#### Analytical method for boscalid (BAS 510 F) in sediment

**Reports:** ECM: EPA MRID No.: 50785704. Andrews, R.S. 2017. Method for

Determination of BAS 510 F Residues in Sediment with Limit of

Determination (LOD) Calculation (Method D1706/01). BASF Registration Document No.: 2017/7017387. Report prepared, sponsored and submitted by BASF Corporation, Research Triangle Park, North Carolina; 128 pages.

Final report issued December 4, 2017.

ILV: EPA MRID No. 50785705. Sheng, L. 2017. Independent Laboratory Validation of BASF Analytical Method D1706/01: "Method for the determination of BAS 510 F (Reg. No. 300355) in sediment by LC-MS/MS". BASF Study ID No.: 828759. BASF Registration Document No.: 2017/7015827. EPL Study ID No.: 137G1603. Report prepared by EPL Bio Analytical Services (EPL), Niantic, Illinois, sponsored and submitted by BASF Corporation, Research Triangle Park, North Carolina; 53 pages. Final

report issued October 30, 2017.

**Document No.:** MRIDs 50785704 & 50785705

**Guideline:** 850.6100

**Statements:** ECM: The study was not conducted in accordance with Good Laboratory

Practice (GLP) standards since it was not a study (p. 3 of MRID 50785704).

Signed and dated No Data Confidentiality and GLP statements were

provided; Quality Assurance and Authenticity statements were not provided (pp. 2-3). The ECM was a compilation of the study reports for an analytical method and LOD determination (p. 5). These two study reports (BASF Study # 828758 and 828758\_1) were conducted in accordance with USEPA FIFRA GLP standards, 40 CFR, Part 160 (Appendix A, p. 10; Appendix B, p. 102). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Authenticity statements were provided (Appendix A, pp. 9-12;

Appendix B, pp. 101-104).

ILV: The study was conducted in accordance with USEPA FIFRA GLP standards, 40 CFR, Part 160 (p. 3 of MRID 50785705). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Authenticity statements

were provided (pp. 2-5).

**Classification:** This analytical method is classified as supplemental. The limit of

quantitation was based on the lowest fortification level.

**PC Code:** 128008

**Reviewer:** Taimei Harris, Ph.D.,

Chemist Signature: Harris by Harris, Taimei Date: Taimei 2019/1016

**CDM/CSS-** Lisa Muto, M.S.,

Dynamac JV Environmental Scientist Signature: July Muto
Reviewers:

Date: 11/07/2018

Mary Samuel, M.S., Environmental Scientist

Signature:

Date:

11/14/2019

**EPA Reviewer:** Katrina White, Ph.D. Senior Scientist

KATRINA WHITE Digitally signed by KATRINA WHITE Date: 2019.10.17 07:45:42 -04'00'

# <u>Page numbers cited for MRID 50785704 refer to those appearing on the bottom, right-hand corner of the document.</u>

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, BASF Method D1706/01, is designed for the quantitative determination of boscalid (BAS 510 F) in sediment at the LOQ of 0.005 mg/kg using LC/MS. The LOQ is less than the lowest toxicological level of concern in sediment. The ECM performed BASF Method D1706/01 using two characterized sediments; two sediments were also used in the ILV, but they were not characterized or described in texture. It could not be determined if the ILV was provided with the most difficult matrix with which to validate the method and that the matrices covered the range of soils used in the Terrestrial Field Dissipation (TFD) studies. The ILV validated the ECM method for the quantitation and confirmation analyses of the analytes in the first trial with insignificant modifications to the analytical instruments and parameters, as well as one recommendation which was included in the submitted ECM; however, there was some documented communication between the individuals who conducted the ECM and the ILV. All ECM and ILV data regarding repeatability, accuracy, precision, linearity, and specificity were satisfactory for boscalid.

In a separate study, the method LOD of BASF Analytical Method D1717/01 (0.001 mg/kg) was validated in accordance with the methodology set forth in 40 CFR Ch. 1 Part 136 Appendix B.

