ANALYTICAL METHODOLOGY

Analytical Standards

The following analytical standards were used for fortifications and calibrations:

Standard ID	Lot Number	Purity (Percent)	Expiration Date	Date Received at Maxim	Maxim Reference Number
quinoxyfen (1)	DECO-36-90A	99.8%	30-Apr-02	12-May-99	1007001
3-OH quin. (2)	B463-182	>98%	03-May-01	12-May-99	1007002

Determination of Residues of Quinoxyfen and 3-OH Quinoxyfen

Soil samples were extracted using Dow AgroSciences Analytical Method ERC 94.27 (3). Residues of quinoxyfen and 3-OH quinoxyfen were extracted by shaking with acidic acetone. After the addition of sodium bicarbonate solution to the extract, both analytes were partitioned into hexane which was then evaporated to dryness. The 3-OH quinoxyfen was then methylated, and both analytes were extracted from the aqueous layer into methyl tertiary butyl ether. The ether layer was evaporated to dryness, and the residuum was reconstituted in hexane. The hexane aliquot was then cleaned up using an aminopropyl solid phase extraction column. The compounds of interest were then eluted off the column using 5% acetone in hexane. The eluate was evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane containing 0.2 µg/mL 1,4-dibromonaphthalene as an internal standard. A portion of the tri-methyl pentane extract was then analyzed by capillary gas chromatography with mass selective detection.

Instrumentation - Quinoxyfen and 3-OH Quinoxyfen Analysis

Sample extracts were analyzed using a Hewlett Packard Model 5890 Gas Chromatograph/ Hewlett Packard Model 5971 Mass Selective Detector (MSD) with either a 12 m x 0.2 mm i.d. x 0.33 µm film thickness HP-Ultra 2 or HP-5MS fused silica capillary column. (The HP-5MS is an equivalent substitution for the HP-Ultra 2.) Residues of 3-OH quinoxyfen were quantified as "3-methoxy quinoxyfen" using calibration standards that were prepared in the laboratory via methylation of 3-OH quinoxyfen standards.

The ions that were monitored for each of the compounds are listed below.

- 1,4-dibromonaphthalene: 286 amu
- quinoxyfen: 237 amu (target) and 272 amu (confirmatory)
- 3-methoxy quinoxyfen: 337 amu (target) and 320 amu (confirmatory)

CALCULATIONS

Determination of Quinoxyfen Residues in Soil

The calculations for 3-OH quinoxyfen (which was quantified as "methylated 3-OH quinoxyfen") are similar to quinoxyfen; therefore, a separate section will not be added. Any differences from the quinoxyfen calculations for calculating 3-OH quinoxyfen residues will be noted in this section (if applicable).

A series of calibration standards ranging from 0.05 to 1.00 µg/mL each quinoxyfen and "methylated 3-OH quinoxyfen" (identified on chromatograms as "3-hydroxy-quinoxyfen), each containing 0.2 µg/mL 1,4-dibromonaphthalene as an internal standard, were injected with each set of samples. A five (5) concentration level standard curve, injected in duplicate (i.e., once prior to and once following injection of sample extracts for a total of ten (10) standard injections), was used to construct a calibration curve for quantitation of residues. The calibration

curve was prepared by plotting the analyte concentration of the standards on the abscissa (x-axis) and the corresponding quantitation ratio (i.e., peak area of quinoxyfen/peak area of internal standard) on the ordinate (y-axis). Linear regression was used to determine the slope (m) and y-intercept (b) for the calibration curve.

$$y = mx + b$$

To solve for "x," the equation is rearranged as follows:

$$x = \frac{(y - b)}{m},$$

where: y = *m/z* 237/286 peak area (i.e., Peak Area Quinoxyfen / Peak Area Internal Standard)
(*m/z* 337/286 peak area for "3-OH Quinoxyfen")
x = Quinoxyfen concentration (µg/mL)

The concentration in each sample extract was determined by substituting the *m/z* 237/286 peak area ratio obtained for quinoxyfen into the equation for the calibration curve and solving for the concentration.

$$\text{Quinoxyfen Conc. } (\mu g/mL) = \frac{(m/z\ 237/286 \text{ peak area ratio} - b)}{m}$$

For example, using the data for 7817416A from Figure 1:

$$\text{Quinoxyfen Conc. } (\mu g/mL) = \frac{((10574992 / 5457744) - (-0.12246))}{7.63389}$$

$$= 0.270\ \mu g/mL$$

The "ppm" (µg/g) of quinoxyfen in each soil sample was determined as follows:

$$\text{ppm Found} = \frac{C \times FV \times DF}{W},$$

where: C = Quinoxyfen concentration (µg/mL) from curve
FV = final volume (mL) = 1.0
DF = dilution factor
W = sample weight (g) = 5.0

For example, using the data for 7817416A from Figure 1:

$$\text{ppm Found} = \frac{0.270\ \mu g/mL \times 1.0\ mL \times 1}{5.0\ g}$$

$$= 0.054\ \mu g/g$$

If an interfering peak was detected in the *reagent blank*, then the quantitation ratio was used to calculate a "ppm Found" value which was then *subtracted from the "ppm Found" values for the treated samples* extracted with that set. If an interfering peak was detected in the *control soil sample*, then the quantitation ratio was used to calculate a "ppm Found" value which was then *subtracted from the "ppm Found" values for the recovery samples* extracted with that set.

Determination of Percent Recovery for Quinoxyfen

The percent recovery was determined by dividing the "ppm Found" for each recovery sample by the theoretical amount ("ppm") added.

$$\% \text{ Recovery} = \frac{\text{ppm Found}}{\text{ppm Added}} \times 100\%$$

For example, using the data for 7817406A from Figure 1:

$$\% \text{ Recovery} = \frac{0.813 \text{ ppm}}{1.000 \text{ ppm}} \times 100\%$$

$$= 81.3 \%$$

Determination of "Corrected" Quinoxyfen Residues in Soil

The "corrected ppm" (i.e., ppm value which includes correction for the average sequence percent recovery and the percent soil moisture) of quinoxyfen in each soil sample was determined as follows:

$$\text{ppm Found} = \frac{C \times FV \times DF \times (1 + (M/100))}{W \times \text{Avg. Sequence \% Recovery}},$$

where: C = Quinoxyfen concentration (µg/mL) from curve
FV = final volume (mL) = 1.0
DF = dilution factor
M = percent soil moisture
W = sample weight (g) = 5.0

For example, using the data for 7817416A from Figure 1:

$$\text{ppm Found} = \frac{0.270 \text{ µg/mL} \times 1.0 \text{ mL} \times 1 \times (1 + (11.6/100))}{5.0 \text{ g} \times .899}$$

$$= 0.067 \text{ µg/g}$$