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JUN 17 1998

MEMORANDUM

SUBJECT: Tribufos (DEF) and Dibutyldisulfide Method  
Evaluation Report No. ECM0128S1-S2

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

TO: Elizabeth Leovey, Chief  
EFED/Environmental Risk Branch II (H7507C)

THRU: Donald A. Marlow, Lab Coordinator *DM*  
BEAD/(H7503W)

The EFED/Environmental Risk Branch II has requested Environmental Chemistry Lab Evaluations for Tribufos (DEF) and Dibutyldisulfide in soil, MRID #433255/02, using a method submitted by the Agriculture Division of Miles Inc., entitled "The Determination of DEF and Dibutyldisulfide Residues in Soil Samples". *42350006*

This method was evaluated with soil fortified at three levels, 3.0, 10.0 and 100 ppb, and with quadruplicate analyses at each level.

The attached method evaluation reports include three parts:

Part I: Summary and conclusions

In this section any problems encountered with the method and how they were handled are discussed. ECS'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 these reports, please contact Henry Shoemaker at (601) 688-1222 or me at (601) 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 0128S1-S2

A GAS CHROMATOGRAPHY METHOD FOR THE  
DETERMINATION OF RESIDUES OF TRIBUFOS  
AND DIBUTYLDISULFIDE IN SOIL.

ENVIRONMENTAL CHEMISTRY LABORATORY (ECL)

ENVIRONMENTAL CHEMISTRY BRANCH

BIOLOGICAL AND ECONOMIC ANALYSIS DIVISION

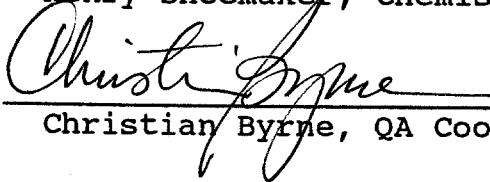
05/11/98

PREPARED BY:



Henry Shoemaker, Chemist/ECL

REVIEWED BY:



Christian Byrne, QA Coordinator/ECL

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PART I

SUMMARY AND CONCLUSION

We have completed an Environmental Chemistry Method Evaluation of Tribufos(DEF) and Dibutyldisulfide(DBDS) in soil. This method, MRID# 433255-02, submitted by the Agriculture Division of Miles Inc., is titled "The Determination of DEF and Dibutyldisulfide Residues in Soil Samples".

In order to evaluate this method we fortified a soil matrix with DEF and Dibutyldisulfide at 3.0, 10.0 and 100 ppb. All samples were done in replicates of four at each level. The registrant did not specify a method limit of detection (LOD), therefore we estimated the (LOD) to be 3.0 ppb. The Miles method limit of quantitation (LOQ) of 10 ppb of each analyte in soil was confirmed by our data. 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 9.3% at the (LOQ) of 10 ppb for DEF and 4.9% at the 100 ppb level. The mean recoveries for DEF of 79% at the 10 ppb level and 85% at the 100 ppb level are well within the target range of 70% to 120%.

The precision of the Dibutyldisulfide analyses was also within limits, 1.7% RSD and 4.0% RSD at 10 ppb and 100 ppb, respectively. The mean recovery at the LOQ of 10 ppb was 74% and was 79% at the 100 ppb level. All of our data matched very well with that of the registrant.

This method involves a triple extraction of soil samples by shaking with acetone/hexane solvent, followed by a florisil clean-up. The resulting sample extract is evaporated under a stream of nitrogen to an appropriate volume and analyzed by FPD Gas Chromatography.

PART II

ANALYTICAL RESULTS FOR TRIBUFOS(DEF)

EPA RECOVERIES IN SOIL

Sample	Added (ppb)	Found (ppb)	Recovery (%)	Statistics
BLK 01	0	0		
BLK 02	0	0		
BLK 03	0	0		
BLK 04	0	0		
SOIL 01	3.00	2.79	93.0	mean(Rec) = 66.8% sd = 20.2 rsd = 30.2%
SOIL 02	3.00	2.16	72.0	
SOIL 03	3.00	1.46	48.7	
SOIL 04	3.00	1.60	53.3	
SOIL 05	10.0	8.56	85.6	mean(Rec) = 78.7% sd = 7.35 rsd = 9.34%
SOIL 06	10.0	7.90	79.0	
SOIL 07	10.0	8.16	81.6	
SOIL 08	10.0	6.84	68.4	
SOIL 09	100.0	79.0	79.0	mean(Rec) = 84.6% sd = 4.12 rsd = 4.88%
SOIL 10	100.0	85.7	85.7	
SOIL 11	100.0	88.9	88.9	
SOIL 12	100.0	84.6	84.6	

PART II  
ANALYTICAL RESULTS FOR DIBUTYLDISULFIDE  
EPA RECOVERIES IN SOIL

Sample	Added (ppb)	Found (ppb)	Recovery (%)	Statistics
BLK 01	0	0		
BLK 02	0	0		
BLK 03	0	0		
BLK 04	0	0		
SOIL 01	3.00	2.64	88.0	mean(Rec) = 69.9% sd = 12.63 rsd = 18.1%
SOIL 02	3.00	1.94	64.7	
SOIL 03	3.00	2.03	67.7	
SOIL 04	3.00	1.77	59.0	
SOIL 05	10.0	7.30	73.0	mean(Rec) = 73.6% sd = 1.211 rsd = 1.65%
SOIL 06	10.0	7.54	75.4	
SOIL 07	10.0	7.32	73.2	
SOIL 08	10.0	7.28	72.8	
SOIL 09	100.0	82.3	82.3	mean(Rec) = 79.0% sd = 3.156 rsd = 3.99%
SOIL 10	100.0	79.5	79.5	
SOIL 11	100.0	79.5	79.5	
SOIL 12	100.0	74.7	74.7	

PART III  
EXPERIMENTAL SUMMARY

(a) Method Procedure

A 100 gram soil sample was extracted by shaking with three portions of 5% acetone in hexane. The sample extract was filtered and then dried by passing through anhydrous sodium sulfate. The extract was reduced by roto-evaporation to approximately 5 ml, transferred to the barrel of a 10 ml syringe and then passed through a florisil sep-pac. The syringe barrel and sep-pac were rinsed with about 5 ml of extraction solvent. The final solution was reduced under nitrogen to 2 ml for samples SOIL01 thru SOIL08 and 5 ml for samples SOIL09 thru SOIL12. Gas chromatographic(GC) analysis was performed using a GC equipped with flame photometric detector(sulfur mode) and a fused silica capillary DB-225 column.

(b) Source of Analytical Reference Standard

Analytical standards were obtained from the Bayer Corporation, Agriculture Division, Stillwell, Kansas (formerly Miles, Inc.) and certified to be DEF at 98.6% purity and Dibutylidissulfide at 98% purity.

(c) Source of Sample Matrix

The soil(batch I) used was obtained from Iowa State University and was 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 (FPD) in the sulfur mode.
2. Column used was capillary DB-225, 15 m x 0.53 mm with 1.0  $\mu$ m film thickness (J & W Scientific).



(e) Modification of Method

Manual injection was used instead of an autosampler as specified in the method.

(f) Calculations

1. Standard Curve

Since FPD is a non-linear detector in the sulfur mode, a Ln\Ln (concentration\area) plot was made with a set of calibration standards consisting of four concentrations, 250 ng/ml, 500 ng/ml, 1000 ng/ml, and 2000 ng/ml. Linear regression was used to calculate the linear equation;

$$\text{Ln } Y = m(\text{Ln } X) + b$$

Where: Y = peak area  
X = concentration (ng/ml)  
m = slope of line  
b = Y-intercept

2. Calculation of Analytes in Samples

The calibration equation generated by the calibration standards in the sample set is used to solve for the concentration by substituting known quantities.

$$\text{Analyte conc(ng/ml)} = e^a$$

where:  $a = (\text{Ln } Y - b)/m$

Then to find the concentration in ppb use the equation:

$$\text{ppb} = \{\text{conc(ng/ml)}\} \{\text{extract vol./ sple wt.}\}$$

3. Example Calculation

Sample no. SOIL 05 for Tribufos (DEF)

Calibration equation is  $\ln Y = 1.660(\ln X) - 4.711$

Peak area = 210.0

Sample wt. = 100g

Extract vol. = 2 ml

$$\begin{aligned}\text{Conc}(\text{ng/ml}) &= e^{(5.347 + 4.711)/(1.660)} \\ \text{conc.} &= e^{6.0590} \\ \text{conc.} &= 428 \text{ ng/ml}\end{aligned}$$

Then to find ppb

$$\begin{aligned}\text{ppb} &= (428 \text{ ng/ml}) (2 \text{ ml} / 100\text{g}) \\ \text{ppb} &= 8.56\end{aligned}$$

(g) Graphs and Data

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

1. Pages 9 thru 20, calibration curves and raw data for calibration standards.
2. Pages 21 thru 24, chromatograms and raw data for selected samples.

