Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Dimethenamide

MRID #: 422662-05

Matrix: Soil

Analysis: GC/NPD

This method is provided to you by the Environmental Protection Agency's (EPA) Environmental Chemistry Laboratory (ECL). This method *is not* an EPA method but one which was submitted to EPA by the pesticide manufacturer to support product registration. EPA recognizes that the methods may be of some utility to state, tribal, and local authorities, but makes no claim of validity by posting these methods. Although the Agency reviews *all* Environmental Chemistry Methods submitted in support of pesticide registration, the ECL evaluates only about 30% of the currently available methods. Most methods perform satisfactorily but some, particularly the older methods, have deficiencies. Moreover, the print quality of the methods varies considerably because the methods originate from different sources. Therefore, the methods offered represent the best available copies.

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.

Cover Page for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Dichlormid

MRID #: [illegible]

Matrix: Soil

Analysis: GC/NPD

This method is provided to you by the Environmental Protection Agency's (EPA) Environmental Chemistry Laboratory (ECL). This method is not an EPA method but one which was submitted to EPA by the pesticide manufacturer to support product registrations. EPA recognizes that the methods may be of some utility to state, tribal, and local authorities, but makes no claim of validity by posting these methods. Although the Agency reviews all Environmental Chemistry Methods submitted in support of pesticide registration, the ECL evaluates only about 30% of the currently available methods. Most methods perform satisfactorily but some, particularly the older methods, have deficiencies. Moreover, the quality of the methods varies considerably because there are no specific format requirements. Therefore, the methods provided are sometimes lacking.

If you have difficulties in downloading the method, or further questions concerning the methods, you may contact Elizabeth Flynn at [illegible] or via e-mail at flynn.elizabeth@epa.gov.

DETERMINATION OF

SAN-582H AND OXALAMIDE IN SOIL

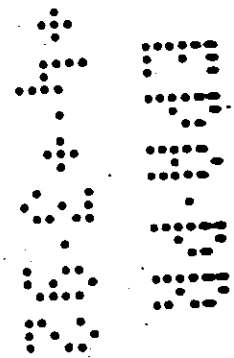
METHOD AM-0830-0290-2

DATE ISSUED: 2/12/92

Sandoz Agro, Inc.
Residue Chemistry Group

Kenton Smith

Thomas Bade



APPENDIX IV

ANALYTICAL METHOD

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ANALYTICAL METHOD

SANDOZ CROP PROTECTION CORPORATION

Location: ◉ 1300 E. TOUHY AVE.
DES PLAINES, IL 60018

◉ DEVELOPMENT ○ QUALITY CONTROL

Method Number AM-0830-0290-2
Addendum AM-0830-0290-1
Supersedes AM-0830-0889-0
Approved BAB Date 2-26-92

Reviewed _____ Date _____
Reviewed _____ Date _____
Reviewed _____ Date _____
Reviewed _____ Date _____

DETERMINATION OF SAN-582H AND ITS OXALAMIDE METABOLITE IN SOIL

1. SUMMARY

1.1 This method has been developed for the analysis of SAN-582H and its Oxalamide metabolite (see Figure 1. for structures) from soil at concentrations in the sub ppm range. It has been reissued to adequately describe areas where particular attention is required for acceptable recoveries. Recoveries now listed in this report are only from experiments performed using these precautions. No changes have been made in how the method is performed, only in the specifications of steps which must be tightly controlled.

1.2 SAN-582H and the Oxalamide are extracted from soil with 1:1 methanol/aqueous sodium hexametaphosphate, an aliquot is diluted with 1N HCl and partitioned with 1:1 ethyl ether:methylene chloride. The organic phase is concentrated and the Oxalamide is derivatized with diazomethane. SAN-582H and Oxalamide (as the methyl ester) are quantitated by gas chromatography using either a nitrogen phosphorous detector (NPD) and an HP-5 Megabore capillary column or a Mass Selective Detector (MSD) and an HP-1 capillary column. In most cases the MSD was necessary to obtain an adequate signal in relation to background. The Nitrogen phosphorous detector sometimes showed backgrounds that were significantly higher than the stated limit of detection. A comparison of some GC/NPD and GC/MSD results for background and recoveries is given in Table 3.

1.3 Sample background, from validation analyses performed with particular attention to all precautions on steps which need tight control, are listed in Table 1. These values are for Gas Chromatographic analysis using the Mass Selective Detector (GC/MSD) since it has been showed that this is the preferred quantitation tool for the best result the first time, without the necessity of confirmation reanalysis. See Table 3 for a comparison of GC/MSD and GC/NPD results.

