2-12-97

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

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

FEB 12 1997
FEB 12 '97

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP# 4F04407. Sulfentrazone(Authority Herbicide)on Soybeans and Rotational Crops. Method Validation Report.

FROM: Juan F. Negron, Chemist
Mark W. Law, Chemist
Analytical Chemistry Section

THRU: Dallas P. Wright, Jr, Acting Head
Analytical Chemistry Section

THRU: Donald A. Marlow, Chief
Analytical Chemistry Branch

TO: E. T. Haeberer, Acting Chief
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 its major metabolite 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) on soybean seed at the 0.025 and 0.050 ppm levels. We were also requested to run a method validation for sulfentrazone plus the metabolites 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone (N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-5-oxo-1H-1,2,4-triazol-1-yl] phenyl] methanesulfonamide) on wheat forage, wheat straw, and wheat grain. The requested fortification levels were 0.025, 0.100, and 0.200 ppm for wheat forage; 0.050, 0.300, and 0.600 ppm for wheat straw; and 0.025, 0.050, and 0.100 ppm for wheat grain. ACL used FMC Corporation Method 162MVL96R1, "Analytical Methodology for the Determination of Sulfentrazone, 3-Desmethyl Sulfentrazone, and 3-Hydroxymethyl Sulfentrazone in/on Various Matrixes", dated 09/13/96 (Appendix B of MRID No. 441188-01) for the validation study.

1

METHOD SUMMARY:

A 10g sample is extracted by refluxing with acetone/0.25N HCl for an hour. After concentration, the aqueous concentrate is acidified to 1 N, refluxed for 2 hours, cooled, and filtered. The sample is then passed through C-8 and silica gel solid phase extraction (SPE) cartridges for clean-up. After concentrating, the HMS analyte is derivatized with N,O-bis [trimethylsilyl] trifluoracetamide (BSTFA). The derivatized solutions passed through a second silica gel SPE cartridge for additional clean-up. The eluate is then concentrated and brought to a final volume with acetonitrile. Analysis is performed on a GC equipped with a 35% phenyl methyl silicone megabore column and an electrolytic conductivity (ELCD) detector.

COMMENTS

1. The analytical standard was not available from the EPA standards repository in RTP as 12/10/96. Standards and commodities for this method trial were provided by the petitioner.

2. Our instrument has been upgraded from an ELCD to the more modern O.I. model 5360 XSD (Halogen Specific Detector) connected to a model 5300 detector controller. This detector works on the same principle as the ELCD but uses only air (or oxygen) and eliminates the need for isopropanol and the associated resin and pump. The petitioner agreed that the XSD was considered equivalent equipment to the ELCD.

3. A set of eight samples can be extracted, cleaned up, and injected into the GC/XSD in two working days.

4. **CRITICAL STEP**. It was determined that step #7 on page 51 is a very critical step in the method. If the samples are not taken to near dryness at this stage, the small amount of derivitizing reagent remaining with the sample will reduce the chromatographic response of the 3-HMS metabolite and result in poor recovery of that compound.

5. ACL discovered that the DB-35 GC column installed at the beginning of the method trial was defective. While waiting for the new DB-35 column to arrive, we used a DB-17 megabore column for preliminary work The DB-17 column performed satisfactorily and could be included in the method as an alternate column.

6 A copy of ACL's pre-review is attached as additional information for this study.

7. This method meets the requirements in the Pesticide Assessment Guidelines, Subdivision O, Section 171-4(b) for soybean seed, wheat forage, wheat straw, and wheat grain provided these comments and those in the pre-review report are incorporated into the method.

8. The limit of detection has been estimated to be 0.008 PPM. We concur with the LOQ of 0.025 PPM as stated by the petitioner in the method description..

METHOD: "Analytical Methodology for the Determination of Sulfentrazone, 3-Desmethyl Sulfentrazone, and 3-Hydroxymethyl Sulfentrazone in/on Various Matrixes", dated 09/13/96 (Appendix B of MRID No. 441188-01).

	CHEMICALS ADDED								
	SULFENTRAZONE			3-Hydroxymethyl Sulfentrazone			3-DESMETYL SULFENTRAZONE		
Commodity	PPM Added	PPM Found	Percent Recovery	PPM Added	PPM Found	Percent Recovery	PPM Added	PPM Found	Percent Recovery
Soybean Seed	0	N.D.*		0	N.D.*				
	0	N.D.*		0	N.D.*				
	0.0250	0.0196	78.4	0.0250	0.0205	82.0			
	0.0249	0.0194	77.9	0.0249	0.0198	79.5			
	0.0504	0.0401	79.6	0.0500	0.0400	80.0			
	0.0504	0.0426	84.5	0.0499	0.0442	88.6			
Wheat Forage	0	N.D.*	80.1 ± 2.8	0	N.D.*	82.5 ± 4.2	0	N.D.	
	0	N.D.*		0	N.D.*		0	N.D.	
	0.0252	0.0207	82.1	0.0251	0.0311	124	0.025	0.0226	90.4
	0.0252	0.0177	70.2	0.0251	0.0311	124	0.025	0.0194	77.6
	0.101	0.0724	71.7	0.100	0.114	114	0.100	0.0757	75.7
	0.101	0.0655	64.9	0.100	0.0958	95.8	0.100	0.0696	69.6
	0.202	0.161	79.7	0.201	0.223	111	0.201	0.173	86.1
	0.202	0.151	74.8	0.201	0.193	96.0	0.201	0.152	75.6
			73.9 ± 6.3			111 ± 13			79.2 ± 7.7

*N.D. = Not Detected at a LOD of 0.008 ppm.

METHOD: "Analytical Methodology for the Determination of Sulfentrazone, 3-Desmethyl Sulfentrazone, and 3-Hydroxymethyl Sulfentrazone in/on Various Matrixes", dated 09/13/96 (Appendix B of MRID No. 441188-01).

	CHEMICALS ADDED								
	SULFENTRAZONE			3-Hydroxymethyl Sulfentrazone			3-DESMETYL SULFENTRAZONE		
Commodity	PPM Added	PPM Found	Percent Recovery	PPM Added	PPM Found	Percent Recovery	PPM Added	PPM Found	Percent Recovery
WHEAT STRAW	0	N.D.*		0	N.D.*		0	N.D.*	
	0	N.D.*		0	N.D.*		0	N.D.*	
	0.0505	0.0505	100	0.0502	0.0505	101	0.0502	0.0494	98.4
	0.0505	0.0479	94.9	0.0502	0.0567	113	0.0502	0.0431	85.9
	0.302	0.242	80.1	0.300	0.318	106	0.301	0.272	90.4
	0.303	0.245	80.9	0.301	0.316	105	0.301	0.238	79.1
	0.605	0.545	90.1	0.602	0.740	123	0.602	0.546	90.7
	0.605	0.485	80.2	0.602	0.723	120	0.602	0.428	71.1
WHEAT GRAIN	0	N.D.*	87.7 ± 8.6	0	N.D.*	111 ± 5.5	0	N.D.*	85.9 ± 9.6
	0	N.D.*		0	N.D.*		0	N.D.*	
	0.0252	0.0231	91.7	0.0251	0.0155	61.8	0.0251	0.0226	90.0
	0.0252	0.0262	104	0.0251	0.0210	83.7	0.0251	0.0265	106
	0.0504	0.0497	98.6	0.0502	0.0311	62.0	0.0501	0.0459	91.6
	0.0504	0.0484	96.0	0.0501	0.0335	66.9	0.0502	0.0461	91.8
	0.101	0.114	113	0.100	0.0707	70.7	0.100	0.103	103
	0.101	0.121	120	0.100	0.0722	72.2	0.100	0.116	116

104 ± 11 61.6 ± 5.2 99.7 ± 10.4

*N.D. = Not Detected at a LOD of 0.008 ppm.

Modifications to method (major or minor):

See Comments

Special precautions to be taken:

None

Source of analytical reference standards:

Standards were provided by FMC Corporation.

If derivatized standards used, give source:

Standards derivatized as per method.

Instrument for Confirmation:

None

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

Halogen Specific Detector (XSD) used instead of Electrolytic Conductivity Detector (ELCD).
GC temperature ramp modified to improve chromatography:

Initial Temp. 180^0 C.
Hold 1 min.
Rate 20^0 C/min.
Final Temp. 290^0 C.
Final Time 10 min.

Injection temperature was also raised from 250^0 C to 270^0 C.

Commercial source for any special chemicals or apparatus:

Sources for all equipment and reagents given in method.

Comments:

See attached report.

Chromatograms:

Representative chromatograms attached.

TMV Pre-review of Sulfentrazone

Reviewed by: Everett S. Greer, Jr. ESG

Date: 10-30-96

Laboratory assignment numbers: B97-1,2,3,4

Petitioner: FMC Corporation

Independent validation laboratory: Centre Analytical Laboratories, INC.

Analyte: Sulfentrazone, 3-Hydroxymethyl Sulfentrazone and 3-Desmethyl Sulfentrazone

Commodities: Soybean seed, wheat forage, wheat straw and wheat grain

Method: Analytical Methodology for the Determination of Sulfentrazone, 3-Desmethyl sulfentrazone, and 3-Hydroxymethyl Sulfentrazone in/on Various Matrices

V. Analytical procedure

A. Residue method

5. C_8 cartridge solid phase extraction (SPE)

This step requires the SPE cartridge to be "completely" dried under nitrogen after elution of the sample extract. The method uses a Supelco manifold drying attachment capable of drying up to 12 cartridges simultaneously. Cartridges can probably be dried individually using a cartridge adapter fitted to a nitrogen source.

Additional reviewer's comments

1. Recovery data are included with the method for all of the commodities listed in the validation request. Recovery data are provided for wheat samples spiked with parent sulfentrazone, sulfentrazone-3-carboxylic acid(SCA) and 3-hydroxymethyl sulfentrazone(HMS). CBTS has asked ACL to validate the method for samples spiked with 3-desmethyl sulfentrazone(DMS) instead of SCA. No recovery data are provided for samples spiked with DMS, but SCA is converted to DMS during the HCl reflux step. No soybean recovery data are included for SCA.

Representative chromatograms are included with the method for soybeans but not for the other requested commodities (wheat forage, straw and grain). A chromatogram for a wheat hay sample spiked with sulfentrazone, SCA and DMS is provided.

2. The independent validation laboratory validated the method for wheat forage spiked with sulfentrazone, SCA and HMS at the 0.025 ppm and 0.2 ppm levels. Representative chromatograms are included with the ILV report. The report stated that the trial was successful on the first attempt without any modifications.

3. The analyst should be aware of the potential problems listed in Section H.

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ANALYTICAL CHEMISTRY BRANCH
SCREEN FOR RESIDUE METHODS FOR TMV

1. LABORATORY ASSIGNMENT NUMBER: B97-1,2,3,4

2. PP#: 4F04407

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

4. DATE: 10-30-96

5. ANALYTES/LEVEL: Sulfentrazone, 3-hydroxymethyl sulfentrazone, 3-desmethyl Sulfentrazone / 0.025, 0.05, 0.1, 0.2, 0.3, 0.6 ppm

6. COMMODITIES: Soybean seed, Wheat straw, forage, grain

7. METHOD: Analytical Methodology for the Determination of Sulfentrazone, 3-desmethyl Sulfentrazone and 3-Hydroxymethyl Sulfentrazone in/on various Matrices

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?		✓
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?	✓	

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	YES	NO
4. Has recovery data been provided to ACL for the residues that are specified in the TMV request?	See narrative review	
5. Are recovery values between 70% and 120% at all levels and for all commodity types?	Three recoveries are between 131% and 125%	
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?	✓	
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.

