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OPERATION SAFE REMOVAL: SPRING VALLEY, WASHINGTON, D.C. ANALYTICAL RESULTS: JANUARY - FEBRUARY 1993

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On 5 January 1993, laboratory debris/soils and munitions from WWI chemical warfare (CW) agent studies at American University were discovered in the Spring Valley development in Washington, D.C. Ninety-eight separate items/samples, including nine intact munitions, were packaged and delivered by the Technical Escort Unit (TEU) to the U.S. Army Edgewood Research, Development and Engineering Center for chemical characterization.

This report summarizes the analytical results obtained on the samples submitted from the Spring Valley site. It identifies the various samples delivered for analysis and describes the analytical methodology used to identify and confirm the chemicals contained in each sample. Spectra for each sample in which chemicals were identified are included in the appendices.

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PREFACE

The work described in this report was authorized under Project No. 3WBBRX, Operation Safe Removal, Spring Valley, Washington, D.C. This work was started in January 1993 and completed in February 1993.

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1. INTRODUCTION

On 5 January 1993, laboratory debris and munitions from World War I chemical warfare (CW) agent studies at American University were discovered in the Spring Valley development in Washington, D.C. U.S. Army experts in munitions, environmental hazards, and CW agent removal and transportation accepted responsibility for the identification and removal of any hazardous materials. The U.S. Army Edgewood Research, Development and Engineering Center (ERDEC) effort in support of Operation Safe Removal included: (1) selection of suspect samples/items from the area by the Technical Escort Unit (TEU); (2) packaging of the samples in sealed, stainless steel shipping containers (i.e., "pigs"); (3) transportation of the samples to ERDEC for chemical analysis; and, (4) chemical characterization of each sample/item.

The samples consisted of a variety of materials including soil; various solids; crystals; fibers; charcoal; liquids; laboratory glassware and equipment; household items; munition fills, components, and residues; and metal pellets. All samples underwent a screening protocol. Granular and fibrous materials were first analyzed by thermal desorption/mass spectroscopy and direct exposure probe/mass spectrometry. All samples were subjected to solvent extraction/leaching. The recovered solvents were analyzed by nuclear magnetic resonance (NMR) spectroscopy, direct exposure probe/mass spectrometry (DEP/CI), gas chromatography/mass spectrometry (GC/MS), high performance liquid chromatography/ion chromatography (HPLC/IC), and infrared (IR) spectrometry. Elemental analyses were performed using inductively coupled plasma (ICP) emission spectrophotometry and atomic absorption (AA) spectrometry on selected samples, as appropriate.

This report summarizes the analytical results obtained on the samples submitted from the Spring Valley site. The report identifies the various samples delivered to ERDEC (Bldg E3300) for analysis and describes the analytical methodology used to identify and confirm the chemicals contained in each sample. Spectra for each sample in which chemicals were identified are included in the appendices.

During the course of these analyses, informal interim reports on the results were dispatched by E-mail to personnel involved in Operation Safe Removal. The final E-mail report summarizing the analytical results for all of the samples received through February 1993 is included as Appendix A.

2. EXPERIMENTAL PROCEDURES

2.1 Sample Preparation

Each sample was prepared for analysis according to a set protocol depending on the type of sample, i.e., broken glass, pieces of hardware, soils, powders, rocks, rubber stoppers, liquids, etc. "Neat" (without solvent) liquids (such as from the Livens projectiles), powders, etc. were analyzed as such and in certain cases extracted with solvent. Pieces of broken glass, ceramic pieces, glass and rubber stoppers, glass tubing, small metallic pieces (such as spray adapters, probes and lead balls) were leached first with deuterated chloroform, $CDCl_3$ (99.8 atom% D, MSD Isotopes, Montreal, CAN) and then with deuterated water, D_2O (99.9 atom% D, MSD Isotopes, Montreal, CAN). Separate portions of soil samples, mixtures of granular substances and other powders or fillers were extracted with separate aliquots of the two deuterated solvents.

Each of the sample/solvent combinations was initially homogenized using a vortex mixer for 30 sec and then sonicated, in ice, for 20 min. The extract (or leachate for bulky samples such as glassware) was then removed with a Pasteur pipette using a cotton plug as a filtering aid; other samples were centrifuged before solvent removal. Depending on the amount of solvent used and recovered, some samples were concentrated to smaller volumes by passing a stream of nitrogen over the liquid. The extracts/leachates were then distributed for analysis.

All sample weights (other than leachates of large bulky items), sample extraction volumes, recovery volumes, etc. were measured and are tabulated in Appendix B.

2.2 Analytical Procedures

2.2.1 Nuclear Magnetic Resonance Spectroscopy (NMR), Appendix C

Approximately 1 mL of each sample to be analyzed was transferred to a new, clean 5-mm o.d. Pyrex NMR tube (No. 507-PP, Wilmad Glass Co., Inc., Buena, NJ), and the top of the tube was capped and then wrapped with Parafilm (American Can Company). The NMR spectra were obtained at probe temperature (22 $\pm$ 1 °C) using either a Varian VXR-400S superconducting Fourier transform (FT) NMR spectrometer system or a Varian XL-200 superconducting FTNMR system (Varian Associates, Palo Alto, CA). ^1H NMR spectra were obtained on each $CDCl_3$ and D_2O extract and on the neat (without solvent) munition fills. ^{13}C NMR spectra were obtained on some of the more concentrated extracts to aid in the

interpretation of unknown compounds. All two-dimensional spectra (COSY and HETCOR) were accumulated using the standard Varian software.

^{1}H: The ^{1}H NMR spectra were obtained in double precision at either 400 MHz (VXR-400S) or 200 MHz (XL-200) using a sweep width of 20 ppm, a pulse angle of 90°, an acquisition time of at least 3.0 sec, and a pulse delay of 1.0 sec. At least 196 transients were accumulated for each spectrum so that compounds present as low as 20 µg/mL (20 ppm) would be detected. Samples in $CDCl_3$ were referenced to internal tetramethylsilane (TMS, δ0.0) using the $CHCl_3$ resonance from the solvent (δ7.26) as a secondary reference. Samples in D_2O were referenced to the HDO peak set at δ4.80. All quantitative data were obtained by digital integration of the peak areas of interest.

^{13}C: The ^{13}C NMR spectra were obtained in double precision at 100 MHz (VXR-400S) using a sweep width of 20K Hz, a pulse width of 12 µsec (90°), an acquisition time of 1.6 sec, a pulse delay of at least 2 sec, and full proton WALTZ decoupling. Spectra were run overnight or until the desired signal-to-noise ratio was achieved. The samples were referenced to internal TMS (δ0.0) using the $CDCl_3$ peak (δ77.0) as a secondary reference. Quantitative data were obtained by digital integration of the peak areas of interest.

All spectra were reviewed by two experienced NMR spectroscopists. Spectra were interpreted manually and assignments confirmed by comparison with spectra of authentic samples from the in-house database. For those compounds not in the database, structure elucidation by NMR was complemented by spectroscopic data from GC/MS, HPLC/IC and/or IR analyses.

2.2.2 Gas Chromatography/Mass Spectrometry (GC/MS)

2.2.2.1 Chemical Ionization (CI) Analyses, Appendix D

Samples were analyzed as required by GC/MS and/or direct inlet direct exposure probe (DEP) mass spectrometry using methane chemical ionization. Samples were analyzed either neat (without solvent) or as the $CDCl_3$ extract. The following conditions were used:

GC/MS/CI: Samples were analyzed with a Finnigan model 5100 GC/MS (Finnigan MAT, San Jose, CA) equipped with a 15 m x 0.25 mm DB-5 column with a 0.25 micron film thickness (J&W Scientific, Folsum, CA). The oven temperature was programmed from 60 to 270 °C at 10 °C/min with a 10 min hold at 270 °C. The injection port temperature was 200 °C and the GC/MS interface temperature was 230 °C. The CI reagent gas was methane with a source pressure of 0.6 Torr. The mass range was scanned from 60-

550 amu at 1 scan/sec. Neat samples were injected with a split ratio of 20:1. Extracts were injected in the splitless mode. The instrument was tuned daily with perfluorotributylamine according to the manufacturer's specifications. Solvent blanks were run periodically. Retention times are reported in seconds.

DEP/CI: Solid samples and extracts were analyzed as required by placing the sample on a Finnigan direct exposure probe tip. After evaporation of the solvent at room temperature, the probe was ramped from 0 to 1 amp at 0.2 amp/sec and the mass spectrum recorded under CI conditions. Mass to charge ratios (m/z) are reported for each compound identified.

2.2.2.2 Electron Impact (EI) Analyses, Appendix E

Samples were analyzed in the EI mode using a Hewlett-Packard 5890 Gas Chromatograph interfaced with a Hewlett-Packard MSD 5970A Mass Spectrometer (Hewlett Packard Company, San Fernando, CA). Vapor samples were collected in glass tubes (1/4" x 7") packed with 15 mg of Tenax GR (60/80 mesh). The tubes were desorbed at 190 °C with helium at a flow of 20 mL/min using a Tekmar 5010 Automatic Thermal Desorber (Tekmar Corporation, Cincinnati, OH) equipped with a liquid nitrogen cryofocus. The analytes were transferred at 200 °C into the HP 5890 Gas Chromatograph containing a 30 m DB-5 capillary column (0.25 mm ID; 0.25 μm film thickness) (Restek Corporation, Bellefonte, PA). The oven was held at 25 °C for 3 min and then ramped at 10 °C/min to 250 °C and held for 1 min. A head pressure of 12 psi and a helium flow of 0.8 mL/min directed the analytes into the source of the HP 5970A Mass Selective Detector (MSD). The mass spectra were recorded under EI conditions.

Liquid samples of 1 μL were injected directly into the HP 5890 Gas Chromatograph in the splitless mode and analyzed by the HP 5970 MSD using the same conditions as those for the vapor samples.

The HP 5970A MSD was set at 70 eV, a scan range of 30-400 amu at 1 scan/sec. The multiplier was set at 2400 V (autotune voltage) and an emission current of 0.3 mA. The instrument was tuned daily with perfluorobutylamine, and blanks were run after each analysis.

Retention times (in seconds) and m/z values are reported for each compound identified. The fragmentation patterns were referenced to the Wiley Library incorporated into the Chem Data Station and also to the Eight Peak Index of Mass Spectra (Mass Spectrometry Data Centre, AWRE, Aldermaston, Reading, RG7 4PR, UK).

2.2.2.3 Ion Trap Detector (ITD) Analyses, Appendix F

Samples were analyzed in the EI mode using a Perkin Elmer 8500 Gas Chromatograph (Perkin Elmer Corporation, Norwalk, CT) equipped with a Finnigan-Mat ITD-40 Ion Trap Detector having a sensitivity of 100 ppb (split injection). The column was a Restek RT-5 (5% diphenyl/95% dimethyl polysiloxane) (Restek Corporation, Bellefonte, PA), 30 m x 0.25 mm (1.0 μm film thickness) with a column flow of 1 mL/min of helium with a make-up of 0.7 mL/min. The flows were checked with a methanol injection at 60 °C. The baseline sensitivity was set to 7051 based on FC-43, and the multiplier voltage and emission current were 1700 V and 80 μA, respectively. The injection port was heated to 280 °C while the transfer line and manifold were heated to 260 °C. The column program was 60 °C for 5 min then a 10 °C/min ramp to 250 °C for 5 min. The split/splitless injection liner was run in the splitless mode with the split coming on at a range of 0.2-0.5 min with a split ratio between 10:1 and 100:1 depending on concentration. A 1 μL injection was performed on all samples.

2.2.2.4 Gas Chromatography/Flame Ionization Detector (GC/FID), Appendix F

Selected samples were analyzed by the flame ionization detector (FID) using the Hewlett Packard 5880 Gas Chromatograph (Hewlett Packard Company, San Fernando, CA). One μL liquid samples were injected on-column and separated on a Restek RT-35 column (35% diphenyl/65% polysiloxane, Restek Corporation, Bellefonte, PA) having a length of 30 m, an interior diameter of 0.33 mm and a film thickness of 1.0 μm. Column flow was 10 mL/min of helium, and the hydrogen and air flows were 30 mL/min and 300 mL/min, respectively, for the flame.

Samples were screened by the FID before being analyzed by the highly-sensitive Ion Trap. FID was used to estimate concentrations and to obtain better peak separation.

2.2.3 Fourier Transform Infrared Spectroscopy (FTIR), Appendix G

Approximately 0.5 μL of sample was placed on 5-10 mg of potassium bromide (spectroscopic grade, International Crystal Laboratories, Irvington, NJ). The mixture was ground in a crucible, and the solvent ($CDCl_3$) and water were pulled off under vacuum. The mixture was then placed in a press, and 10 tons of pressure were applied under vacuum for 5 min to form a clear, uniform, 13-mm diameter pellet. The pellet was placed in a

special holder that allowed the analysis results to be directly compared to results from liquid reference spectra.

The IR spectra were recorded using a Nicolet 60SX Fourier transform infrared (FTIR) spectrometer (Nicolet Instruments, Madison, WI) having a resolution set to 1 wavenumber and calibrated using a standard sample of polystyrene. Identification of compounds was based on comparisons with library reference spectra and by interpretation in conjunction with the other spectroscopic techniques (NMR and MS).

2.2.4 High Performance Liquid Chromatography/Ion Chromatography (HPLC/IC), Appendix H

2.2.4.1 Analysis of 2-Chlorovinylarsonous Acid (CVAA) and Phenyldichloroarsine (PD)

Reverse phase liquid chromatographic (RPLC) analyses were carried out using a Waters/Millipore Liquid Chromatograph (Waters/Millipore Corporation, Milford, MA) consisting of two Model 6000A pumps, a Model U6K injector, a Model 490 ultraviolet/visible (UV/VIS) detector, and a Model 840 data chromatography control station. Analytical separations were performed under the following conditions:

Sample Size:	100 µL
Column:	Ultrosphere ODS, 5 µm, 25 cm x 4.6 mm I.D. (Beckman Instruments, San Ramon, CA)
Mobile Phase:	10% Acetonitrile/90% Deionized distilled water
Flow Rate:	1.5 mL/min
Detection:	225 nm (0.05 AUFS)

The detectable limit for CVAA is 0.1 µg/mL (100 ppb) and for PD, 1 µg/mL (1 ppm).

2.2.4.2 Analysis of Thiodiglycol (TDG), Thiodiglycol Sulfoxide (TDGO), Thiodiglycol Sulfone (TDGO2), Arsenite Anion (AsO_2^-), and Arsenate Anion (AsO_4^{-2})

Chromatography was performed using a Dionex Model DX-300 Ion Chromatograph (Dionex Corporation, Sunnyvale, CA) equipped serially with both a variable wavelength UV detector and a Dionex Model PAD-1 pulsed amperometric detector operating with a platinum working electrode. Samples were introduced by an air-activated valve injector with a 200 µL sample loop. This system was connected to a Dionex Model 4270 Recorder-Integrator that measured UV and pulsed amperometric detector response in terms of peak area.

Ion-exclusion separations of TDG, TDGO, TDGO2, AsO_2^-, and AsO_4^{-2} were performed using the following chromatographic parameters:

Column:	Dionex HPICE-AS1
Column Temperature:	Ambient
Eluent:	100 mM Perchloric acid
Flow Rate:	1 mL/min

The UV detection of TDG, TDGO, AsO_2^-, and AsO_4^{-2} was carried out at the 210 nm wavelength (0.05 AUFS). Pulsed amperometric detection of TDGO2 was determined at an output range of 1000 nanoamps. The working electrode potentials used were E_1 = 0.30 V, E_2 = 1.25 V, E_3 = 0.10 V, where E_1 was the sampling voltage, and E_2 and E_3 were the cleaning and regenerating voltages, respectively. The duration of each potential was 60, 60, and 240 msec for E_1, E_2, and E_3.

The detectable limit for each of the compounds is 1 μg/mL (1 ppm).

2.2.4.3 Analysis of 2-Chlorovinylarsonic Acid (CVAOA), Chloride (Cl^-), Nitrate (NO_3^-), Sulfate (SO_4^{-2}) and Other Ions

Chromatography was performed using a Dionex Corporation Model DX-300 Ion Chromatograph equipped with both a variable wavelength UV and a 5 μL flow-through conductivity detector in series. Samples were introduced by an air-activated valve injector with a 25 μL sample loop. This system was connected to a Model 4270 Dionex Recorder-Integrator that measured conductivity detector response in terms of peak area. Ion exchange separations were performed under the following conditions:

Column:	Dionex HPIC-AS4A
Eluent:	0.75 mM sodium bicarbonate/2.2 mM sodium carbonate buffer
Flow Rate:	0.5 mL/min

Conductivity detection was determined at a sensitivity of 30 μS, full scale (Dionex-type anion fiber suppression).

The detectable limit for CVAOA is 0.1 μg/mL (100 ppb) and for the anions, 1 μg/mL (1 ppm).

2.2.5 Inductively Coupled Plasma (ICP) Emission Spectrophotometry

Elemental analyses were run on selected samples using a

Plasma II Inductively Coupled Plasma Emission Spectrophotometer (Perkin Elmer Corporation, Norwalk, CT).

One mL of each water extract to be analyzed was placed into the microwave cup, and 1 mL of concentrated nitric acid and 8 mL of water were added. The microwave (CEM, Matthewes, NC) was set to 100 psi and 165 °C for 10 min. The sample was then cooled and diluted to 25 mL before ICP or GFAA analysis.

Each $CDCl_3$ extract was placed into a test tube, and dry air was used to evaporate the solvent. One mL of concentrated nitric acid and 10 mL of water were added to the sample. The sample was then placed in a microwave cup and microwaved at 100 psi at ~165 °C for 10 min. The sample was then cooled and diluted to 25 mL with distilled water for analysis.

The ICP was set to screen for ~40 elements. The sample and a blank were run with no calibration, and the data are given in Emission Counts. If the sample and blank had roughly the same number of counts, it was concluded that the element was not present. A library search was performed on marginal elements to determine any interferences that might be occurring. A standard solution of the elements expected was run, then the blank sample. To determine the detection limits, the Lab 0 sample was analyzed and the standard run as a quality control (QC) check. The unknown sample was then run for the calibrated elements. The detection limits (in ppm) for the elements using a 1/25 dilution are given below:

Ag	(Silver)	0.025	Li	(Lithium)	0.022
Al	(Aluminum)	0.080	Mg	(Magnesium)	0.005
B	(Boron)	0.05	Mn	(Manganese)	0.01
Ba	(Barium)	0.025	Mo	(Molybdenum)	0.25
Be	(Beryllium)	0.002	Na	(Sodium)	0.20
Bi	(Bismuth)	0.080	Ni	(Nickel)	0.2
Ce	(Cerium)	0.25	P	(Phosphorus)	0.75
Cd	(Cadmium)	0.025	Pb	(Lead)	0.50
Co	(Cobalt)	0.050	S	(Sulfur)	1.3
Cr	(Chromium)	0.050	Sb	(Antimony)	1.5
Cu	(Copper)	0.022	Si	(Silicon)	0.075
Fe	(Iron)	0.025	Sn	(Tin)	1.0
Ga	(Gallium)	0.25	Sr	(Strontium)	0.01
Hg	(Mercury)	0.50	Ti	(Titanium)	0.050
I	(Iodine)	0.75	Tl	(Thallium)	1.00
K	(Potassium)	1.25	V	(Vanadium)	0.050
			Zn	(Zinc)	0.025

NOTE: In this report, all results for the elemental analyses are rounded off to the nearest whole number. These analyses were performed to gain an understanding of the chemical composition of

the samples and may not be the same as the values that would be obtained using Certified EPA methods of extraction and testing.

2.2.6 Graphite Furnace Atomic Absorption Spectrophotometry (GFAA)

Arsenic was determined using a Perkin Elmer 5100 Atomic Absorption Spectrophotometer equipped with a Zeeman 5100 graphite furnace module and an AS-60 autosampler (Perkin Elmer Corporation, Norwalk, CT). Instrument control and data collection were accomplished using an Epson Equity III+ personal computer running Perkin Elmer 5100 PC software. An electrodeless discharge lamp (EDL) at a wavelength of 193.7 nm was used for these determinations; the slit width was set at 0.7 nm.

Standard arsenic solutions were prepared by diluting a 1000 ppm commercial arsenic standard with distilled, deionized water. After the primary dilution was made, the AS-60 autosampler was used to prepare secondary dilutions. Prior to analysis, the samples were digested with nitric acid and hydrogen peroxide at 95 °C in order to convert organically bound arsenic to an inorganic form. A nickel nitrate solution (1005 ppm Ni in 2% nitric acid) was added to each sample to serve as a matrix modifier to prevent arsenic volatilization during sample heating. The sample size was 20 μL with 10 μL of the matrix modifier being taken at the same time as the sample.

A L'vov platform and pyrolytically coated graphite sample holders were used in the furnace; argon was used as the support gas. Furnace temperatures were as follows: drying, 130 °C for 50 sec; pretreatment, 1200 °C for 30 sec; cooling, 20 °C for 15 sec; and, atomization, 2225 °C for 5 sec. Each sample was followed by heating to 2600 °C to clean the platform for the next sample. The detection limit for arsenic determined in this manner is 0.005 ppm (5 ppb).

3. RESULTS AND DISCUSSION

A total of 98 items/samples were delivered to Bldg E3300 between 9 January 1993 and 2 February 1993 for chemical analysis by Research and Technology Directorate personnel. Copies of the chain of custody forms for each delivery are in Appendix I. The results of the analyses are summarized in Tables 1 through 9. "NR" indicates that the sample was "Not Run" by that particular technique, and "BDL" indicates that if any of the compound were present, it was below the detectable limit of the analytical technique used. The samples are grouped according to their delivery dates except for the samples from the intact munitions. The results for these samples are all presented in

Table 9. Details of the analyses are given below. NMR, DEP/CI, GC/MS/CI, GC/MS/EI, GC/FPD/FID/ITD, FTIR, and LC/IC spectra for each sample in which chemicals were identified are included in Appendices C through H. All original spectra for each of the samples from the Spring Valley site are filed within the respective analytical laboratories within the Research and Technology Directorate of ERDEC.

3.1 Pigs #1, #2, #3 and #5 (1400 Hrs, 9 Jan 93), Table 1

3.1.1 Pig #1

193-1a. The ^1H NMR spectrum of the $CDCl_3$ extract showed small amounts of aliphatic, aromatic and unsaturated organic compounds to be present. A doublet at δ6.30 (J = 15.9 Hz) is consistent for the presence of a trans-vinyl moiety; however, no single component in the extract could be identified. Only background hydrocarbon spectra were obtained by all of the GC/MS techniques. No resonances were observed by NMR in the D_2O extract; however, HPLC detected 20 ppm CVAA, a breakdown product of the CW agent Lewisite (L). It should be noted that the HPLC technique analyzes both L and CVAA as CVAA, so that one would not be able to distinguish between them by HPLC, alone. However, no L, itself, was observed in the $CDCl_3$ extract by GC/MS, and the ease with which L degrades would make it highly unlikely for the intact agent to survive on broken glass buried in the ground for over 70 years.[1] HPLC/IC was also used to analyze for PD, arsenite and arsenate anion, TDG, TDGO and TDGO2. None were detected.

193-1b. The ^1H NMR spectrum of the $CDCl_3$ extract showed the usual hydrocarbon background (δ0.9 and 1.25) as well as several peaks in the δ6.3-7.6 region indicative of trans-vinyl moieties similar to Lewisite. Three compounds were observed:

Area %		
72%	(a)	δ6.98, δ7.56 (J = 13.6 Hz) (Cl)CH=CH[As(O)]~
18%	(b)	δ6.38, δ6.48 (J = 14.5 Hz) L-3
9%	(c)	δ6.72, δ6.87 (J = 14.5 Hz) L-2

Based on literature values and spectra of authentic L, L-2, and L-3 in $CDCl_3$, it was concluded that no Lewisite, itself, was present. The compound at δ6.72 and δ6.87 was identified as bis(2-chlorovinyl)chloroarsine, L-2, and the compound at δ6.38

Table 1. Summary of Analytical Results for Pigs #1, #2, #3 and #5 Delivered by TEU to Bldg E3300 at 1400 Hrs on 9 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #1 OTH-193-1a	Broken plate, glass	Aliphatic, aromatic hydrocarbons; t-Vinyl compound	Background	Background	NR	CVAA: 20 ppm	NR
Pig #1 OTH-193-1b	Broken glass, jars & carboys	L-2, L-3, CVAOA, L analogs/degradation products	L-2, L-3	Background	NR	CVAA: 40 ppm CVAOA: 20 ppm	NR
Pig #2 OTH-193-2a	Glass test tube w/ dark green oily substance	DM, Diphenylamine	DM, Diphenylamine	Diphenylamine Diphenyl sulfide	NR	NR	As: >5000 ppm
Pig #2 OTH-193-2b	Gray/tan fine powder	Background	Nothing detected	Diphenyl sulfide	NR	Background	As: BDL
Pig #2 OTH-193-2c	Gray fine powder	Background	Nothing detected	Background	NR	Background	As: BDL
Pig #2 OTH-193-2d	Dark grainy greenish/brown substance	Background	Nothing detected	Background	NR	Background	As: BDL
Pig #3 OTH-193-3a	Rusted conical fuse w/dark brown viscous liquid	CN, Acetophenone, CN degradation products	CN, Acetophenone, Benzoic acid, Benzaldehyde, Diphenyl sulfide, CN degradation products	CN, Acetophenone, Benzoic acid, Diphenyl sulfide	NR	NR	NR
Pig #3 OTH-193-3b	Leatherman's tool w/dark brown substance	CN, Acetophenone, CN degradation products	CN	CN, Acetophenone	NR	NR	NR
Pig #3 OTH-193-3c	Plastic vial w/cap & dark brown substance	CN, Acetophenone, CN degradation products	CN, Benzoic acid, Acetophenone, Diphenyl sulfide, Benzaldehyde, CN degradation products	CN, Benzaldehyde, Acetophenone	NR	Background	NR

Table 1 (Continued). Summary of Analytical Results for Pigs #1, #2, #3 and #5 Delivered by TEU to Bldg E3300 at 1400 Hrs on 9 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #5 OTH-193-5a	Brownish soil	Background	Nothing detected	Background	NR	Background	NR
Pig #5 OTH-193-5b	Soil adhering to and with pink waxy fiber	Polystyrene	Polystyrene	Background	NR	Background	NR
Pig #5 OTH-193-5c	Tannish sandy soil w/o pink waxy material	Background	Nothing detected	Background	NR	NR	NR

Table 2. Summary of Analytical Results for Pigs #4 and #6 Delivered by TEU to Bldg E3300 at 2015 Hrs on 14 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #4 OTH-393-4	Broken glass/ ceramic/dirt	TNT, Long chain hydrocarbons	TNT, Unknowns with m/z: 256, 282, and 284	TNT, Octanoic acid, Nonanoic acid	TNT, hydrocarbons > C10	Background	NR
Pig #6 OTH-393-6a	Hard, black solid, sweet smell from 75mm round #43 (soil)	Aliphatic & aromatic hydrocarbons, Acetophenone, (2-Oxo-2-phenyl)ethyl benzoate, metals indicated	Acetophenone, Diphenyl sulfide, Benzoic Acid, (2-Oxo-2-phenyl)-ethyl benzoate, Phenol, Diphenyl sulfoxide, Diphenyl sulfone, Benzaldehyde, CN	Acetophenone; Diphenyl sulfide; 1,2-Diphenyl ethanone	Acetophenone, CN, Benzoic acid, Diphenyl sulfide	Background	NR
Pig #6 OTH-393-6b	Solid whitish/ tan substance, clay-like	Nothing detected	Background	NR	NR	Background	NR
Pig #6 OTH-393-6c	Black tar-like substance from bomb windshield	Background	Elemental sulfur	NR	NR	Background	Al: 1910 ppm Ca: 476 ppm Co: 141 ppm Fe: 46000 ppm K: 5380 ppm Mg: 3040 ppm Mn: 173 ppm Pb: 2080 ppm Zn: 1360 ppm
Pig #6 OTH-393-6d	Cotton fiber type wadding	Aliphatic hydrocarbons, metals indicated	Hydrocarbons, metal isotopes	NR	NR	Background	Al: 1580 ppm Ca: 952 ppm Co: 30 ppm Fe: 23900 ppm K: 7300 ppm Mg: 3250 ppm Mn: 397 ppm Pb: 1250 ppm Zn: 5060 ppm
Pig #6 OTH-393-6e	Broken glass/ rock/ceramic	Background	Styrene	NR	NR	CVAA: 10 ppm	NR

Table 3. Summary of Analytical Results for Pigs #9 and #10 Delivered by TEU to Bldg E3300 at 1240 Hrs on 15 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #9-1 OTH-293-9a	Green glass	TNT, Polyether unknown	TNT, Unknowns with m/z: 336, 354, 372, 390 and 408	NR	Background	Background	NR
Pig #9-1 OTH-293-9b	Clear glass plus dirt	TNT	TNT, Arsenic acids	NR	NR	Background	NR
Pig #9-1 OTH-293-9c	Clear glass plus dirt	Unknown L analog/ degradation product, L-3, TNT	L-3	L-3	NR	Background	NR
Pig #9-1 OTH-293-9d	Thick clear and green glass & black dirt	Several aromatic compounds (monosubstituted benzenes)	Triphenyl arsine, Diphenyl sulfide, Other aromatic arsenic compounds	NR	NR	CVAA: 2 ppm	NR
Pig #10-1 OTH-293-10a	Brown waxy material	Tetryl	Tetryl	Background	NR	Background	NR
Pig #10-1 OTH-293-10b	Fine yellow powder	Tetryl, TNT	Tetryl, TNT	Background	NR	Background	NR
Pig #10-1 OTH-293-10c	White rubber tubing w/coating of dirt	L-3, Aliphatic hydrocarbons	L-3	L-3	NR	CVAA: 1 ppm	Al: 1100 ppm As: 1520 ppm Ba: 256 ppm Fe: 403 ppm Mg: 536 ppm Zn: 1880 ppm
Pig #10-1 OTH-293-10d	Black solids & soil	Background	Background	Background	NR	CVAA: 1 ppm	Al: 1680 ppm As: BDL Ca: 404 ppm Fe: 1210 ppm K : 274 ppm Mg: 1440 ppm
Pig #10-1 OTH-293-10e	Black solid	Background	Background	NR	NR	Background	NR
Pig #10-1 OTH-293-10f1	Solid (light sand)	Background	Background	Background	NR	Background	Al: 1470 ppm

Table 3 (Continued). Summary of Analytical Results for Pigs #9 and #10 Delivered by TEU to Bldg E3300 at 1240 Hrs on 15 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #10-1 OTH-293-10f2	Solid (dark sand)	Background	Background	Background	NR	CVAA: 1 ppm	Al: 1320 ppm
Pig #10-1 OTH-293-10g	Small rocks w/ soil	TNT, metals indicated	Background	NR	NR	Background	NR

Table 4. Summary of Analytical Results for the Second Pig #9 Delivered by TEU to Bldg E3300 at 1430 Hrs on 21 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #9-2 OTH-893-1 SOL 40	Broken glass bottle w/charred purple substance	Background	Background	Background	NR	Background	Background
Pig #9-2 OTH-893-2 SOL 43	Broken glass from various pit depths	Background	Background	Background	NR	Background	NR
Pig #9-2 OTH-893-3 SOL 44	Glass/ceramic pieces (55" deep) & dirt	Background	Background	Background	NR	Background	NR
Pig #9-2 OTH-893-4 SOL 45	Rubber stopper w/glass tube (55" deep)	Aliphatic hydrocarbons	Background	NR	NR	Background	Ca: 224 ppm Mg: 143 ppm Zn: 184 ppm

Table 5. Summary of Analytical Results for the Second Pig #10 Delivered by TEU to Bldg E3300 at 1530 Hrs on 22 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #10-2 OTH-993-1	Bright orange dirt around 75 mm round (50" deep)	Background	Background	Background	NR	Background	Al: 20500 ppm Ba: 180 ppm Ca: 90 ppm Cd: 19 ppm Cu: 114 ppm Fe: 96200 ppm K: 11200 ppm Mg: 92 ppm Mn: 137 ppm Na: 3540 ppm Pb: 3520 ppm S: 6360 ppm V: 32 ppm Zn: 32 ppm
Pig #10-2 OTH-993-2	White/brown powder from Livens Item #59	Background	Background	Background	NR	Background	Al: 7380 ppm Ba: 41 ppm Ca: 3033 ppm Cd: 58 ppm Cu: 47 ppm Fe: 5800 ppm K: 4200 ppm Mg: 71300 ppm Mn: 148 ppm Na: 20100 ppm V: 8 ppm Zn: 193000 ppm
Pig #10-2 OTH-993-3	Yellow/orange dirt at end of 2 suspect Livens	Background	Background	Background	NR	Background	Al: 9280 ppm Ba: 141 ppm Ca: 148 ppm Cd: 14 ppm Cu: 22 ppm Fe: 41900 ppm K: 12800 ppm Mg: 252 ppm Mn: 217 ppm Na: 8810 ppm V: 24 ppm

Table 5 (Continued). Summary of Analytical Results for the Second Pig #10 Delivered by TEU to Bldg E3300 at 1530 on 22 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #10-2 OTH-993-4	Black square of unknown material	Aliphatic hydrocarbons, Smaller amount of aromatic hydrocarbons	Hydrocarbons up to mass 300	Background	NR	Background	NR
Pig #10-2 OTH-993-5	Probe from flash channel	Background	Elemental sulfur (S8)	Background	NR	Background	Cu: 44 ppm K: 93 ppm Mg: 62 ppm Zn: 17 ppm
Pig #10-2 OTH-993-6	Green stained large nail	Background	Background	Background	NR	Background	NR
Pig #10-2 OTH-993-7	Small tube	Background, metals indicated	Background	Background	NR	Background	Al: 383 ppm Ca: 23 ppm Fe: 188 ppm Mg: 84 ppm

Table 6. Summary of Analytical Results for Pigs #11, #12, #13, #14 and #15 Delivered by TEU to Bldg E3300 at 1000 Hrs on 25 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #11 OTH-1093-1a SOL 56	Glass adapter w/ copper tubes	Background	Background	Background	NR	Background	NR
Pig #11 OTH-1093-1b SOL 62	Two clear glass bottles	Background	Background	Background	NR	Background	NR
Pig #11 OTH-1093-1c SOL 59	Clear glass bottle	Background	Background	Background	NR	Background	NR
Pig #11 OTH-1093-1d	White ceramic jar w/flaky solid	Long chain aliphatic hydrocarbons; Smaller amounts of aromatic hydrocarbons; cis-1,4-Polyisoprene	Hydrocarbons up to mass 500	Background	NR	Background	NR
Pig #12 OTH-1093-2a SOL 63	Amber glass pieces	Background	Background	Background	NR	Background	As: BDL
Pig #12 OTH-1093-2b SOL 64	Clear glass pieces	Background	Background	Background	NR	Background	NR
Pig #13 OTH-1093-3a SOL 71	Cotton fiber wadding	Background	Background	Background	NR	Background	NR
Pig #13 OTH-1093-3b SOL 73	Cloth from around Item #98	Background	Background	Background	NR	Background	NR
Pig #13 OTH-1093-3c SOL 66	Stoppers w/glass tubing	Aliphatic hydrocarbons; cis-1,4-Polyisoprene	Hydrocarbons; Unknowns with m/z: 101 & 83	Background	Hydrocarbons	Background	NR
Pig #13 OTH-1093-3d SOL 74	Wooden pieces	Background, metals indicated	Background	Background	NR	Background	NR

Table 6 (Continued). Summary of Analytical Results for Pigs #11, #12, #13, #14 and #15 Delivered by TEU to Bldg E3300 at 1000 Hrs on 25 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #13 OTH-1093-3e SOL 68	Large stopper w/one hole (not rubber)	Aliphatic hydrocarbons; cis-1,4-Polyisoprene	Hydrocarbons up to m/z 250	Aliphatic hydrocarbons	NR	Background	NR
Pig #13 OTH-1093-3f SOL 67	Copper piping	Background	Background	NR	NR	Background	NR
Pig #13 OTH-1093-3g SOL 69	Clear broken glass w/black tar substance	Background, metals indicated	Background	NR	NR	Background	NR
Pig #14 OTH-1093-4a SOL 60	Green solid from lead lined 75 mm Item #96	Background	Copper sulfate, possibly	Background	NR	Background	NR
Pig #14 OTH-1093-4b SOL 58	2-holed rubber stopper w/glass tubing	Background	Background	Background	NR	Background	NR
Pig #14 OTH-1093-4c SOL 53	Glass lab bottle	Background	Background	Background	NR	Background	NR
Pig #14 OTH-1093-4d SOL 70	Black/white granular solid	Background	Background	Background	NR	Background	NR
Pig #14 OTH-1093-4e SOL 57	Multiple glass pieces	Background	Background	Background	NR	Background	NR
Pig #14 OTH-1093-4f SOL 75	Metal section from wall of spray tank Item #98	Background	Background	NR	NR	Background	NR
Pig #14 OTH-1093-4g SOL 76	Metallic tubing/fitting	Aliphatic hydrocarbons	Hydrocarbons	Background	Hydrocarbons	Background	NR
Pig #15 OTH-1093-5a SOL 72	White flaky material from ceramic jar	Background	Background	Background	NR	Background	NR

Table 6 (Continued). Summary of Analytical Results for Pigs #11, #12, #13, #14 and #15 Delivered by TEU to Bldg E3300 at 1000 on 25 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC IC	ICP GFAA
Pig #15 OTH-1093-5b SOL 65	Broken glass tubing	Background	Background	Background	NR	Background	NR
Pig #15 OTH-1093-5c SOL 55	Erlenmeyer flask	Background	Background	Background	NR	NR	NR

Table 7. Summary of Analytical Results for Pigs #17, #18, #19, #21 and #22 Delivered by TEU to Bldg E3300 at 1010 Hrs on 27 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #21 OTH-1193-1 LIQ 5	Liquid from inside 75 mm, #126	Thiodiglycol; Thiodiglycol polymers; 1,4-Dithiane; 1,4-Thioxane	Thiodiglycol; Ether/sulfide polymers of thiodiglycol; 1,4-Thioxane; 1,4-Dithiane; Ethyl 2-hydroxyethyl sulfide;1,4-Dioxa-7-thionane	Thiodiglycol; Ethyl 2-hydroxy ethyl sulfide; 1,4-Dithiane; 1,4-Thioxane	NR	TDG: 200 ppm; TDGO: 50 ppm; TDGO2: trace; Chloride Ion: 2 mg/mL	Ca: 69 ppm Cd: 35 ppm Cu: 1800 ppm Fe: 135000 ppm Mg: 50 ppm Mn: 455 ppm Na: 75 ppm S: 48900 ppm Zn: 82 ppm
Pig #18 OTH-1193-2a SOL 78	Glass pipette in glass jar top, Item #1	Background	Background	Background	NR	Background	NR
Pig #18 OTH-1193-2b SOL 81	Broken glass test tubes, Item #2	Background	Background	Background	NR	Background	NR
Pig #18 OTH-1193-2c SOL 87	White powder from 75 mm, Item #3	Background	Arsenic; Arsenic (III) oxide	NR	NR	Background	NR
Pig #18 OTH-1193-2d SOL 86	Clear glass pieces w/black tar, Item #4	Background	Background	NR	NR	CVAA: 10 ppm PD: 20 ppm	NR
Pig #18 OTH-1193-2e SOL 82	Broken small glass bottle w/tube Item #5	Cis(1,4-polyisoprene); Aliphatic hydrocarbons	Hydrocarbons	NR	Hydrocarbons	Background	NR
Pig #18 OTH-1193-2f SOL 79	Glass stopper, Item #6	Background	Background	NR	NR	Background	NR
Pig #18 OTH-1193-2g SOL 77	Glass bottle from trap w/copper fitting	Background	Background	NR	NR	Background	NR
Pig #18 OTH-1193-2h SOL 80	Metal handles, Item #8	Background	Background	NR	NR	Background	NR

Table 7 (Continued). Summary of Analytical Results for Pigs #17, #18, #19, #21 and #22 Delivered by TEU to Bldg E3300 at 1010 on 27 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #18 OTH-1193-2i SOL 84	Cloth fiber pieces, Item #9	Background	Background	NR	NR	Background	NR
Pig #19 OTH-1193-3a SOL 91	75mm base w/ black substance lining interior, Item #1	Background	Background	Background	NR	Chloride Ion: 0.5 mg/mL; Nitrate Ion: 1.0 mg/mL	NR
Pig #19 OTH-1193-3b SOL 90	Black burnt substance from 2 qt can, Item #2	Background	Background	Background	NR	Background	NR
Pig #19 OTH-1193-3c SOL 92	White granular substance from around glassware, Item #3	Background	Background	Background	NR	Background	NR
Pig #19 OTH-1193-3d SOL 93	Tan solid from base of 75 mm, Item #4	Background	Background	Background	NR	Background	NR
Pig #17 OTH-1193-4a SOL 83	Various clear bottle pieces	Background	Background	Background	NR	Background	NR
Pig #17 OTH-1193-4b	Various amber jar broken pieces	Background	Background	Background	NR	Background	NR
Pig #22 OTH-1193-5a SOL 89	Clay-like filler from 75 mm fuse, #114, Item #1	Background	Red phosphorus	Background	NR	Background	P: 11.3%
Pig #22 OTH-1193-5b SOL 94	White powder from Item #75, unfired projectile	Background	Arsenic, Arsenic (III) oxide, Related arsenic acids and oxides	NR	NR	Background	NR
Pig #22 OTH-1193-5c SOL 96	Tubing, Item #3	L-3, Lewisite analogs/degradation products, Aliphatic hydrocarbons	L-3, Unknown chlorinated compound	L-3	NR	Background	NR

Table 7 (Continued). Summary of Analytical Results for Pigs #17, #18, #19, #21 and #22 Delivered by TEU to Bldg E3300 at 1010 Hrs on 27 Jan 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #22 OTH-1193-5d SOL 95	Part of Item #118, smoke can, Item #4	Background, metals indicated	Nothing detected	NR	NR	Background	NR

Table 8. Summary of Analytical Results for Pig #23 Delivered by TEU to Bldg E3300 on 1 Feb 1993.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
Pig #23 OTH-1393-1a SOL 97	Broken glass bottle w/black substance	Background	Nothing detected	Background	NR	Background	NR
Pig #23 OTH-1393-1b SOL 98	Brown filler from inside 75mm #141 + lead ball	Degradation products of CN, Small amounts of CN & acetophenone	Acetophenone, Benzoic acid, Diphenyl sulfide, Degradation products of CN	Acetophenone, CN, Benzoic acid, Degradation products of CN, Diphenyl disulfide	Acetophenone, Diphenyl sulfide, Benzoic acid	Chloride Ion: 1 mg/mL	Fe: 810000 ppm Mn: 6700 ppm
Pig #23 OTH-1393-1c SOL 99	Lead balls & filler material from 75mm #137	Aliphatic (branched) & aromatic hydrocarbons	Hydrocarbons up to mass 300	Background	NR	Background	NR
Pig #23 OTH-1393-1d	Lead balls from inside 75mm #100	Degradation products of CN	Degradation products of CN	Acetophenone	NR	Background	NR

Table 9. Summary of Analytical Results for the Munitions Sampled by CTF Personnel.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
18 Jan 93 1030 hrs OTH-493-1c	Fuming brown viscous liquid from Item #71	Only H+ observed	Sulfuric Acid	NR	NR	NR	S: 32.2%
18 Jan 93 1515 hrs OTH-493-2c	Fuming dark brown liquid from Item #82	Only H+ observed	NR	NR	NR	NR	S: 32.2%
19 Jan 93 1250 hrs OTH-593-1c	Bluish-gray liquid from Livens Projectile #65	Water	Traces of xylene, trimethylbenzene, naphthalene	Background	NR	Background	Ba: 205 ppm Ca: 31100 ppm K: 1198 ppm Mg: 72100 ppm Na: 1160 ppm
21 Jan 1993 1300 hrs Pig #8 OTH-793-2 SOL 42	3016-1 pipe w/ capped ends	Nitrobenzene, Small amounts of dinitrobenzene and trinitrobenzene, Excess H+	Nitrobenzene, Small amounts of dinitrobenzenes	Nitrobenzene, Dinitrobenzenes, Carbon tetrachloride, Nitrobenzonitrile	NR	NR	Ca: 2 ppm Fe: 532 ppm Mg: 4 ppm Mn: 1 ppm Na: 62 ppm
1 Feb 93 OTH-1293-1c	Bluish/gray liquid from Livens Projectile #87	Water, metals indicated	Background	Background	NR	Chloride Ion: 50 mg/mL	Al: 16900 ppm Ba: 16100 ppm Ca: 72700 ppm Fe: 346 ppm K: 9020 ppm Mg: 159000 ppm Mn: 489 ppm Na: 144000 ppm
2 Feb 93 OTH-1493-1c	Gray liquid w/ dark sediment from 4.7" Livens, Item #67	Water with small amounts of thiodiglycol; 1,4-dithiane; 1,4-thioxane	Trace thiodiglycol	Background	NR	TDG: 100 ppm; Nitrate Ion: 2 mg/mL	Al: 83 ppm As: 13 ppm Ca: 130 ppm Cu: 1500 ppm Fe: 98000 ppm Mg: 70 ppm Mn: 610 ppm Na: 570 ppm S: 3300 ppm

Table 9 (Continued). Summary of Analytical Results for the Munitions Sampled by CTF Personnel.

Sample Number	Description	NMR	GC/MS/CI DEP/CI	GC/MS/EI	FTIR, GC/FID GC/ITD	HPLC/IC	ICP GFAA
2 Feb 93 OTH-1493-2	Fibrous solids from 75 mm, Item #147	Aliphatic hydrocarbons	Straight chain (C9-C16) aliphatic hydrocarbons	Hydrocarbons (C10-C18)	NR	Nitrate Ion: 2 mg/mL	As: 6 ppm Ca: 1940 ppm Cu: 110 ppm Fe: 15000 ppm Mg: 1300 ppm Mn: 93 ppm Na: 49500 ppm
2 Feb 93 OTH-1593-1c	Dark viscous liquid from 75 mm Item #90	HD (>60%), HD degradation products, metals indicated	HD, numerous HD degradation products	HD; Thiodiglycol dimer; 1,4-Thioxane; 1,4-Dithiane	NR	Thiodiglycol: 1 mg/mL	NR
2 Feb 93 OTH-1693-1c	Dark mobile liquid from 75 mm Item #113	Water, Thiodiglycol, HD degradation products, metals indicated	Thiodiglycol; Thiodiglycol polymers; 1,4-Dithiane; 1,4-Thioxane	Thiodiglycol; 1,4-Dithiane; 1,4-Thioxane	NR	Chloride Ion: 50 mg/mL	Ca: 384 ppm Cd: 17 ppm Co: 26 ppm Fe: 67300 ppm Mg: 423 ppm Mn: 696 ppm
2 Feb 93 OTH-1793-1C	Gray liquid w/ dark sediment from 75 mm Item #142	Nothing detected	Nothing detected	NR	NR	Chloride Ion: 50 mg/mL; Sulfate Ion: 1 mg/mL	NR

and δ6.48 was identified as tris(2-chlorovinyl)arsine, L-3. The major component (δ6.98 and δ7.56) was not conclusively identified but the low-field chemical shift values for the two protons indicate that the arsenic in this compound is probably oxidized.The presence of L-2 (361 sec; m/z: 197/199, 171/173) and L-3 (463 sec; m/z: 197/199, 259/261) were confirmed by both GC/MS/CI and DEP/CI in the $CDCl_3$ extract.

The ^1H NMR spectrum of the D_2O extract contained resonances for a trans-vinyl moiety also (δ6.77 and 7.30, J~15 Hz). These were attributed to (2-chlorovinyl)arsonic acid (CVAOA) based on a comparison to the spectrum of an authentic sample. The presence of CVAOA was confirmed by HPLC/IC which found 20 ppm CVAOA and 40 ppm CVAA in the sample. HPLC/IC was also used to analyze for PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

3.1.2 Pig #2

193-2a: The ^1H NMR spectrum of the $CDCl_3$ extract showed the following:

Mole %

67% (a) Diphenylamine [H2,2′: δ7.09 (4H, "doublet"); H3,3′: δ7.29 (4H, "triplet"); H4,4′: δ6.95 (2H, "triplet")]

27% (b) Adamsite (DM) [H1,9: δ7.89 (2H, doublet of doublets, J = 1.3, 7.6 Hz); H2,8: δ7.07 (2H, doublet of triplets, J = 0.9, 7.5 Hz); H3,7: δ7.38 (2H, multiplet, J = 1.6, 7.4, 8.4 Hz); H4,6: δ6.94 (2H, doublet, J = N.O.)]

6% (c) Other aromatic compounds

There was a significant amount of green solid which did not dissolve in the $CDCl_3$ solution. This may be excess DM since the DM was observed to have a limited solubility in the $CDCl_3$. Even so, the excellent signal-to-noise obtained by NMR would indicate that the concentrations of these compounds were in the mg/mL range.

The presence of the DM and its degradation product diphenylamine were confirmed by ^{13}C NMR (DM: C9a,10a: δ120.5; C1,9: δ132.5; C2,8: δ120.9; C3,7: δ135.4; C4,6: δ116.4; C4a,5a: 139.8; and diphenylamine: C1,1′: 143.1; C2,2′: 117.7; C3,3′: 129.3; C4,4′: 120.9). The assignments were confirmed by two-dimensional NMR experiments (COSY and HETCOR) and by comparison with authentic samples.

A monosubstituted benzene (δ7.21, doublet; δ7.38, triplet) was the only compound observed by NMR in the D_2O extract. It may be some diphenylamine present as a salt or it may be another water soluble product from the degradation of DM. No identification was made.

The presence of DM and diphenylamine was confirmed by DEP/CI (DM, m/z: 278, 242; diphenylamine, m/z: 170); they were found in approximately equal amounts. The presence of DM was also confirmed by the large amount of arsenic found in the sample (>5000 ppm), and the presence of diphenylamine (730 sec; m/z: 169, 168, 141, 115, 83 and 77) was also confirmed by GC/MS/EI which also detected diphenyl sulfide (1071 sec; m/z: 186, 151, 109, 77 and 51).

193-2b: No resonances were observed by NMR in the D_2O extract. Background hydrocarbons were observed in the $CDCl_3$ extract along with very small amounts of aliphatic and aromatic compounds for which no identification was possible. No compounds were detected by GC/MS/CI whereas a small amount of diphenyl sulfide was observed by GC/MS/EI (1072 sec; m/z: 186, 152 and 51). HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected. GFAA was run for arsenic which was found to be below the detectable limit.

193-2c and 193-2d: No resonances were observed in the D_2O extracts of either sample by NMR, and only hydrocarbon background was observed in the $CDCl_3$ extracts. Nothing was detected in either sample by DEP/CI or GC/MS/EI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were found in either sample. GFAA was run for arsenic which was below the detectable limit in both cases.

3.1.3 Pig #3

193-3a: The ^1H NMR spectrum of the $CDCl_3$ extract indicates that the main component is chloroacetophenone, CN (CH_2: δ4.72, singlet;H2,6: δ7.96, doublet; H3,5: δ7.50, triplet; H4: δ7.60). The CN was identified by comparison with the spectrum of an authentic sample and confirmed by ^{13}C NMR [CH_2: δ46.0; C1: δ134.2; C2,3,5,6: δ128.5 and 128.9; C4: δ134.0; C(O): δ191.0]. Acetophenone was also detected as evidenced by the singlet at δ2.61 in the ^1H spectrum. The sample also appears to contain other degradation products of CN based on the presence of other peaks in the aromatic region (δ7.0-8.2). The excellent signal-to-noise of the NMR spectra indicates that these compounds are present in the mg/mL range.

GC/MS/CI found the following compounds:

Area %		
86.0%	(a)	CN (343 sec; m/z: 155/157, 141, 119, 105, 77)
9.4%	(b)	Acetophenone (180 sec; m/z: 121, 105)
3.4%	(c)	Benzoic acid (268 sec; m/z: 123, 105)
1.3%	(d)	Benzaldehyde (122 sec; m/z: 107)

In addition, the DEP/CI spectrum suggests the possible presence of small amounts of higher molecular weight CN degradation products and diphenyl sulfide (m/z: 187).

GC/MS/EI also detected acetophenone (590 sec; m/z: 120, 105, 77, 51 and 50), benzoic acid (779 sec; m/z: 122, 105, 77, 51 and 50), CN (850 sec; m/z: 154, 105, 77 and 51), and diphenyl sulfide (1085 sec; m/z: 186, 152, 77 and 51).

193-3b: The ^{1}H and ^{13}C NMR spectra of the $CDCl_3$ extract are identical to the spectra obtained for sample 193-3a, above, except this sample does not appear to be as concentrated. The sample is a mixture of CN (^{1}H: δ4.72, 7.97, 7.50 and 7.63; ^{13}C: δ46.0, 128.6, 128.9, 134.0, and 134.2), degradation products of CN, and a small amount of acetophenone (^{1}H: δ2.61). The presence of CN (348 sec; m/z: 155,157, 141, 119, 105, 77) was confirmed by GC/MS/CI, however, DEP/CI did not show the higher molecular weight compounds observed in sample 193-3a. GC/MS/EI also detected CN (823 sec; m/z: 154, 105, 77 and 51) and acetophenone (589 sec; m/z: 120, 105, 77, 51, 50).

193-3c: The ^{1}H NMR spectrum of the $CDCl_3$ extract is identical to the spectra of 193-3a and 193-3b, above. The main single component is CN (δ4.72, 7.97, 7.50 and 7.62); some acetophenone was also observed (δ2.62). Other CN degradation products are present as evidenced by the additional peaks and broadening in the aromatic region. The ^{1}H NMR spectrum of the D_2O extract showed 72% CN (δ4.80, 7.68, 7.25 and 7.40), 5% acetophenone (δ2.35), and 24% other degradation products.

The DEP/CI spectrum was similar to that of sample 193-3a, and the following compounds were detected by GC/MS/CI:

Area %		
80.7%	(a)	CN (346 sec; m/z: 155/157, 141, 119, 105, 77)
11.8%	(b)	Benzoic acid (276 sec; m/z: 123, 105)
5.6%	(c)	Acetophenone (176 sec; m/z: 121, 105)

1.3% (d) Diphenyl sulfide (580 sec; m/z: 187)
0.6% (e) Benzaldehyde (121 sec; m/z: 107)

GC/MS/EI confirmed the presence of benzaldehyde (683 sec; m/z: 106, 105, 77, 52, 51 and 50), acetophenone (815 sec; m/z: 120, 105, 77, 51 and 50), and CN (1053 sec; m/z: 154, 105, 77, 51 and 50). HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

3.1.4 Pig #5

193-5a: No resonances were observed by NMR in the D_2O extract, and the $CDCl_3$ extract showed only background hydrocarbons. Nothing was detected by GC/MS/CI or GC/MS/EI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

193-5b: No resonances were observed by NMR in the D_2O extract. Both the ^1H (CH_2: δ1.44; CH: δ1.85; H2,6: δ6.54; H3,4,5: δ7.08) and ^{13}C (CH_2: δ40.3; CH: δ41-45; C1: δ147.2; C2,3,4,5,6: δ125.6, 127.6 and 127.9) NMR spectra of the $CDCl_3$ extract were consistent for the presence of isotactic polystyrene probably from the pink waxy (construction?) material in the sample.

Nothing was detected by GC/MS/CI or GC/MS/EI in this sample. The DEP/CI spectrum was consistent with polystyrene (m/z: 91, 105, 117, 131, 195 and 235). HPLC/IC showed no CVAA, PD, arsenite or arsenate anion, TDG, TDGO, or TDGO2.

193-5c: No resonances were observed by NMR in the D_2O extract, and the $CDCl_3$ extract showed only background hydrocarbons. Nothing was detected by GC/MS/CI or GC/MS/EI.

3.2 Pigs #4 and #6 (2015 Hrs, 14 Jan 93), Table 2

3.2.1 Pig #4

393-4: The ^1H NMR spectrum of the $CDCl_3$ extract showed the presence of a large amount of long chain aliphatic hydrocarbons (δCH_3: 0.88, triplet; δCH_2: 1.25, 1.63, 2.02 and 2.35, triplet). The spectrum is consistent for the $CH_3(CH_2)_n$- moiety with n>10 based on the integration. A smaller resonance (δ5.35) was observed in the unsaturated region (CH=CH) of the spectrum indicating the presence of an alkene. The singlets at δ2.72 and 8.84 were attributed to the methyl and aromatic protons, respectively, of trinitrotoluene (TNT). This assignment was based on comparison with the spectrum of an authentic sample of TNT and on the fact that TNT was also found in other samples

from the Spring Valley site (see below). The spectrum of the D_2O extract showed several broad resonances in the CH_3/CH_2 region (δ1.30, 1.57 and 2.17) which would be consistent with the long chain acids observed in this sample by GC/MS/EI.

The presence of TNT was confirmed by GC/MS/CI (639 sec; m/z: 228), DEP/CI and GC/MS/EI (1126 sec;m/z: 210, 89 and 63). Three additional small peaks were observed by GC/MS/CI with molecular weights of 256, 282 and 284; no identification was possible. GC/MS/EI also detected octanoic acid (631 sec; m/z: 101, 85, 73 and 60), and nonanoic acid (721 sec; m/z: 158, 129, 115, 73 and 60). The TNT was also confirmed by GC/ITD and determined to be 150 μg/mL by GC/FID. FTIR and GC/FID showed hydrocarbons above C_{10} at ~15 μg/ mL. No CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 was detected by HPLC/IC.

3.2.2 Pig #6

393-6a: The ^1H NMR spectra of both the D_2O and the $CDCl_3$ extracts were very broad. This indicates that paramagnetic metal ions may be present. In spite of the broad resonances, acetophenone was observed in the D_2O spectrum (CH_3: δ2.62; H2,6: δ7.96; H3,5: δ7.21; H4: δ7.64). The spectrum of the $CDCl_3$ extract was extremely broad; however, it did show resonances for the presence of aliphatic and aromatic-type hydrocarbon protons. The broad singlet at δ2.60 indicates the presence of acetophenone, and the peak at δ5.57 suggests the presence of (2-oxo-2-phenyl)ethyl benzoate, Ph-C(O)OCH_2C(O)-Ph, a degradation product of CN (see 3.8, 1393-1b, below).

The following compounds were observed by GC/MS/CI:

Area %		
40.1%	(a)	Acetophenone (174 sec;m/z: 121, 105)
15.6%	(b)	(2-Oxo-2-phenyl)ethyl benzoate, Ph-C(O)OCH_2C(O)-Ph (858 sec; m/z: 241, 121, 105)
14.8%	(c)	Diphenyl sulfide (555 sec; m/z: 187)
14.0%	(d)	Benzoic acid (275 sec; m/z: 123, 105)
3.0%	(e)	Unknown MW 222 (822 sec; m/z: 223, 167, 145, 105)
2.5%	(f)	Diphenyl sulfone (770 sec; m/z: 219, 141)
2.5%	(g)	Phenol (126 sec; m/z: 95)
1.7%	(h)	Benzaldehyde (113 sec;m/z: 107)
1.6%	(i)	CN (331 sec; m/z: 155/157, 105, 77)
1.5%	(j)	Diphenyl sulfoxide (737 sec; m/z: 203, 187)
0.9%	(k)	Unknown MW 238 (923 sec;m/z: 239, 161, 121, 105)

The unknown compounds appear to be acetophenone condensation products.

GC/MS/EI confirmed the presence of acetophenone (510 sec; m/z: 120, 105, 89, 77, 63, 51 and 50), diphenyl sulfide (988 sec; m/z: 186, 185, 152, 92, 77, 65 and 51), and 1,2-diphenyl ethanone (723 sec; m/z: 122, 105, 77, 51 and 50). The GC/ITD also showed a large quantity of benzoic acid in the sample (874 sec; m/z: 105, 120, 77, 51 and 50) but it was difficult to quantitate because of poor chromatography of the acid. Diphenyl sulfide was also apparent by GC/ITD (1321 sec; m/z: 186, 185, 51 and 77) at ~1 mg/mL. In addition to a multitude of hydrocarbons, acetophe-none and CN were identified by the ITD; CN was found to be below 10 μg/mL. FTIR supported the identifications made by the ITD.

HPLC/IC was used to check for the presence of CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

393-6b: No resonances were observe by NMR in the D_2O extract. Only background hydrocarbons were detected in the $CDCl_3$ extract by both NMR and DEP/CI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

393-6c: No resonances were observed by NMR in the D_2O extract, and only background hydrocarbons (δ0.85 and 1.25) were detected in the $CDCl_3$ extract. Nothing was detected by GC/MS/CI, but DEP/CI observed protonated molecular ions at m/z 257, 225 and 193 indicative of elemental sulfur (S_8, S_7, and S_6, respectively). HPLC/IC showed no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2. In addition to the metals reported in Table 2, ICP found 7 ppm barium, 60 ppm copper, and 37 ppm sodium. The arsenic concentration was below detectable limits by GFAA.

393-6d: No resonances were observed by NMR in the D_2O extract. The $CDCl_3$ extract showed a large amount of aliphatic hydrocarbons and smaller amounts of aromatic compounds. No single component could be identified, however, the broadness of the resonances is indicative of the presence of a large amount of metal ions. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO, and TDGO2; none were detected. In addition to the metals listed in Table 2, ICP detected 67 ppm barium, 182 ppm copper, and 46 ppm sodium. The arsenic concentration was below detectable limits by GFAA.

393-6e: No resonances were observed by NMR in the D_2O extract, and only background hydrocarbons were detected in the

$CDCl_3$ extract. Nothing was detected by GC/MS/CI but DEP/CI showed a protonated molecular ion at m/z 105 suggestive of styrene. HPLC/IC found 10 µg/mL of CVAA; but no PD, arsenite or arsenate anion, TDG, TDGO and TDGO2 were detected. The presence of the CVAA could not be confirmed by a second technique.

3.3 Pigs #9 and #10 (1240 Hrs, 15 Jan 93), Table 3

3.3.1 Pig #9

293-9a: No resonances were observed by NMR in the D_2O extract. TNT (δ2.72 and 8.84) was observed in the $CDCl_3$ extract, along with an unknown compound possessing the following moieties:

$$\underset{\delta 0.92}{CH_3} - \underset{\delta 1.37}{CH_2} - \underset{\delta 1.57}{CH_2} - \underset{\delta 3.48}{CH_2} - X \qquad - \underset{\delta 3.59}{CH_2} - \underset{\delta 3.68}{CH_2} - Z - \underset{\delta 3.74}{CH_2} - \underset{\delta 3.63}{CH_2} - Y$$

where X, Y, Z are electronegative groups (i.e., O, Cl, SO_2, etc.).

Based on the NMR data, the unknown might be diethylene glycol monobutyl ether (butyl Carbitol®), $CH_3(CH_2)_3OCH_2CH_2OCH_2CH_2OH$, but this was not confirmed. The mole ratio of TNT to the unknown was determined to be 26:74.

Nothing was detected by GC/MS/CI but the protonated molecular ion of TNT (m/z 228) was observed by DEP/CI, confirming its presence. Several unidentified ions were also detected at m/z 336, 354, 372, 390 and 408. Only background hydrocarbons (C>10) were observed by GC/FID, and no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 were detected by HPLC/IC.

293-9b: No resonances were observed by NMR in the D_2O extract. TNT (δ2.72 and 8.84) was observed in the $CDCl_3$ extract and confirmed by DEP/CI (m/z: 228). Ions at m/z 187/189, 229, 301 and 397 in the DEP/CI spectrum of the D_2O extract are indicative of arsenic acids. However, HPLC/IC detected no arsenite or arsenate anion present. In addition, no CVAA, PD, TDG, TDGO or TDGO2 were detected.

293-9c: No organic compounds were detected in the ^1H NMR spectrum of the D_2O extract, but several compounds were observed in $CDCl_3$:

Mole %

60% (a) Unknown trans-vinyl compound (δ6.48 and 7.11; J = 14.7 Hz)
23% (b) L-3 (δ6.38 and 6.48; J = 14.7 Hz)
6% (c) TNT (δ2.72 and 8.84)
8% (d) Unknown (δ6.49, singlet)
3% (e) Unknown (δ6.72, singlet)

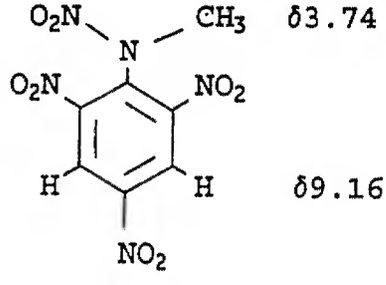
The presence of L-3 (461 sec) was confirmed by GC/MS/CI and DEP/CI (m/z: 197/199/201), and GC/MS/EI (1140 sec; m/z: 147, 145, 136, 110, 100 and 77). No CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 were detected by HPLC/IC.

293-9d: Resonances for a monosubstituted benzene compound (δ7.77, 2H, doublet; δ7.62, 2H, triplet; δ7.68, 1H, triplet) were observed by NMR in the D_2O extract; however, no identification was possible. The NMR spectrum of the $CDCl_3$ extract showed the presence of several aromatic compounds (δ7.2-8.2) but no single component could be identified.

Two major compounds were detected by GC/MS/CI and DEP/CI: 31 area% triphenyl arsine (923 sec; m/z: 307, 229); and 19 area % diphenyl sulfide (557 sec; m/z: 187). Other related compounds containing arsenic and phenyl moieties with molecular weights of 230, 304, 336 and 378 were also detected but not identified. HPLC/IC detected 2 μg/mL CVAA but no PD, arsenite or arsenate anion, TDG, TDGO and TDGO2 were found.

3.3.2 Pig #10

293-10a: The ^1H NMR spectrum of the D_2O extract showed two small resonances at δ3.69 and 3.73 but no identification was possible. The $CDCl_3$ extract was found to contain the explosive tetryl based on interpretation of the ^1H and ^{13}C NMR spectra obtained for sample 293-10b, below, and on comparison with a ^1H spectrum in the in-house database:



Tetryl

The presence of tetryl was confirmed by DEP/CI (m/z: 288, 242). No significant peaks were observed by GC/MS/EI, and HPLC/IC found no evidence for CVAA, PD, arsenite or arsenate anion, TDG, TDGO, or TDGO2.

293-10b: The fine yellow powder that dissolved in $CDCl_3$ was found by NMR to be:

Mole %		
88.9%	(a)	Tetryl (^1H: CH_3: δ3.73; H3,5: δ9.16; ^{13}C: CH_3: δ41.1; C1: δ131.9; C2,6: δ147.1; C3,5: δ124.5; C4: δ147.5)
10.5%	(b)	TNT (^1H: CH_3: δ2.71; H3,5: δ8.84; ^{13}C: CH_3: δ15.6; C3,5: δ122.2; C1,2,4,6: N.O.)
0.5%	(c)	Other (^1H: δ3.39)

A small amount of TNT (δ2.66 and 9.03) and tetryl (δ3.72 and 9.13) were also observed by ^1H NMR in the D_2O "extract" of the powder.

DEP/CI also identified the yellow powder as tetryl (m/z: 288, 242). A small amount of TNT (m/z: 228) was detected in the $CDCl_3$ solution by DEP/CI and by GC/MS/CI (640 sec). Tetryl was observed not to chromatograph. Only background peaks were observed by GC/MS/EI; and HPLC/IC found no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

293-10c: No resonances were observed by NMR in the D_2O extract; however, L-3 (δ6.38 and 6.47; J = 14.6 Hz) was observed in the $CDCl_3$ extract by ^1H NMR along with a significant amount of aliphatic hydrocarbons. The presence of L-3 was confirmed by DEP/CI, GC/MS/CI (465 sec; m/z: 197/199, 259/261), and GC/MS/EI (960 sec; m/z: 147, 145, 110, 100 and 77). HPLC/IC found 1 ppm CVAA; however, this could not be confirmed by a second technique. No PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 were detected. In addition to the arsenic determined by GFAA and the other metals reported in Table 3, ICP also found:

Ag	BDL	Cr	1 ppm	Mo	6 ppm	Sb	BDL
Be	BDL	Cu	4 ppm	Na	12 ppm	Se	BDL
Ca	65 ppm	Hg	BDL	Ni	BDL	Sn	BDL
Cd	BDL	K	85 ppm	P	9 ppm	Tl	BDL
Co	BDL	Mn	6 ppm	Pb	21 ppm	V	BDL

293-10d: Only background hydrocarbons were observed by ^1H NMR, DEP/CI, and GC/MS/EI. HPLC/IC found 1 ppm CVAA present but no PD, arsenite or arsenate anion, TDG, TDGO or TDGO2. In

addition to the metals reported in Table 3, ICP found the following:

Ag	BDL	Cr	1 ppm	Na	8 ppm	Se	BDL
Ba	10 ppm	Cu	2 ppm	Ni	1 ppm	Sn	BDL
Be	BDL	Hg	BDL	P	22 ppm	Tl	BDL
Cd	BDL	Mn	33 ppm	Pb	21 ppm	V	2 ppm
Co	1 ppm	Mo	4 ppm	Sb	BDL	Zn	BDL

293-10e: Only background hydrocarbons were observed by NMR and DEP/CI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

293-10f1: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI, and no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 were detected by HPLC/IC. In addition to the high amount of aluminum in this sample (see Table 3), ICP found the following:

Ag	BDL	Cr	BDL	Mn	BDL	Sb	BDL
Ba	3 ppm	Cu	BDL	Mo	2 ppm	Se	BDL
Be	BDL	Fe	99 ppm	Na	4 ppm	Sn	BDL
Ca	4 ppm	Hg	BDL	Ni	BDL	Tl	BDL
Cd	BDL	K	71 ppm	P	6 ppm	V	BDL
Co	BDL	Mg	130 ppm	Pb	BDL	Zn	BDL

The arsenic was also found to be below detectable limits.

293-10f2: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI; nothing was detected by GC/MS/CI. HPLC/IC found 1 ppm CVAA but no PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 were detected. In addition to the high aluminum content (see Table 3), ICP found the following metals:

Ag	BDL	Cr	BDL	Mn	2 ppm	Sb	BDL
Ba	2 ppm	Cu	BDL	Mo	2 ppm	Se	BDL
Be	BDL	Fe	73 ppm	Na	3 ppm	Sn	BDL
Ca	7 ppm	Hg	BDL	Ni	BDL	Tl	BDL
Cd	BDL	K	53 ppm	P	BDL	V	BDL
Co	BDL	Mg	142 ppm	Pb	2 ppm	Zn	BDL

293-10g: No resonances were observed in the ^1H NMR spectrum of the D_2O extract; however, the broadness of the HDO solvent peak may indicate the presence of metal ions. In addition to background hydrocarbon resonances, a small singlet at δ2.71 ppm in the ^1H NMR spectrum of the $CDCl_3$ extract is consistent for the presence of a small amount of TNT (see 3.3.1,

293-9b, above). Nothing was detected by GC/MS/CI, and only background hydrocarbons were observed by DEP/CI. HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

3.4 Pig #9, Second Delivery (1430 Hrs, 21 Jan 93), Table 4

893-1, 893-2 and 893-3: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI in all three samples. HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2. Chloride, nitrate and sulfate IC analyses were also run on sample 893-1; all were below detectable limits. ICP was run for metals only on sample 893-1; the following results were obtained:

Al	19 ppm	Cu	BDL
Ba	BDL	Fe	16 ppm
Ca	39 ppm	Mg	23 ppm
Cd	BDL	Mn	BDL
Co	BDL	Na	41 ppm

In addition, the arsenic in sample 893-1 was found to be below the detectable limit by GFAA.

893-4: No resonances were detected in the ^1H NMR spectrum of the D_2O extract; however, the spectrum of the $CDCl_3$ extract showed the presence of a significant amount of aliphatic hydrocarbons. No single component could be identified. DEP/CI detected only background hydrocarbons; and HPLC/IC observed no CVAA, PD, arsenite or arsenate anion, TDG, TDGO, TDGO2, chloride, nitrate or sulfate. In addition to the metals reported in Table 4, ICP found the following:

Al	BDL	K	BDL
Ba	BDL	Mn	2 ppm
Be	BDL	Na	57 ppm
Fe	BDL	Pb	BDL

3.5 Pig #10, Second Delivery (1530 Hrs, 22 Jan 93), Table 5

993-1, 993-2 and 993-3: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI in all three samples. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2 for all three samples; and chloride, nitrate and sulfate were also run on 993-1 and 993-3. None were found. In addition to the metals reported in Table 5, ICP found below

detectable limits of lead in both 993-2 and 993-3 and 38 ppm zinc in 993-3.

993-4: No resonances were observed by NMR in the ^1H NMR spectrum of the D_2O extract. The spectrum of the $CDCl_3$ extract, however, showed the presence of large amounts of aliphatic hydrocarbons (δ0.90 and 1.30) with smaller amounts of aromatic compounds being present (δ6.4-8.0). No single component could be identified. The presence of hydrocarbons up to mass 300 was confirmed by DEP/CI. No identifiable peaks were observed by GC/MS/EI; and HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

993-5: Only background hydrocarbons were observed by NMR and GC/MS/EI. The DEP/CI spectrum showed peaks indicative of elemental sulfur (m/z: 257, 225, 193, and 161). This sample appeared to be primarily S_8. HPLC/IC showed no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2. In addition to the metals reported in Table 5, ICP was run for aluminum (11 ppm) and sodium (31 ppm) on the D_2O extract.

993-6: Only background hydrocarbons were detected by NMR, DEP/CI, and GC/MS/EI; and no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 were detected by HPLC/IC.

993-7: No resonances were observed in the ^1H NMR spectrum of the D_2O extract; however, the broadness of the HDO solvent peak may indicate the presence of metal ions. Only background hydrocarbons were observed in the $CDCl_3$ extract by NMR, GC/MS/EI and DEP/CI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were found. In addition to the metals reported in Table 5, ICP was run for sodium (34 ppm), barium (1 ppm) and manganese (4 ppm).

3.6 Pigs #11, #12, #13, #14 and #15 (1000 Hrs, 25 Jan 93), Table 6

3.6.1 Pig #11

1093-1a, 1093-1b and 1093-1c: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI and nothing was detected for the two samples (1093-1a and 1093-1b) run by GC/MS/CI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2 on all three samples; none were found.

1093-1d: No resonances were observed in the ^1H NMR spectrum of the D_2O extract; however, the $CDCl_3$ extract was found to contain a significant amount of long chain aliphatic hydrocarbons and a significant amount of the alkene cis(1,4-polyisoprene)(i.e., rubber) as evidenced by the resonances at

δ1.68, 2.05 and 5.13. (see 3.6.3, 1093-3c, below). The presence of long chain hydrocarbons was confirmed by DEP/CI which observed a continuous band of peaks 14 mass units apart up to mass 500, with a maximum at about mass 370 (C_{26}). Similarly, the GC/MS/EI showed the presence of high molecular weight hydrocarbons (retention times > 1230 sec). HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were found.

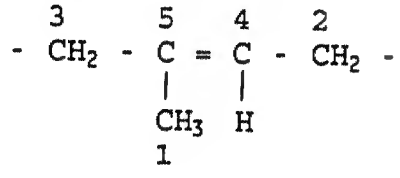
3.6.2 Pig #12

1093-2a and 1093-2b: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI; no single identifiable component was detected. No CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 was observed by HPLC/IC. GFAA was run for arsenic on sample 1093-2a; any arsenic was below detectable limits.

3.6.3 Pig #13

1093-3a and 1093-3b: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI. No CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 was detected by HPLC/IC.

1093-3c: No resonances were observed in the ^1H NMR spectrum of the D_2O extract; however, broadness of the HDO solvent peak may indicate the presence of metal ions. The ^1H NMR spectrum of the $CDCl_3$ extract showed the presence of a significant amount of aliphatic hydrocarbons over and above the background. One main component was observed which appeared to be present at a concentration of ~15-20 mg/mL or more based on the excellent signal-to-noise obtained for the NMR spectra. This compound contained the moiety:



^1H NMR Data (δ):

1	1.67
2&3	2.04
4	5.12

^{13}C NMR Data (δ):

1	23.4
2	26.4
3	32.2
4	125.0
5	135.2

It was identified as rubber, cis(1,4-polyisoprene), which had been leached from the rubber stopper during sample preparation.

The presence of the aliphatic hydrocarbons was confirmed by DEP/CI along with peaks indicative of the cis(1,4-polyisoprene) (m/z: 101 and 83), but GC/MS/EI detected no significant peaks. GC/FID, GC/ITD and FTIR showed no identifiable peaks except trace hydrocarbons. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

1093-3d: Only background hydrocarbons were detected by NMR, DEP/CI; and GC/MS/EI observed no significant peaks. HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

1093-3e: No resonances were observed by ^{1}H NMR in the D_2O extract; however, a significant amount of aliphatic hydrocarbon resonances were observed in the $CDCl_3$ extract along with resonances from δ0.6 to δ6.0. One of the major components was cis(1,4-polyisoprene) (rubber, δ1.67, 2.03 and 5.11), leached from the stopper. The two singlets at δ4.55 and 4.68 were originally thought to be part of an organofluorine compound since the distance between the two peaks is consistent for proton-fluorine coupling (52 Hz). However, a ^{19}F NMR spectrum showed no fluorine present to confirm this hypothesis.

GC/MS/EI confirmed the presence of long chain aliphatic compounds (e.g., hexadecanoic acid, 1000 sec;m/z: 256, 213, 129, 85, 55 and 43), and DEP/CI observed hydrocarbon peaks (m/z <250) and peaks at m/z 101 and 83 which appear to be from the cis(1,4-polyisoprene). HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were found.

1093-3f and 1093-3g: Only background hydrocarbons were observed by NMR and DEP/CI. The HDO solvent resonance, however, was broadened in the ^{1}H NMR spectrum of the D_2O extract of 1093-3f. This is indicative of the presence of metals in this sample. No evidence for CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 was observed by HPLC/IC in either sample.

3.6.4 Pig #14

1093-4a through 1093-4f: Only background hydrocarbons were observed on all six samples by NMR. Only hydrocarbon background was observed by DEP/CI in the $CDCl_3$ extracts of all six samples, but a case can be made for the presence of copper sulfate in sample 1093-4a on the basis of the DEP/CI spectrum of the solid. No confirmation for the presence of copper sulfate was sought. All samples except 1093-4f were analyzed by GC/MS/EI; no significant peaks were detected. HPLC/IC on all six

samples showed no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 to be present.

(NOTE: The rubber stopper was not leached in sample 1093-4b. Consequently, no cis(1,4-polyisoprene) was observed in the $CDCl_3$ extract by NMR).

1093-4g: No resonances were observed in the ^1H NMR spectrum of the D_2O extract; however, the spectrum of the $CDCl_3$ extract showed the presence of a significant amount of aliphatic hydrocarbons. Small resonances were observed throughout the unsaturated region (CH=CH; $\delta 5$ to $\delta 6$), but no aromatic resonances were detected; no single component was identifiable. The two singlets at $\delta 4.57$ and 4.69 are conspicuous but cannot be assigned to any particular compound. A ^{19}F NMR spectrum was run to ascertain the presence of an FCH_2- moiety; no fluorine resonances were observed.

The presence of hydrocarbons was confirmed by DEP/CI, GC/ITD and FTIR while GC/MS/EI saw no significant peaks. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were found.

3.6.5 Pig #15

1093-5a, 1093-5b and 1093-5c: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2 on samples 1093-5a and 1093-5b. None were detected.

3.7 Pigs #17, #18, #19, #21 and #22 (1010 Hrs, 27 Jan 93), Table 7

3.7.1 Pig #17

1193-4a and 1193-4b: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were found.

3.7.2 Pig #18

1193-2a and 1193-2b: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI. HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

1193-2c: No resonances were observed by NMR in the D_2O extract or in the $CDCl_3$ extract. DEP/EI and CI of the solid white powder suggest the presence of a mixture of arsenic and arsenic (III) oxide, As_2O_3. The peaks assigned to As_2O_3 [m/z (EI): 396, 289, 182, 91; and m/z (CI): 397, 381] agree with those obtained for an authentic sample of the oxide. HPLC/IC of the extracts showed no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2 present.

1193-2d: Only background hydrocarbons were observed by NMR and DEP/CI; however, the broadness of the HDO solvent peak in the D_2O extract may indicate the presence of metal ions. HPLC/IC detected 10 ppm CVAA and 20 ppm PD in this sample. No arsenite or arsenate anion, TDG, TDGO or TDGO2 was observed. The presence of CVAA and PD could not be confirmed by a second technique.

1193-2e: No resonances were observed in the ^1H NMR spectrum of the D_2O extract; however, both the ^1H and ^{13}C NMR spectra of the $CDCl_3$ extract showed significant amounts of cis-(1,4-polyisoprene), (rubber, ^1H: δ1.67, 2.04 and 5.12; ^{13}C: δ23.4, 26.4, 32.2, 125.0 and 135.2) and aliphatic hydrocarbons (^1H: δ0.8 to δ1.5) to be present. GC/ITD and FTIR, and DEP/CI confirmed the presence of the aliphatic hydrocarbons; and, the peaks associated with the cis(1,4-polyisoprene) (m/z: 101 and 83) were also observed by DEP/CI. HPLC/IC found no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

1193-2f through 1193-2i: Only background hydrocarbons were observed by NMR and DEP/CI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2 on all samples. None were detected.

3.7.3 Pig #19

1193-3a through 1193-3d: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI for all four samples. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO, and TDGO2 on each sample. In addition, chloride, nitrate and sulfate were run on samples 1193-3a through 1193-3c. In sample 1193-3a, 0.5 mg/mL chloride and 1.0 mg/mL nitrate were detected; none of the others were found.

3.7.4 Pig #21

1193-1: The ^1H NMR of the neat liquid showed that the sample was primarily water. However, both the ^1H and ^{13}C NMR spectra of a $CDCl_3$ extract of the liquid showed the presence of thiodiglycol (^1H: CH_2S: δ2.77; CH_2O: δ3.75; ^{13}C: CH_2S: δ36.0; CH_2O: δ60.9); 1,4-thioxane (^1H: CH_2S: δ2.62; CH_2O: δ3.91; ^{13}C: CH_2S:

δ27.1; CH_2O: δ68.8); and 1,4-dithiane (^1H: δ2.88; ^{13}C: δ29.2), the water soluble degradation products from the blister agent, mustard (HD). The presence of several large peaks in both the ether region of the ^{13}C spectrum (δ70-71) and the SCH_2 region (δ30-32) suggest that ether/sulfide polymers of thiodiglycol are also major components of this sample.

DEP/CI characterization of the neat liquid shows predominately the following compounds:

(a) Thiodiglycol (m/z: 123, 105)
(b) $(HOCH_2CH_2SCH_2CH_2)_2O$ (m/z: 227)
(c) $(HOCH_2CH_2SCH_2CH_2OCH_2CH_2)_2S$ (m/z: 331)

In addition, the following compounds were observed by GC/MS/CI, in lesser amounts, since thiodiglycol and thiodiglycol "polymers" are poorly chromatographed:

Area %		
56.0	(a)	1,4-Thioxane (78 sec; m/z: 105, 87)
19.0	(b)	1,4-Dithiane (168 sec; m/z: 121, 89)
16.5	(c)	1-Oxa-4,5-dithiapane (405 sec; m/z: 137, 121)
5.4	(d)	Ethyl 2-hydroxyethyl sulfide (95 sec; m/z: 107, 89)
2.1	(e)	1,4-Dioxa-7-thionane (221 sec; m/z: 149, 133, 105)
1.1	(f)	1,2,5-Trithiepane (445 sec; m/z: 153)

GC/MS/EI confirmed the presence of ethyl 2-hydroxyethyl sulfide (341 sec; m/z: 106, 75, 62, 61, 47 and 45); thiodiglycol (636 sec; m/z: 122, 104, 91, 61, 48 and 45); 1,4-thioxane (300 sec; m/z: 104, 74, 61, 46 and 45); and 1,4-dithiane (505 sec; m/z: 120, 92, 73, 64, 61, 60, 46 and 45).

No HD, itself, was observed by any of the three methods.

HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, nitrate or sulfate. However, 200 μg/mL of TDG (thiodiglycol), 50 μg/mL of TDGO (thiodiglycol sulfoxide) and a trace of TDGO2 (thiodiglycol sulfone) were observed. Furthermore, 2 mg/mL of chloride ion were detected indicating that these degradation products could have come from HD rather than just from degraded thiodiglycol.

3.7.5 Pig #22

1193-5a: No resonances were observed in the ^1H NMR spectrum of the D_2O extract, and only background hydrocarbons

were observed in the $CDCl_3$ extract. GC/MS/CI and GC/MS/EI observed only background hydrocarbons; and HPLC/IC found no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

DEP/CI and DEP/EI of the solid, itself, indicate the sample is red phosphorus [m/z (EI): 155, 124, 93 62; and m/z (CI): 125, 93]. The presence of phosphorus was confirmed by ICP (11.3% of the solid, as received). Furthermore, after drying in an oven, the sample was a free-flowing reddish solid which burned with a dense, white smoke on ignition. Both of these characteristics are consistent with red phosphorus.[2]

1193-5b: No resonances were observed by ^1H NMR in the D_2O extract, and only background hydrocarbons were observed in the $CDCl_3$ extract. HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2. The DEP/CI spectrum of the solid (m/z: 397, 381) was almost identical to that of sample 1193-2c (see 3.7.2, above) indicating the presence of arsenic, arsenic (III) oxide, and other related arsenic oxides and/or acids.

1193-5c: No resonances were observed in the ^1H NMR spectrum of the D_2O extract. The spectrum of the $CDCl_3$ extract showed that the sample contained a significant amount of aliphatic hydrocarbons (δ0.8 to 2.6) over and above the background. Resonances were observed in the SCH_2 and OCH_2 regions of the spectrum (δ2 to δ4), but a COSY experiment showed no cross peaks in the regions for mustard-type compounds. Tris(2-chlorovinyl)arsine, L-3, was observed (δ6.38 and 6.48, J = 14.8 Hz), but no Lewisite nor L-2 was detected. At least three other unidentified vinyl compounds were observed to be present (δ6.50 and 7.12; δ6.40 and 6.78; and, δ6.40 and 6.53) but could not be identified.

The presence of L-3 (m/z: 197/199) was confirmed by DEP/CI, and GC/MS/CI observed L-3 (491 sec; m/z: 197/199) as well as several unidentified chlorinated compounds (190 sec; m/z: 159/161/163, 123/125; and 207 sec; m/z: 159/161/163, 123/125). GC/MS/EI observed no Lewisite nor HD, but L-3 was detected [491 sec, m/z: 197/199; and 877 sec; m/z: 145, 136, 110, 100, 77, 51 (two different instruments used)]. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected.

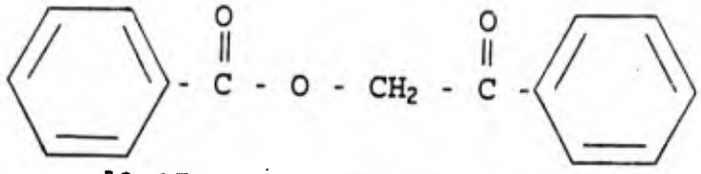
1193-5d: Only background hydrocarbons were observed by NMR and DEP/CI. The HDO solvent peak in the ^1H NMR spectrum of the D_2O extract was broad indicating the presence of metal ions

in the sample. HPLC/IC detected no CVAA, PD, arsenite or arsenate anion, TDG, TDGO or TDGO2.

3.8 Pig #23 (1 Feb 93), Table 8

1393-1a: Only background hydrocarbons were observed by NMR, DEP/CI and GC/MS/EI. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO, TDGO2, chloride, nitrate and sulfate; none were found.

1393-1b: The ^{1}H and ^{13}C NMR spectra of the $CDCl_3$ extract showed some aliphatic hydrocarbons (^{1}H: δ0.6 to δ4.0) and the presence of one major component. The NMR parameters for this compound are consistent with the following structure which appears to be a degradation/condensation product of CN, chloroacetophenone.



δ8.15 δ5.58 δ7.98

(2-Oxo-2-phenyl)ethyl benzoate

^{13}C NMR Data:

CH_2: δ66.4
Phenyl C: δ127.7 (2C); 128.4 (2C); 128.8 (2C); 129.9 (2C), 133.3 (1C); 134.2 (1C)
CC(O): δ192.1
OC(O): δ166.0

The sample also appears to contain smaller amounts of a second degradation/condensation product (^{1}H: δ3.47, see sample 1393-1d, below) and even smaller amounts of CN (^{1}H: δ4.79) and acetophenone ^{1}H: δ2.61. The ^{1}H NMR spectrum of the D_2O extract showed a small amount of aromatic resonances (δ7.47, 7.85) indicating the presence of some acids (e.g., benzoic acid) or other water soluble degradation products of CN.

DEP/CI also indicated the presence of acetophenone-related products. GC/MS/CI found the following compounds:

Area %		
40.9	(a)	Acetophenone (222 sec; m/z: 121, 105)
27.5	(b)	(2-Oxo-2-phenyl)ethyl benzoate, PhC(O)OCH$_2$C(O)Ph (964 sec;m/z: 241, 105)
13.3	(c)	Benzoic acid (328 sec; m/z: 123, 105)

10.2 (d) Diphenyl sulfide (641 sec; m/z: 187)
8.1 (e) CN (403 sec; m/z: 155/157, 141, 105, 77)

GC/MS/EI confirmed the presence of acetophenone (702 sec; m/z: 120, 105, 77, 51 and 50), CN (799 sec; m/z: 105,77 and 51) and benzoic acid (935 sec; m/z: 122, 105, 77, 51 and 50) as well as detecting diphenyl disulfide (1358 sec; m/z: 218, 185, 154, 109, 65 and 51).

The sample by FTIR showed bands indicating that the major component was an aromatic compound, possibly a monosubstituted ketone or ester (monosubstitution: 711, 688 cm^{-1}; aromatic bending: 1599, 1473 and 1448 cm^{-1}; C=O stretch: 1705 cm^{-1}; aromatic stretch: 3062 cm^{-1}; ketone: 1279 cm^{-1}; C=O overtones: 3210 cm^{-1}). These results are consistent with the structure proposed by NMR. Acetophenone, diphenyl sulfide and benzoic acid were also confirmed by GC/ITD.

HPLC/IC found 1 mg/mL chloride which is consistent with this sample being degraded CN, chloroacetophenone. HPLC/IC was also run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO, TDGO2, nitrate and sulfate; none were detected. ICP found high levels of iron and manganese; however, aluminum, barium, calcium, copper, lead, magnesium, sodium, sulfur, and zinc were all below detectable limits.

1393-1c: No resonances were observed by NMR in the ^1H spectrum of the D_2O extract. Numerous resonances were observed in the ^1H spectrum of the $CDCl_3$ extract indicating the presence of branched aliphatic hydrocarbons (δ0.6 to δ1.8), unsaturated (CH=C) compounds (δ4.7 to δ6.0) and aromatic compounds (δ6.8 to δ8.2). No single component could be identified, however.

DEP/CI observed hydrocarbon bands up to mass 300, and GC/MS/EI detected only background hydrocarbons indicating that the compounds present are not directly chromatographable. HPLC/IC found no CVAA, PD, arsenite or arsenate anion, TDG, TDGO, TDGO2, chloride, nitrate or sulfate present.

1393-1d: No resonances were observed in the ^1H NMR spectrum of the D_2O extract. The ^1H and ^{13}C spectra of the $CDCl_3$ extract, however, showed that the sample was primarily a mixture of two compounds both of which appear to be degradation/condensation products of CN.

Mole %

66 (a) (2-Oxo-2-phenyl)ethyl benzoate, $PhC(O)OCH_2C(O)Ph$ (^1H: CH_2: δ5.59, singlet; $CC(O)CCH$: δ8.15,

doublet; OC(O)CCH: δ7.98, doublet; Ph's: δ7.44-7.66; ^{13}C: CH_2: δ66.5; Ph's: δ127.8 (CH, 2C); δ128.4 (CH, 2C); δ128.9 (CH, 2C); δ130.0 (CH, 2C); δ133.2 (CCH, 1C); δ133.9 (CH, 1C); quats: N.O.).

34 (b) 1,4-Diphenyl-1,4-butanedione, $PhC(O)CH_2CH_2C(O)Ph$ (^1H: CH_2: δ3.48, singlet; C(O)CCH: δ8.05, doublet; Ph's: δ7.44-7.66; ^{13}C: CH_2: δ32.6; Ph's: δ128.1 (CH, 2C); δ128.6 (CH, 2C); δ133.3 (CH, 1C); quats: N.O.).

GC/MS/CI confirmed the presence of $PhC(O)OCH_2C(O)Ph$ (910 sec; m/z: 241, 105), and the DEP/CI spectrum indicated the presence of acetophenone-related products (m/z: 241, 123, 105). GC/MS/EI detected the presence of acetophenone (693 sec; m/z: 120, 105, 77, 51 and 43). Based on the NMR data, however, the acetophenone (^1H, $CDCl_3$: δ2.62) is a very minor component of the sample.

HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO, TDGO2, chloride, nitrate and sulfate; none were found.

3.9 Munition Samples (Table 9)

3.9.1 Item #71, 75mm Projectile (1030 Hrs, 18 Jan 93)

493-1c: The sample, as received, was a dark amber viscous liquid which emitted a slight white vapor when exposed to air. Prior to subjecting the sample to the analysis protocol, a vapor sample was obtained and analyzed by thermal desorption/mass spectrometry, and the pH of a 50% solution of the unknown in water was measured using a colorpHast pH strip (pH 0-14, EM Science, Gibbstown, NJ). No significant peaks were observed by GC/MS/EI from the vapor sample, and the pH was found to be <1 (extremely acidic).

^1H, ^{13}C, ^{31}P and ^{119}Sn NMR spectra were run on the neat sample; no resonances were observed for carbon, phosphorus or tin. A large, broad exchangeable proton (H^+) peak was observed in the ^1H NMR spectrum (δ11.4) indicating that the sample is probably an inorganic acid.

Based on its DEP/CI spectrum (m/z: 197, 117, 99, 81, 65, 48), the sample was identified as sulfuric acid, H_2SO_4. The identification was confirmed by ICP which found that the sample contained 32.2 wt% sulfur.

3.9.2 Item #82 (1515 Hrs, 18 Jan 93)

493-2c: The sample, as received, had the same physical appearance and characteristics as the sample from Item #71. It was a fuming, dark amber, viscous liquid with a pH<1. The GC/MS/EI of the vapor showed no significant peaks, and no resonances were observed by ^{13}C NMR. A large broad, exchangeable proton peak was observed (δ11.52) in the ^{1}H NMR spectrum, and ICP found 32.2 wt% sulfur present. Thus, it was concluded that this sample is also concentrated sulfuric acid.

3.9.3 Item #65, Livens (1250 Hrs, 19 Jan 93)

593-1c: The sample, as received, was a mobile liquid with a slight blue color. Thermal desorption/mass spectrometry of the vapor samples showed no significant peaks by GC/MS/EI; neither were any significant peaks observed in a $CDCl_3$ extract of the sample. No ^{13}C resonances were observed by NMR in the neat liquid, and ^{1}H NMR showed only a broad water peak at δ4.36. This chemical shift is consistent with the neutral pH measured for this sample using the colorpHast pH indicator strip. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO and TDGO2; none were detected. Traces of xylene (112 sec; m/z: 107), trimethylbenzene (173 sec; m/z: 121) and naphthalene (256 sec; m/z: 129) were observed by GC/MS/CI and could possibly arise from dissolution of some of the explosive components in the munition. A gray solid and a green-blue solid were isolated from the liquid but were not identifiable by DEP/CI. ICP for metals was run on both the neat liquid and on the mixed solid. In addition to the high levels of barium, calcium, magnesium, potassium, and sodium in the liquid (Table 9), ICP found:

Al	4 ppm	Mn	2 ppm
Be	BDL	Pb	BDL
Fe	3 ppm	Zn	BDL

The solid was found to contain the following:

Al	76 ppm	Cu	140 ppm	Na	1100 ppm
As	5 ppm	Fe	99000 ppm	Pb	1000 ppm
Ba	2600 ppm	Mg	93000 ppm	S	630 ppm
Ca	19000 ppm	Mn	380 ppm	Zn	BDL

It was concluded that this munition sample is a water solution of inorganic salts, mainly calcium and magnesium. Ion chromatography on another munition sample having a similar fill indicates that the anion is chloride (see below, sample 1293-1c).

3.9.4 Pig #8 (1300 Hrs, 21 Jan 93)

793-2: The sample, as received, was a heavy metal 3016-1 pipe with threaded ends that were capped. The container resembled older laboratory equipment that was used to perform pressure reactions. The pipe/container previously had been drilled by personnel at the U.S. Army Medical Research Institute of Infectious Diseases (USAMRIID) at Ft. Detrick, MD during their analysis for biological contamination. When a brown vapor escaped from the drilled hole, USAMRIID personnel plugged the hole with a latex surgeon's glove which came wrapped around the container/pipe. The glove was removed, and a vapor sample was taken and analyzed by thermal desorption/mass spectrometry. No organic compounds were detected by GC/MS/EI in the brown vapor. Subsequently, a total of 40 mL of a dark green, non-viscous liquid was removed from the cooled container with a needle and syringe. The liquid was allowed to come to room temperature, and a brown vapor was emitted.

The ^{1}H NMR spectrum of the neat sample showed broad resonances in the aromatic and H^{+} regions of the spectrum (δ8 to δ10). The ^{13}C spectrum of the neat sample showed that the sample was mainly nitrobenzene (C1: δ149.0; C2,6: δ124.3; C3,5: δ130.5; C4: δ136.1) with smaller amounts of 1,3-dinitrobenzene (C1: δ149.4; C2: δ119.7; C3: N.O.; C4: δ130.1; C5: δ132.2; C6: N.O.), and 1,4-dinitrobenzene (C1,4: δ154.4; C2,3,5,6: N.O.).

A small amount of sample was dissolved in deuterated methanol, CD_3OD (99.8 atom% D, MSD Isotopes, Montreal, CAN) in order to obtain a better ^{1}H NMR spectrum of the sample. The following compounds were identified:

Mole %		
41.5	(a)	Nitrobenzene [H2,6: δ8.23 (2H, "doublet"); H3,5: δ7.60 (2H, "triplet"); H4: δ7.76 (1H, "triplet")]
2.8	(b)	1,3-Dinitrobenzene [H2: δ9.00 (1H, triplet, J = 2.2 Hz); H4: δ8.62 (1H, doublet of doublets, J = 2.2, 8.2 Hz); H5: δ7.89 (1H, triplet, J = 8.2 Hz); H6: N.O.)]
1.7	(c)	1,4-Dinitrobenzene [H2,3,5,6: δ8.45 (4H, singlet)]
0.1	(d)	1,3,5-Trinitrobenzene [H2,4,6: δ9.32 (3H, singlet)]

1.4	(e)	Other aromatics
52.6	(f)	Excess H^+ (δ~5)

No sulfur was found by ICP indicating that the excess H^+ was probably due to nitric acid (HNO_3) rather than sulfuric acid (H_2SO_4).

The presence of nitrobenzene was confirmed by DEP/CI which observed protonated molecular ions at m/z 124 and 169 indicative of nitrobenzene and dinitrobenzene, respectively. GC/MS/EI (Finnigan 5100) observed the following compounds:

Area %		
95.6	(a)	Nitrobenzene (217 sec; m/z: 123, 107, 93, 77, 65)
2.8	(b)	Dinitrobenzene isomer (472 sec; m/z: 168, 122, 92, 76, 75, 64, 50)
0.6	(c)	Dinitrobenzene isomer (452 sec; m/z: 168, 122, 92, 76, 75, 64, 50)
0.4	(d)	Unknown MW 77, contains 1 nitrogen (49 sec; m/z: 46, 77)
0.4	(e)	Unknown MW 61, contains 1 nitrogen (26 sec; m/z: 46, 61)
0.2	(f)	Carbon tetrachloride (36 sec; m/z: 117/119, 82/84, 47/49)
0.1	(g)	Nitrobenzonitrile (398 sec; m/z: 148, 102, 90, 75, 64, 51)

A second vapor sample of the contents of the pipe/container did show the presence of nitrobenzene (754 sec; m/z: 123, 93, 77, 65, 51 and 50) by thermal desorption GC/MS/EI. Direct injection of an ether extract of the liquid confirmed the presence of nitrobenzene (560 sec); 1,4-dinitrobenzene (855 sec; m/z: 168, 122, 92, 75 and 50); and 1,3-dinitrobenzene (876 sec; m/z: 168, 122, 92, 76, 75 and 50). ICP found no significant metals present except for iron, probably from the container, itself.

It was concluded that this sample was most likely a pressure reaction to prepare trinitrobenzene from nitrobenzene and nitric acid. On a weight percent basis, the amount of acid present calculates to be 30-35%. Although no direct analysis of the brown vapor was obtained, the physical appearance (reddish brown) is consistent for nitrogen dioxide (NO_2/N_2O_4) which is a highly toxic gas used in the nitration of organic compounds.[3]

3.9.5 Item #87, Livens (1 Feb 93)

1293-1c: The sample, as received, was a bluish-gray mobile liquid. The ^{13}C NMR spectrum of the neat liquid showed no

resonances indicating that no organic compounds were present. This was further confirmed by a more sensitive ^1H NMR spectrum of a $CDCl_3$ extract of the liquid which showed only a small amount of background hydrocarbons to be present. The ^1H NMR spectrum of the neat liquid showed a broad singlet (δ4.52 ppm) consistent for neutral water.

Only background hydrocarbons were detected by DEP/CI and GC/MS/EI. HPLC/IC found 50 mg/mL chloride ion; but no CVAA, PD, arsenite or arsenate anion, TDG, TDGO, TDGO2, nitrate or sulfate were detected. In addition to the metals reported in Table 9, ICP found 16 ppm zinc and below detectable limits of vanadium, lead and cadmium in the liquid. ICP analysis of the solid residue in the sample gave the following results:

Al	51 ppm	Cu	12 ppm	Na	1400 ppm
As	0.5 ppm	Fe	5600 ppm	Pb	BDL
Ba	1700 ppm	Mg	53000 ppm	S	510 ppm
Ca	36000 ppm	Mn	BDL	Zn	BDL

Based on the analyses, it was concluded that this munition fill is similar to that of Item #65 (sample 593-1c), above: a water solution of inorganic salts, primarily chlorides of calcium, magnesium and sodium, in this case.

3.9.6 Item #67, Livens (2 Feb 93)

1493-1c: This sample, as received, was a gray, mobile liquid with a dark sediment. The ^1H NMR spectrum of the neat sample showed an extremely broad peak (peak width at half-height ~350 Hz) indicating that the sample was primarily water containing an abundance of metals. The ^1H NMR spectrum of a $CDCl_3$ extract of the sample showed the presence of 1,4-dithiane (δ2.90); 1,4-thioxane (SCH_2: δ2.63; OCH_2: δ3.92); and thiodiglycol (SCH_2: δ2.79; OCH_2: δ3.76). Other unidentifiable components were also present.

DEP/CI provided additional evidence for the presence of thiodiglycol (m/z: 105); however, GC/MS/EI observed only background hydrocarbons probably because low concentrations of thiodiglycol do not readily chromatograph. HPLC/IC observed 100 ppm thiodiglycol (TDG) along with 2 mg/mL of nitrate ion; no CVAA, PD, arsenite or arsenate anion, TDGO, TDGO2, chloride or sulfate were found.

Suspicion that this sample was merely the aqueous layer from a degraded mustard round and not representative of the true nature of the entire munition fill led to the analysis of a separated lower sludge layer to ensure that the Livens contained

no intact HD. The ^1H NMR spectra of the D_2O and $CDCl_3$ extracts of this sludge showed only very broad peaks. The sample appeared to contain a significant amount of metals, but no HD nor HD-type compounds could be detected. DEP/CI showed only background peaks except for a strong ion at m/z 65 which is sometimes indicative of sulfonate. In addition to the metals and high sulfur content listed in Table 9, ICP of the bottom sludge layer also found 62 ppm zinc and below detectable limits of barium and lead.

3.9.7 Item #147, 75mm Projectile (2 Feb 93)

1493-2: The sample, as received, was a fibrous solid that looked like wadding material. The ^1H NMR spectrum of the $CDCl_3$ extract of the solid showed mostly aliphatic hydrocarbon resonances (δ0.88 and 1.26). A smaller amount of aromatic resonances were present (δ7.0-8.0) as well as some resonances in the unsaturated (CH=C) and OCH region (δ4.6-5.4). Two small OCH_2 quartets were observed; one consistent for an ethyl ester, $CH_3CH_2OC(O)\sim$ (δ4.12) and one consistent for ethanol, CH_3CH_2OH (δ3.72). There was no evidence for the presence of any HD-type compounds.

The ^1H NMR spectrum of the D_2O extract of the fibrous material showed a triplet (δ1.17) and a quartet (δ3.64) consistent with ethanol; and, a singlet at δ1.91 which could be due to either a $CH_3C(O)\sim$ moiety (i.e., acetone or acetic acid) or to acetonitrile, CH_3CN. Some aliphatic hydrocarbon resonances (δ0.83 and 1.22), perhaps from a long chain acid or alcohol, were also observed.

GC/MS/CI observed aliphatic hydrocarbons in the $CDCl_3$ extract, primarily straight chain C_9-C_{16} compounds. This was confirmed by GC/MS/EI which found significant amounts of C_{10}-C_{18} hydrocarbons. HPLC/IC detected 2 mg/mL of nitrate ion, extracted perhaps from the explosives in the round. No CVAA, PD, arsenite or arsenate anion, TDG, TDGO, TDGO2, chloride or sulfate were observed. In addition to the metals listed in Table 9, ICP found 12 ppm barium and below detectable limits of aluminum, cadmium and cobalt.

3.9.8 Item #90, 75mm Projectile (2 Feb 93)

1593-1c: The sample, as received, was a dark, viscous liquid. The ^1H NMR spectrum of the neat sample showed no resonances; however, the ^{13}C NMR spectrum showed two large, very broad peaks (peak width at half-height ~150 Hz) and several smaller "humps". The two largest peaks represent ~59% of the total area in the spectrum. In our laboratory, this type of spectrum has been observed for samples of mustard (HD) that have

been stored in munitions and contain an abundance of dissolved metals.

The ^{1}H and ^{13}C NMR spectra of a $CDCl_3$ solution of this liquid confirmed the presence of a large amount of HD (^{1}H: SCH_2: δ2.92; $ClCH_2$: δ3.65; ^{13}C: SCH_2: δ34.5; $ClCH_2$: δ43.0). In addition, thiodiglycol (^{1}H: SCH_2: δ2.76; OCH_2: δ3.77), dioxane (^{1}H: δ3.73) and several other HD degradation products were observed. In this sample, the HD represented 72% of the total area.

The presence of HD and its various degradation products were confirmed by GC/MS/CI. The following compounds were observed:

Area %		
64.2	(a)	HD (284 sec; m/z: 159/161, 123/125)
11.8	(b)	HD disulfide, $(ClCH_2CH_2S)_2$ (443 sec; m/z: 191/193, 155/157)
9.7	(c)	1,4-Dithiane (183 sec; m/z: 121, 89)
3.7	(d)	HD trisulfide, $(ClCH_2CH_2S)_2S$ (619 sec; m/z: 223/225, 187/189, 159/161)
3.7	(e)	$ClCH_2CH_2SCH_2CH_2SCH_2CH_2Cl$ (667 sec; m/z: 183/185, 155/157, 123/125)
1.4	(f)	1,2,3-Trithiolane (220 sec; m/z: 125, 97)
1.1	(g)	1,4-Thioxane (91 sec; m/z: 105, 87)
0.9	(h)	1,2,5-Trithiepane (410 sec; m/z: 153, 125)
0.8	(i)	1,2,3,4-Tetrathiane (384 sec; m/z: 157, 125)
0.7	(j)	$ClCH_2CH_2SCH_2CH_2OCH_2CH_2Cl$ (503 sec; m/z: 167/169, 123/125)
0.4	(k)	$Cl(CH_2)_3SCH_2CH_2Cl$ (299 sec; m/z: 173/175, 137/139)
0.3	(l)	$ClCH_2CH_2SCH_2CH_2OCH_2CH_3$ (316 sec; m/z: 133, 123/125)
0.2	(m)	$ClCH_2CH_2SCH_2CH_2OCH_3$ (235 sec; m/z: 123/125, 119, 155)
0.1	(n)	$(ClCH_2CH_2)_2O$ (136 sec; m/z: 143/145, 107/109)
0.1	(o)	$(ClCH_2CH_2CH_2)_2S$ (349 sec; m/z: 187/189, 151/153)

GC/MS/EI of the $CDCl_3$ extract observed HD (624 sec; m/z: 158, 123, 111, 109, 73, 63, 59 and 45); $HOCH_2CH_2SCH_2CH_2SCH_2CH_2OH$ (816 sec; m/z: 164, 121, 105, 61 and 60); 1,4-dithiane (479 sec; m/z: 120, 92, 73, 61, 46 and 45); and 1,4-thioxane (273 sec; m/z: 104, 74, 61 and 46). HPLC/IC found 1 mg/mL of thiodiglycol (TDG) in the water extract but no CVAA, PD, TDGO or TDGO2 were detected.

3.9.9 Item #113, 75mm Projectile (2 Feb 93)

1693-1c: The sample, as received, was a dark mobile liquid with suspended solids. No resonances were observed in the ^{1}H NMR spectrum of the neat sample; the peaks were apparently broadened into the baseline due to the presence of paramagnetic

metals (especially the iron). Two broad peaks were observed in the ^{13}C NMR spectrum; however, no identification was possible. ICP confirmed the presence of metals. In addition to those listed in Table 9, ICP found 2 ppm copper, 50 ppm sodium and below detectable limits of barium.

The sample was miscible with water and immiscible with $CDCl_3$. Upon dilution with water, the ^1H and ^{13}C NMR spectra indicated the sample was better than 70% thiodiglycol (^1H, D_2O: δ2.19 and 3.32; ^{13}C: δ28.9 and 57.4). The spectra of the $CDCl_3$ extract confirmed the presence of thiodiglycol (^1H, $CDCl_3$: δ2.78 and 3.76; ^{13}C; δ35.1 and 60.9) and also showed 1,4-dithiane (^1H: δ2.89; ^{13}C: δ29.1); 1,4-thioxane (^1H: δ2.63 and 3.92; ^{13}C: δ27.0 and 68.8) and thiodiglycol polymers/ethers (^{13}C: δ70.8). No evidence for the presence of intact HD was observed even when the ^{13}C spectrum was allowed to accumulated overnight for better sensitivity.

DEP/CI indicated that thiodiglycol (m/z: 105) and thiodiglycol polymers/ethers (m/z: 121, 137, 152, 164, 209, 228) were the major components. GC/MS/CI indicated the presence of the following compounds as minor constituents:

Area %	
29.0	(a) 1,4-Dithiane (177 sec; m/z: 121, 89)
27.7	(b) 1,4-Thioxane (88 sec; m/z: 105, 87)
22.8	(c) Unknown MW 120 (260 sec; m/z: 121)
20.6	(d) Unknown MW 136 (426 sec; m/z: 137, 121, 89, 77)

No intact HD was detected.

GC/MS/EI confirmed the presence of thiodiglycol (712 sec; m/z: 122, 104, 91, 61 and 47); 1,4-dithiane (513 sec; m/z: 120, 92, 61 and 46); 1,4-thioxane (318 sec; m/z: 104, 74, 61 and 46). Again, no intact HD was found.

HPLC/IC was run for chloride, nitrate and sulfate. 50 mg/mL of chloride was found; but no nitrate or sulfate were detected. The presence of so much chloride ion would indicate that this sample is probably degraded HD and not just degraded thiodiglycol.

Since this sample appeared to be a "water layer" from a degraded HD sample, the second sludge layer from the projectile was also analyzed to make sure that no intact HD was present in the munition. ^1H and ^{13}C NMR spectra of the $CDCl_3$ and D_2O extracts of the sludge layer showed the same compounds present as in the upper water layer: thiodiglycol (^1H, $CDCl_3$: δ2.78 and 3.76; ^{13}C, $CDCl_3$: δ35.1 and 61.0); 1,4-dithiane (^1H, $CDCl_3$: δ2.89; ^{13}C, $CDCl_3$: δ29.1); 1,4-thioxane (^1H, $CDCl_3$: δ2.63 and 3.92); and

thiodiglycol polymers/ethers (^{3}C, $CDCl_3$: δ70.8). No intact HD was observed indicating that this lower layer was not an HD layer as first suspected.

3.9.10 Item #142, 75mm Projectile (2 Feb 93)

1793-1c: The sample, as received, was a gray liquid with dark sediment. No resonances were observed by ^{1}H and ^{13}C NMR for the neat liquid, probably due to an abundance of dissolved metal ions. The ^{1}H NMR spectrum of a $CDCl_3$ extract of the liquid showed only background hydrocarbons indicating that the munition fill was not organic in nature. This observation was confirmed by DEP/CI which also observed only background hydrocarbons. HPLC/IC was run for CVAA, PD, arsenite and arsenate anion, TDG, TDGO, TDGO2, chloride, nitrate and sulfate. 50 mg/mL of chloride and 1 mg/mL of sulfate were observed; all other were below their detectable limits. Based on the analyses, the fill appears to be primarily water with dissolved metal salts.

4. SUMMARY/CONCLUSIONS

A total of 98 separate items/samples relating to Operation Safe Removal at Spring Valley, Washington, D.C. were analyzed by the Research and Technology Directorate Analysis Team during January and February 1993. Individual soil and debris samples from the site were given identification numbers/descriptors, packaged in numbered, stainless steel pigs and transported to ERDEC by the Technical Escort Unit (TEU). At ERDEC, the samples were removed from the pigs, given sample identification numbers and subjected to an analysis protocol. The analytical techniques used to characterize the samples included nuclear magnetic resonance (NMR) spectroscopy, gas chromatography/mass spectrometry (GC/MS), direct exposure probe mass spectrometry (DEP/CI), high performance liquid chromatography/ion chromatography (HPLC/IC), infrared (IR) spectrometry, inductively coupled plasma (ICP) emission spectrophotometry, and atomic absorption spectrophotometry.

The following compounds were found to be present in one or more of the soil/debris samples collected at the Spring Valley site:

Acetophenone
Adamsite (DM)
Arsenic
Arsenic III oxide
Benzaldehyde
Benzoic acid
Chloroacetophenone (CN)
bis-(2-Chlorovinyl)chloroarsine (L-2)

tris-(2-Chlorovinyl)arsine (L-3)
Chlorovinylarsonous acid (CVAA)
2-Chlorovinylarsonic acid (CVAOA)
1,4-Dioxa-7-thionane
Diphenylamine
1,4-Diphenyl-1,4-butanedione
Diphenyl sulfide
Diphenyl sulfone
Diphenyl sulfoxide
1,4-Dithiane
Ethyl 2-hydroxethyl sulfide
(2-Oxo-2-phenyl)ethyl benzoate
Phenyldichloroarsine
Red phosphorus
cis(1,4-Polyisoprene) (rubber)
Polymeric ethers/sulfides of thiodiglycol
Polystyrene
Sulfur (elemental, S8)
Tetryl
Thiodiglycol
Thiodiglycol sulfone
Thiodiglycol sulfoxide
1,4-Thioxane
Trinitrotoluene (TNT)
Triphenyl arsine

Nine intact munitions were downloaded at the ERDEC Chemical Transfer Facility (CTF); the following munition fills were identified:

Item #90:	60% mustard (HD) 40% HD hydrolysis/degradation products
Items #67 & #113:	Primarily water Some HD hydrolysis/degradation products (No intact HD detected)
Items #71 & #82:	Sulfuric acid
Items #65, #87 & #142:	Water solutions of inorganic salts, primarily the chlorides of magnesium, calcium and sodium
Item #147:	Fibrous solid (wadding material) Only aliphatic hydrocarbons extracted

In addition, one intact reaction vessel (SOL 42, 3016-1 pipe with capped ends) was received for analysis. The vessel was found to contain nitrobenzene and nitric acid along with small

amounts of dinitrobenzenes, trinitrobenzene, carbon tetrachloride and nitrobenzonitrile.

NMR, DEP/CI, GC/MS/CI, GC/MS/EI, GC/FPD/FID/ITD, FTIR and HPLC/IC spectra for each sample in which chemicals were identified are presented.

LITERATURE CITED

1. Doak, G. O., and Freedman, L. D., Organometallic Compounds of Arsenic, Antimony and Bismuth, John Wiley & Sons, New York, 1970, pp. 65, 89-90, 103-104, 109-110.

2. The Merck Index, 9th Edition, Windholz, M., Budavari, S., Stroumtsos, L., and Noether Fertig, M., eds. Merck & Co., Inc., Rahway, NJ, 1976, p. 7162.

3. Ibid, p. 858.

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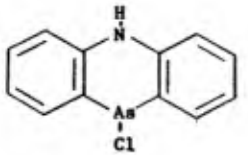
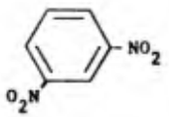
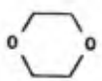
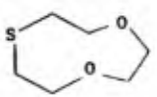
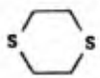
GLOSSARY

COMMON NAME/SYNONYM	IUC NAME	CAS NUMBER	STRUCTURE/FORMULA
Acetophenone; phenyl methyl ketone; acetylbenzene	1-Phenylethanone	98-86-2	C_6H_5-C(O)CH_3
Adamsite; DM; phenarsazine chloride; diphenylaminechlorarsine; phenazarsine chloride	10-Chloro-5-10-dihydrophenarsazine	578-94-9	
Arsenic	Arsenic	7440-38-2	As
Arsenic (III) oxide; arsenolite; arsenous acid; arsenous oxide; white arsenic; arsenic sesquioxide	Arsenic trioxide	1327-53-3	As_2O_3
Benzaldehyde; benzoic aldehyde	Benzaldehyde	100-52-7	C_6H_5-C(O)H
Benzoic acid; benzenecarboxylic acid; phenylformic acid	Benzoic acid	65-85-0	C_6H_5-C(O)OH
Butyl Carbitol®; diethylene glycol monobutyl ether	2-(2-Butoxyethoxy)ethanol	112-34-5	$CH_3(CH_2)_2OCH_2CH_2OCH_2CH_2OH$
Calcium chloride	Calcium chloride	10043-52-4	$CaCl_2$
Carbon tetrachloride; tetrachlormethane	Carbon tetrachloride	56-23-5	CCl_4
2-Chloroacetophenone; phenacyl chloride; CN	2-Chloro-1-phenylethanone	532-27-4	C_6H_5-C(O)CH_2Cl
2-Chlorovinyldichloroarsine; chlorovinylarsine dichloride; dichloro(2-chlorovinyl)arsine; Lewisite; L	(2-Chloroethenyl)arsonous dichloride	541-25-3	(ClCH=CH)$AsCl_2$

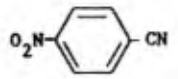
GLOSSARY (Continued)

COMMON NAME/SYNONYM	IUC NAME	CAS NUMBER	STRUCTURE/FORMULA
bis(2-Chlorovinyl)chloroarsine; L-2	bis(2-Chloroethenyl)arsinous chloride	40334-69-8	$(ClCH=CH)_2AsCl$
tris(2-Chlorovinyl)arsine; L-3	tris(2-Chloroethenyl)arsine	40334-70-1	$(ClCH=CH)_3As$
2-Chlorovinylarsonous acid; CVAA	(2-Chloroethenyl)arsonous acid	85090-33-1	$(ClCH=CH)As(OH)_2$
2-Chlorovinylarsonic acid; CVAOA	(2-Chloroethenyl)arsonic acid	-	$(ClCH=CH)As(O)(OH)_2$
Cupric sulfate; copper (II) sulfate	Copper sulfate	7758-98-7	$CuSO_4$
1,3-Dinitrobenzene; m-dinitrobenzene	1,3-Dinitrobenzene	99-65-0	
1,4-Dinitrobenzene; p-dinitrobenzene	1,4-Dinitrobenzene	100-25-4	
Dioxane; 1,4-diethylene dioxide	1,4-Dioxane	123-91-1	
1,4-Dioxa-7-thionane	1,4,7-Dioxathionane	40254-03-3	

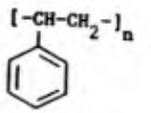
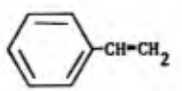
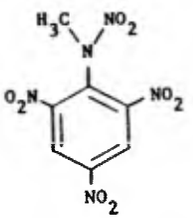
GLOSSARY (Continued)

COMMON NAME/SYNONYM	IUC NAME	CAS NUMBER	STRUCTURE/FORMULA
Diphenylamine	N-Phenylbenzeneamine	122-39-4	$(C_6H_5)_2NH$
1,4-Diphenyl-1,4-butanedione	1,4-Diphenyl-1,4-butanedione	495-71-6	C_6H_5-C(O)CH_2CH_2C(O)-C_6H_5
Diphenyl sulfide; phenyl sulfide	1,1'-Thiobis[benzene]	139-66-2	$(C_6H_5)_2S$
Diphenyl sulfoxide; phenyl sulfoxide	Sulfinylbis[benzene]	945-51-7	$(C_6H_5)_2S(O)$
1,4-Dithiane	1,4-Dithiane	505-29-3	
Ethyl 2-hydroxyethyl sulfide	2-(Ethylthio)ethanol	110-77-0	CH_3CH_2-S-CH_2CH_2OH
Magnesium chloride	Magnesium chloride	7786-30-3	$MgCl_2$
Mustard; mustard gas; H; HD; sulfur mustard; 2,2'-dichlorodiethyl sulfide; bis(2-chloroethyl)sulfide	1,1'-Thiobis[2-chloroethane]	505-60-2	$(ClCH_2CH_2)_2S$
Nitric acid	Nitric acid	52583-42-3	HNO_3
Nitrobenzene; nitrobenzol; oil of mirbane	Nitrobenzene	98-95-3	C_6H_5-NO_2

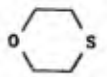
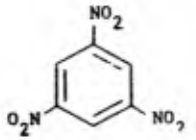
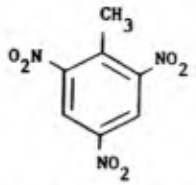
GLOSSARY (Continued)

COMMON NAME/SYNONYM	IUC NAME	CAS NUMBER	STRUCTURE/FORMULA
Nitrobenzonitrile	4-Nitrobenzonitrile	619-72-7	
Nitrogen dioxide; nitrogen tetroxide; dinitrogen tetroxide; nitrogen peroxide	Nitrogen dioxide	10102-44-0	NO_2/N_2O_4
Nonanoic acid; pelargonic acid; nonylic acid	Nonanoic acid	112-05-0	$CH_3(CH_2)_7C(O)OH$
Octanoic acid; caprylic acid	Octanoic acid	124-07-2	$CH_3(CH_2)_6C(O)OH$
(2-Oxo-2-phenyl)ethyl benzoate	(2-Oxo-2-phenyl)ethyl benzoate	-	$C_6H_5\text{-}C(O)OCH_2C(O)\text{-}C_6H_5$
Phenol; carbolic acid; phenic acid; phenyl hydroxide; hydroxybenzene; oxybenzene	Phenol	108-95-2	$C_6H_5\text{-}OH$
Phenyldichloroarsine; PD	Phenylarsonous dichloride	696-28-6	$C_6H_5\text{-}AsCl_2$
Red phosphorus	Phosphorus, red	7723-14-0	P
cis-1,4-Polyisoprene; natural rubber	cis-1,4-Polyisoprene	104389-31-3	$[\text{-}CH_2CH{=}C(CH_3)CH_2\text{-}]_n$

GLOSSARY (Continued)

COMMON NAME/SYNONYM	IUC NAME	CAS NUMBER	STRUCTURE/FORMULA
Polymeric ethers/sulfides of thiodiglycol	-	-	$(HOCH_2CH_2\text{-}S\text{-}CH_2CH_2)_2O$ $(HOCH_2CH_2\text{-}S\text{-}CH_2CH_2\text{-}O\text{-}CH_2CH_2)_2O$ $HOCH_2CH_2\text{-}S\text{-}CH_2CH_2\text{-}S\text{-}CH_2CH_2OH$
Polystyrene; poly(vinylbenzene)	Polystyrene	9003-53-6	
Sodium chloride; table salt	Sodium chloride	7647-14-5	NaCl
Sulfur; brimstone; sulphur	Sulfur	7704-34-9	S
Fuming sulfuric acid; oleum	Fuming sulfuric acid	8014-95-7	H_2SO_4
Styrene; vinylbenzene; phenylethylene	Ethenylbenzene	100-42-5	
Tetryl; nitramine; Tetralite; picrylmethylnitramine; N-methyl-N,2,4,6-tetranitroaniline	N-Methyl-N,2,4,6-tetranitrobenzenamine	479-45-8	

GLOSSARY (Continued)

COMMON NAME/SYNONYM	IUC NAME	CAS NUMBER	STRUCTURE/FORMULA
Thiodiglycol; 2-mercaptoethanol; 2-hydroxy-1-ethanol; 2-hydroxyethyl mercaptan; monothioethyleneglycol; bis(2-hydroxyethyl)-sulfide; 2,2'-thiodiethanol	2,2'-Thiobis[ethanol]	111-48-8	$(HOCH_2CH_2)_2S$
Thiodiglycol sulfone; TDGO2	2,2'-Sulfonylbis[ethanol]	2580-77-0	$(HOCH_2CH_2)_2S(O)_2$
Thiodiglycol sulfoxide; TDGO	2,2'-Sulfinylbis[ethanol]	3085-45-8	$(HOCH_2CH_2)_2S(O)$
1,4-Thioxane	1,4-Oxathiane	15980-15-1	
1,3,5-Trinitrobenzene; benzite; sym-trinitrobenzene	1,3,5-Trinitrobenzene	99-35-4	
2,4,6-Trinitrotoluene; TNT; trotyl; Tolit; Trilit; 1-methyl-2,4,6-trinitrobenzene	2-Methyl-1,3,5-trinitrobenzene	118-96-7	
Triphenylarsine	Triphenylarsine	603-32-7	$(C_6H_5)_3$-As

APPENDIX A

FINAL E-MAIL REPORT (UNEDITED) OF ANALYTICAL RESULTS FOR ALL SAMPLES RECEIVED THROUGH FEBRUARY 1993

MRS. MARGUERITE E. BROOKS, TEAM LEADER, SENDS:

1. In support of the U.S. Army Edgewood Research, Development and Engineering Center (ERDEC) "Operation Safe Removal" at the Spring Valley development in Washington, DC, selected suspect samples and munitions from the area were packed in "pigs"and transported to ERDEC. The samples were subjected to chemical analysis by scientists assigned to the Chemistry Department, Research and Technology Directorate, ERDEC. All samples underwent a screening protocol. Samples of granular and fiber materials were first analyzed by thermal desorption/mass spectrometry and direct inlet mass spectrometry. Each sample was subjected to solvent extraction/leaching. The recovered solvents were analyzed by nuclear magnetic resonance (NMR), direct inlet mass spectrometry, gas chromatography/mass spectrometry (GC/MS), liquid chromatography/ion chromatography (LC/IC) and infrared spectrometry (IR). Elemental analysis was performed using inductively coupled plasma (ICP) spectrometry on selected samples, as appropriate. The chemical analyses of these samples follows.