The highest measured background (signal noise) was 0.002 ppm for Oxalamide and 0.003 ppm for SAN-582H. These values are below the validated limit of detection and are given as indications of expected noise and not to suggest quantitation at this level. Average measured background was 0.0013 ppm (SD=±0.0005, N=4) for Oxalamide with two zero values not included in this average, and 0.0017 ppm (SD=±0.0008, N=6) for SAN-582H with four zero values not included in this average. Five times these noise levels would give a limit of detection of 0.0065 ppm and 0.0085 ppm for Oxalamide and SAN-582H, respectively. The validated limit of detection for both compounds is 0.01 ppm.

1.4 Recoveries of Oxalamide and SAN-582H are given in Table 2. The average recovery for Oxalamide was 99.1% ± 11.4, (N=24). The average recovery for SAN-582H was 94.5% ± 13.7, (N=40). Sample fortifications ranged from 0.01 ppm (N=10) to 0.1 ppm (N=20). All recoveries at each fortification level are given in Table 2.

2. SAFETY

2.1 The oral LD_{50} of SAN-582H in rats is 1570mg/kg.

2.2 Normal laboratory precautions are sufficient for safe handling of SAN-582H.

2.3 Ethyl ether is extremely flammable and methanol and toluene are flammable and should not be used near heat, sparks or open flames.

2.4 All solvents should be used only in well ventilated laboratories.

2.5 Methylene chloride is a suspected carcinogen. Solvent resistant gloves and adequate ventilation should be used when handling methylene chloride.

2.6 Diazomethane and the Diazald® reagent are suspected carcinogens. The methylation step should be done in a hood and gloves should be worn.

2.7 Protective glasses should be worn during all laboratory procedures.

2.8 Disposal of samples and standards must be done in compliance with on-site safety policies and procedures.

3. MATERIALS / METHODS

3.1 Apparatus

3.1.1 Balance, electronic, top-loading, 1000 g capacity, 0.01 g sensitivity.

3.1.2 Balance, electronic, 240 g capacity, 0.1 mg sensitivity.

3.1.3 Bottles, screw cap with Poly-Seal® liners, 8-oz.

3.1.4 Centrifuge, 8 oz. capacity (e.g Damon/IEC Model CU-5000).

3.1.5 Distillation receivers, 15-mL.

3.1.6 Evaporator, nitrogen, (e.g. N-Evap, Organomation Associates, Northborough, MA. 01532)

3.1.7 Funnels, 60°, 75mm.

3.1.8 Gas Chromatograph, Hewlett-Packard model 5890 with Nitrogen Phosphorous Detector (NPD), or similar instrument.

3.1.9 Gas Chromatograph, Hewlett-Packard model 5880 with 5970 Mass Selective Detector (MSD) and autosampler, or similar instrument.

3.1.10 Glass wool, pyrex.

3.1.11 Graduated cylinders, 50-mL.

3.1.12 Kudarna-Danish flasks, 125-mL.

3.1.13 Methylation apparatus(see Figure 2).

3.1.14 Pipets, disposable, 10-mL.

3.1.15 Pipets, volumetric, 1-mL, 2-mL, 5-mL.

3.1.16 Separatory funnels, with Teflon® stoppers, 125-mL.

3.1.17 Vigreaux condensers, with 24/40 joint, 38 cm.

3.1.18 Volumetric flasks, 100-mL.

3.1.19 Water bath, 65 C.

3.2 Reagents

3.2.1 Analytical Reference Standards, SAN-582H and Oxalamide, Sandoz Crop Protection.

3.2.2 Diazald®, Aldrich Chemical Co.

3.2.3 Carbitol®, 2-(2-ethoxyethoxy)ethanol, Aldrich Chemical Co.

3.2.4 Ethyl ether, "Distilled in Glass", Burdick and Jackson, Muskegon, MI 49442.

3.2.5 Hydrochloric acid, concentrated, reagent grade.

3.2.6 Methanol, "Distilled in Glass", Burdick and Jackson, Muskegon, MI 49442.

3.2.7 Methylene chloride, "Distilled in Glass", Burdick and Jackson, Muskegon, MI 49442.

3.2.8 Potassium hydroxide, reagent grade.

3.2.9 Sodium Hexametaphosphate, reagent grade.

3.2.10 Sodium sulfate, anhydrous granular, reagent grade.

3.2.11 Toluene, "Distilled in Glass", Burdick and Jackson, Muskegon, MI 49442.

3.2.12 Water, de-ionized, organic-free.

3.3 Preparation of Standards

3.3.1 Accurately prepare stock solutions (10^{-4}g/µL) of 100 mg SAN-582H and Oxalamide in 100 mL methanol in 100-mL volumetric flasks. Accurately prepare a stock solution (10^{-4}g/µL) of 100 mg Oxalamide Methyl Ester in 100 mL toluene in a 100-mL volumetric flask.

