2-28-96

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

Analytical Chemistry Section
Building 306, BARC-East
Beltsville, Maryland 20705

FEB 28 1996

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP# 4F04407. Sulfentrazone in/on Rotational Crops. Method Validation Report.

FROM: Mark W. Law, Chemist
Analytical Chemistry Section

THRU: Harvey K. Hundley, Head
Analytical Chemistry Section

THRU Donald A. Marlow, Chief
Analytical Chemistry Branch

TO: F.B Suhre, Acting Section Head
Tolerance Petition Section I
Chemistry Branch I-Tolerance Support
Health Effects Division (7509C)

INTRODUCTION:

The Analytical Chemistry Section was requested to run a method validation for the analysis of the herbicide sulfentrazone (N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1H-1,2,4-triazol-1-yl]phenyl]methanesulfonamide), and the metabolites 3-desmethyl sulfentrazone (N-[2,4 dichloro-5-[4-(difluoromethyl)-4,5-dihydro-5-oxo-1H-1,2,4-trizol-1-yl]phenyl]methanesulfonamide) and 3- hydroxymethyl sulfentrazone (N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-hydroxymethyl-5-oxo-1h-1,2,4-triazol-1-yl]phenyl]methanesulfonamide) in/on Wheat Grain, Wheat Forage, and Wheat Straw. The requested fortification levels were 0.025, 0.05, and 0.1 PPM for all analytes on all commodities. The validation was requested for method P-2982M, Analytical Methodology for the Determination of Sulfentrazone and its Metabolites in/on Winter Wheat, 4/25/95, by I. Kim, FMC Co., MRID #436510-14. This TMV is a follow-up to the TMV for sulfentrazone and 3-hydroxymethyl sulfentrazone in/on soybeans.

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METHOD SUMMARY:

Three separate procedures are described in the method provided. Sulfentrazone and 3-desmethyl sulfentrazone are done by one method, 3-hydroxymethyl sulfentrazone is done by a second procedure, and a third procedure was provided for a metabolite we were not requested to validate. In both of the procedures validated, 2.5 g. samples are extracted by refluxing with 75/25 Acetone / 0.25N HCl. After filtering to remove solids, the acetone is removed by rotary evaporation and the samples are again filtered. At this point, the sulfentrazone and 3-desmethyl sulfentrazone samples are cleaned up on a C-8 SPE cartridge. The C-8 cartridge is completely dried with a Nitrogen flow at 30 psi for 30 minutes. The samples are then eluted with 20/80 ethyl acetate/hexane and further cleaned up on a Silica SPE cartridge. The filtered 3-hydroxymethyl sulfentrazone samples are partitioned with hexane, then partitioned into dichloromethane. After evaporation, they are cleaned up on a silica SPE cartridge, derivatized with BFSTA, and again cleaned up on another silica SPE cartridge. The final determination for all samples is by GLC using a HP-50+ megabore capillary column with detection by electron capture detector.

COMMENTS:

1. The analytical standard was not available from the EPA standards repository in RTPon 10/19/95. The standards and commodity samples used in this method trial were provided by the petitioner.

2. A copy of ACL's method validation pre-review is attached as additional information for this study and should be referenced for additional minor modifications that should be made to the proposed enforcement method.

3. The method trial was started using a regular HP injection liner rather than the Restek Cyclo Uniliner specified in the method since the Cyclo Uniliner was not specified in the soybean method and the regular liner gave good results. Recoveries averaging 150 to 200% were obtained for several sets of samples of grain and forage spiked with sulfentrazone and 3-desmethyl sulfentrazone. The Cyclo Uniliers were purchased and the trial was repeated. All data used in this report is from analyses using the Cyclo Uniliers as specified in the method.

4. It was noted in the TMV request that the metabolite 3-hydroxymethyl sulfentrazone was not included in the ILV. CBTS felt it should not be required because of the method similarity to the method for Soybeans. This is not correct. The method for soybeans determines sulfentrazone and 3-hydroxymethyl sulfentrazone by a method exactly like the wheat method for sulfentrazone and 3-desmethyl sulfentrazone with the addition of the derivatization step. The 3-hydroxymethyl sulfentrazone procedure in the wheat method is quite different. It adds hexane partitioning, and extracting into dichloromethane. It also uses two silica gel SPE cartridges instead of one C-8 and one silica. The method summary on page 7 of the method implies that this method is similar to the soybean method, and glosses over the significant

method, and glosses over the significant differences in the two methods. The schematics for the two procedures, given on pages 9 and 10 of the method, show that these are clearly two very different procedures.

5. The clean-up procedure for sulfentrazone and 3-desmethyl sulfentrazone requires one and a half days for a set of 8 samples. The procedure for the 3-hydroxymethyl sulfentrazone requires 2 working days to clean up and derivatize a set of 8 samples. The GC analyses require an overnight run to acquire data.

6. The oven temperature program was modified from that specified in the method to further separate the compounds of interest from the interferences present in the sample extracts. Even with the improved chromatography parameters, there was too much interference in the wheat straw extracts for quantitation of sulfentrazone and 3-desmethyl sulfentrazone. Recoveries for 3-hydroxymethyl sulfentrazone were very low in the wheat grain and wheat forage samples, and no attempt was made to analyze wheat straw for 3-hydroxymethyl sulfentrazone.

7. In section d. of the procedure for 3-hydroxymethyl sulfentrazone, the instructions call for eluting 15 ml of 25% acetonitrile/DCM into a clean 13 ml centrifuge tube. In section e. of the same procedure, the sample must be evaporated to about 0.1 ml to obtain an oily film which must not be allowed to darken or harden. It is very difficult to be sure all acetonitrile is removed from the sample and that the film has not dried.

8. This method does not meet the requirements in the Pesticide Assessment Guidelines, Subdivision O, Section 171-4(b) for any of the analytes in wheat straw or for 3-hydroxymethyl sulfentrazone in any of the commodities evaluated. The sulfentrazone and 3-desmethyl sulfentrazone procedure works well for wheat grain and wheat forage, but not for wheat straw. The procedure for 3-hydroxymethyl sulfentrazone gave very poor recoveries for both wheat grain and wheat forage samples. This procedure is also quite different from the soybean method and a successful ILV should be required on this procedure before a new TMV is requested.

METHOD: Analytical Methodology for the Determination of Sulfentrazone and Its Metabolites in/on Winter Wheat. 3/ 7/95 By I. Kim. FMC Co. MRID# 436510-14.

Commodity	Chemical Added	PPM ADDED	PPM FOUND	PERCENT RECOVERY
Wheat Grain	Sulfentrazone	0	N.D.*	
		0	N.D.	
		0.0251	0.0206	82.1
		0.0251	0.0193	76.9
		0.0502	0.0367	73.1
		0.0502	0.0425	84.7
		0.1004	0.0709	70.6
		0.1004	0.0675	67.2
		0	N.D.	
		0.0250	.0240	96.0
		0.0250	.0210	84.0
		0.0500	.0414	82.8
		0.0500	.0478	95.6
		0.1001	.0767	76.6
		0.1001	.0753	75.2
Wheat Grain	3-Hydroxymethyl Sulfentrazone	0	N.D.*	
		0	N.D.	
		0.0247	0.0096	38.9
		0.0247	0.0064	25.9
		0.0493	0.0067	13.6
		0.0493	0.0081	16.4
		0.0987	0.0326	33.0
		0.0987	0.0153	15.5

*N.D. = Not Detected at a sensitivity of 0.01 ppm.

Commodity	Chemical Added	PPM ADDED	PPM FOUND	PERCENT RECOVERY
Wheat Forage	Sulfentrazone	0	N.D.*	
		0	N.D.	
		0.0251	0.0226	90.0
		0.0251	0.0280	112
		0.0502	0.0445	88.6
		0.0502	0.0491	97.8
		0.1004	0.0859	85.6
		0.1004	0.0810	80.7
		0	N.D.	
		0.0250	0.0243	97.2
		0.0250	0.0281	112
		0.0500	0.0469	93.8
		0.0500	0.0464	92.8
		0.1001	0.0886	88.5
		0.1001	0.0826	82.5
Wheat Forage	3-Hydroxymethyl Sulfentrazone	0	N.D.*	
		0	N.D.	
		0.0247	0.0068	27.6
		0.0247	0.0071	28.7
		0.0493	0.0300	60.8
		0.0493	0.0194	39.3
		0.0987	0.0330	33.4
		0.0987	0.0358	36.3

*N.D. = Not Detected at a sensitivity of 0.01 ppm.

Commodity	Chemical Added	PPM ADDED	PPM FOUND	PERCENT RECOVERY
Wheat Straw	Sulfentrazone	0	N.D.*	
		0	N.D.	
		0.0251	0.0061	24.3
		0.0251	0.0115	45.8
		0.0502	0.0283	56.4
		0.0502	0.0231	46.0
		0.1004	0.0937	93.3
		0.1004	0.0365	36.4
		0	N.D.	
		0.0250	0.0096	38.4
		0.0250	0.0166	66.4
		0.0500	0.0406	81.2
		0.0500	0.0348	69.6
		0.1001	0.0609	60.8
		0.1001	0.0249	24.9
Wheat Straw	3-Hydroxymethyl Sulfentrazone	0		
		0		
		0.0247		
		0.0247		
		0.0493		
		0.0493		
		0.0987		
		0.0987		

*N.D. = Not Detected at a sensitivity of 0.01 ppm.

Modifications to method (major or minor):

None

Special precautions to be taken:

None

Source of analytical reference standards:

Standards were not available from RTP, and were provided by petitioner.

If derivatized standards used, give source:

None--Standards are derivatized along with samples.

Instrument for Confirmation:

None

If instrument parameters differ from method as written, list parameters actually used:

HP 50+ GC column substituted for DB-17 with permission of petitioner. HP 50+ is used in the soybean method. Temperature ramp changed to improve peak separation. 185 deg for 1 min, 20 deg/min to 265 deg. Hold 8 min, then 20 deg/min to 285 deg, and hold 8 min. Retention times are: 7/8 min for 3-desmethyl sulfentrazone, 8.4 min. for sulfentrazone, and 10.9 min. for 3-hydroxymethyl sulfentrazone.

Commercial source for any special chemicals or apparatus:

Sources for all equipment and reagents given in method.

Comments:

Attached

Chromatograms:

Representative chromatograms attached.

TMV Pre-review of Sulfentrazone
in Wheat Grain, Straw and Forage

Reviewed by: Everett S. Greer, Jr. ESG

Date: 9-25-95

Laboratory assignment number: 95-76,77,78

Analytes: Sulfentrazone, 3-desmethyl sulfentrazone and 3-hydroxymethyl sulfentrazone

Commodity: Wheat grain, straw and forage

Petitioner: FMC Corporation

Method: Analytical Methodology for the Determination of Sulfentrazone and Its Metabolites in/on Winter Wheat

V. Analytical procedure

A. Residue Method

1.Sulfentrazone and 3-desmethyl sulfentrazone (grain, forage and straw)

a. Extraction

No directions are given for preparing the wheat commodities for the reflux extraction step. A statement should be given describing how the commodities are ground up prior to weighing into the extraction flask.

The extraction solution is evaporated using a Turbo Vap Evaporator. The ILV substituted a rotary evaporator in this step.

b. C_8 Cartridge solid phase extraction

A Sulpelco Visidry vacuum manifold is used for drying the SPE cartridge after addition of the sample extract. The temperature for evaporating the ethyl acetate/hexane eluate under nitrogen is not given.

c. Silica gel cartridge solid phase extraction

The temperature for the nitrogen evaporation step is not given.

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2. 3-Hydroxymethyl sulfentrazone (grain, forage, and straw)

The temperature of the nitrogen evaporation in steps d, e and f are not given.

3. 3-Desmethyl-4-desfluoromethyl sulfentrazone (forage only)

The temperature of the nitrogen evaporation in steps b, c and d are not given.

D. Method of calculation

This section states that residues found in control samples were subtracted from fortified controls.

F. Confirmatory techniques

A complete set of GC/MSD instrument parameters are not given.

Additional reviewer's comments

1. Recovery data are provided for forage and grain fortified at the 0.025 ppm, 0.05 ppm and 0.1 ppm levels with the three requested compounds. Recovery data is provided for straw fortified at 0.025 ppm and 0.05 ppm with these compounds, but no 0.1 ppm straw data is included. For some of the data only one recovery is provided for an analyte/matrix combination.

Chromatograms are included for wheat straw and forage fortified with all three compounds at the lowest level (0.025 ppm) requested by CB-1 The lowest level chromatogram for wheat grain fortified with sulfentrazone and 3-desmethyl sulfentrazone is for 0.05 ppm. A wheat grain chromatogram for a 0.025 fortification with 3-hydroxymethyl sulfentrazone is presented.

2. The ILV laboratory validated the method for sulfentrazone and 3-desmethyl sulfentrazone on wheat grain fortified at the 0.05 ppm and 0.1 ppm levels. The ILV recoveries are within the 70% to 120% range. The ILV laboratory did not validate the method for 3-hydroxymethyl sulfentrazone. The 3-hydroxymethyl compound, which includes a derivatization step not included in the parent and desmethyl procedure, is determined separately through a different analytical scheme.

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The ILV used a slightly different solvent composition for eluting the analyte from the silica gel cartridge in step V.A.1.c. The ILV report states that new lots of both the C_8 and silica SPE cartridges should be evaluated prior to the analysis.

ANALYTICAL CHEMISTRY BRANCH
SCREEN FOR RESIDUE METHODS FOR TMV

1. LABORATORY ASSIGNMENT NUMBER: B95- 76, 77, 78

2. PP#: 4F04407

3. TECHNICAL REVIEWER: Everett S. Green, Jr.

4. DATE: 9-25-95

5. ANALYTES/LEVEL: Sulfentrazone, 3-Desmethyl Sulfentrazone, 3-Hydroxymethyl Sulfentrazone / 0.025, 0.05, 0.10 ppm

6. COMMODITIES: Wheat grain, straw, forage

7. METHOD: Analytical Methodology for the Determination of Sulfentrazone and Its Metabolites in/on Winter Wheat

The Analytical Chemistry Section has been asked to screen the residue chemistry methods submitted by the registrant in order to determine if they contain the essential requirements identified in the Residue Chemistry Guidelines. Full scientific review and laboratory evaluation of those methods will take place after the initial screen. The following items need to be resolved before the analytical method can be evaluated.

