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June 5, 2001

MEMORANDUM

DP Barcode: 262813

SUBJECT: Thiamethoxam Method Evaluation-Report No. ECM0171S1-S6

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The BEAD/Environmental Chemistry Lab has performed an Environmental Chemistry Method Evaluation (ECME) on Thiamethoxam in soil using the method, "Analytical Method For The Determination of CGA-293343 And Its Degradates CGA-322704, CGA-355190, CGA-353042, NOA-404617, And NOA-407475 In Soil By High Performance Liquid Chromatography With Mass Spectrometric Detection Including Validation Data".

The attached method evaluation report includes three parts:

Part I: Summary and Conclusions

In this section any problems encountered with the method and how they were handled are discussed. ECL's opinion of how well the method performed is also presented.

Part II: Analytical Results

In this section the individual results of each sample at each spiking level of each analyte is listed. The arithmetical means and descriptive statistics for each spiking level are also presented here.

Part III: Experimental Details

In this section any modification(s) that were made to the method, along with instrument parameters, spiking levels, example calculations, representative samples and standard chromatograms and standard curves are listed and/or discussed.

If you have questions concerning this report, please contact Charles Kennedy at (228) 688-2443 or Aubry Dupuy at (228) 688-3212.

cc: Christian Byrne, QA Officer
BEAD/ECL

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BEAD/ECL

Environmental Chemistry Method Evaluation Report

Analytical Method for The Determination of CGA-293343 and its Degradates CGA-322704, CGA-355190, CGA-353042, CGA-353042, NOA-404617, and NOA-407475 in Soil By High Performance Liquid Chromatography with Mass Spectrometric Detection Including Validation Data

Report Number ECM0171S1-S6

Final Report

Environmental Chemistry Laboratory
Biological and Economic Analysis Division

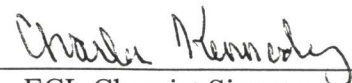
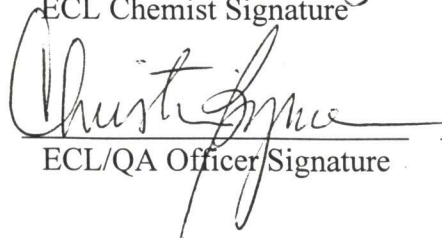
Prepared by: Charles Kennedy	 ECL Chemist Signature	<u>5/23/01</u> Date
Reviewed by: Christian Byrne	 ECL/QA Officer Signature	<u>5/29/01</u> Date

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Part 1
Summary and Conclusions

We have completed the Environmental Chemistry Method Evaluation (ECME) of "Analytical Method for the Determination of CGA-293343 and its Degradates CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 in Soil by High Performance Liquid Chromatography With Mass Spectrometric Detection Including Validation Data", MRID #449754-02. The method used to accomplish the analyses was submitted by Novartis Crop Protection, Inc. in support of registration of CGA-293343 (Thiamethoxam) and its degradates.

CGA-293343 and its degradates (CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475) were fortified in the original test soil twice at the 0.001 ppm (LOQ) and 0.010 ppm (10 x LOQ). We were unable to achieve all of target ranges for recovery (70-120%) and precision ($\leq 20\%$) of the compounds at 0.001 ppm (LOQ) as the registrant and independent laboratory. For this reason we think that this method may not be as easily employed to attain the specified reported LOQ. Discussion by one of the ECB chemists with a representative that performed the independent laboratory validation (ILV) confirmed difficulties with at least one of the degradates (CGA-353042). The results of the first fortification set at the LOQ are presented in the Analytical Results in Part 2. The recoveries for CGA-293343, CGA-322704, and CGA-353042 were low (i.e., 56.9, 53.5 and 30.3%), although the precision as measured by relative standard deviation (RSD), approximated the target range of less than 20%. The second fortification set at the LOQ resulted in higher recoveries for three of the compounds and lower for the other three, but with reduced precision.

We were able to achieve all of the target ranges for recovery (70-120%) and precision ($\leq 20\%$) of the compounds at 0.010 ppm (10 x LOQ) as the registrant and independent laboratory. The results of the second fortification set at 10 x LOQ are presented in the Analytical Results. The recoveries were satisfactory for all of the compounds with the exception of CGA-353042 (57.6%). However, all of the compounds exhibited increased recoveries during the second fortification set.

It is thought from these results that additional experience with the method would further improve the recovery and precision of the compounds at 0.010 ppm (10 x LOQ). However, there is inconsistency with the method at (LOQ); however, due to the complexity of the isolation procedures of the method, the number of analytes to be measured, and the difficulties at the limit of quantitation (LOQ), it might be expected that additional experience might greatly improve the recovery and precision at this limit.

Therefore, we think that this method can be used to quantitate CGA-293343 and its degradates in soil at or above 0.010 ppm (10 x LOQ) and monitor the presence of these compounds in soil at 0.001 ppm (LOQ).

Residues of CGA-293343 and its five degradates are extracted from the soil matrices by shaking with ammonium acetate/acetonitrile. After centrifuging and filtering, the sample is evaporated to 20-25 mL. The sample is then made basic and passed through an ENVI-Carb SPE cartridge attached on top of a C-18 cartridge. CGA-293343, CGA-322704, CGA-355190, NOA-404617, NOA-407475 and a large portion of CGS-353042 are retained on the ENVI-Carb SPE. The remaining CGA-353042 is retained on the lower C-18 cartridge. Analytes in the ENVI-Carb cartridge are eluted and collected. Water is added and the organic solvent is evaporated. The sample is then passed through the C-18 cartridge used previously. The eluate is collected along with the analytes that are eluted from the cartridge with 50% MeOH/0.1 N acetic acid. After evaporation to near dryness, the sample is reconstituted to a final concentration for analysis by LC/MS.

In order to evaluate this method we fortified a soil matrix with CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 at 0.0003 ppm (ECL's estimated MDL), 0.001 ppm (registrant's LOQ), and 0.010 ppm (10 x LOQ). All samples were extracted and analyzed in replicates of four at each fortification level. We found the precision to be well within our target limits of $\leq 20\%$ relative standard deviation (RSD) for CGA-293343 (the parent compound), CGA-322704, CGA-355190, and NOA-404617 at the LOQ. CGA-353042 (20.3%) and NOA-407475 (21.7%) were just slightly above the RSD ($\leq 20\%$) at the LOQ levels. At 10 x LOQ we found the precision to be within our target limits of $\leq 20\%$ RSD for CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475. The precision as measured by RSDs at the LOQ for CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 were 2.8, 17.7, 9.6, 20.3, 5.5, and 21.7%, respectively. At 10 x LOQ the RSDs for the compounds were 6.1, 12.0, 7.2, 7.8, 6.1 and 11.5% respectively.

The mean recoveries at 10 x LOQ for CGA-293343 (74.9%), CGA-322704 (74.7%), CGA-355190 (87.9%), NOA-404617 (95.2%) and NOA-407475 (97.4%) were within the target range of 70% to 120% with the exception of CGA-353042 (57.6%). Mean recoveries at the LOQ for the degradates CGA-355190 (72.7%), and NOA-404617 (85.8%), are all well within the target range. However, the recoveries of the parent CGA-293343 (56.9%), and the other degradates CGA-322704 (53.5%), CGA-353042 (30.3%), NOA-407475 (66.9%) were below the target range at the LOQ.

Part II

EPA Analytical Results

Results:

1. CGA-293343 (Parent Compound)

Recovery Values for Soil Fortified at 0.0003, 0.001, and 0.010 ppm in four replicates on
 Turbo Ionspray API 300 LC/MS/MS

(3) Fortified (ppm)	(4) Found (ppm)	(5) Recovery %	(7) SD	(8) RSD %
Matrix Blk (1)	—	—	—	--
Sample1-0.0003 Sample2-0.0003 Sample3-0.0003 Sample4-0.0003 (2)	0.000166 0.000104 0.000109 0.0001			
Sample#1-0.001 Sample#2-0.001 Sample#3-0.001 Sample#4-0.001 Mean Recovery(6)	0.000592 0.000569 0.000561 0.000555 0.000569	59.2 56.9 56.1 55.5 56.9	1.6	2.8
Sample#1-0.010 Sample#2-0.010 Sample#3-0.010 Sample#4-0.010 Mean Recovery	0.00727 0.00730 0.00722 0.00818 0.00749	72.7 73.0 72.2 81.8 74.9	4.6	6.1

Results:

2. CGA-322704 (Degradate)

Recovery Values for Soil Fortified at 0.0003, 0.001, and 0.010 ppm in four replicates on Turbo Ionspray API 300 LC/MS/MS

(3) Fortified (ppm)	(4) Found (ppm)	(5) Recovery (%)	(7) SD	(8) RSD %
Matrix Blk (1)	—	—	—	—
Sample1-0.0003 Sample2-0.0003 Sample3-0.0003 Sample4-0.0003 (2)	0.000182 0.000146 0.000163 0.000170			
Sample#1-0.001 Sample#2-0.001 Sample#3-0.001 Sample#4-0.001 Mean Recovery(6)	0.000614 0.000588 0.000401 0.000540 0.000535	61.4 58.8 40.1 54.0 53.5	9.5	17.7
Sample#1-0.010 Sample#2-0.010 Sample#3-0.010 Sample#4-0.010 Mean Recovery	0.00732 0.00647 0.00717 0.00874 0.00743	73.2 64.7 71.7 87.4 74.3	9.5	12.7

Results:

3. CGA-355190 (Degradate)

Recovery Values for Soil Fortified at 0.0003, 0.001, and 0.010 ppm in four replicates on
Turbo Ionspray API 300 LC/MS/MS

(3) Fortified (ppm)	(4) Found (ppm)	(5) Recovery %	(7) SD	(8) RSD %
Matrix Blk (1)	-	-	-	-
Sample1-0.0003 Sample2-0.0003 Sample3-0.0003 Sample4-0.0003 (2)	0.000302 0.000223 0.000250 0.000218			
Sample#1-0.001 Sample#2-0.001 Sample#3-0.001 Sample#4-0.001 Mean Recovery(6)	0.000706 0.000768 0.000637 0.000794 0.000727	70.6 76.8 63.7 79.4 72.7	7.0	9.6
Sample#1-0.010 Sample#2-0.010 Sample#3-0.010 Sample#4-0.010 Mean Recovery	0.00905 0.00798 0.00867 0.00946 0.00879	90.5 79.8 86.7 94.6 87.9	6.3	7.2

Results:

4. CGA-353042 (Degradate)

Recovery Values for Soil Fortified at 0.0003, 0.001, and 0.010 ppm in four replication on Turbo Ionspray API 300 LC/MS/MS

(3) Fortified (ppm)	(4) Found (ppm)	(5) Recovery %	(7) SD	(8) RSD %
Matrix Blk (1)	-	-	-	-
Sample1-0.0003 Sample2-0.0003 Sample3-0.0003 Sample4-0.0003 (2)	0.000072 0.000035 0.000038 0.0001			
Sample#1-0.001 Sample#2-0.001 Sample#3-0.001 Sample#4-0.001 Mean Recovery(6)	0.000386 0.000238 0.000287 0.000302 0.000303	38.6 23.8 28.7 30.2 30.3	6.2	20.3
Sample#1-0.010 Sample#2-0.010 Sample#3-0.010 Sample#4-0.010 Mean Recovery	0.00610 0.00551 0.00617 0.00525 0.00576	61.0 55.1 61.7 52.5 57.6	4.5	7.8

Results:

5. NOA-404617 (Degradate)

Recovery Values for Soil Fortified at 0.0003, 0.001, and 0.010 ppm in four replication on Turbo Ionspray API 300 LC/MS/MS

(3) Fortified (ppm)	(4) Found (ppm)	(5) Recovery %	(7) SD	(8) RSD %
Matrix Blk (1)	-	-	-	-
Sample1-0.0003 Sample2-0.0003 Sample3-0.0003 Sample4-0.0003 (2)	0.000303 0.000237 0.000250 0.000244			
Sample#1-0.001 Sample#2-0.001 Sample#3-0.001 Sample#4-0.001 Mean Recovery(6)	0.000892 0.000895 0.000851 0.000793 0.000858	89.2 89.5 85.1 79.3 85.8	4.8	5.5
Sample#1-0.010 Sample#2-0.010 Sample#3-0.010 Sample#4-0.010 Mean Recovery	0.00914 0.00919 0.00936 0.01038 0.00952	91.4 91.9 93.6 103.8 95.2	5.8	6.1

Results:

6. NOA-407475

Recovery Values for Soil Fortified at 0.0003, 0.001, and 0.010 ppm in four replication on Turbo Ionspray API 300 LC/MS/MS

(3) Fortified (ppm)	(4) Found (ppm)	(5) Recovery %	(7) SD	(8) RSD %
Matrix Blk (1)	-	-	-	-
Sample1-0.0003 Sample2-0.0003 Sample3-0.0003 Sample4-0.0003 (2)	0.000167 0.000155 0.000070 0.0001			
Sample#1-0.001 Sample#2-0.001 Sample#3-0.001 Sample#4-0.001 Mean Recovery(6)	0.000713 0.000466 0.000809 0.000690 0.000669	71.3 46.6 80.9 69.0 66.9	14.5	21.7
Sample#1-0.010 Sample#2-0.010 Sample#3-0.010 Sample#4-0.010 Mean Recovery	0.01121 0.00848 0.00964 0.00963 0.00974	112.1 84.8 96.4 96.3 97.4	11.2	11.5

Notes:

(1) Minimum Detection Level (MDL), equivalent to 0.0003 ppm in soil sample.

Limit of Quantitation (LOQ), equivalent to 0.001 ppm in soil sample.

(2) The four values (Sample#1, Sample#2, Sample#3, Sample#4) are replicate soil samples at each of three concentration levels of 0.0003, 0.001, and 0.010 ppm.

(3) Fortified (ppm) = CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 Fortification Levels.

(4) Found (ppm) = CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 Recovery Levels in Terms of Concentration.

(5) Recovery % = Percent Recovery of CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 as referred to in the Calculation Section.

(6) Mean Recovery = Average Recovery of Sample#1, Sample#2, Sample#3 and Sample#4.

(7) SD = Standard Deviation of % Recovery of Four Replicate Samples of CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475.

(8) RSD = Relative Standard Deviation of % Recovery of Four Replicate Samples of CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475.

Part III

Experimental DetailsGeneral description of method:

A 20 gram soil sample was weighted into a 250 mL centrifuge bottle. The sample was fortified with standards and left to equilibrate for 15-20 minutes. A volume of 100 mL of 20% 10mM ammonium acetate in acetonitrile was added to the centrifuge bottle and the sample was placed in a mechanical shaker for ~30 minutes. The sample was then centrifuged for 10 minutes to separate the supernatant. The supernatant was filtered through filter paper into a 500 mL round bottom flask. Another 100 mL of extraction solvent was added to the sample and was shaken another 30 minutes. After centrifuging as before, the supernatant was added through filter paper into the same round bottom flask. The extract in the round bottom flask was then evaporated to 20-25 mL on a rotary evaporator at ~40°C.

A 1 gram ENVI-Carb column was conditioned with 10 mL 0.2% formic acid in 20% methanol/dichloromethane, then 3 mL methanol, then 10 mL 2% acetic acid/water, then 24 mL water. All washes were discarded. The flow was stopped and 1 mL water was added to the top of the cartridge.

A 1 gram C18 SPE column was conditioned with 5 mL of methanol and then 5 mL water. All washes were discarded. Another 5 mL water was added and half was drained through and discarded. The flow was stopped and the conditioned ENVI-Carb was connected to the top using an adapter. Approximately 200 µl of ammonium hydroxide was added to the sample extract in the roundbottom flask and was poured into the connected columns and allowed to elute through at 1-2 drops/second. The eluate was discarded. The flask was rinsed with 10-12 mL water and the rinse was passed through the columns. The columns were further rinsed with 2 mL water. All rinses were discarded. The ENVI-Carb and C18 columns were disconnected.

Analytes were eluted from the ENVI-Carb with 3 mL of methanol followed by 24 mL 20% methanol in dichloromethane with 0.2% formic acid and collected in a 100 mL round bottom flask. Approximately 3 mL of water was added (as a keeper) and the organic solvent was evaporated to 2-3 mL on a rotary evaporator at ~40°C. This extract was then passed through the C18 SPE and the eluate was collected in a new 100 mL roundbottom flask. The old flask was rinsed with 12 ml 50% methanol in 0.1N acetic acid/water and sonicated. This rinse was passed through the C18 SPE and the eluate collected in the new 100 mL roundbottom flask. The extract was then evaporated to near dryness on a rotary evaporator at ~40°C. Four milliliters of 10% acetonitrile/water with 0.1% acetic acid was added to the nearly dry residue and sonicated. The final extract was transferred to a 15 mL vial and stored in a refrigerator until LC/MS/MS analysis.

The structural formula of CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475 are shown in Appendix A.

Modification to method:

Major Problem

Consistent recoveries values were a major problem in the evaluation. Various modifications were examined as they related to the extraction procedures. For example, the LC/MS chemist at ECB initially had problems with background interferences when performing data analysis. This problem was resolved by requesting soil used in the method by the registrant (Putnam County, Florida). Several other contacts had to be made with the registrant throughout the evaluation to clarify instrumental condition and recovery values stated in the method.

Due to the complexity of the extraction procedures of the method, another modification was saving the separate eluates in steps 2.12 thru 2.15 and 2.18; so they could be analyzed for CGA-322704 and its degradates. This resulted in some of the compounds of interests hanging up on the

C-18 column at step 2.18. It was decided to increase Step 2.18 to 24 mL of 50% MeOH/0.1N acetic acid instead of 12 mL suggested in the method. This 24 mL of 50% MeOH/0.1N acetic acid was then transferred into a C-18 column and the eluate collected in a 100 mL round bottom flask. It was found that a generally more consistent recovery of CGA-293343 (Thiamethoxam) and its degradates resulted due to this modification. This modification, however, did not account for acceptable recoveries for a number of the compounds at the LOQ. As a result of this modification, fortification and extraction of soil samples at the LOQ and 10 x LOQ levels were repeated.

