REGISTRATION REPORT

Part B

Section 7

Metabolism and Residues

Detailed summary of the risk assessment

Product code: 102000028562

Product name: Deltamethrin + Flupyradifurone EC 85 (10+75 g/L)

Chemical active substances:

Deltamethrin, 10 g/L
Flupyradifurone, 75 g/L

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

(authorization)

Applicant: Bayer Crop Science Division

Submission date: 01/08/2019 (intended)

MS Finalisation date: June 2021 (initial Core Assessment)

February 2022 (final Core Assessment)

Version history

When	What
01/08/2019	Original Bayer Crop Science Division submission
June 2021	Initial zRMS assessment. The report in the dRR format has been prepared by the Applicant, therefore all comments, additional evaluations and conclusions of the zRMS are presented in grey commenting boxes. Minor changes are introduced directly in the text and highlighted in grey. Not agreed or not relevant information are ~~struck through and shaded for transparency~~.
February 2022	Final report (Core Assessment updated following the commenting period) Additional information/assessments included by the zRMS in the report in response to comments recieved from the cMS and the Applicant are highlighted in yellow. Information no longer relevant ~~is struck through and shaded~~.

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7 Metabolism and residue data (KCA section 6)

7.1 Summary and zRMS Conclusion

zRMS summary:
This application is submitted for the authorization of the product Deltamethrin + Flupyradifurone EC 85 (10+75 g/L), an EC formulation containing 10 g of the active substance deltamethrin and 75 g of the active substance flupyradifurone per liter for use on:
- Oilseed rape for the control of pests such as *Ceutorhynchus napi*, *Ceutorhynchus quadridens (pallidactylus)*, *Ceutorhynchus obstrictus (assimilis)*, *Brassicogethes (Meli-gethes) aeneus* or *Dasineura brassica*,
- White and black mustard for the control of pests such as *Brassicogethes (Meligethes) aeneus*.

Oilseed rape is the major crop in Northern Europe (Technical Guidelines SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

White and black mustard is a minor crop in the Northern zone of Europe. Therefore, 4 NEU trials are therefore required to support the use of Deltamethrin + Flupyradifurone EC 85 on mustard in NEU.

In accordance with Technical Guidelines SANTE/2019/12752 eight residue trials on rapeseeds/canola seeds (0401060) for foliar treatments before and after forming of the edible part can be extrapolated to support mustard seeds (0401080). Therefore the data cover the use on white and black mustard.

All comments, additional evaluations and conclusions of the zRMS are presented in grey commenting boxes below.

Conclusion:
~~Considering the intended uses in oilseed rape, an exceedance of the default MRL of 0.01 mg/kg for flupyradifurone (expressed as flupyradifurone), as established in Commission Regulation (EU) 2016/1902, is expected. The current MRL of 0.05 mg/kg for DFA (expressed as DFA) will also also be exceeded in oilseed rape seed. Therefore until the new MRLs for flupyradifurone and its metabolite DFA come into force, authorisation of the GAP (oilseed rape and mustard) will not be possible.~~
In the meantime new MRLs (0.3 mg/kg) for oilseed rape and mustard seed were set for flupyradifurone and DFA in Commission Regulation 2021/1842 of 20 October 2021, applying from 10 November 2021 on.
Available results of residue trials show that the in force MRLs of flupyradifurone and DFA on oilseed rape seed and mustard of 0.3 mg/kg (Reg. (EU) 2021/1842) will not be exceeded. The current EU MRLs for flupyradifurone and DFA are sufficient to support the proposed uses.
Additionally, there is no sufficient data to support application of DLT+FPF EC 85 at flowering oilseed rape. Following the review done by zRMS, the applicant would like to request to restrict the use of the product to applications outside of flowering.

7.1.1 Critical GAP(s) and overall conclusion

Selection of critical uses and justification
The critical GAPs of the central registration zone with respect to consumer intake and risk assessment for the preparation Sivanto Energy (DLT+FPF EC 85) are presented in Table 7.1-1. All these GAPs are less critical as compared to the cGAP of the southern registration zone which is the overall critical GAP for Europe. All supervised residue trials have been conducted with the cGAP of the southern zone in order to follow the risk envelope approach. A list of all intended uses within the central zone is given in Part B, Section 0.

The critical GAPs for the use of Sivanto Energy EC 85 in oilseed rape (rapeseeds) were identified based on the rationale below. As deltamethrin shows no residues above the LOQ in oilseed rape, potential flupyradifurone and DFA residues were used as basis for the cGAP selection:

The foliar use of Sivanto Energy in oilseed rape (OSR) will be supported by three GAPs in the central zone and by one in the southern zone (see also Appendix A 2.1.3).
The GAP of the southern zone comprises two applications of the product Sivanto Energy, each at 0.75 L/ha, corresponding to 56.25 g flupyradifurone/ha. The applications can be done at crop stages

ranging from BBCH 30 to BBCH 79. The minimum interval between applications is 14 days and the PHI is 45 days.
S-EU: 2 x 56.25 g/ha, interval 14 d, BBCH 30-79, PHI 45 d
In the beginning, BCS intended to support this GAP also in the central zone. As a consequence a full trial package was conducted in both zones following this GAP. However due to several reasons, the GAP for the central zone had to be adapted.
The three current GAPs to be registered in the central zone are less critical with respect to possible residues as compared to the intended GAP of the southern zone. The first GAP is very similar to the GAP of the southern zone, however with lower application rates and a smaller application window (BBCH 65-79). As a consequence, lower flupyradifurone (FPF) and difluoroacetic acid (DFA) residues can be expected in mature seeds (expected residues can be estimated by scaling down the residues detected in the available residue trials).
The second and the third GAP support the same application rates as the GAP of the southern zone, however application is only possible before flowering (BBCH 30-49 or BBCH 50-59) and therefore no PHI has to be considered.
N-EU:
(1) 2 x 37.5 g/ha, interval 14 d, BBCH 65-79, PHI 45 d
(2) 2 x 56.25 g/ha, interval 14 d, BBCH 30-49, PHI n.a.
(3) 2 x 56.25 g/ha, interval 14 d, BBCH 50-59, PHI n.a.
In order to show that the second and third GAP are also less critical with respect to possible FPF and DFA residues, the available decline trials have been evaluated. The decline trials show two to three sampling points, covering the harvest of mature seeds at the intended PHI ± 3 days, and an earlier and/or a later sampling.

The residue levels of FPF and DFA are summarized below (for more details please refer to Appendix C):

Trial No./ Location/ EU zone/ Year	**Portion analysed**	**Residues (mg/kg)**				**PHI (days)**
		FPF	**DFA as FPF**	**Sum of FPF and DFA**	**DFA as DFA**	
15-2132-01 15-2132-01-T France, north 37310 Chambourg sur Indre Europe, North F 2015	seed	**0.11** 0.088 0.074	0.21 0.23 0.20	0.32 0.31 0.27	0.070 **0.075** 0.065	42 45 52
15-2132-02 15-2132-02-T Germany 51399 Burscheid Europe, North F 2015	seed	**0.21** 0.21	0.29 0.28	0.50 0.50	**0.095** 0.094	46 53
15-2132-03 15-2132-03-T Germany 59609 Anröchte-Berge Europe, North F 2015	seed	0.12 **0.13** 0.13	0.12 0.14 0.14	0.24 0.27 0.27	0.039 **0.048** 0.047	37 44 51
15-2132-04 15-2132-04-T Belgium 6210 Villers-Perwin Europe, North F 2015	seed	0.088 **0.065** 0.055	0.16 0.20 0.19	0.25 0.27 0.24	0.052 **0.068** 0.063	39 48 54
16-2044-01	seed	0.13	0.21	0.34	0.069	38

Trial No./ Location/ EU zone/ Year	Portion analysed	Residues (mg/kg)				PHI (days)
		FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA	
16-2044-01-T Germany 04824 Beucha OT Wolfshain Europe, North F 2016		**0.15** 0.10	0.20 0.23	0.35 0.32	0.067 **0.075**	45 52
16-2044-02 16-2044-02-T Germany 59609 Anröchte-Berge Europe, North F 2016	seed	0.11 **0.15** 0.087	0.23 0.20 0.16	0.33 0.35 0.25	**0.075** 0.068 0.054	36 42 51
16-2044-03 16-2044-03-T Belgium 6221 Saint-Amand Europe, North F 2016	seed	**0.13** 0.12 0.090	0.14 0.13 0.13	0.26 0.25 0.22	**0.045** 0.043 0.043	34 43 50
16-2044-04 16-2044-04-T France, north 37310 Chambourg sur Indre Europe, North F 2016	seed	**0.066** 0.064	0.19 0.19	0.25 0.25	**0.063** 0.062	48 52

Evaluation of FPF residues:

- in six of eight trials, FPF residues were highest at the intended PHI (42-48 days after the second application) and declined thereafter
- in two of eight trials, FPF residues were already highest 34/42 days after the second treatment

Overall it can be concluded that FPF residues peak approx. 45 days after the last treatment (or even earlier) and decline thereafter.

As the second and third GAP supported in the central registration zone show an earlier application (latest application at BBCH 59, i.e. before flowering), lower FPF residues can be expected in mature seeds at harvest as the time period between the last application and harvest will be longer in the intended GAP as compared to the tested GAP.

Evaluation of DFA residues:

- in five of eight trials, DFA residues were highest at the intended PHI (44-48 days after the second application) and declined thereafter
- in two of eight trials, DFA residues were highest already 34-36 days after the last treatment
- only in one of eight trials, DFA residues were highest after the intended PHI days (52 days after the second application)

As for the FPF residues, it can be concluded that the maximum DFA residues are also detected approx. 45 days after the last treatment and decline thereafter.

As the second and third GAP supported in the central registration zone show an earlier application (latest application at BBCH 59, i.e. before flowering), lower DFA residues can be expected in mature seeds at harvest as the time period between the last application and harvest will be longer in the intended GAP as compared to the tested GAP.

Based on the evaluation of the decline trials it was shown that the overall cGAP for Europe is the GAP of the southern zone. All GAPs to be authorized in the central zone will be less critical with respect to possible flupyradifurone and DFA residues.

Overall conclusion

Flupyradifurone
Thus far, EU MRLs in oilseed rape have only been established for DFA based on rotational crops (EFSA Journal 2015;13(2):4020). Considering the intended uses in oilseed rapes, an exceedance of the default MRL of 0.01 mg/kg for flupyradifurone (expressed as flupyradifurone), as established in Commission Regulation (EU) 2016/1902, is expected. The current MRL of 0.05 mg/kg for DFA (expressed as DFA) will also ~~also~~ be exceeded in oilseed rape seed. Therefore, an MRL Evaluation Report (ER) has already been submitted to the Netherlands (Ctgb) as designated EMS in February 2019 (update March 2019) in order to support this submission for oilseed rape.

The data available are considered sufficient for risk assessment. The chronic and the short-term intakes of flupyradifurone residues are unlikely to present a public health concern.

Deltamethrin
The data available are considered sufficient for risk assessment. An exceedance of the current MRL of 0.2 mg/kg for deltamethrin for oilseed rape as laid down in Reg. (EU) 396/2005 is not expected.
The chronic and the short-term intakes of deltamethrin residues are unlikely to present a public health concern.
As far as consumer health protection is concerned, the zRMS (Poland) agrees with the authorization of the intended uses in oilseed rape.

According to available data, no specific mitigation measures should apply.

Data gaps

Data gaps identified for **flupyradifurone** in the EFSA Conclusion (EFSA Journal 2015; 13(2):4020) are:

Field rotational crop studies considering realistic plant back intervals for the crops considered and providing information on the flupyradifurone and DFA residue levels in soil are required.

Animal feeding studies conducted with the DFA metabolite are required.

These field rotational crop studies have been conducted showing flupyradifurone and DFA residues in re-cropped plants (root and tuber crops, small grain cereals, brassica vegetables, oilseeds and fruits). These additional data are presented within this submission as they were not yet evaluated at EU level, however submitted to the Netherlands (Evaluation Report prepared under Article 8 of Regulation (EC) No 396/2005; MRL application on the setting of MRLs in various rotational crops and animal commodities; March 2018, updated July 2018).

The additional feeding studies with the metabolite DFA were also not yet evaluated at EU level. Therefore summaries are given in this dRR. As for the rotational crop data, the data were already submitted to the Netherlands (see above).

Data gaps were identified for **deltamethrin** in EFSA's reasoned opinion on the review of the existing MRLs for deltamethrin according to Article 12 of Regulation (EC) N° 396/2005 (EFSA Journal 2015;13(11):4309). Bayer has submitted on October 2018 an evaluation report to the RMS, which compiled the available MRL confirmatory information or a justification as to why the data are not available yet.

Table 7.1-1: Acceptability of critical GAPs (northern residue zone, only)

1	2	3	4	5	6	7		8				9			10	11
GAP number (see part B.0)*	**Crop and/ or situation ****	**Zone**	**Product code**	**F, Fn, Fpn G, Gn, Gpn or I*****	**Pests or Group of pests controlled**	**Formulation**		**Application**				**Application rate per treatment**			**PHI (days)**	**Conclusion**
						Type	**Conc. of as**	**method kind**	**growth stage & season**	**number min max**	**interval between applications (min)**	**kg as/hL min max**	**water L/ha min max**	**kg as/ha min max**		
1, 2,5, 6, 7, 8, 11, 12, 17, 18, 23, 24, 27, 28	Oilseed rape (0401060)	NEU	Sivanto Energy (DLT+FPF EC 85)	F	Biting insects	EC	085	spray	BBCH 30-49	2	14	DLT: 1.25-3.75 FPF: 9.375-28-125	200-600	DLT: 7.5 FPF: 56.25	As per growth stage	A
13, 14, 19, 20, 31 to 34	Oilseed rape (0401060) Mustard seed (0401080)	NEU	Sivanto Energy (DLT+FPF EC 85)	F	Biting insects	EC	085	spray	BBCH 50-59	2	14	DLT: 1.25-3.75 FPF: 9.375-28-125	200-600	DLT: 7.5 FPF: 56.25	As per growth stage	A
3, 4, 9, 10, 15, 16, 21, 22, 25, 26, 29, 30	Oilseed rape (0401060)	NEU	Sivanto Energy (DLT+FPF EC 85)	F	Biting insects	EC	085	spray	~~65~~ 71-79	2	14	DLT: 0.83-2.5 FPF: 6.25-18.75	200-600	DLT: 5 FPF: 37.5	45	A See remark below

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

** Use also code numbers according to Annex I of Regulation (EU) No 396/2005

*** F: professional field use, Fn: non-professional field use, Fpn: professional and non-professional field use, G: professional greenhouse use, Gn: non-professional greenhouse use, Gpn: professional and non-professional greenhouse use, I: indoor application

Explanation for Column 11 "Conclusion"

A	Exposure acceptable without risk mitigation measures, safe use
R	Further refinement and/or risk mitigation measures required
N	Exposure not acceptable, no safe use

Remark:

~~The intended use patterns supported for Sivanto Energy in oilseed rape and mustard seeds will not be covered by the currently approved MRLs for flupyradifurone and its metabolite DFA. Until the new MRLs for flupyradifurone and its metabolite DFA come into force, authorisation of the GAP (oilseed rape and mustard) will not be possible.~~

In the meantime new MRLs (0.3 mg/kg) for oilseed rape and mustard seed were set for flupyradifurone and DFA in Commission Regulation 2021/1842 of 20 October 2021, applying

from 10 November 2021 on.
Available results of residue trials show that the in force MRLs of flupyradifurone and DFA on oilseed rape seed and mustard of 0.3 mg/kg (Reg. (EU) 2021/1842) will not be exceeded. The current EU MRLs for flupyradifurone and DFA are sufficient to support the proposed uses.
Additionally, there is no sufficient data to support application of DLT+FPF EC 85 at flowering oilseed rape (BBCH 60-69). Following the review done by zRMS, the applicant would like to request to restrict the use of the product to applications outside of flowering (BBCH 60-69).
Authorisation of the GAP with BBCH 30-49, BBCH 50-59 and BBCH 71-79 (oilseed rape and mustard) is possible only.

7.1.2 Summary of the evaluation

The preparation DLT+FPF EC 85 is composed of deltamethrin and flupyradifurone.

Table 7.1-2: Toxicological reference values for the dietary risk assessment of flupyradifurone and deltamethrin

Reference value	Source	Year	Value	Study relied upon	Safety factor
Parent compound flupyradifurone					
ADI	EFSA	2015	0.064 mg/kg bw day	2-generation reproductive toxicity study in rats	100
ARfD	EFSA	2015	0.15 mg/kg bw	rabbit developmental toxicity study	100
Metabolite DFA (from flupyradifurone)					
ADI	EFSA	2015	0.064 mg/kg bw day [1]	2-generation reproductive toxicity study in rats	100
ARfD	EFSA	2015	0.15 mg/kg bw [1]	rabbit developmental toxicity study	100
Parent compound cis-deltamethrin					
ADI	Review report 6504/VI/99-final	2002	0.01 mg/kg bw/d	1 year dog and 90 days dog	100
ARfD	Review report 6504/VI/99-final	2002	0.01 mg/kg bw/d	1 year dog and 90 days dog	100

[1] After an expert consultation it was concluded that the reference values of the parent flupyradifurone are also applicable to DFA (when DFA residues are expressed in flupyradifurone equivalents)

7.1.2.1 Summary for flupyradifurone

Table 7.1-3: Summary for flupyradifurone

Use-No.*	Crop	Plant metabolism covered?	Sufficient residue trials?	PHI sufficiently supported?	Sample storage covered by stability data?	MRL compliance	Chronic risk for consumers identified?	Acute risk for consumers identified?
1 to 30	oilseed rape (OSR)	Yes	Yes OSR 8 NEU (+ 8 SEU)	Yes	Yes	~~No~~[1] Yes	No	No
31 to 34	mustard seed	Yes	Yes OSR 8 NEU (+ 8 SEU)	Yes	Yes	~~No~~[1] Yes	No	No

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

[1] A draft Evaluation Report has already been submitted to cover these use of Sivanto Energy in OSR

~~The intended use patterns supported for Sivanto Energy in oilseed rape will not be covered by the currently approved MRLs for flupyradifurone and its metabolite DFA. As a consequence an MRL Evaluation Report (ER) has been submitted to the Netherlands (Ctgb) as designated EMS in February 2019 (update March 2019) in order to support this submission for oilseed rape.~~

In the meantime new MRLs (0.3 mg/kg) for oilseed rape and mustard seed were set for flupyradifurone and DFA in Commission Regulation 2021/1842 of 20 October 2021, applying from 10 November 2021 on.

Available results of residue trials show that the in force MRLs of flupyradifurone and DFA on oilseed rape seed and mustard of 0.3 mg/kg (Reg. (EU) 2021/1842) will not be exceeded. The current EU MRLs for flupyradifurone and DFA are sufficient to support the proposed uses.

According to Technical Guidelines SANTE/2019/12752, extrapolation from rape oilseeds to mustard seeds is possible for treatments before and after the edible part has formed and thus the proposed MRL (dER, submitted in February 2019) should apply to both oilseeds.

The effects of processing on the nature of flupyradifurone residues have been investigated. Processing studies are available for the oilseeds soybean and cotton. In addition, processing studies are available for potato, barley, wheat, corn (maize), and oilseed rape (as a representative oilseed) which all can be grown in rotation to oilseed rape. Some of the processed commodities of these crops are potential feed items and therefore of interest when estimating the dietary burden of livestock.

Numerous field rotational crop trials have been conducted to support the use of flupyradifurone. Apart from the "main" field study (containing data for three rotations with three crop groups: leafy, root, and cereal crops), further data on individual crops after one rotation simulating crop failure (“small” rotational crop studies on tuber, stem, fruiting, bulb, and legume vegetables, as well as on pulses and oilseeds) have been submitted for the approval of the active substance. These studies have been assessed and summarized in the EFSA peer review (EFSA Journal 2015;13(2):4020). The amount of flupyradifurone, applied either to bare soil or lettuce as target crop, amounted to 200 or 250 g a.s./ha (which was based on the worst-case application rate in the "home & garden" uses, which are more critical as compared to the corresponding agricultural uses).
The design of the field rotational crop studies was discussed at EU level (Pesticides Peer Review expert meeting TC 107). It was questioned if the applied application rate reflects the plateau concentration of flupyradifurone reached in soil following several years of consecutive applications as flupyradifurone is a persistent compound. Moreover, the experts were of the opinion that testing a single plant back interval (PBI) of 30 days (worst-case: crop failure) reflects an unrealistic situation for several crops. It was therefore concluded that further studies conducted at realistic PBIs and providing information on the flupyradifurone and DFA residue levels in soil are required.
In order to overcome above mentioned deficiencies, an assessment was done to identify the most reasonable plateau concentration of flupyradifurone reached in soil following several years of consecutive applications. Based on the results, additional field rotational studies (including soil sampling) were initiated in selected crops in the seasons 2016, 2017 and 2018.
Two scenarios were considered: The multi-year use of flupyradifurone in a “Home & Garden” product and the multi-year use of flupyradifurone by farmers. New field rotational crop studies were conducted in cereals, maize and oilseed rape after application of 175 g a.s./ha and in potato, brassica vegetables and strawberries after application of 300 g a.s./ha to bare soil. This approach considers that crops like cereals, maize and oilseed rape are generally not grown in gardens and thus the agricultural application rates have to be used as reference. In contrast for all crops which can be grown by non-professionals, the "home & garden" use rate has to be taken into account. Considering the possible accumulation of flupyradifurone after multi-year use, exaggerated rates of 175 g a.s./ha and 300 g a.s./ha were chosen to represent the agricultural use rates and the "home & garden" use rates, respectively as agreed with the RMS (Spiegel, K.; 2016; M-617654-01-1). The rates tested cover all the intended uses of this dRR.

The results of these additional field rotational crop studies are summarized in the present dRR as they are not yet approved on EU level, although already submitted (dER March (confirmatory data), dER June (import tolerances), updated in July 2018).

The use in oilseed rape relevant to this submission as well as the new mode of calculation, modified the theoretical maximum daily intake for animals as compared to the intake estimated in the EFSA Conclusion on the peer review (EFSA, 2015). However, all increased MRLs will be already covered by the Evaluation Reports which have been submitted in March and July 2018 (dER (confirmatory data), dER (import tolerances) and dER for SL 200) in order to establish import tolerances for plant and animal commodities and also to support the intended uses of this dRR.

It should be noted that there is no sufficient data to support application of DLT+FPF EC 85 at flowering oilseed rape (BBCH 60-69). Following the review done by zRMS, the applicant would like to request to restrict the use of the product to applications outside of flowering.

The proposed uses of flupyradifurone in the formulation Sivanto Energy (DLT+FPF EC 85) do not represent unacceptable chronic or acute risks for the consumer.

7.1.2.2 Summary for deltamethrin

Table 7.1-4: Summary for deltamethrin

Use-No.*	Crop	Plant metabolism covered?	Sufficient residue trials?	PHI sufficiently supported?	Sample storage covered by stability data?	MRL compliance	Chronic risk for consumers identified?	Acute risk for consumers identified?
1 to 30	oilseed rape (OSR)	Yes	Yes OSR 8 NEU (+ 8 SEU)	Yes	Yes	Yes	No	No
31 to 34	mustard seed	Yes	Yes OSR 8 NEU (+ 8 SEU)	Yes	Yes	Yes	No	No

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

7.1.2.3 Summary for DLT+FPF EC 85

Table 7.1-5: Information on DLT+FPF EC 85 (KCA 6.8)

Crop	PHI for DLT+FPF EC 85 proposed by applicant	PHI/ Withholding period* sufficiently supported for		PHI for DLT+FPF EC 85 proposed by zRMS	zRMS Comments (if different PHI proposed)
		Deltamethrin	Flupyradifurone		
Oilseed rape	45 days	NR	Yes	45 days	for BBCH ~~65~~71-79 PHI - 45 days, for BBCH 30-49 and 50-59 PHI - F
Mustard seed	45 days	NR	Yes	F	see GAP for mustard seeds: BBCH 50-59, PHI is defined by the application stage at last treatment (F)

NR: not relevant
* Purpose of withholding period to be specified
** F: PHI is defined by the application stage at last treatment (time elapsing between last treatment and harvest of the crop).

Table 7.1-6: Waiting periods before planting succeeding crops

Waiting period before planting succeeding crops			Overall waiting period proposed by zRMS for DLT+FPF EC 85
Crop group	Deltamethrin	Flupyradifurone	
Oilseed rape	NR	NR	NR
Mustard seed	NR	NR	NR

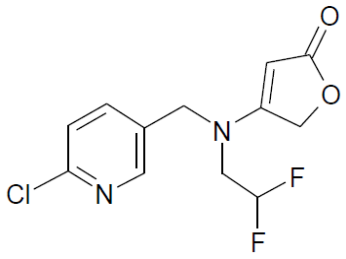
NR: not relevant

Assessment

7.2 Flupyradifurone

General data on flupyradifurone are summarized in the table below.

Table 7.2-1: General information on flupyradifurone

Active substance (ISO Common Name)	Flupyradifurone
IUPAC	4-[(6-chloro-3-pyridylmethyl)(2,2- difluoroethyl)amino]furan-2(5H)-one
Chemical structure	
Molecular formula	$C_{12}H_{11}ClF_2N_2O_2$
Molar mass	288.68 g/mol
Chemical group	Butenolide
Mode of action (if available)	Nicotinic acetylcholine receptor (nAChR) agonist
Systemic	Yes
Company (ies)	Bayer AG, Crop Science Division*
Rapporteur Member State (RMS)	The Netherlands
Approval status	Approved Commission Regulation 2015/2084 of 18 Nov. 2015 (entry into force: 9 Dec 2015)
Restriction (e.g. is restricted to use as "...")	Confirmatory information has to be submitted, see Commission Regulation 2015/2084
Review Report	SANTE/11649/2015 rev 1 (Oct 9, 2015)
Current MRL regulation	Commission Regulation (EU) 2016/1902 of 27 October 2016, (entry into force: 24 November 2016); SANTE/10757/2016 [Annex IIIA] SANTE/11428/2020 Commission Regulation 2021/1842 of 20 October 2021, applying from 10 November 2021.
Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed	No
EFSA Journal : Conclusion on the peer review	Yes, EFSA Journal 2015; 13(2):4020 (updated version of March 2016)
EFSA Journal: conclusion on article 12	No
Current MRL applications on intended uses	An MRL application was submitted to the NL (EMS) in February 2019 in order to apply for a modification of the MRL in rapeseeds/canola seeds, and mustard seeds EFSA Journal 2020;18(11):6298

* Notifier in the EU process to whom the a.s. belong(s)

7.2.1 Stability of Residues (KCA 6.1)

7.2.1.1 Stability of residues during storage of samples

Available data

The stability of flupyradifurone and its metabolite difluoroacetic acid (DFA) was evaluated in the final

addendum to the DAR (NL, February 2015) and peer reviewed by EFSA (EFSA Journal 2015;13(2):4020).
At the time of evaluation, the storage stability data for plant matrices were based on an interim report, only. In the meantime storage data are available for up to 52 months which are submitted in the framework of this application (Netzband, D.; 2015; M-428412-03-1). The results are summarized in Table 7.2-2 and in more detail in Appendix 2.

During the transport and storage of samples from supervised residue trials of oilseed rape (studies 16-2131 and 16-2169), storage temperature deviations occurred. The worst-case being a temperature raise during a time period of 13 hours with a maximum temperature of -8.5°C, resulting in an average temperature of -15°C during these 15 hours. Since none of the samples thawed, the stability of residues can be assumed, particularly when considering that several short-term temperature increases ≥0°C (up to 6.5°C) were also observed in the long-term stability study.
Two short-term storage stability studies at exaggerated temperatures are available, which show the stability of flupyradifurone and DFA residues in matrices of high water (forage) and high fat content (seeds) under similar conditions:

- The first study is available for matrices with high water, high acid, high protein and high starch content (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1). Stability of flupyradifurone and DFA residues was shown when samples were stored for 8 hours at +1°C, followed by 6-8 days at -7°C. Unfortunately no matrices with high oil content were included in this study.
- Within the second study (Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1) residues of flupyradifurone and DFA were found to be stable in samples of oilseed rape (seeds and straw) when stored for 10 days at -8.5°C followed by 5 hours at +6°C.

Both studies are summarized in Appendix 2.

The available storage stability studies for flupyradifurone and DFA at exaggerated temperatures confirmed the storage stability of relevant flupyradifurone residues during storage and validate the residue results of the residue studies.

The longest storage duration relevant to this submission was detected for oilseed rape seed and amounted to 799 days (approx. 27 months). As the long-term storage stability study covers a period of at least 52 months, the maximum storage duration relevant to this submission is well covered by storage stability data for flupyradifurone and DFA.

During the in-life portion of the difluoroacetic acid feeding studies animal samples were briefly stored at temperatures above -18ºC for a period of about 24 hours with a maximum recorded temperature of -2.5ºC. Therefore a short-term storage stability study was conducted as a part of the poultry feeding study ([redacted] 2017; M-611007-01-1) which confirmed the stability of difluoroacetic acid in animal matrices when stored for 24 hours at refrigerator conditions (cf. ~~Błąd! Nie można odnaleźć źródła odwołania.~~). The results are summarized in Table 7.2-2.

Table 7.2-2: Summary of stability data achieved at ≤ - 18°C (unless stated otherwise)

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference
Data relied on in EU			
Plant products			
Sugar cane Spinach leaves Tomato fruit	High water content	Flupyradifurone (BYI 02960), Difluoroacetic acid (DFA): 18 months	Netzband, D.; Timberlake, B. C.; Harbin, A. M.; 2012; M-428412-02-1 RARVP046-1 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
Soybean seed Coffee bean	High oil content		
Bean seed	High protein content		
Wheat grain	High starch content		
Orange fruit	High acid content		

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference
Animal Products			
Poultry For parent flupyradifurone and metabolite DFA, no storage stability testing in poultry matrices is required, as the longest storage duration of the samples was < 1 month in the poultry feeding study.			
Ruminant			
For parent flupyradifurone, no storage stability testing for ruminant matrices is required, as the longest storage duration of the samples was < 1 month in the cattle feeding study.			
Bovine	Muscle	Difluoroacetic acid (DFA): 43 days	[redacted] 2012; M-428416-02-1, RARVP050 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
Bovine	Liver		
Bovine	Kidney		
Bovine	Fat		
New data			
Plant products			
Sugar cane Spinach leaves Tomato fruit	High water content	Flupyradifurone (BYI 02960), Difluoroacetic acid (DFA): 52 months	Netzband, D.; 2015; M-428412-03-1 RARVP046-2 (amended version of RARVP046-1), Appendix 2
Soybean seed Coffee bean	High oil content		
Bean seed	High protein content		
Wheat grain	High starch content		
Orange fruit	High acid content		
Plant products Storage at exaggerated temperature			
Tomato fruit Wheat green material	High water content	Flupyradifurone (BYI 02960), Difluoroacetic acid (DFA): 8 hours at +1°C followed by 6-8 days of storage at -7°C	Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1 S13-03307 Appendix 2
Peas, dry	High protein content		
Wheat grain Potato tuber	High starch content		
Grape bunches	High acid content		
Oilseed rape seeds	High oil content	Flupyradifurone (BYI 02960), Difluoroacetic acid (DFA): 10 days at -8.5°C followed by 5 hours at +6°C	Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1 Appendix 2
Oilseed rape straw	Dry commodity		
Animal Products			
Poultry/Ruminant Storage at exaggerated temperature			
Poultry	Eggs	Difluoroacetic acid (DFA): 2 days (at refrigerator conditions)	[redacted] 2017; M-611007-01-1 RARVN150 Appendix 2
Poultry	Muscle		
Poultry	Liver		
Poultry	Fat		
Dairy cow	Milk		
Dairy cow	Kidney		

Conclusion on stability of residues during storage

Residues of flupyradifurone and its metabolite difluoroacetic acid (DFA) were shown to be stable for up

to 52 months in soybean (seed), coffee (bean), sugarcane, spinach (leaves) and tomato (fruit), bean seed, wheat grain and orange fruit in frozen storage at ≤ -18°C.
The maximum storage period of frozen samples obtained from the plant residue studies (field residue, rotational crop or processing studies) included in this dRR are shown in the table below.

The maximum storage period of oilseed rape seed in residue studies was 799 days (approx. 27 months) and is the longest storage duration relevant to this submission.
Hence, the results of the storage stability studies validate the residue values with respect to the stability of flupyradifurone and its metabolite DFA in deep-frozen samples relevant to this submission.

Two studies performed at exaggerated temperatures during storage validate the results of the residue studies with respect to stability of flupyradifurone and DFA in rape seed.

The results of the storage stability study performed at exaggerated temperatures for animal matrices validate the results obtained from the feeding studies with respect to the stability of DFA in egg, muscle, liver, fat, milk and kidney.

Sample material		Longest storage	Study	
Crop	**Matrix**	**duration (d)**	**Report No.**	**dRR**
Oilseed rape	green material	171		
	straw	147	15-2132	A 2.1.3.1.1
	seed	116		
	green material	619		
	straw	571	16-2044	A 2.1.3.1.2
	seed	571		
	green material	799		
	straw	766	16-2131	A 2.1.3.1.3
	seed	756		
	green material	736		
	straw	702	16-2169	A 2.1.3.1.4
	seed	684		
	green material	218	16-2511	A 2.1.6.1.2
	seed	113		
	green material	157	16-2521	A 2.1.6.1.3
	seed	97		
Soybean	soybean seed	254		
	aspirated grain fractions	221		
	meal	254		
	hulls	169	RARVY029	A 2.1.5.2.1
	refined oil	171		
	soymilk	170		
	defatted flour	162		
Cotton	cotton seed	449		
	oil, refined	176		
	oil, solvent extracted	187		
	crude oil, pre-clarified	189	RARVY033	A 2.1.5.2.2
	crude oil, neutralized	189		
	meal	190		
	hulls	185		
Potato	tuber	204		
	tuber, washed	197		
	tuber with peel, cooked	209		
	tuber, peeled	198	RARVY038	0
	tuber, steamed, mashed	209		
	starch	201		
	flakes	202		
	crisps (chips)	215		
	tuber	699		
	tuber, washed	701		
	peel washed	701		
	tuber, peeled	701		
	tuber, cooked	701		
	tuber with peel, cooked	701		
	fibre	699	13-3149	A 1.1.1.1.1
	pulp, dry	699		
	starch	700		
	waste	701		
	waste, dried	701		
	flakes	700		
	crisps (Am.: chips)	699		
	tuber	161	16-2510	A 2.1.6.1.1

Sample material		Longest storage	Study	
Crop	Matrix	duration (d)	Report No.	dRR
Barley	grain	328		
	malt sprouts	256		
	brewer´s malt	256		
	brewer´s grain	229		
	hops draff	229	10-3410	A 2.1.5.2.4
	brewer´s yeast	222		
	beer	195		
	pearl barley rub off	266		
	pearl barley	266		
	green material	119		
	grain	92	16-2512	A 2.1.6.1.4
	straw	89		
	green material	139		
	grain	116	16-2522	A 2.1.6.1.5
	straw	119		
Wheat	grain	159		
	semolina bran	160		
	semolina	160		
	white-flour bran	160		
	white flour	160	10-3409	A 2.1.5.2.5
	white bread	160		
	whole meal	160		
	whole-meal bread	160		
	wheat germ	160		
	grain	372		
	aspirated grain fractions	147		
	bran	170		
	cooking water	107		
	cooking water 2	89		
	germ	177		
	gluten	145		
	middlings	126		
	pasta, cooked	90		
	pasta, dried and cooked	90	RARVY031	A 2.1.5.2.6
	pasta, dry	95		
	pasta, fresh	100		
	shorts	170		
	starch	126		
	white bread	106		
	white flour	170		
	whole meal	106		
	whole meal bread	154		
Maize/field corn	grain	218		
	forage	272		
	silage	249		
	aspirated grain fractions	158		
	bran	182		
	flour	166		
	germ, dry milling	173	RARVY030	A 1.1.1.1.1
	germ, wet milling	169		
	grits	172		
	meal, dry milled	181		
	oil, dry milled	176		
	oil, wet milled	179		
	starch	175		
	steep water	182		
	green material	70		
	kernel	47	16-2514	A 2.1.6.1.6
	rest of plant	47		
	green material	92		
	kernel	61	16-2524	A 2.1.6.1.7
	rest of plant	61		

Evaluator comment:
Information given by the Applicant is sufficient.
The endpoints, as presented in EFSA Journal 2015;13(2):4020 are summarized below:

Stability of residues (Annex IIA, point 6 introduction, Annex IIIA, point 8 Introduction)
Flupyradifurone DFEAF, and DFA stable when stored frozen (≤-20°C) for at least 18 months in:
- high acid (orange)
- high water (spinach, sugar cane and tomato)
- high starch (wheat grain)
- high protein (bean seed)
- and high oil matrices (soybean seed)
- specific matrix (coffee bean)

DFA is stable in animal fat, kidney, liver, and muscle for at least 43 days, when stored frozen at (≤-20°C).

In the EFSA Journal 2020;18(6):6133 it is stated that the storage stability of flupyradifurone and DFA has been investigated in the EU pesticides peer review; on the basis of an interim study report, it was concluded that both compounds are stable for at least 18 months in high water, high acid, high oil, high protein and high starch content matrices when stored frozen at approximately -18°C (EFSA, 2015).
The applicant now submitted the fnal report of the above-mentioned study, where stability of flupyradifurone and DFA residues was investigated for longer storage periods of 35 and 52 months.The submitted data confrm freezer storage stability of both compounds at -18°C for 52 months in matrices with high water content (spinach, tomato, sugarcane), high acid content (orange fruit), high oil content (soyabean), high protein content (bean), high starch content (wheat grain) and coffee beans.
It should be noted that address storage stability in one commodity from each of the five commodity categories, an extrapolation to all other crops is possible (in line with paragraph 25 of OECD Guideline 506) and accepted.
The residue trial samples of crops under consideration were stored under conditions for which the stability of flupyradifurone and DFA residues has been demonstrated.
No further data are required.

7.2.1.2 Stability of residues in sample extracts (KCA 6.1)

Since the validity of the methods depends on factors such as reproducibility and the possibility of interruption during the work process, it must be ensured that the stability of relevant residues during possible storage of extracts is always guaranteed. The storage stability of flupyradifurone-related residues in extracts was tested during development of the analytical methods.
During the course of the method validations, stability was shown in all tested matrix extracts for at least 4-8 days, when stored in the dark in a refrigerator at 4°C ± 3°C.

Additionally, relevant information on the stability of residues in the final or any intermediate extracts can be derived from the fortification experiments performed during sample analysis. Every analytical batch does contain at least one freshly fortified sample for concurrent recovery determination. The extracts of the fortified samples and of the study samples are handled and stored in parallel. If the recoveries in the fortified samples are within acceptable ranges, the stability of the sample extracts is considered as sufficiently proven.

Conclusion on stability of residues in sample extracts
It can be concluded, that flupyradifurone and its metabolite DFA were stable for at least 4-8 days when stored in the dark in a refrigerator at 4°C ± 3°C. In addition, relevant information on the stability of residues in the final or any intermediate extracts can be derived from the fortification experiments performed during sample analysis. Every analytical batch contained at least one freshly fortified sample for concurrent recovery determination. The extracts of the fortified samples and of the study samples were handled and stored in parallel. Since the recoveries in the fortified samples were within acceptable ranges, the stability of the sample extracts was considered to be sufficiently proven.

7.2.2 Nature of residues in plants, livestock and processed commodities

7.2.2.1 Nature of residue in primary crops (KCA 6.2.1)

Available data

The metabolism of flupyradifurone has been investigated in apple, tomato, potato, cotton, and rice (representing five diverse crops) after foliar, soil, or seed treatment application with ^{14}C-fluprydifurone. These studies have already been reported and evaluated in the final addendum to the DAR (NL, 2015) and peer reviewed by EFSA (EFSA Journal 2015;13(2):4020). The results are summarized in Table 7.2-3.

No new data are submitted in the framework of this application.

Table 7.2-3: Summary of plant metabolism studies

Crop Group	Crop	Label position	Application and sampling details					Reference
			Method, F or G (a)	Rate (g a.s./ha)	No	Sampling (DALT)	Remarks	
EU data								
Fruits and fruiting vegetable	Apple	[furanone-4-^{14}C]	Foliar, F[1]	86 g a.s./(ha x mCH)	1	leaves and fruit: 98 days; BBCH 87-89	-	Justus, K.; 2011; M-422562-01-1 MEF-11/499 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
				86 g a.s./(ha x mCH)	2	leaves and fruit: 14 days; BBCH 87-89		
		[pyridinyl-methyl-^{14}C]	Foliar, F[1]	87 g a.s./(ha x mCH)	1	leaves and fruit: 98 days; BBCH 87-89	-	Justus, K.; 2011; M-414678-01-2 MEF-11/198 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
				86 g a.s./(ha x mCH)	2	leaves and fruit: 14 days; BBCH 87-89		
	Tomato	[furanone-4-^{14}C]	Soil drench, G	300	2	flowers: 6-36 days[2]; BBCH 61-69 fruit: 69-92 days; BBCH 81-89	-	Justus, K.; 2011; M-411352-01-3 MEF-11/016 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
		[pyridinyl-methyl-^{14}C]		300	2	flowers: 3-36 days[2]; BBCH 61-69 fruit: 73-92 days; BBCH 81-89	-	Justus, K.; 2011; M-411500-01-2 MEF-11/498 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020

Crop Group	Crop	Label position	Application and sampling details					Reference
			Method, F or G (a)	Rate (g a.s./ha)	No	Sampling (DALT)	Remarks	
Fruits and fruiting vegetable (cont.)		[ethyl-1-^{14}C]		300	2	flowers: 1-32 days[2]; BBCH 61-69 fruit: 56-86 days; BBCH 81-89	-	Unold, M.; Justus, K; 2011; M-413996-01-2 MEF-11/016 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
Root and tuber vegetables	Potato	[furanone-4-^{14}C]	Tuber treatment, F[1]	10 g a.s./dt = 254 g a.s./ha	1	tuber: 97 days; BBCH 97	-	Justus, K.; 2011; M-415234-01-2 MEF-10/769 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
			In-furrow appl. at planting, F[1]	626	1	tuber: 97 days, BBCH 97		
		[pyridinyl-methyl-^{14}C]	Tuber treatment, F[1]	10 g a.s./dt = 254 g a.s./ha	1	tuber: 97 days, BBCH 97	-	Justus, K.; 2011; M-415078-01-2 MEF-10/710 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
			In-furrow appl. at planting, F[1]	626	1	tuber: 97 days, BBCH 97		
Pulses and oilseeds	Cotton	[furanone-4-^{14}C]	Foliar, G	209	1	intermediate: 28 days BBCH 22-24 gin trash, lint, seed: 169 days; BBCH 99	-	Schmeling, S.; Weber, E.; 2011; M-421625-01-1 MEF-11/392 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
			Foliar, G	193	2	gin trash, lint, seed: 14 days; BBCH 99		
		[pyridinyl-methyl-^{14}C]	Foliar, G	1) 206 2) 176	1	intermediate: 28 days BBCH 22-24 gin trash, lint, seed: 169 days; BBCH 99	-	Schmeling, S.; Weber, E.; 2011; M-421691-01-1 MEF-11/393 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
			Foliar, G	1) 206 2) 177	2	gin trash, lint, seed: 15 days; BBCH 99		
Cereals	Paddy rice	[furanone-4-^{14}C]	Soil granule at transplanting, G	0.409	1	kernels, husks, straw: 127 days, BBCH 89-92	-	Schmeling, S.; Weber, E.; 2011; M-414219-01-2 MEF-11/058 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
			Foliar, G	1) 175 2) 240	2	kernels, husks, straw: 29 days, BBCH 89-92		
		[pyridinyl-methyl-^{14}C]	Soil granule at transplanting, G	0.434	1	kernels, husks, straw: 127 days, BBCH 89-92	-	Schmeling, S.; Weber, E.; 2011; M-414328-01-2 MEF-11/059 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
			Foliar, G	1) 178 2) 236	2	kernels, husks, straw: 29 days, BBCH 89-92		

[1] The plants were grown in the glass-roofed vegetation area of the test facility which allows cultivation similar to natural

temperature and light conditions, but protected from rainfall.

[2] Tomato: newly opened tomato flowers were sampled 2-3 times a week starting at 6 days after the last application and continuing over a period of 30 days (until end of flowering). Fully ripe fruits were harvested 2-3 times a week from the remaining three plants, until no fruits were produced anymore (BBCH 89).

(a) Outdoor/field application (F) or glasshouse/protected/indoor application (G)

(b) F or G not reported

Summary of plant metabolism studies reported in the EU

Excerpt from EFSA Conclusion on the peer review, 2015:

Flupyradifurone metabolism in primary crops was investigated in four crop groups either by foliar applications (apple, cotton, rice), by soil granule/drench applications (tomato, potato, rice) and by seed dressing (potato). Studies were conducted using ^{14}C-flupyradifurone labelled on the pyridinyl or furanone moiety and using application rates representative of the supported uses. One study on tomato using soil drench application and a ^{14}C-labelling on the difluoroethyl amino group was also submitted.

The metabolism in primary crops was seen to be similar in all plant groups investigated. Flupyridafurone was consistently observed as the major component of the radioactive residues, accounting for ca. 25% to 88% TRR in all plant parts analysed. Besides flupyradifurone, the following metabolites were identified in different plant matrices:

- *the conjugate flupyradifurone-hydroxy-glycoside, up to 36%TRR in apple leaves,*
- *the conjugate CHPM-diglycoside, up to 37% TRR (0.06 mg/kg) and the metabolite 6-CNA in the range of 13% to 22% TRR in tomato fruit, potato tuber and cotton seed at ca. 0.02 mg/kg, both resulting from the cleavage of the molecule at ethylamine bond and containing the pyridinyl moiety.*

In contrast, metabolites containing the furanone moiety were almost not detected and the radioactivity in the ^{14}C-furanone studies was mostly recovered as incorporated in natural glucoside and carbohydrate components, indicating an extensive degradation of the furanone counterpart. In addition, information was given by the study conducted on tomato with the ^{14}C-labelling on the difluoroethyl amino group that, following soil drench application, significant proportions (87% TRR) and levels (0.17 mg/kg) of difluoroacetic acid (DFA) are present in tomato fruits. Samples from the radiolabelled studies were therefore re-analysed for non-radiolabelled DFA and residues, expressed as DFA equivalent, were measured in the range of 0.04 to 0.23 mg/kg in apple fruits, potato tuber, cotton seed and rice grain, irrespective of the mode of application. (…)

In primary crops, flupyradifurone is not extensively degraded and the metabolism in plant proceed via the hydroxylation of the furanone ring leading to the M8 metabolite and its glycoside conjugates and via the cleavage of the parent molecule at the ethylamine bond resulting in the formation of metabolites containing the pyridinyl moiety (CHMP-diglycoside, 6-CNA free and conjugated). The furanone counterpart is extensively metabolised and incorporated in natural glycoside or carbohydrate components. (…)

Based on these studies, and considering the outcome of the Peer Review Meeting 122 on toxicology where it was concluded that the toxicological reference values set for flupyradifurone are also applicable to DFA, the residue definition for risk assessment was proposed as "sum of flupyradifurone and DFA expressed as flupyradifurone". For enforcement, the residue definition was intensively discussed in the teleconference meeting 107 (…). It was finally agreed that two separate residue definitions would be required, as "flupyradifurone"; to consider the residues resulting from the uses of the active substance on primary crops, and as "DFA, expressed as DFA"; to consider DFA residues in rotational crops.

Conclusion on metabolism in primary crops

Target crop metabolism studies have been performed on apple, tomato, potato, cotton, and rice (representing five diverse crops from four different crop groups) using foliar, soil, or seed treatment application with ^{14}C-flupyradifurone (labelled in the furanone ring or pyridinylmethyl position) formulated as 200 SL (for foliar and soil applications) and 480 FS (for seed treatment applications).

The metabolism studies were conducted at rates covering the anticipated worst case GAPs. The submitted foliar treatment uses on oilseed rape are covered by the available metabolism studies.

Evaluator comment:
Information given by the Applicant is sufficient.
Flupyradifurone metabolism in primary crops was investigated in the framework of the EUpesticides peer review in four crop groups either by foliar applications (apple, cotton, rice), by soil granule/drench applications (tomato, potato, rice) and by seed treatment (potato). Studies were conducted using ^{14}C-flupyradifurone labelled on the pyridinyl and furanone moiety. One study on tomato using soil drench application and a ^{14}C-labelling on the difuoroethyl amino group was also submitted (EFSA, 2015).The metabolism in primary crops was seen to be similar in all plant groups investigated.

In EFSA Journal 2020;18(6):6133 it is stated that based on the metabolic pattern identifed in primary and rotational crop metabolism studies, the results of hydrolysis studies, the toxicological signifcance of metabolites and/or degradation products,the capabilities of enforcement analytical methods, the following **residue defnitions** were agreed by the EU pesticides peer review (EFSA, 2015):
- Residue defnition for risk assessment: Sum of flupyradifurone and DFA, expressed as flupyradifurone.
- Residue defnition for enforcement: 1) Flupyradifurone; 2) DFA, expressed as DFA.

The same residue defnitions are applicable to rotational crops and processed products.
The residue defnition for enforcement set in Regulation (EC) No 396/2005 is identical with the above mentioned residue defnition for enforcement.
No further data are required.

7.2.2.2 Nature of residue in rotational crops (KCA 6.6.1)

Available data
The nature of the residues in rotational crops has been investigated in three representative crop types, a leafy crop, a root crop and a cereal after application of either [pyridinylmethyl-^{14}C]flupyradifurone or [furanone-4-^{14}C]flupyradifurone to bare soil. These studies have already been reported and evaluated in the final addendum to the DAR (NL, 2015) and peer reviewed by EFSA (EFSA Journal 2015; 13(2):4020). A summary of the confined rotational crop studies is presented in Table 7.2-4.

No new data were submitted in the framework of this application.

Table 7.2-4: Summary of metabolism studies in rotational crops

Crop group	Crop	Label position	Application and sampling details					Reference
			Method, F or G *	Rate (g a.s./ha)	Sowing intervals (DAT)	Harvest Intervals (DAT)	Remarks	
EU data								
Leafy vegetables	Swiss chard	[furanone-4-^{14}C]	Bare soil, F[1], G	436	29, 135 and 296	immature SC DAT: 58, 170, 344 days, BBCH 44-46 mature SC DAT: 76, 197, 359 days BBCH 49	-	Klempner, A.; 2011; M-421861-01-1 MEF 11/365 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
Root and tuber vegetables	Turnips					turnip leaves and roots DAT: 85, 209, 380 days BBCH 45-47		
Cereals	Wheat					wheat forage DAT: 69, 167, 350 days, BBCH 29-31 wheat hay DAT: 101, 237, 401 days, BBCH 79-83 wheat grain and		

Crop group	Crop	Label position	Application and sampling details					Reference
			Method, F or G *	Rate (g a.s./ha)	Sowing intervals (DAT)	Harvest Intervals (DAT)	Remarks	
						straw DAT:134, 294, 440 BBCH 87-92		
Leafy vegetables	Swiss chard	[pyridinyl-methyl-^{14}C]	Bare soil, F[1], G	436	29, 135 and 296	immature SC DAT: 58, 170, 344 days, BBCH 44-46 mature SC DAT: 76, 197, 359 days BBCH 49	-	Breuer-Rehm, M.; 2012; M-419853-02-1 MEF 10/892 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
Root and tuber vegetables	Turnips					turnip leaves and roots DAT: 85, 209, 380 days BBCH 45-47		
Cereals	Wheat					wheat forage DAT: 69, 167, 350 days, BBCH 29-31 wheat hay DAT: 100, 237, 401 days, BBCH 79-83 wheat grain and straw DAT:134, 294, 440 BBCH 87-92		

F[1], G Depending on the season, the different rotations were either conducted in the vegetation hall (F) or in the greenhouse (G) of the test facility.
* Outdoor/field application (F) or glasshouse/protected/indoor application (G)

Summary of plant metabolism studies reported in the EU

Excerpt from EFSA Conclusion on the peer review, 2015:

A similar metabolic profile [compared to primary crops] *was observed in the confined rotational studies conducted with a ^{14}C-pyridinyl and ^{14}C-furanone labelling, where flupyradifurone and its metabolites flupyradifurone-hydroxy, 6-CNA and their conjugates were seen to be the major components of the radio-active residues. These radiolabelled studies are however incomplete as they did not include a labelling on the difluoroethyl amino group. Information provided by rotational crop field studies, indicates that DFA is the major component of the residues in rotational crops. The presence of DFA is mostly the result of a preferential uptake from soil, where this metabolite was identified as a major metabolite. (…)*

Based on these studies, and considering the outcome of the Peer Review Meeting 122 on toxicology where it was concluded that the toxicological reference values set for flupyradifurone are also applicable to DFA, the residue definition for risk assessment was proposed as "sum of flupyradifurone and DFA expressed as flupyradifurone". For enforcement, the residue definition was intensively discussed in the teleconference meeting 107 (…). It was finally agreed that two separate residue definitions would be required, as "flupyradifurone"; to consider the residues resulting from the uses of the active substance on primary crops, and as "DFA, expressed as DFA"; to consider DFA residues in rotational crops.

Additional information:

The metabolism of flupyradifurone was investigated in the rotational crops wheat, Swiss chard and turnips from three consecutive rotations in two separate experiments with [furanone-4-^{14}C]flupyradifurone and [pyridinylmethyl-^{14}C]flupyradifurone.

Since no confined rotational crop study with [ethyl-1-^{14}C]flupyradifurone was available, all samples from the confined rotational crop study with [furanone-4-^{14}C]flupyradifurone were analyzed in addition by LC-MS/MS for difluoroacetic acid (DFA). The analyses revealed that DFA was the main metabolite in

wheat grains (all rotations) and in turnip roots (1st rotation) and was a major metabolite in all other RACs. As a consequence, DFA was included in the data collection method for the field rotational crop studies.

The plant metabolism studies have shown a reasonably consistent metabolic profile across both foliar and soil application. The only residues of flupyradifurone that were consistently observed at significant levels across all primary and succeeding crops were the parent compound flupyradifurone and its metabolite DFA, both of which are specific to flupyradifurone use.

Conclusion on metabolism in rotational crops
These data were peer reviewed as part of the registration decision and were considered to be acceptable (see EFSA Journal 2015;13(2):4020). No additional confined rotational crop studies need to be conducted to support the submitted foliar treatment use on oilseed rape.

Evaluator comment:
Information given by the Applicant is sufficient.
The nature of flupyradifurone in rotational crops (turnips, Swiss chard and wheat) was investigatedin the framework of the EU pesticides peer review. Flupyradifurone, labelled at ^{14}C-pyridinyl and^{14}C-furanone moiety, was applied on a bare soil at an application rate of 436 g/ha. Rotational crops were planted 29, 135 and 296 days after the soil treatment. In rotational crops, flupyradifurone and its metabolites Flupyradifurone-hydroxy, 6-CNA and their conjugates were found to be the major components of the radioactive residues (EFSA, 2015). These radiolabelled studies did not include the labelling on the difluoroethyl amino group. Additional field rotational crop studies indicated that DFA is the major component of the residues in rotational crops. The presence of DFA is mostly resulting from the uptake from soil, where this metabolite was identified as a major metabolite (EFSA, 2015).
No further data are required.

7.2.2.3 Nature of residues in processed commodities (KCA 6.5.1)

Available data
Studies investigating the nature of flupyradifurone residues in processed commodities were reported and evaluated in the final addendum to the DAR (NL, 2015) and peer reviewed by EFSA (EFSA Journal 2015;13(2):4020). A summary of the nature of the residues in processed commodities is presented in Table 7.2-5.

No new data is submitted in the framework of this application.

Table 7.2-5: Nature of the residues in processed commodities

Conditions (Duration, Temperature, pH)	Identified compound(s) (%)	Reference
EU data		
Pasteurisation (20 minutes, 90°C, pH 4)	Parent (100%)	Weber, E.; 2011; M-402311-01-2 MEF-10/856 DAR, NL, 2015 EFSA Journal 2015; 13(2):4020
Baking, boiling, brewing (60 minutes, 100°C, pH 5)	Parent (100%)	
Sterilisation (20 minutes, 120°C, pH 6)	Parent (100%)	

The test compound flupyradifurone was stable under all representative conditions of high temperature hydrolysis for simulation of food processing. No significant hydrolysis products of flupyradifurone were detected above an estimated LOD of 0.5% of the total radioactivity.
It was also confirmed that no additional high-temperature hydrolyses have to be conducted with DFA since no additional metabolites will be expected in significant amounts.

Excerpt from EFSA Conclusion on the peer review, 2015:
Flupyradifurone was seen to be stable under standard hydrolysis conditions.

Conclusion on nature of residues in processed commodities
The conditions of high temperature hydrolysis tested (pasteurization, baking, brewing, boiling and sterilisation) are also representative for the processing of the crops relevant to this dossier, such as preparation of oil and rapeseed meal from oilseed rape. Thus, for the central zone, the use to be supported on oilseed rape is sufficiently covered by the metabolism studies on the nature of residues in processed commodities.

Evaluator comment:
Information given by the Applicant is sufficient.
In the EFSA Journal 2020;18(6):6133 it is stated that the effect of processing on the nature of flupyradifurone was investigated in the framework of the EU pesticides peer review (EFSA, 2015). Standard hydrolysis studies showed that flupyradifurone is hydrolytically stable under standard processing conditions of pasteurisation, baking/brewing/boiling and sterilisation.The effect of processing on the nature of difluoroacetic acid (DFA) has not been investigated. Considering the similarity of the structures between trifluoroacetic acid (TFA) and DFA, the applicant proposed a read-across for both acids. The TFA, due to its stability in environment, has been widely studied and is, due to its structure (complete fluoride ion substitution), very stable and thus has no potential for hydrolytic degradation (Lifongo et al., 2010).
EFSA agrees with the EMS that there is sufficient evidence that difluoroacetic acid is stable under standard hydrolysis conditions.
No further data are required.

7.2.2.4 Conclusion on the nature of residues in commodities of plant origin (KCA 6.7.1)

Table 7.2-6: Summary of the nature of residues in commodities of plant origin

Endpoints	
Plant groups covered	Fruits and fruiting vegetable: Tomato (soil drench application), apple (foliar application) Root and tuber vegetables: Potato (seed treatment and in-furrow spray application) Pulses and oilseeds: Cotton (foliar application) Cereals: Paddy rice (foliar application and granule application at transplanting) (EFSA Journal 2015;13(2):4020)
Rotational crops covered	Leafy vegetable: Swiss chard Root and tuber vegetable: Turnip Cereal: Wheat (EFSA Journal 2015;13(2):4020)
Metabolism in rotational crops similar to metabolism in primary crops?	Yes (EFSA Journal 2015; 13(2):4020)
Processed commodities	Hydrolysis studies simulating pasteurisation, boiling and sterilisation: Flupyradifurone was shown to be stable under standard hydrolysis conditions (EFSA Journal 2015; 13(2):4020)
Residue pattern in processed commodities similar to pattern in raw commodities?	Yes (EFSA Journal 2015;13(2):4020)
Plant residue definition for monitoring	Two separate residue definitions (EFSA Journal 2015;13(2):4020): 1) Flupyradifurone (expressed as flupyradifurone) 2) DFA (expressed as DFA)
Plant residue definition for risk assessment	Sum of flupyradifurone and DFA, expressed as flupyradifurone (EFSA Journal 2015;13(2):4020)
Conversion factor from enforcement to RA	not appropriate (EFSA Journal 2015;13(2):4020)

7.2.2.5 Nature of residues in livestock (KCA 6.2.2-6.2.5)

Available data

The metabolism of flupyradifurone has been investigated in lactating goats and laying hens using flupyradifurone radiolabelled in the pyridinylmethyl or the furanone moiety. These studies have already been reported and evaluated in the final addendum to the DAR (NL, 2015) and peer reviewed by EFSA (EFSA Journal 2015;13(2):4020). The results are summarized in Table 7.2-7.

No new data are submitted in the framework of this application.

Table 7.2-7: Summary of animal metabolism studies

Group	Species	Label position	No of animal	Application details		Sample details		Reference
				Rate (mg/kg bw/d)	Duration (days)	Commodity	Time of sampling	
EU data								
Lactating ruminants	Goat	[furanone-4-^{14}C]	1	1.0	5	Milk	Twice daily[1]	[redacted]; 2011; M-419701-01-1 MEF-11/269 DAR, NL, 2015; EFSA Journal 2015;13(2):4020
						Urine and faeces	Daily	
						Tissues (liver, kidney, round and loin muscle, perirenal and omental fat)	After sacrifice	
		[pyridinyl-methyl-^{14}C]	1	1.0	5	Milk	Twice daily[1]	[redacted] 2011; M-421995-01-1 MEF-11/268 DAR, NL, 2015 EFSA Journal 2015;13(2):4020
						Urine and faeces	Daily	
						Tissues (liver, kidney, round and loin muscle, perirenal and omental fat)	After sacrifice	
Laying poultry	Laying hen	[furanone-4-^{14}C]	6	1.05	14	Eggs	Daily[2]	[redacted] 2012; M-422263-01-1 MEF-11/200 DAR, NL, 2015 EFSA Journal 2015;13(2):4020
						Excreta	Daily	
						Tissues (leg and breast muscle, fat, liver, kidney, skin and eggs from ovary/ oviduct)	After sacrifice	
Laying poultry	Laying hen	[pyridinyl-methyl-^{14}C]	6	1.02	14	Eggs	Daily[3]	[redacted]; 2012; M-422162-01-1 MEF-11/199 DAR, NL, 2015 EFSA Journal 2015;13(2):4020
						Excreta	Daily[3]	
						Tissues (leg and breast muscle, fat, liver, kidney, skin and eggs from ovary/ oviduct)	After sacrifice	

[1] Milk was collected twice per day (morning milk directly before the administration and evening milk approx. 8 h after the administration), milk collected 24 to 102 hours after the first administration was pooled and used for metabolic profiling

[2] Eggs were collected every morning before administration; for metabolic profiling, two egg pools were prepared: eggs of days 2 to 7 and eggs of day 8 to sacrifice, Excreta was collected daily, but only the excreta collected 13 days after the first administration was used for metabolic profiling

[3] Eggs were collected every morning before administration; for metabolic profiling, an egg pool was prepared from eggs

collected 3 days after the first administration to sacrifice; Excreta was collected daily, but only the excreta collected 13 days after the first administration was used for metabolic profiling

Summary of animal metabolism studies reported in the EU

EFSA Journal 2015;13(2):4020 summarises and concludes on the livestock metabolism studies.
Excerpt from EFSA Conclusion on the peer review, 2015:
Livestock metabolism studies conducted with ^{14}C-flupyradifurone labelled on the pyridinyl [pyridinylmethyl] *or furanone moiety at the dose rate of ca. 1 mg/kg bw over 5-6 days (goat) or 14 days (hen) were submitted. In ruminant, the degradation of the parent compound was limited, flupyradifurone accounting for 24%-35% in milk and kidney up to 81%-99% in fat. The metabolism was more extensive in hen, and flupyradifurone almost not detected in any poultry matrices in the ^{14}C-furanone study (<3% TRR) and in the range of 1% (liver) to 20% TRR (eggs) in the ^{14}C-pyridinyl study. The main components identified in hen were the hydroxy-flupyradifurone metabolites (18% TRR in eggs) and its sulphate conjugated in fat and liver (16-23% TRR) and the acetyl-AMCP metabolite in egg, fat and muscle (23 to 40% TRR). As for plants, the metabolic picture given by these studies is incomplete as the 14C labelling on the difluoroethyl amino group is missing. The feeding studies conducted with animals dosed with flupyradifurone alone, revealed that DFA is a major marker of the residues in poultry matrices and in a lesser extent, in ruminant matrices. Based on these studies the residue definitions were proposed as "sum of flupyradifurone and DFA expressed as flupyradifurone" for risk assessment and as flupyradifurone and as DFA as for plant two separate residue definitions were proposed for enforcement respectively.*

Additional information and correction of typos in EFSA Conclusion:
Livestock metabolism studies conducted with ^{14}C-flupyradifurone labelled on the pyridinylmethyl or furanone moiety at the dose rate of approx. 1 mg/kg bw over 5-6 days (goat) or 14 days (hen) were submitted. In ruminant, the degradation of the parent compound was less pronounced compared to poultry:
In the ^{14}C-pyridinylmethyl study, flupyradifurone accounted for 89% of the TRR in milk and for 85-99% in liver, muscle and fat; in kidney 35% of the TRR was identified as flupyradifurone. In the ^{14}C-furanone study, flupyradifurone accounted for 24% in milk, the major radioactive residue was assigned to a natural compound (lactose). In fat and muscle again high percentages of flupyradifurone were detected (81-88%), in kidney and liver flupyradifurone accounted for 51-60%. The metabolism was more extensive in hen, and flupyradifurone was almost not detected in any poultry matrices in the ^{14}C-furanone study (<3% of TRR) and in the range of <1% (liver) to 20% (eggs) in the ^{14}C-pyridinyl study. The main components identified the ^{14}C-pyridinylmethyl study was the hydroxy-flupyradifurone metabolite (up to 18% of the TRR in eggs) and its sulphate conjugate (16-23% in fat and liver), as well as the acetyl-AMCP metabolite, which was mainly detected in egg, fat and muscle (23 to 40% of TRR). In the ^{14}C-furanone study, again the instability of the furanone ring was seen resulting in incorporation of C1 and C2-fragments in natural compounds. Endogenous fatty acids accounted for 52-96% in liver, eggs and fat. Since no metabolism studies were conducted with [ethyl-1-^{14}C]flupyradifurone, selected livestock samples were analyzed for difluoroacetic acid (DFA) by high resolution LC-MS. The analysis revealed rather high DFA concentrations and thus it was concluded that DFA has to be included in the data collection method for livestock matrices, besides parent compound, the hydroxy and the acetyl-AMCP metabolite.

Conclusion on metabolism in livestock
Livestock metabolism studies have been conducted in goat and hen. These data were peer reviewed as part of the approval process and were considered to be acceptable (EFSA Journal 2015;13(2):4020).
As forage of oilseed rape, and as well rape meal can be fed to cattle and laying hens, the transfer of flupyradifurone and DFA residues into edible animal matrices cannot be excluded. However, the dose levels in the livestock metabolism studies (approx. 1 mg/kg bw/d) were significantly higher compared to the dietary burdens expected after feeding of oilseed rape matrices which were treated with Sivanto Energy (DLT+FPF EC 85) according to the label.

Evaluator comment:
Information given by the Applicant is sufficient.
The nature of flupyradifurone residues in livestock was investigated in the framework of the EU pesticides peer review (EFSA, 2015).
Based on these studies, the residue definitions were proposed as 'Sum of flupyradifurone and DFA,expressed as flupyradifurone' for risk assessment. For enforcement, to align with the residue definition set for plants, two separate residue definitions were established: 1) flupyradifurone and 2) DFA, expressed as DFA (EFSA, 2015).
No further data are required.

7.2.2.6 Conclusion on the nature of residues in commodities of animal origin (KCA 6.7.1)

Table 7.2-8: Summary on the nature of residues in commodities of animal origin

	Endpoints
Animals covered	Lactating goats (EFSA Journal 2015; 13(2):4020)
	Laying hens (EFSA Journal 2015; 13(2):4020)
Time needed to reach a plateau concentration	9 days in milk, confirmed by cattle feeding study (EFSA Journal 2015; 13(2):4020)
	6 days in eggs, confirmed by feeding study (EFSA Journal 2015; 13(2):4020)
Animal residue definition for monitoring	Two separate residue definitions (EFSA Journal 2015; 13(2):4020): 1) Flupyradifurone (expressed as flupyradifurone) 2) DFA (expressed as DFA)
Animal residue definition for risk assessment	Sum of flupyradifurone and DFA, expressed in flupyradifurone (EFSA Journal 2015; 13(2):4020)
Conversion factor	Not applicable (EFSA Journal 2015;13(2):4020)
Metabolism in rat and ruminant similar	Yes (EFSA Journal 2015;13(2):4020)
Fat soluble residue	No (EFSA Journal 2015;13(2):4020)

7.2.3 Magnitude of residues in plants (KCA 6.3)

7.2.3.1 Summary of European data and new data supporting the intended uses

New studies on the magnitude of residue have been submitted by the applicant in the framework of this application. These studies are summarized in the table below. The detailed assessment of these studies is presented in Appendix 2.

Remark: The tested GAP in the available residue trials (2 x 56.25 g a.s./ha, interval 14 days, BBCH 30-79, PHI 45 days) differs from the intended GAPs of this dRR submission for the central zone as (1) 2 x 37.5 g a.s./ha, interval 14 d, BBCH 65-79, PHI 45 d and 2 x 56.25 g a.s./ha, interval 14 d, (2) BBCH 30-49 and (3) BBCH 50-59, no PHI.
The first GAP (1) is very similar to the tested GAP, however with lower application rates and a smaller application window. As a consequence lower FPF and DFA residues can be expected in mature seeds. The second and the third intended GAPs support the same application rates as the tested GAP, however application is only possible before flowering (BBCH 30-49 and BBCH 50-59) and therefore no PHI has to be considered. In the available decline trials (study 15-2132, A 2.1.3.1.1 and study 16-2044, A 2.1.3.1.2), it can be shown that the residues of FPF and DFA peak approx. 45 days after the last treatment and decline therafter.
Therefore lower FPF and DFA residues can be expected in mature seeds at harvest as the time period between the last application and harvest will be longer in the intended GAPs as compared to the tested GAP.

Please note that the intended cGAP of the southern zone is equivalent to the tested GAP (which was identified as the cGAP for entire Europe). Residues of this cGAP will drive the risk assessment and will be the basis for MRL setting as well (risk envelope approach).

The use pattern supports the harvest of mature seeds of rape and is not intended for harvesting forage as feed item.

Table 7.2-9: Summary of EU reported and new data supporting the intended uses of DLT+FPF EC 85 and conformity to existing MRL

Commodity	Source	Residue zone (N-EU, S-EU, EU, outside EU)	Evaluation GAP Residue levels (mg/kg) E = according to enforcement residue definition RA = according to risk assessment residue definition	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg)[1]	MRL compliance
Oilseed rape, seed → extrapolated to mustard seeds	EFSA, 2015 11-2554 (M-443092-01-1)	EU	GAP on which MRL/EU a.s. assessment is based (rotational crop study): 2 x 0.125 kg a.s./ha on bare soil, re-cropping after 27-41 days E (FPF): 4 x <0.01 E (DFA): 0.017; 0.024; 0.030; 0.051	n.a.			0.01 FPF: 0.3 0.05[4] DFA: 0.3	yes yes
	New trials 15-2132 (M-578527-02-1) 16-2044 (M-645539-01-1)	N-EU	GAP: 2 x 56.25 g a.s./ha, interval = 14 days, PHI = 45 days E (FPF): 0.065; 0.066; 0.088; 0.12; 0.13; 0.15; 0.15; 0.21 E (DFA): 0.043; 0.048; 0.063; 0.068; 0.068; 0.075; 0.075*; 0.095 RA (sum): 0.25; 0.25; 0.27; 0.27; 0.31; 0.35; 0.35; 0.50 Residue values downscaled to the intended application rate: E (FPF): 0.043; 0.044; 0.059; 0.080; 0.087; 0.10; 0.10; 0.14 E (DFA): 0.029; 0.032; 0.042; 0.045; 0.045; 0.050; 0.050*; 0.063 RA (sum): 0.17; 0.17; 0.18; 0.18; 0.21; 0.23; 0.23; 0.33	0.13 0.068 0.29 FPF: 0.084 DFA: 0.045 RA: 0.195	0.21 0.095 0.50 FPF: 0.14 DFA: 0.063 RA: 0.33	n.a. n.a. n.a. FPF: 0.245 DFA: 0.134	FPF: 0.3 DFA: 0.3	yes yes
	New trials 16-2131 (M-643446-01-1) 16-2169 (M-645539-01-1)	S-EU	GAP: 2 x 56.25 g a.s./ha, interval = 14 days, PHI = 45 days E (FPF): 0.024; 0.036; 0.058; 0.066; 0.11; 0.11, 0.13; 0.16* E (DFA): 0.015; 0.033; 0.050*; 0.057; 0.060; 0.063; 0.088; 0.16* RA (sum): 0.069; 0.21; 0.22; 0.22; 0.28; 0.30; 0.32; 0.63*	0.088 0.059 0.25	0.16 0.16 0.63	n.a. FPF: 0.279 n.a. DFA:0.241 n.a	FPF: 0.3 DFA: 0.3	yes yes
	Overall supporting data for cGAPs of this dRR**	EU (N-EU + S-EU)	GAP: 2 x 56.25 g a.s./ha, interval = 14 days, PHI = 45 days E (FPF): 0.024; 0.036; 0.043; 0.044; 0.058; 0.059; 0.065; 0.066; 0.066; 0.080; 0.087; 0.10; 0.10; 0.11; 0.11, 0.12; 0.13; 0.13; 0.14; 0.15; 0.15; 0.16* 0.21 E (DFA): 0.015; 0.029; 0.032; 0.033; 0.043; 0.043; 0.042; 0.045; 0.045; 0.050; 0.050*; 0.050*; 0.057; 0.060; 0.063; 0.063; 0.063; 0.068; 0.068; 0.075; 0.075*; 0.088; 0.095; 0.16* RA (sum): 0.069; 0.17; 0.17; 0.18; 0.18; 0.21; 0.21; 0.22; 0.22; 0.23; 0.23; 0.25; 0.25; 0.27; 0.27; 0.28; 0.30; 0.31; 0.32; 0.33; 0.35; 0.35; 0.50; 0.63*	0.44 0.084 0.062 0.05 0.26 0.22	0.210 0.16 0.16 0.63	0.4 (0.31) FPF: 0.253 0.2 (0.20) DFA: 0.182	0.01 FPF: 0.3 0.05 DFA: 0.3	no yes no yes
	MRL setting for FPF and DFA should be based on the combined datasets of N-EU and S-EU[3]**							

[1] according to SANTE/10572/2016 (Annex IIIA) and Commission Regulation (EU) 2018/1881 according to the Reg. (EU) 2021/1842

[2] A dER was submitted in Q1 2019 to apply for adequate MRLs

[3] Since N-EU and S-EU datasets are not significantly different (U-Test, 5%), both datasets were pooled together to derive an MRL proposal.

(+) Temporary MRL according to Commission Regulation (EU) 2016/1902, 27 October 2016; the European Food Safety Authority identified some information as unavailable. Based on new field rotational crop studies, the MRL has been confirmed and the new data were submitted in an dER im March 2018 and are presented in this dRR .

* Peak residues detected after PHI.

** The intended GAPs of this dRR submission for the central zone ((1) 2 x 37.5 g a.s./ha, interval 14 d, BBCH 65-79, PHI 45 d and 2 x 56.25 g a.s./ha, interval 14 d, (2) BBCH 30-49 and (3) BBCH 50-59, no PHI) differ from the tested GAP in the available residue trials (2 x 56.25 g a.s./ha, interval 14 days, BBCH 30-79, PHI 45 days). However the tested GAP is equivalent to the intended cGAP in the southern zone, which is the cGAP for whole Europe. Residues of this cGAP will drive the risk assessment and will be the basis for MRL setting as well (risk envelope approach).

FPF = Flupyradifurone (BYI 02960); DFA = difluoroacetic acid; HR: Highest residue; STMR: Supervised Trials Median Residue; n.a. = not applicable

7.2.3.2 Conclusion on the magnitude of residues in plants

According to the available data, the intended uses on oilseed rape in the central zone are considered acceptable. It can be expected that even lower residues of flupyradifurone and DFA will be obtained following the intended GAPs of the central registration zone as compared to the tested GAP which is the critical GAP of the southern European registration zone and therefore the overall cGAP of Europe. The MRLs will be established on the basis of the combined residue data collected from the available trials from N-EU and S-EU (conducted according to the cGAP of southern Europe following the risk envelope approach). According to SANCO 7525/VI/95 Rev. 10.3 it is possible to extrapolate from rapeseeds to mustard seeds.

The data submitted show that the MRLs for flupyradifurone and DFA need to be increased for both oilseed rape and mustard seed. An MRL application has already been submitted to the Netherlands as EMS to support the intended uses of this draft registration report.

Please note that the use pattern supports the harvest of mature seeds of rape and is not intended for harvesting forage as feed item.

Evaluator comment:
Oilseed rape is the major crop in Northern Europe (Technical Guidelines SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

White and black mustard is a minor crop in the Northern zone of Europe. Therefore, 4 NEU trials are therefore required to support the use of Deltamethrin + Flupyradifurone EC 85 on mustard in NEU.

In accordance with Technical Guidelines SANTE/2019/12752 eight residue trials on rapeseeds/canola seeds (0401060) for foliar treatments before and after forming of the edible part can be extrapolated to support mustard seeds (0401080). Therefore the data cover the use on white and black mustard.

The proposed GAP of this dRR submission for the central zone is as follows:
Crop: oilseed rape
(1) 2 x 37.5 g FPF/ha, interval 14 d, BBCH 65-79, PHI 45 d;
(2) 2 x 56.25 g FPF/ha, interval 14 d, BBCH 30-49, no PHI,
(3) 2 x 56.25 g FPF/ha, interval 14 d, BBCH 50-59, no PHI.
Crop: mustard seed
(1) 2 x 56.25 g FPF/ha, interval 14 d, BBCH 50-59, no PHI.
FPF - flupyradifurone

The Applicant submitted eight residue trials on oilseed rape, which were conducted in France, Germany and Belgium in 2015 and 2016. Residue trials were designed as decline trials (two/three sampling points at the preharvest intervals (PHIs) of 34–38 days, 42–48 days and 50–54 days) and performed with two applications at a nominal application rate of 56.2 g/ha; the last treatment took place at the BBCH of 73–80.
Residue trials are not fully compliant with any of the intended NEU GAPs.
The Applicant argues that the GAPs with earlier applications at a higher rate are less critical than the GAP with lower rate but later applications (i.e. GAP with a PHI of 45 days). This is confirmed in residue trials, where at longer PHIs of 50–54 days a decline of residues was observed in all trials, except one. In the EFSA Journal 2020;18(11):6298 it is

stated that the EMS disagrees with the Applicant and concludes that intended NEU GAPs number 2 and 3 are not supported by residue trials.
In order to support the NEU GAP number 1, the EMS proposes and EFSA agrees to apply the proportionality principle to potentially account for residues in oilseed rape at a lower application rate of 37.5 g/ha.

1. The study of Schulte, G.; 2017; 15-2132; M-578527-02-1
Since the application rate of the trials is higher than in the NEU GAP number 1, residues should be scaled down:
Residues used for monitoring and risk assessment:
Trial GAP: 2 x 56.25 g a.s./ha, interval = 14 d, PHI = 45 d
Mo (FPF): 0.065; 0.088; 0.13; 0.21 mg/kg
Mo (DFA): 0.048; 0.068; 0.075; 0.095 mg/kg
RA (sum): 0.27; 0.27; 0.31; 0.50 mg/kg

Residue values downscaled to the intended application rate:
Mo (FPF): 0.043; 0.059; 0.087; 0.14 mg/kg
Mo (DFA): 0.032; 0.045; 0.050; 0.063 mg/kg
RA (sum): 0.18; 0.18; 0.21; 0.33 mg/kg.

2. The study of Kaussmann, M.; Kerkering, S.; 2018; M-641044-01-1:
Since the application rate of the trials is higher than in the NEU GAP number 1, residues should be scaled down:
Residues used for monitoring and risk assessment:
Trial GAP: 2 x 56.25 g a.s./ha, interval = 14 d, PHI = 45 d
Mo (FPF): 0.066; 0.12; 0.15; 0.15 mg/kg,
Mo (DFA): 0.043; 0.063; 0.068; 0.075 mg/kg,
RA (sum): 0.25; 0.25; 0.35; 0.35 mg/kg.

Residue values downscaled to the intended application rate:
Mo (FPF): 0.044; 0.080; 0.10; 0.10 mg/kg,
Mo (DFA): 0.032; 0.042; 0.045; 0.050 mg/kg,
RA (sum): 0.17; 0.17; 0.23; 0.23 mg/kg.

The MRL required for the GAP is 0.3 mg/kg. ~~The current MRL for oilseed rape and mustard is 0.01 mg/kg for flupyradifurone and 0.05 mg/kg for DFA. The current MRLs do not support the proposed GAP.~~
~~An EFSA reasoned opinion on the Article 6 application has since been published (EFSA Journal 2020;18(11):6298) and the draft regulation SANTE/11428/2020 is now available regarding raising the MRL for flupyradifurone and its metabolite difluoroacetic acid (DFA) in rapeseeds/canola seeds and mustard seeds, from 0.01 mg/kg (LOQ) to 0.3 mg/kg for flupyradifurone and from 0.05 mg/kg to 0.3 mg/kg for DFA. This is not currently published.~~
~~**Considering the intended uses in oilseed rape, an exceedance of the default MRL of 0.01 mg/kg for flupyradifurone (expressed as flupyradifurone), as established in Commission Regulation (EU) 2016/1902, is expected. The current MRL of 0.05 mg/kg for DFA (expressed as DFA) will also also be exceeded in oilseed rape seed. Therefore until the new MRLs for flupyradifurone and its metabolite DFA come into force, authorisation of the GAP (oilseed rape and mustard) will not be possible.**~~
In the meantime new MRLs (0.3 mg/kg) for oilseed rape and mustard seed were set for flupyradifurone and DFA in Commission Regulation 2021/1842 of 20 October 2021, applying from 10 November 2021 on.

Available results of residue trials show that the in force MRLs of flupyradifurone and DFA on oilseed rape seed and mustard of 0.3 mg/kg (Reg. (EU) 2021/1842) will not be exceeded. The current EU MRLs for flupyradifurone and DFA are sufficient to support the proposed uses.

7.2.4 Magnitude of residues in livestock

7.2.4.1 Dietary burden calculation

Median and maximum dietary burdens were estimated for parent flupyradifurone and separately for its metabolite DFA. In agreement with the EFSA peer review (EFSA, 2015), the dietary burden was calculated based on European crop residue data only. As residues in rotational crops cannot be excluded, the residues on potential succeeding feed items were used as input parameters besides the primary crop data already evaluated and those relevant to this submission. Always the higher residues were taken as input values irrespective if they originated from primary or rotational crops.
Dietary burden calculations are performed using the Excel calculator proposed by EFSA (Animal model 2017a.xls), which considers a total of 9 animal species. Input values are summarized in Table 7.2-10 for flupyradifurone and in Table 7.2-11 for DFA. In agreement with the information given in the calculator, flupyradifurone-related residues in rape forage were not considered in the dietary burden calculation as no specific GAP on forage is intended (the proposed GAP supports the harvest of mature seeds).

Table 7.2-10: Input values for the dietary burden calculation of flupyradifurone (considering the uses authorized in the EU and the use in crops under consideration, as well as the residues in succeeding crops)

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Feed items related to previous evaluation(s)				
Root and tuber vegetables, culls or roots	0.010	STMR (rot. crop): potato (NL / EFSA, 2015)	0.010	HR (rot. crop): potato (NL / EFSA, 2015)
Apple, wet pomace	0.11	STMR (primary crop): apple fruit (0.07) x PF (1.5) (NL, 2015)	n.a.	
Feed items related to the MRL application on rotational crops (March 2018) as well as primary crops relevant to this submission				
Forage and silage of grass	0.010 => 0.010	STMR (rot. crop): barley green material (NL, 2015) => extrapolation to 175 g/ha (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.030 => 0.026	HR (rot. crop): barley green material (NL, 2015) => extrapolation to 175 g/ha (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Straw of cereals and grasses, except maize	0.014	STMR (rot. crop): barley straw (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.032	HR (rot. crop): barley straw (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Forage of legume animal feeds	0.010 => 0.010	STMR (rot. crop): barley green material (NL, 2015) => extrapolation to 175 g/ha (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.030 => 0.026	HR (rot. crop): barley green material (NL, 2015) => extrapolation to 175 g/ha (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Hay and fodder of legume animal feeds	0.014	STMR (rot. crop): barley straw (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.032	HR (rot. crop): barley straw (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Cabbage, head Kale, leaves	0.010	STMR (rot. crop): brassica veg. (cf. Table 7.2-21 and Appendix A 2.1.6.1.10)	0.010	HR (rot. crop): brassica veg. (cf. Table 7.2-21 and Appendix A 2.1.6.1.10)
Fodder beet, mangel	0.010 => 0.015	STMR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. Table 7.2-21)	0.030 => 0.045	HR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. Table 7.2-21)
Tops of beets and turnips	0.010 => 0.015	STMR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. Table 7.2-21)	0.030 => 0.045	HR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. Table 7.2-21)
Corn (maize), stover	0.015	STMR (rot. crop): maize stover (cf. Table 7.2-21 and Appendices A 2.1.6.1.6 and A 2.1.6.1.7)	0.020	HR (rot. crop): maize stover (cf. Table 7.2-21 and Appendices A 2.1.6.1.6 and A 2.1.6.1.7)
Cereal, grains including corn kernels	0.010	STMR (rot. crop): barley grain (cf. Table 7.2-21 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	n.a.	
Oilseedrape, seeds	0.11	STMR (primary crop): rapeseed (cf. Table 7.2-9 and Appendix A 2.1.3.1)	n.a.	
Oilseeds, other than oilseed rape	0.010	STMR (rot.crop): rapeseed (cf. Table 7.2-21 and Appendices A 2.1.6.1.2 and A 2.1.6.1.3)	n.a.	
Pulses, seeds	0.010 => 0.012	STMR (rot. crop): field pea, (NL, 2015) => extrapolation to 300 g/ha (cf. Table 7.2-21)	n.a.	

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Brewer´s grain	0.010	STMR (rot. crop): barley grain (0.01) x PF (1.0) (cf. Table 7.2-21 and Appendix A 2.1.5.2.4) PF = 1 (no concentration of FPF in proc. grain)	n.a.	
Canola (rape seed), meal	0.22	STMR (primary crop): rapeseed (0.11) x PF (2.0) (cf. Table 7.2-9 and default processing factor for canola, meal)	n.a.	
Corn, milled by-products Corn, hominy meal Corn, gluten feed Corn, gluten, meal	0.010	STMR (rot. crop): maize kernel (0.01) x PF (1.0) (cf. Table 7.2-21 and Appendix A 2.1.5.2.7) PF = 1 (no concentration of FPF in proc. com)	n.a.	
Cotton, meal	0.010	STMR (rot. crop): rapeseed (0.01) x PF (1.0) (cf. Table 7.2-21 and Appendix A 2.1.5.2.2) PF = 1 (no concentration of FPF in proc. cotton)	n.a.	
Distiller´s grain (dried)	0.033	STMR (rot. crop): wheat grain (0.01) x PF (3.3) (cf. Table 7.2-21 and default PF for distiller´s grain)	n.a.	
Linseed, peanut, safflower and sunflower meal	0.020	STMR (rot. crop): rapeseed (0.01) x PF (2.0) (cf. Table 7.2-21 and default PF for linseed, meal)	n.a.	
Lupin seed, meal	0.013	STMR (rot. crop): field pea (0.012) x PF (1.1) (cf. Table 7.2-21 and default PF for lupin seed, meal)	n.a.	
Potato, process waste	0.20	STMR (rot.crop): potato (0.01) x PF (20) (cf. Table 7.2-21 and default processing factor for potato, process waste)	n.a.	
Potato, dried pulp	0.38	STMR (rot.crop): potato (0.01) x PF (38) (cf. Table 7.2-21 and default processing factor for potato, dried pulp)	n.a.	
Rape, meal	0.22	STMR (primary crop): rapeseed (0.11) x PF (2.0) (cf. Table 7.2-9 and Appendix A 2.1.3.1) (default PF for rape, meal)	n.a.	
Soybean, meal	0.013	STMR (rot.crop): rapeseed (0.01) x PF (1.3) (cf. Table 7.2-21 and default PF for soybean, meal)	n.a.	
Soybean, hulls	0.13	STMR (rot.crop): rapeseed (0.01) x PF (13) (cf. Table 7.2-21 and default PF for soybean, hull)	n.a.	

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Wheat gluten, meal Wheat, milled by-products	0.010	STMR (rot. crop): wheat grain (0.01) x PF (1.0) (cf. Table 7.2-21 and Appendices A 2.1.5.2.6) PF = 1 (no concentration of FPF in gluten and middlings)	n.a.	

n.a. = not applicable
STMR = supervised trials median residue HR = highest residue
PF = processing factor

Table 7.2-11: Input values for the dietary burden calculation of DFA (expressed as DFA) (considering the uses authorized in the EU and the use in crops under consideration, as well as the residues in succeeding crops)

Feed commodity	Median dietary burden		Maximum dietary burden	
	(mg/kg)	Comment	(mg/kg)	Comment
Feed items related to previous evaluation(s)				
Apple, wet pomace	0.0072	STMR (primary crop): apple fruit (0.009) x PF (0.8) (NL, 2015)	n.a.	
Feed items related to the MRL application on rotational crops (March 2018) as well as primary crops relevant to this submission				
Forage and silage of grass	0.021	STMR (rot. crop): barley green material (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.045	HR (rot. crop): barley green material (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Straw of cereals and grasses, except maize	0.017	STMR (rot. crop): barley straw (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.048	HR (rot. crop): barley straw (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Forage of legume animal feeds	0.021	STMR (rot. crop): barley green material (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.045	HR (rot. crop): barley green material (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Hay and fodder of legume animal feeds	0.017	STMR (rot. crop): barley straw (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	0.048	HR (rot. crop): barley straw (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)
Cabbage, head Kale, leaves	0.039	STMR (rot. crop): brassica veg. (cf. **Table 7**.2-22 and Appendix A 2.1.6.1.10)	0.065	HR (rot. crop): brassica veg. (cf. **Table 7**.2-22 and Appendix A 2.1.6.1.10)
Fodder beet, mangel	0.014 => 0.021	STMR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. **Table 7**.2-22)	0.037 => 0.056	HR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. **Table 7**.2-22)
Tops of beets and turnips	0.014 => 0.021	STMR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. **Table 7**.2-22)	0.037 => 0.056	HR (rot. crop): lettuce (NL, 2015) => extrapolation to 300 g/ha (cf. **Table 7**.2-22)
Corn (maize), stover	0.011	STMR (rot. crop): maize, stover (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.6 and A 2.1.6.1.7)	0.022	HR (rot. crop): maize, stover (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.6 and A 2.1.6.1.7)
Root and tuber vegetables, culls or roots	0.033	STMR (rot. crop): potato (cf. **Table 7**.2-22 and Appendix A 2.1.6.1.1)	0.088	HR (rot. crop): potato (cf. **Table 7**.2-22 and Appendix A 2.1.6.1.1)

Feed commodity	Median dietary burden		Maximum dietary burden	
	(mg/kg)	Comment	(mg/kg)	Comment
Cereal, grains, except maize	0.068	STMR (rot. crop): barley grain (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.4 and A 2.1.6.1.5)	n.a.	
Maize, kernels	0.028	STMR (rot. crop): maize kernel (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.6 and A 2.1.6.1.7)	n.a.	
Oilseed rape, seeds	0.063	STMR (primary crop): rapeseed (cf. Table 7.2-9 and Appendix A 2.1.3.1)	n.a.	
Oilseeds, other than oilseed rape	0.014	STMR (rot. crop): rapeseed (cf. **Table 7**.2-22 and Appendices A 2.1.6.1.2 and A 2.1.6.1.3)	n.a.	
Pulses, seeds	0.51 => 0.61	STMR (rot. crop): field pea (NL, 2015) => extrapolation to 300 g/ha (cf. **Table 7**.2-22)	n.a.	
Brewer´s grain	0.068	STMR (rot. crop): barley grain (0.068) x PF (1.0) (cf. **Table 7**.2-22 and Appendix A 2.1.5.2.4) PF = 1 (no concentration of DFA in proc. grain)	n.a.	
Canola (rape seed), meal	0.13	STMR (primary crop) rapeseed (0.063) x PF (2.0) (cf. Table 7.2-9 and default PF for canola, meal)	n.a.	
Corn, milled by-products Corn, hominy meal Corn, gluten feed Corn, gluten, meal	0.028	STMR (rot. crop): maize kernel (0.028) x PF (1.0) (cf. **Table 7**.2-22 and Appendix A 2.1.5.2.7) PF = 1 (no DFA residues in overdose experiment)	n.a.	
Cotton, meal	0.018	STMR (rot. crop): rapeseed (0.014) x PF (1.3) (cf. **Table 7**.2-22 and default PF for cotton, meal)	n.a.	
Distiller´s grain (dried)	0.22	STMR (rot. crop): barley grain (0.068) x PF (3.3) (cf. **Table 7**.2-22 and default processing factor for distiller´s grain)	n.a.	
Linseed, peanut, safflower and sunflower meal	0.028	STMR (rot. crop): rapeseed (0.014) x PF (2.0) (cf. **Table 7**.2-22 and default PF for linseed seed, meal)	n.a.	
Lupin seed, meal	0.67	STMR (rot. crop): field pea (0.61) x PF (1.1) (cf. **Table 7**.2-22 and default PF for lupin seed, meal)	n.a.	

Feed commodity	Median dietary burden		Maximum dietary burden	
	(mg/kg)	Comment	(mg/kg)	Comment
Potato, process waste Potato, dried pulp	0.033	STMR (rot. crop): potato (0.033) x PF (1.0) (cf. **Table 7**.2-22 and Appendices 0 and A 2.1.5.2.3) PF = 1 (no concentration of DFA in proc. potato)	n.a.	
Rape, meal	0.13	STMR (primary crop): rapeseed (0.063) x PF (2) (cf. Table 7.2-9 and default PF for rape, meal)	n.a.	
Soybean, meal	0.018	STMR (rot. crop): rapeseed (0.014) x PF (1.3) (cf. **Table 7**.2-22 and default PF for soybean, meal)	n.a.	
Soybean, hulls	0.18	STMR (rot. crop): rapeseed (0.014) x PF (13) (cf. **Table 7**.2-22 and default PF for soybean, hull)	n.a.	
Wheat, gluten Wheat, milled by-products	0.068	STMR (rot. crop): barley grain (0.068) x PF (1.0) (cf. **Table 7**.2-22 and Appendix 0) PF = 1 (no concentration of DFA in gluten and middlings)	n.a.	

n.a. = not applicable
STMR = supervised trials median residue HR = highest residue
PF = processing factor

Results of the animal burden calculations are reported for flupyradifurone in Table 7.2-12 and for DFA in Table 7.2-13 (automatically generated by Excel calculator).

Table 7.2-12: Results of the dietary burden calculation for flupyradifurone

Relevant groups	Median dietary burden (mg/kg bw/d)	Maximum dietary burden (mg/kg bw/d)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)*
Flupyradifurone (FPF, expressed as FPF)					
Cattle (all diets)	0.021	0.023	Potato, process waste	0.79[1]	yes
Cattle (dairy only)	0.021	0.023	Potato, process waste	0.60	yes
Sheep (all diets)	0.024	0.024	Potato, process waste	0.73	yes
Sheep (ewe only)	0.024	0.024	Potato, process waste	0.73	yes
Swine (all diets)	0.009	0.010	Potato, process waste	0.42	yes
Poultry (all diets)	0.007	0.008	Potato, dried pulp	0.11	yes
Poultry (layer only)	0.006	0.006	Potato, dried pulp	0.09	yes

* Trigger exceeded if dietary burden is > 0.004 mg/kg bw

[1] Max. dietary burden for beef cattle: 0.79 mg/kg DM, corresponding to 0.019 mg/kg bw/day

Remarks:

When several diets are relevant (e.g. cattle, sheep and poultry "all diets"), the most critical diet is identified from the maximum dietary burdens expressed as "mg/kg bw per day"

The most critical commodity is the major contributor identified from the maximum dietary burden expressed as "mg/kg bw per day".

Table 7.2-13: Results of the dietary burden calculation for DFA (expressed as DFA)

Relevant groups	Median dietary burden (mg/kg bw/d)	Maximum dietary burden (mg/kg bw/d)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)*
Difluoroacetic acid (DFA, expressed as DFA)					
Cattle (all diets)	0.016	0.022	Swede, roots	0.74[1]	yes
Cattle (dairy only)	0.016	0.022	Swede, roots	0.57	yes
Sheep (all diets)	0.018	0.026	Swede, roots	0.65	yes
Sheep (ewe only)	0.015	0.022	Swede, roots	0.65	yes
Swine (all diets)	0.011	0.018	Swede, roots	0.63	yes
Poultry (all diets)	0.018	0.022	Swede, roots	0.33	yes
Poultry (layer only)	0.018	0.022	Swede, roots	0.33	yes

* Trigger exceeded if dietary burden is > 0.004 mg/kg bw

[1] Max. dietary burden for beef cattle: 0.74 mg/kg DM, corresponding to 0.018 mg/kg bw/day

Remarks:
When several diets are relevant (e.g. cattle, sheep and poultry "all diets"), the most critical diet is identified from the maximum dietary burdens expressed as "mg/kg bw per day"
The most critical commodity is the major contributor identified from the maximum dietary burden expressed as "mg/kg bw per day".

Evaluator comment:
Information given by the Applicant is sufficient.
The median and maximum dietary burdens for livestock were estimated for parent flupyradifurone and separately for its metabolite DFA and were calculated using the animal model calculator developed by EFSA (Animal model 2017).
The calculated dietary burdens for flupyradifurone and for its metabolite DFA were found to exceed the trigger value of 0.1 mg/kg DM (or 0.004 mg/kg bw/d, respectively) for all groups. Further investigation of residues is therefore required.

7.2.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

Available data
Due to the nature of flupyradifurone (BYI 02960), measurable residues have been determined in virtually every crop tested. Many of these crops are relevant as feed items. The major part of the residue in these feed items is formed by parent compound flupyradifurone and its metabolite DFA, in varying ratios. Animal metabolism studies showed that these two components are also the major contributors to the relevant residue in animals.

After consultation of the RMS (Ctgb, NL), BCS decided to feed parent flupyradifurone and derive separate transfer factors for flupyradifurone (by analyzing parent flupyradifurone plus DFA), and for DFA. The estimation of the DFA transfer in poultry and cattle matrices is possible since flupyradifurone shows a fast absorption in livestock and a fast degradation to the terminal metabolite DFA which itself is excreted fast and nearly completely via the urine. Thus the systemic exposure to DFA - and therefore a theoretical minimal DFA dose fed - can be estimated.
For ruminants, the total amount of systemically available DFA (which is equivalent to the theoretical dose of DFA) represented 2.9% of the amount administered as flupyradifurone. Thus dosing the animals with 4.9 mg flupyradifurone/kg bw/day is equivalent to a feeding rate of 0.14 mg DFA (expressed in flupyradifurone equivalents)/kg bw/day. For poultry, the amount of DFA which was systemically available represented 32.1% of the amount administered as flupyradifurone (EFSA, 2015).

This approach was not fully supported in the EFSA Conclusions. However EFSA agreed to derive provisional MRLs for DFA in animal commodities on the basis of this approach until the submission of additional feeding studies conducted with DFA. These temporary MRLs were published in Commission Regulation (EU) 2016/486 in March 2016 and in Commission Regulation (EU) 2016/1902 in October 2016.

In order to fulfil the EFSA requirements, additional feeding studies were conducted with DFA at three different feeding levels in ruminants (cattle) and poultry (laying hens). These new studies have already been submitted to the Netherlands (draft Evaluation Report: MRL application on the setting of MRLs in various rotational crops and animal commodities; March 2018) and are also presented in this submission (Appendix 2) as the data was not yet evaluated at EU level. A summary of the study results (HR, STMR) and the proposed MRL values for DFA (expressed as DFA equivalents) are summarized in

Table 7.2-16.

The transfer of parent residues into animal matrices was already evaluated based on the feeding studies conducted with flupyradifurone (EFSA, 2015). Considering the potential residues arising from all registered flupyradifurone uses and as well from the residues in rotational crops, no flupyradifurone residues above 0.01 mg/kg were expected in milk and organs/tissues of livestock. As a consequence the MRLs for flupyradifurone were established at the default value of 0.01 mg/kg in all animal matrices (Commission Regulation (EU) 2016/486 and Commission Regulation (EU) 2016/1902).
However, when estimating the flupyradifurone MRLs based on the actual dietary burden and using the new Excel calculator "Animal model 2017.xls", the default MRLs of 0.01 mg/kg are exceeded for liver and kidney of cattle and sheep and for kidney of swine.

New MRL proposals (as indicated in red in Table 7.2-14) are as follows:
Cattle and sheep: 0.03 mg/kg for liver and kidney
Swine: 0.015 mg/kg for kidney

These flupyradifurone MRLs exceed the established MRLs, however will be covered by earlier MRL applications (which were triggered by the confirmatory data requested by EFSA and several import tolerances). A draft Evaluation Report with more critical MRL proposals has already been submitted to the EMS (NL) in June 2018, and updated in July 2018.

Table 7.2-14: Overview of the values derived from livestock feeding studies with flupyradifurone (*flupyradifurone expressed as flupyradifurone*)

Animal commodity	Residues at the closest feeding level (mg/kg)		Estimated value at 1N level		MRL proposal (mg/kg)	CF	STMR (mg/kg)	HR (mg/kg)
	Mean	Highest	$STMR_{Mo}$ (mg/kg)	HR_{Mo} (mg/kg)				
Cattle (all diets)								
Closest feeding level[a]:	0.184	mg/kg bw	8.0	N Dairy cattle (highest diet)				
Meat	-	-	-	-	-	-	0.004	0.005
Muscle	0.043	0.048	0.005	0.006	**0.006**	n.c.	0.005	0.006
Fat	0.021	0.028	0.002	0.003	**0.004**	n.c.	0.002	0.003
Liver	0.145	0.172	0.017	0.021	**0.030**	n.c.	0.017	0.021
Kidney	0.159	0.222	0.018	0.028	**0.030**	n.c.	0.018	0.028
Cattle (dairy only)								
Closest feeding level[a]:	0.184	mg/kg bw	8.0	N Dairy cattle				
Milk[b]	0.023	0.026	0.003	0.003	**0.003**	n.c.	0.003	0.003
Sheep (all diets)								
Closest feeding level[a]:	0.184	mg/kg bw	7.6	N Ram/Ewe (highest diet)				
Meat	-	-	-	-	-	-	0.005	0.006
Muscle	0.043	0.048	0.006	0.006	**0.007**	n.c.	0.006	0.006
Fat	0.021	0.028	0.003	0.004	**0.004**	n.c.	0.003	0.004
Liver	0.145	0.172	0.019	0.023	**0.030**	n.c.	0.019	0.023
Kidney	0.159	0.222	0.020	0.029	**0.030**	n.c.	0.020	0.029
Sheep (dairy only)								
Closest feeding level[a]:	0.184	mg/kg bw	7.6	N Ewe				
Milk[b]	0.023	0.026	0.003	0.003	**0.003**	n.c.	0.003	0.003

Animal commodity	Residues at the closest feeding level (mg/kg)		Estimated value at 1N level		MRL proposal (mg/kg)	CF	STMR (mg/kg)	HR (mg/kg)
	Mean	Highest	$STMR_{Mo}$ (mg/kg)	HR_{Mo} (mg/kg)				
Swine								
Closest feeding level[(a)]:	0.184	mg/kg bw	18.9	N Breeding (highest diet)				
Meat	-	-	-	-	-	-	0.002	0.002
Muscle	0.043	0.048	0.002	0.003	**0.003**	n.c.	0.002	0.003
Fat	0.021	0.028	0.001	0.001	**0.002**	n.c.	0.001	0.001
Liver	0.145	0.172	0.007	0.009	**0.009**	n.c.	0.007	0.009
Kidney	0.159	0.222	0.008	0.012	**0.015**	n.c.	0.008	0.012
Poultry (all diets)								
Closest feeding level[(a)]:	0.1	mg/kg bw	13.2	N Broiler (highest diet)				
Meat	-	-	-	-	-	-	0.004	0.004
Muscle	0.004	0.004	0.004	0.004	**0.004**	n.c.	0.004	0.004
Fat	0.003	0.003	0.003	0.003	**0.003**	n.c.	0.003	0.003
Liver	0.003	0.003	0.003	0.003	**0.003**	n.c.	0.003	0.003
Poultry (layer only)								
Closest feeding level[(a)]:	0.1	mg/kg bw	15.8	N Layer				
Eggs[(c)]	0.004	0.004	0.004	0.004	**0.004**	n.c.	0.004	0.004

(a): Closest feeding level and N dose rate related to the maximum dietary burden
(b): Highest residue level from day D1 to day D2 (daily mean of 3 cows).
(c): Highest residue level from day D1 to day D28 (daily mean of 12 laying hens).
Residues for meat were calculated considering a certain fat proportion in the consumed meat. For mammal meat, a ratio muscle/fat of 80/20 is assumed, whereas for poultry meat a ratio muscle/fat of 90/10 is assumed (FAO, 2009).

Remark: For the MRL estimations, three different approaches [transfer factor (1), linear regression (2) and interpolation between the two closest feeding levels (3)] were applied as recommended in the OECD Guidance 73 on residues in livestock. The highest value resulting from the 3 different calculation approaches was chosen as the basis for the MRL estimation. The value derived by linear regression is disregarded when R^2 <0.9. In such a case, the highest value derived by the transfer factor or interpolation approach is selected. If not stated otherwise the transfer factor approach (closest level) was used for estimating the residues.
Residues for meat were calculated considering a certain fat proportion in the consumed meat. For mammal meat, a ratio muscle/fat of 80/20 is assumed, whereas for poultry meat a ratio muscle/fat of 90/10 is assumed (FAO, 2009).

When estimating the residues of DFA in the animal matrices, it has to be considered that the DFA residues can have two sources; either DFA is directly taken up via the feed and can be transferred into the edible matrices, or parent compound flupyradifurone is taken up via the feed and is metabolized to DFA in the animal. Thus the contribution of direct feeding of DFA, as well as the contribution due to degradation of flupyradifurone has to be considered when estimating MRLs for DFA in animal matrices.

However when considering the estimated dietary burdens based on the current EU uses, the contribution of DFA after feeding of flupyradifurone can be neglected (Table 7.2-15). At the estimated 1N level, DFA residues were always below the LOQ. The residues after direct feeding of DFA are summarized in

Table 7.2-16. The DFA residues in

Table 7.2-16 are expressed in DFA equivalents.

When estimating the DFA MRLs based on the actual dietary burden and using the new Excel calculator "Animal model 2017.xls", the temporary MRL for DFA of 0.1 mg/kg in sheep fat as well as in chicken eggs of 0.03 mg/kg was exceeded (MRL proposal of 0.15 mg/kg for sheep fat, and 0.05 mg/kg for eggs). Therefore, these DFA MRLs have to be raised.

New MRL proposals (as indicated in red in

Table 7.2-16) are as follows:
Sheep: 0.15 mg/kg for fat
Poultry: 0.05 mg/kg for egg

These DFA MRLs exceed the currently established temporary MRLs, however will be covered by the MRLs proposed in the draft Evaluation Reports submitted in March 2018 (confirmatory data) and in June 2018 (import tolerances).

For the dietary risk assessment the sum of flupyradifurone and DFA residues has to be considered. As a consequence, the estimated residues for flupyradifurone and DFA detected after feeding of flupyradifurone were added to the DFA residues detected after feeding of DFA, only Table 7.2-17. The total residue (expressed as flupyradifurone equivalents) is shown in Table 7.2-18.

Table 7.2-15: Overview of the values derived from livestock feeding studies with flupyradifurone *(DFA expressed as flupyradifurone)*

Animal commodity	Residues at the closest feeding level (mg/kg)		Estimated value at 1N level		MRL proposal (mg/kg)	CF	STMR (mg/kg)	HR (mg/kg)
	Mean	Highest	$STMR_{Mo}$ (mg/kg)	HR_{Mo} (mg/kg)				
Cattle (all diets)								
Closest feeding level[a]:	0.184	mg/kg bw	8.0	N Dairy cattle (highest diet)				
Meat	-	-	-	-	-	-	0.013*	0.013*
Muscle	0.012*	0.014*	0.013*	0.013*	-	n.c.	0.013*	0.013*
Fat	0.010*	0.013*	0.012*	0.012*	-	n.c.	0.012*	0.012*
Liver	0.012*	0.014*	0.014*	0.014*	-	n.c.	0.014*	0.014*
Kidney	0.017*	0.022	0.002*	0.003*	-	n.c.	0.002*	0.003*
Cattle (dairy only)								
Closest feeding level[a]:	0.184	mg/kg bw	8.0	N Dairy cattle				
Milk[b]	0.004*	0.005*	0.004*	0.004*	-	n.c.	0.004*	0.004*
Sheep (all diets)								
Closest feeding level[a]:	0.184	mg/kg bw	7.6	N Ram/Ewe (highest diet)				
Meat	-	-	-	-	-	-	0.013*	0.013*
Muscle	0.012*	0.014*	0.013*	0.013*	-	n.c.	0.013*	0.013*
Fat	0.010*	0.013*	0.012*	0.012*	-	n.c.	0.012*	0.012*
Liver	0.012*	0.014*	0.014*	0.014*	-	n.c.	0.014*	0.014*
Kidney	0.017*	0.022	0.002*	0.003*	-	n.c.	0.002*	0.003*
Sheep (dairy only)								
Closest feeding level[a]:	0.184	mg/kg bw	7.6	N Ewe				
Milk[b]	0.004*	0.005*	0.004*	0.004*	-	n.c.	0.004*	0.004*
Swine								
Closest feeding level[a]:	0.184	mg/kg bw	18.9	N Breeding (highest diet)				
Meat	-	-	-	-	-	-	0.013*	0.013*
Muscle	0.012*	0.014*	0.013*	0.013*	-	n.c.	0.013*	0.013*
Fat	0.010*	0.013*	0.012*	0.012*	-	n.c.	0.012*	0.012*
Liver	0.012*	0.014*	0.014*	0.014*	-	n.c.	0.014*	0.014*
Kidney	0.017*	0.022	0.001*	0.001*	-	n.c.	0.001*	0.001*
Poultry (all diets)								

Animal commodity	Residues at the closest feeding level (mg/kg)		Estimated value at 1N level		MRL proposal (mg/kg)	CF	STMR (mg/kg)	HR (mg/kg)
	Mean	Highest	$STMR_{Mo}$ (mg/kg)	HR_{Mo} (mg/kg)				
Closest feeding level[(a)]:	0.1	mg/kg bw	13.2	N Broiler (highest diet)				
Meat	-	-	-	-	-	-	0.005*	0.006*
Muscle	0.083	0.096	0.006*	0.007*	-	n.c.	0.006*	0.007*
Fat	0.029	0.035	0.002*	0.003*	-	n.c.	0.002*	0.003*
Liver	0.104	0.112	0.008*	0.009*	-	n.c.	0.008*	0.009*
Poultry (layer only)								
Closest feeding level[(a)]:	0.1	mg/kg bw	15.8	N Layer				
Eggs[(c)]	0.047	0.051	0.003*	0.004*	-	n.c.	0.003*	0.004*

Please note:
Data marked with * are below the LOQ of 0.02 mg/kg (expressed as flupyradifurone equivalents). Detailed residue values below the LOQ were taken as input values to get exact values to be used for the calculation of the total residues of flupyradifurone relevant for consumer risk assessment.

Table 7.2-16: Overview of the values derived from livestock feeding studies with DFA *(DFA expressed as DFA)*

Animal commodity	Residues at the closet feeding level (mg/kg)		Estimated value at 1N level		MRL proposal (mg/kg)	CF	STMR (mg/kg)	HR (mg/kg)
	Mean	Highest	$STMR_{Mo}$ (mg/kg)	HR_{Mo} (mg/kg)				
Cattle (all diets)								
Closest feeding level[a]:	0.032	mg/kg bw	1.5	N Dairy cattle (highest diet)				
Meat	-	-	-	-	-	-	0.035	0.067
Muscle	0.070	0.090	0.035	0.061	**0.060**	n.c.	0.035	0.061
Fat	0.074	0.131	0.037	0.089	**0.090**	n.c.	0.037	0.089
Liver	0.053	0.065	0.027	0.044	**0.050**	n.c.	0.027	0.044
Kidney	0.106	0.119	0.053	0.081	**0.090**	n.c.	0.053	0.081
Cattle (dairy only)								
Closest feeding level[a]:	0.032	mg/kg bw	1.5	N Dairy cattle				
Milk[b]	0.015	0.020	0.007	0.010	**0.010**	n.c.	0.007	0.010
Sheep (all diets)								
Closest feeding level[a]:	0.032	mg/kg bw	1.2	N Lamb (highest diet)				
Meat	-	-	-	-	-	-	0.039	0.085
Muscle	0.070	0.090	0.039	0.073	**0.080**	n.c.	0.039	0.073
Fat	0.074	0.131	0.041	0.130[2]	**0.150**	n.c.	0.041	0.130[2]
Liver	0.053	0.065	0.030	0.053	**0.060**	n.c.	0.030	0.053
Kidney	0.106	0.119	0.059	0.098	**0.100**	n.c.	0.059	0.098
Sheep (dairy only)								
Closest feeding level[a]:	0.032	mg/kg bw	1.5	N Ewe				
Milk[b]	0.015	0.020	0.007	0.010	**0.010**	n.c.	0.007	0.010
Swine								
Closest feeding level[a]:	0.032	mg/kg bw	1.8	N Finishing (highest diet)				
Meat	-	-	-	-	-	-	0.024	0.054
Muscle	0.070	0.090	0.023	0.050	**0.050**	n.c.	0.023	0.050
Fat	0.074	0.131	0.025	0.073	**0.080**	n.c.	0.025	0.073
Liver	0.053	0.065	0.018	0.036	**0.040**	n.c.	0.018	0.036
Kidney	0.106	0.119	0.036	0.066	**0.070**	n.c.	0.036	0.066
Poultry (all diets)								

Animal commodity	Residues at the closet feeding level (mg/kg)		Estimated value at 1N level		MRL proposal (mg/kg)	CF	STMR (mg/kg)	HR (mg/kg)
	Mean	Highest	$STMR_{Mo}$ (mg/kg)	HR_{Mo} (mg/kg)				
Closest feeding level[(a)]:	0.018	mg/kg bw	0.8	N Turkey (highest diet)				
Meat	-	-	-	-	-	-	0.025	0.033
Muscle	0.029	0.030	0.029	0.039[(3)]	**0.040**	n.c.	0.029	0.039[(3)]
Fat	0.007	0.009	0.007	0.011	**0.015**	n.c.	0.007	0.011
Liver	0.052	0.055	0.052	0.071[(3)]	**0.080**	n.c.	0.052	0.071[(3)]
Poultry (layer only)								
Closest feeding level[(a)]:	0.018	mg/kg bw	0.8	N Layer				
Eggs[(c)]	0.035	0.040	0.035	0.050	**0.050**	n.c.	0.035	0.050

(a): Closest feeding level and N dose rate related to the maximum dietary burden
(b): Highest residue level from day D1 to day D28 (daily mean of 3 cows).
(c): Highest residue level from day D1 to day D28 (daily mean of 12 laying hens).