2. Four pigs containing samples for analysis were delivered by U.S. Army Technical Escort Unit (TEU) to bldg. E3300 at 1400, 9 January 1993.

a. Pig #1 contained two packages of broken glass. Each of the samples appeared to contain the same chemical components. These consisted of multiple aromatic and vinyl compounds related to Lewisite (L), including chlorovinylarsonic acid (CVAA), chlorovinylarsonic acid oxide (CVAOA), bis(2-chlorovinyl)chloroarsine (L2), and tris(2-chlorovinyl)arsine (L3). CVAA was determined to be 20 ppm and 40 ppm in samples #1 and #2, respectively CVAOA was no detected in samples #1, but was determined to be 20 ppm in sample #2. It should be noted that L and CVAA can not be differentiated since tris(2-chlorovinyl)arsine (L3). The CVAA was determined as 1 ppm and 4 ppm, respectively, in samples #1 and #2. It should be noted that L and CVAA can not be differentiated since each is determined as CVAA.

b. Pig #2 contained four samples. Sample #1 consisted of a test tube packed with soil having an oily sheen. The presence of the vomiting agent DM (Adamsite, diphenyl chloroarsine), and its precursor/degradation product diphenyl amine were identified and confirmed. They were present in approximately a 65:30 molar

ratio respectively, with two or three other compounds as minor components. Elemental analysis showed arsenic present at 250 ppm. Quantitation of the DM using the standard ultraviolet method gave a concentration of 42 mg DM/g sample. The sample met the quantitative requirement criteria that each of the absorbance wavelengths matched those of a standard of DM perfectly. Samples #2 and #3 consisted of tan and gray powders, respectively. Each was identified as containing background hydrocarbons with as the major component and long chain hydrocarbons as trace components. Sample #4 consisted of solid chunks of bituminous material having a green color on the surface. Analysis of the sample identified components similar to those detected in samples #2 and #3 from this pig.

c. Pig #3 contained three samples: A fuse leaking a dark viscous liquid; a leatherman tool apparently used to sample the liquid; and, a vial containing some of the liquid. The liquid in each sample was identified and confirmed to be the lachrymator CN, chloroacetophenone, with minor quantities of its aromatic degradation products, acetophenone, benzaldehyde and benzoic acid.

d. Pig #5 contained three samples. Sample #1 consisted of a sandy material containing water and trace quantities of background hydrocarbons. Sample #2 consisted of a pink colored polymeric foam with traces of soil adhered to it. The styrene structure of the polymeric foam was the only compound detected. Sample #3 was similar to sample #1 in appearance and analysis.

3. Two pigs containing samples for analyses were delivered by TEU to bldg. E3300 at 2015, 14 January 1993.

a. Pig #4 contained a mixture of various types of glass, household ceramic pieces, and both loose and packed soil. A mixture of these materials was leached with solvent. The presence of numerous aromatic and aliphatic compounds was observed. Trinitrotoluene (TNT) and aliphatic hydrocarbons having a carbon chain greater than 10 were detected. Three unknown compounds having mass weights of 256, 282 and 284 were detected, but do not appear to be related to CW agents.

b. Pig #6 contained five samples. Sample #1 consisted of a hard, black solid from a 75 mm round and was marked #43. Numerous aliphatic (C14 to C16) and aromatic compounds were detected. Identification of acetophenone, benzoic acid, and diphenylsulfide by GC/MS were also quantitated by GC/MS as 40% acetophenone, 16% benzoic acid, and 15% diphenylsulfide with smaller quantities of diphenylsulfone, phenol, diphenylsulfoxide, and benzaldehyde. The presence of acetophenone was confirmed by NMR. The possible presence of chloroacetophenone (CN) at <10 ug/g was detected by IR. Sample #2 consisted of a white and tan

solid and soil. The only compound identified in this sample was possibly benzoic acid. Sample #3 consisted of soil and a black tar-like material cited as being from a bomb windshield. Analyses were not definitive, but direct inlet/MS showed three mass peaks, which indicate the presence of sulfur (S8). Sample #4 was a fibrous material which was completely soluble in chloroform. It contained primarily aliphatic hydrocarbons, with traces of aromatic compounds. Heteroatoms such as oxygen, chlorine, and/or nitrogen could possibly be present. Sample #5 consisted of broken glass. Direct inlet/MS identified a mass peak of 104 related to styrene. Liquid chromatography identified the presence of 9 ppm of the Lewisite hydrolysate 2-chlorovinylarsonic acid (CVAA). No other compounds were identified.

4. Two pigs containing samples for analysis were delivered by TEU to bldg. E3300 at 1240, 15 January 1993.

a. Pig #9 contained four samples. Sample #1 consisted of green glass fragments with adhering soil particles. 2,4,6-trinitrotoluene, TNT, was identified and confirmed as a trace component. An additional aliphatic compound having ether-like linkages was detected, but this compound was not to be related to CW agents. Sample #2 consisted of clear glass with soil clumped on some surfaces. Trace amounts of TNT were also identified and confirmed as a component of this sample. Sample #3 also consisted of clear clear glass with soil clumped on some surfaces. Tris(2-chlorovinyl)arsine (L3) and TNT were identified and confirmed at higher than trace levels. Sample #4 consisted of clear and green glass mixed with black soil. The L hydrolysate, CVAA, was present at 2 ppm. Higher concentrations of triphenylarsine were detected (31% of the total organic compounds present) and confirmed. Diphenylsulfide was also detected at a relatively high (19%) concentration. Several other unidentified aromatic compounds were detected at trace levels.

b. Pig #10 contained eight samples. Sample #1 consisted of a brown waxy solid. It was identified and confirmed to be the explosive tetryl (85% pure). Sample #2 was finely divided yellow powder consisting of a mixture of tetryl and TNT in a molar ratio of 89:10.5. A small amount (0.5%) of a compound thought to be tetryl without the methyl moiety was also detected. The molar ratios were determined by NMR and the identification was confirmed by direct inlet/MS. Sample #3 consisted of white rubber tubing, coated with dirt. This consisted primarily of L3 and approximately 1 ppm of the L hydrolysate, CVAA. Additional hydrocarbons were also detected. The majority of the arsenic (300 ppm) can be accounted for by the presence of these L related compounds. However, there could be low ppm levels of arsenic present as inorganic salts or the oxides of arsenic. Sample #4 was of black soil which appeared to contain charcoal

fragments. The only organic component detected was the L hydrolysate, CVAA, at approximately 1 ppm. Elemental analysis detected low levels of aluminum, iron, lead, arsenic, trace quantities of molybdenum and zinc, and levels of potassium and magnesium expected in soil. Sample #5 consisted of a black solid, resembling charcoal. Only background hydrocarbons were detected in this sample. Sample #6A consisted of a light colored sand. Barely detectable levels of CVAA were identified. Arsenic quantitated at 2 ppm supports the presence of this compound. Sample #6B consisted of a dark colored sand. Again a low level of CVAA, 1 ppm, was detected. Quantitation of arsenic at 70 ppm suggests the presence of inorganic arsenic salts or arsenic oxides along with the CVAA. Sample #7 consisted of a mixture of small rocks and soil. The only compound detected was a trace of TNT.

5. Samples of the fills from two munitions, #71 and #82 were delivered by Chemical Transfer Facility (CTF) personnel to bldg. E3300, 18 January 1993, at 1030 and 1515 hours, respectively. Each sample consisted of a dark viscous liquid, which emitted a slight white vapor when exposed to air. Vapor samples of each were analyzed by thermal desorption/mass spectrometry prior to subjecting the liquid to the analysis protocol. The pH of a 1:1 water solution was also attempted. Analysis of each of the munition fills was exactly the same. They contained no organic compounds, no halogenated compounds, but consisted of fuming sulfuric acid.

6. A sample of the fill from the #65 munition was delivered to bldg. E3300 by CTF personnel at 1250, 19 January 1993. The sample was a mobile liquid with a slight blue color. Vapor samples were analyzed by thermal desorption/mass spectrometry prior to subjecting the liquid sample to the analysis protocol. A solid which was separated from the liquid was subjected to elemental analysis. The major component detected was water. Traces of aromatics, including one substituted benzenes and naphthalene were detected. Ion chromatography detected 50 mg chloride ion/ml of liquid. Elemental analysis of the liquid and the separated solid identified the following:

Element	Liquid ppm	Solid ppm
Aluminum	4	76
Arsenic	1	5
Barium	205	2600
Calcium	31,100	19,000
Copper		140
Iron	3	99,000
Lead		1000
Magnesium	72,100	93,000

Manganese		380
Potassium	1,195	
Sodium	1,160	1100
Sulfur	143	630

7. Pig #8, containing the sample opened at the U.S. Army Medical Research Institute for Infectious Diseases (USAMRIID), then sent to ERDEC, was delivered to bldg. E3300 at 1300, 21 January 1993.

a. The container, which had been drilled and plugged by USAMRIID personnel, was a heavy metal pipe with threaded ends, resembling laboratory equipment known to have been used to perform pressure reactions. A vapor sample was analyzed by thermal desorption/mass spectrometry prior to subjecting the sample to the analysis protocol. A total of 40 mL. of a dark green non-viscous liquid was removed from the container. At room temperature, a brown vapor, resembling nitrogen dioxide, was emitted by the liquid.

b. The major component in the liquid was identified and confirmed to be nitrobenzene. Minor components were identified as isomers of dinitro and trinitrobenzene, and nitric acid dissolved in the liquid. Elemental analysis gave the following data:

Element	ppm
Calcium	1
Iron	532
Magnesium	4
Manganese	2
Sodium	62

8. The second #9 pig, containing four samples was delivered by TEU to bldg. E3300 at 1430, 21 January 1993.

a. Sample #1, marked SOL 40, consisted of pieces of glass and a broken bottle which appeared to have a charred purple substance on the surface. The sample showed no organic compounds present.

b. Sample #2, marked SOL 43, consisted of broken glass. The analyses detected only background hydrocarbons.

c. Sample #3, marked SOL 44, consisted of broken glass. Only background hydrocarbons were detected in this sample.

d. Sample #4, marked SOL 45, consisted of a rubber stopper with a piece of glass tubing. Numerous aliphatic hydrocarbons, unsaturates and aromatic compounds were detected. No single

component could be identified. Elemental analysis gave the following data:

Element	ppm
Arsenic	<0.05
Calcium	224
Magnesium	143
Manganese	2
Sodium	57
Sulfur	288
Zinc	184

9. A second #10 pig was delivered to bldg. E3300 by TEU at 1530, 22 January 1993. The pig contained 7 samples.

a. Sample #1 described as "bright orange dirt around 75 mm projectile NE corner of pit 50 inch deep" had been identified on site as possibly containing trinitrotoluene, (TNT). No TNT or CW related organic compounds were detected. Only typical soil background hydrocarbons were found. Elemental analysis gave the following data:

Element	ppm
Aluminum	20,500
Arsenic	35
Barium	180
Cadmium	19
Calcium	90
Copper	114
Iron	96,200
Lead	3520
Magnesium	92
Manganese	137
Potassium	11,200
Sodium	3,540
Sulfur	6,360
Vanadium	32
Zinc	32

b. Sample #2, "a white powder taken from a livens (item #59)" also showed no organic species beyond a hydrocarbon background. An elemental analysis provided the following data:

Element	ppm
Aluminum	2030
Arsenic	1
Barium	64

Element	ppm
Iron	4450
Lead	203
Magnesium	24,700
Cadmium	67
Calcium	1430
Copper	50
Manganese	188
Potassium	2290
Sodium	7620
Vanadium	5
Zinc	234,000

c. Sample #3, "bright yellow dirt at end of 2 suspect livens NW corner 4 1/2 in." also showed no organic species beyond a hydrocarbon background. An elemental analysis provided the following data:

Element	ppm
Aluminum	9280
Arsenic	2
Barium	141
Cadmium	14
Calcium	148
Copper	22
Iron	41,900
Magnesium	252
Manganese	217
Potassium	12,800
Sodium	8810
Vanadium	24
Zinc	38

d. Sample #4, "black square of unknown material 3 in. x 3 in." resembled a roofing material. Analysis identified a large number of aliphatic compounds having high molecular weights. This is typical of tar paper and roofing material. The concentration of these compounds would totally obscure trace concentrations of other organic compounds.

e. Sample #5, "a probe placed in flash channel" also showed no organic species beyond a hydrocarbon background. The potential presence of elemental sulfur was identified by mass spectrometry. Elemental analysis identified the following:

Element	ppm
Aluminum	11.3
Arsenic	<0.08

Copper	44.5
Magnesium	61.6
Potassium	92.6
Sodium	30.8
Zinc	17.4

f. Sample #6, "green stained nail from 3 ft." also showed no organic species. The nail was made of copper. No anionic species were detected.

g. Sample #7, "a small tube 1 1/2 in. L x 1/4 in. dia." showed only a hydrocarbon background typical of soil. A trace of phthalate was detected by direct inlet/MS. Elemental analysis identified the following:

Element	ppm
Aluminum	383
Barium	1
Calcium	28
Iron	188
Magnesium	84
Manganese	4
Sodium	34

10. Five pigs, #11, #12, #13, #14, and #15 were delivered to bldg. E3300 by TEU 1000, 25 January 1993.

a. Pig #11 contained 4 samples. Sample #1, SOL 56, consisted of a small glass spray adaptor or trap with copper tubing. Only background hydrocarbon was detected in this sample. Sample #2, SOL 62, consisted of a glass bottle. Analysis showed mainly hydrocarbon background plus a small amount of what might be a propyl or butyl sulfide or ketone. Sample #3, SOL 59, consisted of another glass bottle. Analysis showed only background hydrocarbons. Sample #4 consisted of glass pieces. Long chain aliphatic compounds with molecular weights up to 400 were detected.

b. Pig #12 contained 2 samples. Sample #1, SOL 63, consisted of amber glass pieces. Only hydrocarbon background and water were detected on this sample. Sample #2, SOL 64, consisted of clear glass pieces. Only background hydrocarbons were detected in this sample.

c. Pig #13 contained 7 samples. Sample #1, SOL 71, consisted of fiber wadding. Only background hydrocarbons were detected. Sample #2, SOL 73, consisted of "cloth from around item # 98". Only background hydrocarbons were detected in this sample. Sample #3, SOL 66, consisted of stoppers with glass

tubing. The glass tubing was removed and analyzed to prevent the rubber stopper components from interfering with the analysis. Only background hydrocarbons were detected in this sample. Sample #4, SOL 74, consisted of wooden pieces. Only background hydrocarbons were detected. Sample #5, SOL 68, consisted of a "large rubber stopper with one hole". This was not a rubber stopper but resembled a cork ring used in laboratories to hold round bottom flasks in. It was very porous. Analysis identified only background hydrocarbons. Sample #6, SOL 67, consisted of a section of copper tubing. Only background hydrocarbons were detected on this sample. Sample #7, SOL 69, consisted of clear broken glass with a black tar substance adhered to it. Again only background hydrocarbons were detected.

d. Pig #14 contained 7 samples. Sample #1, SOL 60, consisted of "a green solid from a lead lined 75 mm, # 96". Sample #2, SOL 58, consisted of a "2-holed stopper with glass tubing". Only the glass tubing was evaluated from this sample. Sample #3, SOL 53, consisted of a "glass lab bottle". Sample #4, SOL 70, consisted of a "black and white granular solid". Sample #5, SOL 57, consisted of multiple glass pieces. Sample #6, SOL 75, was a "metal section from wall of item # 98 (spray tank)". Analysis of each of these samples showed only background hydrocarbons. Sample #7, SOL 76, consisted of "metallic tubing/fitting". An unusually high concentration of aliphatic hydrocarbons was detected.

e. Pig #15 contained 3 samples. Sample #1, SOL 72, was "a white flaky material from inside a ceramic jar". Only background hydrocarbons were detected in this sample. Sample #2, SOL 65, consisted of "broken glass tubing". Very low, trace levels of unidentified compounds containing OCH2, SCH2, and/or NCH2 moieties were detected beyond the hydrocarbon background. Sample #3, SOL 55, consisted of an "Erlenmeyer flask". Only background hydrocarbons were detected in this sample.

11. Five pigs, #17, #18, #19, #21 and #22 were delivered to bldg. E3300 by TEU at 1010, 27 January 1993.

a. Pig #17 contained 2 samples. Sample #1, SOL 83, consisted of "various clear bottle pieces". Sample #2 consisted of "various amber jar broken pieces". Each sample showed only background hydrocarbons, with no evidence of ionic or metal species.

b. Pig #18 contained 9 samples. Sample #1, SOL 78, was a "glass pipet stuck in glass jar top". Sample #2, SOL 81, consisted of "broken glass test tubes". Each of these samples contained only background hydrocarbons. Sample #3, SOL 87, was a "white powder from inside a 75 mm". The only organic compounds present were background hydrocarbons. However, direct inlet MS

suggested the presence of a mixture of arsenic and arsenic (III) oxides. Peaks assigned to arsenic oxide agree with those obtained for an authentic sample. Sample #4, SOL 86, consisted of "clear glass pieces w/black tar-like substances". Only background hydrocarbons were detected on this sample. Sample #5, SOL 82, consisted of a "broken small glass bottle with tube". Sample #6 was a "glass stopper". Sample #7, SOL 77, consisted of a "glass bottle from aspirator/trap w/copper fitting". Sample #8, SOL 80, consisted of "metal handles". Sample #9, SOL 84, consisted of "cloth fiber pieces". Each of these samples contained only background hydrocarbons.

c. Pig #19 contained 4 samples. Sample #1, SOL 91, consisted of a "75 mm base w/black substance lining interior". Sample #2, SOL 90, consisted of a "black burnt substance found in 2 qt can". Sample #3, SOL 92, was a "white granular substance found around glassware". Sample #4, SOL 93, consisted of "a tan solid from base of 75 mm." Each of these samples contained only background hydrocarbons. There was indication of some metals present in sample #4.

d. Pig #21, LIQ 5, contained a "liquid from inside 75 mm, #126". This sample had been identified as containing components potentially related to mustard, (H). The sample consisted of a slightly viscous yellow liquid with a fine particulate suspension. The pH of the liquid was 5, slightly acid. Compounds identified and confirmed as being present included thiodiglycol (TDG), 20 %; the TDG sulfoxide, 0.5%; the TDG sulfone, 1,4 dithiane and 1,4 oxathiane in trace quantities. IC identified the chloride content of the sample at 2%. Elemental analysis found the following metals:

Element	ppm
Arsenic	<0.05
Cadmium	35
Calcium	69
Copper	1800
Iron	135,000
Magnesium	50
Manganese	455
Sodium	75
Sulfur	48,900

e. Pig #22 contained 4 samples. Sample #1, SOL 89, consisted of "clay-like filler from inside fuse of 75 mm, # 114". This sample appeared to be insoluble in the solvents used. No organic structures were detected. However, direct inlet mass spectrometry identified the sample as red phosphorus. Elemental and total phosphorus analysis confirmed the presence of phosphorus at 25% and detected arsenic at 40 ppm. Sample #2, SOL

94, was a "white powder from item # 75, unfired projectile". The only organic species detected were background hydrocarbons. However, direct inlet MS spectrum of the solid was almost identical to that of sample #3, pig #18, indicating the presence of arsenic, arsenic oxide, and possibly other related oxides and/or acids. Sample #3, SOL 96, consisted of glass "tubing". The presence of L3 was identified and confirmed in this sample. Several other trans-vinyl compounds which appear to be L analogs or degradation products were also detected. Numerous aliphatic hydrocarbons were detected at much higher concentrations than expected for background levels. No H type compounds, nor L1 or L2 were detected. Sample #4, SOL 95, consisted of "part of item #118, smoke can". Only background hydrocarbons were detected. There was indication of possible metals present.

12. Pig #23 was delivered to bldg. E3300 by TEU 1 February 1993. Samples from it had previously been evaluated at USAMRIID and found not to contain biologically active components. The pig contained four samples. Sample #1A, SOL 97, consisted of a "broken glass bottle with a black substance". Only background hydrocarbons were detected in this sample. Sample #1B, SOL 98, consisted of "brown filler from inside 75 mm # 141". Aromatic ketone and ester type compounds (condensation/degradation products of CN) were detected. Mass spectrometry identified the presence of diphenylsulfide and low levels of CN and acetophenone with possible trace levels of a dimer and and trimer formed from the acetophenone. Chloride was detected at a concentration of 1.5 mg/g. Sample #1C, SOL 99, consisted of "lead balls and filler material from 75 mm #137". Branched alkyl and aromatic compounds detected could be related to diesel fuel. No evidence of any components related to H, L, or hazardous materials were detected. Sample #1D consisted of "lead from inside 75 mm #100". Traces of acetophenone, benzoic acid and possible alcohols were detected. A mixture of two condensation products of CN, having a molecular weight of 240, have been identified as approximately

67 mole% phenyl-C(O)OCH2C(O)-phenyl
33 mole% phenyl-C(O)-CH2CH2-C(O)-phenyl

13. Six munitions were opened and sampled by CTF personnel 1 and 2 February 1993. Samples from each were delivered to bldg. E3300 for analysis by CTF personnel. The items are listed in order of sample receipt.

Item #	Descriptor	Sample Appearance
87	livens	Light blue colored mobile liquid
67	4.7"	Gray mobile liquid, dark sediment
147	75 mm	Fibrous solids
90	75 mm	Dark slightly viscous liquid, solid particles suspended
113	75 mm	Dark mobile liquid, solid particles suspended
142	75 mm	Gray mobile liquid, dark sediment

a. The sample from item #87 appears to contain water as the primary component. Ion chromatography identified 50 mg chloride/mL. Elemental analysis of the sample gave the following data:

Element	ppm
Arsenic	5
Aluminum	16,900
Barium	16,100
Calcium	72,700
Copper	5
Iron	346
Manganese	189
Magnesium	159,000
Potassium	9020
Sodium	144,000
Zinc	16

b. The sample from item #67 also appeared to have water as the primary component. The mustard hydrolysate thiodiglycol (TDG) was detected at 100 ppm levels. Other mustard degradation products were also detected. Ion chromatography detected 2 mg nitrate/mL. A separated lower layer from the munition was analyzed to ensure it did not contain intact H. The analysis identified the same components as in the original sample. No intact H was present.

c. The sample from item #147 appeared to contain only innocuous aliphatic and aromatic hydrocarbons.

d. The sample from item #90 was identified and confirmed to contain a high concentration of intact mustard (at least 60% pure), in addition to numerous mustard hydrolysates and degradation products. Thiodiglycol (TDG) was detected at the 1 mg/mL level. Indicators point to a high metal content.

e. The sample from item #113, while primarily water, was found to contain significant amounts of H hydrolysates and

degradation products. A separated lower layer from the munition was analyzed to ensure it did not contain intact H. The analysis identified the same components as in the original sample. No intact H was present. Indicators point to a high metals content.

f. The sample from item #142 appeared to have water as the primary component. The analysis showed the only organic components to be background hydrocarbons. Ion chromatography detected 50 mg chloride/mL and 1 mg sulfate/mL.

Summary

1. A total of 104 samples related to the Spring Valley "Operation Safe Removal" were analyzed by the Research and Technology Directorate Analysis Team.

2. Of the nine munitions downloaded at the CTF, only # 90 contained an intact CW agent. It was identified and confirmed to contain a high concentration of intact mustard (H), at least 60% pure, in addition to numerous H hydrolysis and degradation products. Samples of two munitions, #67 and #113, contained water as the major component, and hydrolysis and degradation products of H as minor components. Samples of two munitions, #71 and #82, contained no organic compounds, no halogenated compounds, but consisted of fuming sulfuric acid. Samples of three other munitions, #65, #87, #142, were water solutions of inorganic salts, primarily the chlorides of magnesium, calcium and sodium. The sample from munition #147 appeared to contain only innocuous aliphatic and aromatic compounds.

3. Individual soil and debris samples from the site were given identification numbers or descriptors, packed in numbered pigs, and transported to ERDEC. Samples from pig #1 contained compounds related to the vesicant Lewisite (L), including CVAA, CVAOA, L2 and L3. Samples from pig #2 contained the vomiting agent Adamsite (DM) and its precursor/degradation product diphenylamine. Sample 3 from pig #3 contained the lachrymator chloroacetophenone (CN) and its degradation products. Samples from pig #4 contained detectable quantities of TNT. Samples from pig #5 contained only background components. Sample #5 from Pig #6 contained low levels of CVAA. Sample #1 from pig #6 contained 40% acetophenone, 16% benzoic acid, 15% diphenylsulfide, and possible traces of CN. Pig #8 contained an item resembling a pressure reactor. The liquid inside identified and confirmed to be nitrobenzene. Minor components were identified as isomers of dinitro and trinitrobenzene.

4. Three of the four samples in the first pig #9 contained from trace levels to high concentrations of TNT. Sample #4 in the

first pig #9 contained the L hydrolysate CVAA at low levels in addition to higher concentrations of triphenylarsine and diphenyl sulfide. Samples in the second pig #9 showed no evidence of any CW related or hazardous compounds. Three samples in the first pig #10 were identified and confirmed to contain the explosives TNT and tetryl in concentrations ranging from traces to a nearly neat mixture. Other samples in the first pig #10 contained low ppm levels of CVAA. No evidence of any CW related compounds was detected in the second pig #10. However three samples in the second pig #10 showed unusually high levels of metals.

5. No evidence of CW related compounds, explosives or other compounds of interest was found in pigs #11, #12, #13, #14, #15, #17, and #19.

6. Arsenic and arsenic (III) oxide were identified in one sample in pig #18. The other samples in pig #18 contained only background hydrocarbons. The pig #21 sample contained numerous compounds which are known to be H hydrolysis or degradation products. No intact H was detected. The detection of chloride ion indicated that the organic components detected were originally from the CW vesicant H and not from thiodiglycol (TDG).

7. One sample in pig #22 consisted of red phosphorus. Arsenic and arsenic (III) oxide were identified in another sample. A third sample from pig #22 contained L3 and several other possible L analogs or degradation products.

8. Degradation and condensation products of CN were the only compounds of interest detected in the samples in pig #23.

9. A formal ERDEC-SP report is being prepared for documentation and historical records.

10. Sample Analysis Team:

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APPENDIX B

SPRING VALLEY SAMPLES, WEIGHTS AND VOLUMES OF EXTRACTS

3/31/93
The following is a list of Spring Valley samples as of 3/31/93:

SAMPLE		DESCRIPTION	
OTH193-1	-	PIG #1	
OTH193-1a		broken glass, plate glass type	
-1b		broken glass from jars or carboys	
OTH193-2	-	PIG #2	
OTH193-2a		glass test tube with dark green oily substance	
-2b		grey/tan fine powder	
-2c		grey fine powder	
-2d		dark grainy greenish/brown substance	
OTH193-3	-	PIG #3	
OTH193-3a		rusted conical fuse with dark brown viscous liquid	[Item #1]
-3b		leatherman's tool with dark brown substance	[Item #2]
-3c		plastic vial with cap/dark brown substance	[Item #3]
OTH193-5	-	PIG #5	
OTH193-5a		brownish soil	[Item #1]
-5b		soil adhereing to/and with pink waxy looking fiber	[Item #2]
-5c		tannish sandy soil without pink waxy material	[Item #3]
OTH293-9	-	PIG #9	
OTH293-9a		glass, green	
-9b		glass, clear plus dirt	
-9c		glass, clear plus dirt	
-9d		glass, thick clear/green plus black dirt	
OTH293-10	-	PIG #10	
OTH293-10a		brown waxy material	[Item #1]
-10b		fine yellow powder	[also Item #1]
-10c		white rubber tubing with coating of dirt	[Item #2]
-10d		black solids plus soil (carbon/charcoal?)	[Item #3]
-10e		black solid (carbon?)	[also Item #3]
-10f1		solid (light sand)	[also Item #3]
-10f2		solid (dark sand)	[also Item #3]
-10g		small rocks with soil	[also Item #3]
OTH393-6	-	PIG #6	
OTH393-6a		hard black solid "sweet smell" from 75mm round #43 (soil)	
-6b		solid whitish/tan substance - clay-like	
-6c		black tar-like? substance from bomb windshield	
-6d		cotton fiber type wadding	
-6e		broken glass/rock/ceramic	

OTH393-4	- PIG #4	
OTH393-4	broken glass/ceramic/dirt	
OTH493-1	- MUNITION/DRILL&TAP	
OTH493-1a	Tenax tubes - Item #71 - 75mm projectile - GR0106	
-1b	'' '' '' - TA0105	
-1c	brown viscous liquid from Item #71 - fuming	
OTH493-2	- MUNITION/DRILL&TAP	
OTH493-2a	Tenax tube GR - GR0206	
-2b	Tenax tube TA - TA0205	
-2c	dark brown fuming liquid Item #82	
OTH593-1	- MUNITION/DRILL&TAP	
OTH593-1a	Tenax tube - LIVENS PROJECTILE - #65 - Tenax GR - GR0306	
-1b	'' '' '' Tenax TA - TA0305	
-1c	bluish/grey liquid	
OTH693	- MUNITION/DRILL&TAP	
OTH693	large CTF storage container of OTH593-1c	[Item #65]
OTH793-1	- PIG #8	
OTH793-1a	Tenax tube GR sample taken by us of -2 sample	
-1b	'' '' '' '' '' '' ''	
-2	#8 SOL-42 3016-1 pipe	[SOL 42]
OTH893	- PIG #9 AGAIN!!!!!!	
OTH893-1	glass broken bottle w/ charred purple substance	[SOL 40]
-2	broken glass collected from various depths of pit	[SOL 43]
-3	glass & ceramic pieces (55 inches deep) + dirt	[SOL 44]
-4	rubber stopper w/ glass tube piece (55 inches deep)	[SOL 45]
OTH993	- PIG #10 AGAIN!!!!!!!	
OTH993-1	bright orange dirt around 75mm projectile - 50" deep	
-2	white/ brown powder from Livens (item #59)	
-3	yellow/orange dirt at end of two suspect Livens	
-4	black square of unknown material	
-5	probe from flash channel	
-6	green stained large nail	
-7	small tube	
OTH1093-1	- PIG #11	
OTH1093-1a	small glass spray adapter or trap w/copper tubes	[SOL 56]
-1b	two clear glass bottles	[SOL 62]
-1c	clear glass bottle	[SOL 59]
-1d	white ceramic jar w/flaky solid inside	
OTH1093-2	- PIG #12	
OTH1093-2a	amber glass pieces	[SOL 63]
-2b	clear glass pieces	[SOL 64]

OTH1093-3 -	PIG #13	
OTH1093-3a	cotton fiber wadding	[SOL 71]
-3b	cloth from around the outside of item #98	[SOL 73]
-3c	stoppers w/glass tubing	[SOL 66]
-3d	wooden pieces	[SOL 74]
-3e	large rubber stopper w/one hole (NOT RUBBER)	[SOL 68]
-3f	copper piping	[SOL 67]
-3g	clear broken glass w/black tar substance	[SOL 69]
OTH1093-4 -	PIG #14	
OTH1093-4a	green solid from lead lined 75mm, #96	[SOL 60]
-4b	2-holed rubber stopper w/glass tubing	[SOL 58]
-4c	glass lab bottle	[SOL 53]
-4d	black/white granular solid	[SOL 70]
-4e	multiple glass pieces	[SOL 57]
-4f	metal section from wall of item #98 (spray tank)	[SOL 75]
-4g	metallic tubing/fitting	[SOL 76]
OTH1093-5 -	PIG #15	
OTH1093-5a	white flaky material from inside ceramic jar	[SOL 72]
-5b	broken glass tubing	[SOL 65]
-5c	Erlenmeyer flask	[SOL 55]
OTH1193-1 -	PIG #21	
OTH1193-1	liquid from inside 75mm, #126	[LIQ 5]
OTH1193-2 -	PIG #18	
OTH1193-2a	glass pipet stuck in glass jar top, item #1	[SOL 78]
-2b	broken glass test tubes, item #2	[SOL 81]
-2c	white powder from the inside of 75mm, item #3	[SOL 87]
-2d	clear glass pieces w/black tar like substances, I#4	[SOL 86]
-2e	broken small glass bottle w/tube, item #5	[SOL 82]
-2f	glass stopper, item #6	[SOL 79]
-2g	glass bottle from aspirator/trap w/copper fitting	[SOL 77]
-2h	metal handles, item #8	[SOL 80]
-2i	cloth fiber pieces, item #9	[SOL 84]
OTH1193-3 -	PIG #19	
OTH1193-3a	75mm base w/black substance lining interior, I#1	[SOL 91]
-3b	black burnt substance found in 2qt can, item #2	[SOL 90]
-3c	white granular substance found around glassware, I#3	[SOL 92]
-3d	tan solid from base of 75mm, item #4	[SOL 93]
OTH1193-4 -	PIG #17	
OTH1193-4a	various clear bottle pieces	[SOL 83]
-4b	various amber jar broken pieces	
OTH1193-5 -	PIG #22	
OTH1193-5a	clay-like filler from inside fuse of 75mm, #114, I#1	[SOL 89]

-5b	white powder from item #75; unfired projectile, I#2	[SOL 94]
-5c	tubing, item #3	[SOL 96]
-5d	part of item #118, smoke can, item #4	[SOL 95]

OTH1293 - ITEM #87
OTH1293-1a Tenax TA tubes from CTF-001
-1b '' GR '' '' CTF-002
-1c liquid from #87 Livens projector "R" , item #87

OTH1393 - PIG #23 TO AND FROM USAMRIID

OTH1393-1a	broken glass bottle w/black substance	[SOL 97]
-1b	brown filler from inside 75mm #141 + lead ball	[SOL 98]
-1c	lead balls and filler material from 75mm #137	[SOL 99]
-1d	lead balls from inside 75mm #100	

-2	small silver metal containers	

OTH1493 - ITEM 67
OTH1493-1a Tenax tubes
-1b Tenax tubes
-1c frozen, 2 layers, top/clear, bottom/dark, 75mm, #67
-1d sludge from -1c
-2 fibers from #147, 4.7"

OTH1593 - ITEM #90
OTH1593-1a Tenax tube
-1b Tenax tube
-1c liquid sample
-1d metal turnings

OTH1693 - ITEM #113
OTH1693-1a Tenax tube
-1b Tenax tube
-1c liquid sample
-1d metal shavings
-1e sludge

OTH1793 - ITEM #142
OTH1793-1a Tenax tube
-1b Tenax tube
-1c liquid sample
-1d metal shavings

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SPRING VALLEY SAMPLES -- DEUTERATED CHLOROFORM EXTRACTS

SAMPLE	GRAMS WEIGHT	MLS USED	MLS RECVD	MLS EVAPD
OTH193-1A	leach	8.0	6.74	1.0
OTH193-1B	leach	6.0	5.10	1.21
OTH193-2A	2.366	2.0	1.27	
OTH193-2B	2.791	2.0	1.32	
OTH193-2C	2.590	2.0	1.12	
OTH193-2D	1.909	2.0	1.55	
OTH193-3A	not weighed diluted into NMR tube			
OTH193-3B	leach	4.0	3.20	
OTH193-3C	0.0422	1.0	0.75	
OTH193-5A	3.136	2.0	1.65	
OTH193-5B	0.201	2.0	1.65	
OTH193-5C	3.305	2.0	1.52	
OTH293-9A	leach	6.0	4.70	2.00
OTH293-9B	leach	6.0	4.65	2.20
OTH293-9C	leach	6.0	3.55	1.24
OTH293-9D	leach	6.0	2.35	
OTH293-10A	0.048	2.0	1.71	
OTH293-10B	0.051	2.0	1.81	
OTH293-10C	0.044	2.0	1.58	
OTH293-10D	0.867	2.0	1.40	
OTH293-10E	1.279	2.0	1.63	
OTH293-10F1	1.304	2.0	1.67	
OTH293-10F2	0.713	2.0	1.45	
OTH293-10G	leach	4.0	3.25	
OTH393-6A	0.844	2.0	1.63	
OTH393-6B	0.275	2.0	1.80	
OTH393-6C	0.868	2.0	1.61	
OTH393-6D	0.994	4.0	3.48	
OTH393-6E	leach	6.0	3.75	1.64
OTH393-4	leach	6.0	2.45	1.23
OTH493-1A	Tenax tube			
OTH493-1B	Tenax tube			
OTH493-1C	fuming liquid	1.6920 g/ml		

OTH493-2A	Tenax tube			
OTH493-2B	Tenax tube			
OTH493-2C	fuming liquid	1.7334 g/ml		
OTH593-1A	Tenax tube			
OTH593-1B	Tenax tube			
OTH593-1C	liquid bluish	1.0859 g/ml		
OTH693	more of OTH593-1C			
OTH793-1A	Tenax tube			
OTH793-1B	Tenax tube			
OTH793-2	fuming liquid from 3016-1 pipe			
OTH893-1	leach	8.0	3.0	1.60
OTH893-2	leach	8.0	4.3	2.18
OTH893-3	leach	8.0	2.1	2.10
OTH893-4	leach	8.0	5.2	1.77
OTH993-1	1.358	2.0	1.50	
OTH993-2	0.967	2.0	1.37	
OTH993-3	1.559	2.0	1.40	
OTH993-4	3.676	4.0	2.90	
OTH993-5	0.388	2.0	1.70	
OTH993-6	4.084	2.0	1.75	
OTH993-7	1.048	2.0	1.74	
OTH1093-1A	leach	4.0	3.64	2.27
OTH1093-1B	leach	6.0	3.40	2.00
OTH1093-1C	leach	4.0	2.70	
OTH1093-1D	leach	2.0	0.55	
OTH1093-2A	leach	6.0	2.30	
OTH1093-2B	leach	6.0	1.82	
OTH1093-3A	1.132	4.0	3.40	
OTH1093-3B	0.972	2.0	1.64	
OTH1093-3C	8.385	4.0	1.82	
OTH1093-3D	0.713	2.0	1.00	
OTH1093-3E	leach	6.0	????	
OTH1093-3F	2.289	2.0	1.73	
OTH1093-3G	1.236	2.0	1.70	
OTH1093-4A	0.204	2.0	1.81	
OTH1093-4B	1.335	2.0	1.71	
OTH1093-4C	7.933	2.0	1.61	
OTH1093-4D	0.543	2.0	1.58	
OTH1093-4E	5.193	2.0	1.48	
OTH1093-4F	2.969	2.0	1.58	
OTH1093-4G	6.633	4.0	2.70	

OTH1093-5A	0.3545	2.0	1.78	
OTH1093-5B	1.4315	2.0	1.68	
OTH1093-5C	6.0495	4.0	3.70	
OTH1193-1	1.274	2.0	1.30	
OTH1193-2A	0.537	2.0	1.64	
OTH1193-2B	1.077	2.0	1.60	
OTH1193-2C	0.469	2.0	1.80	
OTH1193-2D	6.248	4.0	3.70	
OTH1193-2E	0.906	2.0	1.30	
OTH1193-2F	0.487	2.0	1.59	
OTH1193-2G	leach	3.0	2.55	
OTH1193-2H	0.635	2.0	1.72	
OTH1193-2I	leach	4.0	3.53	
OTH1193-3A	0.7679	2.0	1.64	
OTH1193-3B	0.3063	2.0	1.55	
OTH1193-3C	0.4238	2.0	1.77	
OTH1193-3D	0.7344	2.0	1.74	
OTH1193-4A	leach	6.0	3.80	
OTH1193-4B	leach	8.0	5.50	2.40
OTH1193-5A	0.413	2.0	1.86	
OTH1193-5B	0.542	2.0	1.65	
OTH1193-5C	1.000	2.0	1.37	
OTH1193-5D	7.626	4.0	3.05	
OTH1293-1A	Tenax tube			
OTH1293-1B	Tenax tube			
OTH1293-1C	liquid not extracted			
OTH1393-1A	leach	3.0	2.40	
OTH1393-1B	2.4545	2.0	0.90	
OTH1393-1C	leach	2.0	1.50	
OTH1393-1D	leach	2.0	1.54	
OTH1393-2	separate item - not Spring Valley			
OTH1493-1A	Tenax tube			
OTH1493-1B	Tenax tube			
OTH1493-1C	liquid extracted as 2ml liquid with 1 ml d. chloroform			
OTH1493-1D	0.1819	2.0		
OTH1493-2	0.0318	2.0	1.8	
OTH1593-1A	Tenax tubes			
OTH1593-1B	Tenax tubes			

OTH1593-1C	liquid not extracted
OTH1593-1D	metal turnings

OTH1693-1A	Tenax tube		
OTH1693-1B	Tenax tube		
OTH1693-1C	liquid not extracted		
OTH1693-1D	1.2588	2.0	1.82
OTH1693-1E	0.3102	2.0	

OTH1793-1A	Tenax tube
OTH1793-1B	Tenax tube
OTH1793-1C	2ml liquid extracted with 1 ml d. chloroform
OTH1793-1D	metal shavings

3/31/93

SPRING VALLEY SAMPLES -- DEUTERATED WATER EXTRACTS

SAMPLE	WEIGHT	MLS USED	MLS RECVD	MLS EVAPD
OTH193-1A	leach	8.0	5.5	
OTH193-1B	leach	8.0	5.4	
OTH193-2A	2.542	2.0	1.55	
OTH193-2B	2.850	2.0	1.28	
OTH193-2C	1.979	2.0	1.17	
OTH193-2D	1.974	2.0	1.30	
OTH193-3A	not weighed diluted into NMR tube			
OTH193-3B	leach	4.0	3.04	
OTH193-3C	0.0569	1.0	0.85	
OTH193-5A	3.241	2.0	1.15	
OTH193-5B	0.295	2.0	1.45	
OTH193-5C	3.085	2.0	1.31	
OTH293-9A	leach	6.0	4.10	
OTH293-9B	leach	6.0	3.85	
OTH293-9C	leach	6.0	3.65	
OTH293-9D	leach	6.0	3.55	
OTH293-10A	0.034	2.0	1.35	
OTH293-10B	0.068	2.0	1.75	
OTH293-10C	0.280	2.0	1.71	
OTH293-10D	0.539	2.0	1.37	
OTH293-10E	0.871	2.0	1.43	
OTH293-10F1	0.974	2.0	1.64	
OTH293-10F2	0.742	2.0	1.58	
OTH293-10G	leach	4.0	3.15	
OTH393-6A	0.969	2.0	1.57	
OTH393-6B	0.411	2.0	1.74	
OTH393-6C	0.814	2.0	1.55	
OTH393-6D	0.749	4.0	2.17	
OTH393-6E	leach	6.0	2.40	
OTH393-4	leach	10.0	1.80	
OTH493-1A	Tenax tube			
OTH493-1B	Tenax tube			
OTH493-1C	fuming liquid	1.6920 g/ml		

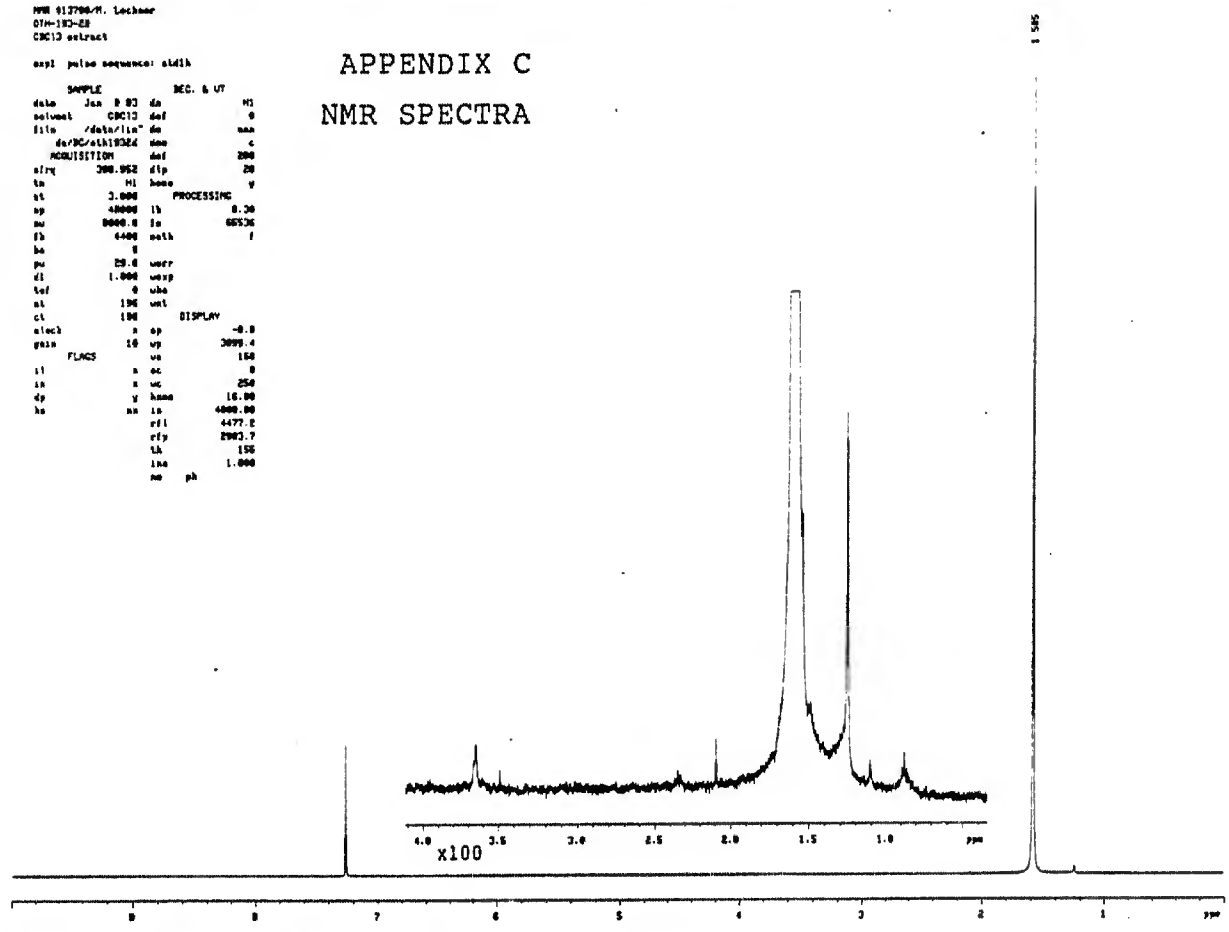
OTH493-2A	Tenax tube		
OTH493-2B	Tenax tube		
OTH493-2C	fuming liquid	1.7334 g/ml	
OTH593-1A	Tenax tube		
OTH593-1B	Tenax tube		
OTH593-1C	bluish liquid	1.0859 g/ml	
OTH693	more of OTH593-1C		
OTH793-1A	Tenax tube		
OTH793-1B	Tenax tube		
OTH793-2	fuming liquid from 3016-1 pipe		
OTH893-1	leach	8.0	2.03
OTH893-2	leach	8.0	1.77
OTH893-3	leach	8.0	2.84
OTH893-4	leach	8.0	5.57
OTH993-1	1.236	2.0	1.47
OTH993-2	1.187	2.0	1.35
OTH993-3	1.428	2.0	1.40
OTH993-4	2.611	4.0	2.80
OTH993-5	0.376	2.0	1.66
OTH993-6	leach	2.0	1.70
OTH993-7	1.122	2.0	1.60
OTH1093-1A	leach	4.0	3.60
OTH1093-1B	leach	6.0	2.54
OTH1093-1C	leach	4.0	1.58
OTH1093-1D	leach	2.0	1.65
OTH1093-2A	leach	6.0	1.70
OTH1093-2B	leach	6.0	1.60
OTH1093-3A	0.987	4.0	2.65
OTH1093-3B	1.089	2.0	1.50
OTH1093-3C	8.242	4.0	2.70
OTH1093-3D	0.768	2.0	1.34
OTH1093-3E	leach	6.0	????
OTH1093-3F	1.869	2.0	1.70
OTH1093-3G	0.870	2.0	1.75
OTH1093-4A	0.373	2.0	1.80
OTH1093-4B	0.924	2.0	1.70
OTH1093-4C	10.450	2.0	1.28
OTH1093-4D	0.920	2.0	1.59
OTH1093-4E	7.134	2.0	1.20
OTH1093-4F	5.167	2.0	1.44
OTH1093-4G	leach	4.0	3.20

OTH1093-5A	0.3551	2.0	1.65	1.86
OTH1093-5B	1.0534	2.0	1.57	
OTH1093-5C	5.0901	4.0	3.10	
OTH1193-1	liquid	put in NMR tube		
OTH1193-2A	0.891	2.0	1.62	
OTH1193-2B	1.488	2.0	1.40	
OTH1193-2C	0.542	2.0	1.62	
OTH1193-2D	5.496	4.0	3.25	
OTH1193-2E	1.038	2.0	1.65	
OTH1193-2F	0.569	2.0	1.54	
OTH1193-2G	leach	3.0	2.10	
OTH1193-2H	1.605	2.0	1.65	
OTH1193-2I	leach	3.0	2.20	
OTH1193-3A	0.8240	2.0	1.40	
OTH1193-3B	0.1972	2.0	1.35	
OTH1193-3C	0.6550	2.0	1.57	
OTH1193-3D	0.4540	2.0	1.75	
OTH1193-4A	leach	8.0	2.35	
OTH1193-4B	leach	6.0	3.97	
OTH1193-5A	0.499	2.0	1.50	
OTH1193-5B	0.584	2.0	1.42	
OTH1193-5C	1.176	2.0	1.65	
OTH1193-5D	3.470	4.0	3.14	
OTH1293-1A	Tenax tube			
OTH1293-1B	Tenax tube			
OTH1293-1C	liquid not extracted			
OTH1393-1A	leach	4.0	2.90	
OTH1393-1B	2.4098	4.0	2.70	
OTH1393-1C	leach	3.0	2.45	
OTH1393-1D	leach	3.0	2.42	
OTH1393-2	separate item - not Spring Valley			
OTH1493-1A	Tenax tube			
OTH1493-1B	Tenax tube			
OTH1493-1C	liquid extracted with d. chloroform			
OTH1493-1D	0.4348	2.0		
OTH1493-2	0.0411	2.0	1.72	
OTH1593-1A	Tenax tube			
OTH1593-1B	Tenax tube			

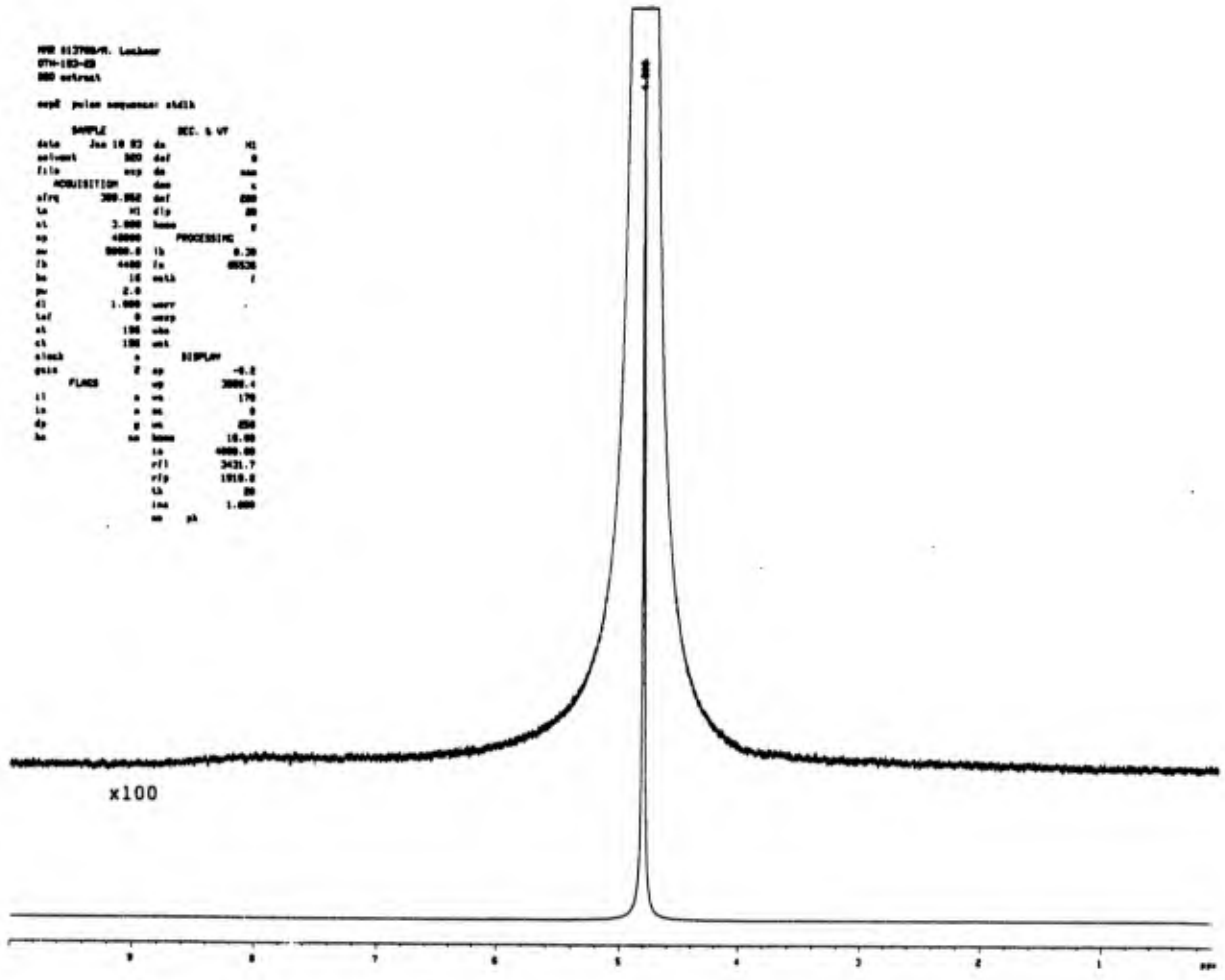
OTH1593-1C	liquid not extracted
OTH1593-1D	metal turnings
OTH1693-1A	Tenax tube
OTH1693-1B	Tenax tube
OTH1693-1C	liquid not extracted
OTH1693-1D	1.1652 3.0
OTH1693-1E	0.4511 2.0
OTH1793-1A	Tenax tube
OTH1793-1B	Tenax tube
OTH1793-1C	liquid extracted with chloroform
OTH1793-1D	metal shavings

APPENDIX C

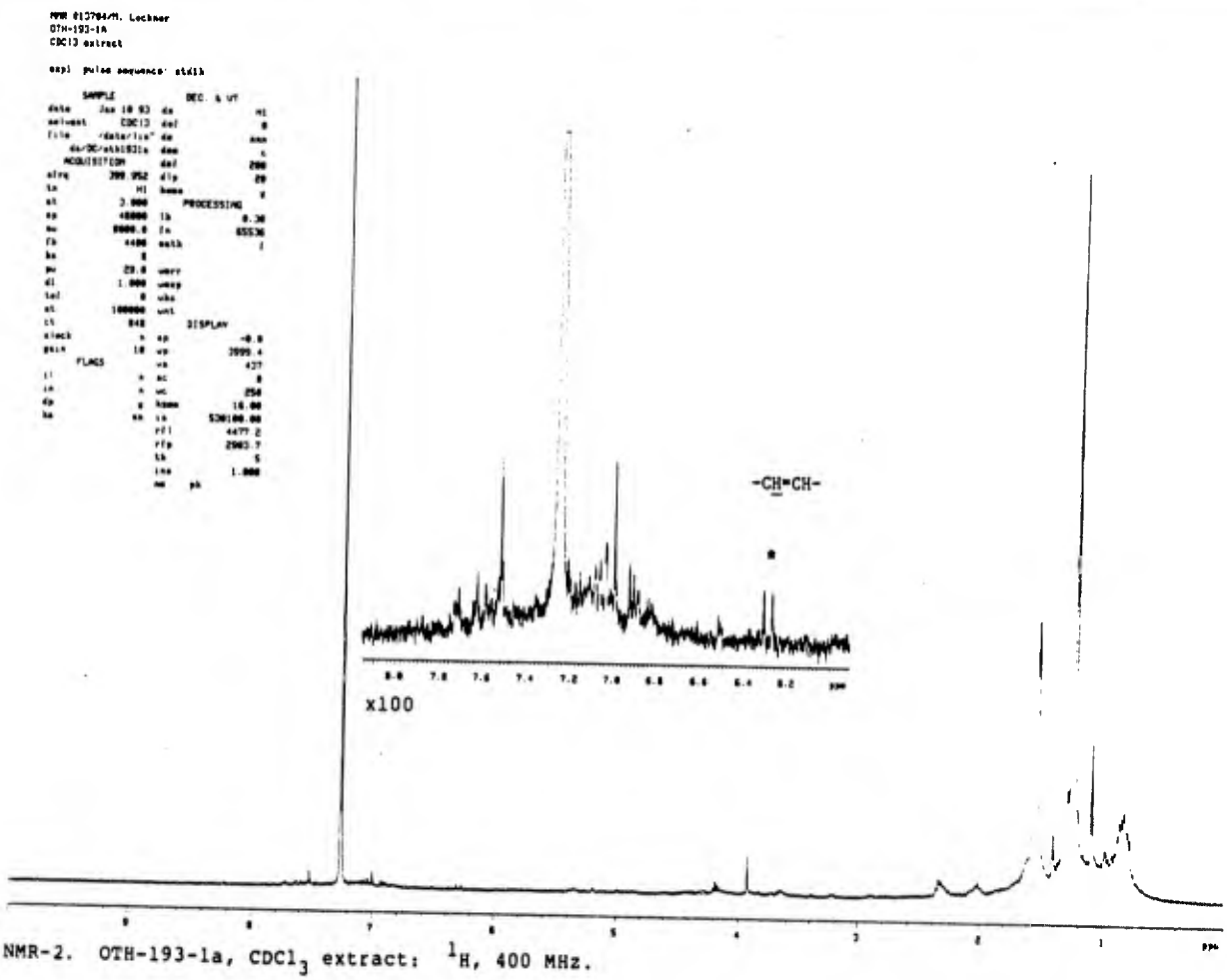
NMR SPECTRA



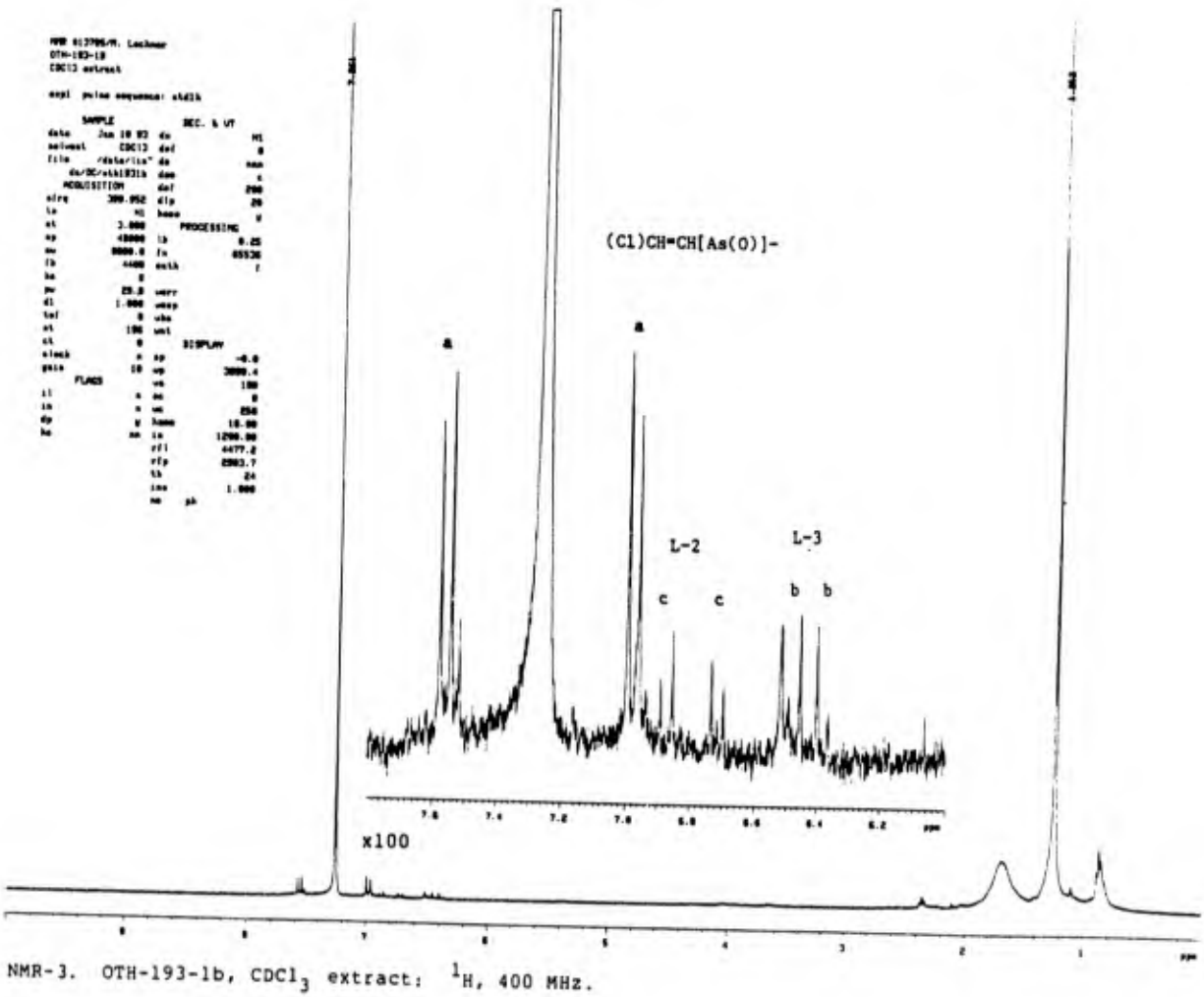
NMR-1a. Typical $CDCl_3$ background spectrum.



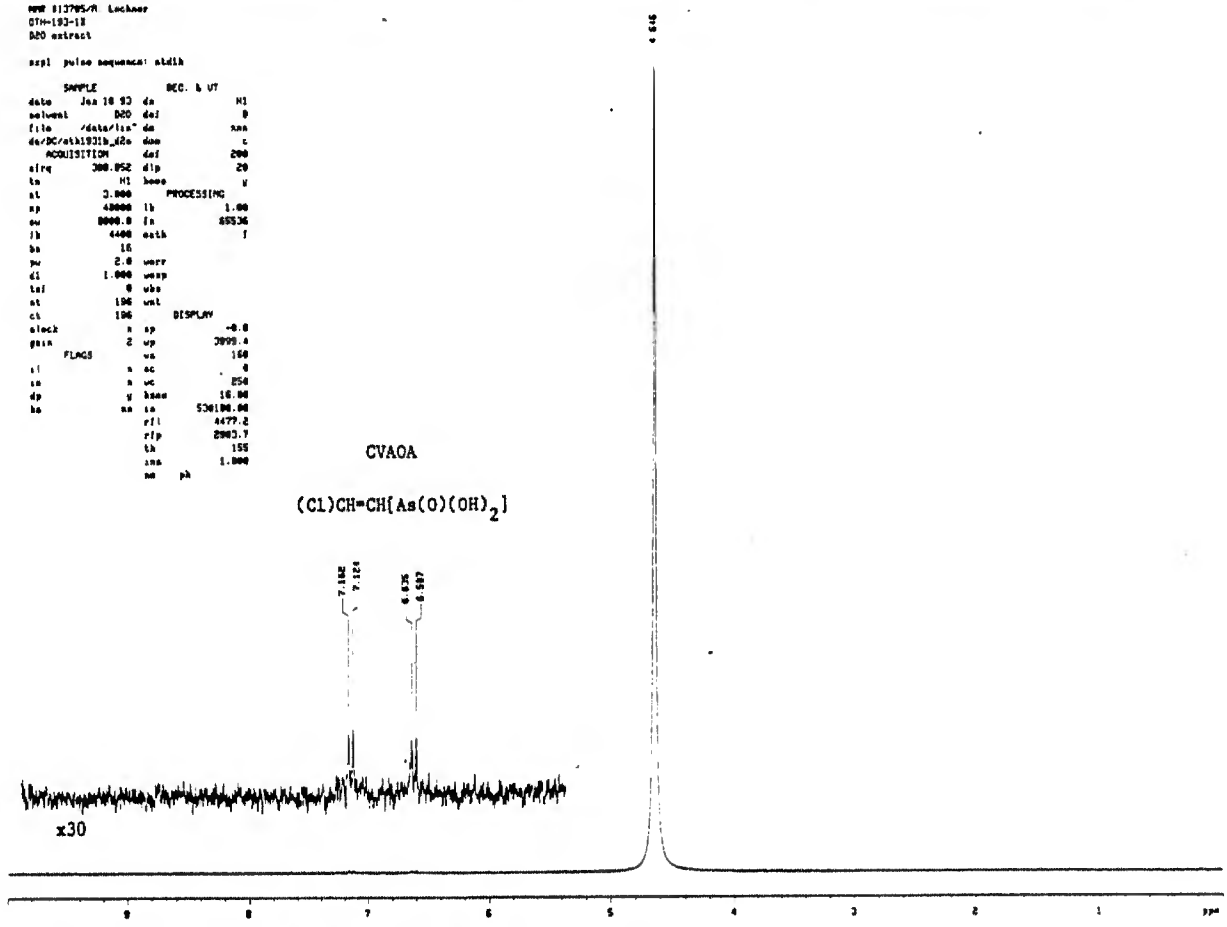
NMR-1b. Typical D_2O background spectrum.



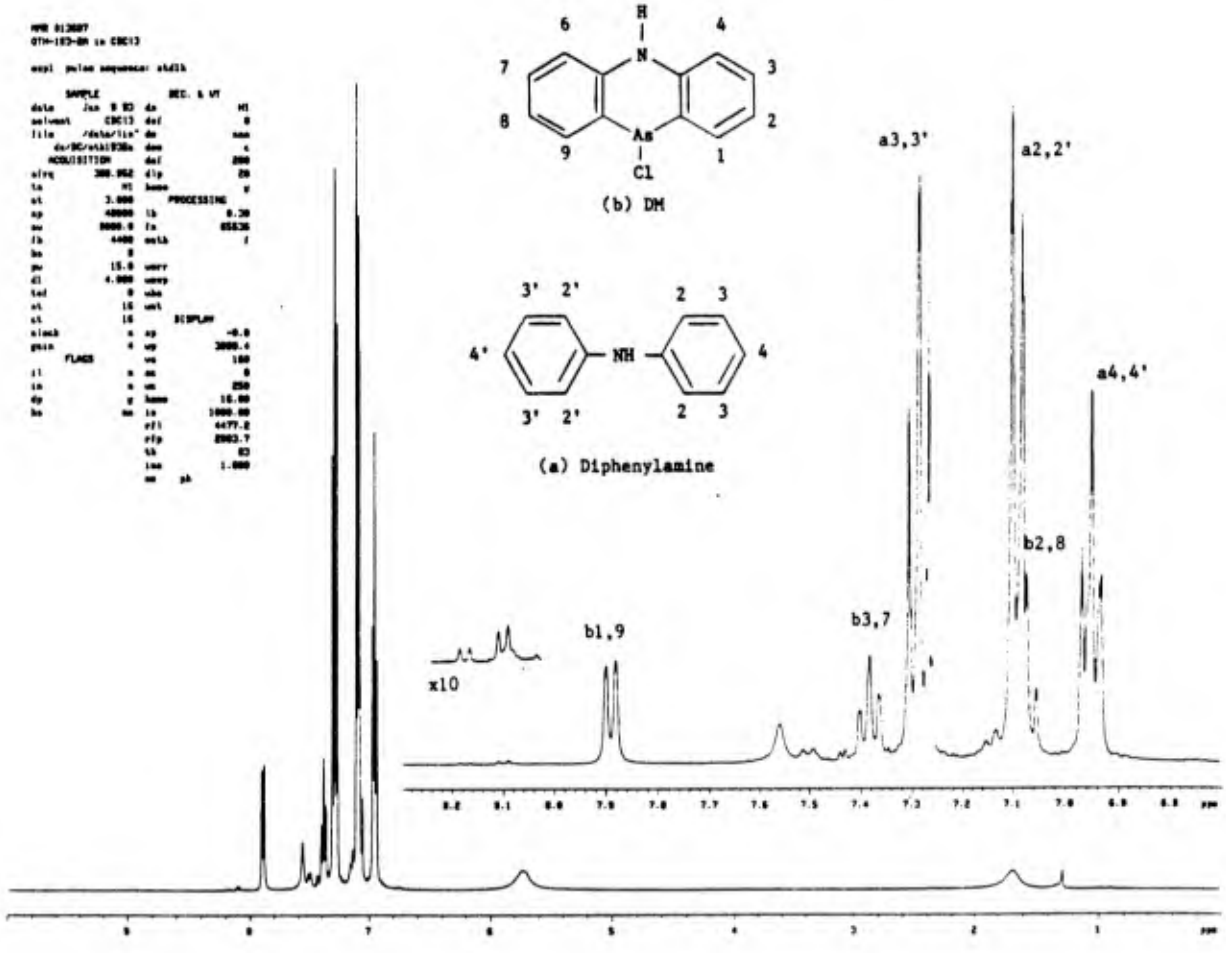
NMR-2. OTH-193-1a, $CDCl_3$ extract: ^1H, 400 MHz.



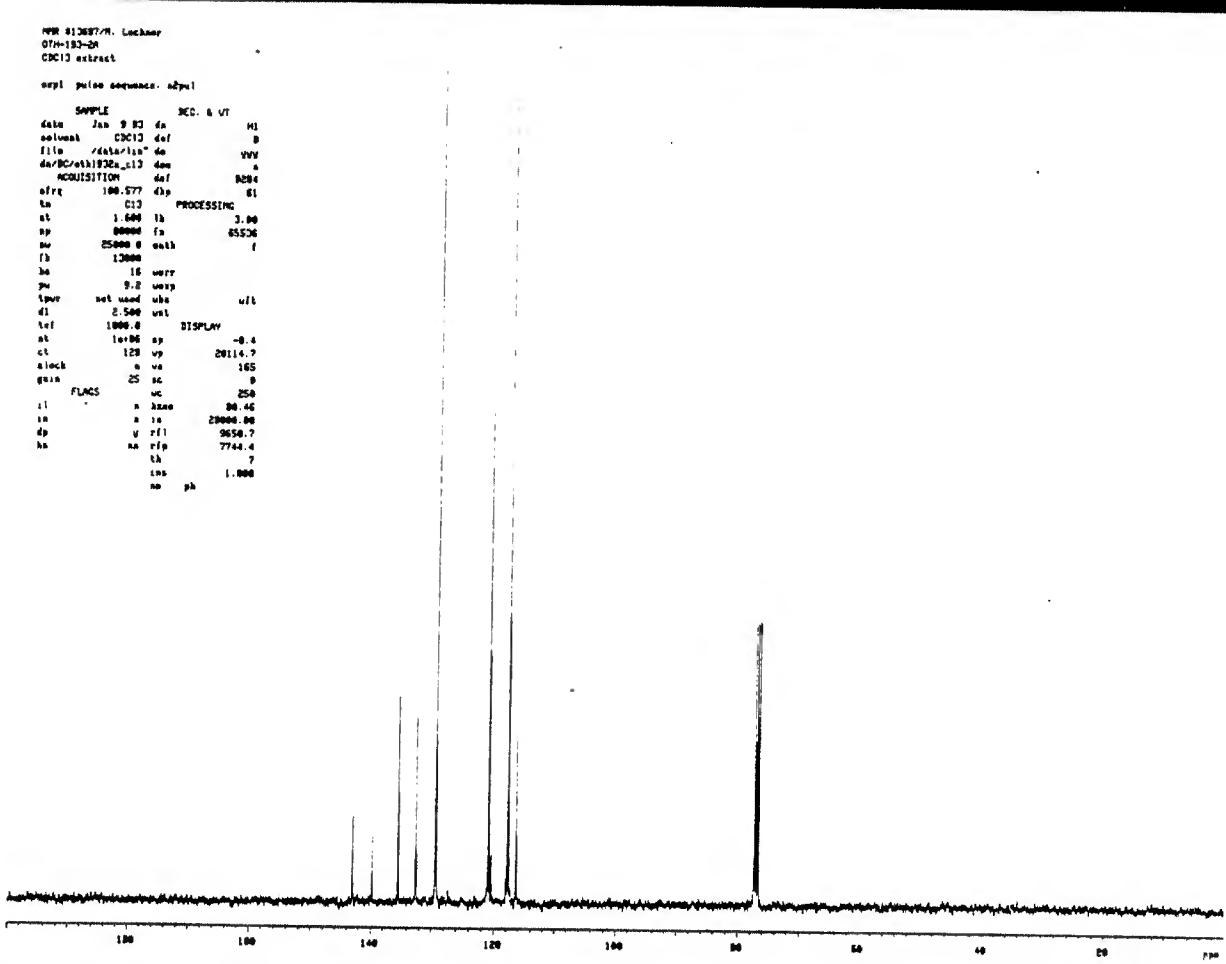
NMR-3. OTH-193-1b, $CDCl_3$ extract: ^1H, 400 MHz.



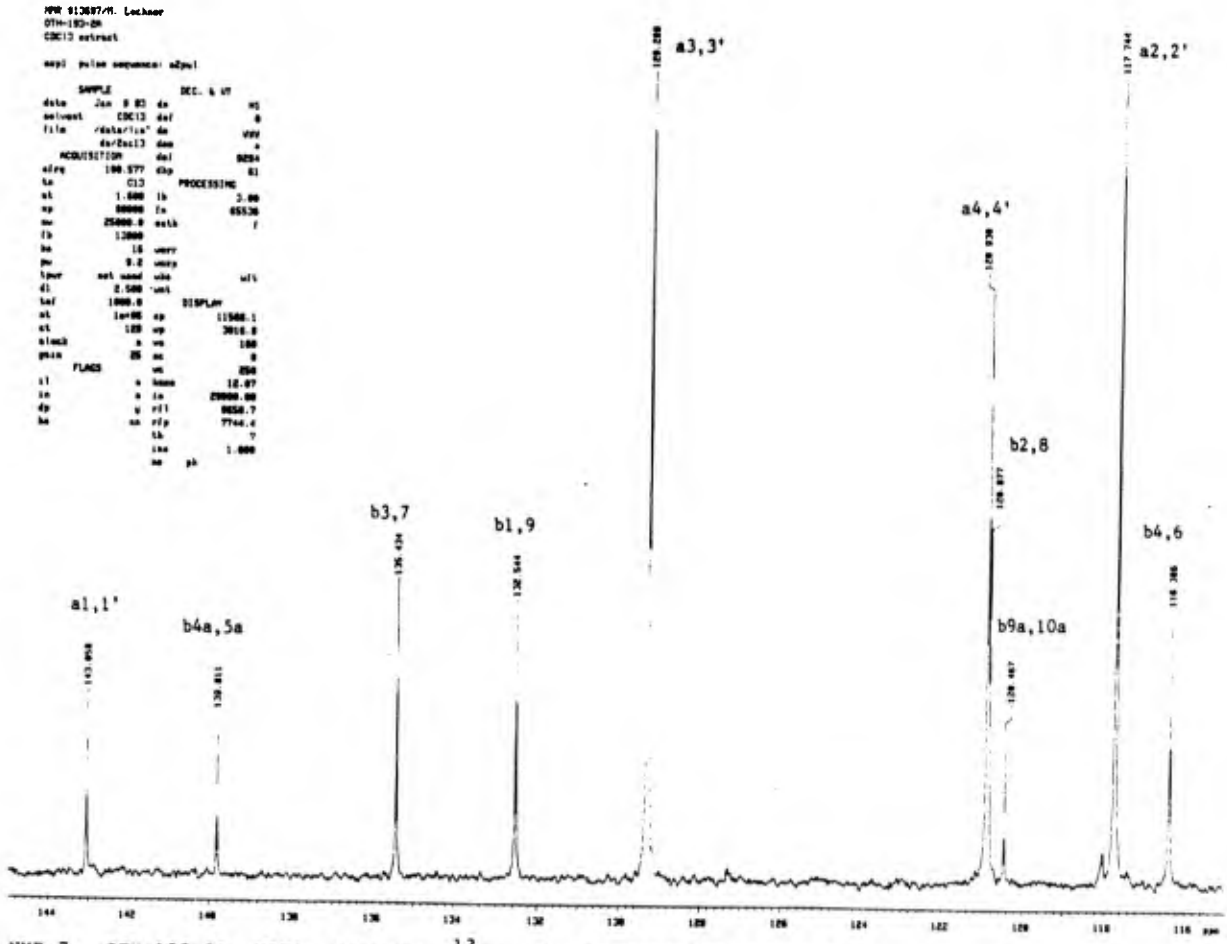
NMR-4. OTH-193-1b, D_2O extract: ^1H, 400 MHz.



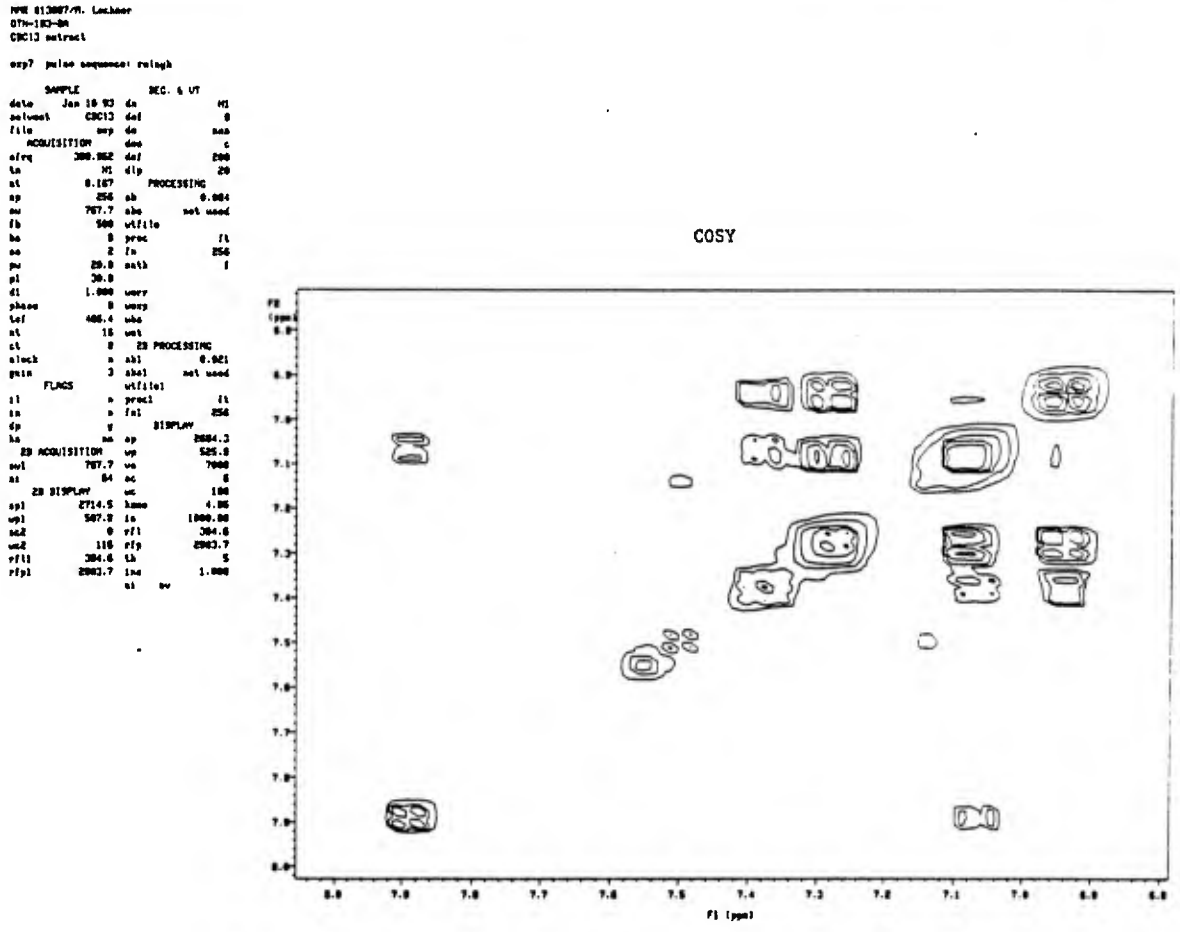
NMR-5. OTH-193-2a, $CDCl_3$ extract: ^1H, 400 MHz.



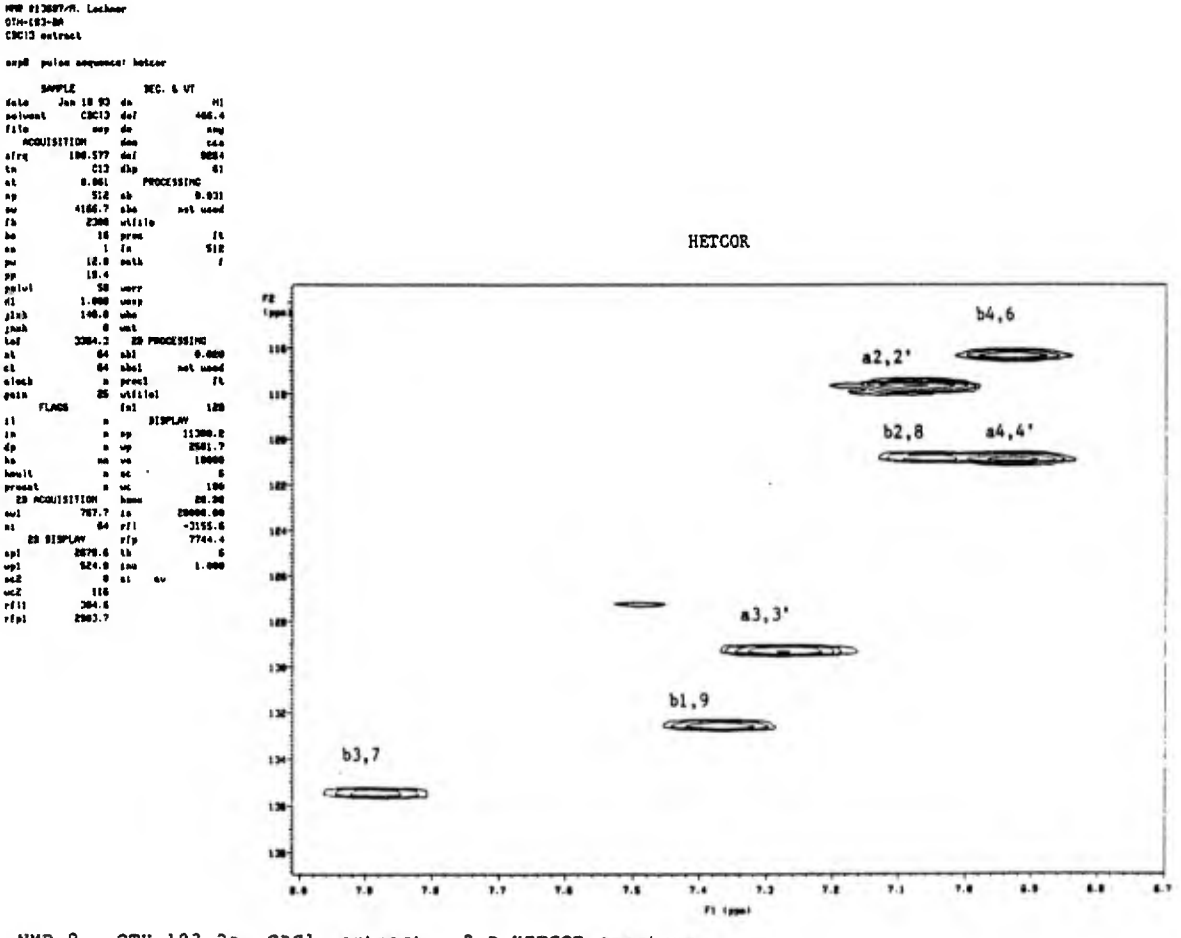
NMR-6. OTH-193-2a, $CDCl_3$ extract: ^{13}C, 100 MHz.



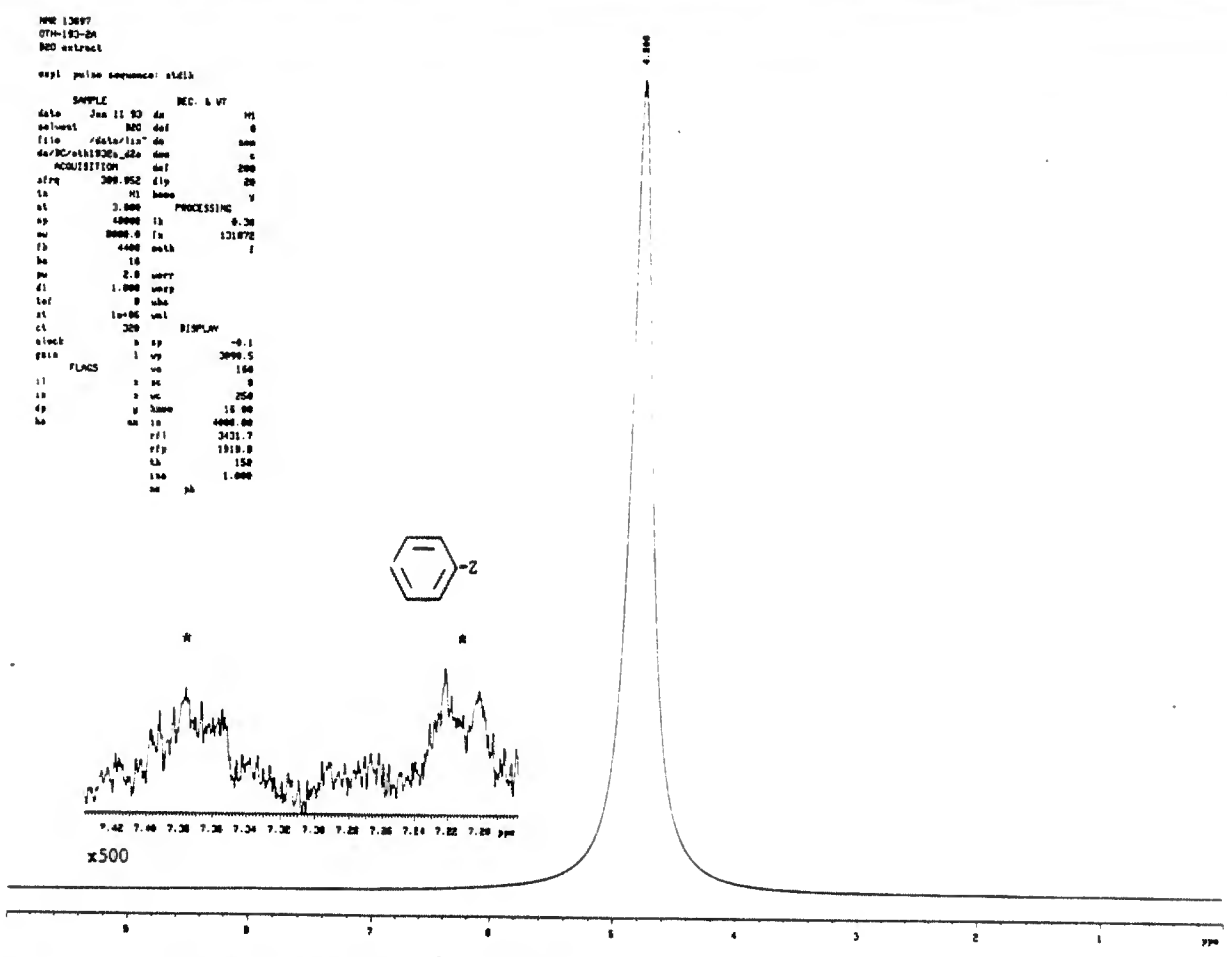
NMR-7. OTH-193-2a, $CDCl_3$ extract: ^{13}C, 100 MHz, expanded spectrum.



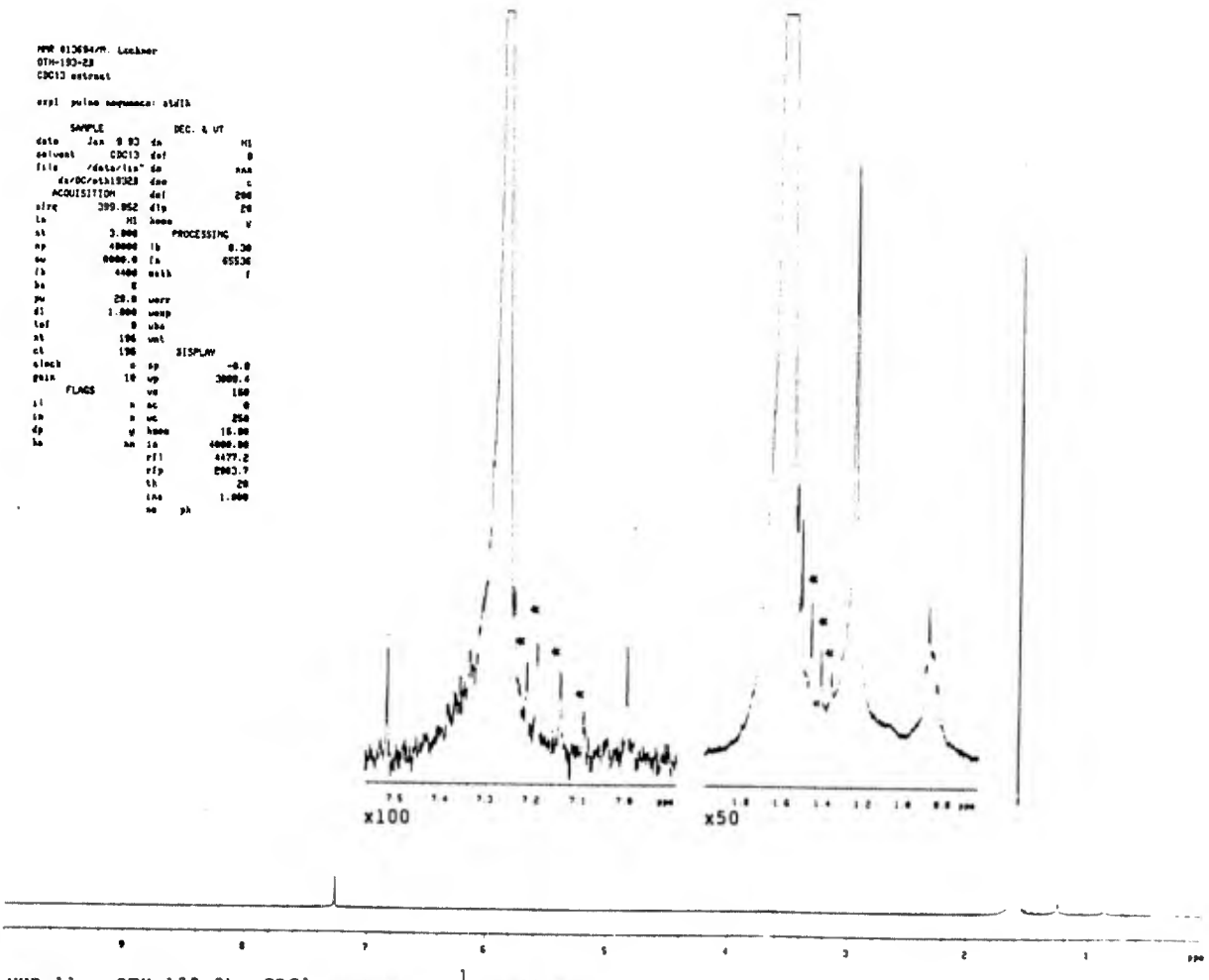
NMR-8. OTH-193-2a, $CDCl_3$ extract: 2-D COSY spectrum.



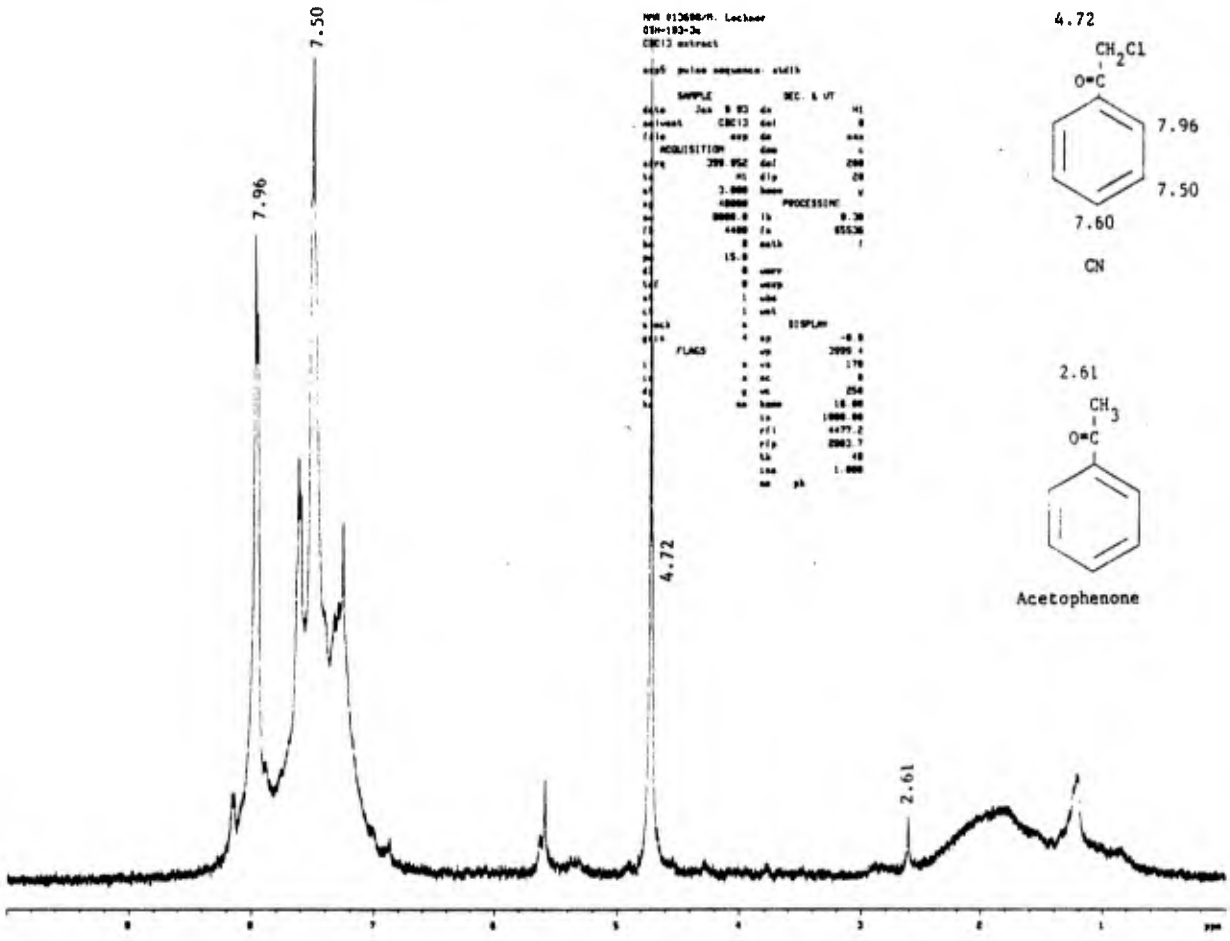
NMR-9. OTH-193-2a, $CDCl_3$ extract: 2-D HETCOR spectrum.



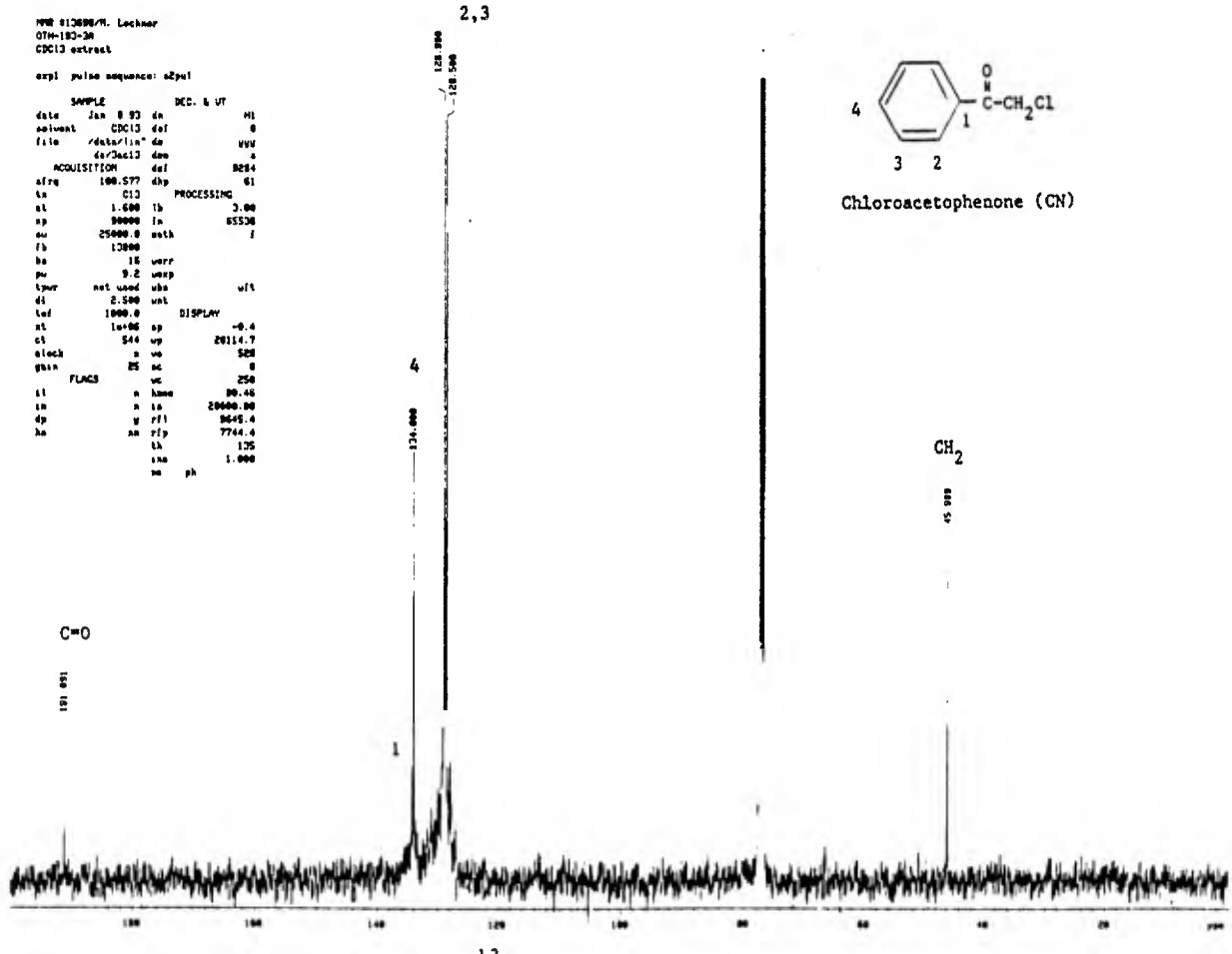
NMR-10. OTH-193-2a, D_2O extract: ^1H, 400 MHz.



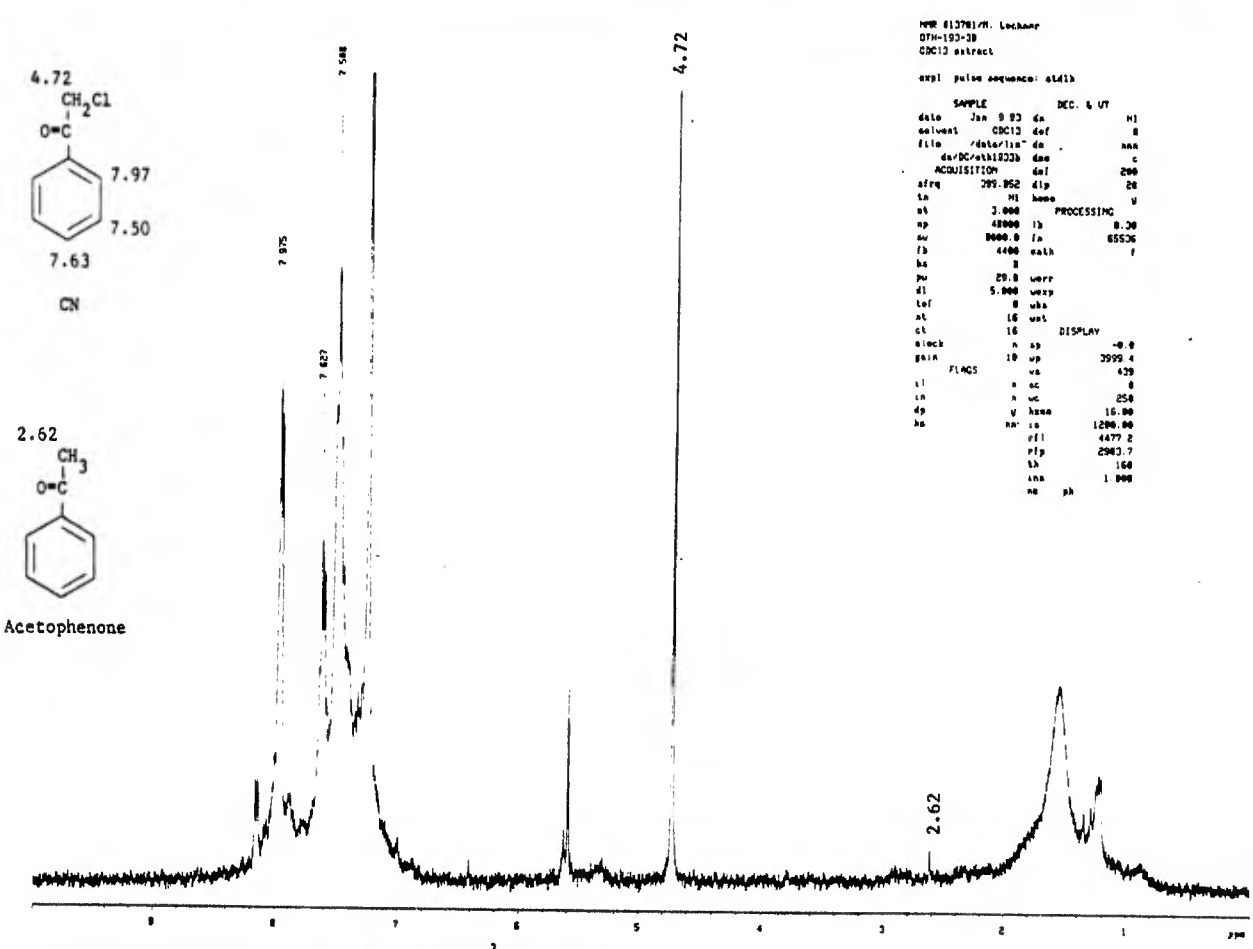
NMR-11. OTH-193-2b, $CDCl_3$ extract: ^1H, 400 MHz.



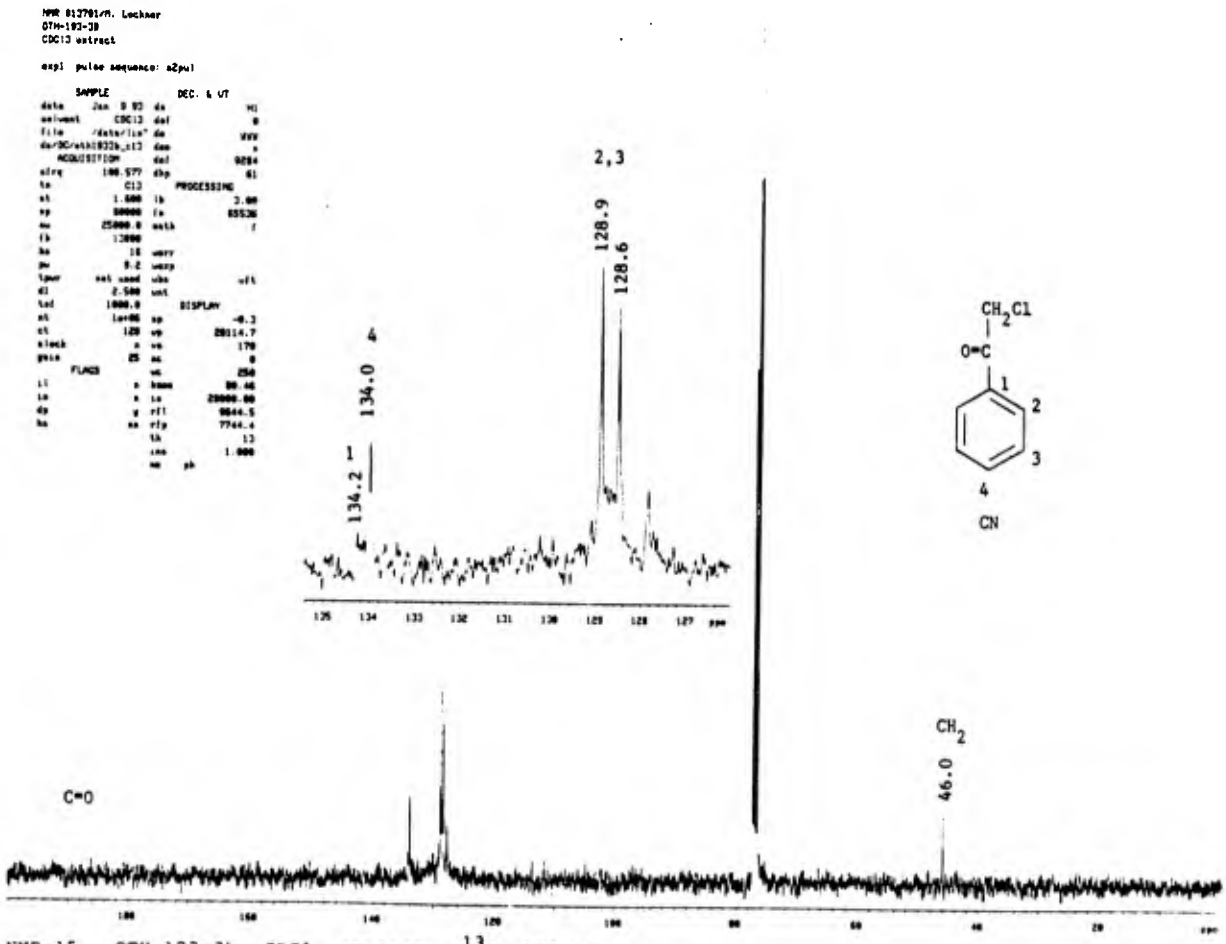
NMR-12. OTH-193-3a, $CDCl_3$ extract: ^1H, 400 HMz.



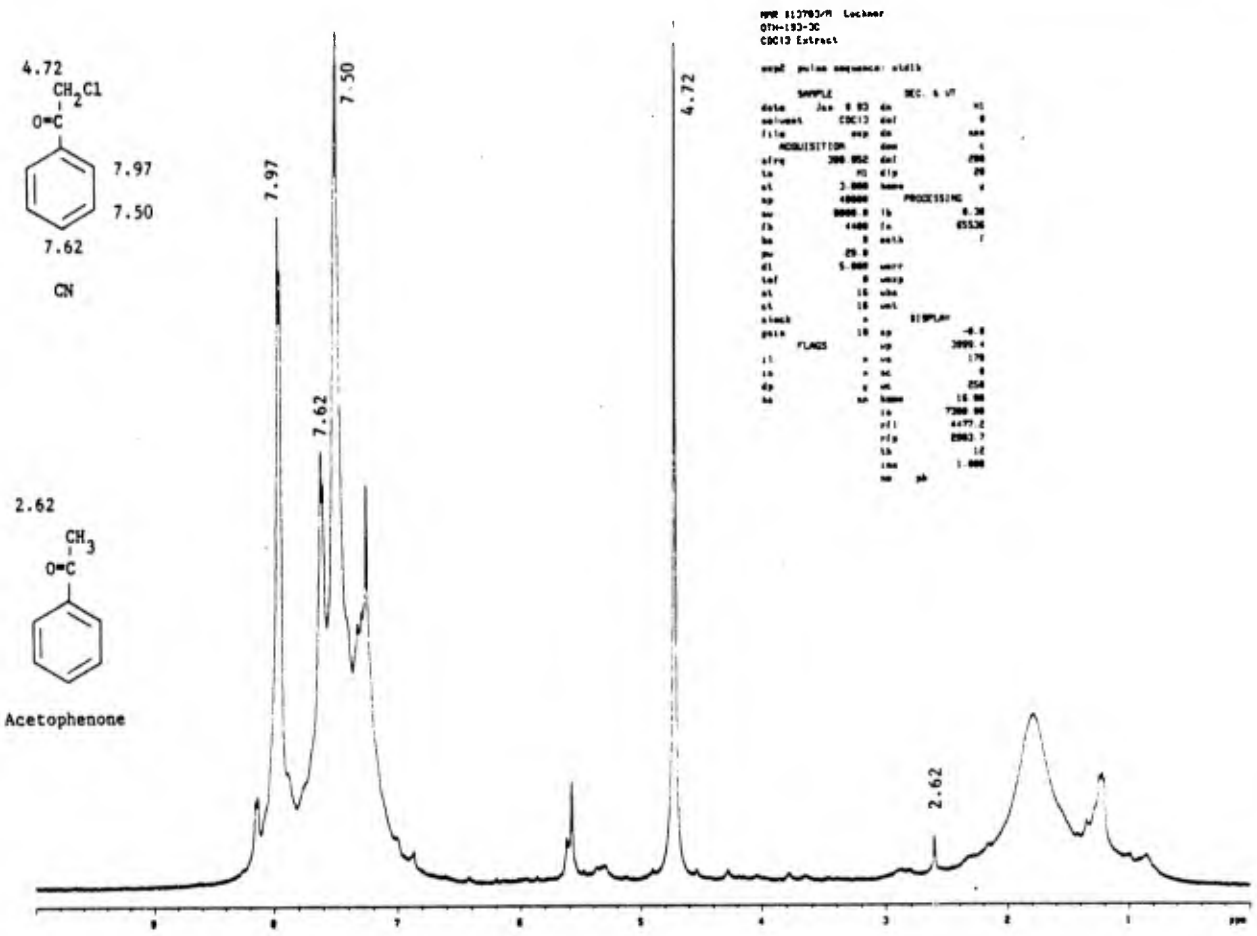
NMR-13. OTH-193-3a, $CDCl_3$ extract: ^{13}C, 100 MHz.



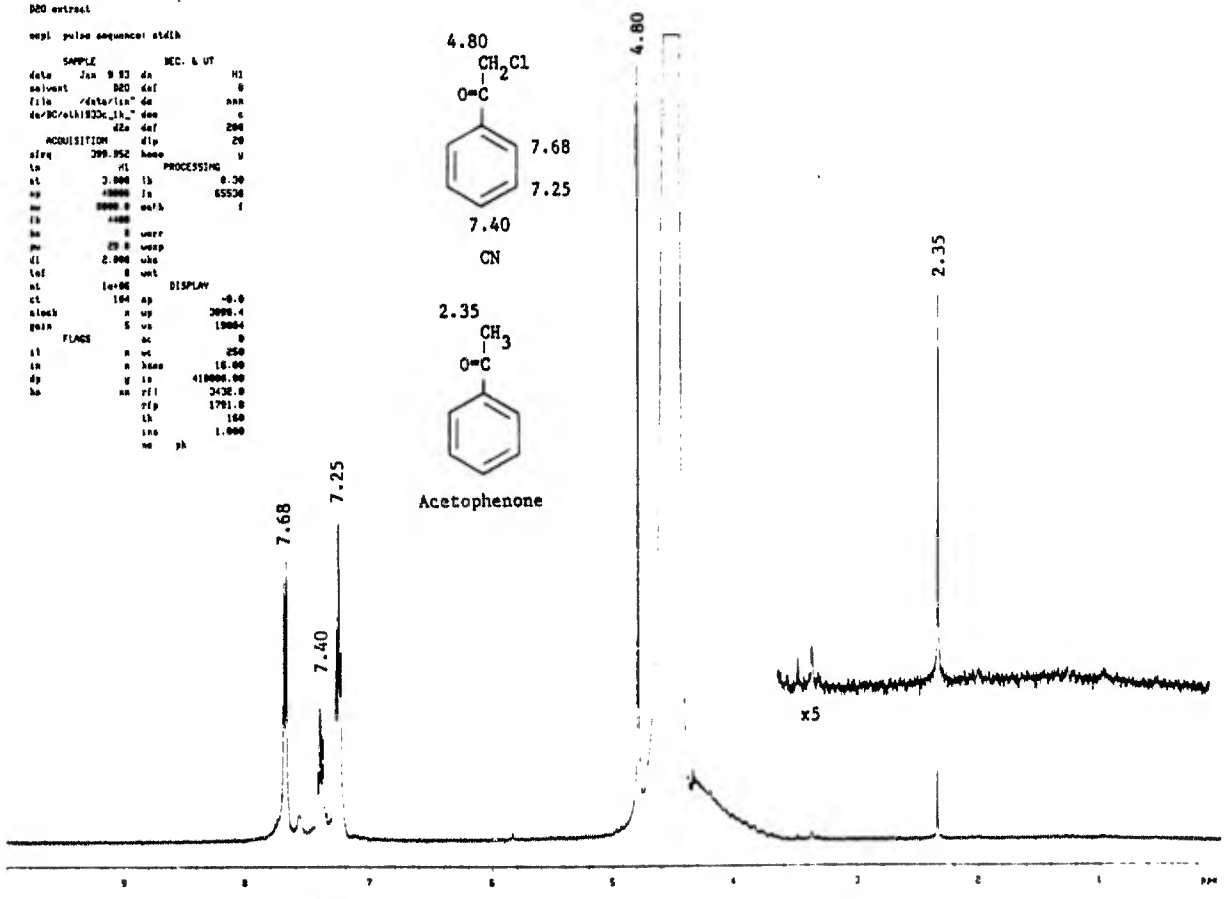
NMR-14. OTH-193-3b, $CDCl_3$ extract: ^1H, 400 MHz.



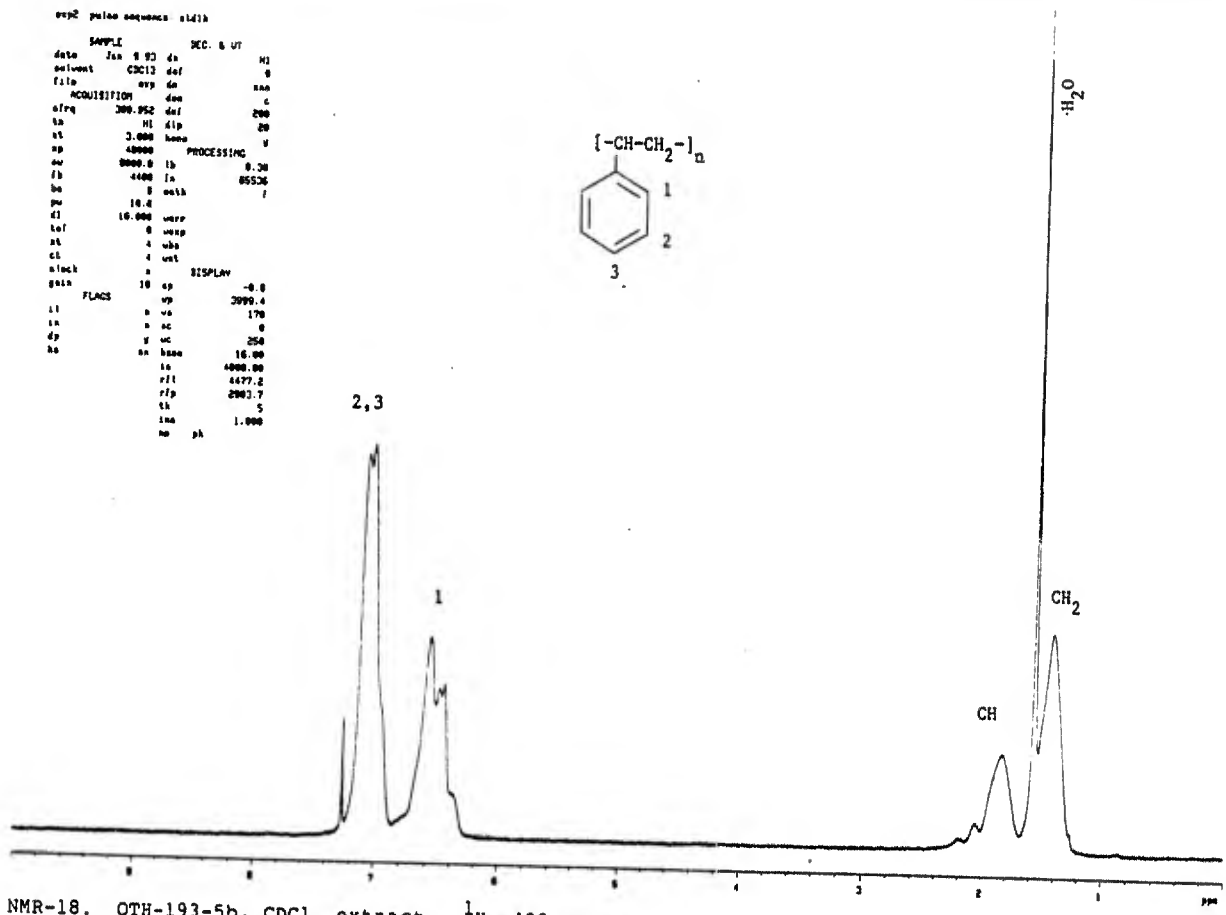
NMR-15. OTH-193-3b, $CDCl_3$ extract: ^{13}C, 100 MHz.



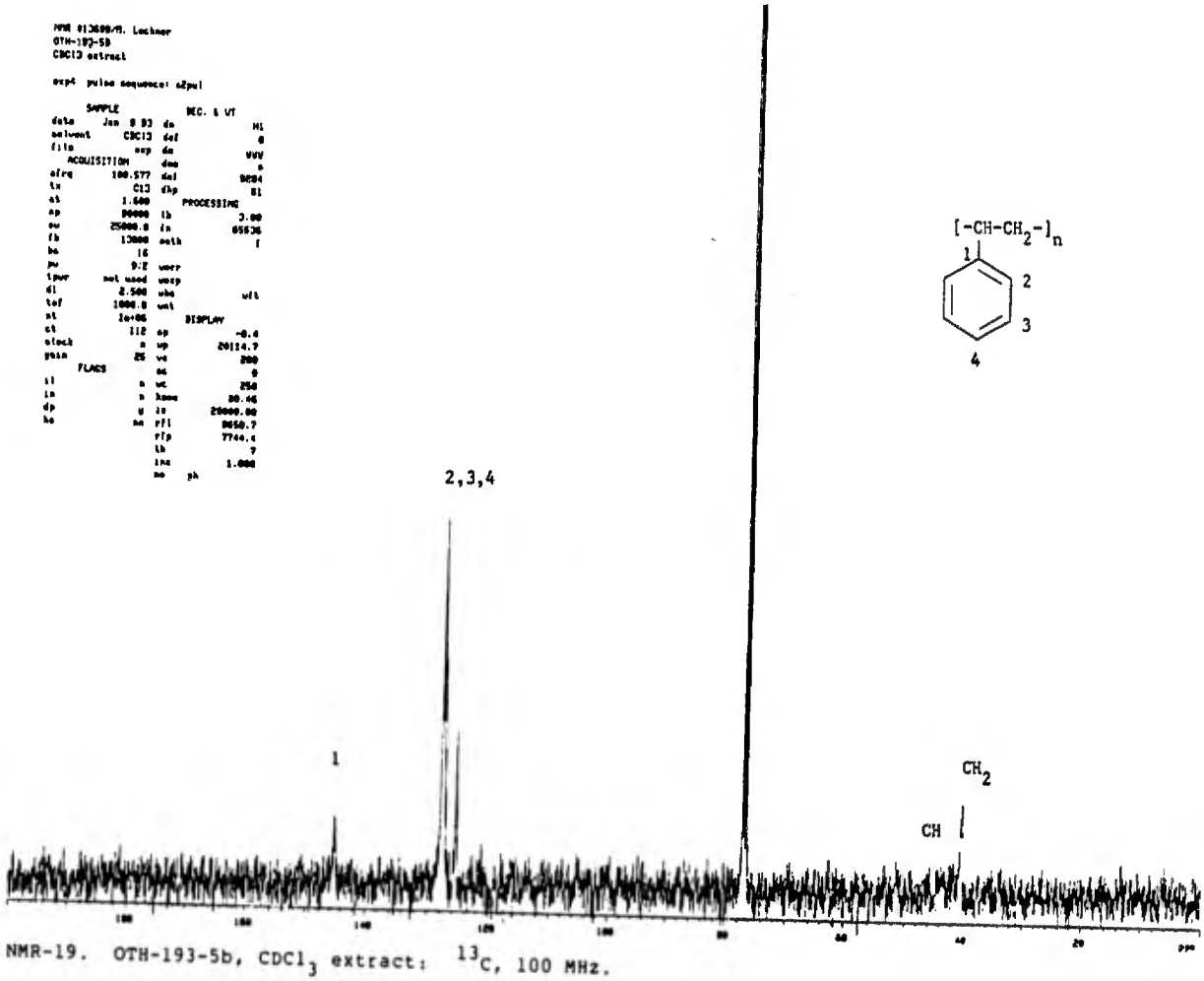
NMR-16. OTH-193-3c, $CDCl_3$ extract: ^1H, 400 MHz.



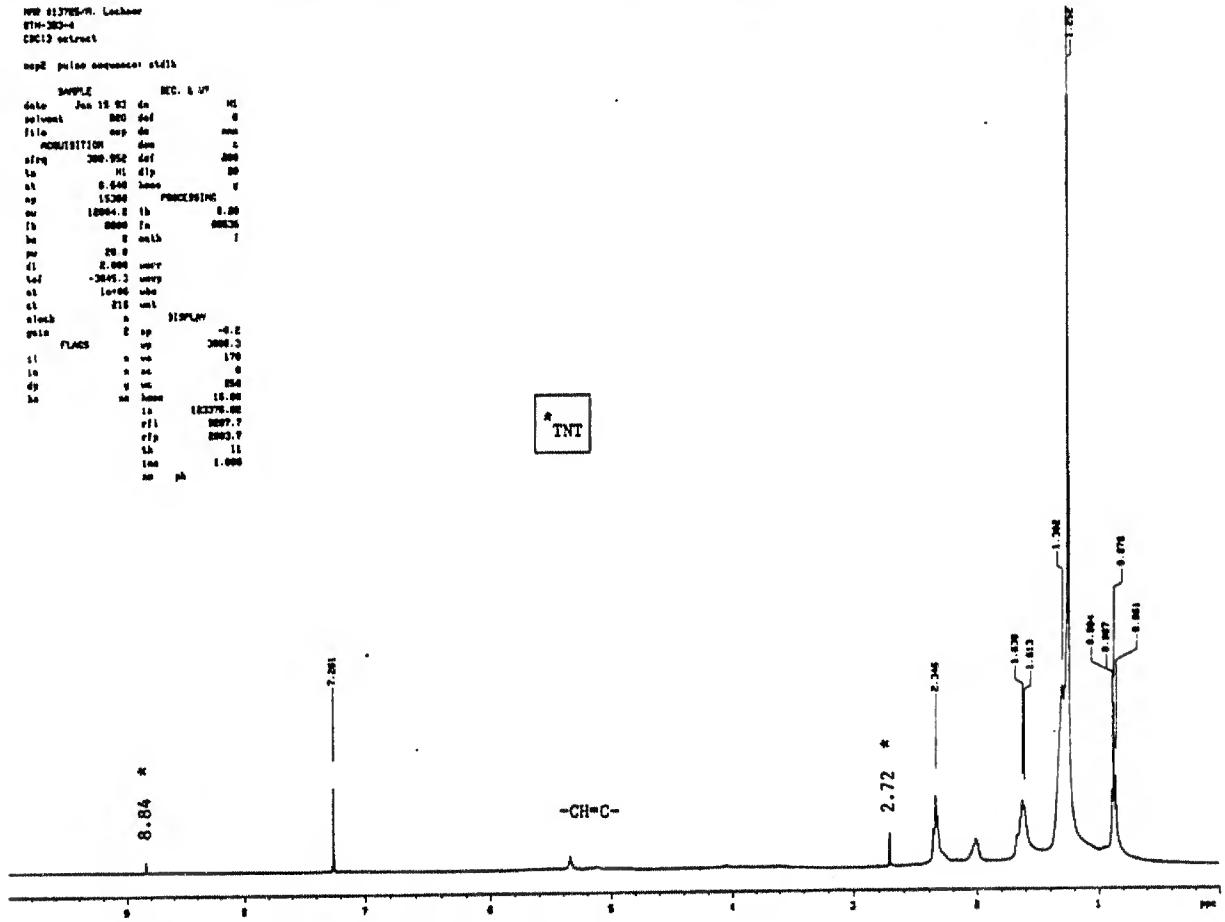
NMR-17. OTH-193-3c, D_2O extract: ^1H, 400 MHz.



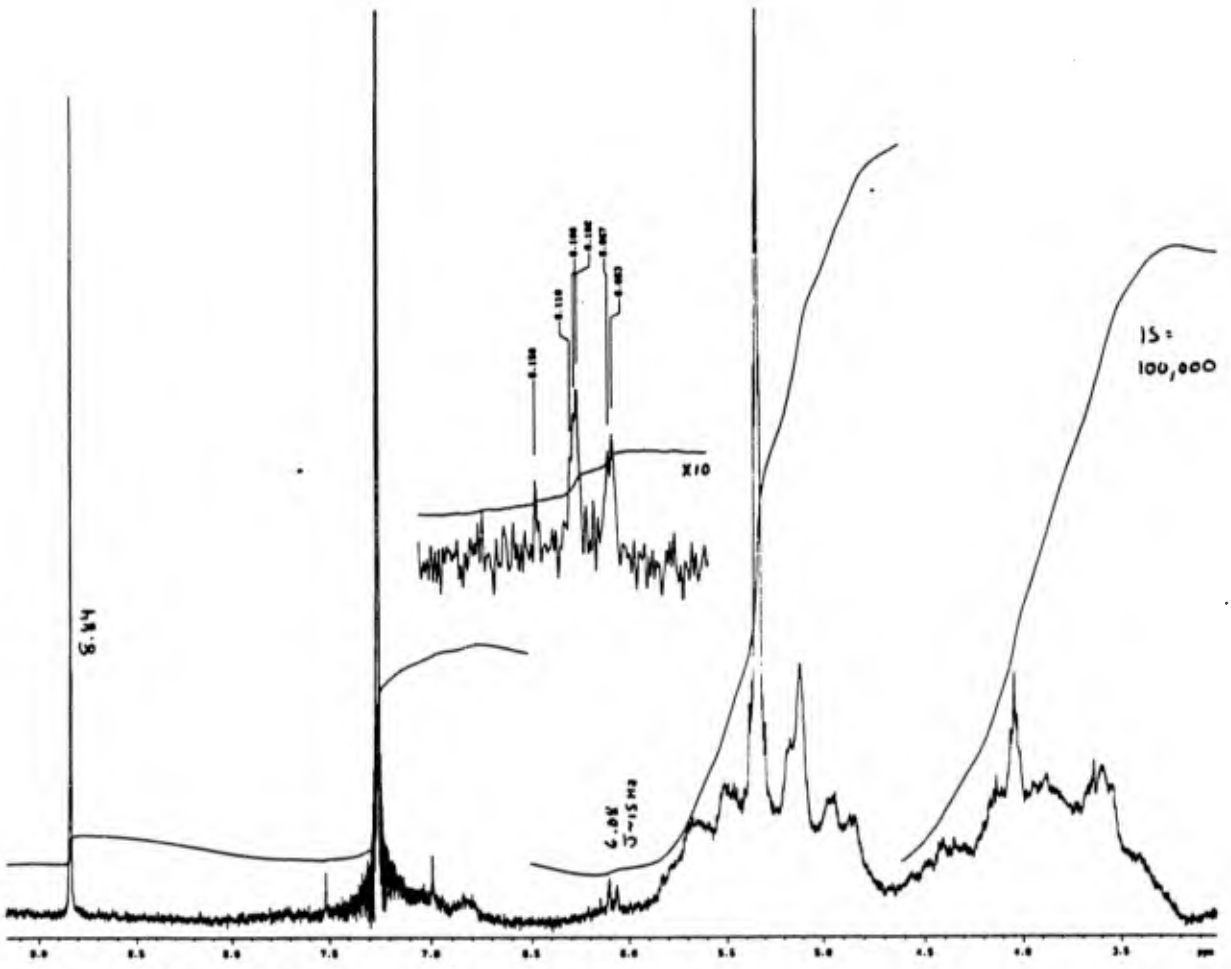
NMR-18. OTH-193-5b, $CDCl_3$ extract: ^1H, 400 MHz.



