Analytical Method for the Determination of Cyromazine and Melamine Residues in Soil Using LC-UV and GC-MSD

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A method is reported for the determination of cyromazine and melamine residues in soil. Soil samples are extracted twice via mechanical shaking, each time with 70% acetonitrile/30% 0.050 M ammomium carbonate for 30 min. An aliquot portion of the pooled extracts is subjected to strong cation exchange (SCX) purification on AG 50W-X4 resin. Final analysis is accomplished using liquid chromatography−ultraviolet (LC-UV) detection at a wavelength of 214 nm. Confirmatory analyses can be performed using gas chromatography−mass selective detection (GC-MSD) in the selected ion monitoring (SIM) mode. The limit of detection (LOD) is 2.5 ng injected and the limit of quantification (LOQ) is 10 ppb when using LC-UV for the analysis of *N*-cyclopropyl-1,3,5-triazine-2,4,6-triamine (cyromazine) and 1,3,5-triazine-2,4,6-triamine (melamine). The LOD is 0.050 ng injected and the LOQ is 10 ppb when using GC-MSD for confirmatory analyses. The mean procedural recoveries were 97 and 95% and the standard deviations were 16 and 11% for cyromazine and melamine, respectively ($n = 24$), when using LC-UV. The mean procedural recoveries were 107 and 92% and the standard deviations were 9.9 and 16% for cyromazine and melamine, respectively ($n = 29$), when using GC-MSD. The method validation study was conducted under U.S. EPA FIFRA Good Laboratory Practice Guidelines 40 CFR 160. The method also passed an Independent Laboratory Validation (ILV) as per U.S. EPA FIFRA Subdivision N.

Keywords: *Cyromazine; melamine; soil; liquid chromatography−ultraviolet detection; gas chromatography−mass selective detection; Good Laboratory Practices (GLP)*

INTRODUCTION

Cyromazine is an insect growth regulator used as a feed-through larvicide to control flies on animals and as a foliar spray to control leafminers on ornamental plants, fruits, and vegetables. The active ingredient is manufactured and formulated by Novartis Crop Protection, Inc., and registered with the U.S. EPA under the trademarks Larvadex and Trigard. It can metabolize via dealkylation reactions in both plants and animals and undergo environmental degradation to form melamine (Cook and Hutter, 1981; Cook et al., 1984; Lim et al., 1990; Roberts and Hutson, 1998). Melamine is routinely used in the manufacture of plastics that are molded into tableware and cups. It can migrate from these household products into food at higher temperatures and acidic pH (Inque et al., 1985; Ishiwata et al., 1986). Other uses of melamine include applications in electrical equipment, adhesives, laminates, permanent-press fabrics (Updegraff et al., 1979), fire-retardant textile finishes and tarnish inhibitors (May, 1979), coatings and paper (Green and Li, 1983), and fertilizer urea mixtures as a slow release source of nitrogen (Arcement and Levy, 1988). Melamine is found as a contaminant in wastewater (Burrows et al., 1984). Due to its classification as a noncarcinogen in humans, melamine is no longer included as part of the tolerance expression for cyromazine residues. There is no evidence that cyromazine is a carcinogen (*Federal Register*, 1999). The chemical names and structures of these compounds are shown in Figure 1.

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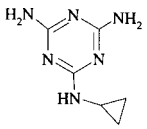
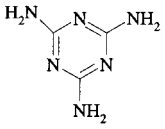


Common Name: Cyromazine

Chemical Name: N-cyclopropyl-1,3,5-triazine-2,4,6-triamine

CAS Registry No.: 66215-27--8

Experimental No.: G-28522



Common Name: Melamine

Chemical Name: 1,3,5-triazine-2,4,6-triamine

CAS Registry No.: 108-78-1

Figure 1. Chemical names and structures for cyromazine and melamine.

Terrestrial field dissipation of Trigard studies were conducted by Novartis Crop Protection, Inc., according to U.S. EPA registration guidelines (U.S. EPA, 1982, 1986). These studies were designed to evaluate the mobility and persistence of cyromazine and its degradation product melamine in a worst-case scenario (i.e.,

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when applied to bare soil) and were conducted at locations in California that were representative of Trigard use (Speth and Rezaaiyan, 1999; Speth and Yokley, 1999). The results of these terrestrial field dissipation studies are reported elsewhere (Jacobson and Speth, 1999; Mester et al., 1999). To support these studies, an analytical method for the analysis of cyromazine and melamine in soil was required.

