

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Ethion

MRID #: 421137-01

Matrix: Soil

Analysis: GC/NPD

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If you have difficulties in downloading the method, or further questions concerning the methods, you may contact Elizabeth Flynt at 228-688-2410 or via e-mail at flynt.elizabeth@epa.gov.

10-11-55

MEMORANDUM FOR THE DIRECTOR

TO: SAC, NEW YORK

FROM: SAC, CHICAGO

SUBJECT: [Illegible]

RE: [Illegible]

[Illegible typed text]

[Illegible typed text]

B. Analytical Method for Ethion and its Oxygen Analogs
in Soil

1. Analytical Procedure:

- a. Weigh about 50.0 grams (\pm 0.1 gram) of soil/sand into a 250 ml polypropylene centrifuge container.
- b. Reference standards are added to check samples (for method recovery determination) using a Hamilton syringe. After samples have been fortified, allow the solvent to evaporate.
- c. Add 100 ml of 9:1 acetone:water (v:v) to each container and seal the container with a screw cap top fitted with an O-ring to prevent leakage.
- d. Place the samples, horizontally, on a reciprocal laboratory shaker and shake for 30 minutes at high speed.
- e. After shaking is completed, centrifuge the samples for 3 minutes at 2000 rpm.
- f. A 10.0 ml aliquot of the solvent extract from each sample is taken and transferred to a 25mm X 150mm screw cap test tube.
- g. The samples are then placed on the robotics system for a liquid/liquid partition (can be done manually).
 1. Each 10 ml sample (all samples are done one at a time) is evaporated under nitrogen to approximately 1 ml.
 2. Add 9.0 ml of aqueous 5% NaCl and 10.0 ml of 3:1 hexane:ethyl acetate (v:v) to the sample.
 3. Vortex to provide a good whirl for 13 seconds followed by a 2-second pause. This cycle is repeated four times.
 4. A 7.0 ml aliquot is taken from the organic layer and transferred to a clean 25mm X 150mm test tube.

5. An additional 7.0 ml of the 3:1 hexane:ethyl acetate solvent is added to the tube containing the salt solution. Repeat steps 3, 4, and 5 twice for a total of three partitions.

6. After partitioning is completed, the 21.0 mls of 3:1 hexane:ethyl acetate is evaporated under nitrogen to approximately 1 ml. (Robotics phase is complete.)

h. The 1 ml sample from the liquid:liquid partition is transferred to a 15 ml graduated centrifuge tube. Rinse the 25mm X 150mm tube two times with approximately 1 ml of ethyl acetate and place in centrifuge tube. Adjust the volume to 10.0 ml with ethyl acetate.

i. The sample is concentrated to approximately 1.0 - 0.5 ml and final volume is adjusted to 1.0 ml using ethyl acetate.

j. Samples are analyzed and quantified using a GC equipped with an N-P detector.

2. Gas Chromatographic System:

Hewlett-Packard (HP) 5890 GC
HP 7673A Autosampler
HP 3396 Integrator

3. GC-NPD Operating Conditions:

Gas Flow Rates: He, carrier, -7.75 ml/min
H₂, -4 ml/min
He, make-up, -30 ml/min
Air, -100 ml/min

Inlet: Splitless injection fitted with a Gooseneck Splitless Sleeve (4mm I.D.); 225°C

Column: 15 m x 0.53 mm, 1.5 µm film thickness, DB-5 capillary column (J & W Scientific).

Oven Temp: 210°C Isothermal

Detector: HP Nitrogen/Phosphorus

Detector Temp: 280°C

Injection Volume: 2 μ l
Run Time: 10 min
Chart Speed: 0.5 cm/min

4. Quantitation

A Hewlett-Packard 3396A Integrator was used to quantitate both ethion and metabolites of ethion by peak area integration and external standard calibration. Prior to analysis, the HP 5890A was calibrated with the proper standards and showed a linear response between ng injected and area counts. An external standard calibration method was entered at the start of analysis of a series of samples. The method was verified by injecting a standard after every two samples. The amount of both ethion and metabolites of ethion in each sample was reported in parts per million (ppm) using appropriate dilution factors calculated from sample aliquot, amount injected, and final volume of samples.

5. Zymate[®] Robotic System (FMC-1)
Capacity - 20 Samples/Run

Zymate II Robot
ZyMark Max-12 Computer System
EasyLab Controller
Printer, H-P ThinkJet[®]
Liquid/Liquid station
Evaporation station for 25 x 150 tubes
Capping/Uncapping station
Vortex station (25 x 150 mm tubes) and liquid
dispenser nozzles
Evaporation station for 15 ml centrifuge tubes
Hand A
Hand B
Power and Event Controller (PEC)
Master Lab Stations (MLS) with 3 syringes
Masterflex[®] pump

6. Reagents

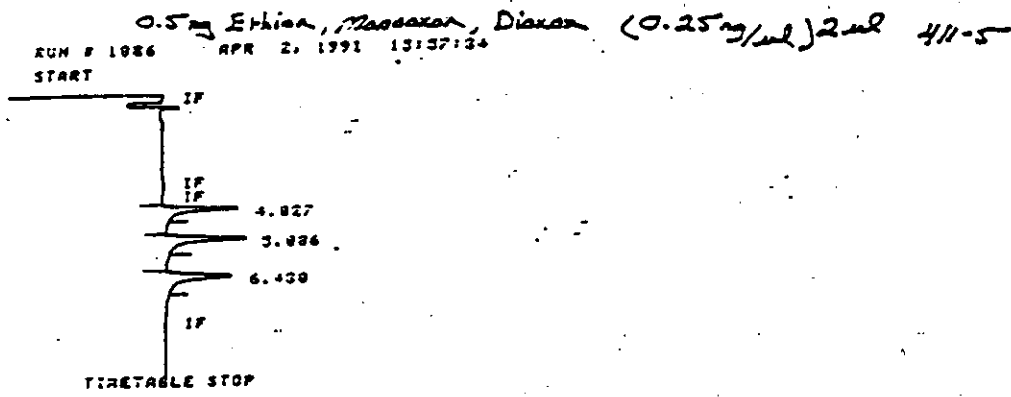
Acetone, Resi-Analyzed, JT Baker
Sodium Chloride, Reagent Grade, JT Baker
Ethyl Acetate, Resi-Analyzed, JT Baker
Hexane, Burdick & Jackson
Hexanes, Resi-Analyzed, JT Baker