Minor Problem

ECB found the analytical extraction portion of this method validation to be long and labor intensive. A stopping point, (overnight freezer storage) of fortified samples was not suggested in any of the steps of the method during the extraction of the soil. The registrant suggested that Step 2.7 would be the only place in the procedure at which extraction could be stopped. This resulted in extra time being needed to process a set of six samples through completion in a 9 hour day.

Sources of analytical reference standards:

CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617 and NOA-407475 analytical standards were obtained from Novartis Crop Protection, P.O. Box 18300, Greensboro, NC, 27419-8300. Telephone: (336)-632-6000. Fax (336) 632-6682

1. CGA-293343, Code # S96-1883, 98.9% purity, Expiration Date: 05/2001.
2. CGA-322704, Code # DAH-XXVII, 96.6% purity, Expiration Date: 09/30/01.
3. CGA-355190, Code # DAH-XXVI-63, 99.0% purity, Expiration Date: 02/28/01.
4. CGA-353042, Code # NEH-XVII-47, 98.4% purity, Expiration Date: 12/31/01.
5. NOA-404617, Code # DAH-XXV-19, 99.5% purity, Expiration Date: 12/31/01.
6. NOA-407475, Code # DAH-XXVI-77, 99.3% purity, Expiration Date: 02/28/01.

Source of sample matrix:

Some of the original soil used by Novartis (DAA 0-6") to validate this method was requested by ECB from the Novartis Crop Protection, Inc. Our purpose for requesting this soil was to eliminate background interference and to improve analytical recovery values for Thiamethoxam and its degradates. A copy of the characterization report is included in Appendix B.

Instrumentation for quantitation (listed only if different from that listed in method)

HPLC Pump Perkin Elmer Series 200
PE SCIEX Turbo Ionspray/API 300 LC/MS/MS System

HPLC Operating Conditions For CGA-353042 and NOA-407475

Column: Zorbax SCX, 5 μ m, 150 x 4.6 mm

Column Guard: Zorbax SCX Guard Column

Mobile Phase: 50% acetonitrile/25 mM Ammonium Acetate

Flow Rate: 1.5 mL/min (1/5 split)

Injection Vol: 20 μ l

Run Time: 12 min per sample

LC/MS/MS Operating Conditions:

Mode: Positive Ion
TurboIonSpray
Nebulizer gas 10 L/min
Curtain gas: 10 L/min
CAD gas: 3 L/min

Acquisition Parameters

Step size: 0.042 amu
Dwell: 0.5 sec
Scan speed: 1.01 sec
Pause: 2 msec
Centre/Width 115.8/86.0 AMU
247.0/160.8 AMU

ISV: 4000
TEM: 450
OR: 16
RNG: 80
Q0 -3.0
IQ1 -4.0
ST -9.0
RO1 -3.5
IQ2 -75
RO2 -80
IQ3 -95
RO3 -85
DF -200
CEM 2200

HPLC Operating Conditions For GCA-293343, CGA-322704, GGA-355190 and NOA-40617

Column:	Inertsil ODS-2, 5 μ m, 100 x 4.6 mm																								
Column Guard:	Inertsil ODS-2																								
Mobile Phase A:	Water with 0.1% acetic acid																								
Mobile Phase B:	0.1% acetic acid in acetonitrile																								
Gradient (A/B):	<table> <thead> <tr> <th><u>Time</u></th> <th><u>%A</u></th> <th><u>%B</u></th> <th><u>Curve</u></th> </tr> </thead> <tbody> <tr> <td>0</td> <td>90</td> <td>10</td> <td></td> </tr> <tr> <td>3</td> <td>30</td> <td>70</td> <td>1</td> </tr> <tr> <td>3</td> <td>30</td> <td>70</td> <td></td> </tr> <tr> <td>0.1</td> <td>90</td> <td>10</td> <td>0</td> </tr> <tr> <td>8</td> <td>90</td> <td>10</td> <td></td> </tr> </tbody> </table>	<u>Time</u>	<u>%A</u>	<u>%B</u>	<u>Curve</u>	0	90	10		3	30	70	1	3	30	70		0.1	90	10	0	8	90	10	
<u>Time</u>	<u>%A</u>	<u>%B</u>	<u>Curve</u>																						
0	90	10																							
3	30	70	1																						
3	30	70																							
0.1	90	10	0																						
8	90	10																							
Flow Rate:	1.5 mL/min (1/5 split)																								
Injection Vol:	20 μ l																								
Run Time:	15 min per sample																								

LC/MS/MS Operating Conditions

Acquisition Parameters

Mode:	Positive Ion TurboIonSpray
Nebulizer gas:	10 L/min
Curtain gas:	10 L/min
CAD gas:	3 L/min

Step size:	0.042 amu
Dwell:	0.25 sec
Scan speed:	1.01 sec
Pause:	2 msec
Centre/Width:	292.2/210.8 AMV 249.8/168.8 AMV 247.8/174.8 AMV 236.8/174.8 AMV

IS:	4000
TEM:	450
OR:	16
RNG:	80
Q0:	-3
IQ1:	-4
ST:	-9
R01:	-3.500
IQ2:	-75
R02:	-80
IQ3:	-95
R03:	-85
DF:	-200
CEM:	2200

Instrumentation for confirmation: Not applicable.

Relative retention parameters for the present evaluation:

Table 1

Analyte	Chemical Abstracts Registry No.	Retention Time (Minutes)
CGA-293343 (Parent)	S96-1883	5.22
CGA-322704 (Degradate)	DAH-XXVII-52	5.27
CGA-355190 (Degradate)	DAH-XXVI-63	5.28
CGA-353042 (Degradate)	NEH-XVII-47	4.33
NOA-404617 (Degradate)	DAH-XXV-19	6.37
NOA-407475 (Degradate)	DAH-XXVI-77	5.04

Notes on analytical procedures:

Extraction

The extraction procedure proceeded well for CGA-293343, CGA-322704, CGA-355190, CGA-353042, NOA-404617, and NOA-407475. Six samples could be extracted, cleaned-up and ready for LC/MS analysis in two 9 hour days.

Instrument Calibration

Along with the set of samples, calibration standards were analyzed over the range of 1.5 ng/mL to 100 ng/mL. A linear regression equation was generated by the MacQuan Version 1.5 software that accompanies the PE SCIEX API 300 LC/MS system on the calibration standards which were analyzed with each set of samples.

Calculation

The concentration of CGA-293343 and its degradates in the sample in ng/mL was determined by direct substitution of the sample peak area counts into the linear regression equation derived from the analysis of the calibration standards analyzed with the set of samples. This was done by the MacQuan Version 1.6 software that accompanies the PE SCIEX API 300 LC/MS.

To convert the concentration of the extract in ng/ml to ppm in the soil sample the following formula was used:

$$\frac{\text{Concentration (ng/mL)} \times \text{Final Volume (4 mL)} \times 1 \mu\text{g} \times \text{*Dilution Factor (5)}}{\text{Initial Weight of Soil (20 g)} \quad 1000 \text{ ng}} = \text{ppm} \quad (1)$$

*Dilution Factor used only at 10 x LOQ.

To calculate the percent recovery of the sample, the following formula was used:

$$\frac{\text{ppm of compound (found) in sample} \times 100}{\text{ppm of compound (fortified) in sample}} = \text{Percent Recovery.}$$

Example: Spike A Fortification of NOA-404617 at 0.001 ppm (LOQ)

$$\frac{4.4594 \text{ ng/mL} \times 4 \text{ mL} \times 1 \mu\text{g}}{20 \text{ g} \quad 1000 \text{ ng}} = 0.0008918 \text{ ppm}$$

$$\frac{0.000892 \text{ ppm} \times 100}{0.001 \text{ ppm}} = 89.2\%$$

Chromatograms and Calibration Curves:

- A. CGA-293343 Calibration Curve (used with 10 x LOQ level only)
- B. CGA-293343 Calibration Standards and Soil Sample Chromatograms
 - B-1. Calibration Standard 25 ng/mL
 - B-2. Calibration Standard 10 ng/ml
 - B-3. Calibration Standard 5 ng/mL
 - B-4. Calibration Standard 2.5 ng/mL
 - B-5. Soil Matrix Blank at MDL
 - B-6. Soil Sample Fortified at 0.0003 ppm (MDL)
 - B-7. Soil Matrix Blank at LOQ
 - B-8. Soil Sample Fortified at 0.001 ppm (LOQ)
 - B-9. Soil Matrix Blank at 10 x LOQ

- B-10. Soil Sample Fortified at 0.010 ppm (10 x LOQ)
- C. CGA-322704 Calibration Curve (used with 10 x LOQ level only)
- D. CGA-322704 Calibration Standards and Soil Chromatograms
 - C-1. Calibration Standard 25 ng/mL
 - C-2. Calibration Standard 10 ng/mL
 - C-3. Calibration Standard 5 ng/mL
 - C-4. Calibration Standard 2.5 ng/mL
 - C-5. Soil Matrix Blank at MDL
 - C-6. Soil Sample Fortified at 0.0003 ppm (MDL)
 - C-7. Soil Matrix Blank at LOQ
 - C-8. Soil Sample Fortified at 0.0001 ppm (LOQ)
 - C-9. Soil Matrix Blank at 10 x LOQ
 - C-10. Soil Sample Fortified at 0.010 ppm (10 x LOQ)
- D. CGA-355190 Calibration Curve (used with 10 x LOQ level only)
- E. CGA-355190 Calibration Standards and Soil Sample Chromatograms
 - E-1. Calibration Standard 25 ng/mL
 - E-2. Calibration Standard 10 ng/mL
 - E-3. Calibration Standard 5 ng/mL
 - E-4. Calibration Standard 2.5 ng/mL
 - E-5. Soil Matrix Blank at MDL
 - E-6. Soil Sample Fortified at 0.0003 ppm (MDL)

- E-7. Soil Matrix Blank at LOQ
 - E-8. Soil Sample Fortified at 0.001 ppm (LOQ)
 - E-9. Soil Matrix Blank at 10 x LOQ
 - E-10. Soil Sample Fortified at 0.010 (10 x LOQ)
- F. CGA-353042 Calibration Curve (used with 10 x LOQ level only)
- G. CGA-353042 Calibration Standards and Soil Sample Chromatograms
- G-1. Calibration Standard 25 ng/mL
 - G-2. Calibration Standard 10 ng/mL
 - G-3. Calibration Standard 5 ng/mL
 - G-4. Calibration Standard 2.5 ng/mL
 - G-5. Soil Matrix Blank at MDL
 - G-6. Soil Sample Fortified at 0.0003 ppm (MDL)
 - G-7. Soil Matrix Blank at LOQ
 - G-8. Soil Sample Fortified at 0.001 ppm (LOQ)
 - G-9. Soil Matrix Blank 10 x LOQ
 - G-10. Soil Sample Fortified at 0.010 ppm (10 x LOQ)
- H. NOA-404617 Calibration Curve (used with 10 x LOQ only)
- I. NOA-404617 Calibration Standards and Soil Sample Chromatograms
- I-1. Calibration Standard 25 ng/mL
 - I-2. Calibration Standard 10 ng/mL
 - I-3. Calibration Standard 5 ng/mL

- I-4. Calibration Standard 2.5 ng/mL
- I-5. Soil Matrix Blank at MDL
- I-6. Soil Sample Fortified at 0.0003 ppm (MDL)
- I-7. Soil Matrix Blank at LOQ
- I-8. Soil Sample Fortified at 0.001 ppm (LOQ)
- I-9. Soil Matrix Blank at 10 x LOQ
- I-10. Soil Sample Fortified at 0.010 ppm (10 x LOQ)
- J. NOA-407475 Calibration Curve (used with 10 x LOQ level only)
- K. NOA-407475 Calibration Standards and Soil Sample Chromatograms
 - K-1. Calibration Standard 25 ng/mL
 - K-2. Calibration Standard 10 ng/mL
 - K-3. Calibration Standard 5 ng/mL
 - K-4. Calibration Standard 2.5 ng/mL
 - K-5. Soil Matrix Blank at MDL
 - K-6. Soil Sample Fortified at 0.0003 ppm (MDL)
 - K-7. Soil Matrix Blank at LOQ
 - K-8. Soil Sample Fortified at 0.001 ppm (LOQ)
 - K-9. Soil Matrix Blank at 10 x LOQ
 - K-10. Soil Sample Fortified at 0.010 (10 x LOQ)

A. CGA-293343 Calibration Curve (used with 10 X LOQ level only)

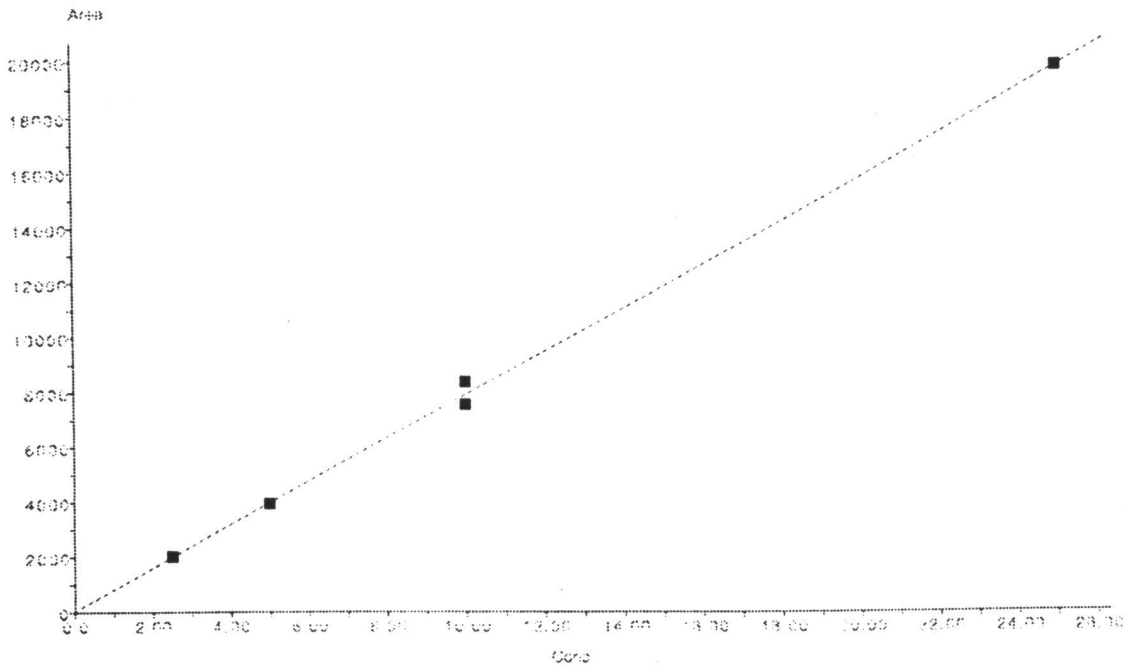
CGA293343 292.2->210.8 No Internal Standard

Linear

Intercept = 32.313

Slope = 787.305

Correlation Coeff. = 0.99911



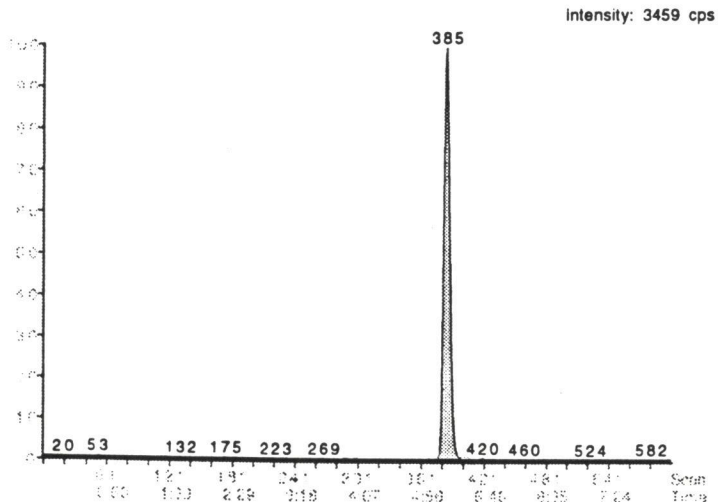
B. CGA-293343 Calibration Standards and Soil Sample Chromatograms

09250003 No Samplename 12:23
- User: DDM - Compound: Q1

B-1. 20 µl @ 25 ng/mL

8:12 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 1.6
Quant Thres. 0.8
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:21
Area 19695
Height 3447
Start Time 5:06
End Time 5:30
Integration Width 0:23.8
Retention Time 5:16
Integration Type A - BV

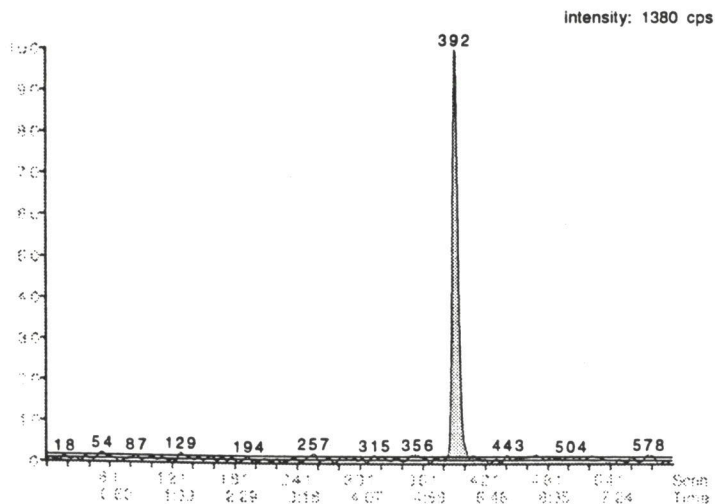


09250002 No Samplename 12:08
- User: DDM - Compound: Q1

B-2. 20 µl @ 10 ng/ml

8:12 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 1.6
Quant Thres. 0.8
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:21
Area 7537
Height 1389
Start Time 5:12
End Time 5:33
Integration Width 0:20.5
Retention Time 5:22
Integration Type A - BV

