Analytical method for tetraniliprole (BCS-CL73507) and its transformation products, BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056, in soil and sediment

Reports:	ECM 1: EPA MRID No.: 50170146 (Appendix 6, pp. 144-157). Freitag, Th. 2015. Amendment No. 1 to Final Report No: MR-13/100 – Analytical method 01373 for the determination of BCS-CL73507 and the metabolites BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in soil and sediment by HPLC-MS/MS. Report prepared and sponsored by Bayer CropScience AG, Monheim am Rhein, Germany, and submitted by Bayer CropScience, Research Triangle Park, North Carolina; 14 pages. Study ID: P601121801. Activity ID: RAFVP019. Amendment to final report issued January 12, 2015. ECM 2: EPA MRID No.: 50170146 (Appendix 6, pp. 158-312). Freitag, Th., V. Koch. 2014. Analytical method 01373 for the determination of BCS-CL73507 and the metabolites BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in soil and sediment by HPLC-MS/MS. Report prepared and sponsored by Bayer CropScience AG, Monheim am Rhein, Germany, and submitted by Bayer CropScience, Research Triangle Park, North Carolina; 155 pages. Study ID: P601121801. Activity ID: RAFVP019. Final report issued May 12, 2014. ILV: EPA MRID No.: 50170146. Netzband, D.J., M.G. Jenks. 2016. Independent Laboratory Validation of "Analytical Method 01373 for the Determination of BCS-CL73507 and the Metabolites BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in Soil and Sediment by HPLC-MS/MS". Report prepared, sponsored and submitted by Bayer CropScience, Research Triangle Park, North Carolina; 312 pages. Study and Activity ID: RAFVP017. Final report issued May 4, 2016.
Document No.:	MRID 50170146
Guideline:	850.6100
Statements:	ECM 1: The study was conducted in compliance with OECD and German Good Laboratory Practice (GLP) standards (Appendix 6, p. 146 of MRID 50170146). Signed and dated Data Confidentiality and GLP statements were provided (Appendix 6, pp. 145-146). The Quality Assurance and Authenticity statements were not included. A statement of the Reasons for the Amendment was provided (Appendix 6, p. 148). ECM 2: The study was conducted in compliance with OECD and German GLP standards (Appendix 6, p. 160; Appendix 6, Appendix 9, pp. 311-312 of MRID 50170146). Signed and dated Data Confidentiality and GLP statements were provided (Appendix 6, pp. 159-160; Appendix 6, Appendix 9, pp. 311-312). The Quality Assurance and Authenticity statements were not included. ILV: The study was conducted in compliance with USEPA FIFRA (40 CFR 160) GLP standards (p. 3 of MRID 50170146). Signed and dated Data

	Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-3, 5). The statement of authenticity was not included.		
Classification:	This analytical method is classified as Acceptable. It could not be determined if the ILV was provided with the most difficult matrices with which to validate the method. In the ECM 2, insufficient chromatographic support was provided for the method validation.		
PC Code:	090097		
EFED Final Reviewer:	Ideliz Negrón-Encarnación, Chemist	Signature: Date: 7/23/18	Digitally signed by IDELIZ NEGRON-ENCARNACION Date: 2018.07.23 11:32:12 -04'00'
CDM/CSS-Dynamac JV Reviewers:	Lisa Muto, Environmental Scientist	Signature: Date:	12/13/17
	Kathleen Ferguson, Ph.D., Environmental Scientist	Signature: Date:	12/13/17
Secondary Reviewer:	Maria Papiez	Signature:	
PMRA, Health Canada	Chemistry Evaluation Section	Date:	07/19/18

This Data Evaluation Record may have been altered by the Environmental Fate and Effects Division subsequent to signing by CDM/CSS-Dynamac JV personnel.

Executive Summary

The analytical method, Bayer Method 01373, is designed for the quantitative determination of tetraniliprole (BCS-CL73507) and its transformation products BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673, and BCS-CU81056 in soil and sediment at the stated LOQ of 2 µg/kg using HPLC/MS/MS. The LOQ is less than the lowest toxicological level of concern in soil/sediment for all analytes. The ILV validated the method with the first trial for all analytes with insignificant modifications to the analytical instrumentation; however, it could not be determined if the ILV was provided with the most difficult matrices with which to validate the method. Both ILV matrices were sand soils; no sediment was included in the ILV. The ECM matrices were three soils of various textures containing high clay contents and organic matter percentages, as well as a sediment matrix. Based on the quantitation ion analysis, all ILV data regarding repeatability, accuracy, precision, and specificity were satisfactory for all analytes; linearity was satisfactory for all analytes, except BCS-CQ63359, BCS-CU81055, BCS-CT30673, and BCS-CU81056. Based on the quantitation ion analysis, all ECM data regarding repeatability, accuracy, precision, and specificity were satisfactory for all analytes, except for BCS-CQ63359 in the sediment matrix at the LOQ (mean 68%); linearity was satisfactory for all analytes, except BCS-CR60014, BCS-CU81055, BCS-CT30673, and BCS-CU81056. In the ECM, insufficient chromatographic support was provided for the method validation since no 10×LOQ chromatograms

were provided. The confirmation ion analyses of the ILV and ECM contained many unacceptable performance data and calibration data results. Additionally, confirmation ion chromatograms were not provided in the ILV. However, since a confirmatory method is not usually required when LC/MS and GC/MS is the primary method, the unacceptable or incomplete support for the confirmation ion analysis did not affect the validity of the method.

Table 1. Analytical Method Summary

<table>
<tr><th rowspan="2">Analyte(s) by Pesticide</th><th colspan="2">MRID</th><th rowspan="2">EPA Review</th><th rowspan="2">Matrix</th><th rowspan="2">Method Date (dd/mm/yyyy)</th><th rowspan="2">Registrant</th><th rowspan="2">Analysis</th><th rowspan="2">Limit of Quantitation (LOQ)</th></tr>
<tr><th>Environmental Chemistry Method</th><th>Independent Laboratory Validation</th></tr>
<tr><td>Tetraniliprole (BCS-CL73507)</td><td rowspan="7">50170146 (Appendix 6)[1]</td><td rowspan="7">50170146[2]</td><td rowspan="7"></td><td rowspan="7">Soil</td><td rowspan="7">12/05/2014 (Original Report)
12/01/2015 (Amendment No. 1)</td><td rowspan="7">Bayer CropScience</td><td rowspan="7">LC/MS/MS</td><td rowspan="7">2 µg/kg</td></tr>
<tr><td>BCS-CQ63359</td></tr>
<tr><td>BCS-CR60014</td></tr>
<tr><td>BCS-CR74541</td></tr>
<tr><td>BCS-CU81055</td></tr>
<tr><td>BCS-CT30673</td></tr>
<tr><td>BCS-CU81056</td></tr>
</table>

1 In the ECM, Höfchen Silt Loam Soil [4.3% sand 76.3% silt 19.4% clay, pH 6.7 (in $CaCl_2$), 1.58% organic matter], Laacher Hof Sandy Loam Soil [69.7% sand 18.3% silt 12.0% clay, pH 6.8 (in $CaCl_2$), 2.06% organic matter], Dollendorf Clay Loam Soil [31% sand 38% silt 31% clay, pH 7.3 (in $CaCl_2$), 8.6% organic matter], and Sediment [OECD 218-219; 4% peat, 20% kaolin, 75% quartz sand, 1% $CaCO_3$] were well-characterized (USDA soil texture characterization for soils; Appendix 6, p. 179 of MRID 50170146). Specific sources were not reported, but all soils and sediment matrices were from Germany.