### LINEAR REGRESSION ANALYSIS

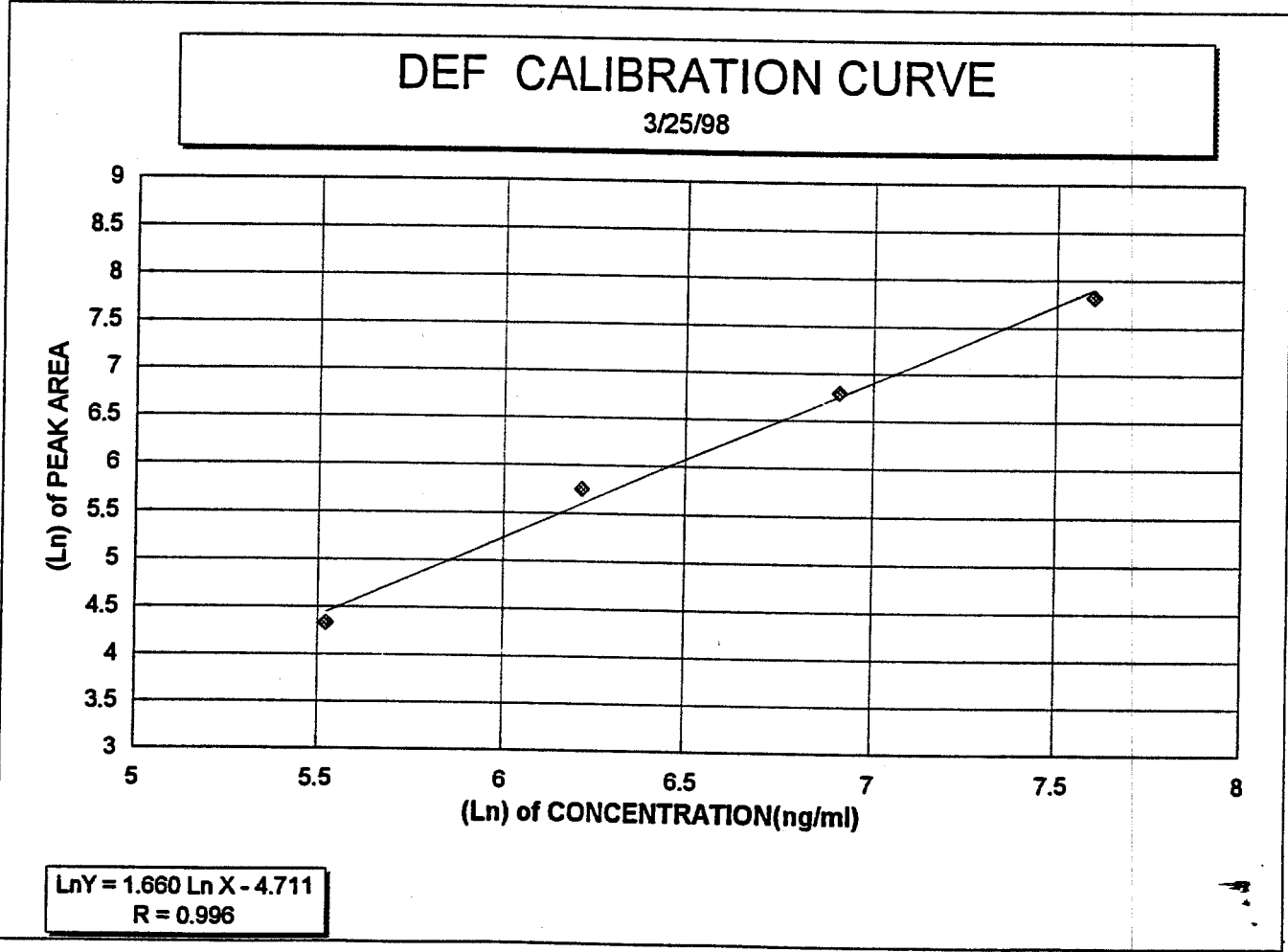
Std.(ng/ml)	Ln(conc)	pk.area	Ln of area	<u>Predicted</u>
250	5.521	76.24	4.334	4.452
500	6.215	317.01	5.759	5.603
1000	6.908	893.51	6.795	6.753
2000	7.601	2499.83	7.824	7.904

I Regression Output:

Constant	-4.711
Std Err of Y Est	0.152
R Squared	0.993
No. of Observations	4.000
Degrees of Freedom	2.000

X Coefficient(s)	1.660
Std Err of Coef.	0.098



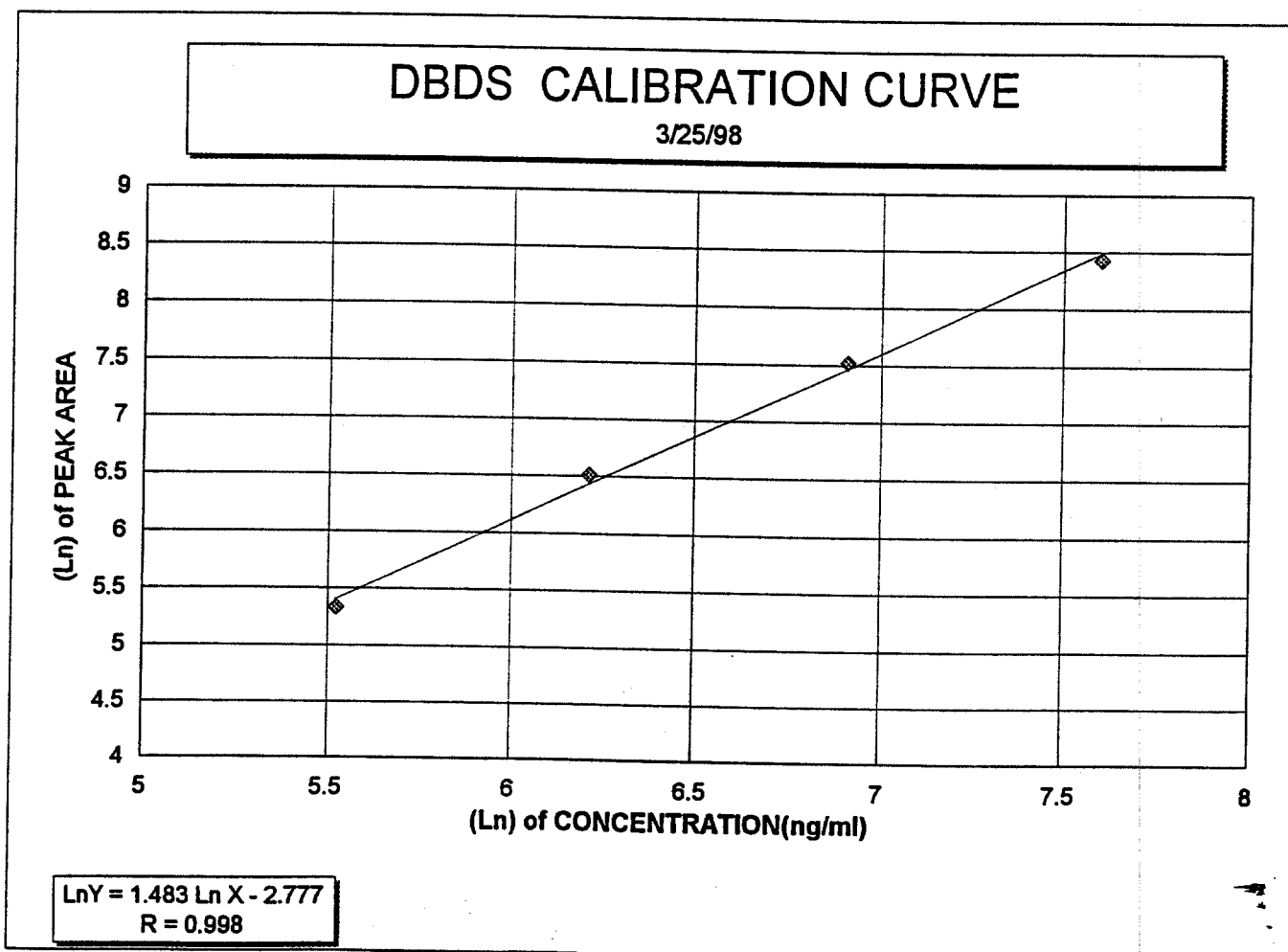
**LINEAR REGRESSION ANALYSIS**

Std.(ng/ml)	Ln (conc)	pk.area	Ln of area	Predicted
250	5.521	207.91	5.337	5.408
500	6.215	675.34	6.515	6.437
1000	6.908	1844.84	7.52	7.464
2000	7.601	4573.92	8.428	8.491

I Regression Output:

Constant	-2.777
Std Err of Y Est	0.096
R Squared	0.997
No. of Observations	4.000
Degrees of Freedom	2.000

