FINAL REGISTRATION REPORT

Part B

Section 5

Analytical Methods

Detailed summary of the risk assessment

Product code: **CHR/F/PYRA 250 EC**

Product name(s): **Etiuda 250 EC, Fermata 250 EC**

Chemical active substance:

Pyraclostrobin, 250 g/L

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

Applicant: Innvigo Sp. z o.o.

Submission date: October 2021

MS Finalisation date: 15/12/2022

Version history

When	What
December 2021	Dossier sent for evaluation
August 2022	Updates based on feedback from zRMS Poland
September 2022	zRMS evaluation of dRR
December 2022	Final version prepared by zRMS after Commenting period

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zRMS comments:
The text highlighted in grey was provided by the evaluator.

5 Analytical methods

New and additional information is highlighted in yellow.

5.1 Conclusion and summary of assessment

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

Sufficiently sensitive and selective analytical methods are available for all analytes included in the residue definitions.
Noticed data gaps are: none

Commodity/crop	Supported/ Not supported
Winter and spring cereals (winter wheat,triticale and rye, spring barley and rye)	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 pyraclostrobin in plant protection product is provided as follows:

Comments of zRMS:	This method is validated and can be applied for analysing of pyraclostrobin in the PPP.

Reference:	KCP 5.1.1/01
Report	*Validation of the Methods of Determination of Pyraclostrobin in an Emulsifiable Concentrate Formulation, in Compliance with Good Laboratory Practise*, 2020, Pomeroy, D., DNA5716
Guideline(s):	SANCO/3030/99 rev.5.
Deviations:	NO
GLP:	YES
Acceptability:	YES

Materials and methods

Study objective was to validate the methods of analysis used for the determination of Pyraclostrobin within an Emulsifiable Concentrate Formulation containing 250 g/L Pyraclostrobin, in compliance with Good Laboratory Practise.

The content of active substance in **CHR/F/PYRA 250 EC** formulation is:
Pyraclostrobin – **256.4 g/L ± 6%**

It was confirmed that the methods are specific. There were no peaks from the placebo interfering with the determined compounds. The validation parameters (linearity, repeatability and accuracy) are within the acceptance range and fulfil EU requirements given in SANCO/3030/99 rev.5.

Validation - Results and discussions

Table 5.2-1: Methods suitable for the determination of active substance pyraclostrobin in plant protection product CHR/F/PYRA 250 EC

	Pyraclostrobin
Author(s), year	Pomeroy, D., 2021
Principle of method	HPLC-DAD
Linearity (linear between mg/L / % range of the declared content) (correlation coefficient, expressed as r)	According to SPT/31, the parameters obtained as a result of validation should meet the following criteria: - linearity $R^2 \geq 0.99$ The resulting curve is linear in the tested concentrations. Correlation coefficient R^2=0. 9999
Precision – Repeatability Mean and Accuracy	According to SPT/31, the parameters obtained as a result of validation should meet the following criteria: - linearity $R^2 \geq 0.99$ - Horwitz ratio ≤ 1.0 Results obtained: %RSD = 0.713 Hr = 0.432 N = 6
Interference/ Specificity	Pyraclostrobin eluted at 8.2 minutes, and there were no other significant peaks present at the same retention time as Pyraclostrobin. A small background of around 0.01% of the measured concentration is present in the blanks.
Comment	The determined validation parameters such as specificity, linearity, limit of quantification (LOQ), repeatability (precision) and accuracy are compliant with EU requirements given in SANCO/3030/99 rev.5.

Conclusion

It was confirmed that the method is specific. There were no peaks from placebo interfering with determined compounds.
The validation parameters (specificity, linearity, instrument precision, repeatability and accuracy) are within the acceptance range and fulfil EU requirements given in *SANCO/3030/99 -rev.5*.

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

For the active substance the manufacturing impurities considered are of no toxicological or environmental concern.

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

Please refer to PART C – Confidential data.

5.2.1.4 Applicability of existing CIPAC methods (KCP 5.1.1)

For the active substance the manufacturing impurities considered are of no toxicological or environmental concern.

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 pyraclostrobin for the generation of pre-authorization data is given in the following table. For the detailed evaluation of

additional studies it is referred to Appendix 2.

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

Component of residue definition: Pyraclostrobin				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Plants (Wheat forage, straw, grain)	Primary	0.02 mg/kg	LC-MS/MS	Reinhard, K., Mackenroth, C. 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Plants, processed (beer, brewer's yeast, brewing malt spent, grains and flocs, pod barley, malt sprouts)	Primary	0.02 mg/kg	LC-MS/MS	Reinhard, K.., Mackenroth, C., 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Plants (Wheat forage, straw, grain)	Primary	0.02 mg/kg	HPLC-UV	Abdel-Baky, S., Riley, M. E., 2000, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Cow milk, muscle, liver, kidney, fat, hen egg	Primary	0.01 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	

Cow milk, muscle, liver, kidney, fat, hen egg	Primary	0.01 mg/kg	LC-MS/MS	Tilting, N., Lehmann, W., 2000, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.05 mg/kg	GC-MS	
Soil	Primary	0.01 mg/kg	HPLC-UV	Ziegler, G., 1998, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.01 mg/kg	LC-MS	Zangmeister, W., 1999, *DAR - Pyraclostrobin*
Water	Primary	0.05 µg/L	LC-MS	Staab, G., 1998, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.05 µg/L	LC-MS/MS	
Water	Primary	0.05 µg/L	LC-MS/MS	Zangmeister, W., 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Air	Primary	0.0003 µg/L air	HPLC-UV	Zangmeister, W., 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Body fluids and tissues	Primary	Not required, not a toxic compound		

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

Component of residue definition: 500M07				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Plants (Wheat forage, straw, grain)	Primary	0.02 mg/kg	LC-MS/MS	Reinhard, K., Mackenroth, C. 1999, *DAR – Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Plants, processed (beer, brewer's yeast, brewing malt spent, grains and flocs, pod barley, malt sprouts)	Primary	0.02 mg/kg	LC-MS/MS	Reinhard, K.., Mackenroth, C., 1999, *DAR – Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
Plants (Wheat forage, straw, grain)	Primary	0.02 mg/kg	HPLC-UV	Abdel-Baky, S., Riley, M. E., 2000, *DAR – Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	

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

Component of residue definition: 500M35				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Cow milk, muscle, liver, kidney, fat, hen egg	Primary	0.01 mg/kg	LC-MS/MS	Tilting, N., Lehmann, W., 2000, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.05 mg/kg	GC-MS	

Table 5.2-6: Validated methods for the generation of pre-authorization data

Component of residue definition: 500M59, 500M60, 500M62, 500M76, 500M78				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Water	Primary	0.05 µg/L	LC-MS/MS	Zangmeister, W., 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A

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

Data provided on Annex I inclusion is sufficient for post-authorizations methods. All data is described in EU approved documents for :
-*DAR, Pyraclostrobin* - 1 August 2001

5.3.1 Analysis of the plant protection product (KCP 5.2)

For active substance Pyraclostrobin all presented methods are sufficient and no new methods are necessary. Please refer to KCP 5.1.2

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

Reference: KCP 5.2.1

Report: *Validation of an analytical method for the determination of residues of pyraclostrobin in wheat (whole plant)*, G. Paszek, 2021, Authority registration No: VAL/01/2021

Guideline(s): SANTE/2020/12830 Rev.1,

Deviations: NO

GLP: YES

Acceptability: YES

Materials and methods

The purpose of this study was to validate an analytical method for the determination of residues of pyraclostrobin in wheat (whole plant). Specimen extraction and determination of residues of pyraclostrobin was performed using the QuEChERS method. The specimens were prepared, extracted and analyzed following an ANALYTICAL PROCEDURE DPL-02 Determination of pesticide residues in food of plant origin using the QuEChERS technique and liquid chromatography by tandem detection of LC-MS/MS mass spectrometry – (version 02), that is available at the Test Facility.
The DPL-02 analytical procedure was written based on the following standards and scientific materials:

• EN 15662:2018 Foods of plant origin. Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE. Modular QuEChERS-method
Quantification was performed by use of LC-MS/MS detection system. The limit of detection (LOD) and quantification (LOQ) of the analytical method were subsequently 0.003 and 0.010 mg/kg for pyraclostrobin.