2 In the ILV, the Florida Sand Soil [97% sand, 2% silt 1% clay, pH 5.8 (in $CaCl_2$), 2.5% organic matter] and Washington Sand Soil [88% sand 11% silt 1% clay, pH 7.1 (in $CaCl_2$), 1.2% organic matter] were well-characterized (USDA soil texture characterization; p. 19 of MRID 50170146). Specific sources were not reported, but both soils were from terrestrial dissipation studies (Washington soil, Study MEFVN015; Florida soil, Study MEFVP115).

For the purpose of this DER, any following reference to "ECM" refers to ECM 2: EPA MRID No.: 50170146 (Appendix 6, pp. 158-312).

I. Principle of the Method

Soil samples (20 g) were placed in 100-mL wide-neck glass jars with screw-caps and fortified, if necessary (Appendix 6, p. 187; Appendix 6, Appendix 3, p. 219 of MRID 50170146). The samples were extracted with 40 mL of acetonitrile/water/acetic acid (4000/1000/30, v/v/v) via microwave extraction using a magnetic stirrer (0-3 min. at 400 W and ambient temperature to 60°C; 3-15 min. at 110 W and 60°C). Internal standard (200 µL) was added with mixing, and the samples were cooled. After centrifugation (5 min. at > 12000 g), if necessary, 0.1 mL of the supernatant was transferred to a round bottom tube and mixed with 0.9 mL of 0.1% acetic acid in water. After centrifugation (5 min. at > 12000 g and 5°C), the sample was analyzed by LC/MS/MS.

Samples were analyzed for tetraniliprole, BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673, and BCS-CU81056 using an Agilent 1290 LC coupled with an AB Sciex API6500 LC- MS/MS (Appendix 6, pp. 188-189 of MRID 50170146). The following LC conditions were used: YMC Ultra HT Hydrosphere C18 (2 µm, 12 nm, 30 x 2.00 mm i.d.; column temperature 40°C), mobile phase of (A) Milli-Q water + 1.0% formic acid and (B) acetonitrile + 1.0% formic acid [mobile gradient phase of percent A:B (v:v) at 0.0 min. 80:20, 3.0 min. 30:70, 3.10-3.9 min. 5:95, 4.0-4.5 min. 80:20], injection volume of 10 µL into a 5 µL loop, and Multiple Reaction Monitoring (MRM) with TurboIon Spray (400°C) in positive mode.

Retention times	BCS-CL73507 / BCS-CL73507 ISTD approx. 1.9 min BCS-CQ63359 / BCS-CQ63359 ISTD approx. 2.5 min BCS-CR60014 / BCS-CR60014 ISTD approx. 1.3 min BCS-CR74541 / BCS-CR74541 ISTD approx. 1.6 min BCS-CU81055 / BCS-CU81055 ISTD approx. 1.5 min BCS-CT30673 / BCS-CT30673 ISTD approx. 2.1 min BCS-CU81056 / BCS-CU81056 ISTD approx. 1.9 min

Two MRM transitions were monitored, one for quantitation and a second for confirmatory purposes, for each analyte and each soil tested:

BCS-CL73507	m/z 545.070 → 356.100 quantitation)
	m/z 545.070 → 376.000 confirmation)
BCS-CQ63359	m/z 527.080 → 389.100 quantitation)
	m/z 527.080 → 374.100 confirmation)
BCS-CR60014	m/z 563.043 → 356.100 quantitation)
	m/z 563.043 → 394.100 confirmation)
BCS-CR74541	m/z 564.020 → 356.000 quantitation)
	m/z 564.020 → 395.000 confirmation)
BCS-CU81055	m/z 550.040 → 395.100 quantitation)
	m/z 550.040 → 356.000 confirmation)
BCS-CT30673	m/z 546.056 → 408.100 quantitation)
	m/z 546.056 → 267.100 confirmation)
BCS-CU81056	m/z 532.054 → 394.100 quantitation)
	m/z 532.054 → 366.100 confirmation)

The ILV performed the ECM methods for each analyte as written, except that different analytical instrumentation was used (pp. 19, 21-22 of MRID 50170146). The LC/MS/MS analysis was performed using a Shimadzu 20ADXR HPLC coupled to an AB Sciex Triple Quad API 6500 LC/MS/MS system. The chromatographic parameters were the same as those of the ECM.

Retention times	BCS-CL73507 / BCS-CL73507 ISTD approx. 2.22 min BCS-CQ63359 / BCS-CQ63359 ISTD approx. 2.80 min BCS-CR60014 / BCS-CR60014 ISTD approx. 1.65 min BCS-CR74541 / BCS-CR74541 ISTD approx. 1.95 min BCS-CU81055 / BCS-CU81055 ISTD approx. 1.80 min BCS-CT30673 / BCS-CT30673 ISTD approx. 2.37 min BCS-CU81056 / BCS-CU81056 ISTD approx. 2.21 min

Two MRM transitions were monitored, one for quantitation and a second for confirmatory purposes, for each analyte. These were the same as those of the ECM (m/z ± 0.1):

BCS-CL73507	amu 545.1 → 356.0 (quantitation)
	amu 545.1 → 376.0 (confirmation)
BCS-CQ63359	amu 527.0 → 389.0 (quantitation)
	amu 527.0 → 374.1 (confirmation)
BCS-CR60014	amu 563.0 → 356.0 (quantitation)
	amu 563.0 → 394.0 (confirmation)
BCS-CR74541	amu 564.0 → 356.0 (quantitation)
	amu 564.0 → 395.0 (confirmation)
BCS-CU81055	amu 550.0 → 395.1 (quantitation)
	amu 550.0 → 356.0 (confirmation)
BCS-CT30673	amu 545.8 → 408.1 (quantitation)
	amu 545.8 → 267.0 (confirmation)
BCS-CU81056	amu 532.0 → 394.1 (quantitation)
	amu 532.0 → 366.0 (confirmation)

In the ECM and ILV, the Limit of Quantification (LOQ) was 2 µg/kg for tetraniliprole (BCS-CL73507) and its transformation products, BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673, and BCS-CU81056 (pp. 9, 26; Table 15, p. 33; Appendix 6, pp. 168; Appendix 6, Tables 19-25, pp. 196-199 of MRID 50170146). The Limit of Detection (LOD) was 0.7 µg/kg for all analytes in the ECM and ILV; calculated LODs ranged 0.277-1.233 µg/kg and 0.4-0.9 µg/kg in the ECM and ILV, respectively, for all analytes.

