REGISTRATION REPORT

Part B

Section 5

Analytical Methods

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

(Extension of use)

Applicant: Bayer Crop Science Division

Submission date: 31/08/2021

MS Finalisation date: February 2023 (initial Core Assessment)

June 2023 (final Core Assessment)

Version history

When	What
31/08/2021	Original Bayer Crop Science Division submission
February 2023	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~~.
June 2023	Final report (Core Assessment updated following the commenting period) Additional information/assessments included by the zRMS in the report in response to comments received from the cMS and the Applicant are highlighted in yellow. Information no longer relevant ~~is struck through and shaded~~.

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The product Deltamethrin + flupyradifurone EC 85 (10+75 g/L) (DLT+FPF EC 85 / Product Code 102000028562) has been submitted at zonal level to Poland as ZRMS in October 2019 for its use in oilseed rape.

This present dossier is for an extension of use. For such dossier, only new information should be submitted. Currently, there is new information to submit in the present section.
However, because the evaluation of the initial dossier submitted in October 2019 is not finished, no final Registration Report from the ZRMS is available yet.
As a result, all relevant data already submitted in that previous dossier is submitted again and highlighted in purple characters in the present summary part.

zRMS comment:

The product Deltamethrin + flupyradifurone EC 85 (10+75 g/L) (DLT+FPF EC 85 / Product Code 102000028562) has been submitted and evaluated by Poland as zRMS in February 2022 for its use in oilseed rape. The final Registration Report from the zRMS is available yet.

5 Analytical methods

5.1 Conclusion and summary of assessment

zRMS conclusions:

Deltamethrin
Analytical methods for residues (Regulation (EU) N° 283/2013, Annex Part A, point 4.2 & point 7.4.2)
Residue definitions for monitoring purposes

Food of plant origin		Cis-deltamethrin
Food of animal origin		Cis-deltamethrin
Soil		Cis-deltamethrin
Sediment		Cis-deltamethrin
Water	surface	Cis-deltamethrin
	drinking/ground	Cis-deltamethrin
Air		Cis-deltamethrin
Body fluids and tissues		Cis-deltamethrin

Plant residue definition for monitoring (RD-Mo)	Cis - Deltamethrin
Plant residue definition for risk assessment (RD-RA)	Sum of cis - deltamethrin and its alpha-R isomer and trans-isomer
Animal residue definition for monitoring (RD-Mo)	Deltamethrin
Animal residue definition for risk assessment (RD-RA)	Sum of deltamethrin and its alpha-R isomer and trans-isomer

Excerpt from the EFSA Journal 2015;13(11):4309:
*"**Methods of analysis in plants***
During the peer review under Directive 91/414/EEC, an analytical method using GC-ECD, was evaluated for the determination of deltamethrin in plant matrices with a limit of quantification (LOQ) of 0.02 mg/kg in high water content, high fat content, acidic and dry commodities. According to the current guideline, the method is not considered highly specific and a confirmatory method is still required. Independent laboratory validation (ILV) data are available for the determination of deltamethrin in high water content, high fat content and acidic commodities but not for dry commodities (Sweden, 2002; European Commission, 2002). Consequently, an analytical method fully validated and its ILV is still required for dry commodities. An analytical method for the determination of deltamethrin in complex matrices, such as spices and herbal infusions, is not available and it is still required.
Hence it is concluded that there are indications that deltamethrin can be monitored with an LOQ of 0.02 mg/kg in high water content, high fat content, acidic and dry commodities but full validation data are still required (confirmatory method for all the matrices and ILV for dry commodities). A fully validated analytical method for the determination of deltamethrin in spices and herbal infusion is not available and it is still required."

Methods of analysis in livestock
During the peer review under Directive 91/414/EEC, an analytical method using GC-ECD and its ILV, was evaluated for the determination of deltamethrin in food of animal origin with an LOQ of 0.02 mg/kg in milk, meat,

fat, liver, kidney and eggs (Sweden, 2002; European Commission, 2002). The method is not considered highly specific. Consequently, a confirmatory method is still required.
Hence, there are indications that deltamethrin can be monitored in animal tissues, in milk and eggs at the LOQ of 0.02 mg/kg but a confirmatory method is still required."

The Applicant submitted a number of methods for analysis of residues of deltamethrin for the generation of pre-authorization data and methods for post-authorization control and monitoring purposes.
The details of the evaluation of new and additional studies are referred in Appendix 2.
Currently, some methods are evaluated in the AIR process for deltamethrin (DRAR, 2018). This process is not finalized at this time.

Flupyradifurone
According to the EFSA Journal 2015;13(2):4020:

Analytical methods for residues (Annex IIA, point 4.2)
Residue definitions for monitoring purposes

Food of plant origin	Two separate residue definitions: 1) Flupyradifurone 2) DFA, expressed as DFA
Food of animal origin	Two separate residue definitions: 1) Flupyradifurone 2) DFA, expressed as DFA
Soil	Flupyradifurone
Water surface	Flupyradifurone
drinking/ground	Flupyradifurone
Air	Flupyradifurone

Monitoring/ Enforcement methods
Analytical methods for residues (Annex IIA, point 4.2)

Food/feed of plant origin (principle of method and LOQ for methods for monitoring purposes)	HPLC-MS/MS method 01330 with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid extraction. LOQ 0.01 mg/kg for flupyradifurone for lettuce, wheat, orange, rape seed potato, and wheat. LOQ 0.05 mg/kg for hop. LOQ 0.02 mg/kg for DFA for lettuce, wheat, orange, rape seed potato, and wheat. LOQ 0.10 mg/kg for hop.
Food/feed of animal origin (principle of method and LOQ for methods for monitoring purposes)	HPLC-MS/MS acetonitrile/water (4/1, v/v), with the addition of heptane in the cases of fat and milk. LOQ 0.01 mg/kg for flupyradifurone and LOQ 0.02 mg/kg for DFA in fat, liver, kidney, muscle, egg and milk.
Soil (principle of method and LOQ)	HPLC-MS/MS after extraction with acetonitrile. LOQ of 5 µg/kg for flupyradifurone
Water (principle of method and LOQ)	HPLC-MS/MS LOQ for flupyradifurone is 0.05 µg/L in drinking and surface water.
Air (principle of method and LOQ)	HPLC/MS-MS after extraction with acetonitrile. LOQ for flupyradifurone 7 µg /m^3
Body fluids and tissues (principle of method and LOQ)	No methods required.

"Two separate residue definitions for monitoring were proposed in food of plant and animal origin: flupyradifurone and the second, its metabolite DFA expressed as DFA (see Section 3). Appropriate single HPLC-MS/MS methods exist for monitoring residues in food and feed of plant origin with LOQs of 0.01 mg/kg flupyradifurone and with LOQs of 0.02 mg/kg DFA respectively, in all commodities, except for hops, for which the respective LOQs were 0.05 mg/kg a.s. and 0.10 mg/kg DFA. Residues of flupyradifurone and DFA in food of animal origin can be monitored with single HPLC-MS/MS method with LOQs of 0.01 mg/kg a.s. and 0.02 mg/kg DFA respectively, in all matrices.
HPLC-MS/MS methods exist for monitoring flupyradifurone in the environmental matrices with LOQs of 5 µg/kg in soil, 0.05 µg/L in surface water and drinking water and 7 µg/m^3 in the air, respectively. The active substance is not classified or proposed to be classified as toxic according to Regulation (EC) No 1272/2008 (CLP Regulation),6 therefore a method of analysis is not required for body fluids and tissues."

Additionally in EFSA Journal 2020;18(6):6133 it is stated that:
"The availability of analytical enforcement methods for the determination of flupyradifurone and DFA in plant matrices was investigated in the framework of the EU pesticides peer review (EFSA, 2015). It was concluded that a method (method reference number 01330) using HPLC-MS/MS is sufficiently validated for the determination of

flupyradifurone and DFA residues; LOQs achievable with the method were 0.01 and 0.007 mg/kg for flupyradifurone and DFA (expressed as DFA), respectively, in plant matrices with high water (lettuce), high starch (wheat, potato), high acid (oranges) and high oil content (rapeseed). In hops, the validated LOQ for the determination of flupyradifurone is 0.05 mg/kg and for DFA (expressed as DFA), it is 0.03 mg/kg. The validation data for high starch content crop matrix is sufficiently representative to cover high protein content plant matrix (OECD, 2007a–h).
EFSA concludes that a sufficiently validated analytical method is available for the enforcement of flupyradifurone and DFA residues in the crops under consideration."

The Applicant submitted a number of methods for analysis of residues of flupyradifurone and DFA for the generation of pre-authorization data and methods for post-authorization control and monitoring purposes.
The details of the evaluation of new and additional studies are referred in Appendix 2.

An independent laboratory validation (ILV) for the method for the determination of residues of flupyradifurone in drinking or ground water is missing. This data gap should be provided at the renewal of the active substance and plant protection product.

The data are sufficient in order to cover this application.

Sufficiently sensitive and selective analytical methods are available for the active substance(s) and relevant impurities in the plant protection product.
Noticed data gaps are: none

Sufficiently sensitive and selective analytical methods are available for all analytes included in the residue definitions.
Noticed data gaps are:

- an independent laboratory validation (ILV) for the method for the determination of residues of flupyradifurone in drinking or ground water is missing. This data gap should be provided at the renewal of the active substance and plant protection product.

Commodity/crop	**Supported/ Not supported**
Grape (table and wine grape)	Supported
Sweet corn	Supported
Sunflower	Supported
Barley	Supported
Oat	Supported
Wheat	Supported
Millet	Supported
Sorghum	Supported
Corn, field (maize)	Supported

5.2 Methods used for the generation of pre-authorization data (KCP 5.1)

5.2.1 Analysis of the plant protection product (KCP 5.1.1)

5.2.1.1 Determination of active substance and/or variant in the plant protection product (KCP 5.1.1)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin and flupyradifurone in plant protection product is provided as follows:

Comments of zRMS:	The analytical method was assessed in the Registration Report/DLT+FPF EC 85 in February 2022 by zRMS-PL and updated below in the context of the application of SANCO/3030/99 rev.5. The analytical method is considered sufficiently described and fully validated according to SANCO/3030/99 rev.5.

Analytical method

Reference:	**KCP 5.1.1/01**
Title:	Determination of deltamethrin and flupyradifurone in formulations - Assay - HPLC, external standard
Report:	Michel, A.; 2014; AM023614MF1; M-485797-01-1
Authority registration No:	--
Guideline(s):	REGULATION (EC) No 1107/2009 , Comission Regulation 545/2011, 5.1 US EPA OCSPP 830.1800
Deviations:	--
GLP/GEP:	no
Acceptability:	yes
Duplication (if vertebrate study):	no

The same method of analysis (AM023614MF1) is used for both active substances deltamethrin and flupyradifurone. They are separated from formulation components on reversed phase column (XBridge Phenyl from Waters; 50 x 4.6 mm; 2.5 µm) using isocratic elution. After UV detection at 270 nm, the quantitative evaluation is carried out by comparing the peak areas with those of reference items, using an external standard.

Chromatographic conditions
Injection volume: 3 µL
Flow rate: 2 / 3 mL/min
Column temperature: 50 °C
Eluent : A) Phosphoric acid 0.01 mol/L (%v/v)
B) Acetonitrile (%v/v)

	time (min)*	**% A**	**% B**	**flow rate (mL/min)**
Separation	0	80	20	2
	2.0	80	20	2
	2.2	35	65	2
	5.0	35	65	2
Rinsing gradient	5.1	05	95	3
	5.9	05	95	3
	6.0	80	20	2
	7.0	80	20	2

*Adjust the equilibration time according to the pump- and injection system.

Running time: approx. 7 min
Total retention times : deltamethrin approx. 4.4 min
flupyradifurone approx. 1.6 min

Analytical method validation

Reference:	**KCP 5.1.1/02**
Title:	Validation of HPLC-method AM023614MF1 - Determination of deltamethrin and flupyradifurone in formulations - deltamethrin + flupyradifurone EC 85 (10+75 g/L)
Report:	Kienow, A.; Michel, A.; 2014; VB1-AM023614MF1; M-485798-01-1
Authority registration No:	--
Guideline(s):	REGULATION (EC) No 1107/2009 , Comission Regulation (EU) 284/2013, 5.1, SANCO/3030/99 rev. 4, US EPA OCSPP 830.1800
Deviations:	--
GLP/GEP:	no
Acceptability:	yes
Duplication (if vertebrate study):	no

Table 5.2-1 Method suitable for the determination of active substances deltamethrin and flupyradifurone in plant protection product DLT+FPF EC 85

Following parameters were checked		Results
Linearity	For each a.i.: 8 concentrations (single injections); Range: 50-150 % of expected concentration (confer Linearity).	**deltamethrin**: The function is linear in the operation range. Correlation coefficient $r_{k:}$ 0.99995 Regression equation: $y = 0.3313x – 0.0007$ **flupyradifurone**: The function is linear in the operation range. Correlation coefficient $r_{k:}$ 0.99999 Regression equation: $y = 0.3511x + 0.3801$
Precision	6 samples (single injection) from one batch; Assessment of repeatability (confer Precision).	The Relative Standard Deviation (RSD) of the analyte was determined to be: deltamethrin: RSD: 0.09 % Horwitz-Value RSDr(max): 2.74 % Horrat value (Horwitz ratio) Hr: 0.03 flupyradifurone: RSD: 0.06 % Horwitz-Value RSDr(max): 2.02 % Horrat value (Horwitz ratio) Hr: 0.03 The Horrat values (Horwitz ratio, Hr) for both analytes are ≤ 1 and thus, the precision of the analytical method is assessed acceptable. No outliers have been detected.
Accuracy	For each a.i.: 6 samples of laboratory-prepared synthetic formulation containing known weight of analyte; Statistical assessment of the recovery results; Calculation of the confidence interval (confer Accuracy).	**deltamethrin**: Mean Recovery: 99.6 % Confidence interval of recovery: 99.59 ± 0.71 **flupyradifurone**: Mean recovery: 100.2 % Confidence interval of recovery: 100.16 ± 0.29 The method shows no constant systematic error. The method shows no proportional systematic error.
Specificity	Comparison of retention times and UV-spectra from reference items and sample (confer Specificity).	The UV-spectra of analyte in the sample and reference items show no spectral difference; The retention times of analyte and reference items are identical.
Interferences from other substances	Comparison of reference item, sample and blank chromatograms with regard to interferences (confer Interferences).	No interferences were found.
Comment	No deviation from guideline SANCO/3030/99 rev.5.	

Conclusion
The analytical method **AM023614MF1** for the determination of deltamethrin and flupyradifurone in the formulation was found to be valid according to the requirements laid down by SANCO/3030/99 rev.5, all criteria were met.

5.2.1.2 Description of analytical methods for the determination of relevant impurities (KCP 5.1.1)

The preparation Specification 102000028562 does not contain any relevant impurities.

5.2.1.3 Description of analytical methods for the determination of formulants (KCP 5.1.1)

With respect to toxicological, eco-toxicological or environmental aspects the product DLT+FPF EC 85 does not contain any relevant formulants. Therefore, a special analytical method and validation is not needed.

5.2.1.4 Applicability of existing CIPAC methods (KCP 5.1.1)

There is no CIPAC method available for the determination of deltamethrin + flupyradifurone in formulations.

5.2.2 Methods for the determination of residues (KCP 5.1.2)

Deltamethrin
An overview on the acceptable methods and possible data gaps for analysis of residues of deltamethrin for the generation of pre-authorization data is given in the following table. For the detailed evaluation of new/additional studies it is referred to Appendix 2.

For the crop matrices submitted in this Registration Report, additional validations were conducted with a limited dataset of recoveries during the conduct of the residue studies. For the detailed evaluation of these additional validation data (if not already evaluated at EU level), please refer to Appendix 2.

Table 5.2-2: Validated methods for the generation of pre-authorization data

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
Component of residue definition: deltamethrin (cis-, alpha-R-, trans-)				
Plants: strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed) **Further validations:** **Residue studies:** Oilseed rape Grape Sunflower	Primary **Method 00855/M004**	0.01 mg/kg 0.05 mg/kg for wheat (straw), barley (whole plant), oilseed rape (green material), oilseed rape (straw), barley (whole plant), barley (straw), wheat (whole plant), wheat (straw), maize/corn (green material) and maize/corn (rest of plant)	LC-MS/MS	Lakaschus, S, Winter, O., 2009, M-356934-01-1, Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15 **see Appendix 2** **Further validations:** **Reported in residue reports:** M-578527-02-1 (15-2132) M-641044-01-1 (16-2044) M-560047-01-1 (14-2095) M-559743-01-1 (14-2096) M-634135-01-1 (16-2194)

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
Barley Wheat Maize				M-645130-01-1 (16-2145) M-629954-01-1 (16-2195) M-580973-04-1 (15-2131) M-572779-03-1 (15-2130) M-634112-01-1 (16-2034) M-634410-01-1 (16-2035) M-580063-03-1 (15-2127) M-580528-03-1 (15-2129) M-633925-01-1 (16-2032) M-634190-01-1 (16-2033) M-574144-02-1 (15-2133) M-574350-02-1 (15-2134) M-628803-01-1 (16-2192) M-621728-01-1 (16-2100) **see Appendix 2**
Plants: apple (fruit), orange (whole fruit), carrot (root), oilseed rape (seed) and bean (dry seeds) **Further validations:** **Storage stability study:** Tomato fruit Wheat green material Dry peas	Primary / Confirmatory **Method 01207**	0.01 mg/kg	HPLC-MS/MS	Lakaschus, S.; Amann, S.; Winter, O.; Gizler, A., 2013, M-424756-02-1 **Further validations:** **Reported in storage stability study:** M-480441-06-1 (S13-03307) **see Appendix 2**
Component of residue definition: cis-deltamethrin				
Animal products, food of animal origin,... (Residues)	Same as post-authorisation methods			**Please refer to enforcement methods**
Component of residue definition: deltamethrin (cis-, alpha-R-, trans -)				
Soil (Environmental fate)	Not relevant.			
Water (Environmental fate)	Not relevant.			
Soil, water,... (Efficacy)	Not relevant.			
Body fluids (Toxicology)	Primary	50 µg/L (cattle blood)	HPLC-MS/MS	**Method 01127** Krebber, R.; 2009; M-348630-01-1 Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
Air (Exposure)	No relevant.			
Water (Ecotoxicology)	Primary	AE F108565 0.51 mg/L	HPLC-UV	xxx; M-199816-01-1 **see Appendix 2**
	Primary	AE F108565 0.08 mg/L	HPLC-UV	xxx 2001; M-199793-01-1 **see Appendix 2**
	Primary	0.005 µg/L (Deltamethrin	HPLC-MS	**Method 00886**

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
		(alpha-R, cis and trans))		xxx; M-248040-01-1 Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	Primary **Method 00886** Supplementary validation	0.005 µg/L	HPLC-MS	xxx; M-246137-01-2; **see Appendix 2** in support of xxx; M-248040-01-1
	Primary **Method 00886** Supplementary validation	0.005 µg/L	HPLC-MS	xxx; M-246173-01-1; **see Appendix 2** in support of xxx; M-248040-01-1
	Primary	0.002 µg/L	HPLC-MS	**Method 00886/M001** Krebber, R.; Braune, M.; 2007; M-291746-01-1 Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	Primary **Method 00886/M001** Supplementary validation	0.002 µg/L	HPLC-MS	Krebber, R.; Braune, M.; 2007; M-291848-01-1; xxx; M-291885-02-1; **see Appendix 2** in support of Krebber, R.; Braune, M.; 2007; M-291746-01-1
	Primary	1 µg/L in hexane extracts	GC-ECD	xxx; M-256605-01-1 **see Appendix 2**
	Primary	0.0148 µg a.i./L	GC-MS/MS	xxx; M-679497-01-1; **see Appendix 2**
	Primary	0.003 µg a.i./L	GC-MS/MS	xxx; M-686370-01-1; **see Appendix 2**
	Primary	0.003 µg a.i./L	GC-MS/MS	xxx; M-686369-01-1; **see Appendix 2**
Sediment (Ecotoxicology)	Primary	0.1 µg/kg	HPLC-MS/MS	**Method 00877** xxx; M-247896-01-1 Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
	Primary **Method 00877** Supplementary validation	0.1 µg/kg	HPLC-MS/MS	xxx; 2005; M-246137-01-2; **see Appendix 2** in support of xxx; M-247896-01-1
	Primary **Method 00877** Supplementary validation	0.1 µg/kg	HPLC-MS/MS	xxx; M-291818-01-1; xxx; M-291885-02-1; **see Appendix 2** in support of xxx; M-247896-01-1
Avian feed (Ecotoxicology)	Not relevant.			
Bees, flowers/ blossoms, green material, honey/ nectar, pollen and wax (Ecotoxicology)	Primary	0.01 mg/kg	HPLC-MS/MS	**Method 01347** Schoening, R.; Willlmes, J.; 2013; M-444791-01-1 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	Primary **Method 01347** Supplementary validation	0.01 mg/kg	HPLC-MS/MS	Rexer, H. U.; 2013; M-452717-01-1; **see Appendix 2** in support of Schoening, R.; Willlmes, J.; 2013; M-444791-01-1
	Primary **Method 01347** Supplementary validation	0.01 mg/kg	HPLC-MS/MS	Rexer, H. U.; 2013; M-452723-01-1; **see Appendix 2** in support of Schoening, R.; Willlmes, J.; 2013; M-444791-01-1
Employed feeding solutions (Ecotoxicology)	Primary **Method 01347 (modified)**	0.01 mg/kg	HPLC-MS/MS	Kling, A.; 2014; M-477250-01-1 **see Appendix 2**
Spray solution (Ecotoxicology)	Primary	6.24 mg/L	HPLC-PDA	Ripperger, D.; 2016; M-554592-01-1; **see Appendix 2s**
	Primary	6.24 mg/L	HPLC-PDA	Ripperger, D.; 2016; M-554604-01-1; **see Appendix 2**
Solubility in water (Properties)	Primary	0.15 mg/L	HPLC-UV	Wiche, A.; Bogdoll, B.; 2012; M-435779-01-1 **see Appendix 2**

Flupyradifurone

An overview on the acceptable methods for analysis of residues of flupyradifurone for the generation of pre-authorization data is given in the following table. For the detailed evaluation of new/additional studies please refer to Appendix 2.

For the crop matrices submitted in this registration report, additional validations were conducted with a limited dataset of recoveries during the conduct of the residue studies. For the detailed evaluation of these additional validation data (if not already evaluated at EU level), please refer to Appendix 2.

Table 5.2-3: Validated methods for the generation of pre-authorization data

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
Components of residue definition: Plant: Flupyradifurone (FPF), difluoroacetic acid (DFA)				
Plant Dried bean seed Wheat forage Orange fruit Soybean seed Tomato fruit Wheat grain **(Residues)** **Further validations** **Residue studies:** Sunflower Barley Wheat Maize **Storage stability study:** (relevant for DFA) Tomato, fruit Wheat, green material Wheat, grain Grapes, bunches Potato, tuber Pea, dried	Primary / Confirmatory **Method 01304**, also known as RARVP013 to determine - FPF - DFA**	FPF:**0.01 mg/kg** (dried bean seed, wheat forage, orange fruit, soybean seed, tomato fruit, wheat grain) DFA: **0.05 mg/kg*** (dried bean seed, wheat forage, soybean seed, wheat grain) DFA: **0.02 mg/kg*** (orange fruit, tomato fruit)	HPLC-MS/MS	Li, Y.; Schoening, R.; 2012; M-415504-02-2, **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2] For method descriptions please refer also to: Li, Y.; 2010; M-401023-01-2 (RV-001-P10-02); Li, Y.; 2012; M-433355-01-1 (RV-001-P10-03) **Further validations** **Reported in residue reports:** M-645130-01-1 (16-2145) M-634135-01-1 (16-2194) M-629954-01-1 (16-2195) M-634112-01-1 (16-2034) M-634410-01-1 (16-2035) M-633925-01-1 (16-2032) M-634190-01-1 (16-2033) M-628803-01-1 (16-2192) **see Appendix 2** **Reported in storage stability study:** M-480441-06-1 (S13-03307) **see Appendix 2**
Plant Tomato fruit Grape bunch of grape Kidney bean dry seed Barley grain Summer rape seed **(Residues)**	Primary / Confirmatory **Method 01212** to determine - FPF - DFA**	FPF: **0.01 mg/kg** FPF: **0.05 mg/kg** in in barley (straw) and wheat (straw) DFA: **0.02 mg/kg*** DFA: **0.05 mg/kg** in barley (straw) and wheat (straw)	HPLC-MS/MS	Rosati, D.; 2012; M-428017-01-2, **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
Further validations: **Residue studies:** Grape Barley Wheat Maize				**Further validations** **Reported in residue reports:** M-479360-01-1 (12-2126) M-559743-01-1 (14-2096) M-560047-01-1 (14-2095) M-572779-03-1 (15-2130) M-580973-04-1 (15-2131) M-580063-03-1 (15-2127) M-580528-03-1 (15-2129) M-574144-02-1 (15-2133) M-574350-02-1 (15-2134) **see Appendix 2**
Plant **(Residues)** **Storage stability study:** Tomato fruit Wheat green material Wheat grain Grape bunches Potato tuber Dry peas	Primary / Confirmatory (based on QuEChERS) **Method 01207**** to determine - FPF	FPF: 1 mg/kg	HPLC-MS/MS	Lakaschus, S.; Amann, S.; Winter, O.; Gizler, A.; 2013; M-424756-02-1 Not presented in Appendix 2, as method is superseded by supplementary validation in M-480441-06-1 **Reported in storage stability study:** M-480441-06-1 (S13-03307) **see Appendix 2**
Components of residue definition: **Animal: Flupyradifurone (FPF), difluoroacetic acid (DFA)**				
Animal products, Food of animal origin **(Residues)**	Not relevant (no additional studies)			
Components of residue definition: Flupyradifurone				
Soil, water, sediment,... (Environmental fate)	Not relevant.			
Soil, water,... (Efficacy)	Not relevant.			
Feed, body fluids,... (Toxicology)	Not relevant.			
Water (Ecotoxicology)	Primary	0.10 µg/L	HPLC-MS/MS	xxx M-548840-01-1; **see Appendix 2**
	Primary **method 01213**	0.05 µg/L	HPLC-MS/MS	Fargeix, G., Rosati, D., 2012, DAR, NL, 2015[1], **EU agreed**
	Primary **Method 01213** Supplementary validation	0.102 µg a.i./L	HPLC-MS/MS	xxx; M-679497-01-1; **see Appendix 2**
	Primary **Method 01213** Supplementary validation	0.038 µg a.i./L	HPLC-MS/MS	xxx; M-686370-01-1; **see Appendix 2**
	Primary **Method 01213**	0.038 µg a.i./L	HPLC-MS/MS	xxx; M-686369-01-1;

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
	Supplementary validation			**see Appendix 2**
	Primary	0.001 µg/L	HPLC-MS/MS	xxx M-553769-03-1; **see Appendix 2**
	Primary **Method 01182**	0.05 µg/L	HPLC-MS/MS	Krebber, R., Sandau, C., 2010, M-363959-01-1, **see Appendix 2**
	Primary **Method 01182** Supplementary validation	0.0033 µg/L	HPLC-MS/MS	Silke, G.; 2016; M-556348-01-1 **see Appendix 2** in support of Krebber, R., Sandau, C., 2010, M-363959-01-1
	Primary	0.002 mg/L	HPLC-MS/MS	xxx M-547460-01-1; **see Appendix 2**
	Primary	0.0397 mg/L	HPLC-PDA	Aldershof, S.; Bakker, F.; 2019; M-661092-01-1; **see Appendix 2**
	Primary	0.0397 mg/L	HPLC-PDA	Aldershof, S.; Bakker, F.; 2019; M-661091-01-1; **see Appendix 2**
Spray solution (Ecotoxicology)	Primary	47.7 mg/L	HPLC-PDA	Ripperger, D.; 2016; M-554592-01-1; **see Appendix 2**
	Primary	47.7 mg/L	HPLC-PDA	Ripperger, D.; 2016; M-554604-01-1; **see Appendix 2**
Soil,... (Ecotoxicology)	Not relevant.			
Water, buffer solutions,... (Properties)	Not relevant.			

[1] Netherlands, 2015. Draft Assessment Report on the new active substance flupyradifurone prepared by the rapporteur Member State Netherlands in the framework of Regulation (EC) 1107/2009, February, 2015.

[2] 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, 1-106 (doi: 10.2903/j.efsa.2015.4020; update of March 2016)

* LOQ expressed in flupyradifurone equivalents (corresponds to an LOQ of 0.0067 mg/kg when expressed in DFA equivalents (molecular weight of flupyradifurone = 288.68 g/mol; molecular weight of DFA = 96.03 g/mol)).

** These methods are also capable to determine BYI 02960-difluoroethylamino-furanone (DFEAF) and 6-chloronicotinic acid (6-CNA); however, as these compounds are not part of any residue definitions, they will not summarized further below.

5.3 Methods for post-authorization control and monitoring purposes (KCP 5.2)

5.3.1 Analysis of the plant protection product (KCP 5.2)

The analytical method for the determination of the active substance in the plant protection product mentioned under point 5.2.1 can be used for post-authorization control and monitoring purposes.

5.3.2 Description of analytical methods for the determination of residues of Deltamethrin (KCP 5.2)

5.3.2.1 Overview of residue definitions and levels for which compliance is required

Compared to the residue definition proposed in the Draft Assessment Report (incl. its addenda) the current legal residue definition is identical.

Table 5.3-1: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high water content	cis-deltamethrin	0.01 mg/kg (LOQ)	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Plant, high acid content		0.01 mg/kg (LOQ)	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Plant, high protein/high starch content (dry commodities)		~~0.01 mg/kg (LOQ)~~ 0.02 mg/kg (LOQ)	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Plant, high oil content		0.01 mg/kg (LOQ)	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Plant, difficult matrices (hops, spices, tea)		Not available	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Muscle	cis-deltamethrin	~~0.01 mg/kg (LOQ)~~ 0.02 mg/kg	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Milk		~~0.01 mg/kg (LOQ)~~ 0.05 mg/kg	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Eggs		~~0.01 mg/kg (LOQ)~~ 0.02 mg/kg	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Fat		~~0.01 mg/kg (LOQ)~~ 0.1 mg/kg	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Liver, kidney		~~0.01 mg/kg (LOQ)~~ 0.02 mg/kg	~~SANTE/10024/2016~~ Reg. (EU) 2018/832
Soil (Ecotoxicology)	Deltamethrin	0.2 µg/kg (LOQ)	European Commission, Peer review Programme, ECCO Meetings, Deltamethrin, Rapporteur Member State: Sweden, September 1998
		0.05 mg/kg	Common limit SANTE/2020/12830, Rev.1, 24. February 2021
Drinking water (Human toxicology)	Deltamethrin	0.05 µg/L (LOQ)	European Commission, Peer review Programme, ECCO Meetings, Deltamethrin, Rapporteur Member State: Sweden, September 1998
		0.1 µg/L	General limit for drinking water SANTE/2020/12830, Rev.1, 24. February 2021
Surface water (Ecotoxicology)	Deltamethrin	0.05 µg/L (LOQ)	European Commission, Peer review Programme, ECCO Meetings, Deltamethrin, Rapporteur Member State: Sweden, September 1998
Air	Deltamethrin	0.4 µg/m^3 (LOQ: GC-ECD) 0.27 µg/m^3 (LOQ: GC-ECD, GC-MSD)	Deltamethrin, Addendum to Monograph Annex B., Rev.2 July 2002
Tissue (meat or liver)	cis-deltamethrin	LOQ = 0.01 mg/kg	SANTE/2020/12830, Rev.1, 24. February 2021

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Body fluids		LOQ = ~~50 µg/L~~ 0.01 mg/L ~~(cattle blood)~~	~~SANCO/825/00 rev.8.1~~ SANTE/2020/12830, Rev.1, 24. February 2021

5.3.2.2 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin in plant matrices is given in the following tables. For the detailed evaluation of new/additional studies it is referred to Appendix 2.

Table 5.3-2: Validated methods for food and feed of plant origin (required for all matrix types, "difficult" matrix only when indicated by intended GAP)

Component of residue definition: cis-deltamethrin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High water content	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-356076-01-1, report No 00086/M089 **see Appendix 2** *Currently under evaluation in the AIR process of deltamethrin*
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356306-01-1, report No P/B 1681 G **see Appendix 2** *Currently under evaluation in the AIR process of deltamethrin*
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
High acid content	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-356076-01-1, report No 00086/M089 **see Appendix 2** Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin*
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356306-01-1, report No P/B 1681 G **see Appendix 2** Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin*
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
High oil content	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-356076-01-1, report No 00086/M089 **see Appendix 2** Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin*
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356306-01-1, report No P/B 1681 G **see Appendix 2** Peer reviewed in 2016 by BEL for the

Component of residue definition: cis-deltamethrin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
				evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin*
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
High protein/high starch content (dry)	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-356076-01-1, report No 00086/M089 **see Appendix 2** Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin*
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356306-01-1, report No P/B 1681 G **see Appendix 2** Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin*
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
Difficult (if required, depends on intended use)	Not available			

For any special comments or remarkable points concerning the analytical methods for the determination of residues in plant matrices, please refer to Appendix 2.

Table 5.3-3: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	Cross validation of extraction methods for the determination of residues of deltamethrin in plant materials by HPLC-MS/MS. Schoening, R.; Willmes, J.; 2014; MR-14/012; M-481952-02-1
Not required, because:	-

For the detailed evaluation of (additional) studies on extraction efficiency, it is referred to Appendix 2.

5.3.2.3 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin in animal matrices is given in the following tables. For the detailed evaluation of new/additional studies it is referred to Appendix 2.

Table 5.3-4: Validated methods for food and feed of animal origin (if appropriate)

Component of residue definition: cis-deltamethrin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Milk	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-351080-01-1, report No 00086/M090 **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, M; 2009, M-356331-01-1, report No P/B 1682 G **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
Eggs	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-351080-01-1, report No 00086/M090 **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356331-01-1, report No P/B 1682 G **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
Muscle	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-351080-01-1, report No 00086/M090 **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356331-01-1, report No P/B 1682 G **see Appendix 2** Peer reviewed in 2016 byBEL authorities for the evaluation of DLT EW15
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
Fat	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-351080-01-1, report No 00086/M090 **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356331-01-1, report No P/B 1682 G **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15

Component of residue definition: cis-deltamethrin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		
Kidney, liver	Primary	0.01 mg/kg	GC-MS (Three ions monitored)	Weber, H; 2009, M-351080-01-1, report No 00086/M090 **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	ILV	0.01 mg/kg	GC-MS (Three ions monitored)	Merdian, H; 2009, M-356331-01-1, report No P/B 1682 G **see Appendix 2** Peer reviewed in 2016 by BEL authorities for the evaluation of DLT EW15
	Confirmatory (if required)	Not required, due to the specificity of the MS detector		

For any special comments or remarkable points concerning the analytical methods for the determination of residues in animal matrices, please refer to Appendix 2.

Table 5.3-5: Statement on extraction efficiency

	Method for products of animal origin
Required, available from:	The extraction efficiency comparison between methods of metabolism studies and feeding studies was not conducted at the time of the studies, because it was not required. It is not possible to perform an alternative cross-validation study since no incurred animal tissue samples are available.
Not required, because:	

5.3.2.4 Description of methods for the analysis of body fluids and tissues (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin in body fluids and tissues is given in the following table. For the detailed evaluation of new/additional studies it is referred to Appendix 2.

Table 5.3-6: Methods for body fluids and tissues (if appropriate)

Component of residue definition: deltamethrin			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.020 mg/kg (human plasma)	GC-ECD	Tillier, C.; 1988; M-149542-01-1 Sweden, 1998[1]
Primary	0.010 mg/kg (urine)	GC-ECD	Tillier, C.; Devaux, P.; 1981; M-149546-01-1 Sweden, 1998[1]
Primary	0.010 mg/kg (urine faeces)	GC-ECD	Akhtar, M. H.; 1982; M-115658-01-1 Sweden, 1998[1]
Primary **Method 01127**	50 µg/L (cattle blood)	HPLC-MS/MS	Krebber, R.; 2009; M-348630-01-1 Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in*

Component of residue definition: deltamethrin			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
			the AIR process of deltamethrin.

[1] 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.

For any special comments or remarkable points concerning the analytical methods for body fluids and tissues please refer to Appendix 2.

5.3.2.5 Description of methods for the analysis of soil (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin in soil is given in the following tables. For the detailed evaluation of new/additional studies it is referred to Appendix 2.

Table 5.3-7: Validated methods for soil (if appropriate):

Component of residue definition: deltamethrin			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.002 µg/L (Deltamethrin (cis- and trans-)	GC-ECD	Grigor, A.; 1991; M-137727-01-1 EC Peer review Programme[1]
Primary	0.1 µg/kg	HPLC-MS/MS	**Method 00877** xxx; M-247896-01-1 xxx; M-246580-02-1 (including amendment). Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin* **see Appendix 2**
Primary (proposed as new enforcement method)	0.2 µg/kg (cis-Deltamethrin)	HPLC-MS/MS	**Method 01358** Freitag, T.; 2013; M-451547-01-1. *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**

[1] EC Peer review Programme, ECCO Meetings, Deltamethrin, Rapporteur Member State: Sweden, September 1998

For any special comments or remarkable points concerning the analytical methods for soil please refer to Appendix 2.

5.3.2.6 Description of methods for the analysis of water (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin in surface and drinking water is given in the following tables. For the detailed valuation of new/additional studies it is referred to Appendix 2.

Table 5.3-8: Validated methods for water (if appropriate)

Component of residue definition: deltamethrin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water	Primary	0.05 µg/L	GC-ECD	**EM F11/99-0**

Component of residue definition: deltamethrin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
				Martens, R.; 1999; M-192230-01-1 Deltamethrin, Addendum to Monograph Annex B[1]
	Primary (proposed as new enforcement method)	0.05 µg/L (cis-deltamethrin)	HPLC-MS/MS	**Method 01383** Krebber, R.; Braune, M.; 2013; M-464818-01-1 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	ILV	0.05 µg/L (cis-deltamethrin)	HPLC-MS/MS	**ILV to method 01383** Stanislowski, T.; 2013; M-471762-01-1 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
Surface water	Primary	0.05 µg/L	GC-ECD	**EM F11/99-0** Martens, R.; 1999; M-192230-01-1 Deltamethrin, Addendum to Monograph Annex B[1]
	Primary	0.003 µg/L	GC-ECD with MS-MS	**Method 01-31949** Class, T.; 2001; M-240561-01-1 Deltamethrin, Addendum to Monograph Annex B[1]
	Primary	0.005 µg/L (Deltamethrin (alpha-R, cis and trans))	HPLC-MS	**Method 00886** xxx; M-248040-01-1 Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	Primary	0.002 µg/L	HPLC-MS	**Method 00886/M001** Krebber, R.; Braune, M.; 2007; M-291746-01-1 Peer reviewed in 2016 by BEL for the evaluation of DLT EW15 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	Primary (proposed as new enforcement method)	0.05 µg/L (cis-deltamethrin)	HPLC-MS/MS	**Method 01383** Krebber, R.; Braune, M.; 2013; M-464818-01-1 *Currently under evaluation in the AIR process of deltamethrin.* **see Appendix 2**
	ILV	0.05 µg/L (cis-deltamethrin)	HPLC-MS/MS	**ILV to method 01383** Stanislowski, T.; 2013; M-471762-01-1 *Currently under evaluation in the AIR process of deltamethrin* **see Appendix 2**.

[1] Deltamethrin, Addendum to Monograph Annex B, Rev.2 July 2002

For any special comments or remarkable points concerning the analytical methods for water please refer to Appendix 2.

5.3.2.7 Description of methods for the analysis of air (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of deltamethrin in air is given in the following tables. There are no new methods submitted with the present dossier.

Table 5.3-9: Validated methods for air (if appropriate)

Component of residue definition: deltamethrin			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.4 µg/m^3	GC-ECD	Class, T.; 1994; M-203491-01-1 Deltamethrin, Addendum to Monograph Annex B[1]
Primary **Method 01-31951**	0.27 µg/m^3	GC-ECD GC-MSD	Class, T.; 2001; M-075131-01-1 Deltamethrin, Addendum to Monograph Annex B[1]

[1] Deltamethrin, Addendum to Monograph Annex B., Rev.2 July 2002

For any special comments or remarkable points concerning the analytical methods for air it is referred to Appendix 2.

5.3.2.8 Other studies/ information

No other studies or information.

5.3.3 Description of analytical methods for the determination of residues of Flupyradifurone (KCP 5.2)

5.3.3.1 Overview of residue definitions and levels for which compliance is required

Plant and animals:
The residue definitions proposed in the Draft Assessment Report and also supported in the EFSA evaluations (EFSA Journal 2015; 13(2):4020 & EFSA Journal 2020;18(6):6133) are identical with the current legal residue definitions.

Table 5.3-10: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required

Matrix	Residue definition	MRL / limit Flupyradifurone	MRL / limit DFA*	Reference for MRL/level Remarks
Plant, high water content	Two separate residue definitions: - Parent compound flupyradifurone (FPF, expressed in FPF equivalents) - DFA (expressed in DFA equivalents)	0.01 mg/kg (LOQ)	0.02 mg/kg (LOQ)**	~~SANTE/10757/2016; Commission Regulation (EU) 2016/1902~~ Commission Regulation 2021/1842 EFSA, 2020 (Article 12 review)
Plant, high acid content		3.0 mg/kg[1] (MRL grape) 0.01 mg/kg (LOQ)	0.15 mg/kg (MRL grape) 0.02 mg/kg (LOQ)**	
Plant, high protein/high starch content (dry commodities)		0.01 mg/kg (MRL maize kernel) 0.01 mg/kg (LOQ)	~~0.03 mg/kg~~[2] 0.1 mg/kg (MRL maize kernel) 0.02 mg/kg (LOQ)**	
Plant, high oil content		~~0.15 mg/kg~~[2] 0.01* mg/kg (MRL sunflower) 0.01 mg/kg (LOQ)	~~0.08 mg/kg~~[2] 0.05 mg/kg (MRL sunflower) 0.02 mg/kg (lowest MRL)	

Matrix	Residue definition	MRL / limit Flupyradifurone	MRL / limit DFA*	Reference for MRL/level Remarks
			0.02 mg/kg (LOQ)**	
Plant, difficult matrices (e.g. hops, spices, tea)		0.05 mg/kg (LOQ)	0.1 mg/kg (LOQ)	
Muscle	Two separate residue definitions: - Parent compound FPF (expressed in FPF equivalents) - DFA (expressed in DFA equivalents)	0.01 mg/kg (MRL poultry) 0.01 mg/kg (LOQ)	0.15 mg/kg[1] (MRL poultry) 0.02 mg/kg (LOQ)**	
Milk		~~0.15~~ 0.01 mg/kg[1] (MRL ~~bovine~~) 0.01 mg/kg (LOQ)	~~0.04~~ 0.03 mg/kg[2] (MRL sheep) 0.02 mg/kg (LOQ)**	
Eggs		0.01 mg/kg (MRL poultry) 0.01 mg/kg (LOQ)	~~0.15~~ 0.1 mg/kg[2] (MRL poultry) 0.02 mg/kg (LOQ)**	
Fat		0.01 mg/kg (MRL poultry)[1] 0.01 mg/kg (LOQ)	0.03 mg/kg[1] (MRL poultry) 0.02 mg/kg (LOQ)**	
Liver		0.01 mg/kg (MRL poultry)[1] 0.01 mg/kg (LOQ)	0.1 mg/kg[2] (MRL swine) 0.02 mg/kg (LOQ)**	
Kidney		~~0.15~~ 0.01 mg/kg[2] (MRL ~~swine~~ poultry) 0.01 mg/kg (LOQ)	~~0.2~~ 0.02 mg/kg[1] (MRL ~~swine~~ poultry) 0.02 mg/kg (LOQ)**	
Soil (Ecotoxicology)	flupyradifurone	5 µg/kg (LOQ)	-	LOQ below the general limit for soil (0.05 mg/kg)
Drinking water (Human toxicology)	flupyradifurone	0.05 µg/L (LOQ)	-	LOQ below the general limit for drinking water (0. 1 µg/L)
Surface water (Ecotoxicology)	flupyradifurone	0.05 µg/L (LOQ))	-	Lowest endpoint is Chironomus riparius (NOEC, 28d, spiked water) = 6.81 µg/L
Air	flupyradifurone	7 µg/m^3 (LOQ)	-	Lowest endpoint is Rat LC50 inhalation > 4.7 mg/L air/4 h (nose only)
Tissue (meat or liver)	flupyradifurone	0.01 mg/kg (LOQ)	0.02 mg/kg (LOQ)**	~~SANCO/825/00 rev.8.1~~ SANTE/2020/12830, Rev.1, 24. February 2021
Body fluids	flupyradifurone	~~0.05 mg/L (LOQ)~~ 0.01 mg/L	-	~~SANCO/825/00 rev.8.1~~ SANTE/2020/12830, Rev.1, 24. February 2021

* DFA expressed in DFA equivalents

** LOQ expressed in flupyradifurone equivalents (corresponds to an LOQ of 0.007 mg/kg when expressed in DFA equivalents (molecular weight of flupyradifurone = 288.68 g/mol; molecular weight of DFA = 96.03 g/mol))
[1] proposal according to Article 12 review, EFSA Journal 2020;18(6):6133
[2] an evaluation report (dER) was submitted to the Netherlands as EMS in August 2020 in order to propose new MRLs based on the uses supported in this dRR
Remark: MRLs shown in this table refer to the lowest MRLs proposed for the intended uses presented in this dRR.

5.3.3.2 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

An overview on the acceptable methods for analysis of parent flupyradifurone and its metabolite difluoroacetic acid in plant matrices for post-authorization control and monitoring purposes is given in the following tables. No additional methods for the determination of flupyradifurone or difluoroacetic acid have been submitted.

Table 5.3-11: Validated methods for food and feed of plant origin (required for all matrix types, "difficult" matrix only when indicated by intended GAP)

Components of residue definitions: Plant: Flupyradifurone (FPF), difluoroacetic acid (DFA) Animal: Flupyradifurone (FPF), difluoroacetic acid (DFA)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year, Document No (report) / missing / EU agreed**
High water content (lettuce head)	Primary /Confirmatory **Method 01330** to determine - FPF - DFA	0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425848-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Konrad, S.; 2012; M-427133-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
High acid content (orange fruit)	Primary /Confirmatory **Method 01330** to determine - FPF - DFA	0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425848-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Konrad, S.; 2012; M-427133-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
High oil content (Rape seed)	Primary /Confirmatory **Method 01330/M001** to determine - FPF - DFA	0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Teubner, L.; 2012; M-438310-01-1 **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Konrad, S.; 2012; M-439855-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]

Components of residue definitions: Plant: Flupyradifurone (FPF), difluoroacetic acid (DFA) Animal: Flupyradifurone (FPF), difluoroacetic acid (DFA)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year, Document No (report) / missing / EU agreed**
High protein/high starch content (dry) (wheat grain)	Primary /Confirmatory **Method 01330** to determine - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425848-01-1 **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Konrad, S.; 2012; M-427133-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
Difficult (hops cone)	Primary **Method 01330** to determine - FPF - DFA	 0.05 mg/kg 0.1 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425848-01-1 **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV	not required, as the ILV has been performed in three other matrices (lettuce fruit, orange fruit and wheat grain) for this method		

[1] Netherlands, 2015. Draft Assessment Report on the new active substance flupyradifurone prepared by the rapporteur Member State Netherlands in the framework of Regulation (EC) 1107/2009, February, 2015

[2] 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, 1-101

* LOQ expressed in flupyradifurone equivalents (i.e. an LOQ of 0.02 mg/kg corresponds to an LOQ of 0.007 mg/kg when expressed in DFA equivalents (molecular weight of flupyradifurone = 288.68 g/mol; molecular weight of DFA = 96.03 g/mol))

For any special comments or remarkable points concerning the analytical methods for the determination of residues in plant matrices, please refer to Appendix 2.