Ernest S. Grant — Signature

10-30-96 — Date

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

	YES	NO
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?		✓

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Project #	B97-1,2,3,4
Date	12/4/96
Chemists	Juan F. Negron & Mark Lacy
Compound	Sulfentrazone, DMS, 3-HMS
Instrument	GC
Detector	ELCD-XSD
Column	DB-35, 30M, 0.53mm, 0.8uM
Reactor Temp	1100 deg
Range	(0.04 to 1) ug/ml
Injection	3ul
Convertion	1ug/ml = 1Ng/ul

Sulfentrazone

Run #	Pk. Ht 1	Pk. Ht 2	Pk. Ht 3	Pk. Ht avg.	Conc Ng/ul	On col. Ng	
501..503	585	351	506	481	0.04038	0.12117	
504..506	1359	1326	1293	1326	0.10097	0.30291	
507..509	3363	2802	3165	3110	0.3079	0.9237	
510..512	11421	10661	11323	11135	1.0097	3.0291	
						0	22.02619
						4	14611.01

Regression Output:

Constant	22.02619
Std Err of Y Est	248.1394
R Squared	0.998271
No. of Observations	4
Degrees of Freedom	2
X Coefficient(s)	3647.247
Std Err of Coef.	107.3218

DMS

Run #	Pk. Ht 1	Pk. Ht 2	Pk. Ht 3	Pk. Ht avg.	Conc Ng/ul	On col. Ng	
501..503	212	176	191	193	0.04018	0.12054	
504..506	1108	1102	1142	1117	0.1005	0.3015	
507..509	2214	1874	2148	2079	0.3014	0.9042	
510..512	8533	8113	8223	8290	1.0047	3.0141	
						0	-60.8844
						4	10926.46

Regression Output:

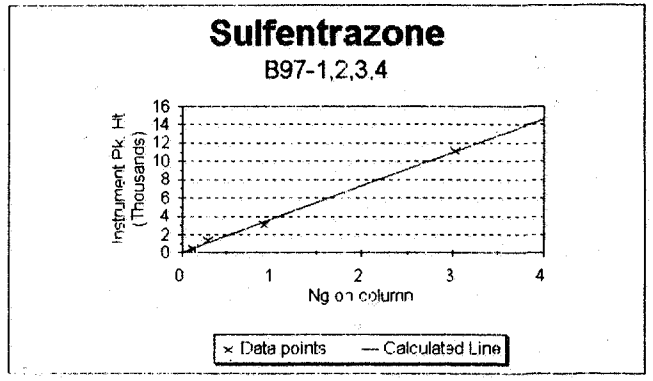
Constant	-60.8344
Std Err of Y Est	354.9732
R Squared	0.993735
No. of Observations	4
Degrees of Freedom	2
X Coefficient(s)	2746.837
Std Err of Coef.	154.2771

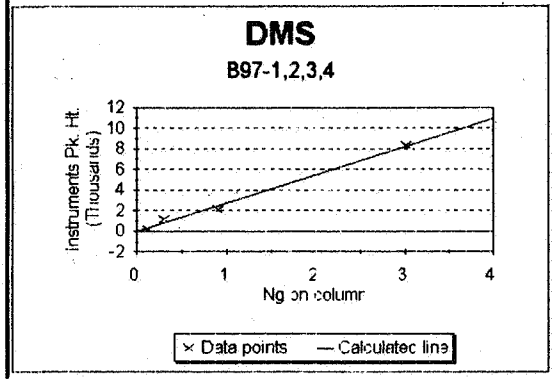
3-HMS

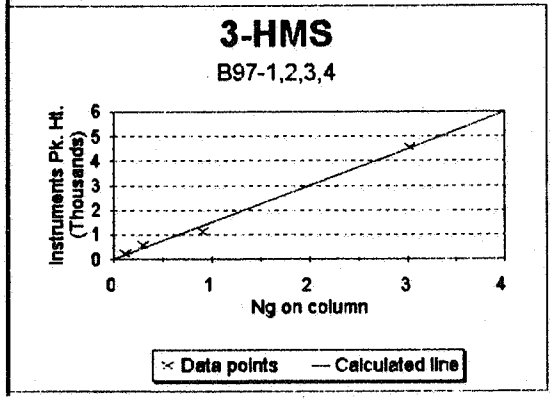
Run #	Pk. Ht 1	Pk. Ht 2	Pk. Ht 3	Pk. Ht avg.	Conc Ng/ul	On col. Ng	
501..503		217	269	243	0.04016	0.12048	
504..506	636	554	599	596	0.1004	0.3012	
507..509	1337	912	1194	1148	0.3012	0.9036	
510..512	4776	4376	4524	4559	1.0041	3.0123	
						0	21.73755
						4	5977.794

Regression Output:

Constant	21.73755
Std Err of Y Est	185.087
R Squared	0.994195
No. of Observations	4
Degrees of Freedom	2
X Coefficient(s)	1489.014
Std Err of Coef.	80.45718







RUN# 1294 JAN 30, 1997 19:09:13

SAMPLE NAME: WG.1-1 SAMPLE# 50
10.0223 G OF WHEAT GRAIN IN 100ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 15 ML: SPIKE LEVEL OF 0.1PPM.

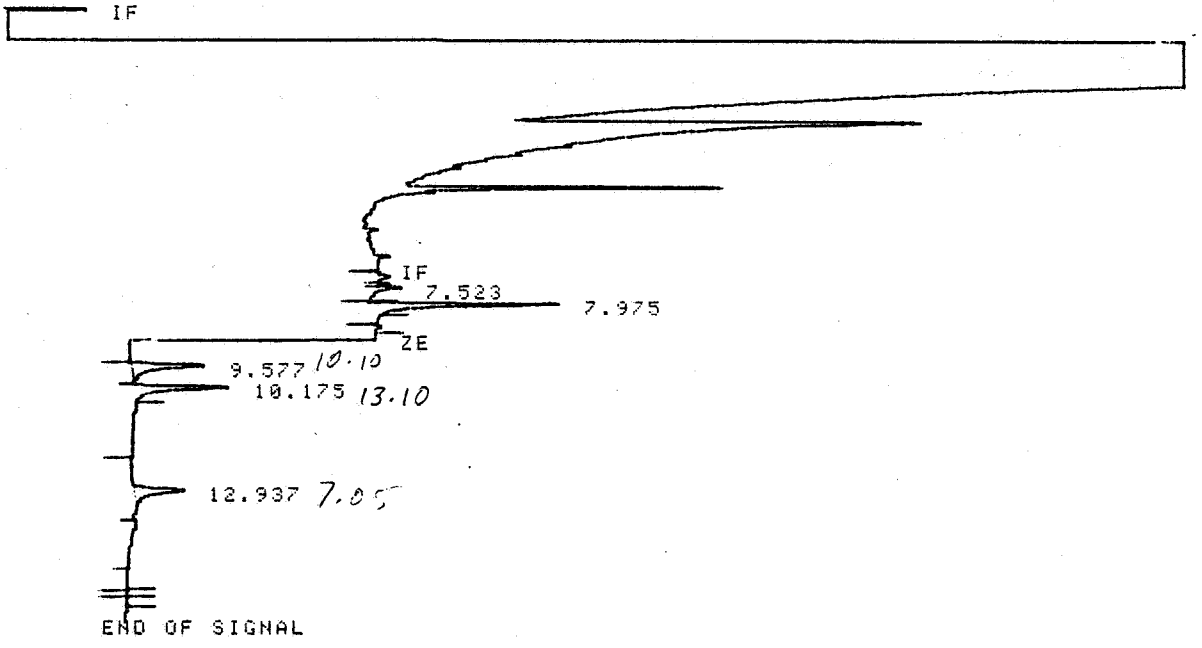
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.577	PP	4011	.138	484	1	.244	DMS
10.175	PB	4853	.127	636	2	.140	SULFENTRAZONE
12.939	BV	3570	.210	283	3	.295	3-HMS

TOTAL AREA= 20727
MUL FACTOR=1.0000E+00

RUN # 1295 JAN 30, 1997 19:27:47
START



RUN# 1295 JAN 30, 1997 19:27:47

SAMPLE NAME: WG.1-1 SAMPLE# 50
10.0223 G OF WHEAT GRAIN IN 100ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 15 ML: SPIKE LEVEL OF 0.1PPM.

2UL,DB17,30M,.53mm,1uM,B97-1..4

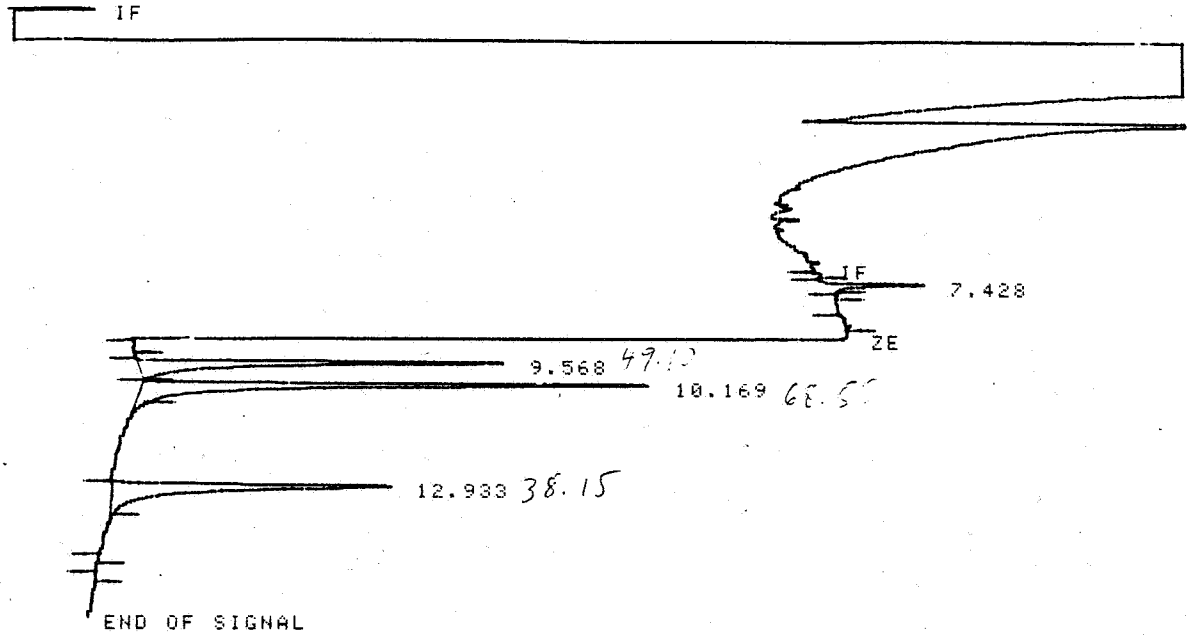
ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.577	PV	4812	.147	546	1	.293	DMS
10.175	VB	5436	.130	698	2	.157	SULFENTRAZONE
12.937	PV	5858	.254	385	3	.484	3-HMS

DATE: FEB 4, 1997 11:18:50

PROCESSING!

RUN # 1274 JAN 30, 1997 12:56:52
START



RUN# 1274 JAN 30, 1997 12:56:52

SAMPLE NAME: STD SAMPLE# 40
0.10097 UG/ML OF SULFENTRAZONE, 0.10041 UG/ML OF 3-HMS & 0.10046 UG/ML OF DMS.