C. Chromatograms

FIGURE NUMBER	DESCRIPTION
<u>Pre-treatment</u>	
4	Standard, 0.25 ng/ μ l x 2 μ l
5	Soil Control Trial 01
6	Fortified Soil Control Trial 01
7	Pre-Treated Soil Trial 01
8	Soil Control Trial 02
9	Fortified Soil Control Trial 02
10	Pre-Treated Soil Trial 02
<u>0-Day Interval</u>	
11	Standard, 0.25 ng/ μ l x 2 μ l
12	Soil Control Trial 01
13	Fortified Soil Control Trial 01
14	Treated Soil Trial 01
15	Soil Control Trial 02
16	Fortified Soil Control Trial 02
17	Treated Soil Trial 02
<u>30-Day Interval</u>	
18	Standard, 0.25 ng/ μ l x 2 μ l
19	Soil Control Trial 01
20	Fortified Soil Control Trial 01
21	Treated Soil Trial 01
22	Soil Control Trial 02
23	Fortified Soil Control Trial 02
24	Treated Soil Trial 02
<u>186-Day Interval</u>	
25	Standard, 0.25 ng/ μ l x 2 μ l
26	Soil Control Trial 01
27	Fortified Soil Control Trial 01
28	Treated Soil Trial 01
29	Soil Control Trial 02
30	Fortified Soil Control Trial 02
31	Treated Soil Trial 02

FIGURE 4

ANALYTICAL STANDARD
411-5
2 μ l, 0.25 ng/ μ l



RUN# 1086 APR 2, 1991 13:57:34

SAMPLE# 1

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	4.827	98767	BB	1R	.383
	5.886	128266	PB	2	.499
	6.439	128039	PS	3	.497

TOTAL AREA= 355092
MUL FACTOR=1.9888E+00

194

FIGURE 5
CONTROL SOIL
TRIAL 01, PRE-TREATMENT
PRG-55, #1C001, 0-6"
2 μ l, 10 mg

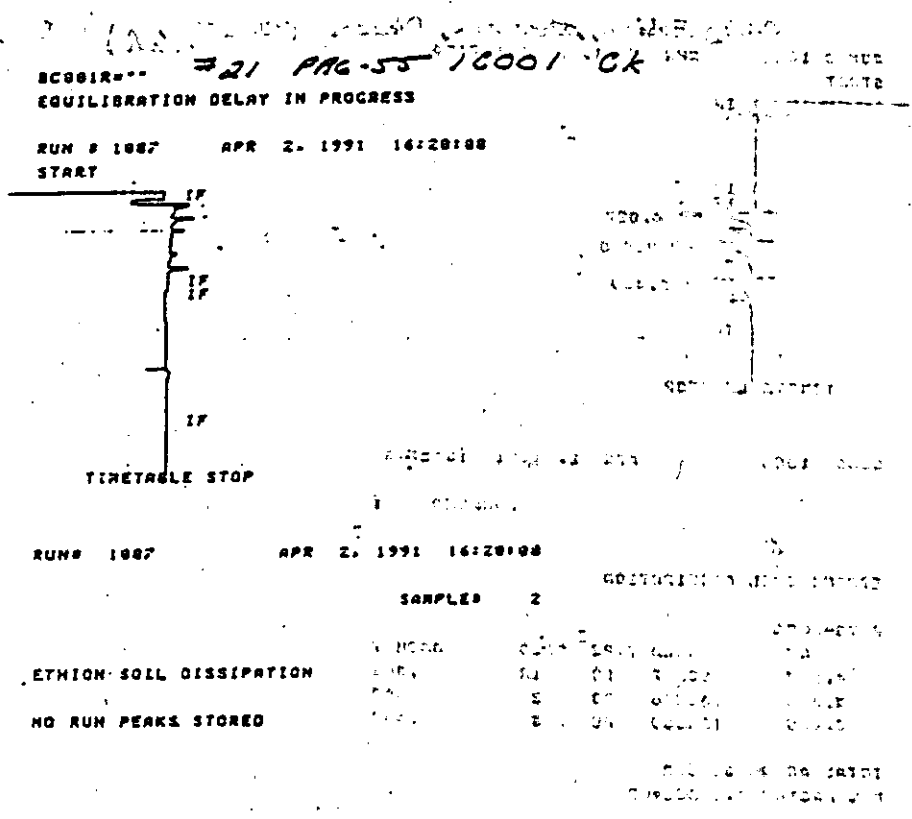
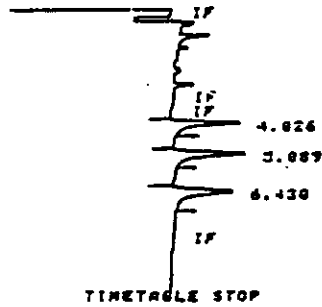


FIGURE 6

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 01, PRE-TREATMENT
PRG-55, #1C001, 0-6"
2 µl, 10 mg

322 PRG-55 1C001 CK + Fort. 0.05 ppm

RUN 8 1888. APR 2, 1991 16:42:39
START



RUN 8 1888 APR 2, 1991 16:42:39
SAMPLES 3

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALS	AMOUNT
	4.826	91029	PS	1R	.466
	5.089	113389	PS	2	.448
	6.430	117339	PS	3	.434

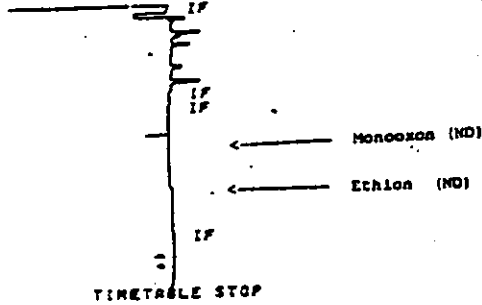
TOTAL AREA= 323977
MUL FACTOR=1.0000E+00