Remark: For the MRL estimations, three different approaches [transfer factor (1), linear regression (2) and interpolation between the two closest feeding levels (3)] were applied as recommended in the OECD Guidance 73 on residues in livestock. The highest value resulting from the 3 different calculation approaches was chosen as the basis for the MRL estimation. The value derived by linear regression is disregarded when R^2 <0.9. In such a case, the highest value derived by the transfer factor or interpolation approach is selected. If not stated otherwise the transfer factor approach (closest level) was used for estimating the residues.
Residues for meat were calculated considering a certain fat proportion in the consumed meat. For mammal meat, a ratio muscle/fat of 80/20 is assumed, whereas for poultry meat a ratio muscle/fat of 90/10 is assumed (FAO, 2009).

Table 7.2-17: Overview of the DFA values derived from livestock feeding studies with flupyradifurone and DFA

Animal matrix	Estimated median residue [mg a.s. equiv./kg]				Estimated maximum residue [mg a.s. equiv./kg]			
	feeding of DFA		feeding of a.s.	Sum DFA	feeding of DFA		feeding of a.s.	Sum DFA
	expr. as DFA	expr. as a.s.	expr. as a.s.	expr. as a.s.	expr. as DFA	expr. as a.s.	expr. as a.s.	expr. as a.s.
Ruminant muscle	0.035	0.104	<0.02	**0.124**	0.061	0.183	<0.02	**0.203**
Ruminant fat	0.037	0.111	<0.02	**0.131**	0.089	0.268	<0.02	**0.288**
Ruminant liver	0.027	0.080	<0.02	**0.100**	0.044	0.132	<0.02	**0.152**
Ruminant kidney	0.053	0.159	<0.02	**0.179**	0.081	0.244	<0.02	**0.264**
Ruminant milk	0.007	0.023	<0.02	**0.043**	0.010	0.031	<0.02	**0.051**
Sheep muscle	0.039	0.117	<0.02	**0.137**	0.073	0.221	<0.02	**0.241**
Sheep fat	0.041	0.125	<0.02	**0.145**	0.130	0.391	<0.02	**0.411**
Sheep liver	0.030	0.090	<0.02	**0.110**	0.053	0.160	<0.02	**0.180**
Sheep kidney	0.059	0.178	<0.02	**0.198**	0.098	0.294	<0.02	**0.314**
Sheep milk	0.007	0.022	<0.02	**0.042**	0.010	0.031	<0.02	**0.051**
Swine muscle	0.023	0.070	<0.02	**0.090**	0.050	0.149	<0.02	**0.169**
Swine fat	0.025	0.075	<0.02	**0.095**	0.073	0.218	<0.02	**0.238**
Swine liver	0.018	0.054	<0.02	**0.074**	0.036	0.108	<0.02	**0.128**
Swine kidney	0.036	0.107	<0.02	**0.127**	0.066	0.199	<0.02	**0.219**
Poultry muscle	0.029	0.088	<0.02	**0.108**	0.039	0.117	<0.02	**0.137**
Poultry fat	0.007	0.021	<0.02	**0.041**	0.011	0.032	<0.02	**0.052**
Poultry liver	0.052	0.157	<0.02	**0.177**	0.071	0.215	<0.02	**0.235**
Poultry eggs	0.035	0.105	<0.02	**0.125**	0.050	0.150	<0.02	**0.170**

Since residue definitions for monitoring are available for both components of the residue definition for risk assessment, no CF factors have to be derived. Nevertheless, the sum of parent flupyradifurone and its metabolite DFA (expressed as flupyradifurone) has to be calculated in order to conduct the dietary risk assessments. The individual residue values for the calculation were taken from Table 7.2-14 to

Table 7.2-16.

Table 7.2-18: Sum of flupyradifurone and DFA residues (*expressed as flupyradifurone*) in ruminant and poultry matrices calculated for dietary risk assessment

Animal matrix	Estimated median residue [mg a.s. equiv./kg]				Estimated maximum residue [mg a.s. equiv./kg]			
	feeding of a.s.		feeding of DFA		feeding of a.s.		feeding of DFA	
	a.s.	DFA	DFA	Total residue	a.s.	DFA	DFA	Total residue
Ruminant muscle	<0.01	<0.02	0.104	**0.134**	<0.01	<0.02	0.183	**0.213**
Ruminant fat	<0.01	<0.02	0.111	**0.141**	<0.01	<0.02	0.268	**0.298**
Ruminant liver	0.017	<0.02	0.080	**0.117**	0.021	<0.02	0.132	**0.173**
Ruminant kidney	0.018	<0.02	0.159	**0.197**	0.028	<0.02	0.244	**0.292**
Ruminant milk	<0.01	<0.02	0.023	**0.053**	<0.01	<0.02	0.031	**0.061**
Sheep muscle	<0.01	<0.02	0.117	**0.147**	<0.01	<0.02	0.221	**0.251**
Sheep fat	<0.01	<0.02	0.125	**0.155**	<0.01	<0.02	0.391	**0.421**
Sheep liver	0.019	<0.02	0.090	**0.129**	0.023	<0.02	0.160	**0.203**
Sheep kidney	0.020	<0.02	0.178	**0.218**	0.029	<0.02	0.294	**0.343**
Sheep milk	<0.01	<0.02	0.022	**0.052**	<0.01	<0.02	0.031	**0.061**
Swine muscle	<0.01	<0.02	0.070	**0.100**	<0.01	<0.02	0.149	**0.179**
Swine fat	<0.01	<0.02	0.075	**0.105**	<0.01	<0.02	0.218	**0.248**
Swine liver	<0.01	<0.02	0.054	**0.084**	<0.01	<0.02	0.108	**0.138**
Swine kidney	<0.01	<0.02	0.107	**0.137**	0.012	<0.02	0.199	**0.231**
Poultry muscle	<0.01	<0.02	0.088	**0.118**	<0.01	<0.02	0.117	**0.147**
Poultry fat	<0.01	<0.02	0.021	**0.051**	<0.01	<0.02	0.032	**0.062**
Poultry liver	<0.01	<0.02	0.157	**0.187**	<0.01	<0.02	0.215	**0.245**
Poultry eggs	<0.01	<0.02	0.105	**0.135**	<0.01	<0.02	0.150	**0.180**

Conclusion on feeding studies

The requested uses in potential feed items relevant to this submission, as well as the new field rotational crop studies and the new mode of calculation modified the theoretical maximum daily intake for animals compared to the intake estimated in the EFSA Conclusion on the peer review (2015). As a consequence there is a clear risk for some animal MRLs to be exceeded.

Draft Evaluation Reports (dERs) have been submitted in March and June 2018 in order to present the confirmatory data requested in Commission Regulation (EU) 2016/1902 and to adopt the US EPA MRLs for flupyradifurone and to apply for DFA MRLs in animal matrices to allow for free trade.

The dER submitted to present the confirmatory data (March 2018) proposes MRLs in livestock which are in agreement with the MRLs of this submission, whereas the proposed MRLs in the dER for the import tolerances are even higher for ruminants and will supersede the EU MRLs.

Evaluator comment:

Information given by the Applicant is sufficient.

Animal metabolism studies showed that flupyradifurone (BYI 02960) its metabolite DFA are the major contributors to the relevant residue in animals.

The livestock exposure was calculated separately for flupyradifurone and the DFA according to the OECD methodology using the EFSA Animal model 2017. The results indicate that the trigger value of 0.004 mg/kg body weight (bw) per day is exceeded for all livestock species, both for fupyradifurone and DFA for relevant livestock diets. Considering the results of livestock dietary burden calculations, the nature and magnitude of flupyradifurone and DFA in animal matrices were further investigated.

The nature of flupyradifurone residues in livestock was investigated in the framework of the EU pesticides peer review (EFSA, 2015).

In the Annex II dossier, feeding studies conducted with the parent flupyradifurone have been provided. These feeding studies were evaluated by EFSA and considered to be acceptable (EFSA, 2015). Additional feeding studies were conducted with DFA at three different feeding levels in poultry (laying hens) and ruminants (cattle). These studies were submitted to Ctgb-NL as confirmatory data (March 2018) and have already been evaluated.

EFSA concluded that the existing EU MRLs for flupyradifurone and DFA should be raised in all animal matrices, except for flupyradifurone in poultry commodities (EFSA Journal 2020;18(6):6133). The data gap identified by the peer review for the livestock feeding studies with DFA was sufficiently addressed.

More details are presented in Appendix 2, point A 2.1.4.1.

In the meantime new MRLs were set for animal commodities for flupyradifurone and DFA in Commission Regulation 2021/1842 of 20 October 2021. In our opinion the residues in animal commodities will not exceed MRLs (Reg. (EU) 2021/1842).
Additionally, in EFSA Journal 2020;18(11):6298, EFSA concluded that "*Since the contribution of flupyradifurone and DFA residues in rapeseed meal to the total EU livestock dietary burden is insignificant, the magnitude of residues in animal commodities was not further assessed.*
EFSA concludes that the residues in rapeseeds from the intended uses will not affect the residues in animal commodities as estimated in the previous EFSA assessment."
No further data are required.

7.2.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation) (KCA 6.5.2-6.5.3)

Numerous studies investigating the effect of processing on the magnitude of flupyradifurone-related residues have been conducted to support the use of flupyradifurone in apple, grape, tomato cucumber and hops. Representative processing studies were conducted with field samples collected from supervised residue trials conducted in Europe, USA or Canada. In an extensive package of processing data, separate Processing Factors (PF) were elucidated for parent flupyradifurone, metabolite DFA and the total residue (sum of flupyradifurone and DFA). These data on processing studies were already reviewed by the RMS and summarized in the final addendum to the DAR (NL, 2015) and the EFSA Conclusion on the peer review (EFSA Journal 2015;13(2):4020).

Excerpt from EFSA Conclusion on the peer review, 2015:
Flupyradifurone was seen to be stable under standard hydrolysis conditions. Processing studies were submitted and processing and conversion factors were proposed for flupyradifurone and DFA respectively, for lettuce, hops, and for the crops included in MRL application (apple, grape, tomato and cucumber).

7.2.5.1 Available data for all crops under consideration

New processing studies are available for the oilseeds soybean and cotton as well as for barley, wheat, corn/maize, and potato. As some of the processed commodities are potential feed items they are of interest when estimating the dietary burden of livestock.

Soybean: Two supervised residue trials were conducted to measure the magnitude of flupyradifurone residues in/on soybean aspirated grain fractions, meal, hulls, refined oil, soymilk, and defatted flour.
The average flupyradifurone residue did not concentrate (processing factor <1X) in soybean milk and oil. Residues of parent remained at approx. the same level in meal, hulls and defatted flour, whereas the flupyradifurone residue did clearly concentrate in aspirated grain fractions (14X).
A similar picture was observed for the metabolite DFA: The average DFA residue did not concentrate (mean processing factor <1X) in soybean milk and oil. Residues of DFA remained also at approx. the same level in hulls. A slight concentration of DFA residues was indicated in meal and defatted flour (mean processing factors of 1.3 and 1.4), whereas the DFA residue again concentrated in aspirated grain fractions (6.5X).

Cotton: Two supervised residue trials were conducted in the US to determine the magnitude of the relevant residues of flupyradifurone in/on processed commodities of cotton, namely oil (refined, solvent extracted, pre-clarified or neutralized), meal and hulls.
Residues of DFA at or above 0.05 mg/kg were neither detected in undelinted cotton seeds (RAC) nor in all different kinds of cotton oil or in hulls. Only the meal samples showed residues of DFA up to 0.065 mg/kg. Hence a slight concentration of DFA residues can be expected for cotton meal during processing. As the raw agricultural commodity did not show any residues, no precise processing factor can be derived and therefore the default processing factor should be applied when estimating the dietary burden of livestock. The flupyradifurone residues did not concentrate (processing factor ≤1X) in all processed commodities including cotton meal.

Potato: Two supervised residue trials were conducted in northern Europe (Germany and the Netherlands) and potatoes were processed into tuber, washed; peel washed; tuber, peeled; tuber, cooked; tuber with peel, cooked; fibre; pulp, dry; starch; waste; waste, dried; flakes; and crisps. Processing factors could only be calculated for DFA as the parent residues were always <0.01 mg/kg in the raw agricultural commodity and as well in the processed products. Concentration of DFA residues was only seen in flakes and crisps. The processing factors for DFA in flakes were <0.8 and 1.7 and in crisps 1.8 and >3.3, respectively. All other processing steps resulted in a dilution of DFA residues.
These results are in line with the results of two supervised residue trials conducted in the USA to determine the magnitude of the relevant residues of flupyradifurone in/on potato and its processed

fractions (crisps, flakes, starch, cooked tuber with peel). Despite the exaggerated application rate, no residues above the LOQ of flupyradifurone and its metabolite DFA were detected in the raw agricultural commodity. A concentration of flupyradifurone residues in the processed commodities was not obvious, whereas a concentration of DFA residues was indicated for flakes and crisps in one trial. The processing factor for DFA in flakes and crisps was >1.9 and >1.5, respectively. All other processing steps resulted in no concentration of flupyradifurone-derived residues.

Barley: Two supervised residue trials were conducted in northern Europe (Germany) to investigate the effect of beer processing on the magnitude of flupyradifurone-related residues in spring barley (malt sprouts; brewer's malt; brewer's grain, hops draff; brewer's yeast; beer; pearl barley and pearl barley rub off).
Concentration of flupyradifurone and DFA residues (and therefore also of the total residue) was only seen in the non-edible commodity pearl barley rub off. The median processing factor for pearl barley rub off was 2.9 for flupyradifurone and the total residue, and 1.8 for DFA. A concentration of DFA residues was also detected in malt sprouts (mean PF = 12), whereas no concentration was detected for the parent flupyradifurone (median PF = 0.40). As a consequence, also no concentration was detected for the total residue. All other processing steps resulted in a dilution of flupyradifurone and DFA residues. Hence, also no concentration of the flupyradifurone and DFA residues is expected in the feed item brewer´s grain.

Wheat: Two supervised residue trials were conducted in northern Europe (Germany) to determine the magnitude of the relevant residues of flupyradifurone (BYI 02960) in/on wheat and its processed fractions (semolina; semolina bran; white flour bran; white flour; white bread; whole meal; whole meal bread and wheat germ).
Flupyradifurone residues concentrate significantly in the bran fractions of semolina and white flour. A transfer of the flupyradifurone residues in semolina or white flour was not detected. Also for DFA a significant concentration was seen in the bran fractions (semolina and white flower), however also a slight transfer of DFA was observed in semolina, white flower, whole meal and wheat germ.
Overall, the total residue of flupyradifurone (sum of flupyradifurone and DFA) did only concentrate in the bran fractions (median processing factor of 5.2 and 6.6 for semolina bran and white flour bran, respectively), in whole meal (median processing factor of 1.5) and in wheat germ (median processing factor of 1.1).
In addition, two field trials were conducted in the USA to measure the magnitude of flupyradifurone residues in/on wheat aspirated grain fractions and the processed commodities of bran, whole meal flour, white flour, middlings, shorts, germ, pasta (dried and cooked), white bread, whole meal bread, gluten, starch, pasta (cooked), pasta (fresh), pasta (dry), and cooking water.
Flupyradifurone and DFA residues concentrate significantly in the aspirated grain fraction. A transfer of residues was also detected in bran. A slight concentration of flupyradifurone was detected in germ and whole meal. The DFA residues in these processed commodities remained at approx. the same level as in the raw agricultural commodity.
Overall, flupyradifurone related residues did not concentrate in most of the processed commodities – especially in commodities for human consumption (like bread and pasta) dilution of residues was observed. Also in the feed items middlings (milled by-products) and gluten, no concentration of residues was detected. The results of the US processing studies are in very good agreement with the results of the EU processing studies and the processing factors of corresponding commodities can be combined.

Maize/corn: Two field trials were conducted in the USA to measure the magnitude of flupyradifurone residues in/on field corn aspirated grain fractions and the processed commodities starch, refined oil, steep water, and germ from wet milling and grits, flour, meal, refined oil, bran and germ from dry milling.
Residues of DFA at or above 0.05 mg/kg were neither detected in field corn grain (RAC) nor in any of the processed commodities – thus no concentration of DFA residues is expected during processing. The flupyradifurone residues did also not concentrate (processing factor $\leq$1X) in the processed commodities of flour, germ (dry or wet milled), grits, meal (dry milled), oil (dry or wet milled), starch and steep water. Therefore no concentration of flupyradifurone or DFA residues is expected in any potential feed item. However, the residues did concentrate significantly in the aspirated grain fraction (median

processing factor of 23), and slightly in bran (median processing factor of 2.8). Overall, the total residue of flupyradifurone (sum of flupyradifurone and DFA) did only concentrate in the aspirated grain fractions (median processing factor of 6.6) and in corn bran (median processing factor of 1.5).

As proposed in the EFSA Conclusion (EFSA Journal 2015;13(2):4020), separate processing factors for flupyradifurone, DFA and as well for the total residue (sum of flupyradifurone and DFA) were derived. The processing factors are summarized in the table below. The detailed results of the studies which were not already evaluated by EFSA are presented in Appendix 2. Please keep in mind that the studies were already submitted in the dER for import tolerances (updated version of July 2018), a dER which will support a label extension of the SL 200 formulation (version July 2018), the dER which will support the use of Sivanto Enery in oilseed rape (version March 2019) and in a dRR for Flupyradifurone AL 0.08 ("home and garden use"). All these dossiers are currently under evaluation.

For the animal burden calculations, a processing factor of 1 was applied when processing resulted in a dilution of residues. If no processing factor was available (e.g. for rape meal), the default processing factors (proposed by EFSA) were considered.

Table 7.2-19: Overview of the available processing studies

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
EU data					
Enforcement residue definition 1: Flupyradifurone (FPF) Enforcement residue definition 2: Difluoroacetic acid (DFA) Risk assesment residue definition: **Sum of flupyradifurone and DFA (Sum)**					
Apple/Wet pomace	4 EU	FPF: *1.5* DFA: 0.8 Sum: 1.4	--	F: *1.3*, 1.4, 1.5, 1.7 D: *0.8*; 3 x n.c. T: *1.1; 1.3; 1.4; 1.6*	Schulte, G.; Bauer, J.; 2012; M-434004-02-1 10-3171 and Schulte, G.; Ballmann, C.; 2012; M-434006-01-2 10-3172 EFSA Journal 2015;13(2): 4020
Apple/Dry pomace	4 EU	FPF: 4.9 DFA: 2.3 Sum: 4.0	--	F: 4.6, 4.9, *4.9*, 5.0, D: *>1.0; >1.5; 2.3*; n.c. T: 3.8; 3.9; 4.0; 4.1	
Apple/Juice	4 EU	FPF: 0.5 DFA: 0.8 Sum: 0.6	--	F: 0.3, 0.4, *0.6*, 0.7, D: *0.8*; 3 x n.c. T: 0.5; 0.6; 0.6; 0.7	
Apple/Sauce	4 EU	FPF: 0.8 DFA: 0.8 Sum: 0.8	--	F: 0.7, *0.7*, 0.8, *0.8*, D: *0.8;* 3 x n.c. T: 0.3; 0.8; 0.8; 0.8	
Apple/Dry peeled fruit	1 EU	FPF: 2.0 DFA: >1.5 Sum: 1.9	--	F: 2.0 D: *>1.5* T: 1.9	
Grape/Red wine	2 EU	FPF: *0.8* DFA: 1.0 Sum: 0.8	--	F: 0.7, *0.8* D: 1.0; n.c. T: 0.7; 0.8	Noss, G.; Bauer, J.; Ruhl, S.; 2012; M-433545-01-1 10-3406 EFSA Journal 2015;13(2): 4020
Grape/White wine	2 EU	FPF: *0.4* DFA: 1.0 Sum: 0.4	--	F: 0.3, 0.4 D: 1.0, n.c. T: 0.3; 0.4	
Grape/Juice (pasteurised)	2 EU	FPF: 0.7 DFA: 1.2 Sum: 0.7	--	F: 0.5, 0.9 D: 1.0, *1.3* T : 0.5; 0.9	
Grape/Jelly	2 EU	FPF: 0.3 DFA: 0.7 Sum: 0.3	--	F: 0.2, 0.4 D: 0.7, 0.7 T: 0.2; 0.4	
Grape/Raisin	2 EU	FPF: 2.5 DFA: 2.3 Sum: 2.5	--	F: 2.1, 2.9 D: *>1.5*; 2.3 T: 2.1; 2.8	

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
Lettuce/outer leaves	4 EU	FPF: 1.8 DFA: n.c. Sum: 1.8	--	F: 1.6, 1.8, 1.8, 2.2 D: 4 x n.c. T: 1.6, 1.8, 1.8, 2.2	Schulte, G.; Bauer, J.; 2012; M-426982-01-1 10-3223 EFSA Journal 2015;13(2): 4020
Lettuce/inner head parts	4 EU	FPF: 0.7 DFA: n.c. Sum: 0.7	--	F: 0.6, 0.7, 0.7, 0.8 D: 4 x n.c. T: 0.6, 0.7, 0.7, 0.8	
Lettuce/inner leaves	4 EU	FPF: 0.9 DFA: n.c. Sum: 0.9	--	F: 0.1, 0.8, 1.0, 1.2 D: 4 x n.c. T: 0.1; 0.8; 1.0; 1.2	
Lettuce/washed inner leaves	4 EU	FPF: *0.7* DFA: n.c. Sum: 0.7	--	F: 0.5, 0.6, 0.7, 0.8, D: 4 x n.c. T: 0.5, 0.6, 0.7, 0.8	
New data					
Enforcement residue definition 1: Flupyradifurone (FPF) Enforcement residue definition 2: Difluoroacetic acid (DFA) Risk assesment residue definition: **Sum of flupyradifurone and DFA (Sum)**					
Soybean, aspirated grain fractions	2 US	FPF: 14 DFA: 6.5 Sum: 12	-	F: 16; 12 D: 8.2; 4.8 S: 15; 8.8	Lenz, C.; 2012; M-428939-01-2 RARVY029 cf. A 2.1.5.2.1
Soybean, meal		FPF: 1.1 DFA: 1.3 Sum: 1.1	-	F: 0.9; 1.3 D: 1.2; 1.4 S: 0.9; 1.3	
Soybean, hulls		FPF: 1.0 DFA: 0.9 Sum: 0.9	-	F: 0.8; 1.2 D: 1.0; 0.78 S: 0.8; 1.0	
Soybean, refined oil		FPF: <0.019 DFA: <0.6 Sum: <0.1	-	F: <0.014; <0.010 D: <1.0; <0.16 S: <0.08; <0.08	
Soybean, milk		FPF: 0.066 DFA: <0.6 Sum: 0.1	-	F: 0.042; 0.089 D: <1.0; <0.16 S: 0.11; 0.12	
Soybean, defatted flour		FPF: 1.1 DFA: 1.4 Sum: 1.2	-	F: 1.0; 1.2 D: 1.5; 1.3 S: 1.1; 1.2	
Cotton, oil refined	2 US	FPF: <0.1 DFA: n.c. Sum: <0.12	-	F: <0.1; <0.1 D: n.c.; n.c. S: <0.04; <0.20	Lenz, C.; Beedle, E.; 2012; M-433122-01-1 RARVY033 cf. A 2.1.5.2.2
Cotton, oil, solvent extracted	2 US	FPF: <0.1 DFA: n.c. Sum: <0.12	-	F: <0.1; <0.1 D: n.c.; n.c. S: <0.04; <0.20	
Cotton, crude oil, preclarified	2 US	FPF: <0.1 DFA: n.c. Sum: <0.12	-	F: <0.1; <0.1 D: n.c.; n.c. S: <0.04; <0.20	
Cotton, crude oil, neutralized	2 US	FPF: <0.1 DFA: n.c. Sum: <0.12	-	F: <0.1; <0.1 D: n.c.; n.c. S: <0.04; <0.20	
Cotton, meal	2 US	FPF: <0.3 DFA: >1.3 Sum: 0.25	-	F: <0.1; 0.3 D: n.c.; >1.3 S: 0.07; 0.43	
Cotton, hull	2 US	FPF: <0.5 DFA: n.c. Sum: 0.53	-	F: <0.1; 0.9 D: n.c.; >n.c. S: 0.13; 0.93	
Potato, flakes	2 US 2 EU	FPF: 1.1 DFA: 1.7	-	F: n.c.; 1.1.; n.c.; n.c. D: n.c.; 0.8; 1.7; 2.0	Lenz, C.; 2012; M-430542-01-2

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
		Sum: 1.3		S: n.c.; 0.8; 1.3; 2.2	RARVY038
Potato, crisps	2 US 2 EU	FPF: n.c. DFA: 1.8 Sum: 1.4	-	F: n.c.; n.c.; n.c.; n.c. D: n.c.; 1.5; 1.8; 3.3. S: n.c.; 1.3; 1.4; 2.7	cf. 0
Potato, starch	2 US 2 EU	FPF: n.c. DFA: <0.8 Sum: <0.8	-	F: n.c.; n.c.; n.c.; n.c. D: n.c.; n.c.; <0.8; <0.8 S: n.c.; n.c.; <0.8; <0.8	Freitag, T.; Hoffmeister, R.; 2016; M-546878-01-1 13-3149
Potato, washed tuber	2 US 2 EU	FPF: ≥1 DFA: 1.1 Sum: 1.2	-	F: n.c.; n.c.; ≥1.; ≥1 D: n.c.; n.c.; 0.9; 1.2 S: ≥1.; ≥1.; 1.0; 1.3	cf. A 2.1.5.2.3
Potato, peeled tuber	2 US 2 EU	FPF: n.c. DFA: 1.0 Sum: 1.0	-	F: n.c.; n.c.; n.c.; n.c. D: n.c.; ≥1; 1.0; 1.0 S: n.c.; ≥1; 1.0; 1.0	
Potato, cooked tuber	2 US 2 EU	FPF: n.c. DFA: <1.0 Sum: <1.0	-	F: n.c.; n.c.; n.c.; n.c. D: n.c; n.c.; <0.9; 1.0 S: n.c.; n.c.; <1.0; 1.0	
Malt sprouts	2 EU	FPF: 0.4 DFA: 12 Sum: 0.77	-	F: 0.38; 0.41 D: 12; 12 S: 0.92; 0.63	Schulte, G.; Ruhl, S.; 2012; M-439853-01-1 10-3410
Brewer´s malt	2 EU	FPF: 0.48 DFA: 0.83 Sum: 0.49	-	F: 0.44; 0.52 D: 1.0; 0.67 S: 0.47; 0.52	cf. A 2.1.5.2.4
Brewer´s grain	2 EU	FPF: 0.05 DFA: <0.70 Sum: 0.07	-	F: 0.08; 0.03 D: <0.5; <0.67 S: 0.10; 0.04	
Hops draff	2 EU	FPF: 0.19 DFA: - Sum: 0.35	-	F: 0.19; 0.18 D: <5.0, <6.7 S: 0.41; 0.30	
Brewer´s yeast	2 EU	FPF: 0.09 DFA: <0.70 Sum: 0.10	-	F: 0.07; 0.11 D: <0.5; <0.67 S: 0.09; 0.06	
Beer	2 EU	FPF: 0.06 DFA: <0.70 Sum: 0.08	-	F: 0.07; 0.05 D: <0.5; < 0.67 S: 0.09; 0.06	
Pearl barley rub off	2 EU	FPF: 2.9 DFA: 1.8 Sum: 2.9	-	F: 3.3; 2.5 D: 2.0; 1.7 S: 3.3; 2.5	
Pearl barley	2 EU	FPF: 0.10 DFA: 0.71 Sum: 0.12	-	F: 0.14; 0.06 D: 0.75; 0.67 S: 0.17; 0.07	
Semolina bran	2 EU	FPF: 4.6 DFA: 25 Sum: 5.2	-	F: 4.8; 4.4 D: 25; >1 S: 6.4; 3.9	Schulte, G.; Bauer, J.; 2012; M-440307-01-1 10-3409
Semolina	2 EU	FPF: 0.9 DFA: 1.5 Sum: 1.0	-	F: 1.4; 0.4 D: 1.5; n.c. S: 1.4; 0.5	cf. A 2.1.5.2.5
White-flour bran	2 EU	FPF: 6.2 DFA: 26 Sum: 6.6	-	F: 6.5; 5.8 D: 26; >1.5 S: 8.0; 5.3	
White flour	2 EU	FPF: 0.2 DFA: 1.5 Sum: 0.3	-	F: 0.3; 0.1 D: 1.5; n.c. S: 0.4; 0.3	
White bread	2 EU	FPF: 0.2 DFA: 1.0	-	F: 0.2, 0.1 D: 1.0, n.c.	

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
		Sum: 0.3		S: 0.3; 0.3	
Whole meal	2 EU	FPF: 1.5 DFA: 1.5 Sum: 1.5	-	F: 1.6; 1.5 D: 1.5; n.c. S: 1.6; 1.4	
Whole-meal bread	2 EU	FPF: 0.9 DFA: 1.0 Sum: 0.9	-	F: 1.0; 0.9 D: 1.0; n.c. S: 1.0: 0.9	
Wheat germ	2 EU	FPF: 1.0 DFA: 1.5 Sum: 1.1	-	F: 1.3; 0.8 D: 1.5; n.c. S: 1.3; 0.8	
Wheat, aspirated grain fractions	2 US	FPF: 17.6 DFA: 6.9 Sum: 10.5	-	F: 21.5; 13.8 D: 5.0; 8.8 S: 10.0; 10.9	Lenz, C.; Fischer, D. R.; 2012; M-427047-01-2 RARVY031 cf. A 2.1.5.2.6
Wheat, bran	2 US	FPF: 2.4 DFA: 1.1 Sum: 1.5	-	F: 2.3; 2.4 D: 1.1; 1.0 S: 1.5; 1.6	
Wheat, cooking water	2 US	FPF: <0.1 DFA: <0.1 Sum: <0.1	-	F: <0.1; <0.1 D: <0.1; <0.1 S: <0.1; <0.1	
Wheat, cooking water 2	2 US	FPF: <0.1 DFA: <0.1 Sum: <0.1	-	F: <0.1; <0.1 D: <0.1; <0.1 S: <0.1; <0.1	
Wheat, germ	2 US	FPF: 1.6 DFA: 1.0 Sum: 1.2	-	F: 1.7; 1.6 D: 1.0; 1.0 S: 1.2; 1.3	
Wheat, gluten	2 US	FPF: 0.4 DFA: 0.4 Sum: 0.4	-	F: 0.5; 0.2 D: 0.7; 0.2 S: 0.6; 0.2	
Wheat, middlings	2 US	FPF: 0.7 DFA: 0.9 Sum: 0.8	-	F: 0.6; 0.8 D: 0.9; 0.9 S: 0.8; 0.8	
Wheat, pasta, cooked	2 US	FPF: <0.1 DFA: 0.2 Sum: 0.1	-	F: <0.1; <0.1 D: 0.2; 0.2 S: 0.2; 0.1	
Wheat, pasta, dried and cooked	2 US	FPF: <0.1 DFA: 0.2 Sum: 0.2	-	F: <0.1; <0.1 D: 0.2; 0.2 S: 0.2; 0.2	
Wheat, pasta, dry	2 US	FPF: 0.3 DFA: 0.9 Sum: 0.7	-	F: 0.3; 0.2 D: 0.9; 0.8 S: 0.7; 0.6	
Wheat, pasta, fresh	2 US	FPF: 0.2 DFA: 0.7 Sum: 0.5	-	F: 0.2; 0.2 D: 0.8; 0.7 S: 0.6; 0.5	
Wheat, shorts	2 US	FPF: 0.2 DFA: 0.9 Sum: 0.9	-	F: 0.6; 1.2 D: 0.9; 1.0 S: 0.8; 1.1	
Wheat, starch	2 US	FPF: <0.1 DFA: <0.1 Sum: <0.1	-	F: <0.1; <0.1 D: <0.1; <0.1 S: <0.1; <0.1	
Wheat, white bread	2 US	FPF: 0.2 DFA: 0.6 Sum: 0.4	-	F: 0.2; 0.1 D: 0.7; 0.5 S: 0.5; 0.4	
Wheat, white flour	2 US	FPF: 0.3 DFA: 0.8	-	F: 0.3; 0.2 D: 0.8; 0.7	

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
		Sum: 0.6		S: 0.7; 0.5	
Wheat, whole meal	2 US	FPF: 1.2 DFA: 1.0 Sum: 1.1	-	F: 1.1; 1.3 D: 1.1; 0.9 S: 1.1; 1.1	
Wheat, whole meal bread	2 US	FPF: 0.7 DFA: 0.7 Sum: 0.7	-	F: 0.7; 08 D: 0.7; 0.7 S: 0.7; 0.7	
Maize, aspirated grain fractions	2 US	FPF: 23.1 DFA: n.c. Sum: 6.6	-	F: 25.4, 20.8 D: n.c., n.c. S: 5.8, 7.4	Lenz, C.; 2012; M-424774-01-1 RARVY030 cf. A 2.1.5.2.7
Maize, bran	2 US	FPF: 2.8 DFA: n.c. Sum: 1.5	-	F: 1.8, 3.8 D: n.c., n.c. S: 1.2, 1.9	
Maize, flour	2 US	FPF: <0.6 DFA: n.c. Sum: <0.9	-	F: <0.8, <0.4 D: n.c., n.c. S: <1.0, <0.8	
Maize, germ, dry milling	2 US	FPF: <1.0 DFA: n.c. Sum: <1.0	-	F: <0.8, 1.2 D: n.c., n.c. S: <1.0, 1.1	
Maize, germ, wet milling	2 US	FPF: <0.7 DFA: n.c. Sum: <0.9	-	F: <0.8, 0.5 D: n.c., n.c. S: <1.0, 0.9	
Maize, grits	2 US	FPF: <0.6 DFA: n.c. Sum: <0.9	-	F: <0.8, <0.4 D: n.c., n.c. S: <1.0, <0.8	
Maize, meal, dry milled	2 US	FPF: <0.6 DFA: n.c. Sum: <0.9	-	F: <0.8, <0.4 D: n.c., n.c. S: <1.0, <0.8	
Oil, dry milled	2 US	FPF: <0.6 DFA: n.c. Sum: <0.9	-	F: <0.8, <0.4 D: n.c., n.c. S: <1.0, <0.8	
Maize, oil, wet milled	2 US	FPF: <0.6 DFA: n.c. Sum: <0.9	-	F: <0.8, <0.4 D: n.c., n.c. S: <1.0, <0.8	
Maize, starch	2 US	FPF: <0.6 DFA: n.c. Sum: <0.9	-	F: <0.8, <0.4 D: n.c., n.c. S: <1.0, <0.8	
Maize, steep water	2 US	FPF: <0.7 DFA: n.c. Sum: <0.9	-	F: <0.8, 0.5 D: n.c., n.c. S: <1.0, 0.8	

* The median processing factor is obtained by calculating the median of the individual processing factors of each processing study.

** The median conversion factor for enforcement to risk assessment is obtained by calculating the median of the individual conversion factors of each processing study.

n.c. not calculated, since residues in RAC and processed commodity were <LOQ.

Remark: Typos/ missing values identified in the DAR/EFSA Conclusion were corrected in the table below (marked in *italic* typeface).

7.2.5.2 Conclusion on processing studies

Processing studies were conducted for soybean, cotton, potato, barley, wheat and corn/maize. As some of the processed commodities are potential feed items and therefore of interest when estimating the dietary burden of livestock these studies are detailed within this submission.

The processing study with soybean indicated a slight concentration of flupyradifurone and DFA residues in the processed product meal. As the experimentally determined processing factors for soybean are

equivalent or lower as compared to the default processing factors proposed by EFSA (Animal model 2017a.xls), the default processing factor was used when estimating the dietary burden of livestock.

Since the flupyradifurone related residues in potato tubers were always <0.01 mg/kg when treated according to the EU label, one processing study was conducted after exaggerated treatment (20X).
Processing of potatoes clearly indicated that domestic processing procedures (washing, peeling, cooking) do not change the potential residues. The residue levels of both analytes (parent flupyradifurone and metabolite DFA) remained at approx. the same level. Industrial processing of potato samples to prepare flakes and crisps (chips) indicated a concentration of DFA residues, whereas the flupyradifurone residues remained at approx. the same level. In potato starch a slight dilution of DFA residues was detected in the study with the exaggerated rate; flupyradifurone residues were not detected in any study. As a worst-case estimate, the default processing factors was applied for process waste and dried pulp of potato when estimating the dietary burden of livestock.

The processing studies for barley, wheat, corn (maize), potato, and cotton - where processed commodities are potential feed items - indicated no concentration of flupyradifurone and DFA residues in any of the EU feed items (brewer´s grain, milled by-products and gluten of wheat, flour, grits and meal (dry milled) of maize/corn as well as process waste and dried pulp of potatoes), except for DFA in cotton meal where a slight increase of DFA was indicated.
As the raw agricultural commodity (cotton, seed) did not show any DFA residues, the processing factor could only be estimated and therefore the default processing factor was applied when calculating the dietary burden of livestock.

Evaluator comment:
Information given by the Applicant is sufficient.
Numerous studies investigating the effect of processing on the magnitude of flupyradifurone-related residues have been conducted. For crops considered in the EU evaluation (lettuce, hops, apple, grape, tomato and cucumber), EFSA derived separate processing factors (PF) for flupyradifurone, DFA and for the total residue (sum of flupyradifurone and DFA) (EFSA, 2015).
Additional processing studies are available for oilseeds, soybean and cotton as well as for barley, wheat, corn/maize, and potato (which can be grown in rotation with oilseed rape). Processing studies on those crops were already summited and evaluated by the EMS-The Netherlands (Evaluation report on MRLs for flupyradifurone in imported commodities, The Netherlands, February 2019). The intended uses are covered with the available processing studies. More details are presented in Appendix 2, point A 2.1.5.
Soybean
Significant concentration of BYI 02960 and DFA residues and of the total residue was only detected in the aspirated grain fraction. The mean processing factor was 14 for BYI 02960, 6.5 for DFA and 12.0 for the total residue. The average total BYI 02960 residue did not concentrate (processing factor <1X) in all soybean processed commodities. A slight concentration could be assumed in meal and defatted flour, with processing factors of slightly above 1 for BYI 02960, DFA and the total residue. The preparation steps for refined oil and milk result in a clear reduction of residues.
Cotton
BYI 02960 residues did not concentrate in any of the processed products (meal, hulls, refined oil (bleached and deodorized), solvent extracted crude oil, pre-clarified crude oil, and neutralized crude oil). DFA residues were only detected in one meal sample; all other commodities showed DFA residues below the LOQ (0.05 mg/kg), so concentration of DFA residues in meal cannot be excluded.
Taking into account the total residue of BYI 02960 (sum of BYI 02960 and DFA), no concentration of the residue can be determined in any of the processed commodities, the processing factors were always <1.
Potato
1) EU study
Processing factors could only be calculated for DFA as the parent residues were always <0.01 mg/kg in the raw agricultural commodity. A concentration of DFA residues due to processing was only seen in flakes and crisps of one study. The processing factor for DFA in flakes and crisps was 1.7 and 1.8 respectively. All other processing steps resulted in a dilution of DFA residues.
2) US study
A slight concentration of BYI 02960 residues was indicated in potato flakes and peel. Washing did not reduce the residues, neither for BYI 02960 not for DFA. Overall, the total BYI 02960 residue (sum of BYI 02960 and DFA) did not concentrate (processing factor ≤1X) significantly in any of the processed commodities. The highest transfer of the total residue was detected for potato flakes.

Barley
BYI 02960 residues and total residue of BYI 02960 (sum of BYI 02960 and DFA) did not concentrate in any processed product, except for non-edible commodity, for rub-off of pearl barley. The mean processing factor for pearl barley rub off equals 2.9 for BYI 02960 and for sum of BYI 02960 and DFA. For the metabolite DFA, the mean processing factors in rub-off of pearl barley are 1.8. In addition DFA concentrates in malt sprouts, for which the mean processing factor are 12.1. It should be noted that no concentration in malt sprouts was detected for the parent BYI 02960 (mean PF = 0.40), so no concentration was detected for the total residue (mean PF = 0.77).
All other processing steps resulted in a dilution of BYI 02960 and DFA residues.
Wheat
1) EU study
BYI 02960 residues concentrate significantly in the bran fractions of semolina (mean processing factor of 4.6) and white flour (mean processing factor of 6.2). A transfer of the residues in semolina or white flour was not detected. Also for DFA a significant concentration was seen in the bran fractions (mean processing factor for semolina of 25 and for white flower of 26), however also a slight transfer of DFA was observed in semolina, white flower, whole flour and wheat germ. Overall, the total residue of BYI 02960 (sum of BYI 02960 and DFA) did only concentrate in the bran fractions (mean processing factor of 5.2 and 6.6 for semolina bran and white flour bran, respectively), in whole flour (mean processing factor of 1.5) and in wheat germ (mean processing factor of 1.1).
2) US study
The results of the study showed that, BYI 02960 and DFA residues concentrate significantly in the aspirated grain fraction (mean processing factor of 17.6 for BYI 02960 and 6.9 for DFA) and in bran (mean processing factor 2.4 for BYI 02960 and 1.1 for DFA). A slight concentration of BYI 02960 was detected in germ (mean processing factor of 1.6 for BYI 02960 and 1.0 for DFA) and whole meal (mean processing factor of 1.2 for BYI 02960 and 1.0 for DFA).
The average total BYI 02960 (sum of BYI 02960 and DFA) residue did not concentrate in most of the processed commodities (processing factor <1X): in cooking water, cooking water 2, gluten, middlings, pasta (cooked), pasta (dried and cooked), pasta (dry), pasta (fresh), shorts, starch, white bread, white flour, whole meal bread, and whole meal flour. However, the total BYI 02960 residue did concentrate in aspirated grain fractions (10X), bran (1.6X), and germ (1.3X) and slightly in whole meal (1.1X).
Corn
The residues of BYI 02960 did not concentrate in the processed commodities of flour, germ (dry or wet milled), grits, meal (dry milled), oil (dry or wet milled), starch and steep water (processing factor ≤1X). However, the BYI 02960 residues did concentrate significantly in the aspirated grain fraction (mean processing factor of 23), and slightly in bran (mean processing factor of 2.8).
Due to the fact that residues of DFA at or above 0.05 mg/kg were neither detected in field corn grain (RAC) nor in any of the processed commodities, it is no concentration of DFA residues expected during processing.
The average total BYI 02960 (sum of BYI 02960 and DFA) residue did not concentrate (processing factor ≤1X) in silage, and the processed commodities of flour, germ (dry milling), germ (wet milling), grits, meal (dry milled), oil (dry milled), oil (wet milled), starch, and steep water. However, the total BYI 02960 residue did concentrate in aspirated grain fractions (mean processing factor of 6.6) and bran (mean processing factor of 1.5).

No further data are required.

7.2.6 Magnitude of residues in succeeding crops

The crop under consideration can be grown in rotation and therefore the possible occurrence of residues in succeeding crops resulting from the primary use has to be investigated.
Data dealing with the magnitude of residues in succeeding crops have already been submitted, but additional field rotational crop studies were conducted in selected crops (cereals, maize, oilseed rape, potato, brassica vegetables and strawberries) at rates reflecting the expected plateau concentration of flupyradifurone in soil reached following several years of consecutive applications. Realistic plant back intervals were considered and soil samples were collected before each re-cropping interval to determine the flupyradifurone and DFA levels in the relevant soil layer (plough layer, 0-20 cm).

7.2.6.1 Field rotational crop studies (KCA 6.6.2)

Available and new data
Numerous field rotational crop trials have been conducted to support the use of flupyradifurone. Apart from the "main" field study (containing data for three rotations with three crop groups: leafy, root, and cereal crops), further data on individual crops after one rotation ("small" rotational crop studies on tuber, stem, fruiting, bulb, and legume vegetables, as well as on pulses and oilseeds) have been submitted. These

studies have already been reported and evaluated in the final addendum to the DAR (NL, 2015) and peer reviewed by EFSA (EFSA Journal 2015;13(2):4020).
The amount of flupyradifurone, applied either to bare soil or lettuce as target crop, amounted to 200 or 250 g a.s./ha (based on the worst-case application rate in the "Home & Garden" uses, which are more critical than the corresponding agricultural uses).

The design of these field rotational crop studies was discussed at EU level (Pesticides Peer Review expert meeting TC 107). It was questioned if the applied application rate reflects the plateau concentration of flupyradifurone reached in soil following several years of consecutive applications as flupyradifurone is a persistent compound. Moreover, the experts were of the opinion that testing a single plant back interval (PBI) of 30 days (worst-case: crop failure) reflects an unrealistic situation for several crops which can lead to an overestimation of MRLs. It was therefore concluded that further studies – conducted at realistic PBIs and providing information on the flupyradifurone and DFA residue levels in soil – are required. Provisionally and since significant DFA residues are expected in rotational crops, it was agreed to derive temporary MRLs values for rotational crops from the available trials conducted at a dose rate of 200-250 g a.s./ha and considering the plant back interval of 30 days and the highest residue values.

In order to overcome this data gap, an assessment was done to identify the most reasonable plateau concentration of flupyradifurone reached in soil following several years of consecutive applications.
Two scenarios were considered: The multi-year use of flupyradifurone in a "Home & Garden" product and the multi-year use of flupyradifurone by farmers. Both scenarios take into account crop rotation and therefore the application of different use rates depending on the crop treated. The plateau concentrations were estimated using either the worst-case DT_{50} value of the field dissipation studies (worst-case trial site was Great Chishill, UK) or the geometric mean DT_{50} of the six available EU field sites (more probable case).

DFOP model parameters (biphasic degradation) to estimate the plateau level:

	DT_{50} fast (d)	**DT_{50} slow (d)**	**k_1**	**k_2**	**g**
worst case	0.02	462	34.66	0.0015	0.27
geo-mean	2.71	224	0.256	0.0031	0.49

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of flupyradifurone (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate allowed for flupyradifurone in Europe.

Scenario	**Mean plateau level [g a.s./ha]**	**Maximum seasonal rate [g a.s./ha]**	**Estimated application rate [g a.s./ha]**	**Application rate already tested [g a.s./ha]**	**Application rate to be tested [g a.s./ha]**
Agricultural uses					
worst-case	43	150[a)]	193	200 or 250	175
more probable	10		160		
"Home & Garden" uses					
worst-case	118	250[b)]	368	200 or 250	300
more probable	29		279		

a) Intended maximum seasonal rate for cereals (use intended in near future); currently the maximum registered application rate is 125 g a.s./ha in lettuce
b) Maximum registered use rate in lettuce

Based on the estimated application rates, it was agreed with the RMS to apply 175 g a.s./ha when growing cereals (wheat/barley), maize and oilseed rape as succeeding crops (agricultural uses, only). Application scenarios were investigated in which a crop was harvested and the field was used again thereafter, in the same season (60-270 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-planting. A plant-back interval of 30 days (1st rotation) was not considered relevant as such a short plant-back interval is not reasonable for a crop with a rather long vegetation period.
It was also agreed that re-cropping of potatoes, strawberries, and the brassica vegetables broccoli and

cauliflower should done after application of 300 g a.s./ha to bare soil. The higher application rate resulted from the fact that "Home & Garden" uses will be registered for these crops. For these crops (except potato), three plant-back intervals were considered relevant (7-30 days, 60-270 days and 270-365 days).
Detailed background information on the estimated application rates and the degradation behaviour of the parent compound and its metabolite DFA in soil can be found in the working paper which was discussed with the RMS (Spiegel, K.; 2016; M-617654-01-1).
Metabolite difluoroacetic acid (DFA) is only formed from flupyradifurone in soil and its degradation follows a single first order kinetic (SFO) with a DT_{50} <150 days and a DT_{90} <500 days. Thus, metabolite DFA itself shows no accumulation potential and the DFA concentration at the plateau is only dependent on the available flupyradifurone amount. Moreover, soil analysis of all soil cores (0-20 cm) sampled in the additional studies - directly before re-planting of a crop at each plant-back interval - showed only flupyradifurone residues and no DFA residues above an LOQ of 0.005 mg DFA/kg. As a consequence, DFA residues in plant matrices can be directly related to the applied amount of flupyradifurone and thus extrapolation of DFA residues based on the applied flupyradifurone rate can be justified.

An overview on the field rotational crop studies already submitted in the EU as well as additional field rotational crop studies is provided in Table 7.2-20. A brief summary of the residue values in the field rotational crop studies is provided in Table 7.2-21.
Unrealistic re-cropping scenarios which do not have to be considered – however were already submitted – are shown with grey background. A summary of the DFA residues in rotational crops (with STMR and HR values) is shown in Table 7.2-22. A consolidated summary comparing the anticipated highest residues in field rotational crop studies with the current MRLs is given in Table 7.2-23 (flupyradifurone) and in Table 7.2-24 (DFA).

In order to comply with the EFSA evaluation (EFSA 2015), the residue levels at the main harvest were considered for root and leafy crops and as well for brassica vegetables which were collected at two sampling points (e.g. for potatoes collected at BBCH 45 and 49, only the residues at BBCH 49 were considered). For strawberries, where an early harvest is very common, the worst-case residue from the two different sampling times was considered for further estimations.
For succeeding crops which were already tested after application of 200 or 250 g a.s./ha, potential residues in the relevant matrices were estimated at an assumed application rate of 175 g a.s./ha or 300 g a.s./ha, respectively by applying the proportionality approach. The different residue scenarios were compared for each crop and the highest relevant residue was selected as basis for MRL setting. In agreement with the EFSA evaluation (EFSA 2015), the MRL was estimated based on the highest residue (HR was rounded up to the subsequent MRL class).

Table 7.2-20: Summary of available studies in field rotational crops

Primary crop	Rate (kg a.s./ha) (GS at application or PHI)	Succeeding crop group	Succeeding crop	Sowing intervals (DAT)	Reference / Remarks
EU data					DAR, NL, 2015 EFSA, 2015
Bare soil/ Lettuce	1 x 0.200 (BBCH 19)	Root and tuber vegetable	Carrot/ turnip (root and top)	1st rotation: 25-30 2nd rotation: 61-145 3rd rotation: 279-329	Schoening, R.; Bauer, J.; 2012; M-429091-01-1
Bare soil/ Lettuce	1 x 0.200 (BBCH 19)	Leafy vegetable	Lettuce (head)	1st rotation: 25-30 2nd rotation: 64-145 3rd rotation: 279-328	
Bare soil/ Lettuce	1 x 0.200 (BBCH 19)	Small grain cereal	Barley (green material, grain and straw)	1st rotation: 25-33 2nd rotation: 116-195 3rd rotation: 266-295	
Bare soil	2 x 0.125 (10 day interval)	Root and tuber vegetable	Potato (tuber)	1st rotation: 25-33	Schulte, G.; 2012; M-438341-01-1
Bare soil	2 x 0.125 (10 day interval)	Stem vegetable	Leek (whole plant	1st rotation: 26-33	Schulte, G.; 2012; M-438384-01-1

Primary crop	Rate (kg a.s./ha) (GS at application or PHI)	Succeeding crop group	Succeeding crop	Sowing intervals (DAT)	Reference / Remarks
			without root)		
Bare soil	2 x 0.125 (10 day interval)	Fruiting vegetable	Cucumber (fruit)	1st rotation: 25-30	Schulte, G.; 2012; M-438343-01-1
Bare soil	2 x 0.125 (10 day interval)	Bulb vegetable	Onion (bulb)	1st rotation: 25-33	Schulte, G.; 2012; M-438397-01-1
Bare soil	2 x 0.125 (10 day interval)	Legume vegetable	Bean (pods)	1st rotation: 25-30	Schulte, G.; 2012; M-438394-01-1
Bare soil	2 x 0.125 (10 day interval)	Pulses	Field pea (dry seed)	1st rotation: 25-35	Schulte, G.; 2012; M-438582-01-1
Bare soil	2 x 0.125 (10 day interval)	Oilseeds	Oilseed rape (seed)	1st rotation: 27-41	Schulte, G.; 2012; M-443092-01-1
New data					
Bare soil	1 x 0.300	Root and tuber vegetable	Potato (tuber)	2nd rotation: 138-181 3rd rotation: 295-343	Freitag, T.; 2018; M-613149-01-1
Bare soil	1 x 0.175	Oilseeds	Oilseed rape (seed)	2nd rotation: 137 3rd rotation: 335	Freitag, T.; 2018; M-617178-01-1
Bare soil	1 x 0.175	Oilseeds	Oilseed rape (seed)	2nd rotation: 158-167 3rd rotation: 273	Kaussmann, M.; van Berkum, S.; 2018; M-613466-01-1
Bare soil	1 x 0.175	Small grain cereal	Barley (green material, grain and straw)	2nd rotation: 137-174 3rd rotation: 335-337	Freitag, T.; Kaussmann, M.; 2018; M-612254-01-1
Bare soil	1 x 0.175	Small grain cereal	Barley (green material, grain and straw)	2nd rotation: 107-163 3rd rotation: 281-283	Kaussmann, M.; van Berkum, S.; 2018; M-613602-01-1
Bare soil	1 x 0.175	Cereals	Maize (green material, kernel, rest of plant)	2nd rotation: 131-167 3rd rotation: 291-365	Freitag, T.; Effertz, C.; 2018; M-617942-02-1
Bare soil	1 x 0.175	Cereals	Maize (green material, kernel, rest of plant)	2nd rotation: 139-204 3rd rotation: 313-324	Freitag, T.; van Berkum, S.; 2018; M-617943-01-1
Bare soil	1 x 0.300	Fruits	Strawberry (fruit)	2nd rotation: 22 3rd rotation: 139-141	Freitag, T.; Hoffmeister, R.; 2018; M-617563-01-1
Bare soil	1 x 0.300	Fruits	Strawberry (fruit)	1st rotation: 29 2nd rotation: 133 3rd rotation: 363	Kaussmann, M; Freitag, T.; van Berkum, S; 2018; M-617923-01-1
Bare soil	1 x 0.300	Brassica vegetables	Cauliflower and broccoli (curd)	1st rotation: 21-30 2nd rotation: 129-161 3rd rotation: 321-339	Freitag, T.; Effertz, C.; 2018; M-617941-02-1

Table 7.2-21: Summary of residues detected in field rotational crops (new studies and *already submitted studies*)

Crop group	Crop	Application	Residue levels (mg/kg)

		PBI (day)	g/ha	on bare soil or primary crop	flupyradifurone [mg FPF/kg]	DFA [mg DFA/kg]
Root crop	Potato (tuber)	*25-33*	*250*	*bare soil*	*4 x <0.01*	*0.0094; 0.012; 0.064; 0.082*
		138-181	300	bare soil	4 x **<0.01**	0.012; 0.023; 0.042; **0.088**
		295-343	300	bare soil	4 x <0.01	<0.007; 0.012; 0.018; 0.019
	Turnip/ carrot (root/cull)	*25-30*	*200*	*lettuce*	*4 x <0.01*	*0.0090; 0.018; 0.020; 0.035*
		25-30	300	extrapolated from 200 g/ha	no extrapolation	0.014; 0.027; 0.030; **0.053**
		61-145	*200*	*bare soil*	*4 x **<0.01***	*<0.007; 0.0092; 0.010; 0.013*
		284-328	*200*	*bare soil*	*4 x <0.01*	*<0.007; 0.0088; 0.011; 0.014*
Bulb veg.	*Onion*	*25-33*	*250*	*bare soil*	*4 x **<0.01***	*<0.007; 0.019; 0.024; 0.054*
		25-33	300	extrapolated from 250 g/ha	no extrapolation	<0.0084; 0.023; 0.029; **0.065**
Stem veg.	*Leek*	*26-33*	*250*	*bare soil*	*4 x <0.01*	*0.007; 0.013; 0.032; 0.077*
		26-33	300	extrapolated from 250 g/ha	no extrapolation	0.0084; 0.016; 0.038; **0.092**
Fruits	Strawberry (fruits)	22-29	300	bare soil	4 x <0.01	0.009; 0.068; 0.098; **0.12**
		133-158	300	bare soil	4 x <0.01	0.012; 0.046; 0.068; 0.12
		270-341	300	bare soil	4 x <0.01	<0.007; 0.017; 0.024; 0.076
Fruiting veg.	*Cucumber*	*25-30*	*250*	*bare soil*	*4 x <0.01*	*0.016; 0.093; 0.11; 0.14*
		25-30	300	extrapolated from 250 g/ha	no extrapolation	0.019; 0.11; 0.13; **0.17**
Leafy veg.	*Lettuce*	*25-30*	*200*	*lettuce*	*2 x <0.01; 0.01; 0.03*	*<0.007; 0.011; 0.016; 0.037*
		25-30	300	extrapolated from 200 g/ha	2 x <0.015; 0.015; **0.045**	<0.011; 0.017; 0.024; **0.056**
		64-145	*200*	*bare soil*	*3 x <0.01; 0.01*	*3 x <0.007; 0.028*
		279-329	*200*	*bare soil*	*4 x <0.01*	*3 x <0.007; 0.015*
Brassica veg.	Cauliflower/ broccoli (curd)	21-30	300	bare soil	4 x <0.01	0.021; 0.023; 0.054; **0.065**
		129-161	300	bare soil	4 x <0.01	0.012; 0.035; 0.037; 0.058
		321-339	300	bare soil	4 x <0.01	0.008; 0.015; 0.023; 0.033
Cereals	Maize (kernel)	131-204	175	bare soil	4 x <0.01	<0.007; 0.021; 0.035; **0.037**
		291-365	175	bare soil	4 x <0.01	<0.007; 0.013; 0.019; 0.030
Cereals	Maize (forage)	131-204	175	bare soil	3 x <0.01; **0.030**	<0.007; 0.011; 0.015; **0.020**
		291-365	175	bare soil	3 x <0.01; 0.012	<0.007; 0.007; 0.008; 0.009
Cereals	Maize (stover)	131-204	175	bare soil	<0.01; 0.01 2 x **0.020**	<0.007; 0.007; 0.014; **0.022**
		291-365	175	bare soil	3 x <0.01; 0.010	3 x <0.007; 0.008
Cereals	Barley (grain)	107-174	175	bare soil	4 x <0.01	0.038; 0.047; 0.088; **0.12**
		281-337	175	bare soil	4 x <0.01	0.025; 0.040; 0.056; 0.088
	Barley (grain)	*25-33*	*200*	*lettuce*	*3 x <0.01*	*0.030; 0.11; 0.21*
		116-195	*200*	*bare soil*	*4 x <0.01*	*0.012; 0.030; 0.034; 0.088*
		116-195	175	extrapolated from 200 g/ha	*4 x <0.01*	0.011; 0.026; 0.030; 0.077

Crop group	Crop	Application			Residue levels (mg/kg)	
		PBI (day)	g/ha	on bare soil or primary crop	flupyradifurone [mg FPF/kg]	DFA [mg DFA/kg]
		266-295	*200*	*bare soil*	*4 x <0.01*	*0.020; 0.030; 0.032; 0.12*
		266-295	175	extrapolated from 200 g/ha	*4 x <0.01*	0.018; 0.026; 0.028; 0.11
Cereals	Barley (forage)	*25-33*	*200*	*lettuce*	*3 x <0.01; 0.02*	*0.0092; 0.014; 0.038; 0.12*
		107-174	175	bare soil	3 x <0.01, 0.013	0.008; 0.014; 0.028; **0.045**
		116-195	*200*	*bare soil*	*3 x <0.01; 0.01*	*0.0093; 0.010; 0.012; 0.034*
		116-195	175	extrapolated from 200 g/ha	4 x <0.01	*0.0081; 0.0088; 0.011; 0.030*
		281-337	175	bare soil	3 x <0.01; 0.010	<0.007; 0.020; 0.025; 0.027
		266-295	*200*	*bare soil*	3 x <0.01; 0.030	2 x <0.007; 0.0078; 0.049
		266-295	175	extrapolated from 200 g/ha	3 x <0.01; **0.026**	2 x <0.007; 0.007; 0.043
Cereals	Barley (straw)	*25-33*	*200*	*lettuce*	*<0.01; 0.01; 0.02, 0.04*	*<0.017; 0.018; 0.042; 0.11*
		107-174	175	bare soil	2 x <0.01, 0.018; **0.032**	3 x <0.03; 0.036
		116-195	*200*	*bare soil*	*3 x <0.01; 0.02*	*3 x <0.017; 0.033*
		116-195	175	extrapolated from 200 g/ha	*3 x <0.01; 0.018*	3 x <0.017; 0.029
		281-337	175	bare soil	4 x <0.01	4 x <0.03
		266-295	*200*	*bare soil*	4 x <0.01	2 x <0.017; 0.017; 0.055
		266-295	*175*	extrapolated from 200 g/ha	4 x <0.01	3 x <0.017; **0.048**
Oilseeds	Rape (seed)	*27-41*	*200*	*bare soil*	*4 x <0.01*	*0.017; 0.024; 0.030; 0.051*
		137-167	175	bare soil	3 x <0.01	0.0091, 0.014; **0.022**
		273-335	175	bare soil	3 x <0.01	0.007; 0.008; 0.013
Oilseeds	Rape (forage)	137-167	175	bare soil	4 x <0.01	0.010; 0.015; 0.056; **0.085**
		273-335	175	bare soil	4 x <0.01	0.007; 0.008; 0.044; 0.046
Pulses	*Field pea*	*25-35*	*250*	*bare soil*	*4 x <0.01*	*0.22; 0.33; 0.69; 0.76*
		25-35	300	extrapolated from 250 g/ha	no extrapolation	0.26; 0.40; 0.83; **0.91**
Legume veg.	*green bean*	*25-30*	*250*	*bare soil*	*4 x <0.01*	*0.087; 0.13; 0.19; 0.36*
		25-30	300	extrapolated from 250 g/ha	no extrapolation	0.10; 0.16; 0.23; **0.43**

Table 7.2-22: DFA residues in rotational crops [mg DFA/kg]

Crop	Rotation	DFA residues (expressed as DFA) in mg a.s./kg individual values	STMR	HR	Remark
Potato (tuber)	2nd rot.	0.012(1); 0.023; 0.042(1); 0.088(1)	**0.033**	**0.088**	cf. A 2.1.6.1.1 (M-613149-01-1)
	3rd rot.	<0.007; 0.012; 0.018(1); 0.019(1)	0.015	0.019	
Carrot/turnip (root)	1st rot.	0.014; 0.027; 0.030; 0.053	**0.029**	**0.053**	Schoening, R.; Bauer, J.; 2012; M-429091-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
	2nd rot.	<0.011; 0.014; 0.015; 0.020	0.014	0.020	
	3rd rot.	<0.011; 0.013; 0.017; 0.021	0.015	0.021	
Onion (bulb)	1st rot.	<0.0084; 0.023; 0.029; 0.065	0.026	0.065	Schulte, G.; 2012; M-438397-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)

Crop	Rotation	DFA residues (expressed as DFA) in mg a.s./kg individual values	STMR	HR	Remark
Leek (stem)	1st rot.	0.0084; 0.016; 0.038; 0.092	0.027	0.092	Schulte, G.; 2012; M-438384-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Oilseed rape (seed)	2nd rot. 3rd rot.	0.009; 0.014; 0.022 0.007; 0.007; 0.013	**0.014** 0.007	**0.022** 0.013	cf. A 2.1.6.1.2 and A 2.1.6.1.3 (M-617178-01-1 and M-613466-01-1)
Barley (green material)	2nd rot. 3rd rot.	0.008; 0.014; 0.028; 0.045 <0.007; 0.020; 0.025; 0.027	**0.021** 0.023	**0.045** 0.027	cf. A 2.1.6.1.4 and A 2.1.6.1.5 (M-612254-01-1 and M-613602-01-1)
Barley (grain)	2nd rot. 3rd rot.	0.038; 0.047; 0.088; 0.12* 0.025; 0.040; 0.056; 0.088*	**0.068** 0.048	**0.12** 0.088	cf. A 2.1.6.1.4 and A 2.1.6.1.5 (M-612254-01-1 and M-613602-01-1)
Barley (straw)	2nd rot. 3rd rot.	3 x <0.03; 0.036 3 x <0.017; 0.048	<0.03 **0.017**	0.036 **0.048**	cf. A 2.1.6.1.4 and A 2.1.6.1.5 (M-612254-01-1 and M-613602-01-1) Schoening, R.; Bauer, J.; 2012; M-429091-01-1, extrapolated to 175 g a.s./ha (original study evaluated by EFSA, 2015)
Maize (kernel)	2nd rot. 3rd rot.	<0.007; 0.021; 0.035; 0.037 <0.007; 0.013; 0.019; 0.030	**0.028** 0.016	**0.037** 0.030	cf. A 2.1.6.1.6 and A 2.1.6.1.7 (M-617942-02-1 and M-617943-01-1)
Maize (stover)	2nd rot. 3rd rot.	<0.007; 0.007; 0.014; 0.022 3 x <0.007; 0.008	**0.011** 0.007	**0.022** 0.008	cf. A 2.1.6.1.6 and A 2.1.6.1.7 (M-617942-02-1 and M-617943-01-1)
Cucumber (fruit)	1st rot.	0.019; 0.11; 0.13; 0.17	0.12	0.17	Schulte, G.; 2012; M-438343-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Strawberries (fruit)	1st rot. 2nd rot. 3rd rot.	0.009(1); 0.068; 0.098; 0.12 0.012; 0.046; 0.068(1); 0.12 <0.007; 0.017(1); 0.024(1); 0.076	**0.083** 0.057 0.021	**0.12** 0.12 0.076	cf. A 2.1.6.1.8 and A 2.1.6.1.9 (M-617563-01-1 and M-617923-01-1)
Lettuce (head)	1st rot. 2nd rot. 3rd rot.	<0.011; 0.017; 0.024; 0.056 <0.011; <0.011; <0.011; 0.042 <0.011; <0.011; <0.011; 0.023	**0.021** <0.011 <0.011	**0.056** 0.042 0.023	Schoening, R.; Bauer, J.; 2012; M-429091-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
French bean (pod)	1st rot.	0.10; 0.16; 0.23; 0.43	0.20	0.43	Schulte, G.; 2012; M-438394-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Cauliflower/ broccoli (corm)	1st rot. 2nd rot. 3rd rot.	0.012; 0.023; 0.054; 0.065 0.012; 0.035; 0.037; 0.058 0.008; 0.015; 0.023; 0.033	**0.039** 0.036 0.019	**0.065** 0.058 0.033	cf. A 2.1.6.1.10 (M-617941-02-1)
Field pea (dry seed)	1st rot.	0.26; 0.40; 0.83; 0.91	0.62	0.91	Schulte, G.; 2012; M-438582-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)

(1) BBCH 85 (if not indicated, fruits were collected at BBCH 87) * plants suffered from drought

Conclusion on rotational crops studies

The highest flupyradifurone and DFA residues (HR) detected in rotational crops were compared with the respective residues in primary crops. If the estimated HR in the rotational crop was higher as compared to

the MRL proposed for the primary crop, the highest residue in the rotational crop was used as basis for MRL setting.
Parent compound flupyradifurone was generally not detected above the LOQ of 0.01 mg/kg in rotational crops which are food items, except in one lettuce trial which showed residues at 0.03 mg/kg in the first rotation. Thus the flupyradifurone MRL is always driven by the residues in the primary crop. If no use is registered in a primary crop, the default MRL of 0.01 mg/kg can be applied to crops grown in rotation.
In contrast, almost all field rotational crop trials showed residues of the metabolite DFA. Therefore DFA MRLs have to be established in all crops that can be grown in rotation. In some cases, the DFA residues in primary crops were higher as compared to the DFA residues detected in the rotational crops. However for several crops/crop groups (e.g. legume vegetables and pulses) the highest DFA residues were estimated to be in the rotational crops and as a consequence these residues have to be used as the basis for MRL setting. The new MRL proposals were submitted in March 2018 (updated in July 2018).

Table 7.2-23: Flupyradifurone residues in rotational crops [mg a.s./kg]

Crop group	Crop tested	HR (rot. crop)	approved MRL[1)]	Remark
berries and small fruits	strawberries	<0.01	0.4	MRL based on residues in primary crop
root and tuber vegetables	potato	<0.01	0.01*	Default MRL for crop group => IT will be more critical [2)]
	turnip/carrot	<0.01	0.01*	
bulb veg.	onion	<0.01	0.01*	Default MRL for crop group => IT will be more critical[2)]
solanacea	cucumber	<0.01	0.7 (tomato) 0.9 (pepper) 0.7 (aubergine)0 0.01* (others)	MRL based on residues in primary crop => IT will be more critical[2)]
cucurbits with edible peel	cucumber	<0.01	0.6	MRL based on residues in primary crops
cucurbits with inedible peel, excl. watermelon	cucumber	<0.01	0.01*	Default MRL for crop group, except watermelon (MRL for watermelon: 0.15 mg/kg) => IT will be more critical[2)]
brassica veg.	cauliflower	<0.01	0.01*	Default MRL for crop group
salad plants, except escaroles	lettuce	0.045	0.03 (+) (salad plants)	new MRL proposal: 6 mg/kg extrapolation from lettuce (leaf varieties; 6 trials available) [2)]
lettuces	lettuce	0.045	5 (lettuces)	MRL based on residues in primary crop (head and leafy varieties) [2)]
escaroles	lettuce	0.045	0.03 (+)	MRL proposal: 0.05 mg/kg [2)]
spinaches and similar leaves	lettuce	0.03	0.03 (+)	new MRL proposal: 6 mg/kg extrapolation from lettuce (leaf varieties, 6 trials available) [2)]
legume veg.	beans with pods	<0.01	0.01*	Default MRL for crop group
stem veg.	leek	<0.01	0.01*	Default MRL for crop group
pulses	field pea	<0.01	0.01*	Default MRL for crop group => IT will be more critical[2)]
oilseeds	oilseed rape	<0.01	0.01*	Default MRL for crop group => IT will be more critical[2)]
cereal grains	barley	<0.01	0.01*	Default MRL for crop group => IT will be more critical[2)]
	maize	<0.01	0.01*	

1) MRL according to Commission Regulation (EU) 2016/1902
2) dER was already submitted in June 2018 (updated in July 2018) in order to adapt the MRL
(+) The European Food Safety Authority identified some information on rotational crops as unavailable. As a consequence the new studies have been presented in an dER submitted in March 2018 (updated in July 2018)
* Indicates the lower limit of analytical determination

Table 7.2-24: DFA residues in rotational crops [mg a.s./kg]

Crop group	Crop tested	HR (rot. crop)	approved MRL[1)]	Remark
berries and small fruits	strawberries	0.12	0.03	new MRL proposal: 0.15 mg/kg[2)]
root and tuber vegetables	potato	0.088	0.09 (+)	MRL of 0.09 mg/kg confirmed => IT will be more critical[2)]
	turnip/carrot	0.053	0.09 (+)	

Crop group	Crop tested	HR (rot. crop)	approved MRL[1)]	Remark
bulb vegetables	onion	0.065	0.06 (+)	new MRL proposal: 0.07 mg/kg => IT will be more critical [2)]
solanacea	cucumber	0.17	0.15 (+)	new MRL proposal: 0.20 mg/kg => IT will be more critical[2)]
cucurbits with edible peel	cucumber	0.17	0.4	rot. crops covered by establ. MRL (based on primary crop)
cucurbits with inedible peel	cucumber	0.17	0.15 (+)	new MRL proposal: 0.20 mg/kg => IT will be more critical[2)]
brassica veg.	cauliflower/ broccoli	0.065	0.02*	MRL proposal: 0.07 mg/kg[2)]
salad plants, except escaroles	lettuce	0.056	0.04 (+) (salad plants)	new MRL proposal: 0.1 mg/kg for whole group (0251000), except escaroles (0251030) and lettuces (0251020) extrapolation from lettuce (leaf varieties; 6 trials available) [2)]
lettuces	lettuce	0.056	0.09 (lettuce)	MRL based on residues in primary crop (head and leafy varieties) [2)]
escaroles	lettuce	0.056	0.04 (+)	new MRL proposal: 0.06 mg/kg[2)]
spinaches and similar leaves	lettuce	0.056	0.04 (+)	new MRL proposal: 0.1 mg/kg extrapolation from lettuce (leaf varieties; 6 trials available) [2)]
legume veg.	beans with pods	0.43	0.4 (+)	new MRL proposal: 0.5 mg/kg[2)]
stem vegetables	leek	0.092	0.08 (+)	new MRL proposal: 0.1 mg/kg[2)]
pulses	field pea	0.91	0.8 (+)	new MRL proposal: 1.0 mg/kg => IT will be more critical[2)]
oilseeds	rape seeds	0.022	0.05 (+)	new MRL proposal: 0.03 mg/kg => IT will be more critical[2)]
cereals	barley grain	0.12	0.3 (+)	new MRL proposal: 0.15 mg/kg => IT will be more critical[2)]
	maize kernel	0.037	0.3 (+)	new MRL proposal: 0.04 mg/kg => IT will be more critical[2)]

1) MRL according to Commission Regulation (EU) 2016/1902
2) dER was already submitted in June 2018 (updated in July 2018) in order to adapt the MRL
(+) The European Food Safety Authority identified some information on rotational crops as unavailable. As a consequence the new studies have been presented in an dER submitted in March 2018 (updated in July 2018)
* Indicates the lower limit of analytical determination

In order to conduct the dietary risk assessments, the total residues (sum of flupyradifurone and DFA, expressed as flupyradifurone) in food items of field rotational crops are needed. A summary of the total residues is given in Table 7.2-25.

Table 7.2-25: Sum of flupyradifurone and DFA residues in rotational crops [mg a.s. equivalents/kg]

Crop	Rotation	Total residues (sum of FPF and DFA, expressed as FPF) in mg a.s./kg individual values	STMR	HR	Remark
Potato (tuber)	2nd rot. 3rd rot.	0.046(1); 0.079; 0.14(1); 0.27(1) <0.03; 0.047; 0.064(1); 0.067(1)	**0.11** 0.056	**0.27** 0.67	cf. A 2.1.6.1.1 (M-613149-01-1)
Carrot/turnip (root)	1st rot. 2nd rot. 3rd rot.	0.06; 0.09; 0.11; 0.17 0.045; 0.060; 0.060; 0.075 0.045; 2 x 0.060; 0.075	**0.098** 0.060 0.060	**0.17** 0.075 0.075	Schoening, R.; Bauer, J.; 2012; M-429091-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Onion (bulb)	1st rot.	<0.036, 0.082; 0.097; 0.20	0.090	0.20	Schulte, G.; 2012; M-438397-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Leek (stem)	1st rot.	0.036; 0.059; 0.13; 0.29	0.095	0.29	Schulte, G.; 2012; M-438384-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Oilseed rape (seed)	2nd rot. 3rd rot.	0.037; 0.053; 0.077 <0.03; 0.033; 0.050	**0.053** 0.033	**0.077** 0.050	cf. A 2.1.6.1.2 and A 2.1.6.1.3 (M-617178-01-1 and M-613466-01-1)
Barley (grain)	2nd rot. 3rd rot.	0.12; 0.15; 0.27; 0.38* 0.085; 0.13; 0.18; 0.27*	**0.21** 0.16	**0.38** 0.27	cf. A 2.1.6.1.4 and A 2.1.6.1.5 (M-612254-01-1 and M-613602-01-1)
Maize (kernel)	2nd rot. 3rd rot.	<0.03; 0.073; 0.11; 0.12 <0.03; 0.050; 0.068; 0.099	**0.092** 0.059	**0.12** 0.099	cf. A 2.1.6.1.6 and A 2.1.6.1.7 (M-617942-02-1 and M-617943-01-1)
Cucumber (fruit)	1st rot.	0.068; 0.35; 0.40; 0.50	0.37	0.50	Schulte, G.; 2012; M-438343-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Strawberries (fruit)	1st rot. 2nd rot. 3rd rot.	0.037(1); 0.22; 0.31; 0.37 0.045(1); 0.15; 0.22(1); 0.38 <0.03; 0.061(1); 0.083(1); 0.24	**0.27** 0.19 0.072	0.37 **0.38** 0.24	cf. A 2.1.6.1.8 and A 2.1.6.1.9 (M-617563-01-1 and M-617923-01-1)
Lettuce (head)	1st rot. 2nd rot. 3rd rot.	<0.045; 0.060; 0.090; 0.21 3 x <0.045; 0.15 3 x <0.045; 0.090	**0.075** <0.045 <0.045	**0.21** 0.15 0.09	Schoening, R.; Bauer, J.; 2012; M-429091-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
French bean (pod)	1st rot.	0.32; 0.47; 0.70; 1.3	0.58	1.3	Schulte, G.; 2012; M-438394-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)
Cauliflower/ broccoli (corm)	1st rot. 2nd rot. 3rd rot.	0.072; 0.080; 0.17; 0.21 0.046; 0.12; 0.12, 0.18 0.033; 0.056; 0.078; 0.11	**0.13** 0.12 0.067	**0.21** 0.18 0.11	cf. A 2.1.6.1.10 (M-617941-02-1)
Field pea (dry seed)	1st rot.	0.79; 1.2; 2.5; 2.8	1.9	2.8	Schulte, G.; 2012; M-438582-01-1 extrapolated to 300 g a.s./ha (original study evaluated by EFSA, 2015)

(1) BBCH 85 (if not indicated, fruits were collected at BBCH 87) * plants suffered from drought

Soil samples collected in selected trials prior to re-cropping showed that the only soil residue that can be detected is parent BYI 02960. Residues of the metabolite DFA were always below the LOQ of 0.005 mg/kg (expressed as DFA). This result is well in agreement with the degradation behaviour of DFA

revealed in the Efate studies. DFA degradation follows a single first order kinetic and shows a rather short half-live (<150 days).
As rotational crops show only very rarely flupyradifurone (BYI 02960) residues (only in some feed items and in lettuce at very low levels), but in almost all cases DFA residues, an immediate uptake of DFA from the soil – as soon as it is formed in soil – by the plants could provide an explanation for the DFA residues in the plant commodities. An alternative explanation would be uptake of flupyradifurone from the soil and immediate degradation to DFA in the plant.
As expected, the highest BYI 02960 residues in soil were detected before sowing/planting the crops of the first rotation (21-30 days after treatment of the bare soil) and the lowest residues before sowing/planting the crops of the third rotation (273-365 days after treatment of the bare soil). The overall highest residue detected in the plough layer (0-20 cm) was 0.1 mg/kg of parent BYI 02960. A summary of the soil analysis is shown in the following table.

Rotation	**Plant-back interval [days]**	**Residues in soil (0-20 cm) [mg/kg]***	
		BYI 02960	**DFA**
1st rotation	21-30	<0.005-0.10	<0.005
2nd rotation	107-204	<0.005-0.067	<0.005
3rd rotation	273-365	<0.005-0.036	<0.005

* expressed in analyte equivalents

Evaluator comment:
Information given by the Applicant is sufficient.

Excerpt from EFSA Journal 2020;18(11):6298:
"A wide range of rotational crop field studies were submitted for the EU pesticides peer review and in the framework of the previous EFSA assessments (EFSA, 2015, 2020; Netherlands, 2017). Flupyradifurone was either applied on a bare soil or on a primary crop lettuce at application rates ranging from 125 to 300 g/ha. Studies indicate a significant uptake of metabolite DFA in rotational crops. Consequently, the EU pesticide peer review derived provisional MRLs for difluoroacetic acid in rotational crops (EFSA, 2015). These provisional MRLs were further assessed by EFSA in the light of rotational crop studies which were submitted by the EMS Netherlands for the assessment of Article 12 confirmatory data and for the setting of MRLs for flupyradifurone and DFA (EFSA, 2020).
Since the total annual application rate in the intended use GAPs on oilseed rape and mustard is significantly lower than the application rates on primary crops for which the residues of DFA in rotational crops were estimated in the previous EFSA assessment (EFSA, 2020), the magnitude of DFA residues in rotational crops was not re-assessed and the conclusions of the previous EFSA opinion are applicable."

Oilseed rape and mustard can be grown in a crop rotation.

New field rotational crop studies were conducted in cereals, maize and oilseed rape after application of 175 g a.s./ha and in potato, brassica vegetables and strawberries after application of 300 g a.s./ha to bare soil. More details are presented in Appendix 2, point A 2.1.6.
It should be noted that *parent compound flupyradifurone was generally not detected above the LOQ of 0.01 mg/kg in rotational crops, except in one lettuce trial which showed residues at 0.03 mg/kg in the first rotation. Thus the flupyradifurone MRL is always driven by the residues in the primary crop.*
In contrast, almost all field rotational crop trials showed residues of the metabolite DFA. Therefore DFA MRLs have to be established in all crops that can be grown in rotation. In some cases, the DFA residues in primary crops were higher as compared to the DFA residues detected in the rotational crops.
The new MRL proposals were submitted in 2018.

EMS-The Netherlands concluded in Evaluation report on MRLs for flupyradifurone in imported commodities, February 2019:
"As rotational crops show only very rarely flupyradifurone (BYI 02960) residues (only in some feed items and in lettuce at very low levels), but in almost all cases DFA residues, an immediate uptake of DFA from the soil – as soon as it is formed in soil – by the plants could provide an explanation for the DFA residues in the plant commodities. An alternative explanation would be uptake of flupyradifurone from the soil and immediate degradation to DFA in the plant."

No waiting periods beyond normal agricultural practice are proposed for succeeding crops to be planted.

7.2.7 Other / special studies (KCA6.10, 6.10.1)

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of Flupyradifurone FS 480. Therefore, other special studies are not needed.

Evaluator comment:
There is no sufficient data to support application of DLT+FPF EC 85 at flowering oilseed rape. Following the review done by zRMS, the applicant would like to request to restrict the use of the product to applications outside of flowering. The table GAP has been corrected.

7.2.8 Estimation of exposure through diet and other means (KCA 6.9)

Toxicological reference values relevant for dietary risk assessment are reported in the summary of the evaluation (see 7.1.2).

7.2.8.1 Input values for the consumer risk assessment

The residue definition for risk assessment comprises the parent compound flupyradifurone and its metabolite DFA (sum of both components, expressed as flupyradifurone).
No TMDI calculations – based on MRLs – have been performed as the two residue definitions for monitoring/enforcement of MRLs are not in agreement with the residue definition for risk assessment.

In order to evaluate the potential chronic and acute exposure to flupyradifurone residues through the diet, the respective International Estimated Daily Intakes (IEDI) and International Estimated Short-Term Intakes (IESTI) were estimated using EFSA PRIMo rev. 3 (Pesticide Residue Intake Model). All crops were considered for which there are registered uses within the EC. An ADI of 0.064 mg/kg bw/d and an ARfD of 0.15 mg/kg bw (EFSA, 2015) were used as toxicological reference values.

To reflect the most recent evaluation on flupyradifurone, the chronic exposure was calculated based on the median residue values reported in the previously issued EFSA Reasoned Opinion on setting new MRLs for flupyradifurone in strawberries, blackberries and raspberries (EFSA Journal 2016;14(3):4423) and the median residues (STMR values) detected in the supervised field trials of primary or field rotational crop trials submitted in this dRR. If data are available for primary and rotational crops, always the higher residue values were chosen as input values.

The acute exposure assessment was performed based on the median residues for oilseed rape (as requested by Primo rev.3 for oilseeds) and highest residue values (HR) of matrices of animal origin.

The input values used for the dietary exposure calculation are summarized in Table 7.2-26.

Table 7.2-26: Input values for the consumer risk assessment

Commodity	Chronic risk assessment (IEDI)		Acute risk assessment (IESTI)	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Risk assessment residue definition (sum of flupyradifurone and DFA, expressed as flupyradifurone)				
Pome fruit	0.115	STMR, primary crop, (EFSA, 2015 & 2016)	-	Acute risk assessment undertaken only with regard to crops under consideration
Grape	0.25	STMR, primary crop, (EFSA, 2015 & 2016)	-	
Strawberries	0.27	STMR succeeding crop, (cf. Table 7.2-25 and Appendices A 2.1.6.1.8 and A 2.1.6.1.9)	-	

Commodity	Chronic risk assessment (IEDI)		Acute risk assessment (IESTI)	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Raspberries, blackberries	0.39	STMR, primary crop, (EFSA, 2016)	-	
Root and tuber vegetables, except carrot and turnip	0.11	STMR, succeeding crop (potato), extrapolated to 300 g a.s./ha, (cf. Table 7.2-25 and Appendix A 2.1.6.1.1)	-	
Carrot/ turnip	0.098	STMR, succeeding crop (carrot/turnip), (EFSA, 2015), extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	
Bulb vegetables	0.090	STMR, succeeding crop (onion), (EFSA, 2016), extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	Acute risk assessment undertaken only with regard to crops under consideration
Fruiting vegetables, except watermelon	0.37	STMR, succeeding crop (cucumber), (EFSA, 2015 & 2016), extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	
Watermelon	0.065	STMR (pulp), primary crop, (EFSA, 2015 & 2016)	-	
Brassica vegetables	0.13	STMR, succeeding crop (cucumber), (EFSA 2015 & 2016), extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	
Lettuces and other salad plants, except lettuce and escaroles	1.05	STMR, primary crop (lettuce), extrapolation from lettuce (leafy varieties only)	-	
Lettuce	0.80	STMR, primary crop, (EFSA, 2015 & 2016)	-	
Escaroles	0.075	STMR, succeeding crop (lettuce), (EFSA, 2015) extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	
Spinach and similar (leaves), herbs and edible flowers	1.05	STMR, primary crop (lettuce), extrapolation from lettuce (leafy varieties only)	-	
Grape leaves and similar species, Watercress Witloofs/Belgian endives	0.075	STMR, succeeding crop (lettuce), (EFSA, 2015) extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	
Legume vegetables	0.58	STMR, succeeding crop (French bean), (EFSA, 2015 & 2016) extrapolated to 300 g a.s./ha (cf. Table 7.2-25)	-	
Stem vegetables	0.095	STMR, succeeding crop (leek), (DAR, NL, 2015) extrapolated to 300 g a.s./ha,	-	

Commodity	Chronic risk assessment (IEDI)		Acute risk assessment (IESTI)	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
		(cf. Table 7.2-25)		
Pulses	1.9	STMR, succeeding crop (field pea), (DAR, NL, 2015) extrapolated to 300 g a.s./ha, (cf. Table 7.2-25)	-	
Oilseed rape seed	0.28	STMR, primary crop (oilseed rape), (cf. Table 7.2-9 and Appendix A 2.1.3.1)	0.28	STMR, primary crop (oilseed rape), cf. Table 7.2-9 and Appendix A 2.1.3.1)
Mustard seed	0.28	STMR, primary crop (oilseed rape), (cf. Table 7.2-9 and Appendix A 2.1.3.1)	0.28	STMR, primary crop (oilseed rape) (cf. Table 7.2-9 and Appendix A 2.1.3.1)
Oilseeds, except seeds of oilseed rape & mustard	0.053	STMR, succeeding crop (oilseed rape), (cf. Table 7.2-25 and Appendices A 2.1.6.1.2 and A 2.1.6.1.3)	0.053	STMR, succeeding crop (oilseed rape) (cf. Table 7.2-25 and Appendices A 2.1.6.1.2 and A 2.1.6.1.3)
Cereals, excluding corn	0.21	STMR, succeeding crop (cereals), (cf. Table 7.2-25 and Appendix A 2.1.6.1.4)	-	Acute risk assessment undertaken only with regard to crops under consideration
Corn	0.092	STMR, succeeding crop (cereals), (cf. Table 7.2-25 and Appendix A 2.1.6.1.6)	-	
Hops	1.08	STMR, primary crop, (EFSA, 2015 & 2016)	-	
Swine: Muscle	0.100	STMR, cf Table 7.2-18	0.179	HR, cf Table 7.2-18
Swine: Meat	0.101	STMR, calculated[1]	0.193	HR, calculated[1]
Swine: Fat	0.105	STMR, cf Table 7.2-18	0.248	HR, cf Table 7.2-18
Swine: Liver	0.084	STMR, cf Table 7.2-18	0.138	HR, cf Table 7.2-18
Swine: Kidney	0.137	STMR, cf Table 7.2-18	0.231	HR, cf Table 7.2-18
Bovine: Muscle	0.134	STMR, cf Table 7.2-18	0.213	HR, cf Table 7.2-18
Bovine: Meat	0.135	STMR, calculated[1]	0.230	HR, calculated[1]
Bovine: Fat	0.141	STMR, cf Table 7.2-18	0.298	HR, cf Table 7.2-18
Bovine: Liver	0.117	STMR, cf Table 7.2-18	0.173	HR, cf Table 7.2-18
Bovine: Kidney	0.197	STMR, cf Table 7.2-18	0.292	HR, cf Table 7.2-18
Sheep: Muscle	0.147	STMR, cf Table 7.2-18	0.251	HR, cf Table 7.2-18
Sheep: Meat	0.149	STMR, calculated[1]	0.285	HR, calculated[1]
Sheep: Fat	0.155	STMR, cf Table 7.2-18	0.421	HR, cf Table 7.2-18
Sheep: Liver	0.129	STMR, cf Table 7.2-18	0.203	HR, cf Table 7.2-18
Sheep: Kidney	0.218	STMR, cf Table 7.2-18	0.343	HR, cf Table 7.2-18
Poultry:Muscle	0.118	STMR, cf Table 7.2-18	0.147	HR, cf Table 7.2-18

Commodity	Chronic risk assessment (IEDI)		Acute risk assessment (IESTI)	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Poultry: Meat	0.111	STMR, calculated[1)]	0.139	HR, calculated[1)]
Poultry: Fat	0.051	STMR, cf Table 7.2-18	0.062	HR, cf Table 7.2-18
Poultry: Liver	0.187	STMR, cf Table 7.2-18	0.245	HR, cf Table 7.2-18
Poultry: Kidney	0.187	based on STMR of liver	0.245	based on HR of liver
Milk	0.053	STMR, cf Table 7.2-18	0.061	HR, cf Table 7.2-18
Eggs	0.135	STMR, cf Table 7.2-18	0.180	HR, cf Table 7.2-18

EFSA, 2015: EFSA Conclusion on the peer review of the pesticide risk assessment of the active substance flupyradifurone, published March 21, 2016 (EFSA Journal 2015;13(2):4020), replaces version of February 10, 2015

EFSA, 2016: EFSA RO: Setting of new maximum residue levels for flupyradifurone in strawberries, blackberries and raspberries, published March 16, 2016 (EFSA Journal 2016;14(3):4423)

ER NL, 2018: Evaluation report: MRL application on the setting of MRLs in various rotational crops and animal commodities, submitted in March 2018 by the applicant, under evaluation of the NL (EMS)

1) Residues for meat were calculated considering a certain fat proportion in the consumed meat. For mammal meat, a ratio muscle/fat of 80/20 is assumed, whereas for poultry meat a ratio muscle/fat of 90/10 is assumed (FAO, 2009).

7.2.8.2 Conclusion on consumer risk assessment

The IEDI, calculated according to the EFSA model, amounts to between 1% (IE child) and 17% (NL toddler) of the ADI. All ADI usage values in this evaluation are well below 100%, and thus no risk to the consumer was identified. As a consequence no additional refinement was considered to be necessary.

The EU uses for oilseeds (oilseed rape and mustard seeds) which are intended for submission did not result in an exceedance of the ARfD. In fact, the IESTI value for the unprocessed commodities of rapeseeds and mustard seeds account for less than 1% of the ARfD (results for children, unprocessed commodities).

Extensive calculation sheets are presented in Appendix 3.

Table 7.2-27: Consumer risk assessment

TMDI (% ADI) according to EFSA PRIMo 3	Not suitable, please refer to IEDI
IEDI (% ADI) according to EFSA PRIMo 3	17% (NL toddler)
IESTI (% ARfD) according to EFSA PRIMo 3*	Results for children (<1% of ARfD): unprocessed and processed rapeseeds: 0.3% unprocessed mustard seeds: 0.2% Results for adults (<1% of ARfD): unprocessed rapeseeds: 0.1% unprocessed mustard seeds: 0.1%
NTMDI (% ADI) **	Not relevant
NEDI (% ADI)**	
NESTI (% ARfD)**	

* include raw and processed commodities if both values are required for PRIMo

** if national model is available

The proposed use of flupyradifurone in the formulation DLT+FPF EC 85 does not represent unacceptable acute and chronic risks for the consumer.

Evaluator comment:

Information given by the Applicant is sufficient.

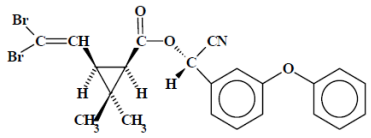
The calculation of the IEDI using EFSA model (version 3) and STMR values reported in EFSA Journal 2016;14(3):4423 and STMR values detected in the supervised field trials of primary or field rotational crop trials submitted in this registration report led to a utilisation of the ADI of 17% with the NL toddler being the population group with the highest value. For this diet, the highest contributor is milk:cattle with 5% of the ADI. The intended uses will not result in a consumer chronic exposure exceeding the ADI.
An acute consumer risk assessment was performed based on the median residues for oilseed rape and highest residue values (HR) of matrices of animal origin. The highest International Estimated Short-Term Intake (IESTI) is at 0.3% and 0.1% of the ARfD for the consumption of rapeseeds/canola by children and by adults respectively.

The data available are considered sufficient for risk assessment. The chronic and the short-term intakes of flupyradifurone residues are unlikely to present a public health concern.

7.3 Deltamethrin

General data on deltamethrin are summarized in the table below.

Table 7.3-1: General information on deltamethrin

Deltamethrin (ISO Common Name)	Active substance
IUPAC	(S)-a-cyano-3-phenoxybenzyl (1R,3R)-3-(2,2- dibromovinyl)-2,2-dimethylcyclopropane carboxylate
Chemical structure	
Molecular formula	$C_{22}H_{19}Br_2NO_3$
Molar mass	505.2
Chemical group	Pyrethroids
Mode of action (if available)	Nervous system
Systemic	No
Company (ies)	Bayer
Rapporteur Member State (RMS)	HUN
Approval status	Approved Date of (01/11/2003) and reference to decision (COMMISSION DIRECTIVE 2003/5/EC. http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32003L0005&from=EN
Restriction (e.g. is restricted to use as "...")	Only uses as insecticide
Review Report	6504/VI/99-final 17 October 2002
Current MRL regulation	Reg. (EU) 2018/832 Reg. (EU) 2018/687 Reg. (EU) 2017/1016 Reg. (EU) 2016/1822 ~~Reg SANTE/10024/2016~~ Reg. (EU) No 441/2012 Reg. (EU) No 524/2011 Reg. (EC) No 839/2008 Reg. (EC) No 149/2008
Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed	Yes
EFSA Journal : Conclusion on the peer review	Yes

EFSA Journal: conclusion on article 12	Yes** (EFSA 2015)
Current MRL applications on intended uses	None

7.3.1 Stability of Residues (KCA 6.1)

7.3.1.1 Stability of residues during storage of samples

Available data

One new stability study has been submitted by the applicant in the framework of this application. Results are summarized in the Table below. The detailed assessment of these studies is presented in Appendix 2.

Table 7.3-2: Summary of stability data achieved at ≤ - 18°C (unless stated otherwise)

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference
Data relied on in EU			
Plant products			
Tomato, cherry tomato	High water content	13.5 months 24 months	Czarnecki, J. J.; 1992, M-152267-01-1 report A73998, FAO 2002
Cabbage	High water content	24 months	Williams, L.; 2000, M-238621-01-1, report B002954, FAO 2002
Ginned cotton	Dry matrices	38 months	McKinney, F. R, ; 1995, M-149576-01-1 report A71103, Sweden 1998
Cotton oil	High oil content	36 months	
Cotton soap stock	High oil content	24 months	
Cotton seed	High oil content	38 months	
Corn grain	High protein content	36 months	Czarnecki, J. J.; 1996, M-139715-01-1, report A55828, submitted in AIR dossier
Corn Forage	High water content	36 months	
Corn Starch	High starch content	36 months	
Corn Flour	Dry matrices	36 months	
Corn Oil	High oil content	36 months	
Animal Products			
Poultry	Muscle	11 months at -12°C	FAO 2002; Sweden 2002
Poultry	Liver	11 months at -12°C	
Poultry	Kidney	11 months at -12°C	
Poultry	Egg	11 months at -12°C	
Cow	Milk	7 months at -20°C	
New data submitted during the renewal process			
Plant products			
Orange	High acid content	24 months	Ballesteros, C., 2012, M-441996-01-1, report Nr 09-07, Appendix 2

During the transport and storage of samples from supervised residue trials of oilseed rape (studies 16-2131 and 16-2169), storage temperature deviations occurred. The worst-case being a temperature raise during a time period of 13 hours with a maximum temperature of -8.5°C, resulting in an average temperature of -15°C during these 15 hours. Since none of the samples thawed, the stability of residues

can be assumed, particularly when considering that several short-term temperature increases ≥0°C (up to 6.5°C) were also observed in the long-term stability study.
Two short-term storage stability studies at exaggerated temperatures are available, which show the stability of deltamethrin residues under similar conditions:

- The first study is available for matrices with high water and high protein content (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1). Stability of deltamethrin residues was shown when samples were stored for 8 hours at +1°C, followed by 6 days at -7°C. Unfortunately no matrices with high oil content were included in this study.
- Within the second study (Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1) residues of deltamethrin were found to be stable in samples of oilseed rape (seeds and straw) when stored for 10 days at -8.5°C followed by 5 hours at +6°C.

Both studies are summarized in Appendix 2.

Conclusion on stability of residues during storage
Data are sufficient to cover the trials supporting intended GAPs of DLT+FPF EC 85

Evaluator comment:
The endpoints, as presented in EFSA Journal 2015;13(11):4309 are summarized below:
"Storage stability of deltamethrin was demonstrated at -20 °C for a period of 24 months in high water content commodities (cabbage and tomatoes) (FAO, 2002) and at -12°C for 30 months in high oil content commodities (cotton seed) and for 9 month in dry commodities (cereals grain) (Sweden, 1998). Storage stability studies on high acid content commodities are not available. However, considering that storage stability in tomatoes (which is borderline between a high water and a high acid content commodity) has been demonstrated for up to 24 months and that in samples from acidic commodities stored for up to 16 months residue levels were not significant different from samples stored for shorter period, a storage stability study on acidic commodities is only desirable."

One new stability study (Ballesteros, C.; 2012) has been submitted by the Applicant in the framework of this application.
Deltamethrin and its isomers (AE F108569 and AE 0035073) were shown to be stable in orange (fruit) for at least 25 months (751 days) under freezer conditions at about -18 °C.
Additionally two storage stability studies at exaggerated temperatures were performed.
1. Lakaschus, S.; Gizler, A.; 2017; S13-03307; M-480441-06-1: Residues of deltamethrin and its isomers, alpha-R-deltamethrin and trans-deltamethrin, in the plant matrices of high water and high protein content were stable (<30% decomposition) after storage at + 1°C for 8 hours, following a storage of 6 days at -7°C.
2. Winter, O.; Amann, S.; Giesler, W.; 2018; S17-05312; M-626405-01-1: Residues of deltamethrin and its isomers, alpha-R-deltamethrin and trans-deltamethrin in the plant matrices of oilseed rape (seed and straw) were stable during 10 days of storage at -8.5°C followed by 5 hours at +6°C.
The studies on the magnitude of residues are valid with regard to the storage stability.
No further data are required.

7.3.1.2 Stability of residues in sample extracts (KCA 6.1)

Available data
Relevant information on the stability of residues in the final or any intermediate extracts can be derived from the fortification experiments performed during sample analysis. Every analytical batch does contain at least one freshly fortified sample for concurrent recovery determination. The extracts of the fortified samples and of the study samples are handled and stored in parallel. If the recoveries in the fortified samples are within acceptable ranges, the stability of the sample extracts is considered as sufficiently proven.
In parallel, during the development of the method 00855/M004, (Lakaschus, S.; Winter; O.; 2009; 00855/M004; M-356934-01-1) a stability check was performed by fortifying control extracts of each matrix in toluene (or acetonitrile for OSR) at 50 ng/mL with trans-, cis-, α-(R)-deltamethrin. The test indicated no significant decline of the recoveries during a storage period of at least seven days in toluene and acetonitrile for all matrices under cool and dark conditions (+4°C).

Conclusion on stability of residues in sample extracts
In the trials submitted in this dossier all analysis were performed within 24 h after extraction. Besides in all studies, recovery experiments were performed concurrently with the analyzed samples. The recovery

rates for the studies presented in this dossier were acceptable or in the 70-110% range, meaning that residues were stable in the sample extracts.

Evaluator comment:
Information given by the Applicant is sufficient.

7.3.2 Nature of residues in plants, livestock and processed commodities

7.3.2.1 Nature of residue in primary crops (KCA 6.2.1)

Available data

The metabolism of deltamethrin has been investigated in apple, tomato, cotton, and maize (representing four diverse crops) after foliar treatment application with ^{14}C-deltamethrin. These studies have already been reported and evaluated by FAO (2002) and peer reviewed by EFSA (2015). The results are summarized in Table 7.3-3.

No new data are submitted in the framework of this application.

Table 7.3-3: Summary of plant metabolism studies

Crop Group	Crop	Label position	Application and sampling details					Reference
			Method, F or G (a)	Rate (kg a.s./ha)	No	Sampling (DAT)	Remarks	
EU data								
Fruits and fruiting vegetable	Apple	[gem-dimethyl-^{14}C]- and [^{14}C-benzyl]-deltamethrin	Foliar, F	0.06	2	28	-	Periasamy, R.; Kimmel, E. C.; Toia, R. F. (1994), M-149515-01-1; FAO, 2002; EFSA, 2015
	Tomato		Foliar, F	0.05	1	4, 14, 28	-	Merricks, L.; Swidersky, P., (1985), M-125042-01-1 FAO 2002; EFSA, 2015
			Local, G	14 µg/tomato	1			
Pulses and oilseeds	Cotton*	[^{14}C-benzyl]-deltamethrin	G	Foliar: 0.009 mg/plant Soil: 0.18 mg/plant hydroponic: 6.7 mg/plant	1	1, 3, 7	Translocation study	Salmon, J.; van Assche, C. J.; Salmon, M., (1977), M-149605-01-2, FAO 2002; EFSA, 2015
	Cotton leaf*	[Dibromo-^{14}C-vinyl]-, ^{14}C-cyano- and [^{14}C-benzyl]-deltamethrin	F, G	3-15 mg/kg leaf	1	14, 42	-	Ruzo, L. O.; Casida, J. E., (1979), M-093407-01-1, FAO, 2002; EFSA, 2015
	Cotton	[gem-dimethyl-^{14}C]- and [^{14}C-benzyl]-deltamethrin	Foliar, F	0.22	2	4, 10, 28	-	O'Grodnick, J. S.; Larson, J. D. (1990 and 1994), M-149567-01-1 and M-191128-02-1, FAO 2002; EFSA, 2015
Cereals	Maize/ corn	[gem-dimethyl-^{14}C]- and [^{14}C-benzyl]-deltamethrin	Foliar, F	0.11	2		-	Periasamy, R., Kimmel,E.C., Toia, R.F. (1994), M-149571-01-1, FAO 2002; EFSA, 2015

(a) Outdoor/field application (F) or glasshouse/protected/indoor application (G) * covers also leafy vegetables

Summary of plant metabolism studies reported in the EU

As stated in EFSA, 2015:

"The metabolism of deltamethrin in primary crops was investigated in the framework of the peer review (Sweden, 1998, 2002). The same studies have been considered in the JMPR Evaluation of deltamethrin (FAO, 2002).

The metabolism of deltamethrin in primary crops following foliar treatment has been investigated in fruits and fruiting vegetables (apples and tomatoes), pulses and oilseeds (cotton seed and leaves) and cereals (corn).

Deltamethrin was the predominant compound in all the crops tested accounting for up 77% total radioactive residue (TRR) (forage corn). In the studies analysing for the single isomers (apples, corn and cotton), the alpha-R-isomer and the trans-isomer accounted for up 34% TRR (apple) and for up to 19% TRR (apples), respectively. In all crops investigated, residues of metabolite 3-phenoxybenzylaldehyde (mPB aldehyde) were found but their levels were never relevant compared to parent and its isomers.

In the study on cotton investigating translocation, hydroponic and soil treatments resulted in a significant root uptake while, after foliar treatment, only a limited translocation throughout the plant was observed. After local applications on cotton leaves, deltamethrin was degraded to its trans-isomer more rapidly under field conditions than under glasshouse conditions.

No studies investigating the metabolism of deltamethrin following post-harvest treatment are available. However, considering the similar metabolic pathway of deltamethrin observed in all crops investigated, it can be assumed that, when applied in post-harvest treatments, deltamethrin will not undergo a more extensive metabolism. EFSA therefore concludes that no additional metabolism studies are required to support post-harvest treatments."

Conclusion on metabolism in primary crops

Sufficient data have been provided to acknowledge the metabolism of deltamethrin in primary crops.

As stated in EFSA, 2015:

"The metabolism of deltamethrin was investigated in apples, tomatoes, cotton seed and leaves, corn. Similar metabolic patterns were observed in these studies and a general residue definition for monitoring is proposed as deltamethrin."

Evaluator comment:

The metabolism of deltamethrin in primary crops was investigated in the framework of the peer review (Sweden, 1998, 2002). Information given by the Applicant is sufficient.

According to the EFSA Journal 2020;18(10):6271: *The metabolism of deltamethrin in primary crops belonging to the group of fruits (apples and tomatoes), pulses and oilseeds (cotton seed) and cereals (maize) was investigated in the framework of the MRL review. The metabolism studies after foliar and local treatment showed that the metabolic pathway is similar in all crop groups investigated. Deltamethrin was the main component of residues (up to 77% of the total radioactive residue (TRR)) with alpha-R-isomer and trans-isomer accounting for approximately 30–40% of the TRR.*

Residue definitions:

Based on the metabolic pattern identified in metabolism studies, the results of hydrolysis studies, the toxicological significance of deltamethrin relevant isomers and the capabilities of enforcement analytical methods, the following residue definitions were proposed by the MRL review (EFSA, 2015).

• residue definition for enforcement: deltamethrin (cis-deltamethrin);

• residue definition for risk assessment: sum of cis-deltamethrin and its alpha-R isomer and trans-isomer (provisional).

The residue definition for enforcement set in Regulation (EC) No 396/2005 is identical with the above-mentioned residue definition.

The same residue definitions are applicable to rotational crops and processed products (EFSA, 2015).

The risk assessment residue definition should be considered on a provisional basis, pending the assessment of further toxicological data investigating the toxicological properties of the alpha-R isomer and trans-isomer of deltamethrin (EFSA, 2015).

Taking into account of the proposed use assessed in this application, it can concluded that these residue definitions are appropriate and no further information is required.

7.3.2.2 Nature of residue in rotational crops (KCA 6.6.1)

Available data

A new metabolism study in rotational crops has been submitted by the applicant in the framework of this application. These studies are summarized in the Table below. The detailed assessment of these studies is presented in Appendix 2.

Table 7.3-4: Summary of metabolism studies in rotational crops

Crop group	Crop	Label position	Application and sampling details					Reference
			Method, F or G *	Rate (kg a.s./ha)	Sowing intervals (DAT)	Harvest Intervals (DAT)	Remarks	
EU data**								
Leafy vegetables	Lettuce	[^{14}C-benzyl]-deltamethrin	F	10 x 0.045	30, 120	139, 176, 190	Bare soil application	Erstfeld, K. M.; Larson, J. D.; Lange, B. D. (1991 / 1994), M-136651-02-2, FAO, 2002; EFSA, 2015
Root and tuber vegetables	Carrot					205, 254		
Cereals	Barley					139, 164, 196, 226		
New data (also submitted during the renewal process)								
Leafy vegetables	Swiss chard	[gem-dimethyl-^{14}C]-deltamethrin	G	0.041	30, 134	69, 80, 174, 188	Bare soil application	Schmeling, S.; Breuer-Rehm, M. (2012); M-431769-01-1, Appendix 2
Root and tuber vegetables	Turnip					86, 202		
Cereals	Wheat					69, 101, 127, 175, 234, 280		

* Outdoor/field application (F) or glasshouse/protected/indoor application (G) ** another study is mentionned in EFSA, 2015 but it is not a confined rotational crop studybut a rotational study (1986, Krebs, *et al*., M-115043-01-2).

Summary of plant metabolism studies reported in the EU

As stated in EFSA, 2015:

"The metabolism of deltamethrin in rotational crops – carrots, lettuce, barley - has been evaluated during the peer review (Sweden, 1998). One confined rotational crop study investigating the nature of residues following different plant-back intervals is available.

Following treatment of a bare soil at 10 × 0.45 kg a.s./ha, residues in rotational crops at harvest were generally found to be below 0.01 mg eq/kg, except in barley straw and foliage where residues amounted to 0.023 and 0.016 mg eq/kg, respectively. Consequently, considering the overdosing factor of the confined study (86N), no significant residues are expected in crops grown in rotation and a specific residue definition for rotational crops is not required."

Summary of new studies in rotational crops

In 2012, a confined rotational crop study was performed with [^{14}C]-gemdimethyl deltamethrin.

The objective of this study was to investigate the metabolism of deltamethrin in rotational crops after one pre-emergent spray application onto bare soil. The application rate of the test compound [gemdimethyl-^{14}C] deltamethrin was aimed to be about 10% above the highest annual field dose of approx. 37.5 g a.s./ha. The rotational crops used were Swiss chard, turnips and spring wheat. The intended timeframe for sowing was approx. 30, 150 and 365 days after soil treatment for the first, the second and the third rotation, respectively. The study was concluded after two rotations due to the very low TRRs (< 0.01 mg/kg) in all of the collected matrices.

No extraction of plant matrices was conducted during this study because of the very low TRR values. The

radioactivity in all samples was exclusively determined by combustion followed by LSC. There was no significant metabolism of deltamethrin detectable in confined rotational crops.

Conclusion on metabolism in rotational crops
Sufficient data have been provided to acknowledge the metabolism of deltamethrin in rotated crops. There is no need for a specific residue definition.

Evaluator comment:
Information given by the Applicant is sufficient.

A new metabolism study in rotational crops (Schmeling, S.; Breuer-Rehm, M.; 2012; MEF-11/669; M-431769-01-1) has been submitted by the applicant in the framework of this application.
The metabolism of deltamethrin was investigated in the rotational crops Swiss chard, turnips and spring wheat after one soil treatment with radiolabelled [gemdimethyl-^{14}C] deltamethrin. The actual application rate corresponded to 41 g a.s./ha, which was slightly above the anticipated maximal seasonal treatment rate for pre-emergent use.
Two rotations were sown at plant-back intervals of 30 and 134 days. The study was concluded after two rotations due to the very low TRRs (< 0.01 mg/kg) in all of the collected matrices.
The TRR values of all samples were very low and ranged from values < LOQ in turnip leaves and roots and wheat forage of the 2nd rotation to 0.009 mg/kg in wheat straw of the 1st rotation. They showed a decrease from the 1st to the 2nd rotation.
No extraction of plant matrices was conducted during this study; the radioactivity in all samples was determined exclusively by combustion followed by LSC.
As this study is not required for this assessment then this is provided for information only.
No further data are required at the moment.

7.3.2.3 Nature of residues in processed commodities (KCA 6.5.1)

Available data
Studies investigating the nature of deltamethrin residues in processed commodities were reported and evaluated in the peer review (Sweden, 2002). A summary of the nature of the residues in processed commodities is presented in Table 7.3-5.
No new data is submitted in the framework of this application.

Table 7.3-5: Nature of the residues in processed commodities

Conditions (Duration, Temperature, pH)	Identified compound(s) (%)	Reference
EU data		
Pasteurisation (20 minutes, 90°C, pH 4)	-Parent (94-96), - 3-phenoxybenzaldehyde (1-2)	Maurer, T., (2001), M-204204-01-1; EFSA, 2015
Baking, boiling, brewing (60 minutes, 100°C, pH 5)	-Parent (91-97), - 3-phenoxybenzaldehyde (3-5)	
Sterilisation (20 minutes, 120°C, pH 6)	-Parent (21-30) -3-phenoxybenzaldehyde (59-75) -(l*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethyl-cyclopropanecarboxylic acid (Br_2CA) (39-47)	

Conclusion on nature of residues in processed commodities
Three different hydrolysis conditions were applied simulating pasteurisation; brewing / baking / boiling and sterilisation. The sterilisation condition was carried out with two different label positions, [^{14}C]-benzyl deltamethrin and [^{14}C]-gemdimethyl deltamethrin. Analysis was performed with LSC and HPLC. The results of the study showed that under simulated pasteurisation (90°C, pH 4, 20 min.), brewing, baking and boiling (100°C, pH 5, 60 min.), deltamethrin is stable. Results of the sterilisation process (120°C, pH 6, 20 min) showed that deltamethrin was degraded under these conditions mainly to two metabolites; 3-phenoxybenzaldehyde and (l*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethyl-cyclopropanecarboxylic acid (Br_2CA).

7.3.2.4 Conclusion on the nature of residues in commodities of plant origin (KCA 6.7.1)

Table 7.3-6: Summary of the nature of residues in commodities of plant origin

Endpoints	
Plant groups covered	Fruit crops (tomato, apple), cereal (maize/corn), pulses and oilseeds (cotton)
Rotational crops covered	Yes
Metabolism in rotational crops similar to metabolism in primary crops?	Yes
Processed commodities	a.s. is stable under pasteurisation and baking/boiling /brewing conditions and not stable under sterilisation conditions
Residue pattern in processed commodities similar to pattern in raw commodities?	Yes
Plant residue definition for monitoring	Parent cis-deltamethrin (tentative, EFSA, 2015 ; Regulation EU 2016/1822)
Plant residue definition for risk assessment	Based on Regulation EU 2016/1822, the sum of *cis*-deltamethrin (paren)t and its isomers *alpha*-R-deltamethrin and *trans*-deltamethrin
Conversion factor from enforcement to RA	1.25 (except asparagus: 1) (EFSA, 2015) Not relevant in the framework of this application for oilseed rape.