3.3.2 Transfer 1.0 mL each of 10^{-4}g/µL SAN-582H and Oxalamide stock solutions to a 100-mL volumetric flask and bring to the mark with methanol. This solution(10^{-4}g/µL each in methanol) is used for fortifying check samples.

3.3.3 Transfer 1.0 mL each of 10^{-4}g/µL SAN-582H and Oxalamide Methyl Ester stock solutions to a 100-mL volumetric flask and bring to the mark with toluene (10^{-4}g/µL each in toluene).

3.3.4 Prepare a range of standards for GC/NPD quantitation by dilution of aliquots of 10^{-4}g/µL SAN-582H and Oxalamide Methyl Ester in toluene solutions to 100 mL with toluene in volumetric flasks, as below:

STOCK SOLUTION VOLUME	G.C. SOLUTION CONCENTRATION
20.0mL	2.0×10^{-6}g/µL
10.0mL	1.0×10^{-6}g/µL
5.0mL	5.0×10^{-10}g/µL
2.0mL	2.0×10^{-10}g/µL
1.0mL	1.0×10^{-10}
0.5mL	5.0×10^{-10}g/µL

3.4 Procedure

3.4.1 Extraction

3.4.1.2 Weigh 50.0 g of soil into an 8-oz. bottle.

If fortifying for recovery determination, add an appropriate volume of fortifying solution(e.g. 500 µL of 10^{-4}g/µL in 50 g soil gives 0.1 ppm) and evaporate the methanol with a stream of nitrogen.

3.4.1.3 Add 50.0 mL of 50 g/L sodium hexametaphosphate in 0.1N HCL to the sample and shake until soil is dispersed (15 to 45 minutes depending on clay content of soil).

3.4.1.4 Add 50.0 mL of methanol to this water/soil mixture and shake for 15 minutes on the reciprocal shaker. Centrifuge at ca. 500 G until the supernatant is clear (15 to 45 minutes depending on soil characteristics).

3.4.1.5 With a disposable volumetric pipet, transfer a 10-mL aliquot (5 g equivalent of sample) of the methanolic extract to a 125-mL separatory funnel containing 40 mL of 1N HCl and 50 mL of 1:1 ethyl ether/methylene chloride. The 1:1 ethyl ether/methylene chloride solution must be made up just before it is used and any remainder discarded. If excess solvent mixture is used at a later time the ratio is not the same as originally prepared.

3.4.1.6 After equilibration, drain the ethyl ether/methylene chloride (bottom layer) through ca. 10 g of sodium sulfate in a 60° funnel plugged with glass wool, into a Kuderna-Danish flask fitted with 15-mL distillation receiver.

3.4.1.7 Add 50 mL of fresh ethyl ether/methylene chloride to the separatory funnel, shake, equilibrate, and drain again through the sodium sulfate.

3.4.1.8 Rinse the sodium sulfate three times with 5-10 mL of ethyl ether/methylene chloride, add 1.0 mL of methanol and a boiling chip, fit with a Vigreaux condenser, and concentrate to ca. 1-2 mL on the 65°C water bath.

3.4.2 Methylation

3.4.2.1 Add 0.5 mL methanol to the concentrated sample and dilute to 5 mL with ethyl ether.

3.4.2.2 Fill tube A of the methylation apparatus (see Figure 2) ca. 2/3 full with ethyl ether and establish a nitrogen flow of about 30 mL/min.

3.4.2.3 Add 2 mL of 9N KOH, 2.8 mL of 1:1 Carbitol®/ethyl ether, and ca. 100 mg of Diazald® to tube B and seat the stopper.

WARNING

Diazald® is a carcinogen, and the diazomethane gas generated is extremely toxic. Wear gloves and work in an efficiently operating hood. Diazomethane may react violently or explode if the solution contains fine powders or there are ground glass joints or scratched or chipped glassware in the system.

3.4.2.4 Place the distillation receiver containing the sample in position as tube C and pass nitrogen/diazomethane through the sample solution until it turns yellow. If the sample extract has too much color to show the yellow of diazomethane, add ca. 3 mL of ethyl ether to tube D and continue until it turns yellow. It is not necessary to "over Methylate" the samples. When the sample has just turned yellow the sample tube can be removed from the apparatus.

3.4.2.5 Cap the sample tube with a Teflon stopper (do not use ground glass), and allow to stand for 20 min. If the yellow color has disappeared, repeat the addition of diazomethane until the sample just turns yellow. Again, it is not desirable to "over Methylate" the sample. If any yellow color remains additional diazomethane is not necessary.

3.4.2.6 Evaporate the sample extract just to dryness and add 1.0 mL of toluene(or more if high residue levels are present). It is important to make sure the sample is dry, and no residual methanol is present. It is suspected that residual methanol might carry varying amounts of moisture which can participate in hydrolysis of the ester in the hot injector port of the gas Chromatograph. Mix the sample thoroughly and quantitate by GC/NPD, or GC/MSD. GC/MSD has been found to be necessary for many soils because of frequent NPD background interferences.

