

Analytical method for pyridate and pyridafol (CL 9673) in water

Reports: ECM: EPA MRID No.: MRID 50235205. Martin, K.H., and T.N. Paz. 2018. Analytical Method Validation for the Determination of Pyridate and Pyridafol in Freshwater and Surface Water. Report prepared by EAG, Inc., Easton, Maryland, and sponsored and submitted by Belchim Crop Protection NV/SA, Londerzeel, Belgium; 98 pages. Project No.: 792C-105. Final report issued November 1, 2018.

ILV: EPA MRID No.: MRID 50235206. Arndt, T. 2018. Independent Laboratory Validation for the Determination of Pyridate and Pyridafol in Surface and Ground Water. Report prepared by EAG Laboratories – Hercules, Hercules, California, sponsored, and submitted by Belchim Crop Protection NV/SA, Londerzeel, Belgium; 98 pages. Project No.: 3113W. Final report issued December 13, 2018.

Document No.: MRIDs 50235205 & 50235206

Guideline: 850.6100

Statements: ECM: The study was conducted in compliance with OCED and USEPA FIFRA (40 CFR Part 160) Good Laboratory Practice (GLP) standards, except for the characterization and stability of the test substances and analyses of the freshwater for contaminants (p. 3 of MRID 50235205). Signed and dated Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). The statement of authenticity was not included. A Report Approval page is provided (p. 5).

ILV: The study was conducted in compliance with USEPA FIFRA GLP standards, except for the certification of the test substances (p. 3 of MRID 50235206). Signed and dated Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). The statement of authenticity was included with the Quality Assurance statement.


Classification: This analytical method is classified as supplemental. The ECM part is acceptable. The ILV linearity was not satisfactory for the surface water quantitation ion transition analysis of pyridate. The number of ILV trials was not specified. It could not be determined if the ILV was provided with the most difficult matrices with which to validate the method.

PC Code: 128834


EFED Final

Reviewer:

James Lin,
Environmental Engineer

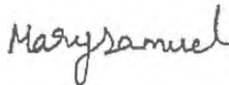
Signature: 
Date: 09/17/2019

Lisa Muto,
Environmental Scientist

Signature: 
Date: 04/22/2019

**CDM/CSS-
Dynamac JV
Reviewers:**

Mary Samuel, M.S.,
Environmental Scientist

Signature: 
Date: 04/22/2019

This Data Evaluation Record may have been altered by the Environmental Fate and Effects Division subsequent to signing by CDM/CSS-Dynamac JV personnel. The CDM/CSS-Dynamac Joint Venture role does not include establishing Agency policies.

Executive Summary

The analytical method, EAG Laboratories-Easton Project No. 792C-105, is designed for the quantitative determination of pyridate and pyridafol (CL 9673) in water at the stated LOQ of 0.005 mg/L. The LOQ is less than the lowest toxicological level of concern in water. The ECM and ILV used two characterized water matrices, a ground/fresh and a surface water. It could not be determined if the ILV was provided with the most difficult matrices with which to validate the method since the ECM water matrices were the same as those of the ILV.

The number of trials was not specified; the reviewer assumed that the ILV validated the ECM in the first trial with only insignificant modifications to the analytical equipment and parameters. All submitted ECM and ILV data pertaining to precision, repeatability, reproducibility, and linearity was acceptable at the LOQ and 10×LOQ in both water matrices, except for the ILV linearity for the surface water quantitation ion transition analysis of pyridate. The reviewer also noted that the specificity of the method was not well-supported for pyridate by the ILV representative quantitation ion transition chromatograms because matrix interferences were *ca.* 13-19% (Q) and *ca.* 17-25% (C) of the LOQ (based on recovery values).

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	MRID 50235205 ¹	MRID 50235206 ²		Water	01/11/2018	Belchim Crop Protection NV/SA	LC/MS/MS	0.005 mg/L
Pyridafol (CL 9673)								

1 In the ECM, the freshwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected from a well *ca.* 40 m deep at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, and surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 14; Appendices 3-5, pp. 92-97 of MRID 50235205).

2 In the ILV, the surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, and groundwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected in the aquatics lab (well water) at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 16; Appendix C, pp. 86-87 of MRID 50235206). These were the same water matrices as those used in the ECM.

I. Principle of the Method

Water samples (10.0 mL) were fortified with either pyridate or pyridafol 0.0100 or 0.00100 mg/mL fortification solutions in glass scintillation vials then diluted immediately with 10.0 mL of 0.2% formic acid in acetonitrile (pp. 15-17; Figure 1, pp. 40-42 of MRID 50235205). An aliquot was transferred to autosampler vials and analyzed by LC/MS/MS.

