

**Analytical method for pyridate and CL-9673 in soil**

**Reports:** ECM: EPA MRID No.: MRID 50164488 (Appendix 6, pp. 53-99). Chambers, J.G., and G. Tsui. 2011. Pyridate and CL-9673: Determination of Residues of Pyridate and the metabolite CL-9673 in Soil – Method Validation. Report prepared by Battelle UK Ltd., Essex, United Kingdom and sponsored Belchim Crop Protection S.A., Londerzeel, Belgium; submitting company not identified. Belchim Crop Protection NV/SA Method, Report, and Study No.: OZ/10/012. Final report issued October 4, 2011.

ILV: EPA MRID No.: MRID 50164488. Rutt, D. 2016. Independent Laboratory Validation of Belchim Method OZ/10/012- Pyridate and CL-9673: Determination of Residues of Pyridate and the Metabolite CL-9673 in Soil. Report prepared by Critical Path Services, LLC (CPS), a knoell company, Garnet Valley, Pennsylvania, and sponsored Belchim Crop Protection S.A., Londerzeel, Belgium; submitting company not identified. Laboratory Project ID and CPS Study No.: 15-CPS-014. Final report issued September 6, 2016.

**Document No.:** MRID 50164488

**Guideline:** 850.6100


**Statements:** ECM: The study was conducted in compliance with OCED and UK Good Laboratory Practice (GLP) standards, which are compatible with GLP regulations throughout Europe, US and Japan. (Appendix 6, p. 55; Appendix 6, Appendix 5, p. 91 of MRID 50164488). Signed and dated GLP and Quality Assurance statements were provided (Appendix 6, pp. 55-56; Appendix 6, Appendix 5, p. 91). The statement of authenticity was included with the Quality Assurance and GLP statements. The Data Confidentiality was included, but not signed and dated (Appendix 6, p. 54).

ILV: The study was conducted in compliance with USEPA FIFRA GLP standards (40 CFR Part 160; p. 3 of MRID 50164488). 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 supplemental. The ILV soil matrix was not representative of a range of soils. For the Solvent Shake extraction sample processing portion of the ECM method, an insufficient number of samples was prepared, and no representative chromatograms were provided.


**PC Code:** 128834

**EFED Final Reviewer:** James Lin  
Environmental Engineer

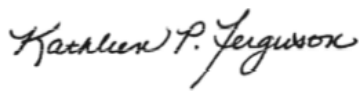
Signature:   
Date: 01/24/18

**CDM/CSS-Dynamac JV**

Lisa Muto,  
Environmental Scientist

Signature:   
Date: 12/13/17

**Reviewers:** Kathleen Ferguson, Ph.D.,  
Environmental Scientist

Signature:   
Date: 12/13/17

*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, Belchim Method OZ/10/012, is designed for the quantitative determination of pyridate and CL-9673 in soil at the stated LOQ of 0.01 mg/kg. The ILV validated the method for pyridate and CL-9673 using the Solvent Shake Extraction sample processing in the first trial using with insignificant modifications to the sample processing and analytical equipment and parameters. The Ultra-Turrax Extraction sample processing was not performed by the ILV. The ILV used characterized sand soil, while the ECM used loamy sand soil; the ILV soil matrix was not representative of a range of soils. Pyridate and CL-9673 was identified using two ion transitions in the ECM and ILV. For the Solvent Shake extraction sample processing, all ILV data regarding repeatability, accuracy, precision, and specificity were satisfactory for pyridate and CL-9673. ILV linearity was not satisfactory for the analysis of CL-9673. In the ECM, an insufficient number of samples was prepared, and no representative chromatograms were provided. For the Ultra-Turrax Extraction sample processing, all ECM data regarding repeatability, accuracy, precision, linearity and specificity were satisfactory for pyridate and CL-9673. The LOD of the method was not reported in the ILV.

**Table 1. Analytical Method Summary**

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Pyridate	Appendix 6 of MRID 50164488 <sup>1</sup>	MRID 50164488 <sup>2</sup>		Soil	04/10/2011		LC/MS/MS	0.01 mg/kg
CL-9673								

<sup>1</sup> In the ECM, the loamy sand soil [Lufa 2.2 (10/008); pH 5.7 (in 1:1 soil:water), 82% sand 11% silt 7% clay, 3.2% organic matter] was characterized by Agvise Laboratories, Northwood, North Dakota (Appendix 6, p. 64; Appendix 6, Appendix 3, p. 89 of MRID 50164488). Soil matrix source was not reported.

