

FINAL REGISTRATION REPORT

Part B

Section 7

Metabolism and Residues

Detailed summary of the risk assessment

Product code: K-300SL-RR

Product name(s): Faworyt 300 SL

Chemical active substance:

Clopyralid, 300 g/L

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT – Art 43

(Renewal of authorisation)

Applicant: CIECH Sarzyna S.A.

Submission date: 12/2021

Correction on: 05/2022

MS Finalisation date: 07/2022; 10/2022; 10/2023

Version history

When	What
December 2021	dRR version 1 submitted by applicant
May 2022	First correction for product authorization
July 2022	zRMS first assessment
October 2022	Final Registration Report
October 2023	Verification of the Report in accordance with the Authority's arrangements regarding the assessment of plant protection products containing the active substance clopyralid.

Table of Contents

7.1	Summary and zRMS Conclusion.....	6
7.1.1	Critical GAP(s) and overall conclusion	8
7.1.2	Summary of the evaluation	11
7.1.2.1	Summary for clopyralid	11
7.1.2.2	Summary for K-300SL-RR.....	12
7.2	Clopyralid	13
7.2.1	Stability of Residues (KCA 6.1)	14
7.2.1.1	Stability of residues during storage of samples	14
7.2.1.2	Stability of residues in sample extracts (KCA 6.1).....	14
7.2.2	Nature of residues in plants, livestock and processed commodities.....	15
7.2.2.1	Nature of residue in primary crops (KCA 6.2.1)	15
7.2.2.2	Nature of residue in rotational crops (KCA 6.6.1).....	16
7.2.2.3	Nature of residues in processed commodities (KCA 6.5.1).....	17
7.2.2.4	Conclusion on the nature of residues in commodities of plant origin (KCA 6.7.1)	18
7.2.2.5	Nature of residues in livestock (KCA 6.2.2-6.2.5)	19
7.2.2.6	Conclusion on the nature of residues in commodities of animal origin (KCA 6.7.1)	20
7.2.3	Magnitude of residues in plants (KCA 6.3).....	21
7.2.3.1	Summary of European data and new data supporting the intended uses	21
7.2.3.2	Conclusion on the magnitude of residues in plants	23
7.2.4	Magnitude of residues in livestock	23
7.2.4.1	Dietary burden calculation	23
7.2.4.2	Livestock feeding studies (KCA 6.4.1-6.4.3)	24
7.2.5	Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation) (KCA 6.5.2-6.5.3).....	28
7.2.5.1	Available data for all crops under consideration	28
7.2.5.2	Conclusion on processing studies	29
7.2.6	Magnitude of residues in representative succeeding crops.....	29
7.2.7	Other / special studies (KCA6.10, 6.10.1)	30
7.2.8	Estimation of exposure through diet and other means (KCA 6.9).....	30
7.2.8.1	Input values for the consumer risk assessment	30
7.2.8.2	Conclusion on consumer risk assessment	30
7.3	Combined exposure and risk assessment	31
7.4	References	32
Appendix 1	Lists of data considered in support of the evaluation.....	33
Appendix 2	Detailed evaluation of the additional studies relied upon	41
A 2.1	Clopyralid	41
A 2.1.1	Stability of residues.....	41
A 2.1.2	Nature of residues in plants, livestock and processed commodities	41
A 2.1.3	Magnitude of residues in plants	48
A 2.1.4	Magnitude of residues in livestock	48
A 2.1.5	Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)	48
A 2.1.6	Magnitude of residues in representative succeeding crops.....	62
A 2.1.7	Other/Special Studies.....	62

Appendix 3	Pesticide Residue Intake Model (PRIMo).....	63
A 3.1	TMDI calculations	63
A 3.2	IEDI calculations	64
A 3.3	IESTI calculations - Raw commodities	64
A 3.4	IESTI calculations - Processed commodities.....	65
Appendix 4	Additional information provided by the applicant	66

CIECH Sarzyna S.A. possess Letter of Access from the Task Force Clopyralid to alternative data package for active substance Clopyralid. Task Force Clopyralid has submitted its Data Matching List to zRMS, Finland. This Data Matching List covers all the protected studies from the main notifier. The Data Matching List was accepted by zRMS.

Metabolism and residue data (KCA section 6)

7.1 Summary and zRMS Conclusion

October 2023 Verification of the Report in accordance with the Authority's arrangements, from the meeting of July 28, 2023, regarding the assessment of plant protection products containing the active substance clopyralid.

Stability of residues during storage of samples

Stability of residues during storage of samples was provided during the EU review of clopyralid.

Residues of clopyralid were found to be stable at $\leq -18^{\circ}\text{C}$ for up to:

13 months in maize fodder and forage (high water content matrix)

13 months in maize grain (high starch content matrix)

17 months in pasture grass (high water content matrix)

24 months in rape seed (high oil content matrix)

Metabolism in plants and animals

Residue definition for monitoring (Commission Regulation (EU) 2021/1807 of 13 October 2021): clopyralid (plants and animals)

Residue definition for risk assessment:

Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of “polar clopyralid” (EFSA Journal 2018;16(7):5389)

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.

EFSA Journal 2021;19(1):6389:

Based on the metabolic pattern identified in metabolism studies with cereals, rotational crops and the results of hydrolysis studies, the residue definitions were proposed as clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) both, for enforcement and risk assessment. These residue definitions are applicable to cereals/grass crop group, rotational crops and processed products.

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.

Authority's arrangements:

- in the case of clopyralid, assessment of residue data for the uses proposed by the Applicants, including, among others, on oilseeds, roots or tubers (crops other than representative crops assessed in RAR (2019) for the substance clopyralid) should be carried out in accordance with the general residue definition for clopyralid proposed by EFSA in the document EFSA Journal 2018;16(8):5389 - applies all administrative proceedings conducted by the Ministry of Agriculture and Rural Development (Article 33, Article 43, Article 40, Article 45, Article 51).

Plant residue definition for monitoring: Clopyralid (Reg. (EU) 2021/1807)

Plant residue definition for risk assessment: clopyralid common moiety (sum of clopyralid, its salts and

conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of “polar clopyralid” (EFSA Journal 2018;16(7):5389).

~~According to EFSA, the residue definition should be limited to cereals/grass only. Taking this into account, application on winter rape and sugar beet are not acceptable until the data gap is filled.~~

The intended uses **on cereals** are supported by the evaluated plant metabolism studies.

One new hydrolysis study has been submitted by the applicant in the framework of this application. The study was submitted as equivalent to protected hydrolysis study and was accepted in data matching (Finland 2022). Study is acceptable. The test compound clopyralid was stable under all conditions of high temperature hydrolysis for simulation of food processing. Equivalent endpoint have been received.

New metabolism studies in rotational crops have been submitted by the applicant in the framework of this application. The study was submitted as equivalent to protected study and was accepted in data matching (Finland 2022). The requirement for alternative tests has been met. This study should be evaluated at EU level.

Magnitude of residues in plants

Winter wheat

Proposed uses: 1 application, BBCH 21-29 (Spring), 90 – 120 g as/ha, PHI: not required

Applicant refers to the unprotected EU data. GAP on which EU a.s. assessment is based: 1 x 0.150 kg as/ha, BBCH 39

Additionally new data are submitted in the framework of this application.

Study S20-04397-01 (1 x 0.153,9 kg as/ha, BBCH 39):

Residues: 0.79 mg/kg

Study S19-01810-01 (1 x 0.159 kg as/ha, BBCH 39)

Residues: 0.76 mg/kg

Sufficient data are available to support the proposed use. The residues arising from the proposed uses will not exceed the MRLs established for wheat (Reg. (EU) 2021/1807)

Winter rape, Sugar beet

~~According to EFSA, the residue definition should be limited to cereals/grass only. Taking this into account, application on winter rape and sugar beet are not acceptable (until the data gap is filled).~~

Winter rape

Proposed use: 1 application, BBCH 10-50 (Spring), 90 – 120 g as/ha, PHI: not required

Applicant refers to unprotected EU data:

Trials GAP: 1 x 0.1 kg as/ha + 1 x 0.2 kg as/ha, outdoor

Residues: <0.01, 0.01, 2 x 0.02, 0.03, 0.04, 0.05, 0.1 mg/kg

Sufficient data are available to support the proposed use. The residues arising from the proposed uses will not exceed the MRLs established for oilseed rape (Reg. (EU) 2021/1807).

Sugar beet

Proposed use: 1 application, BBCH 12-14 (Spring), 90 g as/ha, PHI: not required

Applicant refers to unprotected EU data:

Trials GAP: 1 x 0.1 kg as/ha + 1x 0.2 kg/ha latest timing of BBCH 39

Residues: 0.12, 0.17, 0.21, 0.29, 0.34, 0.35, 0.36, 0.41, 0.56, 0.80

Sufficient data are available to support the proposed use. The residues arising from the proposed uses will not exceed the MRLs established for sugar beet roots (Reg. (EU) 2021/1807).

Livestock 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.

Magnitude of residues in processed commodities

New acceptable, alternative to the protected studies were provided by the applicant (White T., 2021, S19-01810; White T., 2021, S20-04397). No further data is required.

Rotational study

According to the available data following label restriction is proposed: not to use clopyralid on the same field for 125 days after the initial application regardless of the crop grown (see EFSA Journal 2021;19(1):6389).

Other / special studies

Clopyralid is systemic compound and potential residues in honey might occur in honey even from applications before flowering. Therefore, information about residue level in pollen and bee products should be provided by the applicant (post registration requirement).

Estimation of exposure through diet and other means

The accepted uses of clopyralid in the formulation Faworyt 300 SL do not represent unacceptable acute and chronic risks for the consumer. Applicant's calculations are accepted (EFSA PRIMo rev 3.1)

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 K-300SL-RR are presented in Table 7.1-1. They have been selected from the individual GAPs in the zone/EU for winter wheat, winter rape, sugar beet. A list of all intended uses within the zone/EU is given in Part B, Section 0.

Overall conclusion

The data available are considered sufficient for risk assessment. An exceedance of the current MRL of 3 mg/kg in wheat, 0.5 mg/kg in oilseed rape and 1 mg/kg in sugar beet for clopyralid as laid down in Reg. (EU) 396/2005 is not expected.

The chronic and the short-term intakes of clopyralid residues are unlikely to present a public health concern.

EFSA (2021) concluded that the residue definitions only apply for the crop groups cereals and grass.

As far as consumer health protection is concerned, authority, zRMS agrees with the authorization of the use on winter wheat, winter rape and sugar beet.

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

According to the available data following label restriction is proposed: not to use clopyralid on the same field for 125 days after the application regardless of the crop grown.

Data gaps

Data gaps should be listed in the summary to give an overview (especially for cMS).

Noticed data gaps are:

- Information about residue level in pollen and bee products should be provided by the applicant (post registration requirement – within 2 years after authorisation)
- Data gap on residue definitions should be filled at EU level.

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		
						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)	g as/hL min max	water L/ha min max	g as/ha min max				
1	Winter wheat	N-EU	K-300SL-RR	F	Dicotyledonous weeds (from cotyledon stage to the rosette stage)	SL	300 g/L	Broadcast - foliar	BBCH 21-29 (Spring)	1	n.a.	30 - 60	200-300	90 - 120	PHI is covered by the time remaining between application and harvest	A		
2	Winter rape	N-EU	K-300SL-RR	F	Dicotyledonous weeds (from cotyledon stage to the rosette stage)	SL	300 g/L	Broadcast - foliar	BBCH 10-50 (Spring)	1	n.a.	30 - 60	200-300	90 - 120	PHI is covered by the time remaining between application and harvest	A		
3	Sugar beet	N-EU	K-300SL-RR	F	Dicotyledonous weeds (from cotyledon stage to the rosette stage)	SL	300 g/L	Broadcast - foliar	BBCH 12-14 (Spring)	1	n.a.	30 - 45	200-300	90	PHI is covered by the time remaining between application and harvest	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

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 K-300SL-RR is composed of clopyralid.

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	Peer review of the pesticide risk assessment of the active substance clopyralid	2018	0.15	rat, 2-year chronic toxicity and oncogenicity study	100
ARfD	Peer review of the pesticide risk assessment of the active substance clopyralid	2018	0.17	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	Winter wheat	Yes	Yes (17)	NR PHI covered by the time between the last application and harvest	Yes	Yes	No	No
2	Winter oilseed rape	Yes	Yes (8)	NR PHI covered by the time between the last application and harvest	Yes	Yes		
3	Sugar beet	Yes	Yes (10)	NR PHI covered by the time between the last application and harvest	Yes	Yes		

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

The data available are considered sufficient for risk assessment. An exceedance of the current MRL of 3 mg/kg in wheat, 0.5 mg/kg in oilseed rape and 1 mg/kg in sugar beet for clopyralid as laid down in Reg. (EU) 396/2005 is not expected.

The chronic and the short-term intakes of clopyralid residues are unlikely to present a public health concern.

Winter wheat

Sufficient number of trials (minimum 8) are presented for **all crops** and fulfills requirements for Northern Zone. Presented studies represent worst case and since the objective is not new MRL setting but only MRL compliance, it is considered that all the trials are relevant to support registration of Faworyt 300 SL.

