



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PESTICIDES AND TOXIC
SUBSTANCES

January 18, 2000

SUBJECT: Trimethylsulfonium Method Evaluation (Glyphosate
Trimesium: Sulfosate)
PC Code: 128501
DP Barcode: None

FROM: Sid Abel, Environmental Scientist *Sid Abel 1/18/00*
Fate and Monitoring Branch
Environmental Fate and Effects Division (7507C)

THRU: Betsy Behl, Chief *Betsy Behl 1/18/00*
Fate and Monitoring Branch
Environmental Fate and Effects Division (7507C)

TO: Donald Stubbs, Chief
Herbicide Branch
Registration Division (7505C)

The Environmental Fate and Effects Division (EFED) requested and received an evaluation for the determination of residues of trimethylsulfonium (TMS) in soil using a method submitted by Zeneca Ag Products. Attached is a copy of the method evaluation provided by the Environmental Chemistry Laboratory (ECL).

The EFED concludes, along with the ECL, that this method is suitable for monitoring residues of TMS (Touchdown) in soil at or above the suggested analytical limits. The estimated limit of detection (LOD) was determined to be 25 ppb and the estimated limit of quantitation (LOQ) to be 50 ppb. The following modification was noted and will require description if future submission using this method:

The dealkylation step required that the vials be shaken two or three times during the period of heating to improve method precision. This was not pointed out in the method provided by Zeneca.



2057406

If you have any questions concerning this matter, please feel free to contact Sid Abel.

Attachment



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF PESTICIDE PROGRAMS
ENVIRONMENTAL CHEMISTRY LABORATORY
BUILDING 1105—JOHN C. STENNIS SPACE CENTER
STENNIS SPACE CENTER, MISSISSIPPI 39529-6000
TELEPHONE (228) 688-3216 FACSIMILE (228) 688-3536

JAN 11 2000

MEMORANDUM

SUBJECT: Trimethylsulfonium Method Evaluation
Report No. ECM 0144S3

FROM: Aubry E. Dupuy, Jr., Chief *Aubry E. Dupuy, Jr.*
BEAD/Environmental Chemistry Lab

THRU: Elizabeth Leovey, Acting Associate Director
OPP/Environmental Fate and Effects Division (7507C)

THRU: Donald A. Marlow, Acting Associate Division Director
BEAD (7503C)

TO: Jim Carleton, Chemist
OPP/Environmental Fate and Effects Division (7507C)

The EFED/Fate and Monitoring Branch has requested Environmental Chemistry Lab evaluations for trimethylsulfonium (TMS) in soil, MRID #443265-07, using a method submitted by ZENECA Ag Products, entitled "TOUCHDOWN®: Determination of Residues of the Trimethylsulfonium Cation in Soil and Water by Gas Chromatography".

This method was evaluated with soil fortified at three levels, 0.025, 0.05 and 0.5 ppm, and with quadruplicate analyses at each level.

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 are listed. The average percent recovery and relative standard deviation (RSD) for each spiking level is also presented here.

Part III: Experimental Details

In this section any modifications that were made, instrument parameters, representative sample calculations and standard curve are listed and/or discussed.

If you have any questions concerning this report, please contact Henry Shoemaker at (228) 688-1222 or me at (228) 688-3212.

Attachments

cc: Christian Byrne, QA Officer
BEAD/Environmental Chemistry Lab

Henry Shoemaker, Chemist
BEAD/Environmental Chemistry Lab

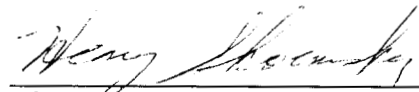
ENVIRONMENTAL CHEMISTRY METHOD EVALUATION REPORT
NUMBER ECM 0144S3

Trimethylsulfonium in Soil

ENVIRONMENTAL CHEMISTRY LABORATORY
BIOLOGICAL AND ECONOMIC ANALYSIS DIVISION

10/12/99

Prepared by:


Henry Shoemaker, ECL Chemist

Reviewed by:

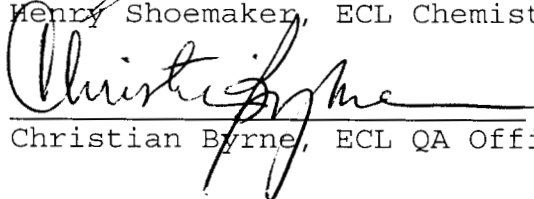

Christian Byrne, ECL QA Officer

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PART I

SUMMARY AND CONCLUSIONS

We have completed an Environmental Chemistry Method Laboratory Evaluation of trimethylsulfonium cation (TMS) in soil. This method, MRID# 443265-07, submitted by ZENECA Ag Products, is titled "TOUCHDOWN®: Determination of Residues of the Trimethylsulfonium Cation in Soil and Water by Gas Chromatography".

24 Feb
40 ppm
In order to evaluate this method, we fortified a soil matrix with trimethylsulfonium iodide at 0.025, 0.05 and 0.5 ppm of TMS equivalents. All samples were done in replicates of four at each level. We estimated the method limit of detection (LOD) to be 0.025 ppm. The registrant's method limit of quantitation (LOQ) of 0.05 ppm of TMS in soil was confirmed by our data. [After modifying the method to include shaking the sample during dealkylation, we found the precision to be well within our target limits of $\leq 20\%$ relative standard deviation (RSD) at or above the (LOQ).] For example, the (RSD) is 18.8% at the (LOQ) of 0.05 ppm for TMS and 15.3% at the 0.5 ppm level. The mean recoveries for TMS of 113.5% at the 0.05 ppm level and 79.7% at the 0.5 ppm level are well within the target range of 70% to 120%.

This method involves extracting TMS-Iodide from soil with a potassium hydroxide solution, which is then cleaned up by acidifying, adding activated charcoal, and filtering. Extraction of TMS cation is accomplished by heating the sample along with potassium hydroxide pellets and toluene in a sealed vial. The TMS is captured by the toluene which is transferred into an auto-sampler vial and analyzed by gas chromatography/flame photometric detector (FPD).

[We believe that this method can be used for monitoring for TMS at the suggested levels]

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PART II
 ANALYTICAL RESULTS FOR TMS
 EPA RECOVERIES IN SOIL

Sample	Added (ppm)	Found (ppm)	Recovery (%)	Statistics
Matrix Blank	0	0		
Matrix Blank	0	0		
Method Blank	0	0		
Method Blank	0	0		
TS 05	0.025	0.033		
TS 06	0.025	0.033		
TS 07	0.025	0.039		
TS 08	0.025	0.039		
TS 09	0.05	0.071	142.0	mean(Rec) = 113.5% SD = 21.38 RSD = 18.8%
TS 10	0.05	0.058	116.0	
TS 11	0.05	0.052	104.0	
TS 12	0.05	0.046	92.0	
TS 13	0.50	0.335	67.0	mean(Rec) = 79.7% SD = 12.17 RSD = 15.3%
TS 14	0.50	0.365	73.0	
TS 15	0.50	0.422	84.4	
TS 16	0.50	0.472	94.4	

PART III

EXPERIMENTAL SUMMARY

(a) Method Procedure

Soil samples weighing 25 grams were combined with 50 ml of 10% KOH solution and mixed for one hour on a mechanical shaker. After centrifuging, 14 ml was pipetted into a vial and two ml of concentrated hydrochloric acid was added for a total volume of 16 ml. Two hundred fifty milligrams of activated charcoal was mixed with the solution, which was then centrifuged and filtered. An aliquot of 8 ml of the cleaned extract was placed into a 15 ml dealkylation vial. A 5 ml portion of a 1.0 $\mu\text{g}/\text{ml}$ standard was also placed into a dealkylation vial. About 225 milligrams of stannous chloride dihydrate, an appropriate amount of toluene, and 5.5 grams of potassium hydroxide pellets were added to each vial. The vials were heated to 100°C for two hours in a heating block. The vials were cooled and centrifuged before a portion of the toluene was pipetted into autosampler vials fitted with limited-volume inserts. Analysis was performed using a gas chromatograph equipped with flame photometric detector (sulfur mode), auto-injection system, and a fused silica capillary DB-5 column.

(b) Source of Analytical Reference Standard

Analytical standard of trimethylsulfonium iodide was obtained from ZENECA Agricultural Products, Western Research Center, Richmond, California (I.D.NO. ASW01441-01A), and certified to be a purity of 99.0%.

(c) Source of Sample Matrix

The soil used was (Iowa Batch 4) obtained from Iowa State University and characterized by A&L Analytical Laboratories. A copy of the characterization report is included in this report.

(d) Instrumentation for Quantitation

1. Hewlett-Packard Model 6890 Gas Chromatograph with a flame photometric detector (sulfur mode) and automatic injector.
2. Column used was capillary DB-5, 30 m x 0.53 mm with 1.5 μm film thickness (J & W Scientific).

(e) Modification of Method

[The dealkylation step needs to be clarified. It is critical that the vials be shaken two or three times during the time they are being heated. This is not pointed out in the ZENECA method.]