<sup>2</sup> In the ILV, the sand soil [ID RS295, CPS sample # GS-15-32-3, SC10 6-12"; 93% sand 6% silt 1% clay, pH 6.0 (in saturated paste), 0.53% organic matter] was provided by Southeast Ag Research, Georgia and characterized by Agvise Laboratories, Northwood, North Dakota (p. 10; Appendix 2, p. 37 of MRID 50164488).

## I. Principle of the Method

### Ultra-Turrax Extraction

Soil (10 g) was fortified in plastic bottles then extracted with 100 mL of methanol:acetic acid (100:0.5, v:v) by blending with an Ultra-Turrax macerator (Appendix 6, p. 64; Appendix 6, Figure 3, p. 78 of MRID 50164488). After centrifugation (conditions not reported), the supernatant was transferred to glass vials and analyzed by LC/MS/MS.

### Solvent Shake Extraction

Soil (10 g) was fortified in plastic bottles then extracted with 100 mL of methanol:acetic acid (100:0.5, v:v) by shaking for 20 minutes (Appendix 6, p. 64; Appendix 6, Figure 3, p. 78 of MRID 50164488). After centrifugation (conditions not reported), the supernatant was transferred to glass vials and analyzed by LC/MS/MS.

### LC/MS/MS Analysis

Samples were analyzed for pyridate and CL-9673 using an Agilent 1100 Series HPLC coupled with an API 5000 Mass Spectrometer with Q Jet Ion Guide using a Turbo-Ion Spray source operated in the positive ion, multiple reaction monitoring (MRM) mode (600°C; Appendix 6, pp. 64-65 of MRID 50164488). The following LC conditions were used: Phenomenex Aqua C18 column (2.0 mm x 50 mm, 5  $\mu$ , 125 Å; column temperature not reported), mobile phase of (A) water:acetic acid (99:1, v:v) and (B) acetonitrile:acetic acid (99:1, v:v) [mobile gradient phase of percent A:B (v:v) at 0.0-0.2 min. 90:10, 1.0-2.0 min. 10:90, 2.65-3.5 min. 90:10] and injection volume of 10  $\mu$ L. Two ion pair transitions were monitored (quantitation and confirmation, respectively):  $m/z$  379.05 $\rightarrow$ 77.20 and  $m/z$  379.05 $\rightarrow$ 68.10 for pyridate and  $m/z$  207.07 $\rightarrow$ 103.97 and  $m/z$  207.07 $\rightarrow$ 67.97 for CL-9673. Observed retention times were *ca.* 2.0 and 1.25 minutes for pyridate and CL-9673, respectively (Appendix 6, Figures 4-7, pp. 79-82).

### ILV

The ILV performed the ECM method using the solvent shake extraction as written, except for the use of a Wrist Action® Shaker, that the fortification solvent was evaporated before extraction, that the centrifugation settings were specified as 10 minutes at 3600 rpm, and insignificant modifications to the analytical equipment and parameters (pp. 12-15 of MRID 50164488). The LC/MS/MS instrument and parameters were similar to those of the ECM. Samples were analyzed for pyridate and CL-9673 using an Agilent Technologies 1200 Infinity Series HPLC coupled with an AB Sciex API 4000 Tandem Mass Spectrometer (550°C). A Phenomenex Synergi Hydro-RP column (2.0 mm x 50 mm, 4  $\mu$ ; column temperature 27.00°C) was used. The other LC conditions were the same as those reported in the ECM. Two ion pair transitions were monitored (quantitation and confirmation, respectively):  $m/z$  379.3 $\rightarrow$ 77.2 and  $m/z$  379.3 $\rightarrow$ 68.1 for pyridate and  $m/z$  207.3 $\rightarrow$ 104.1 and  $m/z$  207.3 $\rightarrow$ 68.0 for CL-9673. Reported retention times were *ca.* 2.7 and 1.6 minutes for pyridate and CL-9673, respectively. No other method modifications were reported.

## LOQ/LOD

In the ECM and ILV, the Limit of Quantification (LOQ) was 0.01 mg/kg for pyridate and CL-9673 in soil (p. 9; Appendix 6, pp. 62, 67 of MRID 50164488). In the ECM, the Limit of Detection (LOD) was determined to be 0.0021 mg/kg for pyridate and 0.002 mg/kg CL-9673. In the ILV, the LOD was not reported.