II. Recovery Findings

ECM (Appendix 6 of MRID 50170146): For the quantitation ion transition analysis, mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of tetraniliprole (BCS-CL73507), BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673, and BCS-CU81056 at fortification levels of 2 µg/kg (LOQ) and 20 µg/kg (10×LOQ) in three soil matrices and one sediment matrix, except for the LOQ analysis in the sediment matrix of BCS-CQ63359 (mean 68%; Appendix 6, Tables 12-18, pp. 193-195 and Tables 49-55, pp. 211-213; DER Attachment 2). For the confirmation ion transition analysis, mean recoveries and RSDs were within guidelines for analysis of tetraniliprole (BCS-CL73507), BCS-CR60014, BCS-CR74541, and BCS-CU81055 at fortification levels of 2 µg/kg (LOQ) and 20 µg/kg (10×LOQ) in three soil matrices and one sediment matrix. For the confirmation ion transition analysis, mean recoveries and RSDs were within guidelines for analysis of BCS-CQ63359, BCS-CT30673, and BCS-CU81056 at the fortification level of 20 µg/kg (10×LOQ) in three soil matrices and one sediment matrix. For the confirmation ion transition analysis, mean recoveries and RSDs were not within guidelines for analysis of BCS-CQ63359, BCS-CT30673, and BCS-CU81056 at the fortification level of 2 µg/kg (LOQ) in all four matrices: BCS-CQ63359 (Höfchen Silt Loam Soil: mean 45%, RSD 103.3%; Laacher Hof Sandy Loam Soil: RSD 20.1%; Dollendorf Clay Loam Soil: mean 57%, RSD 75.6%; sediment: mean 50%, RSD 94.7%); BCS-CT30673 (Höfchen Silt Loam Soil: RSD 137%; Laacher Hof Sandy Loam Soil: mean 29%, RSD 224%; Dollendorf Clay Loam Soil: mean 0%; sediment: RSD 143%); and BCS-CU81056 (Höfchen Silt Loam Soil, Laacher Hof Sandy Loam Soil, Dollendorf Clay Loam Soil, and sediment: means 0%). The unacceptable results of the confirmation ion analysis did not affect the validity of the method since a confirmatory method is not usually required when LC/MS and GC/MS is the primary method. Recovery results of the quantitation ion were comparable to those of the confirmation ion for all analytes/matrices/fortifications, except for the LOQ analyses of BCS-CQ63359, BCS-CT30673, and BCS-CU81056. Höfchen Silt Loam Soil [4.3% sand 76.3% silt 19.4% clay, pH 6.7 (in $CaCl_2$), 1.58% organic matter], Laacher Hof Sandy Loam Soil [69.7% sand 18.3% silt 12.0% clay, pH 6.8 (in $CaCl_2$), 2.06% organic matter], Dollendorf Clay Loam Soil [31% sand 38% silt 31% clay, pH 7.3 (in $CaCl_2$), 8.6% organic matter], and Sediment [OECD 218-219; 4% peat, 20% kaolin, 75% quartz sand, 1% $CaCO_3$] were well-characterized (USDA soil texture characterization for soils; Appendix 6, p. 179). Specific sources were not reported, but all soils and sediment matrices were from Germany.

ILV (MRID 50170146): Mean recoveries and RSDs were within guidelines for analysis of tetraniliprole, BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 at fortification levels of 2 µg/kg (LOQ) and 20 µg/kg (10×LOQ) in two soil matrices, except for the LOQ confirmation analysis in the Florida Sand soil matrix of BCS-CT30673 (RSD 33%) and BCS-CU81056 (RSD 26.5%; p. 25; Tables 1-14, pp. 29-32; DER Attachment 2). For the LOQ confirmation analysis of BCS-CT30673 in the Florida Sand soil matrix and the 10×LOQ confirmation analysis of BCS-CU81056 in the Washington Sand soil matrix, means, s.d.s, and RSDs were reviewer-calculated based on data provided in the study report (n = 5) since these values were calculated by the study authors with the exclusion of one value (n = 4). For the Washington Sand soil, recovery results of the quantitation ion were comparable to those of the confirmation ion; for Florida Sand soil, recovery results of the quantitation and confirmation ion were less comparable. The Florida Sand Soil [97% sand, 2% silt 1% clay, pH 5.8 (in $CaCl_2$), 2.5% organic matter] and Washington Sand Soil [88% sand 11% silt 1% clay, pH 7.1 (in $CaCl_2$), 1.2%

organic matter] were well-characterized (USDA soil texture characterization; p. 19). Specific sources were not reported, but both soils were from terrestrial dissipation studies (Washington soil, Study MEFVN015; Florida soil, Study MEFVP115). The method was validated with the first trial for all analytes with insignificant modifications to the analytical instrumentation (pp. 9-10, 21-22, 26).

Table 2. Initial Validation Method Recoveries for Tetraniliprole (BCS-CL73507), BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673, and BCS-CU81056 in Soil[1,2]

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[3]	Relative Standard Deviation (%)
	Höfchen Silt Loam Soil					
	Quantitation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	78-95	89	7	7.7
	20	5	94-109	99	6	6.4
BCS-CQ63359	2 (LOQ)	5	73-96	86	11	12.4
	20	5	81-91	86	5	5.5
BCS-CR60014	2 (LOQ)	5	86-111	99	10	10.3
	20	5	85-101	93	6	6.1
BCS-CR74541	2 (LOQ)	5	83-106	98	9	8.9
	20	5	83-95	88	5	6.0
BCS-CU81055	2 (LOQ)	5	92-109	103	7	6.9
	20	5	75-99	90	10	11.1
BCS-CT30673	2 (LOQ)	5	72-105	89	14	15.3
	20	5	81-95	89	5	5.8
BCS-CU81056	2 (LOQ)	5	95-117	105	10	9.2
	20	5	69-99	82	12	14.0
	Confirmation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	60-95	78	13	16.7
	20	5	88-109	99	9	9.3
BCS-CQ63359	2 (LOQ)	5	0-109	**45**	46	**103.3**
	20	5	76-112	89	14	15.4
BCS-CR60014	2 (LOQ)	5	72-114	92	18	19.6
	20	5	85-106	95	8	8.0
BCS-CR74541	2 (LOQ)	5	92-116	107	10	8.9
	20	5	84-98	90	5	5.8
BCS-CU81055	2 (LOQ)	5	72-109	96	14	15.0
	20	5	75-96	88	8	9.2
BCS-CT30673	2 (LOQ)[4]	5	0-192	74	101	**137**
	20	5	82-131	106	20	18.9
BCS-CU81056	2 (LOQ)[5]	5	**0**	--	--	--
	20	5	63-97	80	15	18.8