Analytical methods for the analysis of melamine in industrial applications have been reported (Arcement and Li, 1988; Beilstein et al., 1981; Burrows et al., 1984; Inque et al., 1985; Ishiwata et al., 1985, 1987; Stoks and Schwartz, 1979; Sugita et al., 1990). Gas chromatography−mass selective detection (GC-MSD) (Toth and Bardalaye, 1987) and liquid chromatography (Cabras et al., 1990) operating parameters have been described for the separation and analysis of cyromazine and melamine. However, very few sample preparation procedures for these two analytes have been reported (Bardalaye et al., 1987), and none was found for the analysis of soil. Thus, an analytical method for the analysis of cyromazine and melamine in soil, validated under Good Laboratory Practice Standards, and acceptable to the EPA (U.S. EPA, 1989a), was needed to support Novartis Crop Protection, Inc., terrestrial field dissipation studies. In this study, soil samples were extracted and then subjected to purification using a strong cation-exchange resin prior to analysis using either LC-UV or GC-MSD.

EXPERIMENTAL PROCEDURES

Solvents and Reagents. HPLC grade solvents methanol (A452-4) and acetonitrile (A998-4) and HPLC grade ammonium carbonate (A652-3), Optima grade acetone (A949-4), and reagent grade ammonium hydroxide (A669-212) and potassium phosphate monobasic (P285-500) were all obtained from Fisher Scientific Co.

Preparation of Solutions. A 5% ammonium hydroxide solution in methanol was prepared by mixing 5 mL of the concentrated base with 95 mL of methanol. A 90% acetonitrile/10% water solution was prepared by mixing 90 mL of acetonitrile with 10 mL of water (v/v). A 90% methanol/10% water solution was prepared by mixing 90 mL of methanol with 10 mL of water (v/v). The aqueous portion of the extraction solvent was prepared by weighing 4.8 g of ammonium carbonate and diluting to 1 L with deionized water to make a 0.050 M solution. The aqueous portion of the LC mobile phase was prepared by weighing 2.04 g of KH_2PO_4 into 1 L of water followed by adjustment to pH 3.0 using phosphoric acid (~1 mL) to make a 15 mM solution.

Preparation of the SCX Column. A strong cation exchange (SCX) resin was prepared for use by adding 500 mL of deionized water to 200−250 g of resin (Bio-Rad AG-50W, 200−400 mesh, catalog no. 142-1351) in a 1-L Erlenmeyer flask. The resin was gently shaken (to minimize fracturing of the resin) and allowed to settle. The water was then decanted (to remove the fines) and replaced with a fresh 500-mL portion of deionized water. The resin must be allowed to equilibrate overnight. The addition of a couple of drops of concentrated HCl is recommended to help ensure that the resin remains in the hydrogen form if the resin is to be stored for several days prior to use.

A resin column bed was prepared by transferring 20−25 mL (in a 25 mL pipet) of the suspended resin/water slurry to a Poly-prep column (Bio-Rad, 10-mL capacity, catalog no. 731-1550) that was attached to a Vac-Elut processing station (Varian, catalog no. 1223-4001). The slurry was drained from the pipet until a resin bed volume of 2 mL was attained. The water from the slurry was drained from the column under gravity until the water level reached the top of the resin (do not allow the top of the resin bed to dry). The column was ready for use.

Table 1. Characterization Data for the 0−6-in. Layer Soil Collected in Santa Cruz and Madera Counties, California

characteristic	Santa Cruz County	Madera County
% sand	56	76
% silt	28	16
% clay	16	8
USDA textural class (hydrometer method)	Elder sandy loam	Hanford fine sandy loam
bulk density (g/cm^3)	1.23	1.50
cation exchange capacity (mequiv/100 g)	16.9	4.6
% moisture at 0.33 bar	21.1	6.4
% moisture at 15 bar	8.6	2.6
% organic matter	2.2	0.20
pH	6.9	6.9
Olsen phosphorus (ppm)	18	18
% total nitrogen	0.023	0.023
soluble salts (mmhos/cm)	0.060	0.060
calcium (%/ppm)	56.2/1900	54.6/500
magnesium (%/ppm)	21.2/430	21.8/120
sodium (%/ppm)	1.0/39	2.5/26
potassium (%/ppm)	6.9/458	5.4/97
hydrogen (%/ppm)	14.7/25	15.7/7

Standards. Analytical standards of cyromazine (98.5%) and melamine (99%) were obtained from the Analytical and Product Chemistry Department, Novartis Crop Protection, Inc., P.O. Box 18300, Greensboro, NC 27419-8300.

