

Study Title (P-3446)

Magnitude of the Residues of zeta-Cypermethrin in/on Soybeans Treated with Fury[®] 1.5 EC Insecticide or Fury[®] 1.5 EW Insecticide

3.2.4 Analytical Method and Method Modifications

Residue analytical methodology employed for zeta-cypermethrin in/on soybean seed was slightly modified from the analytical method previously reported in FMC report P-3424 (Section 7, Reference 3). The detailed residue analytical methods employed for soybean seed can be found in Appendix II. The method flow scheme is presented in Figure 2.

The analytical methodology for zeta-cypermethrin in/on soybean seed involved extraction of the analyte from a homogenized matrix using an acetone/aqueous mixture. After centrifugation, the resulting extract solution was then partitioned with hexane and cleaned up with a silica gel SPE cartridge. The final solution containing zeta-cypermethrin was quantitated by a GC equipped with an ECD. The LOQ was validated at 0.05 ppm for zeta-cypermethrin by acceptable and reproducible recoveries of the analyte from laboratory fortified control samples. Based on a linear regression in each analytical data set, the LOD was calculated to be 0.03 ppm for zeta-cypermethrin in all soybean samples.

3.2.5 Instrumentation and Quantitation

An HP 6890 GC equipped with an HP 6890 autosampler, an ECD detector, a J&W Scientific DB-5 (95% polymethylsiloxane) megabore capillary column, and a PE Nelson Turbochrom[®] computer software package was used for the quantitation of zeta-cypermethrin. An HP 6890 GC equipped with an HP 6890 autosampler, an HP 5972 MSD, a J&W Scientific DB-5 narrowbore capillary column and a MS ChemStation (Window Based) was used qualitative confirmation of zeta-cypermethrin residues. Detailed instrument parameters for all detector systems are listed in Appendix III.

zeta-Cypermethrin residues were quantitated by a multiple point external standard calibration method. A computer spreadsheet program (Microsoft[®] Excel 97) was used for calculation and reporting. A full description of the calculations for quantitation can be found in Appendix IV.

Appendix II Residue Method (P-2446)

Analytical Method for zeta-Cypermethrin in/on Soybean Seed

1. Weigh 5 grams of sample into a 50 mL polypropylene centrifuge tube. Add ~40 mL of acetone/water (70/30). Screw cap in place.
2. Horizontally shake the centrifuge tube for ~30 minutes. Centrifuge at 2000 rpm for ~3 minutes.
3. Decant the solution into a 100 mL graduated cylinder.
4. Repeat the extraction with another ~40 mL of acetone/water (70/30) and centrifugation.
5. Decant the solution into the above 100 mL graduated cylinder. Bring final volume to 100 mL. Stopper and invert the cylinder to mix.
6. Place a 20 mL aliquot (1 gram) into another 50 mL Corning clear polypropylene centrifuge tube. Add ~2 grams of NaCl.
7. Partition acetone/aqueous twice with ~20 mL hexane and ~10 mL of hexane, respectively. Centrifuge at 2000 RPM for 3 minutes if emulsions occur.
8. Pipet each hexane layer into a 50 mL Corning clear polypropylene centrifuge tube.
9. Concentrate the combined hexane layers on the N-Evap (use a temperature setting at ~40°C and nitrogen setting at ~10 psi) to ~1 mL.
10. Condition a 1 gram Silica gel SPE cartridge with one CV of 10% ethyl acetate/hexane followed by one CV of hexane. Allow hexane to drain to the top of the frit. Load the ~1 mL sample onto the cartridge. Rinse the centrifuge tube with ~1 mL of hexane and add the rinse to the cartridge. Allow hexane to drain to top of frit.
11. Wash the cartridge with 3 mL of hexane. Discard wash.
12. Elute the zeta-cypermethrin off the cartridge with 12 mL of 5% ethyl acetate/hexane and collect in a 13 mL centrifuge tube.
16. Concentrate on the N-Evap to less than 1 mL and adjust final volume with hexane to 1 mL or required final volume.
17. Inject on GC/ECD.

Appendix III

Instrument Parameters

INSTRUMENT:	HP 6890 GC
COLUMN:	J&W DB-5, 5% phenyl methyl silicone, 15 m x 0.53 mm, 1.0 μ m film thickness
INLET:	Splitless Injection Mode (Cyclo-double gooseneck liner)
DETECTOR:	Electron Capture Detector
TEMPERATURE PROGRAM:	
Injection Port:	250°C
Oven:	200°C/1 minute (initial) 14°C/minute (ramp) 280°C/8.2 minutes (hold)
Detector:	300°C
COLUMN GAS FLOW:	He. Carrier, ~11.5 mL/minute
DETECTOR GAS FLOW:	Argon/10% methane, ~60 mL/minute
INJECTION VOLUME:	2 μ L
RUN TIME:	~15 minutes
RETENTION TIME:	~6.2 minutes (zeta-cypermethrin)

Instrument Parameters
Confirmation of zeta-cypermethrin

INSTRUMENT:	HP 6890 GC/5972A MSD
COLUMN:	J&W DB-5MS, 5% phenyl methyl silicone, 15 m x 0.25 mm, 0.25 μ m film thickness
INLET:	Splitless Injection Mode (Cyclo-double gooseneck liner)
DETECTOR:	Mass Selective Detector
TEMPERATURE PROGRAM:	
Injection Port:	250°C
Oven:	120°C/1 minute (initial) 10°C/minute (ramp 1) 200°C/0 minute (hold) 20°C/minute (ramp 2) 280°C/3 minutes (hold)
Transfer line:	280°C
COLUMN GAS FLOW:	He, Carrier, ~1 mL/minute
INJECTION VOLUME:	2 μ L
RUN TIME:	16 minutes
RETENTION TIME:	~6.8 minutes (zeta-cypermethrin)
ION MONITORED:	141, 169, and 198