	YES	NO
1. Does the method use exotic equipment and/or supplies that are not commercially available in the U.S.?		✓
2. Does the method require any new equipment before the laboratory work begins?	See narrative review	
3. Are chromatograms included?	✓	
a. Is (are) peak(s) of interest sufficiently resolved from other peaks?	✓	
b. Has registrant included chromatograms of analyses at or below tolerance on all crop types for which tolerance is requested by HED?	✓	
c. Do the control samples have reasonably low levels of the analyte in relation to the proposed tolerance?	✓	
d. Is the method sufficiently sensitive and specific to measure and identify the residues at levels specified by HED in the TMV request?	✓	

	YES	NO
4. Has recovery data been provided to ACL for the residues that are specified in the TMV request?	✓	
5. Are recovery values between 70% and 120% at all levels and for all commodity types?	✓	
6. Are all procedures clearly written with no ambiguities so that the method can be run without communication with the registrant?	✓	
7. Does the method require correction for a sample of the untreated commodities or a blank?		✓
8. Does the method require the use of an internal or procedural standard to compensate for lost analyte during analysis?		✓
9. Are 2nd laboratory validation data provided with the method?	✓	See narrative review
10. Are there any deficiencies other than those covered above that would prevent ACS from conducting a method trial?		✓
11. Is this method suitable for validation testing?	✓	

Any deficiencies/problems noted for any above items should be addressed in the full scientific review of this method to be attached as an addendum.

Robert S. Kheen — Signature

9-25-95 — Date

The following is to be completed by the analyst performing the TMV.

12. Can a set of 6 samples be run within 24 hours?	✓	
13. a. Are standards available at RTP repository?		✓
b. Are derivatized analytical reference standards available?	N/A	

LINEARITY OF SULFENTRAZONE AND 3-DESMETHYL SULFENTRAZONE

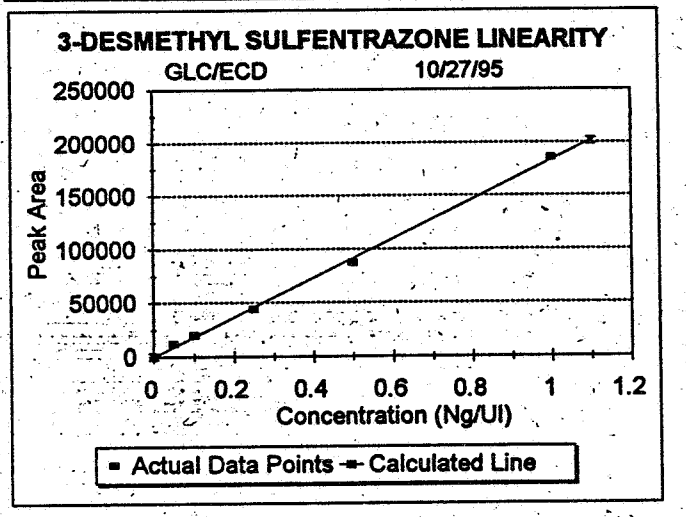
Project Number: B95-(76-78)
Project Name: Sulfentrazone in/on Wheat TMV
Instrument: HP 5890 GC W/ EC Detector
Column: HP-50+, 50% phenylmethylsilicone, 30M x 0.53mm x 1.0 um film thickness.
Injection Volume: 2 ul
Date: 1/25/96
Analyst: Mark W. Law

3-DESMETHYL SULFENTRAZONE

Std. Conc. Ng/Ul	Area 1	Area 2	Area 3	Average Area	Calculate Areas
0.0501	12619	11864	11334	11939	
0.1001	21891	19357	18480	19909	
0.2503	42946	46275	42931	44051	
0.5005	88350	89540	85316	87735	
1.0010	183213	188601	185148	185654	
0.0000					290
1.1000					201469

Regression Output:

Constant	290.1592
Std Err of Y Est	3364.764
R Squared	0.998322
No. of Observations	5
Degrees of Freedom	3
X Coefficient(s)	182889.5
Std Err of Coef.	4328.744

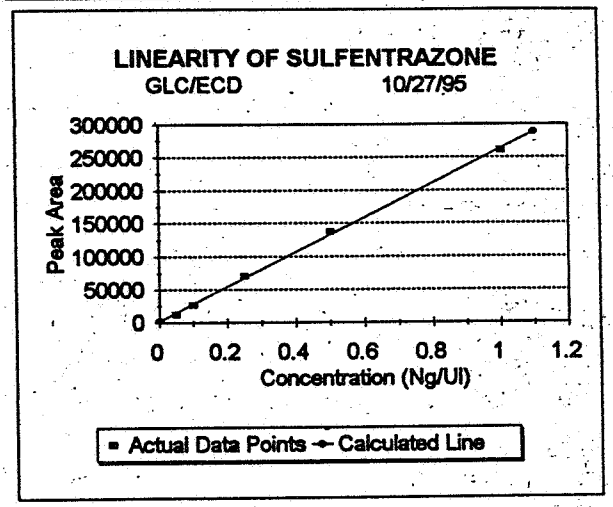


SULFENTRAZONE

Std. Conc Ng/Ul	Area 1	Area 2	Area 3	Average Area	Calculated Areas
0.0502	12871	12824	12590	12762	
0.1004	27123	26998	26528	26883	
0.2511	69910	71335	71326	70857	
0.5022	138249	138714	137580	138181	
1.0044	258198	263251	261076	260842	
0.0000					2743
1.1000					288532

Regression Output:

Constant	2743.423
Std Err of Y Est	4243.881
R Squared	0.998686
No. of Observations	5
Degrees of Freedom	3
X Coefficient(s)	259808
Std Err of Coef.	5441.239



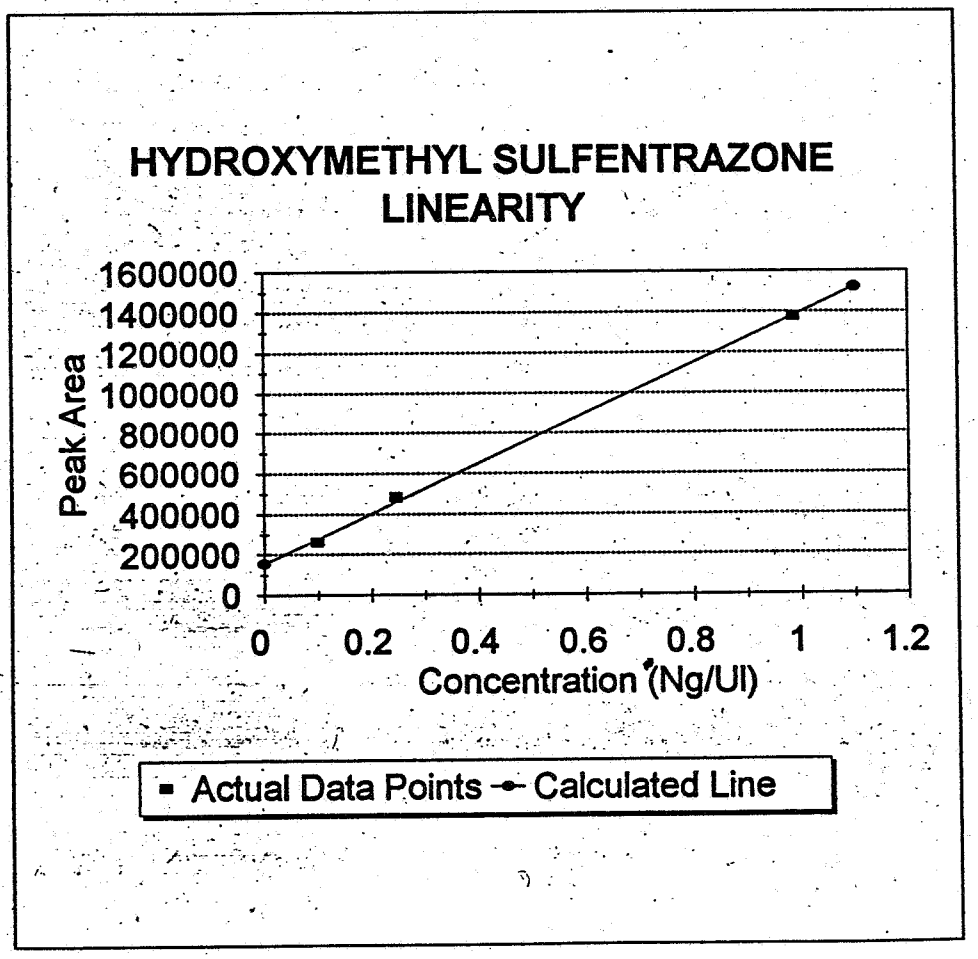
LINEARITY OF 3-HYDROXYMETHYL SULFENTRAZONE

Project Number: B95-(76-78)
Project Name: Sulfentrazone in/on Wheat TMV
Instrument: HP 5890 GC W/ EC Detector
Column: HP-50+, 50% phenylmethylsilicone, 30M x 0.53mm x 1.0 um film thickness.
Injection Volume: 2 ul
Date: 2/7/96
Analyst: Mark W. Law

Std. Conc. Ng/Ul	Area 1	Area 2	Area 3	Average Area	Calculated Areas
0.0494	231222	222549	196221	216664	
0.0987	279206	262345	245403	262318	
0.2467	489500	480930	476727	482386	
0.9870	1388090	1368070		1378080	
0.0000					155920
1.1000					1521780

Regression Output:

Constant	155919.8
Std Err of Y Est	18419.08
R Squared	0.999228
No. of Observations	4
Degrees of Freedom	2
X Coefficient(s)	1241691
Std Err of Coef.	24399.47



Data File C:\HPCHEM\2\DATA\SULF\006F1103.D Sample Name: 0.1 ng/ul Stds
2 ul injection of 0.10044 ng/ul Sulfentrazone and 0.100
10 ng/ul 3-desmethyl sulfentrazone.

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Injection Date : 1/30/96 3:32:27 PM Seq. Line : 11
Sample Name : 0.1 ng/ul Stds Vial : 6
Acq. Operator : Mark W. Law Inj : 3
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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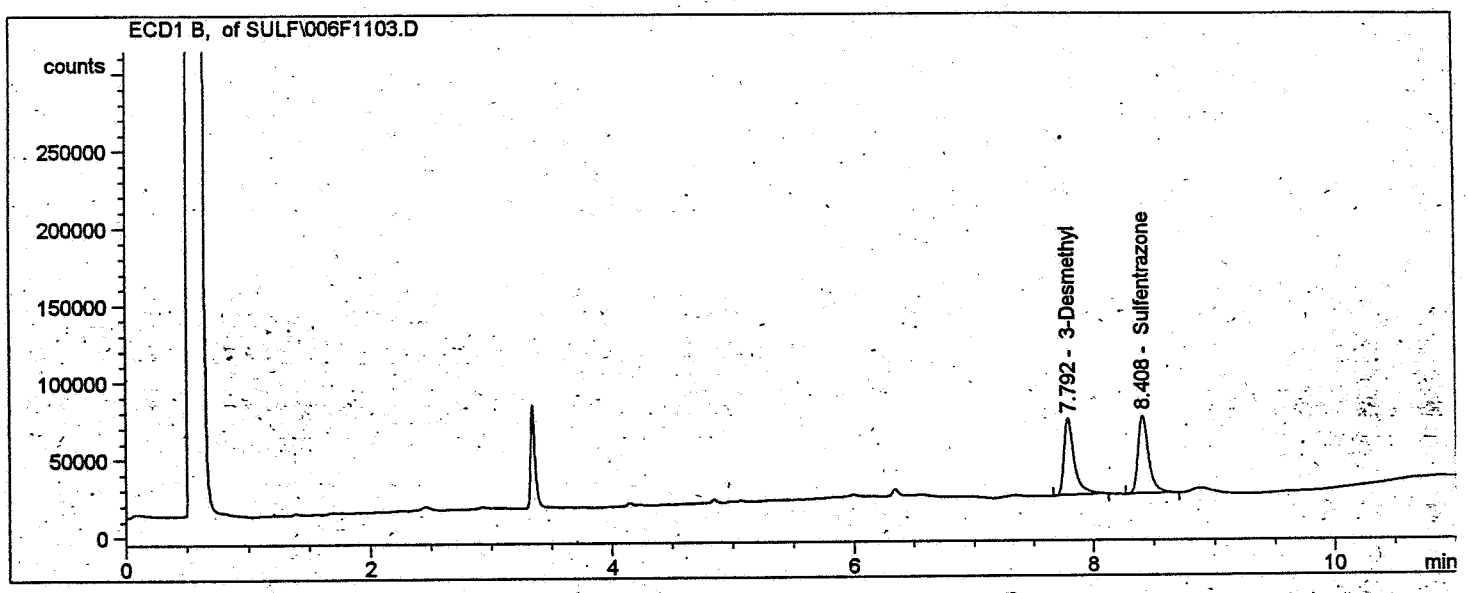

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Tuesday, January 30, 1996 8:39:27 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.792	BB +	0.089	2.96970e5	49.06	3-Desmethyl
2	8.408	BB +	0.092	3.08353e5	50.94	Sulfentrazone