3.5 Quantitation

3.5.1 Gas Chromatographic Conditions

3.5.1.1 The following instrument and operating conditions have been shown to be suitable for analysis of SAN-582H and Oxalamide in soil. Other conditions may be acceptable provided that SAN-582H and Oxalamide are separated from sample interferences and the response is linear over the range of interest. Elution times and standard linearity must be checked with any new instrument or any change in operating conditions.

3.5.1.2 Instrument: Hewlett-Packard, model 5890 equipped with a 7673A autosampler and a nitrogen phosphorous detector (NPD).

Column: 30 m x 0.53 mm(I.D.) x 0.88 µm film thickness bonded FSOT HP-5 (5% phenyl methyl silicone).

G.C. Parameters:

Carrier Gas: Helium at 17.6 mL/min. (15 psi head pressure).

Detector Gases:
Hydrogen at 3.2 mL/min.
Air at 92 mL/min.
Helium(Make-Up) at 24.4 mL/min.

Oven Temperature: 180°C

Injector Temperature: 250°C

Detector Temperature: 300°C

Retention Times:

SAN-582H Retention Time: 4.06 min.

Oxalamide Retention Time: 3.65 min.

Retention times will vary slightly with exact G.C. operating conditions and column length and condition.

3.5.1.3 Instrument: Hewlett-Packard 5880A Gas Chromatograph equipped with 5970 Mass Selective Detector and 59970 MS ChemStation software.

Column: 25m x 0.2mm x 0.11um film thickness bonded FSOT HP-1 (methyl silicone).

G.C. Conditions:

Carrier Gas: Helium at 0.4 mL/min. (10 psi head pressure).

Injection Mode: Splitless, 0.5min. purge delay.

Injector Temperature: 250°C

Oven Temperature: 100°C for 0.5 min. then 30°C/min. to 220°C and hold for 2.5 min.

Interface Temperature: 275°C

M.S.D. Parameters:

MSD Mode: Selected Ion Monitoring

Oxalamide Methyl Ester: m/e 240,213,and 180

SAN-582H: m/e 230,203, and 154

Retention Times:

Oxalamide Retention Time: 6.47 min.

SAN-582H Retention Time: 6.68 min.

Retention times will vary slightly with exact G.C. operating conditions and column length and condition.

3.5.2 Calculations

3.5.2.1 Prepare standard curves for SAN-582H and Oxalamide by injecting fixed volumes (5 µL for NPD, 2.0 µL for MSD) of a series of standards of known concentrations and plotting peak heights versus concentrations on log-log graph paper.

3.5.2.2 Inject the same volume of sample extract and determine the concentrations of SAN-582H and Oxalamide in the injected aliquot from the peak heights and the standard curves prepared from standards run at the same time.

3.5.2.3 Calculate the concentration of the residues in the sample using the following expression:

$$ppm = \frac{C \times A \times V}{W}$$

Where:

ppm = Concentration of the analyte in the sample in parts per million (µg/g).

C = Concentration of the analyte in the sample extract determined from the standard curve (ng/µL).

V = The volume of the sample extract, taking into account all dilutions (mL).

W = The weight of sample represented by the extract (g).

A = Conversion factor necessary for accommodation of the change in molecular weight of Oxalamide after methylation. (A=0.94 for Oxalamide, A=1 for SAN-582H)

3.6 Confirmatory Techniques

3.6.1 The use of GC/NPD for quantitation gives frequent false positive results. It should be used in conjunction with the GC/MSD for confirmation, or the GC/MSD can be used exclusively if residues are expected in most samples. In such cases the use of an NPD should be limited to sample screening, and suspected residues should be confirmed by MSD.

3.6.2 The use of MSD in SIM mode monitoring three ions for quantitation includes confirmation. The presence, in the sample, of peaks for all three ions in the same ratios as observed in standards gives extremely high confidence that the sample response is due to the compound of interest.

This is reflected in the good agreement between values obtained for each ion when standard curves are prepared for each of the monitored ions and results are calculated as described in section 3.5.2 of this method. When the results for all three ions are similar the average of these three responses may be used to get a single value for the analysis. However, sample co-extractives frequently co-elute and contribute to the response of one or two ions, giving a higher calculated value for those ions. In cases where the result for one ion is much larger than the two remaining ions, and the two remaining results agree closely, the high result may be discounted due to a high contribution from interferences, and the remaining two averaged for a result which still has a high degree of confidence. In cases where there is no close agreement between the results from all three ions, the lowest result may be used on the assumption that co-extractives are contributing to the ions giving the higher results. However, there is no guarantee that co-extractives are not also contributing to the lowest result, and the value obtained has much less confidence.

