

Cover Sheet for

Environmental Chemistry Method

Pesticide Name: Triconazole

MRID#: 448021-30

Matrix: Soil

Analysis: LC/MS

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ANALYTICAL METHOD MS 90.01 REVISION 3

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METHOD TITLE:

Method of Analysis for the Determination of RPA 400727
(Triticonazole) and its Metabolites RPA 406203 and RPA
406341 in Soil and Turf by LC/MS (Revision 3)

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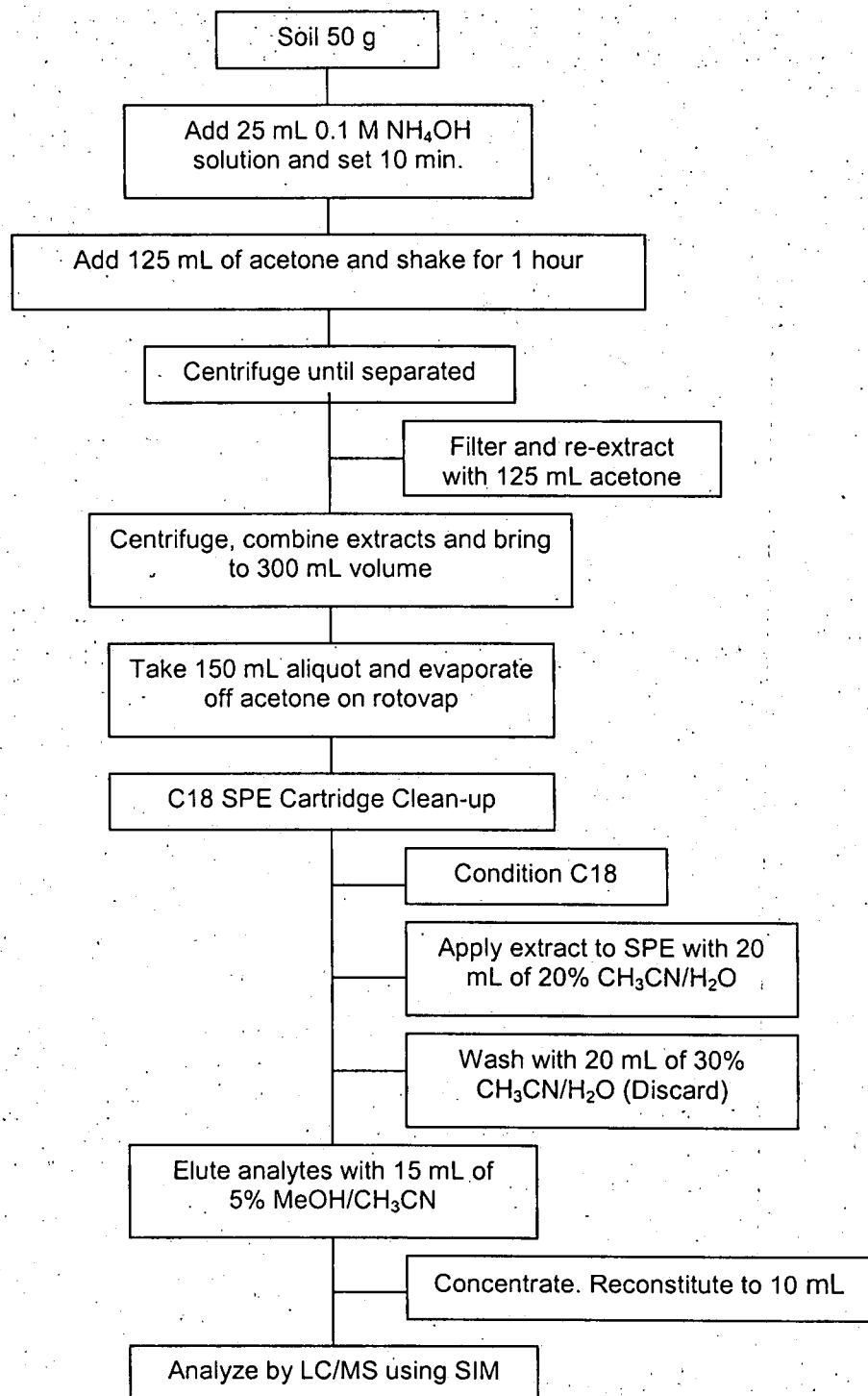
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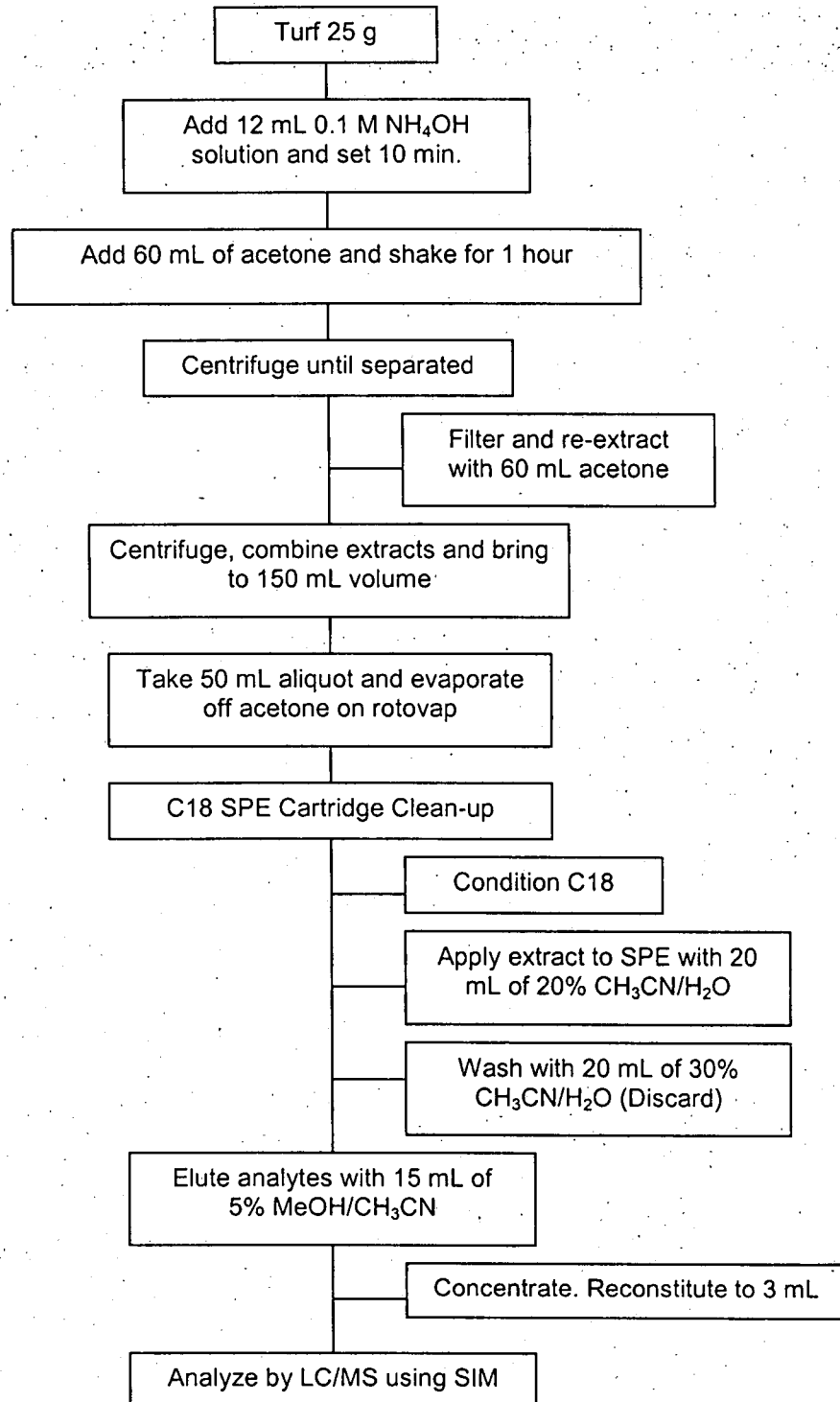
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SUMMARY FLOWCHART OF ANALYTICAL METHOD (SOIL)



SUMMARY FLOWCHART OF ANALYTICAL METHOD (TURF)



1.0 INTRODUCTION

1.1 Scope

This method sets forth the procedure for determining the residues of RPA 400727 (Triticonazole) and its metabolites RPA 406203 and RPA 406341 in soil and turf. The method is based on Rhône Poulenc method no. AR91-82 entitled "RPA 400727 Methode de Dosage Des Residus Dans Le Sol" by M. Guillet/B. Simonin. The method was modified at Enviro-Test Labs to utilize LC/MS rather than GC/ECD.

1.2 Principle

An analytical method is described for the determination of residues of RPA 400727 (Triticonazole) and its metabolites RPA 406203 and RPA 406341 in soil and turf. The inclusion of metabolite RPA 407922 in the method is for demonstrating HPLC resolution of this metabolite from RPA 406341 only. Residues of RPA 400727, RPA 406203 and RPA 406341 are extracted from soil/turf using sonication and shaking with acetone/water. All residue analysis is accomplished by LC/MS (thermospray and/or ionspray) on a C₁₈ column. Quantitation of results is based on a comparison of peak areas with those of known standards. The method has been validated at 5 ppb, 25 ppb, and 50 ppb for RPA 400727, RPA 406203 and RPA 406341 by preparing and analyzing control and fortified soils and turf from California, North Carolina and Washington. The method has also been verified on Canadian soils at levels from 5 - 100 ppb for RPA 400727 and metabolite RPA 406341.

1.3 Method Limits

The method detection level (MDL) and limit of quantitation (LOQ) for RPA 400727, RPA 406203 and RPA 406341 in soil and turf were determined during validation of the method (ETL Report 98RP26.REP). The results are shown in Section 11.0, Table 1 for soil and Table 2 for turf.

The target LOQ of 0.005 ppm for all analytes in soil and turf were close to the calculated LOQ values and consequently 0.005 ppm was established as the LOQ fortification level.

Recovery data at the LOQ for 3 soil types are shown in Section 11.0, Table 3. Recovery data at 5 and 10 times LOQ for soils are shown in Table 4. Recovery data at the LOQ for turf types are shown in Table 5. Recovery data at 5 and 10 times LOQ for turf are shown in Table 6. Precision is < 20% RSD for all analytes at all fortification levels.

2.0 MATERIALS

2.1 Reagents/Solvents

(Equivalent or better grade reagents/solvents may be substituted.)

Acetone - glass distilled, EM Science, OmniSolv®
Acetonitrile (CH₃CN) - glass distilled, EM Science, OmniSolv®
Ammonium acetate - ACS grade, Fisher
Glacial Acetic acid - ACS grade, Fisher
Methanol - glass distilled, EM Science, OmniSolv®
Sodium sulfate - purified by heating to 400°C
Water, deionized - Millipore Purification System

2.2 Equipment and Supplies

(Equivalent equipment may be substituted.)

Balance - Sartorius 1206 MP, VWR Scientific
Bottles, centrifuge - polypropylene, 250 mL, Baxter
Cartridge, C18 SPE - 2 g, Supelco Cat.No. 5-7117
Centrifuge - Sorvall®, RC2-B with 250 mL rotor head, DuPont Instruments
Centrifuge -HN-S with 8 position head, International Equipment Co. (for 40 mL tubes)
Column, HPLC - Symmetry®, C18 5 µm 4.6 × 250 mm, Waters, Cat.No. WAT05475
Culture tubes - 15 mL, screw-top tube, 16 × 25 mm, Kimble Glass Inc.
Cylinders, graduated - 100, 250 and 500 mL
Flasks, round bottom, 50 and 500 mL - Kimble Glass Inc.
Flasks, volumetric - 25 and 100 mL, Class A
Funnels, wide-mouth - polypropylene
Nitrogen evaporator with water bath - Organomation Assoc. Inc., Model No.111
Pipettes, volumetric - 10 mL
Rotoevaporator with water bath - RE51, Yamato
Rubber bulb - Fisher (to apply pressure to SPE cartridge)
Shaker, platform or wrist - Pychotherm
TurboVap tubes, 200 mL
TurboVap® nitrogen evaporator, Zymark
Ultrasonicator - Fisher Scientific, FS-28
Vacuum manifold system for SPE - Visiprep DL, Cat.No. 5-7044, Supelco

2.3 Solutions

The following is a list of solutions used in the analyses of soil and turf.

- 2.3.1 NH_4OH Solution (0.1M): Add 10 mL of concentrated NH_4OH to 4 L of deionized water. Mix by shaking. (Used to pre-soak soil before acetone extraction.)
- 2.3.2 Ammonium Acetate Solution (0.5 M): Add 155 g of ammonium acetate to 4 L of deionized water. Mix by shaking. De-gas by placing 4 L bottle in sonic bath and applying vacuum for ~ 5 minutes.
- 2.3.3 2% Acetic acid in HPLC H_2O : Add 80 mL of glacial acetic acid to 4 L of deionized water and mix by shaking. De-gas by placing 4 L bottle in sonic bath and apply vacuum for at least 5 minutes.
- 2.3.4 2% Acetic acid in acetonitrile: Add 80 mL of glacial acetic acid to 4 L of acetonitrile and mix by shaking. De-gas as above.
- 2.3.5 30% ACN in water (v/v): Add 300 mL of acetonitrile to every 700 mL of deionized water. Mix.
- 2.3.6 5% MeOH in ACN: Add 50 mL of methanol to every 950 mL of acetonitrile. Mix.
- 2.3.7 0.2% Acetic acid in HPLC H_2O : Add 8.0 mL of glacial acetic acid to 4 L of deionized water and mix by shaking. De-gas by placing 4 L bottle in sonic bath and apply vacuum for at least 5 minutes.
- 2.3.8 0.2% Acetic acid in acetonitrile: Add 8.0 mL of glacial acetic acid to 4 L of acetonitrile and mix by shaking. De-gas as above.

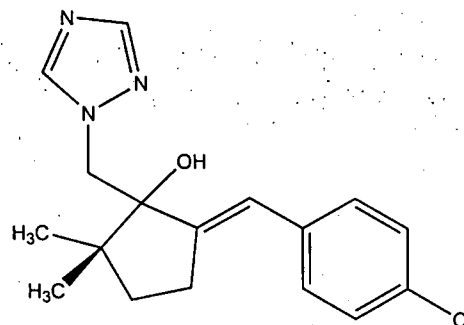
2.4 Analytical Standards and Chemical Structures:

RPA 400727

Chemical Name:

(1RS)-E-2-(4-chlorobenzylidene)-
5,5-dimethyl-1-(1H-1,2,4-triazol-
-1-ylmethyl)cyclopentan-1-ol

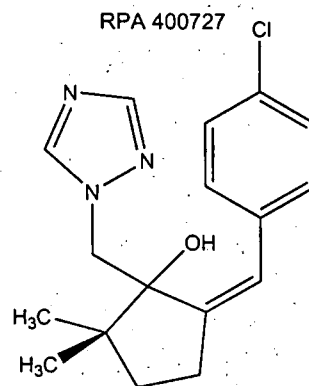
CAS No.: 131983-72-7



RPA 400727

RPA 406203

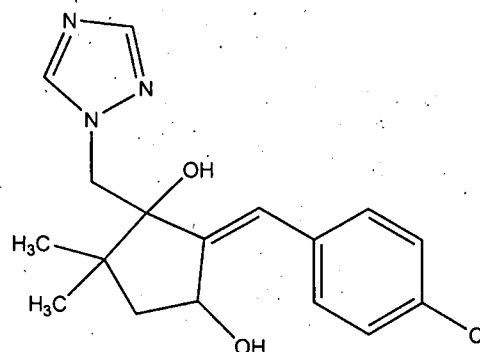
Chemical Name:

(1RS)-(Z)-5-(4-Chlorobenzylidene)-
2,2-dimethyl-1-(1,2,4-triazol-1-
-ylmethyl)cyclopentan-1-ol

RPA 406203

RPA 406341

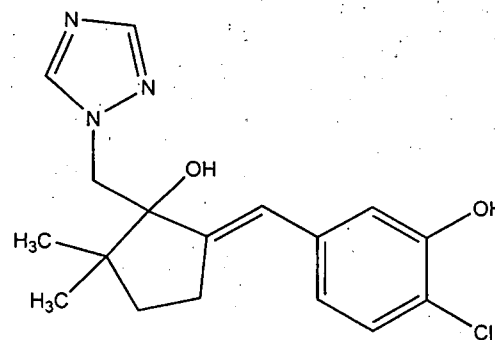
Chemical Name:

E-2-(4-chlorobenzylidene)-5,
5-dimethyl-1-(1H-1,2,4-triazol-
1-ylmethyl)-trans-cyclopentan-
1,3-diol

RPA 406341

RPA 407922*

Chemical Name:

(1RS)-(E)-5-(4-chloro-3-
-hydroxybenzylidene)-2,2-
dimethyl-1-(1,2,4-triazol-
1-ylmethyl)cyclopentan-1-ol

RPA 407922

*Used in the resolution mix standard only.

3.0 FORTIFICATION AND CALIBRATION STANDARD SOLUTIONS

3.1 Preparation

All the standard solutions must be stored in glass at or below 10°C when not in use. Solutions should be allowed to warm to room temperature prior to use. The following is an example procedure for preparing a standard solution. Alternate or additional standards of appropriate weight and volume may be prepared as needed. The "~" symbol indicates approximately.

- 3.1.1 Accurately weigh ~ 0.025 g (corrected for purity) each of RPA 400727, RPA 406203 and RPA 406341 into separate 25 mL volumetric flasks and dilute to the mark with acetonitrile. Cap and mix by inversion. The concentration of these stock standards is ~1000 µg/mL.
- 3.1.2 For the preparation of fortification standards of RPA 400727, RPA 406203 and RPA 406341, transfer 10 mL of each ~1000 µg/mL standard via volumetric class "A" pipettes, to a 100 mL volumetric flask. Dilute to mark with acetonitrile. Cap and mix by inversion. The concentration of this mixed standard is ~100 µg/mL RPA 400727, RPA 406203 and RPA 406341.
- 3.1.3 Calibration standards for soil samples are prepared by serial dilution of the 100 µg/mL fortification standards in 20% acetonitrile/water.

Calibration standards for turf samples are prepared by serial dilution of the 100 µg/mL fortification standards in 100% acetonitrile. The peak shape of RPA 406341 is broadened for the turf samples but acetonitrile is needed in the final turf extracts to prevent precipitation of co-extractives in solution.

A standard of RPA 407922 is also prepared as in 3.1.1 and 3.1.2. A "resolution" mix standard of all 4 analytes at 10 µg/mL to 100 µg/mL is prepared and diluted to 10 and 0.100 µg/mL in 20% acetonitrile/water. It is used only for demonstrating optimization of resolution of the HPLC system.

3.2 Stability

3.2.1 To evaluate the stability, the following formula has been used:

$$\% \text{ Stability} = 1 - \left(\frac{\text{old standard solution}}{\text{new standard solution}} \right) \times 100$$

The old standard solution should give detector responses within 10% of those of the new standard solution in order for the given standard solution to be considered stable under the storage conditions.

3.2.2 Stock solutions: Each product prepared in acetonitrile and stored at $4 \pm 3^\circ\text{C}$ was stable for up to 18 months (ref. 99RP41A.REP).

4.0 METHOD PROCEDURES

4.1 General Notes

- 4.1.1 The "♦" symbol indicates an optional stopping point after completing the indicated step. Samples may be stored overnight in a refrigerator (at or below 10°C).
- 4.1.2 The "~" symbol indicates approximately.
- 4.1.3 The elution profile of RPA 400727, RPA 406203 and RPA 406341 should be checked on each new lot of C_{18} SPE cartridges. This should be done using analytes in solvent and in control matrix. A recovery of over 85% must be achieved on the SPE cartridges before proceeding. If low recoveries are observed additional fractions of elution solvent and the washes should be collected and analyzed to determine if the analytes eluted in the wash or have remained on the column.
- 4.1.4 The analytical C_{18} column must resolve the two metabolites RPA 407922 and RPA 406341 well enough to identify and quantitate RPA 406341. The gradient program may need to be modified in order to obtain this resolution. The cis/trans isomers of Triticinazole (RPA 400727 and RPA 406203) must also be resolved.

Alternate analytical columns may be substituted, provided they meet these criteria.

- 4.1.5 The concentration of the acetic acid in the mobile phases may be reduced to 0.2% acetic in water and 0.2% acetic in acetonitrile for the API 150 EX LC/MS, provided the LC/MS system performance is optimized.
- 4.1.6 It has also been found useful to split the post column flow about 5:1 (waste/source) so that about 200 μ L/min. enters the turbo ionspray interface on the Sciex API 150 EX LC/MS. This has been found to increase sensitivity of all the analytes.

4.2 Soil Analysis of RPA 400727, RPA 406203 and RPA 406341

- ◆ 4.2.1 Weigh 50 g of a prepared¹ subsample of soil into a 250 mL polypropylene centrifuge bottle. Untreated control samples may be fortified at this point for determination of recovery.
- 4.2.2 Add 25 ± 5 mL of 0.1 M NH_4OH solution to the soil, shake to mix and let sit for ~ 10 min.
- 4.2.3 Add 125 ± 5 mL of acetone to the sample and shake on wrist-action or platform shaker for ~ 1 hour.
- ◆ 4.2.4 Remove, shake by hand and place in a sonic bath for ~ 10 minutes.
- 4.2.5 Centrifuge the sample for at least 5 minutes, or until separated and decant the supernatant through a cotton ball placed in a wide-mouth funnel. Collect in a 500 mL graduated cylinder.
- 4.2.6 Add 125 ± 5 mL of acetone to the soil pellet in the centrifuge bottle and shake vigorously by hand.
- 4.2.7 Centrifuge as in 4.2.5 and combine supernatant through the sample funnel in the 500 mL graduated cylinder.
- 4.2.8 Adjust the volume in the cylinder to 300 mL with acetone and mix by pouring into a 500 mL boiling flask and back into the cylinder.
- ◆ 4.2.9 Transfer a 150 ± 2 mL aliquot (representing 25 g of soil) to the same 500 mL flask and evaporate the acetone (until only 10-15 mL of aqueous remains) using a rotoevaporator with the water bath set at $40 \pm 2^\circ\text{C}$. Transfer to a 40 mL screw-cap tube. Rinse 500 mL flask 2 times with 2 mL of acetonitrile and add this rinse to the 40 mL screw-cap tube. Bring to 20 mL with deionized water. Cap and centrifuge for a couple of minutes at ~ 1500 RPM.