For LC-UV calibration purposes, stock solutions of each standard were prepared by weighing 100.0 mg of cyromazine and melamine (all corrected for percent purity) into each of two 100-mL volumetric flasks to provide concentrations of 1000 μg/mL. The cyromazine stock standard was diluted to the mark with methanol. The melamine stock standard was diluted to the mark with 20% water/80% methanol to assist with the dissolution of melamine. A 10.0 μg/mL mixed standard was prepared by mixing 1.0 mL of the cyromazine and melamine stock solution standards in a 100-mL volumetric flask followed by dilution to the mark with water. Serial dilutions of the 10.0 μg/mL mixed standard were then prepared with mobile phase to produce calibration standards of 0.050−3.0 μg/mL. Injections of 50.0 μL of each standard produced a calibration range of 2.5−150 ng. Selected mixed analytical standards can be used to fortify control soil samples for procedural recovery purposes. For example, the addition of 1.0 mL of the 0.20 μg/mL analytical standard to a 20-g soil sample results in analyte concentrations of 10 ppb (the LOQ of the method). The analysis of control and fortified control soil samples provides procedural recovery and method performance data. The volume of standard used to fortify samples should not exceed 2 mL.

For GC-MSD calibration purposes, stock solutions of each standard were prepared by weighing 100.0 mg of cyromazine and melamine (all corrected for percent purity) into each of two 100-mL volumetric flasks to provide concentrations of 1000 μg/mL. The cyromazine stock standard was diluted to the mark with acetone, and the melamine stock standard was diluted to the mark with 85% acetonitrile/15% water to assist with the dissolution of melamine. A 10 μg/mL mixed standard was prepared by transferring 1.0 mL of each stock solution to a 100-mL volumetric flask and reducing the solution to dryness by using a gentle stream of dry nitrogen gas. The analytes were then diluted to the mark with acetone. Serial dilution of the mixed standard in acetone provided calibration standards from 0.025 to 1.0 μg/mL; thus, a 2-μL injection produced a calibration range from 0.050 to 2.0 ng injected. Selected mixed analytical standards can be used to fortify control soil samples for procedural recovery purposes.

Soil Types for the Method Validation. Soil characterization data for the two soil types evaluated in this study are shown in Table 1. These soil samples were obtained from terrestrial field dissipation study sites in Santa Cruz and Madera Counties, California, thus, the need to validate the method using control soil samples from these locations.

Table 2. Retention Time, Target and Qualifier Ions, and Qualifier/Target Ion Ratios Used for the GC-MSD Analyses

analyte	retention time[a] (min)	target ion (*m*/*z*)	Q1 (*m*/*z*)	Q2 (*m*/*z*)	Q1/target ion ± 20% acceptance range[b]
cyromazine	14	151	165	166	24.9−37.4
melamine	13	126	85	68	27.9−41.8

[a] This will vary according to column length and other operating parameters. [b] This confirmation ratio will vary slightly from analytical set to analytical set.

Sample Storage. Soil samples to be analyzed for residues of cyromazine and melamine should be stored frozen (−20 °C) until analyzed. The results of a storage stability study indicate that these compounds are stable in soil under freezer conditions for at least 18 months (Speth and Rezaaiyan, 1999; Speth and Yokley, 1999).

Sample Preparation. Soil core sections (representing each specific depth interval) should be prepared to achieve complete homogeniety. A 20-g portion of soil was weighed directly into a 250-mL Nalgene bottle (Fisher catalog no. 02-893-5D). To this was added 100 mL of extraction solvent (70% acetonitrile/30% 0.050 M ammonium carbonate). The bottle was hand shaken vigorously for 15 s followed by mechanical shaking for 30 min at ∼300 oscillations per minute (e.g., LabLine Orbital shaker or equivalent). The sample was centrifuged for 10 min at 9000 rpm using a Sorvall RC-5B Superspeed centrifuge at ∼0 °C. The supernate was carefully decanted into a 250-mL Erlenmeyer flask (extracts should not be filtered through filter paper because some contain trace levels of melamine). A second 100-mL portion of extraction solvent was added, and the sample was again shaken for 30 min. The sample was centrifuged and the supernate decanted into the same 250-mL Erlenmeyer flask containing the first extract. A 100-mL aliquot portion of the pooled extract was transferred to a 100-mL graduated cylinder and adjusted to pH ≤2.0 with 4 N HCl.