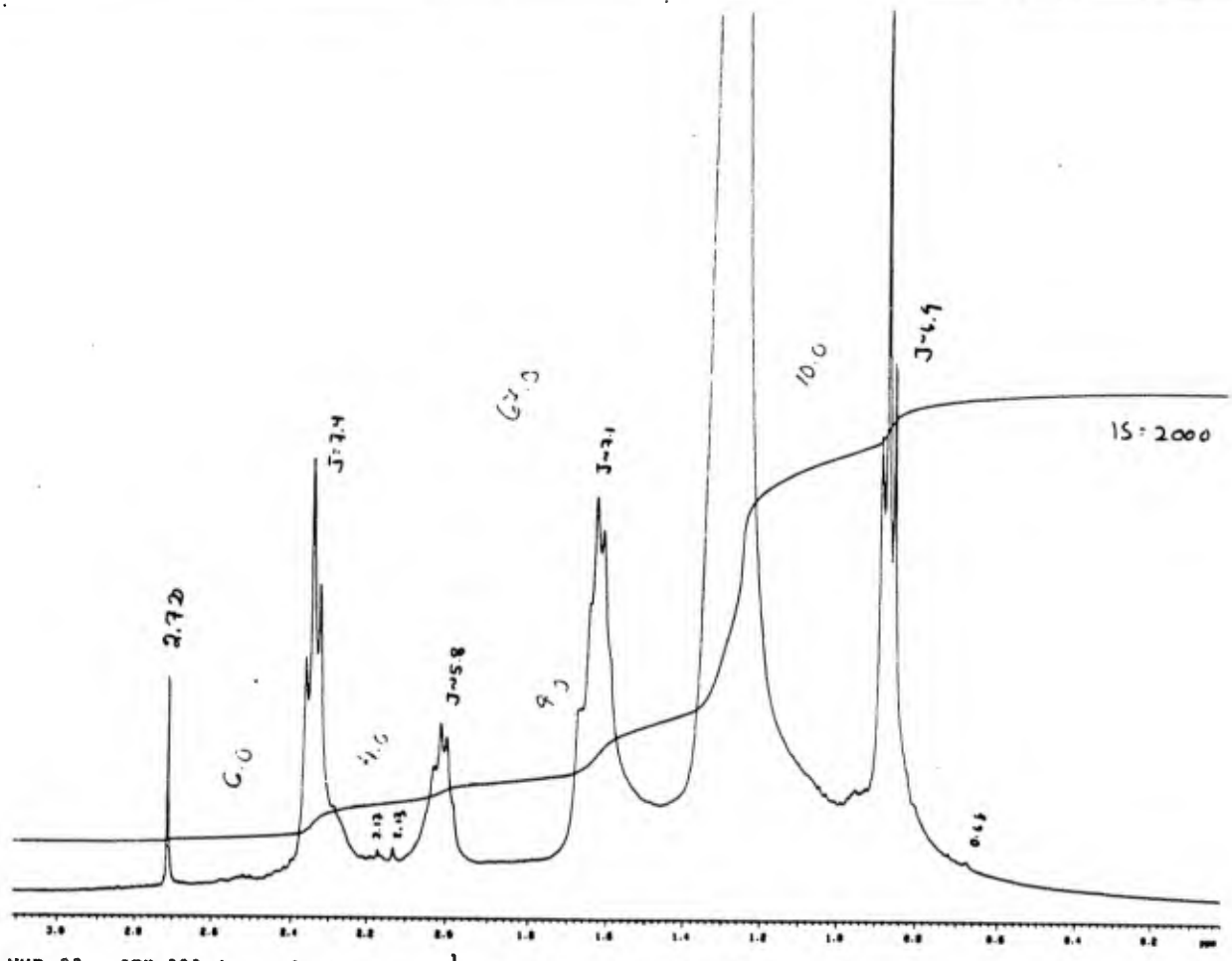
NMR-19. OTH-193-5b, $CDCl_3$ extract: ^{13}C, 100 MHz.



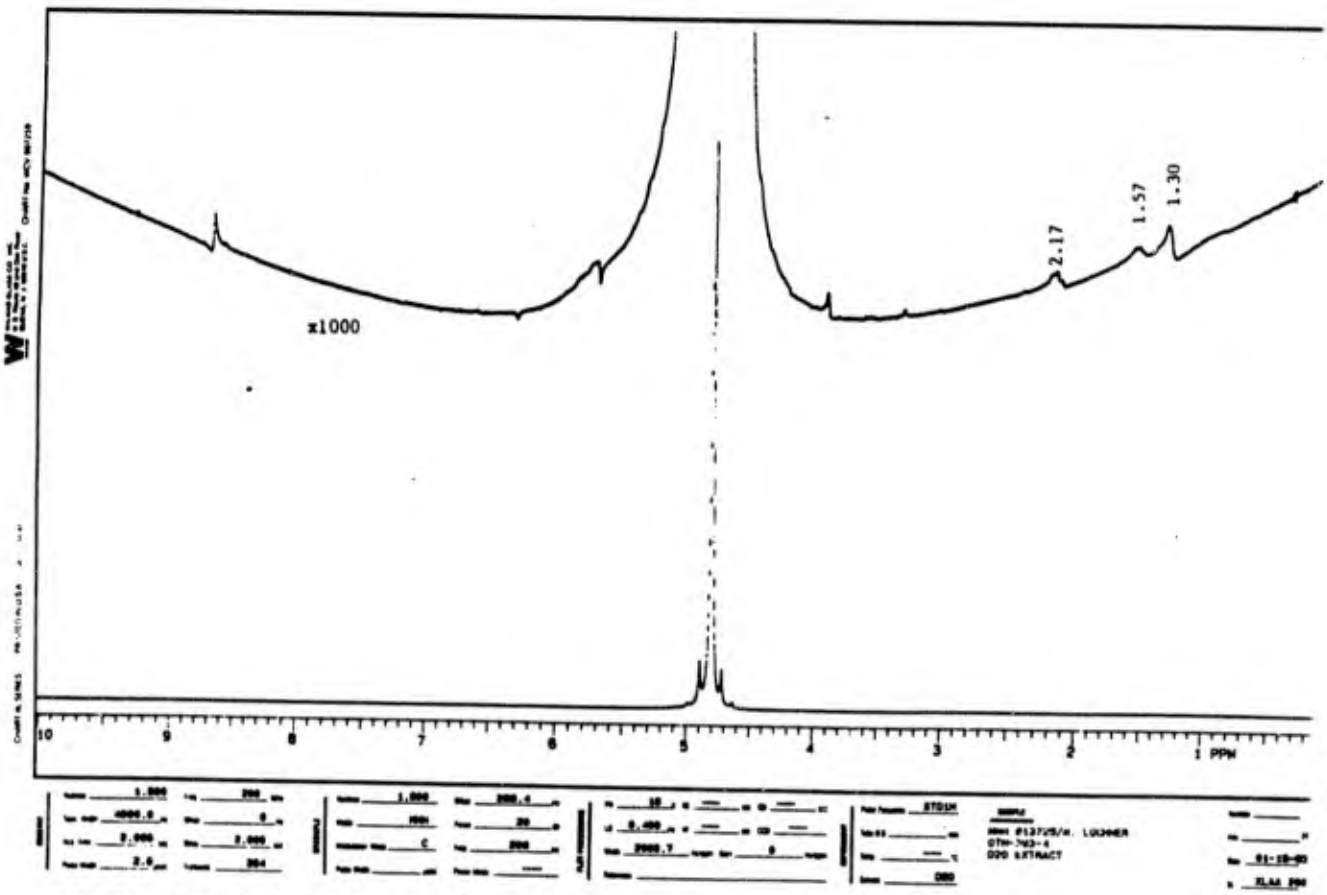
NMR-20. OTH-393-4, $CDCl_3$ extract: ^1H, 400 MHz.



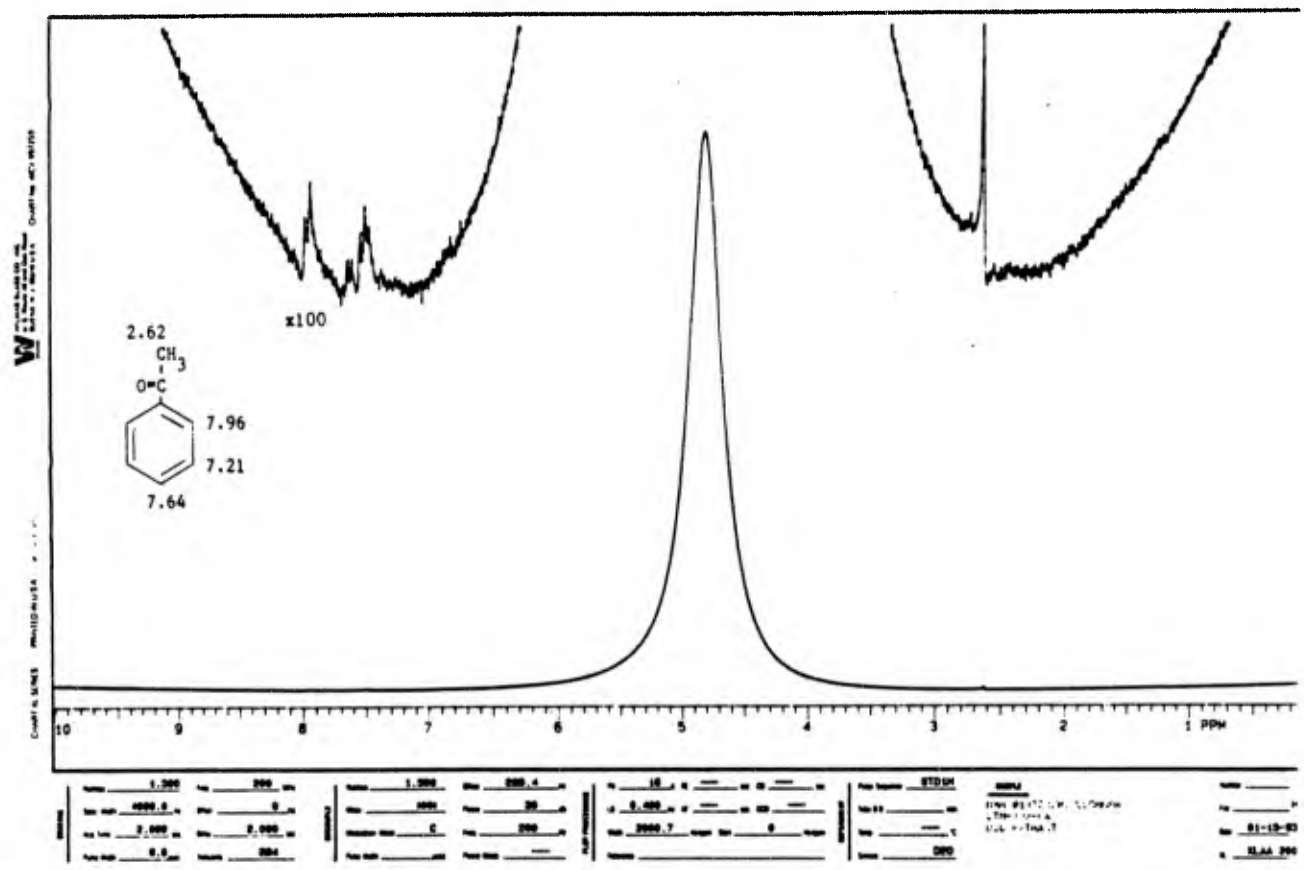
NMR-21. OTH-393-4, $CDCl_3$ extract: ^1H, 400 MHz.



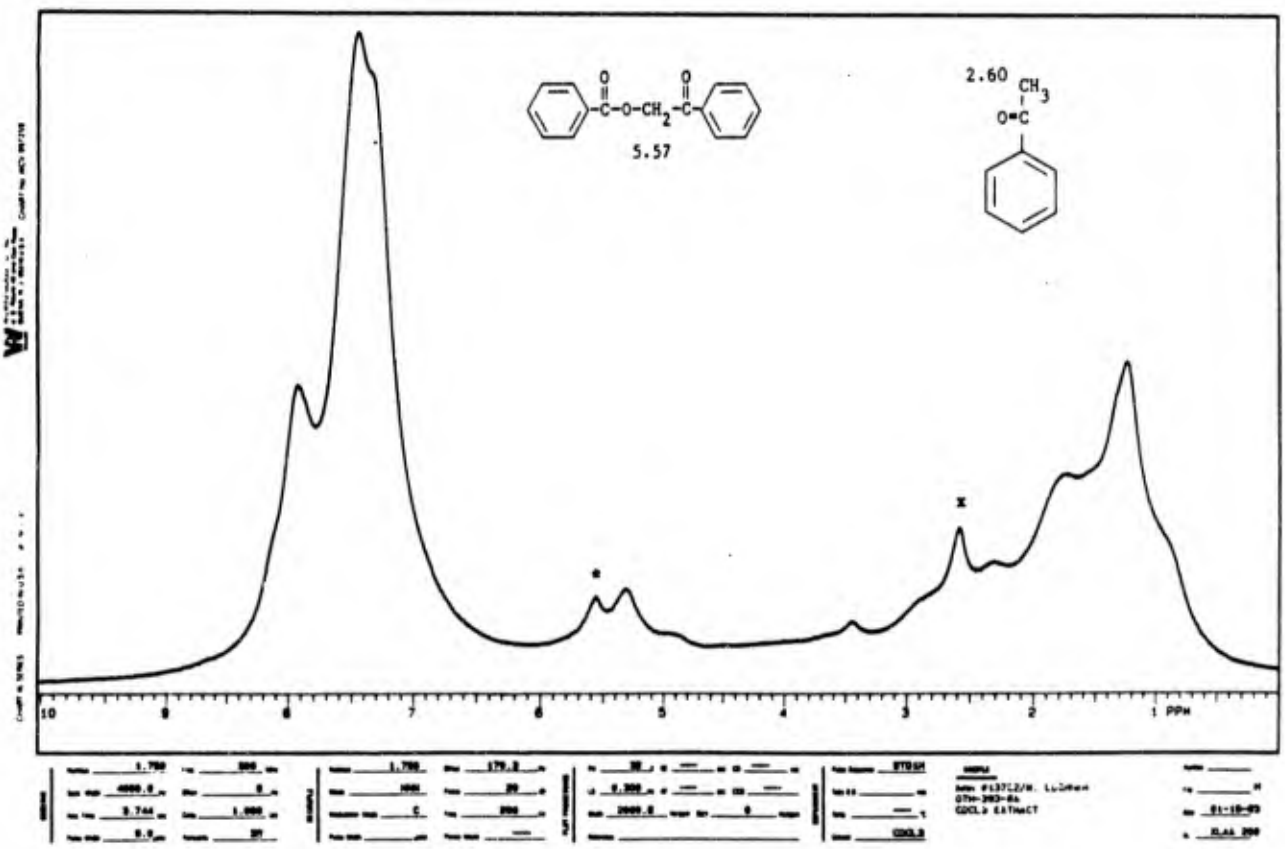
NMR-22. OTH-393-4, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



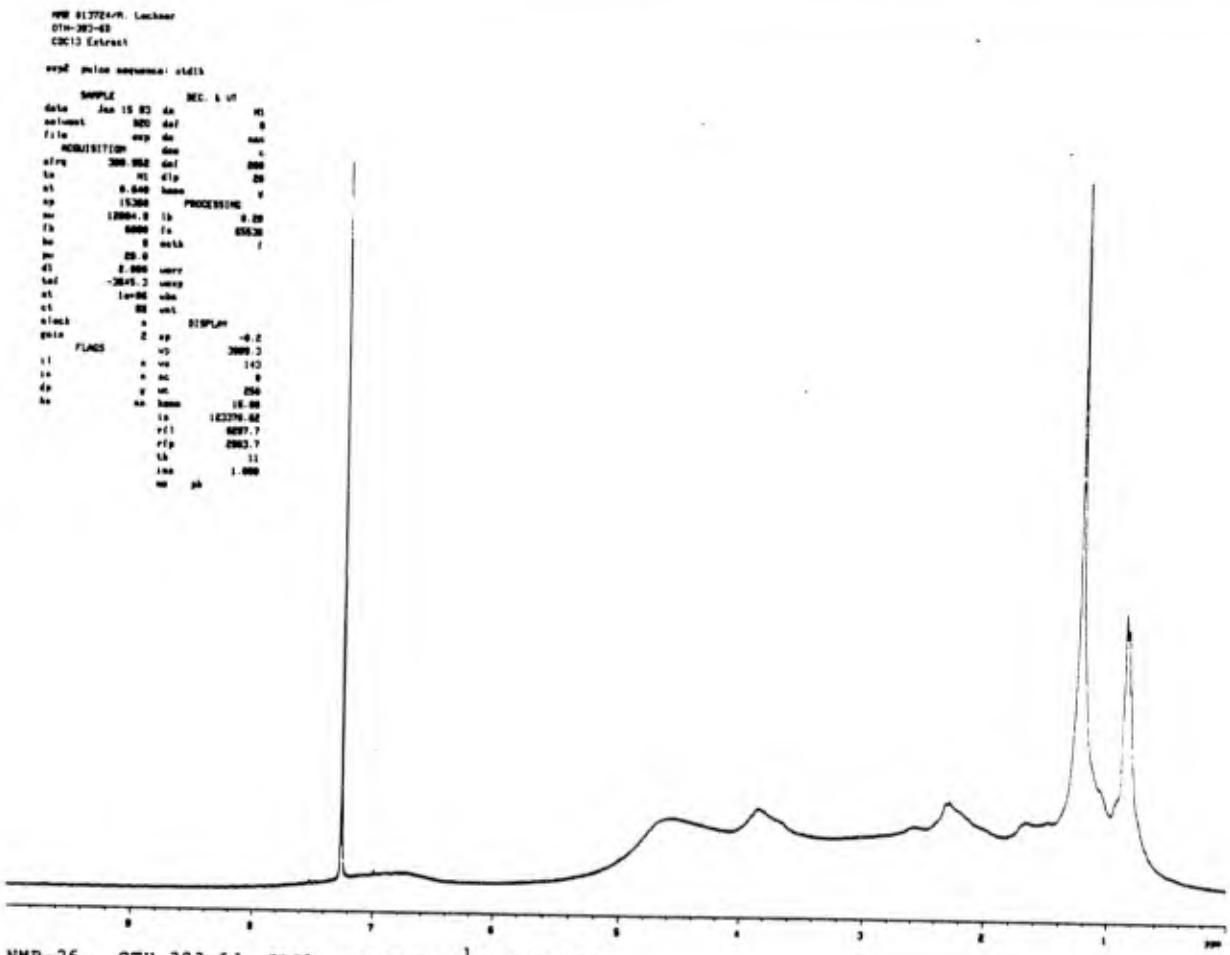
NMR-23. OTH-393-4, D_2O extract, ^1H, 200 MHz.



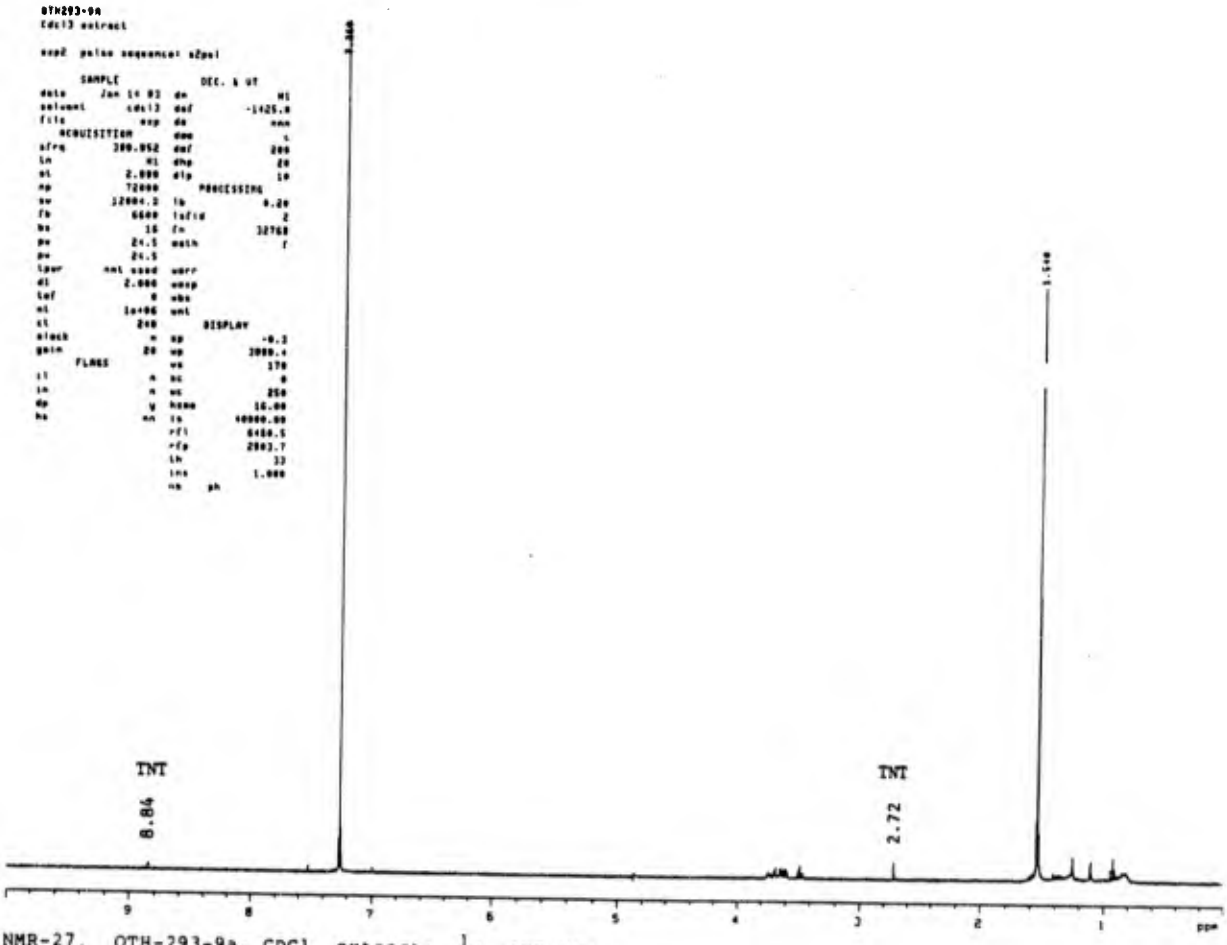
NMR-24. OTH-393-6a, D_2O extract: ^1H, 200 MHz.



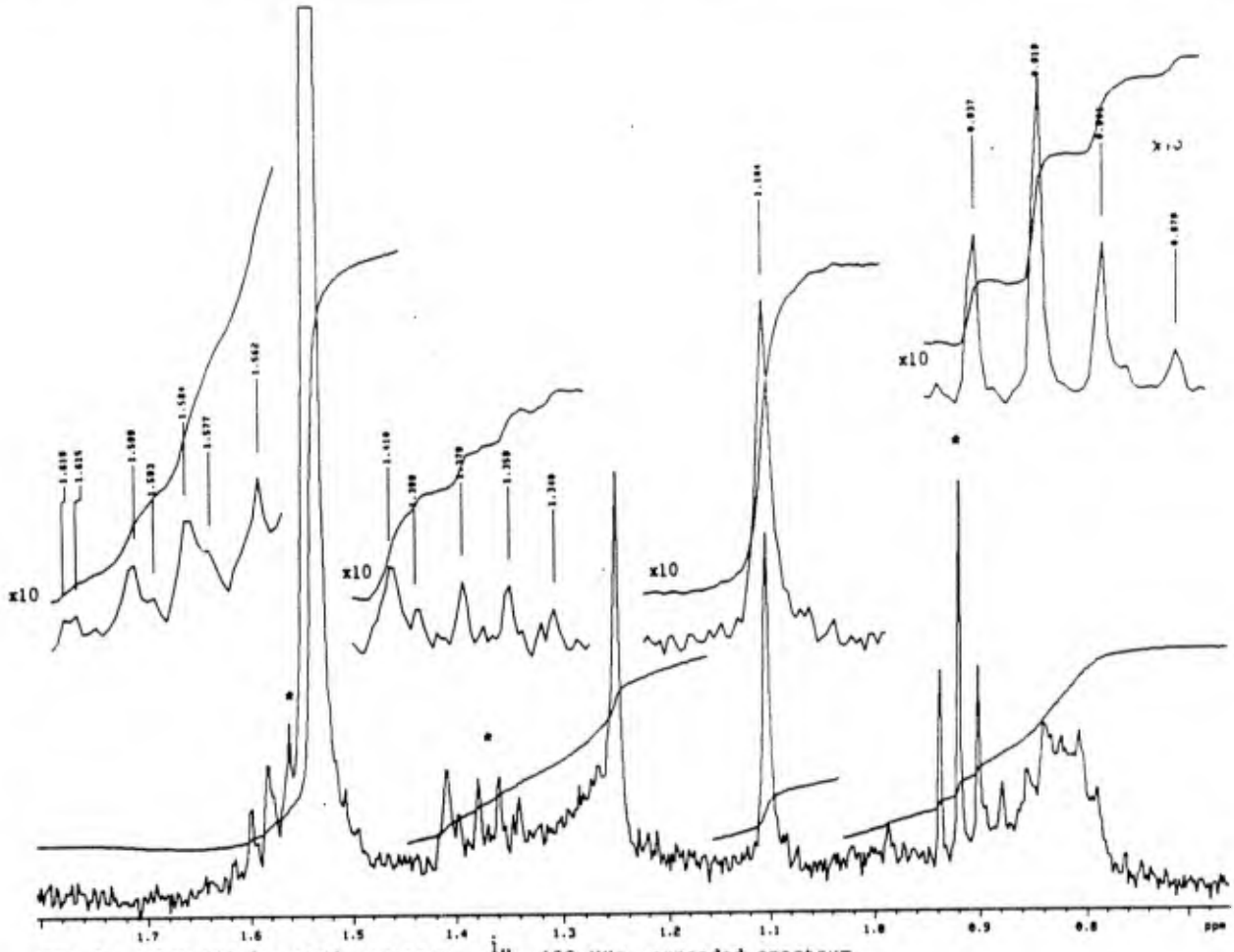
NMR-25. OTH-393-6a, $CDCl_3$ extract: ^1H, 200 MHz.



NMR-26. OTH-393-6d, $CDCl_3$ extract: ^1H, 400 MHz.



NMR-27. OTH-293-9a, $CDCl_3$ extract: ^1H, 400 MHz.



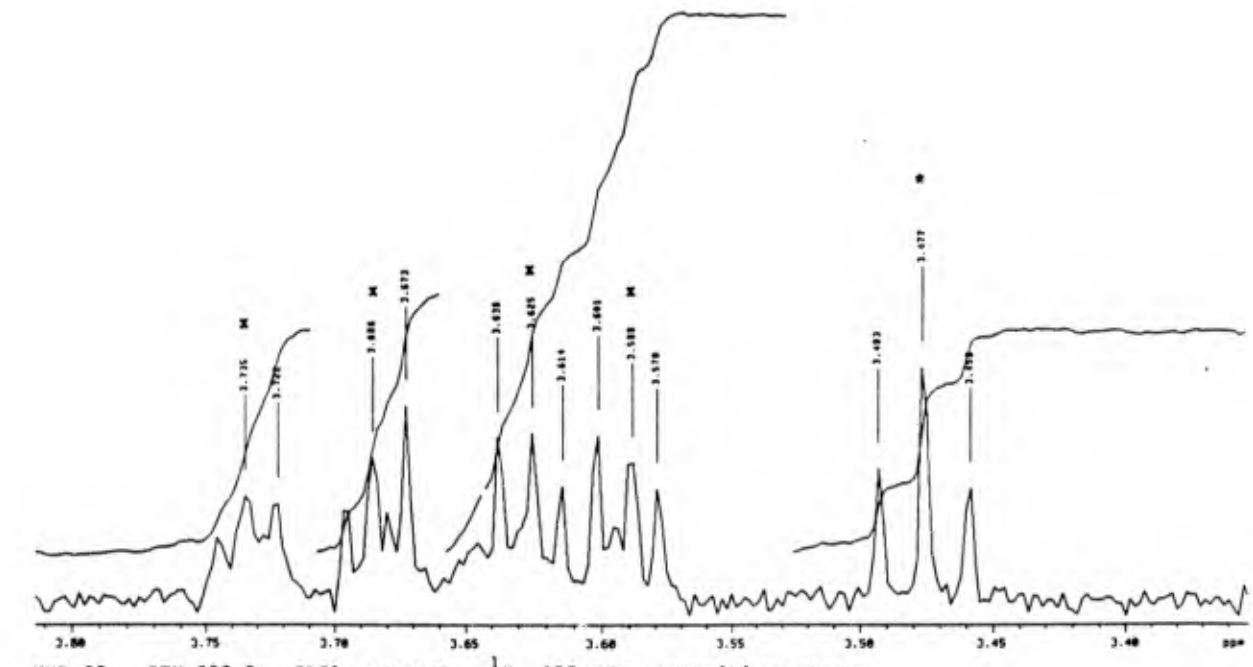
NMR-28. OTH-293-9a, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.

$CH_3 - CH_2 - CH_2 - CH_2 - Z$

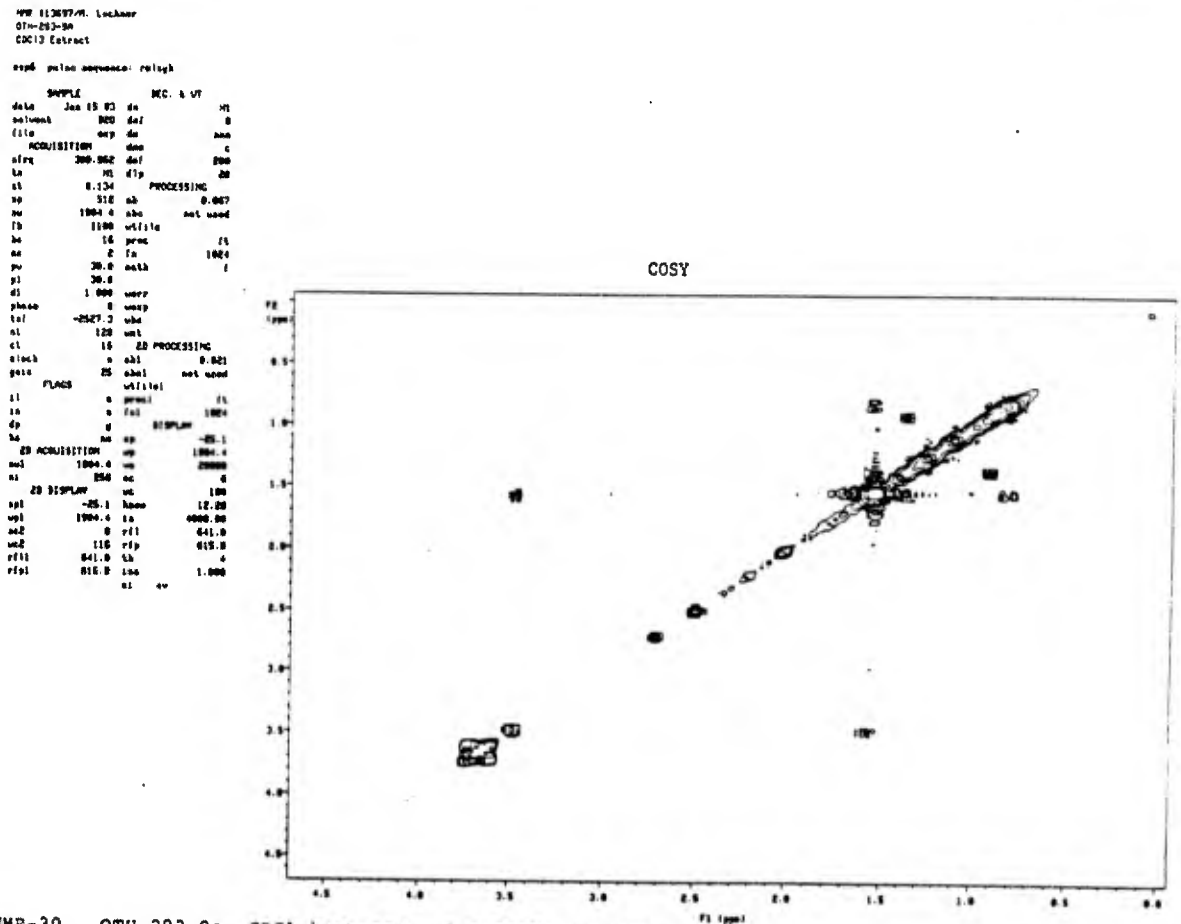
0.92 1.36 1.55 3.47

$Y - CH_2 - CH_2 - Y_1 - CH_2 - CH_2 - Y_2$

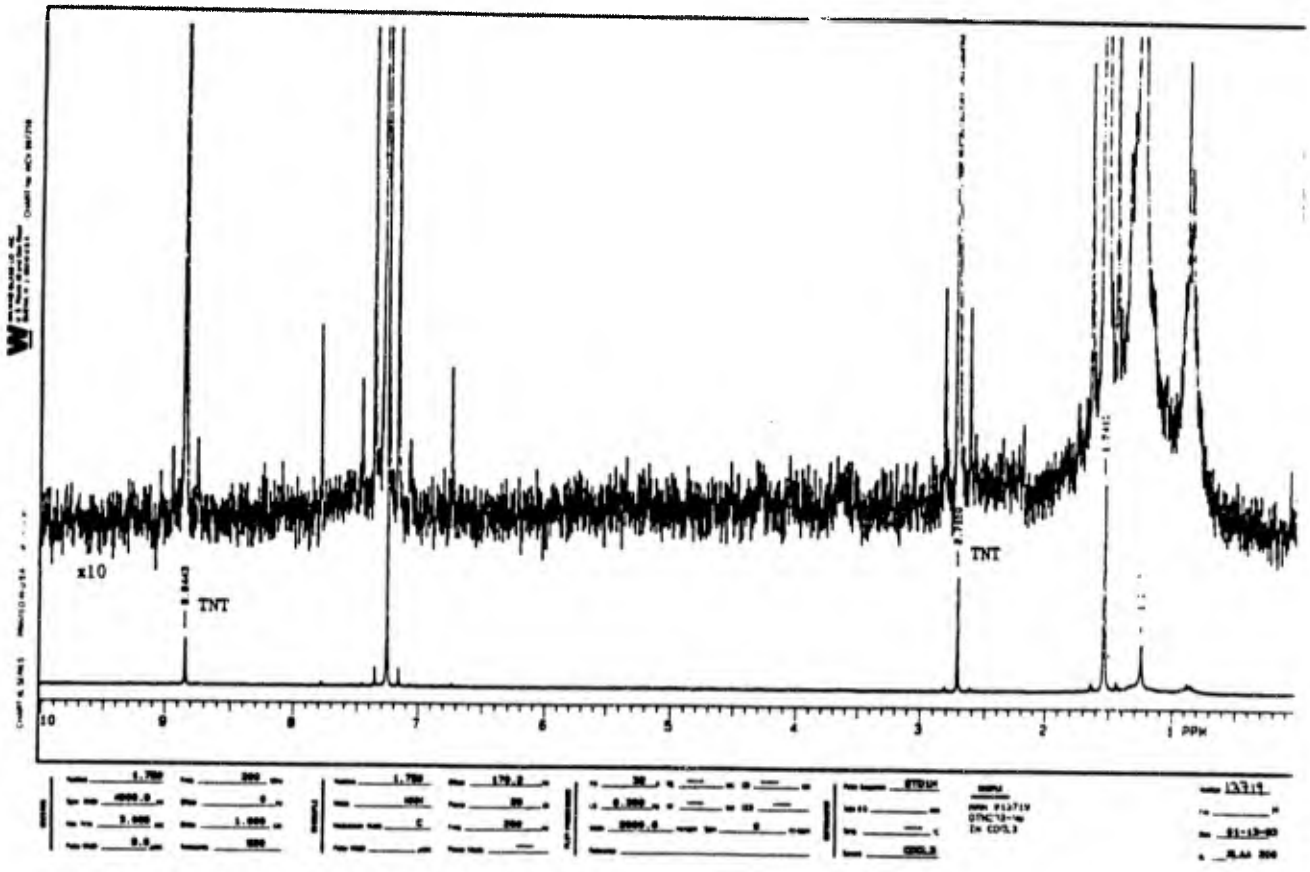
3.59 3.68 3.74 3.63



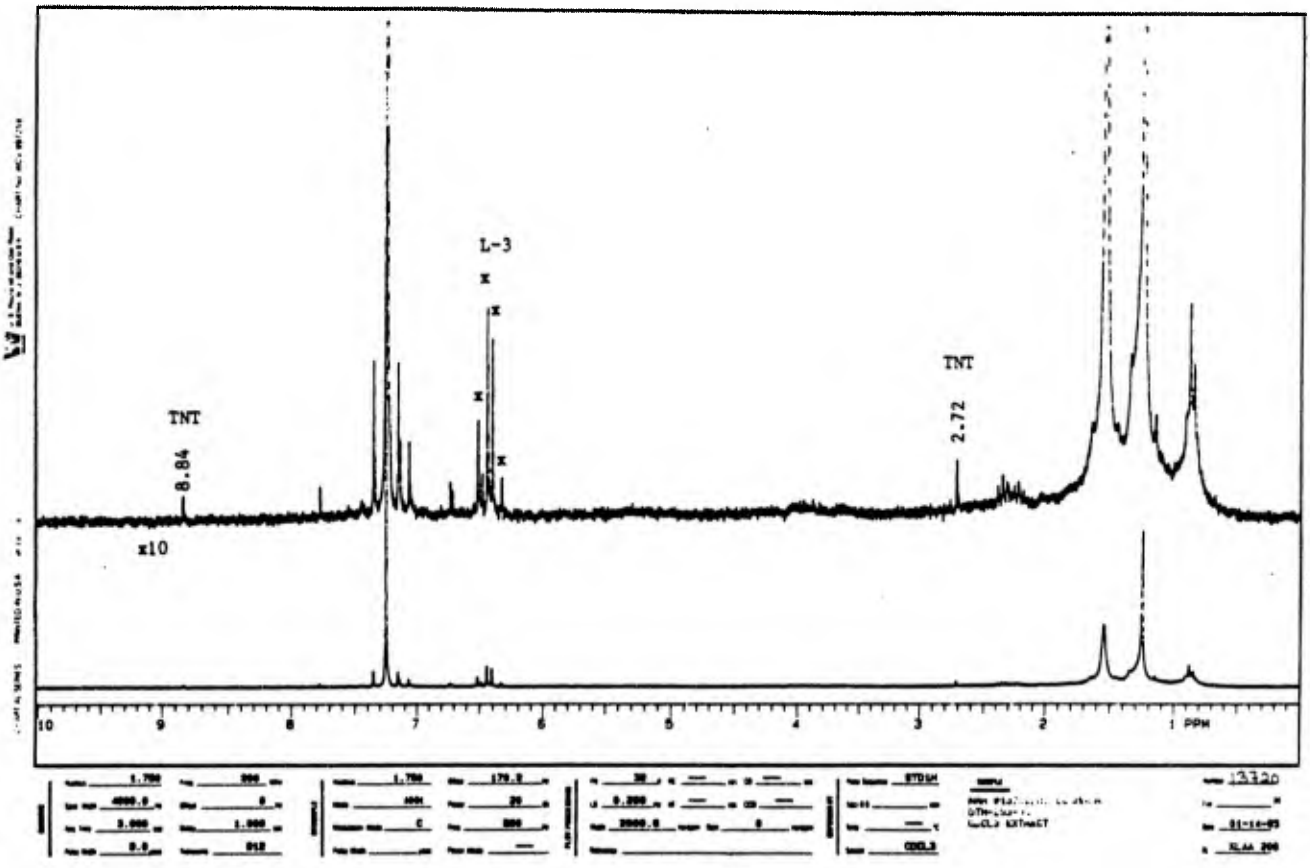
NMR-29. OTH-293-9a, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



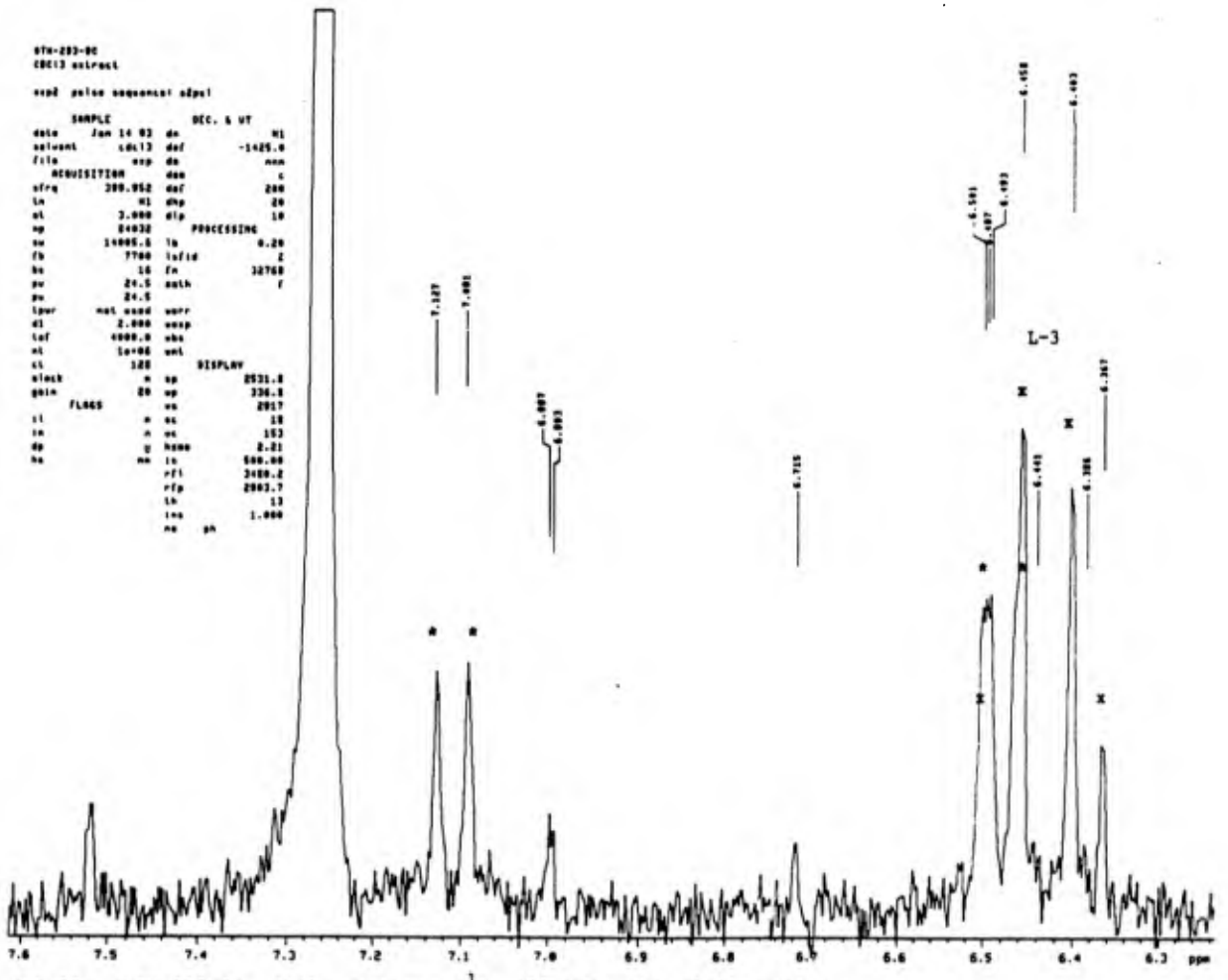
NMR-30. OTH-293-9a, $CDCl_3$ extract: 2-D COSY spectrum.



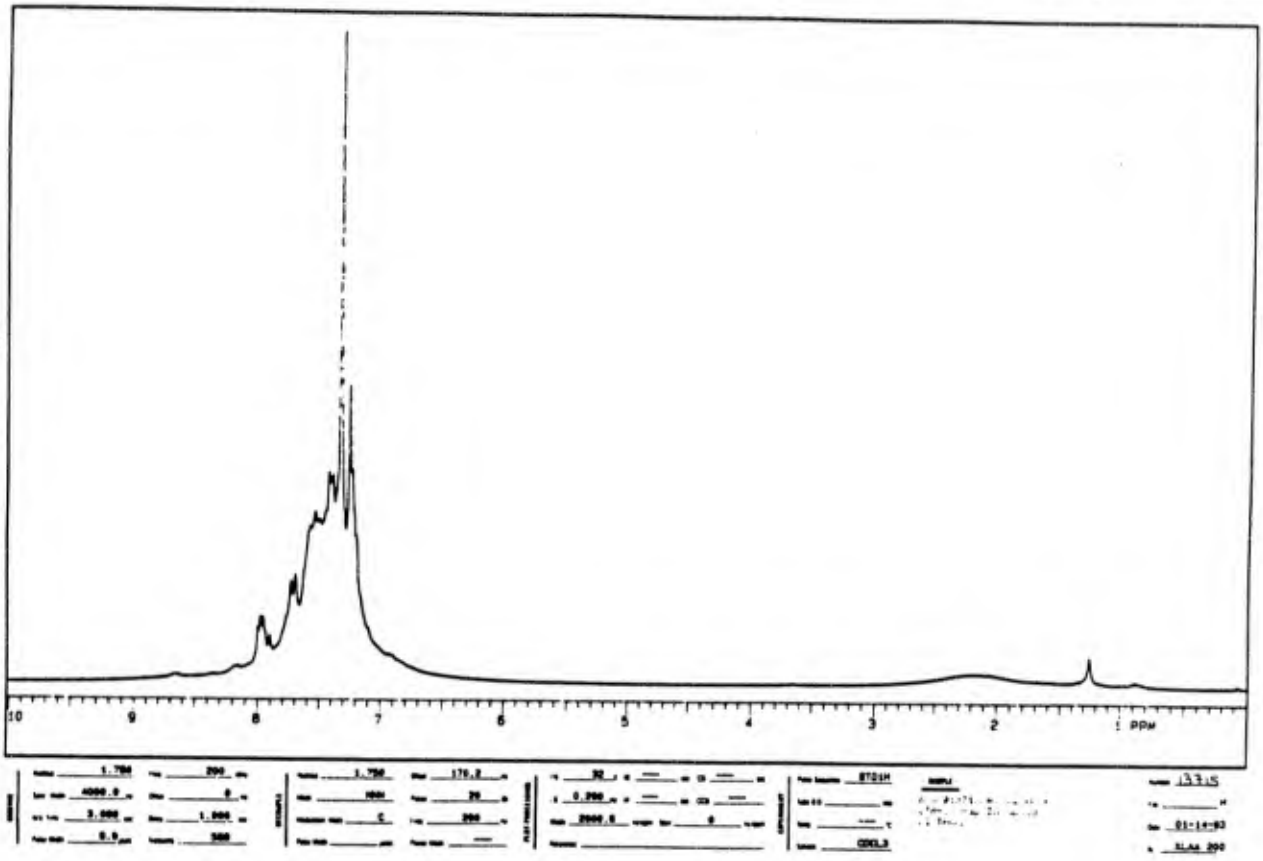
NMR-31. OTH-293-9b, $CDCl_3$ extract: ^1H, 200 MHz.



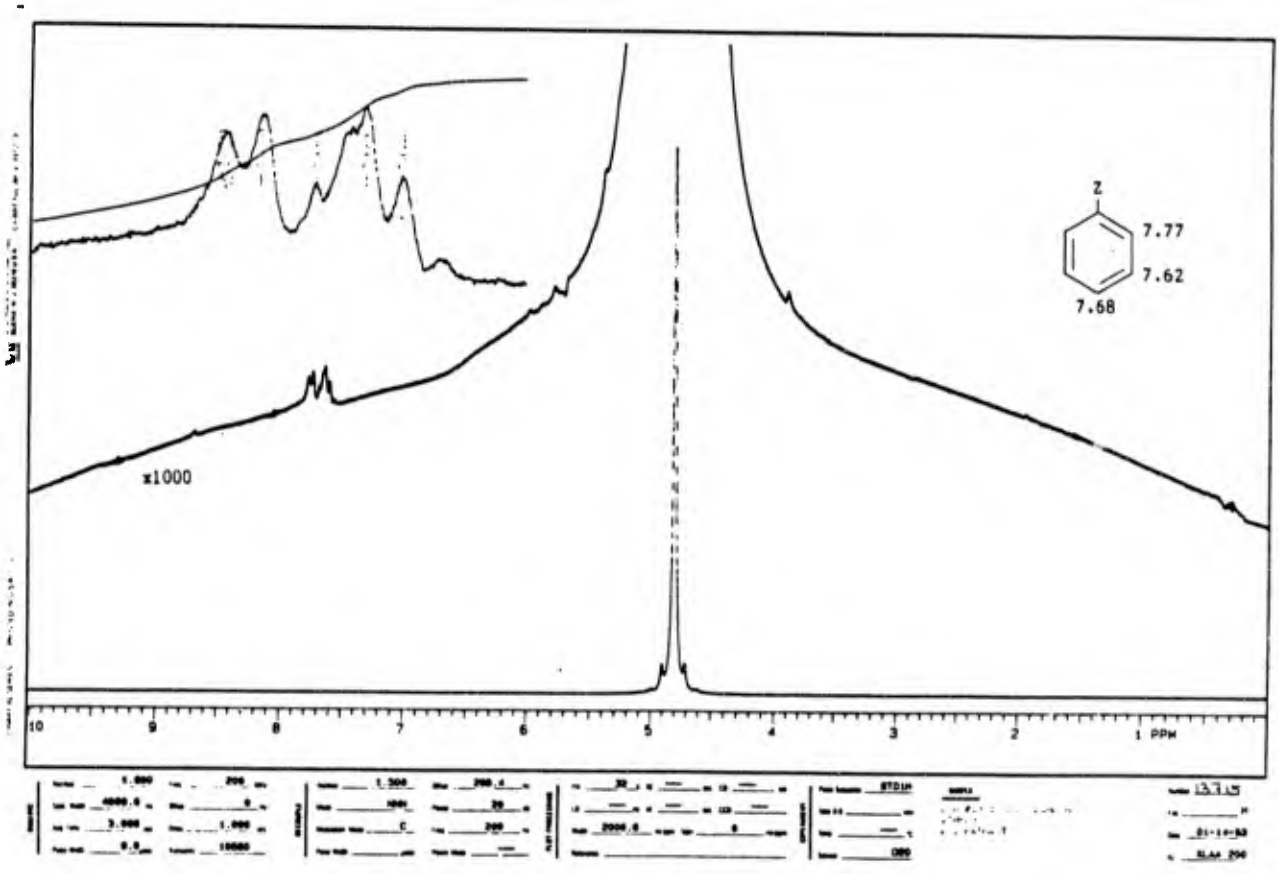
NMR-32. OTH-293-9c, $CDCl_3$ extract: ^1H, 200 MHz.



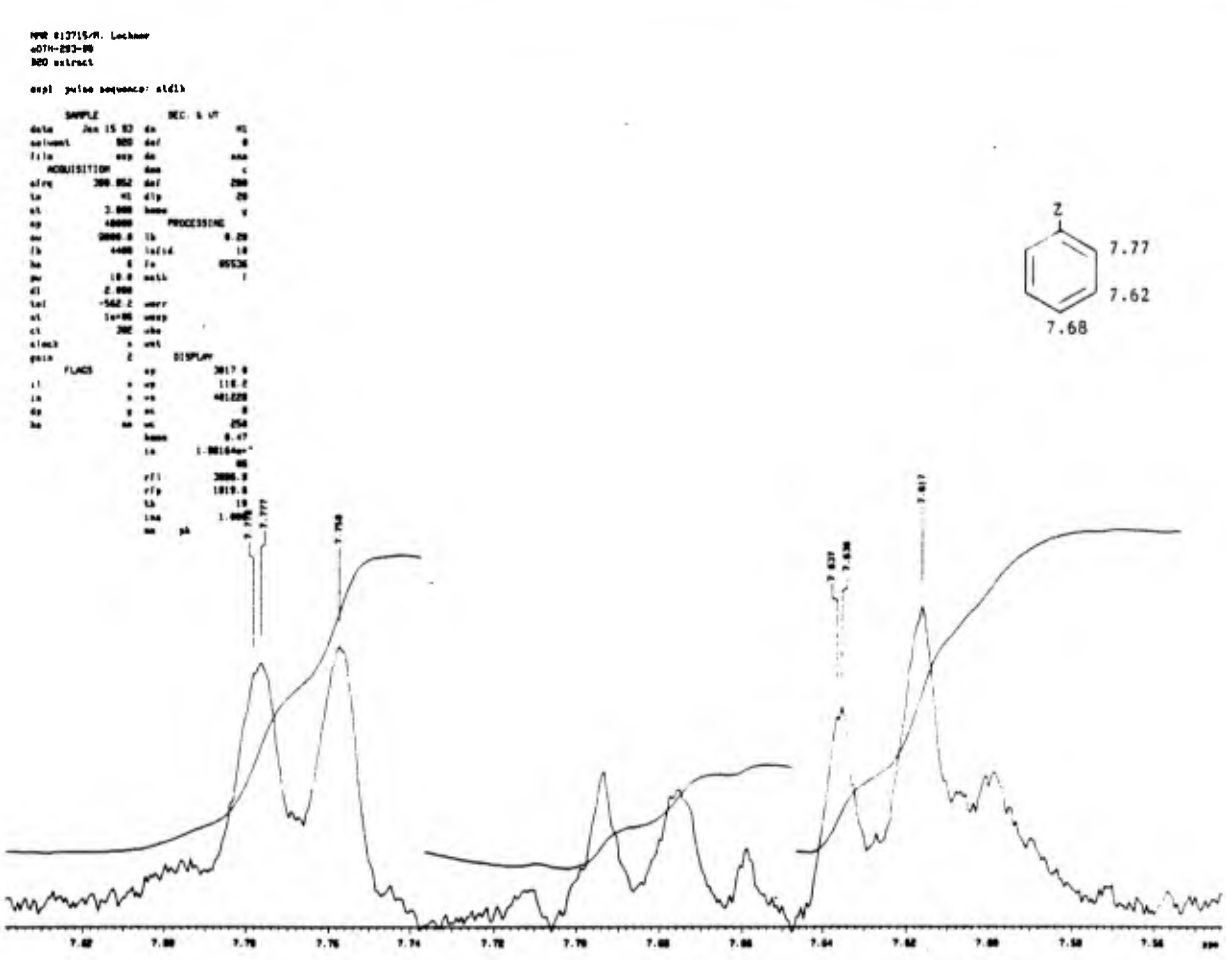
NMR-33. OTH-293-9c, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



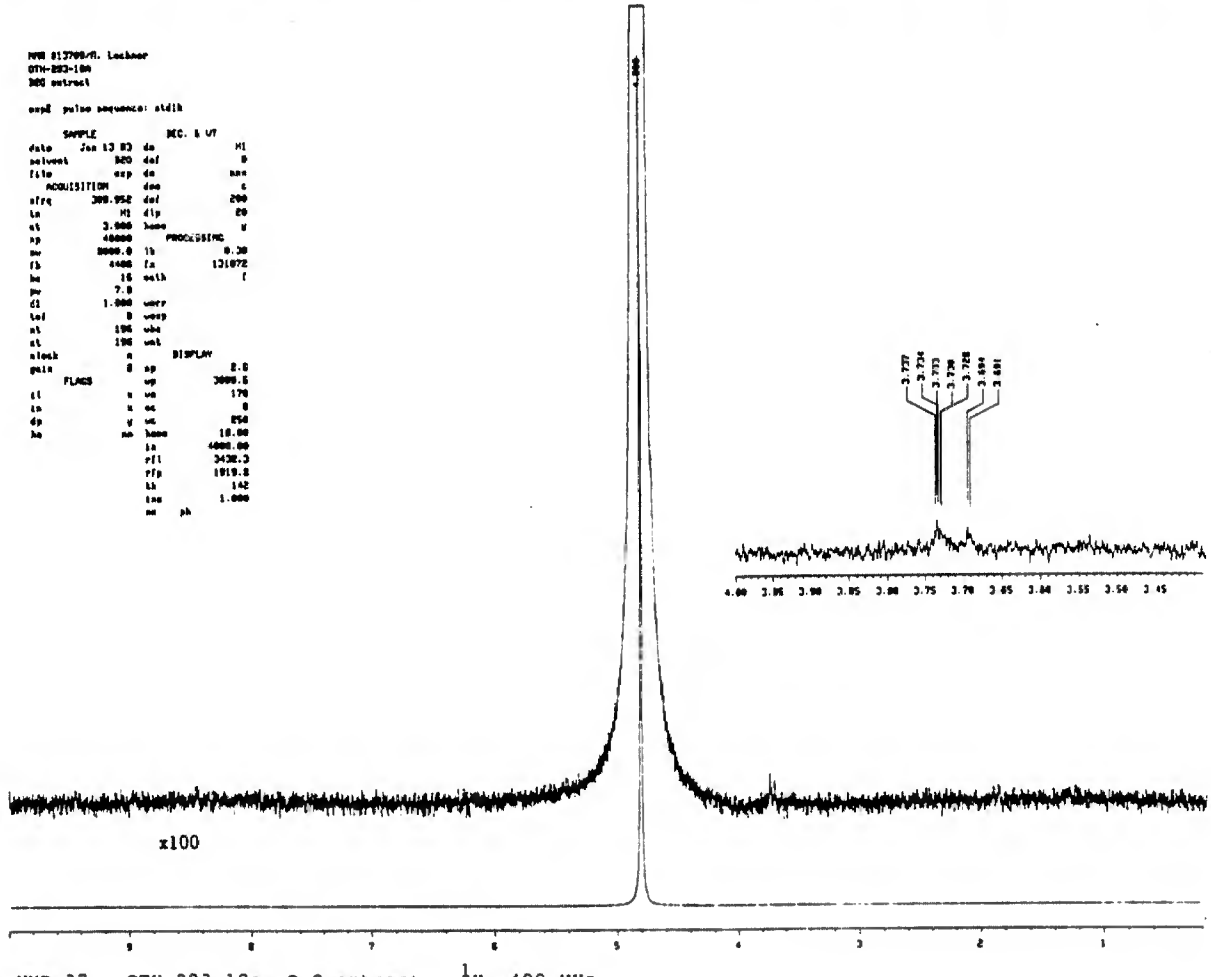
NMR-34. OTH-293-9d, $CDCl_3$ extract: ^1H, 200 MHz.



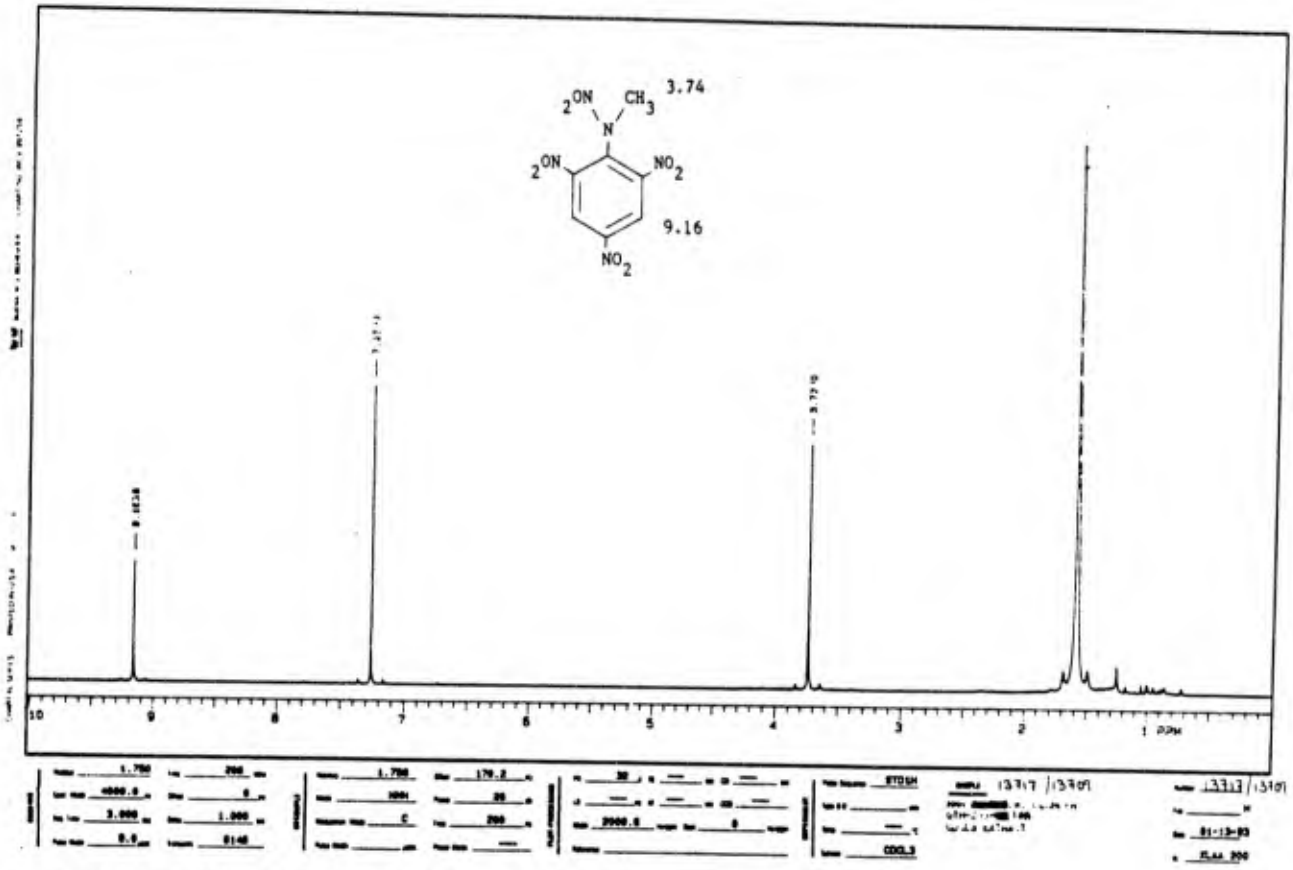
NMR-35. OTH-293-9d, D_2O extract: ^1H, 200 MHz.



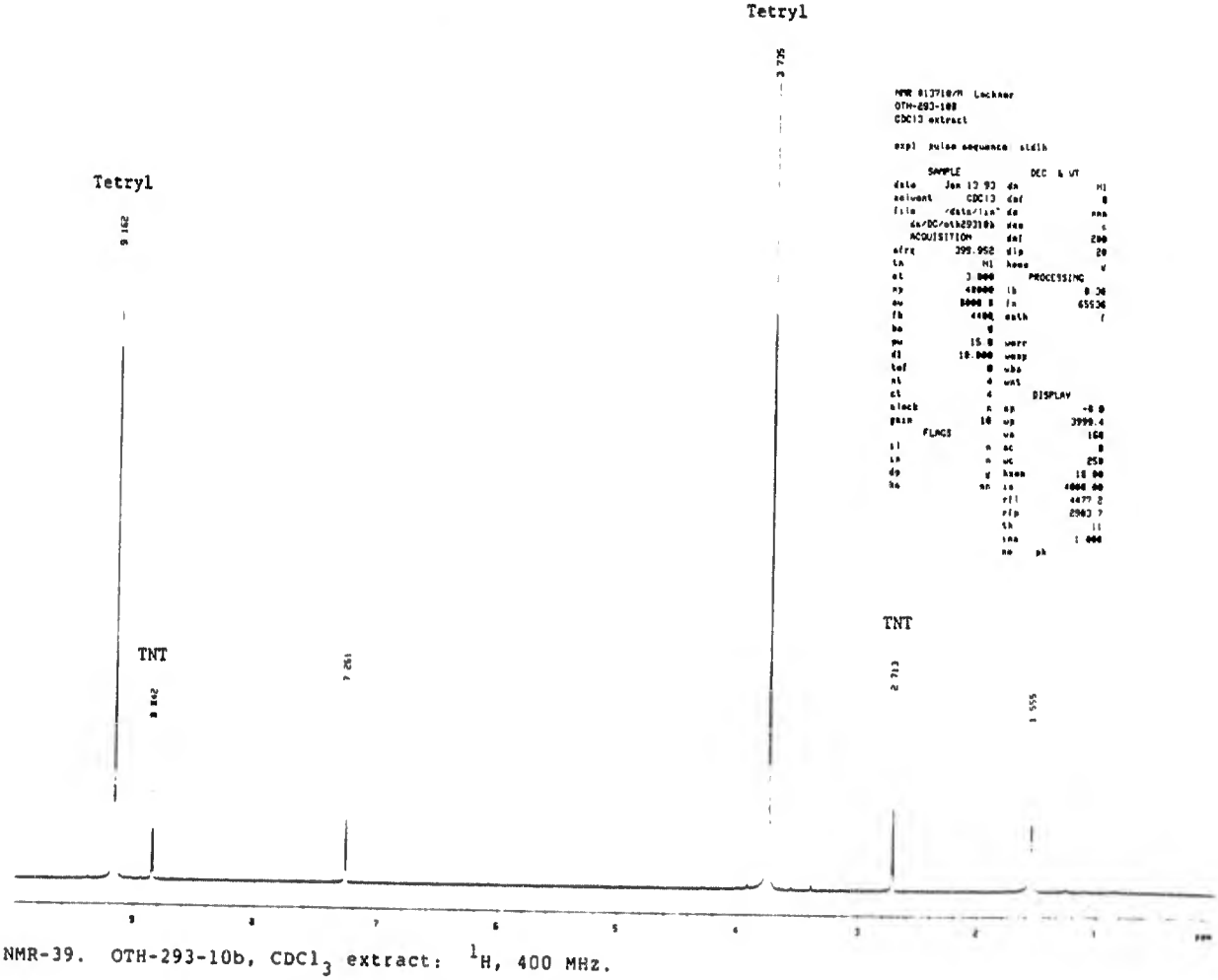
NMR-36. OTH-293-9d, D_2O extract: ^1H, 400 MHz, expanded spectrum.



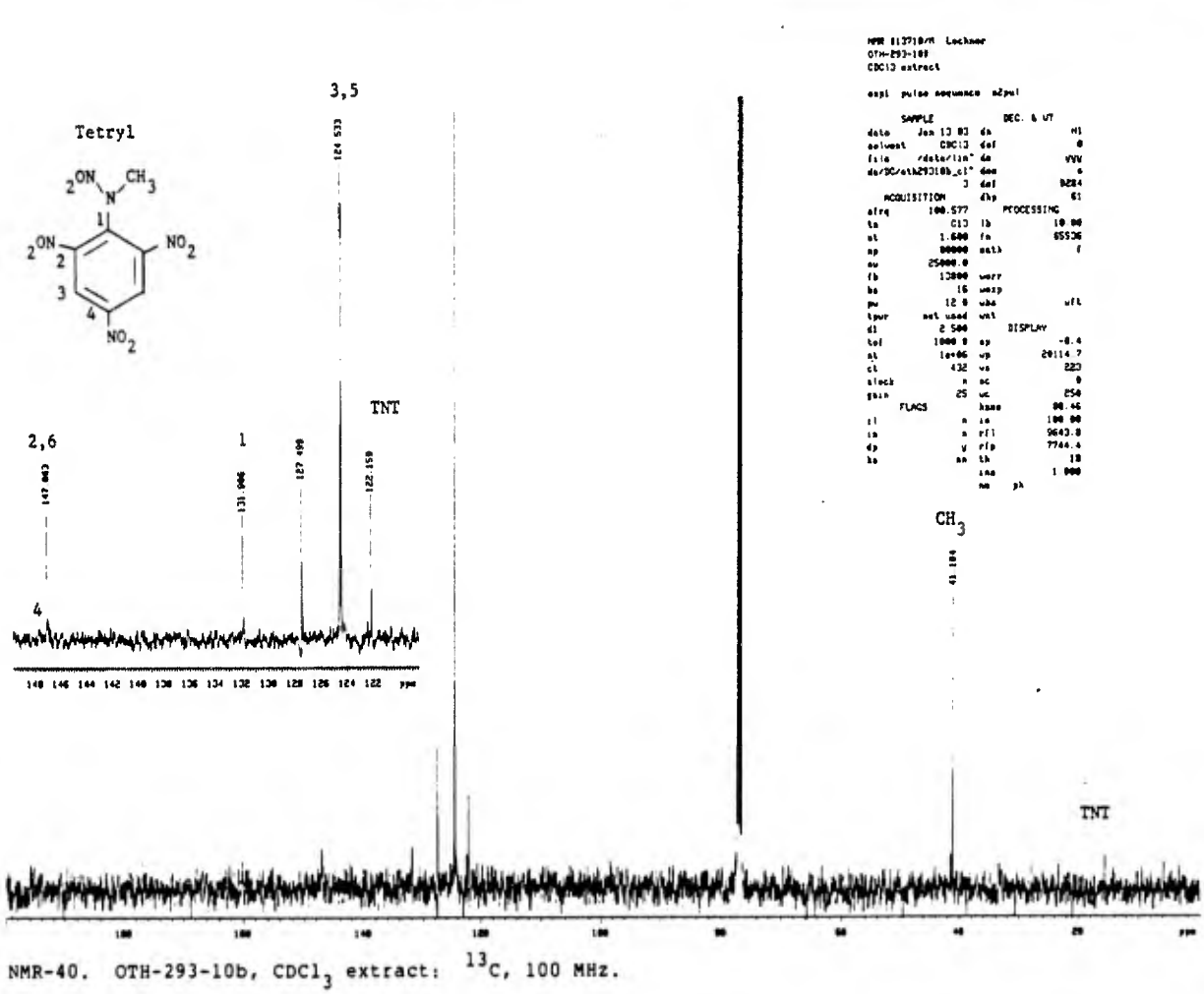
NMR-37. OTH-293-10a, D_2O extract: ^1H, 400 MHz.



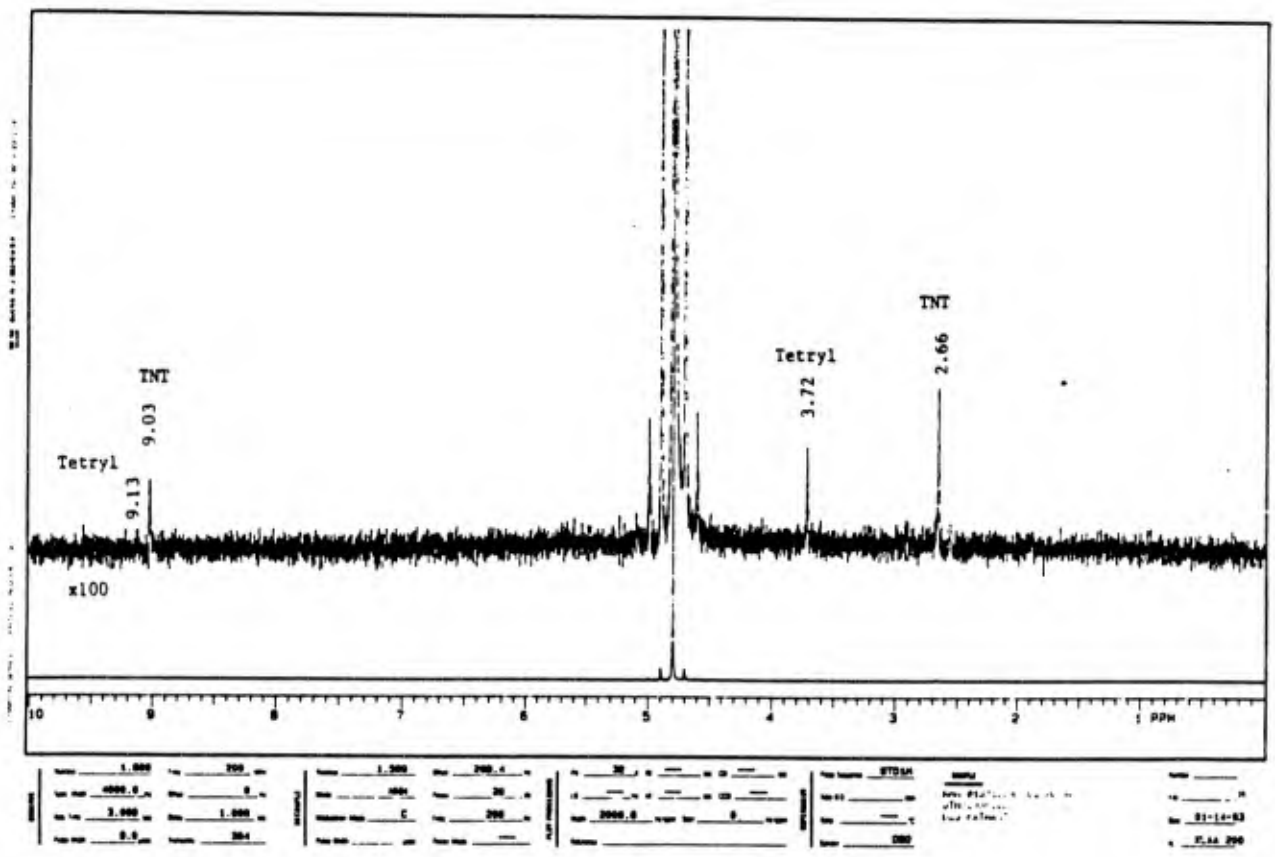
NMR-38. OTH-293-10a, $CDCl_3$ extract: ^1H, 200 MHz.



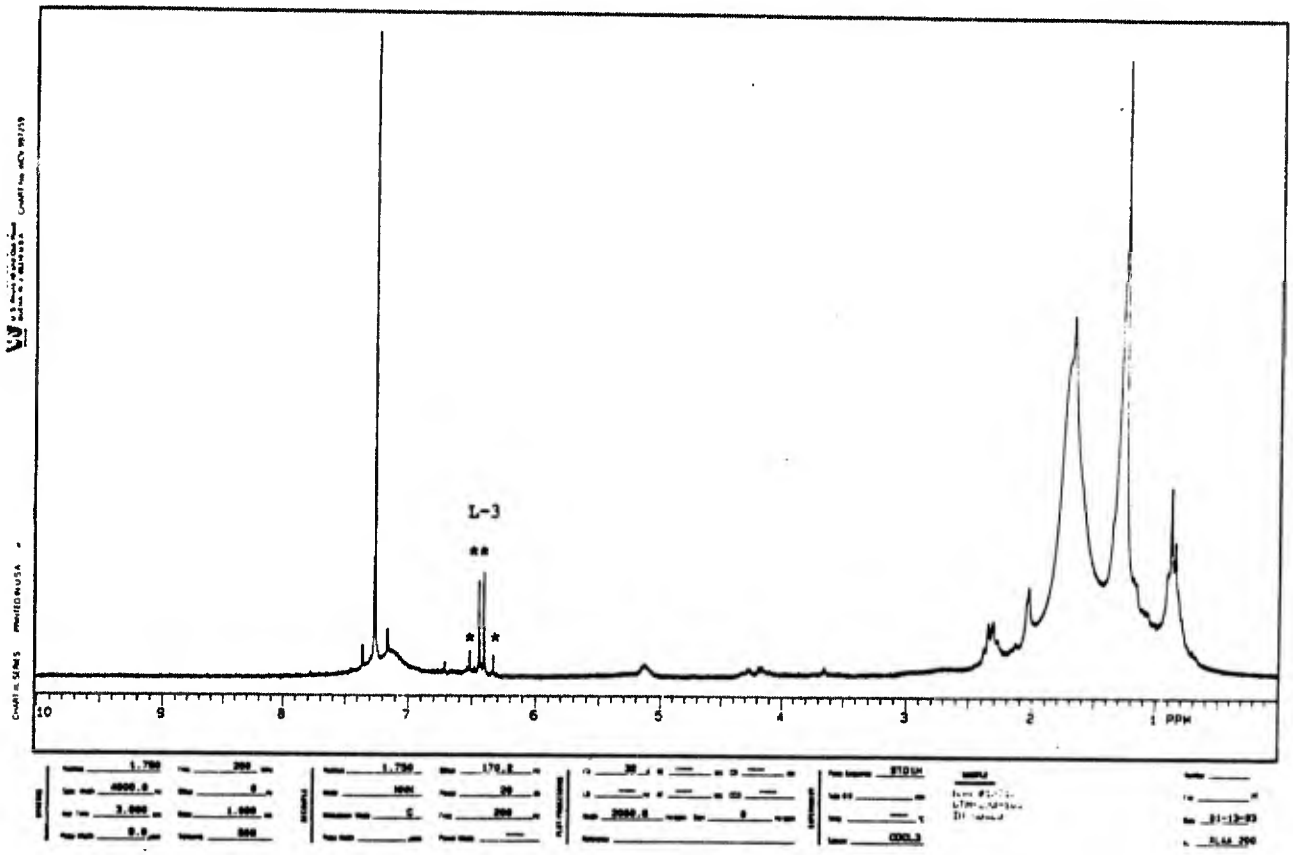
NMR-39. OTH-293-10b, $CDCl_3$ extract: ^1H, 400 MHz.



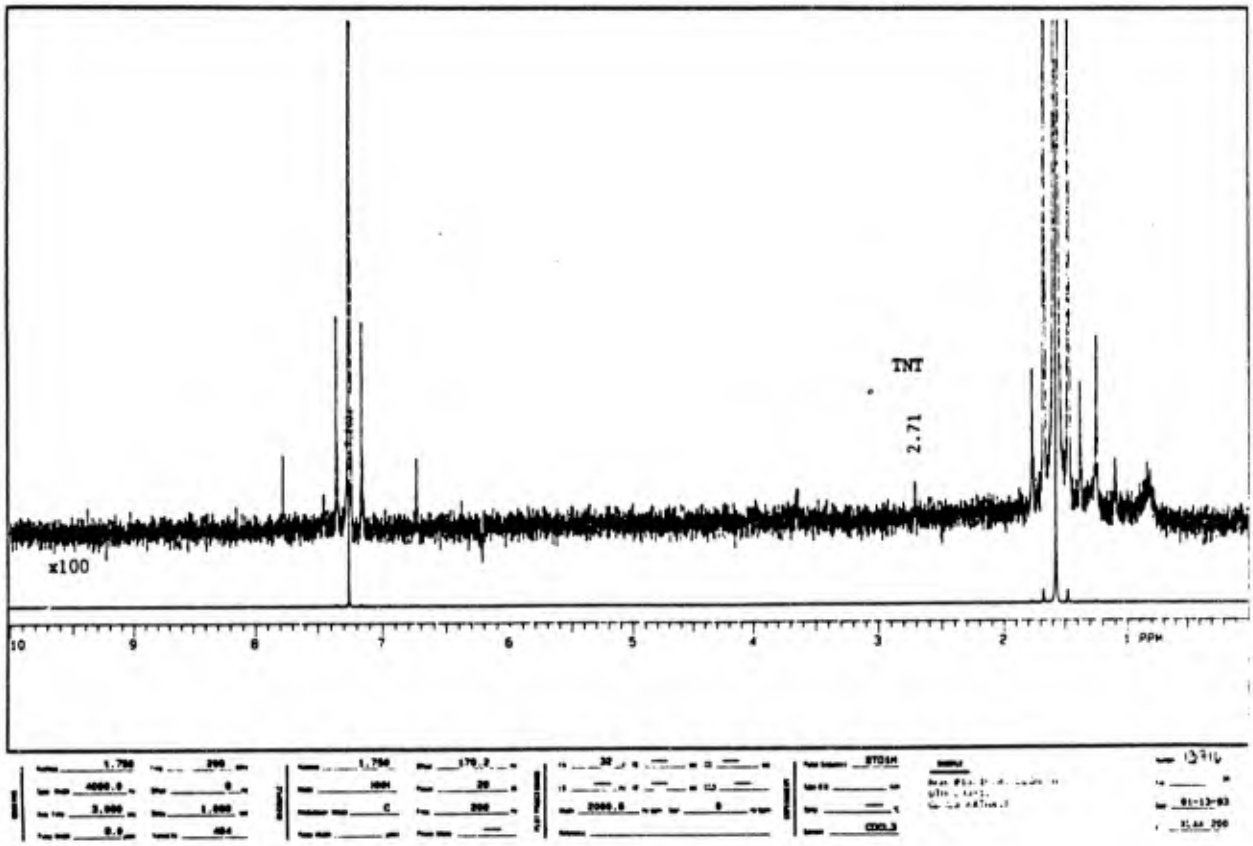
NMR-40. OTH-293-10b, $CDCl_3$ extract: ^{13}C, 100 MHz.



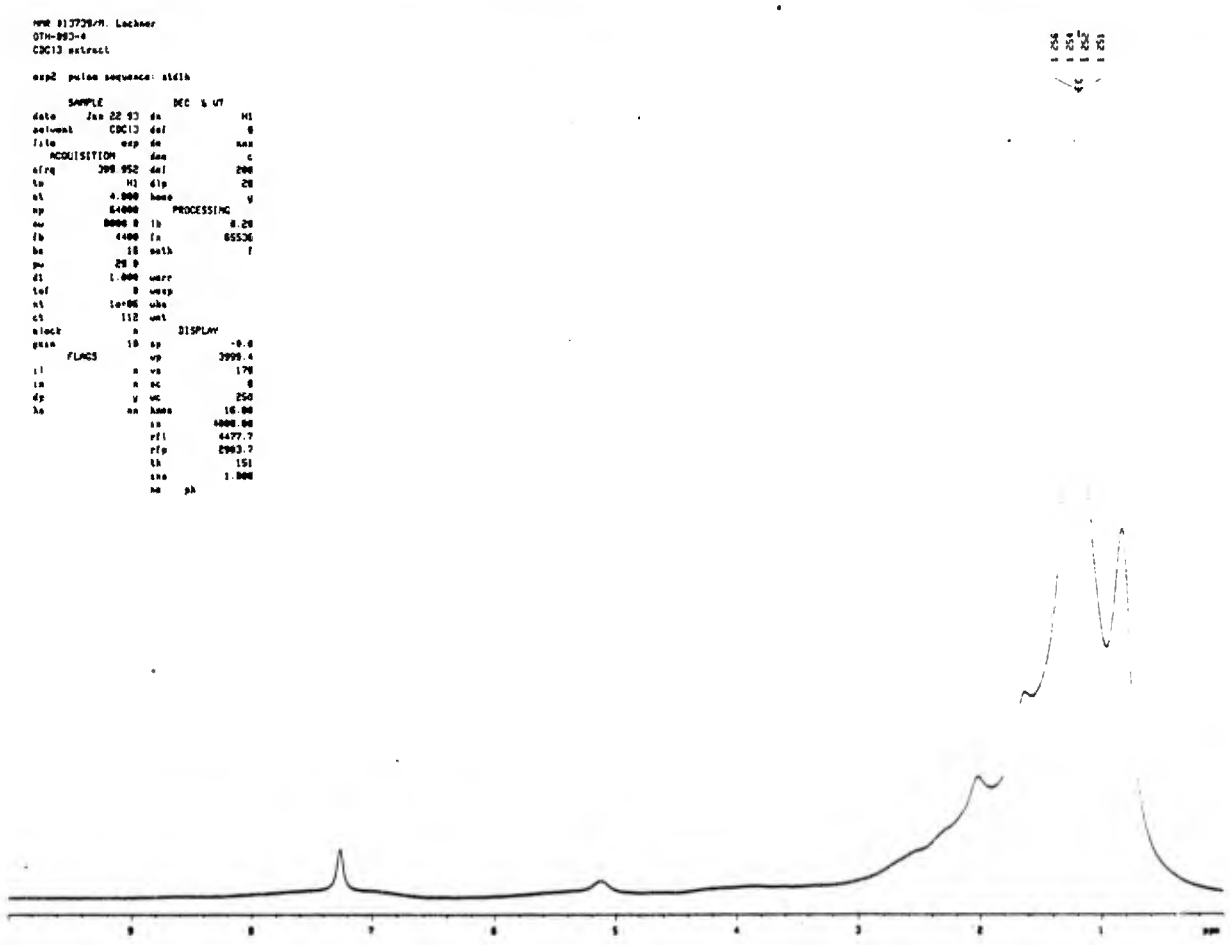
NMR-41. OTH-293-10b, D_2O extract: ^{1}H, 200 MHz.



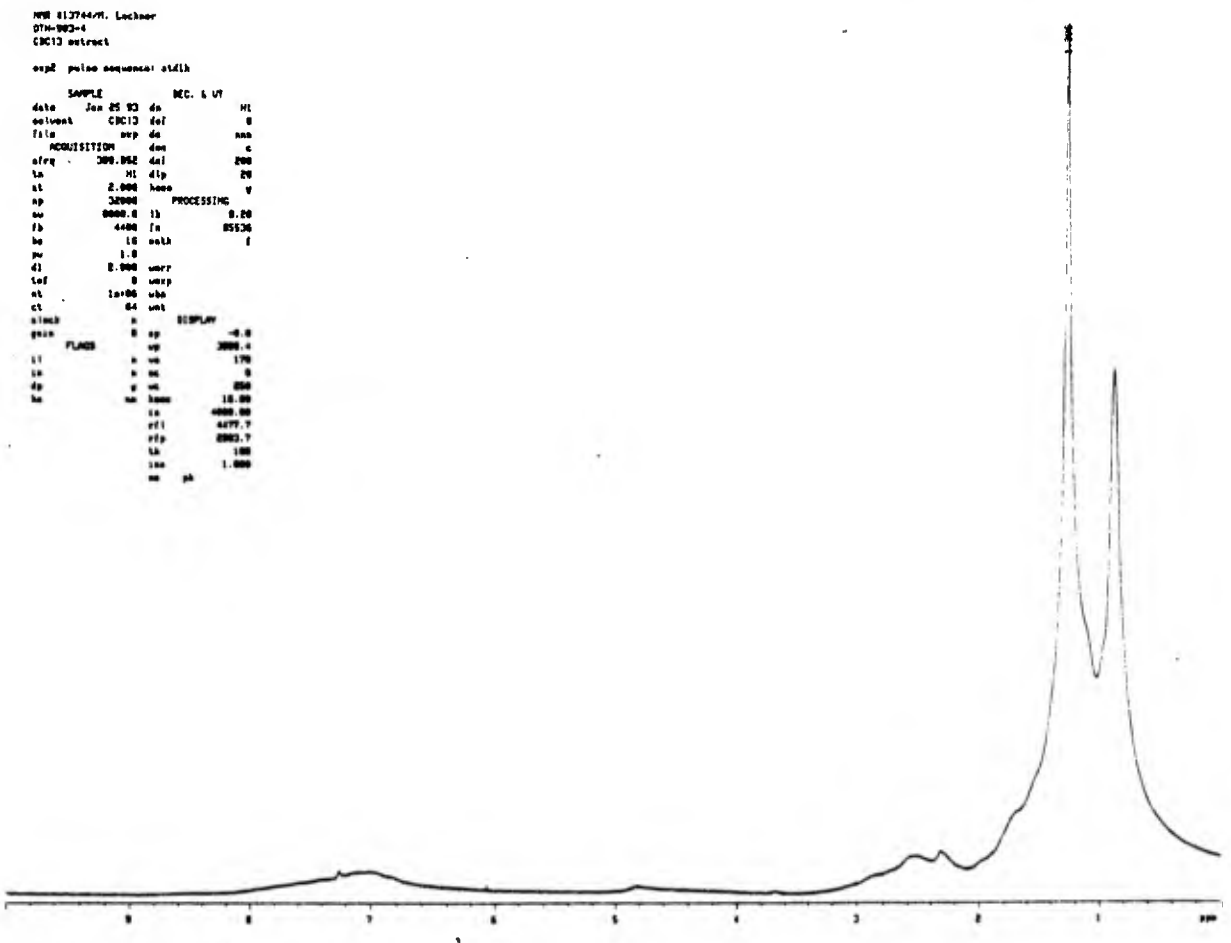
NMR-42. OTH-293-10c, $CDCl_3$ extract: ^1H, 200 MHz.



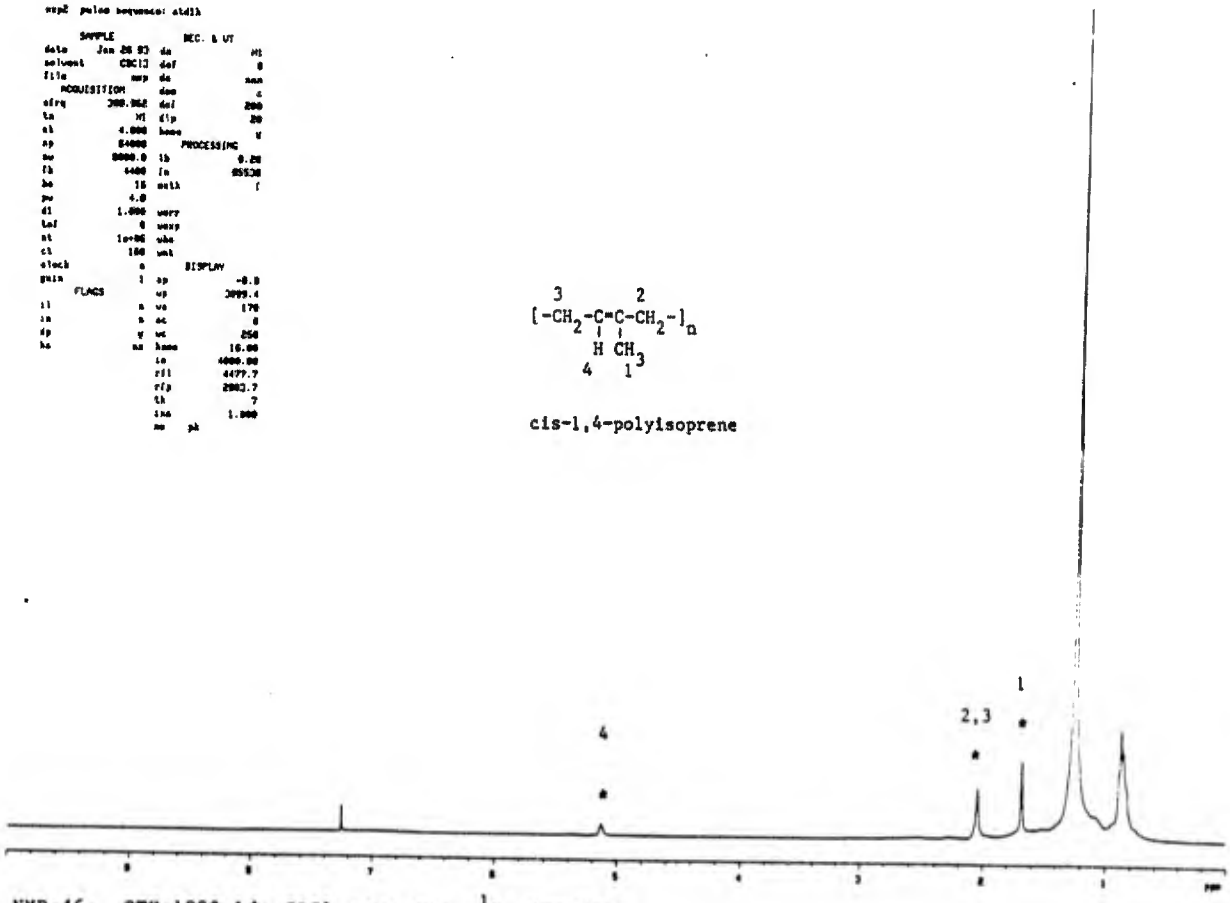
NMR-43. OTH-293-10g, $CDCl_3$ extract: ^1H, 200 MHz.



NMR-44. OTH-893-4, $CDCl_3$ extract: ^1H, 400 MHz.



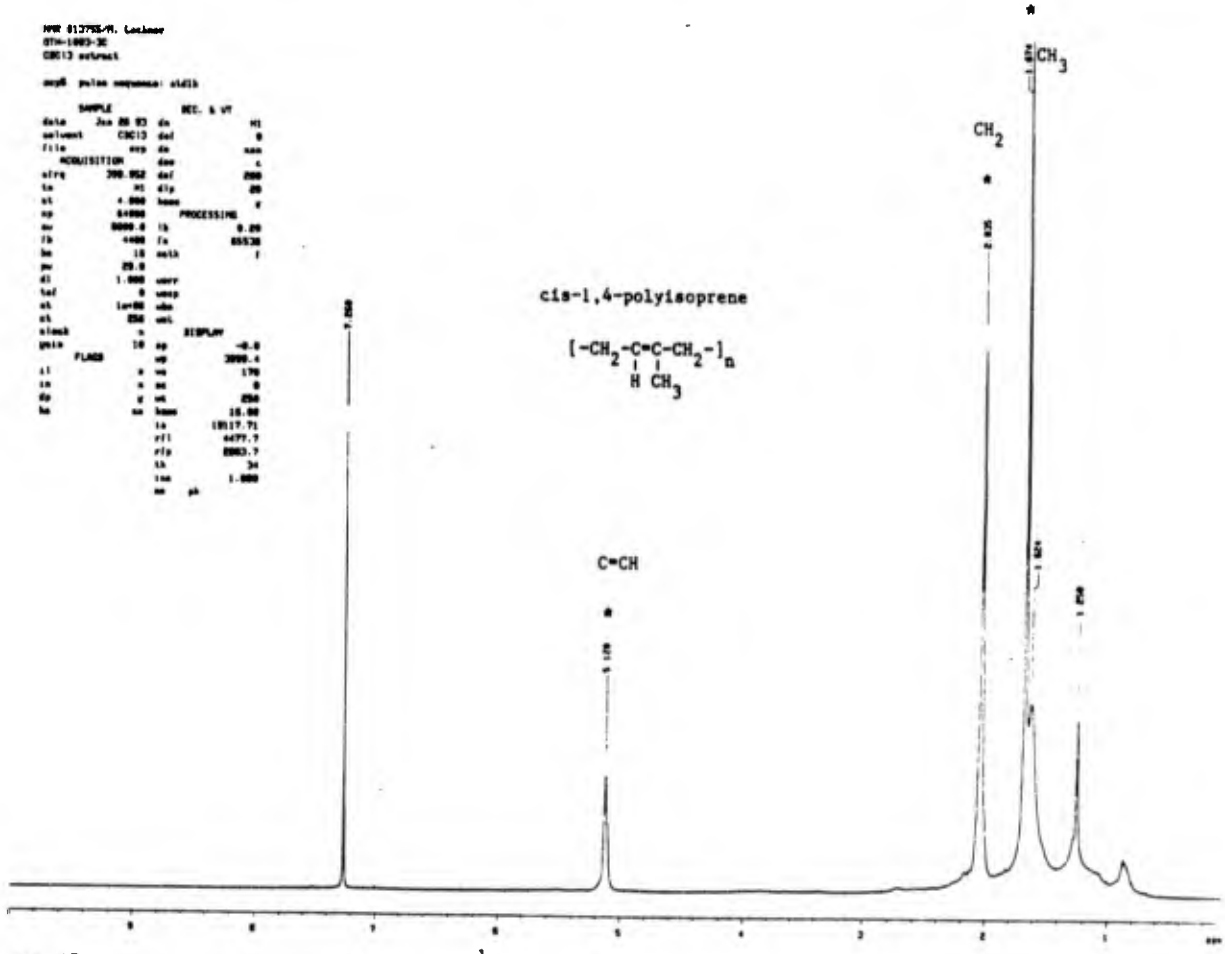
NMR-45. OTH-993-4, $CDCl_3$ extract: ^1H, 400 MHz.



cis-1,4-polyisoprene

$$[-CH_2-\underset{H}{\overset{3}{C}}=\underset{CH_3}{\overset{2}{C}}-CH_2-]_n \quad (4\ 1)$$

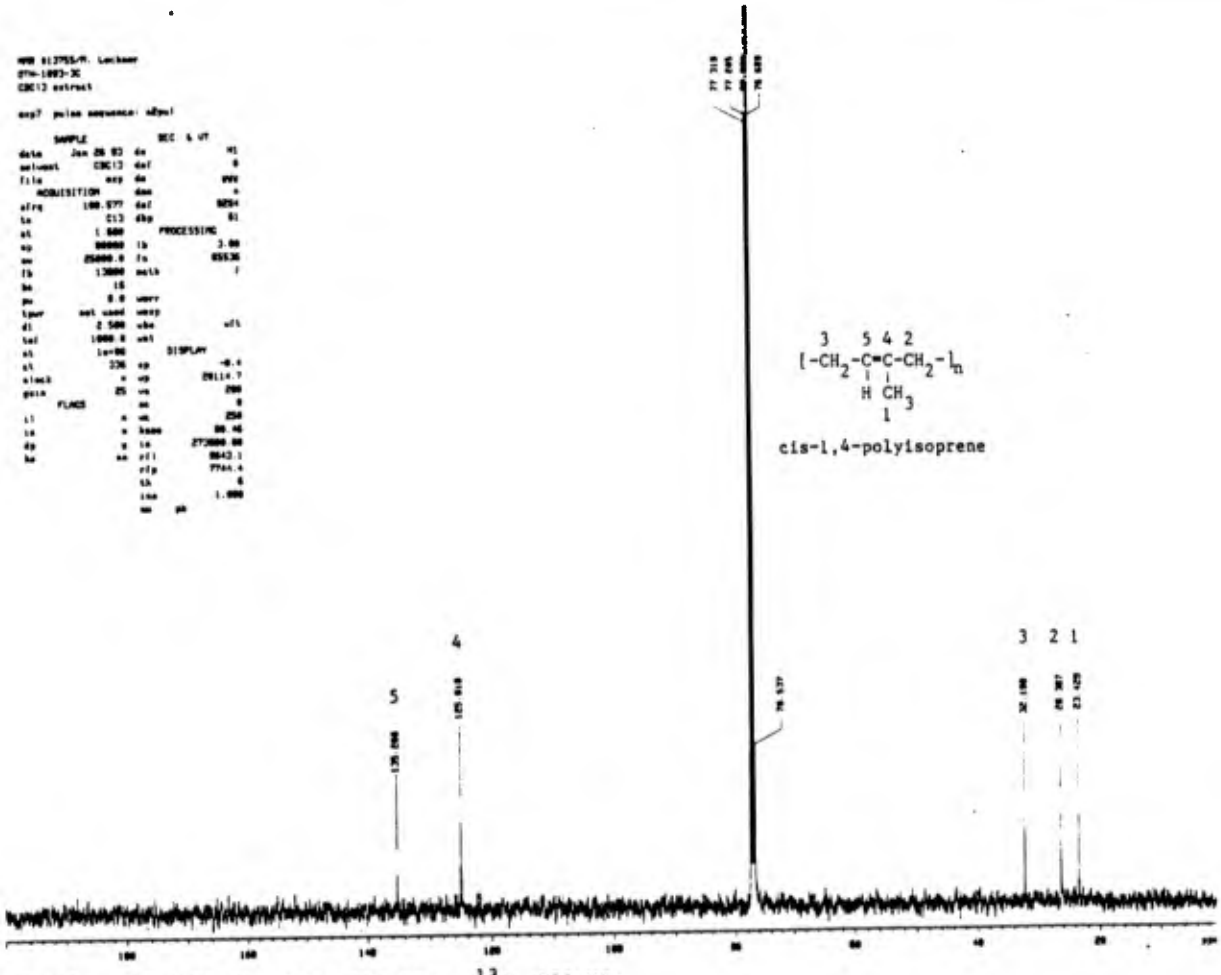
NMR-46. OTH-1093-1d, $CDCl_3$ extract: ^1H, 400 MHz.



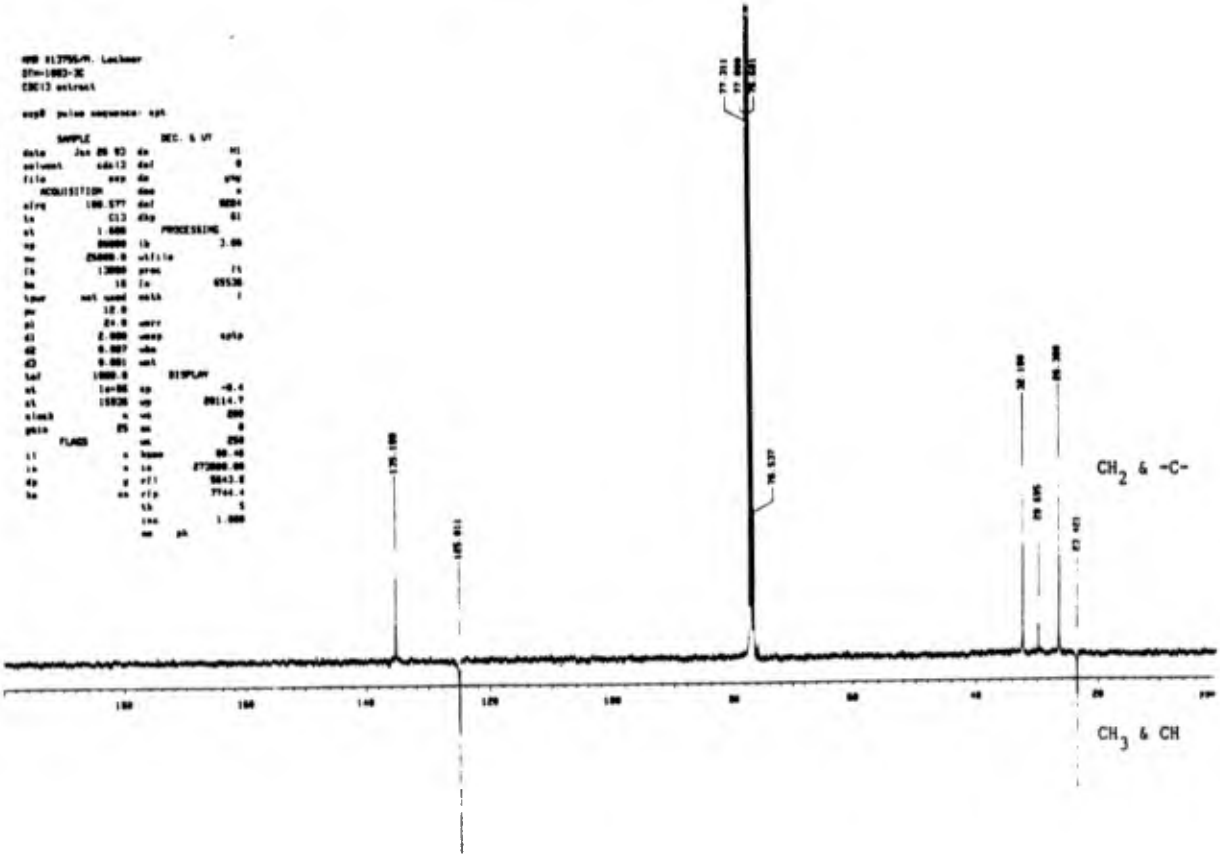
cis-1,4-polyisoprene

$$[-CH_2-\underset{H}{\overset{}{C}}=\underset{CH_3}{\overset{}{C}}-CH_2-]_n$$

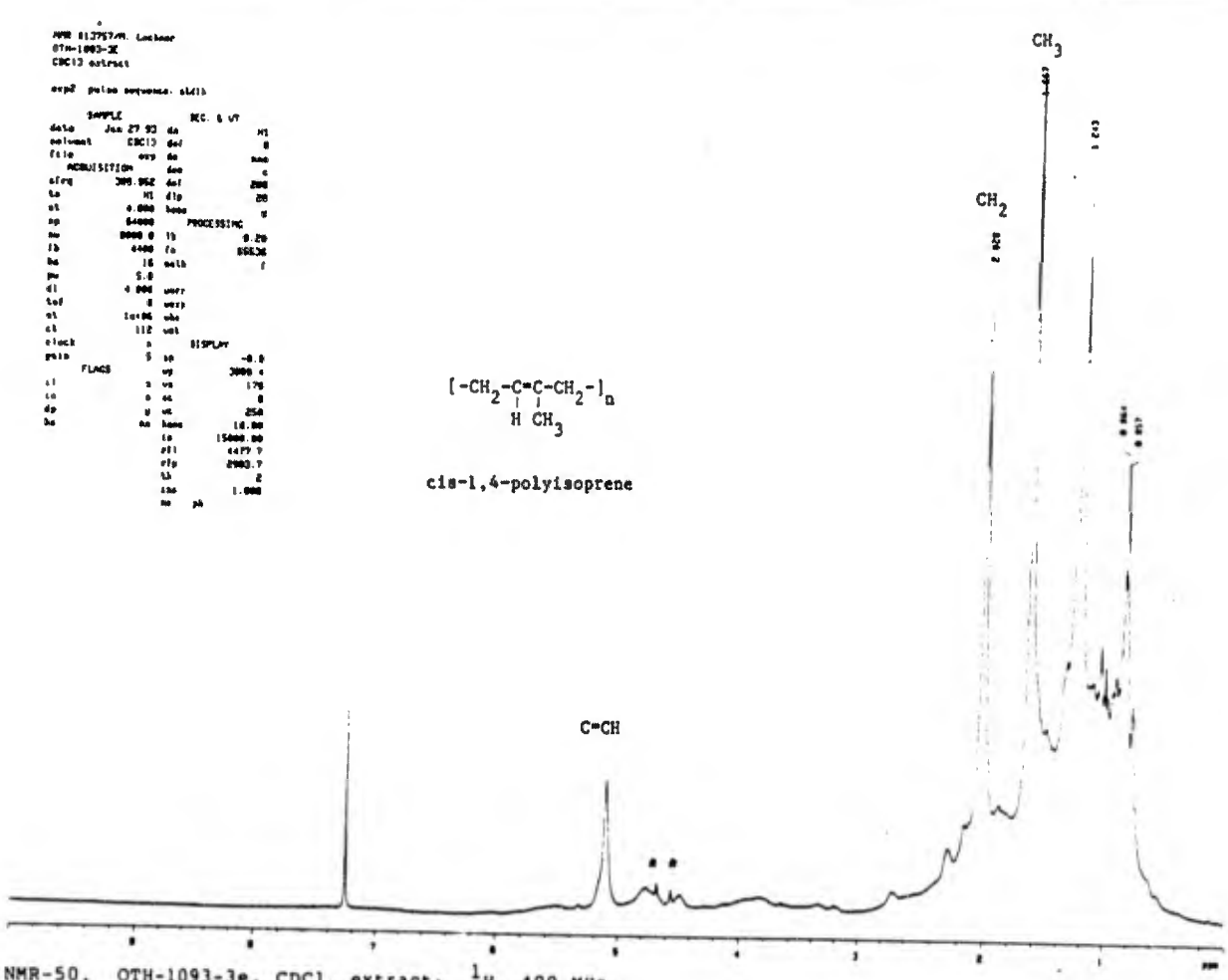
NMR-47. OTH-1093-3c, $CDCl_3$ extract: ^1H, 400 MHz.



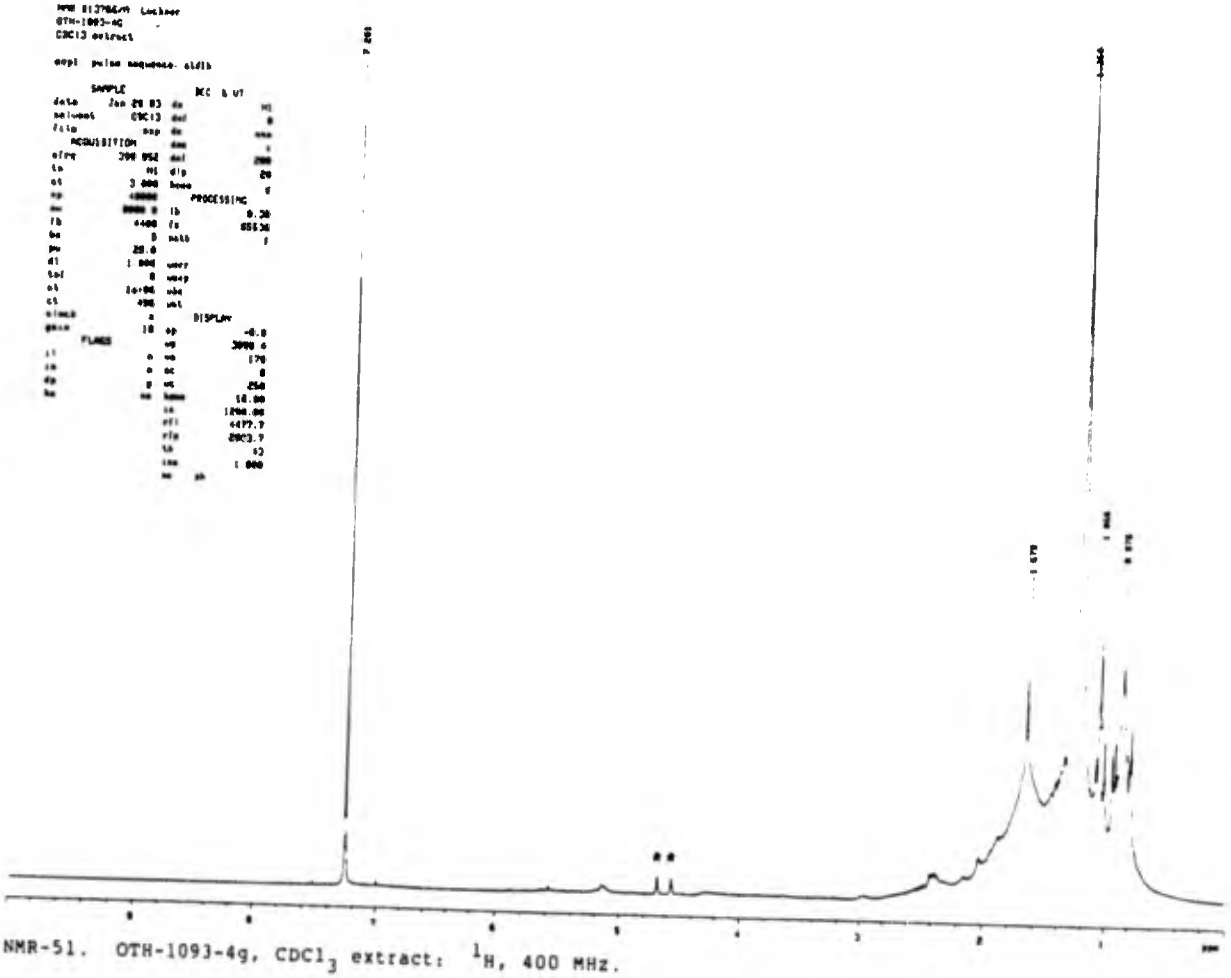
NMR-48. OTH-1093-3c, $CDCl_3$ extract: ^{13}C, 100 MHz.



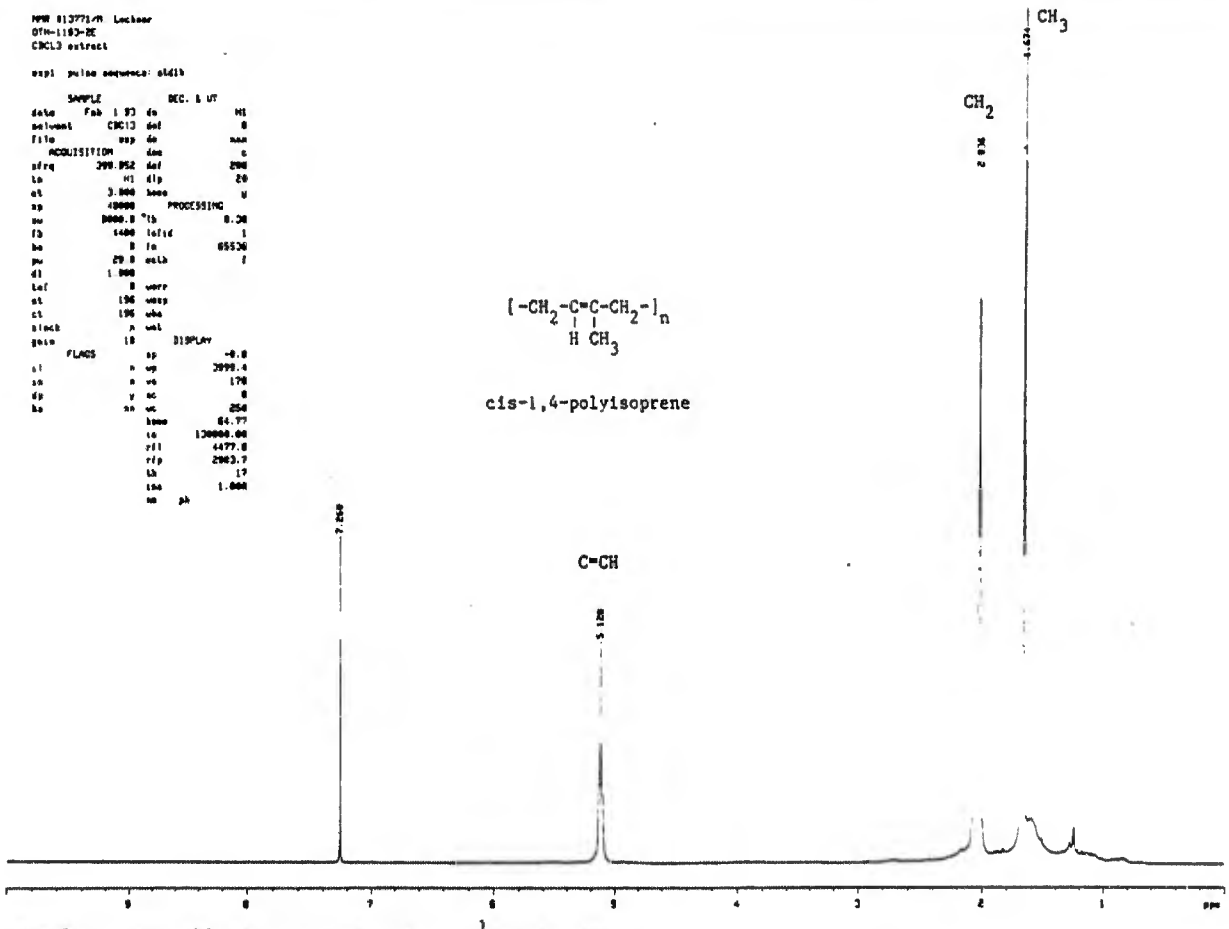
NMR-49. OTH-1093-3c, $CDCl_3$ extract: Attached Proton Test (APT) spectrum.



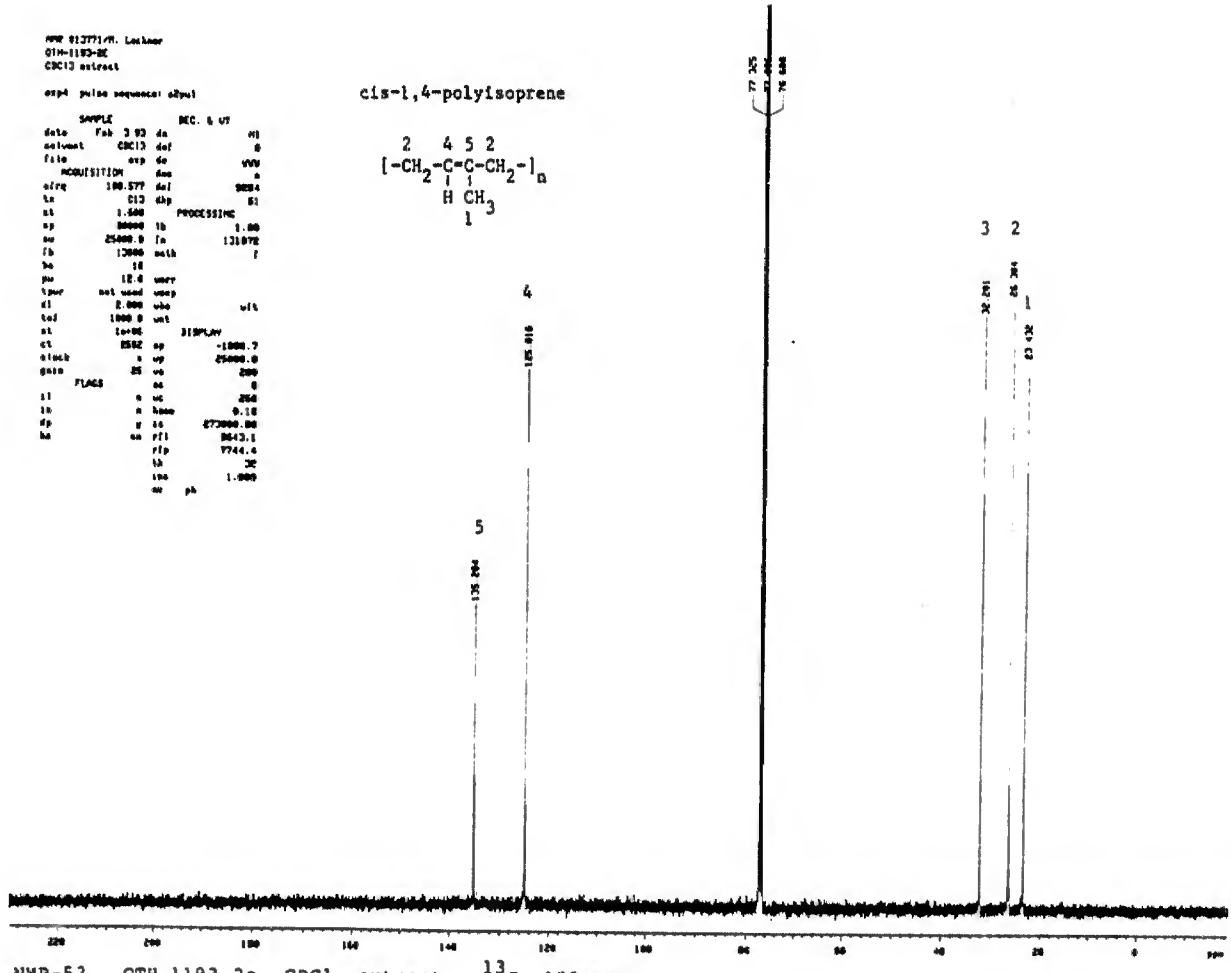
NMR-50. OTH-1093-3e, $CDCl_3$ extract: ^1H, 400 MHz.



NMR-51. OTH-1093-4g, $CDCl_3$ extract: ^1H, 400 MHz.



NMR-52. OTH-1193-2e, $CDCl_3$ extract: ^1H, 400 MHz.

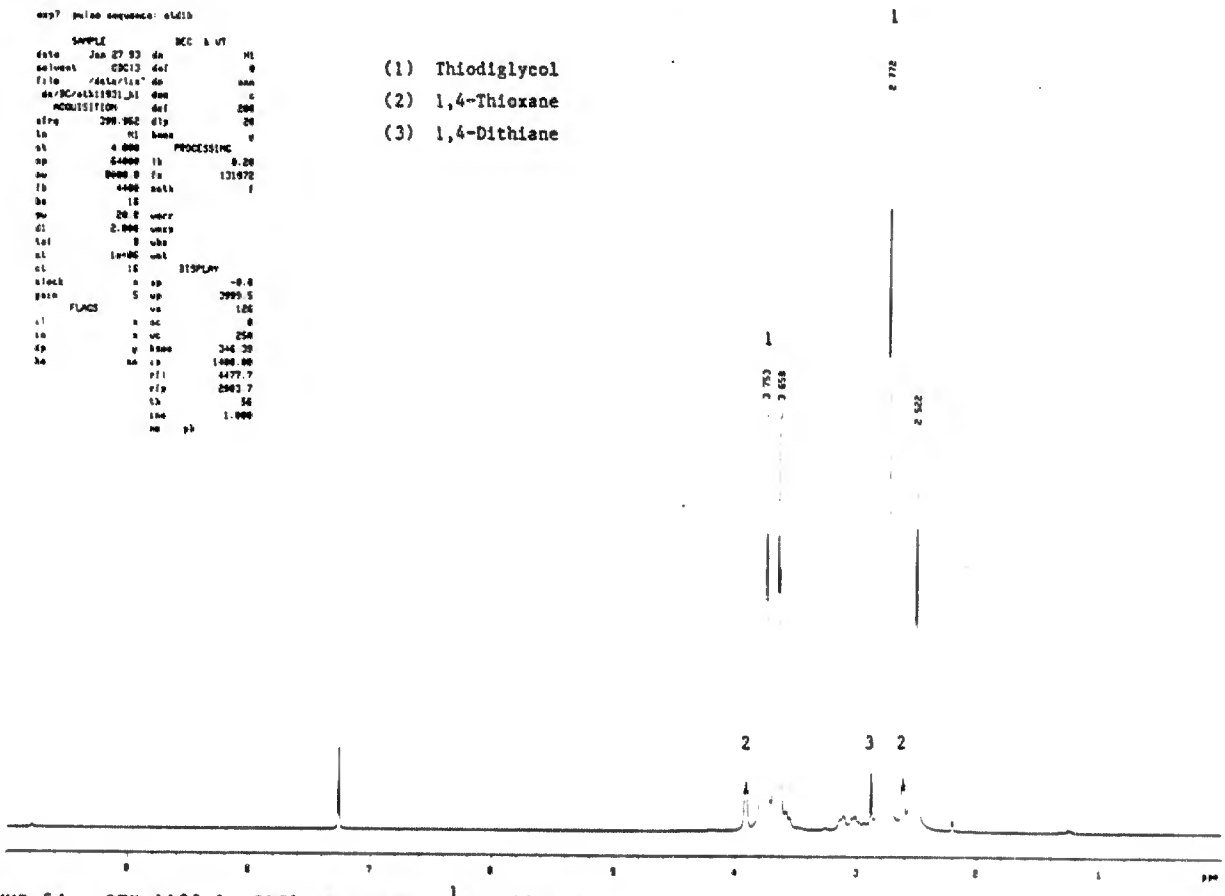


NMR-53. OTH-1193-2e, $CDCl_3$ extract: ^{13}C, 100 MHz.

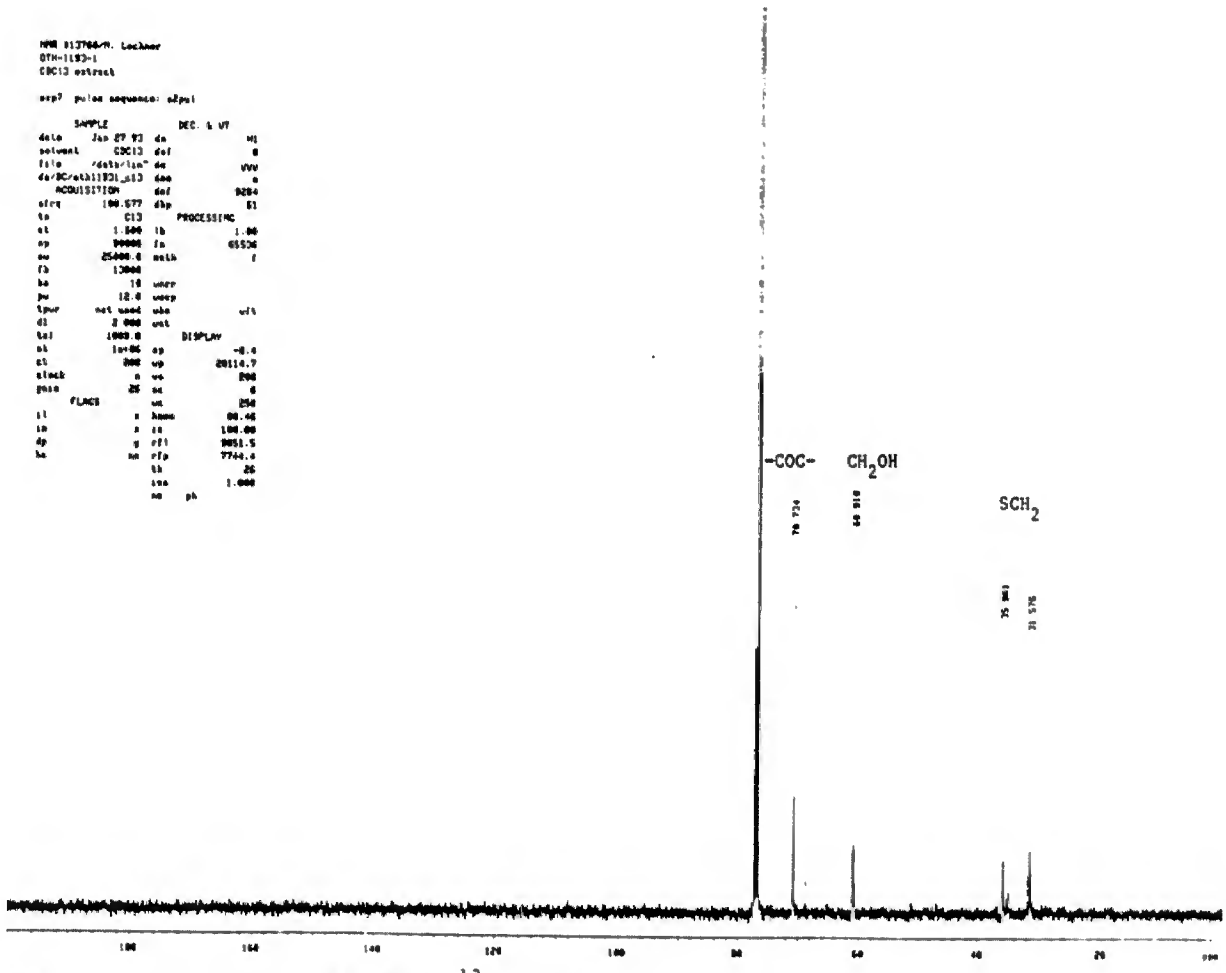
(1) Thiodiglycol

(2) 1,4-Thioxane

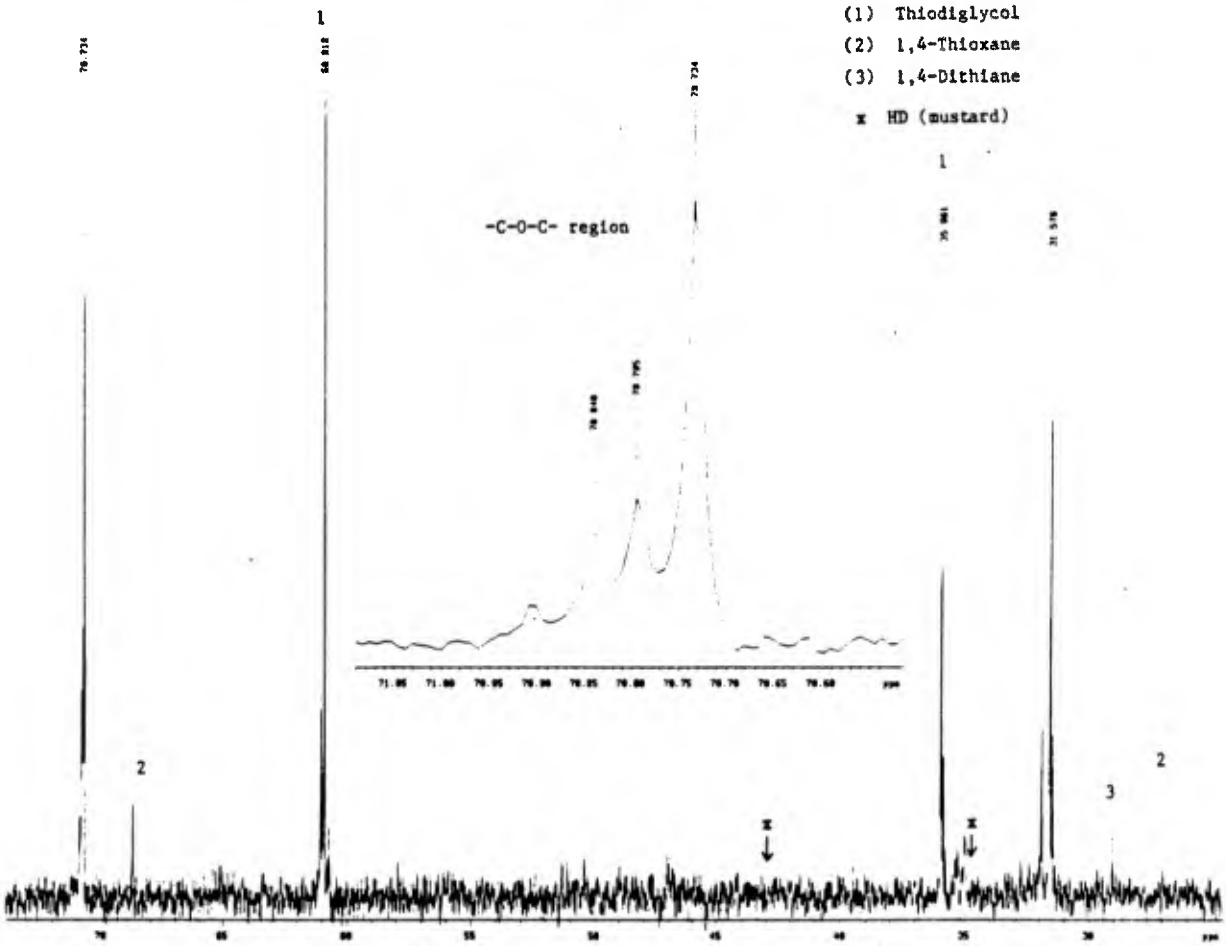
(3) 1,4-Dithiane



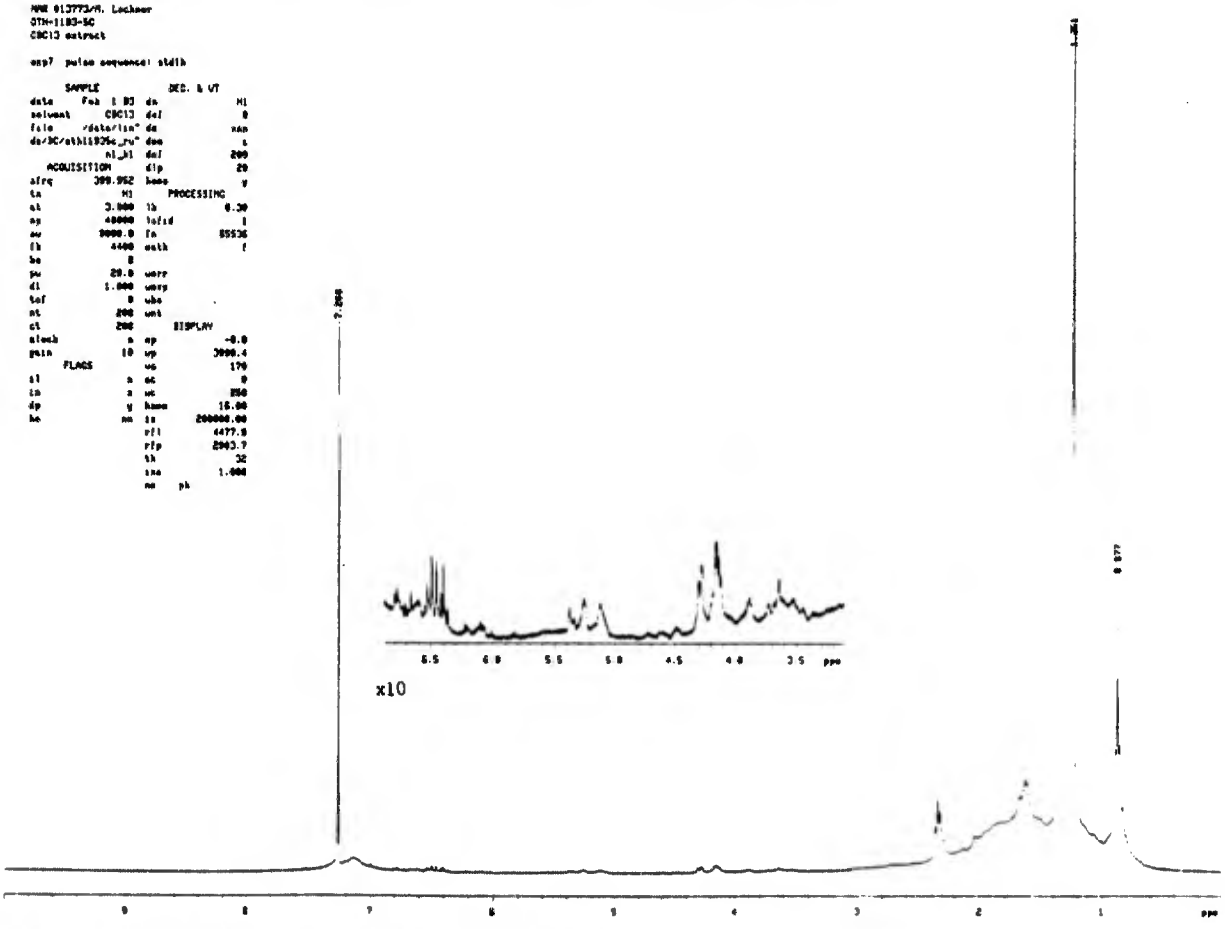
NMR-54. OTH-1193-1, $CDCl_3$ extract: ^1H, 400 MHz.