3.7 Time Required for Analysis

3.7.1 A set of six to eight samples can be prepared in an eight hour day and quantitated overnight using an autosampler. A single sample could be extracted and quantitated in a single eight hour day.

4. RESULTS / DISCUSSION

4.1 Precision and Accuracy

4.1.1 Recoveries from check samples fortified with 0.1 to 0.5 ppm each of SAN-582H and Oxalamide, as quantitated by NPD, are shown in Table 2. The average recovery is 180% with a standard deviation of 317% for SAN-582H (n=77) and 115% with a standard deviation of 203% for Oxalamide (n=77). Also shown in Table 2 are corrected recoveries which have had the background from the corresponding check samples subtracted. The corrected recoveries averaged 120% for SAN-582H (n=75) and 96% for Oxalamide (n=75).

4.1.2 Recoveries from check samples fortified with 0.1 to 0.5 ppm each of SAN-582H and Oxalamide, as quantitated by MSD, are shown in Table 4. The average recovery is 115% with a standard deviation of 50% for SAN-582H (n=93) and 90% with a standard deviation of 53% for Oxalamide (n=93). Also shown in Table 2 are corrected recoveries which have had the background from the corresponding check samples subtracted. The corrected recoveries averaged 115% for SAN-582H (n=85) and 89% for Oxalamide (n=85).

Note

4.2 Limits of Detection

4.2.1 The limit of detection for this method is 0.01 ppm each for SAN-582H and Oxalamide. This limit cannot reliably be achieved using NPD for quantitation, due to frequent interferences, but quantitation using MSD is generally reliable down to the limit of detection. A lower limit may be obtained by concentration of the sample extract to a smaller volume, or by partitioning a larger aliquot of the acidic methanol extract, if sample background permits.

5. CONCLUSION

5.1 This method detects and quantitates SAN-582H and Oxalamide in a variety of soil types with a limit of detection of 0.01 ppm. The use of NPD for detection and quantitation of SAN-582H and Oxalamide gives frequent false positives, and should be considered only for screening purposes. For confirmation and more reliable quantitation, the use of MSD is recommended. Because of the variability of soils, no guarantee of these limits and recoveries for all soil types can be assumed.

6. **CERTIFICATION**

I hereby state that as principal author and chemist of record, that the method herin was conducted within the framework of the GLP program at Sandoz Crop Protection. The description of this method and the supporting data (backgrounds and recoveries) are correct to the best of my knowledge.

KEITH SMITH	
name	signature
SECTION LEADER, RESIDUE CHEMISTRY	2/24/92
title	date
Thomas Bade	Thomas Bade
name	signature
Manager, Residue Chemistry	2/21/92
title	date

Table 1. Background in Check Samples - GC/MSD Analyses

Sample #	Extraction Date	Analysis Date	GC/MSD Response Oxalamide	GC/MSD Response SAN-582H
1 03-001-3C CK	10/04/91	10/07/91	T 0.001	A 0.000
6 03-001-3C CK	10/04/91	10/07/91	T 0.002	A 0.000
11 02-911-0C CK	10/21/91	10/22/91	--	A 0.002
12 02-911-0C CK	10/21/91	10/22/91	--	A 0.003
21 02-911-0C CK	10/22/91	10/23/91	--	T 0.001
22 02-911-0C CK	10/22/91	10/23/91	--	A 0.002
31 02-008-1C CK	11/13/91	11/15/91	T 0.000	A 0.001
32 02-008-1C CK	11/13/91	11/15/91	T 0.001	A 0.001
41 02-012-0C CK	11/19/91	11/21/91	T 0.001	A 0.000
42 02-012-0C CK	11/19/91	11/21/91	T 0.000	A 0.000

