Analytical method for chlorothalonil degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573 and SYN546669 in soil

Reports: ECM: EPA MRID No.: 49659701. Lin, K. and S.-B. Huang. 2015. Chlorothalonil. Analytical Method (GRM005.07A) for the Determination of Chlorothalonil Degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573 and SYN546669 in Soil. Analytical Method. Syngenta Report No. GRM005.07A and Task No. TK0183016. Report prepared by Syngenta Crop Protection, LLC, Greensboro, North Carolina; sponsored by Syngenta Crop Protection, LLC, Greensboro, North Carolina, Arysta LifeScience North America, LLC, Cary, North Carolina, ADAMA Agricultural Solutions, Ltd. (Formerly Makhteshim Agan North America Inc.), Raleigh, North Carolina, and SipcamAdvan, Durham, North Carolina; and submitted by Syngenta Crop Protection, LLC, Greensboro, North Carolina; 126 pages. Final report issued February 13, 2015.

ILV: EPA MRID No. 49659703. Shen, X. 2015. Chlorothalonil. Chlorothalonil - Independent Laboratory Validation of Analytical Method (GRM005.07A) for the Determination of Chlorothalonil Degradates R182281, R611966, R61I968, R611965, R613636, R417888, SYN510573 and SYN546669 in Soil. Final ILV Report. Report No.: PASC-REP-0544. PASC Project No.: 141-1071. Task No.: TK0183017. Report prepared by Primera Analytical Solutions Corp., Princeton, New Jersey; sponsored by Syngenta Crop Protection, LLC, Greensboro, North Carolina, Arysta LifeScience North America, LLC, Cary, North Carolina, ADAMA Agricultural Solutions, Ltd. (Formerly Makhteshim Agan North America Inc.), Raleigh, North Carolina, and SipcamAdvan, Durham, North Carolina; and submitted by Syngenta Crop Protection, LLC, Greensboro, North Carolina; 285 pages. Final report issued February 24, 2015.

Document No.: MRIDs 49659701 & 49659703

Guideline: 850.6100

Statements: ECM: The study was not conducted in accordance Good Laboratory Practice (GLP) standards (p. 3 of MRID 49659701). Signed and dated No Data Confidentiality and GLP statements were provided (pp. 2-3). Quality Assurance and Authenticity statements were not included. A signed and dated Summary of Revisions to Previous Versions was included (p. 4).
ILV: The study was conducted in accordance with the USEPA FIFRA GLP standards (40 CFR Part 160; p. 3 of MRID 49659703). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). A certification of authenticity was not included.

Classification: This analytical method is classified as Supplemental. Only one set of performance data was submitted; ECM MRID 49659701 was a method only. It could not be determined if the ILV was conducted independently of the ECM since the ILV study author communicated directly with Kaijun Lin of Syngenta who was the ECM study author, as well as the ILV Study Monitor. ILV linearity was not satisfactory for R182281 in both soils and SYN546669

in clay loam soil. The specificity of the method for R611965 in sandy loam soil and SYN546669 in both soils was not supported by ILV representative chromatograms. In the ILV, the purity of the SYN546669 test material was reported as 70.7%. It could not be determined that the ILV were provided with the most difficult matrices with which to validate the method and that ILV soil matrices covered the range of soils used in the terrestrial field dissipation studies.

PC Code:	081901		
EFED Final Reviewer:	Sheng Lin, Ph.D. Physical Scientist	Signature: Date: 12/28/20	SHENG LIN Digitally signed by SHENG LIN Date: 2020.12.28 14:16:56 -05'00'
CDM/CSS-Dynamac JV Reviewers:	Lisa Muto, M.S., Environmental Scientist	Signature: Date:	Lisa Muto 03/20/2019
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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

This analytical method, Syngenta Residue Method GRM005.07A, is designed for the quantitative determination in soil at the LOQ of 0.005 mg/kg of chlorothalonil degradates R182281, R611968, R611965, R417888, SYN510573 and SYN546669 using LC/MS/MS and of chlorothalonil degradates R611966 and R613636 using GC/MS. The LOQ of Syngenta Residue Method GRM005.07A is less than the lowest toxicological level of concern in soil for all analytes. Only one set of performance data was submitted**;** ECM MRID 49659701 was a method only**.** The ILV used two characterized soil matrices, but it could not be determined that the ILV were provided with the most difficult matrices with which to validate the method and that ILV soil matrices covered the range of soils used in the terrestrial field dissipation studies since ILV soils were not compared to or derived from terrestrial field dissipation studies. **It could not be determined if the ILV was conducted independently of the ECM** since the ILV study author communicated directly with the ECM study author. The ILV validated Syngenta Residue Method GRM005.07A in the first trial as written, except for the modification of the GC/MS temperature program and insignificant modifications of the analytical instrumentation and equipment method. The optional J-SPE procedure was not used for analysis of R182281, R611965, R611968, R417888, SYN546669 and SYN510573. All ILV data regarding repeatability, accuracy, and precision were satisfactory. ILV linearity was not satisfactory for R182281 in both soils and SYN546669 in clay loam soil. The specificity of the method for R611965 in sandy loam soil and SYN546669 in both soils was not supported by ILV representative chromatograms due to significant baseline noise around the analyte peak which

prevented proper integration and distinction. In the ILV, the purity of the SYN546669 test material was reported as 70.7%; this inferior purity could be the cause of its unacceptable specificity and linearity.

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						
R182281	49659701 (GRM005.07A)	49659703		Soil[1,2]	13/02/2015	Syngenta Crop Protection, LLC	LC/MS/MS	0.005 mg/kg
R611966							GC/MS	
R611968							LC/MS/MS	
R611965								
R613636							GC/MS	
R417888							LC/MS/MS	
SYN510573								
SYN546669								

1 In the ECM, performance data from the ILV was submitted; however, the test soils were reported as clay loam soil (25% sand, 43% silt, 32% clay, pH 6.5 in 1:1 soil:water, 4.2% organic carbon) from Underwood Farm 0-6" (TK0002309) and sandy loam soil (73% sand, 16% silt, 11% clay, pH 7.8 in 1:1 soil:water, 0.89% organic carbon) from Madera, California, 1-15-13 0-6" (TK0002309; USDA soil texture characterization not specified; p. 33; Table 1, p. 38 of MRID 49659701). These soils were not the test soils which were referenced in the ILV.