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.568	BB	22001	.139	2646	1	1.338	DMS
10.169	PB	30733	.141	3631	2	.885	SULFENTRAZONE
12.933	BB	25663	.212	2015	3	2.121	3-HMS

TOTAL AREA= 81677
MUL FACTOR=1.0000E+00

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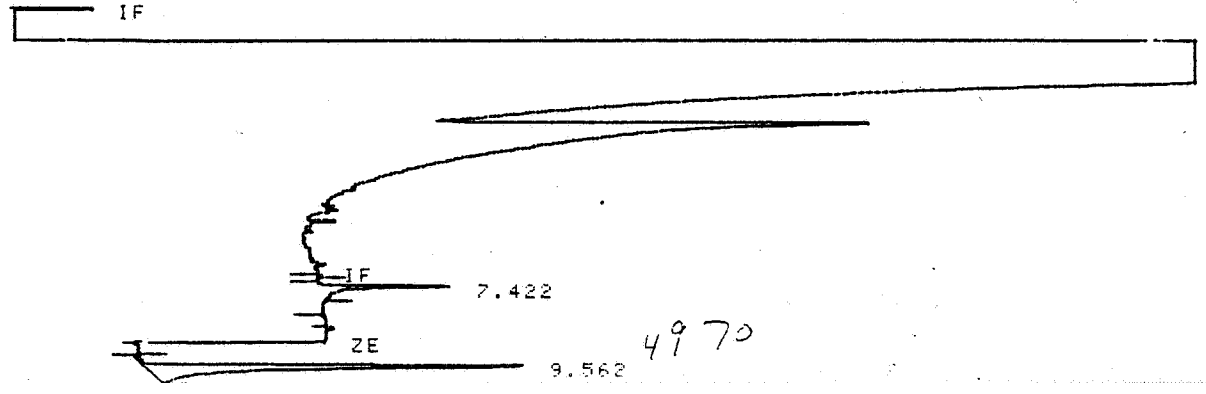

12

2UL,DB17,30M,.53mm,1uM,B97-1..4

NO CALIB PEAKS FOUND
AREA%

RT	AREA	TYPE	WIDTH	AREA%
7.926	2404	PB	.098	100.00000

TOTAL AREA= 2404
MUL FACTOR=1.0000E+00

RUN # 1279 JAN 30, 1997 14:29:48
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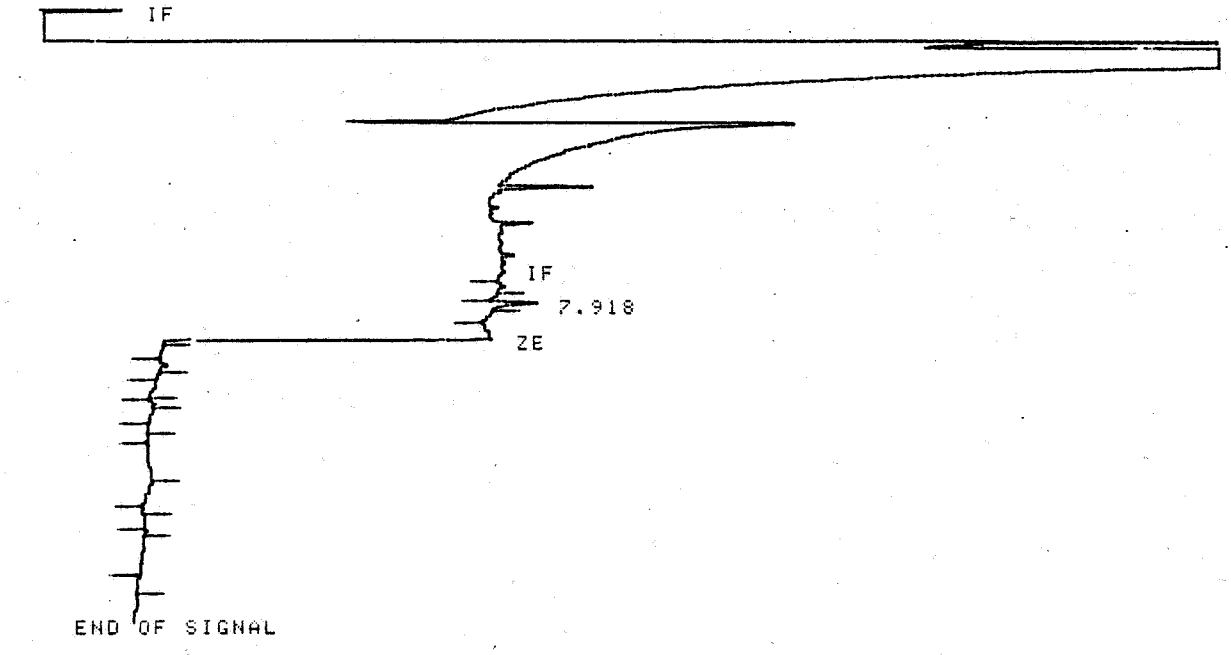

RUN# 1279 JAN 30, 1997 14:29:48

SAMPLE NAME: WG-C-2 SAMPLE# 42
10.0153 G OF WHEAT GRAIN IN 100 ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 3 ML; NO SP
IKE .

2UL,DB17,30M,.53mm,1uM,B97-1..4

NO CALIB PEAKS FOUND
AREA%

RT	AREA	TYPE	WIDTH	AREA%
7.918	1737	BB	.085	100.00000

TOTAL AREA= 1737
MUL FACTOR=1.0000E+00

RUN # 1280 JAN 30, 1997 14:48:22

TOTAL AREA= 19664
MUL FACTOR=1.0000E+00

RUN # 1284 JAN 30, 1997 16:02:35
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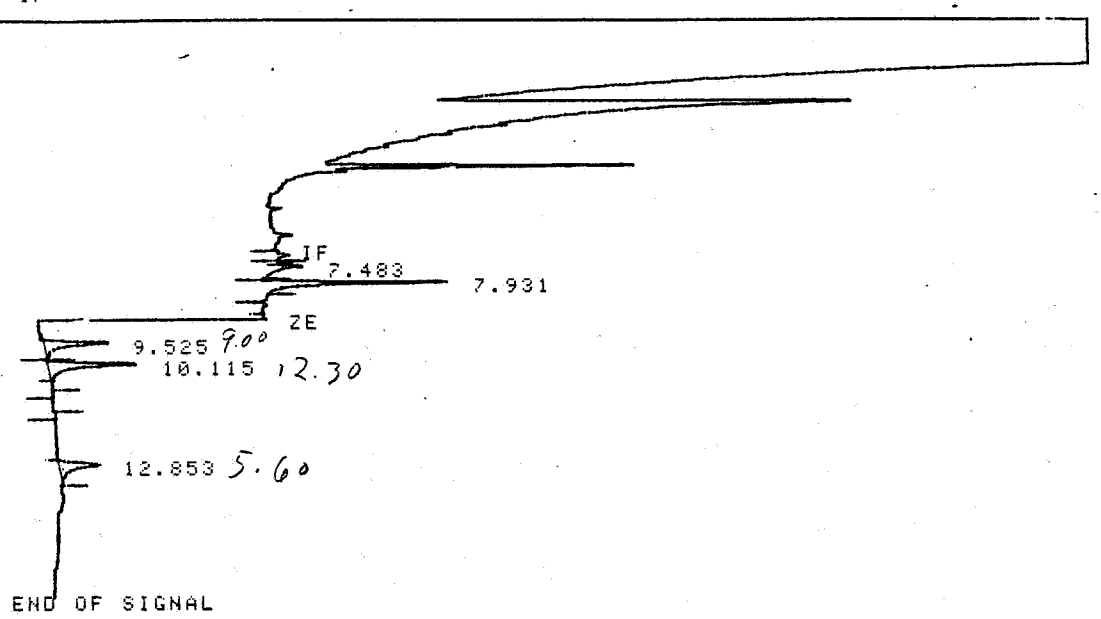

RUN# 1284 JAN 30, 1997 16:02:35

SAMPLE NAME: WG.025-2 SAMPLE# 45
10.0127 G OF WHEAT GRAIN IN 100 ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 3 ML.; SPIK
E LEVEL OF 0.025PPM

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.525	PB	3585	.128	468	1	.218	DMS
10.115	BV	5266	.137	640	2	.152	SULFENTRAZONE
12.853	PB	3186	.190	280	3	.263	3-HMS

TOTAL AREA= 20673
MUL FACTOR=1.0000E+00

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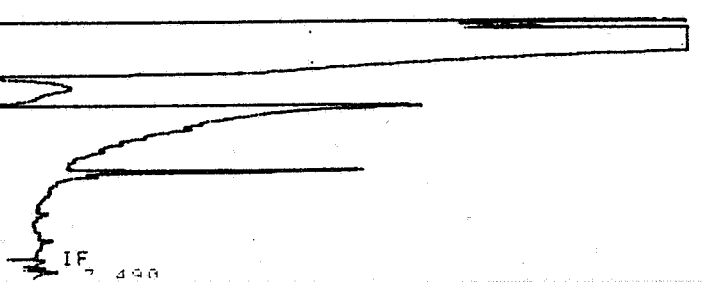
START IF



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ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.571	PP	3915	.139	469	1	.238	DMS
10.169	PB	4563	.128	595	2	.131	SULFENTRAZONE
12.926	BP	3443	.193	297	3	.285	3-HMS

TOTAL AREA= 19884
MUL FACTOR=1.0000E+00

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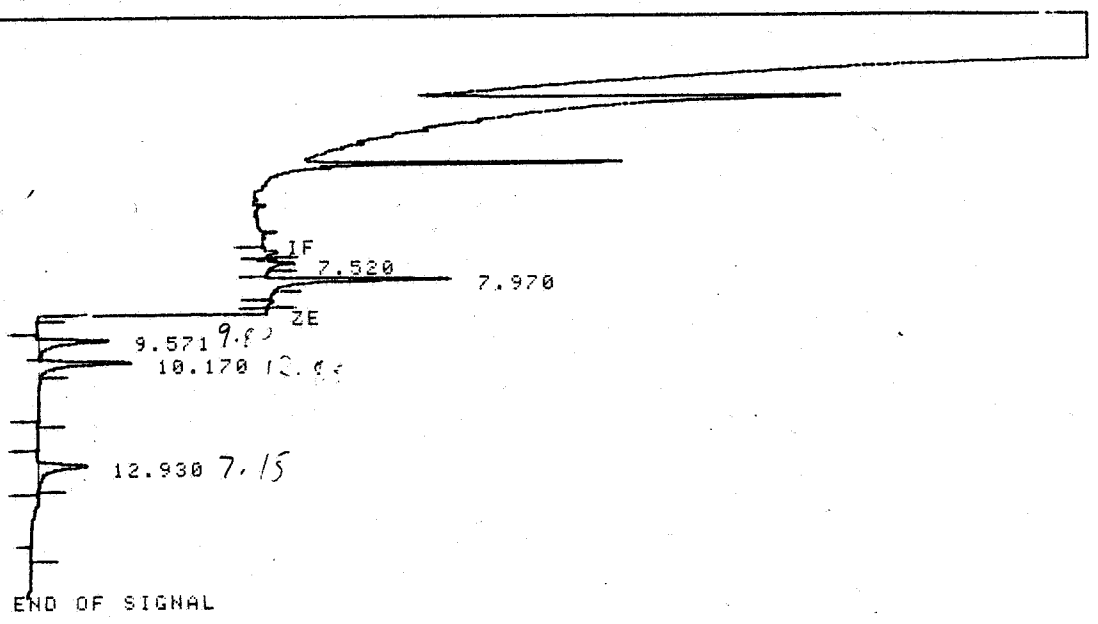
START IF



RUN# 1291 JAN 30, 1997 18:13:33

SAMPLE NAME: WG.05-2 SAMPLE# 48
10.0126 G OF WHEAT GRAIN IN 100ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 5 ML; SPIKE LEVEL OF 0.05PPM.

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

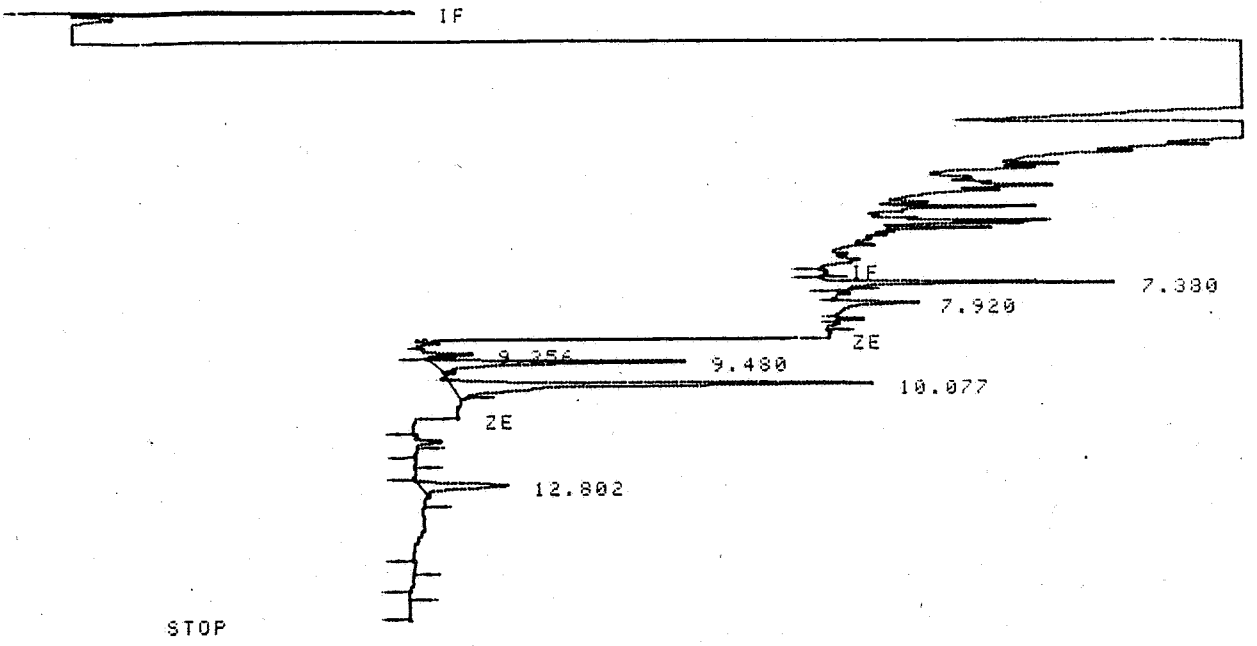
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.571	BV	4526	.145	520	1	.275	DMS
10.170	VB	5222	.130	667	2	.150	SULFENTRAZONE
12.930	BB	4302	.200	359	3	.356	3-HMS