¹Prepared subsamples are samples which have been mixed and chopped with dry ice using a Hobart food chopper. These samples must be free-flowing and homogenous prior to subsampling.

4.2 Soil Analysis of RPA 400727, RPA 406203 and RPA 406341 cont'd

- 4.2.10 Set up a series of 2 g C18 SPE cartridges in a vacuum manifold and elute 15 mL of ACN followed by 15 mL of 30% CH₃CN/water through each cartridge. Use a rubber bulb to force the solvent through the cartridge stopping the solvent at the top of the bed (positive pressure). The conditioning rinses are discarded.
- 4.2.11 Transfer the 20 mL of 20% CH₃CN/aqueous sample extracts to the SPE cartridges leaving any solid residues in the tube. Bring to the top of the column using a combination of vacuum and positive pressure at a low rate of 1-2 drops/second and discard. Do not let the cartridge go dry! Wash cartridge with 20 mL of 30% CH₃CN/H₂O. Dry cartridge using vacuum (>5 mm Hg) for at least 3 minutes.
- 4.2.12 After a 15 mL culture tube is placed under each cartridge, elute analytes with 15 mL of 5% MeOH/acetonitrile using positive pressure.
- 4.2.13 The sample is concentrated on a N-Evap at 35-40°C to less than 1 mL and restored to 2 mL with acetonitrile. 8 mL of HPLC grade water is added to make a 10.0 mL final volume.
- 4.2.14 Transfer an aliquot to HPLC vials and analyze by LC/MS using selective ion monitoring (see section 5.0). These extracts can be stored at -20°C until analyzed.

4.3 Turf Analysis of RPA 400727, RPA 406203 and RPA 406341

- ◆ 4.3.1 Weigh 25 g of a prepared¹ subsample of turf into a 250 mL polypropylene centrifuge bottle. Untreated control samples may be fortified at this point for determination of recovery.
- 4.3.2 Add 12 ± 2 mL of 0.1 M NH₄OH solution to the turf, shake to mix and let sit for ~10 min.
- 4.3.3 Add 60 ± 5 mL of acetone to the sample and shake on wrist-action or platform shaker for ~1 hour.
- ◆ 4.3.4 Remove, shake by hand and place in a sonic bath for ~10 minutes.
- 4.3.5 Centrifuge the sample for at least 5 minutes, or until separated and decant the supernatant through a cotton ball placed in a wide-mouth funnel. Collect in a 250 mL graduated cylinder.
- 4.3.6 Add 60 ± 5 mL of acetone to the pellet in the centrifuge bottle and shake vigorously by hand.
- 4.3.7 Centrifuge as in 4.3.5 and combine supernatant through the sample funnel in the 250 mL graduated cylinder.
- 4.3.8 Adjust the volume in the cylinder to 150 ± 2 mL with acetone and mix by pouring into a 250 mL or 500 mL flask and back into the cylinder.

¹Prepared subsamples are samples which have been mixed and chopped with dry ice using a Hobart food chopper. These samples must be free-flowing and homogenous prior to subsampling.

4.3 Turf Analysis of RPA 400727, RPA 406203 and RPA 406341 cont'd

- ◆ 4.3.9 Transfer a 50 ± 1 mL aliquot (representing 8.3 g of turf) to a 250 or 500 mL flask and evaporate the acetone using a rotoevaporator with the water bath set at $40 \pm 2^\circ\text{C}$. Transfer to a 40 mL screw-cap tube. Rinse flask 2 times with 2 mL of acetonitrile and add this rinse to the 40 mL screw-cap tube. Bring to 20 mL with deionized water. Cap and centrifuge for a couple of minutes at ~ 1500 RPM.
- 4.3.10 Set up a series of 2 g C18 SPE cartridges in a vacuum manifold and elute 15 mL of ACN followed by 15 mL of 30% CH_3CN /water through each cartridge. Use a rubber bulb to force the solvent through the cartridge stopping the solvent at the top of the bed (positive pressure). The conditioning rinses are discarded.
- 4.3.11 Transfer the 20 mL of 20% CH_3CN /aqueous sample extracts to the SPE cartridges leaving any solid residues in the tube. Bring to the top of the column using a combination of vacuum and positive pressure. Wash cartridge with 20 mL of 30% $\text{CH}_3\text{CN}/\text{H}_2\text{O}$. Dry cartridge using vacuum (>5 mm Hg) for at least 3 minutes.
- 4.3.12 After a 15 mL culture tube is placed under each cartridge, elute analytes with 15 mL of 5% MeOH/acetonitrile using positive pressure.
- 4.3.13 The sample is concentrated on a N-Evap to less than 1 mL at $35\text{--}40^\circ\text{C}$ and restored to a 3.0 mL final volume with acetonitrile.
- 4.3.14 Transfer an aliquot to HPLC vials and analyze by LC/MS Turbo Ionspray using selective ion monitoring (see section 5.2). These extracts can be stored at -20°C until analyzed.

5.0 HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY (LC/MS)

Note: Equivalent LC/MS instrumentation may also be used and optimized to meet sensitivity requirements. LC/MS systems which utilize "heated capillary" technology may also require a "column switching" program.

5.1 Finnigan Thermospray LC/MS Conditions

Instruments used:

Finnigan (San Jose, CA) SSQ 710 with thermospray TSP-2 interface

Waters (Milford, MA) 600 MS systems controller

Waters 717 refrigerated autosampler

Guard Column: RP-18 Newguard 7μ (15×3.2 mm), Applied Biosystems, Part # 0711-0092

HPLC column: Symmetry® C18, 5 µm, 4.6 × 250 mm
(Equivalent C18 column or guard column may be used, but metabolite resolution of RPA 406431 and RPA 407922 must be demonstrated.)

Gradient Program: (linear gradient changes)

Time (min.)	% A	% B
Initial	65	35
12	65	35
23	35	65
28	35	65
35	65	35

The above gradients may be modified to improve resolution and/or chromatography

Solvent A - (2% acetic acid/water)
Solvent B - (2% acetic acid/acetonitrile)
Flow rate - 1.2 mL/minute

Post column eluant:

Perkin Elmer HPLC/MS pump continuously adds 0.5 M aqueous ammonium acetate at a flow rate of 0.3 mL/minute.
Injection volume - 100 µL

5.2 PE-Sciex API-150EX LC/MS Conditions

Instruments used:

Sciex API-150EX with Turbo Ionspray Source

Varian 9012 Solvent Delivery System

Rainin AI-200 autosampler

Guard Column: RP-18 Newguard 7 µ (15.2 × 3.2 mm), Applied Biosystems, Part # 0711-0092

HPLC column: Symmetry® C18, 5 µm, 4.6 × 250 mm
(Equivalent C18 or guard column may be used but metabolite resolution of RPA 407922 and RPA 406341 must be demonstrated.)

Gradient Program: (linear gradient changes)

Time (min.)	% A	% B
Initial	70	30
1.0	70	30
14	65	35
23	35	65
28	35	65
28.1	70	30
36	70	30

The above gradient may be modified to improve resolution and/or chromatography:

Solvent A - (0.2% acetic acid/water)

Solvent B - (0.2% acetic acid/acetonitrile)

Flow rate - 0.9 mL/minute

Injection volume - 25 μ L

Split post column flow 5:1 (waste/source)

Approximate Retention Times		
	m/z Target	m/z Qualifier
RPA 406341 - 17.9 min.	334.0	336.0
RPA 407922 - 18.3 min.	334.0	336.0
RPA 400727 - 23.9 min.	318.0	320.0
RPA 406203 - 25.6 min.	318.0	320.0

Retention times may vary from those presented above.

Example chromatograms are attached including a resolution mix standard (see Section 9.0 for API 150 EX Instrument Conditions). Note that the retention times may vary from system to system and may require optimization but peak resolution of RPA 406341 and RPA 407922 must be attained. This can normally be achieved by altering the elution gradient.

Scan type: Q1 SIM

Polarity: Positive

Acquisition mode: Profile

Pause time: 2 ms

Masses requested:

Start Mass (amu)	Stop Mass (amu)	Step (amu)	Dwell Time (ms)
318	318	0	100
320	320	0	100
334	334	0	100
336	336	0	100

ST: -16.500

RO1: -11

IQ2: 0

RO2: 0

IQ3: 0

RO3: 0

DF: -200

CEM: 2500

NEB: 15

CUR: 12

QPE: 0

POL: 0

VCM: 0

IPE: 0

Examples:

Calibration Table Q. Calibration	
Mass	DAC value
59	1098
175.133	3308
616.464	11711
906.673	17236
1254.925	23866
1545.134	29391
2010.469	38245
2242.637	42663

Q1 Peak Widths	
Mass	Offset
30	0.050
100	0.085
1000	0.483
2000	0.917

State Table Parameters	
Parm	Value
IS	5900
NC	0
TEM	475
OR	27
RNG	230
QO	-2.500
IQ1	-9

5.3 Performance Criteria (LC/MS)

First Criterion:

Run a standard solution on LC/MS corresponding to a level at or below the estimated LOQ and obtain a signal to noise ratio of at least 9:1.

If this criterion cannot be met, optimize and change instrument operating parameters.

Optimization may include altering post column split flow and/or altering the concentration of the acetic acid in the LC mobile phase.

Second Criterion:

Run a set of standards of four or more concentration levels, from at or below the LOQ, up to the highest concentration level to be included in the analysis. Generate a calibration curve for each analyte and obtain a linear regression with a correlation coefficient of at least 0.98 for each analyte. If this criterion is met, the samples may be run with standards interspersed.

6.0 CALCULATIONS

Linear regression should be used to generate calibration curves for RPA 400727, RPA 406203 and RPA 406341. After the instrument performance criteria are met, a minimum of four standards over a range of concentration levels should be included with a set of samples. Standards should be interspersed with samples to compensate for any minor change in instrument response. Samples should be diluted such that any peak areas or heights are within the area or height range between the lowest and highest standards injected.

Linear regression coefficients should be calculated on standard concentration ($\mu\text{g/mL}$) versus peak area or height. The data from the analytical standards should then be fit to the linear model.

$$y = mx + b$$

6.0 CALCULATIONS cont'd

The equation to be used to estimate the residues in the samples is:

$$\text{Conc. (ppm or ug/mL)} = \frac{(x - b)}{m} \times \frac{\text{F.V.}}{g} \times \text{A.F.}$$

NOTE: If the LC/MS system generates an intercept (b) that is >20% of the area or peak height of the LOQ spikes (0.005 ppm), then use a 1/x curve for quantitation. This will weight the low level standards and result in a curve passing nearer the origin.

Where: x = Response of analyte of interest (peak area or height)
 b = Intercept from linear regression analysis (peak or height)
 m = Slope from linear regression analysis (response per concentration)
 F.V. = Final sample volume (mL)
 g = Starting weight in grams of sample (g)
 A.F. (Soil)=Aliquot factor= $\frac{\text{Extraction Solvent Volume(mL)}}{\text{Aliquot Volume (mL)}} = \frac{300 \text{ mL}}{150 \text{ mL}} = 2.0$
 A.F. (Turf) = $\frac{150 \text{ mL}}{50 \text{ mL}} = 3.0$

7.0 SAFETY

All available appropriate MSDS's should be available to the study personnel during the conduct of the study. General laboratory safety precautions should be taken. This method does not present any specific risks.

8.0 REVISIONS

- 8.1 Updated and expanded PE-Sciex API 150 EX LC/MS conditions.
- 8.2 Updated example chromatograms.
- 8.3 Added LOQ, 5 times, and 10 times LOQ method validation data tables from ETL Report 99RP48.REP.
- 8.4 Added additional note to Section 4.1, General Notes (0.2% acetic mobile phase).
- 8.5 Added Revisions, section 8.0, indicating what changes to Rev.2 were made.

**9.0 EXAMPLE CHROMATOGRAMS:
FINNIGAN THERMOSPRAY LC/MS**

Elapse: 1 0 8.22
Times: 8.22 > 28.01
Masses: 317 > 336
Client: RPA (USA)
RIC: 704924
Peak ID: 2, 40
7.0E+05

CHRO: 1216rg502.dat (16-DEC-97)
Samp: RPA mix 10.0 ppm
Comm: Set#1
Mode: TSP +QIMS LMR UP LR
Oper: RBG
Peak: 1000.0 mmu
Area: 2, 4, 0
RIC (SM 3)

Study: Trit.smetab. verif.
Intensity: 704924
Baseline: 20, 3

Total 10N Chromatogram (TIC)

RPA mix

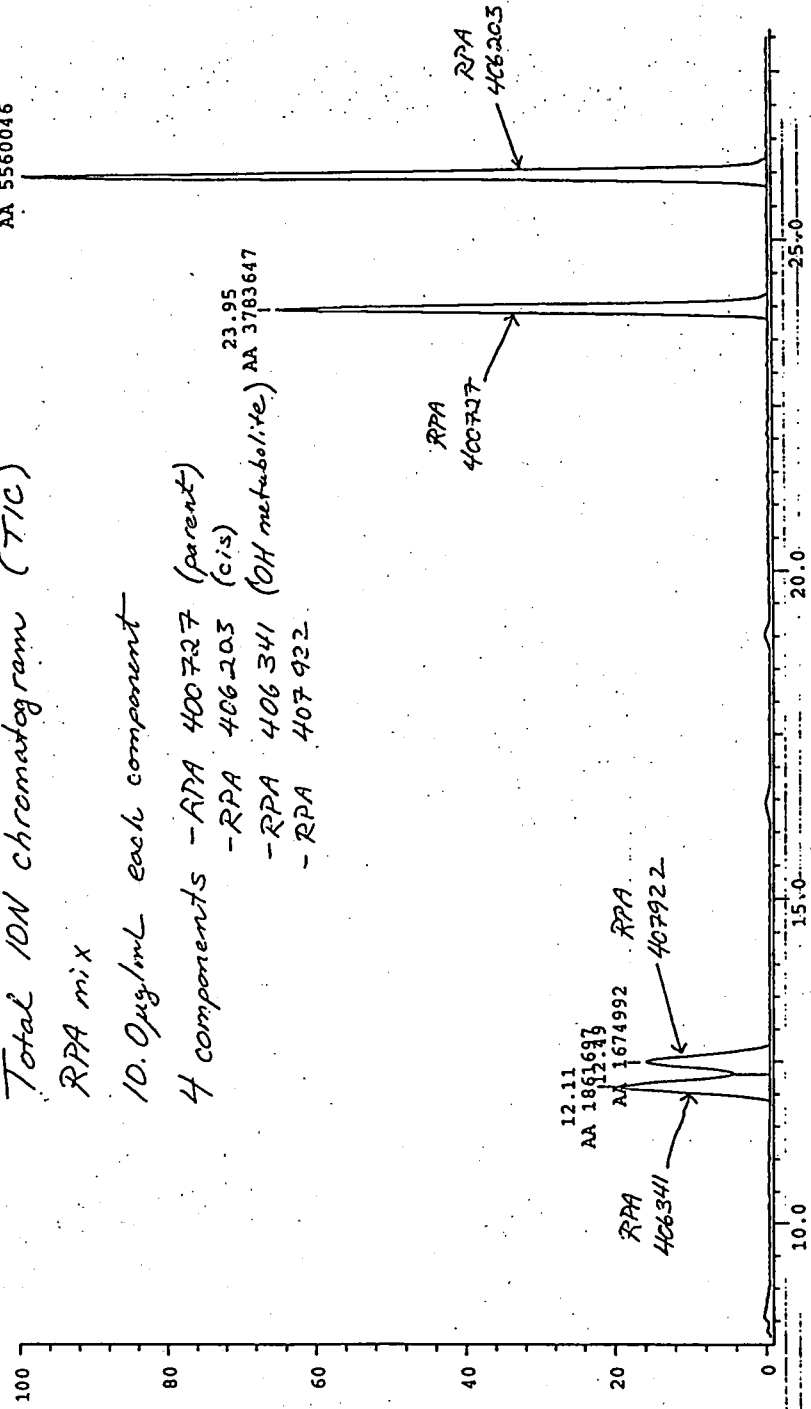
10.0 µg/mL each component

4 components - RPA 400727 (parent)

- RPA 406203 (cis)

- RPA 406341 (OH metabolite)

- RPA 407922



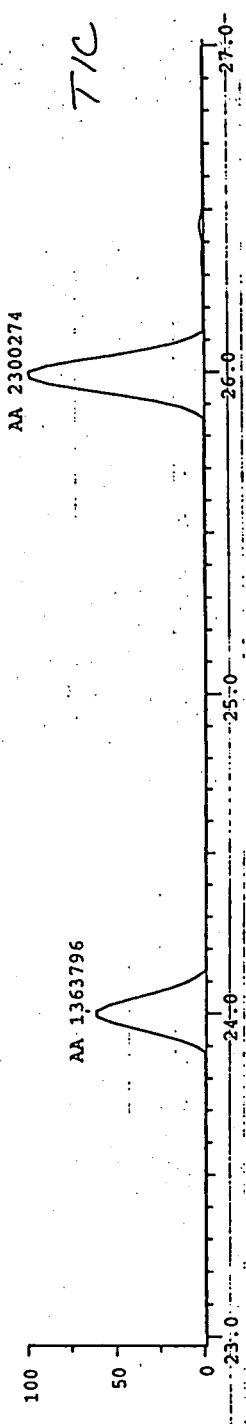
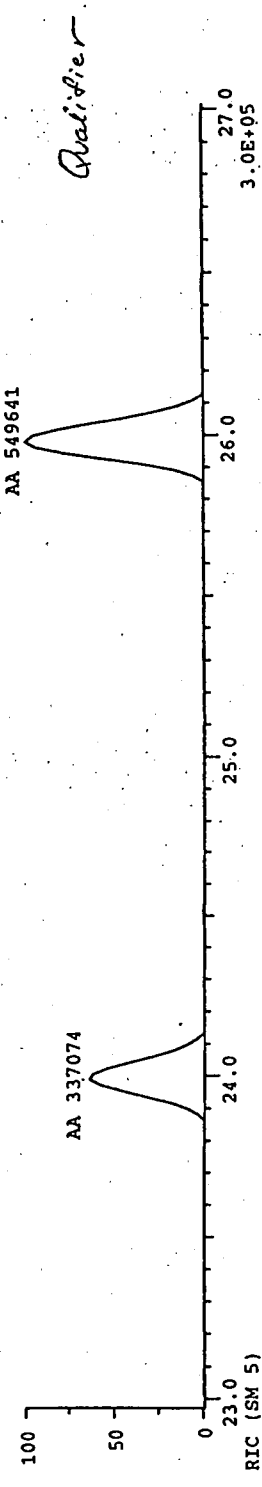
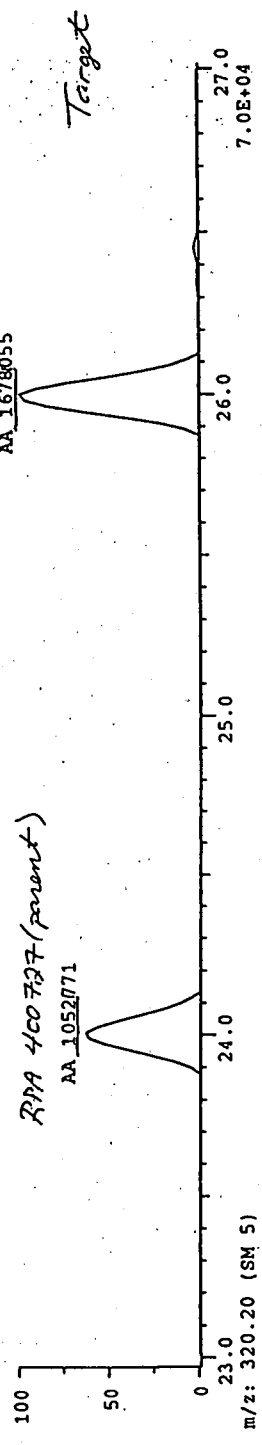
Total Ion Chromatogram (TIC) RPA
Mix 10.0 µg/mL each component

Date: Wed Dec 17 07:39:03 1997 ICIS: 8.3.0 for ULTRIX (4.4)

CHRO: 1216rg501.dat (16-DEC-97)
 Samp: RPA mix 5.00 ppm
 Comm: Set#1
 Mode: TSP +Q1MS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 318.20 (SM 5)

Elapse: 1 @ 8.22
 Times: 8.22 > 28.01
 Masses: 317 > 336
 Client: RPA (USA)
 RIC: 225070
 Peak ID: 12, 5
 2.3E+05

Study: Trit.&metab. verif.
 Intensity: 225070
 Baseline: 10, 10



5.00 µg/mL Standard Mix
 4 components

Date: Thu Dec 18 14:48:54 1997 ICIS: 8.3.0 for ULTRIX (4.4)