Totals : 605323.50000

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*** End of Report ***

15

Data File C:\HPCHEM\2\DATA\SULF\007F1203.D Sample Name: Grain Control 1
2.5 g. Wheat Grain Control, 1.0 ml final, 2 ul inj.

```
==========================================================================
Injection Date  : 1/30/96 4:26:59 PM              Seq. Line :  12
Sample Name     : Grain Control 1                      Vial :   7
Acq. Operator   : Mark W. Law                          Inj :   3
                                                  Inj Volume : 2 µl

Sequence File   : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method          : C:\HPCHEM\2\METHODS\SULF.M
Last changed    : 1/30/96 8:39:27 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
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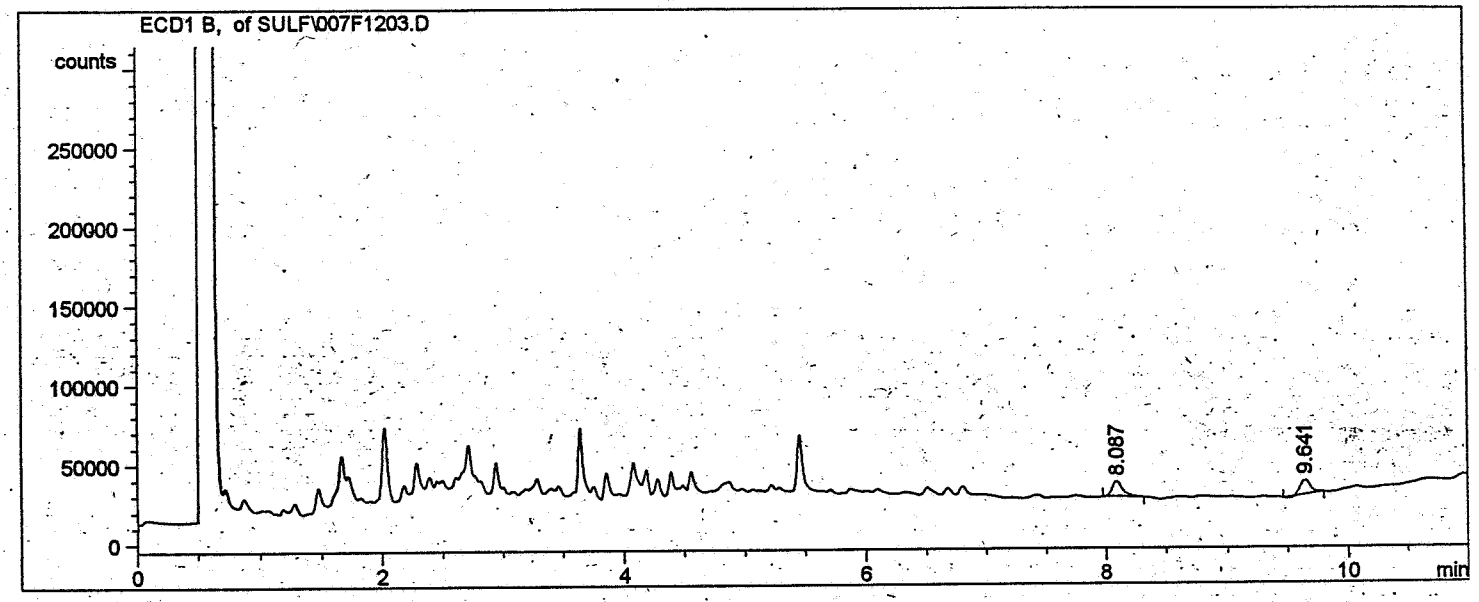


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==========================================================================
                        Area Percent Report
==========================================================================

Sorted by Signal
Calib. Data Modified  :      Tuesday, January 30, 1996 8:39:27 AM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,

 Peak    RT     Type    Width      Area       Area     Name
  #    [min]            [min]    counts*s      %
|----|---------|------|-------|------------|--------|--------------|
   1    8.087   BB      0.091   60068.35938   53.21   ?
   2    9.641   BB      0.087   52810.89062   46.79   ?

Totals :                        112879.25000

1 Warnings or Errors :

Warning : Time reference compound(s) not found

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

/6

Data File C:\HPCHEM\2\DATA\SULF\010F1601.D Sample Name: Grain-0.025-2
2.5 g. Wheat Grain + 0.025 ppm each Sulfentrazone and 3
-desmethyl sulfentrazone, 1 ml final vol., 2 ul inj.

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Injection Date : 1/30/96 7:28:51 PM Seq. Line : 16
Sample Name : Grain-0.025-2 Vial : 10
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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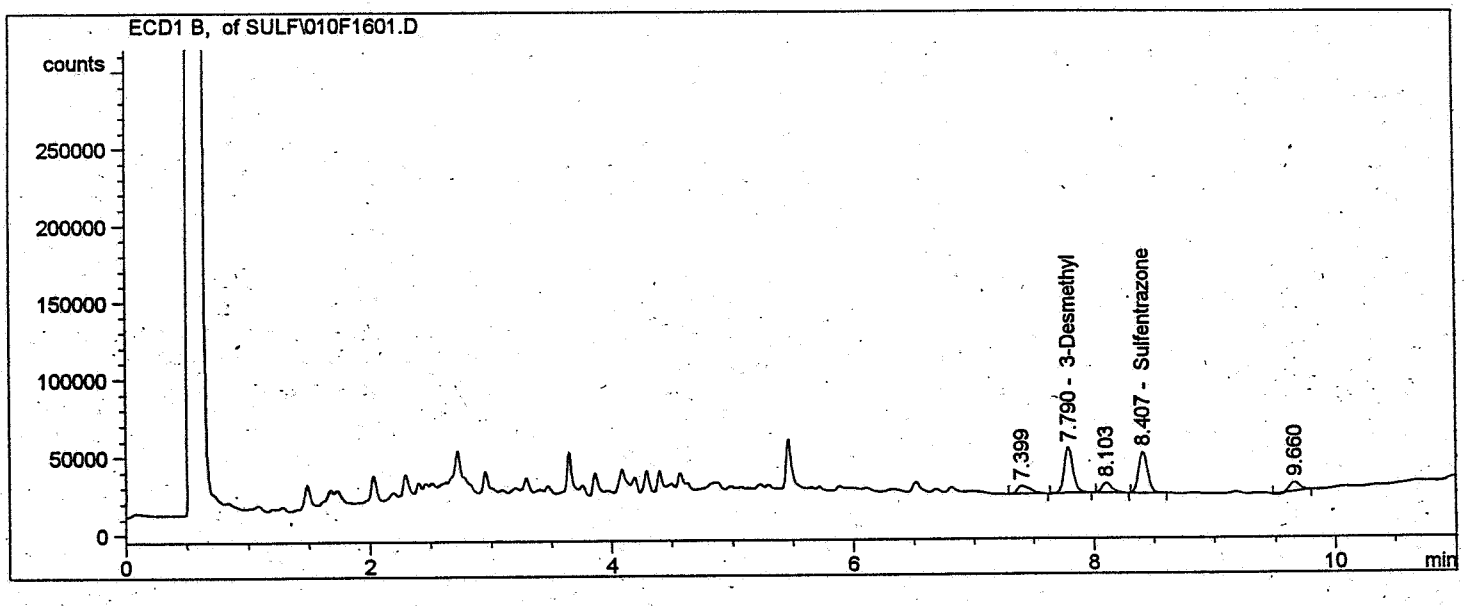

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Tuesday, January 30, 1996 8:39:27 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.399	BB		0.107	41269.90625	10.16	?
2	7.790	BB	+	0.079	1.52574e5	37.57	3-Desmethyl
3	8.103	BB		0.086	37928.52344	9.34	?
4	8.407	BB	+	0.082	1.39806e5	34.43	Sulfentrazone
5	9.660	BB		0.090	34478.87891	8.49	?

Totals : 406057.96875

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*** End of Report ***

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Data File C:\HPCHEM\2\DATA\SULF\013F1901.D Sample Name: Grain-0.05-2

2.5 G. Wheat Grain + 0.05 ppm Sulfentrazone and 3-desme
thyl sulfentrazone. 1 ml final vol. 2 ul injection.

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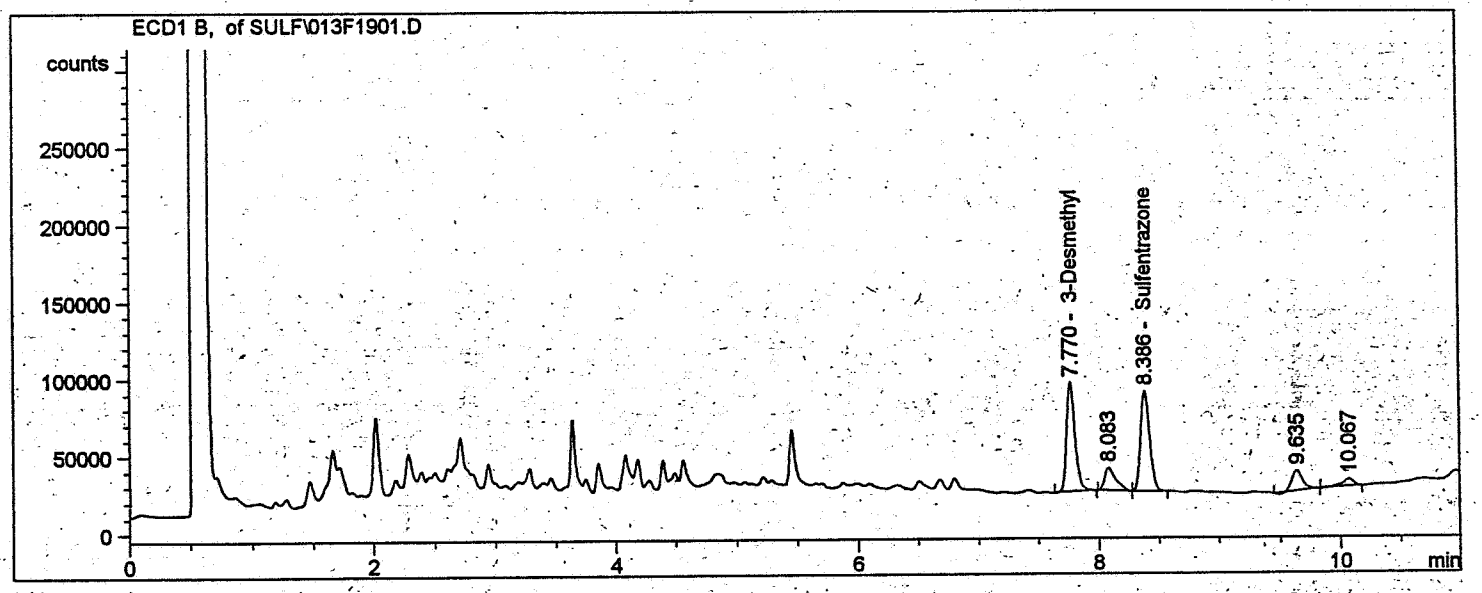
==========================================================================
Injection Date  : 1/30/96 10:12:47 PM          Seq. Line :  19
Sample Name     : Grain-0.05-2                      Vial :  13
Acq. Operator   : Mark W. Law                       Inj :   1
                                               Inj Volume : 2 µl

Sequence File   : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method          : C:\HPCHEM\2\METHODS\SULF.M
Last changed    : 1/30/96 8:39:27 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
==========================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Tuesday, January 30, 1996 8:39:27 AM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.770	BB +	0.074	3.42755e5	38.68	3-Desmethyl
2	8.083	BB	0.094	93853.92969	10.59	?
3	8.386	BB +	0.081	3.36503e5	37.98	Sulfentrazone
4	9.635	BB	0.093	79534.35156	8.98	?
5	10.067	BB	0.104	33439.40234	3.77	?
Totals :				886086.00000		

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

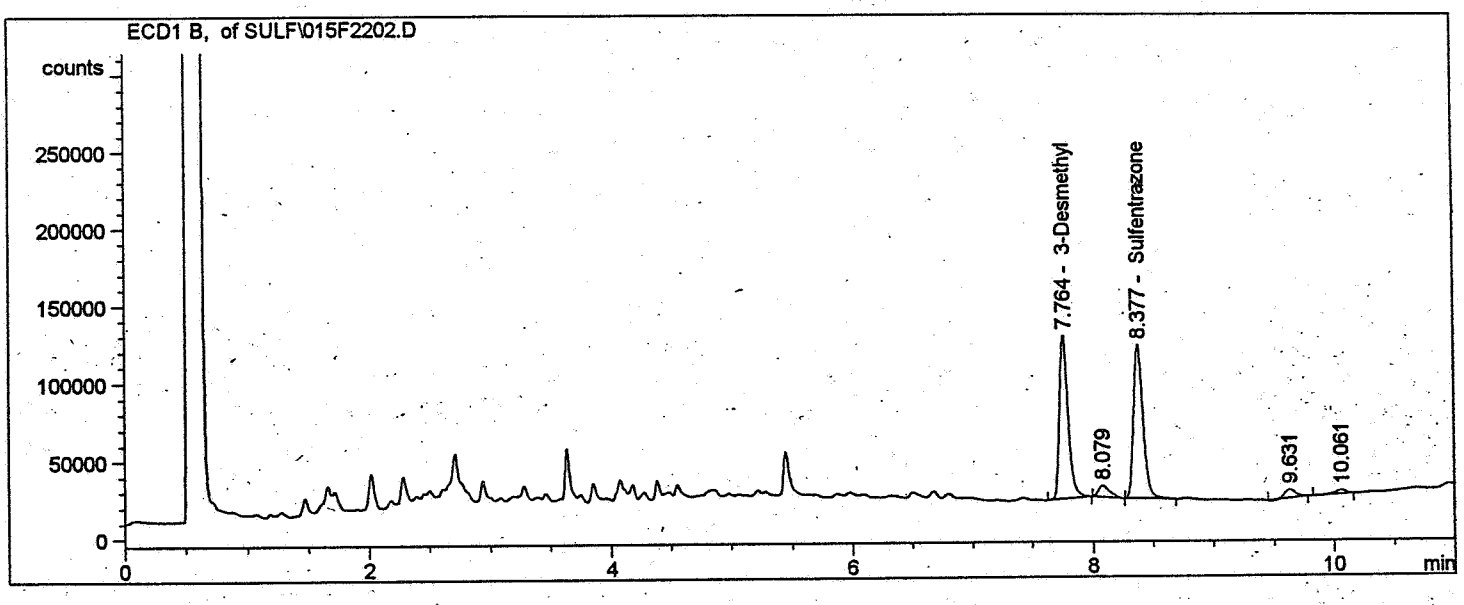
17

Data File C:\HPCHEM\2\DATA\SULF\015F2202.D Sample Name: Grain-0.1-2

2.5 g. Wheat Grain + 0.1 ppm Sulfentrazone and 3-desmet
hyl sulfentrazone. 1 ml final vol. 2 ul injection.

```
==========================================================================
Injection Date  : 1/31/96 1:15:01 AM          Seq. Line :  22
Sample Name     : Grain-0.1-2                      Vial :  15
Acq. Operator   : Mark W. Law                      Inj :   2
                                             Inj Volume : 2 µl

Sequence File   : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method          : C:\HPCHEM\2\METHODS\SULF.M
Last changed    : 1/30/96 8:39:27 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
==========================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Tuesday, January 30, 1996 8:39:27 AM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.764	BB +	0.077	5.26124e5	44.76	3-Desmethyl
2	8.079	BB	0.093	48891.91797	4.16	?
3	8.377	BB +	0.085	5.46720e5	46.51	Sulfentrazone
4	9.631	BB	0.094	35359.24609	3.01	?
5	10.061	BB	0.100	18274.41406	1.55	?

