REGISTRATION REPORT

Part B

Section 5

Analytical Methods

Detailed summary of the risk assessment

Product code: 19202

Product names: **KINVARA**

Chemical active substances:

MCPA, 233 g/L
Fluroxypyr, 50 g/L
Clopyralid, 28 g/L

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

(Renewal of Authorization)

Applicant: XXXX

Submission date: 31/01/2024

Evaluation date: October 2024

MS Finalisation date: March 2025

Version history

When	What
January 2024	The dRR submission
October 2024	Initial RR
March 2025	Version modified by zRMS PL to take into accout cMSs and the applicant

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5 Analytical methods

5.1 Conclusion and summary of assessment

Sufficiently sensitive and selective analytical methods are ~~not~~ available for the active substance(s) and relevant impurities in the plant protection product.
Noticed data gaps are: none

- ~~data gap 1~~
- ~~data gap 2~~
- ~~data gap 3~~

Sufficiently sensitive and selective analytical methods are available for all analytes included in the residue definitions in the context of the authorisation of Kinvara. The applicant's dRR was not rewritten. In the resulting zRMS' RR all comments /corrections/ add-ons were placed on the grey background. In the context of the present authorisation the noticed data gaps are none.

Commodity/crop	Supported/ Not supported
Cereals	Supported

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

5.2.1 Analysis of the plant protection product (KCP 5.1.1)

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

An overview on the acceptable methods and possible data gaps for analysis of Clopyralid, Fluroxypyr and MCPA in Kinvara is provided as follows:

Comments of zRMS:	Accepted. ~~There is the Horrat value missing for precision. However, the lack is not crucial for the method acceptance. This should be required when reassessing the authorisation of PPP~~ This Article 43 assessment results from the renewal for clopyralid. For this reason, data for other substances are not taken into account.

Reference:	KCP 5.1.1
Report	Title: OA02413 – Validation of the analytical methods used to determine the active ingredients in "Kinvara", a micro-emulsion (ME) formulation, containing MCPA, Clopyralid and Fluroxypyr. Author: S. Hubbard, Oxford Analytical Ltd (2014)
Guideline(s):	Method validated in accordance with the guidelines set out in SANCO/3030/99 Rev. 4
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

The analytical method is based on Reverse Phase HPLC with UV-Vis detection. Quantification of each active ingredient is achieved by calibration curve using certified reference standards of active ingredients. Samples and standards are dissolved in acetonitrile and analysed under the HPLC conditions outlined in the report.

Validation - Results and discussions

Table 5.2-1: Methods suitable for the determination of active substances Clopyralid, Fluroxypyr and MCPA in plant protection product Kinvara

	Clopyralid	**Fluroxypyr (meptyl)**	**MCPA**
Author(s), year	S. Hubbard, Oxford Analytical Ltd (2014)		
Principle of method	HPLC-UV		
Linearity (linear between mg/mL / % range of the declared content) n=6 **(correlation coefficient, expressed as r)**	y=19089x-2.77 0.0004 mg/mL – 0.1158 mg/mL, equivalent to 1.6 – 465% of the declared content (r = 1.0000)	0.0011 mg/mL – 0.2982 mg/mL, equivalent to 1.7 – 465% of the declared content (r = 0.9997)	0.0038 mg/mL – 1.0045 mg/mL, equivalent to 1.8 – 484% of the declared content (r = 0.9991)
Precision – Repeatability Mean n = 6 (%RSD)	26.9 g/L (1.10%) The RSDr is 2.3, the Horrat value is 0.48	77.5 g/L (1.45%)	226.8 g/L (0.59%)
Accuracy n = **5** **(% Recovery)**	Range: 100.43 – 103.05% Mean: 102.0% %RSD: 0.95%	Range: 100.1 – 103.1% Mean: 101.4% %RSD: 1.16%	Range: 101.21 – 102.24% Mean: 101.8% %RSD: 0.41%
Interference/ Specificity	**Interference:** Mixed reference standard, solvent blank and Kinvara chromatograms were compared. No interferences were noted at the retention times of the active ingredients. **Specificity:** A standard solution containing MCPA, Clopyralid and Fluroxypyr-meptyl reference standard and a sample of formulation were analysed by HPLC-MS/MS. For all active substances, retention time and molecular ion (m/z) matched. Furthermore, the fragmentation patterns of the		

	Clopyralid	Fluroxypyr (meptyl)	MCPA
	molecular ions were consistent between reference and sample.		
Comment	N/A		

Conclusion

The analytical method has been validated according to EU Guidance SANCO/3030/99 rev.4 and is considered suitable for the determination of MCPA, Clopyralid and Fluroxypyr-meptyl in Kinvara formulation in accordance with SANCO/3030/99 rev.5.

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

N-methyl-2-pyrrolidone (NMP) is deemed to be a relevant impurity in Fluroxypyr-meptyl technical, with a maximum allowable content of 3 g/kg. The dRR for Kinvara was originally submitted in 2015, at which point no relevant impurities were specified for Fluroxypyr-meptyl technical in the EU. Consequently, NMP was not considered as a relevant impurity in Kinvara when the original storage stability study was performed and dossiers were prepared. For this reason the content of NMP in Kinvara was not analysed. In order to address this new requirement in this dRR report XXXX has consulted with each source of Fluroxypyr-meptyl technical and has determined (in writing from each source - please refer to Supplier Statements) that NMP is not and has never been used in the manufacturing process for Fluroxypyr-meptyl technical employed by either of the two sources. For this reason it can be concluded that NMP will not be present in Kinvara.

Additional assurance of compliance with the maximum level of NMP in Kinvara (calculated in table 1.4-2 as approx. 0.009 g/L NMP/L of Kinvara) is provided by analysis commissioned by XXXX on 10 batches of Fluroxypyr-meptyl technical from each of the two sources of this material (please refer to *KCP 1.4.1-01 DNA 4069 & KCP 1.4.1-02 DNA4070).* Details of the analytical method validation for the analysis of NMP in Fluroxypyr-meptyl are presented below.

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

Comments of zRMS:	Clopyralid has no relevant impurities agreed at the EU level. So, the data are not considered for the purpose of the evaluation under art. 43 (renewal of clopyralid)

Reference:	KCP 5.1.1
Report	Validation of the method of a specified impurity in Fluroxypyr-Meptyl technical material, in compliance with Good Laboratory Practice D. Pomeroy, 2017, DNA4071
Guideline(s):	Method validated in accordance with the guidelines set out in SANCO/3030/99 Rev. 4
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

The analytical method is based on Reverse Phase HPLC with UV-Vis detection. Quantification of NMP is

achieved by calibration curve using certified reference standards.

Samples and standards are dissolved in methanol and analysed under the HPLC conditions outlined in the study report.

Validation - Results and discussions

Table 5.2-2: Methods suitable for the determination of the relevant impurity (NMP) in Fluroxypyr-meptyl technical

	N-methyl-2-pyrrolidone **max. content in Fluroxypyr-meptyl technical = 3 g/kg**
Author(s), year	D. Pomeroy, 2017
Principle of method	HPLC-UV
Linearity **(linear between** **mg/L)** **(correlation coefficient, expressed as r)**	0.25 – 100 mg/L (r = 1.0000)
Precision – Repeatability Mean **n = 6** **(%RSD)**	0.105%
Accuracy **n = 6** **(% Recovery)**	Range: 99.09 – 99.35% Mean: 99.21% %RSD: 0.105%
Interference/ Specificity	**Interference:** NMP eluted at 4.79 minutes, and there were no other peaks present at the same elution time. **Specificity:** The UV and MS spectra for NMP confirms the identity.
LOQ	0.125 g/kg

Conclusion

The analytical method has been validated according to EU Guidance SANCO/3030/99 rev.4 and is considered suitable for the determination of NMP in Fluroxypyr-meptyl technical, and by extension the NMP content in Kinvara since Fluroxypyr-meptyl technical is the only potential source of NMP in Kinvara in accordance with SANCO/3030/99 rev.5.

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

Kinvara does not contain any relevant formulants.

5.2.1.4 Applicability of existing CIPAC methods (KCP 5.1.1)

CIPAC methods are not available for the detection of all three active ingredients in a single method. Therefore a bespoke method was developed and validated that is capable of detecting all three active ingredients in Kinvara in a single method.

5.2.2 Methods for the determination of residues (KCP 5.1.2)

An overview on the acceptable methods and possible data gaps for analysis of residues of MCPA, fluroxypyr and clopyralid for the generation of pre-authorization data is given in the following tables.

XXXX was considered to have a complete Annex II data package in support of the inclusion of fluroxypyr on to Annex I. As part of the fluroxypyr product reauthorisations in the UK, CRD concluded the "*methods of analysis for monitoring in food of plant origin*, food of animal origin, environmental samples including soil, water and air, and body tissues and fluids are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed.*
** A confirmatory method, an ILV and further validation of the hydrolysis step were required. However these data are only required at re-renewal so no further data are required at present*."

These EU agreed validated methods are detailed in table 5.2-3 below.

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

Component of residue definition: *1) fluroxypyr and its ester fluroxypyr 1-meptylheptylester expressed as fluroxypyr* *2) the parent molecule fluroxypyr* *3) fluroxypyr 1-MHE, fluroxypyr acid & the metabolites pyridinol & methoxypyridine* *4) Fluroxypyr 1-MHE & fluroxypyr acid*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Plant matrices [**1**]: Cereals & dry commodities (wheat barley oats) (Residues)	Primary	0.05 mg/kg[1] 0.10 mg/kg[2]	GC-MSD	Olberding, E.L & Ng, C.A, 1996 / Method GRM 96.02 / EU agreed
	ILV	0.01mg/kg[3] 0.05 mg/kg[4] 0.25 mg/kg[5]	GC-MSD	McKeller, R.L, MacGregory, A. & Markley, B.J, 1996 / Method GRM 96.02 / EU agreed
Foodstuffs of plant origin [**1**]: Cereal matrices (Residues)	Primary	0.01 mg/kg (cereal matrices)	HPLC-MS/MS	Witte, A., 2015, KCP 5.1.2/42 and KCP 5.1.2/43 (Appendix 2)
Plant matrices [**1**]: Maize (Residues)	Primary	0.10 mg/kg	GC-ECD	Maycock, C. & Teasdale, R,.1992 / Method GRM ERC 89.5 / EU agreed
	ILV	0.05 mg/kg[6] 0.2 mg/kg[7] 0.05 mg/kg[8]	GC-ECD	Teasdale, R, 1995 / Method ERC 89.5 / EU agreed
Animal products [**2**]: Bovine tissue & milk (Residues)	Primary	0.01 mg/kg	GC-MSD	Olberding, E.L. & Huskin, M.A, 1996 / Mehod GRM 96.03 / EU agreed
	ILV	0.01 mg/kg	GC-MSD	Reed, D.E & Bottom, S.N, 2003 / Method GRM 96.03 / EU agreed
Animal products [**2**]: poultry tissues & eggs (residues)	Primary	0.01 mg/kg	LC-MS/MS	Shackelford, D.D, 2009 / Method GRM 08.03 / EU agreed
	ILV	0.01 mg/kg	LC-MS/MS	Sencuic, M & Class. T, 2009 / GRM 08.03 / EU agreed