(f) Calculations

1. Standard Curve

The HP-Chemstation contains preprogrammed data processing capabilities which calculates a standard curve for each analytical set using the responses of the calibration standards; which had concentrations of 0.125 $\mu\text{g/ml}$, 0.250 $\mu\text{g/ml}$, 0.500 $\mu\text{g/ml}$, 0.750 $\mu\text{g/ml}$ and 1.00 $\mu\text{g/ml}$. The calibration curve is constructed using polynomial regression with the concentration ($\mu\text{g/ml}$) on the X-axis and the response on the Y-axis.

The Power curve equation is: $Y = b (X)^m$

Where: Y = peak height
X = concentration ($\mu\text{g/ml}$)

2. Calculation of Analyte in Samples

The HP-Chemstation will calculate the concentration ($\mu\text{g/ml}$) of TMS in each sample extract using the calibration equation generated by the calibration standards in the analytical set.

Then to find the sample concentration (S) in ppm of the TMS use the formula:

$$S = \{ C \} \{ E / W \}$$

C = extract conc. ($\mu\text{g/ml}$)

E = extract volume
(0.7 ml for the LOD and LOQ samples)
(2.0 ml for the 10 X LOQ samples)

W = sample aliquot weight (3.5 grams)
Since portions of the sample were taken
At two different extraction steps an
Aliquot weight is calculated.
 $25\text{g} \times (14\text{ml}/50\text{ml}) \times (8\text{ml}/16\text{ml}) = 3.5\text{g}$

[Since the FPD(sulfur mode) detector is non-linear and somewhat erratic, I followed the registrant's suggestion that the analyst may choose to make duplicate injections of calibration standards and samples.]

[The final reported concentration (ppm) for each sample is the average of the two injections]

3. Example Calculation

Sample no. TS 10 for TMS

Injection 1

Conc. from HP-Chemstation = 0.3115 $\mu\text{g/ml}$

$S = \{0.3115 \mu\text{g/ml}\} \{0.7 \text{ ml}/3.5 \text{ g}\}$

$S = 0.0623 \mu\text{g/g}$

$S = 0.0623 \text{ ppm}$

Injection 2

Conc. From HP-Chemstation = 0.2725 $\mu\text{g/ml}$

$S = (0.2725 \mu\text{g/ml}) (0.7 \text{ ml}/3.5 \text{ g})$

$S = 0.0545 \mu\text{g/g}$

$S = 0.0545 \text{ ppm}$

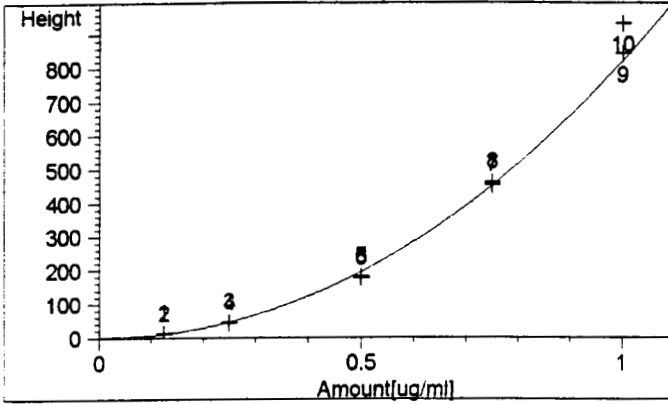
Average of two injections = 0.058 ppm

(g) Graphs and Data

The following pages contain a print-out of the calibration curves and data generated by the TMS standards and representative samples.

1. Pages 9 thru 19, calibration curves and raw data for calibration standards.
2. Pages 20 thru 26, chromatograms and raw data for selected samples.

=====
Calibration Curves
=====



TMS at exp. RT: 1.335
 FPD2 B,
 Correlation: 0.99705
 Residual Std. Dev.: 40.82506
 Formula: $y = b * x^m$
 m: 2.07816
 b: 823.22885
 x: Amount [ug/ml]
 y: Height

=====
Signal 1: FPD2 B,

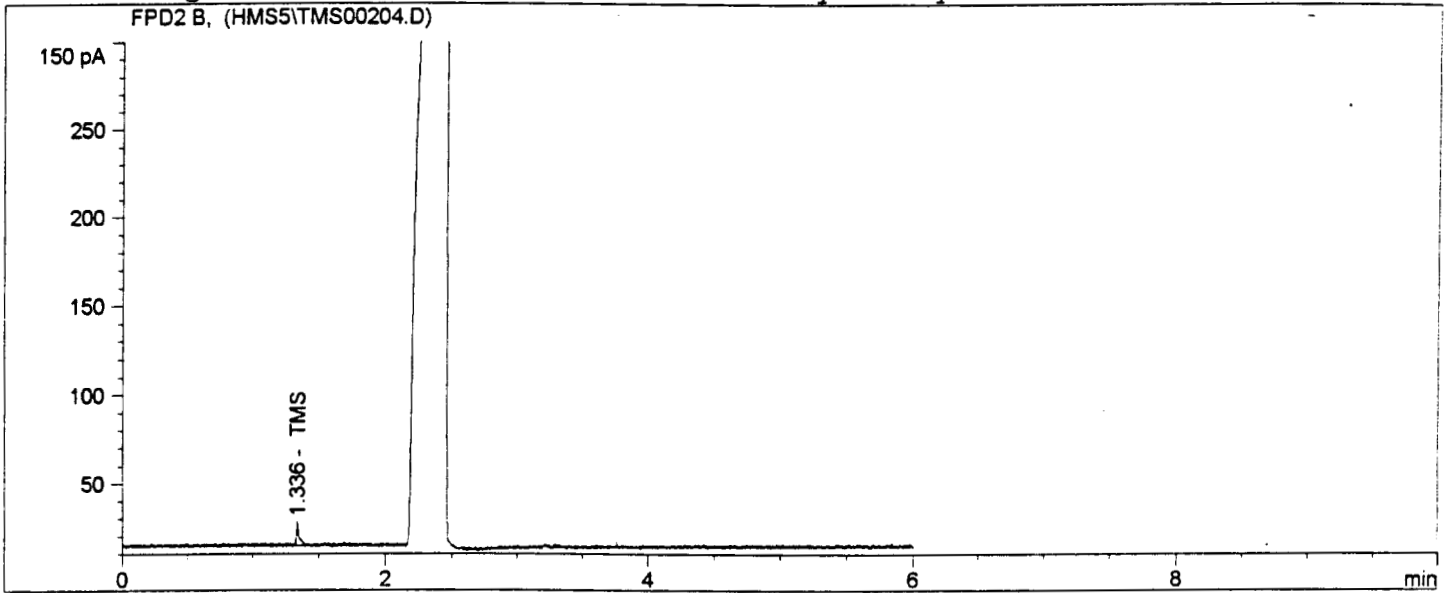
RetTime [min]	Lvl Sig	Amount [ug/ml]	Height	Amt/Height	Ref Grp Name
1.335	1	1.25000e-1	13.25222	9.43238e-3	TMS
		1.25000e-1	10.55258	1.18454e-2	
		2.50000e-1	44.14570	5.66307e-3	
		2.50000e-1	41.47395	6.02788e-3	
		5.00000e-1	181.21370	2.75917e-3	
		5.00000e-1	176.14017	2.83865e-3	
		7.50000e-1	452.96198	1.65577e-3	
		7.50000e-1	461.43347	1.62537e-3	
		1.00000	843.28717	1.18584e-3	
		1.00000	934.05133	1.07060e-3	

=====
Peak Sum Table
=====

```

=====
Injection Date   : 09/08/1999 1:26:25 PM           Seq. Line :    5
Sample Name     : cal 1   0.125 µg/ml             Vial      :    5
Acq. Operator   : Henry Shoemaker                 Inj       :    1
                                                    Inj Volume:  2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.336	PB	13.25222	1.03470e-2	1.37121e-1		TMS

Totals : 1.37121e-1

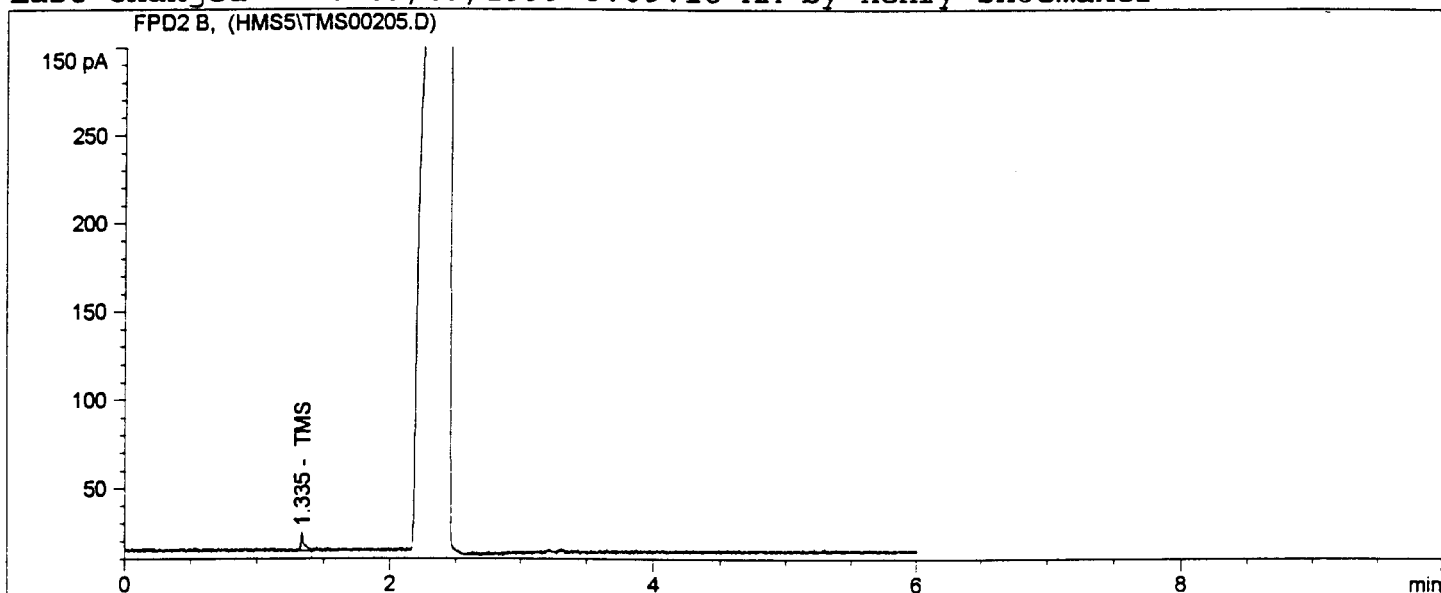
Results obtained with enhanced integrator!

*** End of Report ***

