

REGISTRATION REPORT

Part B

Section 7

Metabolism and Residues

Detailed summary of the risk assessment

Product code: HCV08

Product names: Vivendi 300 SL, Auksendy 300 SL, Cliophar Super

Chemical active substance:

Clopyralid-olamine, 395 g/l (300 g ae/l)

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

(Renewal of Authorization under Art.43)

Applicant: UPL Holdings Coöperatief U.A.

Submission date: 22/12/2021, updated 10/2023

MS Finalisation date: October 2023 (initial Core Assessment)

March 2024 (final Core Assessment), updated January 2025

Version history

When	What
December 2021	Article 43 submission for re-registration of HCV07 following Clopyralid Renewal of approval (Commission Implementing Regulation (EU) 2021/1191)
April 2022	Art 43: renewal of authorization_revision and composition change from HCV07 to HCV08. The proposed change in the composition was accepted, the studies presented for the formulations code HCV07 are still supporting the registration of the plant protection product.
October 2023	Honey study and additional onion study provided by Corteva for assessment
October 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.
March 2024	Final report (Core Assessment updated following the commenting period) No additional information or assessments after the commenting period.
January 2025	Honey MRL update Additional information/assessments included by the zRMS in the report after the MRL for honey change in Commission Regulation (EU) 2024/2612 are highlighted in yellow . Not agreed or not relevant information are struck through and shaded for transparency.

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7 Metabolism and residue data (KCA section 6)

7.1 Summary and zRMS Conclusion

7.1.1 Critical GAP(s) and overall conclusion

Selection of critical uses and justification

The critical GAPs with respect to consumer intake and risk assessment for the preparation HCV07 are presented in Table 7.1-1. They have been selected from the individual GAPs in the Central zone for Fodder beet, Sugar beet, Red Beet, Turnip, Swede, Winter Oilseed rape, Spring Oilseed rape, Mustard, Linseed and Onion from seed. A list of all intended uses within the Central zone is given in Part B, Section 0.

Overall conclusion

The data available are considered sufficient for risk assessment. An exceedance of the current MRLs for clopyralid as laid down in Reg. (EU) 396/2005 (Commission Regulation (EU) 2024/2612) for proposed crops and honey is not expected. However, an exceedance of the current MRL of 0.05 mg/kg for clopyralid in honey as laid down in Reg. (EU) 396/2005 may be expected. Until the new MRL has been set for honey, use on oilseed rape, mustard and linseed cannot be authorized.

The chronic and the short-term intakes of clopyralid residues are unlikely to present a public health concern. As far as consumer health protection is concerned, zRMS-PL agrees with the authorization of the intended use(s) except oilseed rape, mustard and linseed.

Data gaps

Noticed data gaps are: None.

Remark:

In accordance with national harmonization arrangements, for products containing clopyralid, after completing the procedure under Article 12 of Regulation No 396/2005, it will be necessary to verify the assessment performed in terms of the established residues definition for the accepted uses.

Table 7.1-1: Acceptability of critical GAPs (and respective fall-back GAPs, if applicable)

1	2	3	4	5	6	7		8				9			10	11
GAP number (see part B.0)*	Crop and/or situation **	Zone	Product code	F, Fn, Fpn G, Gn, Gpn or I***	Pests or Group of pests controlled	Formulation		Application				Application rate per treatment			PHI (days)	Conclusion
						Type	Conc. of as	method kind	growth stage & season	number min max	interval between applications (min)	kg as/hL min max	water L/ha min max	kg ae/ha min max		
1	Fodder beet, Sugar beet, Red Beet, Turnip, Swede EU MRL Code: 0213010, 0900010, 0213010	CEU	HCV07	F	Broad-leaved weeds (BBBBB) (including but not only Cirsium arvense, Matricaria spp.)	SL	395 g as/L (300 g ae/L)	Foliar	BBCH 12-39 35 (until July 1 st)	1	-		100-400	0.090-0.12	42 F	A
6	Winter Oilseed rape, Spring Oilseed rape, Mustard, Linseed EU MRL Code: 0401060	CEU	HCV07	F	Broad-leaved weeds (BBBBB) (including but not only Cirsium arvense, Centaurea cyanus, Matricaria spp)	SL	395 g as/L (300 g ae/L)	Foliar	BBCH 30-51	1	-		100-400	0.12	NA	NA Current MRL exceeded in honey
7	Onion from seeds EU MRL Code: 0220020	CEU	HCV07	F	Broad-leaved weeds (BBBBB) (including but not only Cirsium arvense, Matricaria spp.)	SL	395 g as/L (300 g ae/L)	Foliar	BBCH 11-16	1	-		100-400	0.12	42	A

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

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

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

NA: Not applicable.

Explanation for Column 11 “Conclusion”

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

7.1.2 Summary of the evaluation

The preparation HCV07 is composed of clopyralid olamine. Clopyralid olamine is the 2-aminoethanol salt of clopyralid and in solution dissociates and behaves as clopyralid. Therefore the toxicological reference values for clopyralid are relevant.

Table 7.1-2: Toxicological reference values for the dietary risk assessment of clopyralid

Reference value	Source	Year	Value	Study relied upon	Safety factor
Clopyralid					
ADI	EFSA	2018a	0.15 mg/kg bw per day	Rat, 2-year chronic toxicity and oncogenicity study	100
ARfD	EFSA	2018a	0.17 mg/kg bw	Rabbit, developmental toxicity	300

7.1.2.1 Summary for clopyralid

Table 7.1-3: Summary for clopyralid

Use-No.*	Crop	Plant metabolism covered?	Sufficient residue trials?	PHI sufficiently supported?	Sample storage covered by stability data?	MRL compliance	Chronic risk for consumers identified?	Acute risk for consumers identified?
1, 2, 3, 4, 5	Fodder beet, Sugar beet, Red Beet	Yes	Yes (8)	Yes No	Yes	Yes	No	No
6	Winter Oilseed rape	Yes	Yes (11)	Yes	Yes	Yes for oilseeds and for honey	No	No
7	Onion	Yes	Yes (8)	Yes	Yes	Yes	No	No

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

The effects of processing on the nature of clopyralid residues have been investigated.

Residues in succeeding crops have been sufficiently investigated taking into account the specific circumstances of the cGAP uses being considered here. No mitigation measures are required for Oilseeds. For all other food and feed commodities except sugar canes, a 30-day plant-back interval is supported. It is recommended that sugar canes not be planted for 125 days after application of clopyralid.

As the dietary burden intakes are within those calculated in the EFSA MRL Reasoned Opinion (EFSA, 2021), the existing animal MRLs accommodate the Article 43 uses presented in this submission (including consideration of rotational residues) and no further evaluation is warranted. The requested uses do not modify the theoretical maximum daily intake for animals, and there is no risk for animal MRLs to be exceeded.

Current MRL for clopyralid in honey is 0.05* mg/kg (Reg. (EU) 2021/1807), and according to the provided study on magnitude of residues in honey, the MRL is potentially exceeded. Until the higher MRL has been set for honey, use on melliferous target crops (oilseed rape, mustard and linseed) cannot be authorized.

In accordance with Article 6 of Regulation (EC) No 396/2005, the applicant Corteva Agriscience International Sarl submitted a request to the competent national authority in Finland to modify the existing maximum residue levels (MRLs) for the active substance clopyralid in honey. The data submitted in support of the request were found to be sufficient to derive MRL proposals for honey. According to the EFSA Journal. 2024;22:e8546 (<https://doi.org/10.2903/j.efsa.2024.8546>) to accommodate clopyralid residues in

honey from the use on oilseed rape, the EMS proposed to raise the existing MRLs in honey from the limit of quantification (LOQ) of 0.05 to 0.15 mg/kg.

The current MRL for clopyralid in honey has been changed and now is 0.15 mg/kg (Commission Regulation (EU) 2024/2612), and according to the provided study on magnitudes of residues in honey (UPL has access to this data *via* a letter of access), the MRL is not potentially exceeded. The uses on melliferous target crops (oilseed rape, mustard and linseed) can be authorized.

7.1.2.2 Summary for HCV07

Table 7.1-4: Information on HCV07 (KCA 6.8)

Crop	PHI and withholding period proposed by applicant	PHI/ Withholding period* sufficiently supported for	PHI proposed by zRMS	zRMS Comments (if different PHI proposed)
		Clopyralid		
Fodder beet, Sugar beet, Red Beet, Mangels	42 days	Yes No	F	PHI of 42 days is not sufficiently supported by residue data
Winter Oilseed rape	F**	Yes	F	-
Onion	42 days	Yes	42 days	-

** F: PHI is defined by the application stage at last treatment (time elapsing between last treatment and harvest of the crop).

Table 7.1-5: Waiting periods before planting succeeding crops

Waiting period before planting succeeding crops		Overall waiting period proposed by zRMS
Crop group	Led by Clopyralid	
Oilseeds	NR 30 days	NR 30 days
All other food and feed crops not specified	30 days	30 days
Sugar canes	125 days	125 days

NR: not relevant

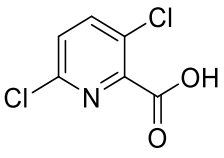
Assessment

7.2 Clopyralid

The preparation HCV07 is composed of clopyralid olamine. Clopyralid olamine is the 2-aminoethanol salt of clopyralid and in solution dissociates and behaves as clopyralid. Therefore data for clopyralid are relevant.

General data on clopyralid are summarized in the table below (last updated 2019/03/25)

Table 7.2-1: General information on clopyralid

Active substance (ISO Common Name)	Clopyralid
IUPAC	3,6-dichloropyridine-2-carboxylic acid or 3,6-dichloropicolinic acid
Chemical structure	
Molecular formula	C ₆ H ₃ Cl ₂ NO ₂
Molar mass	191.96 g/mol
Chemical group	Herbicide, pyridine compound
Mode of action (if available)	Auxin mimic. Taken up via leaves and roots and induces an epinastic response leading to chlorosis, cessation of normal growth and death.
Systemic	Yes
Company (ies)	Dow AgroSciences Ltd (now Corteva Agriscience)*
Rapporteur Member State (RMS)	Finland co-RMS: Poland
Approval status	Approved (01/05/2007, COMMISSION DIRECTIVE 2006/64/EC) Re-approval (01/10/2021) - Commission Implementing Regulation (EU) 2021/1191 http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32021R1191&from=EN
Restriction	Only uses as a herbicide may be authorised
Review Report	SANCO/10012/2006 – rev. 3, 04/04/2006 SANTE/10206/2021– rev. 1 20/05/2021
Current MRL regulation	Reg. (EU) 2021/1807 2024/2612
Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed	Pending (EFSA-Q-2008-513)
EFSA Journal : Conclusion on the peer review	Yes (EFSA, 2018a)
EFSA Journal: conclusion on article 12	No
Current MRL applications on intended uses	EFSA-Q-2018-00576 Modification of the existing maximum residue levels for clopyralid in various commodities Status: Reasoned opinion available (EFSA Journal 2021;19(1):6389); Reg. (EU) 2021/1807 EFSA-Q-2015-00419 (UK) MRLs for clopyralid in spring/green/Welsh onions and leeks Status: Reasoned opinion available (EFSA Journal

	2018;16(1):5149 EFSA-Q-2011-00206 Modification of the existing MRLs for clopyralid in various commodities/ Head cabbage, cauliflower, Brocoli, linseeds (and their by-products), swedes and turnips, animal commodity (bovine sheep goat - meet, liver, kidney; milk) Status: Reasoned Opinion available (EFSA Journal 2011;9(10):2418)
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7.2.1 Stability of Residues (KCA 6.1)

7.2.1.1 Stability of residues during storage of samples

Available data

Available data

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

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

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference
Data relied on in EU			
Plant products			
Pasture grass	High water content	17 months	EFSA, 2018a
Maize fodder/forage	High water content	13 months	EFSA, 2018a
Oilseed rape	High oil content	24 months	EFSA, 2018a
Olive (fruit and oil)	High oil content	10 months	EFSA, 2018a
Maize	High starch content	13 months	EFSA, 2018a
Orange	High acid content	10 months	EFSA, 2018a
Orange peel	Other	10 months	EFSA, 2018a
Animal Products			
Ruminant	Muscle	19 months	EFSA, 2018a
Ruminant	Liver	19 months	EFSA, 2018a
Ruminant	Kidney	19 months	EFSA, 2018a
Ruminant	Milk	19 months	EFSA, 2018a
Ruminant	Fat	24 months	EFSA, 2018a
Poultry	Eggs	19 months	EFSA, 2018a
New data			
Dried Navy Beans	High protein content	13 months	Skaggs, C. S., Penning, B. N; 2021; Corteva Study No. 191728
Strawberries	High acid content	24-18 months	Teasdale, R.; 1996; Corteva Study No. GHE-P-4832
Honey/nectar/pollen	pollinator	18 months	Forbes, T.; Cross, M.; 2020; Corteva Study No. 180869

Reference: EFSA, 2018a

“Stability of conjugates was not tested, though clopyralid conjugates are major metabolites comprising up to 50% of TRR depending on the crop studied. It was assumed that conjugated clopyralid was also stable.”

Conclusion on stability of residues during storage

Clopyralid (free and conjugated) is stable for at least 17 months in high water commodities, 24 months in high oil commodities, 13 months in high starch commodities and 10 months in high acid commodities when stored under frozen ($\leq -18^{\circ}\text{C}$) conditions in the EFSA conclusion 2018a. The additional studies for high protein, and high acid has demonstrated clopyralid is stable across the five commodity categories and therefore is stable in all crop commodities up to 13 months when stored under frozen ($\leq -18^{\circ}\text{C}$) conditions.

Clopyralid (free and conjugated) is stable for at least 19 months in tissues, milk and eggs and up to 24 months in fat when stored under frozen ($\leq -18^{\circ}\text{C}$) conditions.

Clopyralid (free and conjugated) is stable for at least 18 months in honey, pollen and nectar when stored under frozen ($\leq -18^{\circ}\text{C}$) conditions.

zRMS comments:

According to the EFSA Journal 2021;19(1):6389: “The storage stability of clopyralid in plants stored under frozen conditions was investigated in the framework of the EU pesticides peer review (EFSA, 2018d).

It was demonstrated that clopyralid was stable for at least 10 months when stored at -18°C in commodities with high water, high oil, high acid and dry/high starch content (details see Appendix B). In pasture stability was demonstrated up to 17 months, in maize grain and forage/fodder up to 13 months when stored at -20°C .”

The storage stability studies indicated that clopyralid is stable for at least 19 months in animal tissues, milk and eggs and up to 24 months in fat when stored under frozen ($\leq -18^{\circ}\text{C}$) conditions.

Additionally the Applicant submitted three studies on storage stability of clopyralid in plant products, in dried navy beans (Skaggs, C. S., Penning, B. N; 2021), in strawberries (Teasdale, R.; 1996) and in honey/nectar/pollen (Forbes, T.; Cross, M.; 2020). New studies are considered acceptable. Storage stability of clopyralid was demonstrated for a period of 13 months under frozen ($\leq -18^{\circ}\text{C}$) conditions in dried navy beans (commodity with high protein content) and for a period of 18 months in strawberries (commodities with high protein acid content).

It can be concluded that residues of clopyralid are stable for at least 13 months in all crop commodities when stored under frozen (-18°C) conditions.

Clopyralid has been shown to be stable in honey/nectar/pollen for 18 months when stored frozen at -18°C .

The studies on the magnitude of residues are valid with regard to storage stability. The stability of residues during storage of clopyralid in plants and animal products is sufficiently addressed to support the proposed uses of the product HCV08 / Vivendi 300 SL, Auksendy 300 SL, Cliophar Super. No additional study is required.

7.2.1.2 Stability of residues in sample extracts (KCA 6.1)

Available data

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

Conclusion on stability of residues in sample extracts

The stability of residues in sample extracts was demonstrated by acceptable procedural recoveries analysed concurrently with residue trial samples.

zRMS comments:

Procedural recoveries obtained during residue analysis demonstrate the stability of residues of clopyralid in sample extracts. No additional study is required.

7.2.2 Nature of residues in plants, livestock and processed commodities

7.2.2.1 Nature of residue in primary crops (KCA 6.2.1)

Available data

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

Table 7.2-3: Summary of plant metabolism studies

Crop Group	Crop	Label position	Application and sampling details					Reference
			Method, F or G (a)	Rate (kg a.s./ha)	No	Sampling (DAT)	Remarks	
EU data								
Leafy vegetables	Cabbage	¹⁴ C-clopyra- lid	G, foliar	0.42	1	0, 5, 38	Application at BBCH 31 (8-10 leaf stage)	Finland, 2018a
Root and tuber vegetables	Sugar beet	¹⁴ C-clopyra- lid	F, foliar	0.30	1	0, 28, 105 (maturity)	Application at BBCH 36	EFSA, 2018a and Finland, 2018a Finland 2018b†
Pulses and oilseeds	Oilseed rape	¹⁴ C-clopyra- lid	F, foliar	0.30	1	0, 28 (60% final size), 77 (ma- turity)	Application at BBCH 36	EFSA, 2018a and Finland, 2018a Finland 2018b†
Cereals/grass crops	Pasture grass	¹⁴ C-clopyra- lid	F, foliar	1.12	1	1, 2, 4, 8, 18 weeks	Non-GLP, non-guideline compliant. Supporting information only.	Finland, 2018a
Cereals and oilseeds	Oilseed rape and wheat	¹⁴ C-clopyra- lid	G, Hydro- ponic	75 µg/L	-	-	Non-guide- line compli- ant. No me- tabolism data availa- ble, only bio- distribution. Supporting information only.	Finland, 2018a
New data								
Cereals/grass crops	Cereals	¹⁴ C-clopyra- lid	F, foliar (last 3 weeks in- doors, G)	125 g ae/ha	1	3 (forage), 23 (hay), and ma- turity (92 for straw and grain)	Application at BBCH 39,	Morton Lloyd, G, 2020, DAS study No. 191200
Root and tuber vegetables Pulses and oilseeds	Oilseed rape	¹⁴ C-clopyra- lid	F	200 g ae/ha	1	23 (forage) and ma- turity (94) (seeds & trash); pol- len (be- tween 9 and 31)	Application at BBCH 30 Trash is not a RAC	Mackenzie, A, 2021, DAS study No. 200928
Pulses and oilseeds Root and tuber vegetables	Sugar beet	¹⁴ C-clopyra- lid	F	314 g ae/ha	1	21 (imma- ture), 110 (roots & foliage)	Application at BBCH 19	DAS Study No. 210071 Ongoing

(a) F:Field, G: greenhouse
DAT: Days after treatment

† The sugar beet and oilseed rape metabolism studies were evaluated in two separate EU processes. The original reports were evaluated during active substance renewal (EFSA 2018a, Finland RAR 2018a) and the amended reports for the existing studies were evaluated in an MRL Evaluation Report (Finland 2018b).

Summary of plant metabolism studies reported in the EU

Reference: EFSA, 2018a

“Three Good Laboratory Practice (GLP) and guideline-compliant metabolism studies with foliar application of ^{14}C -clopyralid are available for root crop, leafy vegetable and pulses/oilseeds.

The extraction with caustic methanol employed in the cabbage study has led to cleavage of the conjugates and resulted in the presence of free clopyralid at maturity up to 92% and 99% total radioactive residue (TRR) in head and wrapper leaves, respectively. In the studies with oilseed rape and sugar beet, a first extraction was performed with acetonitrile/water and followed with a caustic extraction allowing for investigation of the presence of eventual conjugates. In sugar beet, clopyralid was initially the major residue in the plant (97% TRR at day 0 and 85% TRR at day 28). At maturity, it decreased to 51% TRR in the shoot and to 58% TRR in the root. A ‘polar form of clopyralid’ was observed in shoots and roots up to 37% TRR and 39% TRR, respectively, but only in the mature plant parts. In oilseed rape clopyralid was present at 63% TRR in immature plant and at 32% and 43% TRR in mature straw and seed, respectively. A ‘polar form of clopyralid’ was reported to 32% and 28% TRR in mature straw and seed, respectively and an unknown metabolite B also referred to as ‘clopyralid conjugates’ to 29% and 18% TRR in mature straw and seed, respectively. From the analytical protocol it can be assumed that the ‘polar clopyralid’ refers to the protonated form (clopyralid acid). However, a confirmation that the observed results in the different metabolism studies are consistent with the analytical conditions (pH) used and an explanation why in mature samples of sugar beet and oilseed rape ‘polar clopyralid’ and clopyralid were observed while ‘polar clopyralid’ was not observed in immature samples as the analytical procedure used was always the same within one study is outstanding (data gap). A clarification is also needed whether the term ‘polar clopyralid’ is referring always to a single structure (and not different polar compounds) and that this structure of ‘polar clopyralid’ is identical across all metabolism studies where it has been identified (data gap).”

To address the EFSA data gap, Dow AgroSciences Ltd (DAS, now Corteva Agriscience) has submitted additional data and argumentation. Further explanation of the applicant’s arguments, additional data and conclusions of EFSA and the RMS may be found in Appendix 4. The applicant believes sufficient data have been provided to conclude that the EFSA-proposed plant residue definition is fully justified and can be adopted (‘sum of clopyralid, its salts and conjugates expressed as clopyralid’) and no further data shall be deemed required. Nonetheless, an oilseed rape metabolism study has been completed, and a sugar beet metabolism study is currently under repetition to confirm the residue definition and thereby fill the data gap.

Summary of new plant metabolism studies

Study of Morton Lloyd, G, 2020, DAS study No. 191200

Following a single 125.87 g a.e./ha (104.9% of target) application of [^{14}C]-clopyralid to wheat, TRR levels in forage, hay, straw and grain ranged from 1.879 – 3.717 mg eq/kg.

For all tissues, accountability, between combustion and extraction data, was 95.4 – 107.6%. When normalised with respect to accountability, aqueous acetonitrile recovered 50.6 – 90.5 %TRR and methanolic base extracts released a further 5.0 – 34.7 %TRR. Further extractions were conducted on all samples using mild aqueous base, mild and stronger acid, which released a further 2.6 – 7.5 %TRR. Non-extractable residues were 1.8 – 7.2 %TRR.

The aqueous acetonitrile contained a resolved clopyralid peak (15.4 – 66.0% TRR, 0.311 – 1.240 mg eq/kg) and a less polar, unresolved region (21.0 – 63.3% TRR, 0.394 – 2.351 mg eq/kg). Upon mild base treatment (similar to analytical method 120610 using methanol:10 N NaOH (100:1, v/v), overnight), primarily clopyralid was detected (*ca.* 98%). The methanol base extract, mild aqueous base and acid further extracts contained only clopyralid (*ca.* 99%). No polar clopyralid was detected in any sample.

Experiments on the apolar unresolved region demonstrated that it was completely converted to clopyralid within an hour of treatment with 5% ammonium hydroxide solution (2:1,v/v, sample:base ratio). Any sort of light manipulation of the sample (SPE, changing the pH etc.) to remove endogenous materials resulted

in the elimination of this region, forming clopyralid. This indicates that the unresolved region is a result of weak binding interactions (*e.g.*, electrostatic) between clopyralid and the endogenous plant matrix, which are disrupted by treatment with mild base or removal of endogenous material. The presence of the apolar region in the control sample fortified with clopyralid further adds to the theory that this region is a result of interactions between clopyralid and endogenous materials present in the sample. Overall, it can be strongly hypothesised that this region is the result of chromatographic effects of clopyralid weakly binding/interacting with endogenous material. Therefore, this region can be identified as base-labile clopyralid.

Greater than 90% of the extractable residue was identified as clopyralid. In total, clopyralid accounted for 98.0 %TRR (1.843 mg eq/kg) in forage, 97.2 %TRR (3.608 mg eq/kg) in hay, 92.6 %TRR (1.978 mg eq/kg) in straw and 96.4 %TRR (1.087 mg eq/kg) in grain.

Study of Mackenzie, A, 2021, DAS study No. 200928

The metabolism of clopyralid was investigated in spring oilseed rape at a target application rate of 200 g a.e./ha when the plants were at growth stage BBCH 30. Spring oilseed rape anthers (containing pollen), early forage, seeds and trash (including pods) were harvested. Forage (23 d PHI) contained 1.839 mg eq/kg while seed contained 0.069 mg eq/kg.

All samples were subjected to extraction and characterisation. HPLC analysis of the aqueous acetonitrile extracts from forage contained a resolved clopyralid peak (28.8% TRR, 0.530 mg eq/kg) and a broad less polar (apolar), unresolved region (64.0% TRR, 1.178 mg eq/kg). Trash was similar. The equivalent extracts from seeds contained a resolved clopyralid peak (29.0% TRR, 0.020 mg eq/kg) and two minor, more polar regions (2.1 – 3.3% TRR, 0.001 – 0.002 mg eq/kg). Upon mild base treatment (similar to Dow analytical method 120610, using methanol:10 N NaOH (100:1, v/v), overnight) of the neutral organic extracts, only clopyralid was detected (83.8% and 69.4% TRR in forage and trash, respectively). The methanol base extract and further basic extracts from each sample contained only clopyralid and were therefore characterized as base-labile clopyralid. In total, clopyralid (free and base-labile) accounted for 101.6% TRR (1.871 mg eq/kg) in forage, 95.2% TRR (0.213 mg eq/kg) in trash and 70.3% TRR (0.049 mg eq/kg) in seeds. Only clopyralid was detected in the extracts from the analytical method for all three commodities.

The anthers containing pollen were washed with water and centrifuged to separate the pollen. The isolated pollen was then extracted with methanolic base. Both the water washes and the pollen extract were characterized by HPLC. The majority (63.4%) of the water wash eluted with clopyralid, while the remainder of the water wash eluted in the same apolar region as the forage and trash samples and is therefore characterized as base-labile clopyralid. The majority of the pollen residue was extractable (>85%) and eluted with clopyralid, and is therefore characterized as base-labile clopyralid.

Extensive experiments on the apolar unresolved region demonstrated that this region was readily converted to clopyralid in the presence of dilute base. Any light manipulation readily converted the apolar region to clopyralid, which strongly supports the hypothesis that the apolar region is a result of weak electrostatic interactions with endogenous plant matrix and/or chelation of clopyralid to metal ions in the plant. Although extensive attempts were made, no LC-MS mass or fragment ion related to clopyralid could be found in the apolar region, despite optimization of the mass spectrometer for clopyralid.

In total, clopyralid (free and base-labile) accounted for >70% of the TRR in all samples, including seed, forage, trash, and pollen/anthers. Furthermore, the amount of total clopyralid detected by Dow analytical method 120610 was in excellent agreement with the exhaustive methods used in a metabolism study, at greater than 90%.

Conclusion on metabolism in primary crops

The metabolism of clopyralid in three representative crops from three crop groups was re-evaluated during the EU renewal (cabbage, oilseed rape and sugar beet). In addition, a wheat metabolism study (cereals group) was also completed.

Based on the crop metabolism studies re-evaluated during the EU renewal, EFSA proposed the following residue definitions for plant commodities (EFSA, 2018a):

Residue definition for monitoring (plant): Sum of clopyralid, its salts and conjugates expressed as clopyralid

Residue definition for risk assessment (plant): Sum of clopyralid, its salts and conjugates expressed as clopyralid

On the basis of the additional data submitted by the applicant, to the RMS and EFSA in 2019-2020 in the framework of the MRL application, the applicant believes the EFSA-proposed plant residue definition is fully justified and can be adopted ('sum of clopyralid, its salts and conjugates expressed as clopyralid') and no further data shall be deemed required. Nonetheless, the oilseed rape NOR was repeated which confirms the residue definition above, and sugar beet metabolism studies is currently under repetition to confirm the residue definition, and thereby address the data gaps noted by EFSA. Further explanation may be found in Appendix 4.

Evaluator comments:

The metabolism of clopyralid following foliar application was investigated in the EU pesticides peer review in crops belonging to the groups of root crops, leafy crops and pulses/oilseeds. In the framework of the assessment in accordance with Article 6 of Regulation (EC) No 396/2005, a new metabolism study in wheat, representing cereals/grass crop group was submitted. During the peer review, the data gap related to the identification of an unknown compound observed in sugar beet and oilseed rape metabolism studies was identified and, although the applicant and EMS provided further clarifications under the current assessment, EFSA still considers this data gap as not addressed. In addition, the EU pesticides peer review noted that the extraction conditions in metabolism study with leafy crops were not suitable to allow an elucidation of the metabolism of clopyralid. EFSA concludes that for the intended uses in grass and cereals, the metabolic behaviour of clopyralid in these crops is addressed with the new metabolism study in wheat (EFSA, 2021).

The EU pesticides peer review concluded that, based on the metabolic pattern identified in leafy crops, root crops and cereals/oilseeds crop groups, the results of hydrolysis and rotational crop studies, the data are sufficient to derive the following general residue definitions in primary and rotational crops, pending the outstanding clarification on the nature of 'polar clopyralid' in oilseed rape and sugar beet metabolism:

Residue definition for risk assessment: clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)

Residue definition for enforcement: clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)
(EFSA, 2018)

The residue definition for enforcement set in Regulation (EC) No 396/2005 is currently 'clopyralid' alone. EFSA proposes to change the existing enforcement residue definition by including clopyralid salts and conjugates. The proposed change will not have an impact on the existing MRLs, as the analytical methods used to generate data for risk assessment and for enforcement include a hydrolysis step which is capable to cover the common moiety.

Additionally, EFSA concluded in EFSA Journal 2021;19(1):6389 that *Since the clarification of the unknown polar metabolite (called 'polar clopyralid') in mature sugar beet and oilseeds identified by the EU pesticides peer review was not sufficiently addressed under the current assessment, EFSA concludes that the proposed residue definitions are applicable only to cereals/grass crop group for which a new metabolism study was submitted under the current assessment and for which the data gap identified by the peer review is not relevant. For remaining crop groups, the data gap as identified by the EU pesticides peer review remains open.*

The residue definition for enforcement set in Regulation (EC) No 396/2005 is currently 'clopyralid' alone. EFSA proposes to change the existing enforcement residue definition by including clopyralid salts and conjugates. The proposed change will not have an impact on the existing MRLs, as the analytical methods used to generate data for risk assessment and for enforcement include a hydrolysis step which is capable to cover the common moiety. The residue definitions are applicable to processed products.

In the framework of the current assessment, three new studies on metabolism of clopyralid in cereals, oilseed rape and sugar beet were submitted.

The study by Morton Lloyd (2020) on wheat was evaluated during the MRL evaluation by EMS-Finland (EFSA 2021): *A new metabolism study with wheat covering the group of cereals/grass was submitted in support of the current MRL application (Finland, 2018b). In the wheat plant parts, the parent compound was the only identified residue, representing 20% and 28% of the total radioactive residues (TRR) in straw and grain, respectively. Besides parent, a multitude of partially unresolved and less polar compounds were reported accounting for 30% and 45% TRR in straw and grain, respectively. When the plant extracts were subjected to a base treatment, these compounds disappeared and only clopyralid was observed. It is noted that the unknown less polar fractions of the plant extracts were only characterised as base labile and especially in grain, these residues make up for 48% TRR (0.51 mg eq/kg). In this point, the study does not fulfil the requirements of OECD 501 (OECD, 2007) which stipulates that residues above 10% TRR and above 0.05 mg eq/kg should be identified. The study is reported to be performed*

according to GLP and further clarification could be provided, e.g. individual MS spectra of the partially separated signals eluting after clopyralid which are no longer present after hydrolysis of the extract. An impact on the magnitude of clopyralid in grass and cereals is not expected given the fact that the not identified but characterised as less polar compounds will be converted to clopyralid by the analytical method used in the residue field trials. EFSA concludes that for the intended uses in grass and cereals, the metabolic behaviour of clopyralid is addressed. The study was deemed acceptable.

The study by Mackenzie (2021) investigated the nature of residues in oilseed rape commodities (forage, seed, trash, and anthers and pollen) treated with 1.7N rate at BBCH 30. Only clopyralid (free + base-labile) was identified in all OSR commodities, accounting for a total of 101.6% TRR (1.871 mg eq/kg) in forage, 95.2% TRR (0.213 mg eq/kg) in trash and 70.3% TRR (0.049 mg eq/kg) in seeds. A less polar region eluting before the peak of clopyralid was detected in all aqueous acetonitrile extracts. The less polar region was readily converted to clopyralid by mild base treatment. In anthers, all of the residues were identified as clopyralid, with 63.4% (1.529 mg/kg) being free and 36.7% (0.882 mg/kg) being base-labile clopyralid. In pollen, all extractable clopyralid was characterized as base-labile (85.5% / 0.592 mg/kg), while unextractable residues represented 14.5% / 0.1 mg/kg.

The third study on sugar beet is on-going and no study report was available for the current evaluation.

In zRMS-PL opinion the metabolism studies should be evaluated at EU level, not at the national level.

In the framework of the new MRL application in honey (EFSA, 2024 – „Modification of the existing maximum residue level for clopyralid in honey”), the applicant submitted two new GLP and guidelines - compliant metabolism studies: one on oilseed rape and one on sugar beet (Finland, 2023). Both studies are performed with application patterns similar to the studies assessed in the peer review (foliar application at an early growth stage at the rate of 200 or 300 g a.s./ha).

EFSA, 2024: *The two new studies submitted and assessed in the framework of the present application indicate that clopyralid (free, conjugated or any forms readily converted to free clopyralid after basic hydrolysis) is the only relevant compound found in oilseed rape and sugar beet. No further metabolites were identified, thus a different degradation of the parent compound in these crops is not expected compared to wheat. In the new sugar beet study, the presence of clopyralid conjugates could be demonstrated. In the new oilseed rape study, however, the authors could only formulate the hypothesis that clopyralid may undergo interaction with endogenous plant matrix, in the form of weak chelation effects or electrostatic interactions. Nevertheless, in both cases, an impact on the magnitude of residues in samples analysed in monitoring, or in the residue trials, is not expected provided that the analytical methods used to generate the data also apply basic hydrolysis to convert all the relevant residues to clopyralid. The results of a new study on oilseed rape showed similar results compared to the previously assessed study on wheat, with a significant part of the TRR corresponding to compounds less polar than clopyralid, not properly identified, but which are readily converted to free parent after basic hydrolysis. In both cases, an impact on the magnitude of residues in samples analysed in the residue trials and in monitoring is not expected, provided that the analytical methods used to generate the data also apply basic hydrolysis in order to convert all the relevant residues to clopyralid (EFSA, 2021).*

(...) The available studies submitted after the EU peer review indicate that clopyralid (free, conjugated or any forms readily converted to free clopyralid after basic hydrolysis) is the only relevant compound found in oilseed rape, wheat and sugar beet. However, a conclusion on the metabolism of clopyralid in plants shall be taken in the MRL review according to Article 12 of Regulation (EC) No 396/2005 where all available plant metabolism studies will be reassessed.

(...) A change of the wording of the existing enforcement residue definition might need to be proposed in the framework of the MRL review (art 12 of Reg (EU) 396/2005) to better reflect these aspects of the nature of clopyralid residues in plants.

Furthermore, whether this residue definition is also applicable to crops other than cereals also needs to be concluded in the framework of the MRL review.

Conclusion:

Taking into account EFSA conclusion presented in EFSA Journal 2018;16(8):5389 for clopyralid, a general residue definition for primary and rotational crops for risk assessment and monitoring has been derived.

At present, no additional data are required to support the proposed uses on sugar beet, red beet, turnip, swede, oilseed rape, mustard, linseed and onion.

Taking the above into account and in accordance with national harmonization arrangements (<https://www.gov.pl/web/rolnictwo/ustalenia-dotyczace-sporzadzania-oceny-lub-uwag-w-zakresie-srodkow-ochrony-roslin-przez-podmioty-upowaznione>; 17.12.2024), for products containing clopyralid, after completing the procedure under Article 12 of Regulation No 396/2005, it will be necessary to verify the assessment performed in terms of the established residues definition for the accepted uses.

7.2.2.2 Nature of residue in rotational crops (KCA 6.6.1)

Available data

No new data submitted in the framework of this application.

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

Crop group	Crop	Label position	Application and sampling details						Reference
			Method, F or G *	Rate (kg a.s./ha)	Sowing intervals (DAT)	Harvest Intervals		Remarks	
						DAT	DAP		
EU data									
Leafy vegetables	Cabbage, lettuce	¹⁴ C-2-pyridine-carboxylic acid	F, Bare soil application	0.28-0.30	30 (cabbage), 125, 319 (lettuce)	Cabbage: 78 (immature), 128 (mature)	Lettuce: 38, 60, 81 and 49, 71	30 DAT mature cabbage was harvested at 128 days (9+ leaves/head; heads failed to fully close due to heat, BBCH 53)	Finland, 2018a
Root and tuber vegetables	Turnip, radish	¹⁴ C-2-pyridine-carboxylic acid	F, Bare soil application	0.28-0.30	30 (radish), 125, 319 (turnip)	Radish: 78 (BBCH 53) Turnip: 219, 390	Turnip: 71, 94	-	Finland, 2018a
Cereals	Wheat	¹⁴ C-2-pyridine-carboxylic acid	F, Bare soil application	0.28-0.30	30, 125, 319	Study 1: 244 Study 3: 62 (forage), 78 (hay), 107 (straw and grain)	Study 1: 119 Study 2: 71 (immature), 98 (grain, chaff, straw)	-	Finland, 2018a
Other	Soybean (beans and plant)	¹⁴ C-2-pyridine-carboxylic acid	F, Bare soil application	0.28	125	251	126	-	Finland, 2018a
	Green beans (beans and plant)	¹⁴ C-2-pyridine-carboxylic acid	F, Bare soil application	0.28	319	382, 390	63-71	-	Finland, 2018a

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

DAP: Days after planting

DAT: Days after treatment

Summary of plant metabolism studies reported in the EU

Reference: EFSA, 2018a

“Three nature of residues studies in three rotational crops covering the plant-back intervals (PBI) of ca. 30, 120 and 365 days are available. Only in the most recent study covering PBI of 30 days, identification of residues was performed and besides the parent only conjugated clopyralid is found in wheat, cabbage and radish. As residues in rotational crops cannot be excluded based on the available data, rotational crop field trials according to current guidelines should be submitted (data gap).”

The confined rotational crop studies were conducted at an exaggerated rate compared to the intended GAPs (1.4N), however when applied at the intended maximum use rate, residues above the LOQ (0.01 mg/kg) in rotated crops may occur at 30 days after treatment (Hall, 2015: max. of 0.549 mg/kg identified as clopyralid free and conjugated in wheat grain).

Total radioactive residues (TRR) in rotated crops 125 days after treatment (Yackovich, 1989, GH-C 2277) were a maximum of 0.015 mg/kg in soybean and wheat straw. TRR values in lettuce, turnip roots and tops and in wheat grain were <0.01 mg/kg. The wheat straw, soybean and soybean trash fractions were extracted with dilute aqueous sodium hydroxide and methanol (4 times) followed by concentration, acidification and partitioning residues into diethyl ether. This method has been proven to quantitatively measure clopyralid, free and conjugated. However, for these 125-d PBI samples, 57-65% of the TRR was extracted, with HPLC revealing that the only extracted component was clopyralid, at levels <0.01 mg/kg. The un-extracted residue was <0.01 mg/kg and therefore was not analysed further, but should not be considered to be clopyralid. Therefore, residues of clopyralid in the 125-d PBI samples were all <0.01 mg/kg and a label restriction of 125-d should be acceptable. The Yackovich study was conducted with application to bare soil at 280 g as/ha, therefore supporting uses up to 280 g as/ha and a 125-d Plant Back Interval (“PBI”). TRR values in lettuce, turnip roots and tops and in wheat grain were <0.01 mg/kg.

The metabolism in rotational crops is considered comparable to that in primary crops, therefore the same residue definitions apply.

Conclusion on metabolism in rotational crops

EFSA concluded that even if “*the metabolism studies are not performed with crop groups covering the representative uses in cereals and grass, they are sufficient to derive a general residue definition for primary and rotational crops for risk assessment and monitoring as ‘clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)’ – pending the outstanding clarification on the nature of ‘polar clopyralid’*”.

The rotational crop residue definition for monitoring and risk assessment is: *clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)’ – pending the outstanding clarification on the nature of ‘polar clopyralid’*”.

For the clarification on the nature of “polar clopyralid”, refer to the section on metabolism in primary crop and to Appendix 4 for further details.

The 30-d CRC data indicate residues will be similar or lower than the primary crop residue levels, and if no further application of clopyralid is allowed to the succeeding crops, the MRLs would not be exceeded in the succeeding crops or the current MRL of 0.5 mg/kg set for majority of crops. These data do not include the reduction expected from the primary crop intercept as this could vary depending upon the primary crop and growth stage at application.

For root vegetables, plant back interval of 30-125 days can be accepted without further restriction on the succeeding crops based on the 30-day confined rotational crop study.

In light of results from the field rotational crop study recently performed and submitted in this dossier, the following label restrictions are suggested:

“No mitigation measures are required for Leafy and Brassica vegetables or for Oilseeds. For all other food and feed commodities except sugar canes, a 30-day PBI is supported. It is recommended that sugar canes not be planted for 125 days after application of clopyralid.”

zRMS comments:

Information presented by Applicant is sufficient and acceptable.

zRMS-PL agrees that the same general residue definition set for primary crops can be applied to rotational crop.

The residue definition for monitoring and risk assessment for rotational crops: *clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)’ – pending the outstanding clarification on the nature of ‘polar clopyralid*.

EFSA concluded in EFSA Journal 2021;19(1):6389 that *Clopyralid is proposed to be used on crops that can be grown in rotation with other crops. It is considered to be low to medium persistent, forming no metabolites at levels triggering identification or further assessment; it does not accumulate in soil (EFSA, 2018d).*

From the rotational crop metabolism studies which were assessed in the framework of the EU pesticides peer review, it was concluded that the same metabolic pattern is observed across all rotational crops and that this

corresponds with the findings in primary crops with clopyralid and conjugated clopyralid being the major residues (EFSA, 2018d). The studies covered three plant back intervals (PBI), with quantifiable amounts reported at PBI of 30 days (except for radish roots) and identification performed in all plant parts. Low amounts of radioactive residues were still detected at PBI of 125 days in soybeans (0.013 mg eq/kg), soybean plant (0.015 mg eq/kg) and wheat straw (0.015 mg eq/kg).

As residues in rotational crops cannot be excluded based on these findings, the peer review recommended that rotational crop field trials according to current guidelines should be submitted (EFSA, 2018d).

The EU pesticides peer review identified a data gap for the submission of rotational crop field trials according to current guidelines, since in the metabolism studies the residues of free and conjugated parent were found in all plant parts at PHI 30 days (except in radish roots) in soybean plant and wheat straw up to 0.015 mg eq/kg at PHI 120 days (EFSA, 2018).

In the absence of rotational crop field trials the applicant and EMS proposed to apply risk mitigation measures and to restrict the use for succeeding crops.

EFSA re-iterates the data gap from the EU pesticides peer review, to request rotational crop field studies to address this data requirement. Until these studies become available EFSA suggests, to risk managers, to label clopyralid containing products with the restriction not to use clopyralid on the same field for 125 days after the initial application, regardless of the crop grown, as the risk mitigation measure (EFSA Journal 2021;19(1):6389; Clopyralid-SANTE/10206/2021, Rev. 1, 20 May 2021).

Specific mitigation measures are recommended before planting succeeding crops – see point 7.2.6.1.

No additional data are required.

7.2.2.3 Nature of residues in processed commodities (KCA 6.5.1)

Available data

No new data submitted in the framework of this application.

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

Conditions (Duration, Temperature, pH)	Identified compound(s) (%)	Reference
EU data		
Pasteurisation (20 minutes, 90°C, pH 4)	Clopyralid (99.3)	Finland, 2018a
Baking, boiling, brewing (60 minutes, 100°C, pH 5)	Clopyralid (96.9)	
Sterilisation (20 minutes, 120°C, pH 6)	Clopyralid (97.1)	

Reference: EFSA, 2018a

“Clopyralid proved to be stable under pasteurisation, baking, brewing, boiling and sterilisation conditions.”

Conclusion on nature of residues in processed commodities

Clopyralid is stable under pasteurisation, baking, brewing, boiling and sterilisation conditions. Specific residue definitions for processed commodities are not required.

zRMS comments:

The effect of processing on the nature of clopyralid was investigated in the framework of the EU pesticides peer review (EFSA, 2018). The study showed that clopyralid is hydrolytically stable under standard processing conditions.

No further data are required.

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

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

Endpoints	
Plant groups covered	Root and tuber vegetables (Sugar beet) Leafy crops (Cabbage)

	Pulses/oilseeds (Oilseed rape) Cereals (wheat)
Rotational crops covered	Radish/turnip, cabbage/lettuce, wheat, soybean (green plant and beans)
Metabolism in rotational crops similar to metabolism in primary crops?	Yes (EFSA, 2018a)
Processed commodities	Clopyralid is stable under standard hydrolysis conditions (EFSA, 2018a)
Residue pattern in processed commodities similar to pattern in raw commodities?	Yes
Plant residue definition for monitoring	Clopyralid (Regulation n°2018/1514 Reg. (EU) 2021/1807 2024/2612) During the renewal assessment EFSA proposed a residue definition of: sum of clopyralid, its salts and conjugates, expressed as clopyralid (EFSA, 2018a)*; EFSA, 2024: Cereals: Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) (EFSA, 2021) [Wording of the existing residue definition and extension to other crop groups to be assessed in the MRL review].
Plant residue definition for risk assessment	Sum of clopyralid, its salts and conjugates, expressed as clopyralid (EFSA, 2018a)* EFSA, 2024: Cereals: Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) (EFSA, 2021) [Wording of the existing residue definition and extension to other crop groups to be assessed in the MRL review].
Conversion factor from enforcement to RA	Not required

* Identified by EFSA as pending clarification on the nature of ‘polar clopyralid’. The applicant has since provided sufficient information to address the data gap (Finland, 2018b), therefore these residue definitions should no longer be considered ‘pending’. Further explanation may be found in Appendix 4.

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

Available data

No new data submitted in the framework of this application.

Table 7.2-7: Summary of animal metabolism studies

Table 7.2-1. Summary of animal metabolism studies								
Group	Species	Label position	No of animal	Application details		Sample details		Reference
				Rate (mg/kg bw/d)	Duration (days)	Commodity	Time of sampling	
EU data								
Lactating ruminants	Goat	Clopyralid-2,6- ¹⁴ C	1	0.484 (50.9 mg as/kg dry feed/day)	5	Milk	Twice daily	Finland, 2018a EFSA, 2018a
						Urine and faeces	Daily	
						Tissues	At sacrifice	
Laying poultry	Hen	Clopyralid-2,6- ¹⁴ C	10	0.56-0.65 (11.4 mg as/kg feed/day)	7	Eggs	Twice daily	Finland, 2018a EFSA, 2018a
						Excreta	Daily	
						Tissues	At sacrifice	

Summary of animal metabolism studies reported in the EU

Reference: EFSA, 2018a

“Metabolism studies both for ruminants and poultry are submitted indicating that conjugation is the major pathway; however, significant amounts of glycine conjugates were only found in milk. The conversion

factor of 1.3 for monitoring to risk assessment is only relevant for milk and is based on the new ruminant metabolism study.

The residue definition in products of animal origin for risk assessment is proposed as ‘clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid)’ and ‘clopyralid and its salts’ for monitoring. The plateau in eggs was reached at *ca.* 7 days and in milk at day 1.”

In milk over 21% of TRR (corresponding to 0.002 mg eq/kg) was found as clopyralid-glycine conjugate (X36538). Clopyralid comprised from 54% to over 70% of the TRR in milk, urine and faeces. In the tissues unchanged clopyralid was the major residue along with minor amounts of conjugate X36538.

Conclusion on metabolism in livestock

Metabolism in animals has been thoroughly characterised in rats, poultry and lactating ruminants. Metabolism is similar in the animals tested, and a pig metabolism study is not required.

A fish metabolism study is not required, as clopyralid is not expected to bioaccumulate in animal tissues as indicated by a Log P_{ow} of -2.63 and a fish bioconcentration factor of < 1.

Based on the goat and hen metabolism studies submitted and evaluated during the EU renewal, EFSA proposed the following residue definitions for animal commodities (EFSA, 2018a):

Residue definition for monitoring (animal): Clopyralid and its salts

Residue definition for risk assessment (animal): Sum of clopyralid, its salts and glycine conjugates, expressed as clopyralid

The conversion factor monitoring / risk assessment is only relevant for milk and is based on the new ruminant metabolism study as 1.3.

zRMS comments:

The information provided by the Applicant is sufficient and acceptable.

The residue definition for monitoring for animal: clopyralid (Reg. (EU) ~~2021/1807~~ 2024/2612);
clopyralid and its salts (EFSA, 2018)

The residue definition for risk assessment for animal: clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid) (EFSA, 2018)

The metabolism of clopyralid in livestock is sufficiently addressed to support the proposed uses of HCV08.

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

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

	Endpoints
Animals covered	Lactating goats
	Laying hens
Time needed to reach a plateau concentration	1 day in milk
	7 days in eggs
Animal residue definition for monitoring	Clopyralid (Regulation n°2018/1514 Reg. (EU) 2021/1807 2024/2612) During the renewal assessment EFSA proposed a residue definition of: clopyralid and its salts (EFSA, 2018a).
Animal residue definition for risk assessment	Sum of clopyralid, its salts and glycine conjugates, expressed as clopyralid (EFSA, 2018a)

Conversion factor	Milk only: 1.3 (EFSA, 2018a)
Metabolism in rat and ruminant similar	Yes
Fat soluble residue	No

In accordance with the draft fish guidance (SANCO/11187/2013), metabolism in fish is not required, as the Log Pow for clopyralid is < 3 and it is not considered fat soluble.

7.2.3 Magnitude of residues in plants (KCA 6.3)

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

New studies on the magnitude of residue have been submitted by the applicant in the framework of this application.

Studies that were submitted and evaluated during the original EU peer review of clopyralid (EFSA, 2005) and subsequent MRL evaluations (Finland, 2008 and 2011; EFSA, 2011) are also relied upon. Due to the aforementioned data gap, the relevant magnitude of residue studies have been fully summarized for all uses in Appendix 2.

Table 7.2-9: Summary of EU reported and new data supporting the intended uses of HCV07 and conformity to existing MRL

Commodity	Source	Residue zone (N-EU, S-EU, EU, outside EU)	Evaluation GAP Residue levels (mg/kg) E = RA†	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg) *	MRL compliance
Sugar beet, roots Zonal cGAP: 1 x 150-200 g ae/ha, BBCH 31-39, PHI 42d	New trials (Study No. 200809)	N-EU	Trials GAP: 1 x 125 g ae/ha, BBCH 35, PHI 42d-100-134 d E/RA: 0.10, 0.13, 0.14, 0.16, 0.17, 0.19, 2 x 0.20	N/A				
	Overall supporting data for cGAP	N-EU	Intended cGAP: 1 x 90-120 g ae/ha, BBCH 12-39, PHI 42d, outdoor E/RA: 0.10, 0.13, 0.14, 0.16, 0.17, 0.19, 2 x 0.20	0.165	0.20	0.484	1 (Sugar beet, beetroot) 1.5 (turnip and swede)	Yes
Sugar beet, tops	New trials (Study No. 200809)	N-EU	Trials GAP: 1 x 125 g ae/ha, BBCH 35, PHI 42d-100-134 d E/RA: 2 x 0.12, 0.14, 2 x 0.17, 0.19, 0.20, 0.22	N/A				
	Overall supporting data for cGAP	N-EU	Intended GAP: 1 x 90-120 g ae/ha, BBCH 12-39, PHI 42d, outdoor E/RA: 2 x 0.12, 0.14, 2 x 0.17, 0.19, 0.20, 0.22	0.17	0.22	0.499	Not relevant to animal feed commodities	
Oilseed rape	New trials (Study No. 150534)	N-EU	Trials GAP: 1 x 120 g ae/ha, BBCH 50, outdoor E/RA: <0.01, 3 x 0.02, 2 x 0.03, 0.05, 2 x 0.06, 0.10, 0.30	N/A				
	Overall supporting data for cGAP	N-EU	Intended cGAP: 1 x 120 g ae/ha, BBCH 30-51, PHI N/A, outdoor E/RA: <0.01, 3 x 0.02, 2 x 0.03, 0.05, 2 x 0.06, 0.10, 0.30	0.03	0.30	0.394	0.5 (oilseed rape, mustard seeds) 20 (linseed)	Yes
Onion	Finland, 2008	N-EU	GAP on which MRL/EU a.s. assessment is based: 100 + 200 g ae/ha ^[1] , BBCH 13-16, PHI 42-66d, outdoor E/RA: <0.01, 2 x 0.02, 0.03, 0.04, 0.05, 0.17 (scaled to 120 g ae/ha: <0.01, 0.012, 0.018, 0.03, 0.1)	N/A				

Commodity	Source	Residue zone (N-EU, S-EU, EU, outside EU)	Evaluation GAP Residue levels (mg/kg) E = RA†	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg) *	MRL compliance
	New trials (Study No. GHE-P-12680)	N-EU	Trials GAP: 100 + 200 g ae/ha ^[1] , BBCH 13-16, PHI 44d, outdoor E/RA: 0.025, 0.03 0.028 (scaled to 120 g ae/ha – 0.017 mg/kg)					
	New trials (Study GHE-P-7289)	N-EU	Trials GAP: 147-150 g ae/ha, BBCH 14-15, PHI 62d, outdoor E/RA: 0.075, 0.14 (scaled to 120 g ae/ha – 0.060 and 0.112 mg/kg)					
	Overall supporting data for cGAP	N-EU	Intended GAP: 1 x 120 g ae/ha, BBCH 11-16, PHI 42d, outdoor E/RA: <0.01, 2 0.02, 0.028, 0.025 , 0.03, 0.04 , 0.05, 0.075, 0.14, 0.17 [3] Residues scaled to 120 g ae/ha – mean scaling factor of 0.60 E/RA: <0.01, 2 0.012, 0.014 , 0.017, 0.018, 0.022 , 0.03 3 , 0.060, 0.10, 0.112	0.016 0.024	0.10 0.112	0.148 0.209	0.5	Yes

N/A: not applicable

* Source of EU MRL: Reg. (EU) ~~2021/1807~~ 2024/2612

† Residue definition for risk assessment and monitoring: Sum of clopyralid, its salts and conjugates, expressed as clopyralid.

[1] The RMS agreed that BBCH 32 and BBCH 39 can be considered the same, since cereal grain is not yet formed, only the stems are elongating between these growth stages. Therefore trials conducted at BBCH 39-45 can be considered to support the cGAP.

[2] Application occurred at BBCH 45 and the RMS considered it to be belong in the same cereals residue population with the applications that occurred at BBCH 39.

[3] Although 2 applications were made in the onion residue trials, the applications were ca. 1 month apart and it is generally accepted that it is the final application that determines the final residue, therefore these data are suitable to support the intended 1 application GAP.

7.2.3.2 Conclusion on the magnitude of residues in plants

Sugar beet is a major crop in the N-EU (central/northern regulatory zone) and **Sugar beet, tops** are an animal feed item. The critical GAP is for one application at 90-120 g ae/ha, at BBCH 12-39, PHI 42 days, which is less critical than the zonal cGAP (one application 150-200 g ae/ha, at BBCH 31-39, and 42 day PHI). To support the intended cGAP, eight trials on sugar beet are available (1 x 125 g ae/ha, BBCH 39-35, PHI 100-134). The data submitted show that no exceedance of the current EU MRL will occur. An EU MRL is not relevant for animal feed items. In accordance with SANTE/2019/12752, data on sugar beet may be extrapolated to support fodder beet, red beet, and mangels, which is the case here. The uses are considered acceptable.

zRMS comments:

Sugar beet and beetroot are the major crops in northern Europe (EU guideline Document SANCO 7525/VI/95 – rev.10.3 of 13 June 2017 and SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

Turnip and swede are the minor crops and minimum of four trials are required.

The Applicant provided one study covered a total of 12 field trials conducted on sugar beet. Eight out of twelve trials were conducted in Northern Europe in accordance (within 25% deviation) with the intended GAP of the product HCV08 (90-120 g as/ha, BBCH 12-39) following one foliar application of the formulation GF-1966 containing 720 g as clopyralid/kg, at a nominal application rate of 125 g ae/ha at BBCH 35. Samples of roots and tops were taken at harvest (BBCH 49, PHI 100-134 days).

PHI in all the NEU trials vary between 100 and 134 days with an application stage at BBCH 35. zRMS-PL considers the PHI of 42 days is not sufficiently supported by residue data and proposes F: PHI is defined by the application stage at last treatment (time elapsing between last treatment and harvest of the crop) and the BBCH should be 12-35.

Residues of clopyralid found in the treated sugar beet roots samples (application rate of 125 g ae/ha at BBCH 35) ranged from 0.10 to 0.20 mg/kg (8 NEU trials: 0.10, 0.13, 0.14, 0.16, 0.17, 0.19, 2x 0.20 mg/kg).

Residues of clopyralid found in the treated sugar beet tops samples (application rate of 125 g ae/ha at BBCH 35) ranged from 0.12 to 0.22 mg/kg (8 NEU trials: 2 x 0.12, 0.14, 2 x 0.17, 0.19, 0.20, 0.22 mg/kg).

According to the SANTE/2019/12752 the residue trials on sugar beets (0900010) may be extrapolated to beetroots (0213010), swedes/rutabagas (0213100) and turnips (0213110) when application is done before and after the forming of the edible part.

Available results show that the in force MRL of clopyralid on sugar beets, beetroots of 1 mg/kg and swedes and turnips of 1.5 mg/kg (Reg. (EU) ~~2021/1807~~ 2024/2612) will not be exceeded. The current EU MRLs for clopyralid are sufficient to support the proposed uses.

Therefore, sufficient residue trials are available to support the intended GAP uses on sugar beets, beetroots, swedes and turnips and fodder beets.

Oilseed rape is a major crop in northern Europe (central/northern regulatory zone). The intended critical GAP is for one application at 120 g ae/ha, at BBCH 30-51. To support the zonal cGAP, twelve trials on oilseed rape are available at the cGAP. One trial was not relied upon, as the residue was identified as an outlier according to the Dixon's Q-test. This approach was previously accepted by member states using the same dataset. The data submitted show that no exceedance of the current EU MRLs will occur. The uses are considered acceptable.

zRMS comments:

Oilseed rape is the major crop in northern Europe (EU guideline Document SANCO 7525/VI/95 - rev.10.3 of 13 June 2017 and SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

Linseeds and mustard seeds are the minor crops and minimum of four trials are required.

The Applicant provided one study covered a total of 12 field trials conducted on oilseed rape in Northern Europe in accordance (within 25% deviation) with the intended GAP of the product HCV08 (120 g as/ha, BBCH 30-51) following one foliar application of the formulation GF-3488, an EC formulation containing 120 g ae/L, at a nominal application rate of 120 g ae/ha at BBCH 50.

Samples of seed and straw were taken at normal commercial harvest (NCH) in the eight harvest trials. In the four

decline trials samples of seed and straw were taken at a PHI of 7 days, at NCH, and at 3 and 7 days after NCH. Samples of whole plant were taken at 0, 7 and 14 days after application.

A residue in seed in a single trial (PL07) was identified as an outlier according to the Dixon's Q-test and therefore is not relied upon.

Residues of clopyralid in oilseed rape are in the range <0.01 – 0.30 mg/kg.

According to the SANTE/2019/12752 the residue trials on rapeseeds/canola seeds (0401060) may be extrapolated to linseeds (0401010) and to mustard seeds (0401080) when application is done before and after the forming of the edible part.

