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Chlorothalonil
Chlorothalonil – Independent Laboratory Validation of Analytical Method (GRM005.07A) for the Determination of Chlorothalonil Degradates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573 and SYN546669 in Soil
Final ILV Report

DATA REQUIREMENT(S):

VOLUME 1 OF 1 OF STUDY

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2.0 INTRODUCTION

Described in this report is the independent laboratory validation (ILV) of Syngenta analytical method entitled "Analytical Method (GRM005.07A) for the Determination of Chlorothalonil Degredates R182281, R611966, R611968, R611965, R613636, R417888, SYN510573 and SYN546669 in Soil".

This study was designed to satisfy harmonized guideline requirements described in EPA OCSP 850.6100 (Data Reporting for Environmental Chemistry Methods), EC Guidance Documents SANCO/3029/99 Rev.4 (2000), and SANCO/825/00 Rev.8.1 (2010). This study was conducted in compliance with EPA FIFRA Good Laboratory Practice Standards, 40 CFR Part 160 (3).

BRIEF SUMMARY OF METHOD

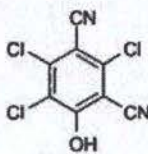
Soil sub-samples were initially extracted with 50/50 (v/v) methanol/water by mechanical shaking followed by a second extraction (mechanical shaking) with 50/50 (v/v) acetonitrile/water at room temperature. The two extracts were combined after centrifugation and adjusted to the final volume. For analysis of R182281, R611968, R611965, R417888, SYN510573 and SYN546669, an aliquot was taken from the combined soil extract and diluted/ injected onto LC-MS/MS with negative electrospray ionization (ESI). A separate aliquot of the combined extract was taken for QuEChERS treatment followed by solid phase extraction (SPE) clean up. After proper sample treatments, the sample was injected for residue analysis of R611966 and R613636 using GC-MSD.

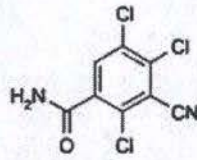
3.0 MATERIALS AND METHODS

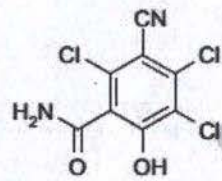
3.1 Test/Reference Substance


The test/reference substances were obtained from Syngenta Crop Protection, LLC. The following test/reference substances were used:


STANDARD	LOT NUMBER	PURITY (%)	EXPIRATION DATE	STORAGE
R182281	DAH-XXIX-83	98.2	February 28, 2015	Refrigerator
R611966	DAH-XXIX-89	99.7	January 31, 2015	Refrigerator
R611968	DAH-XXIX-85-1	99.7	May 31, 2015	Refrigerator
R611965	DAH-XXXV-37	98.3	May 31, 2015	Refrigerator
R613636	DAH-XXIX-95	98.4	April 30, 2015	Refrigerator
R417888	DAH-XXX-70	99.7	March 31, 2015	Refrigerator
SYN510573	DAH-XXXV-36	90.0	June 30, 2015	Refrigerator
SYN546669	DAH-XXXV-55-2	70.7	August 31, 2015	Refrigerator

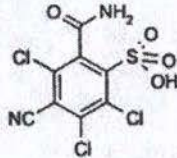
Compound Structure	 <chem>Oc1c(Cl)c(Cl)c(Cl)c1#N</chem>
Syngenta Code:	R182281
Common Name:	None
CAS Name:	N/A
CAS Number:	28343-61-5
Molecular Weight:	247.46
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXIX-83
Purity:	98.2%
Expiration Date:	02/28/15

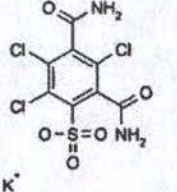
Compound Structure	
Syngenta Code:	R611966
Common Name:	None
CAS Name:	N/A
CAS Number:	Not available
Molecular Weight:	249.48
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXIX-89
Purity:	99.7%
Expiration Date:	01/31/15


Compound Structure	
Syngenta Code:	R611968
Common Name:	None
CAS Name:	N/A
CAS Number:	Not available
Molecular Weight:	265.48
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXIX-85-1
Purity:	99.7%
Expiration Date:	05/31/15

Compound Structure	
Syngenta Code:	R611965
Common Name:	None
CAS Name:	N/A
CAS Number:	Not available
Molecular Weight:	268.48
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXXX-37
Purity:	98.3%
Expiration Date:	05/31/15