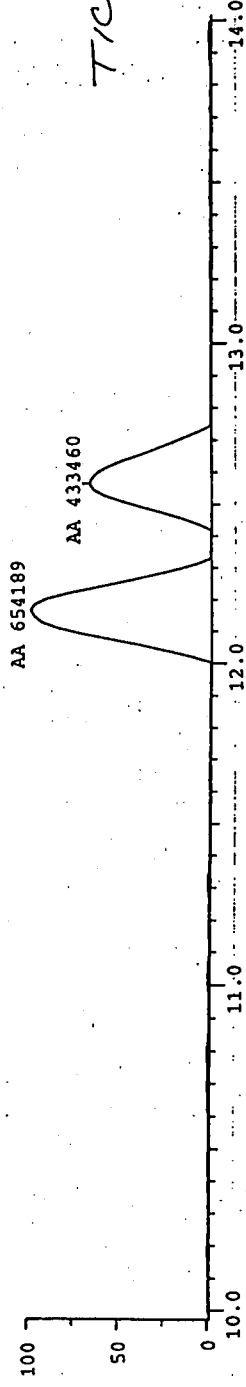
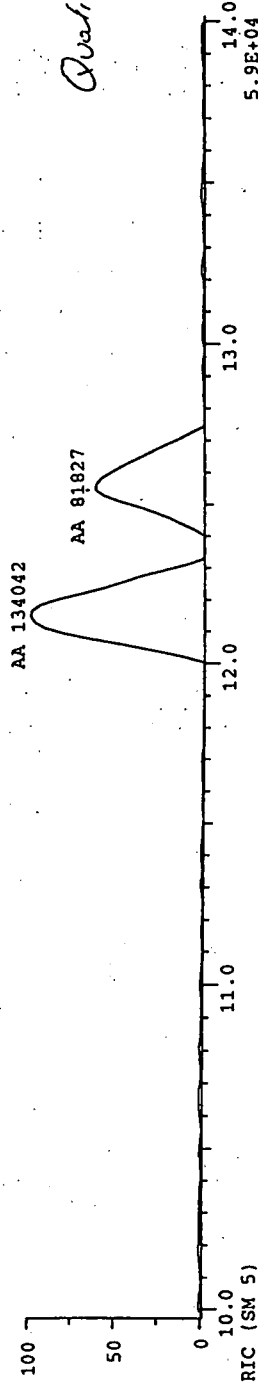
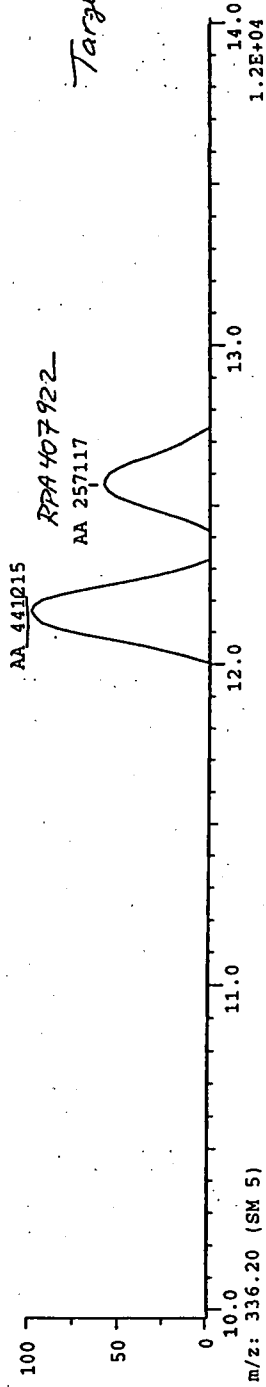
CHRO: 1216rg501.dat (16-DEC-97)
Samp: RPA mix 5.00 ppm
Comm: Set#1
Mode: TSP +Q1MS LMR UP LR
Oper: RBG
Peak: 1000.0 mmu
Area: 2, 2, 0
m/z: 334.20 (SM 5)

Study: Trit.smetab. verif.
Intensity: 39454
Baseline: 10, 10

Elapsed: 1 @ 8.22
Times: 8.22 > 28.01

Masses: 317 > 336
Client: RPA (USA)
RIC: 39454
Peak ID: 12, 5

RPA 406341 (OH metabolite)



5.00 µg/mL Standard Mix
4 components

Date: Thu Dec 18 14:01:07 1997 ICIS: 8.3.0 for ULTRIX (4.4)

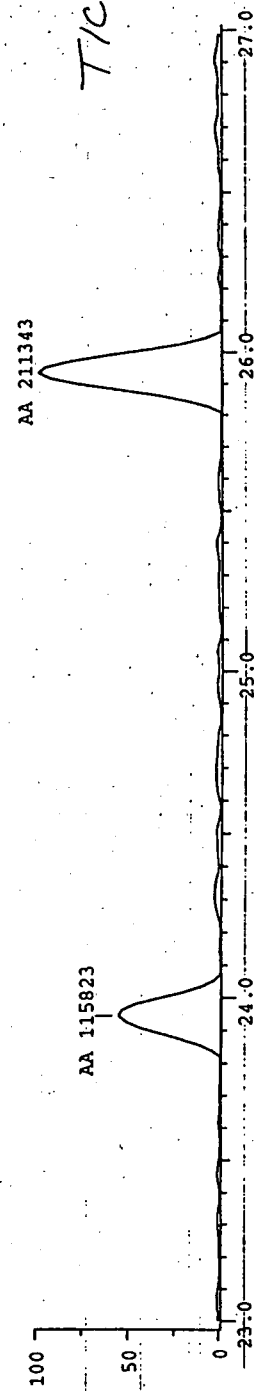
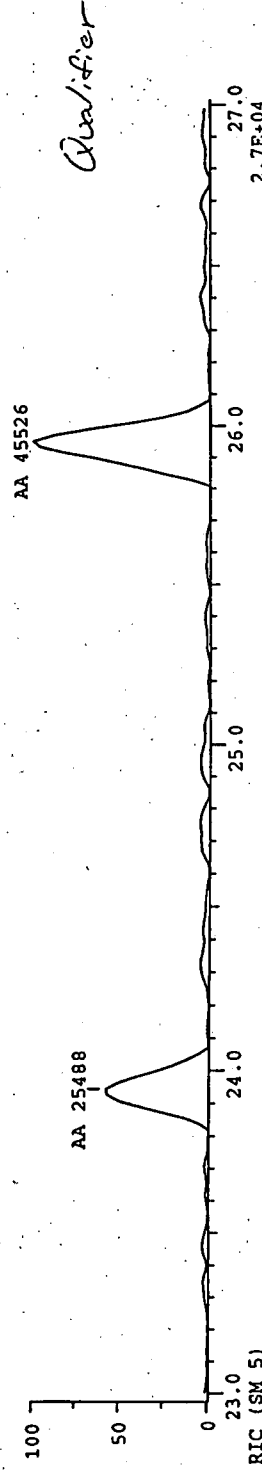
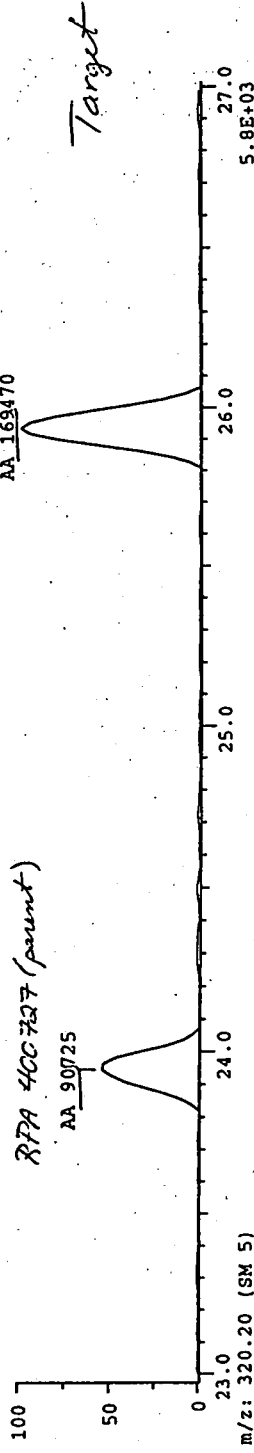
CHRO: 1216rg505.dat (16-DEC-97)
Samp: RPA mix 0.500 ppm
Comm: Set#1
Mode: TSP +Q1MS LMR UP LR
Oper: RBG
Peak: 1000.0 mmu
Area: 2, 2, 0
m/z: 318.20 (SM 5)

Study: Trit.&metab. verif.
Intensity: 22079
Baseline: 10, 10

Elapse: 1 @ 8.25
Times: 8.25 > 28.00

Masses: 317 > 336
Client: RPA (USA)
RIC: 22079
Peak ID: 12, 5
2.2E+04

RPA 406 203 (c.r.)



0.500 µg/mL Standard Mix
4 components

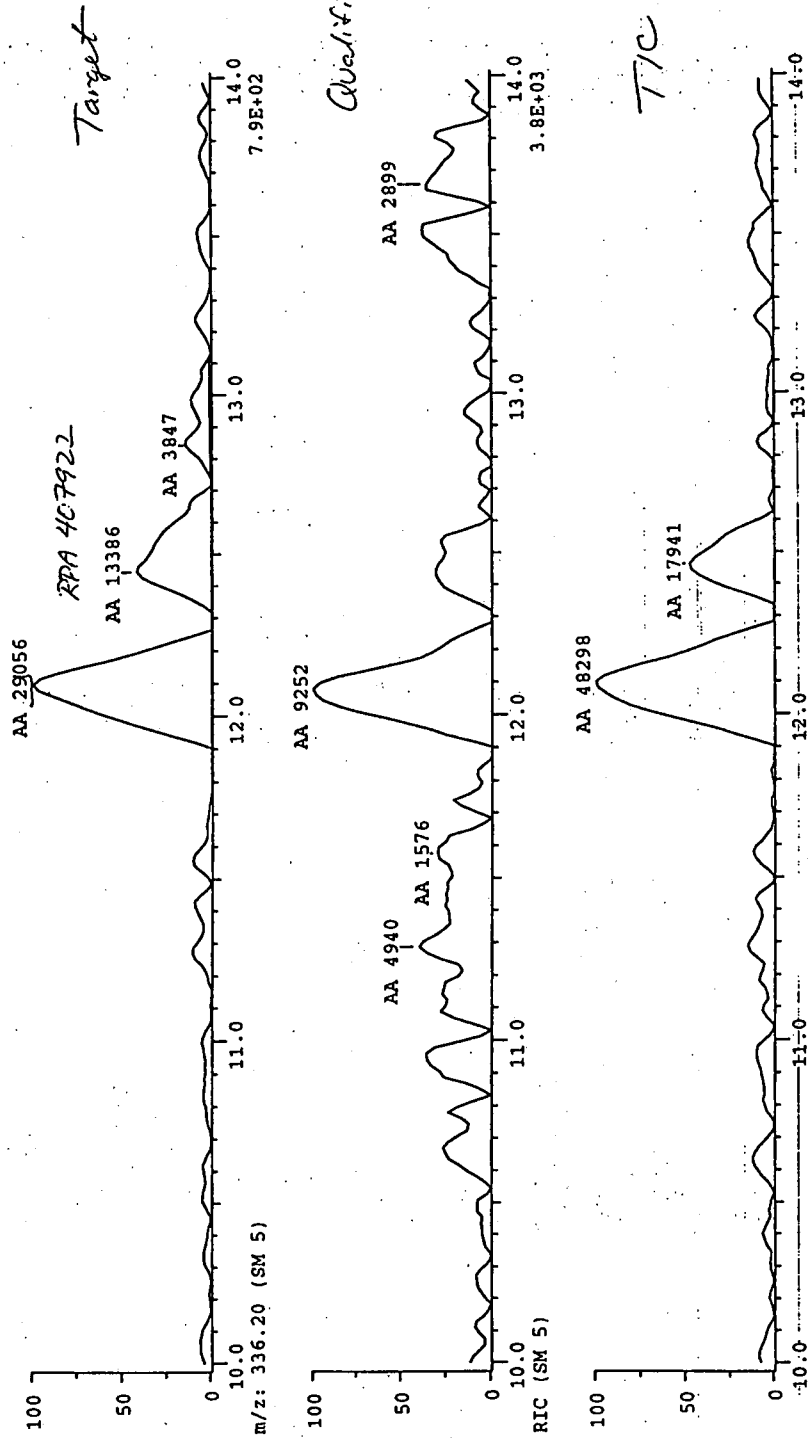
CHRO: 1216rg505.dat (16-DEC-97)
 Samp: RPA mix 0.500 ppm
 Comm: Set#1
 Mode: TSP +Q1MS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 334.20 (SM 5)

Elapse: 1 @ 8.25
 Times: 8.25 > 28.00

Masses: 317 > 336
 Client: RPA (USA)
 RIC: 2473
 Peak ID: 12, 5
 2.5E+03

Study: Trit.Emetab. verif.
 Intensity: 2473
 Baseline: 10, 10

RPA 406.341 (OH metabolite)



0.500 µg/mL Standard Mix
 4 components

Date: Thu Dec 18 14:14:25 1997 ICIS: 8.3.0 for ULTRIX (4.4)

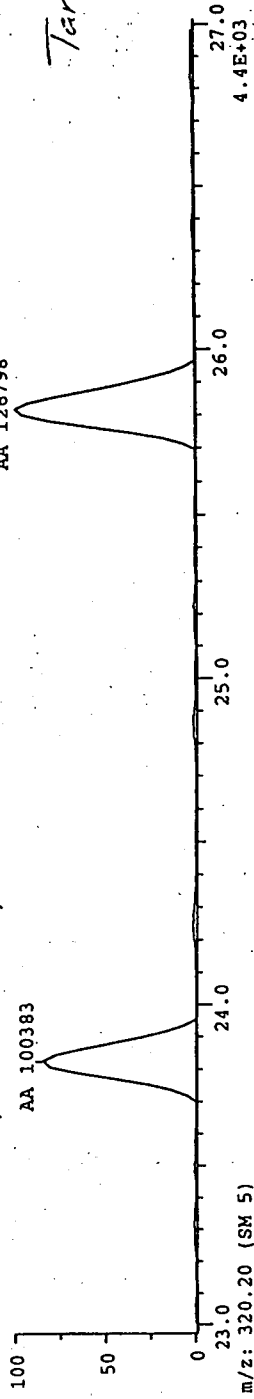
Elapse: 1 @ 8.24
Times: 8.24 > 28.01

Masses: 317 > 336
Client: RPA (USA)
RIC: 16325
Peak ID: 12, 5
1.6E+04

RPA 406203 (c/s)

AA 126798

Target



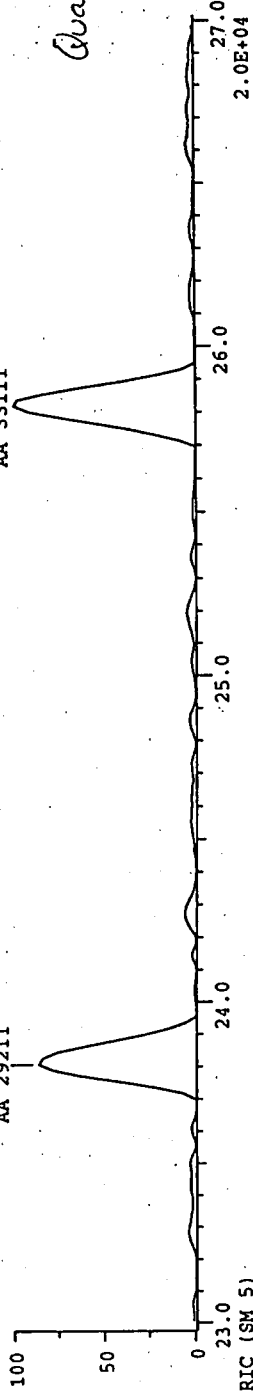
Study: Trit.&metab. verif.
Intensity: 16325
Baseline: 10, 10

RPA 400727 (parent)

AA 100383

AA 33111

Qualifier



TIC

AA 150314

AA 128183

0.025 µg/g Fortification
RS 51037 0-18" Soil WA

CHRO: 1216rg518.dat (17-DEC-97)

Samp: E7-11-412-SA++

Comm: Set#1

Mode: TSP +QIMS LMR UP LR

Oper: RBG

Peak: 1000.0 mmu

Area: 2, 2, 0

m/z: 318.20 (SM 5)

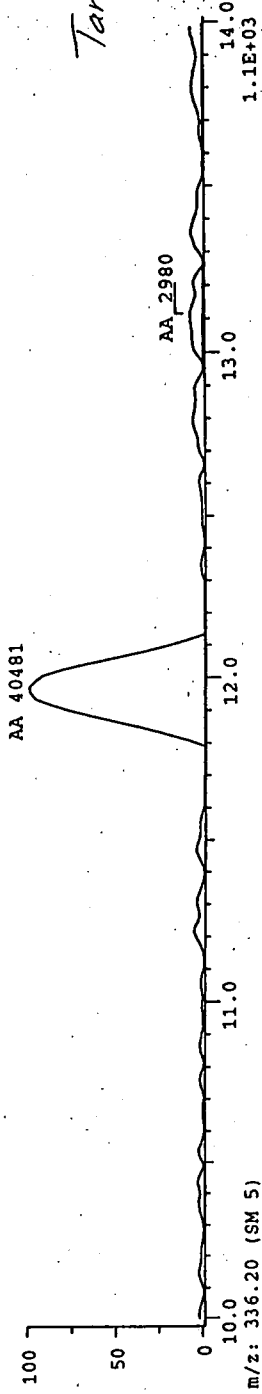
CHRO: 1216rg518.dat (17-DEC-97)
 Samp: E7-11-412-5A++
 Comm: Set#1
 Mode: TSP +QIMS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 334.20 (SM 5)

Elapse: 1 @ 8.24
 Times: 8.24 > 28.01

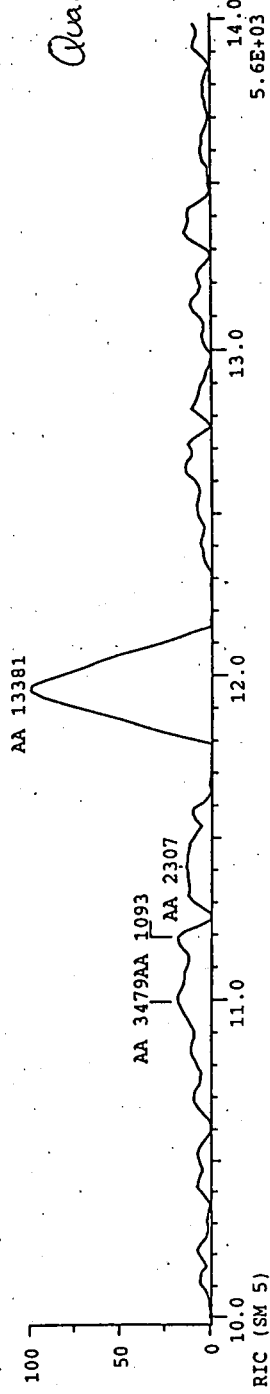
Masses: 317 > 336
 Client: RPA (USA)
 RIC: 3418
 Peak ID: 12, 5
 3.4E+03

Study: Trit.&metab. verif.
 Intensity: 3418
 Baseline: 10, 10

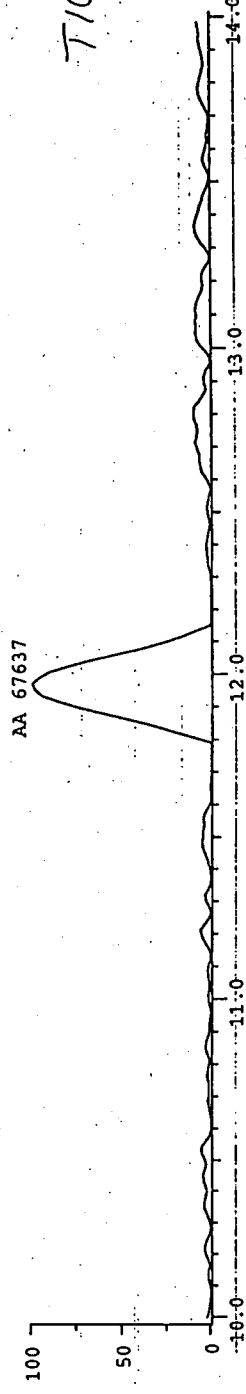
RPA 406341



Target



Qualifier



TIC

0.025 µg/g Fortification
 RS 51037 0-18" Soil WA

Date: Thu Dec 18 14:11:03 1997 ICIS: 8.3.0 for ULTRIX (4.4)