Available results show that the in force MRL of clopyralid on rapeseeds and mustard seeds of 0.5 mg/kg and linseeds of 20 mg/kg (Reg. (EU) 2021/1807 2024/2612) will not be exceeded. The current EU MRLs for clopyralid are sufficient to support the proposed uses.

Therefore, sufficient residue trials are available to support the intended GAP uses on oilseed rape, linseeds and mustard seeds.

It should be noted that current MRL for clopyralid in honey is 0.05* mg/kg (Reg. (EU) 2021/1807), and according to the provided study on magnitude of residues in honey, the MRL is potentially exceeded. Until the higher MRL has been set for honey, use on melliferous target crops (oilseed rape, mustard and linseed) cannot be authorized.

The current MRL for clopyralid in honey has been changed and now is 0.15 mg/kg (Commission Regulation (EU) 2024/2612), and according to the provided study on magnitudes of residues in honey (UPL has access to this data via a letter of access), the MRL is not potentially exceeded. **The uses on melliferous target crops (oilseed rape, mustard and linseed) can be authorized.**

Onion is a major crop in northern Europe (central/northern regulatory zone). The intended critical GAP is for one application at 120 g ae/ha, at BBCH 11-16, PHI 42 days. To support this GAP, eight trials on onion in N-EU are available at a more critical GAP (1 x 100 + 1 x 200 g ae/ha, BBCH 13-16, PHI 42-66 days). The data submitted show that no exceedance of the current EU MRLs will occur. The uses are considered acceptable.

zRMS comments:

Onion is the major crop in northern Europe (EU guideline Document SANCO 7525/VI/95 - rev.10.3 of 13 June 2017 and SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

The Applicant provided four studies covered a total of 8 field trials conducted on onions (UPL has access to this data via a letter of access).

Although the residue trials applied two applications, the interval between applications was ca. 1 month, and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application.

All residues have been scaled to the cGAP rate.

- Study No. CEMS-2030/GHE-P-10805; DAS Study No. CEMS-2030

Four field trials were conducted on onions in the United Kingdom in 2003. Clopyralid was applied as LONTREL 100 (EF-1136), an SL formulation containing 100 g ae/L. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and the second at 200 g ae/ha. The first application was made at growth stage BBCH 10-11 and the second at BBCH 14-16, 42 days before harvest.

A single trial was cancelled, as the farmer harvested the crop before the end of the trial.

Trials CEMS-2030B and CEMS-2030C were conducted in Colchester, Essex (UK), the distance was less than 10 km and the dates of planting and treatment are also the same. **Thus, the two trials are not considered independent.** Therefore, the mean value of two residue results should be used as the representative value for the field trials.

In conclusion two independent trials are available.

Residues of clopyralid in onions bulb are 0.03 mg/kg and 0.05 mg/kg.

Scaled to 120 g ae/ha residues of clopyralid in onions are 0.018 and 0.03 mg/kg.

- Study No. CEMS-2346; DAS Report No. GHE-P-11080

Two independent residue trials on onions were conducted in Northern Europe. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 14-16. Samples of mature onions bulbs were taken 42-46 days after the last application (DALA).

Residues of clopyralid in onions bulb are <0.01 and 0.17 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are <0.01 and 0.1 mg/kg.

- Study No. CEMS-2696; DAS Report No. GHE-P-11272

One residue trial on onions was conducted in Northern Europe. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 13-15. Samples of mature onions bulbs were taken 42 days after the last application (DALA).

Residues of clopyralid in onions bulb are 0.02 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.012 mg/kg.

- DAS Study No. CEMS-4969; DAS Report No. GHE-P-12680

Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 13-16. Samples of mature onions bulbs were taken 44 days after the last application (DALA).

These two residue trials on onions were conducted at the same site: La Chapelle de Guinchay, Burgundy, France. The dates of planting and treatment are also the same. **Thus, the two trials are not considered independent.** Therefore, the mean value of two residue results should be used as the representative value for the field trials.

Residues of clopyralid in onions bulb are 0.025 and 0.03 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.015 and 0.018 mg/kg.

The average of the residue of clopyralid is 0.028 mg/kg, scaled to 120 g ae/ha – 0.017 mg/kg.

6 independent trials are available only.

At the request of the zRMS-PL, the applicant provided one additional study, DAS Report No. GHE-P-7289 (two trials). UPL have access to this data *via* a letter of access.

- Study Plan No. R97-030; DAS Report No. GHE-P-7289

Two residue trials on onions were conducted in Northern Europe, in Belgium. One foliar application was made with EF-I136 at a nominal rate of 1.5 L/ha (150 g as/ha) at growth stage BBCH 14-15. Samples of whole plant were taken at intervals up to harvest when onion bulb was taken, 62 days after application.

Trials R97-030A and R97-030B were conducted in Belgium, not at the same site, but the distance was ca.15 km. However, the dates of planting are different (ca. 80 days apart) and the dates of application (13 days). Thus, in our opinion, **the two trials are considered independent.**

Residues of clopyralid in onions bulb are 0.075 and 0.14 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.060 and 0.112 mg/kg.

In conclusion, Applicant provided 8 independent trials on bulbs to support the proposed use on onion.

Available results show that the in force MRL of clopyralid on onions of 0.5 mg/kg (Reg. (EU) ~~2021/1807~~ 2024/2612) will not be exceeded. The current EU MRL for clopyralid is sufficient to support the proposed use.

Therefore, sufficient residue trials are available to support the intended GAP uses on onions.

7.2.4 Magnitude of residues in livestock

7.2.4.1 Dietary burden calculation

The inputs for the dietary burden are listed below. All uses in the Article 43 procedure have been included at their worst-case residue endpoints across both residue zones (NEU/SEU). In addition, and in line with the approach in EFSA 2021, all other feed crops on which the use of clopyralid is authorized according to Regulation (EC) No 396/2005, e.g., for which the existing EU MRL is set above the LOQ, have been included. Input values for Article 43 uses that i) were not considered or ii) are more critical than those evaluated as part of EFSA 2021 have been summarized at the top of the table for clarity. As risk assessment values could not be retrieved for all uses listed, the MRL value was used. EFSA have previously noted that this can lead to a possible overestimation of the actual livestock dietary exposure to clopyralid residues. Input values can be found in Table 7.2-10.

To avoid over conservative calculations, the potato MRL (0.5 mg/kg) was not included in this assessment.

There is currently no registered GAP for potatoes, and it is unlikely that other register holders would have a registration on potatoes because clopyralid is highly phytotoxic to potatoes. DAS (now Corteva Agriscience) has a label recommendation to kill volunteer potatoes.

Table 7.2-10: Input values for the dietary burden calculation (considering the uses authorized within the Art. 43 procedure and all other feed crops on which the use of clopyralid is authorized according to Regulation (EC) No 396/2005, for which the existing EU MRL is set above the LOQ)

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input Value (mg/kg)	Comment	Input Value (mg/kg)	Comment
Feed items related to the Article 43 product renewal process				
Corn, field, forage/silage	0.60	STMR (NEU)	1.41	HR (NEU)
Corn, field, stover (fodder)	0.81	STMR (NEU)	1.95	HR (NEU)
Corn, pop (stover)	0.81	STMR (NEU)	1.95	HR (NEU)
Millet, straw	0.57	STMR (SEU)	1.86	HR (SEU)
Sorghum, forage	0.70	STMR (SEU)	0.83	HR (SEU)
Sorghum, grain, stover	0.57	STMR (SEU)	1.86	HR (SEU)
Sorghum, silage	0.42	STMR x PF (0.6) ^(a) (SEU)	0.5	HR x PF (0.6) ^(a) (SEU)
Millet, grain	0.11	STMR (SEU)		
Sorghum, grain	0.11	STMR (SEU)		
Corn, field (Maize)	0.30	STMR (NEU)		
Corn, pop	0.30	STMR (NEU)		
Corn, field, milled by-products	0.30	STMR (NEU) x PF(1) ^(a)		
Corn, field, hominy meal	1.80	STMR (NEU) x PF(6) ^(a)		
Corn, field, gluten feed	0.75	STMR (NEU) x PF(2.5) ^(a)		
Corn, field, gluten meal	0.65	STMR (NEU) x PF(1) ^(a)		
Feed items related to EFSA Journal 2021;19(1):6389 https://doi.org/10.2903/j.efsa.2021.6389				
Barley, straw	0.91	STMR (Barley, NEU)	1.86	HR (Barley, NEU)
Grass, forage (fresh)	7.08	STMR (NEU)	15.16	HR (NEU)
Grass, hay	24.78	STMR (NEU) x PF (3.5) ^(a)	53.2	HR (NEU) x PF (3.5) ^(a)
Grass, silage	11.33	STMR (NEU) x PF (1.6) ^(a)	24.32	HR (NEU) x PF (1.6) ^(a)
Oat, straw ^(b)	0.58	STMR (Wheat, SEU)	2.17	HR (Wheat, NEU)
Rye, straw ^(b)	0.58	STMR (Wheat, SEU)	2.17	HR (Wheat, NEU)
Triticale, straw ^(b)	0.58	STMR (Wheat, SEU)	2.17	HR (Wheat, NEU)
Wheat, straw ^(b)	0.58	STMR (Wheat, SEU)	2.17	HR (Wheat, NEU)
Barley, grain	0.33	STMR (Barley, SEU)		
Oat, grain ^(b)	0.72	STMR (Wheat, NEU)		
Rye, grain ^(b)	0.72	STMR (Wheat, NEU)		
Triticale, grain ^(b)	0.72	STMR (Wheat, NEU)		
Wheat, grain ^(b)	0.72	STMR (Wheat, NEU)		
Brewer's grain, dried	1.09	STMR (Barley, SEU) x PF (3.3) ^(a)		
Distiller grain, dried	2.38	STMR (Wheat, NEU) x PF (3.3) ^(a)		
Wheat gluten, meal	1.3	STMR (Wheat, NEU) x PF (1.8) ^(a)		
Wheat, milled by products	5.04	STMR (Wheat, NEU) x PF (7) ^(a)		

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input Value (mg/kg)	Comment	Input Value (mg/kg)	Comment
Feed items related to the previous evaluations (as considered in the evaluation report by the EMS (Finland, 2018b))				
Beet mangel, fodder	0.47	STMR (EFSA, 2005)	1.05	HR (EFSA, 2005)
Beet, sugar (tops)	0.47	STMR (EFSA, 2005)	1.05	HR (EFSA, 2005)
Cabbage, heads (leaves)	0.23	STMR (EFSA, 2011)	1.52	HR (EFSA, 2011)
Corn, field (stover)^(c)	0.46	STMR (Finland, 2018b)	0.88	HR (Finland, 2018b)
Corn, pop (stover)^(c)	0.46	STMR (Finland, 2018b)	0.88	HR (Finland, 2018b)
Kale	1	MRL	1	MRL
Turnip, tops (leaves)	0.47	STMR (EFSA, 2005)	1.05	HR (EFSA, 2005)
Swede, roots	0.35	STMR (EFSA, 2011)	0.80	HR (EFSA, 2011)
Turnip, roots	0.35	STMR (EFSA, 2011)	0.80	HR (EFSA, 2011)
Maize, field (grain)^(c)	0.06	STMR (Finland, 2008)		
Maize, pop (grain)^(c)	0.06	STMR (Finland, 2008)		
Cotton	0.5	MRL		
Pea, seed (dry)	0.5	MRL		
Soybean seed	0.5	MRL		
Apple, pomace (wet)	0.05	STMR (EFSA, 2005) x PF (5) ^(a)		
Beet, sugar (dried pulp)	6.30	STMR (EFSA, 2005) x PF (18) ^(a)		
Beet, sugar (ensiled pulp)	1.05	STMR (EFSA, 2005) x PF (3) ^(a)		
Beet, sugar (molasses)	9.80	STMR (EFSA, 2005) x PF (28) ^(a)		
Canola (Rape seed), meal	0.06	STMR (Finland, 2018b) x PF (2) ^(a)		
Citrus, dried pulp	5.00	MRL x PF (10) ^(a)		
Corn, field, milled by products	0.06	STMR (Finland, 2008) x PF(1)^(c)		
Corn, field, hominy meal	0.36	STMR (Finland, 2008) x PF(6)^(c)		
Corn, field, gluten feed	0.15	STMR (Finland, 2008) x PF(2.5)^(c)		
Corn, field, gluten meal	0.06	STMR (Finland, 2008) x PF(1)^(c)		
Cotton, meal	0.65	MRL x PF (1.3) ^(a)		
Flaxseed/Linseed, meal	8.92	STMR (EFSA, 2011) x PF (2) ^(a)		
Peanut, meal	2.00	MRL x PF (2) ^(a)		
Rape, meal	0.06	STMR x PF (2) ^(a,d)		
Soybean, meal	0.65	MRL x PF (1.3) ^(a)		
Soybean, hulls	6.50	MRL x PF (13) ^(a)		
Sunflower, meal	1.00	MRL x PF (2) ^(a)		

(a): Default processing factors as inserted in the Animal model 2017 were used.

(b): Wheat grain and straw data are extrapolated to oats and rye.

(c): Corn, field (stover) and corn, pop (stover) as well as grain are replaced with more critical data from the submitted maize study 201513.

(d): EFSA 2021 indicates an MRL was used as the input value. However, based on the burden calculation results and the fact that the EU cGAP for canola/rape seed is known, the input value was almost certainly an STMR.

The results of the dietary burden calculations are summarised in the following table.

Table 7.2-11: Results of the dietary burden calculation (without consideration of residues associated with rotational crops)

Relevant groups	Dietary burden expressed in				Most critical diet (a)	Most critical commodity (b)		Trigger exceeded (Yes/No)
	mg/kg bw per day		mg/kg DM					0.004
	Median	Maximum	Median	Maximum				mg/kg bw
Cattle (all diets)	0.761	1.521	19.79	39.55	Dairy cattle	Grass	forage (fresh)	Yes
Cattle (dairy only)	0.761	1.521	19.79	39.55	Dairy cattle	Grass	forage (fresh)	Yes
Sheep (all diets)	0.914	1.934	27.41	58.01	Ram/Ewe	Grass	forage (fresh)	Yes
Sheep (ewe only)	0.914	1.934	27.41	58.01	Ram/Ewe	Grass	forage (fresh)	Yes
Swine (all diets)	0.221	0.407	9.58	17.62	Swine (breeding)	Grass	forage (fresh)	Yes
Poultry (all diets)	0.149	0.204	2.18	2.98	Poultry layer	Cabbage, heads	leaves	Yes
Poultry (layer only)	0.149	0.204	2.18	2.98	Poultry layer	Cabbage, heads	leaves	Yes

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

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

At the time of submission, the dietary burden intakes are within those calculated in the EFSA MRL Reasoned Opinion (EFSA, 2021), which set the animal MRLs in Reg. (EU) 2021/1807 (now Reg. (EU) 2024/2612 is in force; conclusions are still valid). Therefore the new animal MRLs will accommodate the Article 43 uses presented in this submission.

To account for residues in rotational crops, residues associated with study 190557 have been considered as part of an additional dietary burden calculation. Rotational STMR and HR values have been calculated based on the worse case values across different treatment rates at the 30-day PBI (*e.g.*, residues in OSR whole plant were higher in the 80 g a.e./ha plot than the 125 g a.e./ha plot at the 30-day PBI; therefore, the former values were used). These rotational input values have been extrapolated to similar crops (*e.g.*, residues in wheat/barley whole plant have been extrapolated to other cereal forage items). Input values, extrapolations, and overall data selection are detailed in Table 7.2-12.

Calculated rotational STMR and HR values at the 30-day PBI have been summed with any inputs arising from residues associated with primary uses, thereby giving a worse case dietary burden calculation (*i.e.*, an additive scenario). Where the rotational STMR or HR was <0.01 mg/kg (<LOQ), the rotational residue value was not added to the primary crop input value, assuming a primary use existed. Where rotational residues were present and no primary use exists, input values in the dietary burden contain only residues associated with the crop rotation study. Input values used for the dietary burden calculation can be found in Table 7.2-13, which is effectively an extension of Table 7.2-10 to account for rotational residues.

Table 7.2-12: Rotational residue input values, applicability and data source.

Crop	Rate g ae/ha	PBI days	Residue mg/kg	STMR	HR
Radish root → Carrot, Cassava, Swede, Turnips, and Potato	125	30	4x <0.01*, 0.027, 0.042	<0.01	0.042
Radish tops with leaves → Beet, mangel fodder, Beet, sugar tops, and Turnip tops	125	30	3x <0.01, 0.017**, 0.541, 0.627	<0.01	0.627
Oilseed, whole plant → Rape forage	80	30	4x <0.01, 0.113, 0.212	<0.01	0.212
Cereals, whole plant → Barley, Wheat, Maize, Millet, Sorghum, Rye, Triticale, Oat and Sugar canes forage	125	30	2x <0.01, 0.034, 0.195, 0.515, 0.583	0.115	0.583
Cereals, grain → Barley, Wheat, Maize, Millet, Sorghum, Rye, Triticale, and Oat grain	125	30	2x <0.01, 0.035, 0.053, 0.134, 0.167	0.044	0.167
Cereals, straw → Barley, Wheat, Maize, Millet, Sorghum, Rye, Triticale, Oat, and Rice straw	125	30	2x <0.01, 0.10, 0.159, 0.183, 0.279	0.130	0.279

* One residue value of <0.01 is excluded from the evaluation due to exceedance of FSS.

** Residue value 0.017 is excluded from the evaluation due to exceedance of FSS.

Table 7.2-13: Input values for the dietary burden calculation (considering residues in rotation, the uses authorized within the Art. 43 procedure and all other feed crops on which the use of clopyralid is authorized according to Regulation (EC) No 396/2005, for which the existing EU MRL is set above the LOQ)

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input Value (mg/kg)	Comment	Input Value (mg/kg)	Comment
Feed items related to residues in rotated commodities without primary clopyralid uses. Values taken from study 190557				

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input Value (mg/kg)	Comment	Input Value (mg/kg)	Comment
Barley, forage	0.12	STMR _{rot}	0.583	HR _{rot}
Barley, silage	0.15	STMR _{rot} x PF (1.3) ^(a)	0.76	HR _{rot} x PF (1.3) ^(a)
Millet, forage	0.12	STMR _{rot}	0.583	HR _{rot}
Oat, forage	0.12	STMR _{rot}	0.58	HR _{rot}
Oat, hay	0.35	STMR _{rot} x PF (3) ^(a)	1.75	HR _{rot} x PF (3) ^(a)
Rape, forage	0.01	STMR _{rot}	0.212	HR _{rot}
Rice, straw	0.13	STMR _{rot}	0.279	HR _{rot}
Rye, forage	0.12	STMR _{rot}	0.583	HR _{rot}
Triticale, forage	0.12	STMR _{rot}	0.583	HR _{rot}
Triticale, hay	0.12	STMR _{rot}	0.583	HR _{rot}
Wheat, forage	0.12	STMR _{rot}	0.58	HR _{rot}
Wheat, hay	0.40	STMR _{rot} x PF (3.5) ^(a)	2.04	HR _{rot} x PF (3.5) ^(a)
Carrot, culls	0.01	STMR _{rot}	0.042	HR _{rot}
Cassava/tapioca	0.01	STMR _{rot}	0.042	HR _{rot}
Potato	0.01	STMR _{rot}	0.042	HR _{rot}
Potato, process waste	0.01	STMR _{rot} x PF (1)		
Potato, dried pulp	0.01	STMR _{rot} x PF (1)		
Sugarcane, molasses	3.68	STMR _{rot} x PF (32) ^(a)		
Feed items related to the Article 43 product renewal process				
Corn, field, forage/silage	0.72	STMR (NEU) + STMR _{rot}	1.99	HR (NEU) + HR _{rot}
Corn, field, stover (fodder)	0.94	STMR (NEU) + STMR _{rot}	2.23	HR (NEU) + HR _{rot}
Corn, pop (stover)	0.94	STMR (NEU) + STMR _{rot}	2.23	HR (NEU) + HR _{rot}
Millet, straw	0.70	STMR (NEU) + STMR _{rot}	2.14	HR (NEU) + HR _{rot}
Sorghum, forage	0.82	STMR (NEU) + STMR _{rot}	1.41	HR (NEU) + HR _{rot}
Sorghum, grain, stover	0.70	STMR (NEU) + STMR _{rot}	2.14	HR (NEU) + HR _{rot}
Sorghum, silage	0.49	STMR x PF (0.6) ^(a) (SEU) + STMR _{rot}	0.85	HR x PF (0.6) ^(a) (SEU) + HR _{rot}
Millet, grain	0.154	STMR (NEU) + STMR _{rot}		
Sorghum, grain	0.154	STMR (NEU) + STMR _{rot}		
Corn, field (Maize)	0.30	STMR (NEU) + STMR _{rot}		
Corn, pop	0.30	STMR (NEU) + STMR _{rot}		
Corn, field, milled by-products	0.34	STMR (NEU) + STMR _{rot} x PF(1) ^(a)		
Corn, field, hominy meal	2.06	STMR (NEU) + STMR _{rot} x PF(6) ^(a)		
Corn, field, gluten feed	0.86	STMR (NEU) + STMR _{rot} x PF(2.5) ^(a)		
Corn, field, gluten meal	0.34	STMR (NEU) + STMR _{rot} x PF(1) ^(a)		
Feed items related to EFSA Journal 2021;19(1):6389 https://doi.org/10.2903/j.efsa.2021.6389				
Barley, straw	1.04	STMR (Barley, NEU) + STMR _{rot}	2.14	HR (Barley, NEU) + HR _{rot}
Grass, forage (fresh)	7.08	STMR (NEU)	15.16	HR (NEU)
Grass, hay	24.78	STMR (NEU) x PF (3.5) ^(a)	53.2	HR (NEU) x PF (3.5) ^(a)

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input Value (mg/kg)	Comment	Input Value (mg/kg)	Comment
Grass, silage	11.33	STMR (NEU) x PF (1.6) ^(a)	24.32	HR (NEU) x PF (1.6) ^(a)
Oat, straw ^(b)	0.71	STMR (Wheat, SEU) + STMR _{rot}	2.45	HR (Wheat, NEU) + HR _{rot}
Rye, straw ^(b)	0.71	STMR (Wheat, SEU) + STMR _{rot}	2.45	HR (Wheat, NEU) + HR _{rot}
Triticale, straw ^(b)	0.71	STMR (Wheat, SEU) + STMR _{rot}	2.45	HR (Wheat, NEU) + HR _{rot}
Wheat, straw ^(b)	0.71	STMR (Wheat, SEU) + STMR _{rot}	2.45	HR (Wheat, NEU) + HR _{rot}
Barley, grain	0.37	STMR (Barley, SEU) + STMR _{rot}		
Oat, grain ^(b)	0.76	STMR (Wheat, NEU) + STMR _{rot}		
Rye, grain ^(b)	0.76	STMR (Wheat, NEU) + STMR _{rot}		
Triticale, grain ^(b)	0.76	STMR (Wheat, NEU) + STMR _{rot}		
Wheat, grain ^(b)	0.76	STMR (Wheat, NEU) + STMR _{rot}		
Brewer's grain, dried	1.23	STMR (Barley, SEU) + STMR _{rot} x PF (3.3) ^(a)		
Distiller grain, dried	2.52	STMR (Wheat, NEU) + STMR _{rot} x PF (3.3) ^(a)		
Wheat gluten, meal	1.38	STMR (Wheat, NEU) + STMR _{rot} x PF (1.8) ^(a)		
Wheat, milled by products	5.35	STMR (Wheat, NEU) + STMR _{rot} x PF (7) ^(a)		
Feed items related to the previous evaluations (as considered in the evaluation report by the EMS (Finland, 2018b))				
Beet mangel, fodder	0.47	STMR (EFSA, 2005)	1.68	HR (EFSA, 2005) + HR _{rot}
Beet, sugar (tops)	0.47	STMR (EFSA, 2005)	1.68	HR (EFSA, 2005) + HR _{rot}
Cabbage, heads (leaves)	0.23	STMR (EFSA, 2011)	1.52	HR (EFSA, 2011)
Corn, field (stover) ^(c)	0.46	STMR (Finland, 2018b)	0.88	HR (Finland, 2018b)
Corn, pop (stover) ^(c)	0.46	STMR (Finland, 2018b)	0.88	HR (Finland, 2018b)
Kale	1	MRL	1	MRL
Turnip, tops (leaves)	0.47	STMR (EFSA, 2005)	1.08	HR (EFSA, 2005) + HR _{rot}
Swede, roots	0.35	STMR (EFSA, 2011)	0.842	HR (EFSA, 2011) + HR _{rot}
Turnip, roots	0.35	STMR (EFSA, 2011)	0.842	HR (EFSA, 2011) + HR _{rot}
Maize, field (grain)	0.09	STMR (Finland, 2008) + STMR _{rot}		
Maize, pop (grain)	0.09	STMR (Finland, 2008) + STMR _{rot}		
Cotton	0.5	MRL		
Pea, seed (dry)	0.5	MRL		
Soybean seed	0.5	MRL		
Apple, pomace (wet)	0.05	STMR (EFSA, 2005) x PF (5) ^(a)		
Beet, sugar (dried pulp)	6.30	STMR (EFSA, 2005) x PF (18) ^(a)		
Beet, sugar (ensiled pulp)	1.05	STMR (EFSA, 2005) x PF (3) ^(a)		
Beet, sugar (molasses)	9.80	STMR (EFSA, 2005) x PF (28) ^(a)		
Canola (Rape seed), meal	0.06	STMR (Finland, 2018b) x PF (2) ^(a)		
Citrus, dried pulp	5.00	MRL x PF (10) ^(a)		
Corn, field, milled by products	0.30	STMR (Finland, 2008) + STMR _{rot} x PF (1) ^(c)		
Corn, field, hominy meal	1.80	STMR (Finland, 2008) + STMR _{rot} x PF (6) ^(c)		

Feed commodity	Median dietary burden		Maximum dietary burden	
	Input Value (mg/kg)	Comment	Input Value (mg/kg)	Comment
Corn, field, gluten feed	0.75	$STMR_{(Finland, 2008)} + STMR_{rot} \times PF(2.5)^{(a)}$		
Corn, field, gluten meal	0.65	$STMR_{(Finland, 2008)} + STMR_{rot} \times PF(1)^{(a)}$		
Cotton, meal	0.65	MRL x PF (1.3) ^(a)		
Flaxseed/Linseed, meal	8.92	STMR (EFSA, 2011) x PF (2) ^(a)		
Peanut, meal	2.00	MRL x PF (2) ^(a)		
Rape, meal	0.06	STMR x PF (2) ^(a,d)		
Soybean, meal	0.65	MRL x PF (1.3) ^(a)		
Soybean, hulls	6.50	MRL x PF (13) ^(a)		
Sunflower, meal	1.00	MRL x PF (2) ^(a)		

STMR_{rot}: STMR calculated from rotation crop residue data at the 30d plant-back interval or worst case value at a later plant back.

HR_{rot}: HR calculated from rotation crop residue data at the 30d plant-back interval or worst case value at a later plant back.

(a): Default processing factors as inserted in the Animal model 2017 were used.

(b): Wheat grain and straw data are extrapolated to oats and rye.

(c): Corn, field (stover) and corn, pop (stover) as well as grain are replaced with more critical data from the submitted maize study 201513.

(d): EFSA 2021 indicates an MRL was used as the input value. However, based on the burden calculation results and the fact that the EU cGAP for canola/rape seed is known, the input value was almost certainly an STMR.

The results of the dietary burden containing inputs for rotational residues is presented in Table 7.2 14.

Table 7.2-14: Results of the dietary burden calculation (with consideration of residues associated with crop rotation at the 30d plant-back interval)

Relevant groups	Dietary burden expressed in				Most critical diet (a)	Most critical commodity (b)		Trigger exceeded (Yes/No)
	mg/kg bw per day		mg/kg DM					0.004
	Median	Maximum	Median	Maximum				mg/kg bw
Cattle (all diets)	0.761	1.524	19.79	39.63	Dairy cattle	Grass	forage (fresh)	Yes
Cattle (dairy only)	0.761	1.524	19.79	39.63	Dairy cattle	Grass	forage (fresh)	Yes
Sheep (all diets)	0.914	1.934	27.41	58.03	Ram/Ewe	Grass	forage (fresh)	Yes
Sheep (ewe only)	0.914	1.934	27.41	58.03	Ram/Ewe	Grass	forage (fresh)	Yes
Swine (all diets)	0.225	0.414	9.75	17.93	Swine (breeding)	Grass	forage (fresh)	Yes
Poultry (all diets)	0.158	0.214	2.31	3.12	Poultry layer	Cabbage, heads	leaves	Yes
Poultry (layer only)	0.158	0.214	2.31	3.12	Poultry layer	Cabbage, heads	leaves	Yes

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

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

A comparison between the dietary burdens with and without consideration of rotational inputs is presented in Table 7.2-15. For ruminants and swine, consideration of rotational residues results in a maximum burden increase of 1.8%, which illustrates that primary crop residues are driving the dietary burden calculation (principally, grass forage). For poultry, the maximum burden increase is 6.1%, again indicating that primary crop residues are driving the dietary burden calculation (principally, cabbage).

Table 7.2-15: Comparison of dietary burden calculation with consideration of rotated residues expressed as % increase in burden relative to values presented in Table 7.2-11

Relevant groups	Dietary burden expressed in			
	mg/kg bw per day		mg/kg DM	
	Median % Increase	Maximum % Increase	Median % Increase	Maximum % Increase
Cattle (all diets)	0.0	0.2	0.0	0.2
Cattle (dairy only)	0.0	0.2	0.0	0.2
Sheep (all diets)	0.0	0.0	0.0	0.0
Sheep (ewe only)	0.0	0.0	0.0	0.0
Swine (all diets)	1.8	1.7	1.8	1.7
Poultry (all diets)	6.1	4.8	6.1	4.8
Poultry (layer only)	6.1	4.8	6.1	4.8

(%): percent increase when considering rotated residues VS primary crop inputs alone.

For the livestock assessed, the increase in dietary burden results in estimated residues in animal commodities that are within the new EU MRLs (Reg. (EU) ~~2021/1807~~ 2024/2612). Furthermore, no dietary risk assessment concerns are noted. A minimum 30-day PBI for the livestock feed uses assessed within the dietary burden calculation is supported.

zRMS comments:

Calculations presented by Applicant are sufficient and acceptable.

The calculated dietary burdens for all groups of livestock were found to exceed the trigger value of 0.004 mg/kg bw/day. Further investigation of clopyralid residues is therefore required in all commodities of animal origin.

7.2.4.1 Livestock feeding studies (KCA 6.4.1-6.4.3)

Available data

No new data were submitted in the framework of this application. As the dietary burden intakes are within those calculated in the EFSA MRL Reasoned Opinion (EFSA, 2021), the existing animal MRLs accommodate the Article 43 uses presented in this submission (including consideration of rotational residues) and no further evaluation is warranted.

Livestock feeding studies were evaluated during renewal of clopyralid and are summarised below.

Reference: EFSA, 2018a

“GLP- and guideline-compliant feeding studies with poultry and cattle analysing for all compounds covered by the residue definition for risk assessment and within a time period covered by storage stability data were presented. Residues in poultry matrices at the highest dose group at sampling day 28 were highest in eggs (up to 0.046 mg/kg), followed by liver (up to 0.034 mg/kg) and muscle (up to 0.017 mg/kg), whereas very little residue were quantified in fat (0.005 mg/kg). Residues in cow’s milk were in the highest dosing group already at day 2 (up to 0.0175 mg/kg) and remained at this level. Residue levels at the highest dose group were observed also in all other organs (up to 0.484 mg/kg in muscle, up to 1.962 g/kg in liver, up to 25.3 mg/kg in kidney and up to 2.131 mg/kg in average fat).”

Conclusion on feeding studies

The requested uses do not modify the theoretical maximum daily intake for animals, and there is no risk for animal MRLs to be exceeded.

zRMS comments:

Regarding available feeding data, there is no risk for animal MRLs to be exceeded.

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

7.2.5.1 Available data for all crops under consideration

Clopyralid is stable under pasteurisation, baking, brewing, boiling and sterilisation conditions (EFSA, 2018a).

New data are submitted in the framework of this application.

Table 7.2-16: Overview of the available processing studies

Processed commodity	Number of studies	Median PF *	Median CF **	Comments	Reference
EU data					
Sum of clopyralid, its salts and conjugates, expressed as clopyralid					
Oilseed rape / oil	15	0.1	NA		EFSA, 2005
New data					
Sugar beet / pulp	3	0.7	N/A	-	Report GH-C 3305 and Report No. 181493
Sugar beet / molasses	3	4.4	N/A	-	
Sugar beet / brown sugar	2	0.9	N/A	-	
Sugar beet / sugar juice	2	0.5	N/A	-	
Sugar beet / non-refined sugar	2	4.1	N/A	-	
Sugar beet / white sugar	3	0.3	N/A	-	

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

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

7.2.5.2 Conclusion on processing studies

Residues on the intended uses do exceed the trigger value for processing studies (0.1 mg/kg). Data on sugar beet processed commodities summarised above are used to refine dietary burden and consumer intakes where necessary. Default processing factors can be used for all other commodities.

zRMS comments:

Processing data were evaluated in the EU review for clopyralid for wheat and barley (EFSA, 2018). Processing studies are normally required if the supervised residue trials reveal that the total residues in commodities exceed the trigger value of 0.1 mg/kg, or if the total Theoretical Maximum Daily Intake (TMDI) is more than 10% of the ADI. Based on these data requirements, processing studies are required for oilseed rape and sugar beet.

Oilseed rape (Clopyralid – Volume 3, Annex B, B.7 Residue data, DAR 2003)

A total of 15 studies were conducted during 1975-1985 to determine the residues in rapeseed oil and cake following both spring and autumn application of clopyralid. Samples of seed were obtained at normal harvest time. The seed was then pressed to produce oil and cake. The seed, oil and cake were analysed for clopyralid residues by using a gas chromatographic method.

No concentration of clopyralid residues was occurred in oil samples. Instead, concentration in cake was observed in some cases, and a conservative concentration factor is 3.

Remark:

It should be noted that during the renewal of the clopyralid data, the processing studies on oilseed rape were not re-evaluated. Studies investigating residues during oil production from oilseed rape were not reported in Appendix 2.

Therefore, in our opinion, the median PF derived for oil from oilseed rape is considered indicative only.

Sugar beet (new data)

Two new processing studies have been submitted by the Applicant in the framework of this application. The studies demonstrate that clopyralid residues concentrate in sugar beet / molasses and sugar beet / non-refined sugar whereas a reduction is observed in other processed commodities of sugar beet. No additional data are required.

Processing factors (PF) derived for sugar beet and oilseed rape were not used neither in the dietary burden calculation nor in the consumer risk assessment calculation. More critical default PF were used instead.

7.2.6 Magnitude of residues in representative succeeding crops

The crops under consideration can be grown in rotation. Three nature of residues studies in three rotational crops covering the plant-back interval (PBI) of *ca* 30, 120 and 365 days are available. According to the results of the confined rotational crop studies submitted in the framework of clopyralid's renewal process, residues above the LOQ may be expected in rotational crops. Therefore, EFSA recommended the submission of rotational field crop studies to either confirm the proposed MRLs or to modify the proposed MRLs if necessary (EFSA, 2018a).

Data dealing with magnitude of residues in succeeding crops are available/have been submitted and are summarized hereafter.

7.2.6.1 Field rotational crop studies (KCA 6.6.2)

Available data

A new study for residues in succeeding crops has been submitted by the applicant in the framework of this application. The study is summarized in the table below. The detailed results are presented in Appendix 2.

Table 7.2-17: Summary of available studies in field rotational crops

Summary of available studies in field rotational crops					
Primary crop	Rate (g ae/ha) (GS at application or PHI)	Residue levels in succeeding crops			
		Succeeding crop group	Succeeding crop	Sowing intervals (DAT)	Reference / Remarks
New data					
Bare soil	3 rates: 60, 80, 125 (1 application to achieve targeted plantback intervals)	Leafy and Brassica vegetables	Head cabbage	30, 90, 125, 270-365	Devine, C., 2021, Study No. 190557
		Root and tuber vegetables	Radish		
		Oilseeds	Oilseed rape, Sunflower		
		Cereals	Barley, Wheat		

Radish root and radish tops with leaves samples from three sites in Northern France, Poland and Southern France were stored for longer than 443 days and were excluded from further evaluations to determine plant-back intervals and the impact of rotational crops on livestock dietary burdens.

Residues of clopyralid in head cabbage were <0.01 mg/kg at all application rates and at all plantback intervals (PBI). Residues of clopyralid in radish roots ranged from <0.01 to 0.042 mg/kg at all application rates at the shortest PBI of 30 days and were < 0.01 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in oilseed rape and sunflower seeds were <0.01 mg/kg at all application rates and at all PBI. Residues of clopyralid in barley and wheat grain ranged from <0.01 to 0.17 mg/kg at all application rates at the shortest PBI of 30 days and ranged from <0.01 to 0.037 mg/kg at all application rates at a 90-day or longer PBI.

The residues of clopyralid in rotational crops representative of food commodities are all well below the established EU MRLs which, for most food crop commodities, are 0.5 mg/kg or higher (

Table 7.2-18). Further, the EU MRL for chicory roots is set at a lower (default) value of 0.05 mg/kg, which is still higher than the maximum residue value that was observed for the representative root crop (*i.e.*, 0.042 mg/kg for radish roots at 30-day PBI at 125 g ae/ha treatment rate).

Table 7.2-18: Summary of Clopyralid Residues in Representative Food Commodities at Treatment Rate 125 g ae/ha and 30-day PBI and EU MRL Compliance

Representative Food Commodity → Crop Group	Residue levels (mg/kg)	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg) *	MRL compliance
Head cabbage → Leafy and Brassica vegetables	6x <0.01	0.01	0.01	0.010	0.5 (Leafy vegetables) 1 (Leafy brassicas) 3 (Head cabbage cauliflower)	Yes
Radish root → Root and tuber vegetables	4 x <0.01*, 0.027, 0.042	0.01	0.042	0.072	0.5 (Potato, carrot, radish) 1 (Beetroots, sugarbeets) 1.5 (Turnips, swedes)	Yes
Oilseed rape, Sunflower seed → Oilseeds	6x <0.01	0.01	0.01	0.010	0.5 (Oilseeds) 20 (Linseed)	Yes
Barley, Wheat grain → Cereals grain	2x <0.01, 0.035, 0.053, 0.13, 0.17	0.044	0.17	0.335	2 (Barley, maize, rice) 3 (Wheat, oat) 5 (Rye)	Yes

* One residue value of <0.01 mg/kg is excluded from the evaluation due to exceedance of frozen storage stability.

For livestock feed commodities, residues of clopyralid in radish tops with leaves ranged from <0.01 to 0.54 mg/kg at all application rates at the shortest PBI of 30 days and ranged from <0.01 to 0.025 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in oilseed rape and sunflower whole plant (forage commodity) ranged from <0.01 to 0.21 mg/kg at all application rates at the shortest PBI of 30 days and were <0.01 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in the rest of the oilseed rape and sunflower plants after seed harvest ranged from <0.01 to 0.089 mg/kg at all application rates at the shortest PBI of 30 days and were <0.01 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in barley and wheat whole plant (forage commodity) ranged from <0.01 to 0.58 mg/kg at all application rates at the shortest PBI of 30 days, ranged from <0.01 to 0.084 mg/kg at all application rates at the 90-day PBI, and ranged from <0.01 to 0.017 at all application rates at a 125-day or longer PBI. Residues of clopyralid in the rest of the barley and wheat plants after seed harvest ranged from <0.01 to 0.28 mg/kg at all application rates at the shortest PBI of 30 days and ranged from <0.01 to 0.036 mg/kg at all application rates at a 90-day or longer PBI.

The EU MRL for sugar canes, which can be represented by cereal forages, is 0.05 mg/kg (default). Based on the available whole plant data, it is recommended that sugar canes not be planted for 125 days after application of clopyralid.

EU MRLs are not currently set on livestock feed commodities; however, the potential clopyralid residues in rotational crops were considered when estimating livestock dietary burdens. The potential clopyralid residues in rotational crops do not significantly increase livestock dietary burdens, which are driven by registered uses on cereals and pasture. For the livestock assessed, the small increases in dietary burdens

result in estimated residues in animal commodities that are within the new EU MRLs under Reg. (EU) 2021/1807 2024/2612. Therefore, a minimum 30-day PBI for the livestock feed uses assessed within the dietary burden calculation is supported.

Conclusion on rotational crops studies

Residues of clopyralid in rotational crop food commodities are anticipated to be less than the established EU MRLs. Residues in rotational crop livestock feed commodities were taken into consideration when estimating the livestock dietary burdens. No mitigation measures are required for Oilseeds. For all other food and feed commodities except sugar canes, a 30-day PBI is supported. It is recommended that sugar canes not be planted for 125 days after application of clopyralid.

zRMS comments:

The EU pesticides peer review identified a data gap for the submission of rotational crop field trials according to current guidelines, since in the metabolism studies the residues of free and conjugated parent were found in all plant parts at PHI 30 days (except in radish roots) in soybean plant and wheat straw up to 0.015 mg eq/kg at PHI 120 days (EFSA, 2018d).

In the absence of rotational crop field trials the applicant and EMS proposed to apply risk mitigation measures and to restrict the use for succeeding crops.

EFSA re-iterates the data gap from the EU pesticides peer review, to request rotational crop field studies to address this data requirement. Until these studies become available EFSA suggests, to risk managers, to label clopyralid containing products with the restriction not to use clopyralid on the same field for 125 days after the initial application, regardless of the crop grown, as the risk mitigation measure (EFSA Journal 2021;19(1):6389; Clopyralid-SANTE/10206/2021, Rev. 1, 20 May 2021).

To address this data requirement and consequent risk mitigation, the Applicant provided access to a new study (Devine, C., 2021; CEMS-9009/190557). This study has been previously submitted to Poland for a clopyralid product by Corteva (PPP of EF-243) in December 2021 as part of their art. 43 dossier, and was evaluated by zRMS on 05 December 2022. The applicant has access to this study.

Six crop rotational trials were conducted in northern Europe (three trials) and Southern Europe (three trials) to determine residues of clopyralid in rotational crops at harvest, following one application to bare soil. GF-1966 (720 g ae/kg clopyralid) was applied once at a nominal rate of 60, 80, or 125 g ae/ha with an application volume of 200-400 L/ha. Following plant back intervals (PBI) of approximately 30, 125 and 270-365 days the plots were planted with a root crop, leafy crop, cereal, or oilseed.

The application was made on bare soil and the highest dose of 125 g ae/ha is in accordance (within 25% deviation) with the intended annual critical dose for all crops.

Residue above 0.01 mg/kg is expected in rotational crops at 30-day PBI at 125 g ae/ha treatment rate. However the residue levels coming from rotational crops are not expected to lead to MRL exceedances except for sugar cane (Reg. (EU) 2021/1807 2024/2612).

According to available data, the following specific mitigation measures are recommended before planting succeeding crops:

- a 30-day PBI is supported for leafy and brassica vegetables, oilseeds and for all other food and feed commodities except sugar canes;
- sugar canes not be planted for 125 days after application of clopyralid.

No additional data are required.

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

In accordance with SANTE/11956/2016 rev. 9, crops included in this submission are considered melliferous, application of clopyralid is close to flowering and the active substance is systemic, therefore data are required. A honey study was completed in 2021 and will be submitted with a new MRL application in 2022.

A study was conducted to determine residue levels of clopyralid in nectar, pollen, plants, and honey after one application of a clopyralid-containing formulation, GF-1966, under semi-field conditions to winter oilseed rape plants (Study ID DAS 200098).

The study was conducted in 2020 as six separate field trials, with four trials located in N-EU (Northern and Southern Germany, Romania, and The Netherlands) and two trials located in S-EU (Southern France and Spain). The study consisted of two treatment groups per trial: the test item group T (two replicates - Ta and Tb) and an untreated control C (one replicate). There was one application of GF-1966 (720 g a.e./kg SG) in the test item treatment group at a target rate of 125 g a.e./ha in water spray volume of 200-300L/ha at

BBCH 55 for trials -02, -03, -05, -06 and -07 and BBCH 57 for trial -04. Winter oilseed rape plants were collected six times after application (up to 25 days after application [DAA]), pollen and forager bees for nectar preparation were collected six times after application (up to 34 DAA) and honey was collected once in all trials (17-49 DAA) for subsequent residue analysis. The maximum period of frozen storage from the day of sampling until extraction was 313 days for matrices honey, pollen, nectar, and plant. The study supports a proposed MRL of 0.15 mg/kg for clopyralid in honey and was submitted to EMS Finland in 2022 (EFSA-Q-2022-00622). A summary of the study is presented in Appendix 2.

zRMS comments:

According to SANTE/11956/2016 rev. 9, rapeseed and some other oilseeds are considered as melliferous crops. Although clopyralid is proposed to be used before the flowering stage (BBCH 30-51), it is a systemic active substance and its effect on the residue level in pollen and bee products should be investigated. Information about residue levels in pollen and bee products should be provided by the applicant in the framework of this submission.

Applicant has access to new honey study (Study Code: S20-00871) with the LoA.

The data on honey has been submitted to EMS - Finland in the framework of an MRL application. ~~However no Reasoned Opinion is currently available:~~ “Modification of the existing maximum residue level for clopyralid in honey” (EFSA Journal. 2024;22:e8546; DOI: 10.2903/j.efsa.2024.8546).

The study was conducted as six field trials in Germany, Romania, The Netherlands, Southern France and Spain. The rate used in the study was approximately 1N of the proposed use rate for oilseeds (foliar application 1 × 125 g a.s./ha; BBCH 30–51). The residues in honey were 3x <0.01, 0.0179, 0.0274, 0.0695 mg/kg.

HR from six trials equals 0.0695 mg/kg and this value is from the SEU trial. ~~According to the Reg. (EU) 2021/1807 current MRL for clopyralid in honey is 0.05* mg/kg and it is potentially exceeded.~~

It should be noted that the Technical guidelines for determining the magnitude of pesticide residues in honey and setting Maximum Residue Levels in honey (SANTE/11956/2016 rev. 9) do not divide the zones, and therefore all residual trials for honey should be taken into account.

EFSA, 2024: Based on the six GAP-compliant trials from the GLP study, an MRL of 0.15 mg/kg can be proposed for clopyralid in honey using the OECD MRL calculator. It should be noted that currently, MRLs set for honey are not applicable to other apicultural products following Commission Regulation (EU) 2018/62.

The MRL for clopyralid in honey has been changed and now is 0.15 mg/kg (Commission Regulation (EU) 2024/2612), and according to the provided study on magnitudes of residues in honey, the MRL is not potentially exceeded. **The uses on melliferous target crops (oilseed rape, mustard and linseed) can be authorized.**

~~In our opinion, until the new MRL has been set for honey, uses on oilseed rape, mustard and linseed cannot be authorized.~~

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

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

7.2.8.1 Input values for the consumer risk assessment

The input values in the following table were used to estimate consumer risk using the EFSA PRIMo Rev 3.1. The chronic dietary risk assessment is based on current MRLs from Reg ~~2018/1514~~ 2021/1807 2024/2612, apart from indicated animal products, wheat, and oat which considered MRLs adopted in the Article 6 MRL Evaluation Report (EFSA Journal 2021; 19(1):6389).

The acute dietary assessments are performed based on MRL inputs only for the commodities for which GAPs are notified in the clopyralid Article 43 process.

Table 7.2-19: Input values for the consumer risk assessment

Commodity	Chronic-risk assessment		Acute-risk assessment	
	Input-value (mg/kg)	Comment	Input-value (mg/kg)	Comment
Clopyralid common moiety (sum of clopyralid, its salts and conjugates, expressed as clopyralid)				
Onion, Garlic, Shallot	0.5	EU MRL/Reg- 2021/1807	0.5	EU MRL/Reg- 2021/1807
Spring onions	0.7		0.7	
Broccoli	1.5		STMR (NEU) flowering brassica, section 7.2.3.1 of this submission	
	0.27			
Cauliflower	3.0	EU MRL/Reg- 2021/1807	0.85	
	0.27	STMR (NEU) flowering brassica, section 7.2.3.1 of this submission		
Other Flowering brassica	0.5	EU MRL/Reg- 2021/1807	0.85	
	0.27	STMR (NEU) flowering brassica, section 7.2.3.1 of this submission		
Head-cabbage	3.0	EU MRL/Reg- 2021/1807	0.91	HR (NEU) flowering brassica, section 7.2.3.1 of this submission
	0.21	STMR (NEU) flowering brassica, section 7.2.3.1 of this submission		
Brussel sprouts	0.5	EU MRL/Reg- 2021/1807	0.5	EU MRL/Reg- 2021/1807
Spinach, Chard/beet leaves	1.0		1.0	
Oilseed rape, Mustard seed, Borage seed	0.5		0.5	
Linseed	20		20	
Barley, Maize	2.0		2.0	
Rye	5.0		5.0	
Oat, Wheat	3.0	EU MRL/Reg- 2021/1807	3.0	EU MRL/Reg- 2021/1807
Sugar beet	1.0	EU MRL/Reg- 2021/1807	1.0	EU MRL/Reg- 2021/1807
Red beet	1.0		1.0	
Swede	1.5		1.5	
Turnips	1.5		1.5	
All other plant commodities	EU MRL/Reg- 2021/1807		Not relevant for an acute risk assessment	
Clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid)				
Swine, muscle	0.05*	EU MRL/Reg- 2021/1807	0.05*	EU MRL/Reg- 2021/1807
Swine, fat	0.05		0.05	

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Swine, liver	0.05		0.05	
Swine, kidney	0.6		0.6	
Swine, edible offals	0.05		0.05	
Swine, Others	0.05		0.05	
Bovine, muscle	0.05		0.05	
Bovine, fat	0.15		0.15	
Bovine, liver	0.15		0.15	
Bovine, kidney	1.5		1.5	
Bovine, edible offals	0.05 [±]		0.05 [±]	
Bovine, Others	0.05 [±]		0.05 [±]	
Sheep, muscle	0.05		0.05	
Sheep, fat	0.2		0.2	
Sheep, liver	0.2		0.2	
Sheep, kidney	2.0		2.0	
Sheep, edible offals	0.05 [±]		0.05 [±]	
Sheep, Others	0.05 [±]		0.05 [±]	
Goat, muscle	0.05		0.05	
Goat, fat	0.2		0.2	
Goat, liver	0.2		0.2	
Goat, kidney	2.0		2.0	
Goat, edible offals	0.05 [±]		0.05 [±]	
Goat, Others	0.05 [±]		0.05 [±]	
Equine	0.05 [±]		0.05 [±]	
Poultry, muscle	0.05 [±]		0.05 [±]	
Poultry, fat	0.05 [±]		0.05 [±]	
Poultry, liver	0.05		0.05	
Poultry, kidney	0.05 [±]		0.05 [±]	
Poultry, edible offals	0.05 [±]		0.05 [±]	
Poultry, others	0.05 [±]		0.05 [±]	
Milk	0.05 CF of 1.3 (EFSA, 2018a)		0.05 CF of 1.3 (EFSA, 2018a)	
Eggs	0.05 [±]		0.05 [±]	

Commodity	Chronic risk assessment		Acute risk assessment		
	Input value (mg/kg)	Comment	Commodity	Input value (mg/kg)	Comment
Risk assessment residue definition: Clopyralid, including its salts and conjugates, expressed as clopyralid					
All commodities	MRL	Reg (EU) 2021/1807 2024/2612	Sugar beet	1	Acute risk assessment was undertaken only with regard to the crops under consideration. MRL Reg (EU) 2021/1807 2024/2612
			Red beet	1	
			Turnip	1.5	
			Swedes	1.5	
			Oilseed rape	0.5	
			Mustard seeds	0.5	
			Linseed	20	
			Onion	0.5	
Milk	0.05 x CF (1.3)		Milk	0.05 x CF (1.3)	
Animal commodities	MRL		Animal commodities	MRL	

7.2.8.2 Conclusion on consumer risk assessment

The highest Theoretical Maximum Daily Intake (TMDI) is 42% of the ADI (based on NL toddler). The acute risk assessment was undertaken only for the crops under consideration. Children have the highest International Estimated Short-Term Intake (IESTI) for unprocessed commodities at 34% of the ARfD (NL toddler) based on the consumption of beetroots, and for processed commodities at 65% of the ARfD (NL child) from the consumption of sugar beets (root)/sugar.

Extensive calculation sheets are presented in Appendix 3.

Table 7.2-20: Consumer risk assessment


TMDI (% ADI) according to EFSA PRIMo	42 43% (based on NL toddlers)
IEDI (% ADI) according to EFSA PRIMo	Not necessary
IESTI (% ARfD) according to EFSA PRIMo*	MRL inputs only for the commodities for which GAPs are notified in the Article 43 submission: Unprocessed Commodities 34% — Beetroots (based on NL toddlers) 25% — Wheat (based on UK 4-6 year olds) 15% — Wheat (based on UK 15-18 yr) 14% — Beetroots (based on NL general) 13% — Linseeds (based on DE child) 11% — Chards/beet leaves (based on NL general) 11% — Rye (based on UK infant)

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

** National risk assessment calculators

zRMS comments:

A new table 7.2-19 has been created by the zRMS-PL and is highlighted in grey. As no refinement of the chronic risk assessment is required ($\text{TMDI} < 100\% \text{ ADI}$), STMR values do not need to be presented.

 <p>European Food Safety Authority</p> <p>EFSA PRIMO revision 3.1; 2019/03/19</p>		<div> <div>clopyralid</div> <div> <div>LOOs (mg/kg) range from:</div> <div>to:</div> </div> </div>				<div>Input values</div>									
		<div> <div>Toxicological reference values</div> <div> <div>ADI (mg/kg bw/day):</div> <div>0,15</div> <div>ARID (mg/kg bw):</div> <div>0,17</div> </div> </div>				<div> <div>Details - chronic risk assessment</div> <div>Supplementary results - chronic risk assessment</div> </div>									
		<div> <div>Source of ADI:</div> <div>EFSA</div> <div>Source of ARID:</div> <div>EFSA</div> </div>				<div> <div>Details - acute risk assessment/children</div> <div>Details - acute risk assessment/adults</div> </div>									
		<div> <div>Year of evaluation:</div> <div>2018</div> <div>Year of evaluation:</div> <div>2018</div> </div>													
Comments:															
Normal mode															
Chronic risk assessment: JMPR methodology (EDI/TMDI)															
TMDI/NEDIED calculation (based on average food consumption)	Calculated exposure (% of ADI)	MS Diet	Exposure to MS diet (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commodities (in % of ADI)	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities (in % of ADI)	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities (in % of ADI)	Exposure resulting from MRLs set at the LOQ (in % of ADI)	commodities under assessment (in % of ADI)				
	43%	NL toddler	64,28	9%	Maize/corn	8%	Wheat	4%	Apples						
	33%	DK child	50,22	18%	Rye	9%	Wheat	0,8%	Potatoes						
	27%	GEMS/Food G06	40,64	14%	Wheat	2%	Rice	2%	Maize/corn						
	25%	NL child	37,66	8%	Wheat	6%	Sugar beet roots	2%	Apples						
	26%	DE child	37,25	8%	Wheat	4%	Apples	3%	Rye						
	21%	RO general	31,30	10%	Wheat	3%	Head cabbages	1%	Maize/corn						
	21%	GEMS/Food G15	30,89	9%	Wheat	2%	Head cabbages	1%	Potatoes						
	21%	GEMS/Food G08	30,82	8%	Wheat	2%	Rye	1%	Potatoes						
	20%	FR child 3 15 y	30,12	9%	Wheat	2%	Sugar beet roots	1%	Oranges						
	20%	GEMS/Food G10	29,37	8%	Wheat	2%	Rice	1%	Soybeans						
	18%	IT toddler	27,67	13%	Wheat	2%	Other cereals	0,5%	Tomatoes						
	18%	IE adult	27,44	5%	Wheat	2%	Sweet potatoes	2%	Linseeds						
	18%	GEMS/Food G07	27,36	8%	Wheat	1%	Potatoes	0,8%	Barley						
	17%	GEMS/Food G11	25,78	7%	Wheat	1%	Potatoes	1%	Soybeans						
	17%	UK toddler	25,18	8%	Wheat	2%	Sugar beet roots	1%	Potatoes						
	16%	FR toddler 2 3 y	24,00	6%	Wheat	2%	Sugar beet roots	1%	Milk: Cattle						
	16%	UK infant	23,47	5%	Wheat	2%	Milk: Cattle	1%	Maize/corn						
	16%	SE general	23,26	6%	Wheat	1%	Potatoes	1%	Head cabbages						
	15%	PT general	23,04	8%	Wheat	2%	Potatoes	1%	Rice						
	15%	ES child	22,68	9%	Wheat	0,7%	Oranges	0,6%	Rice						
	15%	DE women 14-50 y	22,28	4%	Wheat	3%	Sugar beet roots	2%	Rye						
	15%	DE general	21,93	4%	Wheat	3%	Sugar beet roots	2%	Rye						
	12%	IT adult	18,12	8%	Wheat	0,9%	Other cereals	0,4%	Tomatoes						
	12%	NL general	18,02	4%	Wheat	2%	Sugar beet roots	0,8%	Potatoes						
	12%	FI 3 y	17,37	2%	Wheat	2%	Rye	2%	Potatoes						
	10%	LT adult	14,97	4%	Rye	2%	Wheat	1%	Potatoes						
	9%	ES adult	14,07	5%	Wheat	0,7%	Barley	0,4%	Oranges						
	9%	FI 6 y	13,78	2%	Rye	2%	Wheat	1%	Potatoes						
	9%	FR adult	13,65	4%	Wheat	0,8%	Wine grapes	0,5%	Sugar beet roots						
	8%	UK vegetarian	12,55	4%	Wheat	0,5%	Rice	0,5%	Potatoes						
	8%	FI adult	11,39	2%	Rye	2%	Coffee beans	0,6%	Wheat						
	7%	FR infant	10,69	2%	Wheat	0,9%	Sugar beet roots	0,7%	Milk: Cattle						
	7%	DK adult	10,51	2%	Wheat	2%	Rye	0,4%	Potatoes						

The calculation of the TMDI using EFSA model (PRIMo ver. 3.1) and MRLs values for all plant and animals commodities according to Regulation (EU) ~~2021/1807~~ **2024/2612** led to a utilisation of the ADI of 43% for NL toddler diet with the highest value. For this diet, the highest contributor is Maize/corn with 9% of the ADI.

Additionally, the evaluator performed an acute consumer risk assessment using MRLs for intended uses and MRLs for animal commodities and using a value of ARfD of 0.17 mg/kg bw.