Table 2. Recoveries of SAN-582H and Oxalamide from Fortified Control Samples

Sample #	Extraction Date	Analysis Date	Fort Level	*GC/MSD Recovery Oxalamide	SAN-582H
2 03-001-3C F0	10/04/91	10/07/91	0.01	T 87	A 89
3 03-001-3C F1	10/04/91	10/07/91	0.05	A 98	A 85
4 03-001-3C F2	10/04/91	10/07/91	0.10	A 81	A 80
5 03-001-3C F2	10/04/91	10/07/91	0.10	A 96	A 79
7 03-001-3C F0	10/04/91	10/07/91	0.01	L 79	A 86
8 03-001-3C F1	10/04/91	10/07/91	0.05	T 90	A 83
9 03-001-3C F2	10/04/91	10/07/91	0.10	A 97	A 77
10 03-001-3C F2	10/04/91	10/07/91	0.10	A 89	A 71
13 02-911-0C F0	10/21/91	10/22/91	0.01	--	A 106
14 02-911-0C F0	10/21/91	10/22/91	0.01	--	T 114
15 02-911-0C F1	10/21/91	10/22/91	0.05	--	A 78
16 02-911-0C F1	10/21/91	10/22/91	0.05	--	A 92
17 02-911-0C F2	10/21/91	10/22/91	0.10	--	A 108
18 02-911-0C F2	10/21/91	10/22/91	0.10	--	A 75
19 02-911-0C F2	10/21/91	10/22/91	0.10	--	T 107
20 02-911-0C F2	10/21/91	10/22/91	0.10	--	A 84
23 02-911-0C F0	10/22/91	10/23/91	0.01	--	A 119
24 02-911-0C F0	10/22/91	10/23/91	0.01	--	A 94
25 02-911-0C F1	10/22/91	10/23/91	0.05	--	A 98
26 02-911-0C F1	10/22/91	10/23/91	0.05	--	A 75
27 02-911-0C F2	10/22/91	10/23/91	0.10	--	A 109
28 02-911-0C F2	10/22/91	10/23/91	0.10	--	A 104
29 02-911-0C F2	10/22/91	10/23/91	0.10	--	A 103
30 02-911-0C F2	10/22/91	10/23/91	0.10	--	79
33 02-008-1C F0	11/13/91	11/15/91	0.01	T 90	A 120
34 02-008-1C F0	11/13/91	11/15/91	0.01	L 110	T 120
35 02-008-1C F1	11/13/91	11/15/91	0.05	T 112	A 104
36 02-008-1C F1	11/13/91	11/15/91	0.05	T 112	A 108
37 02-008-1C F2	11/13/91	11/15/91	0.10	T 112	A 100
38 02-008-1C F2	11/13/91	11/15/91	0.10	T 102	A 90
39 02-008-1C F2	11/13/91	11/15/91	0.10	T 108	A 96
40 02-008-1C F2	11/13/91	11/15/91	0.10	T 101	A 89
43 02-012-0C F0	11/19/91	11/21/91	0.01	L 130	L 115
44 02-012-0C F0	11/19/91	11/21/91	0.01	T 100	A 93
45 02-012-0C F1	11/19/91	11/21/91	0.05	T 96	A 99
46 02-012-0C F1	11/19/91	11/21/91	0.05	T 108	A 104
47 02-012-0C F2	11/19/91	11/21/91	0.10	A 91	A 86
48 02-012-0C F2	11/19/91	11/21/91	0.10	A 96	A 81
49 02-012-0C F2	11/19/91	11/21/91	0.10	A 98	A 88
50 02-012-0C F2	11/19/91	11/21/91	0.10	A 95	A 90

* NO BACKGROUND CORRECTIONS WERE MADE

Table 3. Comparison of GC/MSD and GC/NPD Analyses

Sample I.D. #	Soil Source	Background from GC/MSD and GC/NPD Analyses of Soil Samples[1]			
		-------GC/MSD-------		-------GC/NPD-------	
		Oxalamide	SAN-582H	Oxalamide	SAN-582H
0411	N. Carolina	0.001	0.005	0.130*	0.065*
0421	N. Carolina	0.000	0.005	0.144*	0.121*
0401	Illinois	0.001	0.005	0.000	0.009
0411	Illinois	0.001	0.003	0.009	0.015
	Illinois	0.001	0.006	0.009	0.015
	Illinois	0.001	0.000	0.000	0.000
0401	Indiana	0.000	0.001	0.000	2.647*
	Indiana	0.022	0.007	0.000	2.288*
2001	Mississippi	0.001	0.005	0.106*	0.044*

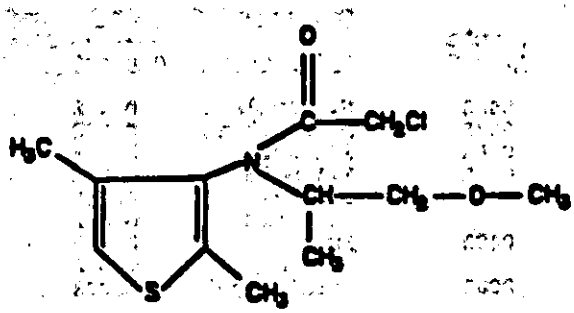
Sample I.D. #	Soil Source	% Recoveries from 0.2 ppm Fortifications of Oxalamide and SAN-582H, After Subtraction of Above Background			
		-------GC/MSD-------		--------GC/NPD--------	
		Oxalamide	SAN-582H	Oxalamide	SAN-582H
0411	N. Carolina	68.7	89.4	37.8*	64.1*
0421	N. Carolina	68.8	93.1	38.1*	65.5*
0401	Illinois	87.5	86.3	89.1	96.7(92.3)[2]
0411	Illinois	67.2	114	90.1(85.6)[2]	95.2(87.5)[2]
	Illinois	104	113	90.1(85.6)[2]	95.2(87.5)[2]
	Illinois	84.3	80.4	83.4	84.3
0401	Indiana	153	157	184*	792(-532)[2]*
	Indiana	77.3	70.5	219*	693(-631)[2]*
2001	Mississippi	68.9	98.8	16.5*	62.3*

[1] Values below 0.01 ppm are below the lowest GC standard and outside the validated range of this method. They are included to demonstrate a typical background which may be observed.