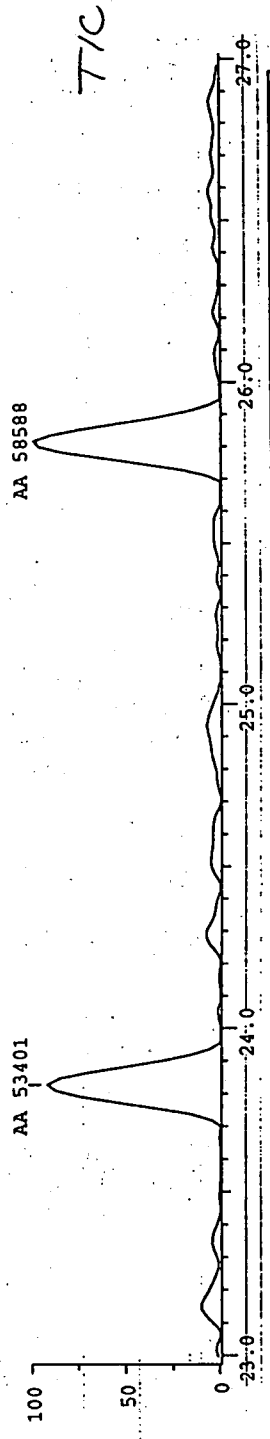
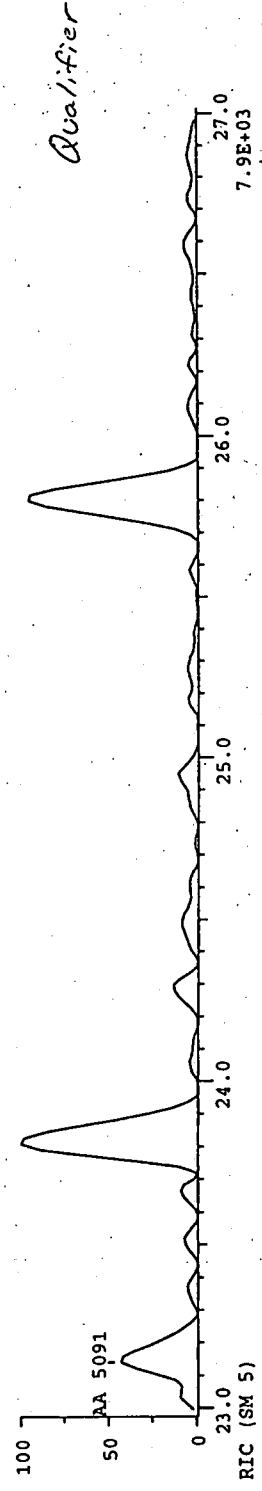
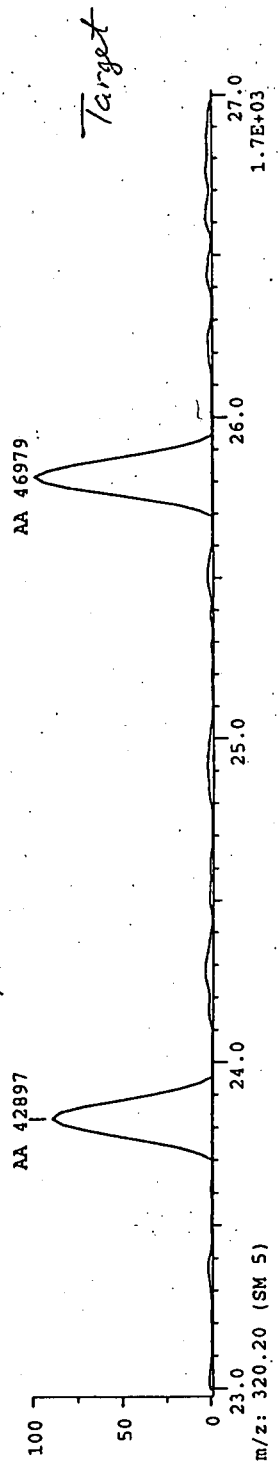
CHRO: 1216rg516.dat (17-DEC-97)
Samp: E7-11-412-16A*
Comm: Set#1
Mode: TSP +Q1MS LMR UP LR
Oper: RBG
Peak: 1000.0 mmu
Area: 2, 2, 0
m/z: 318.20 (SM 5) *RPA 400 727 (percent)*

Study: Trit.smetab. verif.
Intensity: 6290
Baseline: 10, 10

Elapse: 1 @ 8.23
Times: 8.23 > 28.01

Masses: 317 > 336
Client: RPA (USA)
RIC: 6290
Peak ID: 12, 5
6.3E+03

RPA 406 203 (cis)



0.005 µg/g Fortification
RS 51048 0-18" Soil NC

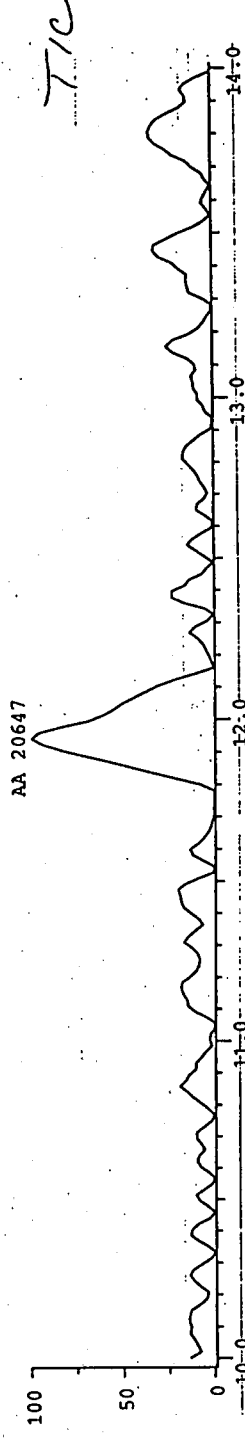
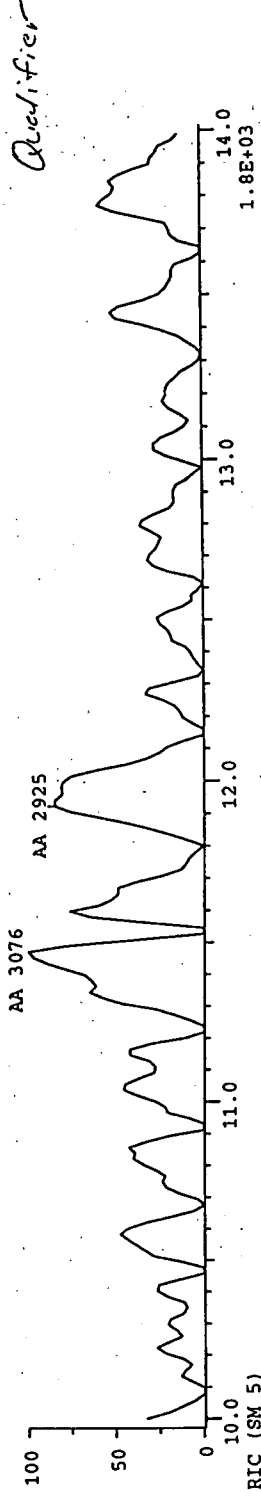
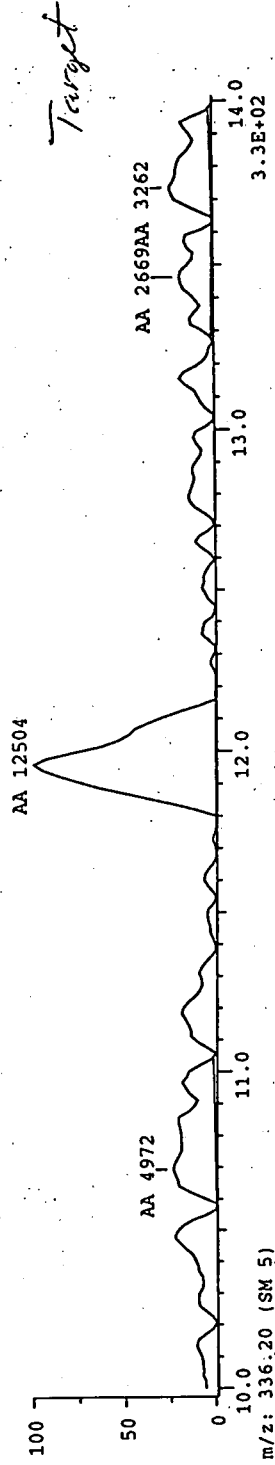
CHRO: 1216rg516.dat (17-DEC-97)
 Samp: E7-11-412-16A+
 Comm: Set#1
 Mode: TSP +Q1MS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 334.20 (SM 5)

Elapse: 1 @ 8.23
 Times: 8.23 > 28.01

Masses: 317 > 336
 Client: RPA (USA)
 RIC: 1132
 Peak ID: 12, 5
 1.1E+03

Study: Trit.&metab. verif.
 Intensity: 1132
 Baseline: 10, 10

RPA 406.341



0.005 µg/g Fortification
 RS 51048 0-18" Soil NC

Date: Thu Dec 18 14:17:48 1997 ICIS: 8.3.0 for ULTRIX (4.4)

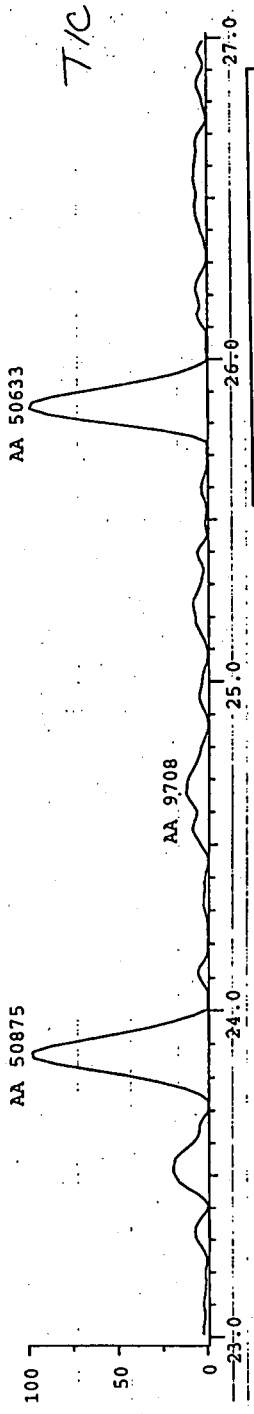
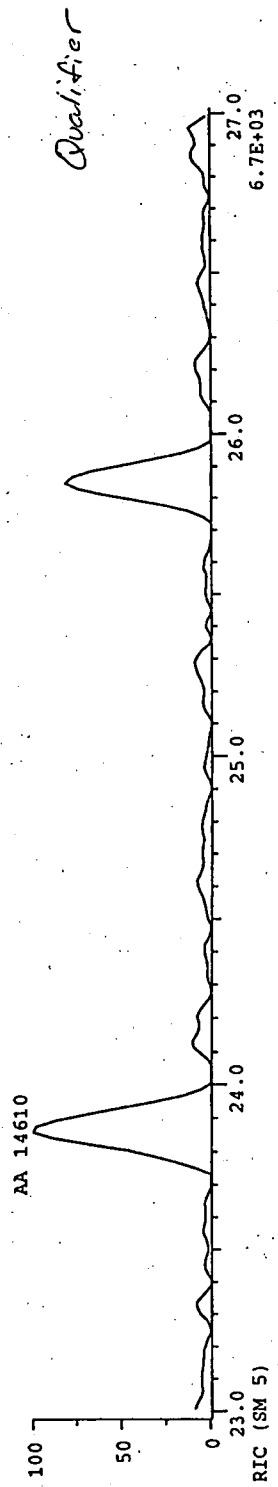
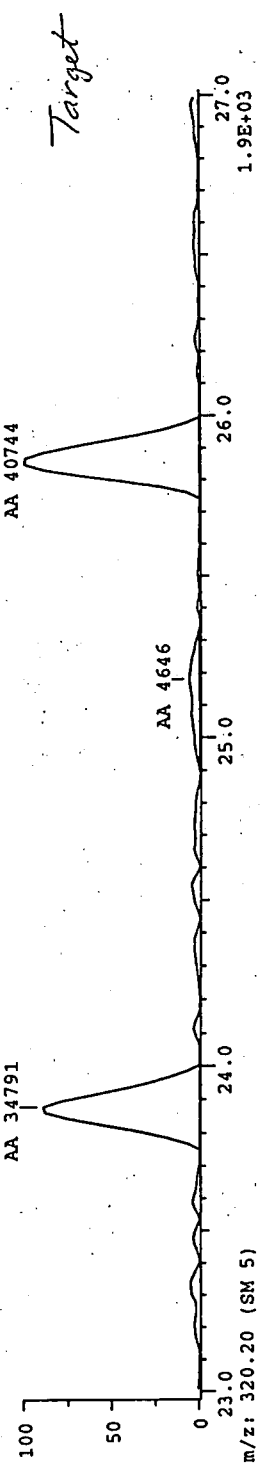
CHRO: 1216rg522.dat (17-DEC-97)
 Samp: E7-11-412-18A+
 Comm: Set#1
 Mode: TSP +Q1MS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 318.20 (SM 5)

Elapse: 1 @ 8.24
 Times: 8.24 > 28.00

Masses: 317 > 336
 Client: RPA (USA)
 RIC: 5263
 Peak ID: 12, 5
 5.3E+03

Study: Trit.smetab. verif.
 Intensity: 5263
 Baseline: 10, 10
 RPA 400787 (paint)

RPA 406203 (cis)

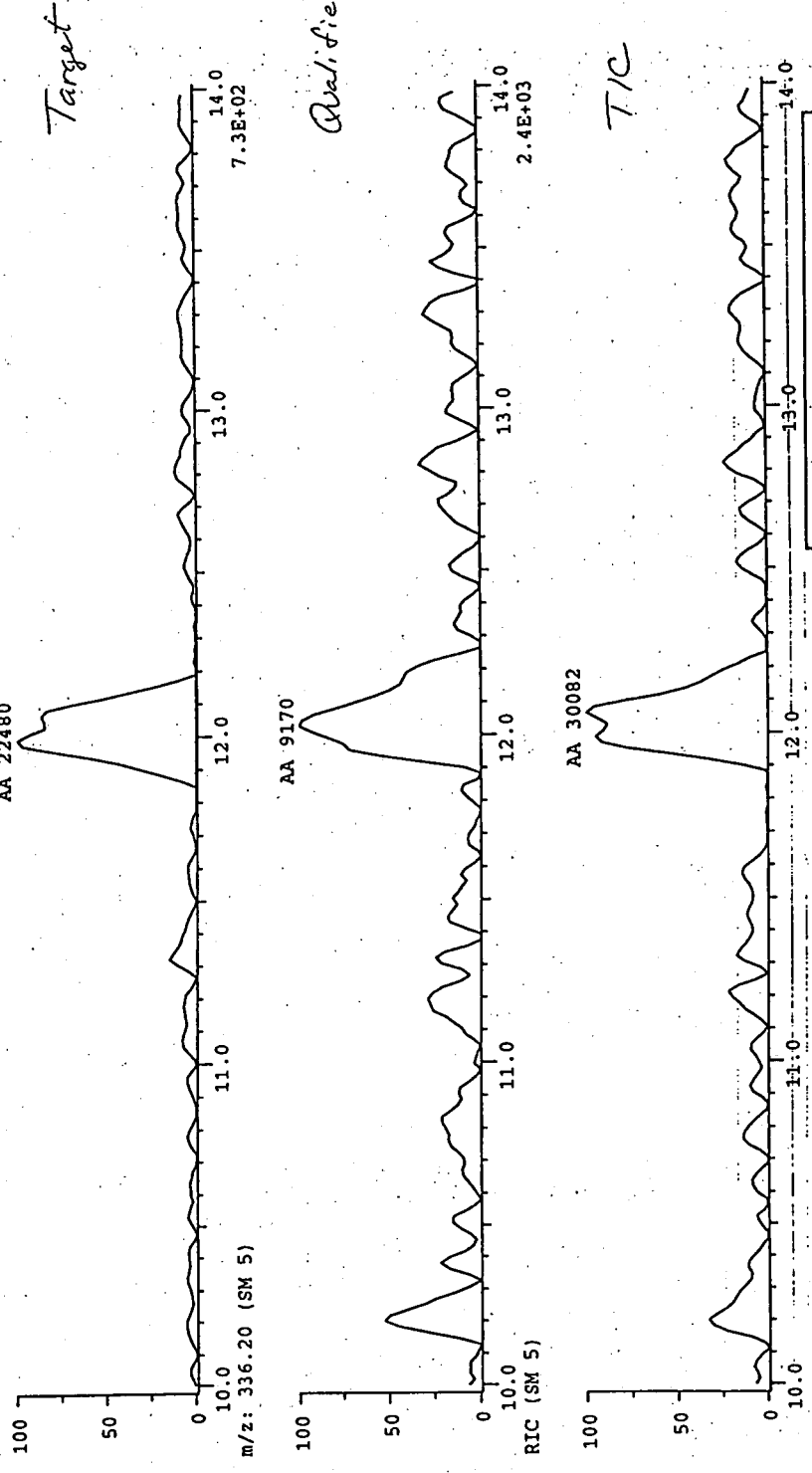


0.005 µg/g Fortification
 RS 51049 0-18" Turf CA

Date: Thu Dec 18 14:59:32 1997 ICIS: 8.3.0 for ULTRIX (4.4)

Elapse: 1 @ 8.24
Times: 8.24 > 28.00
Masses: 317 > 336
Client: RPA (USA)
RIC: 1954
Peak ID: 12, 5 2.0E+03

CHRO: 1216rg522.dat (17-DEC-97)
Samp: E7-11-412-18A+
Comm: Set#1
Mode: TSP +Q1MS LMR UP LR
Oper: RBG
Peak: 1000.0 mmu
Area: 2, 2, 0
m/z: 334.20 (SM 5)
Study: Trit.smetab. verif.
Intensity: 1954
Baseline: 10, 10
RPA 406341



0.005 µg/g Fortification
RS 51049 0-18" Turf CA

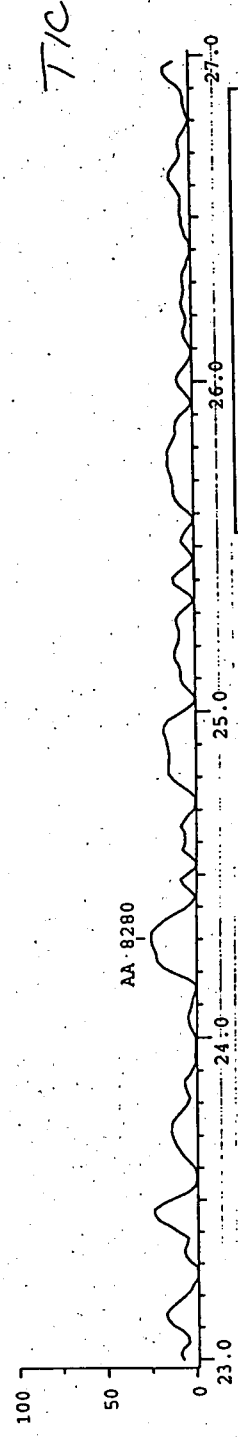
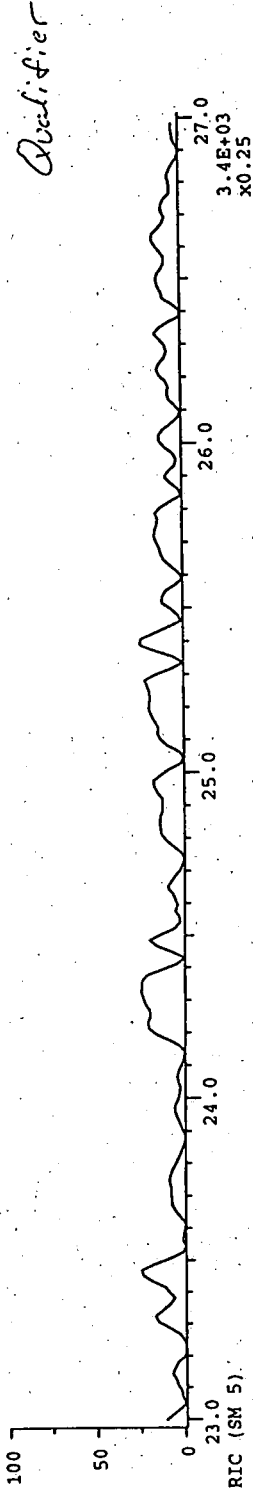
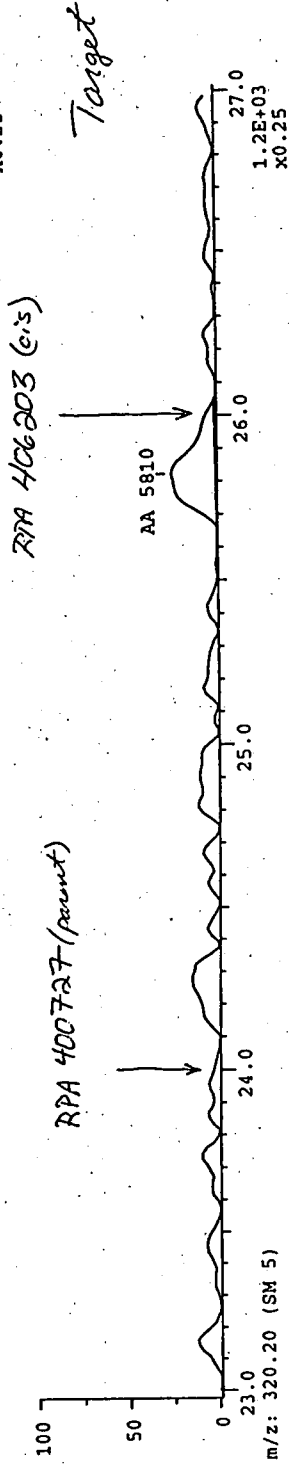
Date: Thu Dec 18 14:12:38 1997 ICIS: 8.3.0 for ULTRIX (4.4)

CHRO: 1216rg511.dat (16-DEC-97)
 Samp: E7-11-412-5A
 Comm: Set#1
 Mode: TSP +Q1MS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 318.20 (SM 5)

Study: Trit.&metab. verif.
 Intensity: 477
 Baseline: 10, 10

Elapse: 1 @ 8.24
 Times: 8.24 > 28.01

Masses: 317 > 336
 Client: RPA (USA)
 RIC: 477
 Peak ID: 5, 12
 1.9E+03
 X0.25



Control Soil
 RS 51037 0-18" Soil WA

Date: Thu Dec 18 15:22:13 1997 ICIS: 8.3.0 for ULTRIX (4.4)

CHRO: 1216rg511.dat (16-DEC-97)
Samp: E7-11-412-5A
Comm: Set#1
Mode: TSP +Q1MS LMR UP LR
Oper: RBG
Peak: 1000.0 mnu
Area: 2, 2, 0
m/z: 334.20 (SM 5)

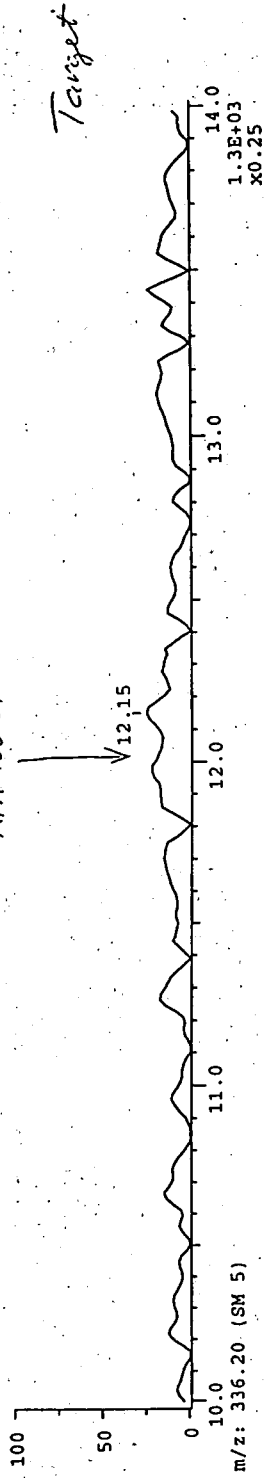
Study: Trit.&metab. verif.
Intensity: 355
Baseline: 10, 10