Under slight vacuum, the cation-exchange resin bed (prepared as described above) was washed with 10 mL of deionized water at a rate of 1−2 drops per second. A reservoir was attached (Varian, 60-mL capacity, catalog no. 1213-1012) to the Poly-Prep column, and the 100 mL of pooled and pH adjusted extract was loaded under slight vacuum at a rate of 5 mL/min. The eluate was discarded. The column was then washed successively, at a rate of 5 mL/min, with 50 mL of 90% acetonitrile/10% water (v/v), 50 mL of 90% methanol/10% water (v/v), and finally 10 mL of methanol. All of the eluates were discarded. The analytes were eluted with 20 mL of 5% ammonium hydroxide/95% methanol (v/v) at a rate of 5 mL/min and collected in a 50-mL centrifuge tube. Water (0.50 mL) was added as a "keeper", and the sample was evaporated to dryness using a rotary evaporator at a water bath temperature of 30−35 °C. Small quantities (1−2 mL) of methanol and then acetone were added to the sample to form an azeotrophe. This assisted with the removal of water and reduced the time required for the concentration step. Upon reaching dryness, the residue was reconstituted in LC mobile phase (for immediate analysis) or water (if >2 days before analysis) for LC-UV analysis or acetone for GC-MSD analysis. Samples reconstituted in water could be injected directly without the need to switch to the LC mobile phase. A final volume of 1.0 mL was used to establish the method LOQ. For LC-UV analysis, the final fraction was filtered through a 0.20-μm nylon acrodisc filter (Gelman catalog no. 4427).

Each method validation sample set consisted of a reagent blank and a control plus three procedural recovery samples at 10 ppb, two at 50 ppb, and one at 100 ppb. The control and recovery samples were 0−6 in. layer soil samples collected in the untreated plots at the field dissipation study sites.

Instrumentation. The LC-UV analyses were performed using a Waters model 600 controller and solvent delivery system, a SpectraFlow model 783 UV detector, and a Waters 717 autosampler (or equivalent system). The separation was achieved using a Zorbax SCX column (4.6 mm × 15 cm, 5-μm particle size) and an isocratic mobile phase consisting of 25%