7.1.2.2 Summary for K-300SL-RR

Table 7.1-4: Information on K-300SL-RR (KCA 6.8)

Crop	PHI for K-300SL-RR proposed by applicant	PHI/ Withholding period* sufficiently supported for	PHI for K-300SL-RR proposed by zRMS	zRMS Comments (if different PHI proposed)
		Clopyralid		
Winter wheat	F	NR	!	
Winter oilseed rape	F	NR	!	
Sugar beet	F	NR	!	

NR: not relevant

* Purpose of withholding period to be specified

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

Table 7.1-5: Waiting periods before planting succeeding crops

Waiting period before planting succeeding crops		Overall waiting period proposed by zRMS for K-300SL-RR
Crop group	Led by clopyralid	
All crops	NR	125 days after the initial application

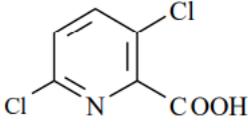
NR: not relevant

Assessment

7.2 Clopyralid

General data on Clopyralid are summarized in the table below

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	Pyridine compound
Mode of action (if available)	Selective, systemic, absorbed through leaves and roots. Synthetic auxin.
Systemic	Yes
Company (ies)	Dow AgroSciences S.A.S.*
Rapporteur Member State (RMS)	Finland co-RMS: Poland
Approval status	Approved Date of (01/10/2021) and reference to decision (COMMISSION IMPLEMENTING REGULATION (EU) 2021/1191 of 19 July 2021)
Restriction	-
Review Report	SANTE/10206/2021 Rev 1 20 May 2021
Current MRL regulation	COMMISSION REGULATION (EU) 2021/1807 of 13 October 2021
Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed	Pending
EFSA Journal : Conclusion on the peer review	Yes**
EFSA Journal: conclusion on article 12	No
Current MRL applications on intended uses	EFSA Q 2018 00576 oat grain, wheat grain and in fat, liver and kidney of bovine, sheep and goat and in swine kidney Status: Evaluation ongoing

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

** EFSA, 2018 - see list of references

7.2.1 Stability of Residues (KCA 6.1)

7.2.1.1 Stability of residues during storage of samples

Available data

No new data submitted in the framework of this application.

Table 7.2-2: Summary of stability data achieved at $\leq -18^{\circ}\text{C}$ (unless stated otherwise)

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference
Data relied on in EU			
Plant products			
Maize	High starch content, dry commodities	13 months	RMS, 2018 Foster, D.R., Blakeslee, B.A., Rutherford, B.S., 1996, Study No. RES93050.01
Maize fodder/forage	High water content	13 months	Clements, B, Bolton, A, 1996, Study No. GHE-P-5350
Pasture grass		17 months	
Oilseed rape	High oil content	24 months	RMS, 2018 Dial, E., Lindsay, D, 2006, Study No. 020122.02
Animal Products			
Ruminant	Liver, kidney, muscle, milk	19 months	DAR, 2004; Study No. 020120.01
Ruminant	Fat	24 months	
Poultry	Egg	19 months	RAR, 2018 Lab Study No. 69209; Study No. 120602

Conclusion on stability of residues during storage

The storage stability of clopyralid in plants stored under frozen conditions was investigated during Annex I inclusion. It was demonstrated that clopyralid was stable for at least 24 months when stored at -18°C in commodities with high oil content. In pasture stability was demonstrated up to 17 months, in maize grain and forage/fodder up to 13 months when stored at -20°C . stability of conjugates has not been tested, though clopyralid conjugates are major metabolites comprising up to 50 % of TRR depending on crop studied. It is assumed that conjugated clopyralid will be also stable

These data are sufficient to demonstrate the stability of clopyralid residues in high protein/starch and high water content commodities.

7.2.1.2 Stability of residues in sample extracts (KCA 6.1)

All extract samples were analyzed directly after extraction and therefore the stability of residues in sample extracts is not necessary.

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

No new data submitted in the framework of this application.

Table 7.2-3: Summary of plant metabolism studies

Crop Group	Crop	Label position	Application and sampling details					Reference
			Method, F or G (a)	Rate (kg a.s./ha)	No	Sampling (DAT)	Remarks	
EU data								
Leafy vegetables	Cabbage	[14C]-Clopyralid	foliar treatment, F	420 g as/ha	1	0, 5 and 38d	-	RMS, 2018 Guo, C., 1996, Study No. RES95095
Root and tuber vegetables	Sugar beet	[14C]-Clopyralid	foliar treatment, F	300 g as/ha	1	0, 28 and 105 d (maturity)	-	RMS, 2018 Chapleo, S. ; Caley, C. Y., 2002, Study No. GHE-P-9939
Pulses and oilseeds	Oilseed rape	[14C]-Clopyralid	foliar treatment, F	300 g as/ha	1	0, 28 and 77 d (maturity)	-	RMS, 2018 Chapleo, S., Caley, C. Y., White, D. E., 2002, Study No. GHE-P 9938

Summary of plant metabolism studies reported in the EU

Plant metabolism was studied by applying clopyralid radio-labelled in two positions as a foliar spray to sugar beet, oilseed rape and cabbage at the intended application rate. The submitted study in pasture was not done in compliance with GLP and thus regarded by RMS as additional information only. At maturity most of the radioactivity was taken up into the plants, the major radioactive compound was unchanged clopyralid, the anionic form (salt) and conjugated forms of clopyralid. Conjugated clopyralid was present at low levels in beet shoots (ca 1% TRR), but at levels 18-30% TRR in oilseed rape matrices. Together all clopyralid fractions accounted for 89 – 97 % of TRR. No other significant metabolites were detected. In sugar beets clopyralid (including salts and conjugates) accounted for 0.36 and 0.38 mg/kg in beets and shoots, respectively. In oilseed rape, clopyralid fractions accounted for 0.71 mg/kg in straw and 0.06 mg/kg in seeds. In cabbage plants unchanged clopyralid was found to be the major component of the residue, accounting for 0.32 mg/kg in cabbage heads and 1.2 mg/kg in wrapper leaves. It was stated that the presence of residues in the cabbage hearts indicates translocation from the immature leaves with the residue level being diluted by growth. Based on the supportive study on pasture, the metabolism of clopyralid in grass is also very limited and the reduction of residue levels (from 13 mg/kg to 0.16 mg/kg) is due to the growth dilution. No extensive metabolism occurred in the crops studied and clopyralid (including anionic form) was found to be the major component of the residue. However, depending on the crop

clopyralid conjugates seem to build a major part of the residue, and furthermore, the analytical methods employed in recent supervised residue trials (Hastings, 2002) include a hydrolysis step covering potentially present conjugated forms of clopyralid as well. The method by Hastings is also the proposed enforcement method for food of plant origin. Therefore, the residue definition in plants should be clopyralid including its salt and conjugates, expressed as clopyralid for risk assessment and monitoring purposes. It is noted that the proposal for a plant residue definition agreed in the experts' meeting for residues was limited to clopyralid only, based on previous RMS information that no hydrolysis step was included in the relevant methods of analysis and on the view that, with the exception of rapeseed, the level of conjugates was negligible in the edible part of the crops studied. There is also indication from supervised residue trials (see below), that clopyralid (including salts) might be a valid alternative to define the residue in plants for monitoring purposes, provided that a validated enforcement method was available. The metabolism of clopyralid was similar in all studied crop groups, thus the metabolic behaviour of clopyralid in plants can be regarded sufficiently studied. The proposed residue definition might apply for plants in general.

Conclusion on metabolism in primary crops

The metabolism of clopyralid was similar in all studied crop groups, thus the metabolic behaviour of clopyralid in plants can be regarded sufficiently studied. The proposed residue definition might apply for plants in general. Although 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)'.

7.2.2.2 Nature of residue in rotational crops (KCA 6.6.1)

Available data

New metabolism studies have been submitted by the applicant in the framework of this application. The study was submitted as equivalent to protected study and was accepted in data matching. CIECH Sarzyna S.A. possess Letter of Access from the Task Force Clopyralid to alternative data package for active substance Clopyralid. Equivalent nature of residue in rotational crop study was included in Data Matching List. This Data Matching List covers all the protected studies from the main notifier. These studies are summarized in the Table below. The detailed assessment of these studies is presented in Appendix 2.

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 (DAT)	Re-marks	
EU data								
Leafy vegetables	Lettuce Cabbages	3,6-dichloro-2,6-14C-2-pyridinecarboxylic acid	F	280 g a.i./ha	30, 125, 319	128, 390	-	RMS, 2018 Yackovich, P. R. ; Lardie, T. S. ; Brink, D. L., 1993
Root and tuber vegetables	Turnip Radishes				30, 125, 319	390		
Pulses and oilseeds	Green bean, soybean				125, 319	390		
Cereals	Wheat				30, 125,	62, 417		
								RMS, 2018 Yackovich, P.R.; Lardie T.S.; Miller

					319			J.H., 1989, Study No. GH-C 2277 RMS, 2018 Hall, L. R.; 2015; Lab Study No. 69725; DAS Study No. 130733	
New data									
Leafy vegeta- bles	Cabbage	[14C]-Clopyralid.	G	0.300 kg/ha	30			Rooney P., FR/001647, 2021	
Root and tuber vegetables	Radish								42 93 114
Cereals	Spring wheat								42 64 63 77 139

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

Summary of plant metabolism studies reported in the EU

Three nature of residues studies in three rotational crops covering the plant-back interval (PBI) of ca 30, 120 and 365 days are available. Negligible residue levels were present in turnips, beans and wheat (immature, chaff, straw, grain) planted after 390 to 417 days after clopyralid treatment. 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.

Conclusion on metabolism in rotational crops

All metabolism data are active substance data and were evaluated in the EU review of clopyralid. The detailed studies about metabolism in rotational crops are presented in Draft Assessment Report (DAR, 2005) and renewal assessment report (RAR, 2018). The metabolic pattern of clopyralid in rotational crops is deemed similar to the one depicted in primary crops, thus the same residue definitions are applicable. Additional studies are not regarded as necessary.

7.2.2.3 Nature of residues in processed commodities (KCA 6.5.1)

Available data

One new hydrolysis study has been submitted by the applicant in the framework of this application. The study was submitted as equivalent to protected hydrolysis study and was accepted in data matching. CIECH Sarzyna S.A. possess Letter of Access from the Task Force Clopyralid to alternative data package for active substance Clopyralid. Equivalent nature of the residues in processed commodities study was included in Data Matching List. This Data Matching List covers all the protected studies from the main notifier. This study is summarized in the table below. The detailed results of this study are presented in Appendix 2.

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

Conditions (Duration, Temperature, pH)	Identified compound(s) (%)	Reference
EU data		
Pasteurisation (20 minutes, 90°C, pH 4)	Parent (99.3%)	Adusumilli, H.; 2014; Lab Study No. 140574; DAS Study No. 14057
Baking, boiling, brewing (60 minutes, 100°C, pH 5)	Parent (96.9%)	
Sterilisation (20 minutes, 120°C, pH 6)	Parent (97.1%)	
New data		
Pasteurisation (20 minutes, 90°C, pH 4)	Clopyralid	Hamnett K., 2019, FR/001648
Baking, boiling, brewing (60 minutes, 100°C, pH 5)		
Sterilisation (20 minutes, 120°C, pH 6)		

Conclusion on nature of residues in processed commodities

In conclusion, no hydrolysis of 14C-clopyralid occurred in pH 4 aqueous buffer heated to 90°C for 20 minutes, pH 5 aqueous buffer heated to 100°C for 60 minutes, or pH 6 aqueous buffer heated to 120°C for 20 minutes.

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 (Sugarbeet) Leafy crops (Cabbage) Pulses/Oilseed (Oilseed rape)
Rotational crops covered	Yes Root and tuber vegetables (Turnip, Radish) Leafy crops (Lettuce, Cabbage) Cereals (Wheat) Other (Soybean)
Metabolism in rotational crops similar to metabolism in primary crops?	Yes
Processed commodities	a.s. is stable under standard hydrolysis conditions
Residue pattern in processed commodities similar to pattern in raw commodities?	No changes proposed in residue definition on basis of hydrolysis test. Only parent has been tested. Clopyralid conjugates are also major residue and included in the proposed residue definitions.
Plant residue definition for monitoring	clopyralid (COMMISSION REGULATION (EU) 2021/1807 of 13 October 2021) Applicable only for cereals/grass: clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) (EFSA, 2021) **
Plant residue definition for risk assessment	clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of “polar clopyralid” (EFSA

	Journal 2018;16(7):5389)
Conversion factor from enforcement to RA	Residue definitions are the same, i.e. conversion factor is not needed.

* If residue pattern in processed commodities is not similar to that in raw commodities

** A more recent proposal by EFSA may be provided as additional information (EFSA RO 2021).

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

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	¹⁴ C-ring labelled 3,6-dichloropicolinic acid (Dowco 290), labelled in the 2,6- position	2	230 and 69 ppm	7	Milk	twice daily	DAR, 2003
						Urine and faeces	daily	
						Tissues	at sacrifice	
	Goat	¹⁴ C-clopyralid	1	50.9 mg a.s./kg dry feed/day equivalent to 0.484 mg/kg bw per day	7	Milk	twice daily	RAR 2018 2015; Lab Study No. 130202; Study No. 130202;
						Urine and faeces	daily	
						Tissues	at sacrifice	
Laying poultry	Hens	¹⁴ C-clopyralid, labelled in the 2,6 position	2	100 ppm of test material in the feed	5	Eggs	daily	DAR, 2003 xxx and xxx, 1974
						Excreta	daily	
						Tissues	at sacrifice	
	Hens	¹⁴ C-clopyralid	6	100 ppm	14	Eggs	daily	DAR, 2003 xxx xxx, xxx 1974
						Excreta	daily	
						Tissues	at sacrifice	
Hens	¹⁴ C-clopyralid pyridine ring radiolabeled	-	11.4 mg/kg dry feed /day, Equivalent to 0.56-0.65 mg/kg bw/d	7	Eggs	twice daily	RAR, 2018, 2014; Lab Study No. 130906; Study No. 130906;	
					Excreta	daily		
					Tissues	at sacrifice		

Summary of animal metabolism studies reported in the EU

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.