Table 1. Analytical Method Summary								
	MRID							Limit of
Analyte(s) by Pesticide	Environmental Chemistry Method	Independent Laboratory Validation	EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Quantitation (LOQ)
Boscalid (BAS 510 F)	50785704 <sup>1</sup>	50785705		Sediment <sup>2,3</sup>	04/12/2017	BASF Corporation	LC/MS/MS	0.005 mg/kg

**Table 1. Analytical Method Summary** 

#### I. Principle of the Method

### BASF Method D1706/01

Sediment samples ( $5 \pm 0.1$  g) were fortified in glass containers (Appendix A, p. 21; Appendix A, Appendix B, pp. 38-41, 45 of MRID 50785704). The sample was extracted once with 50 mL of methanol: 0.098M sodium acetate (aq): 0.102M acetic acid (aq), (80:10:10, v:v:v), via shaking at 300 rpm for 60 minutes. An aliquot (ca. 10 mL) was transferred to a culture tube and centrifuged (ca. 3500 rpm for 5 minutes). An aliquot (0.1 mL) of the supernatant was transferred to a culture tube and diluted with 0.9 mL of water. The sample was diluted with methanol: 9.8mM sodium acetate (aq): 10.2mM acetic acid (aq)/water, (8:1:1:90, v:v:v:v), as needed, and filtered (0.45 µm PTFE filter – syringe filter may be used) and transferred to an LC vial for analysis.

Samples were analyzed for boscalid using a Waters Acquity UPLC system coupled to an AB Sciex API 6500 mass spectrometer (Appendix A, Tables 2-3, pp. 27-28; Appendix A, Appendix B, pp. 43-44 of MRID 50785704). The LC/MS conditions consisted of a Acquity BEH C18 column (50 x 2.1 mm, 1.7  $\mu$ m particle size; column temperature 50°C) with a mobile phase gradient of 0.1% formic acid in water and 0.1% formic acid in methanol [percent A:B (v:v) at 0.00-0.05 min. 75:25, 0.90 min. 55:45, 1.50-2.45 min. 5:95, 2.50-3.00 min. 75:25] and MS/MS detection in ESI positive ion mode (ionization temperature 550°C). Injection volume was 10-50  $\mu$ L. Two ion transitions were monitored (quantitation and confirmatory, respectively) as follows: m/z 343 $\rightarrow$ 307 and 343 $\rightarrow$ 271. Approximate expected retention time was 2.0 minutes.

In the ILV, the ECM was performed as written for boscalid, except for the use of a different LC/MS system (pp. 7, 13, 15-17 of MRID 50785705). Agilent 1290 HPLC system coupled to an AB Sciex API 6500 mass spectrometer was used. All LC/MS conditions were the same, except that the ionization temperature was 750°C and the injection volume was defined as 40  $\mu$ L (p. 18). Two ion transitions were monitored (quantitation and confirmatory, respectively) as follows:

<sup>1</sup> Compilation of two reports: BASF Method D1706/01 & LOD determination.

<sup>2</sup> In the ECM, Goose river clay loam sediment (Sample ID CM17-025; 35% sand, 36% silt, 29% clay; pH 7.8 in 1:1 soil:water ratio; 5.3% organic matter – Walkley Black) and Golden lake loamy sand sediment (Sample ID CM17-026; 83% sand, 12% silt, 5% clay; pH 7.8 in 1:1 soil:water ratio; 2.1% organic matter – Walkley Black) were used in the study (USDA soil texture classification; Appendix A, p. 20; Appendix A, Appendix K, pp. 88-94 of MRID 50785704). Both matrices were collected from Grand Forks, North Dakota and characterized at Agvise Laboratories, Northwood, North Dakota.

<sup>3</sup> In the ILV, Goose river sediment and Golden lake sediment were provided by the Sponsor (BASF) and used in the study (p. 13 of MRID 50785705). The sediments were not characterized, and the soil texture was not reported.

m/z 342.8 $\rightarrow$ 307.2 and 342.8 $\rightarrow$ 270.9. Approximate expected retention time was 1.95 minutes. These parameters were very similar to those of the ECM. No other modifications to the ECM were reported. The ILV reported one recommendation which was to give a weight range for the sample, e.g. 5  $\pm$  0.1 g (pp. 23-24). This recommendation was incorporated into the ECM which was submitted.

The Limit of Quantification (LOQ) and Limit of Detection (LOD) for boscalid in sediment were 0.005 mg/kg and 0.001 mg/kg, respectively, in the ECM and ILV (Appendix A, Table 3, p. 28 of MRID 50785704; pp. 22-23 of MRID 50785705).