Table 5.3-12: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	--
Not required, because:	same extraction procedure was used as in plant metabolism studies

The extraction efficiency of the residue method for the determination of the relevant residues of flupyradifurone in plant matrices, consisting of the parent compound and its metabolite DFA, was assured by choosing the same extraction procedures as used in the plant metabolism studies. Nevertheless, an extraction efficiency study was conducted for the data generation method 01304 (DAR, NL, 2015). As the extraction procedures for the monitoring method 01330 are the same as for the data generation method 01304, the results of the study prove also satisfactory extraction efficiency with method 01330 (DAR, NL, 2015).

5.3.3.3 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

An overview on the acceptable methods for analysis of flupyradifurone in animal matrices is given in the following tables. No additional studies have been submitted.

Table 5.3-13: Validated methods for food and feed of animal origin (if appropriate)

Component of residue definition: Flupyradifurone (FPF), difluoroacetic acid (DFA)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Milk	Primary / Confirmatory **Method 01214** to determine - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425837-01-1 **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	see above	Konrad, S.; 2012; M-427160-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
Eggs	Primary / Confirmatory **Method 01214** to determine - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425837-01-1 **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	see above	Konrad, S.; 2012; M-427160-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
Muscle	Primary / Confirmatory **Method 01214** to determine - FPF -DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425837-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	see above	Konrad, S.; 2012; M-427160-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]

Component of residue definition: Flupyradifurone (FPF), difluoroacetic acid (DFA)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Fat	Primary / Confirmatory **Method 01214** to determine - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425837-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	see above	Konrad, S.; 2012; M-427160-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
Kidney, liver	Primary/ Confirmatory **Method 01214** to determine - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425837-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]
	ILV - FPF - DFA	 0.01 mg/kg 0.02 mg/kg*	see above	Konrad, S.; 2012; M-427160-01-1 **EU agreed:** DAR, NL, 2015[1], EFSA, 2015[2]

[1] Netherlands, 2015. Draft Assessment Report on the new active substance flupyradifurone prepared by the rapporteur Member State Netherlands in the framework of Regulation (EC) 1107/2009, February, 2015

[2] 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, 1-101

* LOQ expressed in flupyradifurone equivalents (corresponds to an LOQ of 0.0067 mg/kg when expressed in DFA equivalents (molecular weight of flupyradifurone = 288.68 g/mol; molecular weight of DFA = 96.03 g/mol))

Table 5.3-14: Statement on extraction efficiency

	Method for products of animal origin
Required, available from:	-
Not required, because:	same extraction procedure was used as in livestock metabolism studies

The extraction efficiency of the residue method for the determination of the relevant residues of flupyradifurone in livestock matrices, consisting of the parent compound and its metabolite DFA, was assured by choosing the same extraction procedures as used in the livestock metabolism studies.

5.3.3.4 Description of methods for the analysis of body fluids and tissues (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of flupyradifurone in body fluids and tissues is given in the following table. For the detailed evaluation of new studies it is referred to Appendix 2.

Table 5.3-15: Methods for body fluids and tissues (if appropriate)

Component of residue definition: Plasma (body fluid): Flupyradifurone (FPF)			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary/ Confirmatory **Method 01495**	0.05 mg/L (plasma)	HPLC-MS/MS (2 MRM transitions)	Kaussmann, M.; 2016; M-570324-01-1

Component of residue definition: Plasma (body fluid): Flupyradifurone (FPF)			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
to determine - FPF			**see Appendix 2**
Component of residue definition: Tissues: Flupyradifurone (FPF)			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary / Confirmatory **Method 01214** to determine - FPF -DFA	 0.01 mg/kg (tissues) 0.02 mg/kg* (tissues)	HPLC-MS/MS 2 transitions (MRM) 2 stationary phases	Schulte, G.; Bauer, J.; 2012; M-425837-01-1 **EU agreed :** DAR, NL, 2015[1], EFSA, 2015[2]

[1] Netherlands, 2015. Draft Assessment Report on the new active substance flupyradifurone prepared by the rapporteur Member State Netherlands in the framework of Regulation (EC) 1107/2009, February, 2015

[2] 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, 1-101

For any special comments or remarkable points concerning the analytical methods for body fluids and tissues please refer to Appendix 2.

zRMS comments:

Applicant provided new analytical method (Kaussmann, M.; 2016) for the determination of Flupyradifurone in plasma with a LOQ of 0.05 mg/L.
According to the guidance document SANTE/2020/12830, rev.1 "*The LOQ shall meet 0.01 mg/L for body fluids and 0.01 mg/kg for body tissues. Higher LOQs are acceptable for analytically challenging analytes, if justified."*
Considering the above, this method for determination of Flupyradifurone in plasma does not fully comply with SANTE/2020/12830, rev.1. Nevertheless, method can be regarded as fit for purpose for the determination of Flupyradifurone in plasma with a LOQ of 0.05 mg/L.
More information is presented in Appendix 2.

During commenting period the Applicant submitted the following information:
Flupyradifurone: for body fluids a new method with an LOQ of 0.01 mg/L is available. This new method was submitted with the Annex I renewal of flupyradifurone (December 2022) and is currently being evaluated at EU level. If requested, the study can also be submitted for this Sivanto Energy submission.
In our opinion these data may be provided at the renewal of the active substance and plant protection product.

5.3.3.5 Description of methods for the analysis of soil (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of flupyradifurone in soil is given in the following table. For the detailed valuation of new studies it is referred to Appendix 2.
The following method for the analysis of soil has already been evaluated and accepted at EU level and therefore, no additional studies for soil have been submitted.

Table 5.3-16: Validated methods for soil (if appropriate)

Component of residue definition: flupyradifurone			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	5 µg/kg	HPLC-MS/MS	xxx **EU agreed**

For any special comments or remarkable points concerning the analytical methods for soil please refer to Appendix 2.

5.3.3.6 Description of methods for the analysis of water (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of flupyradifurone in surface and drinking water is given in the following table. For the detailed valuation of new studies it is referred to Appendix 2.

Table 5.3-17: Validated methods for water (if appropriate)

Component of residue definition: flupyradifurone				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water	Primary **method 01213**	0.05µg/L	HPLC-MS/MS	Fargeix, G., Rosati, D., 2012, DAR, NL, 2015[1], **EU agreed**
Surface water	Primary **method 01213**	0.05µg/L	HPLC-MS/MS	Fargeix, G., Rosati, D., 2012, DAR, NL, 2015[1], **EU agreed**

1 Netherlands, 2015. Draft Assessment Report on the new active substance flupyradifurone prepared by the rapporteur Member State Netherlands in the framework of Regulation (EC) 1107/2009, February, 2015

zRMS comments:

According to the guidance document SANTE/2020/12830, rev.1 *an ILV must be conducted for drinking water or ground water, with the same number of fortification levels and fortified samples per level as for the primary method*.
Applicant did not provide ILV for drinking or ground water.
Considering the above, an independent laboratory validation (ILV) for the method for the determination of residues of flupyradifurone in drinking or ground water is missing. This data gap should be provided at the renewal of the active substance and plant protection product.

5.3.3.7 Description of methods for the analysis of air (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of flupyradifurone in air is given in the following table. For the detailed evaluation of new studies please refer to Appendix 2.
The following method for the analysis of air has already been evaluated and accepted at EU level and therefore, no additional studies for air have been submitted.

Table 5.3-18: Validated methods for air (if appropriate)

Component of residue definition: flupyradifurone			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	7 µg/m^3	HPLC-MS/MS	Heinz, N., 2011, DAR, Netherlands, 2015 **EU agreed**

For any special comments or remarkable points concerning the analytical methods for air it is referred to Appendix 2.

5.3.3.8 Other studies/ information

No other studies/information.

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**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 01	Lakaschus, S.; Winter; O.	2009	Validation of BCS Method 00855/M004 for the Determination of cis-deltamethrin, trans-deltamethrin and alpha-R-deltamethrin in foodstuff of plant origin Report No.: 00855/M004, Edition Number: M-356934-01-1 Method Report No.: BAY-0904V Eurofins Analytik GmbH, Dr. Specht Laboratorien, Hamburg, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y evaluated in the RR for DLT+FPF EC 85 on 02.2022**
KCP 5.1 / 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	**Y evaluated in the RR for DLT+FPF EC 85 on 02.2022**
KCP 5.1 / 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	**Y evaluated in the RR for DLT+FPF EC 85 on 02.2022**
~~KCP 5.1 / 04~~ ~~**... also filed: KCA 6.3.1.1 / 02**~~	~~Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.~~	~~2016~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on grape after high or low-volume spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy~~ ~~Report No.: 14-2095, Edition Number: M-560047-01-1~~ ~~Bayer CropScience AG, Monheim, Germany~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 05 **... also filed: KCA 6.3.1.1 / 01** **KCA 6.3.1.2 / 01**	Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.	2016	Determination of the residues of BYI 02960 and deltamethrin in/on grape after high and low-volume spray application of deltamethrin & flupyradifurone EC 085 in Germany and France (North) Report No.: 14-2096, Edition Number: M-559743-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**

Data Point	Author(s)	Year	Title Company Report No. Source GLP or GEP status published or not	Vertebrate study Y/N	Owner	Previously used Y/N If yes, for which data point?
~~KCP 5.1 / 06~~ ~~**... also filed: KCA 6.3.2.1 / 02**~~	~~Kaussmann, M.; Kowalski, N.~~	~~2018~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in Italy, southern France, Spain and Greece~~ ~~Report No.: 16-2194, Edition Number: M-634135-01-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 07 **... also filed: KCA 6.3.2.1 / 01** **KCA 6.3.2.2 / 01**	Miara, C.; Kowalski, N.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in northern France, Hungary, The United Kingdom and Poland Report No.: 16-2145, Edition Number: M-645130-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
~~KCP 5.1 / 08~~ ~~**... also filed: KCA 6.3.2.1 / 03**~~	~~Kaussmann, M.; Kowalski, N.~~	~~2018~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy~~ ~~Report No.: 16-2195, Edition Number: M-629954-01-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
~~KCP 5.1 / 09~~ ~~**... also filed: KCA 6.3.3.1 / 03**~~	~~Noss, G.~~	~~2017~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in France (South), Italy, Spain and Greece~~ ~~Report No.: 15-2130, Edition Number: M-572779-03-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~**... amended: 2017-10-17**~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 10 **... also filed: KCA 6.3.3.1 / 01** **KCA 6.3.3.2 / 01**	Schulte, G.	2017	Amendment no. 3 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring barley after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and United Kingdom Report No.: 15-2131, Edition Number: M-580973-04-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-09-22** GLP/GEP: Yes unpublished	No	Bayer	**N**

Data Point	Author(s)	Year	Title Company Report No. Source GLP or GEP status published or not	Vertebrate study Y/N	Owner	Previously used Y/N If yes, for which data point?
~~KCP 5.1 / 11~~ ~~**... also filed: KCA 6.3.3.1 / 04**~~	~~Kaussmann, M.; Miara, C.~~	~~2018~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy, Spain and Greece~~ ~~Report No.: 16-2034, Edition Number: M-634112-01-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 12 **... also filed: KCA 6.3.3.1 / 02** **KCA 6.3.3.2 / 02**	Kaussmann, M.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring barley after spray application of deltamethrin & flupyradifurone EC 085 in the Netherlands, Germany and Belgium Report No.: 16-2035, Edition Number: M-634410-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
~~KCP 5.1 / 13~~ ~~**... also filed: KCA 6.3.4.1 / 03**~~	~~Schulte, G.~~	~~2017~~	~~Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in Italy, Spain and Portugal~~ ~~Report No.: 15-2127, Edition Number: M-580063-03-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~**... amended: 2017-09-22**~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 14 **... also filed: KCA 6.3.4.1 / 01** **KCA 6.3.4.2 / 01**	Schulte, G.	2017	Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on spring wheat and winter wheat after spray application of deltamethrin & flupyradifurone EC 085 in Germany, the Netherlands and Belgium Report No.: 15-2129, Edition Number: M-580528-03-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-09-22** GLP/GEP: Yes unpublished	No	Bayer	**N**
~~KCP 5.1 / 15~~ ~~**... also filed: KCA 6.3.4.1 / 04**~~	~~Kaussmann, M.; Kerkering, S.~~	~~2018~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy and Spain~~ ~~Report No.: 16-2032, Edition Number: M-633925-01-1~~ ~~Bayer AG, Crop Science Division, 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	Previously used Y/N If yes, for which data point?
KCP 5.1 / 16 **... also filed:** **KCA 6.3.4.1 / 02** **KCA 6.3.4.2 / 02**	Kaussmann, M.; Kerkering, S.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring wheat after spray application of deltamethrin & flupyradifurone EC 085 in Belgium, Germany and the Netherlands Report No.: 16-2033, Edition Number: M-634190-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
~~KCP 5.1 / 17~~	~~Schulte, G.~~	~~2017~~	~~Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, France (South) and Italy~~ ~~Report No.: 15-2133, Edition Number: M-574144-02-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~**... amended: 2017-05-03**~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 18 **... also filed:** **KCA 6.3.5.1 / 02** **KCA 6.3.5.2 / 02**	Schulte, G.; Kerkering, S.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and the Netherlands Report No.: 16-2192, Edition Number: M-628803-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
~~KCP 5.1 / 19~~	~~Schulte, G.; Kuester, S.; Kerkering, S.~~	~~2018~~	~~Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, southern France and Italy~~ ~~Report No.: 16-2100, Edition Number: M-621728-01-1~~ ~~Bayer AG, Crop Science Division, Monheim, Germany~~ ~~GLP/GEP: Yes~~ ~~unpublished~~	~~No~~	~~Bayer~~	
KCP 5.1 / 20 **... also filed:** **KCA 6.3.5.1 / 01** **KCA 6.3.5.2 / 01**	Schulte, G.	2017	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and the Netherlands Report No.: 15-2134, Edition Number: M-574350-02-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-05-03** GLP/GEP: Yes unpublished	No	Bayer	**N**

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 21	Schoening, R.; Willmes, J.	2014	Cross validation of extraction methods for the determination of residues of deltamethrin in plant materials by HPLC-MS/MS Report No.: MR-14/012, Edition Number: M-481952-02-1 Bayer CropScience AG, Monheim, Germany **... amended: 2014-04-11** GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 22	Lakaschus, S.; Amann, S.; Winter, O.; Gizler, A.	2013	Validation of the BCS method no. 01207 (based on modified QuEChERS method) for the determination of selected BCS analytes and their metabolites in carrot, apple, orange, oilseed rape seed and beans Report No.: S10-00279, Edition Number: M-424756-02-1 Eurofins Agroscience Services Chem GmbH (EAS Chem), Hamburg, Germany **... amended: 2013-12-11** GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 23 **... also filed: KCA 6.1 / 01**	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	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 24 **... also filed: KCP 5.2 / 05**	Krebber, R.	2009	Analytical method 01127 for the determination of cyfluthrin and deltamethrin in blood by HPLC-MS/MS Report No.: MR-08/176, Edition Number: M-348630-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 25 **... also filed: KCP 10.2.1 / 01**	xxx.	2001	Acute toxicity to Oncorhynchus mykiss (rainbow trout) AE F108565 (metabolite of deltamethrin) substance, pure Code: AE F108565 00 1B99 0001 Report No.: C010902, Edition Number: M-199816-01-1 xxx GLP/GEP: Yes unpublished	Yes	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 26 **... also filed:** **KCP 10.2.1 / 02**	Sowig, P.; Gosch, H.	2001	Acute toxicity to Daphnia magna (Waterflea) AE F108565 (Metabolite of deltamethrin) substance, pure Code: AE F108565 00 1B99 0001 Report No.: C010889, Edition Number: M-199793-01-1 Aventis CropScience GmbH, Frankfurt am Main, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 27 **... also filed:** **KCP 5.2 / 11**	Brumhard, B.	2005	Analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS Report No.: C047388, Edition Number: M-248040-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 28 **... also filed:** **KCP 10.2.3 / 02**	xxx	2005	Biological effects and fate of deltamethrin EW 015 in outdoor mesocosm ponds Report No.: HBF/BT 07, Edition Number: M-246137-01-2 xxx GLP/GEP: Yes unpublished	Yes	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 29 **... also filed:** **KCP 10.2.2 / 03**	Heimbach, F.; Arnold, M.	2005	Bioassay on the effects of Deltamethrin EW 015 on Gammarus pulex in mesocosm water Report No.: HBF/BT 08, Edition Number: M-246173-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 30 **... also filed:** **KCP 5.2 / 06**	Brumhard, B.	2005	Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS Report No.: C047210, Edition Number: M-247896-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 31 **... also filed: KCP 5.2 / 07**	Brumhard, B.	2009	Analytical method 00877 for the determination of total residues of Deltamethrin (AE F032640) in/on soil and sediment by HPLC-MS/MS Report No.: 00877, Edition Number: M-246580-02-1 Method Report No.: MR-081/04 Bayer CropScience AG, Monheim, Germany **... amended: 2009-03-31** GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 32	Brumhard, B.; Loehrwald, K.H.	2007	Analysis of deltamethrin concentrations in sediment samples of ECT study no. P1MA Report No.: MR-07/297, Edition Number: M-291818-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 33 **... also filed: KCP 10.2.2 / 08**	xxx	2007	Deltamethrin EW 15 G: Acute and chronic effects to different life stages of the isopod Assellus aquaticus L in a natural water-sediment-system Report No.: P1MA, Edition Number: M-291885-02-1 xxx **... amended: 2007-08-29** GLP/GEP: Yes unpublished	Yes	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 34 **... also filed: KCP 5.2 / 12**	Krebber, R.; Braune, M.	2007	Modification M001 of analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS Report No.: 00886/M001, Edition Number: M-291746-01-1 Method Report No.: MR-07/296 Bayer CropScience AG, Monheim, Germany GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 35	Krebber, R.; Braune, M.	2007	Analysis of deltamethrin concentrations in water samples of ECT study no. P1MA Report No.: MR-07/295, Edition Number: M-291848-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 36 **... also filed: KCP 10.2.3 / 01**	xxx	2005	Effects of Deltamethrin EW 15 on rainbow trout in aquatic outdoor microcosm enclosures Report No.: ALT.JD.2005.1, Edition Number: M-256605-01-1 xxx GLP/GEP: Yes unpublished	Yes	Bayer	**N**
KCP 5.1 / 37 **... also filed: KCP 10.2.1 / 04**	xxx	2020	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to rainbow trout (Oncorhynchus mykiss) in a 96-hour semi-static test Report No.: EBRV0196, Edition Number: M-679497-01-1 xxx GLP/GEP: Yes unpublished	Yes	Bayer	**N**
KCP 5.1 / 38 **... also filed: KCP 10.2.1 / 06**	Bebon, R.; Sonntag, F.	2020	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to Daphnia magna in a semi-static 48-hour immobilisation test - Final report - Report No.: EBRV0195, Edition Number: M-686370-01-1 IBACON GmbH, Rossdorf, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 39 **... also filed: KCP 10.2.1 / 08**	Bebon, R.; Sonntag, F.	2020	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to larvae of Chironomus riparius in a semi-static 48-hour immobilisation test - Final report - Report No.: EBRV0194, Edition Number: M-686369-01-1 IBACON GmbH, Rossdorf, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 40	Schoening, R.; Willlmes, J.	2013	Residue analytical method 01347 for the determination of residues of deltamethrin by HPLC with electrospray and MS/MS - detection Report No.: MR-012/067, Edition Number: M-444791-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 41 **... also filed: KCP 10.3.1.6 / 01**	Rexer, H. U.	2013	Assessment of side effects on the honeybee (Apis mellifera L.), exposed to Phacelia tanacetifolia, sprayed sequentially with deltamethrin during flowering in a long-term field study in North Alsace, France Report No.: S10-03820, Edition Number: M-452717-01-1 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**

Data Point	Author(s)	Year	Title Company Report No. Source GLP or GEP status published or not	Vertebrate study Y/N	Owner	Previously used Y/N If yes, for which data point?
KCP 5.1 / 42 **... also filed: KCP 10.3.1.6 / 02**	Rexer, H. U.	2013	Assessment of side effects on the honeybee (Apis mellifera L.), exposed to Phacelia tanacetifolia, sprayed sequentially with deltamethrin during flowering in a long-term field study in Mid Alsace, France Report No.: S10-03824, Edition Number: M-452723-01-1 Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 43 **... also filed: KCP 10.3.1.2 / 01**	Kling, A.	2014	Deltamethrin EW 15B G - Assessment of chronic effects to the honeybee, Apis mellifera L., in a 10 days continuous laboratory feeding test Report No.: S13-00151, Edition Number: M-477250-01-1 Eurofins-GAB GmbH, Niefern-Oeschelbronn, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 44 **... also filed: KCP 10.6.2 / 02 KCP 6.5.1 / 01**	Ripperger, D.	2016	Deltamethrin + flupyradifurone EC 85 (10+75 g/L): Effects on the seedling emergence of non-target terrestrial plant species under greenhouse conditions Report No.: S15-01670, Edition Number: M-554592-01-1 Eurofins Agroscience Services EcoChem GmbH, Niefern-Oeschelbronn, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 45 **... also filed: KCP 10.6.2 / 01 KCP 6.5.2 / 01**	Ripperger, D.	2016	Deltamethrin + flupyradifurone EC 85 (10+75 g/L): Effects on the vegetative vigour of non-target terrestrial plant species under greenhouse conditions Report No.: S15-01671, Edition Number: M-554604-01-1 Eurofins Agroscience Services EcoChem GmbH, Niefern-Oeschelbronn, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 46 **... also filed: KCA 7.1.4 / 02**	Wiche, A.; Bogdoll, B.	2012	AE F108565 (Br2CA): Solubility in water at pH 5, pH 7 and pH 9 Report No.: PA10/073, Edition Number: M-435779-01-1 Bayer CropScience AG, Frankfurt am Main, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
~~KCP 5.1 / 47~~	~~Meilland-Berthier, I.~~	~~2014~~	~~Determination of the residues of BYI 02960 in/on grape after high and low-volume spray application of BYI 02960 SL 200 in southern France, Spain, Italy and Greece~~ ~~Report No.: 12-2126, Edition Number: M-479360-01-1~~ ~~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**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 48 **... also filed:** **KCP 10.2.1 / 03**	xxx	2016	Acute toxicity of deltamethrin + flupyradifurone EC 85 (10+75 g/L) to the rainbow trout (Oncorhynchus mykiss) under static conditions Report No.: 007SRLS15C08, Edition Number: M-548840-01-1 xxx GLP/GEP: Yes unpublished	Yes	Bayer	**N**
KCP 5.1 / 49 **... also filed:** **KCP 10.2.1 / 05**	Matlock, D.; Moore, S.	2016	Amendment no. 2 - Acute toxicity of deltamethrin + flupyradifurone EC 85 to Daphnia magna under static conditions - Final report - Report No.: EBRVR015, Edition Number: M-553769-03-1 SynTech Research Laboratory Services, LLC, Stilwell, KS, USA **... amended: 2016-10-19** GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 50	Krebber, R.; Sandau, C.	2010	Method 01182 for the determination of BYI 02960 in test water from aquatic toxicity tests by HPLC-MS/MS Report No.: 01182, Edition Number: M-363959-01-1 Method Report No.: MR-10/002 Bayer CropScience AG, Monheim, Germany GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1 / 51 **... also filed:** **KCP 10.2.1 / 07**	Silke, G.	2016	Acute toxicity of deltamethrin + flupyradifurone EC 85 (10+75) G to larvae of Chironomus riparius in a 48 h static laboratory test system Report No.: EBRVN060, Edition Number: M-556348-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 52 **... also filed:** **KCP 10.2.1 / 09**	Matlock, D.; Moore, S.	2015	Toxicity of deltamethrin + flupyradifurone EC 85 to the green algae Pseudokirchneriella subcapitata during a 72 hour exposure Report No.: EBRVR016, Edition Number: M-547460-01-1 SynTech Research Laboratory Services, LLC, Stilwell, KS, USA GLP/GEP: Yes unpublished	No	Bayer	**N**

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.1 / 53 **... also filed: KCP 10.3.2.4 / 03**	Aldershof, S.; Bakker, F.	2019	A field study to assess the effects of deltamethrin + flupyradifurone EC 85 (10+75 g/L) on the non-target, surface- and plant-dwelling, arthropod fauna of a grassland habitat (off-crop) in The Netherlands during spring/summer Report No.: B168FFN, Edition Number: M-661092-01-1 Eurofins MITOX, Amsterdam, Netherlands GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1 / 54 **... also filed: KCP 10.3.2.4 / 04**	Aldershof, S.; Bakker, F.	2019	A field study to assess the effects of deltamethrin + flupyradifurone EC 85 (10+75 g/L) on the non-target, surface- and plant-dwelling, arthropod fauna of a grassland habitat (off-crop) in SW France during spring/summer Report No.: B169FFN, Edition Number: M-661091-01-1 Eurofins MITOX, Amsterdam, Netherlands GLP/GEP: Yes unpublished	No	Bayer	**N**
KCP 5.1.1 / 01	Michel, A.	2014	Determination of deltamethrin and flupyradifurone in formulations - Assay - HPLC, external standard Report No.: AM023614MF1, Edition Number: M-485797-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.1.1 / 02	Kienow, A.; Michel, A.	2014	Validation of HPLC-method AM023614MF1 - Determination of deltamethrin and flupyradifurone in formulations - deltamethrin + flupyradifurone EC 85 (10+75 g/L) Report No.: VB1-AM023614MF1, Edition Number: M-485798-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
NKCP 5.2 / 01	Weber, H.	2009	Validation of enforcement method DFG S19 (L 00.00-34) (BCS method ID 00086/M089) for the determination of cis-deltamethrin (AE F032640) in/on foodstuff of plant origin Report No.: S09-00553, Edition Number: M-351076-01-1 Eurofins Analytik GmbH, Dr. Specht Laboratorien, Hamburg, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.2 / 02	Merdian, H.	2009	Independent laboratory validation of the DFG method S19 (BCS method 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in plant materials, using GC/MS Report No.: P/B 1681 G, Edition Number: M-356306-01-1 PTRL Europe GmbH, Ulm, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 03	Weber, H.	2009	Validation of enforcement method DFG S19 (L 00.00-34) (BCS method ID 00086/M090) for the determination of residues cis-deltamethrin (AE F032640) in/on foodstuff of animal origin Report No.: S09-00551, Edition Number: M-351080-01-1 Eurofins Analytik GmbH, Dr. Specht Laboratorien, Hamburg, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 04	Merdian, H.	2009	Independent laboratory validation of the DFG method S19 (BCS method 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in foodstuffs of animal origin, using GC/MS Report No.: P/B 1682 G, Edition Number: M-356331-01-1 PTRL Europe GmbH, Ulm, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 05 **... also filed: KCP 5.1 / 24**	Krebber, R.	2009	Analytical method 01127 for the determination of cyfluthrin and deltamethrin in blood by HPLC-MS/MS Report No.: MR-08/176, Edition Number: M-348630-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 06 **... also filed: KCP 5.1 / 30**	Brumhard, B.	2005	Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS Report No.: C047210, Edition Number: M-247896-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.2 / 07 **... also filed: KCP 5.1 / 31**	Brumhard, B.	2009	Analytical method 00877 for the determination of total residues of Deltamethrin (AE F032640) in/on soil and sediment by HPLC-MS/MS Report No.: 00877, Edition Number: M-246580-02-1 Method Report No.: MR-081/04 Bayer CropScience AG, Monheim, Germany **... amended: 2009-03-31** GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 08	Freitag, T.	2013	Analytical method 01358 for the determination of cis-deltamethrin in soil by HPLC-MS/MS Report No.: MR-13/002, Edition Number: M-451547-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 09	Krebber, R.; Braune, M.	2013	Analytical method 01383 for the determination of deltamethrin in drinking and surface water by HPLC-MS/MS Report No.: MR-13/053, Edition Number: M-464818-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 10	Stanislowski, T.	2013	Independent laboratory validation of BCS analytical method no. 01383 for the determination of deltamethrin in surface water, using LC/MS/MS Report No.: P 3021 G, Edition Number: M-471762-01-1 PTRL Europe GmbH, Ulm, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 11 **... also filed: KCP 5.1 / 27**	Brumhard, B.	2005	Analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS Report No.: C047388, Edition Number: M-248040-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

Data Point	Author(s)	Year	**Title** **Company Report No.** **Source** **GLP or GEP status** **published or not**	**Vertebrate study Y/N**	**Owner**	**Previously used Y/N** If yes, for which data point?
KCP 5.2 / 12 **... also filed:** **KCP 5.1 / 34**	Krebber, R.; Braune, M.	2007	Modification M001 of analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS Report No.: 00886/M001, Edition Number: M-291746-01-1 Method Report No.: MR-07/296 Bayer CropScience AG, Monheim, Germany GLP/GEP: No unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022
KCP 5.2 / 13	Kaussmann, M.	2016	Analytical method 01495 for the determination of various pesticides and selected pesticide metabolites in blood plasma by HPLC-MS/MS Report No.: 01495, Edition Number: M-570324-01-1 Method Report No.: P683166506 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer	**Y** evaluated in the RR for DLT+FPF EC 85 on 02.2022

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.

Deltamethrine

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 4.1.1 / 01	Giudicelli, J. C.	1990	Deltamethrin: Analytical method to verify certified limits. AgrEvo UK Crop Protection Ltd., Chesterford Park, United Kingdom Bayer Report No.: A70743 Edition Number: M-149238-01-1 Date: 1990-06-30 GLP/GEP: No, unpublished confidential	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 4.1.1 / 02	Budgen, P.	2000	Analytical method deltamethrin Determination of AE F108564 and AE 0034609 in technical grade and pure active ingredient by HPLC Deltamethrin technical grade active ingredient Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C010359 Edition Number: M-198876-01-1 Date: 2000-10-18 GLP/GEP: No, unpublished confidential	No	Bayer
KCA 4.1.1 / 03	Hanks, A. R.	1990	Liquide chromatographic method for determination of technical deltamethrin and deltamethrin in pesticide formulations : CIPAC collaborative study. Association of Official Analytical Chemists -public data- Report No.: A70978 Edition Number: M-149460-01-1 Date: 1990-01-01 GLP/GEP: No, unpublished	No	-public data-
KCA 4.1.1 / 04	Martijn, A.; Dobrat, W.	1988	CIPAC - Analysis of technical and formulated pesticides (Deltamethrin). CIPAC; -public data- Report No.: A71086 Edition Number: M-149559-01-1 Date: 1988-01-01 GLP/GEP: No, unpublished	No	-public data-
KCA 4.1.1 / 05	Budgen, P.; Andel, M.	2000	Validation of the analytical method AL026/00-0 for the determination of AE F108564 and AE 0034609 in AE F032640 Deltamethrin technical grade active ingredient Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C010360 Edition Number: M-198878-01-1 Date: 2000-10-18 GLP/GEP: Yes, unpublished confidential	No	Bayer

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KCA 4.1.1 / 06	Budgen, P.; Patzke, D.	2000	Validation of the analytical method AL027/00-0 for the determination of AE F108569 and AE 0437894 in AE F032640 Deltamethrin technical grade active ingredient Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C010358 Edition Number: M-198874-01-1 Date: 2000-10-18 GLP/GEP: Yes, unpublished confidential	No	Bayer
KCA 4.1.1 / 07	Budgen, P.; Guebert, C.	2000	Validation of the analytical method AL025/00-0 for the determination of AE F108565 and AE F108566 in AE F032640 Deltamethrin technical grade active ingredient Code: AE F032640 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C010356 Edition Number: M-198870-01-1 Date: 2000-10-18 GLP/GEP: Yes, unpublished confidential	No	Bayer
KCA 4.1.1 / 08	Feucht, G.; Michel, A.	2003	Analytical method Quantification of AE F032640 (deltamethrin) in formulations (DP, EC, EG, EW, SC, TB, WDG, WP) and technical grade active ingredient by high performance liquid chromatography (HPLC) Bayer Report No.: AL003/99-3 Edition Number: M-232849-01-1 Date: 2003-07-17 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.1 / 09	Feucht, G.	2010	Validation of the analytical method AL003/99-2 for the determination of AE F032640 (active ingredient) in technical AE F032640 - Amendment 1 of report PA02/074 Bayer Report No.: PA02/074 A1 Edition Number: M-231645-02-2 Method Report No.: AL003/99-2 Date: 2003-03-24 **... amended: 2010-03-31** GLP/GEP: No, unpublished confidential	No	Bayer

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KCA 4.1.1 / 10	Anon.	2004	CIPAC Analytical method for Deltamethrin in TC, WG, WP, EC, SC, DP, WT, EG, EW & UL Bayer Report No.: M-274505-01-1 Date: 2004-12-31 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.1 / 11	Doerner-Rieping, S.; Junker, H.	2013	Determination of the enantiomeric purity of AE F032640 in technical grade and pure deltamethrin by high performance liquid chromatography (HPLC) Bayer Report No.: AM038013FP1 Edition Number: M-472746-01-1 Date: 2013-12-04 GLP/GEP: No, unpublished confidential	No	Bayer
KCA 4.1.1 / 12	Cichy, M.; Junker, H.	2010	Validation of the analytical method AM028810FP2 determination of the enantiomeric purity in technical grade and pure deltamethrin (AE F032640) by high performance liquid chromatography (HPLC) Bayer Report No.: PA10/079 Edition Number: M-398212-01-1 Date: 2010-12-21 GLP/GEP: Yes, unpublished confidential	No	Bayer
KCA 4.1.1 / 13	Doerner-Rieping, S.; Junker, H.	2013	Validation of analytical HPLC method AM038013FP1 - Determination of the enantiomeric purity of AE F032640 in technical grade and pure deltamethrin by high performance liquid chromatography (HPLC) - Deltamethrin (AE F032640) Bayer Report No.: PA13/107 Edition Number: M-472718-01-1 Date: 2013-11-26 GLP/GEP: No, unpublished confidential	No	Bayer
KCA 4.1.1 / 14	Doerner-Rieping, S.; Perez-Diaz, C.	2014	Determination of by-products of deltamethrin (AE F032640) in technical grade and pure active substance by high performance liquid chromatography (HPLC) Bayer Report No.: AM034912FP1 Edition Number: M-469685-02-1 Date: 2013-11-15 **... amended: 2014-02-24** GLP/GEP: No, unpublished confidential	No	Bayer

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KCA 4.1.1 / 15	Doerner-Rieping, S.; Perez-Diaz, C.	2014	Amendment no1 to validation of analytical HPLC method AM034912FP1 - Determination of by-products of deltamethrin (AE F032640) in technical grade and pure active substance by high performance liquid chromatography (HPLC) Bayer Report No.: PA13/112 Edition Number: M-469686-02-1 Date: 2013-11-13 **... amended: 2014-02-24** GLP/GEP: Yes, unpublished confidential	No	Bayer
KCA 4.1.1 / 16	Cichy, M.	2002	Determination of 1,2-dichloroethane (AE C504722) and 2-propanol (AE 0171363) in technical deltamethrin (AE F032640) by GC (analytical method) Code: AE F032640 (Deltamethrin) Bayer Report No.: C025631 Edition Number: M-209701-01-1 Date: 2002-08-02 GLP/GEP: No, unpublished confidential	No	Bayer
KCA 4.1.1 / 17	Doerner-Rieping, S.	2016	Amendment no 1 to validation of the analytical method AL025/02-0 for the determination of 1,2-dichloroethane (AE C504722) and 2-propanol (AE 0171363) in technical deltamethrin (AE F032640) by GC Bayer Report No.: PA02/016 Edition Number: M-209703-01-1 Edition Number: M-209703-02-1 Date: 2002-09-10 **... amended: 2016-05-19** GLP/GEP: Yes, unpublished confidential	No	Bayer
KCA 4.1.1 / 18	Doerner-Rieping, S.; Perez-Diaz, C.	2014	Determination of triethylamine (AE 0171388) in technical grade and pure deltamethrin (AE F032640) by gas chromatography (GC) Bayer Report No.: M-474272-01-1 Method Report No.: AM038413FP1 Date: 2014-01-14 GLP/GEP: No, unpublished confidential	No	Bayer

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KCA 4.1.1 / 19	Doerner-Rieping, S.; Perez-Diaz, C.	2014	Validation of analytical method AM038413FP1 determination of triethylamine (AE 0171388) in technical grade and pure deltamethrin (AE F032640) by gas chromatography (GC) Bayer Report No.: PA13/136 Edition Number: M-474279-01-1 Date: 2014-01-14 GLP/GEP: Yes, unpublished confidential	No	Bayer
KCA 4.1.2 / 01	Taylor, N. W.; Snowdon, P. J.	1996	Deltamethrin; analytical grade; active ingredient; Code: Hoe 032640 - Validation of analytical method; peaches; gas chromatography AgrEvo UK Crop Protection Ltd., Chesterford Park, United Kingdom Bayer Report No.: A56355 Report includes Trial Nos.: 203/07/001 Edition Number: M-140178-01-1 Date: 1996-05-03 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 02	Czarnecki, J. J.; McKinney, F. R.; Clayton, F. B.; Crotts, D. G.	1991	Validation of the analytical methodology for determination of combined residues of deltamethrin & trans-deltamethrin in cottonseed & cottonseed processed fractions. EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A71067 Edition Number: M-149543-02-1 Date: 1990-07-05 **... amended: 1991-08-08** GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 03	Mestres, R.; Espinoza, C.; Chevallier, C.; Marti, G.	1979	Decamethrin residues analysis. Journal: Travaux de la Societe de Pharmacie de Montpellier Volume: 39 Issue: 4 Pages: 329;336 Year: 1979 Report No.: A71066 Edition Number: M-152254-01-2 GLP/GEP: n.a., published	No	published

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KCA 4.1.2 / 04	Martens, R.	1998	Analytical method and validation for the determination of residues of endosulfan and deltamethrin by GC Deltamethrin, endosulfan Code: AE F032640 and AE F002671 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C000413 Report includes Trial Nos.: CR97/028 Edition Number: M-180617-02-1 Date: 1998-08-24 **... amended: 1998-11-30** GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 05	Martens, R.	1998	Validation of analytical method DGM F01/97-0 for residues of endosulfan and deltamethrin in cucumber, orange, melon and tomato Deltamethrin, endosulfan Code: AE F032640, AE F002671 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C001152 Edition Number: M-181877-01-1 Date: 1998-11-18 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 06	Martens, R.	2000	Validation of analytical method DGM F01/97-0 for dry crops (grain) Deltamethrin Endosulfan Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C006935 Edition Number: M-194895-01-1 Date: 2000-04-03 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 07	Thier, W. G.	1979	Analytical method for the determination of Hoe 32640 0I (deltamethrin) in biological materials Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A38979 Edition Number: M-251195-01-2 Date: 1979-09-20 GLP/GEP: No, unpublished	No	Bayer

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KCA 4.1.2 / 08	Akhtar, M. H.	1982	Gas chromatographic determination of Deltamethrin in biological samples Journal: Journal of Chromatography Issue: 246 Pages: 81-87 Year: 1982 Report No.: A34911 Edition Number: M-115658-01-1 GLP/GEP: n.a., published	No	published
KCA 4.1.2 / 09	Baldi, B. G.; McKinney, F. R.	1994	Analytical method for the G.C. determination of cis-deltamethrin, trans-deltamethrin and alpha-R-deltamethrin in selected processed grain fractions, grain dusts and whole grain from corn, wheat, sorghum and rice. EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A71069 Edition Number: M-149544-01-1 Date: 1994-01-01 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 10	Supatto, F.	1995	RU 22974 - Assay procedure in oily crops (method and validation) Roussel Uclaf, Romainville, France Bayer Report No.: C016898 Edition Number: M-203267-01-1 Date: 1995-12-14 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 11	Maffezzoni, M.	2001	Analytical method for the determination of deltamethrin in crop ADME Bioanalyses S.A., Vergeze, France Bayer Report No.: C017436 Edition Number: M-204274-01-1 Date: 2001-11-27 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 12	Martens, R.	2000	Data generation and enforcement method for residues on plant material by GC Deltamethrin, Endosulfan Code: AE F032640, AE F002671 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C007949 Edition Number: M-240943-01-1 Date: 2000-03-28 GLP/GEP: No, unpublished	No	Bayer

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KCA 4.1.2 / 13	Martens, R.	2000	Validation of analytical method DGM F01/97-1 for foodstuff of animal origin (milk, eggs, meat, fat, liver, kidney) Deltamethrin Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C009558 Edition Number: M-198798-01-1 Date: 2000-09-06 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 14	Haines, B.; Tauber, R.	2001	Independent Laboratory Validation for the Determination of Residues of Deltamethrin in Lettuce, Oranges, Milk and Fat and Endosulfan in Lettuce and Oranges Using Method DGM F01/97-1 Xenos Laboratories, Inc., Ottawa, ON, Canada Bayer Report No.: B003259 Edition Number: M-238899-01-1 Date: 2001-03-29 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 15	Benwell, L.	1992	Deltamethrin: The validation of the analytical method for the determination of residues in field beans and soil. Hazleton Lab., United Kingdom Bayer Report No.: A49410 Edition Number: M-138460-01-1 Date: 1992-10-01 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 16	Bixler, T. A.	1990	The GLC determination of the combined residues of deltamethrin and trans-deltamethrin in Mexican cherry tomatoes. Hoechst Roussel Agri-Vet Company, Somerville, NJ, USA Bayer Report No.: A71089 Edition Number: M-149562-01-1 Date: 1990-01-05 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 17	Grigor, A.	1991	Analytical method for the determination of deltamethrin, trans-deltamethrin and degradates in soil by gas chromatography. Chemalysis, Inc., USA Bayer Report No.: A48622 Edition Number: M-137727-01-1 Date: 1991-07-26 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 4.1.2 / 18	Mestres, R.; Chevallier, C.; Espinoza, C.	1978	Dosage des residus de decamethrine dans l'eau. University of Montpellier, Faculte de Pharmacie, Montpellier, France Bayer Report No.: A74147 Edition Number: M-152399-01-1 Date: 1978-07-08 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 19	Class, T.	2001	Analytical Method for the Determination of Deltamethrin in Surface Water PTRL Europe GmbH, Ulm, Germany Bayer Report No.: B003535 Report includes Trial Nos.: 01 G 31949 Edition Number: M-240561-01-1 Date: 2001-10-31 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 20	Martens, R.	1999	Enforcement method and validation for water by GC Deltamethrin, endosulfan Code: AE F032640, AE F002671 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer Report No.: C005528 Report includes Trial Nos.: CR99/023 Edition Number: M-192230-01-1 Date: 1999-10-05 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 21	Mestres, R.; Chevallier, C.; Espinoza, C.	1978	Analytical method for decamethrine residue analysis in water. University of Montpellier, Faculte de Pharmacie, Montpellier, France -public data- Report No.: A20239 Edition Number: M-093408-01-1 Date: 1978-07-08 GLP/GEP: No, unpublished	No	-public data-

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KCA 4.1.2 / 22	Idstein, H.; Merz, H. D.; Klug, R.	1993	Bestimmung von Deltamethrin (Hoe 032640) in Luft mittels GC. Hoechst AG, Frankfurt am Main, Germany Bayer Report No.: A50771 Edition Number: M-131778-01-1 Date: 1993-06-02 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 23	Class, T.	1994	Determination of deltamethrin (Hoe 032640) in air by GC validation of the analytical method No.: AL005/93-0 provided by the sponsor. PTRL Europe GmbH, Ulm, Germany Bayer Report No.: A52594 Edition Number: M-133404-01-1 Date: 1994-03-31 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 24	Class, T.	1994	Validation of an analytical method for the determination of deltamethrin in air (Method and validation) PTRL Europe GmbH, Ulm, Germany Bayer Report No.: C012850 Report includes Trial Nos.: P138G Edition Number: M-203491-01-1 Date: 1994-03-01 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 25	Class, T.	2001	Validation of an Analytical Method for the Determinatin of Deltamethrin in Air PTRL Europe GmbH, Ulm, Germany Bayer Report No.: B003367 Report includes Trial Nos.: 01 G 31951 P 482 G Edition Number: M-240404-01-1 Date: 2001-06-29 GLP/GEP: No, unpublished	No	Bayer

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KCA 4.1.2 / 26	Huff, D. K.; McKinney, F. R.	1994	Method development and validation for the determination of deltamethrin (alpha-R-, cis- and trans-) and tralomethrin in dairy cow tissues (with poultry matrices added by amendment). EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer Report No.: A71076 Edition Number: M-149550-01-1 Date: 1994-07-29 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 27	Brumhard, B.	2005	Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS Bayer Report No.: C047210 Report includes Trial Nos.: 00877 Edition Number: M-247896-01-1 Date: 2005-03-04 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 28	Brumhard, B.	2010	Analytical method 00877 for the determination of total residues of Deltamethrin (AE F032640) in/on soil and sediment by HPLC-MS/MS Bayer Report No.: 00877 Edition Number: M-246580-02-2 Method Report No.: MR-081/04 Date: 2005-03-04 **... amended: 2009-03-31** GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 29	Brumhard, B.	2005	Analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS Bayer Report No.: C047388 Report includes Trial Nos.: 00886 Edition Number: M-248040-01-1 Date: 2005-03-04 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 4.1.2 / 30	Krebber, R.; Braune, M.	2007	Modification M001 of analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS Bayer Report No.: 00886/M001 Edition Number: M-291746-01-1 Method Report No.: MR-07/296 Date: 2007-08-21 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 31	Krebber, R.	2009	Analytical method 01127 for the determination of cyfluthrin and deltamethrin in blood by HPLC-MS/MS Bayer Report No.: MR-08/176 Edition Number: M-348630-01-1 Date: 2009-06-03 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 32	Diot, R.	2004	Modification M001 to the analytical method 00855 for the determination of residues of deltamethrin (AE F032640) in/on apple, kiwi and plum by GC/MSD Bayer Report No.: C040164 Report includes Trial Nos.: 04-03 Edition Number: M-228400-01-1 Date: 2004-05-25 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 33	Diot, R.	2004	Supplement E001 to the analytical method 00855/M001 for the determination of residues of deltamethrin (AE F032640) in/on pear, cherry, tomato, peach, and processed fractions of apple, pear and peach by GC/MSD Bayer Report No.: C042013 Report includes Trial Nos.: 04-05 Edition Number: M-231816-01-1 Date: 2004-05-28 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 4.1.2 / 34	Diot, R.	2004	Supplement E002 to the analytical method 00855/M001 for the determination of residues of deltamethrin (AE F032640) in/on melon, zucchini, artichoke, pepper, sugar beet, field pea and lettuce by GC/MSD Bayer Report No.: C043675 Report includes Trial Nos.: 00855/M001/E002 04-08 Edition Number: M-234987-01-1 Date: 2004-09-07 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 35	Zimmer, D.; Philipowski, C.	2004	Residue analytical method 00855/M002 for the determination of residues of cis-deltamethrin (AE F032640) in/on pepper (fruit), zucchini (fruit), tomato (fruit), olive (fruit), melon (fruit, pulp), sugarbeet (body, leaf with root collar), cob Bayer Report No.: 00855/M002 Report includes Trial Nos.: P602045501 Edition Number: M-236022-01-1 Date: 2004-10-05 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 37	Lakaschus, S.; Winter; O.	2009	Validation of BCS Method 00855/M004 for the Determination of cis-deltamethrin, trans-deltamethrin and alpha-R-deltamethrin in foodstuff of plant origin Eurofins Analytik GmbH, Dr. Specht Laboratorien, Hamburg, Germany Bayer Report No.: 00855/M004 Report includes Trial Nos.: EASSM/S09-02191 Edition Number: M-356934-01-1 Method Report No.: BAY-0904V Date: 2009-09-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 38	xxx	2000	Acute toxicity to Oncorhynchus mykiss (rainbow trout) in a static-renewal system Deltamethrin oil in water emulsion 15 g/L Code: AE F032640 00 EW01 B103 xxx Bayer Report No.: C008365 Edition Number: M-197428-01-1 Date: 2000-07-18 GLP/GEP: Yes, unpublished	Yes	Bayer