TOTAL AREA= 22477
MUL FACTOR=1.0000E+00

15

RUN # 1292 JAN 30, 1997 18:32:07
START
IF

RUN # 1257 JAN 29, 1997 22:38:23
START



Closing signal file B:Q3FB045F.BNC

RUN# 1257 JAN 29, 1997 22:38:23

SAMPLE NAME: WS .6-1 SAMPLE# 38
10.0136 G OF WHEAT STRAW IN 100ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 25 ML; SPIKE LEVEL OF 0.6PPM

SIGNAL FILE: B:Q3FB045F.BNC

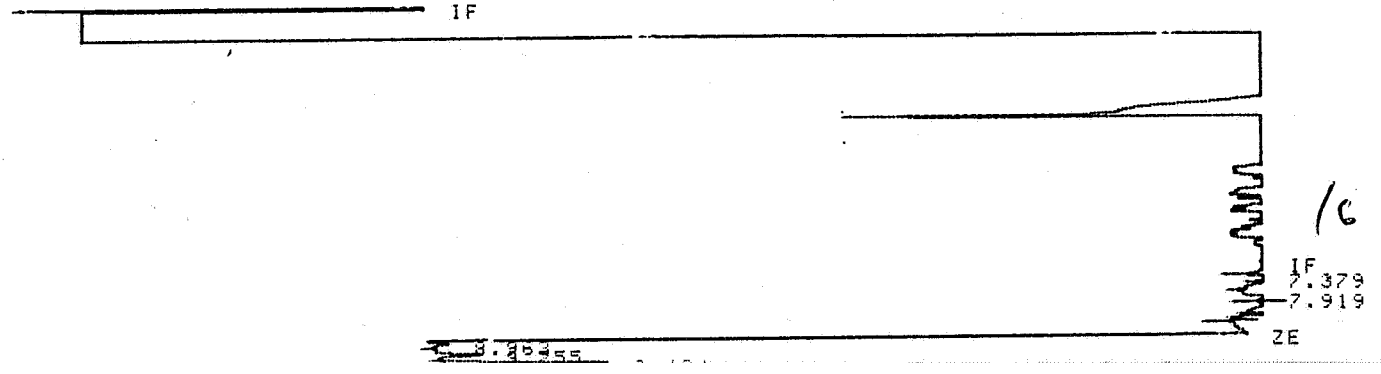
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.480	PV	5765	.104	925	1	.351	DMS
10.077	PB	11101	.121	1531	2	.320	SULFENTRAZONE
12.802	PB	3538	.179	330	3	.292	3-HMS

TOTAL AREA= 27144
MUL FACTOR=1.0000E+00

RUN # 1258 JAN 29, 1997 22:56:56
START



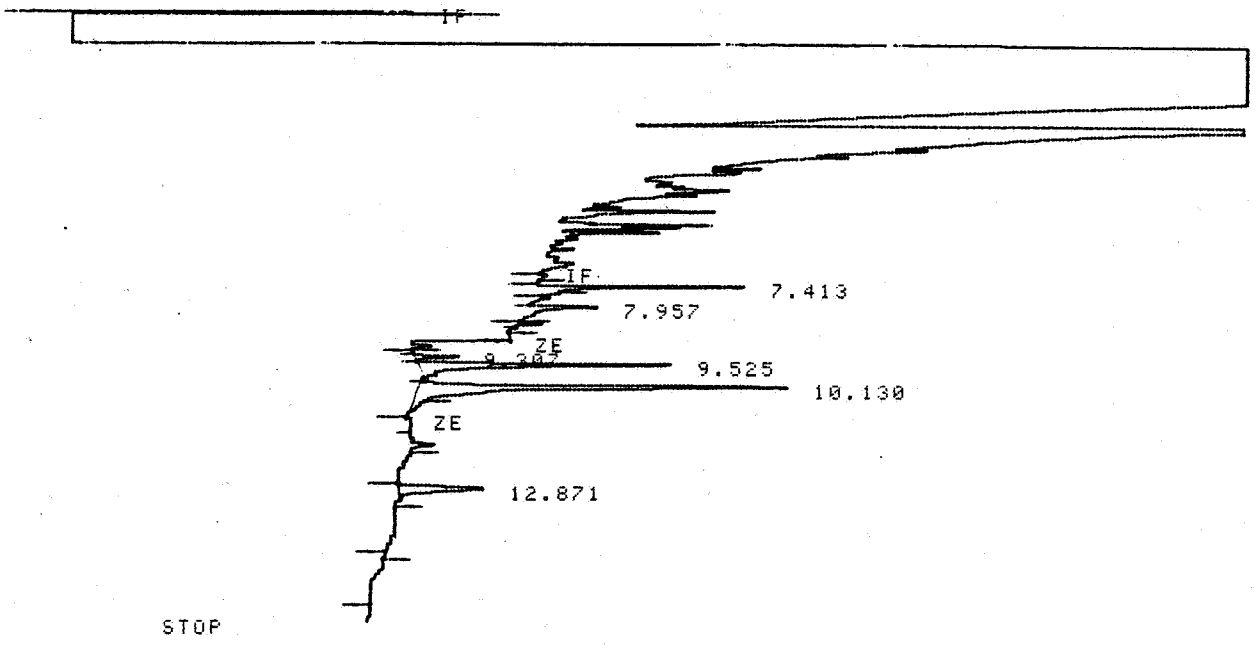
/c

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.561	BB	12211	.119	1715	1	.743	DMS
10.160	PB	19254	.123	2607	2	.554	SULFENTRAZONE
12.940	PV	5828	.182	533	3	.482	3-HMS

TOTAL AREA= 39792
MUL FACTOR=1.0000E+00

RUN # 1251 JAN 29, 1997 20:46:01
START



Closing signal file B:03FAEA0A.BNC

RUN# 1251 JAN 29, 1997 20:46:01

SAMPLE NAME: WS.3-1 SAMPLE# 35
10.0259 G OF WHEAT STRAW IN 100 ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 10 ML; SPIKE LEVEL OF 0.3PPM

SIGNAL FILE: B:03FAEA0A.BNC

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.525	VV	6594	.118	933	1	.401	DMS
10.130	VB	9962	.125	1326	2	.287	SULFENTRAZONE
12.871	PB	3102	.164	315	3	.256	3-HMS

TOTAL AREA= 24539
MUL FACTOR=1.0000E+00

17

RUN # 1252 JAN 29, 1997 21:04:36
START

RUN# 1244   JAN 29, 1997 18:36:07

SAMPLE NAME: STD   SAMPLE#   31
0.10097 UG/ML OF SULFENTRAZONE, 0.10041 UG/ML OF 3-HMS & 0.10046 UG/ML OF DMS

SIGNAL FILE: B:Q3FACB98.BNC

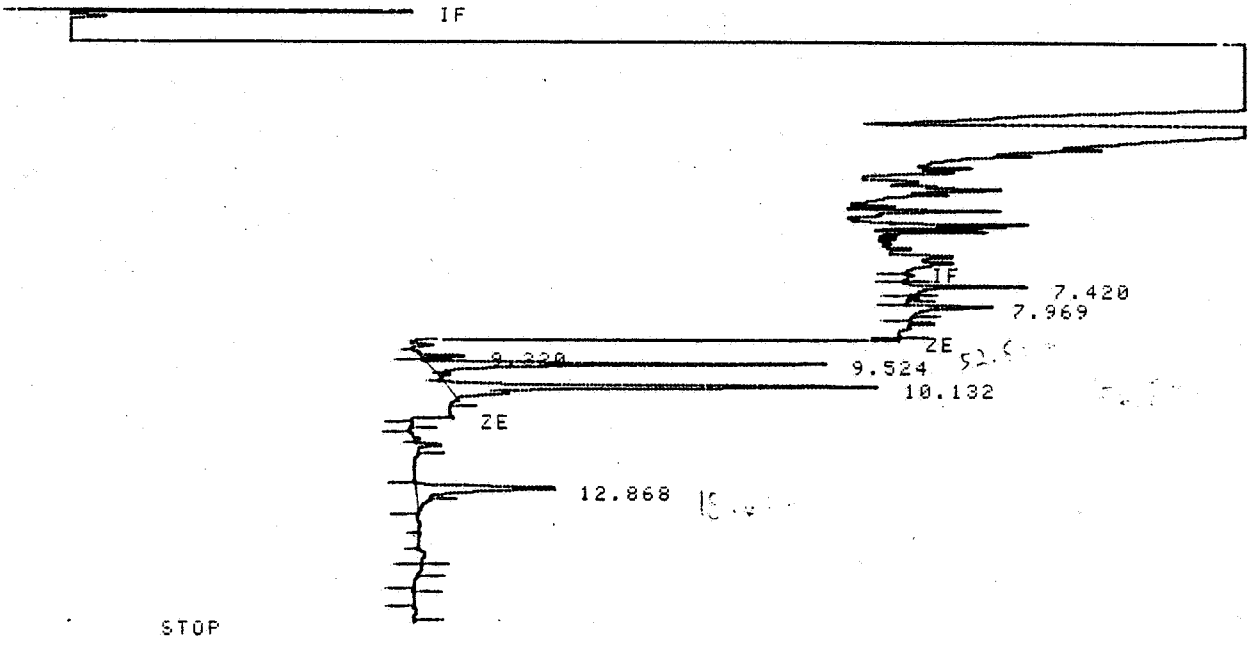
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.534	PB	21859	.092	3966	1	1.329	DMS
10.142	PB	22715	.096	3947	2	.654	SULFENTRAZONE
12.888	BB	11449	.142	1343	3	.946	3-HMS

TOTAL AREA= 56023
MUL FACTOR=1.0000E+00

RUN # 1245   JAN 29, 1997 18:54:41
START



Closing signal file B:Q3FACFF2.BNC

RUN# 1245   JAN 29, 1997 18:54:41

SAMPLE NAME: WS.05-1   SAMPLE#   32
10.0053 G OF WHEAT STRAW IN 100 ML, TOOK ALIQ. OF 50 ML, FINAL VOL.= 5 ML; SPIKE
 LEVEL OF 0.05PPM

SIGNAL FILE: B:Q3FACFF2.BNC

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

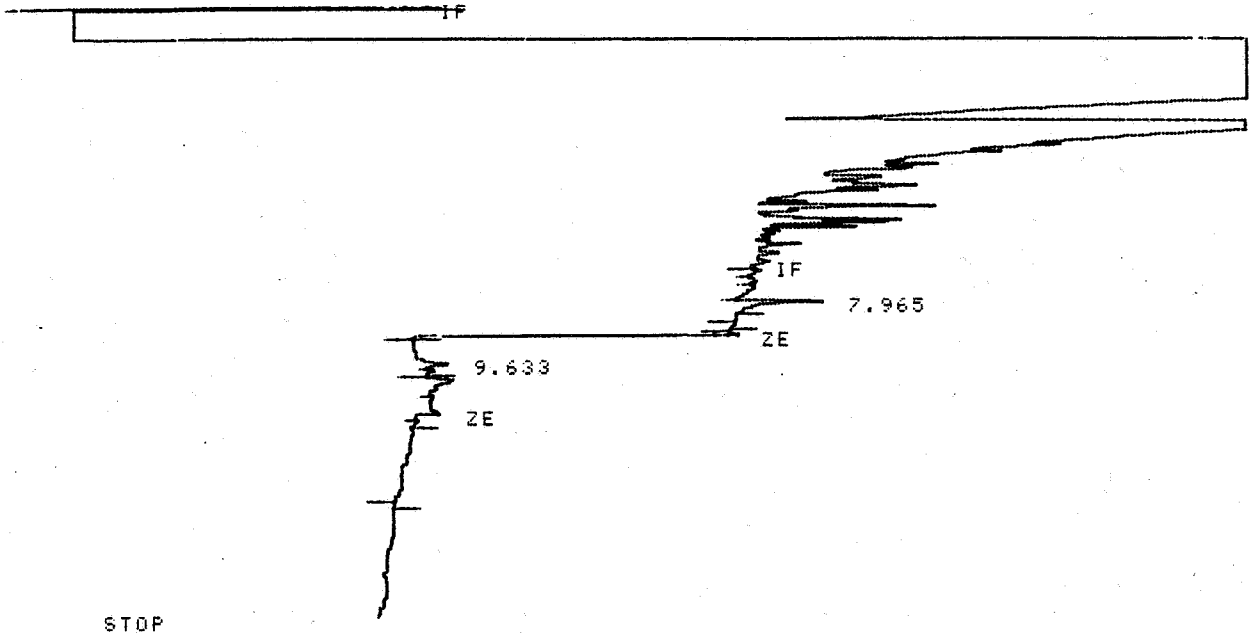
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.524	PV	8070	.093	1443	1	.491	DMS
10.132	VV	9756	.103	1580	2	.281	SULFENTRAZONE
12.868	PB	4257	.146	485	3	.352	3-HMS