2 In the ILV, clay loam soil (PASC ID: 130743-2; 21% sand, 42% silt, 37% clay, pH 7.9 in 1:1 soil:water, 2.8% organic carbon) from SCL Gardener ND 0-6" (TK0002309) and sandy loam soil (PASC ID: 130743-1; 55% sand, 28% silt, 17% clay, pH 7.7 in 1:1 soil:water, 4.9% organic carbon) from McClain Farm 0-6" (TK0002309) were used in the study (USDA soil texture characterization; pp. 26-27; Table 1, p. 47; Appendix 3, pp. 210-211 of MRID 49659703). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

I. Principle of the Method

Syngenta Residue Method GRM005.07A

Soil samples (20 g) in disposable polypropylene centrifuge tube (50 mL) was fortified with mixed fortification standard solution in methanol, if necessary (pp. 18-19 of MRID 49659701). After 5 minutes of equilibration, the soil was extracted with 30 mL of 50:50 (v:v) MeOH:ultrapure water via sonication in a water bath for 10 minutes then shaking on a mechanical shaker (*ca*. 300 cps) for 30 minutes. After centrifugation at *ca*. 6000 rpm with refrigeration at 10°C for about 10 minutes, the supernatant was decanted into a second clean polypropylene centrifuge tube (50 mL). The method noted that, with some soils, particularly those with high clay contents, the solution may still be visibly cloudy even after centrifugation. The extraction was repeated using second 20 mL of 50:50 (v:v) acetonitrile:ultrapure water. The volume of the combined supernatants was adjusted to 50 mL with HPLC grade methanol and stored in a refrigerator if not processed immediately.

For analysis of R182281, R611965, R611968, R417888, SYN546669 and SYN510573, an aliquot (1 mL) from the combined sample extract was transferred to a clean polypropylene centrifuge tube (15 mL) and mixed with 0.5 mL of methanol (pp. 19-20 of MRID 49659701). After diluting the sample to the 10 mL mark with ultrapure water, a *ca*. 1 mL of the aliquot was transferred into a HPLC injection vial for LCMS/MS analysis. If direct injection meets sensitivity or interference problems, the following J-SPE procedure is used. An aliquot (1.5 mL) of the combined extract was applied to a BONDESIL CI8 (100 ± 10 mg; 40µm; Agilent P/N 12213012) with a 2-mL (or an appropriate size) centrifugal filter device (Millipore; Cat. No. UFC40HV00) in top filter cup with 0.5 mL of HPLC grade methanol. 20 µL of freshly prepared 1% ammonium hydroxide solution to the sample was added, and the mixture was homogenized with mild swirling action for *ca*. 30 seconds. The vial was allowed to sit with occasional swirling for *ca*. 5 minutes. After centrifugation at *ca*. 6000 rpm with refrigeration at 10°C for about 45 minutes, an aliquot (1 mL) of the filtered extract was transferred into a separate clean polypropylene centrifuge tube (15 mL). The volume of the sample was reduced to *ca*. 0.4-mL (or less but not dry) under a gentle stream of nitrogen at a water bath temperature of *ca*. 40°C. The volume was adjusted to 1.5 mL with 10:90 (v:v) methanol:ultrapure water; a higher dilution rate may be required for residues higher than 50 ppb in soil. a *ca*. 1 mL of the aliquot was transferred into a HPLC injection vial for LCMS/MS analysis.

Samples were analyzed for R182281, R611965, R611968, R417888, SYN546669 and SYN510573 using a Waters Acquity UPLC system (1 class) coupled to an Applied Biosystems Sciex API 4000 mass spectrometer (pp. 23-28 of MRID 49659701). The LC/MS conditions consisted of a Zorbax SB-CN column (75 x 4.6 mm, 3.5 µm particle size; oven temperature 40°C) with a mobile phase gradient of A) 0.05% formic acid in Optima water and B) 0.05% formic acid in Optima acetonitrile [percent A:B (v:v) at 0-0.5 min. 90:10, 3-8 min. 10:90, 8.1-10 min. 90:10] and TurboIonSpray (ESI) ionization interface MS detection in negative ion mode with MRM (TEM 550°C). Injection volume was 50 µL. Two ion transitions were monitored for each analyte as follows (quantitative and confirmatory, respectively): *m/z* 244.9→182.0 and *m/z* 244.9→174.0 for R182281, *m/z* 265.8→221.8 and *m/z* 267.8→223.8 for R611965, *m/z* 263.0→220.0 and *m/z* 265.0→222.0 for R611968, *m/z* 326.8→220.0 and *m/z* 328.8→222.0 for

R417888, *m/z* 372.9→335.9 and *m/z* 374.9→357.9 for SYN546669, and *m/z* 344.8→302.0 and *m/z* 346.8→304.0 for SYN510573. Expected retention times were *ca*. 4.72, 3.37, 4.03, 3.74, 3.85, and 2.90 minutes for R182281, R611965, R611968, R417888, SYN546669 and SYN510573, respectively. Optional LC/MS/MS instruments and chromatography conditions were provided. The option of a Surveyor Plus LC system coupled with a Thermo Electron TSQ Quantum Ultra MS was suggested. The Optional LC/MS conditions consisted of a Zorbax SB-CN column (75 x 4.6 mm, 3.5 µm particle size; oven temperature 30°C) with a mobile phase gradient of A) 0.05% formic acid in Optima water and B) Optima acetonitrile [percent A:B (v:v) at 0-0.5 min. 90:10, 3.0-6.0 min. 10:90, 6.1-7.0 min. 90:10] and HESI-II Probe (ESI) ionization interface MS detection in negative ion mode with MRM (vaporization temperature 350°C). Injection volume was 50 µL. Two ion transitions were monitored for each analyte as follows (quantitative and confirmatory, respectively): *m/z* 244.9→182.0 and *m/z* 244.9→175.0 for R182281, *m/z* 265.9→222.0 and *m/z* 267.9→224.0 for R611965, *m/z* 262.9→219.9 and *m/z* 264.9→221.9 for R611968, *m/z* 326.9→220.0 and *m/z* 328.9→222.0 for R417888, *m/z* 372.9→291.9 and *m/z* 374.9→293.9 for SYN546669, and *m/z* 344.6→301.9 and *m/z* 346.6→303.9 for SYN510573. Expected retention times were *ca*. 5.32, 3.81, 4.51, 4.17, 4.05, and 3.13 minutes for R182281, R611965, R611968, R417888, SYN546669 and SYN510573, respectively.