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KCA 4.1.2 / 39	Sowig, P.; Gosch, H.; Weller, O.	2000	Acute toxicity to Daphnia magna (waterflea) Deltamethrin oil in water emulsion 15 g/L Code: AE F032640 00 EW01 B103 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C008334 Edition Number: M-197398-01-1 Date: 2000-07-05 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 40	Sowig, P.; Gosch, H.; Weller, O.	2000	Algal growth inhibition - Pseudokirchneriella subcapitata Deltamethrin oil in water emulsion 15 g/L Code: AE F032640 00 EW01 B103 Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer Report No.: C008323 Edition Number: M-197387-01-1 Date: 2000-07-05 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 41	Braune, M.	2011	Method 01307 for the determination of deltamethrin, á-R-isomer of deltamethrin and trans-isomer of deltamethrin in test water from aquatic toxicity tests by HPLC-MS/MS Bayer Report No.: 01307 Edition Number: M-410093-01-1 Method Report No.: MR-10/162 Date: 2011-06-15 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 42	xxx	2005	Fate and effects of Thiacloprid & Deltamethrin OD 100 + 10 in outdoor mesocosm ponds xxx Report No.: BAY-018/4-52 Edition Number: M-259938-01-2 Date: 2005-11-03 GLP/GEP: Yes, unpublished	Yes	Bayer
KCA 4.1.2 / 43	Krebber, R.; Braune, M.	2007	Analysis of deltamethrin concentrations in water samples of ECT study no. P1MA Bayer Report No.: MR-07/295 Edition Number: M-291848-01-1 Date: 2007-08-24 GLP/GEP: Yes, unpublished	No	Bayer

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KCA 4.1.2 / 44	Schoening, R.; Willlmes, J.	2013	Residue analytical method 01347 for the determination of residues of deltamethrin by HPLC with electrospray and MS/MS - detection Bayer Report No.: MR-012/067 Edition Number: M-444791-01-1 Date: 2013-01-14 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 45	xxx	1990	(LX 165-08, deltamethrin technical) - Acute (28-Day) toxicity to rainbow trout (Oncorhynchus mykiss) under flow-through conditions. xxx Report No.: A47111 Edition Number: M-135553-01-1 Date: 1990-04-11 GLP/GEP: Yes, unpublished **... also filed:** **KCA 8.2.1 / 03** **KCA 8.2.2.1 / 01**	Yes	Bayer
KCA 4.1.2 / 46	Freitag, T.; Koch, V.	2011	Analytical method 01306 for the determination of deltamethrin and the metabolites a-R-deltamethrin and trans-deltamethrin in sediment by HPLC-MS/MS Bayer Report No.: MR-10/154 Edition Number: M-418179-01-1 Date: 2011-11-18 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 47	Braune, M.	2013	Method 01369 for the determination of BCS-BY84407 in test water by HPLC-MS/MS Bayer Report No.: MR-13/038 Edition Number: M-451312-01-1 Date: 2013-04-12 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 48	Braune, M.	2013	Analytical method 01371 for the determination of BCS-CW57835 in test water from aquatic toxicity tests by HPLC-UV Bayer Report No.: MR-13/043 Edition Number: M-451531-01-1 Date: 2013-04-17 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 4.1.2 / 49	Schoening, R.; Willmes, J.	2013	Determination of deltamethrin in feeding solutions from a 10d continuous honeybee feeding study (Study Number: S13-00151; Eurofins) Bayer Report No.: MR-13/135 Edition Number: M-469484-01-1 Date: 2013-11-13 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 50	Dix, M. E.	2013	Method validation for seven pyrethroids in formulated sediment by gas chromatography using mass selective detection with negative chemical ionization Smithers Viscient, Wareham, MA, USA Pyrethroid Working Group Report No.: 13656.6124 Edition Number: M-554274-01-1 Date: 2013-10-03 GLP/GEP: No, unpublished	No	Pyrethroid Working Group
KCA 4.1.2 / 51	Dix, M. E.	2015	Method validation for seven pyrethroids in freshwater by gas chromatography using selective detection with negative chemical ionization Smithers Viscient, Wareham, MA, USA Pyrethroid Working Group Report No.: 13656.6125 Edition Number: M-536985-01-1 Date: 2015-10-03 GLP/GEP: No, unpublished	No	Pyrethroid Working Group
KCA 4.1.2 / 52	Dix, M. E.	2013	Method extension for eight pyrethroids in freshwater by gas chromatography using mass selective detection with negative chemical ionization and liquid chromatography with mass spectrometry Smithers Viscient, Wareham, MA, USA Pyrethroid Working Group Report No.: 13656.6174 Edition Number: M-554282-01-1 Date: 2013-10-03 GLP/GEP: No, unpublished	No	Pyrethroid Working Group
KCA 4.1.2 / 53	Brumhard, B.; Loehrwald, K.H.	2007	Analysis of deltamethrin concentrations in sediment samples of ECT study no. P1MA Bayer Report No.: MR-07/297 Edition Number: M-291818-01-1 Date: 2007-08-22 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 4.1.2 / 54	Schoening, R.; Diehl, P.	2013	Analytical phase report - Assessment of side effects on the honeybee (Apis mellifera L.), exposed to Phacelia tanacetifolia, sprayed sequentially with deltamethrin during flowering in a long-term field study in North Alsace, France Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Bayer Report No.: S10-03820 Edition Number: M-451145-01-1 Date: 2013-04-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 55	Schoening, R.; Diehl, P.	2013	Analytical phase report - Assessment of side effects on the honeybee (Apis mellifera L.), exposed to Phacelia tanacetifolia, sprayed sequentially with deltamethrin during flowering in a long-term field study in Mid Alsace, France Bayer Report No.: S10-03824 Edition Number: M-451154-01-1 Date: 2013-04-15 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.1.2 / 56	Desmaris, F.; Diot, R.; Mousques, A.	2017	Deltamethrin - Questions by the RMS CRD on the analytical methods/validation relating to chemical active in the frame of AIR process Bayer Report No.: M-588243-01-1 Date: 2017-05-20 GLP/GEP: n.a., unpublished **... also filed:** **KCA 4.2 / 17**	No	Bayer
KCA 4.1.2 / 36	Diot, R.	2004	Modification M003 to the analytical method 00855 for the determination of residues of deltamethrin (AE F032640) in/on olive and rape by GC/MSD and in/on Brussels sprout, rape and sugar beet by GC-MS/MS Bayer Report No.: C043677 Report includes Trial Nos.: 00855/M003 04-10 Edition Number: M-234990-01-1 Date: 2004-09-07 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 4.1.2 / 57	Desmaris, F.	2018	Additional chromatograms of study reports: RA-2409/03 (sugar beet) - RA-2410/03 (sugar beet) - RA-2436/03 (sugar beet) - 08-2215 (barley post-harvest) - 08-2214 (wheat post-harvest) Bayer Report No.: M-641700-01-1 Date: 2018-11-09 GLP/GEP: No, unpublished	No	Bayer
KCA 4.1.2 / 58	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 6.1 / 06**	No	Bayer
KCA 4.1.2 / 59	Sadler, T.	2019	Validation data for method used in study report 2010/0064/01 Bayer Report No.: M-646878-01-1 Date: 2019-01-18 GLP/GEP: n.a., unpublished **... also filed:** **KCA 8.2.4.1 / 11**	No	Bayer
KCA 4.2 / 01	Tillier, C.; Devaux, P.	1981	Quantitative determination of deltamethrin in urine. Roussel Uclaf, Romainville, France Bayer Report No.: A71071 Edition Number: M-149546-01-1 Date: 1981-09-24 GLP/GEP: No, unpublished	No	Bayer
KCA 4.2 / 02	Tillier, C.	1989	RU 22974: Assay procedure in plasma. Roussel Uclaf, Romainville, France Bayer Report No.: A70887 Edition Number: M-149371-01-1 Date: 1989-12-21 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 4.2 / 03	Tillier, C.	1988	Assay procedure for the analysis of deltamethrin residues in human plasma. Roussel Uclaf, Romainville, France Bayer Report No.: A71065 Edition Number: M-149542-01-1 Date: 1988-05-20 GLP/GEP: No, unpublished	No	Bayer
KCA 4.2 / 04	Weber, H.	2009	Validation of enforcement method DFG S19 (L 00.00-34) (BCS method ID 00086/M089) for the determination of cis-deltamethrin (AE F032640) in/on foodstuff of plant origin Eurofins Analytik GmbH, Dr. Specht Laboratorien, Hamburg, Germany Bayer Report No.: S09-00553 Edition Number: M-351076-01-1 Date: 2009-07-07 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.2 / 05	Merdian, H.	2009	Independent laboratory validation of the DFG method S19 (BCS method 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in plant materials, using GC/MS PTRL Europe GmbH, Ulm, Germany Bayer Report No.: P/B 1681 G Edition Number: M-356306-01-1 Date: 2009-09-23 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.2 / 06	Weber, H.	2009	Validation of enforcement method DFG S19 (L 00.00-34) (BCS method ID 00086/M090) for the determination of residues cis-deltamethrin (AE F032640) in/on foodstuff of animal origin Eurofins Analytik GmbH, Dr. Specht Laboratorien, Hamburg, Germany Bayer Report No.: S09-00551 Edition Number: M-351080-01-1 Date: 2009-07-07 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.2 / 07	Merdian, H.	2009	Independent laboratory validation of the DFG method S19 (BCS method 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in foodstuffs of animal origin, using GC/MS PTRL Europe GmbH, Ulm, Germany Bayer Report No.: P/B 1682 G Edition Number: M-356331-01-1 Date: 2009-09-23 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 4.2 / 08	Justus, K.	2014	Extraction efficiency testing of the residue analytical method 00855/M002 for the determination of residues of cis-deltamethrin (AE F032640) in crops using aged radioactive residues - Surrogate - Bayer Report No.: EnSa-14-0317 Edition Number: M-481954-02-1 Date: 2014-04-01 **... amended: 2014-04-02** GLP/GEP: No, unpublished	No	Bayer
KCA 4.2 / 09	Schoening, R.; Willmes, J.	2014	Cross validation of extraction methods for the determination of residues of deltamethrin in plant materials by HPLC-MS/MS Bayer Report No.: MR-14/012 Edition Number: M-481952-02-1 Date: 2014-04-01 **... amended: 2014-04-11** GLP/GEP: No, unpublished	No	Bayer
KCA 4.2 / 10	Freitag, T.	2013	Analytical method 01358 for the determination of cis-deltamethrin in soil by HPLC-MS/MS Bayer Report No.: MR-13/002 Edition Number: M-451547-01-1 Date: 2013-04-17 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.2 / 11	Krebber, R.; Braune, M.	2013	Analytical method 01383 for the determination of deltamethrin in drinking and surface water by HPLC-MS/MS Bayer Report No.: MR-13/053 Edition Number: M-464818-01-1 Date: 2013-09-02 GLP/GEP: Yes, unpublished	No	Bayer
KCA 4.2 / 12	Stanislowski, T.	2013	Independent laboratory validation of BCS analytical method no. 01383 for the determination of deltamethrin in surface water, using LC/MS/MS PTRL Europe GmbH, Ulm, Germany Bayer Report No.: P 3021 G Edition Number: M-471762-01-1 Date: 2013-11-18 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 4.2 / 13	Schoening, R.; Snowdon, P.	2015	Review of analytical methods for the determination of deltamethrin residues in products of plant and animal origin Bayer Report No.: M-537967-01-1 Date: 2015-11-03 GLP/GEP: n.a., unpublished	No	Bayer
KCA 4.2 / 14	Specht, W.; Thier, H. P.	1987	Organochlorine, organophosphorus, nitrogen-containing and other pesticides - Gas-chromatographic determination after cleanup by gel permeation chromatography and silica gel minicolumn chromatography Publisher: Deutsche Forschungsgemeinschaft / VCH Location: Weinheim Journal: Manual of Pesticide Residue Analysis Volume: I Pages: 383 - 400 Year: 1987 Report No.: 00086 Edition Number: M-006227-01-1 GLP/GEP: No, published	No	published
KCA 4.2 / 15	Anon.	1996	Analytical methods for pesticide residues in foodstuffs Multi-residue method 1 Pesticides amenable to gas chromatography Journal: Ministery of Public Health, Welfare and Sport, NLD Volume: June Issue: Part I Pages: 1;22 Year: 1996 Report No.: C048287 Edition Number: M-249648-01-1 Date: 1996-01-01 GLP/GEP: No, published	No	published
KCA 4.2 / 16	Radix, P.	2016	Deltamethrin - Answer to CRD question Volume 3CA Part B 5: Analytical methods/validation relating to the active Bayer Report No.: M-555924-01-1 Date: 2016-06-01 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 4.2 / 17	Desmaris, F.; Diot, R.; Mousques, A.	2017	Deltamethrin - Questions by the RMS CRD on the analytical methods/validation relating to chemical active in the frame of AIR process Bayer Report No.: M-588243-01-1 Date: 2017-05-20 GLP/GEP: n.a., unpublished **... also filed:** **KCA 4.1.2 / 56**	No	Bayer
KCA 4.2 / 18	Mousquès, A., Sadler, T.	2019	Method validation data in support of the determination of deltamethrin in other crop fractions not assigned to a specific group Bayer Report No.: M-647563-01-1 Date: 2019-01-25 GLP/GEP: n.a., unpublished	No	Bayer
KCA 4.2 / 19	Mousquès, A., Sadler, T.	2019	Method validation data in support of the determination of deltamethrin in high starch commodities Bayer Report No.: M-647564-01-1 Date: 2019-01-25 GLP/GEP: n.a., unpublished	No	Bayer
KCA 4.2 / 20	Mousquès, A., Sadler, T.	2019	Method validation data in support of the determination of deltamethrin in high oil plant commodities Bayer Report No.: M-647565-01-1 Date: 2019-01-25 GLP/GEP: n.a., unpublished	No	Bayer
KCA 4.2 / 21	Sadler, T.; Mousquès, A.	2019	Method validation data in support of the determination of deltamethrin high acid content plant commodities Bayer Report No.: M-647566-01-1 Date: 2019-01-25 GLP/GEP: n.a., unpublished	No	Bayer
KCA 4.2 / 22	Mousquès, A.; Sadler, T.	2019	Method validation data in support of the determination of deltamethrin in high water commodities Bayer Report No.: M-647640-01-1 Date: 2019-01-25 GLP/GEP: n.a., unpublished	No	Bayer

Flupyradifurone

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 4.2.1 /02	Wagner, S.	2011	Validation of AM008809MP1 - Flupyradifurone (BYI 02960) - Determination of technical grade active substance HPLC - ISTD Bayer CropScience, Report No.: VB1-AM008809MP1, Edition Number: M-409002-01-1 Date: 2011-06-06 GLP/GEP: no, unpublished	N	Bayer
KIIA 4.3 /01	Schulte, G.; Bauer, J.	2012	Analytical method 01330 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrix by HPLC-MS/MS - Enforcement method plant Bayer CropScience, Report No.: 01330, Edition Number: M-425848-01-1 Method Report No.: 01330 Date: 2012-02-22 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.3 /02	Konrad, S.	2012	Independent lab validation of BCS method 01330 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrices by HPLC-MS/MS Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 2011/0134/01, Edition Number: M-427133-01-1 Method Report No.: 2011/0134/01 EPA MRID No.: 48843818 Date: 2012-02-28 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.3 /03	Li, Y.; Schoening, R.	2011	Amendment No. 1 - Validation of Bayer CropScience method RV-001-P10-02 - An analytical method for the determination of residues of BYI 02960, 6-chloronicotinic acid, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP013, Edition Number: M-415504-02-1 Method Report No US: RARVP013 Date: 2011-10-12 ...Amended: 2012-01-11 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 4.3 /04	Justus, K.	2011	Extraction efficiency testing of the residue analytical method RV-001-P10-02 for the determination of BYI 02960, 6-chloronicotinic acid, difluoroacetic acid and difluoroethyl-amino-furanone in plant matrices using aged radioactive residues Bayer CropScience, Report No.: MEF-11/793, Edition Number: M-419323-01-1 EPA MRID No.: 48843821 Date: 2011-12-01 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.3 /05	Rosati, D.	2012	Analytical method no 01212 for the determination of residues of BYI 02960 and its metabolites BCS-AA56716 (DFA), AE F161089 (6CNA) and BCS-CC98193 (furanone) in/on plant material by HPLC-MS/MS Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 01212, Edition Number: M-428017-01-1 Method Report No.: MR-10/174 Date: 2012-03-27 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.3 /06	Schulte, G.; Bauer, J.	2012	Analytical method 01214 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on animal matrices by HPLC-MS/MS - Enforcement method animal Bayer CropScience, Report No.: 01214, Edition Number: M-425837-01-1 Method Report No.: 01214 EPA MRID No.: 48843825 Date: 2012-02-22 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.3 /07	Konrad, S.	2012	Independent lab validation of BCS method 01214 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on animal matrices by HPLC-MS/MS Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 2011/0164/01, Edition Number: M-427160-01-1 Method Report No.: 2011/0164/01 EPA MRID No.: 48843826 Date: 2012-02-28 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 4.3 /08	xxx	2012	BYI 02960 - Magnitude of the residue in dairy cows - Amended report xxx 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 6.1.1 /02 ...also filed: KIIA 6.4.2 /01	Y	Bayer
KIIA 4.3 /09	xxx	2012	BYI 02960 - Magnitude of the residue in laying hens xxx Report No.: RARVP041, Edition Number: M-428933-01-1 Date: 2012-04-05 GLP/GEP: yes, unpublished ...also filed: KIIA 6.4.1 /01	Y	Bayer
KIIA 4.3 /10	Schulte, G.; Teubner, L.	2012	Modification M001 of the analytical method 01330 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrix by HPLC-MS/MS- Enforcement method plant Bayer CropScience, Report No.: MR-12/054, Edition Number: M-438310-01-1 Date: 2012-09-10 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.3 /11	Konrad, S.	2012	Independent lab validation of BCS method 01330/M001 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrices by HPLC-MS/MS Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 01330/M001, Edition Number: M-439855-01-1 Date: 2012-10-15 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.4 /01	Brumhard, B.: Reineke, A.	2009	Analytical method 01074 for the determination of BYI 02960 in soil using LC/MS/MS Bayer CropScience, Report No.: 01074, Edition Number: M-337752-01-1 Method Report No.: MR-07/337 Date: 2009-02-24 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 4.5 /01	Fargeix, G.; Rosati, D.	2012	Analytical method no 01213 for the determination of residues of BYI 02960 in drinking and surface water by HPLC-MS/MS Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 01213, Edition Number: M-428019-01-1 Method Report No.: MR-12/022 Date: 2012-03-29 GLP/GEP: yes, unpublished	N	Bayer
KIIA 4.7 /01	Heinz, N.	2011	BYI 02960: Analytical method for determination in air PTRL Europe GmbH, Ulm, Germany Bayer CropScience, Report No.: P 2419 G, Edition Number: M-420657-01-1 EPA MRID No.: 48843838 Date: 2011-12-14 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
KCP 5.1 / 04 **... also filed: KCA 6.3.1.1 / 02**	Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.	2016	Determination of the residues of BYI 02960 and deltamethrin in/on grape after high or low-volume spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy Report No.: 14-2095, Edition Number: M-560047-01-1 Bayer CropScience AG, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 06 **... also filed: KCA 6.3.2.1 / 02**	Kaussmann, M.; Kowalski, N.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in Italy, southern France, Spain and Greece Report No.: 16-2194, Edition Number: M-634135-01-1 Bayer AG, Crop Science Division, Monheim, Germany 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
KCP 5.1 / 08 **... also filed: KCA 6.3.2.1 / 03**	Kaussmann, M.; Kowalski, N.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy Report No.: 16-2195, Edition Number: M-629954-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 09 **... also filed: KCA 6.3.3.1 / 03**	Noss, G.	2017	Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in France (South), Italy, Spain and Greece Report No.: 15-2130, Edition Number: M-572779-03-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-10-17** GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 11 **... also filed: KCA 6.3.3.1 / 04**	Kaussmann, M.; Miara, C.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy, Spain and Greece Report No.: 16-2034, Edition Number: M-634112-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 13 **... also filed: KCA 6.3.4.1 / 03**	Schulte, G.	2017	Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in Italy, Spain and Portugal Report No.: 15-2127, Edition Number: M-580063-03-1 Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-09-22** GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 15 **... also filed: KCA 6.3.4.1 / 04**	Kaussmann, M.; Kerkering, S.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy and Spain Report No.: 16-2032, Edition Number: M-633925-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 17	Schulte, G.	2017	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, France (South) and Italy Report No.: 15-2133, Edition Number: M-574144-02-1	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
			Bayer AG, Crop Science Division, Monheim, Germany **... amended: 2017-05-03** GLP/GEP: Yes unpublished		
KCP 5.1 / 19	Schulte, G.; Kuester, S.; Kerkering, S.	2018	Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, southern France and Italy Report No.: 16-2100, Edition Number: M-621728-01-1 Bayer AG, Crop Science Division, Monheim, Germany GLP/GEP: Yes unpublished	No	Bayer
KCP 5.1 / 47	Meilland-Berthier, I.	2014	Determination of the residues of BYI 02960 in/on grape after high and low-volume spray application of BYI 02960 SL 200 in southern France, Spain, Italy and Greece Report No.: 12-2126, Edition Number: M-479360-01-1 Bayer S.A.S., Bayer CropScience, Lyon, France GLP/GEP: Yes unpublished	No	Bayer

List of data relied on 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 submitted analytical methods

A 2.1 Analytical methods for Deltamethrin

A 2.1.1 Methods used for the generation of pre-authorization data (KCP 5.1)

A 2.1.1.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.1)

A 2.1.1.1.1 Analytical method 1 (00855/M004)

A 2.1.1.1.1.1 Method validation

Comments of zRMS:	The study of Lakaschus, S.; Winter; O. (2009) has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The method 00855/M004 was successfully validated for the determination of residues of cis-deltamethrin, trans-deltamethrin and α-R-deltamethrin in foodstuff of plant origin (strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed)).* *The limit of quantification (LOQ) for cis-, trans- and α-(R)-deltamethrin was 0.01 mg/kg for strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain) and oilseed rape (seed) and 0.05 mg/kg for wheat (straw) and barley (whole plant).* *For all matrices mean recovery values obtained by LC-MS/MS for trans-, cis- and α-(R)-deltamethrin for both fortification levels (LOQ and the ten times LOQ level) comply with the standard acceptance criteria of SANCO/825/00, which requires that the mean recovery at each fortification level should be in the range of 70-110%). Furthermore, as required by the standard acceptance criteria, the overall relative standard deviation as well as the relative standard deviation for each fortification level were <20%.* *Two MRM transitions were monitored for each analyte. Therefore, the LC-MS/MS method is highly specific and an additional confirmatory method is not necessary.* *This method meets all guideline criteria according to SANCO/3029/99 rev. 4 and SANCO/825/00 rev. 8.1.* *The sudy is acceptable.*

Reference:	**KCP 5.1/01**
Title:	Validation of BCS Method 00855/M004 for the Determination of cis-deltamethrin, trans-deltamethrin and alpha-R-deltamethrin in foodstuff of plant origin
Report:	Lakaschus, S.; Winter; O.; 2009; 00855/M004; M-356934-01-1
Authority registration No:	
Guideline(s):	91/414/EEC, 96/46/EC 4.2.1 SANCO/3029/99 rev. 4 of 1/07/2000 SANCO7(825/00 rev.7 of 17/03/2004 OECD (ENV7JM7MONO (2007)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

In order to validate the method for the determination of trans-, cis- and α-(R)-deltamethrin in foodstuff of plant origin a series of recovery experiments was performed by fortifying control (untreated) specimens of strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed).

The BCS Method 00855/M002 for the determination of cis-deltamethrin was used as a basis and extended for the analysis of trans-deltamethrin and α-(R)-deltamethrin. The extended method is referred to as BCS

Method 00855/M004. The conventional extraction of olive from the precursor method 00855/M002 was used for oilseed rape (seed). Additionally ^{13}C-labelled isomers of deltamethrin were used as internal standards for each corresponding unlabelled deltamethrin isomer. In cases where matrix effects were nearly identical the internal standards of cis-^{13}C-deltamethrin and trans-^{13}C-deltamethrin were used for the determination of α-(R)-deltamethrin since these standards had a higher purity than α-(R)-^{13}C-deltamethrin.
The specimen weights taken for analysis were 10 g for strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain) and 5 g for wheat (straw), barley (whole plant) and oilseed rape (seed).
Extraction of residues was achieved by homogenisation with acetone/dichlormethan/n-hexane (1/1/1, v/v/v). Depending on the nature of the sample material, the obtained raw extracts were diluted and directly analyzed by LC-MS/MS without further clean up, or cleaned up by solid phase extraction on a SCX-cartridge, or by gel permeation chromatography (GPC), respectively. The HPLC column was a C18 Ascentis Express. The final extracts were analysed for trans-, cis- and α-(R)-deltamethrin using a Hewlett-Packard Series 1200 HPLC (Agilent Technologies) coupled to a PE-Sciex API 4000 tandem mass spectrometer with IonSpray® interface. Two MRM transitions were monitored for all the isomers m/z 523 → 281 as 1st MRM (quantification) and m/z 525 → 283 as 2nd MRM (confirmation).
For all eight matrices, the control specimens were analysed in duplicate, and fortified specimens were analysed in quintuplicate for each fortification level, limits of quantification (LOQ) and at ten times LOQ. Two characteristic mass transitions are monitored for each deltamethrin isomer.

Results and discussions
In order to ensure unambiguous identification two mass transitions were monitored and evaluated for all isomers (MRM 523→281 and MRM 525→283). No significant interferences from the specimen matrices were detected at the retention time corresponding to the deltamethrin isomers in any of the control specimens.
Analytical grade reference materials of trans-deltamethrin (94.0 %), cis-deltamethrin (99.6 %) and α-(R) - deltamethrin (94.3 %) were used for preparing all required solutions. trans-13C-deltamethrin (99.0 %), cis-13C-deltamethrin (>99 %) and α-(R)-13C-deltamethrin (91 %) were used as internal standards.
The tests for strawberry (fruit), tomato (fruit), green peas (seed) and wheat straw showed matrix effects for α-(R)-deltamethrin analogue to cis-deltamethrin as well as to trans-deltamethrin in oilseed rape seed.
The purity of α-(R)-^{13}C-deltamethrin was less than trans-^{13}C- and cis-^{13}C-deltamethrin. Therefore residues of α-(R)-deltamethrin were determined using cis-13C-deltamethrin for strawberry (fruit), tomato (fruit), green peas (seed) and wheat (straw) and trans-13C-deltamethrin for oilseed rape (seed) as the internal standards.
The ratio of the analyte peak area to the internal standard peak area was determined in each sample. During method calibration a curve with variable analyte concentrations of 1.25 to 100 ng/mL and a constant amount of the internal standard (100 ng/mL) was recorded and used for the calculation of an average response factor. The detector showed linear response for solvent standards of all three deltamethrin isomers ranging from 1.25 ng/mL to 100 ng/mL. The correlation coefficients were found to be 0.9995 and 0.9990 for trans-deltamethrin, 0.9996 and 0.9992 for cis-deltamethrin and ranged between 0.9987 to 0.9999 for α-(R) - deltamethrin.
For all matrices, analysis of control specimens by LC-MS/MS indicated that residues of the test substance were below 30% of the LOQ.
The limit of quantification (LOQ) for cis-, trans- and α-(R)-deltamethrin was 0.01 mg/kg for edible matrices like strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain) and oilseed rape (seed) and the limit of detection (LOD) was 0.003 mg/kg. For not edible matrices like wheat (straw) and barley (whole plant) the limit of quantification (LOQ) was 0.05 mg/kg and the limit of detection (LOD) was 0.01 mg/kg.
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).
The accuracy (analytical recovery) of the method was determined by comparing the measured and nominal concentrations from the recovery experiments.
For all eight matrices, mean recovery values obtained by LC-MS/MS for both fortification levels (LOQ and the ten times LOQ level) for trans-, cis- and α-(R)-deltamethrin, comply with the standard acceptance criteria of SANCO Guideline 825/00, which requires that the mean recovery at each fortification level

should be in the range of 70 - 110 %. Furthermore, the overall relative standard deviation and the relative standard deviation for each fortification level was ≤ 20 %.

Table A 1: Recovery results from method validation of cis-deltamethrin and isomers using the analytical method in different plant matrices

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin					
strawberry (fruit)	0.01	5	103	1.3	m/z 523→281 (quantification)
	0.10	5	103	0.8	
	0.01	5	103	2.5	m/z 525→283 (confirmation)
	0.10	5	104	0.8	
tomato (fruit)	0.01	5	100	1.5	m/z 523→281 (quantification)
	0.10	5	102	3.3	
	0.01	5	100	1.6	m/z 525→283 (confirmation)
	0.10	5	102	2.2	
lambs lettuce	0.01	5	99	2.3	m/z 523→281 (quantification)
	0.10	5	104	3.0	
	0.01	5	100	4.6	m/z 525→283 (confirmation)
	0.10	5	102	3.7	
green peas	0.01	5	89	2.1	m/z 523→281 (quantification)
	0.10	5	91	2.6	
	0.01	5	91	1.8	m/z 525→283 (confirmation)
	0.10	5	90	4.3	
wheat (straw)	0.05	5	80	5.3	m/z 523→281 (quantification)
	0.50	5	73	6.3	
	0.05	5	81	3.5	m/z 525→283 (confirmation)
	0.50	5	72	5.0	
wheat (grain)	0.01	5	100	3.3	m/z 523→281 (quantification)
	0.10	5	97	2.7	
	0.01	5	100	4.4	m/z 525→283 (confirmation)
	0.10	5	95	3.1	
barley (whole plant)	0.05	5	96	2.3	m/z 523→281 (quantification)
	0.50	5	96	1.1	
	0.05	5	96	1.9	m/z 525→283 (confirmation)
	0.50	5	96	2.1	
oilseed rape (seeds)	0.01	5	98	1.9	m/z 523→281 (quantification)
	0.10	5	101	2.9	
	0.01	5	98	3.0	m/z 525→283 (confirmation)
	0.10	5	98	1.5	

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
α-(R)-deltamethrin					
strawberry (fruit)	0.01	5	84	7.3	m/z 523→281 (quantification)
	0.10	5	94	5.2	
	0.01	5	83	5.2	m/z 525→283 (confirmation)
	0.10	5	93	2.9	
tomato (fruit)	0.01	5	91	3.2	m/z 523→281 (quantification)
	0.10	5	100	2.5	
	0.01	5	91	2.7	m/z 525→283 (confirmation)
	0.10	5	99	2.3	
lambs lettuce	0.01	5	98	6.0	m/z 523→281 (quantification)
	0.10	5	90	15	
	0.01	5	97	5.6	m/z 525→283 (confirmation)
	0.10	5	88	16	
green peas	0.01	5	79	8.7	m/z 523→281 (quantification)
	0.10	5	88	1.7	
	0.01	5	79	6.7	m/z 525→283 (confirmation)
	0.10	5	87	2.3	
wheat (straw)	0.05	5	80	5.4	m/z 523→281 (quantification)
	0.50	5	71	6.9	
	0.05	5	80	6.5	m/z 525→283 (confirmation)
	0.50	5	70	6.7	
wheat (grain)	0.01	5	91	3.2	m/z 523→281 (quantification)
	0.10	5	94	0.9	
	0.01	5	93	4.0	m/z 525→283 (confirmation)
	0.10	5	94	1.6	
barley (whole plant)	0.05	5	90	2.8	m/z 523→281 (quantification)
	0.50	5	88	3.4	
	0.05	5	91	3.5	m/z 525→283 (confirmation)
	0.50	5	90	2.6	
oilseed rape (seeds)	0.01	5	80	11	m/z 523→281 (quantification)
	0.10	5	87	2.0	
	0.01	5	83	6.7	m/z 525→283 (confirmation)
	0.10	5	86	2.0	

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
trans-deltamethrin					
strawberry (fruit)	0.01	5	102	2.9	m/z 523→281 (quantification)
	0.10	5	102	2.5	
	0.01	5	102	2.1	m/z 525→283 (confirmation)
	0.10	5	103	2.5	
tomato (fruit)	0.01	5	98	3.9	m/z 523→281 (quantification)
	0.10	5	101	2.3	
	0.01	5	101	3.6	m/z 525→283 (confirmation)
	0.10	5	101	2.4	
lambs lettuce	0.01	5	99	3.6	m/z 523→281 (quantification)
	0.10	5	104	3.2	
	0.01	5	100	5.2	m/z 525→283 (confirmation)
	0.10	5	103	2.6	
green peas	0.01	5	90	2.8	m/z 523→281 (quantification)
	0.10	5	90	3.0	
	0.01	5	90	3.3	m/z 525→283 (confirmation)
	0.10	5	89	2.7	
wheat (straw)	0.05	5	80	6.0	m/z 523→281 (quantification)
	0.50	5	71	5.9	
	0.05	5	80	7.0	m/z 525→283 (confirmation)
	0.50	5	71	8.0	
wheat (grain)	0.01	5	100	3.7	m/z 523→281 (quantification)
	0.10	5	98	3.0	
	0.01	5	102	4.7	m/z 525→283 (confirmation)
	0.10	5	96	4.3	
barley (whole plant)	0.05	5	97	4.4	m/z 523→281 (quantification)
	0.50	5	96	0.9	
	0.05	5	98	6.1	m/z 525→283 (confirmation)
	0.50	5	96	2.0	
oilseed rape (seeds)	0.01	5	103	2.8	m/z 523→281 (quantification)
	0.10	5	104	2.2	
	0.01	5	107	3.5	m/z 525→283 (confirmation)
	0.10	5	104	3.4	

Table A 2: Characteristics for the analytical method used for validation of cis-deltamethrin and its isomers trans- and α-(R)- residues in different plant matrices

	***cis*-Deltamethrin**	***trans*-deltamethrin**	**α-(R)-deltamethrin**
Specificity	mass spectrum is provided in Appendix 4 of the method report blank value < 30 % LOQ)	mass spectrum is provided in Appendix 4 of the method report blank value < 30 % LOQ)	mass spectrum is provided in Appendix 4 of the method report blank value < 30 % LOQ)
Calibration (type, number of data points)	individual calibration data presented in Appendix 1 calibration line equation presented for each MRM number of data points>5 (6) MRM 523→281: R = 0.9996 MRM 525→283: R = 0.9992	individual calibration data presented in Appendix 1 calibration line equation presented for each MRM number of data points>5 (6) MRM 523→281: R = 0.9995 MRM 525→283: R = 0.9990	individual calibration data presented in Appendix 1 calibration line equation presented for each MRM number of data points>5 (6) MRM 523→281: R = 0.9987; 0.9997; 0.9999 with the three internal standards MRM 525→283: R = 0.9987; 0.9990; 0.9999 with the three internal standards
Calibration range	Internal Standard Linearity with one point calibration of the Peak Area Ratio of *cis*-Deltamethrin (MRM 523→281 and MRM 525→283) and *cis*-^{13}C-Deltamethrin (MRM 529→281) in the Concentration Range 1.25-100 ng/mL in HPLC Solvent	Internal Standard Linearity with one point calibration of the Peak Area Ratio of *trans*-Deltamethrin (MRM 523→281 and MRM 525→283) and *cis*-^{13}C-Deltamethrin (MRM 529→281) in the Concentration Range 1.25-100 ng/mL in HPLC Solvent	Internal Standard Linearity with one point calibration of the Peak Area Ratio of *a-(R)*-Deltamethrin (MRM 523→281 and MRM 525→283) and *cis*-^{13}C-Deltamethrin (MRM 529→281)or *trans*-^{13}C-Deltamethrin (MRM 529→281) or *a-(R)*-^{13}C-Deltamethrin (MRM 529→281) in the Concentration Range 1.25-100 ng/mL in HPLC Solvent
Assessment of matrix effects is presented	yes	yes	yes
Limit of determination/quantification	LOQ = 0.01 mg/kg except in wheat (straw) and barley (whole plant) where LOQ=0.05 mg/kg	LOQ = 0.01 mg/kg except in wheat (straw) and barley (whole plant) where LOQ=0.05 mg/kg	LOQ = 0.01 mg/kg except in wheat (straw) and barley (whole plant) where LOQ=0.05 mg/kg

Conclusion

The data presented demonstrate that the validated method permits the determination of residues of trans-, cis-, α-(R)- deltamethrin in strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed) with acceptable accuracy, precision and repeatability being fully compliant with current regulatory requirements.

The analytical method was successfully validated. The demands of SANCO guideline 825/00 rev. 7 (17/03/04) and SANCO 3029/99 rev. 4 (11/07/00) were fulfilled.

A 2.1.1.1.1.2 Method validation for additional plant matrices

Primary crops

Comments of zRMS:	The studies Schulte, G.; 2017 and Kaussmann, M.; Kerkering, S.; 2018 have been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL. 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) for determination of deltamethrin and its isomers, alpha-R-deltamethrin and trans-deltamethrin. Additional validations were condicted for various additional plant matrices during the conduct of the residue studies. The limit of quantification (LOQ) for all analytes is 0.01 mg/kg except in oilseed rape (green material), oilseed rape (straw), barley (whole plant), barley (straw), wheat (whole plant), wheat (straw), maize/corn (green material) and maize/corn (rest of plant) where the LOQ is 0.05 mg/kg. The individual and average recoveries at each fortification level and overall per matrix were within the range of 70 – 110% for all analytes. The RSD values were below 20%. All method validation data complies with the current guideline requirements for data collection methods. The validation of method 00855/M004 can therefore be considered successful for the additional tested matrices.

Reference:	**KCP 5.1/02**
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):	

Reference:	**KCP 5.1/03**
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):	

Reference:	**KCP 5.1/04**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on grape after high or low-volume spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy
Report:	Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.; 2016; 14-2095; M-560047-01-1
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 Guideline No. 860.1500 on Crop Field Trials
Deviations:	yes, see report
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/05**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on grape after high and low-volume spray application of deltamethrin & flupyradifurone EC 085 in Germany and France (North)
Report:	Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.; 2016; 14-2096; M-559743-01-1
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 Guideline No. 860.1500 on Crop Field Trials
Deviations:	yes, see report
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/06**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in Italy, southern France, Spain and Greece
Report:	Kaussmann, M.; Kowalski, N.; 2018; 16-2194; M-634135-01-1
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:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/07**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in northern France, Hungary, The United Kingdom and Poland
Report:	Miara, C.; Kowalski, N.; 2018; 16-2145; M-645130-01-1
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 Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/08**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy
Report:	Kaussmann, M.; Kowalski, N.; 2018; 16-2195; M-629954-01-1
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 Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/09**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in France (South), Italy, Spain and Greece
Report:	Noss, G.; 2017; 15-2130; M-572779-03-1
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:	not specified
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/10**
Title:	Amendment no. 3 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring barley after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and United Kingdom
Report:	Schulte, G.; 2017; 15-2131; M-580973-04-1
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:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/11**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy, Spain and Greece
Report:	Kaussmann, M.; Miara, C.; 2018; 16-2034; M-634112-01-1
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:	Yes (see report)
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/12**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring barley after spray application of deltamethrin & flupyradifurone EC 085 in the Netherlands, Germany and Belgium
Report:	Kaussmann, M.; 2018; 16-2035; M-634410-01-1
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):	

Reference:	**KCP 5.1/13**
Title:	Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in Italy, Spain and Portugal
Report:	Schulte, G.; 2017; 15-2127; M-580063-03-1
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:	none
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/14**
Title:	Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on spring wheat and winter wheat after spray application of deltamethrin & flupyradifurone EC 085 in Germany, the Netherlands and Belgium
Report:	Schulte, G.; 2017; 15-2129; M-580528-03-1
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:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/15**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy and Spain
Report:	Kaussmann, M.; Kerkering, S.; 2018; 16-2032; M-633925-01-1
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:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/16**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring wheat after spray application of deltamethrin & flupyradifurone EC 085 in Belgium, Germany and the Netherlands
Report:	Kaussmann, M.; Kerkering, S.; 2018; 16-2033; M-634190-01-1
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):	

Reference:	**KCP 5.1/17**
Title:	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, France (South) and Italy
Report:	Schulte, G.; 2017; 15-2133; M-574144-02-1
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:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/18**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and the Netherlands
Report:	Schulte, G.; Kerkering, S.; 2018; 16-2192; M-628803-01-1
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):	

Reference:	**KCP 5.1/19**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, southern France and Italy
Report:	Schulte, G.; Kuester, S.; Kerkering, S.; 2018; 16-2100; M-621728-01-1
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:	Yes (see report)
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/20**
Title:	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and the Netherlands
Report:	Schulte, G.; 2017; 15-2134; M-574350-02-1
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

Full validation data for deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin were documented with the method 00855/M004 itself for matrices representing five major crop groups, including strawberry (fruit), tomato (fruit), lambs lettuce, green peas, wheat (grain), wheat (straw), barley (whole plant) and oilseed rape (seed).
For the crops submitted in this application, additional validations were performed for various additional plant matrices during the conduct of the residue studies.

The limit of quantification (LOQ) for all analytes is 0.01 mg/kg except in oilseed rape (green material), oilseed rape (straw), barley (whole plant), barley (straw), wheat (whole plant), wheat (straw), maize/corn (green material) and maize/corn (rest of plant) where the LOQ is 0.05 mg/kg.

Results and discussions

Apparent residues in control samples were below 30% of the LOQ. Mean recoveries per fortification level for both analytes were in a range of 70 – 110% with RSD < 20%.

Studies 14-2095, 16-2194, 16-2195, 15-2130, 16-2034, 15-2127, 16-2032, 15-2133 and 16-2100 are only submitted for information purposes.