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

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

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

Matrix	Species	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high protein/high starch content (dry commodities)	Wheat (grain)	Pyraclostrobin	0.2 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
	Rye (grain)		0.2 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
	Barley (grain)		1.0 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
Muscle		Pyraclostrobin	0.05 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
Milk			0.01 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
Eggs			0.05 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
Fat			0.05 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
Liver, kidney			0.05 mg/kg	*COMMISSION REGULATION (EU) ~~2020/856~~ 2020/1633*
Soil (Ecotoxicology)		Pyraclostrobin	0.05 mg/kg	Default limit
Drinking water (Human toxicology)		Pyraclostrobin	0.1 µg/L	Default limit
Surface water (Ecotoxicology)		Pyraclostrobin	3.0 µg/L	NOEC of Daphnia magna as most sensitive species
Air		Pyraclostrobin	6 µg/m^3	AOEL: 0.02 mg/kg bw/d
Tissue (meat or liver)		Pyraclostrobin	0.1 mg/kg	Not classified as T / T+

Body fluids	Pyraclostrobin	0.05 mg/L	Not classified as T / T+

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

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

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

Component of residue definition: Pyraclostrobin and metabolite 500M07				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High water content (wheat forage)	Primary	0.02 mg/kg mg/kg	LC-MS/MS	Reinhard, K., Mackenroth, C. 1999, *DAR – Pyraclostrobin*
	ILV	-	-	-
	Confirmatory (if required)	N/A	N/A	N/A
	Primary	0.02 mg/kg	HPLC-UV	Abdel-Baky, S., Riley, M. E., 2000, *DAR - Pyraclostrobin*
	ILV	-	-	-
	Confirmatory (if required)	N/A	N/A	N/A
High protein/high starch content (wheat grain)	Primary	0.02 mg/kg	LC-MS/MS	Reinhard, K., Mackenroth, C., 1999, *DAR - Pyraclostrobin*
	ILV	0.02 mg/kg	LC-MS/MS	Perez. R., Perez, S., 2000, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A
	Primary	0.02 mg/kg	HPLC-UV	Abdel-Baky, S., Riley, M. E., 2000, *DAR - Pyraclostrobin*
	ILV	-	-	-
	Confirmatory (if required)	N/A	N/A	N/A

Table 5.3-3: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	Extraction efficiency was investigated in the context of metabolism study in potato that was presented in the DAR for the evaluation of Pyraclostrobin (Bross, M., Mackenroth, C., 1999).
Not required, because:	The extraction procedures was shown to be equivalent to the one from the metabolism study (Bross, M., Mackenroth, C., 1999). Please refer to *DAR – Pyraclostrobin*.

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 pyraclostrobin in animal matrices is given in the following tables. For the detailed evaluation of additional studies it is referred to Appendix 2.

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

Component of residue definition: Pyraclostrobin				
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	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
	ILV	0.01 mg/kg	HPLC-UV	Levsen, K, 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
Eggs	Primary	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
	ILV	-	-	-
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
Muscle	Primary	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
	ILV	0.05 mg/kg	HPLC-UV	Levsen, K, 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
Fat	Primary	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
	ILV	-	-	-
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
Kidney, liver	Primary	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*
	ILV	-	-	-
	Confirmatory (if required)	0.05 mg/kg	HPLC-UV	Kampke,-Thiel, K., 1999, *DAR - Pyraclostrobin*

Table 5.3-5: Statement on extraction efficiency

	Method for products of animal origin
Required, available from:	-
Not required, because:	A statement about the efficiency of the extraction procedure of animal was already peer reviewed.

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 pyraclostrobin in soil is given in the following tables. For the detailed evaluation of additional studies it is referred to Appendix 2.

Table 5.3-6: Validated methods for soil (if appropriate)

Component of residue definition: Pyraclostrobin			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.01 mg/kg	HPLC-UV	Ziegler, G., 1998, *DAR - Pyraclostrobin*
Confirmatory	0.01 mg/kg	LC-MS	Zangmeister, W., 1999, *DAR - Pyraclostrobin*

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 Pyraclostrobin and its metabolites in surface and drinking water is given in the following tables. For the detailed valuation of additional studies it is referred to Appendix 2.

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

Component of residue definition: Pyraclostrobin, 500M04, 500M59, 500M60, 500M62, 500M76, 500M78				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
Water	Primary	0.05 µg/L	LC-MS/MS	Zangmeister, W., 1999, *DAR - Pyraclostrobin*
	Confirmatory (if required)	N/A	N/A	N/A

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 pyraclostrobin in air is given in the following tables. For the detailed evaluation of additional studies please refer to Appendix 2.

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

Component of residue definition: Pyraclostrobin			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	0.0003 µg/L air	HPLC-UV	Zangmeister, W., 1999, *DAR - Pyraclostrobin*
Confirmatory	N/A	N/A	N/A

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

No methods required as Pyraclostrobin is not classified as toxic or highly toxic.

5.3.2.8 Other studies/ information

Not relevant.

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	Pomeroy, D.	2020	*Validation of the Methods of Determination of Pyraclostrobin in an Emulsifiable Concentrate Formulation, in Compliance with Good Laboratory Practise* DNA5716 David Norris Analytical Laboratories Ltd., Dartford, United Kingdom GLP- Yes Unpublished	N	Chemirol
KCP 5.2.1	Paszek, G.	2021	*Validation of an analytical method for the determination of residues of pyraclostrobin in wheat (whole plant)* VAL/01/2021 SGS Polska Sp. z o.o., Pszczyna, Poland GLP- Yes Unpublished	N	Chemirol
KCP 5.2.1	Niewelt, S, Wańczyk, K.	2021	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC in Northern France – 2021* DPL/37/2021, 21SGS39 SGS Poland GLP- Yes Unpublished	N	Chemirol
KCP 5.2.1	Niewelt, S, Wańczyk, K.	2021	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC - in Hungary – 2021* DPL/38/2021, 21SGS40 SGS Poland GLP- Yes Unpublished	N	Chemirol
KCP 5.2.1	Paszek, G., Wańczyk, K.	2021	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC - Germany – 2021* DPL/39/2021, 21SGS41	N	Chemirol

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
			SGS Poland GLP- Yes Unpublished		
KCP 5.2.1	Jędrusik, M. Wańczyk, K.	2021	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC in Poland – 2021* DPL/40/2021, 21SGS42 SGS Poland GLP- Yes Unpublished	N	Chemirol

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	Reinhard, K., Mackenroth, C.	1999	*Validation of BASF method no. 421/0 (Germany), D9808 (USA): Determination of BAS 500 F and its metabolite BF 500-3 in wheat, grape, peanut and orange matrices.* 35509 BASF Not GLP Unpublished	N	BASF
KCP 5.1.2/02	Reinhard, K., Mackenroth, C.	1999	*Validation of BASF method no. 453/0: Determination of BAS 500 F and its metabolite BF 500-3 in matrices / fractions of the processing of barley.* 35513 BASF Not GLP Unpublished	N	BASF