Conclusion on metabolism in livestock

Metabolism in animals has been thoroughly characterized in rats, poultry and lactating ruminants. Metabolism is similar in the animals tested, and no further studies are required.

Referring to the Peer review of the pesticide risk assessment of the active substance clopyralid (EFSA 2018), the log P_{ow} for clopyralid acid is < 3 therefore the active substance will not bio accumulate and it is not necessary to present or conduct a metabolism study in fish.

No further consideration is necessary.

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 (COMMISSION REGULATION (EU) 2021/1807 of 13 October 2021) Clopyralid and its salts OR clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid) (EFSA, 2021)*
Animal residue definition for risk assessment	clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid) (EFSA Journal 2018;16(7):5389)**
Conversion factor	The conversion factor monitoring / risk assessment is only relevant for milk and is based on the new ruminant metabolism study as 1.3. (RMS, 2018, EFSA Journal 2018;16(7):5389)
Metabolism in rat and ruminant similar	Yes
Fat soluble residue	No

* A more recent proposal by EFSA may be provided as additional information (EFSA RO 2021)

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

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

Data supporting the intended uses of K-300SL-RR are summarised in the table below. These data were presented and evaluated in registration report (dated 03.2013) based on which first authorisation on Faworyt 300 SL was granted (authorisation No R - 140/2013). Data are still valid and meet criteria of current guidelines.

Table 7.2-9: Summary of EU reported and new data supporting the intended uses of K-300SL-RR and conformity to existing MRL

Commodity	Source	Residue zone (N-EU, S-EU, EU, outside EU)	Evaluation GAP Residue levels (mg/kg) E = according to enforcement residue definition RA = according to risk assessment residue definition	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg) *	MRL compliance	
Cereals grain	RMS, 2005	N-EU	GAP on which EU a.s. assessment is based: 1 x 0.150 kg as/ha, BBCH 39, outdoor 0.07, 0.14, 0.23, 0.24, 0.34, 0.37, 0.38, 0.47, 0.61, 0.73, 0.79, 0.82, 0.93, 0.95, 1.06, 1.11, 1.26	N/A					
	Overall supporting data for cGAP	N-EU	0.07, 0.14, 0.23, 0.24, 0.34, 0.37, 0.38, 0.47, 0.61, 0.73, 0.79, 0.82, 0.93, 0.95, 1.06, 1.11, 1.26	0.61	1.26	-	3	Yes	
	New study S20-04397-01	N-EU	1 x 0.153,9 kg as/ha, BBCH 39 0.79						
	New study S19-01810-01	N-EU	1 x 0.159 kg as/ha, BBCH 39 0.76						
Cereals straw	RMS, 2005	N-EU	GAP on which EU a.s. assessment is based: 1 x 0.150 kg as/ha, outdoor n.a, 0.12, 0.17, 0.28, 0.31, 2 x 0.32, 0.33, 0.40, 0.43, 0.50, 0.58,	N/A					

			0.63, 0.81, 0.87, 1.05, 1.08,						
	Overall supporting data for cGAP	N-EU	n.a, 0.12, 0.17, 0.28, 0.31, 2 x 0.32, 0.33, 0.40, 0.43, 0.50, 0.58, 0.63, 0.81, 0.87, 1.05, 1.08,	0.4	1.08	-	-	-	
	New study S20-04397-01	N-EU	1 x 0.153,9 kg as/ha, BBCH 39 2.87						
	New study S19-01810-01	N-EU	1 x 0.159 kg as/ha, BBCH 39 2.72						
Oilseed rape	RMS, 2005	N-EU	Trials GAP: 1 x 0.1 kg as/ha + 1 x 0.2 kg as/ha, outdoor <0.01, 0.01, 2 x 0.02, 0.03, 0.04, 0.05, 0.1	N/A					
	Overall supporting data for cGAP	EU	<0.01, 0.01, 2 x 0.02, 0.03, 0.04, 0.05, 0.1	0.025	0.1	-	0.5	Yes	
Sugar beet roots	RMS, 2005	N-EU	GAP on which EU a.s. assessment is based: 1 x 0.1 kg as/ha + 1x 0.2 kg/ha latest timing of BBCH 39 0.12, 0.17, 0.21, 0.29, 0.34, 0.35, 0.36, 0.41, 0.56, 0.80	N/A					
	Overall supporting data for cGAP	N-EU	0.12, 0.17, 0.21, 0.29, 0.34, 0.35, 0.36, 0.41, 0.56, 0.80	0.345	0.8	-	1	Yes	
Sugar beet tops	RMS, 2005	N-EU	GAP on which EU a.s. assessment is based: 1 x 0.1 kg as/ha + 1x 0.2 kg/ha latest timing of BBCH 39 0.13, 0.14, 0.23, 0.36, 0.46, 2 x 0.47, 0.57, 0.62, 1.05	N/A					
	Overall supporting data for cGAP	N-EU	0.13, 0.14, 0.23, 0.36, 0.46, 2 x 0.47, 0.57, 0.62, 1.05	0.465	1.05	-	-	-	

* Source of EU MRL: COMMISSION REGULATION (EU) 2021/1807 of 13 October 2021

7.2.3.2 Conclusion on the magnitude of residues in plants

According to the available data, the intended uses on wheat, ~~oilseed rape and sugar beet~~ are considered acceptable, for outdoor use.

The data submitted show that no exceedance of the MRL will occur.
 The uses are considered acceptable.

7.2.4 Magnitude of residues in livestock

7.2.4.1 Dietary burden calculation

Table 7.2-10: Input values for the dietary burden calculation (considering the intended uses)

Feed Commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid)				
Rape meal	0.025 x 2	Median residue x PF	-	-
Canola meal	0.025 x 2	Median residue x PF	-	-
Wheat grain	0.61	STMR (wheat and barley)	-	-
Distiller's grain dried	0.61	STMR (wheat and barley)	-	-
Wheat gluten meal	0.61	STMR (wheat and barley)	-	-
Wheat milled by pdts	0.61	STMR (wheat and barley)	-	-
Wheat straw	0.4	STMR	1.08	HR
Beet sugar dried pulp	0.345 x 18	Median residue x PF	-	-
Beet sugar ensiled pulp	0.345 x 3	Median residue x PF	-	-
Beet sugar molasses	0.345 x 28	Median residue x PF	-	-
Beet sugar tops	0.47	STMR	1.05	HR

Table 7.2-11: Results of the dietary burden calculation

Animal species	Median dietary burden (mg/kg bw/d)	Maximum dietary burden (mg/kg bw/d)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)
clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid)					
Beef cattle*	0.0577	0.07	Beet, sugar ensiled pulp	2.91	Y
Dairy cattle*	0.137	0.167	Beet, sugar ensiled pulp	4.34	Y
Ram/ewe	0.087	0.134	Beet, sugar dried pulp	4.01	Y
Lamb	0.149	0.170	Beet, sugar dried pulp	4.01	Y
Breeding swine	0.067	0.073	Wheat milled by pdts	3.16	Y

Animal species	Median dietary burden (mg/kg bw/d)	Maximum dietary burden (mg/kg bw/d)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)
Finishing swine*	0.083	0.083	Wheat milled bypds	3.16	Y
Broiler poultry	0.102	0.102	Wheat milled bypds	1.45	Y
Layer poultry*	0.106	0.115	Wheat milled bypds	1.68	Y

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

7.2.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

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

Table 7.2-12: Overview of the values derived from livestock feeding studies

Commodity	Dietary burden		Results of the livestock feeding study				Median residue (mg/kg) ^(b)	Highest residue (mg/kg) ^(c)	Calculated MRL (mg/kg)
	Med. (mg/kg bw/d)	Max. (mg/kg bw/d)	Dose Level (mg/kg bw/d) ^(a)	No	Result for enforcement/RA				
					Mean (mg/kg)	Max. (mg/kg)			
EU data (RAR, 2018)									
Residue definition for enforcement and risk assessment: clopyralid , its salts and conjugates, expressed as clopyralid									
Pig meat	0.147	0.209	0.451	4	<0.01(0.007)	<0.01(0.007)	0.002	0.004	0.05 ^d
			1.670	4	0.023	0.029			
			8.571	4	0.104	0.113			
Pig fat	0.147	0.209	0.451	4	0.023	0.041	0.007	0.019	0.05 ^d
			1.670	4	0.109	0.264			
			8.571	4	0.519	1.048			
Pig liver	0.147	0.209	0.451	4	0.032	0.036	0.010	0.017	0.05 ^d
			1.670	4	0.112	0.145			
			8.571	4	0.502	0.560			
Pig kidney	0.147	0.209	0.451	4	0.429	0.606	0.140	0.281	0.3
			1.670	4	1.460	1.559			
			8.571	4	5.100	6.030			
Ruminant meat	0.411	0.719	0.451	4	<0.01(0.007)	<0.01(0.007)	0.006	0.015	0.08 ^d
			1.670	4	0.023	0.029			
			8.571	4	0.104	0.113			
Ruminant fat	0.411	0.719	0.451	4	0.023	0.041	0.024	0.081	0.09

			1.670	4	0.109	0.264			
			8.571	4	0.519	1.048			
Ruminant liver	0.411	0.719	0.451	4	0.032	0.036	0.033	0.071	0.08
			1.670	4	0.112	0.145			
			8.571	4	0.502	0.560			
Ruminant kidney	0.411	0.719	0.451	4	0.429	0.606	0.445	1.197	1.5
			1.670	4	1.460	1.559			
			8.571	4	5.100	6.030			
Poultry meat	0.145	0.147	0.280	3	<0.01 (0.005)	0.011	0.003	0.006	0.05 ^d
			0.571	3	<0.01 (0.009)	0.011			
			1.086	3	<0.01 (0.005)	<0.01 (0.005)			
Poultry fat	0.145	0.147	0.280	3	ND (<0.003)	ND (<0.003)	0.002	0.002	0.05 ^d
			0.571	3	ND (<0.003)	<0.01 (0.006)			
			1.086	3	ND (<0.003)	ND (<0.003)			
Poultry liver	0.145	0.147	0.280	3	0.019	0.032	0.010	0.017	0.05 ^d
			0.571	3	0.023	0.033			
			1.086	3	0.017	0.027			
Milk	0.411	0.719	0.451	36	ND	N/A	0.002	0.003	0.05 ^d
			1.670	36	<0.01 (0.008)	N/A			
			8.571	36	0.040	N/A			
Eggs	0.145	0.147	0.280	3	ND (<0.003)	<0.01 (0.005)	0.002	0.003	0.05 ^d
			0.571	3	<0.01 (0.004)	0.011			
			1.086	3	0.011	0.018			

N/A: Not applicable – only the mean values are considered for calculating MRLs in milk.

n.r.: Not reported

(*): Indicates that the MRL is set at the limit of analytical quantification.

(F): MRL is expressed as mg/kg of fat contained in the whole product.

- (a): Dose levels from feeding study are displayed in units of mg/kg bw/d and shown here are the corresponding dose levels from the study when expressed as mg/kg dry feed (DM): 0.451 mg/kg bw/d corresponds to 16.7 mg/kg feed DM; 1.670 mg/kg bw to 56.6 mg/kg feed DM; and 8.571 mg/kg bw/d corresponds to 309.8 mg/kg feed DM.
- (b): Median residue value according to the enforcement residue definition, derived by interpolation/extrapolation from the feeding study for the median dietary burden (FAO, 2009).
- (c): Highest residue value (tissues, eggs) or mean residue value (milk) according to the enforcement residue definition, derived by interpolation/extrapolation of the maximum dietary burden between the relevant feeding groups of the study (FAO, 2009).
- (d): Propose to retain the current EU MRL, which is the value displayed, since the data presented here did not indicate an increase in the current EU MRL is needed.

Conclusion on feeding studies

The requested uses (or the new mode of calculation) modify the theoretical maximum daily intake for animals, but regarding available feeding data, there is no risk for animal MRL 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

Summary of processing studies reported in the EU

Rapeseed

A total of 15 studies were conducted during 197-1985 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.

Sugar beet

A number of studies were conducted during 1984-1987 to determine the residues of clopyralid present in commercial samples of the process fractions of sugar beet as a result of treating the crop with clopyralid. Samples of sugar beet molasses and/or massecuite (massecuite is a green syrup from which molasses is prepared) were collected from different factories. No special procedures were set up for a study and samples were taken from normally production runs. Clopyralid were then extracted from the molasses and/or massecuite and analysed by using as chromatographic method.

The data indicated that clopyralid was concentrated during the processing from massecuite to molasses. Since no data for the water content of samples were available, it was not possible to draw conclusion whether this increase was in parallel to the concentration of the sugar content of the syrup. However, despite the fact that clopyralid residues were concentrated during processing of sugar beet, the residues were below the limit of detection in refined sugar. The low levels of residues indicate the changes to the quality to the fresh or processed products are unlikely

Processing studies were performed with rapeseed, and commercial sugar beet processing fractions have been monitored. No concentration of clopyralid was observed in oil samples. Clopyralid residues were concentrated during processing of sugar beet, but residue levels were below the limit of detection in refined sugar.