Table A 3: Recovery results from method validation for deltamethrin residues using the analytical method 00855/M004

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin					
oilseed rape (seeds)	0.01	5	92	9.7	m/z 523 → 281 (16-2044)
	0.10	5	90	13.2	
oilseed rape (green material)	0.05	5	98	0.9	m/z 523 → 281 (15-2132)
	0.50	5	101	1.0	
	0.05	5	103	2.3	m/z 525 → 283 (15-2132)
	0.50	5	99	3.1	
oilseed rape (straw)	0.05	5	99	2.4	m/z 523 → 281 (15-2132)
	0.50	5	101	1.3	
	0.05	5	101	4.1	m/z 525 → 283 (15-2132)
	0.50	5	102	1.3	
grape (bunches of grape)	0.01	5	99	3.1	m/z 523 → 281 (14-2095, 14-2096)
	0.10	5	101	2.7	
grape (berry)	0.01	3	89	4.2	m/z 523 → 281 (14-2095, 14-2096)
	0.10	3	95	9.6	
sunflower (kernel)	0.01	3	85	2.4	m/z 523 → 281 (16-2194)
	0.10	3	84	3.0	
	0.01	4	84	3.8	m/z 523 → 281 (16-2145, 16-2195)
	0.10	4	84	1.5	
	0.01	4	81	8.2	m/z 525 → 283 (16-2145, 16-2195)
	0.10	4	84	2.0	
barley (whole plant without root)	0.05	3	98	2.0	m/z 523 → 281 (15-2130, 15-2131)
	0.50	3	98	0.6	
	0.05	5	101	3.8	m/z 523 → 281 (16-2034)
	0.50	4	96	3.4	
	0.05	4	101	2.2	m/z 525 → 283 (16-2034)
	0.50	4	98	4.1	
	0.05	4	100	3.8	m/z 523 → 281 (16-2035)
	0.50	4	96	3.4	
	0.05	4	101	2.2	m/z 525 → 283 (16-2035)
	0.50	4	98	4.1	
barley (grain)	0.01	3	100	4.4	m/z 523 → 281 (15-2130, 15-2131)
	0.10	3	99	1.7	
	0.01	4	102	5.1	m/z 523 → 281 (16-2034)
	0.10	4	94	5.9	
	0.01	4	96	8.9	m/z 525 → 283 (16-2034)
	0.10	4	93	5.0	
	0.01	4	102	5.1	m/z 523 → 281 (16-2035)
	0.10	4	94	5.9	
	0.01	4	96	8.9	m/z 525 → 283 (16-2035)
	0.10	4	93	5.0	

barley (straw)	0.05	3	88	1.7	m/z 523 → 281 (15-2130, 15-2131)
	0.50	3	90	2.3	
	0.05	5	96	3.6	m/z 523 → 281 (16-2034)
	0.50	4	93	4.2	
	0.05	4	97	7.7	m/z 525 → 283 (16-2034)
	0.50	4	94	4.1	
	0.05	4	95	2.7	m/z 523 → 281 (16-2035)
	0.50	4	93	4.2	
	0.05	4	97	7.7	m/z 525 → 283 (16-2035)
	0.50	4	94	4.1	
wheat (whole plant without root)	0.05	3	98	2.1	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.50	3	98	1.2	
Wheat (grain)	0.01	5	102	1.6	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.10	5	100	1.1	
	0.01	5	102	3.0	m/z 525 → 283 (15-2127)
	0.10	5	103	2.2	
Wheat (straw)	0.05	3	97	2.6	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.50	3	93	2.8	
Maize / Corn (green material)	0.05	3	102	0.6	m/z 523 → 281 (15-2133, 15-2134)
	0.50	3	97	1.2	
Maize / Corn (kernel)	0.01	3	105	4.2	m/z 523 → 281 (15-2133, 15-2134)
	0.10	3	100	1.0	
	0.01	4	86	9.9	m/z 523 → 281 (16-2192, 16-2100)
	0.10	4	85	4.9	
	0.01	4	87	9.5	m/z 525 → 283 (16-2192, 16-2100)
	0.10	4	84	5.8	
Maize / Corn (rest of plant)	0.05	5	95	2.7	m/z 523 → 281 (15-2133, 15-2134)
	0.50	5	99	0.8	
	0.05	5	96	3.0	m/z 525 → 283 (15-2133, 15-2134)
	0.50	5	101	1.1	
	0.05	4	94	7.2	m/z 523 → 281 (16-2192, 16-2100)
	0.50	4	91	3.3	
	0.05	4	94	15	m/z 525 → 283 (16-2192, 16-2100)
	0.50	4	91	2.7	
alpha-R-deltamethrin					
oilseed rape (seeds)	0.01	5	89	11.6	m/z 523 → 281 (16-2044)
	0.10	5	86	13.3	
oilseed rape (green material)	0.05	5	96	2.2	m/z 523 → 281 (15-2132)
	0.50	5	97	1.9	
	0.05	5	97	2.6	m/z 525 → 283 (15-2132)
	0.50	5	99	2.7	
	0.05	5	97	5.2	

oilseed rape (straw)	0.50	5	98	1.6	m/z 523 → 281 (15-2132)
	0.05	5	97	6.6	m/z 525 → 283 (15-2132)
	0.50	5	100	2.9	
grape (bunches of grape)	0.01	5	97	3.0	m/z 523 → 281 (14-2095, 14-2096)
	0.10	5	99	2.2	
grape (berry)	0.01	3	88	3.4	m/z 523 → 281 (14-2095, 14-2096)
	0.10	3	92	8.6	
sunflower (kernel)	0.01	3	80	0.7	m/z 523 → 281 (16-2194)
	0.10	3	80	3.2	
	0.01	4	85	4.9	m/z 523 → 281 (16-2145, 16-2195)
	0.10	4	81	2.7	
	0.01	4	87	6.8	m/z 525 → 283 (16-2145, 16-2195)
	0.10	4	80	5.5	
barley (whole plant without root)	0.05	3	96	1.0	m/z 523 → 281 (15-2130, 15-2131)
	0.50	3	95	1.8	
	0.05	4	96	5.8	m/z 523 → 281 (16-2034)
	0.50	4	93	1.9	
	0.05	4	98	6.9	m/z 525 → 283 (16-2034)
	0.50	4	95	5.2	
	0.05	4	96	5.8	m/z 523 → 281 (16-2035)
	0.50	4	93	1.9	
	0.05	4	98	6.9	m/z 525 → 283 (16-2035)
	0.50	4	95	5.2	
barley (grain)	0.01	3	98	4.4	m/z 523 → 281 (15-2130, 15-2131)
	0.10	3	97	2.4	
	0.01	4	96	8.0	m/z 523 → 281 (16-2034)
	0.10	4	91	4.8	
	0.01	4	91	6.5	m/z 525 → 283 (16-2034)
	0.10	4	88	4.8	
	0.01	4	96	8.2	m/z 523 → 281 (16-2035)
	0.10	4	91	4.8	
	0.01	4	91	6.5	m/z 525 → 283 (16-2035)
	0.10	4	88	4.8	
barley (straw)	0.05	3	89	2.6	m/z 523 → 281 (15-2130, 15-2131)
	0.50	3	93	0.6	
	0.05	4	94	3.3	m/z 523 → 281 (16-2034)
	0.50	4	92	3.9	
	0.05	4	93	4.9	m/z 525 → 283 (16-2034)
	0.50	4	93	6.0	
	0.05	4	94	3.3	m/z 523 → 281 (16-2035)
	0.50	4	92	3.9	
	0.05	4	93	4.9	

	0.50	4	93	6.0	m/z 525 → 283 (16-2035)
wheat (whole plant without root)	0.05	3	96	2.2	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.50	3	97	3.0	
Wheat (grain)	0.01	5	100	1.8	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.10	5	99	3.3	
	0.01	5	101	4.1	m/z 525 → 283 (15-2127)
	0.10	5	104	3.0	
Wheat (straw)	0.05	3	94	2.5	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.50	3	91	3.5	
Maize / Corn (green material)	0.05	3	101	1.0	m/z 523 → 281 (15-2133, 15-2134)
	0.50	3	95	0.0	
Maize / Corn (kernel)	0.01	3	98	6.5	m/z 523 → 281 (15-2133, 15-2134)
	0.10	3	96	1.0	
	0.01	4	88	7.5	m/z 523 → 281 (16-2192, 16-2100)
	0.10	4	85	3.8	
	0.01	4	90	15	m/z 525 → 283 (16-2192, 16-2100)
	0.10	4	84	5.1	
Maize / Corn (rest of plant)	0.05	5	95	1.2	m/z 523 → 281 (15-2133, 15-2134)
	0.50	5	95	1.7	
	0.05	5	96	2.8	m/z 525 → 283 (15-2133, 15-2134)
	0.50	5	99	2.4	
	0.05	4	98	2.3	m/z 523 → 281 (16-2192, 16-2100)
	0.50	4	92	2.1	
	0.05	4	96	3.3	m/z 525 → 283 (16-2192, 16-2100)
	0.50	4	93	2.6	
trans-deltamethrin					
oilseed rape (seeds)	0.01	5	89	12.4	m/z 523 → 281 (16-2044)
	0.10	5	93	13.3	
oilseed rape (green material)	0.05	5	95	2.6	m/z 523 → 281 (15-2132)
	0.50	5	101	1.8	
	0.05	5	98	3.7	m/z 525 → 283 (15-2132)
	0.50	5	100	1.3	
oilseed rape (straw)	0.05	5	98	2.2	m/z 523 → 281 (15-2132)
	0.50	5	98	1.3	
	0.05	5	104	5.7	m/z 525 → 283 (15-2132)
	0.50	5	99	1.7	
grape (bunches of grape)	0.01	5	98	2.8	m/z 523 → 281 (14-2095, 14-2096)
	0.10	5	97	2.8	
grape (berry)	0.01	3	88	6.2	m/z 523 → 281 (14-2095, 14-2096)
	0.10	3	92	8.6	
sunflower (kernel)	0.01	3	83	0.7	

	0.10	3	83	2.8	m/z 523 → 281 (16-2194)
	0.01	4	88	5.1	m/z 523 → 281 (16-2145, 16-2195)
	0.10	4	85	5.0	
	0.01	4	86	1.5	m/z 525 → 283 (16-2145, 16-2195)
	0.10	4	84	4.2	
barley (whole plant without root)	0.05	3	96	2.2	m/z 523 → 281 (15-2130, 15-2131)
	0.50	3	97	1.6	
	0.05	4	102	1.7	m/z 523 → 281 (16-2034)
	0.50	4	96	3.1	
	0.05	4	97	5.2	m/z 525 → 283 (16-2034)
	0.50	4	98	7.5	
	0.05	4	102	1.7	m/z 523 → 281 (16-2035)
	0.50	4	96	3.1	
	0.05	4	97	5.2	m/z 525 → 283 (16-2035)
	0.50	4	98	7.5	
barley (grain)	0.01	3	101	4.0	m/z 523 → 281 (15-2130, 15-2131)
	0.10	3	99	3.5	
	0.01	4	99	5.9	m/z 523 → 281 (16-2034)
	0.10	4	91	3.0	
	0.01	4	96	3.3	m/z 525 → 283 (16-2034)
	0.10	4	92	3.4	
	0.01	4	99	5.9	m/z 523 → 281 (16-2035)
	0.10	4	91	3.0	
	0.01	4	96	3.3	m/z 525 → 283 (16-2035)
	0.10	4	92	3.4	
barley (straw)	0.05	3	91	4.8	m/z 523 → 281 (15-2130, 15-2131)
	0.50	3	91	2.3	
	0.05	4	94	3.4	m/z 523 → 281 (16-2034)
	0.50	4	92	1.6	
	0.05	4	98	8.4	m/z 525 → 283 (16-2034)
	0.50	4	93	4.0	
	0.05	4	94	3.4	m/z 523 → 281 (16-2035)
	0.50	4	92	1.6	
	0.05	4	98	8.4	m/z 525 → 283 (16-2035)
	0.50	4	93	4.0	
wheat (whole plant without root)	0.05	3	94	2.2	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.50	3	97	2.1	
Wheat (grain)	0.01	5	103	3.9	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.10	5	100	2.3	
	0.01	5	103	9.1	m/z 525 → 283 (15-2127)
	0.10	5	104	3.0	

Wheat (straw)	0.05	3	97	3.1	m/z 523 → 281 (15-2127, 16-2032, 16-2033, 15-2129)
	0.50	3	92	4.4	
Maize / Corn (green material)	0.05	3	101	2.6	m/z 523 → 281 (15-2133, 15-2134)
	0.50	3	94	0.6	
Maize / Corn (kernel)	0.01	3	100	0.6	m/z 523 → 281 (15-2133, 15-2134)
	0.10	3	101	2.8	
	0.01	4	84	6.1	m/z 523 → 281 (16-2192, 16-2100)
	0.10	4	84	4.6	
	0.01	4	90	13	m/z 525 → 283 (16-2192, 16-2100)
	0.10	4	82	6.2	
Maize / Corn (rest of plant)	0.05	5	97	3.4	m/z 523 → 281 (15-2133, 15-2134)
	0.50	5	98	1.5	
	0.05	5	101	6.6	m/z 525 → 283 (15-2133, 15-2134)
	0.50	5	98	2.4	
	0.05	4	98	5.8	m/z 523 → 281 (16-2192, 16-2100)
	0.50	4	89	2.0	
	0.05	4	97	11	m/z 525 → 283 (16-2192, 16-2100)
	0.50	4	92	3.1	

Table A 4: Characteristics for the analytical method 00855/M004 used for validation of deltamethrin and its isomers, alpha-R-deltamethrin and trans-deltamethrin

	***cis*-Deltamethrin, *trans*-deltamethrin, α-(R)-deltamethrin**
Specificity	mass spectrum is provided in Appendix 4 of the original method report blank value < 30 % LOQ
Calibration (type, number of data points)	Calibration data presented Calibration line presented number of data points ≥ 5 R > 0.99
Calibration range	Range for all studies: 0.250 – 200 µg/L for all analytes (corresponds to 0.01 to 8 mg/kg). The range may differ in single studies with in-study validations.
Assessment of matrix effects is presented	The use of stable isotopically labelled internal standards compensates for matrix effect.
Limit of determination/quantification	LOQ = 0.01 mg/kg except in oilseed rape (green material), oilseed rape (straw), barley (whole plant), barley (straw), wheat (whole plant), wheat (straw), maize/corn (green material) and maize/corn (rest of plant) where LOQ = 0.05 mg/kg

Conclusion
All method validation data are in compliance with the guideline requirements for data generation methods. Method 00855/M004 can therefore be considered successfully validated for the determination of residues of deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin in all additional plant matrices relevant to this submission.

A 2.1.1.1.1.3 Extraction efficiency - Cross validation of extraction methods

Comments of zRMS:	The study of Schoening, R.; Willmes, J. (2014) has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *Both methods, 00855/M004 and 00086/M089, meet all necessary criteria to sufficiently extract and determine the residues of deltamethrin, cis-deltamethrin, trans-isomer and*

	α-R-isomer of deltamethrin in plant matrices (barley grain, lettuce head, orange fruit and olive fruit). *The study is acceptable.*

Reference:	**KCP 5.1/21**
Title:	Cross validation of extraction methods for the determination of residues of deltamethrin in plant materials by HPLC-MS/MS
Report:	Schoening, R.; Willmes, J.; 2014; MR-14/012; M-481952-02-1
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 and repealing Council Directives 79/117/EEC and 91/414/EEC European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, 11/07/00 Guidance document on residue analytical methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010 US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method
Deviations:	not specified
GLP/GEP:	no
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method 00855/M004 (M-356934-01-1) was developed to determine the residues of parent *cis*-deltamethrin, *trans*-isomer and α-R-isomer of deltamethrin and the multi-residue method 00086/M089 (M-351076-01-1) derived from DFG S19 was developed to determine the residues of parent *cis*-deltamethrin in plant matrices. The objective of this study was to investigate the extraction efficiency of these two methods in comparison to the methods used in the metabolism studies.

For comparison of the extraction efficiency:

- Incurred residues of deltamethrin from barley grain, lettuce head, orange fruit and olive fruit samples from residue trials conducted in 2010 and 2011 were extracted according to the methods 00855/M004 and 00086/M089, as well as according to the procedure used during the metabolism studies 305W-1 (M-149515-01-1), 89-0090 & HLA 6187-110 (M-149567-01-1) and 306W-1 (M-149571-01-1). All thus obtained extracts were measured using the same approach as in the method 00855/M004.

Comparison of the residue levels of *cis*-deltamethrin, *trans-isomer* of deltamethrin and α-R-isomer of deltamethrin determined after extraction according to the methods 00855/M004 and 00086/M089 with the residue levels of *cis*-deltamethrin, *trans-isomer* and α-R-isomer of deltamethrin determined after extraction according to the metabolism study 305W-1 (M-149515-01-1), 89-0090 & HLA 6187-110 (M-149567-01-1) and 306W-1 (M-149571-01-1) provided the extraction efficiency of the methods 00855/M004 and 00086/M089.

Extraction

Barley, lettuce, orange and olive samples were extracted according to the methods 00855/M004 (M-356934-01-1) and 00086/M089 (M-351076-01-1), as well as according to the procedure used during the metabolism studies 305W-1 (M-149515-01-1), 89-0090 & HLA 6187-110 (M-149567-01-1) and 306W-1 (M-149571-01-1). All thus obtained extracts were measured using the same approach as in method 00855/M004.

- The lettuce head and orange fruit samples were extracted once with methanol/water (1/1, v/v) and twice with methanol as described in the metabolism study 305W-1 (M-149515-01-1).

- The olive fruit samples were extracted three times with hexane, three times with $CHCl_3$/methanol (1/3, v/v) and three times with methanol/water (8/3, v/v) as described in the metabolism study 89-0090 & HLA 6187-110 (M-149567-01-1).
- The barley grain samples were extracted three times with methanol, twice with methanol/water (1/2, v/v) and twice with water as described in the metabolism study 306W-1 (M-149571-01-1).
- In method 00855/M004 the samples of barley grain, lettuce head and orange fruit were extracted with a mixture of acetone/dichloromethane/n-hexane (1/1/1, v/v/v). Olive fruit samples were extracted with acetone/dichloromethane/n-hexane (1/1/1, v/v/v), evaporated to dryness, re-dissolved in acetonitrile and afterwards stirred against hexane.
- In method 00086/M089 (M-351076-01-1) the extraction was carried out with acetone/water (2/1, v/v) followed by stirring with ethyl acetate/cyclohexane (1/1, v/v).

The extracts obtained using each of the four procedures were measured using the same approach as in method 00855/M004.

Results and discussions

During the analysis of the samples coming out of the residue trials concurrent recoveries were performed. All results of the concurrent recoveries were acceptable for the purpose of the current study.

Each sample was analysed three times using each extraction procedure.
For each analyte, the extraction efficiency of the methods 00855/M004 and 00086/M89 is calculated as the ratio (expressed as percentage) between the average residues measured after extracting the samples according to these procedures and the average residues measured using the corresponding procedure of the metabolism studies. Either studies 305W-1 (M-149515-01-1) for lettuce and orange samples, or 89-0090 & HLA 6187-110 (M-149567-01-1) for olive samples and or 306W-1 (M-149571-01-1) for barley grain.
It was noticed that for barley grain samples, the extracts resulting of the metabolism procedures presented content **in trans-isomer**. For lettuce head samples and olive fruit samples, the extracts resulting of the metabolism procedures presented content **in α-R-isomer**. It is very unlikely that the metabolism extraction procedures are able to extract these isomers and not the residue extraction procedure. It seems more probably that due to the length and complexity of the metabolism extraction procedure isomerization of the cis-deltamethrin into trans-isomer and α-R-isomer has occurred in some extent.
Based on this hypothesis a second extraction efficiency calculation was performed, using the sum of cis-deltamethrin and trans-isomer or cis-deltamethrin and α-R-isomer observed after extraction according to the metabolism conditions. This second calculation is mentioned at the bottom of Table 1, Table 3 and Table 4 provided in document M-481952-02-1.

No residues above the LOQ were found in the control samples.
For the all samples results were not corrected for concurrent recoveries.

Table A 5: Residue Levels and Extraction Efficiency for cis-deltamethrin and its isomers *α-R*-deltamethrin and trans-deltamethrin in Barley Grain

Analyte	Sample 10-2032-04	Mean residue levels [mg/kg] *Extraction Efficiency [%]*		
		Metabolism conditions (306W-1)	Method 00855/M004	Method 00086/M089
cis-deltamethrin	0011E	0.086 mg/kg *100%*	0.085 mg/kg *99%*	0.069 mg/kg *80%*
trans-isomer		0.015 mg/kg *100%*	-- mg/kg *-- %*	-- mg/kg *-- %*
αlpha-*R*-isomer		-- mg/kg *-- %*	-- mg/kg *--%*	-- mg/kg *--%*
cis-deltamethrin + trans-isomer		0.101 mg/kg *100%*	0.085 mg/kg *84%*	0.069 mg/kg *68%*

Note : The extraction efficiency of the methods 00855/M004 and 00086/M089 is calculated as the ratio (expressed as percentage) between the average residues measured using these extraction procedures and the average residues measured using the extraction procedure of the metabolism study 306W-1 (M-149571-01-1).

Table A 6: Residue Levels and Extraction Efficiency for cis-deltamethrin and its isomers *α-R*-deltamethrin and trans-deltamethrin in Orange Fruit

Analyte	Sample 10-2302	Mean residue levels [mg/kg] *Extraction Efficiency [%]*		
		Metabolism conditions (305W-1)	Method 00855/M004	Method 00086/M089
cis-deltamethrin	110024893	0.028 mg/kg *100%*	0.027 mg/kg *95%*	0.030 mg/kg *106%*
	100215479	0.029 mg/kg *100%*	0.026 mg/kg *89%*	0.030 mg/kg *104%*
trans-isomer	110024893	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %
	100215479	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %
αlpha-R-isomer	110024893	-- mg/kg -- %	-- mg/kg --%	-- mg/kg --%
	100215479	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %

Note : The extraction efficiency of the methods 00855/M004 and 00086/M089 is calculated as the ratio (expressed as percentage) between the average residues measured using these extraction procedures and the average residues measured using the extraction procedure of the metabolism study 305W-1 (M-149515-01-1).

Table A 7: Residue Levels and Extraction Efficiency for cis-deltamethrin and its isomers *α-R*-deltamethrin and trans-deltamethrin in Olive Fruit.

Analyte	Sample S10-00015	Mean residue levels [mg/kg] *Extraction Efficiency [%]*		
		Metabolism conditions (89-0090 & HLA 6187-110)	Method 00855/M004	Method 00086/M089
cis-deltamethrin	03-002A	0.410 mg/kg *100%*	0.466 mg/kg *114%*	0.520 mg/kg *126%*
	04-002A	0.414 mg/kg *100%*	0.540 mg/kg *131%*	0.612 mg/kg *149%*
trans-isomer	03-002A	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %
	04-002A	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %
αlpha-R-isomer	03-002A	-- mg/kg -- %	-- mg/kg --%	-- mg/kg --%
	04-002A	0.017 mg/kg *100%*	-- mg/kg -- %	-- mg/kg -- %
cis-deltamethrin + alpha-R-isomer	03-002A	0.410 mg/kg *100%*	0.466 mg/kg *114%*	0.520 mg/kg *126%*
	04-002A	0.431 mg/kg *100%*	0.540 mg/kg *126%*	0.612 mg/kg *143%*

Note : The extraction efficiency of the methods 00855/M004 and 00086/M089 is calculated as the ratio (expressed as percentage) between the average residues measured using these extraction procedures and the average residues measured using the extraction procedure of the metabolism study 89-0090 & HLA 6187-110 (M-149567-01-1).

Table A 8: Residue Levels and Extraction Efficiency for cis-deltamethrin and its isomers *α-R*-deltamethrin and trans-deltamethrin in Lettuce Head.

Analyte	Sample 11-2052	Mean residue levels [mg/kg] *Extraction Efficiency [%]*		
		Metabolism conditions (305W-1)	Method 00855/M004	Method 00086/M089
cis-deltamethrin	01-0016E	0.275 mg/kg *100%*	0.316 mg/kg *122%*	0.328 mg/kg *124%*
	02-0010E	0.426 mg/kg *100%*	0.528 mg/kg *127%*	0.594 mg/kg *143%*
trans-isomer	01-0015E	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %
	02-0010E	-- mg/kg -- %	-- mg/kg -- %	-- mg/kg -- %
αlpha-R-isomer	01-0015E	0.054 mg/kg *100%*	-- mg/kg --%	-- mg/kg --%
	02-0010E	0.083 mg/kg *100%*	-- mg/kg -- %	-- mg/kg -- %

cis-deltamethrin + alpha-R-isomer	01-0016E	0.329 mg/kg *100%*	0.316 mg/kg *103%*	0.328 mg/kg *105%*
	02-0010E	0.509 mg/kg *100%*	0.528 mg/kg *106%*	0.594 mg/kg *120%*

Note : The extraction efficiency of the methods 00855/M004 and 00086/M089 is calculated as the ratio (expressed as percentage) between the average residues measured using these extraction procedures and the average residues measured using the extraction procedure of the metabolism study 305W-1 (M-149515-01-1).

Conclusion
The methods 00855/M004 and 00086/M089 meet all necessary criteria to sufficiently extract and determine the residues of *cis*-deltamethrin, *trans*-isomer and α-*R*-isomer of deltamethrin in plant matrices.

A 2.1.1.1.2 Analytical ~~method~~ method 2 (01207)

A 2.1.1.1.2.1 Method validation

Comments of zRMS:	The QuEChERS method 01207 has been validated for the determination of deltamethrin (trans, cis and α-R isomers) in apple (fruit), orange (whole fruit), carrot (root), oilseed rape (seed) and bean (dry seeds) with LOQ of 0.01 mg/kg for each compound in each matrix. Mean recoveries for each fortification level and the overall mean recovery were within the 70 - 110% range for all matrices. Relative standard deviations were below 20%. The method is acceptable.

Reference:	**KCP 5.1/22**
Title:	Validation of the BCS method no. 01207 (based on modified QuEChERS method) for the determination of selected BCS analytes and their metabolites in carrot, apple, orange, oilseed rape seed and beans
Report:	Lakaschus, S.; Amann, S.; Winter, O.; Gizler, A.; 2013; S10-00279; M-424756-02-1
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 and repealing Council Directives 79/117/EEC and 91/414/EEC European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, 11/07/00 Guidance document on residue analytical methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010 US EPA Residue Chemistry Test Guideline OCSSP 860.1340: Residue Analytical Method
Deviations:	Not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The QuEChERS multi residue method 01207 was validated for the determination of deltamethrin (trans, cis and α-R isomers) and other analytes in/on plant materials. The analytes were extracted from apple (fruit), orange (whole fruit), carrot (root), oilseed rape (seed) and bean (dry seeds) with acetonitrile/water (4/1, v/v). An aliquot of the extract was taken and the internal stable labeled standards were added. The solution was subjected to LC-MS/MS. The internal standard procedure, using stable isotopically labelled internal standards was used for calibration.

The Limit of Quantification for this method was 0.01 mg/kg for each analyte and sample material.

An aliquot of the sample solution was injected into the high performance liquid chromatograph and

subjected to reversed phase chromatography coupled with tandem mass spectrometry (MS/MS) with electrospray ionisation. The MS/MS instrument was operated in the Multiple Reaction Monitoring mode (MRM). The pseudomolecular ions of the analytes ([M+H]+, [M-H]- or any adducts) were selected by the first quadrupole. These precursor ions were impulsed with nitrogen in the collision cell (second quadrupole) and the resulting fragment ions (product ions) were separated according to their m/z ratio in the third quadrupole. Two of these product ions per analyte were selected: one product ion (MRM-transition) serving for quantitation and the second for confirmation (MRM used for quantification: Deltamethrin (all isomers) m/z 523 → 281; MRM used for confirmation: Deltamethrin (all isomers) m/z 525 → 283).

Results and discussions

Recovery rates were determined at fortification levels of 0.01 mg/kg (= LOQ level), and 0.10 mg/kg. The lowest fortification level providing a mean recovery between 70 and 110% with a relative standard deviation of < 20% per definition corresponding to the Limit of Quantitation (LOQ), provided that the blank values were below 30% at this level. Recovery experiments were conducted by fortifications of untreated control samples with defined amounts of each analyte prior to analysis. Mean recoveries for each fortification level and the overall mean recovery were within the 70 - 110% range for all matrices.

Up to three untreated control samples of different origin were examined. For all analytes the residues found were below the LOD (< 0.003 mg/kg).

As a measure for the precision, the intra-laboratory repeatability (n=5) is given as relative standard deviation (% RSD) for all sample materials at fortification levels of 0.01 and 0.10 mg/kg. Relative standard deviations were below 20%.

Two MRM transitions were monitored for each analyte and each matrix tested. For each compound, a 2nd MRM transitios is considered to be suitable for confirmatory purposes in all tested sample materials at the LOQ level of 0.01 mg/kg.

Table A 9: Recovery rates and precision results (repeatability) of trans-deltamethrin, cis-deltamethrin and α-R-deltamethrin

Analyte	Sample Material	FL [mg/kg]	Single Values [%]	Mean Value [%]	RSD [%]	LOQ [mg/kg]
trans-deltamethrin	Apple MRM 523 → 281 (quantification)	0.01	93, 107, *, 93, 96	97	6.8	0.01
		0.10	104, 118, 106, 103, 110	108	5.6	
			Overall Recovery (n = 9)	**103**	**8.0**	
	Apple MRM 525 → 283 (confirmation)	0.01	92, 91, *, 87, 86	89	3.3	
		0.10	107, 98, 102, 94, 87	98	7.8	
			Overall Recovery (n = 9)	**94**	**7.8**	
	Orange MRM 523 → 281 (quantification)	0.01	72, 78, 97, 93, 98	88	14	
		0.10	84, 94, 102, 94, 103	95	8.0	
			Overall Recovery (n = 10)	**92**	**11**	
	Orange MRM 525 → 283 (confirmation)	0.01	101, 106, 94, 105, 105	102	4.9	
		0.10	83, 90, 100, 92, 102	93	8.3	
			Overall Recovery (n = 10)	**98**	**7.9**	
	Carrot MRM 523 → 281 (quantification)	0.01	109, 92, 97, 75, *	93	15	
		0.10	99, 102, 104, 79, 106	98	11	
			Overall Recovery (n = 9)	**96**	**12**	
	Carrot MRM 525 → 283 (confirmation)	0.01	84, 87, 103, 92, *	92	9.1	
		0.10	100, 108, 99, 79, 104	98	11	
			Overall Recovery (n = 9)	**95**	**11**	
	Dry bean MRM 523 → 281 (quantification)	0.01	88, 88, 75, 86, 83	84	6.5	
		0.10	79, 83, 77, 75, 79	79	3.8	
			Overall Recovery (n = 10)	**81**	**6.2**	
	Dry bean MRM 525 → 283 (confirmation)	0.01	78, 78, 75, 87, 84	80	6.1	
		0.10	75, 84, 70, 79, 78	77	6.7	
			Overall Recovery (n = 10)	**79**	**6.4**	

	Oilseed rape MRM 523 → 281 (quantification)	0.01	90, *, 101, 86, 83	90	8.7	
		0.10	85, 88, 78, 81, 81	83	4.7	
			Overall Recovery (n = 9)	**86**	**7.9**	
	Oilseed rape MRM 525 → 283 (confirmation)	0.01	94, *, 96, 87, 93	93	4.2	
		0.10	78, 80, 71, 82, 78	78	5.3	
			Overall Recovery (n = 9)	**84**	**10**	
cis-deltamethrin	Apple MRM 523 → 281 (quantification)	0.01	86, 83, *, 84, 95	87	6.3	0.01
		0.10	89, 99, 89, 100, 90	93	6.0	
			Overall Recovery (n = 9)	**91**	**6.8**	
	Apple MRM 525 → 283 (confirmation)	0.01	86, 84, *, 81, 87	85	3.1	
		0.10	84, 94, 85, 96, 88	89	6.0	
			Overall Recovery (n = 9)	**87**	**5.6**	
	Orange MRM 523 → 281 (quantification)	0.01	84, 73, 83, 92, 89	84	8.6	
		0.10	84, 93, 97, 95, 99	94	6.2	
			Overall Recovery (n = 10)	**89**	**8.9**	
	Orange MRM 525 → 283 (confirmation)	0.01	83, 74, 87, 96, 93	87	10	
		0.10	79, 89, 93, 91, 96	90	7.2	
			Overall Recovery (n = 10)	**88**	**8.4**	
	Carrot MRM 523 → 281 (quantification)	0.01	89, 93, 92, 86, *	90	3.5	
		0.10	96, 101, 99, 77, 100	95	11	
			Overall Recovery (n = 9)	**93**	**8.4**	
	Carrot MRM 525 → 283 (confirmation)	0.01	89, 98, 98, 96, *	95	4.5	
		0.10	95, 102, 96, 78, 101	94	10	
			Overall Recovery (n = 9)	**95**	**7.7**	
	Dry bean MRM 523 → 281 (quantification)	0.01	78, 87, 62, 86, 86	80	13	
		0.10	79, 84, 85, 81, 83	82	2.9	
			Overall Recovery (n = 10)	**81**	**9.1**	
	Dry bean MRM 525 → 283 (confirmation)	0.01	81, 90, 60, 83, 85	80	14	
		0.10	78, 81, 82, 81, 83	81	2.3	
			Overall Recovery (n = 10)	**80**	**9.7**	
	Oilseed rape MRM 523 → 281 (quantification)	0.01	80, **, 88, 78, 80	82	5.4	
		0.10	78, 79, 81, 79, 79	79	1.4	
			Overall Recovery (n = 9)	**80**	**3.8**	
	Oilseed rape MRM 525 → 283 (confirmation)	0.01	81, **, 80, 79, 77	79	2.2	
		0.10	79, 81, 78, 79, 78	79	1.6	
			Overall Recovery (n = 9)	**79**	**1.7**	
α-R-deltamethrin	Apple MRM 523 → 281 (quantification)	0.01	87, 86, *, 90, 96	90	5.0	0.01
		0.10	89, 99, 91, 102, 96	95	5.7	
			Overall Recovery (n = 9)	**93**	**6.0**	
	Apple MRM 525 → 283 (confirmation)	0.01	76, 79, *, 77, 84	79	4.5	
		0.10	84, 92, 86, 96, 86	89	5.7	
			Overall Recovery (n = 9)	**84**	**7.9**	
	Orange MRM 523 → 281 (quantification)	0.01	87, 73, 86, 95, 91	86	9.6	
		0.10	84, 94, 98, 94, 101	94	6.8	
			Overall Recovery (n = 10)	**90**	**9.0**	
	Orange MRM 525 → 283 (confirmation)	0.01	83, 76, 86, 95, 94	87	9.1	
		0.10	86, 94, 101, 98, 100	96	6.4	
			Overall Recovery (n = 10)	**91**	**9.0**	
	Carrot MRM 523 → 281 (quantification)	0.01	92, 96, 85, 93, *	92	5.1	
		0.10	95, 106, 100, 80, 101	96	10	
			Overall Recovery (n = 9)	**94**	**8.5**	
	Carrot MRM 525 → 283 (confirmation)	0.01	84, 98, 91, 90, *	91	6.3	
		0.10	94, 103, 94, 78, 101	94	10	
			Overall Recovery (n = 9)	**93**	**8.6**	
	Dry bean MRM 523 → 281	0.01	83, 90, 61, 90, 82	81	15	
		0.10	82, 85, 83, 83, 86	84	2.0	

	(quantification)		**Overall Recovery (n = 10)**	**83**	**9.9**	
	Dry bean MRM 525 → 283 (confirmation)	0.01	83, 86, 63, 86, 86	81	12	
		0.10	81, 86, 84, 86, 85	84	2.5	
			Overall Recovery (n = 10)	**83**	**8.6**	
	Oilseed rape MRM 523 → 281 (quantification)	0.01	86, **, 82, 74, 78	80	6.5	
		0.10	78, 76, 79, 78, 81	78	2.3	
			Overall Recovery (n = 9)	**79**	**4.4**	
	Oilseed rape MRM 525 → 283 (confirmation)	0.01	74, **, 84, 75, 71	76	7.4	
		0.10	78, 75, 75, 79, 79	77	2.7	
			Overall Recovery (n = 9)	**77**	**4.9**	

FL = Fortification Level, RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantitation; * Spiking error, not reported; ** not reported, injection error

The stability of the analytes in solvent standard solutions was tested. For this purpose aged solvent standard solutions were quantified against freshly prepared solvent standard solutions. The aged solutions were stored in HPLC vials in the dark in a refrigerator at 4°C ± 3°C until re-analysis. Fresh solutions were prepared at the date of analysis. All three analytes were stable in standard solutions for at least 7 days of storage in a refrigerator at 4°C ± 3°C under dark conditions.

The stability in final plant extracts was checked for the tested sample materials, apple (fruit), carrot (root), orange (whole fruit), oilseed rape (seed) and bean (dry seed). The below tables show the recoveries comparing initial day of analysis and analysis after storage of the final samples at 4 °C ± 3°C under dark conditions over the given periods.

The samples for stability tests were fortified and worked up in the same way as validation samples. Three aliquots were transferred into HPLC vials and mixed with the internal standards at the day of preparation and measurement. After a period of approximately one week again three aliquots of the raw extract were taken and mixed with a freshly prepared internal standard. Calibration solutions were prepared freshly at the day of first and second analysis.

The test showed that deltamethrin (trans, cis and α-R) was stable for at least 7 days.

Table A 10: Storage stability of trans-deltamethrin, cis-deltamethrin and α-R-deltamethrin in plant extracts (using the quantifier mass transition)

Analyte	Sample Material	FL [mg/kg]	Detection	Single Values [%]	Mean Value [%]	Deviation* [%]
trans-deltamethrin	Apple	0.1	initial analysis	93, 97, 97	96	3.0
			7 days reanalysis	99, 99, 98	99	
	Carrot	0.1	initial analysis	95, 98, 92	95	5.0
			7 days reanalysis	98, 100, 102	100	
	Whole orange	0.1	initial analysis	96, 98, 100	98	3.0
			7 days reanalysis	100, 101, 101	101	
	Oilseed rape seed	0.1	initial analysis	78, 76, 79	78	11
			7 days reanalysis	92, 89, 87	89	
	Dry bean seed	0.1	initial analysis	86, 83, 82	84	9.0
			7 days reanalysis	92, 93, 95	93	
cis-deltamethrin	Apple	0.1	initial analysis	96, 96, 96	96	4.0
			7 days reanalysis	102, 100, 99	100	
	Carrot	0.1	initial analysis	99, 101, 96	99	3.0
			7 days reanalysis	101, 102, 104	102	
	Whole orange	0.1	initial analysis	96, 99, 102	99	3.0
			7 days reanalysis	103, 100, 102	102	
	Oilseed rape seed	0.1	initial analysis	78, 79, 76	78	12
			7 days reanalysis	92, 89, 90	90	
	Dry bean seed	0.1	initial analysis	86, 83, 85	85	10
			7 days reanalysis	95, 96, 94	95	
α-R-	Apple	0.1	initial analysis	100, 99, 98	99	0.0

deltamethrin			7 days reanalysis	99, 99, 99	99	
	Carrot	0.1	initial analysis	99, 102, 98	100	1.0
			7 days reanalysis	100, 103, 99	101	
	Whole orange	0.1	initial analysis	97, 98, 103	99	2.0
			7 days reanalysis	102, 101, 101	101	
	Oilseed rape seed	0.1	initial analysis	77, 79, 74	77	10
			7 days reanalysis	88, 84, 88	87	
	Dry bean seed	0.1	initial analysis	87, 83, 83	84	8.0
			7 days reanalysis	94, 92, 89	92	

FL = Fortification Level; * Deviation [%] between mean initial analysis [%] and mean reanalysis [%]

Table A 11: Characteristics for the QuEChERS multi residue method 01207 used for validation of trans-deltamethrin, cis-deltamethrin and α-R-deltamethrin

Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ	
Limit of determination/quantification	LOQ = 0.01 mg/kg for each compound in each matrix	
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.	
	trans-deltamethrin (Quantifier MRM)	**trans-deltamethrin (Confirmatory MRM)**
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation: y = 0.0074 x - 0.0098, Correlation coefficient r: 0.9984 number of data points: 6	Individual calibration data is presented, calibration equation: y = 0.0025 x + 0.0009, Correlation coefficient r: 0.9985 number of data points: 6
	cis-deltamethrin (Quantifier MRM)	**trans-deltamethrin (Confirmatory MRM)**
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation: y = 0.0083 x - 0.005, Correlation coefficient r: 0.9995 number of data points: 6	Individual calibration data is presented, calibration equation: y = 0.0031 x – 0.0001, Correlation coefficient r: 0.9997 number of data points: 6
	α-R-deltamethrin (Quantifier MRM)	**α-R-deltamethrin (Confirmatory MRM)**
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation: y = 0.0143 x - 0.0086, Correlation coefficient r: 0.9996 number of data points: 6	Individual calibration data is presented, calibration equation: $y = 0.0053\ x + 9 \cdot 10^{-5}$, Correlation coefficient r: 0.9999 number of data points: 6
Calibration range	1.25 ng/mL to 100 ng/mL (corresponds to 0.05 – 0.4 mg/kg)	

Cross validation

A cross validation was performed in order to compare the extraction efficiency of the solvents used in a previous method (M-223823-01-1) with the solvent used in this study. This cross validation was performed for cis-deltamethrin. For this purpose, incurred residues were extracted from selected matrices. For cis-deltamethrin lettuce (head) and wheat straw was selected.

For BCS Method 01207, 5 g of sample material was weighed into 50 mL centrifuge tubes. The water content was adjusted to 5 g by addition of water. After adding 20 mL of acetonitrile (leading to an acetonitrile/water ratio of (4/1; v/v)), the sample material of straw was allowed to stand for 10 min. The centrifuge tube was closed and shaken manually for 2 min. After the addition of 6.5 g of a salt mixture (Mg2SO4/NaCl/Na3 citrate 2 H2O/Na2H citrate 6 H2O) (4/1/1/0.5, w/w/w/w), the sample was shaken for 1 min. Then the sample was centrifuged for 5 min at 4000 rpm. 1 mL of the upper acetonitrile phase was transferred into a 10-mL test tube and evaporated to dryness under a stream of nitrogen air (40°C). 2.50 mL of the working solution containing the internal standard was added. The final determination was performed with LC-MS/MS.

For the previous study, 5 g of sample material was weighed into 50 mL centrifuge tubes. After adding 20 mL of acetone/dichloromethane/hexane (1/1/1, v/v/v), the sample material of straw was allowed to stand for 10 min. The centrifuge tube was closed and shaken manually for 2 min, then the sample was centrifuged for 5 min at 4000 rpm. 1 mL of the extract was transferred into a 10-mL test tube and evaporated to dryness

under a stream of nitrogen air (40°C). 2.50 mL of the working solution containing the internal standard was added. The final determination was performed with LC-MS/MS.

Each sample was analysed in triplicate and measured with solvent calibration curves and internal standards for cis-deltamethrin. The results of the cross validation are expressed as average residues of three single extractions.

It should be noted that the extraction steps were compared but not the complete analytical residue methods. Aliquotations and mode of calibrations were kept identical in order to compare the solvents and not any other differences of the methods.

For cis-deltamethrin a slightly higher residue was extracted in lettuce head with method BCS-1207 but not for wheat straw. The latter matrix is considered as more difficult due to lower water content and higher matrix loading of the extracts.

Table A 12: Cross validation results for cis-deltamethrin

Sample Material	Method applied	Rounded residue found (mg/kg)	Average (mg/kg)	RSD (%)
Lettuce head	BCS Method 01207 Acetonitril/water (4/1,v/v)	0.43	0.42	4.2
		0.42		
		0.40		
	Study No. SYN/0270 / DART No. M-223823-01-1 Acetone/CH2Cl2/Hexane (1/1/1,v/v/v)	0.32	0.31	9.2
		0.34		
		0.28		
Wheat straw	BCS Method 01207 Acetonitril/water (4/1,v/v)	0.18	0.17	4.6
		0.17		
		0.16		
	Study No. SYN/0270 / DART No. M-223823-01-1 Acetone/CH2Cl2/Hexane (1/1/1,v/v/v)	0.18	0.19	5.3
		0.18		
		0.20		

The tests indicated similar extraction efficiences for acetonitrile/water (4/1, v/v) and the solvents applied in previous methods.

Conclusion
The QuEChERS multi residue method 01207 was validated for the determination deltamethrin (trans, cis and α-R isomers) and other analytes in/on plant materials by conducting recovery experiments with a broad number of plant matrices (commodity groups with high water, high acid, high protein and high oil content). All results are in accordance with the criteria set by the guideline SANCO/3029/99 rev. 4 with the minor exception of the precision data. For some matrices only four instead of five determinations per fortification level were performed for the analytes presented here. However, this deviation can be regarded as acceptable due to the fact that the recoveries for the confirmatory MRM are also presented. All values are within the acceptable ranges and clearly demonstrate sufficient precision. Therefore, the analytical method 01207 can be seen as fit for purpose in order to determine residues of deltamethrin (trans, cis and α-R isomers) in/on plant materials at an LOQ of 0.01 mg/kg.

A 2.1.1.1.2.2 Method validation for additional plant matrices

Comments of zRMS:	The study of Lakaschus, S.; Gizler, A.; 2017 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The analytical method BCS 01207 was validated for the determination of flupyradifurone in/on samples of tomato (fruit), wheat (green material), onion (bulbs), grape (bunches), wheat (grain), potato (tuber), peas (dry peas) and oilseed rape (seeds).* *The LOQ of BCS Method 01207 is defined as 0.01 mg/kg as validated in study S10-00279.* *This analyte not validated within study S10-00279 were validated within this study (reduced validation set – 5 samples fortified at 1.0 mg/kg).*

	LOQ: 1 mg/kg. *Mean recoveries were within the 70 - 110% range. The RSD values were well below 20%.* *Amendment No. 3 is written to provide additional information for the validation of flupyradifurone* *1. On request of the sponsor a full scan spectrum and the product ion spectra of flupyradifurone are added to the report.* *2. On request of the sponsor the linearity ranges for flupyradifurone are expressed as mass fractions of the original sample in mg/kg and the percentage of the fortification level at the lower and upper level is calculated for the used linearity curves of flupyradifurone.* *3. Starting 2017-01-01 the name of the sponsor changed.* *Accepted.*

Reference:	**KCP 5.1/23**
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 data generation method 01207 was validated for the determination of residues of deltamethrin and its isomers, alpha-*R*-deltamethrin and trans-deltamethrin in tomato fruit, wheat green material and dry peas within the storage stability study S13-03307 (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1). For the additional plant matrices relevant to this study but not included in the original validation, a reduced set of additional validation recoveries was analysed within this study. For the analysis of deltamethrin and its isomers, alpha-R-deltamethrin and trans-deltamethrin the LOQ of the original method was selected (= 0.01 mg/kg).

For the analysis of deltamethrin, the water content of different sample materials was adjusted to 5 g followed by the addition of acetonitrile, leading to a acetonitrile/water ratio of (4/1, v/v) followed by shaking. Thereafter the samples were left to soak under solvent for 15 minutes. After shaking 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 followed by shaking again and centrifugation. A defined aliquot of 1.0 mL of the acetonitrile phase was transferred into a test tube and blown down to dryness in a gentle stream of nitrogen at 40°C. The remainder was dissolved in 1.0 mL of the internal standard in acetonitrile / 10mM ammoniumformate (9/1, v/v). This solution was used for the LC-MS/MS detection.

The MS/MS instrument was operated in the Multiple Reaction Monitoring mode (MRM). One MRM transition was monitored for quantification, m/z 523 → 281, a second one for confirmation, m/z 529 → 283, for all analytes.

Results and discussions

The validation described within the storage stability study S13-03307 using method 01207 was performed on tomato, fruit, wheat, green material, and peas, dried. Recovery rates were determined at the fortification level of 0.1 mg/kg for tomato, fruit and wheat, green material and at 0.04 mg/kg for peas, dried. Mean recoveries were within the 70 - 110% range. The RSD values were well below 20%. The results are summarized in the table below.

Table A 13: Recovery results from method validation of deltamethrin residues using the analytical method 01207

Matrix	Fortification level (mg/kg)	Single recoveries (%)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin						
Tomato, fruit	0.1	93; 93; 96; 95; 93	5	94	1.5	m/z 523 → 281
	0.1	94; 90; 93; 91; 94	5	92	2.0	m/z 529 → 283
Wheat, green material	0.1	84; 80; 85; 89; 92	5	86	5.4	m/z 523 → 281
	0.1	85; 80; 85; 88; 87	5	85	3.6	m/z 525 → 283
Peas, dried	0.04	85; 87; 85; 77; 83	5	83	4.6	m/z 523 → 281
	0.04	87; 85; 83; 77; 81	5	83	4.7	m/z 529 → 283
alpha-R-deltamethrin						
Tomato, fruit	0.1	97; 93; 94; 94; 95	5	95	1.6	m/z 523 → 281
	0.1	96; 96; 97; 98; 91	5	96	2.8	m/z 529 → 283
Wheat, green material	0.1	84; 86; 90; 92; 93	5	89	4.4	m/z 523 → 281
	0.1	84; 86; 89; 91; 89	5	88	3.2	m/z 525 → 283
Peas, dried	0.04	85; 83; 85; 79; 84	5	83	3.0	m/z 523 → 281
	0.04	87; 85; 83; 78; 83	5	83	4.0	m/z 529 → 283
trans-deltamethrin						
Tomato, fruit	0.1	91; 99; 99; 99; 91	5	96	4.6	m/z 523 → 281
	0.1	94; 103; 98; 99; 94	5	98	3.9	m/z 529 → 283
Wheat, green material	0.1	85; 84; 89; 90; 94	5	88	4.6	m/z 523 → 281
	0.1	83; 86; 89; 91; 96	5	89	5.6	m/z 525 → 283
Peas, dried	0.04	85; 85; 88; 78; 85	5	84	4.4	m/z 523 → 281
	0.04	84; 81; 81; 76; 86	5	82	4.6	m/z 529 → 283

Table A 14: Characteristics for the analytical method 01207 used for validation of residues in different plant commodities

	Deltamethrin	alpha-*R*-deltamethrin	trans-deltamethrin
Specificity	blank value < 30% of the respective LOQ mass spectra are presented within the report S13-03307		
Calibration (type, number of data points)	1/x weighted linear regression individual calibration data and calibration line equation presented; number of data points ≥5; r ≥ 0.99		
Calibration range	Accepted calibration range in concentration units : Tomato fruit, wheat green material: 2.5-100µg/L corresponding calibration range in mass ratio units for the sample: 0.01-0.4 mg/kg Dry peas: 1.0-20 µg/L corresponding calibration range in mass ratio units for the sample: 0.004-0.08 mg/kg	Accepted calibration range in concentration units : Tomato fruit, wheat green material: 2.5-100µg/L corresponding calibration range in mass ratio units for the sample: 0.01-0.4 mg/kg Dry peas: 1.0-20 µg/L corresponding calibration range in mass ratio units for the sample: 0.004-0.08 mg/kg	Accepted calibration range in concentration units : Tomato fruit, wheat green material: 2.5-100µg/L corresponding calibration range in mass ratio units for the sample: 0.01-0.4 mg/kg Dry peas: 1.0-20 µg/L corresponding calibration range in mass ratio units for the sample: 0.004-0.08 mg/kg
Assessment of matrix effects is presented	Matrix effects are compensated by using matrix-matched standards		
Limit of quantification	LOQ: 0.01 mg/kg	LOQ: 0.01 mg/kg	LOQ: 0.01 mg/kg

Conclusion
The data collection method 01207 (based on QuEChERS) meets all necessary performance requirements to determine residues of deltamethrin and its isomers alpha-R-deltamethrin and trans-deltamethrin in tomato fruit, wheat, green material and dry peas (pulses) with a limit of quantification of 0.1 mg/kg (spiking level of the study). The exception according to SANCO/3029/99 rev. 4 is that recoveries were only performed at 1 fortification level. Despite this exception, all data presented confirmed the accuracy and linearity of this method for the determination of FPF and its metabolites according to SANCO/3029/99 rev. 4. Therefore, it can be considered as fit for purpose with regard to the presented stability study.

A 2.1.1.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.1)

A 2.1.1.2.1 Analytical method 01127 for the determination of deltamethrin in blood

A 2.1.1.2.1.1 Method validation

Comments of zRMS:	See point A 2.1.2.3.1.1

Reference:	**KCP 5.1/24**
Title:	Analytical method 01127 for the determination of cyfluthrin and deltamethrin in blood by HPLC-MS/MS
Report:	Krebber, R.; 2009; MR-08/176; M-348630-01-1
Authority registration No:	
Guideline(s):	SANCO/825/00 rev. 7 of March 17, 2004; BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998; EU: 96/46/EC amending Council Directive 91/414/EEC of 16 July 1996
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Please refer to section A2.1.2.3.1 for the respective summary of this method as this method was used in support of pre- as well as post-authorization method.

A 2.1.1.3 Description of analytical methods for the determination of residues in support to environmental fate studies (KCP 5.1)

No new or additional studies have been submitted

A 2.1.1.4 Description of analytical methods for the determination of residues in support to toxicological studies (KCP 5.1)

No analytical methods have been submited in support to toxicological studies.

A 2.1.1.5 Description of analytical methods for the determination of residues in support of operator, worker, resident and bystander exposure studies (KCP 5.1)

Not relevant. No operator, worker, resident and bystander exposure studies performed.