```

=====
Injection Date   : 09/08/1999 1:33:26 PM           Seq. Line :    6
Sample Name     : cal 1   0.125 µg/ml             Vial       :    5
Acq. Operator   : Henry Shoemaker                 Inj        :    1
                                                    Inj Volume : 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	BP	10.55258	1.16450e-2	1.22885e-1		TMS

Totals : 1.22885e-1

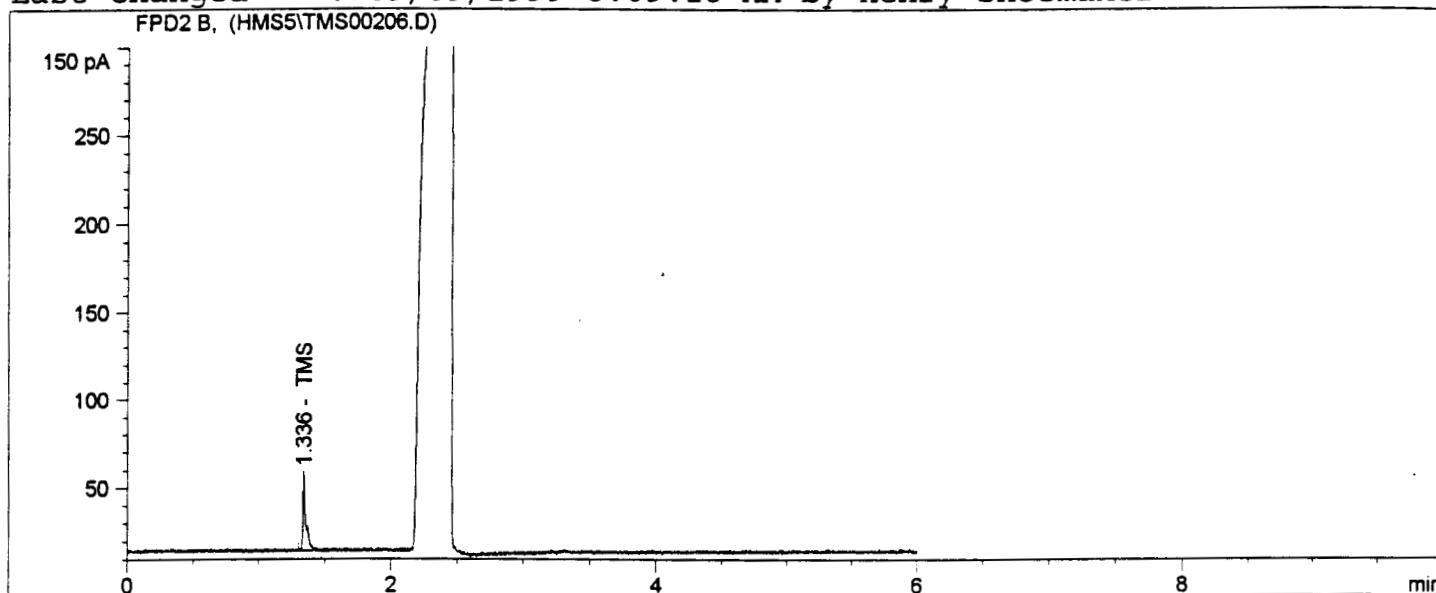
Results obtained with enhanced integrator!