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

CIECH Sarzyna S.A. possess Letter of Access from the Task Force Clopyralid to alternative data package for active substance Clopyralid. Equivalent processing study was included in Data Matching List. This Data Matching List covers all the protected studies from the main notifier.

Table 7.2-13: Overview of the available processing studies

Processed commodity	Number of studies	Median PF*	Median CF*	Comments	Reference
EU data					

Processed commodity	Number of studies	Median PF *	Median CF *	Comments	Reference
clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)					
Wheat / bran	4	6.1/5.2	N/A	-	RMS, 2018 Devine, H.C., 2008., DAS Study / Report number: Study report no. GHE-P-11684
Wheat / white flour	4	0.3/0.3	N/A	-	
Wheat / wholemeal flour	2	1/1	N/A	-	
Wheat / germ	2	3.3	N/A	-	
Wheat / white bread	2	0.1/0.1	N/A	-	
Wheat / wholemeal bread	2	0.6/0.6	N/A	-	
Barley / malt sprouts	2	0.2/0.2	N/A	-	
Barley / brewing malt	2	0.7/0.7	N/A	-	
Barley / spent grains and flocs	2	0.2/0.2	N/A	-	
Barley / brewer's yeast	2	0.1/0.1	N/A	-	
Barley / beer	2	0.1/0.1	N/A	-	
New data					
Total bran	2	1.06/0.82	N/A	-	White T., 2021, S19-01810 White T., 2021, S20-04397
Shorts	2	0.13/0.11	N/A	-	
Middlings	2	0.44/0.31	N/A	-	
White flour	2	0.15/0.11	N/A	-	
White bread	2	0.10/0.08	N/A	-	
Wholemeal flour	2	1.15/0.99	N/A	-	
Wholemeal bread	2	0.75/0.73	N/A	-	
Wheat germs	2	0.89/0.13	N/A	-	

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

7.2.5.2 Conclusion on processing studies

All processing data are active substance data and were evaluated in the EU review of clopyralid. The detailed studies about processing are presented in Draft Assessment Report (DAR, 2005) and renewal assessment report (RAR, 2018). Wheat and barley samples were processed according to the technical procedures on a laboratory scale comparable to the processes used for commercial or household production of the goods. Clopyralid concentrated in wheat germ and bran. Processing did not have effects on the residue levels in whole meal flour. Residue levels of clopyralid reduced in all other processed fractions of wheat. Residue levels were reduced also during malting and brewing of barley. Processing factors have been established. Additional studies are not regarded as necessary

7.2.6 Magnitude of residues in representative succeeding crops

The crops under consideration can be grown in rotation.

Considering available data dealing with nature of residues (see 7.2.2.2), no study dealing with magnitude of residues in succeeding crops is needed.

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

Faworyt 300 SL is intended to be applied in the following crops: wheat, sugar beet and oilseed rape. Wheat and sugar beet are not considered to have melliferous capacity. For oilseed rape Faworyt 300 SL is applied before the flowering stage (BBCH 10-50). Therefore there is no need to determining the magnitude of pesticide residues in honey.

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of Faworyt 300 SL. Therefore, other special studies are not needed.

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

Table 7.2-14: Input values for the consumer risk assessment

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid)				
Wheat grain	2	MRL (according to COMMISSION REGULATION (EU) 2021/1807 of 13 October 2021)	2	MRL (according to COMMISSION REGULATION (EU) 2021/1807 of 13 October 2021)
Oilseed rape seeds	0.5		0.5	
Sugar beet roots	1		1	
Bovine, sheep and goat muscle	0.08		0.08	
Bovine fat and liver	0.15		0.15	
Bovine kidney	1.5		1.5	
Sheep and goat fat and liver	0.2		0.2	
Sheep and goat kidney	2		2	
Bovine, sheep and goat edible offals and other products	0.05		0.05	
Poultry, Equine and Other farm animals origins	0.05		0.05	
Milk	0.05		0.05	
Eggs	0.05		0.05	
Other	various		-	

7.2.8.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented in Appendix 3.

Table 7.2-15: Consumer risk assessment

TMDI (% ADI) according to EFSA PRIMo rev 3.1	39 % (based on NL toddler)
IEDI (% ADI) according to EFSA PRIMo rev 3.1	TMDI does not exceed ADI therefore IEDI calculations are not required
IESTI (% ARfD) according to EFSA PRIMo* rev 3.1	Unprocessed 25 % Wheat (Children) 15 % Wheat (Adults) Processed 65% Sugar beets (roots)/sugar (Children) 26% Sugar beets (roots)/sugar (Adults)
NTMDI (% ADI) **	Not relevant
NEDI (% ADI)**	Not relevant
NESTI (% ARfD) **	Not relevant

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

** if national model is available

The proposed uses of clopyralid in the formulation Faworyt 300 SL 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 Journal 2018;16(8):5389, Peer review of the pesticide risk assessment of the active substance clopyralid

EFSA Journal 2021;19(1):6389, Modification of the existing maximum residue levels for clopyralid in various commodities

Draft Assessment Report (DAR), Volume 3, Annex B, B.7, February 2005

Appendix 1 Lists of data considered in support of the evaluation

Tables considered not relevant can be deleted as appropriate.
 MS to blacken authors of vertebrate studies in the version made available to third parties/public.

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
KCA 6.5.1	Hamnett K	2019	Nature of ¹⁴ C-Clopyralid in Processed Commodities – High Temperature Hydrolysis Fera Study Number: FR/001648 Fera GLP Unpublished	N	Clopyralid TF
KCA 6.6.1	Rooney P.	2021	[¹⁴ C]-Clopyralid Metabolism in Rotational Crops Fera FR/001647 GLP Unpublished	N	Clopyralid TF
KCA 6.5.2-6.5.3/01	White T.	2021	Determination of Residues of Clopyralid after One Application of Major 300 SL (CHR/H/CPD 300SL) in Winter Wheat. One site in Northern France and One Site in Southern France During 2019 S19-01810 GLP Unpublished	N	Clopyralid TF
KCA 6.5.2-6.5.3/02	White T.	2021	Determination of Residues of Clopyralid after One Application of Major 300 SL (CHR/H/CPD 300SL) in Spring Wheat. One site in Northern France During 2020 S20-04397 GLP Unpublished	N	Clopyralid TF

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 6.1/01	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/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.1/02	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/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.1/03	Dial, E., Lindsay, D	2006	Frozen Storage Stability of Clopyralid in Oilseed Rape DAS Study No. 020122.02 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.1/04	-	2015	Frozen Storage Stability of Clopyralid in Bovine Fat Study No. 120602 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KC 6.1/05	-	2004	Frozen Storage Stability of Clopyralid in Beef Muscle, Liver, Kidney, Milk and Chicken Egg Study No. 020120.01 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.2.1/01	Chapleo, S. ; Caley, C. Y.	2002	The Metabolism of [14C]-Clopyralid in Sugar Beet DAS Study No. GHE-P-9939 Inveresk Research International, Tranent, East Lothian, United Kingdom GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA	Chapleo, S.,	2002	The Metabolism of (14C)-Clopyralid in Oilseed Rape	N	DAS

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
6.2.1/02	Caley, C. Y., White, D. E.		DAS Study No. GHE-P 9938 Inveresk Research International, Tranent, East Lothian, UK GLP/GEP (Y/N): Yes Published (Y/N): No		
KCA 6.2.1/03	Guo, C.	1996	Metabolism of ¹⁴ C -Clopyralid in Cabbage DAS Study No. RES95095 DAS Report No. GH-C-4289 ABC Laboratories Inc, Columbia, Missouri, USA GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.2.2- 6.2.5/01	xxx	1974	The Fate of ¹⁴ C-labelled DOWCO 290 Fed as a Single Oral Dose to Broiler Chicken DAS Report No: GH-C 740 GLP/GEP (Y/N): N Published (Y/N): N	N	DAS
KCA 6.2.2- 6.2.5/02	xxx	1974	Fate of ¹⁴ C-DOWCO 290 in Laying Hens DAS Report No: GH-C 726 GLP/GEP (Y/N): N Published (Y/N): N	N	DAS
KCA 6.2.2- 6.2.5/03	-	2014	A Nature of the Residue Study in the Laying Hen with [¹⁴ C]- Clopyralid Study No. 130906 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS
KCA 6.2.2- 6.2.5/04	-	2015	A Nature of the Residue Study in the Ruminant with [¹⁴ C]Clopyralid Study No. 130202 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS
KCA 6.6.1/01	Yackovich, P. R. ; Lardie, T. S. ; Brink, D. L.	1993	A 10-1/2 Month Rotational Crops Study With ¹⁴ C - Labeled Clopyralid - MET90080 DAS Study No. GH-C 2992 Dow AgroSciences LLC, Indianapolis, Indiana, United States GLP/GEP (Y/N): Yes	N	DAS

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
			Published (Y/N): No		
KCA 6.6.1/02	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 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.6.1/03	Hall, L (Y/N): No	2015	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/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/01	Jones E.M., Yuill M.M	1976	Determination od residues of 3,6-dichloropicolinic Acid (DOWCO 290) in Rape Seed, Oil and Cake from 1975 Trials Carried Out by the Boots Company Limited DAS Report No. GHE-P-325 GLP/GEP (Y/N): No Published (Y/N): No	N	DAS
KCA 6.3/02	Jones E.M., Yuill M.M	1976	Determination od 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 DAS Report No. GHE-P-337 GLP/GEP (Y/N): No Published (Y/N): No	N	DAS
KCA 6.3/03	Rawle N.W., Khoshab A.	2002	Residues of Clopyralid in Oilseed Rape at Intervals and at Harvest Following Multiple Applications of Lontrel 100 (EF-1136), EU Northern Zone – 2001 DAS Report No. GHE-P-9380 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/04	Freeman J.M.H, Walker S.S.	1980	Determination od residues of 3,6-dichloropicolinic Acid (DOWCO* 290) in Sugar Beet, Roots and Tops, Treated with FORMAT** - UK 1980 DAS Report No. GHE-P-803	N	DAS

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
			GLP/GEP (Y/N): No Published (Y/N): No		
KCA 6.3/05	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Sugarbeet at Intervals Under Open Field Conditions Following Multiple Applications of Lontrel 100 (EF-1136), Northern France and UK -2000 DAS Report No. GHE-P-9356 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/06	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Sugarbeet at Harvest Under Open Field Conditions Following Multiple Applications of Lontrel 100 (EF-1136), Northern France and UK -2000 DAS Report No. GHE-P-9357 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/07	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Sugarbeet at Intervals and at Harvest Following Multiple Applications of Lontrel 100 (EF-1136), EU Northern Zone -2001 DAS Report No. GHE-P-9381 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/08	Freeman, JHM et al	1982	Effect of Length of Peiod Between Application of Cyronal* and Harvest on Residues of 3,6-dichloropicolinic Acid (DOWO 290**) in Winter Wheat, Winter Barley and Maize- Belgium 1981 DAS Report No. GHE-P-943 GLP/GEP (Y/N): No Published (Y/N): No	N	DAS
KCA 6.3/09	Freeman, JHM	1984	Clopyralid Residues in Wheat Grain and Straw Treated with Either LONPAR* or LONTREL * 100 from French Trials, 1983 DAS Report No. GHE-P-1258 GLP/GEP (Y/N): No Published (Y/N): No	N	DAS
KCA 6.3/10	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Wheat at Intervals Under Open Field Conditions Following a Single Application of Lontrel 100 (EF-1136), UK and Germany -2000	N	DAS

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
			DAS Report No. GHE-P-9358 GLP/GEP (Y/N): Yes Published (Y/N): No		
KCA 6.3/11	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Wheat at Intervals Following a Single Application of Lontrel 100 (EF-1136), EU Northern Zone -2001 DAS Report No. GHE-P-9385 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/12	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Barley at Intervals and at Harvest Following a Single Application of Lontrel 100 (EF-1136), EU Northern Zone -2001 DAS Report No. GHE-P-9383 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/13	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Barley at Intervals Under Open Field Conditions Following a Single Application of Lontrel 100 (EF-1136), EU Northern Zone -2000 DAS Report No. GHE-P-9360 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.3/14	Rawle N.W., Khoshab A	2002	Residues of Clopyralid in Barley at Harvest in Open Field Conditions Following a Single Application of Lontrel 100 (EF-1136), EU Northern Zone -2000 DAS Report No. GHE-P-9359 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
KCA 6.5.1	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/GEP (Y/N): Yes Published (Y/N): No	N	DAS

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 6.4.1-6.4.3	-	2015	Summary of Clopyralid Livestock Feeding Study: Magnitude of Residue in Eggs, Muscle, Liver and Fat of Laying Hens Study No. 150031 Lab Study No. 6921 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS
KCA 6.5.2-6.5.3	Device H	2006	Residues of clopyralid in wheat and process fractions at harvest following a single application of EF-1498, Northern France - 2005 DAS Study No. GHE-P-11274 CEM Analytical Services - UK GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS

The following tables are to be completed by MS.

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

No new or additional studies have been submitted.

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

No new or additional studies have been submitted.