FIGURE 7

PRE-TREATMENT SOIL, TRIAL 01
PRG-55, #1C007, 0-6"
2 μ l, 10 mg

#28 PRG-55 1C007 CK

RUN 8 1897 APR 2, 1991 20:03:31
START



RUN 8 1897 APR 2, 1991 20:03:31

SAMPLES 1201

ETHION SOIL DISSIPATION

NO RUN PEAKS STORED

TIME	AREA	CONC	IDENT
1.1	1.2	0.1	ETHION
1.2	1.5	0.1	ETHION
1.3	1.8	0.1	ETHION
1.4	2.1	0.1	ETHION
1.5	2.4	0.1	ETHION
1.6	2.7	0.1	ETHION
1.7	3.0	0.1	ETHION
1.8	3.3	0.1	ETHION
1.9	3.6	0.1	ETHION
2.0	3.9	0.1	ETHION
2.1	4.2	0.1	ETHION
2.2	4.5	0.1	ETHION
2.3	4.8	0.1	ETHION
2.4	5.1	0.1	ETHION
2.5	5.4	0.1	ETHION
2.6	5.7	0.1	ETHION
2.7	6.0	0.1	ETHION
2.8	6.3	0.1	ETHION
2.9	6.6	0.1	ETHION
3.0	6.9	0.1	ETHION
3.1	7.2	0.1	ETHION
3.2	7.5	0.1	ETHION
3.3	7.8	0.1	ETHION
3.4	8.1	0.1	ETHION
3.5	8.4	0.1	ETHION
3.6	8.7	0.1	ETHION
3.7	9.0	0.1	ETHION
3.8	9.3	0.1	ETHION
3.9	9.6	0.1	ETHION
4.0	9.9	0.1	ETHION
4.1	10.2	0.1	ETHION
4.2	10.5	0.1	ETHION
4.3	10.8	0.1	ETHION
4.4	11.1	0.1	ETHION
4.5	11.4	0.1	ETHION
4.6	11.7	0.1	ETHION
4.7	12.0	0.1	ETHION
4.8	12.3	0.1	ETHION
4.9	12.6	0.1	ETHION
5.0	12.9	0.1	ETHION
5.1	13.2	0.1	ETHION
5.2	13.5	0.1	ETHION
5.3	13.8	0.1	ETHION
5.4	14.1	0.1	ETHION
5.5	14.4	0.1	ETHION
5.6	14.7	0.1	ETHION
5.7	15.0	0.1	ETHION
5.8	15.3	0.1	ETHION
5.9	15.6	0.1	ETHION
6.0	15.9	0.1	ETHION
6.1	16.2	0.1	ETHION
6.2	16.5	0.1	ETHION
6.3	16.8	0.1	ETHION
6.4	17.1	0.1	ETHION
6.5	17.4	0.1	ETHION
6.6	17.7	0.1	ETHION
6.7	18.0	0.1	ETHION
6.8	18.3	0.1	ETHION
6.9	18.6	0.1	ETHION
7.0	18.9	0.1	ETHION
7.1	19.2	0.1	ETHION
7.2	19.5	0.1	ETHION
7.3	19.8	0.1	ETHION
7.4	20.1	0.1	ETHION
7.5	20.4	0.1	ETHION
7.6	20.7	0.1	ETHION
7.7	21.0	0.1	ETHION
7.8	21.3	0.1	ETHION
7.9	21.6	0.1	ETHION
8.0	21.9	0.1	ETHION
8.1	22.2	0.1	ETHION
8.2	22.5	0.1	ETHION
8.3	22.8	0.1	ETHION
8.4	23.1	0.1	ETHION
8.5	23.4	0.1	ETHION
8.6	23.7	0.1	ETHION
8.7	24.0	0.1	ETHION
8.8	24.3	0.1	ETHION
8.9	24.6	0.1	ETHION
9.0	24.9	0.1	ETHION
9.1	25.2	0.1	ETHION
9.2	25.5	0.1	ETHION
9.3	25.8	0.1	ETHION
9.4	26.1	0.1	ETHION
9.5	26.4	0.1	ETHION
9.6	26.7	0.1	ETHION
9.7	27.0	0.1	ETHION
9.8	27.3	0.1	ETHION
9.9	27.6	0.1	ETHION
10.0	27.9	0.1	ETHION

ETHION SOIL DISSIPATION
NO RUN PEAKS STORED

FIGURE 8

CONTROL SOIL
TRIAL 02, PRE-TREATMENT
PRG-56, #2C001, 0-6"
2 μ l, 10 mg

#101 PRG-56 20001

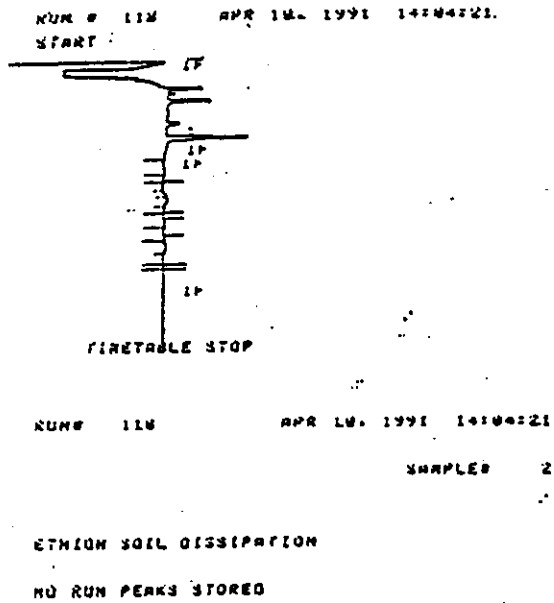
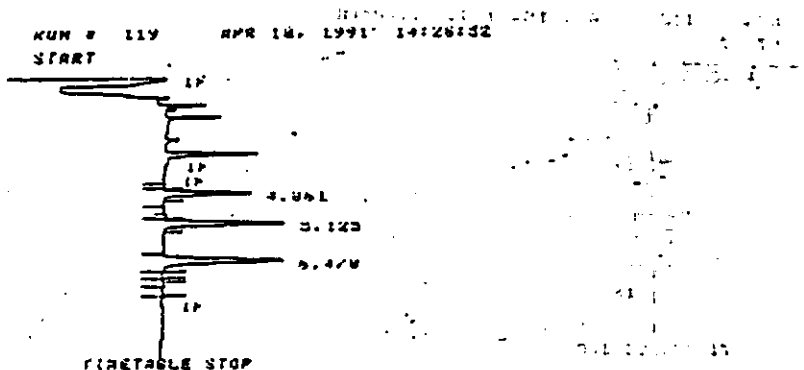


FIGURE 9

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 02, PRE-TREATMENT
PRG-56, #2C001, 0-6"
2 µl, 10 mg