*** End of Report ***

```

=====
Injection Date   : 09/08/1999 1:40:26 PM           Seq. Line :    7
Sample Name     : cal 2      0.250 µg/ml           Vial      :    6
Acq. Operator   : Henry Shoemaker                  Inj       :    1
                                                    Inj Volume:    2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           :      Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          :      1.0000
Dilution            :      1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.336	BP	44.14570	5.54228e-3	2.44668e-1		TMS

Totals : 2.44668e-1

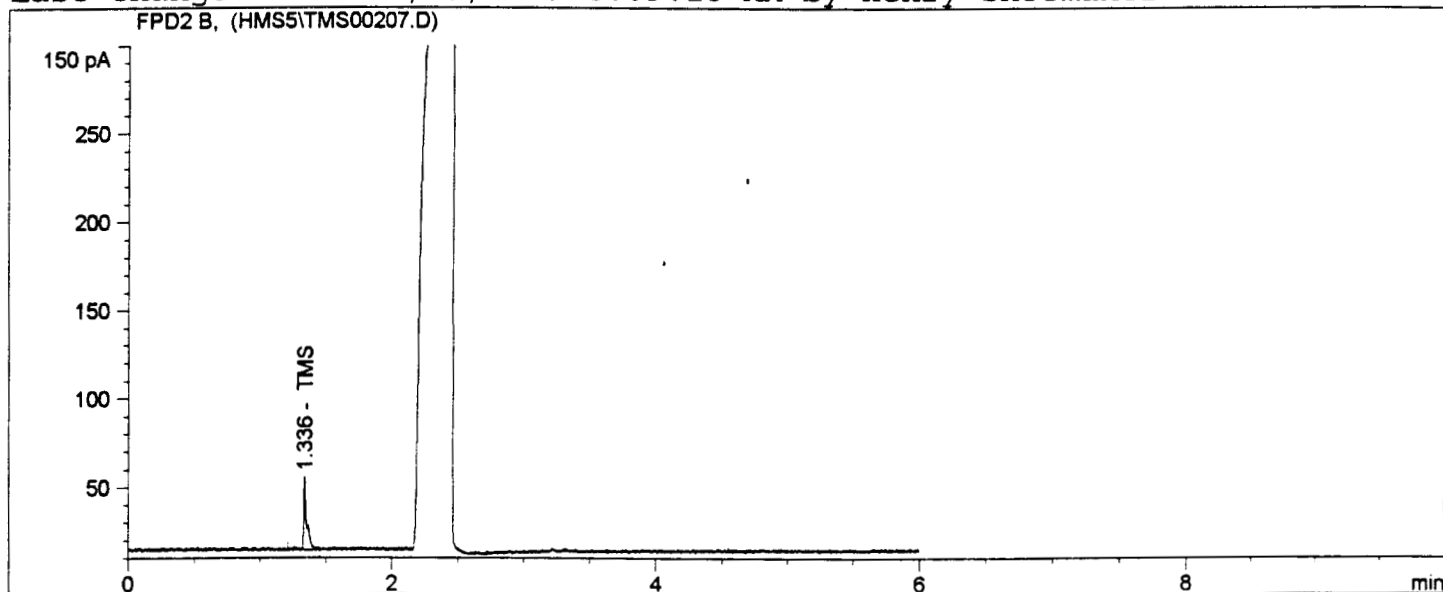
Results obtained with enhanced integrator!

*** End of Report ***


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=====
Injection Date   : 09/08/1999 1:47:25 PM           Seq. Line :    8
Sample Name     : cal 2      0.250 µg/ml           Vial      :    6
Acq. Operator   : Henry Shoemaker                  Inj       :    1
                                                    Inj Volume:    2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier         : 1.0000
Dilution           : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.336	BP	41.47395	5.72473e-3	2.37427e-1		TMS

Totals : 2.37427e-1

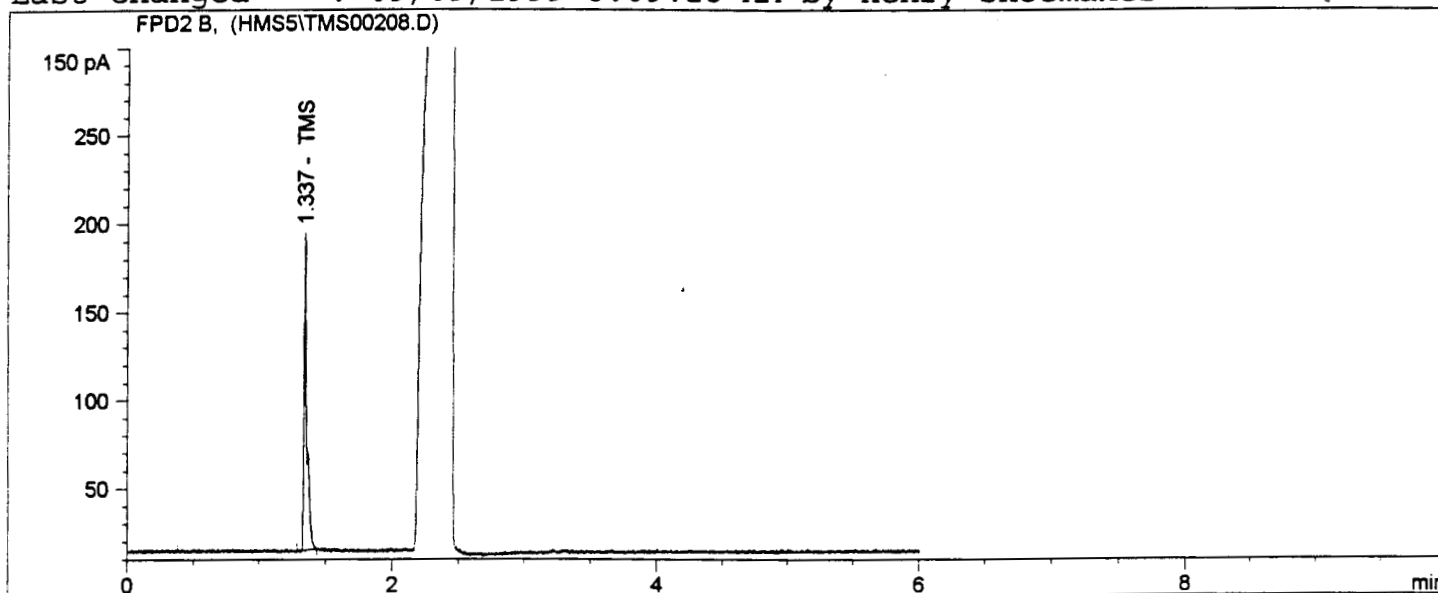
Results obtained with enhanced integrator!

*** End of Report ***

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=====
Injection Date   : 09/08/1999 1:54:24 PM           Seq. Line :    9
Sample Name     : cal 3      0.500 µg/ml           Vial      :    7
Acq. Operator   : Henry Shoemaker                 Inj       :    1
                                                    Inj Volume:  2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.337	PB	181.21370	2.66382e-3	4.82720e-1		TMS

Totals : 4.82720e-1

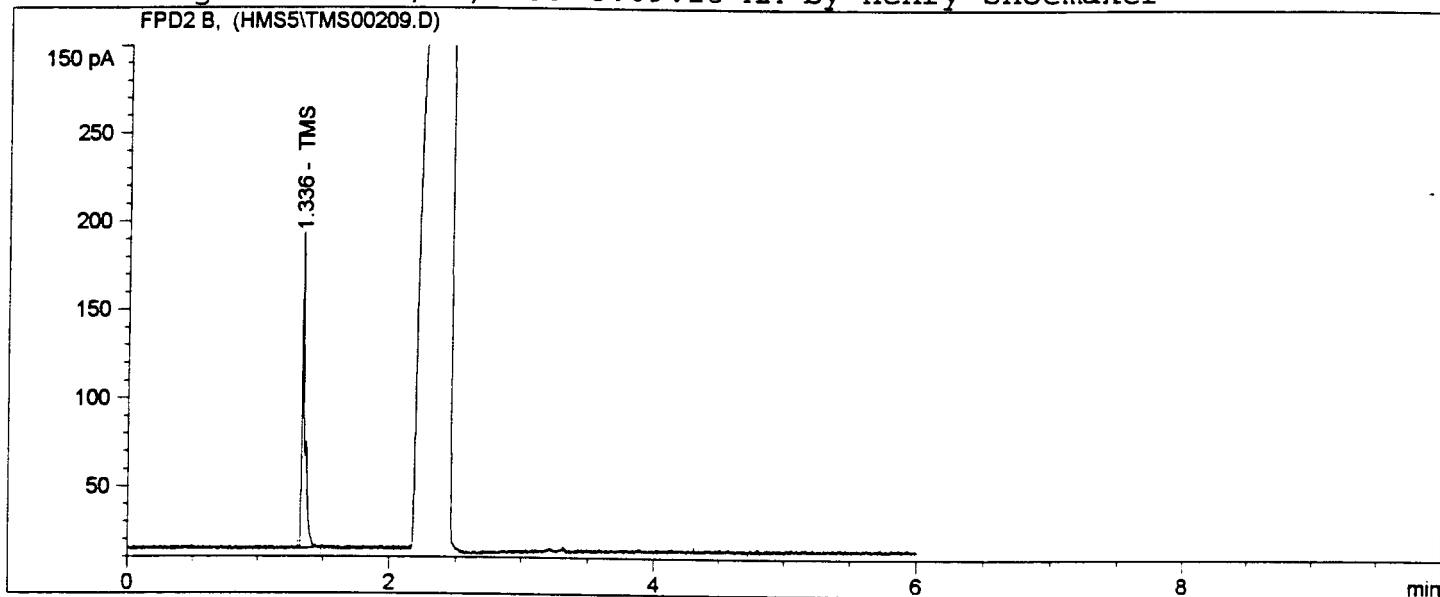
Results obtained with enhanced integrator!

*** End of Report ***

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=====
Injection Date   : 09/08/1999 2:01:23 PM           Seq. Line :   10
Sample Name     : cal 3      0.500 µg/ml           Vial      :    7
Acq. Operator   : Henry Shoemaker                  Inj       :    1
                                                    Inj Volume:  2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
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External Standard Report
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Sorted By           :      Signal
Calib. Data Modified :      09/09/1999 8:09:13 AM
Multiplier          :      1.0000
Dilution            :      1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.336	BB	176.14017	2.70335e-3	4.76169e-1		TMS

Totals : 4.76169e-1

Results obtained with enhanced integrator!

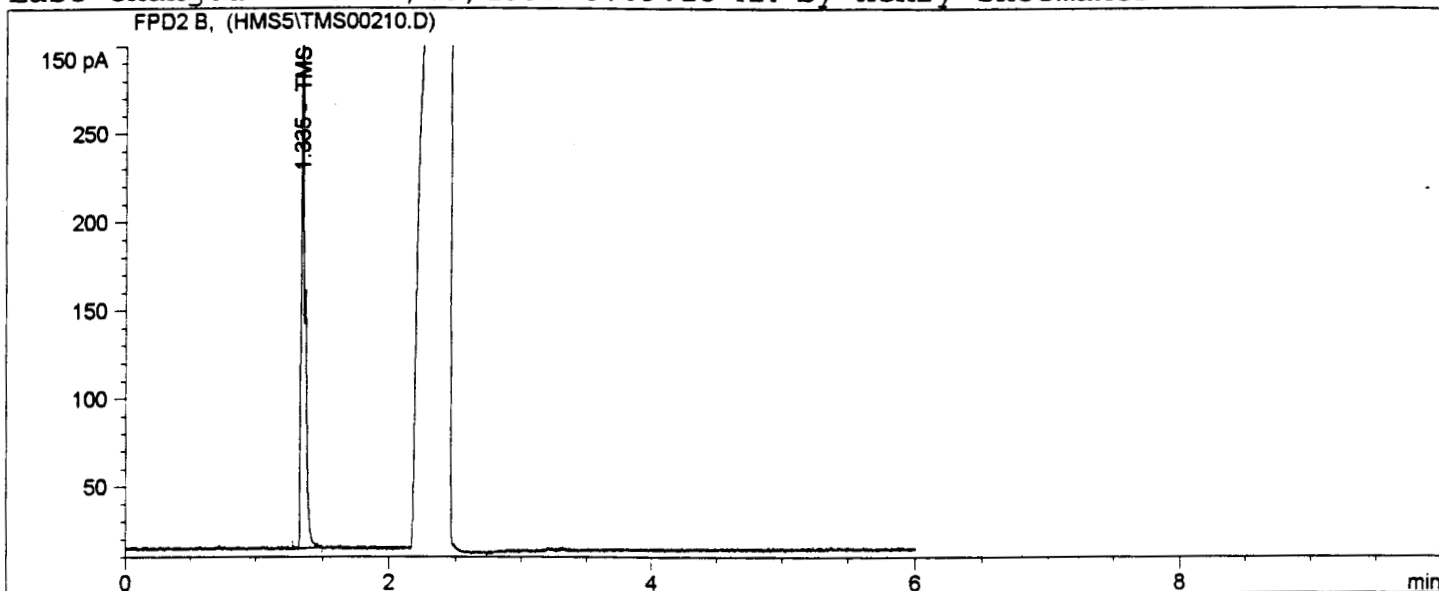
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*** End of Report ***

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=====
Injection Date   : 09/08/1999 2:08:24 PM           Seq. Line : 11
Sample Name     : cal 4      0.750 µg/ml           Vial      : 8
Acq. Operator   : Henry Shoemaker                 Inj       : 1
                                                    Inj Volume: 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
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External Standard Report
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Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000

```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	BB	452.96198	1.65610e-3	7.50152e-1		TMS

```
Totals :                               7.50152e-1
```

Results obtained with enhanced integrator!