X Coefficient(s)	1.483
Std Err of Coef.	0.062

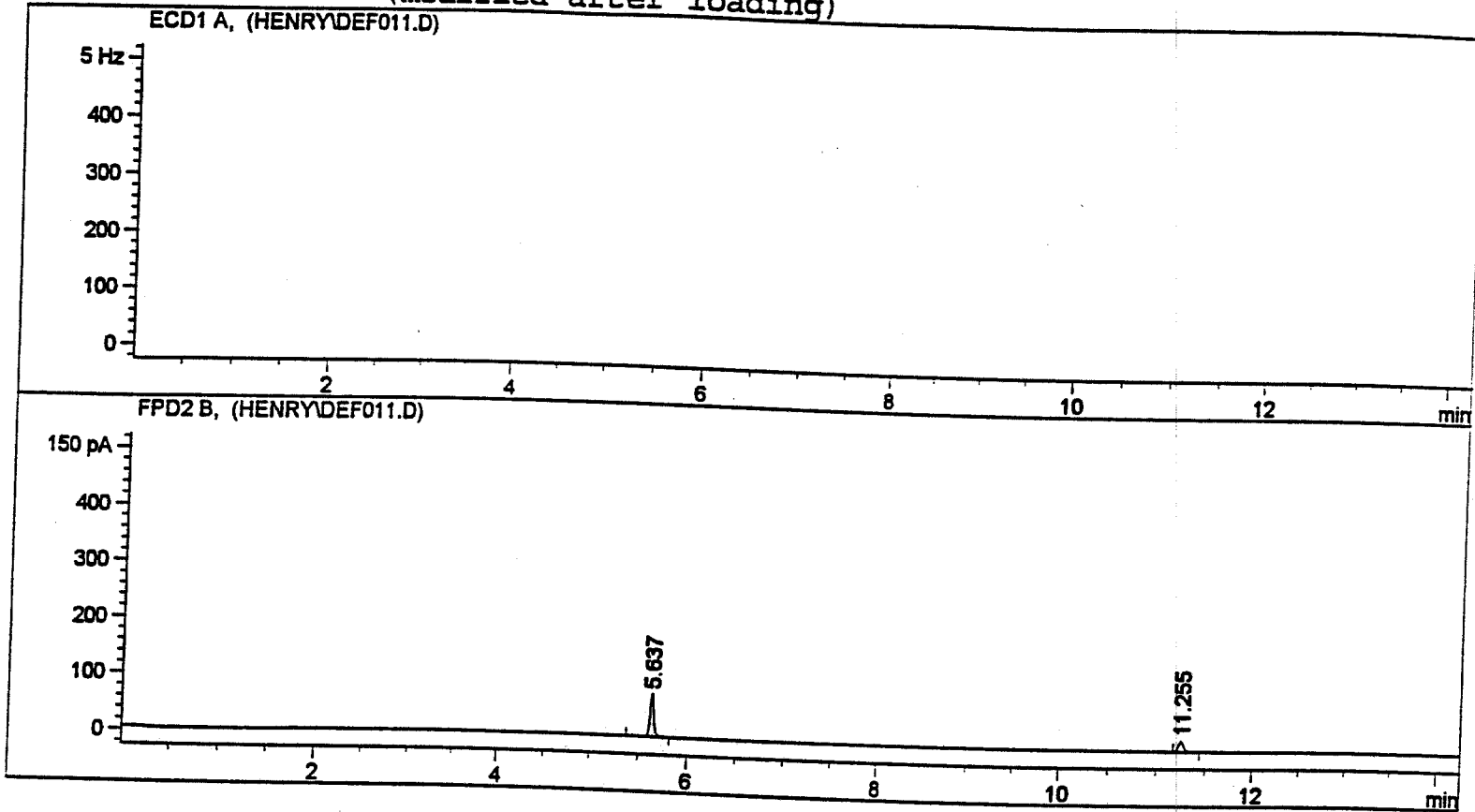


Data File C:\HPCHEM\3\DATA\HENRY\DEF011.D

Sample Name: cal std.-

```

=====
Injection Date   : 3/25/98 12:15:40 PM
Sample Name     : cal std.-1
Acq. Operator  : Henry Shoemaker
Method         : C:\HPCHEM\3\METHODS\HMS.M
Last changed   : 3/25/98 11:21:42 AM by Henry Shoemaker
                (modified after loading)
Vial           : 1
Inj           : 1
Inj Volume    : Manually
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

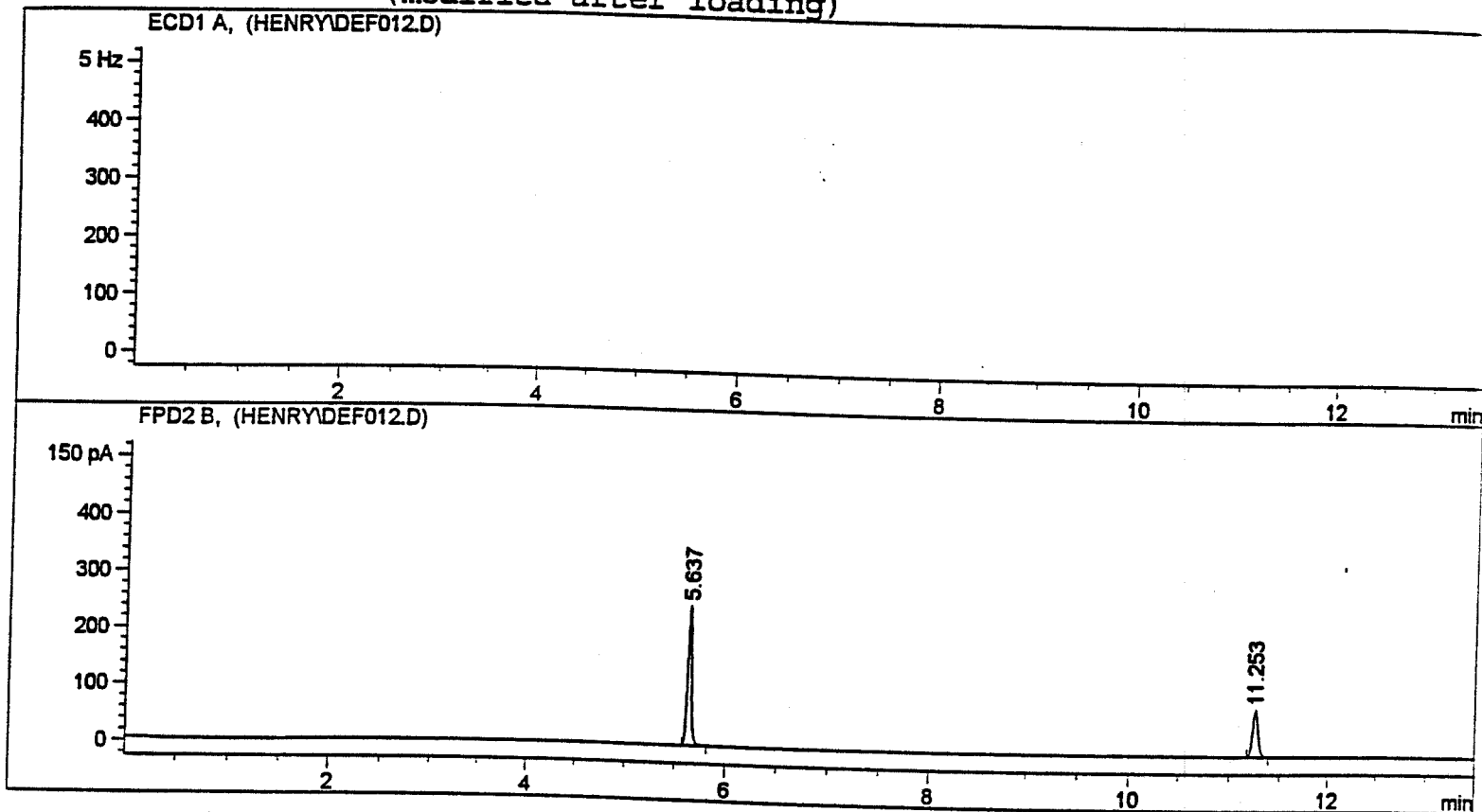
Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.637	BV	0.0429	207.90941	74.89422	73.16917	
2	11.255	BV	0.0509	76.23953	18.80073	26.83083	DBDS DEF
Totals :				284.14894	93.69495		

Data File C:\HPCHEM\3\DATA\HENRY\DEF012.D

Sample Name: cal std.-

```

=====
Injection Date   : 3/25/98 12:39:25 PM
Sample Name     : cal std.-2
Acq. Operator  : Henry Shoemaker
Method         : C:\HPCHEM\3\METHODS\HMS.M
Last changed   : 3/25/98 11:21:42 AM by Henry Shoemaker
                  (modified after loading)
Vial           : 1
Inj           : 1
Inj Volume    : Manually
    
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
    
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

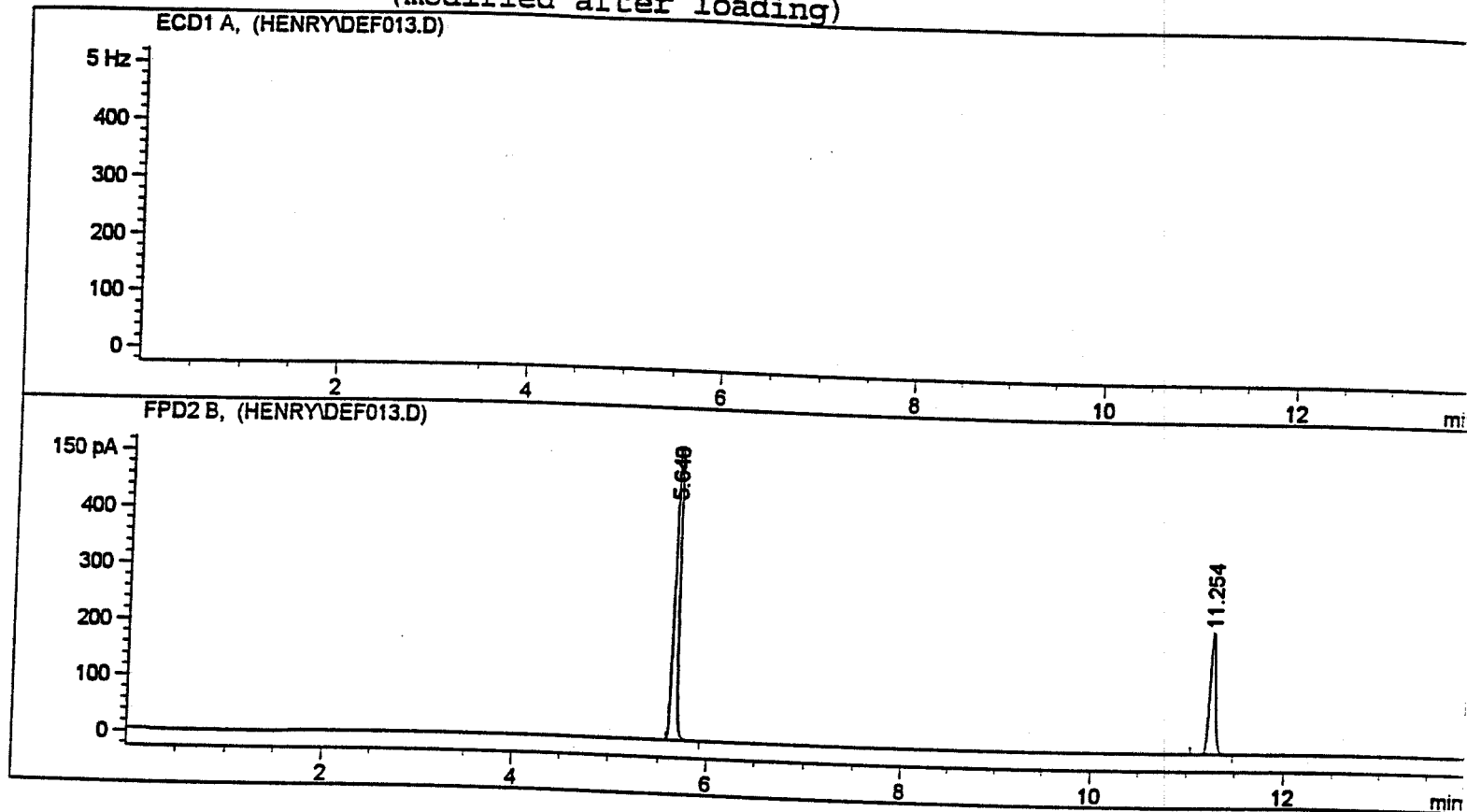
Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.637	BV	0.0421	675.34180	245.23030	68.05482	DBDS
2	11.253	VV	0.0596	317.00793	81.30119	31.94518	DEF
Totals :				992.34973	326.53149		