Acute risk assessment /children				Acute risk assessment / adults / general population				Acute risk assessment /children				Acute risk assessment / adults / general population											
Details - acute risk assessment /children				Details - acute risk assessment/adults				Hide IESTI new calculations				Show IESTI new calculations											
The acute risk assessment is based on the ARfD. The calculation is based on the target portion of the most critical consumer group.								IESTI new calculations: The calculation is performed with the MRL and the peeling/processing factor (PF), taking into account the residue in the edible portion and/or the conversion factor for the residue definition (CF). For case 2a, 2b and 3 calculations a variability factor of 3 is used. Since this methodology is not based on internationally agreed principles, the results are considered as indicative only. Since this methodology is not based on internationally agreed principles, the results are considered as indicative only.															
Show results for all crops																							
Unprocessed commodities	Results for children No. of commodities for which ARfD/ADI is exceeded (IESTI):				Results for adults No. of commodities for which ARfD/ADI is exceeded (IESTI):				IESTI new Results for children No. of commodities for which ARfD/ADI is exceeded (IESTI new):				IESTI new Results for adults No. of commodities for which ARfD/ADI is exceeded (IESTI new):										
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	IESTI				IESTI				IESTI new				IESTI new										
	Highest % of ARfD/ADI	Commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)							
	46%	Swedes/rutabagas	1.5 / 1.5	78	30%	Swedes/rutabagas	1.5 / 1.5	51	27%	Swedes/rutabagas	1.5 / 1.5	47	18%	Swedes/rutabagas	1.5 / 1.5	31							
	34%	Beetroots	1 / 1	57	14%	Beetroots	1 / 1	23	14%	Beetroots	1 / 1	24	6%	Beetroots	1 / 1	10							
	32%	Turnips	1.5 / 1.5	54	10%	Turnips	1.5 / 1.5	17	14%	Turnips	1.5 / 1.5	23	6%	Linseeds	20 / 20	9.6							
	13%	Linseeds	20 / 20	21	6%	Linseeds	20 / 20	9.6	13%	Linseeds	20 / 20	21	5%	Turnips	1.5 / 1.5	8.2							
	7%	Onions	0.5 / 0.5	7.4	4%	Onions	0.5 / 0.5	7.4	5%	Milk: Cattle	0.05 / 0.07	8.1	2%	Onions	0.5 / 0.5	3.2							
	5%	Milk: Cattle	0.05 / 0.07	8.1	2%	Bovine: Kidney	1.5 / 1.5	3.2	3%	Bovine: Kidney	1.5 / 1.5	5.6	2%	Bovine: Kidney	1.5 / 1.5	3.2							
3%	Bovine: Kidney	1.5 / 1.5	5.6	1%	Milk: Cattle	0.05 / 0.07	2.5	3%	Onions	0.5 / 0.5	4.9	1%	Milk: Cattle	0.05 / 0.07	2.5								
0.9%	Milk: Goat	0.05 / 0.07	1.6	0.8%	Swine: Kidney	0.6 / 0.6	1.3	0.9%	Milk: Goat	0.05 / 0.07	1.6	0.8%	Swine: Kidney	0.6 / 0.6	1.3								
0.7%	Bovine: Liver	0.15 / 0.15	1.2	0.7%	Milk: Goat	0.05 / 0.07	1.2	0.7%	Bovine: Liver	0.15 / 0.15	1.2	0.7%	Milk: Goat	0.05 / 0.07	1.2								
0.5%	Poultry: Musclemeat	0.05 / 0.05	0.85	0.6%	Milk: Sheep	0.05 / 0.07	0.98	0.5%	Poultry: Musclemeat	0.05 / 0.05	0.85	0.6%	Milk: Sheep	0.05 / 0.07	0.98								
0.4%	Swine: Kidney	0.6 / 0.6	0.76	0.4%	Bovine: Liver	0.15 / 0.15	0.60	0.4%	Swine: Kidney	0.6 / 0.6	0.76	0.4%	Bovine: Liver	0.15 / 0.15	0.60								
0.4%	Rapeseeds/oils	0.5 / 0.5	0.59	0.3%	Poultry: Muscde	0.05 / 0.05	0.59	0.4%	Rapeseeds/oils	0.5 / 0.5	0.59	0.3%	Poultry: Muscde	0.05 / 0.05	0.59								
0.4%	Eggs: Chicken	0.05 / 0.05	0.62	0.3%	Sheep: Liver	0.2 / 0.2	0.56	0.4%	Eggs: Chicken	0.05 / 0.05	0.62	0.3%	Sheep: Liver	0.2 / 0.2	0.56								
0.3%	Swine: Musclemeat	0.05 / 0.05	0.61	0.3%	Bovine: Muscde	0.08 / 0.08	0.46	0.4%	Swine: Musclemeat	0.05 / 0.05	0.61	0.3%	Bovine: Muscle	0.08 / 0.08	0.46								
0.3%	Bovine: Musclemeat	0.08 / 0.08	0.58	0.2%	Sheep: Musclemeat	0.08 / 0.08	0.38	0.3%	Bovine: Musclemeat	0.08 / 0.08	0.58	0.2%	Sheep: Musclemeat	0.08 / 0.08	0.38								
Expand/collapse list																							
Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)								Total number of commodities found exceeding the ARfD/ADI in children and adult diets (IESTI new calculation)															
Processed commodities	Results for children No. of processed commodities for which ARfD/ADI is exceeded (IESTI):				Results for adults No. of processed commodities for which ARfD/ADI is exceeded (IESTI):				Results for children No. of processed commodities for which ARfD/ADI is exceeded (IESTI new):				Results for adults No. of processed commodities for which ARfD/ADI is exceeded (IESTI new):										
	---				---				---				---										
	IESTI				IESTI				IESTI new				IESTI new										
	Highest % of ARfD/ADI	Processed commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Processed commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Processed commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Processed commodities	MRL /input for RA (mg/kg)	Exposure (µg/kg bw)							
	65%	Sugar beets (root) / sugar	1 / 12	110	26%	Sugar beets (root) / sugar	1 / 12	44	65%	Sugar beets (root) / sugar	1 / 12	110	26%	Sugar beets (root) / sugar	1 / 12	44							
	45%	Turnips / boiled	1.5 / 1.5	76	23%	Beetroots / boiled	1 / 1	39	19%	Turnips / boiled	1.5 / 1.5	33	10%	Beetroots / boiled	1 / 1	17							
	26%	Beetroots / boiled	1 / 1	44	17%	Turnips / boiled	1.5 / 1.5	29	11%	Beetroots / boiled	1 / 1	19	8%	Turnips / boiled	1.5 / 1.5	14							
	0.2%	Rapeseeds / oils	0.5 / 1	0.29	3%	Onions / boiled	0.5 / 0.5	4.7	0.2%	Rapeseeds / oils	0.5 / 1	0.29	2%	Onions / boiled	0.5 / 0.5	3.6							
	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI							
	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI							
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI	#LICZBAI								
Expand/collapse list																							
Conclusion: No exceedance of the toxicological reference value was identified for any unprocessed commodity. A short term intake of residues of Please insert here the MRLs of COM database (use 'base values' function) is unlikely to present a public health risk. For processed commodities, no exceedance of the ARfD/ADI was identified.																							

The highest International Estimated Short-Term Intake (IESTI) is at 46% and 30% of the ARfD for the consumption of swedes by children and by adults respectively and for processed commodities at 65% **and 26%** of the ARfD from the consumption of sugar beets (root)/sugar **by children and by adults, respectively**.
The proposed uses of clopyralid in the product HCV08 do not represent unacceptable acute and chronic risks for the consumer.

7.3 Combined exposure and risk assessment

Not relevant. The product contains only one active substance.

7.4 References

EFSA (European Food Safety Authority), 2005. Conclusion regarding the peer review of the pesticide risk assessment of the active substance clopyralid. Finalised: 14 December 2005. EFSA Scientific Report (2005) 50, 1-65. Available online: www.efsa.europa.eu/efsajournal

EFSA (European Food Safety Authority), 2011. Modification of the existing MRLs for clopyralid in various commodities. EFSA Journal 2011;9(10):2418. [doi:10.2903/j.efsa.2011.2418](https://doi.org/10.2903/j.efsa.2011.2418). Available online: www.efsa.europa.eu/efsajournal

EFSA (European Food Safety Authority), 2018a. Arena M, Auteri D, Barmaz S, Brancato A, Brocca D, Bura L, Carrasco Cabrera L, Chiusolo A, Civitella C, Court Marques D, Crivellente F, Ctverackova L, De Lentdecker C, Egsmose M, Erdos Z, Fait G, Ferreira L, Greco L, Ippolito A, Istace F, Jarrah S, Kardassi D, Leuschner R, Lostia A, Lythgo C, Magrans JO, Medina P, Mineo D, Miron I, Molnar T, Padovani L, Parra Morte JM, Pedersen R, Reich H, Sacchi A, Santos M, Serafimova R, Sharp R, Stanek A, Streissl F, Sturma J, Szentes C, Tarazona J, Terron A, Theobald A, Vagenende B, Van Dijk J and Villamar-Bouza L, 2018. Conclusion on the peer review of the pesticide risk assessment of the active substance clopyralid. EFSA Journal 2018;16(8):5389, 21 pp. <https://doi.org/10.2903/j.efsa.2018.5389>

EFSA (European Food Safety Authority), 2018b. Brancato A, Brocca D, De Lentdecker C, Erdos Z, Ferreira L, Greco L, Jarrah S, Kardassi D, Leuschner R, Lythgo C, Medina P, Miron I, Molnar T, Nougadere A, Pedersen R, Reich H, Sacchi A, Santos M, Stanek A, Sturma J, Tarazona J, Theobald A, Vagenende B, Verani A and Villamar-Bouza L, 2018. Reasoned Opinion on the modification of the existing maximum residue levels for clopyralid in spring/green/Welsh onions and leeks. EFSA Journal 2018;16 (1):5149, 19 pp. <https://doi.org/10.2903/j.efsa.2018.5149>

European Food Safety Authority (EFSA), 2021. Anastassiadou M, Bernasconi G, Brancato A, Carrasco Cabrera L, Ferreira L, Greco L, Jarrah S, Kazocina A, Leuschner R, Magrans JO, Miron I, Nave S, Pedersen R, Reich H, Rojas A, Sacchi A, Santos M, Scarlato AP, Theobald A, Vagenende B and Verani A, 2021. Reasoned Opinion on the modification of the existing maximum residue levels for clopyralid in various commodities. EFSA Journal 2021;19(1):6389, 46 pp. <https://doi.org/10.2903/j.efsa.2018.5149>

Finland, 2005. Revised Draft Assessment Report (DAR) on clopyralid prepared by the rapporteur Member State Finland under Directive 91/414/EEC, September 2005.

Finland, 2008. Evaluation Report on the modification of MRLs for clopyralid in brassica vegetables, linseed, swedes and turnips prepared by the evaluating Member State Finland under Article 8 of Regulation (EC) No 396/2005, 15 December 2008.

Finland, 2018a. Revised Renewal Assessment Report (RAR) on clopyralid prepared by the rapporteur Member State Finland in the framework of Commission Implementing Regulation (EU) 844/2012, May 2018. Available online: <http://www.efsa.europa.eu>

Finland, 2018b. Evaluation Report, prepared under Article 8 of Regulation (EC) No 396/2005. MRL application on the setting of MRL(s) in various commodities. 31 May 2018. Ongoing EFSA evaluation. EFSA-Q-2018-00576. Updated in January 2019 and July 2020.

EFSA (European Food Safety Authority), 2024. Giulia Bellisai, Giovanni Bernasconi, Luis Carrasco Cabrera, Irene Castellan, Monica del Aguila, Lucien Ferreira, German Giner Santonja, Luna Greco, Samira Jarrah, Renata Leuschner, Andrea Mioč, Stefanie Nave, Ragnor Pedersen, Hermine Reich, Silvia Ruocco, Alessia Pia Scarlato, Marta Szot, Anne Theobald, Manuela Tiramani, Alessia Verani. Modification of the existing maximum residue level for clopyralid in honey. EFSA Journal. 2024;22:e8546; <https://doi.org/10.2903/j.efsa.2024.8546>

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 (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner	Previously evaluated
KCA 6.1	Skaggs, C.S., Penning, B.N.	2021	Storage Stability of Clopyralid for One Year in Dried Beans Corteva Report No. 191728 Study No. SGS-19-01-08 SGS North America, Inc GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.1	Teasdale, R.	1996	Frozen Storage Stability of Clopyralid Residues in Strawberries Corteva Report No. GHE-P-4832 Study No. CEMS-235 CEM Analytical Services Ltd. GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.1	Forbes, T., Cross, M	2021	Frozen Storage Stability of Clopyralid in Pollinator Matrices Corteva Report No. 180869 Study No. CEMS-8756 CEM Analytical Services (CEMAS) GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.2.1/01	Morton Lloyd, G.	2020	The Metabolism of [14C]-Clopyralid in Wheat DAS Study No. 191200 Study No. 229882 Charles River Laboratories GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.2.1/02	MacKenzie, A.	2021	The Metabolism of [14C]-Clopyralid in Spring Oilseed Rape DAS Study No. 200928 Study No. 231101 Charles River Laboratories GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)

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	Previously evaluated
KCA 6.3.1/01	Delmotte, R.	2017	Magnitude of the Residues of Halauxifen-methyl and Clopyralid in Oilseed rape (RAC Whole Plant, Seed and Straw), following One Application of GF-3488, Northern Europe - 2015 DAS Report No. 150534 Study No. RDE-15-20400 Staphyt GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.3.2/01	Pirie, D.	2021	Magnitude and Decline of Residues of Clopyralid in Sugar Beet Following Applications of GF-1966 in Northern Europe and the UK, Initiated in 2020. DAS Study No. 200809 Study No. 684083 Charles River Laboratories Edinburgh Ltd. GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.3.5/01	Devine, H.C.	2004	Residues of Clopyralid in Onions at Harvest and at Intervals Following Two Application of Lontrel 100 (EF-1136), UK - 2003 Study No. CEMS-2030 DAS Report No. GHE-P-10805 CEM Analytical Services Ltd (CEMAS) GLP Unpublished	N	Corteva Agriscience	N
KCA 6.3.5/02	Devine, H.C.	2005	Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF-1136), Northern Europe - 2004 Study No. CEMS-2346 DAS Report No. GHE-P-11080 CEM Analytical Services Ltd (CEMAS) GLP Unpublished	N	Corteva Agriscience	N
KCA 6.3.5/03	Devine, H.C.	2006	Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF-1136), Northern Europe-2005 Study No. CEMS-2696 DAS Report No. GHE-P-11272 CEM Analytical Services Ltd (CEMAS) GLP Unpublished	N	Corteva Agriscience	N

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	Previously evaluated
KCA 6.3.3/04	Rawle, N. W.	2012	Residues of Clopyralid in Bulb Onions following Two Applications of EF-1136 - Northern Europe – 2011 Report No. GHE-P-12680 GLP Unpublished	N	Corteva Agriscience	N
KCA 6.3.5/05	Butler, R.E. and Reynens, P.	1998	Determination of Residues of Clopyralid in Onions following a single application of Lontrel 100 (EF-1136), Belguim, 1997; Study No. R97-030 DAS Report No. GHE-P-7289 GLP Unpublished	N	Corteva Agriscience	N
KCA 6.5.3/01	Phillips, A.M.	1994	Determination of residues of clopyralid in sugar beet processed fractions DAS Report No. GH-C 3305 North American Environmental Chemistry Laboratory GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.5.3/02	Devine, H.C.	2020	Residues of Clopyralid in Sugar Beet and Process Fractions Following Multiple Applications of GF-1966 – Northern Europe – 2019 DAS Report No. 181493 Study No. CEMS-8908 CEM Analytical Services Ltd (CEMAS) GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.6.2/01	Devine, C.	2021	Determination of Residues of Clopyralid after One Application of GF-1966 (EC Formulation) on Bare Soil in Rotational Crops at 3 Sites in Northern Europe and 3 Sites in Southern Europe 2019-2020 Corteva Study No. 190557 Study No. CEMS-9009 CEM Analytical Services Ltd (CEMAS) GLP Unpublished	N	Corteva Agriscience	Yes, in RR, Part B7 for EF-243/ Lontrel 300 (02.2023)
KCA 6.7	Appeltauer, A.	2021	Determination of Residues of Clopyralid in Nectar, Pollen, Plants and Honey of Winter Oilseed Rape after One Application of GF-1966 in a Semi-Field Residue Study in Germany, Romania, The Netherlands, Southern France and Spain in 2020 Eurofins Agrosience Services Ecotox GmbH Study Code: S20-00871; Sponsor Study ID.: DAS 200098 GLP/GEP (Y/N): Yes	N	Corteva Agriscience	N

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	Previously evaluated
			Published (Y/N): No			

List of data submitted or referred to by the applicant and relied on, but already evaluated at EU peer review

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.1 (CA 6.1.1/1)	Allen, L.	2013	Frozen Storage Stability of Residues of Clopyralid in Crop Matrices DAS Study No. 120939 CEM Analytical Services (CEMAS), North Ascot, Berkshire, UK GLP Unpublished	N	Corteva Agriscience
KCA 6.1 (CA 6.1.1/2)	Foster, D.R., Blakeslee, B.A., Rutherford, B.S.	1996	Frozen Storage Stability of Clopyralid, 2,4-D in Corn Grain, Straw and Fodder DAS Study No. RES93050.01 DowElanco, Indianapolis, Indiana, US GLP Unpublished	N	Corteva Agriscience
KCA 6.1 (CA 6.1.1/3)	Clements, B., Bolton, A.	1996	Determination of the Stability of Clopyralid Residues in Pasture under Frozen Storage Conditions DAS Study No. GHE-P-5350 CEM Analytical Services (CEMAS), North Ascot, Berkshire, UK GLP Unpublished	N	Corteva Agriscience
KCA 6.1 (CA 6.1.2/1)	xxx	2015	Frozen Storage Stability of Clopyralid in Bovine Fat Study No. 120602 xxx GLP Unpublished	Y	Corteva Agriscience
KCA 6.1 (CA 6.1.2/2)	xxx	2004	Frozen Storage Stability of Clopyralid in Beef Muscle, Liver, Kidney, Milk and Chicken Egg Study No. 020120.01 xxx GLP Unpublished	Y	Corteva Agriscience

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.2.1 (CA 6.2.1/1)	Chapleo, S.; Caley, C. Y.	2002, revised 2019 †	The Metabolism of [14C]-Clopyralid in Sugar Beet DAS Study No. GHE-P-9939 Inveresk Research International, Tranent, East Lothian, United Kingdom GLP Unpublished	N	Corteva Agriscience
KCA 6.2.1 (CA 6.2.1/2)	Guo, C.	1996	Metabolism of 14C -Clopyralid in Cabbage DAS Study No. RES95095 DAS Report No. GH-C-4289 ABC Laboratories Inc, Columbia, Missouri, USA GLP Unpublished	N	Corteva Agriscience
KCA 6.2.1 (CA 6.2.1/3)	Chapleo, S., Caley, C. Y., White, D. E.	2002, revised 2019†	The Metabolism of (14C)-Clopyralid in Oilseed Rape DAS Study No. GHE-P 9938 Inveresk Research International, Tranent, East Lothian, UK GLP Unpublished	N	Corteva Agriscience
KCA 6.2.1	Bauriedel, WR, Miller, JH	1981	A Field Metabolism Study of 14C-Labeled 3,6-Dichloropicolinic acid Applied to Pasture Grass DAS Study ID GH-C 1424 Dow Chemical USA Non-GLP (pre-GLP) Unpublished	N	Corteva Agriscience
KCA 6.2.1 (CA 6.2.1/4)	Gourlay, V.	2015	Plant uptake of 14C -labelled clopyralid in wheat and oilseed rape under greenhouse conditions DAS Study No. 150297 RLP AgroScience GmbH, 67435 Neustadt a.d. Weinstraße, Germany GLP Unpublished	N	Corteva Agriscience
KCA 6.2.2 (CA 6.2.2/1)	xxx	2014	A Nature of the Residue Study in the Laying Hen with [14C]-Clopyralid Study No. 130906 xxx GLP Unpublished	Y	Corteva Agriscience

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.2.3 (CA 6.2.3/1)	xxx	2015	A Nature of the Residue Study in the Ruminant with [14C]Clopyralid Study No. 130202 xxx GLP Unpublished	Y	Corteva Agriscience
KCA 6.3.3/01	Devine, H. C.	2004	Residues of Clopyralid in Onions at Harvest and at Intervals Following Two Application of Lontrel 100 (EF 1136), UK –2003; Report No. GHE P 10805 GLP Unpublished	N	Corteva Agriscience
KCA 6.3.3/02	Devine, H. C.	2005	Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF 1136), Northern Europe –2004 Report No. GHE P 11080 GLP Unpublished	N	Corteva Agriscience
KCA 6.3.3/03	Devine, H. C.	2006	Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF 1136), Northern Europe –2005; Report No. GHE P 11272 GLP Unpublished	N	Corteva Agriscience
KCA 6.4.1 (CA 6.4.1/1)	xxx	1974	Dowco 290 and 2,4-D Chicken Feeding Study Study No. TA-517 xxx Non-GLP Unpublished	Y	Corteva Agriscience
KCA 6.4.1 (CA 6.4.1/2)	xxx	1975	Residues of Dowco 290 (3,6-dichloropicolinic acid) in Tissues of Chickens Fed the Herbicide Study No. GH-C 819 xxx Non-GLP Unpublished	Y	Corteva Agriscience

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.4.1 (CA 6.4.1/3)	xxx	2015	Summary of Clopyralid Livestock Feeding Study: Magnitude of Residue in Eggs, Muscle, Liver and Fat of Laying Hens Study No. 150031 xxx GLP Unpublished	Y	Corteva Agriscience
KCA 6.4.2 (CA 6.4.2/1)	xxx	1974	Milk Residue Study with Dairy Cows Fed Lontrel Herbicide, Nellite Nematocide and 2,4-D Herbicide: Animal Care, Sampling and Production Records Study No. GH-A 579 xxx Unpublished	Y	Corteva Agriscience
KCA 6.4.2 (CA 6.4.2/2)	xxx	1974	Residues of Dowco 290 (3,6-dichloropicolinic acid) in Milk and Cream from Cows Fed the Herbicide Study No. GH-C 745 xxx Non-GLP Unpublished	Y	Corteva Agriscience
KCA 6.4.2 (CA 6.4.2/3)	xxx	1975	Residues of Dowco 290 (3,6-dichloropicolinic acid) in Bovine Tissues from Calves Fed the Herbicide Study No. GH-C 811 xxx Non-GLP Unpublished	Y	Corteva Agriscience
KCA 6.4.2 (CA 6.4.2/4)	xxx	2015	Summary of Clopyralid Livestock Feeding Study: Magnitude of Residue in Milk, Muscle, Liver, Kidney and Fat of Lactating Dairy Cattle Study No. 150030 xxx GLP Unpublished	Y	Corteva Agriscience
KCA 6.4.3 (CA 6.4.3/1)	xxx	1975	Residues of Dowco 290 (3,6-dichloropicolinic acid) in Tissues of Swine Fed the Herbicide Study No. GH-C 874 xxx Non-GLP Unpublished	Y	Corteva Agriscience

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.5.1 (CA 6.5.1/1)	Adusumilli, H.	2014	Processing Study to Determine the Nature of Residues of 14C -Clopyralid Following the Industrial or Household Preparation DAS Study No. 140574 Dow AgroSciences LLC, Indianapolis, Indiana, USA GLP Unpublished	N	Corteva Agriscience
KCA 6.5.3 (IIA 6.5.2/06)	Day, SR	1987	Clopyralid Residues in Rape Plant, Straw, Seed and Oil, Following Application of LONTREL 100, Germany 1985 and 1986 Study No. GHE-P-1740 Non-GLP Unpublished	N	Corteva Agriscience
KCA 6.5.3 (IIA 6.5.2/01)	Jones EM and Yuill MM	1976	Determination of Residues of 3,6-dichloropicolinic Acid (DOWCO 290) in Rape Seed, Oil, Cake and Straw from a 1974-5 Trial Carried Out by Dow Personnel Study No. GHE-P-324 Non-GLP Unpublished	N	Corteva Agriscience
KCA 6.5.3 (IIA 6.5.2/02)	Jones EM and Yuill MM	1976	Determination of Residues of 3,6 dichloropicolinic Acid (DOWCO 290) in Rape Seed, Oil and Cake from 1975 Trials Carried Out by the Boots Company Limited Study No. GHE-P-325 Non-GLP Unpublished	N	Corteva Agriscience
KCA 6.5.3 (IIA 6.5.2/03)	Jones EM and Yuill MM	1976	Determination of Residues of 3,6 dichloropicolinic Acid (DOWCO 290) in Rape Seed, Cake, Oil and Straw from a Trial Carried Out in 1975 in Sweden by BT KEMI Study No. GHE-P-337 Non-GLP Unpublished	N	Corteva Agriscience
KCA 6.5.3 (IIA 6.5.2/04)	Jones EM and Yuill MM	1976	Determination of Residues of 3,6 dichloropicolinic Acid (DOWCO 290) in Rape Seed, Cake and Oil from Five Trial Locations in Sweden, 1975. Cooperator – Astra Ewos Study No. GHE-P-350 Non-GLP Unpublished	N	Corteva Agriscience

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.5.3 (IIA 6.5.2/05)	Jones EM and Yuill MM	1976	Determination of Residues of 3,6 dichloropicolinic Acid (DOWCO 290) in Rape Seed, Cake, Oil and Straw from a Trial Carried Out in Germany, 1975. Cooperator – Schreing AG Study No. GHE-P-395 Non-GLP Unpublished	N	Corteva Agriscience
KCA 6.6.1 (CA 6.6.1/1)	Yackovich, P. R. ; Lardie, T. S. ; Brink, D. L.	1993	A 10-1/2 Month Rotational Crops Study With 14C -Labeled Clopyralid - MET90080 DAS Study No. GH-C 2992 Dow AgroSciences LLC, Indianapolis, Indiana, United States GLP Unpublished	N	Corteva Agriscience
KCA 6.6.1 (CA 6.6.1/2)	Yackovich, P.R.; Lardie T.S.; Miller J.H.	1989	A 125-Day Rotational Crops Study with 14C Labelled Clopyralid DAS Study No. GH-C 2277 DowElanco, Midland, Michigan, USA Unpublished	N	Corteva Agriscience
KCA 6.6.1 (CA 6.6.1/3)	Hall, L. R.	2015 revised 2018	14C -Clopyralid: Metabolism in Confined Rotational Crops with a 30-Day Plant-back Interval DAS Study No. 130733 ABC Laboratories, Inc., Columbia, Missouri 65202, USA GLP Unpublished	N	Corteva Agriscience

† The amended reports (2019) have been evaluated as part of the Article 6 MRL evaluation (Finland 2018b; EFSA, 2021).

List of data submitted by the applicant and not relied on

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

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

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

Appendix 2 Detailed evaluation of the additional studies relied upon

A 2.1 Clopyralid

A 2.1.1 Stability of residues

A 2.1.1.1 Stability of residues during storage of samples

A 2.1.1.1.1 Storage stability of residues in plant products

A 2.1.1.1.1.1 Study 1

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>Study performed according to guideline and GLP requirements. Storage stability of clopyralid was demonstrated for up to 13 months in dried navy beans stored under frozen conditions. The study is acceptable.</i></p>
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Reference:	KCA 6.1
Report	Skaggs, C. S., Penning, B. N; 2021; Storage Stability of Clopyralid for One Year in Dried Beans; SGS North America, Inc., 1405 32nd Avenue, Brookings, SD 57006, USA.; Lab Study No.SGS-19-01-08; Corteva Study No. 191728 ; 27 October 2021
Guideline(s):	Yes, SANCO 825/00 Rev. 8.1, SANCO 3029/99 Rev.4
Deviations:	No
GLP:	Yes
Acceptability:	Yes

MATERIALS AND METHODS

Test Item(s)

Test item (common name):	Clopyralid
Purity:	95.9%
Description (physical state):	Off-white powder
Lot/batch no.:	910905 5P [TSN100167]

Method Scope

This method is applicable for the quantitative determination of residues of clopyralid in dried navy beans (high protein). The method was validated in dried navy beans over the concentration range of 0.01-0.10 mg/kg with a validated limit of quantitation of 0.01 mg/kg.

Method Principle

Residues of clopyralid are extracted from crop samples with 100:1 methanol:10N NaOH by blending for approximately 1 minute and shaking for 1 hour on a reciprocal shaker. The extracts are allowed to set ambient overnight. An aliquot of the extract is submitted to a nitrogen stream to remove the methanol and then brought back to volume with 1N sodium hydroxide. The clean up for crops is affected by partitioning

the basic extract with DCM. An aliquot of the extract is acidified with HCl and submitted to a polymeric reversed-phase solid phase extraction column (Waters, HLB SPE) cleanup and elution with DCM. After removal of the DCM using nitrogen blow down, the sample is reconstituted in 10:90, methanol:0.1% formic acid. The final extract is filtered through a 0.2-µm PTFE syringe filter and then analyzed by liquid chromatography coupled with negative-ion electrospray ionization tandem mass spectrometry (ESI LC/MS/MS).

Linearity

For each analyte, the linearity of detector response was evaluated using solvent standard solutions with calibration range 0.1 – 25 ng/mL (sample equivalence of 0.002 – 0.5 mg/kg). Calibration curves were calculated by linear regression analysis with 1/x weighting.

RESULTS AND DISCUSSION

Method Performance

The validated method performance was further demonstrated by concurrent recovery samples analyzed in this study. Mean concurrent recovery values were 70-110%; RSD ≤ 20%. The storage stability sample concentrations were corrected for the mean recovery values of the procedural samples. The results obtained are summarised in the following tables.

Table 1: Summary of quantitative recovery of clopyralid (m/z 189.8/146.0Q)

Matrix group	Matrix	LOQ	Fortification level	Recovery (%)		SD (%)	RSD (%)	n
			mg/kg	mean	range			
High protein	Dried navy beans	0.01	0.01	90	82-105	5.9	6.5	21
High protein	Dried navy beans	0.01	0.10	83	70-99	7.3	8.7	21

Stability

Storage stability of clopyralid was demonstrated for up to 13 months in dried navy beans.

Table 2: Results of frozen storage stability samples for clopyralid residues – dried navy beans

Days of storage	Spike level µg/g	Uncorrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)	Corrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0	0.10	0.0824	82	83	0.6	0.8	0.0988	99	99	0.6	0.6
0	0.10	0.0820	82				0.0988	99			
0	0.10	0.0832	83				0.1000	100			
31	0.10	0.0736	74	74	1.6	2.2	0.0860	86	86	1.5	1.8
31	0.10	0.0763	76				0.0884	88			
31	0.10	0.0733	73				0.0849	85			
62	0.10	0.0679	68	73	5.6	7.7	0.0861	86	93	7.0	7.6
62	0.10	0.0728	73				0.0924	92			
62	0.10	0.0791	79				0.1000	100			
90	0.10	0.0737	74	74	0.5	0.7	0.0914	91	92	1.2	1.3
90	0.10	0.0740	74				0.0914	91			
90	0.10	0.0747	75				0.0926	93			
182	0.10	0.0775	77	83	6.5	7.9	0.0846	85	91	7.2	7.9
182	0.10	0.0901	90				0.0989	99			

182	0.10	0.0810	81				0.0890	89			
273	0.10	0.0939	94				0.1044	104			
273	0.10	0.0873	87	89	4.8	5.4	0.0967	97	98	5.1	5.2
273	0.10	0.0846	85				0.0944	94			
395	0.10	0.1053	105				0.1458	146			
395	0.10	0.0970	97	99	5.2	5.2	0.1347	135	138	7.0	5.1
395	0.10	0.0957	96				0.1333	133			

Table 3: Summary of concurrent recoveries of clopyralid from dried navy beans

Matrix	Spike level (mg/kg)	Storage Interval (days)	Sample size (n)	Individual procedural recoveries (%)	Mean ± std dev
Clopyralid					
Dried navy beans	0.01	0	3	93 ; 90 ; 84	89 ± 5
		31	3	95 ; 86 ; 90	90 ± 5
		62	3	83 ; 94 ; 91	89 ± 6
		90	3	89 ; 84 ; 90	88 ± 3
		182	3	105 ; 92 ; 100	99 ± 7
		273	3	93 ; 91 ; 83	89 ± 5
		395	3	83 ; 82 ; 89	85 ± 4
High protein group	0.10	0	3	84 ; 83 ; 83	83 ± 1
		31	3	86 ; 87 ; 85	86 ± 1
		62	3	80 ; 78 ; 79	79 ± 1
		90	3	89 ; 79 ; 74	81 ± 8
		182	3	92 ; 93 ; 89	91 ± 2
		273	3	99 ; 85 ; 86	90 ± 8
		395	3	76 ; 71 ; 70	72 ± 3

CONCLUSION

The data indicates that residues of clopyralid are stable for at least 395 days (13 months) in dried navy beans stored under frozen conditions. These conditions are consistent with the storage of actual field samples.

A 2.1.1.1.1.2 Study 2

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/ Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023).</p> <p>The conclusions of the assessment are presented below: <i>Study performed according to method similar to OECD guideline 506 and GLP requirements. The minor deviation was identified – one fortification level instead of two as is recommended in OECD guideline was used. However, the method can be considered to be fit for purpose. The data indicates that residues of clopyralid are stable for at least 24 18 months in strawberries stored under frozen conditions.</i></p> <p>The evaluator added some comments below: It should be noted that the time point of 725 days (~24 months) is not acceptable due to the</p>
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	low recovery values and low means of the values. This is not in line with the requirement of the OECD Guideline for the Testing of Chemicals 506. Thus, in our opinion, the study demonstrates storage stability for up to 18 months for clopyralid in strawberries.
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Reference:	KCA 6.1
Report	Teasdale, R.; 1996; Frozen Storage Stability of Clopyralid Residues in Strawberries; CEM Analytical Services, North Ascot, Berkshire, UK; Lab Study No. CEMS-235; Sponsor Study No. GHE-P-4832 ; 17 June 1996
Guideline(s):	Similar to OECD Guideline 506
Deviations:	No
GLP:	Yes
Acceptability:	Yes

MATERIALS AND METHODS

Test Item(s)

Test item (common name):	Clopyralid
Purity:	N/A
Description (physical state):	Solid
Lot/batch no.:	N/A (TSN100042)

Method Scope

This method is applicable for the quantitative determination of residues of clopyralid in agricultural commodities (acidic crop). The method was independently validated in strawberries over the concentration range of 0.10 mg/kg with a validated limit of quantitation of 0.1 mg/kg.

Method Principle

Residues of clopyralid are extracted from samples by homogenising and shaking with caustic methanol. After centrifugation, an aliquot of the supernatant was acidified and the fluroxypyr partitioned into dichloromethane. The dichloromethane layer was then partitioned into aqueous sodium bicarbonate and, after acidification, into diethyl ether. After removal of the diethyl ether by evaporation, the residue was treated with butylation reagent. The clopyralid (as the butyl ester) was partitioned into hexane in the presence of water prior to a silica Bond Elut clean-up procedure. Analysis was by capillary gas chromatography with electron capture and the external standardisation technique.

Linearity

For each analyte, the linearity of detector response was evaluated using solvent standard solutions with calibration standards range of 1-50 ng/mL (sample equivalence of 0.0125 – 0.625 mg/kg). Calibration curves were calculated by linear regression analysis with 1/x weighting, with a correlation coefficient > 0.995.

RESULTS AND DISCUSSION

Method Performance

The validated method performance was further demonstrated by virtue of concurrent recovery samples analyzed in this study. Mean concurrent recovery values were 70-110%; RSD ≤ 20%. The storage stability sample concentrations were corrected for the mean recovery values of the procedural samples. The results obtained are summarised in the following tables.

Table 1: Summary of quantitative recovery of Clopyralid

Matrix group	Matrix	LOQ	Fortification level	Recovery (%)		SD	RSD	n
			mg/kg	mean	range	(%)	(%)	

Acidic Crop	Strawberries	0.1	0.10	80	68-89	-	9.7	7
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Stability

Storage stability of clopyralid was demonstrated for up to 24 months in strawberries.

Table 2: Results of frozen storage stability samples for Clopyralid residues – Strawberries

Days of storage	Spike level µg/g	Uncorrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)	Corrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0	0.10	0.0872	93	80	8.0	10.0	0.093	107	91	9.3	10.1
0	0.10	0.0740	78				0.078	90			
0	0.10	0.0666	70				0.070	80			
0	0.10	0.0746	79				0.079	91			
0	0.10	0.0703	74				0.074	85			
0	0.10	0.0782	83				0.083	95			
32	0.10	0.0732	78	73	-	-	0.078	107	100		
32	0.10	0.0641	68				0.068	93			
124	0.10	0.0730	77	73	-	-	0.077	113	107		
124	0.10	0.0653	69				0.069	100			
250	0.10	0.0773	81	89	-	-	0.081	91	100		
250	0.10	0.0911	96				0.096	108			
370	0.10	0.0745	78	78	-	-	0.078	98	98		
370	0.10	0.0746	78				0.078	98			
549	0.10	0.0666	72	73	-	-	0.072	94	95		
549	0.10	0.0673	73				0.073	95			
725	0.10	0.0645	68	66	12.8	19.5	0.068	80	77	15.2	19.5
725	0.10	0.0802	85				0.085	100			
725	0.10	0.0460	48				0.048	56			
725	0.10	0.0690	73				0.073	86			
725	0.10	0.0556	58				0.058	68			
725	0.10	0.0586	62				0.062	73			

CONCLUSION

The data indicates that residues of clopyralid are stable for at least 24 months in strawberries stored under frozen conditions. These conditions are consistent with the storage of actual field samples.

A 2.1.1.1.2 Storage stability of residues in animal products

A 2.1.1.1.2.1 Study 1

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/ Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>Study performed according to OECD guideline 506 and GLP requirements. The data indicates that residues of clopyralid are stable for 18 months in pollen, nectar or honey stored under frozen conditions. Study is acceptable.</i></p>
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Reference:	KCA 6.1
Report	Forbes, T.; Cross, M.; 2020; Frozen Storage Stability of Clopyralid in Pollinator Matrices; CEM Analytical Services (CEMAS), Imperial House, Oaklands Business Centre Oaklands Park, Wokingham, Berkshire, RG41 2FD, UK; Lab Study No. CEMS-8756; Corteva Study No. 180869 ; 09 April 2020
Guideline(s):	OECD Guideline 506
Deviations:	No
GLP:	Yes
Acceptability:	Yes

MATERIALS AND METHODS

Test Item(s)

Test item (Common name):	Clopyralid
Purity:	99.9 (% , w/w)
Description (physical state):	White solid
Lot/batch no.:	YC2-106153-68 (TSN301194)

Method Scope

The analytical method used for the determination of clopyralid was Dow AgroSciences Method study ID 171332, “Validation of an Analytical Method for the Determination of Clopyralid in Pollinator Matrices.” This method is applicable for the quantitative determination of residues of clopyralid in pollinator matrices (pollen, nectar and honey). The method was validated over the concentration range of 0.001-0.01 mg/kg with a validated limit of quantitation of 0.001 mg/kg.

Method Principle

Residues of clopyralid are extracted from samples by shaking with a mixture of methanol containing 10 N sodium hydroxide. The final sample extracts are analyzed for clopyralid by gas chromatography with negative ion electrospray ionization mass spectrometry (GC/NCI-MS).

Linearity

The linearity of detector response was evaluated using solvent standard solutions for nectar and honey, but matrix-matched standard solutions for pollen. Calibration curves were calculated by linear regression analysis with 1/x weighting.

RESULTS AND DISCUSSION

Method Performance

The validated method performance was further demonstrated by virtue of concurrent recovery samples analyzed in this study. Mean concurrent recovery values were 70-110%; RSD ≤ 20%. Recoveries of the stored samples were presented as both uncorrected and corrected for the mean procedural recoveries in each analytical batch. The results obtained are summarised in the following tables.

Table 1: Summary of quantitative ion recovery of Clopyralid (m/z 233.0)

Matrix group	Matrix	Fortification level (mg/kg)	Recovery (%)		SD (%)	RSD (%)	n
			mean	range			
	Pollen	0.001	86	67 – 120	11.1	13.0	18
		0.01	83	73 – 95	6.1	7.4	18

Pollinator	Nectar	0.001	97	82 – 110	8.8	9.1	18
		0.01	90	79 – 99	5.2	5.8	18
	Honey	0.001	90	76 – 100	6.3	7.0	18
		0.01	90	76 – 99	5.6	6.2	18

Stability

Storage stability of clopyralid was demonstrated for 550 – 557 days (18 months) in pollen, nectar or honey.

Table 2: Results of frozen storage stability samples for Clopyralid in Pollen

Days of storage	Spike level µg/g	Uncorrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)	Corrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0	0.10	0.0085	85	94	8.1	8.6	0.0103	103	114	9.3	8.2
0	0.10	0.0098	98				0.0118	118			
0	0.10	0.0100	100				0.0120	120			
33	0.10	0.0097	97	91	5.1	5.6	0.0103	103	97	5.1	5.3
33	0.10	0.0090	90				0.0096	96			
33	0.10	0.0087	87				0.0093	93			
91	0.10	0.0097	97	93	3.2	3.4	0.0115	115	111	3.6	3.2
91	0.10	0.0091	91				0.0108	108			
91	0.10	0.0092	92				0.0110	110			
187	0.10	0.0079	79	77	2.0	2.6	0.0098	98	96	2.1	2.2
187	0.10	0.0077	77				0.0097	97			
187	0.10	0.0075	75				0.0094	94			
364	0.10	0.0102	102	98	5.1	5.3	0.0123	123	118	6.2	5.3
364	0.10	0.0092	92				0.0111	111			
364	0.10	0.0099	99				0.0120	120			
557	0.10	0.0094	94	90	9.3	10.4	0.0114	114	108	10.7	9.9
557	0.10	0.0096	96				0.0115	115			
557	0.10	0.0079	79				0.0096	96			

Table 3: Results of frozen storage stability samples for Clopyralid in Nectar

Days of storage	Spike level µg/g	Uncorrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)	Corrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0	0.10	0.0089	89	91	2.7	3.0	0.0090	90	92	2.8	3.0
0	0.10	0.0094	94				0.0095	95			
0	0.10	0.0089	89				0.0090	90			
28	0.10	0.0091	91	97	5.3	5.5	0.0093	93	99	5.3	5.3
28	0.10	0.0099	99				0.0101	101			
28	0.10	0.0101	101				0.0103	103			
89	0.10	0.0106	106	99	6.1	6.1	0.0116	116	109	6.1	5.6
89	0.10	0.0095	95				0.0105	105			
89	0.10	0.0096	96				0.0106	106			
187	0.10	0.0095	95	96	4.2	4.3	0.0102	102	103	4.2	4.0
187	0.10	0.0093	93				0.0100	100			
187	0.10	0.0101	101				0.0108	108			
364	0.10	0.0101	101				0.0107	107			

364	0.10	0.0111	111	106	5.0	4.7	0.0118	118	113	5.5	4.9
364	0.10	0.0106	106				0.0113	113			
550	0.10	0.0102	102	102	3.5	3.5	0.0116	116	116	3.5	3.0
550	0.10	0.0098	98				0.0112	112			
550	0.10	0.0105	105				0.0119	119			

Table 4: Results of frozen storage stability samples for Clopyralid in Honey

Days of storage	Spike level µg/g	Uncorrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)	Corrected µg/g found	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0	0.10	0.0086	86	89	4.4	5.0	0.0099	99	102	5.1	5.0
0	0.10	0.0094	94				0.0108	108			
0	0.10	0.0087	87				0.0100	100			
33	0.10	0.0089	89	93	4.7	5.1	0.0101	101	105	5.1	4.9
33	0.10	0.0091	91				0.0104	104			
33	0.10	0.0098	98				0.0111	111			
91	0.10	0.0106	106	101	5.0	5.0	0.0115	115	110	5.6	5.1
91	0.10	0.0102	102				0.0111	111			
91	0.10	0.0096	96				0.0104	104			
187	0.10	0.0101	101	100	1.2	1.2	0.0111	111	110	1.0	0.9
187	0.10	0.0099	99				0.0109	109			
187	0.10	0.0101	101				0.0110	110			
364	0.10	0.0118	118	112	7.2	6.4	0.0123	123	117	7.2	6.2
364	0.10	0.0104	104				0.0109	109			
364	0.10	0.0114	114				0.0119	119			
557	0.10	0.0105	105	106	0.6	0.5	0.0122	122	123	1.0	0.8
557	0.10	0.0106	106				0.0124	124			
557	0.10	0.0106	106				0.0123	123			

CONCLUSION

The data indicates that residues of clopyralid are stable for 550 – 557 days (18 months) in pollen, nectar or honey stored under frozen conditions. These conditions are consistent with the storage of actual field samples.

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

A 2.1.2.1 Nature of residue in plants

A 2.1.2.1.1 Nature of residue in primary crops

A 2.1.2.1.1.1 Study 1, wheat

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/ Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>The study was performed according to current guidelines and GLP requirements. The study was already evaluated and considered acceptable (EFSA, 2021). Only parent clopyralid was identified in all tested samples, constituting 93-98% of TRR while not other significant</i></p>
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	<i>metabolites were identified.</i>
	Remark: In our opinion, this study should be evaluated at the EU level, not national level. It should be noted that, the study was evaluated during the latest MRL evaluation (ER by Finland 2020 / EFSA 2021). The study was deemed acceptable and supported the current residue definitions for enforcement and risk assessment.

Reference:	KCA 6.2.1/01
Report:	Morton Lloyd, G.; 2020; The Metabolism of [¹⁴ C]-Clopyralid in Wheat; Charles River Laboratories Edinburg Ltd., Tranent, UK; Lab Study No. 229882; DAS Study No. 191200; 19 June 2020; Unpublished
Guidelines:	OECD 501, OPPTS 860.1300
Deviations:	none
GLP:	Yes

BACKGROUND INFORMATION

Clopyralid (3,6-dichloropyridine-2-carboxylic acid) is a selective herbicide developed for use on a variety of agricultural crops including wheat.

MATERIALS AND METHODS

Test Item(s)

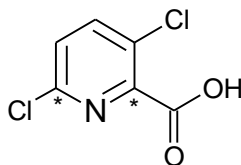
Non-radiolabelled test item #1

Test item (Common name):	Clopyralid
Purity:	99.9%
Description (physical state):	Solid
Lot/batch no.:	YC2-106153-68 (TSN301194)

Radiolabelled test item #1

Test item (Common name):	[¹⁴ C]-Clopyralid
Name:	3,6-Dichloropicolinic acid-2,6- ¹⁴ C, X755015

Structural Formula:
Position of labelling (*)



Lot/batch no.:	INV306082
Radiochemical purity:	99.0%
Specific radioactivity:	35.0 mCi/mmol

Methods

Test Site Information

Testing environment:	outdoor test plots - F, glasshouse when outdoor conditions were unfavourable (early October 2019) - G
Container description:	Plastic, internal dimensions: 0.755 x 0.555 m, 4 used

Soil type: sandy loam (UK and USDA)
Soil characteristics: 1.70% OC
pH 6.8 (in water)
CEC 10.2 meq/100 g
67% Sand (UK)
20% Silt (UK)
13% Clay (UK)

Any adverse weather conditions: No
Any adverse insect or disease problems: No

Study Use Pattern

Application method: Foliar
Formulation type: DMA salt, GF-2895
Application rate: 120 g ae/ha (target), actual 126 g ae/ha
Number of applications: 1
Timing of applications: 23 July 2019, BBCH 39
PHI (Days after Treatment): 3 (forage), 23 (hay), 92 (mature straw & grain)

Test System

Organism (*Species*): Spring Wheat (*Triticum*)
Variety: Mulika
Crop group: Cereal & forage/pulses
Growth stage at application: BBCH 39
Harvested RAC: forage, hay, grain & straw
Growth stage at harvest: Forage: BBCH 43 (26 Jul 2019), hay: BBCH 71 (15 Aug 2019), mature: BBCH 89 (23 Oct 2019)
Harvesting procedure: Approximately $\frac{1}{4}$ plants randomly selected for forage sampling, cut at soil surface. Approximately $\frac{1}{3}$ remaining plants cut at soil surface for hay; hay was air-dried in a glasshouse for 5 days. Remaining plants were harvested at maturity. The heads were removed by hand and grain threshed; the chaff was combined with the straw. After weighing, the samples were stored frozen (-20 °C).

Sample Handling and Preparation

Each frozen plant fraction was homogenised with excess carbon dioxide chips using a Waring PB20 or a Hobart VCB61 blender. The carbon dioxide was then allowed to sublime while frozen prior to removal of sub-samples for combustion.

Sub-samples (five each, *ca.* 0.1 – 0.3 g) of the milled tissue were analysed by oxidative combustion to determine the total radioactive residues in the sample.

Extraction of Sample Residues

Aqueous Acetonitrile Extraction (E1-2)

Approximately 25 g of homogenized tissue was extracted with approx. 125 – 250 mL of 1:1 acetonitrile:water (v/v). The mixture was blended using a Silverson SL2 TopDrive homogenizer for approximately 5 minutes. The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and the extraction was repeated once more using *ca.* 62.5 – 125 mL solvent, after which the extracts were pooled and volume measured. Triplicate aliquots of each pooled extract were analysed by liquid scintillation counting.

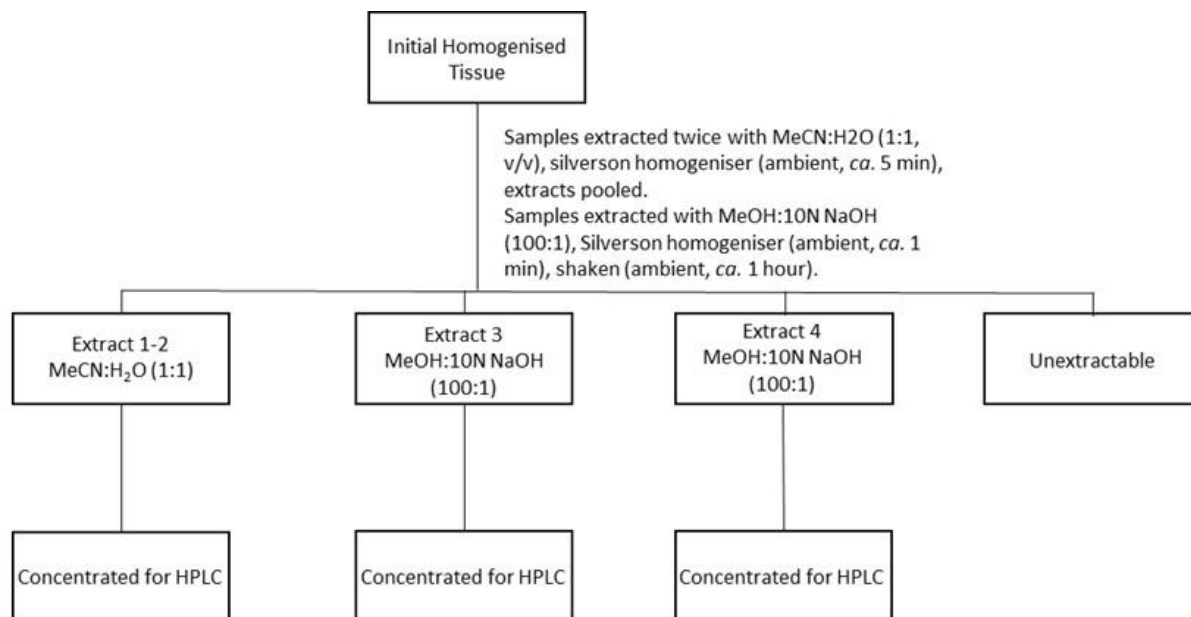
For “dry” samples (hay, straw and grain), the extraction tissue was soaked in the first extract solvent for *ca.* 30 minutes prior to blending.

The remaining solid for each sample was further extracted as described below.

Methanolic Base Extraction (E3 and E4)

Following neutral organic solvent extraction, all samples were subjected to base extraction. The entire remaining residue sample following neutral organic extraction was homogenised in the presence of *ca.* 62.5 – 125 mL methanol:10 N sodium hydroxide (100:1, v/v) for *ca.* 1 minute using a Silverson SL2 TopDrive homogeniser, before being shaken on a reciprocal shaker for *ca.* 1 hour. The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and the extract volume measured. For wheat straw and grain, the extraction was repeated once more using the same volume of solvent (E4). Triplicate aliquots of each extract were analysed by liquid scintillation counting. The post extracted tissue for all samples was dried and aliquots combusted.

Figure 1: Representative Flowchart of Sample Extractions and Concentrations



Note: Above flowchart is a generic overview of sample processing. Selected samples have not had all extractions conducted.

Further Extraction

Aqueous Base Further Extractions (FE1)

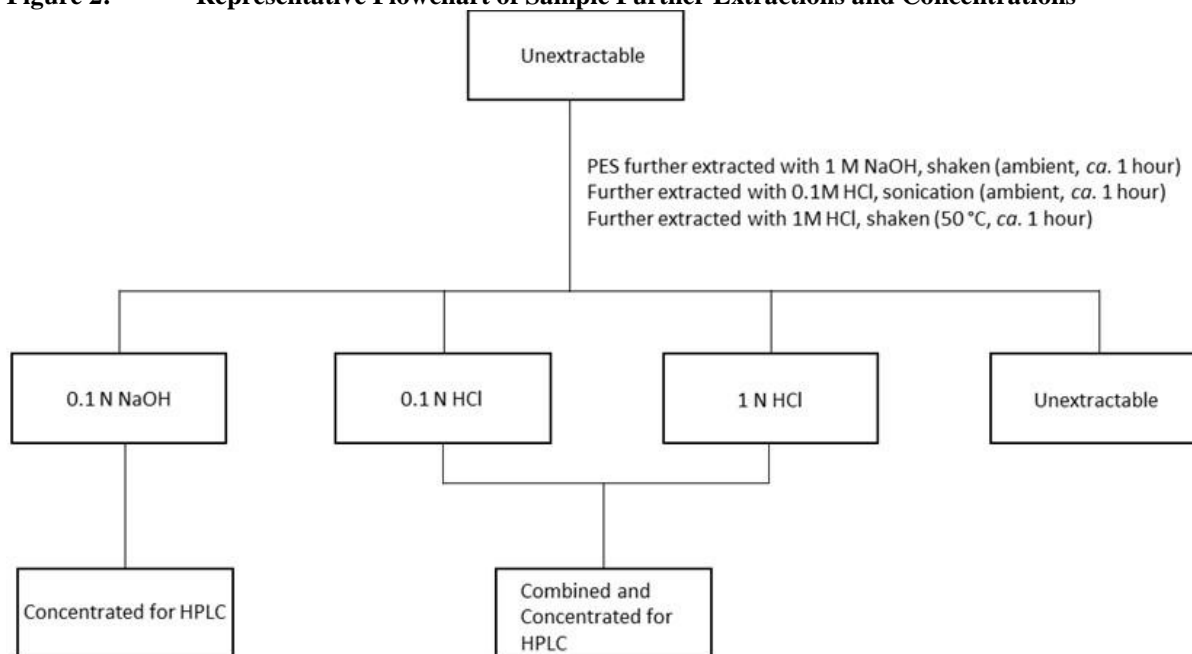
Aliquots of dried forage, hay, straw and grain post extraction solids were subjected to further base extraction. Approximately 1.5 – 10 g of post extraction solids was extracted with 30 - 100 mL of 0.1 M NaOH (20:1, v/w for forage solvent:PES, 10:1, v/w for hay, straw and grain solvent:PES). The mixture was shaken on a reciprocal shaker for *ca.* 1 hour, at ambient, before the solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes). The extract volume for forage, hay and straw was measured and triplicate aliquots of each extract were analysed by liquid scintillation counting. Due to the viscous nature of the grain extract, this was diluted to 100 mL with the addition of Milli-Q water prior to analysis by liquid scintillation counting. The forage post extracted tissue was dried and aliquots combusted.

Acid Extraction (FE2 and FE3)

Following extraction with base (FE1), hay, grain and straw post extraction solids were subjected to acid extraction. The entire remaining residue was extracted with 50 mL of 0.1 M HCl (5:1, v/w solvent:PES) with ultrasonication at ambient for *ca.* 1 hour. The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes) and the extract volume measured. Triplicate aliquots of each extract (FE2) were analysed by liquid scintillation counting. The grain post extracted tissue was dried and aliquots combusted.

Remaining hay and straw residue were further extracted with 50 mL of 1 M HCl (5:1, v/w solvent:PES). Samples were shaken by hand for *ca.* 1 minute, before shaking in a water bath at *ca.* 50 °C for *ca.* 1 hour. The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes) and the extract volume measured. Triplicate aliquots of each extract (FE3) were analysed by liquid scintillation counting. The post extracted tissue was dried and aliquots combusted.

Figure 2: Representative Flowchart of Sample Further Extractions and Concentrations



Note: Above flowchart is a generic overview of sample processing. Selected samples have not had all extractions conducted.

Base Treatment of Suspected Conjugate Fractions

Neutral Organic Extract Base Treatment

To further characterise the neutral-organic sample extracts, aliquots of forage, hay, straw and grain pooled extract 1-2 were subjected to base treatment, using methodology similar to the field trial analytical method (analytical method 120610). The solvent was removed under nitrogen until dryness (ambient temperature) and the sample reconstituted in an equal volume (amount of initial neutral-organic extract aliquot) of methanol:10 N NaOH (100:1, v/v). Samples were homogenised for *ca.* 1 minute using a Silverson SL2 TopDrive homogeniser, shaken for *ca.* 1 hour on a reciprocal shaker and left at ambient overnight. Samples were centrifuged and the supernatants analysed by LSC. Where recoveries were low, a wash of the pellet after centrifuging was conducted using water:acetonitrile, 1:1 (v/v). The wash was centrifuged prior to analysis by LSC. Base treatment samples were combined with the respective wash prior to HPLC analysis. Separate aliquots of forage, hay, straw and grain pooled extract 1-2 were processed using the same procedure with methanol:water (100:1, v/v), no caustic, to act as control samples.

Control Fortification

Homogenised control wheat grain was fortified with [^{14}C]-clopyralid radiodiluted stock solution prior to extraction with aqueous acetonitrile, as described above. The extract was concentrated and analysed by HPLC, as described above for the treated tissue.

Metabolite Isolation and Identification

Non-radiolabeled clopyralid was used as a reference standard for chromatographical comparison. HPLC retention time comparisons were made between this reference standard and the radioactive peaks in the chromatograms in order to provide initial tentative identifications.

Based on the initial HPLC analyses, clopyralid fractions were targeted for isolation and structure confirmation. This was accomplished using a grain aqueous acetonitrile SPE sample and a straw methanolic base SPE sample which were submitted for radio LC-MS and LC-MS/MS analyses. A bulk grain aqueous acetonitrile extract was submitted for MRM-MS analyses.

Analytical Methodology

Total ^{14}C measurement

Oxidative Combustion:

Approximately 0.1-0.3 g sub-samples of each sample were weighed in 3 to 5 replicates into Combustorcones® (Packard Bioscience) and combusted using a Perkin Elmer Tri-Carb 307 Automatic Sample Oxidiser. The resultant $^{14}\text{CO}_2$ was absorbed in Carbo-Sorb® (Packard Bioscience), mixed automatically with Permafluor® scintillation fluid (Packard Bioscience) and the radioactivity determined by LSC.

The efficiency of the oxidiser was determined daily, and at regular intervals throughout each batch of analyses. Carbon-14 standards were combusted at the beginning of each batch of analyses. Combustion efficiencies were in excess of 97.1%. Combustion values were not corrected for the oxidizer efficiencies.

Liquid Scintillation Counting:

The liquid scintillation counters automatically converted the radioactivity counting rate in counts per minute (cpm) to disintegrations per minute (dpm) using an external standard to correct for sample quenching. The instrument was calibrated approximately every three months with a set of ten quenched standards. Each day of use, the instrument was normalized and its performance was checked with respect to background cpm value, unquenched standard cpm value, and quenched standard dpm value for a range of quenched standards. The scintillation counters used were a Packard 2100TR Tri-Carb (Packard Instrument Co.). The dpm value for the liquid samples was determined by LSC after diluting an appropriate aliquot of the sample with Aquasafe 500 Plus scintillation cocktail (Zinsser Analytic, Maidenhead, UK) and counting for at least five minutes.

