

Addendum No. 1 to MRIDs # 43564901 & 49098001

DER Study Titles: ECM: GAS CHROMATOGRAPHIC DETERMINATION OF DICHLOBENIL AND 2,6-DICHLOROBENZAMIDE IN SOIL.

ILV: Validation of an Analytical Method for Determination of Dichlobenil and its Metabolite 2,6-Dichlorobenzamide (BAM) in Soil by GC/ECD, Demonstrating GC/MS as Confirmatory Method.

Guideline Number: 850.6100

- Reasons for changes:
  - Downgrade of study classification for dichlobenil to **unacceptable**.
    - The ECM and ILV reports are unacceptable for both dichlobenil and BAM because the numbers of sample replicates were inadequate for both dichlobenil and BAM and the reports lacked sufficient raw data and example chromatographs. In the ILV, five replicate samples were analyzed by GC/ECD with acceptable recoveries for both dichlobenil and BAM. However, GC/ECD analysis requires confirmation, unlike GC/MS. Only one replicate sample was analyzed by GC/MS, which is insufficient for the confirmation evaluation.
    - To upgrade the unacceptable study classification for both dichlobenil and BAM, a separate ILV report is needed with at least 5 replicates at LOQ and 10x LOQ for the initial analysis and also for any needed confirmatory analysis, with acceptable recoveries, and with guideline-compliant reporting (*i.e.*, sufficient raw data and example chromatographs provided). If the analysis is conducted with GC/MS, a confirmatory analysis is not needed.

Revised by: Greg Orrick

Date: 5-28-15

Secondarily reviewed by: R. David Jones

Date: 5-28-15

**Analytical method for dichlobenil and its metabolite 2,6-dichlorobenzamide in soil**

- Reports:** ECM: MRID No. 43564901 (Appendix E, pp. 40-45). Pouwelse, A.V., T.M.W. van Loo. 1991. GAS CHROMATOGRAPHIC DETERMINATION OF DICHLOBENIL AND 2,6-DICHLOROBENZAMIDE IN SOIL. Laboratory Instructions RES 051. Lab. ID: DUPHAR 56630/87/91. Report prepared, sponsored and submitted by DUPHAR B.V., Analytical Development Dept., Weesp, The Netherlands; 6 pages (ECM); 76 pages (study). ECM dated May 3, 1991 and issued May 17, 1991; study issued June 12, 1991 (p. 10).  
ILV: MRID No. 49098001. Bacher, R. 2002. Validation of an Analytical Method for Determination of Dichlobenil and its Metabolite 2,6-Dichlorobenzamide (BAM) in Soil by GC/ECD, Demonstrating GC/MS as Confirmatory Method. PTRL Study No: P 556 G; Uniroyal Study No. C.201.62.029; 2002-001. Report prepared by PTRL Europe GmbH Helmholtzstr, Ulm, Germany; sponsored by Uniroyal Chemical Europe B.V., Amsterdam, The Netherlands; and submitted by Chemtura Corporation, Middlebury, Connecticut (p. 3); 80 pages. Final report issued July 23, 2001.
- Document No.:** MRIDs 43564901 & 49098001
- Guideline:** 850.6100
- Statements:** ECM: For the ECM Laboratory Instructions, no documents or statements were provided. The MRID study was conducted in compliance with OECD Good Laboratory Practices (p. 3). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4).  
ILV: The study was conducted in accordance with German and OECD GLP (p. 3 and pp. 59-60). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Certification of Authenticity statements were provided (pp. 2-5).
- Classification:** This analytical method is classified as **supplemental** for dichlobenil and **unacceptable** for BAM. The number of samples was inadequate at the LOQ and 10×LOQ in the ECM (dichlobenil and BAM) and ILV (BAM). The confirmation method of the ILV used an insufficient number of samples (dichlobenil and BAM) and yielded unacceptable recovery for BAM at 10×LOQ. The LOQ is greater than the lowest toxicological level of concern in soil for BAM (monocot EC<sub>25</sub> = 6.3 µg/kg). Inadequate data were provided with the ECM report, including a lack of reported recovery results and all chromatograms. The ECM did not use a confirmation method.
- PC Code:** 027401
- Reviewer:** Gregory Orrick, Environmental Scientist  
USEPA
- Signature:**  
**Date:** Dec. 31, 2014

(All page citations refer to MRID 49098001 (ILV/ECM) unless otherwise noted.)