## **II. Recovery Findings**

ECM (Appendix 6 of MRID 50164488): Using the Ultra-Turrax Extraction sample processing, mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD  $\leq$ 20%) for analysis of pyridate and CL-9673 at fortification levels of 0.01 mg/kg (LOQ) and 0.1 mg/kg (10 $\times$ LOQ) in one soil matrix (Appendix 6, Table 1, pp. 68-69). Two ion pair transitions were monitored for pyridate and CL-9673 using LC/MS/MS in positive mode; the quantification and confirmation ion data was comparable. Using the Solvent Shake Extraction sample processing, mean recoveries and RSDs were within guidelines (mean 70-120%; RSD  $\leq$ 20%) for analysis of pyridate and CL-9673 at fortification levels of 0.01 mg/kg (LOQ) and 0.1 mg/kg (10 $\times$ LOQ) in one soil matrix; however, an insufficient number of samples was prepared for all fortifications/analytes (n = 2 or 3; Appendix 6, Table 3, p. 71). For 10 $\times$ LOQ, where n = 2, the study authors calculated means, but not standard deviations and relative standard deviations. The reviewer did not calculate standard deviations and relative standard deviations since n = 2. Two ion pair transitions were monitored for pyridate and CL-9673 using LC/MS/MS in positive mode; the quantification and confirmation ion data were not comparable. Loamy sand soil [Lufa 2.2 (10/008); pH 5.7 (in 1:1 soil:water), 82% sand 11% silt 7% clay, 3.2% organic matter] was characterized by Agvise Laboratories, Northwood, North Dakota (Appendix 6, p. 64; Appendix 6, Appendix 3, p. 89). Soil matrix source was not reported.

ILV (MRID 50164488): Using the Solvent Shake Extraction sample processing, mean recoveries and RSDs were within guidelines for analysis of pyridate and CL-9673 at fortification levels of 0.01 mg/kg (LOQ) and 0.1 mg/kg (10 $\times$ LOQ) in one soil matrix (p. 17; Tables 1-2, pp. 20-21). Two ion pair transitions were monitored for pyridate and CL-9673 using LC/MS/MS in positive mode; the quantification and confirmation ion data was comparable. The Ultra-Turrax Extraction sample processing was not performed. Sand soil [ID RS295, CPS sample # GS-15-32-3, SC10 6-12"; 93% sand 6% silt 1% clay, pH 6.0 (in saturated paste), 0.53% organic matter] was provided by Southeast Ag Research, Georgia and characterized by Agvise Laboratories, Northwood, North Dakota (p. 10; Appendix 2, p. 37). The method for pyridate and CL-9673 using the Solvent Shake Extraction sample processing was validated in the first trial with insignificant modifications to the sample processing and analytical equipment and parameters (pp. 10, 12-15; Tables 1-2, pp. 20-21).

**Table 2. Initial Validation Method Recoveries for Pyridate and CL-9673 in Soil**

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Ultra-Turrax Extraction</b>						
<b>Loamy Sand Soil<sup>1</sup></b>						
Quantitation ion <sup>2</sup>						
Pyridate	0.01 (LOQ)	6	75-98	86	10.6	12.3
	0.1	6	71-96	83	10.5	12.7
CL-9673	0.01 (LOQ)	6	84-94	87	3.7	4.3
	0.1	6	87-97	92	4.4	4.7
Confirmation ion <sup>2</sup>						
Pyridate	0.01 (LOQ)	6	72-89	82	6.5	7.9
	0.1	6	75-101	86	10.8	12.5
CL-9673	0.01 (LOQ)	6	87-99	91	4.5	4.9
	0.1	6	87-96	92	3.9	4.2
<b>Solvent Shake Extraction</b>						
<b>Loamy Sand Soil<sup>1</sup></b>						
Quantitation ion <sup>2</sup>						
Pyridate	0.01 (LOQ)	<b>3</b>	80-88	83	4.3	5.1
	0.1	<b>2<sup>3</sup></b>	83, 96	90	--	--
CL-9673	0.01 (LOQ)	<b>3</b>	73-83	79	5.1	6.4
	0.1	<b>2</b>	75, 77	76	--	--
Confirmation ion <sup>2</sup>						
Pyridate	0.01 (LOQ)	<b>3</b>	88-93	90	2.5	2.7
	0.1	<b>2</b>	73, 76	74	--	--
CL-9673	0.01 (LOQ)	<b>3</b>	79-85	83	3.6	4.3
	0.1	<b>2</b>	74, 82	78	--	--

Data (uncorrected recovery results; Appendix 6, Appendix 4, p. 90) were obtained from Appendix 6, Table 1, pp. 68-69; Appendix 6, Table 3, p. 71 of MRID 50164488.