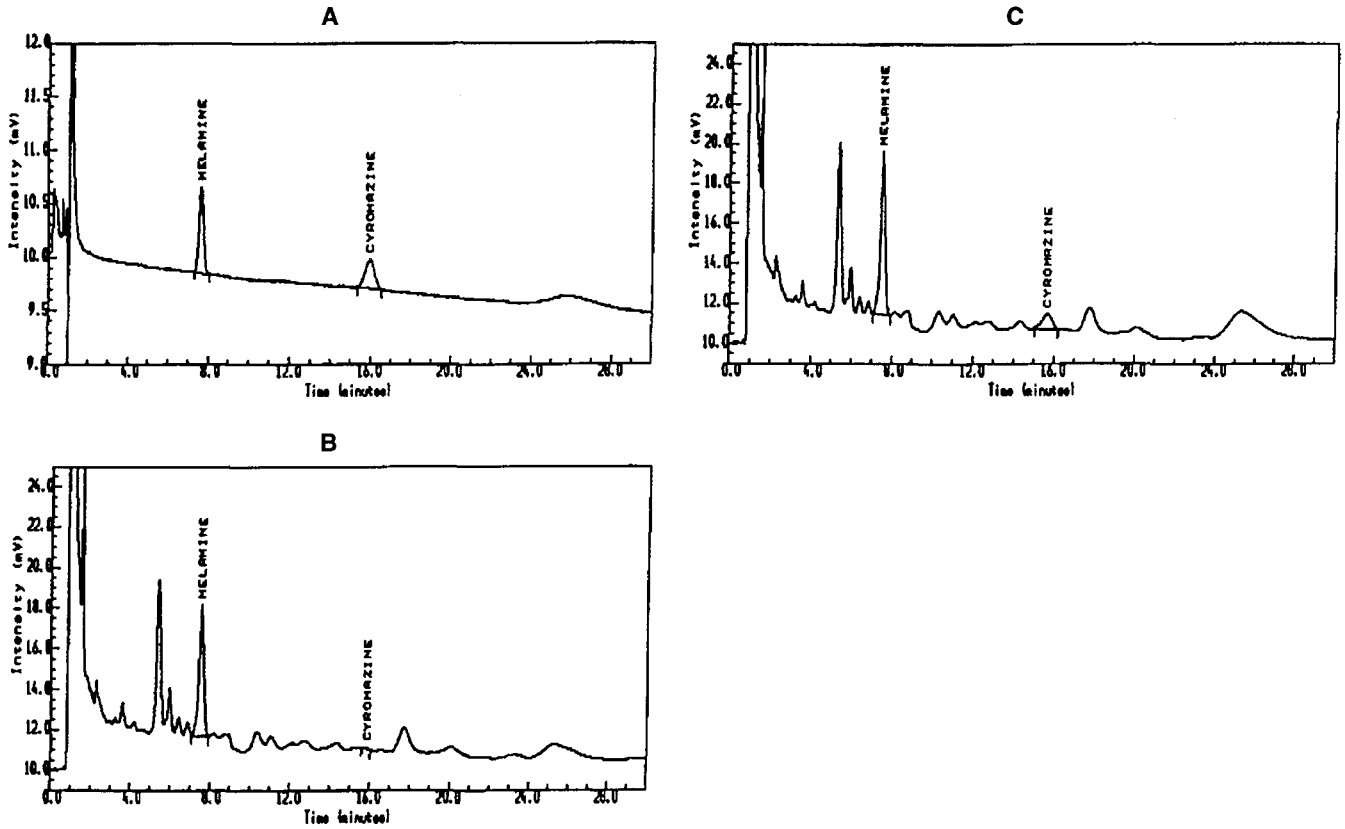

Figure 2. Representative LC-UV chromatograms of cyromazine and melamine in Santa Cruz County soil: (A) 2.5 ng injected concentration level; (B) control; (C) 10 ppb procedural recovery sample.

Table 3. Temperature Program Used during the GC-MSD Analyses

parameter	value
oven, initial temp (°C)	110
oven, initial time (min)	0.5
ramp 1 rate (°C/min)	35
final temp (°C)	250
final time (min)	16
injector temp (°C)	225
MSD temp (°C)	280
column head pressure (0 time) (psi)	12

methanol/75% potassium phosphate buffer at a flow rate of 2 mL/min. The injection volume was 50 μL, and UV detection was performed at a wavelength of 214 nm. Multichrom software was used for data acquisition and processing.

GC-MSD confirmation analyses were performed using either a Hewlett-Packard model 5890 series II gas chromatograph interfaced (capillary direct) to a 5972 mass selective detector (GC-MSD) or a Hewlett-Packard 6890 series GC/MSD, both operated in the selected ion monitoring (SIM) mode. The ions of interest for each analyte, shown in Table 2, were obtained after inspection of their full-scan mass spectra obtained via electron ionization (EI) at 70 eV. The MSD transfer lines were maintained at 280 °C, and tuning was performed on a daily basis with perfluorotributylamine (PFTBA) to ensure accurate mass calibration. The GCs were equipped for splitless injection, and J&W DB-Wax capillary columns (0.25 mm i.d. × 30 m, 0.25-μm film thickness) were employed for the separation. Electronic pressure programming (EPP) was utilized in conjunction with the temperature program detailed in Table 3.

During the method validation, the final fractions analyzed using GC-MSD were obtained from the extraction of separate soil samples from those final fractions analyzed using LC-UV. However, during the conduct of field dissipation studies, a portion of each soil sample extract was analyzed using LC-UV while the remaining portion was stored and analyzed later using GC-MSD when confirmatory analyses were required.

System Suitability Testing. A typical analytical set consisted of at least six analytical standards of various concentrations, a blank, a control, two controls fortified with the two analytes at the 10 and 100 ppb concentration levels for procedural recovery purposes, and 6−10 samples for analysis. Additional standards were dispersed throughout the run as a means of checking the stability of the system for variances in detector sensitivity and/or column performance. They were not used to construct the calibration plot. A minimum of two were used, and an analytical set terminated with one of these "stability check" or "quality control" standards (Jenke, 1996a−c).

RESULTS AND DISCUSSION

LC-UV Analyses. Representative chromatograms of cyromazine and melamine are shown in Figure 2 for a standard at the 2.5 ng injected concentration level and a control and a fortified control (procedural recovery) sample at the 10 ppb fortification level in Santa Cruz County soil. The 2.5 ng injected standard represents the lowest concentration standard [limit of detection (LOD)] used for construction of the calibration plot. The calibration plots were linear and the correlation coefficients were all ≥0.995 for both analytes (based on integrated peak areas). A primary criterion for selection of these sites as locations for the Trigard terrestrial field dissipation studies was documented evidence that this product (cyromazine) had not been used previously at these locations. Nevertheless, the Santa Cruz County soil contained background levels of melamine averaging ~40 ppb. This is demonstrated by the presence of melamine in the control sample chromatogram shown