## Executive Summary

This analytical method, Laboratory Instructions RES 051, is designed for the quantitative determination of dichlobenil and its metabolite 2,6-dichlorobenzamide (BAM) in soil using GC/ECD. The method could not be evaluated based on the ECM (MRID 43564901) due to a lack of reported data and supporting raw data. Mean recoveries were not provided and could not be calculated for the LOQ; only one sample was reported for 10×LOQ. RSDs (reported as coefficients of variation) were only reported for analysis of dichlobenil and BAM in soil at the LOQ (n = 4), although these were satisfactory. Based on the ILV, the method was found to be quantitative for dichlobenil at the stated LOQ of 10 µg/kg; for BAM, an insufficient number of samples was included. The LOQ is similar to and appears to be less than the lowest toxicological level of concern in soil for dichlobenil (dicot EC<sub>25</sub> ≈ 12 µg/kg). However, the LOQ is greater than the lowest toxicological level of concern in soil for BAM (monocot EC<sub>25</sub> = 6.3 µg/kg). GC/MS and GC/MS/MS (dichlobenil only) analyses were included in the ILV for confirmation; however, only one fortification/analyte/analysis was provided.

**Table 1. Analytical Method Summary**

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Dichlobenil and Degradatae BAM	43564901	49098001		Soil	5/03/91	Chemtura Corporation/	GC/ECD GC/MS*	10 µg/kg

\*The ILV included GC/MS and GC/MS/MS (dichlobenil only) analyses for confirmation.

## I. Principle of the Method

Soil (50 g) was extracted with 17 mL of 0.2% NH<sub>4</sub>Cl aqueous solution and 150 mL of acetone/petroleum ether (1:1, v:v) via grinding for 5 minutes with an Ultra Turrax (4000 rpm; pp. 15-16; Appendix 11, pp. 75-79). After filtration through a Büchner funnel, the residue was washed with 30 mL of acetone/petroleum ether (1:1, v:v). The filtrate and wash were combined into a separatory funnel. Water (300 mL) and saturated sodium chloride solution (10 mL) were added. After shaking for 1 minute, the petroleum ether layer was collected through a fluted filter containing anhydrous sodium sulfate. The water layer was extracted three times with 100 mL of petroleum ether (shaking for 1 minute). All petroleum ether layers were passed through the same fluted filter containing anhydrous sodium sulphate as above. The combined petroleum ether layers were reduced to 30 mL at ambient temperature. The residue was transferred to a 50-mL volumetric flask; the volume was brought to 50 mL with petroleum ether. This extract contained the dichlobenil. BAM was partitioned from the water layer with ethyl acetate. The water layer was extracted three times with 100 mL of ethyl acetate. After shaking for 1 minute, each ethyl acetate layer was collected through a fluted filter containing anhydrous sodium sulfate. All ethyl acetate layers were combined and reduced to dryness (evaporation details not reported). The residue was reconstituted with 10.0 mL acetone/petroleum ether (1:1, v:v). An aliquot (5.0 mL) of the dichlobenil extract was purified by passing through a neutral alumina cartridge that was pre-conditioned with 5 mL of petroleum ether under vacuum. The cartridge with extract was washed with 1 mL of petroleum ether then eluted with 8 mL of 2% acetone/petroleum ether solution. The eluate was collected into a 10 mL flask that was brought to volume with 2%

acetone/petroleum ether solution prior to analysis. An aliquot (5.0 mL) of the BAM extract was purified by passing through a neutral alumina cartridge which was pre-conditioned with 5 mL of petroleum ether under vacuum. The cartridge with extract was washed twice with 5 mL of acetone/petroleum ether (1:9, v:v) then eluted with 5 mL of ethanol/petroleum ether (15:85, v:v). The eluate was reduced to dryness via evaporation (40°C) then reconstituted in 5.0 mL of ethyl acetate.