Sample Concentration

The general sample processing procedure for the aqueous acetonitrile pooled extract 1-2 was to concentrate aliquots either under nitrogen or by rotary evaporation (at ambient), to a reduced volume or dryness before reconstitution to a known volume in a suitable solvent (water, water:acetonitrile or water:methanol). Samples were centrifuged (*ca.* 13,000 rpm for *ca.* 10 minutes) and the supernatant analysed by LSC and HPLC. Aliquots of base extracts (extract 3 and 4) were neutralised to *ca.* pH 7 using hydrochloric acid solution before concentration in a similar manner to the neutral organic extracts (E1-2).

Where required, aliquots of base treatment and associated control samples were combined with the respective wash prior to concentration. Base treated samples were processed in a similar manner to the base extracts (*e.g.*, neutralized). Control samples were processed in a similar manner to the neutral organic extracts.

Aliquots of mild base further extracts (FE1) were neutralized to *ca.* pH 7 using hydrochloric acid solution prior to centrifuging (*ca.* 13,000 rpm for *ca.* 10 minutes). The resulting supernatants were concentrated in a similar manner to the neutral organic extracts.

For hay and straw samples, equal proportion of the mild acid (FE2) and stronger acid (FE3) further extracts were combined and neutralised to *ca.* pH 7 using sodium hydroxide solution prior to centrifuging (*ca.* 13,000 rpm for *ca.* 10 minutes). The resulting supernatants were concentrated in a similar manner to the neutral organic extracts.

For grain, pH analysis of the mild acid further extract (FE2) determined it to be basic. This was due to carry-over from the mild base further extraction (FE1). The sample was neutralized to *ca.* pH 7 using hydrochloric acid solution prior to centrifuging (*ca.* 13,000 rpm for *ca.* 10 minutes). The resulting supernatants were concentrated in a similar manner to the neutral organic extracts.

Solid phase extraction (SPE)

In order to remove endogenous material from samples prior to mass spec analysis, selected samples were subjected to solid phase extraction (SPE).

Aliquots of forage concentrated extract 3 (methanolic base), straw concentrated extract 3 (methanolic base), and grain concentrated extract 1-2 (neutral organic) were loaded onto pre-conditioned C18 SPE cartridges (Varian, 6cc, 1 g) and the radioactivity washed off with acetonitrile:water (3:7, v/v). The organic solvent of each sample was removed under nitrogen before each was pre-treated with 5% NH_4OH (250 – 500 μL) prior to loading on to a MAX cartridge (Oasis, 6cc, 500 mg). Where required, samples were split across multiple MAX cartridges and eluates combined. MAX cartridges washed with 5% NH_4OH and eluted sequentially with methanol, 2% formic acid in organic (methanol or methanol:acetonitrile, 40:60) and 2% TFA in methanol. The volume of each eluate was measured and aliquots were removed for LSC analysis. Fractions which contained radioactivity were neutralised, concentrated and reconstituted in a suitable solvent prior to HPLC and MS analysis.

High performance liquid chromatography (HPLC) for quantitation

HPLC analyses of all sample extracts were accomplished using a Waters SPHERISORB ODS 2 column (150 x 4.6 mm, 5 µm particle size; 1.0 mL/min; UV detection at 280 nm) and a 1 step, non-linear gradient.

Mass spectral analysis (LC/MS) for identification of transformation products

A forage methanolic base, straw methanolic base and grain neutral-organic samples were cleaned-up by SPE to aid confirmation of clopyralid by mass spectral analysis. Mass spectral analysis was also conducted on bulk treated wheat grain and fortified wheat grain samples.

RESULTS AND DISCUSSION

Results of In-Life Phase

The [¹⁴C]-clopyralid plot received 21.095 mg, equivalent to 125.87 g a.e./ha (104.9% of target of 120 g a.e./ha). Radiochemical purity and stability of the formulated application solution pre- and post-application were ≥97.7%, indicating the stability of the radiolabelled test item throughout the formulation and application process.

Total Radioactive Residue (TRR) Levels

TRR levels in all samples, expressed as mg/kg of parent equivalents are shown below.

Table 21: Total radioactive residues (TRRs) in Plant Samples Collected for Clopyralid Nature of Residue in Wheat Study

Matrix	Days after Application	TRR (mg eq./kg)
Wheat Forage	3	1.879
Wheat Hay	23	3.717
Wheat Straw	92	2.134
Wheat Grain	92	1.128

¹ = mg eq/kg = mg parent equivalents per kg of tissue

Distribution of Residues Following Extraction

Forage

Accountability, between combustion and extraction data, was 103.0% for forage. When normalised with respect to accountability, aqueous acetonitrile recovered 90.5 %TRR (1.700 mg eq/kg) and methanolic base extracted 5.0 %TRR (0.095 mg eq/kg). The level of non-extractable radioactivity following initial extractions accounted for 3.2 %TRR (0.060 mg eq/kg) and therefore was subjected to further extraction. A further aqueous mild base extraction released 2.6 %TRR (0.050 mg eq/kg). Final unextracted residues accounted for 1.8 %TRR (0.035 mg eq/kg).

Hay

Accountability, between combustion and extraction data, was 107.6% for hay. When normalised with respect to accountability, aqueous acetonitrile recovered 78.6 %TRR (2.923 mg eq/kg) and methanolic base extracted 13.2 %TRR (0.491 mg eq/kg). The level of non-extractable radioactivity following initial extractions accounted for 8.6 %TRR (0.318 mg eq/kg) and therefore was subjected to further extraction. Further aqueous mild base, mild acid and stronger acid extractions (FE1, FE2 and FE3) released a total of 5.3 %TRR (0.197 mg eq/kg). Final unextracted residues accounted for 2.9 %TRR (0.107 mg eq/kg).

Straw

Accountability, between combustion and extraction data, was 101.5% for straw. When normalised with respect to accountability, aqueous acetonitrile recovered 50.6 %TRR (1.081 mg eq/kg) and methanolic base extracts released a further 34.7 %TRR (0.739 mg eq/kg). The level of non-extractable radioactivity following initial extractions accounted for 13.7 %TRR (0.293 mg eq/kg) and therefore was subjected to further extraction. Further mild base, mild acid and stronger acid extractions (FE1, FE2 and FE3) released a total of 7.5 %TRR (0.160 mg eq/kg). Final unextracted residues accounted for 7.2 %TRR (0.154 mg eq/kg).

Grain

Accountability, between combustion and extraction data, was 95.4% for grain. When normalised with respect to accountability, aqueous acetonitrile recovered 72.6%TRR (0.820 mg eq/kg) and methanolic base

extracts released a further 20.4 %TRR (0.229 mg eq/kg). The level of non-extractable radioactivity following initial extractions accounted for 7.2 %TRR (0.082 mg eq/kg) and therefore was subject to further extractions. Further mild base and mild acid extractions released 3.4 %TRR (0.038 mg eq/kg). Final unextracted residues accounted for 3.7 %TRR (0.041 mg eq/kg).

The distribution of the residues in the wheat samples among the fractions generated following the extractions, expressed both as a percentage of the total sample residue and as mg/kg of clopyralid equivalents is shown below.

Table 22: Normalised Characterisation of Residues in Wheat Following a Foliar Treatment with [¹⁴C]-Clopyralid

	Wheat							
	Forage		Hay		Straw		Grain	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR ¹	100.0	1.879	100.0	3.171	100.0	2.134	100.0	1.128
Pooled Extract 1-2 Acetonitrile:water (1/1, v/v)	90.5	1.700	78.6	2.923	50.6	1.081	72.6	0.820
Extract 3 Methanol:10 N NaOH (100/1, v/v)	5.0	0.095	13.2	0.491	26.8	0.571	14.2	0.159
Extract 4 Methanol:10 N NaOH (100/1, v/v)	NC		NC		7.9	0.168	6.2	0.070
Sum of initial extracts (1-4)	95.5	1.795	91.8	3.414	85.3	1.820	93.0	1.049
Further Extract 1 0.1 M NaOH	2.6	0.050	3.3	0.125	4.5	0.097	2.0	0.022
Further Extract 2 0.1 M HCl	NC		1.3	0.048	2.1	0.044	1.4	0.016
Further Extract 3 1 M HCl	NC		0.7	0.024	0.9	0.019	NC	
Sum of further extracts (FE1-3)	2.6	0.050	5.3	0.197	7.5	0.160	3.4	0.038
Sum of neutral and further extracts	98.1	1.845	97.1	3.611	92.8	1.980	96.4	1.087
Characterised by HPLC	98.1	1.845	97.1	3.611	92.8	1.980	96.4	1.087
Unextracted	1.8	0.035	2.9	0.107	7.2	0.154	3.7	0.041
Accountability (%) ²	99.9		100.0		100.0		100.1	

¹ = TRR determined by initial extractions

² = Accountability = %TRR total extractable + %TRR unextractable

NC = Not Conducted

Characterization and Identification of Residues

Forage:

The main component in the aqueous acetonitrile extract was clopyralid, accounting for 66.0 %TRR, 1.240 mg eq/kg (E1-2). A number of less polar components (maximum individual component 5.4 %TRR) were also detected, with the entire region accounting for 21.0 %TRR (0.394 mg eq/kg). Upon mild base treatment, primarily clopyralid was detected (98.7 %ROI). Therefore, the less polar components are identified as base-labile clopyralid. Two more polar components, accounting for 3.4 %TRR (0.064 mg eq/kg) were also detected. These components were also reduced to clopyralid under base treatment, and therefore, are identified as base-labile clopyralid.

Base-labile clopyralid was the only component detected in the organic mild base extract (E3), accounting for 5.0 %TRR, 0.095 mg eq/kg.

Base-labile clopyralid was the only component detected in the aqueous mild base further extract (FE1), accounting for 2.6 %TRR, 0.050 mg eq/kg.

Overall, 98.1% of the radioactivity was extractable and 94.6 %TRR (1.779 mg eq/kg) was shown to be clopyralid. Of this, 66.0 %TRR (1.240 mg eq/kg) was “free” clopyralid and 32.0 %TRR (0.603 mg eq/kg) was base-labile clopyralid.

Hay:

A resolved clopyralid peak accounted for 15.4 %TRR, 0.571 mg eq/kg in the aqueous acetonitrile extracts (E1-2). A large unresolved, less polar, region, accounting for 63.3 %TRR, 2.351 mg eq/kg was also detected. Upon mild base treatment, primarily clopyralid was detected (97.7 %ROI) and therefore is identified as base-labile clopyralid.

Base-labile clopyralid was the only component detected in the organic base extract (E3), accounting for 13.2 %TRR, 0.491 mg eq/kg.

Base labile clopyralid was the main component detected in the aqueous mild base (FE1) further extract, accounting for 3.3 %TRR, 0.125 mg eq/kg. Acid-labile clopyralid was the only component present in the combined acidic further extracts (FE2-3), accounting for 2.0 %TRR, 0.072 mg eq/kg. Overall, 97.1% of the radioactivity was extractable, and 97.2 % TRR (3.608 mg eq/kg) was shown to be clopyralid. Of this, 15.4 %TRR (0.571 mg eq/kg) was “free” clopyralid, 79.8 %TRR (2.965 mg eq/kg) was base-labile clopyralid and 2.0 %TRR (0.072 mg eq/kg) was acid-labile clopyralid.

Straw:

A resolved clopyralid peak accounted for 20.4 %TRR, 0.436 mg eq/kg in the aqueous acetonitrile extracts (E1-2). A large, less polar, unresolved region, accounting for 30.1 %TRR, 0.645 mg eq/kg was also detected. Upon mild base treatment, primarily clopyralid was detected (98.7 %ROI) and therefore is identified as base-labile clopyralid.

Base-labile clopyralid was the major component detected in the organic base extracts (E3 and E4), accounting for 34.6 %TRR, 0.737 mg eq/kg.

Base-labile clopyralid was the only component detected in the mild base further extract (FE1), accounting for 4.5 %TRR, 0.097 mg eq/kg and acid-labile clopyralid was detected in the combined acidic further extracts (FE2-3) 3.0 %TRR, 0.063 mg eq/kg, respectively.

Overall, 92.8% of the radioactivity was extractable, and 92.6 %TRR (1.978 mg eq/kg) was shown to be clopyralid. Of this, 20.4 %TRR (0.436 mg eq/kg) was “free” clopyralid, 69.2 %TRR (1.479 mg eq/kg) was base-labile clopyralid and 3.0 %TRR (0.063 mg eq/kg) was acid-labile clopyralid.

Grain:

A resolved clopyralid peak accounted for 27.5 %TRR, 0.311 mg eq/kg in the aqueous acetonitrile extracts (E1-2). A large, less polar, unresolved region, accounting for 45.1 %TRR, 0.509 mg eq/kg was also detected. Upon mild base treatment, primarily clopyralid was detected (98.2 %ROI) and therefore is identified as base-labile clopyralid.

Base-labile clopyralid was the only component detected in the organic base extracts, accounting for 20.4 %TRR, 0.229 mg eq/kg.

Base-labile clopyralid was the only component detected in the mild base further extract (FE1) accounting for 2.0 %TRR, 0.022 mg eq/kg. As the mild acidic further extract (FE2) remained basic, the clopyralid released in this extraction is determined to be base-labile clopyralid, which accounted for 1.4 %TRR, 0.016 mg eq/kg.

Overall, 96.4% of the radioactivity was extractable, and 96.4 %TRR (1.087 mg eq/kg) was shown to be clopyralid. Of this, 27.5 %TRR (0.311 mg eq/kg) was “free” clopyralid and 68.9 %TRR (0.776 mg eq/kg) was base-labile clopyralid.

Table 23: Clopyralid Levels in Wheat Following a Foliar Treatment with [¹⁴C]-Clopyralid

	Wheat							
	Forage		Hay		Straw		Grain	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR ¹	100	1.879	100.0	3.171	100.0	2.134	100.0	1.128
Total Extractable	98.1	1.845	97.1	3.611	92.8	1.980	96.4	1.087
Total Analysed by HPLC ²	98.1	1.845	97.1	3.611	92.8	1.980	96.4	1.087
Clopyralid ³	66.0	1.240	15.4	0.571	20.4	0.436	27.5	0.311
Base-Labile Clopyralid ⁴	32.0	0.603	79.8	2.965	69.2	1.479	68.9 ⁶	0.776 ⁶
Acid-Labile Clopyralid ⁵	-	-	2.0	0.072	3.0	0.063	- ⁶	- ⁶
Total Identified as Clopyralid (HPLC/MS)	98.0	1.843	97.2	3.608	92.6	1.978	96.4	1.087
Total Characterised by HPLC	-	-	<0.1	0.002	0.1	0.002	-	-
Unextracted	1.8	0.035	2.9	0.107	7.2	0.154	3.7	0.041
Accountability ⁷	99.9		100.0		100.0		100.1	

¹ = TRR determined by combustion

² = Total extracts that were prepared and analysed by HPLC does not include any losses/gains during processing

³ = Total free clopyralid detected in aqueous acetonitrile extracts.

⁴ = Sum of 'unresolved region' in aqueous acetonitrile extracts and clopyralid in base extracts (E3, E4 and FE1). Extensive investigation work was conducted on the 'unresolved region'. Mild base treatment resulted in >97% clopyralid. Further experiments were conducted and the conclusion of these experiments is that this region is base-labile clopyralid.

⁵ = Total clopyralid detected in acidic further extracts.

⁶ = As the mild acidic further extract (FE2) was basic, the clopyralid detected in this extract is base-labile clopyralid.

⁷ = Accountability = %TRR extractable + %TRR unextractable

Metabolite Identification

Confirmation of Clopyralid

The identity of clopyralid was confirmed in a grain aqueous acetonitrile SPE sample and in a straw methanolic base SPE sample by qualitative mass spectrometry using radio LC-MS and LC-MS/MS. Identification was confirmed by expected isotope pattern and comparison of retention time, accurate mass measurement and fragmentation pathway with an authentic reference standard for clopyralid.

Clopyralid was also confirmed in a bulk treated grain aqueous acetonitrile extract and a fortified grain sample using MRM-MS.

Characterisation of Unresolved Region

A large unresolved region, less polar than clopyralid, was detected in each concentrated aqueous acetonitrile extract, eluting from 14 – 22.5 minutes and accounting for 21.0 - 63.3 %TRR (0.394 – 2.351 mg eq/kg). Multiple experiments were conducted to explore the nature and identity of this region.

Base Treatment of Treated Samples:

Initially the aqueous acetonitrile extracts from forage, hay, straw and grain were treated with mild base (methanol:10 N NaOH, 100:1, v/v, ambient, 24h), and subsequent HPLC analysis resulted in >97.7 %ROI clopyralid. The near-complete conversion of the unresolved region after a mild base treatment may be due to hydrolysis and/or a result of the change in sample pH. Therefore, additional investigations into the effect of base on the sample extracts were conducted.

pH Adjustment of Treated Samples:

To investigate the effect of pH on wheat grain extract, the concentrated aqueous acetonitrile extract from the treated grain sample was adjusted to pH 11 with the addition of 5% ammonium hydroxide solution (2:1 ,v/v sample:base ratio). HPLC analysis of the resulting sample was conducted after 1, 2, 4 and 24 hours. Analysis showed that after 1 hour, the unresolved region was not detectable. The region was not seen in any of the subsequent analysis.

However, a second peak eluting at 13.8 minutes was detected in analysis of pH adjusted samples after 1, 2 and 4 hours. At the 24-hour analysis, the second peak was not well resolved but was still present.

To investigate the second peak, an aliquot of the clopyralid standard was analysed by HPLC following addition of 5% ammonium hydroxide. This analysis also showed two peaks, which suggests that the second peak is clopyralid in an altered ionization form. A split clopyralid peak was also detected during MS analysis. The peaks at 10.75 and 11.50 minutes can be tentatively identified as clopyralid, indicating a split peak. The sample was re-prepared for MS and no split peak was detected in that analysis, which was subsequently used for confirmation of clopyralid.

The complete removal of the unresolved region seen through this analysis demonstrates that a significant amount of clopyralid is rapidly released by adjustment of the sample pH. This indicates that the unresolved

region is a result of weak binding interactions between clopyralid and the endogenous plant matrix, which are disrupted by treatment with mild base. The use of mild base and the speed of conversion to clopyralid suggests that clopyralid is not covalently bonded to the plant matrix (*e.g.*, sugars, amino acids etc) but binds *via* weak interactions (*e.g.*, electrostatic) which can be easily disrupted. This was further confirmed by the fortification of control tissue.

Fortification of Control Tissue:

To investigate this hypothesis further, homogenised control grain tissue was fortified with [¹⁴C]-clopyralid. The fortified sample was extracted, concentrated and analysed by HPLC following the same procedure as the treated grain sample. The resulting HPLC analysis showed an unresolved region accounting for 11.1% ROI. As the control tissue had been homogenised and stored frozen it is highly improbable that ‘conjugates’ were formed during the fortification experiment. Therefore, this supports the results from the pH adjustment experiments which show that the unresolved region is likely a result of interactions between clopyralid and endogenous plant matrix. Furthermore, this supports the conclusion that clopyralid likewise interacted with endogenous plant matrix in the treated wheat matrices, especially hay, straw, and grain.

SPE of Treated Samples:

Attempts were made to remove endogenous material from the concentrated aqueous acetonitrile extract from treated wheat grain using SPE. Removal of this endogenous material through ion exchange SPE resulted in the removal of the unresolved region. This further supports the idea that the unresolved region is linked to the endogenous matrix present in the sample. The reduction in the unresolved region could also be due to the use of mild base (ammonium hydroxide) during the SPE process. Use of mild base (ammonium hydroxide) to change sample pH also reduces the unresolved region. Overall, this SPE procedure shows that the unresolved region can be reduced to clopyralid with only very light manipulation of the sample.

LC-MS:

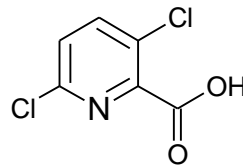
Initial MS analysis on the treated wheat grain sample, performed on a LUMOS Mass Spectrometer, did not detect clopyralid in the unresolved region. Interference from endogenous plant matrix present in the sample caused a number of effects, including suppression of the ionisation and very low intensity peaks. Limited injection volumes restricted the amount of mass injected on the MS, further reducing the intensity of peaks. Attempts to generate a cleaner sample for LC-MS analysis through SPE resulted in the loss of the unresolved region. To further investigate this initial MS analysis, additional MS work was conducted using an API4000 Mass Spectrometer. Analysis of the treated wheat grain and fortified wheat grain sample did not detect clopyralid in the unresolved region. Due to the low injection volumes used during this analysis, not enough mass was injected to detect clopyralid in the unresolved region.

Conclusion of Experiments on Unresolved Region:

Although experiments on this apolar unresolved region did not result in a conclusive identification, it can be strongly hypothesised that this region is the result of chromatographic effects of clopyralid weakly binding and interacting with endogenous material. Conversion to clopyralid is rapid under mild base conditions. Furthermore, clopyralid test material created a similar unresolved region when fortified onto control grain. Therefore, this region can be identified as base-labile clopyralid.

The structure, chemical name and the common name used in the study report for clopyralid are shown below.

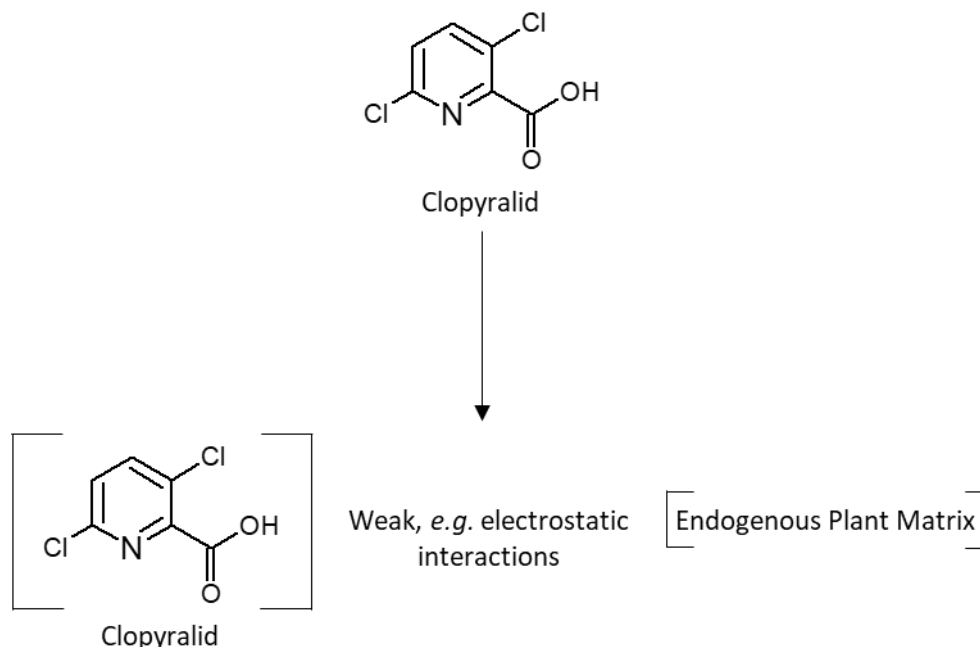
Table 24: Identification of compounds from metabolism study

Common name/code number	Chemical name	Chemical structure
Clopyralid	3,6-Dichloropicolinic Acid	

Metabolic Pathway

A metabolic pathway indicating interactions with endogenous plant material is presented below. No metabolites of clopyralid were detected in this study.

Figure 3: Proposed Metabolic Pathway for Clopyralid in Wheat



Storage Stability

Storage stability data for samples and extracts is provided below. All samples and extracts were stored in a freezer set to maintain -20 °C when not in use. Initial extractions of all samples occurred within 27 days after harvest, while HPLC analyses of initial extracts occurred typically within 49 days of harvest.

Table 25: Summary of storage stability

Matrix	Storage temp. (°C)	Harvest date	Combustion analysis date	Initial extraction date	Initial chromatography date
Forage	-20 °C	26 Jul 2019	05 Aug 2019 (10 DAH)	22 Aug 2019 (27 DAH)	29 Aug 2019 (34 DAH)
Hay	-20 °C	15 Aug 2019	27 Aug 2019 (12 DAH)	03 Sep 2019 (19 DAH)	03 Oct 2019 (49 DAH)
Straw	-20 °C	23 Oct 2019	25 Oct 2019 (2 DAH)	31 Oct 2019 (8 DAH)	07 Nov 2019 (15 DAH)
Grain	-20 °C	23 Oct 2019	25 Oct 2019 (2 DAH)	31 Oct 2019 (8 DAH)	07 Nov 2019 (15 DAH)

DAH = days after harvest

CONCLUSION

The wheat metabolism study is considered scientifically acceptable.

Following a single 125.87 g a.e./ha (104.9% of target) application of [¹⁴C]-clopyralid to wheat, TRR levels in forage, hay, straw and grain ranged from 1.879 – 3.717 mg eq/kg.

For all tissues, accountability, between combustion and extraction data, was 95.4 – 107.6%. When normalised with respect to accountability, aqueous acetonitrile recovered 50.6 – 90.5 %TRR and methanolic base extracts released a further 5.0 – 34.7 %TRR. Further extractions were conducted on all samples using mild aqueous base, mild and stronger acid, which released a further 2.6 – 7.5 %TRR. Non-extractable residues were 1.8 – 7.2 %TRR.

The aqueous acetonitrile contained a resolved clopyralid peak (15.4 – 66.0% TRR, 0.311 – 1.240 mg eq/kg) and a less polar, unresolved region (21.0 – 63.3% TRR, 0.394 – 2.351 mg eq/kg).

Upon mild base treatment (similar to analytical method 120610 using methanol:10 N NaOH (100:1, v/v), overnight), primarily clopyralid was detected (*ca.* 98%). The methanol base extract, mild aqueous base and acid further extracts contained only clopyralid (*ca.* 99%). No polar clopyralid was detected in any sample.

Experiments on the apolar unresolved region demonstrated that it was completely converted to clopyralid within an hour of treatment with 5% ammonium hydroxide solution (2:1,v/v, sample:base ratio). Any sort of light manipulation of the sample (SPE, changing the pH etc.) to remove endogenous materials resulted in the elimination of this region, forming clopyralid. This indicates that the unresolved region is a result of weak binding interactions (*e.g.* electrostatic) between clopyralid and the endogenous plant matrix, which are disrupted by treatment with mild base or removal of endogenous material. The presence of the apolar region in the control sample fortified with clopyralid further adds to the theory that this region is a result of interactions between clopyralid and endogenous materials present in the sample. Overall, it can be strongly hypothesised that this region is the result of chromatographic effects of clopyralid weakly binding/interacting with endogenous material. Therefore, this region can be identified as base-labile clopyralid.

Greater than 90% of the extractable residue was identified as clopyralid. In total, clopyralid accounted for 98.0 % TRR (1.843 mg eq/kg) in forage, 97.2 % TRR (3.608 mg eq/kg) in hay, 92.6 % TRR (1.978 mg eq/kg) in straw and 96.4 % TRR (1.087 mg eq/kg) in grain.

A 2.1.2.1.1.2 Study 2 – Oilseed rape

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the CMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>The study was performed according to OECD guideline and GLP requirements. In total, clopyralid (free and base-labile) accounted for >70% of the TRR in all samples, including seed, forage, trash, and pollen/anthems.</i></p> <p><i>However, the study should be evaluated at EU level.</i></p> <p><u>Remark:</u></p> <p>In our opinion, this study should be evaluated at the EU level, not national level. The study was deemed acceptable and supported the current residue definitions for enforcement and risk assessment.</p>
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Reference: KCA 6.2.1/02

Report: Mackenzie.; 2021; The Metabolism of [¹⁴C]-Clopyralid in Oilseed Rape; Charles River Laboratories Edinburg Ltd., Tranent, UK; Lab Study No. 231101; DAS Study No. 200928; 22 September 2021; Unpublished

Guidelines: OECD 501, OPPTS 860.1300

Deviations: none

GLP: Yes

BACKGROUND INFORMATION

Clopyralid (3,6-dichloropyridine-2-carboxylic acid) is a selective herbicide developed for use on a variety of agricultural crops including oilseed rape.

A previous study had been conducted to investigate the metabolism of clopyralid in oilseed rape. In this study, two regions of unidentified radioactivity were detected in trash and seed. A polar component re-formed clopyralid under modified HPLC conditions or acid hydrolysis; evidence indicated this was an artefact of the HPLC system and it was concluded to represent unchanged clopyralid. An apolar component

was entirely converted to clopyralid by hydrolysis and was concluded to be either unchanged clopyralid or clopyralid in conjugated form. The amount of co-injected organic solvent and potentially matrix effects during chromatography appeared to influence the amount of each component. Therefore, the Sponsor initiated an additional oilseed rape study to further investigate the nature of these unknowns.

MATERIALS AND METHODS

Test Item(s)

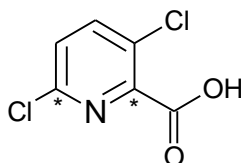
Non-radiolabelled test item #1

Test item (Common name): Clopyralid
Purity: 99.9%
Description (physical state): Solid
Lot/batch no.: TSN301194 (YC2-106153-68)

Radiolabelled test item #1

Test item (Common name): [¹⁴C]-Clopyralid
Name: 3,6-Dichloropicolinic acid-2,6-¹⁴C, X755015

Structural Formula:
Position of labelling (*)



Lot/batch no.: INV306082
Radiochemical purity: 99.0%
Specific radioactivity: 35.0 mCi/mmol

Methods

Test Site Information

Testing environment: F - (outdoors) from application until mature harvest, except one plot moved into glasshouse (25 May 2020) for pollen collection - G

Container description: Plastic, internal dimensions: 0.755 x 0.555 m, 6 used (5 treated and 1 control)

Soil type: Sandy Loam (UK and USDA)

Soil characteristics: 2.16% OM
pH 7.2 (in water)
CEC 8.9 meq/100 g
63% Sand (USDA)
26% Silt (USDA)
11% Clay (USDA)

Any adverse weather conditions: No

Any adverse insect or disease problems: No

Study Use Pattern

Application method: Foliar
Formulation type: Monoethanolamine salt, GF-1966
Application rate: 200 g ae/ha (target), actual 199.09 g ae/ha

Number of applications:	1
Timing of applications	19 May 2020, BBCH 30
PHI (Days after Treatment):	23 (forage), 9-31 (pollen/anthers), 94 (mature seed and trash)

Test System

Organism (<i>Species</i>):	Spring oilseed rape (<i>Brassica napus</i>)
Variety:	Click
Crop group:	Oilseed
Growth stage at application:	BBCH 30
Harvested RAC:	Early forage, seeds
Other harvested commodities:	Pollen/anthers, trash
Growth stage at harvest:	Pollen/anthers: BBCH 60-69 (28 May–19 Jun 2020), Early forage: BBCH 66 (11 Jun 2020), Mature seed and trash: BBCH 89 (21 Aug 2020)
Harvesting procedure:	Plants were randomly sampled for forage harvest (approximately 1/3 from each of the containers except the container dedicated to pollen/anther harvest), cut at soil surface. Remaining plants were sampled at maturity. Pods were removed and seeds separated from pods by hand. Trash was cut at soil surface and combined with pods. Forage, trash, and seeds were stored frozen (-20°C). Anthers (containing pollen) were separated from flowers using tweezers every 1-5 days for <i>ca.</i> 3 weeks and stored at 4°C between collections.

Sample Handling, Preparation, and TRR Determination

Each frozen plant fraction (forage, trash and seeds) was homogenised with excess carbon dioxide chips using a Waring PB20 or a Hobart VCB61 blender. The carbon dioxide was then allowed to sublime while frozen prior to removal of sub-samples for combustion. Sub-samples (five each, *ca.* 0.3 g) of the milled tissue were analysed by oxidative combustion to determine the total radioactive residues in the sample. As part of the extraction sequence, the anther samples were manually separated into anther capsules and pollen using a scalpel blade and washed with water to fully separate the pollen from the anther capsules. This was conducted on the same day following the final anther/pollen harvest and the processed samples stored in a freezer set to maintain -20°C.

Extraction of Sample Residues – Forage, Trash and Seeds

Aqueous Acetonitrile Extraction (E1-2)

Samples of oilseed rape forage, trash and seed were extracted as follows. Approximately 25 g (forage or trash) or 15 g (seeds repeat) of homogenised tissue was extracted with 1:1 acetonitrile:Milli-Q® water (v/v) at a solvent:sample ratio of 5:1 (mL:g) for forage and seed, and a ratio of 10:1 for trash (included a *ca.* 30 minutes soak prior to blending). The mixture was blended using a Silverson L5T TopDrive homogeniser for approximately 5 minutes at room temperature. The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and Extract 1 decanted. The extraction was repeated once more at a solvent:sample ratio of 2.5:1 (mL:g) for forage and seed and 5:1 (mL:g) for trash, and Extract 2 decanted. Extract 1 and Extract 2 were pooled and the volume measured. Where required, extracts were filtered through a 100 µm nylon cell strainer when decanting after centrifuging. Any solid material captured during filtration was returned to the extraction pot. Aliquots of each combined extract (E1-E2) were analyzed by liquid scintillation counting.

For forage aqueous acetonitrile extracts E1-E2 only, where recovery was slightly lower, a flask wash of the associated glassware from concentration including round bottom flask, volumetric flask and centrifuge tube was conducted using acetonitrile. The flask wash was centrifuged (*ca.* 13,000 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) prior to analysis by LSC and direct analysis by HPLC.

Methanolic Base Extraction (E3)

Following neutral organic solvent extraction, the samples were subjected to base extraction. The entire remaining residue sample following neutral organic extraction was homogenised in the presence of methanol:10 N aqueous sodium hydroxide (100:1, v/v) for *ca.* 1 minute at room temperature using a Silverson L5T TopDrive homogeniser before being shaken on a reciprocal shaker for *ca.* 1 hour. Forage and seed samples were extracted at a solvent:sample ratio of 2.5:1 (mL:g) and trash samples were extracted at a solvent:sample ratio of 5:1 (mL:g). The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and the total extract volume measured. Where required, extracts were filtered through a 100 µm nylon cell strainer when decanting after centrifuging. Any solid material captured during filtration was returned to the extraction pot. Aliquots of Extract 3 were analysed by liquid scintillation.

Methanolic Base Extraction (E4-E6) of Seed Only

For seed samples, the entire remaining residue following extract 3 was extracted with methanolic base (E4-6) as follows. For extract 4 (E4) and extract 5 (E5), the remaining seed residue was homogenised in the presence of methanol:10 N sodium hydroxide (100:1, v/v) for *ca.* 1 minute at room temperature, using a Silverson L5T TopDrive homogeniser, before being shaken on a reciprocal shaker for either *ca.* 1 hour (E4) or overnight (E5). Extractions were performed at a solvent:sample ratio of 4:1(mL:g) and 2.5:1(mL:g) for E4 and E5, respectively. For each extraction, solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and the extract volume measured prior to analysis by liquid scintillation counting.

Following E5, the remaining residue was homogenised in the presence of 0.5 N aqueous sodium hydroxide at a solvent:sample ratio of 2.5:1 (mL:g) for *ca.* 5 minutes at room temperature using a Silverson L5T TopDrive homogeniser before being placed in a shaking water bath set to maintain at *ca.* 50°C for *ca.* 4 hours. The sample was allowed to cool prior to separating the solid and liquid phases by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40). The remaining solid residue was then rinsed with methanol at a solvent:sample ratio of 1:1 (mL:g), shaken by hand for 30 seconds and the solid and liquid phases separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40). The methanol rinse was repeated a second time and both rinses and 0.5 N NaOH extract pooled forming Extract E6. The total extract volume was measured prior to analysis by liquid scintillation counting.

Further Extraction 0.1N Sodium Hydroxide (FE1) of Forage and Trash Only

Following the initial extractions (E3) and initial combustion of the remaining debris the remaining residue from forage and trash samples was further extracted in the presence of 0.1 N NaOH at a solvent:sample ratio of 10:1 (mL:g). Extracts were placed in a water bath set to maintain at *ca.* 50°C for *ca.* 4 hours. The extracts were allowed to cool prior to separating the solid and liquid phases by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40). Following Further extract 1 the tissue sample was rinsed with Milli-Q water (5:1 mL/g), and the resulting rinse shaken by hand prior to separation by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40). The water rinse was combined with the 0.1 N NaOH extract. The total extract volume was recorded and aliquots of Further Extract 1 were analysed by liquid scintillation.

Further Extraction 0.5N Sodium Hydroxide (FE1) of Seed Only

Following the extraction of seeds through E6, the remaining residue was homogenised in the presence of 0.5 N sodium hydroxide at a solvent:sample ratio of 8:1 (mL:g) for *ca.* 5 minutes at room temperature using a silverson SL2 TopDrive homogeniser before being placed in a shaking water bath set to maintain at *ca.* 50°C for *ca.* 4 hours. The sample was allowed to cool prior to separating the solid and liquid phases by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40). The remaining solid residue was then rinsed with methanol at a solvent:sample ratio of 1:1 (mL:g), shaken by hand for 30 seconds and the solid and liquid phases separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40). The methanol rinse was repeated a second time. Both rinses and 0.5 N NaOH extract were pooled and total extract volume measured. Aliquots of Further Extract 1 were analysed by liquid scintillation counting.

Figure 4: Flow diagram for the extraction of Forage and Trash

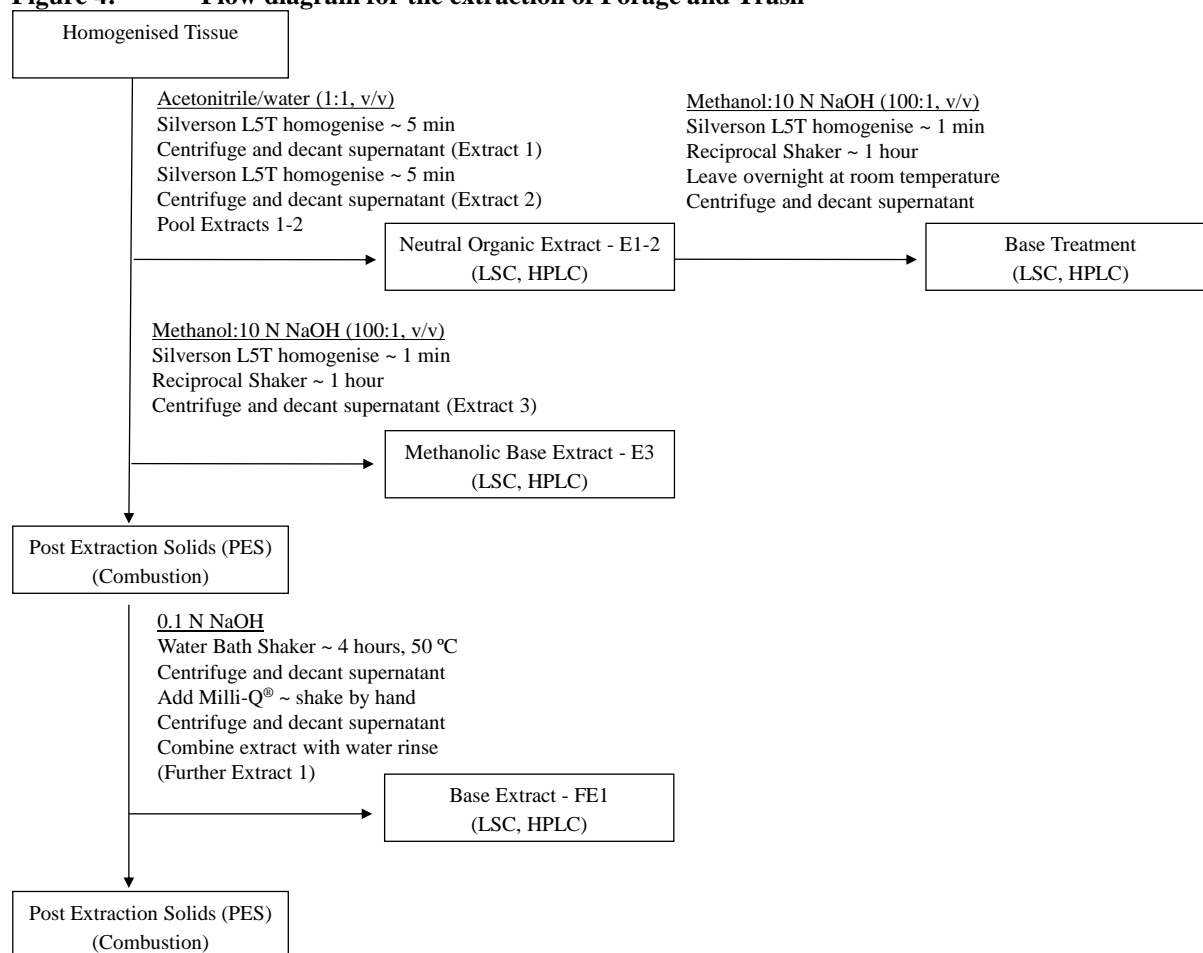
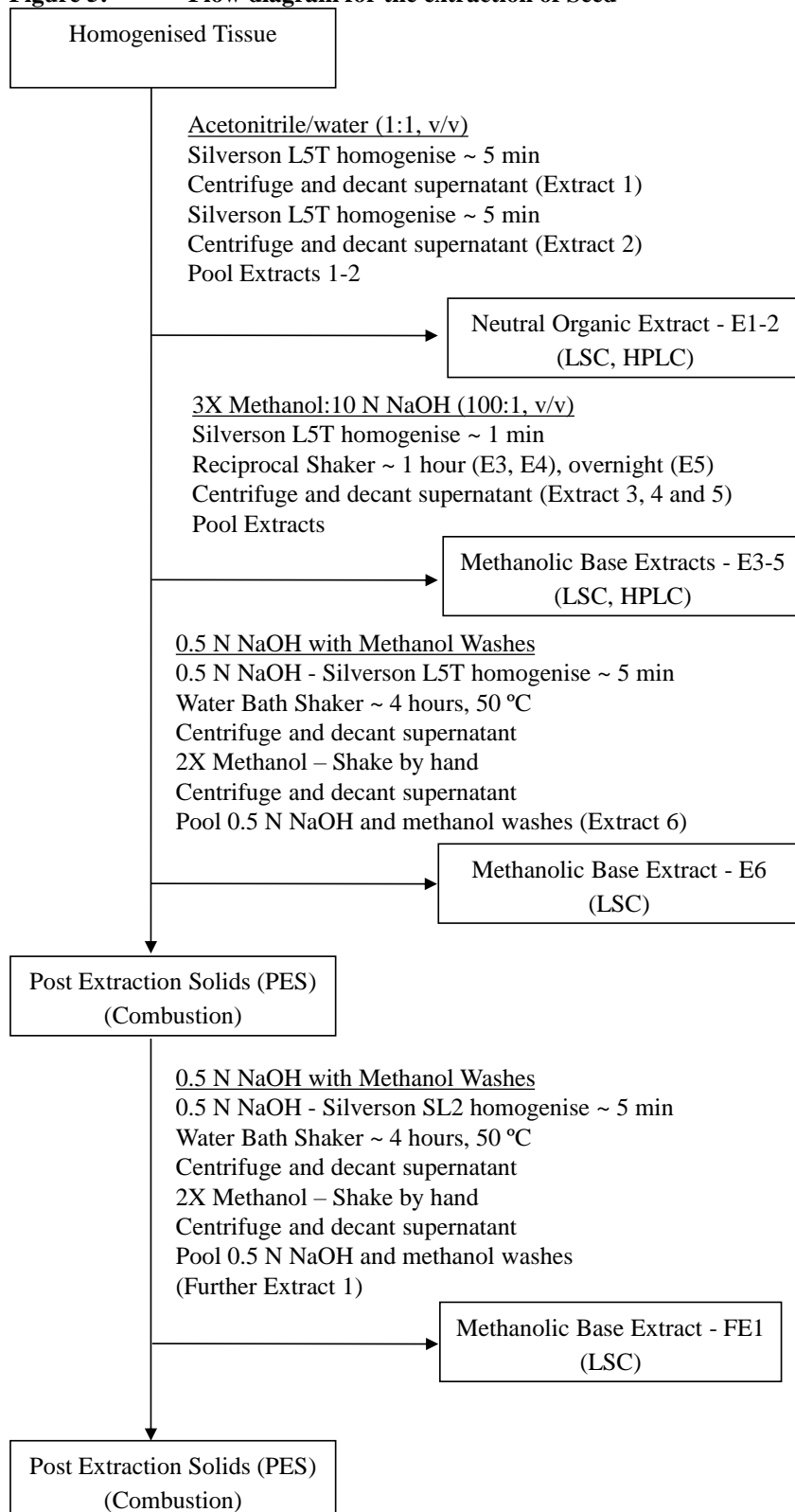


Figure 5: Flow diagram for the extraction of Seed



Extraction of Sample Residues – Anthers/Pollen

Anther/Pollen Processing

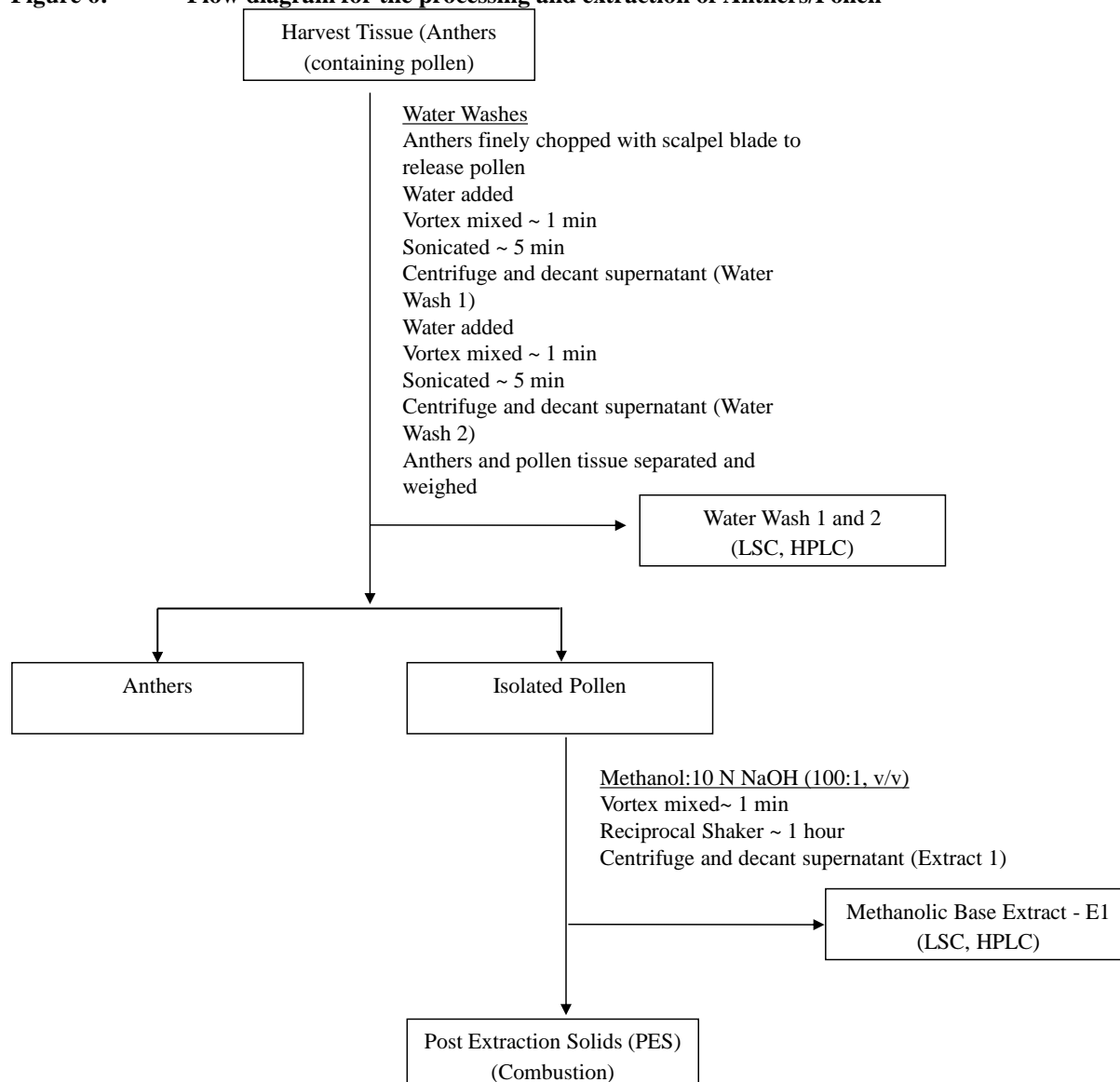
Once all of the sample collection was complete (after approximately three weeks of sampling every 1-5 days), the anthers (including pollen) were finely chopped with a scalpel blade to open up the anther capsules and release the pollen. Water was used to wash the pollen away from the anther capsules; Milli-Q® water

was added to completely cover the sample, still containing the anthers with the pollen, and the mixture was then agitated using a vortex mixer for *ca.* 1 minute and sonicated for *ca.* 5 minutes. The pollen suspension was decanted from the anthers then transferred to a centrifuge tube and centrifuged (*ca.* 13000 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) to separate the pollen from the water wash. The volume of the water wash (Water Wash 1) was measured and transferred to a suitable container. The remaining anthers sample was washed a second time following the same procedure to suspend any remaining pollen. The volume of the water wash (Water Wash 2) was measured and transferred to a suitable container. Water Wash 1 and Water Wash 2 were analysed by LSC. Following the water washes, the individual pollen and anthers fractions were weighed.

Pollen Extraction

Once isolated from the anthers, pollen was extracted with methanol:10N NaOH (100:1 v/v) at a solvent:sample ratio of 25:1 (mL:g). Note that the mass of isolated pollen allowed for a single extraction, not a sequence, and therefore the MOR analytical method solvent was selected. The mixture was vortex mixed for *ca.* 1 minute and then placed on a reciprocating shaker for *ca.* 1 hour at room temperature. The solid and liquid phases were separated by centrifugation (*ca.* 13000 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and the extract decanted and volume measured. The pollen extract was analysed by liquid scintillation counting. The pollen post extraction solid was combusted.

Figure 6: Flow diagram for the processing and extraction of Anthers/Pollen



Analytical Method Extraction (Radiovalidation)

To compare the extractability of the analytical method to the metabolism method, aliquots of forage, trash and seeds homogenised tissue were subjected to base treatment, using methodology from the field trial analytical method (Dow 120610). For each commodity 100 mL of methanol:10 N NaOH (100:1, v/v) was added to *ca.* 5 g of homogenised tissue. Samples were homogenised for *ca.* 1 minute using a Silverson L5T TopDrive homogeniser, shaken for *ca.* 1 hour on a reciprocal shaker and left at ambient temperature overnight. Samples were centrifuged and the supernatants analysed by LSC. One mL (forage) and 5 mL (trash) of extract was neutralized with hydrochloric acid solution prior to concentration under a gentle stream of nitrogen. Samples were reconstituted in 1 mL acetonitrile:water (3:7, v/v) and analysed by LSC prior to HPLC analysis.

For the seeds extracts only, 30 mL of the resulting extract was split between two 50 mL centrifuge tubes (15 mL in each tube) and 3 mL of 1 N NaOH added to each. The samples were then placed under a gentle stream of nitrogen to remove the methanol (*ca.* 3 mL volume remained). The samples were made to 15 mL each with 1 N NaOH. The samples were then partitioned with an equivalent volume of DCM (15 mL), then vortex mixed and sonicated thoroughly prior to centrifuging at *ca.* 3000 rpm for *ca.* 2 minutes. The aqueous layer was removed and both the aqueous and DCM layers pooled prior to analysis by LSC. Five mL of the aqueous layer was concentrated to *ca.* 0.5 mL then made to 1 mL with acetonitrile:water (3:7 v/v). The sample was neutralized with concentrated hydrochloric acid. The neutralized sample was analysed by LSC prior to HPLC analysis.

Characterisation of Apolar Region

Neutral Organic Extract Base Treatment

To further characterise the neutral organic extracts (E1-2) of forage, trash, and seeds, 10 mL aliquots of pooled extract 1-2 were subjected to base treatment, using methodology similar to the field trial analytical method (Dow 120610). The neutral solvent was evaporated under nitrogen, at ambient temperature, until dryness and the sample reconstituted in 10 mL of methanol:10 N NaOH (100:1, v/v). Samples were homogenised for *ca.* 1 minute using a Silverson SL2 TopDrive homogeniser, shaken for *ca.* 1 hour on a reciprocal shaker and left at ambient temperature overnight. Samples were centrifuged (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and the supernatants analysed by LSC. For forage only, where recoveries were low, a wash of the pellet after centrifuging was conducted using 5 mL water:acetonitrile, 1:1 (v/v). The wash was centrifuged (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) prior to analysis by LSC. Base treatment samples were combined with the respective wash prior to concentration and HPLC analysis.

A separate control sample consisting of an aliquot of forage pooled extract 1-2 was processed using the same procedure with methanol:water (100:1, v/v), containing no sodium hydroxide to confirm that any changes during processing resulted from the presence of base in the samples.

pH Adjustment of Treated Samples

To investigate the effects of pH on oilseed rape, the concentrated aqueous acetonitrile extracts (E1-2) of forage were subjected to a number of experiments:

1. An aliquot of concentrated E1-2 was pH adjusted with the addition of 5% ammonium hydroxide solution (4:1, v/v sample:base ratio). The aliquot at each time point, 0.5 hours and 24 h, was acidified to pH 1 immediately prior to injection on the HPLC to aid chromatography.
2. To investigate the second peak, a number of further investigations were conducted. The solvent ratio in the initial pH adjustment experiments described above was acetonitrile:water (3:7, v/v). To investigate possible effects from the organic solvent, two aliquots of forage concentrated E1-2 had the solvent removed under a gentle stream of nitrogen. One aliquot was reconstituted in water, containing a small amount of 5% ammonium hydroxide solution (17:1, v/v sample:base ratio), and one aliquot was reconstituted in 5% ammonium hydroxide solution. After 1 hour, the ammonium hydroxide aliquot was acidified to pH 1 and the water aliquot was neutralized to pH 7 immediately prior to analysis.
3. To investigate the presence of an ammonium adduct, an aliquot of forage concentrated E1-2 was added to two vials. An equal amount of ¹⁴C clopyralid (dpm) was spiked into one of the vials, and both aliquots had the solvent removed under a gentle stream of nitrogen prior to reconstitution in

an equivalent volume of 5% ammonium hydroxide solution. After 1 hour, both samples were acidified to pH 1 immediately prior to injection.

4. The results from the organic solvent experiments detailed above indicated that there may also be an impact from the pH during chromatography. To investigate this, two aliquots of forage concentrated E1-2 had the solvent removed under a gentle stream of nitrogen prior to reconstitution in an equivalent volume of 5% ammonium hydroxide solution. After 1 hour, one aliquot was neutralized to pH 7 and one aliquot was acidified to pH 1 immediately prior to analysis.
5. Based on the structure of clopyralid, it was postulated that it may be an effective chelator in biological systems, similar to picolinic acid. Structural analogues of clopyralid have been shown to weakly chelate to heavy metal ions in matrix. To investigate this, it was suggested that Na⁺ ions may disrupt the chelation similar to the caustic used in the base treatment methods using methanol:10 N NaOH (100:1 v/v). Two aliquots of forage concentrated E1-2 had the solvent removed under a gentle stream of nitrogen. One aliquot was reconstituted in an equivalent volume of 5% ammonium hydroxide solution. Both aliquots had an equivalent volume of 1 M NaCl added to generate an excess of Na⁺ ions in the sample. The aliquot without 5% ammonium hydroxide acted as a control. After 1 hour, both aliquots were acidified to pH 1 immediately prior to analysis.

Control Tissue Fortification

Control oilseed rape forage tissue (*ca.* 5 or 10 g) was fortified with ¹⁴C clopyralid (0.38 or 2.27 µCi) and subjected to aqueous acetonitrile extraction (E1-E2). The extract was concentrated in preparation for HPLC. The same experiment was attempted twice on control oilseed rape forage tissue under this study.

Extraction of Oilseed Rape Forage for Apolar MS

To assist with metabolite identification analysis of the apolar region, an aliquot of oilseed rape forage was extracted as follows. Approximately 15 g of homogenised forage tissue was extracted with 1:1 acetonitrile:Milli-Q[®] water (v/v) at a solvent:sample ratio of 3.6:1 (mL:g). The mixture was blended using a Silverson L5T TopDrive homogeniser for approximately 5 minutes at room temperature. The solid and liquid phases were separated by centrifugation (*ca.* 3500 rpm, *ca.* 10 minutes, Thermo Heraeus Megafuge 40) and Extract 1 decanted. Aliquots of the extract were analyzed by liquid scintillation counting. Immediately following extraction, the extract was analysed by LC-MS/MS. Although initial LC-MS analysis was conducted on the day of extraction, further investigations were conducted on subsequent dates, as appropriate.

Concentration, Molecular Weight Filtration, and Methylation of the Neutral Organic Extract (Including the Apolar Region)

Concentration of the oilseed rape forage neutral organic extract for MS was conducted using a nitrogen stream to avoid any heating of the sample which was suspected to cause degradation of the apolar region back to clopyralid parent which had been observed on previous studies. The bulk extract (38 mL) was concentrated to approximately 3 mL and analysed by LC-MS.

The concentrated OSR Forage extract sample (1.8 mL) was split between 6 x Amicon Ultracel 3K Centrifugal Molecular weight filtration tubes and centrifuged at 13,000 rpm for 2 x 15 minute periods. The supernatant was removed as was the remaining liquid portion on the filter. LSC counting indicated that 47% of the radioactivity was present in the supernatant and 53% present in the unfiltered liquid. LC-MS analysis was conducted on both portions.

An aliquot of molecular weight filtered (3KDa Filter) OSR extract sample was taken forward for methylation using a TMS-diazomethane / methanol methylation procedure. An aliquot, 0.3 mL, of the concentrated OSR extract sample that had been subjected to centrifugal molecular weight filtration was diluted with 1.0 mL of methanol and 1.5 mL of trimethylsilyl diazomethane added. The reaction mixture was stirred at ambient temperature for 1h and the reaction quenched by the addition of 0.2 mL of acetic acid. The reaction mixture was concentrated to dryness and dissolved in 0.5 mL of Milli-Q water. LC-MS analysis was conducted on the reaction mixture.

Metabolite Isolation and Identification

Non-radiolabeled clopyralid was used as a reference standard for chromatographical comparison. HPLC retention time comparisons were made between this reference standard and the radioactive peaks in the chromatograms in order to provide initial tentative identifications.

Based on the initial HPLC analyses, clopyralid fractions were targeted for structure confirmation. This was accomplished using neutral organic extracts (E1-2) both before and after base treatment in oilseed rape forage and in neutral organic extracts (E1-2) before base treatment oilseed rape seeds. These samples were submitted for radio LC-MS and LC-MS/MS analyses.

Analytical Methodology

Total ^{14}C measurement

Oxidative Combustion:

Approximately 0.05-0.3 g sub-samples (3-5 replicates of forage, trash and seeds samples; and one aliquot of pollen PES) of each sample were weighed into Combustococones[®] and combusted using a PerkinElmer Tri-Carb 307 Automatic Sample Oxidiser. The resultant $^{14}\text{CO}_2$ was absorbed in Carbo-Sorb[®], mixed automatically with Permafluor[®] scintillation fluid and the radioactivity determined by LSC.