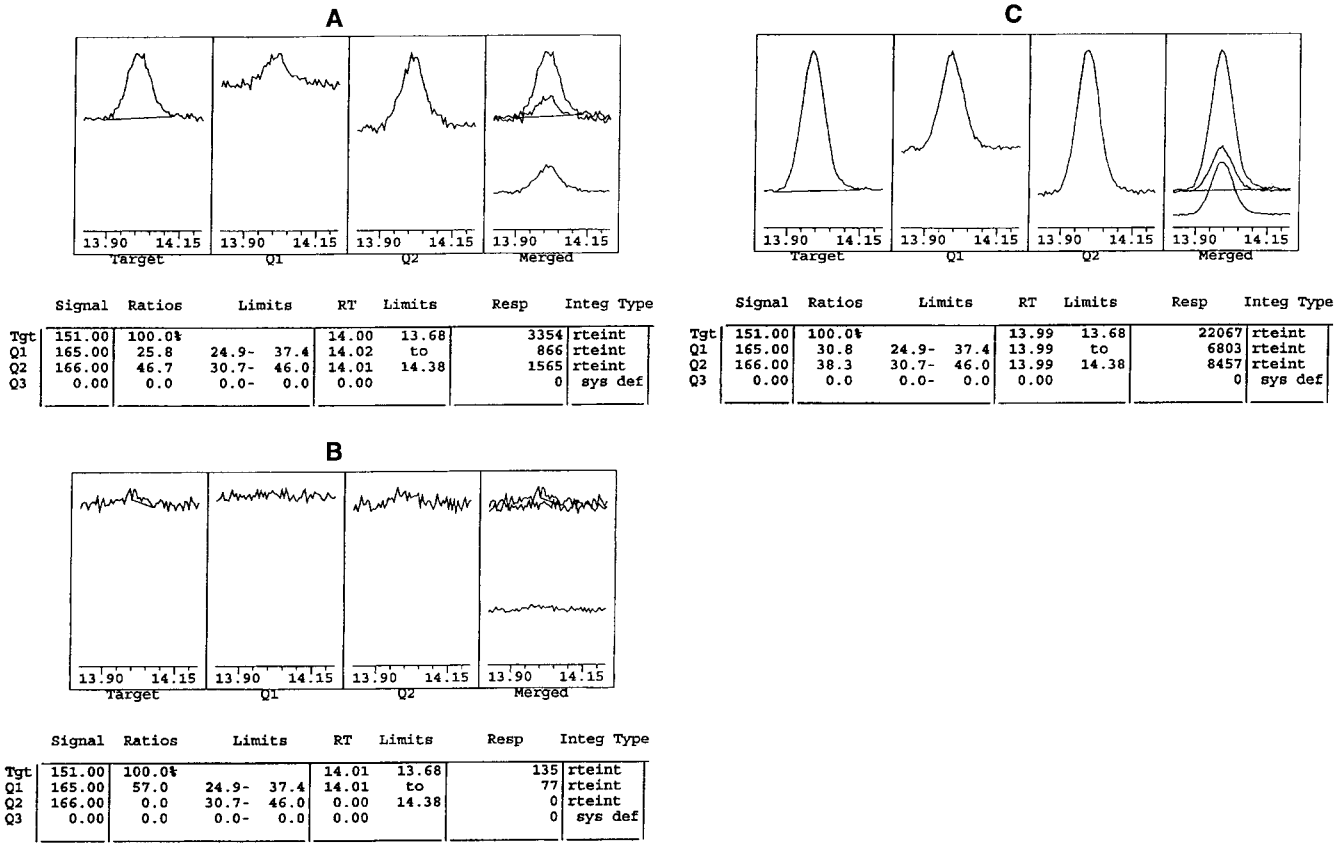

Figure 3. Representative SIM chromatograms for cyromazine in Santa Cruz County soil: (A) 0.05 ng injected concentration level; (B) control; (C) 10 ppb procedural recovery sample.

Table 4. Summary of Procedural Recovery Data Obtained Using LC-UV on Fortified Control Soil Samples Collected in Madera and Santa Cruz Counties, California

		% recovery[a] (standard deviation)	
soil	*n*	cyromazine	melamine
Madera 1	6	90.3 (12.6)	93.5 (9.4)
Madera 2	6	91.2 (9.9)	91.8 (10.4)
Santa Cruz 1	6	91.8 (12.2)	96.3 (17.5)
Santa Cruz 2	6	116 (14.4)	96.8 (7.9)
range[b]		73−142	65−118
all	24	97.3 (15.9)	94.5 (11.4)

[a] All recovery samples were corrected for control residues, if present. [b] Range for both soil types.

in Figure 2B. No background residues of cyromazine were found in any of the soil samples collected from the two study sites.

The procedural recovery data obtained during the method validation on soil obtained from both study sites are listed in Table 4. The mean recovery and standard deviation for cyromazine and melamine were 97.3 (n = 24) and 94.5% (n = 24) and 15.9 and 11.4%, respectively.

The average procedural recoveries for cyromazine obtained during the analysis of soil samples from the two terrestrial field dissipation study sites were 93.9% (n = 149) and 95.0% (n = 132) with standard deviations of 10.2 and 14.3%, respectively, for the Madera and Santa Cruz County soil samples. The average procedural recoveries for melamine obtained during these analyses were 97.5% (n = 149) and 91.6% (n = 132) with standard deviations of 12.2 and 32.3%, respectively, for the Madera and Santa Cruz County soil samples. The standard deviation was larger for the Santa Cruz County soil samples due to the high background levels of melamine in the soil (Speth and Rezaaiyan, 1999; Speth and Yokley, 1999).