#102 PRG-56 2C001 + Fort. 0.05ppm



NUM # 119 APR 18. 1991 14126132
SAMPLES 3

ETHION SOIL DISSIPATION

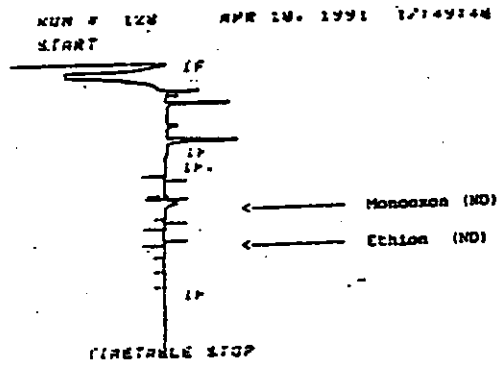
ESTO-AREA	RT	AREA	TYPE	CALS	AMOUNT
	4.861	23199	BB	12	.514
	5.123	36649	BB	2	.496
	6.470	46144	BB	3	.491

TOTAL AREA= 105992
RUL FACTOR=1.8880E+06

FIGURE 10

PRE-TREATMENT SOIL, TRIAL 02
PRG-56, #2C007, 0-6"
2 μ l, 10 mg

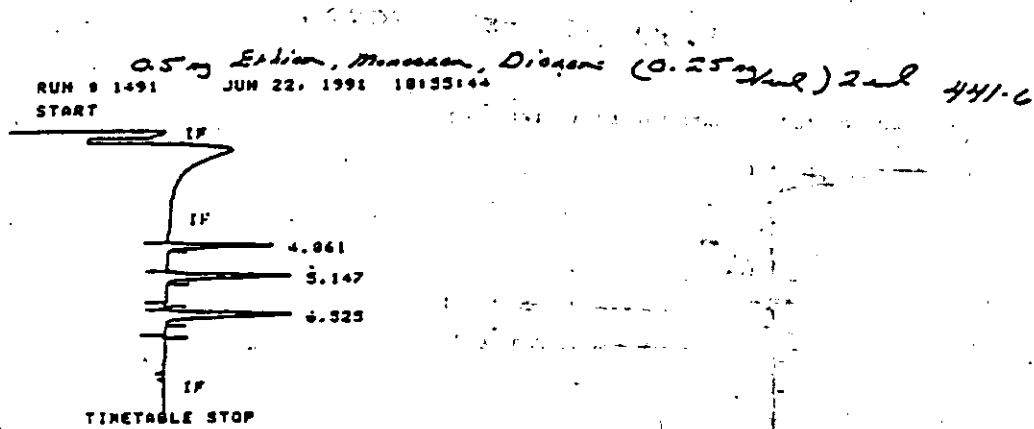
#108 PRG-56 20007



RUN# 128 APR 18. 1991 17:49:48
SAMPLE# 12

ETHIN SOIL DISSIPATION
NO RUN PEEKS STORED

FIGURE 11
ANALYTICAL STANDARD
411-6
2 μ l, 0.25 ng/ μ l



RUN 1491 JUN 22, 1991 18:55:44

SAMPLED 1

ETHION SOIL DISSIPATION

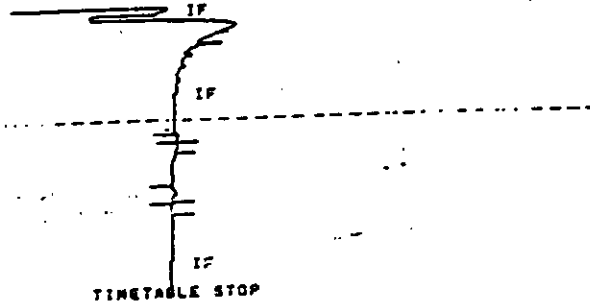
ESTD-AREA	RT	AREA	TYPE	CALS	AMOUNT
	4.061	54723	88	1R	.496
	5.147	79273	88	2	.499
	6.525	100891	88	3	.592

FIGURE 12

CONTROL SOIL
TRIAL 01, 7-DAY
PRG-156, #1C061, 0-6"
2 μ l, 10 mg

#489 PRG-156 1C061

RUN 1492 JUN 22. 1991 11:18:21
START



RUN 1492 JUN 22. 1991 11:18:21

SAMPLES 2

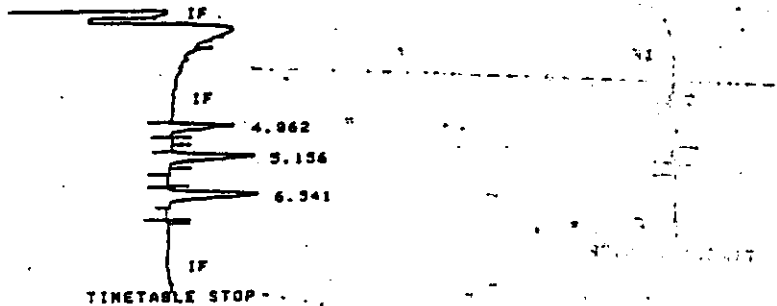
ETHION SOIL DISSIPATION
NO RUN PEAKS STORED

FIGURE 13

PORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 01, 7-DAY
PRG-156, #1C061, 0-6"
2 μ l, 10 mg

#490 PRG-156 1C061 + FOL 0.05PPM

RUN 9 1493 JUN 22, 1991 11:48:47
START



RUN 9 1493 JUN 22, 1991 11:48:47

SAMPLED 3

ETHION SOIL DISSIPATION

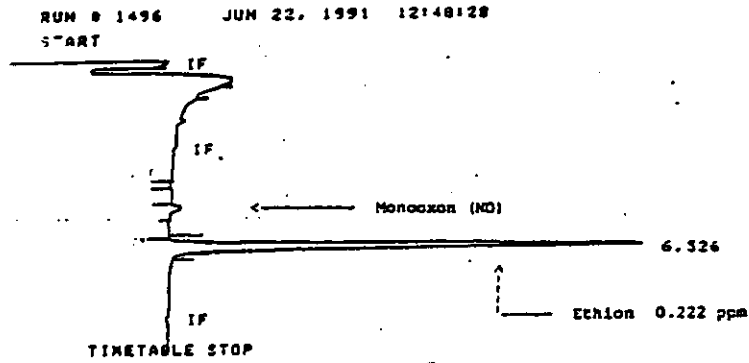
ESTO-AREA	RT	AREA	TYPE	CALC	AMOUNT
	4.862	47731	PS	1R	.433
	5.156	74989	BB	2	.472
	6.341	91374	BV	3	.454