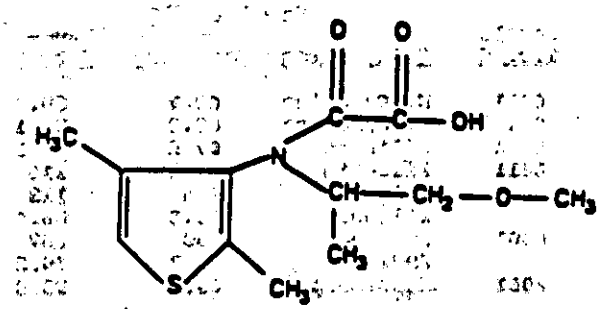
[2] The first number is without background correction. Number in parenthesis is the recovery after background subtraction.

* Examples of NPD analyses which must be confirmed and quantitated by GC/MSD because of interfering background.

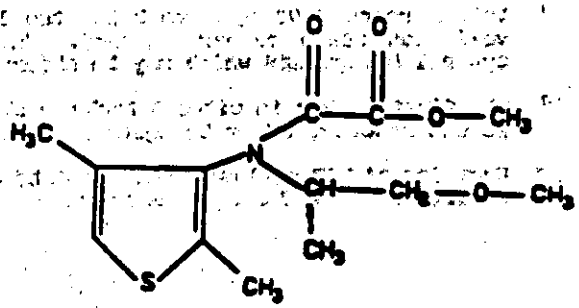
Figure 1. Chemical Structures of SAN-582H and Related Compounds



SAN-582H, 2-chloro-N-(1-methyl-2-methoxy)ethyl)-N-(2,4-dimethyl-thien-3-yl)-acetamide



Oxalamide, N-(1-methyl-2-methoxy)ethyl)-N-(2,4-dimethyl-thien-3-yl)-oxalamide



Oxalamide, N-(1-methyl-2-methoxy)ethyl)-N-(2,4-dimethyl-thien-3-yl)-oxalamide methyl ester

Figure 2. Methylation Apparatus

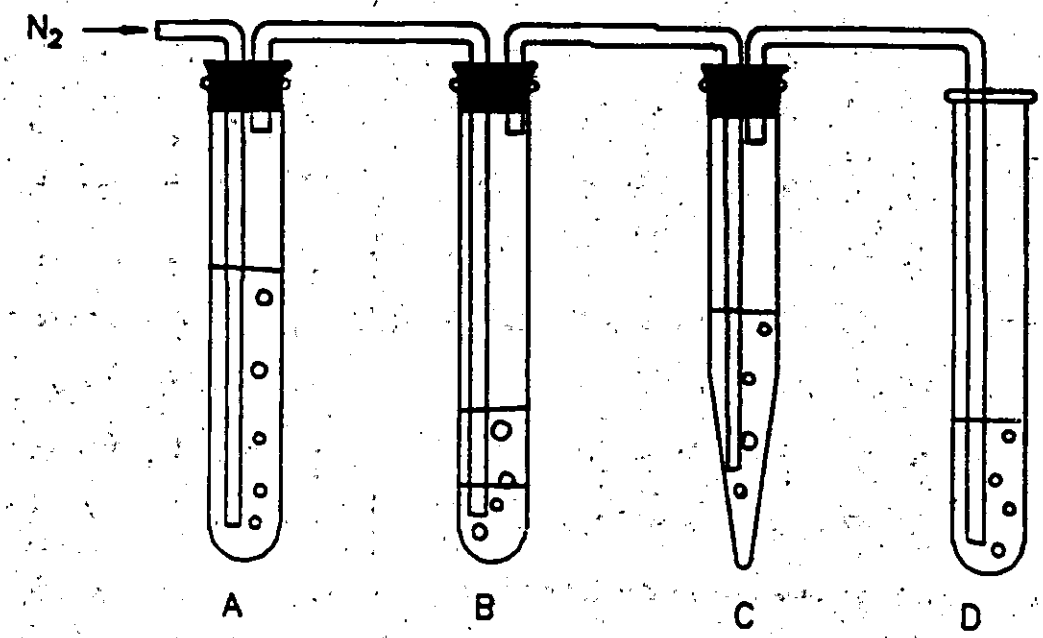
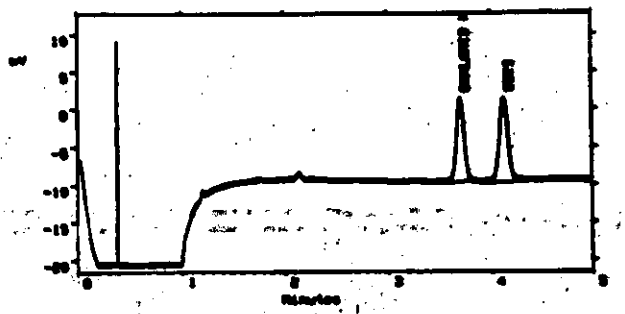
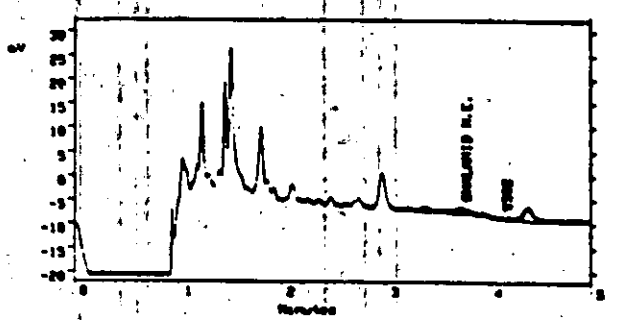