The efficiency of the oxidiser was determined daily, and at regular intervals throughout each batch of analyses. Carbon-14 quality control standards were combusted at the beginning and at regular intervals throughout each batch of analyses. Combustion efficiencies were in excess of 98.3%. Combustion values were not corrected for the oxidiser efficiencies.

Liquid Scintillation Counting:

The liquid scintillation counters automatically converted the radioactivity counting rate in counts per minute (cpm) to disintegrations per minute (dpm) using an external standard to correct for sample quenching. The instrument was calibrated approximately every three months with a set of ten quenched standards. Each day of use, the instrument was normalized and its performance was checked with respect to background cpm value, unquenched standard cpm value, and quenched standard dpm value for a range of quenched standards. The scintillation counters used were Packard 2100TR Tri-Carb (Packard Instrument Co.). The radioactive content (dpm) of liquid samples was determined by LSC. Triplicate aliquots (0.005 – 5 mL) were taken from each sample and mixed with scintillation cocktail (Aquasafe 500 Plus scintillation cocktail, Zinsser Analytic, Maidenhead, UK).

Sample Concentration - E1-3 (Neutral and Base Treated) and FE1 (Caustic Extracts)

The general sample processing procedure for the aqueous acetonitrile pooled extract 1-2 was to concentrate aliquots by rotary evaporation or under a gentle stream of nitrogen, to a reduced volume or dryness before reconstitution to a known volume in a solvent suitable for HPLC (water:acetonitrile 7:3, v/v). Samples were centrifuged (*ca.* 13,000 rpm for *ca.* 10 minutes) and the supernatant analysed by LSC to monitor recovery and then by HPLC.

Aliquots of forage and trash extract 3 in forage and trash (E3, methanol:10 N NaOH, 100:1, v/v) and pollen extract 1 (E1, methanol:10 N NaOH, 100:1, v/v) were neutralised to *ca.* pH 7 using hydrochloric acid solution before concentration using the same techniques as the neutral organic extracts (E1-2).

Where required, aliquots of base treatment neutral organic extracts E1-2 (methanol:10 N NaOH, 100:1, v/v) and associated control samples (methanol:water, 100:1, v/v) were combined with the respective wash prior to concentration. Base treated samples of E1-2 were also neutralized prior to concentration. Control sample extracts were processed in a similar manner to the neutral organic extracts.

Aliquots of mild base further extracts (FE1) were neutralised to *ca.* pH 7 using hydrochloric acid solution before concentration to dryness under a gentle stream of nitrogen or by rotary evaporation to a reduced volume before reconstitution to a suitable volume with water. Samples were then analysed following a similar process to the neutral organic extracts.

Processing and Concentration of Seeds Methanolic Base Extracts

Oilseed rape seeds extracts E3-5 (methanol:10 N NaOH, 100:1, v/v) were combined in equal proportion (5% of each extract; total volume 5.3 mL) and added to a 15 mL centrifuge tube. One mL of 1 N NaOH was added and the sample placed under a gentle stream of nitrogen to remove the methanol. Once the methanol had been removed and *ca.* 1 mL NaOH remained, the sample was made to 5 mL with 1 N NaOH. The sample was then placed in a shaking water bath set to maintain *ca.* 90 °C for 4 hours. The sample was then allowed to cool prior to partitioning with 5 mL DCM. The sample was vortex mixed and sonicated

thoroughly prior to centrifuging (*ca.* 3,000 rpm for *ca.* 2 minutes). The aqueous layer was removed. A further 5 mL 1 N NaOH was added to the DCM layer and a second partition performed (centrifuging at *ca.* 13,000 rpm for *ca.* 10 minutes). The two aqueous layers were acidified using 5 mL of 1 N HCl per aqueous phase and the pH confirmed to be strongly acidic using pH paper.

Two Waters Oasis HLB SPE cartridges (200 mg, 6 cc) were conditioned with 5 mL methanol followed by 5 mL 1 N HCl. The cartridges were dried under vacuum in the SPE manifold for *ca.* 10 seconds. The acidified samples were loaded onto the cartridges and the loading eluates collected separately. The cartridges were eluted with 1 mL 1 N HCl each and this collected into the loading eluates. The cartridges were then eluted with 4.5 mL acetonitrile:1 N formic acid (15:85, v/v) each, which was collected as a separate fraction. The cartridges were dried under vacuum (*ca.* 15 inches Hg) for 30 minutes to ensure that all the solvent was collected from the cartridge. The cartridges were then eluted with 14 mL DCM each which was collected as a separate fraction.

The DCM eluate was concentrated under a gentle stream of nitrogen to dryness and reconstituted in 1 mL 0.1% formic acid (aq): methanol (90:10, v/v). The concentrated sample was analysed by LSC to monitor recovery and then by HPLC.

A similar procedure was attempted for seed E6 without the hydrolysis for 4 hours at *ca.* 90 °C as E6 had already underwent elevated temperature during extraction.

High performance liquid chromatography (HPLC) for quantitation

HPLC analyses of all sample extracts were accomplished using a Waters SPHERISORB ODS 2 column (150 x 4.6 mm, 5 µm particle size; 1.0 mL/min; UV detection at 280 nm) and a 1 step, non-linear gradient.

Mass spectral analysis (LC/MS) for identification of transformation products

Neutral organic extracts (E1-2) both before and after base treatment in oilseed rape forage and in neutral organic extracts (E1-2) before base treatment oilseed rape seeds were used for confirmation of clopyralid by mass spectral analysis. Mass spectral analysis was also conducted on neutral organic extracts (E1-2) of forage which was extracted for apolar MS analysis.

RESULTS AND DISCUSSION

Results of In-Life Phase

An equal volume of the [¹⁴C]-clopyralid application solution was foliarly applied to each of five treated containers when the plants were at growth stage BBCH 30. Each container received 8.34 mg of the test item equivalent to 199.09 g a.e./ha (99.5% of target). Radiochemical purity and stability of the formulated application solution pre- and post-application were ≥98.7%, indicating the stability of the radiolabelled test item throughout the formulation and application process.

Total Radioactive Residue (TRR) Levels

TRR levels in forage, trash and seed samples, expressed as mg/kg of parent equivalents are shown below.

Table 26: Total radioactive residues (TRRs) in Plant Samples Collected for Clopyralid Nature of Residue in Oilseed Rape Study

Matrix	Days after Application	TRR (mg eq./kg ¹)
Forage	23	1.839
Trash	94	0.224
Seed	94	0.069

¹ = mg eq/kg = mg parent equivalents per kg of tissue

Anthers containing pollen were not combusted prior to sample processing. The pollen was separated from the anthers using water and centrifugation. The pollen was characterized by extraction and final debris combustion. The residue in the remaining anthers post-washing fraction was not determined in this study and therefore an overall TRR is not available.

Distribution of Residues Following Extraction

Forage

Extractability was high in forage where 99.1% TRR (1.822 mg eq/kg) was extracted in neutral organic extracts, 5.3% TRR (0.097 mg eq/kg) in methanolic base extract (plus 1.7% TRR in further extract 1, 0.1 N NaOH and water rinses). The final non extractable residue (NER) accounted for 1.2% TRR (0.022 mg eq/kg). Good correlation between the extractable TRR and initial combustion analysis was observed (107.3%).

Trash

Extractability was high in trash, where 76.9% TRR (0.172 mg eq/kg) was extracted in neutral organic extracts and another 15.9% TRR (0.036 mg eq/kg) in methanolic base extract (plus 5.7% TRR in further extract 1, 0.1 N NaOH and water rinses). The final non-extractable residue (NER) accounted for 7.9% TRR (0.018 mg eq/kg). Good correlation between the extractable TRR and initial combustion analysis was observed (106.4%).

Seed

Neutral extractability was less than observed for forage and trash, although overall extractability was nearly 100%. The neutral acetonitrile/water extraction removed 40.8% TRR (0.028 mg eq/kg), while a total 35.9% TRR (0.025 mg eq/kg) was found in methanolic base extracts (E3-5) (plus 15.5% TRR in E6, 0.5 N NaOH and methanol rinses, and 4.5% TRR in Further Extract 1, 0.5 M NaOH). The final non-extractable residue (NER) accounted for 8.0% TRR (0.006 mg eq/kg). Good correlation was observed between the extractable TRR and initial combustion analysis (104.7%).

The distribution of the residues in the oilseed rape forage, trash and seed samples among the fractions generated following the extractions, expressed both as a percentage of the total sample residue and as mg/kg of clopyralid equivalents is shown below.

Table 27: Distribution and Characterisation of Residues in Oilseed Rape Following a Foliar Treatment with [¹⁴C]-Clopyralid

	Spring Oilseed Rape Forage		Trash		Seeds	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR ¹	100	1.839	100	0.224	100	0.069
Pooled Extract 1-2 Acetonitrile:water (1/1, v/v)	99.1	1.822	76.9	0.172	40.8	0.028
Extract 3 Methanol:10 N NaOH (100/1, v/v)	5.3	0.097	15.9	0.036	9.7	0.007
Extract 4 Methanol:10 N NaOH (100/1, v/v)	NC	NC	NC	NC	11.0	0.008
Extract 5 (overnight) Methanol:10 N NaOH (100/1, v/v)	NC	NC	NC	NC	15.2	0.010
Extract 6 0.5 N NaOH with Methanol washes	NC	NC	NC	NC	15.5	0.011
Sum of initial extracts (1-6)	104.4	1.919	92.8	0.208	92.2	0.064
Initial combustions of PES	4.1	0.075	15.1	0.034	16.2	0.011
Further Extract 1 0.1 M NaOH (forage and trash) 0.5 M NaOH (seeds)	1.7	0.031	5.7	0.013	4.5	0.003
Sum of initial and further extracts	106.1	1.950	98.5	0.221	96.7	0.067
Final Unextracted	1.2	0.022	7.9	0.018	8.0	0.006
Accountability (%) ²	107.3		106.4		104.7	

1 = TRR determined by initial combustion

2 = Accountability = % TRR total extractable + % TRR unextractable

NC = Not Conducted

Anthers/Pollen

The residues in the pollen/anther water washes, based on the harvest weight of anthers sample (containing pollen), were found to be 1.095 and 1.316 mg eq/kg for water wash 1 and 2, respectively. This is equivalent to 45.4 and 54.6 % of the total residue in the anthers sample (containing pollen) water washes.

Anther/pollen water washes contained radioactive residues associated with solubilized nectar and sugars. However, due to the high aqueous solubility of clopyralid these water washes, could also contain clopyralid

residues associated with the anthers and pollen. Therefore, it was not possible to determine the total residue in the individual components of the anthers (e.g., pollen).

The isolated pollen residue following initial processing was extracted. The residues in the pollen extract and final solid residue were 0.592 and 0.100 mg eq/kg, based on the wet weight of isolated pollen. This is equivalent to 85.5 and 14.5% of the total residue in the isolated pollen sample. This is a minimum residue associated with the pollen as an unknown proportion of the residue in the water washes of the anthers (containing pollen) may also have been extracted from the pollen. The unextracted residue in isolated pollen accounted for 14.5% of total (0.100 mg eq/kg).

Table 28: Distribution and Characterisation of Residues in Oilseed Rape Anthers/Pollen Following a Foliar Treatment with [¹⁴C]-Clopyralid

	Spring Oilseed Rape			
	Anthers (containing pollen) Water Washes		Isolated Pollen	
	% of Total ²	mg eq/kg	% of Total ³	mg eq/kg
% of Total ¹	100	2.411	100	0.692
Water Wash 1	45.4	1.095	-	-
Water Wash 2	54.6	1.316	-	-
Extract 1 Methanol:10 N NaOH (100:1 v/v)	-	-	85.5	0.592
Unextracted	NA	NA	14.5 ⁴	0.100 ⁴

1 = Sum of % extractable and unextractable, where applicable.

2 = % of total extractable residue in anthers (containing pollen) sample.

3 = % of total extractable and unextractable residue in isolated pollen.

4 = Due to limited sample it was only possible to combust a single aliquot of the post-extraction solid (PES). This aliquot was based on a wet weight of pollen PES as drying was not possible.

NA = Not Applicable

Characterization and Identification of Residues

Forage:

Parent compound clopyralid (free, *i.e.*, neither bound nor conjugated) was a major component in the aqueous acetonitrile extract accounting for 28.8% TRR (0.530 mg eq/kg). The majority of the neutral extract eluted in a poorly resolved region that was less polar than clopyralid and accounted for a total of 64.0% TRR (1.178 mg eq/kg). Upon base treatment and preparation for chromatography, there was a slight loss of radioactivity prior to profiling, however the entire apolar region was converted to clopyralid, therefore, the entire apolar residue was characterized as base-labile clopyralid.

Parent compound clopyralid (free, *i.e.*, neither bound nor conjugated) was a major component in the aqueous acetonitrile extract flask wash accounting for 0.8% TRR (0.014 mg eq/kg). (E1-2 flask wash). As for the neutral extract, the majority of radioactivity in the flask wash was a poorly resolved, apolar region accounting for a total of 1.8% TRR (0.035 mg eq/kg).

Clopyralid was the only component detected in the organic mild base extract (E3), accounting for 5.1% TRR, 0.094 mg eq/kg. Because the extraction solvent and procedure hydrolysed clopyralid complexes to clopyralid, this region is characterised as base-labile clopyralid.

A further aqueous mild base extraction released 1.7% TRR (0.031 mg eq/kg). Clopyralid was the only component detected in the mild base further extract (FE1), accounting for 1.1% TRR, 0.020 mg eq/kg.

Overall, 106.1% of the radioactivity was extractable, of which 101.6% TRR (1.871 mg eq/kg) was shown to be clopyralid (free plus base-labile). Of this, 29.6% TRR (0.544 mg eq/kg) was “free” clopyralid and 72.0% TRR (1.327 mg eq/kg) was base-labile clopyralid.

Trash:

Parent component clopyralid (free, *i.e.*, neither bound nor conjugated) was a major component in the aqueous acetonitrile extract accounting for 40.2% TRR, 0.090 mg eq/kg. The remainder of the neutral extract eluted in a poorly resolved region that was less polar than clopyralid accounting for a total of 37.2% TRR (0.083 mg eq/kg). Upon base treatment and preparation for chromatography there was a slight loss of radioactivity prior to profiling, however the entire apolar region converted to clopyralid, therefore, the entire apolar residue was characterized as base-labile clopyralid.

Clopyralid was the only component detected in the organic mild base extract (E3), accounting for 13.4% TRR, 0.030 mg eq/kg. Because the extraction solvent and procedure hydrolysed clopyralid complexes to clopyralid, this region is characterised as base-labile clopyralid.

A further aqueous mild base extraction released 5.7% TRR (0.013 mg eq/kg). Clopyralid was the only component detected in this mild base further extract (FE1), accounting for 4.3% TRR, 0.010 mg eq/kg. Overall, 98.5% of the radioactivity was extractable, of which 95.2% TRR (0.213 mg eq/kg) was shown to be clopyralid (free plus base-labile). Of this, 40.2% TRR (0.090 mg eq/kg) was “free” clopyralid and 55.0% TRR (0.123 mg eq/kg) was base-labile clopyralid.

Seed:

Parent compound clopyralid (free, *i.e.*, neither bound nor conjugated) was the major component in the aqueous acetonitrile extract accounting for 29.0% TRR (0.020 mg eq/kg). The remainder of the neutral extract eluted earlier than clopyralid and accounted for a total of 5.4% TRR (0.003 mg eq/kg). Upon base treatment all of the radioactivity in the neutral organic extract eluted with clopyralid.

Clopyralid was the only component detected in the methanolic base extracts (E3-5, DCM eluate from SPE), accounting for 35.9% TRR, 0.025 mg eq/kg. Because the extraction solvent and procedure hydrolyzed clopyralid complexes to clopyralid, this region is characterized as base-labile clopyralid.

Although 15.5% TRR of the seed was extracted with 0.5 N NaOH (and associated methanol washes), this fraction (E6) was unable to be concentrated and prepared for HPLC. Attempts to concentrate E6 – either combined with E3-5 or individually – proved unsuccessful despite multiple attempts. Under the extraction conditions used for E6, the remaining oil would have been hydrolysed into smaller subunits (*e.g.*, fatty acids and glycerols). It is theorized that the formation of these hydrolysis products formed an emulsion layer, causing SPE cartridges to readily clog. Significant dilution of the sample with 1 N NaOH prior to the analytical method SPE procedure did not prove successful and low recoveries were observed.

A further mild base extraction (FE1) released 4.5% TRR (0.003 mg eq/kg). Due to the low levels of radioactivity in this extract, further characterisation was not necessary.

Overall, 96.7% of the radioactivity was extractable, of which 70.3% TRR (0.049 mg eq/kg) was shown to be clopyralid (free plus base-labile). Of this, 29.0% TRR (0.020 mg eq/kg) was “free” clopyralid and 41.3% TRR (0.030 mg eq/kg) was base-labile clopyralid.

Table 29: Clopyralid Levels in Oilseed Rape Following a Foliar Treatment with [¹⁴C]-Clopyralid

	Spring Oilseed Rape					
	Forage		Trash		Seeds	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR ¹	100	1.839	100	0.224	100	0.069
Total Extractable	106.1	1.950	98.5	0.221	96.7	0.067
Total Analysed by HPLC ²	101.7	1.870	95.1	0.213	70.3	0.049
Clopyralid ³	29.6	0.544	40.2	0.090	29.0	0.020
Base-Labile Clopyralid ⁴	72.0	1.327	55.0	0.123	41.3	0.028
Total Identified as Clopyralid (HPLC/MS)	101.6	1.871	95.2	0.213	70.3	0.049
Total Characterised by HPLC	101.6	1.871	95.2	0.213	70.3	0.049
Total Characterised Not Analysed by HPLC	-	-	-	-	20.0	0.014
Unextracted	1.2	0.022	7.9	0.018	8.0	0.006
Accountability ⁵	107.3		106.4		104.7	

1 = TRR determined by combustion

2 = Total extracts that were prepared and analysed by HPLC including any losses/gains during processing

3 = Total free clopyralid detected in aqueous acetonitrile extracts.

4 = Sum of ‘unresolved region’ in aqueous acetonitrile extracts and clopyralid in base extracts (E3, E4, E5 and FE1, where applicable). This region readily converts to clopyralid, particularly upon treatment with base. Extensive investigation work was conducted on the ‘unresolved region’. Mild base treatment resulted in only clopyralid. Further experiments were conducted. No further conclusion can be drawn beyond the fact that this region is entirely base-labile clopyralid.

5 = Accountability = %TRR extractable + %TRR unextractable

Anthers/Pollen:

Following processing, the anthers (containing pollen) water washes were analysed by HPLC. The main component of the residue was free clopyralid: 29.2% of total extracted (0.705 mg eq/kg) in water wash 1 and 34.2% of total extracted (0.824 mg eq/kg) in water wash 2. A number of apolar components (maximum individual component 10.8% of total extracted; 0.259 mg eq/kg) were also detected, with the entire apolar region accounting for 16.2% of total extracted (0.391 mg eq/kg) in water wash 1 and 20.5% of total extracted (0.491 mg eq/kg) in water wash 2. Upon mild base treatment on forage and trash aqueous acetonitrile extracts, only clopyralid was detected. This was also demonstrated in the wheat NOR study. Therefore, the apolar components in anther washes is likewise characterised as base-labile clopyralid.

Pollen extract 1 was analysed by HPLC. The only component of the residue was base-labile clopyralid at 85.5% of total (0.592 mg eq/kg). The unextracted residue in isolated pollen accounted for 14.5% of total (0.100 mg eq/kg).

Table 30: Clopyralid Levels in Oilseed Rape Anthers/Pollen Following a Foliar Treatment with [¹⁴C]-Clopyralid

	Spring Oilseed Rape Anthers (containing pollen)		Isolated Pollen	
	% Total ²	mg eq/kg	% Total ³	mg eq/kg
TRR ¹	100	2.411	100	0.692
Total Extractable	100	2.411	85.5	0.592
Total Analysed by HPLC ⁴	100	2.411	85.5	0.592
Clopyralid ⁵	63.4	1.529	-	-
Base-Labile Clopyralid ⁶	36.7	0.882	85.5	0.592
Total Identified as Clopyralid (HPLC/MS)	100.1	2.411	85.5	0.592
Total Characterised by HPLC	100.1	2.411	85.5	0.592
Unextracted	NA	NA	14.5	0.100

1 = Sum of % extractable and unextractable, where applicable.

2 = % of total extractable residue in anthers (containing pollen) sample.

3 = % of total extractable and unextractable residue in isolated pollen

4 = Total water washes and extracts that were prepared and analysed by HPLC not including any losses/gains during processing

5 = Total free clopyralid detected in water washes.

6 = Sum of 'unresolved region' in water washes and clopyralid in base extracts (E1). The apolar region is characterised as base-labile clopyralid is based on results of the oilseed rape forage and trash, as well as characterisation of wheat matrices.

Residue Analytical Method Confirmation:

Oilseed rape forage, trash, and seeds were also extracted following the analytical method (Dow AgroSciences 120610). Following concentration and further processing of the radiovalidation extracts the samples were analysed by HPLC. Chromatographic analysis of the extracts showed that only clopyralid was detected in forage, trash and seeds. These results are summarized in the table below. As shown, the analytical method was >90% efficient for determining the total clopyralid, free plus base-labile.

Table 31: Summary of Extraction Efficiency and Radiovalidation Using Dow Analytical Method 120610

Matrix	TRR (mg eq/kg)	Clopyralid Determined by Dow Method 120610		Total Clopyralid Determined Above		Dow Method 120610 Efficiency ¹
		% TRR	mg eq/kg	% TRR	mg eq/kg	
Forage	1.839	92.2	1.696	101.6	1.871	90.6%
Trash	0.224	91.4	0.205	95.2	0.213	96.2%
Seed	0.069	65.9	0.045	70.3	0.049	91.8%

1 = Method efficiency determined by dividing Clopyralid Determined by Dow Method 120610 into Total Clopyralid Determined Above, multiplied by 100%.

Metabolite Identification

Confirmation of Clopyralid

The identity of clopyralid was confirmed in neutral organic extracts (E1-2) both before and after base treatment in oilseed rape forage and in neutral organic extracts (E1-2) before base treatment oilseed rape seeds by qualitative mass spectrometry using radio LC-MS and LC-MS/MS. Identification was confirmed by comparison of retention time, isotope pattern, accurate mass measurement, and fragmentation with an authentic reference standard for clopyralid.

Characterisation of Apolar Unresolved Region

An unresolved region, less polar than clopyralid, was detected in concentrated aqueous acetonitrile extracts from forage and trash, eluting from 13.75 – 19 minutes and accounting for 37.3 – 65.8 %TRR (0.083 – 1.213 mg eq/kg). Significant attempts and experiments were made to characterize and identify the residue in this region.

Base Treatment of Treated Samples:

Initially the aqueous acetonitrile extracts from forage, trash and seeds were treated with mild base (methanol:10 N NaOH, 100:1, v/v, ambient, overnight, and subsequent HPLC analysis resulted in only clopyralid was observed. The complete conversion of the unresolved region after a mild base treatment

may be due to hydrolysis and/or a result of the change in sample pH. Therefore, additional investigations into the effect of base on the sample extracts were conducted.

pH Adjustment of Treated Samples:

To investigate the effects of pH on oilseed rape, the concentrated aqueous acetonitrile extracts (E1-2) of forage were subjected to a number of experiments as described below:

1. An aliquot of concentrated E1-2 was pH adjusted with the addition of 5% ammonium hydroxide solution (4:1, v/v sample:base ratio). HPLC analysis of the resulting sample was conducted after 0.5 and 24 hours.

Analysis at both 0.5 hours and 24 hours showed that the unresolved region was not present and only clopyralid and a second peak eluting just after clopyralid were observed.

2. To investigate possible effects from the organic solvent, two aliquots of forage concentrated E1-2 had the solvent removed under a gentle stream of nitrogen. One aliquot was reconstituted in water, containing a small amount of 5% ammonium hydroxide solution (17:1, v/v sample:base ratio), and one aliquot was reconstituted in 5% ammonium hydroxide solution. After 1 hour, the ammonium hydroxide aliquot was acidified to pH 1 and the water aliquot was neutralized to pH 7 immediately prior to analysis.

Analysis of both aliquots showed that the formation of the second peak was not dependent on organic solvent being present. However, the amount of the second peak did increase significantly in the ammonium hydroxide aliquot, which suggested that the concentration of ammonium hydroxide had an effect on the ratio of clopyralid to the second peak. Based on these results it was postulated that the second peak may be an ammonium adduct of clopyralid.

3. To investigate the presence of an ammonium adduct, two aliquots of forage concentrated E1-2 were added to separate vials and one fortified with an equivalent amount of ^{14}C clopyralid (dpm). Both aliquots were dried and reconstituted in an equivalent volume of 5% ammonium hydroxide solution. After 1 hour, both samples were acidified to pH 1 immediately prior to injection.

The analysis showed that the addition of more clopyralid did not increase the amount of the second peak, despite the ammonium hydroxide being present in excess. This indicates that the second peak is not an ammonium adduct of clopyralid.

4. The results from the organic solvent experiments detailed above indicated that there may also be an impact from the pH during chromatography. To investigate this, two aliquots of forage concentrated E1-2 were concentrated and reconstituted in an equivalent volume of 5% ammonium hydroxide solution. After 1 hour, one aliquot was neutralized to pH 7 and one aliquot was acidified to pH 1 immediately prior to analysis.

Analysis at both pH 1 and pH 7 showed that the presence or ratio of clopyralid to the second peak (*ca.* 1:1) was not dependent on pH.

5. Based on the structure of clopyralid, it was postulated that it may be an effective chelator in biological systems, similar to picolinic acid. Structural analogues of clopyralid have been shown to weakly chelate to heavy metal ions in matrix. To investigate this, it was suggested that Na^+ ions may disrupt the chelation similar to the caustic used in the base treatment methods methanol:10 N NaOH (100:1 v/v). Two aliquots of forage concentrated E1-2 had the solvent removed under a gentle stream of nitrogen. One aliquot was reconstituted in an equivalent volume of 5% ammonium hydroxide solution. Both aliquots had an equivalent volume of 1 M NaCl added to generate an excess of Na^+ ions in the sample. The aliquot without 5% ammonium hydroxide acted as a control. After 1 hour, both aliquots were acidified to pH 1 immediately prior to analysis.

The analysis showed that the addition of NaCl had no effect on the ratio of clopyralid to the second peak and the Na^+ ions present did not disrupt the potential chelation, however, this does not rule out chelation as a potential property of clopyralid.

Overall, under certain conditions a second peak eluting just after clopyralid was observed during pH adjustment with 5% ammonium hydroxide solution. This was confirmed not to be an ammonium adduct of clopyralid and was shown to be related to clopyralid following treatment with NaOH.

Fortification of Control Tissue:

Fortification experiments were conducted under Charles River Laboratories' study 229882 – 'The Metabolism of [^{14}C] Clopyralid in Spring Wheat' - to support the hypothesis that the unresolved region is clopyralid weakly interacting with endogenous plant matrix. Control grain tissue was fortified with ^{14}C clopyralid and subjected to aqueous acetonitrile extraction. Following HPLC analysis, the unresolved

region was observed to have been generated during the experiment. The same experiment was attempted twice on control oilseed rape forage tissue under this study. The same result was not observed however, with only clopyralid observed and no apolar region generated.

LC-MS of the Apolar Region:

LC-MS analysis of the apolar region was conducted on oilseed rape forage aqueous acetonitrile extract. Several attempts were made to directly analyse the apolar region, but no identifications of the components could be made. In order to characterize the region, molecular weight filtration and derivatization experiments were attempted. Following methylation of the sample, only one peak was detected and identified as the methyl ester of clopyralid. The apolar region was not isolated prior to methylation, and recovery during the procedure was not calculated, it is not clear whether the apolar peak released clopyralid prior to methylation, or if the apolar peak was otherwise degraded and only the original “free” clopyralid was methylated.

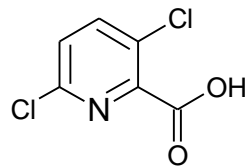
The results from the methylation experiment in conjunction with the other MS experiments indicated that the apolar region was most likely to be a loosely bonded coordinative complex of clopyralid with a low molecular weight matrix component.

Summary of Experiments into Unresolved Apolar Region:

It is hypothesised that this region is the result of chromatographic effects of clopyralid weakly binding and interacting with endogenous material, possibly through weak electrostatic bonds or chelation effects. Conversion to clopyralid is rapid under mild base conditions.

The structures, chemical names and the common names used in the study report for all the residue components that were identified or tentatively identified during this study are shown below.

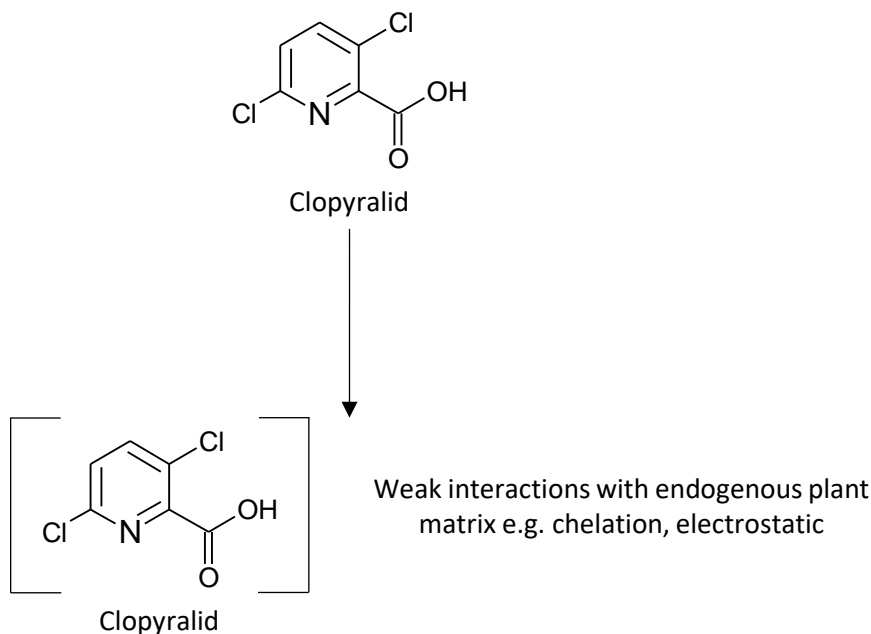
Table 7: Identification of compounds from metabolism study

Common name/code number	Chemical name	Chemical structure
Clopyralid	3,6-Dichloropicolinic Acid	

Metabolic Pathway

A proposed metabolic pathway is presented below. Based on the results obtained from this study clopyralid undergoes interaction with endogenous oilseed rape matrix, in the form of weak chelation effects or electrostatic interactions. No metabolites of clopyralid were detected in this study. This is consistent with previously reported metabolism in wheat.

Figure 4: Proposed metabolic pathway of Clopyralid in Oilseed Rape



Storage Stability

Determination of total radioactive residues (TRR), initial extraction of radioactivity and initial chromatography (including E1-2 for forage, trash and seeds, E3 for forage and trash, water washes of the anthers and methanolic base extraction of pollen) was completed within 41 days of collection.

Following the initial extractions of oilseed rape seed, additional extractions were performed. Characterisation of the principal radioactive residues in oilseed rape seeds was complete within 293 days of sampling.

The profiles from the repeat seed extracts are virtually identical to the initial analysis, and therefore the residues of clopyralid are stable in oilseed rape seed samples under the storage conditions employed on this study.

In addition to the repeat extractions, samples of oilseed rape forage were analysed by LC-MS after 6 months. The radio chromatograms generated during LC-MS analysis are virtually identical to the initial analysis, and therefore the residues of clopyralid are also stable in oilseed rape forage under the storage conditions employed on this study.

Table 32: Summary of storage stability

Matrix	Storage temp. (°C)	Harvest date	Combustion analysis date	Initial extraction date	Initial chromatography date
Forage	-20 °C	11-Jun-2020	19-Jun-2020	01-Jul-2020	08-Jul-2020 (E1-2) 09-Jul-2021 (E3)
Trash	-20 °C	21-Aug-2020	03-Sep-2020	09-Sep-2020	01-Oct-2020 (E1-2) 01-Oct-2020 (E3)
Seed1	-20 °C	21-Aug-2020	03-Sep-2020	29-Jan-2021	04-Feb-2021 (E1-2) 10-Jun-2021 (E3-5)
Pollen/Anther	-20 °C ²	28-May-2020 - 19-Jun-2020 ²	NA	19-Jun-2020/ 02-Jul-2020 ³	21-Jul-2021/ 30-Jul-2020 ⁴

1 = Initial extraction and profiling of seeds was conducted within 41 days of harvest. The extraction and chromatography was repeated to provide full characterisation of the seeds. This repeat extraction data has been used for quantification of seeds.

2 = Anthers/pollen were samples every 1-5 days over for *ca.* 3 weeks. During this time the harvest tissue was stored at *ca.* 4°C. Following the final harvest of anthers/pollen, the sample was processed to separate pollen from anther capsules and water washes conducted on the same day prior to storage of all samples at -20 °C.

3 = Dates separated by: water washes conducted on anthers (containing pollen)/isolated pollen extraction.

4 = Dates separated by: HPLC analysis of water washes/HPLC analysis of isolated pollen.

CONCLUSION

The metabolism of clopyralid was investigated in spring oilseed rape following a single foliar spray application of [2,6-¹⁴C]-clopyralid at an achieved application rate of 199.09 g a.e./ha (99.5% of target) at BBCH 30.

TRR levels in forage, trash and seeds were 1.839, 0.224 and 0.069 mg eq/kg, respectively. Under aqueous acetonitrile conditions, 99.1%, 76.9%, and 40.8% TRR was extracted from forage, trash and seeds, respectively. A further 5.3%, 15.9%, and 35.9% was released upon extraction with methanolic base. Additionally, 15.5% of the TRR (0.011 mg eq/kg) in seeds was released with 0.5 N NaOH with methanol washes. Accountability was 104.7 – 107.3%. Further caustic extractions were conducted on all samples, which released 1.7 – 5.7% TRR. The final non-extractable residues ranged from 1.2 - 8.0% TRR (0.022 mg eq/kg in forage and 0.006 mg eq/kg in seeds). Tissue samples were also extracted following the analytical method (Dow 120610), with extracts accounting for 1.696, 0.205 and 0.045 mg eq/kg. HPLC analysis of the aqueous acetonitrile extracts from forage contained a resolved clopyralid peak (28.8% TRR, 0.530 mg eq/kg) and a broad less polar (apolar), unresolved region (64.0% TRR, 1.178 mg eq/kg). Trash was similar. The equivalent extracts from seeds contained a resolved clopyralid peak (29.0% TRR, 0.020 mg eq/kg) and two minor, more polar regions (2.1 – 3.3% TRR, 0.001 – 0.002 mg eq/kg). Upon mild base treatment (similar to Dow analytical method 120610, using methanol:10 N NaOH (100:1, v/v), overnight) of the neutral organic extracts, only clopyralid was detected (83.8% and 69.4% TRR in forage and trash, respectively). The methanol base extract and further basic extracts from each sample contained only clopyralid and were therefore characterized as base-labile clopyralid. In total, clopyralid (free and base-labile) accounted for 101.6% TRR (1.871 mg eq/kg) in forage, 95.2% TRR (0.213 mg eq/kg) in trash and 70.3% TRR (0.049 mg eq/kg) in seeds. Only clopyralid was detected in the extracts from the analytical method for all three commodities. The anthers containing pollen were washed with water and centrifuged to separate the pollen. The isolated pollen was then extracted methanolic base. Both the water washes and the pollen extract were characterized by HPLC. The majority (63.4%) of the water wash eluted with clopyralid, while the remainder of the water wash eluted in the same apolar region as the forage and trash samples and is therefore characterized as base-labile clopyralid. The majority of the pollen residue was extractable (>85%) and eluted with clopyralid, and is therefore characterized as base-labile clopyralid. Extensive experiments on the apolar unresolved region demonstrated that this region was readily converted to clopyralid in the presence of dilute base. Any light manipulation readily converted the apolar region to clopyralid, which strongly supports the hypothesis that the apolar region is a result of weak electrostatic interactions with endogenous plant matrix and/or chelation of clopyralid to metal ions in the plant. Although extensive attempts were made, no LC/MS mass or fragment ion related to clopyralid could be found in the apolar region, despite optimization of the mass spectrometer for clopyralid. In total, clopyralid (free and base-labile) accounted for >70% of the TRR in all samples, including seed, forage, trash, and pollen/anthers. Furthermore, the amount of total clopyralid detected by Dow analytical method 120610 was in excellent agreement with the exhaustive methods used in a metabolism study, at greater than 90%.

A 2.1.2.1.2 Nature of residue in rotational crops

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

A 2.1.2.1.3 Nature of residues in processed commodities

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

A 2.1.2.2 Nature of residues in livestock

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

A 2.1.3 Magnitude of residues in plants

A 2.1.3.1 Oilseed rape

Table A 1: Comparison of intended and critical EU GAPs

Type of GAP	Number of applications	Application rate per treatment	Interval between application	Growth stage at last application	PHI (days)
cGAP EU (EFSA, 2005; Finland, 2005)	1	100 - 150 g ae/ha	nr	BBCH 12-32	nr
Intended cGAP, Oilseed rape (6*)	1	120 g ae/ha	nr	BBCH 30-51	nr

nr: Not relevant

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

A 2.1.3.1.1 Study 1 – 150534

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/ Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>The study was performed according to guidelines and GLP requirements. The applicant submitted 12 trials consistent with the proposed GAP for oilseed rape. The residues in seeds did not exceed the current MRL for rape of 0.5 mg/kg. Study is acceptable.</i></p>
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Reference: KCA 6.3.1/01

Report Delmotte, R., (2017); Magnitude of the Residues of Halauxifen-methyl and Clopyralid in Oilseed rape (RAC Whole Plant, Seed and Straw), following One Application of GF-3488, Northern Europe - 2015; Study No. RDE-15-20400; DAS Report No. 150534.

Guideline(s): Guidelines for the generation of data concerning residues as provided in Annex II, part A, section 6 and annex III, part A, section 8 of directive 91/414/EEC concerning the placing of plant protection products on the market : foreword 1607/VI/97 rev. 2 and Appendix B - General recommendations for the design, preparation and realisation of residue trials, 7029/VI/95-rev 5, 22.07.97 and amendments.

OECD Guidelines for the Testing of Chemicals, TG 509: Crop Field Trial. Organisation for Economic Co-operation and Development, 7 September 2009.

The OECD Principles of Good Laboratory Practice (as Revised in 1997), OECD Series on Principles of GLP and Compliance Monitoring Number 1, ENV/MC/CHEM(98)17.

The Application of GLP Principles to Field Studies, OECD Series on Principles of GLP and Compliance Monitoring Number 6 (Revised 1999), ENV/JM/MONO(99)22.

The Application of the OECD Principles of GLP to the Organisation and Management of MultiSite Studies, OECD Series on Principles of GLP and Compliance Monitoring Number 14, ENV/JM/MONO(2002)9.

Deviations: None that impact the integrity of the study

GLP: Yes

Acceptability: Yes

Materials and methods

Twelve field trials were conducted on oilseed rape in the United Kingdom (2), Poland (4), Germany (2) and Northern France (4) in 2015. Clopyralid was applied as GF-3488, an EC formulation containing 120 g ae/L. One foliar application was made at a nominal rate of 120 g ae/ha at growth stage BBCH 50.

Samples of seed and straw were taken at normal commercial harvest (NCH) in the eight harvest trials. In the four decline trials samples of seed and straw were taken at a PHI of 7 days, at NCH, and at 3 and 7 days after NCH. Samples of whole plant were taken at 0, 7 and 14 days after application. Samples were stored ≤ -18 °C for a maximum of 337 days. This is accommodated by the available storage stability data on high oil commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined using method 120610, with an LOQ of 0.01 mg/kg. Acceptable procedural recoveries were obtained: whole plant at 0.01-10 mg/kg fortification, mean recovery 101%, RSD=7%, n=15; seed at 0.01-10 mg/kg fortification, mean recovery 96%, RSD=11%, n=15; straw at 0.01-5.0 mg/kg fortification, mean recovery 89%, RSD=11%, n=15.

Results and Discussion

The residue trials are summarized in the following table and residues used for risk assessment are underlined. Residues of clopyralid were <LOQ in control samples.

All trials applied clopyralid in accordance with the intended cGAP rate (within 25%). The trials were conducted at growth stage BBCH 50, compared to the cGAP at BBCH 51. These growth stages can be considered comparable, as both growth stages represent the beginning of inflorescence emergence with flower buds present, but enclosed, and this will not impact the final residue in rape seed. Therefore, the trials can be relied upon to support the intended cGAP. A residue in seed in a single trial (PL07) was identified as an outlier according to the Dixon's Q-test and therefore is not relied upon.

Trials were conducted with an EC formulation but the results can be extrapolated to support the EF-243 SL formulation, as in accordance with OECD 509, formulations diluted in water and with a PHI of >7 days are considered equivalent for residue purposes.

Conclusions

Eleven residue trials on oilseed rape were conducted in northern Europe and are considered supportive of the intended cGAP. Residues of clopyralid in oilseed rape are in the range <0.01-0.30 mg/kg.

Table A 2: Summary of the study 1 trials

Reference:	Magnitude of the Residues of Halauxifen-methyl and Clopyralid in Oilseed rape (RAC Whole Plant, Seed and Straw), following One Application of GF-3488, Northern Europe - 2015, RDE-15-20400; DAS Report No. 150534		
GLP:	Yes	Sample storage conditions:	Max 337 days
Crop/crop group:	Oilseed rape	Analytical method:	120610
Indoor/Outdoor:	Outdoor (field)	Limit of Quantification (mg/kg):	0.01
Formulation:	GF-3488	Limit of Detection (mg/kg):	0.003
Content of active substance (g/kg or g/l):	120 g ae/L clopyralid + 4.797 g ae/L halauxifen-methyl	Residues calculated as:	clopyralid

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/kg)	PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid		
FR01 / 21200 Montagny lès Beaune, Bourgogne, France (EU Northern Zone)/ 2015	Winter oilseed rape / ES Athletic	1. 21-Aug-2014 2. N/A 3. 10-Jul-2015	116	199	59	26-Mar-2015	BBCH.50	Whole plant	3.26	0	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl); NCH = 99 DAT Maximum Storage: 315 days
								Seed	2.33	7	
									2.09	14	
									0.03	92	
								Straw	<u>0.02</u>	99	
									<0.01	102	
									0.01	106	
									0.31	92	
									0.28	99	
									0.30	102	
									0.25	106	
FR02 / 62860 Inchy en Artois, Nord Pas de Calais, France (EU Northern Zone)/ 2015	Winter oilseed rape / Grizzly	1. 08-Sep-2014 2. N/A 3. 23-Jul-2015	120	205	59	02-Apr-2015	BBCH.50	Seed	<u>0.02</u>	112	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 208 days
								Straw	0.18	112	
FR03 / 49700 Doué la Fontaine, Pays de la Loire, France (EU Northern Zone)/ 2015	Winter oilseed rape / DK Expertise	1. 05-Sep-2014 2. N/A 3. 01-Jul-2015	117	200	59	25-Mar-2015	BBCH.50	Seed	<u>0.06</u>	98	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 230 days
								Straw	0.27	98	

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/kg)	PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid		
DE04 / 63863 Eschau, Bayern, Germany (EU Northern Zone)/ 2015	Winter oilseed rape / Avatar	1. 27-Aug-2014 2. N/A 3. 31-Jul-2015	121	207	59	07-Apr-2015	BBCH.50	Whole plant	4.32	0	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl); NCH = 107 DAT Maximum Storage: 303 days
									2.92	7	
									2.30	15	
								Seed	0.02	101	
									<u>0.06</u>	107	
									0.03	115	
								Straw	0.12	101	
									0.43	107	
									0.36	115	
PL05 / 11-010 Bark, Warminsko Marzurskie, Poland (EU Northern Zone)/ 2015	Spring oilseed rape / SW Svinto	1. 14-Apr-2015 2. N/A 3. 24-Aug-2015	120	307	39	04-Jun-2015	BBCH.50	Seed	<u>0.10</u>	81	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 176 days
								Straw	0.26	81	
DE06 / 23919 Rondeshagen, Schleswig-Holstein, Germany (EU Northern Zone)/ 2015	Winter oilseed rape / Exquisite	1. 26-Sep-2014 2. N/A 3. 06-Aug-2015	123	210	59	10-Apr-2015	BBCH.50	Seed	<u><0.01</u>	118	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 194 days
								Straw	0.22	118	
PL07 / 63-040 Chwalecin, Wielkopolska, Poland (EU Northern Zone)/ 2015	Spring oilseed rape / Markus	1. 08-Apr-2015 2. N/A 3. 25-Aug-2015	118	303	39	25-May-2015	BBCH.50	Whole plant	3.87	0	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl); NCH = 86 DAT Maximum Storage: 255 days Data point identified as an outlier according to Dixon's Q-test
									4.17	7	
									2.97	14	
								Seed	0.77	79	
									0.91, 0.67 (0.79)	86	
									0.79	89	
									0.32	92	
								Straw	0.56	79	
									0.42	86	
									0.39	89	
									0.51	92	
PL08 / 64-840 Brzezno, Wielkopolska, Poland (EU Northern Zone)/ 2015	Winter oilseed rape / Granat	1. 30-Aug-2014 2. N/A 3. 29-Jul-2015	124	317	39	14-Apr-2015	BBCH.50	Seed	<u>0.03</u>	106	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 204 days
								Straw	0.17	106	

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/kg)	PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid		
PL09 / 11-034 Tomaszkowo, Warminsko Marzurskie, Poland (EU Northern Zone)/ 2015	Winter oilseed rape / DK Exquisite	1. 25-Aug-2014 2. N/A 3. 10-Aug-2015	120	307	39	16-Apr-2015	BBCH.50	Seed Straw	<u>0.03</u> 0.25	116 116	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 192 days
GB10 / Bucknell, Oxfordshire OX27 7LT, Great Britain (EU Northern Zone)/ 2015	Winter oilseed rape / Harper	1. 04-Sep-2014 2. N/A 3. 30-Jul-2015	121	207	58	25-Mar-2015	BBCH.50	Whole plant Seed Straw	3.67 2.75 2.25 0.12 <u>0.05</u> 0.01 0.02 0.15 0.23 0.09 0.15	0 7 14 105 112 115 118 105 112 115 118	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl); NCH = 112 DAT Maximum Storage: 316 days
FR11 / 02700 Menessis, Picardie, France (EU Northern Zone)/ 2015	Spring oilseed rape / Mosaik	1. 14-May-2015 2. N/A 3. 08-Sep-2015	121	207	58	24-Jun-2015	BBCH.50	Seed Straw	<u>0.30</u> 0.76	76 76	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 163 days
GB12 / Kensworth, Bedfordshire LU6 3QZ, Great Britain (EU Northern Zone)/ 2015	Winter oilseed rape / Charger	1. 16-Aug-2014 2. N/A 3. 28-Jul-2015	121	207	58	25-Mar-2015	BBCH.50	Seed Straw	<u>0.02</u> 0.19	125 125	Appl. rate and conc. are based on actual content of product (117 g a.e./L clopyralid and 5 g a.e./L halauxifen-methyl) Maximum Storage: 205 days

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

A 2.1.3.2 Sugar beet, fodder beet, red beet, mangels

Table A 3: Comparison of intended and critical EU GAPs

Type of GAP	Number of applications	Application rate per treatment	Interval between application	Growth stage at last application	PHI (days)
cGAP EU Sugar beet (EFSA, 2005; Finland, 2005)	2	100 + 200 g ae/ha	21 days	BBCH 10-39	nr
Intended cGAP Sugar beet, Fodder beet, Red beet (1*)	1	120 g ae/ha	nr	BBCH 12-39	42
Zonal cGAP‡	1	150-200 g ae/ha	nr	BBCH 31-39	42

nr: Not relevant

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

‡ A more critical GAP for sugar beet exists in the central EU regulatory zone for product GF-2000.

In accordance with SANTE/2019/12752, data on sugar beet may be extrapolated to fodder beet and red beet, which is the case here.

A 2.1.3.2.1 Study 1 - 200809 – Sugar beet

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>The study was performed according to guidelines and GLP requirements. Twelve residue trials on sugar beet were conducted in Northern and eight trials are considered supportive of the intended cGAP on sugar beet and four trials are considered supportive of the zonal cGAP. Residues of clopyralid found in the treated sugar beet tops samples ranged from 0.080 to 0.56 mg/kg. Residues of clopyralid found in the treated sugar beet roots samples ranged from 0.072 to 0.56 mg/kg (MRL=1.0 mg/kg).</i></p> <p><u>Remark:</u></p> <p>PHI in all the NEU trials vary between 100 and 134 days with an application stage at BBCH 35. zRMS-PL considers the PHI of 42 days is not sufficiently supported by residue data and proposes F: PHI is defined by the application stage at last treatment (time elapsing between last treatment and harvest of the crop) and the BBCH should be 12-35.</p> <p>Residues of clopyralid found in the treated sugar beet roots samples (application rate of 125 g ae/ha at BBCH 35) ranged from 0.10 to 0.20 mg/kg (8 NEU trials: 0.10, 0.13, 0.14, 0.16, 0.17, 0.19, 2x 0.20 mg/kg).</p> <p>Residues of clopyralid found in the treated sugar beet tops samples (application rate of 125 g ae/ha at BBCH 35) ranged from 0.12 to 0.22 mg/kg (8 NEU trials: 2 x 0.12, 0.14, 2 x 0.17, 0.19, 0.20, 0.22 mg/kg).</p>
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Reference: KCA 6.3.2/01

Report Pirie, D., 2021. Magnitude and Decline of Residues of Clopyralid in Sugar Beet Following Applications of GF-1966 in Northern Europe and the UK, Initiated in 2020. Charles River Laboratories Edinburgh Ltd. Study No. 684083/200809.

Guideline(s): OECD Series on Principles of Good Laboratory Practice and Compliance Monitoring Number 13. Consensus Document of the Working Group on Good Laboratory Practice. The application of the OECD principles of GLP

to the Organisation and Management of Multi-Site Studies (June 2002).
OECD Guideline 509, for the Testing of Chemicals, Crop Field Trial (September 2009).

SANCO/3029/99 Revision 4 11/07/00 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 that impact the integrity of the study
GLP:	Yes
Acceptability:	Yes

Materials and methods

Twelve trials were conducted, Poland (2 – Trials 1 and 9), Germany (2 – Trials 2 and 8), The Netherlands (1 – Trial 3), United Kingdom (4 – Trials 4, 10, 11 e 12), Belgium (1 – Trial 5) and North France (2 – Trials 6 and 7), being of this total 6 “Harvest” Trials (Trials 1, 2, 3, 4, 5 and 11) and 6 “Decline” Trials (6, 7, 8, 9, 10 and 12). A single application of the Formulation GF-1966 (a SG – Water Soluble Granules formulation, containing 720 g as clopyralid/kg) was made, at a nominal application rate of 125 g ae/ha (corresponding to 0.174 kg/ha), spray volume 100 - 400 L/ha at BBCH Stage 35 at Trials 1-3 and 5-9; and a single application of the Formulation GF-1966 (a SG – Water Soluble Granules formulation, containing 720 g as clopyralid/kg) was made, at a nominal application rate of 200 g ae/ha (corresponding to 0.278 kg/ha), spray volume 100 - 400 L/ha at BBCH Stage 35 at Trials 4 and 10-12. There were no surfactants/additives included in the spray mixture.

In magnitude of residue tests, control and treated plots were sampled at normal commercial harvest (NCH) and consisted of sugar beet roots and tops. Two control specimens and two treated specimens were submitted for analysis. In decline of residue tests, the control plot was sampled at normal commercial harvest (NCH) and the treated plot was sampled at normal commercial harvest (NCH), 1, 3, 5, 7 and 14 days after NCH and consisted of sugar beet roots and tops. Two control specimens and twelve treated specimens were submitted for analysis.

Specimens were analysed for residues of clopyralid in sugar beet using a method based on analytical method, Dow AgroSciences Study ID 120610, this method was supplied by the Sponsor (Reference 1). The analysis of clopyralid in sugar beet roots and tops samples is reported in the analytical phase report CEMR-9487 “Determination of Residues of Clopyralid in Sugar Beets – Analytical Phase” (Reference Concurrent recoveries from untreated samples of all matrices fortified at the LOQ to as high as 5.0 mg/kg ranged from 75-108%. The determined Limit of Quantification (LOQ) was 0.010 mg/kg for all analytes. The Limit of Detection (LOD) was <0.003 mg/kg for all analytes.

For each test, after collection/preparation the specimens were stored in a freezer set to maintain *ca* -20°C at the Field Test Sites until the date of shipping. During the shipment the samples were kept deep frozen at *ca* -18°C. Treated samples were stored at *ca* -20°C for less than approximately 6 months (maximum storage interval of 161 days) between sampling and analysis.

Samples were analysed in analytical batches, each consisting of two untreated control samples, one reagent blank, one control sample fortified at the limit of detection (LOD) level, at least two control samples fortified at the limit of quantitation (LOQ) and at least two control samples fortified at 10×LOQ or at a higher level. Over all of the analytical batches, there were at least five control samples fortified at the limit of quantitation (LOQ) and at least five control samples fortified at 10×LOQ or at a higher level.

Results and Discussion

All tests show an overall consistent residue profile when the GF-1966 is applied in accordance with the defined good agricultural practice. Residues of clopyralid found in treated samples collected at normal commercial harvest (NCH) or longer sampling intervals, following one foliar application of GF-1966 formulated product are adequate to derive relevant study endpoints. The residue data demonstrate the

following in the residue tests conducted in 2020 in which GF-1966 was applied at 95-105 % of the targeted rate of application:

- Clopyralid residues in treated field-sugar beet roots following foliar application ranged from 0.072 mg/kg to 0.56 mg/kg;
- Clopyralid residues in treated field-sugar beet tops following foliar application ranged from 0.080 mg/kg to 0.56 mg/kg.

Conclusions

Twelve residue trials on sugar beet were conducted in Northern Europe (Poland, Germany, The Netherlands, United Kingdom, Belgium and North France) and eight trials are considered supportive of the intended cGAP on sugar beet and four trials are considered supportive of the zonal cGAP. Residues of clopyralid found in the treated sugar beet tops samples ranged from 0.080 to 0.56 mg/kg. Residues of clopyralid found in the treated sugar beet roots samples ranged from 0.072 to 0.56 mg/kg.