TOTAL AREA= 214114
MUL FACTOR=1.8888E+00

FIGURE 14

TREATED SOIL
TRIAL 01, 0-DAY
PRG-148, #1C212, 0-6"
2 μ L, 10 mg

#492 PRG-148 1C212



RUN# 1496 JUN 22, 1991 12:48:28

SAMPLED 6

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA TYPE	CALC	AMOUNT
	6.326	427235 BB	3	2.124

TOTAL AREA= 427235
MUL FACTOR=1.8808E+00

FIGURE 15

CONTROL SOIL
TRIAL 02, PRE-TREATMENT
PRG-56, #2C001, 0-6"
2 μ l, 10 mg

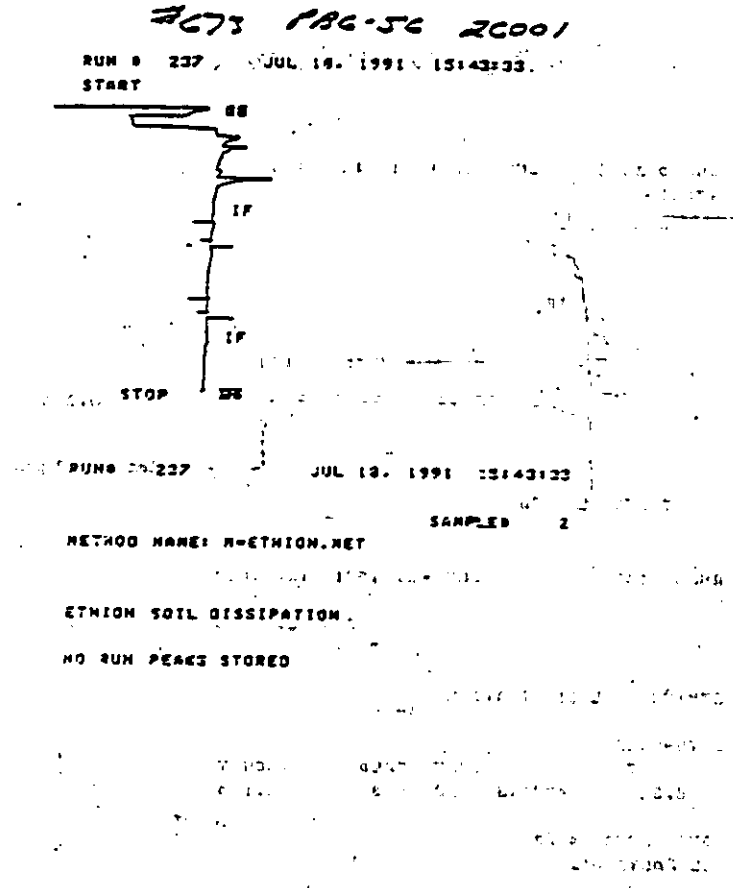
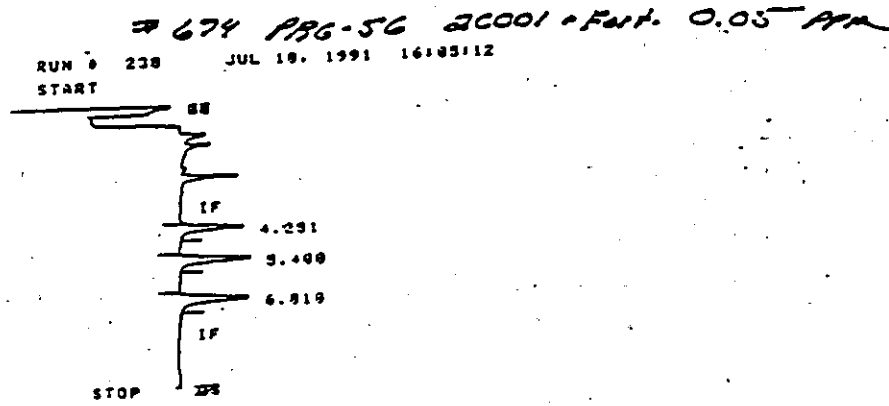


FIGURE 16

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 02, PRE-TREATMENT
PRG-56, #2C001, 0-6"
2 µl, 10 mg



RUN# 238 JUL 18. 1991 16103112

SAMPLES 3

METHOD NAME: M-ETHION.NET

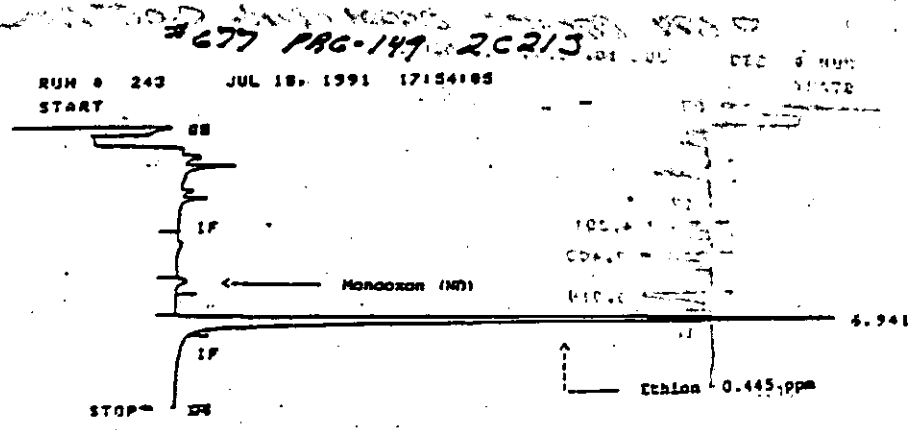
ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	4.231	49794	PS	1R	.417
	5.400	58462	SS	2	.463
	6.310	64832	SS	3	.468