Compound Structure	
Syngenta Code:	R613636
Common Name:	None
CAS Name:	N/A
CAS Number:	61073-19-6
Molecular Weight:	283.92
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXIX-95
Purity:	98.4%
Expiration Date:	04/30/15

Compound Structure	
Syngenta Code:	R417888
Common Name:	None
CAS Name:	N/A
CAS Number:	Not available
Molecular Weight:	329.54
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXX-70
Purity:	99.7%
Expiration Date:	03/31/15

Compound Structure	
Syngenta Code:	SYN510573
Common Name:	None
CAS Name:	N/A
CAS Number:	Not available
Molecular Weight:	385.64
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXXV-36
Purity:	90.7%
Expiration Date:	06/30/15

Compound Structure	
Syngenta Code:	SYN546669
Common Name:	None
CAS Name:	NA
CAS Number:	Not available
Molecular Weight:	375.16
Standard Reference:	Syngenta Crop Protection LLC
Storage Conditions:	4°C
Lot Number:	DAH-XXXV-55-2
Purity:	70.7%
Expiration Date:	08/31/16

Characterization data for the test/reference standards are maintained by Syngenta Crop Protection, LLC.

The test/reference substances (analytical standard) used in this study were procured from the Sponsor and stored as directed on "Analytical Standards Chain of Custody" documents. The stock standard solutions and fortification solutions were stored in a freezer at a nominal temperature of -20°C. The calibration standards were stored in a refrigerator at a nominal temperature of 4°C.

3.2 Test System

The test systems evaluated in this study were sandy loam and clay loam untreated (control) soil samples transferred from Syngenta Crop Protection, LLC and given PASC sample IDs PASC ID 130743-1 and 130743-2, respectively. The soil types were chosen because they are believed to represent the soil samples the method is designed for. These control soil samples were characterized by AGVISE Laboratories of Northwood, North Dakota and reported to Syngenta Archive under Syngenta Study Number TK0002309. GLP characterization results are presented in Table 1 and are summarized below:

Sample ID	Matrix	Sample Description
PASC ID 130743-1	Sandy Loam	Percent Sand: 55% Percent Silt: 28% Percent Clay: 17%
PASC ID 130743-2	Clay Loam	Percent Sand: 21% Percent Silt: 42% Percent Clay: 37%

The samples were received at PASC on August. 29, 2014, and were stored in a freezer at a nominal temperature of -20°C immediately after arrival. The control samples were checked

for contamination prior to use in this study by employing the same extraction and determination as described in Syngenta Method GRM005.07A, and they were found to be free of contamination for all of the analytes.

3.3 Equipment and Reagents

The equipment and reagents used for this study were selected and prepared as outlined in the method. Identical or equivalent equipment and materials may have been used, as permitted by the method.

3.3.1 Equipment

Equipment	Description	Supplier
General lab glassware	General lab glassware	Thermoscientific
General lab plastic ware	General lab plastic ware	Thermoscientific
Autosampler vials	Snap cap, 2 mL size	Thermoscientific
LC-MS/MS system Including HPLC and autosampler units	Acquity UPLC system; Sciex API 6500 triple quadruple mass spectrometer with Analyst TM software version 1.6.2	Waters Corporation Applied Biosystems
GC/MSD system Includes GC, MS and autosampler units	Agilent 6890N/HP 5973	Agilent
HPLC column	Zorbax SB-CN; 3.5µm 4.6 mm i.d x 75 mm	Agilent
GC column	J&W DB-1701, 30 m x 0.25 mm, 0.25 µm film thickness (catalog number 122-0732)	Agilent
Dispersive SPE	BONDESIL C18; 40µm	Agilent
SPE Cartridges	13mm, 0.45µm	Agilent
QuEChERS Dispersive kit (Cat. #5982-4956)	15-mL tube; 150 mg C18, 900 mg MgSO ₄	Agilent
Centrifugal Filter Devices	Ultrafree-CL 2 mL	Millipore

3.3.2 Reagents

Reagent	Description	Supplier
Ultrapure water	In house	Milli-Q water
Acetonitrile	HPLC grade	Pharmco
Methanol	HPLC grade	J.T Baker
Toluene	HPLC grade	Sigma Aldrich
Acetone	HPLC grade	J. T. Baker
Formic acid	A.C.S grade	EMD
Ethyl acetate	A.C.S. grade	Pharmoco
R182281, R611966 R611968, R611965 R613636, R417888 SYN510573 and SYN546669	GLP certified	Syngenta Crop Protection, LLC P.O Box 18300 Greensboro, NC 27419-8300