A 2.1.1.6 Description of analytical methods for the determination of residues in ecotoxicology studies (KCP 5.1)

A 2.1.1.6.1 Analytical method in support of the study M-199816-01-1

A 2.1.1.6.1.1 Method validation

Comments of zRMS:	The study of xxx 2001 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *Chemical analysis of the freshly prepared and aged (96 hours old) test solutions was performed for the metabolite of deltamethrin - AE F108565 using HPLC/UV.* *LOD: 0.31 mg/L in the aqueous sample* *LOQ: 0.51 mg/L in the aqueous sample* *No calibration curve was presented in the report.* *The repeatability precision is expressed by a mean CV of duplicate determinations < 20% for all concentration levels. The accuracy is within 80 - 120% recovery with a CV < 20%.* *The number of recovery replicates is outside the minimum acceptable of five (n ≥5) according to SANCO/3029/99 rev.4. Therefore, it cannot be concluded the method is validated for the determination of the concentration of AE F108565 (metabolite of deltamethrin) in test solutions.* *Remark:* *The method is not satisfactorily validated in accordance with SANCO/3029/99 rev. 4. for the determination of AE F108565 in test solution.*

Reference:	**KCP 5.1/25**
Title:	Acute toxicity to Oncorhynchus mykiss (rainbow trout) AE F108565 (metabolite of deltamethrin) substance, pure Code: AE F108565 00 1B99 0001
Report:	xxx 2001; C010902; M-199816-01-1
Authority registration No:	
Guideline(s):	OECD No. 203; US-EPA E § 72-1; EUC.1 US EPA OPPTS 850.1075
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	This method does not meet all required guideline criteria (SANCO/3029/99 rev 4).
Duplication (if vertebrate study):	No

Materials and methods

Chemical analysis of the freshly prepared and aged (96 hours old) test solutions was performed for the active ingredient AE F108565 using High Performance Liquid Chromatography with ultraviolet detection (HPLC/UV). The concentrations were analysed prior dilution.

The analytical method was validated during the development with respect to linearity range, selectivity, accuracy and the validity monitored during the study period with respect to LOD (limit of detection), LOQ (limit of quantification), precision and accuracy.

Results and discussions

The results of the method validation are summarized in the tables below.

Table A 15: Recovery results from method validation of AE F108565 using the analytical method

Matrix	Analyte	Fortification level (mg/L) (*n* = 2)	Mean recovery (%)	RSD (%)	Mean RSD (%)
Water	AE F108565	9.88	97.9	3.2	4.5
		17.78		0.6	
		31.62		1.1	
		55.33		1.2	
		98.80		1.8	

Table A 16: Characteristics for the analytical method used for validation of AE F108565 residues in water

	AE F108565
Specificity	The representative chromatograms show well-resolved peaks for AE F108565. The identity of the determined compound is established by cochromatography with the corresponding certified reference item.
Calibration (type, number of data points)	No calibration curve presented in the report.
Calibration range	linear working range of the detector response of 25 µg/L to 1000 µg/L.
Assessment of matrix effects is presented	No permanent interferences of the determined compound with matrix or solvent blanks above the LOQ were identified.
Limit of determination/quantification	LOD: 0.31 mg/L in the aqueous sample LOQ: 0.51 mg/L in the aqueous sample

Conclusion
The validation results and chromatograms demonstrate sufficient reliability of the method for the desired application: The lowest concentration level is above the LOQ and all concentration of the analyte solution prepared for HPLC are within the linearity range. The repeatability precision is sufficient expressed by a mean RSD of duplicate determinations < 20% for all concentration levels. The accuracy is within 80 - 120% recovery with a RSD < 20%. The specificity of the method is sufficient: The chromatograms display no matrix interference > LOQ of the determined compound of which the identity is established by co-chromatography with the corresponding certified reference item.

A 2.1.1.6.2 Analytical method in support of the study M-199793-01-1

A 2.1.1.6.2.1 Method validation

Comments of zRMS:	The study of xxx 2001 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *Chemical analysis of the freshly prepared and aged (48 hours old) test solution was performed for metabolite of deltamethrin (AE F108565) using HPLC/UV.* *LOD: 0.05 mg/L in the aqueous sample* *LOQ: 0.08 mg/L in the aqueous sample* *No calibration curve was presented in the report.* *The repeatability precision is expressed by a mean CV of duplicate determinations < 20% for all concentration levels. The accuracy is within 80 - 120% recovery with a CV < 20%.* *The number of recovery replicates is outside the minimum acceptable of five (n ≥5) according to SANCO/3029/99 rev.4.* *Remark:* *The method is not satisfactorily validated in accordance with SANCO/3029/99 rev. 4. for the determination of metabolite of deltamethrin (AE F108565) in test solution.*

Reference:	**KCP 5.1/26**
Title:	Acute toxicity to Daphnia magna (Waterflea) AE F108565 (Metabolite of deltamethrin) substance, pure Code: AE F108565 00 1B99 0001
Report:	xxx; 2001; C010889; M-199793-01-1
Authority registration No:	
Guideline(s):	OECD No. 202; US-EPA E § 72-2 EUC.2; US EPA OPPTS 850.1010
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	This method does not meet all required guideline criteria (SANCO/3029/99 rev 4).
Duplication (if vertebrate study):	

Materials and methods
Chemical analysis of the freshly prepared and aged (48 hours old) test solution was performed for the active

ingredient AE F108565 using High Performance Liquid Chromatography with ultraviolet detection (HPLC/UV). The concentrations were analysed prior dilution.
The analytical method was validated during the development with respect to linearity range, selectivity, accuracy and the validity monitored during the study period with respect to LOD (limit of detection), LOQ (limit of quantification), precision and accuracy.

Results and discussions
The results of the method validation are summarized in the tables below.

Table A 17: Recovery results from method validation of AE F108565 using the analytical method

Matrix	Analyte	Fortification level (mg/L) (*n* = 2)	Mean recovery (%)	RSD (%)	Mean RSD (%)
Water	AE F108565	0.99	94.0	7.4	0.4
		1.78		6.1	
		3.16		2.1	
		5.53		1.1	
		9.88		1.7	
		17.78		2.0	
		31.62		0.4	
		55.83		1.8	
		98.80		1.0	

Table A 18: Characteristics for the analytical method used for validation of AE F108565 residues in water

	AE F108565
Specificity	The representative chromatograms show well-resolved peaks for AE F108565. The identity of the determined compound is established by cochromatography with the corresponding certified reference item.
Calibration (type, number of data points)	No calibration curve presented in the report.
Calibration range	linear working range of the detector response of 25 µg/L to 1000 µg/L.
Assessment of matrix effects is presented	No permanent interferences of the determined compound with matrix or solvent blanks above the LOQ were identified. For the lowest concentration level of 1 mg/L nominal test item, matrix interferences lead to exceeding results (136.9 % of the nominal concentration) despite subtraction of the control blank value.
Limit of determination/quantification	LOD: 0.05 mg/L in the aqueous sample LOQ: 0.08 mg/L in the aqueous sample

Conclusion
The validation results and chromatograms demonstrate sufficient reliability of the method for the desired application: The lowest concentration level is above the LOQ and all concentrations of the analyte solution prepared for HPLC are within the linearity range. The repeatability precision is sufficient expressed by a mean RSD of duplicate determinations < 20% for all concentration levels. The accuracy is within 80 - 120% recovery with a RSD < 20%. The specificity of the method is sufficient: The chromatograms display no matrix interference > LOQ of the determined compound except for the lowest concentration level on day 2 and their identity is established by co-chromatography with the corresponding certified reference item.

A 2.1.1.6.3 Analytical method 00886

A 2.1.1.6.3.1 Method validation

Comments of zRMS:	Please refer to point A 2.1.2.5.2.1

Reference:	**KCP 5.1/27**
Title:	Analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS
Report:	xxx; 2005; C047388; M-248040-01-1
Authority registration No:	
Guideline(s):	--
Deviations:	--
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Please refer to section A 2.1.2.5.2 for the respective summary of this method as this method was used in support of pre- as well as post-authorization method.

A 2.1.1.6.4 Concurrent validation of the analytical method 00886 in support of the studies M-246137-01-2 and M-246173-01-1

Comments of zRMS:	The study of xxx; 2005 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and it has been accepted.

Reference:	**KCP 5.1/28**
Title:	Biological effects and fate of deltamethrin EW 015 in outdoor mesocosm ponds
Report:	xxx; HBF/BT 07; M-246137-01-2
Authority registration No:	
Guideline(s):	OECD Guidance Doc. "Freshwater Lentic Field Teste", 2004 (Draft) ; Guidance Doc. on Testing Procedures for Pesticides in Freshwater Microcosms (SETAC 1991) Community-Level Aquatic System Studies Interpretation Criteria (SETAC 2002)
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Reference:	**KCP 5.1/29**
Title:	Bioassay on the effects of Deltamethrin EW 015 on Gammarus pulex in mesocosm water
Report:	xxx; 2005; HBF/BT 08; M-246173-01-1
Authority registration No:	
Guideline(s):	OECD Guidance Doc. "Freshwater Lentic Field Teste", 2004 (Draft); Guidance Doc. on Testing Procedures for Pesticides in Freshwater Microcosms (SETAC 1991); Community-Level Aquatic System Studies Interpretation Criteria (SETAC 2002)
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Concurrent validation

During the course of both studies the water samples were analysed according to method 00886:

- "Method for the Determination of Deltamethrin in Surface Water by HPLC-MS/MS", xxx; M-248040-01-1.

The limit of quantitation for deltamethrin according to this method is 0.005 µg/L with direct injection of 250 µL of the sample. During the study M-246137-01-2, the method was validated concurrently with the

sample analyses of the study by evaluation of the standard injections. The validation results were also used for study M-246173-01-1.

Table A 19: Method validation for deltamethrin (standard injections)

Concentration [µg/L]		Peak area [area counts]		Retention time	
	n	Mean	RSD [%]	Mean [min]	RSD [%]
0.0059	14	0.036	15.6	1.65	0.6
0.059	14	0.300	3.3	1.68	<0.1
0.59	6	3.410	2.1	1.64	<0.1
0.0059	22	0.036	14.2	1.69	0.6
0.059	22	0.310	6.5	1.69	0.6
0.0059	18	0.080	25.0	1.70	1.2
0.059	18	0.350	5.7	1.73	0.6
0.0059	16	0.033	13.5	1.69	<0.1
0.059	16	0.320	3.1	1.67	0.6
0.0059	12	0.043	7.9	1.72	0.6
0.059	12	0.320	3.1	1.71	0.6
0.0052	13	0.049	8.0	1.71	0.6
0.052	13	0.460	2.2	1.70	0.6

Conclusion
The applicability of the HPLC-MS/MS method 00886 for the analysis of deltamethrin in water samples was tested. The data presented demonstrate that the method allows the determination of deltamethrin with satisfactory precision and repeatability and can be regarded as fit for purpose.

A 2.1.1.6.5 Analytical method 00877 (including amendment)

A 2.1.1.6.5.1 Method validation

Comments of zRMS:	Please refer to point A 2.1.2.4.1.1

Please refer to section A 2.1.2.4.1 for the respective summary of this method as this method was used in support of pre- as well as post-authorization method.

Reference:	**KCP 5.1/30**
Title:	Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS
Report:	xxx; C047210; M-247896-01-1
Authority registration No:	
Guideline(s):	--
Deviations:	--
GLP/GEP:	yes
Acceptability:	
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/31**
Title:	Analytical method 00877 for the determination of total residues of Deltamethrin (AE F032640) in/on soil and sediment by HPLC-MS/MS
Report:	xxx 00877; M-246580-02-1
Authority registration No:	
Guideline(s):	EC Guidance Document on Residue Analytical Methods, SANCO/825/00 rev.7 of March 17,2004 BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998 Commission Directive 96/46/EC amending Council Directive 91/414/EEC of 16 July 1996 US EPA OPPTS 835.6100, 835.6200
Deviations:	With the exception of recognised differences that exist between the GLP principles/standards of OECD and those FIFRA and JMAFF (for instance, authority granted agency inspectors).
GLP/GEP:	yes
Acceptability:	
Duplication (if vertebrate study):	

A 2.1.1.6.6 Concurrent validation of the analytical method 00877 in support of the study M-246137-01-2

Comments of zRMS:	The study of xxx.; 2005 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and it has been accepted.

Reference:	**KCP 5.1/28**
Title:	Biological effects and fate of deltamethrin EW 015 in outdoor mesocosm ponds
Report:	xxx; 2005; HBF/BT 07; M-246137-01-2
Authority registration No:	
Guideline(s):	OECD Guidance Doc. "Freshwater Lentic Field Teste", 2004 (Draft) ; Guidance Doc. on Testing Procedures for Pesticides in Freshwater Microcosms (SETAC 1991) Community-Level Aquatic System Studies Interpretation Criteria (SETAC 2002)
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Concurrent validation

The sediment samples were analysed according to the following method:

- - "Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS"; xxx; M-247896-01-1

The limit of quantitation (LOQ) of the method is 0.1 µg/kg for deltamethrin. The limit of detection (LOD) of the method is 0.03 µg/kg for deltamethrin.

Recovery experiments were conducted concurrently with the analysis of the samples. They were performed to verify the integrity of the analysed residues. These concurrent recoveries were performed with control sediment of the trial investigated in this study. For evaluation of the recovery experiments the measured residue concentrations in the recovery samples were corrected for the blank value in the respective control sample.

Table A 20: Concurrent recovery rates of deltamethrin during analysis of sediment samples using the analytical method 00877

Matrix	Fortification level (µg/kg)	Single Recovery (%)	Mean Recovery (%)
Control Sediment	0.1	122 / 94.9 / 108 / 99.7 / 104 / 95.6 / 95.0 / 88.3 / 98.0 / 91 .4 / 88.6 / 95.0 / 104 / 91.2 /107 / 88.9 / 82.9 / 102 / 89.4 / 103 / 93.5/91.1	98.0
	1.0	105/108/102/104/103/105/96.2 / 94.1 / 92.8 / 96.4 / 97.1 / 94.1 / 93.0 / 104 / 1 1 1 / 92.7 / 102 / 93.9 / 97.5 / 98.3 / 97.3/103/97.0/91.8/95.9	

Conclusion
The applicability of the HPLC-MS/MS method 00877 for the analysis of deltamethrin in sediment samples was tested. The data presented demonstrate that the method allows the determination of deltamethrin with satisfactory accuracy and can be regarded as fit for purpose.

A 2.1.1.6.7 Concurrent validation of the analytical method 00877 in support of the studies M-291818-01-1 and M-291885-02-1

Comments of zRMS:	The study of xxx has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and it has been accepted.

Reference:	**KCP 5.1/32**
Title:	Analysis of deltamethrin concentrations in sediment samples of ECT study no. P1MA
Report:	xxx; MR-07/297; M-291818-01-1
Authority registration No:	
Guideline(s):	not specified
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/33**
Title:	Deltamethrin EW 15 G: Acute and chronic effects to different life stages of the isopod Assellus aquaticus L in a natural water-sediment-system
Report:	xxx; P1MA; M-291885-02-1
Authority registration No:	
Guideline(s):	no guideline available
Deviations:	--
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Concurrent validation
The sediment samples were analysed according to the following method:

- - "Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS"; xxx; M-247896-01-1

The results of study M-291818-01-1 were used in support of the following study:

- - "Amendment No. 1 to the Report: Deltamethrin EW 15 G: Acute and chronic Effects to Different Life Stages of the Isopod *Asellus aquaticus* L. in a Natural Water-Sediment System"; xxx; 2007a; Report No.: P1MA; Doc. No.: M-291885-02-1.

The limit of quantitation (LOQ) of the method is 0.1 µg/kg for deltamethrin. The limit of detection (LOD) of the method is 0.03 µg/kg for deltamethrin.

Recovery experiments were conducted concurrently with the analysis of the samples. They were performed to verify the integrity of the analysed residues.

Table A 21: Concurrent recovery rates of deltamethrin during analysis of sediment samples using the analytical method 00877

Matrix	Fortification level (µg/kg)	Single Recovery (%)	Mean Recovery (%) ± RSD (%)
Sediment	0.1	104	106 ± 2.7
		109	
		104	
	1.0	101	100 ± 1.2
		99	
		99	

Conclusion
The applicability of the HPLC-MS/MS method 00877 for the analysis of deltamethrin in sediment samples was tested. The data presented demonstrate that the method allows the determination of deltamethrin with satisfactory precision and repeatability and can be regarded as fit for purpose.

A 2.1.1.6.8 Analytical method 00886/M001

A 2.1.1.6.8.1 Method validation

Comments of zRMS:	Please refer to point A 2.1.2.5.3.1

Reference:	**KCP 5.1/34**
Title:	Modification M001 of analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS
Report:	Krebber, R.; Braune, M.; 2007; 00886/M001; M-291746-01-1
Authority registration No:	
Guideline(s):	US EPA OPPTS 835.6100, 835.6200
Deviations:	not specified
GLP/GEP:	no
Acceptability:	yes
Duplication (if vertebrate study):	

Please refer to section A 2.1.2.5.3 for the respective summary of this method as this method was used in support of pre- as well as post-authorization method.

A 2.1.1.6.9 Concurrent validation of the analytical method 00886/M001 in support of the studies M-291818-01-1 and M-291885-02-1

Comments of zRMS:	The study of Krebber, R.; Braune, M.; 2007 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and it has been accepted.

Reference:	**KCP 5.1/35**
Title:	Analysis of deltamethrin concentrations in water samples of ECT study no. P1MA
Report:	Krebber, R.; Braune, M.; 2007; MR-07/295; M-291848-01-1
Authority registration No:	
Guideline(s):	not specified
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/33**
Title:	Deltamethrin EW 15 G: Acute and chronic effects to different life stages of the isopod Assellus aquaticus L in a natural water-sediment-system
Report:	xxx; 2007; P1MA; M-291885-02-1
Authority registration No:	
Guideline(s):	no guideline available
Deviations:	--
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Concurrent validation

During the course of the study the water samples were analysed according to method 00886/M001:

- - "Modification M001 of analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS", R. Krebber and M. Braune, Bayer CropScience AG Report No. MR-07/296; M-291746-01-1.

The results of study M-291848-01-1 were used in support of the following study:

- - "Amendment No. 1 to the Report: Deltamethrin EW 15 G: Acute and chronic Effects to Different Life Stages of the Isopod *Asellus aquaticus* L. in a Natural Water-Sediment System"; xxx; 2007a; Report No.: P1MA; Doc. No.: M-291885-02-1.

The analytical method was validated within the present study at fortification levels of 2 and 10 ng/L using the internal standard procedure. The limit of quantitation (LOQ) of the method is 2 ng/L. The limit of detection (LOD) of the method was 0.3 x LOQ for deltamethrin in water. Linearity was determined in the concentration range of 2 to 100 ng/L. The correlation coefficient was 0.9997. 5 concentrations measured.

Table A 22: Method validation for deltamethrin in test water using the analytical method 00886/M001

Sample concentration [ng/L]	Peak area [area counts]			Retention time	
	Single values	Mean	RSD [%]	Mean [min]	RSD [%]
2	0.02137 / 0.02233 / 0.02264 / 0.02148 / 0.02217 / 0.02156 / 0.02109 / 0.02224 / 0.02251 / 0.02195	0.02193	2.4	8.14	0.2
10	0.09592 / 0.096955 / 0.09126 / 0.1002 / 0.09664 / 0.09883 / 0.09467 / 0.09578 / 0.09221 / 0.09051	0.09530	3.3	8.12	0.1

Conclusion

The applicability of the HPLC-MS/MS method 00886/M001 for the analysis of deltamethrin in water samples was tested. The data presented demonstrate that the method allows the determination of deltamethrin with satisfactory precision and repeatability and can be regarded as fit for purpose.

A 2.1.1.6.10 Analytical method in support of the study M-256605-01-1

A 2.1.1.6.10.1 Method validation

Comments of zRMS:	The validation data were not presented in the study report, so the method is not acceptable.

Reference:	**KCP 5.1/36**
Title:	Effects of Deltamethrin EW 15 on rainbow trout in aquatic outdoor microcosm enclosures
Report:	xxx.1; M-256605-01-1
Authority registration No:	
Guideline(s):	OECD Guidance Document "Freshwater Lentic Tests", 2004 (Draft); Guidance Document on Testing Procedures for Pesticides in Freshwater Mesocosm (SEATC-Europe Workshop, July 1991)
Deviations:	none
GLP/GEP:	yes
Acceptability:	no
Duplication (if vertebrate study):	No

Materials and methods
The aim of the study was to assess the effects of repeated applications (3x) of the insecticide Deltamethrin EW 15 G on growth and survival of juvenile rainbow trout under outdoor field conditions. No validation data were presented in the study report.

Nevertheless, to show that the method is suitable for the determination of deltamethrin in water samples, recovery data of test concentration are presented.

After sampling, hexane was added and the bottles were shaken for 0.5 h. As much hexane as possible was removed and, if necessary, the samples were further concentrated by evaporation of the hexane fraction. The residues were then redissolved into an appropriate amount of hexane. The concentration of the test substance in the hexane extracts was measured through gas chromatography using an electron-capture detector. The limit of quantification corresponds to 1 µg/L in the hexane extracts.

Results and discussions
On each day that water samples were taken from the enclosures recovery of the test substance was determined in triplicate at 100 ng a.i./L and in triplicate at 1000 ng a.i./L. One of the samples spiked at 1000 ng a.i./L was lost.

For the 39 determinations at 100 µg/L the observed recoveries ranged from 76% - 154%, resulting in a mean recovery of 109 ± 22%. For the 38 determinations at 1000 µg/L the observed recoveries ranged from 81% - 150%, resulting in a mean recovery of 106 ± 20%.
Reported concentrations are not corrected for recovery.

Table A 23: Concentration of test substance in spraying solutions applied to enclosures; concentrations are not corrected for recoveries.

Enclosure no.	Nominal treatment level (ng l^{-1})	Nominal[A] concentration of test substance in spraying solution for each of the three applications (µg l^{-1})			Measured[B] concentration of test substance in spraying solution for each of the three applications (µg l^{-1})		
		1	2	3	1	2	3
4	0	0.00	0.00	0.00	0.00	0.00	0.00
11	0	0.00	0.00	0.00	0.00	0.00	0.00
5	125	26.0	24.4	26.7	21.9	13.7	21.0
12	125	25.9	24.4	26.8	23.9	14.7	16.7
8	250	51.9	48.8	53.6	45.7	47.2	49.1
9	250	51.8	49.1	53.5	43.7	39.9	41.9
7	500	105	98.9	108	93.1	88.2	84.5

10	500	105	98.5	109	100	85.8	106
2	1000	341	201	219	231	185	206
6	1000	211	198	220	218	198	207

[A] Calculated from the weighed amount of test substance used to prepare solutions.
[B] Mean of duplicate measurement.

Conclusion
Despite the fact that not all validation parameters according to SANCO/3029/99 rev. 4 were met, the method can nevertheless be regarded as fit for purpose with regard to the present study.

A 2.1.1.6.11 Analytical method in support of the study M-679497-01-1

A 2.1.1.6.11.1 Method validation

Comments of zRMS:	The analytical method has been validated to determine concentration of deltamethrin in test water samples. The limit of quantitation (LOQ) for deltamethrin was 1.6 µg test item/L (corresponding to nominal 0.0148 µg a.i./L). The average recoveries were within the acceptable range of 70 – 110% with the RSD values below 20%. The method is acceptable.

Reference:	**KCP 5.1/37**
Title:	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to rainbow trout (Oncorhynchus mykiss) in a 96-hour semi-static test
Report:	xxx; EBRV0196; M-679497-01-1
Authority registration No:	
Guideline(s):	OECD Guideline for Testing of Chemicals, Section 2, No. 203, "Fish, Acute Toxicity Test", June 18, 2019 OECD Series on Testing and Assessment, No. 23, "Guidance Document on Aqueous-phase Aquatic Toxicity Testing of Difficult Test Chemicals", 2nd Ed., February 08, 2019 EPA Guideline 712-C-16-007: OCSPP 850.1075, "Freshwater and Saltwater Fish Acute Toxicity Test", October 2016 SANCO/3029/99 rev.4 11/07/00: Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A; Section 4) and Annex III (part A; Section 5) of directive 91/414
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Materials and methods
The concentrations of the active ingredient Deltamethrin of the test item Deltamethrin + Flupyradifurone EC85 (10+75 g/L) were measured in two of the four taken undiluted test medium and control samples after extraction.

The analytical method for the determination of the analyte is based on liquid/liquid-extraction followed by analysis via GC with MS/MS detection (electron ionization, MRM m/z: 181.2 → 152.1 used for quantification).

A sample volume of 30 mL was extracted twice with 2.5 mL Dichloromethane for 20 minutes on an overhead shaker. The resulting Dichloromethane extracts were unified and diluted further with matrix-matched dichloromethane to match the calibration range, if necessary.

Results and discussions
Recovery rates were determined at fortification levels of 1.6, 3.5 and 200 µg test item/L with 6 replicates

each. The recovery experiments were conducted by fortification of untreated control samples with defined amounts of the analytes prior to analysis.

The overall mean recovery and mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 24: Recovery rates and precision results (repeatability) of Deltamethrin

	Concentration					
Sample description	**Nominal [µg test item/L]**	**Found [µg a.i./L][1]**	**DF**	**Concentration calculated [µg a.i./L][1]**	**Corrected nominal [µg a.i./L][1]**	**Recovery [%][1]**
Anayltical Blank	0	<LOD	0.1667	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	0.1667	n.a.	0.000	n.a.
Fortified Sample	1.6	0.083	0.1667	0.014	0.015	93
	1.6	0.082	0.1667	0.014	0.015	92
	1.6	0.076	0.1667	0.013	0.015	85
	1.6	0.078	0.1667	0.013	0.015	88
	1.6	0.080	0.1667	0.013	0.015	90
	1.6	0.070	0.1667	0.012	0.015	79
Mean value (n = 6):						**88**
RSD (n = 6):						**6**
Fortified Sample	200	2.353	0.8335	1.962	1.864	105
	200	2.176	0.8335	1.814	1.864	97
	200	2.422	0.8335	2.019	1.864	108
	200	2.572	0.8335	2.144	1.855	116
	200	2.534	0.8335	2.112	1.855	114
	200	2.561	0.8335	2.135	1.855	115
Mean value (n = 6):						**109**
RSD (n = 6):						**7**
Overall mean value (n = 12):						**98**
RSD (n = 12):						**13**

1 The tabulated results represent rounded results calculated on the exact raw data; a.i. = active ingredient (= Deltamethrin); LOD: Limit of Detection = 0.02 µg a.i./L; n.a.: not applicable; DF: Dilution factor; RSD: Relative Standard Deviation

Table A 25: Characteristics for the analytical method used for validation of Deltamethrin

	Deltamethrin
Specificity	GC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 46317 x – 1179, Correlation coefficient r: 1.0000, number of data points: 7
Calibration range	0.06 µg/L – 5 µg/L
Limit of determination/quantification	LOQ = 0.0148 µg a.i./L
Assessment of matrix effects is presented	No effects observed

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of Deltamethrin in test water samples via GC-MS/MS.

A 2.1.1.6.12 Analytical method in support of the study M-686370-01-1

A 2.1.1.6.12.1 Method validation

Comments of zRMS:	The analytical method has been validated to determine concentration of deltamethrin in test water samples. The limit of quantitation (LOQ) for deltamethrin was 0.3 µg test item/L (corresponding to

	nominal 0.003 µg a.i./L). The average recoveries were within the acceptable range of 70 – 110% with the RSD values below 20%. The method is acceptable.

Reference:	**KCP 5.1/38**
Title:	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to Daphnia magna in a semi-static 48-hour immobilisation test - Final report -
Report:	xxx; EBRV0195; M-686370-01-1
Authority registration No:	
Guideline(s):	− EPA Guideline 712-C-16-013: OCSPP 850.1010, "Aquatic Invertebrate Acute Toxicity Test, Freshwater Daphnids" October 2016 − OECD Guideline for Testing of Chemicals No. 202: "Daphnia sp., Acute Immobilisation Test" adopted April 13, 2004 − OECD Series on Testing and Assessment, No. 23, "Guidance Document on Aqueous-phase Aquatic Toxicity Testing of Difficult Test Chemicals", 2nd Ed., February 08, 2019 − Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009, Official Journal of the European Union No. L 309: 1 – 50 − SANCO/3029/99 rev.4 11/07/00: Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A; Section 4) and Annex III (part A; Section 5) of directive 91/414
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The concentrations of the active ingredient Deltamethrin of the test item Deltamethrin + Flupyradifurone EC85 (10+75 g/L) were measured in two of the four taken undiluted test medium and control samples after extraction.

The analytical method for the determination of the analyte is based on liquid/liquid-extraction followed by analysis via GC with MS/MS detection (electron ionization, MRM m/z: 181.2 → 152.1 used for quantification).

A sample volume of 30 mL was extracted twice with 3 mL Dichloromethane for 20 minutes on an overhead shaker. The resulting Dichloromethane extracts were unified, evaporated and then re-dissolved in 1 mL Dichloromethane by intense vortexing. The samples were diluted further with matrix-matched dichloromethane to match the calibration range, if necessary.

The fortification level of 0.3 µg test item/L (LOQ level) was validated within the study EBRV0194 (M-686369-01-1). The results are reported in the present study since the fortification samples were prepared in the same test water as in the present study (Elendt/M4 medium) and were prepared and analysed with the same method.

Results and discussions

Recovery rates were determined at fortification levels of 0.3 µg test item/L with 5 replicates and 15 µg test item/L with 6 replicates. The recovery experiments were conducted by fortification of untreated control samples with defined amounts of the analytes prior to analysis.

The overall mean recovery and mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 26: Recovery rates and precision results (repeatability) of Deltamethrin

	Concentration					
Sample	**Nominal [µg**	**Found [µg**	**DF**	**Concentration**	**Corrected**	**Recovery**

description	test item/L]	a.i./L][1]		calculated [µg a.i./L][1]	nominal [µg a.i./L]	[%][1]
Anayltical Blank	0	<LOD	0.0333	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	0.0333	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	0.0333	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	0.0333	n.a.	0.000	n.a.
Fortified Sample	0.30	0.063	0.0333	0.002	0.00279	75
	0.30	0.062	0.0333	0.002	0.00279	74
	0.30	0.059	0.0333	0.002	0.00280	71
	0.30	0.067	0.0333	0.002	0.00280	79
	0.30	0.078	0.0333	0.003	0.00280	93
Mean value (n = 5):						**78**
RSD (n = 5):						**11**
Fortified Sample	15	3.501	0.0333	0.117	0.140	83
	15	3.577	0.0333	0.119	0.140	85
	15	3.238	0.0333	0.108	0.140	77
	15	2.929	0.0333	0.098	0.140	70
	15	2.651	0.0333	0.088	0.140	63
	15	3.508	0.0333	0.117	0.140	83
Mean value (n = 6):						**77**
RSD (n = 6):						**11**
Overall mean value (n = 11):						**78**
RSD (n = 11):						**11**

1 The tabulated results represent rounded results calculated on the exact raw data; a.i. = active ingredient (= Deltamethrin); LOD: Limit of Detection = 0.0327 µg a.i./L; n.a.: not applicable; DF: Dilution factor; RSD: Relative Standard Deviation;

Table A 27: Characteristics for the analytical method used for validation of Deltamethrin

	Deltamethrin
Specificity	GC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Two calibration curves were used to cover the wide calibration range of 0.05 – 7.5 µg reference item with a sufficient linearity. Individual calibration data is presented, calibration equation (1/x weighted) for lower calibration curve: y = 105268 x + 2516, Correlation coefficient r: 0.9994, number of data points: not given Individual calibration data is presented, calibration equation (1/x weighted) for upper calibration curve: y = 103553 x + 3619, Correlation coefficient r: 1.0000, number of data points: 9
Calibration range	lower calibration curve: 0.05 µg/L – 0.5 µg/L upper calibration curve: 0.05 µg/L – 7.5 µg/L
Limit of determination/quantification	LOQ = 0.003 µg a.i./L
Assessment of matrix effects is presented	No effects observed

Conclusion

The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of Deltamethrin in test water samples via GC-MS/MS.

A 2.1.1.6.13 Analytical method in support of the study M-686369-01-1

A 2.1.1.6.13.1 Method validation

Comments of zRMS:	The analytical method has been validated to determine concentration of deltamethrin in test water samples. The limit of quantitation (LOQ) for deltamethrin was 0.3 µg test item/L (corresponding to nominal 0.003 µg a.i./L). The average recoveries were within the acceptable range of 70 – 110% with the RSD values below 20%. The method is acceptable.

Reference:	**KCP 5.1/39**
Title:	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to larvae of Chironomus riparius in a semi-static 48-hour immobilisation test - Final report -
Report:	xxx; EBRV0194; M-686369-01-1
Authority registration No:	
Guideline(s):	− OECD Guideline for Testing of Chemicals 235: "Chironomus sp., Acute Immobilisation Test" adopted July 28, 2011 − OECD Series on Testing and Assessment, No. 23, "Guidance Document on Aqueous-phase Aquatic Toxicity Testing of Difficult Test Chemicals", 2nd Ed., February 08, 2019 − SANCO/3029/99 rev.4 11/07/00: Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A; Section 4) and Annex III (part A; Section 5) of directive 91/414
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The concentrations of the active ingredient Deltamethrin of the test item Deltamethrin + Flupyradifurone EC85 (10+75 g/L) were measured in all of the four taken undiluted test medium and control samples after extraction.

The analytical method for the determination of the analyte is based on liquid/liquid-extraction followed by analysis via GC with MS/MS detection (electron ionization, MRM m/z: 181.2 → 152.1 used for quantification).

A sample volume of 30 mL was extracted twice with 3 mL Dichloromethane for 20 minutes on an overhead shaker. The resulting Dichloromethane extracts were unified, evaporated and then re-dissolved in 1 mL Dichloromethane by intense vortexing. The samples were diluted further with matrix-matched dichloromethane to match the calibration range, if necessary.

Results and discussions
Recovery rates were determined at fortification levels of 0.3 and 30 µg test item/L with 5 replicates each. The recovery experiments were conducted by fortification of untreated control samples with defined amounts of the analytes prior to analysis.

The overall mean recovery and mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 28: Recovery rates and precision results (repeatability) of Deltamethrin

	Concentration					
Sample description	**Nominal [µg test item/L]**	**Found [µg a.i./L][1]**	**DF**	**Concentration calculated [µg a.i./L][1]**	**Corrected nominal [µg a.i./L]**	**Recovery [%][1]**

Anayltical Blank	0	<LOD	0.0333	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	0.0333	n.a.	0.000	n.a.
Fortified Sample	0.30	0.063	0.0333	0.002	0.00279	75
	0.30	0.062	0.0333	0.002	0.00279	74
	0.30	0.059	0.0333	0.002	0.00280	71
	0.30	0.067	0.0333	0.002	0.00280	79
	0.30	0.078	0.0333	0.003	0.00280	93
Mean value (n = 5):						**75**
RSD (n = 5):						**4**
Fortified Sample	30	3.357	0.0666	0.224	0.279	80
	30	3.857	0.0666	0.257	0.279	92
	30	3.693	0.0666	0.246	0.280	88
	30	2.539	0.0666	0.169	0.280	60
	30	2.867	0.0666	0.191	0.280	68
Mean value (n = 5):						**78**
RSD (n = 5):						**17**
Overall mean value (n = 10):						**78**
RSD (n = 10):						**14**

2 The tabulated results represent rounded results calculated on the exact raw data; a.i. = active ingredient (= Deltamethrin); LOD: Limit of Detection = 0.0155 µg a.i./L; n.a.: not applicable; DF: Dilution factor; RSD: Relative Standard Deviation;

Table A 29: Characteristics for the analytical method used for validation of Deltamethrin

	Deltamethrin
Specificity	GC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Two calibration curves were used to cover the wide calibration range of 0.05 – 7.5 µg reference item with a sufficient linearity. Individual calibration data is presented, calibration equation (1/x weighted) for lower calibration curve: y = 79930 x – 683, Correlation coefficient r: 0.9994, number of data points: 8 Individual calibration data is presented, calibration equation (1/x weighted) for upper calibration curve: y = 74847 x + 1994, Correlation coefficient r: 0.9994, number of data points: not given
Calibration range	lower calibration curve: 0.05 µg/L – 2.5 µg/L upper calibration curve: 0.05 µg/L – 7.5 µg/L
Limit of determination/quantification	LOQ = 0.003 µg a.i./L
Assessment of matrix effects is presented	No effects observed

Conclusion

The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of Deltamethrin in test water samples via GC-MS/MS.

A 2.1.1.6.14 Analytical method 01347

A 2.1.1.6.14.1 Method validation

Comments of zRMS:	The study of Schoening, R.; Willlmes, J.; 2013 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below:

	The analytical method 01347 was validated for the determination of deltamethrin residues in/on bees (insects), flowers/blossoms, green material, honey/nectar, pollen and wax by HPLC-ESI-MS/MS using stable isotopic labelled internal standards. *This method determines the sum of deltamethrin isomers.* *The limit of quantitation (LOQ) for deltamethrin is 0.01 mg/kg in all matrices tested.* *The limit of determination (LOD) was estimated for the linearity and the control chromatograms and was set to 0.0025 mg/kg.* *Mean recoveries for each fortification level and the overall mean recovery were within the 70 - 110% range for all matrices. Relative standard deviations were below 20% for all analytes and sample materials.* *Two MRM transitions were monitored for the analyte and each matrix tested m/z 523 → 281 for the MRM and m/z 525 → 283 for the MRM.* *The results of the method validation were confirmed using a MRM transition for confirmation.* *The method meets all guideline criteria (SANCO/825/00/rev. 8.1 and SANCO/3029/99 rev. 4) to determine residues of deltamethrin in/on bees (insects), flowers/blossoms, green material, honey/nectar, pollen and wax at 0.01 mg/kg with exception of wax samples at the LOQ level for the 2nd MRM.* *The study is acceptable.*

Reference:	**KCP 5.1/40**
Title:	Residue analytical method 01347 for the determination of residues of deltamethrin by HPLC with electrospray and MS/MS - detection
Report:	Schoening, R.; Willlmes, J.; 2013; MR-012/067; M-444791-01-1
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 European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, 11/07/00 Guidance document on residue analytical methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010 US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method
Deviations:	not specified
GLP/GEP:	~~No~~ yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method 01347 was developed for the determination of deltamethrin by HPLC with electrospray and MS/MS-detection.

Deltamethrin was extracted from the sample material using a mixture of dichloromethane/ n-hexane/ acetone (1/1/1, v/v/v). After filtration an aliquot of this solution was evaporated to the aqueous remainder and clean-up on a ChemElut® column or by liquid/ liquid partition for wax. After elution of the residues with dichloromethane or separation with acetonitrile the extract was evaporated to dryness and re-dissolved in water/ acetonitrile (1/9, v/v) + 10 mmol ammonium formate/L + 100 µL/L formic acid. The residues were quantified by reversed phase HPLC with ESI MS/MS-detection using internal standards.

Results and discussions

Table A 30: Recovery results from method validation of deltamethrin using the analytical method 01347

Matrix	Analyte	Fortification level (µg/kg) (*n* = 5)	Mean recovery (%)	RSD (%)	Overall mean [%] and RSD [%]
1st MRM transition					
Bees (insects)	deltamethrin	10	95	4.2	99 ± 4.6
		100	102	1.6	
Green material		10	99	5.3	98 ± 5.1
		100	96	5.1	
Flowers/ blossoms		10	95	7.3	98 ± 5.4
		100	100	1.8	
Nectar/ honey		10	97	4.0	97 ± 6.2
		100	97	8.4	
Pollen		10	76	7.6	83 ± 10.8
		100	90	6.0	
Wax		10	86	7.3	87 ± 5.6
		100	87	4.3	
2nd MRM transition					
Bees (insects)	deltamethrin	10	93	4.6	97 ± 5.7
		100	101	2.5	
Green material		10	100	1.2	97 ± 4.4
		100	94	4.7	
Flowers/ blossoms		10	99	6.5	100 ± 4.5
		100	100	1.8	
Nectar/ honey		10	93	6.5	93 ± 7.8
		100	93	9.7	
Pollen		10	80	1.6	84 ± 5.6
		100	88	3.2	
*Wax**		10	86	6.7	83 ± 6.4
		100	80	4.4	

* The evaluation of the 2nd MRM at the LOQ level (0.01 mg/kg) for wax is not according to the guideline (signal to noise >3/1). Therefore the confirmation of the residues of deltamethrin at the LOQ level is not possible for wax.

Table A 31: Characteristics for the analytical method used for validation of deltamethrin residues in pure solvent

	Deltamethrin
Specificity	mass spectrum is provided blank value < 30 % LOQ
Calibration (type, number of data points)	individual calibration data is presented calibration line equations: y =0.922 x + 0.00134 (1st MRM transition) y =0.954 x + 0.000849 (2nd MRM transition) Correlation coefficients r: 0.9995 (1/x weighted, 1st MRM transition) 0.9998 (1/x weighted, 2nd MRM transition) number of data points: 6
Calibration range	0.1 - 50 µg/L

	Deltamethrin
Specificity	mass spectrum is provided blank value < 30 % LOQ
Assessment of matrix effects is presented	Possible matrix effects of deltamethrin are eliminated by using an internal standard solution of the isotopically labelled analytical standard.
Limit of determination/quantification	LOQ = 10 µg/kg (in all sample materials) LOD = 2.5 µg/kg

Conclusion
The method 01347 meets all guideline criteria to determine residues of deltamethrin in/ on bees (insects), flowers/ blossoms, green material, honey /nectar, pollen and wax at 0.01 mg/kg with exception of wax samples at the LOQ level for the 2nd MRM. The evaluation of the 2nd MRM at the LOQ level (0.01 mg/kg) for wax is not according to the guideline (signal to noise >3/1). Therefore the confirmation of the residues of deltamethrin at the LOQ level is not possible for wax.

A 2.1.1.6.15 Concurrent validation of the analytical method 01347 in support of the study M-452717-01-1

Comments of zRMS:	The analytical method 01347 has been concurrently validated to determine concentration of deltamethrin in samples of beehive products from the colonies and of *Phacelia tanacetifolia* flowers. The limit of quantitation (LOQ) for deltamethrin was 0.01 mg/kg for all matrices. The average recoveries were within the acceptable range of 70 – 110% with the RSD values below 20%. The method is acceptable.

Reference:	**KCP 5.1/41**
Title:	Assessment of side effects on the honeybee (Apis mellifera L.), exposed to Phacelia tanacetifolia, sprayed sequentially with deltamethrin during flowering in a long-term field study in North Alsace, France
Report:	Rexer, H. U.; 2013; S10-03820; M-452717-01-1
Authority registration No:	
Guideline(s):	OEPP/EPPO Guideline No. 170 (4) (2010), SANCO/3029/99 rev. 4
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Concurrent validation
The concentration of residues of deltamethrin was determined in samples of beehive products from the colonies and of *Phacelia tanacetifolia* flowers.

The extraction procedures and analysis of beewax from comb / hive, nectar from comb / hive, pollen from comb / hive, and *Phacelia tanacetifolia* flowers via High Performance Liquid Chromatography (HPLC), coupled with electrospray and tandem mass spectrometry (MS/MS) detection, followed the provisions of method 01347 (Schoening, R.; Willmes, J.; 2013; M-444791-01-1). The Limit of Quantitation (LOQ) of deltamethrin, defined as the lowest validated fortification level, was 10 µg/kg (= 0.01 mg/kg) in all tested sample matrices.

The analytical method was validated concurrently during the present study. Recovery experiments were performed by spiking control samples with defined amounts of deltamethrin at fortification levels of 0.01 mg/kg (LOQ) and 0.10 mg/kg (10-fold LOQ). An additional fortification level was applied to flowers/blossoms at 100-fold LOQ level (1.0 mg/kg; 1000 µg/kg).

The average recoveries were within the acceptable range of 70 – 110% and wherever applicable (n ≥ 3), the RSD values were below 20%.

Table A 32: Recovery rates and precision results (repeatability) of deltamethrin

Analyte	Crop/Sample Material	FL [mg/kg]	Single Values [%]	Mean Value [%]	RSD [%]	LOQ [mg/kg]
Deltamethrin	Pollen	0.01	82; 75; 78; 80; 76	78	3.7	0.01
		0.10	86; 79; 84; 88; 90	85	4.9	
			Overall recovery (n = 10)	**82**	**6.2**	
Deltamethrin	Nectar	0.01	92; 88; 86; 91; 91	90	2.8	0.01
		0.10	100; 99; 98; 102; 103	100	2.1	
			Overall recovery (n = 10)	**95**	**6.4**	
Deltamethrin	Beewax	0.01	102; 97; 97; 93; 80	94	8.9	0.01
		0.10	86; 86; 86; 88; 83	86	2.1	
			Overall recovery (n = 10)	**90**	**7.9**	
Deltamethrin	Flowers / blossoms	0.01	102	-	-	0.01
		0.10	85	-	-	
		1.0	82	-	-	
			Overall recovery (n = 3)	**90**	**12.0**	

FL = Fortification Level, RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantitation

Table A 33: Characteristics for the analytical method 01347 used for validation of deltamethrin in beewax from comb / hive, nectar from comb / hive, pollen from comb / hive, and *Phacelia tanacetifolia* flowers

	deltamethrin
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 0.0933 x + 0.00245, Correlation coefficient r: 0.9999 number of data points: 6
Calibration range	0.1 µg/L - 50 µg/L (corresponds to 0.001 mg/kg – 0.5 mg/kg)
Limit of determination/quantification	LOQ = 0.01 mg/kg
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion

The applicability of the analytical method 01347 for the analysis of deltamethrin in samples of beehive products from the colonies and of *Phacelia tanacetifolia* flowers was tested. The data presented demonstrate that the method allows the determination of deltamethrin with satisfactory precision and repeatability and can be regarded as fit for purpose.

A 2.1.1.6.16 Concurrent validation of the analytical method 01347 in support of the study M-452723-01-1

Comments of zRMS:	The analytical method 01347 has been concurrently validated to determine concentration of deltamethrin in samples of beehive products from the colonies and of *Phacelia tanacetifolia* flowers. The limit of quantitation (LOQ) for deltamethrin was 0.01 mg/kg for all matrices. The average recoveries were within the acceptable range of 70 – 110% with the RSD values below 20%. The method is acceptable.

Reference:	**KCP 5.1/42**
Title:	Assessment of side effects on the honeybee (Apis mellifera L.), exposed to Phacelia tanacetifolia, sprayed sequentially with deltamethrin during flowering in a long-term field study in Mid Alsace, France
Report:	Rexer, H. U.; 2013; S10-03824; M-452723-01-1
Authority registration No:	
Guideline(s):	OEPP/EPPO Guideline No. 170 (4) (2010), SANCO/3029/99 rev. 4
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Concurrent validation

The concentration of residues of deltamethrin was determined in samples of beehive products from the colonies and of *Phacelia tanacetifolia* flowers.

The extraction procedures and analysis of beewax from comb / hive, nectar from comb / hive, pollen from comb / hive, and *Phacelia tanacetifolia* flowers via High Performance Liquid Chromatography (HPLC), coupled with electrospray and tandem mass spectrometry (MS/MS) detection, followed the provisions of method 01347 (Schoening, R.; Willmes, J.; 2013; M-444791-01-1). The Limit of Quantitation (LOQ) of deltamethrin, defined as the lowest validated fortification level, was 10 µg/kg (= 0.01 mg/kg) in all tested sample matrices.

The analytical method was validated concurrently during the present study. Recovery experiments were performed by spiking control samples with defined amounts of deltamethrin at fortification levels of 0.01 mg/kg (LOQ) and 0.10 mg/kg (10-fold LOQ). An additional fortification level was applied to flowers/blossoms at 100-fold LOQ level (1.0 mg/kg; 1000 µg/kg).

The average recoveries were within the acceptable range of 70 – 110% and wherever applicable (n ≥ 3), the RSD values were below 20%.