Analytes were identified by gas chromatography equipped with a  $^{63}\text{Ni}$  electron capture detector (GC/ECD; system not specified; Appendix 11, pp. 76-77, 79-80). The gas chromatography column was fused silica (length 30 m, i.d. 0.32 mm) coated with 0.25  $\mu\text{m}$  D3-17, which was coupled with an uncoated deactivated retention gap (length 30 cm, i.d. 0.53 mm) for dichlobenil and coupled with a retention gap coated with 0.05  $\mu\text{m}$  of OV-225 (CP Sil 43 CB, length 30 cm, i.d. 0.53 mm) for BAM. For dichlobenil, GC conditions were as follows: carrier gas pressure 100 kPa; flow 25 mL/min.; oven temp. 75°C; detector temp. 280°C; oven temp. increase 30°C/min until 180°C; injection volume 1  $\mu\text{L}$ ; and retention time *ca.* 5 minutes. For BAM, GC conditions were as follows: carrier gas pressure 125 kPa; flow 25 mL/min.; oven temp. 70°C; detector temp. 280°C; oven temp. increase 30°C/min until 180°C; injection volume 1  $\mu\text{L}$  and retention time *ca.* 11 minutes.

In the ILV, the extraction procedure for dichlobenil and BAM were the same as those in the ECM, aside from two insignificant adjustments (pp. 10, 15-16). As a confirmation of the identification of the analytes, GC/MS and GC/MS/MS (dichlobenil only) analyses were employed. Prior to GC/MS analysis, 5.0 mL of the dichlobenil extract used for GC/ECD analysis was concentrated under a gentle stream of nitrogen to *ca.* 0.3 mL. The volume of the residue was adjusted to 0.5 mL with petroleum ether. Likewise, 3.0 mL of the BAM extract used for GC/ECD analysis was concentrated under a gentle stream of nitrogen to *ca.* 0.4 mL. The volume of the residue was adjusted to 0.6 mL with ethyl acetate. The GC/ECD details indicated only one set of conditions for both analytes: CP-3380 GC with 1079 split/splitless-injector and electron capture detector; Varian Chrompack CP Sil 8 CB Low Bleed column (length 30 m, i.d. 0.32 mm, 0.25  $\mu\text{m}$  film thickness); carrier gas pressure 10 psi; oven temp. 70°C; oven temp. increase 20°C/min until 270°C; injection volume 1  $\mu\text{L}$ ; and retention times 7.4 minutes (dichlobenil) and 9.9 minutes (BAM; p. 17). GC/MS and GC/MS/MS conditions were as follows: 3400 GC with SPI injector; Varian Chrompack CP Sil 8 CB Low Bleed column (length 30 m, i.d. 0.32 mm, 0.25  $\mu\text{m}$  film thickness); carrier gas pressure 8 psi; oven temp. 70°C; oven temp. increase 20°C/min until 270°C; injection volume 3  $\mu\text{L}$ ; and retention times *ca.* 5.5 minutes (dichlobenil) and *ca.* 7.7 minutes (BAM; pp. 20-21). Ions monitored for dichlobenil were 171  $m/z$  (quantification) and 173  $m/z$  (confirmation) in the MS detection and 171  $m/z$  (parent) and 136  $m/z$  (daughter) in the MS/MS detection. Ions monitored for BAM were 173  $m/z$  (quantification), 175  $m/z$  (confirmation) and 189  $m/z$  (confirmation) in the MS detection (not analyzed by MS/MS detection).

The LOQ for both analytes was the same in the ECM and ILV (10  $\mu\text{g}/\text{kg}$ ; p. 19; Appendix 11, p. 80). The LOD for both analytes was determined to be 1  $\mu\text{g}/\text{kg}$  in the ECM and estimated to be 2  $\mu\text{g}/\text{kg}$  in the ILV.