<sup>1</sup> Loamy sand soil [Lufa 2.2 (10/008); pH 5.7 (in 1:1 soil:water), 82% sand 11% silt 7% clay, 3.2% organic matter] was characterized by Agvise Laboratories, Northwood, North Dakota (Appendix 6, p. 64; Appendix 6, Appendix 3, p. 89). Soil matrix source was not reported.

<sup>2</sup> Two ion pair transitions were monitored (quantitation and confirmation, respectively):  $m/z$  379.05→77.20 and  $m/z$  379.05→68.10 for pyridate and  $m/z$  207.07→103.97 and  $m/z$  207.07→67.97 for CL-9673.

<sup>3</sup> For  $n = 2$ , the study authors calculated means, but not standard deviations and relative standard deviations. The reviewer did not calculate standard deviations and relative standard deviations since  $n = 2$ .

**Table 3. Independent Validation Method Recoveries for Pyridate and CL-9673 in Soil**

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Solvent Shake Extraction</b>						
<b>Sand Soil<sup>1</sup></b>						
Quantitation ion <sup>2</sup>						
Pyridate	0.01 (LOQ)	5	92.6-102	95.9	4.1	4.3
	0.1	5	99.1-102	101	1.2	1.2
CL-9673	0.01 (LOQ)	5	80.2-87.6	84.5	2.7	3.3
	0.1	5	102-110	106	3.0	2.8
Confirmation ion <sup>2</sup>						
Pyridate	0.01 (LOQ)	5	90.4-98.7	94.8	3.2	3.4
	0.1	5	99.7-107	103	2.7	2.6
CL-9673	0.01 (LOQ)	5	83.6-90.2	86.6	2.5	2.9
	0.1	5	104-109	107	1.8	1.7

Data (uncorrected recovery results; Appendix 3, p. 38) were obtained from p. 17; Tables 1-2, pp. 20-21 of MRID 50164488.

1 Sand soil [ID RS295, CPS sample # GS-15-32-3, SC10 6-12"; 93% sand 6% silt 1% clay, pH 6.0 (in saturated paste), 0.53% organic matter] was provided by Southeast Ag Research, Georgia and characterized by Agvise Laboratories, Northwood, North Dakota (p. 10; Appendix 2, p. 37).

2 Two ion pair transitions were monitored (quantitation and confirmation, respectively):  $m/z$  379.3→77.2 and  $m/z$  379.3→68.1 for pyridate and  $m/z$  207.3→104.1 and  $m/z$  207.3→68.0 for CL-9673.

### III. Method Characteristics

In the ECM and ILV, the LOQ was 0.01 mg/kg for pyridate and CL-9673 in soil (p. 9; Appendix 6, pp. 62, 67; Appendix 6, Appendix 6, p. 97 of MRID 50164488). In the ECM, the LOQ was reported as the lowest quantifiable level. No calculations were provided for the LOQ in the ECM or ILV. In the ECM, the LOD was determined to be 0.0021 mg/kg for pyridate and 0.002 mg/kg CL-9673. The LOD was estimated based on three times the baseline noise; the calculations were not provided. In the ILV, the LOD was not reported.