Table A 34: Recovery rates and precision results (repeatability) of deltamethrin

Analyte	Crop/Sample Material	FL [mg/kg]	Single Values [%]	Mean Value [%]	RSD [%]	LOQ [mg/kg]
Deltamethrin	Pollen	0.01	77, 77, 80, 82, 79	79	2.7	0.01
		0.10	89, 92, 93, 90, 92	91	1.8	
			Overall recovery (n = 10)	**85**	**7.8**	
Deltamethrin	Nectar	0.01	92, 97, 82, 89, 89	90	6.1	0.01
		0.10	100, 101, 100, 98, 101	100	1.2	
			Overall recovery (n = 10)	**95**	**6.9**	
Deltamethrin	Beewax	0.01	101, 102, 102, 104, 98	101	2.2	0.01
		0.10	81, 79, 84, 87, 88	83	4.0	
			Overall recovery (n = 10)	**92**	**10.7**	
Deltamethrin	Flowers / blossoms	0.01	84	-	-	0.01
		0.10	94	-	-	
		1.0	78	-	-	
			Overall recovery (n = 3)	**85**	**9.5**	

FL = Fortification Level, RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantitation

Table A 35: Characteristics for the analytical method 01347 used for validation of deltamethrin in beewax from comb / hive, nectar from comb / hive, pollen from comb / hive, and *Phacelia tanacetifolia* flowers

	deltamethrin
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 0.851 x + 0.00121, Correlation coefficient r: 0.9995 number of data points: 6
Calibration range	0.1 µg/L - 50 µg/L (corresponds to 0.001 mg/kg – 0.5 mg/kg)
Limit of determination/quantification	LOQ = 0.01 mg/kg
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion
The applicability of the analytical method 01347 for the analysis of deltamethrin in samples of beehive products from the colonies and of *Phacelia tanacetifolia* flowers was tested. The data presented demonstrate that the method allows the determination of deltamethrin with satisfactory precision and repeatability and can be regarded as fit for purpose.

A 2.1.1.6.17 Analytical method 01347 (modified) in support of the study M-477250-01-1

A 2.1.1.6.17.1 Method validation

Comments of zRMS:	The analytical method 01347 has been validated to determine concentration of deltamethrin in the employed feeding solutions. The limit of quantitation (LOQ) for deltamethrin was 0.01 mg/kg for the sample material aqueous sugar solution. The mean recovery was 99% with a relative standard deviation (RSD) of 2%. The method is acceptable.

Reference:	**KCP 5.1/43**
Title:	Deltamethrin EW 15B G - Assessment of chronic effects to the honeybee, Apis mellifera L., in a 10 days continuous laboratory feeding test
Report:	Kling, A.; 2014; S13-00151; M-477250-01-1
Authority registration No:	
Guideline(s):	not applicable
Deviations:	not applicable
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The concentration of deltamethrin was determined in the feeding solution employed to determine the chronic effects of the test item deltamethrin on the honeybee, *Apis mellifera* L, in a 10 days continuous feeding test in the laboratory.

For the determination of deltamethrin the analytical method 01347 (Schoening, R.; Willlmes, J.; 2013; M-444791-01-1) was used. In deviation to the original method 01347, only parts of the analytical method like chromatography conditions, mass transitions etc. were necessary to determine the concentration of deltamethrin in the employed feeding solutions. According to the original method, deltamethrin was extracted from samples of bees (insects), flowers/blossoms, green material, honey/nectar, pollen and wax with a mixture of dichloromethane/n-hexane/acetone. After filtration, an aliquot of this solution was evaporated to the aqueous remainder and cleaned-up on a ChemElut column or by liquid/liquid partition for wax. After elution of the residues with dichloromethane or separation with acetonitrile, the extract was evaporated to dryness and re-dissolved in a mixture of water/acetonitrile. The residues were quantified by

reversed phase HPLC with ESI MS/MS-detection using internal standards (MRM used for quantification m/z: 523 → 281; MRM used for confirmation (ISTD) m/z: 529 → 281).

Due to the fact that the concentration in the feeding solutions of study S13-00151 were at a very high level, it was only necessary to take an aliquot of the feeding solution and to dilute the sample. Thereafter, aliquots of the diluted samples were subjected to reversed phase High Performance Liquid Chromatography (HPLC) coupled with electrospray and mass spectrometry (MS/MS) detection without a further clean-up step.

The limit of quantitation (LOQ) for deltamethrin was 0.01 mg/kg (= 10 µg/kg) for the sample material aqueous sugar solution, corresponding to the lowest fortification level of successfully conducted recovery experiments.

Results and discussions
Recovery experiments were performed by spiking control samples (control sugar solution) with deltamethrin at fortification levels of 0.01 mg/kg, 0.10 mg/kg and 200 mg/kg.

The individual recovery values for deltamethrin ranged from 95 to 102%, with an overall recovery of 99% and a relative standard deviation (RSD) of 2.0 % (n = 13).

Table A 36: Recovery rates and precision results (repeatability) of deltamethrin

Analyte	Crop/Sample Material	FL [mg/kg]	Single Values [%]	Mean Value [%]	RSD [%]	LOQ [mg/kg]
Deltamethrin	Aqueous sugar solution	0.01	96, 95, 98, 99, 99	97	1.9	0.01
		0.10	98, 100, 99, 102, 101	100	1.6	
		200	98, 101, 99	99	1.5	
			Overall recovery (n = 13)	**99**	**2.0**	

FL = Fortification Level, RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantitation

Table A 37: Characteristics for the analytical method 01347 used for validation of deltamethrin in feeding solution (aqueous sugar solution)

	deltamethrin
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 0.942471 x + 0.00115209, Correlation coefficient r: 0.9998 number of data points: 7
Calibration range	0.05 ng/mL - 50 ng/mL (corresponds to 0.0005 mg/kg – 0.5 mg/kg)
Limit of determination/quantification	LOQ = 0.01 mg/kg
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of deltamethrin in feeding solution via HPLC-MS/MS.

A 2.1.1.6.18 Analytical method in support of the studies M-554592-01-1 and M-554604-01-1

A 2.1.1.6.18.1 Method validation

Comments of zRMS:	An analytical method for the determination of deltamethrin and flupyradifurone was succesfully validated with regard to recovery, linearity of detector response, repeatability, specificity, limit of quantification and limit of detection.

	The limit of quantification (LOQ) was 720 mg/L of test item fortification level (6.24 mg/L of deltamethrin and 47.7 mg/L flupyradifurone). Mean recoveries and relative standard deviations per fortification fulfil the criteria of guideline SANCO/3029/99 (70 - 110 % mean recovery, ≤ 20 % RSD). The analytical method is acceptable.

Reference:	**KCP 5.1/44**
Title:	Deltamethrin + flupyradifurone EC 85 (10+75 g/L): Effects on the seedling emergence of non-target terrestrial plant species under greenhouse conditions
Report:	Ripperger, D.; 2016; S15-01670; M-554592-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC Regulation (EC) No. 1107/2009 OCSPP 850.4100 (2012) OECD 208 (2006)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/45**
Title:	Deltamethrin + flupyradifurone EC 85 (10+75 g/L): Effects on the vegetative vigour of non-target terrestrial plant species under greenhouse conditions
Report:	Ripperger, D.; 2016; S15-01671; M-554604-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC Regulation (EC) No. 1107/2009 OCSPP 850.4150 (2012) OECD 227 (2006)
Deviations:	Deviations with no major impact occurred regarding the test conditions
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

In the present study, an analytical method was validated for the determination of deltamethrin and flupyradifurone in spray solution samples used to treat non-target terrestrial plant species. In the following part, only data for deltamethrin is presented.

The test samples are analysed by direct injection in an HPLC-PDA instrument after appropriate dilution.

The same method was used in the study on effects on the vegetative vigour of non-target terrestrial plant species (Ripperger, D.; 2016; M-554604-01-1) for the analytical verification of the spray solutions. No additional validation parameters were presented in this study.

Results and discussions

Recovery samples were prepared by fortification of untreated samples of deionized water with the test item and determined at test item fortification levels of 720 mg/L and 9500 mg/L.

Mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 38: Recovery rates and precision results (repeatability) of deltamethrin

Test item fortification level	deltamethrin				
	Nominal	Effective	Found	Recovery	Mean Recovery

[mg/L]	[mg/L]	[mg/L]	[mg/L]	[%]	± RSD [%]
720	6.24	6.24	5.98 6.06 6.06 5.96 6.06	96 97 97 96 97	97 ± 1
9500	82.4	86.0 83.8 83.7 84.2 82.8	80.7 82.1 85.9 88.7 80.3	94 98 103 105 97	99 ± 5

RSD: relative standard deviation

Table A 39: Characteristics for the analytical method used for validation of deltamethrin

	deltamethrin
Specificity	HPLC-PDA method is specific. Blank values of analyte were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented calibration equation (linear): y = 0.1845 x – 0.0219, Correlation coefficient r: 0.9999 number of data points: 6
Calibration range	2.00 mg/L to 100.0 mg/L
Limit of determination/quantification	LOQ = 6.24 mg/L deltamethrin (corresponds to 720 mg/L of test item)
Assessment of matrix effects is presented	No effects observed.

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4. It was validated successfully and can be seen as fit for purpose.

A 2.1.1.7 Description of analytical methods for the determination of residues in support of physical and chemical properties tests (KCP 5.1)

The analytical method used for the generation of pre-authorization data is the same as the one described in part B section 5.2.1

A 2.1.1.7.1 Analytical method in support of the water solubility study M-435779-01-1

A 2.1.1.7.1.1 Method validation

Comments of zRMS:	The method is acceptable.

Reference:	**KCP 5.1/46**
Title:	AE F108565 (Br2CA): Solubility in water at pH 5, pH 7 and pH 9
Report:	Wiche, A.; Bogdoll, B.; 2012; PA10/073; M-435779-01-1
Authority registration No:	
Guideline(s):	European Commission Council Regulation (EC) No 440/2008, Annex, Part A, method A.6.; OECD-Guideline 105; EPA Product Properties Test Guideline OPPTS 830.7840
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The analytical method described in the report of Wiche, A.; Bogdoll, B.; 2012; AE F108565 (Br2CA): Solubility in water at pH 5, pH 7 and pH 9; M-435779-01-1 was developed to determine solubility of the test item AE F108565 (BR2CA) in water at pH 5, pH 7 and pH 9. The concentration of AE F108565 was

quantified by HPLC analyses.

The concentration of AE F108565 was quantified by HPLC-UV method (reversed phase). The reference item was injected dissolved in acetonitrile and diluted in acetonitrile / water (ratio 50:50 v/v). The concentration of the test item was quantified by comparing integrated peak areas to integrated peak areas measured by injection of solutions with known amounts of test sample (external standard method).

The limit of quantification was defined as the lowest concentration from the linearity test: LOQ = 0.15 mg/L.

Results and discussions
The evaluation of accuracy was obtained by the analysis of five sample solutions (five individual sample weights). Each sample was injected twice. The values found for the precision (0.2 %) and for the accuracy (100.9%) are acceptable. The individual accuracy results expressed as recovery rates and the relative standard deviation determined as the precision of the method are shown in the table below.

Table A 40: Validation parameters accuracy and precision

Reference	Concentration measured [mg/L]	Concentration expected [mg/L]	Recovery
Reference-2	295.7	293.8	100.6
Reference-3	362.4	359.6	100.8
Reference-4	222.9	220.2	101.2
Reference-5	293.9	291.4	100.9
Reference-6	233.1	231.2	100.8
	Mean = Accuracy (average recovery rate) [%]		100.9
	Rel. standard deviation = Precision [%]		0.2

The detector linearity was tested in a concentration range from 0.15 mg/L to 61 mg/L using seven different concentrations.
Equation of the calibration line: y = 0.160754 x + 0.01636
Correlation coefficient: r = 0.99997

Chromatograms of a blank experiment at pH 5, a blank run at pH 7 and the certified test item were checked for interfering compounds. The blank experiment and the blank run were free of interfering compounds.

Conclusion
The analytical method was developed to determine solubility of the test item AE F108565 (BR2CA) in water at pH 5, pH 7 and pH 9. The concentration of AE F108565 was quantified by HPLC method (reversed phase). The detector linearity was tested in a concentration range from 0.15 mg/L to 61 mg/L using seven different concentrations. The values found for the precision (0.2 %) and for the accuracy (100.9%) are acceptable.
The used HPLC-UV method as well as the analytical method were found to be valid and comply with all criteria according to *SANCO/3029/99 rev. 4* and is suitable for the determination of AE F108565 (BR2CA).
The method is fit for purpose with regard to the study Wiche, A.; Bogdoll, B.; 2012; AE F108565 (Br2CA): Solubility in water at pH 5, pH 7 and pH 9; M-435779-01-1.

A 2.1.2 Methods for post-authorization control and monitoring purposes (KCP 5.2)

A 2.1.2.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

A 2.1.2.1.1 Analytical method 00086/M089

A 2.1.2.1.1.1 Method validation

Comments of zRMS:	The study of Weber, H.; 2009 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The the multi method DFG S 19 (L 00.0034) (BCS Method ID 00086/M089) was validated for the the determination of residues of cis-deltamethrin (AE F032640) in tomato (fruit), orange (fruit), barley (grain), dried bean (seed) and oilseed rape (seed).* *The limit of quantitation (LOQ) for cis-deltamethrin is 0.01 mg/kg in all matrices tested.* *For all matrices, analysis of control specimens by GC-MSD indicated that residues of the test substance were below 30% of the LOQ.* *Mean recoveries for each fortification level and the overall mean recovery were within the 70 - 110% range for all matrices. Relative standard deviations were below 20% for cis-deltamethrin and all sample materials.* *Three ions (253, 251, 181) were monitored to fulfil the requirement for validation of this confirmatory method.* *The residue definition for monitoring is cis-deltamethrin. The analytical method is suitable to support this residue definition.* *The method meets all guideline criteria (SANCO/825/00/rev. 8.1 and SANCO/3029/99 rev. 4) to determine residues of cis-deltamethrin in plant matrices such as tomato (fruit), orange (fruit), barley (grain), dried bean (seed) and oilseed rape (seed) with satisfactory accuracy, precision and repeatability.* *The study is acceptable.*

Reference:	**KCP 5.2/01**
Title:	Validation of enforcement method DFG S19 (L 00.00-34) (BCS method ID 00086/M089) for the determination of cis-deltamethrin (AE F032640) in/on foodstuff of plant origin
Report:	Weber, H.; 2009; S09-00553; M-351076-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC as amended by 96/46/EC 4.2.1 Guidance document SANCO/3029/99 rev. 4 of 11/07/00 of the European Commission Guidance document SANCO/0825/00 rev. 7 of 17/03/04 of the European Commission BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of this study was to validate the multi method DFG S 19 (L 00.0034)
(BCS Method ID 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in tomato (fruit), orange (fruit), barley (grain), dried bean (seed) and oilseed rape (seed).
A series of recovery experiments were performed by fortifying control (untreated) specimens of the matrices tomato (fruit), orange (fruit), oilseed rape (seed), dry bean (seed) and barley (grain).
The validation followed the procedures as described in the multi method DFG S 19 (L 00.0034) [1] as well as the guideline SANCO/825/00 rev.7 (17/03/04).
Cis-deltamethrin was extracted from tomato (fruit), orange (fruit), dry bean (seed) and barley (grain) specimens with acetone/water (2/1, v/v). Thereafter, ethyl acetate/cyclohexane (1/1, v/v) and sodium chloride were added for liquid-liquid partition. An aliquot of the organic phase was evaporated to dryness.
From oilseed rape (seeds), cis-deltamethrin was extracted with acetonitrile/acetone (9/1, v/v) in the presence of synthetic calcium silicate (trade name Calflo E) and Celite. The organic phase was filtered and evaporated to dryness.
For all matrices the evaporated extract was cleaned up by gel permeation chromatography (GPC) on Bio Beads S-X3 polystyrene gel using a mixture of ethyl acetate/cyclohexane (1/1, v/v) as eluant. The collected extracts were further cleaned on a mini silicagel column and analysed for residues of deltamethrin by gas chromatography with mass selective detection (GC-MSD).

Three ions were monitored to fulfil the requirement for validation of this confirmatory method. Using GC-MSD, the ions 253, 251 and 181 were used for the determination of cis-deltamethrin in tomato (fruit), orange (fruit), oilseed rape (seed), dried bean (seed) and barley (grain). Control specimens were analysed in duplicate and fortified specimens were analysed in quintuple for each fortification level.

Results and discussions

For cis-deltamethrin in tomato (fruit), orange (fruit), oilseed rape (seed), dried beans (seed) and barley (grain) the limit of quantitation (LOQ) was 0.01 mg/kg.

Fortification experiments were performed for tomato (fruit), orange (fruit), dried bean (seed) and oilseed rape (seed) at the limits of quantitation (LOQ) and tenfold LOQ. For barley (grain), fortification experiments were performed at the LOQ, tenfold LOQ and two hundred fold LOQ.

The linearity of the detector response was confirmed by injecting eight external standard solutions in the range between of 0.00500 to 1.25 µg/mL of deltamethrin. Correlation coefficients R were ≥ 0.9960 for the three monitored ions.

For all matrices, analysis of control specimens by GC-MSD indicated that residues of the test substance were below 30% of the LOQ.

Mean recovery values obtained for tomato (fruit), orange (fruit), oilseed rape (seed), dry bean (seed) and barley (grain) specimens for all fortification levels comply with the standard acceptance criteria of SANCO Guideline 825/00.

Furthermore, as required by the standard acceptance criteria the overall relative standard deviation and the relative standard deviation for each fortification level were ≤ 20 %.

Matrix effects were tested by evaluating the results with solvent standards and with matrix-matched standards. For cis-deltamethrin, signal suppression or enhancement below 10% were observed for all matrices. Therefore solvent standards were used for the analysis.

Cis-deltamethrin in calibration solution is stable during storage under refrigerator conditions for at least 25 days.

Cis-deltamethrin in extract is stable during storage under refrigerator conditions for at least 23 days.

Table A 41: Recovery results from method validation of cis-deltamethrin using the analytical method in different plant matrices

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin					
tomato (fruit)	0.01	5	78	3.9	m/z=253
	0.10	5	84	11	m/z=253
	0.01	5	88	11	m/z=251
	0.10	5	85	12	m/z=251
	0.01	5	81	4.5	m/z=181
	0.10	5	85	11	m/z=181
orange (fruit)	0.01	5	96	6.2	m/z=253
	0.10	5	88	4.5	m/z=253
	0.01	5	95	4.9	m/z=251
	0.10	5	88	5.5	m/z=251
	0.01	5	92	5.0	m/z=181
	0.10	5	90	5.6	m/z=181
dried beans (seed)	0.01	5	101	10	m/z=253
	0.10	5	89	5.3	m/z=253
	0.01	5	98	8.4	m/z=251
	0.10	5	90	5.2	m/z=251

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
	0.01	5	97	10	m/z=181
	0.10	5	89	5.1	m/z=181
barley (grain)	0.01	5	94	10	m/z=253
	0.10	5	79	8.6	m/z=253
	2.0	5	78	9.0	m/z=253
	0.01	5	88	18	m/z=251
	0.10	5	81	7.0	m/z=251
	2.0	5	80	9.0	m/z=251
	0.01	5	94	9.4	m/z=181
	0.10	5	81	9.0	m/z=181
	2.0	5	80	8.9	m/z=181
oilseed rape (seed)	0.01	5	86	6.1	m/z=253
	0.10	5	74	4.5	m/z=253
	0.01	5	81	8.3	m/z=251
	0.10	5	74	4.2	m/z=251
	0.01	5	82	5.3	m/z=181
	0.10	5	74	4.7	m/z=181

Table A 42: Characteristics for the analytical method used for validation of cis-deltamethrin in different plant matrices

	cis-deltamethrin
Specificity	mass spectrum is provided in Appendix 3 of the method report blank value < 30 % LOQ)
Calibration (type, number of data points)	A one point external standard calibration was carried out.individual calibration data presented| in Appendix 2 of the repport calibration line equation presented for each m/z number of data points>5 (8) m/z=253→R = 0.9963 m/z=251→R = 0.9960 m/z=181→R = 0.9967
Calibration range	External standard with one point calibration in the range between of 0.0050 to 1.25 µg/mL of cis-deltamethrin
Assessment of matrix effects is presented	yes
Limit of determination/quantification	LOQ = 0.01 mg/kg

Conclusion

The results demonstrate that the multi method DFG S 19 (L 00.0034) (BCS Method ID 00086/M089) permits the determination of residues of cis-deltamethrin in tomato (fruit), orange (fruit), oilseed rape (seed), dry bean (seed) and barley (grain). The validation proved that the method is valid for the determination of residues of cis-deltamethrin in plant matrices. The method has proven its applicability as an enforcement method.

A 2.1.2.1.1.2 Independent laboratory validation

Comments of zRMS:	The study of Merdian, H.; 2009 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The Bayer CropScience method 00086/M0089 based on DFG method S19 was independently validated successfully for the determination of residues of cis-deltamethrin in plant materials by GC/MS, exemplified for tomato (fruit), barley (grain), orange (fruit), dry beans (seed) and oilseed rape (seed), all with a limit of quantitation (LOQ) of 0.01 mg/kg.* *Specimens were fortified (5 replicates per matrix and fortification level) at the LOQ and at the 10-fold LOQ, and for barley grain at the 200-fold LOQ.* *For all three MS ions monitored, apparent residues in all blank control specimens were below 20% of the LOQ.* *The average recoveries were within the acceptable range of 70% to 110% with relative standard deviations (RSD) ≤ 20%.* *The study is acceptable.*

Reference:	**KCP 5.2/02**
Title:	Independent laboratory validation of the DFG method S19 (BCS method 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in plant materials, using GC/MS
Report:	Merdian, H.; 2009; P/B 1681 G; M-356306-01-1
Authority registration No:	
Guideline(s):	EC Directive 91/414/EEC, EC Guidance document on residue analytical methods, SANCO/825/00 rev. 7 17/03/04 and the OECD Guidance Document on Pesticide Residue Analytical Methods (ENV/JM/Mono (2007) 17, 2007-08-13)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The objective was to independently validate Bayer CropScience method 00086/M0089 based on DFG method S19 for the determination of residues of cis-deltamethrin in plant materials by GC/MS, exemplified for tomato (fruit), barley (grain), orange (fruit), dry beans (seed) and oilseed rape (seed), all with a limit of quantitation (LOQ) of 0.01 mg/kg.

Residue analysis was performed according to the original method validation report, with minor modifications indicated and explained in the report. These minor modifications became necessary due to slightly different laboratory routines and are considered insignificant.

Extraction was done following extraction procedures (modules E1 for tomato, E2 for bean and grain, E3 for orange, and E7 for oilseed rape seed), with subsequent clean-up according to module GPC and mini silica gel chromatography, and gas chromatography with mass selective detection (GC/MS), monitoring three ions for the determination and confirmation (253, 251 and 181 m/z).

Results and discussions

As demonstrated by the independent laboratory validation results, the method allows the determination of cis-deltamethrin with a limit of quantification (LOQ) of 0.01 mg/kg.
Apparent residues in all blank control specimens were below 20 % of the LOQ.

Specimens were fortified (5 replicates per matrix and fortification level) at the LOQ and at the 10-fold LOQ, and for barley grain at the 200-fold LOQ. Additional specimens were kept untreated as blank controls. The linearity of the detector response was confirmed by injecting seven external standard solutions in the range between of 0.005 to 0.500 µg/mL of deltamethrin. Correlation coefficients R were ≥ 0.9960 for the

three monitored ions. Correlation coefficients R were ≥ 0.980 for the three monitored ions.
For all three MS ions monitored, apparent residues in all blank control specimens were below 20 % of the LOQ.

The average recoveries were within the acceptable range of 70 % to 110 % with relative standard deviations (RSD) ≤ 20 %.
All standard solutions were stored refrigerated in amber glass bottles when not in use. The solutions were stable for approximately one month as demonstrated by consistent GC/MS results obtained during the study.

Summaries of the independent laboratory validation results are given below:

Table A 43: Recovery results from method validation of cis-deltamethrin using the analytical method in different plant matrices

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin					
tomato (fruit)	0.01	5	97	3	m/z=253
	0.10	5	89	13	m/z=253
	0.01	5	97	3	m/z=251
	0.10	5	91	11	m/z=251
	0.01	5	91	4	m/z=181
	0.10	5	89	12	m/z=181
orange (fruit)	0.01	5	98	10	m/z=253
	0.10	5	90	15	m/z=253
	0.01	5	98	8	m/z=251
	0.10	5	92	13	m/z=251
	0.01	5	99	7	m/z=181
	0.10	5	91	17	m/z=181
dried beans (seed)	0.01	5	96	5	m/z=253
	0.10	5	83	13	m/z=253
	0.01	5	94	10	m/z=251
	0.10	5	84	15	m/z=251
	0.01	5	97	5	m/z=181
	0.10	5	84	12	m/z=181
barley (grain)	0.01	5	97	7	m/z=253
	0.10	5	90	20	m/z=253
	2.0	5	82	4	m/z=253
	0.01	5	96	10	m/z=251
	0.10	5	90	19	m/z=251
	2.0	5	83	3	m/z=251
	0.01	5	98	6	m/z=181
	0.10	5	90	16	m/z=181
	2.0	5	84	5	m/z=181
oilseed rape (seed)	0.01	5	97	6	m/z=253
	0.10	5	98	7	m/z=253
	0.01	5	94	9	m/z=251

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
	0.10	5	98	5	m/z=251
	0.01	5	98	7	m/z=181
	0.10	5	101	4	m/z=181

Table A 44: Characteristics for the analytical method used for validation of cis-deltamethrin in different plant matrices

	cis-deltamethrin
Specificity	mass spectrum is provided in Figure 3 of the method report blank value < 20 % of the LOQ)
Calibration (type, number of data points)	A one point external standard calibration for cis-deltamethrin was carried out. individual calibration data presented| in Figure 2 of the method report calibration line equation presented for each m/z in Figure 1 of the method report number of data points>5 (7) m/z=253→R = 0.9848 m/z=251→R = 0.9800 m/z=181→R = 0.9902
Calibration range	External standard with one point calibration in the range between of 5.0 to 500 ng/mL of cis-deltamethrin
Assessment of matrix effects is presented	yes
Limit of determination/quantification	LOQ = 0.01 mg/kg

Conclusion
PTRL Europe performed the independent laboratory validation (ILV) of the DFG method S19 originally validated by Harald Weber (Specht Study No.: BAY-0902V, Bayer CropScience Project No.: P682097524, 07-Jul-09).

The method was independently validated successfully for the determination of residues of cis-deltamethrin in/on plant matrices by GC/MS, exemplified for tomato (fruit), barley (grain), orange (fruit), dry beans (seed) and oilseed rape (seed), demonstrating the LOQ of 0.01 mg/kg.

It is concluded that DFG method S19 fulfils the reproducibility requirements as defined in the Council Directive 91/414/EEC, in the EC Guidance document on residue analytical methods (SANCO/825/00 rev. 7 17/03/04) and in the OECD Guidance Document on Pesticide Residue Analytical Methods (ENV/JM/Mono (2007) 17, 2007-08-13) and is, therefore, applicable as an enforcement method.

A 2.1.2.1.1.3 Confirmatory method (if required)

No confirmatory method is required because of the specificity of the detection methods (three transition monitored:

A 2.1.2.1.1.4 Extraction efficiency

Refer to point A 2.1.1.1.1.3

A 2.1.2.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

A 2.1.2.2.1 Analytical method DFG S 19

A 2.1.2.2.1.1 Method validation

Comments of zRMS:	The study of Weber, H.; 2009 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The multi method DFG method S 19 (BCS Method ID 00086/M090) was validated for the the determination of residues of cis-deltamethrin (AE F032640) in milk, egg, bovine muscle, liver, kidney and fat.* *The limit of quantitation (LOQ) for cis-deltamethrin is 0.01 mg/kg in all matrices tested.* *For all matrices, analysis of control specimens by GC-MSD indicated that residues of the test substance were below 30% of the LOQ.* *Mean recoveries for each fortification level and the overall mean recovery were within the 70 - 110% range for all matrices. Relative standard deviations were below 20% for cis-deltamethrin and all sample materials.* *Three ions (253, 251, 181) were monitored to fulfil the requirement for validation of this confirmatory method.* *The residue definition for monitoring is cis-deltamethrin. The analytical method is suitable to support this residue definition.* *The method meets all guideline criteria (SANCO/825/00/rev. 8.1 and SANCO/3029/99 rev. 4) to determine residues of cis-deltamethrin in animal matrices such as milk, egg, bovine muscle, liver, kidney and fat with satisfactory accuracy, precision and repeatability.* *The study is acceptable.*

Reference:	**KCP 5.2/03**
Title:	Validation of enforcement method DFG S19 (L 00.00-34) (BCS method ID 00086/M090) for the determination of residues cis-deltamethrin (AE F032640) in/on foodstuff of animal origin
Report:	Weber, H.; 2009; S09-00551; M-351080-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC as amended by 96/46/EC 4.2.1 Guidance document SANCO/3029/99 rev. 4 of 11/07/00 of the European Commission Guidance document SANCO/0825/00 rev. 7 of 17/03/04 of the European Commission BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The purpose of this study was the examination of the applicability of the DFG Method S 19 (BCS Method ID 00086/M090) for the determination of residues of cis-deltamethrin (AE F032640) in milk, egg, bovine muscle, liver, kidney and fat. This examination followed the extended and revised version of the DFG Method S 19, published as 'Modular Multiple Analytical Method for the Determination of Pesticide Residues in Foodstuffs, L 00.00-34', as part of the Official Collection of Test Methods under Article 64 of the LFGB (German Food, Commodity and Feed Code).

A series of recovery experiments were performed by fortifying control (untreated) specimens of the matrices milk, egg, bovine muscle, liver, kidney and fat. The validation followed the procedures as described in the multi method DFG S 19 (L 00.0034) [1] as well as the guideline SANCO/825/00 rev.7 (17/03/04).

Cis-deltamethrin was extracted from milk (whole milk, 3.5 % fat), poultry egg (yolk and egg white, without shell), bovine muscle, liver (from cattle) and kidney (from cattle) specimens with acetone/water (2/1, v/v). Thereafter, ethyl acetate/cyclohexane (1/1, v/v) and sodium chloride were added for liquid-liquid partition. An aliquot of the organic phase was evaporated to dryness.
For the analysis of cis-deltamethrin in fat the samples were dissolved in ethyl acetate/cyclohexane (1/1,

v/v) and directly cleaned by gel permeation chromatography (GPC).
For all other matrices the evaporated extract was cleaned up by gel permeation chromatography (GPC) on Bio Beads S-X3 polystyrene gel using a mixture of ethyl acetate/cyclohexane (1/1, v/v) as eluant. The collected extracts were further cleaned on a mini silicagel column and analysed for residues of cis-deltamethrin by gas chromatography with mass selective detection (GC-MSD).

Using GC-MSD, three ions were monitored to fulfil the requirement for validation of this confirmatory method. Ions m/z 253 (quantification) and m/z 251 and 181 m/z (confirmation) were used for the determination of cis-deltamethrin in milk, egg, bovine muscle, liver, kidney and fat. Control specimens were analysed in duplicate and fortified specimens were analysed in quintuple for each fortification level.

Results and discussions
For cis-deltamethrin in milk, egg, bovine muscle, liver, kidney and fat the limit of quantitation (LOQ) was 0.01 mg/kg.

Fortification experiments were performed for milk, egg, bovine muscle, liver, kidney and fat at the limits of quantitation (LOQ) and tenfold LOQ.

The linearity of the detector response was confirmed by injecting nine external standard solutions in the range between of 0.00250 to 1.25 µg/mL of cis-deltamethrin. Correlation coefficients R were ≥ 0.9968 for the three monitored ions.

For all matrices, analysis of control specimens by GC-MSD indicated that residues of the test substance were below 30% of the LOQ.

Mean recovery values obtained for milk, egg, bovine muscle, liver, kidney and fat specimens for all fortification levels comply with the standard acceptance criteria of SANCO Guideline 825/00.
Furthermore, as required by the standard acceptance criteria, the overall relative standard deviation and the relative standard deviation for each fortification level were ≤ 20 %.
Matrix effects were tested by evaluating the results with solvent standards and with matrix-matched standards. For cis-deltamethrin, signal suppression or enhancement below 10% were observed for all matrices. Therefore solvent standards were used for the analysis.
Stabilities of cis-deltamethrin in the calibration solution and in selected extracts were assessed at the end of the study. The results prove that stability of the Cis-deltamethrin in calibration solution is stable during storage under refrigerator conditions for at least 25 days and stability of Cis-deltamethrin in extract is stable during storage under refrigerator conditions for at least 24 days

Table A 45: Recovery results from method validation of cis-deltamethrin using the analytical method

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin					
milk	0.01	5	84	15	m/z=253
	0.10	5	87	15	m/z=253
	0.01	5	86	13	m/z=251
	0.10	5	88	15	m/z=251
	0.01	5	86	17	m/z=181
	0.10	5	87	15	m/z=181
egg	0.01	5	94	7.5	m/z=253
	0.10	5	88	9.0	m/z=253
	0.01	5	91	6.2	m/z=251
	0.10	5	86	7.9	m/z=251

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
	0.01	5	96	7.7	m/z=181
	0.10	5	86	8.0	m/z=181
bovine muscle	0.01	5	78	15	m/z=253
	0.10	5	82	8.5	m/z=253
	0.01	5	78	14	m/z=251
	0.10	5	82	10	m/z=251
	0.01	5	81	15	m/z=181
	0.10	5	83	9.2	m/z=181
liver	0.01	5	84	16	m/z=253
	0.10	5	85	13	m/z=253
	0.01	5	85	16	m/z=251
	0.10	5	87	15	m/z=251
	0.01	5	84	18	m/z=181
	0.10	5	87	14	m/z=181
kidney	0.01	5	80	13	m/z=253
	0.10	5	85	13	m/z=253
	0.01	5	77	14	m/z=251
	0.10	5	85	14	m/z=251
	0.01	5	83	11	m/z=181
	0.10	5	86	14	m/z=181
fat	0.01	5	95	6.2	m/z=253
	0.10	5	81	4.8	m/z=253
	0.01	5	81	3.4	m/z=251
	0.10	5	80	5.0	m/z=251
	0.01	5	92	10	m/z=181
	0.10	5	80	4.9	m/z=181

Table A 46: Characteristics for the analytical method used for validation of cis-deltamethrin residues in different animal matrices

	cis-deltamethrin
Specificity	mass spectrum is provided in Appendix 3 of the method report blank value < 30 % LOQ)
Calibration (type, number of data points)	individual calibration data presented| in Appendix 2 of the report calibration line equation presented for each m/z number of data points>5 (9) m/z=253→R = 0.9968 m/z=251→R = 0.9968 m/z=181→R = 0.9975 A one point external standard caliabration was carried out.
Calibration range	External standard with one point calibration in the range between of 0.0025 to 1.25 µg/mL of cis-deltamethrin
Assessment of matrix effects is presented	yes
Limit of determination/quantification	LOQ = 0.01 mg/kg

Conclusion
The results demonstrate that the multi method DFG S 19 (L 00.0034) (BCS Method ID 00086/M090) permits the determination of residues of cis-deltamethrin in milk, egg, bovine muscle, liver, kidney and fat. The validation proved that the method is valid for the determination of residues of cis-deltamethrin in animal matrices. The method can therefore be considered valid and applicable as enforcement method.

A 2.1.2.2.1.2 Independent laboratory validation

Comments of zRMS:	The study of Merdian, H.; 2009 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The Bayer CropScience method 00086/M0090 based on DFG method S19 was independently validated successfully for the determination of residues of cis-deltamethrin in animal matrices by GC/MS, exemplified for whole milk, hen´s egg, bovine muscle, liver, kidney and fat all with a limit of quantitation (LOQ) of 0.01 mg/kg.* *For independent laboratory validation (ILV), specimens were fortified (5 replicates per matrix and fortification level) at the LOQ and at the 10-fold LOQ.* *For all three MS ions monitored, apparent residues in all blank control specimens were below 20% of the LOQ.* *The average recoveries were within the acceptable range of 70% to 110% with relative standard deviations (RSD) ≤ 20%.* *The study is acceptable.*

Reference:	**KCP 5.2/04**
Title:	Independent laboratory validation of the DFG method S19 (BCS method 00086/M089) for the determination of residues of cis-deltamethrin (AE F032640) in foodstuffs of animal origin, using GC/MS
Report:	Merdian, H.; 2009; P/B 1682 G; M-356331-01-1
Authority registration No:	
Guideline(s):	EC Directive 91/414/EEC, EC Guidance document on residue analytical methods, SANCO/825/00 rev. 7 17/03/04 and the OECD Guidance Document on Pesticide Residue Analytical Methods (ENV/JM/Mono (2007) 17, 2007-08-13)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The objective was to independently validate Bayer CropScience method 00086/M090 based on DFG method S19 for the determination of residues of cis-deltamethrin in animal matrices by GC/MS, exemplified for whole milk, hen´s egg, bovine muscle, liver, kidney and fat all with a limit of quantitation (LOQ) of 0.01 mg/kg.

Extraction was done following extraction modules E 1 (milk, egg, muscle, liver and kidney) and E 6 (fat), with subsequent clean-up according to module GPC and mini silica gel chromatography, and gas chromatography with mass selective detection (GC/MS), monitoring three ions for the determination and confirmation(253, 251 and 181 m/z).

Results and discussions
For independent laboratory validation (ILV), specimens were fortified (5 replicates per matrix and fortification level) at the LOQ and at the 10-fold LOQ. Additional specimens were kept untreated as blank controls.

A one-point external standard calibration for cis-deltamethrin was carried out. Peak area in counts from

injection of known standards versus standard concentrations in ng/mL was used to quantify the residues in the samples.

Calibration functions obtained from injections of calibration solutions containing eight different concentrations ranging from 2.0 to 500 ng/mL were used to show the linearity of the detector response. Regression coefficients (r2) were ≥ 0.993 for all three fragment ions.

As demonstrated by the independent laboratory validation results, the method allows the determination of cis-deltamethrin with a limit of quantification (LOQ) of 0.01 mg/kg. For all three MS ions monitored, apparent residues in all blank control specimens were below 20 % of the LOQ.

The average recoveries were within the acceptable range of 70 % to 110 % with relative standard deviations (RSD) ≤ 20 %.

Summaries of the independent laboratory validation results are given below:

Table A 47: Recovery results from method validation of cis-deltamethrin using the analytical method in different animal matrices

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
cis-deltamethrin					
Hen's Egg	0.01	5	108	4	m/z=253
	0.10	5	93	14	m/z=253
	0.01	5	110	3	m/z=251
	0.10	5	94	10	m/z=251
	0.01	5	109	2	m/z=181
	0.10	5	93	12	m/z=181
Whole Milk	0.01	5	91	3	m/z=253
	0.10	5	101	8	m/z=253
	0.01	5	95	4	m/z=251
	0.10	5	103	5	m/z=251
	0.01	5	95	3	m/z=181
	0.10	5	105	9	m/z=181
Bovine Muscle	0.01	5	102	6	m/z=253
	0.10	5	105	5	m/z=253
	0.01	5	102	4	m/z=251
	0.10	5	105	9	m/z=251
	0.01	5	103	5	m/z=181
	0.10	5	105	10	m/z=181
Bovine Liver	0.01	5	104	3	m/z=253
	0.10	5	103	3	m/z=253
	0.01	5	103	3	m/z=251
	0.10	5	103	3	m/z=251
	0.01	5	106	5	m/z=181
	0.10	5	102	3	m/z=181
Bovine Kidney	0.01	5	94	7	m/z=253
	0.10	5	107	9	m/z=253

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
	0.01	5	96	9	m/z=251
	0.10	5	100	7	m/z=251
	0.01	5	97	14	m/z=181
	0.10	5	100	7	m/z=181
Bovine Fat	0.01	5	104	4	m/z=253
	0.10	5	101	11	m/z=253
	0.01	5	97	6	m/z=251
	0.10	5	99	8	m/z=251
	0.01	5	98	3	m/z=181
	0.10	5	96	14	m/z=181

Table A 48: Characteristics for the analytical method used for validation of cis-deltamethrin in different animal matrices

	cis-deltamethrin
Specificity	mass spectrum is provided in Figure 3 of the method report blank value < 20 % LOQ)
Calibration (type, number of data points)	A one point external standard calibration individual was carried out. Calibration data presented in Figure 2 of the method report calibration line equation presented for each m/z in Figure 1 of the method report number of data points>5 (8) m/z=253→R = 0.9981 m/z=251→R = 0.9950 m/z=181→R = 0.9934
Calibration range	External standard with one point calibration in the range between of 0.002 to 0.50µg/mL of cis-deltamethrin
Assessment of matrix effects is presented	yes
Limit of determination/quantification	LOQ = 0.01 mg/kg

Conclusion
PTRL Europe performed the independent laboratory validation (ILV) of the DFG method S19 originally validated by Harald Weber (Specht Study No.: BAY-0903V, Bayer CropScience Project No.: P682097525, 07-Jul-09). The method was independently validated successfully for the determination of residues of cis-deltamethrin in/on animal matrices by GC/MS, exemplified for whole milk, hen´s egg, bovine muscle, liver, kidney and fat, demonstrating the LOQ of 0.01 mg/kg.

It is concluded that DFG method S19 fulfils the reproducibility requirements as defined in the Council Directive 91/414/EEC, in the EC Guidance document on residue analytical methods (SANCO/825/00 rev. 7 17/03/04) and in the OECD Guidance Document on Pesticide Residue Analytical Methods (ENV/JM/Mono (2007) 17, 2007-08-13) and is, therefore, applicable as an enforcement method.

A 2.1.2.2.1.3 Confirmatory method (if required)

No confirmatory method is required

Comments of zRMS:	Accepted.

A 2.1.2.2.1.4 Extraction efficiency

Refer to A 2.1.1.1.1.3

A 2.1.2.3 Description of Methods for the Analysis of Body Fluids and Tissues (KCP 5.2)

A 2.1.2.3.1 Analytical method 01127 for the determination of deltamethrin in blood

A 2.1.2.3.1.1 Method validation

Comments of zRMS:	The study of Krebber, R.; 2009 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The analytical method 01127 was succefully validated for the determination of residues of deltamethrin in blood by HPLC-MS/MS.* *Two MRM transitions were monitored for deltamethrin (m/z 523 → m/z 281 for quantitation and m/z 523 → m/z 181 for confirmation).* *The limit of quantitation (LOQ) for deltamethrin in cattle blood was 50 µg/L.* *Mean recoveries for each fortification level and the overall mean recoveries were within the 70% - 110% range for deltamethrin for both MRM transitions. Relative standard deviations were below 20%.* *The study is acceptable.*

Reference:	**KCP 5.2/05**
Title:	Analytical method 01127 for the determination of cyfluthrin and deltamethrin in blood by HPLC-MS/MS
Report:	Krebber, R.; 2009; MR-08/176; M-348630-01-1
Authority registration No:	
Guideline(s):	SANCO/825/00 rev. 7 of March 17, 2004; BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998; EU: 96/46/EC amending Council Directive 91/414/EEC of 16 July 1996
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
This method describes the determination of deltamethrin (among other analytes not reported here) in cattle blood by HPLC-MS/MS and provides validation data for Multiple Reaction Monitoring (MRM) using electrospray in the positive mode. The sample is extracted and deproteinized by mixing with acetonitrile and subsequent centrifugation. The supernatant is diluted with water and the determination is performed by direct injection in an HPLC-MS/MS instrument. Two MRM transitions were monitored for deltamethrin (m/z 523 → m/z 281 for quantitation and m/z 523 → m/z 181 for confirmation). Quantification is carried out by external calibration using matrix-matched standards.

An aliquot of the sample solution was injected into the high performance liquid chromatograph and subjected to reversed phase chromatography coupled with tandem mass spectrometry (MS/MS) with electrospray ionisation. The MS/MS instrument was operated in the Multiple Reaction Monitoring mode (MRM). The pseudomolecular ions of the analytes ([M+H]+, [M-H]- or any adducts) were selected by the first quadrupole. These precursor ions were impulsed with nitrogen in the collision cell (second quadrupole) and the resulting fragment ions (product ions) were separated according to their m/z ratio in the third quadrupole. Two of these product ions per analyte were selected: one product ion (MRM-transition) serving for quantitation and the second for confirmation.

Results and discussions
The results of the method validation are summarized in the tables below.

Table A 49: Recovery results from method validation of deltamethrin using the analytical method 01127

Matrix	Analyte	Fortification level (µg/L) (n = 5)	Recoveries (%) (single values)					Mean (%)	RSD (%)	Overall mean (%)	RSD (%)
Cattle blood	deltamethrin (m/z 523 → m/z 281, quantitation)	50	111	116	118	117	107	114*	4.2	111*	4.1
		500	107	109	110	108	106	108	1.4		
	deltamethrin (m/z 523 → m/z 181, confirmation)	50	109	117	116	116	101	112*	6.0	110	4.8
		500	107	108	109	106	105	107	1.6		

* The mean recovery rate is slightly above the usual acceptance limit of 110%. This is a result of an inaccuracy of the extract volume which is used for calculation. The extract volume is assumed as 1 mL (0.1 mL blood + 0.9 mL acetonitrile). The real volume is less, between 0.8 and 0.9 mL. Therefore the recovery rates rose to values above 100%. In order to enable a simple and fast analysis these higher recovery rates can be accepted.

Table A 50: Characteristics for the analytical method used for validation of deltamethrin residues in cattle blood

	Deltamethrin
Specificity	mass spectrum is provided blank value < 30% LOQ Two MRM transitions were successfully validated. Therefore the HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary. No signals/peaks interfering with the detection of the analytes were observed in solutions of untreated control specimens.
Calibration (type, number of data points)	individual calibration data is presented calibration line equation (1/x weighted): quantitation: y = 26958.4 x – 2832.5, r = 0.9995 confirmation: y = 3017.54 x – 380.025, r = 0.9988 number of data points: ≥ 5
Calibration range	2 – 50 µg/L
Assessment of matrix effects is presented	yes The MS/MS detection of deltamethrin was affected by the matrix. The peak areas of the quantification and confirmatory ion in a matrix matched sample containing 25 µg/L of deltamethrin were about 30% lower than the corresponding peak areas in deionized water. Therefore, quantification was performed using matrix-matched standards.
Limit of determination/quantification	LOQ = 50 µg/L (cattle blood) LOD = 15 µg/L (cattle blood)

Conclusion
The above data is in line with the requirements outlined in the "Guidance document on residue analytical methods, SANCO/825/00 rev. 7". Method 01127 is therefore suitable for the determination of cis-Deltamethrin in blood matrix representative of body fluids.

A 2.1.2.4 Description of Methods for the Analysis of Soil (KCP 5.2)

A 2.1.2.4.1 Analytical method 00877 (including amendment) for the determination of deltamethrin (cis-deltamethrin) and its isomers alpha-R-deltamethrin and trans-deltamethrin in soil and sediment

A 2.1.2.4.1.1 Method validation

Comments of zRMS:	The study of xxx has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below:

	The analytical method 00877 was validated for the determination of the total residue of deltamethrin in soil and sediment by means of HPLC-MS/MS. The total residue is defined as the sum of cis-deltamethrin (AE F032640), α-R-deltamethrin (AE F108569) and trans-deltamethrin (AE F0035073). The method was optimised and validated using cis-deltamethrin. *The limit of quantitation of the method is 0.1 µg/kg for cis-deltamethrin.* *Mean recoveries for each fortification level and the overall mean recoveries were within the 70% - 110% range for deltamethrin. Relative standard deviations were below 20%.* *For confirmation of deltamethrin identity the mass fragments monitored were 523 m/z → 281 m/z. The mass fragments identified for the internal standard ([phenoxy-^{13}C6]deltamethrin) were 529 m/z →281 m/z.* *This method has been satisfactorily validated in accordance with SANCO 3029/99/rev.4 and SANCO/825/00.*

Reference:	**KCP 5.2/06**
Title:	Analytical method 00877 for the determination of total residues of deltamethrin (AE F032640) in / on soil and sediment by HPLC-MS/MS
Report:	xxx; C047210; M-247896-01-1
Authority registration No:	
Guideline(s):	--
Deviations:	--
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.2/07**
Title:	Analytical method 00877 for the determination of total residues of Deltamethrin (AE F032640) in/on soil and sediment by HPLC-MS/MS
Report:	xxx; 00877; M-246580-02-1
Authority registration No:	
Guideline(s):	EC Guidance Document on Residue Analytical Methods, SANCO/825/00 rev.7 of March 17,2004 BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998 Commission Directive 96/46/EC amending Council Directive 91/414/EEC of 16 July 1996 US EPA OPPTS 835.6100, 835.6200
Deviations:	With the exception of recognised differences that exist between the GLP principles/standards of OECD and those FIFRA and JMAFF (for instance, authority granted agency inspectors).
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method 00877 was developed for the determination of deltamethrin (cis-deltamethrin) and its isomers alpha-R-deltamethrin and trans-deltamethrin in soil and sediment. The method was optimised and validated using cis-deltamethrin. Residues were extracted from soil with acetonitrile/10 mM ammonium acetate solution (9/1, v/v) using a microwave extractor. A stable isotopically labelled analyte was added to the extract. After centrifugation at > 12000 G to remove fine soil particles, the extract was analysed without further clean up by HPLC-MS/MS in positive ion mode. Residues were quantified using an internal standard of isotopically labelled [phenoxy-$^{13}C_6$]deltamethrin to eliminate possible matrix effects. The MRM transitions, m/z 523 → 281 for cis-deltamethrin and m/z 529 → 281 for [phenoxy-$^{13}C_6$]deltamethrin, were monitored for each matrix tested.