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 5.1.2/03 KCP 5.2	Abdel-Baky, S., Riley, M.	2000	*Validation of BASF analytical method D9904, Method for determination of BAS 500 F and its metabolite BF 500-3 residues in plant matrices using HPLC-UV.* 63770 BASF GLP Unpublished	N	BASF
KCP 5.1.2/04 KCP 5.2	Kampke-Thiel, K.	1999	*Validation of BASF method 439/0 for the determination of BAS 500 F (as parent compound) in matrices of animal origin.* 53018 BASF Not GLP Unpublished	N	BASF
KCP 5.1.2/05	Tilting, N., Lehmann, W.	2000	*Validation of analytical method 446 for the determination of BAS 500 F (reg. no. 304428) in sample material of animal origin.* 35636 BASF GLP Unpublished	N	BASF
KCP 5.1.2/06 KCP 5.2	Ziegler, G.	1998	*Validation of analytical method no. 409, Determination of BAS 500 F (parent) in soil.* 35646 BASF Not GLP Unpublished	N	BASF
KCP 5.1.2/07 KCP 5.2	Zangmeister, W.	1999	*Validation of analytical method no. 432, Determination of BAS 500 F, Reg. no. 340266, Reg. no. 369315 and Reg. no 364380 in soil.* 37275 BASF Not GLP Unpublished	N	BASF
KCP 5.1.2/08	Staab, G.	1998	*Validation of analytical method no. 415, Determination of BAS 500 F (parent) in tap and leachate water.*	N	BASF

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 5.2			35886 BASF Not GLP Unpublished		
KCP 5.1.2/09 KCP 5.2	Zangmeister, W.	1999	*Validation of analytical method 455: Determination of BAS 500 F, BF 500-11, BF 500-12, BF 500-13, BF 500-14 and BF 500-15 residues in water (tap water and surface water).* 35888 BASF Not GLP Unpublished	N	BASF
KCP 5.1.2/10 KCP 5.2	Zangmeister, W.	1999	*Validation of analytical method 447: Determination of BAS 500 F (Reg. no 304428) in air by HPLC/UV.* 35892 BASF Not GLP Unpublished	N	BASF
KCP 5.2	Perez. R., Perez, S.	2000	*Independent method validation of BASF method numbers D9808 (USA) and 421/0 (Germany) entitled "Method for determination of BAS 500 F and its metabolite BF 500-3 residues in plant matrices using LC/MS/MS".* 63832 BASF Not GLP Unpublished	N	BASF
KCP 5.2	Levsen, K.	1999	*Independent validation of BASF method 439/0 for the determination of BAS 500 F (as parent compound) in matrices of animal origin.* 15 G 99015 BASF Not GLP Unpublished	N	BASF

Appendix 2 Detailed evaluation of submitted analytical methods

A 2.1 Analytical methods for pyraclostrobin

A 2.1.1 Description of analytical methods for the determination of residues in plant matrices

zRMS comments	The study is accepted.

Reference:	KCP 5.2.1
Report	*Validation of an analytical method for the determination of residues of pyraclostrobin in wheat (whole plant)*, G. Paszek, 2021, Authority registration No: VAL/01/2021
Guideline(s):	SANTE/2020/12830 Rev.1,
Deviations:	NO
GLP:	YES

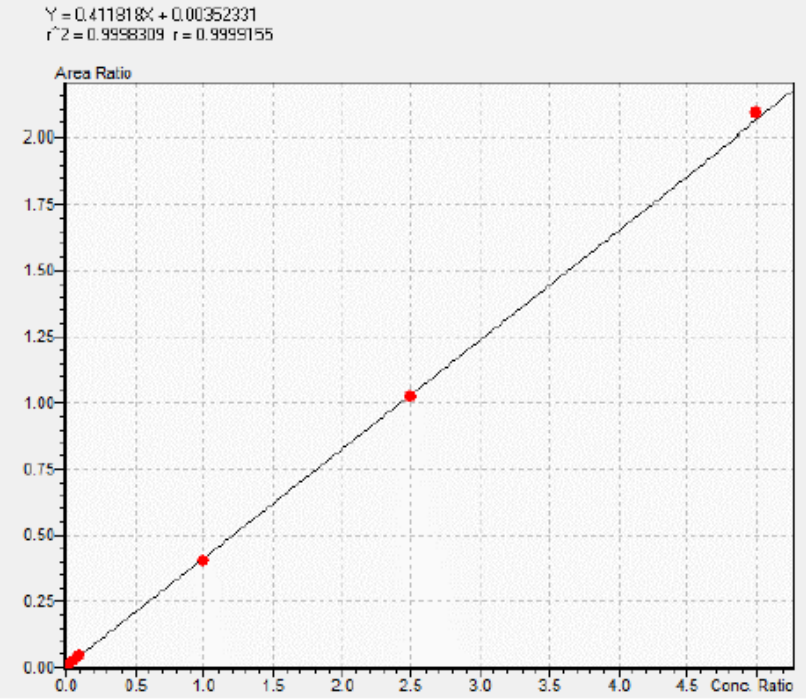
Materials and methods

The purpose of this study was to validate an analytical method for the determination of residues of pyraclostrobin in wheat (whole plant). Specimen extraction and determination of residues of pyraclostrobin was performed using the QuEChERS method. The specimens were prepared, extracted and analyzed following an ANALYTICAL PROCEDURE DPL-02 Determination of pesticide residues in food of plant origin using the QuEChERS technique and liquid chromatography by tandem detection of LC-MS/MS mass spectrometry – (version 02), that is available at the Test Facility.
The DPL-02 analytical procedure was written based on the following standards and scientific materials:
• EN 15662:2018 Foods of plant origin. Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE. Modular QuEChERS-method
Quantification was performed by use of LC-MS/MS detection system. The limit of detection (LOD) and quantification (LOQ) of the analytical method were subsequently 0.003 and 0.010 mg/kg for pyraclostrobin.

The following points were examined during the study:

Linearity

The linearity of the detector response was demonstrated by single determination of calibration standards at six (6) concentration levels ranging from 0.500 to 500 ppb. The coefficient of determination (R2) were determined.
Linear regression analysis with 1/x weighting was used to describe the detector response as a function of the calibration standard concentrations. For the least squares regression equations describing the detector response as a function of the standard calibration curve concentrations, the coefficients of determination (r) were greater than 0.990 for all of the calibration curve determinations during the method validation. The results indicate linearity of the detector response as a function of the standard concentration.



Sensitivity

The LOQ is the lowest validated fortification level for which an average recovery in the range of 70 – 120% and RSD ≤ 20 % is achieved.
For acetamiprid LOQ was successfully established at 0.010 mg/kg for wheat (whole plant).
LOD (limit of detection) was established at 0.003 mg/kg.

Accuracy

Recovery data was generated from five samples fortified at the limit of quantification (LOQ) and five samples fortified at the 10-fold higher concentration than the LOQ (10 x LOQ). Precision of the method was determined as the relative standard deviation (RSD) of recovery at each fortification level.
The mean recovery at each fortification level should be in the range of 70 – 120%. Wherever applicable (n ≥ 3), the relative standard deviation was determined and should be ≤ 20% for each level (RSD were determined only during validation process).
The recovery data was calculated according to equation:

$$R = \frac{C_R \cdot 100}{C_F}$$

Where:

R - recovery [%]
C_R - analyzed concentration of analyte in the fortified specimen [mg/kg]
C_F - nominal concentration of analyte in the fortified specimen [mg/kg]

The recovery values represent were obtained from calculations based on the exact raw data.
Relative Standard Deviation (RSD %) was calculated according to equation:

$$RSD = \frac{s}{x}$$

where:

s – standard deviation obtained from a series of results
x – average received from the result series

whole plant

Fortification level [mg/kg]	Obtained result [mg/kg]	Recovery [%]	Fortification level [mg/kg]	Obtained result [mg/kg]	Recovery [%]
0,0100	0,0098	97,9	0,100	0,095	95,4
	0,0097	97,2		0,093	93,4
	0,0096	96,2		0,097	96,6
	0,0096	96,3		0,094	94,1
	0,0098	98,5		0,099	99,4
Average	0,0097	97,2	Average	0,096	95,8
SD	0,0001	1,001	SD	0,0024	2,368
RSD [%]	1,03		RSD [%]	2,47	
Uncertainty [%]	5,9		Uncertainty [%]	9,8	

Matrix effects

Referring to the requirements of SANTE/2020/12830 Rev.1, 24 February 2021 (Chapter 3.2 Calibration) if the matrix effect exceeds 20% LOQ, the "matrix-matched" calibration should be introduced. This effect was not observed during validation, but the method of preparing working calibration standards based on blank sample was used.