TOTAL AREA= 22021

12.900	BV	6320	.209	504	3	.522	3-HMS

TOTAL AREA= 36728
MUL FACTOR=1.0000E+00

RUN # 1239 JAN 29, 1997 17:02:38
START



STOP

Closing signal file B:03FAB5AF.BNC

RUN# 1239 JAN 29, 1997 17:02:38

SAMPLE NAME: WS-C-1 SAMPLE# 29
10.0101 G OF WHEAT STRAW IN 100 ML, TOOK ALIQ. OF 50ML, FINAL VOL. = 5ML; NO SPI
KE

SIGNAL FILE: B:03FAB5AF.BNC

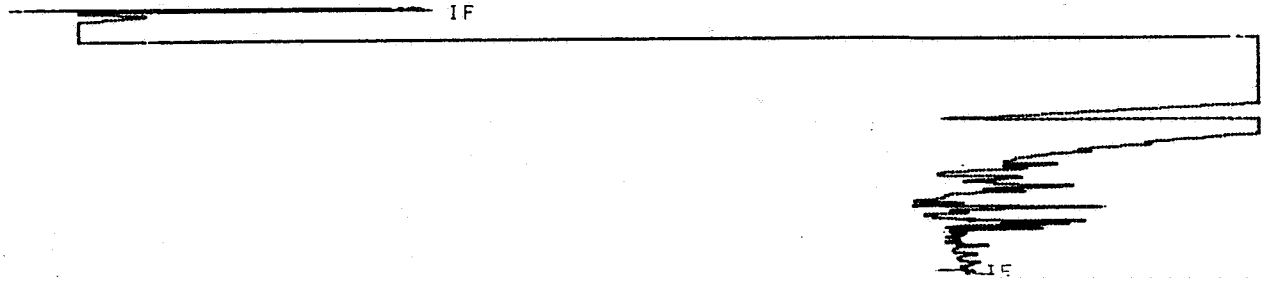
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.633	FV	326	.057	95	1	.020	DMS

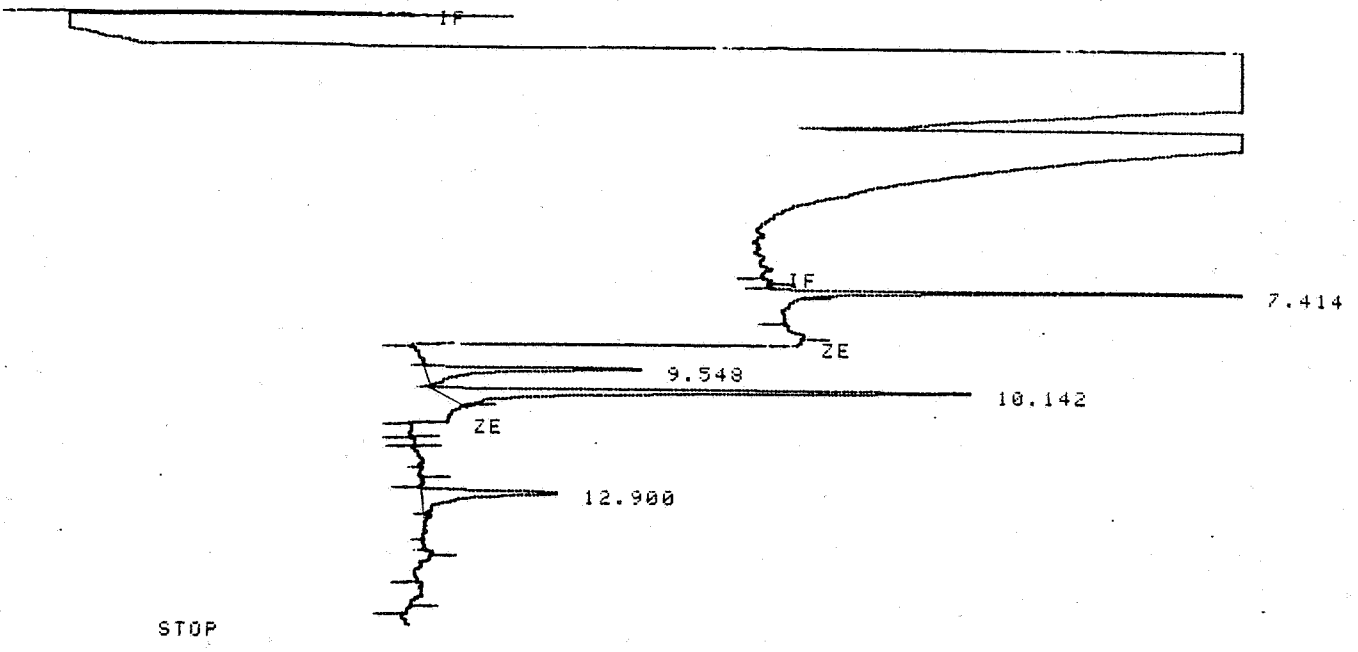
TOTAL AREA= 2249
MUL FACTOR=1.0000E+00

RUN # 1240 JAN 29, 1997 17:21:11
START



TOTAL AREA= 64560
MUL FACTOR=1.0000E+00

RUN # 1238 JAN 29, 1997 16:44:05
START



Closing signal file B:Q3FAB155.BNC

RUN# 1238 JAN 29, 1997 16:44:05

SAMPLE NAME: STD SAMPLE# 28
0.10097 UG/ML OF SULFENTRAZONE, 0.10041 UG/ML OF 3-HMS, & 0.10046 UG/ML OF DMS.

SIGNAL FILE: B:Q3FAB155.BNC

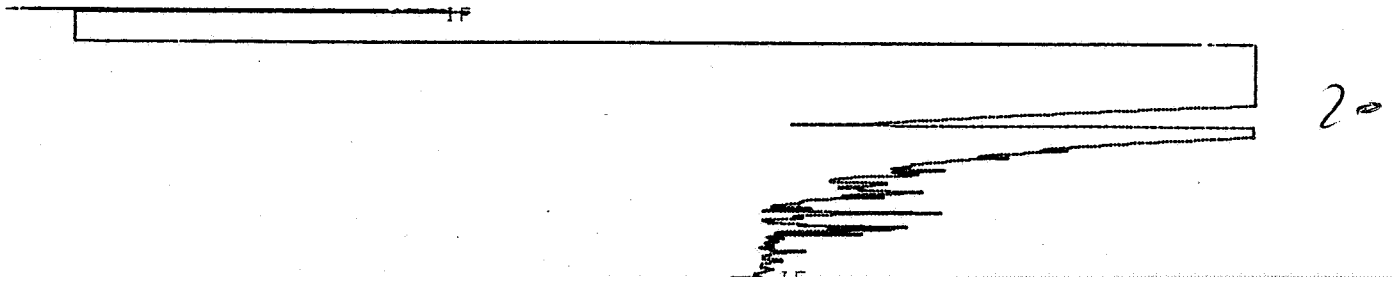
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.548	VP	6710	.140	800	1	.408	DMS
10.142	PB	15419	.134	1923	2	.444	SULFENTRAZONE
12.900	BV	6320	.209	504	3	.522	3-HMS

TOTAL AREA= 36728
MUL FACTOR=1.0000E+00

RUN # 1239 JAN 29, 1997 17:02:38
START

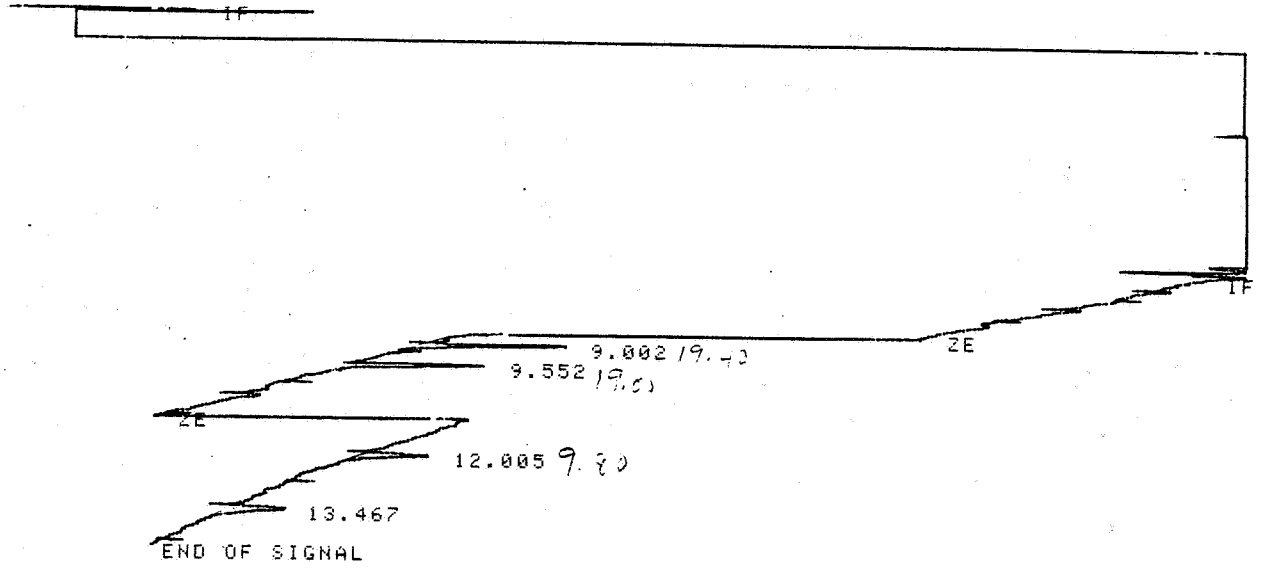


2-

12.004	BB	1585	.105	252	3	.131	3-HMS

TOTAL AREA= 9714
MUL FACTOR=1.0000E+00

RUN # 1210 JAN 27, 1997 23:12:58
START



RUN# 1210 JAN 27, 1997 23:12:58

SAMPLE NAME: WF.2-2 SAMPLE# 27
10G OF WHEAT FORAGE IN 100ML, TOOK ALIQ. OF 50ML, FINAL VOL.= 10 ML.; SPIKE LEVE
L OF 0.2 PPM.