For analysis of R611966 and R613636, the sample cleanup and concentration are accomplished by the use of Agilent Bond Elut QuEChERS Dispersive kit (Cat No. 5982-4956; 150 mg CIS, 900 mg $MgS0_4$,15-mL tube) followed by Agilent Bond Elut solid phase extraction (Cat. No. 12102052; 500 mg, 6-mL) cartridge (pp. 20-21 of MRID 49659701). Condition a QuEChERS tube as follows: 1) ethyl acetate; 5 mL, one time; 2) mix well, then centrifuge at 3000 rpm for *ca*. 2 minutes at room temperature; and 3) decant ethyl acetate to waste leaving solid materials in QuEChERS tube. Ethyl acetate (4 mL) was added to the conditioned QuEChERS tube followed by 2.0 mL of the combined extract from above. The QuEChERS tube was vigorously mixed both by hand and vortex mixer, then centrifuged at 3000 rpm for *ca*. 2 minutes at room temperature. The supernatant was decanted, and the process was repeated with 3 mL of ethyl acetate. 1 mL of ultrapure water to the combined ethyl acetate extracts prior to reducing the sample to aqueous (1 mL or less) under a gentle stream of nitrogen at a bath temperature of *ca*. 40°C. The sample was reconstituted by adding 1 mL of methanol, then bring up to 5 mL with ultrapure water. The sample cleanup and concentration are accomplished using an Agilent Bond Elut solid phase extraction (500 mg, 6-mL) cartridge. The SPE cartridge was conditioned as follows: 3 mL of methanol; 3 mL of acetone; then 3 mL of ultrapure water. After the sample was loaded onto the cartridge, the sample tube was washed with 2 mL of 20:80 (v:v) methanol:ultrapure water which was then transferred to the SPE cartridge. The cartridge was rinsed three times with 2 mL of 20:80 (v:v) methanol:ultrapure water. The cartridge was dried with vacuum for *ca*. 10 minutes. The analytes were eluted with 3 mL of 50:50 (v:v) methanol:acetone and collected into a clean glass tube. The eluant was evaporated to dryness under a gentle stream of nitrogen at a bath temperature of *ca*. 40°C. The sample was reconstituted with 1 mL of toluene using an air-tight syringe to yield the sample final fraction. The final fraction was mixed, and an aliquot (ca. 1 mL) into a GC vial for GC-MSD analysis.

Samples are analyzed for R611966 and R613636 using an Agilent 6890N GC coupled to an Agilent 5973 MSD (p. 29 of MRID 49659701). The following conditions were used: Agilent

DB1701 column (30 m x 0.25 mm, 0.25 µm film thickness), helium carrier gas, injector temperature 250°C, temperature program 120°C for 1.00 minute to 270°C for 7.0 minutes (rate 15°C/min.) to post temperature of 325°C for 2.00 minutes, and EI mode. Injection volume was 2.0 µL. Expected retention times for R611966 and R613636 are *ca*. 12.73 and 15.68 minutes, respecitvely. Analytes were identified using three ions (primary, confirmatory 1, and confirmatory 2, respectively): *m/z* 231.9, 233.9, and 247.9 for R611966 and *m/z* 267.9, 265.9, and 269.9 for R613636.

The method contained precautions for use of different SPE and QuEChERS-SPE equipment, as well as non-HPLC grade solvents (p. 22 of MRID 49659701). Also, to minimize the chance of carry-over of high recovery samples, samples should be diluted and solvent blanks should be injected after high recovery samples. A Method Flow Chart was included (Appendix 4, p. 126).

ILV

The ILV reportedly performed Syngenta Residue Method GRM005.07A as written, except for the modification of the GC/MS temperature program and insignificant modifications of the analytical instrumentation and equipment (pp. 27, 31-36 of MRID 49659703). The J-SPE procedure was not used for analysis of R182281, R611965, R611968, R417888, SYN546669 and SYN510573. Samples were analyzed for R182281, R611965, R611968, R417888, SYN546669 and SYN510573 using a Waters Acquity UPLC system coupled to an Applied Biosystems Sciex API 6500 mass spectrometer. The LC/MS conditions consisted of a Zorbax SB-CN column (75 x 4.6 mm, 3.5 µm particle size; oven temperature 40°C) with a mobile phase gradient of A) 0.05% formic acid in deionized water and B) 0.05% formic acid in acetonitrile [percent A:B (v:v) at 0.01-0.50 min. 90:10, 3.00-8.00 min. 10:90, 8.10-10.00 min. 90:10] and TurboIonSpray (ESI) ionization interface MS detection in negative ion mode with MRM (TEM 550°C). Injection volume was 50 µL. Two ion transitions were monitored for each analyte as follows (quantitative and confirmatory, respectively): *m/z* 244.9→182.0 and *m/z* 244.9→174.9 for R182281, *m/z* 265.8→221.8 and *m/z* 267.8→223.9 for R611965, *m/z* 263.0→220.0 and *m/z* 265.0→222.0 for R611968, *m/z* 326.8→219.8 and *m/z* 328.9→222.0 for R417888, *m/z* 372.9→335.9 and *m/z* 374.9→357.9 for SYN546669, and *m/z* 344.8→302.0 and *m/z* 346.8→304.0 for SYN510573. Expected retention times were *ca*. 4.0, 3.0, 3.5, 3.2, 2.2, and 2.1 minutes for R182281, R611965, R611968, R417888, SYN546669 and SYN510573, respectively. Samples are analyzed for R611966 and R613636 using an Agilent 6890N GC coupled to a HP 5793 MSD. The following conditions were used: J&W DB-1701 column (30 m x 0.25 mm, 0.25 µm film thickness), helium carrier gas, injector temperature 250°C, temperature program 120°C for 1.00 minute to 238°C (rate 18°C/min.) to 242°C (rate 0.5°C/min.) to 260°C (rate 5°C/min.) for 5.0 minutes to 280°C (rate 3°C/min.) for 2.0 minutes to post temperature of 325°C for 2.00 minutes, and EI mode. Injection volume was 3.0 µL. Expected retention times for R611966 and R613636 are *ca*. 14.7 and 20.4 minutes, respectively. Analytes were identified using three ions (primary, confirmatory 1, and confirmatory 2, respectively): *m/z* 231.9, 233.9, and 247.9 for R611966 and *m/z* 267.9, 265.9, and 269.9 for R613636.

In the ECM and ILV, the Limit of Quantification (LOQ) for chlorothalonil degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573, and SYN546669 in Syngenta Residue Method GRM005.07A was reported as 0.005 mg/kg (ppm; pp. 14, 33 of MRID

49659701; pp. 16, 44 of MRID 49659703). The Limit of Detection (LOD) in the ECM was 25 pg injected on column, equivalent to 0.5 pg/µL, when using a 50 µL injection, for chlorothalonil degradates R182281, R611968, R611965, R417888, SYN510573 and SYN546669 using LC/MS/MS and was 6 pg injected on column, equivalent to 2 ng/mL, when using a 3 µL injection, for chlorothalonil degradates R611966 and R613636 using GC/MS. In the ILV, the LODs were reported as 0.05 ng/mL for chlorothalonil degradates R182281, R611968, R611965, R417888, SYN510573, and SYN546669 and as 2 ng/mL for chlorothalonil degradates R611966 and R613636 (Figures 1.1-1.32, pp. 72-87 of MRID 49659703).