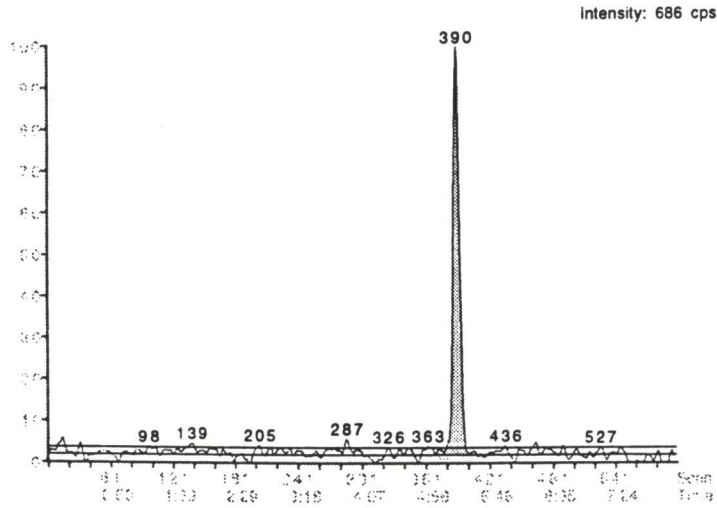


09250005 No Samplename 13:12
- User: DDM - Compound: Q1

B-3. 20 µl @ 5 ng/mL

8:12 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 1.6
Quant Thres. 0.8
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:21
Area 3915
Height 674
Start Time 5:10
End Time 5:28
Integration Width 0:18.1
Retention Time 5:20
Integration Type A - VB

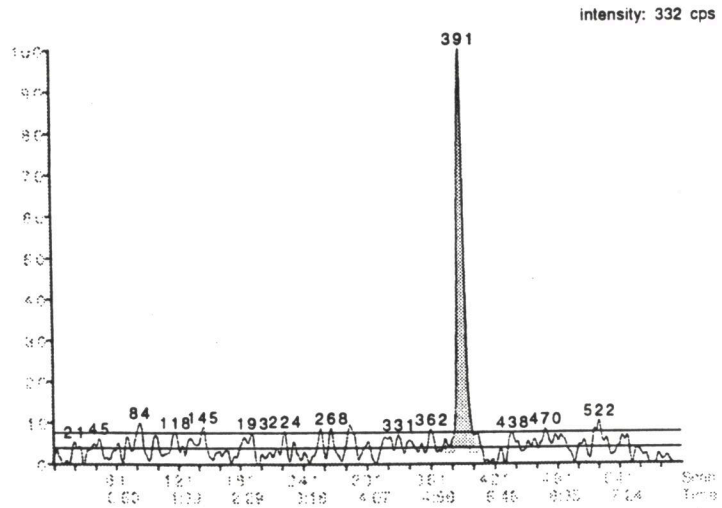


09250006 No Samplename 13:27
- User: DDM - Compound: Q1

B-4. 20 µl @ 2.5 ng/mL

8:12 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 1.6
Quant Thres. 0.8
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:21
Area 1997
Height 320
Start Time 5:11
End Time 5:29
Integration Width 0:18.1
Retention Time 5:21
Integration Type A - VV

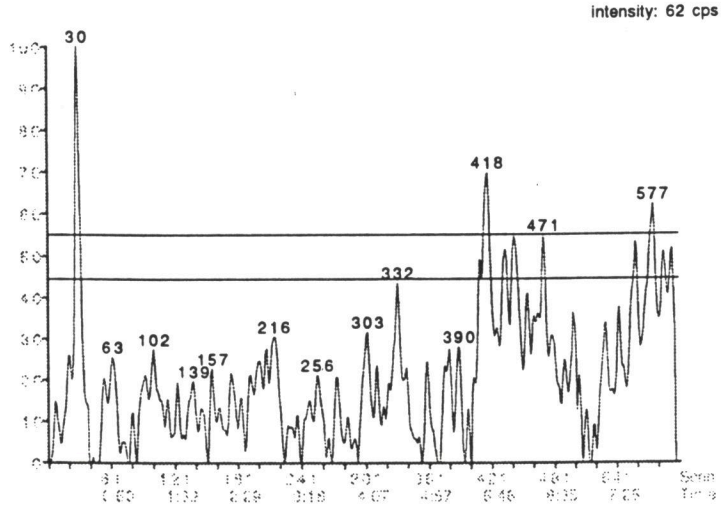


10100024 No Samplename 15:04
- User: DDM - Compound: Q1

B-5. Soil Matrix Blank at MDL

8:13 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 2.6
Quant Thres. 2.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:19
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

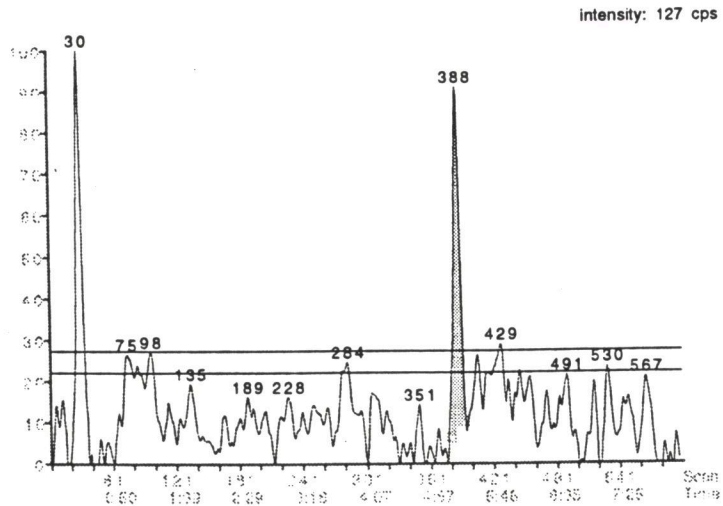


10100027 No Samplename 15:49
- User: DDM - Compound: Q1

B-6. Soil Sample Fortified at 0.0003 ppm (MDL)

8:13 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 2.6
Quant Thres. 2.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:19
Area 611
Height 104
Start Time 5:13
End Time 5:23
Integration Width 0:09.9
Retention Time 5:19
Integration Type M

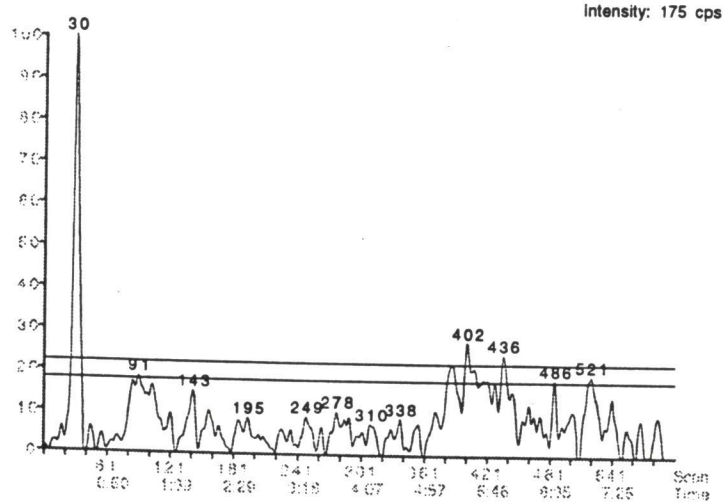


10100025 No Samplename 15:18
- User: DDM - Compound: Q1

B-7. Soil Matrix Blank at LOQ

8:13 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 2.6
Quant Thres. 2.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:19
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

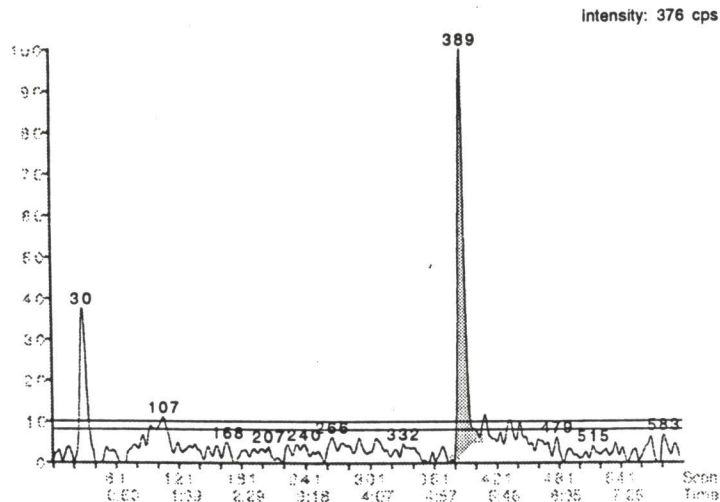


10100020 No Samplename 14:04
- User: DDM - Compound: Q1

B-8. Soil Sample Fortified at 0.001 ppm (LOQ)

8:13 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 2.6
Quant Thres. 2.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:19
Area 2086
Height 366
Start Time 5:13
End Time 5:30
Integration Width 0:17.3
Retention Time 5:20
Integration Type M

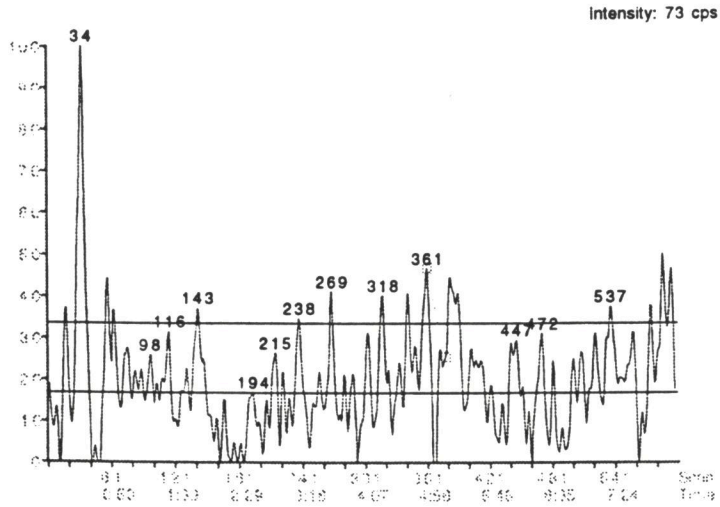


09250010 No Samplename 14:29
- User: DDM - Compound: Q1

B-9. Soil Matrix Blank at 10 X LOQ

8:12 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 1.6
Quant Thres. 0.8
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:21
Area 0
Height 0
Start Time 4:56
End Time 5:10
Integration Width 0:14.0
Retention Time 4:56
Integration Type M

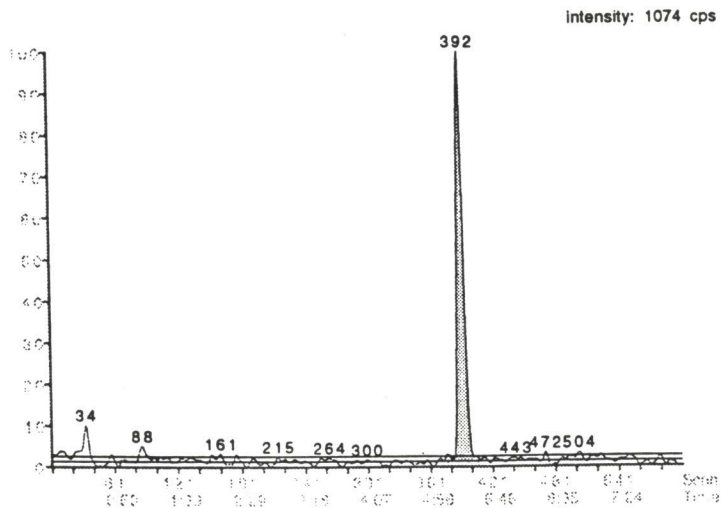


09250009 No Samplename 14:14
- User: DDM - Compound: Q1

B-10. Soil Sample Fortified at 0.010 ppm (10 X LOQ)

8:12 in 1 period
CGA293343
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
292.2->210.8
Noise Thres. 1.6
Quant Thres. 0.8
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:21
Area 6472
Height 1061
Start Time 5:14
End Time 5:30
Integration Width 0:15.6
Retention Time 5:22
Integration Type A - VV

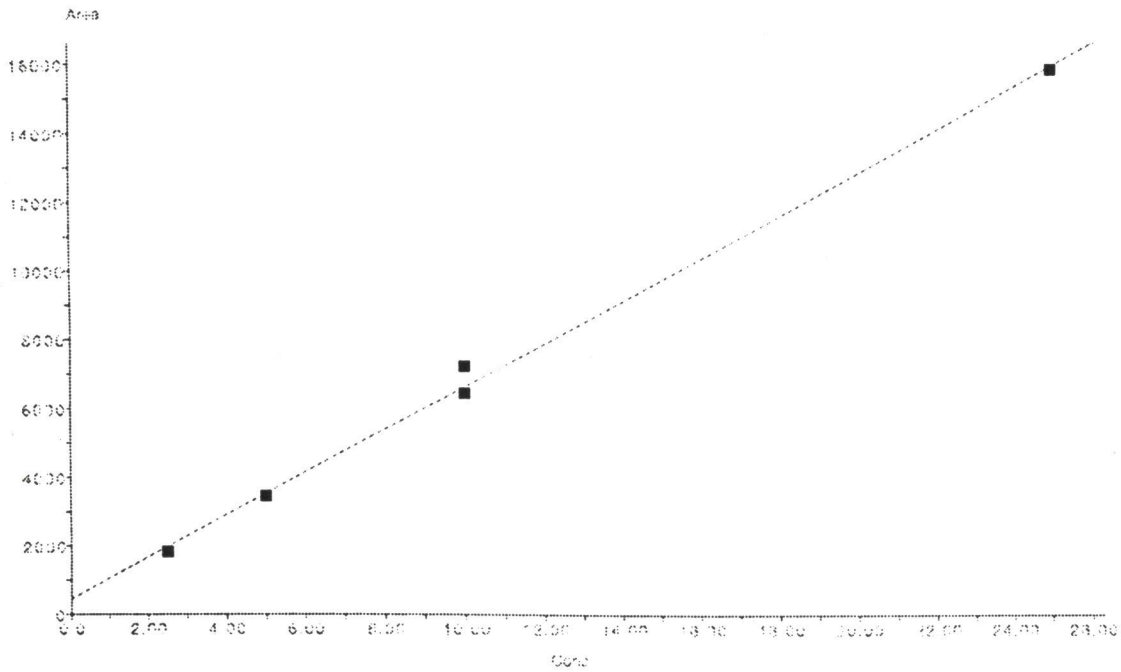


CGA322704 249.8->168.8 No Internal Standard

Linear

Intercept = 434.831
Slope = 619.404
Correlation Coeff. = 0.99818

C. CGA-322704 Calibration Curve (used with 10 X LOQ level only)



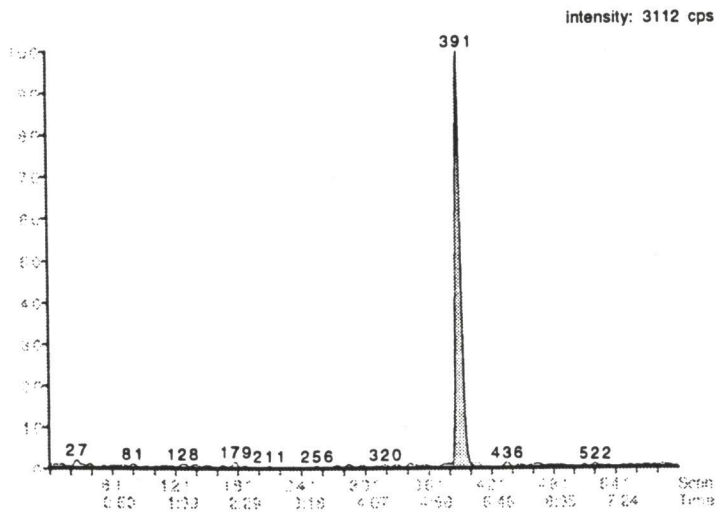
D. CGA-322704 Calibration Standards and Soil Chromatograms

09250003 No Samplename 12:23
- User: DDM - Compound: Q1

C-1. 20 µl @ 25 ng/mL

8:12 in 1 period
CGA322704
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 15804
Height 3109
Start Time 5:15
End Time 5:33
Integration Width 0:18.1
Retention Time 5:21
Integration Type A - VV

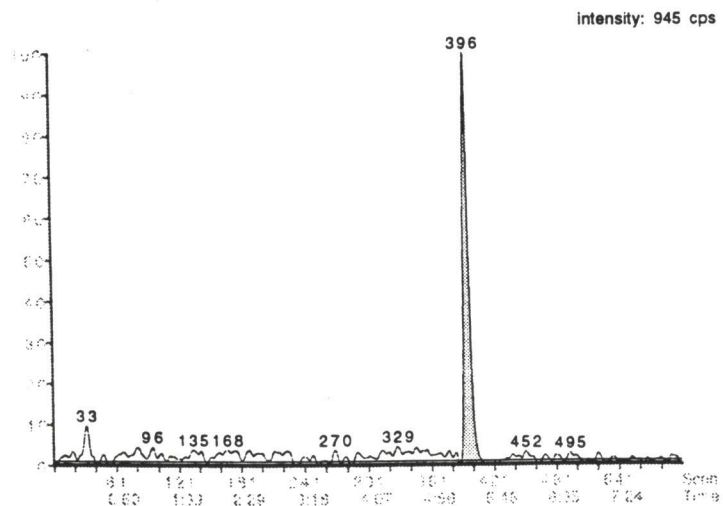


09250004 No Samplename 12:57
- User: DDM - Compound: Q1

C-2. 20 µl @ 10 ng/mL

8:12 in 1 period
CGA322704
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 4969
Height 945
Start Time 5:19
End Time 5:37
Integration Width 0:17.2
Retention Time 5:25
Integration Type A - BB