**Table 4. Method Characteristics for Pyridate and CL-9673 in Soil**

		Pyridate	CL-9673		
Limit of Quantitation (LOQ)	ECM	0.01 mg/kg			
	ILV				
Limit of Detection (LOD)	ECM	0.0021 mg/kg	0.002 mg/kg		
	ILV	<b>Not reported</b>			
Linearity (calibration curve $r^2$ and concentration range) <sup>1</sup>	ECM	$r^2 = 0.9992$ (Q) $r^2 = 0.9998$ (C)	$r^2 = 0.9980$ (Q) $r^2 = 0.9994$ (C)		
	ILV	$r^2 = 0.9988$ (Q) $r^2 = 0.9992$ (C)	$r^2 = 0.9910$ (Q) $r^2 = 0.9914$ (C)		
	Concentration range	0.200-20.0 ng/mL			
Repeatable	ECM <sup>2</sup>	Ultra-Turrax Extraction	Yes at LOQ and 10×LOQ. (characterized loamy sand soil used)		
		Solvent Shake Extraction	Yes at LOQ and 10×LOQ; however, n = 2-3. (characterized loamy sand soil used)		
	ILV <sup>3,4</sup>	Ultra-Turrax Extraction	<b>Not performed</b>		
		Solvent Shake Extraction	Yes at LOQ and 10×LOQ. (characterized sand soil used)		
Reproducible	Ultra-Turrax Extraction		<b>Could not be determined.</b>		
	Solvent Shake Extraction		Yes at LOQ and 10×LOQ.		
Specific	ECM	Ultra-Turrax Extraction	Q	Yes, matrix interferences were <3% of the LOQ (based on peak area), but some minor baseline noise was observed in the control.	Yes, no matrix interferences were observed, but some minor baseline noise was observed in the control.
			C	Yes, matrix interferences were <7% of the LOQ (based on peak area), but some baseline noise was observed in the control.	Analyte peak was barely resolved above the baseline.
	Solvent Shake Extraction		<b>No</b> representative chromatograms provided. <sup>5</sup>		
	ILV	Ultra-Turrax Extraction	<b>No</b> representative chromatograms provided.		
		Solvent Shake Extraction	Yes, matrix interferences were <5% of the LOQ (based on peak height).		

Data were obtained from pp. 9, 13, 17; Tables 1-2, pp. 20-21 (recovery results); Figures 2-5, pp. 24-27 (calibration curve); Figures 6-9, pp. 28-31 (chromatograms); Appendix 6, pp. 62, 66-67; Appendix 6, Table 1, pp. 68-69; Appendix 6, Table 3, p. 71 (ECM recovery results); Appendix 6, Figures 4-7, pp. 79-82 (ECM chromatograms); Appendix 6, Figure 8, pp. 83-84 (ECM calibration curves); Appendix 6, Appendix 6, p. 97 (LOQ/LOD) of MRID 50164488. Q = quantitation ion; C = confirmation ion. All results reported for Q and C ions unless specified otherwise.

1 Correlation coefficients ( $r^2$ ) were reviewer-calculated based on r values (1/x weighted linear regression analysis) reported in the study report (Figures 2-5, pp. 24-27; Appendix 6, Figure 8, pp. 83-84 of MRID 50164488; DER Attachment 2).

2 In the ECM, the loamy sand soil [Lufa 2.2 (10/008); pH 5.7 (in 1:1 soil:water), 82% sand 11% silt 7% clay, 3.2% organic matter] was characterized by Agvise Laboratories, Northwood, North Dakota (Appendix 6, p. 64; Appendix 6, Appendix 3, p. 89 of MRID 50164488). Soil matrix source was not reported.

3 In the ILV, the sand soil [ID RS295, CPS sample # GS-15-32-3, SC10 6-12"; 93% sand 6% silt 1% clay, pH 6.0 (in saturated paste), 0.53% organic matter] was provided by Southeast Ag Research, Georgia and characterized by Agvise Laboratories, Northwood, North Dakota (p. 10; Appendix 2, p. 37 of MRID 50164488).

4 The method for pyridate and CL-9673 using the Solvent Shake Extraction sample processing was validated in the first trial with insignificant modifications to the sample processing and analytical equipment and parameters (pp. 10, 12-15; Tables 1-2, pp. 20-21 of MRID 50164488). The Ultra-Turrax Extraction sample processing was not performed.

5 The reviewer ascertained that the representative chromatograms correlated to the Ultra-Turrax Extraction samples by

comparing recoveries for the first samples of the LOQ and 10×LOQ analyses (REC 1 and REC 7). Linearity is satisfactory when  $r^2 \geq 0.995$ .

#### IV. Method Deficiencies and Reviewer's Comments

1. The TFD MRID 50164489 included the ECM as Appendix 1, Appendix 3, pp. 139-185. That document was the same as that of Appendix 6, pp. 53-99 of ILV MRID 50164488; therefore, MRID 50164489 was not included in the DER to avoid redundancy.

The reviewer noted the Analytical Method of the TFD MRID 50164489 used the Solvent Shake extraction sample processing (pp. 20, 23-24; Appendix 1, pp. 65-66, 70-71 of MRID 50164489). The reviewer did not use the analysis and results of the concurrent analysis of freshly fortified control samples as extra ECM data since the performing laboratory and study author was the same as that of the independent validation.