TOTAL AREA= 164310
MUL FACTOR=1.3000E+00

FIGURE 17

TREATED SOIL
TRIAL 02, 0-DAY
PRG-149, #2C213, 0-6"
2 µl, 10 mg



RUN# 243 JUL 18, 1991 17:54:05

RUN# 243 JUL 18, 1991 17:54:05

METHOD NAME: N-ETHION.MET

SAMPLE# 898-H0101001-10001-00000

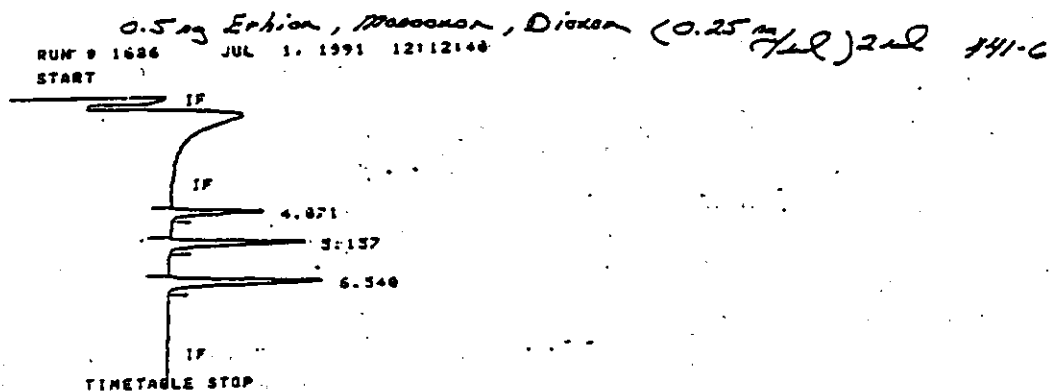
ETHION, SOIL DISSIPATION

ESTD-AREA	RT	AREA TYPE	W/CALS
6.941	623152	PK	100.0
TOTAL AREA= 623152			
MUL FACTOR=1.0000E+00			

AMOUNT	CONC	UNIT
4.49701	0.445	PPM

0.445 ppm

FIGURE 18
ANALYTICAL STANDARD
411-6
2 μ l, 0.25 ng/ μ l



RUN 9 1686 JUL 1, 1991 12:12:40
SAMPLES 1

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALS	AMOUNT
	4.071	63013	00	1R	.511
	5.157	104120	00	2	.529
	6.540	138746	00	3	.525

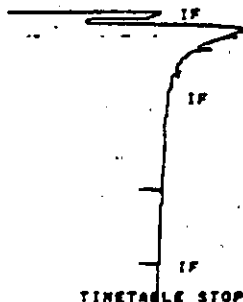
TOTAL AREA= 305887
MUL FACTOR=1.0000E+00

FIGURE 19

CONTROL SOIL
TRIAL 01, 7-DAY
PRG-156, #1C067, 0-6"
2 μ l, 10 mg

#531 PRG-156 1C067

RUN # 1687 JUL 1, 1991 12:35:15
START



RUN# 1687 JUL 1, 1991 12:35:15

	SAMPLES	2
ETHION SOIL DISSIPATION	01	02
NO RUN PEAKS STORED	02	03

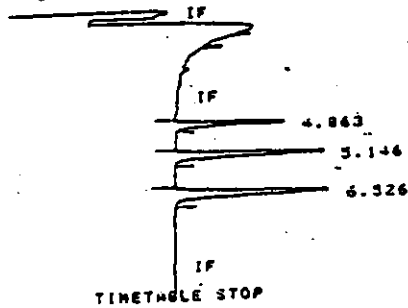
ADDITIONAL INFORMATION

FIGURE 20

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 01, 7-DAY
PRG-156, #1C067, 0-6"
2 µl, 10 mg

#532 PRG-156 1C067 + Fort. 0.05 ppm

RUN 9 1688 JUL 1. 1991 12:57:46
START



RUN 9 1688 JUL 1. 1991 12:57:46
SAMPLE 3

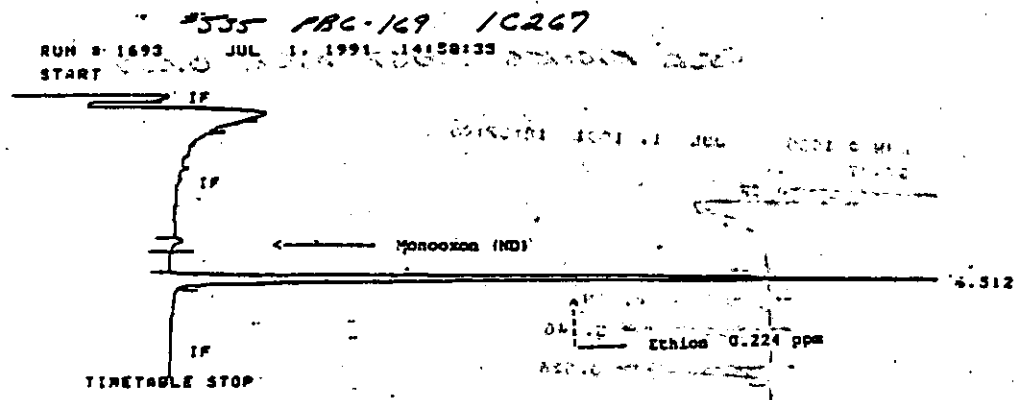
ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
4.863		63735	PB	1R	.517
5.146		104491	PB	2	.522
6.526		108197	PB	3	.493

TOTAL AREA= 298473
AUL FACTOR=1.0000E+00

FIGURE -21

TREATED SOIL
TRIAL 01, 30-DAY
PRG-169, #1C267, 0-6"
2 µl, 10 mg



RUN 8 1693 JUL 1, 1991 14158133

SAMPLED 9

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	6.312	627660	PK	3	2.376

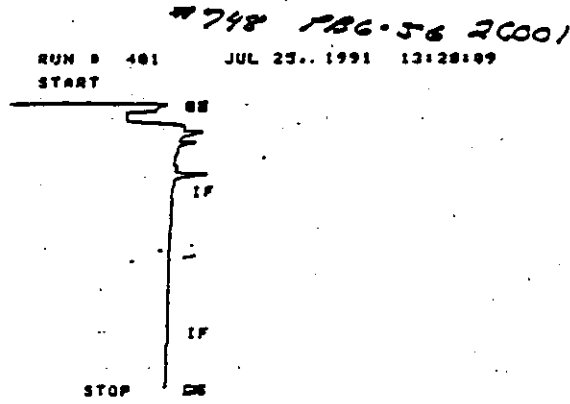
TOTAL AREA= 627660
MUL FACTOR=1.0000E-00

ETHION SOIL DISSIPATION

MONOOXON (ND)