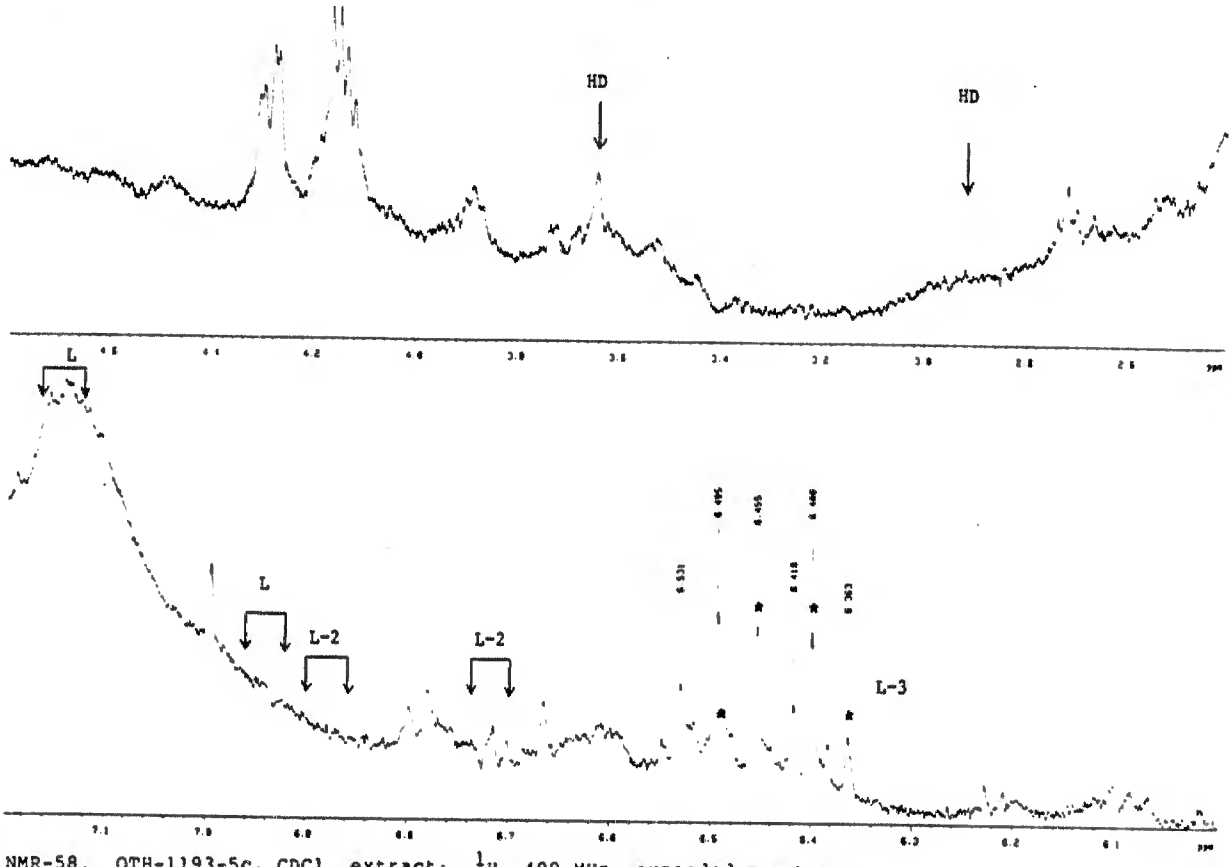
NMR-55. OTH-1193-1, $CDCl_3$ extract: ^{13}C, 100 MHz.



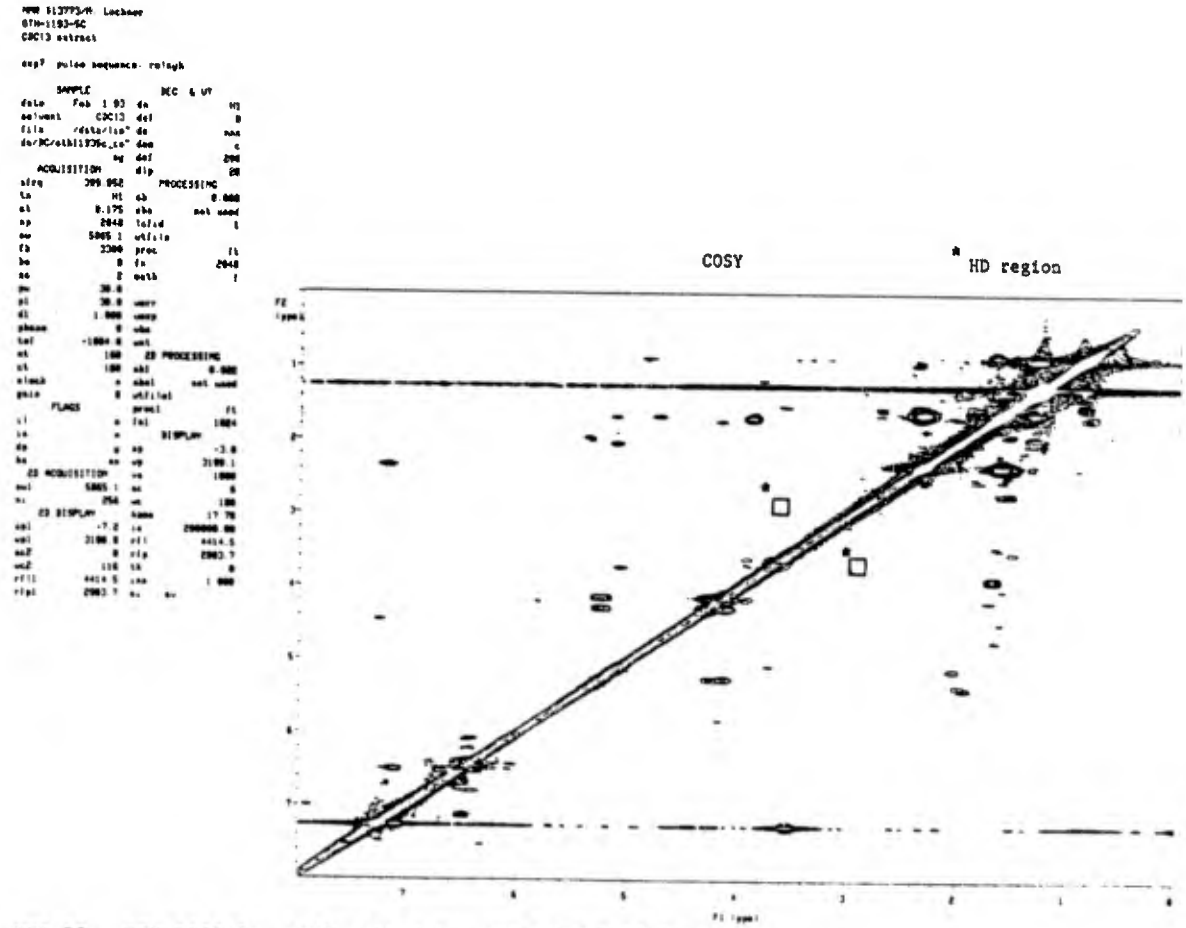
NMR-56. OTH-1193-1, $CDCl_3$ extract: ^{13}C, 100 MHz, expanded spectrum.



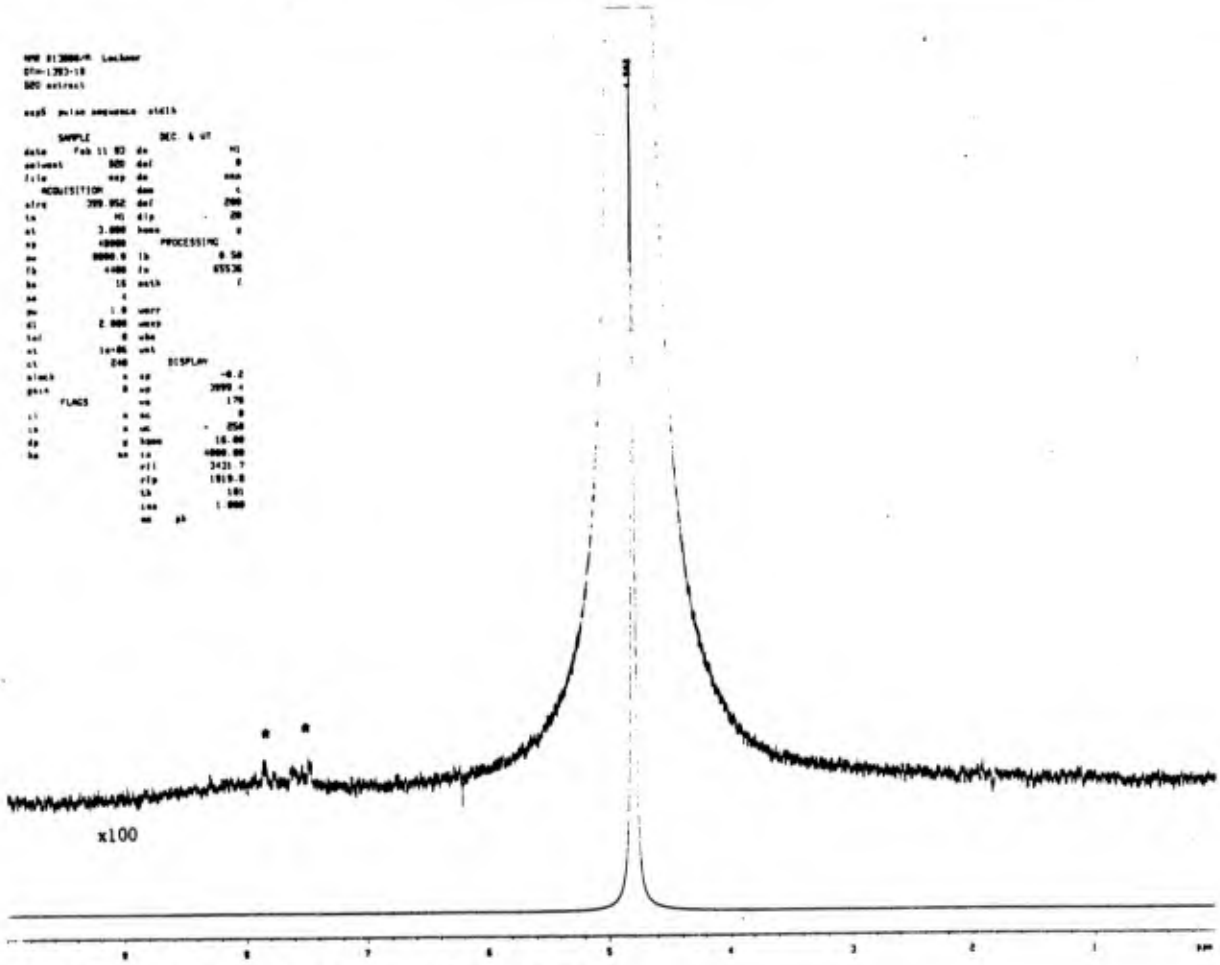
NMR-57. OTH-1193-5c, $CDCl_3$ extract: ^{1}H, 400 MHz.



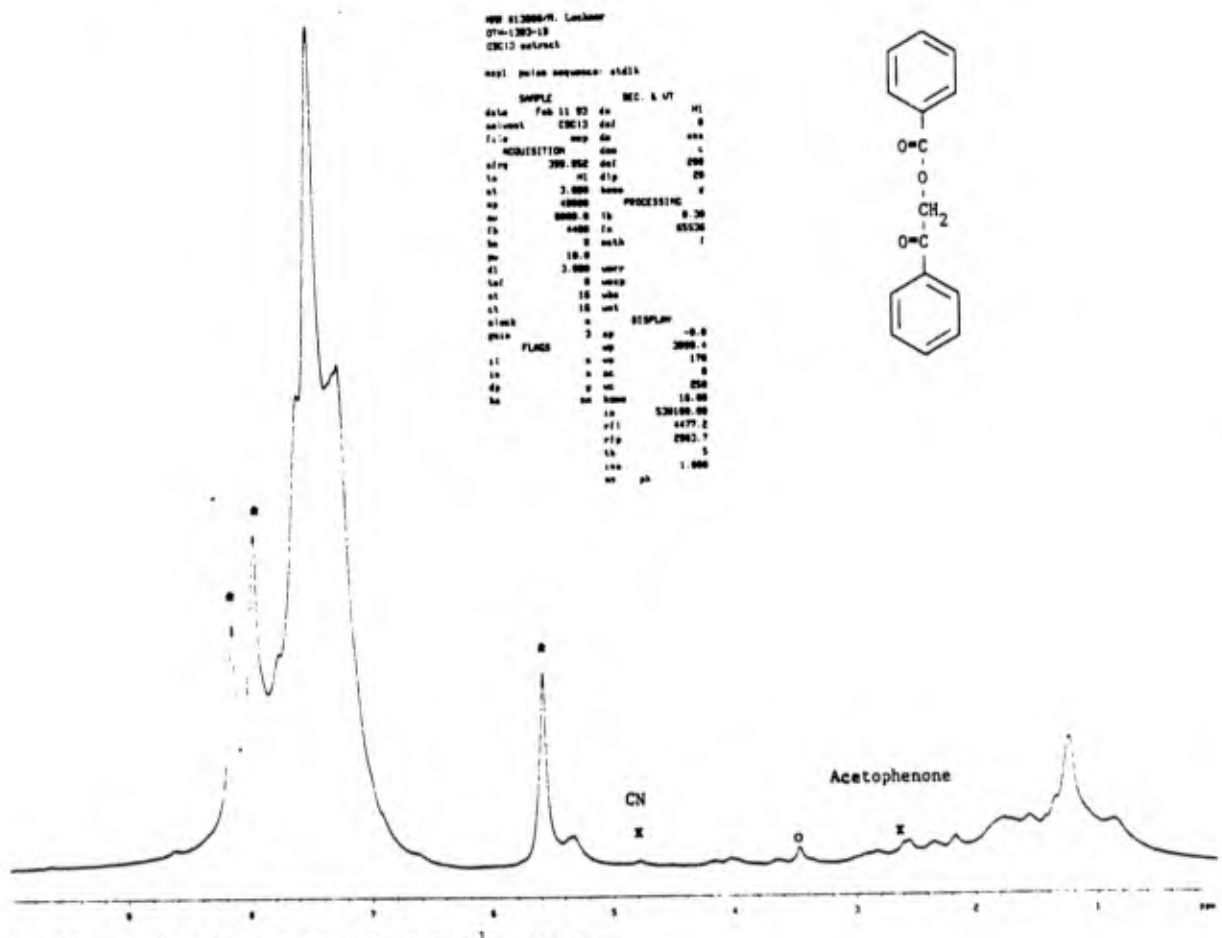
NMR-58. OTH-1193-5c, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectra.



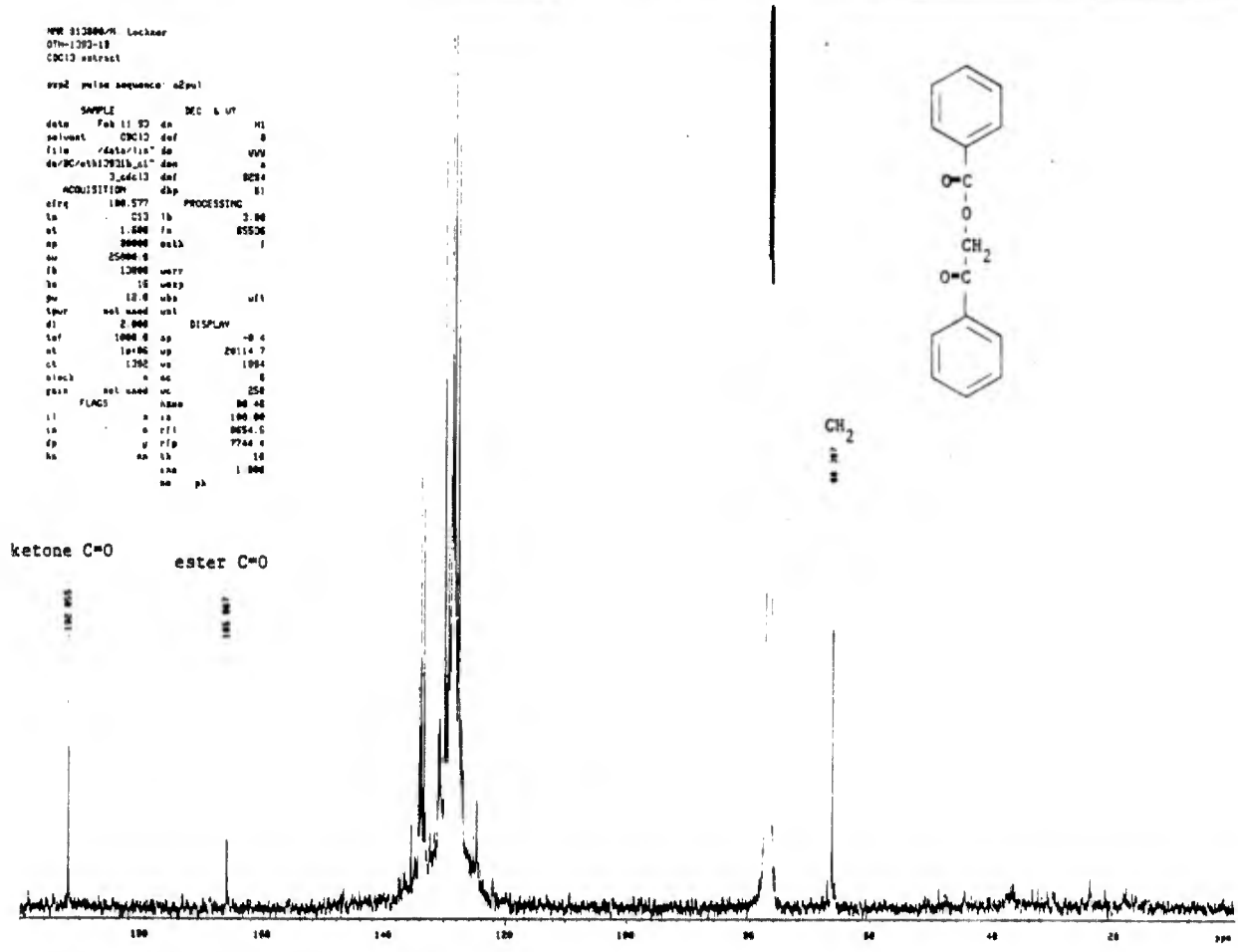
NMR-59. OTH-1193-5c, $CDCl_3$ extract: 2-D COSY spectrum.



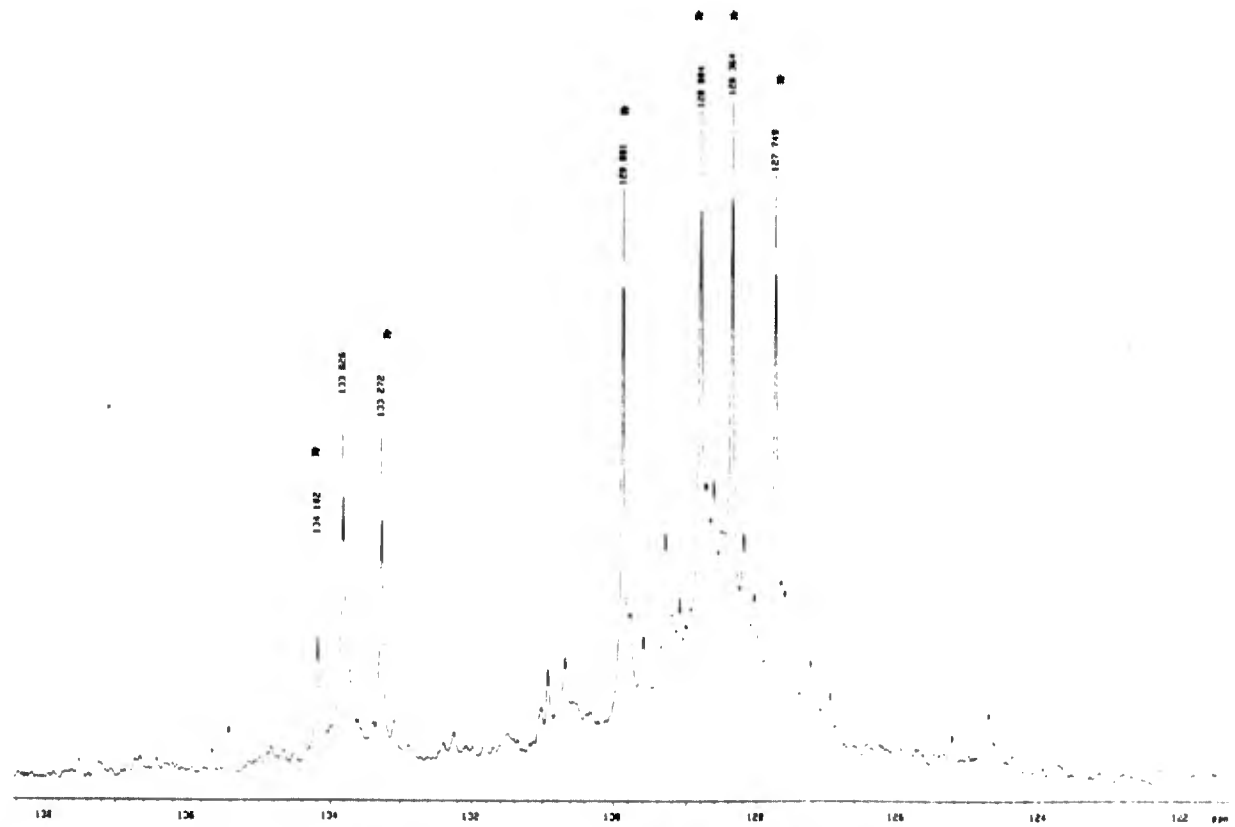
NMR-60. OTH-1393-1b, D_2O extract: ^1H, 400 MHz.



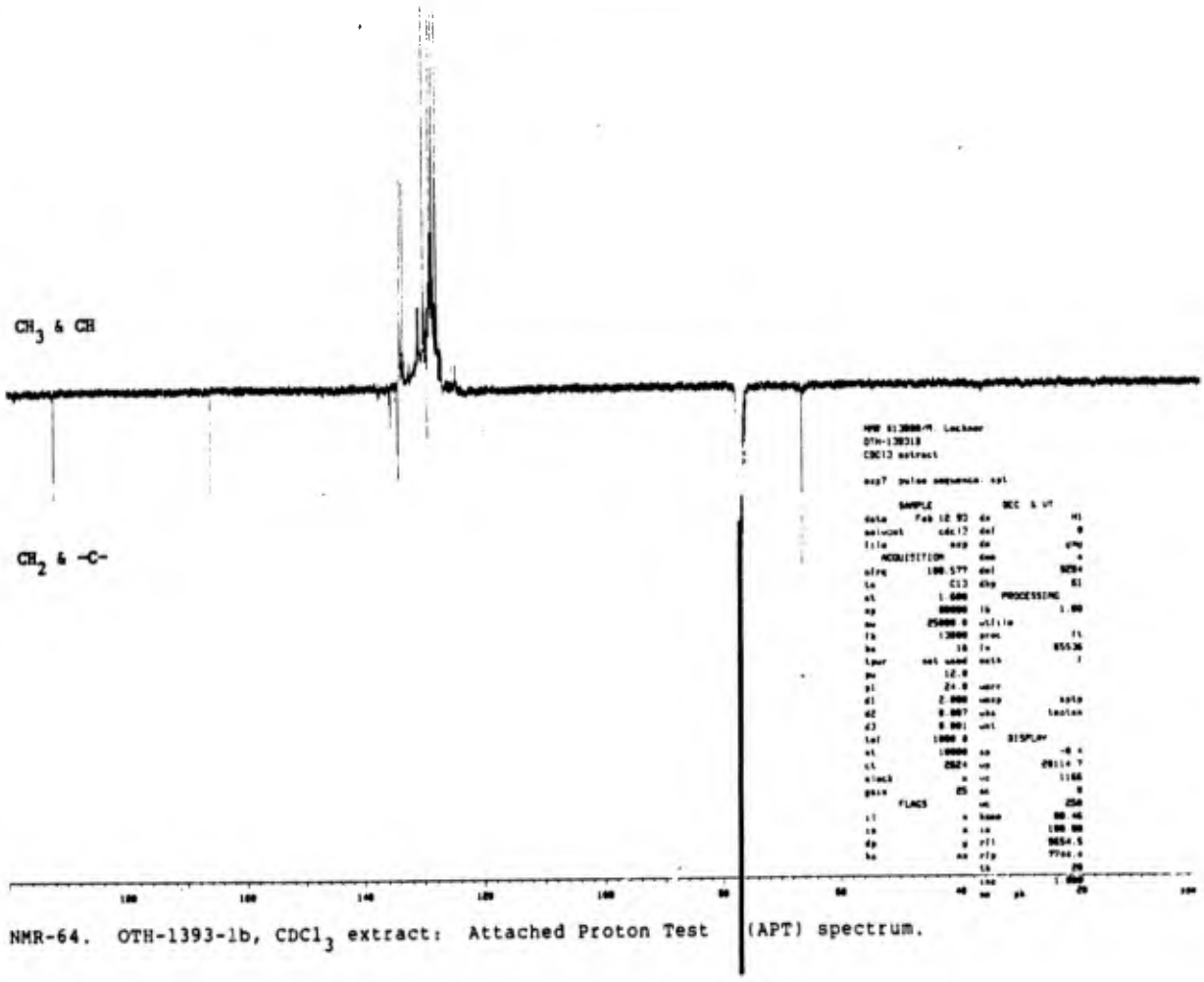
NMR-61. OTH-1393-1b, $CDCl_3$ extract: ^1H, 400 MHz.



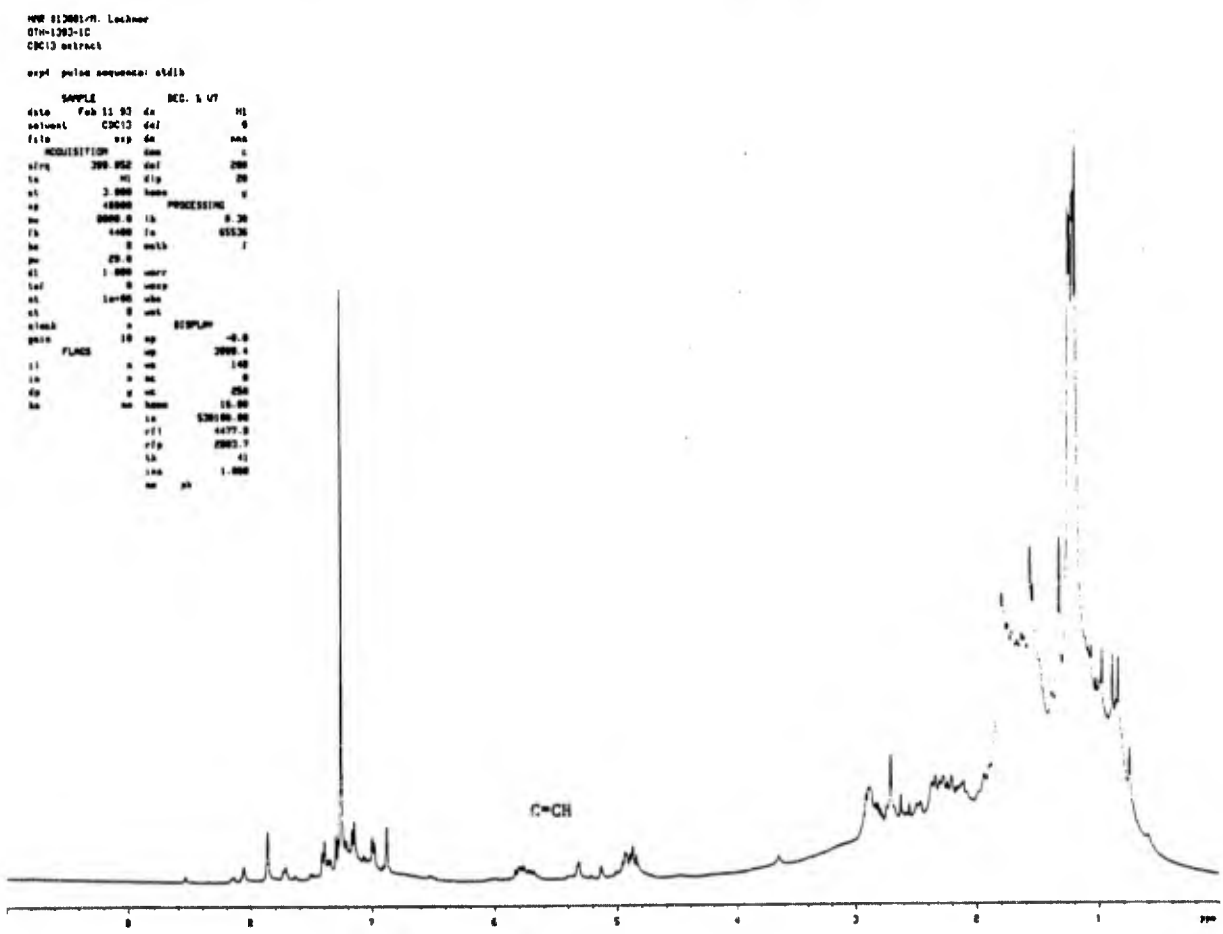
NMR-62. OTH-1393-1b, $CDCl_3$ extract: ^{13}C, 100 MHz.



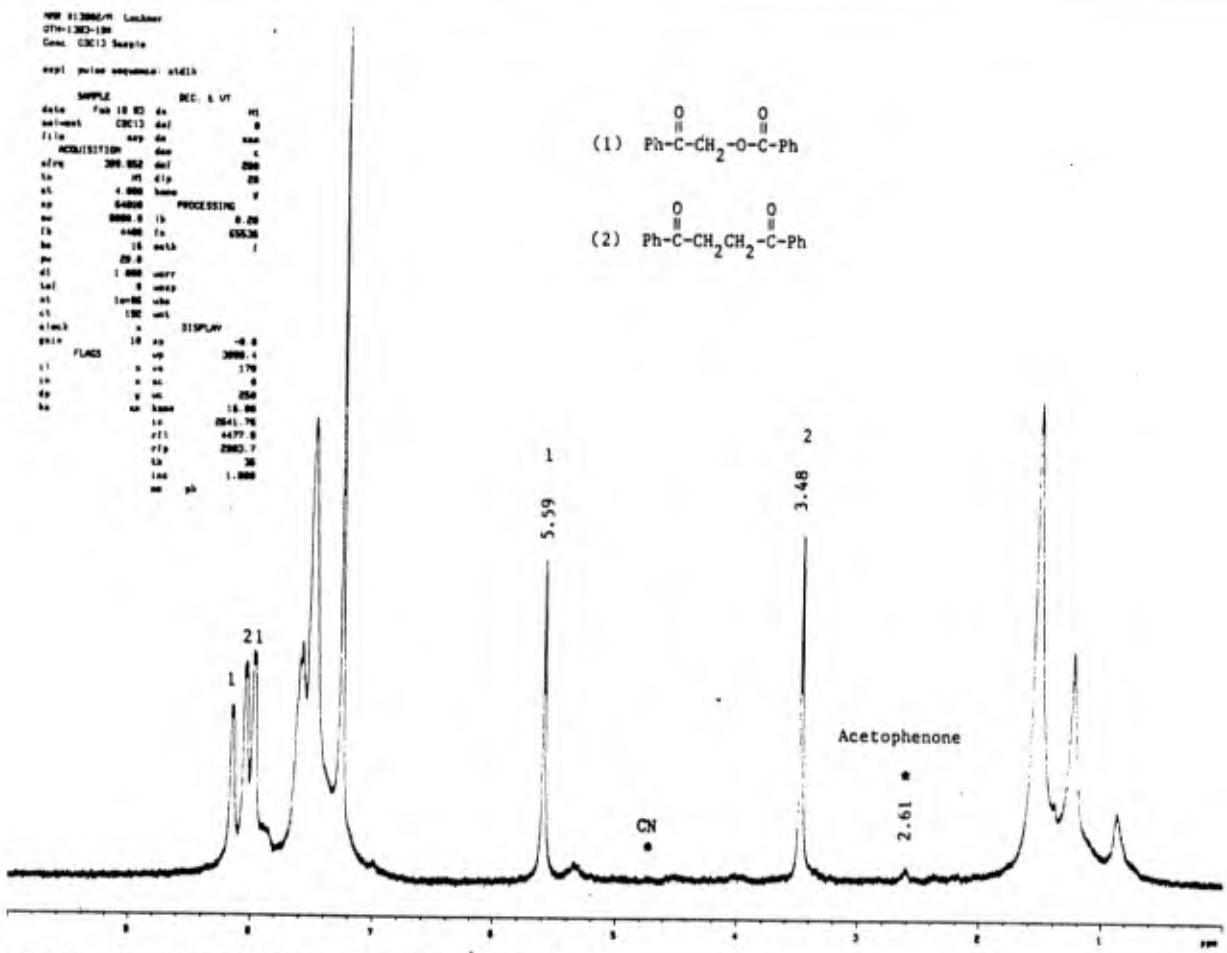
NMR-63. OTH-1393-1b, $CDCl_3$ extract: ^{13}C, 100 MHz, expanded spectrum.



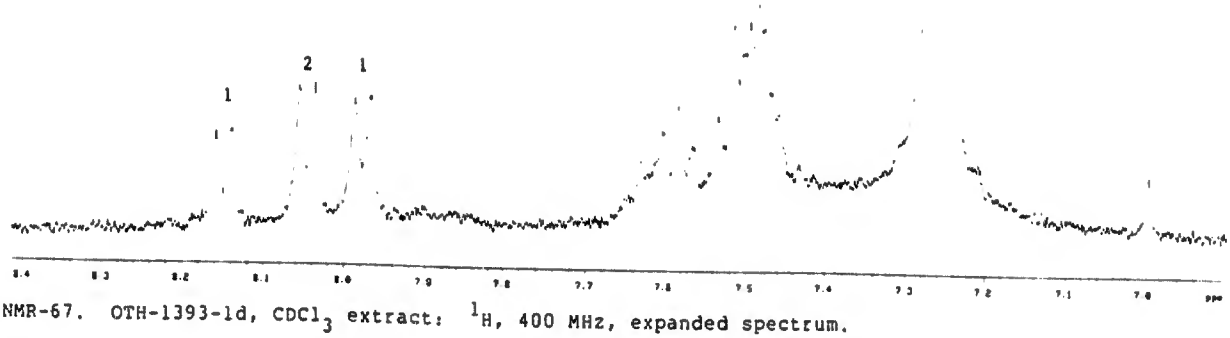
NMR-64. OTH-1393-1b, $CDCl_3$ extract: Attached Proton Test (APT) spectrum.



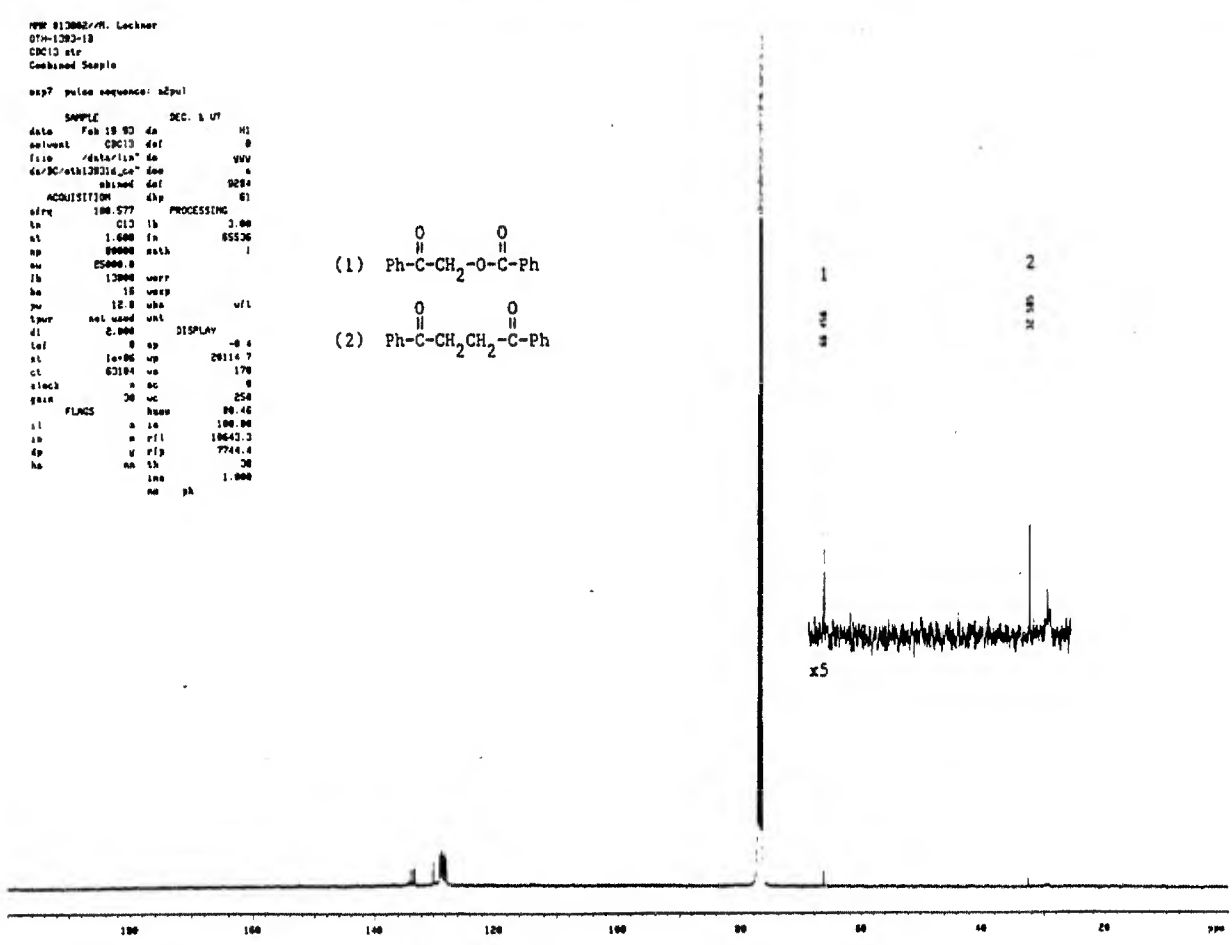
NMR-65. OTH-1393-1c, $CDCl_3$ extract: ^1H, 400 MHz.



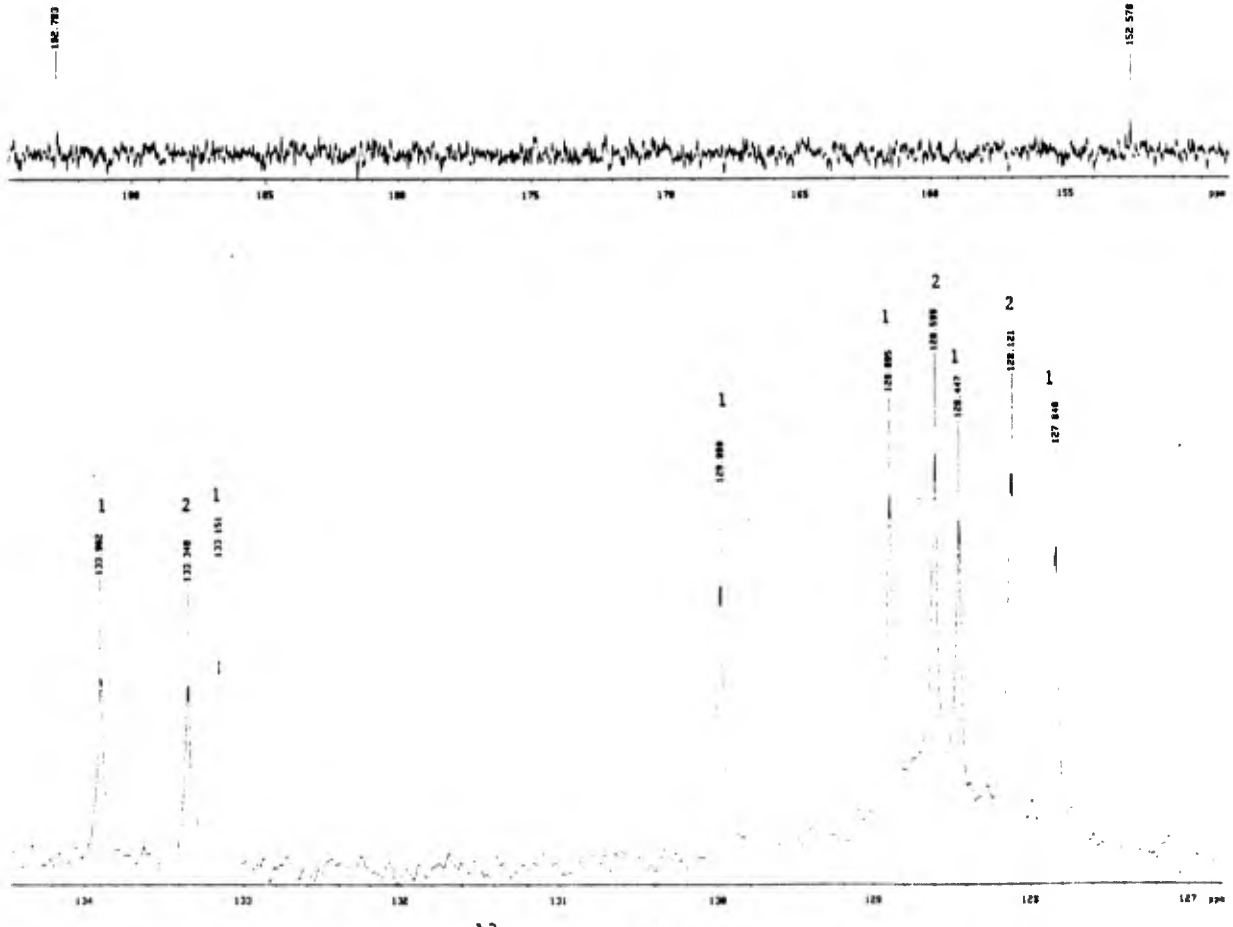
NMR-66. OTH-1393-1d, $CDCl_3$ extract: ^1H, 400 MHz.



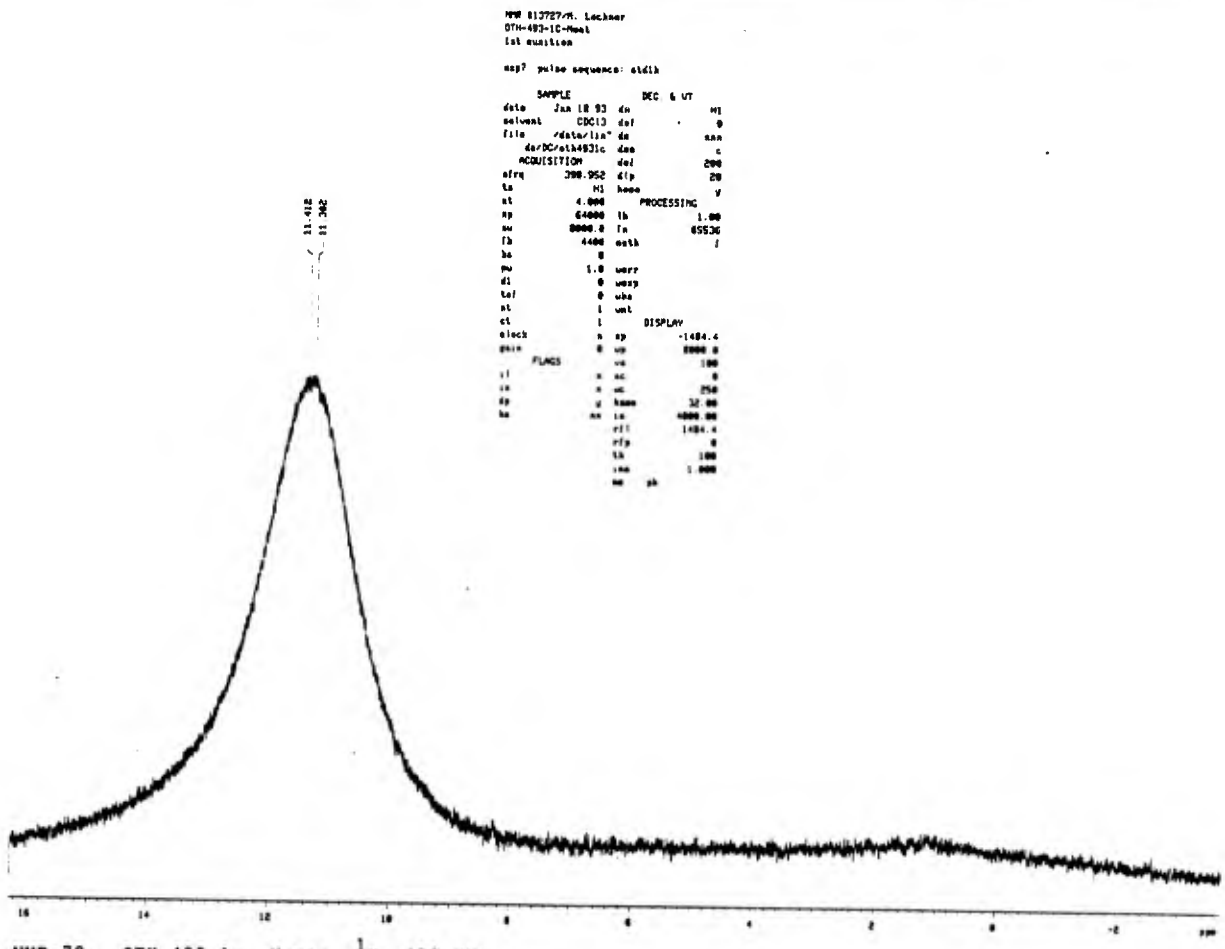
NMR-67. OTH-1393-1d, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



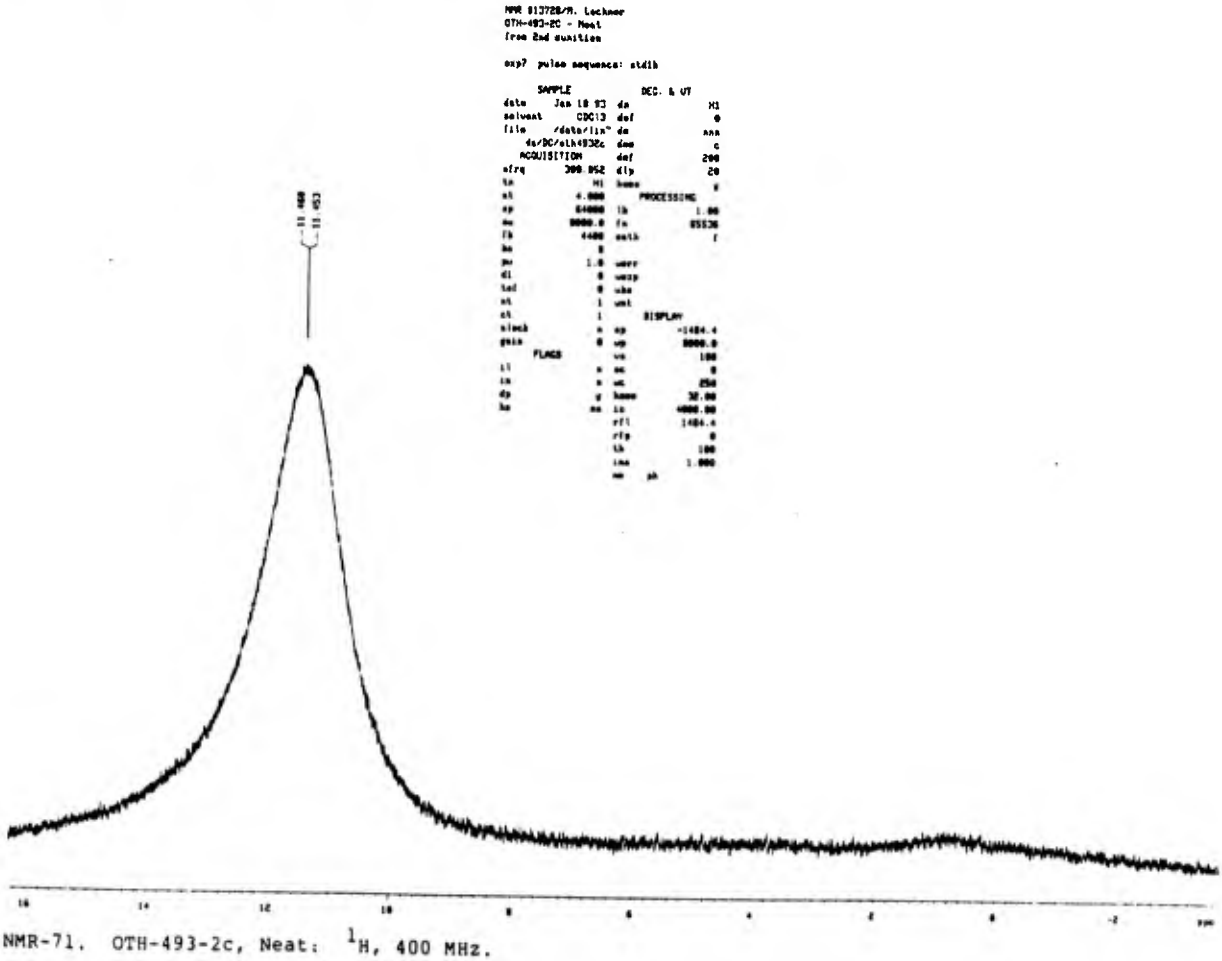
NMR-68. OTH-1393-1d, $CDCl_3$ extract: ^{13}C, 100 MHz.



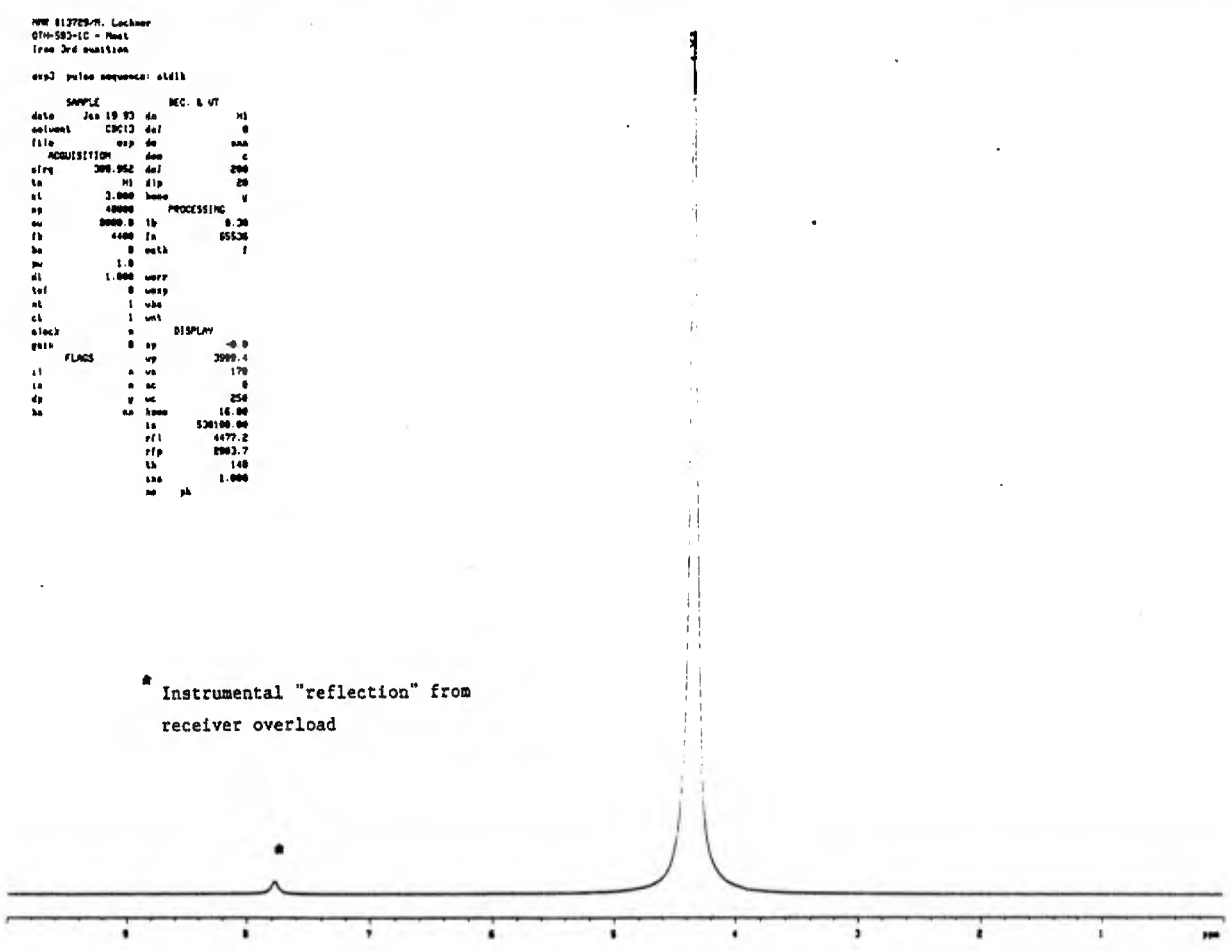
NMR-69. OTH-1393-1d, $CDCl_3$ extract: ^{13}C, 100 MHz, expanded spectra.



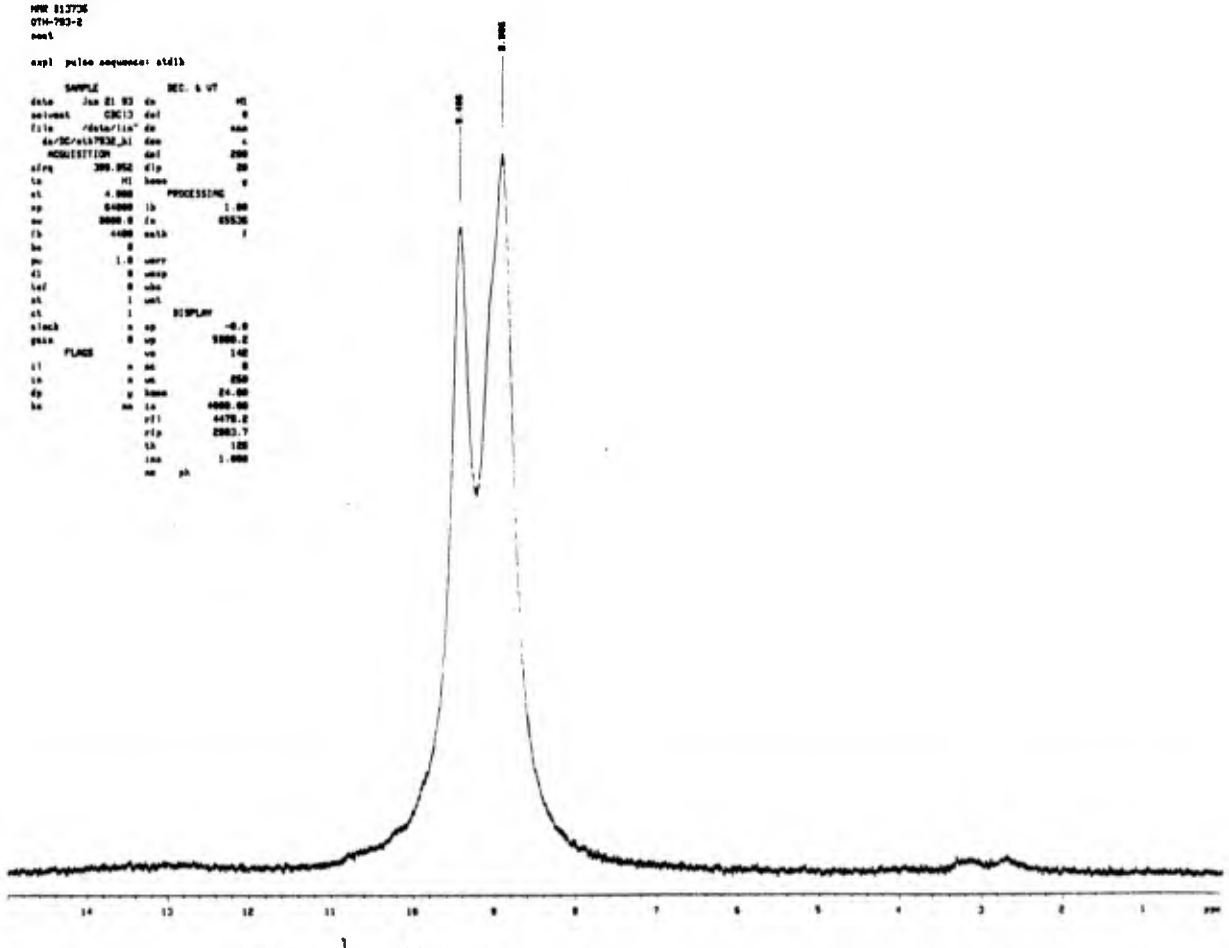
NMR-70. OTH-493-1c, Neat: ^{1}H, 400 MHz.



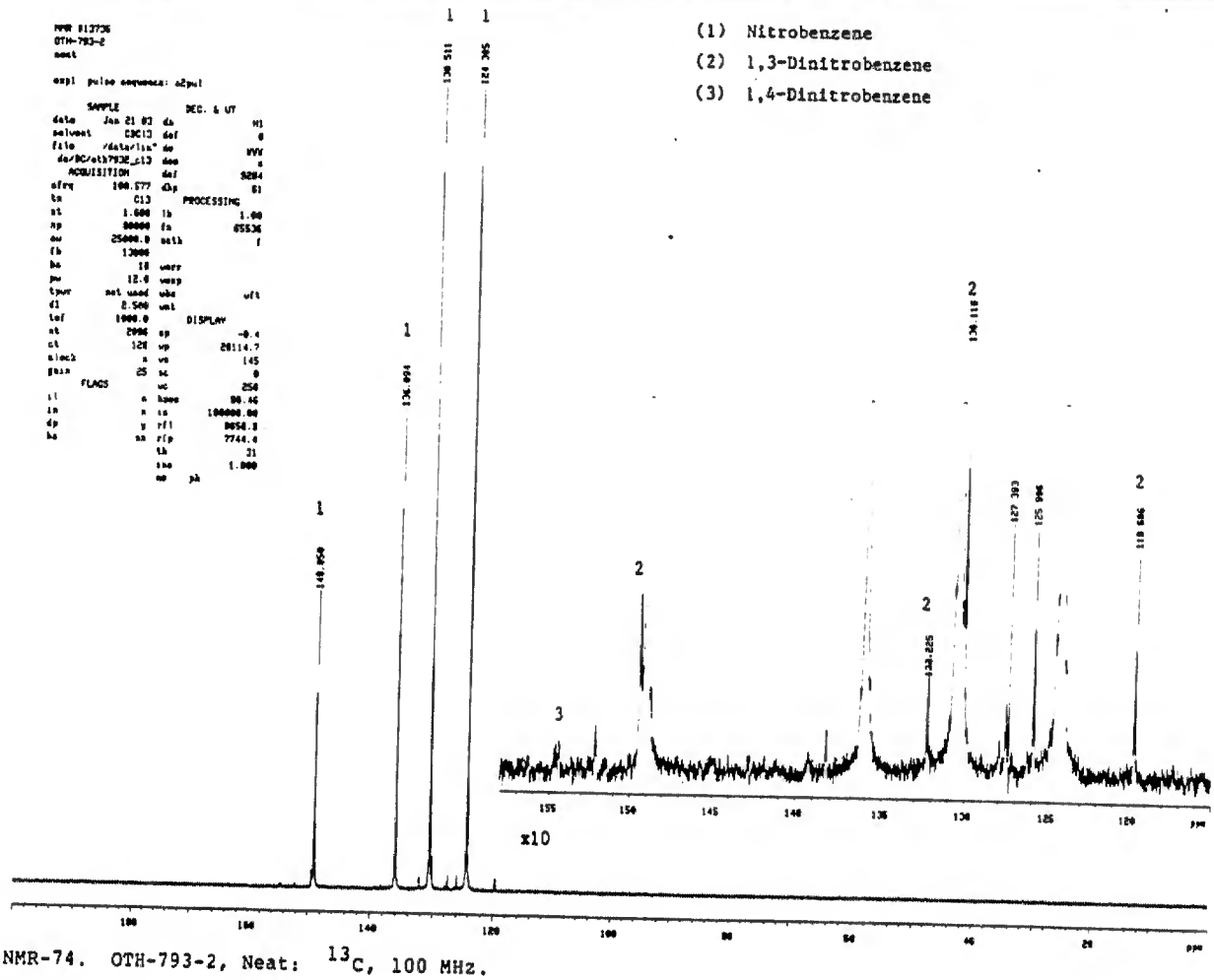
NMR-71. OTH-493-2c, Neat: ^{1}H, 400 MHz.



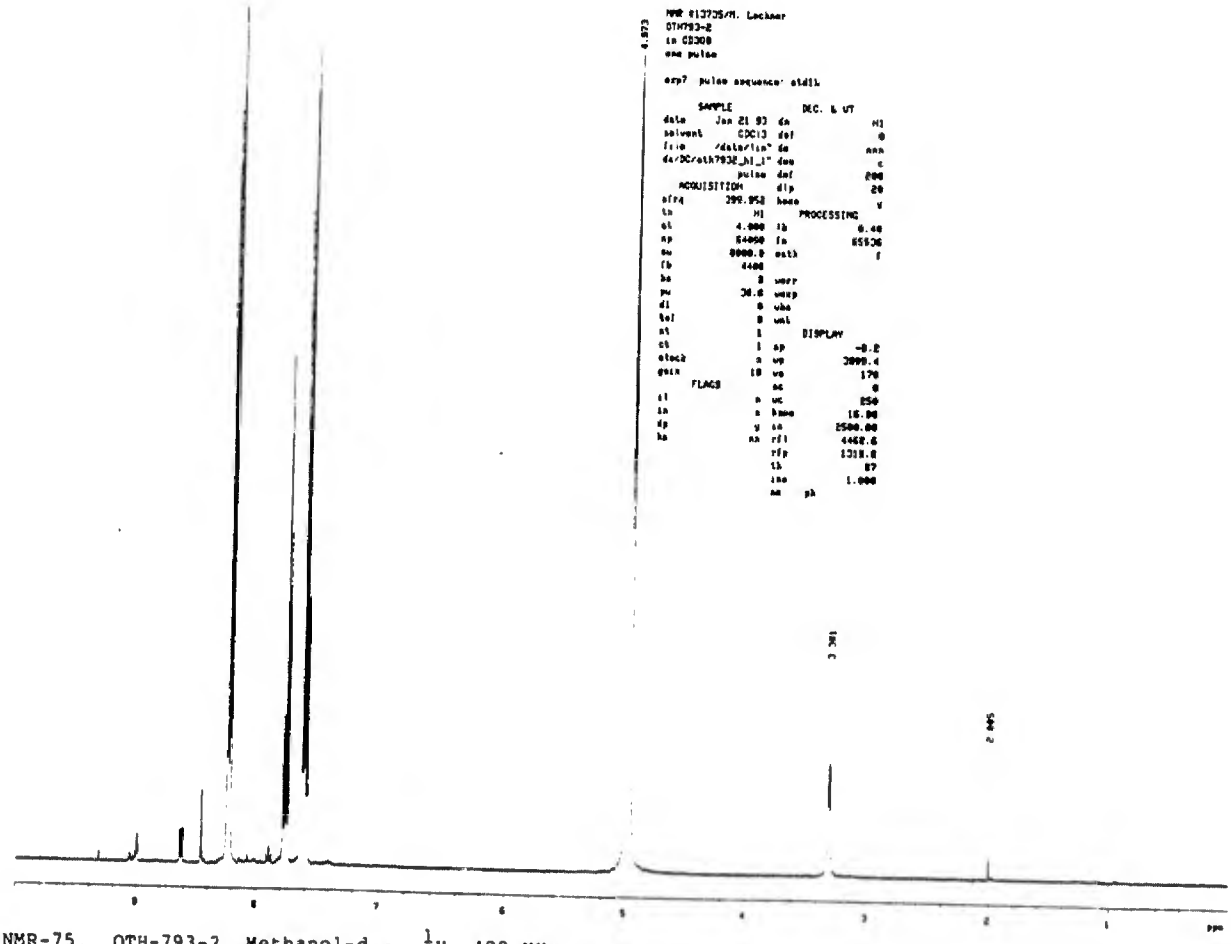
NMR-72. OTH-593-1c, Neat: ^{1}H, 400 MHz.



NMR-73. OTH-793-2, Neat: ^{1}H, 400 MHz.

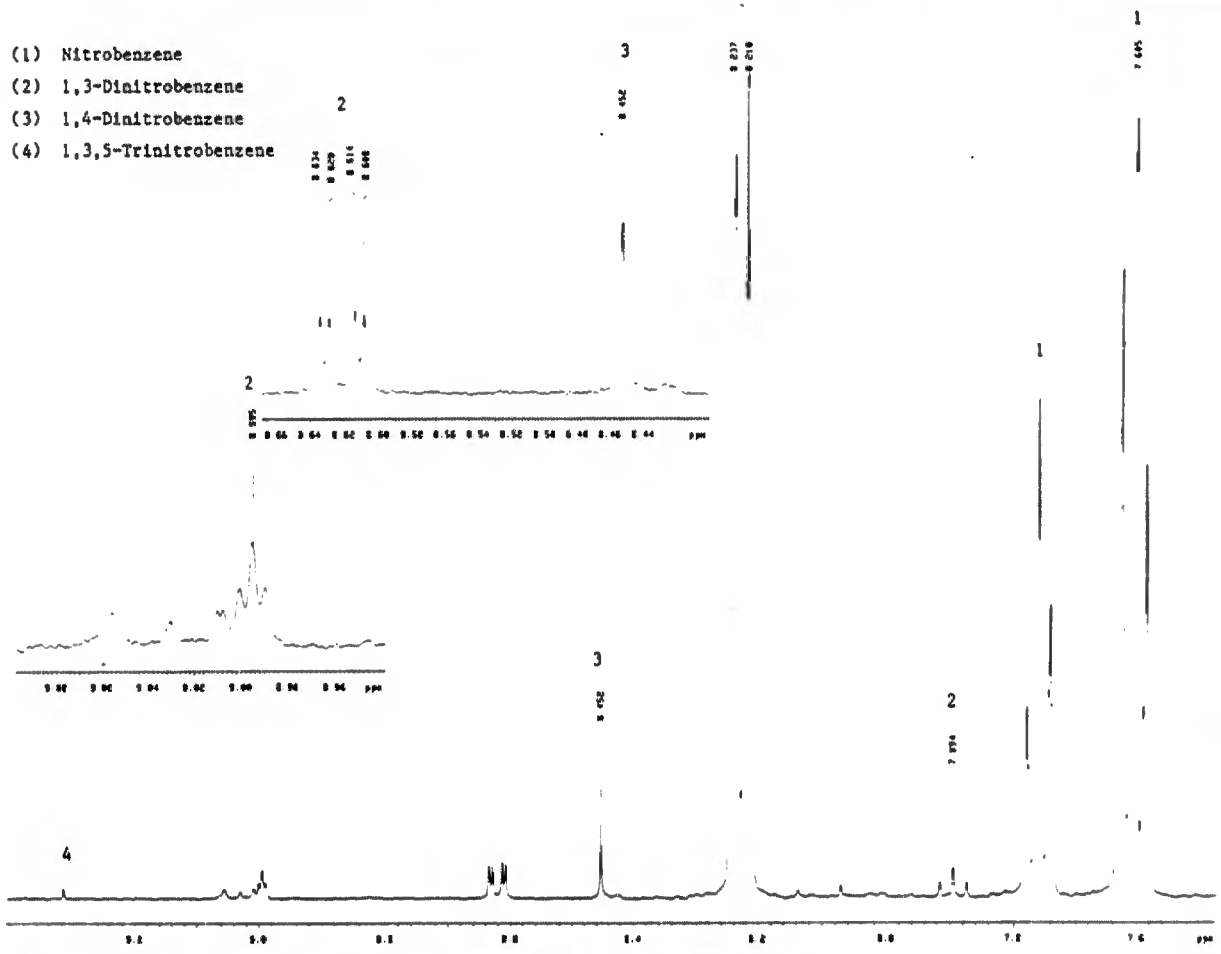


NMR-74. OTH-793-2, Neat: ^{13}C, 100 MHz.

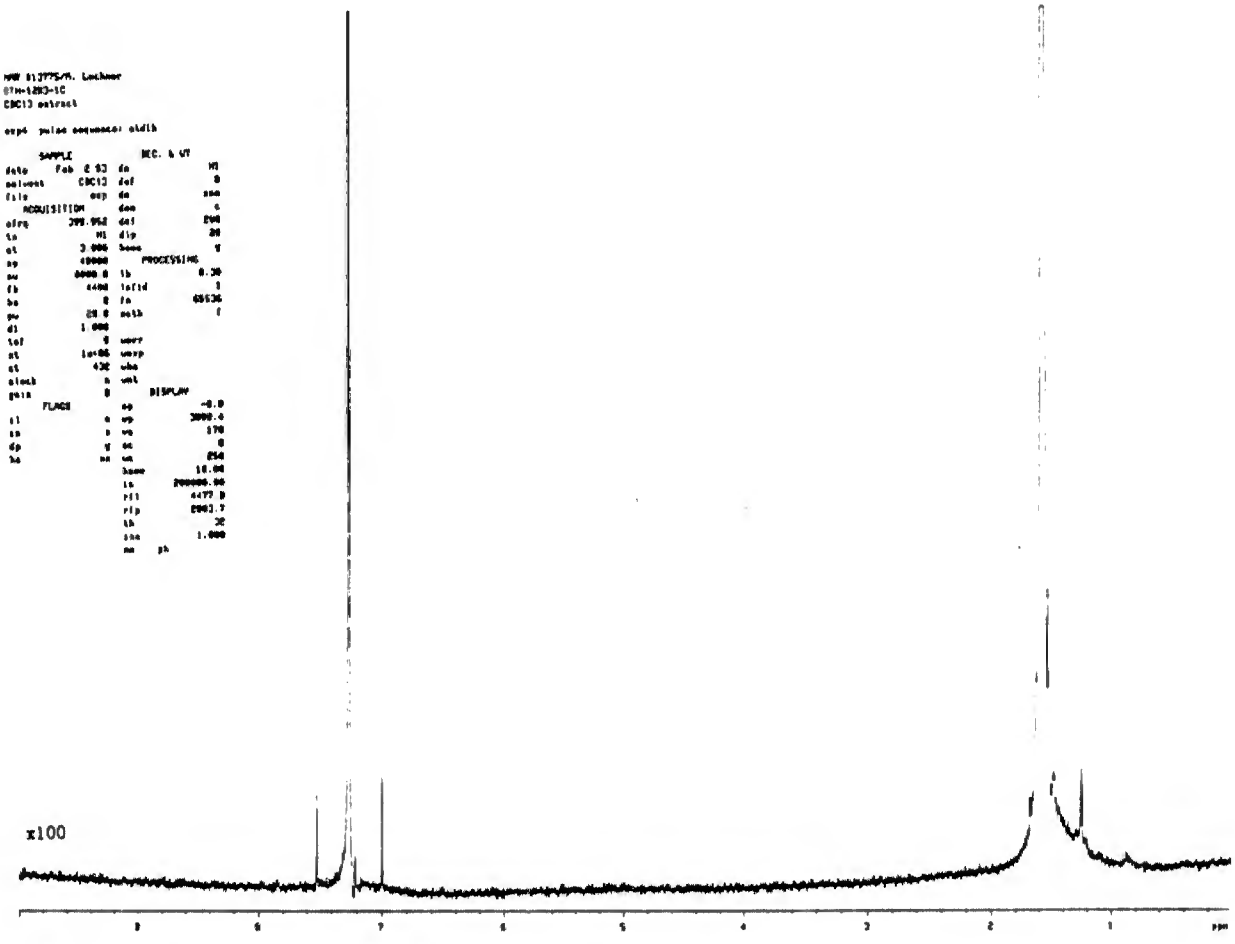


NMR-75. OTH-793-2, Methanol-d_4: ^{1}H, 400 MHz.

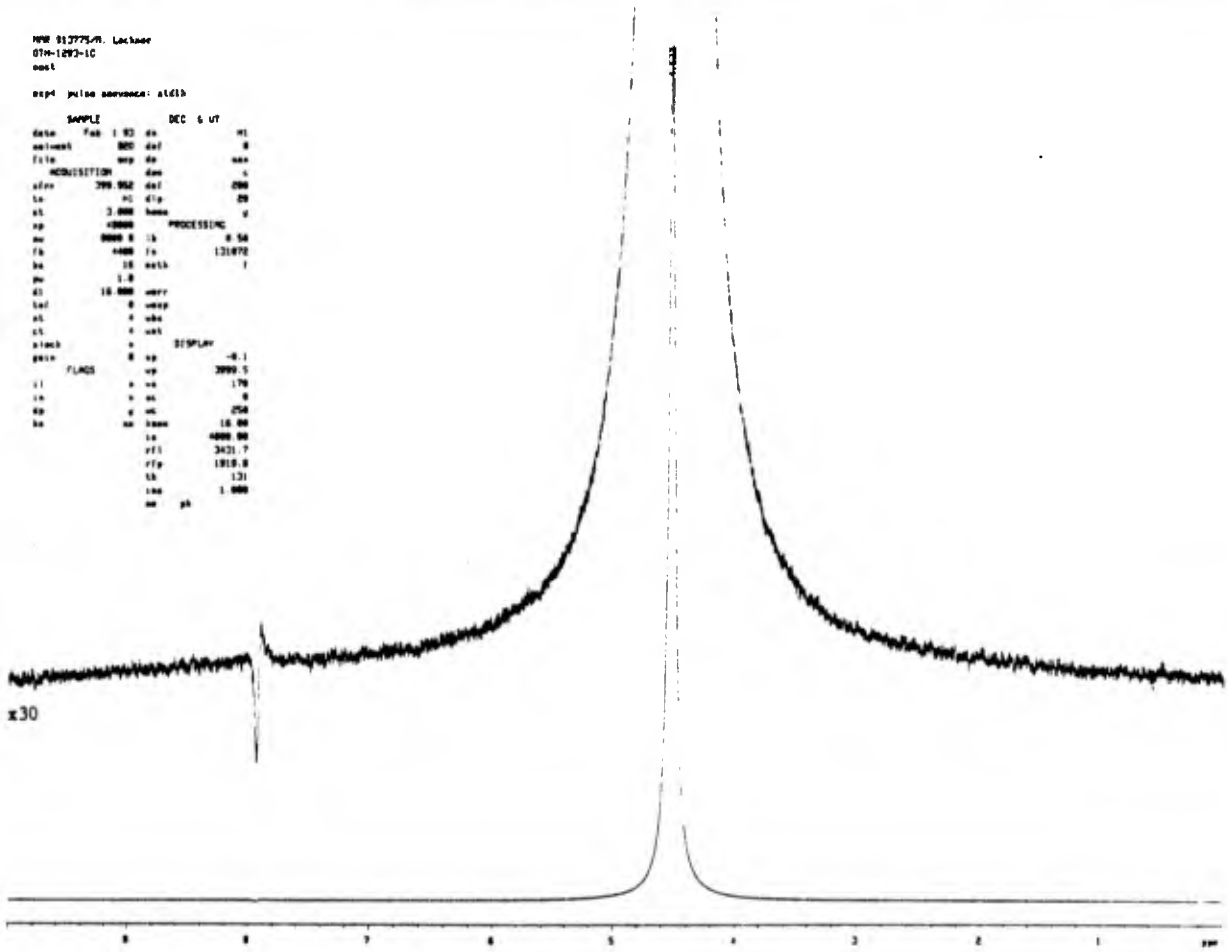
(1) Nitrobenzene
(2) 1,3-Dinitrobenzene
(3) 1,4-Dinitrobenzene
(4) 1,3,5-Trinitrobenzene



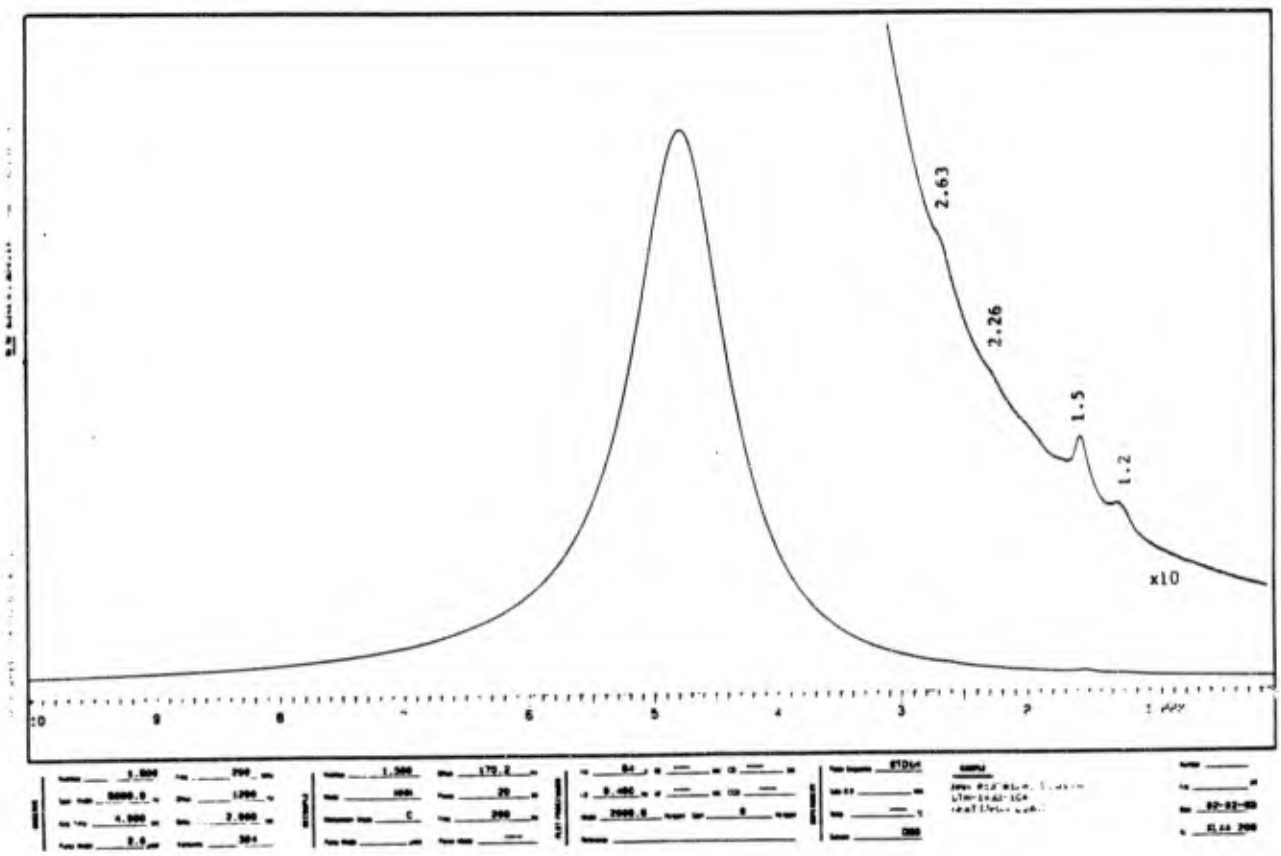
NMR-76. OTH-793-2, Methanol-d_4: ^1H, 400 MHz, expanded spectrum.



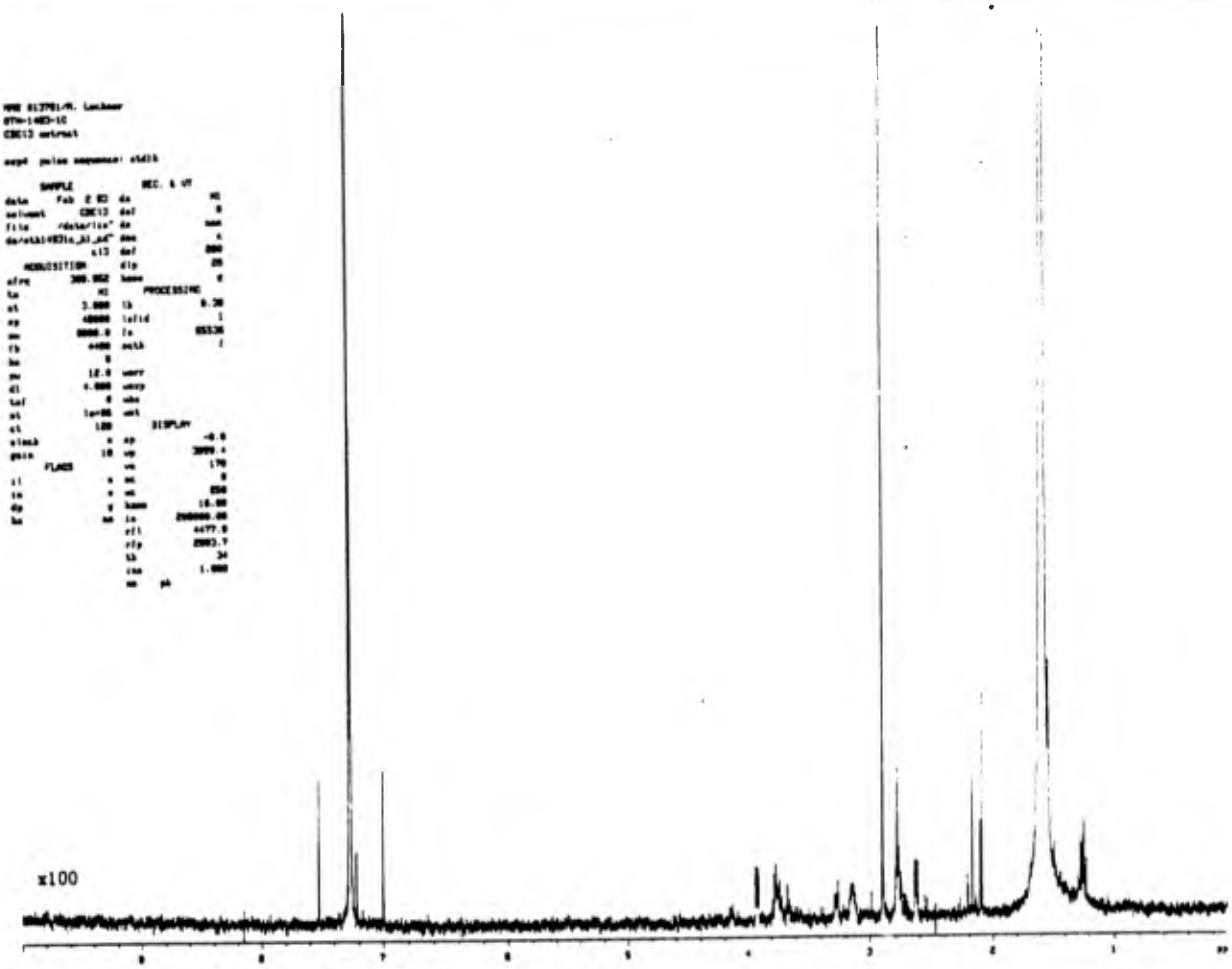
NMR-77. OTH-1293-1c, $CDCl_3$ extract: ^1H, 400 MHz.



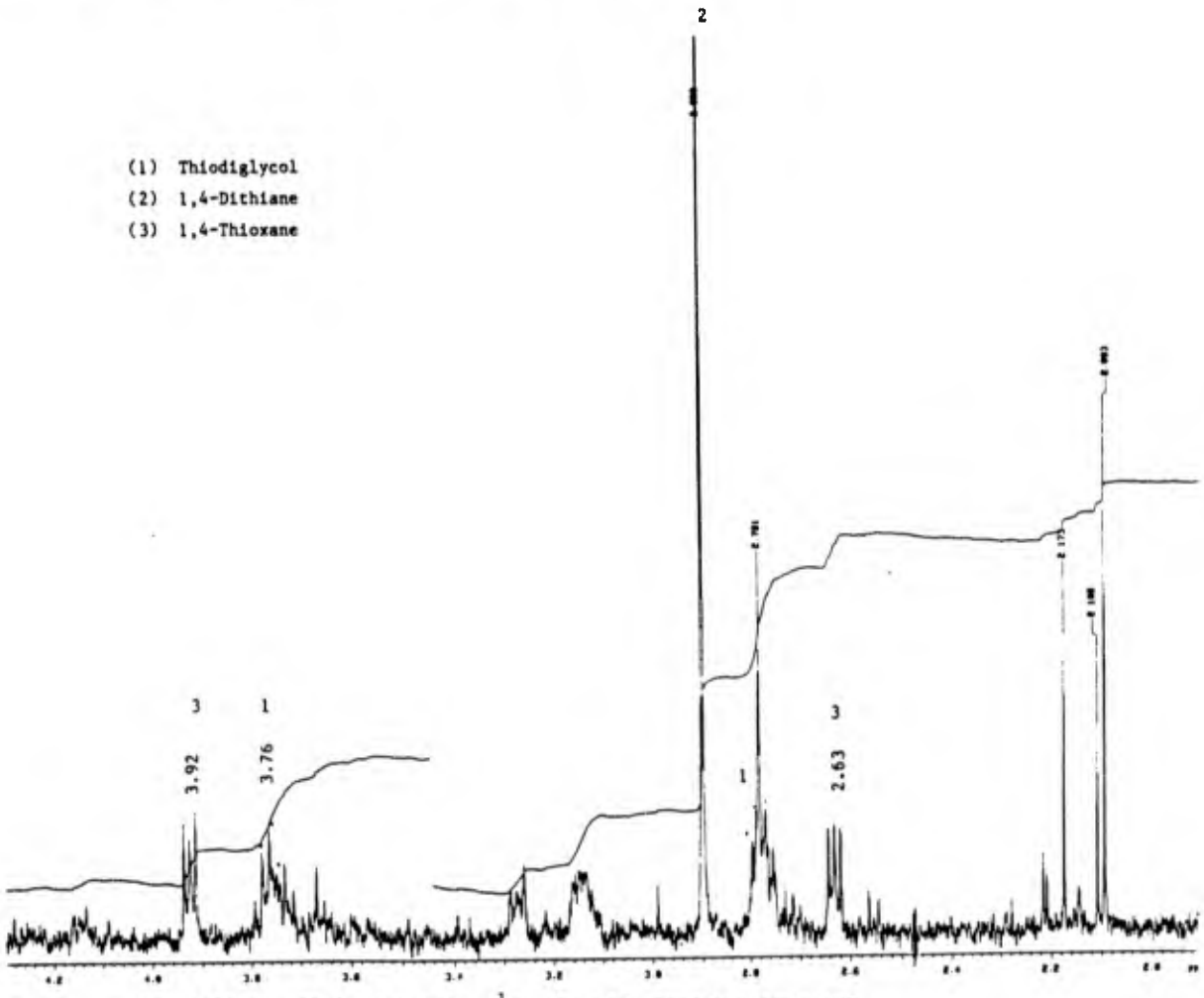
NMR-78. OTH-1293-1c, Neat: ^{1}H, 400 MHz.



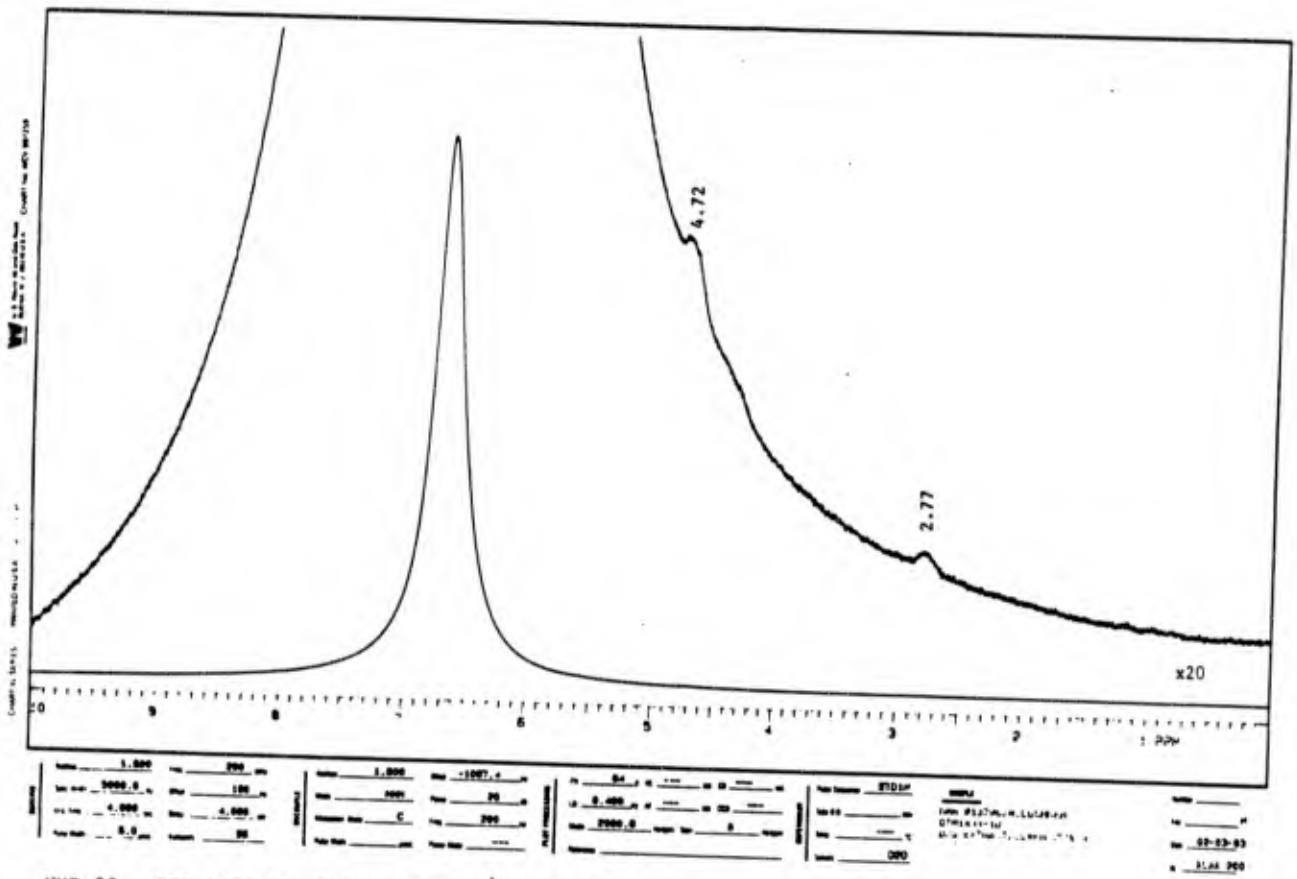
NMR-79. OTH-1493-1c, Neat: ^{1}H, 200 MHz.



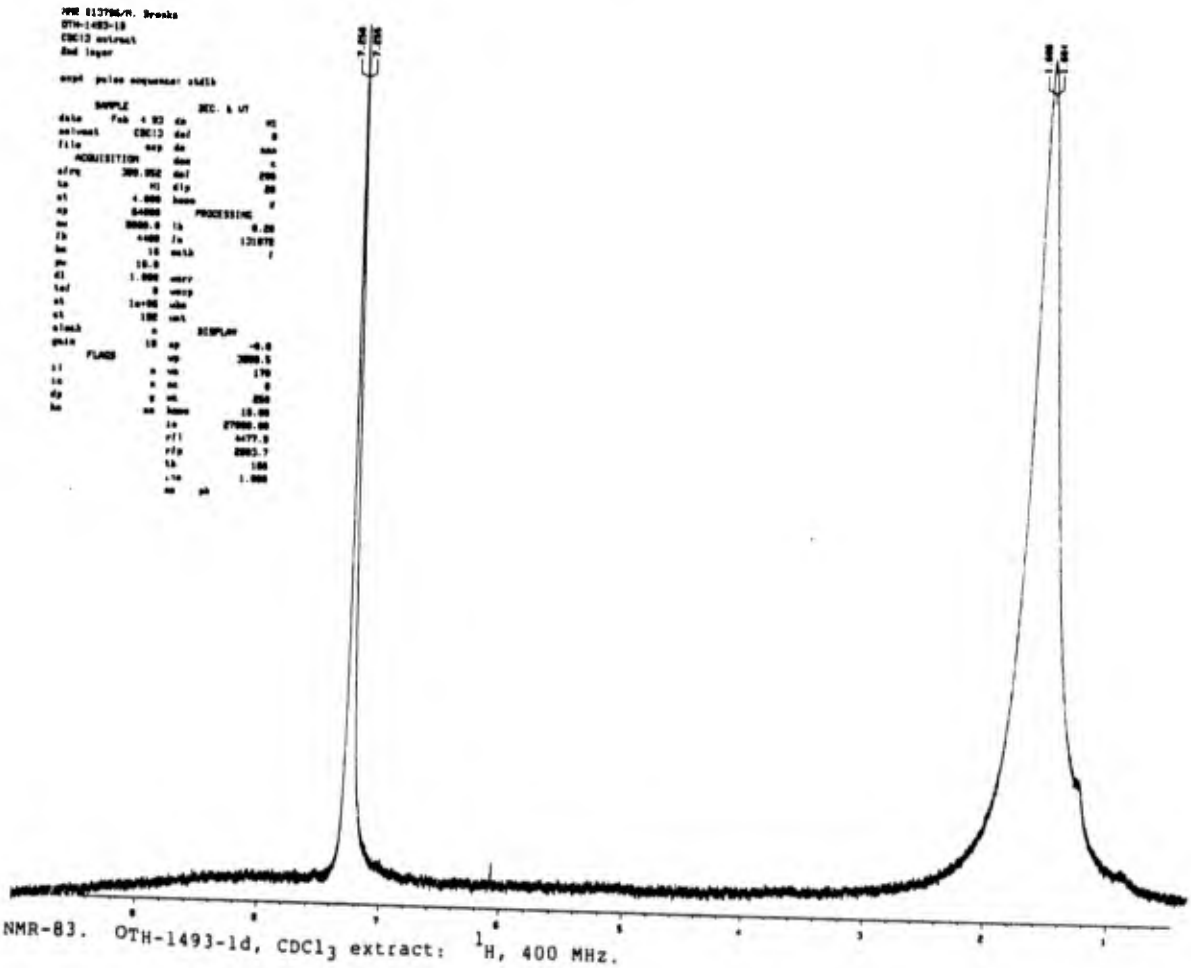
NMR-80. OTH-1493-1c, $CDCl_3$ extract: ^1H, 400 MHz.



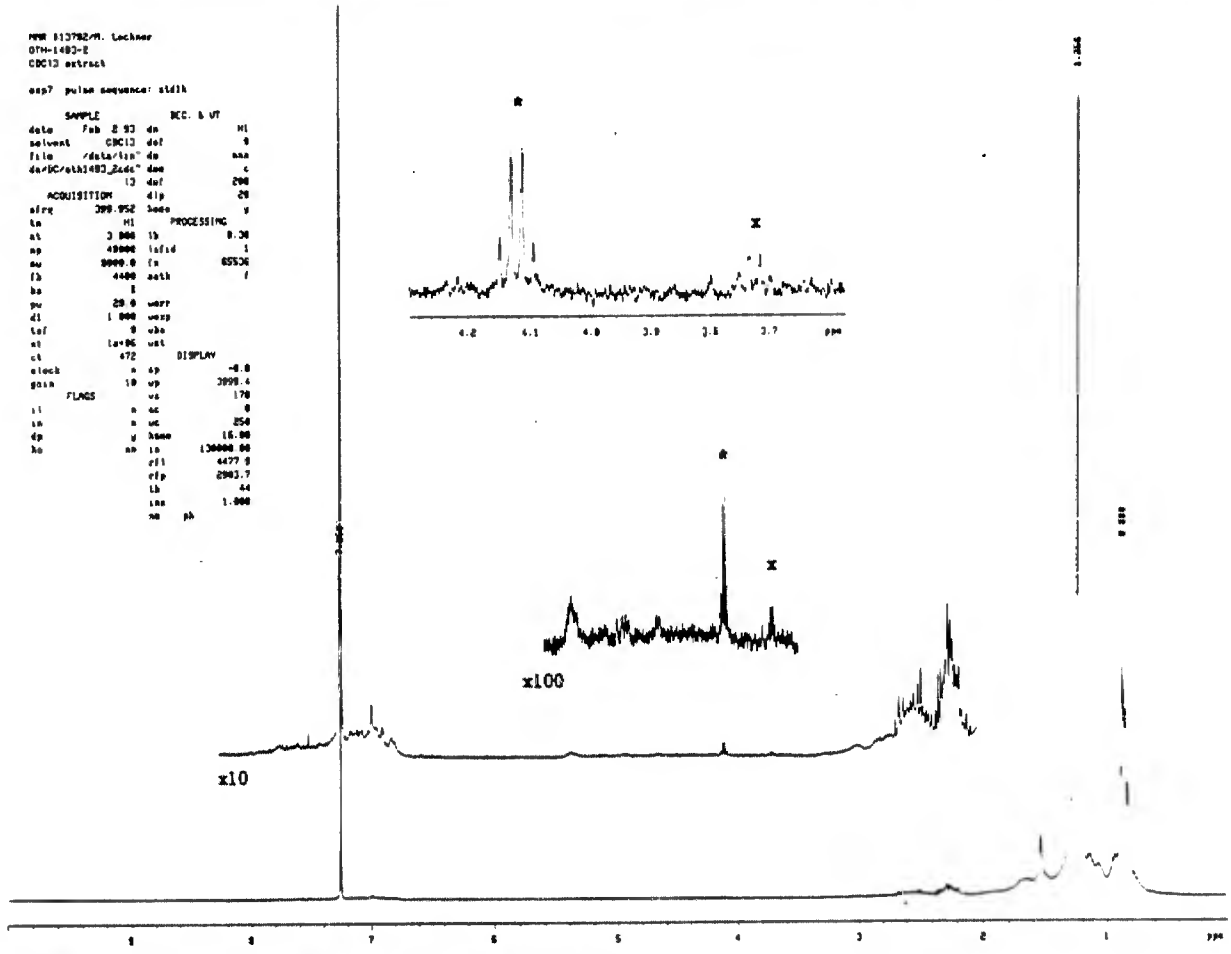
NMR-81. OTH-1493-1c, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



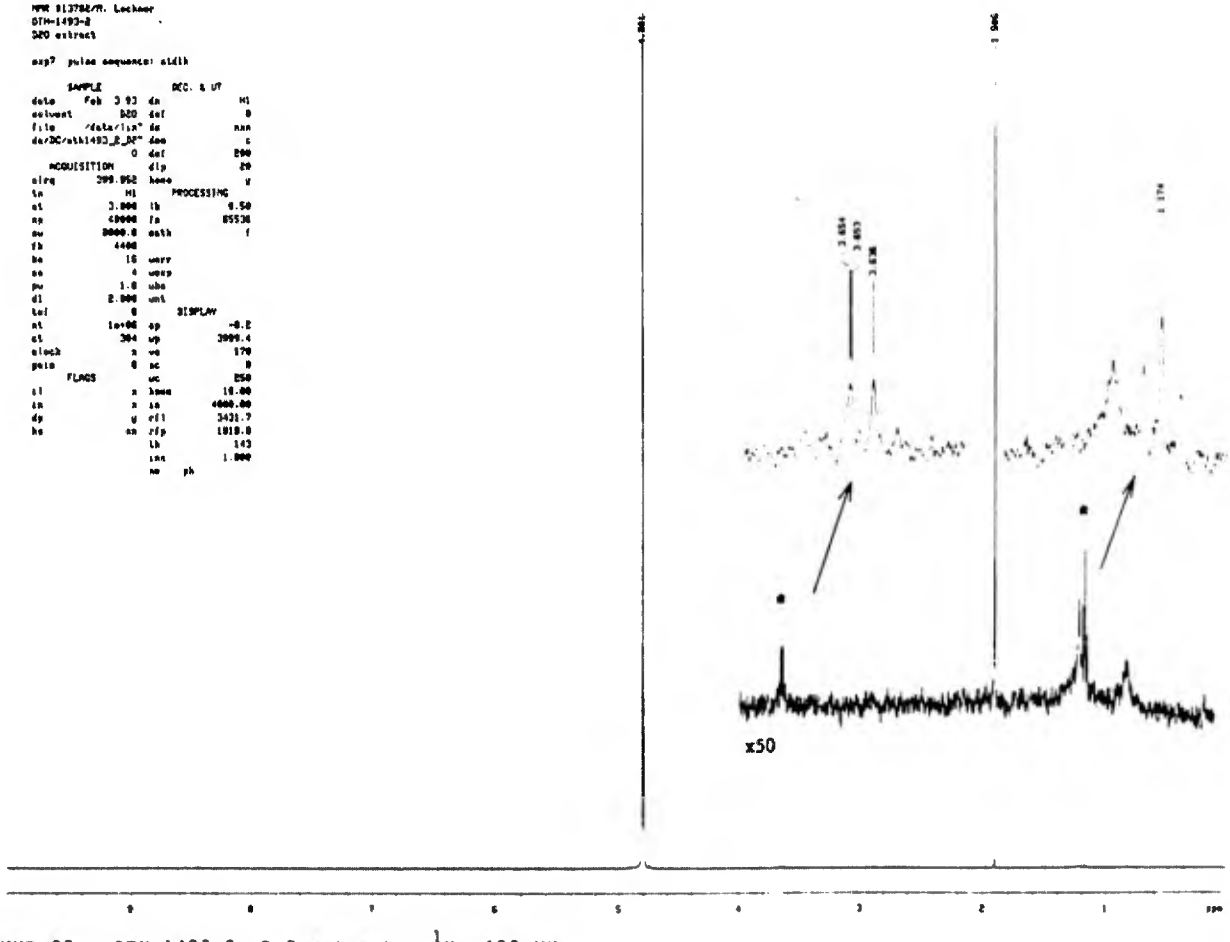
NMR-82. OTH-1493-1d, D_2O extract: ^1H, 200 MHz.



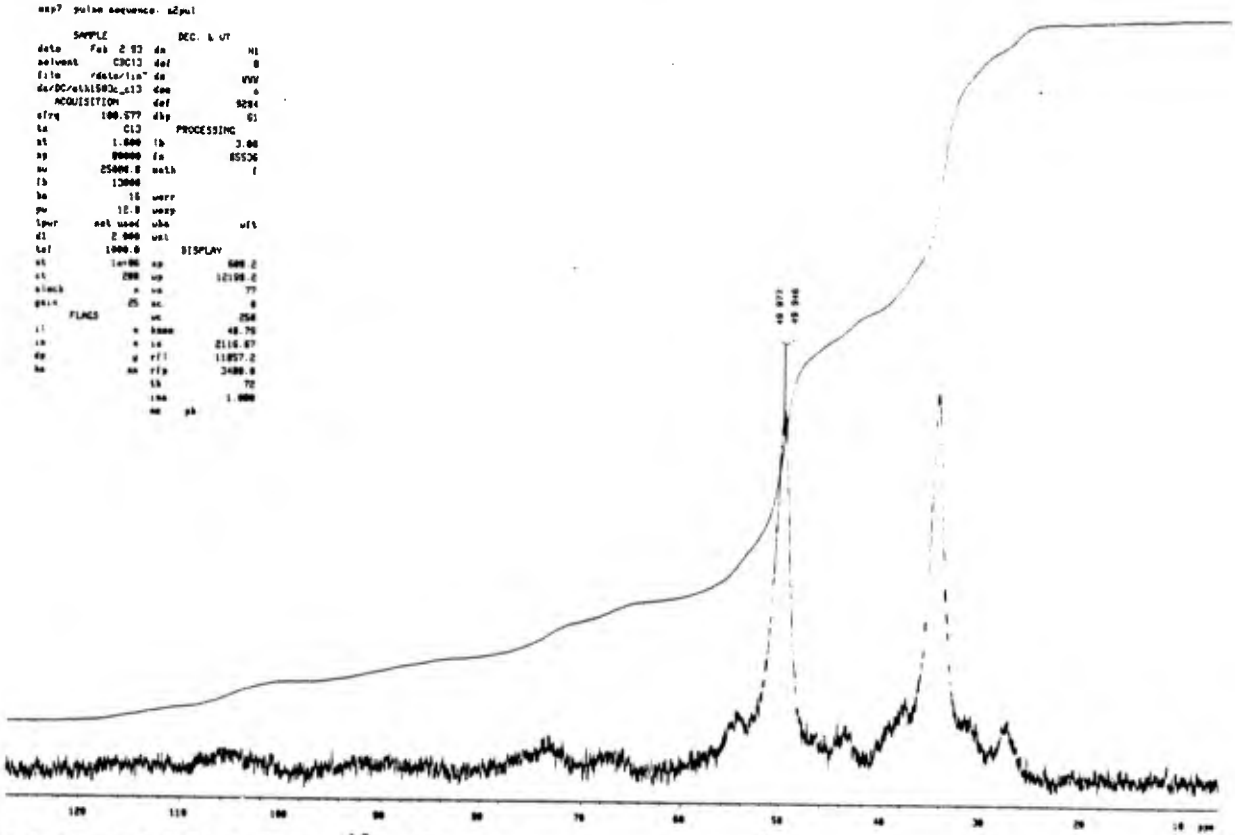
NMR-83. OTH-1493-1d, $CDCl_3$ extract: ^1H, 400 MHz.



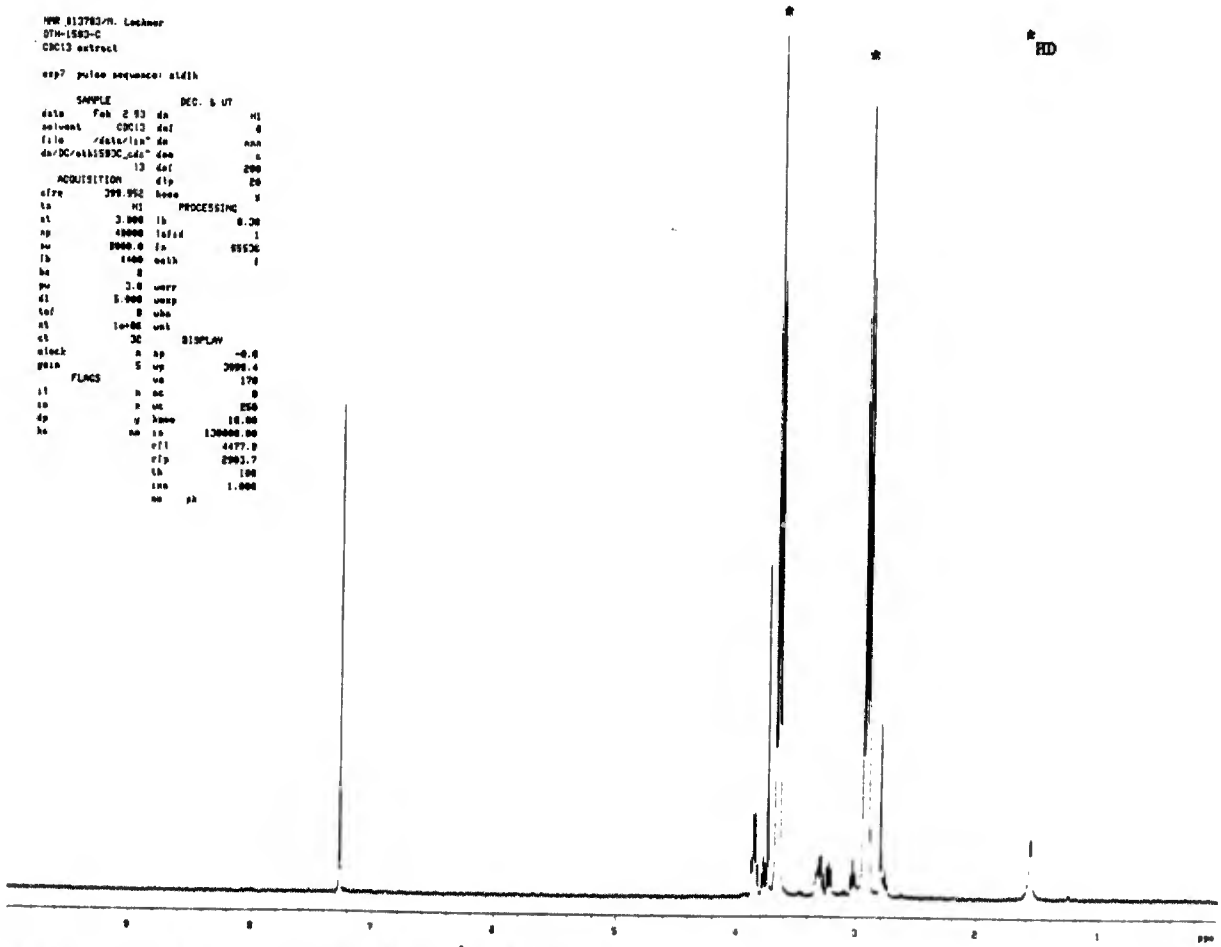
NMR-84. OTH-1493-2, $CDCl_3$ extract: ^1H, 400 MHz.



NMR-85. OTH-1493-2, D_2O extract: ^1H, 400 MHz.

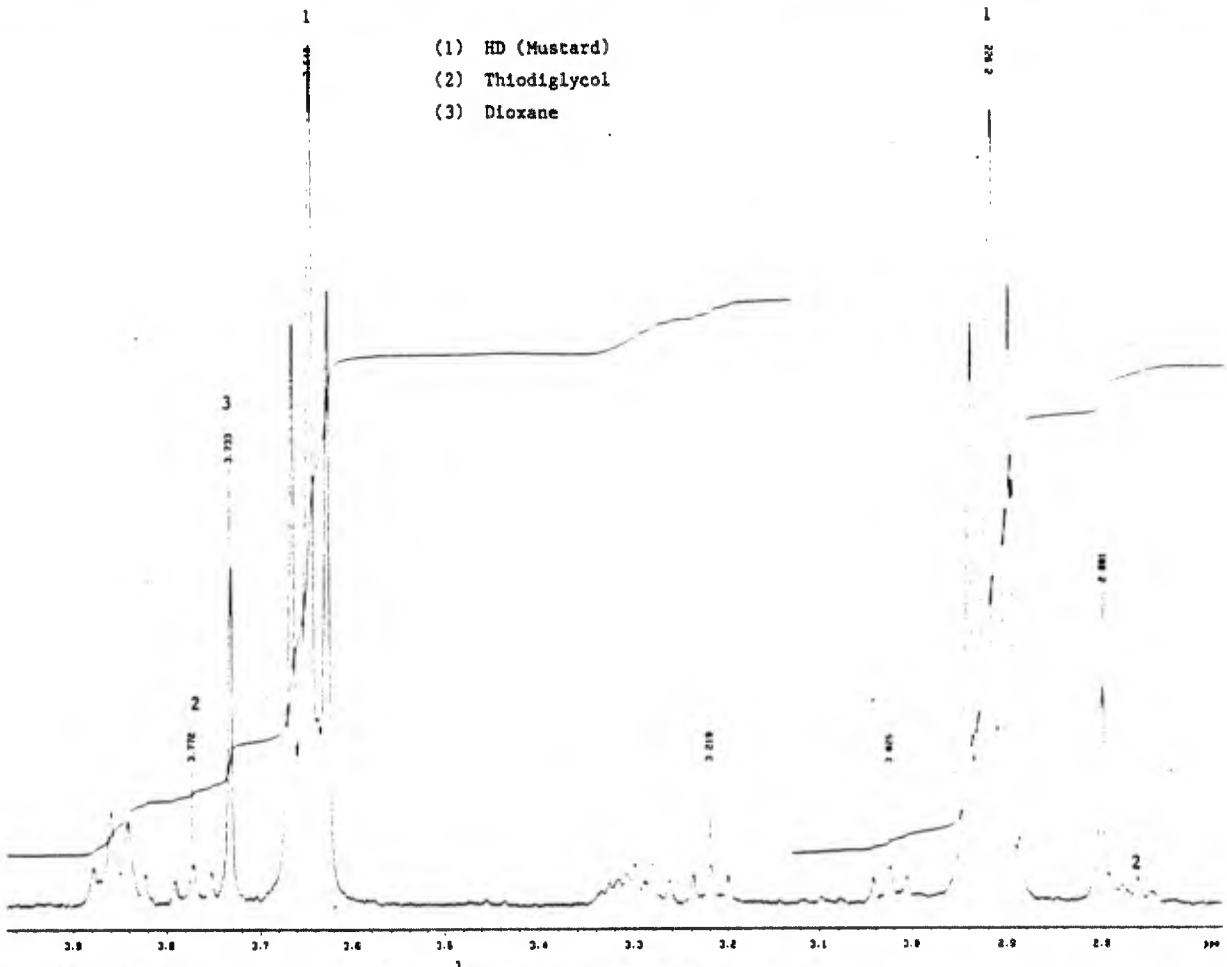


NMR-86. OTH-1593-c, Neat: ^{13}C, 100 MHz.

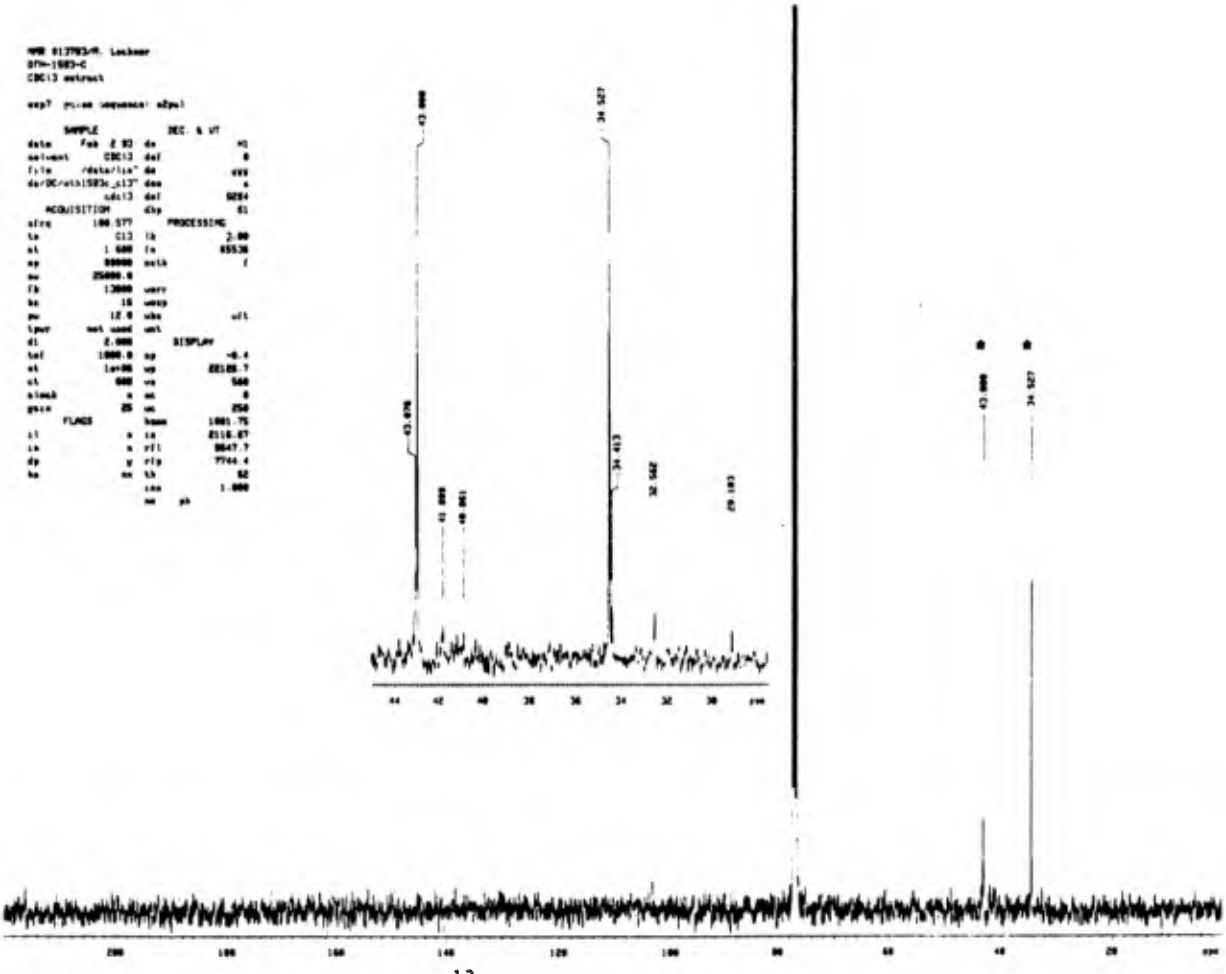


NMR-87. OTH-1593-c, $CDCl_3$ extract: ^{1}H, 400 MHz.

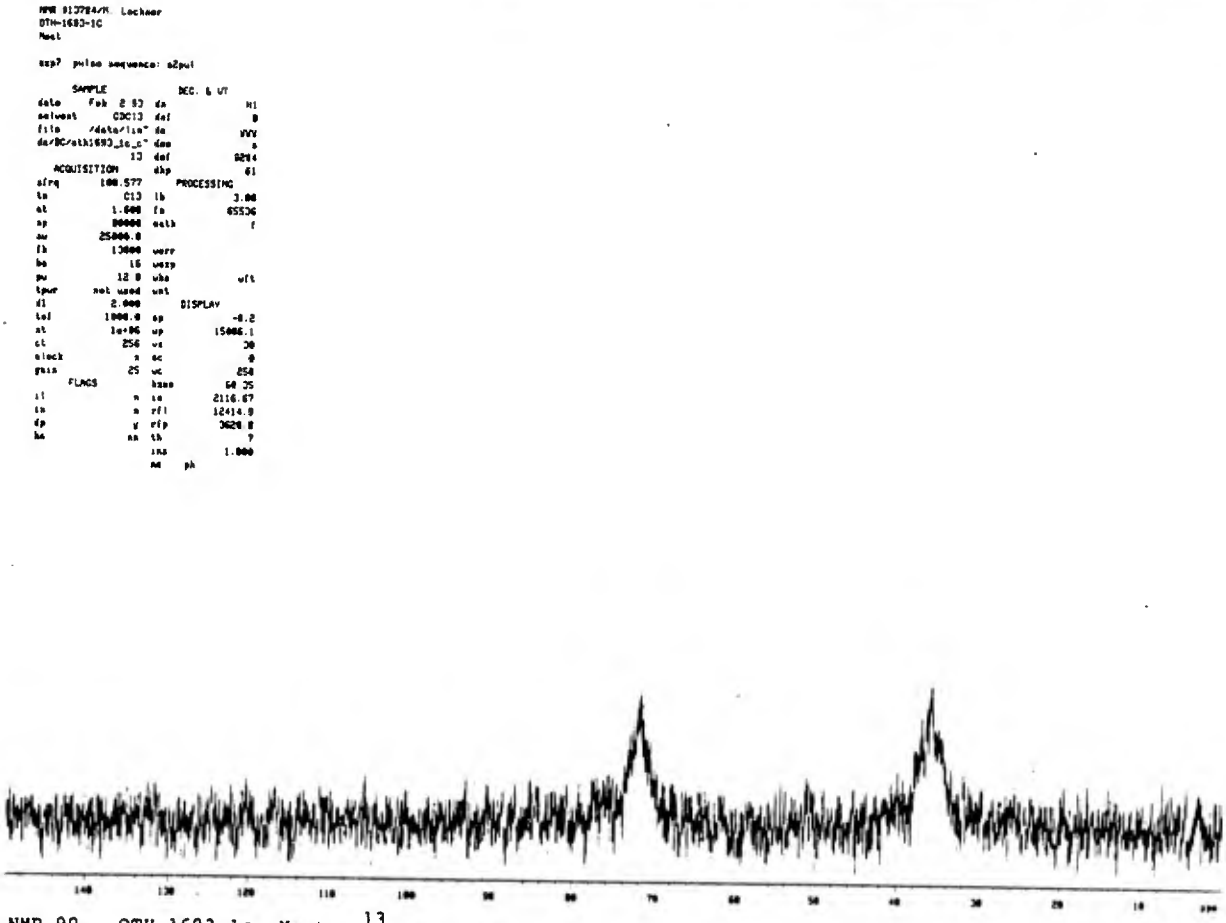
(1) HD (Mustard)
(2) Thiodiglycol
(3) Dioxane



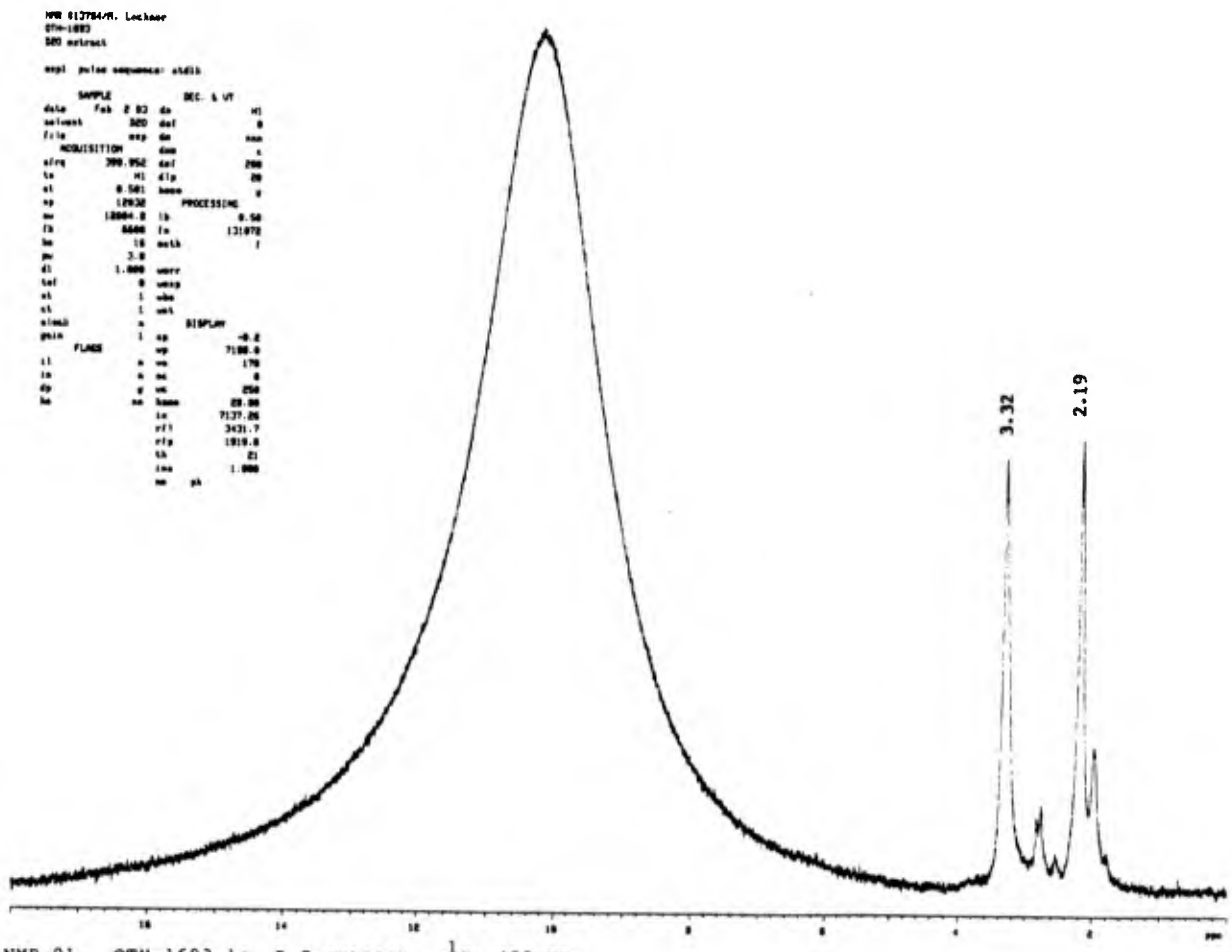
NMR-88. OTH-1593-c, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



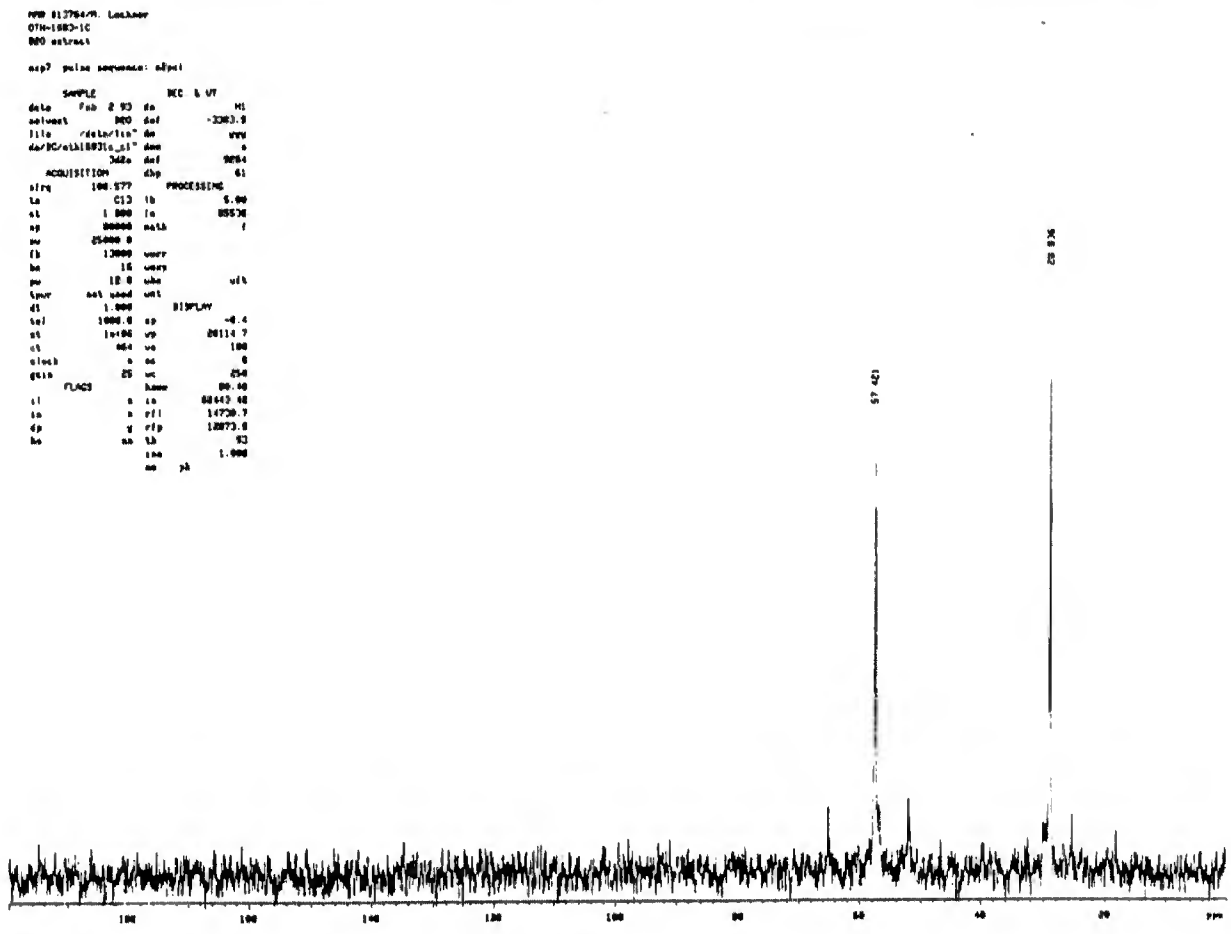
NMR-89. OTH-1593-c, $CDCl_3$ extract: ^{13}C, 100 MHz.