Data File C:\HPCHEM\3\DATA\HENRY\DEF013.D

Sample Name: cal std.

```

=====
Injection Date   : 3/25/98 1:18:21 PM
Sample Name     : cal std.-3
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/25/98 11:21:42 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj             : 1
Inj Volume      : Manually
    
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
    
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

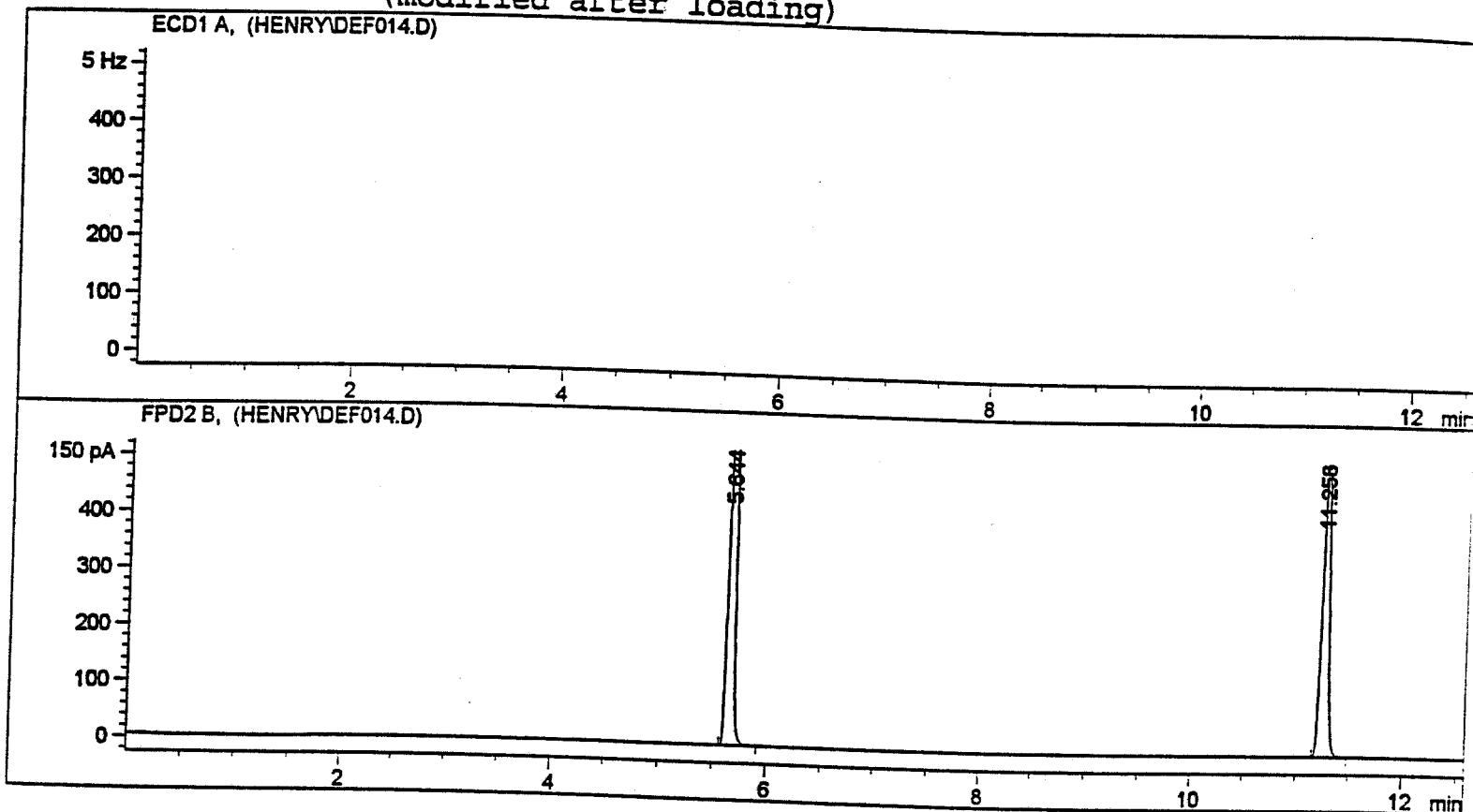
Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.640	BB	0.0450	1844.84387	634.47498	67.37060	DBDS
2	11.254	BB	0.0649	893.50769	214.66574	32.62940	DEF
Totals :				2738.35156	849.14072		

Data File C:\HPCHEM\3\DATA\HENRY\DEF014.D

Sample Name: cal std.-

```

=====
Injection Date   : 3/25/98 1:39:34 PM
Sample Name     : cal std.-4
Acq. Operator  : Henry Shoemaker
Method         : C:\HPCHEM\3\METHODS\HMS.M
Last changed   : 3/25/98 11:21:42 AM by Henry Shoemaker
                (modified after loading)
Vial           : 1
Inj           : 1
Inj Volume    : Manually
    
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
    
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.644	BB	0.0524	4573.91895	1411.13672	64.66048	DBDS
2	11.258	BB	0.0697	2499.82812	566.61407	35.33952	DEF

Totals : 7073.74707 1977.75079



### LINEAR REGRESSION ANALYSIS

Std.(ng/ml)	Ln (conc)	pk.area	Ln of area	<u>Predicted</u>
250	5.521	116.82	4.76	4.823
500	6.215	377.9	5.935	5.893
1000	6.908	1171.3	7.066	6.961
2000	7.601	2825.21	7.946	8.030

I                    Regression Output:

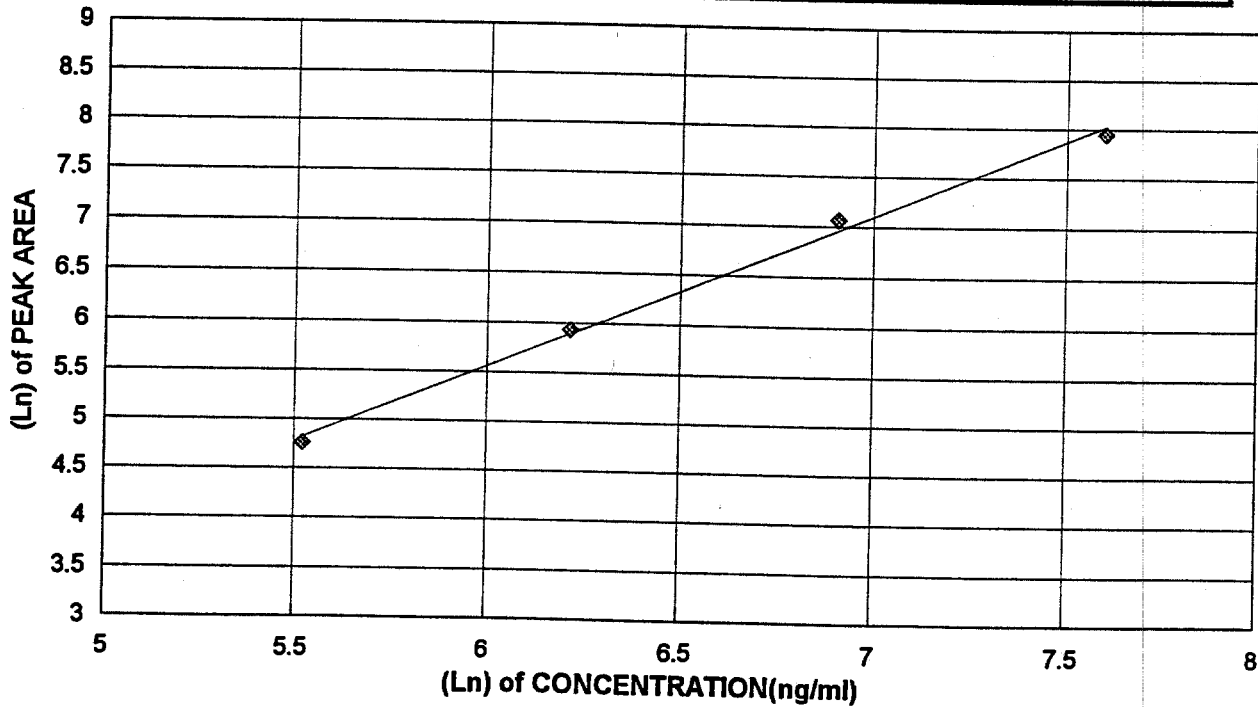
Constant	-3.689
Std Err of Y Est	0.109
R Squared	0.996
No. of Observations	4.000
Degrees of Freedom	2.000

X Coefficient(s)	1.542
Std Err of Coef.	0.070

### DEF CALIBRATION CURVE

3/26/98



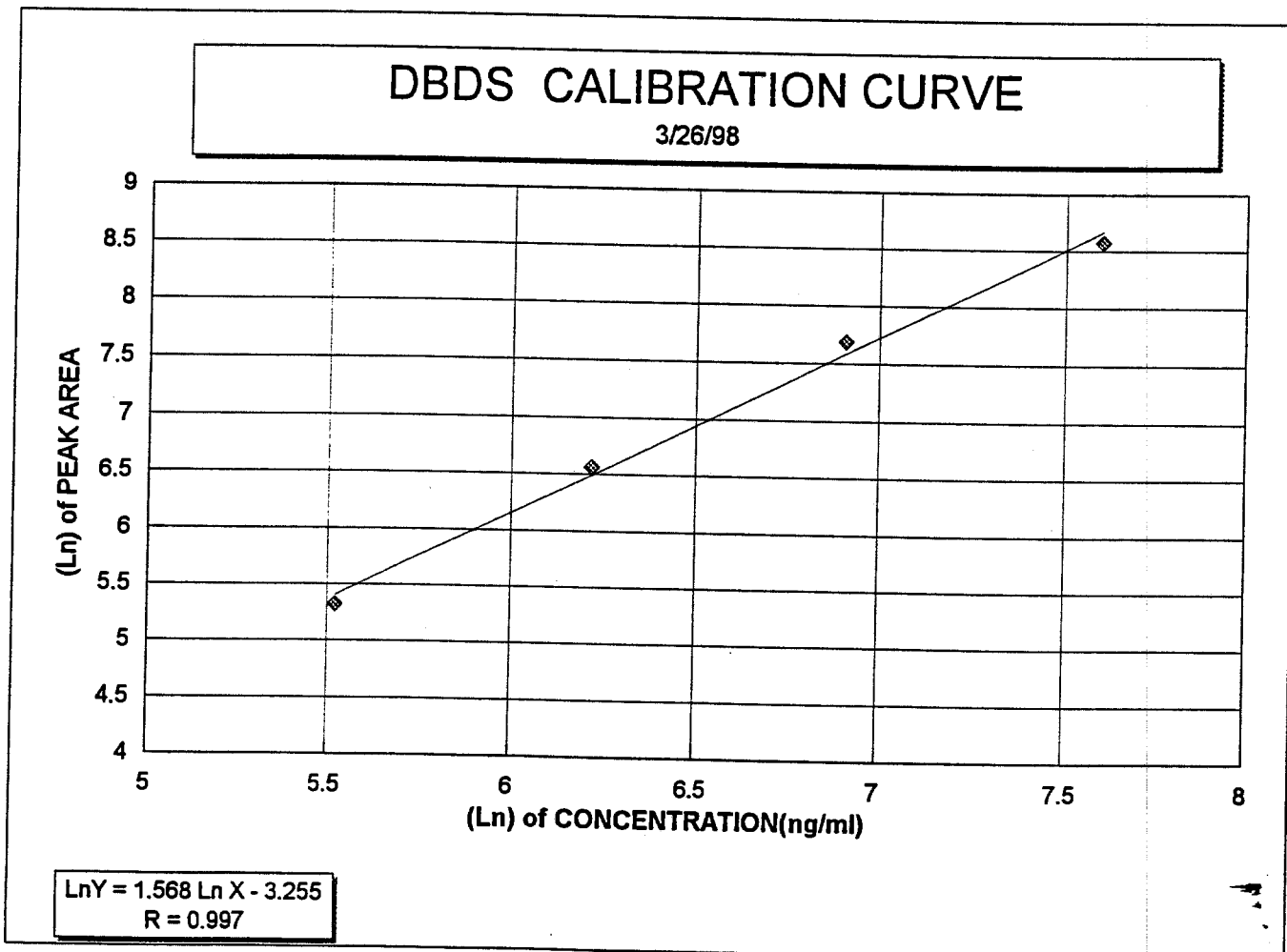
$LnY = 1.542 Ln X - 3.689$   
 $R = 0.998$