## II. Recovery Findings

ECM (MRID 43564901): Reported relative standard deviations (RSDs; reported as coefficients of variation) met requirements ( $RSD \leq 20\%$ ) for analysis of dichlobenil and BAM in soil at the LOQ ( $n = 4$ ; pp. 18-19; Appendix E, p. 45 of MRID 43564901). Reported mean recoveries met requirements (mean 70-120%); however, results were only provided as overall means of all fortifications (0.01 to *ca.* 2 mg/kg) for each analyte. Mean recoveries were not provided and could not be calculated for the LOQ since only one recovery value was reported; mean recoveries and RSDs were not provided and could not be calculated for  $10 \times \text{LOQ}$  since  $n = 1$ . Reported recoveries ( $n = 1$ ) for 0.01 (LOQ), 0.02, 0.05, 0.10-0.11 ( $10 \times \text{LOQ}$ ), 0.51-0.54 and 2.11-2.23 mg/kg ranged 73-91% for dichlobenil and 91-105% for BAM. The soil was sandy loam (not USDA characterization) from Goch-Nierswalde, Germany; it was fully characterized (characterization lab not clear; pp. 14, 18; Appendix A, p. 24 of MRID 43564901). The soil was homogenized from all sampling depths (0-10, 10-20 and 20-30 cm) prior to fortification. A confirmation of the identification of the analytes was not performed.

ILV (MRID 49098001): Mean recoveries and RSDs met requirements for analysis of dichlobenil ( $n = 5$ ) and BAM ( $n = 4$ ) in loamy sand soil (pp. 10, 22; Table 1, p. 23). The number of trials required to validate the method was not reported. The loamy sand soil (LUFAspeyer 2.2) was fully characterized. For confirmation of the identification of the analytes, GC/MS and GC/MS/MS analyses were performed; however, recovery data were provided in graphical rather than tabular or summary form (p. 22). Recovery data obtained from representative chromatograms of one fortification per analyte/analysis demonstrated acceptable results (81-89%) for both analytes, except in the case of the  $10 \times \text{LOQ}$  for BAM (67%; GC/MS analysis; Figures 20-21, pp. 43-44; Figures 26-27, pp. 49-50; Figures 33-34, pp. 56-57).

**Table 2. Initial Validation Method Recoveries for Analytes in Soil**

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%) <sup>1</sup>	Mean Recovery (%) <sup>2</sup>	Standard Deviation (%)	Relative Standard Deviation (%)
Dichlobenil	0.01 (LOQ)	4	91	--	--	7 <sup>3</sup>
	0.02	--	82	--	--	--
	0.05	--	75	--	--	--
	0.11	--	79	--	--	--
	0.54	--	73	--	--	--
	2.23	--	80	--	--	--
2,6-Dichlorobenzamide (BAM)	0.01 (LOQ)	4	91	--	--	10 <sup>3</sup>
	0.02	--	105	--	--	--
	0.05	--	104	--	--	--
	0.10	--	101	--	--	--
	0.51	--	91	--	--	--
	2.11	--	105	--	--	--

Data were obtained from pp. 9, 18-19; Appendix E, p. 45 of MRID 43564901.

1 Only one value was reported for each fortification level.

2 Overall recoveries (mean  $\pm$  s.d.) for all spiked samples ( $n = 6$ ) were reported as  $80 \pm 6.3\%$  for dichlobenil and  $100 \pm 6.7\%$  for BAM.

3 RSDs were reported by the study author for  $n = 4$ , although means and/or all individual recovery values were not reported.