Results and discussions

The results of the method validation are summarized in the tables below.

Table A 51: Recovery results from method validation of deltamethrin using the analytical method 00877

Matrix	Analyte	Fortification level (µg/kg) (*n* = 5)	Mean recovery (%)	RSD (%)	Overall mean recovery (%)	Overall RSD (%)
Höfchen soil	Deltamethrin m/z 523 → 281	0.1	94.6	3.8	97.6	4.1
		1.0	101	1.5		
Laacher Hof		0.1	91.0	7.7	97.0	8.2
		1.0	103	1.7		
Sediment		0.1	102	5.2	102	3.8
		1.0	101	2.1		
Overall Mean		0.1	95.9	7.3	98.8	5.9
		1.0	102	2.0		

Table A 52: Characteristics for the analytical method used for validation of deltamethrin residues in soil

	deltamethrin
Specificity	mass spectrum is provided blank value < 30% LOQ
Calibration (type, number of data points)	individual calibration data is presented calibration line equation: y = 0.509282 x + 0.00420206 correlation coefficient r = 0.9999 number of data points: 8
Calibration range	0.015 – 5 µg/L in solvent standards, corresponding to a soil concentration of 0.03 – 10 µg/kg
Assessment of matrix effects is presented	Yes Possible matrix effects with deltamethrin were eliminated by using an internal standard solution of an isotopically labelled analytical standard.
Limit of determination/quantification	cis-deltamethrin: LOQ = 0.1 µg/kg LOD = 0.03 µg/kg

Conclusion

The method 00877 is suitable to determine residues of cis-deltamethrin (deltamethrin), as well as its isomers (trans-deltamethrin and alpha-R-deltamethrin) in soil and sediment. The method was optimised and validated using cis-deltamethrin. The LOQ of the method is 0.1 µg/kg and the LOD is 0.03 µg/kg for deltamethrin. This method further fulfils the requirements for the SANCO/825/00 rev. 8.1 and is therefore suitable as enforcement method in soil

A 2.1.2.4.2 Analytical method 01358 for the determination of cis-deltamethrin residues in/on soil/sediment

A 2.1.2.4.2.1 Method validation

Comments of zRMS:	The study of Freitag, T.; 2013 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The analytical method 01358 was succefully validated for the determination of residues of cis-deltamethrin in three different soils by HPLC-MS/MS.* *Two MRM transitions were monitored for each soil tested m/z 523 → 281 for quantitation and m/z 525 → 283 for confirmation of cis-deltamethrin. Therefore, the HPLC- MS/MS method is highly specific and an additional confirmatory method is not necessary.* *The limit of quantitation (LOQ) for each single analyte was 0.2 µg/kg in soil. The limit of determination (LOD) for each single analyte was 0.07 µg/kg.* *Mean recoveries for each fortification level and the overall mean recoveries were within the 70% - 110% range for all soils tested. Relative standard deviations were below 20%.* *The analytical method meets all guideline criteria according to SANCO/825/00 rev. 8.1 and SANCO/3029/99 rev. 4.* *The study is acceptable.*

Reference:	**KCP 5.2/08**
Title:	Analytical method 01358 for the determination of cis-deltamethrin in soil by HPLC-MS/MS
Report:	Freitag, T.; 2013; MR-13/002; M-451547-01-1
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 European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, 11/07/00 Guidance document on residue analytical methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010 US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method
Deviations:	not applicable
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method 01358 developed for the determination of cis-deltamethrin residues in/on soil is proposed as new enforcement method for soil (or sediment, if needed).

Soil samples of 20 g were extracted four times in a laboratory rotating mixer with a total of 100 mL of acetone/formic acid (100/1). The extracts were subjected to a liquid/liquid partition step on a ChemElut *CE 1010 column. After clean up and evaporation to dryness the residues were resolved and subsamples of the extracts are centrifuged to remove fine particles of the soil. Identification and quantitation of the active substance is done by high performance liquid chromatography using MS/MS detection in the Multiple Reaction Monitoring mode.

The method was validated using three different soils "Höfchen", "Laacher Hof", and "Dollendorf".
Two MRM transitions were monitored for each soil tested m/z 523 → 281 for quantitation and m/z 525 →

283 for confirmation of cis-deltamethrin.

Results and discussions

The results of the method validation are summarized in the tables below.

Table A 53: Recovery results from method validation of cis-deltamethrin using the analytical method 01358

Matrix	Analyte	Fortification level (µg/kg) (*n* = 5)	Mean recovery (%)	RSD (%)	Overall mean recovery (%)	Overall RSD (%)
soil Höfchen	cis-deltamethrin m/z 523 → 281	0.2	75	10.5	78	12.5
		2	81	14.0		
soil Dollendorf		0.2	81	14.8	81	10.6
		2	80	5.7		
soil Laacher Hof		0.2	96	13.8	97	9.6
		2	97	4.4		
soil Höfchen	cis-deltamethrin m/z 70 → 43	0.2	86	19.7	85	15.5
		2	84	12.1		
soil Dollendorf		0.2	72	8.0	75	7.3
		2	78	4.1		
soil Laacher Hof		0.2	101	12.8	99	9.1
		2	97	2.2		

Table A 54: Characteristics for the analytical method used for validation of cis-deltamethrin residues in soil

	cis-deltamethrin
Specificity	mass spectrum is provided blank value < 30% LOQ
Calibration (type, number of data points)	individual calibration data is presented calibration line equations: quantitation (m/z 523 → 281): soil Höfchen: y = 14872.1 x + 606.636, r = 0.9920 soil Laacher Hof: y = 13690.8 x + 234.938, r = 0.9922 soil Dollendorf: y = 11186.6 x + 118.005, r = 0.9990 confirmation (m/z 525 → 283) : soil Höfchen: y = 8072.27 x – 102.905 r = 0.9940 soil Laacher Hof: y = 7430.17 x + 694.573, r = 0.9935 soil Dollendorf: y = 6219.77 x + 435.711, r = 0.9982 number of data points: ≥ 6
Calibration range	0.125 – 20 µg/L (matrix standards)
Assessment of matrix effects is presented	matrix matched standards were used
Limit of determination/quantification	LOQ = .2 µg/kg (soil) LOD = 0.07 µg/kg

Conclusion

The method meets all guideline criteria to determine residues of cis-deltamethrin in soil at 0.2 µg/kg and 2 µg/kg for the quantifier mass transition. It is proposed as the new enforcement method for soil (or sediment, if needed). This method further fulfils the requirements for the SANCO/825/00 rev. 8.1 and is therefore suitable as enforcement method in soil/sediment.

A 2.1.2.5 Description of Methods for the Analysis of Water (KCP 5.2)

A 2.1.2.5.1 Analytical method 01383 for the determination of cis-deltamethrin residues in drinking and surface water

A 2.1.2.5.1.1 Method validation

Comments of zRMS:	The study of Krebber, R.; Braune, M.; 2013 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The analytical method 01383 was succefully validated for the determination of residues of cis-deltamethrin in surface water by HPLC-MS/MS using two MRM transitions. Two MRM transitions were monitored for deltamethrin (m/z 523 → m/z 281 for quantitation and m/z 523 → m/z 181 for confirmation), so an additional confirmatory method is not necessary.* *In the SANCO/825/00 rev. 8.1 it is stated that "Provided that a method has been successfully validated for surface water at the LOQ required for drinking water, no further validation in drinking water is required."* *The limit of quantitation (LOQ) for deltamethrin is 0.05 µg/L in surface water.* *Because of the direct measurement of the samples recovery rates cannot be calculated. Thus precision data are presented.* *The relative standard deviations for the peak areas were below 20% for all MRM transitions.* *The analytical method meets all guideline criteria to determine concentrations of deltamethrin in drinking and surface water at 0.05 µg/L according to SANCO/825/00 rev. 8.1 and SANCO/3029/99 rev. 4.* *The study is acceptable.*

Reference:	**KCP 5.2/09**
Title:	Analytical method 01383 for the determination of deltamethrin in drinking and surface water by HPLC-MS/MS
Report:	Krebber, R.; Braune, M.; 2013; MR-13/053; M-464818-01-1
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 Document on Residue Analytical Methods, SANCO/825/00 rev. 8.1 of November 16, 2010 • European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, July 11, 2000
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method 01383 developed for the determination of cis-deltamethrin residues in drinking and surface water is proposed as new enforcement method for water. A validation for drinking water is not necessary because the limit of quantitation for surface water is below the drinking water limit (i.e. < 0.1 µg/L).

Water samples were investigated after addition of acetonitrile and formic acid by direct injection into the HPLC-MS/MS instrument using the positive ion mode for deltamethrin without further clean-up. Concentrations were quantified using external matrix-matched standard solutions.

Two MRM transitions were monitored for deltamethrin (m/z 523 → m/z 281 for quantitation and m/z 523

→ m/z 181 for confirmation).

Results and discussions
The results of the method validation are summarized in the tables below.
Because of the direct measurement of the samples, recovery rates cannot be calculated. Thus precision data are presented.

Table A 55: Recovery/ precision results from method validation of cis-deltamethrin using the analytical method 01383

Matrix	Analyte	Fortification level (µg/L) (n = 10)	Mean value (peak area)	RSD (%)
Surface water	cis-deltamethrin m/z 523 → 281 (quantitation)	0.05	11381	2.1
		0.5	102355	3.7
	cis-deltamethrin m/z 523 → 181 (confirmation)	0.05	1149	3.4
		0.5	10122	3.0

Table A 56: Characteristics for the analytical method used for validation of cis-deltamethrin residues in surface water

	cis-deltamethrin
Specificity	mass spectrum is provided blank value < 30% LOQ no signals/peaks interfering with the detection of the analyte
Calibration (type, number of data points)	individual calibration data is presented calibration line equations (1/x weighted): m/z 523 → 281 (quantitation): $y = 2.71*10^5 x – 503$, r = 0.9997 m/z 523 → 181 (confirmation): $y = 2.64*10^4 x + 170$, r = 0.9996 number of data points: ≥ 6
Calibration range	0.014 – 8 µg/L (standard solutions in a mixture of surface water / acetonitrile / formic acid (800/200/0.2, v/v/v)) corresponding to 0.017 - 10 µg/L in surface water
Assessment of matrix effects is presented	yes The MS/MS detection is affected by the matrix. Matrix effects can be eliminated by using matrix-matched standard solutions.
Limit of determination/quantification	LOQ = 0.05 µg/L (surface water)

Conclusion
The method 01383 meets all guideline criteria of SANCO/825/00 rev. 8.1 to determine concentrations of deltamethrin in drinking and surface water at 0.05 µg/L. It is proposed as the new enforcement method for water samples. Therefore, an independent laboratory validation of method no. 01383 was performed, which is summarized in the following.

A 2.1.2.5.1.2 Independent laboratory validation for method 01383

Comments of zRMS:	The study of Stanislowski, T.; 2013 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The method 01383 was independently validated for the determination of deltamethrin in surface water, using LC/MS/MS (LOQ: 0.05 µg/L).* *Two MRM transitions were monitored for deltamethrin. The results of the independent method validation were confirmed using a 2nd MRM transition for confirmation.* *The method was shown to be selective and yields accurate and repeatable results.* *The validation data can be considered sufficient according to SANCO/825/00 rev. 8.1 for ground and drinking water. All validation parameters were within the required range.* *The study is acceptable.*

Reference:	**KCP 5.2/10**
Title:	Independent laboratory validation of BCS analytical method no. 01383 for the determination of deltamethrin in surface water, using LC/MS/MS
Report:	Stanislowski, T.; 2013; P 3021 G; M-471762-01-1
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 European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, 11/07/00 Guidance document on Pesticide Residue Analytical Methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010 Commission Regulation (EU) No. 283/2013 of 1 March 2013 setting out the data requirements for active substances, in accordance with Regulation (EC) No 1107/2009 of the European Parliament and of the Council concerning the placing of plant protection products on the market.
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method no. 01383 was validated for the determination of cis-deltamethrin in surface water. Water samples were determined after addition of acetonitrile and formic acid by direct injection into the LC-MS/MS instrument using the positive ion mode for deltamethrin without further clean-up. The solution was subjected to HPLC¬MS/MS and the concentrations were quantified using matrix matched standards.

Two MRM transitions were monitored for deltamethrin, m/z 523 → 281 as 1st MRM (quantification) and m/z 523 → 181 as 2nd MRM (confirmation).

Results and discussions

The results of the method validation are summarized in the tables below.
Because of the direct measurement of the samples recovery rates cannot be calculated. Thus precision data are presented.

Table A 57: Recovery/ precision results from independent laboratory validation of cis-deltamethrin using the analytical method 01383

Matrix	Analyte	Fortification level (µg/L) (n = 5)	Mean value (peak area)	RSD (%)
Surface water	cis-deltamethrin m/z 523 → 281 (quantitation)	0.05	7611	6.5
		0.5	86110	4.0
	cis-deltamethrin m/z 523 → 181 (confirmation)	0.05	866	5.5
		0.5	8828	4.0

Table A 58: Characteristics for the analytical method used for independent laboratory validation of cis-deltamethrin residues in surface water

	cis-deltamethrin
Specificity	mass spectrum is provided blank value < 30% LOQ no signals/peaks interfering with the detection of the analyte

	cis-deltamethrin
Calibration (type, number of data points)	individual calibration data is presented calibration line equations (1/x weighted): m/z 523 → 281 (quantitation): y = $1.95*10^5$ x – 102, r = 0.9998 m/z 523 → 181 (confirmation): y = $2.01*10^4$ x + 66.3, r = 0.9999 number of data points: 7
Calibration range	0.012 – 8 µg/L (standard solutions in a mixture of surface water / acetonitrile / formic acid (800/200/0.2, v/v/v)), corresponding to 0.015 - 10 µg/L in surface water
Assessment of matrix effects is presented	Matrix effects were be eliminated by using matrix-matched standard solutions.
Limit of determination/quantification	LOQ = 0.05 µg/L (surface water)

Conclusion
The method 01383 meets all guideline criteria of SANCO/825/00/rev. 8.1 to determine concentrations of deltamethrin in surface water at 0.05 µg/L. It can be used as enforcement method for water samples.

A 2.1.2.5.2 Analytical method 00886 for the determination of deltamethrin (cis-deltamethrin) and its isomers α-*R*-deltamethrin and trans-deltamethrin in surface water

A 2.1.2.5.2.1 Method validation

Comments of zRMS:	The study of xxx has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The method 00886 was validated for the determination of total residues of deltamethrin in surface water by means of HPLC-MS/MS. The total residue is defined as the sum of cis-deltamethrin (AE F032640), α-R-deltamethrin (AE F108569) and trans-deltamethrin (AE F0035073).* *It is known that cis-deltamethrin can be transformed by chemical or biological processes to its diasteromeres α-R-deltamethrin and trans-deltamethrin. For ecotoxicological risk assessment purposes α-R- and trans-deltamethrin are assumed to have the same biological/pesticidal activity as cis-deltamethrin. This method is able to quantify cis-deltamethrin as well as its isomers trans-deltamethrin and α-R-deltamethrin. The method was optimised and validated using cis-deltamethrin.* *For confirmation of deltamethrin identity the mass fragments monitored were 523 m/z → 281 m/z. The mass fragments identified for the internal standard ([phenoxy-^{13}C6]deltamethrin) were 529 m/z →281 m/z.* *The limit of quantitation for cis-deltamethrin in surface water is 0.005 µg/L.* *The limit of detection in surface water is 0.002 µg/L.* *The individual and average recoveries at each fortification level were within the range of 70 – 110% for deltamethrin. The RSD values were below 20%.* *This method has been satisfactorily validated in accordance with SANCO 3029/99/rev.4.* *The study is acceptable.*

Reference:	**KCP 5.2/11**
Title:	Analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS
Report:	xxx; C047388; M-248040-01-1
Authority registration No:	
Guideline(s):	--
Deviations:	--
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
The analytical method was developed for the determination of deltamethrin (cis-deltamethrin) and its isomers α-*R*-deltamethrin and trans-deltamethrin in surface water. The method was optimised and validated using deltamethrin. Water samples were diluted with 20% acetonitrile, and then ammonium acetate added to give a 10 mM solution. A stable isotopically labelled analyte was added to the sample, which was then analysed directly by HPLC-MS/MS in positive ion mode. Residues were quantified using an internal standard of isotopically labelled [phenoxy-$^{13}C_6$]deltamethrin to minimise possible matrix effects.
The MRM transitions, m/z 523 → 281 for cis-deltamethrin and m/z 529 → 281 for [phenoxy-$^{13}C_6$]deltamethrin, were monitored for identification and quantification.

Results and discussions
The results of the method validation are summarized in the tables below.

Table A 59: Recovery results from method validation of cis-deltamethrin using the analytical method 00886

Matrix	Analyte	Fortification level (µg/L) (n = 10)	Mean recovery (%)	RSD (%)	Overall mean recovery (%)	Overall RSD (%)
River water	cis-deltamethrin m/z 523 → 281	0.0059	100	5.7	100	4.2
		0.059	100	1.8		

Table A 60: Characteristics for the analytical method used for validation of cis-deltamethrin residues in surface water

	cis-deltamethrin
Specificity	mass spectrum is provided blank value < 30% LOQ
Calibration (type, number of data points)	individual calibration data is presented calibration line equation (1/x weighted): y = 0.746692 x + 0.0053296 correlation coefficient r = 0.99905 number of data points: 6
Calibration range	0.004 – 118.1 µg/L
Assessment of matrix effects is presented	yes The MS/MS detection of cis-deltamethrin was affected by the matrix. Possible matrix effects were minimised by using an internal standard solution of an isotopically labelled analytical standard
Limit of determination/quantification	LOQ = 0.005 µg/L (surface water) LOD = 0.002 µg/L

Conclusion
The method is suitable to determine residues of deltamethrin, as well as its isomers trans-deltamethrin and α-*R*-deltamethrin in surface water. The method was optimised and validated using deltamethrin. The LOQ of the method is 0.005µg/L and the LOD is 0.002 µg/L for deltamethrin.

A 2.1.2.5.3 Analytical method 00886/M001 for the determination of deltamethrin residues in surface water

A 2.1.2.5.3.1 Method validation

Comments of zRMS:	The study of Krebber, R.; Braune, M.; 2007 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The method 00886 developed for total residues of deltamethrin in surface water was*

	modified. The analytical method 00886/M001 was validated for the determination of deltamethrin residues in surface water. *The limit of quantitation (LOQ) for deltamethrin was 2 ng/L.* *For confirmation of deltamethrin identity the mass fragments monitored were 523 m/z → 281 m/z. The mass fragments identified for the internal standard ([phenoxy-^{13}C6]deltamethrin) were 529 m/z →281 m/z.* *The individual and average recoveries at each fortification level were within the range of 70 – 110% for deltamethrin. The RSD values were below 20%.* *This method has been satisfactorily validated in accordance with SANCO 3029/99/rev.4.* *The study is acceptable.*

Reference:	**KCP 5.2/12**
Title:	Modification M001 of analytical method 00886 for the determination of total residues of deltamethrin (AE F032640) in surface water by HPLC-MS/MS
Report:	Krebber, R.; Braune, M.; 2007; 00886/M001; M-291746-01-1
Authority registration No:	
Guideline(s):	US EPA OPPTS 835.6100, 835.6200
Deviations:	not specified
GLP/GEP:	no
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The analytical method 00886/M001 was validated for the determination of deltamethrin residues in surface water. It is a modification of the method developed for total residues of deltamethrin in surface water. Water samples were adjusted to pH 4 with formic acid, and then diluted with acetonitrile (1:1, v/v). A stable isotopically labelled analyte was added to the sample, which was then analysed directly by HPLC-MS/MS in positive ion mode. Residues were quantified using an internal standard of isotopically labelled [phenoxy-$^{13}C_6$]deltamethrin.

The MRM transitions, m/z 523 → 281 for deltamethrin and m/z 529 → 281 for [phenoxy-$^{13}C_6$]deltamethrin, were monitored for identification and quantification.

Results and discussions

The results of the method validation are summarized in the tables below.

Table A 61: Recovery results from method validation of deltamethrin using the analytical method 0086/M001

Matrix	Analyte	Fortification level (µg/L) (n = 10)	Mean recovery (%)	RSD (%)	Overall mean recovery (%)	Overall RSD (%)
Surface Water	Deltamethrin m/z 523 → 281	0.002	105	2.4	101	4.6
		0.01	98	3.3		

Table A 62: Characteristics for the analytical method used for validation of deltamethrin residues in water

	deltamethrin
Specificity	mass spectrum is provided No residues of deltamethrin were detected in the test water control samples.
Calibration (type, number of data points)	individual calibration data is presented calibration line equation (1/x weighted): y = 0.00195024 x + 0.957351 correlation coefficient r = 0.9995 number of data points: 5
Calibration range	2 – 100 ng/L (solvent standards)

	deltamethrin
Assessment of matrix effects is presented	yes The MS/MS detection of deltamethrin was not affected by the matrix.
Limit of determination/quantification	LOQ = 0.002 µg/L (test water) LOD = 0.001 µg/L

Conclusion
The method is suitable to determine residues of deltamethrin in surface water. The LOQ of the method is 0.002 µg/L and the LOD is 0.001 µg/L.

A 2.1.2.6 Description of Methods for the Analysis of Air (KCP 5.2)

No new or additional studies have been submitted.

A 2.1.2.7 Other Studies/ Information

None.

A 2.2 Analytical methods for Flupyradifurone

A 2.2.1 Methods used for the generation of pre-authorization data (KCP 5.1)

A 2.2.1.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.1)

A 2.2.1.1.1 Analytical method 1 (Method 01304)

The analytical method 01304 was fully validated for the determination of residues of flupyradifurone and its metabolite difluoroacetic acid (DFA) in the representative crop matrices dried bean seed, cereal forage, orange fruit, soybean seed, tomato fruit and wheat grain, as reported in the DAR (NL, 2015) and the EFSA Conclusion on the peer review (EFSA Journal 2015;13(2):4020). For the crop submitted in this application, additional validations were conducted with a limited dataset of recoveries during the conduct of the residue studies. Information on the additional recoveries is given below.

A 2.2.1.1.1.1 Method validation

Comments of zRMS:	The analytical method 01304 has been validated for the determination of residues of flupyradifurone and its metabolite difluoroacetic acid (DFA) in additional matrices: sunflower, barley, wheat, maize with following LOQs: FPF: 0.01 mg/kg (expressed as parent) DFA: 0.02 mg/kg (expressed as parent) except in study S13-03307 where the LOQ is 1.0 mg/kg. For all studies the mean recoveries at each fortification level were within the 70 - 110% range. The RSDs were always below 20%. The studies are acceptable.

Primary crop studies

Reference:	**KCP 5.1/07**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in northern France, Hungary, The United Kingdom and Poland
Report:	Miara, C.; Kowalski, N.; 2018; 16-2145; M-645130-01-1
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 Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/06**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in Italy, southern France, Spain and Greece
Report:	Kaussmann, M.; Kowalski, N.; 2018; 16-2194; M-634135-01-1
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:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/08**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on sunflower after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy
Report:	Kaussmann, M.; Kowalski, N.; 2018; 16-2195; M-629954-01-1
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 Guideline No. 860.1500 on Crop Field Trial
Deviations:	None
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/11**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy, Spain and Greece
Report:	Kaussmann, M.; Miara, C.; 2018; 16-2034; M-634112-01-1
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:	Yes (see report)
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/12**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring barley after spray application of deltamethrin & flupyradifurone EC 085 in the Netherlands, Germany and Belgium
Report:	Kaussmann, M.; 2018; 16-2035; M-634410-01-1
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):	

Reference:	**KCP 5.1/15**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in southern France, Italy and Spain
Report:	Kaussmann, M.; Kerkering, S.; 2018; 16-2032; M-633925-01-1
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:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/16**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring wheat after spray application of deltamethrin & flupyradifurone EC 085 in Belgium, Germany and the Netherlands
Report:	Kaussmann, M.; Kerkering, S.; 2018; 16-2033; M-634190-01-1
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):	

Reference:	**KCP 5.1/18**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and the Netherlands
Report:	Schulte, G.; Kerkering, S.; 2018; 16-2192; M-628803-01-1
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):	

Storage stability study

Reference:	**KCP 5.1/23**
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, see below in point A 2.2.1.1.3.3.1
Duplication (if vertebrate study):	

Results and discussions

For matrices relevant in the residue studies submitted in this dRR, but not included in the original validation of method 01304, additional valididation set were conducted in the course of the residue studies. These additional validation recoveries are presented in the table below, if not already evaluated at EU level.

Studies 16-2194, 16-2195, 16-2034 and 16-2032 are only submitted for information purposes.

Table A 63: Recovery results from method validation of flupyradifurone using the analytical method 01304 in various primary crops

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
Flupyradifurone					
sunflower / seed	0.01	3	97	1.8	m/z 289 → 126 (16-2145)
	0.10	3	94	5.0	
	0.01	3	100	1.5	m/z 289 → 126 (16-2195)
	0.10	3	99	4.4	
	0.01	3	93	2.5	m/z 289 → 126 (16-2194)
	0.10	3	89	0.0	
sunflower / kernel	0.01	3	94	2.1	m/z 289 → 126 (16-2145)
	0.10	3	94	2.1	

	0.01	3	107	1.6	m/z 289 → 126 (16-2195)
	0.10	3	100	4.6	
	0.01	3	96	1.6	m/z 289 → 126 (16-2194)
	0.10	3	96	1.6	
barley / whole plant without root	0.01	5	98	4.7	m/z 289 → 126 (16-2034, 16-2035)
	0.10	4	105	7.4	
barley / grain	0.01	5	102	6.9	m/z 289 → 126 (16-2034, 16-2035)
	0.10	4	105	6.6	
barley / straw	0.01	5	99	8.1	m/z 289 → 126 (16-2034, 16-2035)
	0.10	4	104	9.5	
wheat / whole plant without root	0.01	6	103	4.3	m/z 289 → 126 (16-2032, 16-2033)
	0.10	6	102	5.3	
wheat / grain	0.01	6	102	3.7	m/z 289 → 126 (16-2032, 16-2033)
	0.10	6	102	6.7	
wheat / straw	0.01	6	99	4.1	m/z 289 → 126 (16-2032, 16-2033)
	0.10	6	99	8.2	
maize/corn / green material	0.01	3	107	1.6	m/z 289 → 126 (16-2192)
	0.10	3	104	2.5	
maize/corn / kernel	0.01	3	102	2.6	m/z 289 → 126 (16-2192)
	0.10	3	104	0.6	
maize/corn / rest of plant	0.01	3	101	3.0	m/z 289 → 126 (16-2192)
	0.10	3	103	2.8	

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
DFA					
sunflower / seed	0.02	3	80	9.3	m/z 95 → 51 (16-2145)
	0.20	3	81	2.6	
	0.02	3	83	2.5	m/z 95 → 51 (16-2195)
	0.20	3	79	13.2	
	0.02	3	97	5.1	m/z 95 → 51 (16-2194)
	0.20	3	73	0.0	
sunflower / kernel	0.02	3	96	5.2	m/z 95 → 51 (16-2145)
	0.20	3	76	2.8	
	0.02	3	101	5.7	m/z 95 → 51 (16-2195)
	0.20	3	86	0.7	
	0.02	3	82	4.4	m/z 95 → 51 (16-2194)
	0.20	3	77	2.0	
barley / whole plant without root	0.02	5	95	5.1	m/z 289 → 126 (16-2034, 16-2035)
	0.20	4	94	5.0	
braley / grain	0.02	5	94	4.4	m/z 289 → 126 (16-2034, 16-2035)
	0.20	4	86	4.9	
barley / straw	0.02	5	89	8.6	m/z 289 → 126 (16-2034, 16-2035)
	0.20	4	93	5.2	
wheat / whole plant without root	0.02	6	97	4.5	m/z 289 → 126 (16-2032, 16-2033)
	0.20	6	90	3.8	
wheat / grain	0.02	6	95	4.3	m/z 289 → 126 (16-2032, 16-2033)
	0.20	6	83	2.5	
wheat / straw	0.02	6	100	4.2	m/z 289 → 126 (16-2032, 16-2033)
	0.20	6	92	2.2	
maize/corn green material	0.02	3	87	2.0	m/z 95 → 51 (16-2192)
	0.20	3	88	2.4	
maize/corn / kernel	0.02	3	95	2.6	m/z 95 → 51 (16-2192)
	0.20	3	81	3.8	
Maize/corn / rest of plant	0.02	3	102	2.9	m/z 95 → 51 (16-2192)
	0.20	3	91	1.7	
Tomato / fruit	1.0	5	104	4.1	m/z 95 → 51 (S13-03307)
Wheat / green material	1.0	5	93	12	m/z 95 → 51 (S13-03307)
Wheat / grain	1.0	5	104	11	m/z 95 → 51 (S13-03307)
Grapes / bunches	1.0	5	100	14	m/z 95 → 51 (S13-03307)
Potato / tuber	1.0	5	88	3.5	m/z 95 → 51 (S13-03307)
Pea/ dried	1.0	5	87	15	m/z 95 → 51 (S13-03307)

Note: in several cases RSD values were recalculated based on rounded values available in the report.

In the primary crop studies and the storage stability study, the apparent residues in the control samples used for the recoveries were below 30%. Recoveries were not corrected for apparent residues.
For all studies the mean recoveries at each fortification level were within the 70 - 110% range. The RSDs were below 20%.

The limit of quantification (LOQ) for FPF is 0.01 mg/kg. The LOQ for DFA is 0.02 mg/kg (expressed as parent) except in study S13-03307 where the LOQ is 1.0 mg/kg for all matrices.

Table A 64: Characteristics for the analytical method 01304 used for validation of flupyradifurone and its metabolite residues

	Flupyradifurone and DFA
Specificity	Mass spectra are provided in Appendix of the original EU reviewed method 01304 blank values < 30% LOQ
Calibration (type, number of data points)	Calibration data presented Calibration line presented number of data points ≥ 5 R > 0.98
Calibration range (maximum range over all studies)	Range for all studies: 0.125 to 100 µg/L (flupyradifurone) and 0.08 to 66.6 µg/L (DFA, expressed as parent), standards in solvent with internal standards. Corresponding calibration range in mass ratio units: 0.0025 mg/kg to 2 mg/kg (flupyradifurone) and 0.005 to 4 mg/kg (DFA, expressed as parent). The range may differ in single studies with in-study validations.
Assessment of matrix effects is presented	No. The use of stable isotopically labelled internal standards compensates for matrix effect.
Limit of determination/quantification	FPF: 0.01 mg/kg (expressed as parent) DFA: 0.02 mg/kg (expressed as parent) except in study S13-03307 where the LOQ is 1.0 mg/kg

Conclusion
All method validation data are in compliance with the guideline requirements for data generation methods. Method 01304 can therefore be considered successfully validated for the determination of parent flupyradifurone and its metabolite DFA in all additional plant matrices relevant to this submission.

A 2.2.1.1.2 Analytical method 2 (Method 01212)

The analytical method 01212 was fully validated for the determination of residues of flupyradifurone and its metabolite difluoroacetic acid (DFA) in the representative plant matrices tomato fruit, grape and bunch of grape, kidney bean dry seed, barley grain and summer rape seed, as reported in the DAR (NL, 2015) and the EFSA Conclusion on the peer review (EFSA Journal 2015;13(2):4020). For the crop submitted in this application, additional validations were conducted with a limited dataset of recoveries during the conduct of the residue studies. The validation data are shown below

A 2.2.1.1.2.1 Method validation

Comments of zRMS:	The analytical method 01212 has been validated for the determination of residues of flupyradifurone and its metabolite difluoroacetic acid (DFA) in additional matrices: grapes, barley, wheat and corn with following LOQs: FPF: 0.01 mg/kg (expressed as parent) except in barley (straw) and wheat (straw) where the LOQ is 0.05 mg/kg; DFA:0.02 mg/kg (expressed as parent) except in barley (straw) and wheat (straw) where the LOQ is 0.05 mg/kg. For all studies the mean recoveries (or mean corrected recoveries) at each fortification level were within the 70 - 110% range. The RSDs were always below 20%. The studies are acceptable.

Reference:	**KCP 5.1/47**
Title:	Determination of the residues of BYI 02960 in/on grape after high and low-volume spray application of BYI 02960 SL 200 in southern France, Spain, Italy and Greece
Report:	Meilland-Berthier, I.; 2014; 12-2126; M-479360-01-1
Authority registration No:	
Guideline(s):	EC Guidance working document 7029/VI/95 rev.5 (1997-07-22), 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 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial US EPA OCSPP Guideline No. 860.1500
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/05**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on grape after high and low-volume spray application of deltamethrin & flupyradifurone EC 085 in Germany and France (North)
Report:	Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.; 2016; 14-2096; M-559743-01-1
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 Guideline No. 860.1500 on Crop Field Trials
Deviations:	yes, see report
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/04**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on grape after high or low-volume spray application of deltamethrin & flupyradifurone EC 085 in southern France, Spain and Italy
Report:	Schoening, R.; Bouhamadi, S.; Sosniak, A.; Czaja, C.; 2016; 14-2095; M-560047-01-1
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 Guideline No. 860.1500 on Crop Field Trials
Deviations:	yes, see report
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/09**
Title:	Determination of the residues of BYI 02960 and deltamethrin in/on barley after spray application of deltamethrin & flupyradifurone EC 085 in France (South), Italy, Spain and Greece
Report:	Noss, G.; 2017; 15-2130; M-572779-03-1
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:	not specified
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/10**
Title:	Amendment no. 3 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on winter and spring barley after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and United Kingdom
Report:	Schulte, G.; 2017; 15-2131; M-580973-04-1
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:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/13**
Title:	Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on wheat after spray application of deltamethrin & flupyradifurone EC 085 in Italy, Spain and Portugal
Report:	Schulte, G.; 2017; 15-2127; M-580063-03-1
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:	none
GLP/GEP:	yes
Acceptability:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/14**
Title:	Amendment no. 2 to final report - Determination of the residues of BYI 02960 and deltamethrin in/on spring wheat and winter wheat after spray application of deltamethrin & flupyradifurone EC 085 in Germany, the Netherlands and Belgium
Report:	Schulte, G.; 2017; 15-2129; M-580528-03-1
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:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/17**
Title:	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Spain, France (South) and Italy
Report:	Schulte, G.; 2017; 15-2133; M-574144-02-1
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:	Not evaluated
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/20**
Title:	Amendment no. 1: Determination of the residues of BYI 02960 and deltamethrin in/on maize/corn after spray application of deltamethrin & flupyradifurone EC 085 in Germany, Belgium and the Netherlands
Report:	Schulte, G.; 2017; 15-2134; M-574350-02-1
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):	

Results and discussions

For matrices relevant in the residue studies submitted in this dRR, but not included in the original validation of method 01212, a limited dataset of validation recoveries (at least three repetitions at two fortification levels) was conducted. These additional validation recoveries are presented in the table below, if not already evaluated at EU level.

Studies 12-2126, 14-2095, 15-2130, 15-2127 and 15-2133 are only submitted for information purposes.

Table A 65: Recovery results from method validation of flupyradifurone using the analytical method 01212 in various primary crops

Matrix	Fortification level (mg/kg)	n	Mean recovery (%)	RSD (%)	Comments
Flupyradifurone					
Grapes / bunch of grapes	0.01	5	101	4.8	m/z 289 → 126 (14-2096, 14-2095)
	0.10	5	106	5.3	
Grapes / berries	0.01	3	96	2.6	m/z 289 → 126 (12-2126, 14-2096, 14-2095)
	0.10	3	94	1.2	
Barley / whole plant without root	0.01	3	100	1.0	m/z 289 → 126 (15-2130, 15-2131)
	0.10	3	99	0.0	
Barley straw	0.05	3	92	5.0	m/z 289 → 126 (15-2130, 15-2131)
	0.50	3	94	2.1	
Wheat / whole plant without root	0.01	3	98	4.1	m/z 95 → 51 (15-2127, 15-2129)
	0.10	3	101	1.5	
Wheat / grain	0.01	3	105	6.4	m/z 95 → 51 (15-2127, 15-2129)
	0.10	3	107	2.2	
Wheat / straw	0.05	5	102[(1)]	6.3	m/z 95 → 51 (15-2127, 15-2129)
	0.50	5	100	2.3	
Maize/corn / green material	0.01	3	101	1.1	m/z 95 → 51 (15-2133, 15-2134)
	0.10	3	97	3.6	
Maize/corn / kernel	0.01	3	98	1.8	m/z 95 → 51 (15-2133, 15-2134)
	0.10	3	96	6.5	
Maize/corn / rest of plant	0.01	3	92	2.7	m/z 95 → 51 (15-2133, 15-2134)
	0.10	3	89	6.3	

DFA					
Grapes / bunch of grapes	0.02	5	101	3.4	m/z 289 → 126 (14-2096)
	0.20	5	101	2.7	
Grapes / berries	0.02	3	95	1.1	m/z 289 → 126 (12-2126, 14-2096, 14-2095)
	0.20	3	93	1.6	
Barley / whole plant without root	0.02	3	105	1.9	m/z 289 → 126 (15-2130, 15-2131)
	0.20	3	94	1.2	
Barley straw	0.05	3	85	2.4	m/z 289 → 126 (15-2130, 15-2131)
	0.50	3	93	0.0	
Wheat / whole plant without root	0.02	3	88[(2)]	4.0	m/z 95 → 51 (15-2127, 15-2129)
	0.20	3	91	1.9	
Wheat / grain	0.02	3	82	6.7	m/z 95 → 51 (15-2127, 15-2129)
	0.20	3	79	1.5	
Wheat / straw	0.05	5	86[(3)]	4.0	m/z 95 → 51 (15-2127, 15-2129)
	0.50	5	96	2.7	
Maize/corn / green material	0.02	3	98	4.3	m/z 95 → 51 (15-2133, 15-2134)
	0.20	3	93	2.2	
Maize/corn / kernel	0.02	3	95	4.7	m/z 95 → 51 (15-2133, 15-2134)
	0.20	3	86	2.4	
Maize/corn / rest of plant	0.02	3	100	2.1	m/z 95 → 51 (15-2133, 15-2134)
	0.20	3	90	1.1	

(1) The shown mean recovery was calculated from corrected values (107%, 104%, 102%, 106%, 91%); uncorrected values are 119%, 116%, 115%, 119% and 104%

(2) The shown mean recovery was calculated from corrected values (91%, 88%, 84%) uncorrected values are 131%, 128% and 124%

(3) The shown mean recovery was calculated from corrected values (83%, 88%, 85%, 91%, 83%) uncorrected values are 117%, 121%, 118%, 124%, and 116%

In the primary crop studies (grapes, barley, wheat and corn), the apparent residues in the control samples used for the recoveries were below 30%. Recoveries were not corrected for apparent residues, except residues of FPF and DFA in wheat, straw in study 15-2127, where residues of FPF were found at 0.00666 mg/kg in one control sample and at 0.0167 mg/kg of DFA expressed as parent equivalents. In the second control sample of wheat straw, DFA residues amounted to 0.0172 mg/kg, expressed as parent equivalents. Therefore recoveries at 0.05 mg/kg were corrected for the apparent residues. The same was true for wheat, whole plant without root in the same study, where residues of DFA were found at 0.008 mg/kg expressed as parent equivalents and therefore recoveries were also corrected.
For all studies the mean recoveries (or mean corrected recoveries) at each fortification level were within the 70 - 110% range. The RSDs were always below 20%.

The limit of quantification (LOQ) for FPF is 0.01 mg/kg except in barley (straw) and wheat (straw) where the LOQ is 0.05 mg/kg. The LOQ for DFF is 0.02 mg/kg except in barley (straw) and wheat (straw) where the LOQ is 0.05 mg/kg.

Table A 66: Characteristics for the analytical method 01212 used for validation of flupyradifurone and its metabolite residues

	Flupyradifurone and DFA
Specificity	Mass spectra are provided in Appendix of the original EU reviewed method 01212 blank values < 30% LOQ

	Flupyradifurone and DFA
Calibration (type, number of data points)	Calibration data presented Calibration line presented number of data points ≥ 5 R > 0.99
Calibration range	Range for all studies: 0.05 to 20 µg/L (flupyradifurone) and 0.0166 to 16.6 µg/L (DFA, expressed as parent), standards in solvent with internal standards. Corresponding calibration range in mass ratio units: 0.002 mg/kg to 0.8 mg/kg (flupyradifurone) and 0.002 to 2 mg/kg (DFA, expressed as parent). The range may differ in single studies with in-study validations.
Assessment of matrix effects is presented	No. The use of stable isotopically labelled internal standards compensates for matrix effect.
Limit of determination/quantification	FPF: 0.01 mg/kg (expressed as parent) except in barley (straw) and wheat (straw) where the LOQ is 0.05 mg/kg DFA: 0.02 mg/kg (expressed as parent) except in barley (straw) and wheat (straw) where the LOQ is 0.05 mg/kg

Conclusion
All method validation data are in compliance with the guideline requirements for data generation methods. Method 01212 can therefore be considered successfully validated for the determination of parent flupyradifurone and its metabolite DFA in all additional plant matrices relevant to this submission.

A 2.2.1.1.3 Analytical method 3 (01207)

A 2.2.1.1.3.1 Method validation (tomato fruit, wheat green material and grain, grape bunches, potato tuber and dry peas)

Comments of zRMS:	The study of Lakaschus, S.; Gizler, A.; 2017 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The analytical method BCS 01207 was validated for the determination of flupyradifurone in/on samples of tomato (fruit), wheat (green material), onion (bulbs), grape (bunches), wheat (grain), potato (tuber), peas (dry peas) and oilseed rape (seeds).* *The LOQ of BCS Method 01207 is defined as 0.01 mg/kg as validated in study S10-00279. This analyte not validated within study S10-00279 were validated within this study (reduced validation set – 5 samples fortified at 1.0 mg/kg).* *LOQ: 1 mg/kg.* *Mean recoveries were within the 70 - 110% range. The RSD values were well below 20%.* *Amendment No. 3 is written to provide additional information for the validation of flupyradifurone* *1. On request of the sponsor a full scan spectrum and the product ion spectra of flupyradifurone are added to the report.* *2. On request of the sponsor the linearity ranges for flupyradifurone are expressed as mass fractions of the original sample in mg/kg and the percentage of the fortification level at the lower and upper level is calculated for the used linearity curves of flupyradifurone.* *3. Starting 2017-01-01 the name of the sponsor changed.* *Accepted.*

Reference:	**KCP 5.1/23**
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 data generation method 01207 (based on QuEChERS) was validated for the determination of residues of flupyradifurone in tomato fruit, wheat green material and grain, grape bunches, potato tuber and dry peas within the storage stability study S13-03307 (Lakaschus, S.; Gizler, A.; 2017; M-480441-06-1).
The LOQ of BCS Method 01207 is defined as 0.01 mg/kg as validated in study S10-00279 (Lakaschus, S.; Amann, S.; Winter, O.; Gizler, A.; 2013; M-424756-02-1). The analytes (FPF and its metabolites) not validated within study S10-00279 were validated within the present study. A reduced set of validation data was conducted in the different matrices at the spiking level relevant for the storage stability study (1 mg/kg).

For the analysis of flupyradifurone, the water content of different sample materials was adjusted to 5 g followed by the addition of acetonitrile, leading to a acetonitrile/water ratio of (4/1, v/v) followed by shaking. Thereafter the samples were left to soak under solvent for 15 minutes. After shaking 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 followed by shaking again and centrifugation. A defined aliquot was filled up with methanol/water (1/1, v/v) to a defined volume and internal standard solution was added. The residues of flupyradifurone were quantified using reversed HPLC and MS/MS detection.

For the determination of flupyradifurone a high performance liquid Chromatograph was used with a reversed phase chromatography system (Phenomenex Luna C18) coupled with tandem mass spectrometry (MS/MS) with electrospray ionisation (Applied Biosystems API 5500 Triple Quadrupole Mass Spectrometer, AB Sciex Instruments, using Analyst version 1.6.1) operated in the positive ion mode. The MS/MS instrument was operated in the Multiple Reaction Monitoring mode (MRM). One MRM transition was monitored for quantification, m/z 289 → 126, a second one for confirmation, m/z 289 → 90.

Results and discussions

The validation described within the storage stability study S13-03307 using method 01207 was performed on tomato, fruit, wheat, green material, wheat grain, grape, bunches, potato tuber and peas, dried. Mean recoveries were within the 70 - 110% range. The RSD values were well below 20%. The results are summarized in the table below.

Table A 67: Recovery results from method validation of flupyradifurone using the analytical method 01207

Matrix	Fortification level (mg/kg)	Single recoveries (%)	n	Mean recovery (%)	RSD (%)	Comments
Flupyradifurone						
Tomato, fruit	1.0	96, 100, 97, 93, 91	5	95	3.7	m/z 289 → 126
	1.0	105, 117, 75, 99, 89	5	97	16	m/z 289 → 90

Matrix	Fortification level (mg/kg)	Single recoveries (%)	n	Mean recovery (%)	RSD (%)	Comments
Wheat, green material	1.0	107, 100, 114, 97, 85	5	101	11	m/z 289 → 126
	1.0	104, 95, 114, 103, 104	5	104	6.5	m/z 289 → 90
Wheat grain	1.0	98, 119, 110, 103, 112	5	108	7.5	m/z 289 → 126
	1.0	97, 111, 114, 103, 112	5	107	6.7	m/z 289 → 90
Grape, bunches	1.0	97, 95, 128, 97, 81	5	100	17	m/z 289 → 126
	1.0	104, 104, 131, 95, 80	5	103	18	m/z 289 → 90
Potato tuber	1.0	98, 89, 94, 71, 72	5	85	15	m/z 289 → 126
	1.0	84, 85, 96, 71, 69	5	81	14	m/z 289 → 90
Peas, dried	1.0	97, 96, 102, 105, 96	5	99	4.1	m/z 289 → 126
	1.0	93, 86, 103, 87, 97	5	93	7.6	m/z 289 → 90

Fortification level expressed as flupyradifurone parent equivalents

Table A 68: Characteristics for the analytical method used for validation of residues in different plant commodities

	Flupyradifurone (FPF)
Specificity	blank value < 30% of fortification level of 1.0 mg/kg mass spectra are presented within the report S13-03307
Calibration (type, number of data points)	1/x weighted linear regression individual calibration data and calibration line equation presented; number of data points ≥5; $r \geq 0.99$
Calibration range	Accepted calibration range in concentration units : Tomato fruit, wheat green material, bunches of grape and potato tuber: 0.75-10 µg/L corresponding calibration range in mass ratio units for the sample: 0.30-4.0 mg/kg Wheat grain: 0.75-12 µg/L corresponding calibration range in mass ratio units for the sample: 0.30-4.8 mg/kg Dried peas: 1.0-20 µg/L corresponding calibration range in mass ratio units for the sample: 0.40-8.0 mg/kg
Assessment of matrix effects is presented	Matrix effects are compensated by using matrix-matched standards
Limit of quantification	LOQ: 1 mg/kg

Conclusion

The data collection method 01207 (based on QuEChERS) meets nearly all necessary performance requirements to determine residues of flupyradifurone in tomato fruit, wheat, green material and grain, grapes, potatoes and dry peas (pulses) with the limit of quantification of 1 mg/kg for the stability study.
The exception according to SANCO/3029/99 rev. 4 is that recoveries were only performed at 1 fortification level. Despite this exception, all data presented confirmed the accuracy and linearity of this method for the determination of FPF and its metabolites according to SANCO/3029/99 rev. 4. Therefore, it can be considered as fit for purpose with regard to the presented stability study.