Evaluator comment:
Information given by the Applicant is sufficient.
Excerpt from EFSA Journal 2015;13(11):4309:
"The effect of processing on the nature of deltamethrin was investigated in the framework of the peer review (Sweden, 2002). Studies were conducted simulating representative hydrolytic conditions for pasteurisation (20 minutes at 90°C, pH 4), boiling/brewing/baking (60 minutes at 100°C, pH 5) and sterilisation (20 minutes at 120°C, pH 6). Under simulated pasteurisation and brewing, baking and boiling, deltamethrin is stable and only small quantities (0.9 to 5.2% of applied radioactivity) of the plant metabolite 3-phenoxybenzylaldehyde (mPB aldehyde) were detected. Following sterilisation process, deltamethrin represented 21-48% of the applied radioactivity (AR) and it was degraded mainly into two metabolites: mPB aldehyde (59-75% of the AR) and (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethyl-cyclopropanecarboxylic acid (Br2CA) (39-47% of AR). During the peer review these two substances were considered well known plant metabolites with no toxicological relevance. Although their toxicological relevance may be reconsidered during the renewal of the approval of the active substance, the conclusions derived during the initial peer review were applied in the framework of this MRL review. It is also noted that in the hydrolysis studies, residues were always reported as deltamethrin and it is not clear if the analytical method used analysed for the sum of all isomers."

No further data are required at the moment.

7.3.2.5 Nature of residues in livestock (KCA 6.2.2-6.2.5)

Available data
No new data submitted in the framework of this application.

Table 7.3-7: Summary of animal metabolism studies

Group	Species	Label position	No of animal	Application details		Sample details		Reference
				Rate (mg/kg bw/d)	Duration (days)	Commodity	Time of sampling	
EU data								
Laying poultry	Hen	[gem-dimethyl-^{14}C]- and [^{14}C-benzyl]-deltamethrin	3 groups	5	3	Eggs	daily	[redacted] (1985), M-116708-01-1, FAO, 2002; EFSA, 2015
						Excreta	daily	
						Tissues	at sacrifice	
Lactating ruminants	Cow	[gem-dimethyl-^{14}C]- and [^{14}C-benzyl]-deltamethrin	2	10	3	Milk	twice daily	[redacted]. (1986),M-115057-01-1, FAO, 2002; EFSA, 2015
						Urine and faeces	daily	
						Tissues	at sacrifice	

Summary of animal metabolism studies reported in the EU

As stated in EFSA, 2015:

Metabolism of deltamethrin in poultry was investigated in the framework of Directive 91/414/EEC (Sweden, 1998, 2002). Hens were dosed for three consecutive days with 5 mg/kg bw per day (also labelled on both rings). In poultry tissues and eggs, deltamethrin was the main compound found (19-65% of the TRR), except in kidney where, apart for deltamethrin (25-28% TRR), metabolites c-Br2CA and c/t-COOH-c-Br2CA (together 22% TRR), and c-CH2OH-c-Br2CA and t-COOH-c-CH2OH-c-Br2CA-lactone (together 15-22% TRR) were also identified."

"Metabolism of deltamethrin in ruminants was investigated in the framework of Directive 91/414/EEC (Sweden, 1998, 2002). Cows were dosed for three consecutive days with 10 mg/kg body weight (bw) per day (labelled on both the benzyl- and dimethyl-rings, one cow for each label). Deltamethrin was the major compound in all tissues accounting for up to 90% of the TRR in fat. Metabolites Br_2CA and PB acid were present at the same level as parent in liver and kidney (23 and 33% of the TRR, respectively).

Conclusion on metabolism in livestock

Sufficient data have been provided to acknowledge the metabolism of deltamethrin in animal commodities.

As stated in EFSA, 2015:

"The metabolism of deltamethrin was investigated in cows and laying hens. As metabolic pathways are expected to be similar in ruminants and rodents, the results of the cow metabolism study could be extrapolated to pigs. From these studies it was proposed to consider, on a tentative basis, deltamethrin as a sufficient marker for monitoring in livestock commodities."

7.3.2.6 Conclusion on the nature of residues in commodities of animal origin (KCA 6.7.1)

Table 7.3-8: Summary on the nature of residues in commodities of animal origin

	Endpoints
Animals covered	Lactating cow
	Laying hens
Time needed to reach a plateau concentration	Not stated (peak after 3 days)
	2 days in eggs
Animal residue definition for monitoring	Parent cis-deltamethrin (tentative, EFSA, 2015)

Animal residue definition for risk assessment	Based on Regulation EU 2016/1822, the sum of *cis*-deltamethrin parent and its isomers *alpha*-R-deltamethrin and *trans*-deltamethrin
Conversion factor	Data do not allow to derive CFs
Metabolism in rat and ruminant similar	Yes (EFSA, 2015)
Fat soluble residue	Yes (EFSA, 2015)

Evaluator comment:
Information given by the Applicant is sufficient.
Excerpt from EFSA Journal 2015;13(11):4309:
EFSA proposes on a tentative basis, to define the residue for enforcement as deltamethrin only. For risk assessment, the residue definition is proposed as the sum of deltamethrin and its trans-isomer and alpha R-isomer. As metabolic pathways are expected to be similar in ruminants and rodents, the results of the cow metabolism study could be extrapolated to pigs.
No further data are required at the moment.

7.3.3 Magnitude of residues in plants (KCA 6.3)

7.3.3.1 Summary of European data and new data supporting the intended uses

New studies measuring the 3 isomers of deltamethrin according to the new residue definition for risk assessment, have been submitted by the applicant in the framework of this application. These studies are summarized in the Table below. The detailed assessment of these studies is presented in Appendix 2.

Table 7.3-9: Summary of EU reported and new data supporting the intended uses of DLT+FPF EC 85 and conformity to existing MRL

Commodity	Source	Residue zone (N-EU, S-EU, EU, outside EU)	Evaluation GAP Residue levels (mg/kg) E = according to enforcement residue definition RA = according to risk assessment residue definition	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg) *	MRL compliance
Oil seed rape	EFSA 2015	N-EU	GAP on which EU a.s. assessment is based: 4x7.5 g/ha, interval 14 d, PHI 45 d E: <0.01; <0.05; <0.05; <0.05; <0.07; <0.07 RA: -	E: 0.05	E: 0.07	0.16	0.2	
	New trials (15-2132, 16-2044)	N-EU	Tested GAP (cGAP for EU): 2x7.5 g/ha, interval 14 d, PHI 45 d E: 8x<0.01 RA: 8x(3x<0.01)	E: 0.01 RA: 0.01	E: 0.01 RA: 0.01		0.2	Yes

* Source of EU MRL: Reg.(EU) 2018/687 adoption of the Codex MRL for rapeseed

7.3.3.2 Conclusion on the magnitude of residues in plants

The GAPs supported for Deltamethrin on oilseed rape during the EU review of deltamethrin are more critical (4 applications versus 2 applications) and cover the intended use pattern on oilseed rape for the product Deltamethrin+Flupyradifurone EC 85 presented in this dossier. Thus these trials are sufficient to conclude that the intended use with the product Deltamethrin+Flupyradifurone EC 85 in oilseed rape does not result in residues of deltamethrin in the Raw Agricultural Commodities. Thus no supplementary data are deemed necessary. According to the intended use, no deltamethrin-related residue will be detected in mature seeds (<0.01 mg/kg for each isomer). As a consequence an STMR value of 0.01 mg/kg and a HR value of 0.01 mg/kg should be proposed as input values to perform the dietary risk assessment.

Evaluator comment:
Oilseed rape is the major crop in Northern Europe (Technical Guidelines SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

White and black mustard is a minor crop in the Northern zone of Europe. Therefore, 4 NEU trials are therefore required to support the use of Deltamethrin + Flupyradifurone EC 85 on mustard in NEU.

In accordance with Technical Guidelines SANTE/2019/12752 eight residue trials on rapeseeds/canola seeds (0401060) for foliar treatments before and after forming of the edible part can be extrapolated to support mustard seeds (0401080). Therefore the data cover the use on white and black mustard.

The proposed GAP of this dRR submission for the central zone is as follows:
Crop: oilseed rape
(1) 2 x 5 g DLT/ha, interval 14 d, BBCH 65-79, PHI 45 d;
(2) 2 x 7.5 g DLT/ha, interval 14 d, BBCH 30-49, no PHI,
(3) 2 x 7.5 g DLT/ha, interval 14 d, BBCH 50-59, no PHI.
Crop: mustard seed
(1) 2 x 7.5 g DLT/ha, interval 14 d, BBCH 50-59, no PHI.
DLT - deltamethrin

The Applicant submitted eight residue trials on oilseed rape, which were conducted in northern Europe in 2015 and 2016. Two applications were made at an interval of 13-14 days, each at a rate of 0.75 L/ha, corresponding to 7.5 g a.s./ha. The last application was done at a crop growth stage of BBCH 73-80 (30% to 70% of pods have reached final size). The envisaged PHI was 45 days.
Residues of deltamethrin and its isomers AE F108569 and AE 0035073 were determined by LC-MS/MS according to method 00855/M004.
The individual LOQs for deltamethrin and its isomers are 0.05 mg/kg in green material and straw and 0.01 mg/kg in seed.
No deltamethrin-related residue will be detected in mature seeds (<0.01 mg/kg for each isomer).
Residue results (mg/kg) in seed:
E: 8x<0.01
RA: 8x<0.01

Available results show that the in force MRLs of deltamethrin on oilseed rape of 0.2 mg/kg and on mustard seeds of 0.07 mg/kg (Reg. (EU) 2018/832) will not be exceeded. The current EU MRLs for deltamthrin are sufficient to support the proposed uses.
No additional data are required.

7.3.4 Magnitude of residues in livestock

7.3.4.1 Dietary burden calculation

The supported use in this dossier is less critical than the one reviewed in the frame of the Article 12 review (Article 12 of regulation 396/2005) and leads to a no residue situation for all the 3 isomers. Consequently no change in the transfer of Deltamethrin and its isomers is anticipated in animal matrices. Therefore the currently established MRLs don’t need to be modified.

Table 7.3-10: Input values for the dietary burden calculation (considering the uses reviewed in Art. 12 procedure and the use under consideration)

Feed Commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Risk assessment residue definition *Cis*-deltamethrin + *Trans*-isomer + Alpha-R-isomer (EFSA RO 2015)				
Alfalfa (fresh and silage)	0.11	STMRMo × CF (EFSA 2015)	0.23	HRMo × CF (EFSA 2015)
Clover (fresh and silage)	0.11	STMRMo × CF (EFSA 2015)	0.23	HRMo × CF (EFSA 2015)
Grass (fresh and silage)	0.05	STMRMo × CF (EFSA 2015)	0.08	HRMo × CF (EFSA 2015)
Cabbage	0.03	STMRMo × CF (EFSA 2015)	0.08	HRMo × CF (EFSA 2015)
Sugar beet leaves	0.03	STMRMo × CF (EFSA 2015)	0.03	HRMo × CF (EFSA 2015)
Fodder beet leaves	0.03	STMRMo × CF (EFSA 2015)	0.03	HRMo × CF (EFSA 2015)
Citrus pomace	0.03	STMRMo × CF × 2.5 (EFSA 2015)	0.03	STMRMo × CF × 2.5(EFSA 2015)
Apple pomace	0.21	STMRMo × CF × 2.5 (EFSA 2015)	0.21	STMRMo × CF × 2.5(EFSA 2015)
Alfalfa hay	0.43	STMRMo × CF × 4 (EFSA 2015)	0.90	HRMoxCFx4 (EFSA 2015)
Clover hay	0.43	STMRMo × CF × 4 (EFSA 2015)	0.90	HRMoxCFx4(EFSA 2015)
Grass hay	0.20	STMRMo × CF × 4 (EFSA 2015)	0.30	HRMoxCFx4(EFSA 2015)
Cereal grain	0.45	STMRMo × CF (EFSA 2015)	045	STMRMo × CF (EFSA 2015)
Cereal bran	5.00	STMRMo × CF (EFSA 2015)	5.00	STMRMo × CF × 8(EFSA 2015)
Wheat and rye straw	0.32	STMRMo × CF (EFSA 2015)	0.51	HRMo × CF (EFSA 2015)
Barley and oat straw	0.23	STMRMo × CF (EFSA 2015)	0.59	HRMo × CF (EFSA 2015)
Pulses	0.25	STMRMo × CF (EFSA 2015)	0.33	HRMo × CF (EFSA 2015)
Potatoes	0.09	STMRMo × CF (EFSA 2015)	0.14	HRMo × CF (EFSA 2015)
Turnips	0.03	STMRMo × CF (EFSA 2015)	0.03	HRMo × CF (EFSA 2015)
Swedes	0.03	STMRMo × CF (EFSA 2015)	0.03	HRMo × CF (EFSA 2015)

Feed Commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Sugar beet	0.03	STMRMo × CF (EFSA 2015)	0.03	HRMo × CF (EFSA 2015)
Fodder beet	0.03	STMRMo × CF (EFSA 2015)	0.03	HRMo × CF (EFSA 2015)
Rape seed meal	0.01	STMR	0.01	STMR
Cotton seed meal	0.01	STMRMo × CF (EFSA 2015)	0.01	STMRMo × CF (EFSA 2015)
Linseed meal	0.03	STMRMo × CF (EFSA 2015)	0.03	STMRMo × CF (EFSA 2015)
Sunflower seed meal	0.13	STMRMo × CF × PF (EFSA 2015)	0.13	STMRMo × CF × PF (EFSA 2015)

* Indicates that the input value is proposed at the limit of quantification.
STMRMo: supervised trials median residue according to the monitoring residue definition
HRMo: highest residue according to the monitoring residue definition
CF: conversion factor for risk assessment
PF: processing factor
(a): For cotton, rapeseed and linseed meals no default processing factor was applied because residues are expected to be below the LOQ in the raw commodities. Concentration of residues in these commodities is therefore not expected (considering fat solubility of the compound).
(b): For fruit pomace, forage hay and cereal bran, in the absence of processing factors supported by data, default processing factors of 2.5, 4 and 8 were respectively included in the calculation in order to consider the potential concentration of

Table 7.3-11: Results of the dietary burden calculation

Animal species	Median dietary burden (mg/kg bw/d)	Maximum dietary burden (mg/kg bw/d)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)
Risk assessment residue definition *Cis*-deltamethrin + *Trans*-isomer + Alpha-R-isomer (EFSA RO 2015)					
Beef cattle*	0.0123	0.016	Potato culls	0.67	Y
Dairy cattle*	0.0179	0.026	Clover forage	0.67	Y
Ram/ewe	0.0155	0.025	Clover forage	0.76	Y
Lamb	0.0207	0.027	Potato culls	0.64	Y
Breeding swine	0.012	0.015	Clover forage	0.66	Y
Finishing swine*	0.015	0.018	Potato culls	0.61	Y
Broiler poultry	0.030	0.032	Potato culls	0.45	Y
Layer poultry*	0.035	0.038	Clover forage	0.56	Y
Turkey	0.030	0.034	Potato culls	0.48	Y

* These categories correspond to those (formerly) assessed at EU level.

Evaluator comment:
The dietary burden has been calculated with the EFSA Animal Model 2017. Since crops treated with deltamethrin might be fed to livestock, the median and maximum dietary burdens were calculated for different groups of livestock using the agreed European methodology. The calculated dietary burdens for all groups of livestock were found to exceed the trigger value of 0.1 mg/kg DM. Further investigation of residues is therefore required in all commodities of animal origin.

7.3.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

Available data
Poultry:
In EFSA's recent Reasoned Opinion (EFSA Journal 2015;13(11):4309) on the review of the existing maximum residue levels (MRLs), it is stated :
For poultry, although the maximum dietary burden exceeded the threshold of 0.1 mg/kg DM, no residues above the LOQ were expected in poultry matrices at the calculated dietary burden and no feeding study was triggered. Therefore, MRLs can be established at the LOQ in all poultry commodities and no default conversion factors for risk assessment need to be derived.

Ruminant:
No new data were submitted in the framework of this application.

Table 7.3-12: Overview of the values derived from livestock feeding studies

Commodity	Dietary burden		Results of the livestock feeding study						Median residue (mg/kg)[b]	Highest residue (mg/kg)[c]	Calculated MRL (mg/kg)	CF for RA[d]
	Med. (mg/kg bw/d)	Max. (mg/kg bw/d)	Dose Level (mg/kg bw/d)	No	Result for enforcement		Result for RA					
					Mean (mg/kg)	Max. (mg/kg)	Mean (mg/kg)	Max. (mg/kg)				
EU data (RMS, 2010; EFSA, 2015)												
Enforcement residue definition Cis-deltamethrin												
Ruminant meat	0.0179	0.026	1.3	3	0.01*	0.01*	0.01*	0.01*	0.01*	0.01*	0.01	1
			3.3	3	0.01*	0.01*	0.01*	0.01*				
			9.9	3	0.01*	0.01	0.01*	0.01				
Ruminant fat	0.0179	0.026	1.3	3	0.023	0.04	0.023	0.04	0.012	0.029	0.03	1
			3.3	3	0.016	0.02	0.016	0.02				
			9.9	3	0.02	0.03	0.02	0.03				
Ruminant liver	0.0179	0.026	1.3	3	0.01*	0.01*	0.01*	0.01*	0.01*	0.01*	0.01	1
			3.3	3	0.01*	0.01*	0.01*	0.01*				
			9.9	3	0.01*	0.01*	0.01*	0.01*				
Ruminant kidney	0.0179	0.026	1.3	3	0.01*	0.01*	0.01*	0.01*	0.01*	0.01*	0.01	1
			3.3	3	0.01*	0.01*	0.01*	0.01*				
			9.9	3	0.01*	0.01*	0.01*	0.01*				
Poultry meat	0.035	0.038	0.100	4	0.02*	0.02*	0.02*	0.02*	0.02*	0.02*	0.02	1
			0.270	4	0.02*	0.02*	0.02*	0.02*				
			0.950	4	0.02*	0.02*	0.02*	0.02*				
Poultry fat	0.035	0.038	0.100	4	0.05*	0.05*	0.05*	0.05*	0.05	0.084	0.09	1
			0.270	4	0.146	0.285	0.146	0.285				
			0.950	4	0.421	0.533	0.421	0.533				
Poultry liver	0.035	0.038	0.100	4	0.02*	0.02*	0.02*	0.02*	0.02*	0.02*	0.02	1
			0.270	4	0.02*	0.02*	0.02*	0.02*				
			0.950	4	0.02*	0.02*	0.02*	0.02*				

Milk	0.0179	0.026	1.3	3	0.013	0.02	0.013	0.02	0.007	0.01	0.01	1
			3.3	3	0.01	0.01	0.01	0.01				
			9.9	3	0.023	0.03	0.023	0.03				
Eggs	0.035	0.038	0.100	4	0.015*	0.015*	0.015*	0.015*	0.011	0.015	0.015	1
			0.270	4	0.015*	0.015*	0.015*	0.015*				
			0.950	4	0.034	0.037	0.034	0.037				

N/A: Not applicable – only the mean values are considered for calculating MRLs in milk.
n.r.: Not reported
(*): Indicates that the MRL is set at the limit of analytical quantification.
(F): MRL is expressed as mg/kg of fat contained in the whole product.
(b): Median residue value according to the enforcement residue definition, derived by interpolation/extrapolation from the feeding study for the median dietary burden (FAO, 2009).
(c): Highest residue value (tissues, eggs) or mean residue value (milk) according to the enforcement residue definition, derived by interpolation/extrapolation of the maximum dietary burden between the relevant feeding groups of the study (FAO, 2009).
(d): The median conversion factor for enforcement to risk assessment.

Conclusion on feeding studies
The supported use on oilseed rape of Deltamethrin + Flupyradifurone EC 85, being less critical than the one reviewed by EFSA in the frame of Article 12 and leading to a no residue situation for the 3 isomers of deltamethrin. As a consequence a modification of the existing MRLs is not needed in animal products.

Evaluator comment:
Information given by the Applicant is sufficient.
Excerpt from EFSA Journal 2015;13(11):4309:
Deltamethrin is authorised for use on several crops that might be fed to livestock and the dietary burden calculations exceeded the trigger values for all groups of livestock. The metabolism of deltamethrin was investigated in cows and laying hens. As metabolic pathways are expected to be similar in ruminants and rodents, the results of the cow metabolism study could be extrapolated to pigs. From these studies it was proposed to consider, on a tentative basis, deltamethrin as a sufficient marker for monitoring in livestock commodities. A validated analytical method for enforcement of the proposed residue definition is available but not fully validated. For risk assessment, the residue definition was tentatively proposed as the sum of deltamethrin and its trans-isomer and alpha R-isomer. Available livestock feeding studies were sufficient only for deriving tentative MRLs in all relevant tissues of ruminants, pigs (except for kidney) and hens, as well as milk and eggs.

The feeding data available show that no exceedance of the MRL for animal commodities (Reg. (EU) 2018/832) will occur and the intended uses are considered acceptable.
No further data are required at the moment.

7.3.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation) (KCA 6.5.2-6.5.3)

7.3.5.1 Available data for all crops under consideration

No new data were submitted in the framework of this application.
Since the mature seeds are free of residues (<0.01 mg/kg), no processing study is required. Nevertheless for comprehensiveness sake all existing processing studies are mentioned hereafter - however not summarized in Appendix 2 as not relevant for this dRR.
The following table summarises all the existing processing studies that were already reviewed by EFSA in the frame of the Article 12 review of the MRLs and new data that were submitted in the frame of the renewal process and reviewed by the UK rapporteur Member State.

Table 7.3-13: Overview of the available processing studies

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
EU data					
Cis-deltamethrin					
Potatoes, unpeeled and boiled	4	0.26			EFSA 2015
Potatoes, fried	4	0.04			EFSA 2015
Apples, juice	1	0.01			EFSA 2015
Apples, wet pomace	1	5.70			EFSA 2015
Strawberries, canned	1	0.65			EFSA 2015
Tomatoes, paste	1	0.40			EFSA 2015
Tomatoes, juice	1	1.00			EFSA 2015
Tomatoes, ketchup	1	1.00			EFSA 2015
Dry pulses, cooked	1	0.10			EFSA 2015
Sunflower seed, crude oil	1	10			EFSA 2015
Rape seed, crude oil	1	10			EFSA 2015
Cotton seed, crude oil	1	0.02			EFSA 2015

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
Olives, crude oil after warm press	1	1.60			EFSA 2015
Barley, beer	1	0.02			EFSA 2015
Maize, crude oil	1	20			EFSA 2015
Rice, polished	1	0.2			EFSA 2015

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
New data submitted during renewal process					
Cis-deltamethrin					
Wheat flour (type 55)	1	0.27			**Billian, P.; Reineke, A.; 2010**, M-363957-01-1, 08-3214
White flour bran	1	3.3			
White bread	1	0.06			
wholemeal	1	1.2			
Wholemeal bread	1	0.41			
Germ	1	0.81			
Semolina	1	0.13			
Semolina bran	1	0.69			
Pasta, fresh	1	0.09			
Pasta, cooked	1	0.03			
Pasta, dry	1	0.05			
Pasta, dried and cooked	1	0.03			
Brewer's malt	1	0.5			**Billian, P.; Reineke, A.; 2009**, M-361937-01-1, 08-3215
Brewer's grain	1	0.2			
Beer	1	0.03*			
Pearl barley	1	0.03			

* The median processing factor is obtained by calculating the median of the individual processing factors of each processing study.

** The median conversion factor for enforcement to risk assessment is obtained by calculating the median of the individual conversion factors of each processing study.

7.3.5.2 Conclusion on processing studies

The supported use of Deltamethrin +Flupyradifurone EC 85 on oilseed rape represents a situation of no residue in mature seeds. Consequently no concentration is expected in subsequent processing fractions.

Evaluator comment:
Information given by the Applicant is sufficient. No additional data are required.

7.3.6 Magnitude of residues in representative succeeding crops

The crop under consideration can be grown in rotation.

Considering available data dealing with nature of residues (see A 2.2.2.2), no study dealing with magnitude of residues in succeeding crops is needed.
Deltamethrin showed a relatively fast to moderate dissipation from soil under field conditions (field DT_{50} = 8 – 28 days), even after multiple application of exaggerated rates. The respective DT_{90} was calculated to be in a range of 25-94 days. The main soil metabolite Br_2CA was not detected above the limit of quantification (LOQ = 0.01 mg/kg soil) under field conditions. These field dissipation studies demonstrated that deltamethrin and its metabolites are fully degradable under field conditions.
Furthermore, deltamethrin shows a very high adsorption to soil particles with an average Koc of 10 240 000 mL/g. Deltamethrin is not systemic in plants and was not translocated from the application site to other plant parts. Therefore, by considering these results together with the very low uptake of deltamethrin derived soil residues by plants as shown in confined rotational crop studies, the presence of residues in succeeding crops under field conditions is unlikely.

Conclusion on rotational crops studies
No field rotational crop study is considered necessary.

Evaluator comment:
Information given by the Applicant is sufficient.
Excerpt from EFSA Journal 2015;13(11):4309:
"*Most of the crops under consideration may be grown in rotation. According to the soil degradation studies evaluated in the framework of the peer review, period required for 90 percent dissipation (DT_{90}) value of deltamethrin is expected to range between 30 and 390 days which is higher than the trigger value of 100 days (European Commission, 2002). According to the European guidelines on rotational crops (European Commission, 1997b), further investigation of residues in rotational crops is relevant.*

The results of the confined rotational crop study is confirmed by a field rotational crop study analysing residues in spinach, carrots and radishes planted in soil treated once at 0.12 kg a.s./ha. Considering that deltamethrin was applied to a bare soil (interception of active substance by the plants is expected in practice), it can be concluded that residue levels in rotational commodities are not expected to exceed 0.01 mg/kg provided that deltamethrin is used according to the GAPs assessed in the present review."

No waiting periods beyond normal agricultural practice are proposed for succeeding crops to be planted.
No additional data are required.

7.3.7 Other / special studies (KCA6.10, 6.10.1)

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of Deltamethrin + Flupyradifurone EC 85. Therefore, other special studies are not needed.

Evaluator comment:
There is no sufficient data to support application of DLT+FPF EC 85 at flowering oilseed rape. Following the review done by zRMS, the applicant would like to request to restrict the use of the product to applications outside of flowering. The table GAP has been corrected.

7.3.8 Estimation of exposure through diet and other means (KCA 6.9)

Toxicological reference values relevant for dietary risk assessment are reported in the summary of the evaluation (see 7.1.2).

7.3.8.1 Input values for the consumer risk assessment

In its reasoned opinion EFSA has proposed to change the residue definition for risk assessment adding to the parent compound *cis*-deltamethrin its 2 isomers, *trans*-isomer and alpha-R-isomer. Consequently EFSA has performed the consumer chronic and acute risk assessment applying a conservative conversion factor of 1.25 to STMR and HR values. Residues arising directly from the intended use in oilseed rape are considered. The conversion factor of 1.25 was not apllied as the residue trials were analysed according to the new residue definition for risk assessment, *cis*-deltamethrin, *trans*-deltamethrin and *alpha*-R-deltamethrin. Since the supported use leads to a no residue situation for every compound of the residue definition, the STMR and the HR values were considered to be at the LOQ of 0.01 mg/kg.

An ADI of 0.01 mg/kg bw/d and an ARfD of 0.01 mg/kg bw were used as toxicological reference values (EFSA, 2015).
For potatoes a processing factor (PF = 0.26 for unpeeled, boiled potatoes) was also applied.

The input values used for the dietary exposure calculation are summarized below.

Table 7.3-14: Input values for the consumer risk assessment

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Cis-deltamethrin+trans-deltamethrin+*alpha*-R-deltamethrin				
Citrus fruit	0.01	STMRxCF (EFSA, 2015)	-	Acute risk assessment undertaken only with regard to crops under consideration
Tree nuts	0.03	STMRxCF (EFSA, 2015)	-	
Pome fruits	0.04	STMRxCF (EFSA, 2015)	-	
Apricots	0.03	STMRxCF (EFSA, 2015)	-	
Peaches	0.03	STMRxCF (EFSA, 2015)	-	
Cherries	0.04	STMRxCF (EFSA, 2015)	-	
Plums	0.01	STMRxCF (EFSA, 2015)	-	
Cane fruit	0.03	STMRxCF (EFSA, 2015)	-	
Other small fruits and berries	0.10	STMRxCF (EFSA, 2015)	-	
Table and wine grapes	0.08	STMRxCF (EFSA, 2015)	-	
Strawberries	0.03	STMRxCF (EFSA, 2015)	-	
Table olives	0.26	STMRxCF (EFSA, 2015)		
Kiwi	0.03	STMRxCF (EFSA, 2015)	-	
Potatoes	0.02	STMRxCFxPF (EFSA, 2015)	-	
Other root and tuber vegetables	0.03	STMRxCF (EFSA, 2015)	-	
Garlic	0.03	STMRxCF (EFSA, 2015)	-	
Onions	0.03	STMRxCF (EFSA, 2015)	-	
Shallots	0.03	STMRxCF (EFSA, 2015)	-	
Spring onions	0.07	STMRxCF (EFSA, 2015)	-	
Tomatoes	0.03	STMRxCF (EFSA, 2015)	-	
Peppers	0.04	STMRxCF (EFSA, 2015)	-	
Aubergines (egg plants)	0.07	STMRxCF (EFSA, 2015)	-	
Cucurbits edible peel	0.03	STMRxCF (EFSA, 2015)	-	
Cucurbits inedible peel	0.03	STMRxCF (EFSA, 2015)	-	
Sweet corn	0.03	STMRxCF (EFSA, 2015)	-	
Flowering brassica	0.03	STMRxCF (EFSA, 2015)	-	
Brussels sprouts	0.01	STMRxCF (EFSA, 2015)	-	
Head cabbage	0.03	STMRxCF (EFSA, 2015)	-	
Chinese cabbage	0.02	STMRxCF (EFSA, 2015)	-	
Kale	0.05	STMRxCF (EFSA, 2018)	-	
Kohlrabi	0.01	STMRxCF (EFSA, 2015)	-	

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Lamb's lettuce	0.43	STMRxCF (EFSA, 2015)	-	
Lettuce	0.19	STMRxCF (EFSA, 2015)	-	
Scarole	0.04	STMRxCF (EFSA, 2015)	-	
Cress	0.43	STMRxCF (EFSA, 2015)	-	
Land cress	0.43	STMRxCF (EFSA, 2015)	-	
Rocket rucola	0.43	STMRxCF (EFSA, 2015)	-	
Red mustard	0.43	STMRxCF (EFSA, 2015)	-	
Leaves and sprouts of Brassica spp	0.33	STMRxCF (EFSA, 2015)	-	
Spinach and similar leaves	0.43	STMRxCF (EFSA, 2015)	-	
Witloof	0.03	STMRxCF (EFSA, 2015)	-	
Herbs	0.43	STMRxCF (EFSA, 2015)	-	
Beans (fresh, with pods)	0.02	STMRxCF (EFSA, 2015)	-	
Beans (fresh, without pods)	0.03	STMRxCF (EFSA, 2015)	-	
Peas (fresh, with pods)	0.02	STMRxCF (EFSA, 2015)	-	
Peas (fresh, without pods)	0.02	STMRxCF (EFSA, 2015)	-	
Lentils (fresh)	0.02	STMRxCF (EFSA, 2015)	-	
Asparagus	0.02	STMRxCF (EFSA, 2015)	-	
Celery	0.08	STMRxCF (EFSA, 2017)	-	
Florence fennel	0.08	STMRxCF (EFSA, 2017)	-	
Rhubarb	0.08	STMRxCF (EFSA, 2017)	-	
Globe artichokes	0.07	STMRxCF (EFSA, 2015)	-	
Leek	0.07	STMRxCF (EFSA, 2015)	-	
Cultivated fungi	0.03	STMRxCF (EFSA, 2015)	-	
Pulses	0.25	STMRxCF (EFSA, 2015)	-	
Linseed	0.03	STMRxCF (EFSA, 2015)	-	
Poppy seed	0.06	STMRxCF (EFSA, 2015)	-	
Sesame seed	0.01	STMRxCF (EFSA, 2015)	-	
Sunflower seed	0.06	STMRxCF (EFSA, 2015)	-	
Rape seed	0.01	STMR	0.01	HR
Mustard seed	0.01	STMR (based on rape seed extrapolation)	0.01	HR (based on rape seed extrapolation)
Cotton seed	0.01	STMRxCF (EFSA, 2015)		
Pumpkin seed	0.01	STMRxCF (EFSA, 2015)	-	
Safflower	0.01	STMRxCF (EFSA, 2015)	-	

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Borage	0.06	STMRxCF (EFSA, 2015)	-	
Gold of pleasure	0.06	STMRxCF (EFSA, 2015)	-	
Hempseed	0.06	STMRxCF (EFSA, 2015)	-	
Castor bean	0.06	STMRxCF (EFSA, 2015)	-	
Olives for oil production	0.26	STMRxCF (EFSA, 2015)	-	
Barley grain	0.56	STMRxCF (EFSA, 2015)	-	
Buckwheat grain	0.63	STMRxCF (EFSA, 2015)	-	
Maize grain	0.56	STMRxCF (EFSA, 2015)	-	
Millet grain	0.63	STMRxCF (EFSA, 2015)	-	
Oats grain	0.63	STMRxCF (EFSA, 2015)	-	
Rice grain	0.56	STMRxCF (EFSA, 2015)	-	
Rye grain	0.56	STMRxCF (EFSA, 2015)	-	
Sorghum grain	0.63	STMRxCF (EFSA, 2015)	-	
Wheat grain	0.03	STMRxCF (EFSA, 2015)	-	
Herbal infusions (dried flowers)	1.31	STMRxCF (EFSA, 2015)	-	
Herbal infusions (dried leaves)	1.31	STMRxCF (EFSA, 2015)	-	
Herbal infusions (dried roots)	0.09	STMRxCF (EFSA, 2015)	-	
Spices (seeds)	0.06	EUMRLxCF (EFSA, 2015)	-	
Spices (fruits and berries)	1.31	STMRxCF (EFSA, 2015)	-	
Spices (roots and rhizome)	0.09	STMRxCF (EFSA, 2015)	-	
Spices (buds)	1.31	STMRxCF (EFSA, 2015)	-	
Spices (flower stigma)	1.31	STMRxCF (EFSA, 2015)		
Sugar beet (root)	0.03	STMRxCF (EFSA, 2015)	-	
Chicory roots	0.01	STMRxCF (EFSA, 2015)	-	
Swine meat	0.03	$0.8 \times STMR_{muscle} + 0.2\ STMR_{fat}$ (EFSA, 2015)	-	
Swine fat (free of lean meat)	0.06	STMR (EFSA, 2015)	-	
Swine liver	0.02	STMR (EFSA, 2015)	-	
Swine kidney	0.03	EUMRL (EFSA, 2015)	-	
Ruminant meat	0.03	$0.8 \times STMR_{muscle} + 0.2\ STMR_{fat}$ (EFSA, 2015)	-	
Ruminant fat	0.08	STMR (EFSA, 2015)	-	
Ruminant liver	0.02	STMR (EFSA, 2015)	-	
Ruminant kidney	0.03	EUMRL (EFSA, 2015)	-	

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Poultry meat	0.02	$0.9 \times STMR_{muscle} + 0.1 STMR_{fat}$ (EFSA, 2015)	-	
Poultry fat	0.04	STMR (EFSA, 2015)	-	
Poultry liver	0.02	STMR (EFSA, 2015)	-	
Ruminant milk	0.02	STMR (EFSA, 2015)	-	
Birds'eggs	0.02	STMR (EFSA, 2015)	-	

CF: Conversion factor (tentative). For all crops except asparagus, a tentative CF of 1.25 has been proposed for risk assessment (EFSA, 2015)
EFSA, 2015: EFSA Journal 2015;13(11):4309 [104 pp.].Review of the existing maximum residue levels for deltamethrin according to Article 12 of Regulation (EC) No 396/2005.

7.3.8.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented in Appendix 3.

Table 7.3-15: Consumer risk assessment

TMDI (% ADI) according to EFSA PRIMo	Not conducted, please refer to IEDI
IEDI (% ADI) according to EFSA PRIMo Rev.3.0	89% (based on NL toddler)
IESTI (% ARfD) according to EFSA PRIMo Rev.3.0	Rape seed:0.1 % (based on children)

* include raw and processed commodities if both values are required for PRIMo
** if national model is available

The proposed uses of deltamethrin in the formulation DLT+FPF EC 85 do not represent unacceptable acute and chronic risks for the consumer.

Evaluator comment:
Information given by the Applicant is sufficient.
The calculation of the IEDI using EFSA model (version 3) and STMR values reported in EFSA Journal 2015;13(11):4309 and STMR values detected in the supervised field trials submitted in this registration report led to a utilisation of the ADI of 89% with the NL toddler being the population group with the highest value. For this diet, the highest contributor is maize/corn with 62% of the ADI. The intended uses will not result in a consumer chronic exposure exceeding the ADI.
An acute consumer risk assessment was performed based on the highest residues value (HR) for oilseed rape and based on rape seed extrapolation for mustard seed. The highest International Estimated Short-Term Intake (IESTI) is at 0.1% and 0.05% of the ARfD for the consumption of rapeseeds/canola by children and by adults respectively.
The proposed uses of deltamethrin in the product DLT+FPF EC 85 (Sivanto Energy) do not represent unacceptable acute and chronic risks for the consumer.
No further information are required to support the proposed uses.

7.4 Combined exposure and risk assessment

From a scientific point of view it is regarded necessary to take into account potential combination effects. However, the evaluation of cumulative or synergistic effects as requested by Art. 4 (3b) of Regulation (EC) No. 1107/2009 should only be performed when harmonised "scientific methods accepted by the Authority to assess such effects are available."
Currently, no EU-harmonized guidance is available on the risk assessment of combined exposure to multiple active substances; this approach is not mandatory at EU level.

Evaluator comment:
Information given by the Applicant is sufficient.

7.4.1 Acute consumer risk assessment from combined exposure

Please refer to rationale given under point 7.4.

7.4.2 Chronic consumer risk assessment from combined exposure

Please refer to rationale given under point 7.4

7.5 References

FAO 2002 Deltamethrin. In: Pesticide residues in food – 2002- Evaluations – Part I: Residues. Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 175/1, 2002.

Sweden, 1998. Draft assessment report on the active substance deltamethrin prepared by the rapporteur Member State Sweden in the framework of Council Directive 91/414/EEC, October 1998.

Sweden, 2002. Addendum to the draft assessment report on the active substance deltamethrin prepared by the rapporteur Member State Sweden in the framework of Council Directive 91/414/EEC, June 2002.

EFSA Journal 2015;13(11):4309 [104 pp.].Review of the existing maximum residue levels for deltamethrin according to Article 12 of Regulation (EC) No 396/2005.

EC (European Commission), 2011: Appendix D. Guidelines on comparability, extrapo-lation, group tolerances and data requirements for setting MRLs; SANCO 7525/VI/95-rev.10.3

Commission Regulation (EU) 2016/1902 of 27 October 2016 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for acetamiprid, ametoctradin, azoxystrobin, cyfluthrin, difluoroacetic acid, dimethomorph, fenpyrazamine, flonicamid, fluazinam, fludioxonil, flupyradifurone, flutriafol, fluxapyroxad, metconazole, proquinazid, prothioconazole, pyriproxyfen, spirodiclofen and trifloxystrobin in or on certain products

Commission Regulation (EU) 2016/486 of 29 March 2016 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for cyazofamid, cycloxydim, difluoroacetic acid, fenoxycarb, flumetralin, fluopicolide, flupyradifurone, fluxapyroxad, kresoxim-methyl, mandestrobin, mepanipyrim, metalaxyl-M, pendimethalin and tefluthrin in or on certain products

EFSA (European Food Safety Authority), 2015. Conclusion on the peer review of the pesticide risk assessment of the active substance flupyradifurone, EFSA Journal 2015; 13(2):4020 (doi: 10.2903/j.efsa.2015.4020)

EFSA (European Food Safety Authority), 2016. Reasoned Opinion on setting of new maximum residue levels for flupyradifurone in strawberries, blackberries and raspberries, EFSA Journal 2016;14(3):4423 (doi:10.2903/j.efsa.2016.4423)

The Netherlands, 2014: Draft assessment report on the active substance flupyradifurone prepared by the rapporteur Member State the Netherlands in the framework of Regulation (EC) No 1107/2009. February, 2014

Netherlands, 2015. Final addendum to the Draft Assessment Report on the active substance flupyradifurone prepared by the Rapporteur Member State The Netherlands in the framework of Regulation (EC) 1107/2009, February, 2015

Position Paper for Ctgb: Spiegel, K., 2016: Considerations of Bayer CropScience to define the application rates and the crops to be tested for additional field rotational crop studies; M-617654-01-1

Netherlands (Ctgb), 2018a: Evaluation Report - MRL application on the setting of MRLs in various rotational crops and animal commodities (confirmatory data), under evaluation (draft prepared by Bayer AG: March 26, 2018; M-618206-01-1; final document submitted by Ctgb to EFSA in February 2019 as combined document including the import tolerances)

Netherlands (Ctgb), 2018b: Evaluation Report - MRL application on the setting of flupyradifurone MRLs

and Import tolerances in various imported commodities, under evaluation (draft prepared by Bayer AG, June 25, 2018; updated July 9 and August 22, 2018; M-626821-04-1; final document submitted by Ctgb to EFSA in February 2019 as combined document including the confirmatory data)

Netherlands (Ctgb), 2019: MRL application on the setting of MRLs for flupyradifurone in oilseed rape (OSR), under evaluation (draft prepared by Bayer AG: January 31, updated March 19, 2019; M-650217-01-1)

OECD (Organisation for Economic Co-operation and Development), 2011: OECD MRL Calculator: spreadsheet for single data set and spreadsheet for multiple data set, 2 March 2011; In: Pesticide Publications/Publications on Pesticide Residues

OECD (Organisation for Economic Co-operation and Development), 2013: Guidance document on residues in livestock; Series on Pesticides No 73, 10 July 2013, 77 pp.; ENV/JM/MONO(2013)8

EFSA (European Food Safety Authority), 2015: Estimation of animal intakes and HR, STMR and MRL calculation for products of animal origin, September 2015

FAO (Food and Agriculture Organization of the United Nations), 2009. Submission and evaluation of pesticide residues data for the estimation of Maximum Residue Levels in food and feed. Pesticide Residues. 2nd Ed. FAO Plant Production and Protection Paper 197, 264 pp.

Appendix 1 Lists of data considered in support of the evaluation

List of data submitted by the applicant and relied on

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.1 / 01	Netzband, D.	2015	Storage stability of BYI 02960, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices Report No.: M-428412-03-1 Bayer CropScience LP, Stilwell, KS, USA **... amended: 2015-09-03** GLP/GEP: Yes unpublished	No	Bayer
KCA 6.1 / 02	Lakaschus, S.; Gizler, A.	2017	Amendment no. 3 to final report - 7 days freezer storage stability study with different combinations of a total of 61 analytes (parent and metabolite molecules) and five matrix types (high water / acidic / starch / protein / oil) Report No.: S13-03307, Edition Number: M-480441-06-1 Eurofins Agroscience Services Chem GmbH (EAS Chem), Hamburg, Germany **... amended: 2017-08-16** GLP/GEP: Yes unpublished	No	Bayer
KCA 6.1 / 03	Winter, O.; Amann, S.; Giesler, W.	2018	Storage stability of deltamethrin and flupyradifurone in oilseed rape (seed and straw) Report No.: S17-05312, Edition Number: M-626405-01-1 Eurofins Agroscience Services Chem GmbH, Hamburg, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.1 / 04 **... also filed:** **KCA 6.4.1 / 01**	[redacted]	2017	Difluoroacetic acid - Magnitude of the residue in laying hens Report No.: RARVN150, Edition Number: M-611007-01-1 [redacted] GLP/GEP: Yes unpublished	Yes	Bayer
KCA 6.1 / 05	Ballesteros, C.	2012	Storage stability of residues of deltamethrin (AE F032640) and its isomers AE F108569 and AE 0035073 in orange during deep freeze storage for up to 24 months Report No.: 09-07, Edition Number: M-441996-01-1 Bayer S.A.S., Bayer CropScience, Lyon, France GLP/GEP: Yes unpublished	No	Bayer

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3.1.1 / 01 **... also filed:** **KCP 5.1.2.5 / 02**	Schulte, G.	2017	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in France (North), Germany and Belgium Report No.: 15-2132, Edition Number: M-578527-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-05-03** GLP/GEP: Yes unpublished	No	Bayer
KCA 6.3.1.1 / 02 **... also filed:** **KCP 5.1.2.5 / 03**	Kaussmann, M.; Kerkering, S.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and northern France Report No.: 16-2044, Edition Number: M-641044-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.3.1.1 / 03	Miara, C.; Nayyar, B.; Kowalski, N.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Spain, Portugal, southern France and Italy Report No.: 16-2131, Edition Number: M-643446-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.3.1.1 / 04	Miara, C.; Nayyar, B.; Kowalski, N.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Spain and Portugal Report No.: 16-2169, Edition Number: M-645539-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.4.1 / 01 **... also filed:** **KCA 6.1 / 04**	[redacted]	2017	Difluoroacetic acid - Magnitude of the residue in laying hens Report No.: RARVN150, Edition Number: M-611007-01-1 [redacted] GLP/GEP: Yes unpublished	Yes	Bayer
KCA 6.4.2 / 01	[redacted].	2017	Difluoroacetic acid - Magnitude of the residue in dairy cows Report No.: RARVN149, Edition Number: M-608471-01-1 [redacted] GLP/GEP: Yes unpublished	Yes	Bayer

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.5.3 / 01	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on soybean processed commodities Report No.: RARVY029, Edition Number: M-428939-01-2 Bayer CropScience LP, Stilwell, KS, USA GLP/GEP: Yes unpublished	No	Bayer
KCA 6.5.3 / 02	Lenz, C.; Beedle, E.	2012	BYI 02960 200 SL - Magnitude of the residue in cotton processed commodities (crop subgroup 20C) Report No.: RARVY033, Edition Number: M-433122-01-1 Bayer Corporation, Stilwell, KS, USA GLP/GEP: Yes unpublished	No	Bayer
KCA 6.5.3 / 03	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on potato processed commodities Report No.: RARVY038, Edition Number: M-430542-01-2 Bayer CropScience LP, Stilwell, KS, USA GLP/GEP: Yes unpublished	No	Bayer
KCA 6.5.3 / 04	Freitag, T.; Hoffmeister, R.	2016	Determination of the residues of BYI 02960 in/on potato and the processed fractions (tuber, washed; peel washed; tuber, peeled; tuber, cooked; tuber with peel, cooked; fibre; pulp, dry; starch; waste; waste, dried; flakes; and crisps (Am.: chips)) after spray application of BYI 02960 SL 200 in the field in Germany and the Netherlands Report No.: 13-3149, Edition Number: M-546878-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.5.3 / 05	Schulte, G.; Ruhl, S.	2012	Processing study - Determination of the residues of BYI 02960 in/on spring barley and the processed fractions (malt sprouts; brewer's malt; brewer's grain, hops draff; brewer's yeast; beer; pearl barley; pearl barley rub off) after spraying of BYI 02960 SL 200 in the field in Germany Report No.: 10-3410, Edition Number: M-439853-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.5.3 / 06	Schulte, G.; Bauer, J.	2012	Processing study - Determination of the residues of BYI 02960 in/on wheat and the processed fractions (semolina; semolina bran; whiteflour bran; white flour; white bread; whole meal; wholemeal bread and wheat germ) after spraying of BYI 02960 SL 200 in the field in Germany Report No.: 10-3409, Edition Number: M-440307-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.5.3 / 07	Lenz, C.; Fischer, D. R.	2012	BYI 02960 200 SL - Magnitude of the residue in/on wheat processed commodities Report No.: RARVY031, Edition Number: M-427047-01-2 Bayer CropScience LP, Stilwell, KS, USA GLP/GEP: Yes unpublished	No	Bayer
KCA 6.5.3 / 08	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on field corn processed commodities Report No.: RARVY030, Edition Number: M-424774-01-1 Bayer CropScience LP, Stilwell, KS, USA GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.1 / 01	Schmeling, S.; Breuer-Rehm, M.	2012	Metabolism of [gemdimethyl-14C] deltamethrin in confined rotational crops Report No.: MEF-11/669, Edition Number: M-431769-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 01	Freitag, T.	2018	Determination of the residues of BYI 02960 in/on potato and soil after spraying of BYI 02960 SL 200 in the field in Germany, the Netherlands, Italy and Spain Report No.: 16-2510, Edition Number: M-613149-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 02	Freitag, T.; Effertz, C.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop rape and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands - Final report Report No.: 16-2511, Edition Number: M-617178-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2018-12-19** GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 03	Kaussmann, M.; van Berkum, S.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop rape and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Italy and Portugal Report No.: 16-2521, Edition Number: M-613466-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2018-12-19** GLP/GEP: Yes unpublished	No	Bayer

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.6.2 / 04	Freitag, T.; Kaussmann, M.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop barley and soil after spray application of BYI 02960 SL 200 to bare soil in the field in United Kingdom and the Netherlands Report No.: 16-2512, Edition Number: M-612254-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 05	Kaussmann, M.; van Berkum, S.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop barley and soil after spray application of BYI 02960 SL 200 to bare soil in the field in France (South) and Portugal Report No.: 16-2522, Edition Number: M-613602-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 06	Freitag, T.; Effertz, C.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop maize/corn and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands Report No.: 16-2514, Edition Number: M-617942-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2018-09-17** GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 07	Freitag, T.; van Berkum, S.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop maize/corn and soil after spray application of BYI 02960 SL 200 to bare soil in the field in France (South) and Portugal - Final report Report No.: 16-2524, Edition Number: M-617943-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 08	Freitag, T.; Hoffmeister, R.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop strawberry and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands Report No.: 16-2515, Edition Number: M-617563-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCA 6.6.2 / 09	Freitag, T.; van Berkum, S.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop strawberry and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Spain and Italy Report No.: 16-2525, Edition Number: M-617923-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2018-12-19** GLP/GEP: Yes unpublished	No	Bayer

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.6.2 / 10	Freitag, T.; Effertz, C.	2018	Determination of the residues of BYI 02960 in/on the field rotational crop cauliflower, broccoli and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium, the Netherlands, Italy and Spain Report No.: 16-2516, Edition Number: M-617941-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2018-09-19** GLP/GEP: Yes unpublished	No	Bayer

List of data submitted or referred to by the applicant and relied on, but already evaluated at EU peer review
Please note that all data mentioned as part of DAR, RAR, or EFSA journals are considered as relied on.

Deltamethrin

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.1 / 01	Grigor, A. F.	1990	Storage stability study for combined residues of tralomethrin, deltamethrin and trans-deltamethrin in lettuce in a freezer stability study. Chemalysis, Inc., USA Bayer Report No.: A73531 Edition Number: M-151815-01-1 Date: 1990-03-01 GLP/GEP: No, unpublished	No	Bayer
KCA 6.1 / 02	Grigor, A. F.	1991	Supplement to: Determination of the combined residues of tralomethrin, deltamethrin and trans-deltamethrin in lettuce in a freezer stability study. Chemalysis, Inc., USA Bayer Report No.: A71112 Edition Number: M-149583-01-1 Date: 1991-11-11 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.1 / 03	Ballesteros, C.	2012	Storage stability of residues of deltamethrin (AE F032640) and its isomers AE F108569 and AE 0035073 in orange during deep freeze storage for up to 24 months Bayer Report No.: 09-07 Edition Number: M-441996-01-1 Date: 2012-11-19 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.1 / 04	Czarnecki, J. J.	1996	Amended summary report covering submission guidelines for magnitude of the residue (171-4(k)), processed food/feed (171-4(l)), residue methodology (171-4(c)) and storage stability (171-4(e)) for residues of deltamethrin and its metabolites AgrEvo USA Company, Pikeville, NC, USA Bayer Report No.: A55828 Edition Number: M-139715-01-1 Date: 1996-10-22 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.1 / 05	Anon.	2015	Deltamethrin - AIR 3 renewal Bayer - 014/01279 - UK CRD request on residues (dated 09-10-2015) Bayer Report No.: M-536440-01-1 Date: 2015-10-09 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.1 / 06	Sadler, T.	2019	Linearity data for method HRAV-10 and method DGM F01/97-0 Bayer Report No.: M-646787-01-1 Date: 2019-01-18 GLP/GEP: n.a., unpublished **... also filed:** **KCA 4.1.2 / 58**	No	Bayer
KCA 6.1 / 07	Rose, S. J.	1985	One-year storage stability of tralomethrin and deltamethrin in soybean seeds. Tegeris Laboratories, Inc., Laurel, MD, USA Bayer Report No.: A73611 Edition Number: M-151894-01-1 Date: 1985-05-06 GLP/GEP: No, unpublished	No	Bayer
KCA 6.1 / 08	Czarnecki, J. J.	1996	Amended summary report covering submission guidelines for magnitude of the residue, processed food/feed, storage stability, and residue methodology for residues of deltamethrin, and metabolites, trans-deltamethrin and alpha-R-deltamethrin, AgrEvo USA Company, Pikeville, NC, USA Bayer Report No.: A55837 Edition Number: M-139724-01-1 Date: 1996-10-22 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.1 / 09	Hsu, R. S.; Czarnecki, J. J.; Kwiatkoski, D. M.	1993	Summary report covering submission guidelines for magnitude of the residue [171-4 (k)], processed food/feed [171-4 (l)], storage stability [171-4(e)] and residue methodology [171-4 (c)] for residues of tralomethrin and its metabolites delta Huntingdon Analytical Services, Middleport, NY, USA Bayer Report No.: A73997 Edition Number: M-152266-01-1 Date: 1993-08-02 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.1 / 10	Czarnecki, J. J.; Hsu, R. S.; Kwiatkoski, D. M.	1992	Summary report covering magnitude of the residue, storage stability and residue analytical method for residues of tralomethrin and its metabolites (deltamethrin and trans-deltamethrin) in or on tomatoes. Huntingdon Analytical Services, Middleport, NY, USA Bayer Report No.: A73998 Edition Number: M-152267-01-1 Date: 1992-12-21 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.1 / 11	Singer, G. M.	1999	At harvest deltamethrin-derived residues in cabbage following eight applications of DECIS (R) at the maximum proposed rate and the shortest proposed PHI, USA, 1998 AgrEvo USA Company, Pikeville, NC, USA Bayer Report No.: C002732 Edition Number: M-184972-01-1 Date: 1999-04-23 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.1 / 12	McKinney, F. R.; Clayton, F. B.	1991	Stability of Tralomethrin, cis-Deltamethrin and trans-Deltamethrin in cottonseed under freezer storage conditions [with alpha-R-Deltamethrin and stability in processed commodities (cottonseed fractions) added by amendment] EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: C038177 Report includes Trial Nos.: ECRCNC91ENCAA1US890011 Edition Number: M-224446-01-1 Date: 1991-10-09 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.1 / 13	Williams, L.	2000	Stability of Deltamethrin Residues in Cabbage During Frozen Storage, USA, 1999 Aventis CropScience USA LP, RTP, NC, USA Bayer Report No.: B002954 Report includes Trial Nos.: BP99R001 Edition Number: M-238621-01-1 Date: 2000-09-13 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.2 / 01	Ruzo, L. O.; Casida, J. E.	1979	Degradation of Decamethrin on Cotton Plants Journal: Journal of Agricultural and Food Chemistry Volume: 27 Issue: 3 Pages: 572-575 Year: 1979 Report No.: A20237 Edition Number: M-093407-01-1 GLP/GEP: n.a., published	No	published
KCA 6.2 / 02	Salmon, J.; van Assche, C. J.; Salmon, M.	1977	Etude preliminaire de l'absorption et du transport de RU 22974 sur coton. Procida, Les Algorithmes, Gif sur Yvette, France Bayer Report No.: A71134 Edition Number: M-149605-01-1 Date: 1977-09-13 GLP/GEP: No, unpublished	No	Bayer
KCA 6.2 / 03	Merricks, L.; Swidersky, P.	1985	Identification of the residues of (14C)-labeled deltamethrin, Decis, in the tomato plant Agrisearch UK Ltd., Melbourne, United Kingdom Bayer Report No.: A41994 Edition Number: M-125042-01-1 Date: 1985-04-24 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.2 / 04	Krebs, B.; Eickhoff, H.; Raquet, H.; Thier, W.	1986	Deltamethrin - Bestimmung von Rueckstaenden in Gemuesekulturen nach Aufnahme aus kontaminiertem Boden Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A34266 Edition Number: M-115043-01-1 Date: 1986-06-06 GLP/GEP: No, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.2 / 05	Erstfeld, K. M.; Larson, J. D.; Lange, B. D.	1994	C-14 Deltamethrin: Confined Accumulation in Rotational Crops 30 and 120 Day Experiment Pan-Agricultural Laboratories, Inc., Madera, CA, USA Bayer Report No.: A47914 Report includes Trial Nos.: 89-0101 Edition Number: M-136651-02-1 Date: 1991-08-26 **... amended: 1994-05-06** GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.2 / 06	Periasamy, R.; Kimmel, E. C.; Toia, R. F.	1994	Metabolism of [14C-acid] and [14C-alcohol] Decis (deltamethrin) in apples PTRL West, Inc., Richmond, CA, USA Bayer Report No.: A71034 Edition Number: M-149515-01-1 Date: 1994-01-18 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.2 / 07	Periasamy, R.; Kimmel, E. C.; Toia, R. F.	1994	Metabolism of [14C-Acid] and [14C-Alcohol] Decis (deltamethrin) in field corn. PTRL West, Inc., Richmond, CA, USA Bayer Report No.: A71098 Edition Number: M-149571-01-1 Date: 1994-02-11 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.2 / 08	Timme, G.; Frehse, H.; Laska, V.	1986	Statistical interpretation and graphic representation of the degradational behaviour of pesticide residues. II. Journal: Pflanzenschutz-Nachrichten Bayer Volume: 39 Issue: 2 Pages: 188-204 Year: 1986 Report No.: A53503 Edition Number: M-121089-01-2 GLP/GEP: n.a., published	No	published

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.2 / 09	Fisher, P. J.	2000	Conversion of tralomethrin to deltamethrin in mammals (expert summary) Aventis CropScience S.A., Lyon, France Bayer Report No.: C009954 Edition Number: M-199580-01-1 Date: 2000-10-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.2 / 10	[redacted]	1982	Pyrethroid metabolism: comparative fate in rats of tralomethrin, tralocythrin, deltamethrin and (1R,alpha S)-cis- cypermethrin. Journal: Journal of Agricultural and Food Chemistry Volume: 30 Pages: 631-636 Year: 1982 Report No.: A71114 Edition Number: M-149585-01-1 GLP/GEP: n.a., published **... also filed:** **KCA 5.1 / 01**	Yes	published
KCA 6.2 / 11	[redacted]	1990	Metabolism of 14C-tralomethrin in rats. [redacted] Report No.: A73039 Edition Number: M-151332-01-1 Date: 1990-04-05 GLP/GEP: Yes, unpublished **... also filed:** **KCA 5.1.1 / 01**	Yes	Bayer
KCA 6.2 / 12	[redacted]	2016	Deltamethrin - Additional information on residues and metabolism Bayer Report No.: M-560007-01-1 Date: 2016-07-21 GLP/GEP: n.a., unpublished	Yes	Bayer
KCA 6.2 / 13	Christian, I.	2019	Deltamethrin - Occurrence of metabolites in the rat, plants and livestock Bayer Report No.: M-646371-01-1 Date: 2019-01-03 GLP/GEP: n.a., unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.2 / 14	Christian, I.	2019	Summary of the toxicological and metabolism studies for Ddmethrin - ADME studies already evaluated during previous Annex I inclusion Bayer Report No.: M-646370-01-1 Date: 2019-01-14 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.2.1 / 01	O'Grodnick, J. S.; Larson, J. D.	1990	14C-deltamethrin: Nature of the residue in cotton. Hazleton Laboratories America, Inc., USA Bayer Report No.: A71094 Edition Number: M-149567-01-1 Date: 1990-09-12 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.2.1 / 02	Larson, J. D.	1994	14C-deltamethrin: Nature of the residue in cotton (analytical phase - supplements number 1 and 2) Hazleton Laboratories America, Inc., USA Bayer Report No.: A71095 Edition Number: M-191128-02-1 Date: 1991-08-26 **... amended: 1994-05-06** GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.2.1 / 03	Salmon, J.; van Assche, C. J.; Salmon, M.	1977	Preliminary uptake - Translocation studies of Ru 22974 in cotton. Procida, Les Algorithmes, Gif sur Yvette, France Bayer Report No.: A41995 Edition Number: M-149605-01-2 Date: 1977-09-13 GLP/GEP: No, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.2.2 / 01	[redacted]	1985	Metabolism, distribution, and excretion of deltamethrin by leghorn hens. Journal: Journal of Agricultural and Food Chemistry Volume: 33 Issue: 4 Pages: 610-617 Year: 1985 Report No.: A35015 Edition Number: M-116708-01-1 GLP/GEP: n.a., published **... also filed:** **KCA 5.1.1 / 06** **KCA 5.1.2 / 06**	Yes	published
KCA 6.2.2 / 02	[redacted]	1994	Equivalent mixture of (14C-phenyl)-cypermethrin, (14C-benzyl)-deltamethrin and (14C-phenoxyphenyl)-fenvalerate: Distribution - kinetics and excretion after single oral administration to laying hens [redacted] Bayer Report No.: A52315 Edition Number: M-133155-01-1 Date: 1994-03-30 GLP/GEP: Yes, unpublished **... also filed:** **KCA 5.1.1 / 03**	Yes	Bayer
KCA 6.2.2 / 03	[redacted]	1993	(14C-Benzyl)-deltamethrin: Distribution - Kinetics and Excretion after single intravenous Administration to laying Hens [redacted] Report No.: A51514 Edition Number: M-132448-01-1 Date: 1993-08-05 GLP/GEP: Yes, unpublished	Yes	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.2.3 / 01	[redacted]	1986	Fate of 14C-Deltamethrin in lactating dairy cows. Journal: Journal of Agricultural and Food Chemistry Volume: 34 Issue: 4 Pages: 753-758 Year: 1986 Report No.: A34280 Edition Number: M-115057-01-1 GLP/GEP: n.a., published **... also filed:** **KCA 5.1.1 / 07** **KCA 5.1.2 / 07**	Yes	published
KCA 6.2.3 / 02	[redacted]	1995	Metabolism of 14C-benzyl-tralomethrin and 14C-gem-dimethyl-tralomethrin in lactating dairy cattle and storage stability of tralomethrin and deltamethrin in cow milk and tissues. [redacted] Report No.: A70045 Report includes Trial Nos.: HR-01-88 Edition Number: M-148609-02-1 Date: 1991-09-27 **... amended: 1995-11-07** GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.4.2 / 05**	Yes	Bayer
KCA 6.2.5 / 01	[redacted]	1993	Deltamethrin: Bioconcentration exposure with Bluegill Sunfish (Lepomis macrochirus) and identification of resulting metabolites. [redacted] Report No.: A70918 Report includes Trial Nos.: 1719.0393.6231.140 Edition Number: M-149401-01-1 Date: 1993-12-20 GLP/GEP: Yes, unpublished	Yes	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 01	Fuchsbichler, G.	1990	Hoe 032640 (deltamethrin): Storage stability in hops and beer (storage interval 5 1/2 months). Bayer Report No.: A71869 Edition Number: M-150246-01-1 Date: 1990-03-29 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 02	McKinney, F. R.; Clayton, F. B.	1995	Stability of tralomethrin, cis-deltamethrin and trans-deltamethrin in cottonseed under freezer storage conditions (with alpha- R-Deltamethrin and stability in processed commodities, cottonseed fractions, added by amendment). EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A71103 Edition Number: M-149576-01-1 Date: 1995-01-11 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 03	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in animal feed. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71894 Edition Number: M-150269-01-1 Date: 1995-03-22 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 04	de Wilde, G.	1995	Deltamethrin: Residue data summary from supervised trials in legume vegetables (fresh): Beans and peas with pods. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71733 Edition Number: M-150126-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 05	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in stem vegetables: Aspargus. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71770 Edition Number: M-150158-01-1 Date: 1995-03-27 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 06	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in pulses: dry bean, dry lentils, dry peas. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71886 Edition Number: M-150261-01-1 Date: 1995-04-03 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 07	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in hops. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71842 Edition Number: M-150219-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 08	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in cacao. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71872 Edition Number: M-150249-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 09	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in coffee. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71895 Edition Number: M-150270-01-1 Date: 1995-03-30 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 10	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Citrus. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71438 Edition Number: M-149870-01-1 Date: 1995-03-13 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 11	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in stone fruits: Apricots, cherries, peaches, plums. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71519 Edition Number: M-149932-01-1 Date: 1995-03-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 12	de Wilde, G.	1995	Deltamethrin: Residues data summary trials in berries: Grapes (wine and table grapes). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71536 Edition Number: M-149946-01-1 Date: 1995-03-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 13	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in leaf vegetables and fresh herbs: Lettuce and similar. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71692 Edition Number: M-150086-01-1 Date: 1995-03-17 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 14	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in leaf vegetables, fresh herbs - Celery. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71730 Edition Number: M-150123-01-1 Date: 1995-03-20 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 15	Moede, J.	1996	Report on plant protection residue trial. (Hoe 032640, Prunus persica var persica and var nucipersica) AgrEvo UK Crop Protection Ltd., Chesterford Park, United Kingdom Bayer Report No.: A56981 Edition Number: M-140755-01-1 Date: 1996-06-11 GLP/GEP: No, unpublished	No	Bayer

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KCA 6.3 / 16	Klein, E. H. J.; Buerstell, H.	1996	Deltamethrin; Emulsifiable granules 6.25 % w/w; Code: AE F032640 00 EG06 A103 -Residue trials in peas (canning) to confirm maximum residue level compliance. Determination of active substance at harvest following three applications. European Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56787 Edition Number: M-140572-01-1 Date: 1996-10-07 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 17	Klein, E. H. J.; Martens, R.	1997	Residues trials in peaches to confirm Maximum Residue Level compliance. Determination of active substance following 3 applications. European Union, Southern zone, 1996. Deltamethrin - emulsifiable granules 62.5 a.s./kg. Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74186 Edition Number: M-152437-01-1 Date: 1997-08-11 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 18	Klein, E. H. J.; Martens, R.	1997	Residue trials in green beans to confirm Maximum Residue Level compliance. Determination of active substance following two applications. European Union, Northern zone 1996. Deltamethrin - EG emulsifiable granules 62.5 g as/kg Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74188 Edition Number: M-152439-01-1 Date: 1997-07-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 19	Klein, E. H. J.; Martens, R.	1997	Residue trials in green beans to confirm Maximum Residue Level compliance. Determination of active substance following two applications. European Union, Southern zone 1996. Deltamethrin - emulsifiable granules 62.5 g a.s./kg. Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74187 Edition Number: M-152438-01-1 Date: 1997-07-28 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 20	Klein, E. H. J.; Moede, J.	1996	Deltamethrin EG (emulsifiable granules) 6.25 %; Code: Hoe 032640 00 EG06 A102 -Determination of residues of Hoe 032640 to verify the existing maximum residue level following 3 applications in lettuce under field and plastic covered green ho Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A55221 Edition Number: M-139191-01-1 Date: 1996-05-03 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 21	Klein, E. H. J.; Moede, J.	1996	Deltamethrin; emulsifiable granules 6.25 % w/w; Code: AE F032640 00 EG06 A103 -Determination of residues of AE F032640 to verify the existing maximum residue level following three applications in lettuce under field conditions. 1995 Europea Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56782 Edition Number: M-140567-01-1 Date: 1996-09-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 22	McKinney, F. R.	1994	Determination of freezer residue stability for deltamethrin (alpha-R, cis, and trans) and tralomethrin in poultry tissues. EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A54085 Edition Number: M-134680-01-1 Date: 1994-08-12 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.4.1 / 02**	No	Bayer
KCA 6.3 / 23	Klein, E. H. J.	2001	Residues at harvest in kidney beans, green European Union, Southern zone 2000 Deltamethrin, AE F032640 emulsifiable concentrate (EC) 2.81 % w/w (= 25 g/L) Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016472 Edition Number: M-202465-01-1 Date: 2001-11-28 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 24	Klein, E. H. J.; Martens, R.	2000	Residues at harvest in walnuts European Union (northern zone) 1999 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A107 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C009200 Edition Number: M-198192-01-1 Date: 2000-09-28 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 25	Klein, E. H. J.; Martens, R.	2000	Residues at harvest in hazel-and walnuts European union (southern zone),1999 Deltamethrin, AE F032640 emulsifiable granule 6.25 % w/w Code: AE F032640 00 EG06 A107 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C009201 Edition Number: M-198193-01-1 Date: 2000-10-12 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 26	Davies, P.	2001	Residues at harvest in hazelnuts European Union (Southern zone), 2000 Deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (25 g/L) Code: AE F032640 00 EC03 B007 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C014846 Edition Number: M-207403-01-1 Date: 2001-11-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 27	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in tree nuts: Walnut. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71823 Edition Number: M-150203-01-1 Date: 1995-12-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 28	Luttringer, M.; Boltz, V.	1995	Rapport d´essai residus. Analyses de residus de deltamethrine sur noix. Institut National de la Recherche Agronomique, France Bayer Report No.: A70667 Edition Number: M-149168-01-1 Date: 1995-03-10 GLP/GEP: No, unpublished	No	Bayer

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KCA 6.3 / 29	Neuss, B.	2001	Residue data summary from supervised trials Tree nuts: Hazelnuts and walnuts Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015915 Edition Number: M-201403-01-1 Date: 2001-11-05 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 30	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in pome fruits (apples and pears). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71460 Edition Number: M-149883-01-1 Date: 1995-03-13 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 31	Kurzmeier, E.	1998	Residues data summary from supervised trials in pome fruit (apples). Additional data Deltamethrin Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005438 Edition Number: M-192078-01-1 Date: 1998-01-28 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 32	Neuss, B.	2001	Residues data summary from supervised trials - apples Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015916 Edition Number: M-201404-01-1 Date: 2001-11-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 33	Klein, E. H. J.	2001	Decline of residues in apples (bridging data) European Union, Southern zone 2000 Deltamethrin emulsifiable concentrate (EC) emulsifiable granule (EG) oil in water formulation (EW) 25 g/L / 6.25 %/ 15 g/L Codes: AE F032640 00 EC03 B005; AE F Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017035 Edition Number: M-203523-01-1 Date: 2001-11-26 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 34	Klein, E. H. J.; Buerstell, H.	1997	Residue trials in apples to confirm MRL compliance. Determination of a.i. following 5 applications. EU, Southern zone 1996 DT EG 62.5 g/kg Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74166 Edition Number: M-152417-01-1 Date: 1997-07-02 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 35	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in berries and small fruits: Strawberries. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71556 Edition Number: M-149963-01-1 Date: 1995-03-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 36	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Cane fruits: Raspberries, Blackberries. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71565 Edition Number: M-149972-01-1 Date: 1995-03-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 37	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in other small fruits and berries: currants and wild berries. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71567 Edition Number: M-149974-01-1 Date: 1995-03-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 38	Klein, E. H. J.	2001	Decline of residues in strawberries (bridging data) European Union, Northern Zone 2000 Deltamethrin EC (emulsifiable concentrate) 25 g/L EG (emulsifiable granule) 6.25 % EW (oil in water emulsion) 15 g/L Codes: AE F032640 00 EC03 B005; AE F Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016894 Edition Number: M-203252-01-1 Date: 2001-11-27 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 39	Kurzmeier, E.	1998	Residues data summary from supervised trials in berries and small fruits: Strawberries. additional data Deltamethrin Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005441 Edition Number: M-192084-01-1 Date: 1998-01-30 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 40	Klein, E. H. J.; Buerstell, H.	1996	Deltamethrin; EG (emulsifiable granule) 6.25 % w/w; Code: AE F032640 00 EG06 A102 -Residue trials in strawberries to confirm MRL compliance. Determination of active substance at harvest following 3 applications under field conditions. Europ Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56783 Edition Number: M-140568-01-1 Date: 1996-09-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 41	Klein, E. H. J.; Moede, J.	1996	Deltamethrin EG (emulsifiable granule) 6.25 %; Code: Hoe 032640 00 EG06 A102 - Determination of residues of Hoe 032640 to verify the existing maximum residue level following 3 applications in strawberries under field conditions. Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A55222 Edition Number: M-139192-01-1 Date: 1996-04-26 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 42	Neuss, B.	2001	Residues data summary from supervised trials Berries and small fruit: strawberries Additional data Deltmethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017004 Edition Number: M-203463-01-1 Date: 2001-11-19 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 43	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in miscellaneous fruits (figs, kiwi, ananas, olives). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71572 Edition Number: M-149979-01-1 Date: 1995-03-15 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 44	Klein, E. H. J.; Martens, R.	1999	Decline of residues in olives European Union (Southern zone) 1998 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005160 Edition Number: M-191497-01-1 Date: 1999-12-21 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 45	Davies, P.	2001	Decline of residues in olives European Union southern zone 2000 deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (25 g/L) Code: AE F032640 00 EC03 B007; AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016290 Edition Number: M-202111-01-1 Date: 2001-12-04 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 46	Neuss, B.	2001	Residues data summary from supervised trials on olives Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015755 Edition Number: M-201124-01-1 Date: 2001-11-19 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 47	Klein, E. H. J.; Martens, R.	2000	Residues at harvest in olives and processed fractions. European Union, Southern zone 1997 Deltamthrin, AE F032640 emulsifiable granule 6.25 % w/W Code: AE F032640 00 EG06 A105 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C010147 Edition Number: M-238971-01-1 Date: 2000-09-21 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.5.3 / 03**	No	Bayer
KCA 6.3 / 48	Klein, E. H. J.	2001	Residues at harvest in carrots European Union (Southern zone) 2000 Deltamethrin emulsifiable concentrate (EC), 2.81 % w/w = 25 g/L Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016257 Edition Number: M-202047-01-1 Date: 2001-11-05 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 49	Davies, P.	2001	Decline of residues in carrots European Union (Northern zone) 2000 Deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (25 g/L) Code: AE F032640 00 EC03 B007 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016078 Edition Number: M-201688-01-1 Date: 2001-11-14 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 50	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in bulb vegetables: Onions, Shallots, Garlic. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71606 Edition Number: M-150006-01-1 Date: 1995-03-17 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 51	Holzwarth, U.	2001	Residues data summary from supervised trials Bulb vegetables: Bulb onions - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015720 Edition Number: M-201054-01-1 Date: 2001-11-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 52	Klein, E. H. J.; Martens, R.	1999	Decline of residues in onions European Union, northern zone 1998 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C004595 Edition Number: M-188229-01-1 Date: 1999-10-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 53	Klein, E. H. J.; Martens, R.	1999	Decline of residues in onions European Union, southern zone 1998 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C004596 Edition Number: M-188231-01-1 Date: 1999-10-26 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 54	Welcker, H.	1999	Residues at harvest in onions; European Union (northern zone) 1997 - Deltamethrin emulsifiable granule (EG) 6.25% w/w (Code: AE F032640 00 EG06 A105). Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74344 Edition Number: M-152577-01-1 Date: 1999-02-18 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 55	Klein, E. H. J.	1999	Residues at harvest in onions European Union (southern zone) 1997 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A105 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C001500 Edition Number: M-182781-01-1 Date: 1999-02-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 56	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in fruiting vegetables: Solanacea (Tomatoes, Peppers, Aubergines). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71610 Edition Number: M-150010-01-1 Date: 1995-03-20 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 57	Davies, P.	2001	Decline of residues in protected tomatoes European Union (indoor) 2000 Deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (25 g/L) Code: AE F032640 00 EC03 B007 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016111 Edition Number: M-201758-01-1 Date: 2001-11-23 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 58	Klein, E. H. J.; Buerstell, H.	1997	Residue trials in field tomatoes (industrial use) to confirm MRL compliance. Determination of a.i. substance at harvest following 4 applications. EU Southern zone 1996. Deltamethrin EG 62.5 g/kg Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74164 Edition Number: M-152415-01-1 Date: 1997-06-24 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 59	Neuss, B.	2001	Residues data summary from supervised trials - tomatoes Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017003 Edition Number: M-203461-01-1 Date: 2001-11-20 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 60	Klein, E. H. J.	1998	Residue trials in tomatoes to reconfirm a maximum residue level. Determination of a.s. decline following 4 applications under field conditions. European Union (southern zone) 1997 Deltamethrin emulsifiable granule 6.25% Code: AE F032640 00 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C001247 Edition Number: M-182154-01-1 Date: 1998-11-09 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 61	de Wilde, G.	1995	Deltamethrin: Residues data summary supervised trials in Cucurbits with edible peel (Cucumber and gherkin). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71645 Edition Number: M-150039-01-1 Date: 1995-03-13 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 62	Klein, E.H.-J.	1999	Decline of residues in cucumbers or zucchini European Union (southern zone) 1997 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A105 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C002581 Edition Number: M-184758-01-1 Date: 1999-03-16 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 63	Klein, E. H. J.; Buerstell, H.	1999	Residues at harvest in cucumber/zucchini European Union, Southern zone 1998 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005161 Edition Number: M-191499-01-1 Date: 1999-11-11 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 64	Davies, P.	2001	Decline of residues in protected cucumbers 2000 Deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (25 g/L) Code: AE F032640 00 EC03 B007 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015571 Edition Number: M-200743-01-1 Date: 2001-11-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 65	Neuss, B.	2001	Residue data summary from supervised trials Fruit vegetables: cucurbits with edible peel: cucumber and zucchini Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015759 Edition Number: M-201132-01-1 Date: 2001-11-07 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 66	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in fruiting vegetables with inedible peel: Melon. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71654 Edition Number: M-150048-01-1 Date: 1995-03-13 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 67	Klein, E. H. J.; Martens, R.	1999	Decline of residues in melons European Union, southern zone 1998 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005490 Edition Number: M-192157-01-1 Date: 1999-10-28 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 68	Holzwarth, U.	2001	Residues data summary from supervised trials Fruiting vegetables: Cucurbits with inedible peel melon - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015719 Edition Number: M-201052-01-1 Date: 2001-11-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 69	Klein, E. H. J.	1998	Residue trials in melons to reconfirm an maximum residue level. Determination of a.s. decline following 4 applications under field conditions. European union (southern zone) 1997 Deltamethrin emulsifiable granule 62.5 g a.s./kg Code: AE F03 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56781 Edition Number: M-140566-01-1 Date: 1998-09-24 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 70	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in sweet corn. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71661 Edition Number: M-150055-01-1 Date: 1995-03-13 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 71	Klein, E. H. J.	2001	Residues at harvets in sweet corn European Union, Northern zone 2000 Deltamethrin emulsifiable concentrate (EC), 2.81 % w/w (=25 g/L) Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016147 Edition Number: M-201828-01-1 Date: 2001-10-31 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 72	Klein, E. H. J.	2001	Residues at harvest in sweet corn European Union (Southern zone) 2000 Deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (= 25 g/L) Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C014943 Edition Number: M-207608-01-1 Date: 2001-10-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 73	Holzwarth, U.	2001	Residues data summary from supervised trials Sweet corn - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015919 Edition Number: M-201410-01-1 Date: 2001-11-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 74	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in vegetables - Brassica, Leafy brassica. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71689 Edition Number: M-150083-01-1 Date: 1995-03-20 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 75	Holzwarth, U.	2001	Residues data summary from supervised trials Leaf brassica: Curly kale - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015756 Edition Number: M-201126-01-1 Date: 2001-11-03 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 76	Klein, E. H. J.; Martens, R.	2000	Residue on harvest in curly kale , European union (northern zone) 1999 Deltamethrin, AE F032640 emulsifiable granule (EG), 6.25 % w/w Code: AE F032640 00 EG06 A107 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C009198 Edition Number: M-198190-01-1 Date: 2000-10-12 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 77	Klein, E. H. J.; Moede, J.	1996	Deltamethrin; emulsifiable granules 6.25 % w/w; Code: Hoe 032640 00 EG06 A103 - Deltamethrin; emulsifiable concentrate 25 g/l; Code: Hoe 032640 00 EC03 B001 - Residue trials in curly kale to confirm maximum residue level compliance. Determi Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56790 Edition Number: M-140575-01-1 Date: 1996-09-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 78	Klein, E. H. J.; Martens, R.	1999	Decline of residues in curly kale European Union, Northern zone 1998 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005491 Edition Number: M-192159-01-1 Date: 1999-12-07 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 79	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in leaf vegetables and herbs: Spinach and similar. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71710 Edition Number: M-150104-01-1 Date: 1995-03-17 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 80	Neuss, B.	2001	Residue data summary from supervised trials Leaf vegetables and fresh herbs: Spinach Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015920 Edition Number: M-201412-01-1 Date: 2001-11-06 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 81	Klein, E. H. J.	2001	Residues at harvest in spinach European Union (Southern zone) 2000 Deltamethrin emulsifiable concentrate (EC) 25 g/L, emulsifiable granule (EG) 6.25 g/L, oil in water formulation (EW) 15 g/L Code: AE F032640 00 EC03 B005, AE F032640 00 EG06 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015002 Edition Number: M-199679-01-1 Date: 2001-10-31 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 82	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in leaf vegetables: Witlof (endives). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71726 Edition Number: M-150119-01-1 Date: 1995-03-17 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 83	Holzwarth, U.	2001	Residues data summary from supervised trials Leaf vegetables and fresh herbs: Witloof (chickory) - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015722 Edition Number: M-201058-01-1 Date: 2001-11-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 84	Klein, E. H. J.	1998	Residue trials on chicory to reconfirm an maximum residue level. Determination of a.s. decline following 3 applications. European Union (Northern zone) 1997 Deltamethrin emulsifiable granule 6.25 % Code: AE F032640 00 EG06 A105 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C001246 Edition Number: M-182150-01-1 Date: 1998-11-24 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 85	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in stem vegetables: Artichokes. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71771 Edition Number: M-150159-01-1 Date: 1995-03-17 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 86	Holzwarth, U.	2001	Residues data summary from supervised trials Leaf and stem vegetables: Garden artichoke - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015723 Edition Number: M-201060-01-1 Date: 2001-11-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 87	Klein, E. H. J.; Buerstell, H.	1996	Deltamethrin; Emulsifiable granules 6.25 % w/w; Code: AE F032640 00 EG06 A103 - Residue trials in artichokes to confirm maximum residue level compliance Determination of active substance at harvest following four applications European Union Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56780 Edition Number: M-140565-01-1 Date: 1996-10-11 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 88	Klein, E. H. J.; Buerstell, H.	1997	Deltamethrin EG 62.5 g/kg - Residue trials in artichokes to confirm MRL compliance following 4 applications (EU, Southern zone 1996) Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A74152 Edition Number: M-152404-01-1 Date: 1997-06-02 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 89	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Stem vegetables: Leeks. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71781 Edition Number: M-150169-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 90	Holzwarth, U.	2001	Residues data summary from supervised trials Stem vegetables: Leek Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015758 Edition Number: M-201130-01-1 Date: 2001-11-02 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 91	Klein, E. H. J.	1999	Residues at harvest in leeks European Union (southern zone) 1997 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A105 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C001463 Edition Number: M-182694-01-1 Date: 1999-02-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 92	Klein, E. H. J.; Martens, R.	1999	Decline of residues in leek European Union (Northern zone) 1998 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005162 Edition Number: M-191501-01-1 Date: 1999-12-20 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 93	Klein, E. H. J.; Martens, R.	2000	Residues at harvest in leek European Union (northern zone) 1999 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A107 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C009199 Edition Number: M-198191-01-1 Date: 2000-08-28 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 94	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Fungi vegetables: Cultivated mushroom. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71788 Edition Number: M-150175-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 95	Holzwarth, U.	2001	Residues data summary from supervised trials Fungi: Cultivated mushroom - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015925 Edition Number: M-201419-01-1 Date: 2001-11-03 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 96	Balluff, M.	2000	Determination of residues of deltamethrin in mushrooms following two applications of AE F032640 00 EG06 (common name: Decis EG 06) at 2 locations in Germany and France respectively, 1998 Arbeitsgemeinschaft GAB GmbH & IFU GmbH, Germany Bayer Report No.: C008388 Edition Number: M-197450-01-1 Date: 2000-05-02 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 97	Holzwarth, U.	2001	Residues data summary from supervised trials Rape - Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015921 Edition Number: M-201414-01-1 Date: 2001-11-03 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 98	Klein, E. H. J.; Moede, J.	1996	Deltamethrin; Emulsifiable granules 6.25 % w/w; Code: Hoe 032640 00 EG06 A102 - Residue trials in rape to confirm maximum residue level compliance determination of active substance decline following 4 applications European Union (Northern z Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A55220 Edition Number: M-139190-01-1 Date: 1996-08-12 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 99	Neuss, B.	2001	Residue data summary from supervised trials - sunflower Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015923 Edition Number: M-201417-01-1 Date: 2001-12-11 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 100	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in oilseeds: Sunflower seed, Rape seed, Soja bean, Cotton seed. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71790 Edition Number: M-150177-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 101	Klein, E. H. J.; Martens, R.	1999	Decline of residues in sunflower European Union, southern zone, 1998 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C004594 Edition Number: M-188226-01-1 Date: 1999-10-27 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 102	Klein, E. H. J.	2001	Residues at harvest sunflowers European Union, Northern zone 2000 Deltamethrin, AE F032640 emulsifiable concentrate (EC 2.81 % w/w (= 25 g/L) Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016321 Edition Number: M-202174-01-1 Date: 2001-11-28 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 103	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Oilseeds: Sunflower seed, Rape seed, Soja bean, Cotton seed. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71810 Edition Number: M-150194-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 104	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in potatoes. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71877 Edition Number: M-150252-01-1 Date: 1995-04-03 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 105	Neuss, B.	2001	Residue data summary from supervised trials Potatoes Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015924 Edition Number: M-201418-01-1 Date: 2001-11-12 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 106	Kalter, G. J.; Merz, H. D.	1994	Pirimicarb (100 g/l) und Deltamethrin (7.5 g/l) -Emulgierbares Konzentrat- (Code: Hoe 032640 03 EC11 A202) Untersuchung der Rueckstaende in Kartoffeln nach fuenfmaliger Anwendung von Hoe 032640 03 EC11 A202 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A52611 Edition Number: M-133421-01-1 Date: 1994-12-16 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 107	Klein, E. H. J.; Moede, J.	1996	Deltamethrin; Emulsifiable granules 6.25 % w/w; Code: Hoe 032640 00 EG06 A103 - Residue trials in potatoes to confirm maximum residue level compliance. Determination of active substance at harvest following four applications European Union Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56789 Edition Number: M-140574-01-1 Date: 1996-08-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 108	Klein, E. H. J.; Martens, R.; Werner, H. J.	1999	Decline of residues in potatoes European Union (southern zone) 1998 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C004453 Edition Number: M-187966-01-1 Date: 1999-08-27 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 109	Davies, P.	2001	Residues at harvest in potatoes European Union (Southern zone) 2000 Deltamethrin emulsifiable concentrate (EC) 25 g/L (2.81 %) Code: AE F032640 00 EC03 B007, AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015284 Edition Number: M-200206-01-1 Date: 2001-11-14 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 110	Mestres, R.	1982	Residues Analysis; Commercial Product, K-Othrine 0.05 dust; Decamethrine 0.05%; Potatoes (stored) University of Montpellier, Faculte de Pharmacie, Montpellier, France Bayer Report No.: A97788 Edition Number: M-175301-01-1 Date: 1982-07-01 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 111	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in tea. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71829 Edition Number: M-150208-01-1 Date: 1995-03-21 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 112	Klein, E. H. J.	2001	Decline of residues in tea and processed fractions South East Asia 2000/2001 Deltamethrin, AE F032640 emulsifiable concentrate (EC) 2.81 % w/w (= 25 g/L) Code: AE F032640 00 EC03 B0007 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C016591 Edition Number: M-202700-01-1 Date: 2001-11-30 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 113	Neuss, B.	2001	Residues data summary from supervised trials - tea Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017005 Edition Number: M-203465-01-1 Date: 2001-11-23 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 114	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Brassica vegetables - Head brassica: Brussels sprout and Head cabbage. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71678 Edition Number: M-150072-01-1 Date: 1995-03-20 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 115	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in Brassica vegetables: Kohlrabi. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71691 Edition Number: M-150085-01-1 Date: 1995-03-20 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 116	Thier, W.; Gorbach, S.	1978	Pflanzenschutzmittel-Rueckstaende. (Kohlrabi). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A13420 Edition Number: M-065112-01-1 Date: 1978-02-09 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 117	Thier, W.; Gorbach, S.	1978	Pflanzenschutzmittel-Rueckstaende. (Kohlrabi). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A13421 Edition Number: M-065116-01-1 Date: 1978-02-09 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 118	Thier, W.; Gorbach, S.	1978	Pflanzenschutzmittel-Rueckstaende. (Kohlrabi). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A13422 Edition Number: M-065120-01-1 Date: 1978-02-09 GLP/GEP: No, unpublished	No	Bayer

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KCA 6.3 / 119	Thier, W.; Gorbach, S.	1978	Pflanzenschutzmittel-Rueckstaende. (Kohlrabi). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A13423 Edition Number: M-065124-01-1 Date: 1978-02-09 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 120	Neuss, B.	2001	Residue data summary from supervised trials Root and tuber vegetables: Carrots Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015917 Edition Number: M-201406-01-1 Date: 2001-11-05 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 121	de Wilde, G.	1995	Delatmethrin: Residues data summary from supervised trials in 4/ Cereals: Rice. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71984 Edition Number: M-150348-01-1 Date: 1995-04-05 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 122	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in: 3/ Cereals: Corn (Maize). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71957 Edition Number: M-150324-01-1 Date: 1995-04-05 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3 / 123	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in: 2) Cereals: Sorghum (Millet). Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71951 Edition Number: M-150318-01-1 Date: 1995-04-05 GLP/GEP: n.a., unpublished	No	Bayer

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KCA 6.3 / 124	Block, H.	2010	Determination of residues of deltamethrin in/on barley after spraying of Decis EC 025 in the field in Germany 2009 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00281 Edition Number: M-398036-01-1 Date: 2010-11-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 125	Block, H.	2010	Determination of residues of deltamethrin in/on corn after spraying of Decis EC 025 in the field in Germany 2009 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00285 Edition Number: M-398043-01-1 Date: 2010-11-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 126	Ballesteros, C.; Meilland-Berthier, I.	2011	Determination of the residues of deltamethrin and imidacloprid in/on pea, field after spraying of imidacloprid & deltamethrin in the field in France (South) Bayer Report No.: 10-2056 Report includes Trial Nos.: 10-2056-01 Edition Number: M-405310-01-1 Date: 2011-04-12 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 127	Ballesteros, C.	2011	Determination of the residues of deltamethrin and imidacloprid in/on pea, field after spraying of imidacloprid & deltamethrin in the field in France (South) and Italy Bayer Report No.: 09-2007 Report includes Trial Nos.: 09-2007-01 09-2007-03 09-2007-04 Edition Number: M-409261-01-1 Date: 2011-06-14 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3 / 128	Block, H.	2011	Determination of residues of deltamethrin in/on tomato after spraying of deltamethrin EC 25AK G or deltamethrin EC 25 AF G in the greenhouse in France, Spain, Poland and Germany 2010 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00016 Edition Number: M-410061-01-1 Date: 2011-06-28 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3 / 129	Meilland-Berthier, I.	2011	Determination of the residues of deltamethrin in/on spinach after spray application of Decis EC 025 in the field in United Kingdom and Belgium Bayer Report No.: 10-2029 Report includes Trial Nos.: 10-2029-01 10-2029-02 Edition Number: M-410009-01-1 Date: 2011-06-30 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 130	Block, H.	2011	Determination of residues of deltamethrin in/on lettuce after spraying of Decis EC 025 in the greenhouse in France 2009 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00290 Report includes Trial Nos.: S09-00290-02 Edition Number: M-411438-01-1 Date: 2011-07-01 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 131	Block, H.	2011	Determination of residues of deltamethrin in/on oilseed rape after spraying of Decis EC 025 in the field in Spain and France 2010 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00002 Report includes Trial Nos.: S10-00002-01 S10-00002-02 Edition Number: M-410870-01-1 Date: 2011-07-07 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	Owner
KCA 6.3 / 132	Block, H.	2011	Determination of residues of deltamethrin in/on spinach and the processed fractions (washings, leaf washed, cooking water, leaf cooked) after spraying of Decis EC 025 in the field in Poland and Germany 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00001 Report includes Trial Nos.: S10-00001-01 S10-00001-02 S10-00001-03 Edition Number: M-411380-01-1 Date: 2011-07-19 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 133	Block, H.	2011	Determination of residues of Deltamethrin in/on currant after spraying of Decis EC 025 in the field in France and Italy 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00003 Report includes Trial Nos.: S10-00003-01 S10-00003-02 S10-00003-03 Edition Number: M-413402-01-1 Date: 2011-07-22 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 134	Block, H.	2011	Determination of residues of deltamethrin in/on barley after spraying of Decis EC 025 in the field in UK and Germany 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00007 Report includes Trial Nos.: S10-00007-01 S10-00007-02 S10-00007-03 Edition Number: M-411372-01-1 Date: 2011-07-22 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 135	Block, H.	2011	Determination of residues of deltamethrin in/on lettuce after spraying of Decis EC 025 in the greenhouse in Germany 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00014 Edition Number: M-415853-01-1 Date: 2011-07-22 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 136	Block, H.	2011	Determination of residues of deltamethrin in/on barley after spraying of Decis EC 025 in the field in France and Spain 2010 Bayer Report No.: S10-00008 Report includes Trial Nos.: S10-00008-01 S10-00008-02 S10-00008-03 S10-00008-04 Edition Number: M-411558-01-1 Date: 2011-07-29 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 137	Block, H.	2011	Determination of residues of deltamethrin in/on corn after spraying of Decis EC 025 in the field in Bulgaria and France (south) 2010 Bayer Report No.: S10-00012 Report includes Trial Nos.: S10-00012-01 S10-00012-02 S10-00012-03 S10-00012-04 Edition Number: M-411562-01-1 Date: 2011-07-29 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 138	Block, H.	2011	Determination of residues of deltamethrin in/on raspberries after spraying of Decis EC 025 in the field in France, Spain and Italy 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00013 Report includes Trial Nos.: S10-00013-01 S10-00013-02 S10-00013-03 Edition Number: M-411569-01-1 Date: 2011-07-29 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 139	Block, H.	2011	Determination of residues of deltamethrin in/on sweet cherry after spraying of Decis EC 025 in the field in Spain and Italy 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00004 Report includes Trial Nos.: S10-00004-01 S10-00004-02 Edition Number: M-411417-02-1 Date: 2010-07-07 **... amended: 2011-08-15** GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 140	Block, H.	2011	Determination of residues of deltamethrin in/on barley after spraying of Decis EC 025 in the field in France and Spain 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00005 Report includes Trial Nos.: S10-00005-01 S10-00005-02 S10-00005-03 S10-00005-04 Edition Number: M-412389-01-1 Date: 2011-08-17 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 141	Block, H.	2011	Determination of residues of deltamethrin in/on olive and the processed fractions (washings; whole fruit, washed; pomace, wet; vegetation water; oil, crude, oil virgin) after spraying of decis EC 025 in the field in Spain and Italy 2009 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00291 Report includes Trial Nos.: S09-00291-01 S09-00291-02 S09-00291-03 Edition Number: M-412393-01-1 Date: 2011-08-18 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 142	Block, H.	2011	Determination of residues of deltamethrin in/on raspberries after spraying of Decis EC 025 in the field in Germany 2009 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00288 Report includes Trial Nos.: S09-00288-01 Edition Number: M-413294-01-1 Date: 2011-08-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 143	Block, H.	2011	Determination of residues of deltamethrin in/on olive after spraying of Decis EC 025 in the field in France and Italy 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00015 Report includes Trial Nos.: S10-00015-03 S10-00015-04 Edition Number: M-413291-01-1 Date: 2011-08-25 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 144	Ballesteros, C.	2011	Determination of the residues of deltamethrin and imidacloprid in/on kale, curly after spraying of imidacloprid & deltamethrin in the field in Italy and Spain Bayer Report No.: 09-2002 Report includes Trial Nos.: 09-2002-01 09-2002-02 Edition Number: M-413003-01-1 Date: 2011-08-26 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 145	Block, H.	2011	Determination of residues of deltamethrin in/on currant after spraying of Decis EC 025 in the field in Germany 2009 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00277 Report includes Trial Nos.: S09-00277-01 Edition Number: M-414546-01-1 Date: 2011-08-26 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 146	Schulz, H.	2011	Study on the residue behaviour of deltamethrin in oranges after treatment with Decis Fluxx EC 025 under field conditions in Spain, 2010 SGS Institut Fresenius GmbH, Taunusstein, Germany Bayer Report No.: 10-2302 Report includes Trial Nos.: 10-ES-067 10-ES-068 10-ES-069 10-ES-070 Edition Number: M-421031-01-1 Date: 2011-09-22 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 147	Block, H.	2011	Determination of residues of deltamethrin in/on spinach and the processed fractions (washings, leaf washed, cooking water, leaf cooked) after spraying of Decis EC 025 in the field in Germany 2009 Eurofins-GAB GmbH, Stade, Germany Bayer Report No.: S09-00274 Report includes Trial Nos.: S09-00274-02 Edition Number: M-415862-01-1 Date: 2011-10-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 148	Block, H.	2011	Determination of residues of deltamethrin in/on green peas after spraying of deltamethrin EW 15A G in the open field in France and Spain 2009 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00293 Report includes Trial Nos.: S09-00293-01 S09-00293-02 S09-00293-03 S09-00293-04 Edition Number: M-415856-01-1 Date: 2011-10-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 149	Block, H.	2011	Determination of residues of deltamethrin in/on corn after spraying of Decis EC 025 in the field in Hungary and Germany 2010 Eurofins-GAB GmbH, Stade, Germany Bayer Report No.: S10-00011 Report includes Trial Nos.: S10-00011-1 S10-00011-2 S10-00011-3 Edition Number: M-415871-01-1 Date: 2011-10-17 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 150	Meilland-Berthier, I.	2011	Determination of the residues of deltamethrin in/on maize/corn after spray application of Decis EC 025 in the field in Germany, northern France, United Kingdom, the Netherlands, southern France, Spain and Portugal Bayer Report No.: 10-2042 Report includes Trial Nos.: 10-2042-01 10-2042-02 10-2042-03 10-2042-04 10-2042-05 10-2042-06 10-2042-07 10-2042-08 Edition Number: M-416542-01-1 Date: 2011-10-27 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 151	Noss, G.; Krusell, L.; Ruhl, S.	2011	Determination of residues of deltamethrin in/on barley after spraying of Decis EC 025 in the field in France (South), Spain, Italy and Portugal Bayer Report No.: 10-2032 Report includes Trial Nos.: 10-2032-01 10-2032-02 10-2032-03 10-2032-04 Edition Number: M-418208-01-1 Date: 2011-11-23 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 152	Block, H.	2012	Determination of residues of deltamethrin in/on strawberries and the processed Fractions (whole fruit, washings and preserve) after spraying of Decis EC 025 in the field in France and Spain 2009 Eurofins Agroscience Services GmbH, Stade, Germany Bayer Report No.: S09-00275 Report includes Trial Nos.: S09-00275-01 S09-00275-02 S09-00275-03 S09-00275-04 Edition Number: M-414534-02-1 Date: 2011-09-20 **... amended: 2012-06-08** GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 153	Meilland-Berthier, I.	2012	Determination of the residues of deltamethrin in/on winter wheat after spray application of Decis EC 025 in the field in Belgium, northern France, United Kingdom, Germany, southern France, Italy, Spain and Portugal Bayer Report No.: 10-2036 Report includes Trial Nos.: 10-2036-01 10-2036-02 10-2036-03 10-2036-04 10-2036-05 10-2036-06 10-2036-07 10-2036-08 Edition Number: M-435250-01-1 Date: 2012-07-18 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.3.3 / 07**	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 154	Meilland-Berthier, I.	2012	Determination of the residues of deltamethrin in/on winter barley after spray application of Decis EC 025 in the field in Germany, northern France, United Kingdom, southern France, Portugal, Italy and Spain Bayer Report No.: 10-2035 Report includes Trial Nos.: 10-2035-01 10-2035-02 10-2035-03 10-2035-04 10-2035-05 10-2035-07 10-2035-08 Edition Number: M-435247-01-1 Date: 2012-07-19 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 155	Meilland-Berthier, I.	2012	Determination of the residues of deltamethrin in/on tomato after spray application of Decis EC 025 in the field in southern France, Spain, Portugal, Greece and Italy Bayer Report No.: 11-2047 Report includes Trial Nos.: 11-2047-01 11-2047-02 11-2047-03 11-2047-04 11-2047-05 11-2047-06 Edition Number: M-442495-01-1 Date: 2012-11-22 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 156	Meilland-Berthier, I.	2012	Determination of the residues of deltamethrin in/on tomato and cherry tomato after spray of Decis EC 025 in the field in northern France, Germany and Belgium Bayer Report No.: 11-2048 Report includes Trial Nos.: 11-2048-01 11-2048-02 11-2048-03 11-2048-04 Edition Number: M-442496-01-1 Date: 2012-11-26 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 157	Schoening, R.; Diehl, P.	2013	Determination of the residues of deltamethrin and imidacloprid in/on lettuce after spray application of imidacloprid & deltamethrin OD 85 in Spain, Italy, France (south) and Portugal Bayer Report No.: 11-2052 Report includes Trial Nos.: 11-2052-01 11-2052-02 11-2052-03 11-2052-04 Edition Number: M-448490-02-1 Date: 2013-03-04 **... amended: 2013-04-04** GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 158	Meilland-Berthier, I.	2013	Determination of the residues of deltamethrin in/on garden pea after spray application of deltamethrin EW 015 in southern France, Spain, Italy and Greece Bayer Report No.: 12-2063 Report includes Trial Nos.: 12-2063-01 12-2063-02 12-2063-03 12-2063-04 Edition Number: M-451494-01-1 Date: 2013-04-16 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 159	Meilland-Berthier, I.	2013	Determination of the residues of deltamethrin in/on winter barley after spray application of Decis EC 025 in the field in the Netherlands and southern France Bayer Report No.: 11-2107 Report includes Trial Nos.: 11-2107-01 11-2107-02 11-2107-03 11-2107-04 Edition Number: M-456339-01-1 Date: 2013-06-06 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3 / 160	Meilland-Berthier, I.	2013	Amendment No. 1 to Final Report No: 12-2064 - Determination of the residues of deltamethrin in/on cauliflower after spray application of deltamethrin EW 015 in southern France, Spain, Italy and Greece Bayer Report No.: 12-2064 Report includes Trial Nos.: 12-2064-01 12-2064-02 12-2064-03 12-2064-04 Edition Number: M-451517-02-1 Date: 2013-04-16 **... amended: 2013-10-17** GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.3.1 / 16**	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3 / 161	Block, H.	2015	Determination of residues of deltamethrin in/on chinese cabbage and curly kale after spraying of Deltamethrin EW 15A G in the field in France (North and South), Italy, Poland and Germany 2009 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S09-00289 Report includes Trial Nos.: S09-00289-01 S09-00289-02 S09-00289-03 S09-00289-04 S09-00289-05 S09-00289-06 S09-00289-07 S09-00289-08 Edition Number: M-411407-02-1 Date: 2011-06-29 **... amended: 2015-09-25** GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.1 / 01	Krebs, B.; Gorbach, S.	1983	Berichtsbogen fuer Rueckstandsuntersuchungen mit Pflanzenbehandlungsmitteln (Blumenkohl). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A25151 Edition Number: M-098086-01-1 Date: 1983-01-18 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3.1 / 02	Krebs, B.; Gorbach, S.	1983	Berichtsbogen fuer Rueckstandsuntersuchungen mit Pflanzenbehandlungsmitteln (Blumenkohl). Handelslabor Dr. Koerl und Dr. Specht, Hamburg, Germany Bayer Report No.: A25152 Edition Number: M-098087-01-1 Date: 1983-01-18 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3.1 / 03	Krebs, B.; Gorbach, S.	1983	Berichtsbogen fuer Rueckstandsuntersuchungen mit Pflanzenbehandlungsmitteln (Blumenkohl). LUFA, DEU; Bayer Report No.: A25153 Edition Number: M-098088-01-1 Date: 1983-01-18 GLP/GEP: No, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3.1 / 04	Krebs, W.; Gorbach, S.	1983	Berichtsbogen fuer Rueckstandsuntersuchungen mit Pflanzenbehandlungsmitteln (Blumenkohl). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A25154 Edition Number: M-098089-01-1 Date: 1983-01-18 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3.1 / 05	Davies, P.	2001	Decline of residues in cauliflower European Union (Northern zone) 2000 Deltamethrin emulsifiable concentrate (EC) 2.81 % w/w (25 g/L) Code: AE F032640 00 EC03 B007 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015567 Edition Number: M-200735-01-1 Date: 2001-11-14 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.1 / 06	Klein, E. H. J.	1999	Residues at harvest in cauliflowers / broccoli European Union (southern zone) 1997 Deltamethrin emulsifiable granule (EG) 6.25% w/w Code: AE F032640 00 EG06 A105 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C001462 Edition Number: M-182691-01-1 Date: 1999-02-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.1 / 07	Klein, E. H. J.; Buerstell, H.	2006	Residues at harvest in cauliflower and broccoli European Union, Southern zone 1998 Deltamethrin emulsifiable granule (EG) 6.25 % w/w Code: AE F032640 00 EG06 A106 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005163 Edition Number: M-191503-02-1 Date: 1999-11-15 **... amended: 2006-08-07** GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.3.1 / 08	de Wilde, G.	1995	Deltamethrine: Residues data summary from supervised trials in vegetables - Brassica: Broccoli and Cauliflower. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71668 Edition Number: M-150062-01-1 Date: 1995-03-20 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3.1 / 09	Neuss, B.	2001	Residue data summary from supervised trials Flowering brassicas: cauliflower and broccoli Additional data Deltamethrin Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C015757 Edition Number: M-201128-01-1 Date: 2001-11-14 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3.1 / 10	Idstein, H.; Bock, K. D.	1989	Berichtsbogen fuer Rueckstandsuntersuchungen (Hoe 032640, Brassica oleracea var sabellica) Universitaet Muenchen, Muenchen, Germany Bayer Report No.: A41457 Edition Number: M-124552-01-1 Date: 1989-06-01 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3.1 / 11	Idstein, H.; Bock, K. D.	1989	Berichtsbogen fuer Rueckstandsuntersuchungen (Hoe 032640, Brassica oleracea var sabellica) Universitaet Muenchen, Muenchen, Germany Bayer Report No.: A41456 Edition Number: M-124551-01-1 Date: 1989-06-01 GLP/GEP: No, unpublished	No	Bayer
KCA 6.3.1 / 12	Idstein, H.; Bock, K. D.	1989	Berichtsbogen fuer Rueckstandsuntersuchungen (Hoe 032640, Brassica oleracea var sabellica) Universitaet Muenchen, Muenchen, Germany Bayer Report No.: A41458 Edition Number: M-124553-01-1 Date: 1989-06-01 GLP/GEP: No, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3.1 / 13	Sole, C.	2002	Residue study with deltamethrin in cauliflower European Union (Northern Zone) 2001 Deltamethrin emulsifiable concentrate (EC) 24.8 g/L (25 g/L nominal) Code: AE F032640 00 EC03 B018 (= EXP05610A) ADME Bioanalyses S.A., Vergeze, France Bayer Report No.: C023140 Report includes Trial Nos.: 01R054 Edition Number: M-214429-01-1 Date: 2002-05-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.1 / 14	Noss, G.; Schneeberg-Seeba, C.	2008	Determination of the residues of deltamethrin in/on cauliflower after spraying of deltamethrin EW 15 (015 EW) in the field in Spain and Italy Bayer Report No.: RA-2555/06 Report includes Trial Nos.: R 2006 0305/3 = 0305 - 06 R 2006 0306/1 = 0306 - 06 Edition Number: M-303449-01-1 Date: 2008-06-24 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.1 / 15	Noss, G.; Wolters, A.	2008	Determination of the residues of deltamethrin in/on cauliflower after spraying of deltamethrin EW 15 (015 EW) in the field in Spain and Italy Bayer Report No.: RA-2547/07 Report includes Trial Nos.: R 2007 0048/2 = 0048 - 07 R 2007 0049/0 = 0049 - 07 Edition Number: M-308923-01-1 Date: 2008-09-30 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3.1 / 16	Meilland-Berthier, I.	2013	Amendment No. 1 to Final Report No: 12-2064 - Determination of the residues of deltamethrin in/on cauliflower after spray application of deltamethrin EW 015 in southern France, Spain, Italy and Greece Bayer Report No.: 12-2064 Report includes Trial Nos.: 12-2064-01 12-2064-02 12-2064-03 12-2064-04 Edition Number: M-451517-02-1 Date: 2013-04-16 **... amended: 2013-10-17** GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.3 / 160**	No	Bayer
KCA 6.3.2 / 01	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in root and tuber vegetables. Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71585 Edition Number: M-149986-01-1 Date: 1995-03-16 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3.2 / 02	Klein, E. H. J.	2001	Residues at harvest in sugar beets European Union, Northern zone 2000 Deltamethrin, AE F032640 emulsifiable concentrate (EC) 2.81 % w/w (= 25 g/L) Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017186 Edition Number: M-203812-01-1 Date: 2001-12-12 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.2 / 03	Sole, C.	2002	Residue study with deltamethrin in sugar beets European Union (Northern zone) 2001 Deltamethrin emulsifiable concentrate (EC) 24.8 g/L (25 g/L nominal) Code: AE F032640 00 EC03 B018 (=EXP05610A) ADME Bioanalyses S.A., Mougins, France Bayer Report No.: C023401 Edition Number: M-214930-01-1 Date: 2002-05-24 GLP/GEP: Yes, unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	Owner
KCA 6.3.2 / 04	Klein, E. H. J.	2001	Residues at harvest in sugar beets European Union, Southern szone 2000 Deltamethrin, AE F032640 emulsifiable concentrate (EC) 2.81 % w/w (= 25 g/L) Code: AE F032640 00 EC03 B005 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017408 Edition Number: M-204224-01-1 Date: 2001-12-06 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.2 / 05	Sole, C.	2002	Residue study with deltamethrin in sugar beets European Union (Southern zone) 2001 Deltamethrin emulsifiable concentrate (EC) 24.8 g/L (25 g/L nominal) Code: AE F032640 00 EC03 B018 (EXP05610A) ADME Bioanalyses S.A., Mougins, France Bayer Report No.: C024054 Report includes Trial Nos.: 01R052 Edition Number: M-216175-01-1 Date: 2002-06-13 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.3 / 01	McKinney, F. R.	1993	Determination of cis-deltamethrin, trans-deltamethrin and alpha-R-deltamethrin in various grains, grain fractions and grain dusts under 20.C, 30.C and froozen storage conditions. EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A71102 Edition Number: M-149575-01-1 Date: 1993-08-10 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.3 / 02	de Wilde, G.	1995	Deltamethrin: Residues data summary from supervised trials in 1/ Cereals: Wheat, Barley, Oats... Hoechst Schering AgrEvo S.A., France Bayer Report No.: A71908 Edition Number: M-150279-01-1 Date: 1995-04-06 GLP/GEP: n.a., unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.3.3 / 03	Wiesener, G.	1998	Residues data summary from supervised trials in cereals (wheat, winter variety). Additional data Deltamethrin Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005447 Edition Number: M-192094-01-1 Date: 1998-02-06 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.3.3 / 05	Billian, P.; Krusell, L.	2009	Determination of the residues of deltamethrin in/on winter wheat after mixing of Deltamethrin & Piperonylbutoxide EC 275 in the Room, hall, store, ... in Germany, Greece, Portugal and the United Kingdom Bayer Report No.: 08-2214 Report includes Trial Nos.: 08-2214-01 08-2214-02 08-2214-03 08-2214-04 Edition Number: M-360719-01-1 Date: 2009-12-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.3 / 06	Block, H.	2011	Determination of residues of deltamethrin in/on wheat after spraying of Decis EC 025 in the field in UK and Poland 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00009 Edition Number: M-411373-01-1 Date: 2011-07-22 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3.3 / 07	Meilland-Berthier, I.	2012	Determination of the residues of deltamethrin in/on winter wheat after spray application of Decis EC 025 in the field in Belgium, northern France, United Kingdom, Germany, southern France, Italy, Spain and Portugal Bayer Report No.: 10-2036 Report includes Trial Nos.: 10-2036-01 10-2036-02 10-2036-03 10-2036-04 10-2036-05 10-2036-06 10-2036-07 10-2036-08 Edition Number: M-435250-01-1 Date: 2012-07-18 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.3 / 153**	No	Bayer
KCA 6.3.3 / 08	Meilland-Berthier, I.	2011	Determination of the residues of deltamethrin in/on wheat after spray application of Decis EC 025 in the field in Southern France, Italy, Spain and Greece Bayer Report No.: 10-2233 Report includes Trial Nos.: 10-2233-01 10-2233-02 10-2233-03 10-2233-04 Edition Number: M-413097-01-1 Date: 2011-08-30 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.3 / 09	Block, H.	2011	Determination of residues of deltamethrin in/on wheat after spraying of Decis EC 025 in the field in France and Italy 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00006 Edition Number: M-411367-01-1 Date: 2011-07-22 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.3.3 / 10	Block, H.	2011	Determination of residues of deltamethrin in/on wheat after spraying of Decis EC 025 in the field in France and Spain 2010 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-00010 Report includes Trial Nos.: S10-00010-01 S10-00010-04 S10-00010-05 Edition Number: M-433851-01-1 Date: 2011-09-20 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.3.3 / 04	Klein, E. H. J.; Buerstell, H.	1997	Deltamethrin; emulsifiable granules 6.25 % w/w; Code: AE F032640 00 EG06 A103 - Residue trials in winter-wheat to confirm maximum residue level compliance. Determination of active substance at harvest following two applications. European un Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56788 Edition Number: M-140573-01-1 Date: 1997-01-13 GLP/GEP: Yes, unpublished **... also filed:** **Document MCA / 01** **KCA 6.4.2 / 06**	No	Bayer
KCA 6.4 / 01	Anon.	2015	Deltamethrin - AIR 3 renewal Bayer - 2014/01279: UK CRD request (dated 13-10-2015): Residues - Livestock metabolism Bayer Report No.: M-536726-01-1 Date: 2015-10-23 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.4 / 02	Mousques, A.	2016	Compilation of dRR tables for deltamethrin residue studies from 2009 onwards - Results displayed for cis-deltamethrin, trans isomer and alpha-R isomer Bayer Report No.: M-559648-01-1 Date: 2016-07-13 GLP/GEP: n.a., unpublished **... also filed:** **KCA 5.8.1 / 09**	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.4 / 03	Christian, I.; Mousquès, A.	2018	Deltamethrin - Information on the behaviour of the alpha-R isomer and the trans-isomer of cis-deltamethrin in livestock matrices Bayer Report No.: M-628340-01-1 Date: 2018-06-29 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.4.1 / 01	[redacted]	1995	Effects of a supplemented deltamethrin and piperonyl butoxide diet on levels of residues in products of animal origin.~a2. Feeding studies in poultry. Journal: Journal of Agricultural and Food Chemistry Volume: 43 Pages: 1039-1043 Year: 1995 Report No.: A70891 Edition Number: M-149375-01-1 GLP/GEP: n.a., published	Yes	published
KCA 6.4.1 / 02	McKinney, F. R.	1994	Determination of freezer residue stability for deltamethrin (alpha-R, cis, and trans) and tralomethrin in poultry tissues. EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A54085 Edition Number: M-134680-01-1 Date: 1994-08-12 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.3 / 22**	No	Bayer
KCA 6.4.1 / 03	[redacted]	1994	Magnitude of the residues in meat and eggs for tralomethrin (RU 25474) and its major metabolite deltamethrin (RU 22974) in white Leghorn chickens. [redacted] Report No.: A71106 Edition Number: M-149579-01-1 Date: 1994-08-24 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.5.1 / 01**	Yes	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.4.2 / 01	[redacted]	2000	Feeding study of a mixture of tralomethrin and deltamethrin in dairy cows and magnitude of the combined residues of tralomethrin, deltamethrin and trans-deltamethrin in milk and tissues of dairy cattle. (2 volumes). [redacted] Report No.: A70946 Report includes Trial Nos.: HRAV Proj. #87-0123 HRAV Project No. 87-0123 Edition Number: M-149428-03-1 Date: 1988-11-04 **... amended: 2000-05-23** GLP/GEP: Yes, unpublished	Yes	Bayer
KCA 6.4.2 / 02	Akhtar, M. H.; Danis, C.; Trenholm, H. L.; Hartin, K. E.	1992	Deltamethrin residues in milk and tissues of lactating dairy cows. Journal: Journal of Environmental Science and Health : Part B Volume: B27 Issue: 3 Pages: 235-253 Year: 1992 Report No.: A70892 Edition Number: M-149376-01-1 GLP/GEP: n.a., published	No	published
KCA 6.4.2 / 03	Martens, R.	2000	Calculation of the 1x dosage rate for deltamethrin in a ruminant livestock feeding study (according to EU-document 7031/VI/95 rev.4, 22.07.1996 (draft)) Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C009557 Edition Number: M-198796-01-1 Date: 2000-09-06 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.4.2 / 04	Singer, S. S.; Hurst, K.	2001	Survey of Reports on Analysis for Deltamethrin in Milk from Cows and Humans Bayer Report No.: B003480 Edition Number: M-240501-01-1 Date: 2001-09-24 GLP/GEP: n.a., unpublished **... also filed:** **KCA 5.6 / 04** **KCA 6.9 / 01**	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.4.2 / 05	[redacted]	1995	Metabolism of 14C-benzyl-tralomethrin and 14C-gem-dimethyl-tralomethrin in lactating dairy cattle and storage stability of tralomethrin and deltamethrin in cow milk and tissues. [redacted] Report No.: A70045 Report includes Trial Nos.: HR-01-88 Edition Number: M-148609-02-1 Date: 1991-09-27 **... amended: 1995-11-07** GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.2.3 / 02**	Yes	Bayer
KCA 6.4.2 / 06	Klein, E. H. J.; Buerstell, H.	1997	Deltamethrin; emulsifiable granules 6.25 % w/w; Code: AE F032640 00 EG06 A103 - Residue trials in winter-wheat to confirm maximum residue level compliance. Determination of active substance at harvest following two applications. European un Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: A56788 Edition Number: M-140573-01-1 Date: 1997-01-13 GLP/GEP: Yes, unpublished **... also filed:** **Document MCA / 01** **KCA 6.3.3 / 04**	No	Bayer
KCA 6.4.3 / 01	[redacted]	1993	Effects of a supplemented deltamethrin and piperonyl butoxide diet on residues in products of animal origin. I. Feeding study in pigs. Journal: Journal of Agricultural and Food Chemistry Volume: 41 Pages: 2416-2420 Year: 1993 Report No.: A70890 Edition Number: M-149374-01-1 GLP/GEP: n.a., published	Yes	published