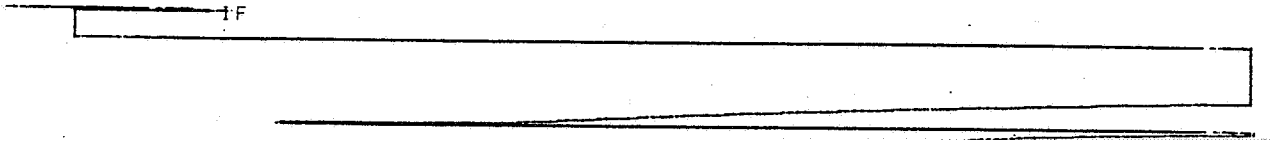
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

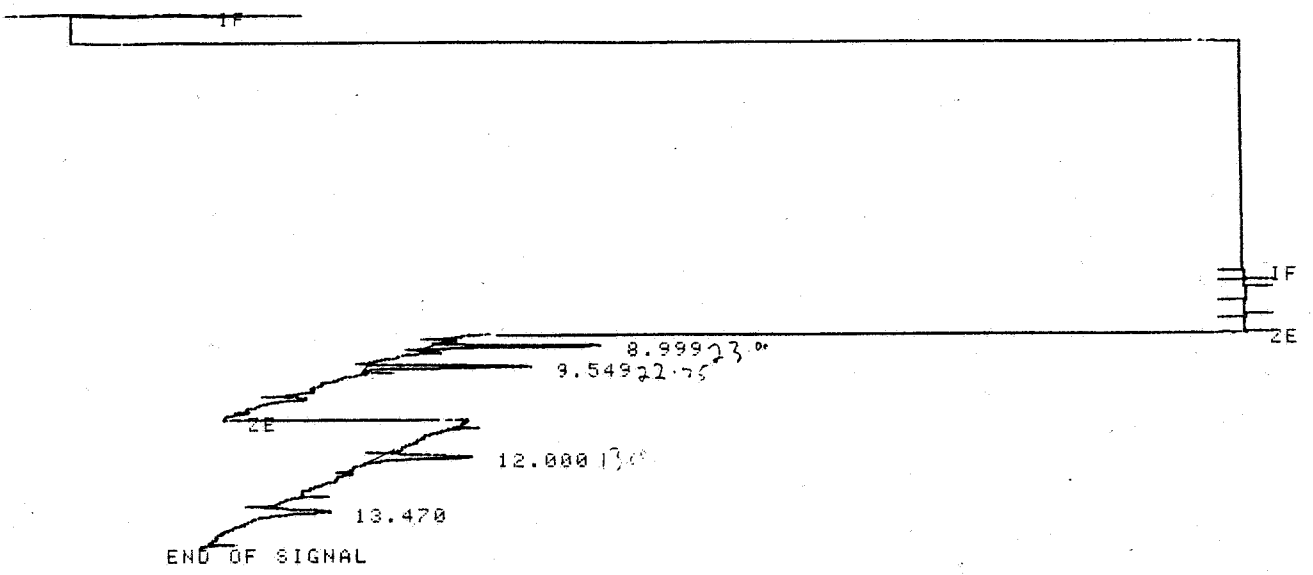
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.002	BV	2039	.066	513	1	.124	DMS
9.552	PB	2776	.091	506	2	.080	SULFENTRAZONE
12.005	BB	1491	.100	248	3	.123	3-HMS

TOTAL AREA= 8697
MUL FACTOR=1.0000E+00

RUN # 1211 JAN 27, 1997 23:29:31
START



RUN # 1201 JAN 27, 1997 20:43:43



RUN# 1201 JAN 27, 1997 20:43:43

SAMPLE NAME: WF.1-1 SAMPLE# 23
10G OF WHEAT FORAGE IN 100 ML,TOOK ALIQ. OF 50ML, FINAL VOL.= 5ML. SPIKE LEVEL O
F .1PPM

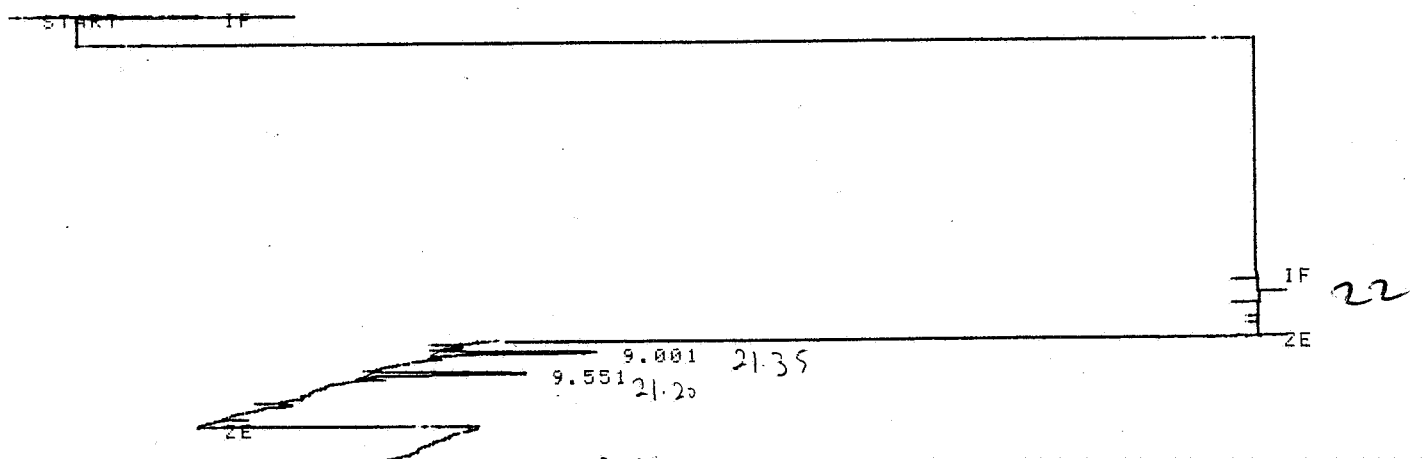
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
8.999	VV	2734	.071	643	1	.166	DMS
9.549	PB	2519	.071	591	2	.073	SULFENTRAZONE
12.000	BP	2156	.112	322	3	.178	3-HMS

TOTAL AREA= 9123
MUL FACTOR=1.0000E+00

RUN # 1202 JAN 27, 1997 21:00:17

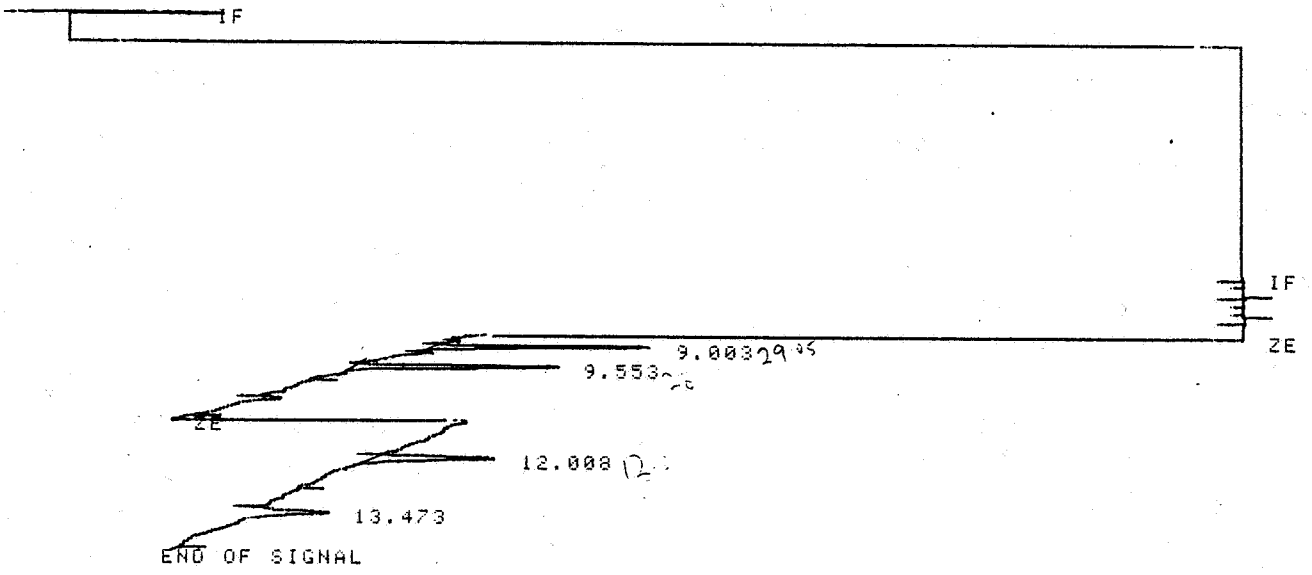


ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.020	PB	10167	.078	2171	1	.618	DMS
9.569	PB	12065	.087	2313	2	.347	SULFENTRAZONE
12.035	BP	7162	.142	839	3	.592	3-HMS

TOTAL AREA= 29394
MUL FACTOR=1.0000E+00

RUN # 1195 JAN 27, 1997 19:04:08
START



RUN# 1195 JAN 27, 1997 19:04:08

SAMPLE NAME: WF-.0251 SAMPLE# 20
3UL-INJ; 10G OF WHEAT FORAGE IN 100ML, TOOK ALIQ. OF 50ML,FINAL VOL.= 2 ML; SPIK
E LEVEL OF 0.025PPM

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.003	PV	3443	.070	814	1	.209	DMS
9.553	PB	3889	.086	751	2	.112	SULFENTRAZONE
12.008	BB	2528	.096	437	3	.209	3-HMS

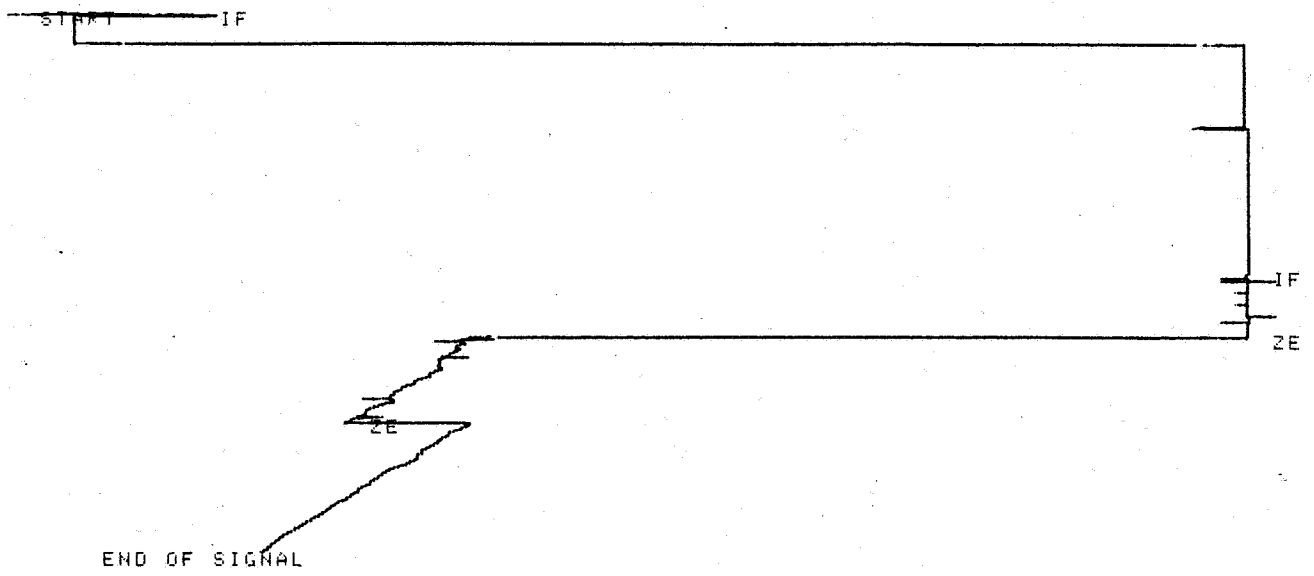
TOTAL AREA= 12548
MUL FACTOR=1.0000E+00

RUN # 1196 JAN 27, 1997 19:20:44
START

R.T.	TP	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.018	PB	6556	.073	1487	1	.399	DMS
9.568	PB	6940	.079	1473	2	.200	SULFENTRAZONE
12.036	PB	3332	.135	410	3	.275	3-HMS