Totals : 1175369.62500

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

(9

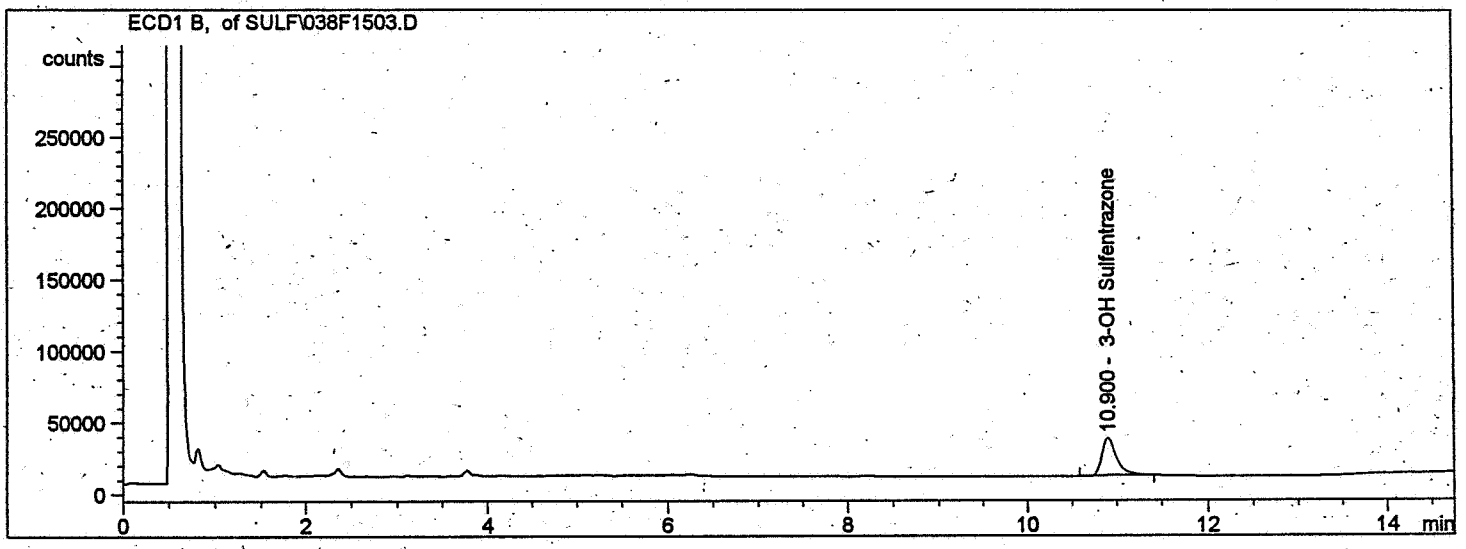
Data File C:\HPCHEM\2\DATA\SULF\038F1503.D Sample Name: 0.1 ng/ul Std.
2 ul injection of Derivitized Standard.
0.0987 ng/ul 3-OH Methyl Sulfentrazone.

==========

Injection Date : 2/8/96 6:36:46 PM Seq. Line : 15
Sample Name : 0.1 ng/ul Std. Vial : 38
Acq. Operator : Mark W. Law Inj : 3
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/8/96 6:29:46 PM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/8/96 6:51:35 PM by Mark W. Law
(recalibrated in sequence after loading)
B95-(76-78) Sulfentrazone on Wheat TMV.

==========



==========

Area Percent Report

==========

Sorted by Signal
Calib. Data Modified : Thursday, February 08, 1996 6:29:46 PM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	10.900	BB +	0.171	3.02065e5	100.00	3-OH Sulfentraz

Totals : 302064.59375

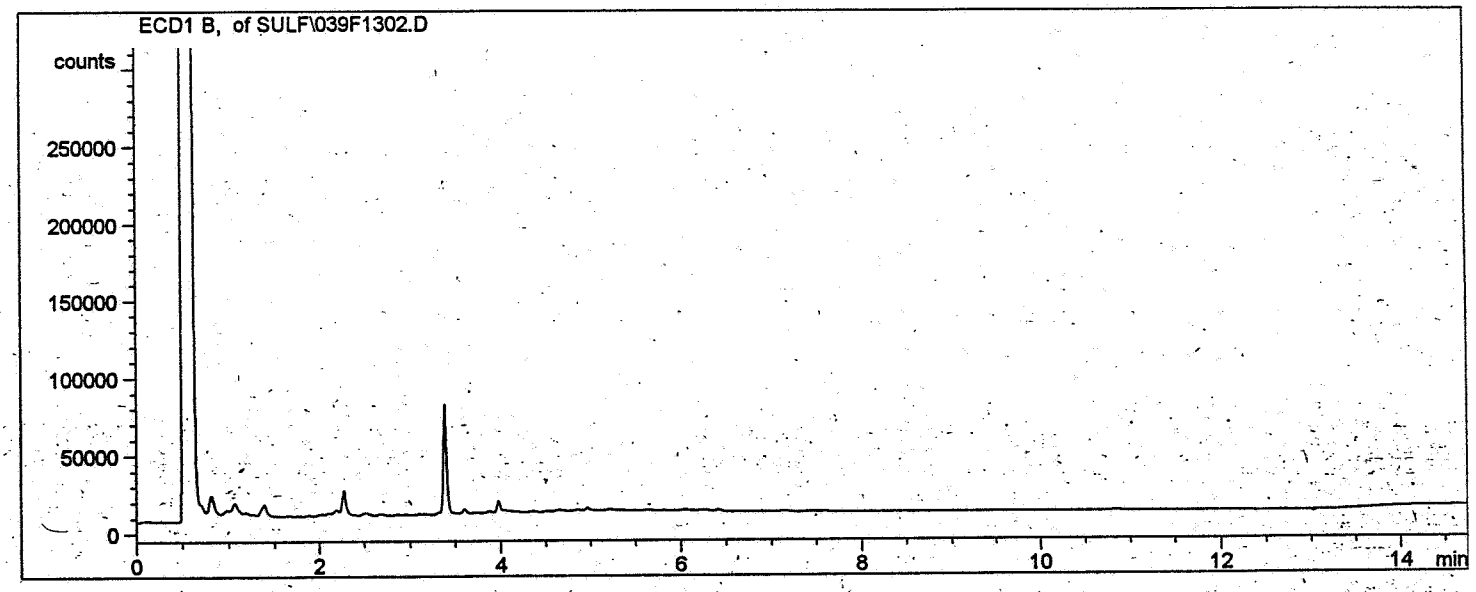
==========

*** End of Report ***

20

```
================================================================================
Injection Date   : 2/8/96 4:03:50 PM                    Seq. Line :  13
Sample Name      : Grain Control-1                          Vial :  39
Acq. Operator    : Mark W. Law                               Inj :   2
                                                      Inj Volume : 2 µl

Sequence File    : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method           : C:\HPCHEM\2\METHODS\SULF2.M
Last changed     : 2/8/96 3:35:07 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
================================================================================
```




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

Sorted by Signal
Calib. Data Modified   :      Thursday, February 08, 1996 3:35:07 PM
Multiplier             :      1.000000
Dilution               :      1.000000

Signal 1: ECD1 B,

1 Warnings or Errors :

Warning : Time reference compound(s) not found

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

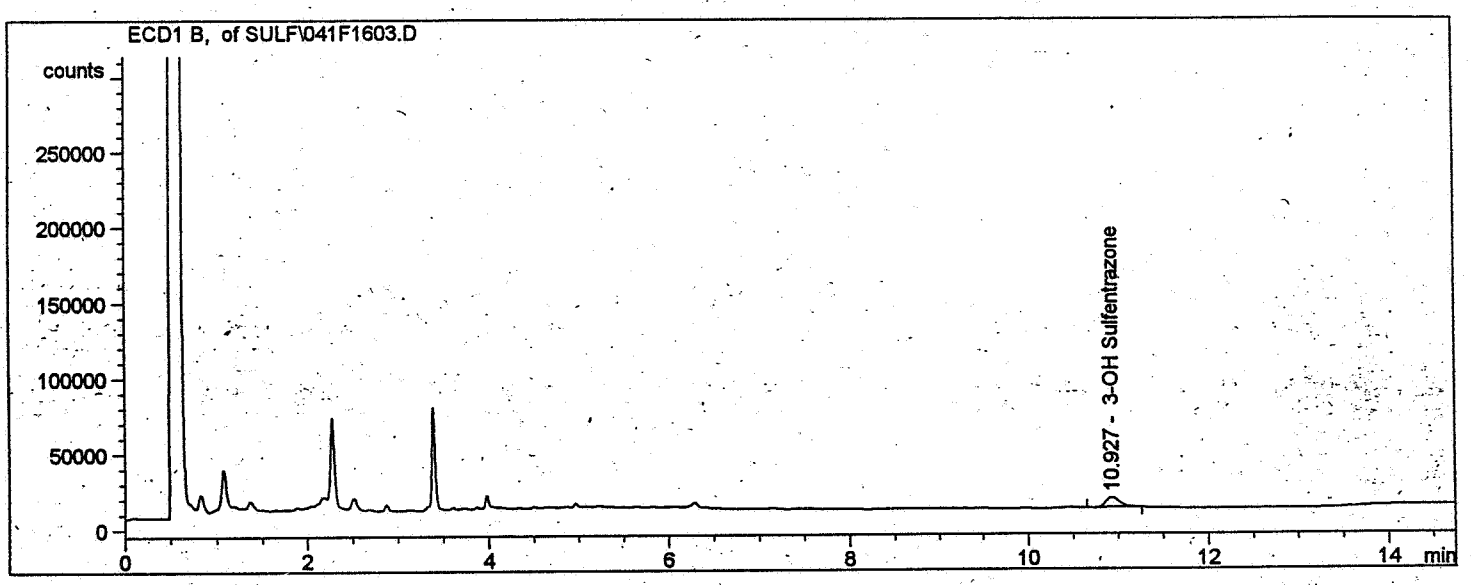

4

Data File C:\HPCHEM\2\DATA\SULF\041F1603.D Sample Name: Grain 0.025-1

2.5 G. Wheat Grain + 0.0247 ppm Spike of
3-OH-Methyl Sulfentrazone.
1 ml final vol., 2 ul injected.

```
================================================================================
Injection Date   : 2/8/96 7:42:24 PM                Seq. Line :  16
Sample Name      : Grain 0.025-1                         Vial :  41
Acq. Operator    : Mark W. Law                           Inj :   3
                                                   Inj Volume : 2 µl

Sequence File    : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method           : C:\HPCHEM\2\METHODS\SULF2.M
Last changed     : 2/8/96 6:51:35 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
================================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Thursday, February 08, 1996 6:51:35 PM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	10.927	BB +	0.164	73754.96875	100.00	3-OH Sulfentraz
Totals :				73754.96875		

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

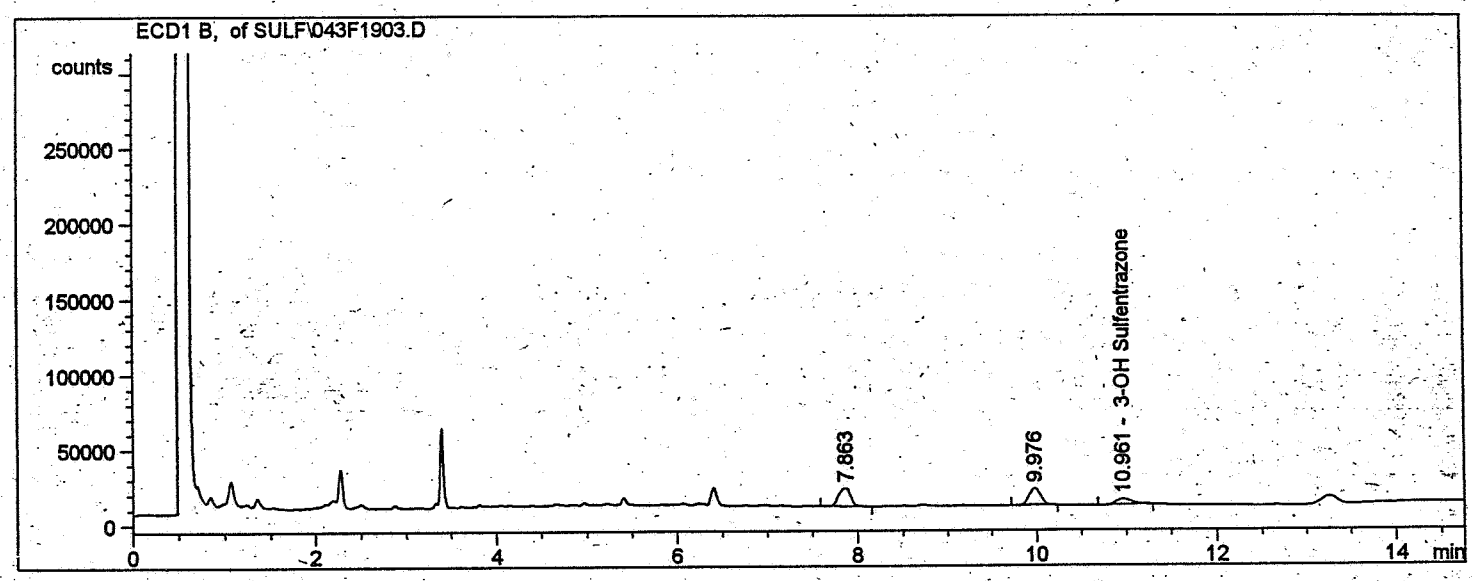
22

Data File C:\HPCHEM\2\DATA\SULF\043F1903.D Sample Name: Grain 0.05-1

2.5 G. Wheat Grain + 0.0493 ppm spike of
3-OH Methyl Sulfentrazone.
1 ml final vol., 2 ul injected.