## Methodology to Evaluate MDL and LOD

Evaluation of LOD of BASF Analytical Method No. D1706/01 required the experimental determination of MDL as defined by 40 CFR Ch.1 Part 136 appendix B. A brief description of the methodology to determine MDL is as follows (Appendix B, pp. 109-110 of MRID 50785704):

- 1. Standards containing all analytes were injected using the LC-MS/MS parameters from Method D1706/01. All transitions were monitored according to the method, including those set forth for the secondary chromatographic techniques. The least sensitive transition of the method was determined qualitatively through visual inspection of factors such as peak height, relative background level, area count, etc. Matrix-fortified control samples were created, and an estimation was made to what level a sample in matrix would produce a S/N of 2-10.
- 2. Using BASF Analytical Method No. D1706/01, seven control samples (7) control samples (5 g) were extracted with methanol/sodium acetate buffer. An aliquot of the extract was transferred to a culture tube and centrifuged. An aliquot (0.1 mL) of the supernatant from the extract was diluted with a calibration standard (water, 0.1 mL) and additional water (0.8 mL) to an appropriate concentration level to make the post-extraction fortified control samples for LOD determination were injected on the LC-MS/MS with bracketing neat standards for quantitation.
- 3. Using the standard curve to calculate the concentrations of the seven matrix-spiked samples, the results are put into the equation:  $MDL = S \times t_{(N-1,1-\infty=.99)}$ , where

MDL = Method detection limit S = Standard deviation of the matrix-spiked sample set concentrations  $t_{(N-1,1-\infty=.99)} = Critical t$  value from a student t-test table at 99% confidence

The acceptance criteria for the MDL calculation was:

- a. The calculated MDL must be able to be seen on the instrument with S/N of  $\geq 2$ .
- b. The concentration of the matrix-spiked samples must be no greater than 10X the calculated MDL.

If either of the above to criteria were not met, the experiment had to be repeated at a

higher or lower spiking concentration, respectively, until all criteria are met. Reinjection of the samples with lower or higher injection volumes could be done in place of preparing new matrix-spiked samples as it changes the amount (pg) injected on the column the same.

4. A matrix-spiked sample at the calculated MDL was injected on the LC-MS/MS in triplicate (no standard curve is required) to verify the MDL can be seen at  $\geq 2$  S/N.

Based on an evaluation of the susceptibility of the analyte of interest to instrument variability, LC-MS/MS drift, unexpected contamination, and untested matrix effects, the MDL was raised to an appropriate value that will mitigate the anticipated issues. This new value is the LOD.

#### **II. Recovery Findings**

ECM (MRID 50785704): Mean recoveries and relative standard deviations (RSDs) were within guideline requirements (mean 70-120%; RSD ≤20%) for analysis of boscalid in sediment matrices at fortification levels of 0.005 mg/kg (LOQ) and 0.05 mg/kg (10×LOQ; Appendix A, Table 1, p. 26). Boscalid was identified using two ion transitions; performance data (recovery results) from primary and confirmatory analyses were comparable. Goose river clay loam sediment (Sample ID CM17-025; 35% sand, 36% silt, 29% clay; pH 7.8 in 1:1 soil:water ratio; 5.3% organic matter – Walkley Black) and Golden lake loamy sand sediment (Sample ID CM17-026; 83% sand, 12% silt, 5% clay; pH 7.8 in 1:1 soil:water ratio; 2.1% organic matter – Walkley Black) were used in the study (USDA soil texture classification; Appendix A, p. 20; Appendix A, Appendix K, pp. 88-94). Both matrices were collected from Grand Forks, North Dakota and characterized at Agvise Laboratories, Northwood, North Dakota.

ILV (MRID 50785705): Mean recoveries and RSDs were within guideline requirements for analysis of boscalid in sediment matrices at fortification levels of 0.005 mg/kg (LOQ) and 0.05 mg/kg (10×LOQ; Table 1, p. 21). Boscalid was identified using two ion transitions. Performance data (recovery results) from primary and confirmatory analyses were comparable; however, less comparability was seen at the LOQ fortification level. Recovery results were corrected for residues quantified in the controls; residues in the controls ranged *ca.* 1-17% of the LOQ (based on quantified residues; pp. 19-20; Appendix C, pp. 45-48). Goose river sediment and Golden lake sediment were provided by the Sponsor (BASF) and used in the study (p. 13). The sediments were not characterized, and the soil texture was not reported. The ILV validated BASF Method D1706/01 in the first trial with insignificant modifications to the analytical instrumentation and parameters, as well as one recommendation which was included in the submitted ECM (pp. 8, 23-24).