3.3.3 Preparation of Reagents

- a) 50/50 (v/v) Methanol/water, prepared by mixing 250 mL HPLC grade methanol with 250 mL Milli-Q water.
- b) 50/50 (v/v) Acetonitrile/water, prepared by mixing 250 ml HPLC grade acetonitrile with 250 mL Milli-Q water.
- c) 10/90 (v/v) Methanol/water, prepared by mixing 50 mL HPLC grade methanol with 450 mL Milli-Q water.
- d) 50/50 (v/v) Methanol/acetone, prepared by mixing 50 mL HPLC grade methanol with 50 mL HPLC grade acetone.
- e) 0.05% Formic acid in HPLC grade water; prepared by mixing 1.0 mL of formic acid with 2000 mL of Milli-Q water
- f) 0.05% Formic acid in acetonitrile; prepared by mixing 1.0 mL of formic acid with 2000 mL of acetonitrile.

3.4 Preparation of Standard Solutions

3.4.1 Stock Standard Solutions

Stock solutions were prepared for all analytes by accurately weighing sufficient analytical standards into individual amber "Class A" volumetric flasks (100 mL) and diluting to the marks with methanol. Final stock solution concentrations were approximately 100 µg/mL each for R182281, R611966, R611968, R611965, R613636 R417888, SYN510573 and SYN546669.

Stock Standard Solution Preparation

Compound	Weight (g)	Diluted to (mL)	Concentration (µg/mL)	Standard Solution ID
R611966	0.010258	100	102.27	S111814-1
R182281	0.010462	100	102.74	S111814-2
R611968	0.010350	100	103.19	S111814-3
R611965	0.011071	100	108.83	S111814-4
R613636	0.010292	100	101.27	S111814-5
R417888	0.010125	100	100.95	S111814-6
SYN510573	0.011309	100	101.78	S111814-7
SYN546669	0.013495	100	95.41	S111814-8

3.4.2 Fortification Standards

Mixed fortification solutions containing R611966, R182281, R611968, R611965, R613636 R417888, SYN510573 and SYN546669 were prepared at two concentrations levels (1.0 µg/mL and 0.1 µg/mL) by mixing appropriate amounts of the individual stock solutions and serial dilutions of with methanol.

Example of Fortification Standard Solution Preparation

Stock Solutions			Fortification			
Standard Solution ID	Compound	Concentration (µg/mL)	Aliquot Taken	Diluted to (mL)	Concentration (µL/ml)	Fortification Solution ID
S111814-1	R611966	102.27	0.489	50.0	1.00	F111814-1
S111814-2	R182281	102.74	0.487			
S111814-3	R611968	103.19	0.485			
S111814-4	R611965	108.83	0.459			
S111814-5	R613636	101.27	0.494			
S111814-6	R417888	100.95	0.495			
S111814-7	SYN510573	101.78	0.491			
S111814-8	SYN546669	95.41	0.524			
F111814-1	mixed	1.00	5.00	50.0	0.1	F111814-2

3.4.3 Calibration Standards

Calibration Standards for LC-MS/MS

Calibration standards were prepared by diluting the 1.0 µg/mL mixed fortification standard with 10/90 (v/v) methanol/ultrapure water. Calibration standard solutions were prepared at concentrations of 0.05 ng/mL, 0.1 ng/mL, 0.2 ng/mL, 0.5 ng/mL, 1.0 ng/mL, 2.0 ng/mL, and 5.0 ng/mL.

The solvent based calibration standards were used to quantify the residues in the validation sets of the two soil types. and the subsequent confirmation of matrix effect, matrix-matched calibration standards were prepared to quantify SYN546669 in the clay loam soil extracts.

The matrix-matched standards were initially prepared in 10/90 (v/v) methanol/ultrapure water, with concentrations of 0.5 ng/mL, 1.0 ng/mL, 2.0 ng/mL, 5.0 ng/mL, 10.0 ng/mL, 20.0 ng/mL, and 50.0 ng/mL as intermediate standards. Then 0.1 mL of each intermediate standard was mixed with 0.9 mL of the clay loam soil control sample extract (final fraction) to yield matrix-matched calibration standard solutions at concentrations of 0.05 ng/mL, 0.1 ng/mL, 0.2 ng/mL, 0.5 ng/mL, 1.0 ng/mL, 2.0 ng/mL, and 5.0 ng/mL. The matrix load is 90% for each standard solution.