Stability of fortification solutions and calibration standards

The stability of the analytes in the final extracts was proven by the corresponding procedural recovery samples, which were stored under the same conditions together with the extracts of the specimens for residue analysis. The recovery values for PK2 0.010 mg/kg and PK2 0.10 mg/kg (in the range of 70 – 120%) confirms the active substance stability during the analytical procedure. The total analytical procedure, from sample extraction till analysis, was performed and completed within 1 day (less than 24 h).

Summary of validation results

Parameter	Criterion of acceptance	Results obtained for transition 223.10>126.0
Specificity/selectivity	Fulfilled	
Linearity	$R^2 \geq 0.99$	Fulfilled
Polarization		positive
Quantification (target) ion	-	390.1>163.1
Qualification (ref) Ion(s)	-	390.1>194.1
Limit of quantification (LOQ)	-	0.010 mg/kg
Limit of detection (LOD)	-	0.003 mg/kg

zRMS comments	The analytical part of the study is accepted. Specimen extraction of pyraclostrobin was performed according to the multi-residue QuEChERS method. Determination was performed using LC-MS/MS. LOQ of the analytical method was 0.01 mg/kg in whole plant without root (wheat). Validation results are presented in the study VAL/01/2021 described above.

Study 1

Reference:	KCP 5.2.1
Report	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC in Northern France - 2021*, S. Niewelt, K. Wańczyk, DPL/37/2021, 21SGS39
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC Commission Regulation (EU) no 283/2013 setting out the data requirements for active substances, in accordance with Regulation (EC) no 1107/2009 Guidance Document on Pesticide Analytical Methods for Risk Assessment and Post-approval Control and Monitoring Purposes, SANTE/2020/12830 Rev.1, 24 February 2021
Deviations:	Yes
GLP:	Yes
Acceptability:	Yes

The objective of the study was the determination of degradation time (DT50) of pyraclostrobin in winter cereals (Raw Agricultural Commodity) after one application of CHR/F/PYRA 250 EC under field conditions under field conditions.

Materials and methods

8.1 TEST ITEM

Trade Name:	CHR/F/PYRA 250 EC
Name:	CHR/F/PYRA 250 EC (Pyraclostrobin 250 g/L)
Batch No.:	04/2020
Active substance (a. s.):	Pyraclostrobin
CAS Number:	175013-18-0
Formulation Name:	EC
Formulation Type:	Emulsion Concentrate
Main uses:	Fungicide
Actual density :	1,0637 g/cm^3 (from CoA)
Expiry date :	23/04/2022
Content of a. s.	nominal: 250,0 g/L
	analysed: 253,1 g/L
Certificate of Analysis dated:	04/08/2020

8.2 TEST SYSTEM

Crop	Winter wheat (*Triticum aestivum*)
Variety, planting date	See Table 2 – Test system information
Crop Group classification	Codex Alimentarius: GC 0654
RACs harvested	Whole plant without root

Field phase desription

One trial was established in Northern France. Trial consisted of one untreated plot U and one treated plot T.

Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.

One typical for fungicide applications of CHR/F/PYRA 250 EC were performed in trial with boom sprayer on the treated plot at the target dose rate of 1,0 l/ha. The reported dose rate actually was 0,991 l/ha.

The target spray volume was 100-400 litres per hectare according to Good Agricultural Practices. The reported spray volume was actually 247,6 l/ha.

Applications were performed at BBCH 29 (foliar).

The spray mixture volumes remaining after applications were measured and the volumes applied to the treated plot were calculated to verify delivery rates. The calculations and the delivery rates were verified by the Study Director.

Deviations to the target rates were all between ± 5% as requested in the study plan (actually it was -0,9 %.

To determinate degradation time 50, RAC specimens for analyses (whole plants without roots) were collected in intervals 0, 2,4,8,12,24,48,72,96,120,144 hours after application.

Quality control measures were taken to maintain specimen integrity and to avoid contamination at the trial sites.

RAC specimens were put in deep freezing conditions at a target temperature
of ≤ -18° C on the day of sampling, within 15 minutes after sampling If period was longer sample was stored on dry ice.

All specimens remained deep frozen during storage at the test site.

Deviations

There were two deviations to the study plan. Application A1 was done at BBCH 29 instead BBCH 25. Second deviation concern increase temperature under -18oC in freezer with retain samples in period after shipment of specimens for analyses. In Both cases there were no impact to the study.

Conclusions- Field phase

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).
Method of determination by LC-MS/MS fulfils the requirements as defined in EC Guidance document on residue analytical methods (SANTE/2020/12830 Rev.1) and is applicable as enforcement and data generation method for determination of pyraclostrobin in wheat after one application of CHR/F/PYRA 250 EC.

Specimen extraction and determination of residues of pyraclostrobin were performed according to the multi-residue QuEChERS method. Quantification was performed by use of LC-MS/MS detection. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg.
Residues of pyraclostrobin were not detected (<LOD) in any of the untreated samples.

Residues concentration detected in analysed field samples:

No	Timing	Study sample code	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	0 DBA	20SGS39-01-1	wheat (whole plant without root)	DPL/37/2021/01U	< LOD
2	0 DAA	20SGS39-01-2	wheat (whole plant without root)	DPL/37/2021/02T	16.10
3	2 HAA	20SGS39-01-3	wheat (whole plant without root)	DPL/37/2021/03T	15.53
4	4 HAA	20SGS39-01-4	wheat (whole plant without root)	DPL/37/2021/04T	14.62
5	8 HAA	20SGS39-01-5	wheat (whole plant without root)	DPL/37/2021/05T	11.95
6	12 HAA	20SGS39-01-6	wheat (whole plant without root)	DPL/37/2021/06T	10.89
7	24 HAA	20SGS39-01-7	wheat (whole plant without root)	DPL/37/2021/07T	10.75
8	48 HAA	20SGS39-01-8	wheat (whole plant without root)	DPL/37/2021/08T	10.64
9	72 HAA	20SGS39-01-9	wheat (whole plant without root)	DPL/37/2021/09T	8.33
10	96 HAA	20SGS39-01-10	wheat (whole plant without root)	DPL/37/2021/10T	5.59
11	120 HAA	20SGS39-01-11	wheat (whole plant without root)	DPL/37/2021/11T	5.68
12	144 HAA	20SGS39-01-12	wheat (whole plant without root)	DPL/37/2021/12U	<LOD
13	144 HAA	20SGS39-01-13	wheat (whole plant without root)	DPL/37/2021/13T	6.28

Study number	Trial number	DT_{50} [h]	DT_{50} [days]	Error[%]
21SGS39	21SGS39-01	89.1	3.71	9.91

Extraction

2 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of deionized water and 10 mL of acetonitrile was added. Next to the sample was added 20 µL of internal standard solution (1.3), and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 10 min for phase separation. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile.

Fortification and control samples

For analytical sequence one sample blank matrix and two procedural recoveries at the level of LOQ and two at the level 10 x LOQ were prepared together with the study samples.

Table 2. Preparation of fortification and control samples

Fortification level	Amount of added standard solution [1.1] [µL]	Amount of added standard solution [1.2] [µL]	Amount of added internal standard solution [1.3] [µL]
Matrix blank	-	-	20.0
PK 0.010 mg/kg (LOQ)	-	20.0	20.0
PK 0.10 mg/kg (10 x LOQ)	20.0	-	20.0

Extraction of all field samples (treated and untreated), as well as control and fortified samples was performed on 21.04.2021 and after that the samples were directly employed for LC-MS/MS analysis, that was started on the same day.

Blank and fortification samples

For each analytical set the method's applicability in terms of accuracy was assessed by fortification of untreated test portions of the respective matrix and subsequent determination of the procedural recoveries upon applying the test method.