A 2.1.2.1 Nature of residue in plants

A 2.1.2.1.1 Nature of residue in primary crops

No new or additional studies have been submitted.

A 2.1.2.1.2 Nature of residue in rotational crops

A 2.1.2.1.2.1 Study 1

Comments of zRMS:	The study was submitted as equivalent to protected study and was accepted in “data matching”. The requirement for alternative tests has been met. This study should be evaluated at EU level.
-------------------	---

Reference: 6.6.1

Report: [14C]-Clopyralid Metabolism in Rotational Crops, Rooney P., 2021, FR/001647

Guideline(s): Yes (OECD Guideline for the Testing of Chemicals, Number 502)

Deviations: Yes (Deviations has no further impact on the study)

GLP: Yes

Acceptability: Yes

Materials and methods

[14C]-Clopyralid, supplied with dual Carbon 14 isotopes labelled within the pyridine function group within the molecule was applied to test plots at a nominal rate of 0.300 kg/ha. Four test plots were setup containing soil. Isotopically diluted [14C]-Clopyralid was applied to the soil containers (test plots), the study objectives were to; quantify the concentrations of total radioactive residue (TRR) in crop samples at various times after application, quantify major components (metabolites) of the TRR in crop samples and show their efficiency of extraction, identify major components (metabolites) of the TRR >0.05 ppm or >10% TRR and characterise those between 0.01 – 0.05 ppm as appropriate and to propose a metabolic

pathway for the metabolism of the test item.

Methods: The field phase of the study was conducted indoors at AgroChemex, Manningtree, Essex. The sandy loam soil for use in this study was obtained by the Test Site. A sample of soil was characterised by CIP. The sample of soil was characterised for soil texture, organic matter content, particle size distribution and pH.

A single application was made to soil containers, pre-filled with the sandy loam soil (FERA ACE19-015). The nominal rate of application was 0.300 kg/ha and at a simulated spray volume of 349 L/ha. The application was made using a spray device based on a single cone nozzle. A water rinse of the spray equipment was applied immediately following application to the soil. The containers were enclosed within closed spray chambers lined with polyethylene sheeting during application which took place on the 17 th April 2019. The spray apparatus and plastic sheeting were returned to the test facility and the quantity of radioactive residues were measured confirming successful application of the test item application solutions. 30 days following the application, Spring Wheat (*Triticum aestivum*), Radish (*Raphanus sativa*) and Cabbage (*Brassica oleracea*) were planted in separate treated tubs. Following growth, the planted crops were sampled and shipped to the test facility for analysis.

Samples were harvested and stored on dry ice and shipped to Fera. Upon receipt at Fera, harvested samples were stored frozen prior to analysis. When required crops were removed from storage, homogenised and sample oxidised to determine total recovered radioactivity. If more than 0.01 mg kg⁻¹ was determined from the sample oxidiser process, then the sample type was taken for radioactive characterisation via extraction and liquid scintillation counting (LSC), and potentially high pressure liquid chromatography connected to ultra violet detection coupled to a beta radioactivity analysis module (HPLC-UV-βRAM) or thin layer chromatography (TLC) analysis.

Analysis using high pressure liquid chromatography coupled to a beta radioactivity analysis module (HPLC-UV-βRAM) was performed to assess the radio-chemical purity of the [14C]- Clopyralid application solution using the gradient method detailed below.

HPLC Method 1: Radiochemical Purity Analysis and reference standards Non-Stop (Direct Injection)

Method ID

FR001647 HPLC M1

HPLC Apparatus

HPLC Instrument: Agilent 1100 HPLC System
UV Detector: Agilent 1100 MWD UV Detector
HPLC Pump: Agilent 1100 Quaternary Pump
Auto Sampler: Agilent 1100 Auto sampler
Column Oven: Agilent 1100 Column Oven
System Controller: Laura 4 (Version 4.1.7.70)

Liquid chromatography settings

Column: ACE Excel 5, C18-PFP
Mobile Phase: A = 0.1% phosphoric acid in water
B = Acetonitrile
Run time: 35 minutes
Flow rate: 1.0 mL min⁻¹
UV Wavelength: 278 nm
Temperature: 25 °C

Radio analysis module

Radio-detector: LabLogic Model 5 βRAM
Cell Type: Liquid
Cell volume: 500 μL
Scintillant / Eluate ratio: 3.0 mL / 1.0 mL
Measurement Type: Direct injection

Gradient Elution

Time (mins)	% A	% B
0	95.0	5.0
5	95.0	5.0
25	10.0	90.0
28	10.0	90.0
35	95.0	5.0

HPLC Method 2: Sample extract analysis, radiochemical purity via stop-flow

Method ID

FR001647 system 4 stop flow & FR/001647 system 5 stop flow

HPLC Apparatus

HPLC Instrument: Agilent 1100 HPLC System
UV Detector: Agilent 1100 MWD UV Detector
HPLC Pump: Agilent 1100 Quaternary Pump
Auto Sampler: Agilent 1100 Auto sampler
Column Oven: Agilent 1100 Column Oven
System Controller: Laura 4 (Version 4.1.7.70)

Liquid chromatography settings

Column: ACE Excel 5, C18-PFP
Mobile Phase: A = 0.1% phosphoric acid in water
B = Acetonitrile
Run time: 35 minutes
Flow rate: 1.0 mL min⁻¹
UV Wavelength: 278 nm
Temperature: 25 °C

Radio analysis module

Radio-detector: LabLogic Model 5 βram
Cell Type: Liquid
Cell volume: 500 μL
Scintillant / Eluate ratio: 2.0 mL / 1.0 mL
Measurement Type: Stop-flow – fraction time 10 s, count time 60 s

Gradient Elution

Time (mins)	% A	% B
0	95.0	5.0
5	95.0	5.0
25	10.0	90.0
28	10.0	90.0
35	95.0	5.0

All RAC samples except Radish roots were progressed to a stage two further extraction. The remaining radioactive residues were >0.01 mg kg⁻¹ for Wheat forage, hay and grain, radish leaves, immature and mature cabbage.

Results and discussion

The mean percentages of total radioactivity recovered (MBq kg⁻¹) from each raw agricultural commodity (RAC) sample type was determined prior to extraction via sample oxidation. Table A1 details the total radioactivity per kg of collected sample type. The results are based on the mean of nine aliquots taken from the homogenised sample.

Table A 1: Mean total radioactivity recovered from Wheat, Radish and Cabbage.

Crop	Raw Agricultural Commodity	Total Radioactivity Recovered ^a		Trigger for further analysis (OECD 502, 2007)
		MBq kg ⁻¹	mg kg ⁻¹	
Wheat	Forage	3.213	2.345	YES
	Hay	3.804	2.777	YES
	Grain	5.048	3.685	YES
	Straw	13.114	9.572	YES
Radish	Leaves	0.871	0.636	YES
	Roots	0.092	0.067	YES
Cabbage	Immature	1.088	0.794	YES
	Mature	0.948	0.692	YES

a – Mean of the total radioactivity calculated from nine aliquots taken of the homogenised sample type.

All RAC samples were taken forward for extraction and analysis via stage one.

Table A 2: Summary of the mean extracted and bound residues after stage one extraction.

Crop	Raw Agricultural Commodity	Extract-S1		Post Extraction Solids (PES)		Trigger for further analysis (OECD 502, 2007)	Mass balance
		% TRR	mg kg ⁻¹	% TRR	mg kg ⁻¹		% TRR
Wheat	Forage	77.55	1.819	18.51	0.434	YES	96.06
	Hay	56.30	1.563	31.46	0.874	YES	87.76
	Grain	13.52	0.498	68.87	2.537	YES	82.38
	Straw ^a	22.19	2.124	57.14	5.470	YES	79.33
Radish	Leaves	81.86 ^a	0.521 ^a	10.07	0.064	YES	85.32
	Roots	98.91	0.067	7.05	0.005	NO	105.96
Cabbage	Immature	82.78	0.658	10.43	0.083	YES	93.21
	Mature	88.66	0.614	8.55	0.059	YES	97.21

a – Mean of the total radioactivity calculated from six aliquots taken of the homogenised sample type.

Due to the amount of bound residue that remained within all Wheat samples, Radish leaves and all Cabbage samples, the post extraction residues for these sample types were taken forward for further extraction using a harsher technique. For Radish roots, < 10 % of the total radioactive residues remained bound to the post extraction residue and accounted for only 0.005 mg kg⁻¹ of test item which is below the trigger value detailed in the OECD 502 guidance document.

Table A 3: Summary of stage two extraction with 0.125 M NaOH methanol:water (1:1 v/v) and post extraction solid analysis

Crop	Raw Agricultural Commodity	Extract-S2 (BASIC)			Post Extraction Solids (PES)		Trigger for further analysis (OECD 502, 2007)
		% TRR	mg kg ⁻¹	% of NER	% TRR	mg kg ⁻¹	
Wheat	Forage	17.25	0.405	93.32	1.04	0.024	NO
	Hay	28.44	0.588	90.46	2.64	0.062	NO
	Grain	55.86	1.029	81.18	13.01	0.305	YES
	Straw ^a	43.26	4.141	76.25	13.88 ^b	0.326 ^b	YES
Radish	Leaves	7.27	0.046	71.85	0.59	0.014	NO
Cabbage	Immature	8.76	0.035	84.27	0.43	0.010	NO
	Mature	5.91	0.021	69.06	0.33	0.008	NO

a – Mean of the total radioactivity calculated from three aliquots taken of the homogenised sample type separated into two ~25 g sub-samples at stage one.

b – Theoretical remaining radioactivity

After the 0.125 NaOH in methanol:water (1:1 v/v) extraction a proportion of residues remained within Wheat grain and straw. Wheat grain met the trigger for additional work as more than 10 % of the total recovered radioactive residues remained within the PES. For Wheat straw, less than 10 % of the total radioactive residues remained within the PES, however as this represented 0.326 mg kg⁻¹ the decision was made to continue extraction using a reflux extraction process.

Table A 4: Summary of stage two reflux extraction for Wheat samples with 1.0 M NaOH methanol:water (1:1 v/v) and post extraction solid analysis

Crop	Raw Agricultural Commodity	Extract-S3			Post Extraction Solids (PES)		Trigger for further analysis (OECD 502, 2007)
		% TRR	mg kg ⁻¹	% of NER	% TRR	mg kg ⁻¹	
Wheat	Grain	10.72	0.395	83.43	5.590	0.131	NO
	Straw ^a	6.22	0.595	52.55	3.420	0.080	NO

a – Mean of the total radioactivity calculated from three aliquots taken of the homogenised sample type separated into two ~25 g sub-samples at stage one. However, only five of the replicates were used due to the loss of one of the replicates.

Clopyralid was taken up into each crop tested in this study. Varied rates of metabolism occurred between the RACs. However, the transformation products produced were similar between crop types. The level of uptake into the crop types demonstrate that limited degradation and elimination occurred prior to the plant of the rotational crops 30 days after application to the bare soil surface. Unk-PM-1 and Unk-PM-4 are the primary metabolites of clopyralid across all sample types. Unk-PM-1 is a highly polar component and is likely generated during the uptake and incorporation of clopyralid within the crop during growth and metabolism. Wheat samples demonstrated the largest amount of uptake with Wheat straw demonstrating 9.572 mg kg⁻¹. Relatively minor uptake of Clopyralid was observed within Radish roots of 0.067 mg kg⁻¹. However, this concentration still required extraction and characterisation by HPLC-UV-βRAM.

Proposed Metabolic Profile of clopyralid in rotational crops

No metabolic pathway was generated as part of this study. This does not impact the integrity of the data within the study.

Conclusions

No notable residues were detected within the application apparatus therefore there is no concern regarding the application process. Determination of radioactive residues within Wheat Forage, Hay, Grain and Straw, Radish Roots and Leaves, and immature and mature Cabbage was performed. All raw agricultural commodities (RAC) collected demonstrated uptake of radioactive residues above the OECD 502 trigger of 0.01 mg kg⁻¹. Therefore, all RAC were subject to an initial extraction process. Following the initial extraction and assessment of non-extracted residues a further extraction and analysis procedure was performed on Wheat Forage, Hay, Grain and Straw, Radish leaves, immature cabbage and mature cabbage. The further extraction performed included a basic 0.125 M NaOH extraction and a further acidic reflux extraction. Once the post extraction residue had been reanalysed, no further extraction or digestion was required. After HPLC-UV-βRAM analysis radioactivity was characterised into parent (Clopyralid) and metabolites.

A 2.1.2.1.3 Nature of residues in processed commodities

Comments of zRMS: Study is accepted

Reference:	KCA 6.5.1
Report	Nature of ¹⁴ C-Clopyralid in Processed , Hamnet K., 2019, FR/001648
Guideline(s):	Yes (OECD Guideline for the testing of Chemicals, number 507 Nature of the Pesticide Residues in Processed Commodities - High Temperature Hydrolysis (OECD 2007))
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

The aim of this study was to determine the route of hydrolysis of clopyralid in aqueous solutions when exposed to three different hydrolytic conditions, used to simulate normal food processing practices. The study was performed to represent the nature of the herbicide in processed commodities based on the OECD Guideline for the testing of Chemicals, number 507 Nature of the Pesticide Residues in Processed Commodities - High Temperature Hydrolysis (OECD 2007).