TOTAL AREA= 19525
MUL FACTOR=1.0000E+00

RUN # 1189 JAN 27, 1997 17:24:01



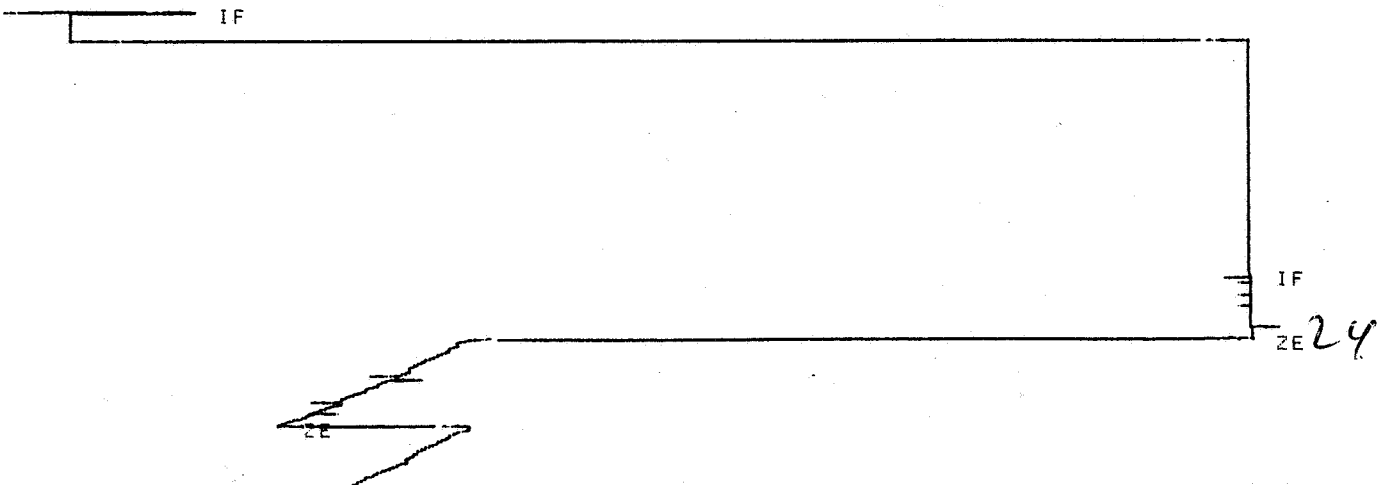
RUN# 1189 JAN 27, 1997 17:24:01

SAMPLE NAME: WF C-1 SAMPLE# 17
10G OF WHEAT FORAGE IN 100 ML, TOOK ALIQ. OF 50 ML, FINAL VOL,= 2 ML. NO SPIKE

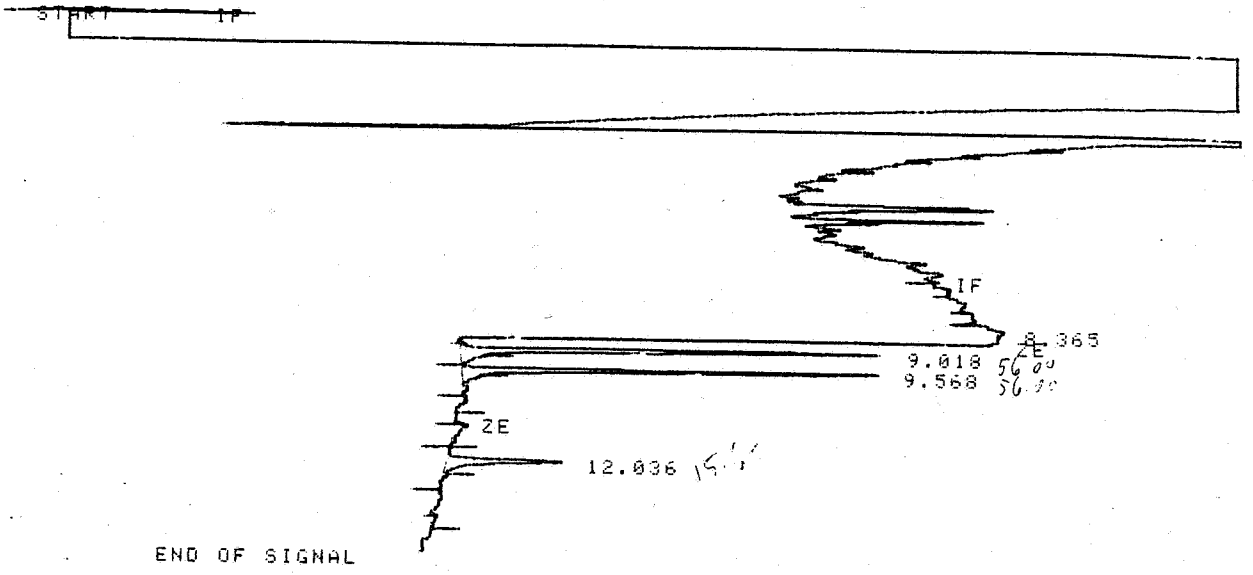
2UL,DB17,30M,.53mm,1uM,B97-1..4

NO RUN PEAKS STORED

RUN # 1190 JAN 27, 1997 17:40:37
START



RUN # 1188 JAN 27, 1997 17:07:25



RUN# 1188 JAN 27, 1997 17:07:25

SAMPLE NAME: STD SAMPLE# 16
0.1UG/ML OF SULFENTRAZONE, DMS, 3-HMS IN ACETONITRILE

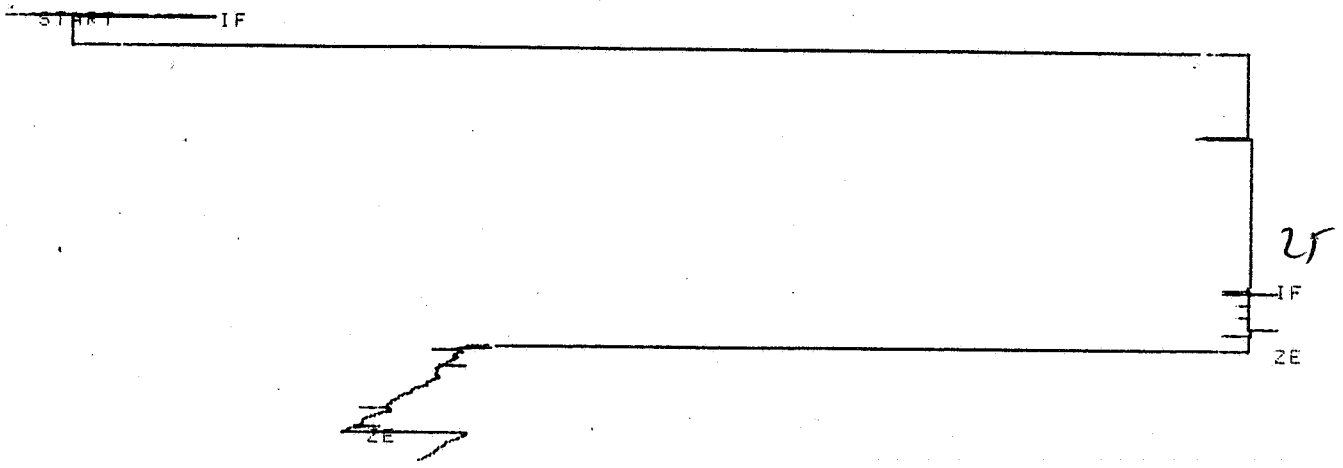
2UL,DB17,30M,.53mm,1uM,897-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.018	PB	6556	.073	1487	1	.399	DMS
9.568	PB	6940	.079	1473	2	.200	SULFENTRAZONE
12.036	PB	3332	.135	410	3	.275	3-HMS

TOTAL AREA= 19525
MUL FACTOR=1.0000E+00

RUN # 1189 JAN 27, 1997 17:24:01



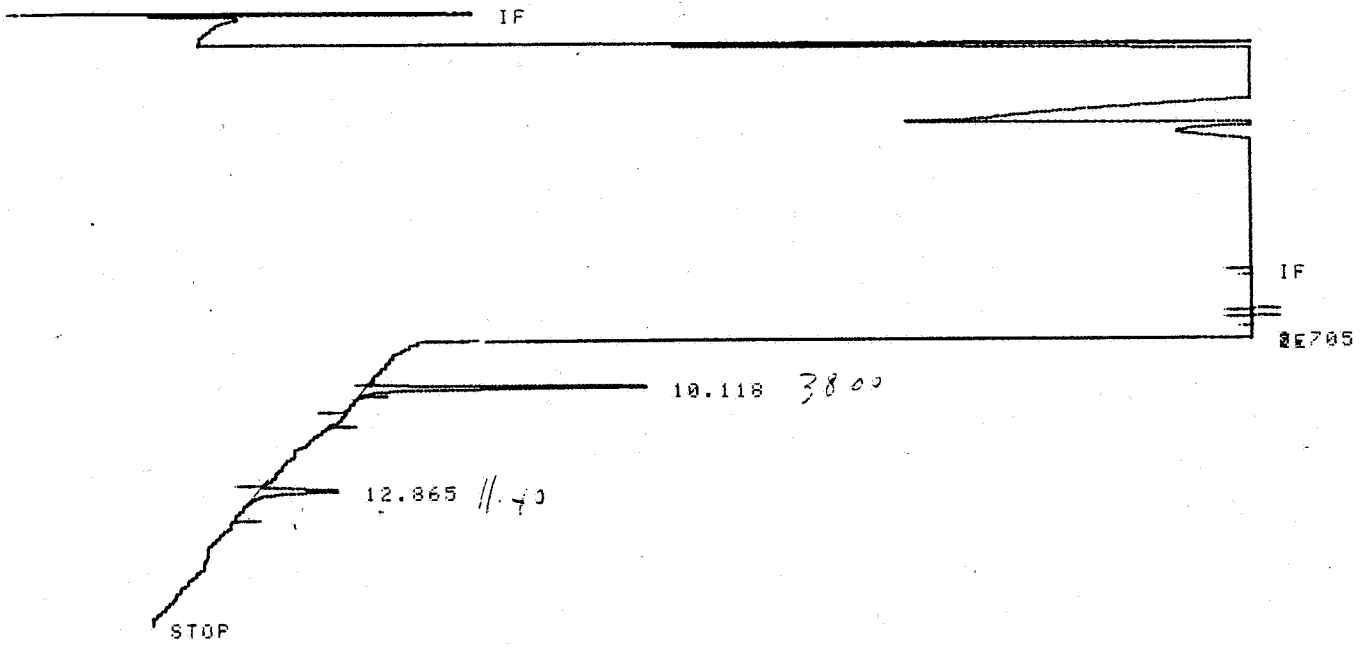
25

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
10.102	BB	27377	.103	4428	2	.788	SULFENTRAZONE
12.840	BB	12943	.154	1398	3	1.070	3-HMS

TOTAL AREA= 40320
MUL FACTOR=1.0000E+00

RUN # 1326 FEB 3, 1997 21:04:52
START



Closing signal file B:040185F5.BNC

RUN# 1326 FEB 3, 1997 21:04:52

SAMPLE NAME: SS.05-1 SAMPLE# 13
10.0071 G OF SOYBEAN SEED IN 100ML, TOOK ALIQ. OF 50ML, FINAL VOL.= 4 ML; SPIKE LEVEL OF 0.05 PPM.

SIGNAL FILE: B:040185F5.BNC

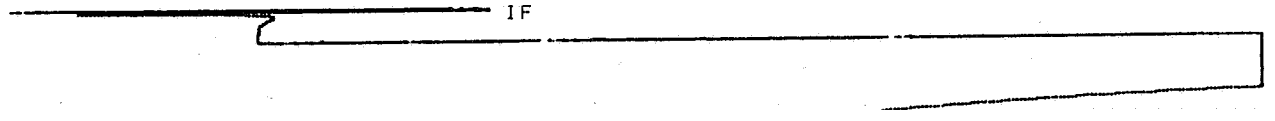
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
10.118	VB	23093	.093	4143	2	.665	SULFENTRAZONE
12.865	BB	9338	.134	1158	3	.772	3-HMS