The LC-UV portion of the method was subjected to an Independent Laboratory Validation (ILV) for ruggedness testing (U.S. EPA, 1989b), and all of the recovery values were in the 93−120% range. The average recoveries were 107 and 98% with standard deviations of 6.3 and 6.0% for cyromazine (n = 10) and melamine (n = 10), respectively, at the 10 and 100 ppb fortification levels (Wedekind and Rezaaiyan, 1999). This method ruggedness testing was performed using Madera County soil.

GC-MSD Analyses. Representative SIM chromatograms of a 0.050 ng injected standard, a control, and 10 ppb procedural recovery samples for cyromazine and melamine in Madera County soil are shown in Figures 3 and 4, respectively. The nanograms injected and respective responses for the target ions for each analyte were used for construction of the calibration plots. The calibration plots were linear with correlation coefficients typically >0.995. The limits of analyte confirmation were established by calculating ±20% of the qualifier ion 1 (Q1)/target ion ratio as measured for the highest concentration of calibrating standard injected. On rare occasions, the Q1/target ion ratio for the lowest injected concentration of standard would be slightly outside this arbitrarily established limit as shown in Figures 3A and 4A. However, consistent analyte confirmation was obtained for all other standards and recovery samples at and above the LOQ of the method (Figures 3C and 4C). Note that the control sample contains melamine. All of the Madera County soil samples had melamine background levels between 2 and 4 ppb.