Component of residue definition: *1) fluroxypyr and its ester fluroxypyr 1-meptylheptylester expressed as fluroxypyr* *2) the parent molecule fluroxypyr* *3) fluroxypyr 1-MHE, fluroxypyr acid & the metabolites pyridinol & methoxypyridine* *4) Fluroxypyr 1-MHE & fluroxypyr acid*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Soil [**3**] (Environmental fate)	Primary & Confirmatory	0.01 mg/kg	GC-MSD	Moore, M, 1996 / Method GRM 93.03 / EU agreed
	Primary & Confirmatory	0.01 mg/kg	GC-MSD	Shackelford, D.D, 1999 / Method GRM 98.04 / EU agreed
	Primary & Confirmatory	0.01 mg/kg	GC-MSD	Van Dyke, M.E., 1991 / Method ARC 91.10 / EU agreed
Water, drinking water, surface water [**3**] (human health)	Primary & Confirmatory (surface water)[9]	5 µg/L	GC-MS	Shackelford, D.D, 2000 / Method GRM 00.21 / EU agreed
Air [**4**] (Exposure)	Primary	≤24 µg/m3	LC-MS/MS	Backer, R, 2009 / Method B1644 G / EU agreed
Body fluids (Exposure)	There is no requirement for a method of analysis for the determination of fluroxypyr or its metabolites in body tissue. Fluroxypyr and its metabolites are not considered to be of toxicological concern.			

1 LOQ for fluroxypyr in forage and straw
2 LOQ for hay due to the high level of fluroxypyr residues found in unfortified control samples
3 LOQ for wheat, barley and oat grain
4 LOQ for fluroxypyr in straw and hay
5 LOQ for fluroxypyr in forage due to the high level of fluroxypyr residues found in unfortified control samples
6 LOQ for fluroxypyr in maize whole plant
7 LOQ for fluroxypyr in maize straw
8 LOQ for fluroxypyr in maize cob
9 The RAR for fluroxypyr needs an LOQ < 0.1 µg/L in drinking water, however UK CRD confirmed during the reauthorization of fluroxypyr products in the UK "*methods of analysis for monitoring in food of plant origin*, food of animal origin, environmental samples including soil, water and air, and body tissues and fluids are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed"*

zRMS: the applicant's data is acceptable.

Table 5.2-4: Validated methods for the generation of pre-authorization data - MCPA

Component of residue definition: **Food of plant origin:** *MCPA and MCPB (MCPA, MCPB including their salts, esters and conjugates expressed as MCPA)*(Reg. (EU) No 491/2014) **Food of animal origin, soil, water (surface, drinking, ground), air:** *MCPA, MCPB and MCPA thioethyl expressed as MCPA.* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Food/feed of plant origin	Primary	0.07 mg/kg	GC-MSD	Flynn S G, 1979/EU agreed
		0.050 mg/kg	GC-MSD	Keller, W, 1980/EU Agreed
	Primary	0.01 mg/kg (cereal matrices)	HPLC-MS/MS	Witte, A., 2015, KCP 5.1.2/42 and KCP 5.1.2/43 (Appendix 2)

Component of residue definition:
Food of plant origin: *MCPA and MCPB (MCPA, MCPB including their salts, esters and conjugates expressed as MCPA)*(Reg. (EU) No 491/2014)
Food of animal origin, soil, water (surface, drinking, ground), air: *MCPA, MCPB and MCPA thioethyl expressed as MCPA.* (Reg. (EU) No 491/2014)

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
	Primary	0.01 mg/kg (grassland)	HPLC-MS/MS	Diebold, J., 2018, KCP 5.1.2/44 (Appendix 2)
Food/feed of animal origin	Primary	0.050 mg/kg	GC-MSD	Keller S G, 1979/EU agreed
		0.050 mg/kg	GC-MSD	Keller, W, 1980/EU Agreed
Soil	Primary	50 mg/kg	GC-MSD	Sattar MA and J Paasivirta, 1979/ EU agreed
Water, drinking, river, ground	Primary	0.1 µg/L	GC-MS	HMSO Publication, "Methods for the examination of waters and associated materials"/1997
Air	Primary	0.6 µg/m3	HPLC-MS	Reichert N, 1994/ EU agreed
		0.24 µg/m3	HPLC/UV	Werrer Zangmeister, 1995/EU agreed
Body fluids (Exposure)	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.			

zRMS: the applicant's data is acceptable.

Table 5.2-5: Validated methods for the generation of pre-authorization data - Clopyralid

Component of residue definition:
Food of plant origin: *Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of "polar clopyralid"* (EFSA, 2018)
Food of animal origin, soil, water (surface, drinking, ground), air: *Clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid)* (EFSA, 2018)
Soil: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Groundwater: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Surface water: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Sediment: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Air: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
Foodstuffs of plant origin (Residues)	Primary	0.01 mg/kg (cereal matrices)	HPLC-MS/MS	Witte, A., 2015, KCP 5.1.2/42 and KCP 5.1.2/43 (Appendix 2)
Foodstuff of animal origin (Residues)	No specific methods for the support of residues (foodstuffs of animal origin) have been developed for the authorisation of Kinvara.			

Component of residue definition:
Food of plant origin: *Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of "polar clopyralid"* (EFSA, 2018)

Food of animal origin, soil, water (surface, drinking, ground), air: *Clopyralid common moiety (sum of clopyralid, its salts and glycine conjugates expressed as clopyralid)* (EFSA, 2018)

Soil: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Groundwater: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Surface water: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)
Sediment: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)

Air: *Clopyralid* (Vol 1 – Level 2, 2017 RAR)

Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
Environmental Fate	No specific methods for the support of environmental fate have been developed for the authorisation of Kinvara.			
Efficacy	No specific methods for the support of efficacy have been developed for the authorisation of Kinvara.			
Toxicology	No specific methods for the support of toxicology have been developed for the authorisation of Kinvara.			
Exposure	No specific methods for the support of exposure have been developed for the authorisation of Kinvara.			
Ecotoxicology	No specific methods for the support of ecotoxicology have been developed for the authorisation of Kinvara.			
Plant protection product (Properties)	Refer to Section 5.2.1.			

zRMS: the applicant's data is acceptable.

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

Table 5.3-1: Validated methods for the generation of post-authorization data – Fluroxypyr

Component of residue definition: **Food of plant origin:** *Fluroxypyr, its esters, salts and its conjugates expressed as fluroxypyr* **Food of animal origin, soil, water (surface, drinking, ground), air:** *Fluroxypyr & its salts expressed as fluroxypyr*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Food/feed of plant origin	Primary	0.05 mg/kg[1] 0.10 mg/kg[2]	GC-MSD	Olberding, E.L & Ng, C.A, 1996 / Method GRM 96.02 / EU agreed
Food/feed of animal origin	Primary	0.01 mg/kg	GC-MSD	Olberding, E.L. & Huskin, M.A, 1996 / Method GRM 96.03 / EU agreed
	Primary	0.01 mg/kg	LC-MS/MS	Shackelford, D.D, 2009 / Method GRM 08.03 / EU agreed
Soil	Primary	0.01 mg/kg	GC-MSD	Shackelford, D.D, 1999 / Method GRM 98.04 / EU agreed
Water, drinking water, surface water	Primary (surface water)[3]	5 µg/L	GC-MS	Shackelford, D.D, 2000 / Method GRM 00.21 / EU agreed
Air	Primary	≤24 µg/m^3	LC-MS/MS	Backer, R, 2009 / Method B1644 G / EU agreed
Body fluids (Exposure)	There is no requirement for a method of analysis for the determination of fluroxypyr or its metabolites in body tissue. Fluroxypyr and its metabolites are not considered to be of toxicological concern.			

[1] LOQ for fluroxypyr in forage and straw
[2] LOQ for hay due to the high level of fluroxypyr residues found in unfortified control samples
[3] The RAR for fluroxypyr needs an LOQ < 0.1 µg/L in drinking water, however UK CRD confirmed during the reauthorization of fluroxypyr products in the UK "*methods of analysis for monitoring in food of plant origin*, food of animal origin, environmental samples including soil, water and air, and body tissues and fluids are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed*

zRMS: the applicant's data is acceptable.

Table 5.3-2: Validated methods for the generation of post-authorization data – MCPA

Component of residue definition: **Food of plant origin:** *MCPA and MCPB (MCPA, MCPB including their salts, esters and conjugates expressed as MCPA)* (Reg. (EU) No 491/2014) **Food of animal origin, soil, water (surface, drinking, ground), air**: *MCPA, MCPB and MCPA thioethyl expressed as MCPA* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Food/feed of plant origin	Primary	0.050 mg/kg	GC-MSD	Wasser C., 2000/Method ATM 592/ EU agreed
Food/feed of animal origin	Primary	0.050 mg/kg	GC-MSD	Pfarl C, 1994/ EU agreed

Component of residue definition: **Food of plant origin:** *MCPA and MCPB (MCPA, MCPB including their salts, esters and conjugates expressed as MCPA)* (Reg. (EU) No 491/2014) **Food of animal origin, soil, water (surface, drinking, ground), air**: *MCPA, MCPB and MCPA thioethyl expressed as MCPA* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Food/feed of plant origin	Primary	0.050 mg/kg	GC-MSD	Pfarl C, 1994/ EU agreed
Food/feed of animal origin	Primary	0.050 mg/kg	GC-MSD	Wasser C., 2000/Method ATM 592/ EU agreed
Soil	Primary	50 mg/kg	GC-MSD	Sattar MA and J Paasivirta, 1979/ EU agreed
Water, drinking, river, ground	Primary	0.1 µg/L	GC-MS	HMSO Publication, "Methods for the examination of waters and associated materials"/1997
Air	Primary	0.6 µg/m^3	HPLC-MS	Reichert N, 1994/ EU agreed
		0.24 µg/m^3	HPLC/UV	Werrer Zangmeister, 1995/EU agreed
Body fluids (Exposure)	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.			

zRMS: the applicant's data is acceptable.