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[3]	Relative Standard Deviation (%)
	Laacher Hof Sandy Loam Soil					
	Quantitation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	100-108	104	4	3.4
	20	5	89-102	97	5	5.1
BCS-CQ63359	2 (LOQ)	5	76-95	86	9	10.1
	20	5	76-98	92	9	10.0
BCS-CR60014	2 (LOQ)	5	76-99	89	9	10.1
	20	5	93-109	100	6	6.5
BCS-CR74541	2 (LOQ)	5	84-110	100	11	10.7
	20	5	84-99	89	6	6.7
BCS-CU81055	2 (LOQ)	5	77-100	92	9	10.3
	20	5	71-91	82	9	11.3
BCS-CT30673	2 (LOQ)	5	73-102	86	12	14.4
	20	5	80-102	91	10	11.1
BCS-CU81056	2 (LOQ)	5	91-108	97	8	7.7
	20	5	77-110	91	15	16.6
	Confirmation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	4[6]	67-99	96 (87)[7]	15	17.0
	20	5	86-100	95	6	6.2
BCS-CQ63359	2 (LOQ)	5	61-98	80	16	**20.1**
	20	5	79-95	88	6	6.9
BCS-CR60014	2 (LOQ)	5	66-101	83	13	15.8
	20	5	92-103	96	4	4.5
BCS-CR74541	2 (LOQ)	5	93-124	109	13	11.8
	20	5	85-100	91	5	5.9
BCS-CU81055	2 (LOQ)	5	82-101	91	10	10.5
	20	5	75-94	83	8	9.1
BCS-CT30673	2 (LOQ)[4]	5	0-146	**29**	65	**224**
	20	5	86-105	96	9	9.5
BCS-CU81056	2 (LOQ)[5]	5	**0**	--	--	--
	20	4[6]	81-96	88	6	7.3
	Dollendorf Clay Loam Soil					
	Quantitation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	81-108	89	11	12.8
	20	5	88-102	96	6	5.6
BCS-CQ63359	2 (LOQ)	5	64-89	77	10	10.0
	20	5	80-87	85	3	12.8
BCS-CR60014	2 (LOQ)	5	89-117	101	11	10.7
	20	5	100-106	102	2	2.2
BCS-CR74541	2 (LOQ)	5	84-99	99	6	12.2
	20	5	87-118	92	12	9.6
BCS-CU81055	2 (LOQ)	5	87-115	101	10	10.3
	20	5	84-97	88	5	6.0
BCS-CT30673	2 (LOQ)	5	77-108	87	13	14.4
	20	5	86-101	94	6	6.0
BCS-CU81056	2 (LOQ)	5	92-118	102	11	10.4

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[3]	Relative Standard Deviation (%)
	20	5	71-99	85	10	11.7
	Confirmation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	81-98	88	7	7.6
	20	5	92-104	98	4	4.5
BCS-CQ63359	2 (LOQ)	5	0-100	**57**	43	**75.6**
	20	5	76-98	85	8	8.9
BCS-CR60014	2 (LOQ)	5	72-99	87	10	11.2
	20	5	87-108	97	10	10.3
BCS-CR74541	2 (LOQ)	5	93-112	102	7	7.2
	20	5	85-102	95	7	7.1
BCS-CU81055	2 (LOQ)	5	72-112	95	16	17.3
	20	5	84-94	90	4	4.4
BCS-CT30673	2 (LOQ)[5]	5	**0**	--	--	--
	20	5	71-115	95	19	19.8
BCS-CU81056	2 (LOQ)[5]	5	**0**	--	--	--
	20	4[6]	76-113	95	18	19.4
	Sediment (OECD 218/219)					
	Quantitation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	73-94	82	9	15.7
	20	5	82-92	87	4	4.6
BCS-CQ63359	2 (LOQ)	5	65-75	**68**	4	3.3
	20	5	76-103	87	11	5.7
BCS-CR60014	2 (LOQ)	5	88-100	94	6	5.9
	20	5	93-110	100	7	7.0
BCS-CR74541	2 (LOQ)	5	70-101	92	13	12.5
	20	5	79-95	87	6	6.6
BCS-CU81055	2 (LOQ)	5	71-103	83	14	16.7
	20	5	74-83	79	3	3.7
BCS-CT30673	2 (LOQ)	5	60-94	83	14	16.4
	20	5	89-103	96	6	6.3
BCS-CU81056	2 (LOQ)	5	75-119	97	16	17.0
	20	5	75-103	86	11	13.1
	Confirmation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	80-102	87	11	19.9
	20	5	82-95	88	5	5.5
BCS-CQ63359	2 (LOQ)	5	0-97	**50**	47	**94.7**
	20	5	72-96	84	9	10.7
BCS-CR60014	2 (LOQ)	5	91-104	96	5	5.6
	20	5	87-103	95	7	7.3
BCS-CR74541	2 (LOQ)	5	81-102	91	9	9.8
	20	5	82-94	87	5	5.6
BCS-CU81055	2 (LOQ)	5	68-114	95	18	19.4
	20	5	75-80	78	3	3.3
BCS-CT30673	2 (LOQ)[4]	5	0-260	85	121	**143**
	20	5	102-113	107	5	4.9

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[3]	Relative Standard Deviation (%)
BCS-CU81056	2 (LOQ)[5]	5	0	--	--	--
	20	5	71-112	87	16	18.3

Data (uncorrected recovery results; Appendix 6, pp. 190-191) were obtained from Appendix 6, Tables 12-18, pp. 193-195 and Tables 49-55, pp. 211-213 of MRID 50170146 and DER Attachment 2.

1 Höfchen Silt Loam Soil [4.3% sand 76.3% silt 19.4% clay, pH 6.7 (in $CaCl_2$), 1.58% organic matter], Laacher Hof Sandy Loam Soil [69.7% sand 18.3% silt 12.0% clay, pH 6.8 (in $CaCl_2$), 2.06% organic matter], Dollendorf Clay Loam Soil [31% sand 38% silt 31% clay, pH 7.3 (in $CaCl_2$), 8.6% organic matter], and Sediment [OECD 218-219; 4% peat, 20% kaolin, 75% quartz sand, 1% $CaCO_3$] were well-characterized (USDA soil texture characterization for soils; Appendix 6, p. 179). Specific sources were not reported, but all soils and sediment matrices were from Germany.

2 Two ion pair transition were monitored for each analyte (see above).

3 Standard deviations (s.d.s) were reviewer-calculated based on data provided in the study report since these values were not provided by the study authors. Rules of significant figures were followed.

4 Means, s.d.s, and RSDs were reviewer-calculated based on data provided in the study report (n = 5) since these values were not calculated by the study authors. Rules of significant figures were followed.

5 Means, s.d.s, and RSDs could not be determined because all values were 0.

6 Only four values were reported in the study report for the fortification/analyte/matrix.

7 Mean value in parenthesis was the reviewer-calculated value based on the values reported in the study report. The s.d. and RSD values of the reviewer matched those reported in the study report.

