2.0 INTRODUCTION

Described in this report is the independent laboratory validation of Syngenta Residue Method GRM043.09A (Reference 1) as performed by PASC.

This study was designed to satisfy guideline requirements described in EPA 850.6100. This study was conducted in compliance with EPA FIFRA Good Laboratory Practice Standards, 40 CFR Part 160.

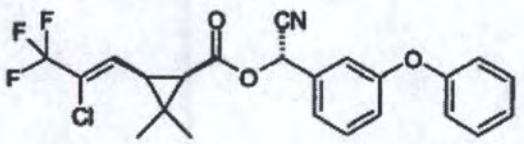
The residue analytical method is deemed suitable for the determination of lambda-cyhalothrin in water.

100 mL of water is transferred to a 200 mL polypropylene bottle, hexane is then added equal to 5% of sample volume (5 mL). The caps are securely tightened and samples placed on a mechanical shaker for two hours. Samples are centrifuged for 30 minutes at 3500 rpm and the upper extract layer (hexane) is transferred to a 15 mL polypropylene centrifuge tube. A 1.0 mL aliquot of final fraction is transferred to a GC autosampler vial. Final determination is performed by gas liquid chromatography with mass selective (GC-NICI) detection using negative ion chemical ionization.

3.0 MATERIALS AND METHODS

3.1 Test/Reference Substance

The test/reference substance was obtained from Syngenta Crop Protection, LLC. The following test/reference substance was used:

Compound Structure	
Batch Identification:	ASJ10012-04
Product Code:	PP321
Common Name:	Lambda-cyhalothrin
Storage Conditions:	Refrigerate < 10°C
Purity:	98.7%
Expiration Date:	03/2018

Characterization data for the test/reference standard are maintained by Syngenta Crop Protection, LLC. The Certificate of Analysis is included in Appendix 2.

The test/reference substance (lambda-cyhalothrin) used in this study was procured from Syngenta Crop Protection, LLC located at the Greensboro facility. All solutions made from lambda-cyhalothrin standard were stored according to Section 2 of the method.

3.2 Test System

The test system evaluated for this ILV was Surface Water (CAPY081512). This matrix was chosen because it is representative of the matrices the method was designed for. The control samples used in this study were provided by Syngenta Crop Protection, LLC. These control water sample were characterized by AGVISE Laboratories of Northwood, North Dakota. GLP characterization results in more detail are presented in Table 1 and summarized below:

Sample ID	pH	Calcium (ppm)	Magnesium (ppm)	Sodium (ppm)	Hardness CaCO3 (mg/L)	SAR	TDS (ppm)	Turbidity (NTU)
Surface water	8.6	4.3	2.4	2.0	21	0.19	84	3.79

3.3 Equipment and Reagents

The equipment and reagents used for the ILV were as outlined in the method. Identical or equivalent equipment and materials were used, as permitted by the method. All solvents and other reagents must be of high purity, e. g. glass distilled/HPLC grade solvents and analytical grade reagents.

3.4 Preparation of Standard Solutions

Standard solutions were prepared and stored as recommended in Section 2 of the method (Reference 1).

3.4.1 Stock Standard

One 101.8 µg/mL stock solution for lambda-cyhalothrin was prepared in hexanes.

3.4.2 Fortification Standard

Sample fortification solutions containing lambda-cyhalothrin were prepared by serial dilution in methanol from the stock solution. The following solutions were prepared: 0.01 µg/mL and 0.1 µg/mL for fortification purposes.

3.4.3 Calibration Standard

Calibration standards were prepared by serially diluting stock standards using hexanes. Using equivalent GC-MS instrumentation described in the method, the following concentration range of standards were prepared and used to construct the calibration plots for *m/z* ions 241, 205, and 243 (0.05, 0.1, 0.2, 0.5, 1.0, and 10 pg/µL).

3.5 Analytical Procedures and Modifications

Analytical Method GRM043.09A (Reference 1) was successfully validated by an independent laboratory as written using the procedures and instrumentation recommended by

the method. A 100 mL sample of surface water is partitioned using 5 mL of hexane for 2 hours by mechanical shaking in a 200 mL Nalgene bottle. The hexane (upper organic layer) is centrifuged and transferred into a suitable autosampler vial. Final determination is by gas chromatography with mass selective (GC-MS) detection using negative ion chemical ionization.

3.5.1 Modifications

Syngenta Analytical Method GRM043.09A (Reference 1) was followed as written. No modifications were made.

3.5.2 Fortifications

Untreated control water samples were fortified using 0.1 mL of known amounts of Lambda-cyhalothrin to LOQ and 10X LOQ concentration levels as per the method. See Table 2 for detailed fortification levels. Fortifications used in this ILV are as follows:

Matrix	Fortification Level	Fortification Volume (mL)	Fortification Conc. (µg/mL)	Final Volume (mL)	Replicates
Surface Water	LOQ	0.1	0.01	100	5
Surface Water	10X LOQ	0.1	0.1	100	5

3.5.3 Method Summary

As per Analytical Method GRM043.09A, a 100 mL sample of surface water is partitioned using 5 mL of hexane for 2 hours by mechanical shaking in a 200 mL Nalgene bottle. The hexane (upper organic layer) is centrifuged and transferred into a suitable autosampler vial. Final determination is by gas chromatography with mass selective (GC-MS) detection using negative ion chemical ionization mode for *(m/z)* ions 241, 205, and 243.

3.5.4 Limit of Detection and Limit of Quantitation

The limit of detection (LOD) of the method is defined as the lowest analyte amount injected on column 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 background noise. The LOD using the instrumentation for this validation was estimated as 0.05 pg/µL injected on column, equivalent to 0.2 pg when using a 4 µL injection volume. Note that the LOD may vary between runs and from instrument to instrument.

The limit of quantitation of the method is defined as the lowest analyte concentration in a sample at which the methodology has been successfully validated with a mean recovery of 70 - 110% and a relative standard deviation of $\leq$ 20%. A LOQ of 10 ng/L (10 ppt) in water was successfully validated in this study.