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KCA 6.5 / 01	Mestres, G.; Espinoza, C.; Chevallier, C.	1986	Effets sur les residus de Deltamethrine de la transformation des produits agricoles en vue de leur consommation Journal: Med.et Nutr. Volume: 22 Issue: 3 Pages: 181-184 Year: 1986 Report No.: A41874 Edition Number: M-124937-01-1 GLP/GEP: n.a., published	No	published
KCA 6.5 / 02	Mestres, G.; Espinoza, C.; Chevallier, C.	1986	Effects of edible crop processing on deltamethrin residues Journal: Unknown Pages: 1;14 Year: 1986 Report No.: A72063 Edition Number: M-150406-01-1 GLP/GEP: n.a., published	No	published
KCA 6.5 / 03	Thier, W.; Gorbach, S.	1980	Berichtsbogen fuer Rueckstandsuntersuchungen mit Pflanzenbehandlungsmitteln (Spinat). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A20420 Edition Number: M-093631-01-1 Date: 1980-11-17 GLP/GEP: No, unpublished	No	Bayer
KCA 6.5 / 04	Thier, W.; Krebs, B.	1983	Berichtsbogen fuer Rueckstandsuntersuchungen mit Pflanzenbehandlungsmitteln (Spinat). Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A25427 Edition Number: M-098339-01-1 Date: 1983-02-15 GLP/GEP: No, unpublished	No	Bayer

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KCA 6.5 / 05	Maurer, T.	2001	Investigation of the nature of the potential residue in the products of industrial processing or household preparation Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017397 Edition Number: M-204204-01-1 Date: 2001-11-27 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.5.3 / 04**	No	Bayer
KCA 6.5 / 06	Martens, R.	1999	Statement on the acute dietary risk assessment for deltamethrin Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C006040 Edition Number: M-193192-01-1 Date: 1999-11-18 GLP/GEP: n.a., unpublished **... also filed:** **KCA 6.9 / 07**	No	Bayer
KCA 6.5.1 / 01	[redacted]	1994	Magnitude of the residues in meat and eggs for tralomethrin (RU 25474) and its major metabolite deltamethrin (RU 22974) in white Leghorn chickens. [redacted] Report No.: A71106 Edition Number: M-149579-01-1 Date: 1994-08-24 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.4.1 / 03**	Yes	Bayer
KCA 6.5.1 / 02	Christian, I.	2013	Deltamethrin: Metabolic behaviour of 3-phenoxybenzaldehyde Bayer Report No.: M-466413-01-1 Date: 2013-10-03 GLP/GEP: n.a., unpublished	No	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	Owner
KCA 6.5.3 / 01	Brady, S. S.	1999	Magnitude of deltamethrin residues in or on apples and processed apple commodities resulting from three applications of Decis (R) insecticide USA, 1998 AgrEvo USA Company, Pikeville, NC, USA Bayer Report No.: C002139 Edition Number: M-183978-01-1 Date: 1999-03-30 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.5.3 / 02	Brady, S. S.	1999	Magnitude of deltamethrin residues in or on tomatoes and processed tomato commodities resulting from six applications of Decis (R) insecticide, USA, 1998 AgrEvo USA Company, Pikeville, NC, USA Bayer Report No.: C002859 Edition Number: M-185190-01-1 Date: 1999-04-20 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.5.3 / 03	Klein, E. H. J.; Martens, R.	2000	Residues at harvest in olives and processed fractions. European Union, Southern zone 1997 Deltamthrin, AE F032640 emulsifiable granule 6.25 % w/W Code: AE F032640 00 EG06 A105 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C010147 Edition Number: M-238971-01-1 Date: 2000-09-21 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.3 / 47**	No	Bayer
KCA 6.5.3 / 04	Maurer, T.	2001	Investigation of the nature of the potential residue in the products of industrial processing or household preparation Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C017397 Edition Number: M-204204-01-1 Date: 2001-11-27 GLP/GEP: Yes, unpublished **... also filed:** **KCA 6.5 / 05**	No	Bayer

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KCA 6.5.3 / 05	Billian, P.; Reineke, A.	2010	Determination of the residues of deltamethrin in/on winter wheat and processed fractions after mixing of Deltamethrin & Piperonylbutoxide EC 275 in the room, hall store ... in Germany, Greece, Portugal and the United Kingdom Bayer Report No.: 08-3214 Report includes Trial Nos.: 08-3214-01 08-3214-02 08-3214-03 08-3214-04 Edition Number: M-363957-01-1 Date: 2010-02-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.5.3 / 06	Schulte, G.	2016	Additional chromatograms of study report 08-3214: Determination of the residues of deltamethrin in/on winter wheat and processed fractions after mixing of deltamethrin & piperonylbutoxide EC 275 in the room, hall store ... in Germany, Greece, Portugal and the United Kingdom Bayer Report No.: M-559765-01-1 Date: 2016-07-19 GLP/GEP: No, unpublished	No	Bayer
KCA 6.6.1 / 01	Schmeling, S.; Breuer-Rehm, M.	2012	Metabolism of [gemdimethyl-14C] deltamethrin in confined rotational crops Bayer Report No.: MEF-11/669 Edition Number: M-431769-01-1 Date: 2012-05-14 GLP/GEP: Yes, unpublished	No	Bayer
KCA 6.6.1 / 02	Erstfeld, K. M.; Larson, J. D.; Lange, B. D.	1994	C-14 Deltamethrin - Confined accumulation in rotational crops 30 and 120 Day Experiment - including - Supplement to the report (amendment) Pan-Agricultural Laboratories, Inc., Madera, CA, USA Bayer Report No.: A73873 Report includes Trial Nos.: 89-0101 Edition Number: M-136651-02-2 Date: 1991-08-26 **... amended: 1994-05-06** GLP/GEP: Yes, unpublished	No	Bayer

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KCA 6.9 / 01	Singer, S. S.; Hurst, K.	2001	Survey of Reports on Analysis for Deltamethrin in Milk from Cows and Humans Bayer Report No.: B003480 Edition Number: M-240501-01-1 Date: 2001-09-24 GLP/GEP: n.a., unpublished **... also filed:** **KCA 5.6 / 04** **KCA 6.4.2 / 04**	No	Bayer
KCA 6.9 / 02	CRD	2012	Data requirements handbook (version 2.2, June 2012) Chemicals Regulation Directorate, CRD, York, United Kingdom Bayer Report No.: M-449685-01-1 Date: 2012-06-30 GLP/GEP: n.a., unpublished	No	Bayer
KCA 6.9 / 03	[redacted]	1999	Analysis of polychlorinated biphenyls, pyrethroid insecticides and fragrances in human milk using a laminar cup liner in the GC injector Publisher: Springer-Verlag Journal: European Food Research Technology Volume: 212 Pages: 247-251 Year: 2001 Report No.: M-449790-01-1 GLP/GEP: n.a., published	Yes	published
KCA 6.9 / 04	[redacted].	1996	The environmental fate of xenobiotics - Pesticides contamination in animal tissues and foods, monitoring results 1993-1994-1995 Publisher: Anon. Journal: Proceedings of the X Symposium Pesticide Chemistry Pages: 473-481 Year: 1996 Report No.: M-449809-01-1 GLP/GEP: n.a., published	Yes	published
KCA 6.9 / 05	Anon.	1997	Guidelines for predicting dietary intake of pesticide residues (revised) Publisher: WHO - World Health Organization Year: 1997 Report No.: M-449680-01-1 GLP/GEP: n.a., published	No	published

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KCA 6.9 / 06	[redacted]	2001	Addendum to monograph Annex B, presented by RMS Sweden, draft dated November 26, 2001 Aventis comments on dietary intake calculations (sections B.6.15.3, page 28) Deltamethrin [redacted] Report No.: C017841 Edition Number: M-205036-01-1 Date: 2001-12-06 GLP/GEP: n.a., unpublished	Yes	Bayer
KCA 6.9 / 07	Martens, R.	1999	Statement on the acute dietary risk assessment for deltamethrin Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C006040 Edition Number: M-193192-01-1 Date: 1999-11-18 GLP/GEP: n.a., unpublished **... also filed:** **KCA 6.5 / 06**	No	Bayer
KCA 6.9 / 08	Shipp, E.	2019	Deltamethrin - In silico assessment of the plant metabolite 3-phenoxybenzaldehyde Bayer Report No.: M-646818-01-1 Date: 2019-01-18 GLP/GEP: n.a., unpublished **... also filed:** **KCA 5.8.1 / 12**	No	Bayer
KCA 6.10.1 / 01	Miles, M.; Radix, P.; Brink, H.	2017	Statement: Label extension Decis forte (EC100): Additional information concerning the presence of residues in nectar and honey following applications in flowering oil seed rape Bayer Report No.: M-598954-01-2 Date: 2017-08-29 GLP/GEP: n.a., unpublished	No	Bayer

Flupyradifurone

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KIIA 6.1.1 /01	Netzband, D.; Timberlake, B. C.; Harbin, A. M.	2012	Storage stability of BYI 02960, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices (18-month data) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP046-1, Edition Number: M-428412-02-1 EPA MRID No.: 48977401 Date: 2012-04-03 ...Amended: 2012-11-01 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.1 /02	[redacted]	2012	BYI 02960 - Magnitude of the residue in dairy cows - Amended report [redacted] Report No.: RARVP050-1, Edition Number: M-428416-02-1 EPA MRID No.: 48843842 Date: 2012-04-03 ...Amended: 2012-05-31 GLP/GEP: yes, unpublished ...also filed: KIIA 4.3 /08 ...also filed: KIIA 6.4.2 /01	Y	Bayer
KIIA 6.2.1 /01	Justus, K.	2011	Metabolism of [furanone-4-14C]BYI 02960 in tomatoes Bayer CropScience, Report No.: MEF-11/016, Edition Number: M-411352-01-3 EPA MRID No.: 48843801 Date: 2011-07-25 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /02	Justus, K.	2011	Metabolism of [pyridinylmethyl-14C]BYI 02960 in tomatoes Bayer CropScience, Report No.: MEF-11/182, Edition Number: M-411500-01-2 EPA MRID No.: 48843802 Date: 2011-07-26 GLP/GEP: yes, unpublished	N	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KIIA 6.2.1 /03	Unold, M.; Justus, K	2011	Metabolism of [ethyl-1-14C]BYI 02960 in tomatoes Bayer CropScience, Report No.: MEF-11/498, Edition Number: M-413996-01-2 EPA MRID No.: 48843803 Date: 2011-09-05 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /04	Justus, K.	2011	Metabolism of [furanone-4-14C]BYI 02960 in potatoes Bayer CropScience, Report No.: MEF-10/769, Edition Number: M-415234-01-2 EPA MRID No.: 48843804 Date: 2011-09-14 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /05	Justus, K.	2011	Metabolism of [pyridinylmethyl-14C]BYI 02960 in potatoes Bayer CropScience, Report No.: MEF-10/710, Edition Number: M-415078-01-2 EPA MRID No.: 48843805 Date: 2011-09-14 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /06	Justus, K.	2011	Metabolism of [furanone-4-14C]BYI 02960 in apples Bayer CropScience, Report No.: MEF-11/499, Edition Number: M-422562-01-1 EPA MRID No.: 48843806 Date: 2011-12-29 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /07	Justus, K.	2011	Metabolism of [pyridinylmethyl-14C]BYI 02960 in apples Bayer CropScience, Report No.: MEF-11/198, Edition Number: M-414678-01-2 EPA MRID No.: 48843807 Date: 2011-09-09 GLP/GEP: yes, unpublished	N	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KIIA 6.2.1 /08	Schmeling, S.; Weber, E.	2011	Metabolism of [furanone-4-14C]BYI 02960 in cotton after spray application Bayer CropScience, Report No.: MEF-11/392, Edition Number: M-421625-01-2 EPA MRID No.: 48843808 Date: 2011-12-12 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /09	Schmeling, S.; Weber, E.	2011	Metabolism of [pyridinylmethyl-14C]BYI 02960 in cotton after spray application Bayer CropScience, Report No.: MEF-11/393, Edition Number: M-421691-01-2 EPA MRID No.: 48843809 Date: 2011-12-12 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /10	Schmeling, S.; Weber, E.	2011	Metabolism of [furanone-4-14C]BYI 02960 in paddy rice Bayer CropScience, Report No.: MEF-11/058, Edition Number: M-414219-01-2 EPA MRID No.: 48843810 Date: 2011-09-09 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /11	Schmeling, S.; Weber, E.	2011	Metabolism of [pyridinylmethyl-14C]BYI 02960 in paddy rice Bayer CropScience, Report No.: MEF-11/059, Edition Number: M-414328-01-2 EPA MRID No.: 48843811 Date: 2011-09-09 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.2.1 /12	Schoening, R.; Ruhl, S.	2012	Determination of residues of difluoroacetic acid in extracts of samples from plant metabolism and confined rotational crops studies after application of BYI 02960 Bayer CropScience, Report No.: MR-11/050, Edition Number: M-422550-01-1 EPA MRID No.: 48843812 Date: 2012-01-17 GLP/GEP: yes, unpublished	N	Bayer

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KIIA 6.2.2 /01	[redacted]	2012	[Pyridinylmethyl-14C]BYI 02960: Metabolism in the laying hen [redacted], Report No.: MEF-11/199, Edition Number: M-422162-01-2 EPA MRID No.: 48843814 Date: 2012-01-10 GLP/GEP: yes, unpublished	Y	Bayer
KIIA 6.2.2 /02	[redacted]	2012	[Furanone-4-14C]BYI 02960: Metabolism in the laying hen [redacted] Report No.: MEF-11/200, Edition Number: M-422263-01-2 EPA MRID No.: 48843813 Date: 2012-01-10 GLP/GEP: yes, unpublished	Y	Bayer
KIIA 6.2.3 /01	[redacted]	2011	[Pyridinylmethyl-14C]BYI 02960: Metabolism in the lactating goat [redacted] Report No.: MEF-11/269, Edition Number: M-419701-01-2 EPA MRID No.: 48843816 Date: 2011-12-07 GLP/GEP: yes, unpublished	Y	Bayer
KIIA 6.2.3 /02	[redacted]	2011	[Furanone-4-14C]BYI 02960: Metabolism in the lactating goat [redacted], Report No.: MEF11/268, Edition Number: M-421995-01-2 EPA MRID No.: 48843815 Date: 2011-12-16 GLP/GEP: yes, unpublished	Y	Bayer

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KIIA 6.3.1.1 /01	Noss, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on lettuce after spraying of BYI 02960 SL 200 in the field in the Netherlands, Belgium, France (North) and Germany Bayer CropScience, Report No.: 10-2223, Report includes Trial Nos.: 10-2223-01 10-2223-02 10-2223-03 10-2223-04 10-2223-05 Edition Number: M-424742-01-1 Date: 2012-02-08 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.1 /02	Uceda, L.	2012	Amendment No. 1 - Determination of the residues of BYI 02960 in/on lettuce after spray application of BYI 02960 SL 200 in the field in Germany, northern France and Belgium Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2082, Report includes Trial Nos.: 11-2082-01 11-2082-02 11-2082-03 11-2082-04 Edition Number: M-425941-02-1 Date: 2012-02-23 ...Amended: 2012-03-05 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.1 /03	Schoening, R.; Bauer, J.	2011	Determination of the residues of BYI 02960 in/on lettuce, head after spray application of BYI 02960 SL 200 in the field in France (South), Spain and Italy - Amendment no. 0001 to report no. 10-2213 Bayer CropScience, Report No.: 10-2213, Report includes Trial Nos.: 10-2213-01 10-2213-02 10-2213-03 10-2213-04 10-2213-05 Edition Number: M-425913-02-1 Date: 2011-09-14 ...Amended: 2012-02-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.1 /04	Uceda, L.	2012	Amendment No.1 - Determination of the residues of BYI 02960 in/on lettuce after spray application of BYI 02960 SL 200 in the field in Spain, Italy, Southern France and Portugal Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2071, Report includes Trial Nos.: 11-2071-01 11-2071-02 11-2071-03 11-2071-04 Edition Number: M-425784-02-1 Date: 2012-02-23 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.1 /05	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on lettuce after spraying of BYI 02960 SL 200 in the greenhouse in France (North), Germany, the Netherlands and Italy Bayer CropScience, Report No.: 10-2212, Report includes Trial Nos.: 10-2212-01 10-2212-02 10-2212-03 10-2212-04 10-2212-05 Edition Number: M-425829-01-1 Date: 2012-02-22 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.1 /06	Uceda, L.	2012	Determination of the residues of BYI 02960 in/on lettuce after spray application of BYI 02960 SL 200 in the greenhouse in northern France, Italy, Spain and Germany Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2070, Report includes Trial Nos.: 11-2070-01 11-2070-02 11-2070-03 11-2070-04 Edition Number: M-425786-01-1 Date: 2012-02-23 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.2 /01	Noss, G.; Ballmann, C.	2012	Determination of the residues of BYI 02960 in/on hop after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-2225, Report includes Trial Nos.: 10-2225-01 10-2225-02 10-2225-03 10-2225-04 Edition Number: M-425351-01-1 Date: 2012-02-13 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.2 /02	Noss, G.; Ballmann, C.	2012	Determination of the residues of BYI 02960 in/on hop after spray application of BYI 02960 SL 200 in Germany Bayer CropScience, Report No.: 11-2076, Report includes Trial Nos.: 11-2076-01 11-2076-02 11-2076-03 11-2076-04 Edition Number: M-425339-01-1 Date: 2012-02-13 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.3 /01	Bauer, J.; Schulte, G.	2012	Determination of the residues of BYI 02960 in/on apple after spraying of BYI 02960 SL 200 in the field in Germany, France (north), the Netherlands and Belgium Bayer CropScience, Report No.: 10-2171, Report includes Trial Nos.: 10-2171-01 10-2171-02 10-2171-03 10-2171-04 10-2171-05 10-2171-06 Edition Number: M-434587-01-1 Date: 2012-07-05 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.3 /02	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on apple after spray application of BYI 02960 SL 200 in the field in Germany, northern France and the United Kingdom Bayer CropScience, Report No.: 11-2077, Report includes Trial Nos.: 11-2077-01 11-2077-02 11-2077-03 11-2077-04 Edition Number: M-438329-01-1 Date: 2012-09-10 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.3 /03	Bauer, J.; Schulte, G.	2012	Determination of the residues of BYI 02960 in/on apple after spraying of BYI 02960 SL 200 in the field in France (south), Italy and Spain Bayer CropScience, Report No.: 10-2172, Report includes Trial Nos.: 10-2172-01 10-2172-02 10-2172-03 10-2172-04 10-2172-05 10-2172-06 Edition Number: M-434603-01-1 Date: 2012-07-05 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.3 /04	Schulte, G.; Bauer, J.; Ruhl, S.	2012	Determination of the residues of BYI 02960 in/on apple after spray application of BYI 02960 SL 200 in the field in southern France, Spain, Italy and Portugal Bayer CropScience, Report No.: 11-2078, Report includes Trial Nos.: 11-2078-01 11-2078-02 11-2078-03 11-2078-04 Edition Number: M-439845-01-1 Date: 2012-10-16 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.4 /01	Rosati, D.; Ratajczak, M.	2012	Determination of the residues of BYI 02960 in/on grape after spraying and spraying, low-volume of BYI 02960 SL 200 in the field in Germany, France (North) and Belgium Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 10-2218, Report includes Trial Nos.: 10-2218-01 10-2218-02 10-2218-03 10-2218-04 10-2218-05 Edition Number: M-437138-01-1 Date: 2012-08-22 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.4 /02	Meilland-Berthier, I.	2012	Determination of the residues of BYI 02960 in/on grape after high or low-volume spray application of BYI 02960 SL 200 in Germany and northern France Bayer CropScience, Report No.: 11-2089, Report includes Trial Nos.: 11-2089-01 11-2089-02 11-2089-03 11-2089-04 Edition Number: M-436857-01-1 Date: 2012-08-17 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.4 /03	Rosati, D.; Ratajczak, M.	2012	Determination of the residues of BYI 02960 in/on grape after spraying and spraying, low-volume of BYI 02960 SL 200 in the field in France (South), Spain and Italy Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 10-2219, Report includes Trial Nos.: 10-2219-01 10-2219-02 10-2219-03 10-2219-04 Edition Number: M-437131-01-1 Date: 2012-08-22 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.4 /04	Meilland-Berthier, I.	2012	Determination of the residues of BYI 02960 in/on grape after high or low-volume spray application of BYI 02960 SL 200 in Southern France, Spain and Italy Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2090, Report includes Trial Nos.: 11-2090-01 11-2090-02 11-2090-03 11-2090-04 Edition Number: M-438482-01-1 Date: 2012-09-18 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.5 /01	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on tomato after spraying of BYI 02960 SL 200 in the field in France (south), Italy, Spain and Portugal Bayer CropScience, Report No.: 10-2186, Report includes Trial Nos.: 10-2186-01 10-2186-02 10-2186-03 10-2186-04 Edition Number: M-438184-01-1 Date: 2012-09-06 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.5 /02	Meilland-Berthier, I.	2012	Determination of the residues of BYI 02960 in/on tomato after spray application of BYI 02960 SL 200 in the field in Spain, Italy, Portugal and Greece Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2087, Report includes Trial Nos.: 11-2087-01 11-2087-02 11-2087-03 11-2087-04 Edition Number: M-438275-01-1 Date: 2012-01-17 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.1.5 /03	Schoening, R.; Ballmann; C.	2012	Determination of the residues of BYI 02960 in/on tomato after spray application of BYI 02960 SL 200 in the greenhouse in Germany, the Netherlands, France (North) and Belgium Bayer CropScience, Report No.: 10-2190, Report includes Trial Nos.: 10-2190-01 10-2190-02 10-2190-03 10-2190-04 Edition Number: M-426300-01-1 Date: 2012-03-01 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.1.5 /04	Meilland-Berthier, I.	2012	Determination of the residues of BYI 02960 in/on tomato and cherry tomato after spray application of BYI 02960 SL 200 in the greenhouse in Germany, the Netherlands, Italy and Spain Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2085, Report includes Trial Nos.: 11-2085-01 11-2085-02 11-2085-03 11-2085-04 Edition Number: M-427056-01-1 Date: 2012-03-08 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.6 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on sweet pepper after spraying of BYI 02960 SL 200 in the field in France (south), Italy, Spain and Portugal Bayer CropScience, Report No.: 10-2187, Report includes Trial Nos.: 10-2187-01 10-2187-02 10-2187-03 10-2187-04 Edition Number: M-439089-01-1 Date: 2012-09-27 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.1.3.6 /02	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on sweet pepper after spray application of BYI 02960 SL 200 in the field in southern France, Spain and Italy Bayer CropScience, Report No.: 11-2083, Report includes Trial Nos.: 11-2083-01 11-2083-02 11-2083-03 11-2083-04 Edition Number: M-439083-01-1 Date: 2012-09-25 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.6 /03	Fargeix, G.	2012	Determination of the residues of BYI 02960 in/on sweet pepper after spraying of BYI 02960 SL 200 in the greenhouse in France, Spain, Italy, Greece and the Netherlands Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2081, Report includes Trial Nos.: 11-2081-01 11-2081-02 11-2081-03 11-2081-04 11-2081-05 11-2081-06 11-2081-07 11-2081-08 Edition Number: M-436855-01-1 Date: 2012-08-17 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.1.3.7 /01	Schulte, G.; Ruhl, S.	2012	Determination of the residues of BYI 02960 in/on cucumber after spraying of BYI 02960 SL 200 in the field in France (south), Spain and Italy Bayer CropScience, Report No.: 10-2184, Report includes Trial Nos.: 10-2184-01 10-2184-02 10-2184-03 10-2184-04 Edition Number: M-438188-01-1 Date: 2012-09-06 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.7 /02	Schulte, G.; Diehl, P.	2012	Determination of the residues of BYI 02960 in/on gherkin after spray application of BYI 02960 SL 200 in the field in southern France, Spain and Italy Bayer CropScience, Report No.: 11-2066, Report includes Trial Nos.: 11-2066-01 11-2066-02 11-2066-03 11-2066-04 Edition Number: M-438326-01-1 Date: 2012-09-10 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.7 /03	Schoening, R.; Oel, D.	2012	Determination of the residues of BYI 02960 in/on cucumber after spray application of BYI 02960 SL 200 in the greenhouse in France (South), the Netherlands, Germany and Italy Bayer CropScience, Report No.: 10-2189, Report includes Trial Nos.: 10-2189-01 10-2189-02 10-2189-03 10-2189-04 Edition Number: M-435235-01-1 Date: 2012-07-23 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.1.3.7 /04	Schulte, G.; Diehl, P.	2012	Determination of the residues of BYI 02960 in/on cucumber after spray application of BYI 02960 SL 200 in the greenhouse in Greece, Italy, Spain and Portugal Bayer CropScience, Report No.: 11-2067, Report includes Trial Nos.: 11-2067-01 11-2067-02 11-2067-03 11-2067-04 Edition Number: M-439079-01-1 Date: 2012-09-24 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.8 /01	Schoening, R.; Ballmann, C.	2012	Determination of the residues of BYI 02960 in/on melon after spray application of BYI 02960 SL 200 in the field in Spain, Italy, France (South) and Portugal Bayer CropScience, Report No.: 10-2185, Report includes Trial Nos.: 10-2185-01 10-2185-02 10-2185-03 10-2185-04 Edition Number: M-439328-01-1 Date: 2012-10-01 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.8 /02	Fargeix, G.	2012	Determination of the residues of BYI 02960 in/on water melon after spray application of BYI 02960 SL 200 in the field in Portugal, Italy and Spain Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2074, Report includes Trial Nos.: 11-2074-01 11-2074-02 11-2074-03 11-2074-04 11-2074-05 Edition Number: M-438099-01-1 Date: 2012-09-10 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.1.3.8 /03	Uceda, L.	2012	Determination of the residues of BYI 02960 in/on melon after spray application of BYI 02960 SL 200 in the greenhouse in the Netherlands, Italy and Spain Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 10-2188, Report includes Trial Nos.: 10-2188-01 10-2188-02 10-2188-03 Edition Number: M-425792-01-1 Date: 2012-02-23 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.1.3.8 /04	Fargeix, G.	2012	Determination of the residues of BYI 02960 in/on water melon after spraying application of BYI 02960 SL 200 in the greenhouse in Spain and Italy Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 11-2075, Report includes Trial Nos.: 11-2075-01 11-2075-02 11-2075-03 11-2075-04 11-2075-05 11-2075-06 Edition Number: M-437681-01-1 Date: 2012-09-04 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.1 /01	Beedle, E.; Niczyporowicz, L. M.	2012	BYI 02960 200 SL - Magnitude of the residue in/on citrus (crop group 10) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY012, Edition Number: M-433259-01-1 EPA MRID No.: 48843913 Date: 2012-06-27 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.1 /02	Woodard, D. L.; Timberlake, B.	2012	BYI 02960 200 SL - Magnitude of the residue in/on mandarin orange (CG 10) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP064, Edition Number: M-432184-01-2 EPA MRID No.: 48843914 Date: 2012-06-05 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.1 /03	Dallstream, K. A.	2012	BYI 02960 200 SL - Magnitude of the residue in/on citrus (including bridging trials to Brazil import tolerance) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP076, Edition Number: M-432687-01-1 EPA MRID No.: 48843915 Date: 2012-06-15 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.1 /04	Resende, G.	2012	Amendment 01 to the final report - Determination of residues of BYI 02960 and its metabolites, in citrus culture after drench application at the base of the plants, followed by foliar spray application of BYI 02960 (200 SL) in field trials in Brazil Bayer CropScience, São Paulo, SP, Brazil Bayer CropScience, Report No.: I11-022, Report includes Trial Nos.: I11-022-01 I11-022-02 I11-022-03 I11-022-04 I11-022-05 Edition Number: M-427041-02-3 EPA MRID No.: 48843945 Date: 2012-03-06 ...Amended: 2012-07-11 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.1 /05	Resende, G.	2012	Amendment 01 to the final report - Determination of residues of BYI 02960 and its metabolites, in citrus after foliar spray application of BYI 02960 (200 SL) in field trials in Brazil Departamento de Registro Bayer CropScience, São Paulo, Brazil Bayer CropScience, Report No.: I11-006, Report includes Trial Nos.: I11-006-03 I11-006-04 I11-006-05 I11-006-06 I11-006-07 Edition Number: M-427468-02-3 EPA MRID No.: 48843946 Date: 2012-03-09 ...Amended: 2012-07-10 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.2 /01	Niczyporowicz, L. M.; Netzband, D.	2012	BYI 02960 200 SL - Magnitude of the residue in/on tree nuts (Crop Group 14) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY016, Edition Number: M-433350-01-1 EPA MRID No.: 48843920 Date: 2012-06-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.3 /01	Lam, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on pome fruits (crop group 11) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY013, Edition Number: M-432703-01-1 EPA MRID No.: 48843916 Date: 2012-06-18 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.4 /01	Dallstream, K. A.	2012	BYI 02960 200 SL - Magnitude of the residue in/on small fruit vine climbing (except Fuzzy kiwifruit) Crop Subgroup 13-07F Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY007, Edition Number: M-432181-01-2 EPA MRID No.: 48843918 Date: 2012-06-04 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.5 /01	Dorschner, K. W.	2012	BYI 02960: Magnitude of the residue on blueberry Rutgers, The State University of New Jersey, Princeton, NJ, USA TF- BCS-IR4, Report No.: IR-4 PR No. 10637, Report includes Trial Nos.: 10637.11-AU01 10637.11-AU02 10637.11-AU04 10637.11-CL01 10637.11-CL02 10637.11-CL03 10637.11-DK01 10637.11-IT01 10637.11-MI01 10637.11-MI02 10637.11-MI03 10637.11-NC01 10637.11-NC02 10637.11-NJ01 10637.11-NJ02 10637.11-NS01 10637.11-NS02 10637.11-NS03 10637.11-NZ01 10637.11-NZ02 10637.11-OR01 10637.11-QC16 10637.11-SP01 10637.11-UK01 10637.11-UK02 Edition Number: M-435476-01-1 EPA MRID No.: 48843917 Date: 2012-07-17 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.6 /01	Dorschner, K.	1996	BYI 02960: Magnitude of the residue on prickly pear cactus U.S. Department of Agriculture, Salinas, CA, USA TF- BCS-IR4, Report No.: IR-4 PR NO. 10722, Report includes Trial Nos.: 10722.11-CA*01 10722.11-CA*02 10722.11-CA*143 10722.11-CA*144 10722.11-CA*160 10722.11-CA*161 10722.11-CA*162 10722.11-CA*163 Edition Number: M-432542-01-1 EPA MRID No.: 48843931 Date: 1996-10-24 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.7 /01	Krolski, M. E.; Harbin, A. M.	2012	BYI 02960 200 SL - Magnitude of the residue in potato - Tuberous and corm vegetables (Crop Subgroup 1C) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY015, Edition Number: M-430532-01-2 EPA MRID No.: 48843902 Date: 2012-05-08 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.7 /02	Ellis, A.	2011	Amendment no.1 - Determination of residues of BYI 02960 in potatoes and sweet potatoes following two or three applications of BYI 02960 200 SL at rates of 100, 150 or 200 g a.i./ha seven days apart Bayer CropScience Pty. Ltd., Eight Mile Plains, QLD, Australia Bayer CropScience, Report No.: BCS-0352, Edition Number: M-415292-02-1 EPA MRID No.: 48843940 Date: 2011-09-30 ...Amended: 2012-07-09 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.7 /03	Ellis, A.	2012	Amendment no.1 - Determination of residues of BYI 02960 in potatoes and sweet potatoes following two or three applications of BYI 02960 200 SL at rates of 100, 150 or 200 g a.i./ha seven days apart Bayer CropScience Pty. Ltd., Residue Laboratory, Eight Mile Plains, QLD, Australia Bayer CropScience, Report No.: BCS-0358, Report includes Trial Nos.: C538 C539 C541 C632 Edition Number: M-426841-02-1 EPA MRID No.: 48843941 Date: 2012-02-27 ...Amended: 2012-07-09 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.8 /01	Miller, A.; Helfrich, K. K.	2012	BYI 02960 200 SL - Magnitude of the residue in/on fruiting vegetables (CG 8); US / Canada import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY022, Edition Number: M-433126-01-1 EPA MRID No.: 48843911 Date: 2012-06-22 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.8 /02	Ellis, A.	2011	Amendment no.1 - Determination of residues of BYI 02960 following three foliar applications of BYI 02960 200 SL to trellis and bush tomatoes at rates of 100, 150 or 200 g a.i./ha seven days apart, and in glass house tomatoes at rates of 10, 15 and 20 g a.i./100L seven days apart Bayer CropScience Pty. Ltd., Residue Laboratory, Eight Mile Plains, QLD, Australia Bayer CropScience, Report No.: BCS-0348.02, Edition Number: M-411773-02-1 EPA MRID No.: 48843936 Date: 2011-07-20 ...Amended: 2012-07-09 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.8 /03	Ellis, A.	2011	Amendment no. 1 - Determination of residues of BYI 02960 following three foliar applications of BYI 02960 200 SL to capsicums at rates of 100, 150 or 200 g a.i./ha seven days apart Bayer CropScience Pty. Ltd., Residue Laboratory, Eight Mile Plains, QLD, Australia Bayer CropScience, Report No.: BCS-0349.02, Edition Number: M-430274-02-1 EPA MRID No.: 48843937 Date: 2011-08-11 ...Amended: 2012-07-09 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.8 /04	Ellis, A.	2012	Determination of residues of BYI 02960 following three foliar applications of BYI 02960 200 SL to trellis and bush tomatoes at rates of 100, 150 or 200 g a.i./ha seven days apart, and in glass house tomatoes at rates of 10, 15 and 20 g a.i./ 100 L seven days apart - Amendment no. 1 to the report BCS-0354 Bayer CropScience Pty. Ltd., Residue Laboratory, Eight Mile Plains, QLD, Australia Bayer CropScience, Report No.: BCS-0354, Report includes Trial Nos.: C525 C526 C683 Edition Number: M-433790-02-1 EPA MRID No.: 48843938 Date: 2012-05-31 ...Amended: 2012-07-09 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.8 /05	Ellis, A.	2012	Determination of residues of BYI 02960 following three foliar applications of BYI 02960 200 SL to capsicums at rates of 100, 150 or 200 g a.i./ha seven days apart - Amendment no. 1 to the report BCS-0355 Bayer CropScience Pty. Ltd., Residue Laboratory, Eight Mile Plains, QLD, Australia Bayer CropScience, Report No.: BCS-0355, Edition Number: M-432144-02-1 EPA MRID No.: 48843939 Date: 2012-05-11 ...Amended: 2012-07-09 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.9 /01	Netzband, D.; Niczyporowicz, L. M.	2012	BYI 02960 SL 200 - Magnitude of the residue in/on leafy vegetables (Crop Group 4) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY005, Edition Number: M-433317-01-1 EPA MRID No.: 48843904 Date: 2012-06-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.10 /01	Hoag, R. E.; Arthur, E. L.; Woodard, D. L.	2012	BYI 02960 200 SL - Magnitude of the residue in/on dried, shelled pea and bean (except soybean), foliage of legume vegetables (except soybean); (CG 6C and 7A) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY028, Edition Number: M-433260-01-1 EPA MRID No.: 48843909 Date: 2012-06-26 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.11 /01	Krolski, M. E.; Harbin, A. H.	2012	BYI 02960 200 SL - Magnitude of the residue in peanut Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY010, Edition Number: M-424313-01-2 EPA MRID No.: 48843930 Date: 2012-01-30 GLP/GEP: no, unpublished	N	Bayer
KIIA 6.3.2.12 /01	Sturdivant, D. W.	2012	BYI 02960 200 SL and BYI 02960 480 FS - Magnitude of the residue in/on soybeans Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY011, Edition Number: M-431214-01-2 EPA MRID No.: 48843910 Date: 2012-05-15 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.13 /01	Timberlake, B. C.; Harbin, A. M.	2012	BYI 02960 200 SL and BYI 02960 480 FS - Magnitude of the residue in/on cotton (Crop Subgroup 20C) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY009, Edition Number: M-431910-01-2 EPA MRID No.: 48843925 Date: 2012-06-01 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.14 /01	Hoag, R. E.	2012	BYI 02960 200 SL and BYI 02960 480 FS - Magnitude of the residue in/on barley Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY001, Edition Number: M-431905-01-2 EPA MRID No.: 48843921 Date: 2012-04-19 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.15 /01	Fisher, D. R.	2012	BYI 02960 200 SL and BYI 02960 480 FS - Magnitude of the residue in/on corn Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY002, Edition Number: M-432754-01-1 EPA MRID No.: 48843922 Date: 2012-06-18 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.16 /01	Krolski, M. E.; Dallstream, K. A.	2012	BYI 02960 200 SL and BYI 02960 480 FS - Magnitude of the residue in/on sorghum Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY004, Edition Number: M-427048-01-2 EPA MRID No.: 48843923 Date: 2012-03-14 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.17 /01	Fischer, D. R.; Niczyporowicz, L. M.	2012	BYI 02960 200 SL and BYI 02960 480 FS - Magnitude of the residue in/on wheat Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY003, Edition Number: M-433258-01-1 EPA MRID No.: 48843924 Date: 2012-06-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.18 /01	Hoag, R. E.	2012	BYI 02960 200 SL - Magnitude of the residue in/on coffee; U.S., Canada and E.U. import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP074, Edition Number: M-433257-01-1 EPA MRID No.: 48843928 Date: 2012-06-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.3.2.18 /02	Resende, G.	2012	Amendment 01 to the final report - Determination of residues of BYI 02960 and its metabolites, in coffee after drench application at the base of the plants, followed by foliar application of BYI 02960 (200 SL) in field trials in Brazil Departamento de Registro Bayer CropScience, São Paulo, Brazil Bayer CropScience, Report No.: I11-008, Report includes Trial Nos.: I11-008-01 I11-008-02 I11-008-04 I11-008-05 Edition Number: M-427469-03-2 EPA MRID No.: 48843944 Date: 2012-03-09 ...Amended: 2012-07-02 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.3.2.19 /01	Krolski, M. E.	2012	BYI 02960 200 SL - Magnitude of the residue in/on hops Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY008, Report includes Trial Nos.: RV047-11HA RV048-11HA RV049-11HA Edition Number: M-432695-01-1 EPA MRID No.: 48843929 Date: 2012-06-12 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.4.1 /01	[redacted]	2012	BYI 02960 - Magnitude of the residue in laying hens [redacted] Report No.: RARVP041, Edition Number: M-428933-01-1 Date: 2012-04-05 GLP/GEP: yes, unpublished ...also filed: KIIA 4.3 /09	Y	Bayer
KIIA 6.4.2 /01	[redacted]	2012	BYI 02960 - Magnitude of the residue in dairy cows - Amended report [redacted] Report No.: RARVP050-1, Edition Number: M-428416-02-1 EPA MRID No.: 48843842 Date: 2012-04-03 ...Amended: 2012-05-31 GLP/GEP: yes, unpublished ...also filed: KIIA 4.3 /08 ...also filed: KIIA 6.1.1 /02	Y	Bayer
KIIA 6.5.1 /01	Weber, E.	2011	Nature of the residues of [pyridinylmethyl-14C]BYI02960 in processed commodities - High temperature hydrolysis Bayer CropScience, Report No.: MEF-10/856, Edition Number: M-402311-01-2 EPA MRID No.: 48843966 Date: 2011-02-09 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4 /01	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on lettuce and head lettuce and the processed fractions (head, inner parts; leaf, outer; leaf, inner; leaf, inner, washed and washings) after spraying of BYI 02960 SL 200 in the field in the Netherlands, Belgium and Germany Bayer CropScience, Report No.: 10-3223, Report includes Trial Nos.: 10-3223-01 10-3223-02 10-3223-04 10-3223-05 Edition Number: M-426982-01-2 EPA MRID No.: 48843947 Date: 2012-03-07 GLP/GEP: no, unpublished ...also filed: KIIA 6.5.4.1 /01	N	Bayer
KIIA 6.5.4 /02	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on hop (cone, green and cone, kiln-dried) and the processed fractions (hops draff, brewer's yeast and beer) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3407, Report includes Trial Nos.: 10-3407-01 10-3407-02 Edition Number: M-425311-01-1 Date: 2012-02-13 GLP/GEP: yes, unpublished ...also filed: KIIA 6.5.4.2 /01	N	Bayer

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KIIA 6.5.4.1 /01	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on lettuce and head lettuce and the processed fractions (head, inner parts; leaf, outer; leaf, inner; leaf, inner, washed and washings) after spraying of BYI 02960 SL 200 in the field in the Netherlands, Belgium and Germany Bayer CropScience, Report No.: 10-3223, Report includes Trial Nos.: 10-3223-01 10-3223-02 10-3223-04 10-3223-05 Edition Number: M-426982-01-2 EPA MRID No.: 48843947 Date: 2012-03-07 GLP/GEP: no, unpublished ...also filed: KIIA 6.5.4 /01	N	Bayer
KIIA 6.5.4.2 /01	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on hop (cone, green and cone, kiln-dried) and the processed fractions (hops draff, brewer's yeast and beer) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3407, Report includes Trial Nos.: 10-3407-01 10-3407-02 Edition Number: M-425311-01-1 Date: 2012-02-13 GLP/GEP: yes, unpublished ...also filed: KIIA 6.5.4 /02	N	Bayer
KIIA 6.5.4.3 /01	Schulte, G.; Teubner, L.	2012	Processing Study - Determination of the residues of BYI 02960 in/on orange (fruit and pulp) and the processed fractions (fruit, stored; whole fruit, washed; washings; raw juice; pomace, wet; pomace, dried; juice; marmalade; pulp; peel; peel washed; peel without oil; oil) after spraying of BYI 02960 SL 200 in the field in Spain Bayer CropScience, Report No.: 10-3405, Report includes Trial Nos.: 10-3405-01 10-3405-02 Edition Number: M-439410-01-1 Date: 2012-10-01 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4.3 /02	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on orange processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY035, Edition Number: M-432186-01-2 EPA MRID No.: 48843954 Date: 2012-06-01 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.4 /01	Schulte, G.; Bauer, J.	2012	Amendment No. 1 - Determination of the residues of BYI 02960 in/on apple and the processed fractions (whole fruit, washed; washings; raw sauce; strain rest; sauce; pomace, wet; pomace, dried; raw juice; juice, retentate; peel; fruit peeled; fruit, dried) after spraying of BYI 02960 SL 200 in the field in Germany and Belgium Bayer CropScience, Report No.: 10-3171, Report includes Trial Nos.: 10-3171-03 10-3171-06 Edition Number: M-434004-02-1 Date: 2012-07-05 ...Amended: 2012-09-28 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.4 /02	Schulte, G.; Ballmann, C.	2012	Determination of the residues of BYI 02960 in/on apple and the processed fractions (whole fruit, washed; washings; raw sauce; strain rest; sauce; pomace, wet; pomace, dried; raw juice; retentate; and juice) after spray application of BYI 02960 SL 200 in the field in Italy and Spain Bayer CropScience, Report No.: 10-3172, Report includes Trial Nos.: 10-3172-03 10-3172-06 Edition Number: M-434006-01-2 Date: 2012-07-05 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4.5 /01	Noss, G.; Teubner, L.	2012	Determination of the residues of BYI 02960 in/on peach and the processed fractions (whole fruit, washed; washings; peel; fruit, peeled and preserve) after spraying of BYI 02960 SL of BYI 02960 SL 200 in the field in Spain and Italy Bayer CropScience, Report No.: 10-3216, Report includes Trial Nos.: 10-3216-02 10-3216-03 Edition Number: M-439376-01-1 Date: 2012-10-08 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.6 /01	Noss, G.; Bauer, J.; Ruhl, S.	2011	Determination of the residues of BYI 02960 in/on grape and the processed fractions (pomace, grape; must; wine at bottling; wine at first taste test, juice, pasteurised; jelly; washings; raisin waste; raisin) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3406, Report includes Trial Nos.: 10-3406-01 10-3406-02 10-3406-03 10-3406-04 Edition Number: M-433545-01-1 EPA MRID No.: 48843953 Date: 2011-09-07 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.7 /01	Schulte, G.; Ruhl, S.	2012	Processing study - Determination of the residues of BYI 02960 in/on beet, sugar and processed fractions (body, washed; washings; pulp; raw juice; thin juice; cake, lime; thick juice; molasses; raw sugar; white sugar; pulp, extracted, wet; press liquor; pulp, extracted, pressed; pulp, extracted, dry; pulp, extracted, ensiled; and refined sugar) after spraying and incorporation of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3408, Report includes Trial Nos.: 10-3408-01 10-3408-02 Edition Number: M-439824-01-1 Date: 2012-10-18 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4.8 /01	Schulte, G.; Ballmann, C.	2012	Amendment No. 2 - Determination of the residues of BYI 02960 in/on tomato and the processed fractions (whole fruit, washed; washings; strain rest; raw juice; juice; raw puree; puree; paste; peel; peeling water; fruit peeled; preserve and tomato, dried) after spraying of BYI 02960 SL 200 in the field in southern France, Italy, Spain and Portugal Bayer CropScience, Report No.: 10-3186, Report includes Trial Nos.: 10-3186-01 10-3186-02 10-3186-03 10-3186-04 Edition Number: M-427003-03-1 Date: 2012-03-08 ...Amended: 2012-09-28 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.9 /01	Schulte, G.; Ruhl, S.	2012	Determination of the residues of BYI 02960 in/on cucumber and the processed fractions (washings; whole fruit, washed; preserve; brine; and fruit, fermented) after spraying of BYI 02960 SL 200 in the field in France (south) - Processing study Bayer CropScience, Report No.: 10-3184, Report includes Trial Nos.: 10-3184-01 10-3184-04 Edition Number: M-438180-01-1 Date: 2012-09-06 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.10 /01	Schulte, G.; Ruhl, S.	2012	Processing study - Determination of the residues of BYI 02960 in/on spring barley and the processed fractions (malt sprouts; brewer's malt; brewer's grain, hops draff; brewer's yeast; beer; pearl barley; pearl barley rub off) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3410, Report includes Trial Nos.: 10-3410-01 10-3410-02 Edition Number: M-439853-01-1 Date: 2012-10-16 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4.11 /01	Schulte, G.; Bauer, J.	2012	Processing study - Determination of the residues of BYI 02960 in/on wheat and the processed fractions (semolina; semolina bran; whiteflour bran; white flour; white bread; whole meal; wholemeal bread and wheat germ) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3409, Report includes Trial Nos.: 10-3409-01 10-3409-02 Edition Number: M-440307-01-1 Date: 2012-10-30 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.11 /02	Lenz, C.; Fischer, D. R.	2012	BYI 02960 200 SL - Magnitude of the residue in/on wheat processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY031, Edition Number: M-427047-01-2 EPA MRID No.: 48843959 Date: 2012-03-13 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.12 /01	Hoag, R. E.	2012	BYI 02960 200 SL - Magnitude of the residue in/on processed commodities for coffee; U.S., Canada and E.U. import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP075, Edition Number: M-433200-01-1 EPA MRID No.: 48843950 Date: 2012-06-26 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.13 /01	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on field corn processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY030, Edition Number: M-424774-01-1 EPA MRID No.: 48843951 Date: 2012-02-09 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4.14 /01	Lenz, C.; Beedle, E.	2012	BYI 02960 200 SL - Magnitude of the residue in cotton processed commodities (crop subgroup 20C) Bayer Corporation, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY033, Edition Number: M-433122-01-1 EPA MRID No.: 48843952 Date: 2012-06-22 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.15 /01	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on peanut processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY032, Report includes Trial Nos.: RV274-10PA RV275-10PA Edition Number: M-430523-01-2 EPA MRID No.: 48843955 Date: 2012-05-08 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.16 /01	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on potato processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY038, Edition Number: M-430542-01-2 EPA MRID No.: 48843956 Date: 2012-05-07 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.5.4.17 /01	Lenz, C.	2012	BYI 02960 200 SL - Magnitude of the residue in/on soybean processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY029, Report includes Trial Nos.: RV268-10PA RV269-10PA Edition Number: M-428939-01-2 EPA MRID No.: 48843957 Date: 2012-04-04 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.5.4.18 /01	Fischer, D. R.	2012	BYI 02960 200 SL - Request for Waiver of the requirements for the BYI 02960 magnitude of the residue in sugarcane processed commodities in Florida (rotational crop regional tolerance) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVX001, Report includes Trial Nos.: RV288-10PA Edition Number: M-432700-01-1 EPA MRID No.: 48843960 Date: 2012-06-18 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.2 /01	A. Klempner	2011	Metabolism of [furanone-4-14C]BYI 02960 in confined rotational crops Bayer CropScience, Report No.: MEF-11/365, Edition Number: M-421861-01-3 EPA MRID No.: 48843961 Date: 2011-12-16 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.2 /02	Breuer-Rehm, M.	2011	Metabolism of [pyridinylmethyl-14C]BYI 02960 in confined rotational crops Bayer CropScience, Report No.: MEF-10/892, Edition Number: M-419853-02-1 EPA MRID No.: 48843962 Date: 2011-12-02 ...Amended: 2012-02-22 GLP/GEP: yes, unpublished	N	Bayer

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KIIA 6.6.3 /01	Schoening, R.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on the field rotational crops barley, carrot, lettuce and turnip after spray application of BYI 02960 SL 200 on lettuce and soil in the field in Germany, the Netherlands, France (South) and Spain Bayer CropScience, Report No.: 10-2503, Report includes Trial Nos.: 10-2503-01 10-2503-02 10-2503-03 10-2503-04 Edition Number: M-429091-02-1 Date: 2012-04-11 ...Amended: 2013-01-24 GLP/GEP: yes, unpublished ...also filed: KIIA 6.6.3.1.1 /01	N	Bayer
KIIA 6.6.3.1.1 /01	Schoening, R.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on the field rotational crops barley, carrot, lettuce and turnip after spray application of BYI 02960 SL 200 on lettuce and soil in the field in Germany, the Netherlands, France (South) and Spain Bayer CropScience, Report No.: 10-2503, Report includes Trial Nos.: 10-2503-01 10-2503-02 10-2503-03 10-2503-04 Edition Number: M-429091-02-1 Date: 2012-04-11 ...Amended: 2013-01-24 GLP/GEP: yes, unpublished ...also filed: KIIA 6.6.3 /01	N	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KIIA 6.6.3.1.2 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on the field rotational crop potato after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, the Netherlands, Spain and Italy - Limited rotational crop study Bayer CropScience, Report No.: 11-2550, Report includes Trial Nos.: 11-2550-01 11-2550-02 11-2550-03 11-2550-04 Edition Number: M-438341-01-1 Date: 2012-09-10 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.3.1.3 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on the field rotational crop leek after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, Germany, Spain and Italy - Limited rotational crop study Bayer CropScience, Report No.: 11-2551, Report includes Trial Nos.: 11-2551-01 11-2551-02 11-2551-03 11-2551-04 Edition Number: M-438384-01-1 Date: 2012-09-13 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.3.1.4 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on the field rotational crop cucumber after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, Germany, Spain and Italy - Limited rotational crop study Bayer CropScience, Report No.: 11-2552, Report includes Trial Nos.: 11-2552-01 11-2552-02 11-2552-03 11-2552-04 Edition Number: M-438343-01-1 Date: 2012-09-13 GLP/GEP: yes, unpublished	N	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KIIA 6.6.3.1.5 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on the field rotational crop onion after spray application of BYI 02960 SL 200 on bare soil in the field in France (North), Germany, Italy and Spain - Limited rotational crop study Bayer CropScience, Report No.: 11-2553, Report includes Trial Nos.: 11-2553-01 11-2553-02 11-2553-03 11-2553-04 Edition Number: M-438397-01-1 Date: 2012-09-14 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.3.1.6 /01	Schulte, G.	2012	Amendment no. 1 to report no: 11.2555 - Determination of the residues of BYI 02960 in/on the field rotational crop french bean after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, Germany, Italy and Spain - Limited rotational crop study Bayer CropScience, Report No.: 11-2555, Report includes Trial Nos.: 11-2555-01 11-2555-02 11-2555-03 11-2555-04 Edition Number: M-438394-02-1 Date: 2012-09-14 ...Amended: 2012-09-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.3.1.7 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on the field rotational crop pea after spray application of BYI 02960 SL 200 on bare soil in the field in France (North), Germany, Italy and Spain - Limited rotational crop study Bayer CropScience, Report No.: 11-2556, Report includes Trial Nos.: 11-2556-01 11-2556-02 11-2556-03 11-2556-04 Edition Number: M-438582-01-1 Date: 2012-09-17 GLP/GEP: yes, unpublished	N	Bayer

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
KIIA 6.6.3.1.8 /01	Schulte, G.	2012	Determination of the residues of BYI 02960 in/on the field rotational crop rape after spray application of BYI 02960 SL 200 on bare soil in the field in nothern France, Germany, Italy and Spain Bayer CropScience, Report No.: 11-2554, Report includes Trial Nos.: 11-2554-01 11-2554-02 11-2554-03 11-2554-04 Edition Number: M-443092-01-1 Date: 2012-12-07 GLP/GEP: yes, unpublished	N	Bayer
KIIA 6.6.3.2 /01	Krolski, M. E.; Dallstream, K. A.	2012	BYI 02960, fenamidone, fluopyram, and spiromesifen - Magnitude of the residue in sugarcane in Florida (rotational crop regional Tolerance) Bayer CropScience LP, RTP, NC, USA Bayer CropScience, Report No.: RARVP030, Report includes Trial Nos.: RV001-11RA RV002-11RA RV286-10RA RV287-10RB Edition Number: M-432179-01-2 EPA MRID No.: 48843964 Date: 2012-05-25 GLP/GEP: yes, unpublished	N	Bayer

List of data submitted by the applicant and not relied on

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
-	-	-	-	-	-

List of data relied on and not submitted by the applicant but necessary for evaluation

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
-	-	-	-	-	-

Appendix 2 Detailed evaluation of the additional studies relied upon

A 2.1 Flupyradifurone

A 2.1.1 Stability of residues

A 2.1.1.1 Stability of residues during storage of samples

A 2.1.1.1.1 Storage stability of residues in plant products

The stability of flupyradifurone-related residues during freezer storage up to 52 months (final storage interval) is summarized in the study below.
Within some residue studies on oilseed rape the temperatures during storage increased. Therefore, two storage stability studies at exaggerated temperatures were performed and summarized within this submission.

A 2.1.1.1.1.1 Study report RARVP046-2

Comments of zRMS:	To support the residue field trials in a wide variety of crops (orange fruit (high acid content representative), spinach leaves and tomato fruit (high water content representatives), wheat grain (high starch content representative), navy bean seed (high protein content representative), coffee and soybean seed (high oil content representatives), and sugar cane), the storage stability of BYI 02960 and its major metabolites, difluoroacetic acid (DFA) and difluoroethyl-amino-furanone (DFEAF), was investigated. Sample analyses for BYI 02960, DFA, and DFEAF were performed with the analytical method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The method was successfully validated in dried beans, forage, orange fruit, soybean seeds, tomato fruit, and wheat grain in that study with the limits of quantitation (LOQs) of 0.010 mg/kg for flupyradifurone and DFEAF and 0.05 mg/kg for DFA (expressed in flupyradifurone equivalents) in all matrices. The mean of the recoveries for each matrix were in an acceptable range (lowest mean recovery was 72% whereas the highest mean recovery was 114%). Residues of BYI 02960, DFA, and DFEAF in all plant matrices were stable (<30% decomposition) during 52 months (1566 to 1572 days) of frozen storage. The study was conducted to comply with the current data requirements (OECD Guideline for the Testing of Chemicals No. 506). The study is acceptable.

Reference:	**KCA 6.1/01**
Title:	Storage stability of BYI 02960, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices
Report:	Netzband, D.; 2015; M-428412-03-1
Authority registration No:	
Guideline(s):	EPA Ref.: OPPTS 860.1380, Storage Stability Data OECD Guideline for the Testing of Chemicals No. 506, Stability of Pesticide Residues in Stored Commodities, Oct. 2007. PMRA Residue Chemistry Guidelines, Regulatory Directive 98-02, Section 5, Storage Stability Data, June 1998
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
To determine the freezer storage stability of the relevant residues of flupyradifurone in plant materials, individual 5-g control samples of orange fruit (high acid content), spinach leaves and tomato fruit (high

water content), wheat grain (high starch content), bean seed (high protein content), coffee bean and soybean seed (high oil content), and sugar cane were separately spiked with either flupyradifurone parent compound, DFA, or DFEAF at a fortification level of 1.0 mg/kg of each analyte. Except for the day -0 analysis, samples were stored in glass containers in a freezer at an average temperature of ≤ - 18°C. Although several short-duration temperatures ≥ 0°C were observed (6.5°C, 4.2°C, and 0.0°C, at different days, respectively), these were too brief to allow samples to thaw as documented by average temperatures not higher than -19°C on those days.
For day -0 analysis, three treated samples of each material were chosen, as well as one control sample of each. Stored samples were analyzed after intervals of 1, 2½, 5-6, 12, 18, 35 and 52 months.

At each storage interval, three 5-gram aliquots of the stored control (unfortified) and two 5-g aliquots of fortified crop matrix were removed from storage. Two of the control samples were fortified at 1.0 mg/kg on the day of extraction to be used as concurrent recoveries. Samples were extracted by blending an aliquot of the matrix with a mixture of ACN/water (4:1, v/v) with 2.2 mL/L formic acid. The extract was diluted, and an aliquot was evaporated to near dryness, reconstituted, and purified through a C-18 SPE column. The eluent was diluted and amended with a mixture of stable, isotopically labelled internal standards. An aliquot was analyzed by high performance liquid chromatography/triple stage quadrupole mass spectrometry (LC-MS/MS).

Quantitation of each analyte was based on the daughter ion transitions of the analytes and the respective internal standard analogs. The responses of the LC-MS/MS system to each analyte and its internal standard were measured in samples and in standards, and a relative response was calculated (calculated as the ratio of the analyte and the stable isotopically labelled internal standard responses). The relative response of the analyte in each sample was compared to the relative response of the analyte in the standards.

Flupyradifurone and its metabolites were analytically determined using analytical method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The method was successfully validated in dried beans, forage, orange fruit, soybean seeds, tomato fruit, and wheat grain in that study. The limits of quantitation (LOQs) were 0.010 mg/kg for flupyradifurone and DFEAF and 0.05 mg/kg for DFA (expressed in flupyradifurone equivalents) in all matrices.

Results and discussions
No flupyradifurone or DFEAF interferences greater than the LOQ were observed in any of the control extracts from any of the matrices analyzed during this study. Apparent DFA residues of 0.01 mg/kg were observed in orange fruit and tomato fruit, and an apparent residue of 0.02 mg/kg was observed in wheat grain. Stored samples and concurrent recoveries were not corrected for interferences in corresponding controls.

Summaries of concurrent recoveries conducted as a part of the study are presented in Table A 1. The mean of the recoveries for each matrix were in an acceptable range (lowest mean recovery was 72% whereas the highest mean recovery was 114%).
Summaries of residues recovered in stored samples are presented in Table A 2 for flupyradifurone and DFA (The recoveries of metabolite DFEAF are not shown in the table since this metabolite is not part of the residue definition).
Residues of flupyradifurone and DFA were stable (<30% decomposition) in spinach, orange fruit, navy bean, sugarcane, green coffee bean, tomato fruit, and wheat grain for approx. 52 months (1566 to 1572 days) of frozen storage. Only for DFA a slight decrease of residues could be presumed in soybean seeds based on the recovery values after storage (71% and 55%). However, when correcting the recoveries of the stored samples with the procedural recoveries (concurrent recoveries), the stability of DFA was also shown in a matrix with high oil content when stored for 52 months at ≤ -18°C. The corrected mean recovery amounted to 88%.

Table A 1: Summary of concurrent recoveries of flupyradifurone and DFA in plant matrices

Matrix	Spike level (mg/kg)	Storage Interval (months)	Sample size (n)	Individual procedural recoveries (%)	Mean ± RSD (%)
Flupyradifurone					
Spinach	1.0	35	2	98, 106	102
		52	2	94, 100	97
Tomato fruit	1.0	35	2	98, 105	102
		52	2	87, 89	88
Orange fruit	1.0	35	2	114, 114	114
		52	2	92, 93	92
Soybean seed	1.0	35	2	103, 102	102
		52	2	80, 97	89
Navy bean	1.0	35	2	110, 95	103
		52	2	92, 104	98
Sugarcane	1.0	35	2	98, 110	104
		52	2	90, 87	89
Coffee green bean	1.0	35	2	90, 106	98
		52	2	85, 86	86
Wheat grain	1.0	35	2	96, 96	96
		52	2	90, 79	85
DFA					
Spinach	1.0	35	2	98, 110	104
		52	2	94, 100	97
Tomato fruit	1.0	35	2	108, 108	108
		52	2	94, 93	94
Orange fruit	1.0	35	2	112, 108	110
		52	2	92, 94	93
Soybean seed	1.0	35	2	83, 81	82
		52	2	71, 73	72
Navy bean	1.0	35	2	92, 92	92
		52	2	87, 83	85
Sugarcane	1.0	35	2	100, 115	108
		52	2	88, 87	88
Coffee green bean	1.0	35	2	83, 88	86
		52	2	80, 83	82
Wheat grain	1.0	35	2	83, 87	85
		52	2	80, 82	81

Table A 2: Stability of flupyradifurone and DFA residues in plant matrices following storage at ≤ -18°C

Matrix	Spike level (mg/kg)	Storage interval (months)	Individual recovered residues (mg/kg)	Individual recoveries (%)
Flupyradifurone				
Spinach	1.0	35	1.043, 1.038	104, 104
		52	0.856, 0.803	86, 80
Tomato fruit	1.0	35	0.975, 1.045	98, 105
		52	0.895, 0.861	90, 86
Orange fruit	1.0	35	1.117, 1.101	112, 110
		52	0.884, 0.903	88, 90
Soybean seed	1.0	35	1.089, 1.070	109, 107
		52	0.857, 0.729	86, 73
Navy bean	1.0	35	1.208, 1.326	121, 133
		52	1.030, 1.040	103, 104
Sugarcane	1.0	35	1.083, 0.975	108, 98
		52	0.779, 0.938	78, 94
Coffee green bean	1.0	35	0.946, 0.954	95, 95
		52	0.805, 0.842	81, 84
Wheat grain	1.0	35	0.740, 0.883	74, 88
		52	0.843, 0.815	84, 82
DFA				
Spinach	1.0	35	0.954, 1.031	95, 103
		52	0.944, 0.914	94, 91
Tomato fruit	1.0	35	1.084, 0.971	108, 97
		52	0.896, 0.894	90, 89
Orange fruit	1.0	35	1.123, 1.005	112, 100
		52	0.887, 0.866	89, 87
Soybean seed	1.0	35	0.830, 0.702	83, 70
		52	0.707, 0.547	71, 55
Navy bean	1.0	35	1.014, 1.080	101, 108
		52	0.724, 0.803	72, 80
Sugarcane	1.0	35	0.946, 1.036	95, 104
		52	0.785, 0.834	79, 83
Coffee green bean	1.0	35	0.869, 0.847	87, 85
		52	0.755, 0.776	76, 78
Wheat grain	1.0	35	0.787, 0.755	79, 75
		52	0.735, 0.747	74, 75

Conclusion

Residues of flupyradifurone and DFA in the plant matrices of orange fruit, spinach leaves, tomato fruit, wheat grain, navy bean seed, coffee, soybean seed, and sugar cane were stable (<30% decomposition) during 52 months (1566 to 1572 days) of frozen storage. As a consequence, the stability of

flupyradifurone and its metabolite DFA was shown in the different matrix types (high water-, high oil-, high protein-, high starch- and high acid-content matrices) and confirms the validity of the residue results in the presented residue trials.

A 2.1.1.1.1.2 Study report S13-03307

Comments of zRMS:	According to the storage stability results it can be concluded that residues of flupyradifurone and DFA in the plant matrices of high water, high acid, high starch and high protein content were stable (<30% decomposition) after storage at + 1°C for 8 hours, following a storage of 6 - 8 days at -7°C. The study is acceptable.

Reference:	**KCA 6.1/02**
Title:	Amendment no. 3 to final report - 7 days freezer storage stability study with different combinations of a total of 61 analytes (parent and metabolite molecules) and five matrix types (high water / acidic / starch / protein / oil)
Report:	Lakaschus, S.; Gizler, A.; 2017; S13-03307; M-480441-06-1
Authority registration No:	
Guideline(s):	Commission Regulation (EU) No 544/2011 of 10 June 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the data requirements for active substances US EPA Residue Chemistry Test Guideline OPPTS 860.1380: Storage Stability Data OECD Test Guideline 506, adopted 16 October 2007
Deviations:	see report
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

To determine the storage stability of relevant residues of flupyradifurone for a period of 8 hours at +1°C followed by 6-8 days of storage at -7°C in tomato fruit, wheat green material (high water content), grape bunches (high acid content), wheat grain, potato tuber (high starch content) and dry peas (high protein content. Individual 5-g control samples of the respective sample material were spiked with a mixture of the relevant analytes flupyradifurone parent compound (separately) or DFEAF, DFA and 6-CNA (jointly) at a fortification level of 1.0 mg/kg of each analyte.
(Results of DFEAF and 6-CNA are not shown in this summary since these metabolites are not a part of the residue definition; for details please refer to the report).

Except for the day-0 analysis, samples were stored in plastic containers at an average temperature of +1°C for eight hours, following 6-8 days at -7°C. For day-0 analysis, five treated samples of each material were chosen, as well as two control samples of each. Samples were then also analyzed after 6, 7 or 8 days of storage.
At each storage interval, five stored samples were analyzed. In addition two concurrent recoveries per commoditiy were conducted.
Flupyradifurone was analytically determined using analytical method 01207 (Schoening, R.; Willmes, J.; 2014; M-476845-01) and its metabolites DFA, DFEAF and 6-CNA were analytically determined using analytical method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02).
For the analysis of flupyradifurone, samples were adjusted with water followed by addition of acetonitrile leading to an acetonitrile/water ratio of (4/1, v/v). The samples fortified with flupyradifurone were left to soak for 15 minutes and then shaken for 15 minutes to enhance the extraction. Afterwards, a salt mixture (Mg_2SO_4/NaCl/Na_3 citrate 2 H_2O/Na_2H citrate 6 H_2O) (4/1/1/0.5, w/w/w/w)) was added and the extracts were centrifuged. The extracts were submitted to LC-MS/MS-analysis and quantification was done using matrix-matched standards.
For DFA the samples were extracted twice with acidified acetonitrile/water (4/1, v/v). After centrifugation the clear supernatant was transferred into a volumetric flask and filled up to volume. An aliquot of the crude extract was evaporated to the aqueous reminder and purified using Bond Elut C_{18} SPE cartridges followed by the addition of internal standards. The final extracts were submitted to LC-MS/MS

analysis with quantification using matrix-matched standards.

The methods were successfully validated at a level of 1 mg/kg relevant to this study in/on samples of tomato (fruit), wheat (green material), grape (bunches), wheat (grain), potato (tuber) and peas (dry peas) in the presented study. For further information on validation of the two methods please refer to Section 5, Appendix A 2.1.1.1).

Results and discussions
In the control samples, the residues were always below 30% of the LOQ.
Summaries of concurrent recoveries conducted as a part of the study are presented in Table A 3. The mean of the recoveries for each matrix were in an acceptable range (mean recoveries were 74% to 105%). The RSDs were in the range of 5.2 to 20%.

Summaries of residues recovered in stored samples are presented in Table A 4 for flupyradifurone and DFA.

Residues of flupyradifurone and DFA were stable (<30% decomposition) in tomato fruit, wheat green material, grape bunches, wheat grain, potato tuber and dry peas after storage for 8 hours at +1°C followed by 6-8 days of storage at -7°C frozen storage.
For flupyradifurone in dry peas the mean recovery in stored samples was at 69% (day 6 analysis). However, after correction for concurrent recoveries, flupyradifurone in dry peas can be considered as stable. The corrected mean recovery amounted to 88%.

Table A 3: Summary of concurrent recoveries of flupyradifurone and DFA in plant matrices

Matrix	Spike level (mg/kg)	Storage Interval (days)	Sample size (n)	Individual procedural recoveries (%)	Mean ± RSD
Flupyradifurone					
Tomato fruit	1.0	0	5	83, 106, 82, 84, 88	89±11
		8	2	86, 83	85
Wheat green material	1.0	0	5	90, 91, 94, 101, 103	96±6.1
		8	2	76, 72	74
Grapes bunches	1.0	0	5	79, 93, 97, 95, 109	95±11
		7	2	96, 88	92
Wheat grain	1.0	0	5	78, 94, 95, 100, 103	94±10
		7	2	76, 81	79
Potato tuber	1.0	0	5	91, 94, 95, 84, 87	90±5.2
		7	2	64, 93	79
Peas, dry	1.0	0	5	117, 107, 86, 132, 81	105±20
		6	2	78, 79	79
DFA					
Tomato fruit	1.0	0	5	102, 79, 68, 99, 84	86±16
		7	2	105, 96	101
Wheat green material	1.0	0	5	89, 80, 77, 61, 75	76±13
		7	2	83, 110	97
Grapes bunches	1.0	0	5	69, 90, 73, 99, 107	88±19
		7	2	85, 92	89
Wheat grain	1.0	0	5	99, 99, 80, 71, 91	88±14
		7	2	92, 87	90
Potato tuber	1.0	0	5	65, 104, 93, 111, 111	97±20
		7	2	91, 90	91
Peas, dry	1.0	0	5	74, 99, 72, 96, 67	82±18
		7	2	88, 76	82

Table A 4: Stability of flupyradifurone and DFA residues in plant matrices following storage at + 1°C for eight hours, following 6 to 8 days at -7°C

Matrix	Spike level (mg/kg)	Storage interval (days)	Individual recovered residues (mg/kg)	Individual recoveries (%)
Flupyradifurone				
Tomato fruit	1.0	0	0.833, 1.061, 0.816, 0.837, 0.875	83, 106, 82, 84, 88
		8	0.883, 0.904, 0.972, 0.885, 0.949	88, 90, 97, 89, 95
Wheat green material	1.0	0	0.896, 0.911, 0.941, 1.007, 1.031	90, 91, 94 101, 103
		8	0.919, 0.755, 0.801, 0.830, 0.869	92, 76, 80, 83, 87
Grapes bunches	1.0	0	0.791, 0.933, 0.967, 0.950, 1.095	79, 93, 97, 95, 109
		7	0.957, 1.016, 0.952, 1.005, 1.055	96, 102, 95, 100, 105
Wheat grain	1.0	0	0.782, 0.938, 0.953, 1.001, 1.029	78, 94, 95, 100, 103
		7	0.720, 0.917, 0.826, 1.061, 0.883	72, 92, 83, 106, 88
Potato tuber	1.0	0	0.914, 0.940, 0.954, 0.842, 0.865	91, 94, 95, 84, 87
		7	0.945, 0.973, 1.024, 1.017, 0.981	94, 97, 102, 102, 98
Peas dry	1.0	0	1.168, 1.070, 0.856, 1.325, 0.812	117, 107, 86, 132, 81
		6	0.778, 0.665, 0.499, 0.727, 0.778	78, 66, 50, 73, 78
DFA				
Tomato fruit	1.0	0	1.025, 0.788, 0.682, 0.989, 0.844	102, 79, 68, 99, 84
		7	0.843, 0.970, 0.778, 0.907, 0.753	84, 97, 78, 91, 75
Wheat green material	1.0	0	0.892, 0.798, 0 765, 0.608, 0.749	89, 80, 77, 61, 75
		7	0.826, 0.788, 0.926, 1.059, 0.928	83, 79, 93, 106, 93
Grapes bunches	1.0	0	0.691, 0.905, 0.731, 0.991, 1.069	69, 90, 73, 99, 107
		7	1.038, 0.932, 1.017, 0.893, 1.021	104, 93, 102, 89, 102
Wheat grain	1.0	0	0.987, 0.991, 0.802, 0.714, 0.911	99, 99, 80, 71, 91
		7	0.999, 0.968, 0.892, 1.184, 1.067	100, 97, 89, 118, 107
Potato tuber	1.0	0	0.645, 1.036, 0.932, 1.110, 1.106	65, 104, 93, 111, 111
		7	0.872, 1.001, 1.072, 1.033, 0.948	87, 100, 107, 103, 95
Peas dry	1.0	0	0.739, 0.986, 0.716, 0.963, 0.673	74, 99, 72, 96, 67
		7	0.821, 0.746, 0.845, 0.653, 0.841	82, 75, 84, 65, 84

Conclusion

Residues of flupyradifurone and DFA in the plant matrices of high water, high acid, high starch and high protein content were stable (<30% decomposition) after storage at + 1°C for 8 hours, following a storage of 6 - 8 days at -7°C.

A 2.1.1.1.1.3 Study report S17-05312

Comments of zRMS:	The stability of flupyradifurone and its metabolites difluoroethyl-amino-furanone (DFEAF), difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA) was investigated in oilseed rape (seed and straw) after 10 days at -8.5°C (target) plus 5 hours at +6°C (target) in the dark. The residues of flupyradifurone and its metabolites were determined in oilseed rape (seed and straw) according to BCS Method 01304/M001. The final determination was performed with HPLC-MS/MS. The Limit of Quantitation (LOQ), defined as the lowest validated fortification level, was 0.01 mg/kg for flupyradifurone, DFEAF and 6-CNA, expressed as parent equivalents and 0.05 mg/kg for DFA expressed as parent equivalents for each matrix. All method validation data are in compliance with the guideline requirements for data generation methods. After 10 days of storage at -8.5°C (target) followed by 5 hours at +6°C (target) the mean recovery in oilseed rape (seed) was 95 % for Flupyradifurone, 90 % for DFEAF, 94 % for 6-CNA and 92 % for DFA. After 10 days of storage at -8.5°C (target) followed by 5 hours at +6°C (target) the mean recovery in oilseed rape (straw) was 104 % for flupyradifurone, 105 % for DFEAF, 91 % for 6-CNA and 78 % for DFA. Furthermore the mean concurrent recoveries of day 0 to day 10 in oilseed rape (seed) determined from freshly fortified samples were in the range of 96-100 % for Flupyradifurone, 94-96 % for DFEAF, 90-102 % for 6-CNA and 91-92 % for DFA. In oilseed rape (straw) the mean concurrent recoveries of day 0 to day 10 determined from freshly fortified samples were in the range of 95-102 % for flupyradifurone, 90-108 % for DFEAF, 93-95 % for 6-CNA and 79-88 % for DFA. The study results demonstrate that the residues of flupyradifurone and its metabolites (DFEAF, 6-CNA and DFA) are stable in oilseed rape (seed and straw) when stored for 10 days at -8.5°C (target) plus 5 hours at +6°C (target). The study is acceptable.

Reference:	**KCA 6.1/03**
Title:	Storage stability of deltamethrin and flupyradifurone in oilseed rape (seed and straw)
Report:	Winter, O.; Amann, S.; Giesler, W.; 2018; S17-05312; M-626405-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC Guideline 7032/VI/95, rev. 5, July 1997; Appendix H of working document 1607/VI/97, rev. 2, June 1999 – Storage Stability of Residue Samples OECD 506, 2007; OECD Guideline for the Testing of Chemicals – Stability of Pesticide Residues in Stored Commodities
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

To determine the storage stability after 10 days at -8.5°C followed by 5 hours at +6°C in the dark of the relevant residues of flupyradifurone in oilseed rape seed and straw, control samples of the respective sample material were spiked with either flupyradifurone parent compound (separately) or DFEAF, DFA and 6-CNA (jointly) at a fortification level of 5.0 mg/kg (expressed as parent equivalent) of each analyte.

(As the metabolites DFEAF and 6-CNA are not part of the residue definition, they are not further considered within this summary).
Except for the day-0 analysis, samples were stored in glass containers in a cooling box at an average temperature of - 8.5°C for ten days, then they were transferred to +6°C in the dark.
For day-0 analysis, five treated samples of each material were chosen, as well as two control samples of each. At the 10-day storage interval, four aliquots of the stored control and five aliquots of fortified crop matrix were removed from storage. One of the control sample was fortified at 5.0 mg/kg with parent compound and DFA. In addition one of the control samples was fortified at 0.1 mg/kg with flupyradifurone or 0.5 mg/kg with DFA (for further details please refer to the report).
Samples were extracted with a mixture of ACN/water (4:1, v/v) with 2.2 mL/L formic acid. For the analysis of flupyradifurone extracts were cleaned-up on a Chromabond XTR column. For DFA no further clean-up steps were performed. After addition of the stable labelled internal standards, the final determination was performed with high performance liquid chromatography/triple quadrupole mass spectrometry (HPLC-MS/MS). Quantitation of each analyte was based on the daughter ion transitions of the analytes and the respective internal standard analogs. The responses of the LC-MS/MS system to each analyte and its internal standard were measured in samples and in standards, and a relative response was calculated (calculated as the ratio of the analyte and the stable isotopically labelled internal standard responses). The relative response of the analyte in each sample was compared to the relative response of the analyte in the standards.

Flupyradifurone and its metabolite were analytically determined using analytical method 01304/M001 (Schoening, R.; Willmes, J.; 2014; M-476845-01). The method was successfully validated in oilseed rape seeds and straw (equivalent to green material) in the storage stability study. The limits of quantitation (LOQs) were 0.01 mg/kg for flupyradifurone and 0.05 mg/kg for DFA (expressed in flupyradifurone equivalents) in all matrices.

Results and discussions
In the control samples, the residues were always below 30% of the LOQ.
Summaries of concurrent recoveries conducted as a part of the study are presented in Table A 5. The mean of the recoveries for each matrix were in an acceptable range (mean recoveries were 79% to 100%). The RSDs were in the range of 3.5 to 9.6%. Additional concurrent recoveries were determined at day 10 for samples spiked at 0.1 mg/kg (flupyradifurone) and 0.5 mg/kg (DFA), these values are not presented in the following table. Recoveries were 97% and 93% in seed and straw respectively, for parent flupyradifurone and 94% and 100% in seed and straw, respectively, for metabolite DFA.
Summaries of residues recovered in stored samples are presented in Table A 6 for flupyradifurone and DFA.
Residues of flupyradifurone and DFA were stable (<30% decomposition) in oilseed rape seed and straw for 10 days of storage at -8.5°C followed by 5 hours at +6°C.

Table A 5: Summary of concurrent recoveries of flupyradifurone and DFA in plant matrices

Matrix	Spike level (mg/kg)	Storage Interval (days)	Sample size (n)	Individual procedural recoveries (%)	Mean ± RSD (%)
Flupyradifurone					
Seed	5.0	0	5	95, 98, 101, 102, 104	100±3.5
		10	1	94	-
Straw	5.0	0	5	89, 95, 95, 98, 99	95±4.1
		10	1	110	-
DFA					
Seed	5.0	0	5	79, 87, 93, 99, 100	92±9.6
		10	1	88	-
Straw	5.0	0	5	75, 78, 80, 80, 83	79±3.7
		10	1	75	-

Only concurrent recoveries for 5.0 mg/kg spiked samples are presented here. In addition samples were also fortified at a level of 0.1 mg/kg (flupyradifurone) or 0.5 mg/kg (DFA). For details, please refer to the report.
RSD values calculated from rounded values of the report.

Table A 6: Stability of flupyradifurone and DFA residues in oilseed rape matrices following storage at -8.5°C for ten days, then +6°C for 5 hours

Matrix	Spike level (mg/kg)	Storage interval (days)	Individual recovered residues (mg/kg)	Individual recoveries (%)
Flupyradifurone				
Seed	5.0	0	4.76, 5.11, 4.88, 5.19, 5.03	95, 102, 98, 104, 101
		10	4.81, 4.72, 4.82, 4.87, 4.72	96, 94, 96, 97, 94
Straw	5.0	0	4.46, 4.74, 4.91, 4.76, 4.94	89, 95, 98, 95, 99
		10	5.50, 5.15, 5.18, 5.42, 4.87	110, 103, 104, 108, 97
DFA				
Seed	5.0	0	4.94, 4.98, 4.63, 4.33, 3.97	99, 100, 93, 87, 79
		10	4.63, 4.58, 4.94, 4.37, 4.38	93, 92, 99, 87, 88
Straw	5.0	0	4.02, 4.14, 3.73, 3.92, 4.01	80, 83, 75, 78, 80
		10	3.78, 3.83, 3.84, 3.60, 4.35	76, 77, 77, 72, 87

Conclusion
Residues of flupyradifurone and DFA in the plant matrices of oilseed rape (seed and straw) were stable during 10 days of storage at -8.5°C followed by 5 hours at +6°C.

A 2.1.1.1.2 Storage stability of residues in animal products

During the in-life portion of feeding study RARVN150 ([redacted]; 2017; M-611007-01-1) and RARVN149 ([redacted]; 2017; M-608471-01-1) samples were briefly stored at temperatures above -18ºC for a period of about 24 hours with a maximum recorded temperature of -2.5 ºC. Therefore a short-term storage stability study to confirm the stability of difluoroacetic acid in animal matrices when stored for 24 hours at refrigerator conditions was conducted as a part of the poultry feeding study (cf A 2.1.4.1.1).

A 2.1.1.1.2.1 Study report RARVN150

Comments of zRMS:	A 48 hour storage stability was conducted to evaluate the stability of difluoroacetic acid in animal matrices e(ggs, milk, muscle, kidney, liver and fat) when stored in a refrigerator. Samples were analyzed at approximately 0, 24 and 48 hour intervals. No loss of difluoroacetic acid (<30% decomposition) was observed in the samples during

	refrigerated storage. It can be concluded that residues of DFA in the animal matrices were stable during 2 days of storage in a refrigerator. This part of study is acceptable.

Reference:	**KCA 6.1/04**
Title:	Difluoroacetic acid - Magnitude of the residue in laying hens
Report:	[redacted] 2017; RARVN150; M-611007-01-1
Authority registration No:	
Guideline(s):	APVMA Residue Guidline No. 23; OPPTS 860.1480; DACO 7.5.1; OECD 505 Residues in Livestook
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Materials and methods

A short storage stability study was conducted as part of the feeding study RARVN0150 ([redacted] 2017; M-611007-01-1) to determine the stability of difluoroacetic acid when stored briefly above -18°C.
Control samples of eggs, liver, muscle and fat were taken from poultry and control samples of milk and kidney were taken from ruminant feeding study (RARVN150 and RARVN149, respectively) and spiked at 0.10 mg/kg (10X LOQ). Samples were stored in a refrigerator and analyzed at approximately 0, 24 and 48 hour intervals.
For day-0 analysis, three concurrent recoveries were prepared and analyzed as well as one control sample per animal matrix.
At each storage interval (24 and 48 hours) three stored samples were removed from storage and analyzed, together with one control (unfortified) sample as well as two freshly fortified samples (concurrent recoveries).

All animal matrices were analyzed according to analytical method RV-006-A17-01 with modifications (Kormos, T.; 2017; M-589558-01-1). Residues of difluoroacetic acid (DFA) were extracted from eggs, egg white and egg yolk using a mixture of 80:20 (v/v) acetonitrile/water acidified with 2.2 mL/L formic acid by blending. Extraction of DFA residues from muscle and liver was done by using a mixture of 85:15 (v/v) acetonitrile/water acidified with 5 mL/L formic acid. Fat samples were extracted using a mixture of 85:15 (v/v) acetonitrile/water acidified with 5 mL/L formic acid in a first step and heptane in a second step. An isotopic internal standard was added to the samples before sample aliquots were cleaned through a stacked C18 and SCX cartridge pre-washed with acetonitrile. The diluted sample was analyzed by high performance liquid chromatography/triple stage mass spectrometry (LC-MS/MS).
Quantitation of the analyte was based on the daughter ion transitions of the analyte and the internal standard analog. The relative response of the analyte in each sample was compared to the relative response of the analyte in the standard. The limit of quantitation (LOQ) and the limit of detection (LOD) are summarized in the table below. The limit of detection (LOD) is defined as the lowest concentration of an analyte that can be determined to be statistically different from a blank. The LODs were determined from method validation data obtained from control samples fortified at the respective analyte LOQs. The LODs were calculated by multiplying the standard deviation of recovery measurements at the LOQ by $t_{0.99}$ [where $t_{0.99}$ is the one-tailed t-statistic at the 99% confidence level for the number of replicates (n)].

Summary of LOQ and LODs:

Matrix	**Analyte**	**LOQ (mg/kg)**	**LOD (mg/kg)**
Eggs	Difluoroacetic acid	0.010	0.0030
Milk	Difluoroacetic acid	0.010	0.0013*
Muscle	Difluoroacetic acid	0.010	0.0031
Kidney	Difluoroacetic acid	0.010	0.0017*
Liver	Difluoroacetic acid	0.010	0.0018
Fat	Difluoroacetic acid	0.010	0.0033

* refers to the range of calculated LOD calculated in study RARVN149 [redacted]; 2017; M-608471-01-1).

Results and discussions

No interferences for DFA greater than the LOQ were observed in any of the control extracts from any of the matrices analyzed during this study. Residues of DFA in control samples ranged between 0.00167 and 0.00558 mg/kg. Recoveries presented in the following tables were not corrected for any interferences in corresponding controls.

Summaries of concurrent recoveries conducted as a part of the study are presented in Table A 7. The mean of the recoveries for each matrix were in an acceptable range (89-113%).
Summaries of residuesof DFA recovered in stored samples are presented in Table A 8. Residues of DFA were stable (<30% decomposition) in all animal matrices (eggs, milk, muscle, kidney, liver and fat) up to 2 days of storage in a refrigerator.

Table A 7: Summary of concurrent recoveries of DFA in poultry matrices (2 days at ca. -2.5 C)

Matrix	Spike level (mg/kg)	Storage Interval (days)	Sample size (n)	Individual procedural recoveries (%)*	Mean ± RSD (%) *
DFA					
Eggs	0.1	0	3	108, 112, 118	112 ± 4.5
		1	2	111, 116	113
		2	2	114, 114	113
Milk	0.1	0	3	114, 113, 110	112 ± 1.9
		1	2	110, 107	108
		2	2	113, 113	113
Muscle	0.1	0	3	95, 105, 105	102 ± 5.7
		1	2	103, 100	101
		2	2	103, 105	104
Kidney	0.1	0	3	113, 111, 114	113 ± 1.4
		1	2	111, 111	111
		2	2	111, 110	110
Liver	0.1	0	3	94, 89, 85	89 ± 5.0
		1	2	108, 108	108
		2	2	108, 112	110
Fat	0.1	0	3	106, 107, 105	106 ± 0.9
		1	2	109, 111	110
		2	2	109, 110	110

* Not stated in the report; recalculated based on values given in mg/kg.

Table A 8: Stability of residues in poultry matrices (2 days at ca. -2.5 C)

Matrix	Spike level (mg/kg)	Storage interval (days)	Individual recovered residues (mg/kg)	Individual recoveries* (%)	Mean recovery (%)
DFA					
Eggs	0.1	1	0.113, 0.114, 0.112	113, 114, 112	113
		2	0.114, 0.115, 0.110	114, 115, 110	113
Milk	0.1	1	0.110, 0.112, 0.112	110, 112, 112	111
		2	0.110, 0.111, 0.108	110, 111, 108	110
Muscle	0.1	1	0.099, 0.103, 0.100	99, 103, 100	101
		2	0.101, 0.103, 0.108	101, 103, 108	104
Kidney	0.1	1	0.112, 0.109, 0.108	112, 109, 108	110
		2	0.111, 0.110, 0.110	111, 110, 110	110
Liver	0.1	1	0.110, 0.110, 0.110	110, 110, 110	110
		2	0.108, 0.110, 0.111	108, 110, 111	109
Fat	0.1	1	0.111, 0.115, 0.111	111, 115, 111	112
		2	0.107, 0.106, 0.110	107, 106, 110	108

* Not stated in the report; recalculated based on values given in mg/kg.

Conclusion

Residues of DFA in the animal matrices of eggs, milk, muscle, kidney, liver and fat were stable (<30%

decomposition) during 2 days of storage in a refrigerator. As a consequence, the stability of DFA confirms the validity of the residue results in the presented feeding studies (cf. A.2.1.4.1).

A 2.1.1.2 Stability of residues in sample extracts

A 2.1.1.2.1 Storage stability of residues in plant sample extracts
No additional studies are required.

A 2.1.1.2.2 Storage stability of residues in animal sample extracts
No additional studies are required.

A 2.1.2 Nature of residues in plants, livestock and processed commodities

A 2.1.2.1 Nature of residue in primary crops
No additional studies are required.

A 2.1.2.2 Nature of residue in rotational crops
No additional studies are required.

A 2.1.2.3 Nature of residues in processed commodities
No additional studies are required.

A 2.1.2.4 Nature of residues in livestock
No additional studies are required.

A 2.1.3 Magnitude of residues in plants

A 2.1.3.1 Rapeseed

The critical GAP (cGAP) for the proposed use of Sivanto Energy (DLT+FPF EC 85) in oilseed rape is the GAP of the southern registration zone. The GAP is more critical with respect to possible flupyradifurone and DFA residues as compared to the GAPs intended for authorization in the central registration zone (for an evaluation of the GAPs please refer to chapter 7.1.1). All GAPs are summarized in Table A 9.

Table A 9: Comparison of intended and critical EU GAPs

Type of GAP	Number of applications	Application rate per treatment (kg a.s./ha)	Interval between application	Growth stage at last application	PHI (days)
cGAP EU (DAR, RMS, year)	Currently there is no EU cGAP in place on oilseed rape for flupyradifurone.				
cGAP EU (Art. 12, EFSA, year)	Currently there is no EU cGAP in place on oilseed rape for flupyradifurone. The intended cGAP for Sivanto Energy (DLT+FPF EC 85) of S-EU will be the EU cGAP.				
S-EU: Intended cGAP (Use No.* in the S-zone dRR to be submitted in 2019-Q4)	2	56.25	14	BBCH 30-79	45
N-EU: Intended cGAP (Use No.* 1, 2, 5, 6, 7, 8, 11, 12, 17, 18, 23, 24, 27, 28)	2	56.25	14	BBCH 30-49	As per growth stage
N-EU: Intended cGAP (Use No.* 13, 14, 19, 20)	2	56.25	14	BBCH 50-59	As per growth stage
N-EU: Intended cGAP (Use No.* 3, 4, 9, 10, 15, 16, 21, 22, 25, 26, 29, 30)	2	37.5	14	BBCH 65-79	45

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0

According to SANCO 7525/VI/95 Rev. 10.3, extrapolation from rapeseeds to mustard seeds is possible based on 4 trials with treatments before and after the edible part has been formed.

A 2.1.3.1.1 Study report 15-2132

Comments of zRMS:	Four field residue trials were conducted in northern Europe in 2015 to determine the magnitude of the relevant residues of deltamethrin and BYI 02960 (flupyradifurone) in/on rape (green material, seed and straw) after two spray applications with Deltamethrin & Flupyradifurone EC 085, an EC formulation containing 10 g/L deltamethrin and 75 g/L BYI 02960. Trial GAP: 2 x 56.25 g a.s./ha, interval = 14 days, PHI = 45 days. This study was conducted according to the study plan and study plan amendment. For deltamethrin and its isomers and for BYI 02960 and its metabolites, no residues above the LOQ were found in the control samples. The mean of the concurrent recoveries per fortification level or the individual recoveries were within the acceptable range of 70 - 110% and the RSD values were below 20% for all compounds and for all sample materials. The residues of BYI 02960 and DFA were determined according to method 01212 (Rosati, D.; 2012;) by LC-MS/MS. Residue results (mg/kg): FPF: 0.065; 0.088; 0.13; 0.21, DFA: 0.048; 0.068; 0.075; 0.095, sum: 0.27; 0.27; 0.31; 0.50.

	The study is acceptable.

Reference:	**KCA 6.3.1.1/01**
Title:	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in France (North), Germany and Belgium
Report:	Schulte, G.; 2017; 15-2132; M-578527-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	yes, see report
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
In 2015, 4 field trials were conducted in northern Europe (northern France, Germany [2] and Belgium) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960).
Two applications were made at an interval of 13-14 days, each at a rate of 0.75 L/ha, corresponding to 56.25 g a.s./ha. The last application was done at a crop growth stage of BBCH 73-77 (30% to 70% of pods have reached final size). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the treatment in all trials and 39 days after the last treatment in the German trial (as collection of immature seeds was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 37-42 days after the last application (BBCH 87-88), 44-48 days after the last application (BBCH 88-89) and 51-54 days after the last application (BBCH 89). All samples were frozen within 24 hours after sampling and stored at a temperature of or below -18°C until analysis (max. 171 days). The maximum storage period of 171 days is covered by the available storage stability study where the analytes have been proven to be stable for a period of at least 52 months (Netzband, D.; 2015; M-428412-03-1).

The residues of BYI 02960 and DFA were determined according to method 01212 (Rosati, D.; 2012; M-428017-01-1) by LC-MS/MS using stable isotopically labelled internal standards. The LOQ in all sample matrices was 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA when expressed as BYI 02960 (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of BYI 02960 and DFA, expressed as BYI 02960) was 0.03 mg/kg.
Residues of DFEAF and 6-CNA have been also determined during the study, but since they are not relevant to the residue definition for risk assessment, they are not reported here.

Results
During the course of this study, the method performance was checked/validated by concurrent recoveries. Full validation of method 01212 was done and documented for oilseed rape (seeds) when developing the method. For rape, green material a limited set (1 control, 3 repetitions each at two fortification levels) of additional validation recoveries was done within this study, and a full validation (2 controls, 5 repetitions each at two fortification levels) was done for rape, straw (cf. Section B5). The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always well below 20% (Table A 10).

Details of the application and residue information are summarized in Table A 11.
Low amounts of DFA were detected in three of four untreated samples, however the levels were always below the limit of quantification of the method.

Table A 10: Recovery data for BYI 02960 and its metabolite DFA in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level* (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
15-2132 15-2132-01 15-2132-01-T to 15-2132-04 15-2132-04-T GLP: yes 2015	Rape	green material	BYI 02960	4	0.01	96; 98; 98; 99	96	99	98	1.3
				3	0.1	98; 99; 101	98	101	99	1.5
				1	0.2	98	98	98	98	-
				1	2.0	104	104	104	104	-
				9	overall		96	104	99	2.3
			DFA	4	0.02	104; 104; 105; 110	104	110	106	2.7
				3	0.2	96; 97; 98	96	98	97	1.0
				1	0.4	99	99	99	99	-
				1	4.0	106	106	106	106	-
				9	overall		96	110	102	4.7
		seed	BYI 02960	1	0.01	110	110	110	110	-
				1	0.1	100	100	100	100	-
				1	1.0	105	105	105	105	-
				3	overall		100	110	105	4.8
			DFA	1	0.02	90	90	90	90	-
				1	0.2	84	84	84	84	-
				1	2.0	80	80	80	80	-
				3	overall		80	90	85	5.9
		straw	BYI 02960	5	0.01	102; 102; 103; 108; 109	102	109	105	3.3
				5	0.1	100; 100; 100; 102; 107	100	107	102	3.0
				1	1.0	105	105	105	105	-
				11	overall		100	109	103	3.2
			DFA	5	0.02	92[a]; 94[b]; 95[c]; 99; 102[d]	92	102	96	4.2
				5	0.2	96; 98; 99; 99; 102	96	102	99	2.2
				1	2.0	100	100	100	100	-
				11	overall		92	102	98	3.3

* expressed as BYI 02960

The control 15-2132-01-0024E (0.00679 mg/kg expressed as BYI 02960) was used for fortification for the following values:

[a] This value was corrected with the value of the respective control sample. The uncorrected value was 126%.

[b] This value was corrected with the value of the respective control sample. The uncorrected value was 128%.

The control 15-2132-02-0024E (0.00767 mg/kg expressed as BYI 02960) was used for fortification for the following values:

[c] This value was corrected with the value of the respective control sample. The uncorrected value was 133%.

The control 15-2132-04-0024E (0.00358 mg/kg expressed as BYI 02960) was used for fortification for the following values:

[d] This value was corrected with the value of the respective control sample. The uncorrected value was 120%.

Table A 11: Summary of the trials of study 15-2132

FPF (flupyradifurone, BYI 02960; determined as FPF, calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as FPF), Sum of FPF and DFA (calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as DFA)

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
15-2132-01 15-2132-01-T France, north 37310 Chambourg sur Indre Europe, North F 2015	Rape Arizona; Winter variety	1) 19.08.2014 2) 07.04.2015 - 30.04.2015 3) 01.07.2015 - 10.07.2015	56.25 56.25	300 300	18.75	04.05.2015/0 18.05.2015/14	75	green material	75 75	0.11 1.4	0.081 0.084	0.19 1.5	0.027 0.028	0* 0	(g) 15-2132 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01212 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01212, 15-2132 (m) Storage: straw: 147 days seed: 116 days green material: 171 days
								seed	88 89 89	0.11 0.088 0.074	0.21 0.23 0.20	0.32 0.31 0.27	0.070 0.075 0.065	42 45 52	
								straw	88 89 89	<0.01 0.15 0.12	0.044 0.36 0.30	0.054 0.50 0.42	0.015 0.12 0.10	42 45 52	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
15-2132-02 15-2132-02-T Germany 51399 Burscheid Europe, North F 2015	Rape Visby; Winter variety	1) 25.08.2014 2) 20.04.2015 - 08.05.2015 3) 01.07.2015 - 01.08.2015	56.25 56.25	300 300	18.75	07.05.2015/0 21.05.2015/14	76	green material	76 76 82	0.18 1.4 0.25	0.10 0.11 0.29	0.28 1.5 0.54	0.034 0.037 0.097	0* 0 39	(g) 15-2132 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01212 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01212, 15-2132 (m) Storage: straw: 140 days seed: 109 days green material: 168 days
								seed	88 89	0.21 0.21	0.29 0.28	0.50 0.50	0.095 0.094	46 53	
								straw	88 89	0.070 0.13	0.37 0.45	0.44 0.58	0.12 0.15	46 53	
15-2132-03 15-2132-03-T Germany 59609 Anröchte-Berge Europe, North F 2015	Rape Avatar; Winter variety	1) 22.08.2014 2) 20.04.2015 - 20.05.2015 3) 15.07.2015 - 10.08.2015	56.25 56.25	300 300	18.75	26.05.2015/0 08.06.2015/13	73	green material	73 73	0.056 1.3	0.021 0.021	0.076 1.3	0.007 0.007	0* 0	(g) 15-2132 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01212 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01212, 15-2132 (m) Storage: straw: 131 days seed: 100 days green material: 150 days
								seed	88 89 89	0.12 0.13 0.13	0.12 0.14 0.14	0.24 0.27 0.27	0.039 0.048 0.047	37 44 51	
								straw	88 89 89	0.29 0.39 0.47	0.023 0.025 <0.02	0.32 0.41 0.49	0.008 0.008 <0.0067	37 44 51	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
15-2132-04 15-2132-04-T Belgium 6210 Villers-Perwin Europe, North F 2015	Rape Dynastie ; Winter variety	1) 04.09.2014 2) 20.04.2015 - 18.05.2015 3) 22.07.2015 - 28.07.2015	56.25 56.25	300 300	18.75	21.05.2015/0 04.06.2015/14	77	green material	77 77	0.24 1.1	0.072 0.061	0.31 1.2	0.024 0.020	0* 0	(g) 15-2132 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01212 (k) LOQ: FPF : 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01212, 15-2132 (m) Storage: straw: 133 days seed: 115 days green material: 154 days
								seed	87 89 89	0.088 0.065 0.055	0.16 0.20 0.19	0.25 0.27 0.24	0.052 0.068 0.063	39 48 54	
								straw	87 89 89	0.018 <0.01 0.022	0.035 0.025 0.037	0.053 0.035 0.058	0.012 0.008 0.012	39 48 54	

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation

(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse F field

* prior to last treatment
** residue in control

A 2.1.3.1.2 Study report 16-2044

Comments of zRMS:	Four field residue trials were conducted in northern Europe in 2016 to determine the magnitude of the relevant residues of deltamethrin and BYI 02960 (flupyradifurone) in/on rape (green material, seed and straw) after two spray applications with Deltamethrin & Flupyradifurone EC 085, an EC formulation containing 10 g/L deltamethrin and 75 g/L BYI 02960. Trial GAP: 2 x 56.25 g a.s./ha, interval = 14 days, PHI = 45 days. No residues above the respective LOQs were found in the control samples. The average / individual recoveries per fortification level were within the range of 70 – 110% and the RSD values were below 20%, if applicable ($n \geq 3$). Residues of deltamethrin and its isomers AE F108569 and AE 0035073 were determined by LC-MS/MS according to method 00855/M004. Residues of BYI 02960 and its metabolites were determined by LC-MS/MS according to method method 01304. The individual LOQs for deltamethrin and its isomers are 0.05 mg/kg in green material and straw and 0.01 mg/kg in seed. The individual LOQs for BYI 02960 and difluoroacetic acid are 0.01 and 0.02 mg/kg, respectively in the sample materials green material, seed and straw. Thus a theoretical LOQ of 0.03 mg/kg results for the sum of the two analytes. Residue results (mg/kg): FPF: 0.066; 0.12; 0.15; 0.15 mg/kg, DFA: 0.043; 0.063; 0.068; 0.075 mg/kg, sum: 0.25; 0.25; 0.35; 0.35 mg/kg. The study is acceptable.

Reference:	**KCA 6.3.1.1/02**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and northern France
Report:	Kaussmann, M.; Kerkering, S.; 2018; 16-2044; M-641044-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP 860.1500, Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and method

In 2016, 4 field trials were conducted in northern Europe (northern France, Germany [2] and Belgium) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960). Two applications were made at an interval of 13-14 days, each at a rate of 0.75 L/ha, corresponding to 56.25 g a.s./ha. The last application was done at a crop growth stage of BBCH 73-80 (30% of pods have reached final size to beginning of ripening: seed green). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the treatment in all trials and 38 days after the last treatment in the French trial (as collection of immature seeds was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 34-38 days

after the last application (BBCH 84-89), 42-48 days after the last application (BBCH 89) and 50-52 days after the last application (BBCH 89). All samples were frozen within 24 hours after sampling and stored at a temperature of or below -18°C until analysis (max. 619 days). The maximum storage period of 619 days is covered by the available storage stability study where the analytes have been proven to be stable for a period of at least 52 months (Netzband, D.; 2015; M-428412-03-1).

The samples were analyzed according to method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The LOQ in all sample matrices was 0.01 mg/kg for flupyradifurone and 0.02 mg/kg for DFA when expressed as flupyradifurone (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of flupyradifurone and DFA, expressed as flupyradifurone) was 0.03 mg/kg.
Residues of DFEAF and 6-CNA have been also determined during the study, but since they are not relevant to the residue definition for risk assessment, they are not reported here.

Results
The method performance was checked/validated by concurrent recoveries during the conduct of the study. Full validation of method 01304 was done and documented for matrices representing the 5 major crop groups, including dry seeds of beans, cereal forage, orange fruit, soybean seeds, tomato fruit, and wheat grain when developing the method. For rape, green material, seed and straw a limited set (1 control, 3 repetitions each at two fortification levels) of additional validation recoveries was done within this study. The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always below 20% (Table A 12).

Details of the application and residue information are summarized in Table A 13. No residues of flupyradifurone or DFA at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 12: Recovery data for BYI 02960 and its metabolite DFA in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level* (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2044 16-2044-01 16-2044-01-T to 16-2044-04 16-2044-04-T GLP: yes 2016	Rape	green material	BYI 02960	6 6 1 13	0.01 0.1 4.0 overall	86; 91; 99 90; 94; 95 96	86 90 96 86	99 95 96 99	92 93 96 93	7.1 2.8 - 4.6
			DFA	3 3 1 7	0.02 0.2 8.0 overall	98; 100; 102 92; 95; 95 91	98 92 91 91	102 95 91 102	100 94 91 96	2.0 1.8 - 4.2
		seed	BYI 02960	3 3 1 7	0.01 0.1 0.3 overall	93; 97; 99 91; 98; 100 96	93 91 96 91	99 100 96 100	96 96 96 96	3.2 4.9 - 3.4
			DFA	3 3 1 7	0.02 0.2 0.6 overall	87; 90; 106 83; 86; 88 83	87 83 83 83	106 88 83 106	94 86 83 89	10.8 2.9 - 8.9
		straw	BYI 02960	3 3 6	0.01 0.1 overall	88; 89; 91 88; 92; 95	88 88 88	91 95 95	89 92 91	1.7 3.8 3.0
			DFA	3 3 6	0.02 0.2 overall	93; 101; 107 90; 90; 91	93 90 90	107 91 107	100 90 95	7.0 0.6 7.4

* expressed as BYI 02960

Table A 13: Summary of the trials of study 16-2044

FPF (flupyradifurone, BYI 02960; determined as FPF, calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as FPF), Sum of FPF and DFA (calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as DFA)

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2044-01 16-2044-01-T Germany 04824 Beucha OT Wolfshain Europe, North F 2016	Rape Totem; winter-rape, line variety	1) 25.08.2015 2) 14.04.2016 - 08.05.2016 3) 06.07.2016 - 27.07.2016	56.25 56.25	300 300	18.75 18.75	18.05.2016/0 01.06.2016/14	73	green material	73 73	0.073 0.71	0.064 0.066	0.14 0.78	0.021 0.022	0* 0	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2044 (m) Storage: straw: 566 days seed: 566 days green material: 604 days
								seed	84 89 89	0.13 0.15 0.10	0.21 0.20 0.23	0.34 0.35 0.32	0.069 0.067 0.075	38 45 52	
								straw	84 89 89	0.029 0.050 0.063	0.11 0.097 0.13	0.14 0.15 0.19	0.037 0.032 0.042	38 45 52	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
(a)		(b)				(c)	(d)		(d)					(e)	(f)
16-2044-02 16-2044-02-T Germany 59609 Anröchte-Berge Europe, North F 2016	Rape Elektra; Rape winter	1) 22.08.2015 2) 10.04.2016 - 25.05.2016 3) 20.07.2016 - 10.08.2016	56.25 56.25	300 300	18.75 18.75	30.05.2016/0 13.06.2016/14	79	green material	79 79	0.31 3.4	0.078 0.071	0.38 3.5	0.026 0.024	0* 0	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2044 (m) Storage: straw: 556 days seed: 556 days green material: 592 days
								seed	88 89 89	0.11 0.15 0.087	0.23 0.20 0.16	0.33 0.35 0.25	0.075 0.068 0.054	36 42 51	
								straw	88 89 89	0.017 0.025 0.060	0.072 0.054 0.042	0.090 0.080 0.10	0.024 0.018 0.014	36 42 51	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
(a)		(b)				(c)	(d)		(d)					(e)	(f)
16-2044-03 16-2044-03-T Belgium 6221 Saint-Amand Europe, North F 2016	Rape Visby; Winter rape	1) 05.09.2015 2) 18.04.2016 - 18.05.2016 3) 20.07.2016 - 05.08.2016	56.25 56.25	300 300	18.75 18.75	03.06.2016/0 16.06.2016/13	80	green material	80 80	0.22 0.88	0.081 0.075	0.30 0.95	0.027 0.025	0* 0	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2044 (m) Storage: straw: 555 days seed: 555 days green material: 589 days
								seed	89 89 89	0.13 0.12 0.090	0.14 0.13 0.13	0.26 0.25 0.22	0.045 0.043 0.043	34 43 50	
								straw	89 89 89	<0.01 0.014 0.022	<0.02 0.026 0.029	<0.03 0.040 0.051	<0.0067 0.009 0.010	34 43 50	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2044-04 16-2044-04-T France, north 37310 Chambourg sur Indre Europe, North F 2016	Rape Arizona; Winter variety	1) 21.08.2015 2) 06.04.2016 - 03.05.2016 3) 05.07.2016 - 15.07.2016	56.25 56.25	300 300	18.75 18.75	03.05.2016/0 17.05.2016/14	75	green material	75 75 82	0.11 0.99 0.025	0.081 0.077 0.24	0.19 1.1 0.26	0.027 0.026 0.079	0* 0 38	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2044 (m) Storage: straw: 571 days seed: 571 days green material: 619 days
								seed	89 89	0.066 0.064	0.19 0.19	0.25 0.25	0.063 0.062	48 52	
								straw	89 89	<0.01 <0.01	0.025 0.034	0.035 0.044	0.008 0.011	48 52	

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

G greenhouse F field

<<< intended PHI

(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation

(m) Storage (max)
! based on date of analysis
P based on production date

* prior to last treatment
** residue in control
\# no data available

A 2.1.3.1.3 Study report 16-2131

Comments of zRMS:	This study has not been evaluated by zRMS-PL, because the trials were conducted in southern Europe.

Reference:	**KCA 6.3.1.1/03**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Spain, Portugal, southern France and Italy
Report:	Miara, C.; Nayyar, B.; Kowalski, N.; 2018; 16-2131; M-643446-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP 860.1500, Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	-
Duplication (if vertebrate study):	

Materials and methods
In 2016, 6 field trials were conducted in southern Europe (Spain, Portugal, southern France [2] and Italy [2]) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960). Two applications were made at an interval of 14-15 days, each at a rate of 0.75 L/ha, corresponding to 56.25 g a.s./ha. The last application was done at a crop growth stage of BBCH 67-80 (flowering declining to beginning of ripening: seed green). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the treatment in all trials and 38 days after the last treatment in the French and Portuguese trials (as collection of immature seeds and straw was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 37-40 days after the last application (BBCH 82-88), 44-47 days after the last application (BBCH 87-89) and 49-54 days after the last application (BBCH 89). All samples were frozen within 24 hours after sampling and stored at a temperature of or below -18°C until analysis (max. 799 days), except in trials 16-2131-02 and 16-2131-06 where the required temperature was exceeded for 15 hours (maximum temperature within this period was -9°C) during storage of straw and seed samples. However these exceedances were shown to be acceptable as the stability of all relevant BYI 02960-related residues in oilseed rape matrices was confirmed in a short-term storage stability study (Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1). A storage period of 10 days at -8.5°C, followed by 5 hours at +6°C was covered. Considering that the long-term storage stability study conducted at ≤ -18°C confirmed the stability of the analytes for a period of at least 52 months (Netzband, D.; 2015; M-428412-03-1), the validity of all study results is proven.