```
================================================================================
Injection Date   : 2/8/96 10:59:04 PM                Seq. Line :  19
Sample Name      : Grain 0.05-1                          Vial :  43
Acq. Operator    : Mark W. Law                           Inj :   3
                                                    Inj Volume : 2 µl

Sequence File    : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method           : C:\HPCHEM\2\METHODS\SULF2.M
Last changed     : 2/8/96 10:08:23 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
================================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Thursday, February 08, 1996 10:08:23 PM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.863	BB	0.151	1.13121e5	42.48	?
2	9.976	BB	0.144	1.03506e5	38.87	?
3	10.961	BB +	0.190	49692.17578	18.66	3-OH Sulfentraz

Totals : 266320.03125

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

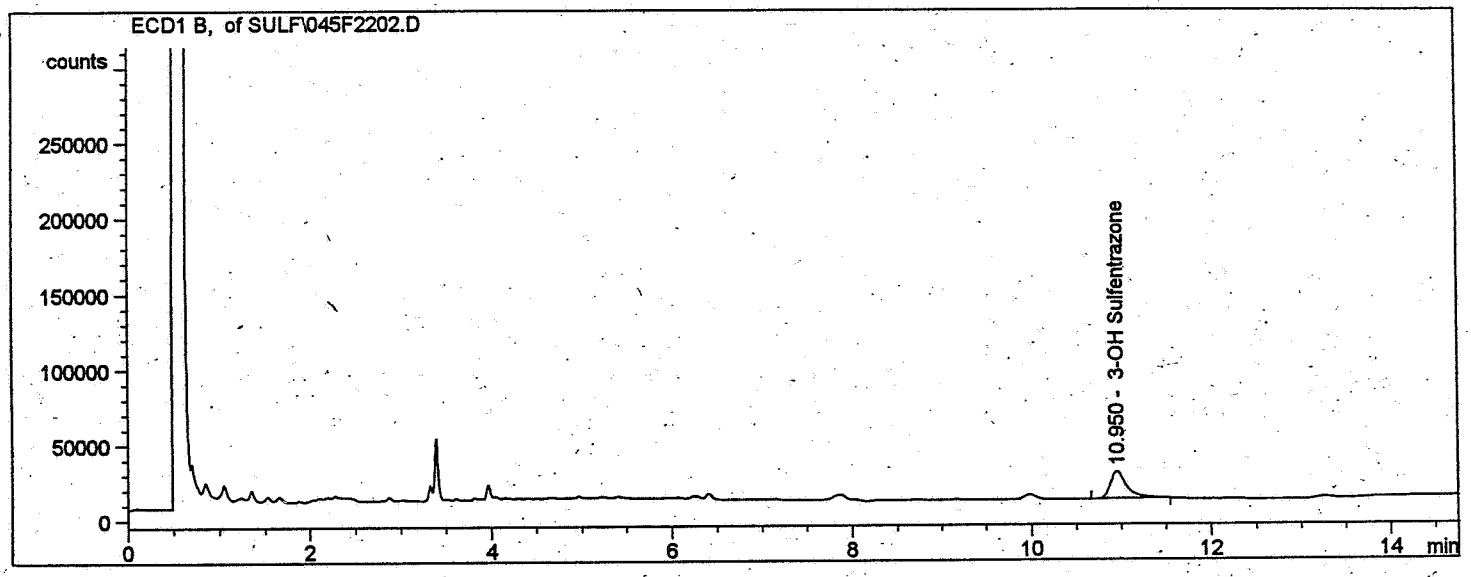
23

Data File C:\HPCHEM\2\DATA\SULF\045F2202.D Sample Name: Grain 0.1-1

2.5 g. Wheat Grain + 0.09968 ppm spike of
3-OH-Methyl Sulfentrazone.
1 ml final vol., 2 ul injected.

```
================================================================================
Injection Date   : 2/9/96 1:53:42 AM                Seq. Line :  22
Sample Name      : Grain 0.1-1                          Vial :  45
Acq. Operator    : Mark W. Law                          Inj :   2
                                                    Inj Volume : 2 µl

Sequence File    : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method           : C:\HPCHEM\2\METHODS\SULF2.M
Last changed     : 2/9/96 1:24:52 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
================================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Friday, February 09, 1996 1:24:52 AM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	10.950	BB +	0.191	2.35299e5	100.00	3-OH Sulfentraz

```
Totals :                              235299.40625

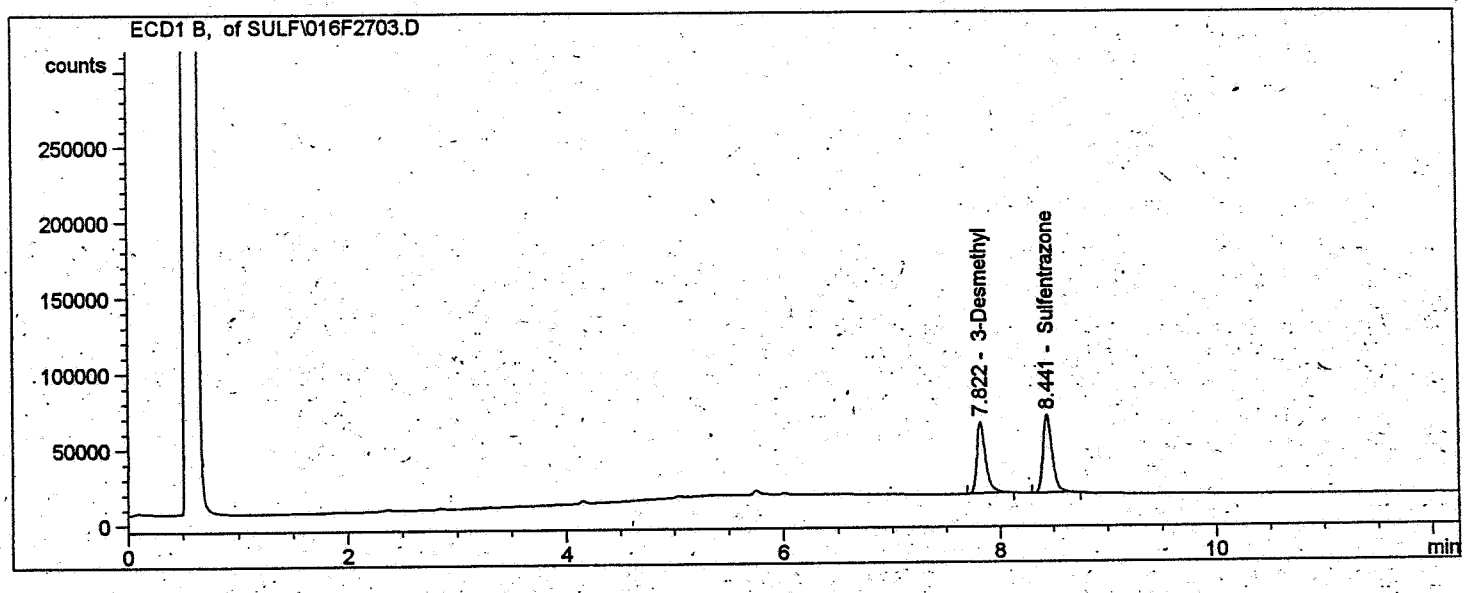
================================================================================
                            *** End of Report ***
```

24

Data File C:\HPCHEM\2\DATA\SULF\016F2703.D Sample Name: 0.1 ng/ul Stds.
3 ul injection of Mixed Standards
0.10014 ng/ul Sulfentrazone and
0.10010 ng/ul 3-Desmethyl Sulfentrazone

Injection Date	: 1/26/96 6:16:00 PM	Seq. Line	: 27
Sample Name	: 0.1 ng/ul Stds.	Vial	: 16
Acq. Operator	: Mark W. Law	Inj	: 3
		Inj Volume	: 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/26/96 2:43:06 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.



Area Percent Report

Sorted by Signal
Calib. Data Modified : Thursday, January 25, 1996 9:38:08 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.822	BB +	0.085	2.66540e5	46.25	3-Desmethyl
2	8.441	BB +	0.092	3.09701e5	53.75	Sulfentrazone
Totals :				576240.12500		

*** End of Report ***

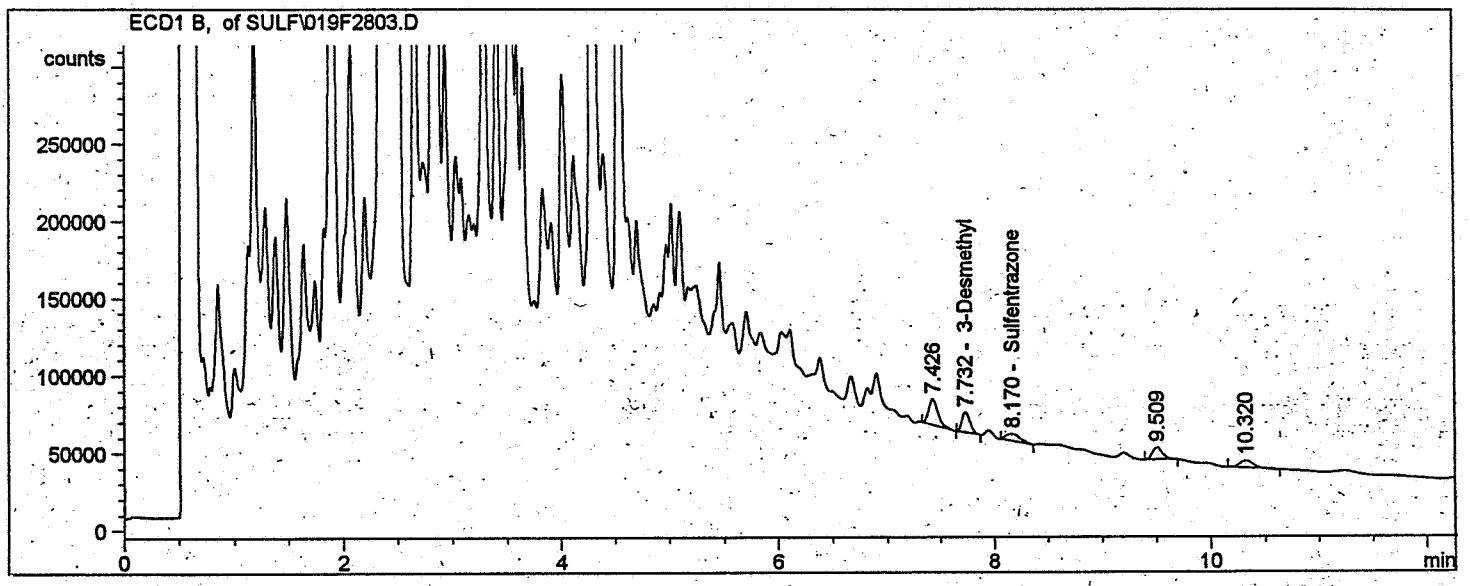
25

Data File C:\HPCHEM\2\DATA\SULF\019F2803.D Sample Name: Forage 0.025-1

2.5 g. wheat forage + 0.025 ppm mixed spike
1 ml final vol., 2 ul injection.

Injection Date	: 1/26/96 7:13:01 PM	Seq. Line :	28
Sample Name	: Forage 0.025-1	Vial :	19
Acq. Operator	: Mark W. Law	Inj :	3
		Inj Volume :	2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/26/96 2:43:06 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.



Area Percent Report

Sorted by Signal
Calib. Data Modified : Thursday, January 25, 1996 9:38:08 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.426	BB		0.091	1.03286e5	33.36	?
2	7.732	BB	+	0.081	70056.35938	22.62	3-Desmethyl
3	8.170	BB	+	0.121	42202.26563	13.63	Sulfentrazone
4	9.509	BB		0.096	50381.91406	16.27	?
5	10.320	BB		0.133	43715.10156	14.12	?

Totals : 309641.50000

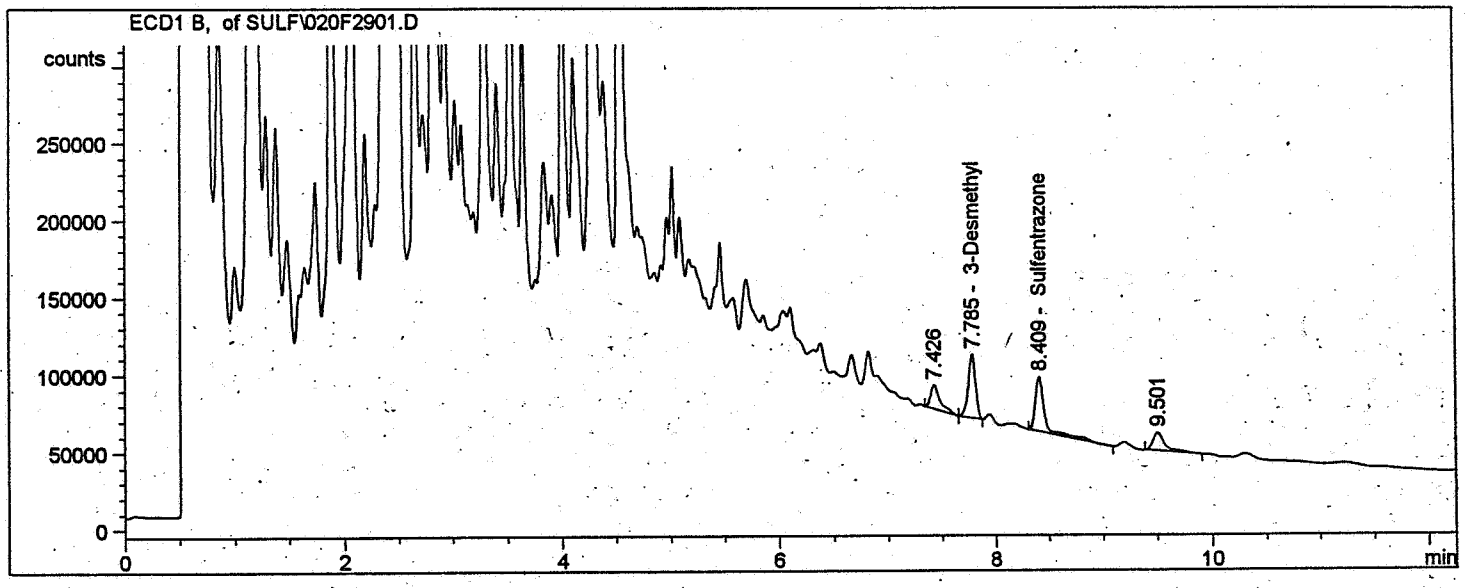
*** End of Report ***

26

Data File C:\HPCHEM\2\DATA\SULF\020F2901.D Sample Name: Forage 0.025-2
2.5 g. wheat forage + 0.025 ppm mixed spike.
1 ml final vol. 2 ml injection.

```
==========================================================================
Injection Date   : 1/26/96 7:32:01 PM          Seq. Line :  29
Sample Name      : Forage 0.025-2                    Vial :  20
Acq. Operator    : Mark W. Law                        Inj :   1
                                               Inj Volume : 2 µl

Sequence File    : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method           : C:\HPCHEM\2\METHODS\SULF.M
Last changed     : 1/26/96 2:43:06 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
==========================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Thursday, January 25, 1996 9:38:08 AM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.426	BB		0.090	99233.26562	16.33	?
2	7.785	BB	+	0.072	1.94683e5	32.05	3-Desmethyl
3	8.409	BB	+	0.093	2.20902e5	36.36	Sulfentrazone
4	9.501	BB		0.113	92670.96875	15.25	?