Table 3. Independent Validation Method Recoveries for Tetraniliprole (BCS-CL73507), BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in Soil[1,2]

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
	Florida Sand Soil					
	Quantitation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	92-110	97	7.5	7.8
	20	5	89-107	96	7.0	7.3
BCS-CQ63359	2 (LOQ)	5	75-88	81	6.3	7.7
	20	5	96-114	102	7.1	6.9
BCS-CR60014	2 (LOQ)	5	92-112	100	8.9	8.9
	20	5	94-112	102	7.2	7.1
BCS-CR74541	2 (LOQ)	5	73-98	88	10.2	11.7
	20	5	89-103	96	5.7	5.9
BCS-CU81055	2 (LOQ)	5	70-102	83	14.8	18.0
	20	5	91-105	99	5.9	6.0
BCS-CT30673	2 (LOQ)	5	66-95	84	11.0	13.1
	20	5	105-120	110	6.3	5.7
BCS-CU81056	2 (LOQ)	5	65-91	76	10.7	14.1
	20	5	88-98	92	4.0	4.4
	Confirmation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	84-105	98	8.6	8.8
	20	5	90-108	97	8.4	8.7
BCS-CQ63359	2 (LOQ)	5	88-122	104	14.0	13.4
	20	5	94-110	102	5.7	5.6

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
BCS-CR60014	2 (LOQ)	5	89-109	99	8.1	8.1
	20	5	95-110	101	7.4	7.3
BCS-CR74541	2 (LOQ)	5	74-111	94	13.8	14.7
	20	5	92-99	96	2.6	2.7
BCS-CU81055	2 (LOQ)	5	59-84	72	10.0	13.9
	20	5	92-97	95	2.2	2.3
BCS-CT30673	2 (LOQ)[3]	5	70-155	100	33	**33**
	20	5	106-119	113	4.8	4.3
BCS-CU81056	2 (LOQ)	5	69-121	91	24.2	**26.5**
	20	5	99-112	106	4.9	4.7
	Washington Sand Soil					
	Quantitation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	93-114	104	9.8	9.4
	20	5	90-101	96	4.5	4.7
BCS-CQ63359	2 (LOQ)	5	78-105	90	9.8	11.0
	20	5	100-113	107	5.5	5.1
BCS-CR60014	2 (LOQ)	5	91-105	98	5.4	5.5
	20	5	91-104	100	5.3	5.3
BCS-CR74541	2 (LOQ)	5	79-110	98	12.1	12.3
	20	5	93-108	99	5.8	5.9
BCS-CU81055	2 (LOQ)	5	73-114	91	17.0	18.8
	20	5	104-117	112	5.7	5.1
BCS-CT30673	2 (LOQ)	5	85-108	96	9.0	9.4
	20	5	107-121	111	5.7	5.1
BCS-CU81056	2 (LOQ)	5	91-107	99	6.7	6.8
	20	5	105-116	110	4.1	3.7
	Confirmation ion					
Tetraniliprole (BCS-CL73507)	2 (LOQ)	5	95-111	101	6.6	6.5
	20	5	90-99	95	3.3	3.5
BCS-CQ63359	2 (LOQ)	5	73-111	94	15.9	17.0
	20	5	97-110	106	5.9	5.6
BCS-CR60014	2 (LOQ)	5	91-123	112	12.6	11.2
	20	5	92-105	98	5.7	5.8
BCS-CR74541	2 (LOQ)	5	88-111	101	9.3	9.2
	20	5	90-108	99	7.1	7.2
BCS-CU81055	2 (LOQ)	5	81-114	94	14.5	15.5
	20	5	105-117	110	4.4	4.0
BCS-CT30673	2 (LOQ)	5	73-105	88	12.0	13.6
	20	5	95-119	106	8.5	8.0
BCS-CU81056	2 (LOQ)	5	78-96	88	7.2	8.1
	20[3]	5	101-143	116	17	15

Data (uncorrected recovery results; p. 23) were obtained from p. 25; Tables 1-14, pp. 29-32 of MRID 50170146.

1 The Florida Sand Soil [97% sand, 2% silt 1% clay, pH 5.8 (in $CaCl_2$), 2.5% organic matter] and Washington Sand Soil [88% sand 11% silt 1% clay, pH 7.1 (in $CaCl_2$), 1.2% organic matter] were well-characterized (USDA soil texture characterization; p. 19). Specific sources were not reported, but both soils were from terrestrial dissipation studies (Washington soil, Study MEFVN015; Florida soil, Study MEFVP115).

2 Two ion pair transition were monitored for each analyte (see above).
3 Means, s.d.s, and RSDs were reviewer-calculated based on data provided in the study report (n = 5) since these values were calculated by the study authors with the exclusion of one value (n = 4). Rules of significant figures were followed.

III. Method Characteristics

In the ECM and ILV, the LOQ was 2 µg/kg for tetraniliprole (BCS-CL73507) and its transformation products, BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 (pp. 9, 26; Table 15, p. 33; Appendix 6, pp. 168, 195; Appendix 6, Tables 19-25, pp. 196-199 of MRID 50170146). In the ECM, the LOQ was defined as the lowest fortification level experimentally providing a mean recovery between 70 and 110% with a relative standard deviation of ≤ 20%, provided that the blank values were below 30% at this level. The LOQ was reported in the ILV from the ECM. No calculations or comparisons to background levels were reported to support the method LOQ. The LOD was 0.7 µg/kg for all analytes in the ECM and ILV. The LOD was calculated using the following equation: LOD = ($t_{0.99}$ × S) + average apparent residue in the untreated control, where $t_{0.99}$ equaled 3.747 (the one-tailed t-statistic at the 99% confidence level for n-1 replicates) and S equaled the standard deviation of n samples fortified at the LOQ. Calculated LODs ranged 0.277-1.233 µg/kg and 0.4-0.9 µg/kg in the ECM and ILV, respectively, for all analytes. In the ILV, the method LOD was defined as one-third of the LOQ.

Table 4. Method Characteristics

Analyte			Tetraniliprole (BCS-CL73507)	BCS-CQ63359	BCS-CR60014	BCS-CR74541	BCS-CU81055	BCS-CT30673	BCS-CU81056
Limit of Quantitation (LOQ)			2 µg/kg						
Limit of Detection (LOD)	ECM		0.7 µg/kg (method) 0.277-1.233 µg/kg (calculated)						
	ILV		0.7 µg/kg (method) 0.4-0.9 µg/kg (calculated)						
Linearity (calibration curve r^2 and concentration range)	ECM[1]		r^2 = 0.9966 (Q) r^2 = 0.9990 (C)	r^2 = 0.9958 (Q) r^2 = 0.9952 (C)	r^2 = **0.9916** (Q) r^2 = 0.9988 (C)	r^2 = 0.9994 (Q) r^2 = 0.9998 (C)	r^2 = **0.9914** (Q) r^2 = **0.9940** (C)	r^2 = **0.9924** (Q) r^2 = **0.9890** (C)	r^2 = **0.9896** (Q) r^2 = **0.9902** (C)[2]
	ILV	WA	r^2 = 0.9974 (Q) r^2 = 0.9984 (C)	r^2 = **0.9914** (Q) r^2 = **0.9924** (C)	r^2 = 0.9980 (Q) r^2 = 0.9984 (C)	r^2 = 0.9982 (Q) r^2 = 0.9986 (C)	r^2 = **0.9886** (Q) r^2 = **0.9851** (C)	r^2 = **0.9938** (Q) r^2 = **0.9932** (C)	r^2 = **0.9924** (Q) r^2 = **0.9930** (C)
		FL	r^2 = 0.9982 (Q) r^2 = 0.9980 (C)	r^2 = 0.9962 (Q) r^2 = 0.9962 (C)	r^2 = 0.9970 (Q) r^2 = 0.9978 (C)	r^2 = 0.9976 (Q) r^2 = 0.9966 (C)	r^2 = **0.9932** (Q) r^2 = **0.9930** (C)	r^2 = **0.9886** (Q) r^2 = **0.9924** (C)	r^2 = **0.9900** (Q) r^2 = **0.9882** (C)
	Range:		0.03-50.0 ng/mL						
Repeatable	ECM[3]	Q	Yes at LOQ and 10×LOQ in three soils and one sediment.	Yes at LOQ and 10×LOQ in three soils. **No** at LOQ in sediment (mean **68**%); yes at 10×LOQ.	Yes at LOQ and 10×LOQ in three soils and one sediment.				
		C[4]		Yes at 10×LOQ, but **No** at LOQ in three soils and one sediment: Höfchen Silt Loam Soil (mean **45**%, RSD **103.3**%); Laacher Hof Sandy Loam Soil (RSD **20.1**%); Dollendorf Clay Loam Soil (mean **57**%, RSD **75.6**%); sediment (mean **50**%, RSD **94.7**%).	Yes at LOQ and 10×LOQ in three soils and one sediment.			Yes at 10×LOQ, but **No** at LOQ in three soils and one sediment: Höfchen Silt Loam Soil (RSD **137**%); Laacher Hof Sandy Loam Soil (mean **29**%, RSD **224**%); Dollendorf Clay Loam Soil (mean **0**%); sediment (RSD **143**%).	Yes at 10×LOQ, but **No** at LOQ in three soils and one sediment (means **0**% for all four matrices).