The samples were analyzed according to method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The LOQ in all sample matrices was 0.01 mg/kg for flupyradifurone and 0.02 mg/kg for DFA when expressed as flupyradifurone (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of flupyradifurone and DFA, expressed as flupyradifurone) was 0.03 mg/kg.
Residues of DFEAF and 6-CNA have been also determined during the study, but since they are not relevant to the residue definition for risk assessment, they are not reported here.

Results

The method performance was checked/validated by concurrent recoveries during the conduct of the study. Full validation of method 01304 was done and documented for matrices representing the 5 major crop groups, including dry seeds of beans, forage, orange fruit, soybean seeds, tomato fruit, and wheat grain when developing the method. For rape, green material, seed and straw a limited set (1 control, 3 repetitions each at two fortification levels) of additional validation recoveries was done within this study. The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always below 20% (Table A 14).

Details of the application and residue information are summarized in Table A 15. No residues of flupyradifurone or DFA at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 14: Recovery data for BYI 02960 and its metabolite DFA in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level* (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2131 16-2131-01 16-2131-01-T to 16-2131-06 16-2131-06-T GLP: yes 2016	Rape	green material	BYI 02960	3	0.01	99; 102; 103	99	103	101	2.1
				3	0.1	94; 96; 102	94	102	97	4.3
				1	5.0	98	98	98	98	-
				7	overall		94	103	99	3.4
			DFA	3	0.02	99; 100; 104	99	104	101	2.6
				3	0.2	94; 96; 104	94	104	98	5.4
				1	10	88	88	88	88	-
				7	overall		88	104	98	5.9
		seed	BYI 02960	3	0.01	96; 103; 103	96	103	101	4
				3	0.1	88; 91; 104	88	104	94	9
				1	1.0	96	96	96	96	
				7	overall		88	104	97	6.5
			DFA	3	0.02	79; 81; 82	79	82	81	1.9
				3	0.2	71; 71; 81	71	81	74	7.8
				1	2.0	70	70	70	70	
				7	overall		70	82	76	7.2
		straw	BYI 02960	3	0.01	95; 97; 101	95	101	98	3.1
				3	0.1	106; 108; 109	106	109	108	1.4
				1	1.0	84	84	84	84	
				7	overall		84	109	100	8.9
			DFA	3	0.02	91; 94; 102	91	102	96	5.9
				3	0.2	102; 103; 105	102	105	103	1.5
				1	2.0	80	80	80	80	
				7	overall		80	105	97	9.3

* expressed as BYI 02960

Table A 15: Summary of the trials of study 16-2131

FPF (flupyradifurone, BYI 02960; determined as FPF, calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as FPF), Sum of FPF and DFA (calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as DFA)

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2131-01 16-2131-01-T Spain 41410 Carmona Europe, South F 2016	Rape Omega 9; Winter rape	1) 23.10.2015 2) 05.02.2016-20.03.2016 3) 20.05.2016-10.06.2016	56.25 56.25	300 300	18.75 18.75	23.03.2016/0 06.04.2016/14	80	green material	80 80	0.43 1.3	0.14 0.10	0.57 1.4	0.047 0.035	0* 0	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2131 (m) Storage: straw: 766 days seed: 756 days green material: 798 days
								seed	85 88 89	0.20 0.10 0.16	0.48 0.46 0.48	0.68 0.56 0.63	0.16 0.15 0.16	40 44 49	
								straw	85 88 89	0.89 0.95 0.88	0.14 0.20 0.13	1.0 1.1 1.0	0.046 0.067 0.045	40 44 49	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2131-02 16-2131-02-T Portugal 2080-187 Almeirim Europe, South F 2016	Rape PR45H73; Spring variety	1) 23.10.2015 2) 10.02.2016-11.04.2016 3) 15.05.2016-15.06.2016	56.25 56.25	300 300	18.75 18.75	21.03.2016/0 05.04.2016/15	78	green material	78 78 82	0.035 0.89 0.16	0.024 0.032 0.15	0.060 0.92 0.31	0.008 0.011 0.049	0* 0 38	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2131 (m) Storage: straw: 762 days seed: 752 days green material: 799 days
								seed	87 89	0.11 0.090	0.17 0.17	0.28 0.26	0.057 0.057	45 52	
								straw	87 89	0.040 0.057	0.060 0.062	0.099 0.12	0.020 0.021	45 52	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2131-03 16-2131-03-T France, south 13103 St Etienne du Gres Europe, South F 2016	Rape Attletick; Rape oil	1) 28.08.2015 2) 21.03.2016-30.04.2016 3) 15.06.2016-25.06.2016	56.25 56.25	300 300	18.75 18.75	14.04.2016/0 29.04.2016/15	69	green material	69 69 79	0.14 1.1 0.051	0.11 0.14 0.29	0.25 1.2 0.34	0.038 0.048 0.096	0* 0 38	g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2131 (m) Storage: straw: 737 days seed: 727 days green material: 775 days
								seed	88 89	0.058 0.038	0.26 0.23	0.32 0.26	0.088 0.075	46 52	
								straw	88 89	0.026 0.024	0.34 0.11	0.37 0.13	0.11 0.036	46 52	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2131-04 16-2131-04-T France, south 31620 Gargas Europe, South F 2016	Rape DK EXPLICIT; Hybrid	1) 20.08.2015 2) 29.03.2016-05.06.2016 3) 23.06.2016-30.06.2016	56.25 56.25	300 300	18.75 18.75	25.04.2016/0 10.05.2016/15	72	green material	72 72	0.18 0.94	0.044 0.051	0.23 1.0	0.015 0.017	0* 0	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2131 (m) Storage: straw: 734 days seed: 724 days green material: 764 days
								seed	88 89 89	0.13 0.11 0.10	0.17 0.19 0.19	0.30 0.30 0.29	0.057 0.063 0.063	38 44 50	
								straw	88 89 89	0.047 0.038 0.028	0.045 0.038 0.047	0.092 0.076 0.075	0.015 0.013 0.016	38 44 50	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2131-05 16-2131-05-T Italy 44022 Comacchio (Ferrara) Europe, South F 2016	Rape PR45D01; Normal ripening cycle variety	1) 21.09.2015 2) 01.04.2016-25.04.2016 3) 01.06.2016-15.06.2016	56.25 56.25	400 400	14 14	07.04.2016/0 20.04.2016/13	67	green material	67 67	0.24 1.8	0.13 0.11	0.37 1.9	0.044 0.037	0* 0	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2131 (m) Storage: straw: 755 days seed: 745 days green material: 785 days
								seed	82 88 89	0.071 0.036 0.020	0.098 0.18 0.18	0.17 0.22 0.20	0.033 0.060 0.059	37 47 54	
								straw	82 88 89	0.038 0.051 0.017	0.16 0.13 0.11	0.20 0.18 0.12	0.053 0.042 0.035	37 47 54	

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
	(a)	(b)				(c)	(d)		(d)					(e)	(f)
16-2131-06 16-2131-06-T Italy 40016 San Giorgio di Piano Bologna Europe, South F 2016	Rape Excalibur; medium variety	1) 15.09.2015 2) 01.04.2016-20.04.2016 3) 06.06.2016-20.06.2016	56.25 56.25	400 400	14 14	12.04.2016/0 26.04.2016/14	73	green material	73 73	0.078 0.81	0.059 0.062	0.14 0.87	0.020 0.021	0* 0	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF l: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2131 (m) Storage: straw: 748 days seed: 738 days green material: 778 days
								seed	86 88 89	0.24 0.066 0.058	0.21 0.14 0.15	0.44 0.21 0.21	0.068 0.048 0.050	38 45 52	
								straw	86 88 89	0.028 <0.01 <0.01	<0.02 <0.02 0.033	0.048 <0.03 0.043	<0.0067 <0.0067 0.011	38 45 52	

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation

(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse F field

<<< intended PHI

* prior to last treatment
** residue in control
\# no data available

A 2.1.3.1.4 Study report 16-2169

Comments of zRMS:	This study has not been evaluated by zRMS-PL, because the trials were conducted in southern Europe.

Reference:	**KCA 6.3.1.1/04**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Spain and Portugal
Report:	Miara, C.; Nayyar, B.; Kowalski, N.; 2018; 16-2169; M-645539-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP 860.1500, Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	-
Duplication (if vertebrate study):	

Materials and method
In 2016, 2 field trials were conducted in southern Europe (Spain and Portugal) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960). Two applications were made at an interval of 14-15 days, each at a rate of 0.75 L/ha, corresponding to 56.25 g a.s./ha. The last application was done at a crop growth stage of BBCH 72-75 (20% to 50% of pods have reached final size). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the last treatment in both trials and 36 days after the last treatment in the Spanish trial (as collection of immature seeds and straw was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 36 days after the last application (BBCH 89), 43 days after the last application (BBCH 89) and 50 days after the last application (BBCH 89-97). All samples were frozen within 24 hours after sampling and stored at a temperature of or below -18°C until analysis (max. 736 days), except in trial 16-2169-01 where the required temperature was exceeded for 13 hours (maximum temperature within this period was -8.5°C) and up to +5.9°C for 4 hours. Also in the trial 16-2169-02 the storage temperature increased for some samples above -18°C (up to -8.5°C during storage and up to +5.9°C for 1 hour. However these exceedances were shown to be acceptable as the stability of all relevant BYI 02960-related residues in high-water content matrices was confirmed in a short-term storage stability study, which covers temperatures up to -7°C for 7 days (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1). Additionally the stability of all relevant BYI 02960-related residues in oilseed rape matrices was confirmed in a short-term storage stability study, which tested the stability of the analytes for 10 days at -8.5°C, followed by 5 hours at +6°C (Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1). Considering that the long-term storage stability study conducted at ≤ -18°C confirmed the stability of the analytes for a period of at least 52 months (Netzband, D.; 2015; M-428412-03-1), the validity of all study results is proven.

The samples were analyzed according to method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The LOQ in all sample matrices was 0.01 mg/kg for flupyradifurone and 0.02 mg/kg for DFA when expressed as flupyradifurone (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of flupyradifurone and DFA, expressed as flupyradifurone) was 0.03 mg/kg.
Residues of DFEAF and 6-CNA have been also determined during the study, but since they are not relevant to the residue definition for risk assessment, they are not reported here.

Results

The method performance was checked/validated by concurrent recoveries during the conduct of the study. Full validation of method 01304 was done and documented for matrices representing the 5 major crop groups, including dry seeds of beans, cereal forage, orange fruit, soybean seeds, tomato fruit, and wheat grain when developing the method. For rape, green material, seed and straw a limited set (1 control, 3 repetitions each at two fortification levels) of additional validation recoveries was done within this study. The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always below 20% (Table A 16).

Details of the application and residue information are summarized in Table A 17. No residues of flupyradifurone or DFA at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 16: Recovery data for BYI 02960 and its metabolite DFA in rapeseed

Study Trial No. Plot No. GLP Year	**Crop**	**Portion analyzed**	**a.s./ metabolite**	**n**	**Fortification level* (mg/kg)**	**Recovery (%) Individual recoveries**	**Min**	**Max**	**Mean**	**RSD**
16-2169 16-2169-01 16-2169-01-T and 16-2169-02 16-2169-02-T GLP: yes 2016	Rape	green material	BYI 02960	3	0.01	103; 103; 105	103	105	104	1.1
				3	0.1	94; 96; 97	94	97	96	1.6
				1	1.0	93	93	93	93	
				7	overall		93	105	99	4.9
			DFA	3	0.02	97; 99; 103	97	103	100	3.1
				3	0.2	90; 90; 93	90	93	91	1.9
				1	2.0	95	95	95	95	
				7	overall		90	103	95	5
		seed	BYI 02960	3	0.01	99; 100; 102	99	102	100	1.5
				3	0.1	91; 93; 93	91	93	92	1.3
				1	0.5	92	92	92	92	
				7	overall		91	102	96	4.7
			DFA	3	0.02	76;77;78	76	78	77	1.3
				3	0.2	75;75;77	75	77	76	1.5
				1	1.0	75	75	75	75	
				7	overall		75	78	76	1.6
		straw	BYI 02960	3	0.01	90; 91; 94	90	94	92	2.3
				3	0.1	77; 86; 86	77	86	83	6.3
				1	0.5	91	91	91	91	
				7	overall		77	94	88	6.4
			DFA	3	0.02	94;96;99	94	99	96	2.6
				3	0.2	78;88;89	78	89	85	7.2
				1	1.0	92	92	92	92	
				7	overall		78	99	91	7.5

* expressed as BYI 02960

Table A 17: Summary of the trials of study 16-2169

FPF (flupyradifurone, BYI 02960; determined as FPF, calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as FPF), Sum of FPF and DFA (calculated as FPF), DFA (difluoroacetic acid; determined as DFA, calculated as DFA)

Trial No./ Location/ EU zone/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Growth stage at sampling	Residues (mg/kg)				PHI (days)	Details on trial
			g a.s./ ha	Water (l/ha)	g a.s./hl					FPF	DFA as FPF	Sum of FPF and DFA	DFA as DFA		
(a)		(b)				(c)	(d)		(d)					(e)	(f)
16-2169-01 16-2169-01-T Spain 25281 Llobera Europe, South F 2016	Rape Hidronel	1) 21.09.2015 2) 25.04.2016 - 25.05.2016 3) 14.07.2016 - 21.07.2016	56.25 56.25	360 360	15.6 15.6	17.05.2016/0 01.06.2016/15	72	green material	72 72 85	0.16 0.76 0.23	0.060 0.041 0.24	0.22 0.80 0.47	0.020 0.014 0.079	0* 0 36	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2169 (m) Storage: straw: 669 days seed: 669 days green material: 736 days
								seed	89 97	0.13 0.099	0.099 0.092	0.22 0.19	0.033 0.031	43 50	
								straw	89 97	0.16 0.065	0.12 0.12	0.28 0.19	0.040 0.040	43 50	
16-2169-02 16-2169-02-T Portugal 2140-408 Vale de Cavalos Europe, South F 2016	Rape PR46W14	1) 08.10.2015 2) 17.04.2016 - 04.05.2016 3) 07.07.2016 - 14.07.2016	56.25 56.25	300 300	18.75 18.75	10.05.2016/0 24.05.2016/14	75	green material	75 75	0.20 0.90	0.064 0.050	0.26 0.95	0.021 0.017	0* 0	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 01304 (k) LOQ: FPF: 0.01 mg/kg DFA: 0.02 mg/kg (expressed as FPF) DFA: 0.0067 mg/kg (expressed as DFA) (l) Method Validation Data: 01304, 16-2169 (m) Storage: straw: 702 days seed: 684 days green material: 720 days
								seed	89 89 89	0.027 0.024 0.019	0.034 0.045 0.029	0.061 0.069 0.048	0.011 0.015 0.010	36 43 50	
								straw	89 89	0.018 0.047 <0.01	<0.02 0.028 <0.02	0.038 0.075 <0.03	<0.0067 0.009 <0.0067	36 43 50	

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included
(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation
(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse
F field

* prior to last treatment
** residue in control

A 2.1.4 Magnitude of residues in livestock

A 2.1.4.1 Livestock feeding studies

In the Annex II dossier, feeding studies conducted with the parent flupyradifurone have been provided. These feeding studies were evaluated by EFSA and considered to be acceptable (EFSA, 2015). Additional feeding studies were conducted with DFA at three different feeding levels in poultry (laying hens) and ruminants (cattle).

A 2.1.4.1.1 Poultry, report RARVN150

Comments of zRMS:	A laying hen feeding study was conducted to measure the difluoroacetic acid residues in hen eggs, egg white, egg yolk, liver, muscle, and fat following 29 consecutive daily oral doses of sodium difluoroacetate at target doses of 0.38 mg/kg (1X group), 1.14 mg/kg (3X group) and 3.80 mg/kg (10X group). These target dose rates corresponded to 1X, 3X, and 10X the anticipated maximum dietary burden of difluoroacetic acid residues in/on livestock feed constructed from maximum reasonably balanced diets for laying hens and a Reasonable Worst Case Diet/Feed (RWCF) approach. The actual dietary burden based on feed consumption data collected in the study for the 1X, 3X, and 10X dosing levels corresponded to 0.38, 1.2, and 3.9 mg sodium difluoroacetate per kg feed, respectively, corresponding to difluoroacetic acid residue concentrations in feed dry matter of 0.31, 0.94, and 3.1 mg/kg, respectively. Difluoroacetic acid residues in eggs reached plateau approximately 7 days after the first dosing. After dosing was discontinued, difluoroacetic acid residue in eggs from the 10X dose level hens declined from 0.23 ppm on Study Day 28 to below the LOQ (<0.010) 14 days after the cessation of dose administration. Difluoroacetic acid residues in the tissues of liver, muscle, and fat from the 10X dose level hens declined during the depuration phase, and were below the LOQ (<0.010) in all tissues by Study Day 43 (14 days after cessation of dose). The residue data provided in this report are suitable for regulatory purposes. The study is acceptable.

Reference:	**KCA 6.4.1/01**
Title:	Difluoroacetic acid - Magnitude of the residue in laying hens
Report:	[redacted] 2017; RARVN150; M-611007-01-1
Authority registration No:	
Guideline(s):	APVMA Residue Guidline No. 23; OPPTS 860.1480; DACO 7.5.1; OECD 505 Residues in Livestook
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

A feeding study was conducted to measure difluoroacetic acid (DFA) residue in eggs, egg white, egg yolk, liver, muscle, and fat from sixty-four ISA Brown (Gold Star) egg-laying hens (*Gallus gallus domesticus*) following 29 consecutive days of morning dosing via oral capsules containing difluoroacetic acid in the form of sodium difluoroacetate.

Materials and methods

Test system, dosing

Two hundred and sixty-five ISA Brown (Gold Star) egg-laying hens (*Gallus gallus domesticus*) were received from a local hatchery and transferred to the test facility approx. fourteen days prior to the initiation of the study (study day -14). Eighty of the delivered hens (20 pens) were placed on acclimation. Sixty-four hens (approximately 23 weeks old at the start of acclimation) were selected for placement on the study based on egg production and health, and the chosen pens were randomly assigned to respective

test groups and subgroups. The animals were allowed *ad libitum* access to water and feed throughout the study.

Dose rates used in this study were calculated using the Reasonable Worst Case Diet/Feed (RWCF) as described in the OECD Guidance Document ENV/JM/MONO(2013)8 in order to cover the anticipated dietary burdens of poultry in Europe. The anticipated dietary burden in Europe (without considering import of feed items as proposed by EFSA) was used as base dose rate (1X) in the feeding study. As difluoroacetic acid (DFA) is a rather strong acid, it was administered in the form of sodium difluoroacetate. The base daily dose of difluoroacetic acid was calculated to be 0.307 mg/kg dry matter feed (0.021 mg/kg body weight per day) by the Reasonable Worst Case Diet/Feed (RWCF) approach.

Dose Group	**Number of Hens**	**Estimated Daily feed Consumption (kg/day)[a]**	**Target Dose (mg salt/kg feed)**	**Estimated Dose Rate (mg salt/hen)[b]**	**Dose level (mg salt/kg bw/day)**
Control	12 (3 groups)	0.13	0.0 (placebo)	0.0 (placebo)	n.a.
1X Dose	12 (3 groups)	0.13	0.38	0.049	0.026
3X Dose	12 (3 groups)	0.13	1.14	0.148	0.078
10X Dose	28 (7 groups)	0.13	3.80	0.494	0.260

[a] The actual feed was determined each week and used to recalculate the dose amounts.
[b] Dose levels are for sodium difluoroacetate and are based on an average weight of 1.9 kg for each laying hen, feed consumption of 0.13 kg dry weight, and dietary burden based on the Reasonable Worst Case model.
n.a. = Not applicable.

The dose was prepared in terms of the dietary burden (mg per kg dry weight feed), and taking into account the weight difference of the salt (118 g/mol) to the acid (96 g/mol). The estimated dietary burden was 0.306 mg/kg feed of difluoroacetic acid which equals 0.377 mg/kg feed of sodium difluoroacetate.

For each dose group, capsules were prepared weekly containing an amount of sodium difluoroacetate at target values of 0.38 mg/kg feed (1X), 1.14 mg/kg feed (3X), and 3.80 mg/kg feed (10X). The capsule dose (mg sodium difluoroacetate) was calculated based on the animal's group average feed consumption, in terms of dry matter, calculated from the feed consumption during the week two weeks prior to dose preparation (e.g., study week 1 feed consumption was used to calculate the dose for study week 3) along with the dose level to which the animal is assigned. Moisture content of the feed was determined weekly prior to capsule preparation. The required capsule dose was recalculated at the beginning of each week.

The actual dose levels based on actual feed intake and actual body weight of the birds are summarized in the table below:

Summary of Actual Sodium Difluoroacetate Dose Administration by Group

Dose Group	**Target Dose (mg/kg feed)**	**Dose (mg/kg) in Study Week**				**Average Dose (mg/kg)**	**Average Dose**	
		1	**2**	**3**	**4**		**(mg salt/kg bw)**	**mg DFA/kg bw**
1X Dose	0.38	0.402	0.355	0.385	0.444	0.396	0.022 [a)]	0.018 [a)]
3X Dose	1.14	1.251	1.077	1.161	1.336	1.206	0.067 [b)]	0.054 [b)]
10X Dose	3.80	4.080	3.698	3.918	4.287	3.996	0.222 [c)]	0.181 [c)]

[a)] Based on the in-life part an average bodyweight (week 1-4) of 2.037 kg and an average feed consumption of 113.8 g dry feed was considered for the calculation of the actual dose
[b)] Based on the in-life part an average bodyweight (week 1-4) of 2.082 kg and an average feed consumption of 115.4 g dry feed was considered for the calculation of the actual dose
[c)] Based on the in-life part an average bodyweight (week 1-4) of 2.023 kg and an average feed consumption of 112.5 kg dry feed was considered for the calculation of the actual dose
Molecular weight of DFA = 96.03 g/mol; molecular weight of sodium salt = 118.01 g/mol

In order to confirm the content and reproducibility and stability of difluoroacetic acid in the dose capsules for at least one week, three capsules were prepared using a nominal feed consumption of 0.13 kg for a 1.9 kg laying hen at the time of Week 1 capsule preparation. These capsules were stored with the test capsules. At the time of the week 2 capsule preparation, three additional stability capsules were prepared. All six capsules were sent to Bayer CropScience for stability analysis. The analysis of the aged capsules (storage time of 10 days) in parallel to the freshly prepared capsules (storage time of 3 days) confirmed

the stability of difluoroacetic acid in the capsules. The average recovered dose of the aged capsules accounted for 119% and the recovery of the freshly prepared capsules for 123%.

Stability of DFA in dose capsules

Treat-ment group	Capsule No.	Date prepared	Date shipped	Nominal concentration (ng DFA/mL)	Date extraction	Date analysis	Measured concentra-tion (ng DFA/mL)	Recovery (%)
1X, aged	-01	2017-05-08	2017-05-17	8.04	2017-05-18	2017-05-18	9.086	113
	-02	2017-05-08	2017-05-17	8.04	2017-05-18	2017-05-18	9.635	120
	-03	2017-05-08	2017-05-17	8.04	2017-05-18	2017-05-18	9.865	123
	Average for aged capsules							**119**
1X, fresh	-01	2017-05-15	2017-05-17	8.04	2017-05-18	2017-05-18	9.847	123
	-02	2017-05-15	2017-05-17	8.04	2017-05-18	2017-05-18	9.737	121
	-03	2017-05-15	2017-05-17	8.04	2017-05-18	2017-05-18	10.034	125
	Average for fresh capsules							**123**

Hens in each dose group were assigned to subgroups. Two control subgroups (A and B) contained six hens each. The 1X and 3X groups contained three subgroups (A, B, and C) of four hens each, and the 10X group contained seven subgroups (A, B, C, A DEP, B DEP, C DEP, and D DEP) of four hens each as animals for a depuration phase were included. During the test period, animals were penned and fed as a subgroup. The hens were fed 16% Layer Poultry Ration ad libitum throughout the study; the quantity of fresh feed offered was sufficient to ensure hens were provided with feed of dry matter weight equal to at least 6.8% of the average body weight. The amount of feed consumed per pen was calculated weekly. Based on the actual feed intake, the does rates simulated sodium difluoroacetate residue concentrations in feed dry matter of 0.38 mg/kg (1X group), 1.2 mg/kg (3X group) and 3.9 mg/kg (10X group), corresponding to difluoroacetic acid residue concentrations in feed dry matter of 0.31, 0.94, and 3.1 mg/kg, respectively.

Eggs were collected twice daily from all dose groups, morning and evening. Doses were administered after the morning egg collection. Eggs were sampled for analysis from the controls and all dose groups on Study days 0, 2, 4, 7, 10, 14, 17, 21, 25, 27, and 28. A composite egg sample was collected by combining the egg white and yolk and discarding the shell from the study day evening eggs and the following morning's eggs collected prior to daily dose administration, with the exception of the eggs collected for study day 27. On day 27, collected eggs from control hens and from all hens of the 10X dose group were separated into egg white and egg yolk samples. Eggs collected on day 27 from the other dosing groups were discarded.
On day 29 (at least 3 hours after the final dose), subgroup A of the control hens (6 birds), all of the 1X and 3X dose group hens (twelve birds per dosing level), and subgroups A, B, and C of the 10X hens (twelve birds) were sacrificed. Liver, muscle, and fat samples were collected from each sacrificed hen for analysis.
The remaining hens (control subgroup B and subgroups A DEP, B DEP, C DEP, and D DEP from the 10X dose level) entered the depuration phase of the study. The 10X depuration hens were sacrificed 3 (A DEP), 7 (B DEP), 14 (C DEP), and 21 (D DEP) days post-dosing (study days 32, 36, 43, and 50). The remaining control hens (subgroup B) were sacrificed on day 50. Additional egg samples were collected from the remaining control and the 10X dose depuration hens on days 31, 35, 38, 42, 45, and 49. Eggs and tissues from the hens sacrificed at each interval were analyzed to monitor the decline of difluoroacetic acid residues.

The collected tissues were stored frozen until homogenized with dry ice. Tissues were kept cold throughout the procedure. The homogenized retention samples were logged into a freezer where they were stored frozen until disposed of or returned to the sponsor for analysis. Storage temperatures for the analytical samples were < -18°C, except for a period of approx. 24 hours when a large amount of fresh tissues were placed in the freezer of the test facility where the in-life part was conducted. The maximum temperature recorded during this period was -2.5°C. In order to confirm the stability of difluoroacetic acid at the increased temperature, a short storage stability run was conducted as part of this study. Control samples of eggs, milk, muscle, kidney, liver and fat were fortified with DFA at a level of 0.1 mg/kg. One set of the fortified samples was analyzed immediately, another set was analyzed after a storage period of 24 hours and a third set of samples after 48 hours.

Overall, the tissue and egg samples in this study were analyzed within 30 days of collection for residue of difluoroacetic acid and do not require any additional freezer storage stability data.

Residues of difluoroacetic acid (DFA) were extracted from eggs, egg white and egg yolk using a mixture of 80:20 (v/v) acetonitrile/water acidified with 2.2 mL/L formic acid by blending. Extraction of DFA residues from muscle and liver was done by using a mixture of 85:15 (v/v) acetonitrile/water acidified with 5 mL/L formic acid. Fat samples were extracted using a mixture of 85:15 (v/v) acetonitrile/water acidified with 5 mL/L formic acid in a first step and heptane in a second step. An isotopic internal standard was added to the samples before sample aliquots were cleaned through a stacked C18 and SCX cartridge pre-washed with acetonitrile. The diluted sample was analyzed by high performance liquid chromatography/triple stage mass spectrometry (LC-MS/MS) according to analytical method RV-006-A17-01 (Kormos, T.; 2017; M-589558-01-1) with modifications.

Quantitation of the analyte was based on the daughter ion transitions of the analyte and the internal standard analog. The relative response of the analyte in each sample was compared to the relative response of the analyte in the standard. The limit of quantitation (LOQ) and the limit of detection (LOD) are summarized in the table below. The limit of detection (LOD) is defined as the lowest concentration of an analyte that can be determined to be statistically different from a blank. The LODs were determined from method validation data obtained from control samples fortified at the respective analyte LOQs. The LODs were calculated by multiplying the standard deviation of recovery measurements at the LOQ by $t_{0.99}$ [where $t_{0.99}$ is the one-tailed t-statistic at the 99% confidence level for the number of replicates (n)].

Summary of LOQ and LODs:

Matrix	Analyte	LOQ (mg/kg)	LOD (mg/kg)
Eggs, egg white, egg yolk	Difluoroacetic acid	0.010	0.0030
Liver	Difluoroacetic acid	0.010	0.0018
Muscle	Difluoroacetic acid	0.010	0.0031
Fat	Difluoroacetic acid	0.010	0.0033

Results and discussion

Residues of difluoroacetic acid in egg and tissue samples were quantitated by high performance liquid chromatography/triple stage mass spectrometry (LC-MS/MS) using a stable, isotopically-labelled internal standard. Acceptable recoveries were achieved for eggs, egg white, and egg yolk at fortification levels of 0.010 and 0.50 mg/kg, with additional recoveries in eggs at 0.020, 0.050, and 0.10 mg/kg. Acceptable recoveries were achieved for liver, muscle, and fat at fortification levels of 0.010 and 0.10 mg/kg, with additional recoveries in liver at 0.85 mg/kg and additional recoveries in muscle at 0.40 mg/kg. The limit of quantitation (LOQ) for difluoroacetic acid was 0.010 mg/kg in all matrices. Details of recovery data are shown in Table A 18. Extraction efficiency was already shown in the corresponding feeding study with flupyradifurone.

The residues of difluoroacetic acid in egg, egg white, egg yolk samples as well as muscle, fat and liver are summarized in Table A 20.

Difluoroacetic acid residues in eggs reached a plateau approximately 7 days after the first dosing. After dosing was discontinued, difluoroacetic acid residue in eggs declined continuously, as shown for the 10X dose level hens.

Mean residues of difluoroacetic acid in muscle were found at 0.029 mg/kg in the 1X dosing group and increased to 0.094 and 0.388 mg/kg in the 3X and 10X dosing groups, respectively.
In fat mean residues of 0.007 mg/kg were determined in the 1X dosing, and increased to 0.021 and 0.069 mg/kg in the 3X and 10X dosing groups, respectively.
In liver mean residues of 0.052 mg/kg were determined in the 1X dosing, and increased to 0.174 and 0.795 mg/kg in the 3X and 10X dosing groups, respectively.

Residues of difluoroacetic acid in eggs as well as in the tissues of liver, muscle, and fat from the 10X

dose level hens declined during the depuration phase, and were below the LOQ (<0.010 mg/kg) in all tissues 14 days after cessation of dose.

Table A 18: Recovery data for DFA residues in poultry matrices

Crop matrix	Analyte	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
Hen eggs	difluoroacetic acid	12	0.010	105, 100, 95, 99, 101, 111, 110, 107, 114, 104, 108, 116	95	116	106	5.4
		4	0.020	107, 101, 96, 82	82	107	97	11.0
		4	0.050	103, 101, 101, 93	93	103	100	4.5
		5	0.10	106, 108, 97, 94, 109	94	109	103	6.6
		5	0.50	95, 99, 96, 97, 97	95	99	97	1.5
		30	overall		82	116	102	7.1
Hen egg white	difluoroacetic acid	3	0.01	95, 94, 92	92	95	94	1.6
		3	0.50	94, 95, 102	94	102	97	4.5
		6	overall		92	102	95	3.6
Hen egg yolk	difluoroacetic acid	3	0.01	89, 87, 96	87	96	91	5.2
		3	0.50	83, 82, 79	79	83	81	2.6
		6	overall		79	96	86	7.1
Hen liver	difluoroacetic acid	5	0.010	101, 93, 100, 99, 98	93	101	98	3.2
		5	0.10	97, 96, 97, 99, 100	96	100	98	1.7
		5	0.85	100, 99, 98, 99, 100	98	100	99	0.8
		15	overall		93	101	98	2.1
Hen muscle	difluoroacetic acid	5	0.010	88, 99, 84, 99, 91	84	99	92	7.3
		5	0.10	91, 93, 92, 94, 87	87	94	91	3.0
		5	0.40	86, 81, 89, 94, 95	81	95	89	6.5
		15	overall		81	99	91	5.7
Hen fat	difluoroacetic acid	5	0.010	99, 96, 99, 100, 93	93	100	97	3.0
		5	0.10	98, 98, 96, 101, 100	96	101	99	2.0
		10	overall		93	101	98	2.5

Mean recoveries and RSD values were recalculated based on rounded values available in the report.

Table A 19: Residue data from poultry feeding study with DFA

Animal dosing level	Matrix/Collection time	Feeding level (target)/actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)	
			Single values	Mean
1X (consists of 3 groups)	Egg/0	(0.38)/0.396 mg/kg (0.017)/0.018 mg DFA/kg bw/d	<LOD, <LOD, <LOD	<LOD
	Egg/2		0.0153, 0.0135, 0.0121	0.0136
	Egg/4		0.0244, 0.0267, 0.0268	0.0260
	Egg/7		0.0320, 0.0375, 0.0318	0.0338
	Egg/10		0.0228, 0.0245, 0.0224	0.0232
	Egg/14		0.0231, 0.0219, 0.0212	0.0221
	Egg/17		0.0308, 0.0312*, 0.0270	0.0301
	Egg/21		0.0325, 0.0343, 0.0276	0.0315
	Egg/25		0.0307, 0.0298, 0.0282	0.0296
	Egg/28		**0.0277, 0.0402, 0.0368**	**0.0349**
3X (consists of 3 groups)	Egg/0	(1.14)/1.206 mg/kg (0.053)/0.054 mg DFA/kg bw/d	<LOD, <LOD, <LOD	<LOD
	Egg/2		0.0364, 0.0506, 0.0304	0.0391
	Egg/4		0.0638, 0.0573, 0.0568	0.0593
	Egg/7		**0.0876, 0.0931, 0.0670**	**0.0826**
	Egg/10		0.0517, 0.0559, 0.0544	0.0540
	Egg/14		0.0446, 0.0541, 0.0505	0.0497
	Egg/17		0.0668, 0.0789, 0.0671	0.0709
	Egg/21		0.0675, 0.0788, 0.0720	0.0728
	Egg/25		0.0652, 0.0745, 0.0653	0.0683
	Egg/28		0.0701, 0.0642, 0.0623	0.0655
10X (consists of 7 groups)	Egg/0	(3.80)/3.996 mg/kg (0.175)/0.181 mg DFA/kg bw/d	<LOD, <LOD, <LOD, <LOD, <LOD, <LOD, <LOD	<LOD
	Egg/2		0.1060, 0.1099, 0.0984, 0.1253, 0.1072, 0.0785, 0.1098	0.1050
	Egg/4		0.1901, 0.1876, 0.2180, 0.1833, 0.2047, 0.1879, 0.2241	0.1994
	Egg/7		**0.2432[a)], 0.2429[a)], 0.2661[a)]**, 0.2208, 0.2484, 0.2572, 0.2715	**0.2500**
	Egg/10		0.2332, 0.2782, 0.2330, 0.2267, 0.2076, 0.2530, 0.2750	0.2438
	Egg/14		0.2424, 0.2192, 0.2471, 0.2191, 0.2479, 0.2347, 0.2487	0.2370
	Egg/17		0.2534, 0.2680, 0.2444, 0.2192, 0.1974, 0.2298, 0.2722	0.2406
	Egg/21		0.2027, 0.2381, 0.2488, 0.2277, 0.2675, 0.2606, 0.2424	0.2411
	Egg/25		0.2265, 0.2311, 0.2290,	0.2233

Animal dosing level	Matrix/Collection time	Feeding level (target)/actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)	
			Single values	Mean
			0.2187, 0.2081, 0.2151, 0.2343	
	Egg/28		0.2324, 0.2618, 0.2294, 0.2160, 0.2550, 0.2134, 0.2092	0.2310
	Egg/31 (3 days after dose cessation)		0.1672, 0.1781, 0.1755, 0.1704	0.1728
	Egg/35 (7 days after dose cessation)		0.0359, 0.0464, 0.0433	0.0419
	Egg/38 (10 days after dose cessation)		0.0142, 0.0134	0.0138
	Egg/42 (14 days after dose cessation)		<LOD, 0.0030	<LOD
	Egg/45 (17 days after dose cessation)		<LOD	-
	Egg/49 (21 days after dose cessation)		<LOD	-
10X	Egg yolk/27	(3.80)/3.996 mg/kg (0.175)/0.181 mg DFA/kg bw/d	0.1120, 0.1168, 0.1432 (values refer to replicate analyses of one sample)	0.1240
10X	Egg white/27	(3.80)/3.996 mg/kg (0.175)/0.181 mg DFA/kg bw/d	0.2540, 0.2494, 0.2535 (values refer to replicate analyses of one sample	0.2523
1X	Muscle/29	(0.38)/0.396 mg/kg (0.017)/0.018 mg DFA/kg bw/d	0.0284, 0.0284, 0.0302	0.0290
3X	Muscle/29	(1.14)/1.206 mg/kg (0.053)/0.054 mg DFA/kg bw/d	0.0850, 0.0938, 0.1026	0.0938
10X	Muscle/29	(3.80)/3.996 mg/kg (0.175)/0.181 mg DFA/kg bw/d	0.3841, 0.3908, 0.3879	0.3876
	Muscle/32: Depuration day 3		0.1345	-
	Muscle/36: Depuration day 7		0.0239	-
	Muscle/43: Depuration day 14		0.0032	-
	Muscle/50: Depuration day 21		<LOD	-
1X	Fat/29	(0.38)/0.396 mg/kg (0.017)/0.018 mg DFA/kg bw/d	0.0085, 0.0064, 0.0059	0.0069
3X	Fat/29	(1.14)/1.206 mg/kg (0.053)/0.054 mg DFA/kg bw/d	0.0202, 0.196, 0.0224	0.0207
10X	Fat/29	(3.80)/3.996 mg/kg (0.175)/0.181 mg DFA/kg bw/d	0.0804, 0.0774, 0.0493	0.0690
	Fat/32: Depuration day 3		0.0182	-
	Fat/36: Depuration day 7		0.0036	-

Animal dosing level	Matrix/Collection time	Feeding level (target)/actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)	
			Single values	**Mean**
	Fat/43: Depuration day 14		<LOD	-
	Fat/50: Depuration day 21		<LOD	-
1X	Liver/29	(0.38)/0.396 mg/kg (0.017)/0.018 mg DFA/kg bw/d	0.0476, 0.0531, 0.0553	0.0520
3X	Liver/29	(1.14)/1.206 mg/kg (0.053)/0.054 mg DFA/kg bw/d	0.1869, 0.1878, 0.1486	0.1744
10X	Liver/29	(3.80)/3.996 mg/kg (0.175)/0.181 mg DFA/kg bw/d	0.7815, 0.8316, 0.7723	0.7951
	Liver/32: Depuration day 3		0.2463	-
	Liver/36: Depuration day 7		0.0429	-
	Liver/43: Depuration day 14		0.0036	-
	Liver/50: Depuration day 21		<LOD	-

* reported value is an average of duplicate analysis

a) values marked in bold refer to eggs taken from animals which were analyzed for organs and tissues. These three values were taken for MRL calculation.

Table A 20: Summary of residue data from poultry feeding study with DFA

Matrix	Feeding level (mg/kg)	Residue levels (mg/kg)					
		n	Min.	Max.	Median (STMdR)	Mean (STMR)	Std. Dev.
Eggs	1X (day 28)	3	0.0277	0.0402	0.0368	0.0349	0.0064
	3X (day 7)	3	0.0670	0.0931	0.0876	0.0826	0.014
	10X (day 7)*	(7) 3	(0.2208) 0.2429	(0.2715) 0.2661	(0.2484) 0.2432	(0.2500) 0.2507	(0.017) 0.0133
	10X (3 days after dose cessation)	4	0.1672	0.1781	0.1729	0.1728	0.0049
	10X (7 days after dose cessation)	3	0.0359	0.0464	0.0433	0.0419	0.0054
	10X (10 days after dose cessation)	2	0.0134	0.0142	0.0138	0.0138	-
	10X (14 days after dose cessation)	2	<LOD	0.0030	<LOD	<LOD	-
	10X (17 days after dose cessation)	1	<LOD	<LOD	-	-	-
	10X (21 days after dose cessation)	1	<LOD	<LOD	-	-	-
Egg yolk	10X (day 27)	1	0.124	0.124	-	-	-
Egg white	10X (day 27)	1	0.252	0.252	-	-	-
Muscle	1X (day 29)	3	0.0284	0.0302	0.0284	0.0290	0.0010
	3X (day 29)	3	0.0850	0.1026	0.0938	0.0938	0.0088
	10X (day 29)	3	0.3841	0.3908	0.3879	0.3876	0.0034
	10X (3 days after dose cessation)	1	0.1345	0.1345	-	-	-
	10X (7 days after dose cessation)	1	0.0239	0.0239	-	-	-
	10X (14 days after dose cessation)	1	0.0032	0.0032	-	-	-
	10X (21 days after dose cessation)	1	<LOD	<LOD	-	-	-
Fat	1X (day 29)	3	0.0059	0.0085	0.0064	0.0069	0.0014
	3X (day 29)	3	0.0196	0.0224	0.0202	0.0207	0.0015
	10X (day 29)	3	0.0493	0.0804	0.0774	0.0690	0.0172
	10X (3 days after dose cessation)	1	0.0182	0.0182	-	-	-
	10X (7 days after dose cessation)	1	0.0036	0.0036	-	-	-
	10X (14 days after dose cessation)	1	<LOD	<LOD	-	-	-
	10X (21 days after dose cessation)	1	<LOD	<LOD	-	-	-
Liver	1X (day 29)	3	0.0476	0.0553	0.0531	0.0520	0.0040
	3X (day 29)	3	0.1486	0.1878	0.1869	0.1744	0.0224
	10X (day 29)	3	0.7723	0.8316	0.7815	0.7951	0.0319
	10X (3 days after dose	1	0.2463	0.2463	-	-	-

Matrix	Feeding level (mg/kg)	Residue levels (mg/kg)					
		n	Min.	Max.	Median (STMdR)	Mean (STMR)	Std. Dev.
	cessation)						
	10X (7 days after dose cessation)	1	0.0429	0.0429	-	-	-
	10X (14 days after dose cessation)	1	0.0036	0.0036	-	-	-
	10X (21 days after dose cessation)	1	<LOD	<LOD	-	-	-

* Remark: values of egg (10X) given in brackets refer to all egg samples analyzed including eggs sampled from animals also investigated during the depuration phase. Values without brackets refer to egg samples from animals which were analyszed for organs and tissues.

Conclusion

The DFA residues detected in eggs and tissues increased linearly dependent on the dose. Highest residues were detected in liver as metabolizing organ and lowest residues were in fat indicating that DFA is not fat soluble.

In eggs, difluoroacetic acid residues reached a plateau approximately 7 days after the first dosing.

Difluoroacetic acid residues did not concentrate in egg yolk (processed commodity concentration factor of 0.54X) and concentrated only slightly in egg white (processed commodity concentration factor 1.1X).

During the depuration phase of the study, the difluoroacetic acid residue in eggs were found to decline rapidly in the 10X dose level hens, from 0.23 mg/kg on study day 28 to below the LOQ (<0.010 mg/kg) by study day 42 (14 days after cessation of dose). Also in tissues, depuration occurred quickly. DFA residues in fat, liver, and muscle - from the 10X dose level hens - declined from 0.069, 0.80, and 0.39 mg/kg, respectively to <0.01 mg/kg at 7 (fat) or 14 days (liver, muscle) after cessation of dose administration. Thus, accumulation of DFA residues in eggs or tissues was nor observed.

Overall, the DFA residues detected in tissues and eggs are in very good agreement with the DFA residues estimates in the flupyradifurone study and confirm the validity of the approach applied in the feeding study submitted in the Annex II dossier.

The residue data provided in this study are suitable for regulatory purposes.

A 2.1.4.1.2 Ruminant, report RARVN149

Comments of zRMS:	A dairy cow feeding study was conducted to measure the difluoroacetic acid residues in cattle milk, cream, skim milk, mesenteric fat, perirenal fat, subcutaneous fat, liver, kidney, and muscle following 29 consecutive daily oral doses of sodium difluoroacetate at target doses of 1.1 mg/kg feed (1X), 5.5 mg/kg feed (5X), and 11 mg/kg feed (10X). These target dose rates corresponded to 1X, 5X, and 10X the anticipated maximum dietary burden of difluoroacetic acid residues in/on livestock feed constructed from maximum reasonably balanced diets for dairy and beef cattle and a Reasonable Worst Case Diet/Feed (RWCF) approach. The actual dietary burden based on feed consumption data collected in the study for the 1X, 5X, and 10X dosing levels corresponded to 1.1, 5.5, and 11 mg sodium difluoroacetate/kg feed, respectively, corresponding to difluoroacetic acid residue concentrations in feed dry matter of 0.89, 4.5, and 8.9 mg/kg, respectively. Difluoroacetic acid residues in milk reached plateau approximately 7 days after the first dosing. After dosing was discontinued, difluoroacetic acid residue in milk from the 10X dose level cows declined from 0.14 ppm to below the LOQ (<0.010) by 10 days after the cessation of dose administration. Difluoroacetic acid residues did not concentrate in cream or skim milk (processed commodity concentration factors = 0.95X and 0.96X, respectively). Difluoroacetic acid residues in the tissues of fat, kidney, liver, and muscle from the 10X dose level cows also declined during the depuration phase and were below the LOQ (<0.010) in all tissues 14 days after the cessation of dose administration. The residue data provided in this report are suitable for regulatory purposes. The study is acceptable.

Reference:	**KCA 6.4.2/01**
Title:	Difluoroacetic acid - Magnitude of the residue in dairy cows
Report:	[redacted] 2017; RARVN149; M-608471-01-1
Authority registration No:	
Guideline(s):	APVMA Residue Guideline No. 23 OCSPP 860.1480 - Meat/Milk/Poultry/Eggs DACO 7.5.1 - Livestock Feeding Study OECD Guideline 505 - Residues in Livestock
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

A feeding study was conducted to measure difluoroacetic acid (DFA) residue in milk, cream, skim milk, mesenteric fat, perirenal fat, subcutaneous fat, liver, kidney, and muscle from fifteen lactating Holstein dairy cows (*Bos taurus*) following 29 consecutive days of morning dosing via oral capsules containing difluoroacetic acid in the form of sodium difluoroacetate.

Materials and methods

Test system, dosing

Twenty Holstein dairy cows (*Bos Taurus*, approximately 3 to 5 years of age) acquired from a local supplier were transferred to the test facility approx. fourteen days prior to the initiation of the study (study day 14). Fifteen of the delivered cows were selected for placement on the study and were randomized to their respective groups. Following randomization, the animals were labelled by study number, animal number, and dose group. The animals were allowed *ad libitum* access to water and feed throughout the study.

Dose rates used in this study were calculated using the Reasonable Worst Case Diet/Feed (RWCF) as described in the OECD Guidance Document ENV/JM/MONO(2013)8 in order to cover the anticipated dietary burdens of ruminant in Europe. The anticipated dietary burden in Europe (without considering import of feed items as proposed by EFSA) was used as base dose rate (1X) in the feeding study. As

difluoroacetic acid (DFA) is a rather strong acid, it was administered in the form of sodium difluoroacetate. The base daily dose of difluoroacetic acid was calculated to be 0.874 mg/kg dry matter feed (0.033 mg/kg body weight per day) by the Reasonable Worst Case Diet/Feed (RWCF) approach. The dose was prepared in terms of the dietary burden (mg/kg feed), and taking into account the weight difference of the salt to the acid, 0.874 mg/kg feed of difluoroacetic acid equals 1.07 mg/kg feed of sodium difluoroacetate. The 1X dose was rounded up to 1.10 mg/kg feed of sodium difluoroacetate.

Dose Group	Number of Cows	Estimated Daily feed Consumption (kg/day)[a]	Target Dose (mg salt/kg feed)	Estimated Dose Rate (mg salt/cow)[b]	Dose level (mg salt/kg bw/day)
Control	2	23	0.0 (placebo)	0.0 (placebo)	n.a.
1X Dose	3	23	1.1	25.3	0.042
5X Dose	3	23	5.5	127	0.21
10X Dose	7	23	11	253	0.42

[a] The actual feed was determined each week and used to recalculate the dose amounts.

[b] Dose levels are for sodium difluoroacetate and are based on an average weight of 600 kg for each dairy cow, feed consumption of 23 kg dry weight, and dietary burden based on the Reasonable Worst Case model.

n.a. = Not applicable.

For each dose group, capsules were prepared weekly containing an amount of sodium difluoroacetate at target values of 1.1 mg/kg feed (1X), 5.5 mg/kg feed (5X), and 11 mg/kg feed (10X). The capsule dose (mg sodium difluoroacetate) was calculated based on the animal's group average feed consumption, in terms of dry matter, calculated from the feed consumption during the first five days of the previous week along with the dose level to which the animal is assigned. Moisture content of the feed was determined weekly prior to capsule preparation. The required capsule dose was recalculated at the beginning of each week. Dosing rates were corrected based on the purity of the test substance (99.7%). The doses were prepared by GML by weighing the appropriate quantity of technical grade sodium difluoroacetate, a powder, directly into gelatin dose capsules. Prepared capsules were stored frozen.

The actual dose levels based on actual feed intake and actual body weight of the animals are summarized in the table below:

Summary of Actual Sodium Difluoroacetate Dose Administration by Group

Dose Group	Target Dose (mg salt/kg feed)	Dose (mg/kg) in Study Week				Average Dose (mg/kg)	Average Actual Dose	
		1	2	3	4		(mg salt/kg bw)	mg DFA/kg bw
1X Dose	1.1	1.02	1.13	1.09	1.27	1.13	0.040 [a)]	0.032 [a)]
5X Dose	5.5	5.34	5.62	5.54	6.32	5.71	0.21 [b)]	0.17 [b)]
10X Dose	11	10.97	11.12	10.36	12.99	11.36	0.41 [c)]	0.33 [c)]

a) Based on the in-life part an average bodyweight (week 1-4) of 634.6 kg and an average feed consumption of 22.33 kg dry feed was considered for the calculation of the actual dose

b) Based on the in-life part an average bodyweight (week 1-4) of 580.6 kg and an average feed consumption of 21.73 kg dry feed was considered for the calculation of the actual dose

c) Based on the in-life part an average bodyweight (week 1-4) of 607.8 kg and an average feed consumption of 21.95 kg dry feed was considered for the calculation of the actual dose

In order to confirm the content, reproducibility and stability of difluoroacetic acid in the dose capsules for at least one week, three capsules were prepared using a nominal feed consumption of 23 kg for a 600 kg dairy cow at the time of Week 1 capsule preparation. These capsules were stored with the test capsules. At the time of the week 2 capsule preparation, three additional stability capsules were prepared. The control and test capsules were stored frozen; storage temperatures ranged from -5.5 °C to -18.5 °C. All six capsules were sent to Bayer CropScience for stability analysis. The analysis of the aged capsules in parallel to the freshly prepared capsules confirmed the stability of difluoroacetic acid in the capsules. The average recovered dose of the aged capsules accounted for 115% and the recovery of the freshly prepared capsules for 118%.

Treat-ment group	Capsule No.	Date prepared	Date shipped	Nominal concentration (ng DFA/mL)	Date prepared	Measured concentration (ng DFA/mL)	Recovery (%)
1X, aged	-01	2017-03-13	2017-03-21	5.07	2017-03-23	6.06	120
	-02	2017-03-13	2017-03-21	5.07	2017-03-23	5.69	112
	-03	2017-03-13	2017-03-21	5.07	2017-03-23	5.81	115
						Average for aged capsules	**115**
1X, fresh	-01	2017-03-15	2017-05-17	5.27	2017-03-23	6.32	120
	-02	2017-03-15	2017-05-17	5.27	2017-03-23	6.21	118
	-03	2017-03-15	2017-05-17	5.27	2017-03-23	6.18	117
						Average for fresh capsules	**118**

Cows were randomly assigned to the four dose groups: control, 1X , 5X and 10X. Two cows were assigned as controls (one of which was designated a depuration cow), thee cows were assigned to the 1X and 5X treatment groups, and seven cows were assigned to the 10X group. Four of the 10X cows were initially designated as depuration cows, but one of these cows fell ill during the course of the study and was sacrificed with the non-depuration cows. Therefore, depuration was only studied using three 10X cows. The animals were dosed orally once per day each morning after milking and feeding. The control animal received a placebo (empty capsule) concurrently with the treated animals. The cows were fed a combination of Genesis Midwest dairy ration (grain), baled hay, and corn silage; the quantity of components offered was adjusted weekly to ensure cows were provided with feed of dry matter weight equal to at least 3.8% of their body weights. The amount of feed consumed was monitored daily. Based on the actual feed intake, the dose rates simulated sodium difluoroacetate residue concentrations in feed dry matter of 1.1 mg/kg (1X group), 5.7 mg/kg (5X group) and 11 mg/kg (10X group), corresponding to difluoroacetic acid residue concentrations in feed dry matter of 0.92, 4.6, and 9.2 mg/kg, respectively. These residue concentrations are equivalent to 0.032 mg DFA/kg bw (1X group), 0.17 mg DFA/kg bw (5X group) and 0.33 mg DFA/kg bw (10X group).

Milk was collected twice daily (evening and morning), and the milk was weighed after each milking. On study days 0, 2, 4, 7, 10, 14, 17, 21, 25 and 28, a subsample of the evening milk (approx. 500 mL) was retained (refrigerated) and then composited with the morning milk proportional to the amount collected at each milking. Additional 25-day milk was collected for processing into whey (skim milk) and cream (milk fat) from both control cows and all seven of the 10X dose group cows. One additional cream sample was collected from each group for crude fat content determination.

On study day 29, all animals except four depuration cows were humanely sacrificed at least three hours following the last dose. One cow (number 25) was initially slated to be a fourth depuration cow but fell ill with persistent diarrhea during the course of dosing and was instead terminated on day 29. The 10X depuration cows were sacrificed 3, 7, and 14 days post-dosing (on study days 32, 36, and 43). The remaining control cow was also sacrificed on study day 43.

Representative samples of liver (distal portions of each lobe), kidney (center and both endsof each kidney), fat (approximately equal composite of omental, perirenal, and subcutaneous), and muscle (composite of loin, round, and flank) were collected. The tissue samples were homogenized in the presence of dry ice and the homogenized samples were placed in a freezer prior to shipping.

The collected tissues were stored frozen until homogenized with dry ice. Tissues were kept cold throughout the procedure. The homogenized retention samples were logged into a freezer where they were stored frozen until disposed of or returned to the sponsor for analysis. Samples were stored at a temperature of ≤-18°C except during a period of approximately 24 hours when a large amount of fresh tissues were placed in the freezer, in which a maximum recorded temperature of -5°C was reached. In order to confirm the stability of difluoroacetic acid at the increased temperature, a short storage stability run was conducted as part of the laying hen feeding study. Control samples of eggs, milk, muscle, kidney, liver and fat were fortified with DFA at a level of 0.1 mg/kg. One set of the fortified samples was analyzed immediately, another set was analyzed after a storage period of 24 hours and a third set of samples after 48 hours.

Overall, the tissue and milk samples in this study were analyzed within 30 days of collection for residue of difluoroacetic acid and do not require any additional freezer storage stability data.

Residues of difluoroacetic acid (DFA) were extracted from milk, cream, skim milk, and as well from muscle, liver, and kidney using a mixture of 85:15 (v/v) acetonitrile/water acidified with 5 mL/L formic acid by blending. Fat samples were extracted using a mixture of 85:15 (v/v) acetonitrile/water acidified with 5 mL/L formic acid in a first step and heptane in a second step. An isotopic internal standard was added to the samples before sample aliquots were cleaned through a stacked C18 and SCX cartridge pre-washed with acetonitrile. The diluted sample was analyzed by high performance liquid chromatography/triple stage mass spectrometry (LC-MS/MS) according to analytical method RV-006-A17-01 (Kormos, T.; 2017; M-589558-01-1).

Quantitation of the analyte was based on the daughter ion transitions of the analyte and the internal standard analog. The relative response of the analyte in each sample was compared to the relative response of the analyte in the standard. The limit of quantitation (LOQ) and the limit of detection (LOD) are summarized in the table below. The limit of detection (LOD) is defined as the lowest concentration of an analyte that can be determined to be statistically different from a blank. The LODs were determined from method validation data obtained from control samples fortified at the respective analyte LOQs. The LODs were calculated by multiplying the standard deviation of recovery measurements at the LOQ by $t_{0.99}$ [where $t_{0.99}$ is the one-tailed t-statistic at the 99% confidence level for the number of replicates (n)].

Summary of LOQ and LODs:

Matrix	Analyte	LOQ (mg/kg)	LOD (mg/kg)
Milk, cream, skim milk	Difluoroacetic acid	0.010	0.0013
Fat (mesenteric, perirenal, subcutaneous)	Difluoroacetic acid	0.010	0.0014
Liver	Difluoroacetic acid	0.010	0.0024
Kidney	Difluoroacetic acid	0.010	0.0017
Muscle	Difluoroacetic acid	0.010	0.0010

Results and discussion

Residues of difluoroacetic acid in milk and tissue samples were quantitated by high performance liquid chromatography/triple stage mass spectrometry (LC-MS/MS) using a stable, isotopically-labelled internal standard. Acceptable recoveries were achieved for milk, cream, and skim milk at fortification levels of 0.010 and 0.50 mg/kg, with additional recoveries in milk at 0.10 mg/kg. Acceptable recoveries were achieved for fat (mesenteric, perirenal, and subcutaneous), liver, kidney, and muscle at fortification levels of 0.010, 0.10, and 1.4 mg/kg, with additional recoveries in liver and kidney at 0.70 mg/kg. The limit of quantitation (LOQ) for difluoroacetic acid was 0.010 mg/kg in all matrices. Details of recovery data are shown in Table A 21. Extraction efficiency was already shown in the corresponding feeding study with flupyradifurone.

Feed consumption and milk production were not adversely affected by treatment with DFA. The overall average daily feed consumption on a dry matter basis per animal across all dosing groups during the test period (study day 1 to study day 28) was 22.6 kg. The average daily feed consumption was 23.6 kg for the control group. Animals were observed to be healthy and normal throughout the study with the exception of cow number 25, a cow in the 10X group initially slated to be part of the depuration phase. Cow 25 experienced persistent health issues (diarrhea) during the dosing phase of the study, and it was decided to sacrifice the cow on Day 29 at the end of the dosing phase instead of continuing it into the depuration phase. No gross health findings were noted for cow 25 at the time of necropsy.

Residues of difluoroacetic acid in milk, cream, and skim milk samples as well as in tissue samples (fat, liver, kidney and muscle) are summarized in the table below (Table A 22).

In addition to the general testing of milk, day-25 milk samples from the 10X dose group were separated into cream and whey, in order to determine if the residues preferentially collect in more aqueous or fatty compartments. The processing/transfer factor (PF) indicated no preference for DFA to concentrate in cream (PF of 0.95) or in whey (PF of 0.96).

With respect to milk, the nominal highest DFA residues were found in the day-25 samples of all dose

groups (with mean concentrations of 0.0150, 0.0674 and 0.1715 mg/kg in 1X, 5X and 10X dose groups, respectively). However the residues started to plateau already 7 to 14 days after the first dose. The residue levels of the samples collected at study day 28 (last dosing) were always at the same level or lower than the residues detected in the samples collected at study day 14.
After dosing was discontinued, difluoroacetic acid residue in milk declined continuously, as shown for the 10X dose level depuration cows. Residues declined from 0.15 mg/kg on study day-28 (last dose administration) to below the LOQ (<0.010 mg/kg) 10 days after the cessation of dose administration (study day 38).

Mean residues of difluoroacetic acid in mesenteric, perirenal and subcutaneous fat were found at 0.0360, 0.0266, and 0.0741 mg/kg in the 1X dosing group and increased to 0.2014, 0.1921 and 0.3362 mg/kg and 0.3934, 0.4232 and 0.7379 mg/kg in the 5X and 10X dosing groups, respectively.
In liver mean residues of 0.0534 mg/kg were determined in the 1X dosing, and increased to 0.2958 and 0.6787 mg/kg in the 5X and 10X dosing groups, respectively.
In kidney mean residues of 0.1062 mg/kg were determined in the 1X dosing, and increased to 0.5084 and 1.0622 mg/kg in the 5X and 10X dosing groups, respectively.

In the depuration phase, DFA residues in omental fat, perirenal fat, subcutaneous fat, liver, kidney, and muscle from the 10X dose group cows declined from 0.39, 0.42, 0.74, 0.68, 1.1, and 0.67 mg/kg, respectively on study day 28 (last dose administration), to <LOQ at 10-14 days after cessation of dose administration (study day 38-43).

Table A 21: Recovery data for DFA residues in ruminant matrices

Crop matrix	Analyte	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
Cattle milk	DFA	17	0.010	77, 70, 82, 76, 81, 90, 86, 90, 90, 120, 72, 73, 81, 76, 72, 82, 71	70	120	82	14.7
		5	0.10	109, 108, 108, 110, 106	106	110	108	1.4
		5	0.50	108, 109, 110, 105, 107	105	110	108	1.8
		27	overall		70	120	91	17.5
Cattle cream	DFA	3	0.01	90, 98, 97	90	98	95	4.6
		3	0.50	104, 108, 106	104	108	106	1.4
		6	overall		90	108	100	6.5
Cattle skim milk	DFA	3	0.01	87, 87, 84	84	87	86	2.0
		3	0.50	104, 107, 106	104	107	106	1.4
		6	overall		84	107	96	11.3
Cattle fat, mesenteric	DFA	5	0.010	98, 97, 90, 82, 87	82	98	91	7.4
		5	0.10	106, 109, 105, 107, 106	105	109	107	1.4
		7	1.4	93, 94, 95, 96, 94, 96, 98	93	98	95	1.8
		17	overall		82	109	97	7.6
Cattle fat, perirenal	DFA	5	0.010	105, 103, 102, 82, 78	78	105	94	13.7
		5	0.10	104, 105, 104, 105, 107	104	107	105	1.2
		7	1.4	92, 99, 98, 97, 98, 92, 82	82	99	94	6.4
		17	overall		78	107	97	9.3
Cattle fat, subcutaneous	DFA	5	0.010	98, 102, 94, 104, 105	94	105	101	4.5
		5	0.10	104, 111, 102, 102, 100	100	111	104	4.1
		7	1.4	102, 88, 100, 93, 87, 90, 88	87	102	93	6.6
		17	overall		87	111	98	7.1
Cattle, liver	DFA	9	0.010	104, 100, 94, 93, 111, 84, 98, 85, 98	84	111	96	8.9
		5	0.10	101, 105, 112, 99, 115	99	115	106	6.5
		1	0.70	102	102	102	-	-
		5	1.4	103, 98, 99, 112, 113	98	113	105	6.8
		20	overall		84	115	101	8.5
Cattle, kidney	DFA	8	0.010	111, 106, 110, 110, 79, 97, 113, 106	79	113	104	10.8
		5	0.10	100, 110, 112, 104, 110	100	112	107	4.7
		1	0.70	109	109	109	-	-
		5	1.4	101, 107, 106, 120, 111	101	120	109	6.5
		19	overall		79	120	106	7.9
Cattle, muscle	DFA	9	0.010	96, 99, 93, 91, 94, 92, 97, 89, 94	89	99	94	3.3
		5	0.10	102, 107, 104, 97, 98	97	107	102	4.1
		5	1.4	94, 104, 108, 110, 114	94	114	106	7.2
		19	overall		89	114	99	7.1

Mean recoveries and RSD values were recalculated based on rounded values available in the report, therefore minor deviations may occur.

Table A 22: Residue data from ruminant feeding study with DFA

Animal dosing level	Matrix/Collection time	Feeding level actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)[a]	
			Single values	Mean
1X 3 animals	Milk/0	1.13 mg/kg 0.032 mg DFA/kg bw/d	<LOD, <LOD, <LOD	<LOD
	Milk/2		0.0039, 0.0031, 0.0071	0.0047
	Milk/4		0.0065, 0.0056, 0.0102	0.0074
	Milk/7		0.0101, 0.0086, 0.0177	0.0121
	Milk/10		0.0133, 0.0092, 0.0161	0.0128
	Milk/14		0.0133, 0.0101, 0.0161	0.0131
	Milk/17		0.0136, 0.0094, 0.0139	0.0123
	Milk/21		0.0148, 0.0101, 0.0193	0.0147
	Milk/25		**0.01515*, 0.01025 *, 0.0197***	**0.0150**
	Milk/28		0.0134, <0.010, 0.0154	0.0129
5X 3 animals	Milk/0	5.71 mg/kg 0.17 mg DFA/kg bw/d	<LOD, <LOD, <LOD	<LOD
	Milk/2		0.0302, 0.0271, 0.0353	0.0308
	Milk/4		0.0445, 0.0399, 0.0538	0.0461
	Milk/7		0.0551, 0.0535, 0.0648	0.0578
	Milk/10		0.0613, 0.0528, 0.0697	0.0613
	Milk/14		0.0655, 0.0631, 0.0714	0.0667
	Milk/17		0.0730, 0.0604, 0.0640	0.0658
	Milk/21		0.0675, 0.0617, 0.0657	0.0650
	Milk/25		**0.06345*, 0.06665*, 0.0722***	**0.0674**
	Milk/28		0.0595, 0.0721, 0.0578	0.0631
10X 7 animals (including four depurations cows)	Milk/0	11.36 mg/kg 0.33 mg DFA/kg bw/d	<LOD, <LOD, <LOD, <LOD, <LOD, <LOD, <LOD	<LOD
	Milk/2		0.0614, 0.0589, 0.0539, 0.1278, 0.0591, 0.0950, 0.0795	0.0765
	Milk/4		0.0897, 0.0893, 0.0850, 0.1648, 0.1069, 0.1587, 0.1313	0.1179
	Milk/7		0.0990, 0.1072, 0.1111, 0.2721, 0.1241, 0.2098, 0.1576	0.1544
	Milk/10		0.1120, 0.1145, 0.1000, 0.1491, 0.1485, 0.2058, 0.1755	0.1436
	Milk/14		0.1112, 0.1180, 0.1089, 0.1538, 0.1498, 0.2090, 0.1778	0.1469
	Milk/17		0.1050, 0.1236, 0.1071, 0.1392, 0.1503, 0.2202, 0.1548	0.1429
	Milk/21		0.1103, 0.1321, 0.1132, 0.2225, 0.1445, 0.2306,	0.1601

Animal dosing level	Matrix/Collection time	Feeding level actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)[a]	
			Single values	**Mean**
			0.1678	
	Milk/25		**0.10645*, 0.1303*, 0.13825*, 0.2685*,** 0.15065*, 0.2297*, 0.17645*	**0.1715**
	Milk/28		0.1003, 0.1323, 0.1278, 0.1758, 0.1411, 0.2126, 0.1912	0.1544
	Milk/31 depuration day 3		0.0669, 0.1028, 0.0579	0.0759
	Milk/35 depuration day 7		0.0238, 0.0092	0.0165
	Milk /38 depuration day 10		<LOD	-
	Milk /42 depuration day 14		<LOD	-
10X	Cream/25	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.1604, 0.1669, 0.1607	0.1627
10X	Skim milk/25	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.1673, 0.1657, 0.1633	0.1654
1X	Mesenteric fat/29	1.13 mg/kg 0.032 mg DFA/kg bw/d	0.0655, 0.0264, 0.0160	0.0360
5X	Mesenteric fat/29	5.71 mg/kg 0.17 mg DFA/kg bw/d	0.2092, 0.3011, 0.0938	0.2014
10X	Mesenteric fat/29	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.6229, 0.2658, 0.4237, 0.2612	0.3934
	Mesenteric fat /32 depuration day 3		0.1941	-
	Mesenteric fat /36 depuration day 7		0.0123	-
	Mesenteric fat /43 depuration day 14		<LOD	-
1X	Perirenal fat/29	1.13 mg/kg 0.032 mg DFA/kg bw/d	0.0517, 0.0183, 0.0099	0.0266

Animal dosing level	Matrix/Collection time	Feeding level actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)[a]	
			Single values	Mean
5X	Perirenal fat/29	5.71 mg/kg 0.17 mg DFA/kg bw/d	0.1723, 0.3495, 0.0544	0.1921
10X	Perirenal fat/29	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.6523, 0.2229, 0.4977, 0.3200	0.4232
	Perirenal fat /32 depuration day 3		0.1329	-
	Perirenal fat /36 depuration day 7		0.0091	-
	Perirenal fat /43 depuration day 14		<LOD	-
1X	Subcutaneous fat/29	1.13 mg/kg 0.032 mg DFA/kg bw/d	0.1312, 0.0598, 0.0313	0.0741
5X	Subcutaneous fat/29	5.71 mg/kg 0.17 mg DFA/kg bw/d	0.2854, 0.5516, 0.1717	0.3362
10X	Subcutaneous fat/29	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.9496, 0.5394, 0.7863, 0.6762	0.7379
	Subcutaneous fat /32 depuration day 3		0.2753	-
	Subcutaneous fat /36 depuration day 7		0.0287	-
	Subcutaneous fat /43 depuration day 14		<LOD	-
1X	Liver/29	1.13 mg/kg 0.032 mg DFA/kg bw/d	0.0648, 0.0471, 0.0484	0.0534
5X	Liver/29	5.71 mg/kg 0.17 mg DFA/kg bw/d	0.3276, 0.3126, 0.2471	0.2958
10X	Liver/29	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.5009, 0.7085, 0.8970, 0.6082	0.6787
	Liver/32 depuration day 3		0.1934	-
	Liver/36 depuration day 7		0.0531	-
	Liver/43 depuration day 14		0.0028*	-
1X	Kidney/29	1.13 mg/kg 0.032 mg DFA/kg bw/d	0.1194, 0.0998, 0.0995	0.1062

Animal dosing level	Matrix/Collection time	Feeding level actual (mg/kg) (mg/kg bw/d)	Residues (mg/kg)[a]	
			Single values	Mean
5X	Kidney/29	5.71 mg/kg 0.17 mg DFA/kg bw/d	0.5265, 0.5234, 0.4754	0.5084
10X	Kidney/29	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.8677, 1.2408, 1.2745, 0.8657	1.0622
	Kidney/32 depuration day 3		0.3341	-
	Kidney /36 depuration day 7		0.0847	-
	Kidney /43 depuration day 14		0.0026*	-
1X	Muscle/29	1.13 mg/kg 0.032 mg DFA/kg bw/d	0.0896, 0.0603, 0.0587	0.0695
5X	Muscle/29	5.71 mg/kg 0.17 mg DFA/kg bw/d	0.3355, 0.3510, 0.3015	0.3293
10X	Muscle/29	11.36 mg/kg 0.33 mg DFA/kg bw/d	0.5705, 0.6681, 0.8013, 0.6502	0.6725
	Muscle/32 depuration day 3		0.2185	-
	Muscle/36 depuration day 7		0.0553	-
	Muscle/43 depuration day 14		<LOD*	-

Remark: To create each milk sample, milk collected on the evening of the noted study day was pooled with milk collected the following morning. Animals were sacrificed post milking the following morning. Thus, for milk samples depuration day 3 is Study Day 31, while for tissue matrices depuration day 3 is Study Day 32, and so on for the remaining depuration days.
* reported value is an average of duplicate analysis (values recalculated based on single values given in the report)
(a): DFA, expressed as DFA

Table A 23: Summary of residue data from ruminant feeding study with DFA

Matrix	Feeding level (mg/kg)	Residue levels (mg/kg)					
		n	Min.	Max.	Median	Mean	Std. Dev.
Milk	1X (day 25)	3	0.01025	0.0197	0.0152	0.0150	0.0047
	5X (day 25)	3	0.0635	0.0722	0.0667	0.0674	0.0045
	10X (day 25)*	(7) 4	(0.1065) 0.1065	(0.2685) 0.2685	(0.1507) 0.1343	(0.1715) 0.1609	(0.0582) 0.0730
Cream	10X (day 25)	3	0.1604	0.1669	0.1607	0.1627	0.0037
Skim milk	10X (day 25)	3	0.1633	0.1673	0.1657	0.1654	0.0020
Mesenteric fat	1X	3	0.0160	0.0655	0.0264	0.0360	0.0261
	5X	3	0.0938	0.3011	0.2092	0.2014	0.1039
	10X	4	0.2612	0.6229	0.3447	0.3934	0.1707
	10X (3 days after dose cessation)	1	0.1941	0.1941	-	-	-
	10X (7 days after dose cessation)	1	0.0123	0.0123	-	-	-
	10X (14 days after dose cessation)	1	<LOD	<LOD	-	-	-
Perirenal fat	1X	3	0.0099	0.0517	0.0183	0.0266	0.0221
	5X	3	0.0544	0.3495	0.1723	0.1921	0.1485
	10X	4	0.2229	0.6523	0.4088	0.4232	0.1904
	10X (3 days after dose cessation)	1	0.1329	0.1329	-	-	-
	10X (7 days after dose cessation)	1	0.0091	0.0091	-	-	-
	10X (14 days after dose cessation)	1	<LOD	<LOD	-	-	-
Subcutaneous fat	1X	3	0.0313	0.1312	0.0598	0.0741	0.0515
	5X	3	0.1717	0.5516	0.2854	0.3362	0.195
	10X	4	0.5394	0.9496	0.7313	0.7379	0.1736
	10X (3 days after dose cessation)	1	0.2753	0.2753	-	-	-
	10X (7 days after dose cessation)	1	0.0287	0.0287	-	-	-
	10X (14 days after dose cessation)	1	<LOD	<LOD	-	-	-

Matrix	Feeding level (mg/kg)	Residue levels (mg/kg)					
		n	Min.	Max.	Median	Mean	Std. Dev.
Liver	1X	3	0.0471	0.0648	0.0484	0.0534	0.0099
	5X	3	0.2471	0.3276	0.3126	0.2958	0.0428
	10X	4	0.5009	0.8970	0.6583	0.6787	0.1684
	10X (3 days after dose cessation)	1	0.1934	0.1934	-	-	-
	10X (7 days after dose cessation)	1	0.0531	0.0531	-	-	-
	10X (14 days after dose cessation)	1	0.0028	0.0028	-	-	-
Kidney	1X	3	0.0995	0.1194	0.0998	0.1062	0.0114
	5X	3	0.4754	0.5265	0.5234	0.5084	0.0287
	10X	4	0.8657	1.2745	1.0543	1.0622	0.2262
	10X (3 days after dose cessation)	1	0.3341	0.3341	-	-	-
	10X (7 days after dose cessation)	1	0.0847	0.0847	-	-	-
	10X (14 days after dose cessation)	1	0.0026	0.0026	-	-	-
Muscle	1X	3	0.0587	0.0896	0.0603	0.0695	0.0174
	5X	3	0.3015	0.3510	0.3355	0.3293	0.0253
	10X	4	0.5705	0.8013	0.6592	0.6725	0.0958
	10X (3 days after dose cessation)	1	0.2185	0.2185	-	-	-
	10X (7 days after dose cessation)	1	0.0553	0.0553	-	-	-
	10X (14 days after dose cessation)	1	<LOD	<LOD	-	-	-

* Remark: values of milk (10X) given in brackets refer to all milk samples analyzed including milk sampled from animals also investigated during the depuration phase. Values without brackets refer to milk samples from animals which were analyszed for organs and tissues.

Conclusion

The DFA residues detected in milk, organs and tissues increased linearly dependent on the dose. Highest residues were detected in liver and kidney as metabolizing organs.

In milk, difluoroacetic acid residues reached a plateau approximately 7 days after the first dosing.

Difluoroacetic acid residues did not concentrate in cream or skim (processed commodity concentration factor of 0.95X and 0.96X, respectively).

During the depuration phase of the study, the difluoroacetic acid residue in milk were found to decline rapidly in the 10X dose level cows, from 0.15 mg/kg on study day 28 to below the LOQ (<0.010 mg/kg) by study day 38 (10 days after cessation of dose). Also in organs and tissues, depuration occurred quickly.

DFA residues in fat, liver, and muscle - from the 10X dose level hens - declined to <0.01 mg/kg (LOQ) at 7 (perirenal fat) or at 14 days (mesenteric and subcutaneous fat, kidney, liver and muscle) after cessation of dose administration. Thus, accumulation of DFA residues in milk, organs or tissues was not observed. The residue data provided in this study are suitable for regulatory purposes.

A 2.1.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

A 2.1.5.1 Distribution of the residue in peel/pulp

No additional studies are submitted.

A 2.1.5.2 Processing studies on a core set of representative processes

Additional processing studies were conducted in commodities with high oil content soybean and cotton as as well as for barley, wheat, and corn (maize), which are presented within this submission as some of the processed commodities are potential feed items and therefore of interest when estimating the dietary burden of livestock.

A 2.1.5.2.1 Soybean, study report RARVY029

Comments of zRMS:	Following a five-fold (5X) exaggerated application of BYI 02960 to two separate trials of soybeans, mature seed was collected. Aspirated grain fractions were then generated and the remaining soybean seed was processed into meal, hulls, refined oil, soymilk, and defatted flour. Significant concentration of BYI 02960 and DFA residues and of the total residue was only detected in the aspirated grain fraction. The mean processing factor was 14 for BYI 02960, 6.5 for DFA and 12.0 for the total residue. The average total BYI 02960 residue did not concentrate (processing factor <1X) in all soybean processed commodities. A slight concentration could be assumed in meal and defatted flour, with processing factors of slightly above 1 for BYI 02960, DFA and the total residue. The preparation steps for refined oil and milk result in a clear reduction of residues. The study is acceptable.

Reference:	**KCA 6.5.3/01**
Title:	BYI 02960 200 SL - Magnitude of the residue in/on soybean processed commodities
Report:	Lenz, C.; 2012; RARVY029; M-428939-01-2
Authority registration No:	
Guideline(s):	EPA Ref.: OPPTS 860.1500, Crop Field Trials PMRA Ref.: DACO 7.4.5. Processed Food/Feed OECD Test Guideline 508: Residues in Processed Commodities (Adopted Oct., 3 2008)
Deviations:	Weather data, pesticide history data, irrigation records, maintenance chemical applications, field sample weights, cultural practices records, soil characterization and plot slope information were not collected under GLP. A protocol amendment identifying the analytical method to be used in the study was not signed until after sample analyses were initiated.
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

Two field trials were conducted to measure the magnitude of flupyradifurone residues in/on soybean seed and processed commodities following two broadcast foliar spray applications of Flupyradifurone 200 SL at exaggerated application rates. Flupyradifurone 200 SL is a soluble concentrate formulation containing 200 g a.s./L.

Flupyradifurone 200 SL was applied twice to soybean plants at individual application rates of 1 kg/ha. The last application was scheduled to be 21 days before harvest. All applications were made using ground-based equipment. The additive, Dyne-Amic 0.25% v/v, was used in all applications.

Single composite soybean seed RAC samples (raw agricultural commodity) were collected at 19 or 21 days after the last treatment from the treated plots. Single composite samples of seed were collected from the control plots on the same day the respective samples were collected from the treated plots.

The residues of flupyradifurone and DFA were quantitated by LC-MS/MS using stable isotopically labelled internal standards using method RV-001-P10-02 (Ying Li; 2010; M-401023-01-2) with some modifactions, also known as method 01304). The limits of quantitation were 0.01 mg/kg for flupyradifurone and 0.05 mg/kg for DFA, when expressed in parent compound equivalents. The individual analyte residues (parent flupyradifurone and DFA, expressed in parent equivalents) were summed to give a total flupyradifurone residue. Residue measurements below the analyte LOQ were summed into the total flupyradifurone residue value as the analyte LOQ value. Prior and parallel to the residue analysis, the method was validated by recovery experiments.

Samples of soybean RAC and processed fractions were were held in frozen storage for a maximum of 254 days prior to extraction (8 months). The storage time is within the acceptable range for storage stability of the residues of flupyradifurone in matrices with high oil content.

Triplicate subsamples of soybean seeds (RAC) were removed and the remaining soybean seed was used to collect aspirated grain fractions and to generate the processed commodities of meal, hulls, refined oil, soymilk and defatted flour. As requested fractions were collected, they were placed in frozen storage within 2 hours of collection.

Soybean grain samples were processed in a way that simulates industrial practice as closely as possible. Because of compliance monitoring requirements and sample size, the samples were processed by batch rather than continuous, as in commercial operation.

Generation of aspirated grain fraction (AGF):
For generation of aspirated grain fractions, the moisture content of the soybean was determined and if above 13.0 percent, the samples were dried in an oven at 43-57°C until the moisture content was 10-13%. To generate aspirated grain fractions, the soybean grain samples were split into two batches. Each batch was placed in the dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved in the system, aspiration was used to remove light impurities (grain dust). Each batch was moved for 120 minutes. Light impurities were classified using the following sieves: 2360 micron (8 mesh); 2000 micron (10mesh); 1180 micron (16 mesh); 850 micron (20 mesh); and 425 micron (40 mesh). After classification of each sample, the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF). A representative sample of the AGF was removed and the ash content was determined.
Following generation, a representative sample of the generated whole soybean grain was collected for further processing.

Cleaning:
Prior to processing, generated samples were cleaned by aspiration and screening. Light material was separated from the sample using a Kice aspirator. After aspiration, the sample was screened using a Hance Corporation cleaner to separate large and small foreign particles (screenings) from the cleaned grain sample.

Processing:
Remainder of whole soybeans were fed into an A.T. Ferrell roller mill to crack the hull and liberate the kernel. After hulling, the material was passed through the Kice aspirator to separate hull and kernel material. Moisture content of the kernel was determined and adjusted to 13.5%. Adjusted kernel was allowed to temper for at least twelve hours at ambient temperature before any further processing. Moisture adjusted kernel material was heated to 71-79°C in a Marion mixer and flaked in an A. T. Ferrell

flaking roll with a gap setting of 0.02-0.03 cm. A portion of the flakes were removed for production of defatted soy flour. Flakes were taken directly to solvent extraction with no additional processing.

Remaining flakes were extruded in a Readco/Teledyne continuous processor, where they were turned into collets by direct steam injection and compression. Collets exited the processor at 93-121°C. After extrusion, the collets were dried in the oven at 66-82°C for 30-40 minutes. Flakes and collets were then solvent extracted to collect crude oil. The flakes and collets were placed in separate stainless steel batch extractors and submerged in 49-60°C hexane (solvent). After 30 minutes, the miscella (crude oil and hexane) was drained and fresh hexane was added to repeat the cycle two more times. Final two washes were for 15 minutes each.
Following the final draining, the extracted collets were desolventized in a Reliance Industries paddle mixer by heating to 99-104°C and removing from the unit. Material exiting the mixer was soybean meal.

Extracted flakes were desolventized with ambient air. Following desolventization, extracted flakes were ground in an Alpine pin mill to produce defatted soy flour.

Miscella was passed through a laboratory vacuum evaporator unit to separate the crude oil and hexane. Crude oil was heated to 91-96°C for hexane removal and filtered.

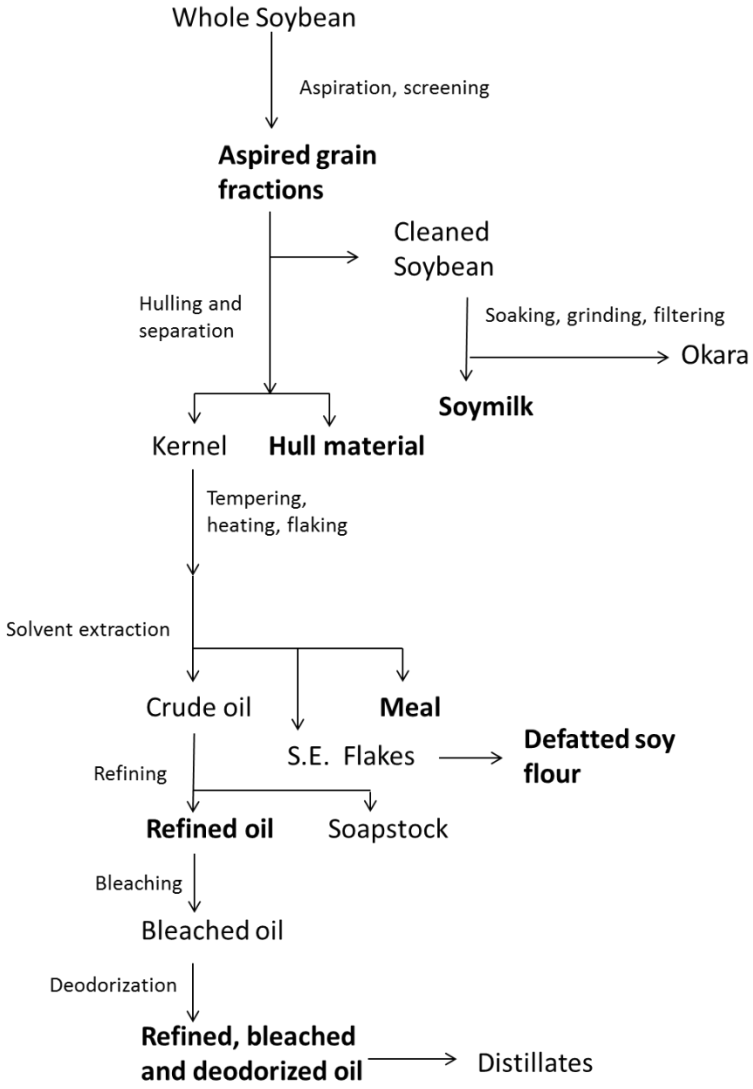
For alkali refining, the percent free fatty acid (FFA) was determined for the crude oil. Based on the FFA, a weighed amount of crude oil and 14° Baume sodium hydroxide was placed in a water bath at 20-24°C and mixed for 90 minutes at high RPM, and then for 20 minutes at low RPM and 63-67°C. Neutralized oil was then centrifuged. Refined oil was decanted and filtered. This step produced alkali refined oil and soapstock. Soapstock was discarded.

For bleaching, alkali refined oil was heated to 40-50°C, activated bleaching earth added (1.0% by weight of oil), and placed under vacuum. Temperature was increased to 85-100°C and held for 10 to 15 minutes. After reducing the temperature, the bleached oil was filtered. A filter aid was used for samples from trial RV268-10PA. Resulting fractions were bleached oil and spent bleaching earth.

For deodorization, bleached oil was steam bathed for 28-32 minutes under vacuum and temperature held between 220-230°C. During the cooling period a 0.5% citric acid solution was added (1 mL per 100 grams of oil deodorized). Resulting fractions were refined-bleached-deodorized oil (RBD oil) and deodorizer distillates.

<u>Soymilk production:</u>
A portion of the cleaned soybean sample was removed for soymilk production. Soybeans for soymilk production were washed and soaked in water for at least 12 hours. Soaked beans were ground and filtered to remove solids and the soymilk was cooked at 91-96 for 9-11 minutes.

Figure A 1: Processing flowchart for soybean



Results and discussions

Concurrent recoveries of flupyradifurone and DFA were measured with each set of samples to verify method performance. All recoveries were corrected for any interferences in corresponding controls. The mean recoveries per fortification level were within the acceptable range of 72 – 111%, and the relative standard deviation values were below 20%.

Table A 24: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soybean seeds and processed commodities

Study Trial No. GLP Year	Portion analyzed	a.s./metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
RARVY029 (RV268-10PA and RV269-10PA) GLP: yes 2010	Soybean seed, dry	BYI 02960	7	0.010	97; 97; 92; 102; 90; 102; 96	90	102	97	4.7
			3	1.0	95; 87; 96	87	96	93	5.3
			10	overall		87	102	95	5.0
		DFA	7	0.050	75; 74; 91; 75; 81; 76; 77	74	91	78	7.6
			3	1.0	77; 79; 87	77	87	81	6.5
			10	overall		74	91	79	7.1
	Soybean aspirated grain fractions	BYI 02960	3	0.25	79; 93; 87	79	93	86	8.1
			3	15	98; 91; 86	86	98	92	6.6
			6	overall		79	98	89	7.4
		DFA	3	0.050	98; 100; 104	98	104	101	3.0
			3	0.25	85; 91; 95	85	95	90	5.6
			3	15	86; 84; 86	84	86	85	1.4
			9	overall		84	104	92	8.0
	Soybean meal	BYI 02960	3	0.010	83; 111; 78	78	111	91	19.6
			3	1.0	85; 93; 104	85	104	94	10.1
			6	overall		78	111	92	14.0
		DFA	3	0.050	75; 71; 74	71	75	73	2.8
			3	1.0	71; 70; 92	70	92	78	16.0
			6	overall		70	92	76	11.0
	Soybean hull	BYI 02960	3	0.010	103; 76; 101	76	103	93	16.1
			3	1.0	91; 90; 87	87	91	89	2.3
			6	overall		76	103	91	10.8
		DFA	3	0.050	84; 84; 83	83	84	84	0.7
			3	1.0	81; 76; 78	76	81	78	3.2
			6	overall		76	84	81	4.1
	Soybean oil, refined	BYI 02960	3	0.010	97; 99; 80	80	99	92	11.3
			3	1.0	104; 109; 92	92	109	102	8.6
			6	overall		80	109	97	10.4
		DFA	3	0.050	101; 109; 101	101	109	104	4.5
			3	1.00	108; 114; 97	97	114	106	8.1
			6	overall		97	114	105	6.1
	Soybean milk	BYI 02960	3	0.010	100; 99; 95	95	100	98	2.7
			3	1.00	99; 97; 98	97	99	98	1.0
			6	overall		95	100	98	1.8
		DFA	3	0.050	96; 98; 92	92	98	95	3.2
			3	1.0	107; 97; 102	97	107	102	4.9
			6	overall		92	107	99	5.3
	Soybean defatted flour	BYI 02960	3	0.010	116; 104; 114	104	116	111	5.8
			3	1.0	100; 87; 91	87	100	93	7.2
			6	overall		87	116	102	11.6
		DFA	3	0.050	88; 78; 81	78	88	82	6.2
			3	1.0	76; 72; 69	69	76	72	4.9
			6	overall		69	88	77	8.7

The residue of flupyradifurone and its metabolite DFA were determined in the harvested soybean seeds (raw agricultural commodity) at day 21 ± 2 and in the processed products. These values were used for the calculation of "processing" factors (cf. Table A 25).

Table A 25: Residue data from soybean processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)						
Soybean seed, dry	0.695	21	aspirated grain fractions	11.0	16	mean PF: 14
			meal	0.593	0.85	mean PF: 1.1
			hull	0.544	0.78	mean PF: 1.0
			oil, refined	<0.010	<0.014	mean PF: <0.019
			milk	0.029	0.042	mean PF: 0.066
			defatted flour	0.728	1.0	mean PF: 1.1
Soybean seed, dry	0.414	19	aspirated grain fractions	4.84	12	mean PF: 14
			meal	0.519	1.3	mean PF: 1.1
			hull	0.485	1.2	mean PF: 1.0
			oil, refined	<0.010	<0.024	mean PF: <0.019
			milk	0.037	0.089	mean PF: 0.066
			defatted flour	0.494	1.2	mean PF: 1.1

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
DFA, expressed as flupyradifurone (RD-Mo)						
Soybean seed, dry	0.052	21	aspirated grain fractions	0.428	8.2	mean PF: 6.5
			meal	0.062	1.2	mean PF: 1.3
			hull	0.054	1.0	mean PF: 0.9
			oil, refined	<0.050	<1.0	mean PF: <0.6
			milk	<0.050	<1.0	mean PF: <0.6
			defatted flour	0.076	1.5	mean PF: 1.4
Soybean seed, dry	0.306	19	aspirated grain fractions	1.47	4.8	mean PF: 6.5
			meal	0.442	1.4	mean PF: 1.3
			hull	0.240	0.78	mean PF: 0.9
			oil, refined	<0.050	<0.16	mean PF: <0.6
			milk	<0.050	<0.16	mean PF: <0.6
			defatted flour	0.390	1.3	mean PF: 1.4
sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)						
Soybean seed, dry	0.747	21	aspirated grain fractions	11.4	15	mean PF: 12
			meal	0.655	0.88	mean PF: 1.1
			hull	0.594	0.80	mean PF: 0.9
			oil, refined	<0.060	<0.08	mean PF: <0.1
			milk	0.079	0.11	mean PF: 0.1
			defatted flour	0.804	1.1	mean PF: 1.2
Soybean seed, dry	0.720	19	aspirated grain fractions	6.3	8.8	mean PF: 12
			meal	0.96	1.3	mean PF: 1.1
			hull	0.73	1.0	mean PF: 0.9
			oil, refined	<0.06	<0.08	mean PF: <0.1
			milk	0.087	0.12	mean PF: 0.1
			defatted flour	0.884	1.2	mean PF: 1.2

* processing factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Following an exaggerated application rate of flupyradifurone to two separate trials of soybeans, mature seeds were collected. Aspirated grain fractions were then generated and the remaining soybean seeds were processed into meal, hulls, refined oil, soymilk, and defatted flour. The average flupyradifurone residue did not concentrate (processing factor <1X) in soybean milk and oil. Residues of parent remained at approx. the same level in meal, hulls and defatted flour, whereas the flupyradifurone residue did clearly concentrate in aspirated grain fractions (14X). A similar picture was observed for the metabolite DFA: The average DFA residue did not concentrate (mean processing factor <1X) in soybean milk and oil. Residues of DFA remained also at approx. the same level in hulls. A slight concentration of DFA residues was indicated in meal and defatted flour (mean processing factors of 1.3 and 1.4, respectively), whereas the DFA residue again concentrated in aspirated grain fractions (6.5X).

A 2.1.5.2.2 Cotton , study report RARVY033

Comments of zRMS:	The study was conducted in accordance with OECD Test Guideline 508: Residues in Processed Commodities. Following a five-fold (5X) exaggerated application of BYI 02960 to two separate trials of cotton plants, BYI 02960 residues did not concentrate in any of the processed products (meal, hulls, refined oil (bleached and deodorized), solvent extracted crude oil, pre-clarified crude oil, and neutralized crude oil). DFA residues were only detected in one meal sample; all other commodities showed DFA residues below the LOQ (0.05 mg/kg), so concentration of DFA residues in meal cannot be excluded. Taking into account the total residue of BYI 02960 (sum of BYI 02960 and DFA), no concentration of the residue can be determined in any of the processed commodities, the processing factors were always <1. The residue data and subsequent calculated processing factors provided in this report are suitable for regulatory purposes. The study is acceptable.

Reference:	**KCA 6.5.3/02**
Title:	BYI 02960 200 SL - Magnitude of the residue in cotton processed commodities (crop subgroup 20C)
Report:	Lenz, C.; Beedle, E.; 2012; RARVY033; M-433122-01-1
Authority registration No:	
Guideline(s):	EPA Ref.: OPPTS 860.1520, Processed Food/Feed PMRA Ref.: DACO 7.4.5. Processed Food/Feed OECD Test Guideline 508: Residues in Processed Commodities (Adopted Oct., 3 2008)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

Two field trials were conducted in the USA to measure the magnitude of BYI 02960 residues in/on cotton processed commodities following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. The additive, Dyne-Amic 0.25% v/v was used in all applications. BYI 02960 SL 200 was sprayed twice at an exaggerated application rate ranging from 596 to 1000 g a.s./ha. Seasonal application rates ranged from 1980 to 2000 g a.s./ha. The first applications were made between BBCH growth stages 87 to 88.The interval between applications was 9 to 10 days. The spray volumes for all treated plots ranged from 10 to 20 GPA (93 to 187 L/ha).

Single composite seed cotton samples were harvested at a 13 to 14-day pre-harvest interval (PHI). Single composite samples of seed cotton samples were harvested from the control plots on the same day the respective samples were collected from the treated plots.

After ginning, triplicate subsamples of undelinted seed, the raw agricultural commodity (RAC), were removed, and the remaining undelinted seed was used to generate the processed commodities of meal, hulls, refined oil (bleached and deodorized (RBD) oil), solvent extracted crude oil, pre-clarified crude oil, and neutralized crude oil.

The maximum storage period of deep-frozen samples intended for the analysis BYI 02960 and its metabolites was 449 days, which is within the acceptable rage for the storage stability of the residues of flupyradifurone in high oil content commodities.

The residues of BYI 02960 and DFA were analyzed using method RV-001-P10-02 (Ying Li; 2010; M-401023-01-2) with some modifactions, also known as method 01304. The individual residues of parent BYI 02960 and DFA were determined and a total BYI 02960 residue was calculated (sum of BYI 02960

and DFA). The limits of quantitation were 0.01 mg/kg for BYI 02960 and 0.05 mg/kg for DFA, expressed in parent compound equivalents, yielding a calculated total-residue LOQ of 0.06 mg/kg, in all matrices. Residue measurements below the analyte LOQ were summed into the total BYI 02960 residue value as the analyte LOQ value. Prior and parallel to the residue analysis, the method was validated by recovery experiments.

Processing procedures:
Drying, ginning and delintering: Lint moisture of the cotton seed sample was determined with a cotton moisture meter. If the moisture was greater than 8.0% the sample was dried in a cotton dryer system or tray oven at 54-71°C until the moisture was less than 8.0%. Cotton seed was cleaned with a stick extractor to remove the gin byproducts (gin trash) and ginned to separate ginned cotton seed (undelinted cotton seed) and lint in a saw gin. Resulting products were ginned cotton seed, lint and gin trash. Ginned seed fractions were collected amd placed at frozen storage. With approximately 11-15% remaining lint, the ginned cotton seed samples were saw delinted in a delinter to remove most remaining lint and produce delinted cotton seed with approximately 3% lint remaining on the seed. Delinting produced delintered cotton seed, linters and linter motes.

Hulling and separation: Delinted cotton seed was mechanically cracked in a roller mill. Kernel and hull material was separated with a screen cleaner equipped with 4.8 mm screen. Hull material fractions were collected and placed in frozen storage. Kernel material moisture was determined and adjusted to 13.5%.

Oil and meal production: Kernel material was heated in a steam heated mixer to 79-91°C and held for 28-32 minutes. After heating, kernel material was flaked in a flaking roll with a gap setting of 0.254-3.30 mm. Flaked kernel material was fed into a continuous processor (extruder). As the material moved through the extruder, steam was injected directly on the product. Maximum exiting temperature range of the material (collets) was 93-121°C. Collets were dried in an oven at 66-82°C for 30-40 minutes and then taken to solvent extraction. Collets were placed in stainless steel batch extractors and submerged in 49-60°C solvent (hexane). After 30 minutes, the miscella (hexane and crude oil) was drained and fresh hexane added to repeat the cycle two more times, Final two washings were for 15 mimnutes each. After the final draining, the spent collets (meal) were desolvnetized by heating to 99-104°C in a steam heated paddle mixer to remove residual hexane. Resulting fraction was cotton seed meal which was collected and stored frozen. Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was heated to 195-205°C for hexane removal during the separation process. Crude oil was filtered. Fractions of crude oil were collected and stored frozen.

Degumming an de-sliming: A weighted amount of crude oil was heated to 70-76°C and reverse osmosis water was added in the amount of 2.5% of weight of crude oil. The mixture was stirred for 60 minutes in this temperature range and filtered to remove the gums produced. Resulting products were degummed oil and gums.
For de-sliming, the degummed oil was treated with 85% phosphoric acid. Amount f phosphoric acid added was equal to 0.03% of the total weight of the degummed oil. Mixture was stirred for 29-31 minutes at 40-44°C then filtered/decanted to remove the slime produced. Resulting products were pre-clraified oil and slime. Fractions of pre-clarified oil were collected and stored frozen.

Alkali refining, bleaching and deodorization:
Pre-clarified oil and 16° Baume sodium hydroxide was placed in a water bath and mixed for 15 minutes at high RPM at 20-24°C followed by 12 minutes at low RPM at 63-67°C. Neutralized oil was centrifuged to separate the refined oil and soapstock. Refined oil was decanted from the soapstock and filtered. Refined oil fractions were collected and placed oin frozen storage. Soapstock was discarded.
Refined oil was heated to 40-50°C, an activated bleching earth added (1.0% by weight of oil), and placed under vacuum. Temperature was increased to 85-100°C and held for 10-15 minutes. After reducing the temperature, the bleached oil was filtered.
Bleached oil was steam bathed for 28-32 minutes under vacuum and termperture held between 220-230°C. During the cooling period a 0.5% citric acid solution was added (1mL/100g of oil deodorized). Resulting fractions were deodorized oil (RBD oil) and deodorizer distillates. Deodorized oil was collected and placed in frozen storage.

Results

Concurrent recoveries of BYI 02960 and DFA were measured with each set of samples to verify method performance. All recoveries were corrected for any interferences in corresponding controls. The overall mean of the recoveries for each matrix at each fortification level was within the acceptable range of 70 to 110%, and the standard deviation (SD) values were less than 20%.

The residue values and processing factors of BYI 02960 and DFA, as well as calculated total BYI 02960 residue data (sum of parent compound BYI 02960 and DFA, expressed as BYI 02960 equivalents) for the cotton seed RAC and the processed commodities are provided in Table A 27.

Table A 26: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in cotton seeds and processed fractions

Study Trial No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
RARVY033 RV276-10PA and RV277-10PA GLP: yes 2010	Cotton	seed, undelinted	BYI 02960	3	0.010	117; 97; 101	97	117	105	10.1
				3	3.0	100; 98; 99	98	100	99	1.0
				6	overall		97	117	102	7.3
			DFA	3	0.050	68; 93; 77	68	93	79	16.0
				3	3.0	88; 92; 91	88	92	90	2.3
				6	overall		68	93	85	11.9
		crude oil, neutralized	BYI 02960	4	0.010	121; 90; 103; 83	83	121	99	16.8
				4	overall		83	121	99	16.8
			DFA	4	0.050	74; 84; 103; 101	74	103	91	15.4
				4	overall		74	103	91	15.4
		meal	BYI 02960	3	0.010	103; 97; 115	97	115	105	8.7
				3	1.0	102; 108; 106	102	108	105	2.9
				6	overall		97	115	105	5.8
			DFA	3	0.050	73; 79; 79	73	79	77	4.5
				3	1.0	83; 83; 83	83	83	83	0.0
				6	overall		73	83	80	4.9
		hull	BYI 02960	3	0.010	97; 87; 93	87	97	92	5.5
				3	1.0	102; 105; 89	89	105	99	8.6
				6	overall		87	105	96	7.5
			DFA	3	0.050	75; 72; 69	69	75	72	4.2
				3	1.0	87; 90; 92	87	92	90	2.8
				6	overall		69	92	81	12.4

Remark re. cotton crude oil, neutralized: also covers crude oil, pre-clarified; oil, refined and oil, solvent extracted

Table A 27: Residue data from cotton seed processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)									
Cotton seed	1.42	0.25	13/14	Oil, refined	<0.01	<0.01	<0.1	<0.1	Median: <0.1
				Oil, solvent extracted	<0.01	<0.01	<0.1	<0.1	Median: <0.1
				Crude oil, pre-clarified	<0.01	<0.01	<0.1	<0.1	Median: <0.1
				Crude oil, neutralized	<0.01	<0.01	<0.1	<0.1	Median: <0.1
				Meal	**0.05**	**0.06**	**<0.1**	**0.3**	Median: **<0.3**
				Hull	0.14	0.23	<0.1	0.9	Median: <0.5
DFA, expressed as DFA (RD-Mo)									
Cotton seed	<0.05	<0.05	13/14	Oil, refined	<0.05	<0.05	n.c.	n.c.	n.c.
				Oil, solvent extracted	<0.05	<0.05	n.c.	n.c.	n.c.
				Crude oil, pre-clarified	<0.05	<0.05	n.c.	n.c.	n.c.
				Crude oil, neutralized	<0.05	<0.05	n.c.	n.c.	n.c.
				Meal	**<0.05**	**0.065**	**n.c.**	**>1.3**	**Median: >1.3**
				Hull	<0.05	<0.05	n.c.	n.c.	n.c.
Sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)									
Cotton seed	1.47	0.30	13/14	Oil, refined	<0.06	<0.06	<0.04	<0.20	<0.12
				Oil, solvent extracted	<0.06	<0.06	<0.04	<0.20	<0.12
				Crude oil, pre-clarified	<0.06	<0.06	<0.04	<0.20	<0.12
				Crude oil, neutralized	<0.06	<0.06	<0.04	<0.20	<0.12
				Meal	**0.10**	**0.13**	**0.07**	**0.43**	**0.25**
				Hull	0.19	0.28	0.13	0.93	0.53

* PF = Processing Factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

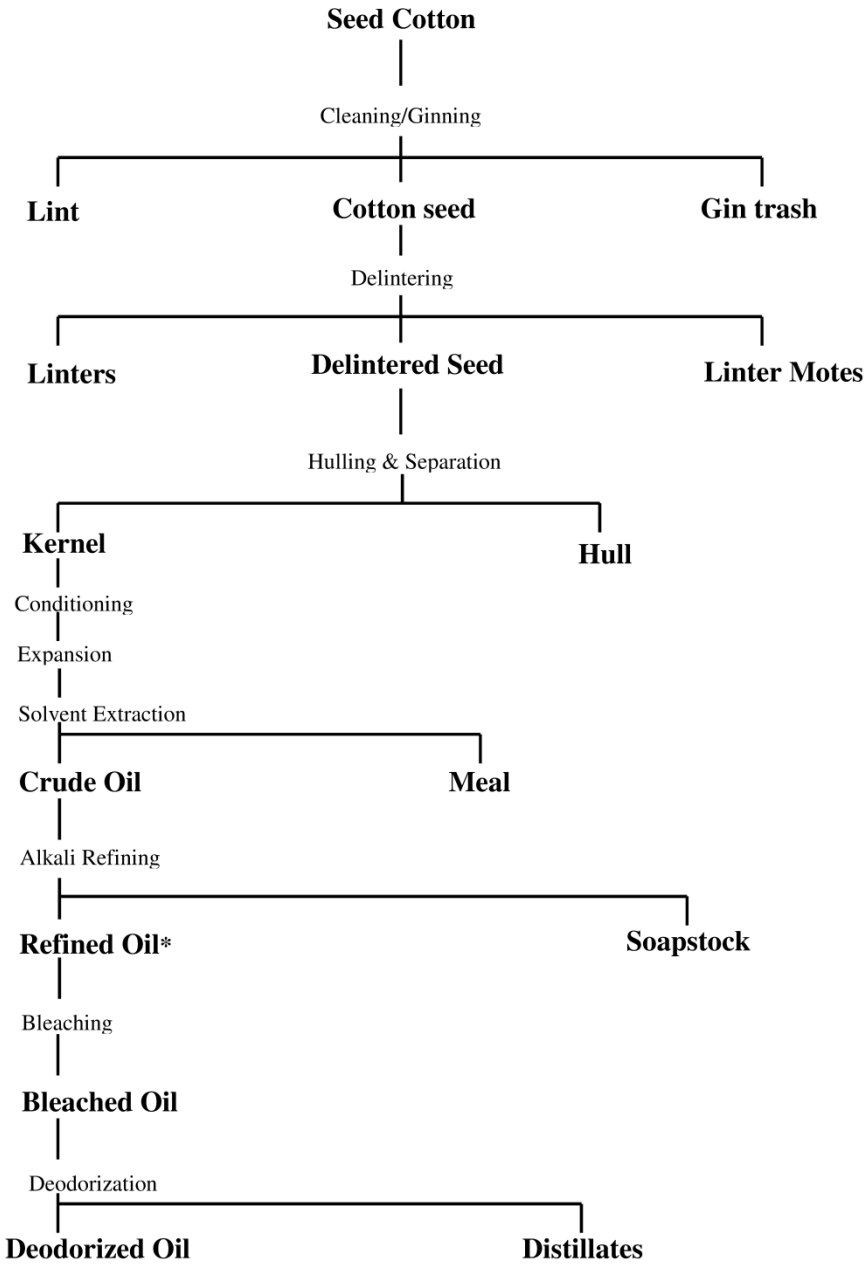
Conclusion

Following an exaggerated application of BYI 02960 SL 200 to two separate trials of cotton, BYI 02960 related residues were detected in cotton seeds as raw agricultural commodity (RAC), and in processed commodities. Processing factors were determined separately for parent BYI 02960, DFA and the total residue (sum of BYI 02960 and DFA).

BYI 02960 residues did not concentrate in meal, hulls, refined oil (bleached and deodorized), solvent extracted crude oil, pre-clarified crude oil, and neutralized crude oil. DFA residues were only detected in one meal sample; all other commodities showed DFA residues below the LOQ (0.05 mg/kg). Thus concentration of DFA residues in meal cannot be excluded.

Considering the total residue of BYI 02960 (sum of BYI 02960 and DFA), no concentration of the residue can be determined in any of the processed commodities, the processing factors were always below 1.

Figure A 2: Processing flowchart of cotton seed into meal and cotton oil



*Per protocol request crude oil was degummed and pre-clarified/de-slime prior to alkali refining

A 2.1.5.2.3 Potato, study report RARVY038

Comments of zRMS:	Two field trials were conducted in US to measure the magnitude of BYI 02960 residues in/on potato processed commodities (tuber, washed; wet peel; tuber, peeled; tuber, peeled, steamed and mashed; tuber with peel, cooked; starch; flakes; and crisps after two foliar applications at rate of 1000 g a.s./ha. A slight concentration of BYI 02960 residues was indicated in potato flakes and peel. Washing did not reduce the residues, neither for BYI 02960 not for DFA. Overall, the total BYI 02960 residue (sum of BYI 02960 and DFA) did not concentrate (processing factor ≤1X) significantly in any of the processed commodities. The highest transfer of the total residue was detected for potato flakes. The study is acceptable.

Reference:	**KCA 6.5.3/03**
Title:	BYI 02960 200 SL - Magnitude of the residue in/on potato processed commodities
Report:	Lenz, C.; 2012; RARVY038; M-430542-01-2
Authority registration No:	
Guideline(s):	EPA Ref.: OPPTS 860.1520, Processed Food/Feed PMRA Ref.: DACO 7.4.5. Processed Food/Feed OECD Test Guideline 508: Residues in Processed Commodities (Adopted Oct., 3 2008)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	
Duplication (if vertebrate study):	

Materials and methods

Two field trials were conducted to measure the magnitude of flupyradifurone residues in/on potato processed commodities following two broadcast foliar spray applications of Flupyradifurone SL 200 at exaggerated application rates. Flupyradifurone SL 200 is a soluble concentrate formulation containing 200 g a.s./L.

Flupyradifurone SL 200 was applied twice to potato plants at individual application rates of approx. 1 kg/ha. The last application was scheduled to be 7 days before harvest. All applications were made using ground-based equipment. The additive, Dyne-Amic 0.25% v/v, was used in all applications.

Single composite potato tuber samples were dug at a 7 days after the last treatment from the treated plots. Single composite samples of potato tubers were dug from the control plots on the same day the respective samples were dug from the treated plots.

The residues of flupyradifurone and DFA were quantitated by LC-MS/MS using stable isotopically labelled internal standards (method 01304, Li, Y.; Schoening, R.; 2012; M-415504-02-1). The limits of quantitation were 0.01 mg/kg for flupyradifurone and 0.05 mg/kg for DFA, when expressed in parent compound equivalents. The individual analyte residues (parent flupyradifurone and DFA, expressed in parent equivalents) were summed to give a total flupyradifurone residue. Residue measurements below the analyte LOQ were summed into the total flupyradifurone residue value as the analyte LOQ value. Prior and parallel to the residue analysis, the method was validated by recovery experiments.

Samples of potato processed fractions were were held in frozen storage for a maximum of 197 to 209 days prior to extraction (12 months). The storage time is within the acceptable range for storage stability of the residues of flupyradifurone in matrices with high starch content.

Triplicate subsamples of unwashed potato tubers, the raw agricultural commodity (RAC), were removed. The remaining potato tubers were washed, (triplicate subsamples removed), washings collected (wash water) and the potato processed commodities of crisps, flakes, wet peel, starch, tuber with peel cooked, peeled tuber, tuber cooked, (steamed, peeled, and mashed), and cooking (boiling) water generated.

Processing of potato flakes and starch:
Potatoes were washed followed by pealing using a steam peeler. The potatoes were scrubbed using a restaurant style peeler fitted with a rubber scrubber. The peel was then hydraulically pressed to increase the solids content. Water from the scrubber was collected for starch processing.
Peeled potatoes were individually inspected for any rot, green or otherwise seriously damaged potato tissue followed by cutting into slabs of 1.0-1.5 cm and washing off the free starch. Potato slices were precooked at ~70-77°C for 20-22 min, cooled down to less than 32°C under running cold water. The cooled slices are steam cooked at 94°-100°C for 40-42 minutes using an atmospherically flowing steam batch style steam cooker. the cooked slices are mashed using a restaurant style meat grinder without the grinding attachment. The mashed potatoes are placed in a bakery style mixer where an emulsion containing the additives is poured into the mashed potatoes and mixed for ~37 seconds. The potato mash is hand fed onto a laboratory single drum dryer where the potato mash is dried into a thin sheet. The product is initially broken by hand to reduce the sheet to large flakes. The large flakes are then hand fed into a hammermill for uniform sizing of the finished flakes. If moisture content of the potatoflakes exceeds 9%, the flakes are dried on the fluidized bed dryer to less than or equal to 9% moisture.

Starch water from potato scrubbing and wet peel pressing was filtered using a series of sieves to remove pieces of peel and pulp. The filtered starch water was centrifuged to separate the water from the starch until required amount of starch sample was obtained.