Totals : 607489.50000

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

27

Data File C:\HPCHEM\2\DATA\SULF\021F3101.D Sample Name: Forage 0.05-1

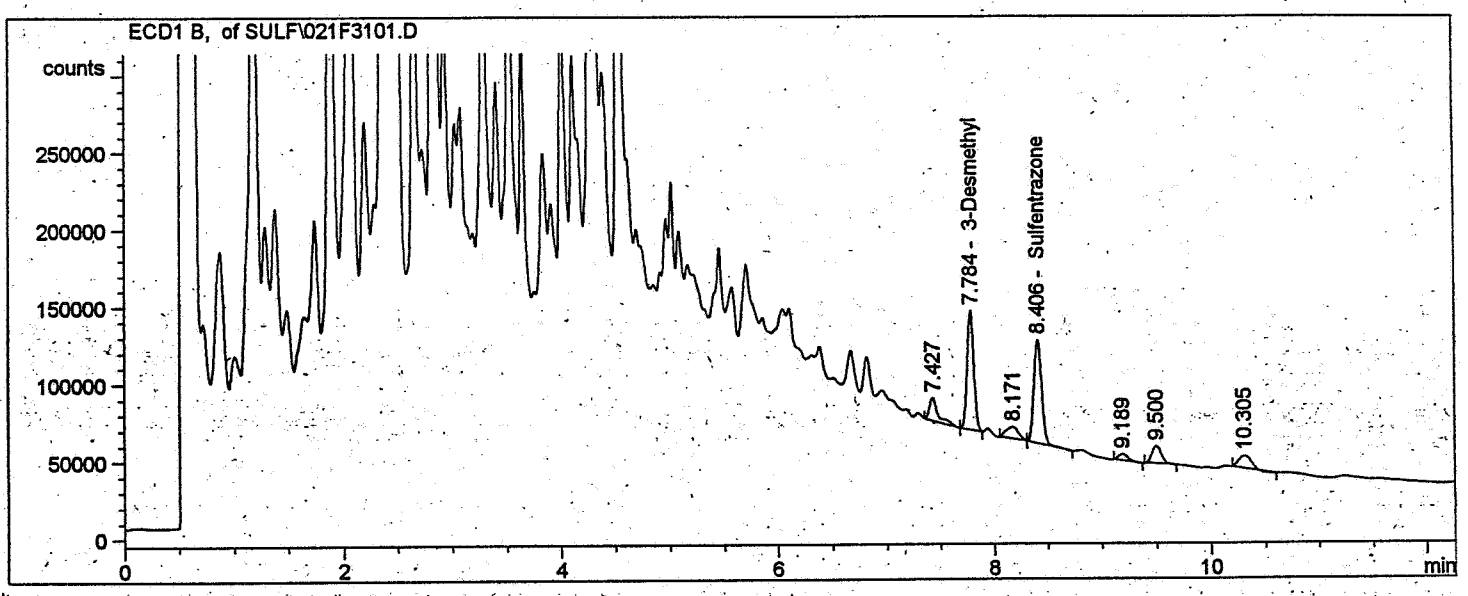
2.5 g. wheat forage + 0.05 ppm mixed spike.
1 ml final vol. 2 ul injection.

==========

Injection Date : 1/26/96 9:26:20 PM Seq. Line : 31
Sample Name : Forage 0.05-1 Vial : 21
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/26/96 2:43:06 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

==========



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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Thursday, January 25, 1996 9:38:08 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.427	BB		0.084	87077.61719	8.83	?
2	7.784	BB	+	0.067	3.36013e5	34.08	3-Desmethyl
3	8.171	BB		0.110	62554.21875	6.34	?
4	8.406	BB	+	0.079	3.40827e5	34.57	Sulfentrazone
5	9.189	BB		0.092	26629.29102	2.70	?
6	9.500	BB		0.093	69018.60937	7.00	?
7	10.305	BB		0.129	63811.48438	6.47	?

Totals : 985930.87500

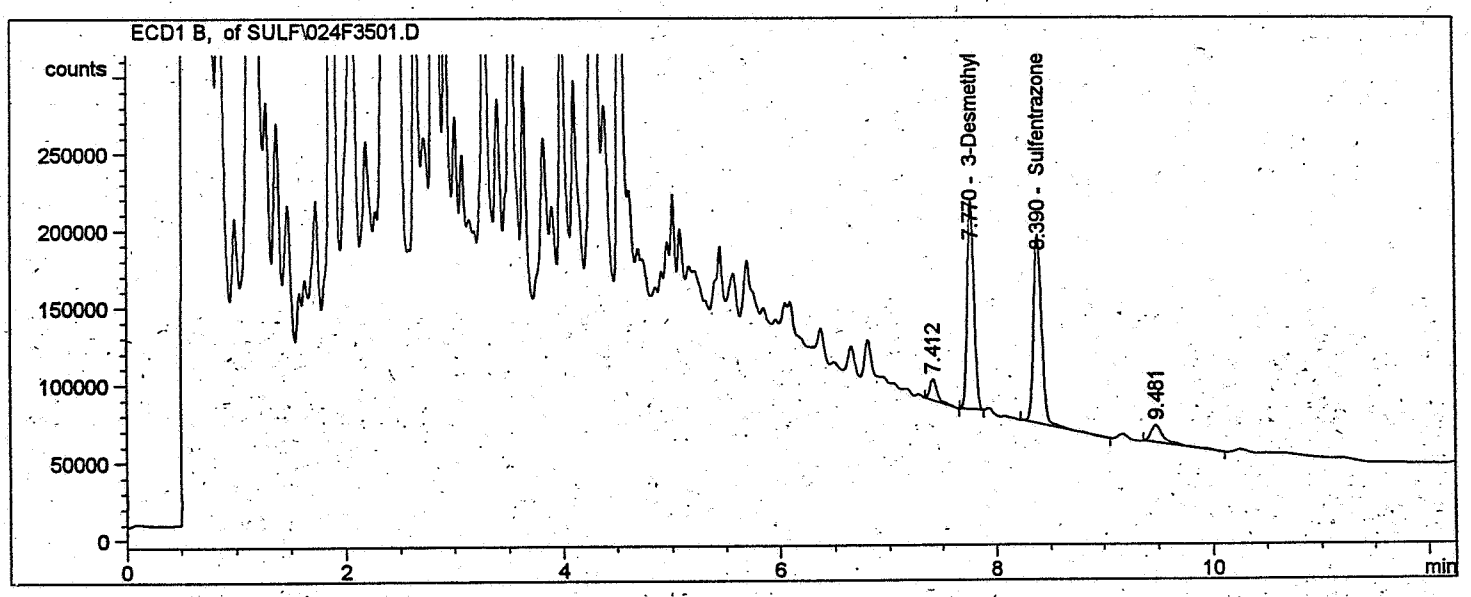
==========28

Data File C:\HPCHEM\2\DATA\SULF\024F3501.D Sample Name: Forage 0.1-1

2.5 g. wheat forage + 0.1 ppm mixed spike.
1 ml final vol. 2 ul injection.

```
================================================================================
Injection Date   : 1/27/96 1:14:46 AM              Seq. Line :  35
Sample Name      : Forage 0.1-1                        Vial :  24
Acq. Operator    : Mark W. Law                          Inj :   1
                                                  Inj Volume : 2 µl

Sequence File    : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Method           : C:\HPCHEM\2\METHODS\SULF.M
Last changed     : 1/26/96 2:43:06 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.
================================================================================
```




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

Sorted by Signal
Calib. Data Modified  :      Thursday, January 25, 1996 9:38:08 AM
Multiplier            :      1.000000
Dilution              :      1.000000

Signal 1: ECD1 B,
```

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.412	BB		0.072	65432.13281	4.68	?
2	7.770	BB	+	0.067	5.93721e5	42.46	3-Desmethyl
3	8.390	BB	+	0.081	6.39217e5	45.71	Sulfentrazone
4	9.481	BB		0.128	99904.71094	7.14	?

```
Totals :                    1398274.62500

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

29

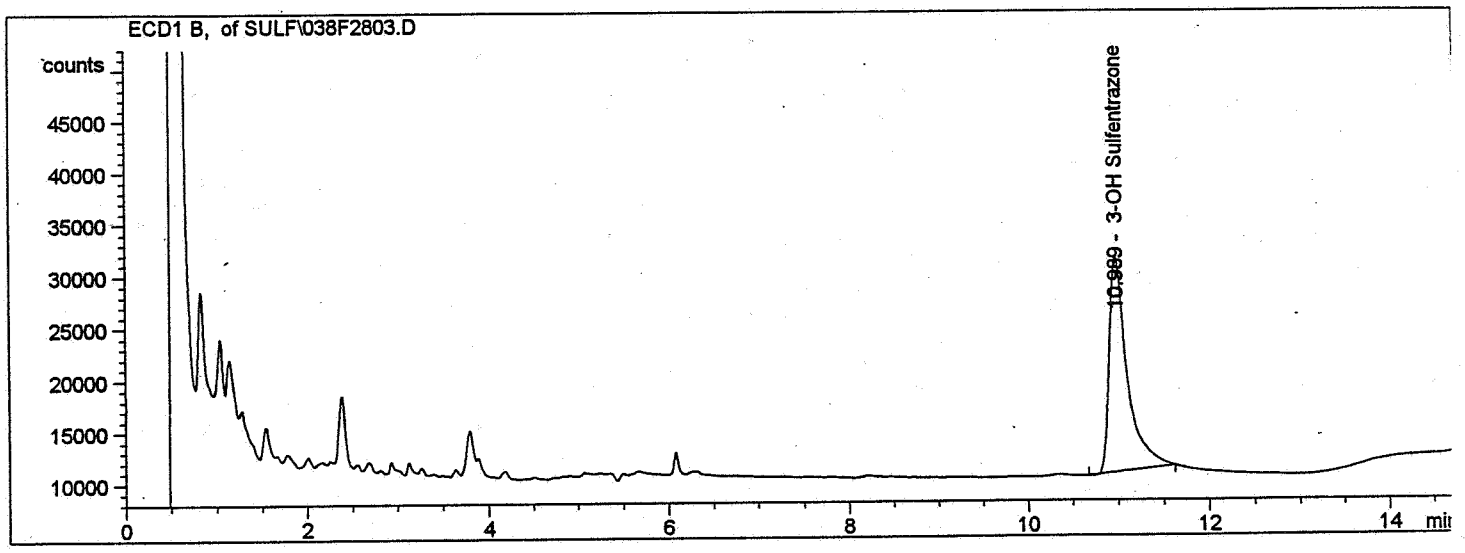
Data File C:\HPCHEM\2\DATA\SULF\038F2803.D Sample Name: 0.1 ng/ul Std.
2 ul injection of Derivitized Standard.
0.0987 ng/ul 3-OH Methyl Sulfentrazone.

==========

Injection Date : 2/14/96 12:27:05 PM Seq. Line : 28
Sample Name : 0.1 ng/ul Std. Vial : 38
Acq. Operator : Mark W. Law Inj : 3
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/14/96 12:20:01 PM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/14/96 12:41:53 PM by Mark W. Law
(recalibrated in sequence after loading)
B95-(76-78) Sulfentrazone on Wheat TMV.

==========



==========

Area Percent Report

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Sorted by Signal
Calib. Data Modified : Wednesday, February 14, 1996 12:20:01 PM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	10.989	BB +	0.205	2.87365e5	100.00	3-OH Sulfentraz

Totals : 287365.09375

==========

*** End of Report ***

30

Data File C:\HPCHEM\2\DATA\SULF\047F2601.D Sample Name: Forage Control 1

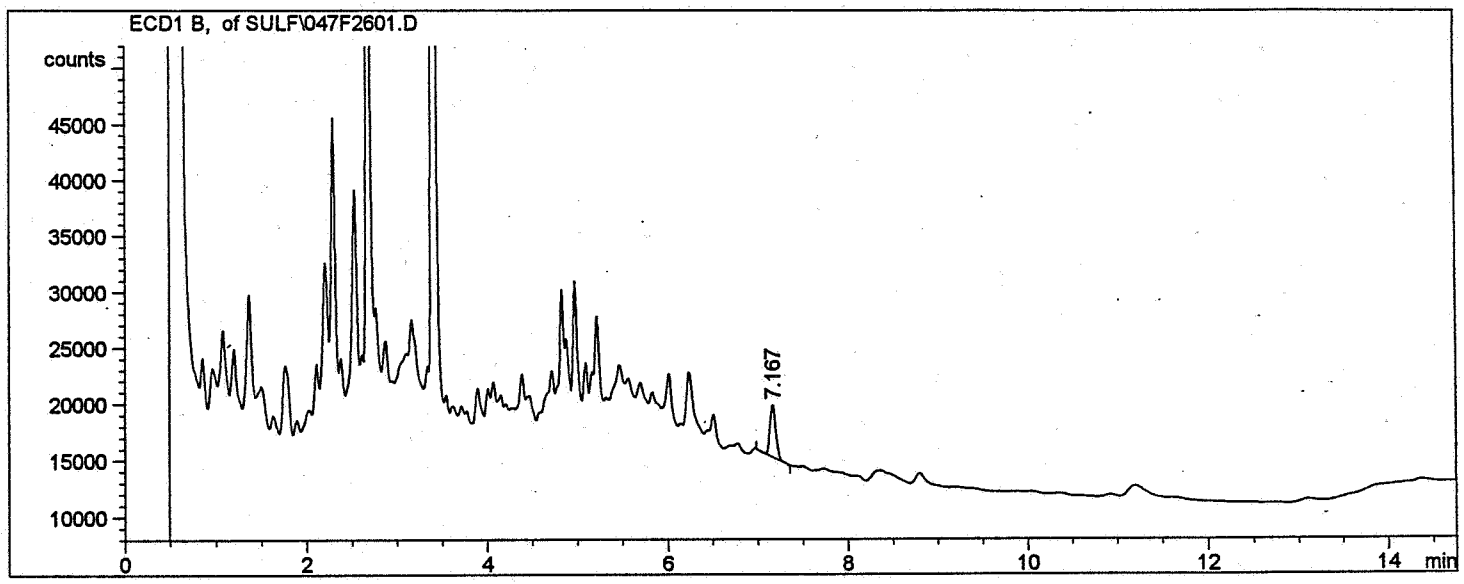
2.5 G. Wheat Forage Control.
1 ml final vol. 2 ul injected.

==========

Injection Date : 2/14/96 9:32:05 AM Seq. Line : 26
Sample Name : Forage Control 1 Vial : 47
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/14/96 9:25:02 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

==========



==========

Area Percent Report

==========

Sorted by Signal
Calib. Data Modified : Wednesday, February 14, 1996 9:25:02 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.167	BB	0.071	20782.40820	100.00	?