Elapse: 1 @ 8.24
Times: 8.24 > 28.01

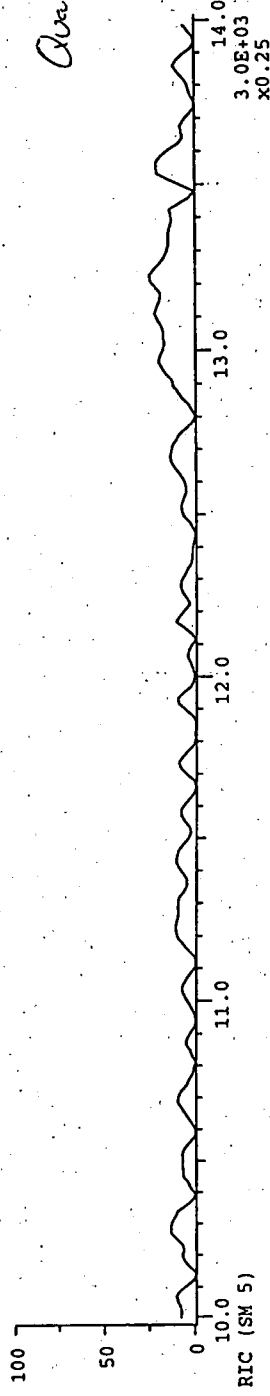
Masses: 317 > 336
Client: RPA (USA)
RIC: 355
Peak ID: 5, 12

1.4E+03
x0.25

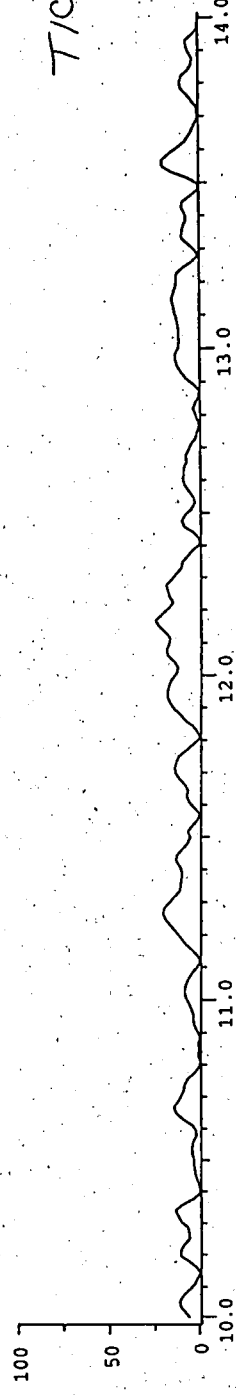
RPA 406341



Qualifier



TIC



Control Soil
RS 51037 0-18" Soil WA

Date: Fri Dec 19 11:47:25 1997 ICIS: 8.3.0 for ULTRIX (4.4)

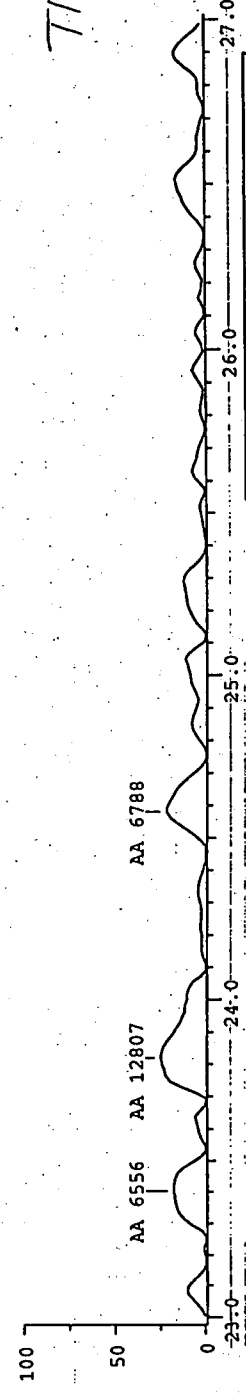
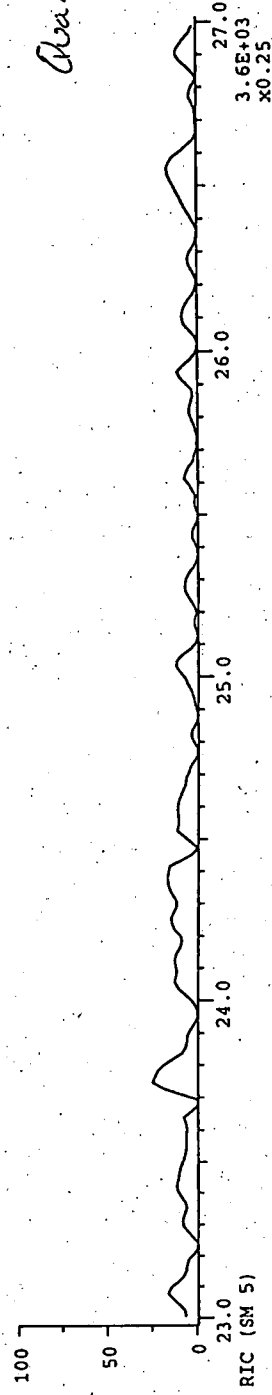
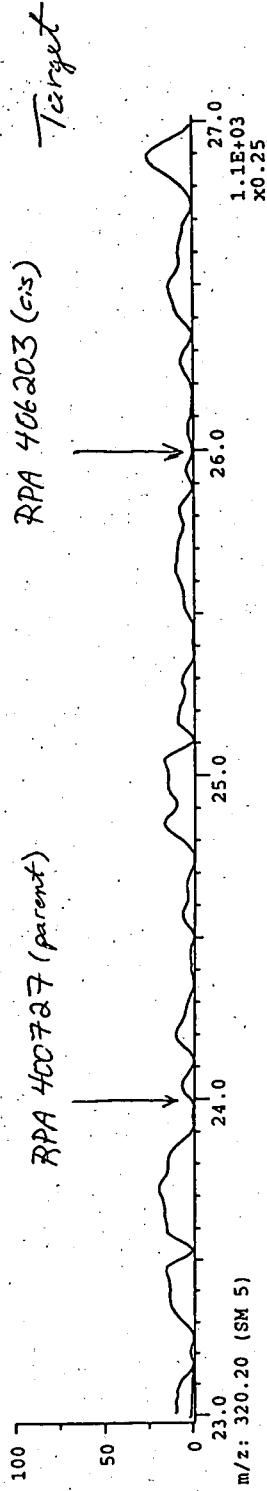
CHRO: 1216rg521.dat (17-DEC-97)
 Samp: E7-11-412-18A
 Comm: Set#1
 Mode: TSP +Q1MS LMR UP LR
 Oper: RBG
 Peak: 1000.0 mmu
 Area: 2, 2, 0
 m/z: 318.20 (SM 5)

Elapse: 1 @ 8.24
 Times: 8.24 > 28.02

Masses: 317 > 336
 Client: RPA (USA)
 RIC: 345
 Peak ID: 5, 12

Study: Trit.&metab. verif.
 Intensity: 345
 Baseline: 10, 10

1.4E+03
 x0.25



Date: Thu Dec 18 15:24:40 1997 ICIS: 8.3.0 for ULTRIX (4.4)

CHRO: 12169521.dat (17-DEC-97)
Samp: E7-11-412-18A
Comm: Set#1
Mode: TSP +QIMS LMR UP LR
Oper: RBG
Study: Trit.smetab. verif.
Peak: 1000.0 mmu
Area: 2, 2, 0
m/z: 334.20 (SM 5)

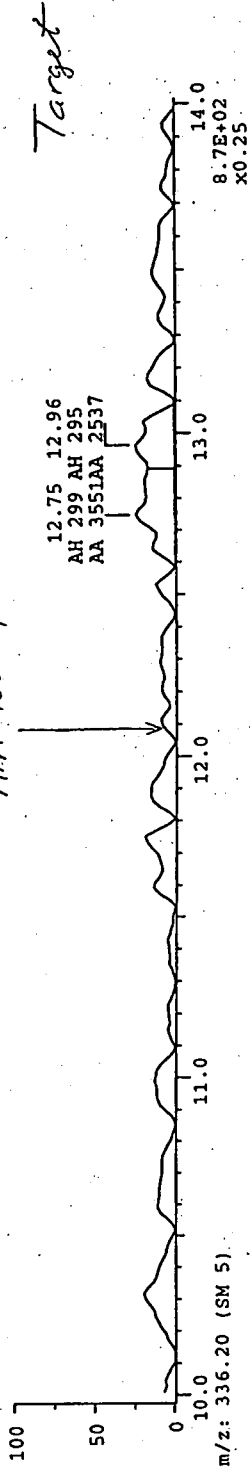
Intensity: 302
Baseline: 10, 10

Elapsed: 1 @ 8.24
Times: 8.24 > 28.02

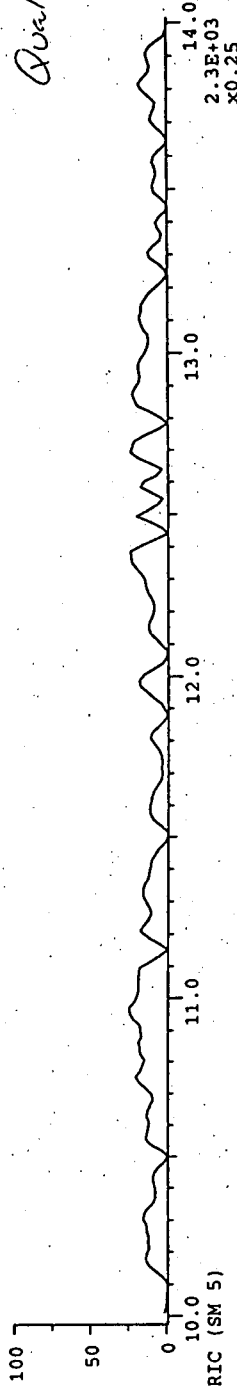
Masses: 317 > 336
Client: RPA (USA)
RIC: 302
Peak ID: 5, 12

1.2E+03
x0.25

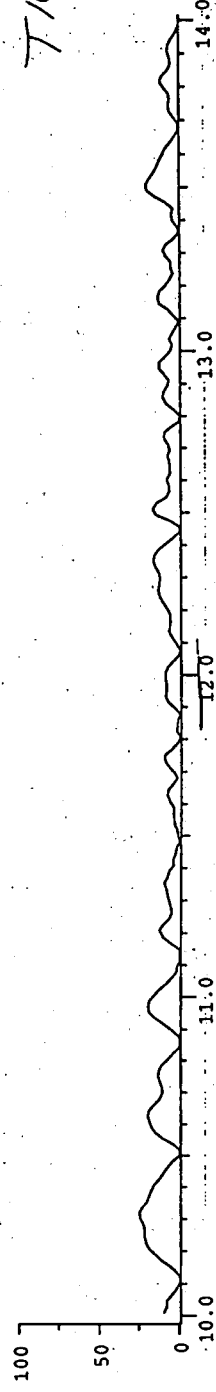
RPA 406.341



Qualifier



TIC



Control Turf
RS 51049 0-18" Turf CA

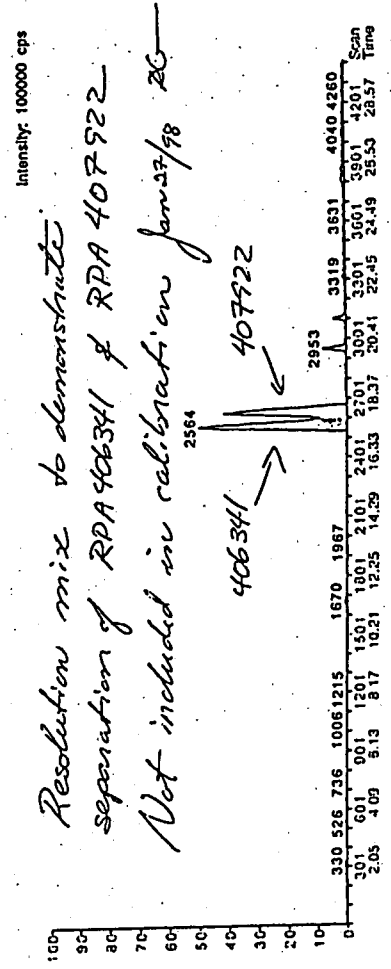
Date: Fri Dec 19 11:49:23 1997 ICIS: 8.3.0 for ULTRIX (4.4)

**10.0 EXAMPLE CHROMATOGRAMS:
SCIEX API-150EX LC/MS**

MacQuan, version 1.5
Printed: Wed, Jan 21, 1998 10:05
Calibration File: NM012098Cal Path: Macintosh HD:AP1150EX #040:DATA:RHONPO:TRIT:NM012098:
Comments: No comments

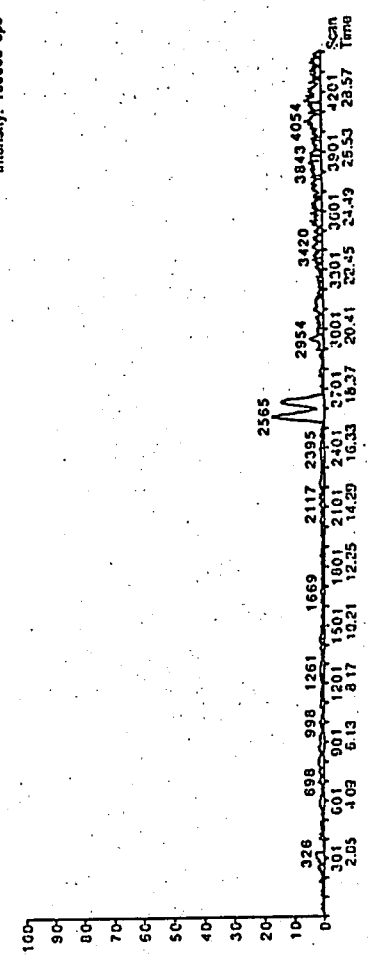
001 Resolution mix 0.100 ppm Tue, Jan 20, 1998 14:39
No Comment

29.98 In 1 period
RPA 406341
No Internal Standard
Use Area
1: 29.99 01 MI, 4411 scans
Noise Thres. 10.0
Quant Thres. 5.0
Min. Width 3
Mult. Width 10
Base. Width 100
RT Wn. (secs) 20
Smooth 2
Expected RT 17.44
Area 804648
Height 50197
Start Time 17.22
End Time 17.69
Integration Width 0.46
Retention Time 17.44
Integration Type A - BV



001 Resolution mix 0.100 ppm Tue, Jan 20, 1998 14:39
No Comment

29.98 In 1 period
RPA 406341 (conf)
No Internal Standard
Use Area
1: 29.99 01 MI, 4411 scans
Noise Thres. 10.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 5
Base. Width 100
RT Wn. (secs) 20
Smooth 2
Expected RT 17.44
Area 290126
Height 17728
Start Time 17.20
End Time 17.67
Integration Width 0.46
Retention Time 17.44
Integration Type A - BV



MacQuan, version 1.5

Printed: Fri, Jan 30, 1998 13:53

Calibration File: RG012998CAL Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:RG012998:

Comments: No comments

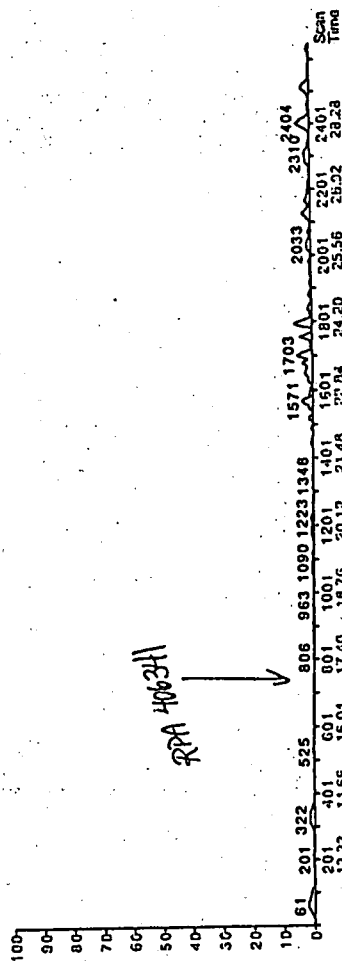
004 E7-11-412-17A(-2) 3ml Thu, Jan 29, 1998 16:24

NC TURF

No Comment
29.95 In 1 period
RPA 406341
No Internal Standard
Use Area

1: 17.99 Q1 M1, 2647 scans
334.0
Noise Thres. 50.0
Quant Thres. 5.0
Min. Width 3
Mult. Width 10
Base. Width 100
RT Win. (secs) 20
Smooth 2
Expected RT 17.31
Area 0
Height 0
Start Time -0.00
End Time -0.00
Integration Width -0.00
Retention Time -0.00
Integration Type

Intensity: 100000 cps



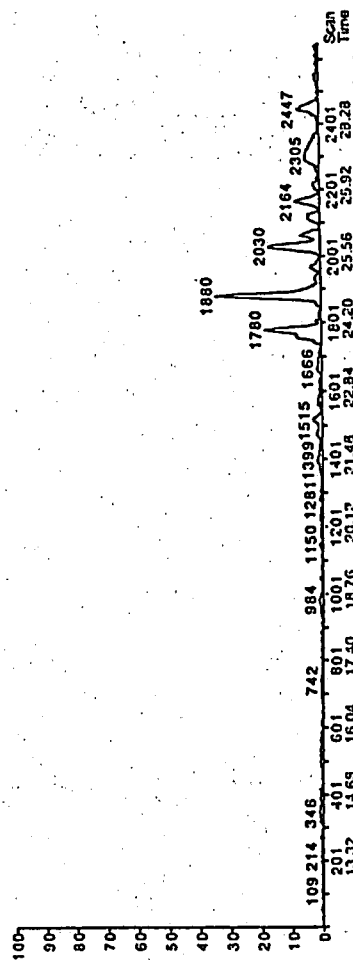
004 E7-11-412-17A(-2) 3ml Thu, Jan 29, 1998 16:24

No Comment

29.95 In 1 period
RPA 406341 (cont)
No Internal Standard
Use Area

1: 17.99 Q1 M1, 2647 scans
338.0
Noise Thres. 50.0
Quant Thres. 2.0
Min. Width 8
Mult. Width 10
Base. Width 100
RT Win. (secs) 20
Smooth 2
Expected RT 17.49
Area 0
Height 0
Start Time -0.00
End Time -0.00
Integration Width -0.00
Retention Time -0.00
Integration Type

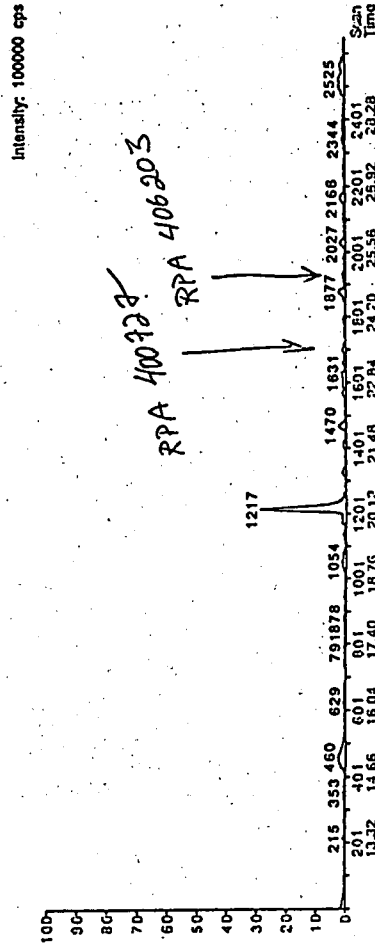
Intensity: 100000 cps



MacQuan, version 1.5
Printed: Fri, Jan 30, 1998 13:53
Calibration File: RG012998CAL Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:RG012998:
Comments: No comments

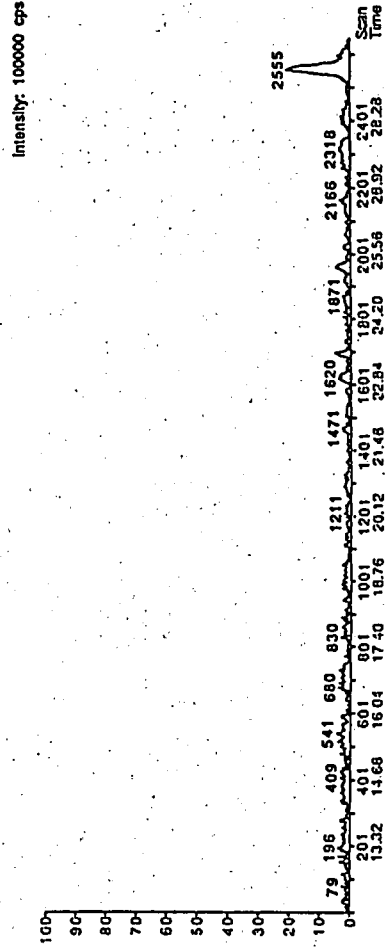
004 E7-11-412-17A(-2) 3ml Thu, Jan 29, 1998 16:24
No Comment

29.95 In 1 period
RPA 400727
No Internal Standard
Use Area
1: 17.99 Q1 MI, 2647 scans
116.0
Noise Thres. 50.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 5
Base. Width 100
RT Wn. (secs) 20
Smooth 2
Expected RT 23.56
Area 0
Height 0
Start Time -0.00
End Time -0.00
Integration Width -0.00
Retention Time -0.00
Integration Type