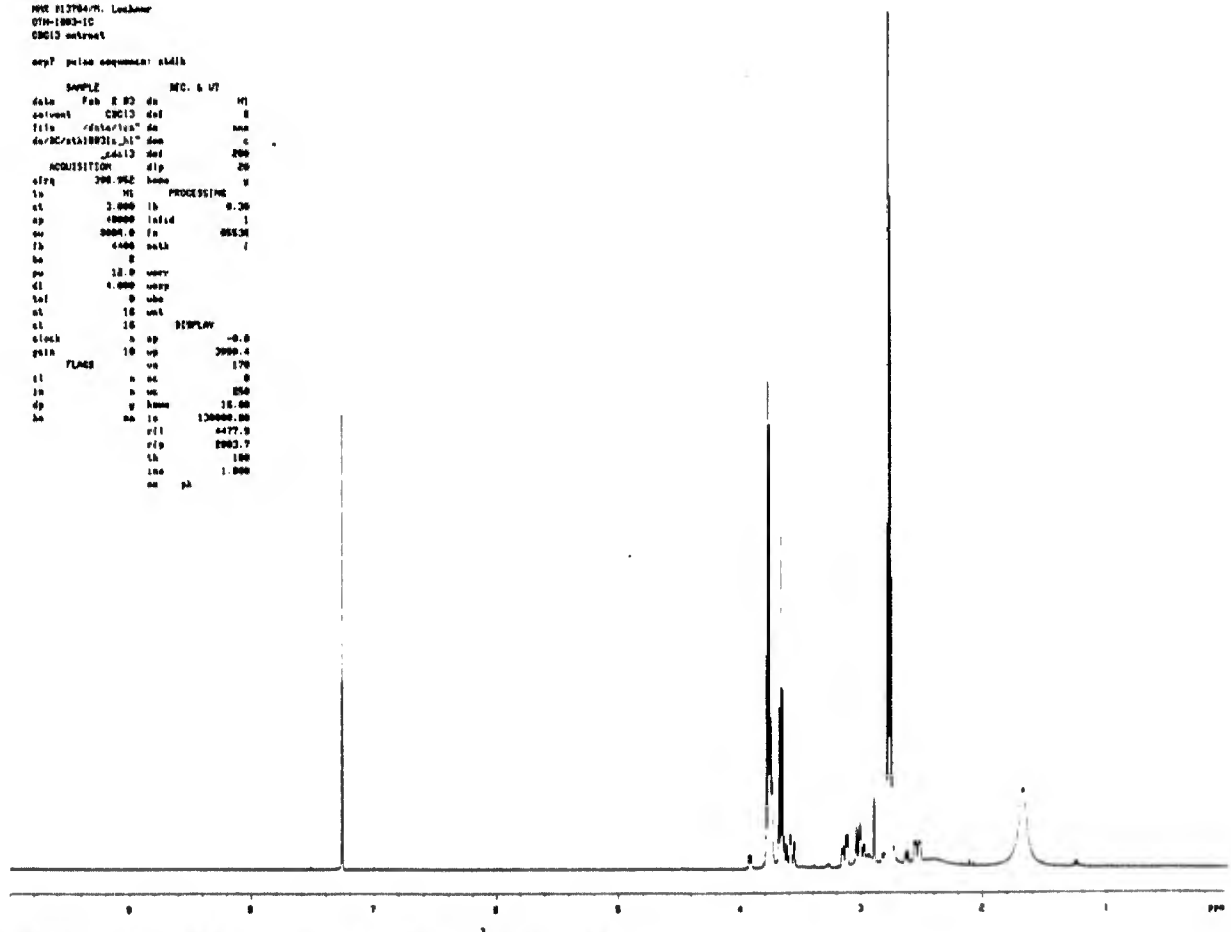
NMR-90. OTH-1693-1c, Neat: ^{13}C, 100 MHz.



NMR-91. OTH-1693-1c, D_2O extract: ^{1}H, 400 MHz.



NMR-92. OTH-1693-1c, D_2O extract: ^{13}C, 100 MHz.

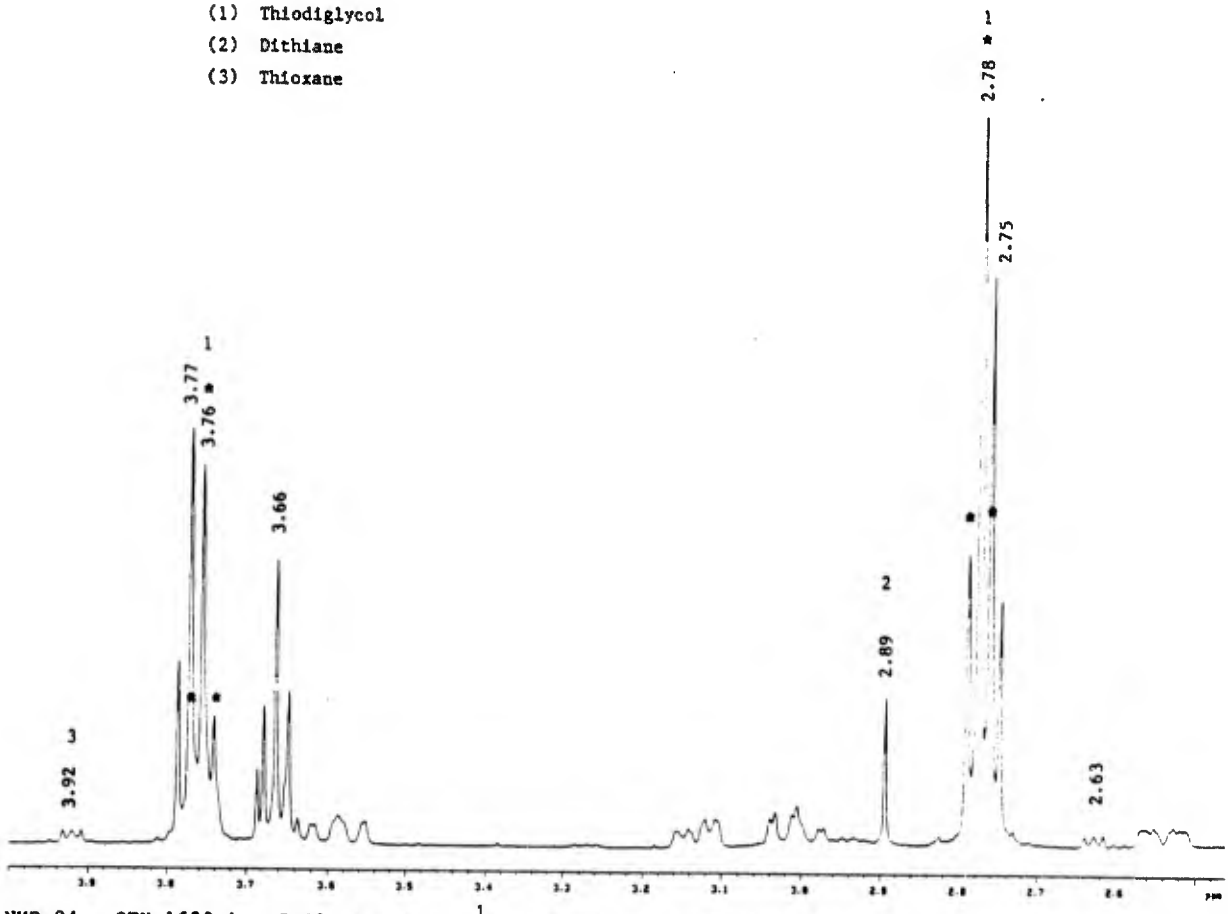


NMR-93. OTH-1693-1c, $CDCl_3$ extract: ^{1}H, 400 MHz.

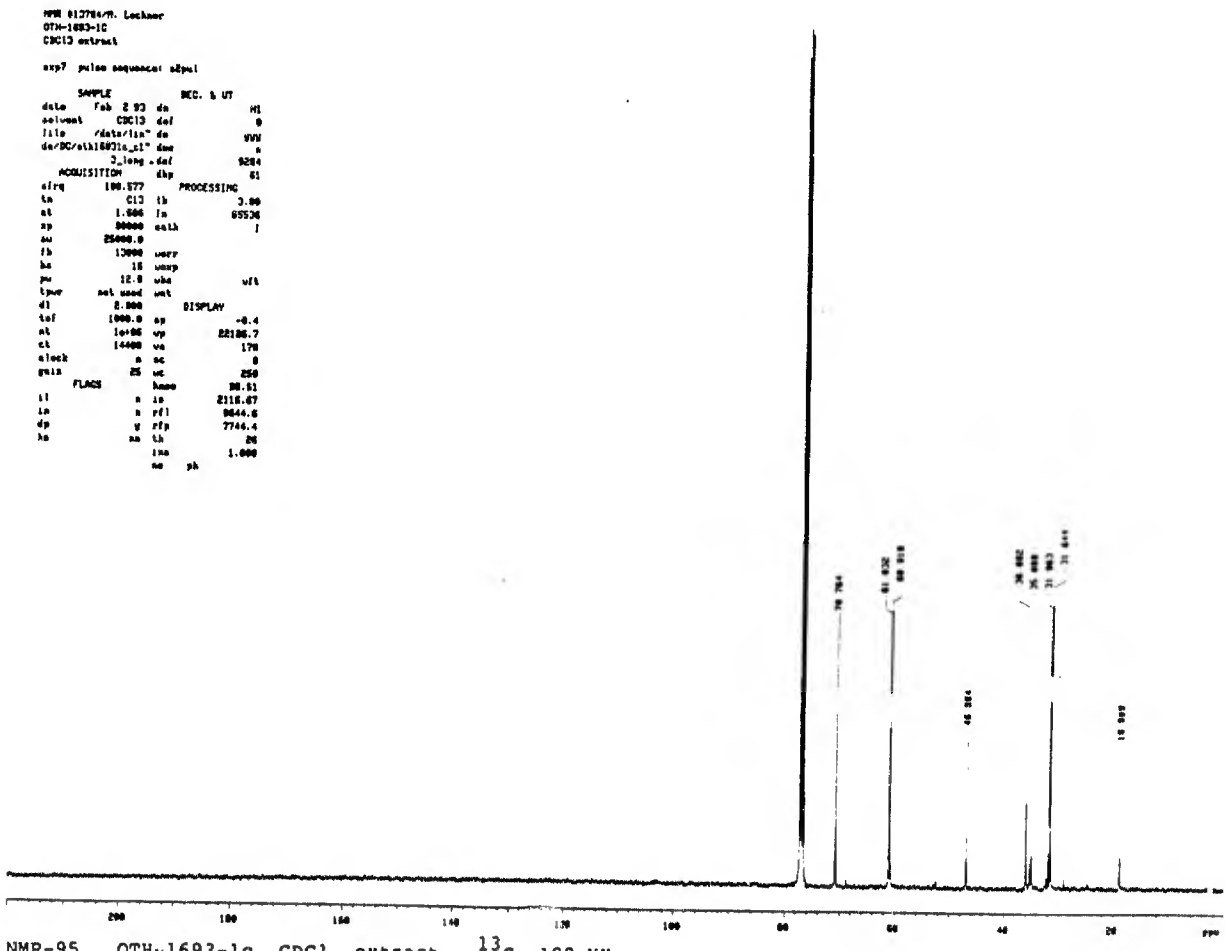
(1) Thiodiglycol

(2) Dithiane

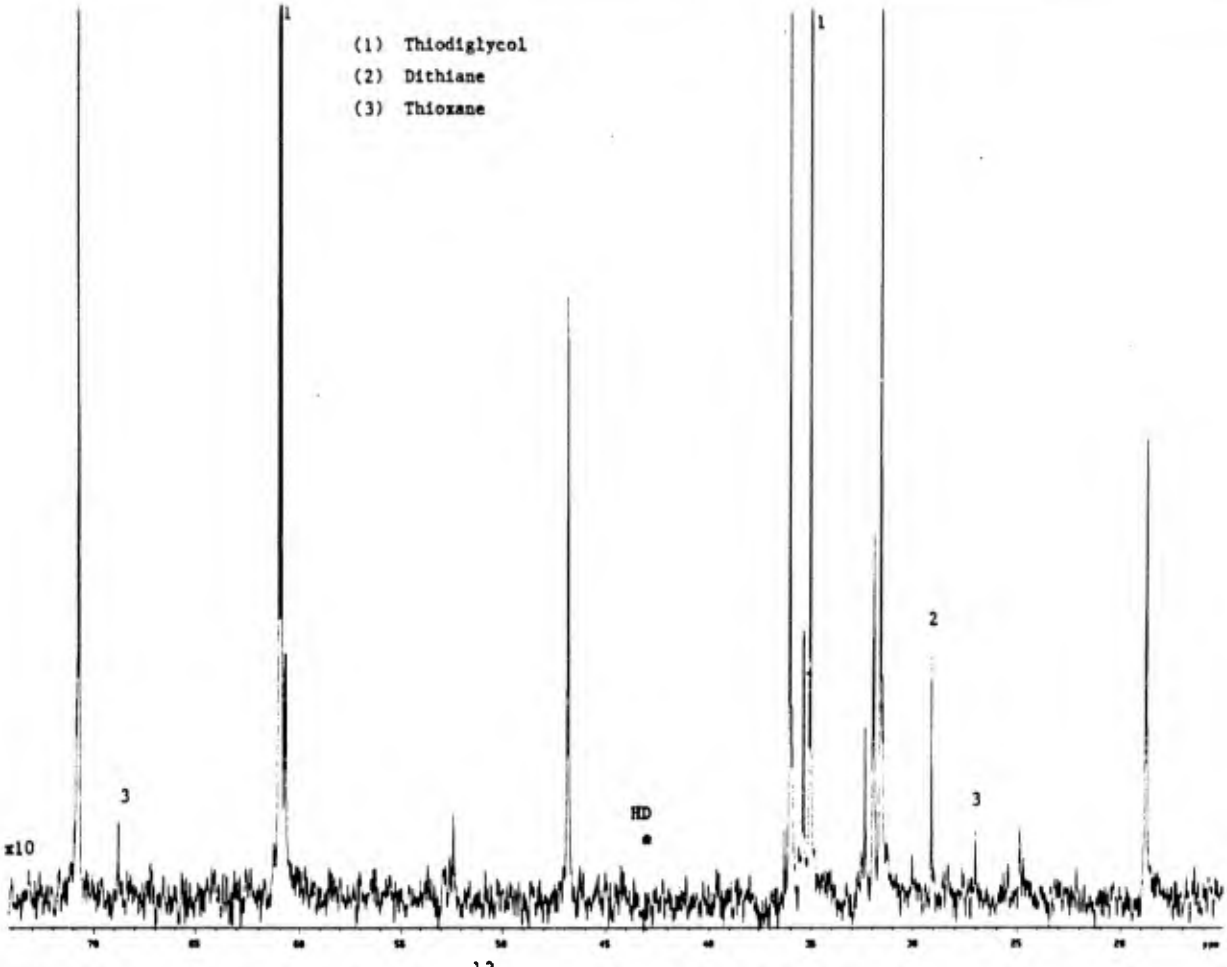
(3) Thioxane



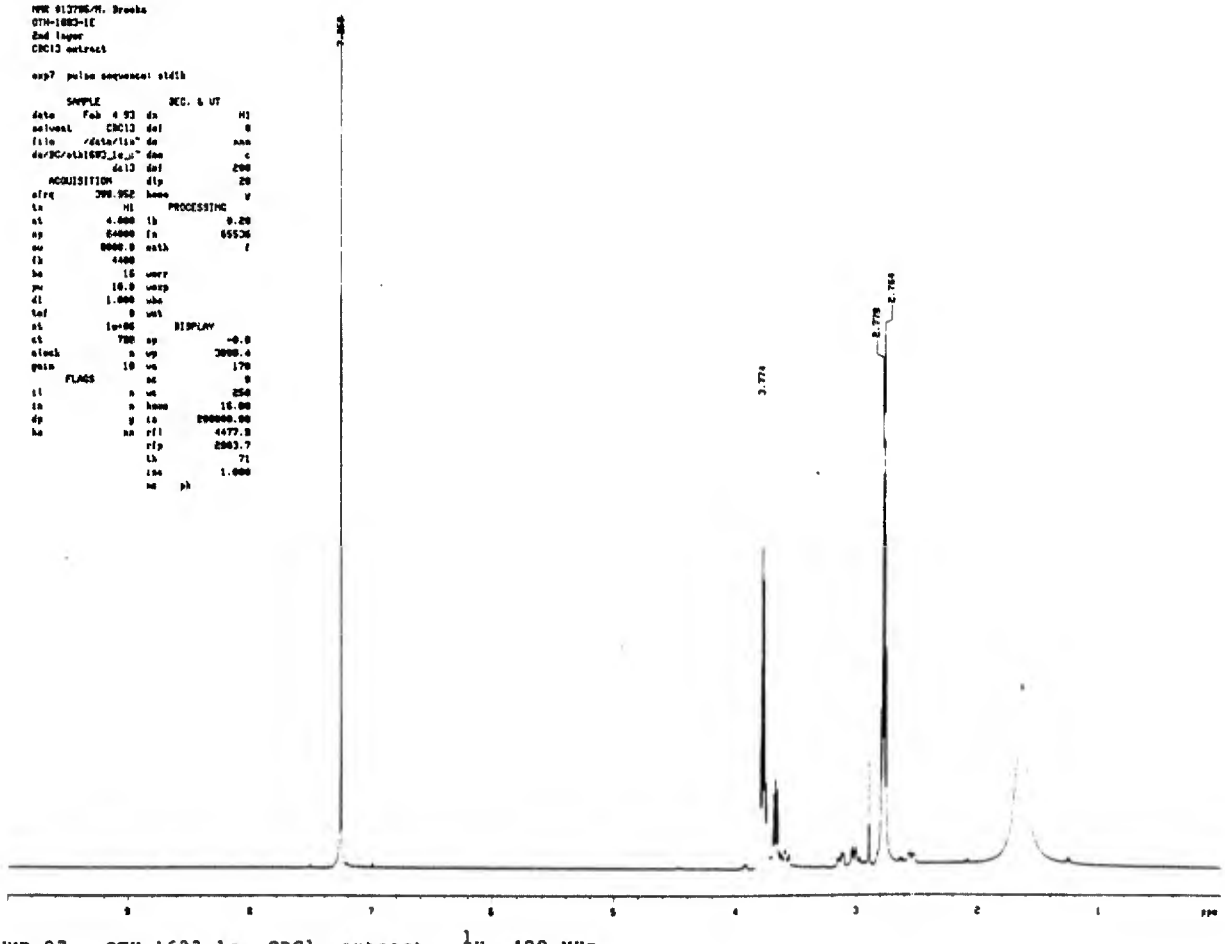
NMR-94. OTH-1693-1c, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



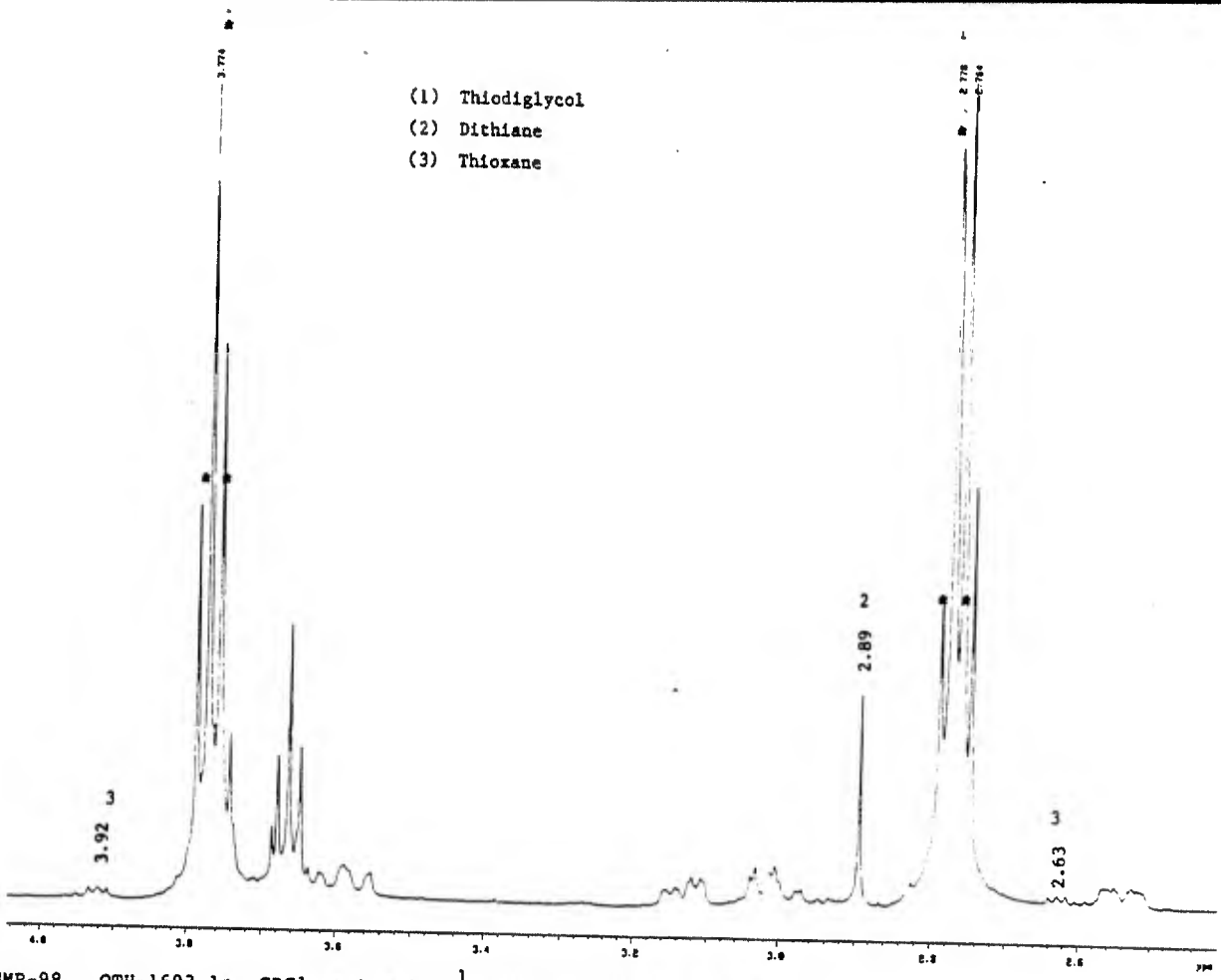
NMR-95. OTH-1693-1c, $CDCl_3$ extract: ^{13}C, 100 MHz.



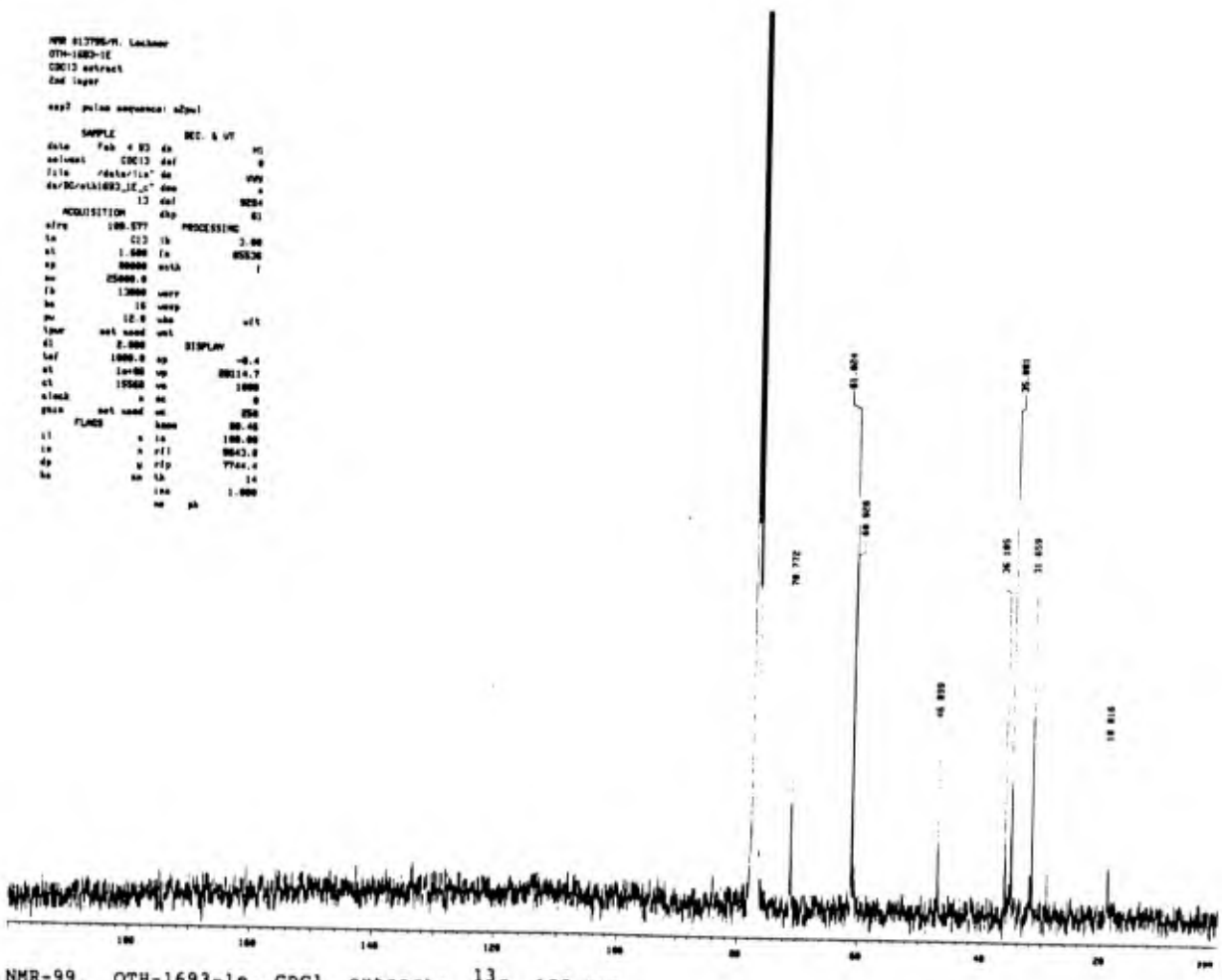
NMR-96. OTH-1693-1c, $CDCl_3$ extract: ^{13}C, 100 MHz, expanded spectrum.



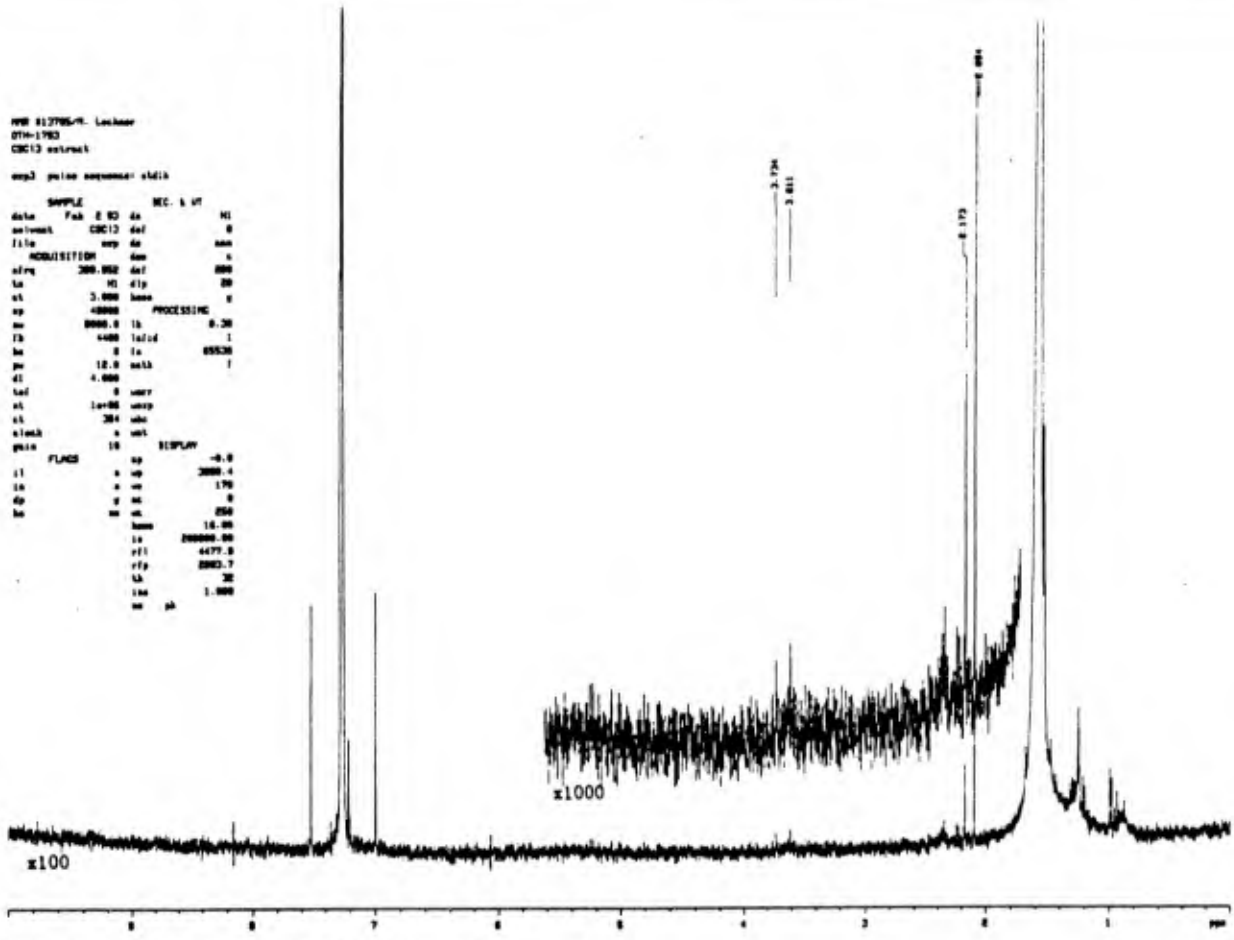
NMR-97. OTH-1693-1e, $CDCl_3$ extract: ^{1}H, 400 MHz.



NMR-98. OTH-1693-1e, $CDCl_3$ extract: ^1H, 400 MHz, expanded spectrum.



NMR-99. OTH-1693-1e, $CDCl_3$ extract: ^{13}C, 100 MHz.



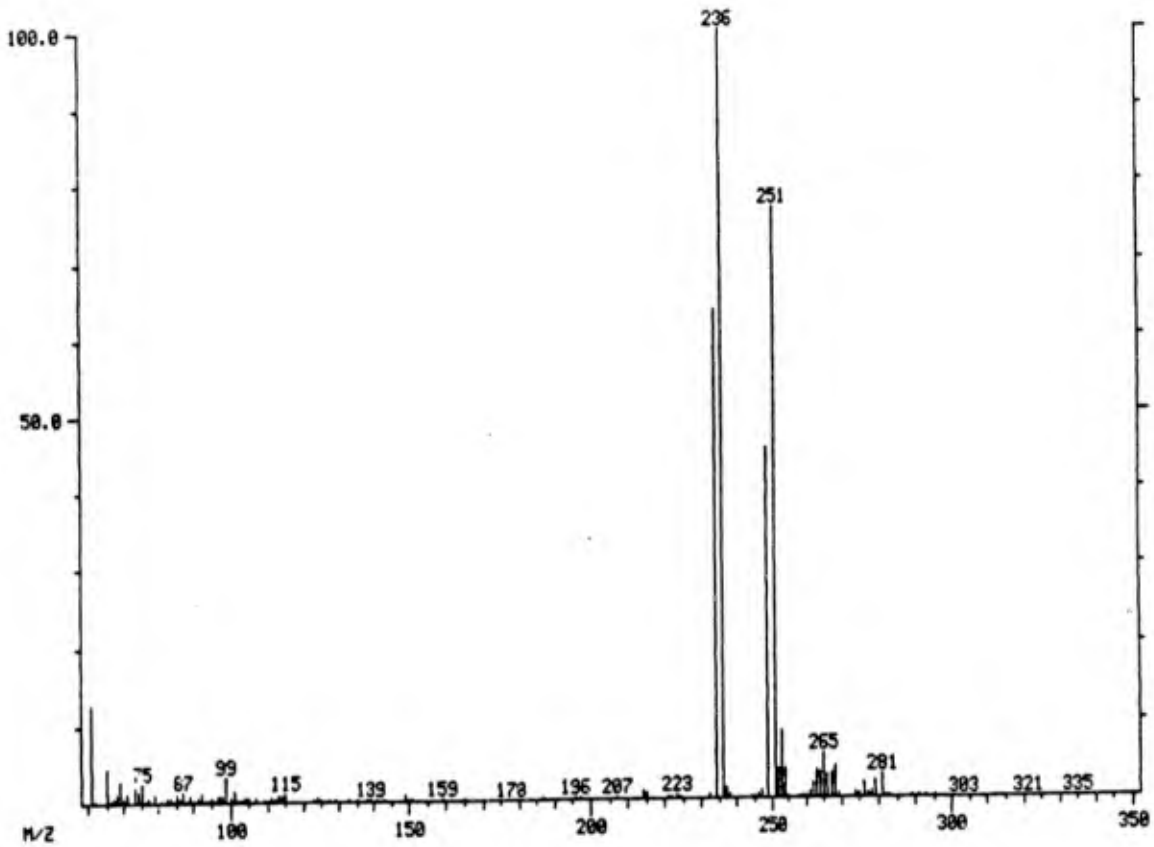
NMR-100. OTH-1793, $CDCl_3$ extract: ^1H, 400 MHz.

Blank

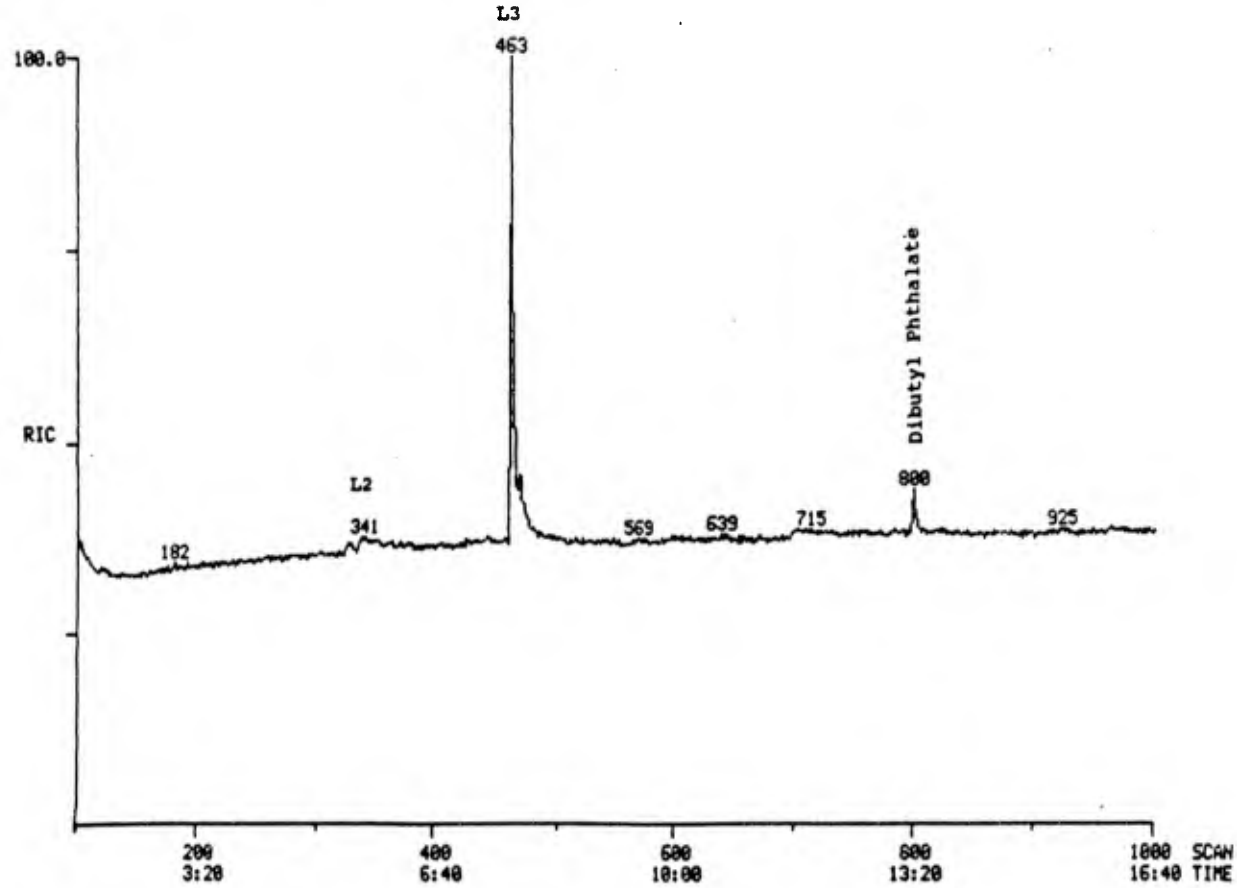
APPENDIX D

GC/MS/CI AND DEP/CI SPECTRA

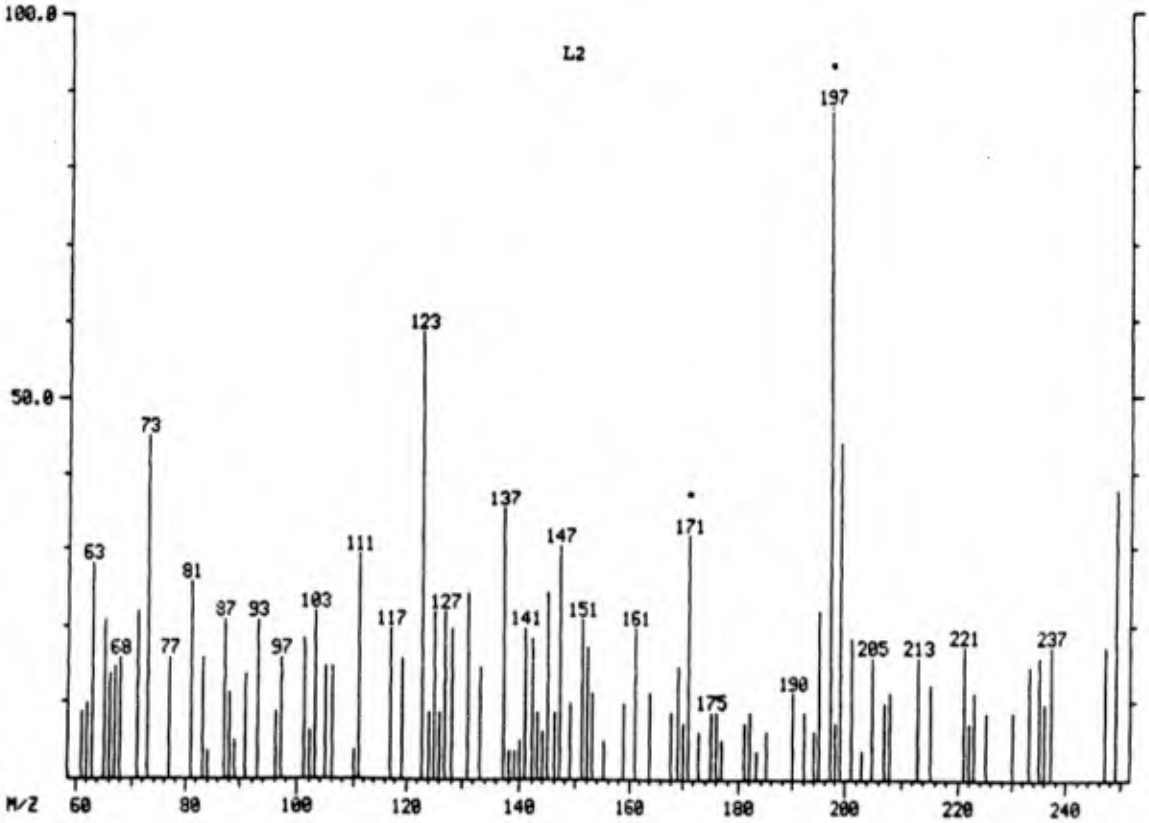
Blank DEP/CI Mass Spectrum



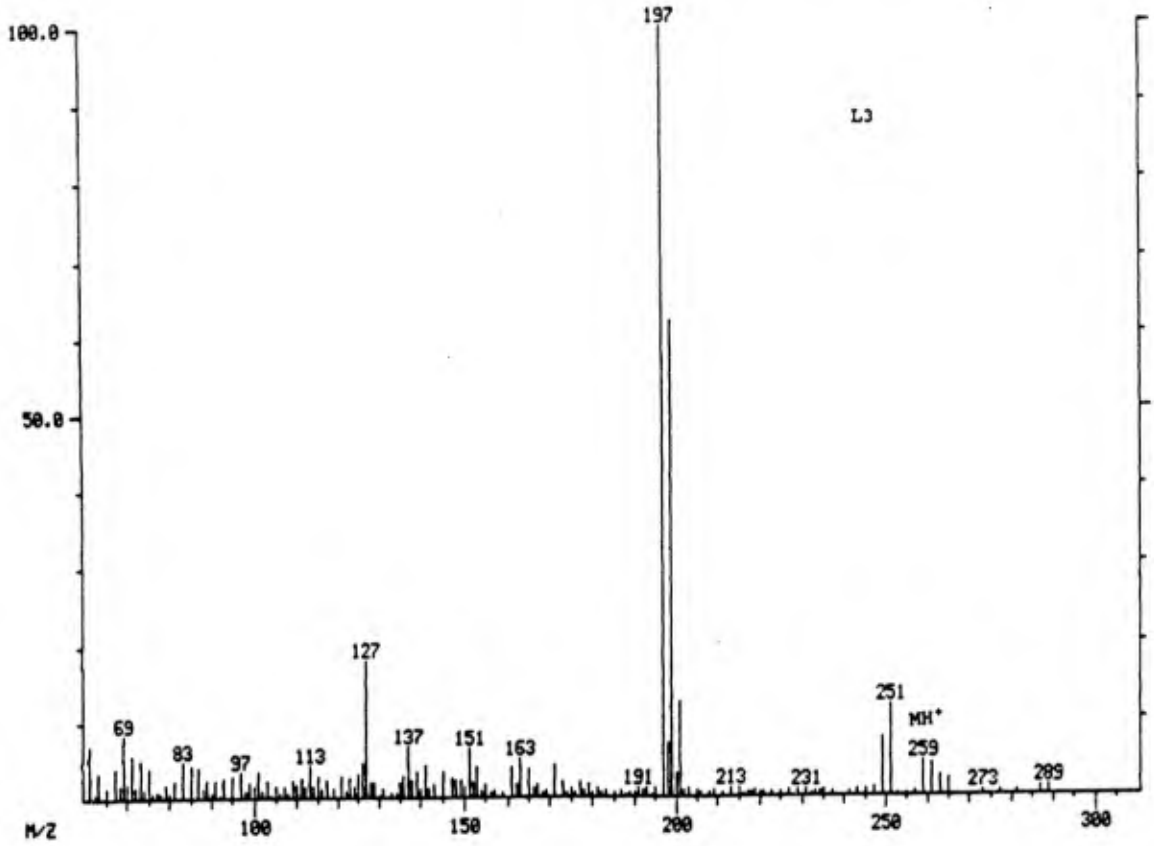
GC/MS/CI Chromatogram of OTH-193-1b (Pig #1)



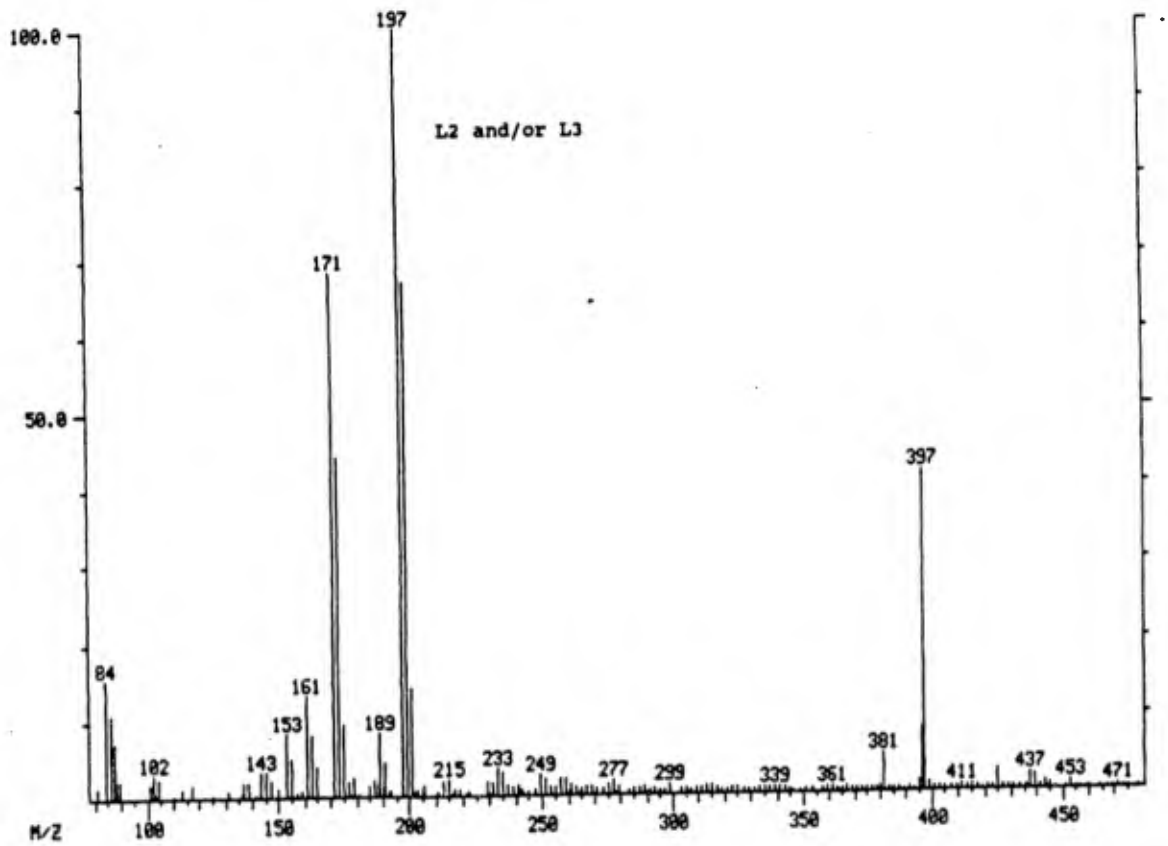
CI Mass Spectrum of OTH-193-1b Scan 341



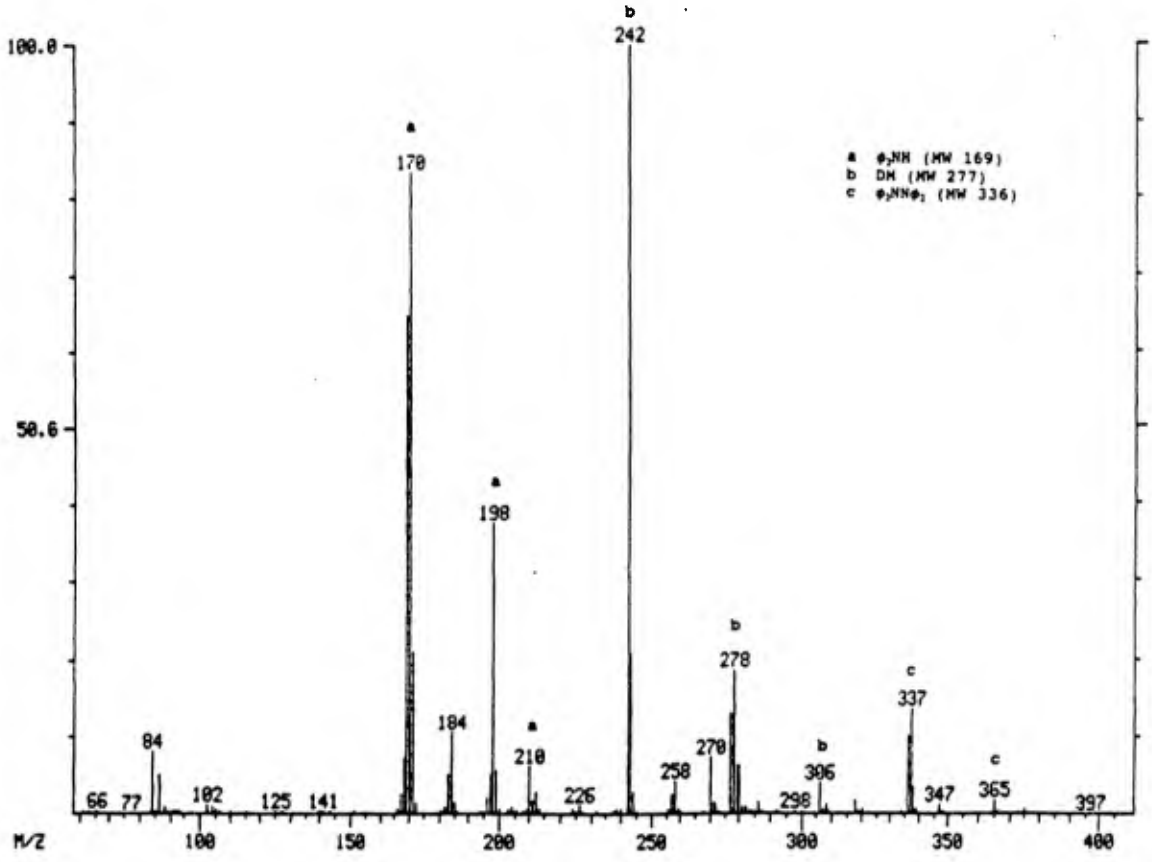
CI Mass Spectrum of OTH-193-1b Scan 463



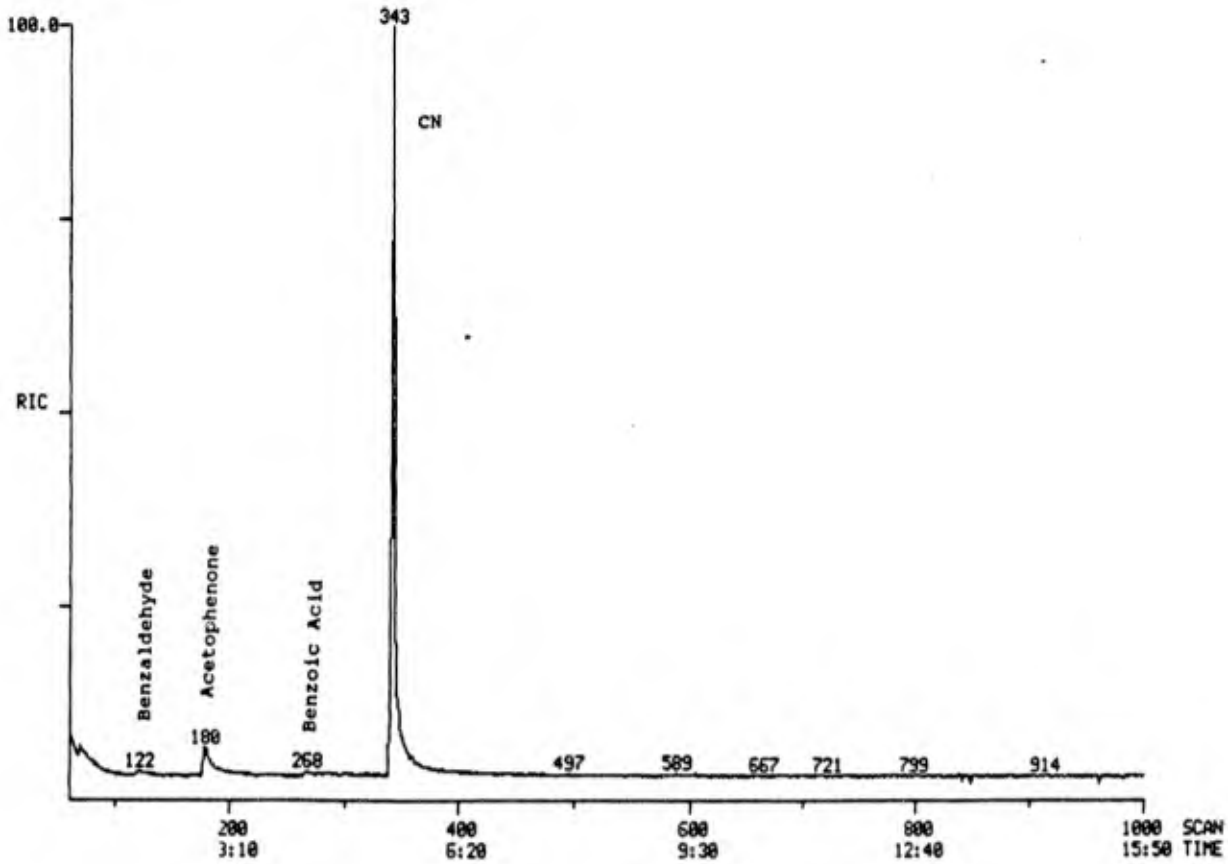
DEP/CI Mass Spectrum of OTH-193-1b (Pig #1)



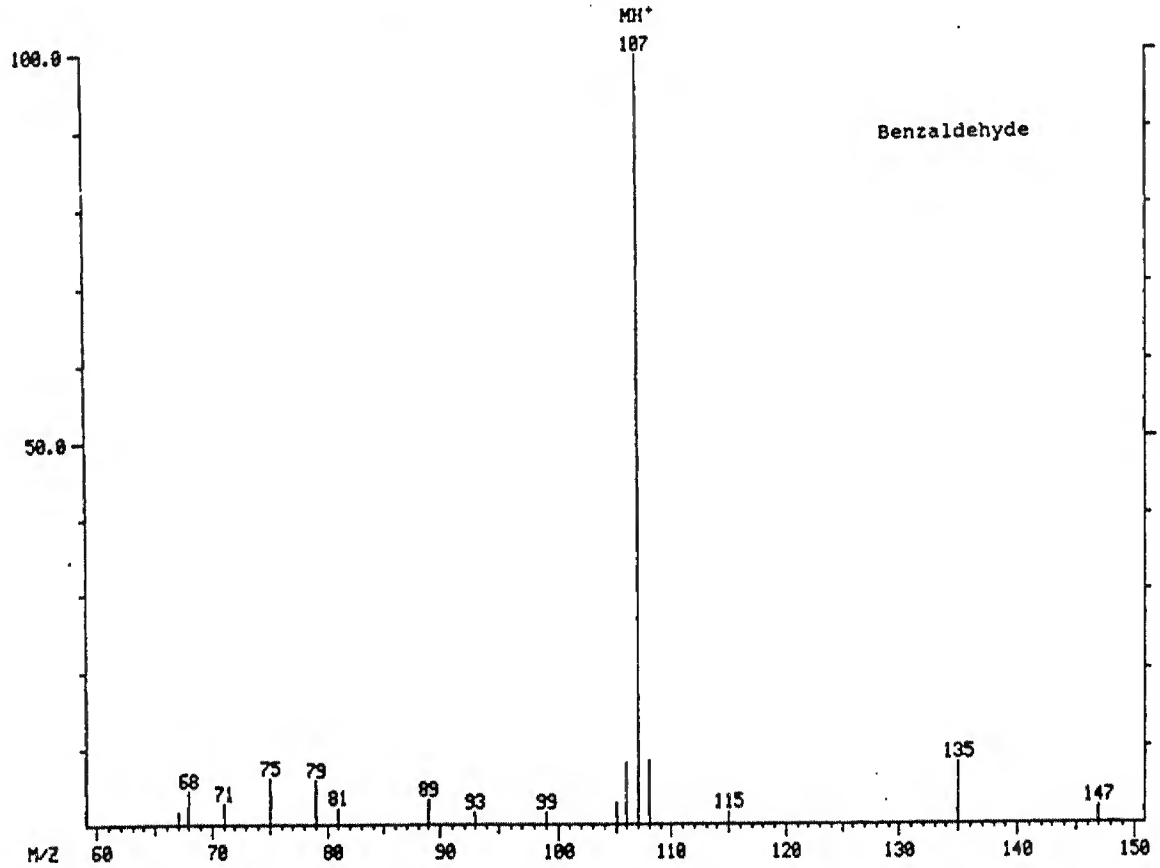
DEP/CI Mass Spectrum of OTH-193-2a (Pig #2)



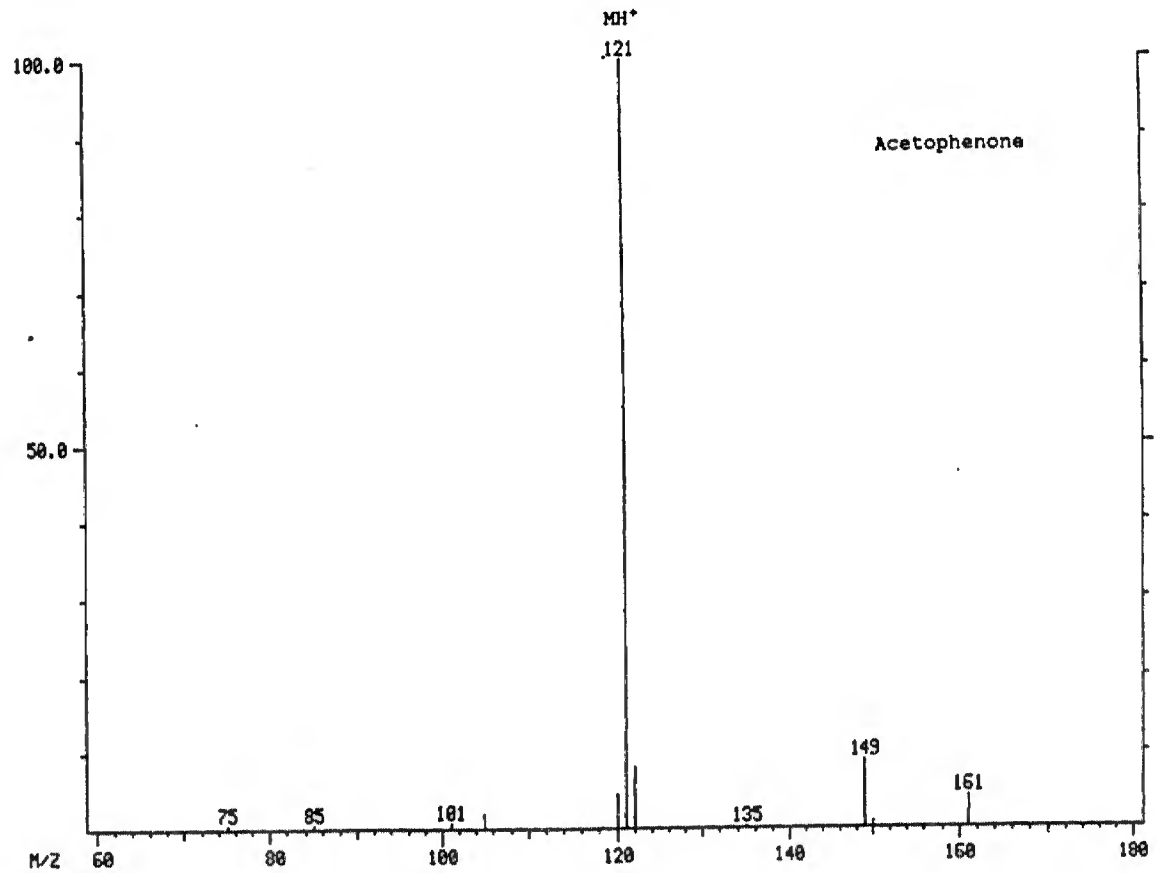
GC/MS/CI Chromatogram of OTH-193-3a (Pig #3)



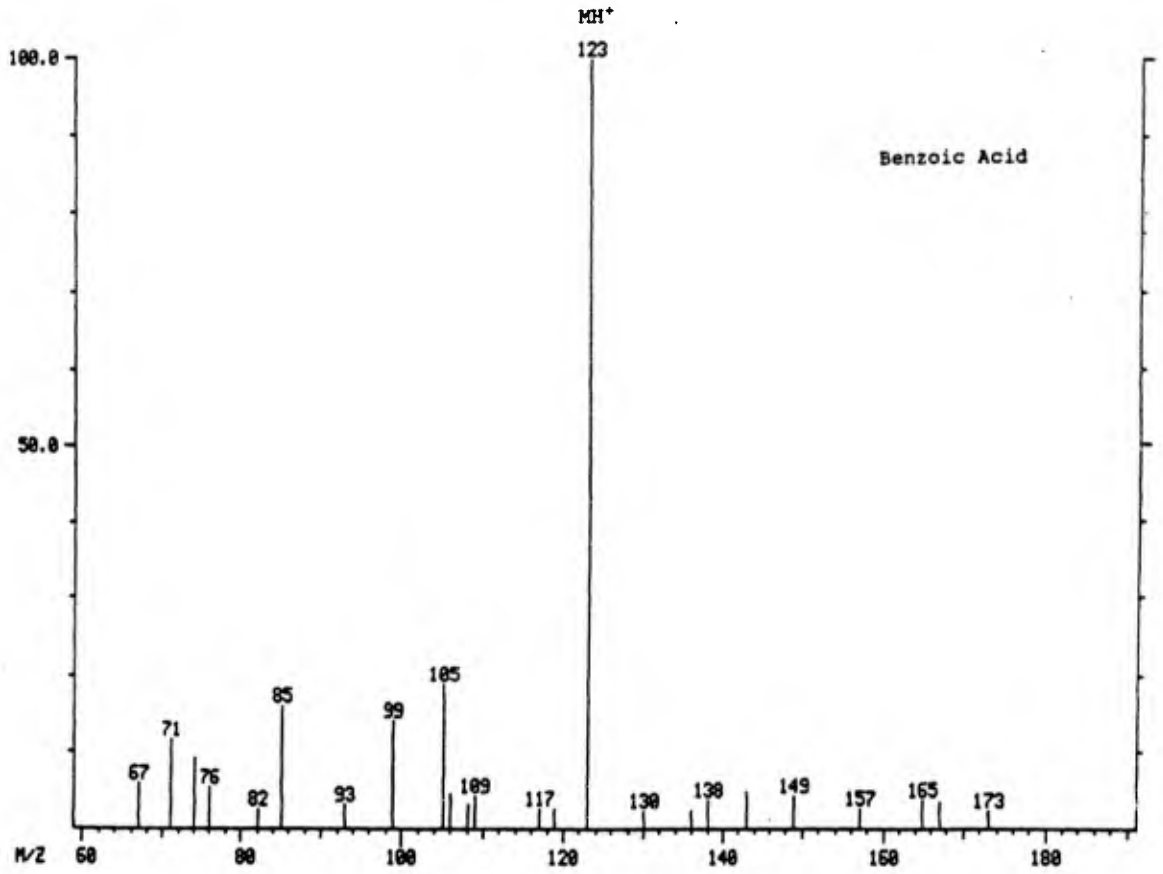
CI Mass Spectrum of OTH-193-3a Scan 122



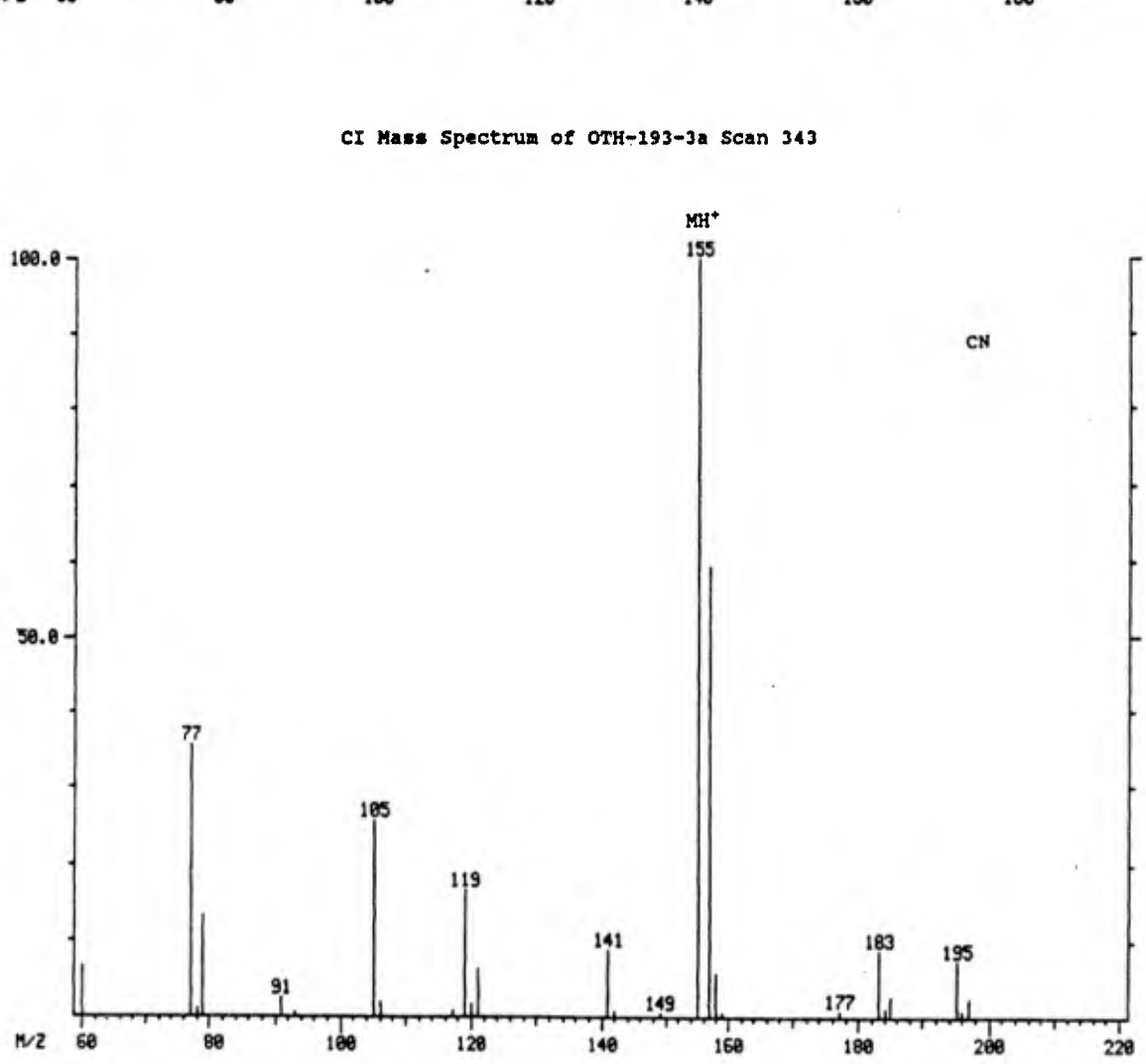
CI Mass Spectrum of OTH-193-3a Scan 179



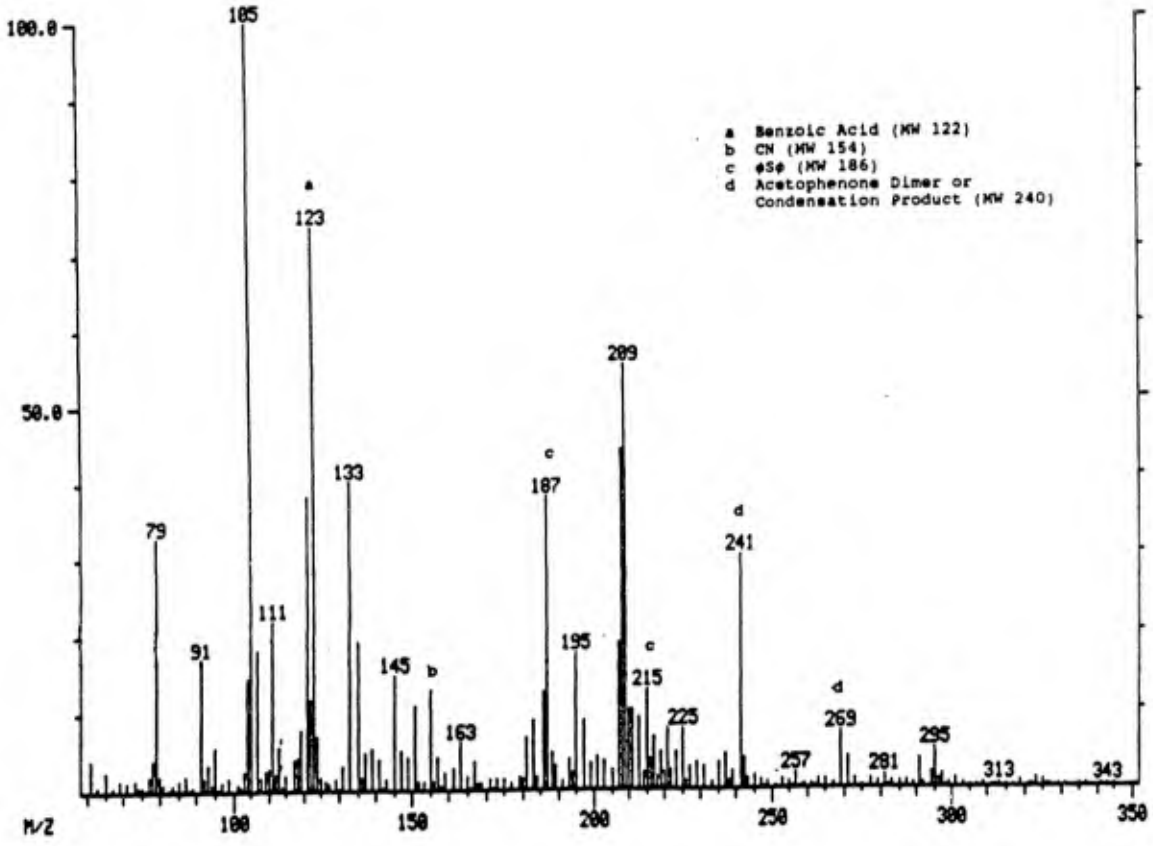
CI Mass Spectrum of OTH-193-3a Scan 268



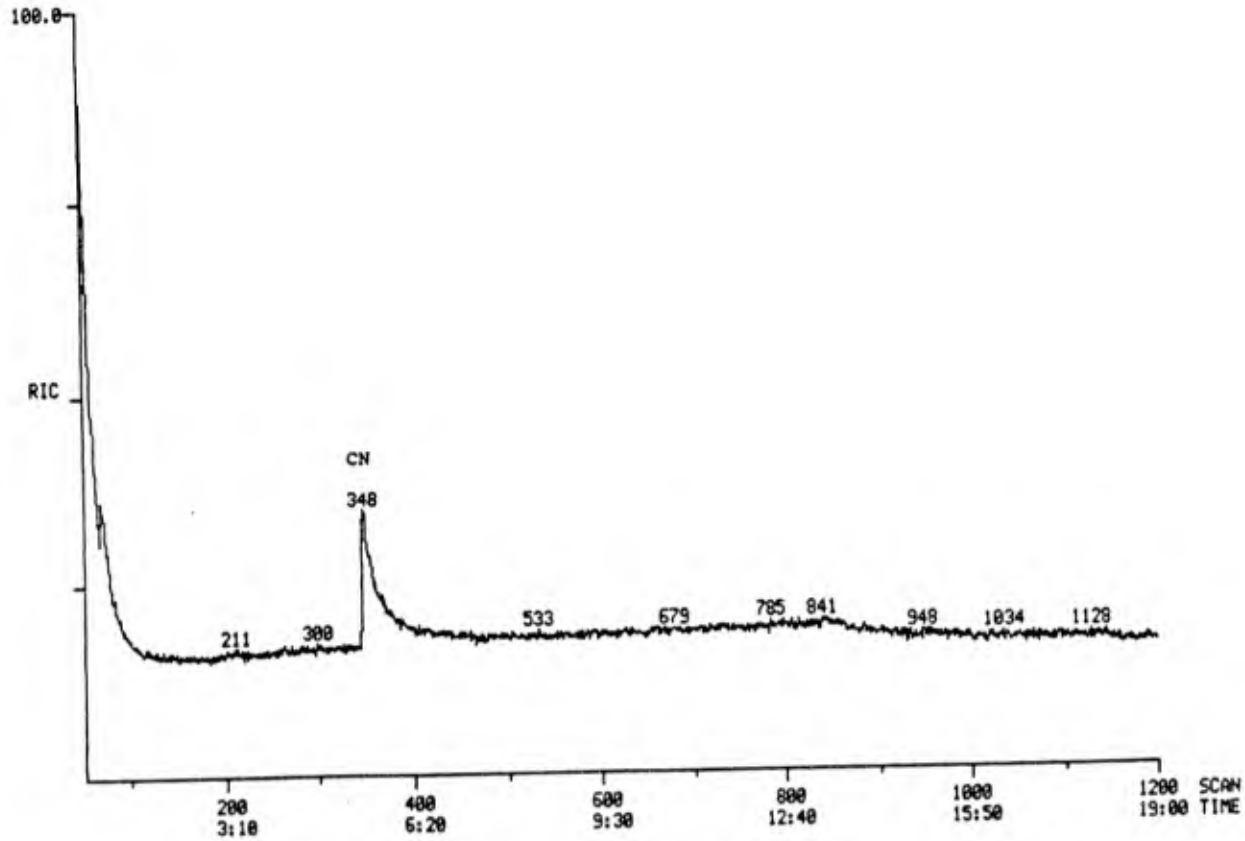
CI Mass Spectrum of OTH-193-3a Scan 343



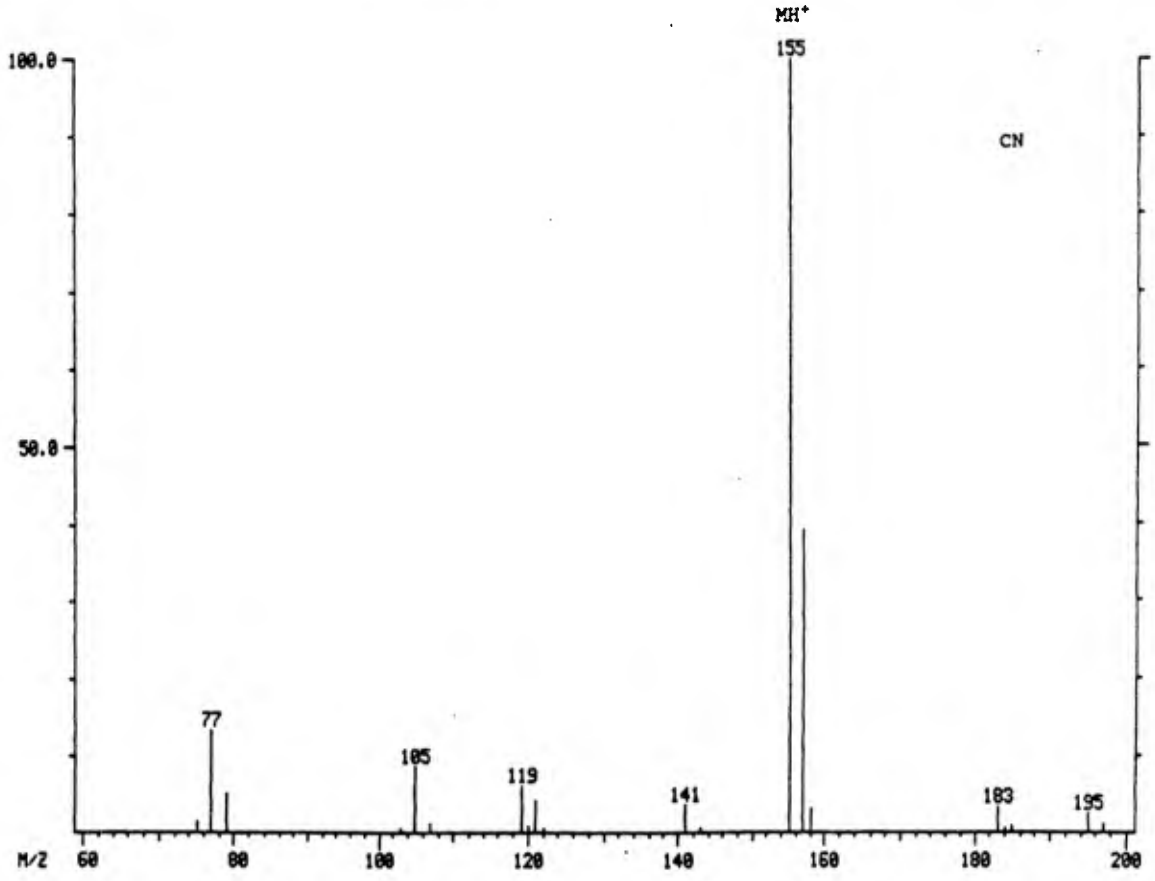
DEP/CI Mass Spectrum of OTH-193-3a (Pig #3)



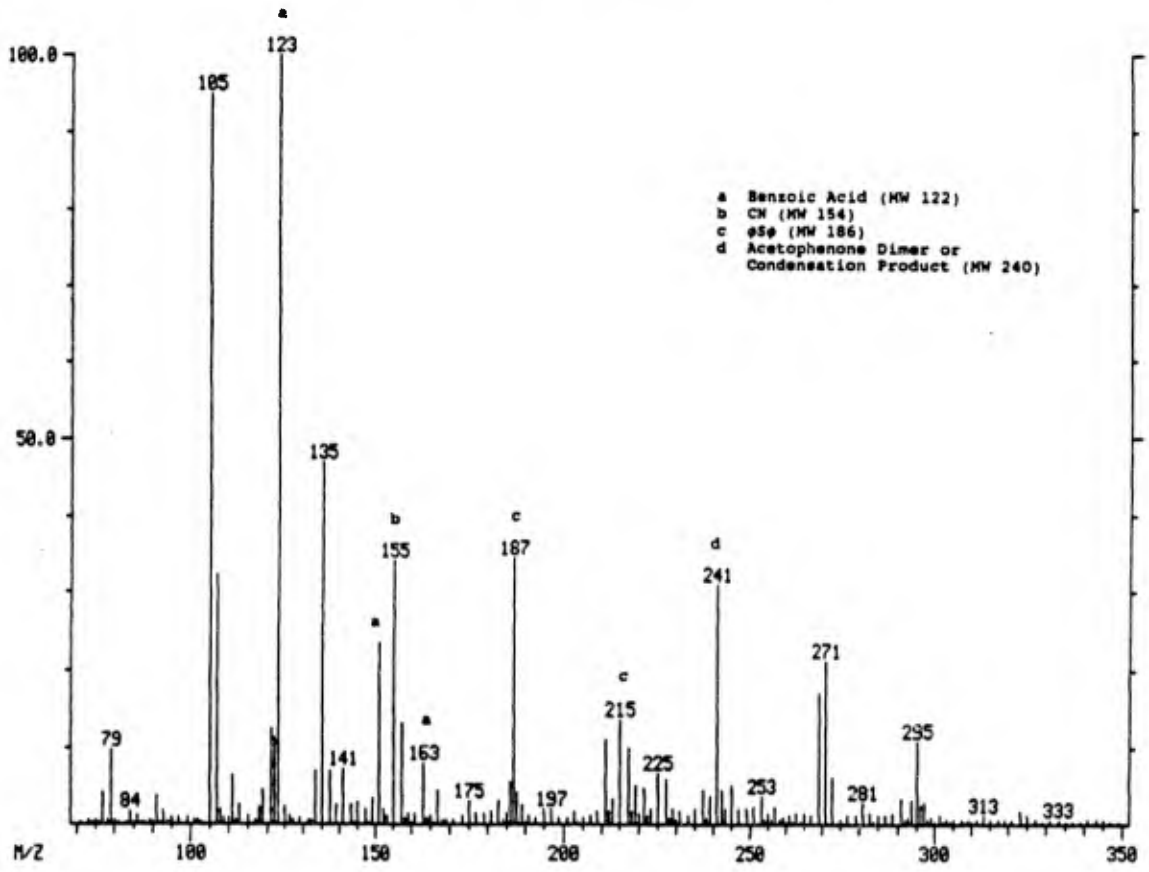
GC/MS/CI Chromatogram of OTH-193-3b (Pig #3)



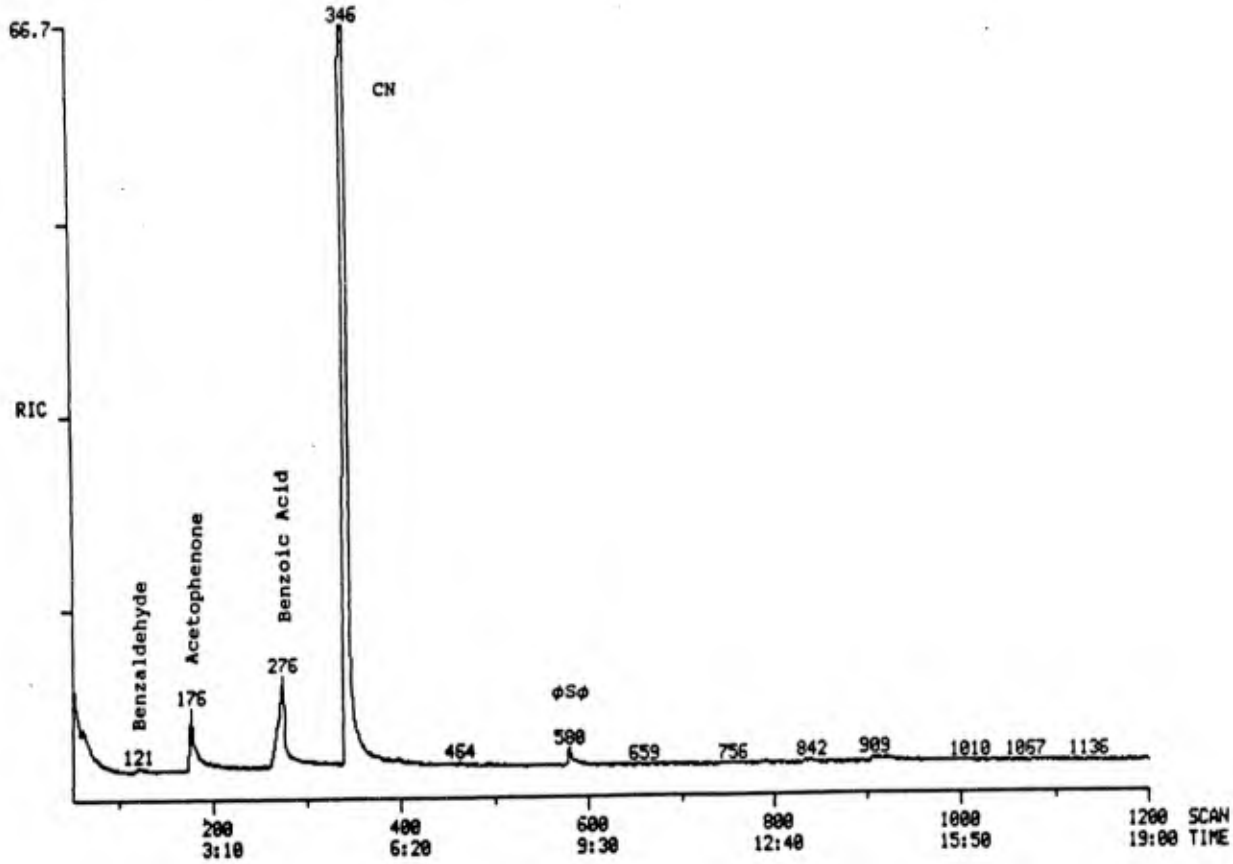
CI Mass Spectrum of OTH-193-3b Scan 348



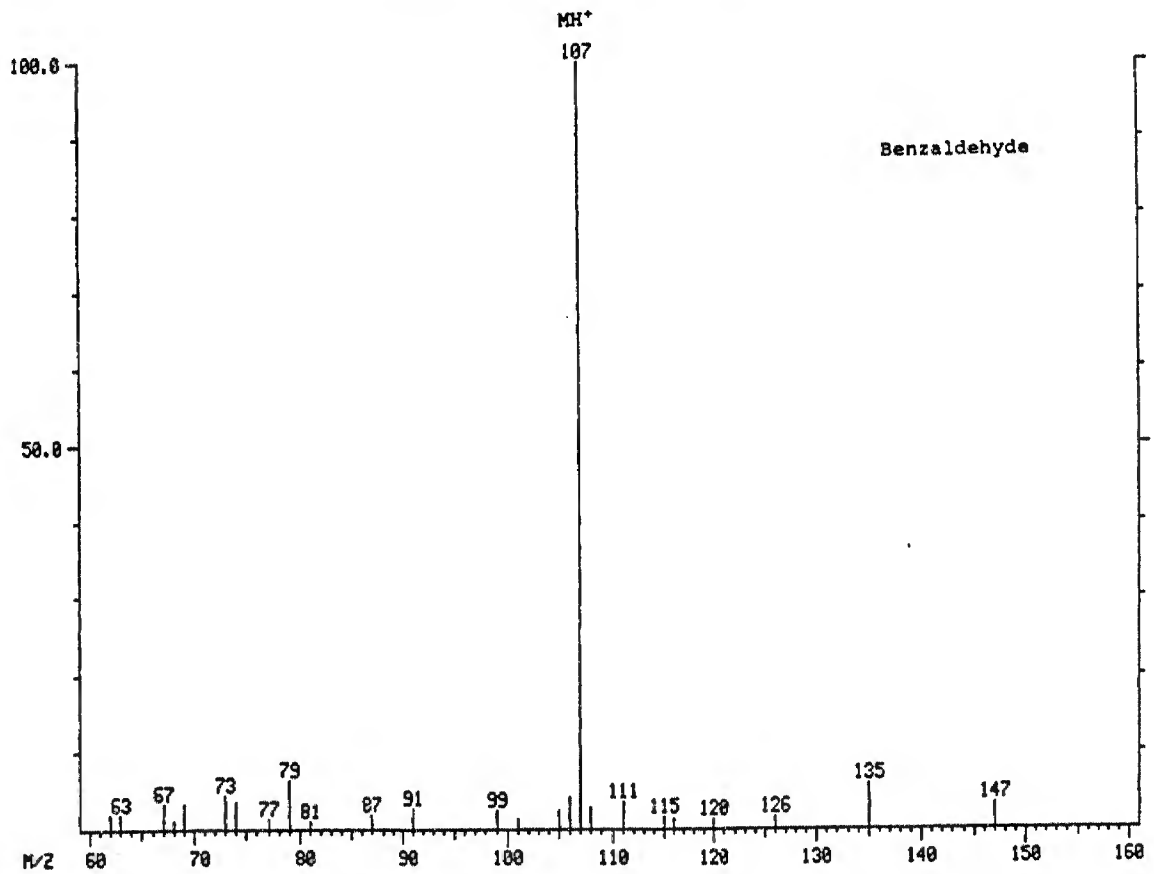
DEP/CI Mass Spectrum of OTH-193-3c (Pig #3)



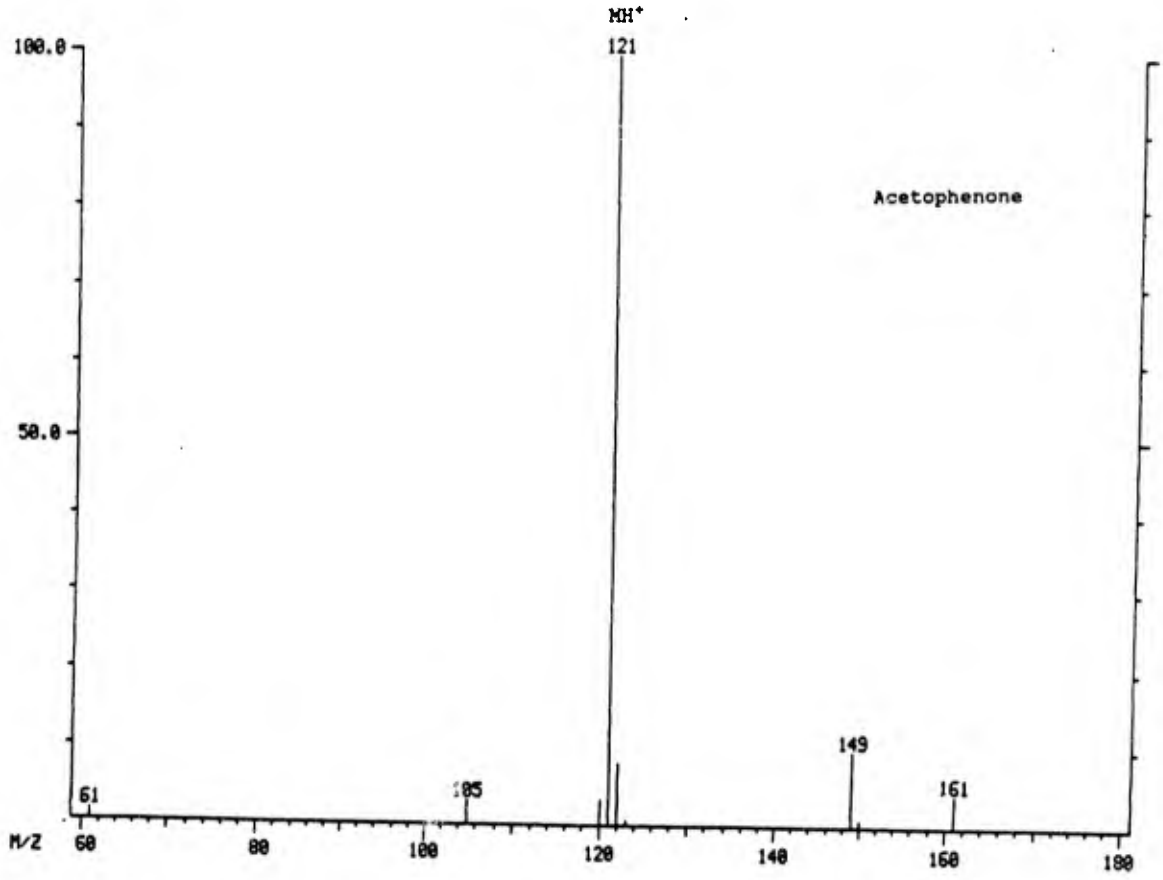
GC/MS/CI Chromatogram of OTH-193-3c (Pig #3)



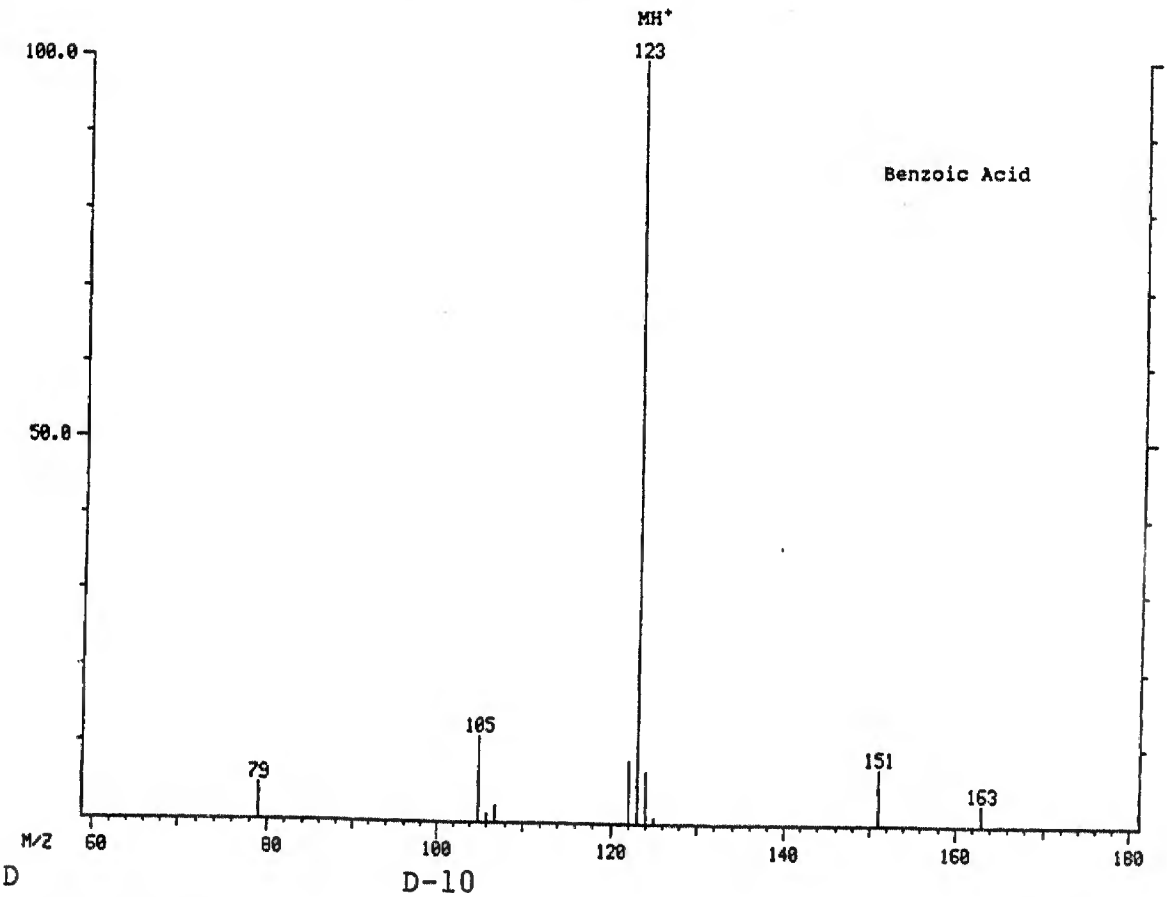
CI Mass Spectrum of OTH-193-3c Scan 121



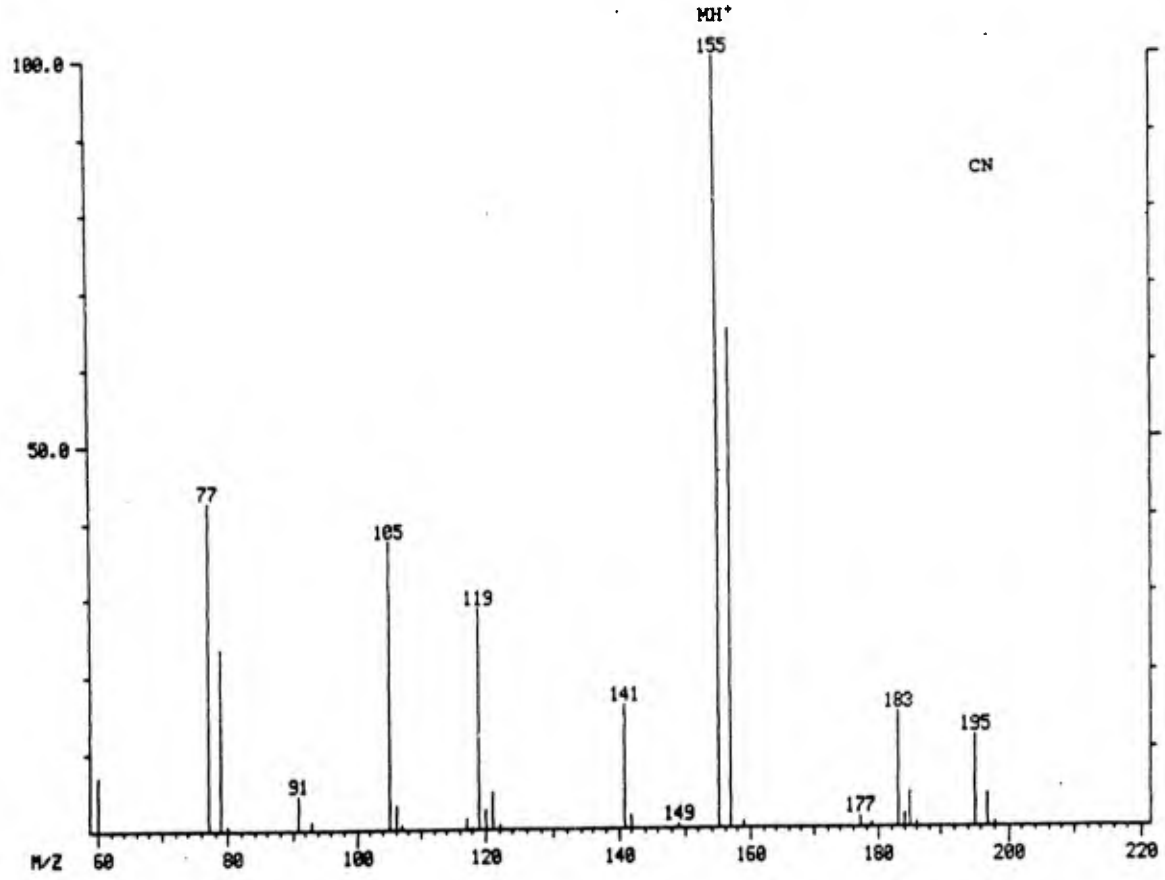
CI Mass Spectrum of OTH-193-3c Scan 176



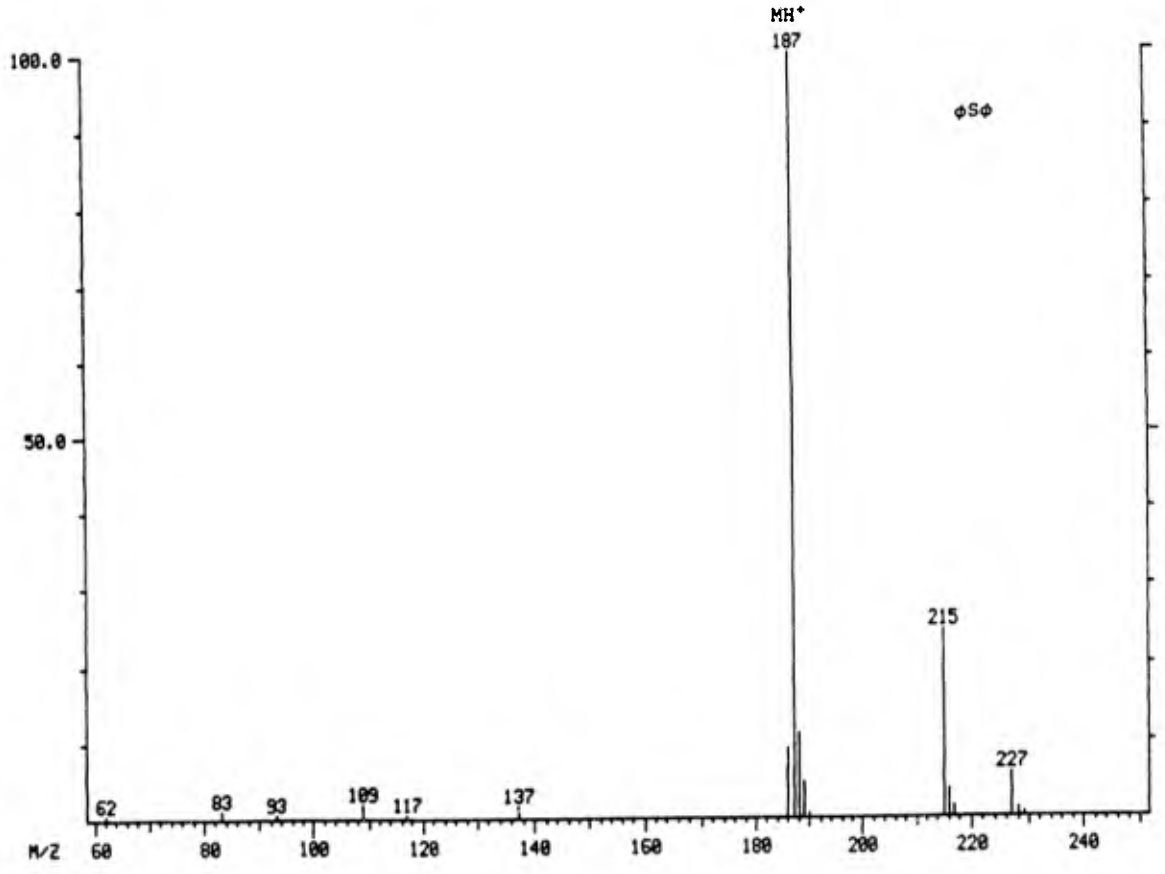
CI Mass Spectrum of OTH-193-3c Scan 276



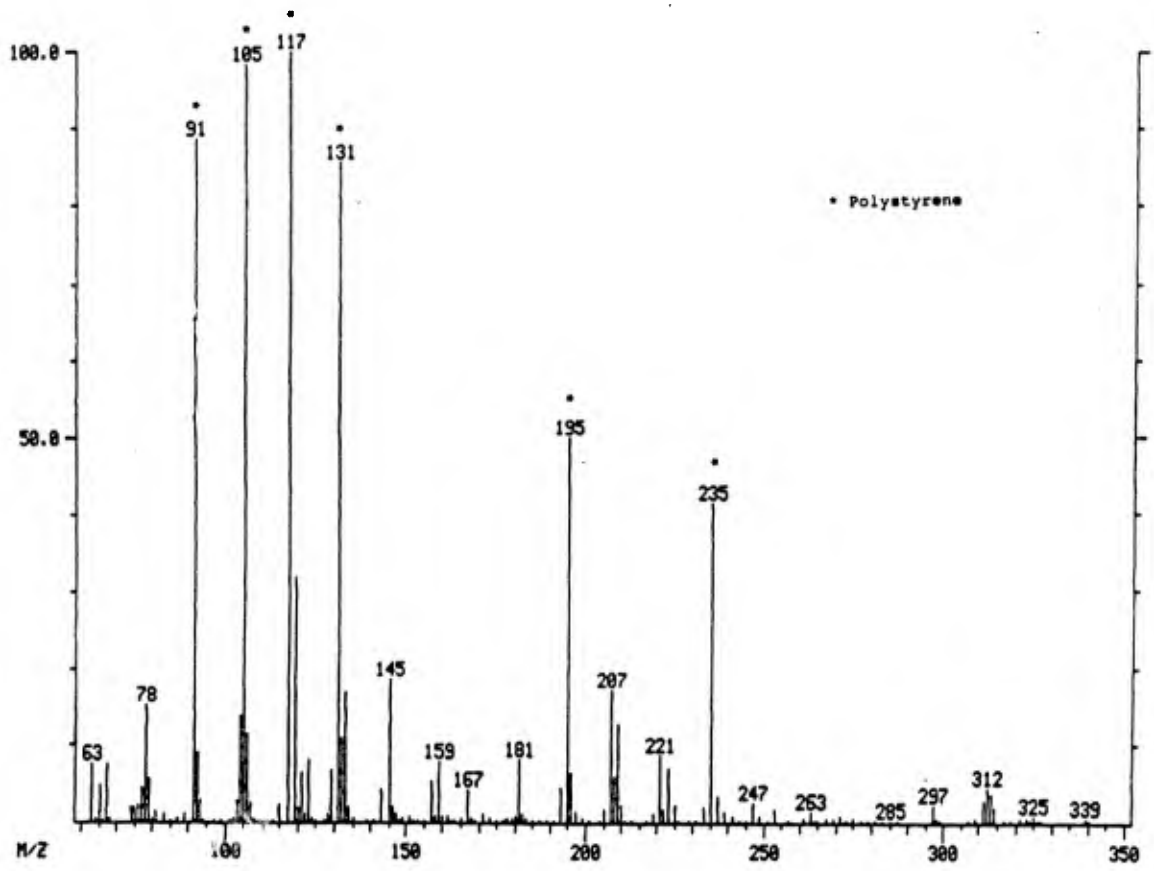
CI Mass Spectrum of OTH-193-3c Scan 346



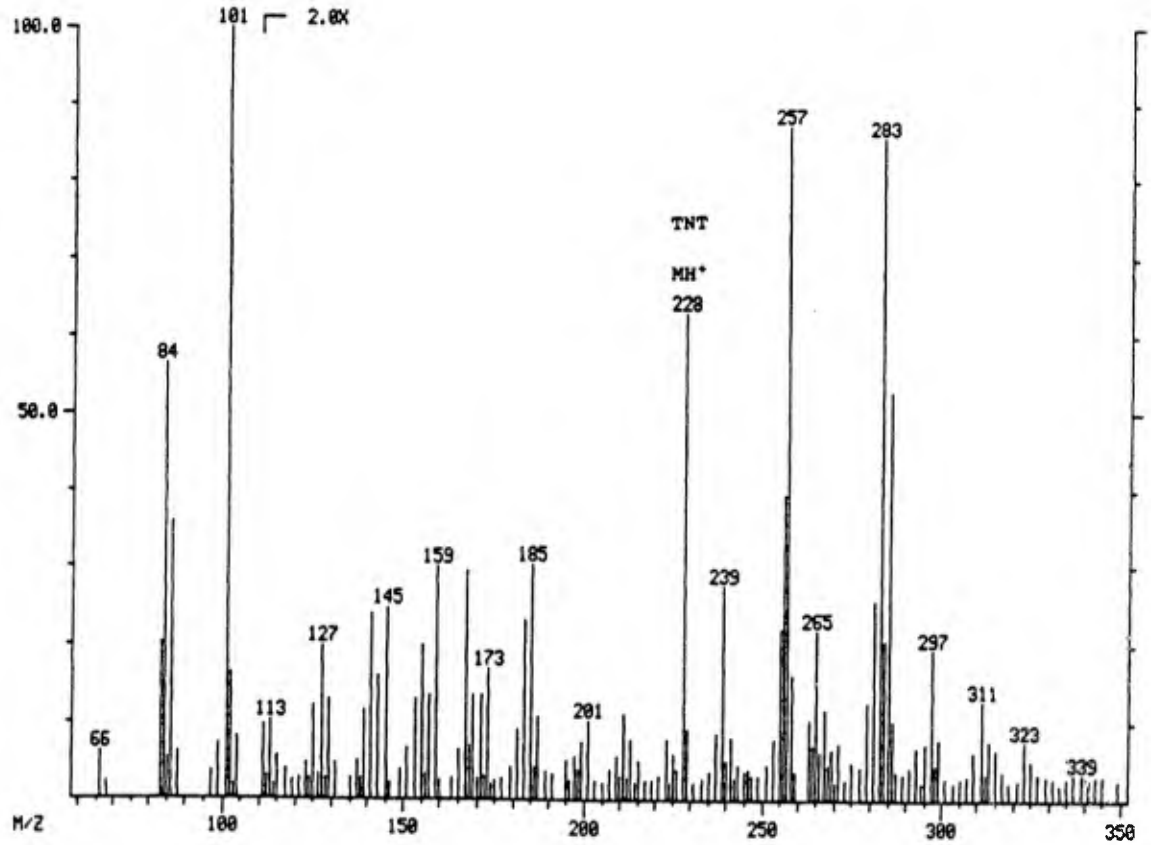
CI Mass Spectrum of OTH-193-3c Scan 580



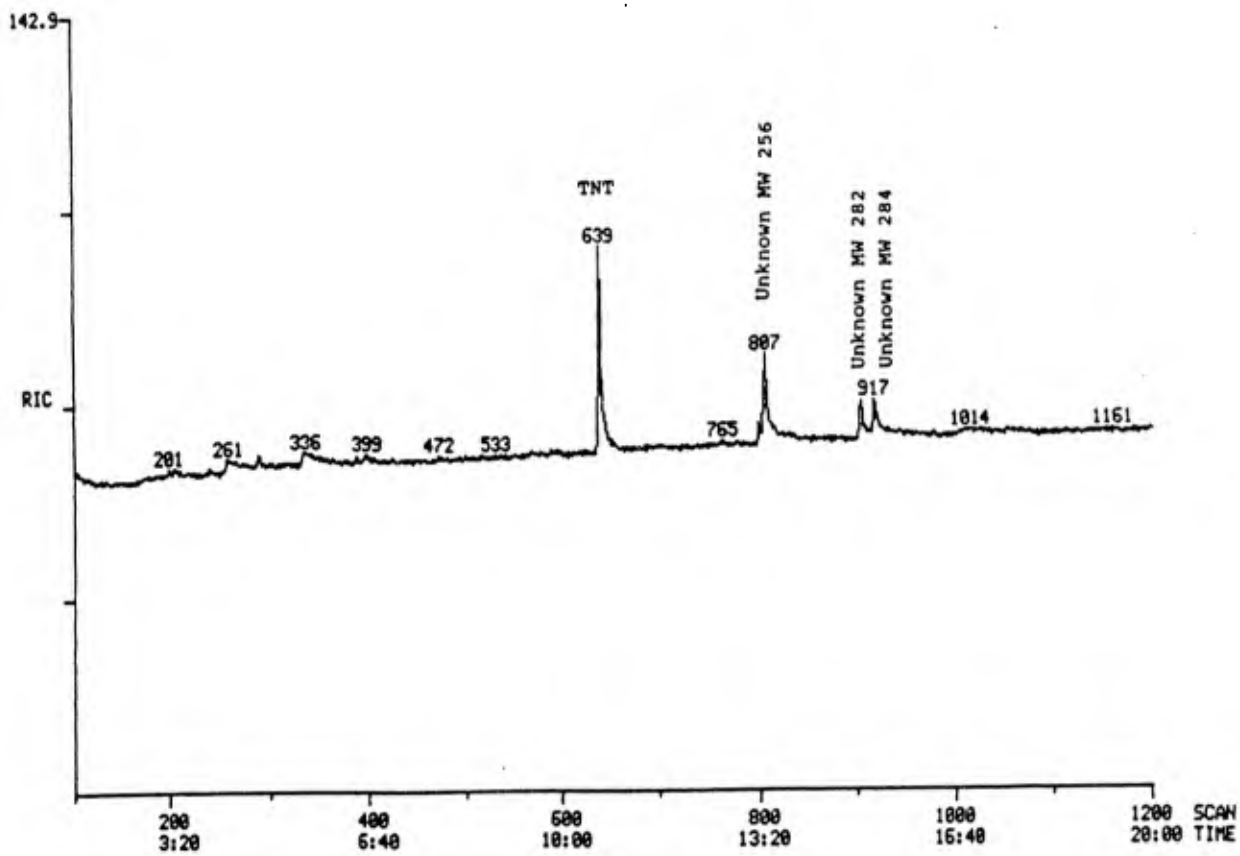
DEP/CI Mass Spectrum of OTH-193-5b (Pig #5)



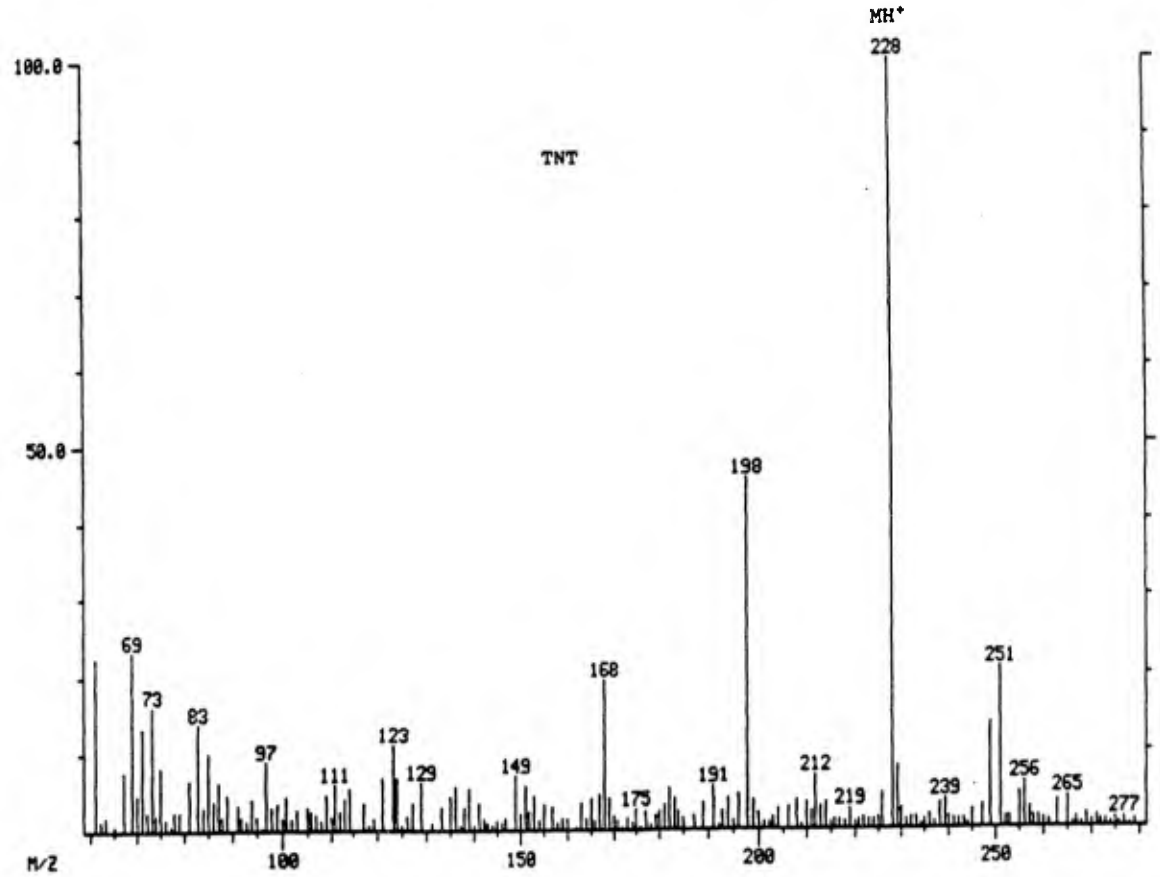
DEP/CI Mass Spectrum of OTH-393-4 (Pig #4)



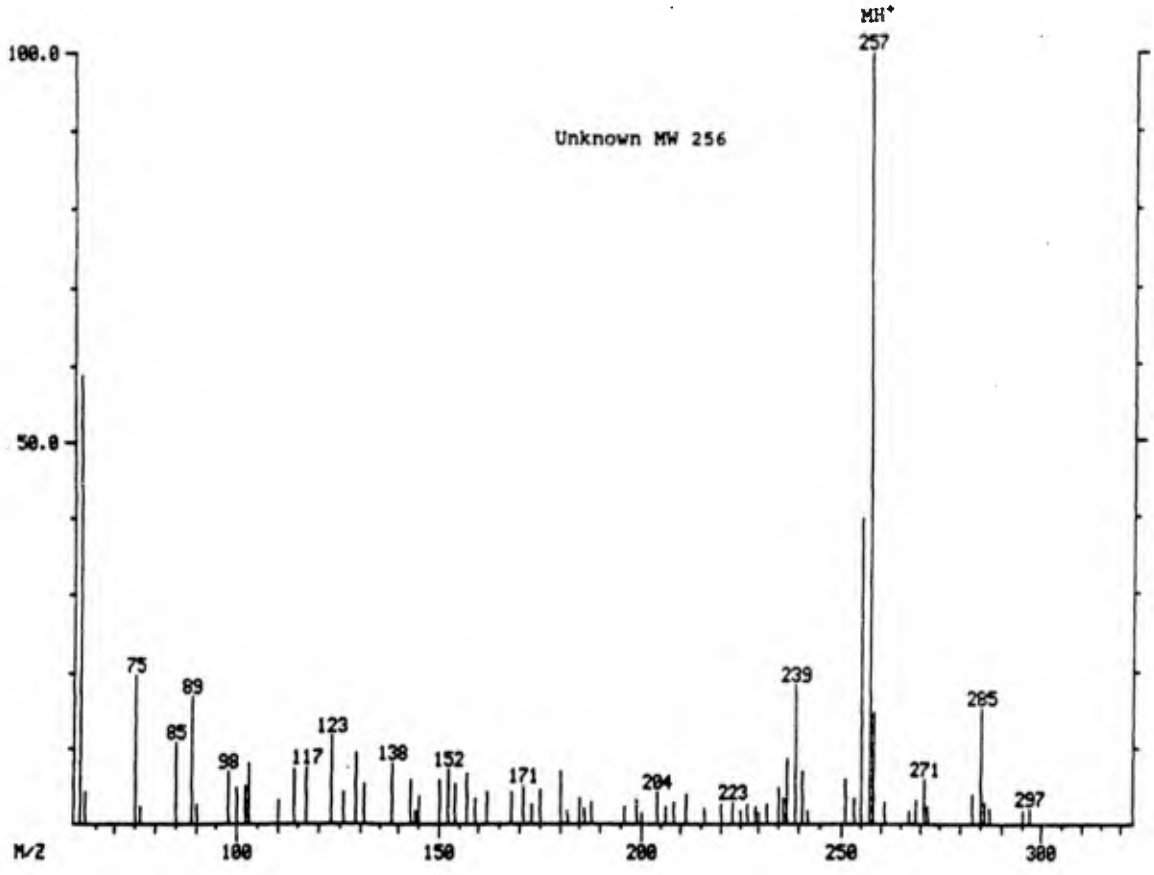
GC/MS/CI Chromatogram of OTH-393-4 (Pig #4)



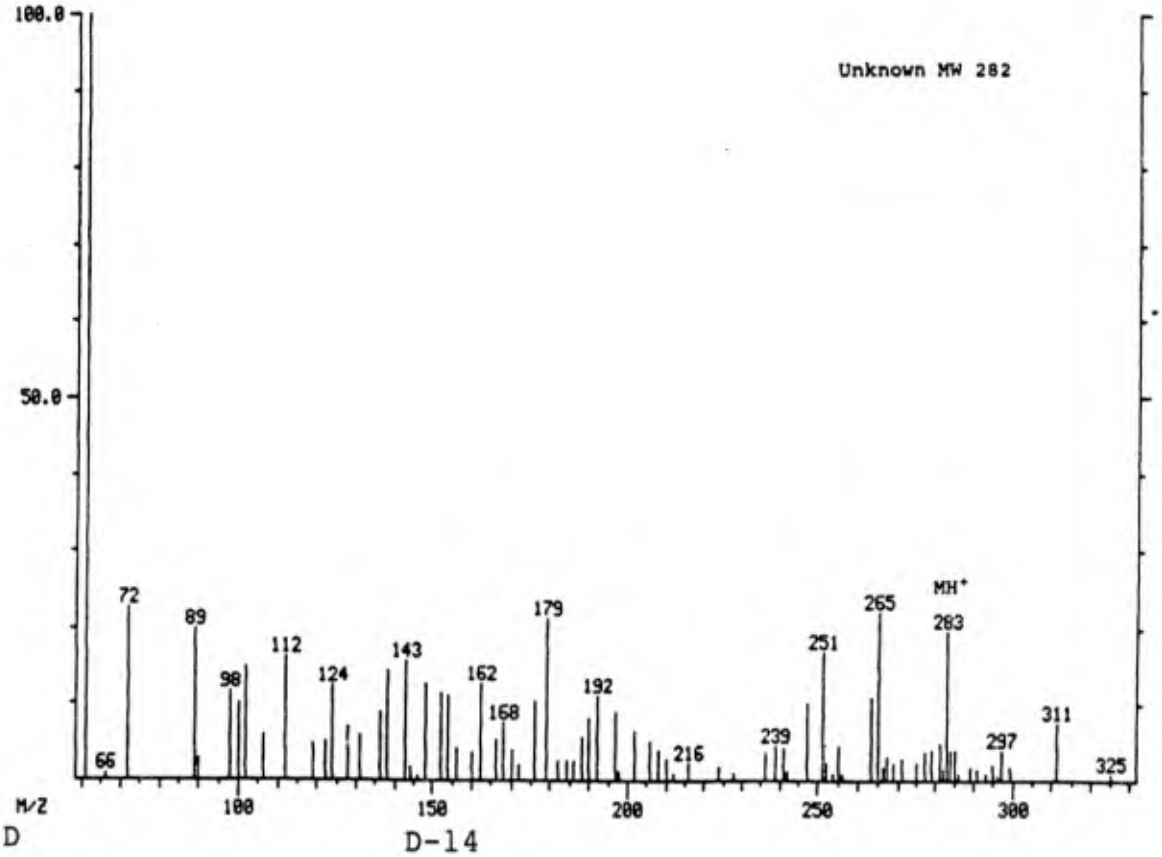
CI Mass Spectrum of OTH-393-4 Scan 639



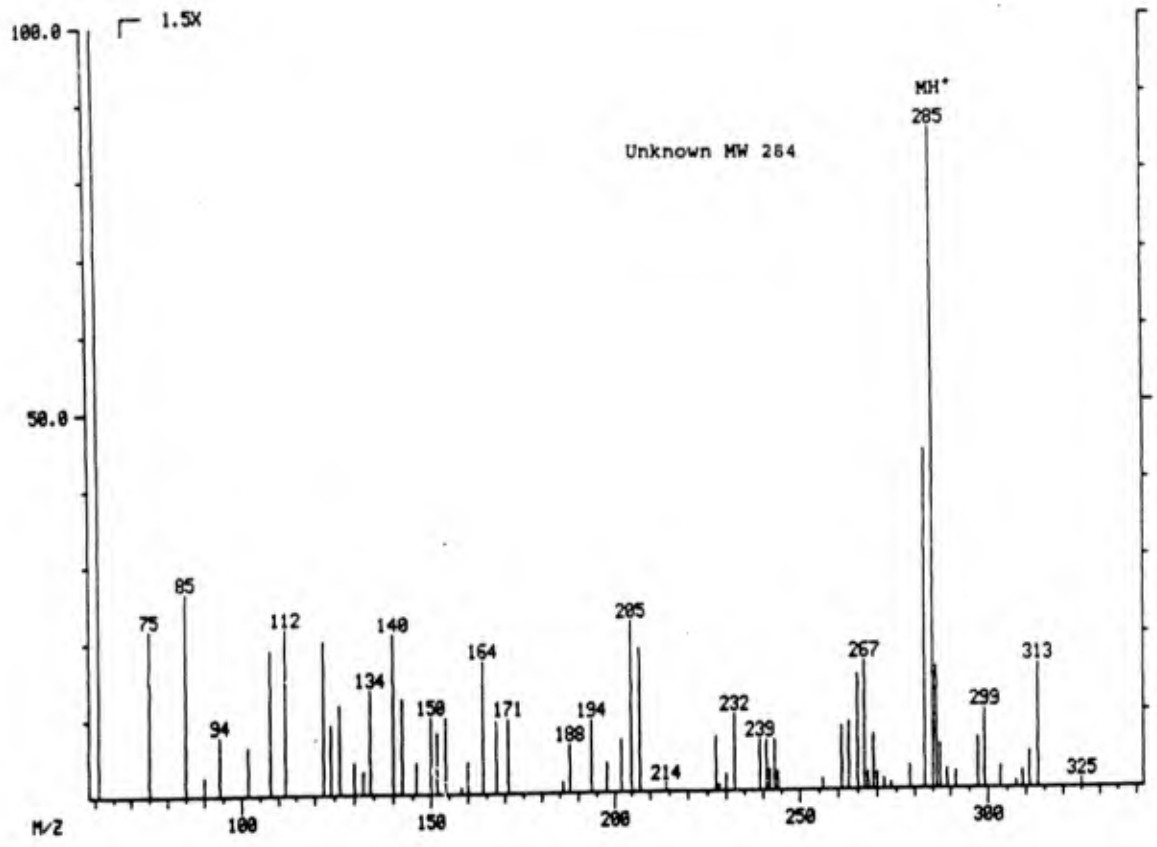
CI Mass Spectrum of OTH-393-4 Scan 807



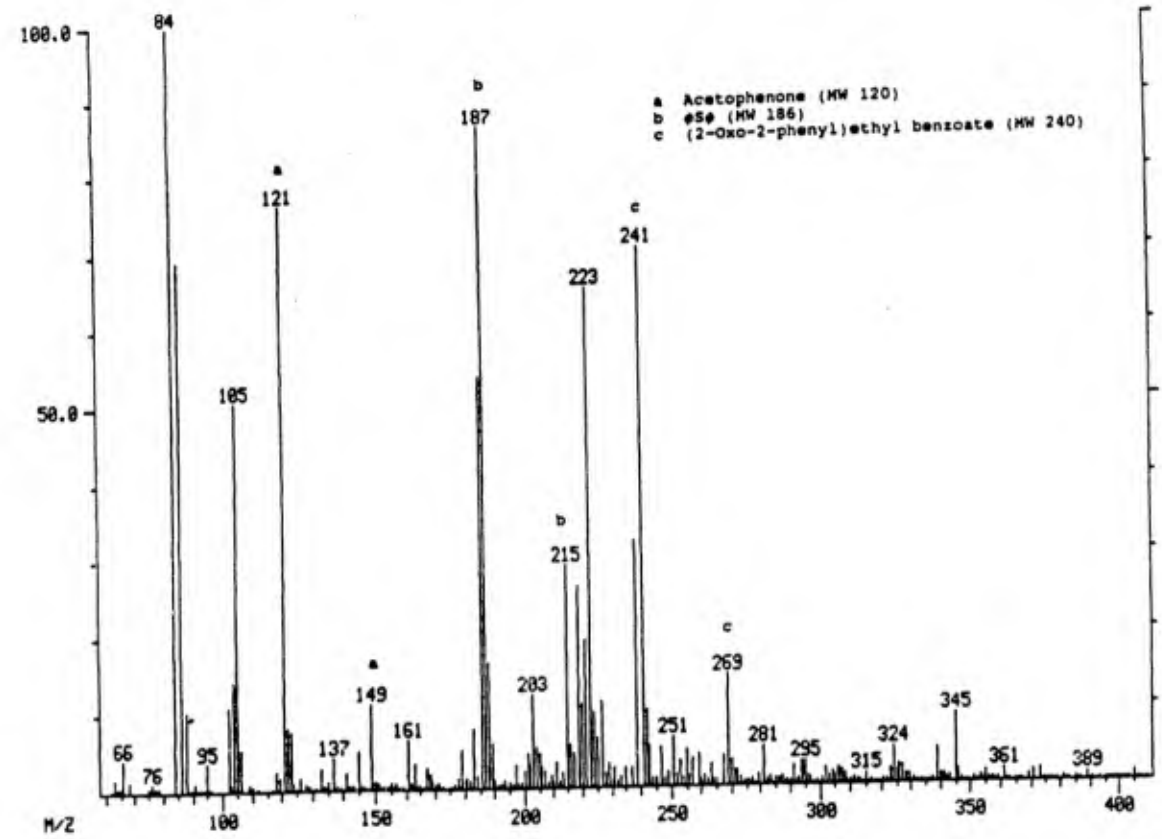
CI Mass Spectrum of OTH-393-4 Scan 903



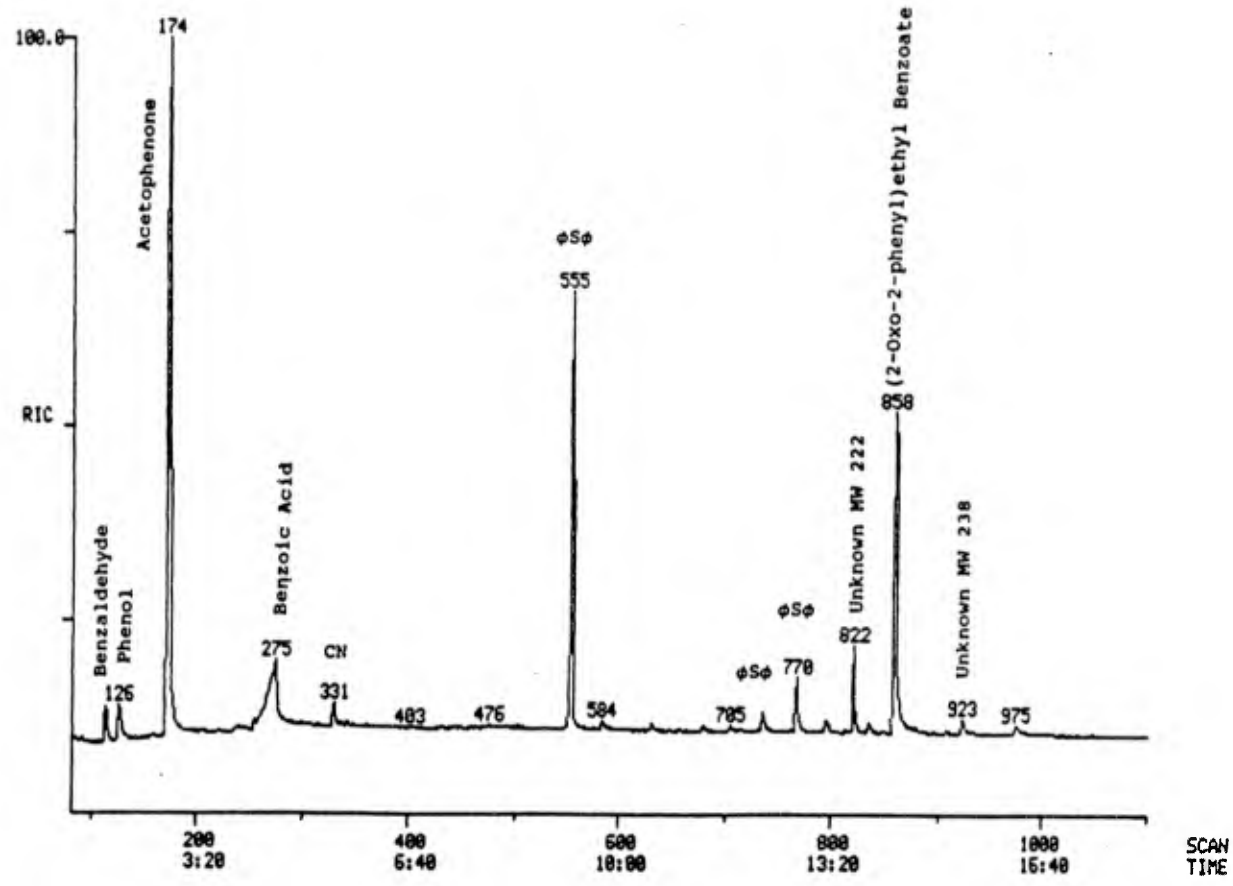
CI Mass Spectrum of OTH-393-4 Scan 917



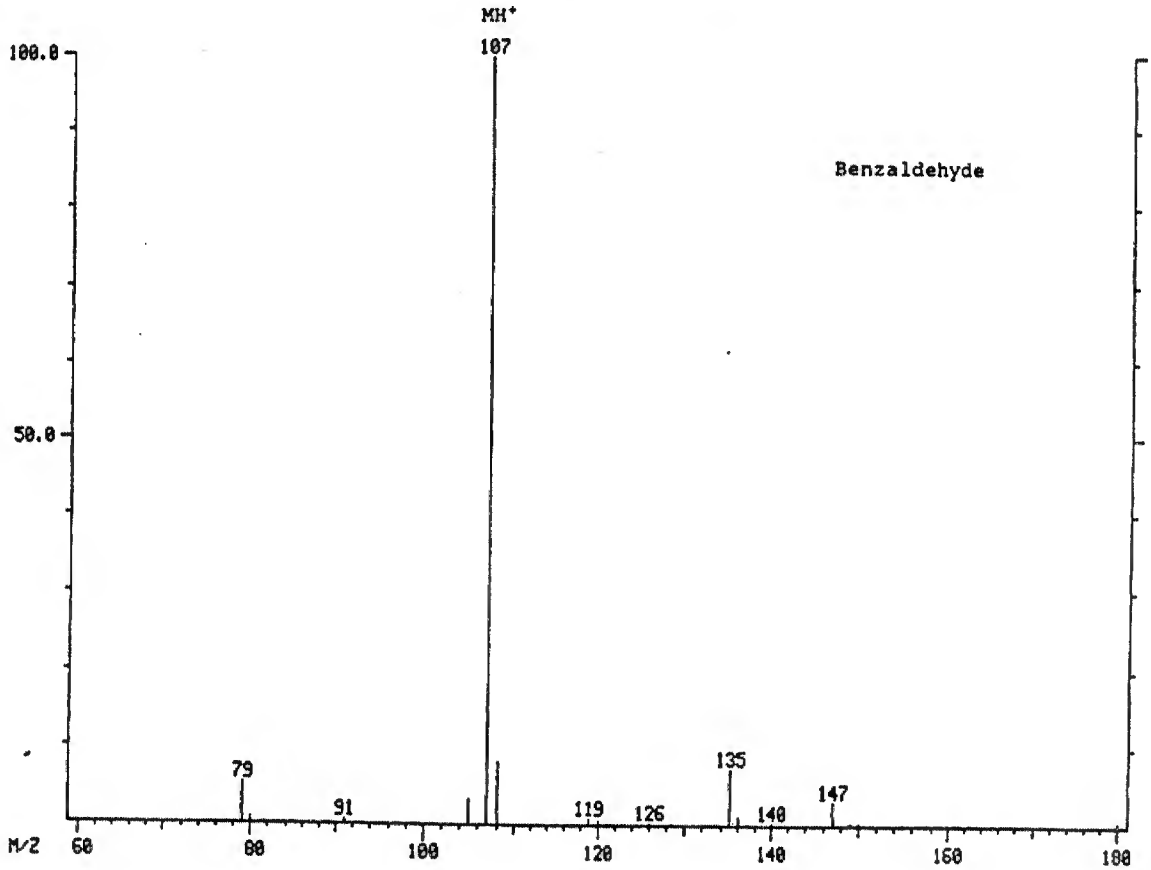
DEP/CI Mass Spectrum of OTH-393-6a (Pig #6)