Calibration curves were generated to quantify the residues of R182281, R611968, R611965, R417888, SYN510573 and SYN546669 using a 1/x weighing factor.

Calibration Standards for GC-MSD

Significant matrix effects were observed for determination of R611966 and R613636 by GC-MSD. Matrix-matched calibration standards for GC-MSD were prepared by diluting the 1.0 µg/mL mixed standards of R611966 and R613636 with the final soil extract solution. Calibration standard solutions were prepared at concentrations of 2.0 ng/mL, 5.0 ng/mL, 10 ng/mL, 20 ng/mL, 50 ng/mL, and 100 ng/mL. The matrix load is 90% and higher for each standard solution.

Example of Matrix-Matched Calibration Standard Solution Preparation for GC-MSD

Stock Solution			Calibration Solution				
Standard Solution ID	Compound	Conc. (ng/mL)	Aliquot Taken (µL)	Diluted To (µL)	Solvent	Conc. (ng/mL)	Intermediate Calibration Solution ID
S111814-1	R611966	102270	97.8	10,000	Toluene	1000	C120714-1
S111814-5	R613636	101270	98.7				
C120714-1	Mixed: R611966 and R613636	1000	20	200	Clay Soil Final Solution	100	C120714-2
C120714-2		100	100	200		50	C120714-3
C120714-3		50	80	200		20	C120714-4
C120714-4		20	100	200		10	C120714-5
C120714-5		10	100	200		5	C120714-6
C120714-6		5	80	200		2	C120714-7

3.5 Analytical Procedures and Modifications

3.5.1 Modifications

No modifications were made to the method except for the GC temperature program which was modified to yield higher peak resolutions. The details are listed in Section 3.6.1.

3.5.2 Sample Preparation and Fortifications

To conduct sample analysis, control soil bulk samples were taken from the storage freezer and thawed to room temperature prior to use. For each of the soil types, a total of 12 samples (20 grams each) were weighed into individual polypropylene centrifuge tubes (50 mL size) and analyzed as an analytical set: two control samples, five LOQ recovery samples and five 10X LOQ samples. Appropriate amounts of mixed fortification standard solutions were spiked into samples to yield the LOQ (0.005 mg/kg) and 10X LOQ (0.05 mg/kg) concentrations. A calibrated micropipette was used for the fortification. Two samples (untreated soil) were not spiked with fortification solutions and were used as control samples. In addition, one reagent blank sample was analyzed along with each of the analytical set.

Details about the fortifications are summarized as follows:

Matrix	Fortification Vol. (µL)	Fortification Conc. (µg/mL)	Sample Wt.(g)	Final Conc.(mg/kg)	Replicates
Sandy Loam Or Clay Loam	1000	0.1	20	0.005	5
	1000	1	20	0.05	5

3.5.3 Extraction Procedure

- a) To each of the samples 30 mL of 50/50 (v/v) methanol/ultrapure water was added and shaken at 300 csp for 30 minutes. Samples were centrifuged at approximately 6000 rpm with refrigeration at 10°C for about 10 minutes to separate liquid and solid phases.
- b) For each of the samples, the supernatant was transferred to a clean 50 mL polypropylene centrifuge tube and the soil solids were extracted again with 20 mL of 50/50 (v/v) acetonitrile/ultrapure water.
- c) For each of the samples, two supernatants were combined and added with methanol to a final volume of 50 mL.

3.5.4 Analysis of R182281, R611965, R611968, R417888, SYN546669, and SYN510573

For each of the samples, an aliquot of 1.00 mL was taken from the combined sample extract and transferred into a clean 15 mL polypropylene centrifuge tube. Next, 0.5 mL of methanol was added to the sample and diluted to 10 mL with ultrapure water for LC-MS/MS analysis

3.5.5 Analysis of R611966 and R613636

Each sample was treated with QuEChERS to extract R611966 and R613636 from aqueous component to volatile organic solvent. Additional solid phase extraction (SPE) was conducted for cleanup and concentration. The procedures are summarized below.