004 E7-11-412-17A(-2) 3ml Thu, Jan 29, 1998 16:24
No Comment

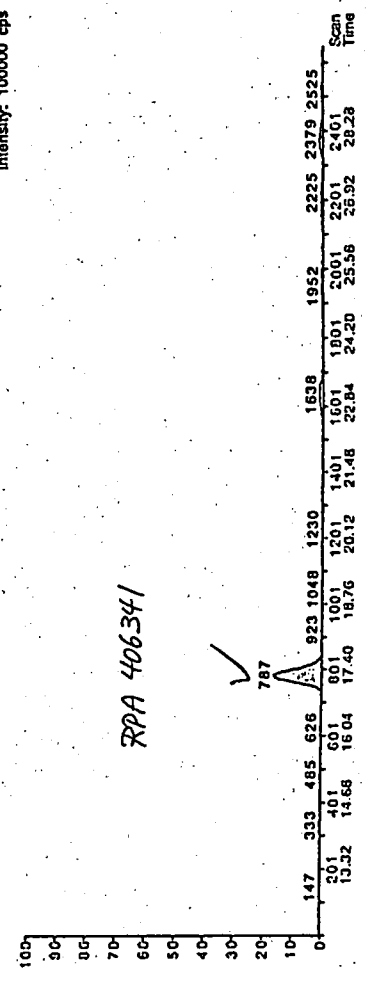
29.95 In 1 period
RPA 400727 (conf)
No Internal Standard
Use Area
1: 17.99 Q1 MI, 2647 scans
320.0
Noise Thres. 70.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 8
Base. Width 100
RT Wn. (secs) 20
Smooth 2
Expected RT 23.60
Area 29659
Height 5067
Start Time 23.39
End Time 23.58
Integration Width 0.18
Retention Time 23.50
Integration Type A-WV



MacQuan, version 1.5
 Printed: Fri, Jan 30, 1998 13:53
 Calibration File: RG012998CAL Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:RG012998:
 Comments: No comments

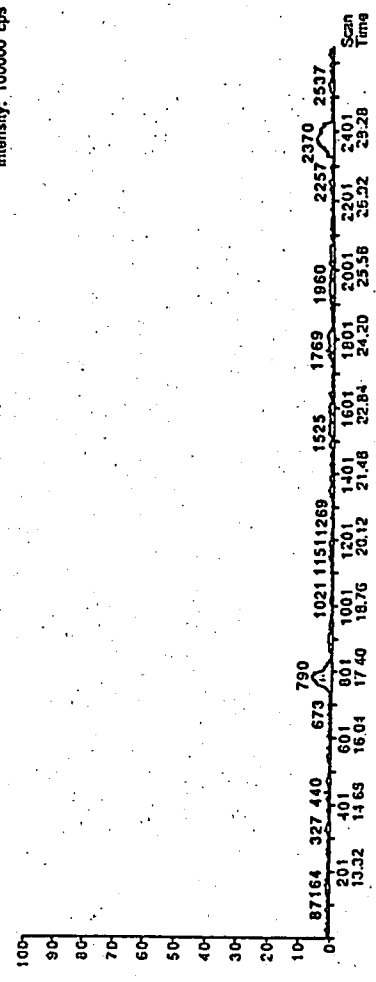
006 RPA CAL Mix #2 0.025 ppm Thu, Jan 29, 1998 17:40
 No Comment

29.95 in 1 period
 RPA 406341
 No Internal Standard
 Use Area
 1: 17.99 01 Mi. 2647 scans
 334.0
 Noise Thres. 50.0
 Quant Thres. 5.0
 Min. Width 3
 Mult. Width 10
 Base. Width 100
 RT Win. (secs) 20
 Smooth 2
 Expected RT 17.31
 Area 310228
 Height 16670
 Start Time 16.99
 End Time 17.67
 Integration Width 0.68
 Retention Time 17.31
 Integration Type A-BB



006 RPA CAL Mix #2 0.025 ppm Thu, Jan 29, 1998 17:40
 No Comment

29.95 in 1 period
 RPA 406341 (cont)
 No Internal Standard
 Use Area
 1: 17.99 01 Mi. 2647 scans
 336.0
 Noise Thres. 50.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 10
 Base. Width 100
 RT Win. (secs) 20
 Smooth 2
 Expected RT 17.49
 Area 96370
 Height 6495
 Start Time 17.21
 End Time 17.73
 Integration Width 0.52
 Retention Time 17.33
 Integration Type A-VB



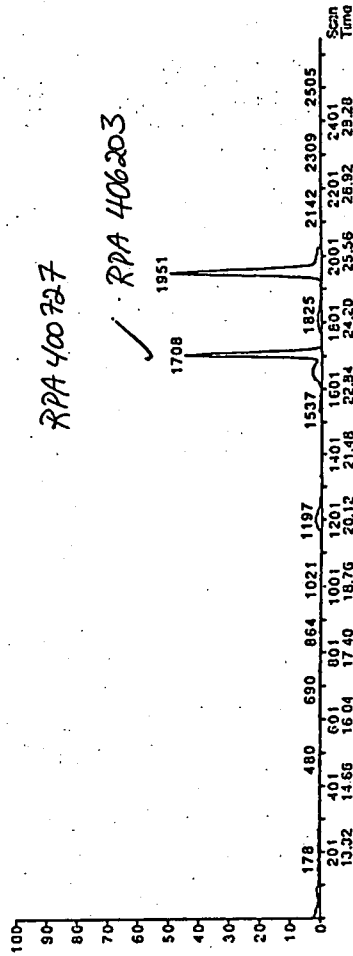
006 RPA CAL Mix #2 0.025 ppm Thu, Jan 29, 1998 17:40

No Comment

29.95 In 1 period
RPA 400727
No Internal Standard
Use Area

1: 17.99 Q1 MI, 2647 scans

318.0
Noise Thres. 50.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 5
Base Width 100
RT Wrt. (secs) 2.0
Smooth 2
Expected RT 23.56
Area 298220
Height 44895
Start Time 23.43
End Time 23.70
Integration Width 0.27
Retention Time 23.57
Integration Type A - VB



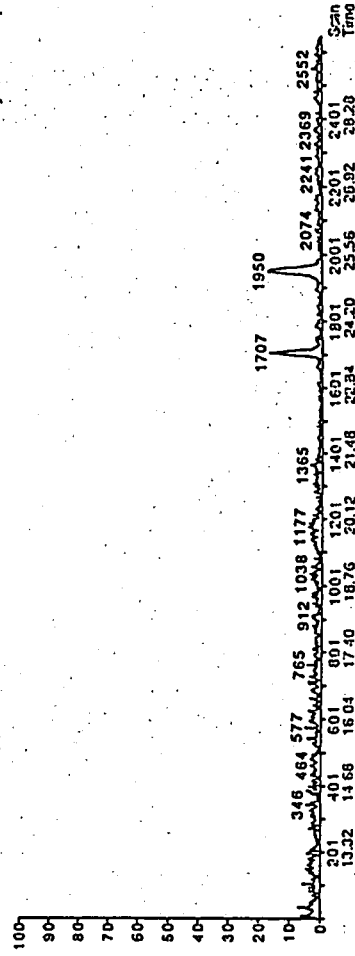
006 RPA CAL Mix #2 0.025 ppm Thu, Jan 29, 1998 17:40

No Comment

29.95 In 1 period
RPA 400727 (conf)
No Internal Standard
Use Area

1: 17.99 Q1 MI, 2647 scans

320.0
Noise Thres. 70.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 8
Base Width 100
RT Wrt. (secs) 2.0
Smooth 2
Expected RT 23.60
Area 117479
Height 16976
Start Time 23.45
End Time 23.74
Integration Width 0.29
Retention Time 23.56
Integration Type A - VV



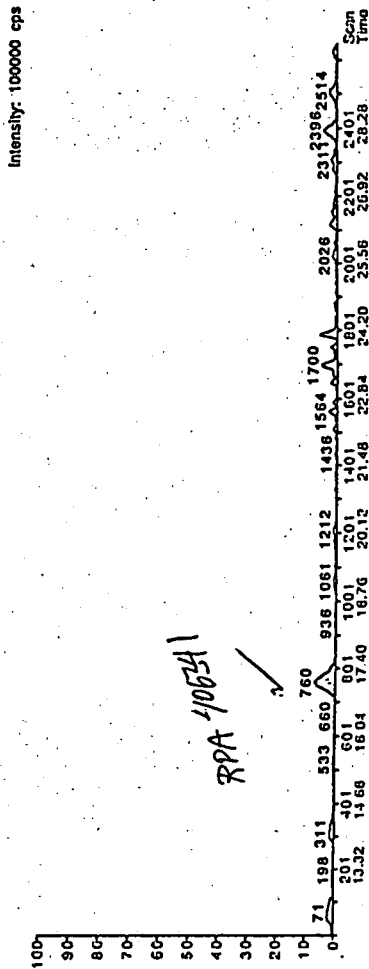
MacQuan, version 1.5
Printed: Fri, Jan 30, 1998 13:53
Calibration File: RG012998CAL Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:RG012998:
Comments: No comments

007 E7-11-412-17A(+4) 3mL Thu, Jan 29, 1998 16:18

No Comment

29.95 in 1 period
RPA 406341
No Internal Standard
Use Area

1: 17.99 01 Ml, 2647 scans
334.0
Noise Thres. 50.0
Count Thres. 3
Min. Width 5.0
Mult. Width 10
Base. Width 100
RT Wrt. (secs) 20
Smooth 2
Expected RT 17.31
Area 150388
Height 7341
Start Time 16.84
End Time 17.52
Integration Width 0.69
Retention Time 17.12
Integration Type A-BB



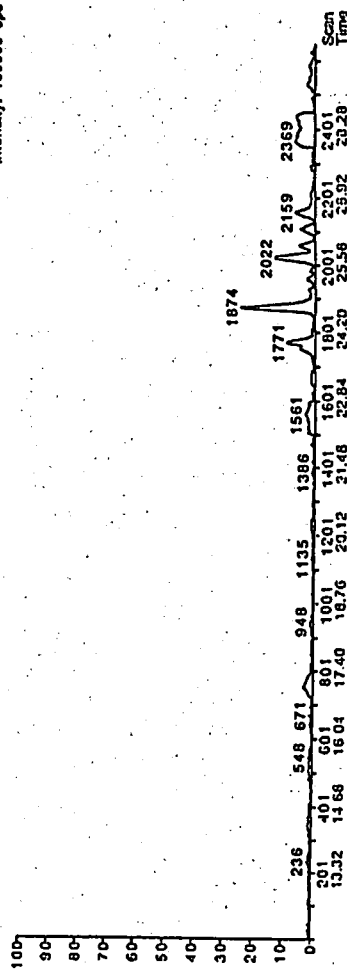
Analytical Method
MS 90.01 Revision 3

007 E7-11-412-17A(+4) 3mL Thu, Jan 29, 1998 16:18

No Comment

29.95 in 1 period
RPA 406341 (cont)
No Internal Standard
Use Area

1: 17.99 01 Ml, 2647 scans
336.0
Noise Thres. 50.0
Count Thres. 2.0
Min. Width 6
Mult. Width 10
Base. Width 100
RT Wrt. (secs) 20
Smooth 2
Expected RT 17.49
Area 0
Height 0
Start Time -0.00
End Time -0.00
Integration Width -0.00
Retention Time -0.00
Integration Type



007 E7-11-412-17A(+4) 3mL Thu, Jan 29, 1998 18:18

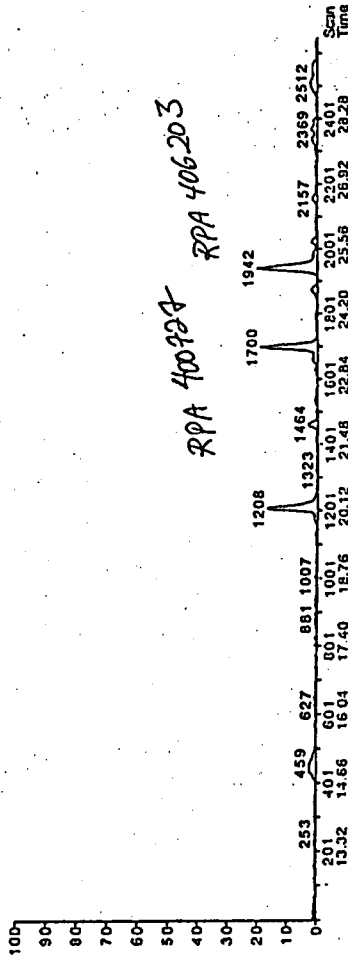
No Comment

29.95 in 1 period
RPA 400727
No Internal Standard
Use Area

1: 17.99 Q1 MI, 2647 scans
318.0
Noise Thres. 50.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 5
Base. Width 100
RT Wdn. (secs) 20
Smooth 2
Expected RT 23.56
Area 145284
Height 19381

Start Time 23.32
End Time 23.68
Integration Width 0.36
Retention Time 23.51
Integration Type A-VB

Intensity: 100000 cps



RPA 400727 RPA 406203

007 E7-11-412-17A(+4) 3mL Thu, Jan 29, 1998 18:18

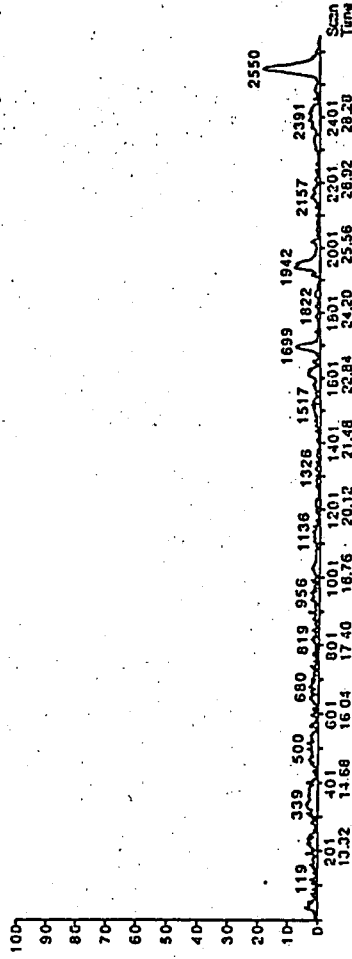
No Comment

29.95 in 1 period
RPA 400727 (cont)
No Internal Standard
Use Area

1: 17.99 Q1 MI, 2647 scans
320.0
Noise Thres. 70.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 8
Base. Width 100
RT Wdn. (secs) 20
Smooth 2
Expected RT 23.60
Area 73230
Height 8124

Start Time 23.38
End Time 23.66
Integration Width 0.30
Retention Time 23.51
Integration Type A-VV

Intensity: 100000 cps



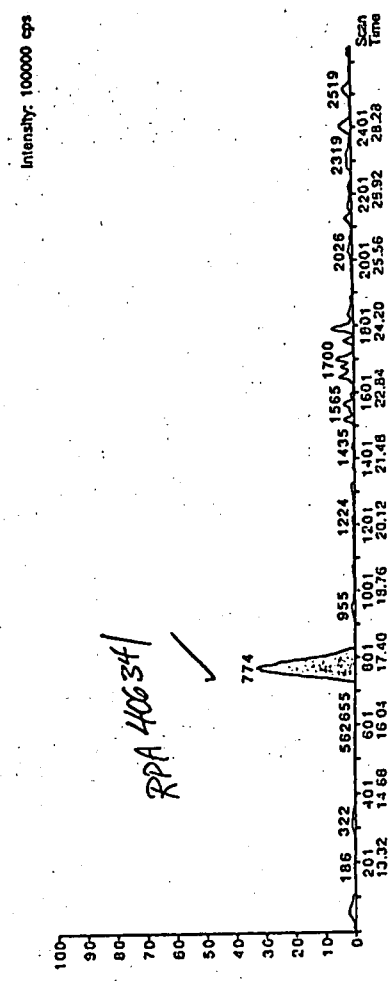
NC Control Turf spiked at LOQ
of 0.005 ppm
(RPA 400727/RPA 406203)

Juan, version 1.5
Printed: Fri, Jan 30, 1998 13:54
Calibration File: RG012998CAL Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:RG012998:
Comments: No comments

010 E7-11-412-17A(++3) 3mL Thu, Jan 29, 1998 20:11
No Comment

29.95 In 1 period
RPA 406341
No Internal Standard
Use Area
1: 17.99 01 Mi, 2647 scans
Noise Thres. 50.0
Quant Thres. 5.0
Min. Width 3
Mult. Width 10
Base. Width 100
RT Wn. (secs) 20
Smooth 2
Expected RT 17.31
Area 725297
Height 33593
Start Time 16.90
End Time 17.58
Integration Width 0.68
Retention Time 17.22
Integration Type A - BB

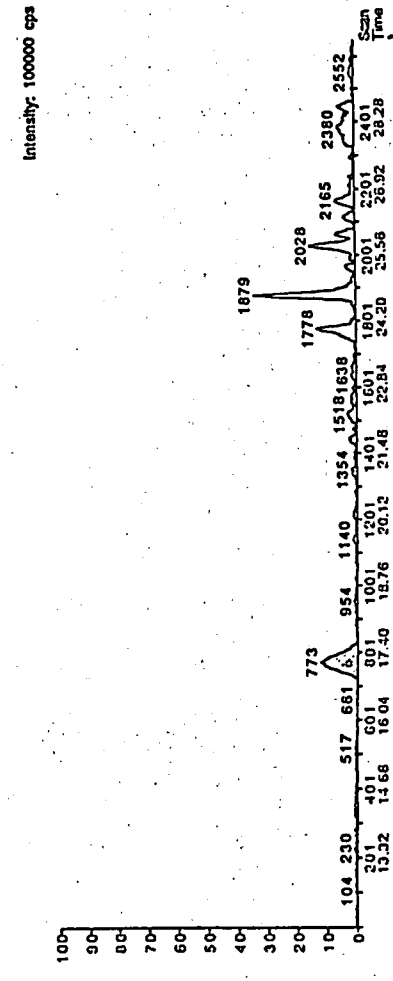
RPA 406341 ✓



010 E7-11-412-17A(++3) 3mL Thu, Jan 29, 1998 20:11
No Comment

29.95 In 1 period
RPA 406341 (cont)
No Internal Standard
Use Area
1: 17.99 01 Mi, 2647 scans
Noise Thres. 50.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 10
Base. Width 100
RT Wn. (secs) 20
Smooth 2
Expected RT 17.49
Area 271469
Height 12639
Start Time 16.89
End Time 17.64
Integration Width 0.75
Retention Time 17.21
Integration Type A - BB

NC Control Turf spiked at 5 x
LOQ (0.025 ppm)
(RPA 406341)



1 Juan, version 1.5

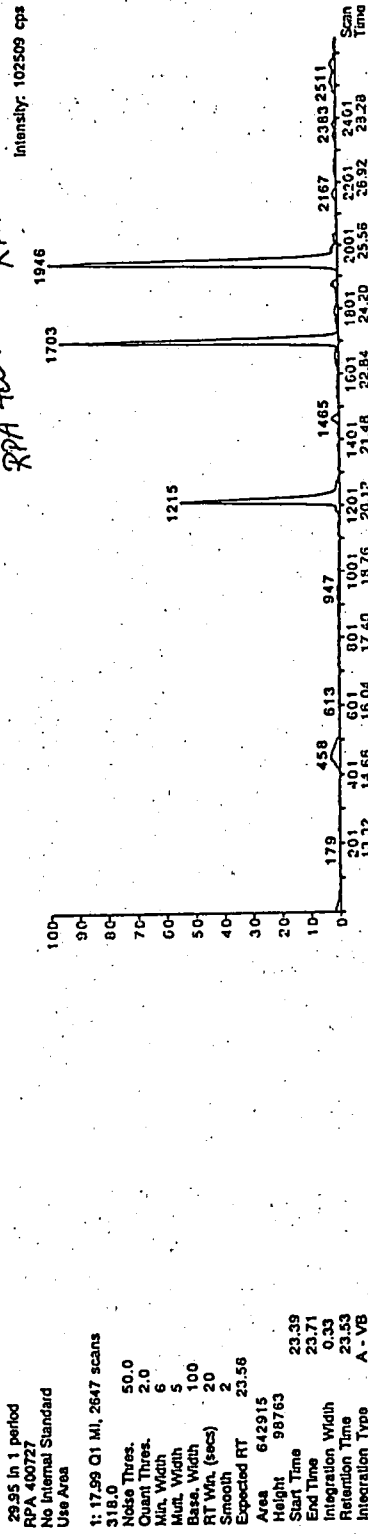
Printed: Fri, Jan 30, 1998 13:54

Calibration File: RG012998CAL Path: Macintosh HD-AP1150EX #040:DATA-RHONPO:TRIT:RG012998:

Comments: No comments

010 E7-11-412-17A(++3) 3mL Thu, Jan 29, 1998 20:11

RPA 400727 RPA 406203

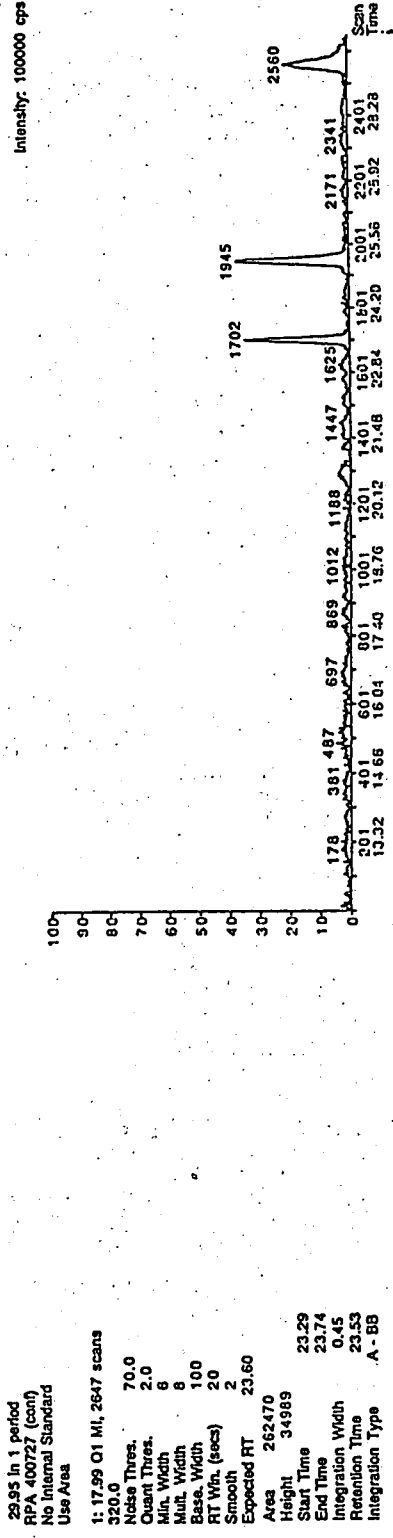


No Comment

29.95 In 1 period
 RPA 400727
 No Internal Standard
 Use Area

1: 17.59 Q1 M1, 2647 scans
 318.0
 Noise Thres. 50.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 5
 Base. Width 100
 RT Wn. (secs) 20
 Smooth 2
 Expected RT 23.56
 Area 642915
 Height 98763
 Start Time 23.39
 End Time 23.71
 Integration Width 0.33
 Retention Time 23.53
 Integration Type A-VB

010 E7-11-412-17A(++3) 3mL Thu, Jan 29, 1998 20:11



No Comment

29.95 In 1 period
 RPA 400727 (conf)
 No Internal Standard
 Use Area

1: 17.59 Q1 M1, 2647 scans
 320.0
 Noise Thres. 70.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 8
 Base. Width 100
 RT Wn. (secs) 20
 Smooth 2
 Expected RT 23.60
 Area 262470
 Height 34989
 Start Time 23.29
 End Time 23.74
 Integration Width 0.45
 Retention Time 23.53
 Integration Type A-BB

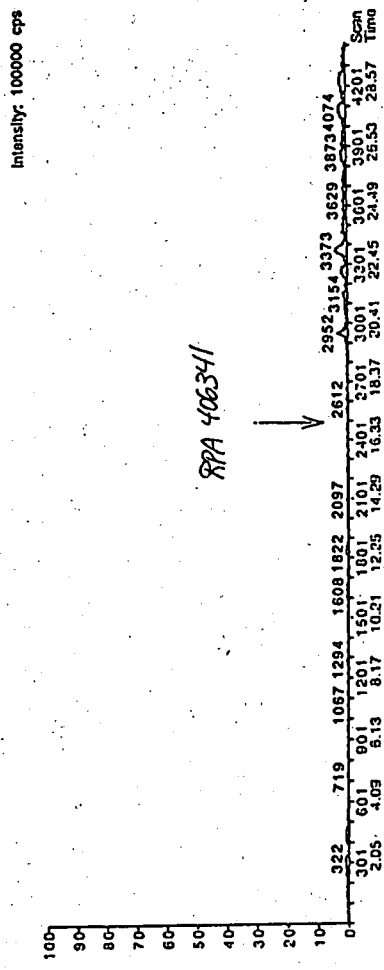
NC Control Turf spiked at 5 x
 LOQ (0.025 ppm)
 (RPA 400727/RPA 406203)

hvacQuan, version 1.5
 Printed: Wed, Jan 21, 1998 10:07
 Calibration File: NM012098Cal Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:NM012098:
 Comments: No comments

005 E7-11-412-05A(-1) Tue, Jan 20, 1998 17:29

No Comment
 29.98 In 1 period
 RPA 406341
 No Internal Standard
 Use Area

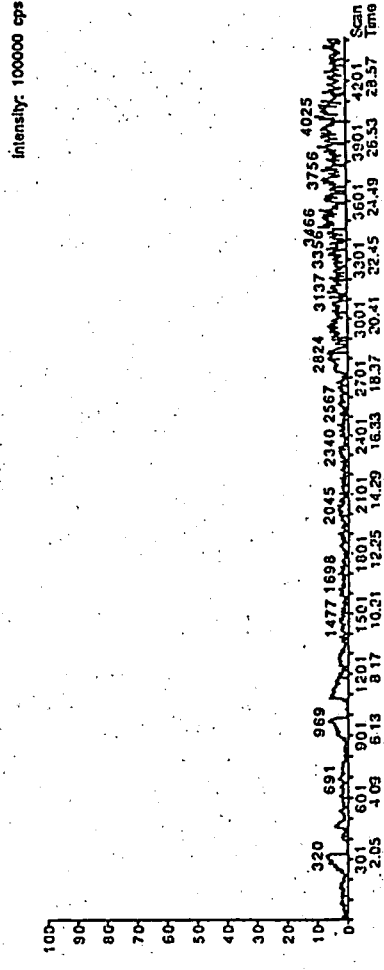
1: 29.99 Q1 MI, 4411 scans
 334.0
 Noise Thres. 10.0
 Quant Thres. 5.0
 Min. Width 3
 Mult. Width 10
 Base. Width 100
 RT Win. (secs) 20
 Smooth 2
 Expected RT 17.44
 Area 0
 Height 0
 Start Time -0.00
 End Time -0.00
 Integration Width -0.00
 Retention Time -0.00
 Integration Type M



005 E7-11-412-05A(-1) Tue, Jan 20, 1998 17:29

No Comment
 29.98 In 1 period
 RPA 406341 (cont)
 No Internal Standard
 Use Area

1: 29.99 Q1 MI, 4411 scans
 336.0
 Noise Thres. 10.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 5
 Base. Width 100
 RT Win. (secs) 20
 Smooth 2
 Expected RT 17.44
 Area 14101
 Height 3342
 Start Time 17.41
 End Time 17.51
 Integration Width 0.10
 Retention Time 17.46
 Integration Type A-VV



A Juan, version 1.5

Printed: Wed, Jan 21, 1998 10:07

Calibration File: NM012098Cal Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:NM012098:

Comments: No comments

005 E7-11-412-05A(-1) Tue, Jan 20, 1998 17:29

No Comment

29.98 In 1 period

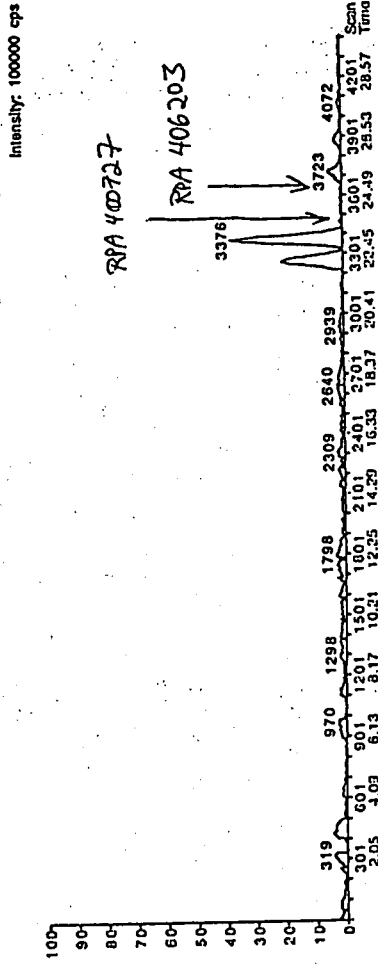
RPA 400727

No Internal Standard

Use Area

1: 29.99 Q1 MI, 4411 scans

318.0
 Noise Thres. 10.0
 Quant Thres. 2.0
 Min. Width 5
 Mult. Width 5
 Base. Width 100
 RT Wn. (secs) 20
 Smooth 2
 Expected RT 23.60
 Area 0
 Height 0
 Start Time -0.00
 End Time -0.00
 Integration Width -0.00
 Retention Time -0.00
 Integration Type M



005 E7-11-412-05A(-1) Tue, Jan 20, 1998 17:29

No Comment

29.98 In 1 period

RPA 400727 (cont)

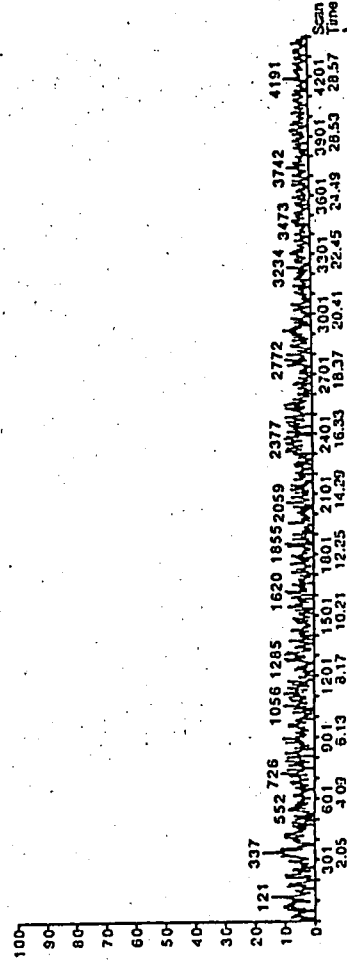
No Internal Standard

Use Area

1: 29.99 Q1 MI, 4411 scans

320.0
 Noise Thres. 10.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 5
 Base. Width 100
 RT Wn. (secs) 20
 Smooth 2
 Expected RT 23.60
 Area 23134
 Height 5792
 Start Time 23.70
 End Time 23.70
 Integration Width 0.10
 Retention Time 23.62
 Integration Type A-VV

Intensity: 100000 cps



Juan, version 1.5

Printed: Wed, Jan 21, 1998 10:10

Calibration File: NMO12098Cal Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:NMO12098:

Comments: No comments

011 E7-11-412-05A(+3) Tue, Jan 20, 1998 21:22

No Comment

29.98 In 1 period

RPA 406341

No Internal Standard

Use Area

1: 29.99 01 MI, 4411 scans

334.0

Noise Thres. 10.0

Quant Thres. 5.0

Min. Width 3

Mult. Width 10

Base. Width 100

RT Wn. (secs) 20

Smooth 2

Expected RT 17.44

Area 296818

Height 17538

Start Time 16.78

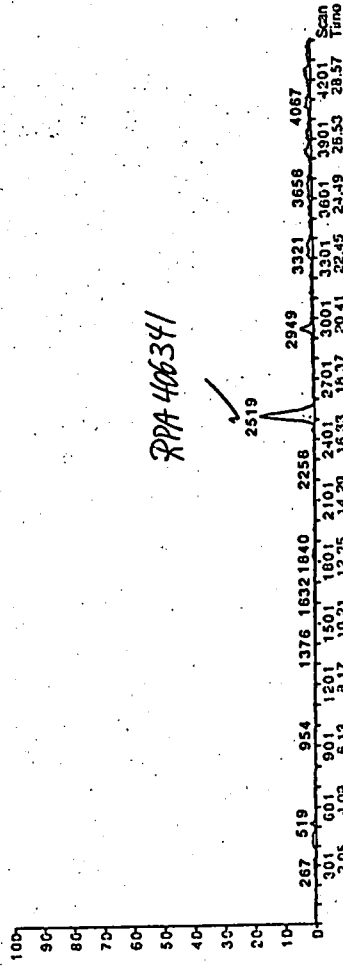
End Time 17.48

Integration Width 0.69

Retention Time 17.13

Integration Type M

Intensity: 100000 cps



011 E7-11-412-05A(+3) Tue, Jan 20, 1998 21:22

No Comment

29.98 In 1 period

RPA 406341 (conf)

No Internal Standard

Use Area

1: 29.99 01 MI, 4411 scans

336.0

Noise Thres. 10.0

Quant Thres. 2.0

Min. Width 6

Mult. Width 5

Base. Width 100

RT Wn. (secs) 20

Smooth 2

Expected RT 17.44

Area 2810

Height 1078

Start Time 17.59

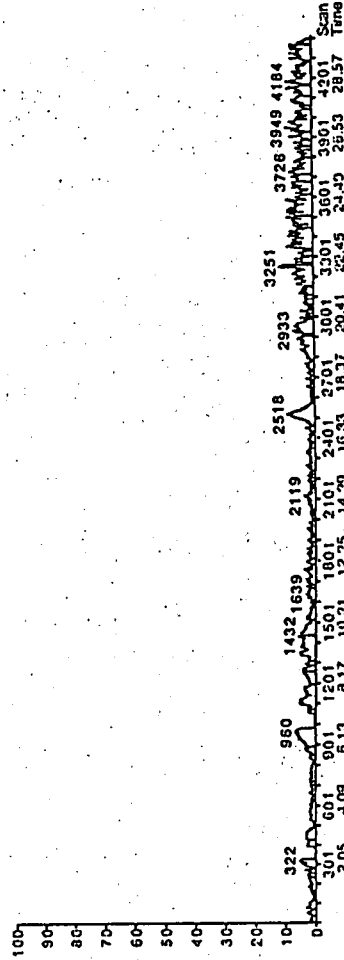
End Time 17.66

Integration Width 0.07

Retention Time 17.65

Integration Type A-BB

Intensity: 100000 cps



NC Control Soil spiked at LOQ
of 0.005 ppm
(RPA 406341)

011 E7-11-412-05A(+3) Tue, Jan 20, 1998 21:22

No Comment

29.98 In 1 period

RPA 400727

No Internal Standard

Use Area

1: 29.99 Q1 M1, 4411 scans

318.0

Noise Thres. 10.0

Quant Thres. 2.0

Min. Width 6

Mult. Width 5

Base. Width 100

RT Wrt. (secs) 20

Smooth 2

Expected RT 23.60

Area 265738 ✓

Height 44722

Start Time 23.38

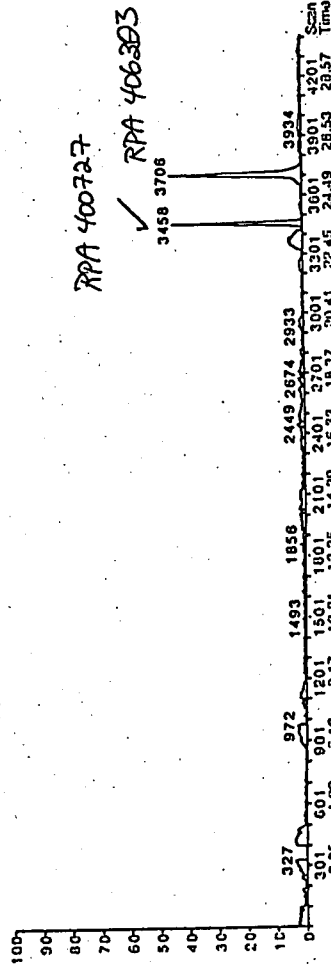
End Time 23.63

Integration Width 0.25

Retention Time 23.51

Integration Type A-BB

Intensity: 100000 cps



011 E7-11-412-05A(+3) Tue, Jan 20, 1998 21:22

No Comment

29.98 In 1 period

RPA 400727 (conf)

No Internal Standard

Use Area

1: 29.99 Q1 M1, 4411 scans

320.0

Noise Thres. 10.0

Quant Thres. 2.0

Min. Width 6

Mult. Width 5

Base. Width 100

RT Wrt. (secs) 20

Smooth 2

Expected RT 23.60

Area 6982

Height 4499

Start Time 23.60

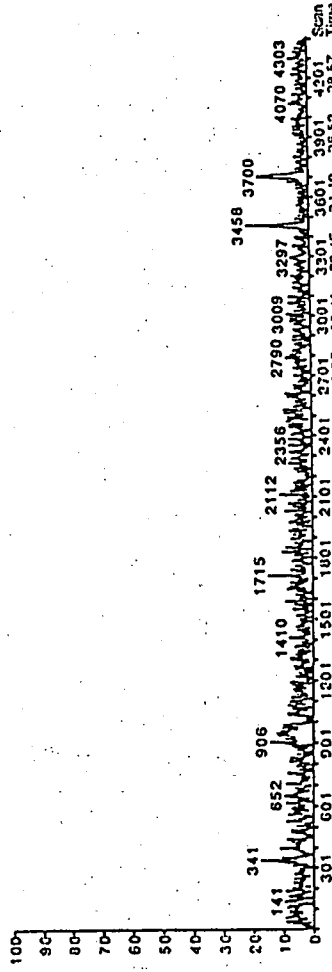
End Time 23.63

Integration Width 0.03

Retention Time 23.62

Integration Type A-W

Intensity: 100000 cps



NC Control Soil spiked at LOQ of 0.005 ppm (RPA 400727/RPA 406203)

Juan, version 1.5

Printed: Wed, Jan 21, 1998 10:11

Calibration File: NM012098Cal Path: Macintosh HD:AP1150EX #040:DATA:RHONPO:TRIT:NM012098:

Comments: No comments

012 RPA CAL Mix#2 0.025 ppm Tue, Jan 20, 1998 22:01

No Comment

29.98 In 1 period

RPA 406341

No Internal Standard

Use Area

1: 29.99 01 MI, 4411 scans

334.0 Noise Thres. 10.0

5.0 Guard Thres. 3

3 Min. Width 10

10 Mult. Width 100

100 Base. Width 2

2 RT Wn. (secs) 17.44

2 Smooth

Expected RT 17.44

Area 473385 ✓

Height 27539

Start Time 17.07

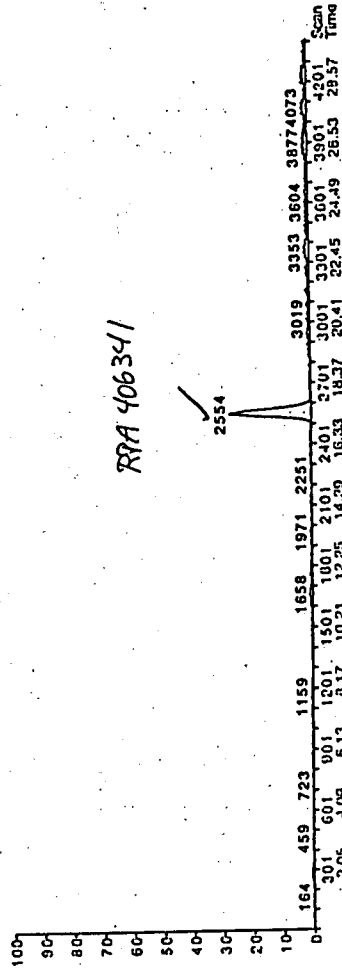
End Time 17.75

Integration Width 0.66

Retention Time 17.37

Integration Type A-BB

Intensity: 100000 cps



RPA 406341

012 RPA CAL Mix#2 0.025 ppm Tue, Jan 20, 1998 22:01

No Comment

29.98 In 1 period

RPA 406341 (cont)

No Internal Standard

Use Area

1: 29.99 01 MI, 4411 scans

336.0 Noise Thres. 10.0

2.0 Guard Thres. 6

6 Min. Width 5

5 Mult. Width 100

100 Base. Width 2

2 RT Wn. (secs) 17.44

2 Smooth

Expected RT 17.44

Area 99691

Height 9667

Start Time 17.37

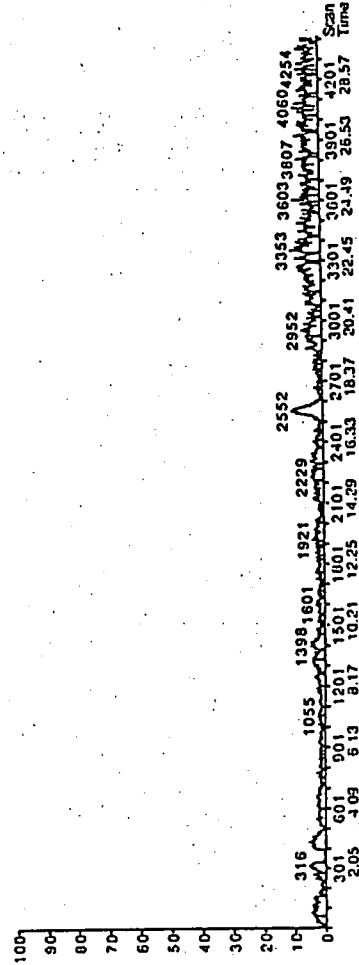
End Time 17.66

Integration Width 0.29

Retention Time 17.39

Integration Type A-W

Intensity: 100000 cps

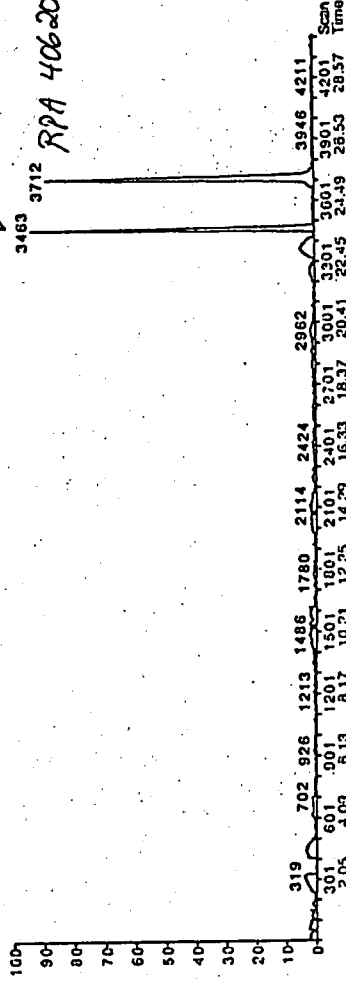


Standard at 0.025 ppm
(RPA 406341)

Juan, version 1.5
Printed: Wed, Jan 21, 1998 10:11
Calibration File: NM012098Cal Path: Macintosh HD:API150EX #040:DATA:RHONPO:TRIT:NM012098:
Comments: No comments