Samples were analyzed for pyridate and pyridafol using an Agilent 1200 Series Infinity HPLC coupled with an API 4000 Mass Spectrometer using a Turbo-Ion Spray source operated in the positive ion, multiple reaction monitoring (MRM) mode (600°C; p. 15; Table 1, p. 23 of MRID 50235205). The following LC conditions were used: Thermo Betasil C-18 column (2.1 mm x 50 mm, 3 µm; oven temperature 40°C), Betasil C-18 guard column (2.1 x 10 mm), mobile phase of (A) 0.1% formic acid in HPLC-grade water and (B) 0.1% formic acid in acetonitrile [mobile gradient phase of percent A:B (v:v) at 0.00-0.25 min. 85.0:15.0, 2.00 min. 40.0:60.0, 2.30-5.50 min. 5.0:95.0, 5.51-9.00 min. 85.0:15.0] and injection volume of 5.00 µL. Two ion pair transitions were monitored (quantitation and confirmation, respectively): m/z 379.100→207.100 and m/z 379.100→351.300 for pyridate and m/z 207.000→104.100 and m/z 207.000→126.100 for pyridafol. Expected retention times were *ca.* 5.65 and 3.09 minutes for pyridate and pyridafol, respectively.

The ILV performed the ECM method as written, except for insignificant modifications to the analytical equipment and parameters (pp. 18-19, 23; Figure 1, p. 38 of MRID 50235206). The LC/MS/MS instrument and parameters were similar to those of the ECM. Samples were analyzed for pyridate and pyridafol using an Applied Biosystems MDS Sciex API4000 triple quadrupole LC-MS/MS system with Turbo Ionspray (ESI +) source, Thermo Scientific Dionex Ultimate 3000 (550°C). A Thermo BDS Hypersil C18 column (2.1 mm x 50 mm, 3 µm; column temperature 40°C) was used. Injection volume was 5 µL. 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 379.1→207.1 and m/z 379.1→351.3 for pyridate and m/z 207.0→104.1 and m/z 207.0→126.1 for pyridafol. Reported retention times were *ca.* 4.21 and 1.52 minutes for pyridate and pyridafol, respectively.

In the ECM and ILV, the Limit of Quantification (LOQ) was 0.005 mg/L for pyridate and pyridafol in water (p. 18 of MRID 50235205; pp. 24-25 of MRID 50235206). In the ECM, the Limit of Detection (LOD) was calculated to be 0.00000123-0.00000372 mg/L for pyridate and 0.0000186-0.0000576 mg/L for pyridafol. In the ILV, the method LOD was determined to be 0.0015 mg/L (30% of the LOQ) for pyridate and pyridafol; the LODs were also calculated to be 0.00000112-0.00000270 mg/L for pyridate and 0.0000307-0.000121 mg/L for pyridafol.

II. Recovery Findings

ECM (MRID 50235205): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD \leq 20%) for analysis of pyridate and pyridafol at fortification levels of 0.005 mg/L (LOQ) and 0.05 mg/L (10 \times LOQ) in two water matrices (Tables 2-17, pp. 24-39). Two ion pair transitions were monitored for pyridate and pyridafol using LC/MS/MS in positive mode; the quantification and confirmation ion data was comparable. freshwater [pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected from a well *ca.* 40 m deep at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, and surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 14; Appendices 3-5, pp. 92-97).

ILV (MRID 50235206): Mean recoveries and RSDs were within guidelines for analysis of pyridate and pyridafol at fortification levels of 0.005 mg/L (LOQ) and 0.05 mg/L (10 \times LOQ) in two water matrices (Tables III-IV, pp. 32-33). Two ion pair transitions were monitored for pyridate and pyridafol using LC/MS/MS in positive mode; the quantification and confirmation ion data was comparable. The surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, and groundwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected in the aquatics lab (well water) at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (USDA water texture classification; p. 16; Appendix C, pp. 86-87). These were the same water matrices as those used in the ECM. Although the number of trials was not specified, the reviewer assumed that the ILV validated the ECM in the first trial with only insignificant modifications to the analytical equipment and parameters (pp. 18-19, 23; Figure 1, p. 38).