**Table 3. Independent Validation Method Recoveries for Analytes in Soil**

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%) <sup>1</sup>	Relative Standard Deviation (%)
<b>GC/ECD Analysis</b>						
Dichlobenil	0.010 (LOQ)	5	75-105	89	12	14
	0.10	5	70-96	82	10	12
2,6-Dichlorobenzamide (BAM)	0.010 (LOQ)	4 <sup>2</sup>	72-87	79	7	8
	0.10	4 <sup>2</sup>	73-83	78	4	5
<b>GC/MS Analysis</b>						
Dichlobenil	0.010 (LOQ)	1 <sup>3</sup>	89	--	--	--
	0.10	1 <sup>3</sup>	87	--	--	--
2,6-Dichlorobenzamide (BAM)	0.010 (LOQ)	1 <sup>3</sup>	85	--	--	--
	0.10	1 <sup>3</sup>	67	--	--	--
<b>GC/MS/MS Analysis</b>						
Dichlobenil	0.010 (LOQ)	1 <sup>3</sup>	81	--	--	--
	0.10	1 <sup>3</sup>	82	--	--	--

Data were obtained from pp. 10, 22; Table 1, p. 23; Figures 20-21, pp. 43-44; Figures 26-27, pp. 49-50; Figures 33-34, pp. 56-57 of MRID 49098001.

1 Standard deviations were reviewer-calculated based on data provided in the study report (see DER Attachment 2).

2 The value from a fifth sample (recovery of 53-57%) was excluded from the final results due to 90% probability as an outlier by the Dixon statistical test.

3 Data obtained from representative GC/MS and GC/MS/MS chromatograms; raw data were not provided in a tabular or summary form.

### III. Method Characteristics

The LOQ for both analytes was the same in the ECM and ILV (0.010 mg/kg; p. 19 of MRID 43564901; p. 22 of MRID 49098001). The LOD for both analytes was determined to be 0.001 mg/kg in the ECM and estimated to be 0.002 mg/kg in the ILV. No calculations or justifications for the LOQ or LOD were provided in the ECM or ILV.

**Table 4. Method Characteristics<sup>1</sup>**

		Dichlobenil	BAM
Limit of Quantitation (LOQ)		10 µg/kg	
Limit of Detection (LOD)		2 µg/kg	
Linearity (calibration curve r <sup>2</sup> and concentration range) <sup>2</sup>	GC/ECD	r <sup>2</sup> = 0.9969 (1.0-100 ng/mL)	r <sup>2</sup> = 0.9959 (5-1000 ng/mL)
	GC/MS	r <sup>2</sup> = 0.9997 (10-1000 ng/mL)	r <sup>2</sup> = 1.000 (20-5000 ng/mL)
	GC/MS/MS	r <sup>2</sup> = 0.9997 (10-1000 ng/mL)	Not performed
Repeatable		Yes	Yes <sup>3</sup>
Reproducible		Yes <sup>4</sup>	Yes <sup>4</sup>
Specific		No <sup>5,6</sup>	No <sup>5,6</sup>

Data were obtained from pp. 10, 17, 21-22; Table 1, p. 23; Figure 1, p. 24; Figure 8, p. 31; Figure 16, p. 39; Figures 20-21, pp. 43-44; Figure 23, p. 46; Figures 26-27, pp. 49-50; Figure 29, p. 52; Figures 33-34, pp. 56-57 of MRID 49098001.

1 All data based on the ILV since inadequate raw/supporting data were provided with the ECM.

2 Calibration curves could not be verified by the reviewer because all of the raw data were not provided; raw chromatograms of only 2 or 3 calibration standards were provided.

3 n = 4; one outlier identified by Dixon statistical test.

- 4 Based on the fact that the ILV followed the ECM method with only minor modifications.
- 5 Trace amounts (< LOD) of dichlobenil and BAM were found in the control soil GC/ECD chromatograms (Figure 7, p. 30; Figure 14, p. 37). GC/MS and GC/MS/MS chromatograms of control soil showed no peak at the retention time of dichlobenil (Figure 22, p. 45; Figure 28, p. 51). For BAM, the GC/MS chromatogram of the control soil showed some noise along the baseline at the retention time of the analyte (Figure 35, p. 58).
- 6 In the ILV, confirmation of the identification of the analytes was performed with GC/MS and GC/MS/MS (dichlobenil only); however, data were only provided for one fortification/analyte and to show the linearity of detector response to analyte concentration. In the ECM, only GC/ECD analysis was performed; without confirmation.