012 RPA CAL Mix#2 0.025 ppm Tue, Jan 20, 1998 22:01
No Comment

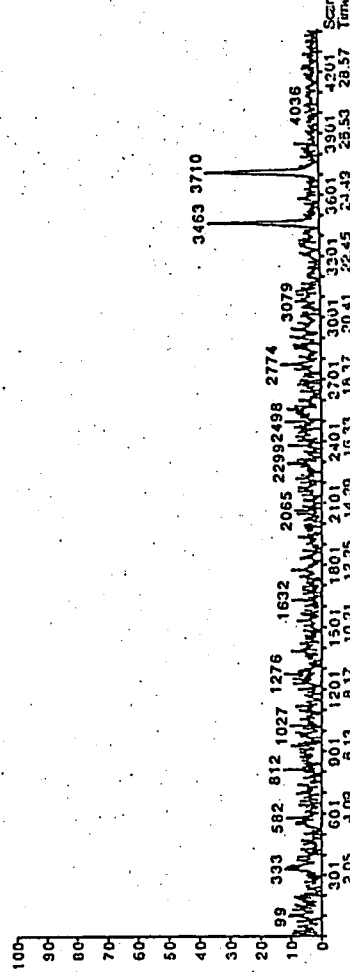
29.98 In 1 period
RPA 400727
No Internal Standard
Use Area
1: 29.99 01 MI, 4411 scans
Noise Thres. 10.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 5
Base. Width 100
RT Wtd. (secs) 20
Smooth 2
Expected RT 23.60
Area 582068
Height 93989
Start Time 23.38
End Time 23.68
Integration Width 0.31
Retention Time 23.55
Integration Type A - BB



RPA 400727
Intensity: 100000 cps
RPA 406203

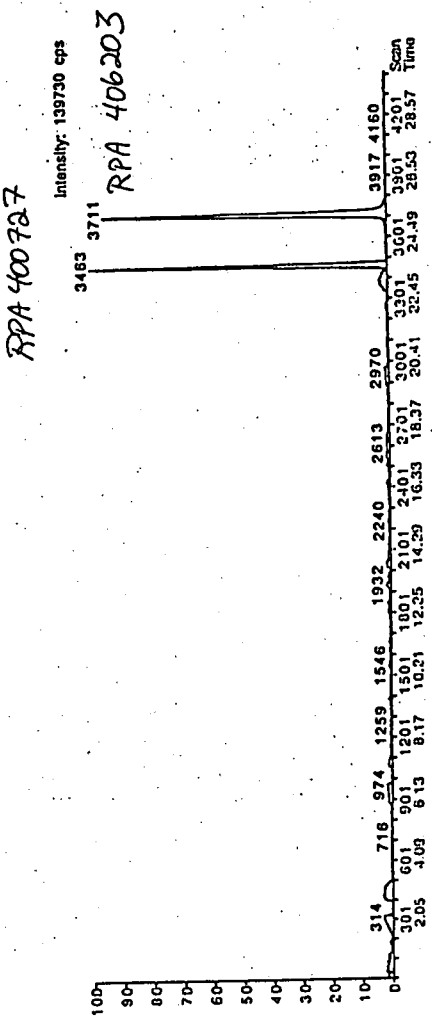
012 RPA CAL Mix#2 0.025 ppm Tue, Jan 20, 1998 22:01
No Comment

29.98 In 1 period
RPA 400727 (conf)
No Internal Standard
Use Area
1: 29.99 01 MI, 4411 scans
Noise Thres. 10.0
Quant Thres. 2.0
Min. Width 6
Mult. Width 5
Base. Width 100
RT Wtd. (secs) 20
Smooth 2
Expected RT 23.60
Area 284172
Height 36763
Start Time 23.39
End Time 23.69
Integration Width 0.31
Retention Time 23.55
Integration Type A - BV



Standard at 0.025 ppm
(RPA 400727/RPA 406203)

013 E7-11-412-05A(++1) Tue, Jan 20, 1998 22:40

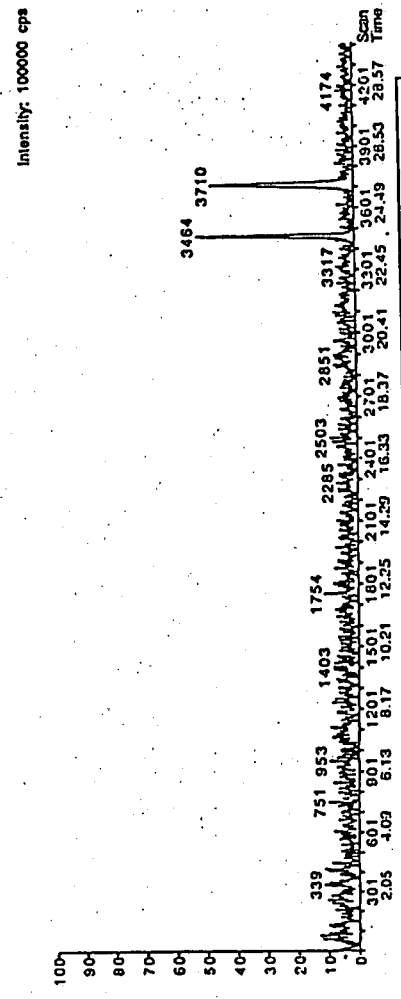


No Comment

29.99 In 1 period
 RPA 400727
 No Internal Standard
 Use Area

1: 29.99 Q1 Ml, 4411 scans
 Noise Thres. 10.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 5
 Base. Width 100
 RT Wn. (secs) 20
 Smooth 2
 Expected RT 23.60
 Area 880384
 Height 139730
 Start Time 23.39
 End Time 23.68
 Integration Width 0.30
 Retention Time 23.55
 Integration Type A-BB

013 E7-11-412-05A(++1) Tue, Jan 20, 1998 22:40



No Comment

29.99 In 1 period
 RPA 400727 (conf)
 No Internal Standard
 Use Area

1: 29.99 Q1 Ml, 4411 scans
 Noise Thres. 10.0
 Quant Thres. 2.0
 Min. Width 6
 Mult. Width 5
 Base. Width 100
 RT Wn. (secs) 20
 Smooth 2
 Expected RT 23.60
 Area 328957
 Height 52071
 Start Time 23.42
 End Time 23.66
 Integration Width 0.24
 Retention Time 23.56
 Integration Type A-VV

11.0 METHOD VALIDATION DATA FOR SOIL AND TURF

TABLE 1: DETERMINATION OF METHOD DETECTION LEVEL (MDL) AND LIMIT OF QUANTITATION (LOQ)
FOR RPA 400727, RPA 406341 AND RPA 406203 IN SOIL (LUCAMA, NORTH CAROLINA)

LAB SAMPLE #	ANALYSIS DATE (m/d/y)	SPIKE LEVEL (ppm)	RPA 400727 RESULT (ppm)	RPA 406341 RESULT (ppm)	RPA 406203 RESULT (ppm)
MDL Soil C-1	1/07/98	---	0	0	0
MDL Soil C-2	1/07/98	---	0	0	0
MDL Soil S+1	1/07/98	0.0025	0.0025	0.0037	0.0022
MDL Soil S+2	1/07/98	0.0025	0.0022	0.0034	0.0018
MDL Soil S+3	1/07/98	0.0025	0.0027	0.0033	0.0020
MDL Soil S+4	1/07/98	0.0025	0.0031	0.0033	0.0025
MDL Soil C-3	1/08/98	---	0.0005	0	0.0006
MDL Soil C-4	1/08/98	---	0.0005	0	0.0004
MDL Soil C-5	1/08/98	---	0.0004	0	0
MDL Soil S+5	1/08/98	0.0025	0.0022	0.0029	0.0018
MDL Soil S+6	1/08/98	0.0025	0.0029	0.0039	0.0024
MDL Soil S+7	1/08/98	0.0025	0.0025	0.0035	0.0028

RPA 400727 RPA 406341 RPA 406203
 Avg. Result - 0.0026 Avg. Result - 0.0034 Avg. Result - 0.0022
 SD - 0.00034 SD - 0.00032 SD - 0.00038

Average Result - 0.00028 Average Result - 0 Average Result - 0.0002

Untreated Controls (UTCs)

Calculations MDL and LOQ

MDL = (Standard deviation(SD) of 7 spikes in ppm x 3) + Average Result of 5 UTCs in ppm
 LOQ = (SD of 7 spikes in ppm x 10) + Average Result of 5 UTCs in ppm

RPA 400727 RPA 406341 RPA 406203
 MDL = 0.001 ppm MDL = 0.001 ppm MDL = 0.001 ppm
 LOQ = 0.003 ppm LOQ = 0.003 ppm LOQ = 0.004 ppm

TABLE 2: DETERMINATION OF METHOD DETECTION LIMIT (MDL) AND LIMIT OF QUANTITATION FOR RPA 400727, RPA 406341 AND RPA 406203 IN TURF (LUCAMA, NC)

LAB SAMPLE #	ANALYSIS DATE (m/d/y)	SPIKE LEVEL (ppm)	RPA 400727 RESULT (ppm)	RPA 406341 RESULT (ppm)	RPA 406203 RESULT (ppm)
MDL Turf C-1	1/07/98	---	0.0012	0	0
MDL Turf C-2	1/07/98	---	0.0011	0	0
MDL Turf S+1	1/07/98	0.0025	0.0025	0.0026	0.0023
MDL Turf S+2	1/07/98	0.0025	0.0029	0.0031	0.0029
MDL Turf S+3	1/07/98	0.0025	0.0032	0.0028	0.0028
MDL Turf S+4	1/07/98	0.0025	0.0022	0.0025	0.0023
MDL Turf C-3	1/08/98	---	0	0	0
MDL Turf C-4	1/08/98	---	0.0008	0	0
MDL Turf C-5	1/08/98	---	0	0	0
MDL Turf S+5	1/08/98	0.0025	0.0027	0.0026	0.0020
MDL Turf S+6	1/08/98	0.0025	0.0019	0.0022	0.0017
MDL Turf S+7	1/08/98	0.0025	0.0020	0.0023	0.0015
			RPA 400727	RPA 406341	RPA 406203
			Avg. Result - 0.0025	Avg. Result - 0.0026	Avg. Result - 0.0022
			SD - 0.00048	SD - 0.00030	SD - 0.00052
Untreated Controls (UTCs)			Average Result - 0.0006	Average Result - 0	Average Result - 0

Calculations MDL and LOQ

MDL = (Standard deviation(SD) of 7 spikes in ppm x 3) + Average Result of 5 UTCs in ppm
 LOQ = (SD of 7 spikes in ppm x 10) + Average Result of 5 UTCs in ppm

RPA 400727	RPA 406341	RPA 406203
MDL = 0.002 ppm	MDL = 0.001 ppm	MDL = 0.002 ppm
LOQ = 0.005 ppm	LOQ = 0.003 ppm	LOQ = 0.005 ppm

TABLE 3: LOQ (0.0050 ppm) RECOVERY DATA FOR RPA 400727, RPA 406341 AND RPA 406203 IN SOIL

LAB SAMPLE #	SAMPLE I.D.	ANALYSIS DATE (m/d/y)	SPIKE LEVEL (ppm)	RPA 400727		RPA 406341		RPA 406203	
				AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.
E7-11-412-16A-1	RS51048 0-18 SOIL	1/14/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-16A+2	RS51048 0-18 SOIL	1/14/98	0.0050	0.0043	86	0.0053	106	0.0049	98
E7-11-412-16A+3	RS51048 0-18 SOIL	1/14/98	0.0050	0.0039	78	0.0043	86	0.0044	88
E7-11-412-16A+1	RS51048 0-18 SOIL	1/20/98	0.0050	0.0036	72	0.0043	86	0.0039	78
E7-11-412-16A+4	RS51048 0-18 SOIL	1/20/98	0.0050	0.0037	74	0.0042	84	0.0035	70
E7-11-412-16A+5	RS51048 0-18 SOIL	1/20/98	0.0050	0.0038	76	0.0046	92	0.0046	92
				Mean Rec. - 77%	SD - 5.4%	Mean Rec. - 91%	SD - 9.0%	Mean Rec. - 85%	SD - 11%
				RSD - 7.0%		RSD - 9.9%		RSD - 13%	
E7-11-412-11A-1	RS51043 0-18 SOIL	1/14/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-11A-2	RS51043 0-18 SOIL	1/14/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-11A-3	RS51043 0-18 SOIL	1/14/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-11A+1	RS51043 0-18 SOIL	1/14/98	0.0050	0.0038	76	0.0048	96	0.0043	86
E7-11-412-11A+2	RS51043 0-18 SOIL	1/14/98	0.0050	0.0039	78	0.0053	106	0.0044	88
E7-11-412-11A+3	RS51043 0-18 SOIL	1/14/98	0.0050	0.0043	86	0.0056	112	0.0050	100
				Mean Rec. - 80%	SD - 5.3%	Mean Rec. - 105%	SD - 8.1%	Mean Rec. - 91%	SD - 7.6%
				RSD - 6.6%		RSD - 7.7%		RSD - 8.4%	
E7-11-412-05A-1	RS51037 0-18 SOIL	1/20/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-05A-2	RS51037 0-18 SOIL	1/20/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-05A-3	RS51037 0-18 SOIL	1/20/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-05A+1	RS51037 0-18 SOIL	1/20/98	0.0050	0.0053	106	0.0062	124	0.0055	110
E7-11-412-05A+2	RS51037 0-18 SOIL	1/20/98	0.0050	0.0043	86	0.0054	108	0.0051	102
E7-11-412-05A+3	RS51037 0-18 SOIL	1/20/98	0.0050	0.0048	96	0.0064	128	0.0053	106
				Mean Rec. - 96%	SD - 10%	Mean Rec. - 120%	SD - 11%	Mean Rec. - 106%	SD - 4.0%
				RSD - 10%		RSD - 8.8%		RSD - 3.8%	

TABLE 4: 5 X AND 10 X LOQ (0.025 ppm AND 0.050 ppm) RECOVERY DATA FOR RPA 400727, RPA 406341 AND RPA 406203 IN SOIL

LAB SAMPLE #	SAMPLE I.D.	ANALYSIS DATE (m/d/y)	SPIKE LEVEL (ppm)	RPA 400727		RPA 406341		RPA 406203	
				AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.
E7-11-412-16A-2	RS51048 0-18 SOIL	1/20/98	---	<0.0010	---	<0.0010	---	<0.0010	---
E7-11-412-16A++1	RS51048 0-18 SOIL	1/14/98	0.025	0.019	76	0.023	92	0.019	76
E7-11-412-16A++2	RS51048 0-18 SOIL	1/14/98	0.025	0.022	88	0.027	108	0.021	84
E7-11-412-16A++3	RS51048 0-18 SOIL	1/20/98	0.025	0.022	88	0.027	108	0.018	72
E7-11-412-16A++4	RS51048 0-18 SOIL	1/20/98	0.025	0.026	104	0.029	116	0.024	96
E7-11-412-16A++5	RS51048 0-18 SOIL	1/20/98	0.025	0.021	84	0.030	120	0.019	76
				Mean Rec. - 88%		Mean Rec. - 109%		Mean Rec. - 81%	
				SD - 10%		SD - 11%		SD - 9.5%	
				RSD - 11%		RSD - 10%		RSD - 12%	
E7-11-412-11A++1	RS51043 0-18 SOIL	1/14/98	0.050	0.040	80	0.047	94	0.043	86
E7-11-412-11A++2	RS51043 0-18 SOIL	1/14/98	0.050	0.043	86	0.049	98	0.044	88
E7-11-412-11A++3	RS51043 0-18 SOIL	1/14/98	0.050	0.032	64	0.044	88	0.033	66
				Mean Rec. - 77%		Mean Rec. - 93%		Mean Rec. - 80%	
				SD - 11%		SD - 5.0%		SD - 12%	
				RSD - 14%		RSD - 5.4%		RSD - 15%	
E7-11-412-05A(++1)	RS51037 0-18 SOIL	1/20/98	0.025	0.019	76	0.020	80	0.017	68
E7-11-412-05A(++2)	RS51037 0-18 SOIL	1/20/98	0.025	0.025	100	0.025	100	0.022	88
E7-11-412-05A(++3)	RS51037 0-18 SOIL	1/20/98	0.025	0.019	76	0.023	92	0.018	72
				Mean Rec. - 84%		Mean Rec. - 91%		Mean Rec. - 76%	
				SD - 14%		SD - 10%		SD - 11%	
				RSD - 17%		RSD - 11%		RSD - 14%	

TABLE 5: LOQ (0.0050 ppm) RECOVERY DATA FOR RPA 400727, RPA 406341 AND RPA 406203 IN TURF

LAB SAMPLE #	SAMPLE I.D.	ANALYSIS DATE (m/d/y)	SPIKE LEVEL (ppm)	RPA 400727		RPA 406341		RPA 406203	
				AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.
E7-11-412-17A-1	RS51049 0-18 TURF	3/04/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-17A+1	RS51049 0-18 TURF	3/04/98	0.0050	0.0034	68	0.0042	84	0.0036	72
E7-11-412-17A+2	RS51049 0-18 TURF	3/04/98	0.0050	0.0053	106	0.0046	92	0.0038	76
E7-11-412-17A+3	RS51049 0-18 TURF	3/04/98	0.0050	0.0045	90	0.0048	96	0.0043	86
E7-11-412-17A+4	RS51049 0-18 TURF	1/29/98	0.0050	0.0039	78	0.0037	74	0.0040	80
E7-11-412-17A+5	RS51049 0-18 TURF	1/29/98	0.0050	0.0040	80	0.0039	78	0.0039	78
				Mean Rec. - 84%		Mean Rec. - 85%		Mean Rec. - 78%	
				SD - 14%		SD - 9.2%		SD - 5.2%	
				RSD - 17%		RSD - 11%		RSD - 6.7%	
E7-11-412-18A-1	RS51054 0-18 TURF	1/27/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-18A-2	RS51054 0-18 TURF	1/27/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-18A-3	RS51054 0-18 TURF	1/27/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-18A+1	RS51054 0-18 TURF	1/27/98	0.0050	0.0042	84	0.0049	98	0.0034	68
E7-11-412-18A+2	RS51054 0-18 TURF	1/27/98	0.0050	0.0060	120	0.0054	108	0.0046	92
E7-11-412-18A+3	RS51054 0-18 TURF	1/27/98	0.0050	0.0051	102	0.0062	124	0.0047	94
				Mean Rec. - 102%		Mean Rec. - 110%		Mean Rec. - 85%	
				SD - 18%		SD - 13%		SD - 14%	
				RSD - 18%		RSD - 12%		RSD - 16%	
E7-11-412-06A-1	RS51038 0-18 TURF	1/22/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-06A-2	RS51038 0-18 TURF	1/22/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-06A-3	RS51038 0-18 TURF	1/22/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-06A+1	RS51038 0-18 TURF	1/22/98	0.0050	0.0041	82	0.0042	84	0.0030	60
E7-11-412-06A+2	RS51038 0-18 TURF	1/23/98	0.0050	0.0045	90	0.0050	100	0.0041	82
E7-11-412-06A+3	RS51038 0-18 TURF	1/23/98	0.0050	0.0041	82	0.0045	90	0.0042	84
				Mean Rec. - 85%		Mean Rec. - 91%		Mean Rec. - 75%	
				SD - 4.6%		SD - 8.1%		SD - 13%	
				RSD - 5.4%		RSD - 8.9%		RSD - 17%	

TABLE 6: 5 X AND 10 X LOQ (0.025 ppm AND 0.050 ppm) RECOVERY DATA FOR RPA 400727, RPA 406341 AND RPA 406203 IN TURF

LAB SAMPLE #	SAMPLE I.D.	ANALYSIS DATE (m/d/y)	SPIKE LEVEL (ppm)	RPA 400727		RPA 406341		RPA 406203	
				AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.	AMOUNT FOUND (ppm)	% REC.
E7-11-412-17A-2	RS51049 0-18 TURF	1/29/98	---	<0.001	---	<0.001	---	<0.001	---
E7-11-412-17A++1	RS51049 0-18 TURF	3/04/98	0.025	0.019	76	0.024	96	0.019	76
E7-11-412-17A++2	RS51049 0-18 TURF	3/04/98	0.025	0.021	84	0.022	88	0.018	72
E7-11-412-17A++3	RS51049 0-18 TURF	1/29/98	0.025	0.022	88	0.023	92	0.020	80
E7-11-412-17A++4	RS51049 0-18 TURF	1/29/98	0.025	0.022	88	0.021	84	0.019	76
E7-11-412-17A++5	RS51049 0-18 TURF	1/30/98	0.025	0.020	80	0.022	88	0.019	76
				Mean Rec. - 83%	Mean Rec. - 90%	Mean Rec. - 76%			
				SD - 5.2%	SD - 4.6%	SD - 2.8%			
				RSD - 6.3%	RSD - 5.1%	RSD - 3.7%			
<hr/>									
E7-11-412-18A++1	RS51054 0-18 TURF	1/27/98	0.050	0.034	68	0.046	92	0.034	68
E7-11-412-18A++2	RS51054 0-18 TURF	1/27/98	0.050	0.038	76	0.041	82	0.039	78
E7-11-412-18A++3	RS51054 0-18 TURF	1/27/98	0.050	0.044	88	0.051	102	0.044	88
				Mean Rec. - 77%	Mean Rec. - 92%	Mean Rec. - 78%			
				SD - 10%	SD - 10%	SD - 10%			
				RSD - 13%	RSD - 11%	RSD - 13%			
<hr/>									
E7-11-412-06A++1	RS51038 0-18 TURF	1/23/98	0.025	0.021	84	0.023	92	0.019	76
E7-11-412-06A++2	RS51038 0-18 TURF	1/23/98	0.025	0.018	72	0.022	88	0.018	72
E7-11-412-06A++3	RS51038 0-18 TURF	1/23/98	0.025	0.018	72	0.019	76	0.017	68
				Mean Rec. - 76%	Mean Rec. - 85%	Mean Rec. - 72%			
				SD - 6.9%	SD - 8.3%	SD - 4.0%			
				RSD - 9.1%	RSD - 9.8%	RSD - 5.6%			