Totals : 20782.40820

1 Warnings or Errors :

Warning : Time reference compound(s) not found

==========

*** End of Report ***

31

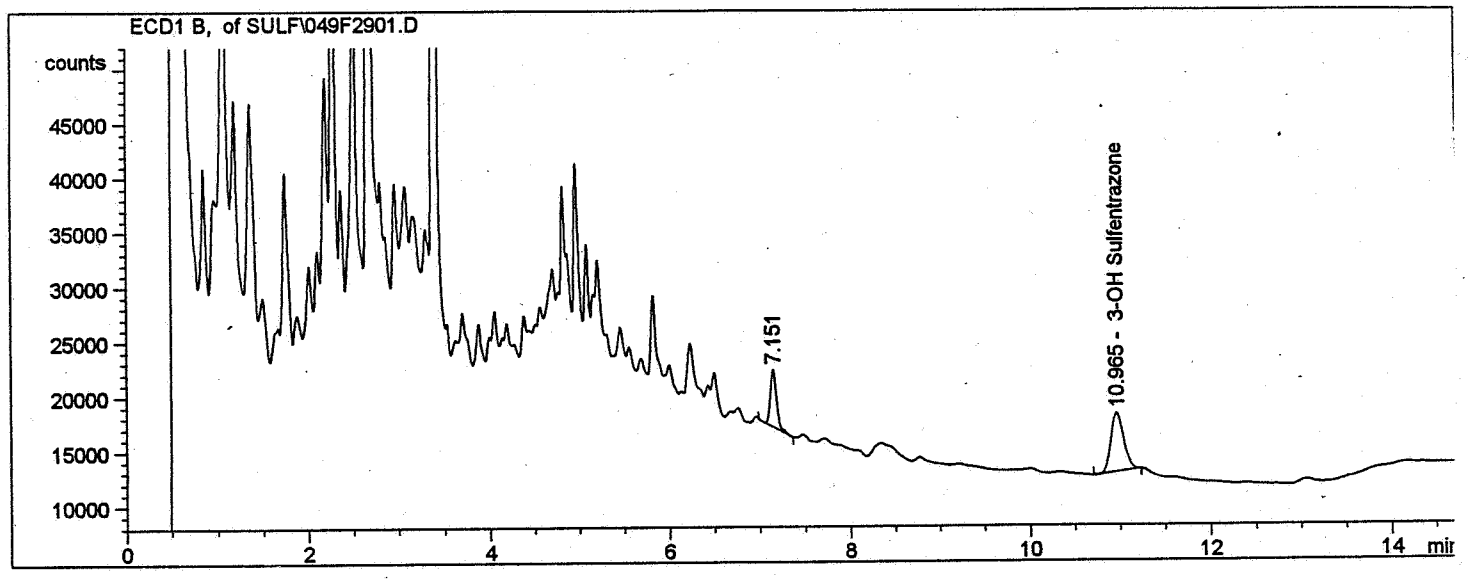
Data File C:\HPCHEM\2\DATA\SULF\049F2901.D Sample Name: Forage 0.025-1
2.5 G. Wheat Forage + 0.0247 ppm Spike
3-Hydroxymethyl Sulfentrazone.
1 ml final vol. 2 ul injection.

==========

Injection Date : 2/14/96 12:48:55 PM Seq. Line : 29
Sample Name : Forage 0.025-1 Vial : 49
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/14/96 12:41:53 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

==========



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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Wednesday, February 14, 1996 12:41:53 PM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.151	BB		0.073	24772.66211	31.81	?
2	10.965	BB	+	0.152	53109.60547	68.19	3-OH Sulfentraz

Totals : 77882.26563

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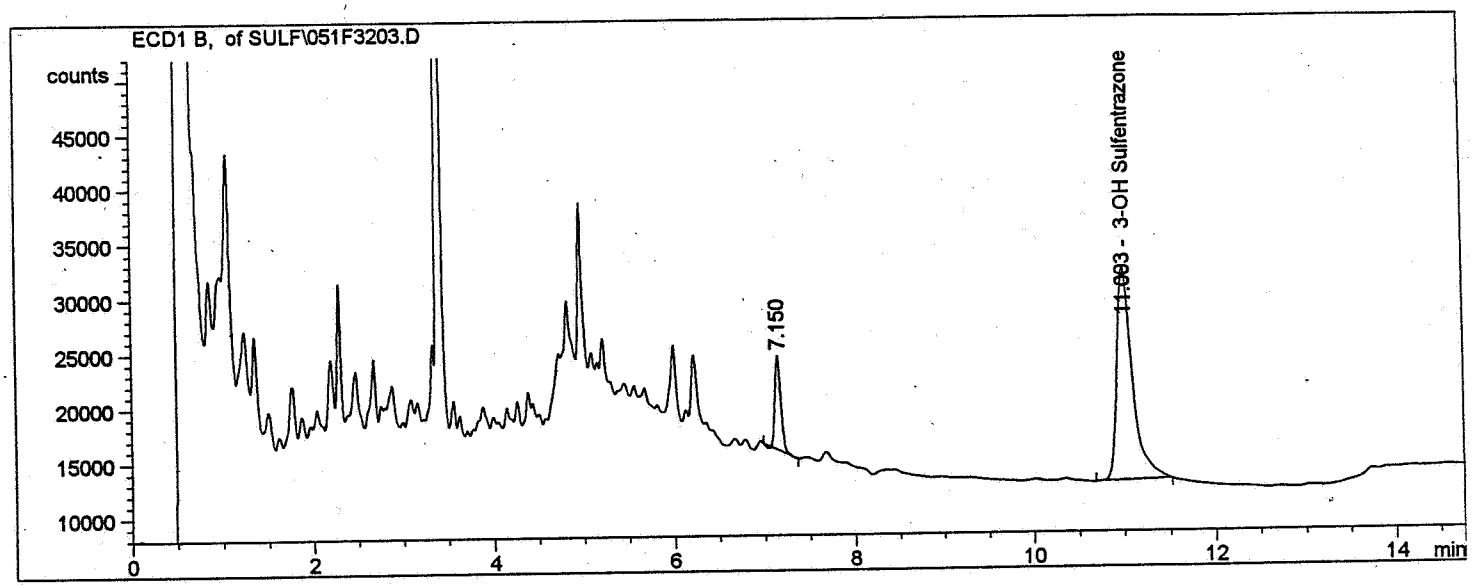
*** End of Report ***

32

Data File C:\HPCHEM\2\DATA\SULF\051F3203.D Sample Name: Forage 0.05-1
2.5 G. Wheat forage + 0.0493 ppm Spike
3-Hydroxymethyl Sulfentrazone.
1 ml final vol. 2 ul injection.

Injection Date	: 2/14/96 4:49:32 PM	Seq. Line	: 32
Sample Name	: Forage 0.05-1	Vial	: 51
Acq. Operator	: Mark W. Law	Inj	: 3
		Inj Volume	: 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/14/96 3:58:32 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.



Area Percent Report

Sorted by Signal
Calib. Data Modified : Wednesday, February 14, 1996 3:58:32 PM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.150	BB	0.069	38165.56641	14.21	?
2	11.003	BB +	0.178	2.30490e5	85.79	3-OH Sulfentraz

Totals : 268655.87500

*** End of Report ***

33

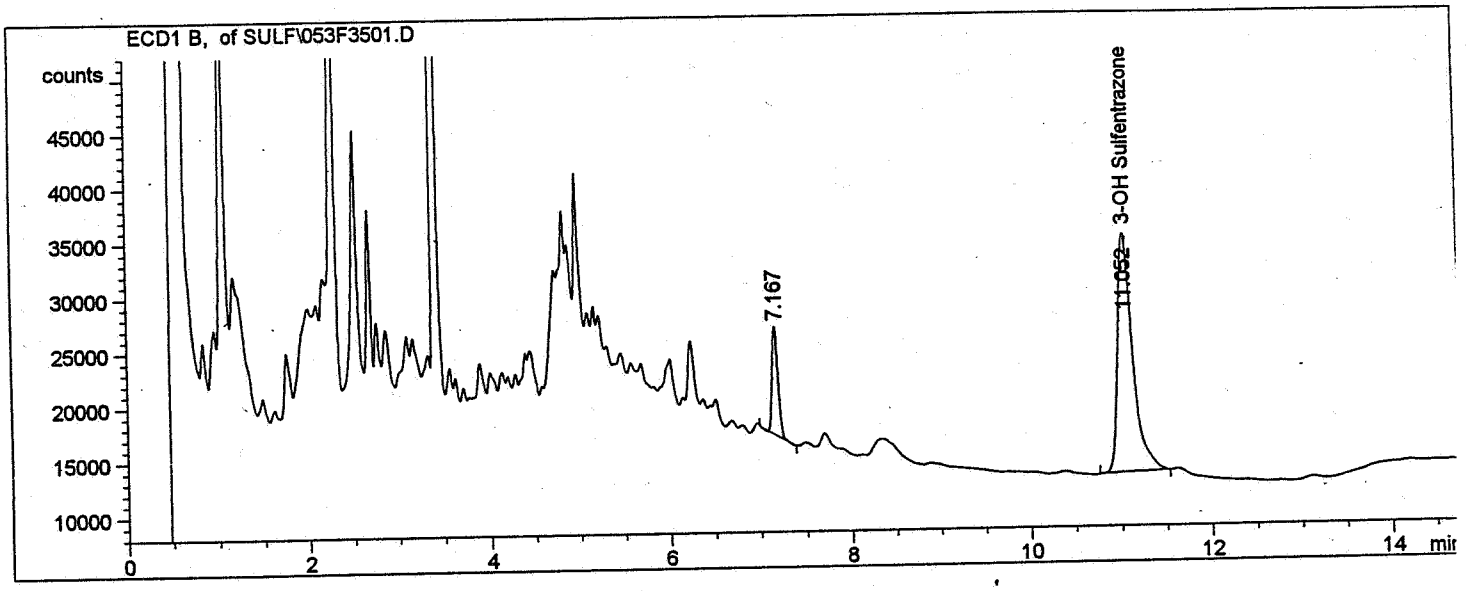
Instrument 2 2/14/96 5:04:24 PM Mark W. Law

Data File C:\HPCHEM\2\DATA\SULF\053F3501.D
2.5 G. Wheat Forage + 0.09968 ppm Spike
3-Hydroxymethyl Sulfentrazone.
1 ml final vol. 2 ul injection.

Sample Name: Forage 0.1-1

Injection Date	: 2/14/96 7:22:34 PM	Seq. Line	: 35
Sample Name	: Forage 0.1-1	Vial	: 53
Acq. Operator	: Mark W. Law	Inj	: 1
		Inj Volume	: 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\OH-SULF.S
Method : C:\HPCHEM\2\METHODS\SULF2.M
Last changed : 2/14/96 7:15:35 PM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.



Area Percent Report

Sorted by Signal
Calib. Data Modified : Wednesday, February 14, 1996 7:15:35 PM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.167	BB	0.069	43382.83203	14.63	?
2	11.052	BB +	0.177	2.53120e5	85.37	3-OH Sulfentraz

Totals : 296502.62500

*** End of Report ***

34

Instrument 2 2/14/96 7:37:24 PM Mark W. Law

Data File C:\HPCHEM\2\DATA\SULF\016F3601.D Sample Name: 0.1 ng/ul Stds.

3 ul injection of Mixed Standards
0.10014 ng/ul Sulfentrazone and
0.10010 ng/ul 3-Desmethyl Sulfentrazone

==========

Injection Date : 2/1/96 9:17:09 AM Seq. Line : 36
Sample Name : 0.1 ng/ul Stds. Vial : 16
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 2/5/96 9:16:59 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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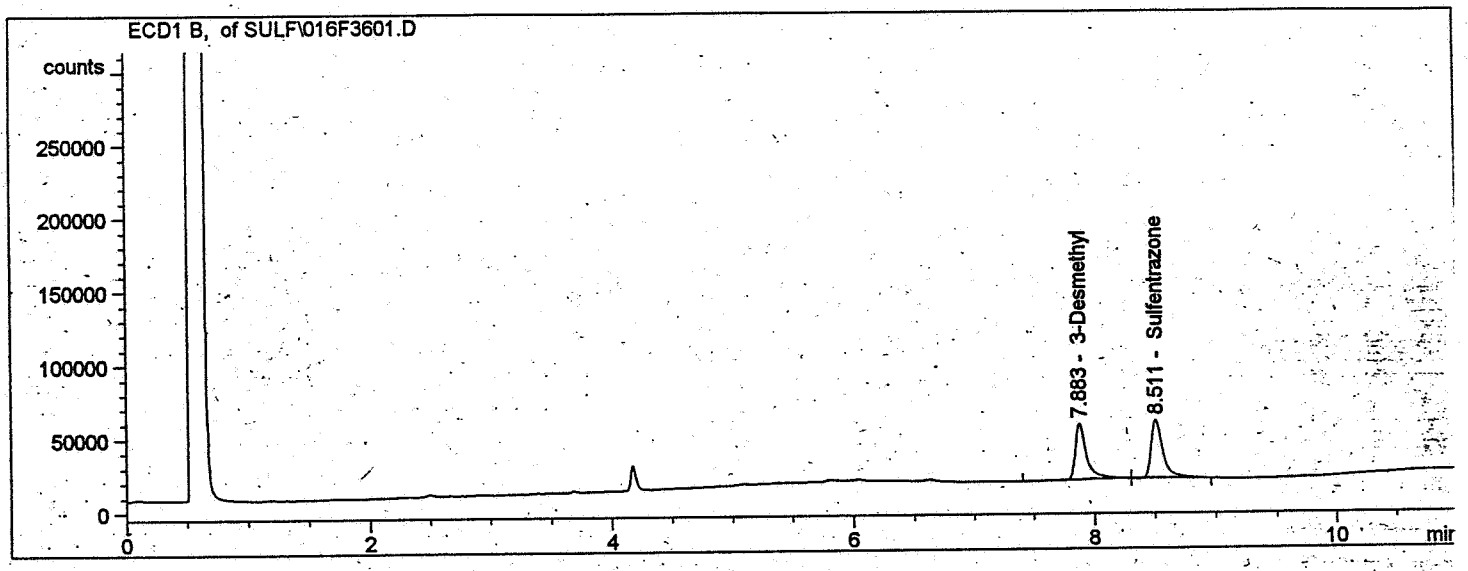

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Monday, February 05, 1996 9:12:26 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.883	BB +	0.096	2.49397e5	48.10	3-Desmethyl
2	8.511	BB +	0.103	2.69085e5	51.90	Sulfentrazone

Totals : 518481.43750

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Time reference compound(s) not found

55

Data File C:\HPCHEM\2\DATA\SULF\025F3702.D Sample Name: Straw Control 1

2.5 g. Wheat Straw
1 ml. final vol. 2 ul inj.