Table A 4: Summary of the study 1 trials

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting :	E. I. du Pont de Nemours and Company	Method of treatment (e) :	Foliar application using an air compressed boom sprayer (Agrotop PL2)
(name, address) :	Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Indoor/Outdoor :	Outdoor
Country :	Poland	Other active substance in the formulation :	None
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance content (g/kg or g/L) :	None
Formulation (e.g., WP) :	SG	Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Urbanowice, 47-270, Kędzierzyńsko- Kozielski, Opole, Poland	Sugar Beet / Graciana	1. 03 Apr 2020 2. NA 3. 19 Oct 2020	--	--	--	Untreated	--	49	Top	ND	--
									Roots	ND	
			127.44*	306	41.7	15 Jun 2020	35	49	Top	0.17	126 (NCH)
									Roots	0.10	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

* g a.e/ha rounded to 2 decimal places

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) : GF-1966 (Clopyralid) Commercial product (name) : GF-1966
Crop/crop group : Sugar Beet / Beet Producer of commercial product : Corteva Agriscience
Responsible body for reporting : E. I. du Pont de Nemours and Company Method of treatment (e) : Foliar application using an air compressed boom sprayer (Agrotop PL1)
(name, address) : Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA Indoor/Outdoor : Outdoor
Country : Germany Other active substance in the formulation : None
Content of active substance: (g/kg) : 719 g/kg acid equivalence Other active substance content (g/kg or g/L) : None
Formulation (e.g., WP) : SG Residues calculated as : Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Blankenhagen, Rostock, 18182 Mecklenburg-Western Pomerania, Germany	Sugar Beet / Advena	1. 02 Apr 2020 2. NA 3. 09 Oct 2020	--	--	--	Untreated	--	49	Top	<LOQ (0.008)	--
									Roots	ND	
			126.77*	304	41.7	10 Jun 2020	35	49	Top	0.12	121 (NCH)
									Roots	0.20	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

* g a.e/ha rounded to 2 decimal places

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) : GF-1966 (Clopyralid) Commercial product (name) : GF-1966
Crop/crop group : Sugar Beet / Beet Producer of commercial product : Corteva Agriscience
Responsible body for reporting : E. I. du Pont de Nemours and Company Method of treatment (e) : Foliar application using a portable knapsack boom sprayer
(name, address) : Member of the Corteva Agriscience Group of Indoor/Outdoor : Outdoor
Companies, Wilmington, Delaware, 19805, USA
Country : The Netherlands Other active substance in the formulation : None
Content of active substance: (g/kg) : 719 g/kg acid equivalence Other active substance content (g/kg or g/L) : None
Formulation (e.g., WP) : SG Residues calculated as : Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Ottersum, 6595 ME, Gennep, Limburg The Netherlands	Sugar Beet / NEENA	1. 06 Apr 2020 2. NA 3. 14 Sep 2020	--	--	--	Untreated	--	49	Top	0.015	--
									Roots	ND	
			123.4	393	31.4	06 Jun 2020	35	49	Top	<u>0.12</u>	100 (NCH)
									Roots	<u>0.14</u>	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (.). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) : GF-1966 (Clopyralid) Commercial product (name) : GF-1966
Crop/crop group : Sugar Beet / Beet Producer of commercial product : Corteva Agriscience
Responsible body for reporting : E. I. du Pont de Nemours and Company Method of treatment (e) : Foliar application using a Knapsack sprayer
(name, address) : Member of the Corteva Agriscience Group of Indoor/Outdoor : Outdoor
Companies, Wilmington, Delaware, 19805, USA
Country : United Kingdom Other active substance in the formulation : None
Content of active substance: (g/kg) : 719 g/kg acid equivalence Other active substance content (g/kg or g/L) : None
Formulation (e.g., WP) : SG Residues calculated as : Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) ^(f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Holme-on- Spalding-Moor, YO43 4DQ, East Yorkshire, United Kingdom	Sugar Beet / Haydn	1. 17 Apr 2020 2. NA 3. 08 Oct 2020	--	--	--	Untreated	--	49	Top	<LOQ (0.007)*	--
									Roots	ND*	
			207.79	312	66.60	15 Jun 2020	35	49	Top	0.56	115 (NCH)
									Roots	0.28	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

* Quantified using Confirmatory ion

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	Belgium	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) ^(f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Templeuve, 7520, Hainaut, Belgium	Sugar Beet / Tessilia	1. 09 Apr 2020 2. NA 3. 20 Oct 2020	--	--	--	Untreated	--	49	Top	ND	--
									Roots	ND	
			128.70	258	49.88	08 Jun 2020	35	49	Top	<u>0.14</u>	134 (NCH)
									Roots	<u>0.16</u>	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	North France	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) ^(f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Senlis-le-Sec, 80300, Hauts-de- France North France	Sugar Beet / Tisserin	1. 30 Mar 2020 2. NA 3. 07 Oct 2020	--	--	--	Untreated	--	49	Top	ND*	--
									Roots	ND*	
			124.39	248	50.16	03 Jun 2020	35	49	Top	0.10	126 (NCH)
									Roots	0.18	
								49	Top	0.085	127
									Roots	0.11	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

* Quantified using Confirmatory ion

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	North France	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(^a)	(^b)	g a.e/ha	Water (L/ha)	g a.e/hL	(^c)	A	S		Clopyralid	(^d)
Sponsor Reference No.: 200809 Senlis-le-Sec, 80300, Hauts-de- France North France	Sugar Beet / Tisserin	1. 30 Mar 2020 2. NA 3. 07 Oct 2020	124.39	248	50.16	03 Jun 2020	35	49	Top	0.20	129
								Roots	0.18		
								49	Top	0.12	131
								Roots	0.19		
								49	Top	0.080	133
								Roots	0.16		
								49	Top	0.14	140
								Roots	0.17		

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	North France	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(^a)	(^b)	g a.e/ha	Water (L/ha)	g a.e/hL	(^c)	A	S		Clopyralid	(^d)
Sponsor Reference No.: 200809 Beuvraignes, 80700, Hauts-de-France North France	Sugar Beet / Frisbee	1. 25 Mar 2020 2. NA 3. 16 Sep 2020	--	--	--	Untreated	--	49	Top	ND*	--
									Roots	ND*	
			131.58	262	50.22	03 Jun 2020	35	49	Top	0.12	105 (NCH)
									Roots	<u>0.20</u>	
								49	Top	0.11	106
									Roots	0.12	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

* Quantified using Confirmatory ion

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	North France	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	
Sponsor Reference No.: 200809 Beuvraignes, 80700, Hauts-de-France North France	Sugar Beet / Frisbee	1. 25 Mar 2020 2. NA 3. 16 Sep 2020	131.58	262	50.22	03 Jun 2020	35	49	Top	0.19	108
									Roots	0.17	
								49	Top	0.16	110
									Roots	0.15	
								49	Top	0.14	112
									Roots	0.13*	
								49	Top	0.14	119
									Roots	0.13	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

* Mean result of two analysis

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting :	E. I. du Pont de Nemours and Company	Method of treatment (e) :	Foliar application using a portable knapsack boom sprayer
(name, address) :	Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Indoor/Outdoor :	Outdoor
Country :	Germany	Other active substance in the formulation :	None
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance content (g/kg or g/L) :	None
Formulation (e.g., WP) :	SG	Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Velen-Ramsdorf, 46342, Borken, North Rhine- Westphalia Germany	Sugar Beet / MARLEY	1. 14 Apr 2020 2. NA 3. 15 Sep 2020	--	--	--	Untreated	--	49	Top	ND	--
									Roots	ND	
			126.7	406	31.2	03 Jun 2020	35	49	Top	0.22	104 (NCH)
									Roots	0.12	
								49	Top	0.17	105
									Roots	0.14	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting :	E. I. du Pont de Nemours and Company	Method of treatment (e) :	Foliar application using a portable knapsack boom sprayer
(name, address) :	Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Indoor/Outdoor :	Outdoor
Country :	Germany	Other active substance in the formulation :	None
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance content (g/kg or g/L) :	None
Formulation (e.g., WP) :	SG	Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Velen-Ramsdorf, 46342, Borken, North Rhine- Westphalia Germany	Sugar Beet / MARLEY	1. 14 Apr 2020 2. NA 3. 15 Sep 2020	126.7	406	31.2	03 Jun 2020	35	49	Top	0.19	107
									Roots	0.12	
								49	Top	0.15	109
									Roots	0.15	
								49	Top	0.17	111
									Roots	0.17	
								49	Top	0.15	118
									Roots	0.13	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting :	E. I. du Pont de Nemours and Company	Method of treatment (e) :	Foliar application using an Air compressed boom sprayer (Agrotop PL2)
(name, address) :	Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Indoor/Outdoor :	Outdoor
Country :	Poland	Other active substance in the formulation :	None
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance content (g/kg or g/L) :	None
Formulation (e.g., WP) :	SG	Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Peckowo, 64-520, Greater Poland Poland	Sugar Beet / Kujavia	1. 06 Apr 2020 2. NA 3. 30 Sep 2020	--	--	--	Untreated	--	49	Top	ND	--
									Roots	ND	
			119.27*	286	41.7	19 Jun 2020	35	49	Top	0.084	103 (NCH)
									Roots	0.079	
								49	Top	0.093	104
									Roots	0.093	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

*g a.e/ha rounded to 2 decimal places

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting :	E. I. du Pont de Nemours and Company	Method of treatment (e) :	Foliar application using an Air compressed boom sprayer (Agrotop PL2)
(name, address) :	Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Indoor/Outdoor :	Outdoor
Country :	Poland	Other active substance in the formulation :	None
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance content (g/kg or g/L) :	None
Formulation (e.g., WP) :	SG	Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Peckowo, 64-520, Greater Poland Poland	Sugar Beet / Kujavia	1. 06 Apr 2020 2. NA 3. 30 Sep 2020	119.27*	286	41.7	19 Jun 2020	35	49	Top	0.095	106
									Roots	0.081	
								49	Top	0.091	108
									Roots	0.093	
								49	Top	0.17	110
									Roots	0.072	
49	Top	0.13	117								
	Roots	0.13									

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

*g a.e/ha rounded to 2 decimal places

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	United Kingdom	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Slingsby, York, YO62 4AY, North Yorkshire United Kingdom	Sugar Beet / Haydn	1. 26 Apr 2020 2. NA 3. 09 Oct 2020	--	--	--	Untreated	--	49	Top	ND	--
									Roots	ND	
			199.16	299	66.61	26 Jun 2020	35	49	Top	0.41	105 (NCH)
									Roots	0.40	
								49	Top	0.44	106
									Roots	0.37	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	United Kingdom	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Slingsby, York, YO62 4AY, North Yorkshire United Kingdom	Sugar Beet / Haydn	1. 26 Apr 2020 2. NA 3. 09 Oct 2020	199.16	299	66.61	26 Jun 2020	35	49	Top	0.42	108
									Roots	0.56	
								49	Top	0.55	110
									Roots	0.50	
								49	Top	0.45	112
									Roots	0.47	
								49	Top	0.39	119
									Roots	0.40	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	United Kingdom	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Moulton Eaugate, PE12 OSZ Spalding, Lincolnshire, United Kingdom	Sugar Beet / Daphna	1. 03 Apr 2020 2. NA 3. 29 Sep 2020	--	--	--	--	--	49	Top	ND	--
									Roots	ND	
			200.60	301	66.64	12 Jun 2020	35	49	Top	<u>0.12</u>	109 (NCH)
									Roots	<u>0.14</u>	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. \geq LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	United Kingdom	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Moreby, York, YO19 6HJ, North Yorkshire United Kingdom	Sugar Beet / Hornet	1. 24 Apr 2020 2. NA 3. 05 Oct 2020	--	--	--	Untreated	--	49	Top	ND	--
									Roots	ND	
			204.20	306	66.73	17 Jun 2020	35	49	Top	0.29	110 (NCH)
									Roots	<u>0.35</u>	
								49	Top	0.28	111
									Roots	0.25	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

NCH = Normal commercial harvest

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

Active ingredient (a.i.)(common name) :	GF-1966 (Clopyralid)	Commercial product (name) :	GF-1966
Crop/crop group :	Sugar Beet / Beet	Producer of commercial product :	Corteva Agriscience
Responsible body for reporting (name, address) :	E. I. du Pont de Nemours and Company Member of the Corteva Agriscience Group of Companies, Wilmington, Delaware, 19805, USA	Method of treatment (e) :	Foliar application using a Knapsack sprayer
Country :	United Kingdom	Indoor/Outdoor :	Outdoor
Content of active substance: (g/kg) :	719 g/kg acid equivalence	Other active substance in the formulation :	None
Formulation (e.g., WP) :	SG	Other active substance content (g/kg or g/L) :	None
		Residues calculated as :	Clopyralid

1	2	3	4			5	6		7	8	9
Report No. Location including Postal Code	Commodity / Variety	Date of: 1. Sowing or Planting 2. Flowering 3. Harvest	Application rate (A1)			Dates of treatment(s)	BBCH Growth stage at Application (A) and at Sampling (S)		Portion Analyzed	Residues (mg/kg) (f)	DALA
	(a)	(b)	g a.e/ha	Water (L/ha)	g a.e/hL	(c)	A	S		Clopyralid	(d)
Sponsor Reference No.: 200809 Moreby, York, YO19 6HJ, North Yorkshire United Kingdom	Sugar Beet / Hornet	1. 24 Apr 2020 2. NA 3. 05 Oct 2020	204.20	306	66.73	17 Jun 2020	35	49	Top	0.20	113
									Roots	0.29	
								49	Top	0.28	115
									Roots	0.27	
								49	Top	0.56	117
									Roots	0.30	
								49	Top	0.24	124
									Roots	0.26	

^a According to EEC and Codex Class Classification (both) should be used

^b Only if relevant

^c Year must be indicated

^d Days after last application

NA = Not applicable

^e High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

^f Analytical method: Based on Dow AgroSciences Study ID 120610

LOQ 0.010 mg/kg; Samples below the LOQ is reported as <LOQ

Residue values below the LOD (0.003 mg/kg) are reported as 'ND' (not detected).

Residue values below the LOQ (0.01 mg/kg) but greater than or equal to the LOD are reported as LOQ with the uncorrected concentration in parentheses (). Values are rounded to 1 significant figure. Residues equal to or greater than 0.01 mg/kg (i.e. ≥LOQ) are reported as their values rounded to 2 significant figures.

A 2.1.3.3 Onion

Table A 5: Comparison of intended and critical EU GAPs

Type of GAP	Number of applications	Application rate per treatment	Interval between application	Growth stage at last application	PHI (days)
cGAP EU (Finland, 2008)	1	100 - 200 g ae/ha	nr	BBCH 13-16	42
Intended cGAP, Onion (7*)	1	120 g ae/ha	nr	BBCH 11-16	42

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

A 2.1.3.3.1 Study 1 - GHE-P-10805

Comments of zRMS:	<p>Three residue trials on onions were conducted in Northern Europe. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 14-16. Samples of mature onions bulbs were taken 42 days after the last application (DALA).</p> <p>Although the residue trials applied two applications, the interval between applications was ca. 1 month, and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application.</p> <p>The maximum storage of samples period was 155 days. Residues of clopyralid were determined using method GRM 01.16 with an LOQ of 0.01 mg/kg.</p> <p>Residues of clopyralid in onions bulb are in the range 0.02 – 0.05 mg/kg (0.02, 0.04, 0.05 mg/kg). Scaled to 120 g ae/ha residues of clopyralid in onions are in the range 0.012 – 0.03 mg/kg.</p> <p>Remark: Trials CEMS-2030B and CEMS-2030C were conducted in Colchester, Essex (UK), the distance was less than 10 km and the dates of planting and treatment are also the same. Thus, the two trials are not considered independent. According to the SANTE/2019/12752 the following factors should be considered separately to decide whether supervised residue trials are independent: - Geographical location and site – Trials at different geographic locations are considered independent, - Dates of planting (annual crops) and treatments – Trials involving significantly different planting dates or treatment dates (> 30 days apart) are considered independent.</p> <p>Therefore, the mean value of two residue results should be used as the representative value for the field trials (0.03 mg/kg).</p> <p>In conclusion, of the three, only two independent trials are available. Residues of clopyralid in onions bulb are 0.03 mg/kg and 0.05 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.018 and 0.03 mg/kg.</p> <p>The study is acceptable.</p>
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Reference: KCA 6.3.3/01

Report Devine, H.C., (2004); Residues of Clopyralid in Onions at Harvest and at Intervals Following Two Application of Lontrel 100 (EF-1136), UK - 2003; DAS Study No. CEMS-2030; DAS Report No. GHE-P-10805

Guideline(s): Commission Directive 96/68/EC amending Council Directive 91/414/EEC

concerning the placing of plant protection products on the market and is designed to comply with the FAO Guidelines on Producing Pesticide Residue Data from Supervised Trials, Rome 1990.

Deviations: None that impact the integrity of the study

GLP: Yes

Acceptability: Yes.

Study No. CEMS-2030/GHE-P-10805 was submitted and evaluated by RMS Finland as part of an MRL evaluation (Finland, 2008) but was not reviewed at EU level. The data also supports the intended critical GAP for onion in C-EU.

Materials and methods

Four field trials were conducted on onions in the United Kingdom in 2003. Clopyralid was applied as LONTREL 100 (EF-1136), an SL formulation containing 100 g ae/L. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and the second at 200 g ae/ha. The first application was made at growth stage BBCH 10-11 and the second at BBCH 14-16, 42 days before harvest.

Samples of mature onion bulbs were taken 42 days after the last application (DALA). Samples were stored ≤ -18 °C for a maximum of 155 days. This is accommodated by the available storage stability data on high water commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined using method GRM 01.16, with an LOQ of 0.01 mg/kg. Acceptable procedural recoveries were obtained: whole plant at 0.01-1.0 mg/kg fortification, mean recovery 87%, RSD=5%, n=3; bulbs at 0.01-0.1 mg/kg fortification, mean recovery 87%, n=2.

Results and Discussion

The residue trials are summarized in the following table, and residues used for risk assessment are underlined. Residues of clopyralid were <LOQ in control samples.

A single trial was cancelled, as the farmer harvested the crop before the end of the trial, so three independent trials are available. Although the residue trials applied two applications, the interval between applications was *ca.* 1 month, and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application. All residues have been scaled to the cGAP rate with respect to the final application rate.

Trials were conducted with an SL formulation but the results can be extrapolated to support the GF-1966 SG formulation, as in accordance with OECD 509, formulations diluted in water, with no organic solvents and with a PHI of >7 days are considered equivalent for residue purposes.

Conclusions

Three residue trials on onions were conducted in the UK and are considered supportive of the intended cGAP. Residues have been scaled with respect to the final application rate. Scaled residues of clopyralid in onions are in the range 0.012-0.033 mg/kg.

Table A 6: Summary of the study 1 trials

Reference: Residues of Clopyralid in Onions at Harvest and at Intervals Following Two Application of Lontrel 100 (EF-1136), UK - 2003, CEMS-2030; DAS Report No. GHE-P-10805

GLP: Yes

Crop/crop group: Onions

Indoor/Outdoor: Outdoor (field)

Formulation: EF-1136

Content of active substance (g/kg or g/l): 100 g ae/L clopyralid-olamine

Sample storage conditions: Max 155 days

Analytical method: GRM 01.16

Limit of Quantification (mg/kg): 0.01

Limit of Detection (mg/kg): 0.002

Residues calculated as: clopyralid

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/kg)		PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid	Scaled to 120.24 g ae/ha		
CEMS-2030A / Thetford, Norfolk, IP24 1LS UK (EU Northern Zone)/ 2003	Onions / Red Baron	1. 09-Mar-2003	98	207	--	01-May-2003 10-Jun-2003	BBCH.16	Bulb	0.05	<u>0.033</u>	<u>42</u>	Maximum Storage: 155 days
		2. N/A	184	185	--			Whole plant	0.10		<0	
		3. 22-Jul-2003							2.82		0	
									0.34		14	
									0.35		28	
CEMS-2030B / Nayland, Colchester, Essex, CO6 4JB UK (EU Northern Zone)/ 2003	Onions / Renate	1. 13-Mar-2003	85	178	--	01-May-2003 10-Jun-2003	BBCH.14	Bulb	0.02	<u>0.012</u>	<u>42</u>	Maximum Storage: 66 days
		2. N/A	206	207	--			Whole plant	0.09		<0	
		3. 22-Jul-2003							0.59		0	
									0.16		14	
									0.10		28	
CEMS-2030C / Great Hor- kesley, Col- chester, Es- sex, CO6 4AP UK (EU Northern Zone)/ 2003	Onions / Hyfort	1. 10-Mar-2003	108	227	--	01-May-2003 10-Jun-2003	BBCH.14	Bulb	0.04	<u>0.022</u>	<u>42</u>	Maximum Storage: 9 days
		2. N/A	222	223	--							
		3. 22-Jul-2003										

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

A 2.1.3.3.2 Study 2 - GHE-P-11080

Comments of zRMS:	<p>Two independent residue trials on onions were conducted in Northern Europe. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 14-16. Samples of mature onions bulbs were taken 42-46 days after the last application (DALA).</p> <p>Although the residue trials applied two applications, the interval between applications was ca. 1 month, and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application.</p> <p>The maximum storage of samples period was 238 days.</p> <p>Residues of clopyralid were determined using method GRM 01.16 with an LOQ of 0.01 mg/kg.</p> <p>Residues of clopyralid in onions bulb are in the range <0.01 – 0.17 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are in the range <0.01 – 0.1 mg/kg.</p> <p>The study is acceptable.</p>
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Reference:	KCA 6.3.3/02
Report	Devine, H. C., (2005); Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF-1136), Northern Europe - 2004; Study No. CEMS-2346; DAS Report No. GHE-P-11080
Guideline(s):	Commission Directive 96/68/EC amending Council Directive 91/414/EEC concerning the placing of plant protection products on the market and is designed to comply with the FAO Guidelines on Producing Pesticide Residue Data from Supervised Trials, Rome 1990.
Deviations:	None that impact the integrity of the study
GLP:	Yes
Acceptability:	Yes.

Study No. CEMS-2346/GHE-P-11080 was submitted and evaluated by RMS Finland as part of an MRL evaluation (Finland, 2008) but was not reviewed at EU level. The data also supports the intended critical GAP for onion in C-EU.

Materials and methods

Two field trials were conducted on onions in the United Kingdom (1) and Northern France (1) in 2004. Clopyralid was applied as LONTREL 100 (EF-1136), an SL formulation containing 100 g ae/L. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and the second at 200 g ae/ha. The first application was made a growth stage BBCH 10-11 and the second at BBCH 14-16, 42-66 days before harvest.

Samples of mature onion bulbs were taken 42-66 days after the last application (DALA). Samples were stored ≤-18 °C for a maximum of 238 days. This is accommodated by the available storage stability data on high water commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined using method GRM 01.16, with an LOQ of 0.01 mg/kg. Acceptable procedural recoveries were obtained: whole plant at 0.01-0.1 mg/kg fortification, mean recovery 105%, RSD=4%, n=4; bulbs at 0.01-0.1 mg/kg fortification, mean recovery 92%, n=2.

Results and Discussion

The residue trials are summarized in the following table and residues used for risk assessment are underlined. Residues of clopyralid were <LOQ in control samples.

Although the residue trials applied two applications, the interval between applications was *ca.* 1 month and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application. All residues have been scaled to the cGAP rate with respect to the final application rate. The PHI in one trial was 66 days, but the trial was conducted at a matching growth stage to the intended GAP; therefore, it is considered suitable to support the intended GAP.

Trials were conducted with an SL formulation but the results can be extrapolated to support the GF-1966 SG formulation, as in accordance with OECD 509, formulations diluted in water, with no organic solvents and with a PHI of >7 days are considered equivalent for residue purposes.

Conclusions

Two residue trials on onions were conducted in northern Europe and are considered supportive of the intended cGAP. Residues have been scaled with respect to the final application rate. Scaled residues of clopyralid in onions are in the range <0.01-0.10 mg/kg.

Table A 7: Summary of the study 2 trials

Reference: Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF-1136), Northern Europe - 2004, CEMS-2346; DAS Report No. GHE-P-11080

GLP: Yes

Crop/crop group: Onions

Indoor/Outdoor: Outdoor (field)

Formulation: EF-1136

Content of active substance (g/kg or g/l): 100 g ae/L clopyralid-olamine

Sample storage conditions: Max 238 days

Analytical method: GRM 01.16

Limit of Quantification (mg/kg): 0.01

Limit of Detection (mg/kg): 0.002

Residues calculated as: Clopyralid

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/kg)		PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid	Scaled to 120.24 g ae/ha		
CEMS-2346A / Higham, Suffolk UK (EU Northern Zone)/ 2004	Onions / Samira	1. 03-Mar-2004 2. N/A 3. 11-Aug-2004	107	213	--	20-May-2004 30-Jun-2004	BBCH.14 to 15	Bulb	<0.01	<u><0.01</u>	<u>42</u>	Maximum Storage: 223 days
			183	187	--			Whole plant	0.06		<0	
									1.02		0	
									0.16		14	
									0.04		28	
CEMS-2346B / Vinzelles (71) France (EU Northern Zone)/ 2004	Onions / Bonus	1. 20-Feb-2004 2. N/A 3. 20-Aug-2004	106	319	--	05-May-2004 15-Jun-2004	BBCH.15 to 16	Bulb	0.17	<u>0.10</u>	<u>66</u>	Maximum Storage: 238 days
			204	307	--			Whole plant	0.08		<0	
									0.71		0	
									0.25		14	
									0.12		28	
									0.17		44	

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated

- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

A 2.1.3.3.3 Study 3 - GHE-P-11272

Comments of zRMS:	<p>One residue trial on onions was conducted in Northern Europe. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 13-15. Samples of mature onions bulbs were taken 42 days after the last application (DALA).</p> <p>Although the residue trials applied two applications, the interval between applications was ca. 1 month, and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application.</p> <p>The storage of samples period was 182 days.</p> <p>Residues of clopyralid were determined using method GRM 01.16 with an LOQ of 0.01 mg/kg.</p> <p>Residues of clopyralid in onions bulb are 0.02 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.012 mg/kg.</p> <p>The study is acceptable.</p>
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Reference:	KCA 6.3.3/03
Report	Devine, H. C., (2006); Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF-1136), Northern Europe-2005; Study No. CEMS-2696; DAS Report No. GHE-P-11272
Guideline(s):	Commission Directive 96/68/EC amending Council Directive 91/414/EEC concerning the placing of plant protection products on the market and is designed to comply with the FAO Guidelines on Producing Pesticide Residue Data from Supervised Trials, Rome 1990.
Deviations:	None that impact the integrity of the study
GLP:	Yes
Acceptability:	Yes.

Study No. CEMS-2696/GHE-P-11272 was submitted and evaluated by RMS Finland as part of an MRL evaluation (Finland, 2008) but was not reviewed at EU level. The data also supports the intended critical GAP for onion in C-EU.

Materials and methods

A single field trial was conducted on onions in the United Kingdom in 2005. Clopyralid was applied as LONTREL 100 (EF-1136), an SL formulation containing 100 g ae/L. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and the second at 200 g ae/ha. The first application was made a growth stage BBCH 10-11 and the second at BBCH 13-15, 42 days before harvest.

Samples of mature onion bulbs were taken 42 days after the last application (DALA). Samples were stored ≤ -18 °C for a maximum of 182 days. This is accommodated by the available storage stability data on high water commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined using method GRM 01.16, with an LOQ of 0.01 mg/kg. Acceptable procedural recoveries were obtained: whole plant at 0.01-1.0 mg/kg fortification, mean recovery 105%, n=2; bulbs at 0.01-0.1 mg/kg fortification, mean recovery 98%, n=2.

Results and Discussion

The residue trials are summarized in the following table and residues used for risk assessment are underlined. Residues of clopyralid were <LOQ in control samples.

Although the residue trial applied two applications, the interval between applications was *ca.* 1 month and it

is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application. All residues have been scaled to the cGAP rate with respect to the final application rate.

Trials were conducted with an SL formulation but the results can be extrapolated to support the GF-1966 SG formulation, as in accordance with OECD 509, formulations diluted in water, with no organic solvents and with a PHI of >7 days are considered equivalent for residue purposes.

Conclusions

One residue trial on onions was conducted in the UK and is considered supportive of the intended cGAP. Residues have been scaled with respect to the final application rate. Scaled residues of clopyralid in onions are 0.012 mg/kg.

Table A 8: Summary of the study 3 trials

Reference: Residues of Clopyralid in Onions at Intervals Following Two Applications of Lontrel 100 (EF-1136), Northern Europe-2005, CEMS-2696; DAS Report No. GHE-P-11272

GLP: Yes

Crop/crop group: Onions

Indoor/Outdoor: Outdoor (field)

Formulation: EF-1136

Content of active substance (g/kg or g/l): 100 g ae/L clopyralid-olamine

Sample storage conditions: Max 182 days

Analytical method: GRM 01.16

Limit of Quantification (mg/kg): 0.01

Limit of Detection (mg/kg): 0.002

Residues calculated as: clopyralid

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treat- ment			Dates of treatment or number and last date	Growth Stage at last treat- ment	Portion an- alysed	Residues (mg/kg)		PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid	Scaled to 120.24 g ae/ha		
CEMS-2696 / Scrooby, Doncaster, Nottingham- shire UK (EU Northern Zone)/ 2005	Onions / Hyskin	1. 21-Mar-2005	101	202	--	11-May-2005	BBCH.13	Bulb	0.02	<u>0.012</u>	<u>42</u>	Maximum Storage: 182 days
		2. N/A	197	306	--	22-Jun-2005	to 15	Whole plant	0.10		<0	
		3. 03-Aug-2005							5.16		0	
									0.24		14	
									0.11		28	

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated

- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

A 2.1.3.3.4 Study 4 - GHE-P-12680

Comments of zRMS:	<p>Two residue trials on onions were conducted in Northern Europe. The field phase was performed at the field station of SynTech Research, at La Chapelle de Guinchay (Northern France). Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and at growth stage BBCH 10-11 and the second at 200 g ae/ha and at BBCH 13-16. The interval between applications was 8-14 days. Samples of mature onions bulbs were taken 44 days after the last application (DALA).</p> <p>The maximum storage of samples period was 108 days. Residues of clopyralid were determined using Dow AgroSciences method GRM 01.16 with an LOQ of 0.01 mg/kg.</p> <p>Residues of clopyralid in onions bulb are 0.025 and 0.03 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.015 and 0.018 mg/kg.</p> <p>Remark: These two residue trials on onions were conducted at the same site: La Chapelle de Guinchay, Burgundy, France. The dates of planting and treatment are also the same. Thus, the two trials are not considered independent. According to the SANTE/2019/12752 the following factors should be considered separately to decide whether supervised residue trials are independent: - Geographical location and site – Trials at different geographic locations are considered independent, - Dates of planting (annual crops) and treatments – Trials involving significantly different planting dates or treatment dates (> 30 days apart) are considered independent. For those trials being considered as not independent the measured residues should be treated as being replicates.</p> <p>The average of the residue of clopyralid is 0.028 mg/kg, scaled to 120 g ae/ha – 0.017 mg/kg.</p> <p>The study is acceptable.</p>
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Reference:	KCA 6.3.3/04
Report	Rawle, N. W., (2012); Residues of Clopyralid in Bulb Onions following Two Applications of EF-1136 - Northern Europe - 2011; DAS Study No. CEMS-4969; DAS Report No. GHE-P-12680.
Guideline(s):	Commission Directive 96/68/EC amending Council Directive 91/414/EEC concerning the placing of plant protection products on the market, Oct.21, 1996 and "Commission Working Document 7029NII95 Rev. 5, General Recommendations for the Design, Preparation and Realisation of Residue Trials, July 22, 1997".
Deviations:	None that impact the integrity of the study
GLP:	Yes
Acceptability:	Yes.

Materials and methods

Two field trials were conducted on onions in Northern France in 2011. Clopyralid was applied as LON-TREL 100 (EF-1136), an SL formulation containing 100 g ae/L. Two foliar applications were made, the first at a nominal rate of 100 g ae/ha and the second at 200 g ae/ha. The first application was made a growth stage BBCH 10-11 and the second at BBCH 13-16, 44 days before harvest.

Samples of mature onion bulbs were taken 44 days after the last application (DALA). Samples were stored ≤18 °C for a maximum of 108 days. This is accommodated by the available storage stability data on high

water commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined using method GRM 01.16, with an LOQ of 0.01 mg/kg. Acceptable procedural recoveries were obtained: bulbs at 0.01 mg/kg fortification, mean recovery 96%, n=2.

Results and Discussion

The residue trials are summarized in the following table and residues used for risk assessment are underlined. Residues of clopyralid were <LOQ in control samples.

Although the residue trials applied two applications, the interval between applications was 8-14 days and it is recognised that the final application generally determines residues in samples at harvest so the trials with two applications may be used to support the proposed GAP with a single application. All residues have been scaled to the cGAP rate with respect to the final application rate. The two trials were conducted in the same location, but used different varieties and different application dates and are therefore considered independent.

Trials were conducted with an SL formulation but the results can be extrapolated to support the GF-1966 SG formulation, as in accordance with OECD 509, formulations diluted in water, with no organic solvents and with a PHI of >7 days are considered equivalent for residue purposes.

Conclusions

Two residue trials on onions were conducted in Northern France and are considered supportive of the intended cGAP. Residues have been scaled with respect to the final application rate. Scaled residues of clopyralid in onions are in the range 0.014-0.018 mg/kg.

Table A 9: Summary of the study 4 trials

Reference: Residues of Clopyralid in Bulb Onions following Two Applications of EF-1136 - Northern Europe - 2011, CEMS-4969; DAS Report No. GHE-P-12680

GLP: Yes

Crop/crop group: Bulb onion

Indoor/Outdoor: Outdoor (field)

Formulation: EF-1136

Content of active substance (g/kg or g/l): 100 g ae/L clopyralid-olamine

Sample storage conditions: Max 108 days

Analytical method: GRM 01.16

Limit of Quantification (mg/kg): 0.01

Limit of Detection (mg/kg): 0.003

Residues calculated as: clopyralid

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/kg)		PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid	Scaled to 120.24 g ae/ha		
CEMS-4969A / La Chapelle de Guinchay, Burgundy (1) France (EU Northern Zone)/ 2011	Bulb onion / Stuttgarter	1. 25-Mar-2011 2. N/A 3. 09-Jun-2011	105 203	263 254	40 80	12-Apr-2011 26-Apr-2011	BBCH.14 to 15	Bulb	0.03	<u>0.018</u>	<u>44</u>	Maximum Storage: 104 days
CEMS-4969B / La Chapelle de Guinchay, Burgundy (2) France (EU Northern Zone)/ 2011	Bulb onion / Red Baron	1. 25-Mar-2011 2. N/A 3. 05-Jun-2011	108 218	271 272	40 80	14-Apr-2011 22-Apr-2011	BBCH.13 to 14	Bulb	0.025	<u>0.014</u>	<u>44</u>	Maximum Storage: 108 days

(a) According to CODEX Classification / Guide (c) Year must be indicated (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are include

(b) Only if relevant (d) Days after last application (Label pre-harvest interval, PHI, underline)

A 2.1.3.3.5 Study 5 - GHE-P-7289

Comments of zRMS:	<p>Two residue trials on onions were conducted in Northern Europe, in Belgium. One foliar application was made with EF-I136 at a nominal rate of 1.5 L/ha (150 g as/ha) at growth stage BBCH 14-15. Samples of whole plant were taken at intervals up to harvest when onion bulb was taken, 62 days after application.</p> <p>The maximum storage of samples period was 361 days.</p> <p>Residues of clopyralid were determined using method ERC 97.20 with an LOQ of 0.02 mg/kg.</p> <p>Residues of clopyralid in onions bulb are 0.075 and 0.14 mg/kg. Scaled to 120 g ae/ha residues of clopyralid in onions are 0.060 and 0.112 mg/kg.</p> <p>Remark: Trials R97-030A and R97-030B were conducted in Belgium, not at the same site, but the distance was ca.15 km. However, the dates of planting are different (ca. 80 days apart) and the dates of application (13 days). Thus, in our opinion, the two trials are considered independent.</p> <p>According to the SANTE/2019/12752 the following factors should be considered separately to decide whether supervised residue trials are independent:</p> <ul style="list-style-type: none"> - Geographical location and site – Trials at different geographic locations are considered independent, - Dates of planting (annual crops) and treatments – Trials involving significantly different planting dates or treatment dates (> 30 days apart) are considered independent. <p>The study is acceptable.</p>
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Reference:	KCA 6.3.5/01
Report	Butler, R. E.; Reynens, P. (1998); Determination of Residues of Clopyralid in Onions following a single application of Lontrel 100 (EF-1136), Belgium, 1997; Study Plan No. R97-030; DAS Report No. GHE-P-7289
Guideline(s):	OECD Principles of Good Laboratory Practice.
Deviations:	None that impact the integrity of the study
GLP:	Yes
Acceptability:	Yes.

Materials and methods

Two trials were undertaken in Belgium during 1997 and each consisted of one untreated and one treated plot. The treated plots were sprayed with EF-I136 at a nominal rate of 1.5 L/ha (150 g as/ha) at growth stage BBCH 14-15. Samples of whole plant were taken at intervals up to harvest when onion bulb was taken, 62 days after application.

Residues in onions were determined using Dow AgroSciences Analytical Method ERC 97.20 which has a lowest validated level of 0.02 mg /kg clopyralid. All residue values equivalent to less than 20% of lowest validated level are classified as "not detected" (ND). All analyses were performed in duplicate.

Clopyralid was extracted from onions by macerating and shaking with caustic methanol. An aliquot was acidified and clopyralid was partitioned into methyl-tertiary-butyl ether (MTBE) then into aqueous sodium bicarbonate, which was acidified and clopyralid was extracted back into MTBE. The organic phase was

evaporated to dryness and the residuum treated with 4% v/v concentrated sulphuric acid / n-butanol to form clopyralid butyl ester. Following the addition of water, clopyralid butyl ester was partitioned into hexane. The hexane extract was then analysed by capillary gas chromatography using mass selective detection.

Results and Discussion

The detector response was checked and found to be linear over the range 0.0025 – 0.12 µg/mL for clopyralid butyl ester.

The mean procedural recovery for onions was 99%. No residues were detected in any of the untreated samples in either trial. Samples were stored up to 361 days, which is covered by storage stability data.

In trial R97-030A, residues of clopyralid in whole plant immediately after application were 0.60 mg/kg declining to 0.14 mg/kg in onion bulb after 62 days. In trial R97-030B, residues of clopyralid in whole plant immediately after application were 0.44 mg/kg declining to 0.08 mg/kg in onion bulb after 62 days.

Conclusions

Two residue trials on onions were conducted in Belgium and are considered supportive of the intended cGAP. Residues have been scaled with respect to the final application rate. Scaled residues of clopyralid in onion are in the range 0.06-0.114 mg/kg.

Table A 10: Summary of the study 5 trials

Reference:	Determination of Residues of Clopyralid in Onions at Intervals Following a Single Application of Lontrel 100 (EF-1136), Belgium, 1997, R97-030		
GLP:	Yes	Sample storage conditions:	Max 361 days
Crop/crop group:	Bulb onion	Analytical method:	ERC 97.20
Indoor/Outdoor:	Outdoor (field)	Limit of Quantification (mg/Kg):	0.02
Formulation:	EF-1136	Limit of Detection (mg/Kg):	0.004
Content of active substance (g/kg or g/l):	100 g ae/L clopyralid-olamine	Residues calculated as:	Clopyralid

Residue trial summary for Bulb onion												
Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/Kg)		PHI (days)	Remarks:
			g ae/ha	Water (L/ha)	g ae/hL				clopyralid	Scaled to 120 g ae/ha		
R97-030A / Chemin de la Siberie, 4, B-5030 Gembloux (EU Northern Zone)/ 1997	Bulb onion / Hystar	1. 06-Mar-1997 2. N/A 3. 10-Sep-1997	147	204.5	71.9	10-Jul-1997	BBCH.14 to 15	Bulb	0.14	0.114	62	Ave Recovery: 99%
								Whole plant	0.595		0	Maximum Storage: 361 days
									0.14		14	
									0.235		28	
								0.22		49		
R97-030B / Rue Xavier Dumont de Chassart, 16, B-6210 Villers-Perwin (EU Northern Zone)/ 1997	Bulb onion / Rocky	1. 27-May-1997 2. N/A 3. 23-Sep-1997	149.63	207	72.3	23-Jul-1997	BBCH.14 to 15	Bulb	0.075	0.060	62	Ave Recovery: 99%
								Whole plant	0.445		0	Maximum Storage: 348 days
									0.165		14	
									0.295		28	
									0.095		49	

A 2.1.4 Magnitude of residues in livestock

A 2.1.4.1 Livestock feeding studies

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

A 2.1.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

A 2.1.5.1 Distribution of the residue in peel/pulp

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

A 2.1.5.2 Processing studies on a core set of representative processes

A 2.1.5.2.1 Sugar beet

Study 1 - GH-C 3305

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/ Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>The study was performed according to guidelines and GLP requirements, however before revision of GLP principles in 1997. Root (RAC) pulp, sugar and molasses were analysed. The processing factor for pulp was 0.77, for white sugar was 0.14 and for molasses was 9.2. Residues of clopyralid are reduced when processed into pulp and white sugar, but concentrated in molasses.</i></p>
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Reference:	KCA 6.5.3/01
Report	Phillips, A.M., (1994); Determination of residues of clopyralid in sugar beet processed fractions; DAS Report No. GH-C 3305.
Guideline(s):	Environmental Protection Agency under Section 171-4(c)(2)(iv), Subdivision 0 of the EPA Pesticide Assessment Guideline.
Deviations:	None
GLP:	Yes
Acceptability:	Yes

Materials and methods

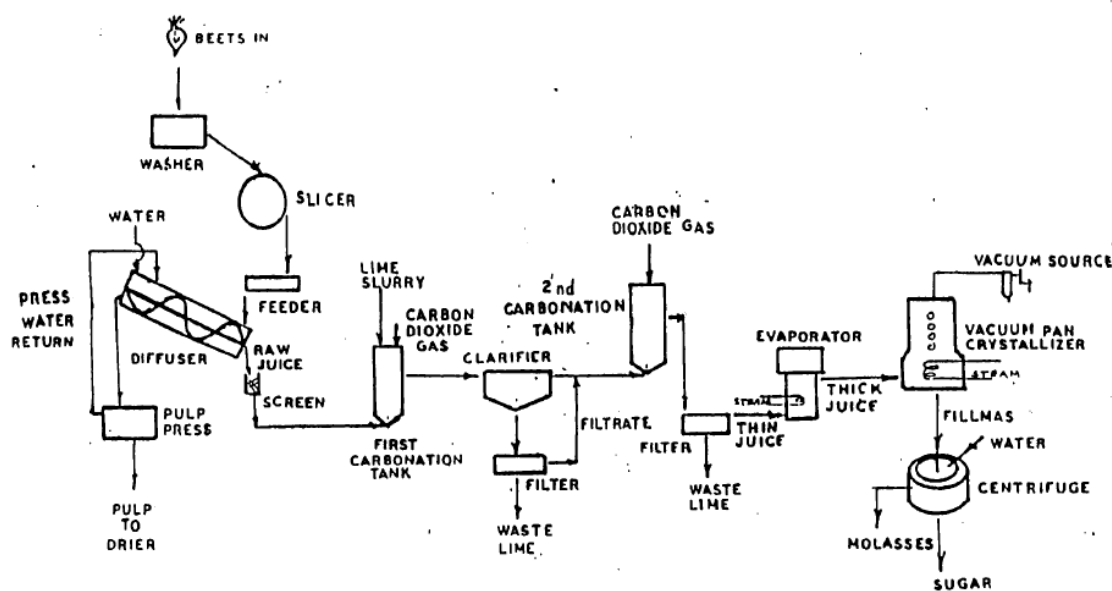
Sugar beet was treated with clopyralid formulation Stinger (formulation XRM-3972) at 841 g ae/ha in 1974/5 in the USA.

Samples of mature sugar beets were taken at normal commercial harvest (131 days after treatment). Processing was conducted at Spreckels Sugar Company, Woodlawn, California to obtain samples of pulp, molasses and sugar in procedures representative of commercial production. All samples were stored frozen (-20°C) for a maximum of 96 days prior to analysis. This is accommodated by the available storage stability data on high water and high starch commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined by method GRM 94.04, with an LOQ of 0.05 mg/kg. The method is based on extraction of samples with a solution of 0.5% sodium hydroxide in water. An aliquot of the sample was extracted into methyl t-butyl ether (MTBE) and then into 0.25M sodium bicarbonate. The sample was acidified and again extracted into MTBE. After evaporating the MTBE, the sample was derivatised with 14% boron trifluoride in methanol to form the methyl derivative. The sample was treated with potassium permanganate. Excess potassium permanganate was reduced with sodium sulfite and then the sample was extracted into 20:80 diethyl ether:hexane. An aliquot of the sample in diethyl ether:hexane was solvent exchanged into toluene and internal standard was added. The final sample in toluene was then transferred to an auto sampler vial for analysis by gas chromatography (GC). Average procedural recoveries of fortified control samples were 80% for sugar beet roots (n=18, 0.05-1.0 mg/kg, %RSD=5); 85% for pulp (n=18, 0.05-1.0 mg/kg, %RSD=10); 92% for molasses (n=18, 0.05-1.0 mg/kg, %RSD=11) and 90% for sugar (n=18, 0.05-1.0 mg/kg, %RSD=7).

Processing phase: The sugar beets were washed with water and hand cleaned with a brush. Clean beets were sliced and the sugar extracted with a mixture of hot water and pulp press water. The spent pulp was mechanically pressed and solar dried to about 20-30% and then further dried using a combination of oven drying at 130 °C and a microwave oven. Diffusion juice was treated with approximately 1.8% lime (CaO) and carbonated, filtered and concentrated to a thick juice. Thick Juice was heated to 75-80 °C and filtered. Sugar (sucrose) was crystallized from filtered thick juice in a vacuum pan by concentrating the juice to a state of supersaturation and then seeding with very fine sugar crystals. Crystallized sugar was separated from its mother liquor (molasses) by centrifugation and washed with hot deionized water. The resulting wet, white sugar was agitated in a stainless steel bowl and dried with warm air.

Figure A1: Processing phase for sugar beet



Results and discussions

Details of the trials and the results of the processing trials are displayed in the tables below.

Table A 11: Summary of processing study 1

Reference: Determination of residues of clopyralid in sugar beet processed fractions; DAS Report No. GH-C 3305
GLP: Yes Sample storage conditions: 96 days
Crop/crop group: Sugar beet Analytical method: GRM 94.04
Indoor/Outdoor: Outdoor (field) Limit of Quantification (mg/Kg): 0.05 mg/kg
Formulation: XRM-3972 Limit of Detection (mg/Kg): -
Content of active substance (g/kg or g/l): 42.6% monoethanolamine salt Residues calculated as: clopyralid

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment		Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/Kg)	PHI (days)	Remarks:
			g ae/ha	Water (L/ha)				clopyralid		
Fresno, California, USA/ 1975	Sugar beet / SS-502	1. 05.05.93 2. - 3. 26.10.93	841	-	17.06.93	6-8 leaf	Root (RAC) Pulp Sugar Molasses	0.64, 0.57 (0.61) 0.47, 0.46 (0.47) 0.084, 0.082 (0.083) 5.3, 5.72 (5.6)	131	

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

Table A 12: Residue data from sugar beet processing study with clopyralid

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/ Reference
Sugar beet	0.61	131	Pulp	0.47	0.77	
			Sugar (white)	0.083	0.14	
			Molasses	5.6	9.2	

* processing factor

Conclusion

The processing factor for pulp was 0.77, for white sugar was 0.14 and for molasses was 9.2. Residues of clopyralid are reduced when processed into pulp and white sugar, but concentrated in molasses.

Study 2 - 181493

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the cMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application. The conclusions of the assessment are presented below:</p> <p><i>The study was performed according to guidelines and GLP requirements. Root (RAC), top with leaves, roots prior to processing, sugar juice molasses, non-refined sugar, pulp brown sugar and white sugar were analysed. Residues of clopyralid do not concentrate on processing to pulp (PF 0.733), brown sugar (PF 0.912), sugar juice (PF 0.514) or white sugar (PF 0.439). Residues of clopyralid do concentrate on processing to molasses (PF 4.236) and non-refined sugar (PF 4.061).</i></p>
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Reference:	KCA 6.5.3/02
Report	Devine, H.C., (2020); Residues of Clopyralid in Sugar Beet and Process Fractions Following Multiple Applications of GF-1966 – Northern Europe – 2019; Study No. CEMS-8908; DAS Report No. 181493..
Guideline(s):	Commission Regulations (EU) No.283/2013 and 284/2013, implementing Regulation (EC) No. 1107/2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC and are designed to comply with the “Commission Working Document 7029/VI/95 Rev. 5, General Recommendations for the Design, Preparation and Realization of Residue Trials, July 22, 1997” and 7035/VI/95 rev.5 July 22 1997 (Processing Studies).
Deviations:	None
GLP:	Yes
Acceptability:	Yes.

Materials and methods

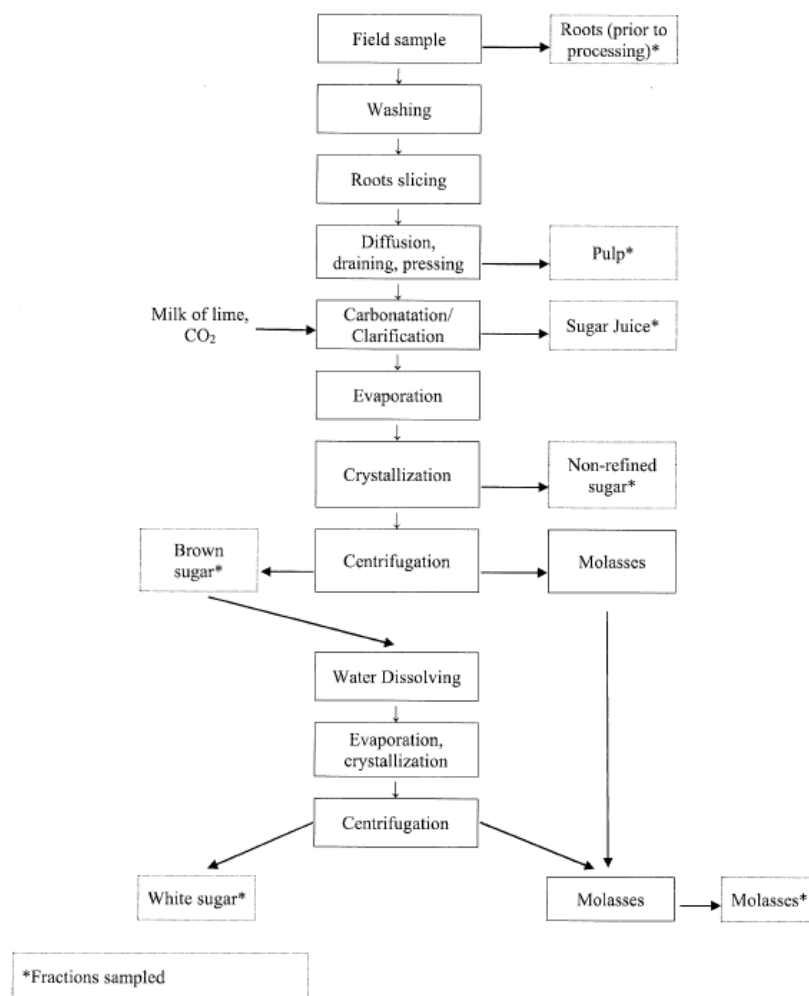
Sugar beet was treated with clopyralid formulation GF-1966 (an SG formulation containing 720 g ae/kg) in 2019 in the Northern France. Two applications were made at 100 g ae/ha + 200 g ae/ha, at growth stages BBCH 18 + BBCH 35.

Samples of mature sugar beets and tops with leaves were taken at normal commercial harvest. Processing was conducted at SAGEA Centro di Saggio, Italy to obtain samples of pulp, molasses, brown sugar, sugar juice, non-refined sugar and white sugar in procedures representative of commercial production. All samples were stored frozen (-20°C) for a maximum of 163 days prior to analysis. This is accommodated by the available storage stability data on high water and high starch commodities reviewed during the EU evaluation (EFSA, 2018a).

Residues of clopyralid were determined by method 120610, with an LOQ of 0.01 mg/kg. Average procedural recoveries of fortified control samples were 90% for tops (n=10, 0.01-1.0 mg/kg, %RSD=8); 78% for roots (n=17, 0.01-5.0 mg/kg, %RSD=7); 81% for juice (n=10, 0.01-1.0 mg/kg, %RSD=11); 86% for molasses (n=17, 0.01-5.0 mg/kg, %RSD=5); 74% for non-refined sugar (n=10, 0.01-5.0 mg/kg, %RSD=3); 76% for pulp (n=10, 0.01-5.0 mg/kg, %RSD=6); 81% for brown sugar (n=10, 0.01-5.0 mg/kg, %RSD=6) and 83% for white sugar (n=10, 0.01-5.0 mg/kg, %RSD=8).

Processing phase: Sugar beet roots were rapidly washed, sliced and immersed in water at 80°C for 2 hours. The juice and pulp were separated and the pulp was pressed and a sample taken. The juice was carbonated/clarified with lime and the clear juice separated and sampled. The sugar juice was concentrated by heating and seeded with sugar crystals to enhance crystallisation of the sugar, non refined sugar was obtained and a sample taken. The sugar was centrifuged to separate brown sugar and molasses. Brown sugar was washed with steam to obtain white sugar. The white sugar was oven dried to obtain the final white sugar sample.

Figure A2: Processing phase for sugar beet



Results and discussions

Details of the trials and the results of the processing trials are displayed in the tables below.

Table A 13: Summary of processing study 2

Reference:	Residues of Clopyralid in Sugar Beet and Process Fractions Following Multiple Applications of GF-1966 – Northern Europe – 2019; Study No. CEMS-8908; DAS Report No. 181493			
GLP:	Yes	Sample storage conditions:	max. 161 days	
Crop/crop group:	Sugar beet	Analytical method:	120610	
Indoor/Outdoor:	Outdoor (field)	Limit of Quantification (mg/Kg):	0.01	
Formulation:	GF-1966	Limit of Detection (mg/Kg):	0.003	
Content of active substance (g/kg or g/l):	720 g ae/kg	Residues calculated as:	clopyralid	

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment		Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/Kg)	PHI (days)	Remarks:
			g ae/ha	Water (L/ha)				clopyralid		
CEMS-8908A La Chapelle de Guinchay, Bourgogne-Franche-Comté, France / 2019	Sugar beet / Millenia KWS	1) 25 Apr 19 2) N/A 3) 02 Sep 19	105.2 198.0	262.8 247.4	13 Jun 19 05 Jul 19	BBCH 18 BBCH 35	Root (RAC) Top with leaves Roots prior to processing Sugar juice Molasses Non-refined sugar Pulp Brown sugar White sugar	1.001* 0.586 0.799* 0.399 3.220 3.059 0.590 0.700 0.231	59	Maximum storage (days): 163 tops 161 roots 86 roots pre-process 98 pulp 88 molasses 95 brown sugar 86 sugar juice 90 non refined sugar 96 white sugar
CEMS-8908B Effiat, Auvergne Rhone Alpes, France / 2019	Sugar beet / Nautille	1) 22 Mar 19 2) N/A 3) 09 Sep 19	105.4 202.3	315.8 303.3	04 Jun 19 18 Jun 19	BBCH 18 BBCH 35	Root (RAC) Top with leaves Roots prior to processing Sugar juice Molasses Non-refined sugar Pulp Brown sugar White sugar	0.530* 0.703 0.614* 0.325 2.728 2.637 0.447 0.582 0.361	83	Maximum storage (days): 156 tops 154 roots 99 roots pre-process 111 pulp 101 molasses 108 brown sugar 99 sugar juice 103 non refined sugar 109 white sugar

N/A Not applicable

- * Mean of 3 analyses
- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

Table A 14: Residue data from sugar beet processing study with clopyralid

Trial	RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	PF*	Comments/Reference
CEMS-8908A	Sugar beet root (prior to processing)	0.799		Pulp	0.590	0.738	
				Molasses	3.220	4.029	
				Brown sugar	0.700	0.875	
				Sugar juice (after clarification)	0.399	0.499	
				Non-refined sugar	3.059	3.828	
				White sugar	0.231	0.289	
CEMS-8908B	Sugar beet root (prior to processing)	0.614	83	Pulp	0.447	0.727	
				Molasses	2.728	4.442	
				Brown sugar	0.582	0.948	
				Sugar juice (after clarification)	0.325	0.529	
				Non-refined sugar	2.637	4.293	
				White sugar	0.361	0.588	
Mean PF							
				Pulp	0.733		
				Molasses	4.236		
				Brown sugar	0.912		
				Sugar juice	0.514		
				Non-refined sugar	4.061		
				White sugar	0.439		

* processing factor, Pf = Residue level in processed commodity / Residue level in sugar beet root prior to processing. Calculated using unrounded data.

Overview of sugar beet processing factors

Table A 15: Summary of sugar beet processing study with clopyralid

Processed commodity	Individual PFs	Mean/median PF
Pulp	0.727, 0.738, 0.77	0.738
Molasses	4.029, 4.442, 9.2	4.442
Brown sugar	0.875, 0.948	0.912
Sugar juice (after clarification)	0.499, 0.529	0.514
Non-refined sugar	3.828, 4.293	4.061
White sugar	0.14, 0.289, 0.588	0.289

Conclusion

Residues of clopyralid do not concentrate on processing to pulp (PF 0.733), brown sugar (PF 0.912), sugar juice (PF 0.514) or white sugar (PF 0.439). Residues of clopyralid do concentrate on processing to molasses (PF 4.236) and non-refined sugar (PF 4.061).

A 2.1.6 Magnitude of residues in representative succeeding crops

One new field crop rotation study (Study No.190557) is available for clopyralid.

A 2.1.6.1 Study 1: 190557

Comments of zRMS:	<p>The study has been evaluated and accepted by zRMS-PL in RR – Part B7 for EF-243/ Lontrel 300 (February 2023, Applicant: Corteva Agriscience). The Registration Report for EF-243/ Lontrel 300 is after the commenting period, but the final report is not currently available. It should be noted that during the commenting period, none of the CMS submitted comments to new studies evaluated by zRMS-PL (Appendix 2 of EF-243/Lontrel 300, February 2023), so this study has not been reassessed in the framework of this application.</p> <p>The conclusions of the assessment are presented below: <i>The study was performed according to guidelines and GLP requirements. Study is acceptable. Six crop rotational trials were conducted in northern Europe (three trials) and Southern Europe (three trials) to determine residues of clopyralid in rotational crops. The study included the following crop groups: leafy and brassica vegetables, root and tuber vegetables, tops of root and tuber vegetables, oilseeds and cereals. The study demonstrated that no residues are expected in succeeding leafy crops or oilseed seeds even at the shortest PBI of 30 days. In root and tuber crops, no significant residues in roots are expected at the PBI of 90 days or longer, while in tops, no significant residues are expected at PBI of 125 days and longer. In cereal grain and straw, significant residues are expected at all PBIs. The residue levels in did not exceed the current MRLs for any of the crops in respective crop groups.</i></p>
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Reference:	KCA 6.6.2/01
Report	Devine, C., 2021; Determination of Residues of Clopyralid after One Application of GF-1966 (EC Formulation) on Bare Soil in Rotational Crops at 3 Sites in Northern Europe and 3 Sites in Southern Europe 2019-2020; CEM Analytical Services Ltd (CEMAS); Study No. CEMS-9009/190557.
Guideline(s):	Commission Regulations (EU) No.283/2013 and 284/2013, implementing Regulation (EC) No. 1107/2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC and are designed to comply with the "Commission Working Document 7029/VI/95 Rev. 5, General Recommendations for the Design, Preparation and Realization of Residue Trials, July 22, 1997".
Deviations:	None that impact the integrity of the study
GLP:	Yes
Acceptability:	Yes

Materials and methods

Six crop rotational trials were conducted in northern Europe (three trials) and Southern Europe (three trials) to determine residues of clopyralid in crops grown as rotational crops at harvest following one application of GF-1966 to bare soil in 2019 and 2020. One additional trial in Southern Europe was added after a few plots were lost and could not be restarted at the original trial site.

GF-1966 is an SG (Water Soluble Granule) formulation, containing a nominal concentration of 949 g/kg clopyralid monoethanolamine, corresponding to 720 g ae/kg of clopyralid. The formulation was applied using appropriate application equipment at the proposed normal use rates and timings. The trials took place in regions typical of Northern and Southern European growing areas for the crops.

GF-1966 was applied once to bare soil at a nominal rate of 60, 80, or 125 g ae/ha with a nominal application volume of 200-400 L/ha. Following plant back intervals (PBI) of a nominal 270-365, 125, 90 or 30 days before replanting the plot were planted with root crop, leafy crop, cereal, or oilseed.

Specimens of radish roots and tops with leaves were collected at normal commercial harvest for root crop. Specimens of whole cabbage were collected at normal commercial harvest for leafy crop at normal commercial harvest. Specimens of barley or wheat whole plant at BBCH 30-39 and barley or wheat straw and grain were collected at normal commercial harvest for cereals. Specimens of oilseed rape whole plant at BBCH 61-69 or sunflower whole plant at BBCH 51-55 and oilseed rape or sunflower seed and rest of plant were collected at normal commercial harvest for oilseed. Samples of soil were taken from each plot

before each planting/sowing but after tillage. Analysis of these was not required. A sample of soil was taken from each trial for GLP characterization.

All samples for residue analysis were placed in freezers within 8 hours of sampling and transported frozen to CEMAS except for 2 soil specimens (CSR-9009-002 & 006) which were put in the freezer within 8.5 hours of sampling. Samples were stored at CEMAS in a freezer set to maintain a sample temperature of < -18 °C. Radish root and radish tops with leaves samples from three sites in Northern France, Poland and Southern France were stored for longer than 443 days and were excluded from further evaluations.

Residues of clopyralid were determined using the Dow Agrosiences Method described in Study No. 120610 using LC-MS/MS with a limit of quantification of 0.01 mg/kg.

Results and discussion

Clopyralid procedural recoveries were in the range 72% to 99% (87% mean) for radish root, 87% to 109% (87% mean) for radish roots, 73% to 110% (90% mean) for radish tops with leaves, 69% to 95% (82% mean) for whole cabbage, 76% to 107% (92% mean) for barley whole plant, 70% to 99% (83% mean) for barley grain, 71% to 102% (88% mean) for barley straw, 85% to 102% (92% mean) for wheat whole plant, 78% to 105% (91% mean) for wheat grain, 80% to 122% (95% mean) for wheat straw, 74% to 105% (85% mean) for oilseed rape whole plant, 68% to 101% (84% mean) for oilseed rape seed, 70% to 109% (85% mean) for oilseed rape rest of plant, 72% to 92% (82% mean) for sunflower whole plant, 73% to 95% (85% mean) for sunflower seed and 78% to 93% (84% mean) for sunflower rest of plant.

The detector response was shown to be linear over the range of 0.5–50 ng/mL (equivalent to 0.0025 – 0.25 mg/kg for clopyralid).

No residues of Clopyralid were detected in any untreated samples with the exception of trial CEMs-9009C where residues were detected in one sample of wheat straw at (0.003 mg/kg) and two samples of wheat grain at (0.009) and 0.011 mg/kg. The retention samples for all untreated wheat straw and grain from this trial were analyzed. For wheat straw these were all not detected. For wheat grain these were all not detected except for the samples from the two plots that had a residue in the shipped sample. These both had residues of (0.004) mg/kg.

Residue data presented in this report are not corrected for procedural recovery except for the wheat straw batch BIS 1564/21 where recovery values are corrected for the residue as the residue in the control is >0.003 mg/kg.

The following table is a summary of the clopyralid residues for all representative commodities at treatment rates 60, 80, and 125 g ae/ha at each PBI.