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*** End of Report ***
=====

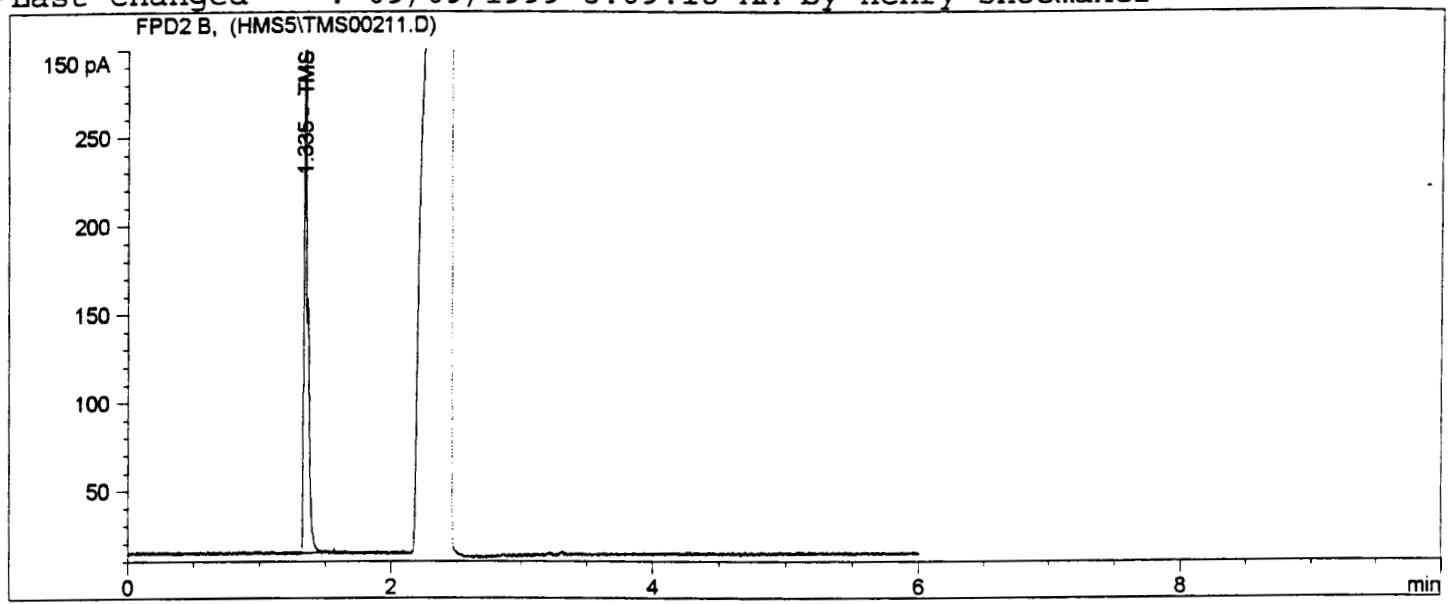
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=====
Injection Date   : 09/08/1999 2:15:25 PM           Seq. Line : 12
Sample Name     : cal 4      0.750 µg/ml           Vial      : 8
Acq. Operator   : Henry Shoemaker                 Inj       : 2
                                                    Inj Volume: 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
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External Standard Report
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Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000

```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	BB	461.43347	1.64026e-3	7.56871e-1	TMS	

Totals : 7.56871e-1

Results obtained with enhanced integrator!

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*** End of Report ***
=====

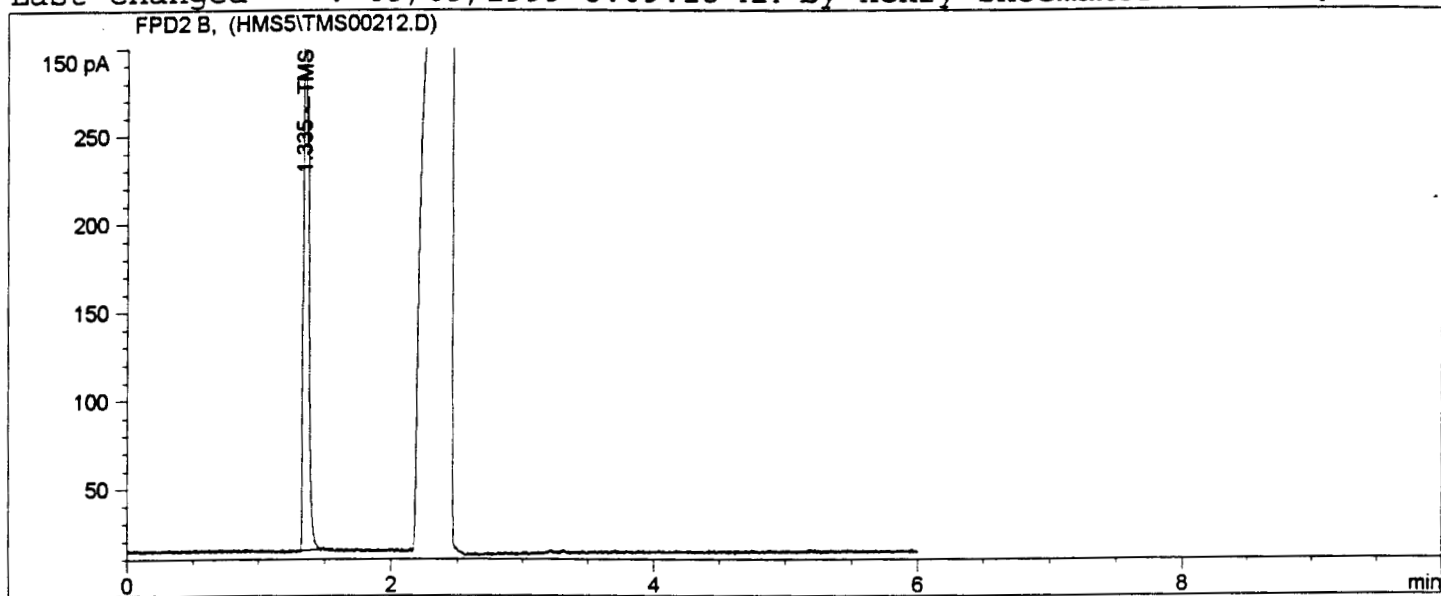
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=====
Injection Date   : 09/08/1999 2:22:24 PM           Seq. Line : 13
Sample Name     : cal 5      1.00 µg/ml           Vial      : 9
Acq. Operator   : Henry Shoemaker                 Inj       : 1
                                                    Inj Volume: 2 ul
    
```

```

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
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External Standard Report

```

Sorted By      : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier    : 1.0000
Dilution      : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	BB	843.28717	1.19965e-3	1.01165		TMS

Totals : 1.01165

Results obtained with enhanced integrator!

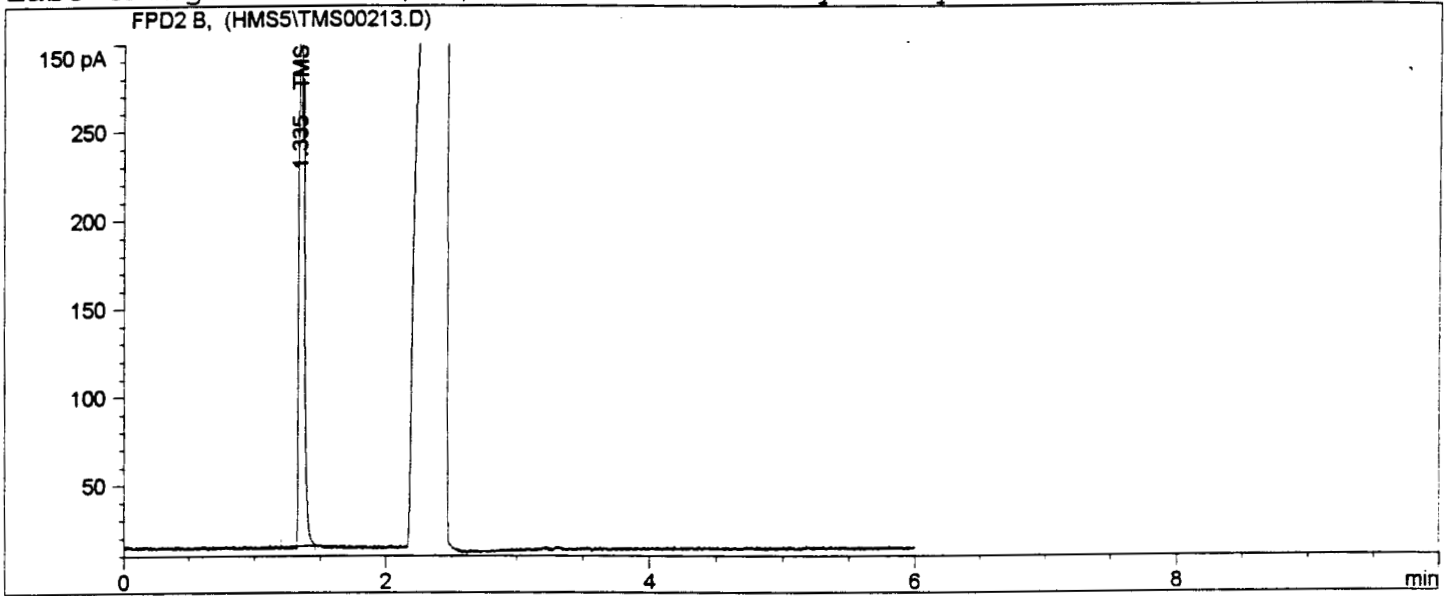
*** End of Report ***

```

=====
Injection Date   : 09/08/1999 2:29:24 PM      Seq. Line : 14
Sample Name     : cal 5      1.00 µg/ml      Vial      : 9
Acq. Operator   : Henry Shoemaker           Inj       : 2
                                           Inj Volume: 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier         : 1.0000
Dilution           : 1.0000