Hydrolysis tests were conducted at a nominal test item concentration of 1.0 mg L⁻¹ in sterile citrate buffer solutions. The study was performed with three different buffer solutions at pH 4, 5 and 6 exposed to conditions selected to simulate typical processing practices. These conditions were selected to represent pasteurisation, baking, brewing and boiling and sterilisation.

HPLC-UV-βRAM was performed to assess the radiochemical purity of the [¹⁴C]-Clopyralid application solution and treated samples, using the gradient method detailed below (HPLC Method 1). All treated samples were analysed by HPLC within 24 hours of sampling. All HPLC analysis was conducted co-injected with 10 µL cold standard Clopyralid to confirm parent retention times.

Results and discussion

Table A 5: Identification of compounds from high temperature hydrolysis study

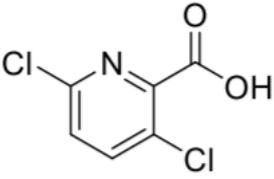
Common name/code ID No.	Chemical name	Chemical structure
Clopyralid	3,6-dichloropyridine-2-carboxylic acid	

Table A 6: Standard hydrolysis study of clopyralid

Process represented	T° (°C)	Time (min)	pH	Parent [14C]-Clopyralid (mg/L) pre exposure	Parent [14C]-Clopyralid (mg/L) post exposure	Recovery of Parent [14C]-Clopyralid (%TAR) pre-exposure	Recovery of Parent [14C]-Clopyralid (%TAR) post-exposure
pasteurization	90 ± 5	20 ± 1	4 ± 0.1	0.97	0.97	98.65	98.85
baking, brewing and boiling	100 ± 5	60 ± 1	5 ± 0.1	0.98	1.00	99.77	101.87
sterilisation	120 ± 5	20 ± 5	6 ± 0.1	0.96	0.96	99.51	99.76

Conclusions

In conclusion, no hydrolysis of [14C]-Clopyralid was observed when exposed to conditions designed to represent high processing procedures; pasteurisation, baking, brewing and boiling and sterilisation. Over the course of all three testing conditions, no hydrolytic products were formed.

A 2.1.3 Magnitude of residues in plants

No new or additional studies have been submitted.

A 2.1.4 Magnitude of residues in livestock

No new or additional studies have been submitted.

A 2.1.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

No new or additional studies have been submitted.

A 2.1.5.1 Distribution of the residue in peel/pulp

No new or additional studies have been submitted.

A 2.1.5.2 Processing studies on a core set of representative processes

A 2.1.5.2.1 Study 1

Comments of zRMS: Study is accepted

Reference: KCA 6.5.2-6.5.3/01

Report Determination of Residues of Clopyralid after One Application of Major 300 SL (CHR/H/CPD 300SL) in Winter Wheat. One site in Northern France and One Site in Southern France During 2019, White T., S19-01810, 2021

Guideline(s): Yes (OECD 508)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods

The objective of the study was to determine residue levels of total clopyralid (clopyralid and clopyralid conjugate (X36538)) in the raw agricultural commodity winter wheat.

Two residue trials were conducted on winter wheat during 2019, one in Northern France (S19-01810-01) and one in Southern France (S19-01810-02). One application of Major 300SL (300 g/L, clopyralid) was applied at 160 g as/ha, diluted with water immediately prior to application to a nominal spray volume of 200 L/ha.

Samples of winter wheat (grain and straw) from the untreated and treated plots were taken by hand 80-93 days (normal commercial harvest) after the single application.

Table A 7: Summary of the study 1 trials – Residues samples

1 Report No. Location (region)	2 Commodity/Variety (a)	3 Date of 1) Sowing or Planting 2) Flowering 3) Harvest (b)	4 Method of Treatment (c)	5 Application rate per treatment			6 Dates of treatment(s) or no. of treatment(s) and last date (d)	7 Growth stage at last treatment or date (e) BBCH	8 Portion analysed (a)	9 Residues (mg/kg) (*)	10 PHI (days) (f)	11 Remarks (g)
				g as/hL	Water (L/ha)	g as/ha						
S19-01810-01 45, Rouvres saint Jean, Loiret, France	Winter wheat/ EPPO code TRZAW / Sacramento	1) 19 Oct 2018 2) 24-31 May 2019 3) 15 Jul 2019	Foliar using a boom sprayer	79.9	199	159	26 Apr 2019	32	Grain Straw	0.76 2.72	80	No residues >LOQ were found in any untreated samples

1 Report No. Location (region)	2 Commodity/Variety (a)	3 Date of 1) Sowing or Planting 2) Flowering 3) Harvest (b)	4 Method of Treatment (c)	5 Application rate per treatment			6 Dates of treatment(s) or no. of treatment(s) and last date (d)	7 Growth stage at last treatment or date (e) BBCH	8 Portion analysed (a)	9 Residues (mg/kg) (*)	10 PHI (days) (f)	11 Remarks (g)
				g as/hL	Water (L/ha)	g as/ha						
S19-01810-02 Castelsarrasin 82100, Tarn et Garonne, France	Winter wheat/ EPPO code TRZAW / Rebelde	1) 10 Nov 2018 2) 10-20 May 2019 3) 04 Jul 2019	Foliar using a boom sprayer	80	204	163	02 Apr 2019	32	Grain Straw	0.70 2.25	93	No residues >LOQ were found in the untreated grain sample. Residues >LOQ were detected in the untreated straw sample

(a) According to EPPO codes

(b) Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

(d) Year must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline); DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

(*) Limit of quantification = 0.044 mg/kg; limit of detection = 0.01 mg/kg; n/d = not detectable

Samples of winter wheat grain taken from both trials were processed into total bran, shorts, middlings, white flour, white bread, wholemeal flour, wholemeal bread and wheat germs at the processing test site.

Table A 8: Summary of the study 1 trials - Processing samples

1 Report No. Location (region)	2 Commodity/Variety (a)	3 Date of 1) Sowing or Planting 2) Flowering 3) Harvest (b)	4 Method of Treatment (c)	5 Application rate per treatment			6 Dates of treatment(s) or no. of treatment(s) and last date (d)	7 Growth stage at last treatment or date (e) BBCH	8 Portion analysed (a)	9 Residues (mg/kg) (*)	10 PHI (days) (f)	11 Remarks (g)
				g as/hL	Water (L/ha)	g as/ha						
S19-01810-01 45, Rouvres saint Jean, Loiret, France	Winter wheat/ EPP0 code TRZAW / Sacramento	1) 19 Oct 2018 2) 24-31 May 2019 3) 15 Jul 2019	Foliar using a boom sprayer	79.9	199	159	26 Apr 2019	32	RAC Grain Total bran Shorts Middlings White flour White bread Wholemeal flour Wholemeal bread Wheat germs	0.68 0.72 0.09 0.30 0.10 0.07 0.78 0.51 Not available	80	No residues >LOQ were found in any untreated samples

(a) According to EPP0 codes

(b) Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

(d) Year must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline); DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

(*) Limit of quantification = 0.044 mg/kg; limit of detection = 0.01 mg/kg; n/d = not detectable

1 Report No. Location (region)	2 Commodity/Variety (a)	3 Date of 1) Sowing or Planting 2) Flowering 3) Harvest (b)	4 Method of Treatment (c)	5 Application rate per treatment			6 Dates of treatment(s) or no. of treatment(s) and last date (d)	7 Growth stage at last treatment or date (e) BBCH	8 Portion analysed (a)	9 Residues (mg/kg) (*)	10 PHI (days) (f)	11 Remarks (g)
				g as/hL	Water (L/ha)	g as/ha						
S19-01810-02 Castelsarrasin 82100, Tarn et Garonne, France	Winter wheat/ EPPO code TRZAW / Rebelde	1) 10 Nov 2018 2) 10-20 May 2019 3) 04 Jul 2019	Foliar using a boom sprayer	80	204	163	02 Apr 2019	32	RAC Grain Total bran Shorts Middlings White flour White bread Wholemeal flour Wholemeal bread Wheat germs	0.71 0.58 0.08 0.22 0.08 0.06 0.70 0.52 0.63	93	No residues >LOQ were found in any untreated samples

(a) According to EPPO codes

(b) Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

(d) Year must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline); DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

(*) Limit of quantification = 0.044 mg/kg; limit of detection = 0.01 mg/kg; n/d = not detectable

The objective of the processing phase was to produce and sample processing fractions (specimens of end products (white flour, white bread, wholemeal flour, wholemeal bread, germs) and specimens of intermediate fractions (bran, shorts, middlings) from winter wheat raw agricultural commodity.

5.2. Equipment and Material

The following list details the equipment and material used during the course of this study:

- Balances - Mettler Toledo
- Desiccator (for moisture analysis) – Ohaus
- Stopwatch – Fisher
- Grinder Mill – 3100 – Perten
- Bran duster – QU100LB4 – BBC Brown Baveri
- Sample cleaner – SLN3 – Pfeuffer
- Oven – 64EXUM2 – Bartsher
- Kneading machine – Franstal
- Drying oven – France Etuve
- Mill – MLU-202 – Bühler
- Centrifuge – G412 – Jouan
- Grinder – Robot Coupe
- Laboratory mill – Biomat senior M3
- Climate chamber – MLR-350HT – Sanyo
- Muffle oven – Mabertherm
- Centrifuge – AN080 X/2 – Abencor

5.3. Reagents

- Table salt – La Baleine – LA190221053
- Yeast – Soframa – 18T1670411

Results and discussions

Table A 9: Residue data from grain processing study with clopyralid

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	TF*	Comments/Reference
Grain	0.68	80	Total bran	0.72	1.06	
			Shorts	0.09	0.13	
			Middlings	0.30	0.44	
			White flour	0.10	0.15	
			White bread	0.07	0.10	
			Wholemeal flour	0.78	1.15	
			Wholemeal bread	0.51	0.75	
			Wheat germs	N/A	Not calculated	Due to processing issues the grains did not germinate and therefore were not available to analyse

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	TF*	Comments/Reference
Grain	0.71		Total bran	0.58	0.82	
			Shorts	0.08	0.11	
			Middlings	0.22	0.31	
			White flour	0.08	0.11	
			White bread	0.06	0.08	
			Wholemeal flour	0.70	0.99	
			Wholemeal bread	0.52	0.73	
			Wheat germs	0.63	0.89	

* transfer factor

Table A 10: Overall Mean Transfer Factors

Process	S19-01810-01-005A & S19-01810-02-005A
Total bran	0.94
Shorts	0.12
Middlings	0.38
White flour	0.13
White bread	0.09
Wholemeal flour	1.07
Wholemeal bread	0.74
Wheat germs	Not calculated

Figure A 1: Processing flowchart for wheat grain - cleaning

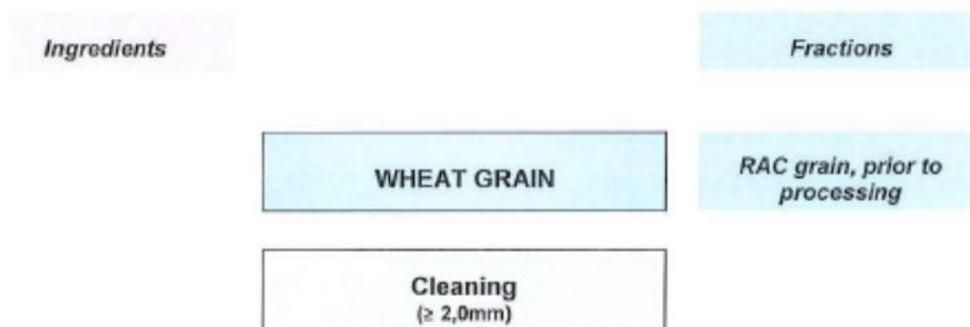


Figure A 2: Processing flowchart for wheat grain – milling (white flour type 550)