Processing of potato chips:
Washed potatoes were peeled using a restaurant style peeler for 30 seconds. The collected peel was weighed and discarded. The peeled potatoes were inspected by hand and trimmed if necessary, to remove rot, green or otherwise damaged potato tissue. Any trim waste collected was weighed and discarded. An aliquot of the remaining peeled potatoes were cut into thin ~0.16 cm slices using a restaurant style slicer. Any abrasion peeled potatoes in excess were discarded. The sliced potatoes were placed in a tub of hot water to remove free starch. The slices were drained over a screen to remove excess water and were fried at ~163 -191°C frying oil for ~ 90 seconds. The fried potato chips were drained, salted and inspected. Undesirable chips were removed. A sample of the drained, salted and inspected potato chips were removed, packaged, labeled and placed in the freezer for the required sample fraction.

Processing of cooked potatoes:
For producing unpeeled boiled potatoes, quartered potatoes were placed into boiling water and boiled until an internal temperature of 88-92°C was reached. After the desired temperature was reached, the unpeeled boiled potato fraction was removed from the water and packaged, labeled and placed into frozen storage.

Figure A 3: Processing flowchart for potato –potatoes chips

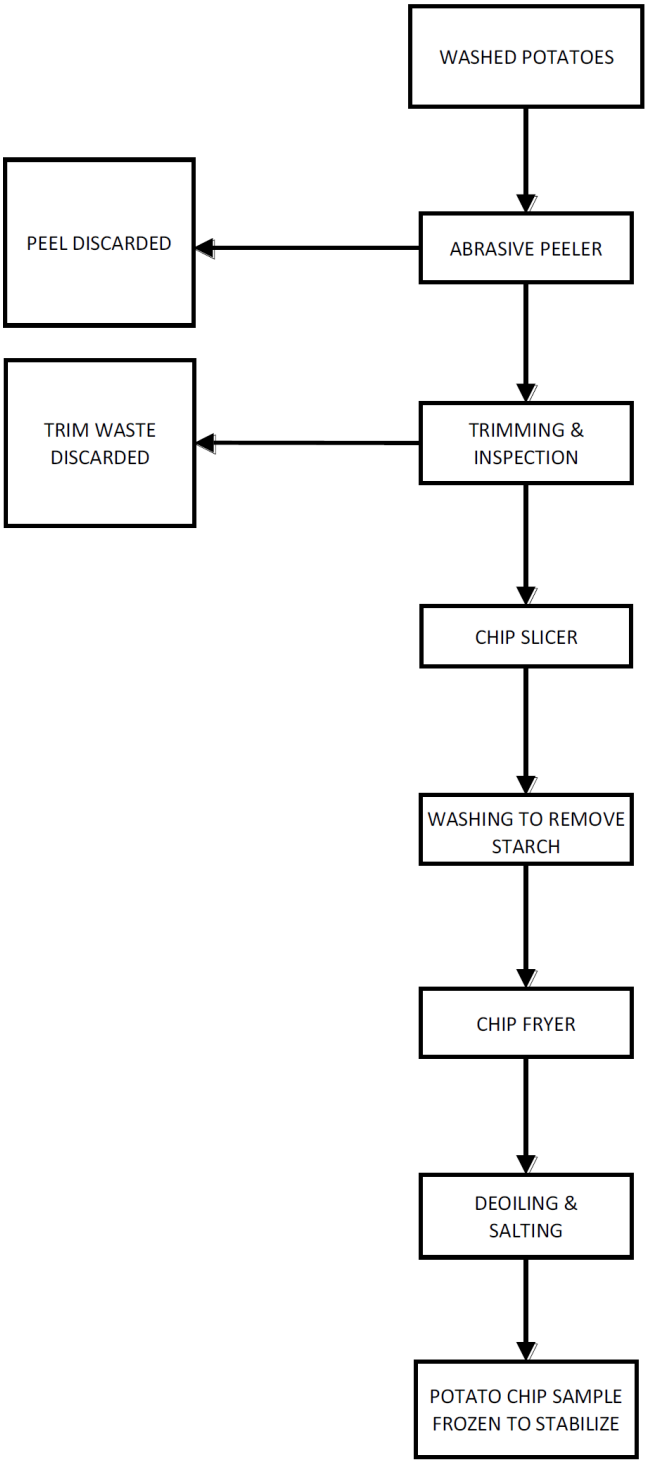


Figure A 4a: Processing flowchart for potato – flakes and starch part 1

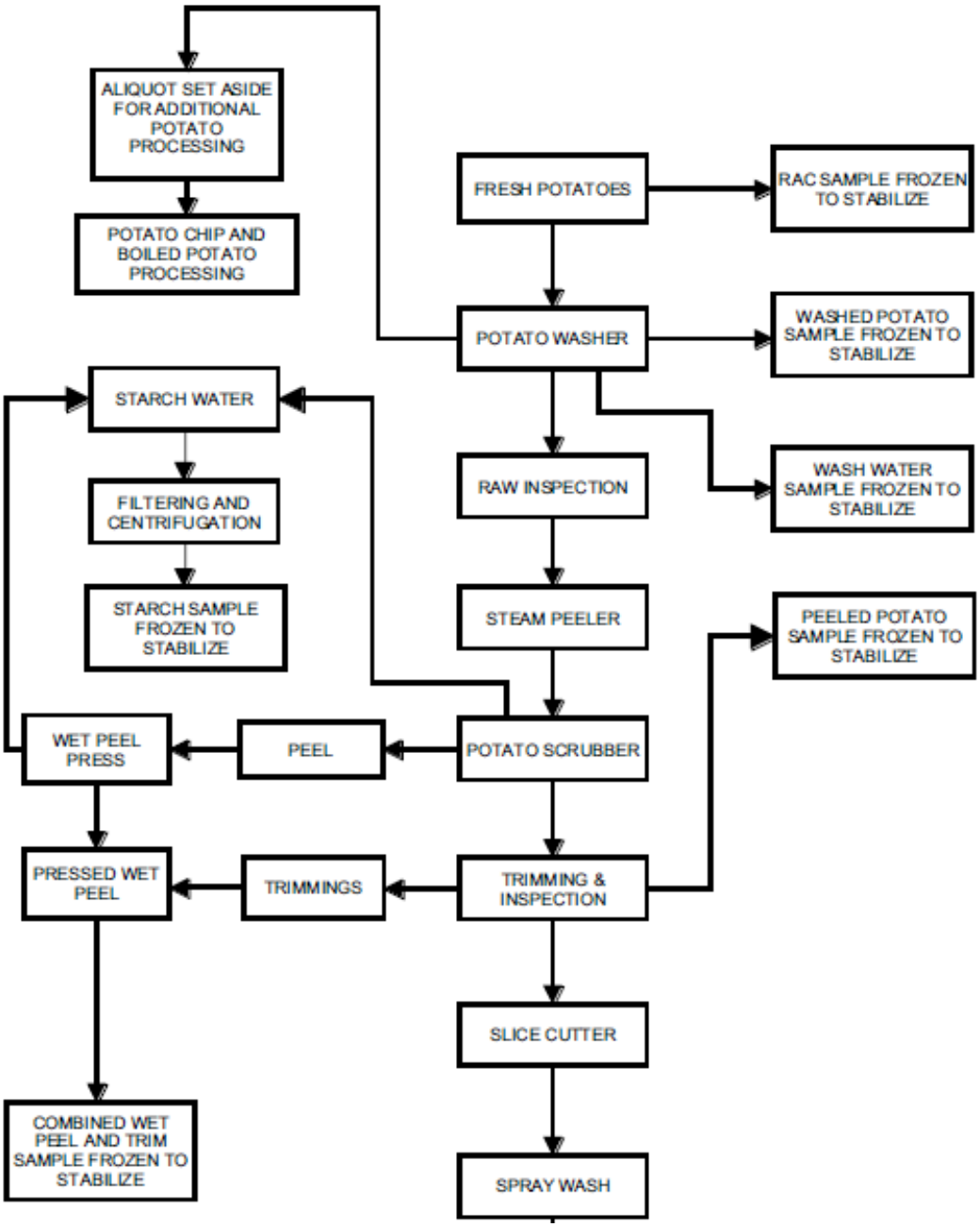


Figure A 2b: Processing flowchart for potato – flakes part 2 (following spray wash)

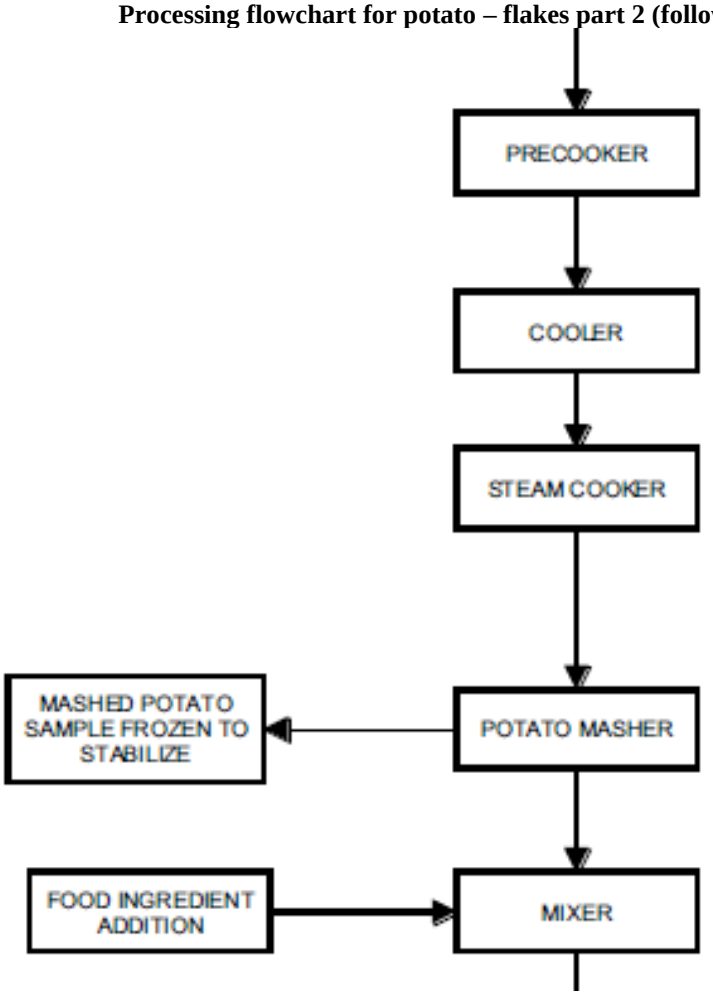


Figure A 2c: Processing flowchart for potato – flakes part 2 (following mixer)

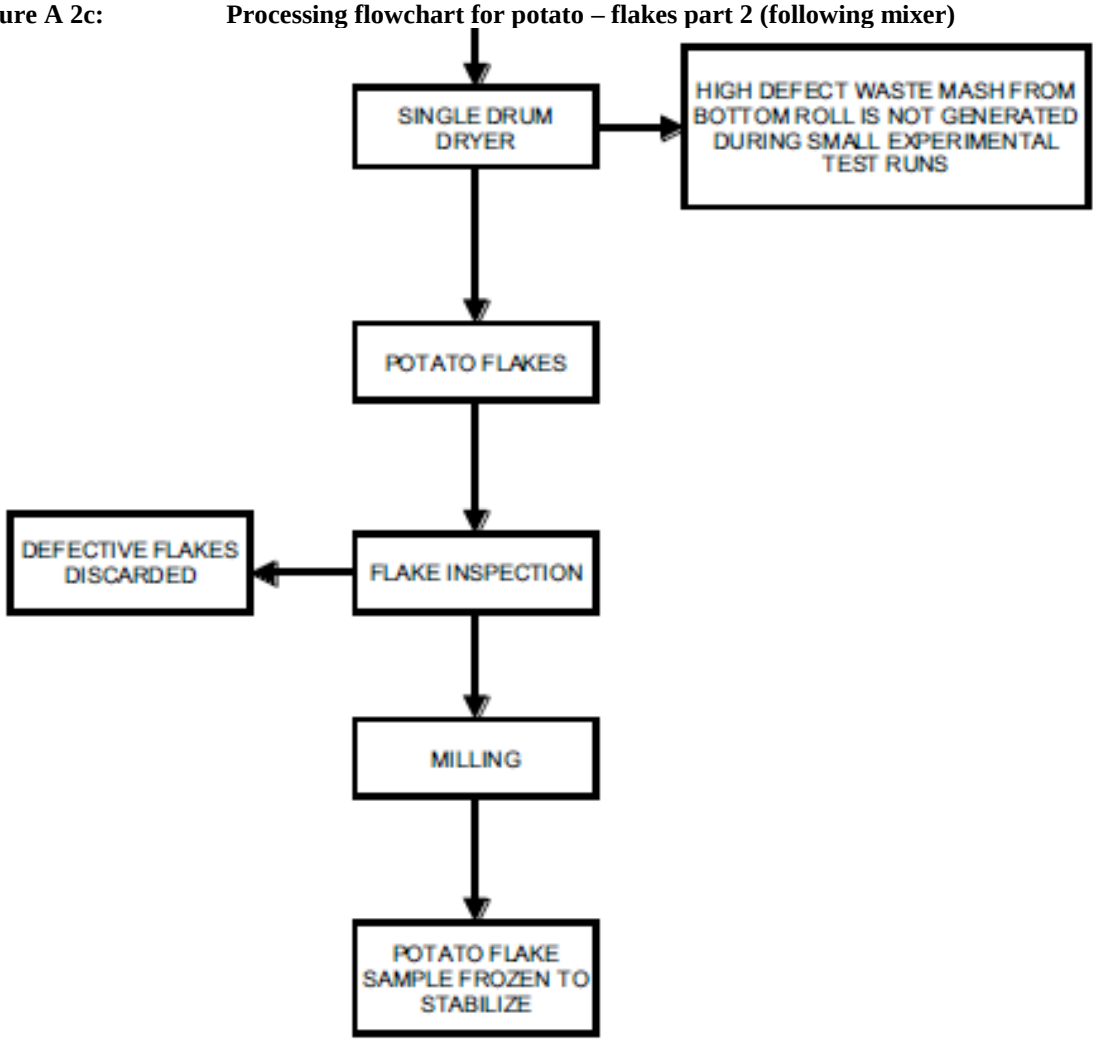
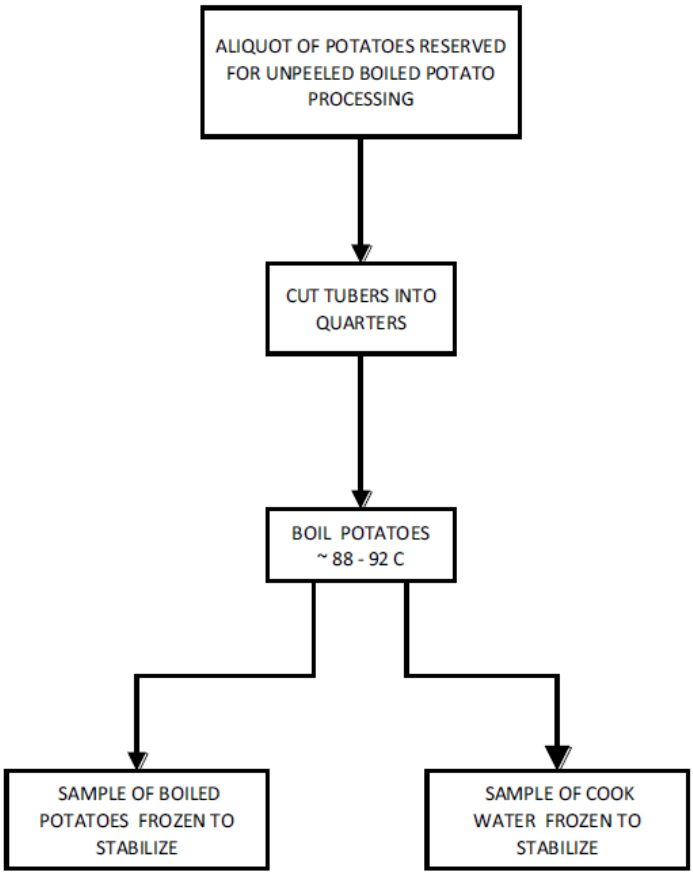


Figure A 5: Processing flowchart for potato – cooked potatoes



Results and discussions

Concurrent recoveries of flupyradifurone and DFA were measured with each set of samples to verify method performance. All recoveries were corrected for any interferences in corresponding controls. The mean recoveries per fortification level were within the acceptable range of 70 – 110%, and the relative standard deviation values were below 20% (Table A 28).

Table A 28: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in potato tuber and processed commodities

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
RARVY038 RV202-11PA and (RV203-11PA) GLP: yes 2011	potato	tuber	BYI 02960	7	0.010	98; 90; 88; 79; 76; 77; 115	76	115	89	15.7
				7	overall		76	115	89	15.7
			DFA	7	0.50	106; 106; 98; 115; 93; 91; 108	91	115	102	8.5
				7	overall		91	115	102	8.5
		tuber, washed	BYI 02960	3	0.10	87; 83; 83	83	87	84	2.7
				3	overall		83	87	84	2.7
			DFA	3	0.10	89; 92; 111	89	111	97	12.3
				3	overall		89	111	97	12.3
		washings	BYI 02960	3	0.010	87; 93; 113	87	113	98	13.9
				3	0.10	92; 94; 79	79	94	88	9.2
				6	overall		79	113	93	12.1
			DFA	3	0.050	89; 90; 98	89	98	92	5.3
				3	0.10	84; 84; 84	84	84	84	0.0
				6	overall		84	98	88	6.3
		crisps (Am.: chips)	BYI 02960	3	0.010	117; 78; 75	75	117	90	26.0
				3	0.10	76; 81; 78	76	81	78	3.2
				6	overall		75	117	84	19.3
			DFA	3	0.050	78; 83; 79	78	83	80	3.3
				3	0.10	84; 74; 74	74	84	77	7.5
				6	overall		74	84	79	5.4
		flakes	BYI 02960	3	0.010	78; 81; 104	78	104	88	16.2
				3	0.50	94; 85; 110	85	110	96	13.1
				6	overall		78	110	92	14.1
			DFA	3	0.050	83; 99; 74	74	99	85	14.8
				3	0.50	79; 78; 77	77	79	78	1.3
				6	overall		74	99	82	11.0
		peel, wet	BYI 02960	3	0.010	89; 95; 84	84	95	89	6.2
				3	0.10	95; 92; 82	82	95	90	7.6
				6	overall		82	95	90	6.2
			DFA	3	0.050	90; 90; 91	90	91	90	0.6
				3	0.10	83; 86; 83	83	86	84	2.1
				6	overall		83	91	87	4.2

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
		starch	BYI 02960	3	0.010	90; 106; 78	78	106	91	15.4
				3	0.50	86; 100; 106	86	106	97	10.5
				6	overall		78	106	94	12.2
			DFA	3	0.050	107; 101; 94	94	107	101	6.5
				3	0.50	89; 96; 94	89	96	93	3.9
				6	overall		89	107	97	6.5
		tuber with peel, cooked	BYI 02960	3	0.010	81; 110; 74	74	110	88	21.6
				3	0.10	78; 81; 87	78	87	82	5.6
				6	overall		74	110	85	15.1
			DFA	3	0.050	86; 84; 76	76	86	82	6.5
				3	0.10	83; 74; 77	74	83	78	5.9
				6	overall		74	86	80	6.2
		tuber, peeled	BYI 02960	2	0.10	106; 90	90	106	98	-
				2	overall		90	106	98	-
			DFA	2	0.10	108; 104	104	108	106	-
				2	overall		104	108	106	-
		tuber, steamed, mashed	BYI 02960	3	0.010	88; 98; 85	85	98	90	7.5
				3	0.10	83; 82; 90	82	90	85	5.1
				6	overall		82	98	88	6.7
			DFA	3	0.050	88; 92; 90	88	92	90	2.2
				3	0.10	80; 82; 83	80	83	82	1.9
				6	overall		80	92	86	5.6
		cooking water	BYI 02960	3	0.010	87; 103; 81	81	103	90	12.6
				3	0.10	86; 92; 92	86	92	90	3.8
				6	overall		81	103	90	8.3
			DFA	3	0.050	104; 101; 101	101	104	102	1.7
				3	0.10	93; 98; 96	93	98	96	2.6
				6	overall		93	104	99	4.0

The residue of flupyradifurone and its metabolite DFA were determined in the harvested potato tubers (raw agricultural commodity) at day 7 ± 1 and in the processed products. These values were used for the calculation of "processing" factors (

Table A 29).

Table A 29: Residue data from potato processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)						
potato, tuber	<0.010	7	tuber, washed	0.010	≥1	artifact (variability of residue data)
			washings	<0.010	n.c.	
			crisps (Am.: chips)	<0.010	n.c.	
			flakes	<0.010	n.c.	
			peel, wet	0.011	≥1	artifact (variability of residue data)
			starch	<0.010	n.c.	
			tuber with peel, cooked	<0.010	n.c.	
			tuber, peeled	<0.010	n.c.	
			tuber, steamed, mashed	<0.010	n.c.	
			cooking water	<0.010	n.c.	
potato, tuber	<0.010	7	tuber, washed	0.014	≥1	artifact (variability of residue data)
			washings	<0.010	n.c.	
			crisps (Am.: chips)	<0.010	n.c.	
			flakes	0.011	≥1.1	
			peel, wet	<0.010	n.c.	
			starch	<0.010	n.c.	
			tuber with peel, cooked	<0.010	n.c.	
			tuber, peeled	<0.010	n.c.	
			tuber, steamed, mashed	<0.010	n.c.	
			cooking water	<0.010	n.c.	
DFA, expressed as DFA (RD-Mo)						
potato, tuber	<0.017	7	tuber, washed	<0.017	n.c.	
			washings	<0.017	n.c.	
			crisps (Am.: chips)	<0.017	n.c.	
			flakes	<0.017	n.c.	
			peel, wet	<0.017	n.c.	
			starch	<0.017	n.c.	
			tuber with peel, cooked	<0.017	n.c.	
potato, tuber	<0.017	7	tuber, peeled	<0.017	n.c.	
			tuber, steamed, mashed	<0.017	n.c.	
			cooking water	<0.017	n.c.	
			tuber, washed	<0.017	n.c.	
			washings	<0.017	n.c.	

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
			crisps (Am.: chips)	0.025	>1.5	
			flakes	0.033	>2.0	
			peel, wet	<0.017	n.c.	
			starch	<0.017	n.c.	
			tuber with peel, cooked	<0.017	n.c.	
			tuber, peeled	0.019	≥1.1	artifact (variability of residue data)
			tuber, steamed, mashed	<0.017	n.c.	
			cooking water	<0.017	n.c.	
sum of flupyradifurone and DFA, expressed as flupyradifurone						
potato, tuber	<0.060	7	tuber, washed	0.060	≥1.0	
			washings	<0.060	n.c.	
			crisps (Am.: chips)	<0.060	n.c.	
			flakes	<0.060	n.c.	
			peel, wet	0.061	≥1.0	
			starch	<0.060	n.c.	
			tuber with peel, cooked	<0.060	n.c.	
			tuber, peeled	<0.060	n.c.	
			tuber, steamed, mashed	<0.060	n.c.	
			cooking water	<0.060	n.c.	

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
potato, tuber	<0.060	7	tuber, washed	0.064	≥1	artifact (variability of residue data)
			washings	<0.060	n.c.	
			crisps (Am.: chips)	0.084	>1.4	
			flakes	0.11	>2.2	
			peel, wet	<0.060	n.c.	
			starch	<0.060	n.c.	
			tuber with peel, cooked	<0.060	n.c.	
			tuber, peeled	0.067	≥1.1	artifact (variability of residue data)
			tuber, steamed, mashed	<0.060	n.c.	
			cooking water	<0.060	n.c.	

* PF = processing factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Conclusion

Following an exaggerated application rate of flupyradifurone (2 x 1 kg a.s./ha compared to 1 x 0.1 kg a.s./ha) to two separate trials of potatoes, mature tubers were collected for subsequent processing. The fresh potatoes were processed into crisps, flakes and starch. In addition potatoes were cooked (cooked tuber with peel), peeled and steamed and subsequently mashed. Despite the exaggerated application rate (approx. 20X), no residues above the LOQ of flupyradifurone and its metabolite DFA were detected in the raw agricultural commodity. A concentration of flupyradifurone residues in the processed commodities was not obvious, whereas a concentration of DFA residues was indicated for flakes and crisps.

A 2.1.5.2.4 Potato, study report 13-3149

Comments of zRMS:	Two supervised residue trials were conducted in northern Europe (Germany and the Netherlands) after one application at rate of 100 g a.s./ha. Equivalent processed fractions (tuber, washed; peel washed; tuber, peeled; tuber, cooked; tuber with peel, cooked; fibre; pulp, dry; starch; waste; waste, dried; flakes; and crisps) were prepared and analysed for flupyradifurone and DFA. Processing factors could only be calculated for DFA as the parent residues were always <0.01 mg/kg in the raw agricultural commodity. A concentration of DFA residues due to processing was only seen in flakes and crisps of one study. The processing factor for DFA in flakes and crisps was 1.7 and 1.8 respectively. All other processing steps resulted in a dilution of DFA residues. The study is acceptable.

Reference:	**KCA 6.5.3/04**
Title:	Determination of the residues of BYI 02960 in/on potato and the processed fractions (tuber, washed; peel washed; tuber, peeled; tuber, cooked; tuber with peel, cooked; fibre; pulp, dry; starch; waste; waste, dried; flakes; and crisps (Am.: chips)) after spray application of BYI 02960 SL 200 in the field in Germany and the Netherlands
Report:	Freitag, T.; Hoffmeister, R.; 2016; 13-3149; M-546878-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC, EC Guidance working document 7029/VI/95 rev.5 (1997-07-22), OECD 508, Adopted 2008-10-03, OECD Guideline for the Testing of Chemicals, Magnitude of Pesticide Residues in Processed Commodities US EPA OCSPP Guideline No. 860.152
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study 13-3149 was to determine the magnitude of the relevant residues of flupyradifurone in/on potato and the processed fractions (tuber, washed; peel washed; tuber, peeled; tuber, cooked; tuber with peel, cooked; fibre; pulp, dry; starch; waste; waste, dried; flakes; and crisps (Am.: chips)).

The raw agricultural commodity (mature potatoes) were sampled during the conduct of the study13-2149, which included four supervised residue trials conducted in the field in Northern Europe (United Kingdom, Germany and The Netherlands) during the 2013 season. From two trials (Germany and The Netherlands) samples were taken for processing.

Flupyradifurone SL 200 was applied once to potato plants at an application rate of 0.1 kg/ha. Mature potato tuber samples (BBCH 49) to be processed were sampled 14 days after the application. The samples were stored at ambient temperature until the beginning of the processing procedures.

The samples were analyzed according to method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The LOQ was 0.01 mg/kg for flupyradifurone and 0.02 mg/kg for DFA when expressed as flupyradifurone (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of flupyradifurone and DFA, expressed as flupyradifurone) was 0.03 mg/kg. In parallel to the residue analysis, the method was validated by recovery experiments.

Samples of potato processed fractions were were held in frozen storage for a maximum of 699 to 701 days prior to extraction (~23 months). The storage time is within the acceptable range for storage stability of the residues of flupyradifurone in matrices with high starch content.

Potato tubers were processed according to household practice into tuber, cooked and tuber with peel, cooked. Industrial processing of potato tuber samples was done into crisps, flakes and starch.

Processing of potato into cooked tuber:
If necessary, unripe, damaged or rotten tubers were sorted out. The remaining fresh potatoes were weighed. The sorted tubers were manually washed for about 3 minutes with luke warm standing water in a vessel. The ratio of water to tuber was approx. 2:1. The water covered the tubers. After washing an aliquot of the laboratory sample "tuber, washed" was taken. The remaining washed tubers were peeled by hand using a peeler. Aliquots of "peel, washed" and "tuber, peeled" were sampled. The remaining peeled tubers were halved, rinsed for a short-term with cold water and then covered with water in a saucepan. Salt was added (7.5 g salt / kg potatoes). Tubers were cooked until they were done. Depending on the size of the tubers, the boiling time was approx. 25 min. "Tuber, cooked" was taken as laboratory sample and stored frozen until analysis.

Processing of potato into cooked tuber with peel:
If necessary, unripe, damaged or rotten tubers were sorted out. The remaining fresh potatoes were weighed. The sorted tubers were manually washed for about 3 min with luke warm standing water in a vessel. The ratio of water to tuber was approx. 2:1. The water covered the tubers. The washed tubers were completely covered with water in a saucepan. Salt was added (7.5 g salt / kg potato). Tubers were cooked until they were done. Depending on the size of the tubers, the boiling time was approx. 25 minutes. The laboratory sample "tuber, with peel, cooked" was taken and stored frozen until analysis.

Processing of potato into starch:
If necessary, unripe, damaged or rotten tubers were sorted out. The remaining fresh potatoes were weighed. The sorted tubers were manually washed for about 3 min with cold tap water in a vessel. The ratio of water to tuber was approx. 1:1. The water covered the tubers. The washed tubers were grated using a potato grater. The resulting pulp was sieved through 3 sieves with different sizes (500 µm, 250 µm, 125 µm) to separate into fibre and starch with tuber water for approx. 60 min. The tuber water containing the starch was collected. The obtained intermediate samples were starch with tuber water and fibre. An aliquot of "fibre" was taken as laboratory sample. Starch with tuber water was separated in a settling tank to obtain the products wet starch and tuber water. Subsequently the remaining fibre and starch were dried seperately at approx. 50°C until a dry mass content of approx. 90% was reached (drying time: 50 hours). The dried fibre "pulp, dry" and "starch" were taken as laboratory samples stored deep-frozen until analysis.

Processing of potato into flakes:
If necessary, unripe, damaged or rotten tubers were sorted out. The remaining fresh potatoes were weighed. The sorted tubers were manually washed for about 3 min with cold tap water in a vessel. The ratio of water to tuber was approx. 1:1. The water covered the tubers. The washed tubers were peeled using an industrial peeler for approx. 3 min. Peeling water with potato peel was separated by sieving. An aliquot of the obtained peel-sample "waste" was taken as laboratory sample. Subsequently the remaining sample of waste was dried at approx. 50°C until a dry mass content of approx. 90% was reached. "Waste, dried" was taken as laboratory sample. Further the peeled potatoes were quartered or halved, in depending on the potato size. Now three temperature treatments followed. First they were blanched in water (staying time approx. 75°C for about 5 min). Second, the blanched potatoes were cooled down (water temperature approx. 22°C) for 15 min. The third treatment was steaming in a special steamer at 100°C for approx. 90 min. This cooked intermediate was mashed. The cooled puree was placed on a roller dryer and dried for approx. 25 sec at approx. 140°C. The obtained "flakes" were taken as laboratory sample and stored frozen until analysis.

Processing of potato into crisps:
If necessary, unripe, damaged or rotten tubers were sorted out. The remaining fresh potatoes were weighed. The sorted tubers were manually washed for about 3 min with cold tap water in a vessel. The ratio of water to tuber was approx. 1:1. The water covered the tubers. The washed potatoes were peeled using an industrial peeler. Afterwards the tubers were sliced by means of a slicing machine in approx. 1

mm potato discs (special discs). These discs were washed with cold tap water in a vessel. The ratio of water to discs was approx. 1:1. The water covered the discs. Finally the washed slices were deep-fried in a fat fryer with pure vegetable oil at 175°C for approx. 6 min. “Crisps” were taken as laboratory sample and stored frozen until analysis.

Results and discussions

The individual and average recoveries for flupyradifurone were always within the acceptable range of 70 – 110% and the RSD values were below 20%. For DFA some recoveries at the LOQ level were slightly above 110% (washed peel: 117%, cooked tuber: 113%, crisps: 118%), however were considered to be acceptable. The DFA average recovery for flakes at the 10X LOQ level was rather low (66%), however since all residues detected were in the range of the LOQ, no additional recovery experiments were conducted.

The residue of flupyradifurone and its metabolite DFA were determined in the harvested potato tubers (raw agricultural commodity) at day 14 and in the processed products. These values were used for the calculation of "processing" factors (Table A 30).

Separate processing factors were determined for parent flupyradifurone, DFA and the total residue (sum of flupyradifurone and DFA).

Table A 30: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in potato tuber and processed commodities

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
13-3149 13-3149-01 13-3149-01-T and 13-3149-02 13-3149-02-T GLP: yes 2013	Potato	peel washed	BYI 02960	3	0.01	92; 93; 93	92	93	93	0.6
				3	0.10	91; 93; 96	91	96	93	2.7
				6	overall		91	96	93	1.8
			DFA	3	0.02	112; 119; 120	112	120	117	3.7
				3	0.20	99; 99; 101	99	101	100	1.2
				6	overall		99	120	108	9.2
		tuber, cooked	BYI 02960	3	0.01	92; 92; 93	92	93	92	0.6
				3	0.10	92; 93; 94	92	94	93	1.1
				6	overall		92	94	93	0.9
			DFA	3	0.02	108; 113; 117	108	117	113	4.0
				3	0.20	94; 95; 95	94	95	95	0.6
				6	overall		94	117	104	9.9
		pulp, dry	BYI 02960	3	0.01	77; 77; 80	77	80	78	2.2
				3	0.10	77; 78; 80	77	80	78	2.0
				6	overall		77	80	78	1.9
			DFA	3	0.02	94; 94; 95	94	95	94	0.6
				3	0.20	73; 73; 74	73	74	73	0.8
				6	overall		73	95	84	13.7
		starch	BYI 02960	3	0.01	92; 94; 95	92	95	94	1.6
				3	0.10	94; 94; 96	94	96	95	1.2
				6	overall		92	96	94	1.4
			DFA	3	0.02	102; 107; 107	102	107	105	2.7
				3	0.20	92; 97; 97	92	97	95	3.0
				6	overall		92	107	100	6.0
		flakes	BYI 02960	3	0.01	72; 76; 82	72	82	77	6.6
				3	0.10	68; 82; 83	68	83	78	10.8
				6	overall		68	83	77	8.0
			DFA	3	0.02	98; 109; 110	98	110	106	6.3
				3	0.20	63; 67; 69	63	69	66	4.6
				6	overall		63	110	86	25.6
		crisps (Am.:	BYI 02960	3	0.01	84; 88; 89	84	89	87	3.0
				3	0.10	87; 89; 92	87	92	89	2.8

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Individual recoveries	Recovery (%) Min	Max	Mean	RSD
		chips)		6	overall		84	92	88	3.0
			DFA	3	0.02	118; 118; 119	118	119	118	0.5
				3	0.20	80; 80; 89	80	89	83	6.3
				6	overall		80	119	101	19.5
		tuber, stored	BYI 02960	4	0.01	94; 95; 95; 96	94	96	95	0.9
				4	0.10	88; 92; 94; 96	88	96	93	3.7
				8	overall		88	96	94	2.8
			DFA	4	0.02	98; 101; 113; 118	98	118	108	8.9
				4	0.20	96; 97; 101; 101	96	101	99	2.7
				8	overall		96	118	103	7.7

The residue of flupyradifurone and its metabolite DFA were determined in the harvested potato tubers (raw agricultural commodity) at day 7 ± 1 and in the processed products. These values were used for the calculation of "processing" factors (Table A 31).

Table A 31: Residue data from potato processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)						
potato, tuber (stored)	<0.010 <0.010 mean: <0.010	14	tuber, washed	<0.01	n.c.	
			peel washed	<0.01	n.c.	
			tuber, peeled	<0.01	n.c.	
			tuber, cooked	<0.01	n.c.	
potato, tuber (stored)	<0.010 <0.010 mean: <0.010	14	tuber with peel, cooked	<0.01	n.c.	
			fibre	<0.01	n.c.	
			pulp, dry	<0.01	n.c.	
			starch	<0.01	n.c.	
potato, tuber (stored)	<0.010 <0.010 mean. <0.010	14	waste	<0.01	n.c.	
			waste, dried	<0.01	n.c.	
			flakes	<0.01	n.c.	
potato, tuber (stored)	<0.010 <0.010 mean: <0.010	14	crisps (Am.: chips)	<0.01	n.c.	
potato, tuber (stored)	<0.010 <0.010 mean: <0.010	14	tuber, washed	<0.01	n.c.	
			peel washed	<0.01	n.c.	
			tuber, peeled	<0.01	n.c.	
			tuber, cooked	<0.01	n.c.	
potato, tuber (stored)	<0.010 <0.010 mean: <0.010	14	tuber with peel, cooked	<0.01	n.c.	
			fibre	<0.01	n.c.	
			pulp, dry	<0.01	n.c.	
			starch	<0.01	n.c.	
potato, tuber	<0.010	14	waste	<0.01	n.c.	

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
(stored)	<0.010 mean: <0.010		waste, dried	<0.01	n.c.	
			flakes	<0.01	n.c.	
potato, tuber (stored)	<0.010 <0.010 mean: <0.010	14	crisps (Am.: chips)	<0.01	n.c.	
DFA, expressed as DFA (RD-Mo)						
potato, tuber (stored)	0.0073 0.0070 mean: 0.0072	14	tuber, washed	0.009	1.2	Median: PF: 1.1
			peel washed	0.0067	0.9	Median: PF: 0.9
			tuber, peeled	0.0073	1.0	Median: PF: 1.0
			tuber, cooked	<0.0067	<0.9	Median: PF: <0.9
potato, tuber (stored)	0.0073 <0.0067 mean: 0.0070	14	tuber with peel, cooked	0.0073	1.0	Median: PF: 1.0
potato, tuber (stored)	0.0103 <0.0067 mean: 0.0087	14	fibre	<0.0067	<0.8	Median: PF: <0.8
			pulp, dry	<0.0067	<0.8	Median: PF: <0.8
			starch	<0.0067	<0.8	Median: PF: <0.8
potato, tuber (stored)	0.0106 <0.0067 mean: 0.0087	14	waste	<0.0067	<0.8	Median: PF: <0.8
			waste, dried	<0.0067	<0.8	Median: PF: <0.8
			flakes	<0.0067	<0.8	Median: PF: 1.3
potato, tuber (stored)	<0.0067 <0.0067 mean: <0.0067	14	crisps (Am.: chips)	0.022	>3.3	Median: PF: >2.6
potato, tuber (stored)	0.008 0.007 mean: 0.008	14	tuber, washed	0.007	0.9	Median: PF: 1.1
			peel washed	0.007	0.9	Median: PF: 0.9
			tuber, peeled	0.008	1.0	Median: PF: 1.0
			tuber, cooked	<0.0067	<0.9	Median: PF: <0.9
potato, tuber (stored)	0.0067 0.0080 mean: 0.0074	14	tuber with peel, cooked	<0.0067	<0.9	Median: PF: 1.0
potato, tuber (stored)	0.0080 0.0083 mean: 0.0082	14	fibre	<0.0067	<0.8	Median: PF: <0.8
			pulp, dry	<0.0067	<0.8	Median: PF: <0.8
			starch	<0.0067	<0.8	Median: PF: <0.8
potato, tuber (stored)	0.0080 0.0087 mean; 0.0084	14	waste	<0.0067	<0.8	Median: PF: <0.8
			waste, dried	<0.0067	<0.8	Median: PF: <0.8
			flakes	0.014	1.7	Median: PF: 1.3
potato, tuber (stored)	0.009 0.007 mean: 0.008	14	crisps (Am.: chips)	0.014	1.8	Median: PF: >2.6

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)						
potato, tuber (stored)	0.03 0.03 mean: 0.03	14	tuber, washed	0.04	1.3	Median: PF: 1.2
			peel washed	0.03	1.0	Median: PF: 1.0
			tuber, peeled	0.03	1.0	Median: PF: 1.0
			tuber, cooked	<0.03	<1.0	Median: PF: <1.0
potato, tuber (stored)	0.03 <0.03 mean: 0.03	14	tuber with peel, cooked	0.03	1.0	Median: PF: 1.0
potato, tuber (stored)	0.04 <0.03 mean: 0.04	14	fibre	<0.03	<0.8	Median: PF: <0.8
			pulp, dry	<0.03	<0.8	Median: PF: <0.8
			starch	<0.03	<0.8	Median: PF: <0.8
potato, tuber (stored)	0.04 <0.03 mean: 0.04	14	waste	<0.03	<0.8	Median: PF: <0.8
			waste, dried	<0.03	<0.8	Median: PF: <0.8
			flakes	<0.03	<0.8	Median: PF: 1.1
potato, tuber (stored)	<0.03 <0.03 mean: <0.03	14	crisps (Am.: chips)	0.08	>2.7	Median: PF: >2.0
potato, tuber (stored)	0.03 0.03 0.03	14	tuber, washed	0.03	1.0	Median: PF: 1.2
			peel washed	0.03	1.0	Median: PF: 1.0
			tuber, peeled	0.03	1.0	Median: PF: 1.0
			tuber, cooked	<0.03	<1.0	Median: PF: <1.0
potato, tuber (stored)	0.03 0.03 0.03	14	tuber with peel, cooked	<0.03	<1.0	Median: PF: 1.0
potato, tuber (stored)	0.03 0.04 mean: 0.04	14	fibre	<0.03	<0.8	Median: PF: <0.8
			pulp, dry	<0.03	<0.8	Median: PF: <0.8
			starch	<0.03	<0.8	Median: PF: <0.8
potato, tuber (stored)	0.03 0.04 mean: 0.04	14	waste	<0.03	<0.8	Median: PF: <0.8
			waste, dried	<0.03	<0.8	Median: PF: <0.8
			flakes	0.05	1.3	Median: PF: 1.1
potato, tuber (stored)	0.03 0.04 mean: 0.04	14	crisps (Am.: chips)	0.05	1.3	Median: PF: >2.0

* PF = processing factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Conclusion

Following the intended application rate of flupyradifurone in northern Europe (1 x 0.1 kg a.s./ha) to two separate trials of potatoes, mature tubers were collected for subsequent processing. The potatoes stored at ambient temperature were processed into crisps, flakes and starch. In addition peeled potatoes and potatoes with peel were cooked (cooked tuber with peel). No residues of flupyradifurone, at or above the LOQ were detected in the raw commodity or in any processing product. Low residues of the metabolite DFA (up to 0.0087 mg/kg) were detected in some of the stored potato samples (RAC samples). A concentration of the DFA residues due to processing was only observed for flakes and crisps. Washing, peeling and cooking of potatoes resulted neither in a reduction nor an increase of the DFA residues.

The results of the EU processing studies are in good agreement with the results of the US processing studies and the processing factors for the total residue (sum of flupyradifurone and DFA) of flakes and crisps could be combined to estimate median processing factors.

Figure A 6: Processing flowchart for potato – cooked potatoes

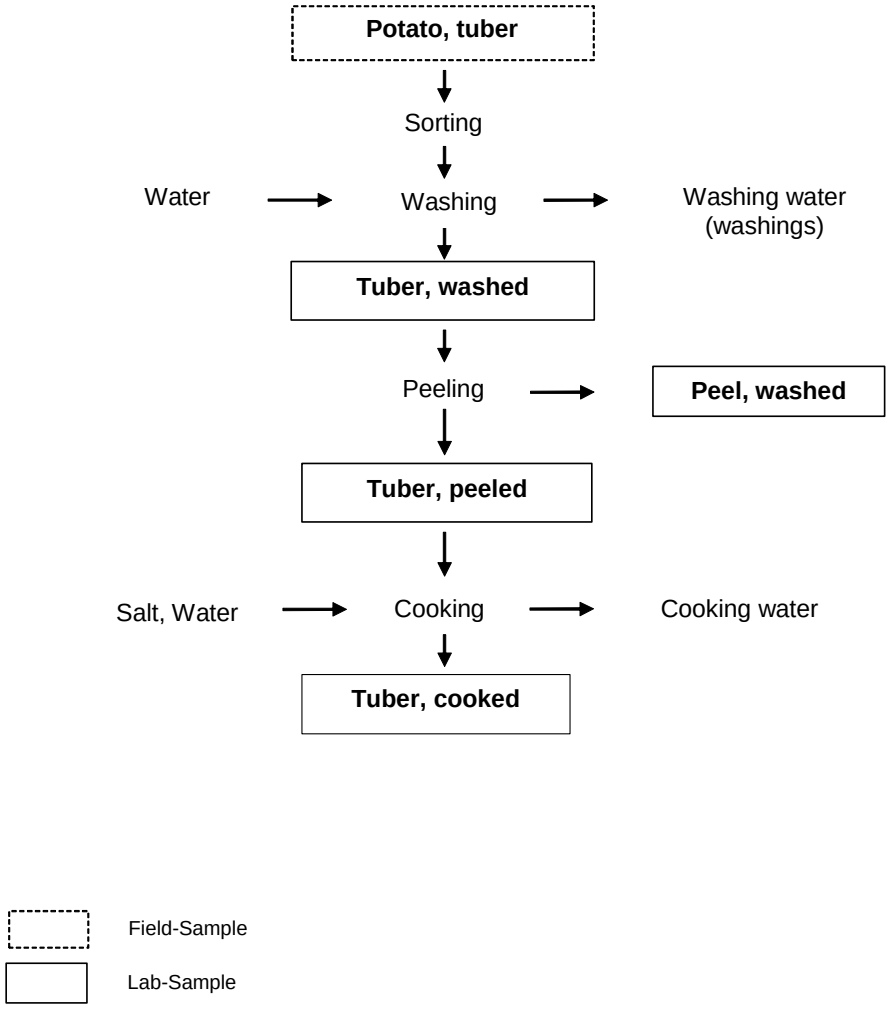


Figure A 7: Processing flowchart for potato – cooked potatoes with peel

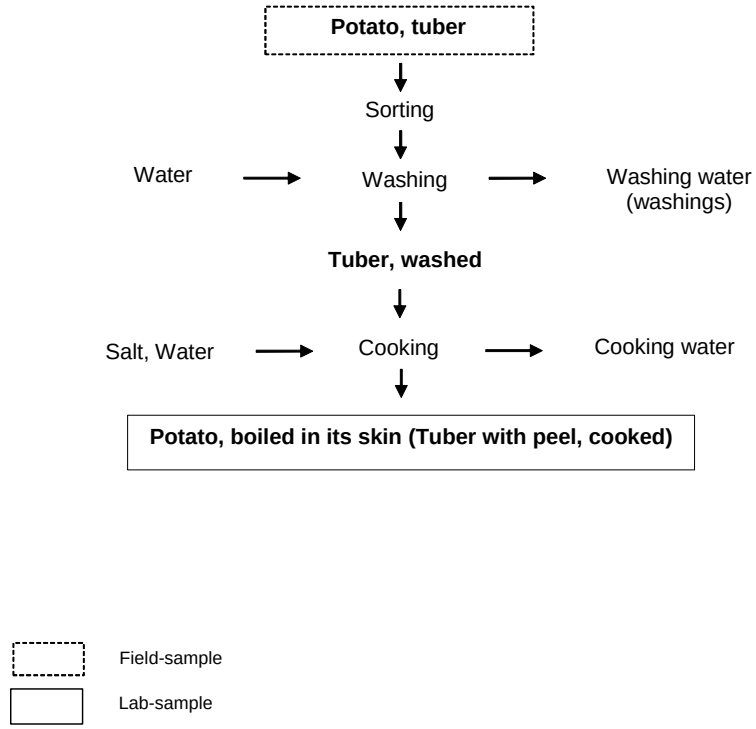


Figure A 8: Processing flowchart for potato – starch

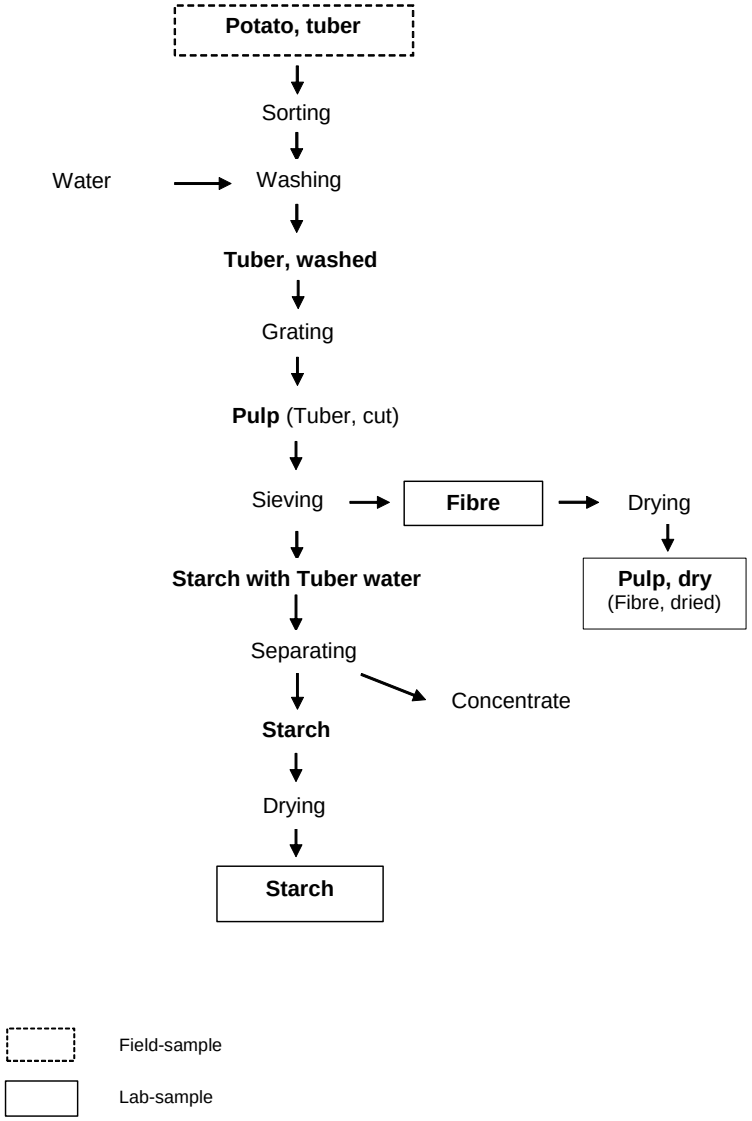


Figure A 9: Processing flowchart for potato – flakes

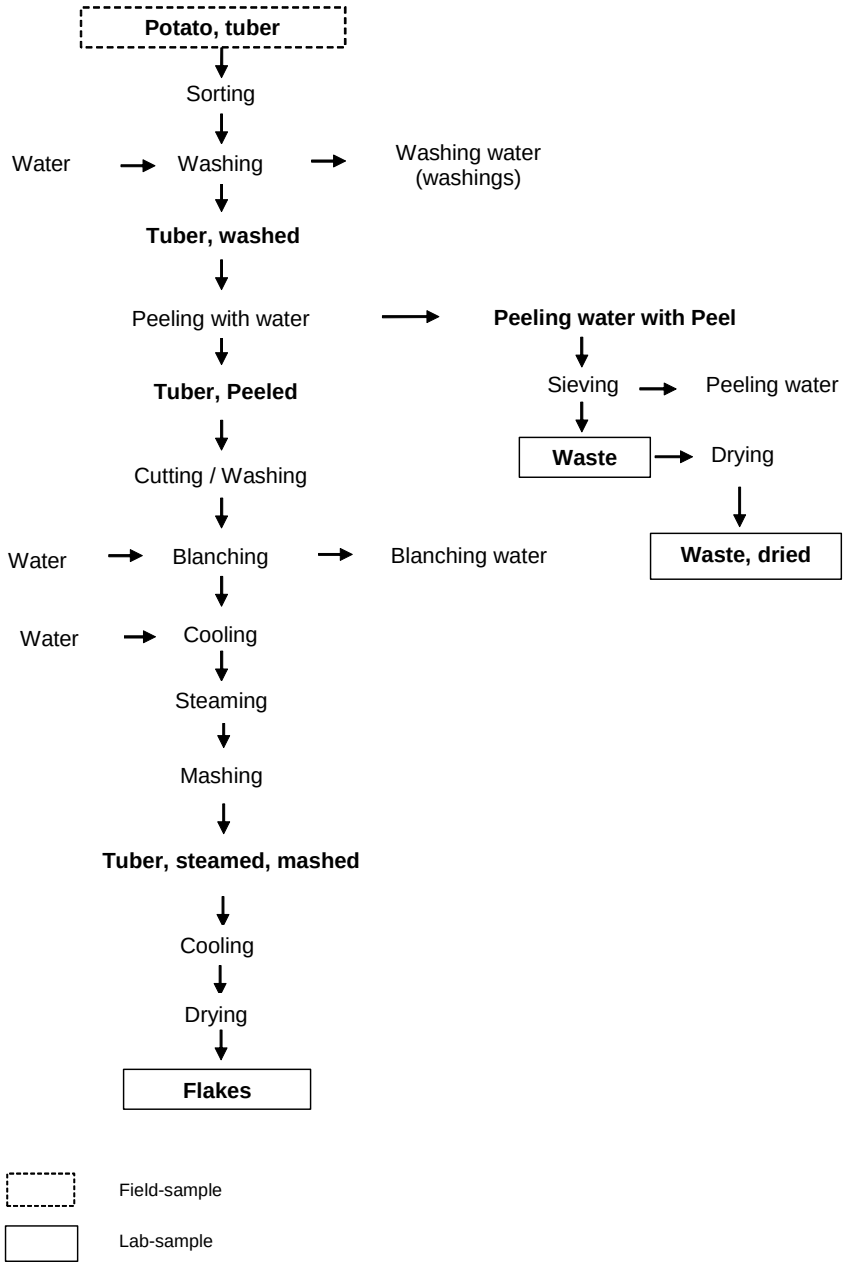
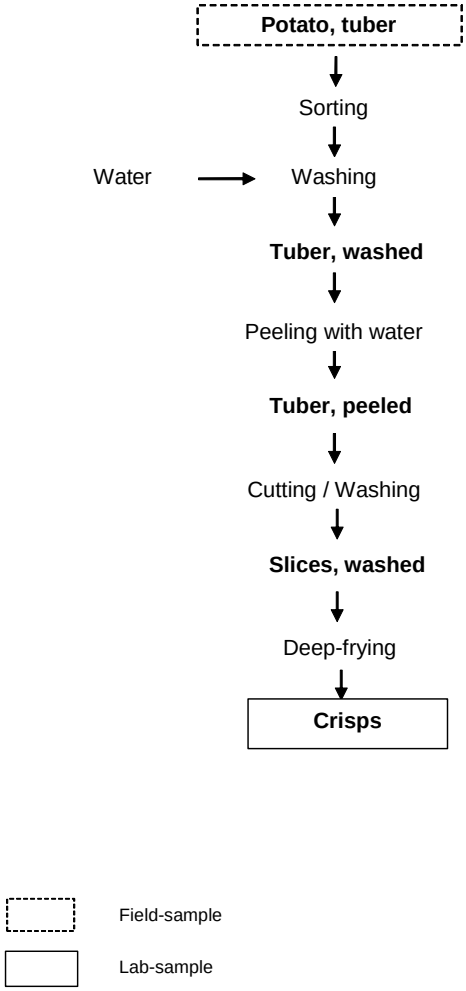


Figure A 10: Processing flowchart for potato – crisps



A 2.1.5.2.5 Barley , study report 10-3410

Comments of zRMS:	Two residue trials were conducted in Germany to determine the magnitude of the relevant residues of BYI 02960 in/on spring barley and its processed fractions (malt sprouts; brewer's malt; brewer's grain, hops draff; brewer's yeast; beer; pearl barley and pearl barley rub off) after one application of BYI 02960 at an exaggerated rate of approx. 460 g a.s. /ha. The results show that BYI 02960 residues and total residue of BYI 02960 (sum of BYI 02960 and DFA) did not concentrate in any processed product, except for non-edible commodity, for rub-off of pearl barley. The mean processing factor for pearl barley rub off equals 2.9 for BYI 02960 and for sum of BYI 02960 and DFA. For the metabolite DFA, the mean processing factors in rub-off of pearl barley are 1.8. In addition DFA concentrates in malt sprouts, for which the mean processing factor are 12.1. It should be noted that no concentration in malt sprouts was detected for the parent BYI 02960 (mean PF = 0.40), so no concentration was detected for the total residue (mean PF = 0.77). All other processing steps resulted in a dilution of BYI 02960 and DFA residues. The study is acceptable.

Reference:	**KCA 6.5.3/05**
Title:	Processing study - Determination of the residues of BYI 02960 in/on spring barley and the processed fractions (malt sprouts; brewer's malt; brewer's grain, hops draff; brewer's yeast; beer; pearl barley; pearl barley rub off) after spraying of BYI 02960 SL 200 in the field in Germany
Report:	Schulte, G.; Ruhl, S.; 2012; 10-3410; M-439853-01-1
Authority registration No:	
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed, EC Guidance working document 7029/VI/95 rev.5 (1997-07-22) EC Guidance working document 7035/VI/95 rev.5 (1997-07-22) OECD Guideline for the Testing of Chemicals, Magnitude of the Pesticide Residues in Processed Commodities, 508 (2008-10-03) US EPA Ref: OCSPP 860.1520. SUPP
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

Two supervised residue trials were conducted in northern Europe (Germany) to determine the magnitude of the relevant residues of BYI 02960 in/on spring barley and its processed fractions. The samples of barley grain (raw agricultural commodity = RAC) to be processed were obtained after one spray application with BYI 02960 SL 200, an SL formulation containing 200 g/L BYI 02960.

BYI 02960 SL 200 was applied once at a rate of approx. 460 g a.s./ha. The application was conducted approx. 20-22 days before harvest at a BBCH growth stage of 83-87. The application scheme reflected an overdosing (approx. 3x higher than the envisaged EU worst case). Grain samples for processing were harvested at maturity (BBCH 89).

Field samples for analysis of the raw agricultural commodity were deep-frozen immediately after sampling and were stored deep-frozen until shipment to the Laboratory for Sampling, Preparation Technique and Sample Logistics (PVTL), Bayer CropScience AG in D-40789 Monheim am Rhein. The field samples for processing were stored at ambient temperature (room temperature) until the start of processing. The processed samples (lab samples) were deep-frozen after sampling and were stored deep-frozen until shipment to the specimen consignee for subsequent analysis.

The samples were analyzed according to method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1).

The LOQ was 0.01 mg/kg for flupyradifurone and 0.02 mg/kg for DFA when expressed as flupyradifurone (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of flupyradifurone and DFA, expressed as flupyradifurone) was 0.03 mg/kg. In parallel to the residue analysis, the method was validated by recovery experiments.

The maximum storage period of deep-frozen samples intended for the analysis of BYI 02960 and its metabolites was 321 days (approx. 11 months), which is within the acceptable range for storage stability of the residues of flupyradifurone in matrices with high starch content.

The sample materials to be analyzed were grain, beer, brewer's grain, brewer's yeast, brewer's malt, malt sprouts, pearl barley rub off, pearl barley, and hops draff. The processing of barley was performed in a specialized pilot plant to simulate industrial procedures at a laboratory scale.

Processing procedures:
In order to obtain acceptable results, grain with a moisture content of approx. 14% is required. As the optimal moisture content was already reached at the sampling date, no drying of the field specimens was necessary before cleaning.

Malting:
Sieving: Before malting (after cleaning) is started, the grain is sieved (sieve mesh 2.5 mm).

Steeping: Sieved barley grain is transferred in a special steeping vessel. During steeping, water is supplied to the interior of the kernel. As a result the enzymes become active and the germination process begins. Water uptake depends on the steeping time, steeping temperature, kernel size, barley variety and harvest year. The final steeping degree was in the range of 44.2 to 44.4%.

Germination: The processes during germination can be divided into growth processes, enzyme formation and metabolic changes. Towards the end of steeping the rootlets break through the base of the grain and become visible. Activation of enzymes and formation of new enzymes are essential processes during germination (starch degrading enzymes, cytolytic enzymes, protein degrading enzymes and phosphoric acid splitting enzymes). For proper performance of germination it is necessary to control the duration of germination, the mean temperature of wet air and the relative humidity of the air.

Kiln-drying: After germination, the life processes are terminated by kilning. During kilning the water content is lowered, germination and modification are stopped; colour and flavor compounds are formed. The malt becomes stable and storable. Kiln-drying was conducted in a dry chamber. The maximum temperature during the kiln-drying process was 80.0°C. After kiln-drying the germs were removed mechanically by a trimmer. Brewer's malt and malt sprouts were sampled 2-4 days after malting. Until brewing the malt was stored at room temperature.

Brewing (Processing of Malt to Beer):
Mashing: Mashing is the homogeneous mixing of ground malt and water according to a definite temperature time regime (mash program). The main purpose of mashing (the dissolution and enzymatic conversion of ingredients) is to form as much extract and as good an extract as possible. Before mashing, the brewer's malt was dry milled in a special malt mill. The crushed malt was mixed with brew water. To produce Pilsener beer, mashing was started in a heatable tun.

Lautering (Wort extraction and separation): After mash boiling, the wort was separated from the insoluble malt components (brewer's grain). The extract remaining in the brewer's grain was extracted by washing with hot water (first filter runnings). The wort separation was done using a refining vat. After separation, the brewer's grain was sampled.
Wort boiling and conditioning: After addition of hop pellets, the separated wort was boiled (about 90 min at normal pressure). This process deactivates the malt enzymes, sterilizes the wort, extracts and isomerises the essential components of the hops, precipitates high molecular proteins (called "Bruch") and expels unwanted aromatic substances.

After boiling, the floes (hops draff) were separated in a whirlpool causing the sludge to deposit on the bottom in the shape of a cone. For cooling and ventilating the wort, an intra-plant circulation was used. By adding oxygen (intra-plant circulation) the conditions for the start of the fermentation were established. Hops draff was sampled.

Fermentation and maturation: The pure culture yeast ferments sugar of the wort to alcohol and carbon dioxide, but in the course of the yeast metabolism, unwanted by-products are also formed (diacetyl, higher alcohols and others). In the pilot plant, the classical primary fermentation (low fermentation) was carried out in bottom fermentation containers. Fermentation heat was dissipated by means of room ventilation.
The duration of main fermentation depends on temperature, on the starting extract concentration of the finished wort, on the ratio of non-fermentable sugars to the extract, on the final attenuation and on the yeast cell number (exact duration was recorded). As soon as the extract content of the fermented young beer was 2% higher than the final attenuation, the storing time began. Before maturation, the young beer was cooled down. During the main fermentation the yeast deposits on the tank bottom and was sampled as brewer's yeast.
At the beginning of maturation the young beer was stored at room temperature (warm maturation to break down the diacetyl) in casks. Then the young beer was stored under pressure (approx. 0.8 - 1.8 bar) at approx. 0 - 2 °C (cold maturation) for about 3-4 weeks. In this time the remaining extract was fermented. Unwanted flavour and odorous substances were decomposed or expelled. Sludge particles and yeast settle at the bottom. The rack beer was filtered using a special filter combination. During filtration, all organisms harming the beer (bacteria and yeast) were removed and sludge particles.

Pearl barley production:
Conditioning: Before beginning of pearl barley production, an optimal moisture content in the barley grain of about 15% is needed. As the optimal moisture content was already reached at the sampling date, no drying of the field specimens was necessary starting the pearl barley production.

Hulling: The specimens of spring barley were hulled using a "Vertikal-Schaelmaschine" (vertical hulling machine). Each sample was hulled until the stipulated abrasion for pearl barley of 30 - 35% was reached. The degree of abrasion (pearling dust/bran and flour) was determined by the proportion of pearl barley with respect to the total portion of cleaned grain used for hulling process. Pearl barley and pearl barley rub off were sampled.

Results and discussions
Parallel to the residue analysis, the analytical method was validated by recovery experiments. Concurrent recoveries were obtained from samples of grain (RAC), brewer´s yeast, beer, and malt sprouts. The recoveries for the sample material grain are also representative for the sample materials brewer's malt, brewer's grain, pearl barley, and pearl barley rub off.

The recovery determinations for the sample material hops draff were performed in study 10-3407 with hop.

In all matrices, concurrent recovery samples for parent compound were spiked at levels of 0.01 mg/kg, as well as at 0.10 mg/kg. A control sample of barley grain was additionally spiked at a level of 5.0 mg/kg. The spiking levels for hops draff were 0.1 mg/kg and 1.0 mg/kg. Mean recoveries for all matrices were always in the acceptable range of 70-110%, RSD values per fortification level (if applicable, $n \geq 3$) were well below 20%. For DFA, concurrent recovery samples for the barley matrices beer, brewer´s yeast, and malt sprouts were spiked at levels of 0.02 mg/kg as well as at 0.20 mg/kg (expressed in BYI 02960 equivalents). The spiking levels for barley grain were 0.02 mg/kg; 0.50 mg/kg and 5.0 mg/kg, and for hops draff 0.20 mg/kg and 1.0 mg/kg. Mean recoveries per fortification level were between 70-110% for all matrices, with RSD values <20%, if applicable ($n \geq 3$). In grain the mean recovery at the LOQ level was slightly above 110% (114%), however was considered to be acceptable. As the control sample of malt sprout showed already DFA residues, the recoveries were corrected by subtracting the blank value at the LOQ level. The method was considered acceptable as the mean recoveries were all within the acceptable range and the RSD values were ≤20%.

Residues of BYI 02960, DFA and the total residue (sum of parent compound and DFA) were determined in the raw agricultural commodity barley grain harvested 20-22 days after the application, and in the processed commodities. On the basis of these values, separate processing factors for BYI 02960, DFA and the total residue were calculated (Table A 33).

Table A 32: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in barley grain and processed grain matrices

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
10-3410 (10-3410-01 and 10-3410-02) GLP: yes 2010	barley, spring	grain*	BYI 02960	1	0.01	107	107	107	107	-
				1	0.1	109	109	109	109	-
				1	5	89	89	89	89	-
				3	overall		89	109	102	10.8
			DFA	1	0.02	114	114	114	114	-
				1	0.50	100	100	100	100	-
				1	5.0	80	80	80	80	-
				3	overall		80	114	98	17.4
		malt sprouts	BYI 02960	5	0.01	76; 66;73; 82; 88	66	88	77	10.9
				3	0.1	84; 79; 85	79	85	83	3.9
				8	overall		66	88	79	9.1
			DFA	5	0.02	72; 78;84; 85; 85	72	85	81	7.1
				3	0.20	83; 83; 87	83	87	84	2.7
				8	overall		72	87	82	5.9
		brewer's yeast	BYI 02960	5	0.01	102;109; 106;109; 99	99	109	105	4.2
				3	0.1	106; 108; 111	106	111	108	2.3
				8	overall		99	111	106	3.8
			DFA	5	0.02	92; 96;97; 98; 101	92	101	97	3.4
				3	0.20	91; 90; 87	87	91	89	2.3
				8	overall		87	101	94	5.0
		beer	BYI 02960	5	0.01	95;103; 96; 94; 103	94	103	98	4.5
				3	0.1	97; 92; 94	92	97	94	2.7
				8	overall		92	103	97	4.3
			DFA	5	0.02	97;99; 101; 99; 100	97	101	99	1.5
				3	0.20	92; 98; 95	92	98	95	3.2
				8	overall		92	101	98	3.0

* Grain covers also brewer´s malt, brewer´s grain, pearl barley and pearl barley rub off

Remark: Additional concurrent recoveries for the matrix barley grain were conducted in the residue studies in order to validate the method in the respective matrix. In addition, a full validation set was conducted in the course of method 01304 for wheat grain.

Table A 33: Residue data from barley grain processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)	DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)								
Barley	0.84	1.6	22/20	malt sprouts	0.32	0.66	0.38	0.41	Median: 0.40
				brewer´s malt	0.37	0.83	0.44	0.52	Median: 0.48
				brewer´s grain	**0.07**	**0.04**	**0.08**	**0.03**	**Median: 0.05**
				hops draff	0.16	0.29	0.19	0.18	Median: 0.19
				brewer´s yeast	0.06	0.17	0.07	0.11	Median: 0.09

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
				beer	0.06	0.08	0.07	0.05	Median: 0.06
				pearl barley rub off	2.8	4.0	3.3	2.5	Median: 2.9
				pearl barley	0.12	0.10	0.14	0.06	Median: 0.10
DFA, expressed as DFA (RD-Mo)									
Barley	0.04	0.03	22/20	malt sprouts	0.49	0.36	12	12	Median: 12
				brewer´s malt	0.04	0.02	1.0	0.67	Median: 0.83
				brewer´s grain	**<0.02**	**<0.02**	**<0.5**	**<0.67**	**Median: <0.70**
				hops draff	<0.20	<0.20	<5.0	<6.7	Median: <5.9
				brewer´s yeast	<0.02	<0.02	<0.5	<0.67	Median: <0.70
				beer	<0.02	<0.02	<0.5	<0.67	Median: <0.70
				pearl barley rub off	0.08	0.05	2.0	1.7	Median: 1.8
				pearl barley	0.03	<0.02	0.75	0.67	Median: 0.71
Sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)									
Barley	0.88	1.6	22/20	malt sprouts	0.81	1.0	0.92	0.63	Median: 0.77
				brewer´s malt	0.41	0.85	0.47	0.52	Median: 0.49
				brewer´s grain	**0.09**	**0.06**	**0.10**	**0.04**	**Median: 0.07**
				hops draff	0.36	0.49	0.41	0.30	Median: 0.35
				brewer´s yeast	0.08	0.19	0.09	0.12	Median: 0.10
				beer	0.08	0.10	0.09	0.06	Median: 0.08
				pearl barley rub off	2.9	4.1	3.3	2.5	Median: 2.9
				pearl barley	0.15	0.12	0.17	0.07	Median: 0.12

* PF = Processing Factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Conclusion

Concentration of BYI 02960 and DFA residues (and therefore also of the total residue) was only seen in the non-edible commodity pearl barley rub off. The mean processing factor for pearl barley rub off was 2.9 for BYI 02960 and the total residue, and 1.8 for DFA. A concentration of DFA residues was also detected in malt sprouts (mean PF = 12), whereas no concentration was detected for the parent BYI 02960 (mean PF = 0.40). As a consequence, also no concentration was detected for the total residue (mean PF = 0.77).
All other processing steps resulted in a dilution of BYI 02960 and DFA residues.

Figure A 11: Processing flowchart of grain into malt

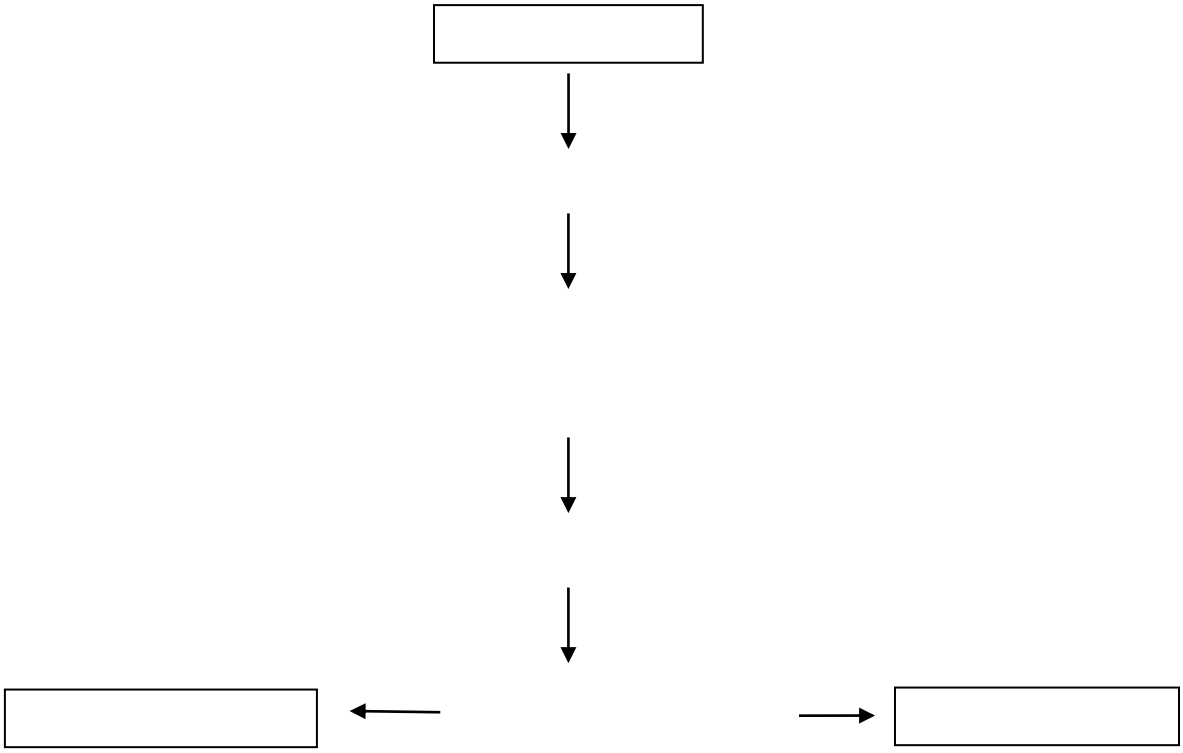


Figure A 12: Processing flowchart of malt into beer

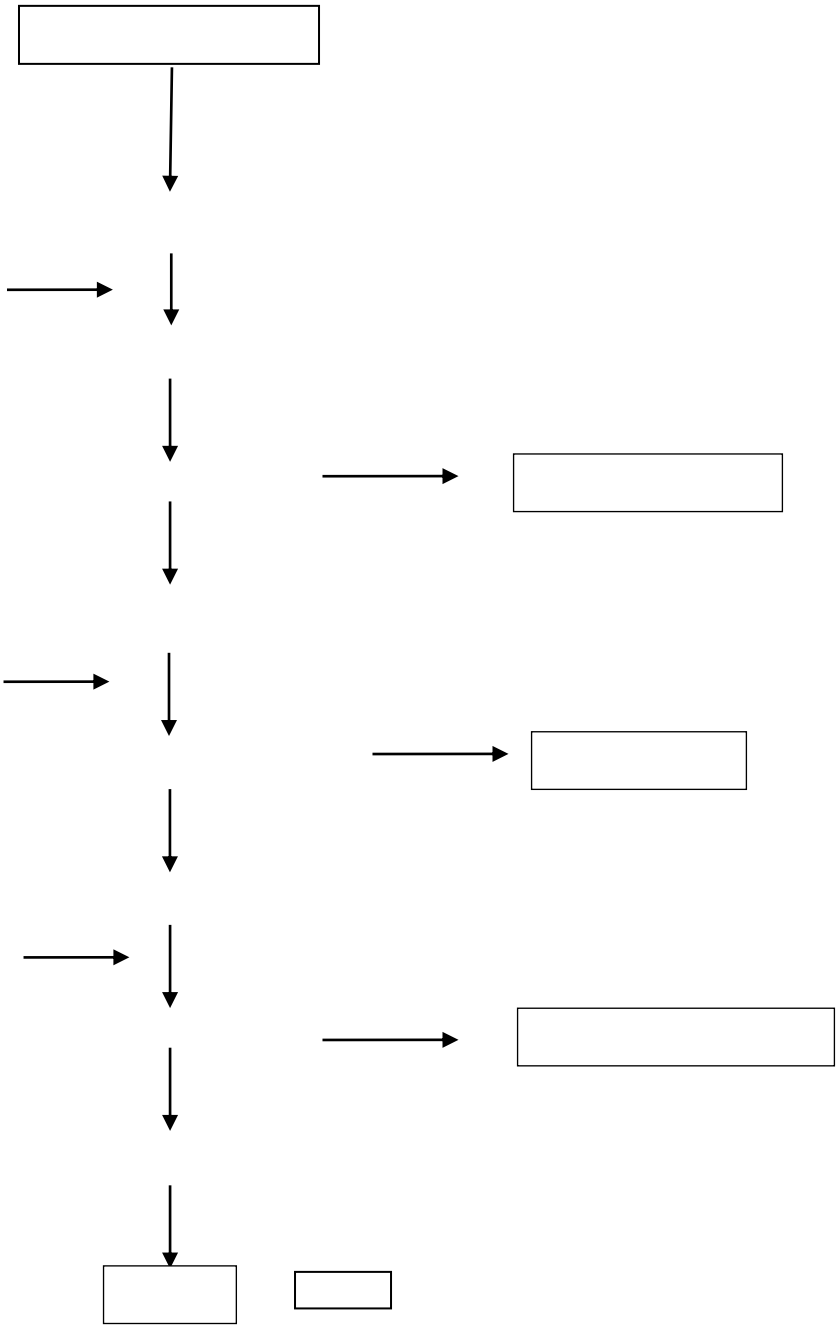
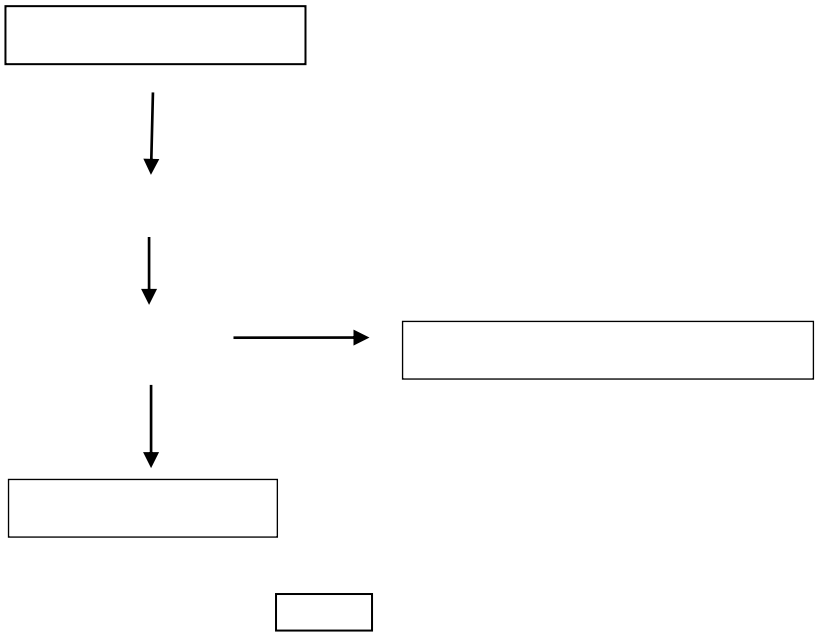


Figure A 13: Processing flowchart of grain into pearl barley



A 2.1.5.2.6 Wheat , study report 10-3409

Comments of zRMS:	The purpose of the study 10-3409 was to determine the magnitude of the relevant residues of BYI 02960 in/on processed fractions of wheat (semolina; semolina bran; white flour bran; white flour; white bread; whole meal; whole meal bread and wheat germ). The samples of wheat grain to be processed and reference raw agricultural commodity (RAC) samples were obtained after one spray application with BYI 02960 at rate of 460 g a.s./ha. The study included two supervised residue trials conducted in northern Europe (Germany) during the 2010 season. BYI 02960 residues concentrate significantly in the bran fractions of semolina (mean processing factor of 4.6) and white flour (mean processing factor of 6.2). A transfer of the residues in semolina or white flour was not detected. Also for DFA a significant concentration was seen in the bran fractions (mean processing factor for semolina of 25 and for white flower of 26), however also a slight transfer of DFA was observed in semolina, white flower, whole flour and wheat germ. Overall, the total residue of BYI 02960 (sum of BYI 02960 and DFA) did only concentrate in the bran fractions (mean processing factor of 5.2 and 6.6 for semolina bran and white flour bran, respectively), in whole flour (mean processing factor of 1.5) and in wheat germ (mean processing factor of 1.1). The study is acceptable.

Reference:	**KCA 6.5.3/06**
Title:	Processing study - Determination of the residues of BYI 02960 in/on wheat and the processed fractions (semolina; semolina bran; whiteflour bran; white flour; white bread; whole meal; wholemeal bread and wheat germ) after spraying of BYI 02960 SL 200 in the field in Germany
Report:	Schulte, G.; Bauer, J.; 2012; 10-3409; M-440307-01-1
Authority registration No:	
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22) EC guidance working document 7035/VI/95 rev. 5 (1997-07-22) OECD Guideline for the Testing of Chemicals, Magnitude of the Pesticide Residues in Processed Commodities, 508 (2008-10-03) US EPA Ref: OCSSP Guideline No. 860. 1520. SUPP
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	Yes bn
Duplication (if vertebrate study):	

Materials and methods

Two field trials were conducted in the northern European residue region to measure the magnitude of BYI 02960 residues in/on wheat processed commodities following one foliar spray application of BYI 02960 200 SL at an application rate of approx. 460 g a.s./ha and a water volume of approx. 300 L/ha. The application scheme (exaggerated rate) was chosen in order to ensure that detectable residues can be found in the relevant raw commodities at harvest, thus allowing elucidation of processing factors. Wheat samples to be processed (grain) were sampled 22 days after the treatment at commercial harvest (BBCH 89).

The maximum storage period of deep-frozen samples intended for the analysis BYI 02960 and its metabolites was 160 days (approx. 5 months), which is within the acceptable rage for the storage stability of the residues of flupyradifurone in high starch matrices.

After processing (described below), the samples were analyzed according to method 01304 (Li, Y.; Schoening, R.; 2012; M-415504-02-1). The LOQ was 0.01 mg/kg for flupyradifurone and 0.02 mg/kg for DFA when expressed as flupyradifurone (corresponding to an LOQ of 0.0067 mg/kg, when expressed as DFA). The calculated LOQ for the total residue (sum of flupyradifurone and DFA, expressed as

flupyradifurone) was 0.03 mg/kg. Prior and parallel to the residue analysis, the method was validated by recovery experiments.

Processing procedures:
The processing of wheat samples into the processed fractions semolina, semolina bran, white-flour bran; white flour, white bread, whole meal, whole meal bread, and wheat germ was performed to simulate industrial procedures at a laboratory scale.

Drying, cleaning, and conditioning of wheat grain: Frozen field samples for processing were defrosted, followed by drying for 12 hours at 30°C at the test site. The dried field samples were cleaned and then shipped at ambient temperature for further processing.
Weight and moisture content of grain specimens were determined on arrival. If the moisture content in the grain was above 15–16%, the specimens were dried at 30°C for approx. 9 hours. If the moisture content in the grain was below 15–16%, the specimens were moistened by addition of tap water. All grain samples were adjusted to a moisture content of 15.5-15.6%.

Milling of white flour (type 550) and baking of white bread: In a closed system with different pairs of smooth rollers and sifter passages, the grain was milled to straight flour, semolina, and semolina bran. Samples of semolina and semolina bran were collected.
In a further processing step, low grade meal (toppings) was separated from the semolina bran using a centrifuge/scouring machine. This process resulted in white flour bran and low grade meal (toppings). A sample of white-flour bran was collected.
The mineral contents of straight flour and low grade meal were determined. If the mineral content of straight flour exceeded 510-630 g/100 kg flour, both fractions were mixed until a mineral content of 510-630 g/100 kg flour was achieved, forming the processed fraction white flour (type 550). A sample of white flour (type 550) was taken.
For baking a 1.0 kg loaf of white bread, white flour (approx. 1.0 kg), yeast (approx. 40 g), salt (approx. 20 g), and water (approx. 0.7 L) were mixed. Subsequently, the dough was kneaded for 7 min and fermented for about 20 min. It was then moved for 10 min followed by a second fermentation period of 50 min. The baking process was conducted at 220°C for 30 min. Afterwards, a sample of white bread was taken (dependent on the wheat grain quality and the baking process/course, the processing parameters were adjusted and may vary in comparison to the above-mentioned values).

Milling of whole meal and baking of whole-meal bread: For the generation of whole meal and whole-meal bread, the same milling procedure was used as described for the production of white flour (type 550). After milling, the bran was cracked with an impact mill into smaller pieces. All milling products of the process were mixed homogeneously in a special flour mixer to generate the processed fraction whole meal. A sample of the whole meal was collected.
For baking a 1.0 kg loaf of whole-meal bread, whole meal (approx. 1.0 kg), yeast (approx. 40 g), salt (approx. 20 g), and water (approx. 0.7 L) were mixed. Subsequently, the resulting dough was kneaded for 7 min and fermented for about 20 min. It was then moved for 10 min followed by a second fermentation period of 40 min. The baking process was conducted at 220°C for 30 min. A sample of whole-meal bread was taken (dependent on the wheat grain quality and the baking process/course, the processing parameters were adjusted and may vary in comparison to the above-mentioned values).

Production of wheat germ: Wheat grain was broken to "bruised grain" in a special mill (roller mill with 0.5 mm roller distance). The fraction 400-1000 µm was collected, the fraction above 1000 µm was broken once more (0.3 mm roller distance). This milling/sieving process was performed a total of three times, with a final roller distance of 0.2 mm. The fractions obtained below 400 µm and the fractions above 1000 µm were completely excluded from further processing.
The fraction 400-1000 µm was put in a special separator ("Leichtgewichtsausleser"). Due to the different specific weights of the bran, semolina, and germ, the semolina/germ mixture was separated from most parts of the bran.
Subsequently, the semolina/germ mixture was milled to flour and small wheat germ discs (incl. parts of bran) in a mill with a pair of smooth rollers. The wheat germ with parts of bran was then sieved to

separate the various fractions (germ with small parts of bran, and bran). From the separated germ discs, small parts of bran were removed manually. A sample of wheat germ was taken.

Results and discussions

The validation of the sample material wheat grain was done during the main validation for method 01304 and was already evaluated at EU level.

Concurrent recoveries were obtained from samples of wheat grain, meal, semolina bran, whole-meal bread. The recoveries for the sample material grain are also representative for whole meal, white flour, semolina, semolina bran, white-flour bran, and wheat germ; those for the sample material whole-meal bread are also representative for white bread.

In wheat grain and in whole-meal bread samples, concurrent recovery samples for parent compound were spiked at levels of 0.01 mg/kg and 0.50 mg/kg; in wheat meal, the spiking level was 0.50 mg/kg. In semolina, recovery samples for parent compound were spiked at 0.01 mg/kg. Mean recoveries for parent compound for all matrices were 89 - 110%, with RSDs in the larger validation sets (n>2) of 1.2 - 6.3%; n=3 - 5.

For DFA, concurrent recovery samples for wheat grain and for whole-meal bread were spiked at levels of 0.02 mg/kg and 0.50 mg/kg (expressed in BYI 02960 equivalents); the fortification level in wheat meal was 0.50 mg/kg. In semolina samples, recovery samples DFA were spiked at 0.02 mg/kg. Mean recoveries for DFA in all matrices were 72 - 89%, with RSDs in the larger validation sets (n>2) of 3.8 - 8.6%; n=3 - 5.

The residue values and processing factors of BYI 02960 and DFA, as well as calculated total BYI 02960 residue data (sum of parent compound BYI 02960 and DFA, expressed as BYI 02960 equivalents) for the wheat RAC and the processed commodities are provided in Table A 35.

Table A 34: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in wheat grain and processed matrices

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Forti-fication level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
10-3409 (10-3409-01) and (10-3409-02) GLP: yes 2010	wheat	grain	BYI 02960	5	0.01	95; 96; 96; 99; 110	95	110	99	6.3
				3	0.50	87; 88; 91	87	91	89	2.3
				8	overall		87	110	95	7.6
			DFA	5	0.02	84; 89; 89; 91; 93	84	93	89	3.8
				3	0.50	71; 73; 81	71	81	75	7.1
				8	overall		71	93	84	9.9
		meal	BYI 02960	3	0.50	88; 91; 93	88	93	91	2.8
			DFA	3	0.50	68; 73; 76	68	76	72	5.6
		semolina bran	BYI 02960	5	0.01	104; 107; 110; 110; 119	104	119	110	5.1
			DFA	5	0.02	80; 82; 86; 90; 99	80	99	87	8.6
		wholemeal bread	BYI 02960	5	0.01	98; 101; 102; 102; 106	98	106	102	2.8
				3	0.50	95; 97; 97	95	97	96	1.2
				8	overall		95	106	100	3.6
			DFA	5	0.02	80; 80; 84; 88; 89	80	89	84	5.1
				3	0.50	73; 76; 82	73	82	77	6.0
				8	overall		73	89	82	6.8

Remark re. *grain*: also covers *whole meal*, *white flour*, *semolina*, *semolina bran*, *white-flour bran*, and *wheat germ*
Remark re. *whole-meal bread*: also covers *white bread*

Table A 35: Residue data from wheat grain processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)									
Wheat grain	0.23	0.14	22	Semolina bran	1.1	0.61	4.8	4.4	Median: 4.6
				Semolina	0.32	0.06	1.4	0.4	Median: 0.9
				White-flour bran	1.5	0.81	6.5	5.8	Median: 6.2
				White flour	0.07	0.02	0.3	0.1	Median: 0.2
				White bread	0.05	0.02	0.2	0.1	Median: 0.2
				Whole meal	0.36	0.21	1.6	1.5	Median: 1.5
				Whole-meal bread	0.22	0.12	1.0	0.9	Median: 0.9
				Wheat germ	0.30	0.11	1.3	0.8	Median: 1.0
DFA, expressed as DFA (RD-Mo)									
Wheat grain	0.02	<0.02	22	Semolina bran	0.5	0.02	25	>1	Median: 25
				Semolina	0.03	<0.02	1.5	n.c.	Median: 1.5
				White-flour bran	0.52	0.03	26	>1.5	Median: 26
				White flour	0.03	<0.02	1.5	n.c.	Median: 1.5
				White bread	0.02	<0.02	1.0	n.c.	Median: 1.0
				Whole meal	0.03	<0.02	1.5	n.c.	Median: 1.5
				Whole-meal bread	<0.02	<0.02	1.0	n.c.	Median: 1.0
				Wheat germ	0.03	<0.02	1.5	n.c.	Median: 1.5
Sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)									
Wheat grain	0.25	0.16	22	Semolina bran	1.6	0.63	6.4	3.9	Median: 5.2
				Semolina	0.35	0.08	1.4	0.5	Median: 1.0
				White-flour bran	2.0	0.84	8.0	5.3	Median: 6.6
				White flour	0.10	0.04	0.4	0.3	Median: 0.3
				White bread	0.07	0.04	0.3	0.3	Median: 0.3
				Whole meal	0.39	0.23	1.6	1.4	Median: 1.5
				Whole-meal bread	0.24	0.14	1.0	0.9	Median: 0.9
				Wheat germ	0.33	0.13	1.3	0.8	Median: 1.1

* PF = Processing Factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Conclusion

Following application of BYI 02960 SL 200 to two separate trials of wheat, BYI 02960 residues concentrate significantly in the bran fractions of semolina and white flour. A transfer of the residues in semolina or white flour was not detected.

Also for DFA a significant concentration was seen in the bran fractions (semolina and white flour), however also a slight transfer of DFA was observed in semolina, white flour, whole meal and wheat germ.

Overall, the total residue of BYI 02960 (sum of BYI 02960 and DFA) did only concentrate in the bran fractions (mean processing factor of 5.2 and 6.6. for semolina bran and white flour bran, respectively), in whole meal (mean processing factor of 1.5) and in wheat germ (mean processing factor of 1.1).

Figure A 14: Processing flowchart of wheat grain cleaning, drying and conditioning

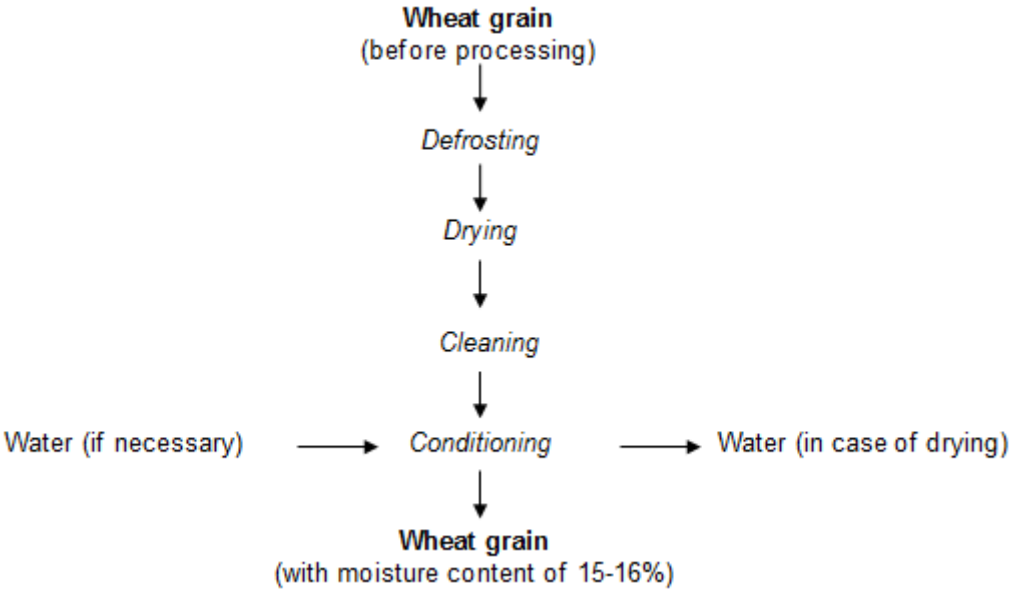


Figure A 15: Processing flowchart for milling of white flour (Type 550) and baking of white bread

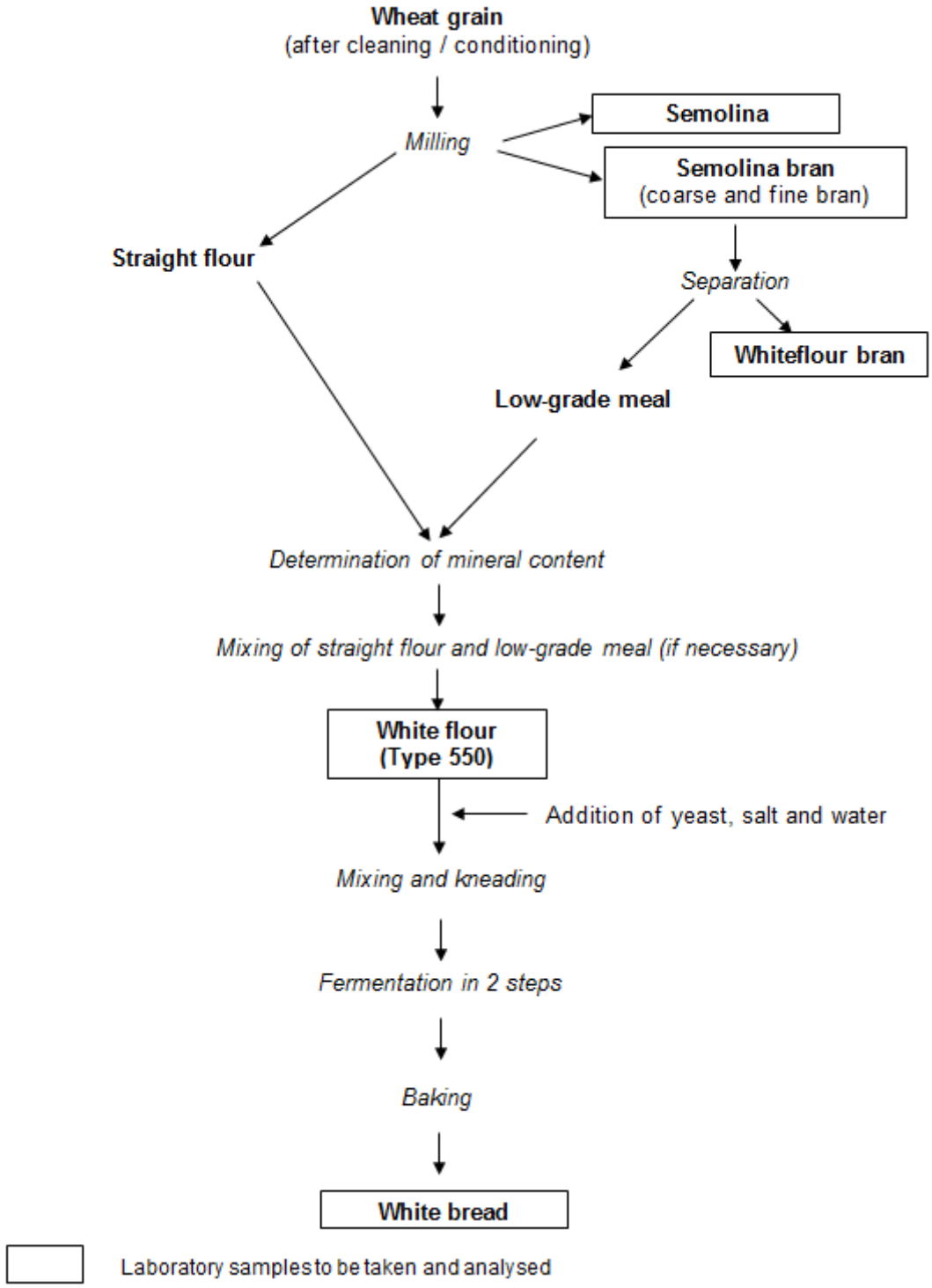


Figure A 16: Processing flowchart for milling of whole meal and baking of whole-meal bread

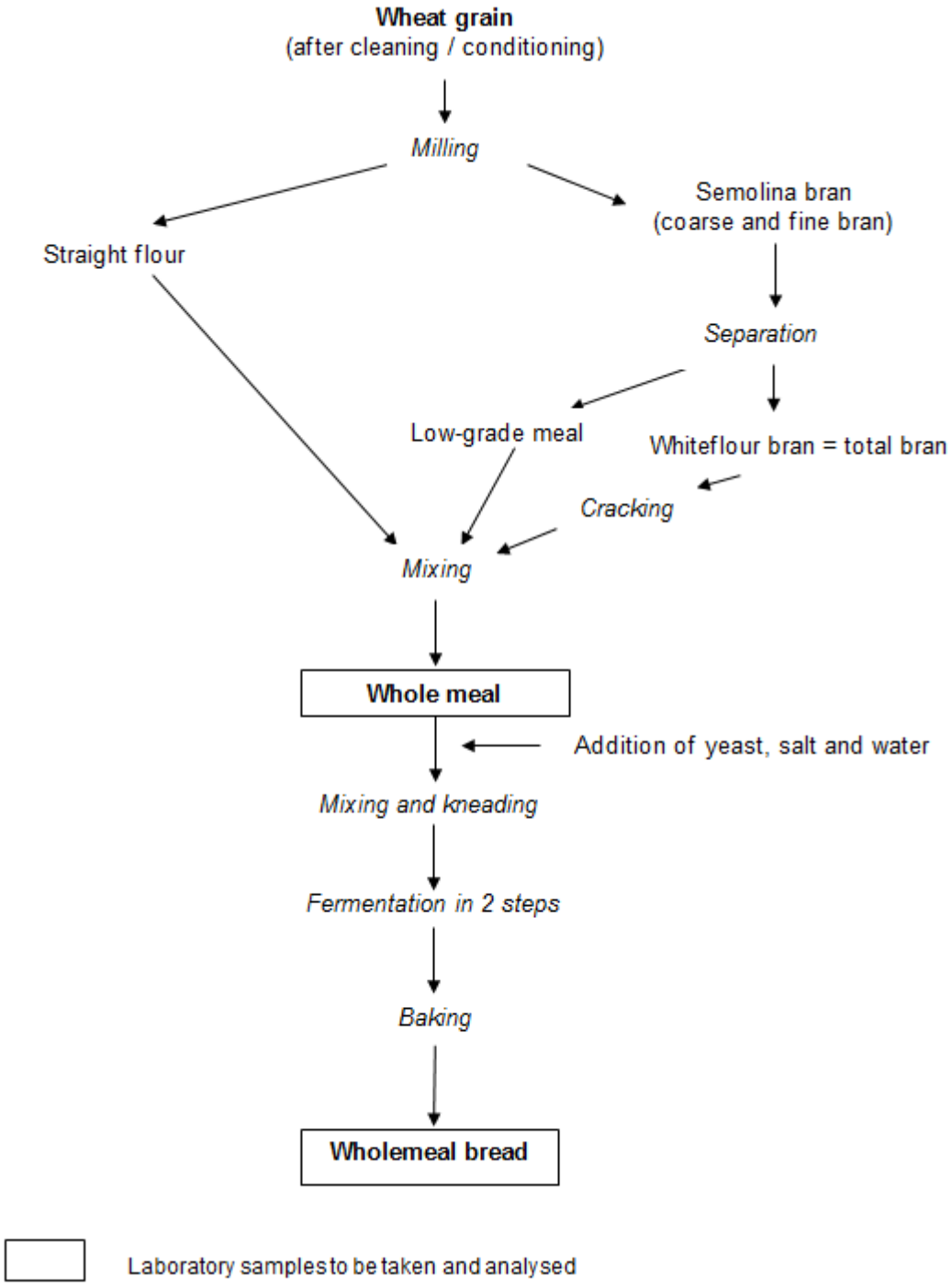
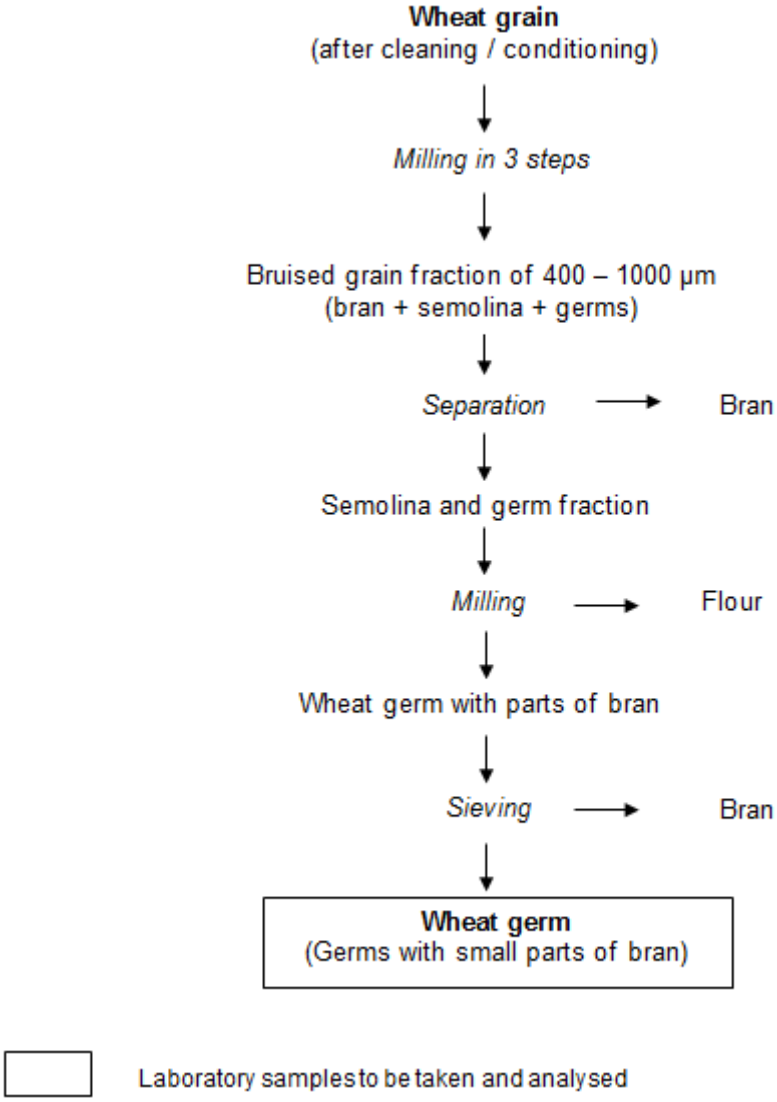


Figure A 17: Processing flowchart for production of wheat germ



A 2.1.5.2.7 Wheat , study report RARVY031

Comments of zRMS:	Two field trials were conducted to measure the magnitude of BYI 02960 residues in/on wheat processed commodities following two broadcast foliar spray applications of BYI 02960 200 SL at exaggerated rates of 1000 g a.s./ha. The results of the study showed that, BYI 02960 and DFA residues concentrate significantly in the aspirated grain fraction (mean processing factor of 17.6 for BYI 02960 and 6.9 for DFA) and in bran (mean processing factor 2.4 for BYI 02960 and 1.1 for DFA). A slight concentration of BYI 02960 was detected in germ (mean processing factor of 1.6 for BYI 02960 and 1.0 for DFA) and whole meal (mean processing factor of 1.2 for BYI 02960 and 1.0 for DFA). The average total BYI 02960 (sum of BYI 02960 and DFA) residue did not concentrate in most of the processed commodities (processing factor <1X): in cooking water, cooking water 2, gluten, middlings, pasta (cooked), pasta (dried and cooked), pasta (dry), pasta (fresh), shorts, starch, white bread, white flour, whole meal bread, and whole meal flour. However, the total BYI 02960 residue did concentrate in aspirated grain fractions (10X), bran (1.6X), and germ (1.3X) and slightly in whole meal (1.1X). The study is acceptable.

Reference:	**KCA 6.5.3/07**
Title:	BYI 02960 200 SL - Magnitude of the residue in/on wheat processed commodities
Report:	Lenz, C.; Fischer, D. R.; 2012; RARVY031; M-427047-01-2
Authority registration No:	
Guideline(s):	OPPTS 860.1520, Processed Food/Feed PMRA Ref.: DACO 7.4.5. Processed Food/Feed OECD Test Guideline 508: Residues in Processed Commodities (Adopted Oct. 3, 2008)
Deviations:	Weather data, pesticide history data, irrigation records, maintenance chemical applications, field sample weights, and cultural practices records were not collected under GLP.
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

Two field trials were conducted in the USA to measure the magnitude of BYI 02960 residues in/on wheat aspirated grain fractions and processed commodities following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. The additive, Dyne-Amic 0.25% v/v was used in all applications. BYI 02960 SL 200 was sprayed twice at an exaggerated application rate of approx. 1.0 kg a.s. /ha and a water volume of approx. 150-200 L/ha. Wheat grains to be processed were sampled 19-21 days after the last treatment (DALT 21 intended) at BBCH growth stage 75-83 (medium milk stage to early dough stage).

The maximum storage period of deep-frozen samples intended for the analysis BYI 02960 and its metabolites was 372 days (approx. 12 months), which is within the acceptable rage for the storage stability of the residues of flupyradifurone in high starch matrices.

Triplicate subsamples of wheat grain RAC were removed, and the remaining wheat grain was used to collect aspirated grain fractions and generate the processed commodities of bran, whole meal flour, white flour, middlings, shorts, germ, pasta (dried and cooked), white bread, whole meal bread, gluten, starch, pasta (cooked), pasta (fresh), pasta (dry), and cooking water.

The residues of BYI 02960 and DFA were analyzed by method RV-001-P10-02 (Ying Li; 2010; M-401023-01-2), also known as method 01304 using LC-MS/MS with stable isotopically labelled internal standards. The individual analyte residues (parent BYI 02960 and DFA, expressed in parent equivalents) were summed to give a total BYI 02960 residue. The limits of quantitation were 0.01 mg/kg for BYI 02960 and 0.05 mg/kg for DFA, expressed in parent compound equivalents, yielding a calculated total-residue LOQ of 0.06 mg/kg, in all matrices. Residue measurements below the analyte LOQ were summed into the total BYI 02960 residue value as the analyte LOQ value. Prior and parallel to the residue analysis, the method was validated by recovery experiments.

Processing procedures:
Aspirated grain fractions: For generation of aspirated grain fractions, the moisture content of the whole wheat was determined and if above 13%, the samples were dried in an oven at 43-57°C until the moisture content was 10-13%. All samples required drying. To generate aspirated grain fractions, the wheat samples were placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved in the system, aspiration was used to remove light impurities (grain dust). Samples were moved for 120 minutes. Grain dust samples were classified on a W. S. Tyler rotap using the following sieves: 2360 micron (8 mesh); 2000 micron (10 mesh); 1180 micron (16 mesh); 850 micron (20 mesh); and 425 micron (40 mesh). After classification of each sample, the material through the 2360 micron sieve was recombined to produce one aspired grain fraction.

Wheat processing: Following generation of aspirated grain fractions, the whole wheat was cleaned by aspiration and screening. Light material was removed from the whole grain using a Kice aspirator. After aspiration, samples were screened in a Hance Corporation screen cleaner to separate large and small foreign particles (screenings) from the wheat. The cleaned wheat was separated into three process lines: germ recovery; whole meal flour; and milling for bran, flour and shorts.

Germ recovery process: Cleaned wheat was moisture adjusted (tempered) to 16%. After tempering for 1-1.5 hours, the wheat was passed through a Glen Mills disc mill. Ground material was sifted with a Sweco Dynascreen equipped with a 8 and 14 mesh sieve and a Great Western sample sifter with a 34 mesh sieve. Material on top of the 34 mesh sieve was aspirated to remove bran from the germ fraction. Germ (with endosperm) was passed through a Chopin CD-1 reduction mill. Germ and reduced endosperm were sifted to separate the germ from the endosperm. Germ material was also aspirated again to remove additional bran and milled/sieved to remove additional endosperm. A 0.5-1.0% recovery of germ was expected.

Milling process: Cleaned wheat was moisture conditioned depending on the physical property of the wheat. This particular wheat was determined to be a floury structure and was moisture conditioned to 16.5% and allowed to temper for 24 hours (±30 minutes). Tempered wheat was fed through the break side of a Chopin CD1 wheat mill. Breaking of the wheat was accomplished by three rolls. After passing through the break rolls, the material was fed onto the break sifter screen. This screen consists of two sizes. Screens sizes are 120 mesh (140 micron) and 25 mesh (800 micron). Material exiting the break rolls passed over the 120 first. Material passing through the 120 screen is "Break Flour." Material not passing through was conveyed over the 25 screen. Material passing through the 25 screen is middlings. Material not passing through was conveyed to the end of the sifter is bran. After breaking, it is necessary to determine if more than one reduction will be required. If the "Middling" fraction is 48% or less of the starting weight, then one reduction is required. If more than 48% is obtained, two reductions are required. All samples required one reduction. After sampling the middlings, the remainder was fed into the reduction side of the mill. Reduction was achieved through two rolls. After passing through the reduction rolls, the material was fed onto the reduction sifter screen. This screen is a 100 mesh (160 micron). Material passing through the screen is reduction flour. Material not passing through and conveyed to the end of the sifter is shorts. Bran from the break sieve was fed into the reductions side screen of the mill. It was notreduced with the rollers. Bran was conveyed by beater bars over the 100 mesh screen. Material passing through the screen was shorts and was added to shorts produced from the middlings. Material passing over the screen and exiting the end was bran. A portion of the break and reduction flours were

mixed to white flour (straight flour). This flour was used to produce white bread. Remaining reduction flour and break flour were used to produce starch, gluten and pasta.

Processing into white bread: Straight flour, sugar, dry milk, salt, margarine, water and dry yeast were placed in a Sunbeam bread machine. The machine was set for type bread (Basic), and medium color. The machine automatically mixed the ingredients, let the dough rise and baked the bread.

Processing into whole meal flour and whole meal bread: Cleaned wheat was ground in an Alpine 160Z pin mill. Ground material was whole meal flour. Whole meal flour, brown sugar, salt, margarine, water and dry yeast were placed in a Sunbeam bread machine. The machine automatically mixed the ingredients, let the dough rise and baked the bread.

Gluten and starch production: For gluten and starch, break flour was mixed with water at a rate of 0.18-0.36 kg of water per 0.45 kg of flour. After mixing, dough was allowed to rest for two hours. After resting, the dough was kneaded as water washed away the starch, leaving the gluten. Product was water washed until water ran clear, indicating all starch was removed, leaving gluten. Starch and water were separated using centrifugation to separate out the water. Starch was dried in an oven at 43-50°C (maximum recorded 59°C) until the moisture content was 15% or less. Dried starch was then ground and collected. Two method were used for drying gluten. Samples from RV272-10PA were dried at 43-50°C in an oven. Maximum recorded temperature using this method was 53°C. Dried gluten was ground and collected. Samples from RV273-10PA were dried utilizing a steam heated drum dryer with a gap setting of 0.1778-0.254 mm. As gluten passed through the rollers, it was dried into a thin sheet of gluten. Temperature is not monitored during use of the drum dryer. Dried gluten was ground and collected.

Pasta production: Equal parts of break and reduction flour were mixed with water and salt to form a dough. This dough was kneaded and allowed to rest for 20-40 minutes. Dough was fed into a Lello pasta machine to produce a fresh Asian noodle. A portion of the fresh noodle was removed and collected. A portion of the fresh noodles were cooked in boiling water for 1-4 minutes, allowed to cool. After cooling, a fresh cooked noodle fraction was collected.
Remaining extruded noodles were dried for eight hours at 24-35°C (maximum recorded temperature was 36.5°C) to produce dried noodles. A portion of the dried noodles were removed and collected. Remaining dried noodles were then cooked in boiling water for 1-4 minutes, removed, and allowed to cool. After cooling, a fraction of dried cooked noodles was collected. Fractions of the cooking water were collected from both fresh and dried

Results and discussions
Concurrent recoveries of BYI 02960 and DFA were measured with each set of samples to verify method performance. All recoveries were corrected for any interference in corresponding controls. The overall mean of the recoveries for each matrix ranged between 74 to 112%, and the standard deviation values were below 20%.

The residue values and processing factors of BYI 02960 and DFA, as well as calculated total BYI 02960 residue data (sum of parent compound BYI 02960 and DFA, expressed as BYI 02960 equivalents) for the wheat RAC and the processed commodities are provided in Table A 37.
noodles.