II. Recovery Findings

ECM (MRID 49659701): Syngenta Residue Method GRM005.07A was a method only. No internal validation performance data was submitted. Performance data from the ILV was submitted; however, the test soils were reported as clay loam soil (25% sand, 43% silt, 32% clay, pH 6.5 in 1:1 soil:water, 4.2% organic carbon) from Underwood Farm 0-6" (TK0002309) and sandy loam soil (73% sand, 16% silt, 11% clay, pH 7.8 in 1:1 soil:water, 0.89% organic carbon) from Madera, California, 1-15-13 0-6" (TK0002309; USDA soil texture characterization not specified; p. 33; Table 1, p. 38). These soils were not the test soils which were referenced in the ILV.

ILV (MRID 49659703): For Syngenta Residue Method GRM005.07A, mean recoveries and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%) for analysis of chlorothalonil degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573, and SYN546669 at the LOQ (0.005 mg/kg) and 10×LOQ (0.05 mg/kg) in two soil matrices (pp. 17-20; Tables 3-18, pp. 49-64). Two ion transitions were monitored for R182281, R611965, R611968, R417888, SYN546669 and SYN510573 using LC/MS/MS. R611966 and R613636 were identified using three ions via GC/MS; however, only the primary and confirmatory 1 were quantified. Performance data (results) of the quantitation and confirmation analyses were comparable. Clay loam soil (PASC ID: 130743-2; 21% sand, 42% silt, 37% clay, pH 7.9 in 1:1 soil:water, 2.8% organic carbon) from SCL Gardener ND 0-6" (TK0002309) and sandy loam soil (PASC ID: 130743-1; 55% sand, 28% silt, 17% clay, pH 7.7 in 1:1 soil:water, 4.9% organic carbon) from McClain Farm 0-6" (TK0002309) were used in the study (USDA soil texture characterization; pp. 26-27; Table 1, p. 47; Appendix 3, pp. 210-211). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota. The ILV validated Syngenta Residue Method GRM005.07A in the first trial as written, except for the modification of the GC/MS temperature program and insignificant modifications of the analytical instrumentation and equipment method (pp. 27, 31-37 of MRID 49659703). The J-SPE procedure was not used for analysis of R182281, R611965, R611968, R417888, SYN546669 and SYN510573.

Table 2. Initial Validation Method Recoveries for Chlorothalonil Degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573, and SYN546669 in Soil[1,2]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
	Soil					
R182281	0.005	5	Not performed			
	0.05	5				
R611966	0.005	5				
	0.05	5				
R611968	0.005	5				
	0.05	5				
R611965	0.005	5				
	0.05	5				
R613636	0.005	5				
	0.05	5				
R417888	0.005	5				
	0.05	5				
SYN510573	0.005	5				
	0.05	5				
SYN546669	0.005	5				
	0.05	5				

Data (uncorrected recovery results; pp. 31-32) were obtained from Tables 2-17, pp. 39-54 of MRID 49659701.

1 The clay loam soil (25% sand, 43% silt, 32% clay, pH 6.5 in 1:1 soil:water, 4.2% organic carbon) from Underwood Farm 0-6" (TK0002309) and sandy loam soil (73% sand, 16% silt, 11% clay, pH 7.8 in 1:1 soil:water, 0.89% organic carbon) from Madera, California, 1-15-13 0-6" (TK0002309) were used in the study (USDA soil texture characterization not specified; Table 1, p. 38).

2 Two ion transitions were monitored for R182281, R611965, R611968, R417888, SYN546669 and SYN510573 using LC/MS/MS as follows (quantitative and confirmatory, respectively): *m/z* 244.9→182.0 and *m/z* 244.9→174.0 for R182281, *m/z* 265.8→221.8 and *m/z* 267.8→223.8 for R611965, *m/z* 263.0→220.0 and *m/z* 265.0→222.0 for R611968, *m/z* 326.8→220.0 and *m/z* 328.8→222.0 for R417888, *m/z* 372.9→335.9 and *m/z* 374.9→357.9 for SYN546669, and *m/z* 344.8→302.0 and *m/z* 346.8→304.0 for SYN510573**.** R611966 and R613636 were identified using three ions via GC/MS (primary, confirmatory 1, and confirmatory 2, respectively): *m/z* 231.9, 233.9, and 247.9 for R611966 and *m/z* 267.9, 265.9, and 269.9 for R613636.

Table 3. Independent Validation Method Recoveries for Chlorothalonil Degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573, and SYN546669 in Soil[1,2]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
	Sandy Loam Soil					
	Quantitation ion transition or Quantitation ion					
R182281	0.005	5	77-92	84	5	6
	0.05	5	83-87	85	1	2
R611966	0.005	5	93-100	97	3	3
	0.05	5	86-93	90	3	3
R611968	0.005	5	86-91	88	2	2
	0.05	5	80-90	85	3	4
R611965	0.005	5	77-92	82	5	7
	0.05	5	91-93	92	2	2
R613636	0.005	5	95-108	102	5	5
	0.05	5	93-101	98	3	3
R417888	0.005	5	95-96	95	1	1
	0.05	5	92-94	93	1	1
SYN510573	0.005	5	87-95	91	4	4
	0.05	5	90-91	90	1	1
SYN546669	0.005	5	81-88	84	3	4
	0.05	5	79-86	82	4	4
	Confirmation ion transition or Confirmation ion					
R182281	0.005	5	81-90	85	3	4
	0.05	5	82-89	84	3	3
R611966	0.005	5	95-98	97	1	2
	0.05	5	86-93	90	3	3
R611968	0.005	5	85-91	89	3	3
	0.05	5	84-88	86	2	2
R611965	0.005	5	92-102	94	5	5
	0.05	5	87-94	90	3	3
R613636	0.005	5	91-104	98	5	5
	0.05	5	93-99	97	2	2
R417888	0.005	5	95-98	96	1	1
	0.05	5	90-96	92	2	3
SYN510573	0.005	5	85-100	92	5	6
	0.05	5	88-93	90	2	2
SYN546669	0.005	5	87-101	94	5	5
	0.05	5	86-90	88	2	2
	Clay Loam Soil					
	Quantitation ion transition or Quantitation ion					
R182281	0.005	5	65-94	85	12	14
	0.05	5	86-91	89	2	3
R611966	0.005	5	111-120	116	4	3
	0.05	5	95-105	99	4	4
R611968	0.005	5	87-93	90	3	3
	0.05	5	85-88	86	1	1