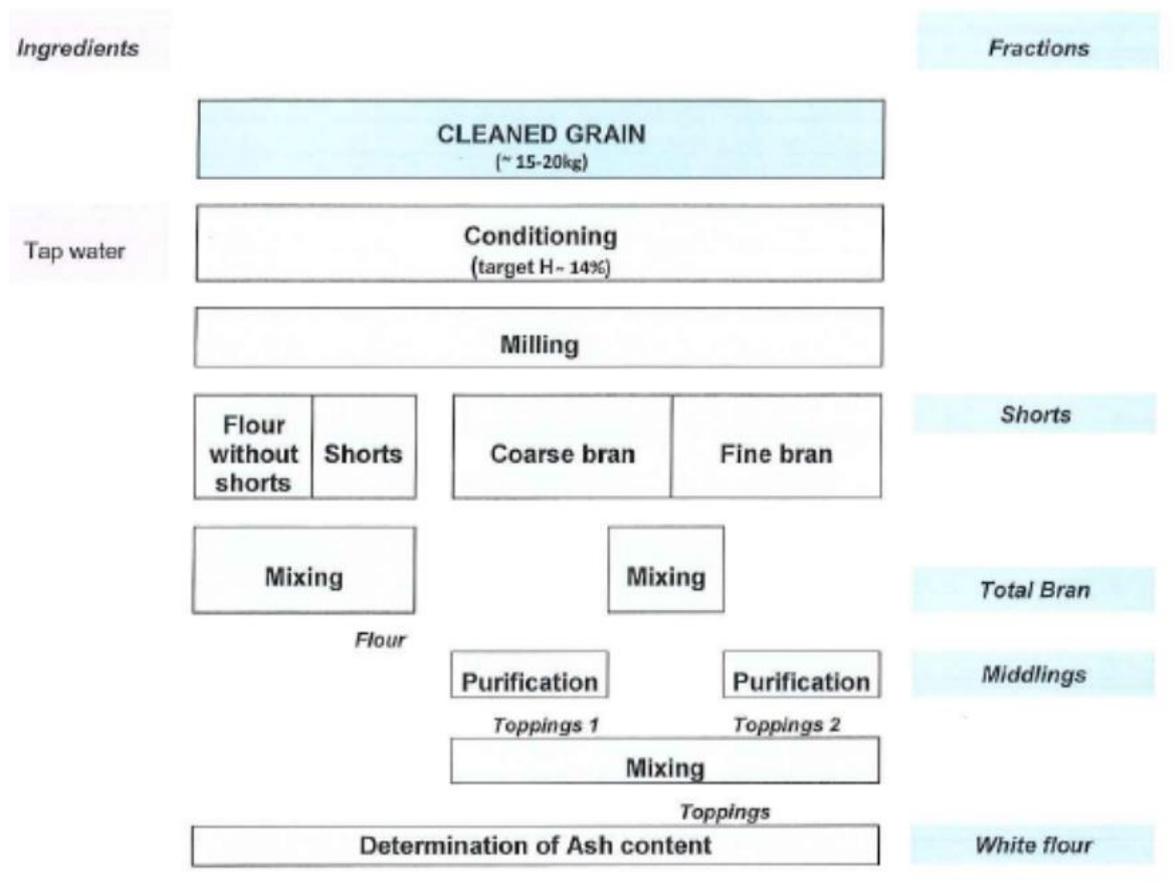


Figure A 3: Processing flowchart for wheat grain – white bread processing

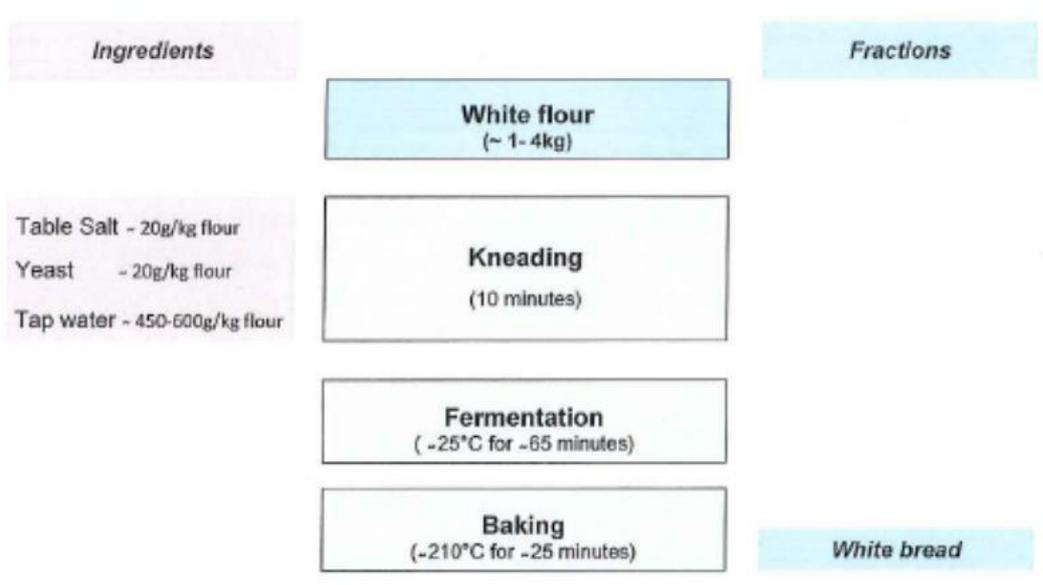


Figure A 4: Processing flowchart for wheat grain – wholemeal flour

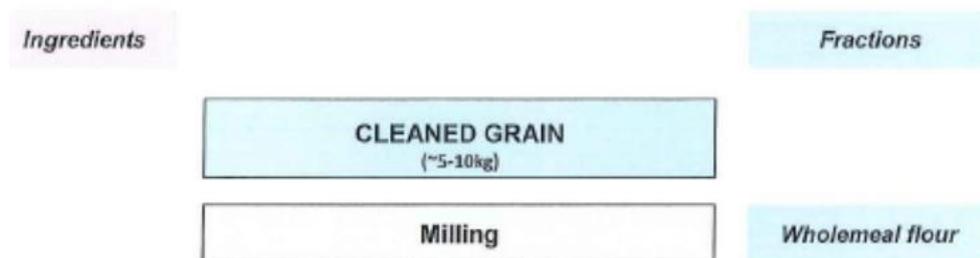


Figure A 5: Processing flowchart for wheat grain – wholemeal bread processing

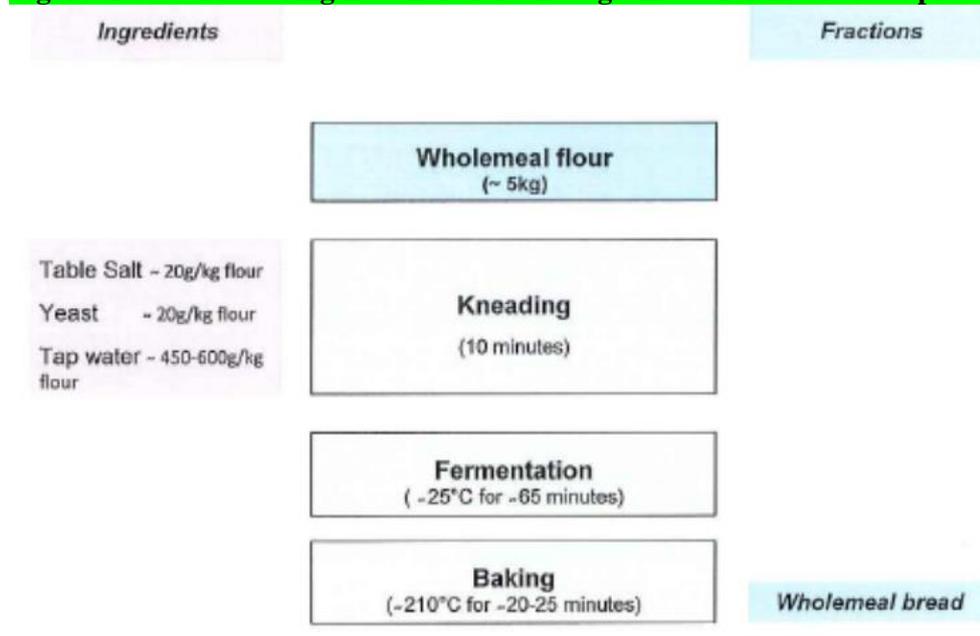
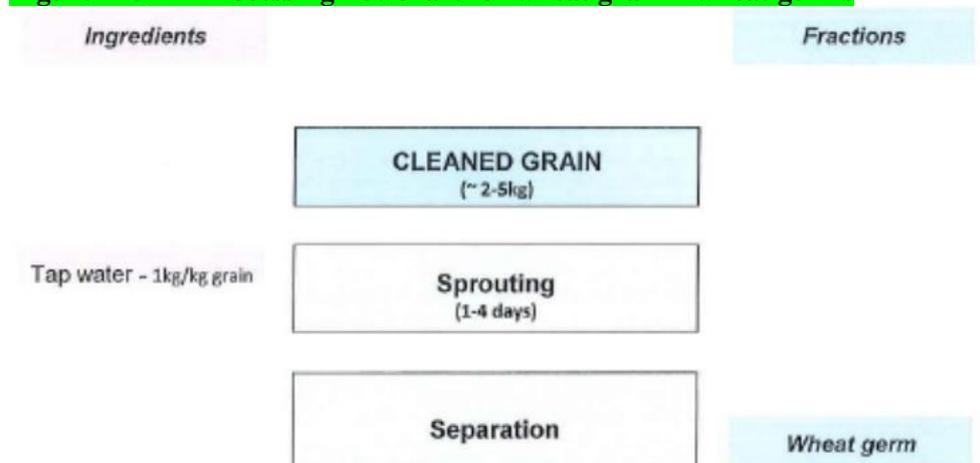


Figure A 6: Processing flowchart for wheat grain – wheat germs



The analytical method was initially validated for the determination of clopyralid in wheat grain, wheat straw, wheat germ, white flour, white bread and total bran according to SANCO/3029/99, rev. 4 within analytical phase S19-01810-L2 by fortification of control (untreated) test portions of the respective matrix and subsequent determination of the recoveries.

Five (5) fortifications of untreated control samples at the level of LOQ (0.01 mg/kg) and five (5) fortifications at the level of tenfold LOQ (0.1 mg/kg) were performed.

All processes and results relating to the initial analysis for clopyralid can be found in the analytical phase report for S19-01810-L2 (Please see Section B5).

As the objective of the study was to determine the residue levels of total clopyralid (clopyralid and clopyralid conjugate (X36538)), a further analytical phase was included within the study (S19-01810-L3, please see section B5), and it is the results from this phase that are the primary focus of the study.

The analytical method was validated for the determination of total clopyralid (clopyralid and clopyralid conjugate (X36538)) in white flour, total bran and white bread matrices according to SANCO/3029/99, rev. 4 within analytical phase S19-01810-L3 by fortification of control (untreated) test portions of the respective matrix and subsequent determination of the recoveries.

Five (5) fortifications of untreated control samples at the level of LOQ (0.044 mg/kg) and five (5) fortifications at the level of tenfold LOQ (0.44 mg/kg) were performed. The validation data was generated in sole analytical sets, i.e. separately from the analytical sets for residue sample analysis.

Due to their similarity, the validation results generated for white flour, total bran and white bread are considered to be representative respectively for wholemeal flour, for shorts, middling and for wholemeal bread.

Quantification was performed by use of LC-MS/MS detection.

The limit of quantification (LOQ) of the analytical method was 0.044 mg/kg for total clopyralid and each matrix with a limit of detection (LOD) set at 0.013 mg/kg (30 % of the LOQ).

No residues above 30 % of the LOQ were detected in the control (untreated) test portions used for recovery determinations.

The accuracy and precision of the method during sample analysis were considered to be acceptable since single recoveries were in the range of 60 - 120 % and the mean recoveries at each fortification level were in the range of 70 – 110 % with relative standard deviation(s) below 20 % for all combinations of matrices.

All samples for storage were fortified with clopyralid at the beginning of the experimental phase. Analysis was done directly at day 0, at 45 days and after Tmax interval.

Stability was demonstrated for clopyralid in homogenates of white flour and wholemeal flour for 840 days, total bran for 837 days, white bread for 640 days, wholemeal bread for 625 days and wheat germs for 638 days upon storage at $\leq -18^{\circ}\text{C}$.

Conclusion

Overall, the process went as expected, with no impact on the quality of specimens.

A 2.1.5.2.2 Study 2

Comments of zRMS:	Study is accepted
-------------------	-------------------

Reference: KCA 6.5.2-6.5.3/02

Report: Determination of Residues of Clopyralid after One Application of Major 300 SL (CHR/H/CPD 300SL) in Spring Wheat. One site in Northern France During 2020, White T., S20-04397, 2021

Guideline(s): Yes (OECD 508)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods

The objective of the study was to determine residue levels and behaviour of total clopyralid (clopyralid and clopyralid conjugate (X36538)) in the raw agricultural commodity spring wheat treated with Major 300SL.

One residue trial was conducted on spring wheat during 2020 in Northern France (S20-04397-01).

One application of Major 300SL (300 g/L, clopyralid) was applied at 160 g as/ha, diluted with water immediately prior to application to a spray volume of 200 L/ha.

Samples of spring wheat (grain and straw) from the untreated and treated plots were taken by hand 71 days (normal commercial harvest) after the single application.

Table A 11: Summary of the study 2 trials – Residues samples

1 Report No. Location (region)	2 Commodity/Variety (a)	3 Date of 1) Sowing or Planting 2) Flowering 3) Harvest (b)	4 Method of Treatment (c)	5 Application rate per treatment			6 Dates of treatment(s) or no. of treatment(s) and last date (d)	7 Growth stage at last treatment or date (e) BBCH	8 Portion analysed (a)	9 Residues (mg/kg) (*)	10 PHI (days) (f)	11 Remarks (g)
				g as/hL	Water (L/ha)	g as/ha						
S20-04397-01 45300, Sermaises, Loiret, France	Spring wheat/ EPPO code TRZAS / Lennox	1) 18 Mar 2020 2) 15 Jun-26 Jun 2020 3) 29 Jul 2020	Foliar using a boom sprayer	80	192	153.9	19 May 2020	32	Grain Straw	0.79 2.87	71	No residues >LOQ were found in any untreated samples

(a) According to EPPO codes
 (b) Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

(d) Year must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline); DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

(*) Limit of quantification = 0.044 mg/kg; limit of detection = 0.01 mg/kg; n/d = not detectable

Samples of spring wheat grain taken from trial S20-04397-01 were processed into wheat germs at the processing test site.