Procedural recoveries were handled and stored in the same way and for the same time period as the samples extracts that were generated within the same analytical set. Two of the fortification samples (LOQ and 10 x LOQ) were run at the very end of analytical sequence in order to ensure the active substance stability during the analytical method workflow.

Sample blank matrix, two procedural recoveries at the level of LOQ and two at the level of 10 x LOQ per analytical set of respective matrix were analyzed during sequence.

The following results for matrix blank and fortified samples were obtained during analysis of untreated and treated samples 21.04.2021:

Table 10. Quality control samples

Sample Name	Result [mg/kg]	Recovery [%]
DPL-37-2021, matrix blank.lcd	< LOD	-
DPL-37-2021, PK1 0,010 mg-kg.lcd	0.0090	89.5
DPL-37-2021, PK1 0,10 mg-kg.lcd	0.091	90.7
DPL-37-2021, PK2 0,010 mg-kg.lcd	0.0089	89.0
DPL-37-2021, PK2 0,10 mg-kg.lcd	0.083	82.8

LOD = 0.003 mg/kg, LOQ = 0.01 mg/kg

All recovery values at fortification levels of 0.010 mg/g and 0.10 mg/kg comply with the standard acceptance criteria of the guidance documents to SANTE/2020/12830 Rev.1.

The stability of the analytes in the final extracts was proven by the corresponding procedural recovery samples, which were stored under the same conditions together with the extracts of the specimens for residue analysis. The recovery values for PK2 0.010 mg/kg and PK2 0.10 mg/kg (in the range of 70 – 120%) confirms the active substance stability during the analytical procedure. The duration of the extraction process was about 3 hours, the duration of the chromatographic analysis was 560 min (9.3 h). The

total analytical procedure, from sample extraction till analysis, was performed and completed within 1 day (less than 13 h).

Conclusions- Analytical phase
The study was conducted using analytical method validated according to SANTE/2020/12830 Rev.1 guideline.
The limit of detection and quantification of the method was established at 0.003 and 0.010 mg/kg for wheat plant, respectively.
The performance of the method during the analytical study complies with SANTE/2020/12830 Rev.1 criteria (accuracy in the range 70 – 120%).
There were no interfering signals at retention time of analyzed compound in examined control matrix.

zRMS comments	The analytical part of the study is accepted. Specimen extraction of pyraclostrobin was performed according to the multi-residue QuEChERS method. Determination was performed using LC-MS/MS. LOQ of the analytical method was 0.01 mg/kg in whole plant without root (wheat). Validation results are presented in the study VAL/01/2021 described above.

Study 2

Reference:	KCP 5.2.1
Report	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC - in Hungary - 2021*, S. Niewelt, K. Wańczyk, DPL/38/2021, 21SGS40
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC Commission Regulation (EU) no 283/2013 setting out the data requirements for active substances, in accordance with Regulation (EC) no 1107/2009 Guidance Document on Pesticide Analytical Methods for Risk Assessment and Post-approval Control and Monitoring Purposes, SANTE/2020/12830 Rev.1, 24 February 2021
Deviations:	No
GLP:	Yes
Acceptability:	Yes

The objective of the study was the determination of degradation time (DT50) of pyraclostrobin in winter cereals (Raw Agricultural Commodity) after one application of CHR/F/PYRA 250 EC under field conditions under field conditions.

Materials and methods

8.1 TEST ITEM

Trade Name:	CHR/F/PYRA 250 EC
Name:	CHR/F/PYRA 250 EC (Pyraclostrobin 250 g/L)
Batch No.:	04/2020
Active substance (a. s.):	Pyraclostrobin
CAS Number:	175013-18-0
Formulation Name:	EC
Formulation Type:	Emulsion Concentrate
Main uses:	Fungicide
Actual density :	1,0637 g/cm^3 (from CoA)
Expiry date :	23/04/2022
Content of a. s.	nominal: 250,0 g/L
	analysed: 253,1 g/L
Certificate of Analysis dated:	04/08/2020

8.2 TEST SYSTEM

Crop	Winter wheat (*Triticum aestivum*)
Variety, planting date	See Table 2 – Test system information
Crop Group classification	Codex Alimentarius: GC 0654
RACs harvested	Whole plant without root

Field phase desription

One trial was established in Hungary. Trial consisted of one untreated plot U and one treated plot T divided in 2 subplots (subplot 1 and subplot 2). Sampling was done from both subplots randomly.
Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.
One typical for fungicide applications of CHR/F/PYRA 250 EC were performed in trial with boom sprayer on the treated plot at the target dose rate of 1,0 l/ha. The reported dose rate actually was 0,985 l/ha (subplot 1) and 0,982 l/ha (subplot 2).
The target spray volume was 100-400 litres per hectare according to Good Agricultural Practices. The reported spray volume was actually 216,7 l/ha and 216,0 l/ha.
Applications were performed at BBCH 25 (foliar).
The spray mixture volumes remaining after applications were measured and the volumes applied to the treated plot were calculated to verify delivery rates. The calculations and the delivery rates were verified by the Study Director.
Deviations to the target rates were all between ± 5% as requested in the study plan (actually it was -1,5% and -1,8 %).
To determinate degradation time 50, RAC specimens for analyses (whole plants without roots) were collected in intervals 0, 2,4,8,12,24,48,72,96,120,144 hours after application.
Quality control measures were taken to maintain specimen integrity and to avoid contamination at the trial sites.
RAC specimens were put in deep freezing conditions at a target temperature
of ≤ -18° C on the day of sampling, within 15 minutes after sampling If period was longer sample was stored on dry ice.
All specimens remained deep frozen during storage at the test site.

Conclusions- Field phase

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).
Method of determination by LC-MS/MS fulfils the requirements as defined in EC Guidance document on

residue analytical methods (SANTE/2020/12830 Rev.1) and is applicable as enforcement and data generation method for determination of pyraclostrobin in Wheat after one application of CHR/F/PYRA 250 EC.

Specimen extraction and determination of residues of pyraclostrobin were performed according to the multi-residue QuEChERS method. Quantification was performed by use of LC-MS/MS detection. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg.

Residues of pyraclostrobin were not detected (<LOD) in any of the untreated samples.

Residues concentration detected in analysed field samples:

No	Timing	Study sample code	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	0 DBA	20SGS40-01-1	wheat (whole plant without root)	DPL/38/2021/01U	< LOD
2	0 DAA	20SGS40-01-2	wheat (whole plant without root)	DPL/38/2021/02T	16.45
3	2 HAA	20SGS40-01-3	wheat (whole plant without root)	DPL/38/2021/03T	16.25
4	4 HAA	20SGS40-01-4	wheat (whole plant without root)	DPL/38/2021/04T	16.02
5	8 HAA	20SGS40-01-5	wheat (whole plant without root)	DPL/38/2021/05T	15.93
6	12 HAA	20SGS40-01-6	wheat (whole plant without root)	DPL/38/2021/06T	15.71
7	24 HAA	20SGS40-01-7	wheat (whole plant without root)	DPL/38/2021/07T	14.76
8	48 HAA	20SGS40-01-8	wheat (whole plant without root)	DPL/38/2021/08T	11.68
9	72 HAA	20SGS40-01-9	wheat (whole plant without root)	DPL/38/2021/09T	7.08
10	96 HAA	20SGS40-01-10	wheat (whole plant without root)	DPL/38/2021/10T	3.97
11	120 HAA	20SGS40-01-11	wheat (whole plant without root)	DPL/38/2021/11T	3.67
12	144 HAA	20SGS40-01-12	wheat (whole plant without root)	DPL/38/2021/12U	<LOD
13	144 HAA	20SGS40-01-13	wheat (whole plant without root)	DPL/38/2021/13T	3.47

Study number	Trial number	DT_{50} [h]	DT_{50} [days]	Error[%]
21SGS40	21SGS40-01	59.8	2.49	7.19

Extraction

2 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of deionized water and 10 mL of acetonitrile was added. Next to the sample was added 20 µL of internal standard solution (1.3), and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 10 min for phase separation. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile.