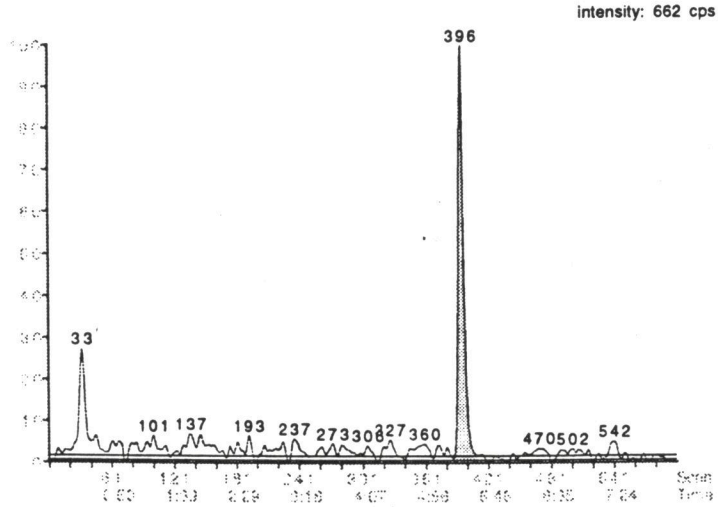


09250005 No Samplename 13:12
- User: DDM - Compound: Q1

C-3. 20 µl @ 5 ng/mL

8:12 In 1 period
CGA322704
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 3438
Height 659
Start Time 5:19
End Time 5:37
Integration Width 0:18.1
Retention Time 5:25
Integration Type A - VV

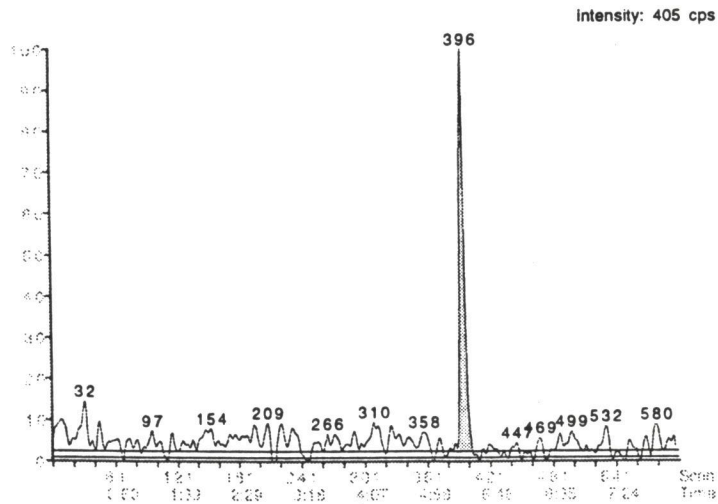


09250006 No Samplename 13:27
- User: DDM - Compound: Q1

C-4. 20 µl @ 2.5 ng/mL

8:12 In 1 period
CGA322704
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 1815
Height 395
Start Time 5:20
End Time 5:33
Integration Width 0:12.3
Retention Time 5:25
Integration Type A - VV



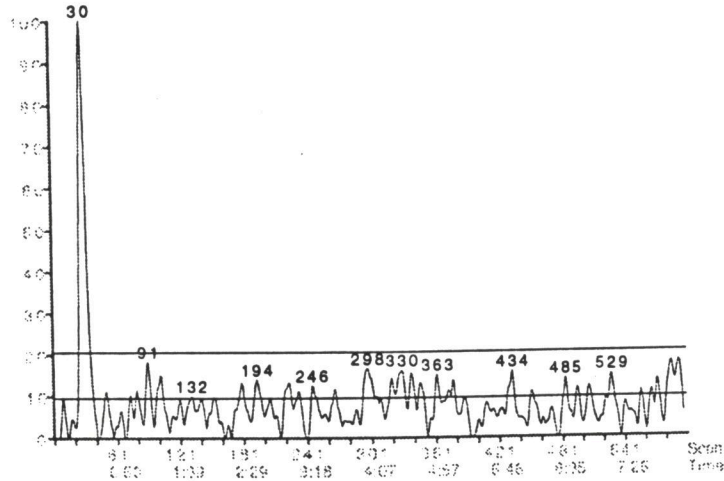
10100024 No Samplename 15:04
- User: DDM - Compound: Q1

C-5. Soil Matrix Blank at MDL

8:13 in 1 period
CGA322704
No Internal Standard
Use Area

Intensity: 183 cps

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 2.6
Quant Thres. 1.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:24
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type



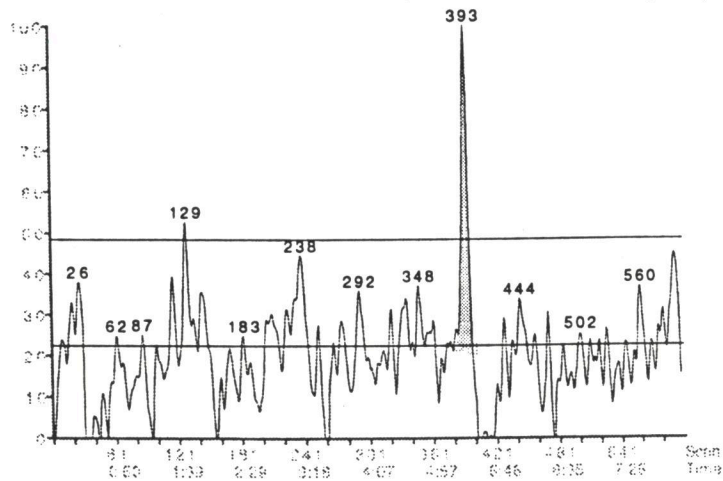
10100027 No Samplename 15:49
- User: DDM - Compound: Q1

C-6. Soil Sample Fortified at 0.0003 ppm (MDL)

8:13 in 1 period
CGA322704
No Internal Standard
Use Area

Intensity: 83 cps

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 2.6
Quant Thres. 1.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:24
Area 346
Height 65
Start Time 5:16
End Time 5:27
Integration Width 0:10.7
Retention Time 5:23
Integration Type A - VB



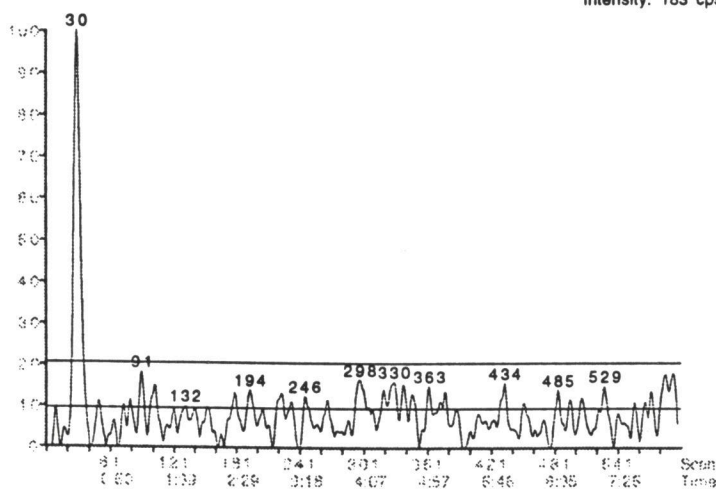
10100024 No Samplename 15:04
 - User: DDM - Compound: Q1

C-7. Soil Matrix Blank at LOQ

8:13 in 1 period
 CGA322704
 No Internal Standard
 Use Area

1: 8:12 MRM, 600 scans
 249.8->168.8
 Noise Thres. 2.6
 Quant Thres. 1.2
 Min. Width 6
 Mult. Width 2
 Base. Width 100
 RT Win. (secs) 20
 Smooth 5
 Expected RT 5:24
 Area 0
 Height 0
 Start Time 0:00.0
 End Time 0:00.0
 Integration Width 0:00.0
 Retention Time 0:00.0
 Integration Type

Intensity: 183 cps



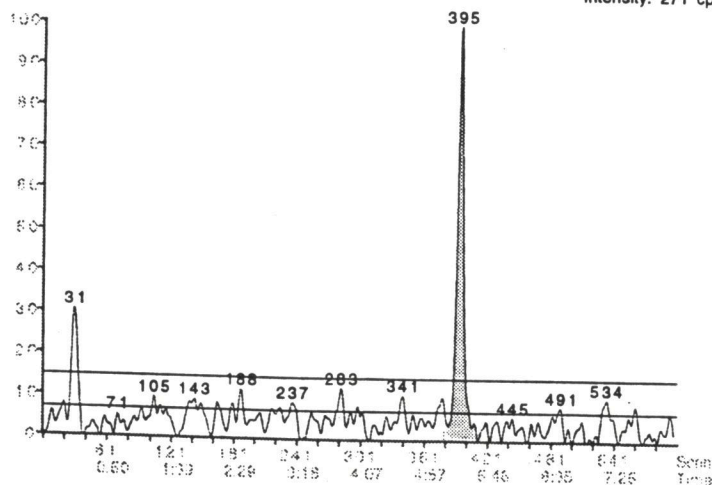
10100020 No Samplename 14:04
 - User: DDM - Compound: Q1

C-8. Soil Sample Fortified at 0.0001 ppm (LOQ)

8:13 in 1 period
 CGA322704
 No Internal Standard
 Use Area

1: 8:12 MRM, 600 scans
 249.8->168.8
 Noise Thres. 2.6
 Quant Thres. 1.2
 Min. Width 6
 Mult. Width 2
 Base. Width 100
 RT Win. (secs) 20
 Smooth 5
 Expected RT 5:24
 Area 1633
 Height 269
 Start Time 5:16
 End Time 5:37
 Integration Width 0:21.4
 Retention Time 5:25
 Integration Type M

Intensity: 271 cps

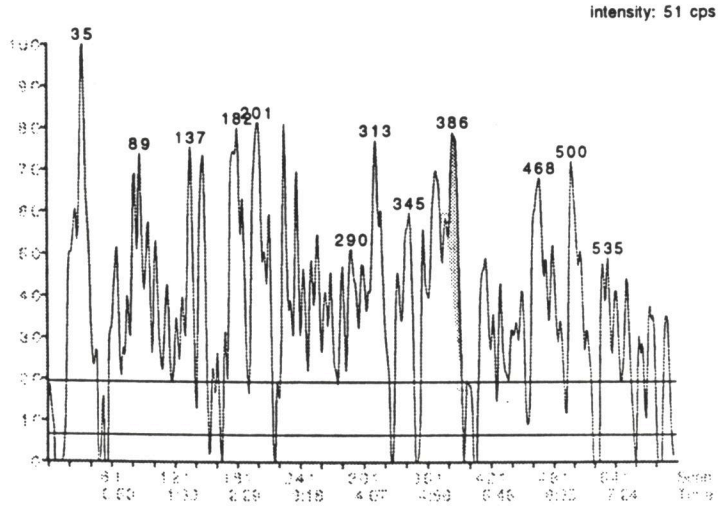


09250010 No Samplename 14:29
- User: DDM - Compound: Q1

C-9. Soil Matrix Blank at 10 X LOQ

8:12 in 1 period
CGA322704
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 151
Height 24
Start Time 5:11
End Time 5:23
Integration Width 0:11.5
Retention Time 5:19
Integration Type M

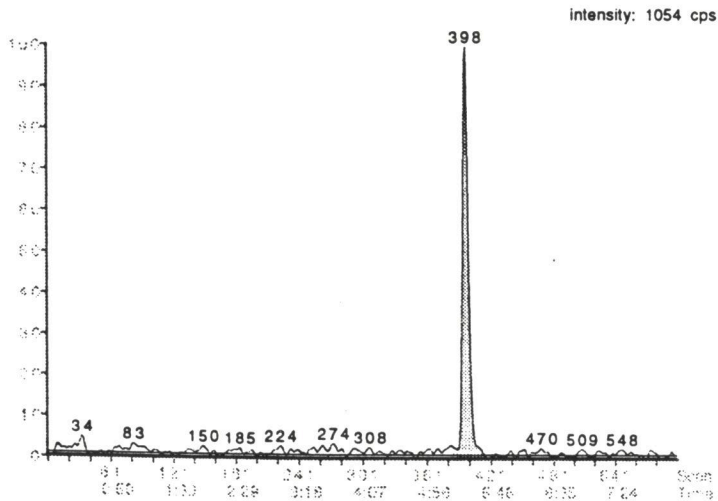


09250009 No Samplename 14:14
- User: DDM - Compound: Q1

C-10. Soil Sample Fortified at 0.010 ppm (10 X LOQ)

8:12 in 1 period
CGA322704
No Internal Standard
Use Area

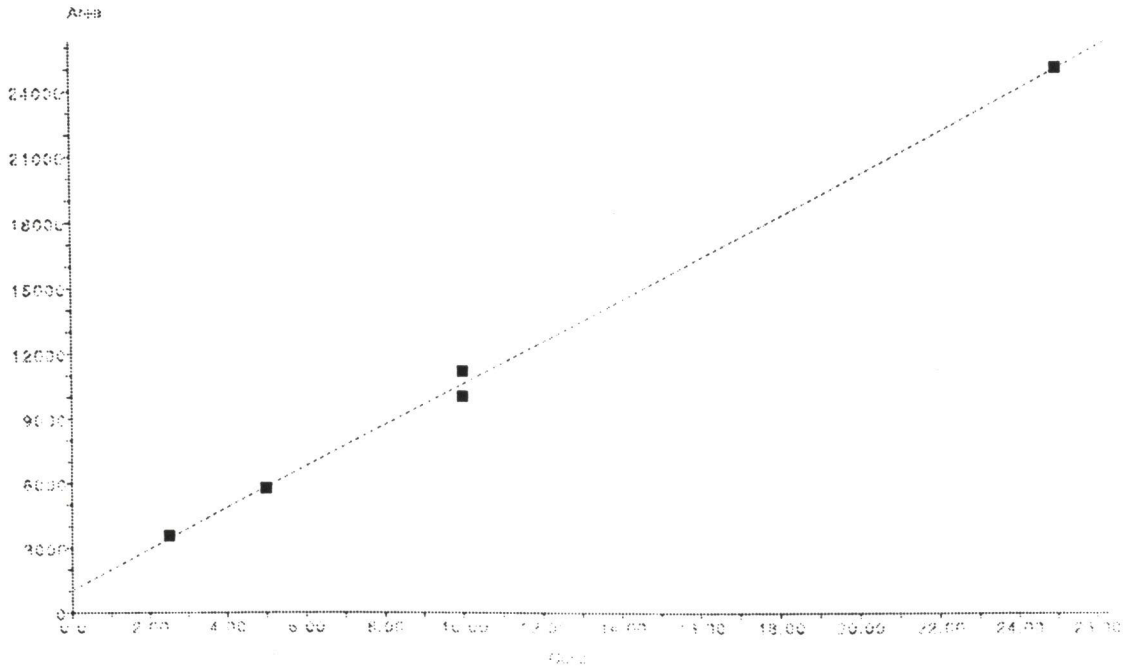
1: 8:12 MRM, 600 scans
249.8->168.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 5849
Height 1051
Start Time 5:20
End Time 5:42
Integration Width 0:22.2
Retention Time 5:27
Integration Type A - VB



CGA355190 247.8->174.8 No Internal Standard
Linear

Intercept = 1040.008
Slope = 957.802
Correlation Coeff. = 0.99876

D. CGA-355190 Calibration Curve (used with 10 X LOQ level only)



E. CGA-355190 Calibration Standards and Soil Sample Chromatograms

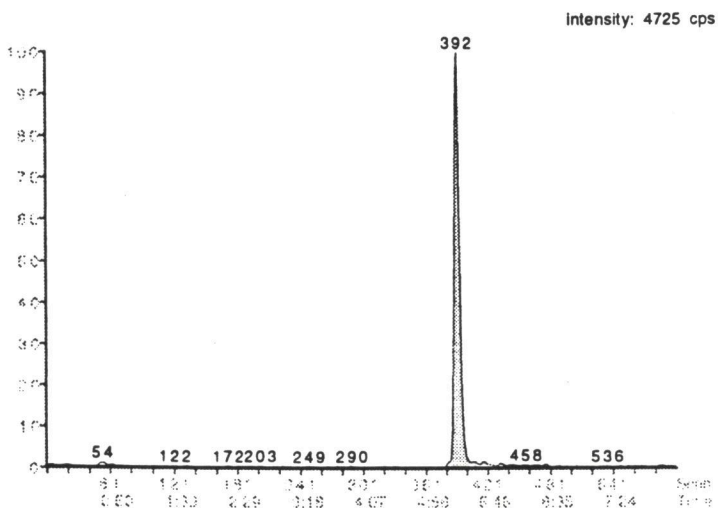
09250003 No Samplename 12:23
- User: DDM - Compound: Q1

E-1. 20 µl @ 25 ng/mL

8:12 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 1.0
Quant Thres. 0.9
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:26

Area 25006
Height 4719
Start Time 5:12
End Time 5:32
Integration Width 0:19.7
Retention Time 5:22
Integration Type A - BV



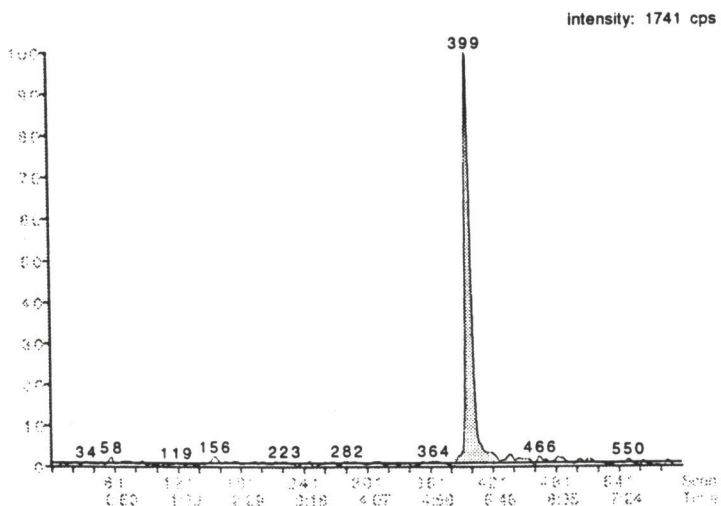
09250002 No Samplename 12:08
- User: DDM - Compound: Q1

E-2. 20 µl @ 10 ng/mL

8:12 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 1.0
Quant Thres. 0.9
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:26

Area 10022
Height 1729
Start Time 5:16
End Time 5:42
Integration Width 0:26.3
Retention Time 5:28
Integration Type A - BV