Table 5.3-3: Validated methods for the generation of post-authorization data – Clopyralid

Component of residue definition: **Food of plant and animal origin:** *Clopyralid* (Reg. (EU) No 322/2012) **Soil, water (surface, drinking, ground), air, body fluids and tissues**: *Clopyralid* (EFSA, 2018)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Food/feed of plant origin	Primary	0.01 mg/kg (all matrices)	LC-MS/MS	Vogl, E., 2012, CA 4.2 (a)/1 (EFSA, 2018) EU agreed
	ILV	0.01 mg/kg (high water, high acid)	LC-MS/MS	Austin, R., 2012, CA 4.2 (a)/2 (EFSA, 2018) EU agreed
	Primary	0.01 mg/kg (high acid, high water)	LC-MS/MS (QuEChERS)	Author names redacted, 2013, CA 4.2 (a)/5 (EFSA, 2018) EU agreed
	ILV	0.01 mg/kg (high acid, high water)	LC-MS/MS (QuEChERS)	Author names redacted, 2014, CA 4.2 (a)/6 (EFSA, 2018) EU agreed
Food/feed of animal origin	Primary	0.01 mg/kg (all matrices)	LC-MS/MS	Author names redacted, 2012, CA 4.2 (a)/3 (EFSA, 2018) EU agreed
	ILV	0.01 mg/kg (milk, muscle,	LC-MS/MS	Author names redacted, 2012, CA 4.2 (a)/4 (EFSA, 2018)

Component of residue definition: **Food of plant and animal origin:** *Clopyralid* (Reg. (EU) No 322/2012) **Soil, water (surface, drinking, ground), air, body fluids and tissues**: *Clopyralid* (EFSA, 2018)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
		liver, egg)		EU agreed
	Primary	0.01 mg/kg (fat, milk)	LC-MS/MS (QuEChERS)	Author names redacted, 2013, CA 4.2 (a)/5 (EFSA, 2018) EU agreed
	ILV	0.01 mg/kg (fat)	LC-MS/MS (QuEChERS)	Author names redacted, 2014, CA 4.2 (a)/6 (EFSA, 2018) EU agreed
Soil	Primary	0.50 µg/kg	LC-MS/MS	Vincent, T. P., 2013, CA 4.2 (b)/1 (EFSA, 2018) EU agreed
Water, drinking, ground, surface	Primary	0.05 µg/L	LC-MS/MS	Shaffer, S., 2012, CA 4.2 (b)/3 (EFSA, 2018) EU agreed
	ILV	0.05 µg/L	LC-MS/MS	Austin, R., Turner, R., 2013, CA 4.2 (b)/4 (EFSA, 2018) EU agreed
Air	Primary	4.5 µg/m^3	LC-MS/MS	Bacher, R. 2012, CA 4.2 (c)/1 (EFSA, 2018) EU agreed
Body fluids (Exposure)	Primary	0.05 mg/L (blood, urine)	LC-MS/MS	Author names redacted, 2014, CA 4.2 (d)/1 (EFSA, 2018) EU agreed

zRMS: the applicant's data is acceptable.

5.3.1 Analysis of the plant protection product (KCP 5.2)

See section 5.2.1 above. No further information on analysis of the plant protection product is needed.

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

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

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

Table 5.3-2: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high protein/high starch content (dry	fluroxypyr and its ester fluroxypyr 1-	0.05 mg/kg[1] (LOQ) 0.10 mg/kg[2] (LOQ)	Olberding, E.L & Ng, C.A, 1996 / Method GRM 96.02 /

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
commodities)	meptylheptylester expressed as fluroxypyr		EU agreed
Muscle	fluroxypyr & its salts expressed as fluroxypyr	0.01 mg/kg	Olberding, E.L. & Huskin, M.A, 1996 / Method GRM 96.03 / EU agreed & Shackelford, D.D, 2009 / Method GRM 08.03 / EU agreed
Milk			
Eggs			
Fat			
Liver, kidney			
Soil (Ecotoxicology)	fluroxypyr & its salts expressed as fluroxypyr	0.01 mg/kg	Shackelford, D.D, 1999 / Method GRM 98.04 / EU agreed
Drinking water (Human toxicology)	fluroxypyr & its salts expressed as fluroxypyr	0.1 µg/L	general limit for drinking water
Surface water (Ecotoxicology)	fluroxypyr & its salts expressed as fluroxypyr	5 µg/L	Shackelford, D.D, 2000 / Method GRM 00.21 / EU agreed
Air	fluroxypyr & its salts expressed as fluroxypyr	≤ 24 µg/m^3	Backer, R, 2009 / Method B1644 G / EU agreed
Tissue (meat or liver)	N/A	Not required	not classified as T / T+
Body fluids		Not required	not classified as T / T+

[1] LOQ for fluroxypyr in forage and straw
[2] LOQ for hay due to the high level of fluroxypyr residues found in unfortified control samples

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

An overview on the acceptable methods and possible data gaps for analysis of fluroxypyr in plant matrices is given in the following tables.

XXXX was considered to have a complete Annex II data package in support of the inclusion of fluroxypyr on to Annex I. As part of the fluroxypyr product reauthorisations in the UK, CRD concluded the "*methods of analysis for monitoring in food of plant origin*…are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed.*
** A confirmatory method, an ILV and further validation of the hydrolysis step were required. However these data are only required at re-renewal so no further data are required at present*".

Table 5.3-3: Validated methods for food and feed of plant origin (required for all matrix types, "difficult" matrix only when indicated by intended GAP)

Component of residue definition: *fluroxypyr and its ester fluroxypyr 1-meptylheptylester expressed as fluroxypyr*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High protein/high starch content	Primary	0.05 mg/kg[1] 0.10 mg/kg[2]	GC-MSD	Olberding, E.L & Ng, C.A, 1996 / Method GRM 96.02 / EU agreed
	ILV	0.01 mg/kg[3]	GC-MSD	McKeller, R.L, MacGregory, A.

Component of residue definition: *fluroxypyr and its ester fluroxypyr 1-meptylheptylester expressed as fluroxypyr*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
(dry)		0.05 mg/kg[4] 0.25 mg/kg[5]		& Markley, B.J, 1996 / Method GRM 96.02 / EU agreed

[1] LOQ for fluroxypyr in forage and straw
[2] LOQ for hay due to the high level of fluroxypyr residues found in unfortified control samples
[3] LOQ for wheat, barley and oat grain
[4] LOQ for fluroxypyr in straw and hay
[5] LOQ for fluroxypyr in forage due to the high level of fluroxypyr residues found in unfortified control samples

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

Table 5.3-4: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	-
Not required, because:	SANTE 2017/10632 Rev. 5 of 22 November 2017 (i.e. the Technical Guideline on the Evaluation of Extraction Efficiency of Residue Analytical Methods), with application from May 2023, states that: *"**For renewal of product authorisations or for new product authorisations** or extension of uses **for which no change of the MRL is needed**, the data requirements used for the latest renewal or approval should be considered. This means that no additional proof of extraction efficiency is required if it had not been required in the renewal of approval/approval procedure itself. Extraction efficiency should be addressed if for a product authorization a different analytical methodology (in methods for risk assessment and/or monitoring) is used, compared to that of the approval/renewal procedure of the active substance."* The guidance document did not apply when the data for the latest renewal of approval of fluroxypyr were submitted. As such, no additional data are required to address extraction efficiency to support authorisation of KINVARA.

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

An overview on the acceptable methods and possible data gaps for analysis of fluroxypyr in animal matrices is given in the following tables.

XXXX was considered to have a complete Annex II data package in support of the inclusion of fluroxypyr on to Annex I. As part of the fluroxypyr product reauthorisations in the UK, CRD concluded the "*methods of analysis for monitoring in… food of animal origin…are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed."*

Table 5.3-5: Validated methods for food and feed of animal origin (if appropriate)

Component of residue definition: *fluroxypyr & its salts expressed as fluroxypyr*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Milk	Primary	0.01 mg/kg	GC-MSD	Olberding, E.L. & Huskin, M.A, 1996 / Method GRM 96.03 / EU agreed
	ILV	0.01 mg/kg	GC-MSD	Reed, D.E & Bottom, S.N, 2003 / Method GRM 96.03 / EU agreed
Eggs	Primary	0.01 mg/kg	LC-MS/MS	Shackelford, D.D, 2009 / Method GRM 08.03 / EU agreed
	ILV	0.01 mg/kg	LC-MS/MS	Sencuic, M & Class. T, 2009 / Method GRM 08.03 / EU agreed
Muscle	Primary	0.01 mg/kg	GC-MSD	Olberding, E.L. & Huskin, M.A, 1996 / Method GRM 96.03 / EU agreed
	ILV	0.01 mg/kg	GC-MSD	Reed, D.E & Bottom, S.N, 2003 / Method GRM 96.03 / EU agreed
Fat	Primary	0.01 mg/kg	GC-MSD	Olberding, E.L. & Huskin, M.A, 1996 / Method GRM 96.03 / EU agreed
	ILV	0.01 mg/kg	GC-MSD	Reed, D.E & Bottom, S.N, 2003 / Method GRM 96.03 / EU agreed
Kidney, liver	Primary	0.01 mg/kg	GC-MSD	Olberding, E.L. & Huskin, M.A, 1996 / Method GRM 96.03 / EU agreed
	ILV	0.01 mg/kg	GC-MSD	Reed, D.E & Bottom, S.N, 2003 / Method GRM 96.03 / EU agreed

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

Table 5.3-6: Statement on extraction efficiency

	Method for products of animal origin
Required, available from:	-
Not required, because:	SANTE 2017/10632 Rev. 5 of 22 November 2017 (i.e. the Technical Guideline on the Evaluation of Extraction Efficiency of Residue Analytical Methods), with application from May 2023, states that: *"**For renewal of product authorisations or for new product authorisations for which no change of the MRL is needed**, the data requirements used for the latest renewal or approval should be considered. In case this document did not yet apply, when the data for the latest renewal or approval were submitted, at this stage no new studies or data related to extraction efficiency are required. This means in practice that for renewal of product authorisations or for new product authorisations for which no new MRL is required, no additional proof of extraction efficiency will be required."* The guidance document did not apply when the data for the latest renewal of approval of fluroxypyr were submitted. As such, no additional data are required to address

	Method for products of animal origin
	extraction efficiency to support authorisation of KINVARA.

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

An overview on the acceptable methods and possible data gaps for analysis of fluroxypyr in soil is given in the following tables.