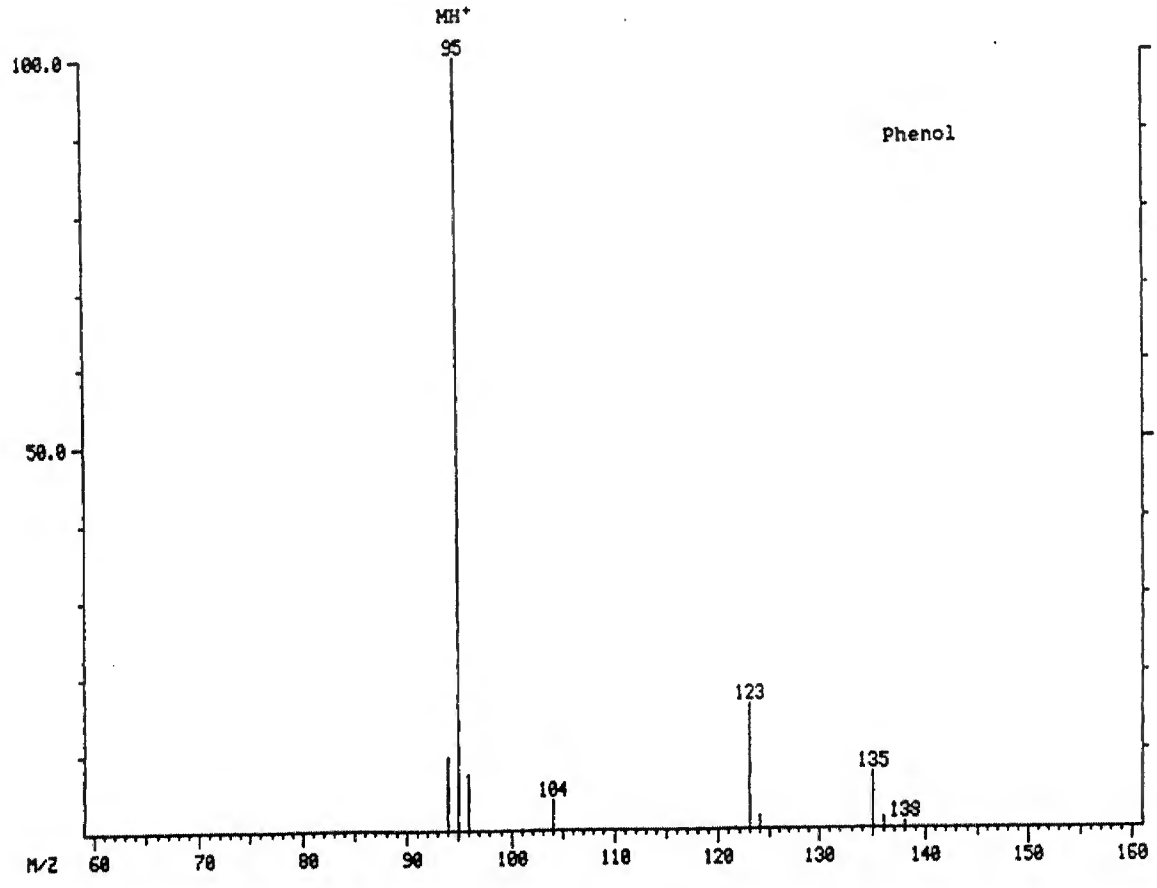
GC/MS/CI Chromatogram of OTH-393-6a (Pig #6)



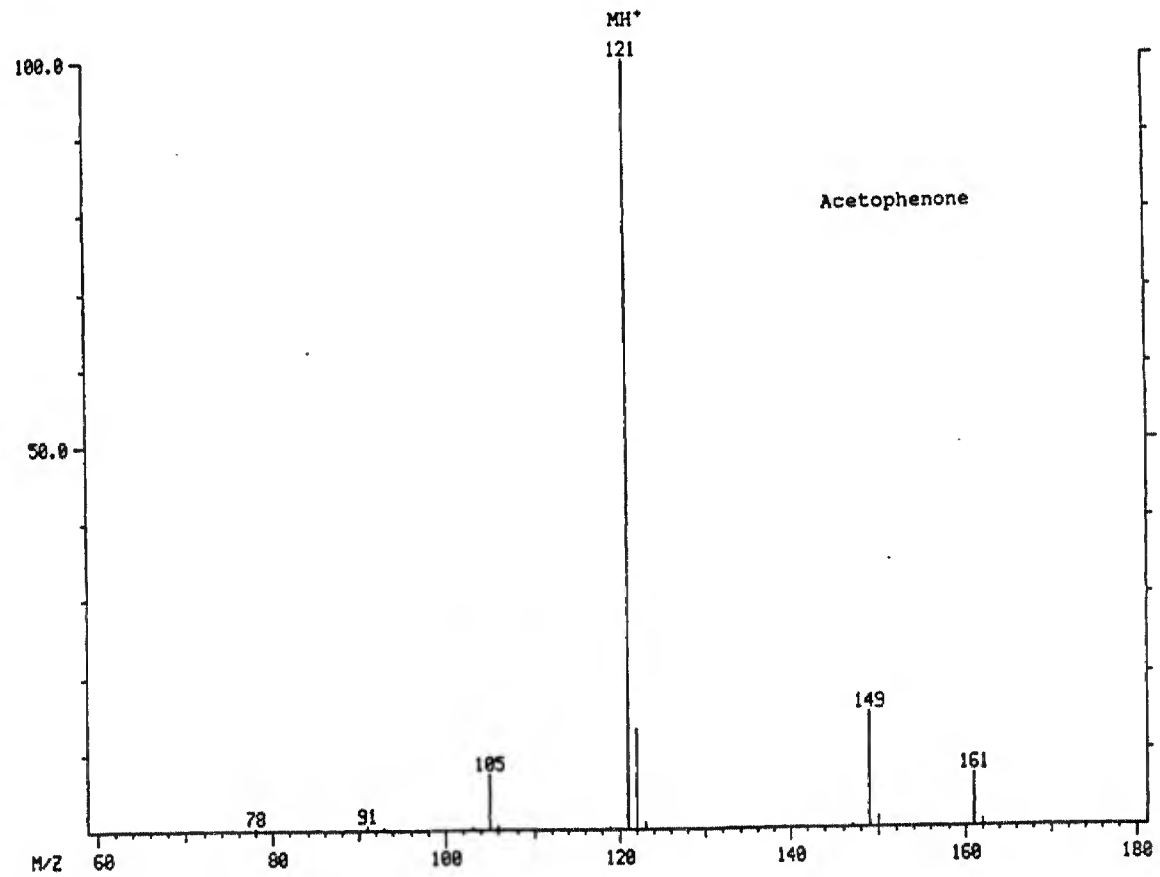
CI Mass Spectrum of OTH-393-6a Scan 113



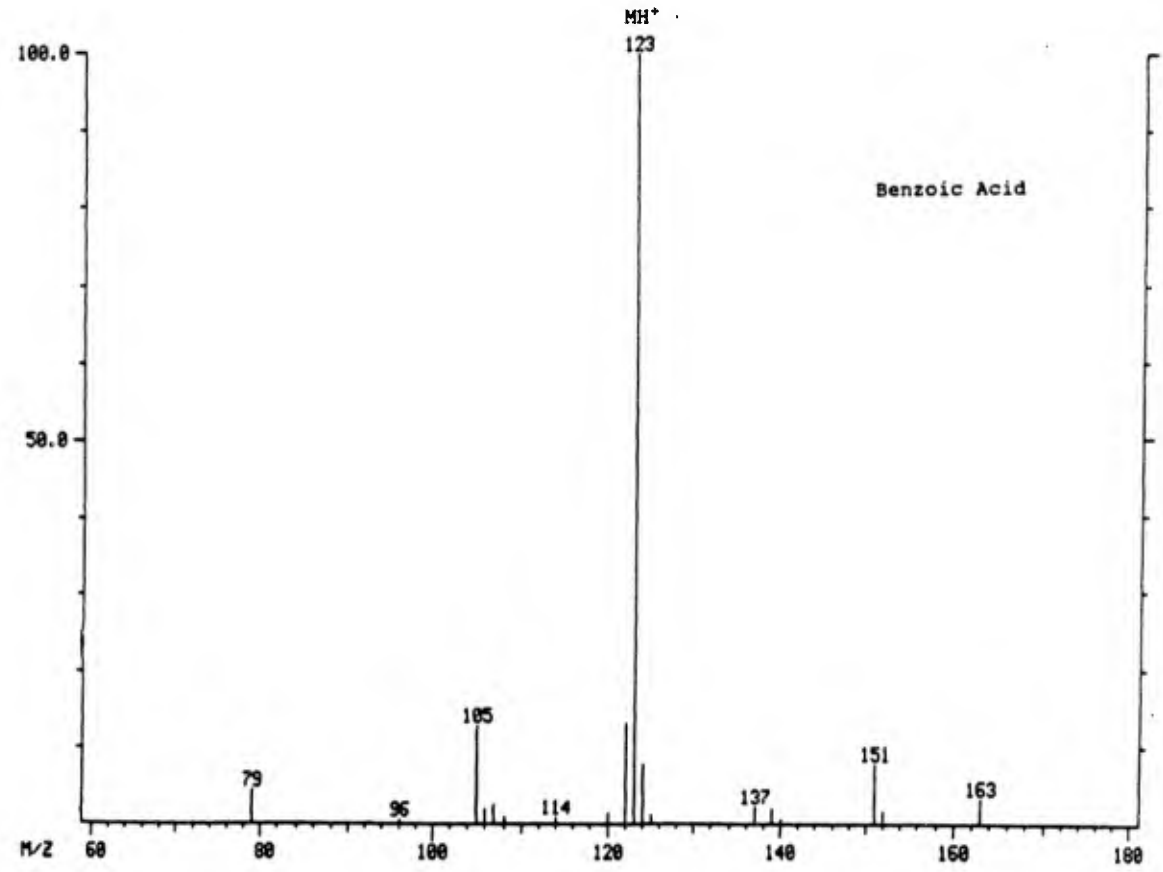
CI Mass Spectrum of OTH-393-6a Scan 126



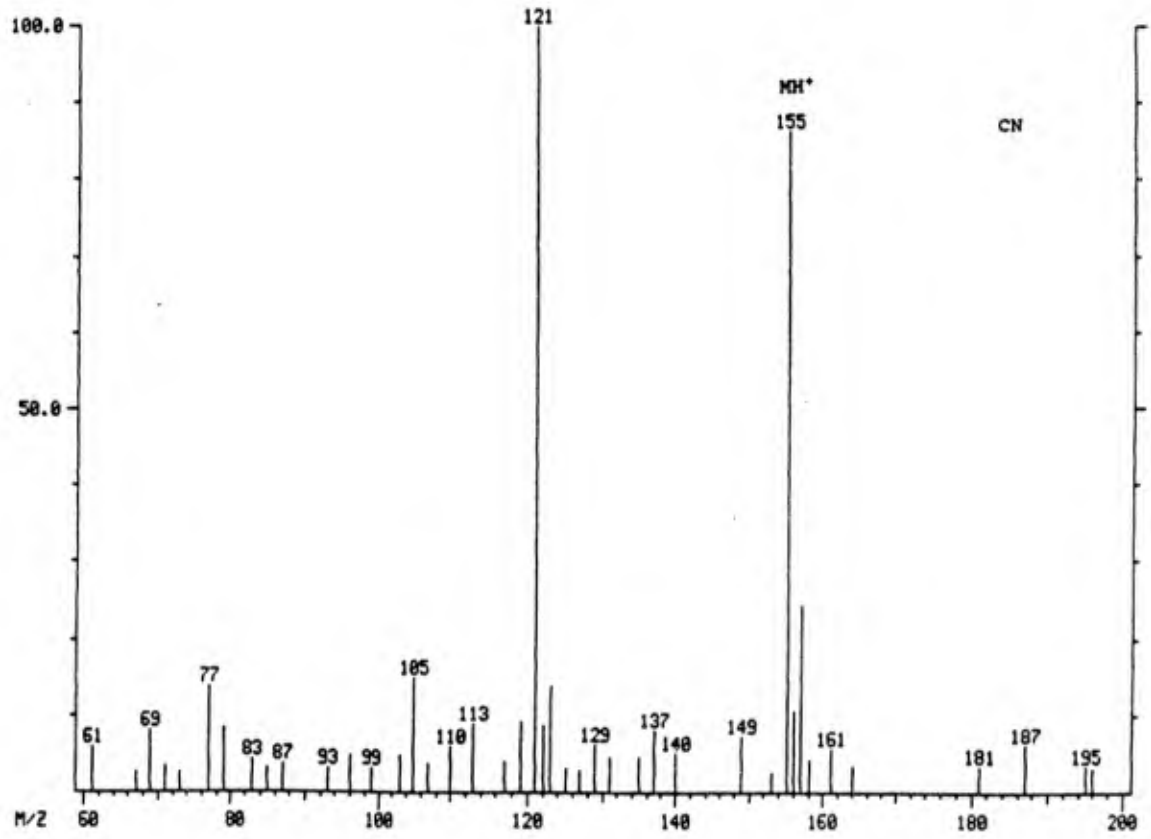
CI Mass Spectrum of OTH-393-6a Scan 174



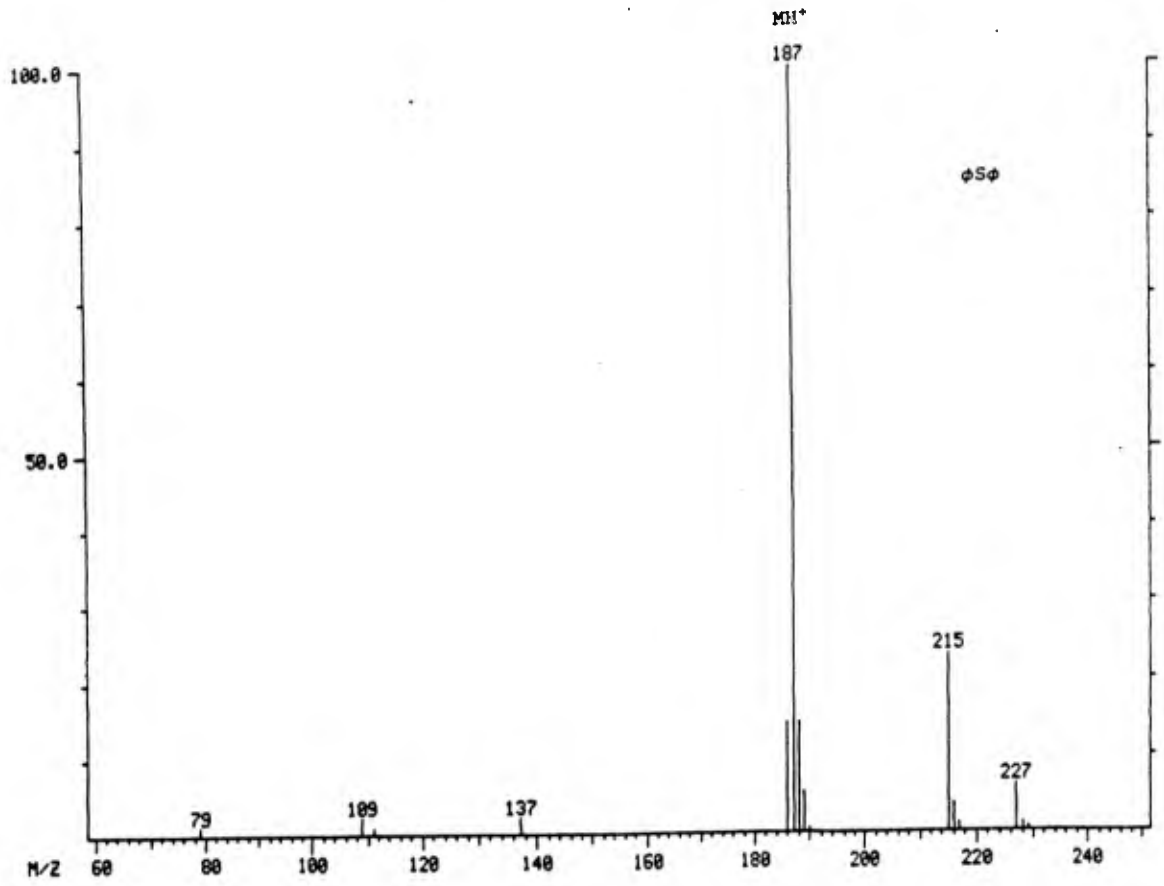
CI Mass Spectrum of OTH-393-6a Scan 275



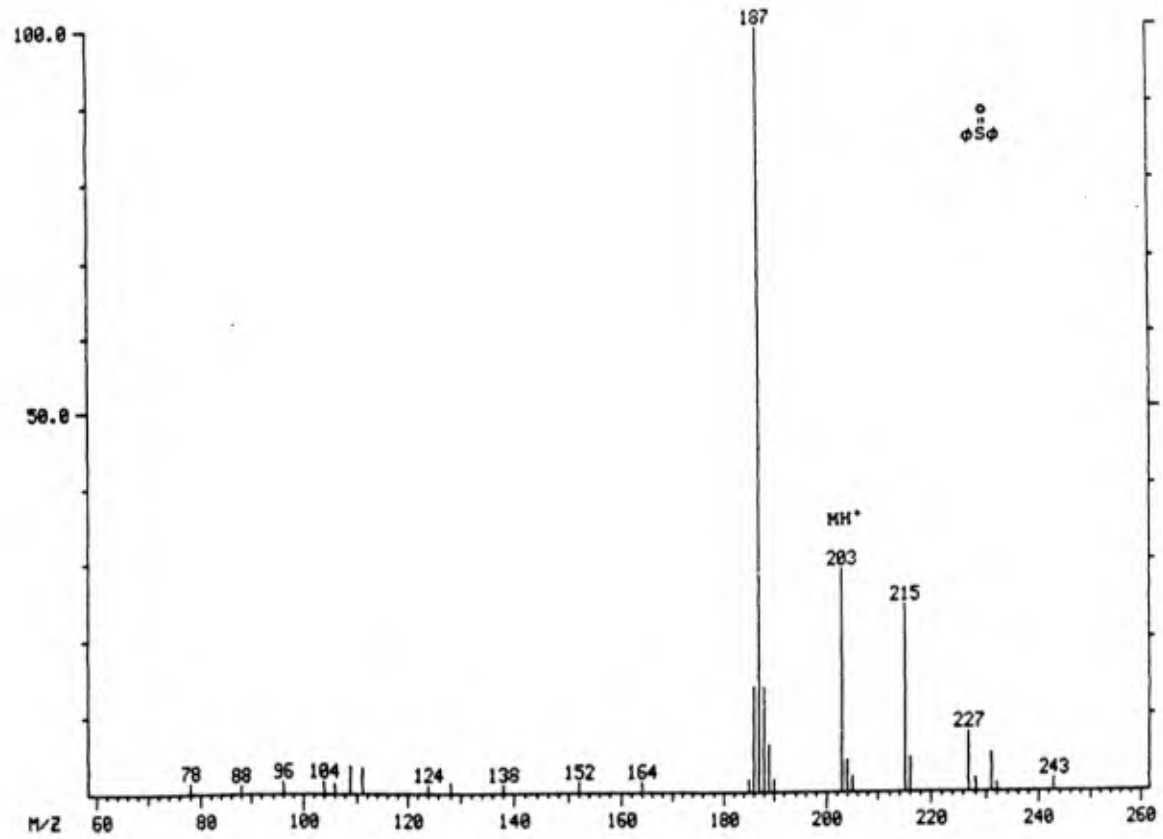
CI Mass Spectrum of OTH-393-6a Scan 331



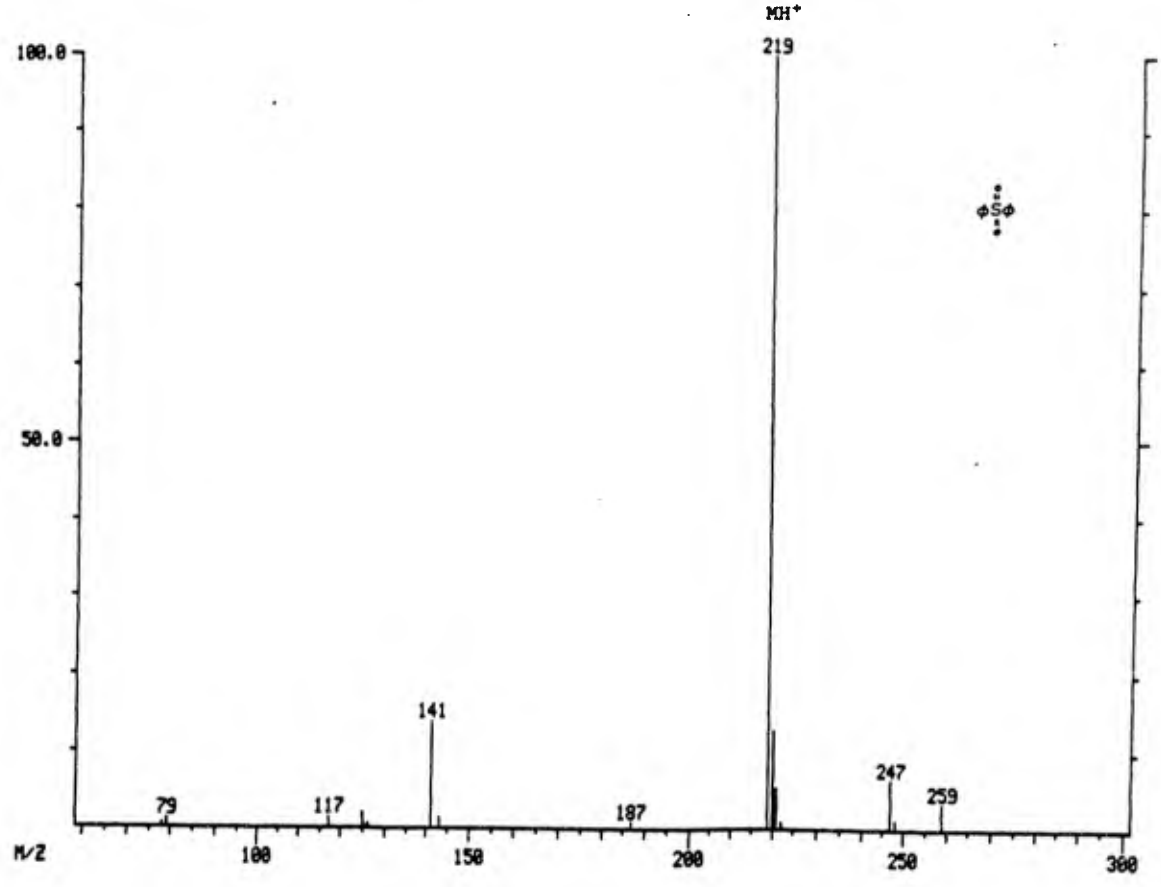
CI Mass Spectrum of OTH-393-6a Scan 555



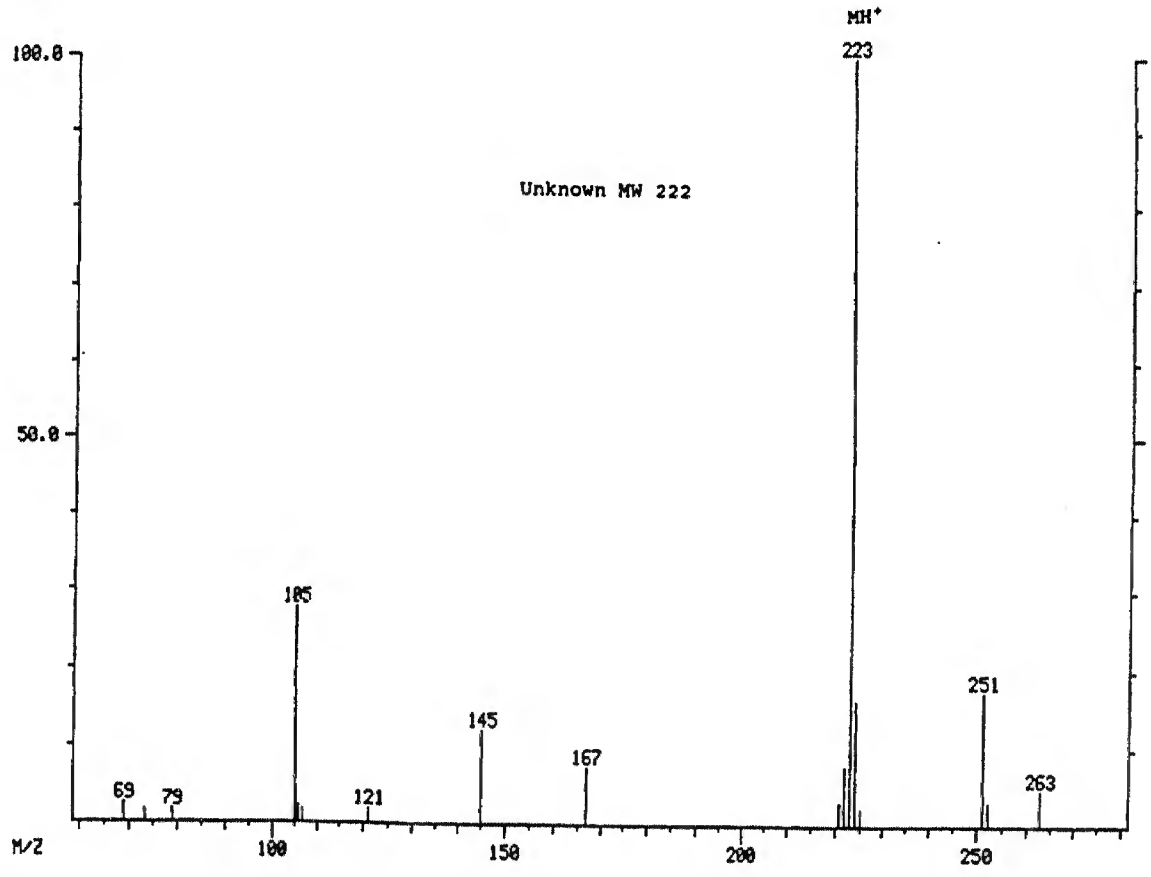
CI Mass Spectrum of OTH-393-6a Scan 737



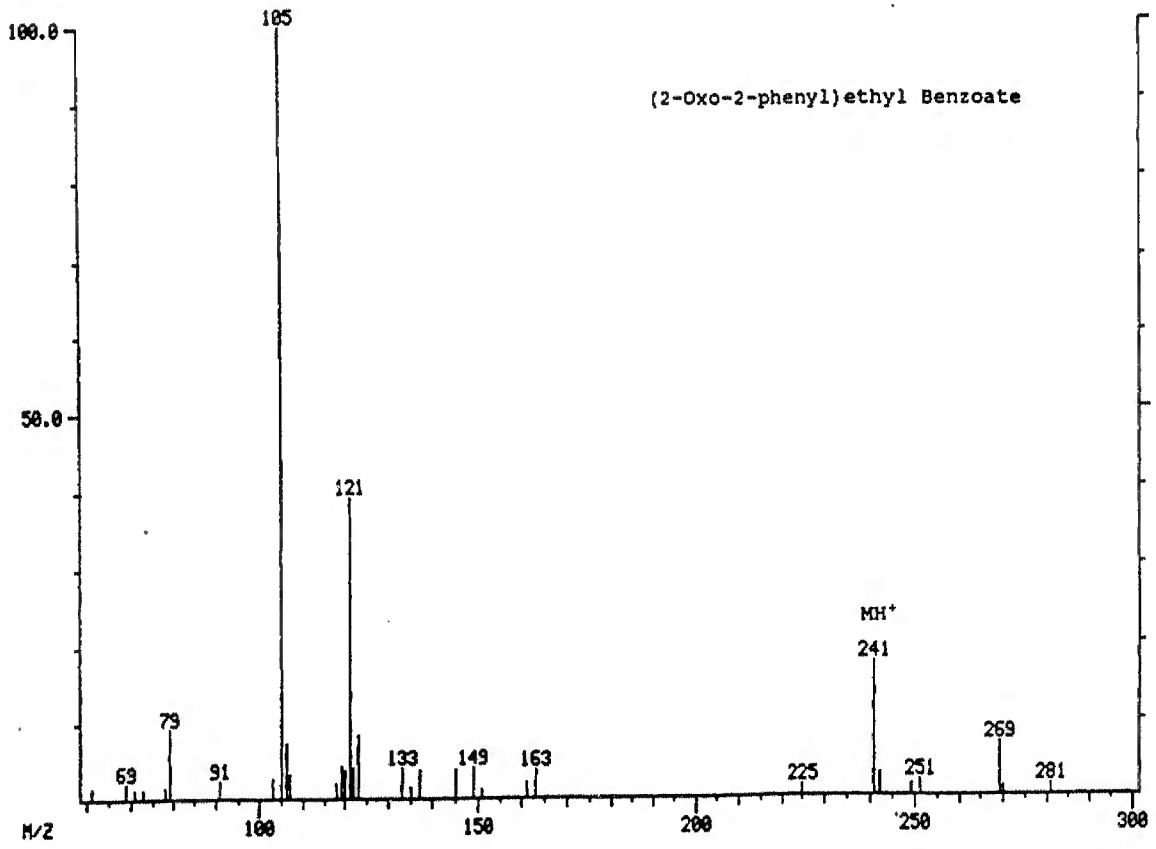
CI Mass Spectrum of OTH-393-6a Scan 770



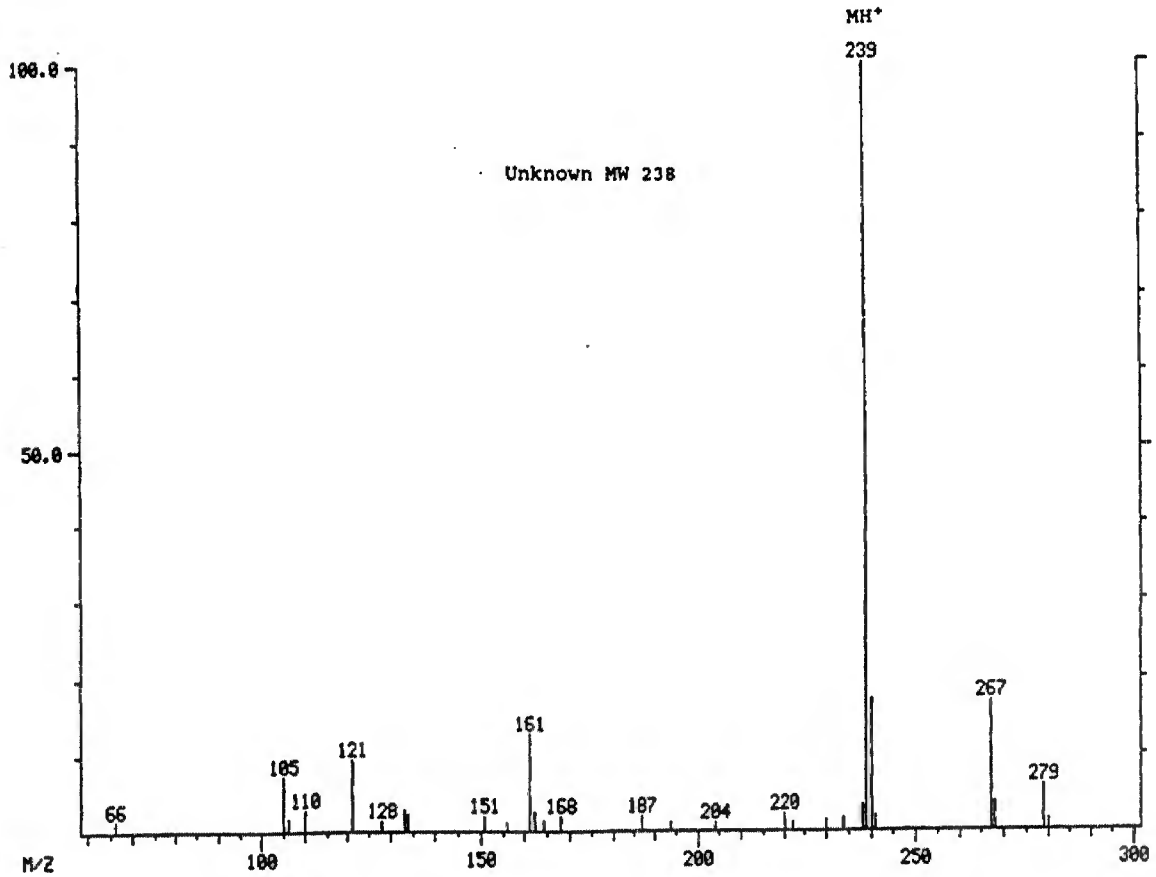
CI Mass Spectrum of OTH-393-6a Scan 822



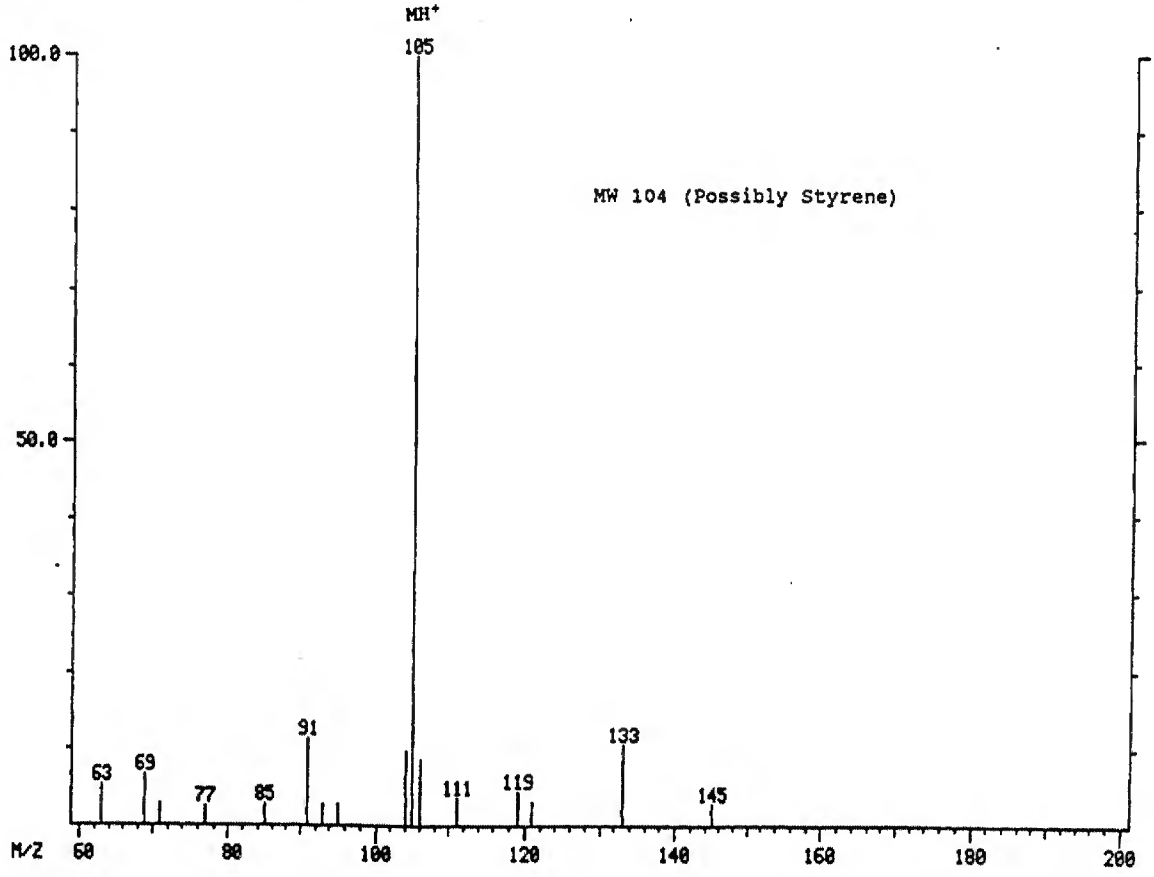
CI Mass Spectrum of OTH-393-6a Scan 858



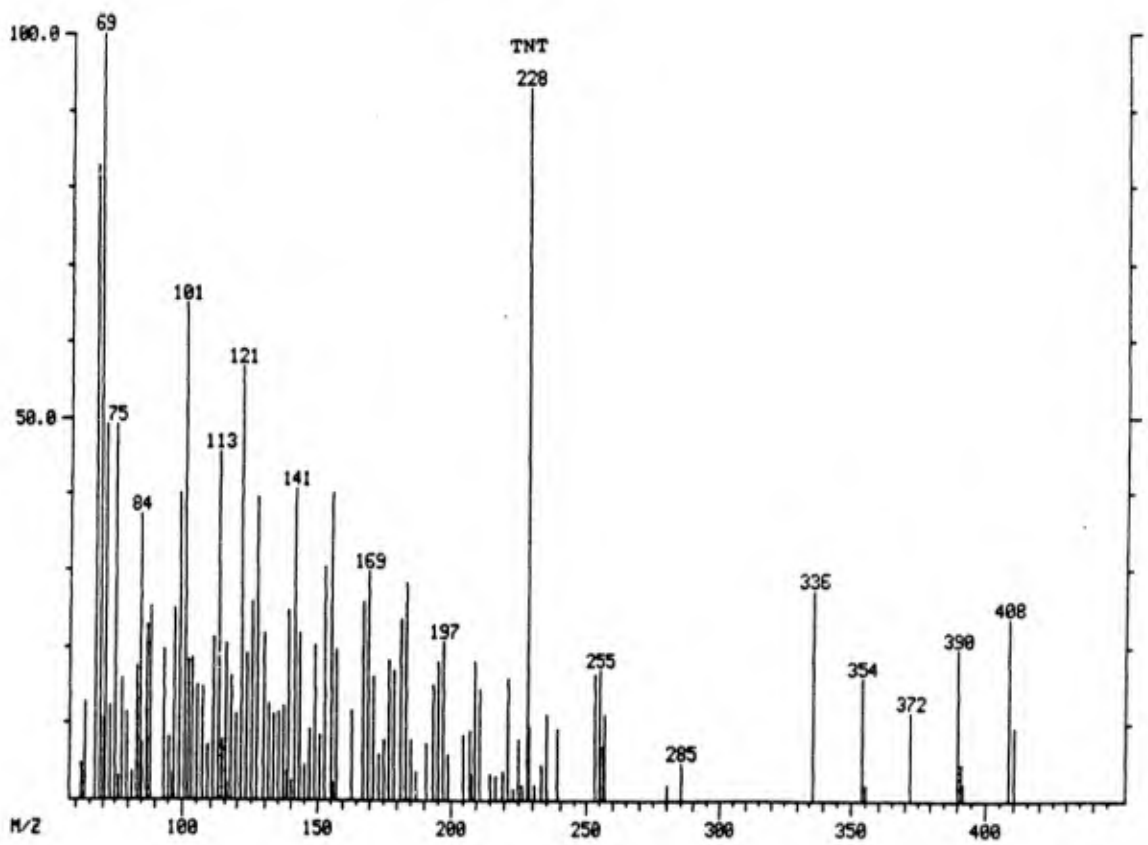
CI Mass Spectrum of OTH-393-6a Scan 923



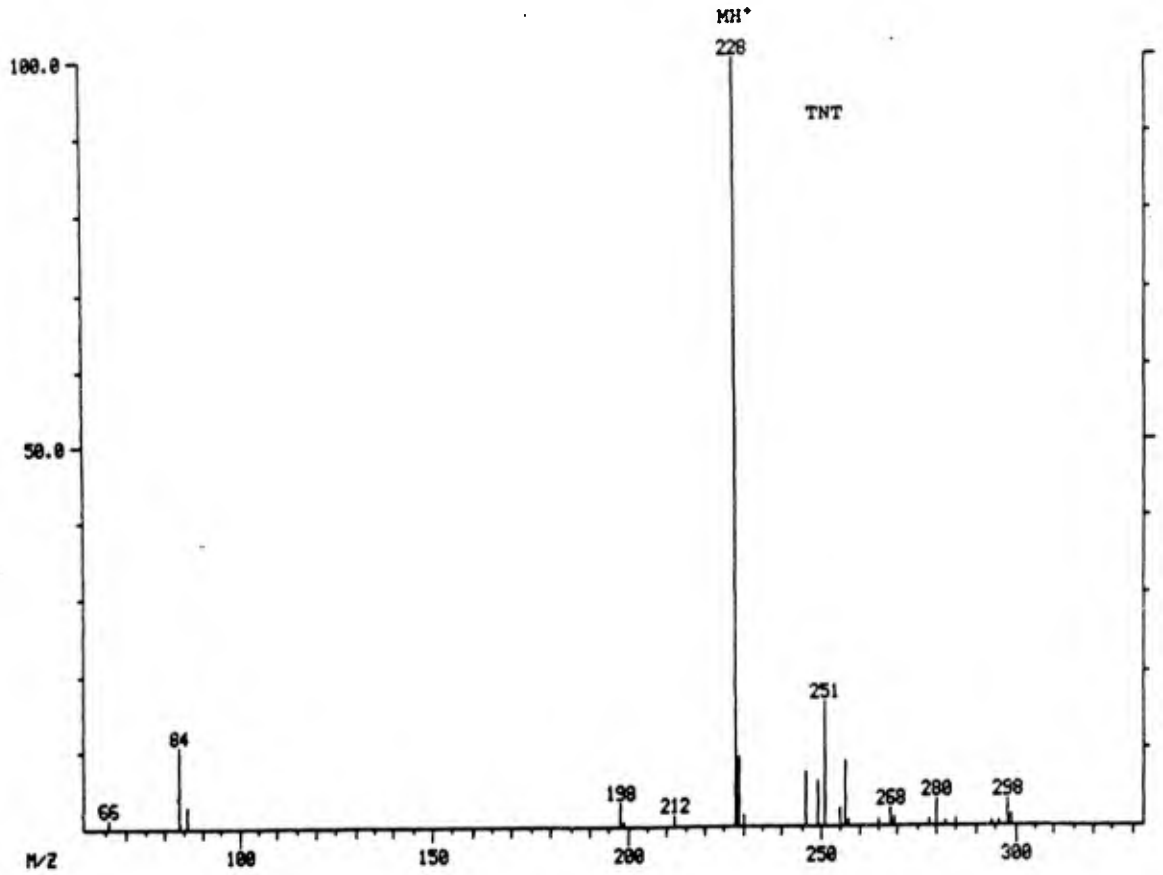
DEP/CI Mass Spectrum of OTH-393-6e (Pig #6)



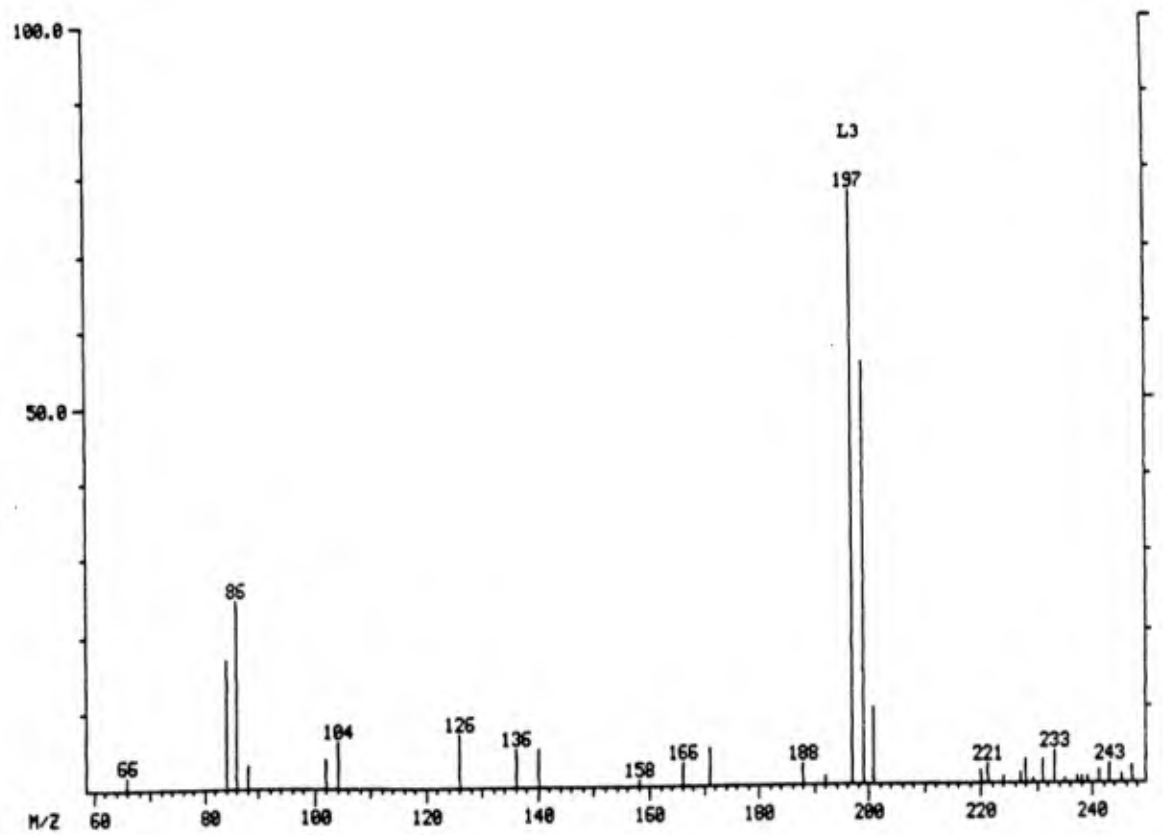
DEP/CI Mass Spectrum of OTH-293-9a (Pig #9)



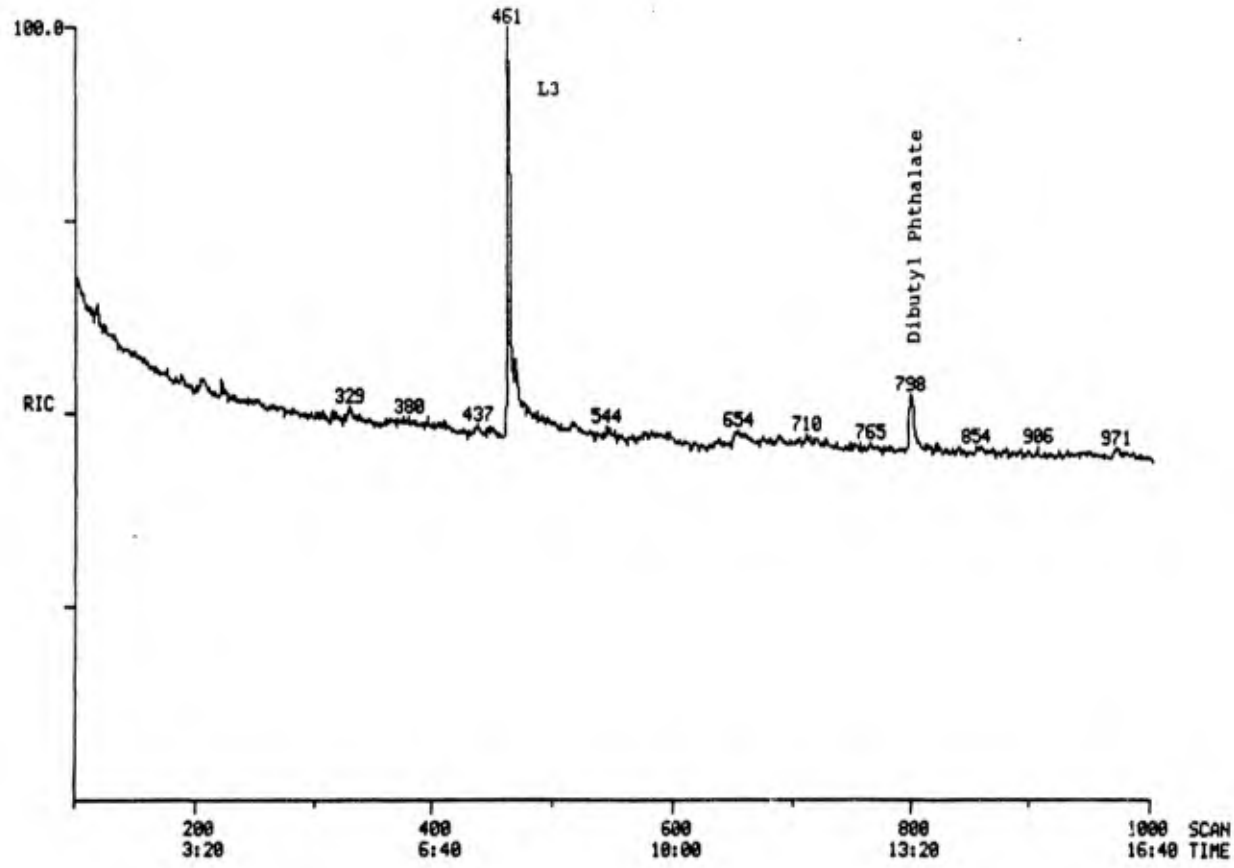
DEP/CI Mass Spectrum of OTH-293-9b (Pig #9)



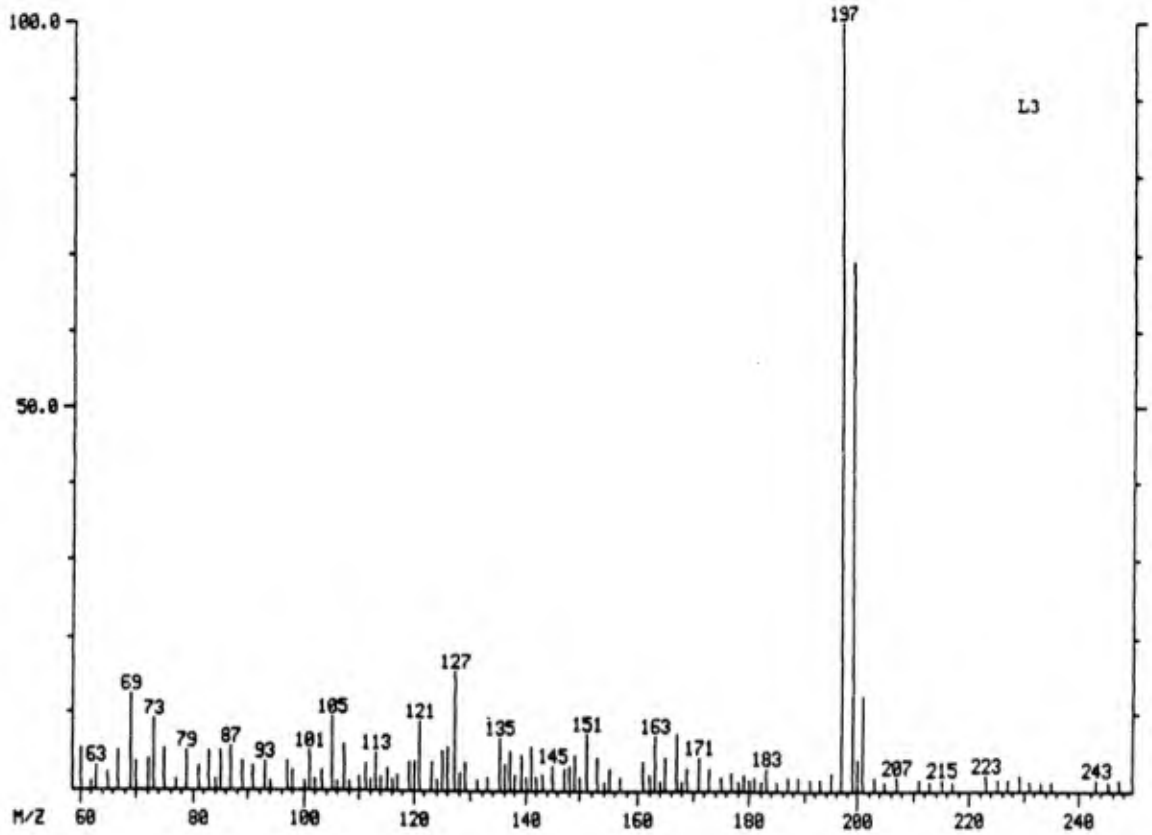
DEP/CI Mass Spectrum of OTH-293-9c (Pig #9)



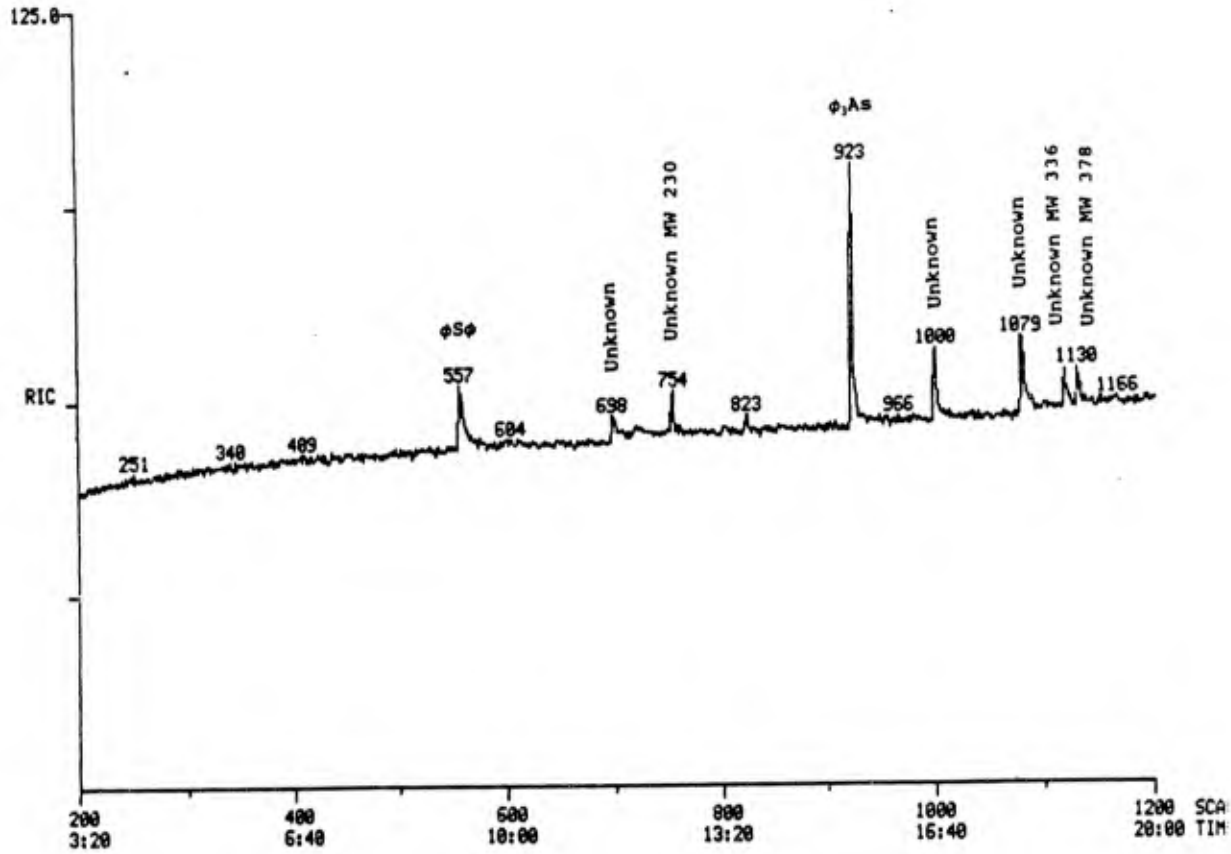
GC/MS/CI Chromatogram of OTH-293-9c (Pig #9)



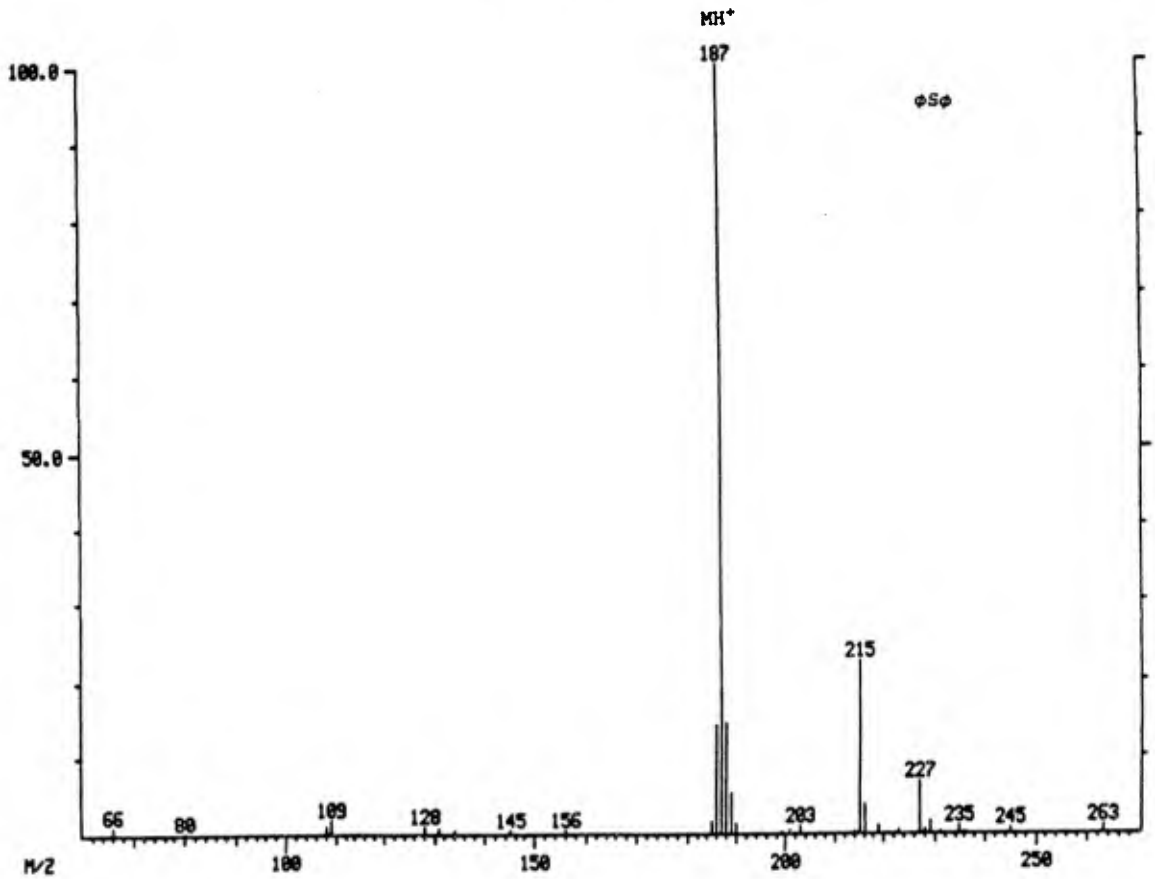
CI Mass Spectrum of OTH-293-9c Scan 461



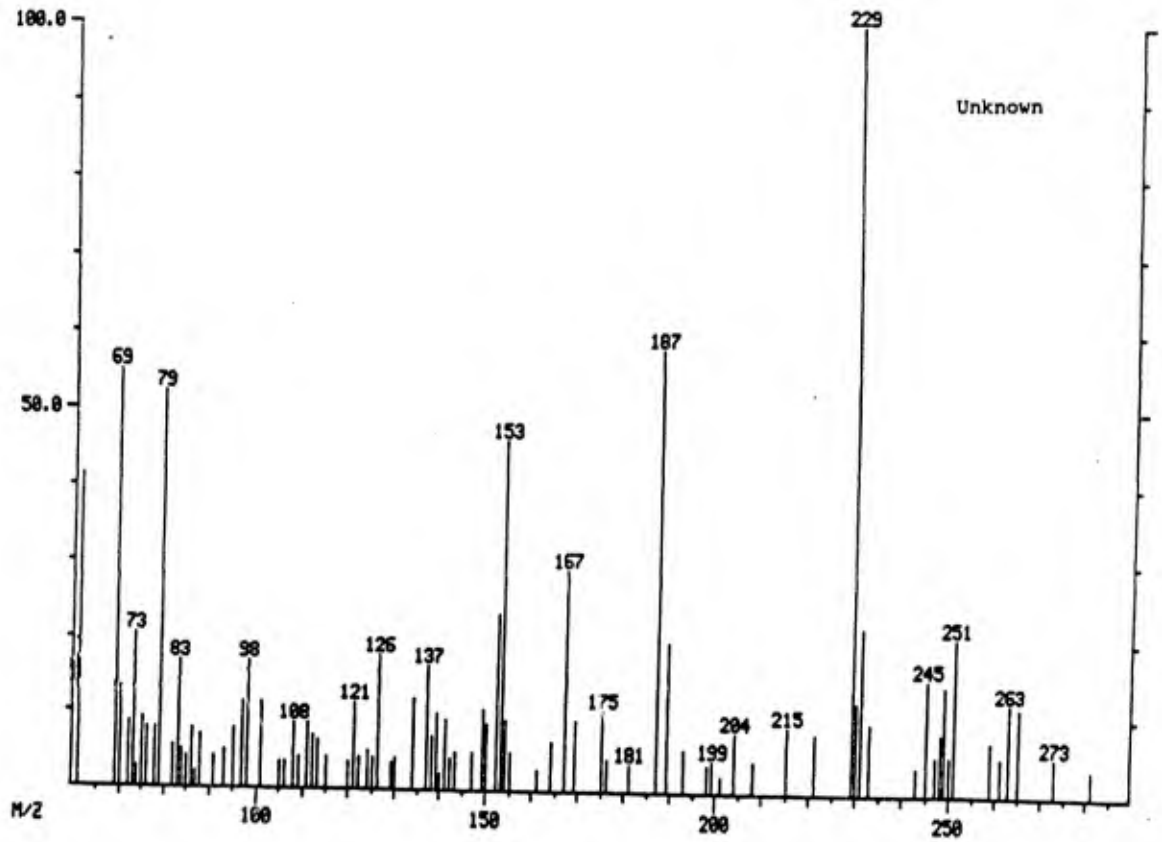
GC/MS/CI Chromatogram of OTH-293-9d (Pig #9)



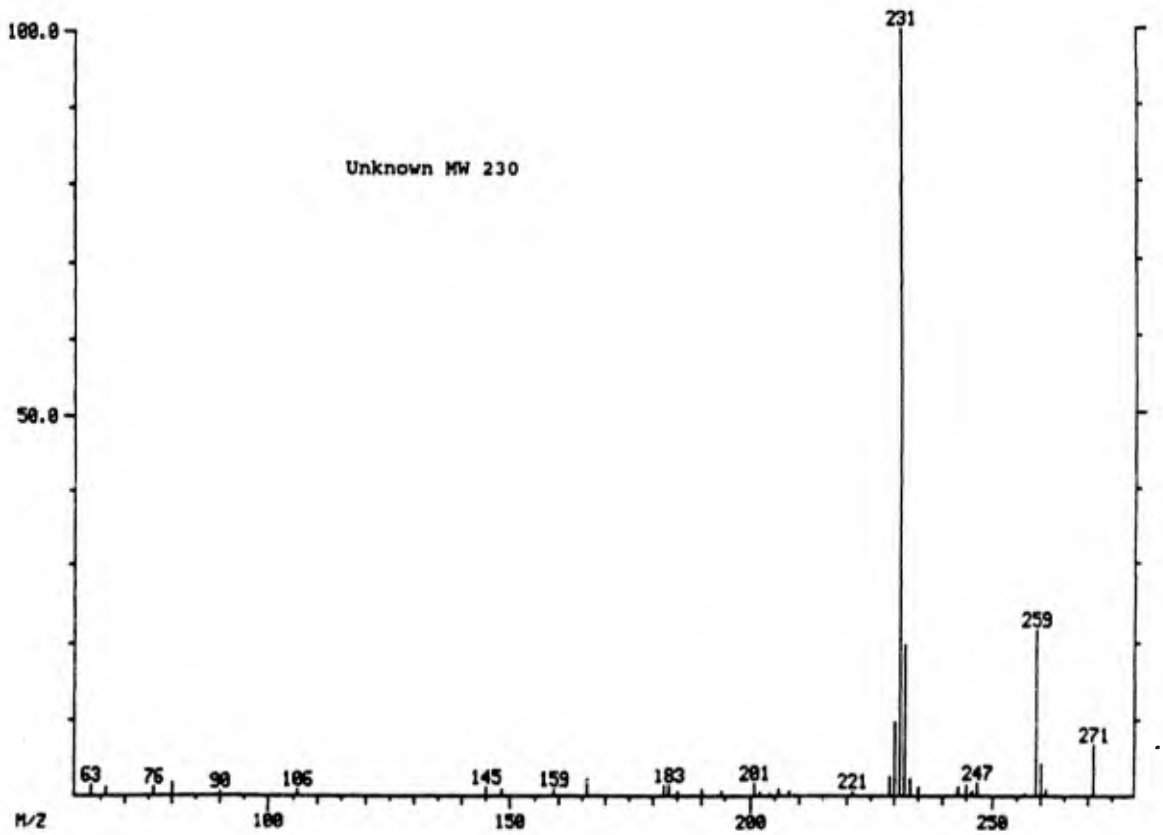
CI Mass Spectrum of OTH-293-9d Scan 557



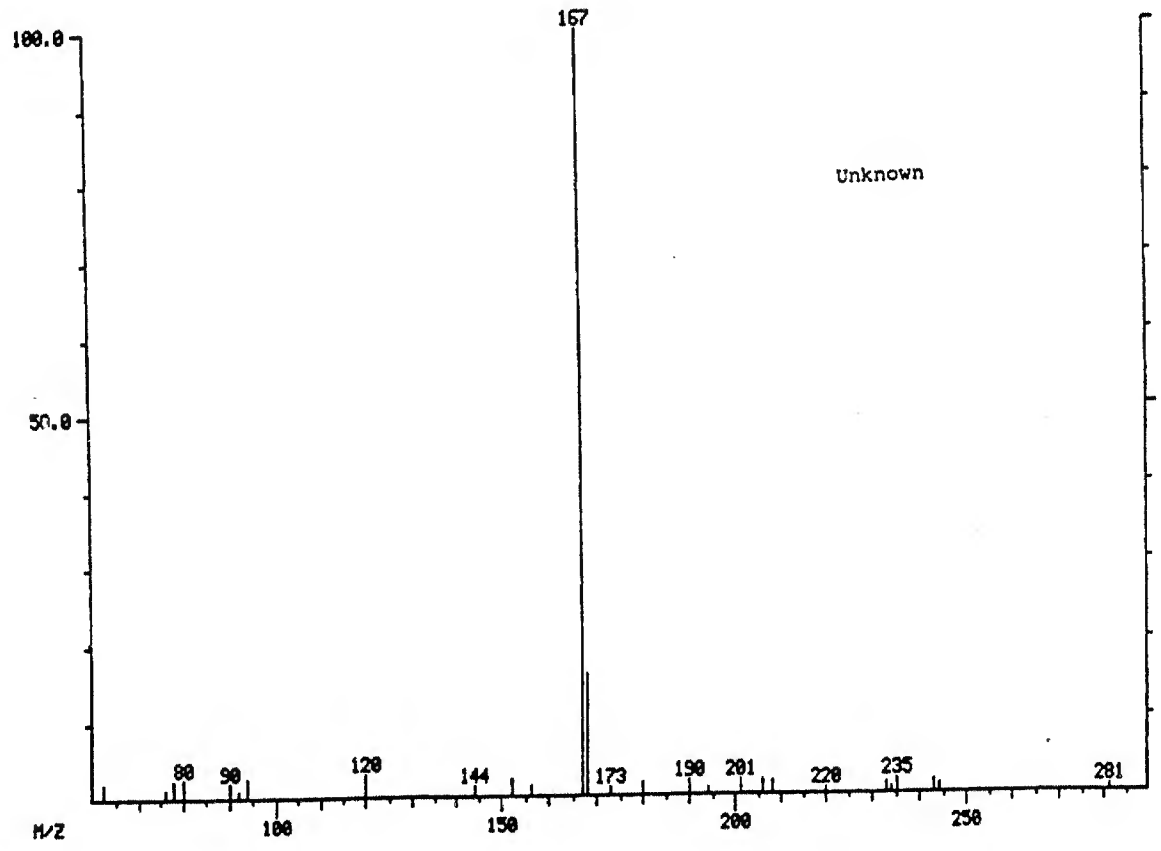
CI Mass Spectrum of OTH-293-9d Scan 698



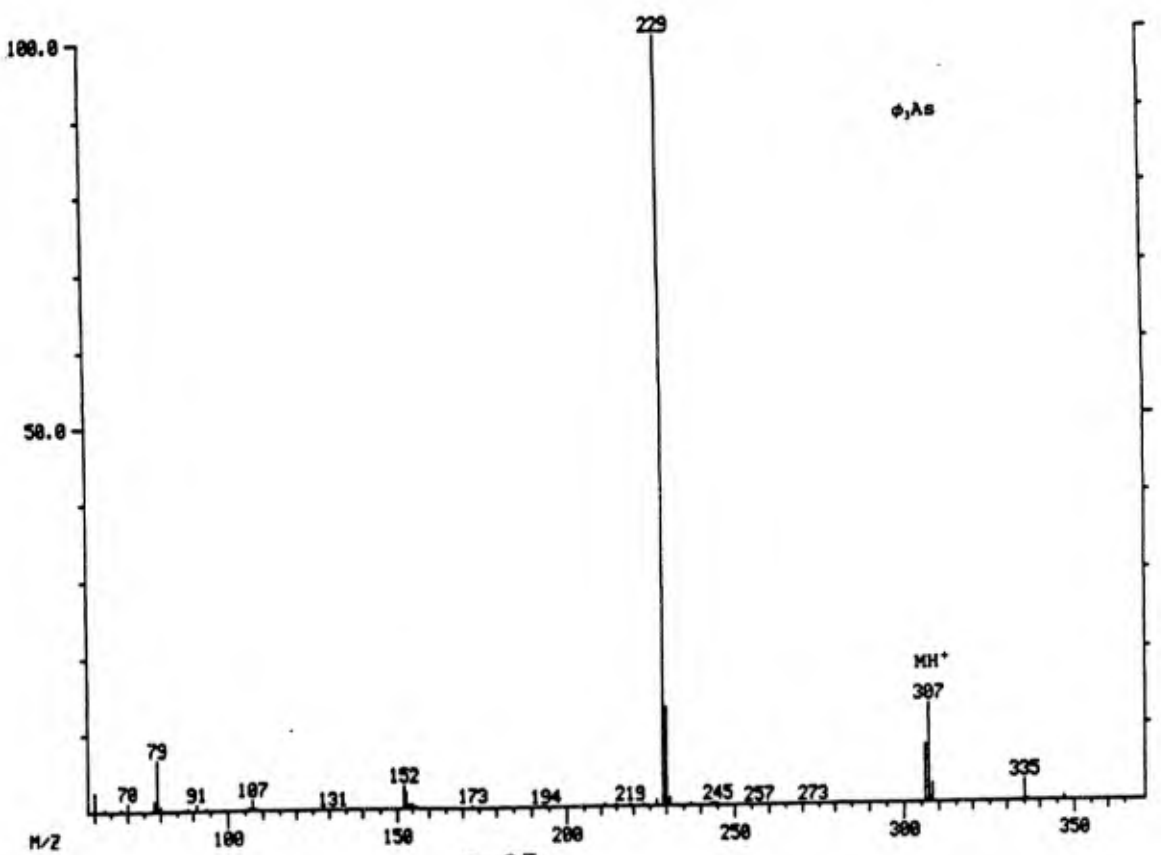
CI Mass Spectrum of OTH-293-9d Scan 754



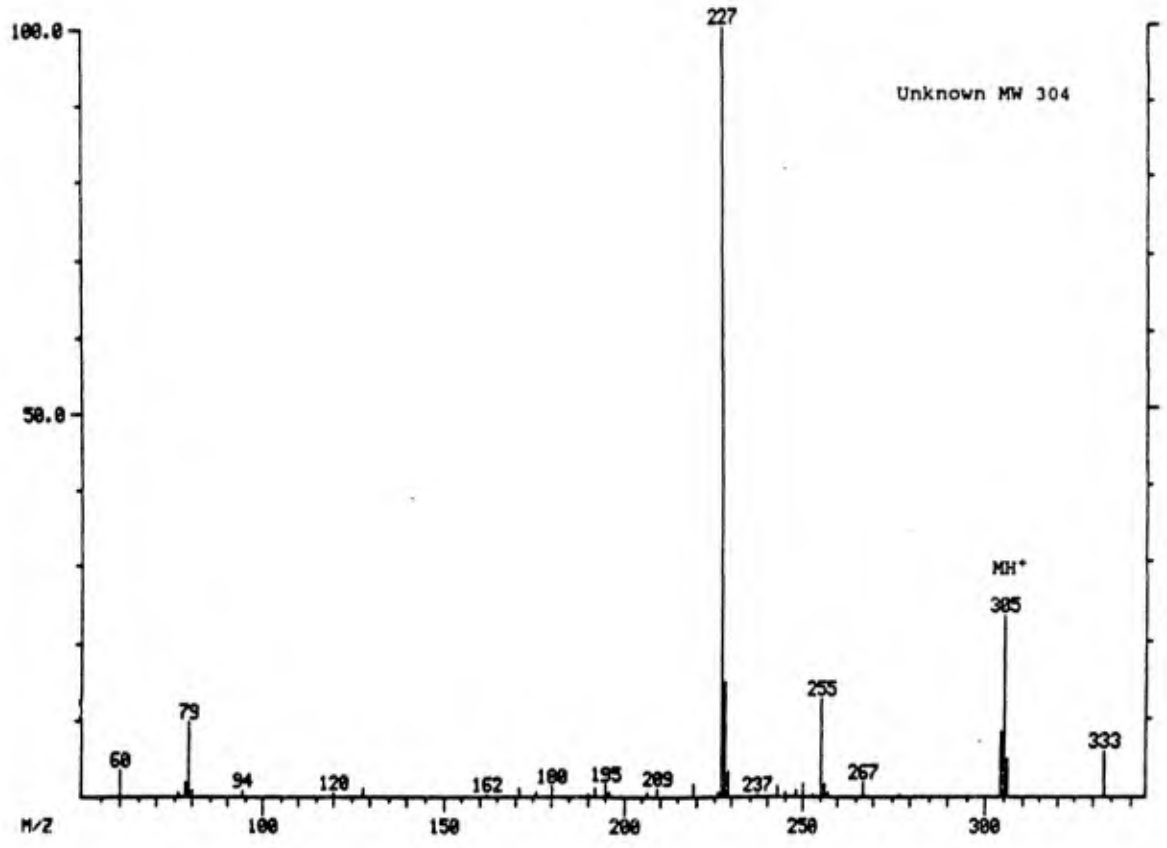
CI Mass Spectrum of OTH-293-9d Scan 823



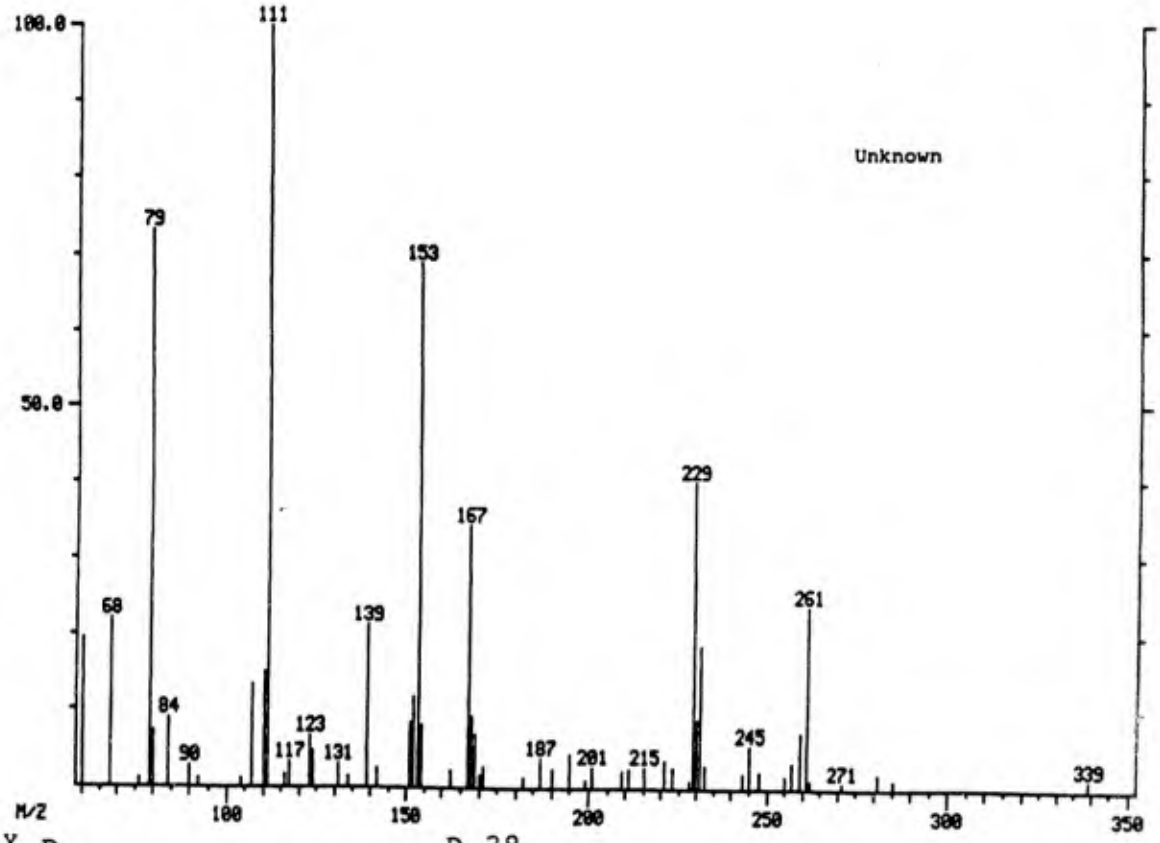
CI Mass Spectrum of OTH-293-9d Scan 923



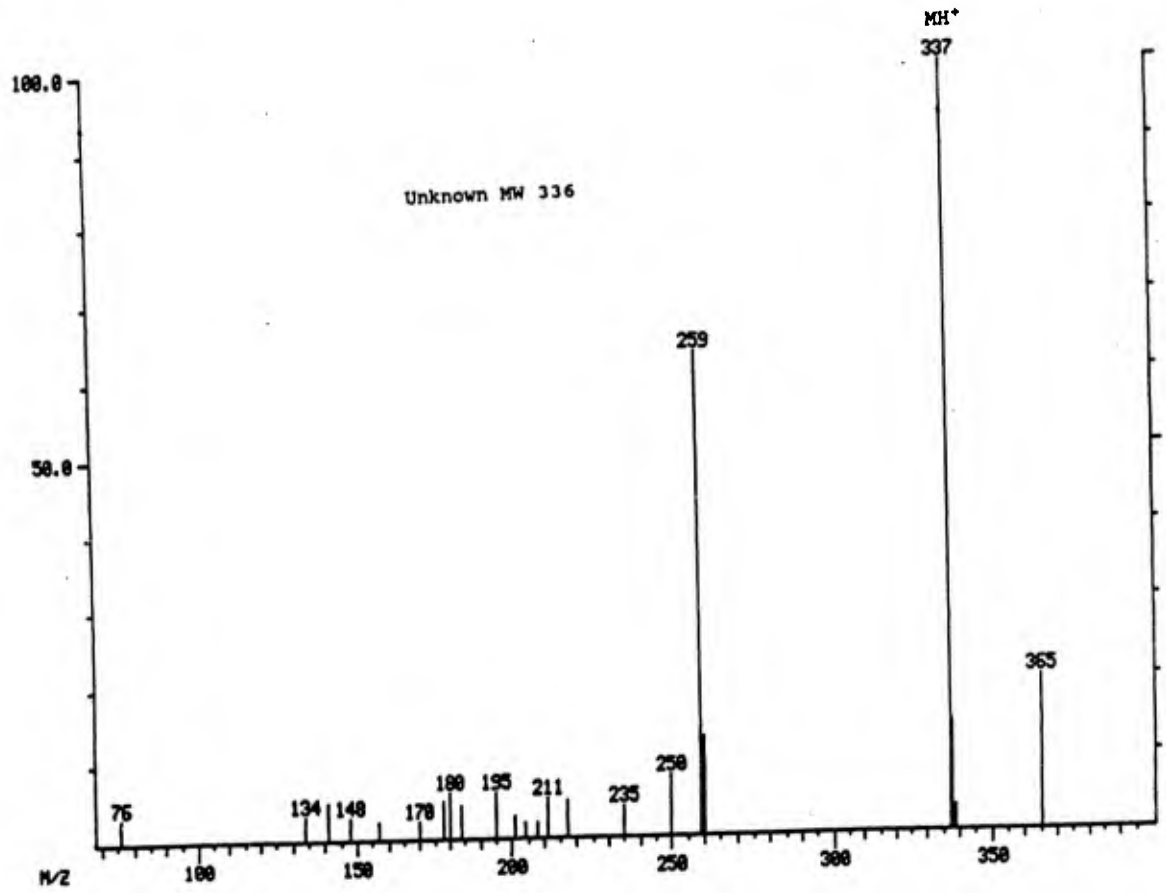
CI Mass Spectrum of OTH-293-9d Scan 1000



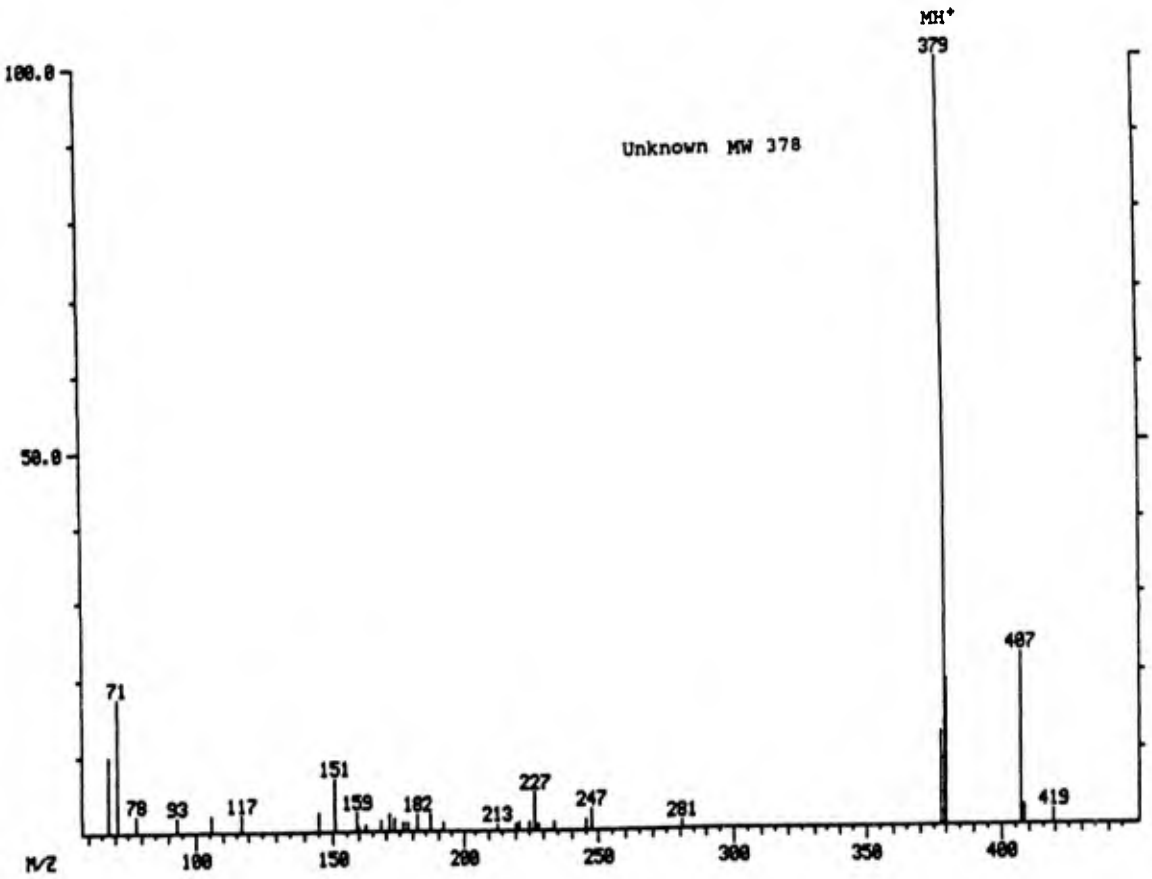
CI Mass Spectrum of OTH-293-9d Scan 1079



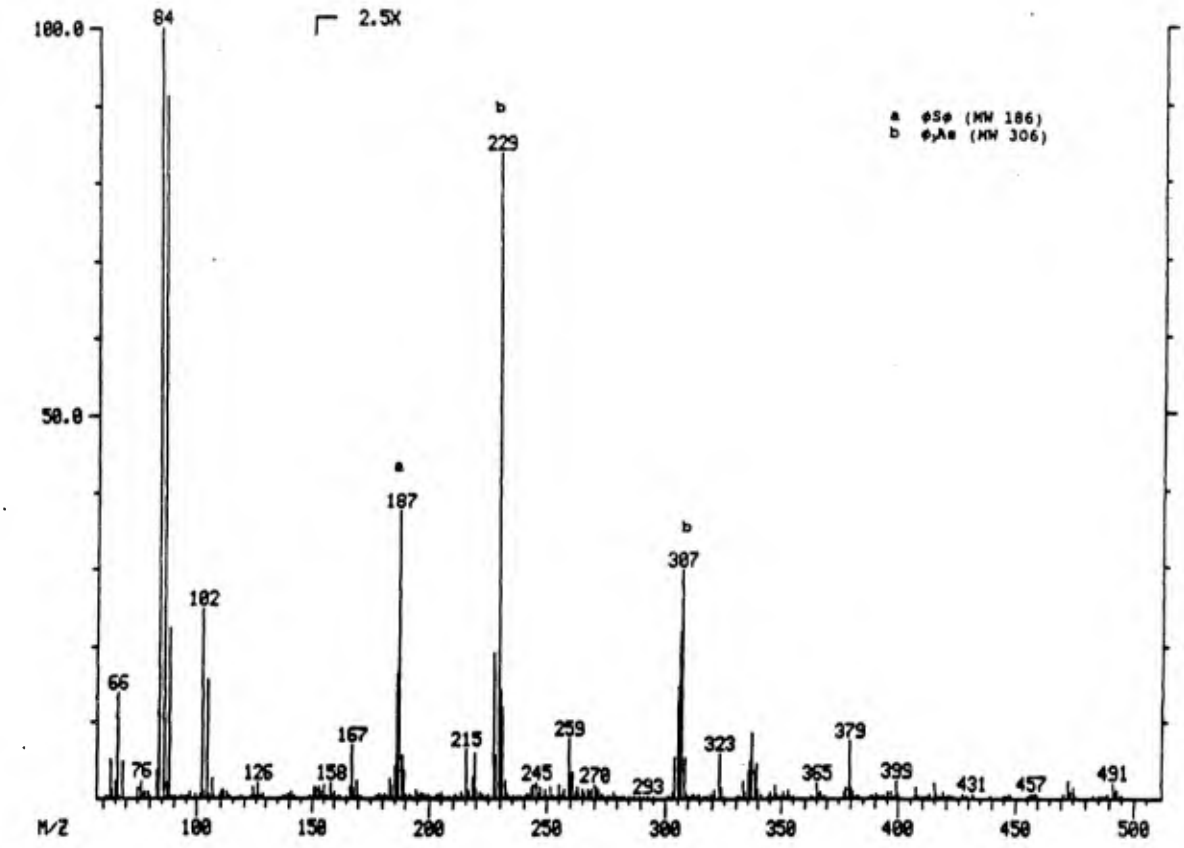
CI Mass Spectrum of OTH-293-9d Scan 1117



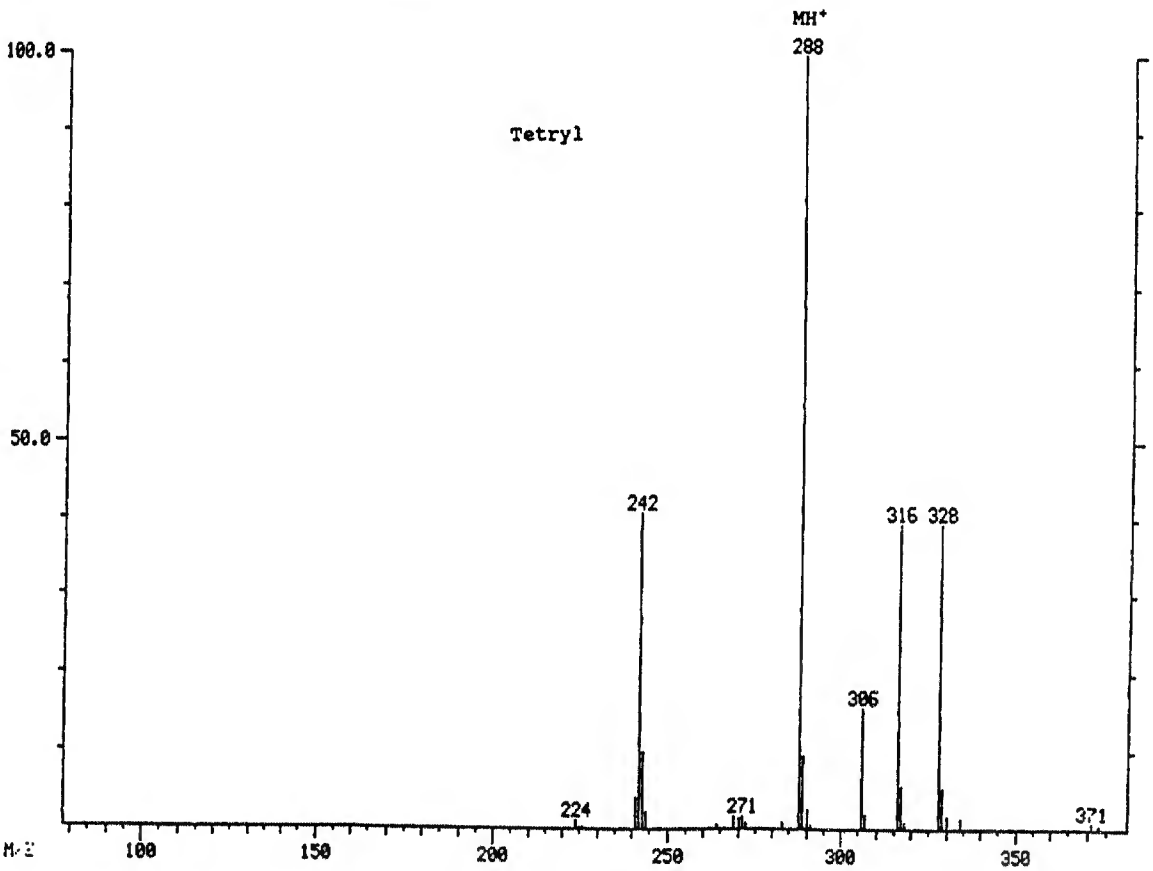
CI Mass Spectrum of OTH-293-9d Scan 1130



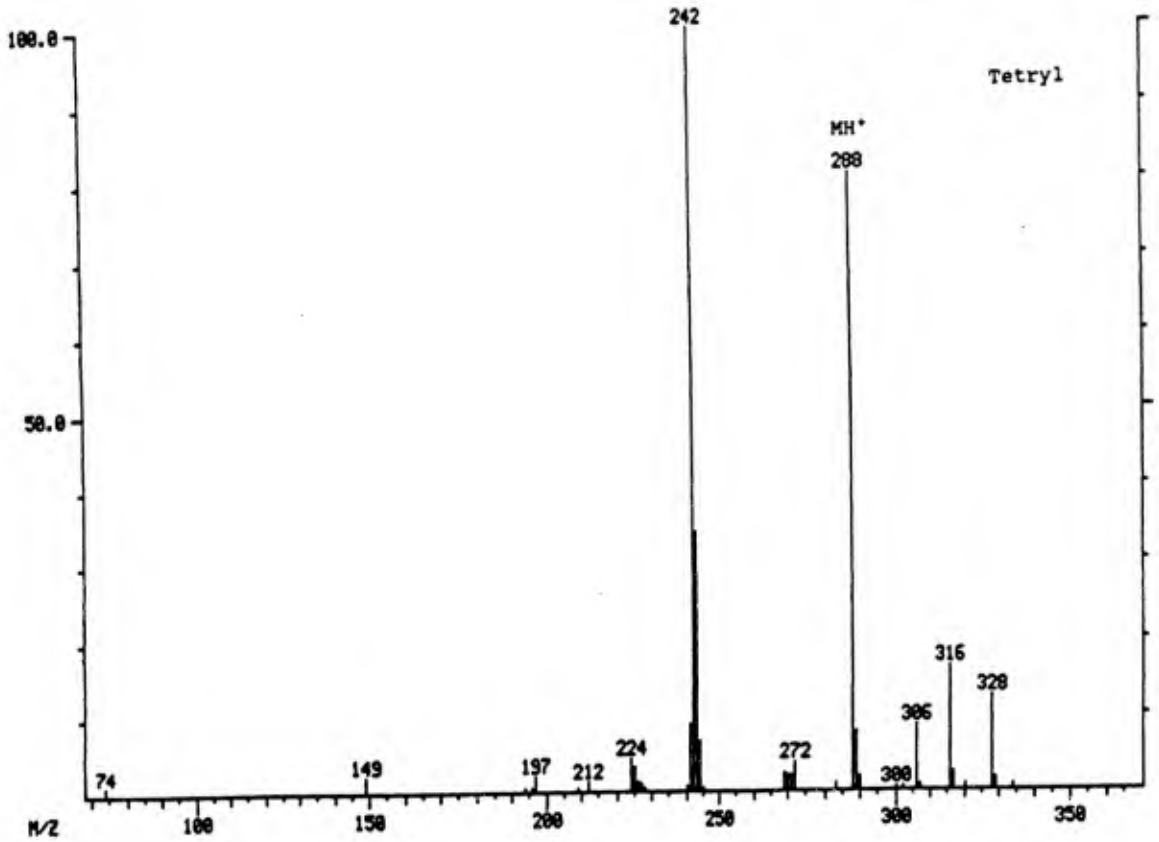
DEP/CI Mass Spectrum of OTH-293-9d (Pig #9)



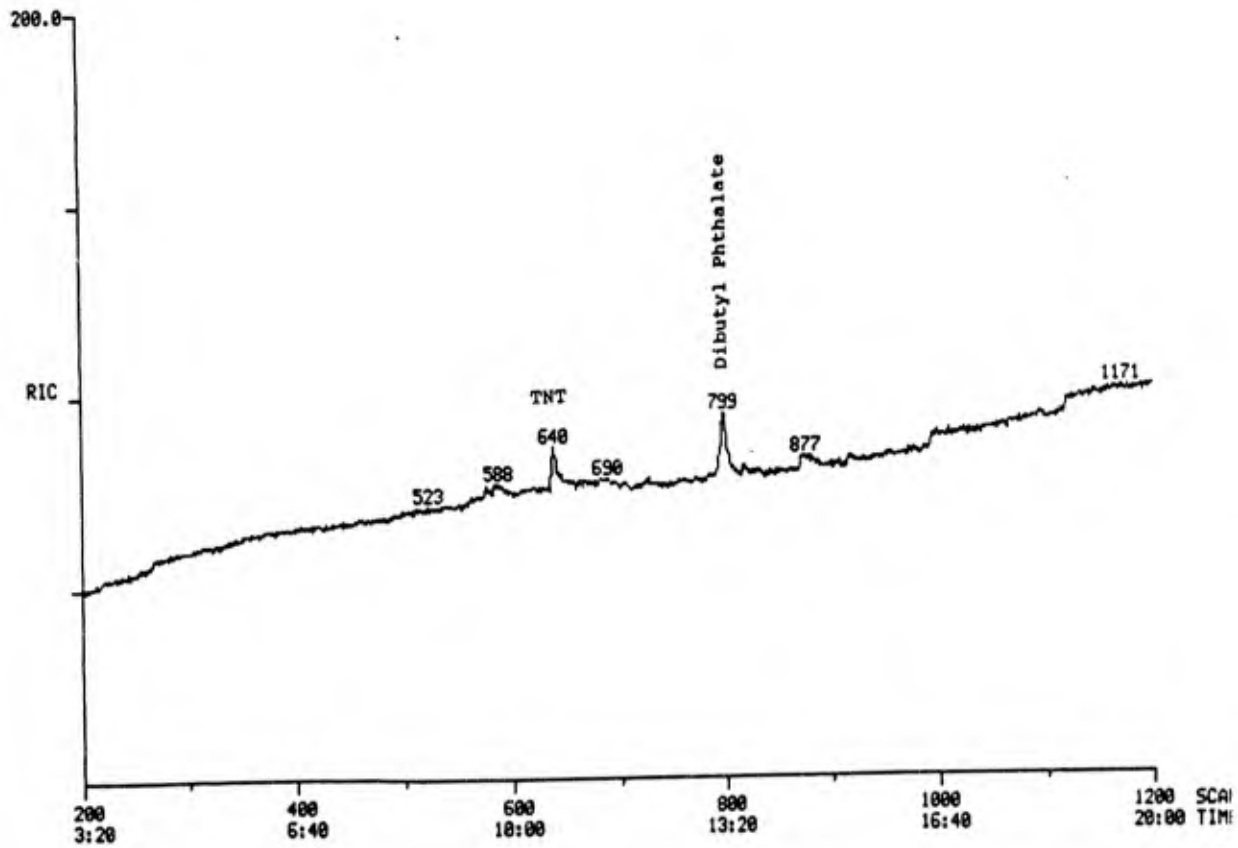
DEP/CI Mass Spectrum of OTH-293-10a (Pig #10)



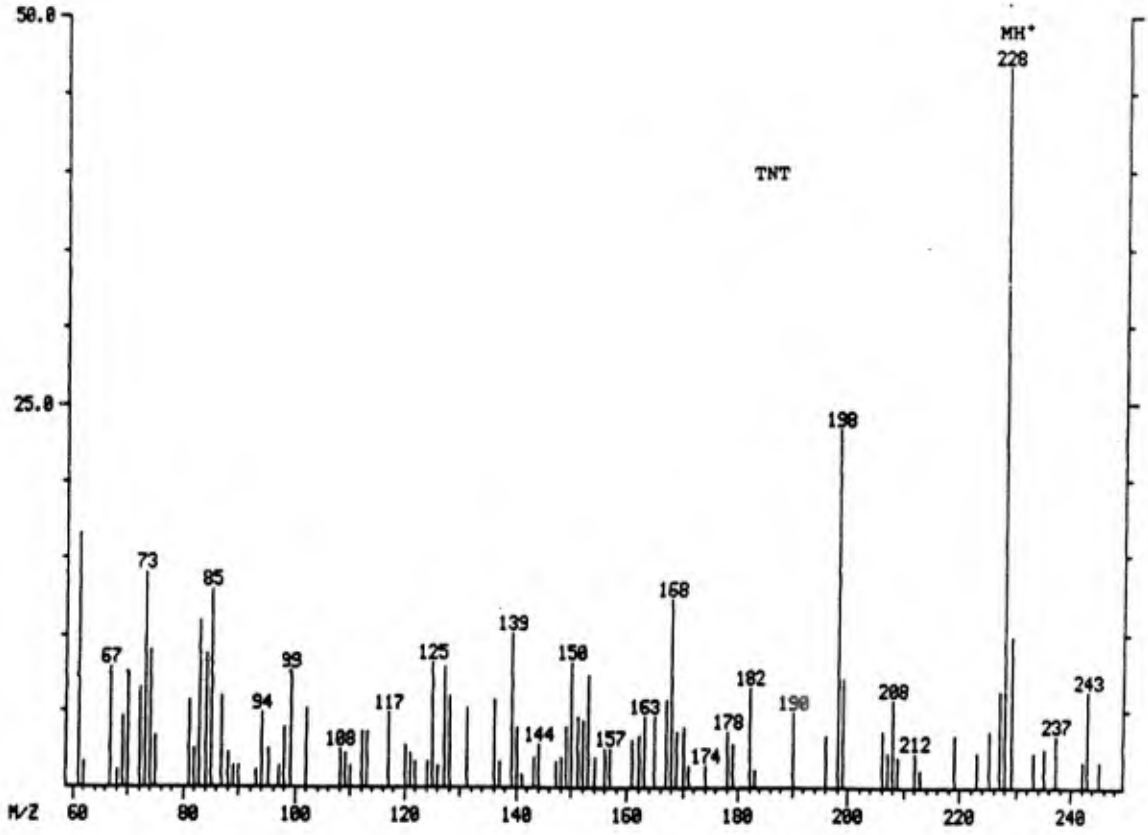
DEP/CI Mass Spectrum of OTH-293-10b (Pig #10)



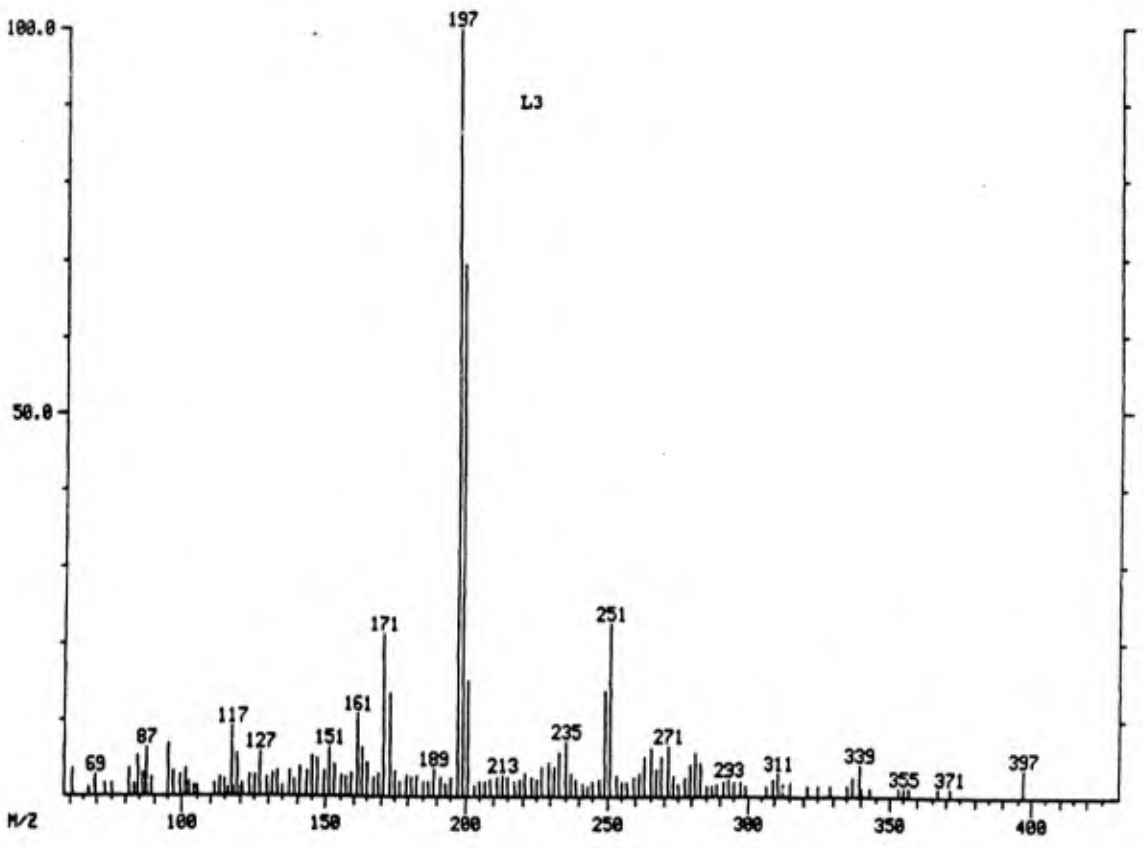
GC/MS/CI Chromatogram of OTH-293-10b (Pig #10)



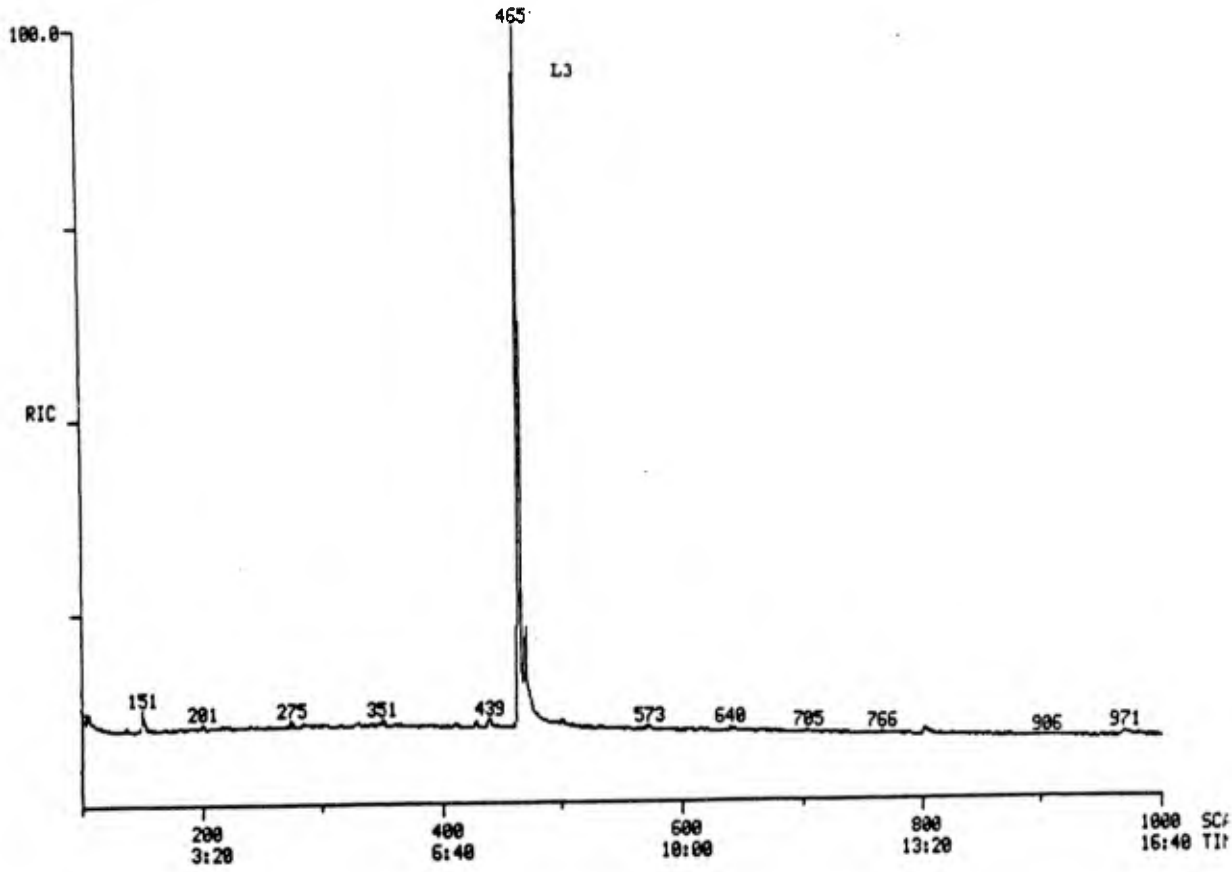
CI Mass Spectrum of OTH-293-10b Scan 640



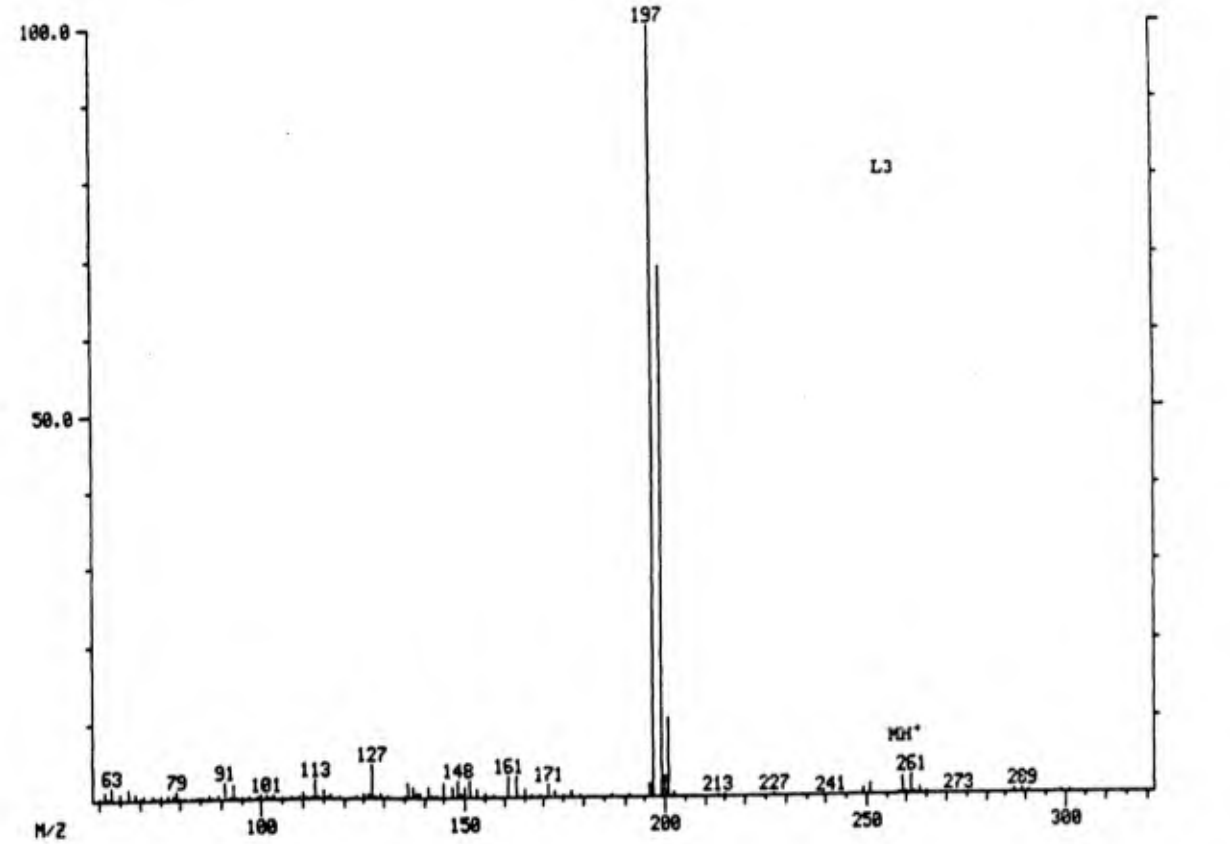
DEP/CI Mass Spectrum of OTH-293-10c (Pig #10)



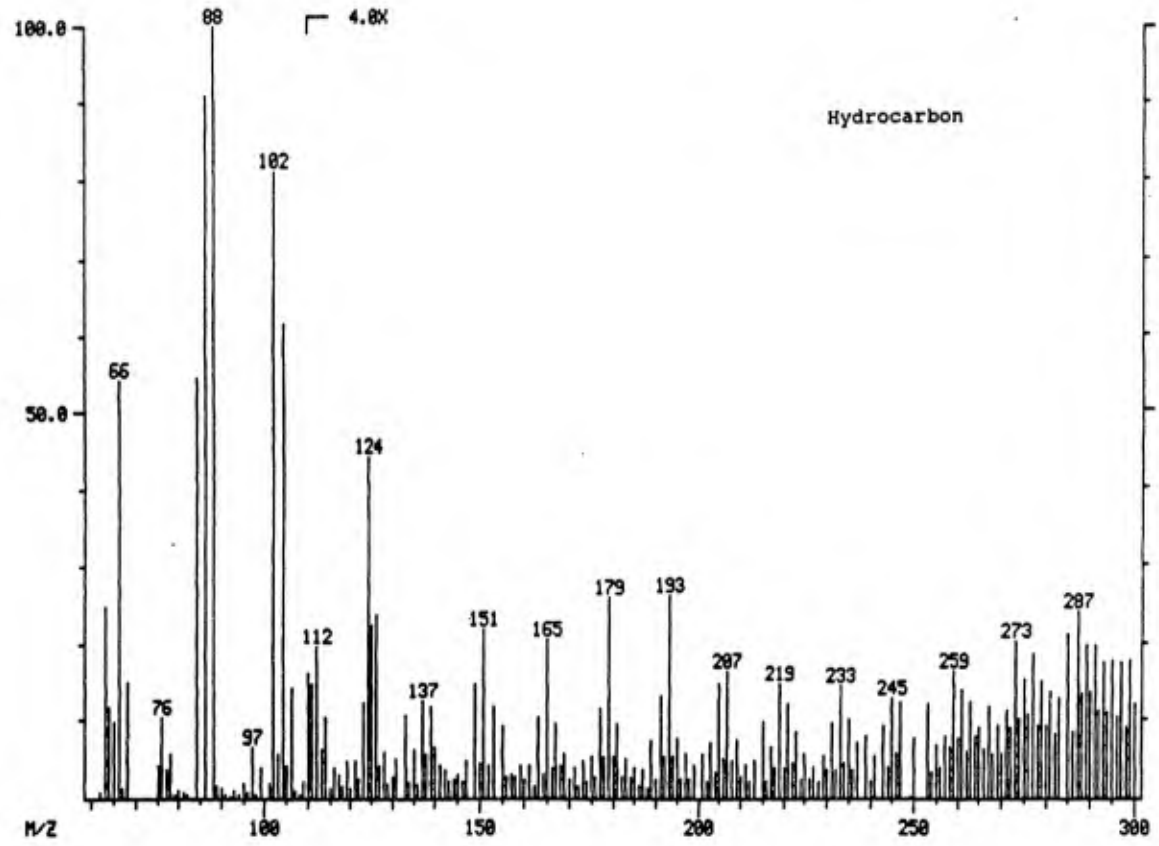
GC/MS/CI Chromatogram of OTH-293-10c (Pig #10)



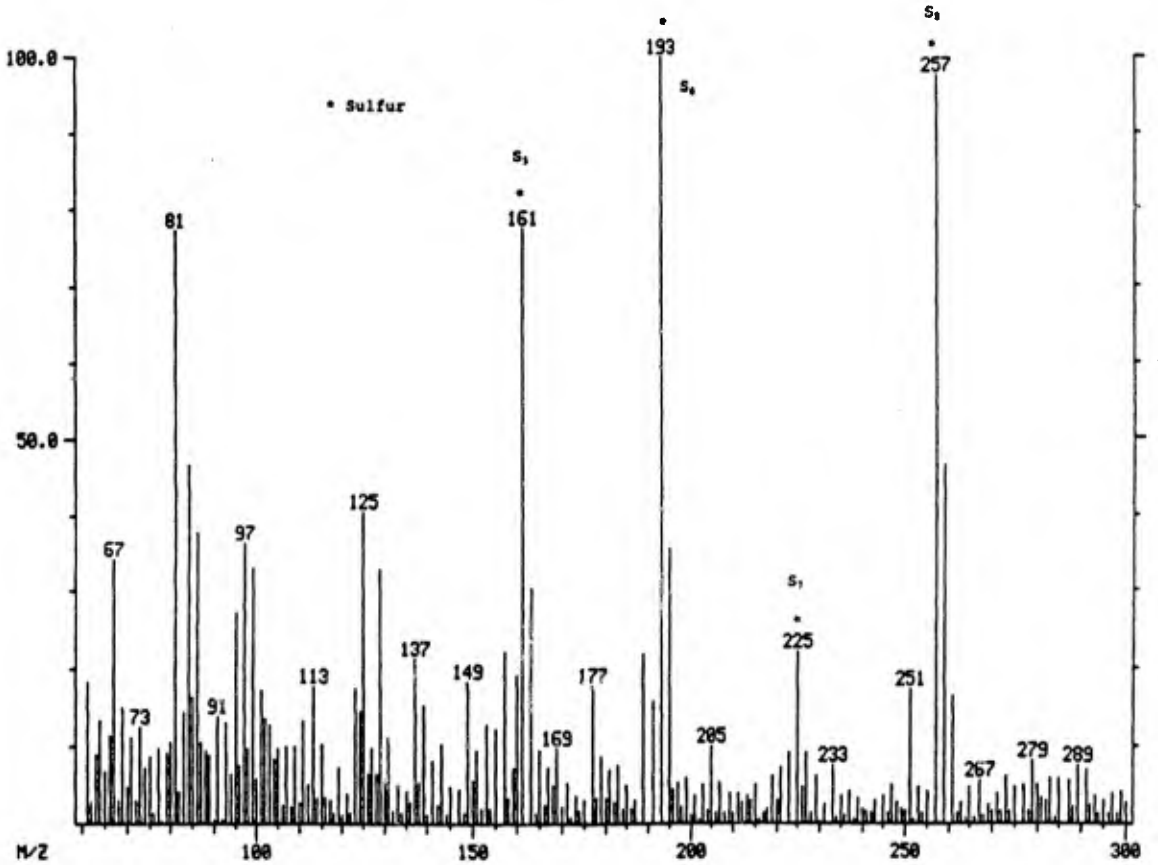
CI Mass Spectrum of OTH-293-10c Scan 465



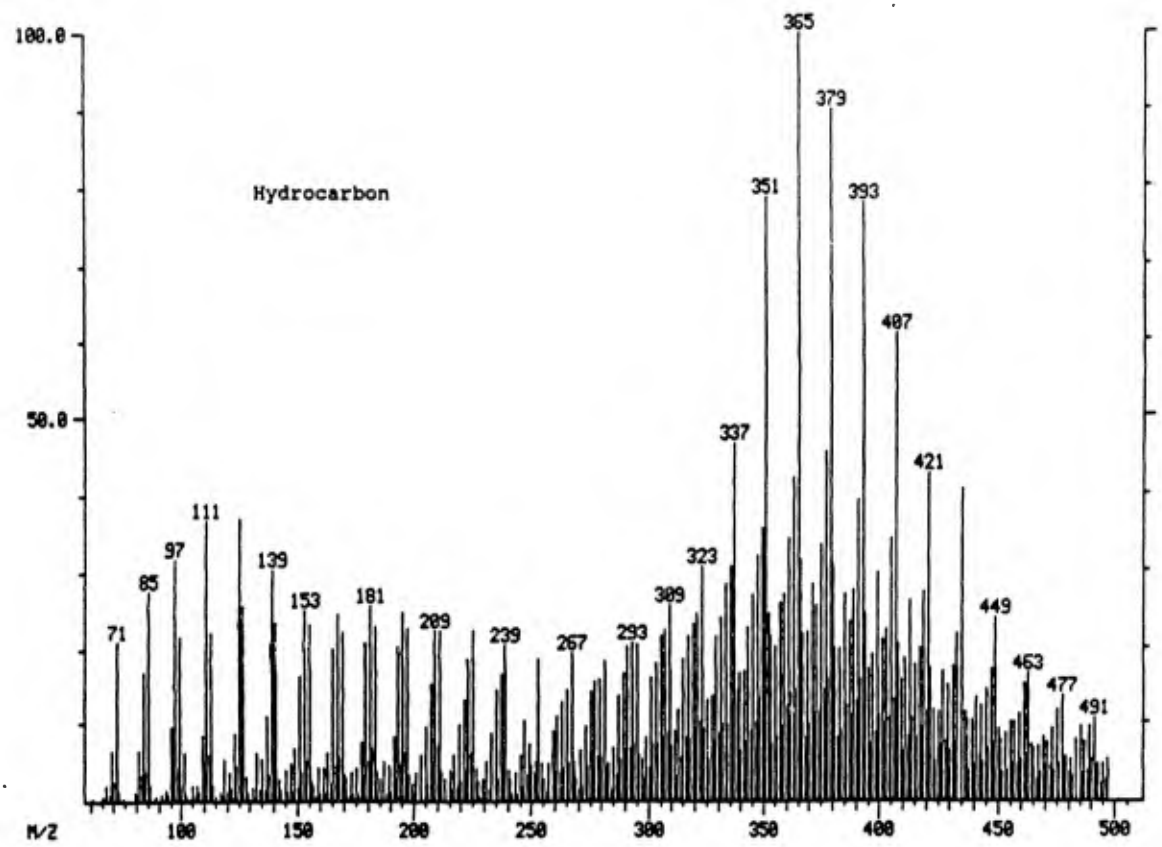
DEP/CI Mass Spectrum of OTH-993-4 (Pig #10)