Table A 36: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in wheat grain and processed matrices

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
RARVY031 RV272-10PA and RV273-10PA GLP: yes 2010	Wheat	grain	BYI 02960	7	0.01	116; 74; 112; 90; 102; 77; 80	74	116	93	18.4
				3	2.5	90; 99; 105	90	105	98	7.7
				10	overall		74	116	95	15.5
			DFA	7	0.05	80; 78; 79; 89; 89; 92; 81	78	92	84	6.9
				3	2.5	79; 87; 87	79	87	84	5.5
				10	overall		78	92	84	6.2
		aspirated grain fractions	BYI 02960	3	0.2	103; 101; 93	93	103	99	5.3
				3	40	100; 104; 95	95	104	100	4.5
				6	overall		93	104	99	4.4
			DFA	3	0.2	100; 110; 109	100	110	106	5.2
				3	40	104; 99; 96	96	104	100	4.1
				6	overall		96	110	103	5.5
		bran	BYI 02960	3	0.01	89; 116; 84	84	116	96	17.9
				3	4	99; 102; 108	99	108	103	4.4
				6	overall		84	116	100	11.9
			DFA	3	0.05	97; 94; 85	85	97	92	6.8
				3	4	87; 85; 83	83	87	85	2.4
				6	overall		83	97	89	6.4
		cooking water	BYI 02960	3	0.01	97; 98; 106	97	106	100	4.9
				3	0.2	93; 97; 91	91	97	94	3.3
				6	overall		91	106	97	5.3
			DFA	3	0.05	96; 98; 95	95	98	96	1.6
				3	0.2	99; 101; 96	96	101	99	2.6
				6	overall		95	101	98	2.3
		germ	BYI 02960	3	0.01	95; 114; 80	80	114	96	17.7
				3	4	98; 99; 114	98	114	104	8.6
				6	overall		80	114	100	12.8
			DFA	3	0.05	79; 77; 75	75	79	77	2.6
				3	4	71; 76; 77	71	77	75	4.3
				6	overall		71	79	76	3.6
		gluten	BYI 02960	3	0.01	115;116;106	106	116	112	4.9
				3	1	109;92;96	92	109	99	9.0
				3	4	99;93;96	93	99	96	3.1
				9	overall		92	116	102	9.1
			DFA	3	0.05	88; 91; 118	88	118	99	16.7
				3	1	88; 101; 104	88	104	98	8.7
				3	4	101; 102; 101	101	102	101	0.6
				9	overall		88	118	99	9.5
		pasta, fresh	BYI 02960	4	0.01	97; 71; 81; 106	71	106	89	17.7
				3	4	111; 106; 100	100	111	106	5.2
				7	overall		71	111	96	15.3
			DFA	4	0.05	94; 96; 94; 98	94	98	96	2.0
				3	4	95; 105; 100	95	105	100	5.0
				7	overall		94	105	97	4.1
		shorts	BYI 02960	3	0.01	87; 91; 96	87	96	91	4.9
				3	4	98; 118; 106	98	118	107	9.4
				6	overall		87	118	99	11.3
			DFA	3	0.05	87; 83; 85	83	87	85	2.4
				3	4	85; 87; 89	85	89	87	2.3
				6	overall		83	89	86	2.4
		starch	BYI 02960	3	0.01	101; 95; 101	95	101	99	3.5
				3	0.2	94; 81; 112	81	112	96	16.3
				6	overall		81	112	97	10.5
			DFA	3	0.05	90; 86; 88	86	90	88	2.3

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
				3 6	0.2 overall	79; 82; 90	79 79	90 90	84 86	6.8 5.2
		white bread	BYI 02960	2 2	0.01 overall	110; 105	105 105	110 110	108 108	
			DFA	2 2	0.05 overall	89; 92	89 89	92 92	91 91	
		white flour	BYI 02960	3 3 6	0.01 4 overall	102; 98; 111 105; 118; 113	98 105 98	111 118 118	104 112 108	6.4 5.9 6.9
			DFA	3 3 6	0.05 4 overall	88; 89; 99 71; 76; 76	88 71 71	99 76 99	92 74 83	6.6 3.9 12.7
		whole meal	BYI 02960	2 2	0.01 overall	103; 117	103 103	117 117	110 110	
			DFA	2 2	0.05 overall	73; 87	73 73	87 87	80 80	
		wholemeal bread	BYI 02960	2 3 5	0.01 4 overall	103; 106 85; 88; 91	103 85 85	106 91 106	105 88 95	 3.4 9.9
			DFA	2 3 5	0.05 4 overall	88; 94 83; 83; 82	88 82 82	94 83 94	91 83 86	 0.7 5.9

Table A 37: Residue data from wheat grain processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)									
Wheat grain	1.29	1.66	21/19	aspirated grain fractions	27.7	22.9	21.5	13.8	Median: 17.6
				bran	3.00	3.95	2.3	2.4	Median: 2.4
				cooking water	0.01	<0.01	<0.1	<0.1	Median: <0.1
				cooking water 2	0.01	0.01	<0.1	<0.1	Median: <0.1
				germ	2.13	2.73	1.7	1.6	Median: 1.6
				gluten	**0.66**	**0.36**	**0.5**	**0.2**	**Median: 0.4**
				middlings	**0.78**	**1.32**	**0.6**	**0.8**	**Median: 0.7**
				pasta, cooked	0.11	0.10	<0.1	<0.1	Median: <0.1
				pasta, dried and cooked	0.12	0.14	<0.1	<0.1	Median: <0.1
				pasta, dry	0.42	0.40	0.3	0.2	Median: 0.3
				pasta, fresh	0.30	0.301	0.2	0.2	Median: 0.2
				shorts	0.79	2.05	0.6	1.2	Median: 0.2
				starch	<0.01	0.02	<0.1	<0.1	Median: <0.1
				white bread	0.29	0.24	0.2	0.1	Median: 0.2
				white flour	0.42	0.36	0.3	0.2	Median: 0.3
				whole meal	1.45	2.10	1.1	1.3	Median: 1.2
				whole meal bread	0.84	1.25	0.7	0.8	Median: 0.7
DFA, expressed as DFA (RD-Mo)									
Wheat grain	2.93	2.18	21/19	aspirated grain fractions	14.7	19.1	5.0	8.8	Median: 6.9
				bran	3.21	2.20	1.1	1.0	Median: 1.1
				cooking water	0.14	0.08	<0.1	<0.1	Median: <0.1
				cooking water 2	0.12	0.10	<0.1	<0.1	Median: <0.1
				germ	2.86	2.17	1.0	1.0	Median: 1.0
				gluten	**1.95**	**0.40**	**0.7**	**0.2**	**Median: 0.4**
				middlings	**2.73**	**1.91**	**0.9**	**0.9**	**Median: 0.9**
				pasta, cooked	0.56	0.37	0.2	0.2	Median: 0.2
				pasta, dried and cooked	0.66	0.53	0.2	0.2	Median: 0.2
				pasta, dry	2.69	1.84	0.9	0.8	Median: 0.9
				pasta, fresh	2.23	1.43	0.8	0.7	Median: 0.7
				shorts	2.63	2.08	0.9	1.0	Median: 0.9
				starch	0.07	0.11	<0.1	<0.1	Median: <0.1
				white bread	1.94	1.15	0.7	0.5	Median: 0.6
				white flour	2.46	1.58	0.8	0.7	Median: 0.8
				whole meal	3.26	2.04	1.1	0.9	Median: 1.0
				whole meal bread	1.97	1.45	0.7	0.7	Median: 0.7
Sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)									

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
Wheat grain	4.22	3.84	21/19	aspirated grain fractions	42.40	42.00	10.0	10.9	Median: 10.5
				bran	6.21	6.15	1.5	1.6	Median: 1.5
				cooking water	0.16	0.09	<0.1	<0.1	Median: <0.1
				cooking water 2	0.13	0.11	<0.1	<0.1	Median: <0.1
				germ	4.99	4.90	1.2	1.3	Median: 1.2
				gluten	**2.61**	**0.76**	**0.6**	**0.2**	**Median: 0.4**
				middlings	**3.51**	**3.23**	**0.8**	**0.8**	**Median: 0.8**
				pasta, cooked	0.67	0.47	0.2	0.1	Median: 0.1
				pasta, dried and cooked	0.78	0.67	0.2	0.2	Median: 0.2
				pasta, dry	3.11	2.24	0.7	0.6	Median: 0.7
				pasta, fresh	2.53	1.74	0.6	0.5	Median: 0.5
				shorts	3.42	4.13	0.8	1.1	Median: 0.9
				starch	0.08	0.13	<0.1	<0.1	Median: <0.1
				white bread	2.23	1.39	0.5	0.4	Median: 0.4
				white flour	2.88	1.94	0.7	0.5	Median: 0.6
				whole meal	4.71	4.14	1.1	1.1	Median: 1.1
				whole meal bread	2.81	2.70	0.7	0.7	Median: 0.7

* PF = Processing Factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Conclusion

Following application of BYI 02960 SL 200 to two separate trials of wheat, BYI 02960 and DFA residues concentrate significantly in the aspirated grain fraction. A transfer of residues was also detected in bran. A slight concentration of BYI 02960 was detected in germ and whole meal. The DFA residues in these processed commodities remained at the same level.

Overall, flupyradifurone related residues did not concentrate in most of the processed commodities – especially in commodities for human consumption (like bread and pasta) even dilution of residues was observed. The results of the US processing studies are in very good agreement with the results of the EU processing studies and the processing factors of corresponding commodities can be combined.

Figure A 18: Processing flowchart for milling process of wheat

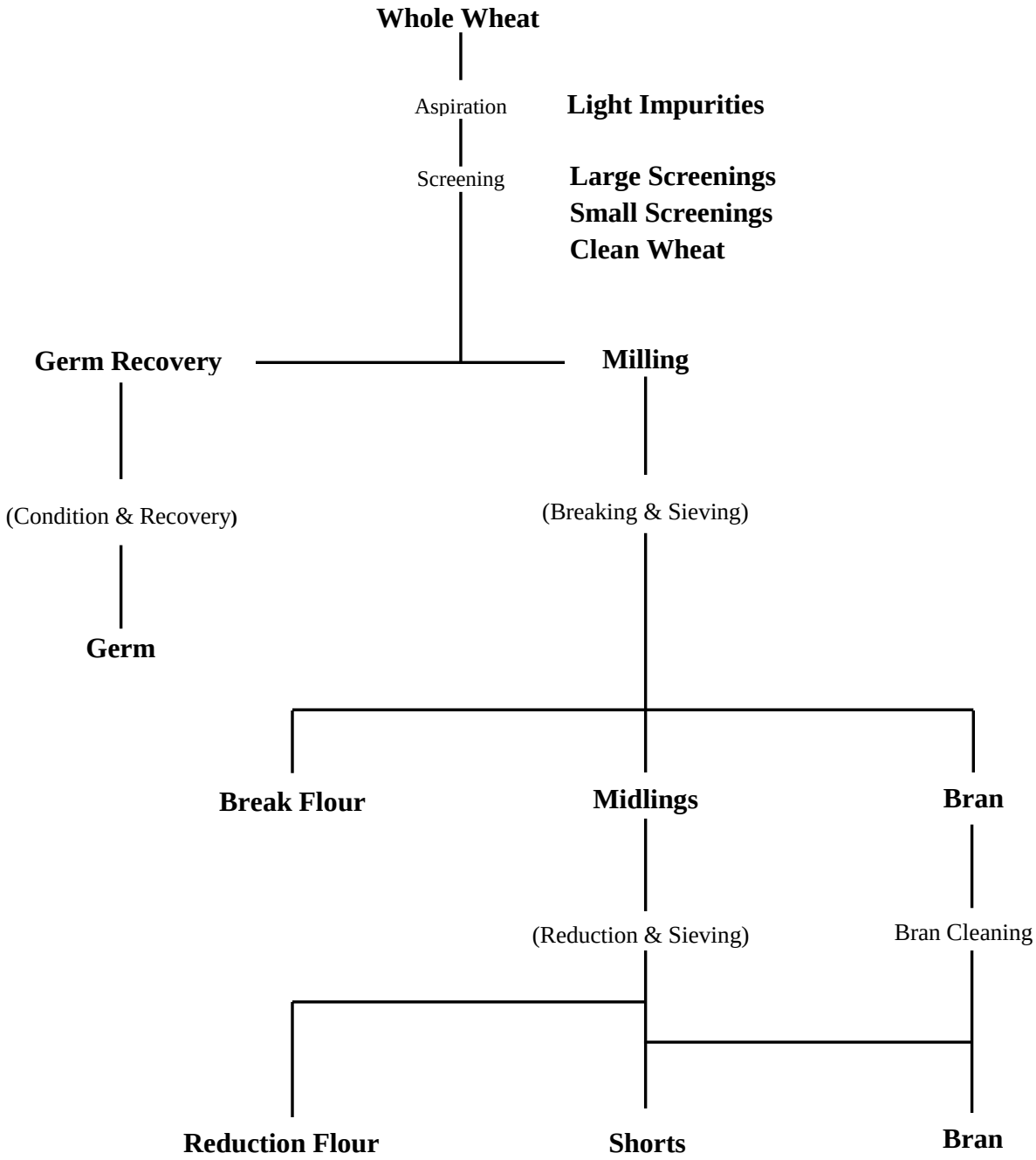


Figure A 19: Processing flowchart for production of gluten and starch

Starch and Gluten Production

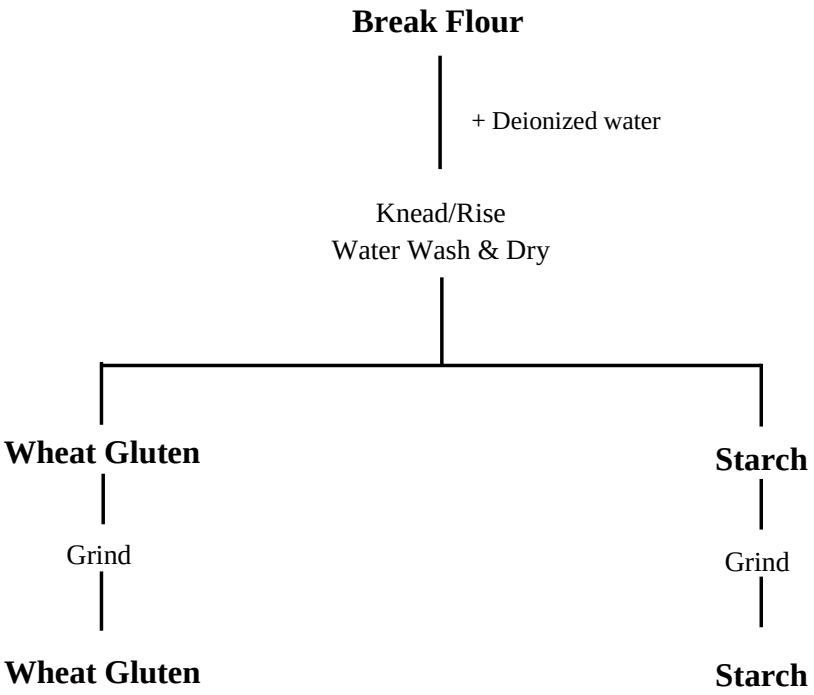
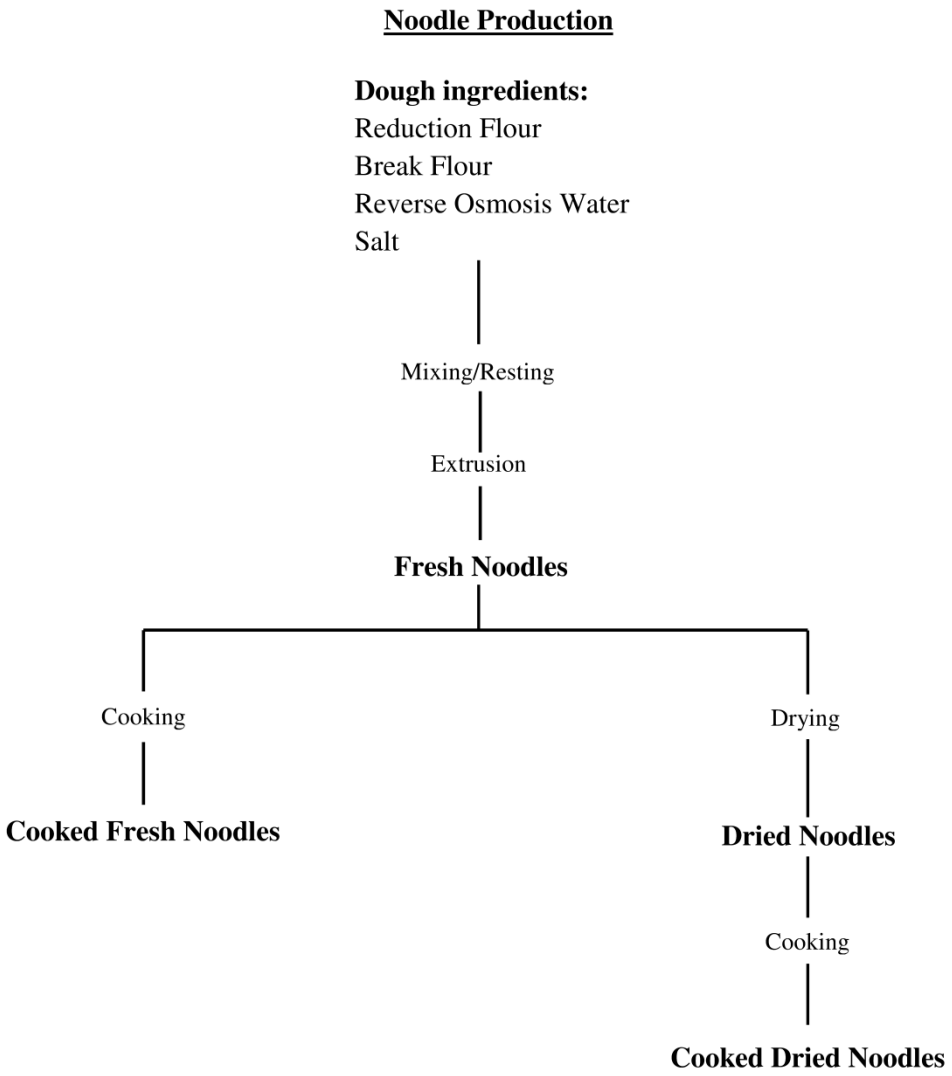


Figure A 20: Processing flowchart for production of pasta



A 2.1.5.2.8 Corn, study report RARVY030

Comments of zRMS:	Two field trials were conducted to measure the magnitude of BYI 02960 residues in/on field corn processed commodities (aspirated grain fractions and starch, refined oil, steep water, and germ from wet milling and grits, flour, meal, refined oil, bran and germ from dry milling) following two broadcast foliar spray applications of BYI 02960 200 SL at exaggerated rates of 1000 g a.s./ha. The residues of BYI 02960 did not concentrate in the processed commodities of flour, germ (dry or wet milled), grits, meal (dry milled), oil (dry or wet milled), starch and steep water (processing factor ≤1X). However, the BYI 02960 residues did concentrate significantly in the aspirated grain fraction (mean processing factor of 23), and slightly in bran (mean processing factor of 2.8). Due to the fact that residues of DFA at or above 0.05 mg/kg were neither detected in field corn grain (RAC) nor in any of the processed commodities, it is no concentration of DFA residues expected during processing. The average total BYI 02960 (sum of BYI 02960 and DFA) residue did not concentrate (processing factor ≤1X) in silage, and the processed commodities of flour, germ (dry milling), germ (wet milling), grits, meal (dry milled), oil (dry milled), oil (wet milled), starch, and steep water. However, the total BYI 02960 residue did concentrate in aspirated grain fractions (mean processing factor of 6.6) and bran (mean processing factor of 1.5). The study is acceptable.

Reference:	**KCA 6.5.3/08**
Title:	BYI 02960 200 SL - Magnitude of the residue in/on field corn processed commodities
Report:	Lenz, C.; 2012; RARVY030; M-424774-01-1
Authority registration No:	
Guideline(s):	OPPTS 860.1520, Processed Food/Feed PMRA Ref.: DACO 7.4.5, Processed Food/Feed OECD Test Guideline 508: Residues in Processed Commodities (adopted Oct., 3 2008)
Deviations:	weather data, pesticide history data, irragation records, maintenance chemical applications, field sample weights, cultural practices records and soil characterization information were not collected under GLP.
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

Two field trials were conducted in the USA to measure the magnitude of BYI 02960 residues in/on field corn aspirated grain fractions and processed commodities following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. The additive, Dyne-Amic 0.25% v/v was used in all applications. BYI 02960 SL 200 was sprayed twice at an exaggerated application rate ranging from 603 to 1000 g a.s. /ha and a water volume of approx. 300 L/ha. The first applications for the 3F (forage) plots were made between BBCH growth stages 79 to 89. The interval between applications was 5 to 8 days. Field corn grains (RAC) to be processed were sampled 21 days after the last treatment at maturity.

Triplicate subsamples of the field corn grain RAC were removed, and the remaining field corn grain was used to collect aspirated grain fractions and generate the processed commodities of starch, refined oil, steep water, and germ from wet milling and grits, flour, meal, refined oil, bran and germ from dry milling.

The maximum storage period of deep-frozen samples intended for the analysis BYI 02960 and its metabolites was 218 days (approx. 7 months) for maize/field corn grain and its processed fractions, which is within the acceptable range for the storage stability of the residues of flupyradifurone in high starch matrices. The maximum storage period for raw commodities which are not relevant for processing was

detected in forage and was 272 days, which is also within the acceptable range for the storage stability of the residues of flupyradifurone in matrices with high water content.

The residues of BYI 02960 and DFA were analyzed using method RV-001-P10-02 (Ying Li; 2010; M-401023-01-2) with some modifactions, also known as method 01304. The individual residues of parent BYI 02960 and DFA were determined and a total BYI 02960 residue was calculated (sum of BYI 02960 and DFA). The limits of quantitation were 0.01 mg/kg for BYI 02960 and 0.05 mg/kg for DFA, expressed in parent compound equivalents, yielding a calculated total-residue LOQ of 0.06 mg/kg, in all matrices. Residue measurements below the analyte LOQ were summed into the total BYI 02960 residue value as the analyte LOQ value. Prior and parallel to the residue analysis, the method was validated by recovery experiments.

Processing procedures:
Ensilage: A representative sub-sample of the forage was weighed and fed into a chipper/shredder. Multiple passes through the machine was permissible. A representative sample of the final chopped material was collected for pH determination and percent dry matter. Chopped material was placed in a plastic container ("micro-silo") and packed several times utilizing a 45 kg weight. After filling, the container was sealed. The lid had a one-way valve and inlet for a thermocouple. A thermocouple was inserted in the chopped material to monitor the minimum and maximum temperatures for the duration of the ensiling procedure. Containers were placed in a temperature controlled room. A chart recorder monitored the room temperature. After 21 days, the lid was removed and ensiled samples are collected by collecting representative core samples from the container. A portion of the sample was used for pH and dry matter determination. pH of the ensiled sample should be 5.0 or less. Ensiled samples are placed into frozen storage.

Aspirated grain fractions: Samples of maize corn were dried (if necessary) to a moisture content of 10-13%. The whole corn samples were placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved in the system, aspiration was used to remove light impurities (grain dust). Samples were moved for 120 minutes. Light impurities were classified using the following sieves: 2360 micron (8 mesh); 2000 micron (10 mesh); 1180 micron (16 mesh); 850 micron (20 mesh); and 425 micron (40 mesh). After classification of each sample, the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF).

Dry milling process: Representative samples of cleaned whole corn were moisture conditioned to 21.0% and tempered for approximately 2 hours. After tempering, the samples were fed into a disc mill to crack the kernel. Corn stock from the mill was dried in an oven for 30 minutes at 54-71°C. Dried cornstock was screened on a cleaner fitted with a 1/8" screen to separate bran, germ, and large grits from grits, meal and flour. Material below 3.175 mm (grits, meal, and flour) was separated using the following two screens: 14 mesh (~1.37 mm) and 62 mesh (~0.25 mm sieves. The fraction on top of 14 mesh sieve was "grits"; the fraction on top of the 62 mesh sieve was “meal” and the fration through the 62 mesh sieve was “flour”.

Material on top of the 3.175 mm screen (bran, germ, and large grits) was screened again using a 5.2 mm screen (or similar size). Material above the screen was aspirated using an aspirator to remove hull from germ with attached hull and endosperm. Germ with attached hull and endosperm was passed through the disc mill and screened.

All material through the 5.2 mm screen (large grits and detached germ) was passed over a separator to separate germ and large grits. If necessary, germ may be milled, screened, and aspirated to remove endosperm. The two germ fractions are combined and dried at 54-71°C to a final moisture of 14.0-16.0%. Grits, meal, flour, bran, and germ samples were collected and placed into freezer storage.

Germ material was heated to 71-79°C in a mixer and held for 10 minutes. Heated material was flaked in a flaking roll with a gap setting of 0.18-0.025 mm. After flaking, germ from trial RV271-10PA was screened to remove additional endosperm not separated during milling. Flaked germ material was placed in stainless steel batch extractors and submerged in 48-60°C solvent (hexane). After 30 minutes, the

miscella (crude oil and hexane) was drained and fresh hexane was added to repeat the cycle two more times. Final two washings were for 15 minutes each. Following the final draining, the spent flakes were desolventized with ambient air to remove residual hexane. Resulting fractions from solvent extraction were miscella (crude oil and hexane) and solvent extracted germ meal. Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was then heated to 91-96°C for hexane removal, filtered, and collected for refining. A weighed amount of crude oil and 16 degree Baume sodium hydroxide was placed in a water bath and mixed for 15 minutes at high RPM at 20-24°C and then for 12 minutes at low RPM at 63-67°C. The neutralized oil was then centrifuged. The refined oil was decanted and filtered. The refined oil samples were collected and placed in freezer storage. Soapstock was discarded. During bleaching, refined oil was heated to 40-50°C, activated bleaching earth added (1.0% by weight of oil), and placed under vacuum. Temperature was increased to 85-100°C and held for 10 to 15 minutes. After reducing the temperature, the bleached oil was filtered. A filter aid was utilized during filtering of bleached oil from trial RV270-10PA (dry and wet mill processes). Resulting fractions were bleached oil and spent bleaching earth. For deodorization, bleached oil was steam bathed for 28-32 minutes under vacuum and temperature held between 220-230°C. During the cooling period a 0.5% citric acid solution was added (1 ml per 100 grams of oil deodorized). Resulting fractions were refined-bleached-deodorized oil (RBD oil) and deodorizer distillates. Deodorized oil was collected and placed into frozen storage.

Wet milling process:A representative sample of dried and cleaned corn was steeped in 49-54°C water containing 0.1-0.2% sulfur dioxide (sulfurous acid) for 22-48 hours. At the end of the steeping period, the whole corn was passed through a bell disc mill and a majority of the germ and hull was removed using a water centrifuge. Germ and hull were dried at 74-91°C to obtain a final moisture between 5-10%. After drying, the germ and hull were separated using aspiration and screening. Requested germ fractions were collected and placed into frozen storage. Cornstock (without germ and hull) ground in the disc mill was passed over a Dynascreen equipped with a 325 mesh (50 micron) screen. In commercial industry, only bran (hull material) remains on top of the screen. Material on top of the screen was discarded. Process water (with starch and gluten) passing through the screen was separated into starch and gluten using batch centrifugation. Starch was dried in a dehydrator oven at 54-71°C until the moisture content was less than 15.0%. Requested starch samples were collected and placed into freezer storage. Germ samples were moisture conditioned to 12%, heated to 88-104°C in a mixer, flaked in a flaking roll with a gap setting of 0.20 - 0.30 mm, and pressed in a expeller to liberate part of the crude oil. Resulting fractions are expelled crude oil and presscake with residual crude oil. Presscake was placed in stainless steel batch extractors and submerged in 49-60°C hexane. After 30 minutes, the miscella was drained and fresh hexane was added to repeat the cycle two more times. Final two washings were for 15 minutes each. Following the final draining, the spent presscake was desolventized with ambient air to remove residual hexane. Resulting fractions from solvent extraction were miscella and solvent extracted presscake (germ cake). Miscella (crude oil and hexane) was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was then heated to 91-96°C for hexane removal. Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was then heated to 91-96°C for hexane removal. Crude oils from expelling and solvent extraction were filtered and combined for refining. Crude oil samples from the wet milling process were refined, bleached, and deodorized with the same methods used for during the dry milling procedure resulting in refined-bleached-deodorized oil (RBD oil).

Results and discussions
Concurrent recoveries of BYI 02960 and DFA were measured with each set of samples to verify method performance. All recoveries were corrected for any interferences in corresponding controls. The overall mean of the recoveries for all matrices ranged between 72 to 107%, and the standard deviation values were below 20%.

The residue values and processing factors of BYI 02960 and DFA, as well as calculated total BYI 02960 residue data (sum of parent compound BYI 02960 and DFA, expressed as BYI 02960 equivalents) for the field corn grain RAC and the processed commodities are provided in Table A 39.

Table A 38: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in maize/field corn and processed matrices

Study Trial No. / GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
RARVY030 RV270-10PA and RV271-10PA (RV270-10PA-B) and (RV271-10PA-B) GLP: yes 2010	maize/ corn	grain	BYI 02960	7	0.01	91; 104; 93; 94; 112; 89; 96	89	112	97	8.4
				3	1	96; 90; 86	86	96	91	5.6
				10	overall		86	112	95	8.1
			DFA	7	0.05	81; 82; 80; 85; 103; 103; 94	80	103	90	11.4
				3	1	89; 80; 84	80	89	84	5.3
				10	overall		80	103	88	10.2
		aspirated grain fractions	BYI 02960	4	0.01	83; 98; 101; 89	83	101	93	8.9
				3	1	101; 118; 102	101	118	107	8.9
				7	overall		83	118	99	11.2
			DFA	4	0.05	80; 90; 95; 82	80	95	87	8.1
				3	1	91; 104; 101	91	104	99	6.9
				7	overall		80	104	92	9.8
		bran	BYI 02960	3	0.01	94; 107; 108	94	108	103	7.6
				3	1	83; 84; 94	83	94	87	7.0
				6	overall		83	108	95	11.3
			DFA	3	0.05	86; 84; 86	84	86	85	1.4
				3	1	85; 79; 86	79	86	83	4.5
				6	overall		79	86	84	3.2
		flour	BYI 02960	3	0.01	105; 81; 89	81	105	92	13.3
				3	overall		81	105	92	13.3
			DFA	3	0.05	78; 76; 74	74	78	76	2.6
				3	overall		74	78	76	2.6
		germ, dry milling	BYI 02960	3	0.01	90; 97; 99	90	99	95	5.0
				3	overall		90	99	95	5.0
			DFA	3	0.05	74; 86; 86	74	86	82	8.4
				3	overall		74	86	82	8.4
		germ, wet milling	BYI 02960	3	0.01	93; 84; 93	84	93	90	5.8
				3	1.0	83; 80; 91	80	91	85	6.7
				6	overall		80	93	87	6.5
			DFA	3	0.05	76; 70; 71	70	76	72	4.4
				3	1.0	71; 74; 70	70	74	72	2.9
				6	overall		70	76	72	3.4
		maize/ corn grits	BYI 02960	3	0.01	74; 98; 95	74	98	89	14.7
				3	overall		74	98	89	14.7
			DFA	3	0.05	80; 82; 80	80	82	81	1.4
				3	overall		80	82	81	1.4
		meal, dry milled	BYI 02960	3	0.01	76; 75; 97	75	97	83	15.0
				3	1.0	97; 97; 93	93	97	96	2.4
				6	overall		75	97	89	12.0
			DFA	3	0.05	72; 72; 78	72	78	74	4.7
				3	1.0	84; 85; 91	84	91	87	4.4
				6	overall		72	91	80	9.5
		oil, dry milled	BYI 02960	3	0.01	100; 76; 112	76	112	96	19.1
				3	1.0	104; 102; 103	102	104	103	1.0
				6	overall		76	112	100	12.3
			DFA	3	0.05	97; 79; 108	79	108	95	15.5
				3	1.0	105; 104; 105	104	105	105	0.6
				6	overall		79	108	100	10.8
		maize/ corn oil, wet milled	BYI 02960	3	0.01	85; 80; 95	80	95	87	8.8
				3	1.0	110; 106; 103	103	110	106	3.3
				6	overall		80	110	97	12.4
			DFA	3	0.05	83; 82; 89	82	89	85	4.5
				3	1.0	95; 105; 103	95	105	101	5.2
				6	overall		82	105	93	10.6

Study Trial No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
		silage	BYI 02960	3	0.01	109; 89; 110	89	110	103	11.5
				3	2.0	93; 100; 99	93	100	97	3.9
				3	10	94; 92; 103	92	103	96	6.1
				9	overall		89	110	99	7.6
			DFA	3	0.05	82; 98; 82	82	98	87	10.6
				3	2.0	100; 99; 99	99	100	99	0.6
				3	10	87; 89; 90	87	90	89	1.7
				9	overall		82	100	92	8.0
		forage	BYI 02960	3	0.01	99; 100; 90	90	100	96	5.7
				3	2.0	87; 95; 85	85	95	89	5.9
				3	15	89; 91; 85	85	91	88	3.5
				9	overall		85	100	91	6.2
			DFA	3	0.05	94; 113; 108	94	113	105	9.4
				3	2.0	88; 96; 94	88	96	93	4.5
				3	15	89; 90; 86	86	90	88	2.4
				9	overall		86	113	95	9.7
		starch	BYI 02960	3	0.01	90; 90; 105	90	105	95	9.1
				3	1.0	92; 95; 99	92	99	95	3.7
				6	overall		90	105	95	6.2
			DFA	3	0.05	105; 79; 81	79	105	88	16.4
				3	1.0	82; 85; 79	79	85	82	3.7
				6	overall		79	105	85	11.7
		steep water	BYI 02960	3	0.01	89; 98; 100	89	100	96	6.1
				3	1.0	103; 101; 106	101	106	103	2.4
				6	overall		89	106	100	5.9
			DFA	3	0.05	105; 96; 89	89	105	97	8.3
				3	1.0	105; 103; 105	103	105	104	1.1
				6	overall		89	105	101	6.6

Table A 39: Residue data from maize/field corn/ grain processing study with flupyradifurone

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
Flupyradifurone, expressed as flupyradifurone (RD-Mo)									
Maize/field corn grain	0.012	0.024	21	Aspirated grain fractions	0.31	0.50	25.4	20.8	Median: 23.1
				Bran	0.02	0.09	1.8	3.8	Median: 2.8
				Flour	**<0.01**	**<0.01**	**<0.8**	**<0.4**	Median: **<0.6**
				Germ, dry milling	<0.01	0.03	<0.8	1.2	Median: <1.0
				Germ, wet milling	<0.01	0.01	<0.8	0.5	Median: <0.7
				Grits	**<0.01**	**<0.01**	**<0.8**	**<0.4**	Median: **<0.6**
				Meal, dry milled	**<0.01**	**<0.01**	**<0.8**	**<0.4**	Median: **<0.6**
				Oil, dry milled	<0.01	<0.01	<0.8	<0.4	Median: <0.6
				Oil, wet milled	<0.01	<0.01	<0.8	<0.4	Median: <0.6
				Starch	<0.01	<0.01	<0.8	<0.4	Median: <0.6
				Steep water	<0.01	0.01	<0.8	0.5	Median: <0.7
DFA, expressed as DFA (RD-Mo)									
Maize/field corn grain	<0.05	<0.05	21	Aspirated grain fractions	<0.05	<0.05	n.c.	n.c.	n.c.
				Bran	<0.05	<0.05	n.c.	n.c.	n.c.

RAC	Residues in RAC (unwashed sample, mg/kg)		DALT (days)	Processed commodity	Residue (mg/kg)		PF*		Comments/ Reference
				Flour	**<0.05**	**<0.05**	**n.c.**	**n.c.**	**n.c.**
				Germ, dry milling	<0.05	<0.05	n.c.	n.c.	n.c.
				Germ, wet milling	<0.05	<0.05	n.c.	n.c.	n.c.
				Grits	**<0.05**	**<0.05**	**n.c.**	**n.c.**	**n.c.**
				Meal, dry milled	**<0.05**	**<0.05**	**n.c.**	**n.c.**	**n.c.**
				Oil, dry milled	<0.05	<0.05	n.c.	n.c.	n.c.
				Oil, wet milled	<0.05	<0.05	n.c.	n.c.	n.c.
				Starch	<0.05	<0.05	n.c.	n.c.	n.c.
				Steep water	<0.05	<0.05	n.c.	n.c.	n.c.
Sum of flupyradifurone and DFA, expressed as flupyradifurone (RD-RA)									
Maize/field corn grain	0.062	0.074	21	Aspirated grain fractions	0.36	0.55	5.8	7.4	Median: 6.6
				Bran	0.072	0.14	1.2	1.9	Median: 1.5
				Flour	**<0.06**	**<0.06**	**<1.0**	**<0.8**	Median: **<0.9**
				Germ, dry milling	<0.06	0.08	<1.0	1.1	Median: <1.0
				Germ, wet milling	<0.06	0.06	<1.0	0.9	Median: <0.9
				Grits	**<0.06**	**<0.06**	**<1.0**	**<0.8**	Median: **<0.9**
				Meal, dry milled	**<0.06**	**<0.06**	**<1.0**	**<0.8**	Median: **<0.9**
				Oil, dry milled	<0.06	<0.06	<1.0	<0.8	Median: <0.9
				Oil, wet milled	<0.06	<0.06	<1.0	<0.8	Median: <0.9
				Starch	<0.06	<0.06	<1.0	<0.8	Median: <0.9
				Steep water	<0.06	0.06	<1.0	0.8	Median: <0.9

* PF = Processing Factor
RD-Mo = Residue definition for Monitoring
RD-RA = Residue definition for Risk Assessment

Conclusion

Residues of DFA at or above 0.05 mg/kg were neither detected in field corn grain (RAC) nor in any of the processed commodities - thus no concentration of DFA residues is expected during processing. The BYI 02960 residues did also not concentrate (processing factor ≤1X) in the processed commodities of flour, germ (dry or wet milled), grits, meal (dry milled), oil (dry or wet milled), starch and steep water. However, the residues did concentrate significantly in the aspirated grain fraction (mean processing factor of 23), and slightly in bran (mean processing factor of 2.8).

Overall, the total residue of BYI 02960 (sum of BYI 02960 and DFA) did only concentrate in the aspirated grain fractions (mean processing factor of 6.6) and in corn bran (mean processing factor of 1.5).

Figure A 21: Processing flowchart for production of maize/corn into ensilage

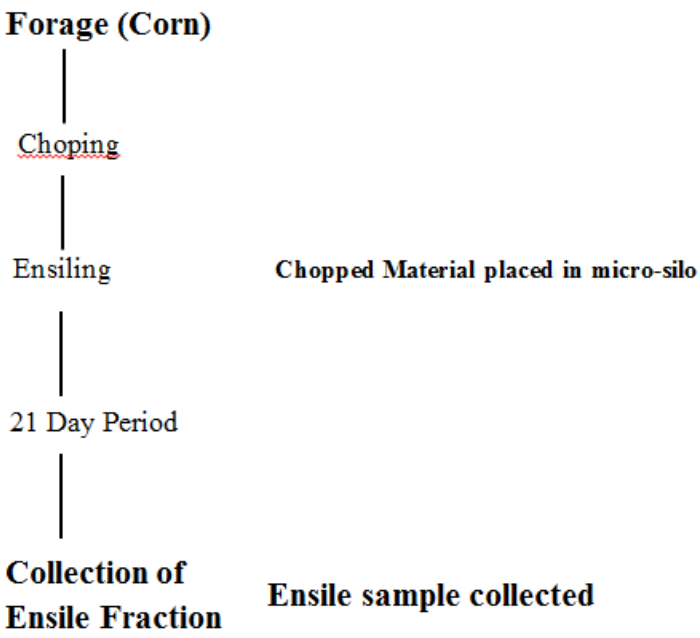
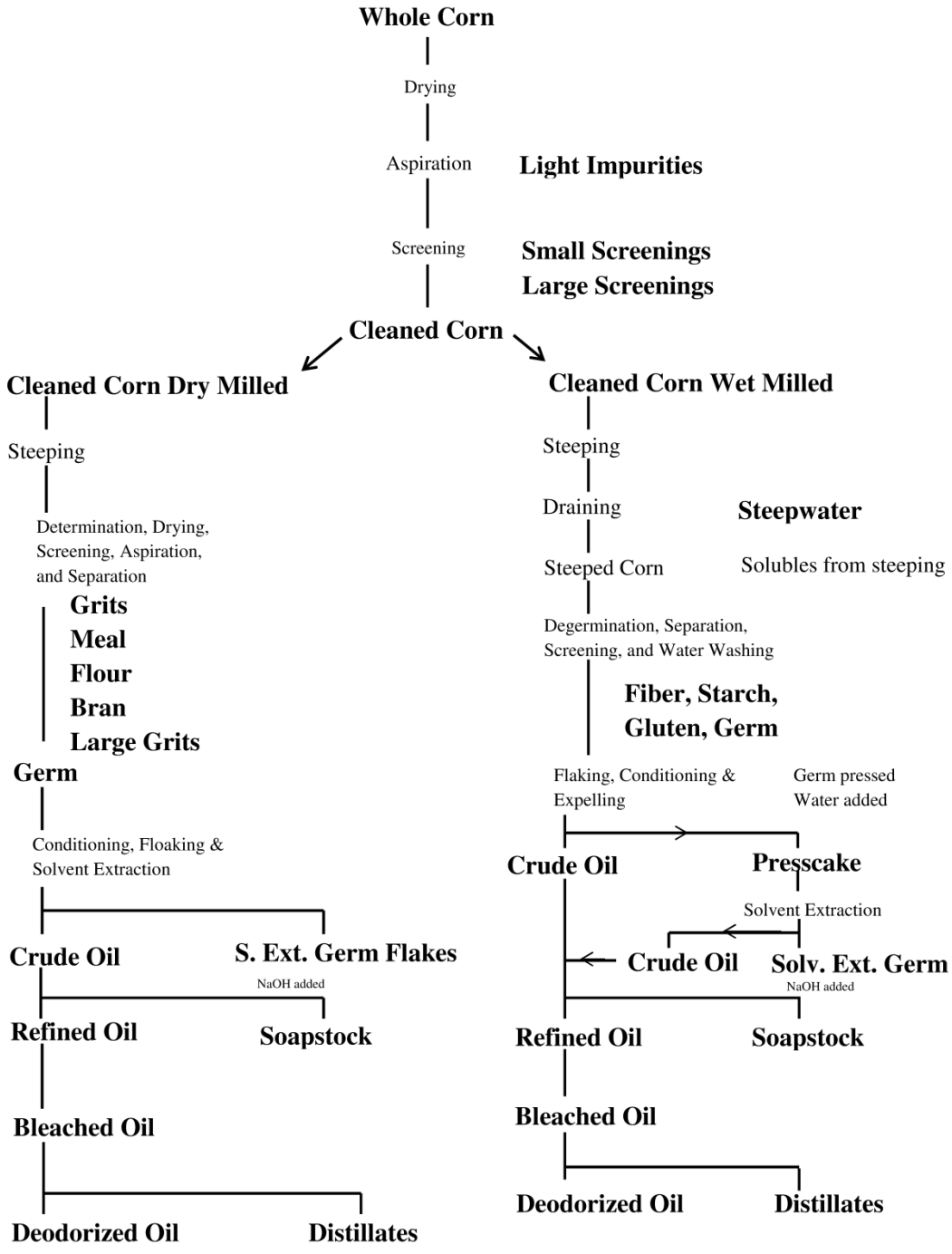


Figure A 22: Processing flowchart for dry and wet milling of corn



A 2.1.6 Magnitude of residues in representative succeeding crops

New field rotational crop studies were conducted in cereals, maize and oilseed rape after application of 175 g a.s./ha and in potato, brassica vegetables and strawberries after application of 300 g a.s./ha to bare soil.

Note: soil samples were taken and also analyzed for flupyradifurone-derived residues. Details on analytcal method 01074/M001 and relevant storage stability information (Netzband, D. J.; 2014; M-428408-02-1) can be provided on request.

A 2.1.6.1.1 Rotational crop study on potato, study report 16-2510

Comments of zRMS:

Four supervised residue trials were conducted in southern and northern Europe (Germany, the Netherlands, Italy and Spain) during the 2016 and 2017 seasons to determine the magnitude of the relevant residues of BYI 02960 in/on potato grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 300 g a.s./ha.
Plant-back intervals were 138 to 181 days (2nd rotation) and 295 to 343 days (3rd rotation).
Plant material to be analysed was potato tuber. The analyzed substances were BYI 02960 and its metabolite difluoroacetic acid. Soil samples were analysed for BYI 02960, difluoroacetic acid and 6-chloronicotinic acid.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.
The residue levels determined in the treated samples are summarized in the following table:

Table 26: Residues in soil and potato samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA* (calculated as BYI 02960) [mg/kg] min - max	sum of BYI 02960 and DFA [mg/kg] min - max	DFA* (calculated as DFA) [mg/kg] min - max	6-CNA (calculated as 6-CNA) [mg/kg] min - max
potato	tuber	T-2A	4	45 - 49	222 - 322	< 0.01	0.034 – 0.26	0.044 – 0.27	0.011 – 0.088	**
		T-3A			389 - 488	< 0.01	< 0.02 – 0.057	< 0.03 – 0.067	< 0.007 – 0.019	**
soil	0-20 cm [0-8 in]	T-2A	4	-	138 - 181	0.019 – 0.061	-	-	< 0.005	< 0.005
		T-3A			292 - 349	0.007 – 0.026	-	-	< 0.005	< 0.005

DAT = Days after treatment
*: The exact LOQ value is 0.00665 mg/kg. This is reported as 0.007 mg/kg if calculated as DFA (equivalent to 0.02 mg/kg if calculated as BYI 02960).
** see Appendix 10

Residues of BYI 02960 were always <0.01 mg/kg in potato tubers. DFA residues expressed as DFA were between 0.011 and 0.088 mg/kg and sum of BYI 02960 and DFA equals 0.044-0.27 mg/kg of the second rotation.It should be noted that DFA residues in potatoes of the second rotation were always higher as compared to the DFA residues in potatoes of the third rotation.
Soil samples were taken prior to planting the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.019-0.061 mg/kg.
The study is acceptable.

Reference:	**KCA 6.6.2/01**
Title:	Determination of the residues of BYI 02960 in/on potato and soil after spraying of BYI 02960 SL 200 in the field in Germany, the Netherlands, Italy and Spain
Report:	Freitag, T.; 2018; 16-2510; M-613149-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on potato grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 138 to 181 days (2nd rotation) and 295 to 343 days (3rd rotation). The study included four supervised residue trials conducted in the field in northern Europe (Germany and the Netherlands) and in southern Europe (Italy and Spain) during the seasons of 2016 and 2017.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 1.5 L/ha (equating to 0.3 kg/ha active substance) and using a water rate of approximately 300 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate allowed for BYI 02960 in Europe for home and garden uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-planting. A plant-back interval of 30 days (1st rotation) was not considered relevant for potatoes as such a short plant-back interval is not reasonable for a crop with a rather long vegetation period.

After spray application, the test item was incorporated into the soil. Prior to planting potato for the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 239 days) until analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Samples of the rotational crop potato were taken at BBCH growth stage of 45 (50% of total final tuber mass reached) and at harvest time (BBCH 49) and stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 161 days). The samples were analyzed for the parent compound and its metabolites DFA using method 01304. The LOQ was 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA (expressed as parent equivalent, corresponding to 0.0067 mg/kg when expressed as analyte equivalent).

Results and discussions

In order to evaluate the potential residues in potatoes grown as following crops, samples were taken at an early harvest time (BBCH growth stage 45) and at maturity (BBCH 49) and were analyzed for

BYI 02960-related residues. Residues of BYI 02960 were always <0.01 mg/kg in potato tubers whereas DFA residues were in the vast majority of the samples above the LOQ of 0.0067 mg/kg (expressed as DFA). DFA residues in potatoes of the second rotation were always higher as compared to the DFA residues in potatoes of the third rotation. Early harvested potatoes showed also slightly higher DFA residues or residues at the same level as potatoes sampled at BBCH growth stage 49. Overall, DFA residues ranged between <0.0067 mg/kg and 0.088 mg/kg (expressed as DFA).

The corresponding soil samples collected prior to planting the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.019-0.061 mg/kg.

BYI 02960 and DFA residues detected in potato tubers grown as rotational crops and in the corresponding soil samples are summarized in Table A 42.

Parallel to the residue analysis, analytical method 01304 was successfully validated for potato tuber. As this sample matrix was not included in the original validation, a limited set (3 repetitions at the LOQ level and 3 at the 10x LOQ level) of additional validation recoveries was performed within the course of the study. These concurrent (procedural) recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. The recoveries (including the concurrent recovery conducted at the 100x LOQ level) are summarized in Table A 40.

Concurrent recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 41.

Table A 40: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in potato (tuber)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
16-2510	potato	tuber	BYI 02960	3	0.01	99; 99; 101	99	101	100	1.2
				3	0.1	95; 96; 96	95	96	96	0.6
(16-2510-01)				1	1.0	88	-	-	-	-
				7	overall		88	101	96	4.4
			DFA	3	0.02	99; 100; 104	99	104	101	2.6
GLP: yes 2018				3	0.20	92; 92; 94	92	94	93	1.2
				1	2.0	87	-	-	-	-
				3	overall		87	104	95	6.1

Table A 41: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	BYI 02960	24 22 46	0.005 0.05 overall	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79 72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	70 72 70	92 88 92	82 81 82	7.2 5.9 6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	DFA	24 22 46	0.005 0.05 overall	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86 66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	72 66 66	93 90 93	82 78 80	5.9 7.8 7.3

Table A 42: Rotational trial summary for potato, study report 16-2510

Reference:	Determination of the residues of BYI 02960 in/on potato and soil after Spraying of BYI 02960 SL 200 in the Field in Germany, the Netherlands, Italy and Spain, Freitag, T., 2018, 16-2510, M-613149-01-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 161 days at ≤ -18°C soil: max. 239 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	potato	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA (expressed as BYI 02960) soil: 0.005 mg/kg for BYI 0296 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT /PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2510 16-2510-01 T-2A Germany 49377 Vechta-Langförden	Bare soil Potato Seresta	1) 24.04.2017 2) 15.06.2017 – 05.07.2017 3) 01.09.2017 – 30.09.2017	SPI and incorporation	0.300	300	0.1	25.10.2016	 45 49	soil 0-20 cm [0-8 in] tuber 2nd rotation (PB 181 days)	0.019 <0.01 <0.01	 0.036 0.034	 0.046 0.044	<0.005 0.012 0.011	181 287 322
16-2510 16-2510-02 T-2A The Netherlands 1681 ND Zwaagdijk	Bare soil Potato Kennebec	1) 12.04.2017 2) 01.07.2017 – 15.07.2017 3) 24.07.2017 – 01.08.2017	SPI and incorporation	0.300	400	0.075	25.11.2016	 45 49	soil 0-20 cm [0-8 in] tuber 2nd rotation (PB 138 days)	0.061 <0.01 <0.01	 0.13 0.084	 0.14 0.094	<0.005 0.042 0.028	138 222 243
16-2510 16-2510-03 Italy T-2A 37050 Albaro di Ronco All'Adige	Bare soil potato Kennebec	1) 02.03.2017 2) 19.06.2017 – 28.06.2017 3) 14.07.2017 – 31.07.2017	SPI and incorporation	0.300	300	0.1	12.10.2016	 45 49	soil 0-20 cm [0-8 in] tuber 2nd rotation (PB 141 days)	0.035 <0.01 <0.01	 0.26 0.21	 0.27 0.22	<0.005 0.088 0.071	147 243 286
16-2510 16-2510-04 Spain T-2A 46230 Alginet	Bare soil Potato Desiree	1) 04.02.2017 2) 15.04.2017 – 08.05.2017 3) 01.06.2017 – 10.06.2017	SPI and incorporation	0.300	400	0.075	06.09.2016	 45 49	soil 0-20 cm [0-8 in] tuber 2nd rotation (PB 151 days)	0.030 <0.01 <0.01	 0.049 0.069	 0.059 0.079	<0.005 0.016 0.023	148 245 268
16-2510 16-2510-01 T-3A Germany 49377 Vechta-Langförden	Bare soil Potato Seresta	1) 24.04.2017 2) 15.06.2017 – 05.07.2017 3) 01.09.2017 – 30.09.2017	SPI and incorporation	0.300	300	0.1	09.06.2016	 45 49	soil 0-20 cm [0-8 in] tuber 3rd rotation (PB 319 days)	0.014 <0.01 <0.01	 <0.02 <0.02	 <0.03 <0.03	<0.005 <0.0067 <0.0067	319 425 460

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT /PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2510 16-2510-02 T-3A The Netherlands 1681 ND Zwaagdijk	Bare soil Potato Kennebec	 1) 12.04.2017 2) 01.07.2017 – 15.07.2017 3) 24.07.2017 – 01.08.2017	SPI and incorporation	0.300	400	0.075	11.05.2016	 45 49	soil 0-20 cm [0-8 in] tuber 3rd rotation (PB 336 days)	0.026 <0.01 <0.01	 0.054 0.042	 0.064 0.052	<0.005 0.018 0.014	336 420 441
16-2510 16-2510-03 Italy T-3A 37050 Albaro di Ronco All'Adige	Bare soil Potato Kennebec	 1) 02.03.2017 2) 19.06.2017 – 28.06.2017 3) 14.07.2017 – 31.07.2017	SPI and incorporation	0.300	300	0.1	24.03.2016	 45 49	soil 0-20 cm [0-8 in] tuber 3rd rotation (PB 343 days)	0.007 <0.01 <0.01	 0.057 0.044	 0.067 0.054	<0.005 0.019 0.015	349 445 488
16-2510 16-2510-04 Spain T-3A 46230 Alginet	Bare soil Potato Desiree	 1) 04.02.2017 2) 15.04.2017 – 08.05.2017 3) 01.06.2017 – 10.06.2017	SPI and incorporation	0.300	400	0.075	15.04.2016	 45 49	soil 0-20 cm [0-8 in] tuber 3rd rotation (PB 295 days)	0.026 <0.01 <0.01	 0.033 0.037	 0.043 0.047	<0.005 0.011 0.012	292 389 412

A 2.1.6.1.2 Rotational crop study on oilseed rape, study report 16-2511

Comments of zRMS:

Two supervised residue trials were conducted in northern Europe (Belgium and the Netherlands) during the 2016 and 2017 seasons to determine the magnitude of the relevant residues of BYI 02960 in/on rape grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 175 g a.s./ha.
Plant-back intervals for were 137 to 147 days (2nd rotation) and 330 to 335 days (3rd rotation).
Samples of representative plant commodities (rape, green material and rape, seed) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes were combined to the sum of BYI 02960 and difluoroacetic acid).
Soil samples were taken at the same day of sowing of the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.

The residue levels determined in the treated samples are summarized in the following table:

Table 20: Analytical results of treated samples for BYI 02960, difluoroacetic acid (DFA), and the sum of BYI 02960 and difluoroacetic acid, test system: rape

Trial no. Country	Plot	PBI (DAT)	Sample no.	Growth stage [BBCH]	DAT	Sample material	BYI 02960 [mg/kg]	DFA [mg/kg] (calculated as BYI 02960)	sum of BYI 02960 DFA [mg/kg] (calculated as BYI 02960)	DFA [mg/kg] (calculated as DFA)*
16-2511-01 The Netherlands	T-2A	137	0022E	59	187	green material	< 0.01	0.045	0.055	0.015
			0024E	89	263	seed	< 0.01	0.027	0.037	0.009
	T-3A	335	0028E	59	385	green material	< 0.01	0.023	0.033	0.008
			0030E	89	461	seed	< 0.01	0.02	< 0.03	0.007
16-2511-02 Belgium	T-2A	147	0022E	59	354	green material	< 0.01	0.029	0.039	0.010
			0024E	89	459	seed	< 0.01	0.043	0.053	0.014
	T-3A	330	0028E	59	549	green material	< 0.01	0.02	< 0.03	0.007
			0030E	89	629	seed	< 0.01	0.023	0.033	0.007

Table 21: Analytical results of treated samples for BYI 02960, difluoroacetic acid (DFA), and 6-chloronicotinic acid (6-CNA), test system: soil

Trial no. Country	Plot	Sample no.	DAT	Sample material	BYI 02960 [mg/kg]	DFA [mg/kg]	6-CNA [mg/kg]
16-2511-01 The Netherlands	T-2A	0020E	137	0-20 cm [0-8 in] plastic tube removed	0.046	< 0.005	< 0.005
	T-3A	0026E	335	0-20 cm [0-8 in] plastic tube removed	0.019	< 0.005	< 0.005
16-2511-02 Belgium	T-2A	0020E	147	0-20 cm [0-8 in] plastic tube removed	0.016	< 0.005	< 0.005
	T-3A	0026E	330	0-20 cm [0-8 in] plastic tube removed	0.015	< 0.005	< 0.005

Residues of BYI 02960 were always <0.01 mg/kg in green material and mature seeds of oilseed rape. DFA residues expressed as DFA were between 0.01 and 0.015 mg/kg in green material and between 0.009 and 0.014 mg/kg in seeds in the second rotation. DFA residues
expressed as DFA were lower in the third rotation and ranged between 0.007-0.008 mg/kg for green material and equal 0.007 mg/kg for seeds.
Soil samples were taken prior to planting the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.016-0.046 mg/kg.

The study is acceptable.

Reference:	**KCA 6.6.2/02**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop rape and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands - Final report
Report:	Freitag, T.; Effertz, C.; 2018; 16-2511; M-617178-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of flupyradifurone (BYI 02960) in/on oilseed rape grown as a rotational crop after one spray application to bare soil with Flupyradifurone SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 137 to 147 days (2nd rotation) and 330 to 335 days (3rd rotation). The study included two supervised residue trials conducted in the field in northern Europe (Belgium and the Netherlands) during the seasons of 2016 and 2017.

One spray application of Flupyradifurone SL 200 was made at an intended application rate of 0.875 L/ha (equating to 0.175 kg/ha active substance) and using a water rate of approximately 300 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate intended for BYI 02960 in Europe for agricultural uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping. A plant-back interval of 30 days (1st rotation) was not considered relevant for oilseed rape as such a short plant-back interval is not reasonable for a crop that can only cultivated as spring or winter rape.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 274 days) prior to analysis according to method 01074/M001. The relevant analytes in soil were BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent).

Samples of rape seeds were taken at harvest time (BBCH 89) and green material was collected at an earlier interval (BBCH growth stage 59: First petals visible, flower buds still closed ("yellow bud")). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 218 days). The crop samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg and for DFA 0.02 mg/kg (expressed as parent equivalent, corresponding to 0.0067 mg/kg when expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Results and discussions

In order to evaluate the potential residues in oilseed rape grown as following crops, samples of green material and mature rape seeds were analyzed for BYI 02960-related residues. Residues of parent

BYI 02960 were always <0.01 mg/kg in green material and mature seeds of oilseed rape. DFA residues in crop matrices were generally highest in the second rotation and were 0.010 mg/kg and 0.015 mg/kg (expressed as DFA) in green material and 0.0091 mg/kg and 0.014 mg/kg (expressed as DFA) in mature seeds. DFA residues in green material and seeds of the third rotation ranged around the limit of quantitation.
The corresponding soil samples collected prior to sowing the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.016-0.046 mg/kg.
All BYI 02960 and DFA residues detected in matrices of oilseed rape grown as rotational crops and in the corresponding soil samples are summarized in Table A 45.

Parallel to the analysis of the plant matrices, concurrent (procedural) recoveries were conducted at the LOQ level and the 50x LOQ level for each analyte included in method 01304 in order to confirm the successful performance of the method during the conduct of the study. For the matrices relevant to this study (rape green material and rape seed), but not included in the original validation, a limited set (3 repetitions at two fortification levels) of additional validation recoveries were analyzed within the course of study 16-2521 which was conducted in parallel. Mean recoveries conducted for the validation and concurrent recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level (if applicable, n ≥ 3) were well below 20%. The procedural recoveries are summarized in Table A 43.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 44.

Table A 43: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in oilseed rape (green material and seed)

Study **Trial No.** **Plot No.** **GLP** **Year**	**Crop**	**Portion analyzed**	**a.s./ metabolite**	**n**	**Fortification level (mg/kg)**	**Recovery (%)**				
						Individual recoveries	**Min**	**Max**	**Mean**	**RSD**
16-2511 (16-2511-01) GLP: yes 2018	oilseed rape	green material	BYI 02960	1 1 2	0.01 0.5 overall	83 90	83 90 83	83 90 90	- - 87	- - -
		seed		1 1 2	0.01 0.5 overall	100 93	100 93 93	100 93 100	- - 97	- - -
		green material	DFA	1 1 2	0.02 1.0 overall	98 89	98 89 89	98 89 98	- - 94	- - -
		seed		1 1 2	0.02 1.0 overall	89 83	89 83 83-	89 83 89	- - 86	- - -

Table A 44: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	BYI 02960	24 22 46	0.005 0.05 overall	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79 72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	70 72 70	92 88 92	82 81 82	7.2 5.9 6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	DFA	24 22 46	0.005 0.05 overall	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86 66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	72 66 66	93 90 93	82 78 80	5.9 7.8 7.3

Table A 45: Rotational trial summary for oilseed rape, study report 16-2511

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop rape and soil after spray application of BYI 02960 SL 200 to bare soil in the Field in Belgium and the Netherlands, Freitag, T., 2018, 16-2511, M-617178-02-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 218 days at ≤ -18°C soil: max. 274 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	oilseed rape	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA (expressed as BYI 02960) soil: 0.005 mg/kg for BYI 0296 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2511 16-2511-01 T-2A The Netherlands 1681 ND Zwaagdijk	Bare soil Rape, summer Campino	 1) 11.04.2017 2) 01.06.2017 – 01.07.2017 3) 12.08.2017 – 22.08.2017	SPI and incorporation	0.175	400	0.04	25.11.2016	 59 89	soil 0-20 cm [0-8 in] green material seed 2nd rotation (PB 137 days)	0.046 <0.01 <0.01	 0.045 0.027	 0.055 0.037	<0.005 0.015 0.009	137 187 263
16-2511 16-2511-02 T-2A Belgium 6221 Saint-Amand	Bare soil Rape DK Explicit	 1) 08.09.2016 2) 07.04.2017 - 15.05.2017 3) 17.07.2017 – 25.07.2017	SPI and incorporation	0.175	300	0.06	14.04.2016	 59 89	soil 0-20 cm [0-8 in] green material seed 2nd rotation (PB 147 days)	0.016 <0.01 <0.01	 0.029 0.043	 0.039 0.053	<0.005 0.010 0.014	147 174 279
16-2511 16-2511-01 T-3A The Netherlands 1681 ND Zwaagdijk	Bare soil Rape, summer Campino	 1) 11.04.2017 2) 01.06.2017 – 01.07.2017 3) 12.08.2017 – 22.08.2017	SPI and incorporation	0.175	300	0.06	11.05.2016	 59 89	soil 0-20 cm [0-8 in] green material seed 3rd rotation (PB 335 days)	0.019 <0.01 <0.01	 0.023 0.020	 0.033 <0.03	<0.005 0.008 0.007	335 385 461
16-2511 16-2511-02 T-3A Belgium 6221 Saint-Amand	Bare soil Rape Diffusion	 1) 06.09.2017 2) 17.04.2018 – 07.05.2018 3) 02.07.2018 – 07.07.2018	SPI and incorporation	0.214	367	0.06	11.10.2016	 59 89	soil 0-20 cm [0-8 in] green material seed 3rd rotation (PB 330 days)	0.015 <0.01 <0.01	 0.020 0.023	 <0.03 0.033	<0.005 0.007 0.007	330 549 629

n.a. not available

A 2.1.6.1.3 Rotational crop study on oilseed rape, study report 16-2521

Comments of zRMS:

Two supervised residue trials were conducted in southern Europe (Italy and Portugal) during the seasons of 2016 and 2017 to determine the magnitude of the relevant residues of BYI 02960 in rape (green material and seed) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 175 g a.s./ha.
Plant-back intervals were 158 to 167 days (2nd rotation) and 273 days (3rd rotation). Samples of representative plant commodities of rape (green material and seed) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid.
Soil samples were taken prior to sowing the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The individual LOQ for BYI 02960 is 0.01 mg/kg in the sample materials of rape (green material and seed). The individual LOQ for difluoroacetic acid is 0.02 mg/kg (expressed in parent BYI 02960 equivalents) for both sample materials.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.

The residue levels determined in the treated samples are summarized in the following table:

Table 4: Residue summary in/on rape

Trial No. Country	Plot	PBI	Sample material	BBCH	DAT	Residues [mg/kg] a.s. BYI 02960			
						BYI 02960	DFA (calculated as BYI 02960)	sum of BYI 02960 and DFA (calculated as BYI 02960)	DFA (calculated as DFA)
16-2521-01 Italy	T-2A	158	green material	59	207	< 0.01	0.17	0.18	0.056
			seed	89	267	< 0.01	0.067	0.077	0.022
	T-3A	273	green material	59	322	< 0.01	0.13	0.14	0.044
			seed	89	382	< 0.01	0.040	0.050	0.013
16-2521-02 Portugal	T-2A	167	green material	59	337	< 0.01	0.25	0.26	0.085
	T-3A	273	green material	59	443	< 0.01	0.14	0.15	0.046

DAT = Days after treatment a.s. = Active substance PBI = plant-back interval

Table 5: Residue summary in/on soil

Trial No. Country	Plot	Sample material	DAT	Residues [mg/kg] a.s. BYI 02960		
				BYI 02960	DFA	6-CNA
16-2521-01 Italy	T-2A	soil, 0-20 cm [0-8 in] plastic tube removed	158	0.032	< 0.005	< 0.005
	T-3A	soil, 0-20 cm [0-8 in] plastic tube removed	273	0.023	< 0.005	< 0.005
16-2521-02 Portugal	T-2A	soil, 0-15 cm [0-6 in] plastic tube removed*	166	0.040	< 0.005	< 0.005
	T-3A	soil, 0-15 cm [0-6 in] plastic tube removed*	272	0.034	< 0.005	< 0.005

Unfortunately, only green material, but no seed samples could be collected from trial 16-2521-02 as birds destroyed the harvest.
Residues of BYI 02960 were always <0.01 mg/kg in green material and mature seeds of oilseed rape. DFA residues expressed as DFA were between 0.056 and 0.085 mg/kg in green material and 0.022 mg/kg in seeds in the second rotation. DFA residues expressed as DFA were lower in the third rotation and ranged between 0.044-0.046 mg/kg for green material and equal 0.013 mg/kg for seeds.
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.032-0.040 mg/kg in the second rotation and 0.023-0.034 mg/kg in the third rotation.

The study is acceptable.

Reference:	**KCA 6.6.2/03**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop rape and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Italy and Portugal
Report:	Kaussmann, M.; van Berkum, S.; 2018; 16-2521; M-613466-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	Yes, but acceptable
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on oilseed rape grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 158 to 167 days (2nd rotation) and 273 days (3rd rotation). The study included two supervised residue trials conducted in the field in southern Europe (Italy and Portugal) during the seasons of 2016 and 2017.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 0.875 L/ha (equating to 0.175 kg/ha active substance) and using a water rate of approximately 300 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate intended for BYI 02960 in Europe for agricultural uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping. A plant-back interval of 30 days (1st rotation) was not considered relevant for oilseed rape as such a short plant-back interval is not reasonable for a crop that can only cultivated as spring or winter rape.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 443 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Samples of rape seeds were taken at harvest time (BBCH 89) and green material was collected at an earlier interval (BBCH growth stage 59: First petals visible, flower buds still closed ("yellow bud")). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 390 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg and for DFA 0.02 mg/kg (expressed as parent equivalent, corresponding to 0.0067 mg/kg when expressed as analyte equivalent).

Results and discussions

In order to evaluate the potential residues in oilseed rape grown as following crops, samples of green material and mature rape seeds were analyzed for BYI 02960-related residues. Residues of parent BYI 02960 were always <0.01 mg/kg in green material and mature seeds of oilseed rape. DFA residues were higher in the second rotation and amounted to 0.056 mg/kg (expressed as DFA) in green material and to 0.022 mg/kg (expressed as DFA) in mature seeds in the Italian trials.

The soil samples collected prior to sowing the crop for the individual rotations in Italy showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and were 0.040 mg/kg. All available BYI 02960 and DFA residues detected in matrices of oilseed rape grown as rotational crops and in the corresponding soil samples are summarized in Table A 48.

Parallel to the residue analysis, analytical method 01304 was successfully validated for green material and mature seeds of oilseed rape. As this sample matrix was not included in the original validation, a limited set (3 repetitions at the LOQ level and 3 at the 10x LOQ level) of additional validation recoveries were performed within the course of the study. These concurrent (procedural) recoveries were always in the acceptable range of 70-110%, except for a single DFA recovery in seeds conducted at the fortification level representing the 100x LOQ level (69%). This recovery was not repeated as the highest DFA residue in seeds was 0.022 mg/kg and therefore in the range which was covered by acceptable recoveries. The RSD values per analyte and fortification level were always well below 20%, if applicable ($n \geq 3$). The recoveries (including the concurrent recovery conducted at the 100x LOQ level) are summarized in Table A 46.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 47.

Table A 46: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in oilseed rape (green material and seed)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2521	oilseed rape	green material	BYI 02960	3	0.01	103; 106; 107	103	107	105	2.0
				3	0.10	95; 96; 98	95	98	96	1.6
(16-2521-01)				1	1.0	95	95	95	-	-
				7	overall		95	107	100	5.2
GLP: yes 2018		seed		3	0.01	96; 100; 101	96	101	99	2.7
				3	0.10	98; 101; 103	98	103	101	2.5
				1	1.0	94	94	94	-	-
				7	overall		94	103	99	3.2
		green material	DFA	3	0.02	97; 101; 110	97	110	103	6.5
				3	0.20	92; 100; 105	92	105	99	6.6
				1	2.0	92	92	92	-	-
				7	overall		92	110	100	6.6
		seed		3	0.02	94; 97; 101	94	101	97	3.6
				3	0.20	79; 84; 87	79	87	83	4.8
				1	2.0	69*	69	69	-	-
				7	overall		69	101	87	12.7

* This recovery was accepted since it is very close to 70% and all residues detected in the rape samples were below 0.2 mg/kg

Table A 47: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	BYI 02960	24	0.005	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79	70	92	82	7.2
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	72	88	81	5.9
GLP: yes 2018				46	overall		70	92	82	6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	DFA	24	0.005	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86	72	93	82	5.9
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	66	90	78	7.8
GLP: yes 2018				46	overall		66	93	80	7.3

Table A 48: Rotational trial summary for oilseed rape, study report 16-2521

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop rape and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Italy and Portugal, Kaussmann, M.; van Berkum, S., 2018, 16-2521, M-613466-02-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 390 days at ≤ -18°C soil: max. 443 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	oilseed rape	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA (expressed as BYI 02960) soil: 0.005 mg/kg for BYI 0296 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2521 16-2521-01 T-2A Italy 12050 Castagnito d'Alba	Bare soil Rape Delight	 1) 27.03.2017 2) 17.05.2017 – 06.05.2017 3) 07.2017	SPI and incorporation	0.175	300	0.06	20.10.2016	 59 89	soil 0-20 cm [0-8 in] green material seed 2nd rotation (PB 158 days)	0.032 <0.01 <0.01	 0.17 0.067	 0.18 0.077	<0.005 0.056 0.022	158 207 267
16-2521 16-2521-02 T-2A Portugal 2070-316 Ereira	Bare soil Rape DK Expower	 1) 21.03.2017 2) 25.06.2018 – 15.07.2018 3) 20.08.2018 – 30.08.2018	SPI and incorporation	0.175	300	0.06	05.10.2016	 59 n.a.	soil[1)] 0-15 cm [0-6 in] green material seed 2nd rotation (PB 167 days)	0.040 <0.01. n.a.	 0.25 n.a.	 0.26 n.a.	<0.005 0.085 n.a.	166 337 n.a.
16-2521 16-2521-01 T-3A Italy 12050 Castagnito d'Alba	Bare soil Rape Delight	 1) 27.03.2017 2) 17.05.2017 – 06.05.2017 3) 07.2017	SPI and incorporation	0.175	300	0.06	27.06.2016	 59 89	soil 0-20 cm [0-8 in] green material seed 3rd rotation (PB 273 days)	0.023 <0.01 <0.01	 0.13 0.040	 0.14 0.050	<0.005. 0.044 0.013	n.a. 322 382
16-2521 16-2521-02 T-3A Portugal 2070-316 Ereira	Bare soil Rape DK Expower	 1) 21.03.2017 2) 25.06.2018 – 15.07.2018 3) 20.08.2018 – 30.08.2018	SPI and incorporation	0.175	300	0.06	21.06.2016	 59 n.a.	soil[1)] 0-15 cm [0-6 in] green material seed 3rd rotation (PB 273 days)	0.034 <0.01 n.a.	 0.14 n.a.	 0.15 n.a.	<0.005 0.046 n.a.	272 443 n.a.

[1)] Due to very hard soil, sampling was only possible to a limited depth (0-15 cm)

n.a. not available as harvest was destroyed by birds

A 2.1.6.1.4 Rotational crop study on barley, study report 16-2512

Comments of zRMS:

Two supervised residue trials were conducted in northern Europe (the United Kingdom and the Netherlands) during the seasons of 2016 and 2017 to determine the magnitude of the relevant residues of BYI 02960 in/on barley (green material, grain, and straw) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 175 g a.s./ha.
Plant-back intervals were 137 to 174 days (2nd rotation) and 335 to 337 days (3rd rotation).
Samples of representative plant commodities (barley green material, grain and straw) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes combined to the sum of BYI 02960 and difluoroacetic acid).
Soil samples were taken prior to planting the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The individual LOQ for BYI 02960 is 0.01mg/kg in the sample materials barley green material, grain and straw. The individual LOQ for difluoroacetic acid is 0.02 mg/kg in the sample materials barley green material and grain and 0.1 mg/kg in the sample material straw (expressed in parent BYI 02960 equivalents).
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.

The residue levels determined in the treated samples are summarized in the following table:

Table 21: Residues in soil and barley samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA (calculated as DFA) [mg/kg] min - max	sum of BYI 02960 and DFA (calculated as BYI 02960) [mg/kg] min - max
barley	green material	T-2A	2	75	230 - 406	< 0.01	0.008 – 0.014	0.034 – 0.051
		T-3A			421 - 428	< 0.01	< 0.007* – 0.02	< 0.03 – 0.07
	grain	T-2A	2	89	262 - 441	< 0.01	0.038 – 0.047	0.12 – 0.15
		T-3A			460	< 0.01	0.025 - 0.04	0.085 – 0.13
	straw	T-2A	2	89	262 - 441	< 0.01	< 0.03**	< 0.11
		T-3A			460	< 0.01	< 0.03**	< 0.11
soil	0-20 cm [0-8 in]	T-2A	2	-	137 - 180	0.024 – 0.036	< 0.005	-
		T-3A			335	0.017	< 0.005	-

DAT = Days after treatment
*: The exact LOQ value is 0.00665 mg/kg. This is reported as 0.007 mg/kg if calculated as DFA (equivalent to 0.02 mg/kg if calculated as BYI 02960).
**: The exact LOQ value is 0.033267 mg/kg. This is reported as 0.03 mg/kg if calculated as DFA (equivalent to 0.1 mg/kg if calculated as BYI 02960).

Residues of BYI 02960 were always <0.01 mg/kg in green material, straw and mature seeds of barley. DFA residues expressed as DFA were between 0.008 and 0.014 mg/kg in green material and between 0.038 and 0.047 mg/kg in seeds in the second rotation. DFA residues expressed as DFA were lower in the third rotation and ranged between 0.007-0.02 mg/kg for green material and between 0.025-0.04 mg/kg for seeds. DFA residues expressed as DFA were always below 0.03 mg/kg in straw of barley in the second and third rotations.
Soil samples were taken prior to planting the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.024-0.036 mg/kg in the second rotation and equals 0.017 mg/kg in the third rotation.
The study is acceptable.

Reference:	**KCA 6.6.2/04**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop barley and soil after spray application of BYI 02960 SL 200 to bare soil in the field in United Kingdom and the Netherlands
Report:	Freitag, T.; Kaussmann, M.; 2018; 16-2512; M-612254-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	Yes, but acceptable
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on barley grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 137 to 174 days (2nd rotation) and 335 to 337 days (3rd rotation). The study included two supervised residue trials conducted in northern Europe (the United Kingdom and the Netherlands) during the seasons of 2016 and 2017.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 0.875 L/ha (equating to 0.175 kg/ha active substance) and using a water rate of approximately 200 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate intended for BYI 02960 in Europe for agricultural uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping. A plant-back interval of 30 days (1st rotation) was not considered relevant for oilseed rape as such a short plant-back interval is not reasonable for a crop that can only cultivated as spring or winter barley.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 227 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Samples of the rotational crops were taken at their respective harvest times, as well as at one earlier interval (forage = "green material" at BBCH growth stage 75: Medium milk - grain content milky, grains reached final size, still green). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 119 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg in all plant matrices and for DFA 0.02 mg/kg in green material and grains and 0.1 mg/kg in straw (expressed as parent equivalent, corresponding to 0.0067 mg/kg or 0.03 mg/kg, respectively when expressed as analyte

equivalent).

Results and discussions

In order to evaluate the potential residues in barley grown as following crops, samples of green material were taken at an intermediate growth stage, as well as grain and straw samples at maturity. The samples were analyzed for BYI 02960-related residues. Residues of parent BYI 02960 were <0.01 mg/kg in all sample materials, irrespective from the sampling time. The highest residues of DFA were detected in mature grains collected from the second rotation – however grains from the third rotation showed comparable residues. Overall, DFA residues ranged from 0.025-0.047 mg/kg (expressed as DFA) in mature grains. The corresponding straw samples showed always DFA residues below the LOQ of 0.03 mg/kg. DFA residues in green material collected from the second and third rotation were also at the same level and ranged between <0.0067-0.020 mg/kg (expressed as DFA).

The corresponding soil samples collected prior to sowing the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.024-0.036 mg/kg. All BYI 02960 and DFA residues detected in matrices of barley grown as rotational crops and in the corresponding soil samples are summarized in Table A 51.

Parallel to the residue analysis, analytical method 01304 was successfully validated for the relevant matrices of barley (green material, grain and straw). As these sample matrices were not included in the original validation, a limited set (3 repetitions at the LOQ level and 3 at the 10x LOQ level) of additional validation recoveries were performed within the course of the study. These concurrent (procedural) recoveries were always in the acceptable range of 70-110%, and the RSD values per analyte and fortification level were always well below 20%, if applicable ($n \geq 3$). The recoveries are summarized in Table A 49.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 50.

Table A 49: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in barley (green material, grain and straw)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2512 (16-2512-01) and (16-2512-02) GLP: yes 2018	barley	green material	BYI 02960	3	0.01	98; 105; 107	98	107	103	4.6
				3	0.10	95; 101; 102	95	102	99	3.8
				6	overall		95	107	101	4.4
		grain		3	0.01	99; 106; 114	99	114	106	7.1
				3	0.10	99; 104; 105	99	105	103	3.1
				6	overall		99	114	105	5.3
		straw		3	0.01	88; 93; 98	88	98	93	5.4
				3	0.05	90; 93; 94	90	94	92	2.3
				3	0.50	92; 92; 97	92	97	94	3.1
				9	overall		88	98	93	3.4
		green material	DFA	3	0.02	101; 105; 106	101	106	104	2.5
				3	0.20	95; 99; 101	95	101	98	3.1
				6	overall		95	106	101	4.0
		grain		3	0.02	94 (128)*; 94 (128)*; 94 (128)*	94	94	94	0.0
				3	0.20	91; 97; 103	91	103	97	6.2
				6	overall		91	103	96	4.3
		straw		3	0.10	89; 90; 91	89	91	90	1.1
				3	1.0	88; 88; 91	88	91	89	1.9
				6	overall		88	91	90	1.5

* corrected for apparent residues found in the control sample used for fortification (16-2512-02-0027E: 0.00667 mg/kg expressed as parent equivalents). The uncorrected values are shown in brackets.

Table A 50: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	BYI 02960	24 22 46	0.005 0.05 overall	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79 72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	70 72 70	92 88 92	82 81 82	7.2 5.9 6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	DFA	24 22 46	0.005 0.05 overall	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86 66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	72 66 66	93 90 93	82 78 80	5.9 7.8 7.3

Table A 51: Rotational trial summary for barley, study report 16-2512

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop barley and soil after spray application of BYI 02960 SL 200 to bare soil in the field in United Kingdom and the Netherlands, Freitag, T.; Kaussmann, M., 2018, 16-2512, M-612254-01-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 119 days at ≤ -18°C soil: max. 227 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	barley	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 in all matrices 0.02 mg/kg for DFA (expressed as BYI 02960) in green material and grain 0.10 mg/kg for DFA (expressed as BYI 02960) in straw soil: 0.005 mg/kg for BYI 02960 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT/PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2512 16-2512-01 T-2A United Kingdom CB22 5EU Cambridge	Bare soil Barley, winter Cassia	 1) 19.10.2016 3) 10.07.2017 – 31.07.2017	SPI and incorporation	0.175	200	0.09	28.04.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 2nd rotation (PB 174 days)	0.024 <0.01 <0.01 <0.01	 0.041 0.14 <0.1	 0.051 0.15 <0.11	<0.005 0.014 0.047 <0.03	180 406 441 441
16-2512 16-2512-02 T-2A The Netherlands 1681 ND Zwaagdijk	Bare soil Barley, spring Kwisirina	 1) 11.04.2017 3) 10.08.2017 – 20.08.2017	SPI and incorporation	0.175	400	0.04	25.11.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 2nd rotation (PB 137 days)	0.036 <0.01 <0.01 <0.01	 0.024 0.11 <0.1	 0.034 0.12 <0.11	<0.005 0.008 0.038 <0.03	137 230 262 262
16-2512 16-2512-01 T-3A United Kingdom CB22 5EU Cambridge	Bare soil Barley, spring Laureate	 1) 31.03.2017 3) 24.07.2017 – 15.08.2017	SPI and incorporation	0.175	200	0.09	28.04.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 3rd rotation (PB 337 days)	0.017 <0.01 <0.01 <0.01	 0.06 0.12 <0.1	 0.07 0.13 <0.11	<0.005 0.02 0.047 <0.03	335 421 460 460

16-2512 16-2512-02 T-3A The Netherlands 1681 ND Zwaagdijk	Bare soil Barley, spring Kwisirina	 1) 11.04.2017 3) 10.08.2017 – 20.08.2017	SPI and incorporation	0.175	300	0.06	11.05.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 3rd rotation (PB 335 days)	0.017 <0.01 <0.01 <0.01	 <0.02 0.075 <0.1	 <0.03 0.085 <0.11	<0.005 <0.0067 0.025 <0.03	335 428 460 460

A 2.1.6.1.5 Rotational crop study on barley, study report 16-2522

Comments of zRMS:

Two supervised residue trials were conducted in southern Europe (Southern France and Portugal) during the 2016 / 2017 season to determine the magnitude of the relevant residues of BYI 02960 in/on barley (green material, grain, and straw) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 175 g a.s./ha.
Plant-back intervals were 107 to 163 days (2nd rotation) and 281 to 283 days (3rd rotation).
Samples of representative plant commodities of barley (green material, grain and straw) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes combined to the sum of BYI 02960 and difluoroacetic acid)
Soil samples were taken prior to sowing the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The individual LOQ for BYI 02960 is 0.01mg/kg in the sample materials barley (green material, grain and straw). The individual LOQ for difluoroacetic acid is 0.02 mg/kg in the sample materials barley green material and grain and 0.1 mg/kg in the sample material straw (expressed in parent BYI 02960 equivalents).
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.

The residue levels determined in the treated samples are summarized in the following table:

Table 21: Residues in soil and barley samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA (calculated as DFA) [mg/kg] min - max	sum of BYI 02960 and DFA (calculated as BYI 02960) [mg/kg] min - max
barley	green material	T-2A	2	75	195 - 245	<0.01 - 0.013	0.028-0.045	0.093 - 0.15
		T-3A			365 - 369	< 0.01	0.025-0.027	0.084 - 0.091
	grain	T-2A	2	89	218 - 274	< 0.01	0.088-0.12	0.27 - 0.38
		T-3A			392 - 394	< 0.01	0.056-0.088	0.18 - 0.27
	straw	T-2A	2	89	218 - 274	0.018 - 0.032	< 0.03 -0.036*	0.12 - 0.14
		T-3A			392 - 394	< 0.01 - 0.019	<0.03*	< 0.11 - 0.12
soil	0-20 cm [0-8 in]	T-2A	2	-	107 - 162	0.020 - 0.035	< 0.005	-
		T-3A			281 - 282	0.018 - 0.036	< 0.005	-

Residues of parent BYI 02960 were <0.01 mg/kg in green material in the third rotation and <0.01 – 0.013 mg/kg in the second rotation. In grain of barley residues of BYI 02960 were always below 0.01 mg/kg. BYI 02960 residues were detected in straw samples and ranged between 0.018 – 0.032 mg/kg in the second rotation and between <0.01 – 0.019 mg/kg in the third rotation.
DFA residues expressed as DFA were detected in all samples of green material and grain. DFA residues (expressed as DFA) in green material collected from the second and third rotation ranged between 0.025-0.045 mg/kg, and in grain collected from the second and third rotation ranged between 0.056-0.12 mg/kg. DFA were below 0.03 mg/kg in straw of barley in the second and third rotations, except one sample (0.036 mg/kg).
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were comparable to the residues detected in the soil samples collected prior to the third rotation and ranged between 0.020 – 0.035 mg/kg prior to the second rotation and 0.018-0.036 mg/kg prior to the third rotation.
No deviations occurred during the conduct of this study which have any negative impact on the quality of this study.

The study is acceptable.

Reference:	**KCA 6.6.2/05**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop barley and soil after spray application of BYI 02960 SL 200 to bare soil in the field in France (South) and Portugal
Report:	Kaussmann, M.; van Berkum, S.; 2018; 16-2522; M-613602-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	Yes (see report)
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on barley grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 107 to 163 days (2nd rotation) and 281 to 283 days (3rd rotation). The study included two supervised residue trials conducted in southern Europe (Southern France and Portugal) during the 2016 / 2017 season.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 0.875 L/ha (equating to 0.175 kg/ha active substance) and using a water rate of approximately 300 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate intended for BYI 02960 in Europe for agricultural uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping. A plant-back interval of 30 days (1st rotation) was not considered relevant for oilseed rape as such a short plant-back interval is not reasonable for a crop that can only cultivated as spring or winter barley.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 221 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Samples of the rotational crops were taken at their respective harvest times (BBCH 89), as well as at one earlier interval (forage = "green material" at BBCH growth stage 75: Medium milk - grain content milky,

grains reached final size, still green). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 139 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg in all plant matrices and for DFA 0.02 mg/kg in green material and grains and 0.1 mg/kg in straw (expressed as parent equivalent, corresponding to 0.0067 mg/kg or 0.03 mg/kg, respectively when expressed as analyte equivalent).

Results and discussions

In order to evaluate the potential residues in barley grown as following crops, samples of green material were taken at an intermediate growth stage, as well as grain and straw samples at maturity. The samples were analyzed for BYI 02960-related residues. Residues of parent BYI 02960 were <0.01 mg/kg in green material and grain collected from the 2nd rotation. BYI 02960 residues were detected in three of four straw samples and ranged between 0.018-0.032 mg/kg. The samples of the third rotation showed slightly higher residues than the samples from the 2nd rotation. Also one green material sample collected from the 2nd rotation showed BYI 02960 residues above the LOQ (0.013 mg/kg). Although BYI 02960 residues were detected in some sample materials (which is exceptional for rotational crops), the DFA residues were more pronounced in the samples collected. The highest residues of DFA were detected in mature grains from the 2nd and 3rd rotation and amounted for 0.088 mg/kg (expressed as DFA). Overall, DFA residues ranged from 0.056-0.088 mg/kg (expressed as DFA) in mature grains. The corresponding straw samples showed always DFA residues below the LOQ of 0.03 mg/kg. DFA residues in green material collected from the second and third rotation were also very similar and ranged between 0.025-0.045 mg/kg (expressed as DFA).

The corresponding soil samples collected prior to sowing the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the third rotation were slightly higher than the residues detected in the soil samples collected prior to the second rotation and ranged between 0.035-0.036 mg/kg. All BYI 02960 and DFA residues detected in matrices of barley grown as rotational crops and in the corresponding soil samples are summarized in Table A 54.

Parallel to the analysis of the plant matrices, concurrent (procedural) recoveries were conducted at the LOQ level and the 10x LOQ level for each analyte included in method 01304 in order to confirm the successful performance of the method during the conduct of the study. For the matrices relevant to this study (barley green material, grain and straw), but not included in the original validation, a limited set (3 repetitions at two fortification levels) of additional validation recoveries were analyzed within the course of study 16-2512 which was conducted in parallel. Mean recoveries conducted for the validation and concurrent recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level (if applicable, n ≥ 3) were well below 20%. The procedural recoveries are summarized in Table A 52.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 53.

Table A 52: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in barley (green material, grain and straw)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2522 (16-2522-01) and (16-2522-02) GLP: yes 2018	barley	green material	BYI 02960	3	0.01	93; 99; 101	93	101	98	4.3
				3	0.10	92; 93; 93	92	93	93	0.6
				1	1.0	94	94	94	-	-
				7	overall		92	101	95	3.7
		grain		3	0.01	97; 100; 101	97	101	99	2.1
				3	0.10	91; 92; 96	91	96	93	2.8
				1	1.0	97	97	97	-	
				7	overall		91	101	96	3.9
		straw		3	0.01	88; 89; 94	88	94	90	3.6
				1	0.05	96	96	96	-	-
				2	0.50	88; 93	88	93	91	-
				6	overall		88	96	91	3.8
		green material	DFA	3	0.02	94; 98; 106	94	106	99	6.2
				3	0.20	90; 93; 95	90	95	93	2.7
				1	2.0	92	92	92	-	-
				7	overall		90	106	95	5.5
		grain		3	0.02	99; 103; 106	99	106	103	3.4
				3	0.20	84; 89; 93	84	93	89	5.1
				1	2.0	90	90	90	-	-
				7	overall		84	106	95	8.5
		straw		1	0.10	85	85	85	-	-
				2	1.0	88; 89	88	89	89	-
				3	overall		85	89	87	2.4

Table A 53: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	BYI 02960	24 22 46	0.005 0.05 overall	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79 72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	70 72 70	92 88 92	82 81 82	7.2 5.9 6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	DFA	24 22 46	0.005 0.05 overall	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86 66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	72 66 66	93 90 93	82 78 80	5.9 7.8 7.3

Table A 54: Rotational trial summary for barley, study report 16-2522

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop barley and soil after spray application of BYI 02960 SL 200 to bare soil in the field in France (South) and Portugal, Kaussmann, M.; van Berkum, S., 2018, 16-2522, M-613602-01-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 139 days at ≤ -18°C soil: max. 221 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	barley	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 in all matrices 0.02 mg/kg for DFA (expressed as BYI 02960) in green material and grain 0.10 mg/kg for DFA (expressed as BYI 02960) in straw soil: 0.005 mg/kg for BYI 02960 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT/PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2522 16-2522-01 T-2A France 30000 Nîmes	Bare soil Barley Calcule	 1) 03.03.2017 2) 04.05.2017 – 15.05.2017 3) 19.06.2017 – 30.06.2017	SPI and incorporation	0.175	300	0.06	16.11.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 2nd rotation (PB 107 days)	0.020 <0.01 <0.01 0.018	 0.083 0.26 <0.1	 0.093 0.27 0.12	<0.005 0.028 0.088 <0.03	107 195 218 218
16-2522 16-2522-02 T-2A Portugal 2070-316 Ereira	Bare soil Barley Pewter	 1) 17.03.2017 2) 25.05.2017 – 07.06.2017 3) 01.07.2017 – 10.07.2017	SPI and incorporation	0.175	300	0.06	05.10.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 2nd rotation (PB 163 days)	0.035 0.013 <0.01 0.032	 0.13 0.37* 0.11	 0.15 0.38* 0.14	<0.005 0.045 0.12* 0.036	162 245 274 274
16-2522 16-2522-01 T-3A France 30000 Nîmes	Bare soil Barley Calcule	 1) 03.03.2017 2) 04.05.2017 – 15.05.2017 3) 19.06.2017 – 30.06.2017	SPI and incorporation	0.175	300	0.06	26.05.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 3rd rotation (PB 281 days)	0.018 <0.01 <0.01 <0.01	 0.074 0.17 <0.1	 0.084 0.18 <0.11	<0.005 0.025 0.056 <0.03	281 369 392 392

16-2522 16-2522-02 T-3A Portugal 2070-316 Ereira	Bare soil Barley Pewter	 1) 17.03.2017 2) 25.05.2017 – 07.06.2017 3) 01.07.2017 – 10.07.2017	SPI and incorporation	0.175	300	0.06	07.06.2016	 75 89 89	soil 0-20 cm [0-8 in] green material grain straw 3rd rotation (PB 283 days)	0.036 <0.01 <0.01 0.019	 0.081 0.26* <0.1	 0.091 0.27* 0.12	<0.005 0.027 0.088* <0.03	282 365 394 394

* Reduced amount of field sample due to draught. Amount of sample was sufficient for analysis. Due to the draught the residues of DFA seem to be slightly increased compared to the other trial.

A 2.1.6.1.6 Rotational crop study on maize, study report 16-2514

Comments of zRMS:

Two supervised residue trials were conducted in northern Europe (the Netherlands and Belgium) during the 2016/2017 season to determine the magnitude of the relevant residues of BYI 02960 in/on maize/corn grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 175 g a.s./ha.
Plant-back intervals were 131 to 167 days (2nd rotation) and 291 to 365 days (3rd rotation).
Samples of representative plant commodities of maize/corn (green material, rest of plant and kernel) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes were combined to the sum of BYI 02960 and difluoroacetic acid).
Soil samples were taken for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The individual LOQ for BYI 02960 is 0.01mg/kg in the sample materials of maize/corn, green material maize/corn, kernel maize/corn and rest of plant. The individual LOQ for difluoroacetic acid is 0.02 mg/kg in all of the maize/corn sample materials.
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.

The residue levels determined in the treated samples are summarized in the following table:

Table 22: Residues on soil and maize/corn samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA* (calculated as BYI 02960) [mg/kg] min - max	sum of BYI 02960 and DFA [mg/kg] min - max	DFA* (calculated as DFA) [mg/kg] min - max	6-CNA (calculated as 6-CNA) [mg/kg] min - max
maize/ corn	green material	T-2A	2	85	259-277	< 0.01	< 0.02 – 0.045	< 0.03 – 0.055	< 0.007 – 0.015	**
		T-3A			419-475	< 0.01	< 0.02 – 0.022	< 0.03 – 0.032	< 0.007 – 0.007	**
	kernel	T-2A	2	89	300-307	< 0.01	< 0.02 – 0.10	< 0.03 – 0.11	< 0.007 – 0.035	**
		T-3A			467-498	< 0.01	< 0.02 – 0.058	< 0.03 – 0.068	< 0.007 – 0.019	**
	rest of plant	T-2A	2	89	300-307	< 0.01	< 0.02 – 0.022	< 0.03 – 0.032	< 0.007 – 0.007	**
		T-3A			467-498	< 0.01	< 0.02	< 0.03	< 0.007	**
soil	0-20 cm [0-8 in]	T-2A	2	-	131- 167	0.029– 0.041	-	-	< 0.005	< 0.005
		T-3A			291 - 365	0.022 – 0.025	-	-	< 0.005	< 0.005

Residues of parent BYI 02960 were always <0.01 mg/kg in green material, kernel and rest of plant.
DFA residues expressed as DFA in green material collected from the second and third rotation ranged between <0.007-0.015 mg/kg, in kernel collected from the second and third rotation ranged between <0.007-0.035 mg/kg and in rest of plant - <0.007-0.019 mg/kg.
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were higher that the residues detected in the soil samples collected prior to the third rotation and ranged between 0.029 – 0.041 mg/kg prior to the second rotation and 0.022-0.025 mg/kg prior to the third rotation.

The study is acceptable.

Reference:	**KCA 6.6.2/06**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop maize/corn and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands
Report:	Freitag, T.; Effertz, C.; 2018; 16-2514; M-617942-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on maize (corn) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 131 to 167 days (2nd rotation) and 291 to 365 days (3rd rotation). The study included two supervised residue trials conducted in the field in northern Europe (the Netherlands and Belgium) during the 2016 season.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 0.875 L/ha (equating to 0.175 kg/ha active substance) and using a water rate of approximately 300 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate intended for BYI 02960 in Europe for agricultural uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping. A plant-back interval of 30 days (1st rotation) was not considered relevant for oilseed rape as such a short plant-back interval is not reasonable for a crop with a long vegetation period.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 157 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent).
Samples of the rotational crops were taken at their respective harvest times, as well as at one earlier interval ("green material" at BBCH growth stage 85: Dough stage: kernels yellowish to yellow (variety dependent), about 55% dry matter). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 140 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg in all plant matrices and for DFA 0.02 mg/kg (expressed as parent equivalent, corresponding to 0.0067 mg/kg or 0.03 mg/kg, respectively when expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Results and discussions

In order to evaluate the potential residues in maize grown as following crops, samples of green material were taken at an intermediate growth stage, as well as kernel and stover ("rest of plant") samples at maturity. The samples were analyzed for BYI 02960-related residues. Residues of parent BYI 02960 were <0.01 mg/kg in all sample materials collected from the Dutch trials, irrespective from the sampling time. Also the DFA residues were always below the LOQ (0.02 mg/kg, expressed as BYI 02960) for all sample materials at this trial site. Samples from the Belgian trial site showed DFA residues which were approx. by a factor of two higher in the samples collected from the 2nd rotation as compared to the samples of the 3rd rotation. The overall highest residues of DFA were detected in mature kernels from the 2nd rotation (0.035 mg/kg, expressed as DFA). DFA residues in green material and stover were quite low and were at or below the LOQ of 0.0067 mg/kg in samples collected from the 3rd rotation.

The corresponding soil samples collected prior to sowing the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.029-0.041 mg/kg. All BYI 02960 and DFA residues detected in matrices of maize grown as rotational crops and in the corresponding soil samples are summarized in Table A 57.

Parallel to the analysis of the plant matrices, concurrent (procedural) recoveries were conducted at the LOQ level and the 10x LOQ level for each analyte included in method 01304 in order to confirm the successful performance of the method during the conduct of the study. For the matrices relevant to this study (maize green material/stover and kernel), but not included in the original validation, a limited set (3 repetitions at two fortification levels) of additional validation recoveries were analyzed within the course of study 16-2524 which was conducted in parallel. Mean recoveries conducted for the validation and concurrent recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level (if applicable, $n \geq 3$) were well below 20%. The procedural recoveries are summarized in

Table A 55.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 56.

Table A 55: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in maize (green material (rest of plant) and kernel)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2514	maize	green material	BYI 02960	3	0.01	89; 93; 97	89	97	93	4.3
				3	0.10	90; 97; 102	90	102	96	6.3
(16-2514-01)				3	overall		89	102	96	8.2
and		kernel		3	0.01	87; 96; 101	87	101	95	7.5
(16-2514-02)				3	0.10	87; 104; 106	87	106	99	10.5
				6	overall		87	106	97	8.6
GLP: yes		stover (rest of plant)		3	0.01	90; 91; 103	90	103	95	7.6
2018				3	0.10	98; 100; 109	98	109	102	5.7
				6	overall				99	7.3
		green material	DFA	3	0.02	83; 87; 92	83	92	87	5.2
				3	0.20	86; 91; 94	86	94	90	4.5
				6	overall		83	94	89	4.7
		kernel		3	0.02	93; 101; 104	93	104	99	5.7
				3	0.20	84; 96; 97	84	97	92	7.8
				6	overall		84	104	96	7.3
		stover (rest of plant)		3	0.02	89; 89; 98	89	98	92	5.6
				3	0.20	88; 92; 92	88	92	91	2.5
				6	overall		88	92	91	4.0

Table A 56: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	BYI 02960	24	0.005	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79	70	92	82	7.2
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	72	88	81	5.9
GLP: yes 2018				46	overall		70	92	82	6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	DFA	24	0.005	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86	72	93	82	5.9
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	66	90	78	7.8
GLP: yes 2018				46	overall		66	93	80	7.3

Table A 57: Rotational trial summary for maize, study report 16-2514

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop maize/corn and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands, Freitag, T.; Effertz, C., 2018, 16-2514, M-617942-02-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 140 days at ≤ -18°C soil: max. 157 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	maize (corn)	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 in all matrices 0.02 mg/kg for DFA (expressed as BYI 02960) in all matrices soil: 0.005 mg/kg for BYI 02960 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT /PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2514 16-2514-01 T-2A The Netherlands 1681 ND Zwaagdijk	Bare soil Maize/corn Stacey	 1) 11.05.2017 2) 15.07.2017 – 15.08.2017 3) 20.09.2017 – 30.09.2017	SPI and incorporation	0.175	400	0.04	25.11.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 2nd rotation (PB 167 days)	0.041 <0.01 <0.01 <0.01	 <0.02 <0.02 <0.02	 <0.03 <0.03 <0.03	<0.005 <0.0067 <0.0067 <0.0067	167 277 300 300
16-2514 16-2514-02 T-2A Belgium 6221 Saint-Amand	Bare soil Maize/corn NK Gitago	 1) 24.04.2017 2) 15.07.2017 – 31.07.2017 3) 13.10.2017 – 26.10.2017	SPI and incorporation	0.175	300	0.06	14.12.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 2nd rotation (PB 131 days)	0.029 <0.01 <0.01 <0.01	 0.045 0.10 0.022	 0.055 0.11 0.032	<0.005 0.015 0.035 0.007	131 259 307 307
16-2514 16-2514-01 T-3A The Netherlands 1681 ND Zwaagdijk	Bare soil Maize/corn Stacey	 1) 11.05.2017 2) 15.07.2017 – 15.08.2017 3) 20.09.2017 – 30.09.2017	SPI and incorporation	0.175	400	0.04	11.05.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 3rd rotation (PB 365 days)	0.025 <0.01 <0.01 <0.01	 <0.02 <0.02 <0.02	 <0.03 <0.03 <0.03	<0.005 <0.0067 <0.0067 <0.0067	365 475 498 498
16-2514 16-2514-02 T-3A Belgium 6221 Saint-Amand	Bare soil Maize/corn NK Gitago	 1) 24.04.2017 2) 15.07.2017 – 31.07.2017 3) 13.10.2017 – 26.10.2017	SPI and incorporation	0.175	300	0.06	07.07.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 3rd rotation (PB 291 days)	0.022 <0.01 <0.01 <0.01	 0.022 0.058 <0.02	 0.032 0.068 <0.03	<0.005 0.007 0.019 <0.0067	291 419 467 467

A 2.1.6.1.7 Rotational crop study on maize/corn study report 16-2524

Comments of zRMS:

Two supervised residue trials were conducted southern Europe (Southern France and Portugal) during the 2016/2017 season to determine the magnitude of the relevant residues of BYI 02960 in/on maize/corn (green material, kernel and rest of plant) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 175 g a.s./ha.
Plant-back intervals were 139 to 204 days (2nd rotation) and 313 to 324 days (3rd rotation).
Samples of representative plant commodities of maize/corn (green material, kernel and rest of plant) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes combined to the sum of BYI 02960 and difluoroacetic acid).
Soil samples were taken prior to sowing the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The individual LOQ for BYI 02960 is 0.01mg/kg in the sample materials of maize/corn, green material maize/corn, kernel maize/corn and rest of plant. The individual LOQ for difluoroacetic acid is 0.02 mg/kg in all of the maize/corn sample materials.
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.

The residue levels determined in the treated samples are summarized in the following table:

Table 21: Residues in soil and maize/corn samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA (calculated as DFA) [mg/kg] min - max	sum of BYI 02960 and DFA (calculated as BYI 02960) [mg/kg] min - max	6-CNA (calculated as 6-CNA) [mg/kg] min - max
maize/corn	green material	T-2A	2	85	251 - 337	< 0.01 - 0.030	0.011 - 0.020	0.044 - 0.091	**
		T-3A			425 - 457	< 0.01 - 0.012	0.008 - 0.009	0.034 - 0.039	**
	kernel	T-2A	2	89	282 - 344	< 0.01	0.021 - 0.037	0.073 - 0.12	**
		T-3A			456 - 464	< 0.01	0.013 - 0.030	0.050 - 0.099	**
	rest of plant	T-2A	2	89	282 - 344	0.020	0.014 - 0.022	0.063 - 0.086	**
		T-3A			456 - 464	< 0.01 - 0.010	< 0.007 - 0.008	< 0.03 - 0.035	**
soil*	0-20 cm [0-8 in] plastic tube removed	T-2A	2	-	139 - 204	0.018 - 0.028	< 0.005	-	< 0.005
		T-3A			313 - 324	0.011 - 0.019	< 0.005	-	< 0.005

Residues of parent BYI 02960 were <0.01 mg/kg in kernel, and rest of plant. BYI 02960 residues in green material were detected and ranged between 0.012 mg/kg (3rd rotation) and 0.030 mg/kg (2nd rotation). BYI 02960 residues in rest of plant ranged between <0.010-0.020 mg/kg. DFA residues expressed as DFA in maize of the second rotation were higher than the DFA residues in maize of the third rotation. The highest DFA residues were detected in maize kernel and ranged between 0.021-0.037 mg/kg by the second rotation and 0.013-0.030 mg/kg by the third rotation. DFA residues in green material and rest of plant ranged between 0.011-0.022 mg/kg by the second rotation.
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were higher that the residues detected in the soil samples collected prior to the third rotation and ranged between 0.018 – 0.028 mg/kg prior to the second rotation and 0.011-0.019 mg/kg prior to the third rotation.

The study is acceptable.

Reference:	**KCA 6.6.2/07**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop maize/corn and soil after spray application of BYI 02960 SL 200 to bare soil in the field in France (South) and Portugal - Final report
Report:	Freitag, T.; van Berkum, S.; 2018; 16-2524; M-617943-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on maize (corn) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 139 to 204 days (2nd rotation) and 313 to 324 days (3rd rotation). The study included two supervised residue trials conducted in the field in southern Europe (Southern France and Portugal) during the 2016 / 2017 season.

One spray application of BYI 02960 SL 200 was made, either at the intended application rate of 0.875 L/ha (equating to 0.175 kg/ha active substance) or at a slightly higher rate of 0.932 L/ha (equating to 0.186 kg/ha active substance) and using a water rate of approximately 300 L/ha.

The intended application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate intended for BYI 02960 in Europe for agricultural uses. Application scenarios were investigated which simulate the re-use of a field after the harvest of a crop directly treated with BYI 02960, either in the same season (100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping. A plant-back interval of 30 days (1st rotation) was not considered relevant for oilseed rape as such a short plant-back interval is not reasonable for a crop with a long vegetation period.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 189 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent).
Samples of the rotational crops were taken at their respective harvest times, as well as at one earlier interval ("green material" at BBCH growth stage 85: Dough stage: kernels yellowish to yellow (variety dependent), about 55% dry matter). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 92 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ was 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA (expressed as parent equivalent, corresponding to 0.0067 mg/kg when expressed as analyte equivalent) for all plan materials. The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Results and discussions

In order to evaluate the potential residues in maize grown as following crops, samples of green material were taken at an intermediate growth stage, as well as kernel and "rest of plant" (= stover) samples at maturity. The samples were analyzed for BYI 02960-related residues. Residues of parent BYI 02960 were <0.01 mg/kg in all kernel samples, irrespective from the sampling time. BYI 02960 residues in green material were detected only at the Portuguese trial site and ranged between 0.012 mg/kg (3rd rotation) and 0.030 mg/kg (2nd rotation). BYI 02960 residues in stover samples were also rather low and ranged between <0.010-0.020 mg/kg. In contrast, almost all sample materials of both trial sites showed DFA residues. DFA residues in maize of the second rotation were always higher as compared to the DFA residues in maize of the third rotation. Highest DFA residues were detected in maize kernels (0.021-0.037 mg/kg by the 2nd rotation and 0.013-0.030 mg/kg by the 3rd rotation). DFA residues in green material and stover were comparable and ranged between 0.011-0.022 mg/kg (expressed as DFA) by the 2nd rotation.

The corresponding soil samples collected prior to sowing the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the third rotation and ranged between 0.018-0.028 mg/kg. All BYI 02960 and DFA residues detected in matrices of maize grown as rotational crops and in the corresponding soil samples are summarized in Table A 60.

Parallel to the residue analysis, analytical method 01304 was successfully validated for the relevant matrices of maize (green material/stover and kernel). As these sample matrices were not included in the original validation, a limited set (3 repetitions at the LOQ level and 3 at the 10x LOQ level) of additional validation recoveries were performed within the course of the study. These concurrent (procedural) recoveries were always in the acceptable range of 70-110%, and the RSD values per analyte and fortification level were always well below 20%, if applicable ($n \geq 3$). The recoveries are summarized in Table A 58.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 59.

Table A 58: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in maize/corn (green material (rest of plant), and kernel)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2524	maize	green material/ rest of plant*	BYI 02960	3	0.01	91; 92; 99	91	99	94	4.6
				3	0.10	70; 83; 93	70	93	82	14.1
(16-2524-02)				1	0.50	90	90	90	-	-
				7	overall		70	99	88	10.6
		kernel		3	0.01	107; 109; 110	107	110	109	1.4
GLP: yes				3	0.10	97; 102; 104	97	104	101	3.6
2018				1	0.50	102	102	102	-	-
				7	overall		97	110	104	4.4
		green material/ rest of plant*	DFA	3	0.02	83; 90; 94	83	94	89	6.3
				3	0.20	85; 87; 91	85	91	88	3.5
				1	1.0	87	87	87	-	-
				7	overall		83	94	88	4.3
		kernel		3	0.02	107; 108; 109	107	109	108	0.9
				3	0.20	85; 88; 89	85	89	87	2.4
				1	1.0	88	88	88	-	-
				7	overall		85	109	96	11.5

* The recoveries for the sample material green material are also representative for sample material rest of plant (= stover) and vice versa

Table A 59: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	BYI 02960	24	0.005	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79	70	92	82	7.2
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	72	88	81	5.9
GLP: yes 2018				46	overall		70	92	82	6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	DFA	24	0.005	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86	72	93	82	5.9
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	66	90	78	7.8
GLP: yes 2018				46	overall		66	93	80	7.3

Table A 60: Rotational trial summary for maize/corn, study report 16-2524

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop Maize/Corn and Soil after spray application of BYI 02960 SL 200 to bare soil in the field in France (South), Freitag, van Berkum, S., 2018, 16-2524, M-617943-01-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 92 days at ≤ -18°C soil: max. 189 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	maize (corn)	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 in all matrices 0.02 mg/kg for DFA (expressed as BYI 02960) in all matrices soil: 0.005 mg/kg for BYI 02960 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2524 16-2524-01 T-2A France, south 30000 Nîmes	Bare soil Maize/corn Capuzi CS	 1) 04.04.2017 2) 26.06.2017 – 06.07.2017 3) 22.08.2017 – 09.09.2017	SPI and incorporation	0.175	300	0.06	16.11.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 2nd rotation (PB 139 days)	0.018 <0.01 <0.01 0.020	 0.034 0.063 0.043	 0.044 0.073 0.063	<0.005 0.011 0.021 0.014	139 251 282 282
16-2524 16-2524-02 T-2A Portugal 2070-316 Ereira	Bare soil Maize/corn Sangria	 1) 27.04.2017 2) 11.07.2017 – 31.08.2017 3) 15.09.2017 – 30.09.2017	SPI and incorporation	0.186	319	0.06	05.10.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 2nd rotation (PB 204 days)	0.028 0.030 <0.01 0.020	 0.061 0.11 0.066	 0.091 0.12 0.086	<0.005 0.020 0.037 0.022	204 337 344 344
16-2524 16-2524-01 T-3A France, south 30000 Nîmes	Bare soil Maize/corn Capuzi CS	 1) 04.04.2017 2) 26.06.2017 – 06.07.2017 3) 22.08.2017 – 09.09.2017	SPI and incorporation	0.175	300	0.06	26.05.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 3rd rotation (PB 313 days)	0.011 <0.01 <0.01 <0.01	 0.024 0.040 <0.02	 0.034 0.050 <0.03	<0.005 0.008 0.013 <0.0067	313 425 456 456
16-2524 16-2524-02 T-3A Portugal 2070-316 Ereira	Bare soil Maize/corn Sangria	 1) 27.04.2017 2) 11.07.2017 – 31.08.2017 3) 15.09.2017 – 30.09.2017	SPI and incorporation	0.175	300	0.06	07.06.2016	 85 89 89	soil 0-20 cm [0-8 in] green material kernel rest of plant 3rd rotation (PB 324 days)	0.019 0.012 <0.01 0.010	 0.026 0.089 0.025	 0.039 0.099 0.035	<0.005 0.009 0.030 0.008	324 457 464 464

A 2.1.6.1.8 Rotational crop study on strawberry, study report 16-2515

Comments of zRMS:

Two supervised residue trials were conducted in northern Europe (the Netherlands and Belgium) during the seasons of 2016 and 2017 to determine the magnitude of the relevant residues of BYI 02960 in/on strawberry grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 300 g a.s./ha.
Plant-back intervals were 22 days (1st rotation), 139 and 141 days (2nd rotation) and 301 and 337 days (3rd rotation).
Samples of representative plant commodities (strawberry fruit) were taken from each of the subplots and analyzed for the residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes combined to the total residue BYI 02960 calc 3.)
Soil samples were taken at the same day of planting the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The LOQ for BYI 02960 is 0.01mg/kg in the fruit sample material of strawberry, the LOQ for difluoroacetic acid is 0.02 mg/kg.
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.
No residues above the LOQ were found in the control samples.

The residue levels determined in the treated samples are summarized in the following table:

Table 22: Residues in soil and on strawberry samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA (calculated as BYI 02960) [mg/kg] min - max	sum of BYI 02960 and DFA [mg/kg] min - max	DFA (calculated as DFA) [mg/kg] min - max	6-CNA (calculated as 6-CNA) [mg/kg] min - max
straw-berry	fruit	T-1A	2	85 - 87	70 - 78	< 0.01	0.17 - 0.30	0.18 - 0.31	0.058 - 0.098	"
		T-2A			196 - 210	< 0.01	0.12 - 0.37	0.13 - 0.38	0.040 - 0.12	"
		T-3A			356 - 408	< 0.01	0.069 - 0.23	0.079 - 0.24	0.023 - 0.076	"
soil	0-20 cm [0-8 in]	T-1A	2	-	22	0.034 - 0.059	-	-	< 0.005	< 0.005
		T-2A			139 - 141	0.052 – 0.086	-	-	< 0.005	< 0.005
		T-3A			301 - 337	0.030 - 0.055	-	-	< 0.005	< 0.005

Residues of parent BYI 02960 were always <0.01 mg/kg in strawberry samples. DFA residues were detected in all three rotations. The highest DFA residue expressed as DFA was detected in a fruit sample of the second rotation and equaled 0.12 mg/kg.
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were higher that the residues detected in the soil samples collected prior to the first and third rotation and ranged between 0.034 – 0.059 mg/kg prior to the first rotation, 0.052 – 0.086 mg/kg prior to the second rotation and 0.030 – 0.055 mg/kg prior to the third rotation.

The study is acceptable.