Table 2. Initial Validation Method Recoveries for Boscalid (BAS 510 F) in Sediment<sup>1,2</sup>

Analyte	Fortification Level (ppb)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)		
	Goose River Clay Loam Sediment							
	Quantitation ion transition							
Boscalid	0.005 (LOQ)	5	87-96	90	4	4		
(BAS 510 F)	0.05	5	80-87	84	3	3		
	Confirmatory ion transition							
Boscalid	0.005 (LOQ)	5	84-89	87	2	2		
(BAS 510 F)	0.05	5	82-87	85	2	3		
		Golden Lake Loamy Sand Sediment						
	Quantitation ion transition							
Boscalid	0.005 (LOQ)	5	82-90	85	3	4		
(BAS 510 F)	0.05	5	75-87	84	5	6		
	Confirmatory ion transition							
Boscalid	0.005 (LOQ)	5	85-87	86	1	1		
(BAS 510 F)	0.05	5	83-84	84	1	1		

Data (uncorrected recovery results, Appendix A, Appendix C, p. 49; Appendix A, Appendix F, pp. 58-63) were obtained from Appendix A, Table 1, p. 26 of MRID 50785704.

<sup>1</sup> The Goose river clay loam sediment (Sample ID CM17-025; 35% sand, 36% silt, 29% clay; pH 7.8 in 1:1 soil:water ratio; 5.3% organic matter – Walkley Black) and Golden lake loamy sand sediment (Sample ID CM17-026; 83% sand, 12% silt, 5% clay; pH 7.8 in 1:1 soil:water ratio; 2.1% organic matter – Walkley Black) were used in the study (USDA soil texture classification; Appendix A, p. 20; Appendix A, Appendix K, pp. 88-94). Both matrices were collected from Grand Forks, North Dakota and characterized at Agvise Laboratories, Northwood, North Dakota.

<sup>2</sup> Two ion transitions were monitored (quantitation and confirmatory, respectively) as follows: m/z 343 $\rightarrow$ 307 and 343 $\rightarrow$ 271.

Table 3. Independent Validation Method Recoveries for Boscalid (BAS 510 F) in Sediment  $^{1,2}$ 

Analyte	Fortification Level (ppb)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)	
	Goose River Sediment						
	Quantitation ion transition						
Boscalid	0.005 (LOQ)	5	71.2-85.8	76.5	6.69	8.75	
(BAS 510 F)	0.05	5	66.2-78.9	75.5	5.26	6.97	
	Confirmatory ion transition						
Boscalid	0.005 (LOQ)	5	76.8-114	88.9	14.7	16.5	
(BAS 510 F)	0.05	5	66.6-79.7	75.5	5.14	6.80	
	Golden Lake Sediment						
	Quantitation ion transition						
Boscalid	0.005 (LOQ)	5	68.4-77.7	73.2	3.65	4.98	
(BAS 510 F)	0.05	5	71.7-86.3	81.0	5.54	6.83	
	Confirmatory ion transition						
Boscalid	0.005 (LOQ)	5	82.4-104	91.1	8.24	9.04	
(BAS 510 F)	0.05	5	75.9-88.3	82.7	4.44	5.38	

Data (recovery results were corrected for residues quantified in the controls, pp. 19-20; Appendix C, pp. 45-48) were obtained from Table 1, p. 21 of MRID 50785705.

#### III. Method Characteristics

The LOQ and LOD for boscalid in sediment were 0.005 mg/kg and 0.001 mg/kg (20% of the LOQ), respectively, in the ECM and ILV (Appendix A, pp. 14, 25; Appendix A, Table 3, p. 28; Appendix B, p. 108 of MRID 50785704; pp. 22-23 of MRID 50785705). In the ECM, the LOQ was defined as the lowest fortification level tested of an analyte in the matrix, before extraction, and is determined by the proposed tolerance. In the ECM and ILV, the LOQ was defined as the lowest fortification level successfully tested. In the ECM, the LOD was defined as the lowest fortification level that can be reliably brought through the method and quantitated, as well as 20% of the LOQ. It was also noted that the LOD is above the MDL. In the ILV, the LOD was defined as the absolute amount of analyte injected into the LC/MS parameters using the lowest calibration standard.