2. The ILV linearity was not satisfactory for the analysis of CL-9673 ( $r^2 = 0.9910$  and  $r^2 = 0.9914$  for the quantitation and confirmation ions, respectively; Figures 4-5, pp. 26-27 of MRID 50164488; DER Attachment 2). Linearity is satisfactory when  $r^2 \geq 0.995$ .
3. The ILV matrix was a sand soil, while the ECM matrix was a loamy sand soil. In the TFD studies MRIDs 50164489, 50164490, the test soils were characterized as sandy loam, loamy sand, loam and silt loam (Table 11, p. 40 of MRID 50164489; Table 3, p. 33 of MRID 50164490; Appendix 8, p. 72 of MRID 50164496; Appendix 8, p. 74 of MRID 50164497). It did not appear that the ILV was provided with the most difficult matrix with which to validate the method and that the ILV soil matrix covered the range of soils used in the terrestrial field dissipation studies.
4. The ILV did not perform the Ultra-Turrax Extraction sample processing; therefore, only one set of performance data was provided for this portion of the ECM method.
5. For the Solvent Shake extraction sample processing, the ECM contained the following deficiencies:

An insufficient number of samples was prepared for all fortifications/analytes ( $n = 2$  or  $3$ ; Appendix 6, Table 3, p. 71). OCSPP guidelines state that a minimum of five spiked replicates should be analyzed at each concentration (*i.e.*, minimally, the LOQ and 10× LOQ) for each analyte.

The specificity of the method could not be assessed because no representative chromatograms provided. The reviewer ascertained that the representative chromatograms correlated to the Ultra-Turrax Extraction samples by comparing recoveries for the first samples of the LOQ and 10×LOQ analyses (REC 1 and REC 7).

6. The determinations of the LOD and LOQ in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (p. 9; Appendix 6, pp. 62, 67; Appendix 6, Appendix 6, p. 97 of MRID 50164488). In the ECM, the LOQ was reported as the lowest quantifiable level. No calculations were provided for the LOQ in the ECM or



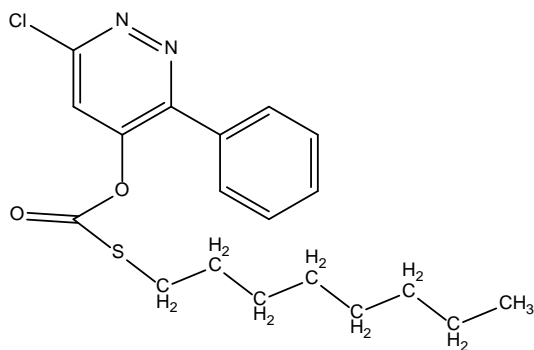
- ILV. In the ECM, the LOD was estimated based on three times the baseline noise; the calculations were not provided. In the ILV, the LOD was not reported.
7. The reviewer noted that the GLP statement of the ECM contained a contradictory statement at the end: “Data is presented for typical soil analysis but no claim is made of GLP compliance” (Appendix 6, p. 55 of MRID 50164488).
  8. The communications between the ECM and ILV were reportedly limited to email exchange regarding study progress (p. 17 of MRID 50164488). Detailed communication records were not provided.
  9. In the ECM, the stability of the final extracts was assessed and determined to be up to 5 days when stored under frozen conditions (nominally -18°C; Appendix 6, Table 2, p. 70 of MRID 50164488).
  10. In the ILV, the time required to complete the extraction of one set of 13 samples (one reagent blank, two matrix controls and ten fortified samples) was reported as *ca.* 4 hours, followed by *ca.* 4 hours for LC/MS/MS analysis (p. 17 of MRID 50164488). The total time requirement of the method was reported as *ca.* one working day in the ILV.

## 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: Chemical Names and Structures****Pyridate**

**IUPAC Name:** *O*-6-Chloro-3-phenylpyridazin-4-yl *S*-octyl thiocarbonate  
**CAS Name:** *O*-6-Chloro-3-phenylpyridazin-4-yl *S*-octyl carbonothioate  
**CAS Number:** 55512-33-9  
**SMILES String:** c1cccc1c2nnc(Cl)cc2OC(=O)SCCCCCCCC

**CL-9673 (Pyridafol)**

**IUPAC Name:** 6-Chloro-3-phenylpyridazin-4-ol  
**CAS Name:** Not reported  
**CAS Number:** 40020-01-7  
**SMILES String:** ClC1=CC(O)=C(N=N1)C2=CC=CC=C2