XXXX was considered to have a complete Annex II data package in support of the inclusion of fluroxypyr on to Annex I. As part of the fluroxypyr product reauthorisations in the UK, CRD concluded the "*methods of analysis for monitoring in…environmental samples including soil, …are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed."*

Table 5.3-7: Validated methods for soil

Component of residue definition: *fluroxypyr & its salts expressed as fluroxypyr*			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary & Confirmatory	0.01 mg/kg	GC-MSD	Shackelford, D.D, 1999 / Method GRM 98.04 / EU agreed

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

An overview on the acceptable methods and possible data gaps for analysis of fluroxypyr in surface and drinking water is given in the following tables.

XXXX was considered to have a complete Annex II data package in support of the inclusion of fluroxypyr on to Annex I. As part of the fluroxypyr product reauthorisations in the UK, CRD concluded the "*methods of analysis for monitoring in…environmental samples including water, …are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed."*

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

Component of residue definition: *fluroxypyr & its salts expressed as fluroxypyr*				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water	Primary	As per the EFSA final conclusions on fluroxypyr [*EFSA Journal 2011; 9(3) :2091*] an acceptable method of analysis is required for drinking water. However these data are only required at re-renewal so no further data are required at present.		
	ILV			
Surface water	Primary & confirmatory	5 µg/L	GC-MS	Shackelford, D.D, 2000 / Method GRM 00.21 / EU agreed

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

An overview on the acceptable methods and possible data gaps for analysis of fluroxypyr in air is given in the following table.

XXXX was considered to have a complete Annex II data package in support of the inclusion of fluroxypyr on to Annex I. As part of the fluroxypyr product reauthorisations in the UK, CRD concluded the "*methods of analysis for monitoring in …environmental samples including air, …are fully validated in the EU DAR for fluroxypyr. These data are out of protection. Therefore no further data have been assessed."*

Table 5.3-9: Validated methods for air

Component of residue definition: *fluroxypyr & its salts expressed as fluroxypyr*			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	≤24 µg/m3	LC-MS/MS	Backer, R, 2009 / Method B1644 G / EU agreed

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

No analytical methods were provided for the analysis of fluroxypyr in body fluids and tissues at the time of renewal as methods were not required if the active substance was not classified as toxic or highly toxic. It is noted that, in accordance with SANTE/2020/128360 rev.2 and the data requirements outlined in Regulation (EC) No 283/2013, methods for the analysis of body fluids and tissues are required. However, this should be addressed as part of the active substance renewal program.

5.3.2.8 Other studies/ information

No other studies or information is provided for the active substance fluroxypyr.

zRMS: the applicant's data in **5.3.2** is acceptable.

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

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

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

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high protein/high starch content (dry commodities)	MCPA and MCPB (MCPA, MCPB including their salts, esters and conjugates expressed as MCPA) (Reg. (EU) No 491/2014)	0.05 mg/kg (LOQ)	Wasser C., 2000/Method ATM 592/ EU agreed; Pfarl C, 1994/ EU agreed
Muscle	MCPA, MCPB and MCPA thioethyl expressed as MCPA. (Reg. (EU) No 491/2014)	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.	
Milk		Method not described in the DAR; XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.	King D.L., Johnson T, 2001/EU Agreed
Eggs		XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.	
Fat			
Liver, kidney			
Soil (Ecotoxicology)	MCPA, MCPB and MCPA thioethyl expressed as MCPA (Reg. (EU) No 491/2014)	50 mg/kg (LOQ)	Sattar MA and J Paasivirta, 1979/ EU agreed
Drinking water (Human toxicology)	MCPA, MCPB and MCPA thioethyl expressed as MCPA (Reg. (EU) No 491/2014)	0.1 µg/L (LOQ)	General limit for drinking water
Surface water (Ecotoxicology)	MCPA, MCPB and MCPA thioethyl expressed as MCPA. (Reg. (EU) No 491/2014)	5 µg/L (LOQ)	HMSO Publication, "Methods for the examination of waters and associated materials"/1997
Air	MCPA, MCPB and MCPA thioethyl expressed as MCPA (Reg. (EU) No 491/2014)	0.6 µg/m^3 (LOQ)	Reichert N, 1994/ EU agreed
		0.24 µg/m^3 (LOQ)	Werrer Zangmeister, 1995/EU agreed
Tissue (meat or liver)	MCPA, MCPB and MCPA thioethyl expressed as MCPA (Reg. (EU) No 491/2014)	Not required	not classified as T / T+
Body fluids		Not required	not classified as T / T+

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

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

Component of residue definition: **Food of plant origin:** *MCPA and MCPB (MCPA, MCPB including their salts, esters and conjugates expressed as MCPA)* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High protein/high starch content (dry)	Primary	0.05 mg/kg	GC-MSD	Wasser C., 2000/Method ATM 592/ EU agreed; Pfarl C, 1994/ EU agreed
	ILV	0.05 mg/kg	GC-MSD	Harper, H 2005/ATM 592/EU Agreed

Table 5.3-12: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	-
Not required, because:	SANTE 2017/10632 Rev. 5 of 22 November 2017 (i.e. the Technical Guideline on the Evaluation of Extraction Efficiency of Residue Analytical Methods), with application from May 2023, states that: *"**For renewal of product authorisations or for new product authorisations** or extension of uses **for which no change of the MRL is needed**, the data requirements used for the latest renewal or approval should be considered. This means that no additional proof of extraction efficiency is required if it had not been required in the renewal of approval/approval procedure itself. Extraction efficiency should be addressed if for a product authorization a different analytical methodology (in methods for risk assessment and/or monitoring) is used, compared to that of the approval/renewal procedure of the active substance."* The guidance document did not apply when the data for the latest renewal of approval of MCPA were submitted. As such, no additional data are required to address extraction efficiency to support authorisation of KINVARA.

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

Table 5.3-13: Validated methods for food and feed of animal origin

Component of residue definition: *MCPA, MCPB and MCPA thioethyl expressed as MCPA* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Milk	Primary	Method not described in the DAR; XXXX have access to a full Annex II data package		King D.L., Johnson T, 2001/EU Agreed

Component of residue definition: *MCPA, MCPB and MCPA thioethyl expressed as MCPA* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
		from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
	ILV	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
Eggs	Primary	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
	ILV	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
Muscle	Primary	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
	ILV	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
Fat	Primary	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
	ILV	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
Kidney, liver	Primary	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		
	ILV	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		

Table 5.3-14: Statement on extraction efficiency

	Method for products of animal origin
Required, available from:	-
Not required, because:	SANTE 2017/10632 Rev. 5 of 22 November 2017 (i.e. the Technical Guideline on the Evaluation of Extraction Efficiency of Residue Analytical Methods), with application from May 2023, states that: *"**For renewal of product authorisations or for new product authorisations** or extension of uses **for which no change of the MRL is needed**, the data requirements used for the latest renewal or approval should be considered. This means that no additional proof of extraction efficiency is required if it had not been required in the renewal of approval/approval procedure itself. Extraction efficiency should be addressed if for a product authorization a different analytical methodology (in methods*

	Method for products of animal origin
	for risk assessment and/or monitoring) is used, compared to that of the approval/renewal procedure of the active substance." The guidance document did not apply when the data for the latest renewal of approval of MCPA were submitted. As such, no additional data are required to address extraction efficiency to support authorisation of KINVARA.

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

Table 5.3-15: Validated methods for soil

Component of residue definition: *MCPA, MCPB and MCPA thioethyl expressed as MCPA* (Reg. (EU) No 491/2014)			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	50 mg/kg	Sattar MA and J Paasivirta, 1979/ EU agreed	Sattar MA and J Paasivirta, 1979/ EU agreed
Confirmatory	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		

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

Table 5.3-16: Validated methods for water

Component of residue definition: *MCPA, MCPB and MCPA thioethyl expressed as MCPA.* (Reg. (EU) No 491/2014)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water	Primary	0.1 µg/L	GC-MS	general limit for drinking water
	ILV			XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.
Surface water	Primary	5 µg/L	GC-MS	HMSO Publication, "Methods for the examination of waters and associated materials"/1997
	Confirmatory	XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.		

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

Table 5.3-17: Validated methods for air

Component of residue definition: *MCPA, MCPB and MCPA thioethyl expressed as MCPA* (Reg. (EU) No 491/2014)			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.6 µg/m3	HPLC-MS	Reichert N, 1994/ EU agreed
	0.240.6 µg/m3	HPLC/UV	Werrer Zangmeister, 1995/EU agreed

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

XXXX have access to a full Annex II data package from the notifier Nufarm in respect of the active substance MCPA which grants access to all Annex II data including renewal data. Please refer to enclosed LoA.

5.3.3.8 Other studies/ information

No other studies or information is provided for the active substance MCPA.

zRMS: the applicant's data in **5.3.3** is acceptable.

5.3.4 Description of analytical methods for the determination of residues of Clopyralid (KCP 5.2)

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

Table 5.3-18: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required

Matrix	**Residue definition**	**MRL / limit**	**Reference for MRL/level Remarks**
Food of plant origin	**Existing:** Clopyralid (Reg. (EU) No 2021/1807) **Proposed:** Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of "polar clopyralid" (EFSA, 2018)	0.5 mg/kg (all groups except for sugar plants) 0.05 mg/kg (LOQ) (sugar plants)	Reg. (EU) No 2021/1807

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Food of animal origin	**Existing:** Clopyralid (Reg. (EU) No 2021/1807) **Proposed:** Clopyralid, its salts and conjugates, expressed as clopyralid (EFSA, 2018)	0.05 mg/kg (LOQ) (all animal matrices)	Reg. (EU) No 2021/1807
Soil (Ecotoxicology)	Clopyralid (EFSA, 2018)	0.05 mg/kg	Common limit
Drinking water (Toxicology)	Clopyralid (EFSA, 2018)	0.1 µg/L (LOQ)	General limit for drinking water
Surface water (Ecotoxicology)	Clopyralid (EFSA, 2018)	3 mg a.s./L	EC50 (*Myriophyllum spicatum*)
Air	Clopyralid (EFSA, 2018)	45 µg/m^3	AOEL$_{systemic}$: 0.15 mg/kg bw/d (EFSA, 2018)
Body fluids and tissues	Clopyralid (EFSA, 2018)	Body fluids: 0.01 mg/L[1] Body tissue: 0.01 mg/kg[2]	Common limit

[1] The common limit according to the guidance which applied at the latest renewal of clopyralid (SANTE/825/00 rev 8.1) was 0.05 mg/L, which is higher than the LOQ of 0.01 mg/L specified in SANTE/2020/12830.
[2] Residues in body tissues can be determined by using the monitoring methods for residues in food of animal origin (EFSA, 2018).