#### Results of Evaluation of the LOD for Method D1513/01

In the ECM, the LOD was calculated to validate the method LOD using the methodology reported above. The calculation of MDL for BAS 510 F was conducted according to the table provided in 40 CFR Ch. 1 Part 136 appendix B (Appendix B, pp. 111-112 of MRID 50785704). The MDL was calculated to be 0.0503 pg on-column for BAS 510 F. Based on this calculated MDL, the LOD for BAS 510 F was set at 0.2 pg on-column (*i.e.* 0.01 ng/mL injected at 0.02 mL [20 µL]). The calculated LOD was equivalent to 0.001 mg/kg in the sediment samples.

<sup>1</sup> The Goose river sediment and Golden lake sediment were provided by the Sponsor (BASF) and used in the study (p. 13). The sediments were not characterized, and the soil texture was not reported.

<sup>2</sup> Two ion transitions were monitored (quantitation and confirmatory, respectively) as follows: m/z 342.8 $\rightarrow$ 307.2 and 342.8 $\rightarrow$ 270.9.

**Table 4. Method Characteristics** 

Analyte		Boscalid (BAS 510 F)				
Limit of Quantitation (LOQ)	ECM ILV	0.005 mg/kg				
Limit of Detection (LOD)	ECM	0.001 mg/kg (20% of LOQ)				
		0.2 pg on-column (i.e. 0.01 ng/mL injected at 0.02 mL [20 μL])				
	ILV	0.001 mg/kg (20% of LOQ)				
		$r^2 = 0.9992 (Q)$				
	ECM	$r^2 = 0.9998$ (C)				
Linearity (calibration		(0.01-0.50 ng/mL)				
curve r <sup>2</sup> and	ILV	$r^2 = 0.99970 (Q)$				
concentration range) <sup>1</sup>		$r^2 = 0.99980 (C)$				
		(0.0101-1.010 ng/mL)				
Repeatable	ECM <sup>2</sup>	Yes at LOQ and 10×LOQ				
		(two characterized sediments)				
	ILV <sup>3,4</sup>	Yes at LOQ and 10×LOQ				
	ILV <sup>3</sup> ,	(two uncharacterized sediments)				
Reproducible		Yes at LOQ and 10×LOQ				
Specific	ECM	Yes, no matrix interferences were observed in Q (ca. 10% of LOQ in C,				
		based on peak area). Some non-uniform peak integration was noted. A				
		significant peak at RT ca. 2.5 was observed in all Q chromatograms.				
	ILV	Yes, matrix interferences were <i>ca.</i> 1-5% (Q) and <i>ca.</i> 4-17% (C) of the LOQ (based on quantified residues).				
	1: 4	LOQ (based on quantified residues).				

Data were obtained from Appendix A, pp. 14, 25; Appendix A, Table 3, p. 28; Appendix B, p. 108, 111-112 (LOQ/LOD); Appendix A, Table 1, p. 26 (recovery data); Appendix A, Appendix H, pp. 68-69 (calibration curves); Appendix A, Appendix I, pp. 78-84 (chromatograms) of MRID 50785704; pp. 22-23 (LOQ/LOD); Table 1, p. 21 and Appendix C, pp. 45-48 (recovery data); Appendix A, p. 27 and Appendix B, p. 36 (calibration curves); Appendix A, pp. 31-34 and Appendix B, pp. 40-43 (chromatograms) of MRID 50785705; DER Attachment 2. Q = Quantitation ion transition; C = Confirmatory ion transition.