	ILV[5,6]	Q	Yes at LOQ and 10×LOQ in two sand soils.						
		C[4]	Yes at LOQ and 10×LOQ in two sand soils.					Yes at LOQ and 10×LOQ in Washington Sand soil. Yes at 10×LOQ, but **No** at LOQ in Florida Sand soil (RSD **33**%).	Yes at LOQ and 10×LOQ in Washington Sand soil. Yes at 10×LOQ, but **No** at LOQ in Florida Sand soil (RSD **26.5**%).
Reproducible			Yes at LOQ and 10×LOQ (based on quantitation ion results and use of only sand soil matrices in the ILV) (no sediment matrix was included in the ILV)						
Specific	ECM		Yes; minor baseline noise interfered with peak integration at the LOQ.	Q: Yes; minor baseline noise interfered with peak integration at the LOQ. C[4]: Significant baseline noise (up to 100% of LOQ peak height) surrounded the LOQ peak.	Yes; minor baseline noise interfered with peak integration at the LOQ.		Yes; minor baseline noise interfered with peak integration at the LOQ. A significant contaminant was observed in the C chromatogram which did not interfere with the LOQ peak.	Yes; minor baseline noise interfered with peak integration at the LOQ.	Q: Yes; minor baseline noise interfered with peak integration at the LOQ. A significant contaminant was observed in the C chromatogram which did not interfere with the LOQ peak. C[4]: Significant baseline noise (up to 50% of LOQ peak height) surrounded the LOQ peak.
			No 10×LOQ chromatograms were provided.						
	ILV		**No** confirmation ion chromatograms were provided.						
		WA	Yes, matrix interferences were <20% of the LOQ (based on peak area).	Yes, matrix interferences were <10% of the LOQ (based on peak area).	Yes, matrix interferences were <5% of the LOQ (based on peak area).	Yes, matrix interferences were <10% of the LOQ (based on peak area). Significant contaminants were observed which did not interfere	Yes, matrix interferences were <10% of the LOQ (based on peak area). Some minor baseline noise interfered with peak integration at	Yes, matrix interferences were <15% of the LOQ (based on peak area).	Yes, matrix interferences were <15% of the LOQ (based on peak area). Significant contaminants were observed which did not interfere

						with the LOQ or 10×LOQ peaks.	the LOQ.		with the LOQ or 10×LOQ peaks.
		FL	Yes, matrix interferences were <5% of the LOQ (based on peak area).	Yes, matrix interferences were <10% of the LOQ (based on peak area). Some minor baseline noise interfered with peak integration at the LOQ.	Yes, matrix interferences were <5% of the LOQ (based on peak area). Some minor baseline noise interfered with peak integration at the LOQ.			Yes, matrix interferences were <7% of the LOQ (based on peak area). Some minor baseline noise interfered with peak integration at the LOQ.	Yes, matrix interferences were <10% of the LOQ (based on peak area). Significant contaminants were observed which did not interfere with the LOQ or 10×LOQ peaks. Some minor baseline noise interfered with peak integration at the LOQ.

Data were obtained from pp. 9, 26; Table 15, p. 33; Appendix 6, pp. 168, 195; Appendix 6, Tables 19-25, pp. 196-199 (LOQ/LOD); p. 25; Tables 1-14, pp. 29-32 (ILV recovery results); Appendix 1, pp. 34-62 (ILV calibration curves); Appendix 2, pp. 63-135 (ILV chromatograms); Appendix 6, Tables 12-18, pp. 193-195 and Tables 49-55, pp. 211-213 (ECM recovery results); Appendix 6, Appendix 6, pp. 231-244 (ECM calibration curves); Appendix 6, Appendix 7, pp. 245-309 (ECM chromatograms) of MRID 50170146; DER Attachment 2. WA = Washington Sand soil; FL = Florida Sand soil. Q = Quantitation ion transition; C = Confirmation ion transition.

1 Correlation coefficients (r^2) were reviewer-calculated based on r values (1/x weighted linear regression analysis) reported in the study report; solvent standards were used (Appendix 6, Appendix 6, pp. 231-244 of MRID 50170146; DER Attachment 2).

2 Calibrant concentration range was 0.25-50.0 ng/mL.

3 In the ECM, Höfchen Silt Loam Soil [4.3% sand 76.3% silt 19.4% clay, pH 6.7 (in $CaCl_2$), 1.58% organic matter], Laacher Hof Sandy Loam Soil [69.7% sand 18.3% silt 12.0% clay, pH 6.8 (in $CaCl_2$), 2.06% organic matter], Dollendorf Clay Loam Soil [31% sand 38% silt 31% clay, pH 7.3 (in $CaCl_2$), 8.6% organic matter], and Sediment [OECD 218-219; 4% peat, 20% kaolin, 75% quartz sand, 1% $CaCO_3$] were well-characterized (USDA soil texture characterization for soils; Appendix 6, p. 179 of MRID 50170146). Specific sources were not reported, but all soils and sediment matrices were from Germany.

4 A confirmatory method is not usually required when LC/MS and GC/MS is the primary method.

5 In the ILV, Florida Sand Soil [97% sand, 2% silt 1% clay, pH 5.8 (in $CaCl_2$), 2.5% organic matter] and Washington Sand Soil [88% sand 11% silt 1% clay, pH 7.1 (in $CaCl_2$), 1.2% organic matter] were well-characterized (USDA soil texture characterization; p. 19). Specific sources were not reported, but both soils were from terrestrial dissipation studies (Washington soil, Study MEFVN015; Florida soil, Study MEFVP115).