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

Table 5.3-19: Validated methods for food and feed of plant origin (required for all matrix types, "difficult" matrix only when indicated by intended GAP)

Component of residue definition: *Clopyralid* (Reg. (EU) No 2021/1087)[1]				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High protein/high starch content (dry) High oil High acid High water	Primary (wheat forage, wheat grain, orange, canola seed)	0.01 mg/kg	LC-MS/MS	Vogl, E., 2012, CA 4.2 (a)/1 (EFSA, 2018) EU agreed
	Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		
	ILV (wheat whole plant, canola seed)	0.01 mg/kg	LC-MS/MS	Austin, R., 2012, CA 4.2 (a)/2 (EFSA, 2018) EU agreed
	Primary (lettuce, lemon)	0.01 mg/kg	LC-MS/MS (QuEChERS)	Author names redacted, 2013, CA 4.2 (a)/5 (EFSA, 2018) EU agreed
	Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		

Component of residue definition: *Clopyralid* (Reg. (EU) No 2021/1087)[1]				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
	ILV (lettuce, lemon)	0.01 mg/kg	LC-MS/MS (QuEChERS)	Author names redacted, 2014, CA 4.2 (a)/6 (EFSA, 2018) EU agreed

[1] A residue definition of *Clopyralid common moiety (sum of clopyralid, its salts and conjugates expressed as clopyralid) – pending the outstanding clarification on the nature of "polar clopyralid"* is proposed in EFSA, 2018.

Table 5.3-20: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	-
Not required, because:	SANTE 2017/10632 Rev. 5 of 22 November 2017 (i.e. the Technical Guideline on the Evaluation of Extraction Efficiency of Residue Analytical Methods), with application from May 2023, states that: *"**For renewal of product authorisations or for new product authorisations** or extension of uses **for which no change of the MRL is needed**, the data requirements used for the latest renewal or approval should be considered. This means that no additional proof of extraction efficiency is required if it had not been required in the renewal of approval/approval procedure itself. Extraction efficiency should be addressed if for a product authorization a different analytical methodology (in methods for risk assessment and/or monitoring) is used, compared to that of the approval/renewal procedure of the active substance."* The guidance document did not apply when the data for the latest renewal of approval of clopyralid were submitted. As such, no additional data are required to address extraction efficiency to support authorisation of KINVARA.

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

Table 5.3-21: Validated methods for food and feed of animal origin

Component of residue definition: *Clopyralid* (Reg. (EU) 2021/1807)[1]				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Milk, egg, fat, liver, kidney, muscle	Primary (muscle, fat, kidney, liver, milk, egg)	0.01 mg/kg	LC-MS/MS	Author names redacted, 2012, CA 4.2 (a)/3 (EFSA, 2018) EU agreed
	Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		
	ILV (muscle, liver, eggs, milk)	0.01 mg/kg	LC-MS/MS	Author names redacted, 2012, CA 4.2 (a)/4 (EFSA, 2018) EU agreed
	Primary (fat, milk)	0.01 mg/kg	LC-MS/MS (QuEChERS)	Author names redacted, 2013, CA 4.2 (a)/5 (EFSA, 2018) EU agreed

Component of residue definition: *Clopyralid* (Reg. (EU) 2021/1807)[1]				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
	Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		
	ILV (fat)	0.01 mg/kg	LC-MS/MS (QuEChERS)	Author names redacted, 2014, CA 4.2 (a)/6 (EFSA, 2018) EU agreed

[1] A residue definition of *Clopyralid, its salts and conjugates, expressed as clopyralid* is proposed in EFSA, 2018.

Table 5.3-22: Statement on extraction efficiency

	Method for products of animal origin
Required, available from:	-
Not required, because:	SANTE 2017/10632 Rev. 5 of 22 November 2017 (i.e. the Technical Guideline on the Evaluation of Extraction Efficiency of Residue Analytical Methods), with application from May 2023, states that: *"**For renewal of product authorisations or for new product authorisations** or extension of uses **for which no change of the MRL is needed**, the data requirements used for the latest renewal or approval should be considered. This means that no additional proof of extraction efficiency is required if it had not been required in the renewal of approval/approval procedure itself. Extraction efficiency should be addressed if for a product authorization a different analytical methodology (in methods for risk assessment and/or monitoring) is used, compared to that of the approval/renewal procedure of the active substance."* The guidance document did not apply when the data for the latest renewal of approval of clopyralid were submitted. As such, no additional data are required to address extraction efficiency to support authorisation of KINVARA.

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

Table 5.3-23: Validated methods for soil

Component of residue definition: *Clopyralid* (EFSA, 2018)			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.50 µg/kg	LC-MS/MS	Vincent, T. P., 2013, CA 4.2 (b)/1 (EFSA, 2018) EU agreed
Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		

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

Table 5.3-24: Validated methods for water

Component of residue definition: *Clopyralid* (EFSA, 2018)				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water, ground water, surface water	Primary	0.05 µg/L	LC-MS/MS	Shaffer, S., 2012, CA 4.2 (b)/3 (EFSA, 2018) EU agreed
	Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		
	ILV	0.05 µg/L	LC-MS/MS	Austin, R., Turner, R., 2013, CA 4.2 (b)/4 (EFSA, 2018) EU agreed

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

Table 5.3-25: Validated methods for air

Component of residue definition: *Clopyralid* (EFSA, 2018)			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	4.5 µg/m^3	LC-MS/MS	Bacher, R. 2012, CA 4.2 (c)/1 (EFSA, 2018) EU agreed

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

Table 5.3-26: Validated methods for body fluids and tissues

Component of residue definition: *Clopyralid* (EFSA, 2018)			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.05 mg/L (blood, urine)	LC-MS/MS	2014, CA 4.2 (d)/1 (EFSA, 2018) EU agreed
Confirmatory	Not required – the primary method monitoring two ion transitions per analyte/matrix is considered highly specific.		

The common limit according to the guidance which applied at the latest renewal of clopyralid (SANTE/825/00 rev 8.1) was 0.05 mg/L, which is higher than the LOQ of 0.01 mg/L specified in SANTE/2020/12830 rev. 2. As this guidance document did not apply when the data for the latest renewal of approval of clopyralid were submitted, and the monitoring method was accepted during the latest EU per review (EFSA, 2018), the analytical method is considered sufficient.

Clopyralid residues in body tissues can be determined by using the monitoring methods for residues in food of animal origin (EFSA, 2018) to an LOQ of 0.01 mg/kg.

5.3.4.8 Other studies/ information

No other studies or information is provided for the active substance Clopyralid

zRMS: the applicant's data in **5.3.4** is acceptable.

Appendix 1 Lists of data considered in support of the evaluation

List of data submitted by the applicant and relied on

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCP 5.1.1	Hubbard, S.	2014	Validation of the analytical methods used to determine the active ingredients in “Kinvara”, a micro-emulsion (ME) formulation, containing MCPA, Clopyralid and Fluroxypyr. Report No. OA02413 Oxford Analytical Ltd GLP Unpublished	N	XXXX
KCP 5.1.1	Pomeroy, D.	2017	Validation of the method of a specified impurity in Fluroxypyr-Meptyl technical material, in compliance with Good Laboratory Practice, Report No. DNA4071 David Norris Analytical Ltd. GLP Unpublished	N	XXXX
KCP 5.1.2/42 (KCA 6.3.1/01)	A. Witte	2015	Magnitude of Fluroxypyr, MCPA and Clopyralid Residues in Wheat Following One Application with Kinvara (Fluroxypyr-meptyl 72 g/L + MCPA 233 g/L + Clopyralid 28 g/L, EW) in Southern and Northern Europe in 2014 Report: TRC14-045 GLP Published: No	N	XXXX
KCP 5.1.2/43 (KCA 6.3.2/01)	A. Witte	2015	Magnitude of Fluroxypyr, MCPA and Clopyralid Residues in Barley Following One Application with Kinvara (Fluroxypyr-meptyl 72 g/L + MCPA 233 g/L + Clopyralid 28 g/L, EW) in Southern and Northern Europe in 2014 Report: TRC14-059 GLP Published: No	N	XXXX

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCP 5.1.2/44 (KCA 6.3.3/01)	Diebold, J.	2018	Determination of MCPA Residues in Grass Following One Foliar application with KINVARA under Field Conditions in Northern Europe in 2018 Report: R B8208 GLP Published: No	N	XXXX

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
KCP 5.1.2/01 KCP 5.2/01	Olberding, E.L., NG, C.A.	1996	Validation report for the determination of residues of fluroxypyr and fluroxypyr-1-mepthylhepylester as the acid equivalent in the grain, forage, straw and hay of wheat, barley and oats by capillary gas chromatography with mass selective detection (GRM 96.02 supplementary) 1996-06-04 Including: determination of residues of fluroxypyr-1-mepthylheptylester as the acid equivalent in the grain, forage, straw and hay of wheat, barley and oats by capillary gas chromatography with mass selective detection GRM 96.02. Company Report No. GH-C 4049 (RES 95118) Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/02 KCP 5.2/02	Mc Kellar, R.L., Mac Gregor, J.A. & Markley, B.J.	1996	Independent laboratory validation of method GRM 96.02 - determination of residues of fluroxypyr-1-mepthylheptylester as the acid equivalent in the grain, forage, straw and hay of wheat, barley and oats by capillary gas chromatography with mass selective detection. Company Report No. GH-C 4166 (RES 96044) Source; fluroxypyr GLP Unpublished	N	DOW

KCP 5.1.2/03	Maycock, C. & Teasdale, R.	1990	Determination of fluroxypyr residues in maize plant and kernels, ERC 89.5. Company Report No. DOWM 10073 DE90A (O38) Source; fluroxypyr Non-GLP Unpublished	N	DOW
KCP 5.1.2/04	Maycock, C. & Teasdale, R.	1992	Determination of fluroxypyr mepthylheptyl ester in immature maize plants, supplement to ERC 89.3 Company Report No. 35402 Source; fluroxypyr Non-GLP Unpublished	N	DOW
KCP 5.1.2/05	Teasdale, R	1995b	Independent validation of Dow Elanco analytical method ERC 89.5 for the determination of fluroxypyr residues in maize plant and cob Company Report No. 42375, GHE-P-4647 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/06 KCP 5.2/03	Olberding, E.L, Huskin M.A	1996b	Validation report for the determination of residues of fluroxypyr in ruminant tissues and milk by capillary gas chromatography with mass selective detection (validation for analytical method GRM 96.03) Company Report No. GCH-C-4048 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/07 KCP 5.2/04	Reed, D.E., Bottom, S.N	2003	Independent laboratory validation of Dow Agro Sciences LLC method GRM 96.03 - determination of residues of fluroxypyr in ruminant tissues and milk by capillary gas chromatography with mass selective detection. Company Report No. 30198040-5008-1 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/08 KCP 5.2/05	Shackelford, D.D	2009	Determination of residues of fluroxypyr in poultry tissues and eggs by liquid chromatography with tandem mass spectrometry. Company Report No. 081043 Source; fluroxypyr GLP Unpublished	N	DOW