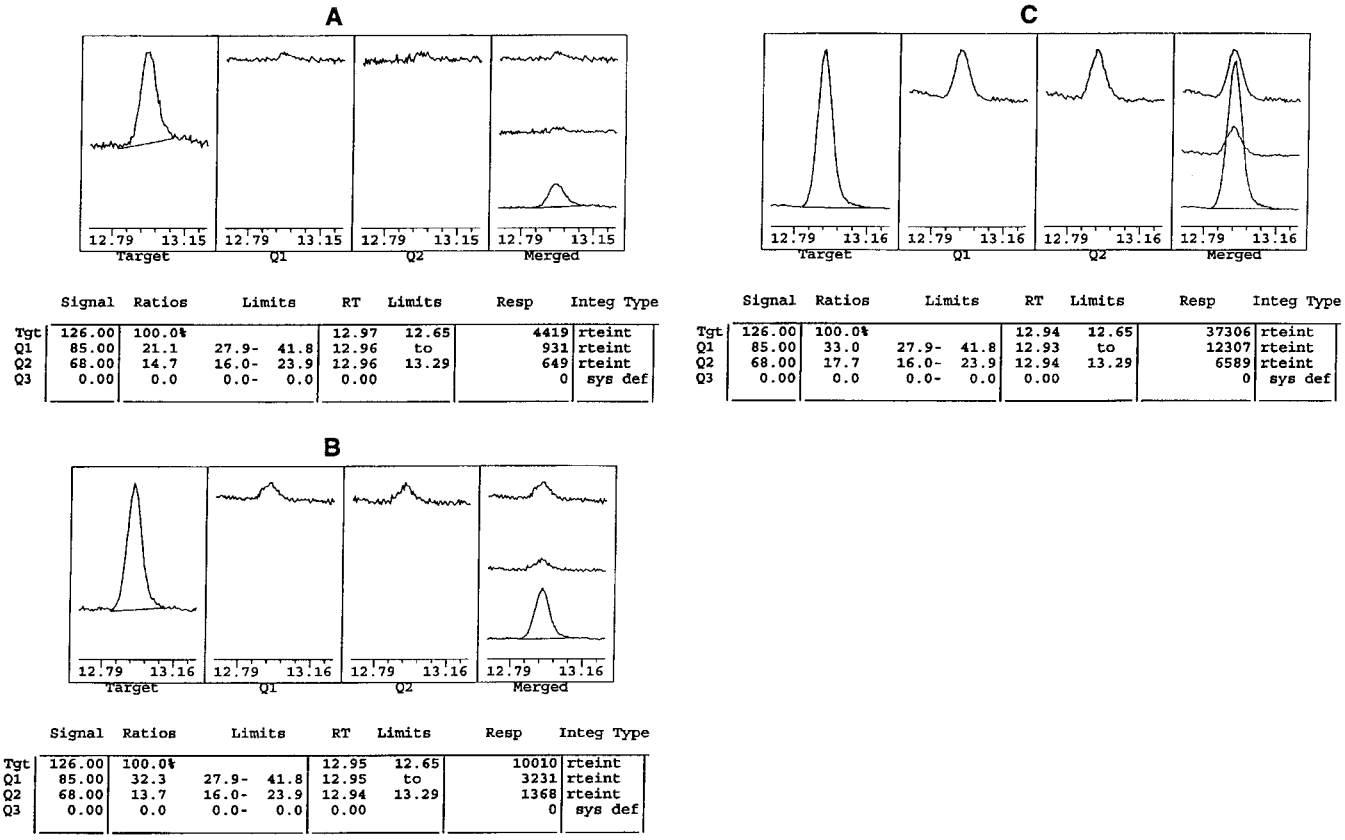

Figure 4. Representative SIM chromatograms for melamine in Santa Cruz County soil: (A) 0.05 ng injected concentration level; (B) control; (C) 10 ppb procedural recovery sample.

Table 5. Summary of Procedural Recovery Data Obtained Using GC-MSD on Fortified Control Soil Samples Collected in Madera and Santa Cruz Counties, California

		% recovery[a] (standard deviation)	
soil	*n*	cyromazine	melamine
Madera 1	5	121 (3.4)	101 (9.2)
Madera 2	6	111 (8.6)	95 (4.5)
Santa Cruz 1	6	102 (4.8)	79 (6.6)
Santa Cruz 2	6	107 (4.4)	105 (22)
Santa Cruz 3	6	96 (2.0)	78 (12)
range[b]		93−127	59−135
all	29	107 (9.9)	92.0 (16)

[a] All recovery samples were corrected for control residues, if present. [b] Range for both soil types.

A summary of the procedural recovery data obtained during the GC-MSD method validation is shown in Table 5. The standard deviation and range of recovery values are greater for the melamine recoveries in Santa Cruz soil due to background levels of melamine in the soil at concentration levels between 20 and 70 ppb. The range of background levels is likely due to the difficulties associated with obtaining completely homogenized soil samples for analysis. Small differences in melamine concentration between the soil sample used for the control and the control sample fortified with 10 ppb of melamine for procedural recovery purposes can adversely affect the recovery values. For example, if the control contains 40 ppb of melamine and another portion of the same control sample selected for procedural recovery purposes contains 50 ppb of melamine, a laboratory fortification of 10 ppb could easily result in a recovery of 200%. The fact that the recoveries range from only 59 to 135% for melamine in the Santa Cruz County soil indicates the great amount of care and effort displayed by the sample preparation laboratory in producing soil samples as homogeneous as possible. The analysis for cyromazine was much less complicated because of the complete absence of detectable levels of cyromazine in the control samples. As a result, the standard deviation was <10% and the range of recoveries was 93−127% (Table 5). The vast majority of the obtained recoveries for both analytes were in the EPA acceptable range of 70−120%.

This method validation was conducted on 0−6-in. layer soil samples from both study sites. However, field dissipation studies require the analysis of soil samples, in 6-in. intervals, to a potential depth of 48 in. depending on the mobility of the compounds in the study. During the analysis of soil samples of deeper depths during field dissipation studies conducted by Novartis Crop Protection, Inc., it was discovered that the GC-MSD method was not as rugged as the LC-UV method. The final fractions for instrumental analysis from soil samples collected at deeper depths contained a new coextractive that behaved as an adhesive in the GC injection syringes. Sometimes after an overnight set of injections, the plunger was found to be held too tightly in the barrel of the syringe even after using numerous syringe solvent rinses between injections. This did not hinder the collection of reliable data, but it did significantly increase the day-to-day GC-MSD maintenance and syringe replacement frequency. Thus, analysis using GC-MSD is recommended primarily for confirmatory purposes when needed on a smaller number of samples.

Conclusions. The results presented in this paper demonstrate that the method presented herein and validated according to FIFRA GLP 40 CFR Part 160 standards is valid, accurate, and precise for the determination of cyromazine and melamine in soil. The LC-UV portion of the method passed an Independent Laboratory Validation as per U.S. EPA FIFRA Subdivision N. The method LOQ is 10 ppb (defined as the lowest recovery value evaluated during the study) and the LOD is 2.5 ng injected (defined as the lowest concentration standard injected and used to construct the calibration plot) when using LC-UV for the final determinative measurement. The LOD is 0.050 ng and the LOQ is 10 ppb when using GC-MSD. The use of LC-UV is recommended for the analysis of large numbers of soil samples requiring method ruggedness, and GC-MSD is recommended when mass spectral confirmatory evidence is required.

In the method validation study, melamine was detected in all of the soil samples analyzed (0−6-in. layer) at concentrations ranging from 2 to ∼80 ppb. Melamine was also detected in all of the soil samples collected at the two field dissipation study sites prior to application of cyromazine to create the treated plots. Thus, it appears that melamine has a source other than cyromazine at these two study site locations in California.

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