3.5.5.1 QuEChERS to force distribution/extraction of analytes

- a) 5 mL of ethyl acetate was added into each QuEChERS tube. After vortexing and being centrifuged, ethyl acetate was discarded, and the solid material was left in the tube.
- b) 4.0 mL of ethyl acetate was added into each conditioned QuEChERS tube, followed by 2.0 mL of the combined extract from Section 3.5.3 (c). The content of the tube was vigorously mixed, centrifuged and the liquid was collected into a clean 15 mL polypropylene centrifuge tube.
- c) A second extraction of 3 mL of ethyl acetate was performed. The liquid was combined into the respective 15 mL centrifuge tube in Section 3.5.5.1 (b).
- d) 1 mL of ultrapure water was added to the ethyl acetate fractions and evaporated the sample until aqueous (1 mL or less) under a gentle stream of N₂ in a bath set at a temperature of approximately 40°C.
- e) Samples were re-constituted by adding 1 mL of methanol, and then brought up to the 5 mL mark with ultrapure water.
- f) Each sample was vortexed and proceeded to the SPE clean-up procedure.

3.5.5.2 Solid Phase Extraction (SPE) Clean-up Procedure

- a) The SPE cartridges (Agilent Bond Elute, 500 mg, 6-mL) was conditioned with 3 mL of methanol, 3 mL of acetone and 3 mL of ultrapure water sequentially.
- b) The sample from Section 3.5.5.1 (f) was loaded onto the SPE cartridge. The flow rate was maintained at approximately 1 mL/min and the eluent was discarded.
- c) The used sample tube was rinsed with 2 mL of 20/80 (v/v) methanol/ultrapure water and the rinse was transferred to the SPE cartridge. The SPE cartridge was again washed three times each with 2 mL of 20/80 (v/v) methanol/ultrapure water.
- d) A vacuum was applied to each cartridge to allow complete dry for 10 minutes.
- e) For each sample, 3 mL of 50/50 (v/v) methanol/acetone was used to elute the analytes off the SPE cartridge into a clean glass tube.
- f) The eluent was evaporated to dryness under a gentle stream of N₂ in a bath set at a temperature of 40°C.
- g) For each glass tube, the sample was re-constituted with 1 mL toluene to yield the sample final fraction.
- h) The final fraction was vortexed and transferred into a GC vial for GC-MSD analysis.

3.6 Instrumentation

3.6.1 LC-MS/MS Instrument Description

LC System:	Waters Acquity UPLC system
MS Detector:	Applied Biosystems Sciex API 6500 triple quadrupole mass spectrometer with Analyst TM software version 1.6.2

Chromatographic Conditions

Flow Rate:	0.6 mL/min
Column:	Zorbax SB-CN, 4.6 x75 mm, 3.5 μ m (Agilent Cat. No. 866953-905)
Column Oven Temp:	40°C
Injection Vol.:	50 μ L
Run Time:	10 minutes
Mobile Phase A:	0.05% formic acid in DI water
Mobile Phase B:	0.05% formic acid in acetonitrile

Isocratic/Gradient Flow:

Time	% A	% B	Flow Rate (mL/min)
0.01	90	10.0	0.60
0.50	90	10.0	0.60
3.00	10	90.0	0.60
8.00	10	90.0	0.60
8.10	90	10.0	0.60
10.00	90	10.0	0.60

Approximate Retention Times of Analytes:

Analyte	Retention Time (min)
R182281	4.0
R611968	3.5
R611965	3.0
R417888	3.2
SYN510573	2.1
SYN546669	2.2

Mass Spectrometer Conditions

Interface: TurboIon Spray
Polarity: Negative
Curtain gas (CUR): Nitrogen set at 25
Temperature (TEM): 550°C
Ionspray voltage: -4200
Collision gas setting (CAD): Nitrogen set at 12 (arbitrary units)
Gas 1 (GS1): Air set at 55.00
Gas 2 (GS2): Air set at 50.00
Interface heater (ihe): On
Scan type: MRM