KCP 5.1.2/09 KCP 5.2/06	Senciuc, M & Class, T	2009	Independent laboratory validation of Dow Agro Sciences LLC Method GRM 08.03 - Determination of residues of fluroxypyr in poultry tissues and eggs by liquid chromatography with tandem mass spectrometry. Company Report No. P. 1545G Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/10	Moore, M	1996	Determination of residues of fluroxypyr (4-amino-3,5-dichloro-6-fluoro-2-oxy acetic acid), methoxypyridine (4-amino-3,5-dichloro-6-flouro-2-methoxy)pyridine) and 3,5-dichloropyridinol ((4-amino-3,5-dichloro-6-fluoro-2-hydroxy)pyridine) in soil by capillary gas chromatography using mass selective detection, GRM 9.03 Company Report No. GH-C 4170 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/11	Phillips, A.M., Miller R.N.	1997	Independent laboratory validation method, Dow Elanco, GRM 93.03 Company Report No. GH-C 4390 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/12 KCP 5.2/07	Shackleford, D.D	1999a	Method validation for the determination of fluroxypyr and its major metabolites in soil by gas chromatography with mass selective detection. GRM 98.04 Company Report No. GH-C 4908 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/13 KCP 5.2/08	Shackleford, D.D	1999b	Independent laboratory validation method GRM 98.04 - determination of fluroxypyr and its major metabolites in soil by gas chromatography with mass selective detection. GRM 98.04 Company Report No. GH-C 4985 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/14	Van Dyke, M.E.	1991	Determination of fluroxypyr meptylester in soil by gas chromatography / mass spectrometry. Company Report No. MET94-00058 Source; fluroxypyr GLP Unpublished	N	DOW

KCP 5.1.2/14 KCP 5.2/09	Shackleford, D. D	2000	Validation report for the determination of fluroxypyr 1-mepthylheptyl ester, fluroxypyr acid, fluroxypyr 2-pyridinol and fluroxypyr-2-methoxypyridine in surface water by capillary gas chromatography with mass spectrometric detection, GRM 00.21 Company Report No. GH-C 5157 Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/15 KCP 5.2/10	Bacher, R.	2009	The development and validation of a method for the analysis of fluroxypyr-acid and fluroxypyr-1-mepthylheptyl ester in air Company Report No. B1644G Source; fluroxypyr GLP Unpublished	N	DOW
KCP 5.1.2/16 KCP 5.2/11	Flynn S G	1979	Determination of 2-methyl-4-chlorophenoxy acetic acid (MCPA) residues in spring barley grain and straw, oat grain and straw, grass and hay Generated by: Nufarm Belvedere former Rhône Poulenc Submitted by: MCPA Dossier Preparation Working Group File No: 1707-35/1/2 Not GLP, Unpublished	N	-
KCP 5.1.2/17 KCP 5.2/12	Keller, W	1980	Method for determination of MCPA residues in grass, wheat and grapes. Generated by: BASF AG/TPH Submitted by: MCPA Dossier Preparation Working Group File No: 143 (173) Not GLP, Unpublished	N	-
KCP 5.1.2/18 KCP 5.2/13	Pfarl C	1994	Pfarl C Validation of an analytical method for determination of residues of MCPA in cereals. Generated by: Agrolinz Mclamin GmbH Submitted by: MCPA Dossier Preparation Working Group File No: 1172a GLP, Unpublished	N	MCPA Task Force
KCP 5.1.2/19 KCP 5.2/14	C. Wasser	2000	Validation of analysis for MCPA, MCPB and HMCPA residue in Cereals Final report N. R9110 Anadiag GLP, Unpublished	N	Task Force AH Marks
KCP 5.1.2/20 KCP 5.2/15	Harper	2005	MCPA – Independent validation of methodology for the determination of MCPA in grain and straw and validation of the methodology for the determination of MCPA in apples Method ATM 592	N	DOW

KCP 5.1.2/21 KCP 5.2/16	Hastings, M.J.	2002a	Determination of residues of clopyralid on agricultural crops by gas chromatography with negative-ion chemical ionization mass spectrometry. Dow AgroSciences LLC Report:GH-C-5439 (O97) GLP, Unpublished	N	DOW
KCP 5.1.2/22 KCP 5.2/17	Rawle, N.	2002	Independent laboratory validation of a method for the determination of clopyralid residues in crops. CEM Analytical Services Ltd Report: CEMS-1671 (O95) GLP, Unpublished	N	DOW
KCP 5.1.2/23 KCP 5.2/18	Wood, S.J.	1994	Determination of residues of clopyralid in grass Dow Elanco Europe Report: RV94.08 (O47) GLP, Unpublished	N	DOW
KCP 5.1.2/24 KCP 5.2/19	Freeman, J.M.H. and Smith, D.W.	1983	Analytical method: Determination of Clopyralid residues in wheat grain and straw Dow Chemical Company Report: ERC 83.23 (O25) Not GLP, Unpublished	N	DOW
KCP 5.1.3/25 KCP 5.2/20	Kutschinski, A.H.	1979	Determination of residues of 3,6-dicloropicolinic acid and 2,4-D in barley and wheat by gas chromatography. Dow Chemical USA Report No: ACR 79.5 Non GLP, Unpublished (O38)	N	DOW
KCP 5.1.3/26 KCP 5.2/21	Jones, E.M.	1975a	Determination of 3,6-dicloropicolinic acid (DOWCO 290) in wheat and barley grain and straw by gas chromatography Dow Chemical Company Report No: ERC 77.4 Not GLP, Unpublished (O23)	N	DOW
KCP 5.1.2/27 KCP 5.2/22	Kutschinski, A.H.	1974	Determination of DOWCO 290 in milk and cream by gas chromatography Dow Chemical Report No: ACR 74.3 Non GLP., Unpublished (O12)	N	DOW
KCP 5.1.2/28 KCP 5.2/23	Kuper, A.W.	1975	Determination of 3,6-dicloropicolinic acid in chicken tissues and eggs by gas chromatography Dow Chemical Report No: ACR 75.2 Non GLP. Unpublished (O13)	N	DOW

KCP 5.1.2/29 KCP 5.2/24	Kuper, A.W.	1974a	Determination of residues of 3,6 –dicloropicolinic acid in chicken tissues and eggs by gas chromatography Dow Chemical Report No: ACR 74.2 Non GLP, Unpublished(O11)	N	XXXX
KCP 5.1.2/30 KCP 5.2/25	Hastings, M.J.	2002b	Determination of residues of clopyralid in animal tissues by gas chromatography with negative ion chemical ionization mass spectrometry. Dow AgroSciences LLC Report No: GH-C-5440 (GRM 02.14) GLP, Unpublished(O96)	N	XXXX
KCP 5.1.2/31 KCP 5.2/26	Kuper, A.W	1974b	Determination of 3,6-dicloropicolinic acid in bovine tissues by gas chromatography Doe Chemical Report No: ACR 74.9 Non GLP, Unpublished (O40)	N	MCPA Task Force
KCP 5.1.2/34 KCP 5.2/29	HMSO Publication, “Methods for the examination of waters and associated materials”	1997	The determination of acid herbicides in waters by GC-MS	N	HMSO Publication
KCP 5.1.2/35 KCP 5.2/30	HMSO Publication, “Methods for the examination of waters and associated materials”	1997	The determination of acidic herbicides in waters by di-isopropyl ether extraction (a note)	N	HMSO Publication
KCP 5.1.2/37 KCP 5.2/32	Reichert N	1994	Development and validation of a method for the determination of 2,4-D, MCPA, Dichlorprop-P and Mercoprop-P in air Submitted by: MCPA Dossier Preparation Working Group File No: RCC 439705 GLP, Unpublished	N	MCPA Task Force
KC KCP 5.1.2/38 KCP 5.2/33	Werrer Zangmeister	1995	Recovery of 2,4-DK, MCPA-DMA, Mercoprop-P-DMA and Dichlorprop-P-DMA after elution from TENAX-supplement to analytical method RCC project 439 705	N	-

KCP 5.1.2/41 KCP 5.2/36	King DL. Johnson	2001	Validated Analytical Method for the Determination of MCPA, MCPA glycine conjugate, HMCPA and HMCPA glucose conjugate in beef tissues, milk and cream MCPA DPWG PTRL USA GLP, Unpublished	N	-
CA 4.2 (a)/1	Vogl, E.	2012	Method Validation Study for the Determination of Residues of Clopyralid and Picloram in Agricultural Commodities by LC-MS/MS ABC Laboratories, Inc., Columbia, Missouri, USA DAS Report No. 120610 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
CA 4.2 (a)/2	Austin, R.	2012	Independent Laboratory Validation of Dow AgroSciences Method 120610, "Method Validation Study for the Determination of Residues of Clopyralid and Picloram in Agricultural Commodities by LC-MS/MS" Battelle UK Ltd, Ongar, Essex, United Kingdom DAS Report No. 120614 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
CA 4.2 (a)/3	-	2012	Method Validation Study for the Determination of Residues of Clopyralid in Bovine and Poultry Matrices by Liquid Chromatography with Tandem Mass Spectrometry Detection XXXX Report No. 120483 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS
CA 4.2 (a)/4	-	2012	Independent Laboratory Validation of an Analytical Method for the Determination of Clopyralid in Animal Matrices XXXX Report No. 120484 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS
CA 4.2 (a)/5	-	2013	Validation of a Multi-residue Method Following the QuEChERS Sample Preparation Technique for the Determination of Clopyralid in Matrices of Plant and Animal Origin XXXX Report No. 130729 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS

CA 4.2 (a)/6	-	2014	Independent Laboratory Validation of a Multi-residue Method Following the QuEChERS Sample Preparation Technique for the Determination of Clopyralid in Matrices of Plant and Animal Origin XXXX Report No. 130728 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS
CA 4.2 (b)/1	Vincent, T. P.	2013	Method Validation Study for the Determination of Residues of Clopyralid and Picloram in Soil by LC-MS/MS ABC Laboratories, Inc., Columbia, Missouri, USA DAS Report No. 120612 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
CA 4.2 (b)/2	Austin, R., Turner, R.	2014	Independent Laboratory Validation of a Dow AgroSciences Method for the Determination of Residues of Clopyralid and Picloram in Soil by LC-MS/MS Battelle UK Ltd, Chelmsford, Essex, United Kingdom DAS Report No. 140079 GLP/GEP (Y/N): Yes Published (Y/N): N	N	DAS
CA 4.2 (b)/3	Shaffer, S.	2012	Method Validation Study for the Determination of Residues of Clopyralid and Picloram in Drinking Water, Ground Water, and Surface Water by LC-MS/MS ABC Laboratories, Inc., Columbia, Missouri, USA DAS Report No. 120611 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
CA 4.2 (b)/4	Austin, R., Turner, R.	2013	Independent Laboratory Validation of Dow AgroSciences Method 120611, “Method Validation Study for the Determination of Residues of Clopyralid and Picloram in Drinking Water, Ground Water, and Surface Water by LC-MS/MS” Battelle UK Ltd, Ongar, Essex, United Kingdom DAS Report No. 120613 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS

CA 4.2 (c)/1	Bacher, R.	2012	The Development and Validation of a Method for the Analysis of Clopyralid in Air PTRL Europe GmbH, D-89081 Ulm, Germany DAS Report No. 120601 GLP/GEP (Y/N): Yes Published (Y/N): No	N	DAS
CA 4.2 (d)/1	-	2014	Development and Validation of an Analytical Method for the Determination of Clopyralid in Body Fluid(s) XXXX Report No. 130727 GLP/GEP (Y/N): Yes Published (Y/N): No	Y	DAS

Appendix 2 Detailed evaluation of submitted analytical methods

A 2.1 Analytical methods for MCPA, Fluroxypyr, Clopyralid

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

The following additional studies are being submitted by XXXX to provide further information in the form of studies TRC14-045 (wheat), TRC14-059 (barley) and R B8208 (grassland).