```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	PB	934.05133	1.13769e-3	1.06266	TMS	

Totals : 1.06266

Results obtained with enhanced integrator!

```

=====
*** End of Report ***
=====

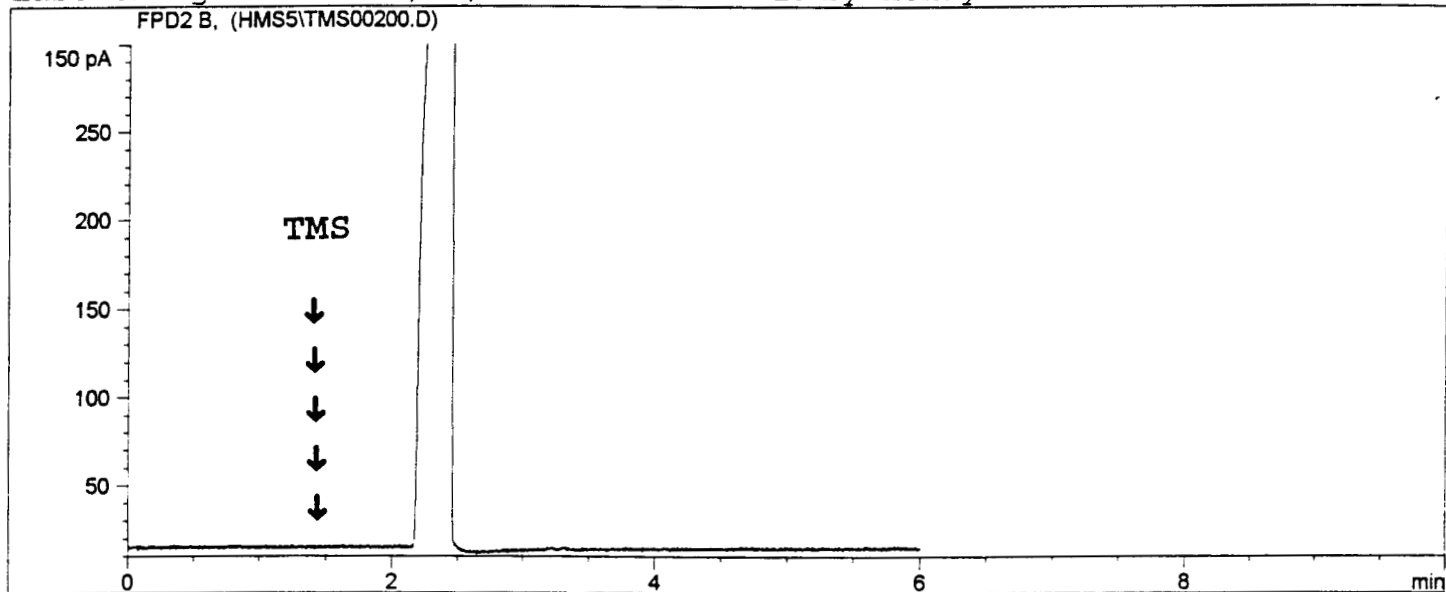
```

Matrix Blank

```

=====
Injection Date   : 09/08/1999 12:58:23 PM      Seq. Line :    1
Sample Name     : TS 01 UNFORTIFIED SOIL MATRIX  Vial      :    1
Acq. Operator   : Henry Shoemaker              Inj       :    1
                                                    Inj Volume: 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 10:29:15 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By      : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier     : 1.0000
Dilution       : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	-	-	-	-	-	TMS

Totals : 0.00000

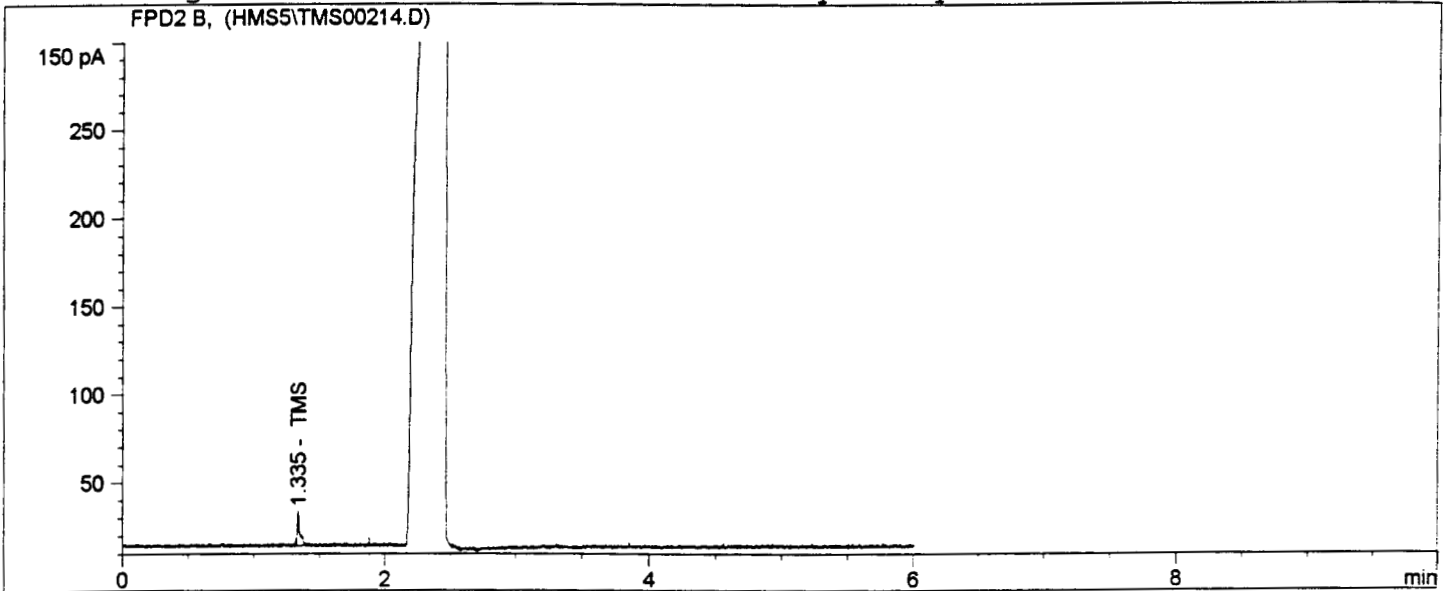
Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found


```

=====
Injection Date   : 09/08/1999 2:36:25 PM           Seq. Line   : 15
Sample Name     : TS 05 FORTIFIED AT 0.025 ppm      Vial        : 10
Acq. Operator   : Henry Shoemaker                  Inj         : 1
                                                    Inj Volume  : 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	PB	18.81901	8.62576e-3	1.62328e-1		TMS

Totals : 1.62328e-1

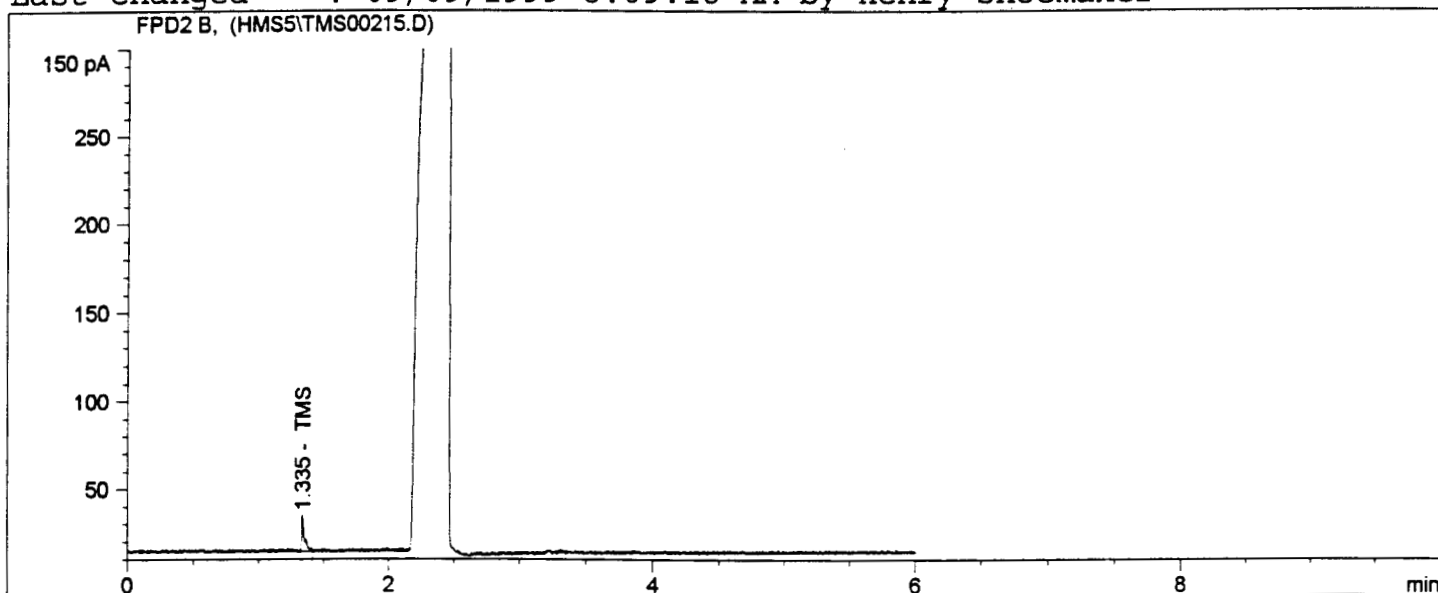
Results obtained with enhanced integrator!