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
R611965	0.005	5	82-87	84	2	3
	0.05	5	81-86	84	2	2
R613636	0.005	5	99-109	103	4	4
	0.05	5	96-107	101	5	5
R417888	0.005	5	92-101	98	4	4
	0.05	5	90-93	91	1	2
SYN510573	0.005	5	72-82	75	4	5
	0.05	5	69-71	70	1	1
SYN546669	0.005	5	89-117	103	12	11
	0.05	5	94-107	98	5	5
	Confirmation ion transition or Confirmation ion					
R182281	0.005	5	62-92	84	13	15
	0.05	5	89-93	90	2	2
R611966	0.005	5	104-110	107	2	2
	0.05	5	93-103	97	4	4
R611968	0.005	5	87-95	90	3	4
	0.05	5	87-88	87	1	1
R611965	0.005	5	88-95	91	3	3
	0.05	5	83-86	85	2	2
R613636	0.005	5	97-103	102	3	3
	0.05	5	96-107	101	5	5
R417888	0.005	5	89-94	93	3	3
	0.05	5	88-95	93	3	3
SYN510573	0.005	5	73-83	77	4	6
	0.05	5	70-72	71	1	1
SYN546669	0.005	5	102-118	108	6	6
	0.05	5	93-109	102	6	6

Data (recovery results were corrected when residues were quantified in the controls; Appendix 4, pp. 229-230) were obtained from pp. 17-20; Tables 3-18, pp. 49-64 of MRID 49659703.

1 The clay loam soil (PASC ID: 130743-2; 21% sand, 42% silt, 37% clay, pH 7.9 in 1:1 soil:water, 2.8% organic carbon) from SCL Gardener ND 0-6" (TK0002309) and sandy loam soil (PASC ID: 130743-1; 55% sand, 28% silt, 17% clay, pH 7.7 in 1:1 soil:water, 4.9% organic carbon) from McClain Farm 0-6" (TK0002309) were used in the study (USDA soil texture characterization; pp. 26-27; Table 1, p. 47; Appendix 3, pp. 210-211). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

2 Two ion transitions were monitored for R182281, R611965, R611968, R417888, SYN546669 and SYN510573 using LC/MS/MS as follows (quantitative and confirmatory, respectively): *m/z* 244.9→182.0 and *m/z* 244.9→174.9 for R182281, *m/z* 265.8→221.8 and *m/z* 267.8→223.9 for R611965, *m/z* 263.0→220.0 and *m/z* 265.0→222.0 for R611968, *m/z* 326.8→219.8 and *m/z* 328.9→222.0 for R417888, *m/z* 372.9→335.9 and *m/z* 374.9→357.9 for SYN546669, and *m/z* 344.8→302.0 and *m/z* 346.8→304.0 for SYN510573. R611966 and R613636 were identified using three ions via GC/MS (primary, confirmatory 1, and confirmatory 2, respectively): *m/z* 231.9, 233.9, and 247.9 for R611966 and *m/z* 267.9, 265.9, and 269.9 for R613636; however, only the primary and confirmatory 1 were quantified. Ion transitions were similar to those of the ECM.

III. Method Characteristics

In the ECM and ILV, the LOQ for chlorothalonil degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573 and SYN546669 in Syngenta Residue Method GRM005.07A was reported as 0.005 mg/kg (ppm; pp. 14, 33 of MRID 49659701; pp. 16, 44 of MRID 49659703). In the ECM, the LOQ was defined as the lowest analyte concentration in a sample at which the methodology has been validated, i.e. which yielded a mean recovery of 70-110% and relative standard deviation of ≤20%. No justifications were reported in the ILV. The LOD in the ECM was 25 pg injected on column, equivalent to 0.5 pg/µL, when using a 50 µL injection, for chlorothalonil degradates R182281, R611968, R611965, R417888, SYN510573 and SYN546669 using LC/MS/MS and was 6 pg injected on column, equivalent to 2 ng/mL, when using a 3 µL injection, for chlorothalonil degradates R611966 and R613636 using GC/MS. In the ECM, the LOD was defined as the lowest analyte concentration detectable above the mean amplitude of the background noise in an untreated sample at the corresponding retention time. An estimate of the LOD can be taken as three times the mean amplitude of the background noise. The ECM study authors noted that the LOD may vary between runs and from instrument to instrument. In the ILV, the LODs were reported as 0.05 ng/mL for chlorothalonil degradates R182281, R611968, R611965, R417888, SYN510573, and SYN546669 and as 2 ng/mL for chlorothalonil degradates R611966 and R613636 (Figures 1.1-1.32, pp. 72-87 of MRID 49659703). No calculations for LOQ and LOD were reported in the ECM or ILV. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples.

Table 4. Method Characteristics in Soil

Analyte			**R182281**	**R611966**	**R611968**	**R611965**	**R613636**	**R417888**	**SYN510573**	**SYN546669**
Analysis[1]			LC/MS/MS	GC/MS	LC/MS/MS		GC/MS	LC/MS/MS		
Limit of Quantitation (LOQ)	ECM		0.005 mg/kg							
	ILV									
Limit of Detection (LOD)	ECM		25 pg injected on column, equivalent to 0.5 pg/µL, when using a 50 µL injection	6 pg injected on column, equivalent to 2 ng/mL, when using a 3 µL injection	25 pg injected on column, equivalent to 0.5 pg/µL, when using a 50 µL injection		6 pg injected on column, equivalent to 2 ng/mL, when using a 3 µL injection	25 pg injected on column, equivalent to 0.5 pg/µL, when using a 50 µL injection		
	ILV		0.05 ng/mL	2 ng/mL	0.05 ng/mL		2 ng/mL	0.05 ng/mL		
Linearity (calibration curve r^2 and concentration range)	ECM[2]		**None submitted**							
	ILV[3]	Sandy loam	r^2 = **0.9936** (Q) r^2 = 0.9950 (C)	r^2 = 0.9995 (Q) r^2 = 0.9990 (C)	r^2 = 0.9992 (Q) r^2 = 0.9994 (C)	r^2 = 0.9970 (Q) r^2 = 0.9990 (C)	r^2 = 0.9986 (Q) r^2 = 0.9994 (C)	r^2 = 0.9996 (Q) r^2 = 0.9998 (C)	r^2 = 0.9996 (Q & C)	r^2 = 0.9982 (Q) r^2 = 0.9986 (C)
		Clay loam	r^2 = **0.9942** (Q) r^2 = **0.9940** (C)	r^2 = 0.9981 (Q) r^2 = 0.9987 (C)	r^2 = 0.9988 (Q & C)	r^2 = 0.9980 (Q) r^2 = **0.9938** (C)	r^2 = 0.9975 (Q) r^2 = 0.9979 (C)	r^2 = 0.9982 (Q) r^2 = 0.9986 (C)	r^2 = 0.9996 (Q & C)	r^2 = **0.9906** (Q) r^2 = **0.9940** (C)
		Range	0.05-5.0 ng/mL (solvent-based)	2.0-100 ng/mL (matrix-matched)	0.05-5.0 ng/mL (solvent-based)		2.0-100 ng/mL (matrix-matched)	0.05-5.0 ng/mL (solvent-based)		0.05-5.0 ng/mL (solvent-based for sandy loam; matrix-matched for clay loam)
Repeatable	ECM[4]		**Not performed**							
	ILV[5,6]		Yes at LOQ and 10×LOQ (two characterized soil matrices).							
Reproducible			**Could not be determined**; only one set of performance data was submitted.							