A 2.1.1.1.1 Analytical method 1

Comments of zRMS:	The method validations have been accepted. The analytical methods for analysis of Fluroxypyr acid and Fluroxypyr-meptyl, Clopyralid and MCPA in cereal matrices (wheat whole plant, grain and straw) were validated in the present study. A full set of recoveries (5 recoveries at LOQ, 5 recoveries at least at one higher level, two blank samples) was prepared for each matrix under investigation.

Report:	KCP 5.1.2/42 Witte, A. (2015)
Title:	Magnitude of Fluroxypyr, MCPA and Clopyralid Residues in Wheat Following One Application with Kinvara (Fluroxypyr-meptyl 72 g/L + MCPA 233 g/L + Clopyralid 28 g/L, EW) in Southern and Northern Europe in 2014
Document No:	TRC14-045
Guidelines:	EU Regulation 1107/2009, 21 October 2009 Guidance document SANCO/3029/99 rev. 4
GLP	Yes

Executive Summary
The aim of the analytical phase of the current study was to determine residues of Fluroxypyr (analysed as sum of Fluroxypyr and Fluroxypyr meptyl ester according to the MRL definition), MCPA and Clopyralid in wheat specimens (whole plant, grain and straw) after a single application of Kinvara (formulation containing Fluroxypyr-meptyl, MCPA and Clopyralid). Specimens for determination of residues of Fluroxypyr and Fluroxypyr meptyl ester are extracted and the extracts cleaned up following an analytical method described in the DAR of Fluroxypyr (method GRM 96.02, DOW Agrosciences, 1996). Esters of Fluroxypyr are hydrolysed during a hydrolysis step. The total residue of Fluroxypyr acid and its esters are analysed as Fluroxypyr acid by HPLC-MS/MS.

Specimens for determination of residues of MCPA and Clopyralid are analysed following a modification of the published QuEChERS method (DIN EN 15662), involving a hydrolysis step to ensure extraction of the acidic compounds during extraction.

Materials and Methods

Test material:	Fluroxypyr (free acid)
Description:	Not stated
Lot/Batch #:	SZBB329XV
Purity:	98.9%
Development code:	Not applicable
CAS No:	69377-81-7

Test material:	Fluroxypyr meptyl ester
Description:	Not stated
Lot/Batch #:	SZBC066XV
Purity:	99.3%
Development code:	Not applicable
CAS No:	81406-37-3

Test material:	MCPA
Description:	Not stated
Lot/Batch #:	SZBC129XV
Purity:	99.8%
Development code:	Not applicable
CAS No:	94-74-6

Test material:	Clopyralid
Description:	Not stated
Lot/Batch #:	90909
Purity:	97.7%
Development code:	Not applicable
CAS No:	1702-17-6

Results

Calibration (linearity)
The linearity of the detector response was confirmed by injecting at least five matrix-matched standard solutions covering the following working ranges:

Fluroxypyr acid:	0.05 ng/mL to 50 ng/mL for *whole plant* and *straw* samples 0.2 ng/mL to 200 ng/mL for *grain* samples
Clopyralid, MCPA:	1 ng/mL to 1000 ng/mL for *whole plant* and *grain* samples 0.75 ng/mL to 500 ng/mL for *straw* samples

The correlation coefficients (r^2) were ≥ 0.99. The lower margin of the linearity test was below 30 % of the LOQ. Representative calibration graphs are shown in Appendix 3 in TRC14-045.

Specificity
The concentration of the analytes in the final extracts was determined by high performance liquid chromatography with MS/MS detection, which is considered to be a highly specific detection method.

Example chromatograms of standards and specimens are presented in Appendix 4 (Fluroxypyr acid) and Appendix 5 (Clopyralid and MCPA) of study report TRC14-045.

Accuracy and precision
Recovery values obtained by HPLC-MS/MS at all fortification levels comply with the standard acceptance criteria of SANCO/3029/99, which demands that the mean recovery at each fortification level should be in the range of 70 – 110 %.

Detailed data on preparation and analysis of the recovery samples are presented in Appendix 2 of study report TRC14-045.

Limit of quantification (LOQ)
The limit of quantification (LOQ) was defined as the lowest fortification level with mean recoveries ranging from 70 % to 110 % at a relative standard deviation (RSD) of ≤ 20 % and blanks not exceeding 30 % of the LOQ. These criteria were fulfilled for the 0.01 mg/kg fortification for all analytes. The limit of detection

(LOD) was defined as 30 % of the limit of quantification as required in the guideline SANCO/3029/99 rev.4 for residues in control samples (i.e. 0.003 mg/kg). Residues in the untreated specimens used for recovery experiments and blank samples were below 30 % of the LOQ, respectively below the limit of detection.

Conclusion
The method was successfully validated and is acceptable for data generation or as an enforcement method. Although the analytical method validation was performed prior to the publication of the SANTE/2020/12830 rev. 2 guidelines, the method is considered fit for the intended purpose (risk assessment) based on the criteria outlined in section 4.2 of the SANTE/2020/12830 rev. 2 guidelines - *Minimum validation requirement for the assessment of existing methods for risk assessment.*

A 2.1.1.1.2 Analytical method 2

Comments of zRMS:

The method validations have been accepted.
The study is also presented in the RR B7 in the context of the residue assessment.
The analytical methods for analysis of Fluroxypyr acid and Fluroxypyr-meptyl, Clopyralid and MCPA in cereal matrices (wheat whole plant, grain and straw) were validated in study TRC14-045 (analytical part 14T05029-01-RACE). A full set of recoveries (5 recoveries at LOQ, 5 recoveries at least at one higher level, two blank samples) was prepared for each matrix under investigation.
In the current study, a reduced validation set (3 recoveries at LOQ, 3 recoveries at least at one higher level, two blank samples) was prepared for the similar barley matrices under investigation. For Fluroxypyr-meptyl and Fluroxypyr acid, the analytical method was basically validated separately for each analyte to check the efficiency of the hydrolysis step of the analytical method. Together with analysis of Fluroxypyr in field samples, procedural recoveries were analyzed for the Fluroxypyr meptyl ester to have a complete check (including hydrolysis step) on the validity.
Accuracy of the analytical method for barley whole plant, straw and grain samples was studied by means of recovery experiments with blank samples fortified at different concentration levels with Fluroxypyr acid, Fluroxypyr-meptyl, Clopyralid and MCPA, at the LOQ level and at a higher level. Precision and repeatability was also estimated from these experiments.
Recovery results are summarized in the following tables.

		Barley whole plant				Barley grain				Barley straw			
Analyte	Fortification Level	Recoveries		Overall recovery		Recoveries		Overall recovery		Recoveries		Overall recovery	
		Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
	[mg/kg]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
Fluroxypyr meptyl (as Fluroxypyr acid*)	0.01	95	12.9	92	11.7	86	6.3	89	7.3	100	2.2	97	3.6
	1.0	89	13.6			93	5.9			97	1.5		
	10	89	1.9			--	--			93	3.3		
Fluroxypyr acid	0.01	94	8.0	94	5.7	90	9.3	95	8.3	94	1.1	96	2.3
	1.0	93	3.9			100	1.2			98	1.2		
Clopyralid	0.01	87	4.7	85	4.0	77	8.4	74	7.5	92	5.6	85	12.8
	1.0	85	3.4			71	4.9			71	3.3		
	50	82	0.7			--	--			91	2.5		
MCPA	0.01	103	3.3	100	4.1	101	1.7	102	1.6	102	2.3	99	3.6
	1.0	100	2.6			103	1.1			98	1.2		
	50	95	1.8			--	--			98	2.2		

** concentrations/residues calculated as Fluroxypyr acid*
RSD: Relative Standard Deviation

Report:	KCP 5.1.2/43 Witte, A. (2015)
Title:	Magnitude of Fluroxypyr, MCPA and Clopyralid Residues in Barley Following One Application with Kinvara (Fluroxypyr-meptyl 72 g/L + MCPA 233 g/L + Clopyralid

	28 g/L, EW) in Southern and Northern Europe in 2014
Document No:	TRC14-059
Guidelines:	EU Regulation 1107/2009, 21 October 2009 Guidance document SANCO/3029/99 rev. 4
GLP	Yes

Executive Summary

The aim of the analytical phase of the current study was to determine residues of Fluroxypyr (analysed as sum of Fluroxypyr and Fluroxypyr meptyl ester according to the MRL definition), MCPA and Clopyralid in barley specimens (whole plant, grain and straw) after a single application of Kinvara (formulation containing Fluroxypyr-meptyl, MCPA and Clopyralid). Specimens for determination of residues of Fluroxypyr and Fluroxypyr meptyl ester are extracted and the extracts cleaned up following an analytical method described in the DAR of Fluroxypyr (method GRM 96.02, DOW Agrosciences, 1996). Esters of Fluroxypyr are hydrolysed during a hydrolysis step. The total residue of Fluroxypyr acid and its esters are analysed as Fluroxypyr acid by HPLC-MS/MS.

Specimens for determination of residues of MCPA and Clopyralid are analysed following a modification of the published QuEChERS method (DIN EN 15662), involving a hydrolysis step to ensure extraction of the acidic compounds during extraction.