### LINEAR REGRESSION ANALYSIS

Std.(ng/ml)	Ln (conc)	pk.area	Ln of area	Predicted
250	5.521	204.93	5.323	5.405
500	6.215	710.08	6.565	6.493
1000	6.908	2170.73	7.683	7.580
2000	7.601	5298.54	8.575	8.667

Regression Output:  
 Constant -3.255  
 Std Err of Y Est 0.124  
 R Squared 0.995  
 No. of Observations 4.000  
 Degrees of Freedom 2.000

X Coefficient(s) 1.568  
 Std Err of Coef. 0.080

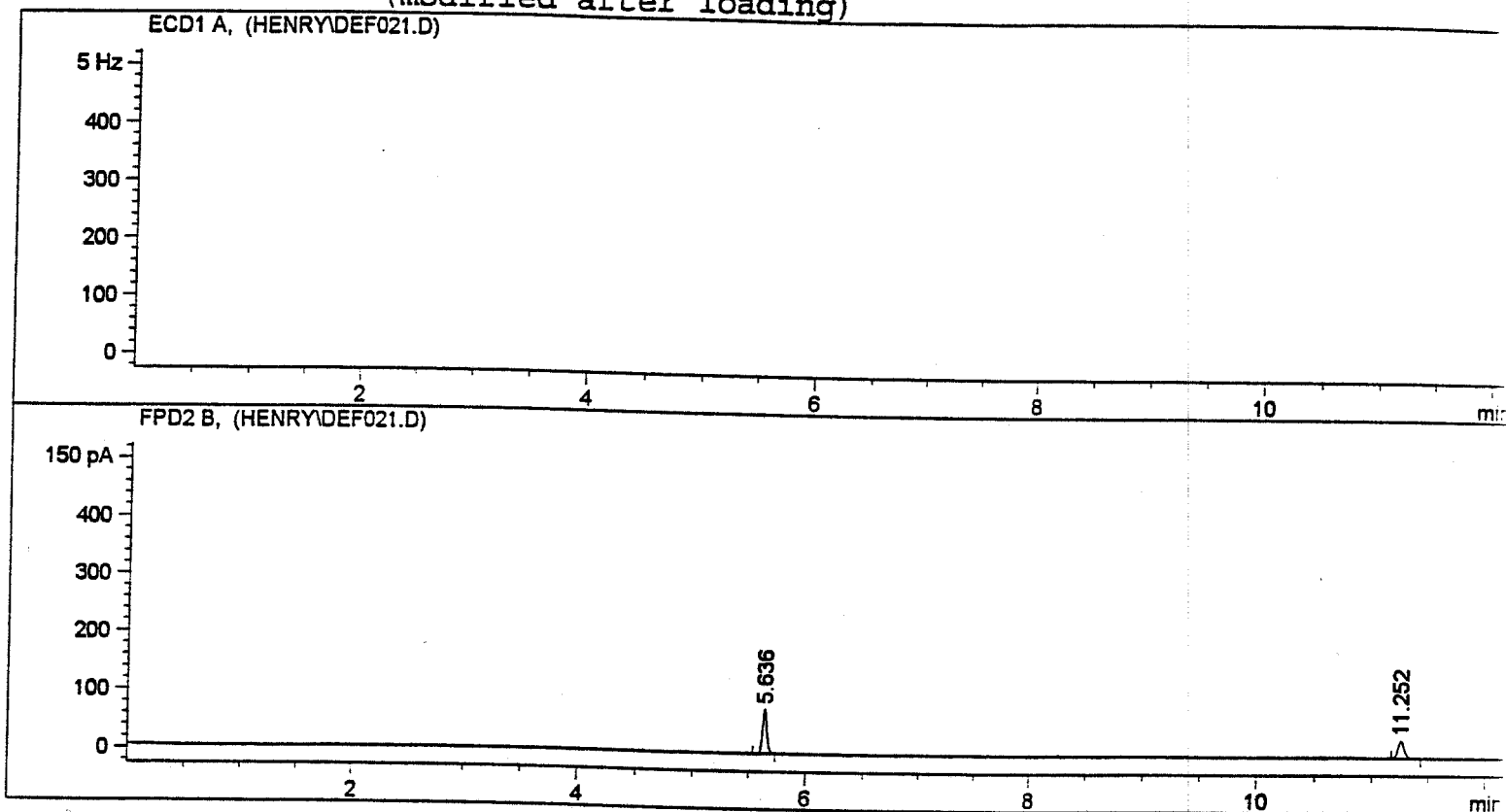


Data File C:\HPCHEM\3\DATA\HENRY\DEF021.D

Sample Name: cal std..

```

=====
Injection Date   : 3/26/98 6:17:47 AM
Sample Name     : cal std.-01
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/26/98 5:59:08 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj            : 1
Inj Volume     : Manually
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %
1	5.636	PV	0.0411	204.92728	75.58924	63.69146
2	11.252	BB	0.0564	116.82273	29.62630	36.30854

DBDS  
 DEF

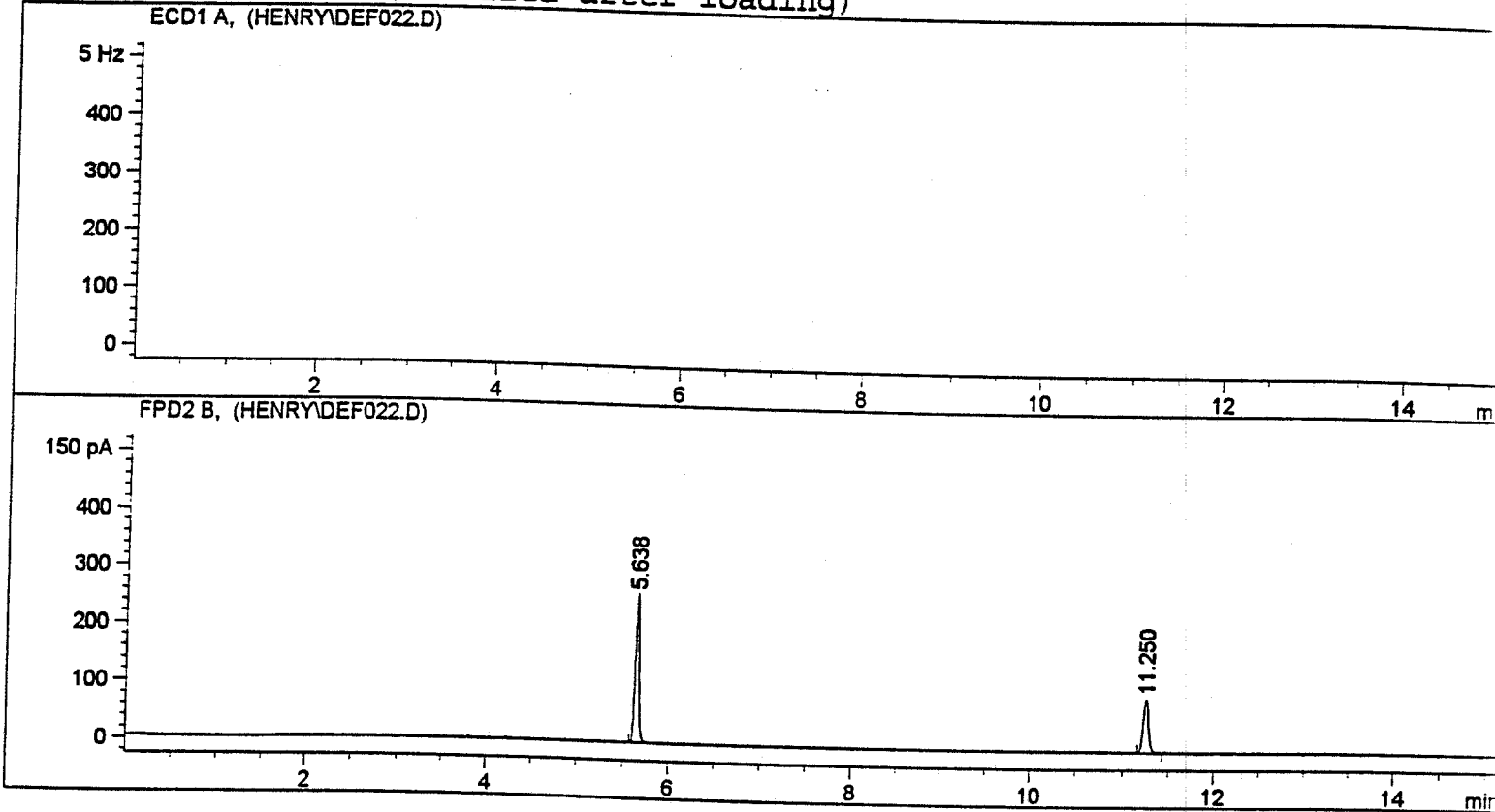
Totals : 321.75001 105.21555

Data File C:\HPCHEM\3\DATA\HENRY\DEF022.D

Sample Name: cal std.-