Analyte		**R182281**	**R611966**	**R611968**	**R611965**	**R613636**	**R417888**	**SYN510573**	**SYN546669**
Analysis[1]		LC/MS/MS	GC/MS	LC/MS/MS		GC/MS	LC/MS/MS		
Specific	ECM[2]	**None submitted**							
	ILV	In general, clay loam soil representative chromatograms were cleaner than sandy loam soil representative chromatograms.							
		Yes, matrix interferences were <6 % of the LOQ (based on peak area).	Yes, no matrix interferences were observed in control samples. Some baseline noise was observed.	Yes, matrix interferences were <2 % of the LOQ (based on peak area).	**No** in sandy loam soil, significant baseline noise around analyte peak prevented proper integration.[7] Yes, in clay loam soil, only some baseline noise observed. No matrix interferences identified as analyte were observed in control samples or either soil.	Yes, no matrix interferences were observed in control samples.	Yes, matrix interferences were <2 % of the LOQ (based on peak area)	Yes, some nearby baseline noise was observed which did not significantly interfere with peak attenuation and integration in sandy loam soil. No matrix interferences identified as analyte were observed in control samples or either soil.	**No**, LOQ analyte peak was not distinguished from baseline noise in both soils; baseline noise significantly interfered with peak attenuation and integration.[8] 10×LOQ peak was wide and displayed peak-splitting or peak-shouldering.[9] Matrix interferences were <2 % of the LOQ (based on peak area).

Data were obtained from pp. 14, 33 (LOQ/LOD) of MRID 49659701; pp. 16, 44 (LOQ/LOD); pp. 17-20; Tables 3-18, pp. 49-64 (recovery results); Figures 9.1-9.16, pp. 144-151; Appendix 5, pp. 232-257, 270-273 (calibration curves); Figures 1.1-8.16, pp. 72-143 (chromatograms) of MRID 49659703; DER Attachment 2. Q = Quantitation ion transition; C = Confirmation ion transition.

1 Two ion transitions were monitored for R182281, R611965, R611968, R417888, SYN546669 and SYN510573 using LC/MS/MS. R611966 and R613636 were identified using three ions via GC/MS; however, only the primary and confirmatory 1 were quantified.

2 Syngenta Residue Method GRM005.07A was a method only. No internal validation performance data was submitted.

3 ILV correlation coefficients (r^2) values for LC/MS/MS analyses were reviewer-calculated from r values provided in the study report (Figures 9.1-9.16, pp. 144-151; Appendix 5, pp. 232-257, 270-273 of MRID 49659703; DER Attachment 2).

4 In the ECM, performance data from the ILV was submitted; however, the test soils were reported as clay loam soil (25% sand, 43% silt, 32% clay, pH 6.5 in 1:1 soil:water, 4.2% organic carbon) from Underwood Farm 0-6" (TK0002309) and sandy loam soil (73% sand, 16% silt, 11% clay, pH 7.8 in 1:1 soil:water, 0.89% organic carbon) from Madera, California, 1-15-13 0-6" (TK0002309; USDA soil texture characterization not specified; p. 33; Table 1, p. 38 of MRID 49659701). These soils were not the test soils which were referenced in the ILV.

5 In the ILV, clay loam soil (PASC ID: 130743-2; 21% sand, 42% silt, 37% clay, pH 7.9 in 1:1 soil:water, 2.8% organic carbon) from SCL Gardener ND 0-6" (TK0002309) and sandy loam soil (PASC ID: 130743-1; 55% sand, 28% silt, 17% clay, pH 7.7 in 1:1 soil:water, 4.9% organic carbon) from McClain Farm 0-6" (TK0002309) were used in the study (USDA soil texture characterization; pp. 26-27; Table 1, p. 47; Appendix 3, pp. 210-211 of MRID 49659703). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

6 The ILV validated Syngenta Residue Method GRM005.07A in the first trial as written, except for the modification of the GC/MS temperature program and insignificant modifications of the analytical instrumentation and equipment method (pp. 27, 31-37 of MRID 49659703). The J-SPE procedure was not used for analysis of R182281, R611965, R611968, R417888, SYN546669 and SYN510573.

7 Based on Figures 5.5-5.6, p. 114 of MRID 49659703.

8 Based on Figures 5.11-5.12, p. 117 and Figures 6.11-6.12, p. 125 of MRID 49659703.

9 Based on Figures 7.11-7.12, p. 133 and Figures 8.11-8.12, p. 141 of MRID 49659703.

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

IV. Method Deficiencies and Reviewer's Comments

1. The reproducibility of Syngenta Residue Method GRM005.07A could not be determined since only one set of performance data was submitted. ECM MRID 49659701 was a method only; no internal validation was performed. Performance data, calibration curves, and representative chromatograms from the ILV were submitted in the ECM as laboratory support for the method. OCSPP guidelines state that two sets of performance data should be submitted, one for the initial or other internal validation and one for the ILV, and each set of performance data should contain a minimum of five spiked replicates which were analyzed at each concentration (*i.e.*, minimally, the LOQ and 10× LOQ) for each analyte.

   The Draft of Syngenta Residue Method GRM005.07A was included in Appendix 2 of the ILV MRID 49659703, but "the Table Section and Figure Section in the draft method are removed in this appendix because they are for information only" (Appendix 2, p. 174 of MRID 49659703).