*** End of Report ***

```

=====
Injection Date   : 09/08/1999 2:43:27 PM           Seq. Line :   16
Sample Name     : TS 05 FORTIFIED AT 0.025 ppm      Vial       :   10
Acq. Operator   : Henry Shoemaker                  Inj        :    1
                                                    Inj Volume : 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	BP	19.87597	8.38465e-3	1.66653e-1		TMS

Totals : 1.66653e-1

Results obtained with enhanced integrator!

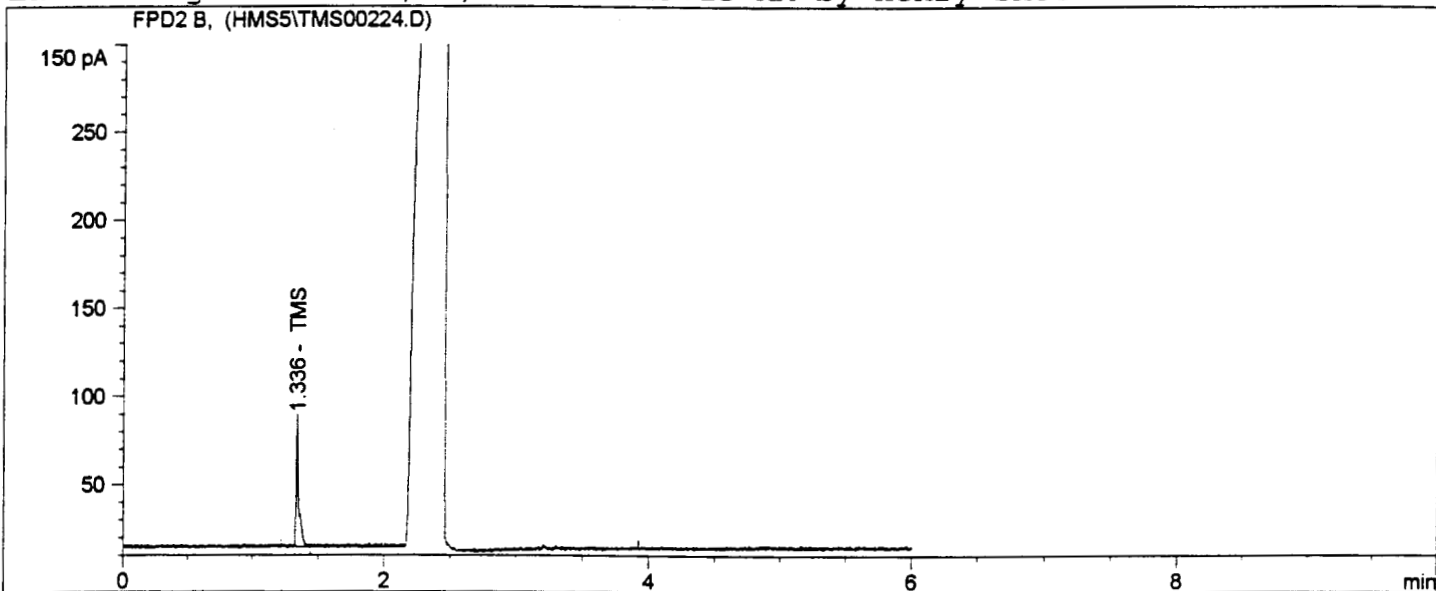
*** End of Report ***

```

=====
Injection Date   : 09/08/1999 3:46:31 PM           Seq. Line :   25
Sample Name     : TS 10      FORTIFIED AT 0.05 ppm   Vial      :   15
Acq. Operator   : Henry Shoemaker                   Inj       :    1
                                                    Inj Volume:  2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 10:29:15 AM by Henry Shoemaker
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           :      Signal
Calib. Data Modified :      09/09/1999 8:09:13 AM
Multiplier         :      1.0000
Dilution           :      1.0000

```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.336	BP	72.93948	4.27121e-3	3.11540e-1		TMS

Totals : 3.11540e-1

Results obtained with enhanced integrator!

```

=====
*** End of Report ***
=====

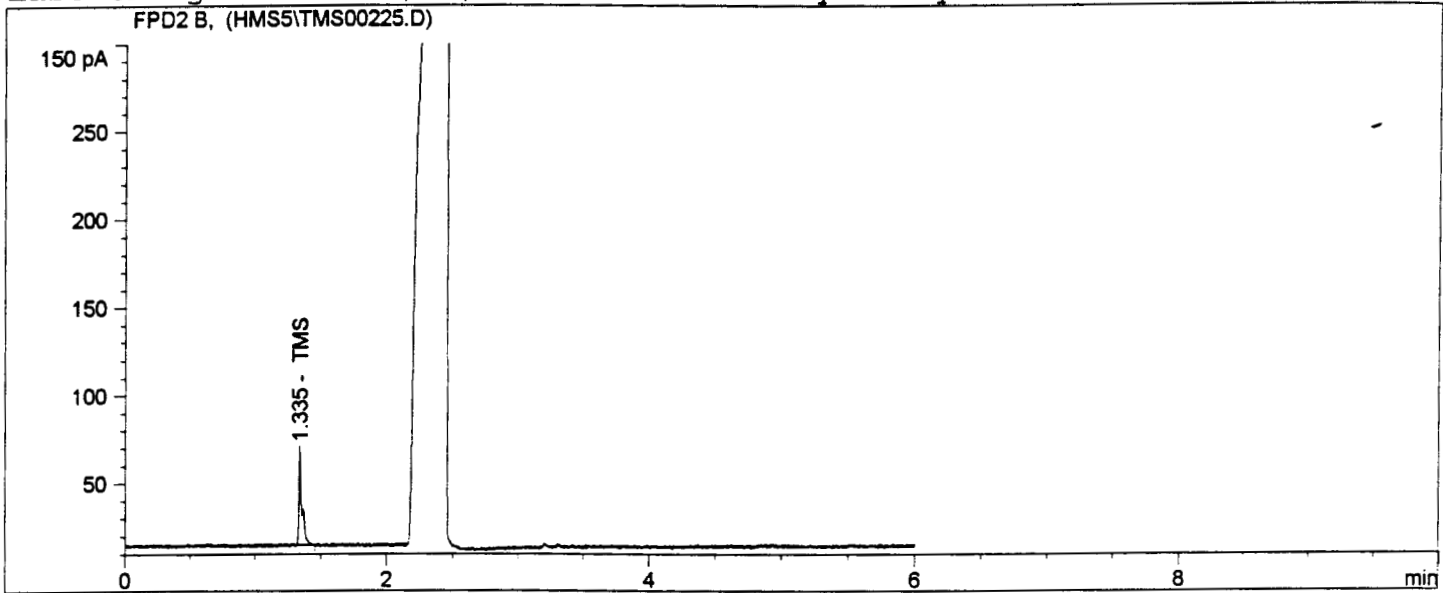
```

```

=====
Injection Date   : 09/08/1999 3:53:31 PM           Seq. Line :   26
Sample Name     : TS 10      FORTIFIED AT 0.05 ppm   Vial       :   15
Acq. Operator   : Henry Shoemaker                   Inj        :    1
                                                    Inj Volume : 2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000

```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.335	BB	55.22371	4.93448e-3	2.72500e-1		TMS

Totals : 2.72500e-1

Results obtained with enhanced integrator!