Table A 12: Summary of the study 1 trials - Processing samples

1 Report No. Location (region)	2 Commodity/Variety (a)	3 Date of 1) Sowing or Planting 2) Flowering 3) Harvest (b)	4 Method of Treatment (c)	5 Application rate per treatment			6 Dates of treatment(s) or no. of treatment(s) and last date (d)	7 Growth stage at last treatment or date (e) BBCH	8 Portion analysed (a)	9 Residues (mg/kg) (*)	10 PHI (days) (f)	11 Remarks (g)
				g as/hL	Water (L/ha)	g as/ha						
S20-04397-01 45300, Sermaises, Loiret, France	Spring wheat/ EPPO code TRZAS / Lennox	4) 18 Mar 2020 5) 15 Jun-26 Jun 2020 6) 29 Jul 2020	Foliar using a boom sprayer	80	192	153.9	19 May 2020	32	RAC Grain Wheat germs	0.52 – 0.67 0.08	71	No residues >LOQ were found in any untreated samples

(a) According to EPPO codes
 (b) Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type of equipment used must be indicated

(d) Year must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline); DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

(*) Limit of quantification = 0.044 mg/kg; limit of detection = 0.01 mg/kg; n/d = not detectable

The objective of the processing phase was to produce and sample processing fractions (specimens of end products (wheat germs and wheat before process) from wheat raw agricultural commodity.

5.2. Equipment and Material

The following list details the equipment and material used during the course of this study:

- Balances - Mettler Toledo
- Sample cleaner SLN3 - Pfeuffer

Results and discussions

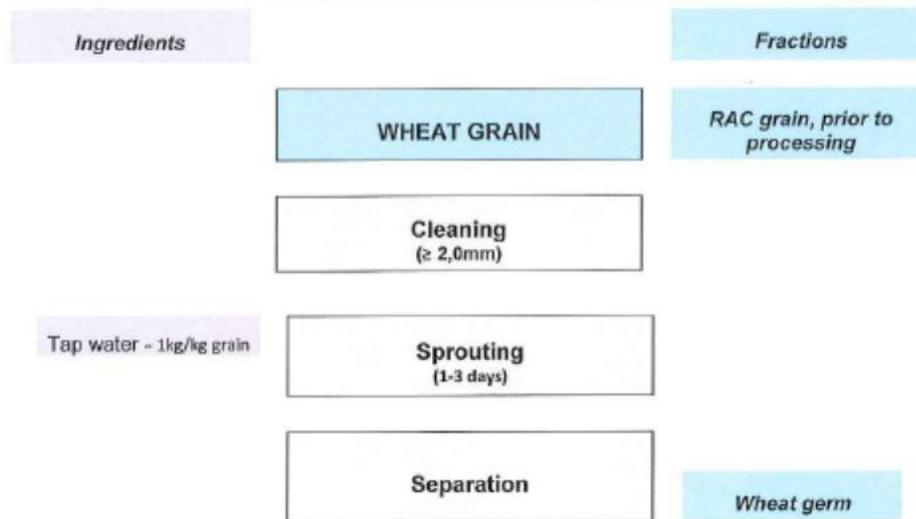
Table A 13: Residue data from grain processing study with clopyralid

RAC	Residues in RAC (unwashed sample, mg/kg)	PHI (days)	Processed commodity	Residue (mg/kg)	TF*	Comments/Reference
Grain	0.60**	80	wheat germs	0.06		

* transfer factor

** Mean of two values (0.52 mg/kg and 0.67 mg/kg)

Figure A 7: Processing flowchart for wheat grain – cleaning and germs



The analytical method was validated for the determination of total clopyralid in wheat straw, wheat grain and wheat germs according to SANCO/3029/99, rev. 4 within analytical phase S20-04397-L2 by fortification of control (untreated) test portions of the respective matrix and subsequent determination of the recoveries. Five (5) fortifications of untreated control samples at the level of LOQ (0.044 mg/kg) and five (5) fortifications at the level of tenfold LOQ (0.44 mg/kg) were performed.

Sample extraction and determination of residues was performed according to an analytical procedure that was validated within the analytical phase of this study (S20-04397-L2) for wheat grain, straw and germ matrices.

Quantification was performed by use of LC-MS/MS detection.

The limit of quantification (LOQ) of the analytical method was 0.044 mg/kg for total clopyralid and each matrix with a limit of detection (LOD) set at 0.01 mg/kg (30 % of the LOQ).

No residues above 30 % of the LOQ were detected in the control (untreated) test portions used for recovery determinations.

The accuracy and repeatability of the method during sample analysis were considered to be acceptable since single recoveries were in the range of 60 - 120 % and the mean recoveries at each fortification level

were in the range of 70 – 110 % with relative standard deviation(s) below 20 % for all combinations of matrices. For details please refers to Section B5.

Conclusion

Overall, the process went as expected, with no impact on the quality of specimens.

A 2.1.6 Magnitude of residues in representative succeeding crops

No new or additional studies have been submitted.

A 2.1.7 Other/Special Studies

No new or additional studies have been submitted.

Appendix 3 Pesticide Residue Intake Model (PRIMo)

A 3.1 TMDI calculations

Chronic risk assessment: JMPR methodology (IEDI/TMDI)											
No of diets exceeding the ADI : ---											Exposure resulting from
TMDI/NEDI/IEDI calculation (based on average food consumption)	Calculated exposure (% of ADI)		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)
	MS Diet										
	39%	NL toddler	57,98	9%	Maize/corn	5%	Wheat	4%	Apples	2%	9%
	22%	GEMS/Food G08	33,25	10%	Wheat	2%	Rice	2%	Maize/corn	0,2%	11%
	22%	NL child	32,59	8%	Sugar beet roots	5%	Wheat	2%	Apples	0,9%	11%
	20%	DE child	30,14	8%	Wheat	4%	Apples	1%	Oranges	0,7%	8%
	19%	DK child	28,71	7%	Rye	8%	Wheat	0,8%	Potatoes	0,5%	8%
	17%	RO general	26,06	7%	Wheat	3%	Head cabbages	1%	Maize/corn	0,5%	8%
	17%	GEMS/Food G15	25,49	8%	Wheat	2%	Head cabbages	1%	Potatoes	0,4%	8%
	17%	FR child 3 15 yr	25,07	8%	Wheat	2%	Sugar beet roots	1%	Oranges	0,9%	9%
	17%	GEMS/Food G10	24,95	5%	Wheat	2%	Rice	1%	Soyabeans	0,3%	5%
	17%	GEMS/Food G08	24,78	5%	Wheat	1%	Potatoes	1%	Barley	0,3%	8%
	16%	IE adult	24,39	3%	Wheat	2%	Sweet potatoes	2%	Linseeds	0,2%	3%
	15%	GEMS/Food G07	22,74	8%	Wheat	1%	Potatoes	0,8%	Barley	0,4%	8%
	15%	GEMS/Food G11	21,92	5%	Wheat	1%	Potatoes	1%	Soyabeans	0,4%	5%
	14%	IT toddler	21,02	9%	Wheat	2%	Other cereals	0,5%	Tomatoes		9%
	14%	UK toddler	20,86	5%	Wheat	2%	Sugar beet roots	1%	Potatoes	0,7%	7%
	14%	FR toddler 2 3 yr	20,40	4%	Wheat	2%	Sugar beet roots	1%	Apples	1%	8%
	13%	UK infant	19,98	3%	Wheat	1%	Maize/corn	1%	Milk: Cattle	1%	4%
	13%	SE general	18,99	4%	Wheat	1%	Potatoes	1%	Head cabbages	0,4%	4%
	12%	PT general	18,88	5%	Wheat	2%	Potatoes	1%	Rice		5%
	12%	DE women 14-50 yr	18,40	3%	Sugar beet roots	3%	Wheat	0,9%	Apples	0,5%	8%
	12%	ES child	18,05	8%	Wheat	0,7%	Oranges	0,8%	Rice	0,5%	8%
	12%	DE general	18,01	3%	Sugar beet roots	3%	Wheat	0,8%	Apples	0,5%	5%
	10%	NL general	16,71	3%	Wheat	2%	Sugar beet roots	0,8%	Potatoes	0,3%	5%
	9%	IT adult	13,99	8%	Wheat	0,9%	Other cereals	0,4%	Tomatoes		8%
	9%	FI 3 yr	13,84	2%	Wheat	2%	Potatoes	0,9%	Rye	0,0%	2%
	8%	ES adult	11,64	3%	Wheat	0,7%	Barley	0,4%	Oranges	0,2%	3%
	8%	FR adult	11,33	3%	Wheat	0,8%	Wine grapes	0,5%	Sugar beet roots	0,2%	4%
	7%	FI 6 yr	10,65	1%	Wheat	1%	Potatoes	0,8%	Rye	0,0%	1%
	7%	LT adult	10,54	1%	Rye	1%	Wheat	1%	Potatoes	0,2%	1%
	7%	UK vegetarian	10,35	3%	Wheat	0,5%	Rice	0,5%	Potatoes	0,1%	3%
	6%	FR infant	9,85	1%	Wheat	0,9%	Sugar beet roots	0,8%	Potatoes	0,6%	2%
	6%	FI adult	8,82	2%	Coffee beans	0,9%	Rye	0,4%	Wheat		0,4%
	6%	UK adult	8,65	2%	Wheat	0,5%	Rice	0,5%	Potatoes	0,1%	3%
	5%	DK adult	7,72	1%	Wheat	0,7%	Rye	0,4%	Potatoes	0,2%	1%
	4%	PL general	6,17	1%	Potatoes	0,7%	Head cabbages	0,7%	Apples		
	3%	IE child	4,46	2%	Wheat	0,4%	Rice	0,2%	Potatoes	0,1%	2%

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.
 DISCLAIMER: Dietary data from the UK were included in PRIMo when the UK was a member of the European Union.

A 3.2 IEDI calculations

TMDI does not exceed ADI therefore IEDI calculations are not required

A 3.3 IESTI calculations - Raw commodities

Show results for all crops															
Results for children No. of commodities for which ARID/ADI is exceeded (IESTI): ---				Results for adults No. of commodities for which ARID/ADI is exceeded (IESTI): ---				IESTI new Results for children No. of commodities for which ARID/ADI is exceeded (IESTI new): ---				IESTI new Results for adults No. of commodities for which ARID/ADI is exceeded (IESTI new): ---			
IESTI				IESTI				IESTI new				IESTI new			
Highest % of ARID/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARID/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARID/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)	Highest % of ARID/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
25%	Wheat	3 / 3	43	15%	Wheat	3 / 3	25	25%	Wheat	3 / 3	43	15%	Wheat	3 / 3	25
4%	Milk: Cattle	0,05 / 0,05	6,2	2%	Bovine: Kidney	1,5 / 1,5	3,2	4%	Milk: Cattle	0,05 / 0,05	6,2	2%	Bovine: Kidney	1,5 / 1,5	3,2
3%	Bovine: Kidney	1,5 / 1,5	5,6	1%	Milk: Cattle	0,05 / 0,05	1,9	3%	Bovine: Kidney	1,5 / 1,5	5,6	1%	Milk: Cattle	0,05 / 0,05	1,9
0,7%	Bovine: Liver	0,15 / 0,15	1,2	0,8%	Swine: Kidney	0,6 / 0,6	1,3	0,7%	Bovine: Liver	0,15 / 0,15	1,2	0,8%	Swine: Kidney	0,6 / 0,6	1,3
0,7%	Milk: Goat	0,05 / 0,05	1,2	0,5%	Milk: Goat	0,05 / 0,05	0,92	0,7%	Milk: Goat	0,05 / 0,05	1,2	0,5%	Milk: Goat	0,05 / 0,05	0,92
0,5%	Poultry: Muscle/meat	0,05 / 0,05	0,85	0,4%	Milk: Sheep	0,05 / 0,05	0,76	0,7%	Poultry: Muscle/meat	0,05 / 0,05	0,85	0,4%	Milk: Sheep	0,05 / 0,05	0,76
0,4%	Swine: Kidney	0,6 / 0,6	0,76	0,4%	Bovine: Liver	0,15 / 0,15	0,60	0,5%	Swine: Kidney	0,6 / 0,6	0,76	0,4%	Bovine: Liver	0,15 / 0,15	0,60
0,4%	Rapeseeds/canola seeds	0,5 / 0,5	0,69	0,3%	Poultry: Muscle	0,05 / 0,05	0,59	0,4%	Rapeseeds/canola seeds	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
0,3%	Sheep: Muscle/meat	0,08 / 0,08	0,43	0,2%	Other farmed animals:	0,05 / 0,05	0,28	0,3%	Sheep: Muscle/meat	0,08 / 0,08	0,43	0,2%	Other farmed animals:	0,05 / 0,05	0,28
0,2%	Bovine: Edible offals (other	0,05 / 0,05	0,36	0,2%	Rapeseeds/canola seeds	0,5 / 0,5	0,26	0,3%	Bovine: Edible offals (other	0,05 / 0,05	0,36	0,2%	Rapeseeds/canola seeds	0,5 / 0,5	0,26
0,2%	Other farmed animals:	0,05 / 0,05	0,35	0,1%	Swine: Muscle/meat	0,05 / 0,05	0,24	0,2%	Other farmed animals:	0,05 / 0,05	0,35	0,1%	Swine: Muscle/meat	0,05 / 0,05	0,24
0,2%	Bovine: Fat tissue	0,15 / 0,15	0,31	0,1%	Equine: Muscle/meat	0,05 / 0,05	0,24	0,2%	Bovine: Fat tissue	0,15 / 0,15	0,31	0,1%	Equine: Muscle/meat	0,05 / 0,05	0,24

Expand/collapse list

Appendix 4 Additional information provided by the applicant

Additional information are not provided.