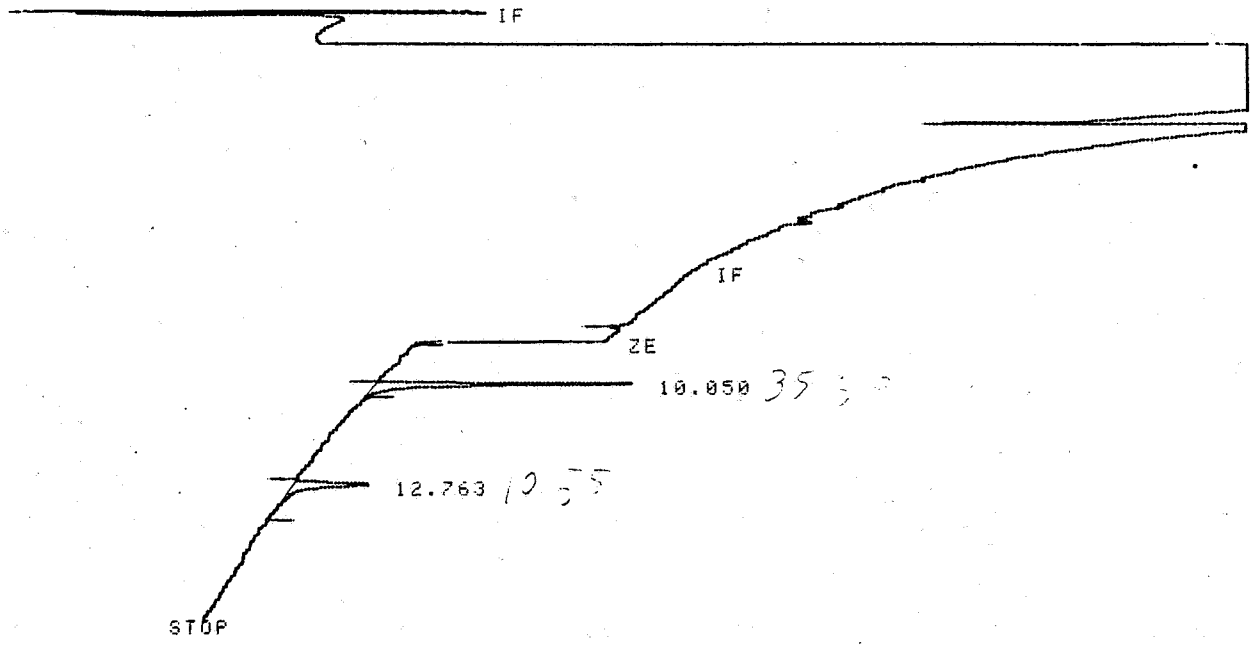
TOTAL AREA= 74325
MUL FACTOR=1.0000E+00

RUN # 1327 FEB 3, 1997 21:23:27
START



TOTAL AREA= 84943
MUL FACTOR=1.0000E+00

RUN # 1320 FEB 3, 1997 19:13:10
START



Closing signal file B:Q4016BC7.BNC

RUN# 1320 FEB 3, 1997 19:13:10

SAMPLE NAME: SS.025-1 SAMPLE# 10
10.0202 G OF SOYBEAN IN 100ML, TOOK ALIQ. OF 50ML, FINAL VOL.= 2ML;SPIKE LEVEL O
F 0.025 PPM.

SIGNAL FILE: B:Q4016BC7.BNC

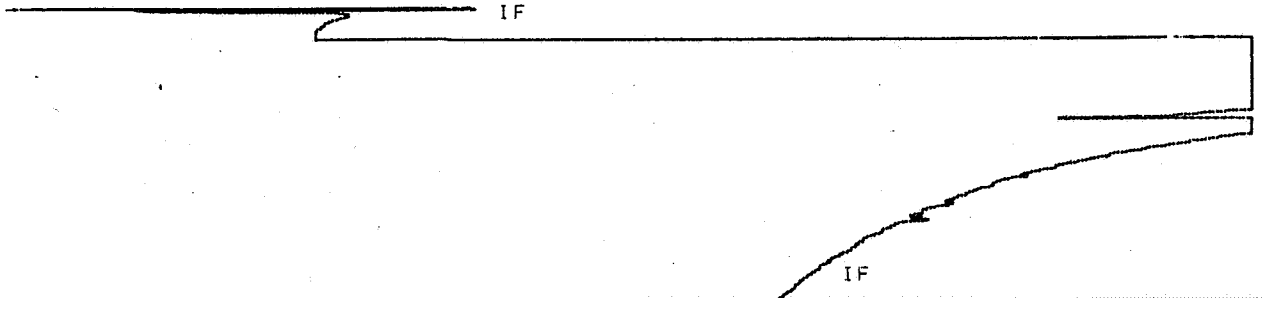
2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
10.050	BB	22869	.101	3760	2	.658	SULFENTRAZONE
12.763	BB	11639	.171	1132	3	.962	3-HMS

TOTAL AREA= 34508
MUL FACTOR=1.0000E+00

RUN # 1321 FEB 3, 1997 19:31:48
START

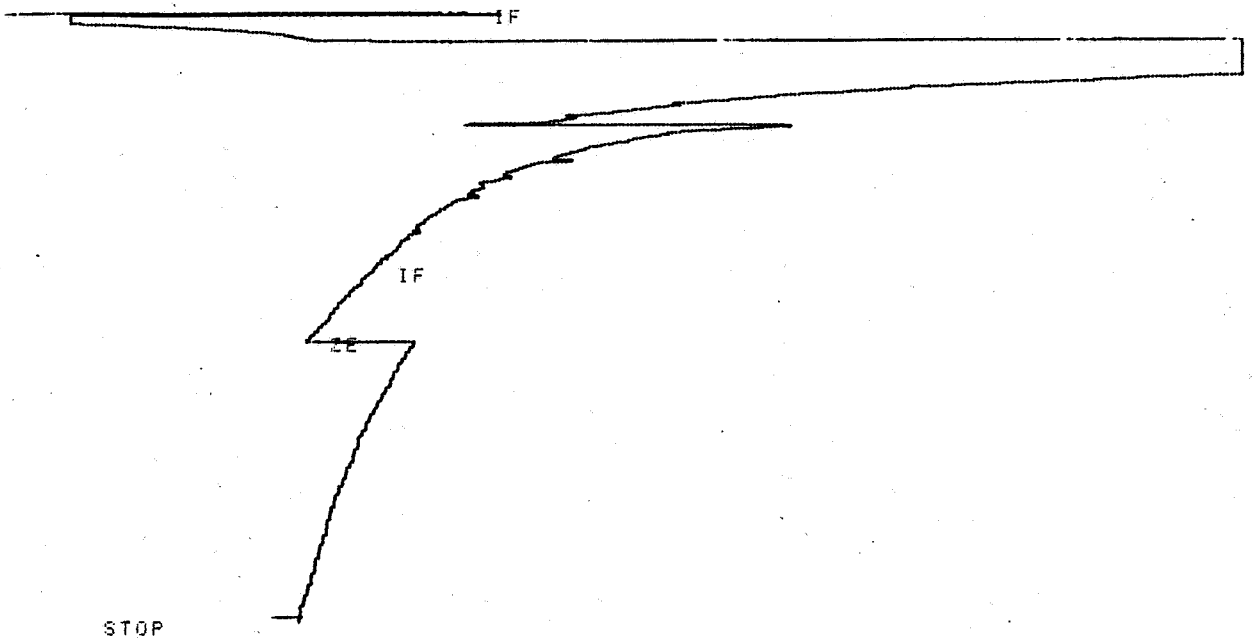


ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.815	VB	8140	.217	624	2	.234	SULFENTRAZONE

TOTAL AREA= 65408
MUL FACTOR=1.0000E+00

RUN # 1315 FEB 3, 1997 17:39:32
START



Closing signal file B:Q40155D4.BNC

RUN# 1315 FEB 3, 1997 17:39:32

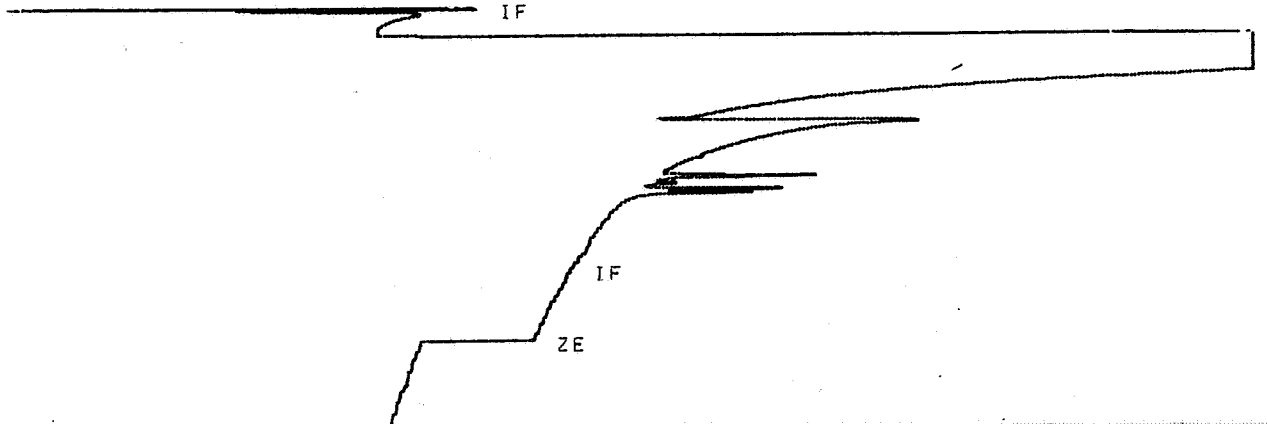
SAMPLE NAME: SS-C-1 SAMPLE# 7
10.0160 G OF SOYBEAN SEED IN 100 ML,TOOK ALIQ. OF 50ML, FINAL VOL.= 2ML; NO SPIK
E

SIGNAL FILE: B:Q40155D4.BNC

2UL,DB17,30M,.53mm,1uM,B97-1..4

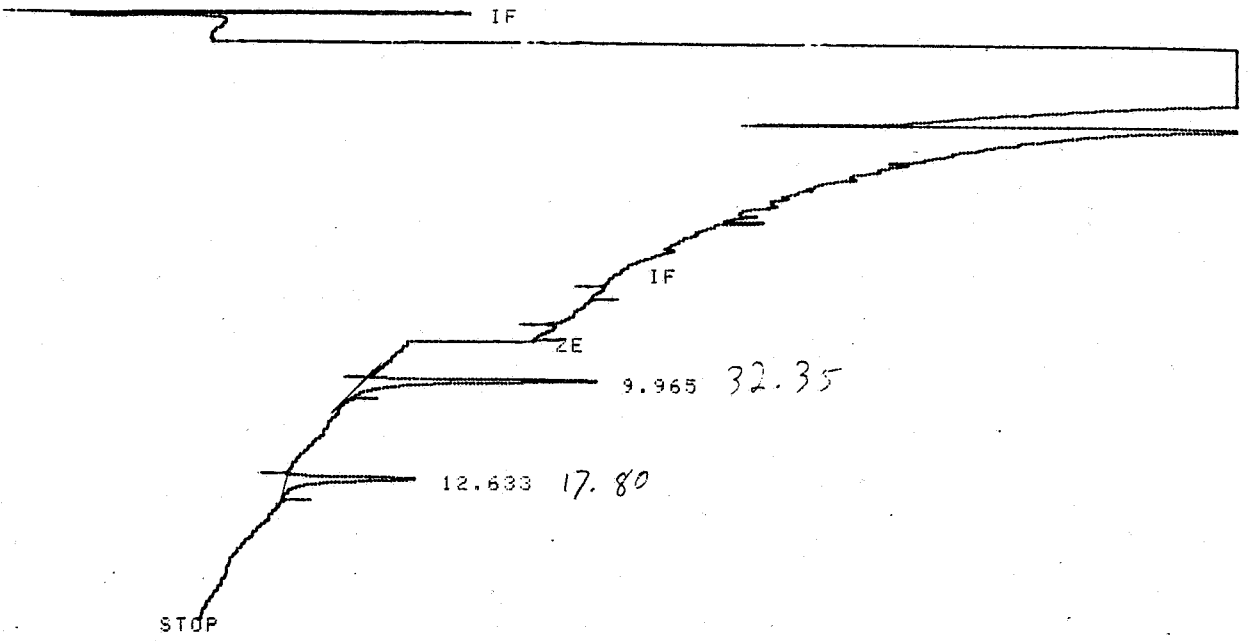
NO RUN PEAKS STORED

RUN # 1316 FEB 3, 1997 17:58:08
START



Device [8*1: BREAK

* SEQ START
Waiting for system readiness
RUN # 1312 FEB 3, 1997 16:43:37
START



Closing signal file B:Q40148BA.BNC

RUN# 1312 FEB 3, 1997 16:43:37

SAMPLE NAME: STD SAMPLE# 6
.10097 UG/ML OF SULFENTRAZONE & 0.10041 UG/ML OF 3-HMS

SIGNAL FILE: B:Q40148BA.BNC

2UL,DB17,30M,.53mm,1uM,B97-1..4

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
9.965	BB	25249	.126	3347	2	.727	SULFENTRAZONE
12.633	BB	17755	.157	1881	3	1.468	3-HMS

TOTAL AREA= 43004
MUL FACTOR=1.0000E+00

RUN # 1313 FEB 3, 1997 17:02:16
START