A 2.2.1.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.1)

No new or additional studies have been submitted.

A 2.2.1.3 Description of analytical methods for the determination of residues in support to environmental fate studies (KCP 5.1)

No new or additional studies have been submitted.

A 2.2.1.4 Description of analytical methods for the determination of residues in support to toxicological studies (KCP 5.1)

No new or additional studies have been submitted.

A 2.2.1.5 Description of analytical methods for the determination of residues in support of operator, worker, resident and bystander exposure studies (KCP 5.1)

No new or additional studies have been submitted.

A 2.2.1.6 Description of analytical methods for the determination of residues in of ecotoxicology studies (KCP 5.1)

A 2.2.1.6.1 Analytical method in support of the study M-548840-01-1

A 2.2.1.6.1.1 Method validation

Comments of zRMS:	The analytical method has been validated for the determination of flupyradifurone in test water samples according to the SANCO/3029/99 rev. 4. The LOQ was 0.10 µg/L. The mean recovery was 96% with an overall relative standard deviation (RSD) of 2.2%. The study is acceptable.

Reference:	**KCP 5.1/48**
Title:	Acute toxicity of deltamethrin + flupyradifurone EC 85 (10+75 g/L) to the rainbow trout (Oncorhynchus mykiss) under static conditions
Report:	xxx; 007SRLS15C08; M-548840-01-1
Authority registration No:	
Guideline(s):	OCSPP Guideline 850.1075, OECD Guideline 203. The afore mentioned guidelines were harmonized for various test parameters (i.e. temperature, light, etc.) to achieve optimal environmental conditions for the test organisms. Scientific discretion was implemented where guideline parameters do not fully converge.
Deviations:	not applicable
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Materials and methods
Test solutions from the study were analyzed to determine the concentrations of BYI 02960. The analysis was performed using Liquid Chromatography-Mass Spectrometry/Mass Spectrometry (LC-MS/MS; ESI positive, mass transitions m/z 289 → 126 for quantitation and m/z 295 → 130 for confirmation) and a stable isotopically labelled internal standard.

A 2.0 mL aliquot of the sample was taken and diluted as needed, to bring the samples within the calibration curve. Next, 0.020 mL of the 0.10 µg/mL of the BYI 02960-IS standard was added to each sample and mixed well. The sample was extracted three times with MtBE and evaporated to dryness. It was reconstituted with ACN/ water, 1:4, containing 0.1% acetic acid and 50 µL was injected for analysis via LC-MS/MS.

Results and discussions
The analytical method was validated by spiking control hard water with BYI 02960 (DLT + FPF EC 85) formulation. The limit of quantitation (LOQ) for BYI 02960 was 0.10 µg/L. During method validation, 10 spikes were prepared: five spikes each at 0.10 µg/L and 20.0 µg/L concentrations.

The individual recoveries ranged from 93% to 99%. The mean recovery was 96% for the 0.10 µg/L level and 97% and for the 20.0 µg/L level. The relative standard deviations (RSD) were of 2.6% and 1.9%, respectively. The mean recovery from 10 spikes was 96% with an overall relative standard deviation (RSD) of 2.2%.

Table A 69: Recovery rates and precision results (repeatability) of BYI 02960

Analyte	Spiked concentration [µg/L]	BYI 02960 measured concentration [µg/L]	Single Values [%]	Mean Value [%]	RSD [%]
BYI 02960	0.10	0.10	94, 96, 93, 98, 99	96	2.6
	20.0	20.0	97, 99, 94, 96, 97	97	1.9
			Overall mean (n = 10)	**96**	**2.2**

RSD = Relative Standard Deviation

In addition to the analysis of fortified samples for validation purposes, laboratory spikes were prepared and analyzed with each set of samples for quality control (QC) purposes. Three QC samples were analyzed during the study at the 1.0 µg/L level. The mean recovery of the laboratory spikes was 97%.

Table A 70: Recovery rates of BYI 02960 in QC samples

Analyte	Test level [µg/L]	Recovery [%]	Sample day
BYI 02960	1.0	97	0
	1.0	97	0 (retain)
	1.0	98	4

Table A 71: Characteristics for the analytical method used for validation of BYI 02960

	BYI 02960
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 0.907107 x – 0.00101482, Correlation coefficient r: 0.9999, number of data points: 7
Calibration range	0.020 µg/L – 4.0 µg/L
Limit of determination/quantification	LOQ = 0.10 µg/L
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of BYI 02960 in test water samples via HPLC-MS/MS.

A 2.2.1.6.2 Analytical method 01213 in support of the study M-679497-01-1

A 2.2.1.6.2.1 Method validation

Comments of zRMS:	The analytical method has been validated for the determination of flupyradifurone in test water samples according to the SANCO/3029/99 rev. 4. The LOQ was 1.6 µg test item/L (corresponding to nominal 0.102 µg a.i./L). The mean recovery was 93% with an overall relative standard deviation (RSD) of 7%. The study is acceptable.

Reference:	**KCP 5.1/37**
Title:	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to rainbow trout (Oncorhynchus mykiss) in a 96-hour semi-static test
Report:	xxx; EBRV0196; M-679497-01-1
Authority registration No:	
Guideline(s):	OECD Guideline for Testing of Chemicals, Section 2, No. 203, "Fish, Acute Toxicity Test", June 18, 2019 OECD Series on Testing and Assessment, No. 23, "Guidance Document on Aqueous-phase Aquatic Toxicity Testing of Difficult Test Chemicals", 2nd Ed., February 08, 2019 EPA Guideline 712-C-16-007: OCSPP 850.1075, "Freshwater and Saltwater Fish Acute Toxicity Test", October 2016 SANCO/3029/99 rev.4 11/07/00: Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A; Section 4) and Annex III (part A; Section 5) of directive 91/414
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	No

Materials and methods

The concentrations of the active ingredient Flupyradifurone of the test item Deltamethrin + Flupyradifurone EC85 (10+75 g/L) were measured in the entire taken diluted test medium and control samples. The water samples are analysed by direct injection in an HPLC-MS/MS instrument after appropriate dilution. The HPLC-MS/MS was operated in the positive ionization mode using the mass transitions m/z 289.141 → 126.000 for the quantitation of Flupyradifurone and m/z 289.141 → 90.100 for confirmation.

For the determination of Flupyradifurone, the analytical method 01213 was used in the present study which is fully validated and EU-agreed (Fargeix, G.; Rosati, D.; 2012; M-428019-01-1).

Results and discussions

Recovery rates were determined at fortification levels of 1.6, 3.5 and 200 µg test item/L with 5 replicates each. The recovery experiments were conducted by fortification of untreated control samples with defined amounts of the analytes prior to analysis.

The overall mean recovery and mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 72: Recovery rates and precision results (repeatability) of Flupyradifurone

	Concentration					
Sample description	**Nominal [µg test item/L]**	**Found [µg a.i./L][1]**	**DF**	**Concentration calculated [µg a.i./L][1]**	**Corrected nominal [µg a.i./L][1]**	**Recovery [%][1]**
Anayltical Blank	0	<LOD	1.25	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	1.25	n.a.	0.000	n.a.
Fortified Sample	1.6	0.071	1.25	0.088	0.102	87
	1.6	0.072	1.25	0.090	0.102	88
	1.6	0.071	1.25	0.088	0.102	86
	1.6	0.068	1.25	0.084	0.102	82
	1.6	0.074	1.25	0.092	0.102	90
Mean value (n = 5):						**87**
RSD (n = 5):						**3**
Fortified Sample	3.5	0.166	1.25	0.208	0.223	93
	3.5	0.166	1.25	0.208	0.223	93
	3.5	0.182	1.25	0.227	0.224	101
	3.5	0.174	1.25	0.218	0.224	97
	3.5	0.158	1.25	0.198	0.224	88

Mean value (n = 5):						**95**
RSD (n = 5):						**5**
Fortified Sample	200	1.965	6.25	12.280	12.720	97
	200	1.946	6.25	12.164	12.720	96
	200	1.934	6.25	12.087	12.802	94
	200	2.113	6.25	13.207	12.802	103
	200	2.064	6.25	12.898	12.802	101
Mean value (n = 5):						**98**
RSD (n = 5):						**4**
Overall mean value (n = 15):						**93**
RSD (n = 15):						**7**

1 The tabulated results represent rounded results calculated on the exact raw data; a.i. = active ingredient (= Flupyradifurone); LOD: Limit of Detection = 0.004 µg a.i./L; n.a.: not applicable; DF: Dilution factor; RSD: Relative Standard Deviation

Table A 73: Characteristics for the analytical method used for validation of Flupyradifurone

	Flupyradifurone
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 16181 x + 107, Correlation coefficient r: 1.0000, number of data points: 8
Calibration range	0.05 µg/L – 5 µg/L
Limit of determination/quantification	LOQ = 0.102 µg a.i./L
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion

The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of Flupyradifurone in test water samples via HPLC-MS/MS.

A 2.2.1.6.3 Analytical method 01213 in support of the study M-686370-01-1

A 2.2.1.6.3.1 Method validation

Comments of zRMS:	The analytical method has been validated for the determination of flupyradifurone in water test medium according to the SANCO/3029/99 rev. 4. The LOQ was 0.6 µg test item/L (corresponding to nominal 0.038 µg a.i./L). The mean recovery was 100% with an overall relative standard deviation (RSD) of 5%. The study is acceptable.

Reference:	**KCP 5.1/38**
Title:	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to Daphnia magna in a semi-static 48-hour immobilisation test - Final report -
Report:	xxx; EBRV0195; M-686370-01-1
Authority registration No:	
Guideline(s):	− EPA Guideline 712-C-16-013: OCSPP 850.1010, "Aquatic Invertebrate Acute Toxicity Test, Freshwater Daphnids" October 2016 − OECD Guideline for Testing of Chemicals No. 202: "Daphnia sp., Acute Immobilisation Test" adopted April 13, 2004 − OECD Series on Testing and Assessment, No. 23, "Guidance Document on Aqueous-phase Aquatic Toxicity Testing of Difficult Test Chemicals", 2nd Ed., February 08, 2019 − Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009, Official Journal of the European Union No. L 309: 1 – 50 − SANCO/3029/99 rev.4 11/07/00: Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A; Section 4) and Annex III (part A; Section 5) of directive 91/414
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The concentrations of the active ingredient Flupyradifurone of the test item Deltamethrin + Flupyradifurone EC85 (10+75 g/L) were measured in the entire taken diluted test medium and control samples. The water samples are analysed by direct injection in an HPLC-MS/MS instrument after appropriate dilution. The HPLC-MS/MS was operated in the positive ionization mode using the mass transitions m/z 289.141 → 126.000 for the quantitation of Flupyradifurone and m/z 289.141 → 90.100 for confirmation.

For the determination of Flupyradifurone, the analytical method 01213 was used in the present study which is fully validated and EU-agreed (Fargeix, G.; Rosati, D.; 2012; M-428019-01-1).

Results and discussions

Recovery rates were determined at fortification levels of 0.6, 1.0 and 15 µg test item/L with 5 replicates each. The recovery experiments were conducted by fortification of untreated control samples with defined amounts of the analytes prior to analysis.

The overall mean recovery and mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 74: Recovery rates and precision results (repeatability) of Flupyradifurone

	Concentration					
Sample description	**Nominal [µg test item/L]**	**Found [µg a.i./L][1]**	**DF**	**Concentration calculated [µg a.i./L][1]**	**Corrected nominal [µg a.i./L][1]**	**Recovery [%][1]**
Anayltical Blank	0	<LOD	1.25	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	1.25	n.a.	0.000	n.a.
Fortified Sample	0.6	0.029	1.25	0.036	0.038	94
	0.6	0.029	1.25	0.036	0.038	95
	0.6	0.031	1.25	0.039	0.038	102
	0.6	0.028	1.25	0.036	0.038	93
	0.6	0.031	1.25	0.039	0.038	102
Mean value (n = 5):						**97**
RSD (n = 5):						**5**
Fortified Sample	1.0	0.050	1.25	0.063	0.064	99
	1.0	0.050	1.25	0.062	0.064	98
	1.0	0.049	1.25	0.061	0.064	96

	1.0	0.052	1.25	0.065	0.064	102
	1.0	0.052	1.25	0.065	0.064	101
Mean value (n = 5):						**99**
RSD (n = 5):						**3**
Fortified Sample	15	0.765	1.25	0.957	0.953	100
	15	0.768	1.25	0.960	0.953	101
	15	0.812	1.25	1.015	0.956	106
	15	0.827	1.25	1.034	0.956	108
	15	0.834	1.25	1.043	0.956	109
Mean value (n = 5):						**102**
RSD (n = 5):						**4**
Overall mean value (n = 15):						**100**
RSD (n = 15):						**5**

1 The tabulated results represent rounded results calculated on the exact raw data; a.i. = active ingredient (= Flupyradifurone); LOD: Limit of Detection = 0.0063 µg a.i./L; n.a.: not applicable; DF: Dilution factor; RSD: Relative Standard Deviation

Table A 75: Characteristics for the analytical method used for validation of Flupyradifurone

	Flupyradifurone
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 45194 x + 105, Correlation coefficient r: 0.9999, number of data points: 9
Calibration range	0.01 µg/L – 1.25 µg/L
Limit of determination/quantification	LOQ = 0.038 µg a.i./L
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion

The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of Flupyradifurone in test water samples via HPLC-MS/MS.

A 2.2.1.6.4 Analytical method 01213 in support of the study M-686369-01-1

A 2.2.1.6.4.1 Method validation

Comments of zRMS:	The analytical method has been validated for the determination of flupyradifurone in water test medium according to the SANCO/3029/99 rev. 4. The LOQ was 0.6 µg test item/L (corresponding to nominal 0.038 µg a.i./L). The mean recovery was 103% with an overall relative standard deviation (RSD) of 8%. The study is acceptable.

Reference:	**KCP 5.1/39**
Title:	Deltamethrin + flupyradifurone EC85 (10+75 g/L): Acute toxicity to larvae of Chironomus riparius in a semi-static 48-hour immobilisation test - Final report -
Report:	xxx; EBRV0194; M-686369-01-1
Authority registration No:	
Guideline(s):	− OECD Guideline for Testing of Chemicals 235: "Chironomus sp., Acute Immobilisation Test" adopted July 28, 2011 − OECD Series on Testing and Assessment, No. 23, "Guidance Document on Aqueous-phase Aquatic Toxicity Testing of Difficult Test Chemicals", 2nd Ed., February 08, 2019 − SANCO/3029/99 rev.4 11/07/00: Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A; Section 4) and Annex III (part A; Section 5) of directive 91/414
Deviations:	None
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

The concentrations of the active ingredient Flupyradifurone of the test item Deltamethrin + Flupyradifurone EC85 (10+75 g/L) were measured in the entire taken diluted test medium and control samples. The water samples are analysed by direct injection in an HPLC-MS/MS instrument after appropriate dilution. The HPLC-MS/MS was operated in the positive ionization mode using the mass transitions m/z 289.141 → 126.000 for the quantitation of Flupyradifurone and m/z 289.141 → 90.100 for confirmation.

For the determination of Flupyradifurone, the analytical method 01213 was used in the present study which is fully validated and EU-agreed (Fargeix, G.; Rosati, D.; 2012; M-428019-01-1).

Results and discussions

Recovery rates were determined at fortification levels of 0.6, 1.5 and 30 µg test item/L with 5 replicates each. The recovery experiments were conducted by fortification of untreated control samples with defined amounts of the analytes prior to analysis.

The overall mean recovery and mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 76: Recovery rates and precision results (repeatability) of Flupyradifurone

	Concentration					
Sample description	**Nominal [µg test item/L]**	**Found [µg a.i./L][1]**	**DF**	**Concentration calculated [µg a.i./L][1]**	**Corrected nominal [µg a.i./L][1]**	**Recovery [%][1]**
Anayltical Blank	0	<LOD	1.25	n.a.	0.000	n.a.
Anayltical Blank	0	<LOD	1.25	n.a.	0.000	n.a.
Fortified Sample	0.6	0.032	1.25	0.040	0.038	105
	0.6	0.034	1.25	0.043	0.038	112
	0.6	0.032	1.25	0.040	0.038	105
	0.6	0.031	1.25	0.039	0.038	101
	0.6	0.037	1.25	0.046	0.038	121
Mean value (n = 5):						**109**
RSD (n = 5):						**7**
Fortified Sample	1.5	0.079	1.25	0.098	0.095	103
	1.5	0.088	1.25	0.110	0.095	115
	1.5	0.078	1.25	0.098	0.096	102
	1.5	0.075	1.25	0.094	0.096	98
	1.5	0.081	1.25	0.101	0.096	105
Mean value (n = 5):						**105**
RSD (n = 5):						**6**

Fortified Sample	30	0.757	2.5	1.893	1.908	99
	30	0.774	2.5	1.934	1.908	101
	30	0.682	2.5	1.705	1.919	89
	30	0.752	2.5	1.879	1.919	98
	30	0.738	2.5	1.844	1.919	96
Mean value (n = 5):						**97**
RSD (n = 5):						**5**
Overall mean value (n = 15):						**103**
RSD (n = 15):						**8**

1 The tabulated results represent rounded results calculated on the exact raw data; a.i. = active ingredient (= Flupyradifurone); LOD: Limit of Detection = 0.0019 µg a.i./L; n.a.: not applicable; DF: Dilution factor; RSD: Relative Standard Deviation

Table A 77: Characteristics for the analytical method used for validation of Flupyradifurone

	Flupyradifurone
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 35914 x – 90, Correlation coefficient r: 0.9999, number of data points: 9
Calibration range	0.01 µg/L – 1.5 µg/L
Limit of determination/quantification	LOQ = 0.038 µg a.i./L
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of Flupyradifurone in test water samples via HPLC-MS/MS.

A 2.2.1.6.5 Analytical method in support of the study M-553769-03-1

A 2.2.1.6.5.1 Method validation

Comments of zRMS:	The analytical method was validated to determine the concentrations of BYI 02960 in test water samples with LOQ of 0.001 µg/L. The mean recovery was 99% with an overall relative standard deviation (RSD) of 1.5%. The method is acceptable and fit for purpose.

Reference:	**KCP 5.1/49**
Title:	Amendment no. 2 - Acute toxicity of deltamethrin + flupyradifurone EC 85 to Daphnia magna under static conditions - Final report -
Report:	xxx; EBRVR015; M-553769-03-1
Authority registration No:	
Guideline(s):	OCSPP Guideline 850.1010 [10], OECD Guideline 202 [3]. The afore- mentioned guidelines were harmonized for various test parameters (i.e. temperature, light, etc.) to achieve optimal environmental conditions for the test organism. Scientific discretion was implemented where guideline parameters do not fully converge EU Directive 91/414/EEC Regulation (EC) No. 1107/2009 US EPA OCSPP 850.1010
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
Test solutions from the study were analyzed to determine the concentrations of BYI 02960. The analysis

was performed using Liquid Chromatography-Mass Spectrometry/Mass Spectrometry (LC-MS/MS; ESI positive, mass transitions m/z 289 → 126 for quantitation and m/z 295 → 130 for confirmation) and a stable isotopically labelled internal standard.

A 50.0 mL aliquot of the sample was diluted with hard water containing 0.1% formic acid to bring the sample within the calibration curve, if needed and then added to a 50-mL plastic centrifuge tube or equivalent. A 0.10 mL aliquot of the 0.020 µg/mL of the BYI 02960-IS standard was added to each sample and mixed well. The following SPE steps utilized gravity only (no vacuum). Waters 0.2g HLB SPE columns were conditioned with 5 mL of ACN followed by 5 mL of 0.1% aqueous formic acid in water. The sample solutions were transferred onto the SPE columns and discarded to waste. Sample tubes were rinsed with 5 mL of 0.1% formic acid in water and the SPE columns were washed with the rinse which was discarded afterwards. The sample tubes were rinsed with 5 mL of an ACN/Water (15:85, v:v) solution containing 0.1% formic acid, then the SPE columns were washed with the rinse and pulled dry for at least 45 minutes under full vacuum. The SPE columns were eluted with two column volumes of ACN containing 0.1% formic acid and the elute was collected in glass test tubes. The ACN was evaporated to dryness. Samples were reconstituted in 1.0 mL of ACN/Water (1:4, v:v) containing 0.1% acetic acid solution then sonicated and vortexed. The samples were transferred to an autosampler vial and a 50µL aliquot was injected into the LC-MS/MS.

Results and discussions

The analytical method was validated by spiking control hard water with BYI 02960 (DLT + FPF EC 85) formulation. The limit of quantitation (LOQ) for BYI 02960 was 0.001 µg/L. During method validation, 10 spikes were prepared: five spikes each at 0.001 µg/L and 0.80 µg/L concentrations.

The individual recoveries ranged from 97% to 101%. The mean recovery for each level was 99% for the 0.001 µg/L, and 99% for the 0.80 µg/L levels respectively with relative standard deviations (RSD) of 2.1% and 1.0% respectively. The mean recovery from 10 spikes was 99% with an overall relative standard deviation (RSD) of 1.5%.

Table A 78: Recovery rates and precision results (repeatability) of BYI 02960

Analyte	Spiked concentration [µg/L]	BYI 02960 measured concentration [µg/L]	Single Values [%]	Mean Value [%]	RSD [%]
BYI 02960	0.001	0.00097	97	99	2.1
	0.001	0.00101	101		
	0.001	0.00100	100		
	0.001	0.00101	101		
	0.001	0.00097	97		
	0.80	0.79767	100	99	1.0
	0.80	0.78729	98		
	0.80	0.78123	98		
	0.80	0.80235	100		
	0.80	0.78808	99		
			Overall mean (n = 10)	**99**	**1.5**

RSD = Relative Standard Deviation

In addition to the analysis of fortified samples for validation purposes, laboratory spikes were prepared and analyzed with each set of samples for quality control (QC) purposes. Three QC samples were analyzed during the study at the 0.010 µg/L level. The mean recovery of the laboratory spikes was 104%.

Table A 79: Recovery rates of BYI 02960 in QC samples

Analyte	Test level [µg/L]	Recovery [%]	Sample day
BYI 02960	0.010	102	0
	0.010	106	0 (retain)
	0.010	104	2

Table A 80: Characteristics for the analytical method used for validation of BYI 02960

	BYI 02960
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 22.2812 x + 0.000406993, Correlation coefficient r: 0.9998, number of data points: 7
Calibration range	0.0002 µg/L – 0.040 µg/L
Limit of determination/quantification	LOQ = 0.001 µg/L
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of BYI 02960 in test water samples via HPLC-MS/MS.

A 2.2.1.6.6 Analytical method 01182 in support of the study

A 2.2.1.6.6.1 Method validation

Comments of zRMS:	The study of Krebber, R.; Sandau, C.; 2010 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *This method was developed for the determination of BYI 02960 in test water from aquatic toxicity tests.* *Aliquots of the water samples are directly injected into the HPLC instrument. Identification and quantitative determination are done by means of electrospray MS/MS-detection in the positive ionisation mode.* *For method validation test water samples were fortified with BYI 02960 at 0.05 µg/L and at 0.5 µg/L.* *The relative standard deviation for the peak areas of BYI 02960 was 1.8% (0.05 µg/L) and 1.4% (0.5 µg/L). The relative standard deviation for the retention time was ≤ 0.3% (0.05 µg/L and 0.5 µg/L).* *BYI 02960 was not detected in test water control samples.* *The limit of quantitation was 0.05 µg/L. The limit of detection (LOD) for BYI 02960 was 0.02 µg/L.* *According to the SANCO/3029/99 rev.4:* *GLP: The development of a method is not subject to GLP, however where the method is used to generate data for safety purposes, for example residues data, data generation must be conducted to GLP.*

Reference:	**KCP 5.1/50**
Title:	Method 01182 for the determination of BYI 02960 in test water from aquatic toxicity tests by HPLC-MS/MS
Report:	Krebber, R.; Sandau, C.; 2010; 01182; M-363959-01-1
Authority registration No:	
Guideline(s):	not specified
Deviations:	not specified
GLP/GEP:	no
Acceptability:	yes
Duplication (if vertebrate study):	

This method 01182 was developed for the determination of BYI 02960 in test water from aquatic toxicity tests via HPLC-MS/MS.

Materials and methods
Aliquots of the water samples are directly injected into the HPLC instrument. Identification and quantitative determination are done by means of electrospray MS/MS-detection in the positive ionisation mode. For quantitation a MRM transition was monitored for BYI 02960 (m/z 289 → m/z 126).

Results and discussions
The results of the method validation are summarized in the tables below.

Table A 81: Characteristics for the analytical method used for validation of BYI 02960 residues in water

	BYI 02960
Specificity	No residues of BYI 02960 were detected in the test water control sample
Calibration	For test water the mass spectrometric detector showed linear response in the concentration range of 0.05 µg/L to 11 µg/L for the quantification ion with a correlation coefficient of 0.9993 (1/x weighted).
Recovery and precision	Because of the direct measurement of fortified samples without separate extraction and clean-up steps it is not possible to determine recovery rates and an estimate of the accuracy of the analytical technique was made by an assessment of the linearity of calibration and by determination of the reproducibility of sample analysis. For method validation test water samples were fortified with BYI 02960 at 0.05 µg/L and at 0.5 µg/L. These test solutions were injected ten times each into the HPLC-MS/MS instrument. The relative standard deviation for the peak areas of BYI 02960 was 1.8 % (0.05 µg/L) and 1.4 % (0.5 µg/L). The relative standard deviation for the retention time was ≤ 0.3 % (0.05 µg/L and 0.5 µg/L).
Recovery of validation samples	For an additional demonstration of the reliability of the method, the validation samples were evaluated like recovery rates. The mean recoveries were 93 ± 2.1% at 0.05 µg/L and 106 ± 1.3 at 0.5 µg/L fortification level.
Limit of determination/quantification	The limit of quantitation (LOQ) for BYI 02960 is 0.05 µg/L. The limit of detection (LOD) for BYI 02960 is 0.02 µg/L.

Conclusion
In conclusion, the results summarized in the tables above show that the method presented here is satisfactory for the determination of BYI 02960 in test water from aquatic toxicity tests via HPLC-MS/MS.

A 2.2.1.6.7 Concurrent validation of the analytical method 01182 in support of the study M-556348-01-1

Comments of zRMS:	The water samples were analysed according to method 01182. The method 01182 was validated concurrently with the test solution analyses. For this purpose the BYI 02960 standard injections were evaluated. The LOQ was 0.0033 µg/L. The method is acceptable and can be regarded as fit for purpose.

Reference:	**KCP 5.1/51**
Title:	Acute toxicity of deltamethrin + flupyradifurone EC 85 (10+75) G to larvae of Chironomus riparius in a 48 h static laboratory test system
Report:	Silke, G.; 2016; EBRVN060; M-556348-01-1
Authority registration No:	
Guideline(s):	OECD Guideline No. 235 (Guideline for Testing of Chemicals, Chironomus sp., Acute Immobilisation Test, adopted July 28, 2011) US EPA OCSPP 850.SUPP
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Concurrent validation

The water samples were analysed according to the following method:

- - "Method 01182 for the determination of BYI 02960 in test water from aquatic toxicity tests by HPLC-MS/MS"; Krebber, R.; Sandau, C.; 2010; M-363959-01-1

The water samples containing deltamethrin + flupyrafifurone EC 85 (10+75) G were analysed for flupyradifurone (BYI 02960). The water samples are analysed by direct injection in an HPLC-MS/MS instrument after appropriate dilution. The HPLC-MS/MS was operated in the positive ionization mode using the mass transitions m/z 289 → 126 for the quantitation of Flupyradifurone. The evaluation of measurements based on HPLC-MS/MS was done by comparison of the peak areas of the samples with the peak areas of the external standard solutions.

In the present study the method was validated concurrently with the sample analyses of the study by evaluation of the standard injections. A lower limit of quantitation of 0.0033 µg/L was validated and the linearity of the MS-detector was checked for BYI 02960 in the concentration range from 0.0033 µg/L to 1 µg/L with an injection volume of 20 µL. The correlation coefficient was 0.9999 (1/x weighted).

Because of the direct measurement of the samples recovery rates cannot be calculated. Thus, the presented precision data is based on the injections of five different standard solutions. The relative standard deviations for the peak areas were <1% for all measured concentration levels.

Table A 82: Recovery rates and precision results (repeatability) of BYI 02960

BYI 02960 standard concentration [µg/L]	n	Peak area		Retention Time	
		Mean Value	RSD	Mean Value	RSD
		[area counts]	[%]	[min]	[%]
0.0033	10	8886	0.5	1.30	0.4
0.033	6	75305	1.9	1.30	0.7
0.100	4	227748	1.0	1.30	0.4
0.500	4	1149734	1.5	1.31	0.7
1.00	6	2285576	1.5	1.30	0.6

RSD: Relative Standard Deviation

Conclusion

The applicability of the HPLC-MS/MS method for the analysis of BYI 02960 in test water was assessed. The data presented demonstrate that the method allows the determination of this substance with satisfactory precision given that the relative standard deviations were below 1% for all measured concentration levels. Thus, this method can be regarded as fit for purpose with regard to the present study.

A 2.2.1.6.8 Analytical method in support of the study M-547460-01-1

A 2.2.1.6.8.1 Method validation

Comments of zRMS:	The analytical method has been validated to determine the concentrations of BYI 02960 in media water with LOQ of 0.002 mg/L. The mean recovery was 97% with an overall relative standard deviation (RSD) of 3.8%. The method is acceptable and suitable for the determination of BYI 02960 in test water.

Reference:	**KCP 5.1/52**
Title:	Toxicity of deltamethrin + flupyradifurone EC 85 to the green algae Pseudokirchneriella subcapitata during a 72 hour exposure
Report:	xxx; EBRVR016; M-547460-01-1
Authority registration No:	
Guideline(s):	OCSPP Guideline 850.4500, OECD Guideline 201. The afore- mentioned guidelines were harmonized for various test parameters (i.e. temperature, light, etc.) to achieve optimal environmental conditions for the test organism. Scientific discretion was implemented where guideline parameters do not fully converge.
Deviations:	not applicable
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

Test solutions from the study were analyzed to determine the concentrations of BYI 02960. The analysis was performed using Liquid Chromatography-Mass Spectrometry/Mass Spectrometry (LC-MS/MS; ESI positive, mass transitions m/z 289 → 126 for quantitation and m/z 295 → 130 for confirmation) and a stable isotopically labelled internal standard.

A 1.0 mL aliquot of the sample was diluted with control 1X AAP media water to bring the sample within the calibration curve, if needed and then added to a 15-mL plastic centrifuge tube or equivalent. A 0.20 mL aliquot of the 0.10 µg/mL of the BYI 02960-IS standard was added to each sample, diluted to the 10 mL mark with ACN/Water (1:4) with 0.1% acetic acid and mixed well. The samples were transferred to an autosampler vial and 50 µL was injected into the LC-MS/MS.

Results and discussions

The analytical method was validated by spiking control 1X AAP media water with DLT + FPF EC 85 formulation. The limit of quantitation (LOQ) was 0.002 mg/L. During method validation, ten spikes were prepared: five spikes each at 0.002 mg/L and 5.0 mg/L concentrations.

The individual recoveries ranged from 90 to 102%. The mean recovery for each level was 94% and 100% for the 0.002 mg/L and the 5.0 mg/L levels respectively with a relative standard deviation (RSD) of 2.9 and 2.3% respectively. The mean recovery from 10 spikes was 97% with an overall relative standard deviation (RSD) of 3.8%.

Table A 83: Recovery rates and precision results (repeatability) of BYI 02960

Analyte	Spiked concentration [µg/L]	BYI 02960 measured concentration [µg/L]	Single Values [%]	Mean Value [%]	RSD [%]
BYI 02960	0.002	0.00180	90	94	2.9
	0.002	0.00189	95		
	0.002	0.00195	97		
	0.002	0.00193	96		
	0.002	0.00187	93		
	5.0	4.8692	97	100	2.3
	5.0	4.9699	99		
	5.0	5.0761	102		
	5.0	4.8905	98		
	5.0	5.0834	102		

			Overall mean (n = 10)	**97**	**3.8**

RSD = Relative Standard Deviation

In addition to the analysis of fortified samples for validation purposes, laboratory spikes were prepared and analyzed with each set of samples for quality control (QC) purposes. Three QC samples were analyzed during the study at the 0.032 mg/L level. The mean recovery of the laboratory spikes was 97%.

Table A 84: Recovery rates of BYI 02960 in QC samples

Analyte	Test level [µg/L]	Recovery [%]	Sample day
BYI 02960	0.032	99	0
	0.032	95	0 (retain)
	0.032	97	3

Table A 85: Characteristics for the analytical method used for validation of BYI 02960

	BYI 02960
Specificity	HPLC-MS/MS method is highly specific. Blank values of all analytes were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): $y = 47.5614\ x + 6.75582 \cdot 10^{-5}$, Correlation coefficient r: 0.9999, number of data points: 7
Calibration range	0.0004 mg/L – 0.080 mg/L
Limit of determination/quantification	LOQ = 0.002 mg/L
Assessment of matrix effects is presented	Matrix effects not monitored, as the internal standard procedure using stable isotopically labelled internal standards compensates for matrix effects.

Conclusion

The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of BYI 02960 in test water samples via HPLC-MS/MS.

A 2.2.1.6.9 Analytical method in support of the studies M-661092-01-1 and M-661091-01-1

A 2.2.1.6.9.1 Method validation

Comments of zRMS:	The analytical method for the determination of flupyradifurone was validated with regard to recovery (97-98%), linearity of detector response ($R^2 > 0.999$), repeatability, specificity, limit of quantification (0.0397 mg flupyradifurone/L) and limit of detection (0.012 mg flupyradifurone/L). The analytical method fulfills the requirements of SANCO/3029/99 rev. 4, 11/07/2000.

Reference:	**KCP 5.1/53**
Title:	A field study to assess the effects of deltamethrin + flupyradifurone EC 85 (10+75 g/L) on the non-target, surface- and plant-dwelling, arthropod fauna of a grassland habitat (off-crop) in The Netherlands during spring/summer
Report:	Aldershof, S.; Bakker, F.; 2019; B168FFN; M-661092-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC Regulation (EC) no. 1107/2009 IOBC (Hassan, 1992), Anonymous (1992), Brown (1998), IOBC, BART and EPPO Joint Initiative (Candolfi et al., 2000, 2001), De Jong et al. (2010) US EPA OCSPP Not Applicable
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/54**
Title:	A field study to assess the effects of deltamethrin + flupyradifurone EC 85 (10+75 g/L) on the non-target, surface- and plant-dwelling, arthropod fauna of a grassland habitat (off-crop) in SW France during spring/summer
Report:	Aldershof, S.; Bakker, F.; 2019; B169FFN; M-661091-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC Regulation (EC) no. 1107/2009 IOBC (Hassan, 1992), Anonymous (1992), Brown (1998), IOBC, BART and EPPO Joint Initiative (Candolfi et al., 2000, 2001), De Jong et al. (2010) US EPA OCSPP Not Applicable
Deviations:	--
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods
In the analytical phases of the two studies the flupyradifurone content of the test solutions was determined. The following method validation is applicable for both studies.

After sampling, the samples were stored deep-frozen (<-18 °C) until analysis. At the analytical laboratory, the samples were thawed and shaken well using a Vortex mixer. Recovery samples were prepared by fortification of untreated samples of tap water with the test item. The samples were diluted with deionised water prior to analysis by HPLC-PDA.

The limit of quantification was defined as 0.60 mg/L of test item (0.0397 mg/L of Flupyradifurone).

Results and discussions
The analytical method was validated by fortification of untreated samples of tap water with the test item at 0.60 mg/L (0.0397 mg/L flupyradifurone) and 250 mg/L (16.6 mg/L flupyradifurone).

Mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 86: Recovery rates and precision results (repeatability) of flupyradifurone

Test item concentration	flupyradifurone

(mg/L)	Concentration (mg/L)	Concentration found (mg/L)	Recovery (%)	Mean Recovery ± RSD (%)
0.60	0.0397	0.0383	96	97 ± 1
		0.0384	97	
		0.0385	97	
		0.0378	95	
		0.0389	98	
250	16.6	16.4	99	98 ± 0
		16.2	98	
		16.3	98	
		16.3	98	
		16.3	98	

RSD = Relative Standard Deviation

The maximum storage period from sampling to analysis was 68 days within this study. Residues are regarded as stable if the samples are stored deep-frozen for up to 30 days between sampling and analysis. Therefore, the storage stability of Flupyradifurone was verified.

Fortified samples were analysed immediately after fortification and additionally after storage for 79 days below -18 °C.

As indicated below, the mean recovery in stored samples was within the same range obtained from the freshly fortified samples (see method validation). Hence, Flupyradifurone can be regarded as stable in deep-frozen samples for at least 79 days of storage.

Table A 87: Storage stability of flupyradifurone

Test item concentration (mg/L)	flupyradifurone				
	Concentration (mg/L)	Storage period	Concentration found (mg/L)	Recovery (%)	Mean Recovery ± RSD (%)
250	16.6	79 days	17.9	108	104 ± 4
			16.8	101	
			16.9	102	

RSD = Relative Standard Deviation

Table A 88: Characteristics for the analytical method used for validation of flupyradifurone

	flupyradifurone
Specificity	HPLC-PDA method is specific. Blank value was below 30% of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented, calibration equation (1/x weighted): y = 17.5260 x + 0.0033, Correlation coefficient r: 0.9995, number of data points: 8
Calibration range	0.01 mg/L – 7 mg/L
Limit of determination/quantification	LOQ = 0.60 mg/L of test item (0.0397 mg/L of Flupyradifurone).
Assessment of matrix effects is presented	No effects observed.

Conclusion

The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4 and is suitable for the determination of flupyradifurone in test water samples via HPLC-PDA.

A 2.2.1.6.10 Analytical method in support of the studies M-554592-01-1 and M-554604-01-1

A 2.2.1.6.10.1 Method validation

Comments of zRMS:	An analytical method for the determination of deltamethrin and flupyradifurone was succesfully validated with regard to recovery, linearity of detector response, repeatability, specificity, limit of quantification and limit of detection. The limit of quantification (LOQ) was 720 mg/L of test item fortification level (6.24 mg/L of deltamethrin and 47.7 mg/L flupyradifurone). Mean recoveries and relative standard deviations per fortification fulfil the criteria of guideline SANCO/3029/99 (70 - 110 % mean recovery, ≤ 20 % RSD). The analytical method is acceptable.

Reference:	**KCP 5.1/44**
Title:	Deltamethrin + flupyradifurone EC 85 (10+75 g/L): Effects on the seedling emergence of non-target terrestrial plant species under greenhouse conditions
Report:	Ripperger, D.; 2016; S15-01670; M-554592-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC Regulation (EC) No. 1107/2009 OCSPP 850.4100 (2012) OECD 208 (2006)
Deviations:	not specified
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Reference:	**KCP 5.1/45**
Title:	Deltamethrin + flupyradifurone EC 85 (10+75 g/L): Effects on the vegetative vigour of non-target terrestrial plant species under greenhouse conditions
Report:	Ripperger, D.; 2016; S15-01671; M-554604-01-1
Authority registration No:	
Guideline(s):	EU Directive 91/414/EEC Regulation (EC) No. 1107/2009 OCSPP 850.4150 (2012) OECD 227 (2006)
Deviations:	Deviations with no major impact occurred regarding the test conditions
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Materials and methods

In the present study, an analytical method was validated for the determination of deltamethrin and flupyradifurone in spray solution samples used to treat non-target terrestrial plant species. In the following part, only data for flupyradifurone is presented.

The test samples are analysed by direct injection in an HPLC-PDA instrument after appropriate dilution.

The same method was used in the study on effects on the vegetative vigour of non-target terrestrial plant species (Ripperger, D.; 2016; M-554604-01-1) for the analytical verification of the spray solutions. No additional validation parameters were presented in this study.

Results and discussions

Recovery samples were prepared by fortification of untreated samples of deionized water with the test item and determined at test item fortification levels of 720 mg/L and 9500 mg/L.

Mean recoveries per fortification level were within the acceptable range of 70 - 110% and RSD values were below 20%.

Table A 89: Recovery rates and precision results (repeatability) of flupyradifurone

Test item fortification level [mg/L]	flupyradifurone				
	Nominal [mg/L]	Effective [mg/L]	Found [mg/L]	Recovery [%]	Mean Recovery ± RSD [%]
720	47.7	47.7	44.8	94	95 ± 1
			45.8	96	
			45.0	94	
			45.0	94	
			45.4	95	
9500	629	657	585	89	93 ± 4
		640	590	92	
		639	614	96	
		643	634	99	
		632	574	91	

RSD: relative standard deviation

Table A 90: Characteristics for the analytical method used for validation of flupyradifurone

	flupyradifurone
Specificity	HPLC-PDA method is specific. Blank values of analyte were below 30 % of the respective LOQ.
Calibration (type, number of data points)	Individual calibration data is presented calibration equation (linear): $y = 0.3243 x – 0.0601$, Correlation coefficient r: 0.9994 number of data points: 6
Calibration range	10.00 mg/L to 100.0 mg/L
Limit of determination/quantification	LOQ = 47.7 mg/L flupyradifurone (corresponds to 720 mg/L of test item)
Assessment of matrix effects is presented	No effects observed.

Conclusion
The analytical method complies with all guideline criteria according to SANCO/3029/99 rev. 4. It was validated successfully and can be seen as fit for purpose.

A 2.2.1.7 Description of analytical methods for the determination of residues in support of physical and chemical properties tests (KCP 5.1)

Analytical methods used for the generation of pre-authorization data are the same as the ones described in part B section 5.

A 2.2.2 Methods for post-authorization control and monitoring purposes (KCP 5.2)

A 2.2.2.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

No new or additional studies have been submitted.

A 2.2.2.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

No new or additional studies have been submitted.

A 2.2.2.3 Description of Methods for the Analysis of Body Fluids and Tissues (KCP 5.2)

A 2.2.2.3.1 Analytical method 1 (Method 01495)

A 2.2.2.3.1.1 Method validation

Comments of zRMS:	The study of Kaussmann, M.; 2016 has been evaluated in Registration Report for 102000028562/ DLT+FPF EC 85 in February 2022 by zRMS-PL and the summary is presented below: *The method 01495 was validated for the determination of residues of flupyradifurone in blood plasma by HPLC-MS/MS with electrospray ionization and Multiple Reaction Monitoring (MRM).* *The limit of quantitation (LOQ) for flupyradifurone in plasma is 0.05 mg/L.* *The limit of detection (LOD) is 0.015 mg/L.* *Fortification experiments were performed at the limit of quantitation (LOQ) and 10xlimit of quantitation. Mean recoveries for each fortification level and the overall mean recoveries were within the 70% - 110% range for flupyradifurone for both MRM transitions. Relative standard deviations were below 20% for flupyradifurone for both MRM transitions.* *Two MRM transitions were successfully validated for plasma. Therefore, an additional confirmatory method is not necessary.* *All method validation data are in compliance with the guideline requirements according to the SANCO/825/00 rev. 8.1 and SANCO/3029/99 rev. 4.* *Accepted.*

Reference:	**KCP 5.2/13**
Title:	Analytical method 01495 for the determination of various pesticides and selected pesticide metabolites in blood plasma by HPLC-MS/MS
Report:	Kaussmann, M.; 2016; 01495; M-570324-01-1
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 Guidance Document on Residue Analytical Methods, SANCO/825/00 rev. 8.1 of November 16, 2010 European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99 rev. 4, July 11, 2000
Deviations:	none
GLP/GEP:	yes
Acceptability:	yes
Duplication (if vertebrate study):	

Method 01495 is a multi-residue method for the determination of several active substances of plant protection products - including flupyradifurone - and/or their metabolites in plasma of blood by HPLC-MS/MS. Only the results relevant to flupyradifurone are reported here.

Materials and methods

In a first step, the plasma samples were denaturated by mixing with a solution of acetonitrile/water (6/1, v/v) containing 56 mg/L ammonium acetate and 0.14 mL/L formic acid. The sample was subjected to centrifugation to separate sediment and supernatant. An aliquot of the supernatant was subjected to HPLC-MS/MS analysis and the residues were quantified using matrix matched standards. All compounds (including flupyradifurone) were measured in positive ion mode.

The quantification of flupyradifurone was done using the following mass transitions:

- primary method (for quantification): m/z 289 → 126
- confirmatory method: m/z 289 → 90

Results and discussions

Specificity

Apparent residues in control samples were below 0.3 x LOQ. Two MRM transitions were monitored for each analyte. Therefore, the HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary.

Linearity

At least five calibration points were used. The correlation between the injected amount of flupyradifurone and the detector response was linear (1/x weighted) for matrix matched standard solutions ranging from 0.0015 to 0.075 µg/mL (corresponding to 1.5 µg/L to 75 µg/L in plasma).
The correlation coefficients were ≥ 0.99.

Accuracy

Fortification experiments were performed at the limit of quantitation (LOQ) and 10 x limit of quantitation. Mean recoveries for each fortification level were within the 70 - 110 % range for both MRM transitions (see table below).

Precision

For both mass transitions monitored, relative standard deviations (RSD) per fortification level were below 20% (see table below).

Stability of Analytes

The analytes were stable in plasma for at least 3 days when stored in a freezer at ≤ -18 °C. In addition the stability of the analytes in extracts was demonstrated for a period of at least 3 days when stored in a refrigerator at ≤ +6 °C under dark conditions.

Table A 91: Recovery results from method validation of flupyradifurone using the analytical method 01495

Matrix	Fortification level (mg/L)	n	Mean recovery (%)	RSD (%)	Comments
Flupyradifurone (MRM: m/z 289 →126, quantification)					
Plasma	0.05	5	105	2.8	
	0.5	5	104	3.5	
Flupyradifurone (MRM: m/z 289 →90, confirmation)					
Plasma	0.05	5	106	2.1	
	0.5	5	104	3.6	

Table A 92: Characteristics for the analytical method used for validation of flupyradifurone residues in plasma

	Flupyradifurone
Specificity	Blank value < 30% LOQ. Two mass transitions were used for quantification for both mass transitions. Quantification: m/z 289 →126 Confirmation: m/z 289 → 90 Therefore, this LC/MS-MS method can be considered as highly specific and the development of additional confirmatory detection techniques are not necessary. Mass spectra are provided in the original report.
Calibration (type, number of data points)	1/x weighted linear regression Using matrix-matched standards, for both MRM transitions, the correlation coefficients (r) were above 0.99 Number of data points: 7
Calibration range	In matrix matched standard (quantitative and confirmatory MRM): Plasma – Concentration range 1.5 to 75 µg/L
Assessment of matrix effects is presented	Matrix matched standards were used for the evaluation of all analytes which compensate for matrix effects.
Limit of determination/quantification (LOD/LOQ)	LOD: 0.015 mg/L LOQ: 0.05 mg/L

Conclusion

The method meets the guideline criteria (SANCO/825/00 rev. 8.1) to determine residues of flupyradifurone in plasma (body fluids) with at a limit of quantitation (LOQ) of 0.05 mg/L and is therefore suitable as monitoring method.

A 2.2.2.4 Description of Methods for the Analysis of Soil (KCP 5.2)

No new or additional studies have been submitted.

A 2.2.2.5 Description of Methods for the Analysis of Water (KCP 5.2)

No new or additional studies have been submitted.

A 2.2.2.6 Description of Methods for the Analysis of Air (KCP 5.2)

No new or additional studies have been submitted.

A 2.2.2.7 Other Studies/ Information

No new or additional studies have been submitted.