6 The ILV validated the method with the first trial for all analytes with insignificant modifications to the analytical instrumentation (pp. 9-10, 21-22, 26 of MRID 50170146).

Linearity is satisfactory when $r^2 \geq 0.995$.

IV. Method Deficiencies and Reviewer's Comments

1. The full ECM, including Amendment No. 1, was provided in the Appendix of the ILV MRID 50170146. The full ECM, including Amendment No. 1, was separately submitted as MRID 50216525. The study reports provided in MRID 50216525 and Appendix 6, pp. 144-312 of MRID 50170146 were identical. MRID 50216525 was provided to CDM Smith after the DER for MRID 50170146 was completed; therefore, the ECM citations in this DER applied to Appendix 6, pp. 144-312 of MRID 50170146 and were not updated to apply to MRID 50216525.

2. In the ILV quantitation ion analyses, linearity was not satisfactory for BCS-CQ63359 (r^2 = 0.9914), BCS-CU81055 (r^2 = 0.9886), BCS-CT30673 (r^2 = 0.9938), and BCS-CU81056 (r^2 = 0.9924) in the Washington sand soil and for BCS-CU81055 (r^2 = 0.9932), BCS-CT30673 (r^2 = 0.9886), and BCS-CU81056 (r^2 = 0.9900) in the Florida sand soil (Appendix 1, pp. 34-62 of MRID 50170146).

   In the ECM quantitation ion analyses, linearity was not satisfactory for BCS-CR60014 (r^2 = 0.9916), BCS-CU81055 (r^2 = 0.9914), BCS-CT30673 (r^2 = 0.9924), and BCS-CU81056 (r^2 = 0.9896; Appendix 6, Appendix 6, pp. 231-244 of MRID 50170146).

   OPPTS 850.6100 Guideline indicate that linearity is satisfactory when $r^2 \geq 0.995$. PMRA adopted the APVMA criteria for r^2 to be ≥ 0.99. Overall, the linearity is considered acceptable.

3. In the ECM quantitation ion analyses, performance data for BCS-CQ63359 in the sediment matrix did not meet OCSPP guidelines requirements for precision and accuracy at the LOQ (mean 68%; Appendix 6, Tables 12-18, pp. 193-195; DER Attachment 2). OCSPP Guideline 850.6100 criteria for precision and accuracy states that means for replicates at each spiking level are between 70% and 120%.

4. It could not be determined if the ILV was provided with the most difficult matrices with which to validate the method. Both ILV matrices were sand soils. No sediment was included in the ILV. The ECM matrices contained high clay contents and organic matter percentages, as well as a sediment matrix.

5. In the ECM, insufficient chromatographic support was provided for the method validation since no 10×LOQ chromatograms were provided. Representative chromatograms for the controls and each fortification level should be provided for all matrices tested so that the specificity of the method can be fully evaluated.

6. The communications between the ILV Study Director and method developer were reported (p. 26; Appendix 5, p. 142 of MRID 50170146). The ILV reported that communication involved comments regarding the study protocol and communication regarding the successful trial.

7. In the ILV confirmation ion analyses, linearity was not satisfactory for BCS-CQ63359 (r^2 = 0.9924), BCS-CU81055 (r^2 = 0.9851), BCS-CT30673 (r^2 = 0.9932), and BCS-CU81056 (r^2 = 0.9930) in the Washington sand soil and for BCS-CU81055 (r^2 = 0.9930), BCS-CT30673

($r^2 = 0.9924$), and BCS-CU81056 ($r^2 = 0.9882$) in the Florida sand soil (Appendix 1, pp. 34-62 of MRID 50170146).

In the ECM confirmation ion analyses, linearity was not satisfactory for BCS-CU81055 ($r^2 = 0.9940$), BCS-CT30673 ($r^2 = 0.9890$), and BCS-CU81056 ($r^2 = 0.9902$; Appendix 6, Appendix 6, pp. 231-244 of MRID 50170146).

Linearity is satisfactory when $r^2 \geq 0.995$; however, a confirmatory method is not usually required when LC/MS and GC/MS is the primary method.

8. In the ILV confirmation ion analyses, performance data for BCS-CT30673 (RSD 33%) and BCS-CU81056 (RSD 26.5%) in the Florida Sand soil matrix did not meet OCSPP guidelines requirements for precision and accuracy at the LOQ (p. 25; Tables 1-14, pp. 29-32 of MRID 50170146).

   In the ECM confirmation ion analyses, performance data did not meet OCSPP guidelines requirements for precision and accuracy for analyses of BCS-CQ63359, BCS-CT30673, and BCS-CU81056 at the fortification level of 2 µg/kg (LOQ) in all four matrices: BCS-CQ63359 (Höfchen Silt Loam Soil: mean 45%, RSD 103.3%; Laacher Hof Sandy Loam Soil: RSD 20.1%; Dollendorf Clay Loam Soil: mean 57%, RSD 75.6%; sediment: mean 50%, RSD 94.7%); BCS-CT30673 (Höfchen Silt Loam Soil: RSD 137%; Laacher Hof Sandy Loam Soil: mean 29%, RSD 224%; Dollendorf Clay Loam Soil: mean 0%; sediment: RSD 143%); and BCS-CU81056 (Höfchen Silt Loam Soil, Laacher Hof Sandy Loam Soil, Dollendorf Clay Loam Soil, and sediment: means 0%; Appendix 6, Tables 49-55, pp. 211-213; DER Attachment 2).

   OCSPP Guideline 850.6100 criteria for precision and accuracy states that means for replicates at each spiking level are between 70% and 120% and RSDs are ≤ 20%; however, a confirmatory method is not usually required when LC/MS and GC/MS is the primary method. Therefore, the unacceptable results of the confirmation ion analysis did not affect the validity of the method.

9. In the ILV, incomplete chromatographic support was provided for the method validation since no chromatograms of the confirmation ion analysis were provided. Representative chromatograms for the controls and each fortification level should be provided for all ion transitions tested so that the specificity of the method can be fully evaluated. However, a confirmatory method is not usually required when LC/MS and GC/MS is the primary method; therefore, the lack of chromatograms of the confirmation ion analysis did not affect the validity of the method.

10. The determination of the LOQ in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 9, 26; Table 15, p. 33; Appendix 6, pp. 168, 195; Appendix 6, Tables 19-25, pp. 196-199 of MRID 50170146). In the ECM, the LOQ was defined as the lowest fortification level experimentally providing a mean recovery between 70 and 110% with a relative standard deviation of ≤ 20%, provided that the blank values were below 30% at this level. The LOQ was reported in the ILV from the ECM. No calculations or comparisons to background levels were reported to support the

method LOQ. The LOD was calculated using the following equation: LOD = ($t_{0.99}$ × S) + average apparent residue in the untreated control, where $t_{0.99}$ equaled 3.747 (the one-tailed t-statistic at the 99% confidence level for n-1 replicates) and S equaled the standard deviation of n samples fortified at the LOQ. In the ILV, the method LOD was defined as one-third of the LOQ.