```

=====
Injection Date   : 3/26/98 6:38:42 AM
Sample Name     : cal std.-02
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/26/98 5:59:08 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj            : 1
Inj Volume     : Manually
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.638	BB	0.0429	710.08167	255.74754	65.26587	DBDS
2	11.250	BB	0.0616	377.90152	91.88990	34.73413	DEF

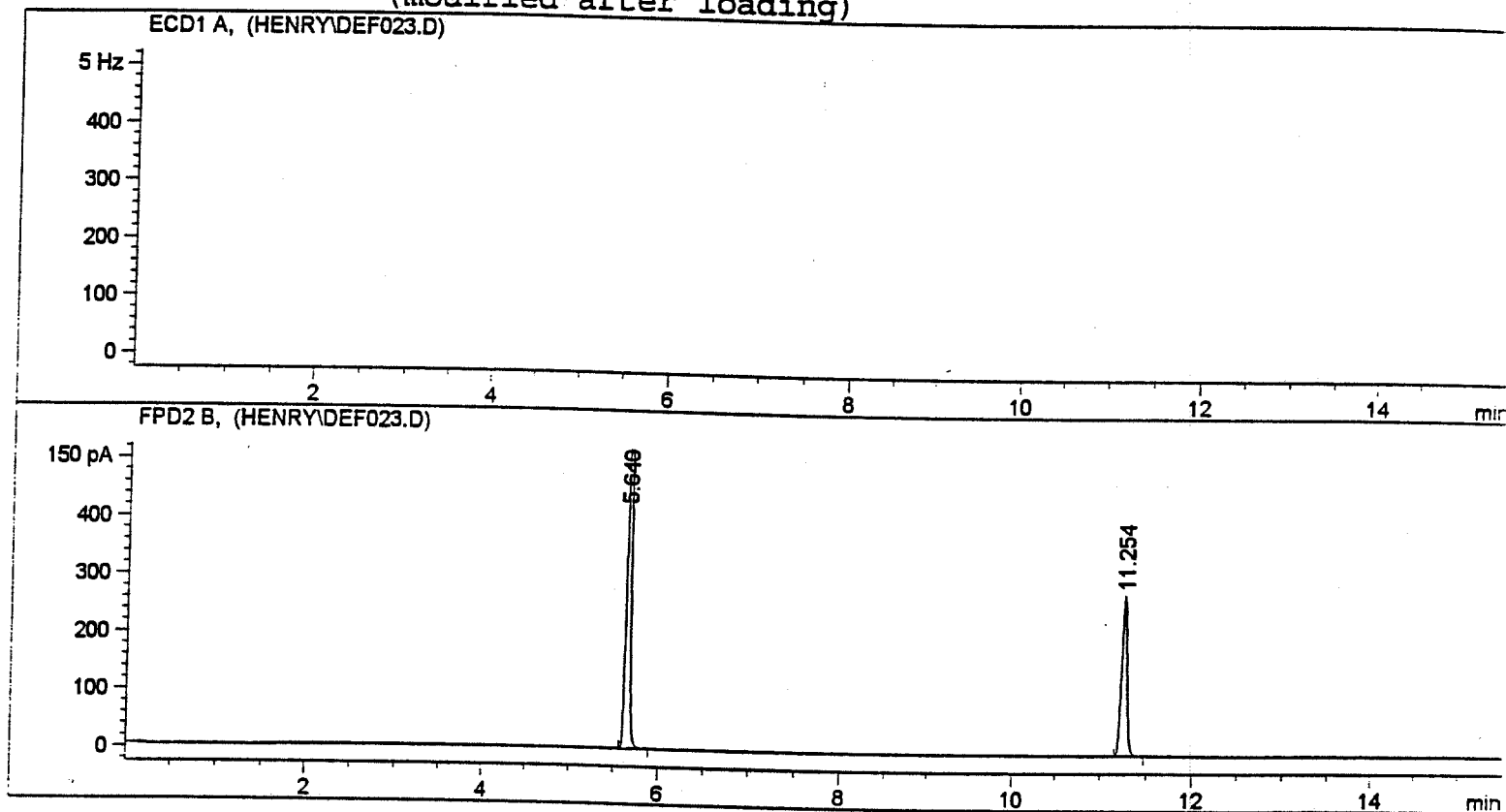
Totals 1087.98319

Data File C:\HPCHEM\3\DATA\HENRY\DEF023.D

Sample Name: cal std.-c

```

=====
Injection Date   : 3/26/98 7:03:37 AM
Sample Name     : cal std.-03
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/26/98 5:59:08 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj             : 1
Inj Volume      : Manually
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.640	BB	0.0470	2170.72998	721.73737	64.95245	DBDS
2	11.254	BB	0.0639	1171.29956	279.67935	35.04755	DEF

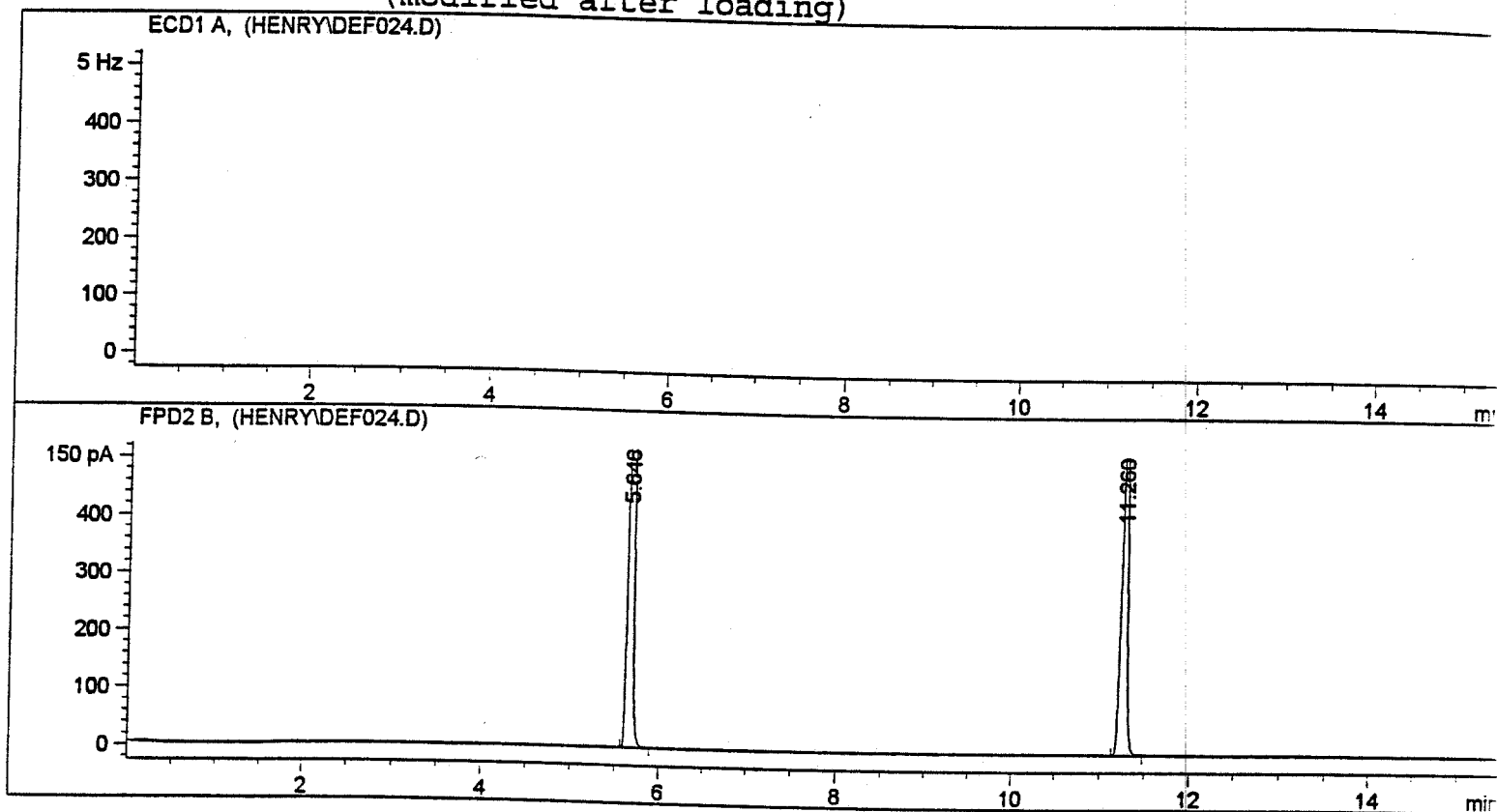
Totals : 3342.02954 1001.41672

Data File C:\HPCHEM\3\DATA\HENRY\DEF024.D

Sample Name: cal std.-

```

=====
Injection Date   : 3/26/98 7:28:05 AM
Sample Name     : cal std.-04
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/26/98 5:59:08 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj             : 1
Inj Volume      : Manually
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %
1	5.646	BB	0.0539	5298.53613	1584.14722	65.22283
2	11.260	BV	0.0686	2825.20825	639.88318	34.77717