FIGURE 22

CONTROL SOIL
TRIAL 02, PRE-TREATMENT
PRG-56, #2C001, 0-6"
2 μ l, 10 mg



RUN# 401 JUL 25. 1991 13:28:09

SAMPLE# 2

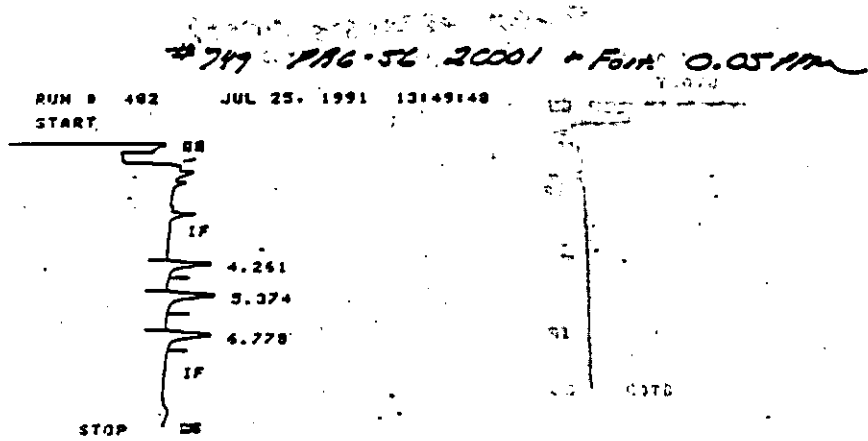
METHOD NAME: M-ETHION.MET

ETHION SOIL DISSIPATION

NO RUN PEAKS STORED

FIGURE 23

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 02, PRE-TREATMENT
PRG-56, #2C001, 0-6"
2 µl, 10 mg



RUN 402 JUL 25 1991 13149148
START

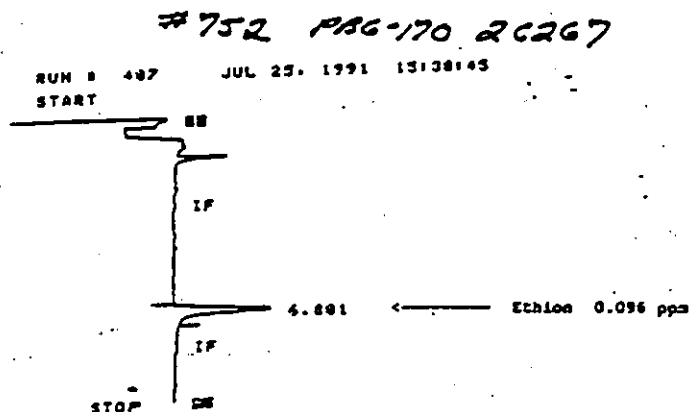
RUN 402 JUL 25 1991 13149148
METHOO NAME: R-ETHION.MET
ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALS	AMOUNT
	4.261	47679	PP	1R	.426
	5.374	86374	PP	2	.475
	6.778	96598	PP	3	.449

TOTAL AREA= 244851
MUL FACTOR=1.2000E+00

FIGURE 24

TREATED SOIL
TRIAL 02, 30-DAY
PRG-170, #2C267, 0-6"
2 µl, 10 mg



RUN# 487 JUL 25, 1991 15132145
METHOD NAME: N-ETHION.MET SAMPLE# 3

ETHION SOIL DISSIPATION

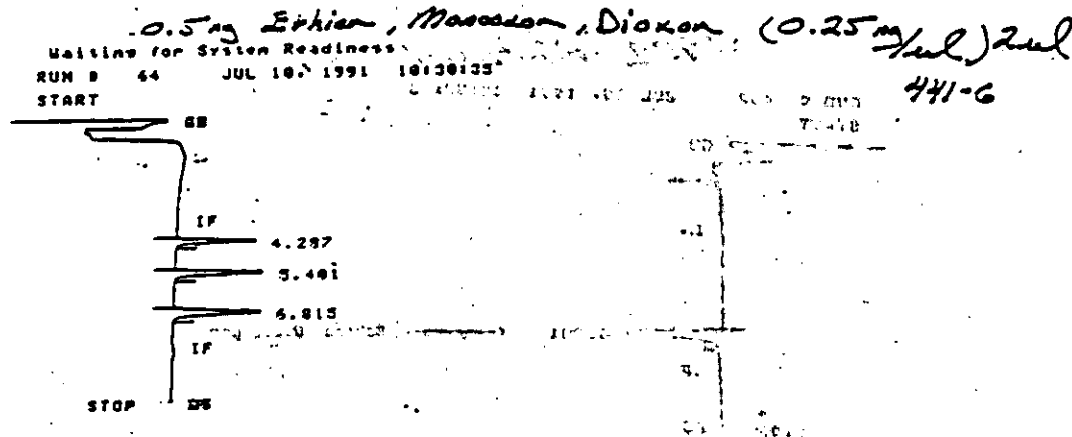
ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	6.801	184134	PK	3	.911

TOTAL AREA= 184134
MUL FACTOR=1.0000E-08

FIGURE 25

ANALYTICAL STANDARD

411-6
2 µl, 0.25 ng/µl



RUN# 64 JUL 10 1991 10:30:35

METHOD NAME: NIETHION.MET

SAMPLES 1

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	4.297	39438	BB	1R	0.2517
	5.491	53756	BB	2	0.3380
	6.815	63373	BB	3	0.508