2. It could not be determined if the ILV was conducted independently of the ECM since the ILV study author (Xiaorong Shen) communicated directly with Kaijun Lin of Syngenta who was the ECM study author, as well as the ILV Study Monitor (pp. 5, 42-43; Appendix 6, pp. 277-285 of MRID 49659703). These communications included exchange of protocols, acquisition of analytical standard and control sample, and pre-validation evaluation and method establishment including calibration curve linearity and modification of the GC-MSD method, and acceptance of LOQ % recovery of R611966 (116%) in clay soil (p. 38 of MRID 49659703). The ECM study author requested details about the problem which occurred in the GC/MS calibration and provided approval for the ILV solution. OCSPP guidelines state that ILV validations are performed without collusion with the ECM personnel. The reviewer noted that Myra Manuli of Syngenta was also involved in ILV communications, although her role was not specified.

3. ILV linearity was not satisfactory for the quantitation ion transition analyses of R182281 in both soils (sandy loam soil $r^2 = 0.9936$ and clay loam soil $r^2 = 0.9942$) and SYN546669 in clay loam soil ($r^2 = 0.9906$; Figures 9.1-9.16, pp. 144-151; Appendix 5, pp. 232-257, 270-273 (of MRID 49659703; DER Attachment 2). Linearity is satisfactory when $r^2 \geq 0.995$.

   ILV linearity was not satisfactory for the confirmation ion transition analyses of R182281 in clay loam soil ($r^2 = 0.9940$), R611965 in clay loam soil ($r^2 = 0.9938$), and SYN546669 in clay loam soil ($r^2 = 0.9940$; Figures 9.1-9.16, pp. 144-151; Appendix 5, pp. 232-257, 270-273 of MRID 49659703; DER Attachment 2). Linearity is satisfactory when $r^2 \geq 0.995$. The reviewer noted that these deviations in linearity did not affect the validity of the method since a confirmatory method is not usually required when LC/MS/MS or GC/MS is the primary method used to generate study data.

4. The specificity of the method for R611965 in sandy loam soil and SYN546669 in both soils was not supported by ILV representative chromatograms. There was significant

baseline noise around analyte peak of R611965 in representative chromatograms of sandy loam soil which prevented proper integration (Figures 5.5-5.6, p. 114 of MRID 49659703). The SYN546669 LOQ analyte peak was not distinguished from baseline noise in representative chromatograms of both soils; baseline noise significantly interfered with peak attenuation and integration (Figures 5.11-5.12, p. 117; Figures 6.11-6.12, p. 125; Figures 7.11-7.12, p. 133; and Figures 8.11-8.12, p. 141). Also, the SYN546669 10×LOQ peak was wide and displayed peak-splitting or peak-shouldering in representative chromatograms of both soils. In general, clay loam soil representative chromatograms were cleaner than sandy loam soil representative chromatograms.

5. The purity of SYN546669 test material was reported as 70.7% in the ILV; this inferior purity could be the cause of its unacceptable specificity and linearity (p. 26 of MRID 49659703). Test material purities should be >90%.

6. It could not be determined that the ILV was provided with the most difficult matrices with which to validate the method. OCSPP 850.6100 guidance suggests for a given sample matrix, the registrant should select the most difficult analytical sample condition from the study (*e.g.*, high organic content versus low organic content in a soil matrix) to analyze from the study to demonstrate how well the method performs. Soil characterization data was provided for the ILV soils (USDA soil texture characterization; pp. 26-27; Table 1, p. 47; Appendix 3, pp. 210-211 of MRID 49659703). The reviewer noted that soil characterization was performed by Agvise Laboratories, Northwood, North Dakota. Additionally, since no terrestrial field dissipation studies were submitted, it not be determined if the ILV soil matrices covered the range of soils used in the terrestrial field dissipation studies. The ILV test soils were reportedly from Syngenta Study # TK0002309, but the title, reference, and description for this study was not reported. The reviewer noted that a certain number of soil matrices is not specified in the OCSPP guidelines in order to cover the range of soils used in the terrestrial field dissipation studies.

   The reviewer noted that the ECM, which provided performance data from the ILV, reported that the test soils were clay loam soil (25% sand, 43% silt, 32% clay, pH 6.5 in 1:1 soil:water, 4.2% organic carbon) from Underwood Farm 0-6" (TK0002309) and sandy loam soil (73% sand, 16% silt, 11% clay, pH 7.8 in 1:1 soil:water, 0.89% organic carbon) from Madera, California, 1-15-13 0-6" (TK0002309; USDA soil texture characterization not specified; p. 33; Table 1, p. 38 of MRID 49659701). These soils were not the test soils which were referenced in the ILV.

   The reviewer also noted that the ILV soil matrices reported in this ILV study (MRID 49659703) matched the soil descriptions and soil characteristics reported in another soil ILV performed by the same ILV for chlorothalonil only (MRID 49659702); however, the soil sources (sites) differed. The reviewer believed that a typographical error occurred within the two ILVs.

7. The estimations of the LOQ and LOD in ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 14, 33 of MRID 49659701; pp.

16, 44; Figures 1.1-1.32, pp. 72-87 of MRID 49659703). In the ECM, the LOQ was defined as the lowest analyte concentration in a sample at which the methodology has been validated, i.e. which yielded a mean recovery of 70-110% and relative standard deviation of ≤20%. In the ECM, the LOD was defined as the lowest analyte concentration detectable above the mean amplitude of the background noise in an untreated sample at the corresponding retention time. An estimate of the LOD can be taken as three times the mean amplitude of the background noise The ECM study authors noted that the LOD may vary between runs and from instrument to instrument. No calculations for LOQ and LOD were reported in the ECM or ILV. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples.

The reviewer noted that the ILV communications with the ILV Study Monitor reported GC sensitivity problems where the sensitivity of R611966 was fine (LOD of 2 ng/ml), but the LOD for R613636 was about 10 ng/mL (the LOQ equivalent was 4 ng/ml; Appendix 6, p. 279 of MRID 49659703). The ILV noted that they needed to improve the instrument conditions. The reviewer assumed that they did by the ILV modifications to the GC/MS parameters.

8. In the ILV, the matrix effects were determined to be insignificant (<±20%) for all analytes/matrices, but R182281 in sandy loam soil, SYN510573 and SYN546669 in clay loam soil, and R611966 and R613636 in GC-MSD for both soil types (p. 43; Table 19, p. 65 of MRID 49659703). Matrix-matched calibration standards were recommended for these analytes/matrices. These ILV results were included in the ECM, as well.