Analyte	MS/MS Transition	Dwell (ms)	DP	CE	CXP
R182281: ESI Negative					
Quantification	244.9→182.0	150	-37	-39	-9
Confirmation	244.9→174.9	150	-51	-38	-9
R611968: ESI Negative					
Quantification	263.0→220.0	150	-33	-23	-12
Confirmation	265.0→222.0	150	-22	-24	-10
R611965: ESI Negative					
Quantification	265.8→221.8	150	-7	-11	-12
Confirmation	267.8→223.9	150	-7	-11	-15
R417888: ESI Negative					
Quantification	326.8→219.8	150	-52	-33	-11
Confirmation	328.9→222.0	150	-53	-34	-12
SYN510573: ESI Negative					
Quantification	344.8→302.0	150	-71	-29	-16
Confirmation	346.9→304.0	150	-60	-30	-17
SYN546669: ESI Negative					
Quantification	372.9→355.9	150	-21	-18	-20
Confirmation	374.9→357.9	150	-25	-18	-20

3.6.2 GC-MSD Instrument Description

The following instrument and parameters were used to analyze R611966 and R613636.

Gas chromatography: Agilent 6890N

Mass spectrometry: HP 5973

Chromatographic Conditions

Column: J&W DB-1701, 30 m × 0.25 mm, 0.25 µm film thickness
(catalog number 122-0732)

Injection Port: Split/splitless injector, pulsed splitless mode
Restek drilled Uniliner[®] direct connect liner, with hole near top
and top taper (catalog number 20508)

Carrier Gas and Head Pressure: Helium at a constant flow of 1.0 ml/min with a pressure pulse of
30 psi for 1 minute

Splitless Time: 1.0 minute

Injection Volume: 3.0 µL

Injector Temperature: 250 °C

Temperature Program: Initial Temperature: 120°C Initial Time: 1.0 min

Rate	Final Temperature	Final Time
18°C/min	238°C	0.0 min
0.5°C/min	242°C	0.0 min
5°C/min	260°C	5.0 min
30°C/min	280°C	2.0 min

Post Temperature: 325°C Post Time: 2.0 min

MS Conditions

Ionization mode: EI
Electron energy: 70eV

Analyte	Low Mass Resolution	SIM Mode	
R611966	Yes	Target Ion	231.9 <i>m/z</i>
		Qualifier 1	233.9 <i>m/z</i>
		Qualifier 2	247.9 <i>m/z</i>
		Retention Time	14.7 min
R613636	Yes	Target Ion	267.9 <i>m/z</i>
		Qualifier 1	265.9 <i>m/z</i>
		Qualifier 2	269.9 <i>m/z</i>
		Retention Time	20.4 min

3.7 Data Acquisition

For LC-MS/MS analysis, peak integration and peak area count quantitation were performed by PE Sciex Analyst® (Version 1.6.2). A best-fit linear regression equation was derived and used in conjunction with the analyte response in each sample to calculate the concentration of analyte. The correlation coefficient (r) for the calibration curves for each analytical set was greater than 0.99. Recovery results were computed for each sample.

For GC-MSD analysis, peak response was generated by MSD Security ChemStation G1732BA B.02.00.589. Linear regressions were applied using Microsoft Office Excel package and the corresponding regression coefficients were greater than 0.99. The residue amounts were calculated based on the calibration curves and the recoveries were calculated per the equations provided by the method (Section 5.0).

A statistical treatment of the data includes the calculation of averages, standard deviations, and relative standard deviations. Mean percent recoveries, standard deviations, and relative standard deviations were calculated using a current Microsoft Office Excel package.

TABLE 1 Characterization Data of Soil Types Used for Method Validation

Sample ID	Soil Type	pH (1:1 soil:water)	Sand Content (%w/w)	Silt Content (%w/w)	Clay Content (%w/w)	Organic Carbon (%)
PASC ID; 130743-1	Sandy Loam ¹	7.7	55	28	17	4.9
PASC ID; 130743-2	Clay Loam ²	7.9	21	42	37	2.8

¹Soil from MCLAIN FARM 0-6"; TK0002309

² Soil from SCL GARDNER ND 0-6"; TK0002309

Note: The GLP characterization of this soil was performed by AGVISE Laboratories, 604 Highway 15, P.O. Box 510, Northwood, ND 58267.

TABLE 2 Soil Fortification Levels

Soil Type	Fortification Level (mg/kg)	Number of Recovery Samples
Clay Loam	Control	2
LOQ	0.005	5
10×LOQ	0.05	5

Soil Type	Fortification Level (mg/kg)	Number of Recovery Samples
Sandy Loam	Control	2
LOQ	0.005	5
10×LOQ	0.05	5