Reference:	**KCA 6.6.2/08**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop strawberry and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands
Report:	Freitag, T.; Hoffmeister, R.; 2018; 16-2515; M-617563-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on strawberries grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 22 days (1st rotation), 139 and 141 days (2nd rotation) and 301 and 337 days (3rd rotation). The study included two supervised residue trials conducted in the field in northern Europe (the Netherlands and Belgium) during the seasons of 2016 and 2017.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 1.5 L/ha (equating to approx. 0.3 kg/ha active substance) and using a water rate of approximately 300 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate registered for BYI 02960 in Europe for home and garden uses. Application scenarios were investigated in which an original crop was destroyed (crop failure) or harvested and the field was used again thereafter, in the same season (20-30 days after the initial application = 1st rotation and 100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 379 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent).

Samples of the rotational crops were taken at the earliest possible harvest time (BBCH growth stage 85: First fruits have cultivar-specific colour), and at the main harvest (BBCH growth stage 87: Main harvest: more fruits coloured). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 433 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg in all plant matrices and for DFA 0.02 mg/kg (expressed as parent equivalent, corresponding to 0.0067 mg/kg or 0.03 mg/kg, respectively when expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Results and discussions

In order to evaluate the potential residues in strawberries grown as following crops, fruits were collected at two sampling times (early and main harvest). The samples were analyzed for BYI 02960-related residues. Residues of BYI 02960 were always <0.01 mg/kg in strawberry fruits, irrespective from the sampling time. DFA residues were detected in all three rotations. The highest DFA residue was detected in a fruit sample of the second rotation and accounted for 0.12 mg/kg (expressed as DFA). The highest median DFA residue was calculated from the samples of the first rotation (0.068 mg/kg). Samples collected at an early harvest time (BBCH 85) and samples collected at main harvest (BBCH 87) showed comparable residues. DFA residues in strawberry fruit ranged between 0.023-0.12 mg/kg (expressed as DFA).

~~The corresponding soil samples collected prior to sowing the crop for the individual rotations showed also neither BYI 02960 nor DFA residues above the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents).~~ The corresponding soil samples collected prior to sowing the crop for the individual rotations showed no DFA residues above the respective LOQs of 0.005 mg/kg (expressed as analyte equivalent). All BYI 02960 and DFA residues detected in matrices of strawberry grown as rotational crops and in the corresponding soil samples are summarized in Table A 60Table A 63.

Parallel to the residue analysis, analytical method 01304 was successfully validated for the relevant matrix strawberry fruit. As this sample matrix was not included in the original validation, a limited set (3 repetitions at the LOQ level and 3 at the 10x LOQ level) of additional validation recoveries were performed within the course of the study. These concurrent (procedural) recoveries were always in the acceptable range of 70-110%, and the RSD values per analyte and fortification level were always well below 20%, if applicable ($n \geq 3$). The recoveries (including the concurrent recovery conducted at the 100x LOQ level) are summarized in Table A 61.

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 62.

Table A 61: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in strawberry (fruit)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2515	straw-berry	fruit	BYI 02960	3	0.01	98; 105; 106	98	106	103	4.2
				3	0.10	101; 103; 105	101	105	103	1.9
(16-2515-01)				1	1.0	101	101	101	-	-
				7	overall		98	106	103	2.8
		fruit	DFA	3	0.02	101; 103; 108	101	108	104	3.5
GLP: yes 2018				3	0.20	96; 97; 99	96	99	97	1.6
				1	2.0	93	93	93	-	-
				7	overall		93	108	100	5.0

Table A 62: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	BYI 02960	24 22 46	0.005 0.05 overall	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79 72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	70 72 70	92 88 92	82 81 82	7.2 5.9 6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514* *16-2524* *16-2515* *16-2525* *16-2516* GLP: yes 2018	soil	0-20 cm	DFA	24 22 46	0.005 0.05 overall	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86 66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	72 66 66	93 90 93	82 78 80	5.9 7.8 7.3

Table A 63: Rotational trial summary for strawberry, study report 16-2515

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop strawberry and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium and the Netherlands, Freitag, T.; Hoffmeister, R., 2018, 16-2515, M-617563-01-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 433 days at ≤ -18°C soil: max. 379 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	strawberry (fruit)	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 0.02 mg/kg for DFA (expressed as BYI 02960) soil: 0.005 mg/kg for BYI 02960 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2515 16-2515-01 T-1A The Netherlands 1681 ND Zwaagdijk	Bare soil Strawberry Felicita Frigo	1) 02.06.2016 2) 22.06.2016 – 01.08.2016 3) 22.07.2016 – 01.08.2016	SPI and incorporation	0.300	400	0.075	11.05.2016	 85 87	soil 0-20 cm [0-8 in] fruit 1st rotation (PB 22 days)	<0.005 0.034 0.034 <0.01 <0.01	 0.17 0.21	 0.18 0.22	<0.005 0.058 0.068	22 72 78
16-2515 16-2515-02 T-1A Belgium 6221 Saint-Amand	Bare soil Strawberry Elsanta	1) 26.05.2016 2) 06.06.2016 – 19.06.2016 3) 15.07.2016 – 26.07.2016	SPI and incorporation	0.300	300	0.1	04.05.2016	 85 87	soil 0-20 cm [0-8 in] fruit 1st rotation (PB 22 days)	<0.005 0.059 0.059 <0.01 <0.01	 0.25 0.30	 0.26 0.31	<0.005 0.082 0.098	22 70 77
16-2515 16-2515-01 T-2A The Netherlands 1681 ND Zwaagdijk	Bare soil Strawberry Rumba Frigo	1) 13.04.2017 2) 15.05.2017 – 15.06.2017 3) 19.06.2017 – 29.09.2017	SPI and incorporation	0.300	400	0.075	25.11.2016	 85 87	soil 0-20 cm [0-8 in] fruit 2nd rotation (PB 139 days)	<0.005 0.053 0.051 <0.01 <0.01	 0.12 0.14	 0.13 0.15	<0.005 0.040 0.046	139 206 210
16-2515 16-2515-02 T-2A Belgium 6221 Saint-Amand	Bare soil Strawberry Sonata	1) 04.05.2017 2) 31.05.2017 – 10.06.2017 3) 01.07.2017 – 12.07.2017	SPI and incorporation	0.300	300	0.1	14.12.2016	 85 87	soil 0-20 cm [0-8 in] fruit 2nd rotation (PB 141 days)	<0.005 0.086 0.087 <0.01 <0.01	 0.37 0.37	 0.38 0.38	<0.005 0.12 0.12	141 196 203
16-2515 16-2515-01 T-3A The Netherlands 1681 ND Zwaagdijk	Bare soil Strawberry Rumba Frigo	1) 13.04.2017 2) 15.05.2017 – 15.06.2017 3) 19.06.2017 – 29.09.2017	SPI and incorporation	0.300	400	0.075	11.05.2016	 85 87	soil 0-20 cm [0-8 in] fruit 3rd rotation (PB 337 days)	<0.005 0.030 0.031 <0.01 <0.01	 0.073 0.069	 0.083 0.079	<0.005 0.024 0.023	337 404 408

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analyzed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2515 16-2515-02 T-3A Belgium 6221 Saint-Amand	Bare soil Strawberry Sonata	 1) 04.05.2017 2) 31.05.2017 – 10.06.2017 3) 01.07.2017 – 12.07.2017	SPI and incorporation	0.317	317	0.1	07.07.2016	 85 87	soil 0-20 cm [0-8 in] fruit 3rd rotation (PB 301 days)	~~<0.005~~ 0.055 0.055 <0.01 <0.01	 0.078 0.23	 0.088 0.24	<0.005 0.026 0.076	301 356 363

A 2.1.6.1.9 Rotational crop study on strawberry, study report 16-2525

Comments of zRMS:

Two supervised residue trials were conducted in southern Europe (Spain and Italy) during the seasons of 2016 and 2017 to determine the magnitude of the relevant residues of BYI 02960 in/on strawberry grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 300 g a.s./ha.
Three plant-back intervals were tested. Actual plant-back intervals were 27-29 days (1st rotation), 133-158 days (2nd rotation) and 341-363 days (3rd rotation). In addition soil samples were collected at all plant-back intervals for analysis.
Samples of representative plant commodities (strawberry fruit) were taken from each of the subplots and analyzed for the residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid.
Soil samples were taken at the same day of planting the crop for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The LOQ for BYI 02960 is 0.01mg/kg in the fruit sample material of strawberry, the LOQ for difluoroacetic acid is 0.02 mg/kg.
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.
No residues above the LOQ were found in the control samples.

The residue levels determined in the treated samples are summarized in the following table:

Table 22: Residues in strawberry and soil samples after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA (calculated as BYI 02960) [mg/kg] min - max	sum of BYI 02960 and DFA (calculated as BYI 02960) [mg/kg] min - max	DFA (calculated as DFA) [mg/kg] min - max	6-CNA (calculated as 6-CNA) [mg/kg] min - max
strawberry	fruit	T-1A	2	85-87	133 - 224	< 0.01	< 0.02 - 0.36	< 0.03 - 0.37	< 0.007 - 0.12	**
		T-2A		85-87	188 - 355	< 0.01	< 0.02 - 0.21	< 0.03 - 0.22	< 0.007 - 0.068	**
		T-3A		85-87	481 - 543	< 0.01	< 0.02 - 0.051	< 0.03 - 0.061	< 0.007 - 0.017	**
soil*	0-20 cm [0-8 in] plastic tube removed	T-1A	2	-	27 - 29	0.008 - 0.053	-	-	< 0.005	< 0.005
		T-2A			134 - 158	0.022 - 0.067	-	-	< 0.005	< 0.005
		T-3A			341 - 364	0.007 - 0.039	-	-	< 0.005	< 0.005

Residues of parent BYI 02960 were always <0.01 mg/kg in strawberry samples. DFA residues were detected in several samples in all three rotations. The highest DFA residue expressed as DFA was detected in a fruit sample of the first rotation and equaled 0.12 mg/kg.
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the second rotation were slightly higher that the residues detected in the soil samples collected prior to the first and third rotation and ranged between 0.008 – 0.053 mg/kg prior to the first rotation, 0.022 – 0.067 mg/kg prior to the second rotation and 0.007 – 0.039 mg/kg prior to the third rotation.

The study is acceptable.

Reference:	**KCA 6.6.2/09**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop strawberry and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Spain and Italy
Report:	Freitag, T.; van Berkum, S.; 2018; 16-2525; M-617923-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on strawberries grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. The study included two supervised residue trials conducted in southern Europe (Spain and Italy) during the seasons of 2016 and 2017. Investigations of three plant-back intervals have been planned. Field and analytical data of the Spanish trial site (16-2525-01) are available for the first and second rotation. The plant-back intervals were 27-29 days (1st rotation) and 133-158 days (2nd rotation) and 270-341 days (3rd rotation).

One spray application of BYI 02960 SL 200 was made at an intended application rate of 1.5 L/ha (equating to 0.3 kg/ha active substance) and using a water rate of approximately 300 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate registered for BYI 02960 in Europe for home and garden uses. Application scenarios were investigated in which an original crop was destroyed (crop failure) or harvested and the field was used again thereafter, in the same season (20-30 days after the initial application = 1st rotation and 100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping.

After spray application, the test item was incorporated into the soil. Prior to planting strawberries for the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 340 days) until analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Samples of the rotational crops were taken at the earliest possible harvest time (BBCH growth stage 85: First fruits have cultivar-specific colour), and at the main harvest (BBCH growth stage 87: Main harvest: more fruits coloured). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 266 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg in all plant matrices and for DFA 0.02 mg/kg (expressed as parent equivalent, corresponding to 0.0067 mg/kg or 0.03 mg/kg, respectively when expressed as analyte equivalent).

Results and discussions

In order to evaluate the potential residues in strawberries grown as following crops, samples were taken at an early harvest time (BBCH growth stage 85) and at main harvest (BBCH 87) and were analyzed for BYI 02960-related residues. Residues of BYI 02960 were always <0.01 mg/kg in strawberry fruits whereas DFA residues were detected in several samples. Highest DFA residues (up to 0.12 mg/kg) were detected in mature fruits collected in the first rotation. Generally, the samples collected from the Italian trial showed higher DFA residues as compared to the samples from the Spanish trial. Samples of the third rotation showed very low or no residues above the LOQ.

The corresponding soil samples collected prior to planting the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the second rotation were slightly higher than the residues detected in the soil samples collected prior to the first rotation and amounted to 0.022-0.067 mg/kg. These residues declined and ranged finally between 0.007-0.039 mg/kg prior to the 3rd rotation.
BYI 02960 and DFA residues detected in strawberry fruits grown as rotational crops and in the corresponding soil samples are summarized in Table A 66.

Parallel to the analysis of the plant matrices, concurrent (procedural) recoveries were conducted at the LOQ level and the 100x LOQ level for each analyte included in method 01304 in order to confirm the successful performance of the method during the conduct of the study. For the matrices relevant to this study (strawberry fruit), but not included in the original validation, a limited set (3 repetitions at two fortification levels) of additional validation recoveries were analyzed within the course of study 16-2515 which was conducted in parallel. Mean recoveries conducted for the validation and concurrent recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level (if applicable, $n \geq 3$) were well below 20%. The procedural recoveries are summarized in Table A 64.

Concurrent recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 65.

Table A 64: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in strawberry (fruit)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2525	straw-berry	fruit	BYI 02960	4	0.01	91; 94; 94; 95	91	95	94	1.9
				3	0.1	94; 98; 102	95	102	98	4.1
(16-2525-01)				1	1.0	99	99	99		-
				8	overall		91	102	96	3.7
			DFA	4	0.02	94; 97; 99; 100	94	100	98	2.7
GLP: yes 2018				3	0.2	99; 101; 104	99	104	101	2.5
				1	2.0	98	98	98		-
				8	overall		97	98	99	3.0

Table A 65: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	BYI 02960	24	0.005	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79	70	92	82	7.2
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	72	88	81	5.9
GLP: yes 2018				46	overall		70	92	82	6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	DFA	24	0.005	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86	72	93	82	5.9
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	66	90	78	7.8
GLP: yes 2018				46	overall		66	93	80	7.3

Table A 66: Rotational trial summary for strawberry, study report 16-2525

Reference:	Determination of the residues of BYI 02960 in/on potato and soil after Spraying of BYI 02960 SL 200 in the Field in Germany, the Netherlands, Italy and Spain, Kaussmann, M., Freitag, T., 2018, 16-2525, M-617923-02-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 266 days at ≤ -18°C soil: max. 340 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	strawberry	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 and 0.02 mg/kg for DFA (expressed as BYI 02960) soil: 0.005 mg/kg for BYI 0296 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT /PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2525 16-2525-01 T-1A Spain 11140 Conil de la Frontera	Bare soil Strawberry San Andrea	 1) 04.11.2016 2) 01.01.2017 – 30.03.2017 3) 14.02.2017 – 30.03.2017	SPI and incorporation	0.300	300	0.1	06.10.2016	 85 87	soil[1)] 0-20 cm [0-8 in] fruit 1st rotation (PB 29 days)	0.053 <0.01 <0.01	 0.027 <0.02	 0.037 <0.03	<0.005 0.009 <0.0067	29 133 175
16-2525 16-2525-02 T-1A Italy 74019 Palagiano (TA)	Bare soil Strawberry	 1) 17.10.2017 2) 01.04.2018 – 31.05.2018 3) 19.04.2018 – 31.05.2018	SPI and incorporation	0.300	300	0.1	20.09.2017	 85 87	soil 0-20 cm [0-8 in] fruit 1st rotation (PB 27 days)	0.008 <0.01 <0.01	 0.23 0.36	 0.24 0.37	<0.005 0.078 0.12	27 211 224
16-2525 16-2525-01 T-2A Spain 11140 Conil de la Frontera	Bare soil Strawberry San Andrea	 1) 16.02.2017 2) 13.03.2017 – 20.06.2017 3) 04.04.2017 – 20.06.2017	SPI and incorporation	0.300	300	0.1	06.10.2016	 85 87	soil[1)] 0-20 cm [0-8 in] fruit 2nd rotation (PB 133 days)	0.067 <0.01 <0.01	 <0.02 0.035	 <0.03 0.045	<0.005 <0.0067 0.012	134 188 242
16-2525 16-2525-02 T-2A Italy 74019 Palagiano (TA)	Bare soil Strawberry	 1) 17.10.2017 2) 01.04.2018 – 31.05.2018 3) 19.04.2018 – 31.05.2018	SPI and incorporation	0.300	300	0.1	12.05.2017	 85 87	soil 0-20 cm [0-8 in] fruit 2nd rotation (PB 158 days)	0.022 <0.01 <0.01	 0.21 0.18	 0.22 0.19	<0.005 0.068 0.059	158 342 355

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT /PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2525 16-2525-01 T-3A Spain 11140 Conil de la Frontera	Bare soil Strawberry San Andrea	 1) 04.10.2017 2) 15.12.2017 – 30.05.2018 3) 01.02.2018 – 30.05.2018	SPI and incorporation	0.300	300	0.1	06.10.2016	 85 87	soil[1)] 0-20 cm [0-8 in] fruit 3rd rotation (PB 270 days)	0.039 <0.01 <0.01	 <0.02 <0.02	 <0.03 <0.03	<0.005 <0.0067 <0.0067	364 481 543
16-2525 16-2525-02 T-3A Italy 74019 Palagiano (TA)	Bare soil Strawberry	 1) 17.10.2017 2) 01.04.2018 – 31.05.2018 3) 19.04.2018 – 31.05.2018	SPI and incorporation	0.300	300	0.1	10.11.2016	 85 87	soil 0-20 cm [0-8 in] fruit 3rd rotation (PB 341 days)	0.007 <0.01 <0.01	 0.051 <0.02	 0.061 <0.03	<0.005 0.017 <0.0067	341 525 538

[1)] The field sample collected was 0-20 cm instead of 0-30 cm due to soil aggregation during the sampling process, therefore the whole field sample was used to prepare the examination sample. The residues can only seen as indicative residues as the sample is most probably not representative for the upper soil layer of 0-20 cm.

A 2.1.6.1.10 Rotational crop study on brassica vegetables, study report 16-2516

Comments of zRMS:

Four supervised residue trials were conducted in northern Europe (the Netherlands and Belgium) and in southern Europe (Italy and Spain) during the seasons of 2016 and 2017 to determine the magnitude of the relevant residues of BYI 02960 in/on cauliflower or broccoli grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200 at rates of 300 g a.s./ha.
Plant-back intervals were 21 to 30 days (1st rotation), 129 to 161 days (2nd rotation) and 321 to 339 days (3rd rotation).
Samples of representative plant commodities (cauliflower, curd and broccoli, curd) were taken from each of the subplots and analyzed for the relevant residues of BYI 02960, comprising the parent compound BYI 02960 and difluoroacetic acid (both analytes were combined to the sum of BYI 02960 and difluoroacetic acid).
Soil samples were taken for each individual rotation from each of the subplots and were analyzed for the residues of BYI 02960 and its metabolites difluoroacetic acid and 6-chloronicotinic acid.
The LOQ for BYI 02960 is 0.01mg/kg in cauliflower and broccoli (curd) samples, the LOQ for difluoroacetic acid is 0.02 mg/kg.
The LOQ values for BYI 02960, its metabolites difluoroacetic acid and 6-chloronicotinic acid in the sample material soil are 0.005 mg/kg each.
The average recoveries were within the acceptable range of 70 – 110% for each analyte and the RSD values were below 20%.
No residues above the LOQ were found in the control samples.

The residue levels determined in the treated samples are summarized in the following table:

Table 26: Residues on cauliflower, broccoli and soil after application of BYI 02960 SL 200

Sample material		Plot	No of trials	Growth stage [BBCH]	DAT	BYI 02960 [mg/kg] min - max	DFA* (calculated as BYI 02960) [mg/kg] min - max	sum of BYI 02960 and DFA [mg/kg] min - max	DFA* (calculated as DFA) [mg/kg] min - max	6-CNA (calculated as 6-CNA) [mg/kg] min - max
cauliflower & broccoli	curd	T-1A	4	45 – 49	86 - 130	< 0.01	0.062 – 0.20	0.072 – 0.21	0.021 – 0.065	**
		T-2A		45 – 49	221 - 244	< 0.01	0.036 - 0.18	0.046 – 0.19	0.012 – 0.061	**
		T-3A		45 - 49	389 - 442	< 0.01	0.021 – 0.12	0.031 – 0.13	0.007 – 0.041	**
soil	0-20 cm [0-8 in]	T-1A	4	-	22 - 30	0.046 - 0.10	-	-	< 0.005	< 0.005
		T-2A			130 - 161	0.039 - 0.074	-	-	< 0.005	< 0.005
		T-3A			321 - 342	0.019 - 0.064	-	-	< 0.005	< 0.005

Residues of parent BYI 02960 were always <0.01 mg/kg in cauliflower and broccoli samples. DFA residues were detected in all samples in all three rotations. The highest DFA residue expressed as DFA was detected in curd sample of the first rotation and equaled 0.065 mg/kg.
Soil samples were taken prior to sowing the crop for each individual rotation. The residues of DFA were always below the LOQ of 0.005 mg/kg. Residues of BYI 02960 in soil collected before starting the first rotation were higher that the residues detected in the soil samples collected prior to the second and third rotation and ranged between 0.046 – 0.10 mg/kg prior to the first rotation, 0.039 – 0.074 mg/kg prior to the second rotation and 0.019 – 0.064 mg/kg prior to the third rotation.

The study is acceptable.

Reference:	**KCA 6.6.2/10**
Title:	Determination of the residues of BYI 02960 in/on the field rotational crop cauliflower, broccoli and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium, the Netherlands, Italy and Spain
Report:	Freitag, T.; Effertz, C.; 2018; 16-2516; M-617941-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guidelines for the Testing of Chemicals. Residues in Rotational Crops (Limited Field Studies). 504. 2007-01-08 OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1900, Field Accumulation in Rotational Crops US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of the study was to determine the magnitude of the relevant residues of BYI 02960 (flupyradifurone) in/on brassica vegetables (cauliflower and broccoli have been selected as representative crops) grown as a rotational crop after one spray application to bare soil with BYI 02960 SL 200, a soluble liquid concentrate (SL) formulation containing 200 g/L BYI 02960. Plant-back intervals were 21 to 30 days (1st rotation), 129 to 161 days (2nd rotation) and 321 to 339 days (3rd rotation). The study included four supervised residue trials conducted in northern Europe (the Netherlands and Belgium) and in southern Europe (Italy and Spain) during the seasons of 2016 and 2017.

One spray application of BYI 02960 SL 200 was made at an intended application rate of 1.5 L/ha (equating to 0.3 kg/ha active substance) and using a water rate of approximately 300 - 400 L/ha.

The application rate was calculated by adding the estimated mean plateau (background) concentration detected after multi-year use of BYI 02960 (assuming crop rotation and therefore different application rates in consecutive years) to the overall maximum seasonal application rate registered for BYI 02960 in Europe for home and garden uses. Application scenarios were investigated in which an original crop was destroyed (crop failure) or harvested and the field was used again thereafter, in the same season (20-30 days after the initial application = 1st rotation and 100-180 days after the initial application = 2nd rotation) or in the following season (270-365 days = 3rd rotation). As such, the use patterns used reflected a typical use in a non-perennial crop followed by a defined interval for re-cropping.

After the spray application, the test item was incorporated into the soil. Prior to sowing the rotational crop of the respective rotation, soil samples were taken from the different subplots. The samples were stored deep-frozen (in maximum 372 days) prior to analysis according to method 01074/M001. The relevant analytes were BYI 02960 (flupyradifurone) and its metabolites difluoroacetic acid and 6-chloronicotinic acid. The LOQs were 0.005 mg/kg for each analyte (expressed as analyte equivalent). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 52 months.

Samples of the rotational crops were taken at their respective harvest times (BBCH growth stage 49: Typical size and form reached; head tightly closed), and as well as at one earlier interval (immature RAC at BBCH growth stage 45: 50% of the expected head diameter reached). The samples were stored deep-frozen (≤ -18°C) prior to homogenization and until analysis (in maximum 462 days). The samples were analyzed for the parent compound and its metabolite DFA according to method 01304. The LOQ for BYI 02960 was 0.01 mg/kg in the cauliflower and broccoli curd and for DFA 0.02 mg/kg (expressed as parent equivalent, corresponding to 0.0067 mg/kg when expressed as analyte equivalent).

Results and discussions

In order to evaluate the potential residues in brassica vegetables grown as following crops, cauliflower and broccoli curds were collected at two sampling times (immature RAC and harvest). The samples were analyzed for BYI 02960-related residues. Residues of BYI 02960 were in all cauliflower and broccoli samples and at all sampling times <0.01 mg/kg, whereas DFA residues were detected in all samples. DFA residues were highest in the samples collected from the first rotation and decreased continuously with time. By the 3rd rotation, DFA residues ranged between 0.007-0.041 mg/kg (expressed as DFA). Residues in the immature RACs were in the same range as the residues in the mature samples. Overall, DFA residues of the 1st rotation ranged between 0.021 mg/kg and 0.065 mg/kg (expressed as DFA).

The corresponding soil samples collected prior to planting the crop for the individual rotations showed only BYI 02960 residues; DFA and 6-CNA residues were always below the respective LOQs of 0.005 mg/kg (expressed as analyte equivalents). BYI 02960 residues in soil collected before starting the first rotation were higher than the residues detected in the soil samples collected prior to the second and third rotation and ranged between 0.046-0.10 mg/kg.

BYI 02960 and DFA residues detected in potato tubers grown as rotational crops and in the corresponding soil samples are summarized in Table A 70.

Parallel to the analysis of the plant matrices, concurrent (procedural) recoveries were conducted at the LOQ level, 10x LOQ level, 50x LOQ level and the 200x LOQ level for BYI 02960 and at the LOQ level, 10x LOQ level, 50x LOQ level and 200x LOQ level for the metabolite DFA in order to confirm the successful performance of the method during the conduct of the study. For the matrices relevant to this study (cauliflower and broccoli curd), but not included in the original validation, a limited set (3 repetitions at two fortification levels) of additional validation recoveries were performed within the course of the study and also within study 16-2147 which was conducted in parallel. Mean recoveries conducted for the validation and concurrent recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level (if applicable, n ≥ 3) were well below 20%. The procedural recoveries of the present study are summarized in Table A 67 (cauliflower) and Table A 68 (broccoli).

Concurrent (procedural) recoveries were also conducted in parallel to the soil analysis. Control samples were fortified at the LOQ level and the 10x LOQ level for each analyte in order to confirm the successful performance of the method. The recoveries were performed during the conduct of the field rotational studies 16-2510, 16-2511, 16-2512, 16-2513, 16-2514, 16-2515, 16-2516, 16-2521, 16-2522, 16-2524, 16-2525 which were all analyzed in parallel. Mean recoveries were always in the acceptable range of 70-110%, and the RSD values per fortification level were well below 20%. All results are summarized in Table A 69.

Table A 67: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in cauliflower (curd)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2516 (16-2516-01) and (16-2516-02) GLP: yes 2018	cauliflower	curd	BYI 02960	3 3 6	0.01 0.50 overall	91; 98; 99 92; 97; 101	91 92 91	99 101 101	96 97 96	4.5 4.7 4.1
		curd	DFA	3 3 6	0.02 1.0 overall	93; 101; 109 95; 96; 97	93 95 93	109 97 109	101 96 99	7.9 1.0 5.9

Table A 68: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in broccoli (curd)

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2516	broccoli	curd	BYI 02960	3	0.01	96; 98; 102	96	102	99	3.1
				3	0.10	97; 91; 100	97	100	96	4.8
(16-2516-03)				1	2.0	90	90	90	-	-
				7	overall		90	102	96	4.6
GLP: yes 2018			DFA	3	0.02	107; 109; 112	107	112	109	2.3
				3	0.20	95; 100; 103	95	103	99	4.1
				1	4.0	106	106	106	-	-
				7	overall		95	112	105	5.5

Table A 69: Procedural recoveries for flupyradifurone (BYI 02960) and DFA in soil (0-20 cm)

Study Trial No. Plot No. GLP Year	Matrix	Portion analyzed	a.s./ metabolite	n	Fortifi-cation level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	BYI 02960	24	0.005	70; 70; 75; 75; 75; 83; 84; 84; 84; 86; 87; 87; 89; 79; 81; 81; 81; 85; 85; 87; 88; 89; 92; 79	70	92	82	7.2
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	72; 73; 74; 74; 76; 77; 79; 82; 82; 83; 85; 85; 85; 80; 82; 83; 84; 84; 85; 87; 88; 78	72	88	81	5.9
GLP: yes 2018				46	overall		70	92	82	6.6
16-2510 *16-2511* *16-2521* *16-2512* *16-2522* *16-2514*	soil	0-20 cm	DFA	24	0.005	72; 76; 76; 79; 79; 80; 81; 81; 82; 84; 84; 87; 89; 76; 78; 79; 81; 82; 84; 84; 85; 89; 93; 86	72	93	82	5.9
16-2524 *16-2515* *16-2525* *16-2516*				22	0.05	66; 70; 71; 71; 72; 73; 73; 77; 77; 77; 80; 85; 90; 79; 80; 80; 81; 82; 82; 85; 86; 73	66	90	78	7.8
GLP: yes 2018				46	overall		66	93	80	7.3

Table A 70: Rotational trial summary for brassica vegetables, study report 16-2516

Reference:	Determination of the residues of BYI 02960 in/on the field rotational crop cauliflower, broccoli and soil after spray application of BYI 02960 SL 200 to bare soil in the field in Belgium, the Netherlands, Italy and Spain, Freitag, T.; Effertz, C., 2018, 16-2516, M-617941-02-1		
GLP:	Yes	Sample storage conditions:	plant matrices: max. 462 days at ≤ -18°C soil: max. 372 days at ≤ -18°C
Preceding crop:	none, application on bare soil	Analytical methods:	plant: 01304, validated soil: 01074/M001, validated
Succeeding crop:	brassica vegetables cauliflower and broccoli (curd)	Limit of Quantification (mg/kg):	plant: 0.01 mg/kg for BYI 02960 0.02 mg/kg for DFA (expressed as BYI 02960) soil: 0.005 mg/kg for BYI 02960 0.005 mg/kg for DFA 0.005 mg/kg for 6-CNA
Indoor/Outdoor:	outdoor	Limit of Detection (mg/kg):	30% of LOQ
Formulation:	200 SL (SP102000021884)	Residues calculated as:	plant: BYI 02960 and DFA equivalents soil: analyte equivalents
Content of active substance (g/kg or g/l):	200 g/L		

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2516 16-2516-01 T-1A The Netherlands 1681 ND Zwaagdijk	Bare soil Cauliflower Clarina	 1) 01.06.2016 3) 12.08.2016 – 26.08.2016	SPI and incorporation	0.300	400	0.075	11.05.2016	 45 49	soil 0-20 cm [0-8 in] curd 1st rotation (PB 21 days)	0.10 <0.01 <0.01	 0.075 0.070	 0.085 0.080	<0.005 0.025 0.023	22 93 107
16-2516 16-2516-02 T-1A Belgium 6221 Saint-Amand	Bare soil Cauliflower Korlanu	 1) 10.06.2016 3) 15.08.2016 – 19.08.2016	SPI and incorporation	0.300	300	0.1	11.05.2016	 45 49	soil 0-20 cm [0-8 in] curd 1st rotation (PB 30 days)	0.046 <0.01 <0.01	 0.18 0.20	 0.19 0.21	<0.005 0.059 0.065	30 86 100
16-2516 16-2516-03 T-1A Italy 70032 Bitonto	Bare soil Broccoli Marathon F1 - Sakata	 1) 30.09.2016 3) 20.12.2016 – 20.02.2017	SPI and incorporation	0.300	300	0.1	02.09.2016	 45 49	soil 0-20 cm [0-8 in] curd 1st rotation (PB 28 days)	0.079 <0.01 <0.01	 0.13 0.062	 0.14 0.072	<0.005 0.042 0.021	28 116 130
16-2516 16-2516-04 T-1A Spain 46230 Alginet	Bare soil Broccoli Principe F1	 1) 29.09.2016 3) 15.12.2016 – 15.01.2017	SPI and incorporation	0.300	400	0.075	06.09.2016	 45 49	soil 0-20 cm [0-8 in] curd 1st rotation (PB 23 days)	0.069 <0.01 <0.01	 0.17 0.16	 0.18 0.17	<0.005 0.056 0.054	24 115 128

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2516 16-2516-01 T-2A The Netherlands 1681 ND Zwaagdijk	Bare soil Cauliflower Aerospace Summer	 1) 12.04.2017 3) 15.07.2017 – 01.08.2017	SPI and incorporation	0.300	400	0.075	25.11.2016	 45 49	soil 0-20 cm [0-8 in] curd 2nd rotation (PB 138 days)	0.051 <0.01 <0.01	 0.045 0.036	 0.055 0.046	<0.005 0.015 0.012	138 230 244
16-2516 16-2516-02 T-2A Belgium 6221 Saint-Amand	Bare soil Cauliflower Tarifa F1	 1) 24.05.2017 3) 10.08.2017 – 22.08.2017	SPI and incorporation	0.300	300	0.1	14.12.2016	 45 49	soil 0-20 cm [0-8 in] curd 2nd rotation (PB 161 days)	0.039 <0.01 <0.01	 0.15 0.11	 0.16 0.12	<0.005 0.048 0.035	161 229 243
16-2516 16-2516-03 T-2A Italy 70032 Bitonto	Bare soil Broccoli Marathon F1 - Sakata	 1) 29.08.2017 3) 03.11.2017 – 31.01.2018	SPI and incorporation	0.300	300	0.1	30.03.2017	 45 49	soil 0-20 cm [0-8 in] curd 2nd rotation (PB 152 days)	0.074 <0.01 <0.01	 0.087 0.11	 0.097 0.12	<0.005 0.029 0.037	152 222 235
16-2516 16-2516-04 T-2A Spain 46230 Alginet	Bare soil Broccoli Principe F1	 1) 29.09.2016 3) 15.12.2016 – 15.01.2017	SPI and incorporation	0.300	400	0.075	23.05.2016	 45 49	soil 0-20 cm [0-8 in] curd 2nd rotation (PB 129 days)	0.067 <0.01 <0.01	 0.18 0.17	 0.19 0.18	<0.005 0.061 0.058	130 221 234

Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at sampling	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)
				kg a.s./ha	Water (L/ha)	kg a.s./hL				BYI 02960 (a.s.) as BYI 02960	DFA as BYI 02960	Total residue (a.s. plus DFA) as BYI 02960	DFA as DFA	
16-2516 16-2516-01 T-3A The Netherlands 1681 ND Zwaagdijk	Bare soil Cauliflower Aerospace Summer	 1) 12.04.2017 3) 15.07.2017 – 01.08.2017	SPI and incorporation	0.300	400	0.075	11.05.2016	 45 49	soil 0-20 cm [0-8 in] curd 3rd rotation (PB 336 days)	0.031 <0.01 <0.01	 0.021 0.023	 0.031 0.033	<0.005 0.007 0.008	336 428 442
16-2516 16-2516-02 T-3A Belgium 6221 Saint-Amand	Bare soil Cauliflower Tarifa F1	 1) 24.05.2017 3) 10.08.2017 – 22.08.2017	SPI and incorporation	0.300	300	0.1	07.07.2016	 45 49	soil 0-20 cm [0-8 in] curd 3rd rotation (PB 321 days)	0.024 <0.01 <0.01	 0.12 0.098	 0.13 0.11	<0.005 0.041 0.033	321 389 403
16-2516 16-2516-03 T-3A Italy 70032 Bitonto	Bare soil Broccoli Marathon F1 - Sakata	 1) 29.08.2017 3) 03.11.2017 – 31.01.2018	SPI and incorporation	0.300	300	0.1	30.09.2016	 45 49	soil 0-20 cm [0-8 in] curd 3rd rotation (PB 333 days)	0.064 <0.01 <0.01	 0.047 0.068	 0.057 0.078	<0.005 0.016 0.023	333 403 416
16-2516 16-2516-04 T-3A Spain 46230 Alginet	Bare soil Broccoli Parthenon	 1) 11.08.2017 3) 05.10.2017 – 30.10.2017	SPI and incorporation	0.300	400	0.075	06.09.2016	 45 49	soil 0-20 cm [0-8 in] curd 3rd rotation (PB 339 days)	0.019 <0.01 <0.01	 0.053 0.046	 0.063 0.056	<0.005 0.018 0.015	342 400 414

A 2.1.7 Other/Special Studies

No additional studies are submitted.

A 2.2 Deltamethrin

A 2.2.1 Stability of residues

A 2.2.1.1 Stability of residues during storage of samples

A 2.2.1.1.1 Storage stability of residues in plant products

The stability of deltamethrin-related residues during freezer storage up to 24 months in orange fruit is summarized in the study below.
Within some residue studies on oilseed rape the temperatures during storage increased. Therefore, two storage stability studies at exaggerated temperatures were performed and summarized within this submission.

A 2.2.1.1.1.1 Study report 09-07

Comments of zRMS:	The study was conducted to evaluate the stability of deltamethrin and its isomers (AE F108569 and AE 0035073) in frozen condition (≤ -18°C) for a period of 24 months in orange (fruit). Individual aliquots of orange (fruit) were fortified with deltamethrin, AE F108569, AE 0035073 at 0.20 mg/kg. The samples stored in HDPE Nalgene containers at an average temperature of -18°C or below, were analyzed at the nominal storage intervals of 0, 6, 12, 18 and 25 months. Residues of deltamethrin and its isomers AE F108569 and AE 0035073 were determined by HPLC-MS/MS according to method 00855/M004. The limits of quantitation (LOQ) for deltamethrin and its isomers (AE F108569 and AE 0035073) are 0.01 mg/kg, for the sample material tested, corresponding to the lowest fortification level of successfully conducted recovery experiments. For deltamethrin, AE F108569, AE 0035073 all the recovery means were within the acceptable range of 70-110% with corresponding RSD (relative standard deviation) < 20%. All method validation data are in compliance with the guideline requirements for data collection methods. Deltamethrin and its isomers (AE F108569 and AE 0035073) were shown to be stable in orange (fruit) for at least 25 months (751 days) under freezer conditions at about -18 °C or below, after correction of the results by the mean of concurrent recoveries. The study is acceptable.

Reference:	**KCA 6.1/05**
Title:	Storage stability of residues of deltamethrin (AE F032640) and its isomers AE F108569 and AE 0035073 in orange during deep freeze storage for up to 24 months
Report:	Ballesteros, C.; 2012; 09-07; M-441996-01-1
Authority registration No:	
Guideline(s):	Guidelines for the generation of data concerning residues as provided in Annex II part A, section 6 and Annex III, part A, section 8 of Directive 91/414/EEC concerning the placing of plant protection products on the market, No. 7032/VI/95 rev.5: Appendix H Storage Stability of Residues Samples, July, 1997; Commission Regulation (EU) No 544/2011 of 10 June 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the data requirements for active substances; US EPA Residue Chemistry Test Guidelines, OPPTS 860.1380: Storage Stability Data, August 1996; OECD Guideline for the Testing of Chemicals No. 506, adopted 16 October 2007
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of this study was to determine the storage stability of Deltamethrin, alpha-*R*-deltamethrin and trans-deltamethrin in orange stored 25 months in frozen conditions (≤ -18ºC).
Individual aliquots of orange (fruit) were fortified with Deltamethrin, alpha-*R*-deltamethrin and trans-deltamethrin at 0.20 mg/kg. The samples stored in plastic containers at an average temperature of -18ºC or below, were analysed at the nominal storage intervals of 0, 6, 12, 18 and 25 months.

At each storage interval deltamethrin and its isomers were determined in the stored control samples and in the stored spiked samples according to the analytical method 00855/M004 (Lakaschus, S.; Winter; O.; 2009; M-356934-01-1). Concurrent recovery experiments at fortification levels of 0.01 mg/kg and 0.20 mg/kg were also performed for each analyte at each storage intervals. Samples were extracted with a mixture of acetone:dichloromethan:*n*-hexan (1:1:1, *v:v:v*) using a high speed blender. After the extraction step, the extract was evaporated to dryness and re-dissolved first with ethyl acetate. Then the obtained extract (Extract A) was directly diluted 20 times, adding the internal standard in a mixture of acetonitrile/10 mM aqueous ammonium acetate (9:1,*v:v*) in order to obtain the final extract. The residues are quantified by reversed phase HPLC with Electrospray and MS/MS-detection in a single run for the determination of all of the three analytes. The quantification was carried out by internal standardization using internal stable labelled standard for each corresponding analyte. Validation recoveries on orange fruit were conducted in the study. Concurrent recoveries were conducted at 0.01 mg/kg (except at storage interval 0 days) and 0.20 mg/kg at each analytical point.

Results and discussions

As required, in this summary only uncorrected recovery values will be discussed. The recovery values of the concurrent recovery rates per compound, storage interval and fortification level of 0.2 mg/kg were in the range of 90-100%. In case of fortification level of 0.01 mg/kg these recoveries ranged between 74-122% (except for Day-0 for which procedural recoveries were not measured).
At day 0, individual recoveries of Deltamethrin ranged between 107-113% (with an average recovery of 110%), between 103-109%, for alpha-*R*-deltamethrin (with an average recovery of 106%), and between 112-115% for trans-deltamethrin (with an average recovery of 114%)
Details of recovery data in orange fruit are shown in the table below.

Over the whole period of storage (25 months), the average recoveries of Deltamethrin ranged between 87-110%, whereas the average concurrent recoveries (for the same fortification level of 0.2 mg/kg) ranged between 92-100%.
Over the whole period of storage (25 months), the average recoveries of alpha-*R*-deltamethrin ranged between 77-106%, whereas the average concurrent recoveries (for the same fortification level of 0.2 mg/kg) ranged between 92-98%.

The average recoveries of trans-deltamethrin during the whole storage period of orange fruit ranged between 81-114%, whereas the average concurrent recoveries of trans-deltamethrin (for the same fortification level of 0.2 mg/kg) ranged between 90-97%.
The average recovery results after 25 months of storage of fortified samples were 91% for Deltamethrin, 78% for alpha-*R*-deltamethrin, and 97% for trans-deltamethrin.

Table A 71: Summary of concurrent recoveries of deltamethrin residues in plant matrices

Matrix	Spike level (mg/kg)	Storage Interval (months)	Sample size (n)	Individual procedural recoveries (%)	Mean ± RSD (%)
cis-deltamethrin					
Orange fruit	0.01	6	1	91	-
		12	1	100	-
		18	1	106	-
		25	1	87	-
	0.2	0	3	101; 99; 100	100±1.0
		6	3	95; 96; 86	92 ±6.0
		12	5	96; 92; 94; 104; 103	98±5.5
		18	3	102; 94; 94	97±4.8
		25	3	85; 95; 99	93±7.8
alpha-R-deltamethrin					
Orange fruit	0.01	6	1	93	-
		12	1	100	-
		18	1	105	-
		25	1	84	-
	0.2	0	3	96; 99; 95	97±2.2
		6	3	95; 96; 87	93±5.3
		12	5	96; 93; 94; 104; 102	98±5.0
		18	3	101; 94; 94	96±4.2
		25	3	85; 94; 96	92±6.4
trans-deltamethrin					
Orange fruit	0.01	6	1	96	-
		12	1	122	-
		18	1	74	-
		25	1	86	-
	0.2	0	3	102; 100; 79	94±13.6
		6	3	95; 97; 86	93±6.3
		12	5	99; 94; 91; 105; 97	97±5.5
		18	3	103; 92; 92	96±6.6
		25	3	83; 93; 95	90±7.1

alpha-*R*-deltamethrin as AE F108569
trans-deltamethrin as AE 0035073

Table A 72: Stability of deltamethrin residues in plant matrices following storage at ≤ -18°C

Matrix	Spike level (mg/kg)	Storage interval (months)	Individual recovered residues (mg/kg)	Individual recoveries (%)
cis-deltamethrin				
Orange fruit	0.2	0	0.222; 0.226; 0.213	111; 113; 106
		6	0.173; 0.176; 0.169	86; 88; 84
		12	0.172; 0.191; 0.196	86; 95; 98
		18	0.194; 0.193; 0.186	97; 97; 93
		25	0.181; 0.188; 0.177	91; 94; 89
alpha-R-deltamethrin				
Orange fruit	0.2	0	0.206; 0.214; 0.218	103; 107; 109
		6	0.164; 0.168; 0.173	82; 84; 86
		12	0.146; 0.122; 0.161; 0.182; 0.141; 0.179	73; 61; 81; 91; 71; 89
		18	0.155; 0.135; 0.169	77; 68; 85
		25	0.155; 0.141; 0.169	78; 71; 84
trans-deltamethrin				
Orange fruit	0.2	0	0.227; 0.230; 0.224	114; 115; 112
		6	0.161; 0.164; 0.165	80; 82; 82
		12	0.215; 0.212; 0.211; 0.228; 0.219; 0.213	108; 106; 106; 114; 110; 107
		18	0.177; 0.171; 0.178	88; 85; 89
		25	0.200; 0.178; 0.202	100; 89; 101

alpha-*R*-deltamethrin as AE F108569
trans-deltamethrin as AE 0035073

Conclusion
For the sample material orange (fruit), Deltamethrin, alpha-*R*-deltamethrin and trans-deltamethrin were shown to be stable for at least 25 months (751 days) under freezer conditions at about -18°C or below.

A 2.2.1.1.1.2 Study report S13-03307

Comments of zRMS:	According to the storage stability results it can be concluded that residues of deltamethrin and its isomers, alpha-R-deltamethrin and trans-deltamethrin, in the plant matrices of high water and high protein content were stable (<30% decomposition) after storage at + 1°C for 8 hours, following a storage of 6 days at -7°C. The study is acceptable.

Reference:	**KCA 6.1/02**
Title:	Amendment no. 3 to final report - 7 days freezer storage stability study with different combinations of a total of 61 analytes (parent and metabolite molecules) and five matrix types (high water / acidic / starch / protein / oil)
Report:	Lakaschus, S.; Gizler, A.; 2017; S13-03307; M-480441-06-1
Authority registration No:	
Guideline(s):	Commission Regulation (EU) No 544/2011 of 10 June 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the data requirements for active substances US EPA Residue Chemistry Test Guideline OPPTS 860.1380: Storage Stability Data OECD Test Guideline 506, adopted 16 October 2007
Deviations:	see report
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The purpose of this study was to determine the storage stability of relevant residues of deltamethrin for a period of 8 hours at +1°C followed by 6-8 days of storage at -7°C in tomato fruit, wheat green material (high water content) and dry peas (high protein content). Individual 5-g control samples of the respective sample material were spiked with the relevant analytes deltamethrin or alpha-*R*-deltamethrin or trans-deltamethrin (separately) at a fortification level of 0.1 mg/kg of each analyte.

Except for the day-0 analysis, samples were stored in plastic containers at an average temperature of +1°C for eight hours, following 6-8 days at -7°C. For day-0 analysis, five treated samples of each material were chosen, as well as two control samples of each. Samples were then also analyzed after 6, 7 or 8 days of storage.
At each storage interval, five stored samples were analyzed. In addition two concurrent recoveries per commoditiy were conducted.
Deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin, were analytically determined using analytical method 01207 (Schoening, R.; Willmes, J.; 2014; M-476845-01).
For the analysis of deltamethrin, samples were adjusted with water followed by addition of acetonitrile leading to an acetonitrile/water ratio of (4/1, v/v). The samples fortified with deltamethrin were left to soak for 15 minutes and then shaken for 15 minutes to enhance the extraction. Afterwards, a salt mixture (Mg_2SO_4/NaCl/Na_3 citrate 2 H_2O/Na_2H citrate 6 H_2O) (4/1/1/0.5, w/w/w/w)) was added and the extracts were centrifuged. The extracts were submitted to LC-MS/MS-analysis and quantification was done using matrix-matched standards.

The methods were successfully validated at a level of 0.10 mg/kg relevant to this study in/on samples of tomato (fruit), wheat (green material) and peas (dry peas) in the presented study. For further information on validation of the two methods please refer to Section 5, Appendix A 2.1.1.1).

Results and discussions
In the control samples, the residues were always below 30% of the LOQ.
Summaries of concurrent recoveries conducted as a part of the study are presented in Table A 73. The mean of the recoveries for each matrix were in an acceptable range (mean recoveries were 89% to 102%). The RSDs were in the range of 1.2 to 7.3%.

Summaries of residues recovered in stored samples are presented in Table A 74 for deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin.

Residues of deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin were stable (<30% decomposition) in tomato fruit, wheat green material and dry peas after storage for 8 hours at +1°C followed by 6 days of storage at -7°C frozen storage.

Table A 73: Summary of concurrent recoveries of deltamethrin residues in plant matrices

Matrix	Spike level (mg/kg)	Storage Interval (days)	Sample size (n)	Individual procedural recoveries (%)	Mean ± RSD
cis-deltamethrin					
Tomato fruit	0.1	0	5	94; 94;97; 98; 93	95±2.3
		6	2	93; 95	94
Wheat green material	0.1	0	5	100; 98; 99; 97; 98	98±1.2
		6	2	100; 97	99
Peas, dry	0.1	0	5	91; 88; 92; 91; 88	90±2.1
		6	2	87; 90	89
alpha-R-deltamethrin					
Tomato fruit	0.1	0	5	94; 90; 95; 95; 91	93±2.5
		6	2	100; 89	95
Wheat green material	0.1	0	5	101; 115; 98; 96; 102	102±7.3
		6	2	95; 101	98
Peas, dry	0.1	0	5	89; 83; 99; 96; 87	91±7.2
		6	2	91; 95	93
trans-deltamethrin					
Tomato fruit	0.1	0	5	100; 97; 97; 104; 96	99±3.3
		6	2	97; 90	94
Wheat green material	0.1	0	5	101; 106; 97; 102; 97	101±3.8
		6	2	101; 96	99
Peas, dry	0.1	0	5	93; 87; 96; 93; 92	92±3.5
		6	2	95; 97	96

Table A 74: Stability of deltamethrin residues in plant matrices following storage at + 1°C for eight hours, following 6 days at -7°C

Matrix	Spike level (mg/kg)	Storage interval (days)	Individual recovered residues (mg/kg)	Individual recoveries (%)
cis-deltamethrin				
Tomato fruit	0.1	0	0.094; 0.094; 0.097; 0.098; 0.093	94; 94; 97; 98; 93
		6	0.085; 0.081; 0.082; 0.082; 0.084	85; 81; 82; 82; 84
Wheat green material	0.1	0	0.100; 0.098; 0.099; 0.097; 0.098	100; 98; 99; 97; 98
		6	0.096; 0.097; 0.101; 0.097; 0.098	96; 97; 101; 97; 98
Peas dry	0.1	0	0.091; 0.088; 0.092; 0.091; 0.088	91; 88; 92; 91; 88
		6	0.080; 0.079; 0.082; 0.080; 0.076	80; 79; 82; 80; 76
alpha-R-deltamethrin				
Tomato fruit	0.1	0	0.094; 0.090; 0.095; 0.095; 0.091	94; 90; 95; 95; 91
		6	0.076; 0.082; 0.085; 0.084; 0.074	76; 82; 85; 84; 74
Wheat green material	0.1	0	0.101; 0.115; 0.098; 0.096; 0.102	101; 115; 98; 96; 102
		6	0.091; 0.106; 0.113; 0.088; 0.090	91; 106; 113; 88; 90
Peas dry	0.1	0	0.089; 0.083; 0.099; 0.096; 0.087	89; 83; 99; 96; 87
		6	0.081; 0.073; 0.074; 0.070; 0.070	81; 73; 74; 70; 70
trans-deltamethrin				
Tomato fruit	0.1	0	0.100; 0.097; 0.097; 0.104; 0.096	100; 97; 97; 104; 96
		6	0.085; 0.074; 0.085; 0.084; 0.082	85; 74; 85; 84; 82
Wheat green material	0.1	0	0.101; 0.106; 0.097; 0.102; 0.097	101; 106; 97; 102; 97
		6	0.106; 0.096; 0.107; 0.099; 0.097	106; 96; 107; 99; 97
Peas dry	0.1	0	0.093; 0.087; 0.096; 0.093; 0.092	93; 87; 96; 93; 92
		6	0.086; 0.081; 0.079; 0.080; 0.084	86; 81; 79; 80; 84

Conclusion

Residues of deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin, in the plant matrices of high water and high protein content were stable (<30% decomposition) after storage at + 1°C for 8 hours, following a storage of 6 days at -7°C.

A 2.2.1.1.1.3 Study report S17-05312

Comments of zRMS:	The stability of cis-deltamethrin, its isomers AE 0035073 (trans-deltamethrin) and AE F018569 (α-(R)-deltamethrin) was investigated in oilseed rape (seed and straw) after 10 days at -8.5°C (target) plus 5 hours at +6°C (target) in the dark. The residues of deltamethrin were determined in oilseed rape (seed and straw) according to BCS Method 00855/M004. The final determination was performed with HPLC-MS/MS. The Limit of Quantitation (LOQ), defined as the lowest validated fortification level, was 0.01 mg/kg for deltamethrin (cis-, trans- and α-(R)-) in oilseed rape (seed) and 0.05 mg/kg in oilseed rape (straw). All method validation data are in compliance with the guideline requirements for data generation methods. After 10 days of storage at -8.5°C (target) followed by 5 hours at +6°C (target) the mean recovery in oilseed rape (seed) was 78 % for *cis*-deltamethrin, 89 % for *trans*-deltamethrin, 84 % for *α-(R)*-deltamethrin. After 10 days of storage at -8.5°C (target) followed by 5 hours at +6°C (target) the mean recovery in oilseed rape (straw) was 92 % for *cis*-deltamethrin, 96 % for *trans*-deltamethrin, 97 % for *α-(R)*-deltamethrin. Furthermore the mean concurrent recoveries of day 0 to day 10 in oilseed rape (seed) determined from freshly fortified samples were in the range of 80-85 % for cis-deltamethrin, 84-86 % for trans-deltamethrin, 80-83 % for α-(R)-deltamethrin. In oilseed rape (straw) the mean concurrent recoveries of day 0 to day 10 determined from freshly fortified samples were in the range of 90-95 % for cis-deltamethrin, 91-93 % for trans-deltamethrin, 91-93 % for α-(R)-deltamethrin. The study results demonstrate that the residues of deltamethrin (cis-, trans- and α-(R)-) are stable in oilseed rape (seed and straw) when stored for 10 days at -8.5°C (target) plus 5 hours at +6°C (target). The study is acceptable.

Reference:	**KCA 6.1/03**
Title:	Storage stability of deltamethrin and flupyradifurone in oilseed rape (seed and straw)
Report:	Winter, O.; Amann, S.; Giesler, W.; 2018; S17-05312; M-626405-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC Guideline 7032/VI/95, rev. 5, July 1997; Appendix H of working document 1607/VI/97, rev. 2, June 1999 – Storage Stability of Residue Samples OECD 506, 2007; OECD Guideline for the Testing of Chemicals – Stability of Pesticide Residues in Stored Commodities
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

To determine the storage stability after 10 days at -8.5°C followed by 5 hours at +6°C in the dark of the relevant residues of deltamethrin in oilseed rape seed and straw, control samples of the respective sample material were spiked with Deltamethrin or alpha-*R*-deltamethrin or trans-deltamethrin (separately) at a fortification level of 1.0 mg/kg.

Except for the day-0 analysis, samples were stored in glass containers in a cooling box at an average temperature of - 8.5°C for ten days, then they were transferred to +6°C in the dark.

For day-0 analysis, five treated samples of each material were chosen, as well as two control samples of each. At the 10-day storage interval, four aliquots of the stored control and five aliquots of fortified crop

matrix were removed from storage.
Samples were extracted with acetone/dichloromethane/ n-hexane (1/1/1, v/v/v) followed by liquid-liquid partition with acetonitrile and n-hexane (seed) or GPC-clean up using cyclohexane/ethyl acetate as eluent (straw). The final determination was performed with HPLC-MS/MS. All samples were analysed by single extraction and single injection to the detection system. The residues were quantified by internal standardisation using stable-labelled internal standards. The responses of the LC-MS/MS system to each analyte and its internal standard were measured in samples and in standards, and a relative response was calculated (calculated as the ratio of the analyte and the stable isotopically labelled internal standard responses). The relative response of the analyte in each sample was compared to the relative response of the analyte in the standards.

Deltamethrin and its isomers were analytically determined using analytical method 00855/M004 (Lakaschus, S.; Winter; O.; 2009; M-356934-01-1). The method was successfully validated in oilseed rape seeds and straw (equivalent to green material) in the storage stability study. The limits of quantitation (LOQs) were 0.01 mg/kg for deltamethrin residues in all matrices.

Results and discussions
In the control samples, the residues were always below 30% of the LOQ.
Summaries of concurrent recoveries conducted as a part of the study are presented in Table A 75. The mean of the recoveries for each matrix were in an acceptable range (mean recoveries were 80% to 95%). The RSDs were in the range of 1.0 to 4.0%. Additional concurrent recoveries were determined at day 10 for samples spiked at 0.1 mg/kg, these values are not presented in the following table. Recoveries were 92% and 88% in seed and straw respectively, for deltamethrin, 86% and 92% in seed and straw, respectively for alpha-*R*-deltamethrin and 93% and 91% in seed and straw, respectively, for trans-deltamethrin.
Summaries of residues recovered in stored samples are presented in Table A 76.
Residues of deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin were stable (<30% decomposition) in oilseed rape seed and straw for 10 days of storage at -8.5°C followed by 5 hours at +6°C.

Table A 75: Summary of concurrent recoveries of deltamethrin residues in plant matrices

Matrix	Spike level (mg/kg)	Storage Interval (days)	Sample size (n)	Individual procedural recoveries (%)	Mean ± RSD (%)
cis-deltamethrin					
Seed	1.0	0	5	77;79; 80; 81; 83	80±2.8
		10	1	78	-
Straw	1.0	0	5	92; 92; 94; 99; 100	95±4.0
		10	1	91	-
alpha-R-deltamethrin					
Seed	1.0	0	5	79; 79; 80; 80; 81	80±1.0
		10	1	80	-
Straw	1.0	0	5	90; 90; 92; 97; 97	93±3.8
		10	1	90	-
trans-deltamethrin					
Seed	1.0	0	5	82; 83; 84; 84; 85	84±1.4
		10	1	79	-
Straw	1.0	0	5	90; 91; 92; 96; 98	93±3.7
		10	1	91	-

Only concurrent recoveries for 1.0 mg/kg spiked samples are presented here. In addition samples were also fortified at a level of 0.1 mg/kg. For details, please refer to the report.
RSD values calculated from rounded values of the report.

Table A 76: Stability of deltamethrin residues in oilseed rape matrices following storage at -8.5°C for ten days, then +6°C for 5 hours

Matrix	Spike level (mg/kg)	Storage interval (days)	Individual recovered residues (mg/kg)	Individual recoveries (%)
cis-deltamethrin				
Seed	1.0	0	0.79; 0.77; 0.80; 0.81; 0.83	79; 77; 80; 81; 83
		10	0.78; 0.77; 0.79; 0.78; 0.78	78; 77; 79; 78; 78
Straw	1.0	0	0.94; 0.92; 1.00; 0.92; 0.99	94; 92; 100; 92; 99
		10	0.89; 0.89; 1.00; 0.91; 0.91	89; 89; 100; 91; 91
alpha-R-deltamethrin				
Seed	1.0	0	0.80; 0.79; 0.81; 0.79; 0.80	80; 79; 81; 79; 80
		10	0.90; 0.94; 0.81; 0.83; 0.73	90; 94; 81; 83; 73
Straw	1.0	0	0.92; 0.90; 0.97; 0.90; 0.97	92; 90; 97; 90; 97
		10	0.94; 0.96; 0.96; 0.99; 0.98	94; 96; 96; 99; 98
trans-deltamethrin				
Seed	1.0	0	0.82; 0.83; 0.84; 0.84; 0.85	82; 83; 84; 84; 85
		10	0.95; 0.88; 0.89; 0.91; 0.84	95; 88; 89; 91; 84
Straw	1.0	0	0.91; 0.92; 0.98; 0.90; 0.96	91; 92; 98; 90; 96
		10	0.91; 1.01; 0.96; 0.94; 0.98	91; 101; 96; 94; 98

Conclusion
Residues of deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin in the plant matrices of oilseed rape (seed and straw) were stable during 10 days of storage at -8.5°C followed by 5 hours at +6°C.

A 2.2.1.1.2 Storage stability of residues in animal products

No additional studies are required.

A 2.2.1.2 Stability of residues in sample extracts

A 2.2.1.2.1 Storage stability of residues in plant sample extracts

No additional studies are required.

A 2.2.1.2.2 Storage stability of residues in animal sample extracts

No additional studies are required.

A 2.2.2 Nature of residues in plants, livestock and processed commodities

A 2.2.2.1 Nature of residue in primary crops

No additional studies are required.

A 2.2.2.2 Nature of residue in rotational crops

In 2012, a confined rotational crop study was performed with [^{14}C]-gemdimethyl deltamethrin. It is presented below.

A 2.2.2.2.1 Study report MEF-11/669

Comments of zRMS:	The metabolism of deltamethrin was investigated in the rotational crops Swiss chard, turnips and spring wheat after one soil treatment with radiolabelled [gemdimethyl-^{14}C] deltamethrin. The actual application rate corresponded to 41 g a.s./ha, which was slightly above the anticipated maximal seasonal treatment rate for pre-emergent use. Two rotations were sown at plant-back intervals of 30 and 134 days. The study was concluded after two rotations due to the very low TRRs (< 0.01 mg/kg) in all of the collected matrices. The TRR values of all samples were very low and ranged from values < LOQ in turnip leaves and roots and wheat forage of the 2nd rotation to 0.009 mg/kg in wheat straw of the 1st rotation. They showed a decrease from the 1st to the 2nd rotation. No extraction of plant matrices was conducted during this study; the radioactivity in all samples was determined exclusively by combustion followed by LSC.

Reference:	**KCA 6.6.1/01**
Title:	Metabolism of [gemdimethyl-14C] deltamethrin in confined rotational crops
Report:	Schmeling, S.; Breuer-Rehm, M.; 2012; MEF-11/669; M-431769-01-1
Authority registration No:	
Guideline(s):	OECD Guideline for the Testing of Chemicals 502: Metabolism in Rotational Crops US EPA Residue Chemistry Test Guideline OPPTS 860.1850: Confined Accumulation in Rotational Crops European Parliament and Council Regulation (EC) no. 1107/2009
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The objective of this study was to investigate the metabolism of deltamethrin in rotational crops after one pre-emergent spray application onto bare soil. The application rate of the test compound [gemdimethyl-^{14}C] deltamethrin, formulated as an EW015, was aimed to be about 10% above the highest annual field dose of approx. 37.5 g a.s./ha to compensate for possible losses during treatment. The rotational crops used were Swiss chard, turnips and spring wheat. The intended timeframe for sowing was approx. 30, 150 and 365 days after soil treatment for the first, the second and the third rotation, respectively.

Immature raw agricultural commodities (RACs) investigated were Swiss chard intermediate leaves and forage and hay of wheat. All other RACs (Swiss chard leaves, turnip leaves and roots and wheat straw and grain) were harvested at maturity. The study was concluded after two rotations due to the very low TRRs (< 0.01 mg/kg) in all of the collected matrices.

Results and discussion

The TRR values were generally very low and ranged from values below the LOD (=LOQ) in turnip leaves and roots and wheat forage of the 2nd rotation to 0.009 mg/kg in wheat straw of the 1st rotation. The TRRs in the edible RACs were very low. The TRRs of all samples showed a more or less noticeable decrease from the 1st to the 2nd rotation.

The TRR values for all matrices are shown in the following table:

Table A 77: Total Radioactive Residues (TRRs) in Swiss chard, turnip and wheat matrices

Matrix	TRR (mg/kg)	
	First rotation	Second rotation
Swiss chard intermediate leaves	0.007	0.001
Swiss chard leaves	0.002	0.001
Turnip leaves	0.002	<0.001
Turnip roots	0.001	<0.001
Wheat forage	0.002	<0.001
Wheat hay	0.006	0.002
Wheat straw	0.009	0.002
Wheat grain	0.003	0.002

No extraction of plant matrices was conducted during this study because of the very low TRR values. The radioactivity in all samples was exclusively determined by combustion followed by LSC.

Conclusions
There was no significant metabolism of deltamethrin detectable in confined rotational crops. Results from earlier studies show that uptake and translocation of the parent compound and potential metabolites seems to be very limited in plants. It is also known that under aerobic conditions deltamethrin is rapidly metabolised in soil and that the formed metabolites are readily mineralised to carbon dioxide.

A 2.2.2.3 Nature of residues in processed commodities

No additional studies are required.

A 2.2.2.4 Nature of residues in livestock

No additional studies are required.

A 2.2.3 Magnitude of residues in plants

A 2.2.3.1 Rape seed

The critical GAP (cGAP) for the proposed use of Sivanto Energy (DLT+FPF EC 85) in oilseed rape is summarized in Table A 78.

Table A 78: Comparison of intended and critical EU GAPs

Type of GAP	Number of applications	Application rate per treatment (kg a.s./ha)	Interval between application	Growth stage at last application	PHI (days)
cGAP EU (DAR, Sweden, 2002)	3	7.5	14	At any stage	45
cGAP EU (Art. 12, EFSA, 2015)	4	6.25	14	-	45
Intended cGAP (Use No.* 1, 2, 5, 6, 7, 8, 11, 12, 17, 18, 23, 24, 27, 28)	2	7.5	14	BBCH 30-49	As per growth stage
Intended cGAP (Use No.* 13, 14, 19, 20)	2	7.5	14	BBCH 50-59	As per growth stage
Intended cGAP (Use No.* 3, 4, 9, 10, 15, 16, 21, 22, 25, 26, 29, 30)	2	5.0	14	BBCH 65-79	45

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0

According to SANCO 7525/VI/95 rev. 10.3, extrapolation from rapeseed, seeds to mustard, seeds is possible with 8 trials for treatments before and after the edible part has formed.

A 2.2.3.1.1 Study report 15-2132

Comments of zRMS:	Four field residue trials were conducted in northern Europe in 2015 to determine the magnitude of the relevant residues of deltamethrin and BYI 02960 (flupyradifurone) in/on rape (green material, seed and straw) after two spray applications with Deltamethrin & Flupyradifurone EC 085, an EC formulation containing 10 g/L deltamethrin and 75 g/L BYI 02960. Trial GAP: 2 x 5-7.5 g a.s./ha, interval = 14 days, PHI = 45 days. This study was conducted according to the study plan and study plan amendment. For deltamethrin and its isomers no residues above the LOQ were found in the control samples. For deltamethrin and its isomers the average recoveries per fortification level were within the acceptable range of 70 – 110% and the RSD values were below 20% for all compounds and for all sample materials. The samples were analyzed according to the method 00855/M004. No deltamethrin-related residue will be detected in mature seeds (<0.01 mg/kg for each isomer). Residue results (mg/kg) in seed: E: 4x<0.01 RA: 4x<0.01 The study is acceptable.

Reference:	**KCA 6.3.1.1/01**
Title:	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in France (North), Germany and Belgium
Report:	Schulte, G.; 2017; 15-2132; M-578527-02-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Deviations:	yes, see report
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
In 2015, 4 field trials were conducted in northern Europe (northern France, Germany [2] and Belgium) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960).
Two applications were made at an interval of 13-14 days, each at a rate of 0.75 L/ha, corresponding to 7.5 g a.s./ha. The last application was done at a crop growth stage of BBCH 73-77 (30% to 70% of pods have reached final size). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the treatment and 39 days after the last treatment in the German trial (15-2132-02, as collection of immature seeds was not yet possible). Mature seed samples and rape straw were collected at two to three different points in time: 37-42 days after the last application (BBCH 87-88), 44-48 days after the last application (BBCH 88-89) and 51-54 days after the last application (BBCH 89). All samples were frozen within 24 hours after sampling and stored at a temperature at or below -18°C until analysis (max. 168 days). The maximum storage period of

168 days is covered by the available storage stability study where the analytes have been proven to be stable for a period of at least 36 months (Czarnecki, J. J.; 1996, M-139715-01-1).

The samples were analyzed according to method 00855/M004 (Lakaschus, S.; Winter; O.; 2009; M-356934-01-1). The LOQ in green material and straw was 0.05 mg/kg and 0.01 mg/kg in seed for each compound, cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073).

Results
During the course of this study, the method performance was checked/validated by concurrent recoveries. Full validation of method 00855/M004 was done and documented for oilseed rape (seeds) when developing the method. For rape, green material and straw a full validation (2 controls, 5 repetitions each at two fortification levels) was done within this study (cf. Section B5). The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always well below 20% (Table A 79).

Details of the application and residue information are summarized in Table A 80. No residues of cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073) at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 79: Recovery data for deltamethrin and its isomers AE F108569 and AR 0035073 in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
15-2132 15-2132-01 to -04 15-2132-01-T to -04-T GLP: yes 2015	Rape	green material	deltamethrin	6	0.05	97; 97; 97; 98; 99; 99	97	99	98	1.0
				6	0.5	99; 100; 100; 101; 102; 102	99	102	101	1.2
				1	5.0	99	99	99	99	
				13	overall		97	102	99	1.8
			AE F108569	6	0.05	93; 95; 96; 98; 98; 100	93	100	97	2.6
				6	0.5	95; 96; 98; 98; 99; 99	95	99	98	1.7
				1	5.0	100	100	100	100	
				13	overall		93	100	97	2.2
			AE 0035073	6	0.05	92; 95; 95; 95; 97; 99	92	99	96	2.5
				6	0.5	99; 99; 99; 100; 102; 103	99	103	100	1.7
				1	5.0	102	102	102	102	
				13	overall		92	103	98	3.3
		seed	deltamethrin	2	0.01	95; 96	95	96	96	
				2	0.1	89; 99	89	99	94	
				4	overall		89	99	95	4.4
			AE F108569	2	0.01	92; 95	92	95	94	
				2	0.1	90; 93	90	93	92	
				4	overall		90	95	93	2.3
			AE 0035073	2	0.01	100; 101	100	101	101	
				2	0.1	96; 97	96	97	97	
				4	overall		96	101	99	2.4
		straw	deltamethrin	6	0.05	75; 95; 99; 100; 100; 101	75	101	95	10.5
				6	0.5	72; 100; 100; 101; 102; 103	72	103	96	12.4
				1	5.0	72	72	72	72	
				13	overall		72	103	94	12.8

Study Trial No. Plot No. GLP Year	Crop	Portion analyzed	a.s./ metabolite	n	Fortification level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
			AE F108569	6	0.05	75; 92; 93; 96; 100; 104	75	104	93	10.7
				6	0.5	68; 96; 97; 98; 99; 100	68	100	93	13.3
				1	5.0	75	75	75	75	
				13	overall		68	104	92	12.5
			AE 0035073	6	0.05	73; 95; 97; 97; 100; 100	73	100	94	11.0
				6	0.5	71; 97; 97; 98; 99; 100	71	100	94	11.9
				1	5.0	74	74	74	74	
				13	overall		71	100	92	12.2

Table A 80: Summary of the trials of study 15-2132

Analyte 1: deltamethrin (determined as deltamethrin, calculated as deltamethrin), Analyte 2: AE F108569 (determined as AE F108569, calculated as AE F108569), Analyte 3: AE 0035073 (determined as AE 0035073, calculated as AE 0035073)

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
15-2132-01 15-2132-01-T France, north 37310 Chambourg sur Indre Europe, North F 2015	Rape Arizona; Winter variety	1) 19.08.2014 2) 07.04.2015 - 30.04.2015 3) 01.07.2015 - 10.07.2015	7.5 7.5	300 300	2.5 2.5	04.05.2015/0 18.05.2015/14	75	green material	75 75	<0.05 0.18	<0.05 <0.05	<0.05 <0.05	0* 0	(g) 15-2132 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: Analyte 1, 2, 3 straw, seed and green material: 00855/M004 (k) LOQ: Analyte 1, 2, 3 straw: 0.05 mg/kg Analyte 1, 2, 3 seed: 0.01 mg/kg Analyte 1, 2, 3 green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132 (m) Storage: Analyte 1, 2, 3 straw: 127 days Analyte 1, 2, 3 seed: 119 days Analyte 1, 2, 3 green material: 168 days
								seed	88 89 89	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	42 45 52	
								straw	88 89 89	<0.05 <0.05 <0.05	<0.05 <0.05 <0.05	<0.05 <0.05 <0.05	42 45 52	

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
15-2132-02 15-2132-02-T Germany 51399 Burscheid Europe, North F 2015	Rape Visby; Winter variety	1) 25.08.2014 2) 20.04.2015 - 08.05.2015 3) 01.07.2015 - 01.08.2015	7.5 7.5	300 300	2.5 2.5	07.05.2015/0 21.05.2015/14	76	green material seed straw	76 76 82 88 89 88 89	<0.05 0.15 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	0* 0 39 46 53 46 53	(g) 15-2132 (h) EC (deltamethrin 10 g/L ,flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: Analyte 1, 2, 3 straw, seed and green material: 00855/M004 (k) LOQ: Analyte 1, 2, 3 straw: 0.05 mg/kg Analyte 1, 2, 3 seed: 0.01 mg/kg Analyte 1, 2, 3 green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132 (m) Storage: Analyte 1, 2, 3 straw: 120 days Analyte 1, 2, 3 seed: 112 days Analyte 1, 2, 3 green material: 165 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
15-2132-03 15-2132-03-T Germany 59609 Anröchte-Berge Europe, North F 2015	Rape Avatar; Winter variety	1) 22.08.2014 2) 20.04.2015 - 20.05.2015 3) 15.07.2015 - 10.08.2015	7.5 7.5	300 300	2.5 2.5	26.05.2015/0 08.06.2015/13	73	green material seed straw 	73 73 88 89 89 88 89 89	<0.05 0.18 <u>≤0.01</u> <0.01 <0.01 0.082 0.071 0.069	<0.05 <0.05 <u>≤0.01</u> <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <u>≤0.01</u> <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 37 44 51 37 44 51	(g) 15-2132 (h) EC (deltamethrin 10 g/L ,flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: Analyte 1, 2, 3 straw, seed and green material: 00855/M004 (k) LOQ: Analyte 1, 2, 3 straw: 0.05 mg/kg Analyte 1, 2, 3 seed: 0.01 mg/kg Analyte 1, 2, 3 green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132 (m) Storage: Analyte 1, 2, 3 straw: 111 days Analyte 1, 2, 3 seed: 104 days Analyte 1, 2, 3 green material: 147 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
15-2132-04 15-2132-04-T Belgium 6210 Villers-Perwin Europe, North F 2015	Rape Dynastie; Winter variety	1) 04.09.2014 2) 20.04.2015 - 18.05.2015 3) 22.07.2015 - 28.07.2015	7.5 7.5	300 300	2.5 2.5	21.05.2015/0 04.06.2015/14	77	green material seed straw 	77 77 87 89 89 87 89 89	<0.05 0.14 <u><0.01</u> <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <u><0.01</u> <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <u><0.01</u> <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 39 48 54 39 48 54	(g) 15-2132 (h) EC (deltamethrin 10 g/L ,flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: Analyte 1, 2, 3 straw, seed and green material: 00855/M004 (k) LOQ: Analyte 1, 2, 3 straw: 0.05 mg/kg Analyte 1, 2, 3 seed: 0.01 mg/kg Analyte 1, 2, 3 green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132 (m) Storage: Analyte 1, 2, 3 straw: 113 days Analyte 1, 2, 3 seed: 106 days Analyte 1, 2, 3 green material: 151 days

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included
(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation
(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse
F field

* prior to last treatment
** residue in control

A 2.2.3.1.1 Study report 16-2044

Comments of zRMS:	Four field residue trials were conducted in northern Europe in 2016 to determine the magnitude of the relevant residues of deltamethrin and BYI 02960 (flupyradifurone) in/on rape (green material, seed and straw) after two spray applications with Deltamethrin & Flupyradifurone EC 085, an EC formulation containing 10 g/L deltamethrin and 75 g/L BYI 02960. Trial GAP: 2 x 5-7.5 g a.s./ha, interval = 14 days, PHI = 45 days. No residues above the respective LOQs were found in the control samples. The average / individual recoveries per fortification level were within the range of 70 – 110% and the RSD values were below 20%, if applicable ($n \geq 3$). Residues of deltamethrin and its isomers AE F108569 and AE 0035073 were determined by LC-MS/MS according to method 00855/M004. The individual LOQs for deltamethrin and its isomers are 0.05 mg/kg in green material and straw and 0.01 mg/kg in seed. No deltamethrin-related residue will be detected in mature seeds (<0.01 mg/kg for each isomer). Residue results (mg/kg) in seed: E: 4x<0.01 RA: 4x<0.01 The storage period of deep-frozen samples used for the analysis of deltamethrin and its isomers (AG02) was between 409 and 506 days. The study is acceptable.

Reference:	**KCA 6.3.1.1/02**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and northern France
Report:	Kaussmann, M.; Kerkering, S.; 2018; 16-2044; M-641044-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP 860.1500, Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	
Duplication (if vertebrate study):	

Materials and method

In 2016, 4 field trials were conducted in northern Europe (northern France, Germany [2] and Belgium) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960). Two applications were made at an interval of 13-14 days, each at a rate of 0.75 L/ha, corresponding to 7.5 g a.s./ha. The last application was done at a crop growth stage of BBCH 73-80 (30% of pods have reached final size to beginning of ripening: seed green). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the treatment in all trials and 38 days after the last treatment in the French trial (as collection of immature seeds was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 34-38 days after the last application (BBCH 84-89), 42-48 days after the last application (BBCH 89) and 50-52 days

after the last application (BBCH 89). All samples were frozen within 24 hours after sampling and stored at a temperature at or below -18°C until analysis (max. 506 days). The maximum storage period of 506 days is covered by the available storage stability study where the analytes have been proven to be stable for a period of at least 36 months (Czarnecki, J. J.; 1996, M-139715-01-1).

The samples were analyzed according to method 00855/M004 (Lakaschus, S.; Winter; O.; 2009; M-356934-01-1). The LOQ in green material and straw was 0.05 mg/kg and 0.01 mg/kg in seed for each compound, cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073).

Results

During the course of this study, the method performance was confirmed by concurrent recoveries.

Full validation data is documented with the method 00855/M004 itself for matrices representing 5 major crop groups, including strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed). However, for rape (green material and straw), a full validation (2 controls and 5 repetitions each at two fortification levels) of additional recoveries was done within the course of the study 15-2132, conducted in paralell. For rape (seed) a full validation (2 controls and 5 repetitions each at two fortification levels) of additional validation recoveries was analyzed within the course of this study. The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always below 20% (

Table A 81).

Details of the application and residue information are summarized in Table A 82. No residues of cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073) at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 81: Recovery data for deltamethrin and its isomers AE 0035073 and AE F108569 in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analysed	a.s./metabolite	n	Fortification level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
16-2044 16-2044-01 to 16-2044-04 16-2044-01-T to 16-2044-04-T GLP: yes 2016	Rape	green material	deltamethrin	2	0.05	107; 108	107	108	108	
				1	0.5	108	108	108	108	
				3	overall		107	108	108	0.5
			AE F108569	2	0.05	101; 103	101	103	102	
				1	0.5	108	108	108	108	
				3	overall		101	108	104	3.5
			AE 0035073	2	0.05	91; 100	91	100	96	
				1	0.5	110	110	110	110	
				3	overall		91	110	100	9.5
		seed	deltamethrin	7	0.01	76; 94; 95; 95; 96; 97; 113	76	113	95	11.3
				5	0.1	69; 94; 95; 95; 98	69	98	90	13.2
				12	overall		69	113	93	11.8
			AE F108569	7	0.01	71; 85; 91; 92; 94; 97; 108	71	108	91	12.4
				5	0.1	66; 89; 91; 93; 93	66	93	86	13.3
				12	overall		66	108	89	12.5
			AE 0035073	7	0.01	70; 89; 91; 92; 96; 97; 116	70	116	93	14.6
				5	0.1	72; 94; 99; 99; 103	72	103	93	13.3
				12	overall		70	116	93	13.4
		straw	deltamethrin	2	0.05	102; 103	102	103	103	
				1	0.5	107	107	107	107	
				3	overall		102	107	104	2.5
			AE F108569	2	0.05	95; 106	95	106	101	
				1	0.5	107	107	107	107	
				3	overall		95	107	103	6.5
			AE 0035073	2	0.05	98; 104	98	104	101	
				1	0.5	108	108	108	108	
				3	overall		98	108	103	4.9

Table A 82: Summary of the trials of study 16-2044

Analyte 1: deltamethrin (determined as deltamethrin, calculated as deltamethrin), Analyte 2: AE F108569 (determined as AE F108569, calculated as AE F108569); Analyte 3: AE 0035073 (determined as AE 0035073, calculated as AE , 0035073)

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2044-01 16-2044-01-T Germany 04824 Beucha OT Wolfshain Europe, North F 2016	Rape Totem; winter-rape, line variety	1) 25.08.2015 2) 14.04.2016 - 08.05.2016 3) 06.07.2016 - 27.07.2016	7.5 7.5	300 300	2.5 2.5	18.05.2016/0 01.06.2016/14	73	green material seed straw	73 73 84 89 89 84 89 89	<0.05 0.092 <0.01 <u><0.01</u> <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <u><0.01</u> <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <u><0.01</u> <0.01 <0.05 <0.05 <0.05	0* 0 38 <u>45</u> 52 38 <u>45</u> 52	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: Analyte 1, 2, 3: straw: 0.05 mg/kg seed: 0.01 mg/kg green material: 0.05 mg/kg (l) Method Validation Data 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3: straw: 454 days seed: 430 days green material: 491 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2044-02 16-2044-02-T Germany 59609 Anröchte-Berge Europe, North F 2016	Rape Elektra; Rape winter	1) 22.08.2015 2) 10.04.2016 - 25.05.2016 3) 20.07.2016 - 10.08.2016	7.5 7.5	300 300	2.5 2.5	30.05.2016/0 13.06.2016/14	79	green material seed straw	79 79 88 89 89 88 89 89	<0.05 0.35 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 36 42 51 36 42 51	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: Analyte 1, 2, 3: straw: 0.05 mg/kg seed: 0.01 mg/kg green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3: straw: 444 days seed: 420 days green material: 479 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2044-03 16-2044-03-T Belgium 6221 Saint-Amand Europe, North F 2016	Rape Visby; Winter rape	1) 05.09.2015 2) 18.04.2016 - 18.05.2016 3) 20.07.2016 - 05.08.2016	7.5 7.5	300 300	2.5 2.5	03.06.2016/0 16.06.2016/13	80	green material seed straw	80 80 89 89 89 89 89 89	<0.05 0.12 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 34 43 50 34 43 50	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: Analyte 1, 2, 3: straw: 0.05 mg/kg seed: 0.01 mg/kg green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3: straw: 443 days seed: 419 days green material: 476 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2044-04 16-2044-04-T France, north 37310 Chambourg sur Indre Europe, North F 2016	Rape Arizona; Winter variety	1) 21.08.2015 2) 06.04.2016 - 03.05.2016 3) 05.07.2016 - 15.07.2016	7.5 7.5	300 300	2.5 2.5	03.05.2016/0 17.05.2016/14	75	green material seed straw 	75 75 82 89 89 89 89	<0.05 0.14 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	0* 0 38 48 52 48 52	(g) 16-2044 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: Analyte 1, 2, 3: straw: 0.05 mg/kg seed: 0.01 mg/kg green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3: straw: 459 days seed: 441 days green material: 506 days

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation

(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse
F field

<<< intended PHI

* prior to last treatment
** residue in control
\# no data available

A 2.2.3.1.1 Study report 16-2131

Comments of zRMS:	This study has not been evaluated by zRMS-PL, because the trials were conducted in southern Europe.

Reference:	**KCA 6.3.1.1/03**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Spain, Portugal, southern France and Italy
Report:	Miara, C.; Nayyar, B.; Kowalski, N.; 2018; 16-2131; M-643446-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP 860.1500, Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	
Duplication (if vertebrate study):	

Materials and methods
In 2016, 6 field trials were conducted in southern Europe (Spain, Portugal, southern France [2] and Italy [2]) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960). Two applications were made at an interval of 14-15 days, each at a rate of 0.75 L/ha, corresponding to 7.5 g a.s./ha. The last application was done at a crop growth stage of BBCH 67-80 (flowering declining to beginning of ripening: seed green). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the treatment in all trials and 38 days after the last treatment in the French and Portuguese trials (as collection of immature seeds and straw was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 37-40 days after the last application (BBCH 82-88), 44-47 days after the last application (BBCH 87-89) and 49-54 days after the last application (BBCH 89). All samples were frozen within 24 hours after sampling and stored at a temperature at or below -18°C until analysis (max. 545 days), except in trials 16-2131-02 and 16-2131-06 where the required temperature was exceeded for 15 hours (maximum temperature within this period was -9°C) during storage of straw and seed samples. However these exceedances were shown to be acceptable as the stability of deltamethrin residues in high-water content matrices was confirmed in a short-term storage stability study, which covers temperatures up to -7°C for 7 days (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1). Additionally the stability of deltamethrin residues in oilseed rape matrices was confirmed in a short-term storage stability study, which covers stability storage for 10 days at -8.5°C followed by 5 hours at +6°C (Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 36 months (Czarnecki, J. J.; 1996, M-139715-01-1).

The samples were analyzed according to method 00855/M004 (Lakaschus, S.; Winter; O.; 2009; M-356934-01-1). The LOQ in green material and straw was 0.05 mg/kg and 0.01 mg/kg in seed for each compound, cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073).

Results

During the course of the present study, the method performance was confirmed by concurrent recoveries. Full validation data is documented with the method 00855/M004 itself for matrices representing 5 major crop groups, including strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed). However for rape (green material and straw), a full validation (2 controls and 5 repetitions each at two fortification levels) of additional recoveries was done within the course of the study 15-2132, conducted in parallel. For rape (seed) a full validation (2 controls and 5 repetitions each at two fortification levels) of additional validation recoveries was analyzed within the course of study 16-2044, also conducted in paralell. The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always below 20% (Table A 83).

Details of the application and residue information are summarized in Table A 84. No residues of cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073) at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 83: Recovery data for deltamethrin and its isomers AR 0035073 and AE F108569 in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analysed	a.s./metabolite	n	Fortification level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
16-2131 16-2131-01 to -06 16-2131-01-T to -06-T GLP: yes 2016	Rape	green material	deltamethrin	3	0.05	97;97;97	97	97	97	0.0
				1	0.5	99	99	99	99	
				4	overall		97	99	98	1.0
			AE F108569	3	0.05	97;97;100	97	100	98	1.8
				1	0.5	97	97	97	97	
				4	overall		97	100	98	1.5
			AE 0035073	3	0.05	88;102;106	88	106	99	9.6
				1	0.5	93	93	93	93	
				4	overall		88	106	97	8.5
		seed	deltamethrin	3	0.01	98;101;101	98	101	100	1.7
				1	0.1	88	88	88	88	
				4	overall		88	101	97	6.4
			AE F108569	3	0.01	89;92;96	89	96	92	3.8
				1	0.1	86	86	86	86	
				4	overall		86	96	91	4.7
			AE 0035073	3	0.01	102;104;118	102	118	108	8.1
				1	0.1	88	88	88	88	
				4	overall		88	118	103	11.9
		straw	deltamethrin	3	0.05	93;103;104	93	104	100	6.1
				1	0.5	100	100	100	100	
				4	overall		93	104	100	5.0
			AE F108569	3	0.05	86;92;101	86	101	93	8.1
				1	0.5	97	97	97	97	
				4	overall		86	101	94	6.9
			AE 0035073	3	0.05	86;97;101	86	101	95	8.2
				1	0.5	95	95	95	95	
				4	overall		86	101	95	6.7

Table A 84: Summary of the trials of study 16-2131

Analyte 1: deltamethrin (determined as deltamethrin, calculated as deltamethrin), Analyte 2: AE F108569 (determined as AE F108569, calculated as AE F108569) , Analyte 3: AE 0035073 (determined as AE 0035073, calculated as AE 0035073)

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2131-01 16-2131-01-T Spain 41410 Carmona Europe, South F 2016	Rape Omega 9; Winter rape	1) 23.10.2015 2) 05.02.2016 - 20.03.2016 3) 20.05.2016 - 10.06.2016	7.5 7.5	300 300	2.5 2.5	23.03.2016/0 06.04.2016/14	80	green material seed straw	80 80 85 88 89 85 88 89	<0.05 0.16 <0.01 <0.01 <0.01 0.12 0.086 0.073	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 0.083 0.065 0.055	0* 0 40 44 49 40 44 49	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: seed: 0.01 mg/kg green material and straw: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 511 days Analyte 1, 2, 3 seed: 491 days Analyte 1, 2, 3 green material: 544 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2131-02 16-2131-02-T Portugal 2080-187 Almeirim Europe, South F 2016	Rape PR45H73; Spring variety	1) 23.10.2015 2) 10.02.2016 - 11.04.2016 3) 15.05.2016 - 15.06.2016	7.5 7.5	300 300	2.5 2.5	21.03.2016/0 05.04.2016/15	78	green material seed straw	78 78 82 87 89 87 89	<0.05 0.097 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	0* 0 38 <u>45</u> 52 <u>45</u> 52	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: seed: 0.01 mg/kg green material and straw: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 507 days Analyte 1, 2, 3 seed: 487 days Analyte 1, 2, 3 green material: 545 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2131-03 16-2131-03-T France, south 13103 St Etienne du Gres Europe, South F 2016	Rape Attletick; Rape oil	1) 28.08.2015 2) 21.03.2016 - 30.04.2016 3) 15.06.2016 - 25.06.2016	7.5 7.5	300 300	2.5 2.5	14.04.2016/0 29.04.2016/15	69	green material seed straw	69 69 79 88 89 88 89	<0.05 0.13 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <u><0.01</u> <0.01 <0.05 <0.05	0* 0 38 46 52 46 52	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: seed: 0.01 mg/kg green material and straw: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 482 days Analyte 1, 2, 3 seed: 464 days Analyte 1, 2, 3 green material: 521 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2131-04 16-2131-04-T France, south 31620 Gargas Europe, South F 2016	Rape DK EXPLICIT; Hybrid	1) 20.08.2015 2) 29.03.2016 - 05.06.2016 3) 23.06.2016 - 30.06.2016	7.5 7.5	300 300	2.5 2.5	25.04.2016/0 10.05.2016/15	72	green material seed straw	72 72 88 89 89 88 89 89	<0.05 0.10 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 38 44 50 38 44 50	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: seed: 0.01 mg/kg green material and straw: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 479 days Analyte 1, 2, 3 seed: 461 days Analyte 1, 2, 3 green material: 510 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2131-05 16-2131-05-T Italy 44022 Comacchio (Ferrara) Europe, South F 2016	Rape PR45D01; Normal ripening cycle variety	1) 21.09.2015 2) 01.04.2016 - 25.04.2016 3) 01.06.2016 - 15.06.2016	7.5 7.5	400 400	1.9 1.9	07.04.2016/0 20.04.2016/13	67	green material seed straw	67 67 82 88 89 82 88 89	<0.05 0.21 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 37 47 54 37 47 54	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: seed: 0.01 mg/kg green material and straw: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 500 days Analyte 1, 2, 3 seed: 487 days Analyte 1, 2, 3 green material: 530 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2131-06 16-2131-06-T Italy 40016 San Giorgio di Piano Bologna Europe, South F 2016	Rape Excalibur; medium variety	1) 15.09.2015 2) 01.04.2016 - 20.04.2016 3) 06.06.2016 - 20.06.2016	7.5 7.5	400 400	1.9 1.9	12.04.2016/0 26.04.2016/14	73	green material seed straw	73 73 86 88 89 86 88 89	<0.05 0.085 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 38 45 52 38 45 52	(g) 16-2131 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: seed: 0.01 mg/kg green material and straw: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 493 days Analyte 1, 2, 3 seed: 480 days Analyte 1, 2, 3 green material: 524 days

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included
(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation

(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse
F field

<<< intended PHI

* prior to last treatment
** residue in control
\# no data available

A 2.2.3.1.2 Study report 16-2169

Comments of zRMS:	This study has not been evaluated by zRMS-PL, because the trials were conducted in southern Europe.

Reference:	**KCA 6.3.1.1/04**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on rape after spray application of deltamethrin & flupyradifurone EC 085 in Spain and Portugal
Report:	Miara, C.; Nayyar, B.; Kowalski, N.; 2018; 16-2169; M-645539-01-1
Authority registration No:	
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009) US EPA OCSPP 860.1500, Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	
Duplication (if vertebrate study):	

Materials and method
In 2016, 2 field trials were conducted in southern Europe (Spain and Portugal) to support the use of Sivanto Energy EC 085 in oilseed rape (rapeseeds). Sivanto Energy is an emulsifiable concentrate formulation containing 10 g/L deltamethrin and 75 g/L flupyradifurone (BYI 02960). Two applications were made at an interval of 14-15 days, each at a rate of 0.75 L/ha, corresponding to 7.5 g a.s./ha. The last application was done at a crop growth stage of BBCH 72-75 (20% to 50% of pods have reached final size). The envisaged PHI was 45 days.

Samples of green material were collected immediately before and after the last treatment in both trials and 36 days after the last treatment in the Spanish trial (as collection of immature seeds and straw was not yet possible). Seed samples, as well as straw samples were collected at two to three different points in time: 36 days after the last application (BBCH 89), 43 days after the last application (BBCH 89) and 50 days after the last application (BBCH 89-97). All samples were frozen within 24 hours after sampling and stored at a temperature at or below -18°C until analysis (max. 511 days), except in trial 16-2169-01 where the required temperature was exceeded for 13 hours (maximum temperature within this period was -8.5°C) and up to +5.9°C for 4 hours. In the trial 16-2169-02 storage temperature rose for some samples above -18°C (up to -8.5°C during storage and up to +5.9°C for 1 hour. However these exceedances were shown to be acceptable as the stability of deltamethrin residues in high-water content matrices was confirmed in a short-term storage stability study, which covers temperatures up to -7°C for 7 days (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1). Additionally the stability of deltamethrin residues in oilseed rape matrices was confirmed in a short-term storage stability study, which covers stability storage for 10 days at -8.5°C followed by 5 hours at +6°C (Winter, O.; Amann, S.; Giesler, W.; 2018; M-626405-01-1). The storage period is covered by the storage stability studies, i.e. the analytes are proven to be stable for a period of at least 36 months (Czarnecki, J. J.; 1996, M-139715-01-1).

The samples were analyzed according to method 00855/M004 (Lakaschus, S.; Winter; O.; 2009; M-356934-01-1). The LOQ in green material and straw was 0.05 mg/kg and 0.01 mg/kg in seed for each compound, cis-deltamethrin and its isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073).

Results
During the course of the present study, the method performance was confirmed by concurrent recoveries. Full validation data is documented with the method 00855/M004 itself for matrices representing 5 major crop groups, including strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat

(straw), barley (whole plant) and oilseed rape (seed). However for rape (green material and straw), a full validation (2 controls and 5 repetitions each at two fortification levels) of additional recoveries was done within the course of the study 15-2132, conducted in parallel. For rape (seed) a full validation (2 controls and 5 repetitions each at two fortification levels) of additional validation recoveries was analyzed within the course of study 16-2044, also conducted in paralell. The average recoveries per fortification level and analyte were within the acceptable range of 70 – 110%. If applicable, the RSD values were always below 20% (Table A 85).

Details of the application and residue information are summarized in Table A 86. No residues of cis-deltamethrin and its is isomers, alpha-*R*-deltamethrin (AE F108569) and trans-deltamethrin (AE 0035073) at or above the limit of quantification of the method were found in any of the untreated samples.

Table A 85: Recovery data for deltamethrin and its isomers AR 0035073 and AE F108569 in rapeseed

Study Trial No. Plot No. GLP Year	Crop	Portion analysed	a.s./metabolite	n	Fortification level (mg/kg)	Recovery (%)				
						Individual recoveries	Min	Max	Mean	RSD
16-2169	Rape	green material	deltamethrin	4	0.05	97; 97; 104; 107	97	107	101	5.0
16-2169-01				3	0.5	102; 103; 106	102	106	104	2.0
16-2169-01-T				7	overall		97	107	102	3.9
and			AE F108569	4	0.05	95; 99; 102; 106	95	106	101	4.6
16-2169-02				3	0.5	104; 104; 107	104	107	105	1.6
16-2169-02-T				7	overall		95	107	102	4.1
GLP: yes			AE 0035073	4	0.05	97; 102; 109; 117	97	117	106	8.2
2016				3	0.5	102; 104; 113	102	113	106	5.5
				7	overall		97	117	106	6.6
		seed	deltamethrin	2	0.01	97; 99	97	99	98	
				1	0.1	100	100	100	100	
				3	overall		97	100	99	1.5
			AE F108569	2	0.01	93; 100	93	100	97	
				1	0.1	96	96	96	96	
				3	overall		93	100	96	3.6
			AE 0035073	2	0.01	91; 101	91	101	96	
				1	0.1	103	103	103	103	
				3	overall		91	103	98	6.5
		straw	deltamethrin	2	0.05	96; 99	96	99	98	
				1	0.5	94	94	94	94	
				3	overall		94	99	96	2.6
			AE F108569	2	0.05	94; 97	94	97	96	
				1	0.5	93	93	93	93	
				3	overall		93	97	95	2.2
			AE 0035073	2	0.05	100; 108	100	108	104	
				1	0.5	95	95	95	95	
				3	overall		95	108	101	6.5

Table A 86: Summary of the trials of study 16-2169

Analyte 1: deltamethrin (determined as deltamethrin, calculated as deltamethrin), Analyte 2: AE F108569 (determined as AE F108569, calculated as AE F108569) , Analyte 3: AE 0035073 (determined as AE 0035073, calculated as AE 0035073)

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2169-01 16-2169-01-T Spain 25281 Llobera Europe, South F 2016	Rape Hidronel	1) 21.09.2015 2) 25.04.2016 - 25.05.2016 3) 14.07.2016 - 21.07.2016	7.5 7.5	360 360	2.1 2.1	17.05.2016/0 01.06.2016/15	72	green material seed straw	72 72 85 89 97 89 97	<0.05 0.086 <0.05 <0.01 <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <0.01 <0.01 <0.05 <0.05	<0.05 <0.05 <0.05 <0.01 <0.01 <0.05 <0.05	0* 0 36 43 50 43 50	(g) 16-2169 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: straw: 0.05 mg/kg seed: 0.01 mg/kg green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 442 days Analyte 1, 2, 3 seed: 441 days Analyte 1, 2, 3 green material: 490 days

Trial No. / Location / EU zone / Year	Commodity / Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest 4. Transplanting	Application rate per treatment			Dates of treatment / Application interval	Growth stage at last treatment	Portion analyzed	Growth stage at sampling	Residues (mg/kg)			PHI (days)	Details on trial
			g a.s./ha	Water (L/ha)	g a.s./hL					Analyte 1 deltamethrin as deltamethrin	Analyte 2 AE F108569 as AE F108569	Analyte 3 AE 0035073 as AE 0035073		
	(a)	(b)				(c)	(d)		(d)				(e)	(f)
16-2169-02 16-2169-02-T Portugal 2140-408 Vale de Cavalos Europe, South F 2016	Rape PR46W14	1) 08.10.2015 2) 17.04.2016 - 04.05.2016 3) 07.07.2016 - 14.07.2016	7.5 7.5	300 300	2.5 2.5	10.05.2016/0 24.05.2016/14	75	green material seed straw	75 75 89 89 89 89 89 89	<0.05 0.11 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	<0.05 <0.05 <0.01 <0.01 <0.01 <0.05 <0.05 <0.05	0* 0 36 43 50 36 43 50	(g) 16-2169 (h) EC (deltamethrin 10 g/L, flupyradifurone 75 g/L) (i) Application method: Spraying (j) Analytical method: 00855/M004 (k) LOQ: straw: 0.05 mg/kg seed: 0.01 mg/kg green material: 0.05 mg/kg (l) Method Validation Data: 00855/M004, 15-2132, 16-2044 (m) Storage: Analyte 1, 2, 3 straw: 457 days Analyte 1, 2, 3 seed: 456 days Analyte 1, 2, 3 green material: 511 days

Abbreviations:

(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Either growth stage description or BBCH Code
(e) Days after last application (Label pre-harvest interval, PHI, underline)
(f) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included
(g) Study reference
(h) Formulation type
(i) Application method
(j) Method information
(k) LOQ
(l) Method validation
(m) Storage (max)
! based on date of analysis
P based on production date

G greenhouse
F field

* prior to last treatment
** residue in control

A 2.2.4 Magnitude of residues in livestock

A 2.2.4.1 Livestock feeding studies

No new data presented.

A 2.2.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

A 2.2.5.1 Distribution of the residue in peel/pulp

No additional studies are submitted

A 2.2.5.2 Processing studies on a core set of representative processes

No additional studies are submitted

A 2.2.6 Magnitude of residues in representative succeeding crops

No additional studies are submitted

A 2.2.7 Other/Special Studies

No additional studies are required.

Appendix 3 Pesticide Residue Intake Model (PRIMo)

A 3.1 TMDI calculations

Flupyradifurone
Two separate residue definitions for monitoring were proposed in food of plant and animal origin: parent flupyradifurone (expressed as flupyradifurone) and second, its metabolite DFA (expressed as DFA). Since the residue definition for risk assessment comprises the sum of both compounds, a TMDI calculation, based on MRL values, is not reasonable.

Deltamethrin
Since the residue definition for risk assessment and for monitoring are different, a TMDI calculation, based on MRL values, is not reasonable.

A 3.2 IEDI calculations

Flupyradifurone

European Food Safety Authority

EFSA PRIMo revision 3.0; 2017/12/11

Flupyradifurone			
LOQs (mg/kg) range from:	0.01	to:	0.02
Toxicological reference values			
ADI (mg/kg bw/day):	0.064	ARfD (mg/kg bw):	0.15
Source of ADI:	EFSA	Source of ARfD:	EFSA
Year of evaluation:	2015	Year of evaluation:	2015

Input values

Details - chronic risk assessment | Supplementary results - chronic risk assessment

Details - acute risk assessment/children | Details - acute risk assessment/adults

Comments:

Normal mode

Chronic risk assessment: JMPR methodology (IEDI/TMDI)

No of diets exceeding the ADI : ---

	Calculated exposure (% of ADI)	MS Diet	Exposure (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commodities	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities	Exposure resulting from MRLs set at the LOQ (in % of ADI)	Exposure resulting from commodities not under assessment (in % of ADI)
TMDI/NEDI/IEDI calculation (based on average food consumption)	17%	NL toddler	11.00	5%	Milk: Cattle	2%	Apples	1%	Wheat		17%
	10%	DE child	6.51	2%	Apples	2%	Milk: Cattle	1%	Wheat		10%
	10%	GEMS/Food G06	6.38	2%	Wheat	2%	Tomatoes	0.5%	Rice		10%
	9%	FR child 3 15 yr	5.84	2%	Milk: Cattle	2%	Wheat	0.5%	Lentils		9%
	9%	UK infant	5.67	3%	Milk: Cattle	1%	Beans	0.9%	Wheat		9%
	9%	NL child	5.64	2%	Milk: Cattle	1%	Wheat	1%	Apples		9%
	9%	DK child	5.52	2%	Rye	1%	Wheat	1%	Milk: Cattle		9%
	8%	UK toddler	5.42	2%	Beans	2%	Milk: Cattle	1%	Wheat		8%
	8%	FR toddler 2 3 yr	5.20	2%	Milk: Cattle	1%	Wheat	0.7%	Beans (with pods)		8%
	8%	RO general	4.99	2%	Wheat	1%	Tomatoes	1.0%	Milk: Cattle		8%
	8%	GEMS/Food G10	4.95	1%	Wheat	0.8%	Tomatoes	0.5%	Potatoes		8%
	8%	ES child	4.92	1%	Wheat	1%	Milk: Cattle	0.7%	Lentils		8%
	8%	GEMS/Food G15	4.86	1%	Wheat	0.7%	Tomatoes	0.6%	Potatoes		7%
	8%	IE adult	4.84	0.8%	Peas	0.8%	Wheat	0.5%	Wine grapes		8%
	8%	GEMS/Food G07	4.83	1%	Wheat	0.6%	Potatoes	0.6%	Tomatoes		7%
	7%	GEMS/Food G08	4.79	1%	Wheat	0.7%	Potatoes	0.7%	Tomatoes		7%
	7%	SE general	4.51	1%	Wheat	1%	Milk: Cattle	0.9%	Bovine: Muscle/meat		7%
	7%	GEMS/Food G11	4.49	1%	Wheat	0.7%	Potatoes	0.6%	Milk: Cattle		7%
	6%	PT general	3.91	1%	Wheat	1.0%	Wine grapes	0.9%	Potatoes		6%
	6%	IT toddler	3.79	2%	Wheat	0.8%	Tomatoes	0.5%	Other cereals		6%
	5%	ES adult	3.36	0.8%	Wheat	0.7%	Lettuces	0.5%	Tomatoes		5%
	5%	FR adult	3.14	0.9%	Wine grapes	0.7%	Wheat	0.4%	Milk: Cattle		5%
	5%	DE women 14-50 yr	3.13	1%	Milk: Cattle	0.7%	Wheat	0.5%	Apples		5%
	5%	DE general	3.11	1%	Milk: Cattle	0.6%	Wheat	0.4%	Apples		5%
	5%	NL general	3.08	0.7%	Milk: Cattle	0.6%	Wheat	0.4%	Potatoes		5%
	5%	IT adult	3.05	1%	Wheat	0.7%	Tomatoes	0.5%	Lettuces		5%
	5%	FR infant	2.96	1%	Milk: Cattle	0.4%	Spinaches	0.4%	Beans (with pods)		5%
	4%	UK vegetarian	2.79	1%	Beans	0.7%	Wheat	0.4%	Tomatoes		4%
	4%	FI 3 yr	2.70	0.8%	Potatoes	0.6%	Cucumbers	0.4%	Wheat		4%
	4%	FI 6 yr	2.31	0.7%	Potatoes	0.4%	Cucumbers	0.3%	Wheat		4%
	4%	UK adult	2.28	0.7%	Beans	0.6%	Wheat	0.4%	Wine grapes		4%
	3%	LT adult	2.18	0.5%	Potatoes	0.4%	Tomatoes	0.4%	Rye		3%
	3%	DK adult	2.16	0.4%	Milk: Cattle	0.4%	Wine grapes	0.4%	Wheat		3%
	2%	PL general	1.47	0.6%	Potatoes	0.5%	Tomatoes	0.4%	Apples		2%
	2%	FI adult	1.32	0.3%	Tomatoes	0.2%	Rye	0.2%	Cucumbers		2%
	1%	IE child	0.88	0.4%	Wheat	0.3%	Milk: Cattle	0.1%	Potatoes		1%

Conclusion:
The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI.
The long-term intake of residues of Flupyradifurone is unlikely to present a public health concern.

Deltamethrin

European Food Safety Authority

EFSA PRIMo revision 3.0; 2017/12/11

(F)

LOQs (mg/kg) range from: to:

Toxicological reference values

ADI (mg/kg bw/day):	**0.01**	ARfD (mg/kg bw):	**0.01**
Source of ADI:		Source of ARfD:	
Year of evaluation:		Year of evaluation:	

Input values

Details - chronic risk assessment

Supplementary results - chronic risk assessment

Details - acute risk assessment/children

Details - acute risk assessment/adults

Comments:

Normal mode

Chronic risk assessment: JMPR methodology (IEDI/TMDI)

No of diets exceeding the ADI : ---

	Calculated exposure (% of ADI)	MS Diet	Exposure (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commodities	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities	Exposure resulting from MRLs set at the LOQ (in % of ADI)	Exposure resulting from commodities not under assessment (in % of ADI)
TMDI/NEDI/IEDI calculation (based on average food consumption)	89%	NL toddler	8.87	62%	Maize/corn	4%	Apples	4%	Rye		89%
	59%	DK child	5.93	48%	Rye	2%	Oat	2%	Rice		59%
	33%	GEMS/Food G06	3.31	11%	Maize/corn	9%	Rice	2%	Wheat		33%
	31%	GEMS/Food G08	3.12	8%	Barley	5%	Rye	4%	Maize/corn		31%
	30%	GEMS/Food G10	2.96	7%	Rice	6%	Maize/corn	5%	Barley		30%
	26%	GEMS/Food G15	2.62	7%	Barley	6%	Maize/corn	2%	Rye		26%
	24%	DE child	2.45	7%	Rye	5%	Apples	2%	Rice		24%
	22%	GEMS/Food G07	2.25	5%	Barley	3%	Maize/corn	2%	Rice		22%
	22%	UK infant	2.17	9%	Maize/corn	4%	Rice	2%	Tea (dried leaves of Camellia sine		22%
	21%	IE adult	2.09	4%	Tea (dried leaves of Camellia sinensis)	2%	Maize/corn	2%	Peas		21%
	20%	GEMS/Food G11	2.02	7%	Barley	2%	Rice	1%	Maize/corn		20%
	18%	DE general	1.83	5%	Rye	4%	Barley	1%	Sugar beet roots		18%
	18%	RO general	1.82	8%	Maize/corn	1%	Rice	1%	Wheat		18%
	18%	FR child 3 15 yr	1.77	4%	Maize/corn	2%	Rice	1%	Lentils		18%
	18%	PT general	1.76	4%	Rice	4%	Maize/corn	2%	Wine grapes		18%
	17%	NL child	1.75	2%	Maize/corn	2%	Apples	2%	Sugar beet roots		17%
	17%	FI 3 yr	1.72	6%	Rye	4%	Oat	3%	Rice		17%
	17%	LT adult	1.66	9%	Rye	1%	Buckwheat and other pseudo-cereals	1%	Rice		17%
	16%	ES child	1.63	3%	Rice	3%	Maize/corn	2%	Olives for oil production		16%
	15%	DE women 14-50 yr	1.50	4%	Rye	2%	Barley	1%	Sugar beet roots		15%
	14%	FI 6 yr	1.39	5%	Rye	2%	Rice	2%	Oat		14%
	14%	ES adult	1.37	4%	Barley	1%	Rice	1%	Olives for oil production		14%
	13%	SE general	1.27	3%	Bovine: Muscle/meat	3%	Rye	2%	Rice		13%
	13%	UK toddler	1.27	3%	Rice	2%	Beans	1.0%	Wheat		13%
	13%	FR toddler 2 3 yr	1.26	3%	Rice	1%	Apples	0.8%	Wheat		13%
	12%	NL general	1.20	3%	Barley	1.0%	Tea (dried leaves of Camellia sinensis)	0.8%	Maize/corn		12%
	12%	FR adult	1.17	4%	Tea (dried leaves of Camellia sinensis)	2%	Wine grapes	0.9%	Rice		12%
	10%	FI adult	1.00	6%	Rye	0.8%	Oat	0.7%	Rice		10%
	10%	DK adult	0.95	5%	Rye	0.7%	Wine grapes	0.5%	Swine: Muscle/meat		10%
	9%	UK vegetarian	0.87	2%	Rice	1%	Tea (dried leaves of Camellia sinensis)	0.9%	Beans		9%
	8%	UK adult	0.79	2%	Rice	1%	Tea (dried leaves of Camellia sinensis)	0.8%	Wine grapes		8%
	6%	IT toddler	0.61	2%	Wheat	1%	Rice	0.5%	Lettuces		6%
	5%	IT adult	0.51	1%	Wheat	1.0%	Rice	0.7%	Lettuces		5%
	4%	FR infant	0.44	0.6%	Apples	0.4%	Potatoes	0.4%	Rice		4%
	3%	IE child	0.31	2%	Rice	0.3%	Wheat	0.2%	Swine: Fat tissue		3%
	3%	PL general	0.31	0.8%	Potatoes	0.8%	Apples	0.2%	Table grapes		3%

Conclusion:
The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI.
The long-term intake of residues of (F) is unlikely to present a public health concern.

A 3.3 IESTI calculations - Raw commodities

Flupyradifurone

Acute risk assessment /children	Acute risk assessment / adults / general population
Details - acute risk assessment /children	Details - acute risk assessment/adults

The acute risk assessment is based on the ARfD.
The calculation is based on the large portion of the most critical consumer group.

Show results of IESTI calculation only for crops with GAPs under assessment

Unprocessed commodities

Results for children				**Results for adults**			
No. of commodities for which ARfD/ADI is exceeded (IESTI):			---	No. of commodities for which ARfD/ADI is exceeded (IESTI):			---
IESTI				**IESTI**			
Highest % of ARfD/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
0.3%	Rapeseeds/canola	0 / 0.28	0.39	0.1%	Mustard seeds	0 / 0.28	0.20
0.2%	Mustard seeds	0 / 0.28	0.29	0.10%	Rapeseeds/canola seeds	0 / 0.28	0.15
Expand/collapse list							
Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)							

Deltamethrin

Acute risk assessment /children	Acute risk assessment / adults / general population
Details - acute risk assessment /children	Details - acute risk assessment/adults

The acute risk assessment is based on the ARfD.
The calculation is based on the large portion of the most critical consumer group.

Show results of IESTI calculation only for crops with GAPs under assessment

Unprocessed commodities

Results for children				**Results for adults**			
No. of commodities for which ARfD/ADI is exceeded (IESTI):			---	No. of commodities for which ARfD/ADI is exceeded (IESTI):			---
IESTI				**IESTI**			
Highest % of ARfD/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
0.1%	Rapeseeds/canola	0 / 0.01	0.01	0.05%	Rapeseeds/canola seeds	0 / 0.01	0.01
Expand/collapse list							
Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)							

A 3.4 IESTI calculations - Processed commodities

Flupyradifurone

Processed commodities

Results for children

No of processed commodities for which ARfD/ADI is exceeded (IESTI): ---

IESTI

Highest % of ARfD/ADI	Processed commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
0.3%	Rapeseeds / processed (not specified)	0 / 0.56	0.48

Results for adults

No of processed commodities for which ARfD/ADI is exceeded (IESTI): ---

IESTI

Highest % of ARfD/ADI	Processed commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)

Deltamethrin

Processed commodities

Results for children				**Results for adults**			
No of processed commodities for which ARfD/ADI is exceeded (IESTI):			---	No of processed commodities for which ARfD/ADI is exceeded (IESTI):			---
IESTI				**IESTI**			
Highest % of ARfD/ADI	Processed commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Processed commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
0.2%	Rapeseeds / processed (r	0 / 0.02	0.02				

Appendix 4 Additional information provided by the applicant

No additional information.