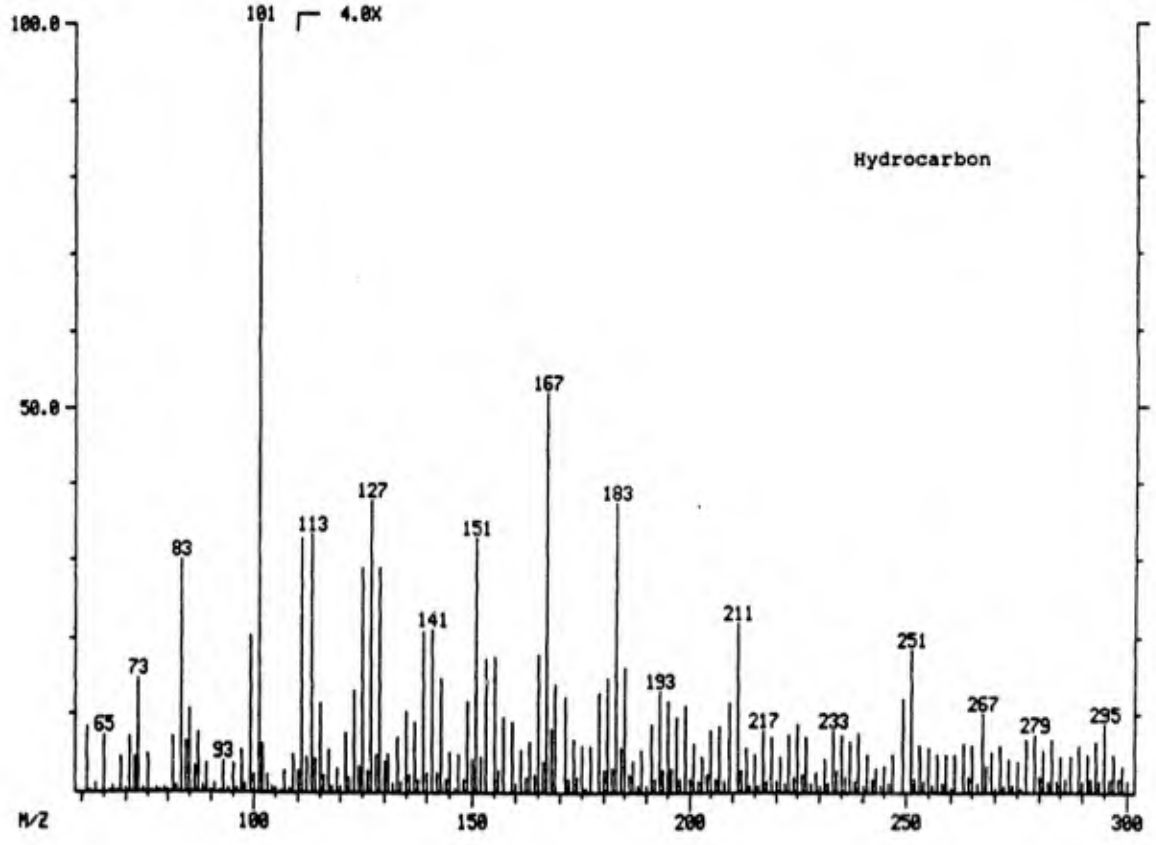
DEP/CI Mass Spectrum of OTH-993-5 (Pig #10)



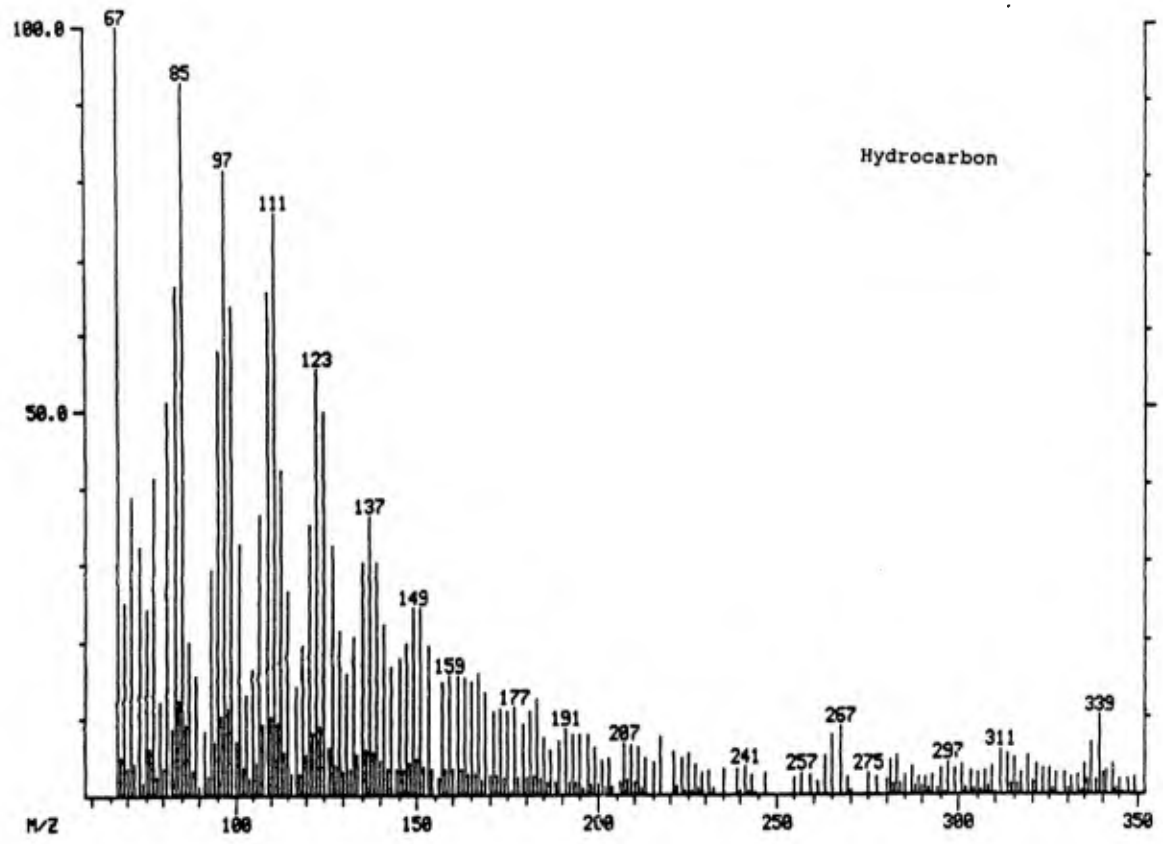
DEP/CI Mass Spectrum of OTH-1093-1d (Pig #11)



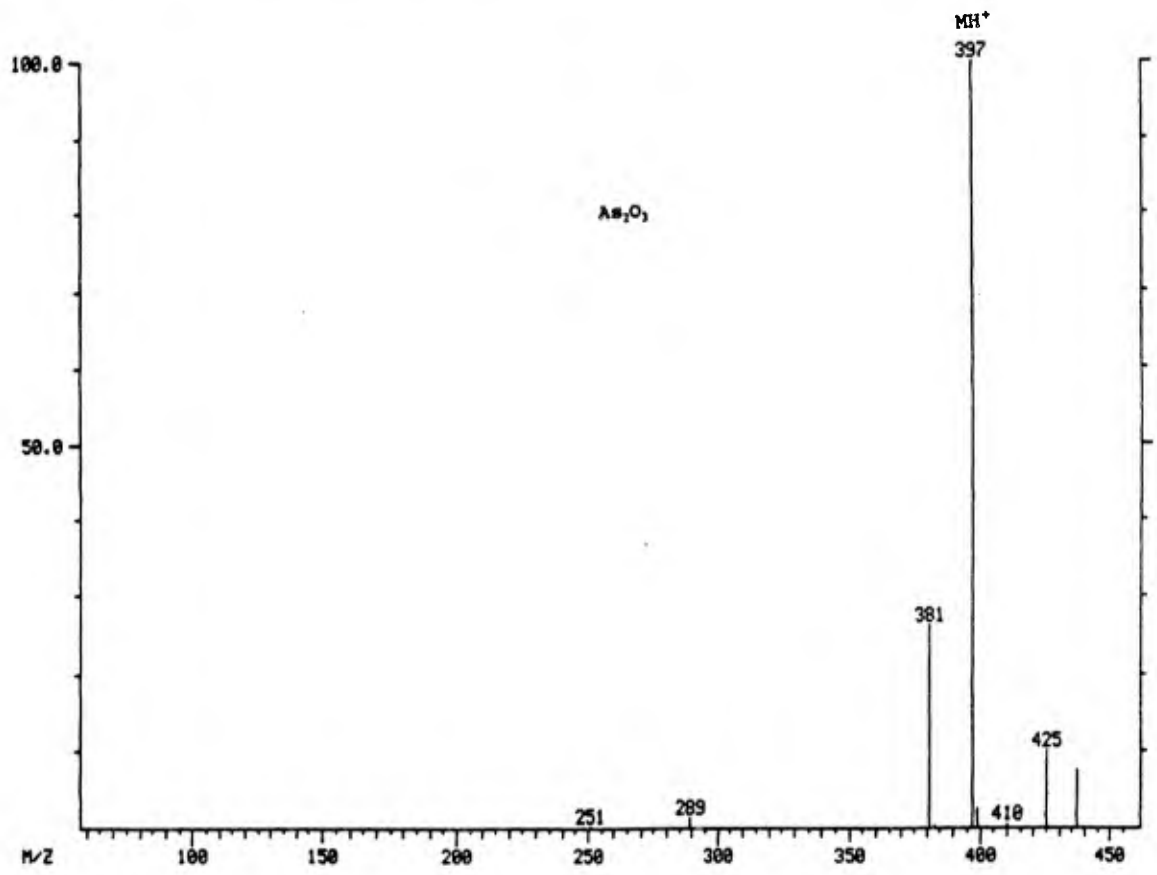
DEP/CI Mass Spectrum of OTH-1093-3c (Pig #13)



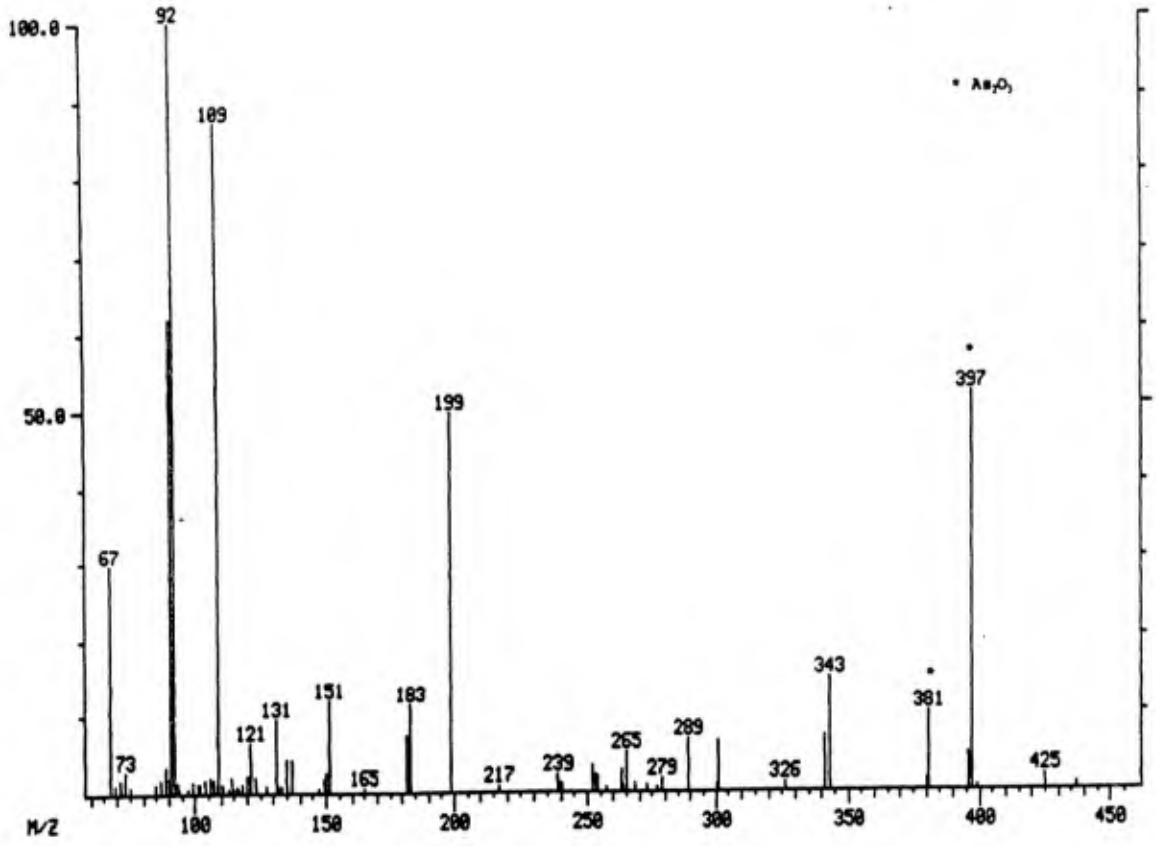
DEP/CI Mass Spectrum of OTH-1093-4g (Pig #14)



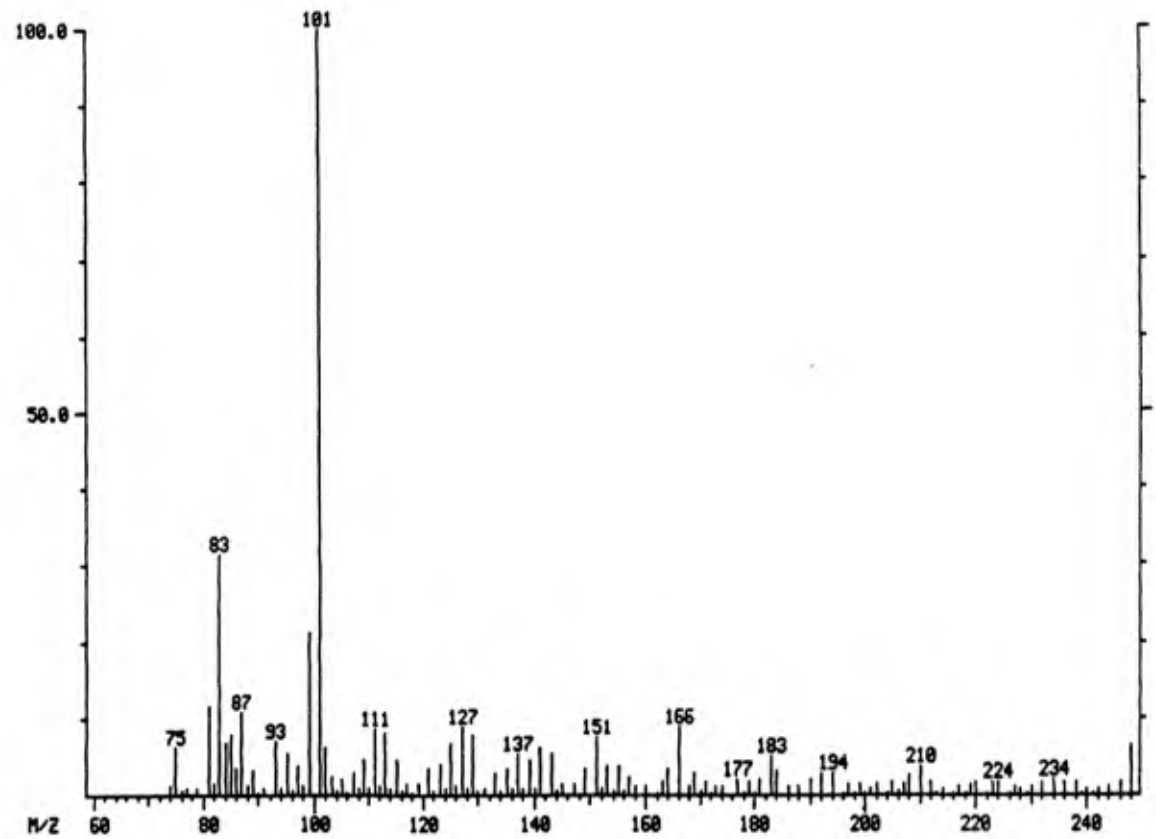
DEP/CI Mass Spectrum of Arsenic Trioxide Reference Standard



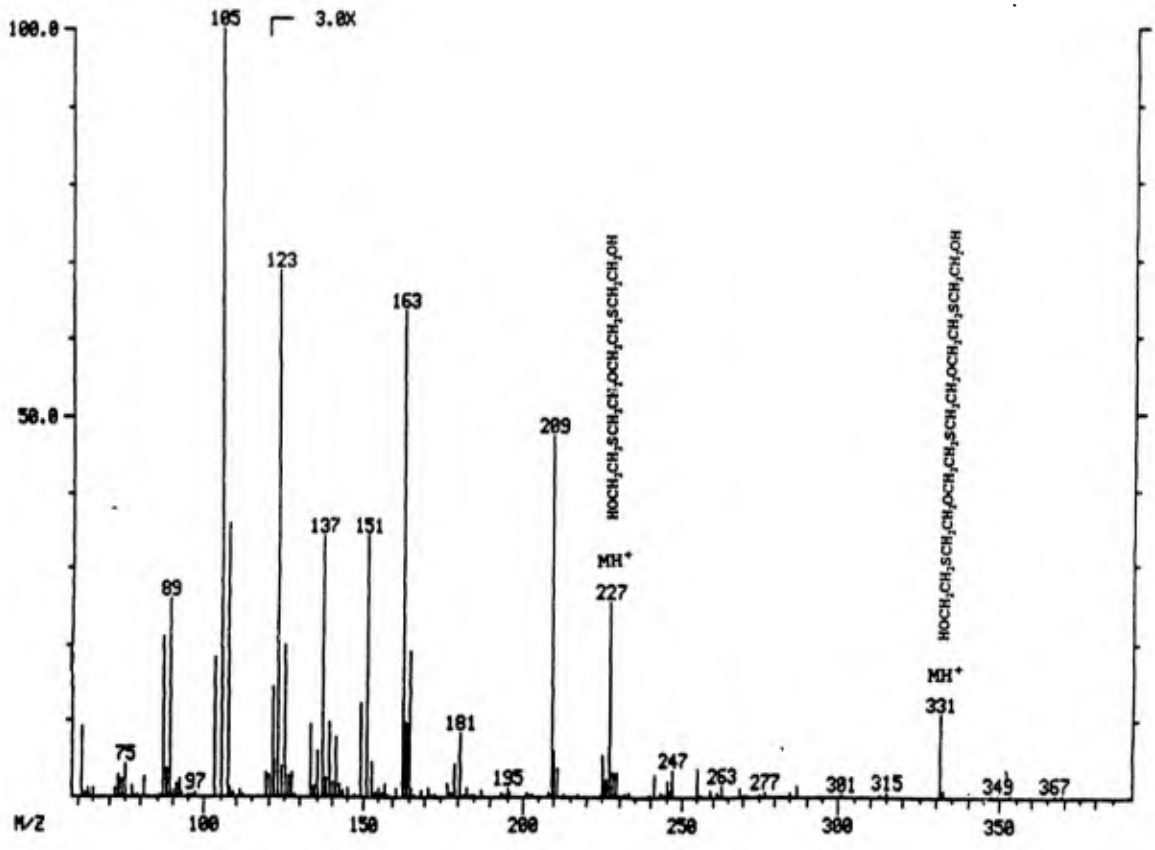
DEP/CI Mass Spectrum of OTH-1193-2c (Pig #18)



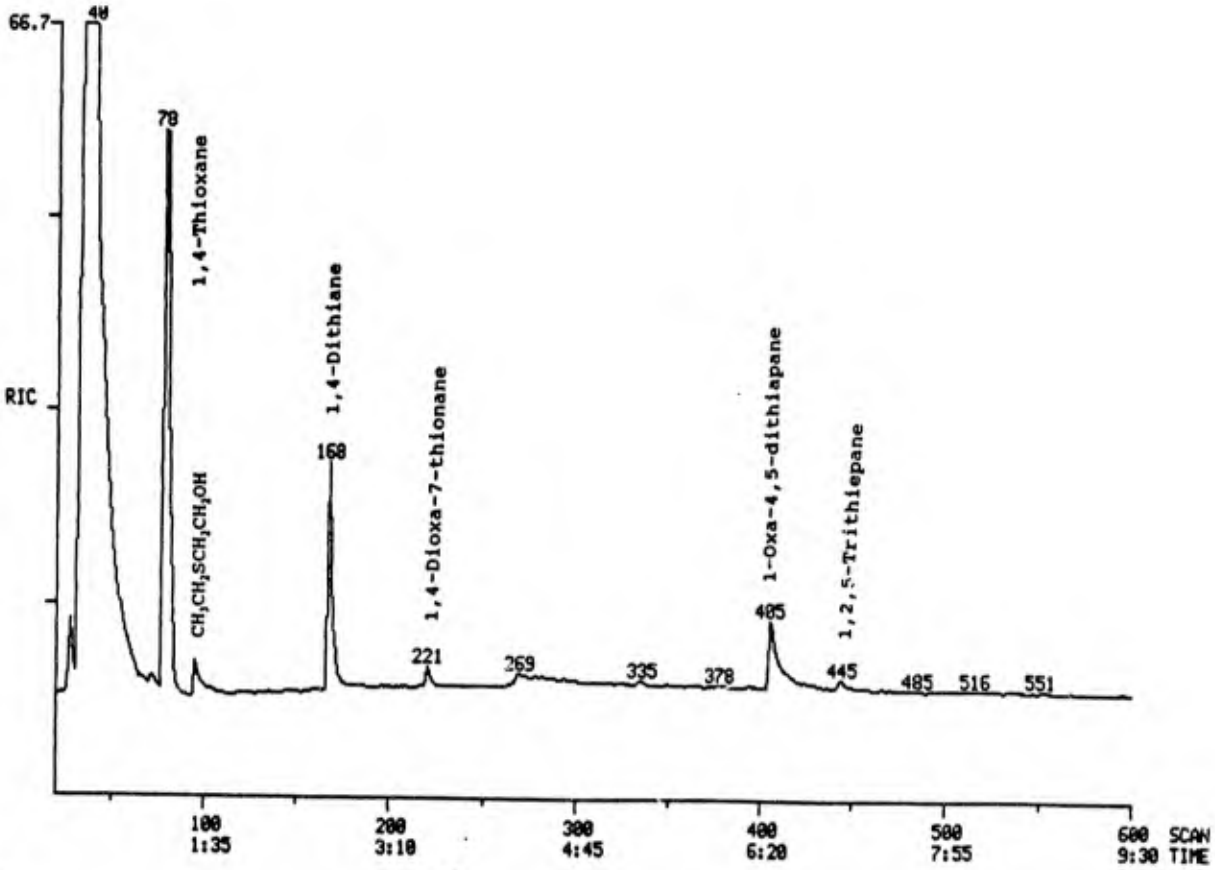
DEP/CI Mass Spectrum of OTH-1193-2e (Pig #18)



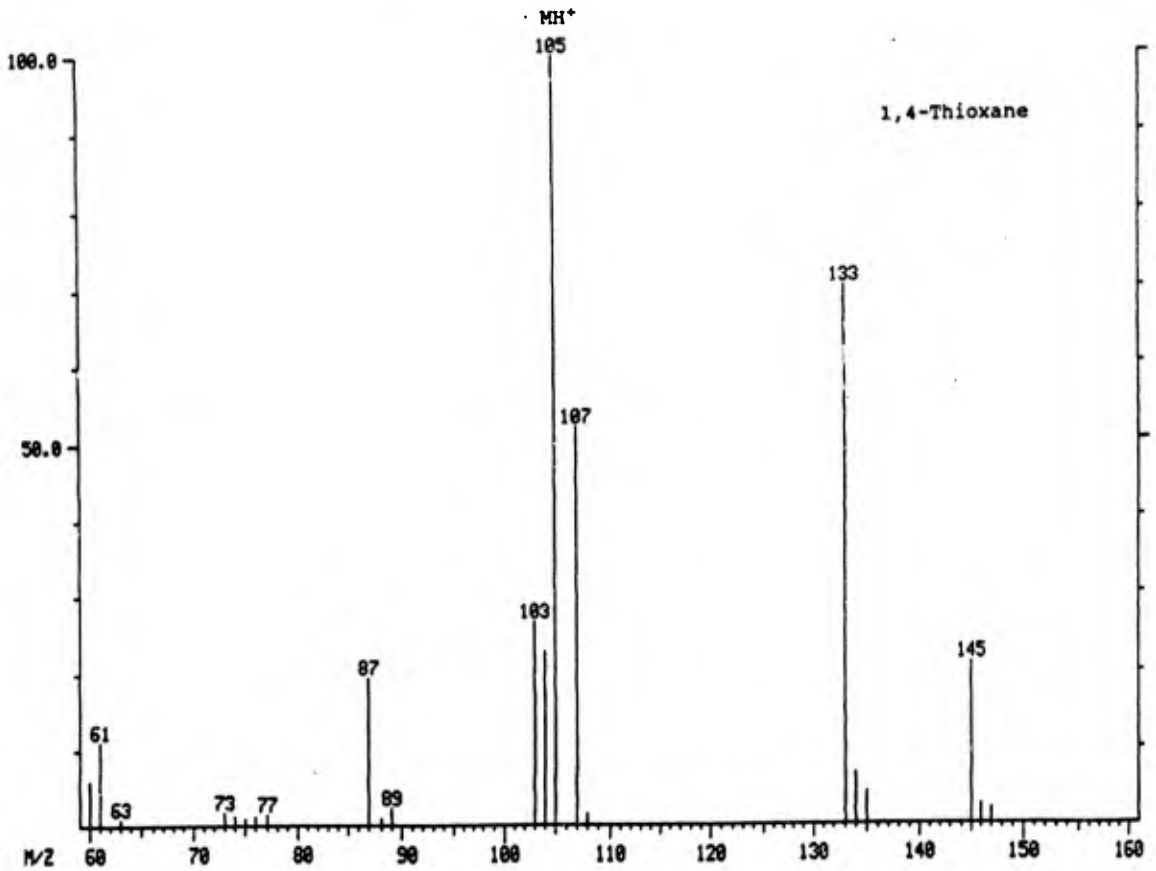
DEP/CI Mass Spectrum of OTH-1193-1 (Pig #21)



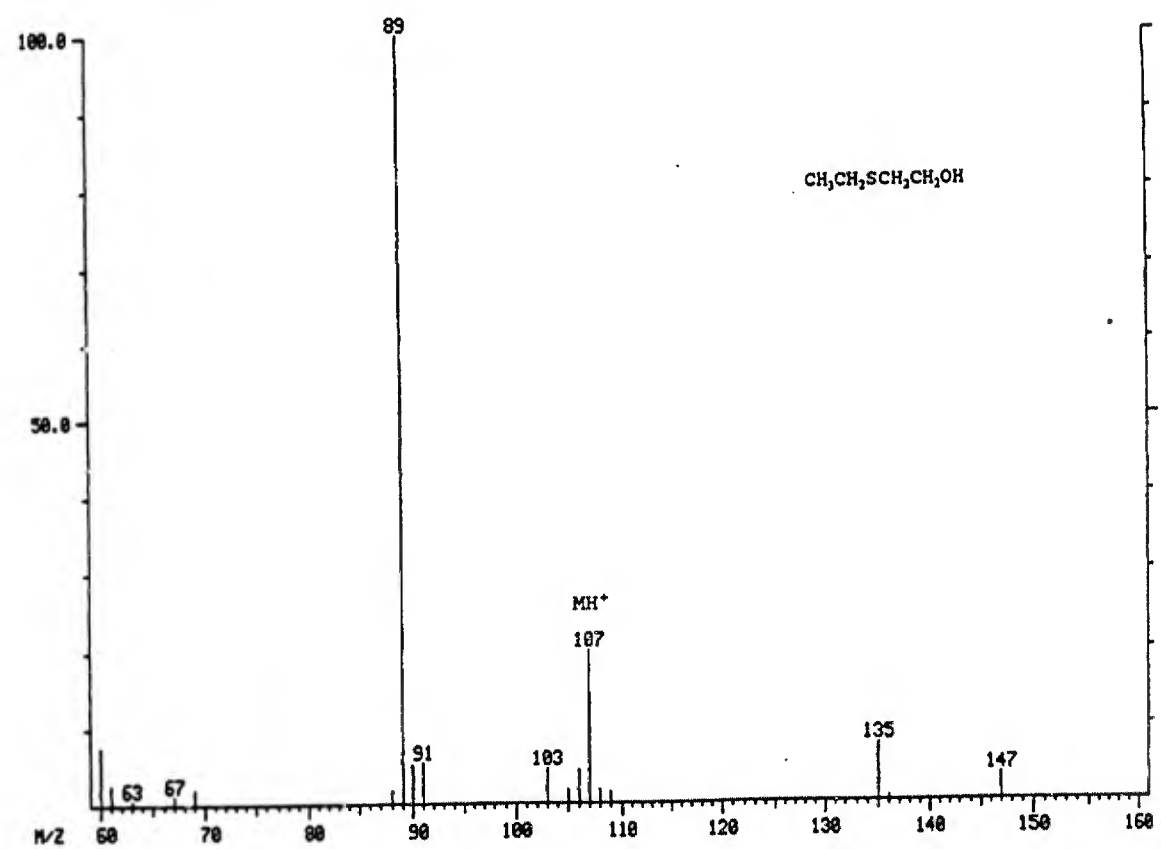
GC/MS/CI Chromatogram of OTH-1193-1 (Pig #21)



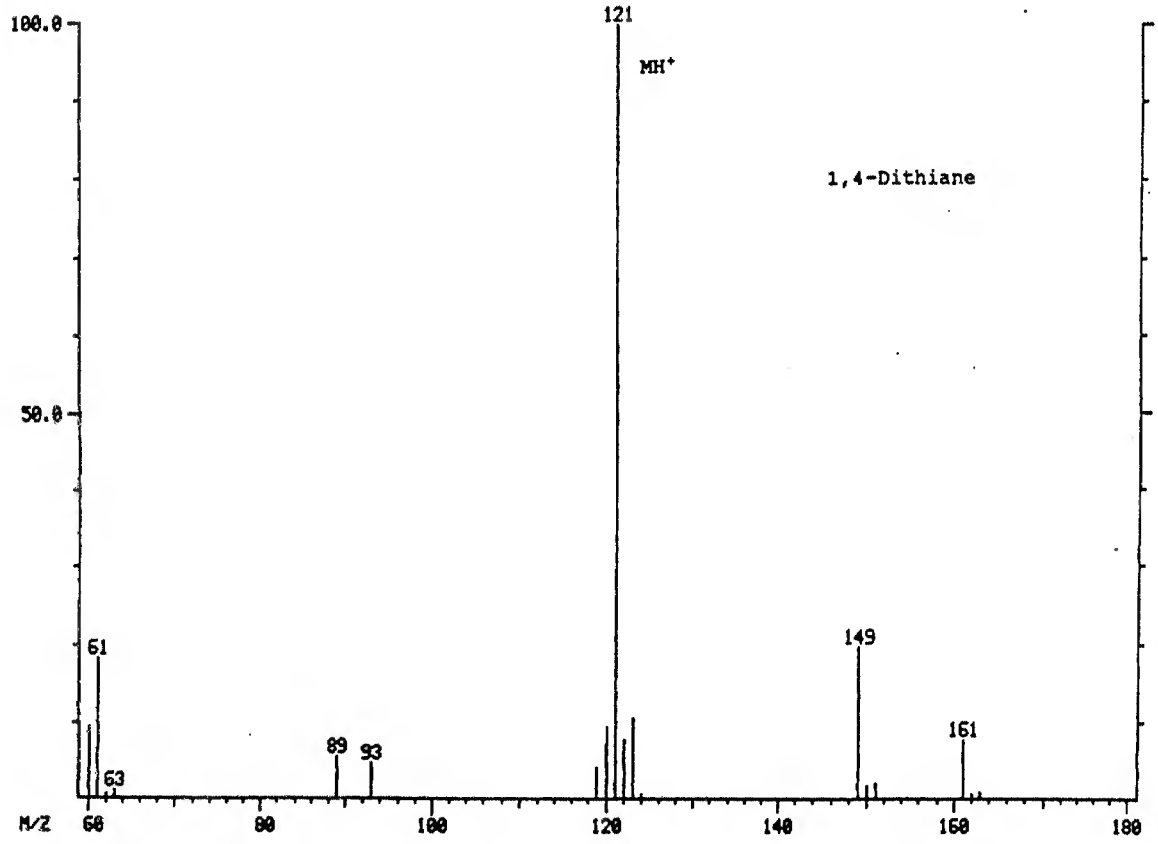
CI Mass Spectrum of OTH-1193-1 Scan 78



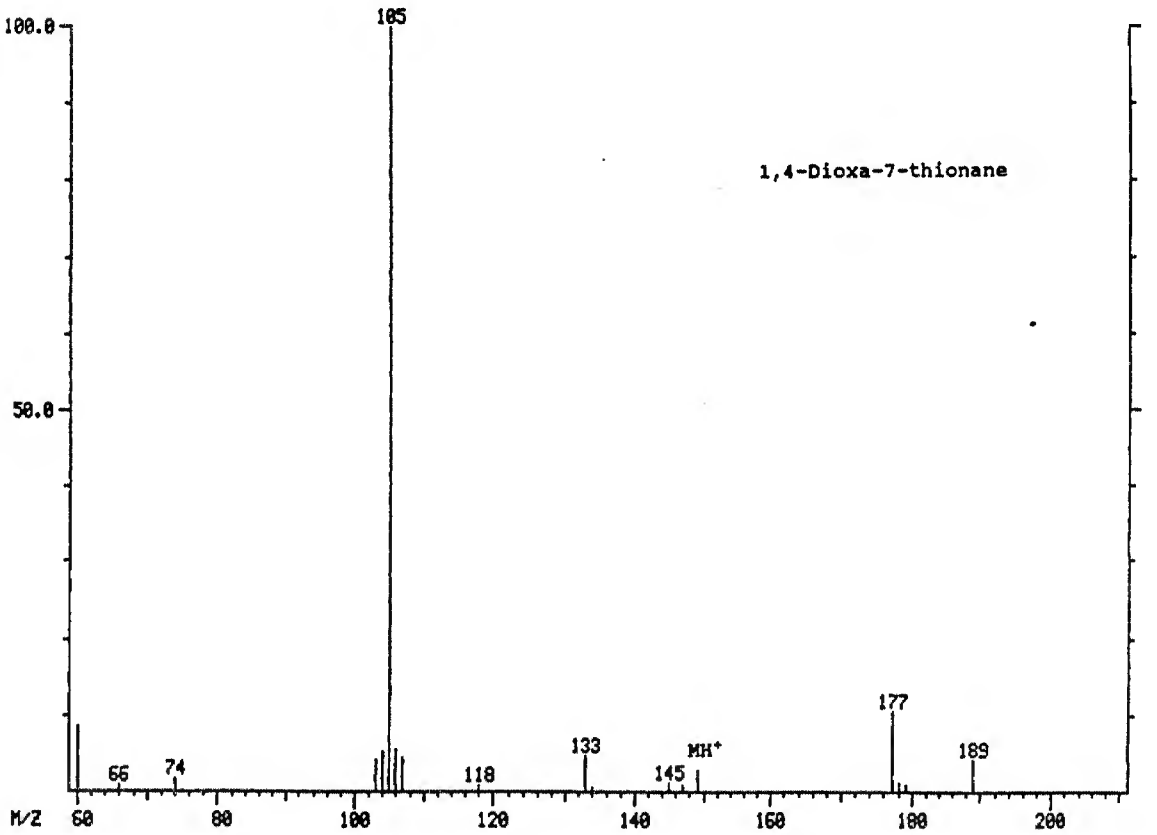
CI Mass Spectrum of OTH-1193-1 Scan 95



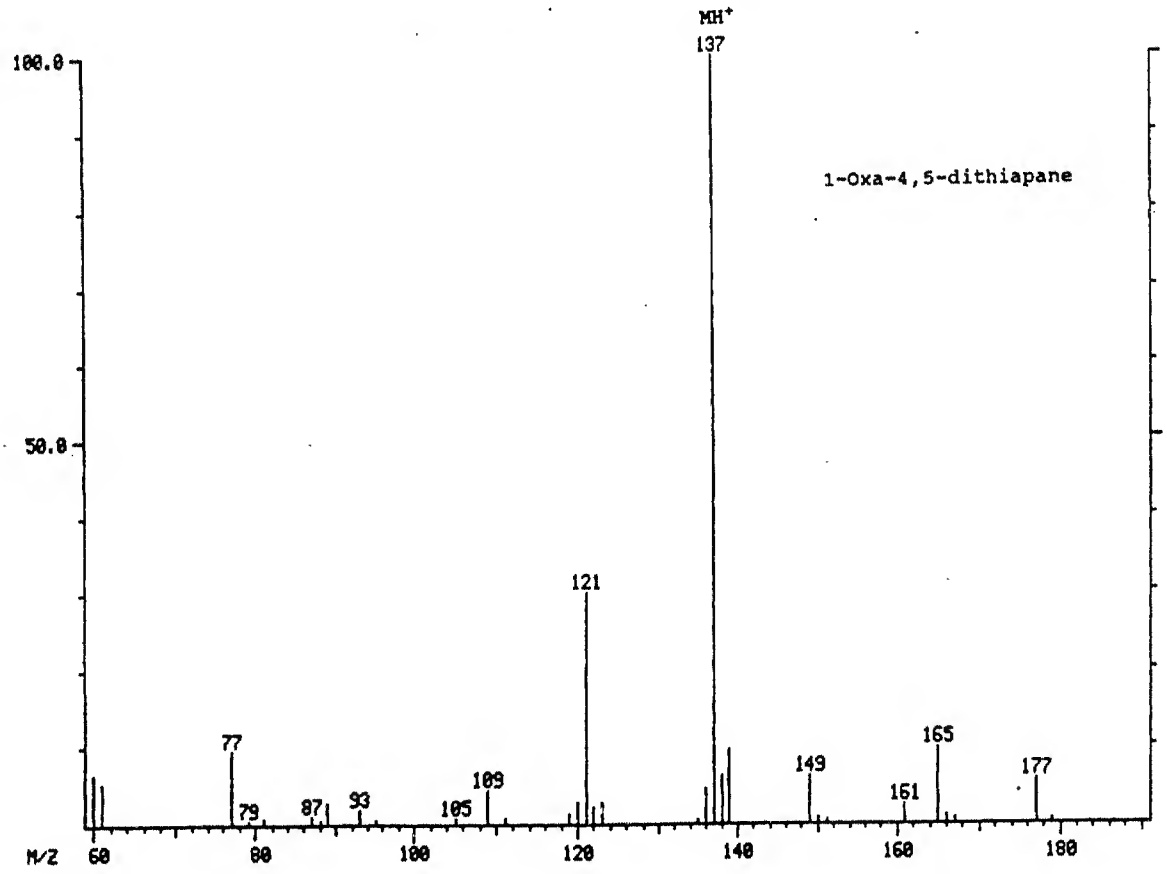
CI Mass Spectrum of OTH-1193-1 Scan 168



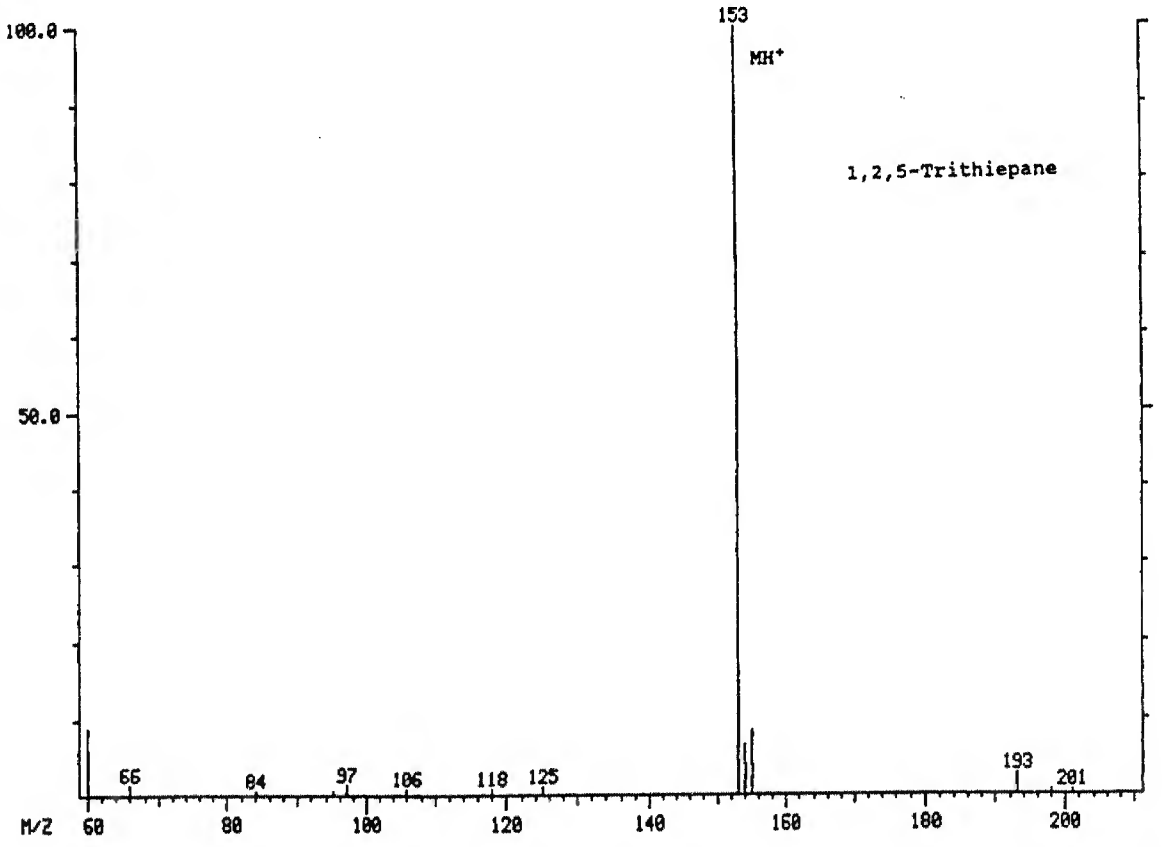
CI Mass Spectrum of OTH-1193-1 Scan 221



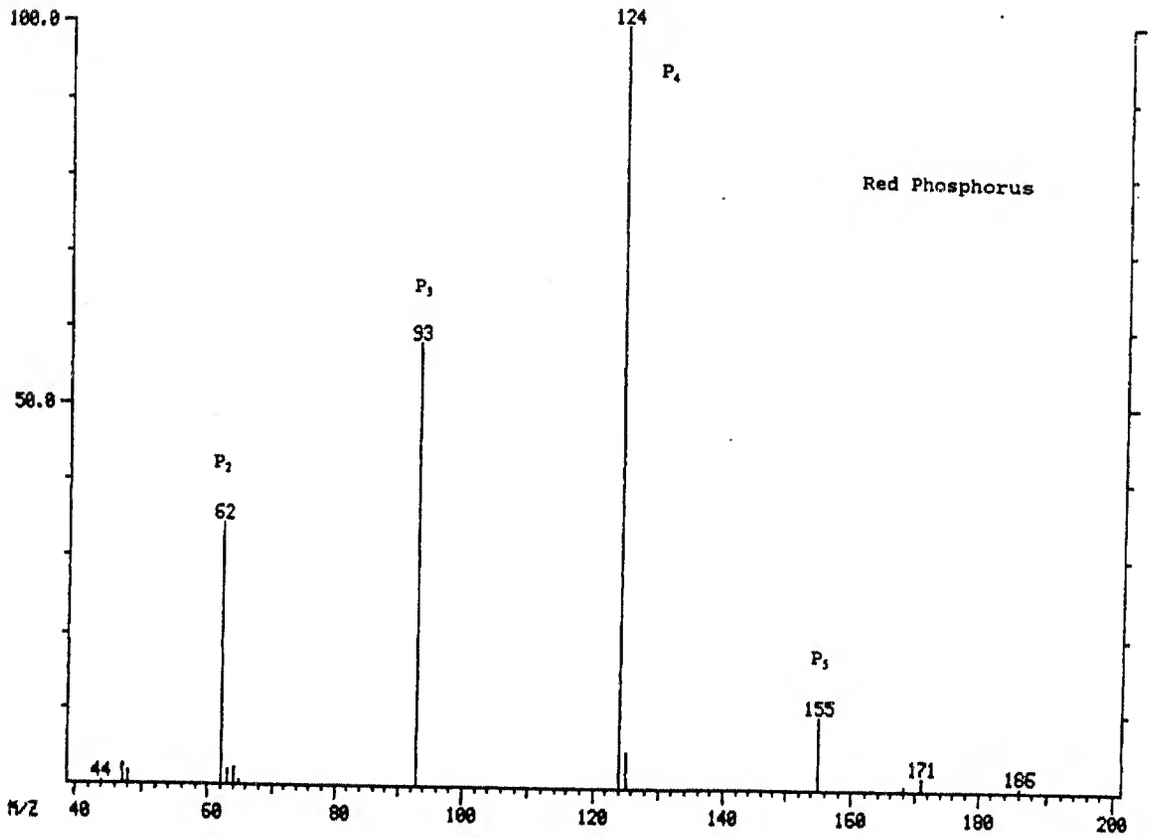
CI Mass Spectrum of OTH-1193-1 Scan 405



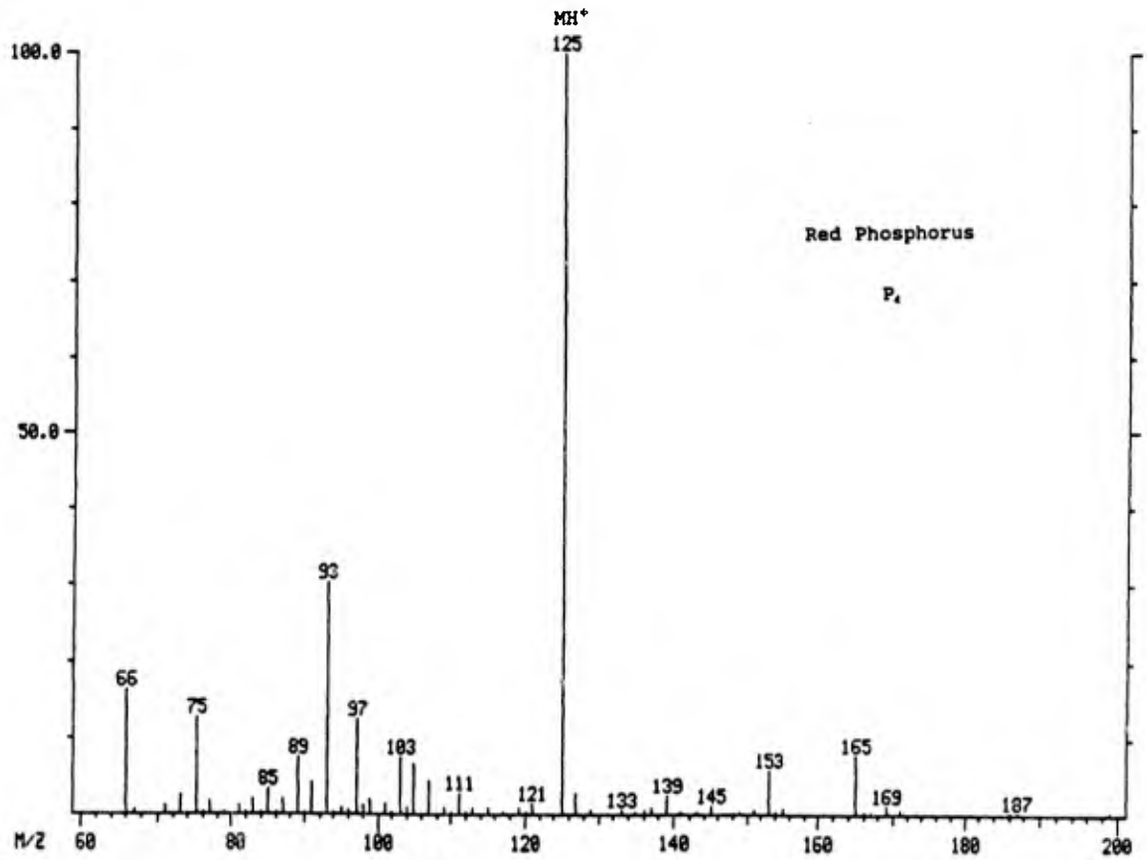
CI Mass Spectrum of OTH-1193-1 Scan 445



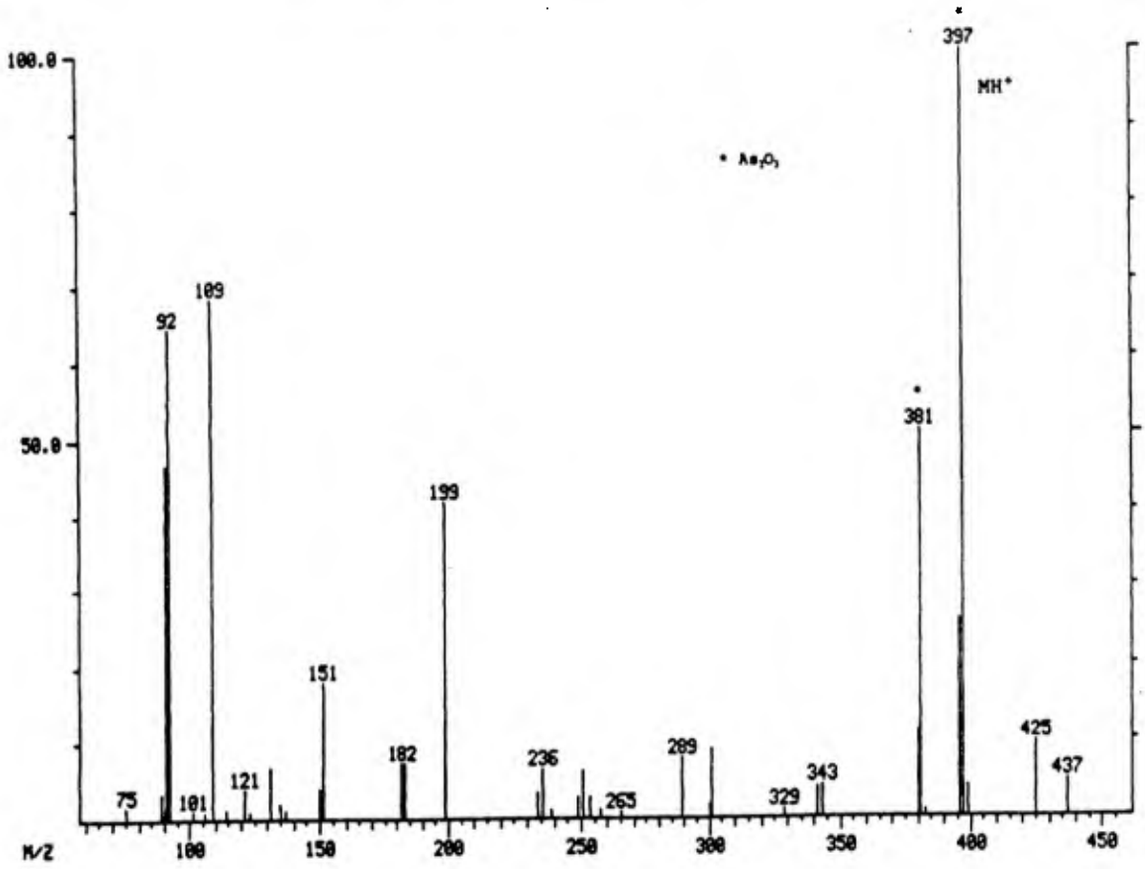
DEP/EI Mass Spectrum of OTH-1193-5a (Pig #22)



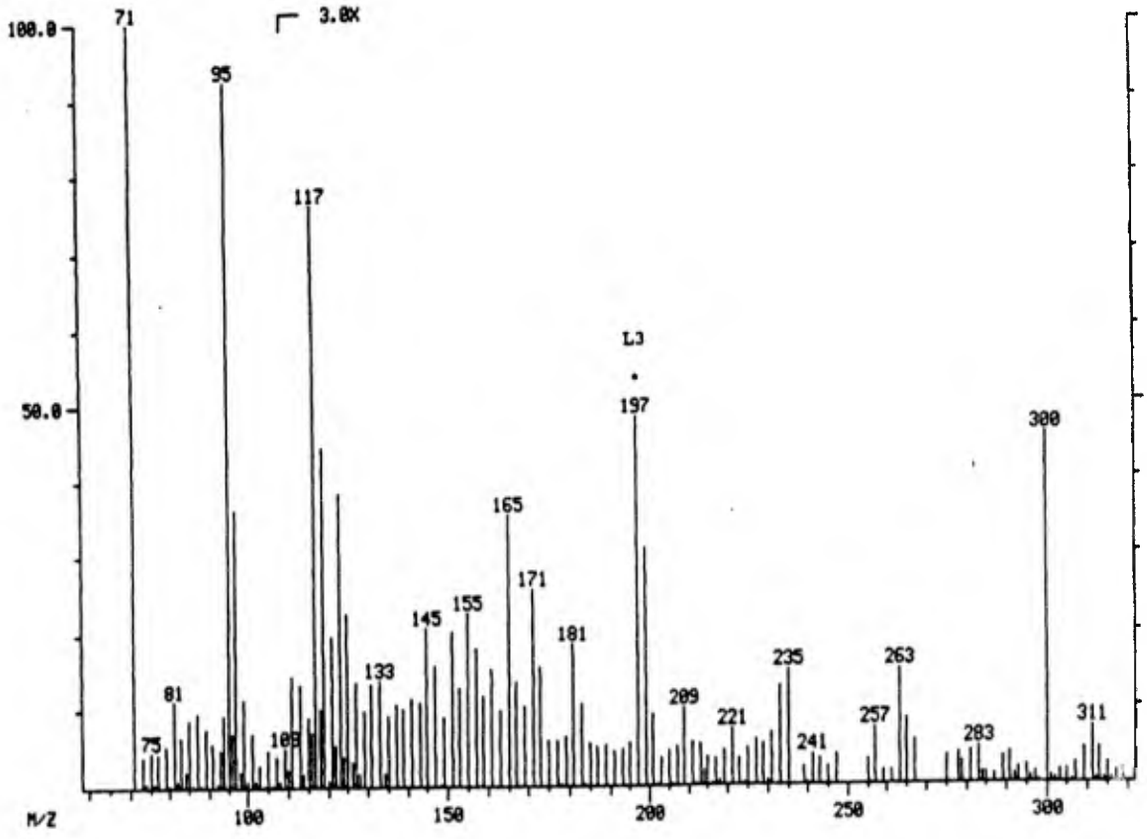
DEP/CI Mass Spectrum of OTH-1193-5a (Pig #22)



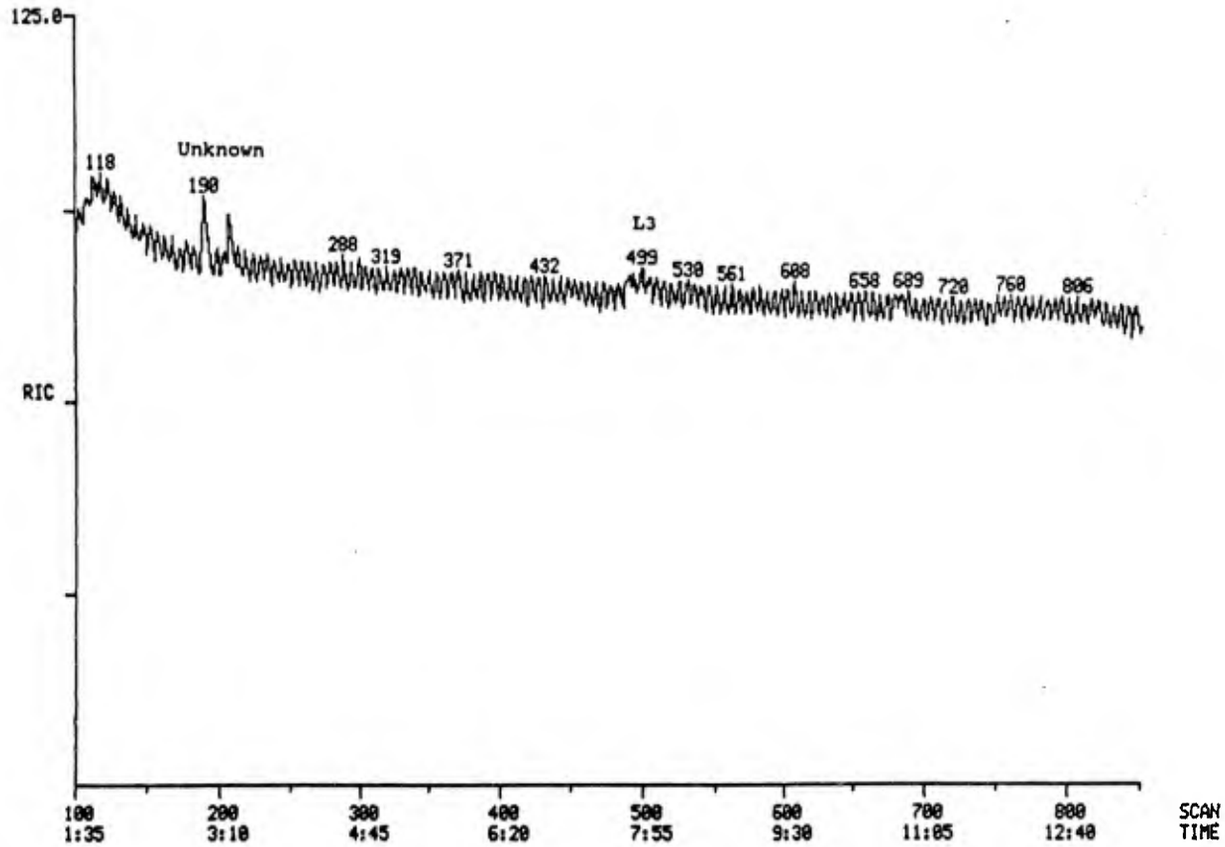
DEP/CI Mass Spectrum of OTH-1193-5b (Pig #22)



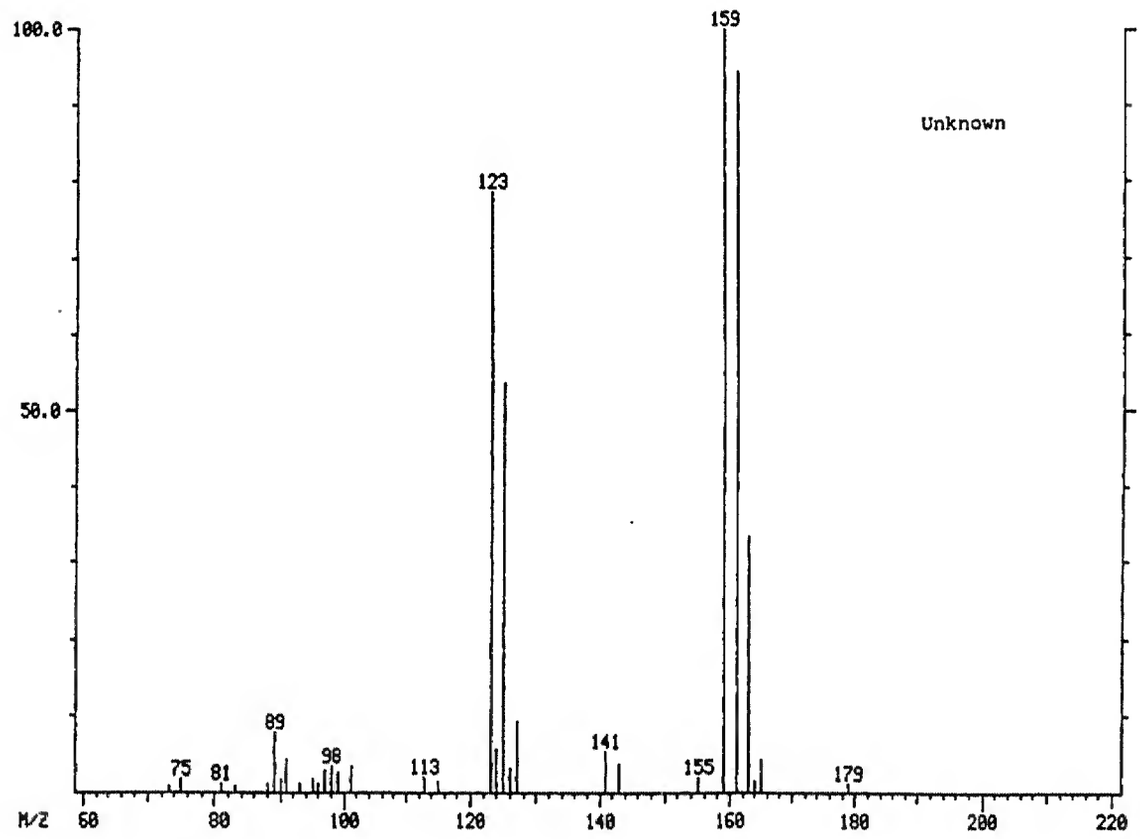
DEP/CI Mass Spectrum of OTH-1193-5c (Pig #22)



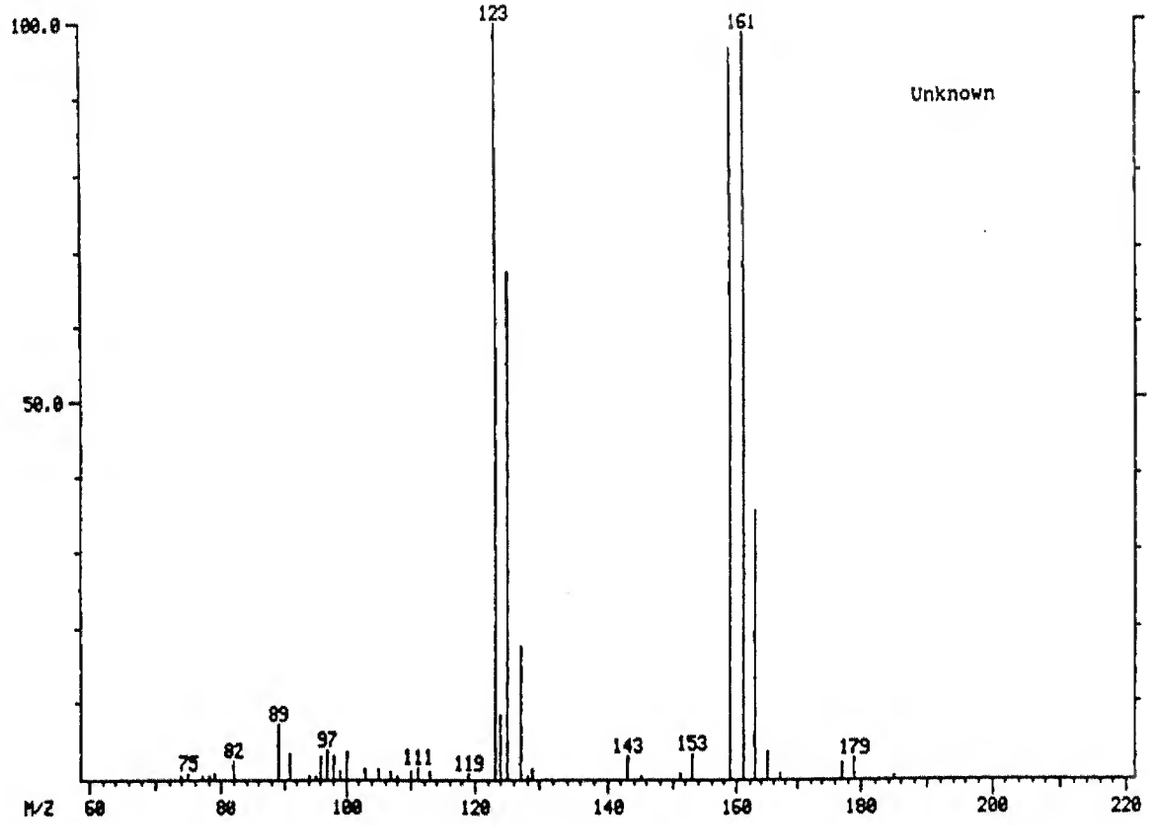
GC/MS/CI Chromatogram of OTH-1193-5c (Pig #22)



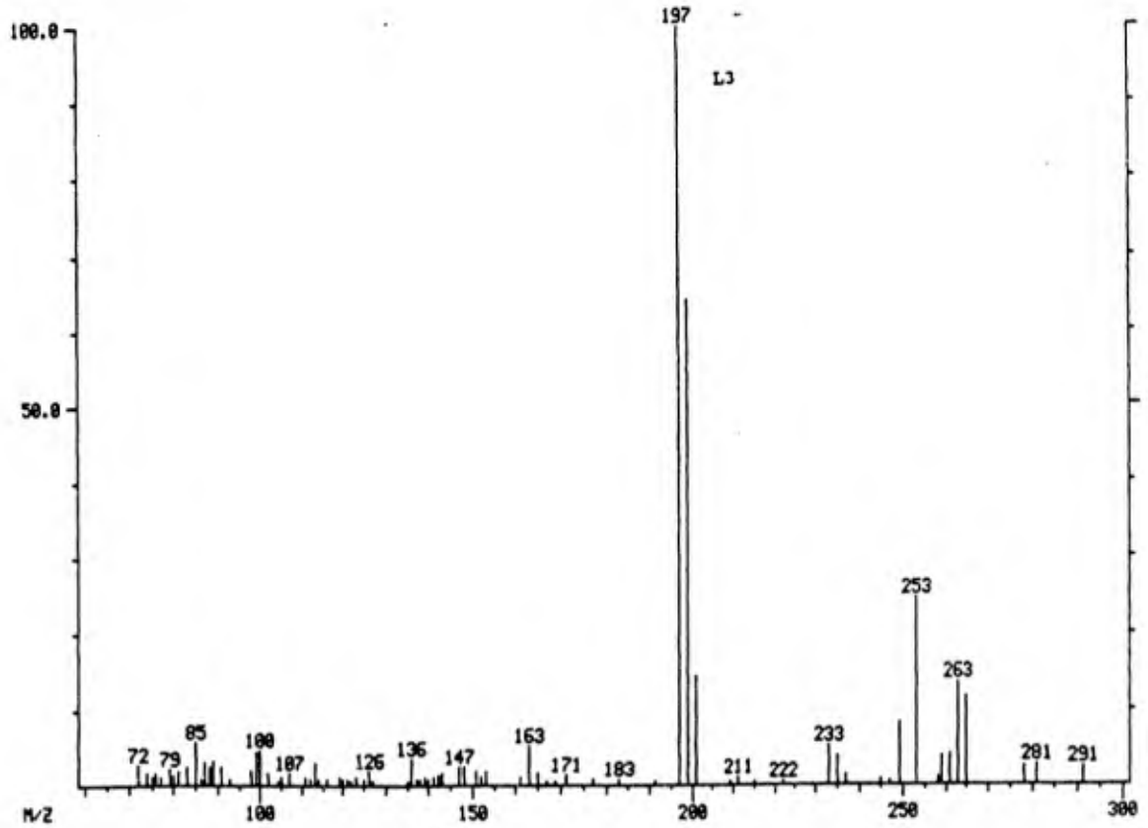
CI Mass Spectrum of OTH-1193-5c Scan 190



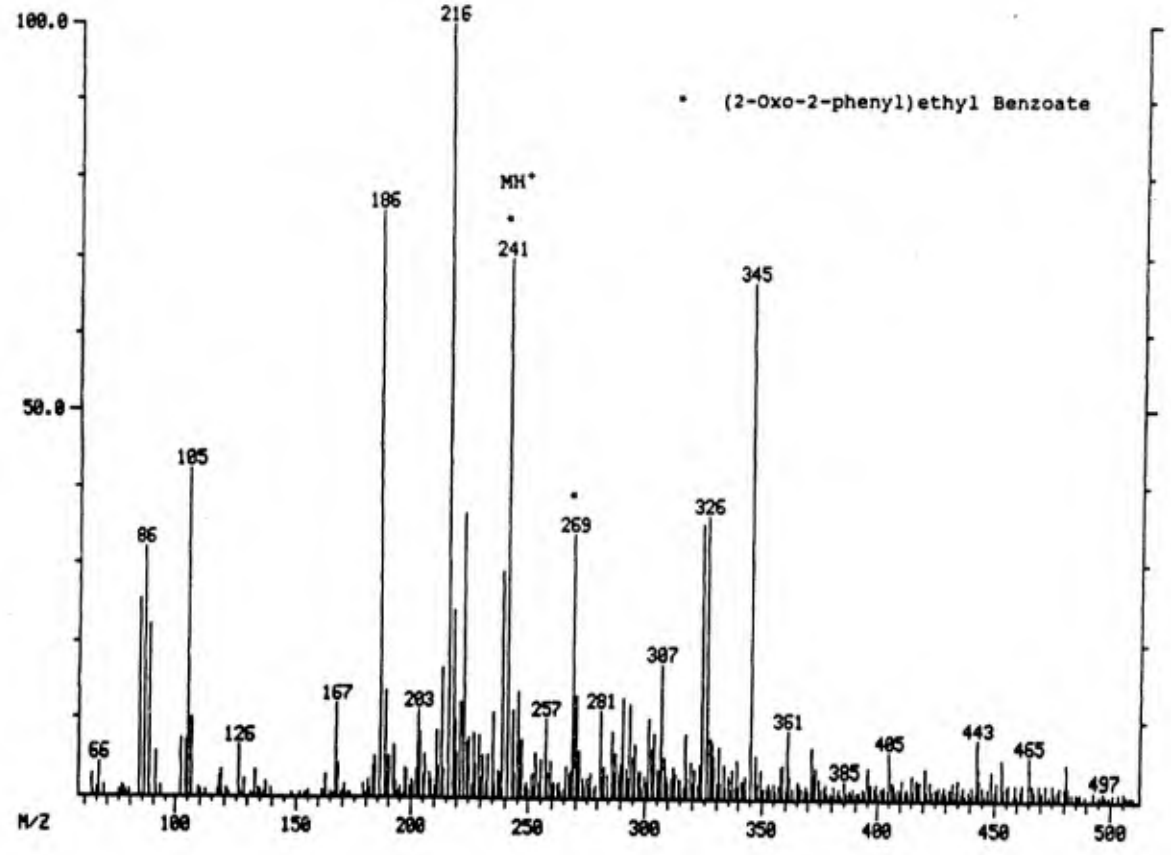
CI Mass Spectrum of OTH-1193-5c Scan 207



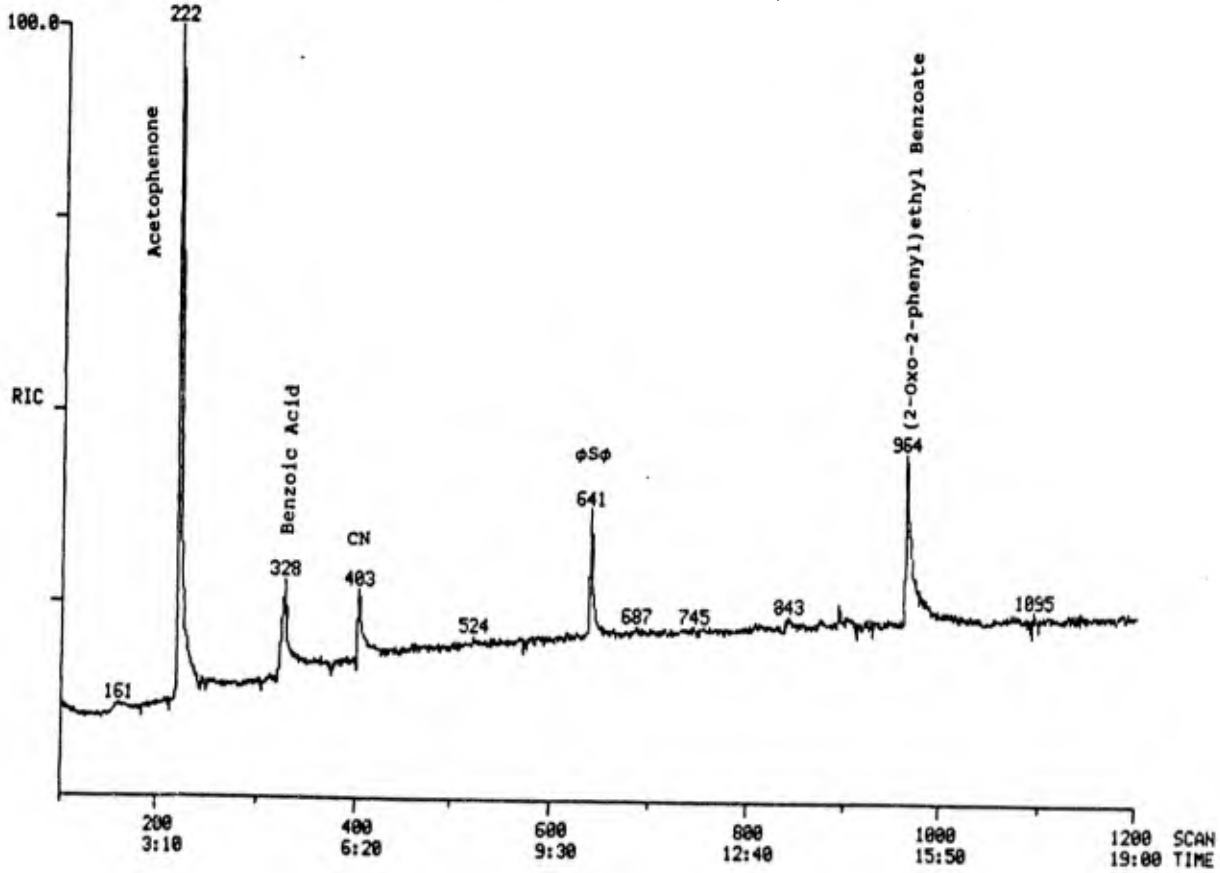
CI Mass Spectrum of OTH-1193-5c Scan 491



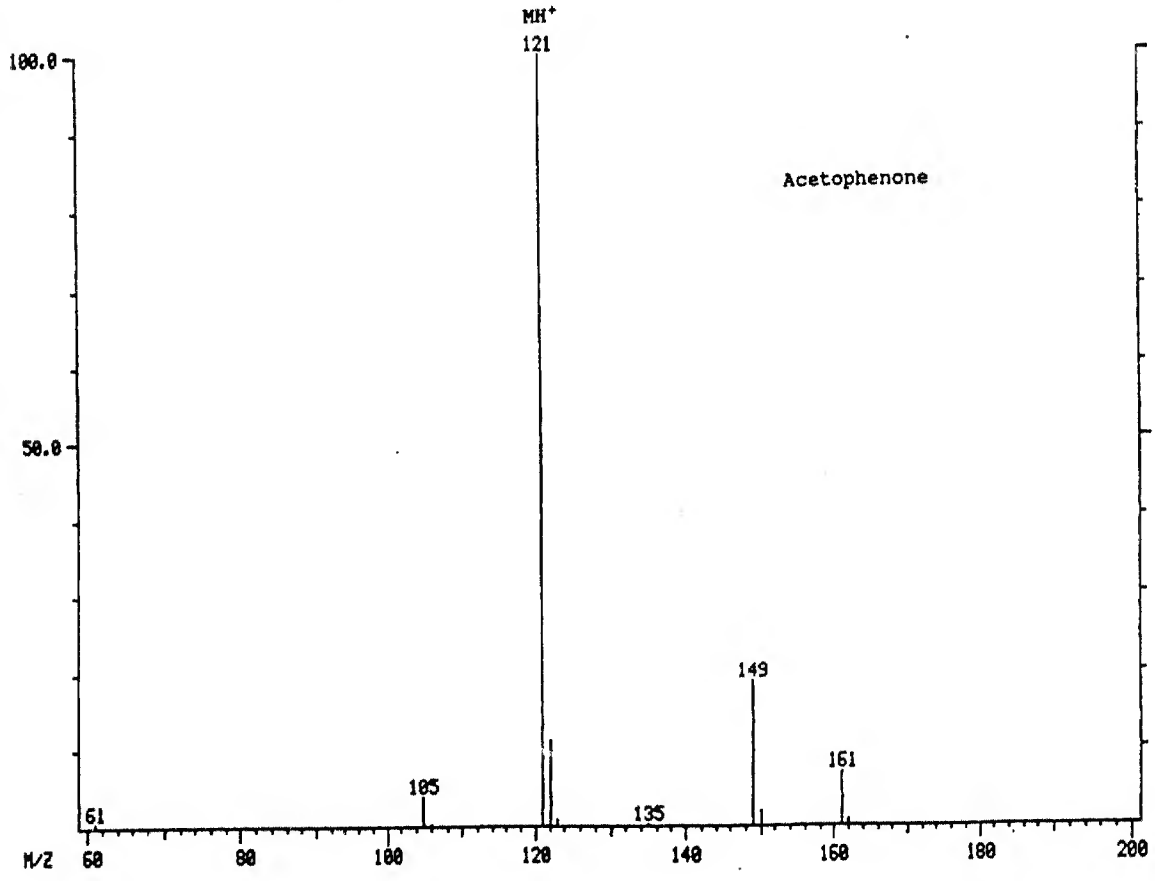
DEP/CI Mass Spectrum of OTH-1393-1b (Pig #23)



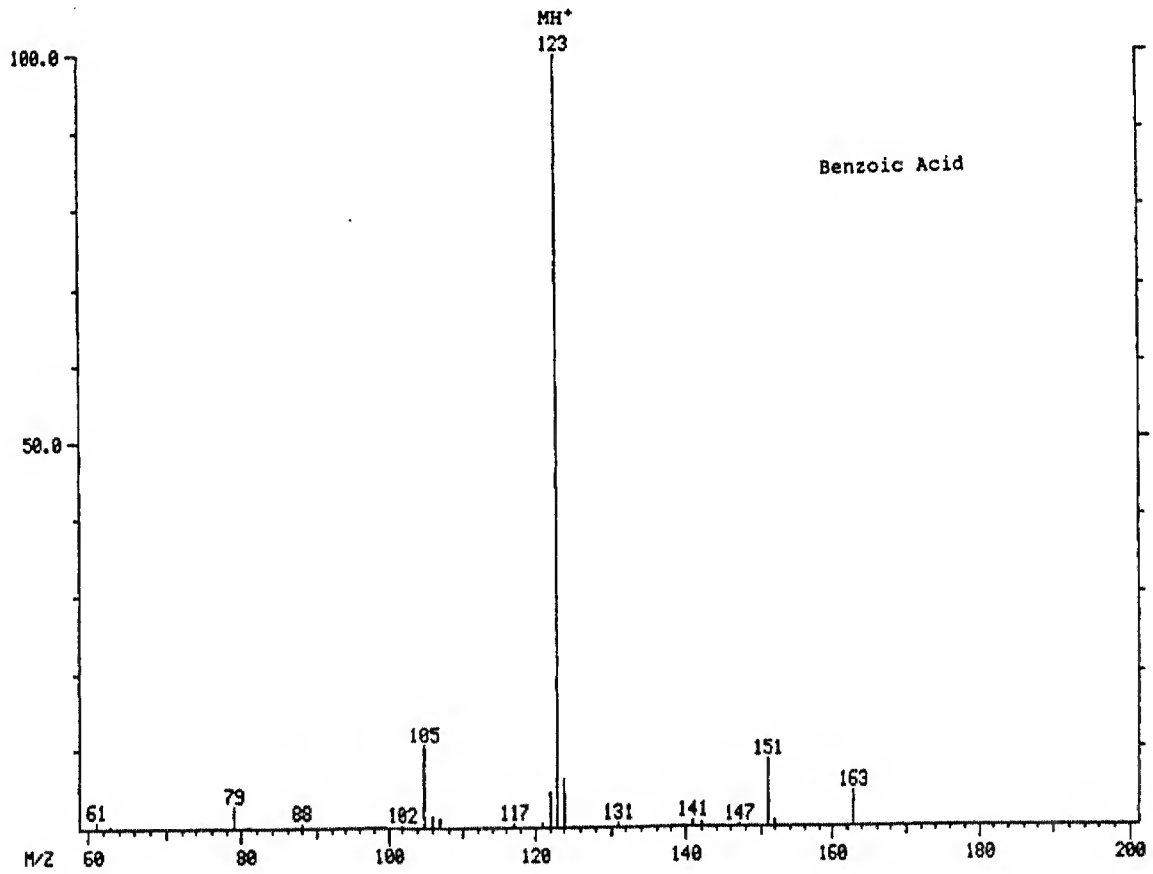
GC/MS/CI Chromatogram of OTH-1393-1b (Pig #23)



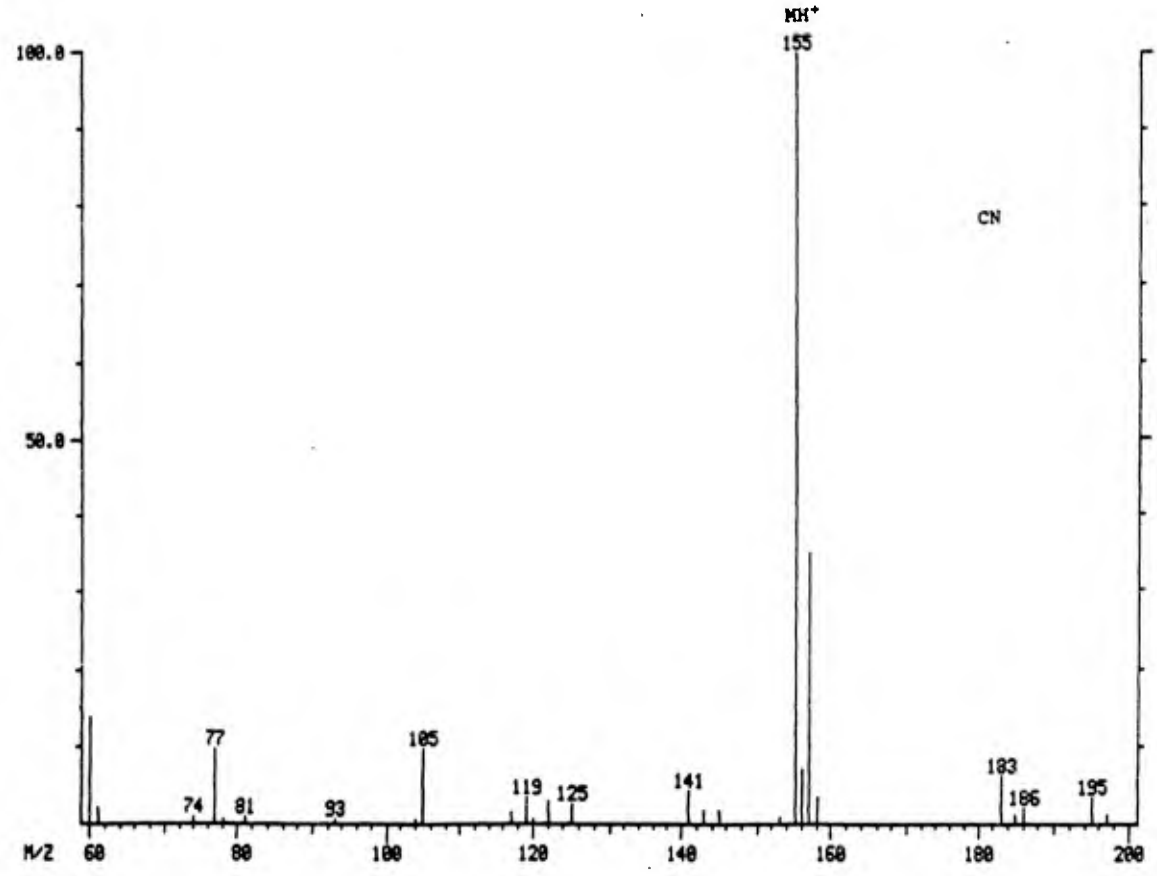
CI Mass Spectrum of OTH-1393-1b Scan 222



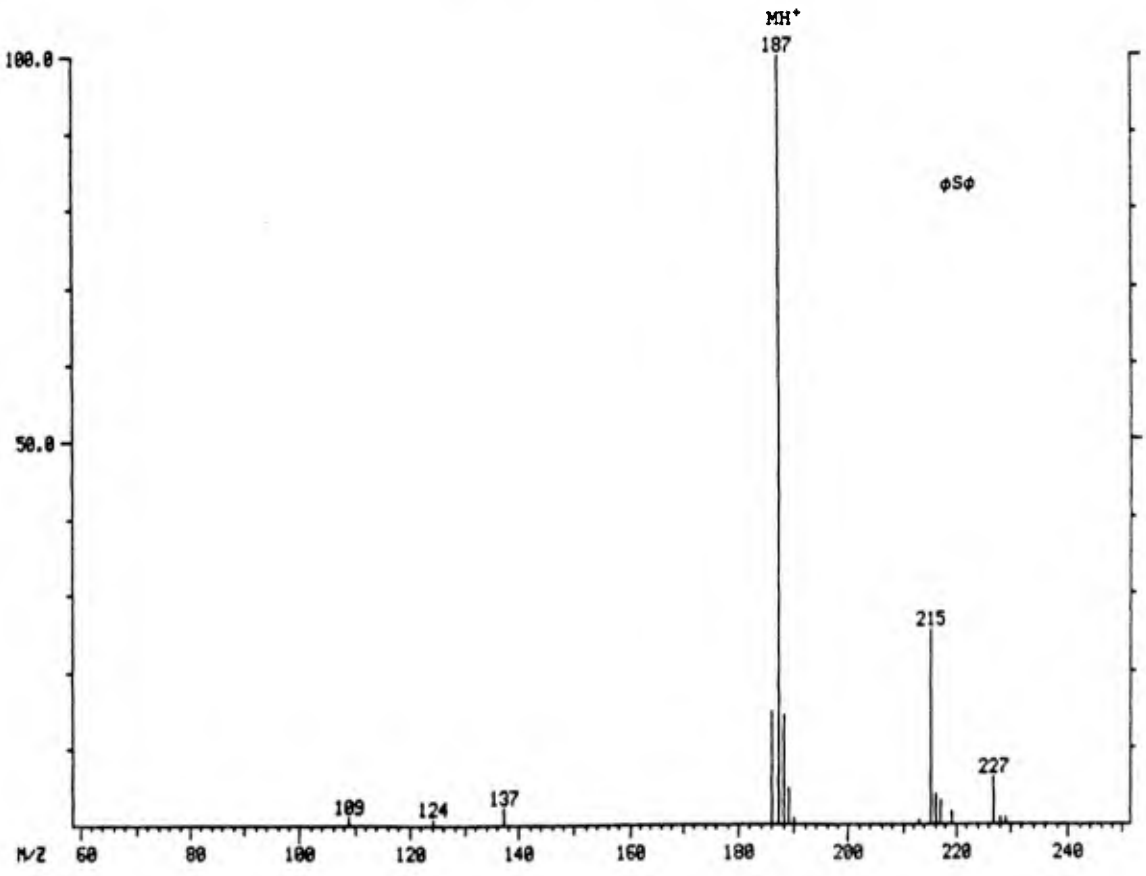
CI Mass Spectrum of OTH-1393-1b Scan 328



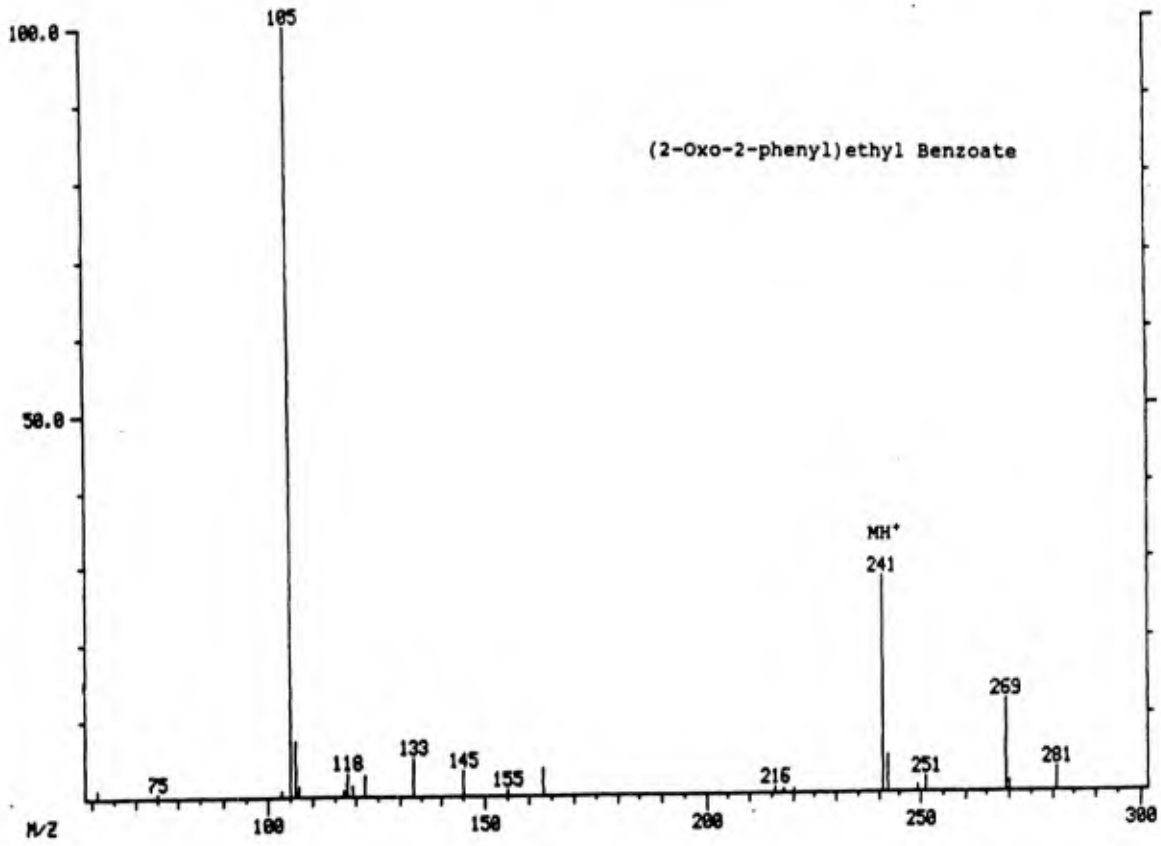
CI Mass Spectrum of OTH-1393-1b Scan 403



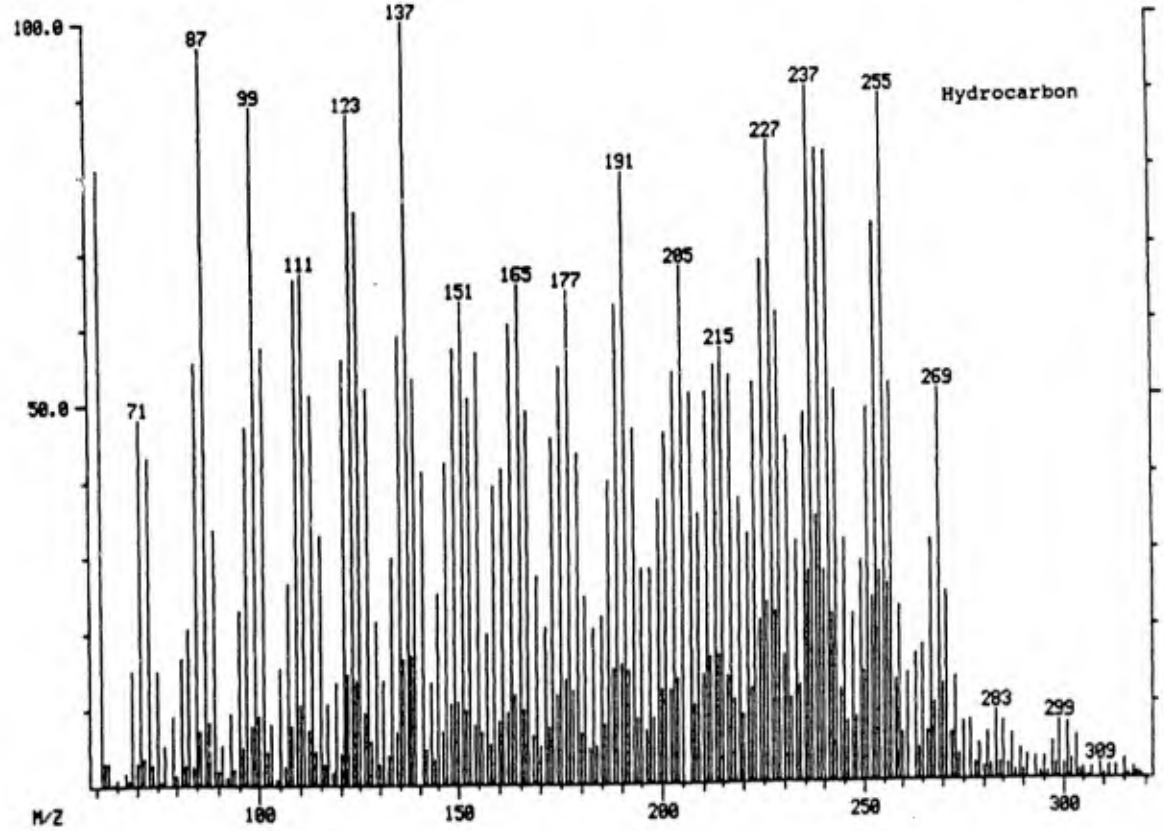
CI Mass Spectrum of OTH-1393-1b Scan 641



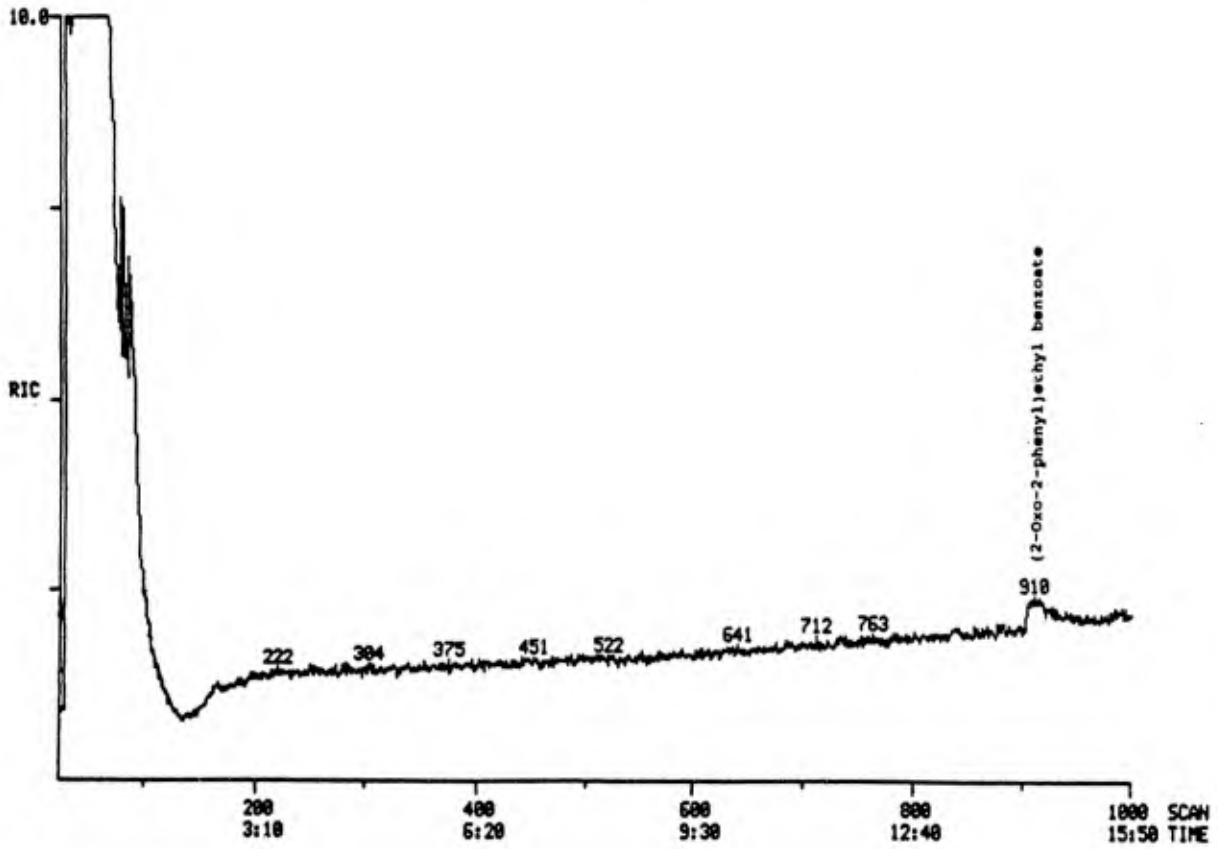
CI Mass Spectrum of OTH-1393-1b Scan 964



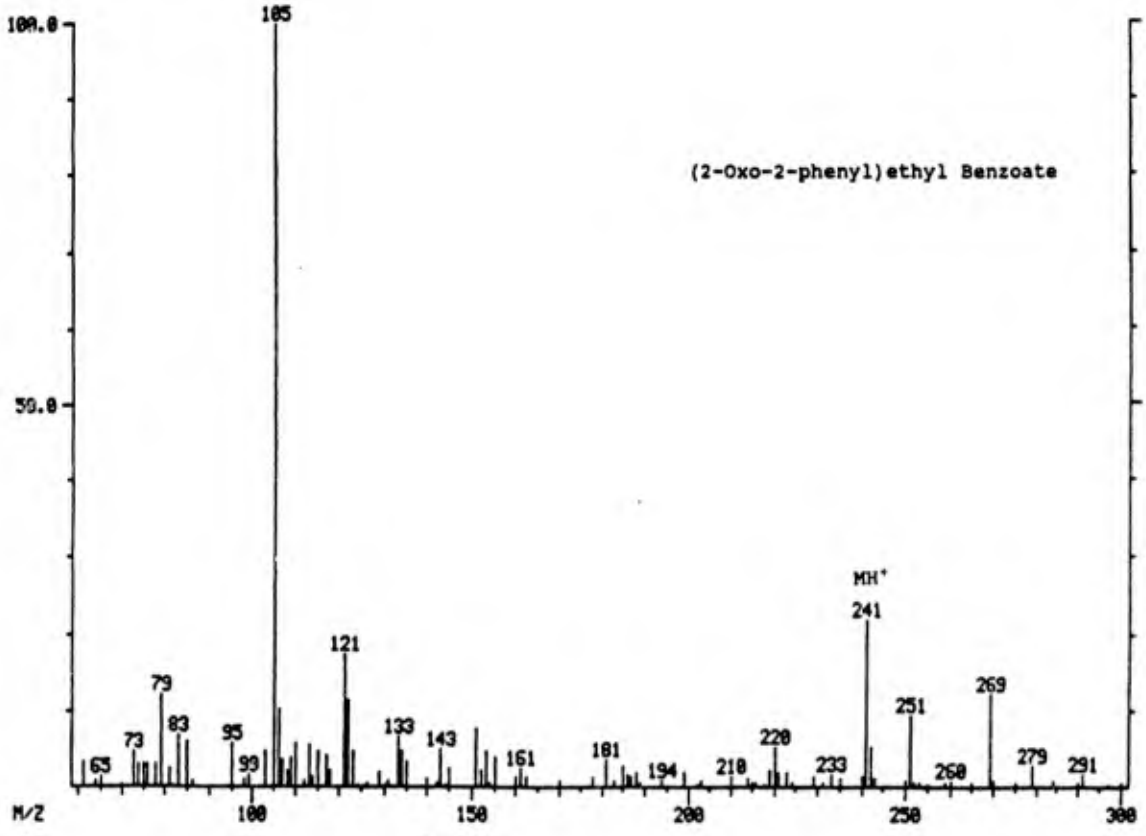
DEP/CI Mass Spectrum of OTH-1393-1c (Pig #23)



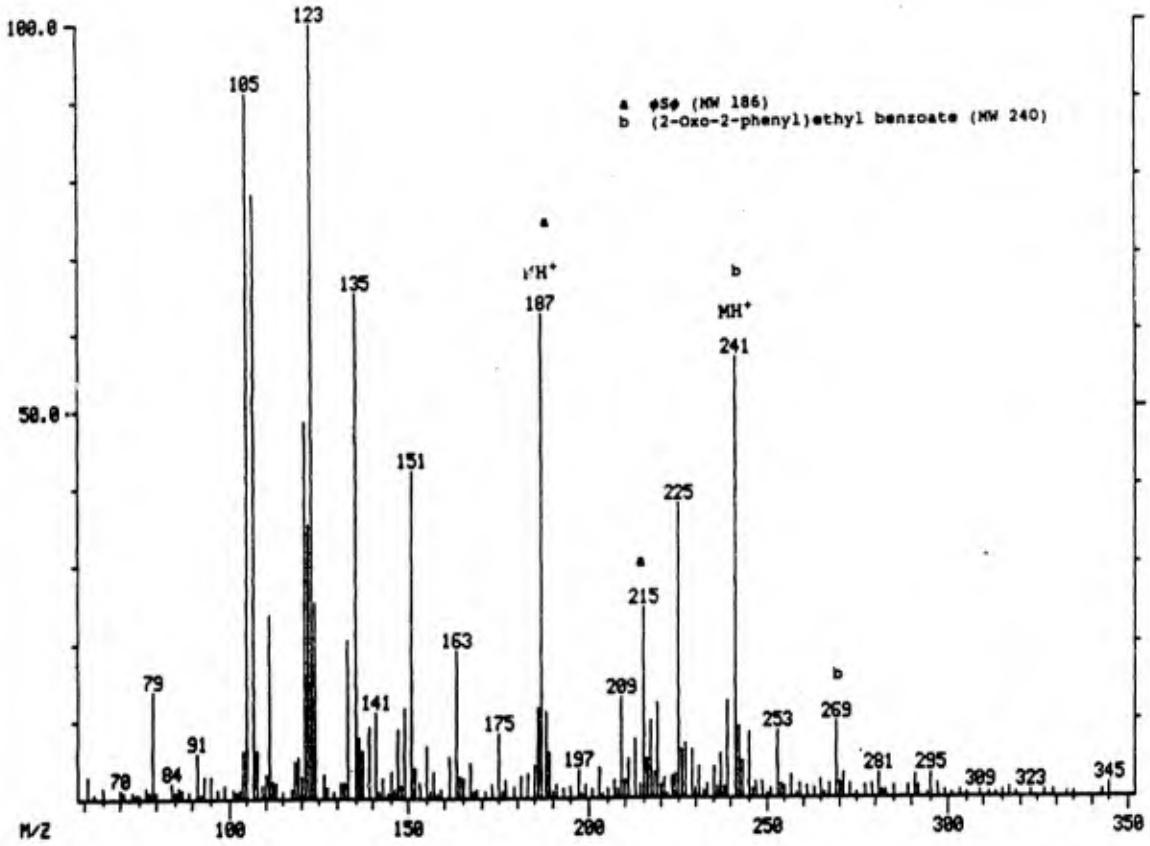
GC/MS/CI Chromatogram of OTH-1393-1d (Pig #23)



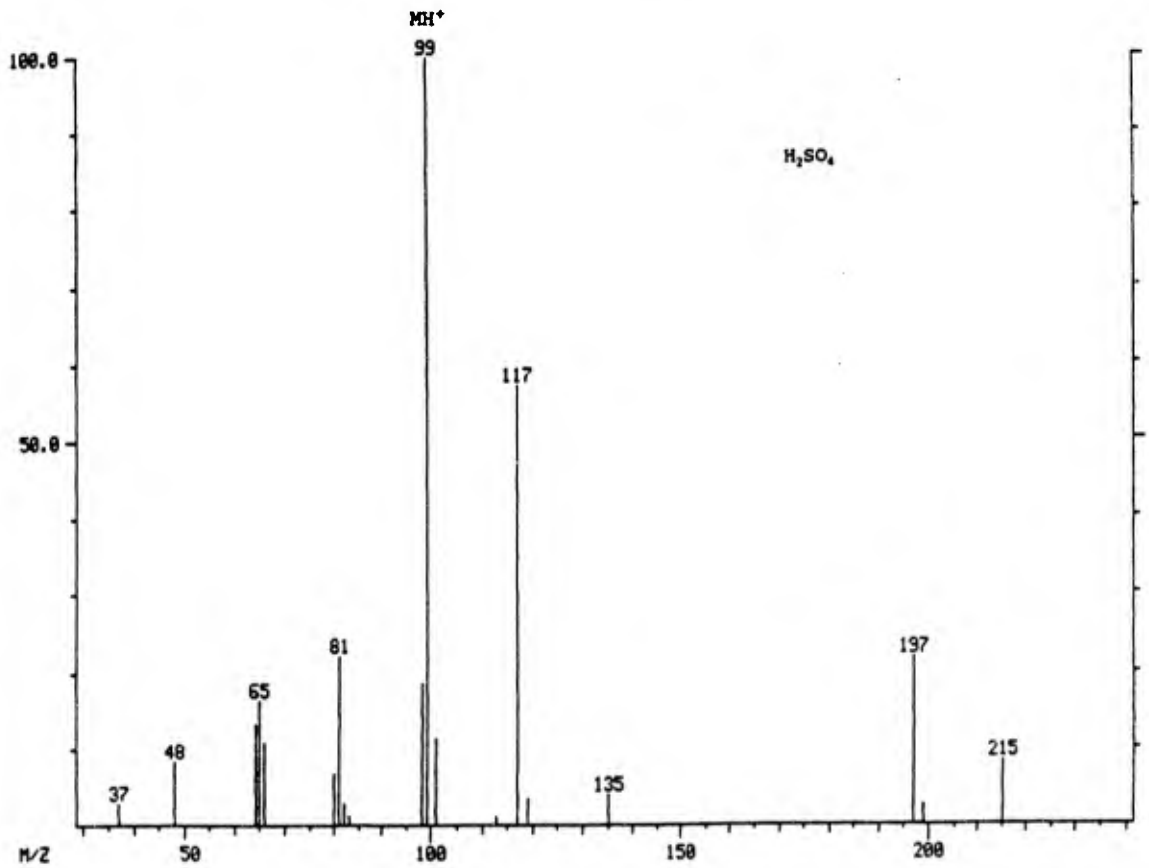
CI Mass Spectrum of OTH-1393-1d Scan 910



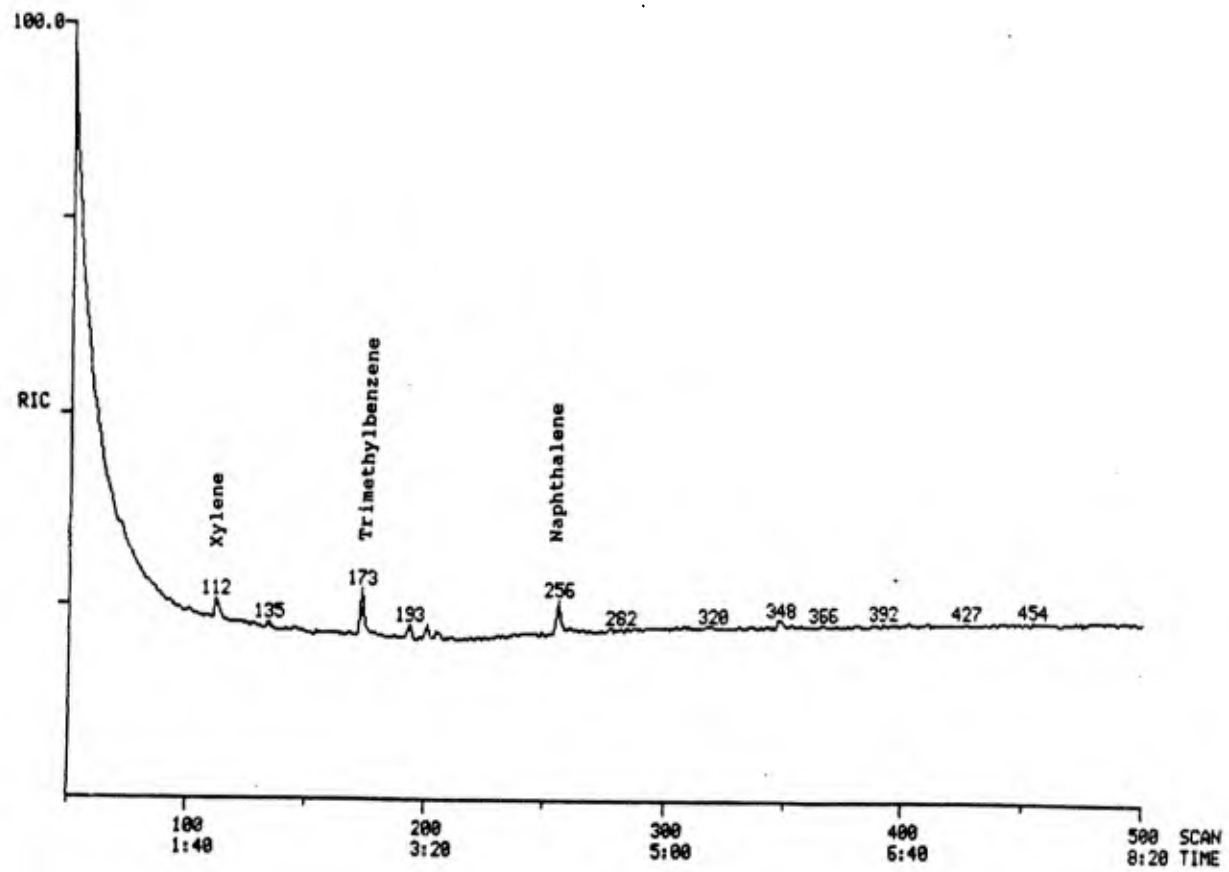
DEP/CI Mass Spectrum of OTH-1393-1d (Pig #23)