Crop Group/Representative Crop matrix	Rate (g ae/ha)	Clopyralid residues (mg/kg) per Plant-back interval (days)			
		30	90	125	270-365
Leafy and Brassica Vegetables / Head Cabbage	60	6 x <0.01	6 x <0.01	6 x <0.01	6 x <0.01
	80	6 x <0.01	6 x <0.01	6 x <0.01	6 x <0.01
	125	6 x <0.01	6 x <0.01	6 x <0.01	6 x <0.01
Root and Tuber vegetables / Radish root	60	5 x <0.01*, 0.041	6 x <0.01 †	6 x <0.01	6 x <0.01
	80	6 x <0.01*	6 x <0.01 †	6 x <0.01	6 x <0.01
	125	4 x <0.01*, 0.027, 0.042	6 x <0.01 †	6 x <0.01	6 x <0.01
Tops of Root and Tuber vegetables / Radish tops with leaves	60	4 x <0.01*, 0.17, 0.38	6 x <0.01 †	6 x <0.01	6 x <0.01
	80	4 x <0.01*, 0.17, 0.52	5 x <0.01 †, 0.025	6 x <0.01	6 x <0.01
	125	3 x <0.01, 0.017**, 0.54, 0.63	6 x <0.01 †	6 x <0.01	6 x <0.01
Oilseeds / Oilseed rape and Sunflower whole plant	60	4 x <0.01, 0.086, 0.15	6 x <0.01	6 x <0.01	6 x <0.01
	80	4 x <0.01, 0.11, 0.21	6 x <0.01	6 x <0.01	6 x <0.01
	125	4 x <0.01, 0.097, 0.11	6 x <0.01	6 x <0.01	6 x <0.01
Oilseeds / Oilseed rape and Sunflower seed	60	6 x <0.01	6 x <0.01	5 x <0.01	6 x <0.01
	80	6 x <0.01	6 x <0.01	6 x <0.01	6 x <0.01
	125	6 x <0.01	6 x <0.01	6 x <0.01	6 x <0.01
Oilseeds / Oilseed rape and Sunflower rest of plant	60	4 x <0.01, 0.022, 0.023	6 x <0.01	5 x <0.01	6 x <0.01
	80	4 x <0.01, 0.024, 0.075	6 x <0.01	6 x <0.01	6 x <0.01
	125	3 x <0.01, 0.015, 0.027, 0.089	6 x <0.01	6 x <0.01	6 x <0.01
Cereals / Barley and Wheat whole plant	60	3 x <0.01, 0.018, 0.083, 0.21	4 x <0.01, 0.012, 0.028	6 x <0.01	6 x <0.01
	80	3 x <0.01, 0.029, 0.14, 0.56	6 x <0.01	6 x <0.01	5 x <0.01, 0.017
	125	2 x <0.01, 0.034, 0.20, 0.52, 0.58	5 x <0.01, 0.084	6 x <0.01	6 x <0.01
Cereals / Barley and Wheat grain	60	6 x <0.01	6 x <0.01	6 x <0.01	6 x <0.01
	80	4 x <0.01, 0.14, 0.15	6 x <0.01	5 x <0.01, 0.015	5 x <0.01, 0.037
	125	2 x <0.01, 0.035, 0.053, 0.13, 0.17	5 x <0.01, 0.011	5 x <0.01, 0.012	5 x <0.01, 0.025
Cereals / Barley and Wheat straw	60	2 x <0.01, 0.018, 0.024, 0.040, 0.12	6 x <0.01	6 x <0.01	4 x <0.01, <0.01, 0.012
	80	2 x <0.01, 0.015, 0.027, 0.12, 0.27	6 x <0.01	6 x <0.01	4 x <0.01, 0.012, 0.015
	125	2 x <0.01, 0.10, 0.16, 0.18, 0.28	6 x <0.01	6 x <0.01	4 x <0.01, 0.016, 0.036

* Residue value of <0.01 mg/kg is excluded from the evaluation due to exceedance of frozen storage stability.

† Two residue values of <0.01 mg/kg are excluded from the evaluation due to exceedance of frozen storage stability.

** Residue value of 0.017 mg/kg is excluded from the evaluation due to exceedance of frozen storage stability.

Residues of clopyralid in head cabbage were <0.01 mg/kg at all application rates and at all plantback intervals (PBI). Residues of clopyralid in radish roots ranged from <0.01 to 0.042 mg/kg at all application rates at the shortest PBI of 30 days and were < 0.01 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in oilseed rape and sunflower seeds were <0.01 mg/kg at all application rates and at all PBI. Residues of clopyralid in barley and wheat grain ranged from <0.01 to 0.17 mg/kg at all application rates at the shortest PBI of 30 days and ranged from <0.01 to 0.037 mg/kg at all application rates at a 90-day or longer PBI.

For livestock feed commodities, residues of clopyralid in radish tops with leaves ranged from <0.01 to 0.54 mg/kg at all application rates at the shortest PBI of 30 days and ranged from <0.01 to 0.025 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in oilseed rape and sunflower whole plant (forage commodity) ranged from <0.01 to 0.21 mg/kg at all application rates at the shortest PBI of 30 days and were <0.01 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in the rest of the oilseed rape and sunflower plants after seed harvest ranged from <0.01 to 0.089 mg/kg at all application rates at the shortest PBI of 30 days and were <0.01 mg/kg at all application rates at a 90-day or longer PBI. Residues of clopyralid in barley and wheat whole plant (forage commodity) ranged from <0.01

to 0.58 mg/kg at all application rates at the shortest PBI of 30 days, ranged from <0.01 to 0.084 mg/kg at all application rates at the 90-day PBI, and ranged from <0.01 to 0.017 at all application rates at a 125-day or longer PBI. Residues of clopyralid in the rest of the barley and wheat plants after seed harvest ranged from <0.01 to 0.28 mg/kg at all application rates at the shortest PBI of 30 days and ranged from <0.01 to 0.036 mg/kg at all application rates at a 90-day or longer PBI.

Conclusions

Six crop rotational trials were conducted in northern Europe (three trials) and Southern Europe (three trials) to determine residues of clopyralid in rotational crops and are considered supportive to propose plant-back intervals for crops groups potentially grown in rotation with clopyralid-treated crops, with the exception of individual samples of radish root and radish tops with leaves that were stored in excess of available frozen storage stability data.

Table A 16: Rotational trial summary for study 1

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Crop group:

Root and tuber vegetables

Crop / EPPO code:

Radish / RAPSR

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Producer of commercial product

Dow AgroSciences

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

320 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Radish / EXPO F1	1) 24 Jun 20 2) N/A 3) 29 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	265	63.32	1	05 Jul 19	N/A	Radish roots Radish tops with leaves	ND ND	390 390	Mean Recovery
				32.0	256	81.82	1	05 Jul 19	N/A	Radish roots Radish tops with leaves	ND ND	390 390	Radish tops with leaves 90%
				50.1	250	125.28	1	05 Jul 19	N/A	Radish roots Radish tops with leaves	ND ND	390 390	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Root and tuber vegetables

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Radish / RAPSr

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

320 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Radish / EXPO F1	1) 24 Jun 20 2) N/A 3) 29 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	265	63.32	1	07 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	173 173	Mean Recovery
				32.0	259	82.77	1	07 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	173 173	Radish tops with leaves 90%
				50.1	262	131.25	1	07 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	173 173	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSR

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

France

49350 Gennes Val De Loire (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

319 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Radish / EXPO F1	1) 24 Jun 20 2) N/A 3) 30 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	250	59.76	1	13 Mar 20	N/A	Radish roots Radish tops with leaves	ND ND	139 139	Mean Recovery
				32.0	259	82.77	1	13 Mar 20	N/A	Radish roots Radish tops with leaves	ND ND	139 139	Radish tops with leaves 90%
				50.1	244	122.30	1	13 Mar 20	N/A	Radish roots Radish tops with leaves	ND ND	139 139	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites

Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Root and tuber vegetables

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Radish / RAPSr

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Max frozen storage time prior to analysis

451 days

Content of active substance (g/kg or g/l):

720 g ae/L

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Radish / EXPO F1	1) 12 Sep 19 2) N/A 3) 22 Oct 19	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	241	57.63	1	12 Aug 19	N/A	Radish roots Radish tops with leaves	ND (0.009)	71 71	Mean Recovery
				32.0	262	83.73	1	12 Aug 19	N/A	Radish roots Radish tops with leaves	ND ND	71 71	Radish tops with leaves 90%
				50.1	241	120.81	1	12 Aug 19	N/A	Radish roots Radish tops with leaves	ND 0.017	71 71	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

216 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Cabbage / Impala	1) 24 Jun 20 2) N/A 3) 18 Nov 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	262	62.61	1	05 Jul 19	N/A	Cabbage	ND	502	Mean Recovery Whole Cabbage 82%
				32.0	253	80.87	1	05 Jul 19	N/A	Cabbage	ND	502	
				50.1	253	126.77	1	05 Jul 19	N/A	Cabbage	(0.005)	502	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

216 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Cabbage / Impala	1) 23 Jun 20 2) N/A 3) 18 Nov 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	262	62.61	1	07 Feb 20	N/A	Cabbage	ND	285	Mean Recovery Whole Cabbage 82%
				32.0	253	80.87	1	07 Feb 20	N/A	Cabbage	(0.006)	285	
				50.1	253	126.77	1	07 Feb 20	N/A	Cabbage	(0.004)	285	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

216 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Cabbage / Impala	1) 22 Jun 20 2) N/A 3) 18 Nov 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	253	60.47	1	13 Mar 20	N/A	Cabbage	ND	250	Mean Recovery Whole Cabbage 82%
				32.0	256	81.82	1	13 Mar 20	N/A	Cabbage	(0.008)	250	
				50.1	253	126.77	1	13 Mar 20	N/A	Cabbage	(0.004)	250	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

315 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Cabbage / Caraflex	1) 12 Sep 19 2) N/A 3) 03 Mar 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	259	61.89	1	12 Aug 19	N/A	Cabbage	ND	204	Mean Recovery Whole Cabbage 82%
				32.0	256	81.82	1	12 Aug 19	N/A	Cabbage	ND	204	
				50.1	262	131.25	1	12 Aug 19	N/A	Cabbage	ND	204	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

370 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Barley / RGT Planet	1) 06 Apr 20 2) N/A 3) 22 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	253	60.47	1	05 Jul 19	N/A	Whole Plant	ND	334	Mean Recovery
										Straw	ND	383	Whole Plant 92%
										Grain	ND	383	
				32.0	250	79.92	1	05 Jul 19	N/A	Whole Plant	ND	334	Straw 88%
										Straw	ND	383	
										Grain	ND	383	Grain 83%
				50.1	253	126.77	1	05 Jul 19	N/A	Whole Plant	ND	334	
										Straw	ND	383	
										Grain	ND	383	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Barley / HORVW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

327 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Barley / KWS Jaguar	1) 18 Nov 19 2) N/A 3) 08 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	241	57.63	1	05 Jul 19	N/A	Whole Plant	ND	285	Mean Recovery
										Straw	ND	369	Whole Plant 92%
										Grain	ND	369	
				32.0	256	81.82	1	05 Jul 19	N/A	Whole Plant	ND	285	Straw 88%
										Straw	ND	369	
										Grain	ND	369	Grain 83%
				50.1	253	126.77	1	05 Jul 19	N/A	Whole Plant	ND	285	
										Straw	ND	369	
										Grain	ND	369	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Barley / HORVW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

327 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Barley / KWS Jaguar	1) 18 Nov 19 2) N/A 3) 07 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	247	59.05	1	07 Aug 19	N/A	Whole Plant	ND	252	Mean Recovery
										Straw	ND	335	Whole Plant 92%
										Grain	ND	335	
				32.0	259	82.77	1	07 Aug 19	N/A	Whole Plant	ND	252	Straw 88%
										Straw	ND	335	
										Grain	ND	335	Grain 83%
				50.1	262	131.25	1	07 Aug 19	N/A	Whole Plant	ND	252	
										Straw	ND	335	
										Grain	ND	335	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

382 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Barley / RGT Planet	1) 13 Mar 20 2) N/A 3) 17 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	256	61.18	1	07 Feb 20	N/A	Whole Plant Straw Grain	ND ND ND	105 161 161	Mean Recovery
				32.0	253	80.87	1	07 Feb 20	N/A	Whole Plant Straw Grain	ND ND ND	105 161 161	Straw 88%
				50.1	259	129.75	1	07 Feb 20	N/A	Whole Plant Straw Grain	(0.005) ND ND	105 161 161	Grain 83%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Sunflower / HELAN

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Max frozen storage time prior to analysis

319 days

Content of active substance (g/kg or g/l):

720 g ae/L

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Sunflower / Serin	1) 02 Jun 20 2) N/A 3) 06 Nov 20	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	259	61.89	1	05 Jul 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	384 490 490	Mean Recovery
				32.0	250	79.92	1	05 Jul 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	384 490 490	Seed 85%
				50.1	256	128.26	1	05 Jul 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	384 490 490	Rest of Plant 84%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

146 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Oilseed Rape / Cadran	1) 19 Oct 19 2) N/A 3) 30 Jul 21	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	259	61.89	1	26 Jun 20	N/A	Whole Plant	ND	298	Mean Recovery
				32.0	256	81.82	1	26 Jun 20	N/A	Whole Plant Seed Rest of Plant	ND ND ND	298 399 399	Whole Plant 85% Seed 84%
				50.1	253	126.77	1	26 Jun 20	N/A	Whole Plant Seed Rest of Plant	ND ND ND	298 399 399	Rest of Plant 85%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

146 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Oilseed Rape / Cadran	1) 28 Sep 19 2) N/A 3) 30 Jul 21	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	265	63.32	1	26 Jun 20	N/A	Whole Plant	ND	298	Mean Recovery
										Seed	ND	399	Whole Plant 85%
										Rest of Plant	ND	399	
				32.0	262	83.73	1	26 Jun 20	N/A	Whole Plant	ND	298	Seed 84%
										Seed	ND	399	
										Rest of Plant	ND	399	Rest of Plant 85%
				50.1	262	131.25	1	26 Jun 20	N/A	Whole Plant	ND	298	
										Seed	ND	399	
										Rest of Plant	ND	399	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

49350 Gennes Val De Loire (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

146 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009A 49350 Gennes Val De Loire, France	Oilseed Rape / Cadran	1) 28 Sep 20 2) N/A 3) 30 Jul 21	Broadcast spraying directed to the ground, knapsack sprayer with 7 x XR TeeJet AI 110015 VS nozzles	23.9	259	61.89	1	26 Aug 20	N/A	Whole Plant	ND	237	Mean Recovery
										Seed	ND	338	Whole Plant 85%
										Rest of Plant	ND	338	
				32.0	256	81.82	1	26 Aug 20	N/A	Whole Plant	ND	237	Seed 84%
										Seed	ND	338	
										Rest of Plant	ND	338	Rest of Plant 85%
				50.1	253	126.77	1	26 Aug 20	N/A	Whole Plant	ND	237	
										Seed	ND	338	
										Rest of Plant	ND	338	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

Krościna Mała, 55-110 Prusice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common

name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

243 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Radish / Krasa TOR	1) 02 Apr 20 2) N/A 3) 29 May 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	302	60.723	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND (0.005)	351 351	Mean Recovery
				26.7	302	80.721	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	351 351	Radish tops with leaves 90%
				41.6	304	126.437	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND (0.004)	351 351	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

Krościna Mała, 55-110 Prusice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

190 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Radish / Krasa TOR	1) 15 Jun 20 2) N/A 3) 21 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	296	59.277	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	159 159	Mean Recovery
				26.7	300	80.000	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	159 159	Radish tops with leaves 90%
				41.7	300	125.000	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND (0.004)	159 159	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

Krościna Mała, 55-110 Prusice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

218 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Radish / Krasa TOR	1) 11 May 20 2) N/A 3) 23 Jun 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	296	59.277	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND (0.006)	131 131	Mean Recovery
				26.7	308	82.162	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	131 131	Radish tops with leaves 90%
				41.8	308	128.592	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND (0.006)	131 131	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Crop group:

Root and tuber vegetables

Crop / EPPO code:

Radish / RAPSr

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Producer of commercial product

Dow AgroSciences

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

243 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Radish / Krasa TOR	1) 02 Apr 20 2) N/A 3) 29 May 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	19.9	298	59.277	1	05 Mar 20	N/A	Radish roots Radish tops with leaves	0.041 0.381	85 85	Mean Recovery
				26.7	300	80.000	1	05 Mar 20	N/A	Radish roots Radish tops with leaves	0.041 0.516	85 85	Radish tops with leaves 90%
				41.6	304	126.437	1	05 Mar 20	N/A	Radish roots Radish tops with leaves	0.042 0.541	85 85	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

132 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Cabbage / Report F1	1) 11 May 20 2) N/A 3) 15 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	302	60.723	1	13 Jun 19	N/A	Cabbage	ND	460	Mean Recovery Whole Cabbage 82%
				26.7	302	80.721	1	13 Jun 19	N/A	Cabbage	ND	460	
				41.8	294	122.845	1	13 Jun 19	N/A	Cabbage	ND	460	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Brassicas

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

94 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Cabbage / Report F1	1) 15 Jun 20 2) N/A 3) 23 Oct 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	292	58.554	1	13 Feb 20	N/A	Cabbage	ND	253	Mean Recovery Whole Cabbage 82%
				26.8	304	81.441	1	13 Feb 20	N/A	Cabbage	ND	253	
				41.8	308	128.592	1	13 Feb 20	N/A	Cabbage	ND	253	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

132 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Cabbage / Report F1	1) 11 May 20 2) N/A 3) 15 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	288	57.831	1	13 Feb 20	N/A	Cabbage	ND	215	Mean Recovery Whole Cabbage 82%
				26.7	308	82.162	1	13 Feb 20	N/A	Cabbage	ND	215	
				41.7	296	123.563	1	13 Feb 20	N/A	Cabbage	ND	215	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

132 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Cabbage / Report F1	1) 12 May 20 2) N/A 3) 15 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	19.9	294	58.554	1	15 Apr 20	N/A	Cabbage	ND	153	Mean Recovery Whole Cabbage 82%
				26.7	300	80.000	1	15 Apr 20	N/A	Cabbage	ND	153	
				41.6	302	125.718	1	15 Apr 20	N/A	Cabbage	ND	153	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Wheat / TRZAS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

238 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Wheat / Telimena	1) 02 Apr 20 2) N/A 3) 12 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	304	60.723	1	13 Jun 19	N/A	Whole Plant Straw Grain	(0.004) ND ND	363 426 426	Mean Recovery
				26.4	292	77.117	1	13 Jun 19	N/A	Whole Plant Straw Grain	0.017 0.015 (0.006)	363 426 426	Straw 95%
				41.6	302	125.718	1	13 Jun 19	N/A	Whole Plant Straw Grain	(0.007) (0.004) (0.003)	363 426 426	Grain 91%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Wheat / TRZAS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

279 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Wheat / JB Asano	1) 10 Oct 19 2) N/A 3) 11 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	308	61.446	1	13 Jun 19	N/A	Whole Plant Straw Grain	ND ND ND	322 425 425	Mean Recovery
				26.8	304	81.441	1	13 Jun 19	N/A	Whole Plant Straw Grain	ND (0.009) ND	322 425 425	Straw 95%
				41.7	300	125.000	1	13 Jun 19	N/A	Whole Plant Straw Grain	(0.006) ND (0.004)	322 425 425	Grain 91%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Wheat / TRZAW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

279 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Wheat / JB Asano	1) 10 Oct 19 2) N/A 3) 11 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	286	57.108	1	12 Jul 19	N/A	Whole Plant	0.012	293	Mean Recovery
										Straw	ND	396	Whole Plant 92%
										Grain	ND	396	
				26.7	292	77.838	1	12 Jul 19	N/A	Whole Plant	(0.005)	293	Straw 95%
										Straw	(0.004)	396	
										Grain	ND	396	Grain 91%
				41.8	294	122.845	1	12 Jul 19	N/A	Whole Plant	(0.007)	293	
										Straw	(0.004)	396	
										Grain	ND	396	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Crop group:

Cereals

Crop / EPPO code:

Spring Wheat / TRZAS

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Producer of commercial product

Dow AgroSciences

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

238 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Wheat / Telimena	1) 02 Apr 20 2) N/A 3) 12 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	306	61.446	1	05 Mar 20	N/A	Whole Plant	0.210	97	Mean Recovery
										Straw	0.108	160	Whole Plant 92%
										Grain	0.082	160	
				26.6	304	80.721	1	05 Mar 20	N/A	Whole Plant	0.561	97	Straw 95%
										Straw	0.274	160	
										Grain	0.153	160	Grain 91%
				41.6	302	125.718	1	05 Mar 20	N/A	Whole Plant	0.515	97	
										Straw	0.159	160	
										Grain	0.134	160	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

230 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Oilseed Rape / Delight	1) 02 Apr 20 2) N/A 3) 12 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	310	62.169	1	13 Jun 19	N/A	Whole Plant	(0.004)	369	Mean Recovery
										Seed	ND	426	Whole Plant 85%
										Rest of Plant	ND	426	
				26.8	304	81.441	1	13 Jun 19	N/A	Whole Plant	ND	369	Seed 84%
										Seed	ND	426	
										Rest of Plant	ND	426	Rest of Plant 85%
				41.6	306	127.155	1	13 Jun 19	N/A	Whole Plant	(0.006)	369	
										Seed	ND	426	
										Rest of Plant	ND	426	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

267 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Oilseed Rape / LG Augusta	1) 29 Sep 19 2) N/A 3) 20 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	302	60.723	1	01 Jun 19	N/A	Whole Plant	ND	334	Mean Recovery
										Seed	ND	415	Whole Plant 85%
										Rest of Plant	ND	415	
				26.6	304	80.721	1	01 Jun 19	N/A	Whole Plant	ND	334	Seed 84%
										Seed	ND	415	
										Rest of Plant	(0.003)	415	Rest of Plant 85%
				41.7	300	125.000	1	01 Jun 19	N/A	Whole Plant	ND	334	
										Seed	ND	415	
										Rest of Plant	(0.005)	415	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

267 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Oilseed Rape / LG Augusta/	1) 06 Sep 19 2) N/A 3) 20 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	300	60.000	1	13 Jun 19	N/A	Whole Plant	ND	322	Mean Recovery
										Seed	ND	403	Whole Plant 85%
										Rest of Plant	ND	403	
				26.7	294	78.559	1	13 Jun 19	N/A	Whole Plant	ND	322	Seed 84%
										Seed	ND	403	
										Rest of Plant	(0.010)	403	Rest of Plant 85%
				41.7	300	125.000	1	13 Jun 19	N/A	Whole Plant	ND	322	
										Seed	ND	403	
										Rest of Plant	(0.003)	403	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

Krościna Mała, 55-110 Prusice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

230 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009B Krościna Mała, 55-110 Prusice, Poland	Oilseed Rape / Delight	1) 02 Apr 20 2) N/A 3) 12 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	308	61.446	1	05 Mar 20	N/A	Whole Plant	0.147	103	Mean Recovery
										Seed	ND	160	Whole Plant 85%
										Rest of Plant	0.023	160	
				26.7	308	82.162	1	05 Mar 20	N/A	Whole Plant	0.212	103	Seed 84%
										Seed	ND	160	
										Rest of Plant	0.075	160	Rest of Plant 85%
				41.6	304	126.437	1	05 Mar 20	N/A	Whole Plant	0.097	103	
										Seed	(0.005)	160	
										Rest of Plant	0.027	160	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

55-011 Siechnice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

383 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Radish / Krasa TOR	1) 06 Apr 20 2) N/A 3) 27 May 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	310	62.169	1	12 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	350 350	Mean Recovery
				26.8	304	81.441	1	12 Jun 19	N/A	Radish roots Radish tops with leaves	ND (0.005)	350 350	Radish tops with leaves 90%
				41.6	302	125.718	1	12 Jun 19	N/A	Radish roots Radish tops with leaves	ND (0.009)	350 350	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

55-011 Siechnice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

329 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Radish / Krasa TOR	1) 12 Jun 20 2) N/A 3) 20 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	315	62.892	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	158 158	Mean Recovery
				26.8	304	81.441	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	158 158	Radish tops with leaves 90%
				41.7	296	123.563	1	13 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	158 158	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Root and tuber vegetables

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Radish / RAPS R

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Max frozen storage time prior to analysis

605 days

Content of active substance (g/kg or g/l):

720 g ae/L

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Radish / Krasa TOR	1) 05 Sep 19 2) N/A 3) 18 Oct 19	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.4	302	61.463	1	12 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	128 128	Mean Recovery
				26.7	292	77.838	1	12 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	128 128	Radish tops with leaves 90%
				41.6	304	126.437	1	12 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	128 128	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

55-011 Siechnice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

383 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Radish / Krasa TOR	1) 06 Apr 20 2) N/A 3) 27 May 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	313	62.892	1	04 Mar 20	N/A	Radish roots Radish tops with leaves	(0.009) 0.174	84 84	Mean Recovery
				26.6	304	80.721	1	04 Mar 20	N/A	Radish roots Radish tops with leaves	(0.008) 0.167	84 84	Radish tops with leaves 90%
				41.7	300	125.000	1	04 Mar 20	N/A	Radish roots Radish tops with leaves	0.027 0.627	84 84	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Brassicac

Head Cabbage / BRSOL

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Poland

55-011 Siechnice (EU Northern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

281 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Cabbage / Report F1	1) 18 May 20 2) N/A 3) 14 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	304	60.723	1	12 Jun 19	N/A	Cabbage	ND	460	Mean Recovery Whole Cabbage 82%
				26.7	300	80.000	1	12 Jun 19	N/A	Cabbage	ND	460	
				41.7	298	124.282	1	12 Jun 19	N/A	Cabbage	ND	460	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites

Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

242 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Cabbage / Report F1	1) 12 Jun 20 2) N/A 3) 23 Oct 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	304	60.723	1	13 Feb 20	N/A	Cabbage	ND	253	Mean Recovery Whole Cabbage 82%
				26.7	308	82.162	1	13 Feb 20	N/A	Cabbage	ND	253	
				41.7	296	123.563	1	13 Feb 20	N/A	Cabbage	ND	253	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

281 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Cabbage / Report F1	1) 18 May 20 2) N/A 3) 14 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	300	60.000	1	13 Feb 20	N/A	Cabbage	ND	214	Mean Recovery Whole Cabbage 82%
				26.8	304	81.441	1	13 Feb 20	N/A	Cabbage	ND	214	
				41.7	288	119.971	1	13 Feb 20	N/A	Cabbage	ND	214	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Brassicas

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

281 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Cabbage / Report F1	1) 18 May 20 2) N/A 3) 14 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	302	60.723	1	16 Apr 20	N/A	Cabbage	ND	151	Mean Recovery Whole Cabbage 82%
				26.7	300	80.000	1	16 Apr 20	N/A	Cabbage	ND	151	
				41.6	304	126.437	1	16 Apr 20	N/A	Cabbage	ND	151	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Wheat / TRZAS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

414 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Wheat / Telimena	1) 06 Apr 20 2) N/A 3) 13 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	306	61.446	1	12 Jun 19	N/A	Whole Plant	ND	364	Mean Recovery
										Straw	(0.006)	428	Whole Plant 92%
										Grain	0.012	428	
				26.7	302	80.721	1	12 Jun 19	N/A	Whole Plant	ND	364	Straw 95%
										Straw	0.012	428	
										Grain	0.037	428	Grain 91%
				41.6	306	127.155	1	12 Jun 19	N/A	Whole Plant	ND	364	
										Straw	(0.010)	428	
										Grain	0.025	428	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Wheat / TRZAW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

406 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Wheat / JB Asano	1) 14 Oct 19 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.4	302	61.463	1	12 Jun 19	N/A	Whole Plant Straw Grain	ND ND (0.009)	328 412 412	Mean Recovery
				26.6	306	81.441	1	12 Jun 19	N/A	Whole Plant Straw Grain	ND (0.006) 0.015	328 412 412	Straw 95%
				41.6	290	120.690	1	12 Jun 19	N/A	Whole Plant Straw Grain	ND (0.005) 0.012	328 412 412	Grain 91%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Wheat / TRZAW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

414 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Wheat / JB Asano	1) 14 Oct 19 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	296	59.277	1	12 Jul 19	N/A	Whole Plant	ND	298	Mean Recovery
										Straw	(0.004)	382	Whole Plant 92%
										Grain	(0.008)	382	
				26.7	292	77.838	1	12 Jul 19	N/A	Whole Plant	ND	298	Straw 95%
										Straw	(0.009)	382	
										Grain	0.015	382	Grain 91%
				41.6	290	120.690	1	12 Jul 19	N/A	Whole Plant	(0.004)	298	
										Straw	ND	382	
										Grain	0.011	382	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Wheat / TRZAS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

398 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Wheat / Telimena	1) 06 Apr 20 2) N/A 3) 13 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	300	60.000	1	04 Mar 20	N/A	Whole Plant	0.083	98	Mean Recovery
										Straw	0.040	162	Whole Plant 92%
										Grain	0.090	162	
				26.6	304	80.721	1	04 Mar 20	N/A	Whole Plant	0.138	98	Straw 95%
										Straw	0.115	162	
										Grain	0.143	162	Grain 91%
				41.8	308	128.592	1	04 Mar 20	N/A	Whole Plant	0.195	98	
										Straw	0.183	162	
										Grain	0.167	162	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

341 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Oilseed Rape / Delight	1) 06 Apr 20 2) N/A 3) 13 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	300	60.000	1	12 Jun 19	N/A	Whole Plant Seed Rest of Plant	ND ND (0.007)	371 428 428	Mean Recovery
				26.7	308	82.162	1	12 Jun 19	N/A	Whole Plant Seed Rest of Plant	(0.004) ND (0.009)	371 428 428	Seed 84%
				41.7	300	125.000	1	12 Jun 19	N/A	Whole Plant Seed Rest of Plant	(0.003) ND (0.008)	371 428 428	Rest of Plant 85%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

384 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Oilseed Rape / LG Augusta	1) 28 Sep 19 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.1	313	62.892	1	31 May 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	340 424 424	Mean Recovery
				26.7	310	82.883	1	31 May 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	340 424 424	Seed 84%
				41.6	285	118.534	1	31 May 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	340 424 424	Rest of Plant 85%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Poland

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

384 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Oilseed Rape / LG Augusta/ 	1) 05 Sep 19 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	300	60.000	1	12 Jun 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	328 412 412	Mean Recovery
				26.7	302	80.721	1	12 Jun 19	N/A	Whole Plant Seed Rest of Plant	ND ND ND	328 412 412	Seed 84%
				41.6	302	125.718	1	12 Jun 19	N/A	Whole Plant Seed Rest of Plant	ND ND (0.007)	328 412 412	Rest of Plant 85%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Oilseed Rape / BRSNS

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

Poland

Residues calculated as:

Clopyralid

Trial location (region):

55-011 Siechnice (EU Northern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

341 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009C 55-011 Siechnice, Poland	Oilseed Rape / Delight	1) 06 Apr 20 2) N/A 3) 13 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet 11002 VS nozzles	20.0	304	60.723	1	04 Mar 20	N/A	Whole Plant Seed Rest of Plant	0.086 ND 0.022	105 162 162	Mean Recovery
				26.7	313	83.604	1	04 Mar 20	N/A	Whole Plant Seed Rest of Plant	0.113 (0.004) 0.024	105 162 162	Seed 84%
				41.6	304	126.437	1	04 Mar 20	N/A	Whole Plant Seed Rest of Plant	0.106 (0.004) 0.089	105 162 162	Rest of Plant 85%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Root and tuber vegetables

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Radish / RAPSR

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

France

Residues calculated as:

Clopyralid

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

308 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Radish / Nelson	1) 15 Jun 20 2) N/A 3) 06 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	195	58.36	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	384 384	Mean Recovery
				40.0	190	75.97	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	384 384	Radish tops with leaves 90%
				62.6	198	123.98	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	384 384	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Root and tuber vegetables

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Radish / RAPSR

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

France

Residues calculated as:

Clopyralid

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

343 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Radish / Nelson	1) 15 Jun 20 2) N/A 3) 06 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	214	63.81	1	12 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	145 145	Mean Recovery
				40.0	195	78.05	1	12 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	145 145	Radish tops with leaves 90%
				62.5	214	133.76	1	12 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	145 145	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Root and tuber vegetables

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Radish / RAPSR

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

France

Residues calculated as:

Clopyralid

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

444 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Radish / Diablus	1) 19 Sep 19 2) N/A 3) 29 Oct 19	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	188	56.03	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	133 133	Mean Recovery
				39.9	198	79.09	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	133 133	Radish tops with leaves 90%
				62.7	195	122.34	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	133 133	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Crop group:

Root and tuber vegetables

Crop / EPPO code:

Radish / RAPSr

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Producer of commercial product

Dow AgroSciences

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

308 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Radish / Nelson	1) 15 Jun 20 2) N/A 3) 06 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	201	59.92	1	13 May 20	N/A	Radish roots Radish tops with leaves	ND ND	54 54	Mean Recovery
				39.9	211	84.29	1	13 May 20	N/A	Radish roots Radish tops with leaves	ND ND	54 54	Radish tops with leaves 90%
				62.7	195	122.34	1	13 May 20	N/A	Radish roots Radish tops with leaves	ND ND	54 54	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Brassicac

Head Cabbage / BRSOL

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

France

47370 Saint Georges (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

243 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Cabbage / Capriccio	1) 15 Jun 20 2) N/A 3) 18 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	216	64.58	1	18 Jun 19	N/A	Cabbage	ND	458	Mean Recovery Whole Cabbage 82%
				39.9	206	82.21	1	18 Jun 19	N/A	Cabbage	ND	458	
				62.5	193	120.71	1	18 Jun 19	N/A	Cabbage	ND	458	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites

Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Brassicas

Head Cabbage / BRSOL

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

France

47370 Saint Georges (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

277 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Cabbage / Capriccio	1) 15 Jun 20 2) N/A 3) 18 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	208	62.25	1	12 Feb 20	N/A	Cabbage	ND	219	Mean Recovery Whole Cabbage 82%
				40.0	216	86.37	1	12 Feb 20	N/A	Cabbage	ND	219	
				62.6	206	128.87	1	12 Feb 20	N/A	Cabbage	ND	219	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Brassicas

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

308 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Cabbage / Dutchman	1) 19 Sep 19 2) N/A 3) 10 Mar 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	193	57.58	1	18 Jun 19	N/A	Cabbage	ND	266	Mean Recovery Whole Cabbage 82%
				40.0	182	72.84	1	18 Jun 19	N/A	Cabbage	ND	266	
				62.5	201	125.61	1	18 Jun 19	N/A	Cabbage	ND	266	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

243 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Cabbage / Capriccio	1) 15 Jun 20 2) N/A 3) 18 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	216	64.58	1	13 May 20	N/A	Cabbage	ND	128	Mean Recovery Whole Cabbage 82%
				39.9	201	80.13	1	13 May 20	N/A	Cabbage	ND	128	
				62.6	206	128.87	1	13 May 20	N/A	Cabbage	ND	128	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

356 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Barley / Olympic	1) 23 Mar 20 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	219	65.36	1	18 Jun 19	N/A	Whole Plant Straw Grain	ND ND ND	331 406 406	Mean Recovery
				40.0	208	83.25	1	18 Jun 19	N/A	Whole Plant Straw Grain	ND ND ND	331 406 406	Straw 88%
				62.6	198	123.98	1	18 Jun 19	N/A	Whole Plant Straw Grain	ND ND ND	331 406 406	Grain 83%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Winter Barley / HORVW

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Max frozen storage time prior to analysis

332 days

Content of active substance (g/kg or g/l):

720 g ae/L

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Barley / KWS Orwell	1) 30 Oct 19 2) N/A 3) 29 Jun 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	190	56.80	1	05 Aug 19	N/A	Whole Plant	ND	249	Mean Recovery
										Straw	ND	329	Whole Plant 92%
										Grain	ND	329	
				39.9	206	82.21	1	05 Aug 19		Whole Plant	ND	249	Straw 88%
										Straw	ND	329	
										Grain	ND	329	Grain 83%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Spring Barley / HORVS

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Max frozen storage time prior to analysis

327 days

Content of active substance (g/kg or g/l):

720 g ae/L

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Barley / Olympic	1) 19 May 20 2) N/A 3) 02 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	62.5	197	123.16	1	12 Feb 20	N/A	Whole Plant Straw Grain	ND ND ND	135 203 203	Mean Recovery Whole Plant 92% Straw 88% Grain 83%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

355 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Barley / Olympic	1) 23 Mar 20 2) N/A 3) 29 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	211	63.03	1	12 Feb 20	N/A	Whole Plant	ND	93	Mean Recovery
										Straw	ND	168	Whole Plant 92%
										Grain	ND	168	
				40.0	216	86.37	1	12 Feb 20	N/A	Whole Plant	ND	93	Straw 88%
										Straw	ND	168	
										Grain	ND	168	Grain 83%
				62.6	206	128.87	1	12 Feb 20	N/A	Whole Plant	ND	93	
										Straw	ND	168	
										Grain	ND	168	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

336 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Oilseed Rape / ES Saoker CL	1) 23 Mar 20 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	201	59.92	1	18 Jun 19	N/A	Whole Plant	ND	358	Mean Recovery
										Seed	ND	406	Whole Plant 85%
										Rest of Plant	ND	406	
				40.0	195	78.05	1	18 Jun 19	N/A	Whole Plant	ND	358	Seed 84%
										Seed	ND	406	
										Rest of Plant	ND	406	Rest of Plant 85%
				62.6	185	115.82	1	18 Jun 19	N/A	Whole Plant	ND	358	
										Seed	ND	406	
										Rest of Plant	ND	406	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Oilseed Rape / BRSNS

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

France

Residues calculated as:

Clopyralid

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

348 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Oilseed Rape / ES Saoker CL	1) 23 Mar 20 2) N/A 3) 28 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	203	60.69	1	30 Oct 19	N/A	Whole Plant	ND	224	Mean Recovery
										Seed	ND	272	Whole Plant 85%
										Rest of Plant	ND	272	
				39.9	206	82.21	1	30 Oct 19	N/A	Whole Plant	ND	224	Seed 84%
										Seed	ND	272	
										Rest of Plant	ND	272	Rest of Plant 85%
				62.7	208	130.50	1	30 Oct 19	N/A	Whole Plant	ND	224	
										Seed	ND	272	
										Rest of Plant	ND	272	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

294 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Oilseed Rape / DK Exclaim	1) 19 Sep 19 2) N/A 3) 29 Jun 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.9	190	56.80	1	18 Jun 19	N/A	Whole Plant	ND	290	Mean Recovery
										Seed	ND	377	Whole Plant 85%
										Rest of Plant	ND	377	
				39.9	193	77.01	1	18 Jun 19	N/A	Whole Plant	ND	290	Seed 84%
										Seed	ND	377	
										Rest of Plant	ND	377	Rest of Plant 85%
				62.7	203	127.24	1	18 Jun 19	N/A	Whole Plant	ND	290	
										Seed	ND	377	
										Rest of Plant	ND	377	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

France

Trial location (region):

47370 Saint Georges (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

336 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009D 47370 Saint Georges, France	Oilseed Rape / ES Saoker CL	1) 23 Mar 20 2) N/A 3) 29 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 8 x XR TeeJet AI 110015 VS nozzles	29.8	214	63.81	1	12 Feb 20	N/A	Whole Plant	ND	119	Mean Recovery
										Seed	ND	168	Whole Plant 85%
										Rest of Plant	ND	168	
				39.9	211	84.29	1	12 Feb 20	N/A	Whole Plant	ND	119	Seed 84%
										Seed	ND	168	
										Rest of Plant	ND	168	Rest of Plant 85%
				62.7	203	127.24	1	12 Feb 20	N/A	Whole Plant	ND	119	
										Seed	ND	168	
										Rest of Plant	ND	168	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Crop group:

Root and tuber vegetables

Crop / EPPO code:

Radish / RAPSR

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Producer of commercial product

Dow AgroSciences

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

290 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Radish / Murciano	1) 13 Apr 20 2) N/A 3) 08 Jun 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	20.0	303	60.48	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	361 361	Mean Recovery
				26.7	305	81.36	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	361 361	Radish tops with leaves 90%
				41.7	292	121.68	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	361 361	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSR

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Spain

Agramon, 02490 Albacete (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

Max frozen storage time prior to analysis

Study no. / DAS Study ID

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

342 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Radish / Murciano	1) 15 Oct 19 2) N/A 3) 20 Feb 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	19.9	307	61.20	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	252 252	Mean Recovery
				26.5	307	81.36	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	252 252	Radish tops with leaves 90%
				41.7	295	123.12	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	252 252	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSR

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Spain

Agramon, 02490 Albacete (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common

name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

372 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Radish / Murciano	1) 16 Sep 19 2) N/A 3) 09 Jan 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	19.9	315	62.64	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND (0.007)	210 210	Mean Recovery
				26.7	305	81.36	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND 0.025	210 210	Radish tops with leaves 90%
				41.7	292	121.68	1	13 Jun 19	N/A	Radish roots Radish tops with leaves	ND (0.006)	210 210	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Root and tuber vegetables

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Radish / RAPSR

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

290 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Radish / Murciano	1) 30 Mar 20 2) N/A 3) 08 Jun 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	19.8	323	64.08	1	27 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	102 102	Mean Recovery
				26.5	307	81.36	1	27 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	102 102	Radish tops with leaves 90%
				41.8	303	126.72	1	27 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	102 102	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Brassicac

Head Cabbage / BRSOL

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Spain

Agramon, 02490 Albacete (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

232 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Cabbage / Avon Crest	1) 13 Apr 20 2) N/A 3) 06 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	312	312	61.92	1	13 Jun 19	N/A	Cabbage	ND	420	Mean Recovery Whole Cabbage 82%
				303	303	80.64	1	13 Jun 19	N/A	Cabbage	ND	420	
				297	297	123.84	1	13 Jun 19	N/A	Cabbage	ND	420	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites

Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Brassicas

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

321 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Cabbage / Jupiter	1) 15 Oct 19 2) N/A 3) 10 Mar 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	20.0	313	62.64	1	13 Jun 19	N/A	Cabbage	ND	271	Mean Recovery Whole Cabbage 82%
				26.6	300	79.92	1	13 Jun 19	N/A	Cabbage	ND	271	
				41.8	293	122.4	1	13 Jun 19	N/A	Cabbage	ND	271	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

330 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Cabbage / Jupiter	1) 16 Sep 19 2) N/A 3) 17 Feb 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	19.9	300	59.76	1	13 Jun 19	N/A	Cabbage	ND	249	Mean Recovery Whole Cabbage 82%
				26.7	302	80.64	1	13 Jun 19	N/A	Cabbage	ND	249	
				41.7	290	120.96	1	13 Jun 19	N/A	Cabbage	ND	249	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

232 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Cabbage / Avon Crest	1) 30 Mar 20 2) N/A 3) 06 Aug 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	20.0	320	64.08	1	27 Feb 20	N/A	Cabbage	ND	161	Mean Recovery Whole Cabbage 82%
				26.5	307	81.36	1	27 Feb 20	N/A	Cabbage	ND	161	
				41.8	293	122.4	1	27 Feb 20	N/A	Cabbage	ND	161	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Cereals

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

286 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Barley / Shakira	1) 13 Apr 20 2) N/A 3) 06 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	19.9	307	61.20	1	13 Jun 19	N/A	Whole Plant	ND	356	Mean Recovery
										Straw	0.012	420	Whole Plant 92%
										Grain	ND	420	
				26.6	308	82.08	1	13 Jun 19	N/A	Whole Plant	ND	356	Straw 88%
										Straw	(0.003)	420	
										Grain	ND	420	Grain 83%
				41.7	295	123.12	1	13 Jun 19	N/A	Whole Plant	ND	356	
										Straw	0.036	420	
										Grain	ND	420	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Barley / HORVW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

360 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Barley / Saratoga	1) 22 Nov 19 2) N/A 3) 08 Jun 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	19.9	308	61.20	1	16 Jul 19	N/A	Whole Plant	ND	241	Mean Recovery
										Straw	ND	328	Whole Plant 92%
										Grain	ND	328	
				26.7	318	84.96	1	16 Jul 19	N/A	Whole Plant	ND	241	Straw 88%
										Straw	ND	328	
										Grain	ND	328	Grain 83%
				41.7	292	121.68	1	16 Jul 19	N/A	Whole Plant	(0.003)	241	
										Straw	ND	328	
										Grain	ND	328	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

272 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Barley / Shakira	1) 06 May 20 2) N/A 3) 19 Sep 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	20.0	320	64.08	1	05 Feb 20	N/A	Whole Plant	0.028	133	Mean Recovery
										Straw	ND	227	Whole Plant 92%
										Grain	ND	227	
				26.6	317	84.24	1	05 Feb 20	N/A	Whole Plant	ND	133	Straw 88%
										Straw	ND	227	
										Grain	ND	227	
				41.7	307	128.16	1	05 Feb 20	N/A	Whole Plant	0.084	133	Grain 83%
										Straw	ND	227	
										Grain	ND	227	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Spring Barley / HORVS

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

Spain

Residues calculated as:

Clopyralid

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

309 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Barley / Shakira	1) 03 Mar 20 2) N/A 3) 26 Jun 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	19.9	315	62.64	1	05 Feb 20	N/A	Whole Plant	0.018	96	Mean Recovery
										Straw	0.024	142	Whole Plant 92%
										Grain	(0.009)	142	
				26.7	313	83.52	1	05 Feb 20	N/A	Whole Plant	0.029	96	Straw 88%
										Straw	0.015	142	
										Grain	(0.007)	142	
				41.8	312	130.32	1	05 Feb 20	N/A	Whole Plant	0.583	96	Grain 83%
										Straw	0.100	142	
										Grain	0.053	142	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

272 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Oilseed Rape / Jura	1) 13 Apr 20 2) N/A 3) 15 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	20.0	317	63.36	1	13 Jun 19	N/A	Whole Plant	ND	376	Mean Recovery
										Seed	ND	460	Whole Plant 85%
										Rest of Plant	ND	460	
				26.1	303	79.20	1	13 Jun 19	N/A	Whole Plant	ND	376	Seed 84%
										Seed	ND	460	
										Rest of Plant	ND	460	Rest of Plant 85%
				41.8	288	120.24	1	13 Jun 19	N/A	Whole Plant	ND	376	
										Seed	ND	460	
										Rest of Plant	ND	460	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

271 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Oilseed Rape / Albatros	1) 15 Oct 19 2) N/A 3) 10 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x LDC 110° / 110-02 ASJ nozzles	19.9	308	61.20	1	13 Jun 19	N/A	Whole Plant	ND	328	Mean Recovery
										Seed	ND	393	Whole Plant 85%
										Rest of Plant	ND	393	
				26.7	297	79.20	1	13 Jun 19	N/A	Whole Plant	ND	328	Seed 84%
										Seed	ND	393	
										Rest of Plant	ND	393	
				41.8	293	122.4	1	13 Jun 19	N/A	Whole Plant	ND	328	Rest of Plant 85%
										Seed	ND	393	
										Rest of Plant	ND	393	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

271 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Oilseed Rape / Albatros	1) 15 Oct 19 2) N/A 3) 10 Jul 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	20.0	317	63.36	1	16 Jul 19	N/A	Whole Plant	ND	295	Mean Recovery
										Seed	ND	360	Whole Plant 85%
										Rest of Plant	ND	360	
				26.7	313	83.52	1	16 Jul 19	N/A	Whole Plant	ND	295	Seed 84%
										Seed	ND	360	Rest of Plant 85%
										Rest of Plant	ND	360	
				41.7	285	118.80	1	16 Jul 19	N/A	Whole Plant	ND	295	
										Seed	ND	360	
										Rest of Plant	ND	360	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Agramon, 02490 Albacete (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

301 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009E Agramon, 02490 Albacete, Spain	Oilseed Rape / Jura	1) 03 Mar 20 2) N/A 3) 28 Aug 20	Broadcast spraying directed to the ground, knapsack boom sprayer with 6 x XR TEEJET 110° 110-05 V8 nozzles	20.0	313	62.64	1	05 Feb 20	N/A	Whole Plant Seed Rest of Plant	ND ND ND	110 205 205	Mean Recovery
				26.5	307	81.36	1	05 Feb 20	N/A	Whole Plant Seed Rest of Plant	ND ND ND	110 205 205	Seed 84%
				41.9	313	131.04	1	05 Feb 20	N/A	Whole Plant Seed Rest of Plant	(0.004) ND 0.015	110 205 205	Rest of Plant 85%

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Spain

Lobosillo, 30331 Murcia (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

272 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Radish / Mallorca	1) 04 May 20 2) N/A 3) 26 Jun 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	20.0	306	61.20	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	374 374	Mean Recovery
				26.7	315	84.24	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	374 374	Radish tops with leaves 90%
				41.7	297	123.84	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	374 374	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Root and tuber vegetables

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Radish / RAPSr

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

363 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Radish / Mallorca	1) 18 Oct 19 2) N/A 3) 30 Jan 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	311	61.92	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	226 226	Mean Recovery
				26.6	300	79.92	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	226 226	Radish tops with leaves 90%
				41.7	307	128.16	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	226 226	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Spain

Lobosillo, 30331 Murcia (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

Study no. / DAS Study ID

415 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Radish / Mallorca	1) 20 Sep 19 2) N/A 3) 27 Nov 19	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	304	60.48	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	162 162	Mean Recovery
				26.6	295	78.48	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	162 162	Radish tops with leaves 90%
				41.6	306	127.44	1	18 Jun 19	N/A	Radish roots Radish tops with leaves	ND ND	162 162	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Crop group:

Crop / EPPO code:

Responsible body for reporting (name & address):

Country:

Trial location (region):

Content of active substance (g/kg or g/l):

Formulation number:

Formulation type (e.g. WP):

Clopyralid

Root and tuber vegetables

Radish / RAPSr

Dow AgroSciences, European Development Centre

3B Park Square, Milton Park, Abingdon

Oxon OX14 4RN, UK

Spain

Lobosillo, 30331 Murcia (EU Southern Zone)

720 g ae/L

GF-1966

SG

Commercial Product (name):

Other active substance in the formulation (common name and content):

Producer of commercial product

Study type

Indoor/Glasshouse/Outdoor:

Residues calculated as:

Residue method and LOQ

Max frozen storage time prior to analysis

Study no. / DAS Study ID

GF-1966

None

Dow AgroSciences

Residue

Outdoor

Clopyralid

120610 / 0.01 mg/kg

325 days

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Radish / Mallorca	1) 04 Mar 20 2) N/A 3) 04 May 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° 110- 05 V8 nozzles.	20.0	303	60.48	1	06 Feb 20	N/A	Radish roots Radish tops with leaves	ND (0.004)	88 88	Mean Recovery
				26.7	315	84.24	1	06 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	88 88	Radish tops with leaves 90%
				41.9	289	120.96	1	06 Feb 20	N/A	Radish roots Radish tops with leaves	ND ND	88 88	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

213 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Cabbage / Brunswick	1) 04 May 20 2) N/A 3) 25 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	307	61.20	1	18 Jun 19	N/A	Cabbage	ND	434	Mean Recovery Whole Cabbage 82%
				26.7	307	82.08	1	18 Jun 19	N/A	Cabbage	ND	434	
				41.8	300	125.28	1	18 Jun 19	N/A	Cabbage	ND	434	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

335 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Cabbage / Brunswick	1) 18 Oct 19 2) N/A 3) 25 Feb 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	308	61.20	1	18 Jun 19	N/A	Cabbage	ND	252	Mean Recovery Whole Cabbage 82%
				26.6	306	81.36	1	18 Jun 19	N/A	Cabbage	ND	252	
				41.6	306	127.44	1	18 Jun 19	N/A	Cabbage	ND	252	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

348 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Cabbage / Brunswick	1) 20 Sep 19 2) N/A 3) 30 Jan 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	297	59.04	1	18 Jun 19	N/A	Cabbage	ND	226	Mean Recovery Whole Cabbage 82%
				26.7	307	82.08	1	18 Jun 19	N/A	Cabbage	ND	226	
				41.8	308	128.88	1	18 Jun 19	N/A	Cabbage	ND	226	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Brassicas**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Head Cabbage / BRSOL

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

245 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Cabbage / Brunswick	1) 04 Mar 20 2) N/A 3) 24 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° 110- 05 V8 nozzles.	20.0	310	61.92	1	06 Feb 20	N/A	Cabbage	ND	169	Mean Recovery Whole Cabbage 82%
				26.6	314	83.52	1	06 Feb 20	N/A	Cabbage	ND	169	
				41.8	296	123.84	1	06 Feb 20	N/A	Cabbage	ND	169	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

279 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Barley / Shakira	1) 04 May 20 2) N/A 3) 20 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	20.0	303	60.48	1	18 Jun 19	N/A	Whole Plant	ND	358	Mean Recovery
										Straw	ND	429	Whole Plant 92%
										Grain	ND	429	
				26.7	310	82.80	1	18 Jun 19	N/A	Whole Plant	ND	358	Straw 88%
										Straw	ND	429	
										Grain	ND	429	
				41.9	301	126.00	1	18 Jun 19	N/A	Whole Plant	ND	358	Grain 83%
										Straw	0.016	429	
										Grain	ND	429	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Cereals

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Winter Barley / HORVW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

357 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Barley / Lagalia	1) 10 Dec 19 2) N/A 3) 17 Jun 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° 110- 05 V8 nozzles.	19.9	300	59.76	1	07 Aug 19	N/A	Whole Plant	ND	222	Mean Recovery
										Straw	ND	315	Whole Plant 92%
										Grain	ND	315	
				26.5	296	78.48	1	07 Aug 19	N/A	Whole Plant	ND	222	Straw 88%
										Straw	ND	315	
										Grain	ND	315	Grain 83%
				41.8	308	128.88	1	07 Aug 19	N/A	Whole Plant	ND	222	
										Straw	ND	315	
										Grain	ND	315	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid
Cereals

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Crop group:

Producer of commercial product

Dow AgroSciences

Crop / EPPO code:

Spring Barley / HORVS

Study type

Residue

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Indoor/Glasshouse/Outdoor:

Outdoor

Country:

Spain

Residues calculated as:

Clopyralid

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Residue method and LOQ

120610 / 0.01 mg/kg

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

279 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Barley / Shakira	1) 04 May 20 2) N/A 3) 20 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° 110- 05 V8 nozzles.	20.0	306	61.20	1	06 Feb 20	N/A	Whole Plant	ND	125	Mean Recovery
										Straw	ND	196	Whole Plant 92%
										Grain	ND	196	
				26.6	311	82.80	1	06 Feb 20	N/A	Whole Plant	ND	125	Straw 88%
										Straw	ND	196	
										Grain	ND	196	
				41.7	292	121.68	1	06 Feb 20	N/A	Whole Plant	(0.006)	125	Grain 83%
										Straw	ND	196	
										Grain	ND	196	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Commercial Product (name):

GF-1966

Crop group:

Cereals

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Spring Barley / HORVS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

320 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Barley / Gustav	1) 04 Mar 20 2) N/A 3) 13 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° 110- 05 V8 nozzles.	19.9	307	61.20	1	06 Feb 20	N/A	Whole Plant	(0.004)	84	Mean Recovery
										Straw	0.018	158	Whole Plant 92%
										Grain	(0.006)	158	
				26.6	319	84.96	1	06 Feb 20	N/A	Whole Plant	(0.005)	84	Straw 88%
										Straw	0.027	158	
										Grain	(0.008)	158	Grain 83%
				41.8	286	119.52	1	06 Feb 20	N/A	Whole Plant	0.034	84	
										Straw	0.279	158	
										Grain	0.035	158	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

269 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Oilseed Rape / Katia	1) 04 May 19 2) N/A 3) 10 Sep 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	304	60.48	1	18 Jun 19	N/A	Whole Plant	ND	374	Mean Recovery
										Seed	ND	450	Whole Plant 85%
										Rest of Plant	ND	450	
				26.6	306	81.36	1	18 Jun 19	N/A	Whole Plant	ND	374	Seed 84%
										Seed	ND	450	
										Rest of Plant	ND	450	Rest of Plant 85%
				41.7	307	128.16	1	18 Jun 19	N/A	Whole Plant	ND	374	
										Seed	ND	450	
										Rest of Plant	ND	450	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

300 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Oilseed Rape / Arsenal	1) 18 Oct 19 2) N/A 3) 11 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.9	307	61.20	1	18 Jun 19	N/A	Whole Plant	ND	294	Mean Recovery
										Seed	ND	289	Whole Plant 85% Seed 84% Rest of Plant 85%
										Rest of Plant	ND	389	
				26.6	308	82.08	1	18 Jun 19	N/A	Whole Plant	ND	294	
										Seed	ND	289	
										Rest of Plant	ND	389	
				41.8	296	123.84	1	18 Jun 19	N/A	Whole Plant	ND	294	
										Seed	ND	289	
										Rest of Plant	ND	389	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNW

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

300 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Oilseed Rape / Arsenal	1) 18 Oct 19 2) N/A 3) 11 Jul 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° / 110-015 VS nozzles	19.8	313	61.92	1	17 Jul 19	N/A	Whole Plant	ND	265	Mean Recovery
										Seed	ND	360	Whole Plant 85%
										Rest of Plant	ND	360	
				26.6	319	84.96	1	17 Jul 19	N/A	Whole Plant	ND	265	Seed 84%
										Seed	ND	360	
										Rest of Plant	ND	360	Rest of Plant 85%
				41.7	318	132.48	1	17 Jul 19	N/A	Whole Plant	ND	265	
										Seed	ND	360	
										Rest of Plant	ND	360	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

**Clopyralid
Oilseeds**

Commercial Product (name):

GF-1966

Crop group:

Other active substance in the formulation (common name and content):

None

Crop / EPPO code:

Oilseed Rape / BRSNS

Producer of commercial product

Dow AgroSciences

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Country:

Spain

Trial location (region):

Lobosillo, 30331 Murcia (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Max frozen storage time prior to analysis

303 days

Formulation number:

GF-1966

Study no. / DAS Study ID

CEMS-9009 / 190557

Formulation type (e.g. WP):

SG

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009F Lobosillo, 30331 Murcia, Spain	Oilseed Rape / Katia	1) 04 Mar 20 2) N/A 3) 20 Aug 20	Broadcast spraying directed to the ground, knapsack sprayer with 6 x XR TEEJET 110° 110- 05 V8 nozzles.	20.0	317	63.36	1	06 Feb 20	N/A	Whole Plant	ND	107	Mean Recovery
										Seed	ND	196	Whole Plant 85%
										Rest of Plant	ND	196	
				26.6	308	82.08	1	06 Feb 20	N/A	Whole Plant	ND	107	Seed 84%
										Seed	ND	196	
										Rest of Plant	ND	196	Rest of Plant 85%
				41.7	283	118.08	1	06 Feb 20	N/A	Whole Plant	ND	107	
										Seed	ND	196	
										Rest of Plant	(0.009)	196	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

DAA = Days after application

RESIDUES DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Active substance (common name):

Clopyralid

Crop group:

Cereals

Crop / EPPO code:

Spring Wheat / TRZAS

Responsible body for reporting (name & address):

Dow AgroSciences, European Development Centre
3B Park Square, Milton Park, Abingdon
Oxon OX14 4RN, UK

Country:

Spain

Trial location (region):

Conil de la Frontera, 11140 Cádiz (EU Southern Zone)

Content of active substance (g/kg or g/l):

720 g ae/L

Formulation number:

GF-1966

Formulation type (e.g. WP):

SG

Commercial Product (name):

GF-1966

Other active substance in the formulation (common name and content):

None

Producer of commercial product

Dow AgroSciences

Study type

Residue

Indoor/Glasshouse/Outdoor:

Outdoor

Residues calculated as:

Clopyralid

Residue method and LOQ

120610 / 0.01 mg/kg

Max frozen storage time prior to analysis

274 days

Study no. / DAS Study ID

CEMS-9009 / 190557

1	2	3	4	5			6		7	8	9	10	11
Trial No. Location (region)	Commodity/Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Method of Treatment	Application rate per treatment			No. of trt(s)	Dates of treatments	Growth stage at treatment	Portion analysed (commodity)	Residues (mg/kg)	DAA	Remarks:
	(a)	(b) – if relevant	(c)	g ae/hL	Water (L/ha)	g ae/ha	(d)		(e)	(a)		(days) (f)	(g)
CEMS-9009G Conil de la Frontera 11140 Cádiz, Spain	Wheat / Gazul	1) 15 Jul 20 2) N/A 3) 10 Jun 21	Broadcast spraying directed to the ground, knapsack sprayer with 9 x Anti-drift Flat Fan Nozzles ASJ CFA-01 (Orange)	28.3	218	61.769	1	13 Mar 20	N/A	Whole Plant	ND	185	Mean Recovery
										Straw	ND	454	Whole Plant 92%
										Grain	ND	454	
				38.0	211	80.086	1	13 Mar 20	N/A	Whole Plant	ND	185	Straw 95%
										Straw	ND	454	
										Grain	ND	454	Grain 91%
				59.5	219	130.262	1	13 Mar 20	N/A	Whole Plant	ND	185	
										Straw	ND	454	
										Grain	ND	454	

(a) According to EEC and Codex classifications (both) should be used.