- 1 ECM correlation coefficients (r²) values were reviewer-calculated from r values provided in the study report (Appendix A, Appendix H, pp. 68-69 of MRID 50785704; DER Attachment 2). For the ECM and ILV, calibration curves were solvent-based.
- 2 In the ECM, Goose river clay loam sediment (Sample ID CM17-025; 35% sand, 36% silt, 29% clay; pH 7.8 in 1:1 soil:water ratio; 5.3% organic matter Walkley Black) and Golden lake loamy sand sediment (Sample ID CM17-026; 83% sand, 12% silt, 5% clay; pH 7.8 in 1:1 soil:water ratio; 2.1% organic matter Walkley Black) were used in the study (USDA soil texture classification; Appendix A, p. 20; Appendix A, Appendix K, pp. 88-94 of MRID 50785704). Both matrices were collected from Grand Forks, North Dakota and characterized at Agvise Laboratories, Northwood, North Dakota.
- 3 In the ILV, Goose river sediment and Golden lake sediment were provided by the Sponsor (BASF) and used in the study (p. 13 of MRID 50785705). The sediments were not characterized, and the soil texture was not reported.
- 4 The ILV validated BASF Method D1706/01 in the first trial with insignificant modifications to the analytical instrumentation and parameters, as well as one recommendation which was included in the submitted ECM (pp. 8, 23-24 of MRID 50785705).

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

- 1. Communications between the ILV Study Director and BASF Study Monitor (R. Stephen Andrews) were reported and demonstrated that technical guidance was provided by the BASF Study Monitor to the ILV study author (Li Sheng; p. 1; Appendix A, p. 8; Appendix B, p. 100 of MRID 50785704; pp. 1, 24; Appendix E, pp. 52-53 of MRID 50785705). In the Communications, Li Sheng requested study protocol clarification and report preparation advice from Robert Stephen Andrews.
- 2. It could not be determined if the ILV was provided with the most difficult matrix with which to validate the method since the ILV sediments were not characterized and the soil texture was not reported. Goose river sediment and Golden lake sediment were provided by the Sponsor (BASF) and used in the study (p. 13 of MRID 50785705).
- 3. The estimation of LOQ in ECM and ILV was not based on procedures as defined in 40 CFR Part 136 (Appendix A, pp. 14, 25; Appendix A, Table 3, p. 28; Appendix B, pp. 108, 111-112 of MRID 50785704; pp. 22-23 of MRID 50785705). In the ECM, the LOQ was defined as the lowest fortification level tested of an analyte in the matrix, before extraction, and is determined by the proposed tolerance. In the ECM and ILV, the LOQ was defined as the lowest fortification level successfully tested. In the ECM, the LOD was defined as the lowest fortification level that can be reliably brought through the method and quantitated, as well as 20% of the LOQ. It was also noted that the LOD is above the method detection limit. In the ILV, the LOD was defined as the absolute amount of analyte injected into the LC/MS parameters using the lowest calibration standard. No calculations were reported to justify the LOD for the method in the ILV. Detection limits should not be based on arbitrary values.

The ECM noted that the LOQ of the method covers the lowest relevant eco-toxicology endpoint for sediment and/or relevant environmental compartment (Appendix A, p. 14 of MRID 50785704).

- 4. The reviewer noted that soil matrices were not included in the method validation.
- 5. The storage stability was investigated by the ECM (Appendix A, p. 23; Appendix A, Table 2, p. 27; Appendix A, Appendices D-E, pp. 51-56 of MRID 50785704). The calibration standards in methanol: 9.8mM sodium acetate (aq): 10.2mM acetic acid (aq)/water (8:1:1:90, v:v:v:v) were determined to be stable for at least 1 month when stored under refrigeration (<5°C). The stock and fortification solutions in methanol were determined to be stable for up to 118 days under refrigeration (<5°C) in a separate study. The stabilities of boscalid sample extracts under refrigeration (<5°C) were determined to be 6 days for both sediments.
- 6. The matrix effects were found to be insignificant ( $< \pm 20\%$ ) for boscalid in the ECM and ILV (Appendix A, p. 23 of MRID 50785704; p. 23 of MRID 50785705).
- 7. Some non-uniform peak integration was noted in the ECM.

- 8. ILV recovery results were corrected for residues quantified in the controls; residues in the controls ranged *ca.* 1-17% of the LOQ (based on quantified residues; pp. 19-20; Appendix C, pp. 45-48)
- 9. It was reported for the ILV that one sample set (e.g. total 13 samples) required *ca*. 4-8 hours of work (p. 22 of MRID 50785705). It was reported for the ECM that one sample set (e.g. total 13 samples) required about 8 hours of work (Table 3, p. 28 of MRID 50785704).

#### 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**

Boscalid (BAS 510 F; Reg. No. 300355)

**IUPAC Name:** 2-Chloro-N-(4'-chlorobiphenyl-2-yl)nicotinamide

CAS Name: Not reported
CAS Number: 188425-85-6
SMILES String: Not found