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

1. The number of samples was inadequate ( $n = 4$ ) for the validation of dichlobenil and BAM at the LOQ in the ECM and for the validation of BAM at the LOQ and  $10 \times \text{LOQ}$  in the ILV (pp. 18-19; Appendix E, p. 45 of MRID 43564901; Table 1, p. 23; Figures 20-21, pp. 43-44; Figures 26-27, pp. 49-50; Figures 33-34, pp. 56-57; Appendix 11, p. 80 of MRID 49098001). The number of samples was inadequate ( $n = 1$ ) for the validation of dichlobenil and BAM at  $10 \times \text{LOQ}$  in the ECM and for the confirmation of the method via GC/MS and GC/MS/MS (dichlobenil only) at the LOQ and  $10 \times \text{LOQ}$  in the ILV. OCSPP guidelines require a minimum of five spiked samples at the LOQ and  $10 \times \text{LOQ}$ . Only the dichlobenil analysis using GC/ECD in the ILV was reported with five samples. For BAM in the GC/ECD analysis of the ILV, the value from a fifth sample (recovery of 53-57%) was excluded from the final results due to 90% probability as an outlier by the Dixon statistical test. Outlier recoveries should not be excluded. No explanation for the number of samples was provided in the ECM.
2. The LOQ ( $10 \mu\text{g}/\text{kg}$ ) is greater than the lowest toxicological level of concern in soil for BAM (monocot  $\text{EC}_{25} = 6.3 \mu\text{g}/\text{kg}$ ).
3. The method could not be evaluated based on the ECM (MRID 43564901) due to a lack of reported data and supporting raw data. RSDs were reported for analysis of dichlobenil and BAM in soil at the LOQ ( $n = 4$ ); however, mean recoveries were not provided and could not be calculated for the LOQ since individual recovery data was only reported for one sample (pp. 18-19; Appendix E, p. 45 of MRID 43564901). Reported mean recoveries were only provided as overall means of all fortifications (0.01 to *ca.* 2 mg/kg) for each analyte. Furthermore, no supporting raw data in the form of chromatograms, calibration curves or spreadsheets were included in the study report. Without this raw data, the reviewer could not evaluate the linearity and specificity of the method.
4. GC/MS/MS analysis was deemed necessary for dichlobenil because of the lack of an intense third confirmation ion  $> 100 m/z$  in GC/MS analysis (p. 22 of MRID 49098001).
5. In the ILV, confirmation of the identification of the analytes was performed with GC/MS and GC/MS/MS (dichlobenil only); however, raw data was only provided as chromatograms for one fortification/analyte and calibration curves to show the linearity of detector response to analyte concentration (Figure 16, p. 39; Figures 20-21, pp. 43-44; Figure 23, p. 46; Figures 26-27, pp. 49-50; Figure 29, p. 52; Figures 33-34, pp. 56-57 of

MRID 49098001). In the ECM, only GC/ECD analysis was performed; there was no confirmation method.

Recovery data obtained from representative chromatograms of one fortification per analyte/analysis demonstrated acceptable results (81-89%) for both analytes, except in the case of the 10×LOQ for BAM (67%; GC/MS analysis; Figures 20-21, pp. 43-44; Figures 26-27, pp. 49-50; Figures 33-34, pp. 56-57 of MRID 49098001).