(b) Only if relevant.

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, -type of equipment must be indicated.

(d) Year must be indicated.

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-429

(f) Minimum number of days after last application

(g) Remarks may include: climatic conditions, references to analytical method, info concerning the metabolites Included, method of storage, storage stability, analysis date and analytical method.

Residues of less than the LOQ, but greater than 0.003 mg/kg, are shown in parentheses, ND = Not detected (<0.003 mg/kg).

A 2.1.7 Other/Special Studies

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

The applicant provided the following study for determination of residues in honey.

Study 1: S20-00871

Comments of zRMS:	<p>The residue study was conducted in 2020 in N-EU (4 trials in Northern and Southern Germany, Romania, and The Netherlands) and in S-EU (2 trials in Southern France and Spain) to determine the residue concentration of clopyralid in winter oilseed rape plants, pollen, nectar and honey following one application of GF-1966 (125 g a.e./ha; 173.85 g product/ha) under semi-field conditions to winter oilseed rape. The rate used in the study was approximately 1N of the proposed use rate for oilseeds.</p> <p>The maximum period of frozen storage from the day of sampling until extraction was 313 days for matrices honey, pollen, nectar, and plant.</p> <p>The analytical method was validated in all matrices according to SANTE/2020/12830, Rev.1, 24. February 2021.</p> <p>The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg for all matrices.</p> <p>The mean recovery at each fortification level was in the range of 70 - 110% with a relative standard deviation of < 20% of all analytes in all tested matrices and thus comply with the standard acceptance criteria of the guidance document SANTE/2020/12830, Rev.1, 24. February 2021.</p> <p>The analyte was determined in the final specimen extracts by use of LC-MS/MS detection.</p> <p>The residues in honey were 3x <0.01, 0.0179, 0.0274, 0.0695 mg/kg.</p> <p>Maximum residues of clopyralid in plant samples were at 3.09 mg/kg.</p> <p>Maximum residues in pollen samples were at 3.30 mg/kg and in nectar samples at 2.71 mg/kg, respectively.</p> <p>The study is acceptable.</p>
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Data point	Point 7.2.1
Report author	Appeltauer A.
Report year	2021
Report title	Determination of Residues of Clopyralid in Nectar, Pollen, Plants and Honey of Winter Oilseed Rape after One Application of GF-1966 in a Semi-Field Residue Study in Germany, Romania, The Netherlands, Southern France and Spain in 2020
Study code	S20-00871
Study number	DAS 200098
Guidelines followed in study	7029/VI/95 (rev. 5) to Directive 91/414/EEC, Reg. (EU) 283/2013 and 284/2013, SANTE/11956/2016 rev. 9
Major deviations from test guideline	None
Previous evaluation	None
GLP/Officially recognised testing facilities	Yes
Acceptability/Reliability	Yes

Materials and methods:

Materials:

Analytical standards:

Analyte	Clopyralid
Lot/batch	200901001

Purity	99.1%
CAS	1702-17-6
Expiry date	21.6.2023

Test formulation:

Description: GF-1966 / formulation SG
Lot/Batch #: D062EAKA04
Content: 94.7 % w/w clopyralid-olamine (947 g/kg) / 71.9 % w/w acid equivalent (719 g/kg)
Expiry date: 8.1.2021

Study design:

Test procedure

The study was conducted as six separate field trials in Northern and Southern Germany, Romania, The Netherlands, Southern France and Spain in 2020. The distance between trials was at least 297 km. The study consisted of two treatment groups per trial: the test item group T (2 replicates- Ta and Tb) and an untreated control C (1 replicate). Each trial consisted of 3 tunnels/plots (Control C, Test item treatment replicate Ta and replicate Tb). The area covered per tunnel was 200 m² for all trials (see Table 3). The approximate dimensions of each tunnel were 40 m length, 5 m width and 3 - 3.5 m height in the center. The tunnels were covered with light plastic gauze (mesh size 1.5 and 1.67 mm). In tunnels C and Ta one colony per tunnel was set up. In tunnel Tb two colonies were set up. The colonies were set up in the tunnels 1-2 days before sampling S6 (equivalent to 9 to 24 days after application). Honeybee colonies (*Apis mellifera* L. (Hymenoptera, Apidae) with a sufficient number of forager bees were used.

There was one application in the test item treatment group at a target rate of 125 g a.e./ha (Ta, Tb; 173.85 g product/ha, analysed) at BBCH 55 for trials -02, -03, -05, -06 and -07 and BBCH 57 for trial -04.

Winter oilseed rape plants, pollen and forager bees for preparation of nectar were collected in the control tunnel and in replicate Tb. Honey was collected in the control tunnel and in replicate Ta. Winter oilseed rape plants were collected six times after application. Sampling of plants for residue analysis was conducted first at 0DAA and last sampling 10-25DAA. Pollen and forager bees were collected six times after application. Sampling of pollen and forager bees for preparation of nectar for residue analysis was conducted first at 10-25DAA and last sampling 17-34DAA.

For plants samples on each sampling day, at least 500 g sample was collected.

From sampling S6 to sampling S11 forager bees were collected for the preparation of nectar from their honey stomachs for residue analysis in replicate Tb. At sampling S6 forager bees were also collected from the control tunnel. On each sampling day an A-sample of at least 300 bees was collected.

From sampling S6 to sampling S11 on each sampling day pollen from winter oilseed rape flowers retrieved by the bees was collected using pollen traps in replicate Tb. At sampling S6 forager bees were also collected from the control tunnel. The hives in the tunnel were equipped with pollen traps.

Honey was collected once mature at the end of flowering or if the water content was < 20 % or after comb closure – whatever occurred first - for subsequent residue analysis in replicate Ta. For honey samples an A1- and, where sufficient sample material was available, an R1-sample of preferable 100g was targeted for sampling, with a minimum of at least 10 g honey as a representative sample of the tunnel area, was collected.

Analytical procedures

Prior sample analysis, the analytical methods were validated in all matrices according to SANCO/3029/99, rev. 4 within the analytical phase. Mean recoveries at each fortification level were in the range of 70 - 110% with relative standard deviation(s) below 20% for all matrices. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg for all matrices with a limit of detection

(LOD) set at 0.003 mg/kg (30 % of the LOQ). The method was considered to be acceptably validated and fit for purpose. No ILV for the method was available.

Hydrolysis of whole plants, honey, nectar and pollen samples were performed based on the analytical method as described in the Sponsor study with ID 120610. Sample workup was modified depending on matrix in the current study and was fully validated for all matrices according to SANCO/3029/99 rev. 4. Quantification was performed by use of LC-MS/MS detection.

Results and discussion:

Residues in honey, nectar, pollen and plants are shown in the table below:

Trial details		Crop	Country	Formulation, Application rate (g a.s./ha)	Crop growth stage2 (BBCH)	DALAI	Residues found (mg/kg)	
							Matrix	clopyralid
Study code: Doc ID: Trial No: GLP: Year:	S20-00871 02 Yes 2020	Oilseed rape	Germany	GF-1966 SG Ta: 127.7	55	35	honey	<0.01
				GF-1966 SG Tb: 125.5	55	0	plant	1.51
						1	plant	1.78
						2	plant	2.15
						4	plant	1.84
						6	plant	1.19
						18	pollen	1.04
							plant	1.04
							nectar	0.0760
						19	pollen	0.808
							nectar	0.0555
						20	pollen	0.620
							nectar	0.0386
						23	pollen	0.0930
							nectar	0.0535
Study code: Doc ID: Trial No: GLP: Year:	S20-00871 03 Yes 2020	Oilseed rape	Romania	GF-1966 SG Ta: 136.1	55	21	honey	0.0179
				GF-1966 SG Tb: 134.8	55	0	plant	0.944
						1	plant	0.168
						3	plant	0.218
						4	plant	0.358
						7	plant	0.433
						10	pollen	0.550
							plant	0.337
							nectar	0.0675
						11	pollen	0.453
							nectar	0.0333
						12	pollen	2.61
							nectar	0.120
						14	pollen	0.249
							nectar	0.0255
						16	pollen	0.310
							nectar	0.0325
						17	pollen	0.169
							nectar	0.0253

Trial details		Crop	Country	Formulation, Application rate (g a.s./ha)	Crop growth stage2 (BBCH)	DALAI	Residues found (mg/kg)	
							Matrix	clopyralid
Study code: Doc ID: Trial No: GLP: Year:	S20-00871	Oilseed rape	The NL	GF-1966 SG Ta: 127.2	57	42	honey	<0.01
				GF-1966 SG Tb: 127.2	57	0	plant	1.45
						1	plant	0.926
						3	plant	1.06
						5	plant	1.05
						7	plant	0.749
						25	pollen	2.35
							plant	1.25
							nectar	0.019
						26	pollen	1.74
							nectar	0.0510
						27	pollen	1.65
							nectar	0.0422
						29	pollen	0.663
							nectar	0.0800
Study code: Doc ID: Trial No: GLP: Year:	S20-00871	Oilseed rape	France (SEU)	GF-1966 SG Ta: 127.4	55	40	honey	0.0695
				GF-1966 SG Tb: 126.4	55	0	plant	2.33
						1	plant	2.30
						2	plant	1.44
						5	plant	3.09
						6	plant	3.02
						12	pollen	3.30
							plant	2.12
							nectar	0.479
						13	pollen	3.17
							nectar	0.520
						14	pollen	0.260
							nectar	1.31
						16	pollen	1.39
							nectar	1.70
						18	pollen	1.68
							nectar	1.58
						20	pollen	1.30
							nectar	1.31
Study code: Doc ID: Trial No: GLP: Year:	S20-00871	Oilseed rape	Spain	GF-1966 SG Ta: 124.1	55	49	honey	<0.01
				GF-1966 SG Tb: 133.5	55	0	plant	0.686
						1	plant	1.16
						2	plant	1.48
						4	plant	1.19
						7	plant	2.02
						14	pollen	1.72
							plant	1.56
							nectar	0.0169
						15	pollen	1.69
							nectar	0.0860
						17	pollen	1.71
							nectar	0.295

Trial details		Crop	Country	Formulation, Application rate (g a.s./ha)	Crop growth stage2 (BBCH)	DALAI	Residues found (mg/kg)	
							Matrix	clopyralid
						18	pollen	1.52
							nectar	0.165
						19	pollen	1.73
							nectar	0.358
						21	pollen	0.855
							nectar	0.157
Study code: Doc ID: Trial No: GLP:	S20-00871 07 Yes	Oilseed rape	Germany	GF-1966 SG Ta: 126.1	55	17	honey	0.0274
				GF-1966 SG Tb: 126.4	55	0	plant	1.49
						1	plant	1.69
						3	plant	1.52
						5	plant	1.07
						7	plant	1.29
						11	pollen	1.33
							plant	1.16
							nectar	0.338
						12	pollen	1.14
							nectar	2.05
						13	pollen	0.610
							nectar	2.71
						15	pollen	0.530
							nectar	0.815
						17	pollen	0.360
							nectar	0.0585
						19	pollen	0.408
							nectar	0.0690

1 Days after last application

2 At last application

Underlined values were used for MRL calculation

The residues in honey were 3x <0.01, 0.0179, 0.0274, 0.0695 mg/kg. Calculated STMR is 0.014 mg/kg and unrounded MRL 0.117 mg/kg (rounded 0.15 mg/kg). Current MRL for clopyralid in honey is 0.05 mg/kg and thus potentially exceeded.

No residues of clopyralid were detected above LOD (0.003 mg/kg) in untreated plant, pollen, nectar and honey specimen, with one exception: In untreated honey specimen of trial -02, residues of clopyralid were determined at 0.0179 mg/kg.

Storage stability

Maximum storage of samples were following:

Honey samples: 222 days; honey extracts 1 day

Pollen samples: 194 days; pollen extracts 3 days

Nectar samples: 15 days; nectar extracts 1 day

Whole plant samples: 188 days; plant extracts 6 days

Maximum storage time of the samples in the test facility was 187 days before dispatch. This should be added to the storage times. Storage times are acceptable when compared to the demonstrated stability of clopyralid in honey (18 months – approx. 540 days).


Stability of residues in extracts of pollen, nectar, whole plant and honey was shown to be at least 12 days in the dark for pollen, and 34 days for whole plants, 8 days for honey and 12 days for nectar.

Appendix 3 Pesticide Residue Intake Model (PRIMo)

A 3.1 TMDI calculations

Normal mode											
Chronic risk assessment: JMPR methodology (IEDI/TMDI)											
			No of diets exceeding the ADI : ---					Exposure resulting from			
	Cal- cu- lated expo- sure (% of ADI)	MS Diet	Expsoure (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commod- ities	2 nd con- tribu- tor to MS diet (in % of ADI)	Commodity / group of commodi- ties	3 rd con- tribu- tor to MS diet (in % of ADI)	Commodity / group of com- modities	MRLs set at the LOQ (in % of ADI)	commodities not under assessment (in % of ADI)
TMDI/IEDI calculation (based on average food consumption)	42.3%	NL toddler	63.42	9.4%	Maize/corn	7.9%	Wheat	3.6%	Apples	0.1%	25.3%
	27.0%	GEMS/Food G06	40.56	14.5%	Wheat	2.1%	Rice	1.7%	Maize/corn	0.1%	18.2%
	26.1%	DK child	39.17	11.0%	Rye	8.8%	Wheat	0.8%	Potatoes	0.1%	21.0%
	24.9%	NL child	37.28	8.2%	Wheat	5.6%	Sugar beet roots	1.9%	Apples	0.1%	15.8%
	23.8%	DE child	35.64	8.4%	Wheat	4.2%	Apples	1.6%	Rye	0.1%	11.6%
	20.9%	RO general	31.29	10.1%	Wheat	2.9%	Head cabbages	1.3%	Maize/corn	0.1%	15.5%
	20.3%	GEMS/Food G15	30.41	9.1%	Wheat	1.6%	Head cabbages	1.2%	Potatoes	0.1%	14.0%
	20.0%	FR child 3 15 yr	30.05	9.2%	Wheat	2.4%	Sugar beet roots	1.1%	Oranges	0.1%	13.3%
	19.8%	GEMS/Food G08	29.63	8.2%	Wheat	1.3%	Potatoes	1.2%	Barley	0.1%	13.1%
	19.4%	GEMS/Food G10	29.13	7.8%	Wheat	1.7%	Rice	1.1%	Soyabeans	0.1%	11.4%
	18.4%	IT toddler	27.67	13.3%	Wheat	2.0%	Other cereals	0.5%	Tomatoes		13.6%
	18.1%	GEMS/Food G07	27.22	8.4%	Wheat	1.3%	Potatoes	0.8%	Barley	0.1%	11.3%
	18.1%	IE adult	27.10	4.6%	Wheat	2.4%	Sweet potatoes	2.0%	Linseeds	0.0%	9.0%
	17.1%	GEMS/Food G11	25.71	7.2%	Wheat	1.3%	Potatoes	1.2%	Soyabeans	0.1%	9.4%
	16.7%	UK toddler	25.12	7.8%	Wheat	2.1%	Sugar beet roots	1.2%	Potatoes	0.0%	10.8%
	16.0%	FR toddler 2 3 yr	23.95	6.2%	Wheat	1.9%	Sugar beet roots	1.3%	Milk: Cattle	0.1%	9.2%
	15.6%	UK infant	23.43	5.2%	Wheat	1.7%	Milk: Cattle	1.4%	Maize/corn	0.1%	9.0%
	15.2%	PT general	22.77	7.8%	Wheat	1.8%	Potatoes	1.0%	Rice		9.0%
	15.1%	ES child	22.63	8.9%	Wheat	0.7%	Oranges	0.6%	Rice	0.1%	9.8%
	15.0%	SE general	22.54	6.4%	Wheat	1.4%	Potatoes	1.3%	Head cabbages	0.0%	8.9%
	14.2%	DE women 14-50 yr	21.31	4.3%	Wheat	3.1%	Sugar beet roots	1.0%	Rye	0.1%	9.6%
	13.8%	DE general	20.76	3.8%	Wheat	2.8%	Sugar beet roots	1.2%	Rye	0.1%	9.5%
	12.1%	IT adult	18.12	8.3%	Wheat	0.9%	Other cereals	0.4%	Tomatoes		8.6%
	11.9%	NL general	17.86	3.9%	Wheat	1.9%	Sugar beet roots	0.8%	Potatoes	0.1%	7.6%
	10.7%	FI 3 yr	16.07	2.4%	Wheat	1.6%	Potatoes	1.3%	Rye	0.0%	5.7%
	9.4%	ES adult	14.04	4.7%	Wheat	0.7%	Barley	0.4%	Oranges	0.1%	5.9%
	9.1%	FR adult	13.62	4.4%	Wheat	0.8%	Wine grapes	0.5%	Sugar beet roots	0.1%	5.7%
	8.5%	LT adult	12.81	2.2%	Rye	2.1%	Wheat	1.1%	Potatoes	0.1%	5.4%
	8.4%	FI 6 yr	12.55	1.9%	Wheat	1.3%	Potatoes	1.2%	Rye	0.0%	4.5%
	8.3%	UK vegetarian	12.51	4.1%	Wheat	0.5%	Rice	0.5%	Potatoes	0.0%	5.3%

[illegible]

 <p>European Food Safety Authority</p> <p>EFSA PRIMo revision 3.1; 2019/03/19</p>		clopyralid				Input values							
		LOQs (mg/kg) range from: _____ to: _____											
		Toxicological reference values								Details - chronic risk assessment		Supplementary results - chronic risk assessment	
		ADI (mg/kg bw/day): 0,15				ARID (mg/kg bw): 0,17				Details - acute risk assessment/children		Details - acute risk assessment/adults	
Source of ADI: EFSA 2018		Year of evaluation: _____		Source of ARID: EFSA 2018		Year of evaluation: _____							
Comments:													
Normal mode													
Chronic risk assessment: JMPR methodology (IED/TMDI)													
				No of diets exceeding the ADI : _____								Exposure resulting from	
	Calculated exposure (% of ADI)	MS Diet	Exposure (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commodities	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities	MRLs set at the LOQ (in % of ADI)	commodities not under assessment (in % of ADI)		
TMDI/NEDI calculation (based on average food consumption)	43%	NL toddler	64,28	9%	Maize/corn	8%	Wheat	4%	Apples				
	33%	DK child	50,22	18%	Rye	9%	Wheat	0,8%	Potatoes				
	27%	GEMS/Food G06	40,64	14%	Wheat	2%	Rice	2%	Maize/corn				
	25%	NL child	37,66	8%	Wheat	6%	Sugar beet roots	2%	Apples				
	25%	DE child	37,25	8%	Wheat	4%	Apples	3%	Rye				
	21%	RO general	31,30	10%	Wheat	3%	Head cabbages	1%	Maize/corn				
	21%	GEMS/Food G15	30,89	9%	Wheat	2%	Head cabbages	1%	Potatoes				
	21%	GEMS/Food G08	30,82	8%	Wheat	2%	Rye	1%	Potatoes				
	20%	FR child 3 15 yr	30,12	9%	Wheat	2%	Sugar beet roots	1%	Oranges				
	20%	GEMS/Food G10	29,37	8%	Wheat	2%	Rice	1%	Soyabeans				
	18%	IT toddler	27,67	13%	Wheat	2%	Other cereals	0,5%	Tomatoes				
	18%	IE adult	27,44	5%	Wheat	2%	Sweet potatoes	2%	Linseeds				
	18%	GEMS/Food G07	27,36	8%	Wheat	1%	Potatoes	0,8%	Barley				
	17%	GEMS/Food G11	25,78	7%	Wheat	1%	Potatoes	1%	Soyabeans				
	17%	UK toddler	25,18	8%	Wheat	2%	Sugar beet roots	1%	Potatoes				
	16%	FR toddler 2 3 yr	24,00	6%	Wheat	2%	Sugar beet roots	1%	Milk: Cattle				
	16%	UK infant	23,47	5%	Wheat	2%	Milk: Cattle	1%	Maize/corn				
	16%	SE general	23,26	6%	Wheat	1%	Potatoes	1%	Head cabbages				
	15%	PT general	23,04	8%	Wheat	2%	Potatoes	1%	Rice				
	15%	ES child	22,68	9%	Wheat	0,7%	Oranges	0,6%	Rice				
	15%	DE women 14-50 yr	22,28	4%	Wheat	3%	Sugar beet roots	2%	Rye				
	15%	DE general	21,93	4%	Wheat	3%	Sugar beet roots	2%	Rye				
	12%	IT adult	18,12	8%	Wheat	0,9%	Other cereals	0,4%	Tomatoes				
	12%	NL general	18,02	4%	Wheat	2%	Sugar beet roots	0,8%	Potatoes				
	12%	FI 3 yr	17,37	2%	Wheat	2%	Rye	2%	Potatoes				
	10%	LT adult	14,97	4%	Rye	2%	Wheat	1%	Potatoes				
	9%	ES adult	14,07	5%	Wheat	0,7%	Barley	0,4%	Oranges				
	9%	FI 6 yr	13,78	2%	Rye	2%	Wheat	1%	Potatoes				
	9%	FR adult	13,65	4%	Wheat	0,8%	Wine grapes	0,5%	Sugar beet roots				
	8%	UK vegetarian	12,55	4%	Wheat	0,5%	Rice	0,5%	Potatoes				
	8%	FI adult	11,39	2%	Rye	2%	Coffee beans	0,6%	Wheat				
	7%	FR infant	10,69	2%	Wheat	0,9%	Sugar beet roots	0,7%	Milk: Cattle				
	7%	DK adult	10,51	2%	Wheat	2%	Rye	0,4%	Potatoes				
	7%	UK adult	10,41	3%	Wheat	0,5%	Rice	0,5%	Potatoes				
4%	PL general	6,17	1%	Potatoes	0,7%	Head cabbages	0,7%	Apples					
4%	IE child	5,69	2%	Wheat	0,4%	Rice	0,2%	Potatoes					
Conclusion: The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI. The long-term intake of residues of clopyralid is unlikely to present a public health concern.													

IESTI calculations – Raw commodities

Show results of IESTI calculation only for crops with GAPs under assessment																
Unprocessed commodities	Results for children No. of commodities for which ARfD/ADI is exceeded (IESTI):				Results for adults No. of commodities for which ARfD/ADI is exceeded (IESTI):											
	---				---											
	IESTI				IESTI											
	Highest % of ARfD/ADI		MRL / input for RA (mg/kg)		Exposure (µg/kg bw)		Highest % of ARfD/ADI		MRL / input for RA (mg/kg)		Exposure (µg/kg bw)					
	Commodities						Commodities									
	45.64%	Swedes/rutabagas	1.5 / 1.5	77.59	30.13%	Swedes/rutabagas	1.5 / 1.5	51.23	33.57%	Beetroots	1 / 1	57.06	22.51%	Head cabbages	3 / 0.91	38.27
	31.70%	Turnips	1.5 / 1.5	53.88	14.83%	Wheat	3 / 3	25.21	28.97%	Cauliflowers	3 / 0.85	49.24	13.54%	Beetroots	1 / 1	23.02
	25.50%	Wheat	3 / 3	43.35	11.92%	Broccoli	1.5 / 0.85	20.26	23.68%	Head cabbages	3 / 0.91	40.26	11.59%	Cauliflowers	3 / 0.85	19.71
	20.80%	Broccoli	1.5 / 0.85	35.36	11.12%	Chards/beet leaves	1 / 1	18.90	13.29%	Spinaches	1 / 1	22.60	9.83%	Turnips	1.5 / 1.5	16.71
	12.60%	Linseeds	20 / 20	21.42	8.56%	Rye	3 / 3	14.56	11.16%	Rye	3 / 3	18.97	5.69%	Barley	2 / 2	9.67
	9.18%	Chards/beet leaves	1 / 1	15.60	5.65%	Linseeds	20 / 20	9.60	7.92%	Maize/corn	2 / 2	13.47	4.37%	Onions	0.5 / 0.5	7.43
	6.69%	Onions	0.5 / 0.5	11.37	2.55%	Maize/corn	2 / 2	4.34	6.60%	Barley	2 / 2	11.22	2.35%	Spinaches	1 / 1	4.00
	6.46%	Spring onions/green onions and Welsh onions	0.7 / 0.7	10.98	1.85%	Spring onions/green onions and Welsh onions	0.7 / 0.7	3.15								
	Expand/collapse list															
	Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)															

Acute risk assessment /children					Acute risk assessment / adults / general population					Acute risk assessment /children					Acute risk assessment / adults / general population					
Details - acute risk assessment /children					Details - acute risk assessment/adults					Hide IESTI new calculations					Show IESTI new calculations					
	The acute risk assessment is based on the ARID. The calculation is based on the large portion of the most critical consumer group.										IESTI new calculations: The calculation is performed with the MRL and the peeling/processing factor (PF), taking into account the residue in the edible portion and/or the conversion factor for the residue definition (CF). For case 2a, 2b and 3 calculations a variability factor of 3 is used. Since this methodology is not based on internationally agreed principles, the results are considered as indicative only. Since this methodology is not based on internationally agreed principles, the results are considered as indicative only.									
	Show results for all crops																			
Unprocessed commodities	Results for children				Results for adults				IESTI new Results for children				IESTI new Results for adults							
	No. of commodities for which ARID/ADI is exceeded (IESTI):				No. of commodities for which ARID/ADI is exceeded (IESTI):				No. of commodities for which ARID/ADI is exceeded (IESTI new):				No. of commodities for which ARID/ADI is exceeded (IESTI new):							
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	IESTI				IESTI				IESTI new				IESTI new							
	Highest % of ARID/ADI		MRL / input for RA (mg/kg)		Exposure (µg/kg bw)		Highest % of ARID/ADI		MRL / input for RA (mg/kg)		Exposure (µg/kg bw)		Highest % of ARID/ADI		MRL / input for RA (mg/kg)		Exposure (µg/kg bw)			
	46%	Swedes/rutabagas	1,5 / 1,5	78	30%	Swedes/rutabagas	1,5 / 1,5	51	27%	Swedes/rutabagas	1,5 / 1,5	47	18%	Swedes/rutabagas	1,5 / 1,5	31				
	34%	Beetroots	1 / 1	57	14%	Beetroots	1 / 1	23	14%	Beetroots	1 / 1	24	6%	Beetroots	1 / 1	10				
	32%	Turnips	1,5 / 1,5	54	10%	Turnips	1,5 / 1,5	17	14%	Turnips	1,5 / 1,5	23	6%	Linseeds	20 / 20	9,6				
	13%	Linseeds	20 / 20	21	6%	Linseeds	20 / 20	9,6	13%	Linseeds	20 / 20	21	5%	Turnips	1,5 / 1,5	8,2				
	7%	Onions	0,5 / 0,5	11	4%	Onions	0,5 / 0,5	7,4	5%	Milk: Cattle	0,05 / 0,07	8,1	2%	Onions	0,5 / 0,5	3,2				
	5%	Milk: Cattle	0,05 / 0,07	8,1	2%	Bovine: Kidney	1,5 / 1,5	3,2	3%	Bovine: Kidney	1,5 / 1,5	5,6	2%	Bovine: Kidney	1,5 / 1,5	3,2				
	3%	Bovine: Kidney	1,5 / 1,5	5,6	1%	Milk: Cattle	0,05 / 0,07	2,5	3%	Onions	0,5 / 0,5	4,9	1%	Milk: Cattle	0,05 / 0,07	2,5				
	0,9%	Milk: Goat	0,05 / 0,07	1,6	0,8%	Swine: Kidney	0,6 / 0,6	1,3	0,9%	Milk: Goat	0,05 / 0,07	1,6	0,8%	Swine: Kidney	0,6 / 0,6	1,3				
	0,7%	Bovine: Liver	0,15 / 0,15	1,2	0,7%	Milk: Goat	0,05 / 0,07	1,2	0,7%	Bovine: Liver	0,15 / 0,15	1,2	0,7%	Milk: Goat	0,05 / 0,07	1,2				
	0,5%	Poultry: Muscle/meat	0,05 / 0,05	0,85	0,6%	Milk: Sheep	0,05 / 0,07	0,98	0,5%	Poultry: Muscle/meat	0,05 / 0,05	0,85	0,6%	Milk: Sheep	0,05 / 0,07	0,98				
0,4%	Swine: Kidney	0,6 / 0,6	0,76	0,4%	Bovine: Liver	0,15 / 0,15	0,60	0,4%	Swine: Kidney	0,6 / 0,6	0,76	0,4%	Bovine: Liver	0,15 / 0,15	0,60					
0,4%	Rapeseeds/canola	0,5 / 0,5	0,69	0,3%	Poultry: Muscle	0,05 / 0,05	0,59	0,4%	Rapeseeds/canola	0,5 / 0,5	0,69	0,3%	Poultry: Muscle	0,05 / 0,05	0,59					
0,4%	Eggs: Chicken	0,05 / 0,05	0,62	0,3%	Sheep: Liver	0,2 / 0,2	0,56	0,4%	Eggs: Chicken	0,05 / 0,05	0,62	0,3%	Sheep: Liver	0,2 / 0,2	0,56					
0,4%	Swine: Muscle/meat	0,05 / 0,05	0,61	0,3%	Bovine: Muscle	0,08 / 0,08	0,46	0,4%	Swine: Muscle/meat	0,05 / 0,05	0,61	0,3%	Bovine: Muscle	0,08 / 0,08	0,46					
0,3%	Bovine: Muscle/meat	0,08 / 0,08	0,58	0,2%	Sheep: Muscle/meat	0,08 / 0,08	0,38	0,3%	Bovine: Muscle/meat	0,08 / 0,08	0,58	0,2%	Sheep: Muscle/meat	0,08 / 0,08	0,38					
Expand/collapse list																				
Total number of commodities exceeding the ARID/ADI in children and adult diets (IESTI calculation)								Total number of commodities found exceeding the ARID/ADI in children and adult diets (IESTI new calculation)												

A 3.2 IESTI calculations - Processed commodities

Processed commodities	Results for children				Results for adults			
	No of processed commodities for which ARfD/ADI is exceeded (IESTI):				No of processed commodities for which ARfD/ADI is exceeded (IESTI):			
	---				---			
	IESTI				IESTI			
	Highest % of ARfD/ADI	Processed commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARfD/ADI	Processed commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
	64.8%	Sugar beets (root) / sugar	1 / 12	110.15	25.8%	Sugar beets (root) / sugar	1 / 12	43.80
	44.7%	Turnips / boiled	1.5 / 1.5	76.07	22.88%	Beetroots / boiled	1 / 1	38.89
	39.4%	Broccoli / boiled	1.5 / 0.85	66.96	20.83%	Cauliflowers / boiled	3 / 0.85	35.41
	34.8%	Cauliflowers / boiled	3 / 0.85	59.17	16.84%	Turnips / boiled	1.5 / 1.5	28.63
	27.4%	Maize / oil	2 / 50	46.57	14.94%	Maize / oil	2 / 50	25.40
	26.1%	Beetroots / boiled	1 / 1	44.33	12.04%	Broccoli / boiled	1.5 / 0.85	20.46
	21.3%	Wheat / milling (flour)	3 / 3	36.27	8.47%	Barley / beer	2 / 0.4	14.40
	18.3%	Chards/beet leaves / boiled	1 / 1	31.12	7.75%	Wheat / bread/pizza	3 / 3	13.17
	9.8%	Wheat / milling (wholemeal)-baking	3 / 3	16.63	7.36%	Chards/beet leaves / boiled	1 / 1	12.52
	8.2%	Spinaches / frozen; boiled	1 / 1	13.91	6.73%	Wheat / pasta	3 / 3	11.44
	6.4%	Rye / boiled	3 / 3	10.88	6.17%	Wheat / bread (whole-meal)	3 / 3	10.48
	6.4%	Oat / boiled	3 / 3	10.88	4.87%	Spinaches / frozen; boiled	1 / 1	8.28
	6.2%	Rye / milling (wholemeal)-baking	3 / 3	10.53	2.77%	Onions / boiled	0.5 / 0.5	4.71
	5.3%	Oat / milling (flakes)	3 / 3	9.00	2.68%	Oat / boiled	3 / 3	4.56
	4.8%	Shallots / boiled	0.5 / 0.5	8.11	1.82%	Shallots / boiled	0.5 / 0.5	3.10
	Expand/collapse list							

Conclusion:

No exceedance of the toxicological reference value was identified for any unprocessed commodity.
A short term intake of residues of Clopyralid is unlikely to present a public health risk.
For processed commodities, no exceedance of the ARfD/ADI was identified.

[illegible]

Appendix 4 Additional information provided by the applicant

zRMS comments:

Additional data on on metabolism of clopyralid in plants has been accepted.

There are two separate processes in which the applicant (Dow AgroSciences, DAS, now Corteva Agriscience), the RMS (FI) and EFSA have communicated to address identified data gaps in the metabolism data package. These are (i) the renewal of approval of clopyralid and (ii) an Article 6 MRL evaluation report, submitted 31 May 2018, updated 14 November 2018, 25 January 2019, 03 May 2019, 14 November 2019 and 17 August 2020.

Additional information on metabolism of clopyralid in plants (RAR, Finland, revised May 2019)

Upon the framework of Art.6 MRL review for clopyralid, the applicant submitted further explanation and data on the metabolism in plants due to requests of EFSA to clarify the nature of the “polar clopyralid” / “metabolite A” detected in OSR and sugar beet metabolism studies. It was specifically requested that an explanation should be provided why in mature samples of sugar beet and oilseed rape ‘polar clopyralid’ and clopyralid were observed, while ‘polar clopyralid’ was not observed in immature samples, as the analytical procedure used was always the same within one study. In addition, EFSA requested further clarification on the nature of the unknown “metabolite B” in the plant metabolism studies, and explanation whether it is identical with the “polar form of clopyralid” or “clopyralid conjugates”.

Metabolite A / “polar clopyralid”

The applicant explained that “polar clopyralid” is the same throughout these studies and is also known as “unknown A” and “unknown 1”. It is observed only in mature OSR straw and seed and mature sugar beet shoots (foliage) and roots. It is the same in both studies where it has been identified as such. Although the extraction procedures are identical, the mature samples were concentrated and reconstituted in 1:1 ACN:H₂O, which would be less likely to retain clopyralid than the 28-d immature samples which were not concentrated and would contain water and therefore contain >50% water in the injected sample. Between the higher organic content and the weaker acid used in HPLC system 1, clopyralid would be less likely to be retained.

The analytical conditions were further explained by the applicant:

“Although the extraction analytical procedures were identical within a study, and similar between OSR and sugar beet metabolism studies, the chromatography was different between immature and mature samples. The samples analyzed by HPLC were of the same general composition, nominally 50/50, ACN/H₂O (v/v). However, after carefully reviewing the raw data of these studies (finalized in 2002), it was determined that a major, and very important difference, was that the HPLC injection volume was 100 µL for the immature samples, versus 200 µL injected for the mature samples. There was sufficient radioactivity in the immature samples for detection of 100 µL, however, a larger volume of 200 µL was needed for the mature samples.

As previously provided, column “break-through” resulting in radioactivity eluting near the solvent front, or peak splitting of a compound, can occur when samples are injected with a high percentage of organic (50% acetonitrile) in a larger injection (200 µL). This is particularly true for a polar compound such as clopyralid.

Evidence of this effect was observed during analysis of the mature samples. On the same day that the mature acetonitrile/water extracts were analyzed and shown to contain 30-70% as “polar clopyralid”, additional analyses were made. These additional analyses combined day 0 acetonitrile/water extract with mature sugar beet shoot, or mature oilseed rape straw acetonitrile/water extracts, in each study. Keep in mind that the day 0 extracts contained 100% clopyralid, and that clopyralid is stable to hydrolysis and stable in crops for >13 months (EFSA 2018;16(7):5389). For sugar beet shoots, 700 µL of Day 0 extract (40,550 dpm/mL, 28,385 dpm, entirely clopyralid, 100 µL injection) plus 800 µL mature shoot extract (10,448 dpm/mL, 8358 dpm, 69.8% “polar clopyralid”, 200 µL injection) were combined (total 36,743

dpm). Based on these results, mathematically, the combined sample would contain ca 16% polar compounds, while at least 77% of the radioactivity should elute with clopyralid. The sample was combined less than 2 months after individually analyzing the day 0 extract.

Rather, upon injection of 200 µL of the combined sample, nearly 86% of the radioactivity eluted near the solvent front, with only 14% of the radioactivity eluting with clopyralid. Similar results were obtained for the mature oilseed rape straw acetonitrile/water extract combined with the Day 0 oilseed rape acetonitrile/water extract, where too much polar material eluted when 200 µL of the combined extract was analyzed. Since the day 0 fraction had recently demonstrated that the combined sample should contain a majority of clopyralid, and clopyralid is stable, yet the combined samples did not chromatograph as clopyralid, these data clearly demonstrate that injecting too much of a 50/50 acetonitrile/water extract onto the HPLC prompts early elution of clopyralid; clopyralid is not fully retained.

Furthermore, there was additional chromatography of the mature oilseed rape straw and mature sugar beet shoots acetonitrile/water extracts, in which the organic solvent was removed, and the samples made entirely aqueous (acetate buffer, pH 5.01), then the samples were heated to 37 °C for 18 hours. This heating cannot be considered significant, due to the low temperature. The slightly acidic pH (pH 5.01) is a very minor modification, and above the pKa of clopyralid (2.3), therefore, not protonating clopyralid. HPLC of the processed sample demonstrated less than 2% polar material in both samples. There were similarly low levels of polar material in sugar beet tubers made entirely aqueous and heated to 60 °C for 1 hour. These data demonstrate that the “polar clopyralid” is an artefact of the sample preparation/ chromatography.

To support this explanation of “polar clopyralid appearing near the solvent front, additional lab work was recently conducted at the Dow AgroSciences Indianapolis lab to confirm the previously reported results above, using non-radiolabeled clopyralid reference standard. Clopyralid was dissolved in 50/50 acetonitrile/water (v/v) at a concentration of ca 1 mg/mL. An HPLC system was set-up similar to the system used for the oilseed rape and sugar beet NORs above. The prepared standard was analyzed, in varying injection volumes of 10, 100, 200, and 250 µL. The clopyralid peak shape was sharp for the 10 µL injection, and gradually broadened, splitted, and included a more polar peak when at least 200 µL was injected. To confirm that the standard had not degraded, another 10 µL injection was made, resulting in sharp peak shape indicating no degradation. These results clearly indicate that although the majority of clopyralid is retained upon injection of larger volumes of acetonitrile/aqueous mixtures, there is peak broadening/splitting as well as an earlier eluting portion of clopyralid. These data clearly demonstrate that the “polar clopyralid” is an artefact of the sample preparation and/or chromatography.”

“metabolite B”

Metabolite B/ Unknown B” (also reported as “Unknown II”) in the sugar beet and oilseed rape metabolism studies was stated to be identical to ‘clopyralid conjugates’. Unknown B is a temperature-labile conjugate of clopyralid forming clopyralid at elevated temperatures (60 °C, 1 h). Unknown B was characterized as less polar than clopyralid. The exact identity of the conjugate was not clarified in the studies and can possibly include more than one compound.

The applicant gave following explanation:

“Unknown B was only observed in the neutral-organic extract (ACN:H₂O). The levels in the sugar beets were too low to warrant further identification. A subsample of oilseed rape ACN:H₂O (1:1 v/v) extract was reduced to dryness under a stream of nitrogen and reconstituted in water. An aliquot (1 mL) of the concentrated extract was combined with an equal volume of 6 M HCl. A separate aliquot of the concentrated extract was combined with an equal volume of water (control). Both were incubated in a shaking water bath at 60 °C for 1 hour, neutralized and analyzed by HPLC system 2. HPLC revealed no unknown B was present in either the acidified or the control (heated only) extract, therefore Unknown B is a temperature-labile conjugate of clopyralid. Unknown B remained following treatment with glucosidase, indicating it is unlikely to be a glucose conjugate of clopyralid, i.e., no evidence for glucose deconjugation.”

“Metabolite B is a mixture of conjugates of clopyralid that are less polar than clopyralid, that easily release clopyralid upon treatment with caustic solution. This has been proven in both the pasture and cabbage NORs (DAS study IDs GH-C 4289 and GH-C 1424), as well as radiovalidation of the caustic extraction that quantitatively extracts clopyralid, as reported in the 30-d CRC (DAS study ID 130733).

Therefore, the analytical methods (e.g., 120610) quantitatively measure clopyralid, free and conjugated, as clopyralid, thus allowing a worst-case residue level for consumer risk assessment purposes.”

RMS conclusions: RMS FI concluded, based on the further data provided by the applicant, that it has been sufficiently demonstrated that “polar clopyralid” / “metabolite A” is indeed an artefact of the sample preparation (content of organic solvent) / chromatography (injection volume), and cannot thus be considered to originate from plant metabolism. In case of “metabolite B”, clopyralid conjugates are a plausible explanation although their nature has not been confirmed. The caustic methanol extraction method used NOR studies and majority of MOR studies can be expected to sufficiently quantitate the conjugates in plant material.

ER, Art.6 MRL application on the setting of MRL(s) in various commodities; 31 May 2018, updated 14 November 2018, 25 January 2019, 3 May 2019, 14 November 2019 and updated for wheat NOR & MOR 17 August 2020

EFSA:

Clarification on the nature of the ‘unknown metabolite B’ in the plant metabolism studies and whether it is identical with the ‘polar form of clopyralid’ and ‘clopyralid conjugates’ should be provided. Furthermore, an explanation should be provided why in mature samples of sugar beet and oilseed rape ‘polar clopyralid’ and clopyralid were observed, while ‘polar clopyralid’ was not observed in immature samples, as the analytical procedure used was always the same within one study.

Reply from EFSA (26.11.2018):

Clarification on the nature of the ‘unknown metabolite B’ in the plant metabolism studies was provided by the applicant as follows: „The author’s OSR NOR conclusions were that “it is more likely that the acidic conditions of the sample optimise retention of clopyralid on the chromatography column...and that this component represents free clopyralid.” Similarly, the sugar beet NOR concluded that “Unknown A represents unchanged clopyralid which is in some way excluded from association with the C18 solid phase. There was no evidence for any deconjugation.

It is understood that the argumentation is meant to clarify the nature of ‘unknown A’¹. Although it sounds logic, for transparency reasons the details of the sample preparation and chromatographic conditions for the NOR studies with OSR and sugar beet should be listed, compared and analysed systematically and to substantiate this argument. It should be also confirmed that the term ‘polar clopyralid’ is referring always to a single structure (and not different polar compounds) and that these structure of ‘polar clopyralid’ is identical across all metabolism studies where identified.

In the same way facts and arguments should be systematically listed and analysed to substantiate the claim that “unknown B” is a clopyralide conjugate.

The argumentation / explanation provided by the applicant why in mature samples of sugar beet and oilseed rape ‘polar clopyralid’ and clopyralid were observed while ‘polar clopyralid’ was not observed in immature samples seems not to be coherent with the observation/interpretation of the RMS. Please clarify.

Reply from EFSA (18.3.2019):

The applicant provided in a tabulated format a summary of the extraction and analysis conditions of the samples of the OSR and sugar beet metabolism studies (Table S1).

¹ It should be noted that the “unknown A” refers to a polar form, whereas “unknown B” is referred to as clopyralide conjugate in the NOR with sugar beet. In the OSR NOR study the “unknown B” is referred to as clopyralide conjugate.

From this summary it is understood, that the same extraction (ACN:H₂O, 1:1) and analysis conditions (HPLC system 1 with 2% acetic acid) were applied to immature and mature OSR and sugar beet plant parts resulting in the occurrence of the “unknown metabolite A” in mature sample extracts only whereas clopyralid was detected in both mature and immature plant material.

The argumentation that “unknown metabolite A” represents a protonated form of clopyralid seems not logic as it was only demonstrated with the mature sample material that a shift in pH resulted in disappearing of the unknown metabolite A. If a protonation of clopyralid occurred during analysis, the “unknown metabolite A” should have also been observed under the same analytical conditions (ACN:H₂O, 1:1) in the chromatograms of the immature plant extracts which also contained clopyralid.

The change in the sample workup and analytical conditions for mature sample parts resulted in the disappearance of the “unknown metabolite A”. The conclusion that it was converted into clopyralid can be challenged without further identification, e.g. by MS. Another explanation could also be the co-elution of any compound with clopyralid due to the pH change.

The applicant is invited to provide further clarification on the nature of “unknown metabolite A”.

RMS:

RMS considered Applicants' responses.

RMS concluded that the ‘polar form of clopyralid’ does seem to easily convert to protonated free clopyralid. Thus, it is probable, that the “polar form” represents either the non-protonated form or some other matrix effect-derived easily transformed form (such as chelates). In such case the form would not originate from plant metabolism but would indeed be an artefact of sample preparation protocol. The current analytical methods as well as those used in the magnitude of residue trials can be considered to effectively analyze also the “polar form” provided that they sufficiently take into account possible matrix effects and the low pK_a of clopyralid. RMS does not consider that the identity of the “polar form” needs to be studied any further, nor is there need to change the definition of residue suggested in the active substance review.

The applicant DAS stated that the metabolite ‘unknown B’ is a temperature-labile conjugate of clopyralid forming clopyralid at elevated temperatures. Unknown B was characterized as less polar than clopyralid. The exact identity of the conjugate was not clarified in the studies and can possibly include more than one compound. As all conjugates are included in the residue definition for monitoring and risk assessment this explanation is considered sufficient, provided that the analytical method for monitoring includes caustic methanol or other equivalent hydrolyzing extraction step capable of releasing clopyralid.

Based on the further clarification (9.1.2019), the identity of Unknown B still cannot be concluded. The plausible conjugate compound/compounds is/are fully degraded to clopyralid with either heat treatment or acid treatment but not with glucosidase enzyme treatment. Based on the data, only clopyralid-glucose conjugates are excluded as possible compounds.

After further explanations by the applicant on **09.01.2019**, EFSA commented on the metabolite issue in the following manner:

The applicant provided in a tabulated format a summary of the extraction and analysis conditions of the samples of the OSR and sugar beet metabolism studies (Table S1). From this summary it is understood, that the same extraction (ACN:H₂O, 1:1) and analysis conditions (HPLC system 1 with 2% acetic acid) were applied to immature and mature OSR and sugar beet plant parts resulting in the occurrence of the “unknown metabolite A” in mature sample extracts only whereas clopyralid was detected in both mature and immature plant material.

The argumentation that “unknown metabolite A” represents a protonated form of clopyralid seems not logic as it was only demonstrated with the mature sample material that a shift in pH resulted in disappearing of the unknown metabolite A. If a protonation of clopyralid occurred during analysis, the “unknown metabolite A” should have also been observed under the same analytical conditions (ACN:H₂O, 1:1) in the chromatograms of the immature plant extracts which also contained clopyralid.

The change in the sample workup and analytical conditions for mature sample parts resulted in the disappearance of the “unknown metabolite A”. The conclusion that it was converted into clopyralid can be challenged without further identification, e.g. by MS. Another explanation could also be the co-elution of any compound with clopyralid due to the pH change. The applicant is invited to provide further clarification on the nature of “unknown metabolite A”.

The applicant responded on **30.4.2019** after going through the material of OSR and beet studies.

“Although the extraction analytical procedures were identical within a study, and similar between studies (DAS study IDs GHE-P 9938 and GHE-P-9939), the chromatography was different between immature and mature samples. The samples analyzed by HPLC were of the same general composition, nominally 50/50, ACN/H₂O (v/v). However, after carefully reviewing the raw data of these studies (finalized in 2002), it was determined that a major, and very important difference, was that the HPLC injection volume was 100 µL for the immature samples, versus 200 µL injected for the mature samples. There was sufficient radioactivity in the immature samples for detection of 100 µL, however, a larger volume of 200 µL was needed for the mature samples.

As previously provided, column “break-through” resulting in radioactivity eluting near the solvent front, or peak splitting of a compound, can occur when samples are injected with a high percentage of organic (50% acetonitrile) in a larger injection (200 µL). This is particularly true for a polar compound such as clopyralid.

Evidence of this effect was observed during analysis of the mature samples. On the same day that the mature acetonitrile/water extracts were analyzed and shown to contain 30-70% as “polar clopyralid”, additional analyses were made. These additional analyses combined day 0 acetonitrile/water extract with mature sugar beet shoot, or mature oilseed rape straw acetonitrile/water extracts, in each study. Keep in mind that the day 0 extracts contained 100% clopyralid, and that clopyralid is stable to hydrolysis and stable in crops for >13 months (EFSA 2018;16(7):5389). For sugar beet shoots, 700 µL of Day 0 extract (40,550 dpm/mL, 28,385 dpm, entirely clopyralid, 100 µL injection) plus 800 µL mature shoot extract (10,448 dpm/mL, 8358 dpm, 69.8% “polar clopyralid”, 200 µL injection) were combined (total 36,743 dpm). Based on these results, mathematically, the combined sample would contain ca 16% polar compounds, while at least 77% of the radioactivity should elute with clopyralid. The sample was combined less than 2 months after individually analyzing the day 0 extract.

Rather, upon injection of 200 µL of the combined sample, nearly 86% of the radioactivity eluted near the solvent front, with only 14% of the radioactivity eluting with clopyralid. Similar results were obtained for the mature oilseed rape straw acetonitrile/water extract combined with the Day 0 oilseed rape acetonitrile/water extract, where too much polar material eluted when 200 µL of the combined extract was analyzed. Since the day 0 fraction had recently demonstrated that the combined sample should contain a majority of clopyralid, and clopyralid is stable, yet the combined samples did not chromatograph as clopyralid, these data clearly demonstrate that injecting too much of a 50/50 acetonitrile/water extract onto the HPLC prompts early elution of clopyralid; clopyralid is not fully retained.

Furthermore, there was additional chromatography of the mature oilseed rape straw and mature sugar beet shoots acetonitrile/water extracts, in which the organic solvent was removed, and the samples made entirely aqueous (acetate buffer, pH 5.01), then the samples were heated to 37 °C for 18 hours. This heating cannot be considered significant, due to the low temperature. The slightly acidic pH (pH 5.01)

is a very minor modification, and above the pKa of clopyralid (2.3), therefore, not protonating clopyralid. HPLC of the processed sample demonstrated less than 2% polar material in both samples. There were similarly low levels of polar material in sugar beet tubers made entirely aqueous and heated to 60 °C for 1 hour. These data demonstrate that the “polar clopyralid” is an artefact of the sample preparation/chromatography.

The majority of data cited above were not included in the final report, however were found upon thorough review of the raw data. These data support the conclusions of the authors: that Unknown A is an artefact and is really unchanged clopyralid.

To support this explanation of “polar clopyralid appearing near the solvent front, additional lab work was recently conducted at the Dow AgroSciences Indianapolis lab to confirm the previously reported results above, using non-radiolabeled clopyralid reference standard. Clopyralid was dissolved in 50/50 acetonitrile/water (v/v) at a concentration of ca 1 mg/mL. An HPLC system was set-up similar to the system used for the oilseed rape and sugar beet NORs above. The prepared standard was analyzed, in varying injection volumes of 10, 100, 200, and 250 µL. The clopyralid peak shape was sharp for the 10 µL injection, and gradually broadened, splitted, and included a more polar peak (5-7% of the Region of Interest eluting ca 4.7 minutes) when at least 200 µL was injected. To confirm that the standard had not degraded, another 10 µL injection was made, resulting in sharp peak shape indicating no degradation. These results clearly indicate that although the majority of clopyralid is retained upon injection of larger volumes of acetonitrile/aqueous mixtures, there is peak broadening/splitting as well as an earlier eluting portion of clopyralid. These data clearly demonstrate that the “polar clopyralid” is an artefact of the sample preparation and/or chromatography.”

The applicant gave further explanation also on the “metabolite B”:

“Metabolite B is a mixture of conjugates of clopyralid that are less polar than clopyralid, that easily release clopyralid upon treatment with caustic solution. This has been proven in both the pasture and cabbage NORs (DAS study IDs GH-C 4289 and GH-C 1424), as well as radiovalidation of the caustic extraction that quantitatively extracts clopyralid, as reported in the 30-d CRC (DAS study ID 130733). Therefore, the analytical methods (e.g., 120610) quantitatively measure clopyralid, free and conjugated, as clopyralid, thus allowing a worst-case residue level for consumer risk assessment purposes.”

RMS FI concluded, based on the further data provided by the applicant, that it has been sufficiently demonstrated that polar clopyralid / “metabolite A” is indeed an artefact of the sample preparation (content of organic solvent) / chromatography (injection volume), and cannot thus be considered to originate from plant metabolism. In case of “metabolite B”, clopyralid conjugates are a plausible explanation although their nature has not been confirmed. The caustic methanol extraction method used in NOR studies and majority of MOR studies can be expected to sufficiently quantitate the conjugates in plant material.

In response to EFSA’s comments on **June 3rd 2019**, the applicant submitted amended study reports of the sugar beet and oilseed rape metabolism studies (study reports 397619 and 397624), and a non-GLP study on behavior of clopyralid chemical standard in the analytical HPLC conditions described in these NoR studies. Detailed evaluation of the submitted study reports were performed and can be found in this report (page 115). Based on the studies, EMS concluded following:

The “polar clopyralid”, also termed as Unknown A, was observed in the new study performed using analytical clopyralid standard. As the standard compound was shown to be stable, the appearance of the compound seems to be an artefact of the HPLC system. Unknown A appeared when high injection volume and high proportion of organic solvent was used.

Compared to the injection volumes, solvents and HPLC systems used in the sugar beet and oilseed rape metabolism studies, the above findings can be mostly confirmed with few exceptions. These exceptions could be explained by other matrix effects in the extract samples. It is possible, that Unknown A is a form of clopyralid that is sensitive to pH changes, especially in complex extracts. The increase of its relative amount in mixing two ACN:H₂O extracts (described in the sugar beet study) also indicates that it can form in situ in extracts, and thus would not be a plant metabolite.

Unknown B was hypothesised to be either heat-labile conjugate(s) or an artefact of the HPLC system. The component was found in the ACN:H₂O extracts of oilseed rape 28 DAT and mature straw and seed samples and at very low level in mature sugar beet shoots. The appearance of the component was not dependent on the HPLC system used or solvent change, while it seemed to be easily converted to clopyralid by heat treatment or long storage. It is thus not clearly resolved if the component is a labile clopyralid conjugate or similar component, or an artefact of the analysis system.

Unknown A and B were only detected in the neutral ACN:H₂O extracts while they were not detected in the caustic methanol (NaOH:MeOH) extracts. Thus, the compounds are degraded/transferred to clopyralid by caustic methanol extraction and can be reliably analysed as clopyralid.

The applicant stated that “the revised oilseed rape and sugar beet NORs, plus the HPLC analyses report, indicate that in all samples, Unknowns A and B were free or conjugated clopyralid, not a new/unidentified metabolite. The elution time may vary due to injection of too much organic and/or matrix effects and/or HPLC conditions (e.g., pH), but the results consistently indicate the entirety of the radioactive residue in immature and mature crop fractions is directly related to clopyralid.”

Further information was provided in **June 2020** by the applicant:

Furthermore, we have completed a **new wheat Nature of Residue study** (*Reference: The Metabolism of [¹⁴C]-Clopyralid in Wheat, 19 June 2020. GLP, unpublished. Dow AgroSciences Study: Study No: 191200*).

This Wheat Nature of Residue (NoR) study has been conducted after applying clopyralid at 125 g/ha (BBCH 39) and confirms the pathway observed in other crops, supporting the residue definition in all crops, especially cereals and pasture grass due to similarity with wheat forage.

No “polar clopyralid” was observed, because proper sample preparation was performed including limiting the amount of organic solvent injected onto the HPLC.

The study confirms the previous NORs in that “free” clopyralid is only detected in the neutral extracts, along with a broad peak similar to the previously reported clopyralid conjugates (“unknown B”). The radioactive residues are completely extracted (same general procedures as used in the original NORs), primarily with neutral followed by mild base; the mild base extracts contain only clopyralid. When the neutral extract containing the clopyralid conjugates is exposed to a mild base (5% ammonium hydroxide) for less than 1 hour, the residue becomes completely clopyralid. Formation of “conjugates” was replicated using clopyralid standard spiked onto control wheat grain – the “conjugates” were observed before treating with caustic, and disappeared after mild, quick caustic treatment. Since true conjugates cannot form so quickly with a frozen, milled tissue, it has been concluded that the “conjugates” are actually weak chromatographic interactions between the clopyralid and the matrix, that are easily interrupted with mild caustic. Any sort of light manipulation of the sample (SPE, changing the pH, etc.) removed the “unknown B” region. Therefore, this region can be identified as base labile-clopyralid. In total, 92.6-97.2% of the residue (normalized) was identified as clopyralid.

In conclusion, the additional evidence from existing plant metabolism studies including a new wheat plant metabolism study confirm that polar clopyralid (also called unknown A and unknown 1) is an artefact of the sample preparation (content of organic solvent)/ chromatography (injection volume) and is clopyralid. Unknown B is mild caustic-labile conjugates of clopyralid. The majority (>90%) of the

residue can be considered to be clopyralid, which is fully quantitated using the existing field trial analytical method. The EFSA proposed plant residue definition for risk assessment and monitoring (clopyralid common moiety as sum of clopyralid, its salts and conjugates expressed as clopyralid) is justified and can be adopted.

RMS comments:

The results of the new wheat metabolism study indicate that the detected wide, less polar region eluting after the main clopyralid peak is composed of clopyralid that is loosely bound to or interacting with coextracted plant material where it is easily released from. The ease of release of this “base-labile” clopyralid does not support the presence of covalently bound clopyralid conjugates or other similar compound that could be regarded as plant metabolite.

EMS considers the new wheat metabolism study as acceptable and it can be used to confirm the previously suggested residue definitions.

The new study does not answer to the questions raised on metabolite A detected in the sugar beet and OSR studies as no such polar clopyralid was detected in the wheat metabolism study. The possible reasons were the use of optimized analytical methods and lower solvent use at injection, assuming that the more polar region is an artefact of the analytical method.

Note that “polar clopyralid” was not observed in the current oilseed rape metabolism study (2021) nor the on-going sugar beet metabolism study. The Sponsor still considers the more polar regions to be an artefact of the analytical procedures.