9. In the ILV, the final soil extracts of R182281, R611968, R611965, R417888, SYN510573, and SYN546669 were found to be stable for up to *ca*. 10 days at *ca*. 4°C (p. 43; Tables 20-27, pp. 66-70 of MRID 49659703). The final fraction residues of R611966 and R613636 in toluene were found to be stable for up to *ca*. 7 days when stored in vials at 4°C. These ILV results were included in the ECM, as well.

10. The ILV reported that 1 sample set of 13 samples each can be completed in 1 day with LC/MS/MS and GC/MS performed overnight (p. 43 of MRID 49659703).

V. References

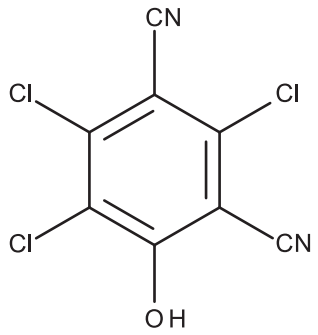
U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

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

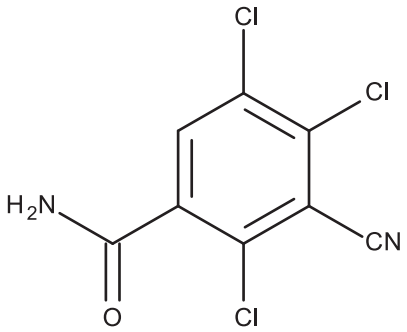
R182281 (SDS3701)

IUPAC Name: 2,4,5-Trichloro-6-hydroxyisophthalonitrile
CAS Name: Not reported
CAS Number: 28343-61-5
SMILES String: C(#N)c1c(c(c(c(c1Cl)C#N)Cl)Cl)O



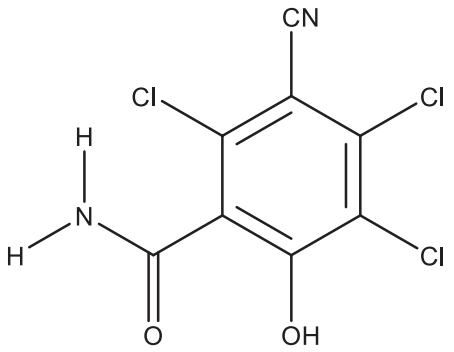
R611966 (SDS47523)

IUPAC Name: 2,4,5-Trichloro-3-cyano-benzamide
CAS Name: Not reported
CAS Number: Not reported
SMILES String: c1c(c(c(c(c1Cl)Cl)C#N)Cl)C(=O)N



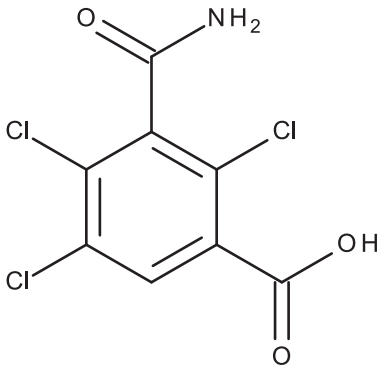
R611968 (SDS47525)

IUPAC Name: 2,4,5-Trichloro-3-cyano-6-hydroxybenzamide
CAS Name: Not reported
CAS Number: Not reported
SMILES String: OC(C(Cl)=C(Cl)C(C#N)=C1Cl)=C1C(N([H])[H])=O



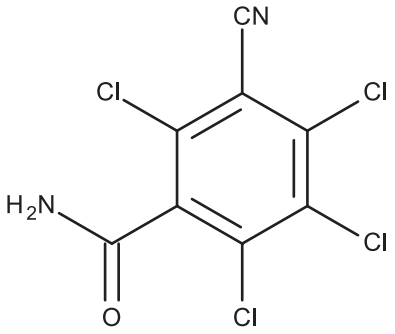
R611965 (SDS46851)

IUPAC Name: 3-Carbamoyl-2,4,5-trichloro-benzoic acid
CAS Name: Not reported
CAS Number: 142733-37-7
SMILES String: c1c(c(c(c(c1Cl)Cl)C(=O)N)Cl)C(=O)O



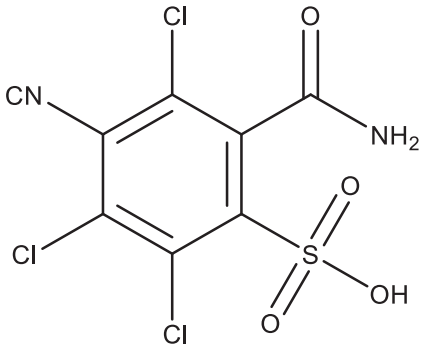
R613636 (SDS19221)

IUPAC Name: 2,3,4,6-Tetrachloro-5-cyano-benzamide
CAS Name: Not reported
CAS Number: 61073-19-6
SMILES String: C(#N)c1c(c(c(c(c1Cl)Cl)Cl)C(=O)N)Cl



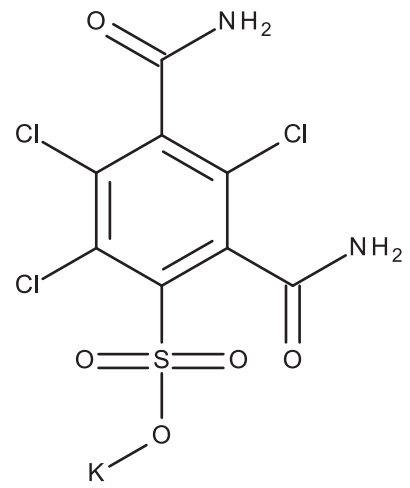
R417888

IUPAC Name: 2-Carbamoyl-3,5,6-trichloro-4-cyanobenzenesulfonic acid
CAS Name: Not reported
CAS Number: Not reported
SMILES String: ClC1=C(C(C#N)=C(Cl)C(C(N)=O)=C1S(O)(=O)=O)Cl



SYN510573 (R471811)

IUPAC Name: Potassium; 2,4-dicarbamoyl-3,5,6-trichlorobenzenesulfonate
CAS Name: Not reported
CAS Number: Not reported
SMILES String: c1(c(c(c(c(c1Cl)C(=O)N)Cl)Cl)S(=O)(=O)O[K])C(=O)N



SYN546669 (R419492)

IUPAC Name: 4-Carbamoyl-2,5-dichloro-6-cyano-benzene-1,3-disulfonic acid
CAS Name: Not reported
CAS Number: Not reported
SMILES String: C(#N)c1c(c(c(c(c1Cl)C(=O)N)S(=O)(=O)O)Cl)S(=O)(=O)O