```

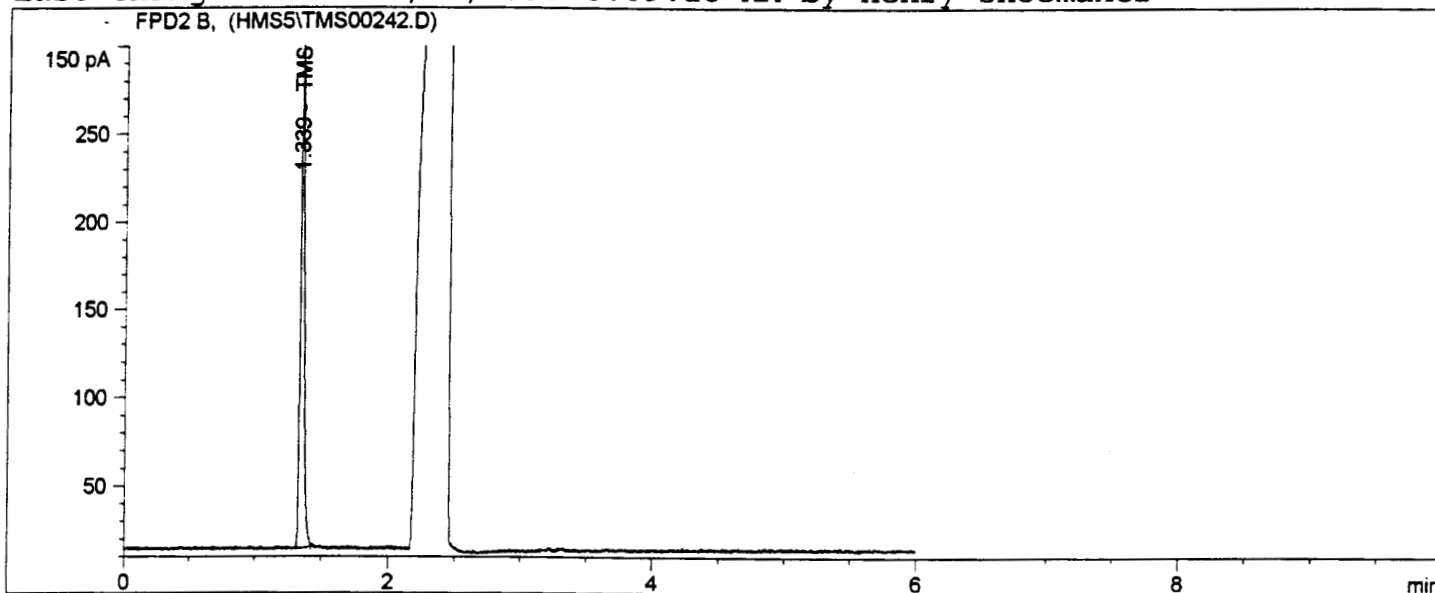
=====
*** End of Report ***
=====

```

```

=====
Injection Date   : 09/08/1999 5:52:28 PM           Seq. Line :   43
Sample Name     : TS 14 FORTIFIED AT 0.50 ppm      Vial      :   24
Acq. Operator   : Henry Shoemaker                 Inj       :    1
                                                    Inj Volume:   2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
    
```



External Standard Report

```

Sorted By           : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier          : 1.0000
Dilution            : 1.0000
    
```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.339	PB	290.75491	2.08437e-3	6.06042e-1		TMS

Totals : 6.06042e-1

Results obtained with enhanced integrator!

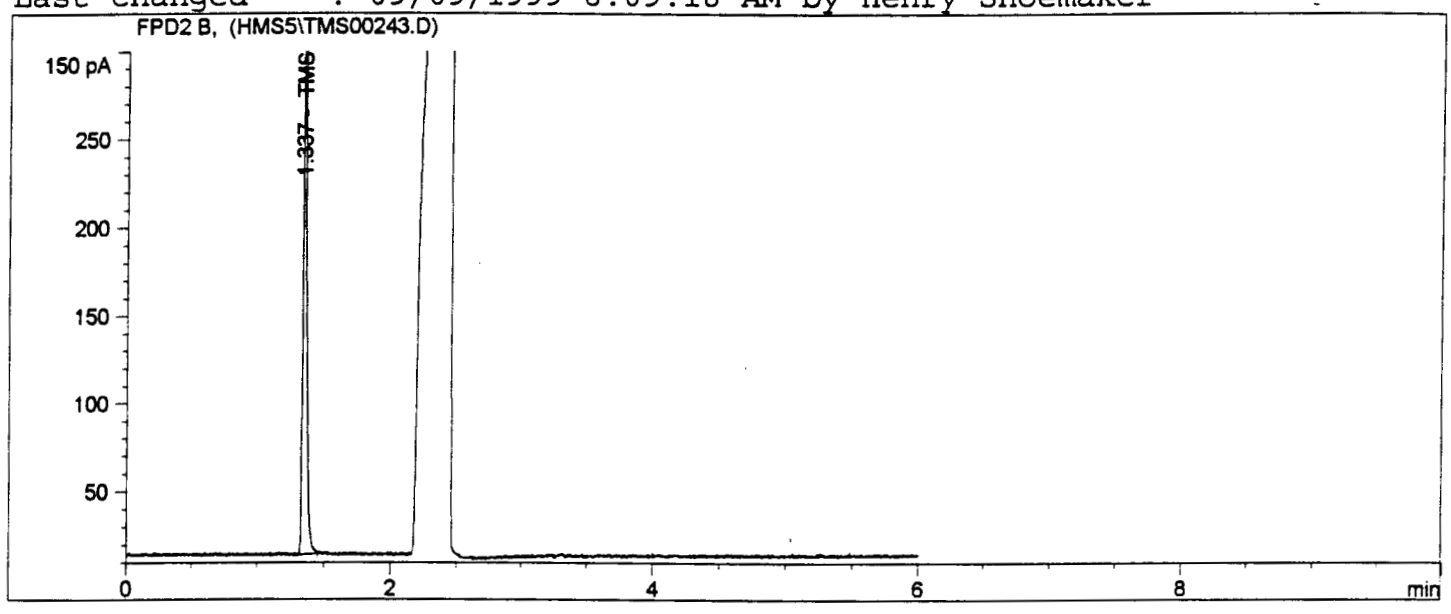
*** End of Report ***

```

=====
Injection Date   : 09/08/1999 5:59:27 PM      Seq. Line :   44
Sample Name     : TS 14 FORTIFIED AT 0.50 ppm  Vial      :   24
Acq. Operator   : Henry Shoemaker             Inj       :    2
                                           Inj Volume:   2 ul

Acq. Method     : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/08/1999 12:56:57 PM by Henry Shoemaker
Analysis Method : C:\HPCHEM\3\METHODS\TMS.M
Last changed    : 09/09/1999 8:09:18 AM by Henry Shoemaker
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : 09/09/1999 8:09:13 AM
Multiplier    : 1.0000
Dilution      : 1.0000

```

Signal 1: FPD2 B,

RetTime [min]	Type	Height [150 pA]	Amt/Height	Amount [ug/ml]	Grp	Name
1.337	PB	359.59552	1.86679e-3	6.71291e-1		TMS

Totals : 6.71291e-1

Results obtained with enhanced integrator!

```

=====
*** End of Report ***
=====

```

APPENDIX I

Touchdown*

BP: ZENECA Agrochemicals (Touchdown*)

Identification

COMMON NAME: Glyphosate-trimesium (ISO draft, BSI).

TRIVIAL NAME: Sulfosate.

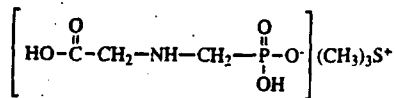
EXP. CODE NUMBERS: ICIA-0224, SC-0224.

OTHER CODE NUMBERS: CAS 81591-81-3; SHA 128501.

Chemistry

COMPOSITION: N-phosphonomethylglycine trimethylsulfonium salt.

PROPERTIES: Vapor pressure 3×10^{-10} Torr (25°C). Specific gravity 1.23 (20°C/20°C). Clear, straw to brown color. Density (20°C) 1.23 - 1.25 g/ml. Viscosity 26.6 centistokes (5°C).



Glyphosate-trimesium

A&L Analytical Laboratories, Inc.

411 North Third Street • Memphis, TN 38105 • (901) 527-2780 • FAX (901) 526-1031



SOIL ANALYSIS

NOV - 6 1995

CLIENT U.S. EPA/ECS BLDG. 1105 STENNIS SPACE CNTR, MS 3952	GROWER U.S. EPA/ECS	REPORT 305-0 DATE 11/03 ACCOUNT 15 PAGE
	DATE RECEIVED: 10/31/95	A & L AGRONOMIST Richard Larz

LAB NUMBER 01087

SAMPLE ID IOWA

SIGNATURE *Richard Larz*

TEST	RESULTS	SOIL TEST RATINGS					CATION EXCHA CAPACITY
		Very Low	Low	Medium	High	Very High	
Soil pH	6.3						19.6 meq/100g
Buffer pH	6.74						
Phosphorus (P)	6 ppm						CATION SATURATION %K 1.0 %Ca 68.0 %Mg 19.5 %H 10.0 %Na .0
Potassium (K)	93 ppm						
Calcium (Ca)	2690 ppm						
Magnesium (Mg)	459 ppm						
Sulphur (S)							
Boron (B)							SOIL TEST METHOD AMMONIUM ACETATE EXTRACT IIC
Copper (Cu)							
Iron (Fe)							
Manganese (Mn)							
Zinc (Zn)							
Sodium (Na)	11 ppm						
Soluble Salts	0.34 mmho/cm						
Organic Matter	3.4 % ENR 112						
NO ₃ -N							

* ADD'L RESULTS TO FOLLOW

SOIL FERTILITY GUIDELINES

CROP:

YIELD GOAL:

LIME	N	P ₂ O ₅	K ₂ O	Mg	S	B	Cu	Mn	Zn

CLIENT: U.S. EPA/ECS

REPORT OF ANALYSIS

REPORT DATE: 11/03/95
DATE RECEIVED: 10/31/95

LAB NO	SAMPLE IDENTIFICATION	PERCENT SAND	PERCENT SILT	PERCENT CLAY	TEXTURAL CLASSIFICATION
01087	IOWA	42	30	28	CLAY LOAM