11. ECM 1 [EPA MRID No.: 50170146 (Appendix 6, pp. 144-157). Freitag, Th. 2015. Amendment No. 1 to Final Report No: MR-13/100 – Analytical method 01373 for the determination of BCS-CL73507 and the metabolites BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in soil and sediment by HPLC-MS/MS. Report prepared and sponsored by Bayer CropScience AG, Monheim am Rhein, Germany, and submitted by Bayer CropScience, Research Triangle Park, North Carolina; 14 pages. Study ID: P601121801. Activity ID: RAFVP019. Amendment to final report issued January 12, 2015.] did not contain any method validation results. The amendment was submitted to correct the description of the solvent used for the fortification solutions and calibration solutions (Appendix 6, p. 148 of MRID 50170146).

12. In the ILV, the total time required to complete one set of 13 samples was reported as three to four hours to complete sample extraction (p. 26 of MRID 50170146). LC/MS/MS analyses were run overnight.

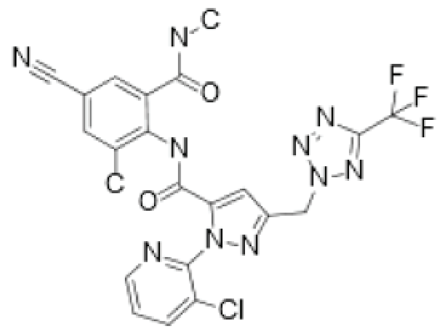
V. References

U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 317-319.

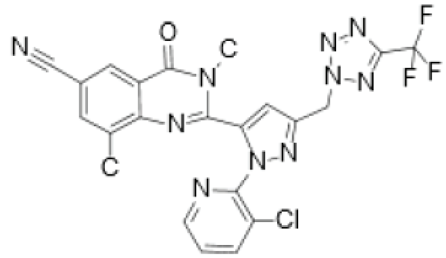
DER ATTACHMENT 1.

BCS-CL73507:



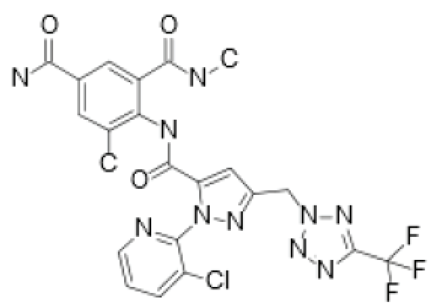
CAS Name:	1-(3-chloropyridin-2-yl)-N-[4-cyano-2-methyl-6-(methylcarbamoyl)phenyl]-3-{[5-(trifluoromethyl-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide
CAS Number	1229654-66-3
Molecular Formula:	$C_{22}H_{16}ClF_3N_{10}O_2$
Molecular Weight:	544.88 g/mol
Standard No.:	K-2056
Purity:	97.9%
Expiration Date:	07/01/2017
Date of Analysis:	07/01/2014
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany

BCS-CQ63359:



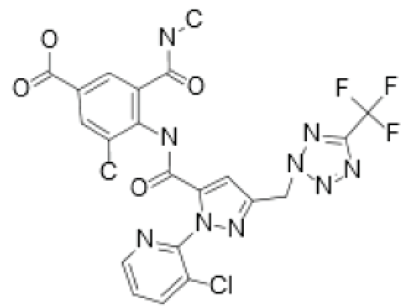
CAS Name:	2-[1[-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]-3,8-dimethyl-4-oxo-3,4-dihydroquinazoline-6-carbonitrile
Molecular Formula:	$C_{22}H_{14}ClF_3N_{10}O$
Molecular Weight:	526.86 g/mol
Standard No.:	K-2118
Purity:	97.7%
Expiration Date:	08/01/2015
Date of Analysis:	08/01/2013
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany

BCS-CR60014:



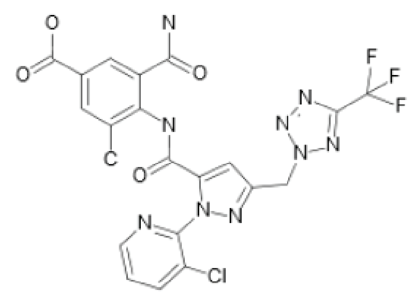
CAS Name:	4-({[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl-1H-pyrazol-5-yl]carbonyl}amino)-N3,5-dimethylisophthalimide
Molecular Formula:	$C_{22}H_{18}ClF_3N_{10}O_3$
Molecular Weight:	562.89 g/mol
Standard No.:	K-2090
Purity:	97.9%
Expiration Date:	06/18/2018
Date of Analysis:	06/20/2013
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany

BCS-CR74541:



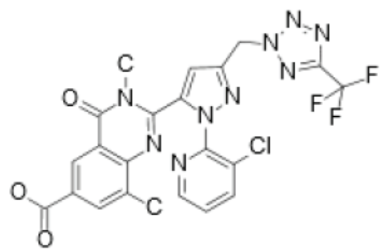
CAS Name:	4-({[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]carbonyl}amino)-3-methyl-5-(methylcarbamoyl)benzoic acid
Molecular Formula:	$C_{22}H_{17}ClF_3N_9O_4$
Molecular Weight:	563.88 g/mol
Standard No.:	K-2117
Purity:	97.2%
Expiration Date:	04/29/2017
Date of Analysis:	05/28/2013
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany

BCS-CU81055:



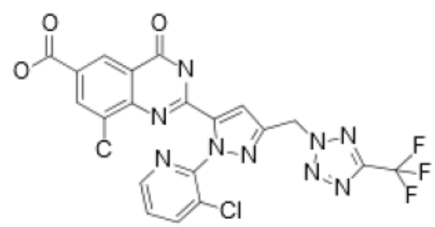
CAS Name:	3-carbamoyl-4-({[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]carbonyl}amino)-5-methylbenzoic acid
Molecular Formula:	$C_{21}H_{15}ClF_3N_9O_4$
Molecular Weight:	549.85 g/mol
Standard No.:	K-2139
Purity:	0.11% (solution 1.021 mg/mL in 1:1 ACN/water)
Expiration Date:	01/16/2016
Date of Analysis:	01/16/2014
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany

BCS-CT30673:



CAS Name:	2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]-3,8-dimethyl-4-oxo-3,4-dihydroquinazoline-6-carboxylic acid
Molecular Formula:	$C_{22}H_{15}ClF_3N_9O_3$
Molecular Weight:	545.86 g/mol
Standard No.:	K-2066
Purity:	97.9%
Expiration Date:	07/24/2016
Date of Analysis:	07/04/2012
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany

BCS-CU81056:



CAS Name:	2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]-8-methyl-4-oxo-3,4-dihydroquinaziline-6-carboxylic acid
Molecular Formula:	$C_{21}H_{13}ClF_3N_9O_3$
Molecular Weight:	531.83 g/mol
Standard No.:	K-2091
Purity:	98.3%
Expiration Date:	06/09/2018
Date of Analysis:	06/19/2013
Storage Conditions:	Frozen
Source:	Bayer CropScience, Frankfurt, Germany