TOTAL AREA= 158799
MUL FACTOR=1.0000E+00

FIGURE 26

CONTROL SOIL
TRIAL 01, 7-DAY
PRG-156, #1C067, 0-6"
2 μ l, 10 mg

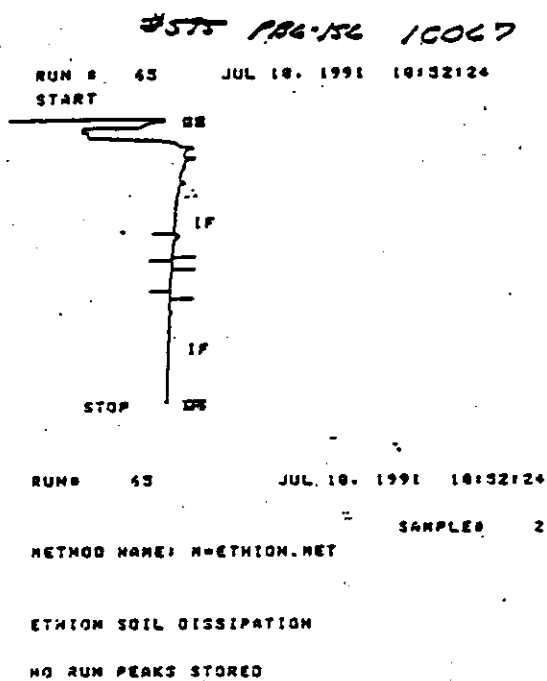
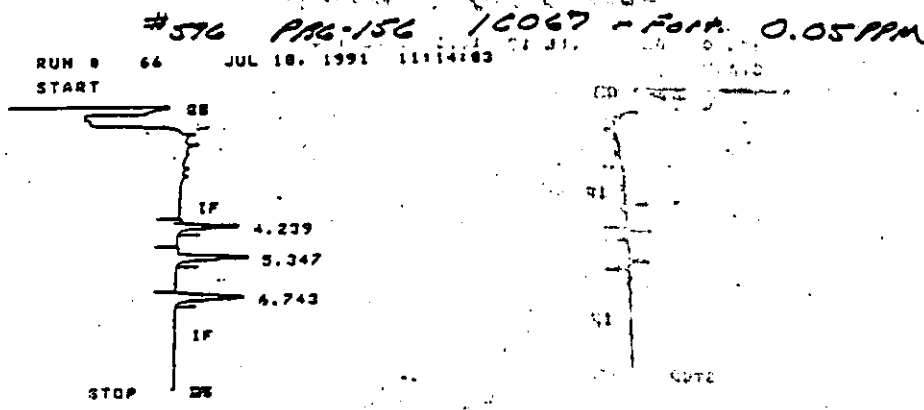


FIGURE 27

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 01, 7-DAY
PRG-156, #1C067, 0-6"
2 µl, 10 mg



RUN# 66 JUL 18. 1991 11114103

METHOD NAME: M-ETHION.MET

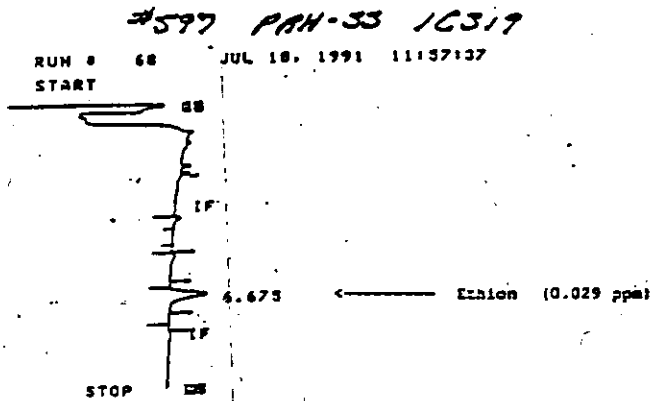
ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALS	AMOUNT
	4.239	34343	VB	1R	.447
	5.347	49834	BB	2	.448
	6.743	55373	PB	3	.438

TOTAL AREA= 139572
MUL FACTOR=1.0868E+08

FIGURE 28

TREATED SOIL
TRIAL 01, 186-DAY
PRH-33, #1C319, 0-6"
2 μ l, 10 mg



RUN# 68 JUL 18. 1991 11:57:37
METHOD NAME: N-ETHION.NET SAMPLED 3

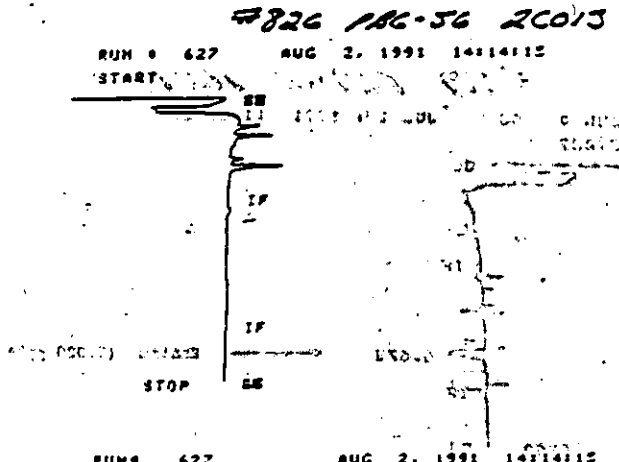
ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	6.673	39877	SB	3	.004

TOTAL AREA= 39877
MUL FACTOR=1.0000E+00

FIGURE 29

CONTROL SOIL
TRIAL 02, PRE-TREATMENT
PRG-56, #2C013, 0-6"
2 µl, 10 mg



RUN# 627 AUG 2, 1991 14:14:12

SAMPLED 2

METHOD NAME: N-ETHION METH

ETHION SOIL DISCIPATION

NO RUN PEAKS STORED

NO. VOLUMES TO BE PRINTED

4276-8713

179

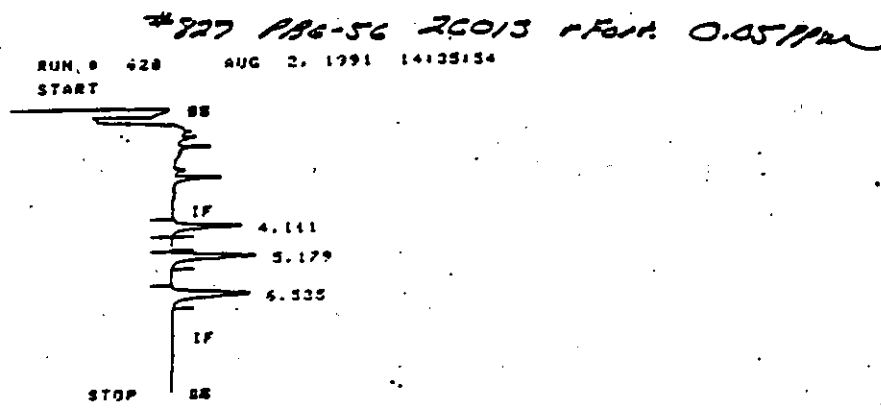
24.00

NO. OF DATA POINTS

COLLECTION TIME

FIGURE 30

FORTIFIED CONTROL SOIL (0.05 ppm)
TRIAL 02, PRE-TREATMENT
PRG-56, #2C013, 0-6"
2 µl, 10 mg



RUN# 428 AUG 2, 1991 14135134
SAMPLED 3
METHOD NAME: N-ETHION.MET

ETHION SOIL DISSIPATION

ESTD-AREA	RT	AREA	TYPE	CALC	AMOUNT
	4.111	44226	BB	1R	.596
	5.179	61593	BB	2	.494
	6.535	49462	BB	3	.432

TOTAL AREA= 175281
MUL FACTOR=1.6888E+00