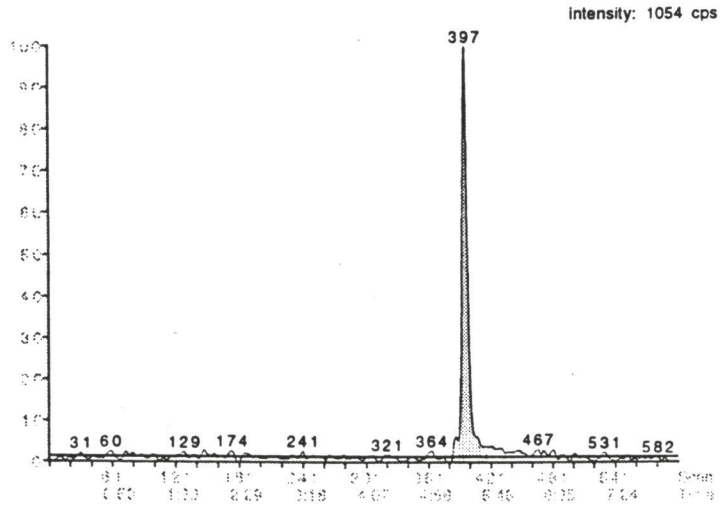


09250005 No Samplename 13:12
- User: DDM - Compound: Q1

E-3. 20 µl @ 5 ng/mL

8:12 In 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 1.0
Quant Thres. 0.9
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 2.0
Smooth 5
Expected RT 5:26
Area 5735
Height 1042
Start Time 5:20
End Time 5:39
Integration Width 0:18.9
Retention Time 5:26
Integration Type A - VV

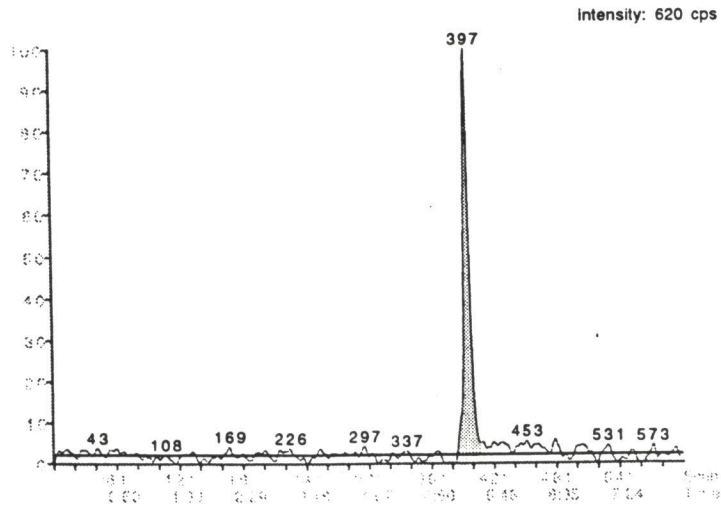


09250006 No Samplename 13:27
- User: DDM - Compound: Q1

E-4. 20 µl @ 2.5 ng/mL

8:12 In 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 1.0
Quant Thres. 0.9
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 2.0
Smooth 5
Expected RT 5:26
Area 3540
Height 614
Start Time 5:16
End Time 5:34
Integration Width 0:18.1
Retention Time 5:26
Integration Type A - BV



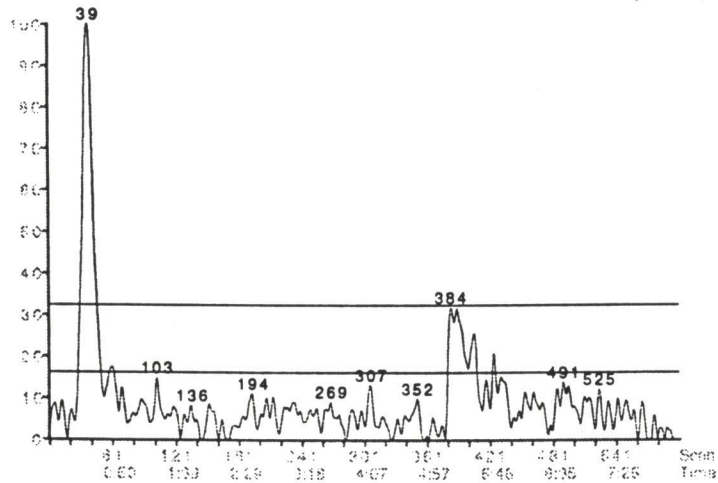
10100024 No Samplename 15:04
- User: DDM - Compound: Q1

E-5. Soil Matrix Blank at MDL

8:13 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 2.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

intensity: 112 cps



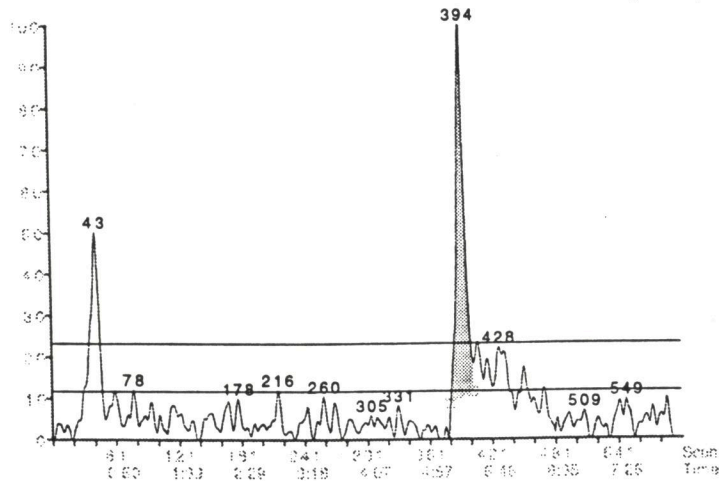
10100032 No Samplename 17:05
- User: DDM - Compound: Q1

E-6. Soil Sample Fortified at 0.0003 ppm (MDL)

8:13 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 2.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:25
Area 974
Height 134
Start Time 5:14
End Time 5:31
Integration Width 0:17.3
Retention Time 5:24
Integration Type A - BV

intensity: 149 cps



10100024 No Samplename 15:04
- User: DDM - Compound: Q1

E-7. Soil Matrix Blank at LOQ

8:13 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans

247.8->174.8

Noise Thres. 2.6

Quant Thres. 1.3

Min. Width 6

Mult. Width 2

Base. Width 100

RT Win. (secs) 20

Smooth 5

Expected RT 5:25

Area 0

Height 0

Start Time 0:00.0

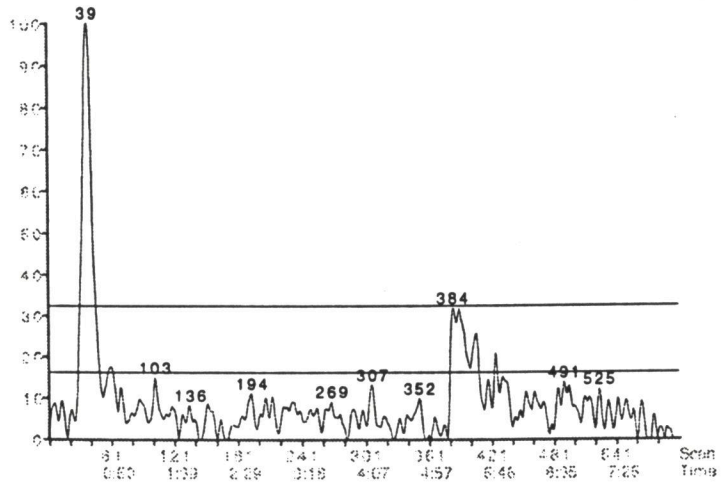
End Time 0:00.0

Integration Width 0:00.0

Retention Time 0:00.0

Integration Type

Intensity: 112 cps



10100020 No Samplename 14:04
- User: DDM - Compound: Q1

E-8. Soil Sample Fortified at 0.001 ppm (LOQ)

8:13 in 1 period

CGA355190

No Internal Standard

Use Area

1: 8:12 MRM, 600 scans

247.8->174.8

Noise Thres. 2.6

Quant Thres. 1.3

Min. Width 6

Mult. Width 2

Base. Width 100

RT Win. (secs) 20

Smooth 5

Expected RT 5:25

Area 2687

Height 386

Start Time 5:12

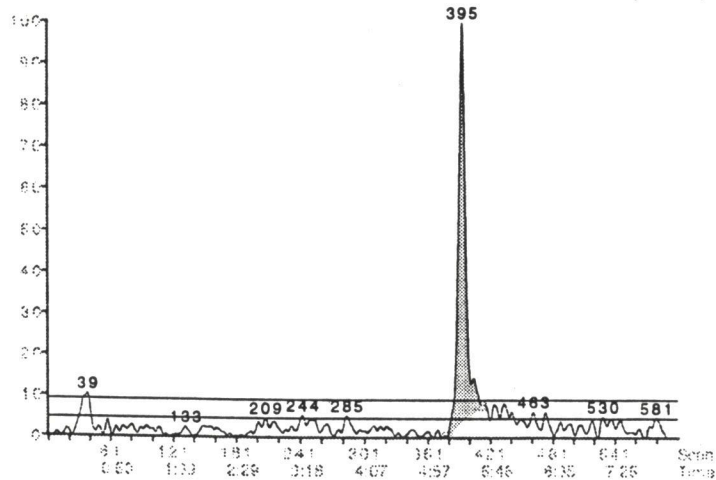
End Time 5:39

Integration Width 0:27.1

Retention Time 5:25

Integration Type M

Intensity: 401 cps



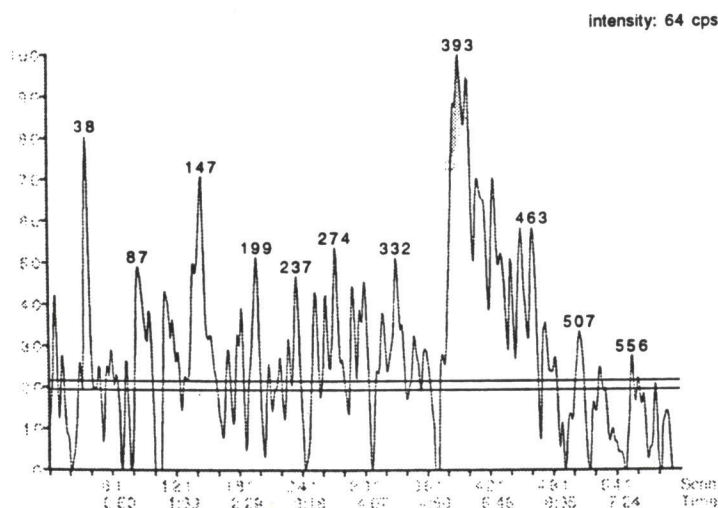
09250010 No Samplename 14:29
- User: DDM - Compound: Q1

E-9. Soil Matrix Blank at 10 X LOQ

8:12 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 1.0
Quant Thres. 0.9
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:26

Area 73
Height 13
Start Time 5:17
End Time 5:27
Integration Width 0:09.9
Retention Time 5:23
Integration Type M



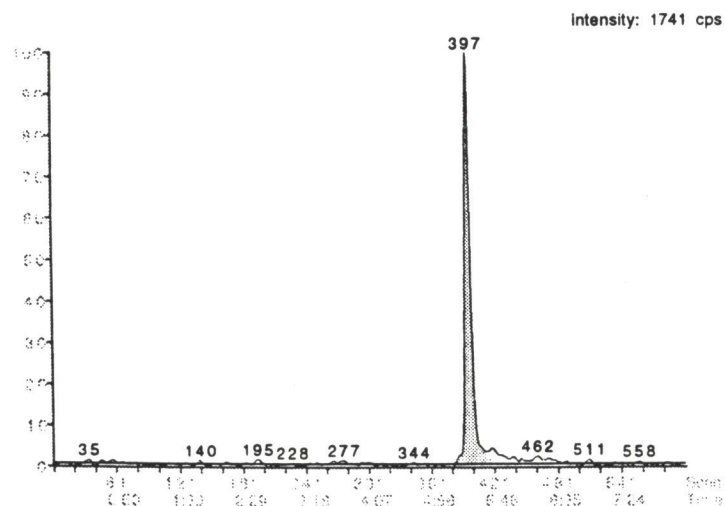
09250004 No Samplename 12:57
- User: DDM - Compound: Q1

E-10. Soil Sample Fortified at 0.010 (10 X LOQ)

8:12 in 1 period
CGA355190
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
247.8->174.8
Noise Thres. 1.0
Quant Thres. 0.9
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:26

Area 9709
Height 1736
Start Time 5:14
End Time 5:39
Integration Width 0:24.6
Retention Time 5:26
Integration Type A - BV



CG A353042 115.8->86.0 No Internal Standard

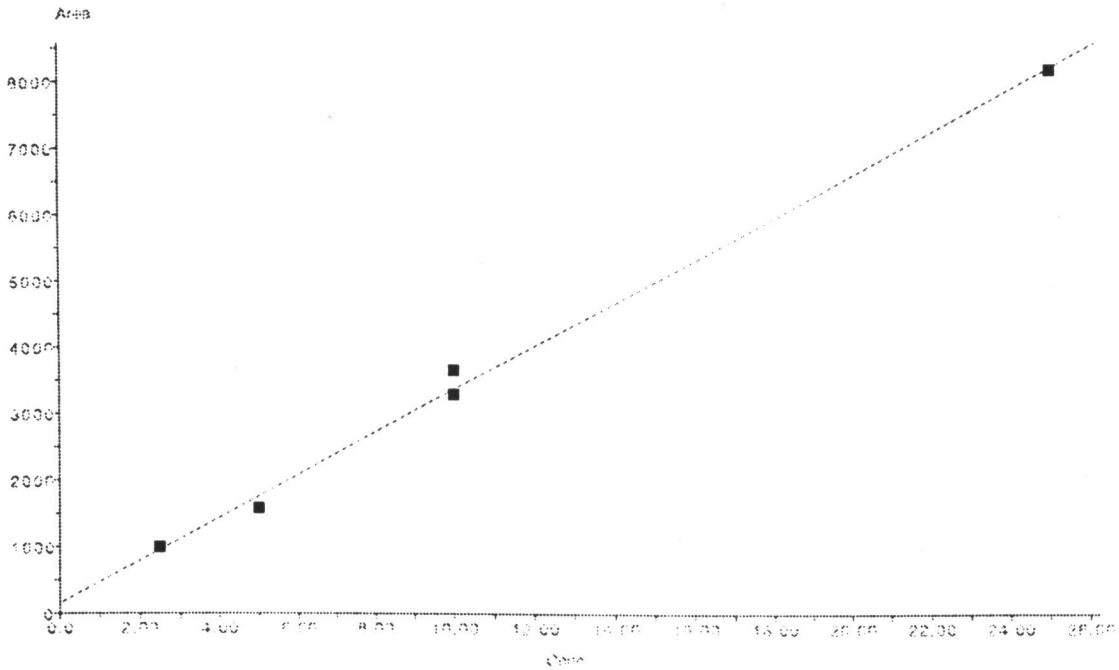
Linear

Intercept = 147.502

Slope = 322.152

Correlation Coeff. = 0.99807

F. CGA-353042 Calibration Curve (used with 10 X LOQ level only)



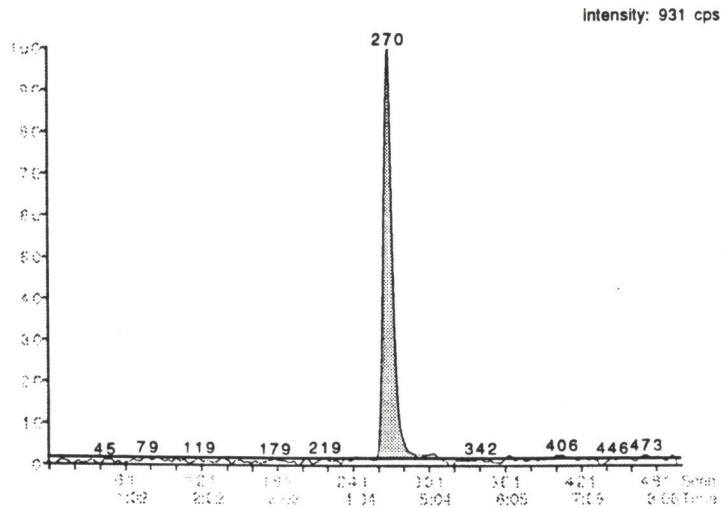
G. CGA-353042 Calibration Standards and Soil Sample Chromatograms

09260001 No Samplename 09:49
- User: DDM - Compound: Q1

G-1. 20 µl @ 25 ng/mL

8:25 in 1 period
CG A353042
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
115.8->86.0
Noise Thres. 1.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 4:32
Area 8158
Height 919
Start Time 4:22
End Time 4:56
Integration Width 0:34.4
Retention Time 4:33
Integration Type A - BV

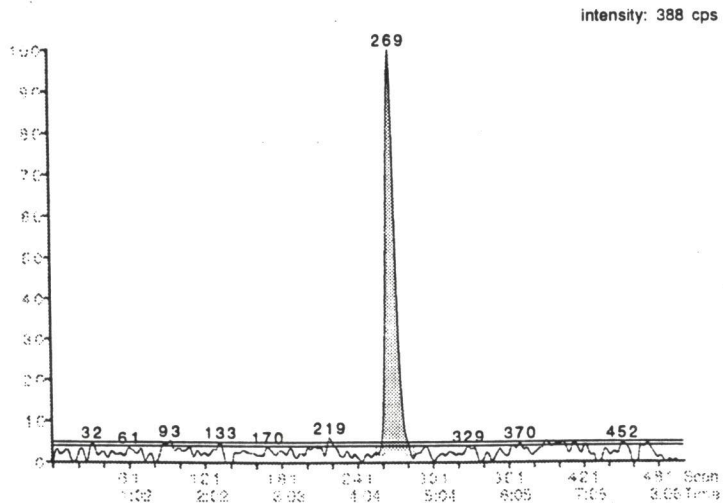


09260002 No Samplename 10:02
- User: DDM - Compound: Q1

G-2. 20 µl @ 10 ng/mL

8:25 in 1 period
CG A353042
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
115.8->86.0
Noise Thres. 1.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 4:32
Area 3281
Height 378
Start Time 4:22
End Time 4:45
Integration Width 0:23.2
Retention Time 4:32
Integration Type A - BB