Fortification and control samples

For analytical sequence one sample blank matrix and two procedural recoveries at the level of LOQ and two at the level 10 x LOQ were prepared together with the study samples.

Table 2. Preparation of fortification and control samples

Fortification level	Amount of added standard solution [1.1] [µL]	Amount of added standard solution [1.2] [µL]	Amount of added internal standard solution [1.3] [µL]
Matrix blank	-	-	20.0
PK 0.010 mg/kg (LOQ)	-	20.0	20.0
PK 0.10 mg/kg (10 x LOQ)	20.0	-	20.0

Extraction of all field samples (treated and untreated), as well as control and fortified samples was performed on 22.04.2021 and after that the samples were directly employed for LC-MS/MS analysis, that was started on the same day.

Blank and fortification samples

For each analytical set the method's applicability in terms of accuracy was assessed by fortification of untreated test portions of the respective matrix and subsequent determination of the procedural recoveries upon applying the test method.

Procedural recoveries were handled and stored in the same way and for the same time period as the samples extracts that were generated within the same analytical set. Two of the fortification samples (LOQ and 10 x LOQ) were run at the very end of analytical sequence in order to ensure the active substance stability during the analytical method workflow.

Sample blank matrix, two procedural recoveries at the level of LOQ and two at the level of 10 x LOQ per analytical set of respective matrix were analyzed during sequence.

The following results for matrix blank and fortified samples were obtained during analysis of untreated and treated samples 22.04.2021:

Table 10. Quality control samples

Sample Name	Result [mg/kg]	Recovery [%]
DPL-38-2021, matrix blank.lcd	< LOD	-
DPL-38-2021, PK1 0,010 mg-kg.lcd	0.011	106.1
DPL-38-2021, PK1 0,10 mg-kg.lcd	0.098	98.2
DPL-38-2021, PK2 0,010 mg-kg.lcd	0.012	117.7
DPL-38-2021, PK2 0,10 mg-kg.lcd	0.10	103.1

LOD = 0.003 mg/kg, LOQ = 0.01 mg/kg

All recovery values at fortification levels of 0.010 mg/g and 0.10 mg/kg comply with the standard acceptance criteria of the guidance documents to SANTE/2020/12830 Rev.1.

The stability of the analytes in the final extracts was proven by the corresponding procedural recovery samples, which were stored under the same conditions together with the extracts of the specimens for residue analysis. The recovery values for PK2 0.010 mg/kg and PK2 0.10 mg/kg (in the range of 70 – 120%) confirms the active substance stability during the analytical procedure. The duration of the extraction process was about 3 hours, the duration of the chromatographic analysis was 518 min (8.6 h). The total analytical procedure, from sample extraction till analysis, was performed and completed within 1 day (less than 12 h).

Extract stability is not considered to be an issue, since working standard that were used for quantification were always prepared on the same day as the work up of the specimen for residue analysis took place.

Conclusions- Analytical phase
The study was conducted using analytical method validated according to SANTE/2020/12830 Rev.1 guideline.
The limit of detection and quantification of the method was established at 0.003 and 0.010 mg/kg for wheat plant, respectively.
The performance of the method during the analytical study complies with SANTE/2020/12830 Rev.1 criteria (accuracy in the range 70 – 120%).
There were no interfering signals at retention time of analyzed compound in examined control matrix.

zRMS comments	The analytical part of the study is accepted. Specimen extraction of pyraclostrobin was performed according to the multi-residue QuEChERS method. Determination was performed using LC-MS/MS. LOQ of the analytical method was 0.01 mg/kg in whole plant without root (wheat). Validation results are presented in the study VAL/01/2021 described above.

Study 3

Reference:	KCP 5.2.1
Report	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC - Germany – 2021,* G. Paszek, K. Wańczyk, DPL/39/2021, 21SGS41
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC Commission Regulation (EU) no 283/2013 setting out the data requirements for active substances, in accordance with Regulation (EC) no 1107/2009 Guidance Document on Pesticide Analytical Methods for Risk Assessment and Post-approval Control and Monitoring Purposes, SANTE/2020/12830 Rev.1, 24 February 2021
Deviations:	No
GLP:	Yes
Acceptability:	Yes

The objective of the study was the determination of degradation time (DT50) of pyraclostrobin in winter cereals (Raw Agricultural Commodity) after one application of CHR/F/PYRA 250 EC under field conditions under field conditions.

Materials and methods

8.1 TEST ITEM

Trade Name:	CHR/F/PYRA 250 EC
Name:	CHR/F/PYRA 250 EC (Pyraclostrobin 250 g/L)
Batch No.:	04/2020
Active substance (a. s.):	Pyraclostrobin
CAS Number:	175013-18-0
Formulation Name:	EC
Formulation Type:	Emulsion Concentrate
Main uses:	Fungicide
Actual density :	1,0637 g/cm^3 (from CoA)
Expiry date :	23/04/2022
Content of a. s.	nominal: 250,0 g/L
	analysed: 253,1 g/L
Certificate of Analysis dated:	04/08/2020

8.2 TEST SYSTEM

Crop	Winter wheat (*Triticum aestivum*)
Variety, planting date	See Table 2 – Test system information
Crop Group classification	Codex Alimentarius: GC 0654
RACs harvested	Whole plant without root

Field phase desription

One trial was established in Germany. Trial consisted of one untreated plot U and one treated plot T.
Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.
One typical for fungicide applications of CHR/F/PYRA 250 EC were performed in trial with boom sprayer on the treated plot at the target dose rate of 1,0 l/ha. The reported dose rate actually was 0,958 l/ha.
The target spray volume was 100-400 litres per hectare according to Good Agricultural Practices. The reported spray volume was actually 191,67 l/ha.
Applications were performed at BBCH 25 (foliar).
The spray mixture volumes remaining after applications were measured and the volumes applied to the treated plot were calculated to verify delivery rates. The calculations and the delivery rates were verified by the Study Director.
Deviations to the target rates were all between ± 5% as requested in the study plan (actually it was -4,2%).
To determinate degradation time 50, RAC specimens for analyses (whole plants without roots) were collected in intervals 0, 2,4,8,12,24,48,72,96,120,144 hours after application.
Quality control measures were taken to maintain specimen integrity and to avoid contamination at the trial sites.
RAC specimens were put in deep freezing conditions at a target temperature
of ≤ -18° C on the day of sampling, within 15 minutes after sampling If period was longer sample was stored on dry ice.
All specimens remained deep frozen during storage at the test site.

Conclusions- Field phase

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).

Method of determination by LC-MS/MS fulfils the requirements as defined in EC Guidance document on residue analytical methods (SANTE/2020/12830 Rev.1) and is applicable as enforcement and data generation method for determination of pyraclostrobin in Wheat after one application of CHR/F/PYRA 250 EC.

Specimen extraction and determination of residues of pyraclostrobin were performed according to the multi-residue QuEChERS method. Quantification was performed by use of LC-MS/MS detection. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg.

Residues of pyraclostrobin were not detected (<LOD) in any of the untreated samples.