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Injection Date : 2/1/96 10:29:51 AM Seq. Line : 37
Sample Name : Straw Control 1 Vial : 25
Acq. Operator : Mark W. Law Inj : 2
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 2/5/96 9:16:59 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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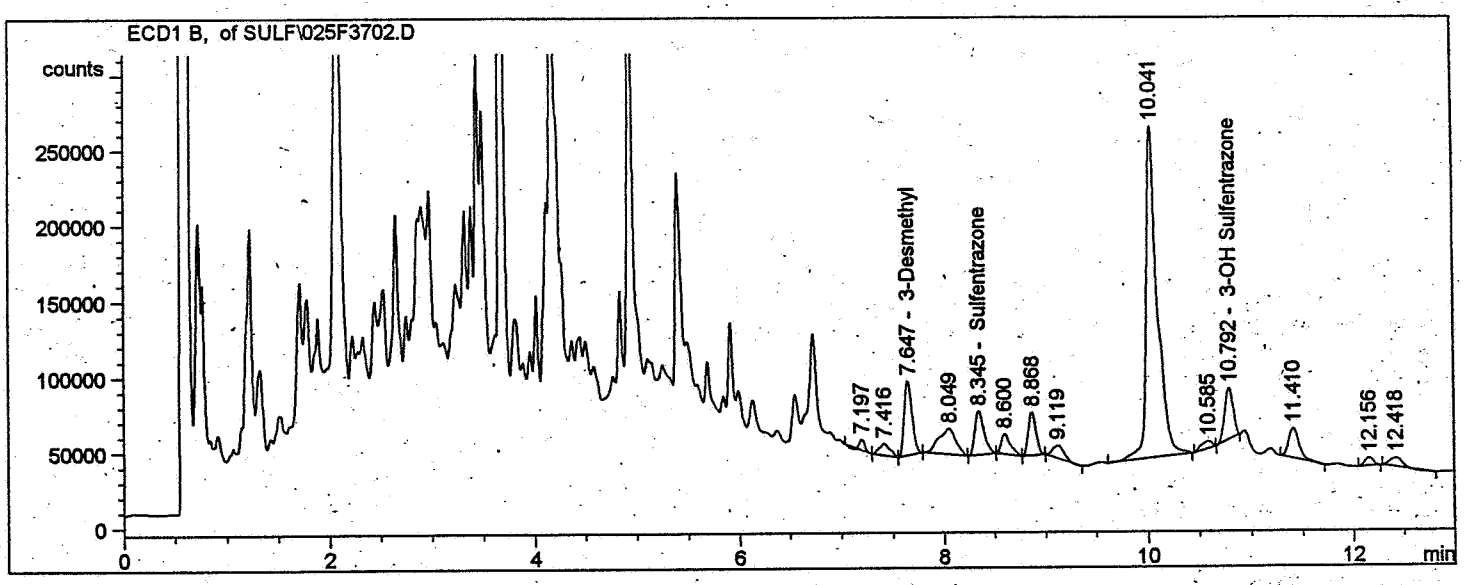

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Monday, February 05, 1996 9:20:31 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type	Width [min]	Area counts*s	Area %	Name
1	7.197	BB	0.054	24738.23438	7.76e-1	?
2	7.416	BB	0.101	62083.89844	1.95	?
3	7.647	BB +	0.086	2.69514e5	8.46	3-Desmethyl
4	8.049	BB	0.161	2.07422e5	6.51	?
5	8.345	BB +	0.098	1.90107e5	5.96	Sulfentrazone
6	8.600	BB	0.095	88918.23437	2.79	?
7	8.868	BB	0.084	1.53459e5	4.81	?
8	9.119	BB	0.121	63040.97266	1.98	?
9	10.041	BB	0.109	1.66903e6	52.36	?
10	10.585	BB	0.115	38690.41797	1.21	?
11	10.792	BB +	0.091	1.96961e5	6.18	3-OH Sulfentraz

36

Data File C:\HPCHEM\2\DATA\SULF\027F4001.D Sample Name: Straw 0.025-1

2.5 g. Wheat Straw + 0.025 ppm mixed spike of
Sulfentrazone and 3-desmethyl sulfentrazone.
1 ml final vol., 2 ul injection.

==========

Injection Date : 2/1/96 1:11:21 PM Seq. Line : 40
Sample Name : Straw 0.025-1 Vial : 27
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 2/5/96 9:21:04 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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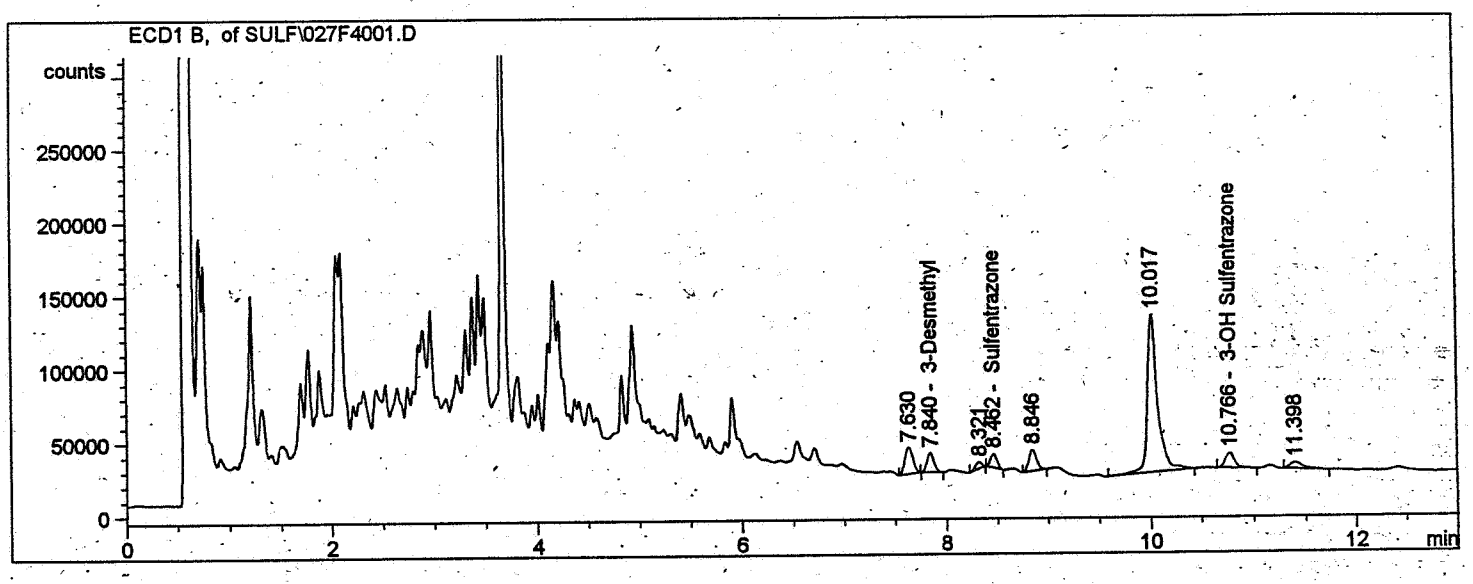

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Monday, February 05, 1996 9:21:13 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.630	BB		0.089	1.01060e5	8.68	?
2	7.840	BB	+	0.074	62167.86328	5.34	3-Desmethyl
3	8.321	BB		0.078	23455.41602	2.01	?
4	8.462	BB	+	0.071	42452.39062	3.64	Sulfentrazone
5	8.846	BB		0.085	77870.19531	6.69	?
6	10.017	BB		0.102	7.51439e5	64.51	?
7	10.766	BB	+	0.097	67381.82031	5.78	3-OH Sulfentraz
8	11.398	BB		0.131	39007.62500	3.35	?

Totals : 1164835.00000

37

Data File C:\HPCHEM\2\DATA\SULF\030F4401.D Sample Name: Straw 0.05-2

2.5 G. Wheat Straw + 0.05 ppm spike of each
Sulfentrazone and 3-desmethyl sulfentrazone.
1 ml final vol., 2 ul injection.

==========

Injection Date : 2/1/96 5:13:26 PM Seq. Line : 44
Sample Name : Straw 0.05-2 Vial : 30
Acq. Operator : Mark W. Law Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 2/5/96 9:21:04 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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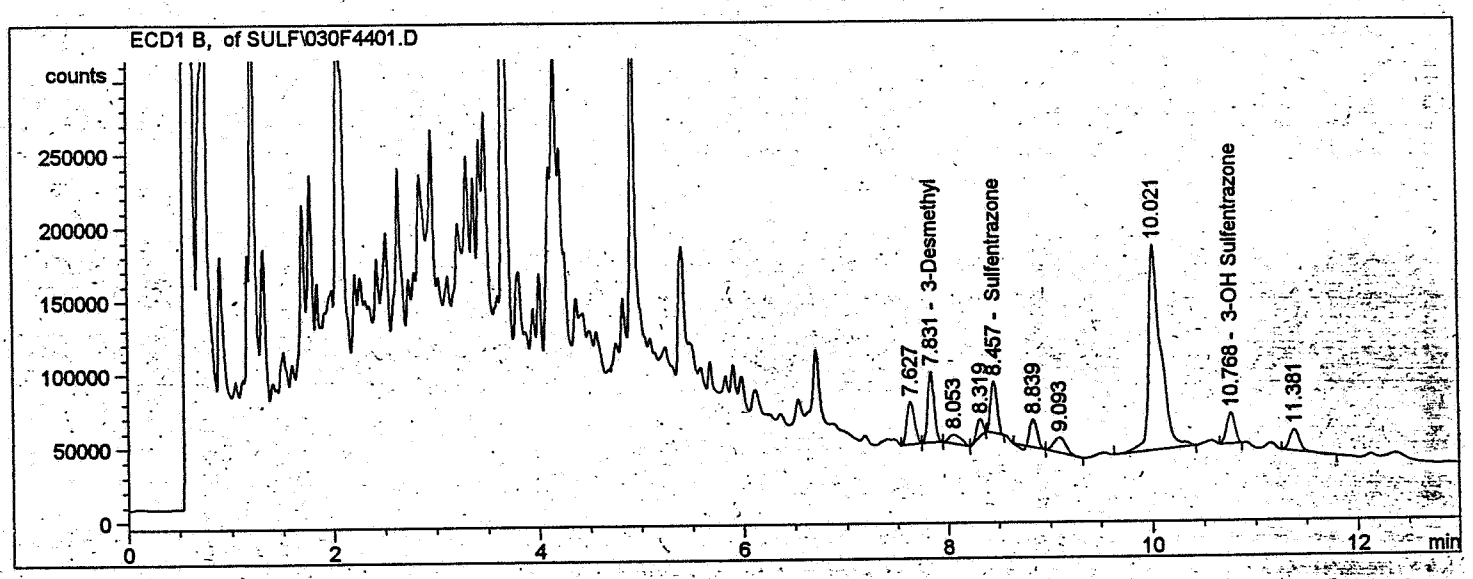

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Monday, February 05, 1996 9:21:58 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.627	BB		0.084	1.54192e5	7.05	?
2	7.831	BB	+	0.068	2.13091e5	9.75	3-Desmethyl
3	8.053	BB		0.152	54266.82813	2.48	?
4	8.319	BB		0.070	52295.62500	2.39	?
5	8.457	BB	+	0.071	1.56547e5	7.16	Sulfentrazone
6	8.839	BB		0.076	87589.32812	4.01	?
7	9.093	BB		0.130	84349.33594	3.86	?
8	10.021	BB		0.117	1.16989e6	53.51	?
9	10.768	BB	+	0.090	1.20384e5	5.51	3-OH Sulfentraz
10	11.381	BB		0.099	93804.20313	4.29	?

78

Data File C:\HPCHEM\2\DATA\SULF\031F4603.D Sample Name: Straw 0.1-1

2.5 g. Wheat Straw + 0.1 ppm each of
Sulfentrazone and 3-desmethyl sulfentrazone
1 ml final vol., 2 ul injection.

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Injection Date : 2/1/96 7:54:58 PM Seq. Line : 46
Sample Name : Straw 0.1-1 Vial : 31
Acq. Operator : Mark W. Law Inj : 3
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\SULFLIN1.S
Acq. Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 1/30/96 8:39:27 AM by Mark W. Law
Analysis Method : C:\HPCHEM\2\METHODS\SULF.M
Last changed : 2/5/96 9:21:04 AM by Mark W. Law
B95-(76-78) Sulfentrazone on Wheat TMV.

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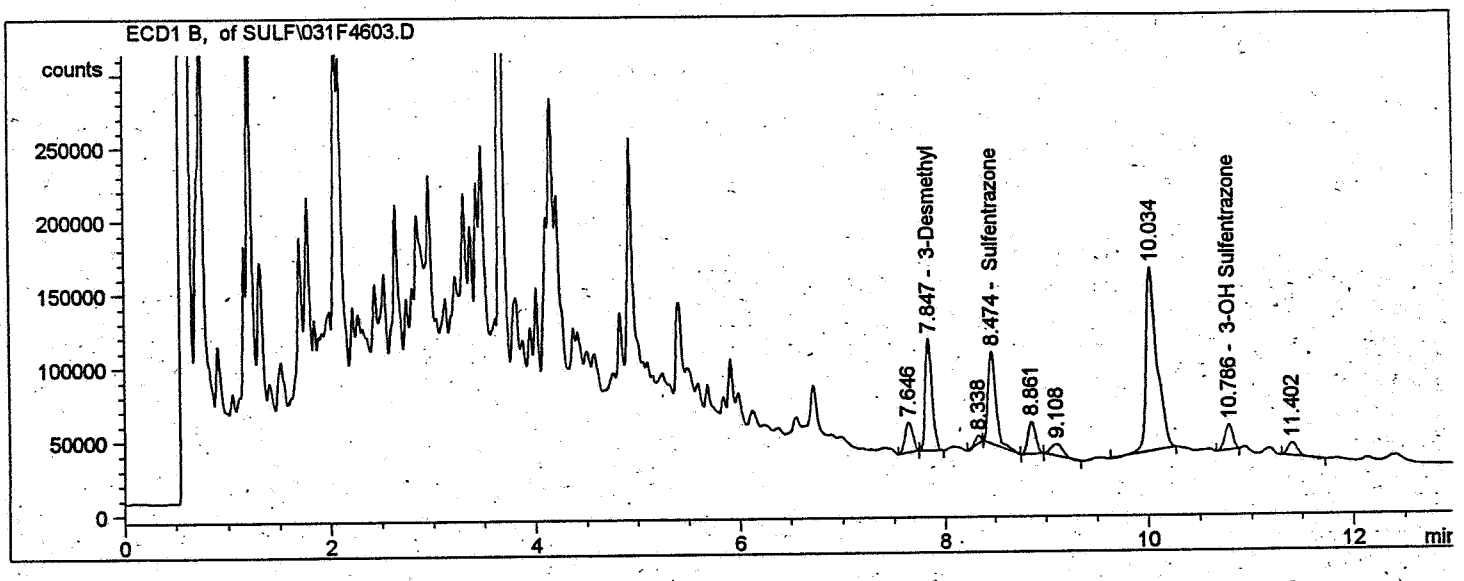

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Area Percent Report

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Sorted by Signal
Calib. Data Modified : Monday, February 05, 1996 9:22:41 AM
Multiplier : 1.000000
Dilution : 1.000000

Signal 1: ECD1 B,

Peak #	RT [min]	Type		Width [min]	Area counts*s	Area %	Name
1	7.646	BB		0.090	1.12686e5	5.44	?
2	7.847	BB	+	0.072	3.52178e5	17.00	3-Desmethyl
3	8.338	BB		0.065	21851.35352	1.05	?
4	8.474	BB	+	0.083	3.32383e5	16.05	Sulfentrazone
5	8.861	BB		0.084	1.16971e5	5.65	?
6	9.108	BB		0.127	62197.24609	3.00	?
7	10.034	BB		0.106	9.19952e5	44.41	?
8	10.786	BB	+	0.087	95174.58594	4.59	3-OH Sulfentraz
9	11.402	BB		0.100	57892.55859	2.80	?

39