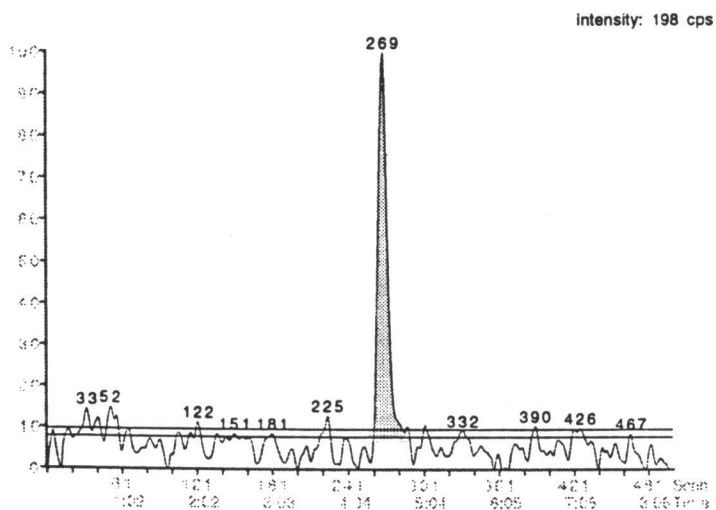


09260003 No Samplename 10:13
- User: DDM - Compound: Q1

G-3. 20 μ l @ 5 ng/mL

8:25 in 1 period
CG A353042
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
115.8->86.0
Noise Thres. 1.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 4:32
Area 1575
Height 183
Start Time 4:24
End Time 4:48
Integration Width 0:24.3
Retention Time 4:32
Integration Type A - BV

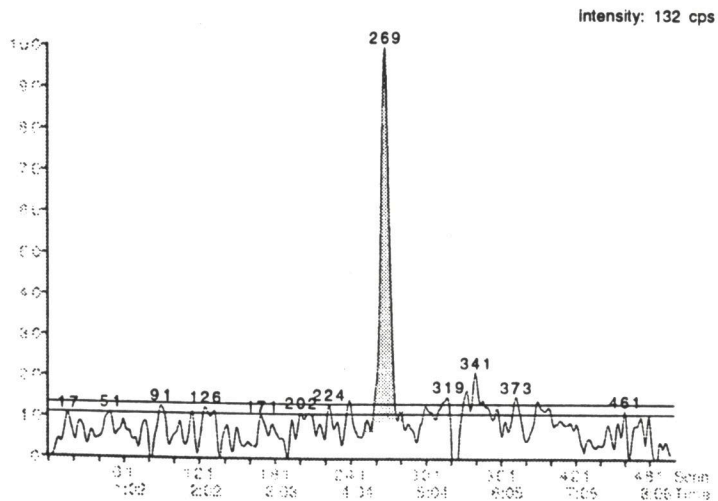


09260004 No Samplename 10:24
- User: DDM - Compound: Q1

G-4. 20 μ l @ 2.5 ng/mL

8:25 in 1 period
CG A353042
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
115.8->86.0
Noise Thres. 1.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 4:32
Area 989
Height 120
Start Time 4:19
End Time 4:42
Integration Width 0:23.2
Retention Time 4:32
Integration Type M

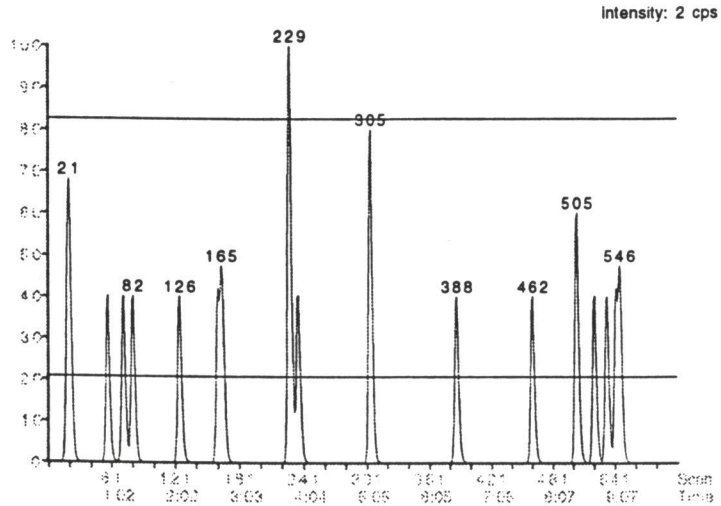


10230012 No Samplename 11:36
- User: DDM - Compound: Q1

G-5. Soil Matrix Blank at MDL

10:07 in 1 period
CGA353042
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans
115.8->86.0
Noise Thres. 0.4
Quant Thres. 0.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:54
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

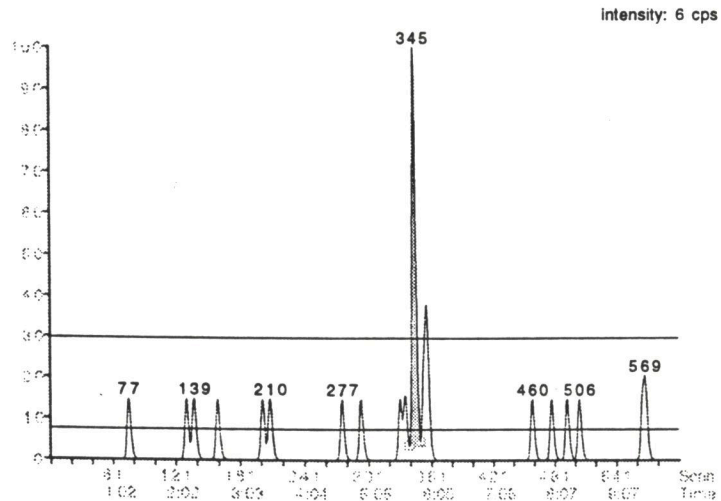


10230014 No Samplename 12:06
- User: DDM - Compound: Q1

G-6. Soil Sample Fortified at 0.0003 ppm (MDL)

10:07 in 1 period
CGA353042
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans
115.8->86.0
Noise Thres. 0.4
Quant Thres. 0.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:54
Area 25
Height 6
Start Time 5:45
End Time 5:54
Integration Width 0:09.1
Retention Time 5:49
Integration Type A - BB



10230011 No Samplename 11:25
- User: DDM - Compound: Q1

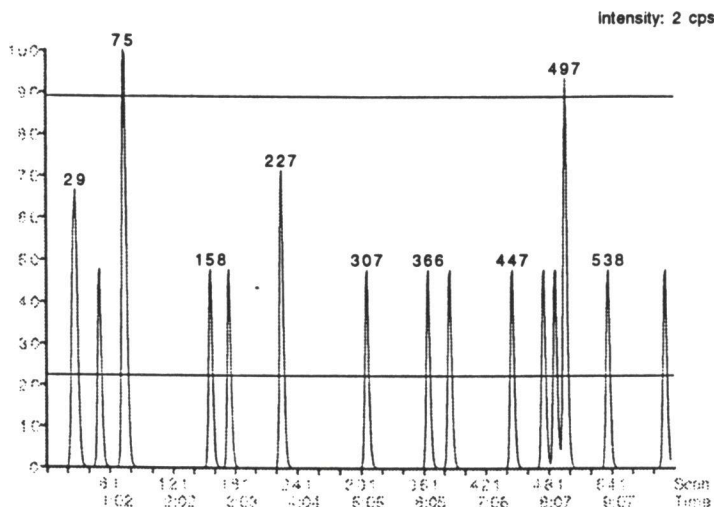
G-7. Soil Matrix Blank at LOQ

10:07 in 1 period
CGA353042
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans

115.8->86.0

Noise Thres.	0.4
Quant Thres.	0.1
Min. Width	6
Mult. Width	2
Base. Width	100
RT Win. (secs)	20
Smooth	5
Expected RT	5:54
Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



10230007 No Samplename 10:34
- User: DDM - Compound: Q1

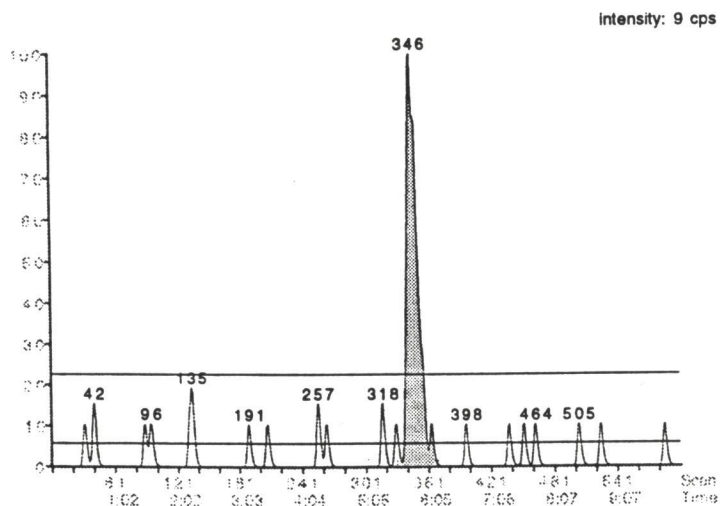
G-8. Soil Sample Fortified at 0.001 ppm (LOQ)

10:07 in 1 period
CGA353042
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans

115.8->86.0

Noise Thres.	0.4
Quant Thres.	0.1
Min. Width	6
Mult. Width	2
Base. Width	100
RT Win. (secs)	20
Smooth	5
Expected RT	5:54
Area	109
Height	9
Start Time	5:40
End Time	6:10
Integration Width	0:30.4
Retention Time	5:50
Integration Type	M

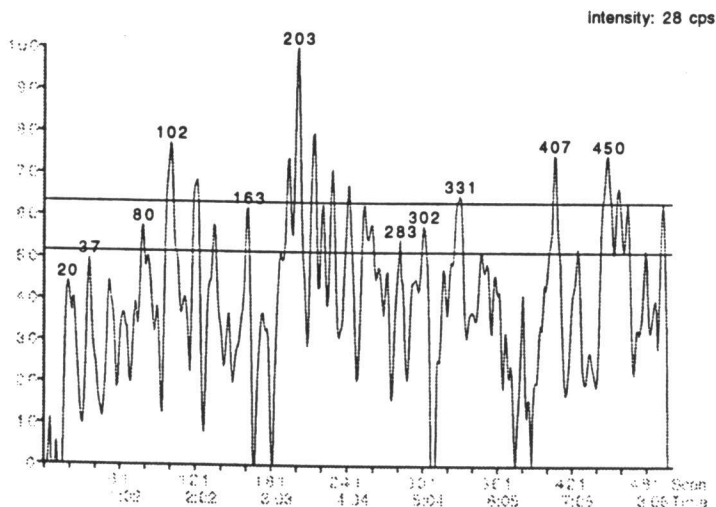


09260010 No Samplename 11:44
- User: DDM - Compound: Q1

G-9. Soil Matrix Blank 10 X LOQ

8:25 in 1 period
CG A353042
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
115.8->86.0
Noise Thres. 1.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 4:32
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

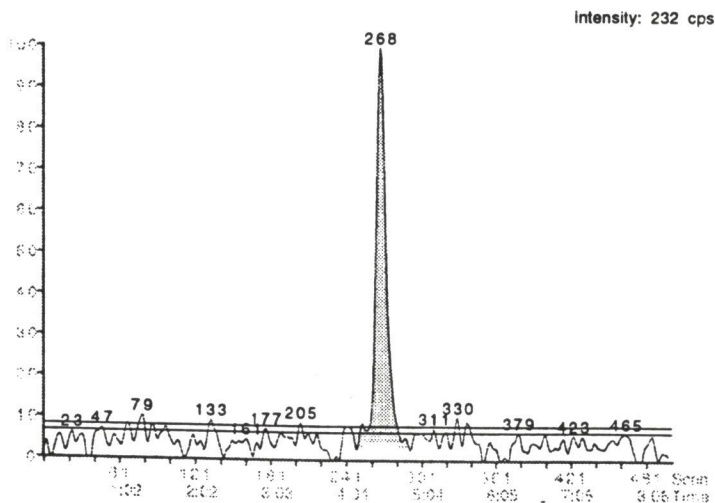


09260005 No Samplename 10:39
- User: DDM - Compound: Q1

G-10. Soil Sample Fortified at 0.010 ppm (10 X LOQ)

8:25 in 1 period
CG A353042
No Internal Standard
Use Area

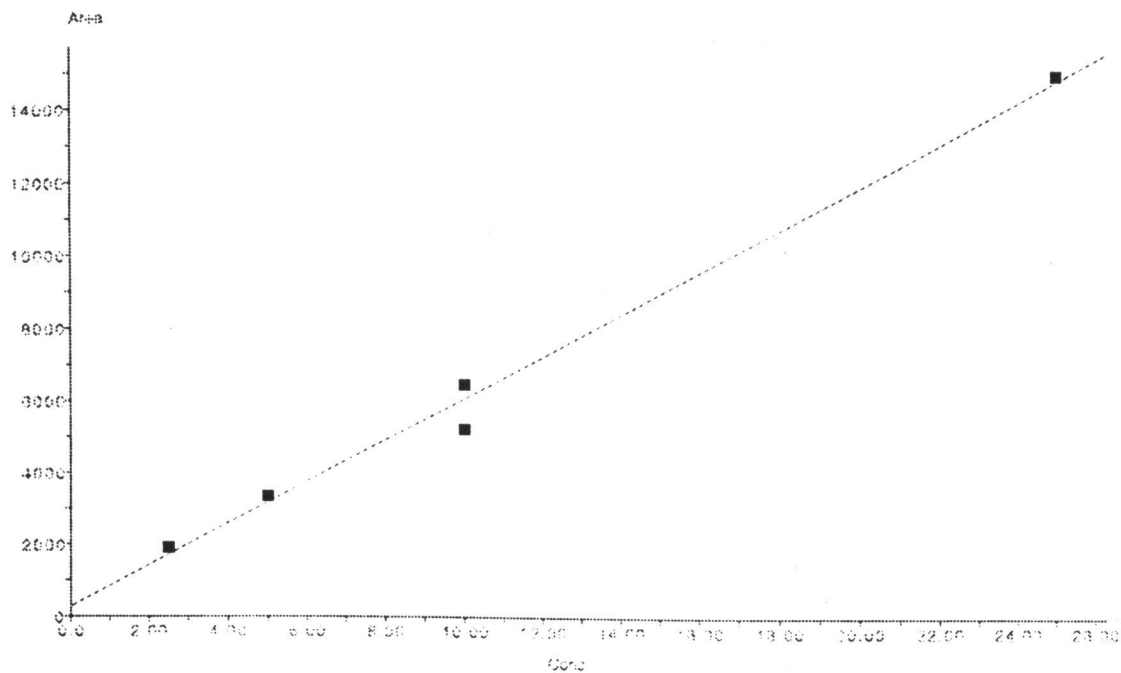
1: 8:24 MRM, 500 scans
115.8->86.0
Noise Thres. 1.6
Quant Thres. 1.3
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 4:32
Area 2112
Height 222
Start Time 4:14
End Time 4:47
Integration Width 0:33.4
Retention Time 4:31
Integration Type M



NOA404617 236.8->174.8 No Internal Standard
Linear

Intercept = 259.034
Slope = 581.901
Correlation Coeff. = 0.99532

H. NOA-404617 Calibration Curve (used with 10 X LOQ only)



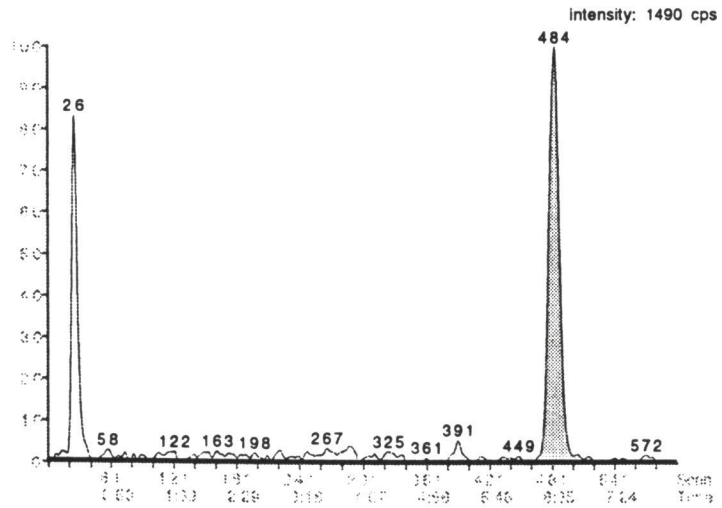
I. NOA-404617 Calibration Standards and Soil Sample Chromatograms

09250003 No Samplename 12:23
 - User: DDM - Compound: Q1

I-1. 20 µl @ 25 ng/mL

8:12 in 1 period
 NOA404617
 No Internal Standard
 Use Area

1: 8:12 MRM, 600 scans
 236.8->174.8
 Noise Thres. 0.6
 Quant Thres. 0.2
 Min. Width 6
 Mult. Width 2
 Base. Width 100
 RT Win. (secs) 20
 Smooth 5
 Expected RT 6:36
 Area 14949
 Height 1488
 Start Time 6:20
 End Time 6:51
 Integration Width 0:31.2
 Retention Time 6:37
 Integration Type A - BV