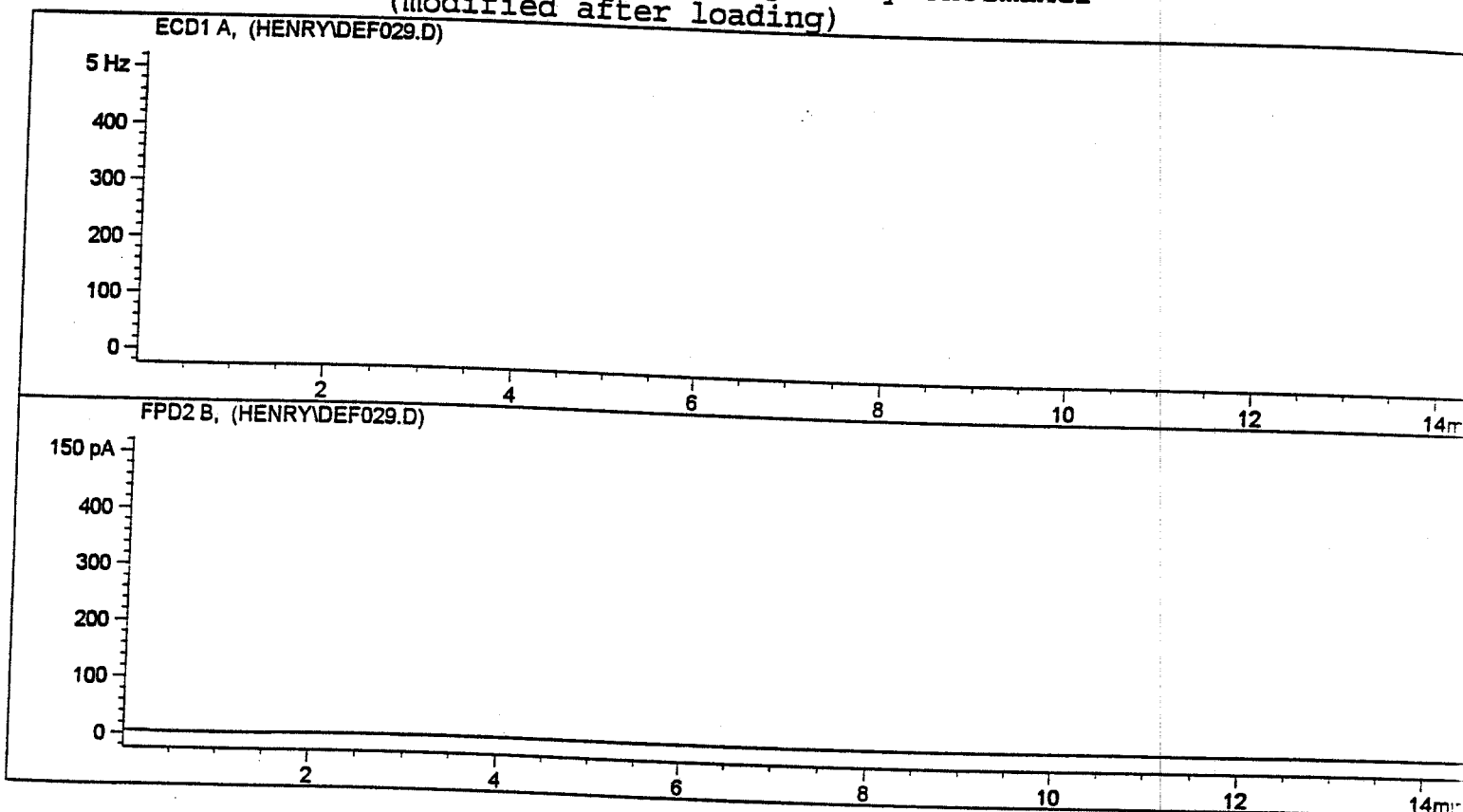
**DBDS**  
**DEF**

Totals : 8123.74438 2224.03040

Data File C:\HPCHEM\3\DATA\HENRY\DEF029.D

Sample Name: blank

=====  
Injection Date : 3/26/98 9:28:35 AM  
Sample Name : blank 01  
Acq. Operator : Henry Shoemaker  
Method : C:\HPCHEM\3\METHODS\HMS.M  
Last changed : 3/26/98 9:02:36 AM by Henry Shoemaker  
(modified after loading)  
Vial : 1  
Inj : 1  
Inj Volume : Manually



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000

No peaks found

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

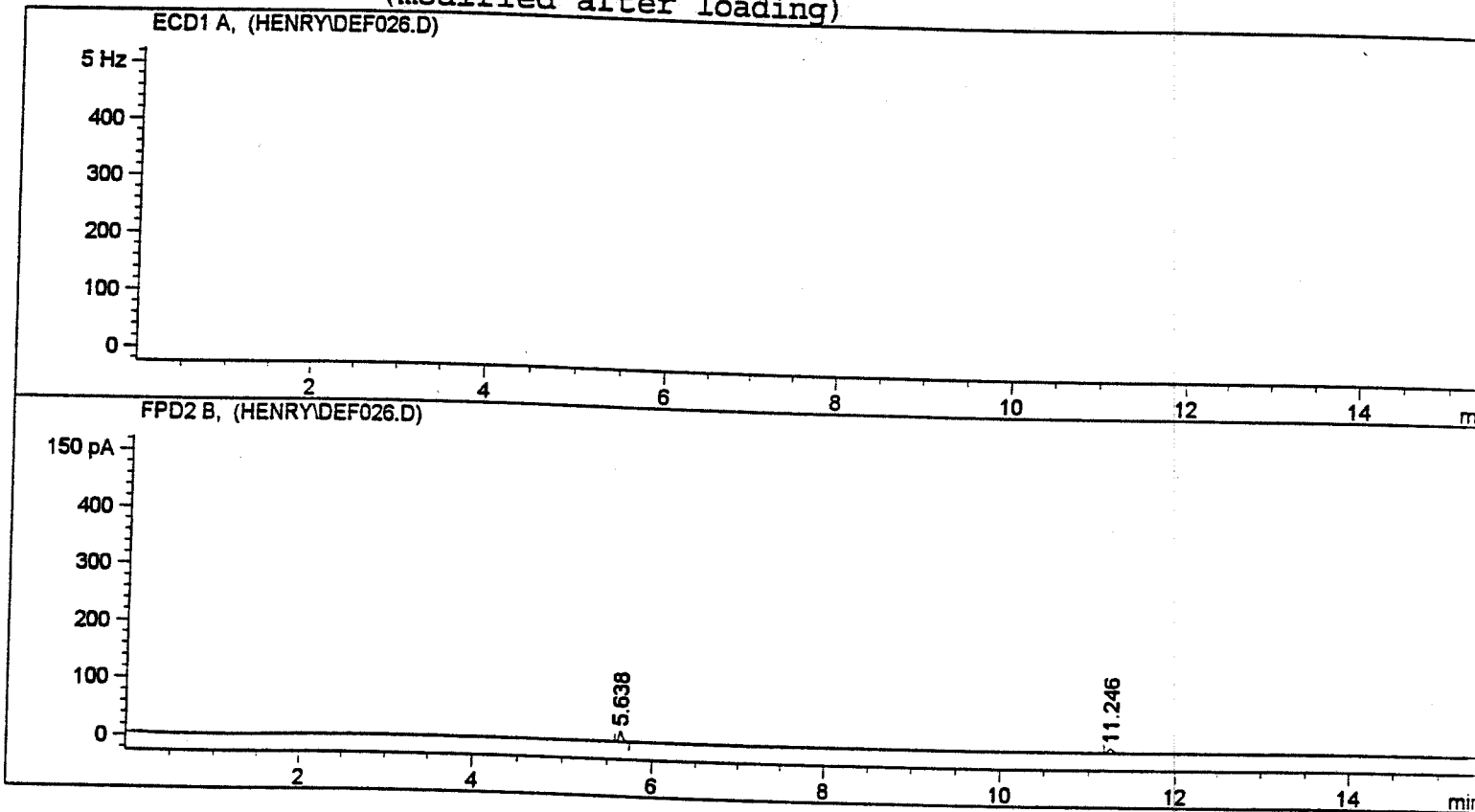
Data File C:\HPCHEM\3\DATA\HENRY\DEF026.D

Sample Name: soil

```

=====
Injection Date   : 3/26/98 8:24:09 AM
Sample Name     : soil 02
Acq. Operator   : Henry Shoemaker
Vial            : 1
Inj             : 1
Inj Volume     : Manually

Acq. Method     : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/26/98 5:59:08 AM by Henry Shoemaker
                  (modified after loading)
Analysis Method : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/26/98 9:02:36 AM by Henry Shoemaker
                  (modified after loading)
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %
1	5.638	BV	0.0405	51.04218	17.97286	60.09324
2	11.246	BV	0.0551	33.89613	8.09159	39.90676

DBDS  
 DEF

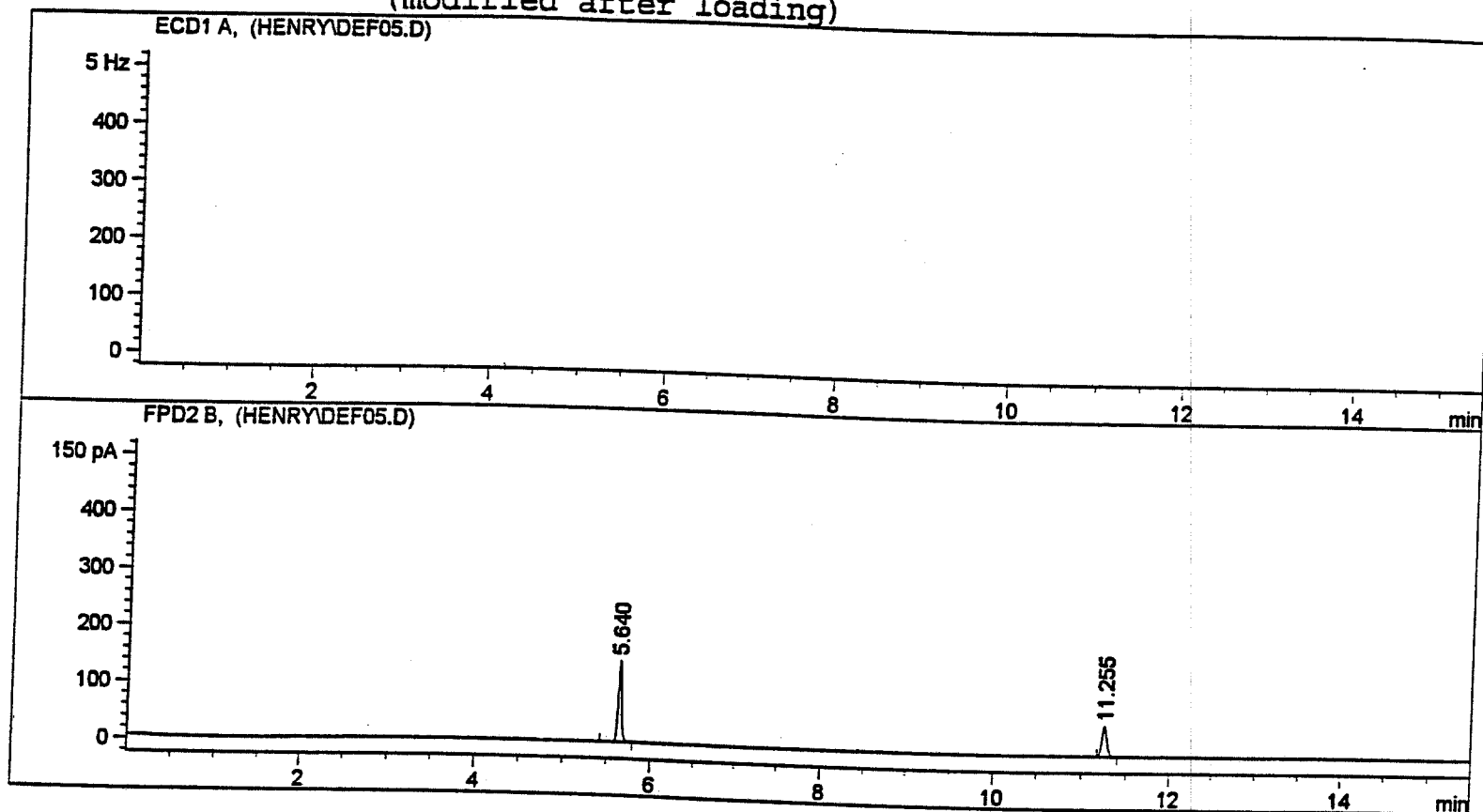


Data File: C:\HPCHEM\3\DATA\HENRY\DEF05.D

Sample Name: Soil 05