Table 2. Initial Validation Method Recoveries for Pyridate and Pyridafol in Water

Analyte	Fortification Level (mg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Freshwater¹						
Quantitation ion ²						
Pyridate	0.005 (LOQ)	5	99.9-106	104	2.59	2.49
	0.05	5	99.8-110	104	3.76	3.61
Pyridafol	0.005 (LOQ)	5	92.9-97.0	94.9	1.76	1.85
	0.05	5	97.9-99.7	99.1	0.722	0.729
Confirmation ion ²						
Pyridate	0.005 (LOQ)	5	104-110	107	2.12	1.98
	0.05	5	99.1-107	102	3.25	3.18
Pyridafol	0.005 (LOQ)	5	93.0-105	99.3	5.38	5.42
	0.05	5	96.3-99.2	98.2	1.16	1.19
Surface Water¹						
Quantitation ion ²						
Pyridate	0.005 (LOQ)	5	95.8-99.4	97.8	1.33	1.36
	0.05	5	96.1-102	99.2	2.33	2.35
Pyridafol	0.005 (LOQ)	5	98.4-106	102	2.95	2.90
	0.05	5	96.8-101	99.5	2.08	2.09
Confirmation ion ²						
Pyridate	0.005 (LOQ)	5	92.5-99.9	97.2	2.82	2.90
	0.05	5	95.3-102	97.1	4.06	4.18
Pyridafol	0.005 (LOQ)	5	92.2-105	98.3	4.78	4.86
	0.05	5	92.3-100	95.6	3.34	3.49

Data (uncorrected recovery results; p. 20) were obtained from Tables 2-17, pp. 24-39 of MRID 50235205.

1 The freshwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected from a well *ca.* 40 m deep at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, and surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 14; Appendices 3-5, pp. 92-97).

2 Two ion pair transitions were monitored (quantitation and confirmation, respectively): *m/z* 379.100→207.100 and *m/z* 379.100→351.300 for pyridate and *m/z* 207.000→104.100 and *m/z* 207.000→126.100 for pyridafol.

Table 3. Independent Validation Method Recoveries for Pyridate and Pyridafol in Water

Analyte	Fortification Level (mg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Surface Water¹						
Quantitation ion ²						
Pyridate	0.005 (LOQ)	5	89-98	93	4.9	5.3
	0.05	5	106-118	112	4.3	3.9
Pyridafol	0.005 (LOQ)	5	100-106	104	2.9	2.8
	0.05	5	104-108	107	1.8	1.7
Confirmation ion ²						
Pyridate	0.005 (LOQ)	5	87-93	90	2.4	2.7
	0.05	5	101-116	110	5.5	5.0
Pyridafol	0.005 (LOQ)	5	94-104	100	4.1	4.2
	0.05	5	106-110	108	1.8	1.7
Ground Water¹						
Quantitation ion ²						
Pyridate	0.005 (LOQ)	5	86-91	88	2.1	2.4
	0.05	5	95-110	106	6.3	5.9
Pyridafol	0.005 (LOQ)	5	97-112	103	6.0	5.8
	0.05	5	98-107	102	3.4	3.4
Confirmation ion ²						
Pyridate	0.005 (LOQ)	5	81-85	83	1.8	2.2
	0.05	5	93-109	104	6.3	6.1
Pyridafol	0.005 (LOQ)	5	96-110	104	5.4	5.2
	0.05	5	96-109	103	4.8	4.6

Data (recovery results were corrected when residues were quantified in the controls; pp. 20-21) were obtained from Tables III-IV, pp. 32-33 of MRID 50235206.

- The surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, and groundwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected in the aquatics lab (well water) at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 16; Appendix C, pp. 86-87). These were the same water matrices as those used in the ECM.
- Two ion pair transitions were monitored (quantitation and confirmation, respectively): m/z 379.1→207.1 and m/z 379.1→351.3 for pyridate and m/z 207.0→104.1 and m/z 207.0→126.1 for pyridafol. These transitions were the same as those of the ECM.

III. Method Characteristics

In the ECM and ILV, the LOQ was 0.005 mg/L for pyridate and pyridafol in water (p. 18 of MRID 50235205; pp. 24-25 of MRID 50235206). In the ECM, the LOQ was reported as the lowest nominal concentration in a fortified sample at which the methodology has been validated and a mean recovery of 70-120% with a relative standard deviation of <20% has been obtained. The LOQ was reported in the ILV from the ECM without justification. No calculations were provided for the LOQ in the ECM or ILV. In the ECM and ILV, the LOD was calculated for each analyte/matrix using the following equation:

$$\text{LOD} = \{[\text{lowest calibrant concentration (mg/L)}]/[(3 \times \text{signal to noise ratio})]\} \times \text{dilution factor}$$

Where, the dilution factor = 2 and the lowest calibrant concentration is 0.001 mg/L.