6. The ILV reported two minor modifications to the extraction/clean-up procedure of the ECM: 1) the elution solvent [ethanol/petroleum ether (15:85, v:v)] volume of BAM was increased from 5 to 7 mL in the fourth step of the clean-up; and 2) 20 g of anhydrous sodium sulfate was used to remove water from the extracts (pp. 10, 16; Appendix 11, p. 79 of MRID 49098001). The ILV also evaluated the GC/ECD chromatograms using peak area instead of peak height. These modifications were not considered significant deviations from the original ECM. However, as stated above, the notable deviation from the ECM was the inclusion of GC/MS and GC/MS/MS (dichlobenil only) analyses as a confirmation method.
7. In the ECM, the study authors reported the necessity for using two different GC columns and solvents due to the polarity differences of dichlobenil and BAM (Appendix E, p. 45 of MRID 43564901). The study authors also advised the periodic replacement of retention gaps to correct peak-tailing.
8. The determination of the LOD and LOQ were not based on scientifically acceptable procedures. The LOQ for both analytes was the same in the ECM and ILV (0.010 mg/kg; p. 19 of MRID 43564901; p. 22 of MRID 49098001). The LOD differed between the ECM and ILV. No calculations or justifications for the LOQ or LOD were provided in the ECM or ILV. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples.
9. Matrix characterization of the sandy loam soil (ECM) and loamy sand soil (ILV) was reported (pp. 14, 18; Appendix A, p. 24 of MRID 43564901; p. 12; Appendix 4, pp. 63-64 of MRID 49098001). The reviewer assumed that the sampling date reported for the ECM soil on p. 18 of MRID 43564901 was a typographical error and should have been written as “June 28, 1989”, corresponding to the sampling date reported in the first column of the table on p. 14 of MRID 43564901.
10. A reagent blank was not included in the ECM or ILV report.
11. It was reported for the ILV that a single analyst completed a sample set consisting of 12 samples in *ca.* 28 hours (14 hours for extraction and clean-up and *ca.* 10 hours for analysis; p. 21).



## V. References

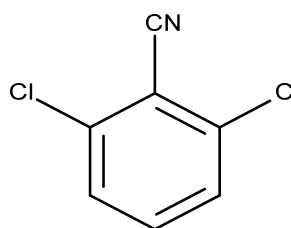
U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

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

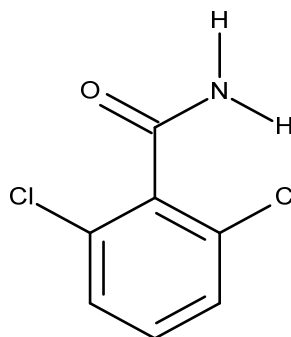
#### Dichlobenil; DCB

**IUPAC Name:** 2,6-Dichlorobenzonitrile  
**CAS Name:** 2,6-Dichlorobenzonitrile  
**CAS Number:** 1194-65-6  
**SMILES String:** c1cc(c(c1)Cl)C#N)Cl



#### 2,6-Dichlorobenzamide; BAM

**IUPAC Name:** 2,6-Dichlorobenzamide  
**CAS Name:** Not reported  
**CAS Number:** 2008-58-4  
**SMILES String:** [H]N([H])C(=O)c1c(ccc1Cl)Cl



### Attachment 2: Statistics Spreadsheet



027401\_43564901+\_  
DER-Fate\_850.6100\_

**Test Material:** Dichlobenil

**MRID:** 43564901

**Title:** 2,6-DICHLOROBENZONITRILE AND 2,6-DICHLOROBENZAMIDE RESIDUES IN SOIL FROM 4 TERRESTRIAL FIELD DISSIPATION TRIALS WITH CASORON G (GERMANY 1989/1990)

**MRID:** 49098001

**Title:** Validation of an Analytical Method for Determination of Dichlobenil and its Metabolite 2,6-Dichlorobenzamide (BAM) in Soil by GC/ECD, Demonstrating GC/MS as Confirmatory Method

**EPA PC Code:** 027401

**OCSPP Guideline:** 850.6100

**For CDM Smith**

**Primary Reviewer:** Lisa Muto

**Signature:**



**Date:** 11/3/14

**Secondary Reviewer:** Lynne Binari

**Signature:**



**Date:** 11/3/14

**QC/QA Manager:** Joan Gaidos

**Signature:**



**Date:** 11/3/14