```

=====
Injection Date   : 3/25/98 9:26:22 AM
Sample Name     : Soil 05
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/25/98 8:52:35 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj             : 1
Inj Volume      : Manually
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
  
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %
1	5.640	BB	0.0420	390.86453	142.31082	65.05044
2	11.255	BB	0.0570	209.99928	53.13925	34.94956

DBDS  
 DEF

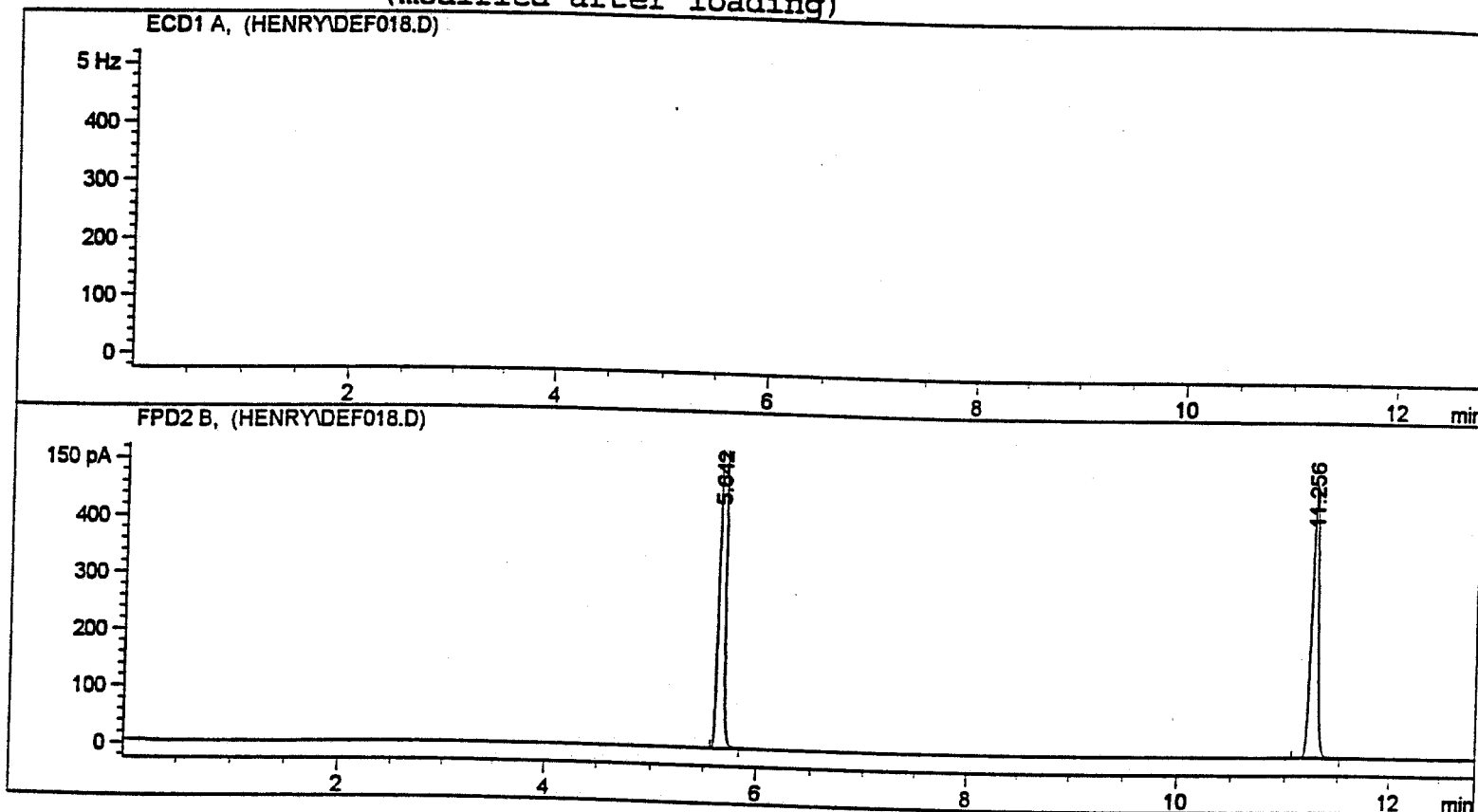
Totals : 600.86382 195.45007

Data File C:\HPCHEM\3\DATA\HENRY\DEF018.D

Sample Name: soil-1

```

=====
Injection Date   : 3/25/98 3:03:02 PM
Sample Name     : soil-10
Acq. Operator   : Henry Shoemaker
Method          : C:\HPCHEM\3\METHODS\HMS.M
Last changed    : 3/25/98 11:21:42 AM by Henry Shoemaker
                  (modified after loading)
Vial            : 1
Inj             : 1
Inj Volume      : Manually
    
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
    
```

Signal 1: ECD1 A,  
 Results obtained with standard integrator!

Signal 2: FPD2 B,  
 Results obtained with standard integrator!

Peak #	RetTime [min]	Type	Width [min]	Area 150 pA*s	Height [150 pA]	Area %	
1	5.642	BV	0.0509	3468.33105	1100.65186	62.35274	DBDS
2	11.256	BB	0.0677	2094.10498	475.90564	37.64726	DEF

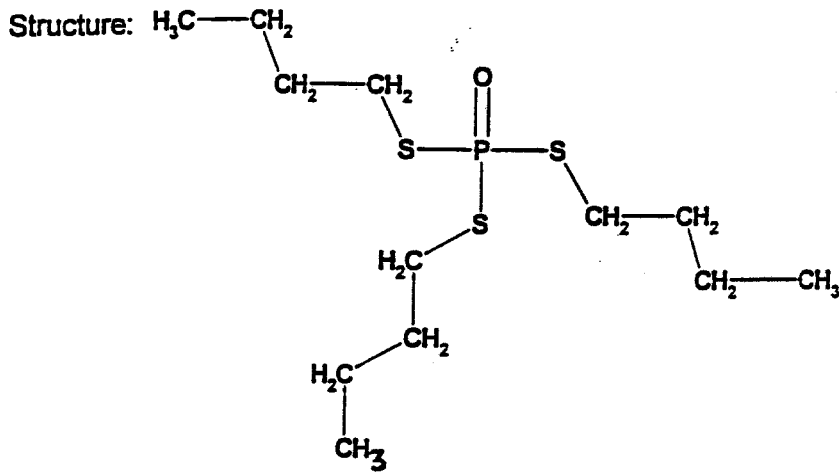
Totals : 5562.43604 1576.55750

# APPENDIX I

Chemical Name: *s,s,s*-tributylphosphorothioate

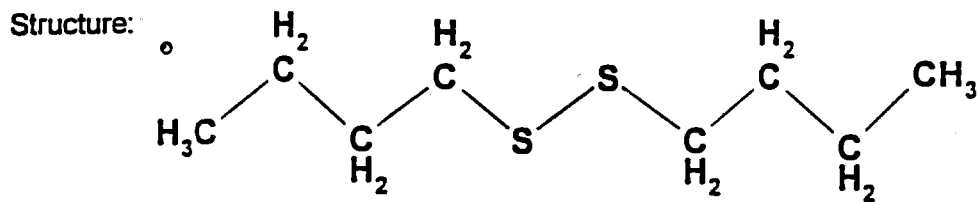
(DEF)

Molecular Weight: 314.51



Chemical Name: Dibutyl disulfide

Molecular Weight: 178.36



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## SOIL ANALYSIS

NOV - 6 1995

<b>CLIENT</b> U.S. EPA/ECS BLDG. 1105 STENNIS SPACE CNTR, MS 3952	<b>GROWER</b> U.S. EPA/ECS	<b>REPORT</b> 305-050 <b>DATE</b> 11/03/95 <b>ACCOUNT</b> 1500 <b>PAGE</b> 1 <b>A &amp; L AGRONOMIST</b> Richard Large
	DATE RECEIVED: 10/31/95	

LAB NUMBER 01087

SAMPLE ID IOWA

SIGNATURE *Richard Large*

TEST	RESULTS	SOIL TEST RATINGS					CATION EXCHANGE CAPACITY
		Very Low	Low	Medium	High	Very High	
Soil pH	6.3						19.6 meq/100g
Buffer pH	6.74						
Phosphorus (P)	6 ppm	██████████					<b>CATION SATURATION</b> %K 1.2 %Ca 68.6 %Mg 19.5 %H 10.6 %Na .2
Potassium (K)	93 ppm	██████████					
Calcium (Ca)	2690 ppm	██████████					
Magnesium (Mg)	459 ppm	██████████					
Sulphur (S)							
Boron (B)							
Copper (Cu)							
Iron (Fe)							
Manganese (Mn)							
Zinc (Zn)							
Sodium (Na)	11 ppm	██████████					<b>SOIL TEST METHOD</b> AMMONIUM ACETATE EXTRACTION
Soluble Salts	0.34 mmho/cm	██████████					
Organic Matter	3.4 % ENR 112						
NO <sub>3</sub> -N							

\* ADD'L RESULTS TO FOLLOW

### SOIL FERTILITY GUIDELINES

CROP:

YIELD GOAL:

LIME	N	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> 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