Residues concentration detected in analysed field samples:

No	Timing	Study sample code	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	0 DBA	20SGS41-01-1	wheat (whole plant without root)	DPL/39/2021/01U	< LOD
2	0 DAA	20SGS41-01-2	wheat (whole plant without root)	DPL/39/2021/02T	19,8
3	2 HAA	20SGS41-01-3	wheat (whole plant without root)	DPL/39/2021/03T	28,1
4	4 HAA	20SGS41-01-4	wheat (whole plant without root)	DPL/39/2021/04T	23,9
5	8 HAA	20SGS41-01-5	wheat (whole plant without root)	DPL/39/2021/05T	23,7
6	12 HAA	20SGS41-01-6	wheat (whole plant without root)	DPL/39/2021/06T	18,0
7	24 HAA	20SGS41-01-7	wheat (whole plant without root)	DPL/39/2021/07T	15,2
8	48 HAA	20SGS41-01-8	wheat (whole plant without root)	DPL/39/2021/08T	10,4
9	72 HAA	20SGS41-01-9	wheat (whole plant without root)	DPL/39/2021/09T	10,1
10	96 HAA	20SGS41-01-10	wheat (whole plant without root)	DPL/39/2021/10T	5,27
11	120 HAA	20SGS41-01-11	wheat (whole plant without root)	DPL/39/2021/11T	4,82
12	144 HAA	20SGS41-01-12	wheat (whole plant without root)	DPL/39/2021/12U	<LOD
13	144 HAA	20SGS41-01-13	wheat (whole plant without root)	DPL/39/2021/13T	2,41

Study number	Trial number	DT_{50} [h]	DT_{50} [days]	Error[%]
21SGS41	21SGS41-01	45.4	1.90	12.5

Extraction

2 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of deionized water and 10 mL of acetonitrile was added. Next to the sample was added 20 µL of internal standard solution (1.3), and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 10 min for phase separation. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetoni-

trile.

Fortification and control samples

For analytical sequence one sample blank matrix and two procedural recoveries at the level of LOQ and two at the level 10 x LOQ were prepared together with the study samples.

Table 2. Preparation of fortification and control samples

Fortification level	Amount of added standard solution [1.1] [µL]	Amount of added standard solution [1.2] [µL]	Amount of added internal standard solution [1.3] [µL]
Matrix blank	-	-	20.0
PK 0.010 mg/kg (LOQ)	-	20.0	20.0
PK 0.10 mg/kg (10 x LOQ)	20.0	-	20.0

Extraction of all field samples (treated and untreated), as well as control and fortified samples was performed on 22.04.2021 and after that the samples were directly employed for LC-MS/MS analysis, that was started on the same day.

Blank and fortification samples

For each analytical set the method's applicability in terms of accuracy was assessed by fortification of untreated test portions of the respective matrix and subsequent determination of the procedural recoveries upon applying the test method.

Procedural recoveries were handled and stored in the same way and for the same time period as the samples extracts that were generated within the same analytical set. Two of the fortification samples (LOQ and 10 x LOQ) were run at the very end of analytical sequence in order to ensure the active substance stability during the analytical method workflow.

Sample blank matrix, two procedural recoveries at the level of LOQ and two at the level of 10 x LOQ per analytical set of respective matrix were analyzed during sequence.

The following results for matrix blank and fortified samples were obtained during analysis of untreated and treated samples 23.04.2021:

Table 10. Quality control samples

Sample Name	Result [mg/kg]	Recovery [%]
p matrix blank.lcd	< LOD	-
p PK1 0,010 mg-kg.lcd	0,0079	79,1
p PK1 0,10 mg-kg.lcd	0,088	87,6
p' matrix blank.lcd	< LOD	-
p PK2 0,010 mg-kg.lcd	0,0086	86,2
p PK2 0,10 mg-kg.lcd	0,086	86,0

LOD = 0.003 mg/kg, LOQ = 0.01 mg/kg

All recovery values at fortification levels of 0.010 mg/g and 0.10 mg/kg comply with the standard acceptance criteria of the guidance document SANTE/2020/12830, Rev.1.

The stability of the analytes in the final extracts was proven by the corresponding procedural recovery samples, which were stored under the same conditions together with the extracts of the specimens for residue analysis. The recovery values for PK2 0.01 mg/kg and PK2 0.10 mg/kg (in the range of 70 –

120%) confirms the active substance stability during the analytical procedure. The duration of the extraction process was about 3 hours, the duration of the chromatographic analysis was 560 minutes (9.3 h). The total analytical procedure, from sample extraction till analysis, was performed and completed within 1 day (less than 13 h).
Extract stability is not considered to be an issue, since working standard that were used for quantification were always prepared on the same day as the work up of the specimen for residue analysis took place.

Conclusions- Analytical phase
The study was conducted using analytical method validated according to SANTE/2020/12830 Rev.1 guideline.
The limit of detection and quantification of the method was established at 0.003 and 0.010 mg/kg for wheat plant, respectively.
The performance of the method during the analytical study complies with SANTE/2020/12830 Rev.1 criteria (accuracy in the range 70 – 120%).
There were no interfering signals at retention time of analyzed compound in examined control matrix.

zRMS comments	The analytical part of the study is accepted. Specimen extraction of pyraclostrobin was performed according to the multi-residue QuEChERS method. Determination was performed using LC-MS/MS. LOQ of the analytical method was 0.01 mg/kg in whole plant without root (wheat). Validation results are presented in the study VAL/01/2021 described above.

Study 4

Reference:	KCP 5.2.1
Report	*Magnitude of residue and degradation time (DT50) of pyraclostrobin in winter wheat (Raw Agricultural Commodity) after one spray application of CHR/F/PYRA 250 EC in Poland - 2021*, M. Jędrusik, K. Wańczyk, DPL/40/2021, 21SGS42
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC Commission Regulation (EU) no 283/2013 setting out the data requirements for active substances, in accordance with Regulation (EC) no 1107/2009 Guidance Document on Pesticide Analytical Methods for Risk Assessment and Post-approval Control and Monitoring Purposes, SANTE/2020/12830 Rev.1, 24 February 2021
Deviations:	No
GLP:	Yes
Acceptability:	Yes

The objective of the study was the determination of degradation time (DT50) of pyraclostrobin in winter cereals (Raw Agricultural Commodity) after one application of CHR/F/PYRA 250 EC under field conditions under field conditions.

Materials and methods

8.1 TEST ITEM

Trade Name:	CHR/F/PYRA 250 EC
Name:	CHR/F/PYRA 250 EC (Pyraclostrobin 250 g/L)
Batch No.:	04/2020
Active substance (a. s.):	Pyraclostrobin
CAS Number:	175013-18-0
Formulation Name:	EC
Formulation Type:	Emulsion Concentrate
Main uses:	Fungicide
Actual density :	1,0637 g/cm^3 (from CoA)
Expiry date :	23/04/2022
Content of a. s.	nominal: 250,0 g/L
	analysed: 253,1 g/L
Certificate of Analysis dated:	04/08/2020

8.2 TEST SYSTEM

Crop	Winter wheat (*Triticum aestivum*)
Variety, planting date	See Table 2 – Test system information
Crop Group classification	Codex Alimentarius: GC 0654
RACs harvested	Whole plant without root

Field phase desription

One trial was established in Poland. Trial consisted of one untreated plot U and one treated plot T divided in 2 subplots (subplot 1 and subplot 2). Sampling was done from both subplots randomly.
Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.
One typical for fungicide applications of CHR/F/PYRA 250 EC were performed in trial with boom sprayer on the treated plot at the target dose rate of 1,0 l/ha. The reported dose rate actually was 0,999 l/ha The target spray volume was 100-400 litres per hectare according to Good Agricultural Practices. The reported spray volume was actually 299,7 l/ha.
Applications were performed at BBCH 25 (foliar).
The spray mixture volumes remaining after applications were measured and the volumes applied to the treated plot were calculated to verify delivery rates. The calculations and the delivery rates were verified by the Study Director.
Deviations to the target rates were all between ± 5% as requested in the study plan (actually it was -0,1 %
To determinate degration time 50, RAC specimens for analyses (whole plants without roots) were collected in intervals 0, 2,4,8,12,24,48,72,96,120,144 hours after application.
Quality control measures were taken to maintain specimen integrity and to avoid contamination at the trial sites.
Sampling dates and weights of collected specimens are presented in Table 15 – Sampling procedures and shipment of RAC specimens.
RAC specimens were put in deep freezing conditions at a target temperature
of ≤ -18° C on the day of sampling, within 15 minutes after sampling If period was longer sample was stored on dry ice.
All specimens remained deep frozen during storage at the test site.