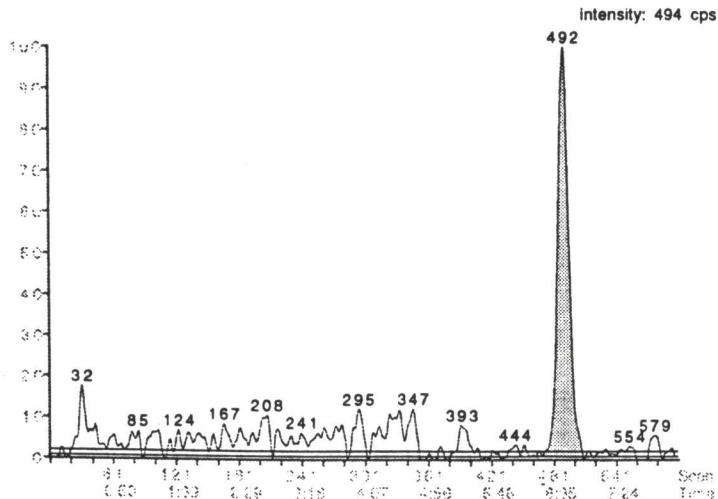


09250002 No Samplename 12:08
 - User: DDM - Compound: Q1

I-2. 20 µl @ 10 ng/mL

8:12 in 1 period
 NOA404617
 No Internal Standard
 Use Area

1: 8:12 MRM, 600 scans
 236.8->174.8
 Noise Thres. 0.6
 Quant Thres. 0.2
 Min. Width 6
 Mult. Width 2
 Base. Width 100
 RT Win. (secs) 20
 Smooth 5
 Expected RT 6:36
 Area 5213
 Height 493
 Start Time 6:26
 End Time 6:58
 Integration Width 0:32.0
 Retention Time 6:44
 Integration Type A - VB

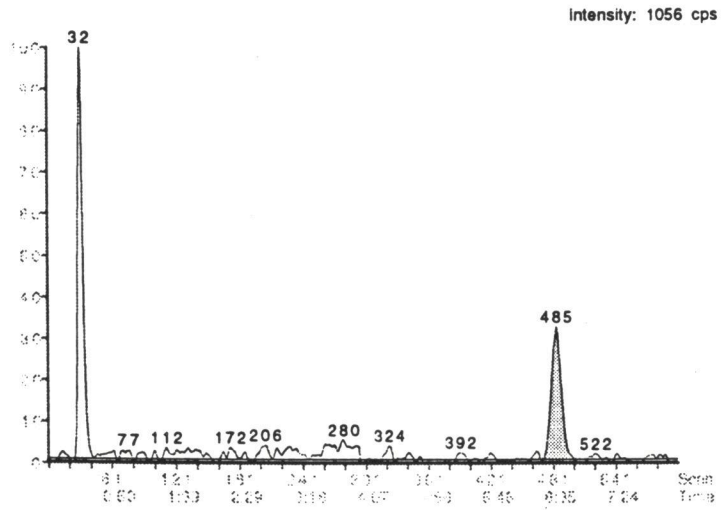


09250005 No Samplename 13:12
- User: DDM - Compound: Q1

I-3. 20 µl @ 5 ng/mL

8:12 In 1 period
NOA404617
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
236.8->174.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:36
Area 3331
Height 345
Start Time 6:27
End Time 6:53
Integration Width 0:26.3
Retention Time 6:38
Integration Type A - VB

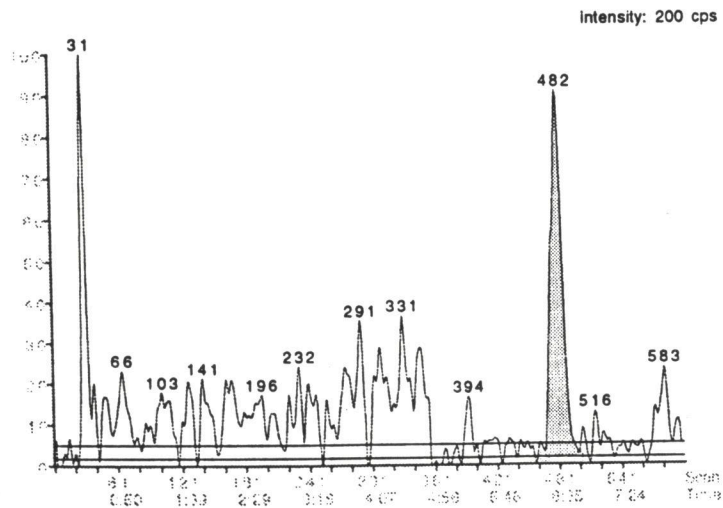


09250006 No Samplename 13:27
- User: DDM - Compound: Q1

I-4. 20 µl @ 2.5 ng/mL

8:12 In 1 period
NOA404617
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
236.8->174.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:36
Area 1891
Height 178
Start Time 6:23
End Time 6:50
Integration Width 0:26.3
Retention Time 6:36
Integration Type A - VV



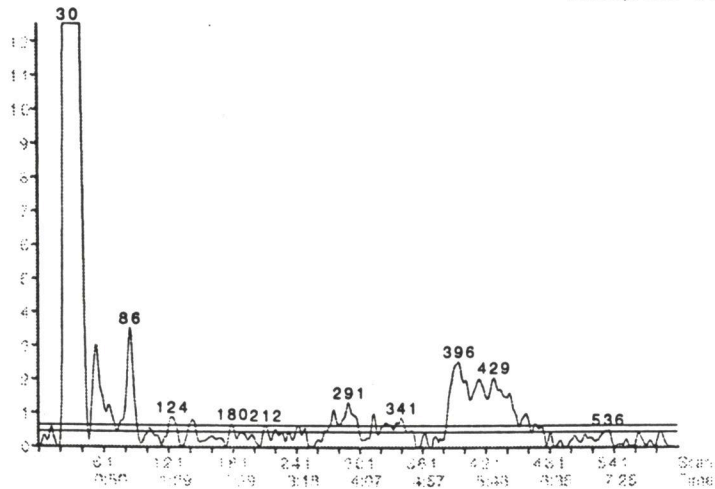
10100025 No Samplename 15:18
 - User: DDM - Compound: Q1

I-5. Soil Matrix Blank at MDL

8:13 in 1 period
 NOA404617
 No Internal Standard
 Use Area

Intensity: 3965 cps

1: 8:12 MRM, 600 scans
 236.8->174.8
 Noise Thres. 1.6
 Quant Thres. 1.1
 Min. Width 6
 Mult. Width 2
 Base. Width 100
 RT Win. (secs) 20
 Smooth 5
 Expected RT 6:36
 Area 0
 Height 0
 Start Time 0:00.0
 End Time 0:00.0
 Integration Width 0:00.0
 Retention Time 0:00.0
 Integration Type M



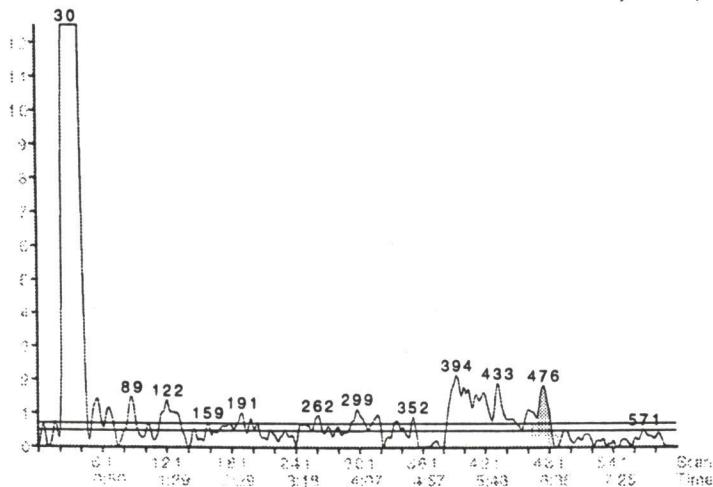
10100027 No Samplename 15:49
 - User: DDM - Compound: Q1

I-6. Soil Sample Fortified at 0.0003 ppm (MDL)

8:13 in 1 period
 NOA404617
 No Internal Standard
 Use Area

Intensity: 3581 cps

1: 8:12 MRM, 600 scans
 236.8->174.8
 Noise Thres. 1.6
 Quant Thres. 1.1
 Min. Width 6
 Mult. Width 2
 Base. Width 100
 RT Win. (secs) 20
 Smooth 5
 Expected RT 6:36
 Area 434
 Height 53
 Start Time 6:25
 End Time 6:38
 Integration Width 0:12.3
 Retention Time 6:31
 Integration Type A - VB

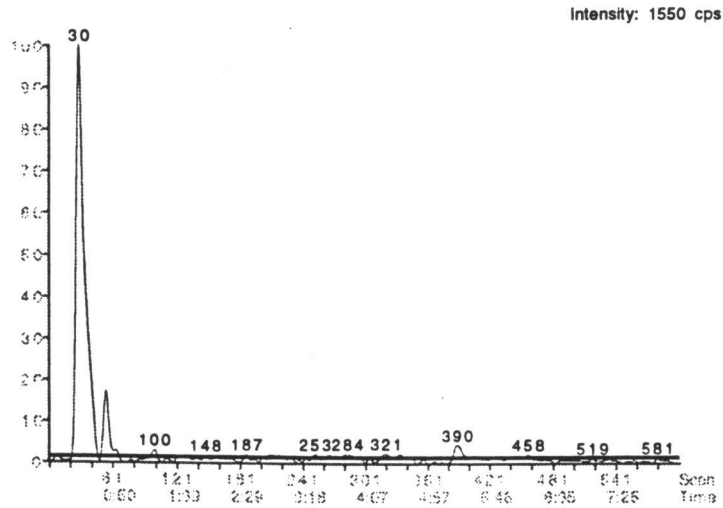


10100024 No Samplename 15:04
- User: DDM - Compound: Q1

I-7. Soil Matrix Blank at LOQ

8:13 in 1 period
NOA404617
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
236.8->174.8
Noise Thres. 1.6
Quant Thres. 1.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:36
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

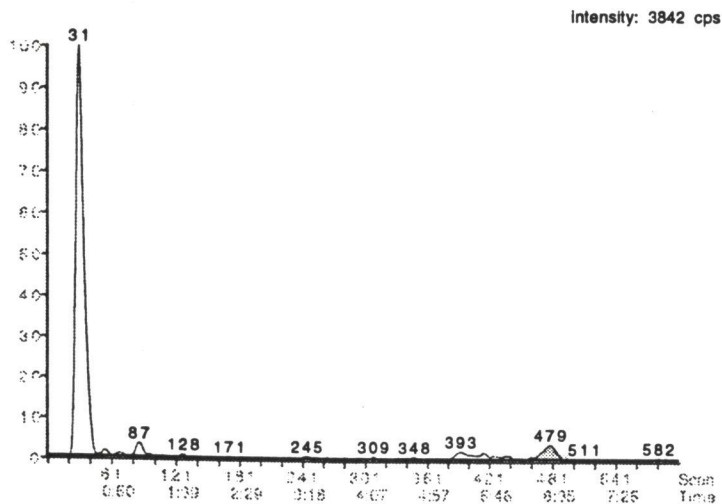


10100021 No Samplename 14:19
- User: DDM - Compound: Q1

I-8. Soil Sample Fortified at 0.001 ppm (LOQ)

8:13 in 1 period
NOA404617
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
236.8->174.8
Noise Thres. 1.6
Quant Thres. 1.1
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:36
Area 1725
Height 143
Start Time 6:21
End Time 6:43
Integration Width 0:21.4
Retention Time 6:34
Integration Type A - VB



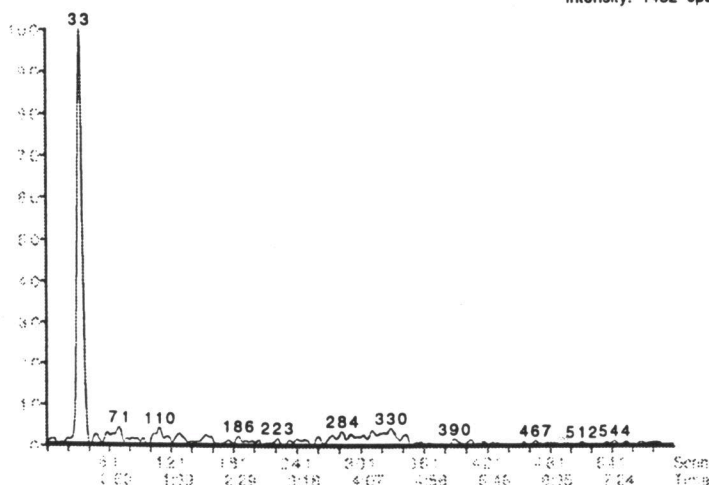
09250010 No Samplename 14:29
- User: DDM - Compound: Q1

I-9. Soil Matrix Blank at 10 X LOQ

8:12 In 1 period
NOA404617
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
236.8->174.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:36
Area 0
Height 0
Start Time 6:44
End Time 6:46
Integration Width 0:01.6
Retention Time 6:44
Integration Type M

Intensity: 1482 cps



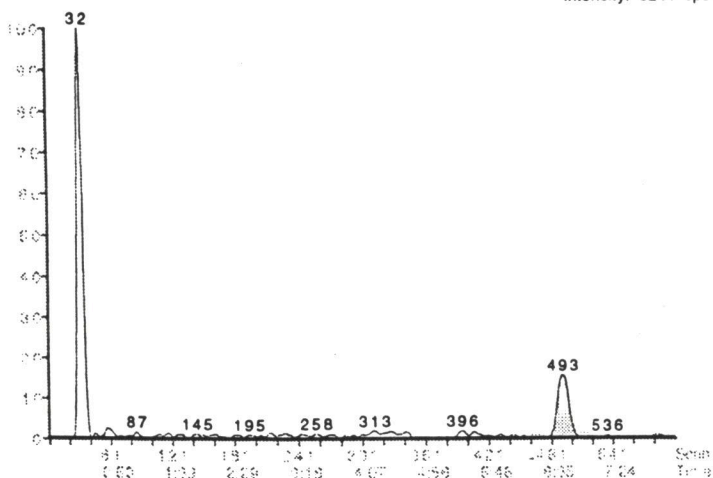
09250004 No Samplename 12:57
- User: DDM - Compound: Q1

I-10. Soil Sample Fortified at 0.010 ppm (10 X LOQ)

8:12 In 1 period
NOA404617
No Internal Standard
Use Area

1: 8:12 MRM, 600 scans
236.8->174.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:36
Area 5578
Height 504
Start Time 6:23
End Time 7:00
Integration Width 0:36.9
Retention Time 6:45
Integration Type M

Intensity: 3241 cps

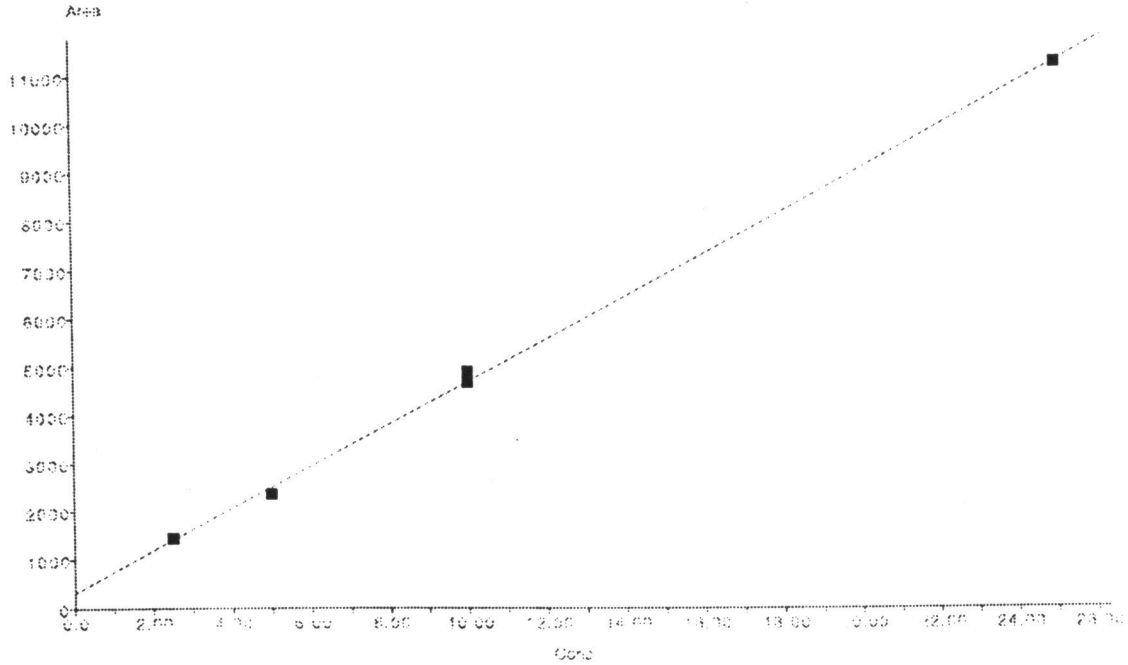


NOA407475 247.0->160.8 No Internal Standard

Linear

Intercept = 311.389
Slope = 438.059
Correlation Coeff. = 0.99943

J. NOA-407475 Calibration Curve (used with 10 X LOQ level only)



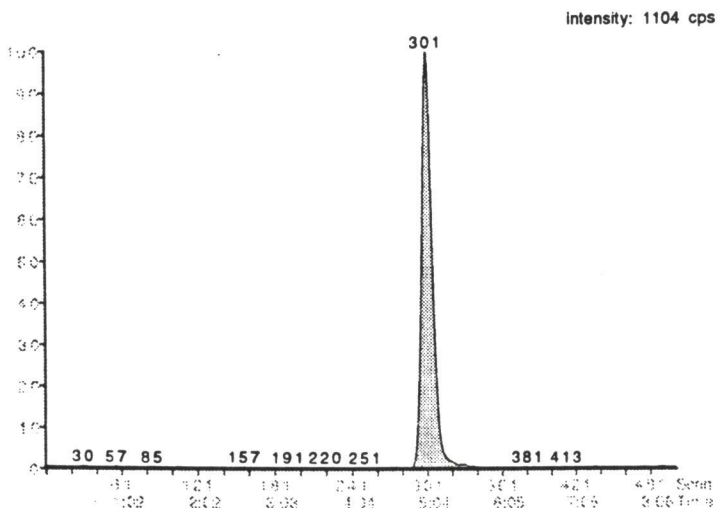
K. NOA-407475 Calibration Standards and Soil Sample Chromatograms