Materials and Methods

Test material:	Fluroxypyr (free acid)
Description:	Not stated
Lot/Batch #:	SZBB329XV
Purity:	98.9%
Development code:	Not applicable
CAS No:	69377-81-7
Test material:	Fluroxypyr meptyl ester
Description:	Not stated
Lot/Batch #:	SZBC066XV
Purity:	99.3%
Development code:	Not applicable
CAS No:	81406-37-3
Test material:	MCPA
Description:	Not stated
Lot/Batch #:	SZBC129XV
Purity:	99.8%
Development code:	Not applicable
CAS No:	94-74-6
Test material:	Clopyralid
Description:	Not stated
Lot/Batch #:	90909
Purity:	97.7%
Development code:	Not applicable
CAS No:	1702-17-6

Results

Calibration (linearity)

The linearity of the detector response was confirmed by injecting at least five matrix-matched standard solutions covering the following working ranges:

Fluroxypyr acid: 0.05 ng/mL to 50 ng/mL for *whole plant* and *straw* samples
0.2 ng/mL to 200 ng/mL for *grain* samples

Clopyralid, MCPA: 1 ng/mL to 1000 ng/mL for *whole plant* and *grain* samples
0.75 ng/mL to 500 ng/mL for *straw* samples

The correlation coefficients (r^2) were ≥ 0.99. The lower margin of the linearity test was below 30 % of the LOQ. Representative calibration graphs are shown in Appendix 3 in TRC14-059.

Specificity
The concentration of the analytes in the final extracts was determined by high performance liquid chromatography with MS/MS detection, which is considered to be a highly specific detection method.

Example chromatograms of standards and specimens are presented in Appendix 4 (Fluroxypyr acid) and Appendix 5 (Clopyralid and MCPA) of study report TRC14-059.

Accuracy and precision
Recovery values obtained by HPLC-MS/MS at all fortification levels comply with the standard acceptance criteria of SANCO/3029/99, which demands that the mean recovery at each fortification level should be in the range of 70 – 110 %.

Detailed data on preparation and analysis of the recovery samples are presented in Appendix 2 of study report TRC14-059.

Limit of quantification (LOQ)
The limit of quantification (LOQ) was defined as the lowest fortification level with mean recoveries ranging from 70 % to 110 % at a relative standard deviation (RSD) of ≤ 20 % and blanks not exceeding 30 % of the LOQ. These criteria were fulfilled for the 0.01 mg/kg fortification for all analytes. The limit of detection (LOD) was defined as 30 % of the limit of quantification as required in the guideline SANCO/3029/99 rev.4 for residues in control samples (i.e. 0.003 mg/kg). Residues in the untreated specimens used for recovery experiments and blank samples were below 30 % of the LOQ, respectively below the limit of detection.

Conclusion
The method was successfully independently validated and is acceptable for data generation or as an enforcement method. Although the analytical method validation was performed prior to the publication of the SANTE/2020/12830 rev. 2 guidelines, the method is considered fit for the intended purpose (risk assessment) based on the criteria outlined in section 4.2 of the SANTE/2020/12830 rev. 2 guidelines - *Minimum validation requirement for the assessment of existing methods for risk assessment.*

A 2.1.1.1.3 Analytical method 3

Comments of zRMS:	The method validation have been accepted. The document was also presented and evaluated within the residue assessment in section B7 of the present RR.

Report:	KCP 5.1.2/44 Diebold, J. (2018)
Title:	Determination of MCPA Residues in Grass Following One Foliar Application with KINVARA Under Field Conditions in Northern Europe in 2018
Document No:	R B8208
Guidelines:	EU Regulation 1107/2009 OECD TG 509, 07/09/2009 SANCO 7525/VI/95 rev. 10.3, 13 June 2017 SANCO/825/00 rev.8.1, 16/11/2010 SANCO/3029/99 rev. 4, 11/07/2000 ENV/JM/MONO(2007)17, 13/08/2007
GLP	Yes

Executive Summary

The objective of this phase of the study was to validate the analytical method for the analysis of MCPA in grass (green matter). Residues are analysed following a modification of the QuEChERS method involving a hydrolysis step to ensure extraction of the acidic compounds during extraction. The extract obtained is analysed by liquid chromatography with MS/MS detection.

Materials and methods

Test material:	MCPA
Description:	Not stated
Lot/Batch #:	BCBW0422
Purity:	99.4%
Development code:	Not applicable
CAS No:	94-74-6

5 g of homogenised grass (green matter) sample was weighed into a 50 mL centrifuge tube, spiking if relevant. 10 mL of water was added, the samples allowed to stand for 5 min, before adding 300 µL of 5N NaOH solution. Samples were shaken with a vortex mixer for 1 min, then left to stand for 30 min with occasional shaking. 300 µL of 5N sulfuric acid was added and the samples homogenised. 10 mL of acetonitrile + 0.5% formic acid mixture was added and the samples were shaken with a vortex mixer for 1 min. 4g (± 0.1g) of magnesium sulfate and 1g (± 0.1g) of sodium chloride were added and the samples were shaken with a vortex mixer for 1 min prior to centrifugation at 3500 rpm for 10 min. Extracts were decanted into 15 mL centrifuge tubes in a freezer over 2 hours. 1 mL of the extract was taken in a vial for analysis using LC-MS/MS.

LOQ: 0.01 mg/kg (all analytes, matrices).
Matrix-matched standards were used for calibration.

Typical liquid chromatography operating conditions are as follows:

Column:	Waters BEH C18 column, 2.1 x 100 mm (1.7 µm particle) ANADIAG number 227
Column Temperature:	40 °C
Injection Volume:	10 µL
Mobile Phase:	A: Water + 0.1 % formic acid B: Methanol + 0.1 % formic acid
Gradient:	**Time (min:sec) / A% / B%** 0:00 / 90 / 10 4:00 / 0 / 100 6:40 / 0 / 100 6:50 / 90 / 10
Ionisation mode:	ESI, negative
Flow rate:	0.4 mL/min

Detector Temperature:	550 °C
Ion transition(s):	200.9 → 143.0 m/z (quantification) 198.9 → 141.0 m/z (qualification)
Retention time:	MCPA ~4.1 min

Validation Results

Linearity
The linearity of the method was checked by injecting into the analytical system matrix-matched calibration solutions of MCPA, at 7 concentration levels, over the range 1.0 ng/mL to 101.0 ng/mL (corresponding to 0.002 to 0.2 mg/kg). An example of typical calibration plot, the equation of the calibration line and the linear correlation coefficient are presented in the report. The equation of the line was in the form y = m.x + c. Linear correlation coefficients were > 0.990, showing a good linearity.

LOD
The limit of detection (LOD) is the lowest measurable standard concentration estimated at 3 times the background noise with the analytical conditions used. It was set at 0.002 mg/kg.

LOQ
The limit of quantification (LOQ) is the lowest validated level where a mean recovery in the range 70-120% with a %RSD less than 20% could be obtained. It was set at 0.01 mg/kg.

Accuracy, precision and repeatability
Recovery tests were performed by untreated control samples spiked with MCPA before extraction. Results are given in the table below.

Recoveries in grass (green matter)

Sample ANADIAG No.	**Fortification level (mg/kg)**	**MCPA % Recovery** (quantifier ion transition 220.9 →143.0 m/z)
B8208 06 01 AA	0.01	90.0%
B8208 06 01 BA		84.7%
B8208 06 01 CA		79.4%
B8208 06 01 DA		95.8%
B8208 06 01 EA		81.4%
B8208 06 01 FA	0.10	91.4%
B8208 06 01 GA		92.3%
B8208 06 01 HA		91.3%
B8208 06 01 IA		87.3%
B8208 06 01 JA		89.4%

Summary of recoveries

Analyte	**Matrix**	**Fortification level (mg/kg)**	**Mean recovery (%)**	**RSD (%)**	**No. fortified samples (n)**
MCPA	Grass (green matter)	0.01	86.3	7.7	5
		0.10	90.3	2.2	5
		All levels	88.3	5.8	10

The accuracy of the method was assessed on the basis of the determined recovery rates:

	MCPA	
Matrix	Grass (green matter)	
Fortification level (mg/kg)	0.01	0.10
Single recovery rates (%)	79.4 – 95.8	87.3 – 92.3
Mean recoveries per fortification level (%)	86.3	90.3

The accuracy of the method fulfils the requirements for residue analytical methods, mean recoveries per fortification level were in the range 70-120% for samples spiked at 1 x LOQ and at 10 x LOQ.

Repeatability tests (5 recoveries at each fortification level) were performed at the LOQ and at 10x LOQ:

	MCPA	
Matrix	Grass (green matter)	
Fortification level (mg/kg)	0.01	0.10
RSD for each fortification level (%)	7.7	2.2

RSD determined was less than 20% for samples spiked at 1 x LOQ and for samples spiked at 10 x LOQ, the method therefore fulfils the requirements for residue analytical methods.

Specificity
Two untreated specimens of grass (green matter) were analysed according to the method. No interferences above 30% of the limit of quantification were recorded. Typical chromatograms of control sample for the analysis of MCPA in grass (green matter) are presented in the study report.
The identity of detected analyte was confirmed by monitoring a second mass transition. Mass spectra for MCPA are presented in the study report, justifying ion choices.

Confirmatory
The specificity of the method was evaluated through the determination of the linearity, the recovery and precision of spiked samples at the LOQ level and by the analysis of 2 blank samples for the confirmatory transition 198.9 → 141.0 m/z. Full details are presented within the study report, but are not reported here as confirmation is not a validation requirement for risk assessment methods.

Matrix Effects
The effect of matrix on the LC-MS/MS response was assessed by analysing standard solution prepared in solvent against matrix-matched calibration solutions. Matrix effects (enhancement or suppression) on the instrument response were considered significant. Consequently, matrix-matched calibration solutions were used for calibration.

Conclusion
The method is able to determine MCPA in the presence of grass (green matter) to an LOQ of 0.01 mg/kg. Although the analytical method validation was performed prior to the publication of the SANTE/2020/12830 rev. 2 guidelines, the method is considered fit for the intended purpose (risk assessment) based on the criteria outlined in section 4.2 of the SANTE/2020/12830 rev. 2 guidelines - *Minimum validation requirement for the assessment of existing methods for risk assessment.*

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

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

No new or additional studies have been submitted.

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

No new or additional studies have been submitted.

A 2.1.2.3 Description of Methods for the Analysis of Soil (KCP 5.2)

No new or additional studies have been submitted.

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

No new or additional studies have been submitted.

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

No new or additional studies have been submitted.

A 2.1.2.6 Description of Methods for the Analysis of Body Fluids and Tissues (KCP 5.2)

No new or additional studies have been submitted.

A 2.1.2.7 A.2.A.9 Other Studies/ Information

No new or additional studies have been submitted.