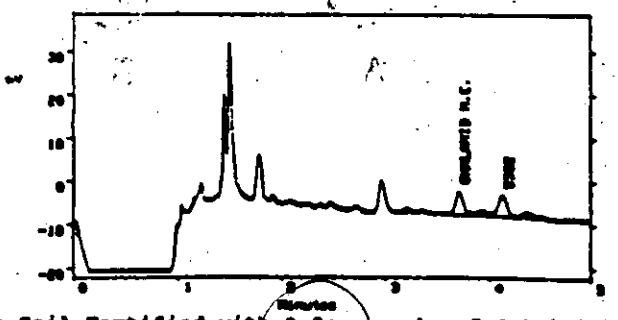
Figure 3. Typical Chromatograms - NPD



a. Standards - 5.0µL injected, $2x10^{-10}$g/µL each SAN-582H and Oxalamide

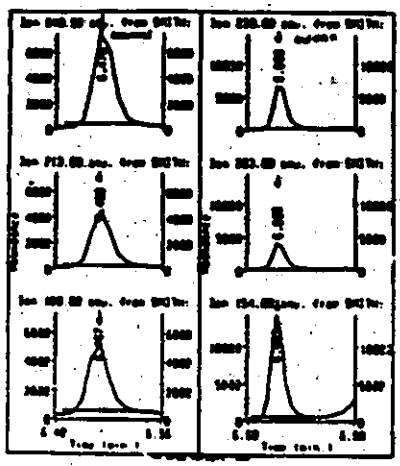


b. Check Soil - 5.0µL injected, 5g/5mL, <0.01ppm SAN-582H, 0.016ppm Oxalamide

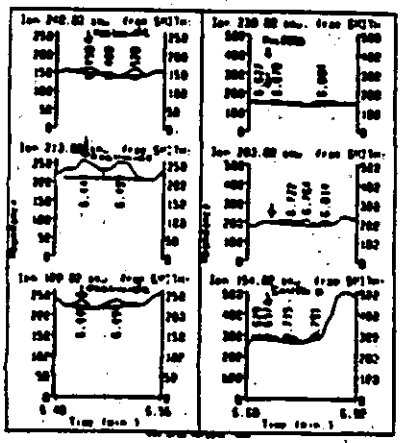


c. Check Soil Fortified with 0.5ppm each - 5.0µL injected, 5g/5mL, 99% Recovery of SAN_582H, 89% Recovery of Oxalamide

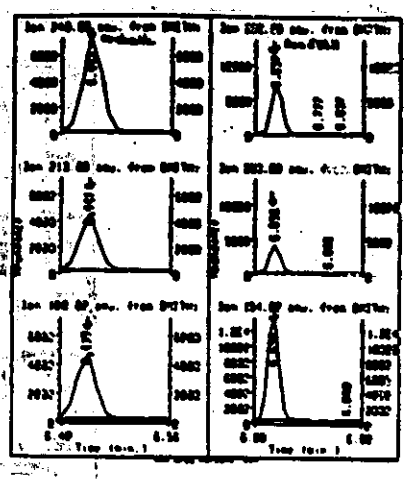
Figure 4. Typical Chromatograms - NSD



a. Standard Injection, 2.0µL, 1.0ng/µL SAN-582H and Oxalamide



b. Check Soil Sample, 2.0µL injected, 5g/1mL, <0.01ppm each SAN-582H and Oxalamide



c. Check Soil Sample Fortified with 0.2ppm each SAN-582H and Oxalamide, 2.0µL injected, 5g/mL, 83% Recovery of SAN-582H, 82% Recovery of Oxalamide