09260001 No Samplename 09:49
- User: DDM - Compound: Q1

K-1. 20 µl @ 25 ng/mL

8:25 in 1 period
NOA407475
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.4
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:04
Area 11221
Height 1103
Start Time 4:52
End Time 5:28
Integration Width 0:36.4
Retention Time 5:04
Integration Type A - BV

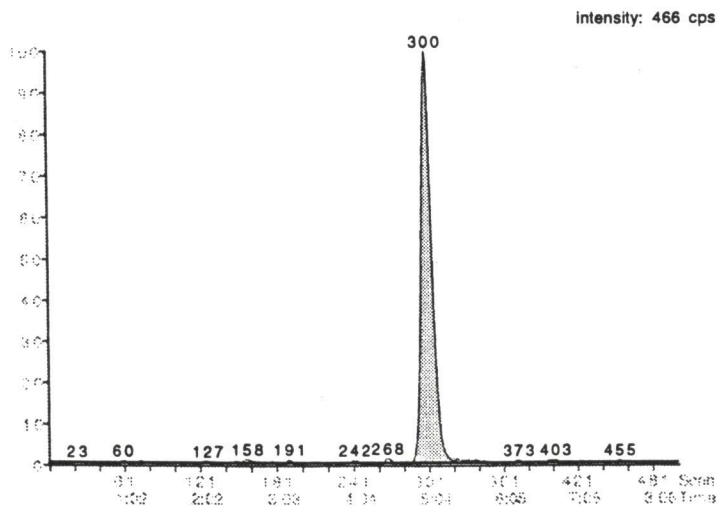


09260002 No Samplename 10:02
- User: DDM - Compound: Q1

K-2. 20 µl @ 10 ng/mL

8:25 in 1 period
NOA407475
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.4
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:04
Area 4674
Height 464
Start Time 4:50
End Time 5:23
Integration Width 0:33.3
Retention Time 5:03
Integration Type A - BV

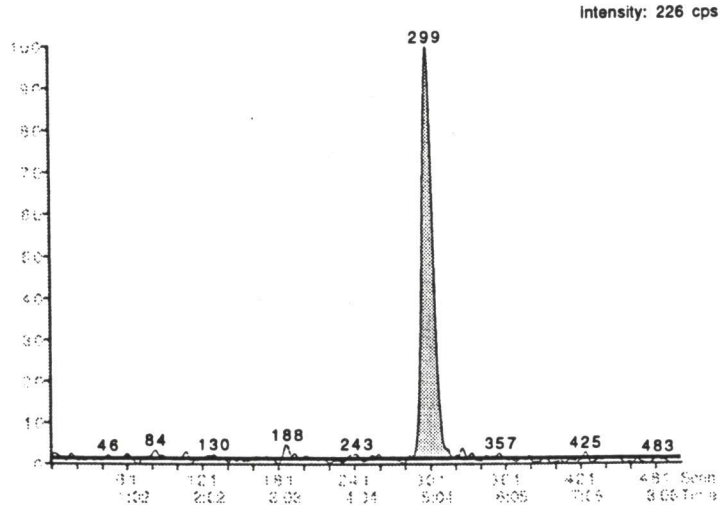


09260003 No Samplename 10:13
- User: DDM - Compound: Q1

K-3. 20 µl @ 5 ng/mL

8:25 in 1 period
NOA407475
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.4
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:04
Area 2343
Height 224
Start Time 4:50
End Time 5:21
Integration Width 0:31.3
Retention Time 5:02
Integration Type A - VV

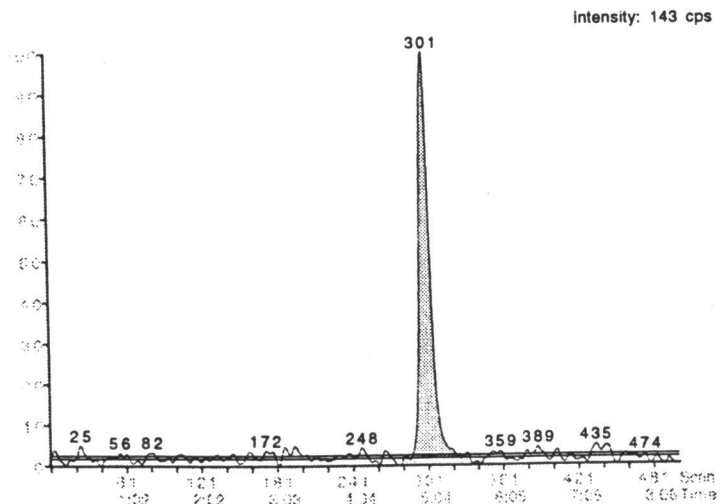


09260004 No Samplename 10:24
- User: DDM - Compound: Q1

K-4. 20 µl @ 2.5 ng/mL

8:25 in 1 period
NOA407475
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.4
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:04
Area 1428
Height 141
Start Time 4:51
End Time 5:26
Integration Width 0:35.4
Retention Time 5:04
Integration Type A - BV

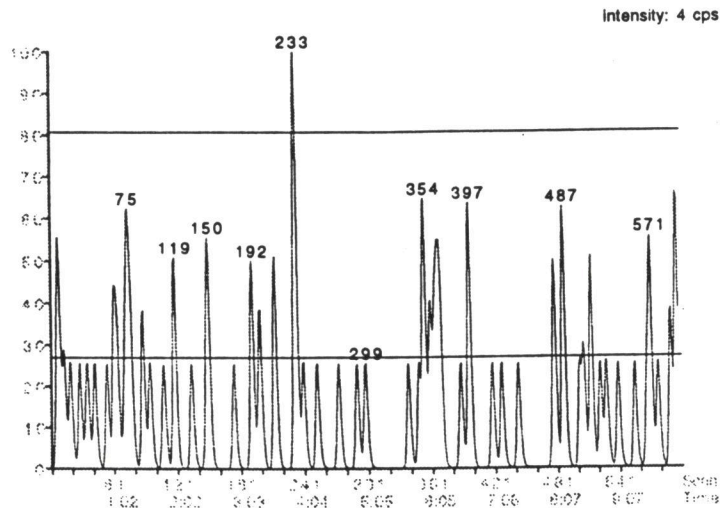


10230011 No Samplename 11:25
- User: DDM - Compound: Q1

K-5. Soil Matrix Blank at MDL

10:07 in 1 period
NOA407475
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:40
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

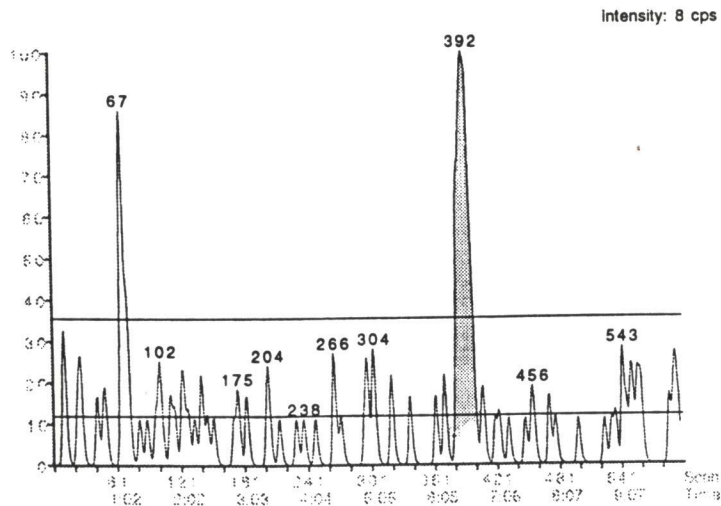


10230014 No Samplename 12:06
- User: DDM - Compound: Q1

K-6. Soil Sample Fortified at 0.0003 ppm (MDL)

10:07 in 1 period
NOA407475
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:40
Area 102
Height 7
Start Time 6:24
End Time 6:46
Integration Width 0:21.2
Retention Time 6:37
Integration Type A - BB

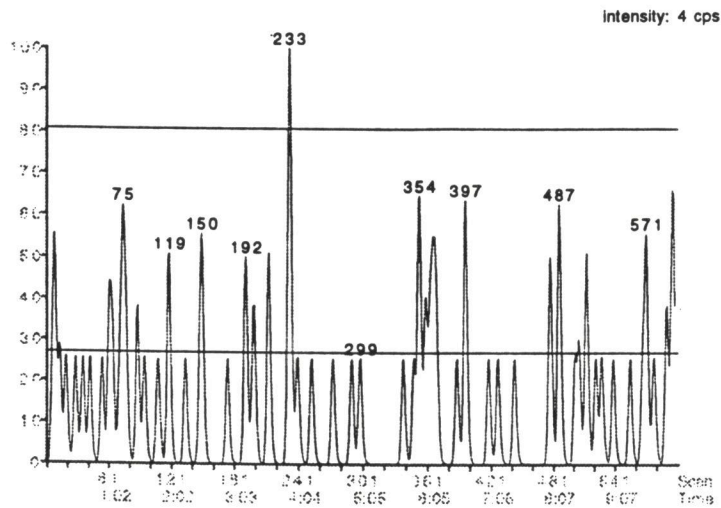


10230011 No Samplename 11:25
- User: DDM - Compound: Q1

K-7. Soil Matrix Blank at LOQ

10:07 In 1 period
NOA407475
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:40
Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

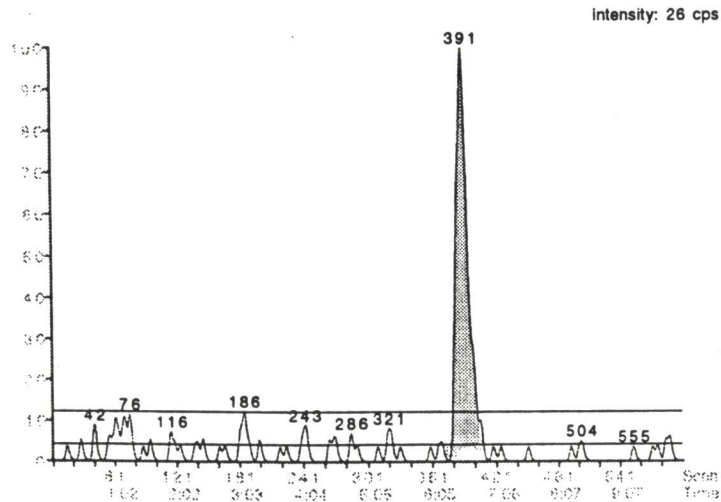


10230007 No Samplename 10:34
- User: DDM - Compound: Q1

K-8. Soil Sample Fortified at 0.001 ppm (LOQ)

10:07 In 1 period
NOA407475
No Internal Standard
Use Area

1: 10:06 MRM, 600 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.2
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 6:40
Area 311
Height 25
Start Time 6:23
End Time 6:49
Integration Width 0:25.3
Retention Time 6:36
Integration Type A - BV

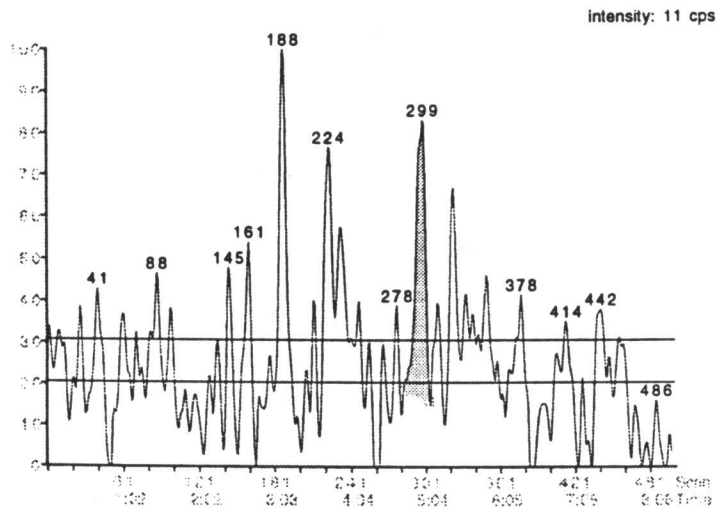


09260009 No Samplename 11:32
- User: DDM - Compound: Q1

K-9. Soil Matrix Blank at 10 X LOQ

8:25 in 1 period
NOA407475
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.4
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:04
Area 67
Height 7
Start Time 4:46
End Time 5:06
Integration Width 0:20.2
Retention Time 5:02
Integration Type A - BB

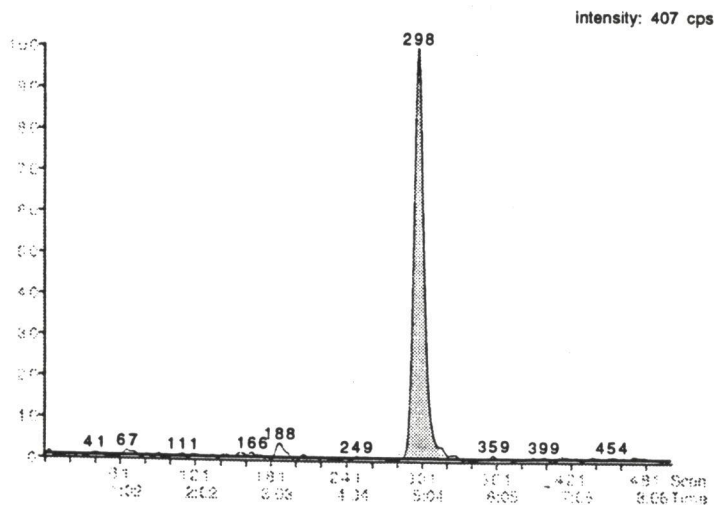


09260006 No Samplename 10:52
- User: DDM - Compound: Q1

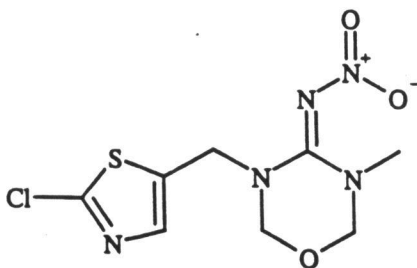
K-10. Soil Sample Fortified at 0.010 (10 X LOQ)

8:25 in 1 period
NOA407475
No Internal Standard
Use Area

1: 8:24 MRM, 500 scans
247.0->160.8
Noise Thres. 0.6
Quant Thres. 0.4
Min. Width 6
Mult. Width 2
Base. Width 100
RT Win. (secs) 20
Smooth 5
Expected RT 5:04
Area 4024
Height 404
Start Time 4:49
End Time 5:17
Integration Width 0:28.3
Retention Time 5:01
Integration Type A - VV



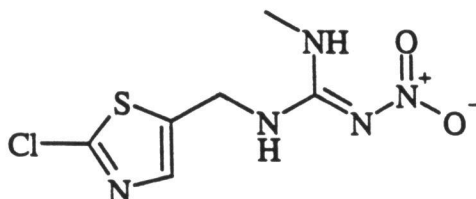
APPENDIX A:

CHEMICAL NAMES AND STRUCTURES

CGA-293343

CAS Number: 153719-23-4

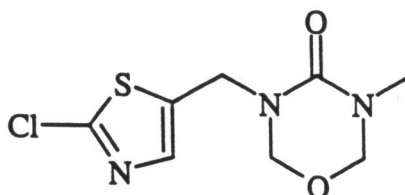
4H-1,3,5-Oxadiazin-4-imine, 3-[(2-chloro-5-thiazolyl)methyl]tetrahydro-5-methyl-N-nitro-



CGA-322704

CAS Number: 131748-59-9

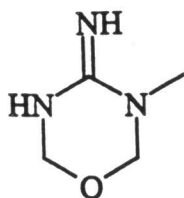
Guanidine, N-[(2-chloro-5-thiazolyl)methyl]-N'-methyl-N''-nitro-



CGA-355190

CAS Number: not available

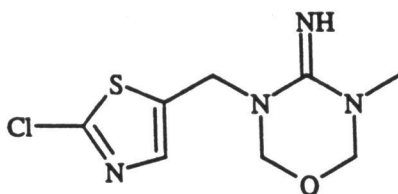
4H-1,3,5-Oxadiazin-4-one, 3-[(2-chloro-5-thiazolyl)methyl]tetrahydro-5-methyl-

CHEMICAL NAMES AND STRUCTURES (Continued)

CGA-353042

CAS Number: not available

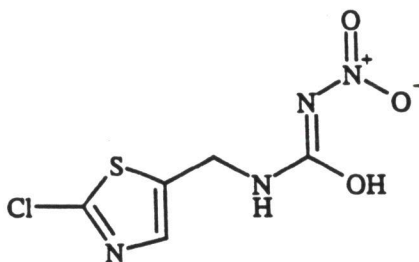
2H-1,3,5-Oxadiazin-4-amine, 3,6-dihydro-3-methyl-



NOA-407475

CAS Number: not available

4H-1,3,5-Oxadiazin-4-imine, 3-[(2-chloro-5-thiazolyl)methyl]tetrahydro-5-methyl-



NOA-404617

CAS Number: not available

Urea, N-[(2-chloro-5-thiazolyl)methyl]-N'-nitro-

APPENDIX B:

SOIL CHARACTERIZATIONS OF STUDY SITE IN PUTNAM COUNTY, FLORIDA

Soil Characteristic	Soil Depth, Inches					
	0-6	6-12	12-18	18-24	24-30	30-36
% Organic Matter	1.0	0.7	0.3	0.2	0.2	1.0
pH	7.7	6.3	6.3	6.4	6.1	5.2
CEC (meq/100g)	9.5	8.8	3.9	3.9	6.3	3.0
Water Holding Capacity (%) at 1/3 bar	3.2	2.8	2.3	2.1	3.0	5.2
Water Holding Capacity (%) at 15 bar	1.7	1.4	1.0	0.9	1.5	3.0
Bulk Density (disturbed) (g/cc)	1.50	1.51	1.54	1.54	1.44	1.49
% Sand	90	90	88	88	88	84
% Silt	3	3	5	3	3	7
% Clay	7	7	7	9	9	9
Texture	Sand	Sand	Loamy Sand	Loamy Sand	Loamy Sand	Loamy Sand

Characterization analysis performed by AGVISE Laboratories in Northwood, North Dakota.