Conclusions- Field phase

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can there-

fore be considered as representative of the crop and of the application timing(s) and rate(s).
Method of determination by LC-MS/MS fulfils the requirements as defined in EC Guidance document on residue analytical methods (SANTE/2020/12830 Rev.1) and is applicable as enforcement and data generation method for determination of pyraclostrobin in Wheat after one application of CHR/F/PYRA 250 EC.

Specimen extraction and determination of residues of pyraclostrobin were performed according to the multi-residue QuEChERS method. Quantification was performed by use of LC-MS/MS detection. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg.

Residues of pyraclostrobin were not detected (<LOD) in any of the untreated samples.

Residues concentration detected in analysed field samples:

No	Timing	Study sample code	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	0 DBA	20SGS42-01-1	wheat (whole plant without root)	DPL/40/2021/01U	< LOD
2	0 DAA	20SGS42-01-2	wheat (whole plant without root)	DPL/40/2021/02T	14.43
3	2 HAA	20SGS42-01-3	wheat (whole plant without root)	DPL/40/2021/03T	13.49
4	4 HAA	20SGS42-01-4	wheat (whole plant without root)	DPL/40/2021/04T	14.69
5	8 HAA	20SGS42-01-5	wheat (whole plant without root)	DPL/40/2021/05T	12.23
6	12 HAA	20SGS42-01-6	wheat (whole plant without root)	DPL/40/2021/06T	12.16
7	24 HAA	20SGS42-01-7	wheat (whole plant without root)	DPL/40/2021/07T	11.31
8	48 HAA	20SGS42-01-8	wheat (whole plant without root)	DPL/40/2021/08T	10.50
9	72 HAA	20SGS42-01-9	wheat (whole plant without root)	DPL/40/2021/09T	6.72
10	96 HAA	20SGS42-01-10	wheat (whole plant without root)	DPL/40/2021/10T	6.10
11	120 HAA	20SGS42-01-11	wheat (whole plant without root)	DPL/40/2021/11T	5.40
12	144 HAA	20SGS42-01-12	wheat (whole plant without root)	DPL/40/2021/12U	<LOD
13	144 HAA	20SGS42-01-13	wheat (whole plant without root)	DPL/40/2021/13T	3.46

Study number	Trial number	DT_{50} [h]	DT_{50} [days]	Error[%]
21SGS42	21SGS42-01	78.7	3.28	5.52

Extraction
2 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of deionized water and 10 mL of acetonitrile was added. Next to the sample was added 20 µL of internal standard solution (1.3), and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 10 min for phase separation. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile.

Fortification and control samples

For analytical sequence one sample blank matrix and two procedural recoveries at the level of LOQ and two at the level 10 x LOQ were prepared together with the study samples.

Table 2. Preparation of fortification and control samples

Fortification level	Amount of added standard solution [1.1] [µL]	Amount of added standard solution [1.2] [µL]	Amount of added internal standard solution [1.3] [µL]
Matrix blank	-	-	20.0
PK 0.010 mg/kg (LOQ)	-	20.0	20.0
PK 0.10 mg/kg (10 x LOQ)	20.0	-	20.0

Extraction of all field samples (treated and untreated), as well as control and fortified samples was performed on 26.04.2021 and after that the samples were directly employed for LC-MS/MS analysis, that was started on the same day.

Blank and fortification samples

For each analytical set the method's applicability in terms of accuracy was assessed by fortification of untreated test portions of the respective matrix and subsequent determination of the procedural recoveries upon applying the test method.

Procedural recoveries were handled and stored in the same way and for the same time period as the samples extracts that were generated within the same analytical set. Two of the fortification samples (LOQ and 10 x LOQ) were run at the very end of analytical sequence in order to ensure the active substance stability during the analytical method workflow.

Two sample blank matrix, two procedural recoveries at the level of LOQ and two at the level of 10 x LOQ per analytical set of respective matrix were analyzed during sequence.

The following results for matrix blank and fortified samples were obtained during analysis of untreated and treated samples 26.04.2021:

Table 10. Quality control samples

Sample Name	Result [mg/kg]	Recovery [%]
p matrix blank.lcd	< LOD	-
p PK1 0,010 mg-kg.lcd	0.011	109.3
p PK1 0,10 mg-kg.lcd	0.10	101.6
p' matrix blank.lcd	< LOD	-
p PK2 0,010 mg-kg.lcd	0.010	98.1
p PK2 0,10 mg-kg.lcd	0.091	90.7

LOD = 0.003 mg/kg, LOQ = 0.01 mg/kg

All recovery values at fortification levels of 0.010 mg/g and 0.10 mg/kg comply with the standard acceptance criteria of the guidance document SANTE/2020/12830, Rev.1.

The stability of the analytes in the final extracts was proven by the corresponding procedural recovery samples, which were stored under the same conditions together with the extracts of the specimens for residue analysis. The recovery values for PK2 0.01 mg/kg and PK2 0.10 mg/kg (in the range of 70 – 120%) confirms the active substance stability during the analytical procedure. The duration of the extraction process was about 3 hours, the duration of the chromatographic analysis was 560 minutes (9.3 h).

The total analytical procedure, from sample extraction till analysis, was performed and completed within 1 day (less than 13 h).
Extract stability is not considered to be an issue, since working standard that were used for quantification were always prepared on the same day as the work up of the specimen for residue analysis took place.

Conclusions- Analytical phase
The study was conducted using analytical method validated according to SANTE/2020/12830 Rev.1 guideline.
The limit of detection and quantification of the method was established at 0.003 and 0.010 mg/kg for wheat plant, respectively.
The performance of the method during the analytical study complies with SANTE/2020/12830 Rev.1 criteria (accuracy in the range 70 – 120%).
There were no interfering signals at retention time of analyzed compound in examined control matrix.

Summary of the field residue trials

1	2	3	4			5	6	7	8	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Timing	Remarks
			kg a.i./ha	Spray volume applied (l/ha)	l prod./ha						
	(a)	(b)				(c)		(a)		(d)	(e)
21SGS39 – Northern France, Bour-gogne	Winter wheat/Nemo	1) 06/11/2020 2) - 3) -	0.248	247.6	0.991	22/03/2021	BBCH 29	Whole plants	16.10 15.53 14.62 11.95 10.89 10.75 10.64 8.33 5.59 5.68 6.28	0 DAA 2 HAA 4 HAA 8 HAA 12 HAA 24 HAA 48 HAA 72 HAA 96 HAA 120 HAA 144 HAA	DT_{50}= 3.71 days
21SGS40 – Hungary, Monok	Winter wheat/ MU Ménròt	1) 22/09/2020 2) - 3) -	0.246	216	0.985	30/03/2021	BBCH 25	Whole plants	16.45 16.25 16.02 15.93 15.71 14.76 11.68 7.08 3.97 3.67 3.47	0 DAA 2 HAA 4 HAA 8 HAA 12 HAA 24 HAA 48 HAA 72 HAA 96 HAA 120 HAA 144 HAA	DT_{50}= 2.49 days

21SGS41 – Germany, Fahrdorf	Winter wheat/ RGT Reform	1) 15/09/2020 2) - 3) -	0.240	210	0.958	25/03/2021	BBCH 25	Whole plants	19.8 28.1 33.9 23.7 18.0 15.2 10.4 10.1 5.27 4.82 2.41	0 DAA 2 HAA 4 HAA 8 HAA 12 HAA 24 HAA 48 HAA 72 HAA 96 HAA 120 HAA 144 HAA	DT_{50}= 1.90 days
21SGS42– Poland, Cerekwica	Winter wheat/ Bataja	1) 23.09.2020 2) - 3) -	0.250	299.7	0.999	30/03/2021	BBCH 25	Whole plants	14.43 13.49 14.69 12.23 12.16 11.31 10.50 6.72 6.10 5.40 3.46	0 DAA 2 HAA 4 HAA 8 HAA 12 HAA 24 HAA 48 HAA 72 HAA 96 HAA 120 HAA 144 HAA	DT_{50}= 3.28 days