In the ECM, the LOD was calculated to be 0.00000123-0.00000372 mg/L for pyridate and 0.0000186-0.0000576 mg/L for pyridafol. In the ILV, the method LOD was determined to be 0.0015 mg/L (30% of the LOQ) for pyridate and pyridafol; the LODs were also calculated to be 0.00000112-0.00000270 mg/L for pyridate and 0.0000307-0.000121 mg/L for pyridafol. The calculated LODs supported the method LOD.

The method detection limit (MDL) was calculated in the ECM and defined as the standard deviation of the measured concentrations in the lowest matrix fortification samples multiplied by the student's t constant corresponding to 10 replicates subtracting 1 degree of freedom (p. 18 of MRID 50235205). The MDLs for pyridate were 0.000608 and 0.000915 mg/L for the primary and confirmatory transitions, respectively. The MDLs for pyridafol were 0.000694 and 0.000782 mg/L for the primary and confirmatory transitions, respectively.

Table 4. Method Characteristics for Pyridate and Pyridafol in Water

		Pyridate	Pyridafol	
Limit of Quantitation (LOQ)	ECM	0.005 mg/L		
	ILV			
Limit of Detection (LOD)	ECM	Freshwater	0.0000151 mg/L (Q) 0.0000372 mg/L (C)	0.0000223 mg/L (Q) 0.0000393 mg/L (C)
		Surface water	0.0000123 mg/L (Q) 0.0000363 mg/L (C)	0.0000186 mg/L (Q) 0.0000576 mg/L (C)
	ILV	Method	0.0015 mg/L (30% of the LOQ)	
		Groundwater	0.0000129 mg/L (Q) 0.0000270 mg/L (C)	0.000121 mg/L (Q) 0.0000386 mg/L (C)
		Surface water	0.0000112 mg/L (Q) 0.0000220 mg/L (C)	0.0000654 mg/L (Q) 0.0000307 mg/L (C)
	Linearity (calibration curve r^2 and concentration range) ¹	ECM	Freshwater	$r^2 = 0.9976$ (Q) $r^2 = 0.9972$ (C)
Surface water			$r^2 = 0.9981$ (Q) $r^2 = 0.9987$ (C)	$r^2 = 0.9985$ (Q) $r^2 = 0.9970$ (C)
ILV		Groundwater	$r^2 = 0.9976$ (Q) $r^2 = 0.9961$ (C)	$r^2 = 0.9987$ (Q) $r^2 = 0.9984$ (C)
		Surface water	$r^2 = 0.9944$ (Q) $r^2 = 0.9954$ (C)	$r^2 = 0.9994$ (Q) $r^2 = 0.9999$ (C)
Range		1-50 ng/mL		
Repeatable		ECM ²	Yes at LOQ and 10×LOQ. (two characterized water matrices)	
	ILV ^{3,4}			
Reproducible		Yes at LOQ and 10×LOQ.		
Specific	ECM	Yes, no matrix interferences were observed.	Yes, no matrix interferences were observed, but some minor baseline noise was observed near LOQ analyte peak in freshwater.	
	ILV	Yes, matrix interferences were <i>ca.</i> 13-19% (Q) and <i>ca.</i> 17-25% (C) ⁴ of the LOQ (based on recovery values). Peak tailing observed.	Yes, no matrix interferences were observed, but baseline was elevated.	

Data were obtained from p. 18 (LOQ/LOD); Tables 2-17, pp. 24-39 (recovery results); p. 17; Figures 2-3, pp. 43-44; Figures 11-12, pp. 52-53; Figures 20-21, pp. 61-62; Figures 29-30, pp. 70-71 (calibration curve); Figures 4-37, pp. 45-78 (chromatograms) of MRID 50235205; pp. 24-25 (LOQ/LOD); Tables III-IV, pp. 32-33 (recovery results); Table I, p. 30; Figures 2-3, pp. 39-40; Figures 11-12, pp. 48-49; Figures 20-21, pp. 57-58; Figures 29-30, pp. 66-67 (calibration curve); Figures 4-37, pp. 41-74 (chromatograms) of MRID 50235206. Q = quantitation ion transition; C = confirmation ion transition. All results reported for Q and C ions unless specified otherwise.

1 In the ECM, the freshwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected from a well *ca.* 40 m deep at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, and surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 14; Appendices 3-5, pp. 92-97 of MRID 50235205).

2 In the ILV, the surface water [SFW-TL-080718; pH 7.5, hardness 61 mg equiv. CaCO₃/L] collected from the surface of Tuckahoe Lake, Ridgely, Maryland, and groundwater [GRW-WL-080718; pH 8.2, hardness 153 mg equiv. CaCO₃/L] collected in the aquatics lab (well water) at the Brooks Drive facility of EAG Laboratories, Easton, Maryland, were used in the study and characterized by Agvise Laboratories, Northwood, North Dakota (p. 16; Appendix C, pp. 86-87 of MRID 50235206). These were the same water matrices as those used in the ECM.

3 Although the number of trials was not specified, the reviewer assumed that the ILV validated the ECM in the first trial with only insignificant modifications to the analytical equipment and parameters (pp. 18-19, 23; Figure 1, p. 38 of MRID 50235206).

4 A confirmatory method is not usually required when LC/MS or GC/MS is used as the primary method with which to generate study data.

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

IV. Method Deficiencies and Reviewer's Comments

1. ILV linearity was not satisfactory for the surface water quantitation ion transition analysis of pyridate ($r^2 = 0.9944$; Figure 2, p. 39 of MRID 50235206). Linearity is satisfactory when $r^2 \geq 0.995$.
2. The number of trials was not specified; the reviewer assumed that the ILV validated the ECM in the first trial with only insignificant modifications to the analytical equipment and parameters (p. 23 of MRID 50234205). OCSPP guidelines state that the ILV should be able to validate the ECM within three trials.
3. It could not be determined if the ILV was provided with the most difficult matrices with which to validate the method since the ECM water matrices were the same as those of the ILV.
4. The specificity of the method was not well-supported for pyridate by the ILV representative quantitation ion transition chromatograms because matrix interferences were *ca.* 13-19% (Q) and *ca.* 17-25% (C) of the LOQ (based on recovery values; Tables III-IV, pp. 32-33 of MRID 50235206). For the quantitation ion analysis chromatograms, the matrix interference of *ca.* 19% was greater than 50% of the method LOD.
5. The communications between the ECM and ILV were reportedly limited to email exchange regarding delivery of method and test materials, as well as clarification of matrix fortification solutions (p. 22; Appendix E, p. 98 of MRID 50235206). The ECM study personnel were not included in contact recipients.
6. In the ILV, it was determined that residual pyridafol was detected in pyridate fortified surface or groundwater samples confirming that no hydrolysis of pyridate occurred during the extraction and analysis for this ILV (p. 10 of MRID 50235206).
7. 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. 18 of MRID 50235205; pp. 24-25 of MRID 50235206). In the ECM, the LOQ was reported as the lowest nominal concentration in a fortified sample at which the methodology has been validated and a mean recovery of 70-120% with a relative standard deviation of <20% has been obtained. The LOQ was reported in the ILV from the ECM without justification. No calculations were provided for the LOQ in the ECM or ILV. In the ECM and ILV, the LOD was calculated for each analyte/matrix using the following equation: $LOD = \{[\text{lowest calibrant concentration (mg/L)}] / [3 \times \text{signal to noise ratio}]\} \times \text{dilution factor}$, where, the dilution factor = 2 and the lowest calibrant concentration is 0.001 mg/L. Detection limits should

not be based on arbitrary values.

8. In the ILV, the time required to complete the extraction of two sets of 19 samples (one reagent blank, two matrix controls, ten fortified samples, and six matrix-matched calibrants) was reported as *ca.* 11 hours (p. 22 of MRID 50235206).

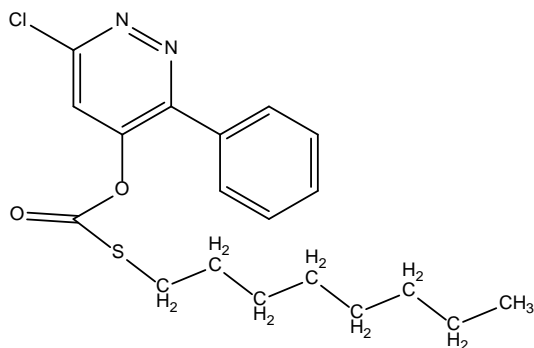
V. References

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.

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: c1ccccc1c2nnc(Cl)cc2OC(=O)SCCCCCCCC

**Pyridafol (CL 9673)**

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