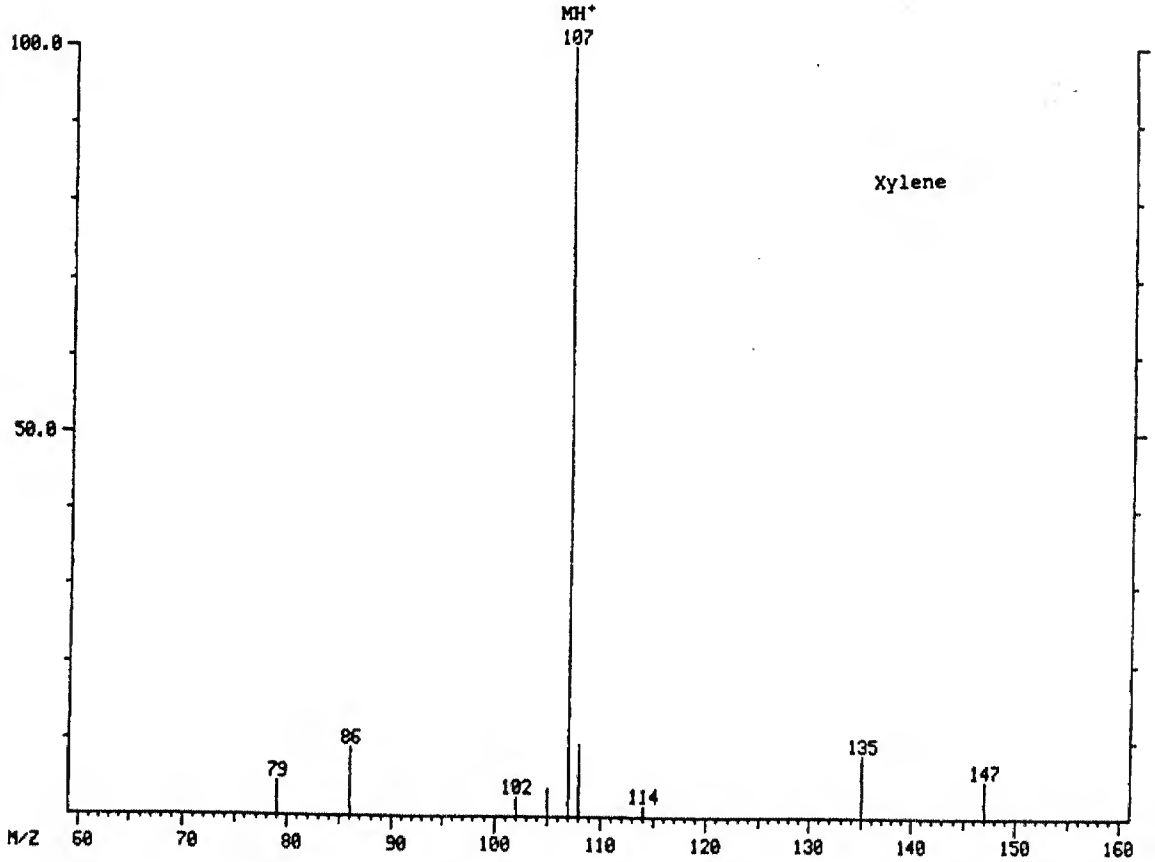
DEP/CI Mass Spectrum of OTH-493-1c



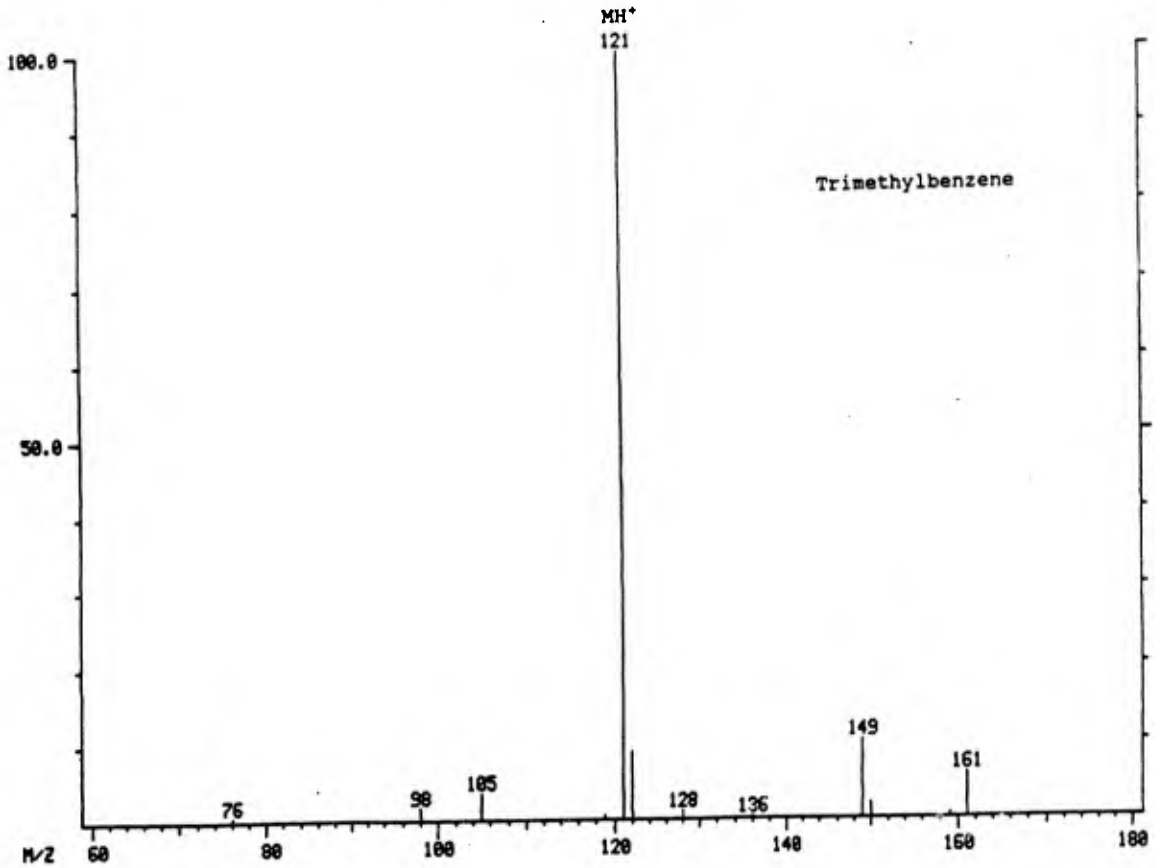
GC/MS/CI Chromatogram of OTH-593-1c



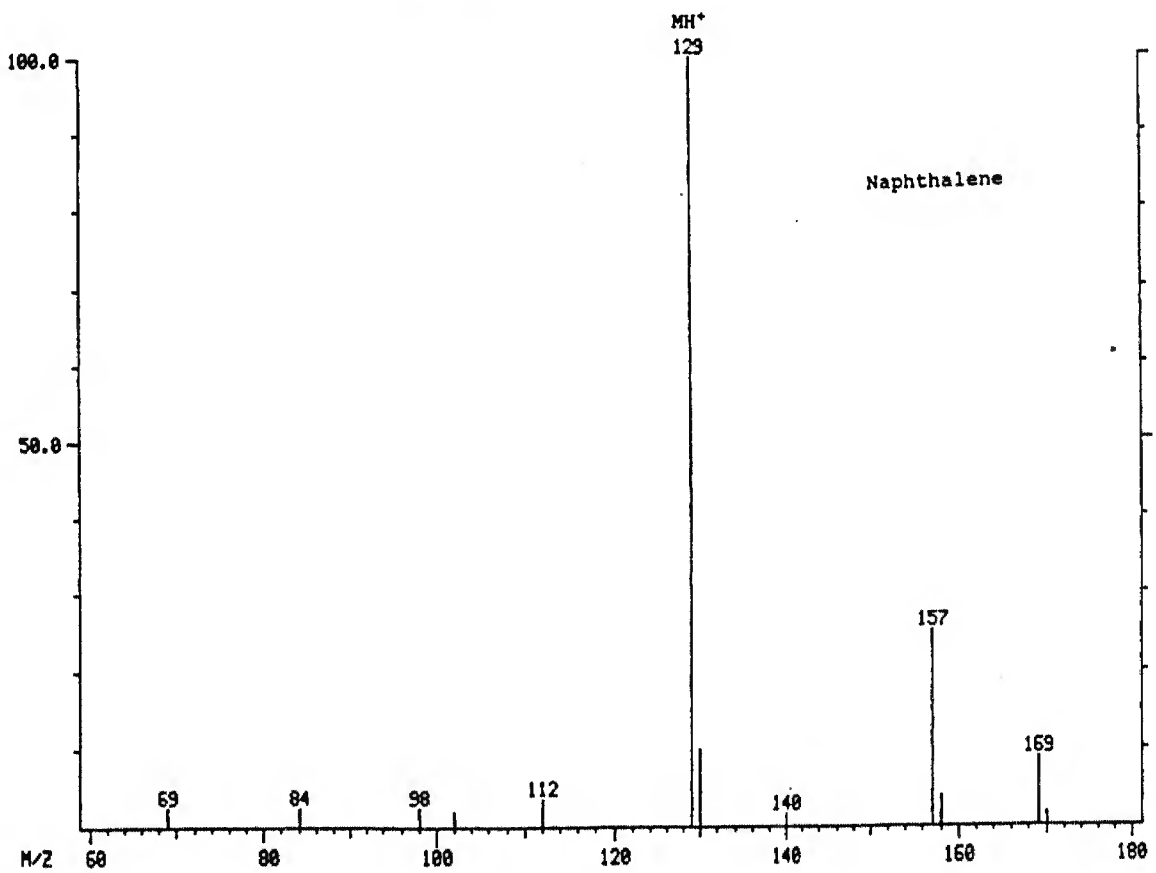
CI Mass Spectrum of OTH-593-1c Scan 112



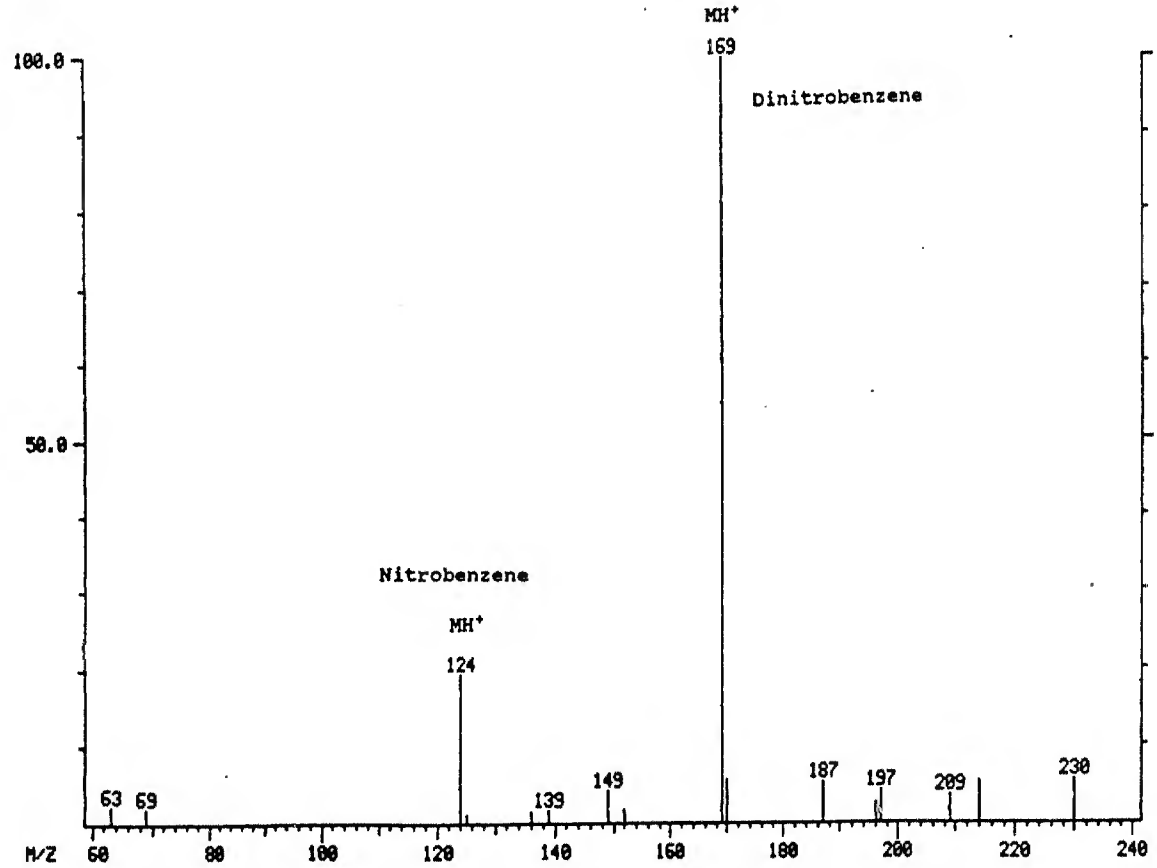
CI Mass Spectrum of OTH-593-1c Scan 173



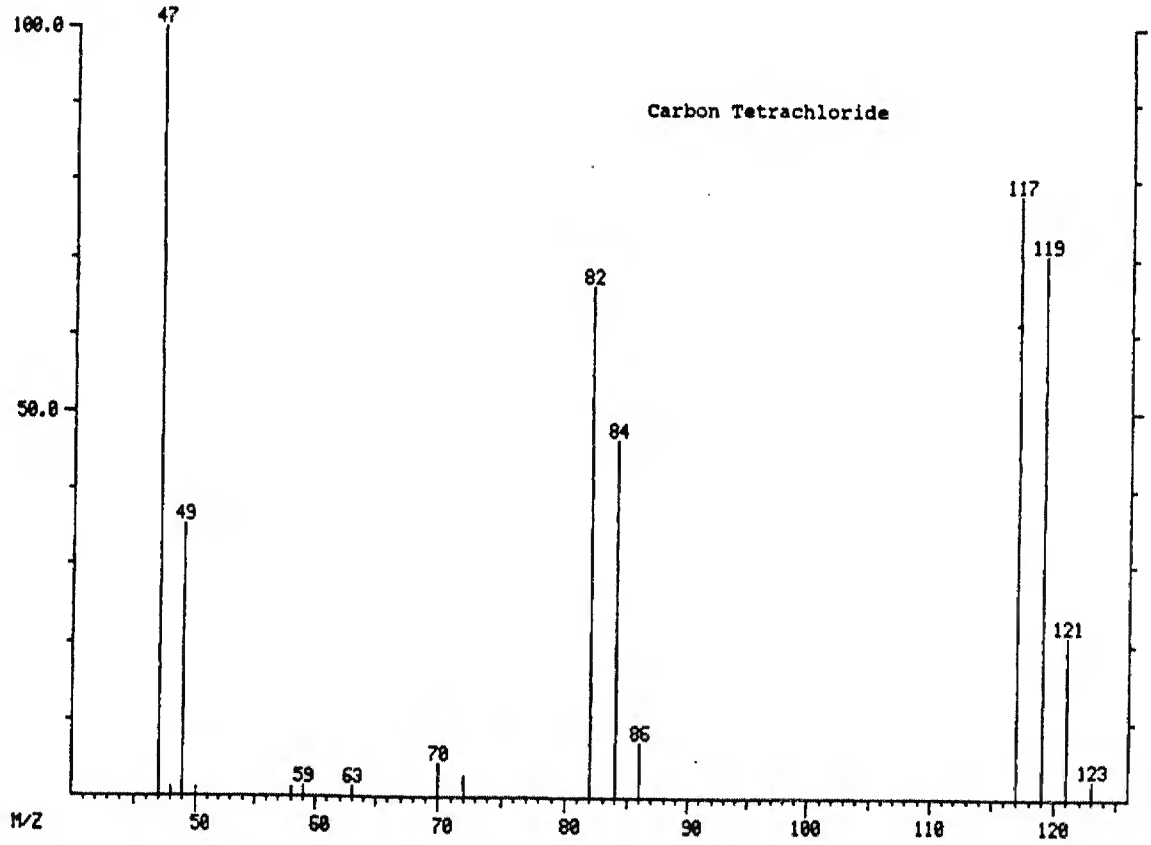
CI Mass Spectrum of OTH-593-1c Scan 256



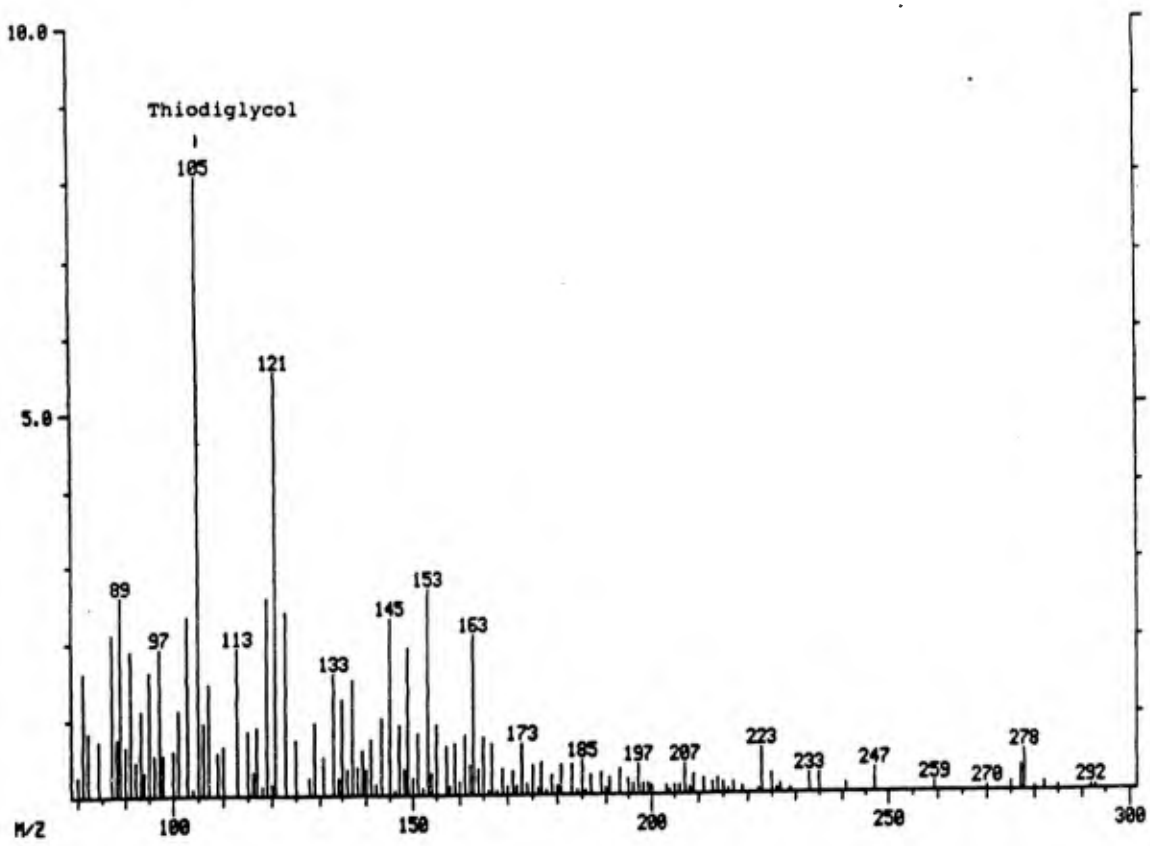
DEP/CI Mass Spectrum of OTH-793-2



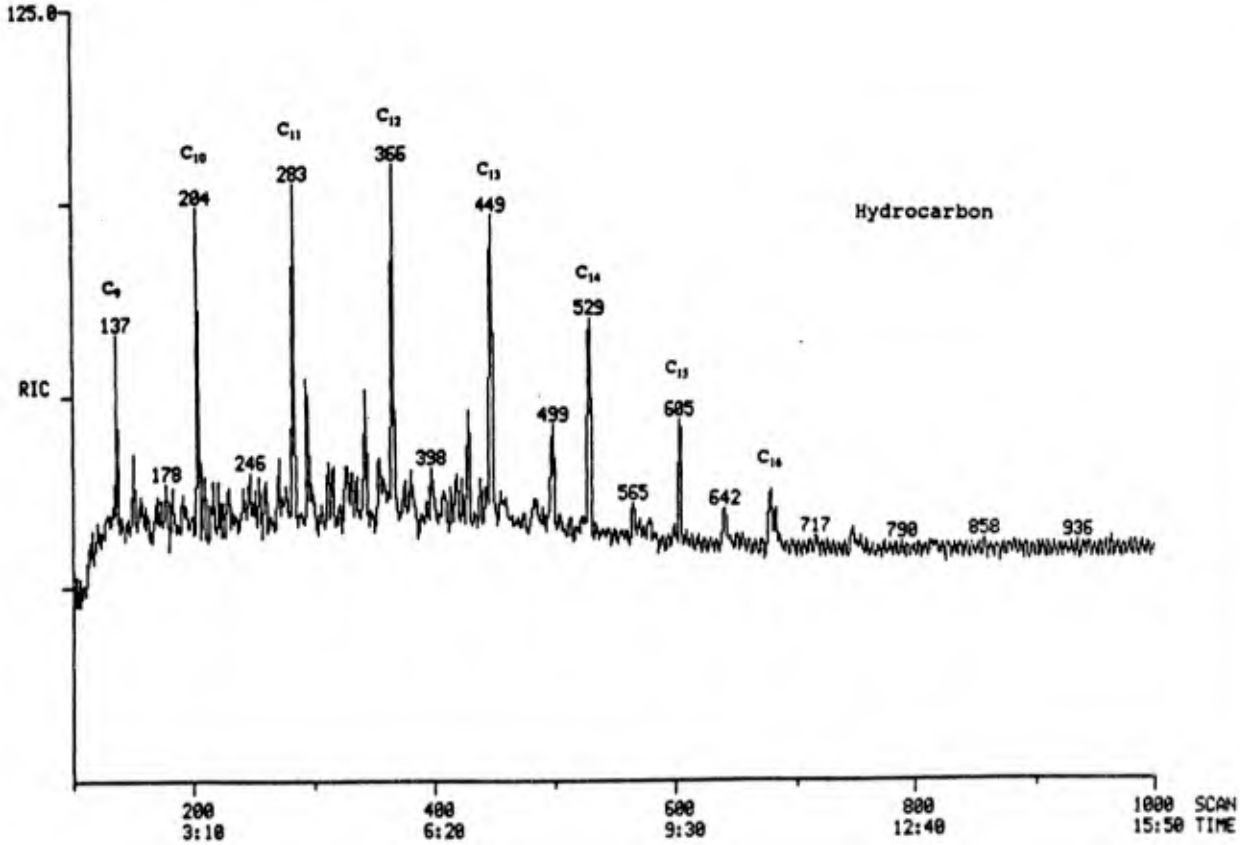
CI Mass Spectrum of OTH-793-2 Scan 36



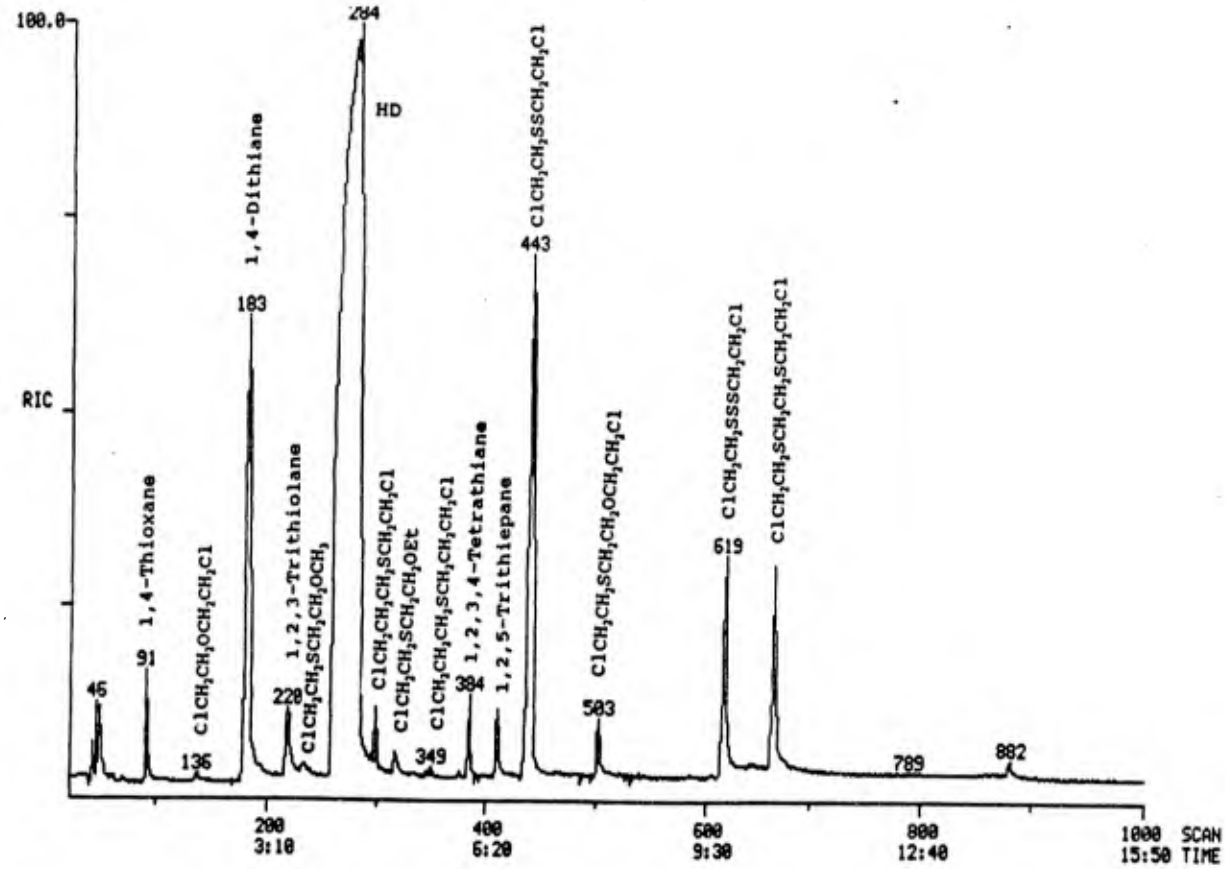
DEP/CI Mass Spectrum of OTH-1493-1c



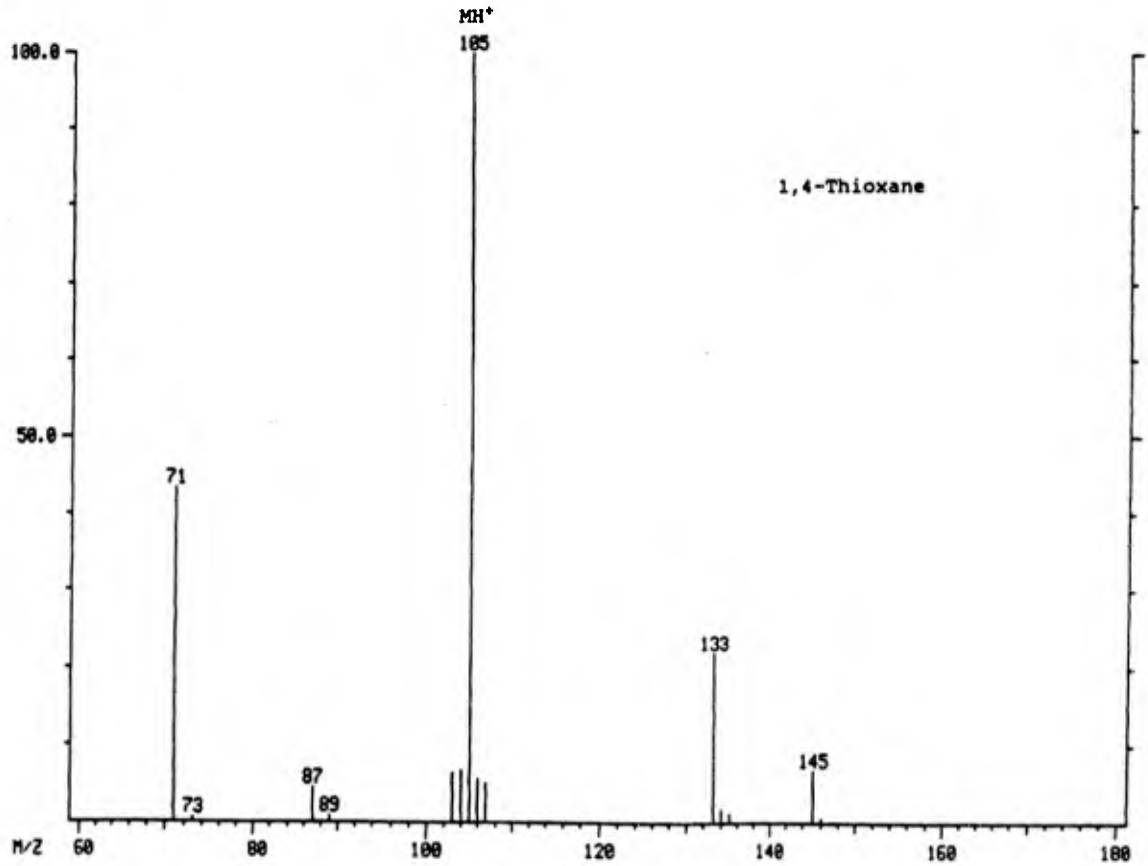
GC/MS/CI Chromatogram of OTH-1493-2



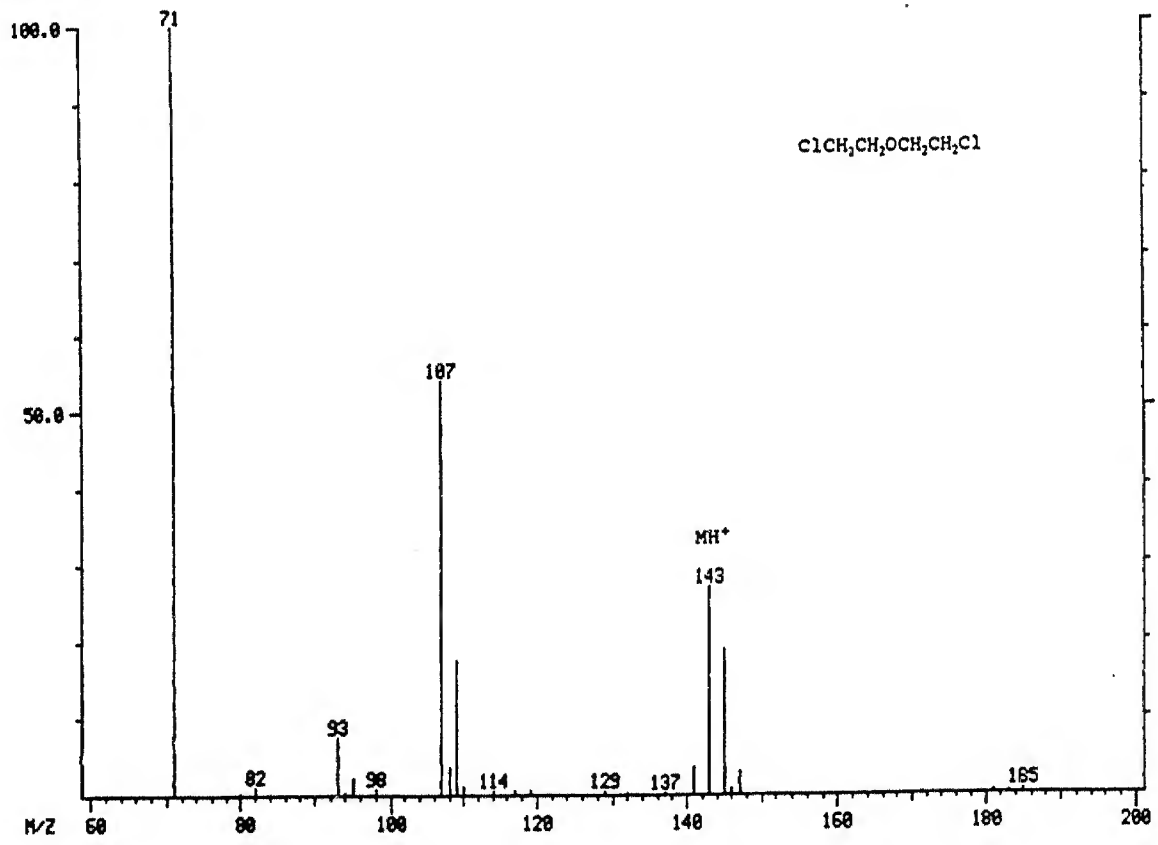
GC/MS/CI Chromatogram of OTH-1593-1c



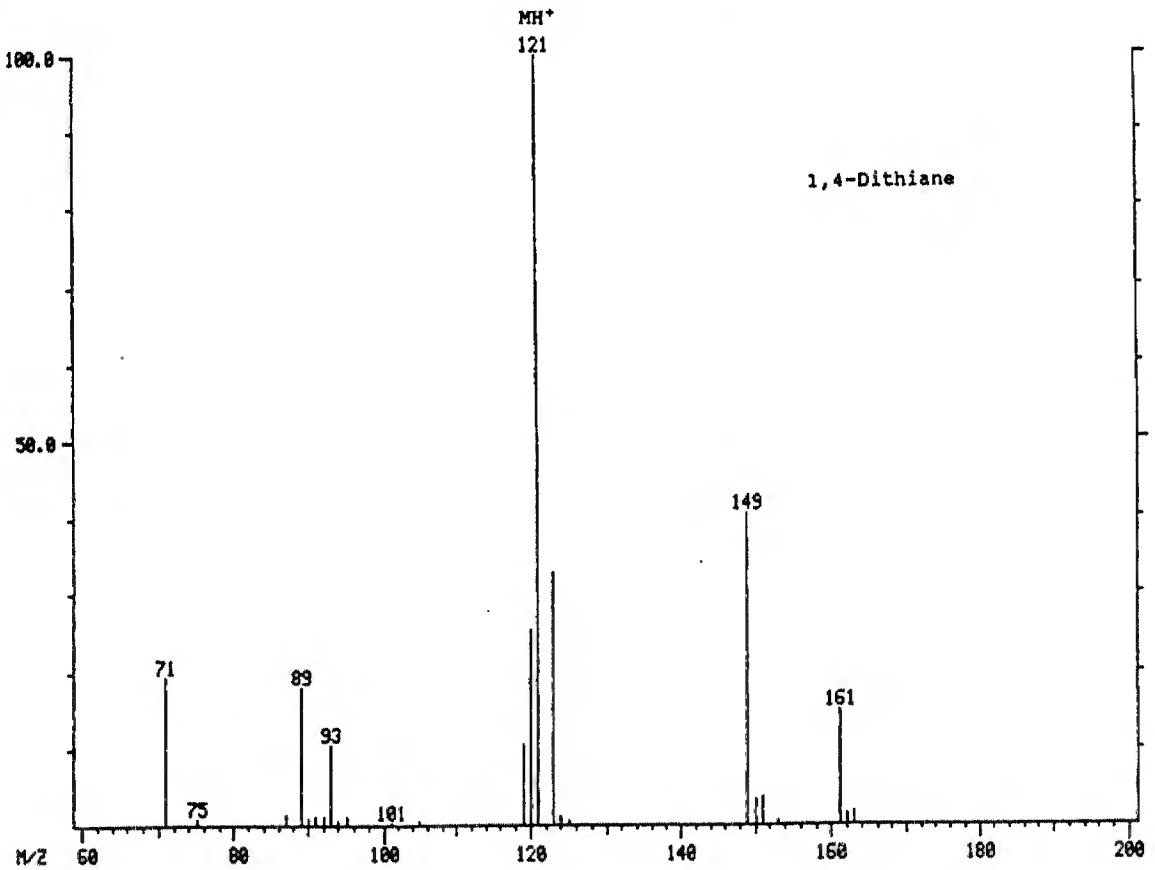
CI Mass Spectrum of OTH-1593-1c Scan 91



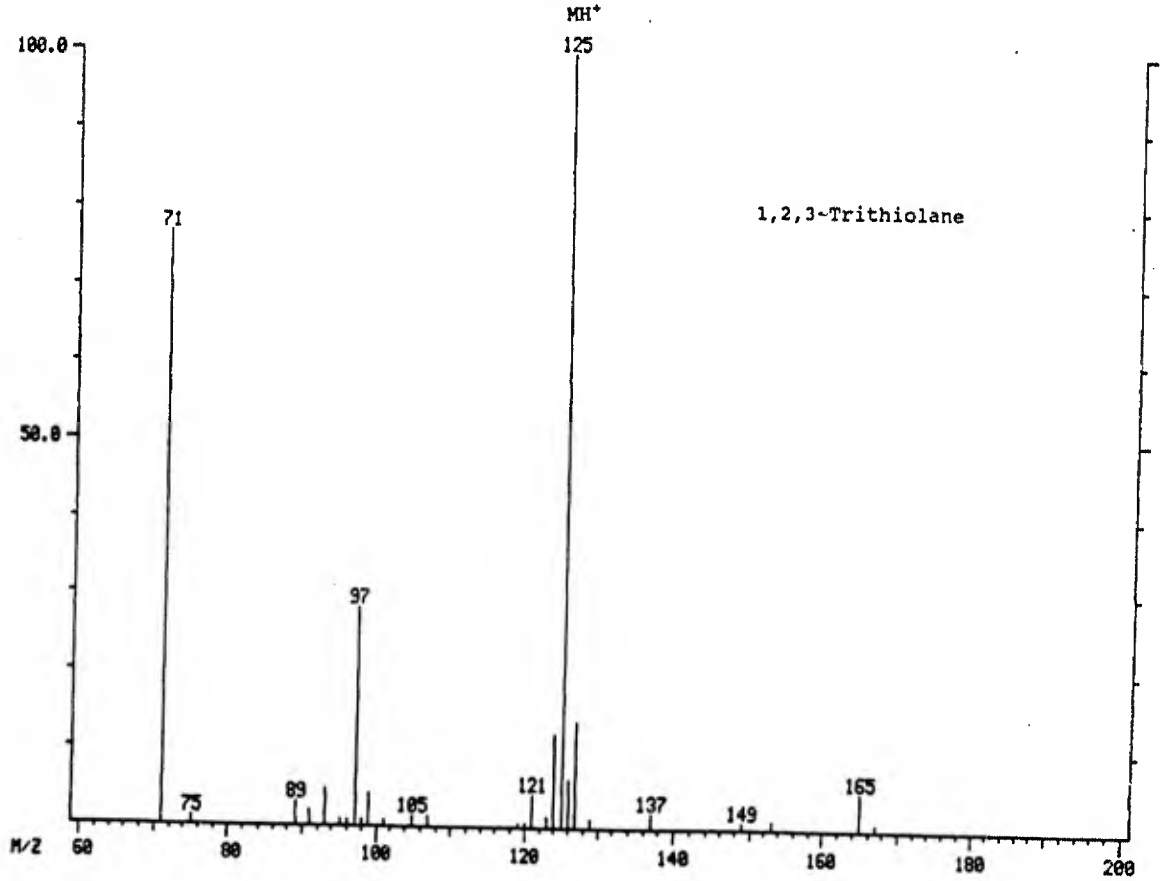
CI Mass Spectrum of OTH-1593-1c Scan 136



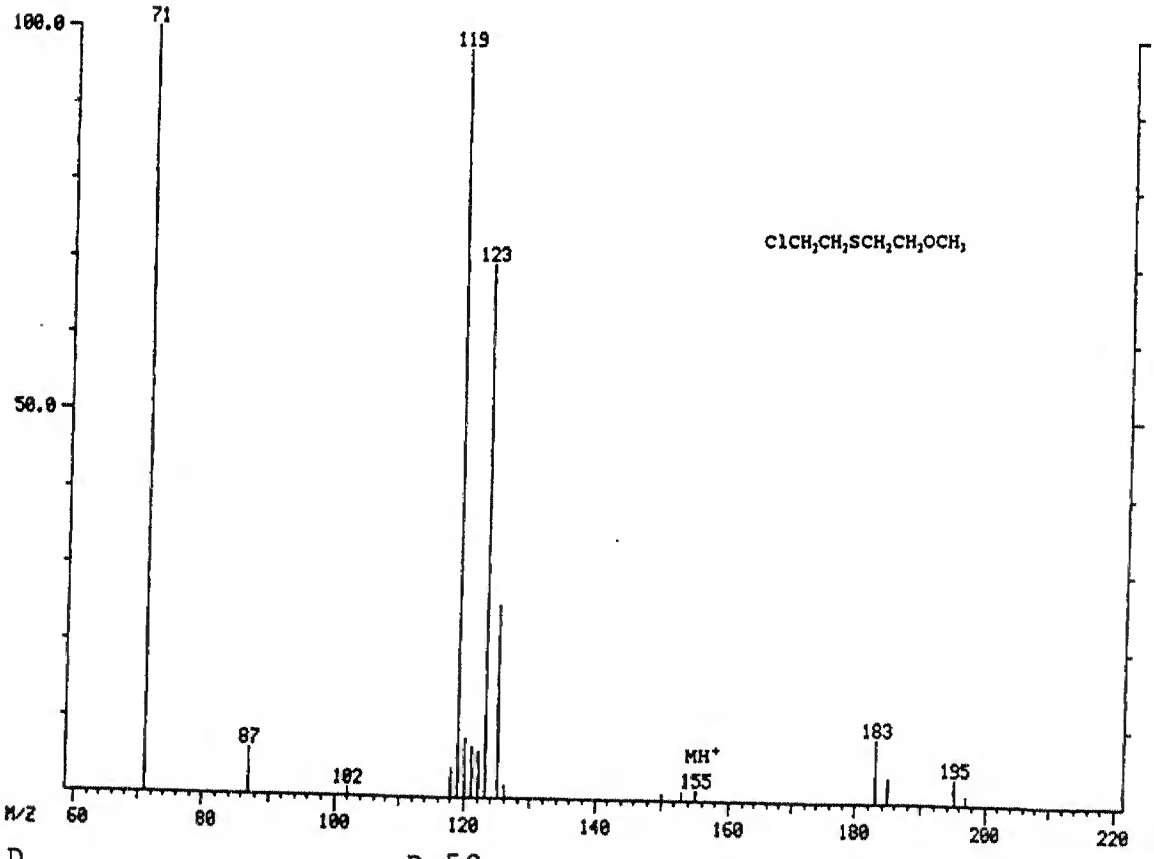
CI Mass Spectrum of OTH-1593-1c Scan 183



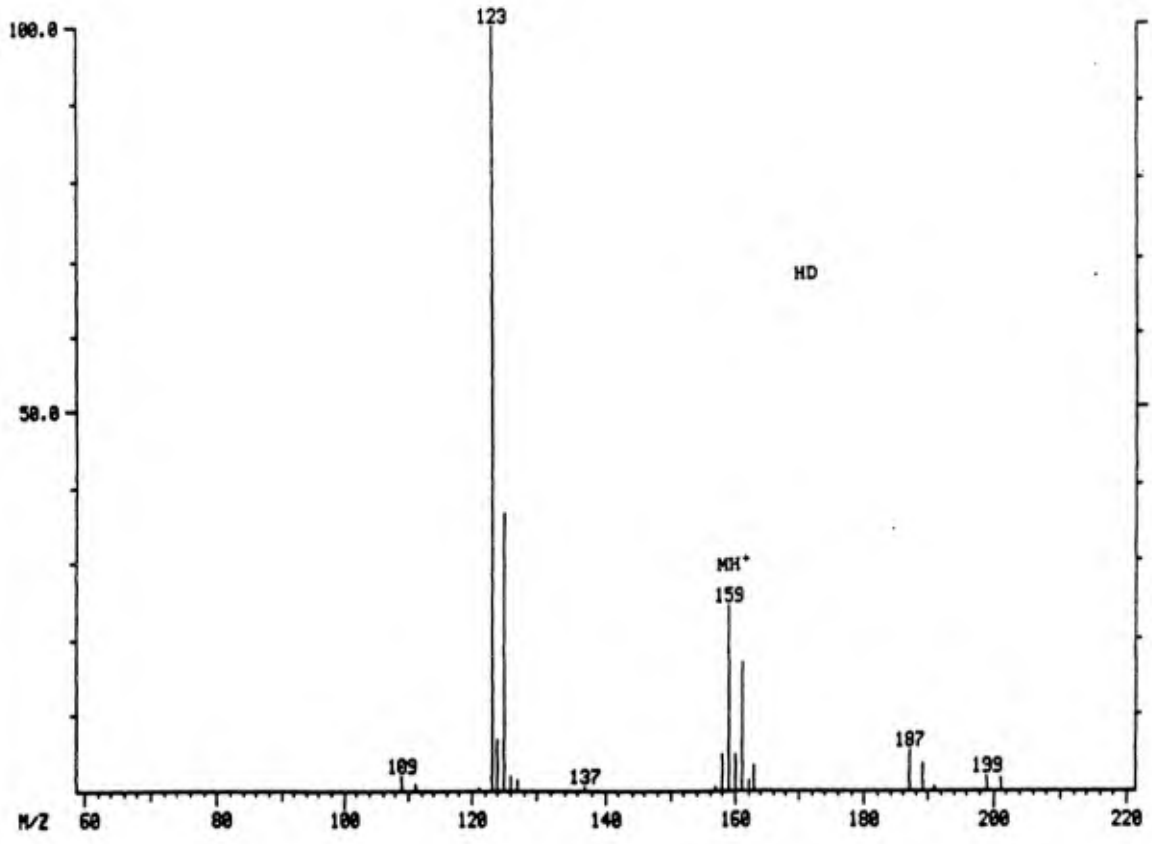
CI Mass Spectrum of OTH-1593-1c Scan 220



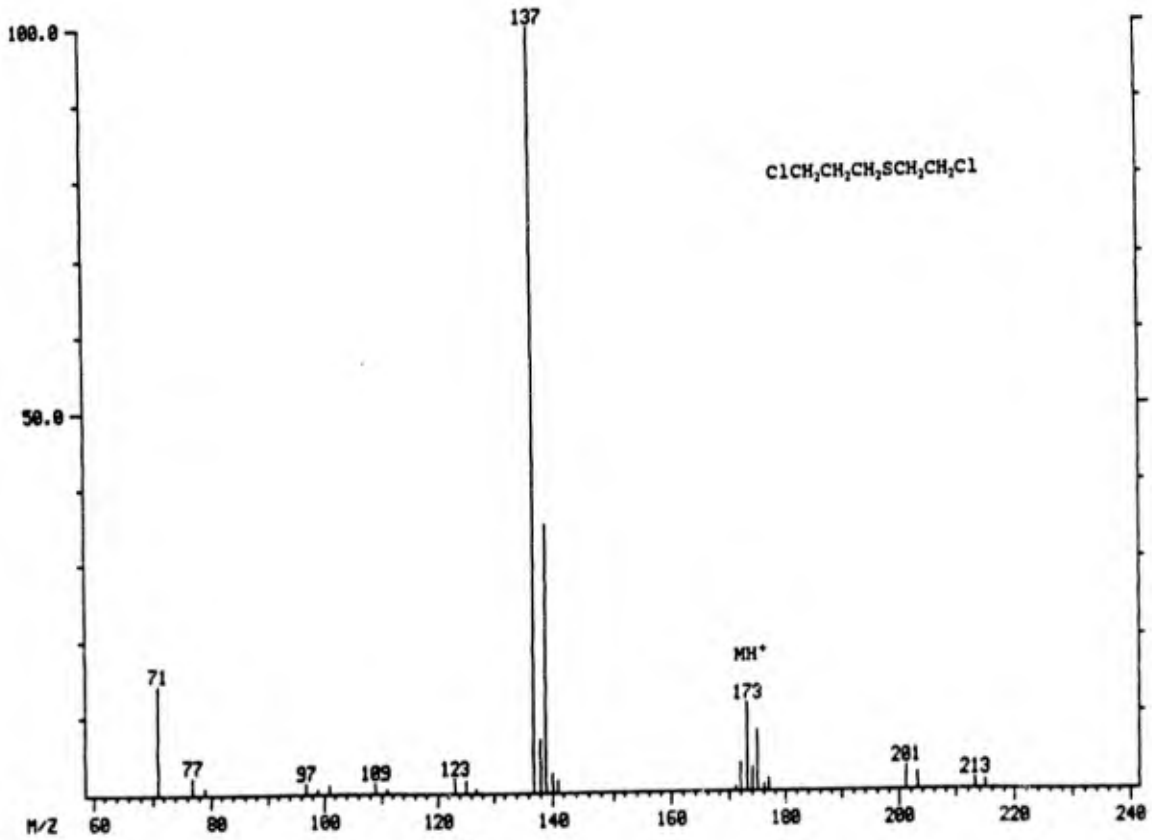
CI Mass Spectrum of OTH-1593-1c Scan 235



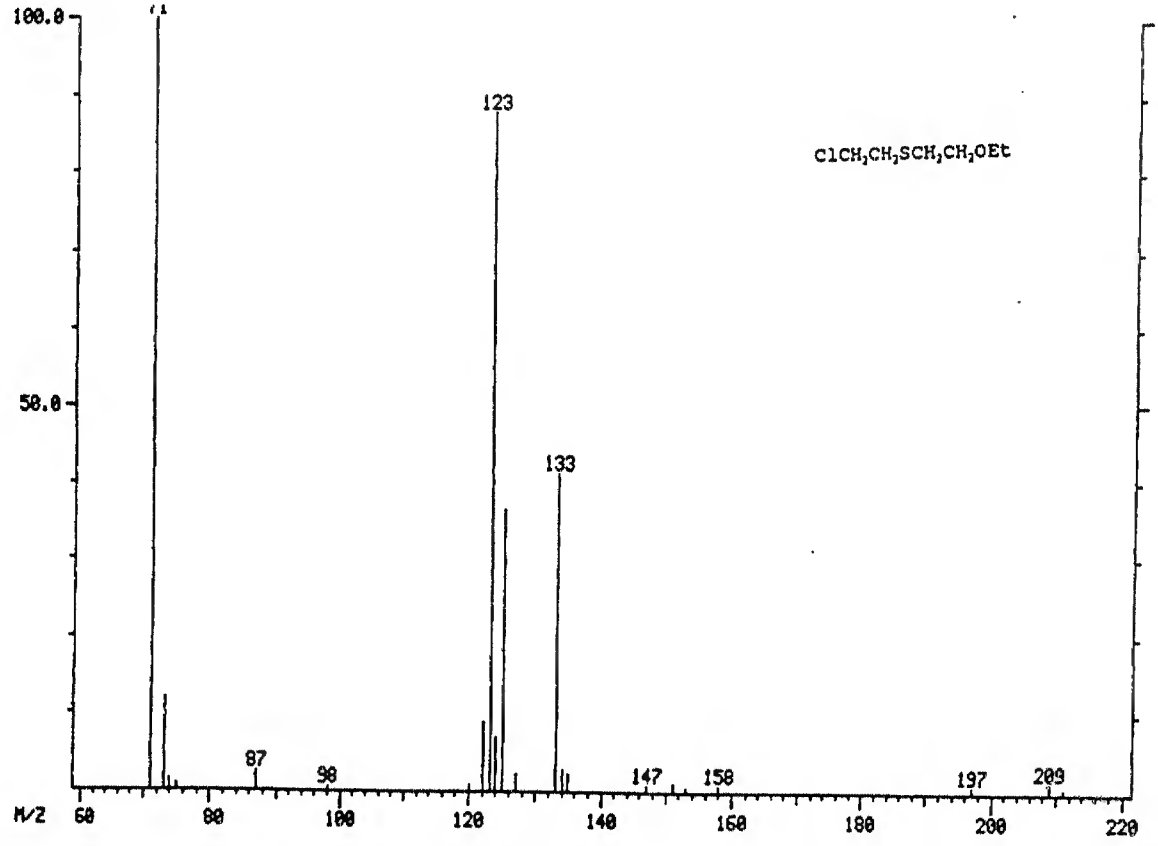
CI Mass Spectrum of OTH-1593-1c Scan 284



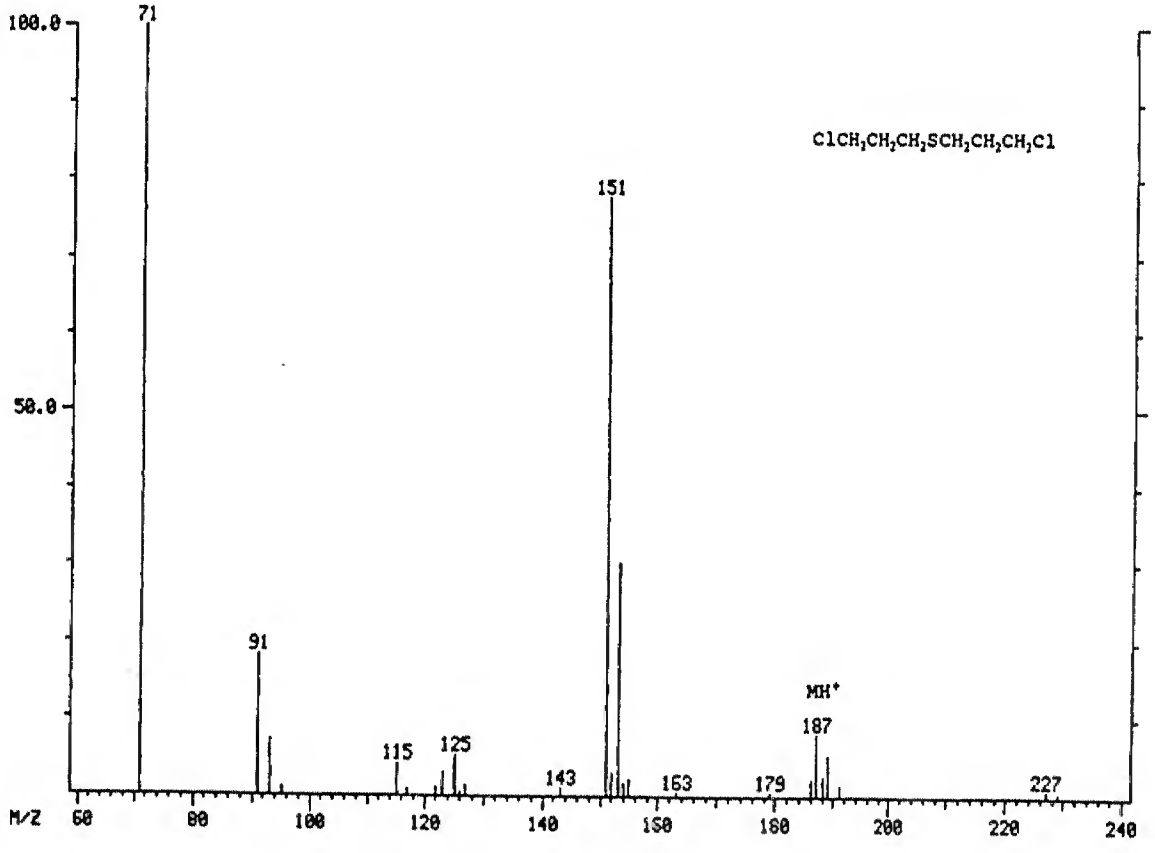
CI Mass Spectrum of OTH-1593-1c Scan 299



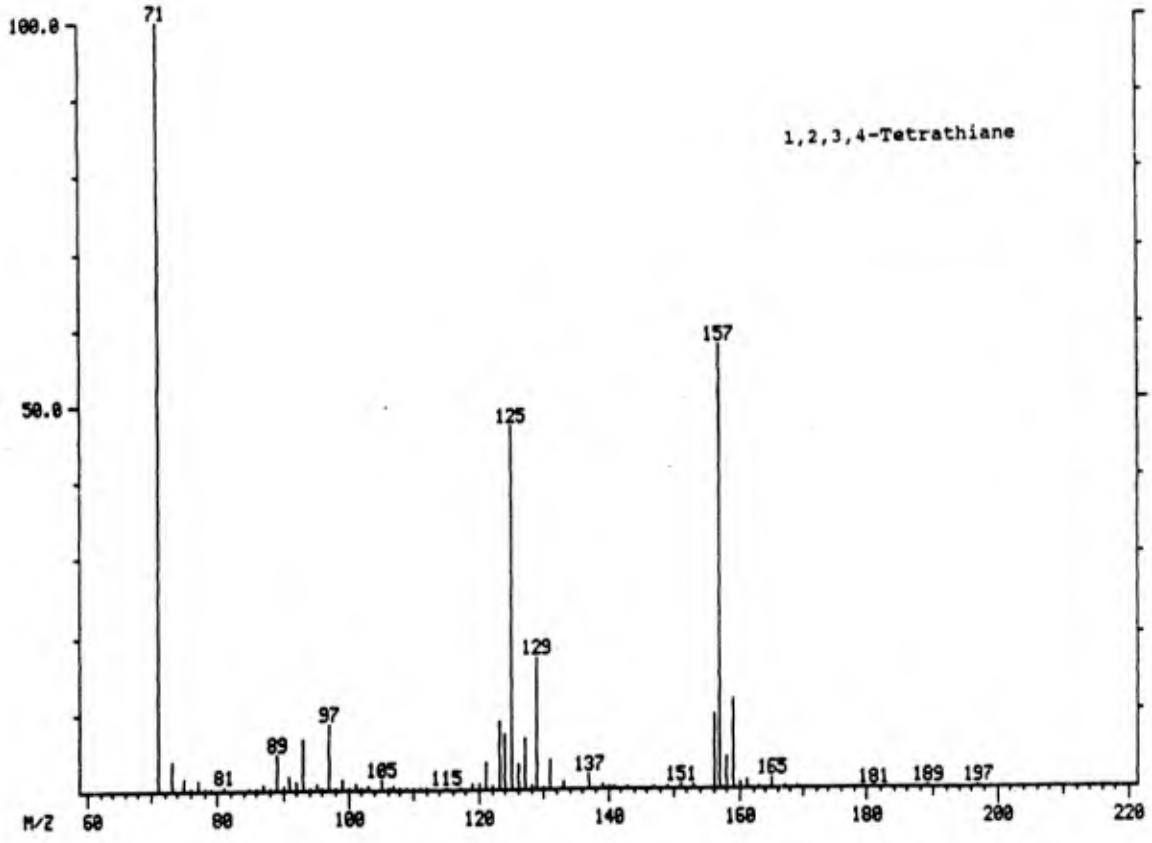
CI Mass Spectrum of OTH-1593-1c Scan 316



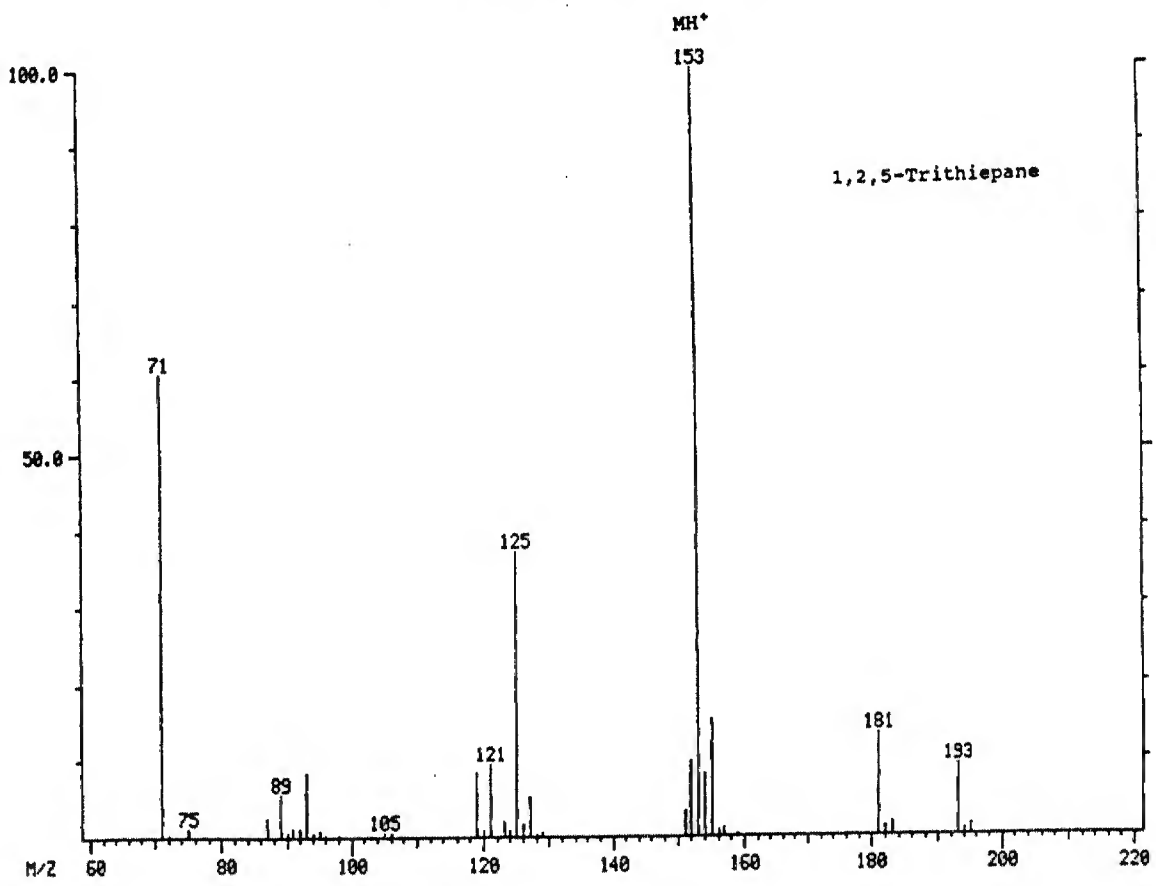
CI Mass Spectrum of OTH-1593-1c Scan 349



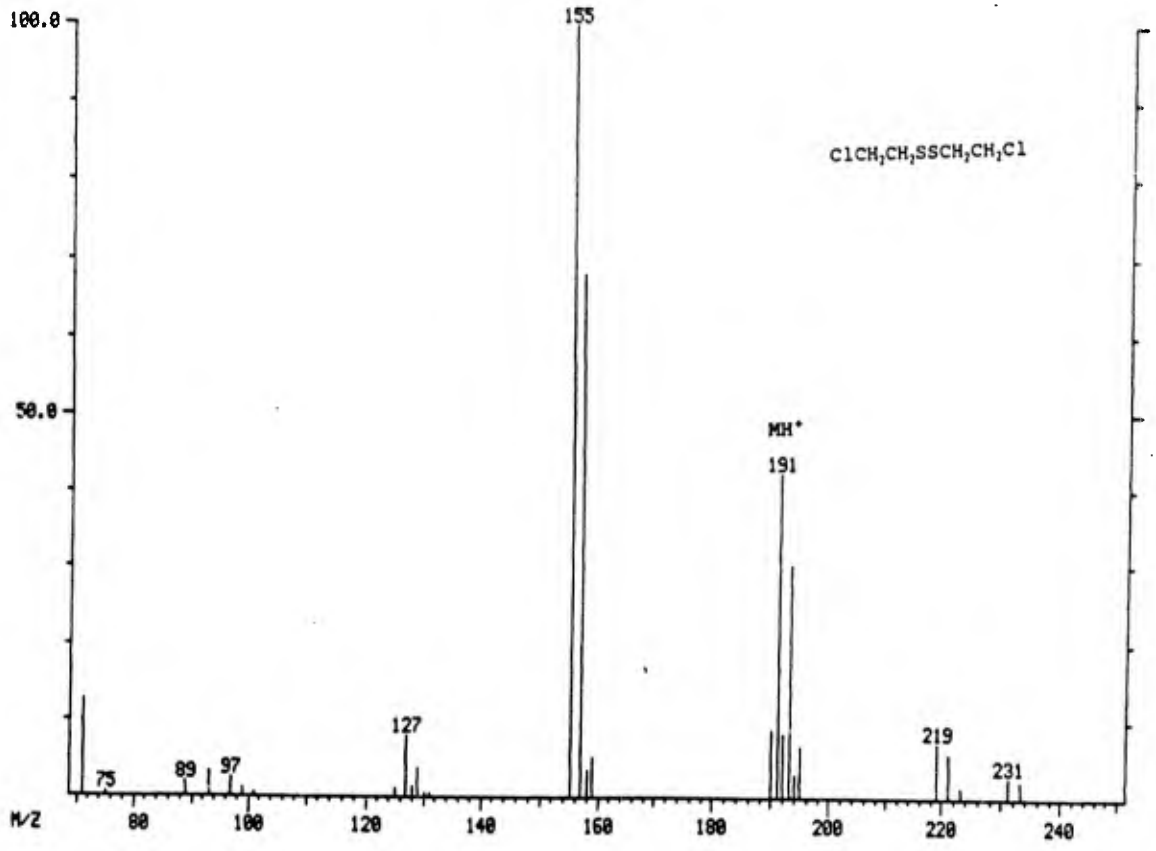
CI Mass Spectrum of OTH-1593-1c Scan 384



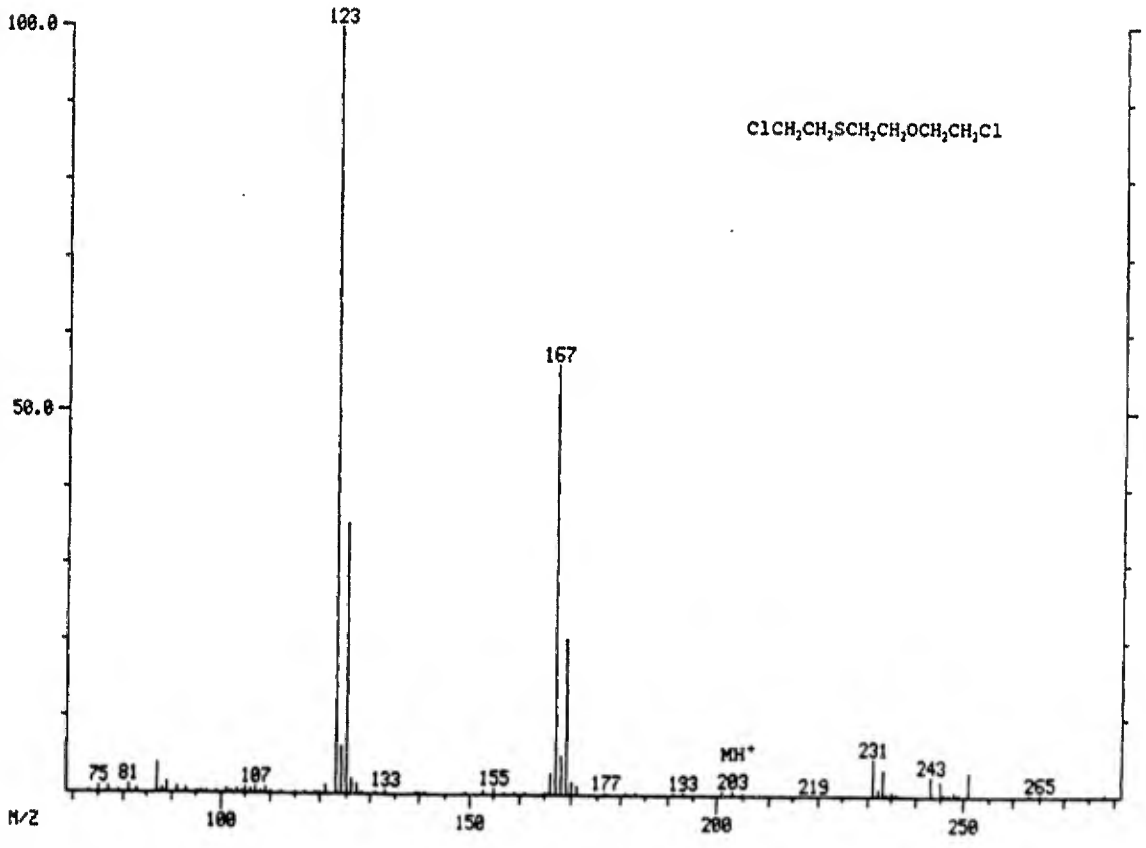
CI Mass Spectrum of OTH-1593-1c Scan 410



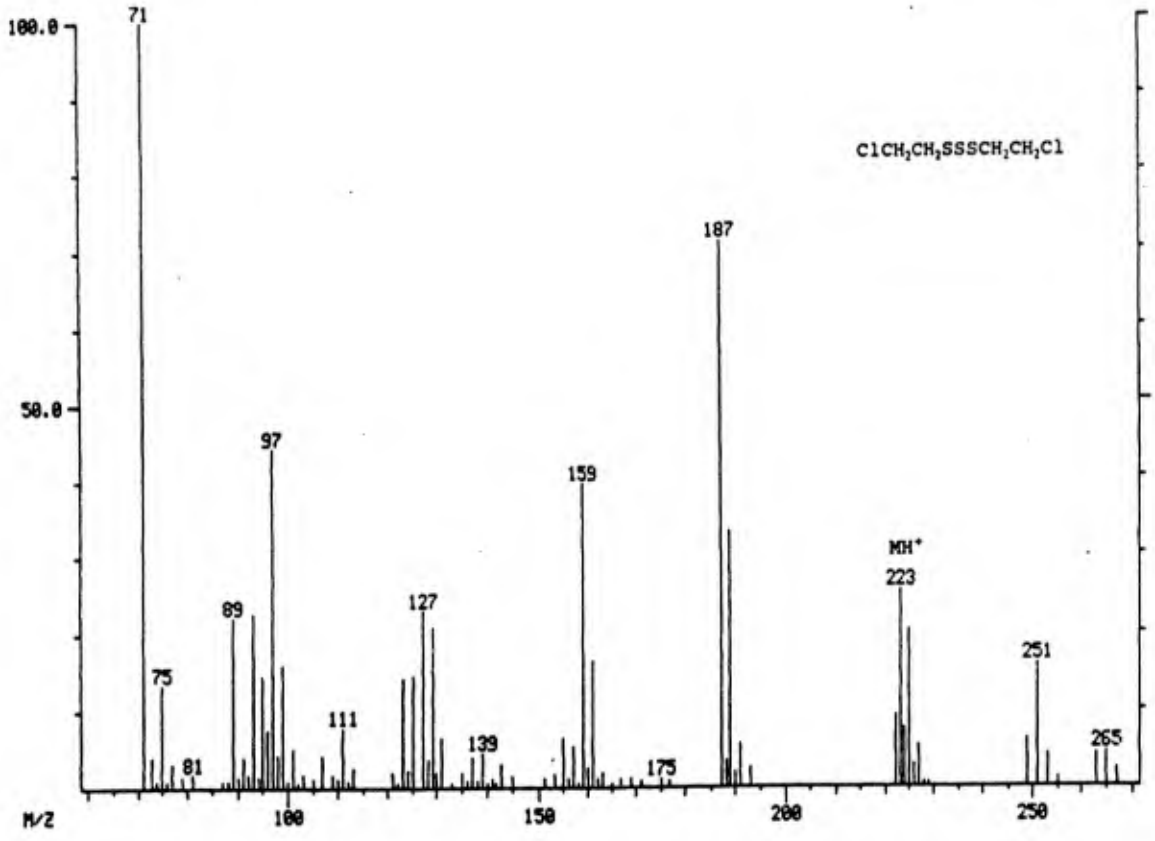
CI Mass Spectrum of OTH-1593-1c Scan 443



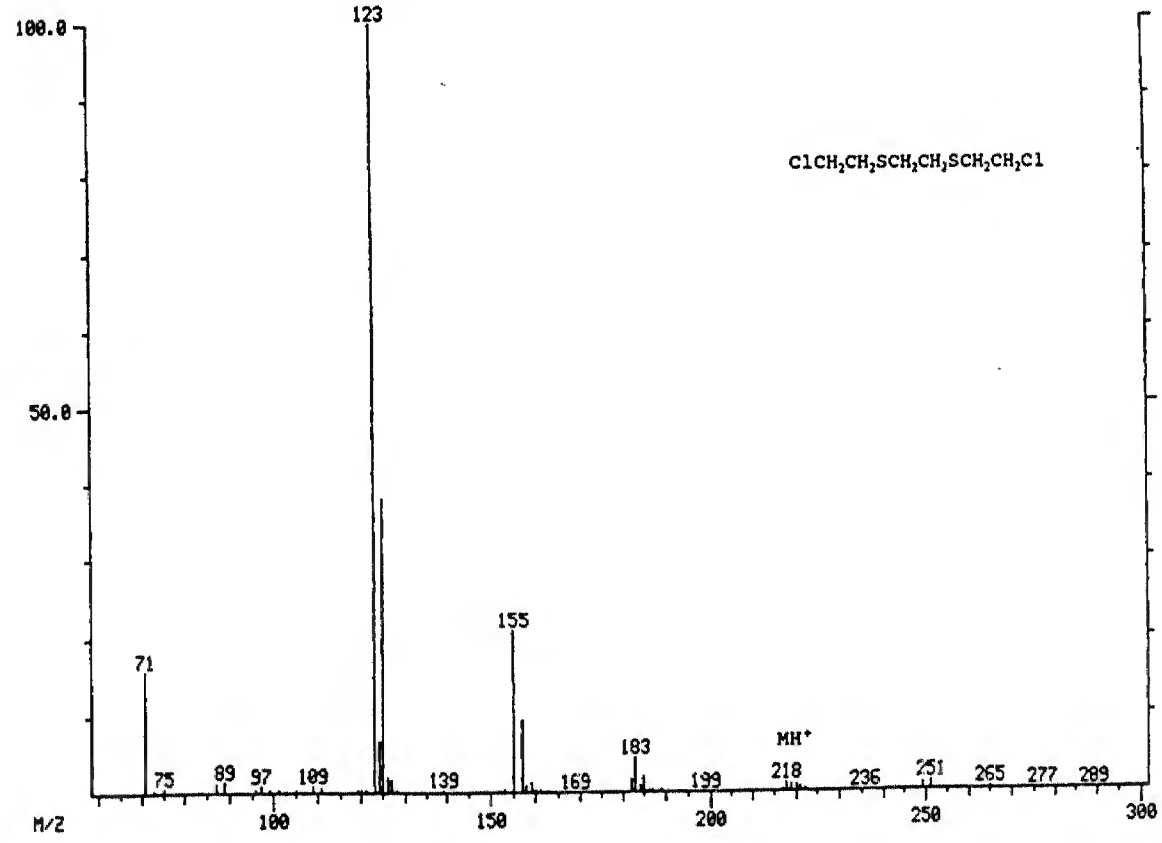
CI Mass Spectrum of OTH-1593-1c Scan 503



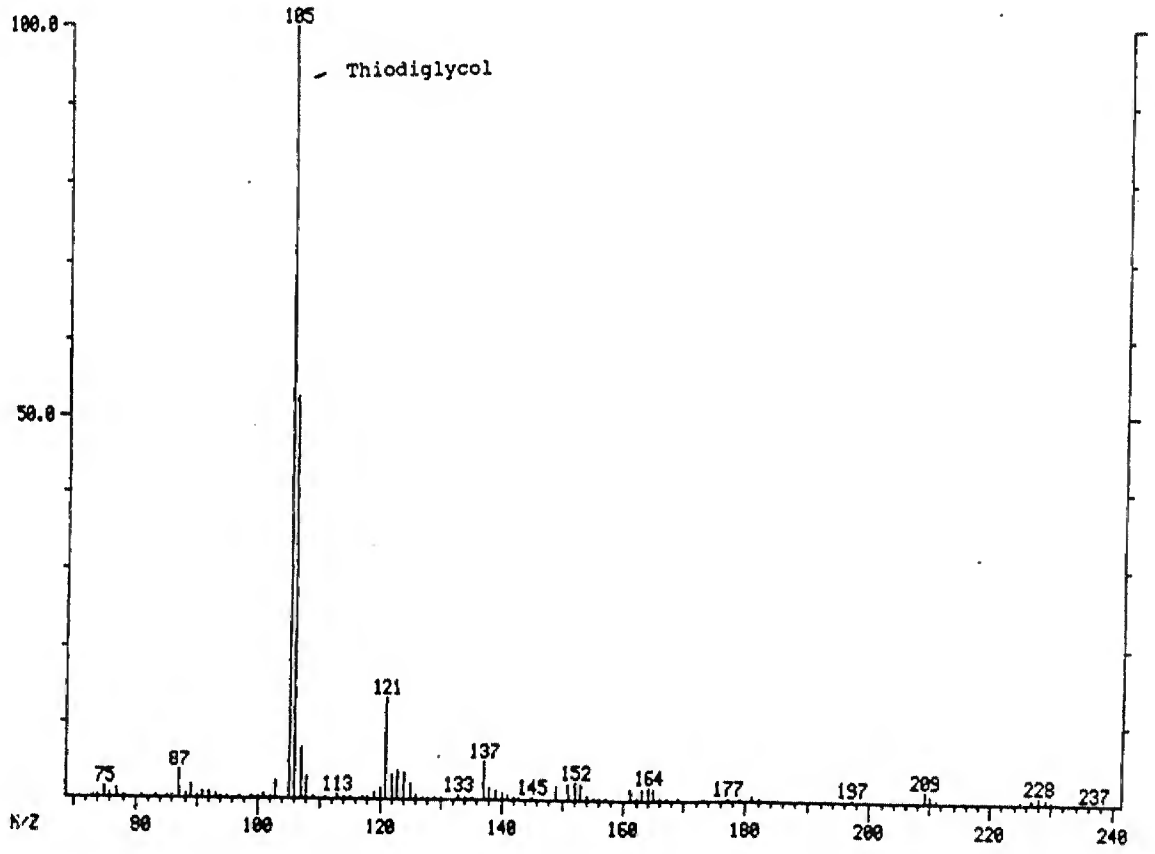
CI Mass Spectrum of OTH-1593-1c Scan 619



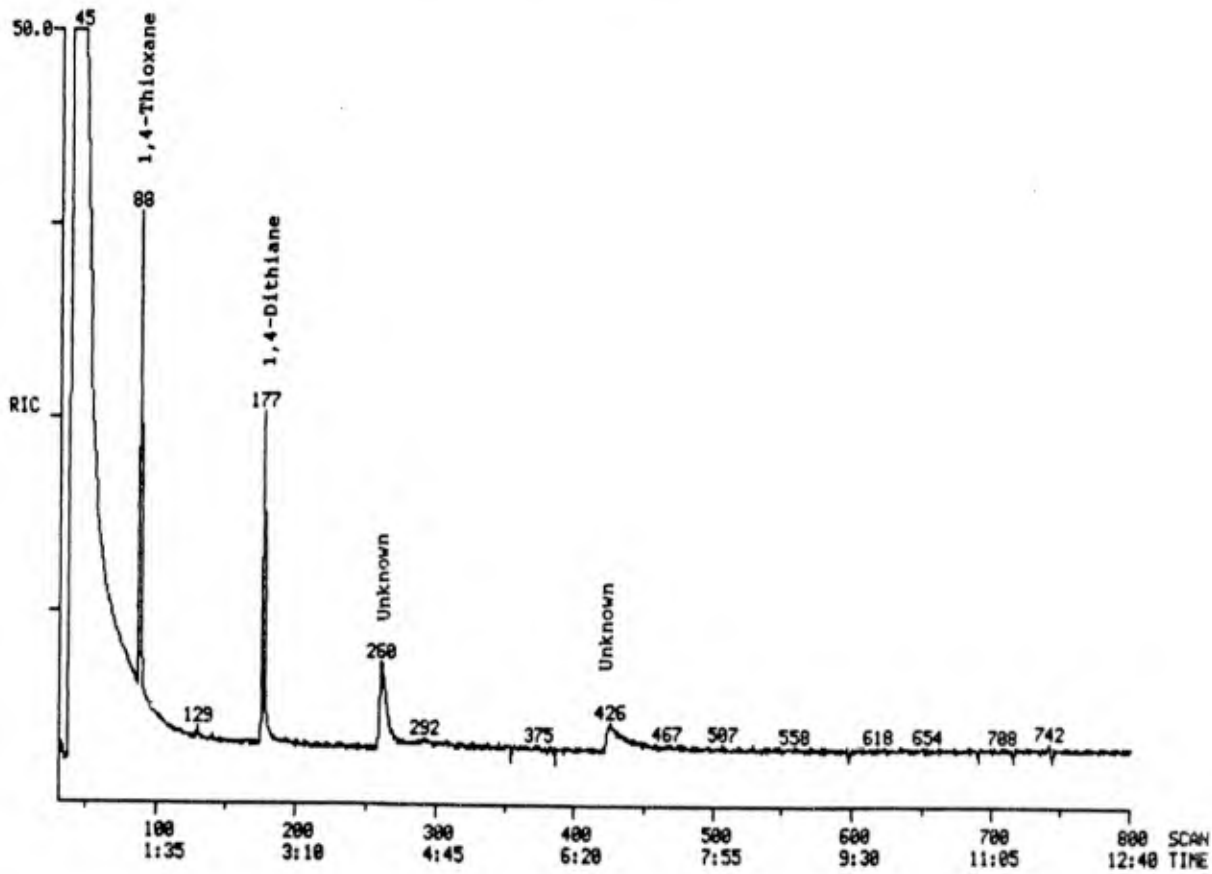
CI Mass Spectrum of OTH-1593-1c Scan 667



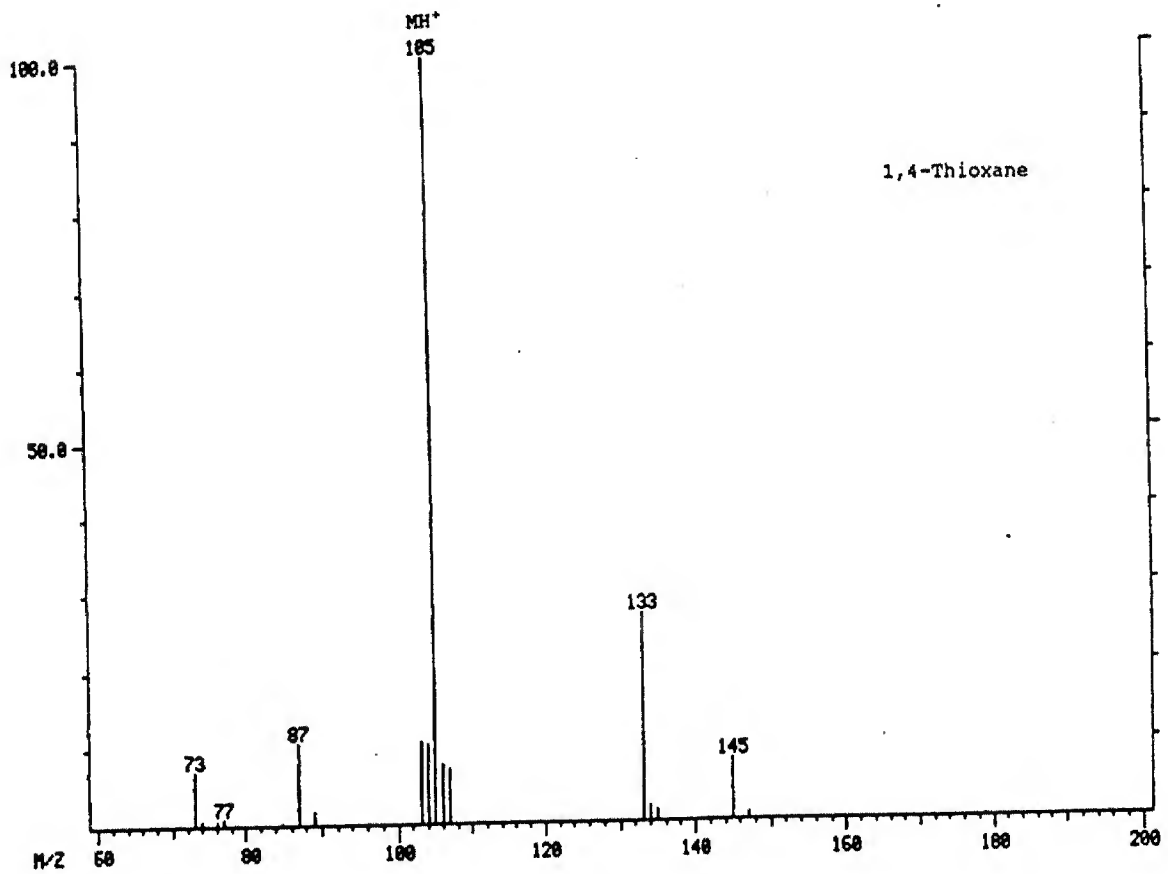
DEP/CI Mass Spectrum of OTH-1693-1c



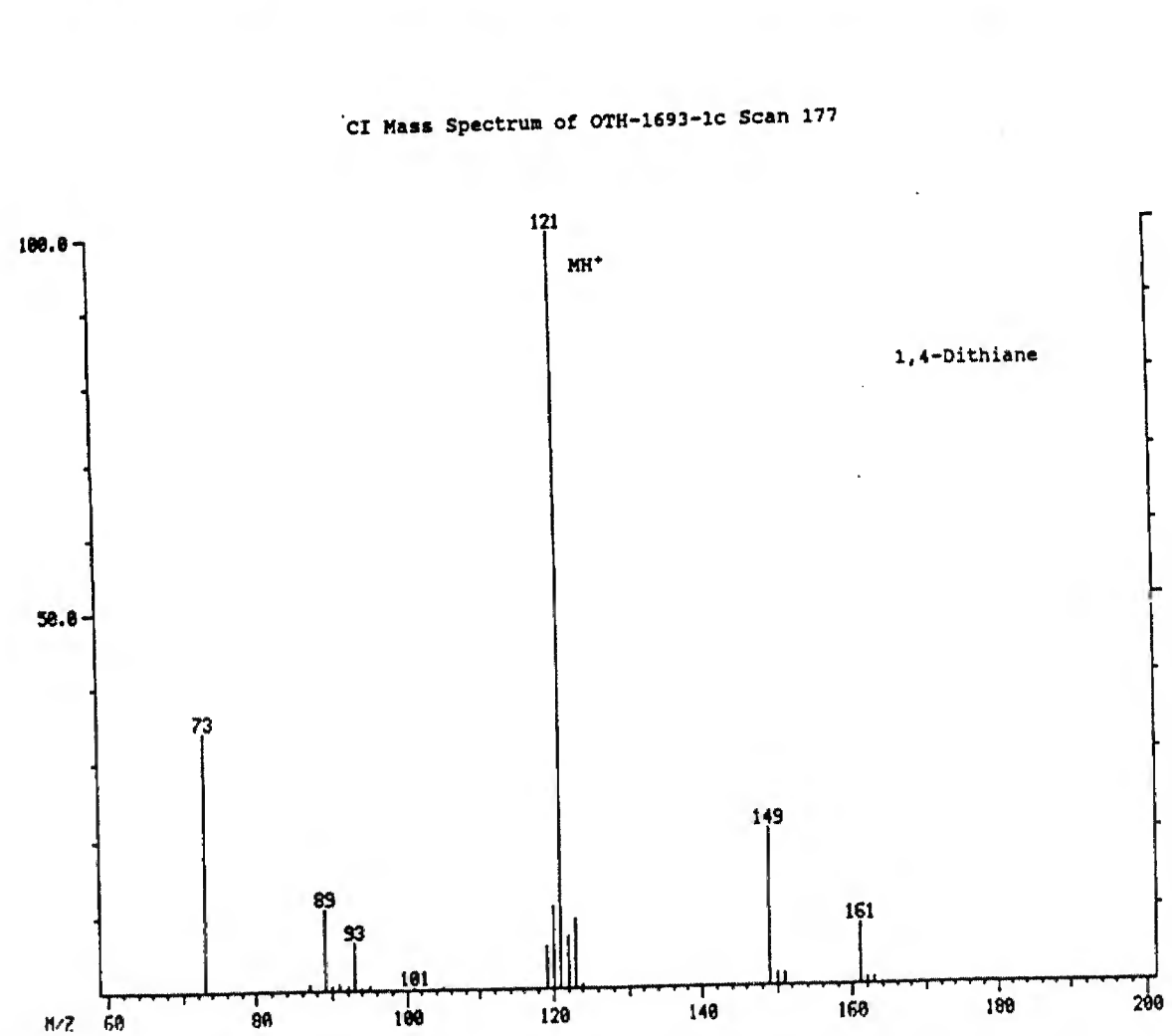
GC/MS/CI Chromatogram of OTH-1693-1c



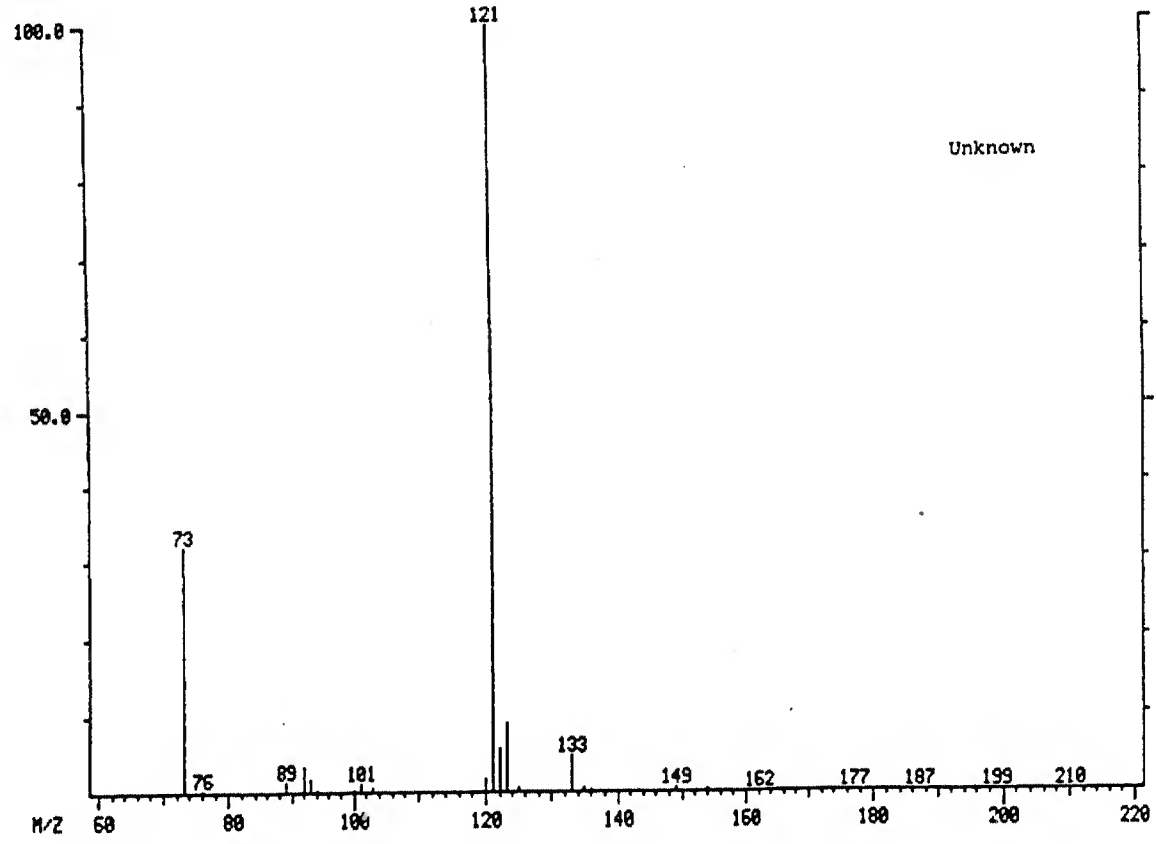
CI Mass Spectrum of OTH-1693-1c Scan 88



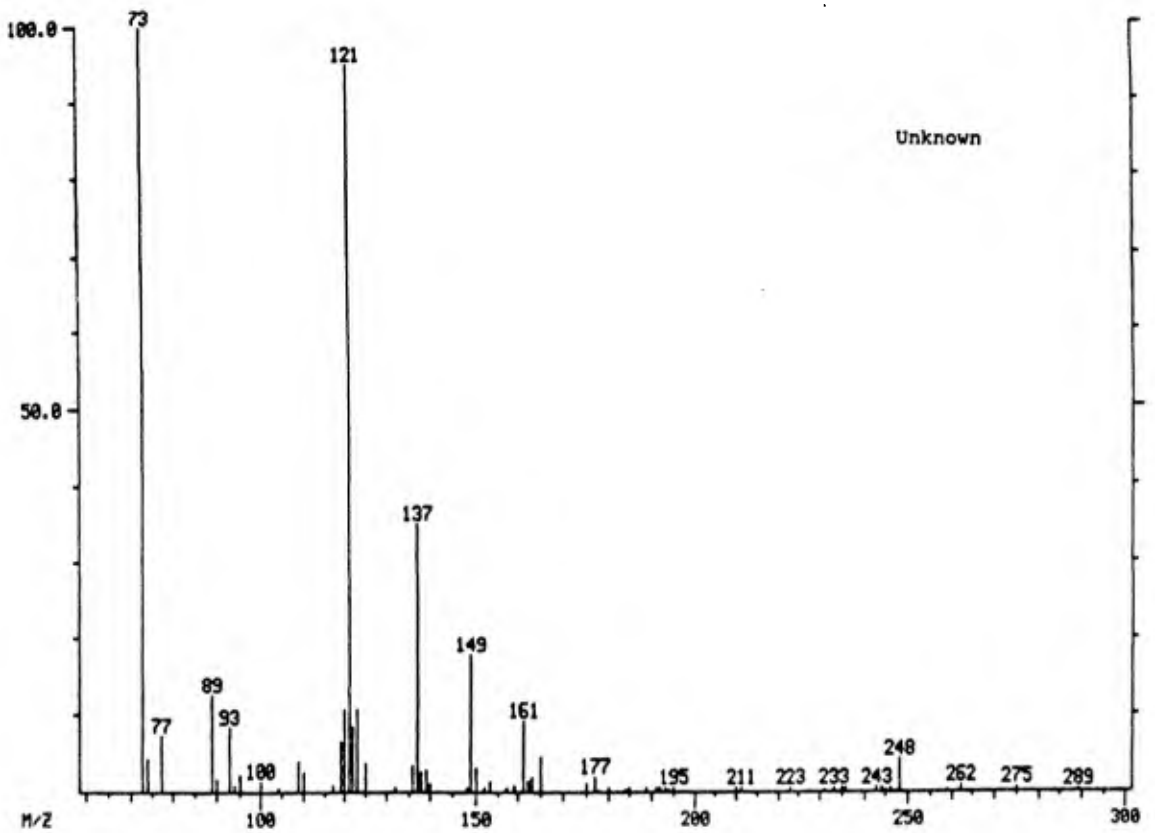
CI Mass Spectrum of OTH-1693-1c Scan 177



CI Mass Spectrum of OTH-1693-1c Scan 260



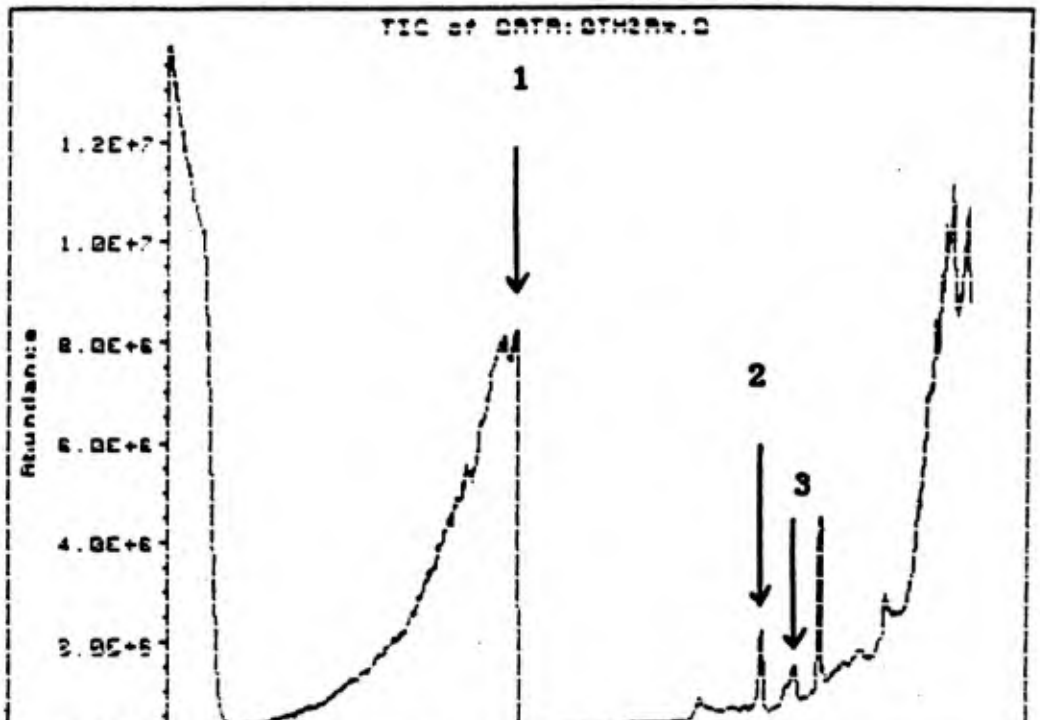
CI Mass Spectrum of OTH-1693-1c Scan 426



APPENDIX E

GC/MS/EI AND DEP/EI SPECTRA

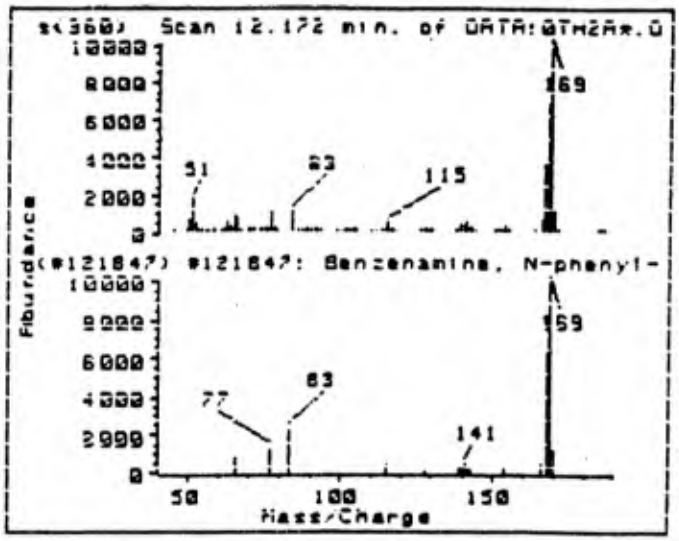
Sample OTH-193-2a



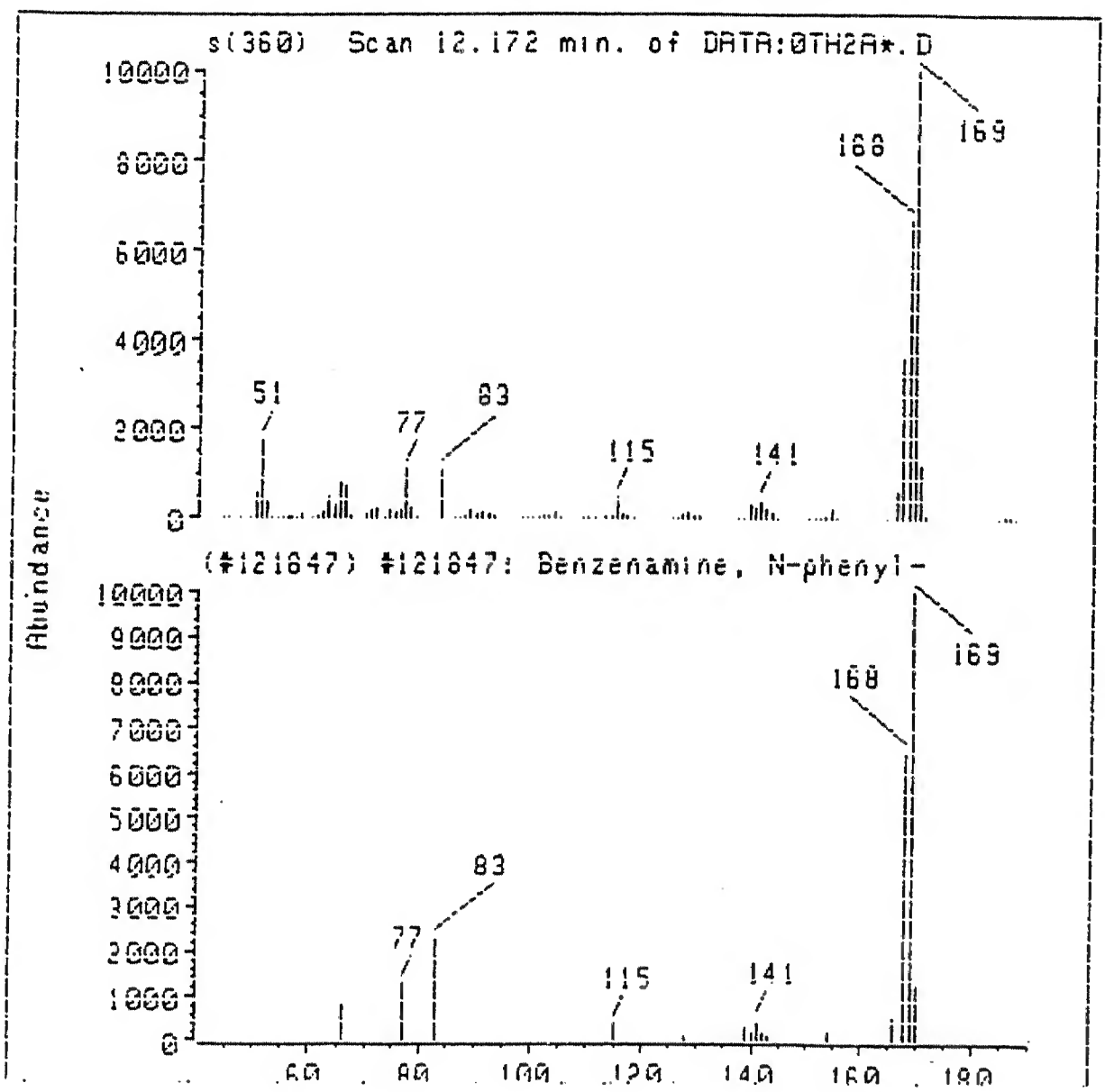
Sample OTH-193-2a

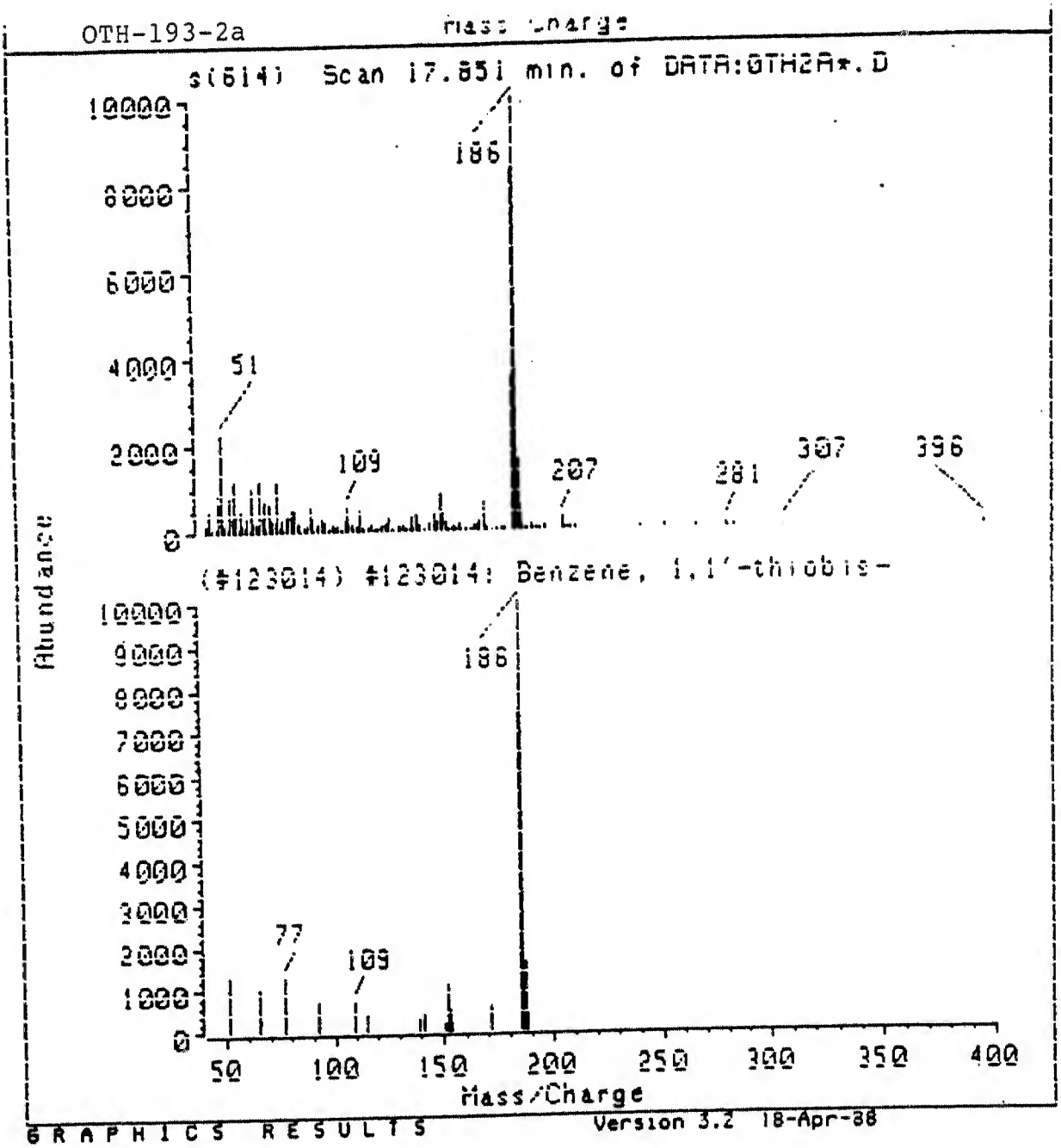
1. **Diphenylamine**
2. **Diphenyl sulfide**
3. **1,1-biphenyl-4-methyl**

OTH-193-2a



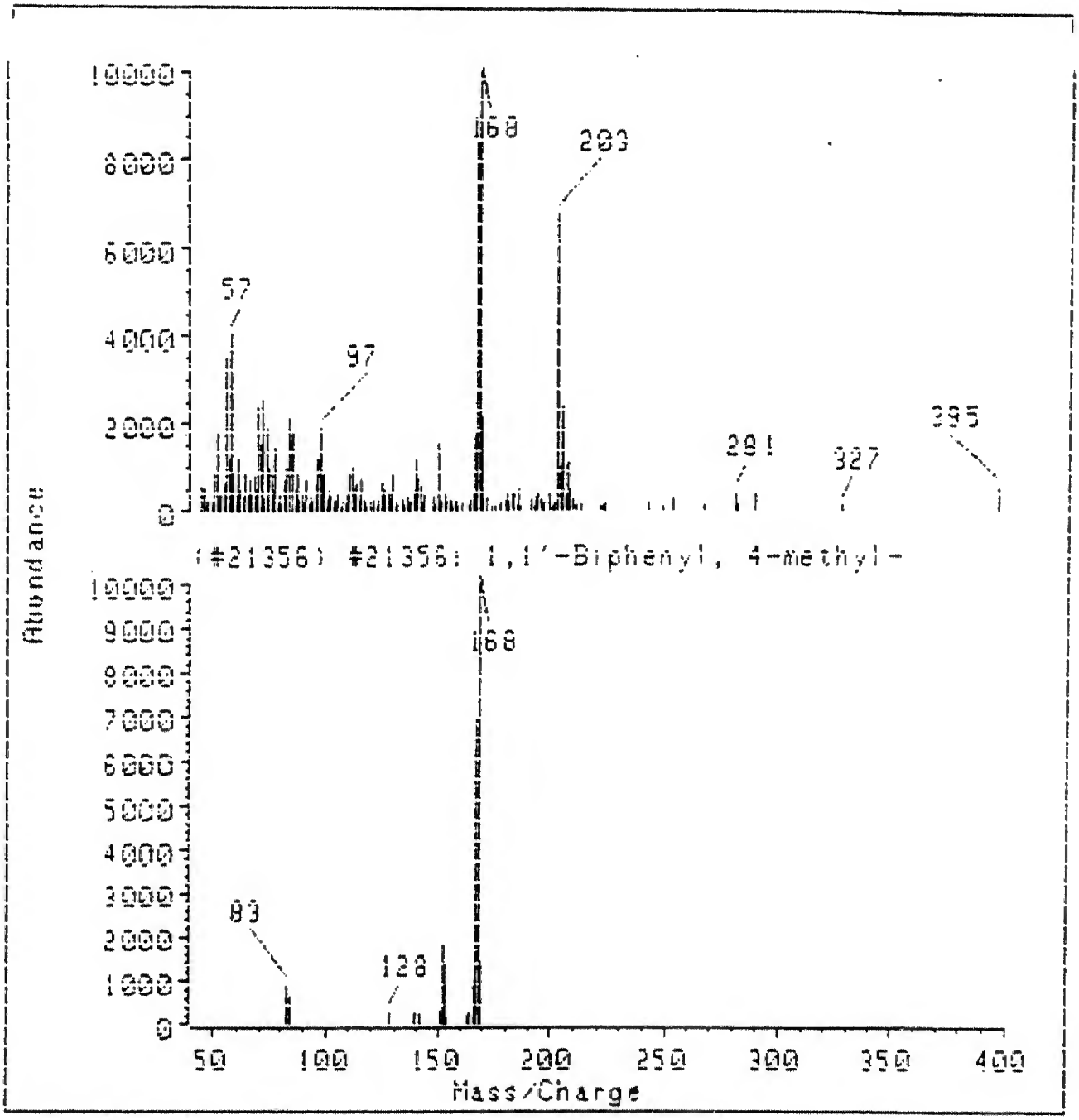
Diphenylamine



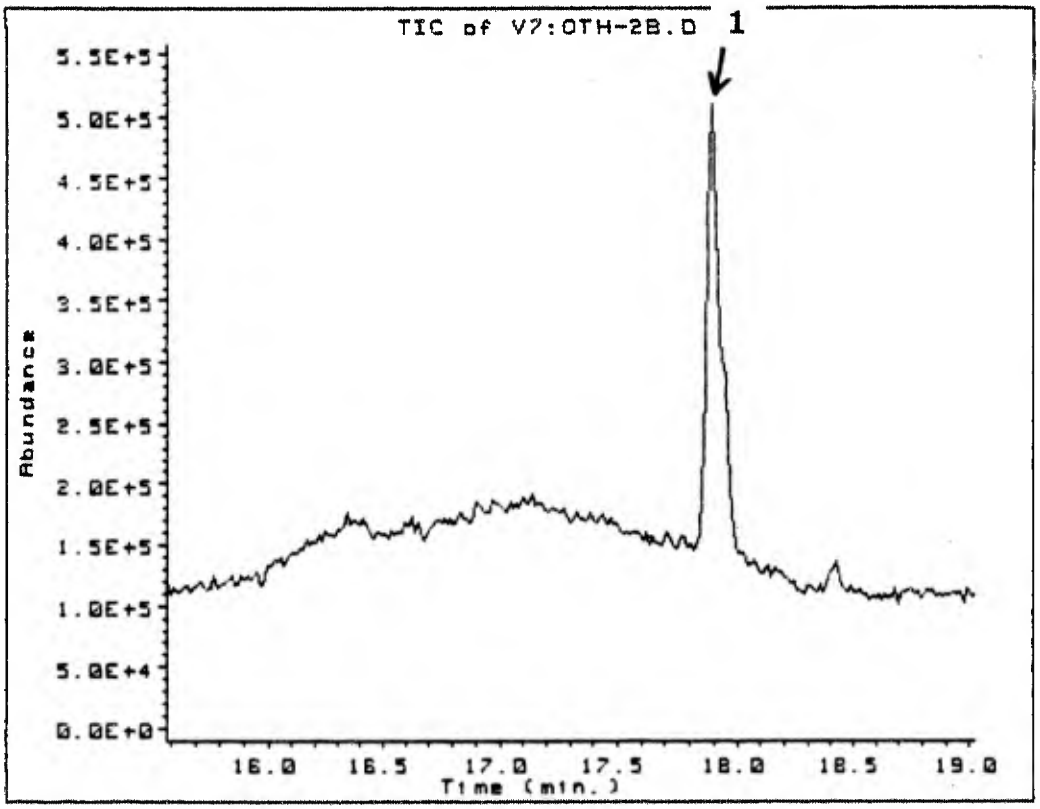


Benzene, 1,1'-thiobi
000139-66-2 97
Benzene, 1,1'-thiobi
000139-66-2 96
Benzene, 1,1'-thiobi
000139-66-2 70
Sulfadiazine
000068-35-9 42
TRIMETHYLSILYL ESTER
27
1,4-Naphthalenedione
002197-57-1 27
[1,1'-Biphenyl]-4,4'
000092-88-6 27
P-PHENOXYPHENYL ESTE
27
Phenol, phenoxy-
05474-79-7 25

Diphenyl sulfide


Abundance
10000
8000
6000
4000
2000
0
57
97
168
203
281
327
395
(#21356) #21356: 1,1'-Biphenyl, 4-methyl-
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
83
128
168
50
100
150
200
250
300
350
400
Mass/Charge

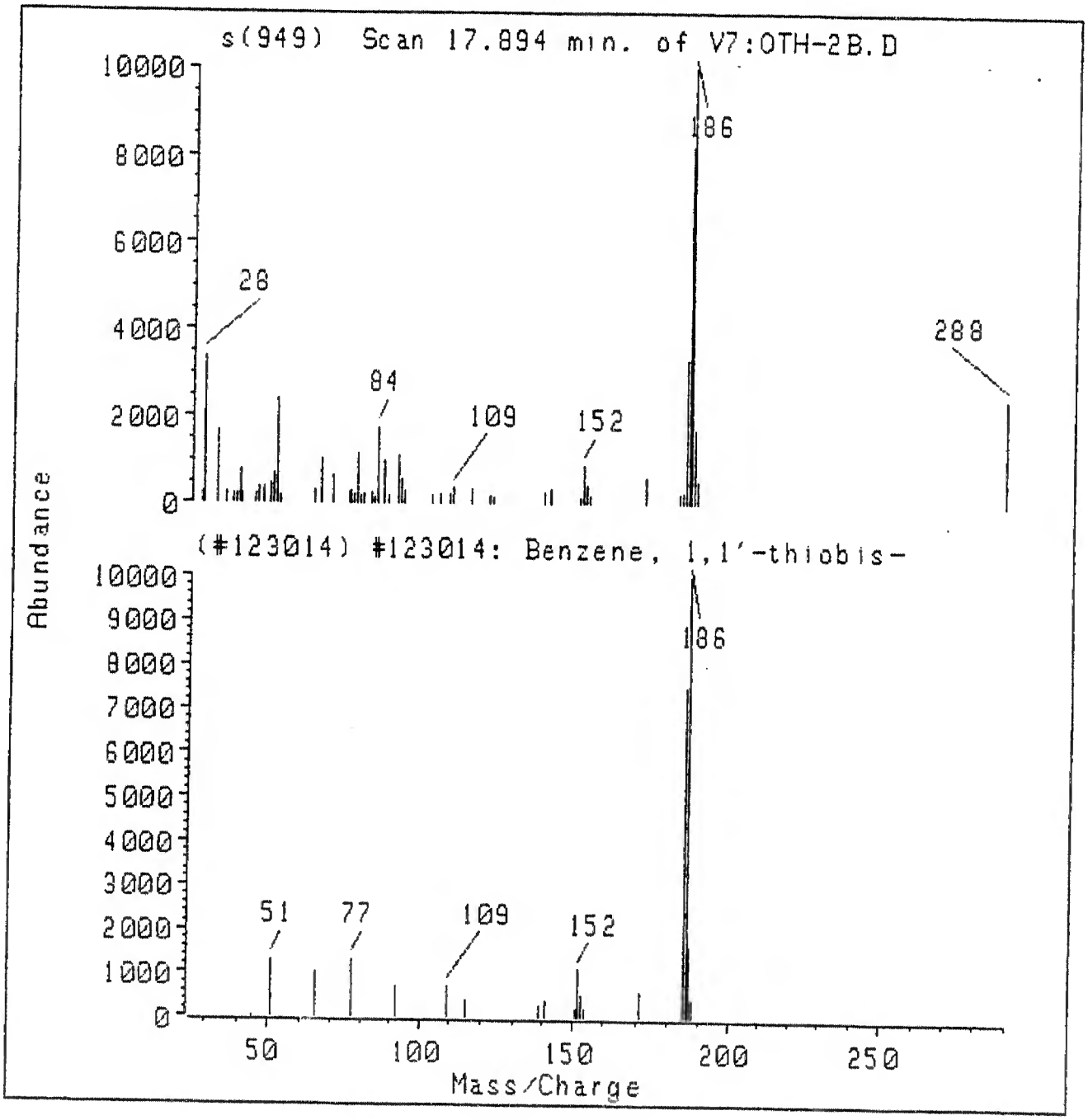
Sample OTH-193-2b



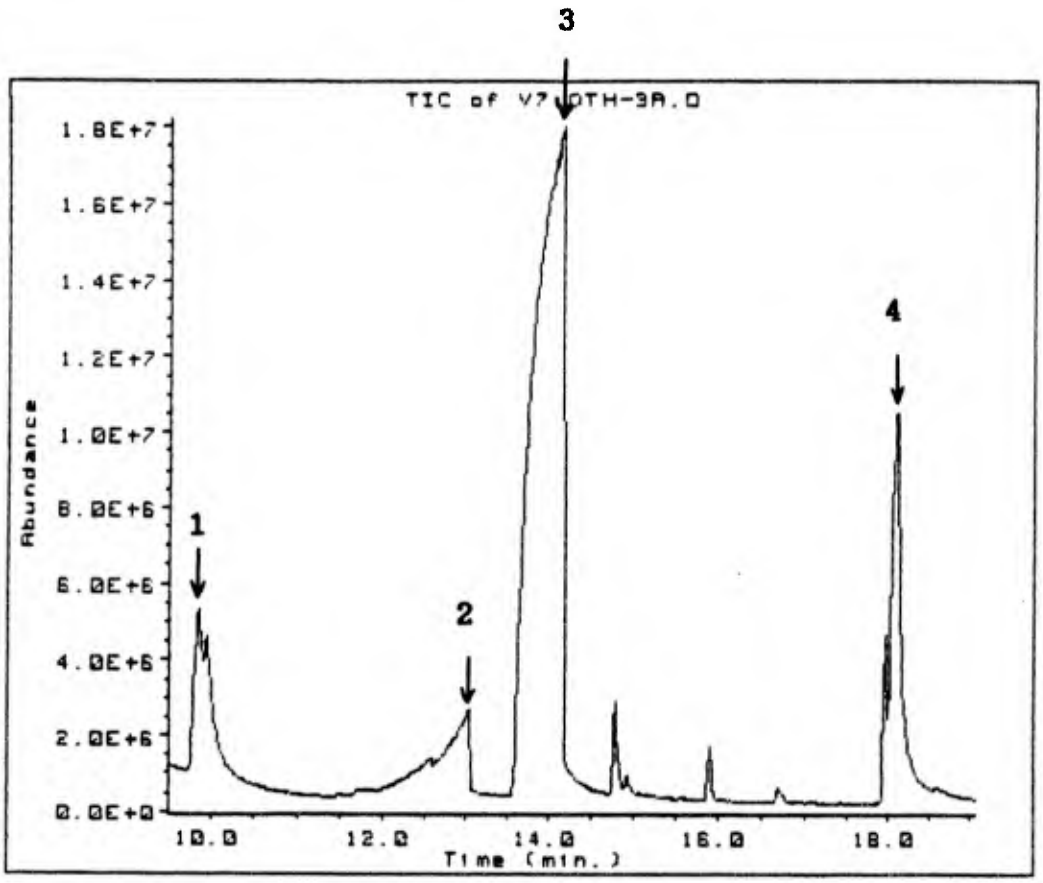
1. Diphenyl sulfide

Diphenyl sulfide

OTH-193-2b



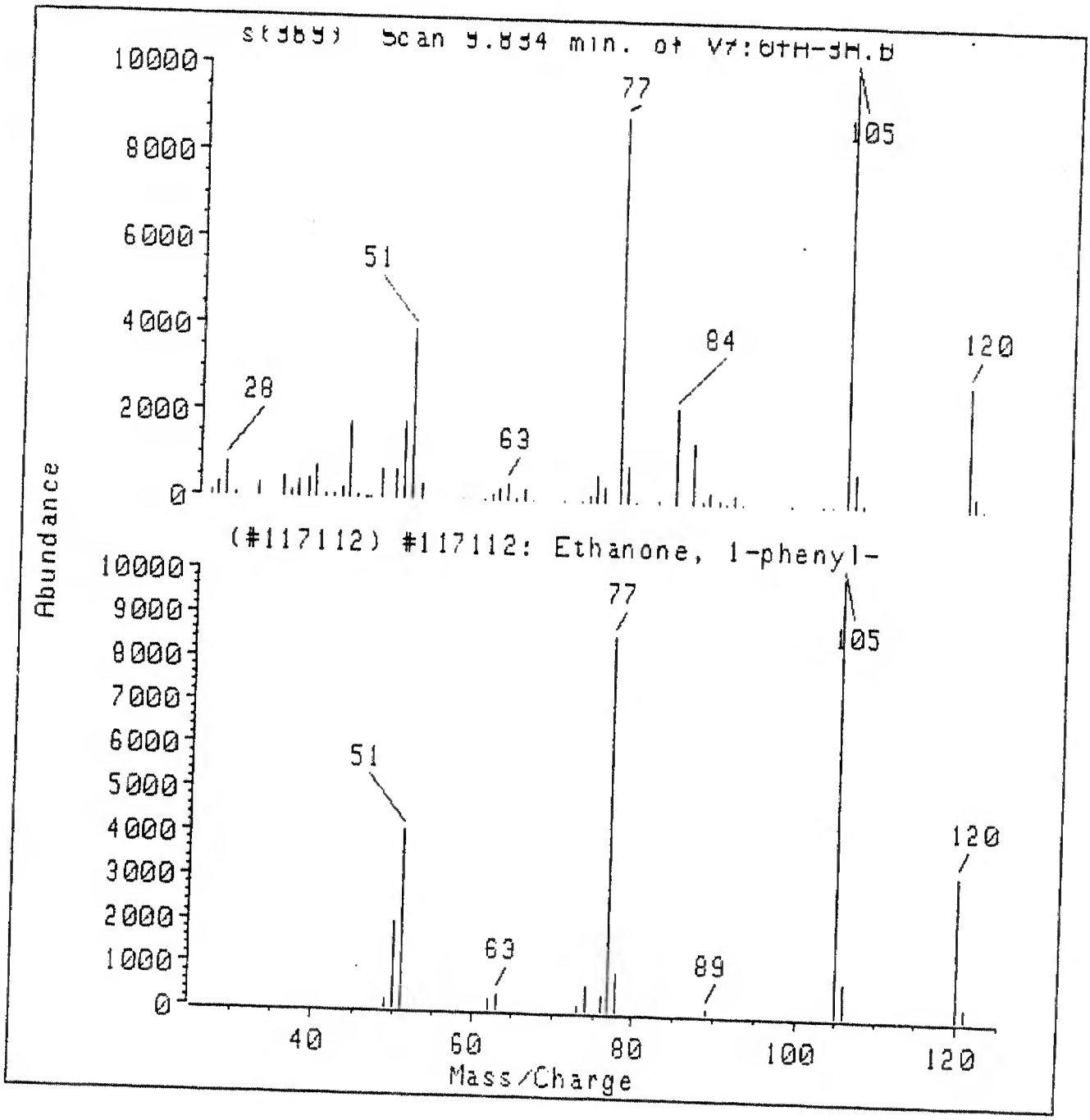
Sample OTH-193-3a



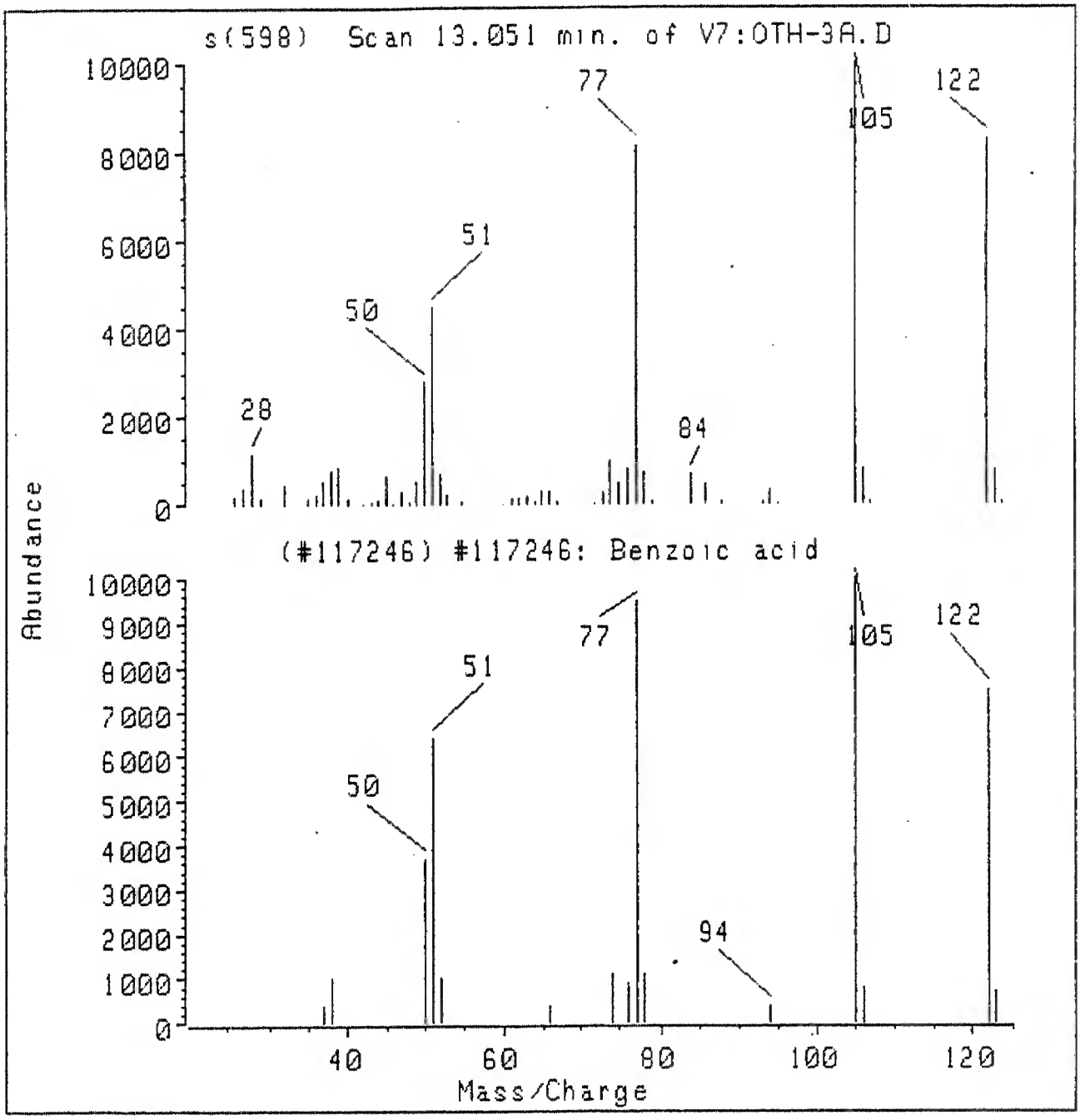
1. Acetophenone
2. Benzoic acid
3. Chloroacetophenone
4. Diphenyl sulfide

Acetophenone

OTH-193-3a

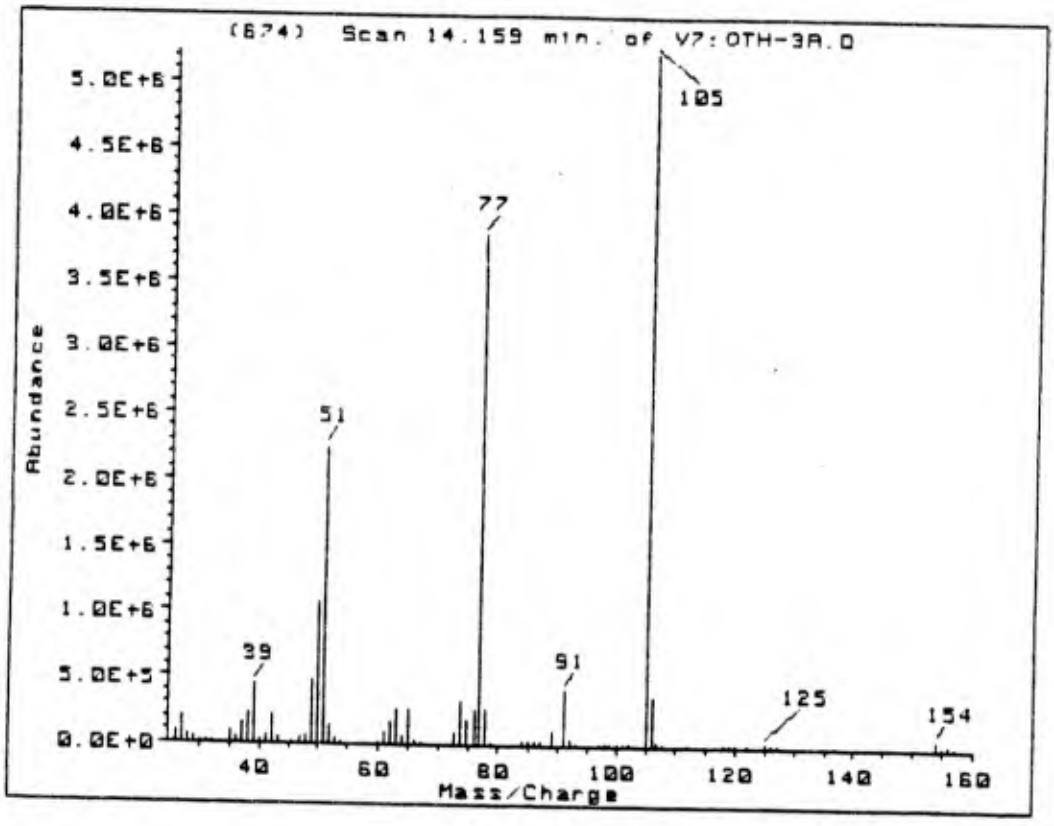


OTH-193-3a


s(598) Scan 13.051 min. of V7:OTH-3A.D
10000
8000
6000
4000
2000
0
77
105
122
51
50
28
84
(#117246) #117246: Benzoic acid
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
77
105
122
51
50
94
Abundance
40
60
80
100
120
Mass/Charge

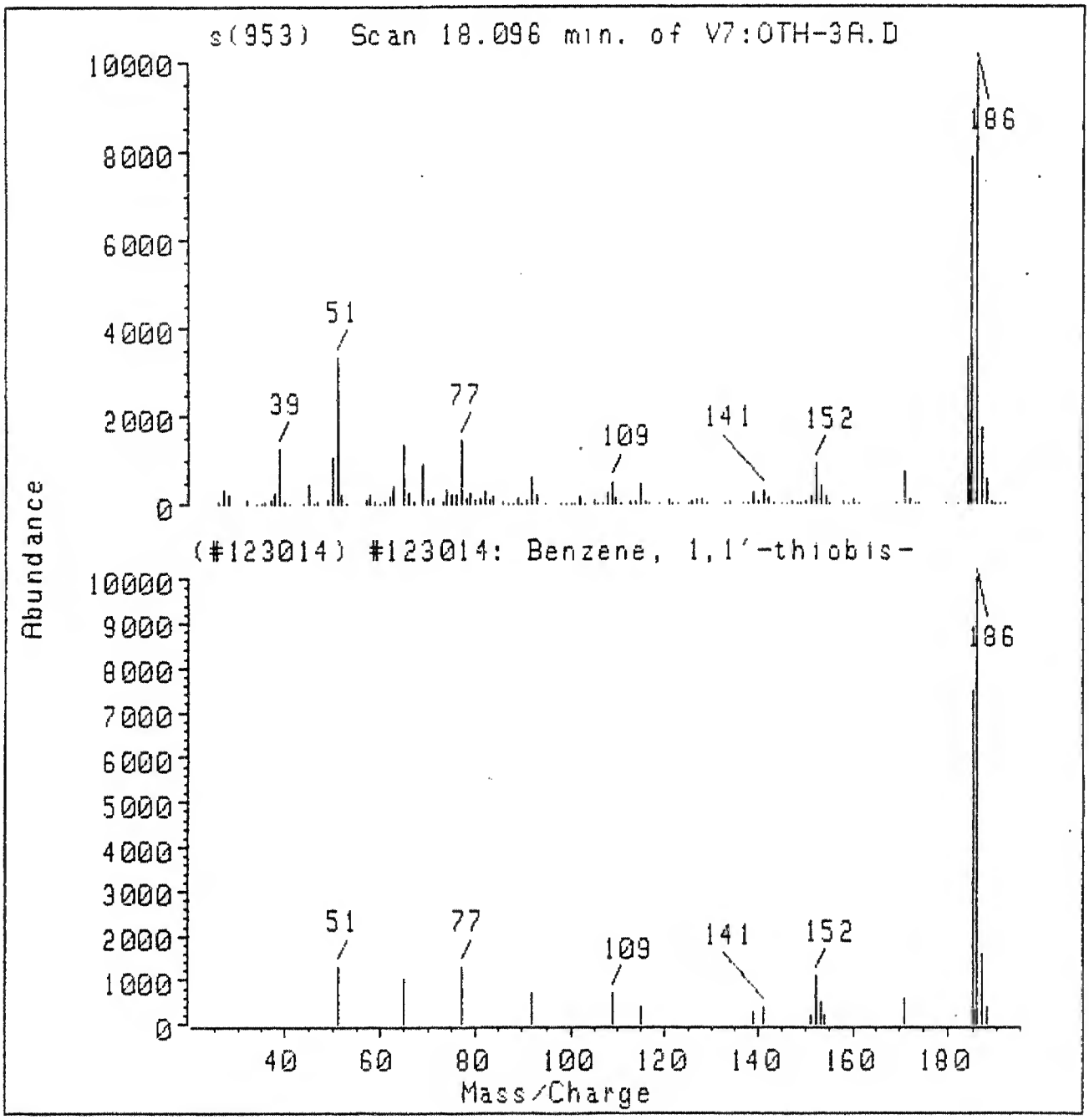
Chloroacetophenone

OTH-193-3a

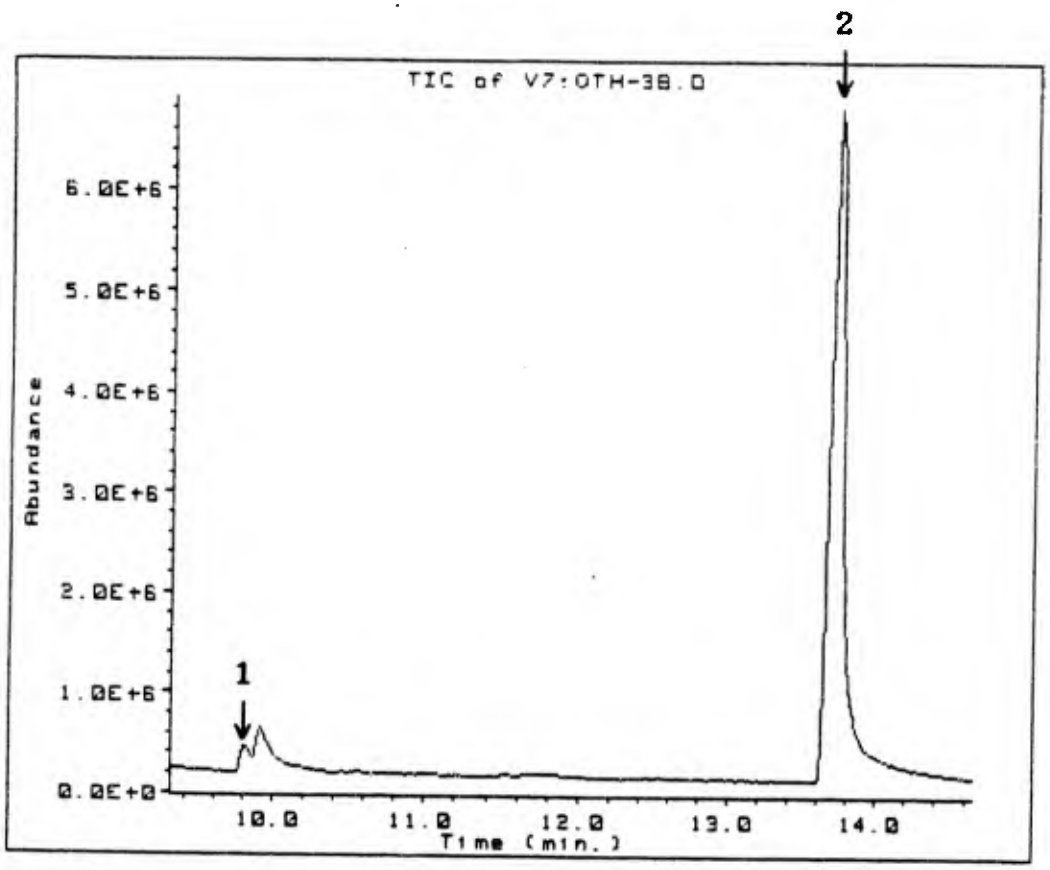


Diphenyl sulfide

OTH-193-3a



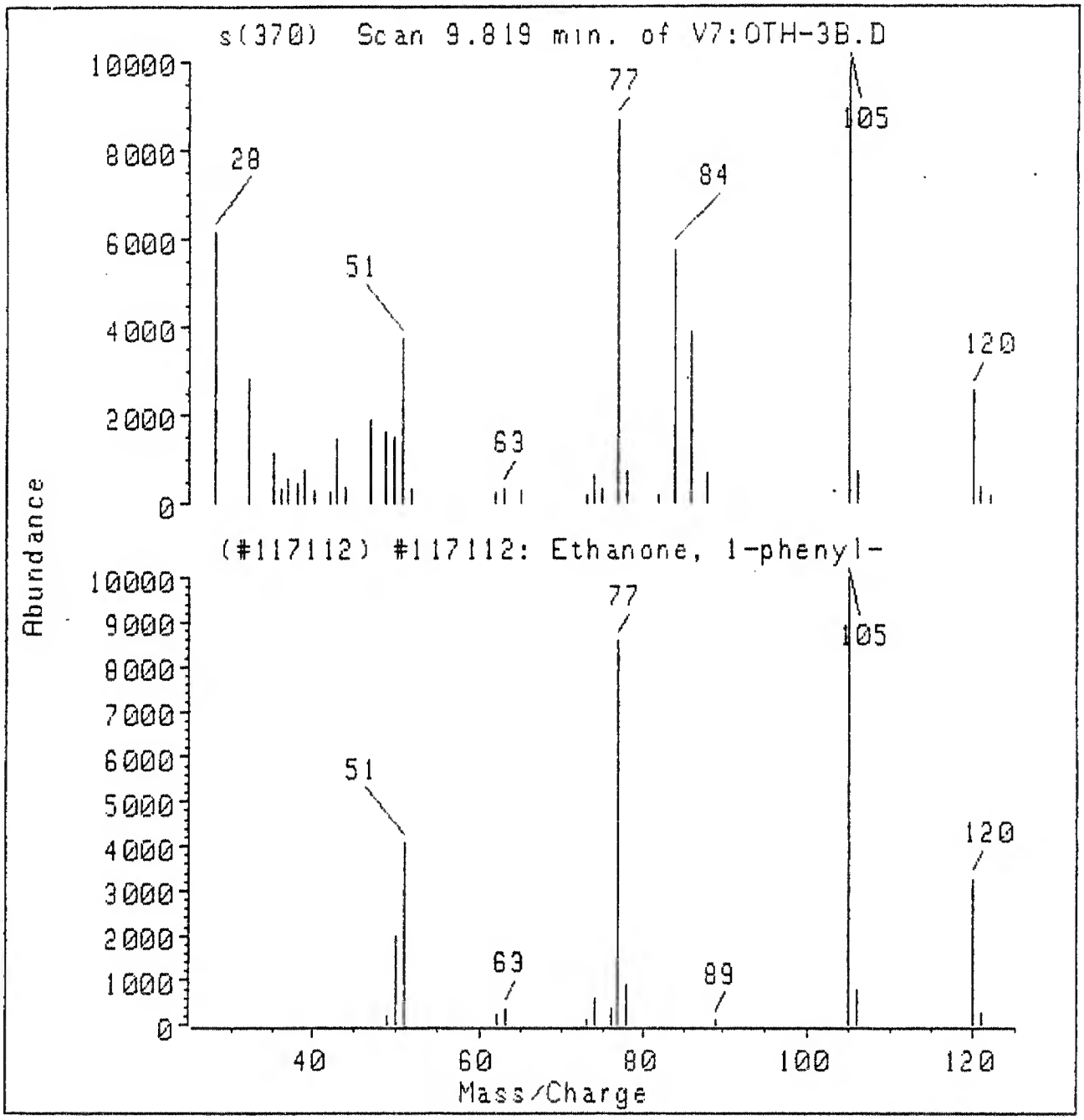
Sample OTH-193-3b



1. Acetophenone
2. Chloroacetophenone

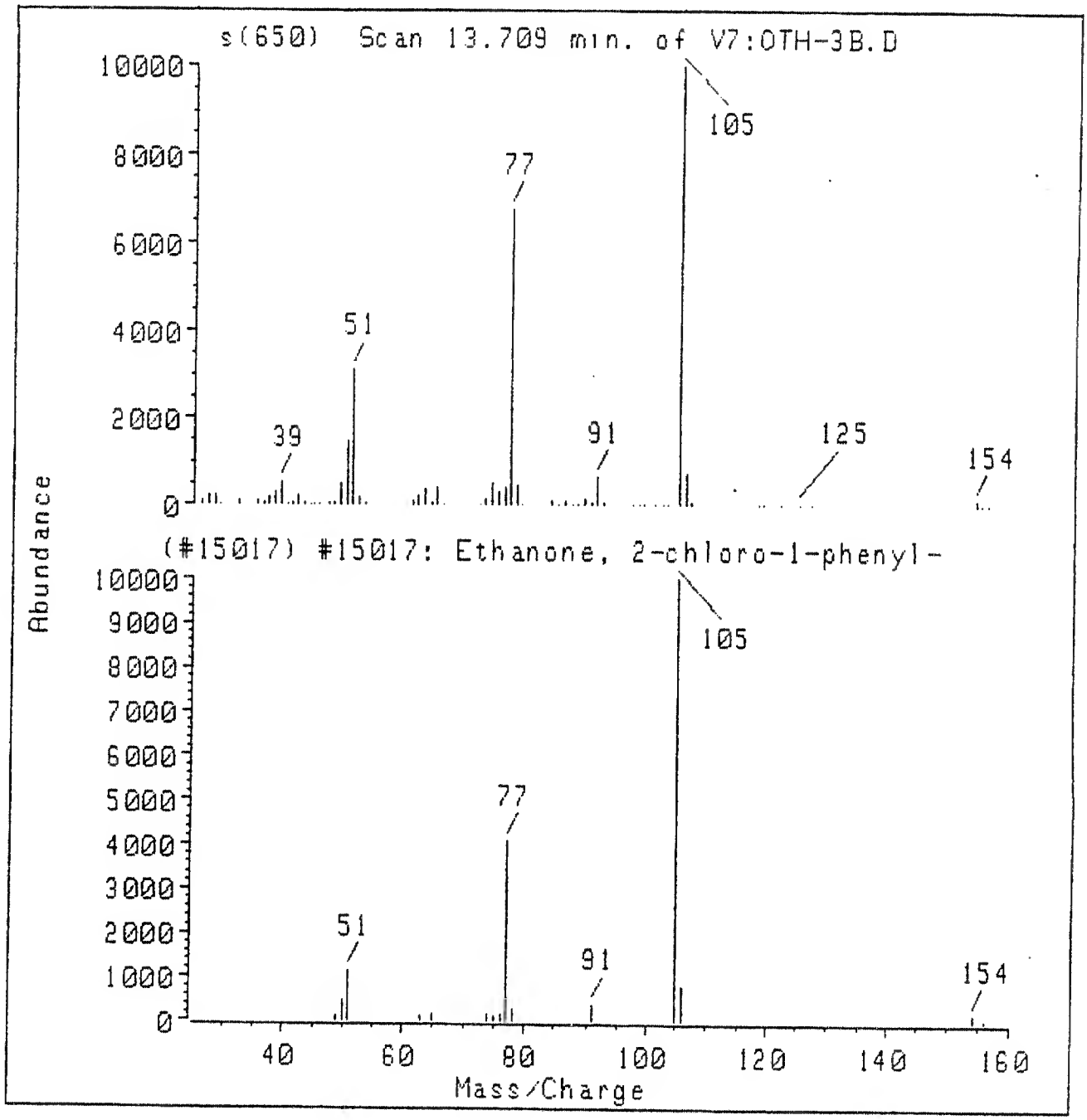
Acetophenone

OTH-193-3b

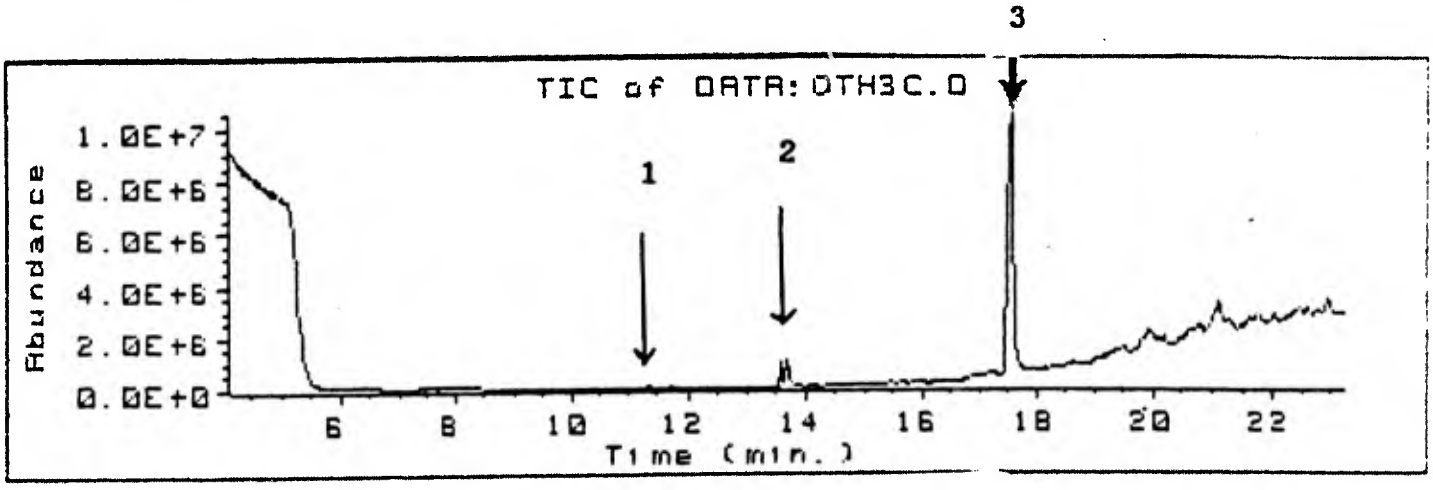


Chloroacetophenone

OTH-193-3b

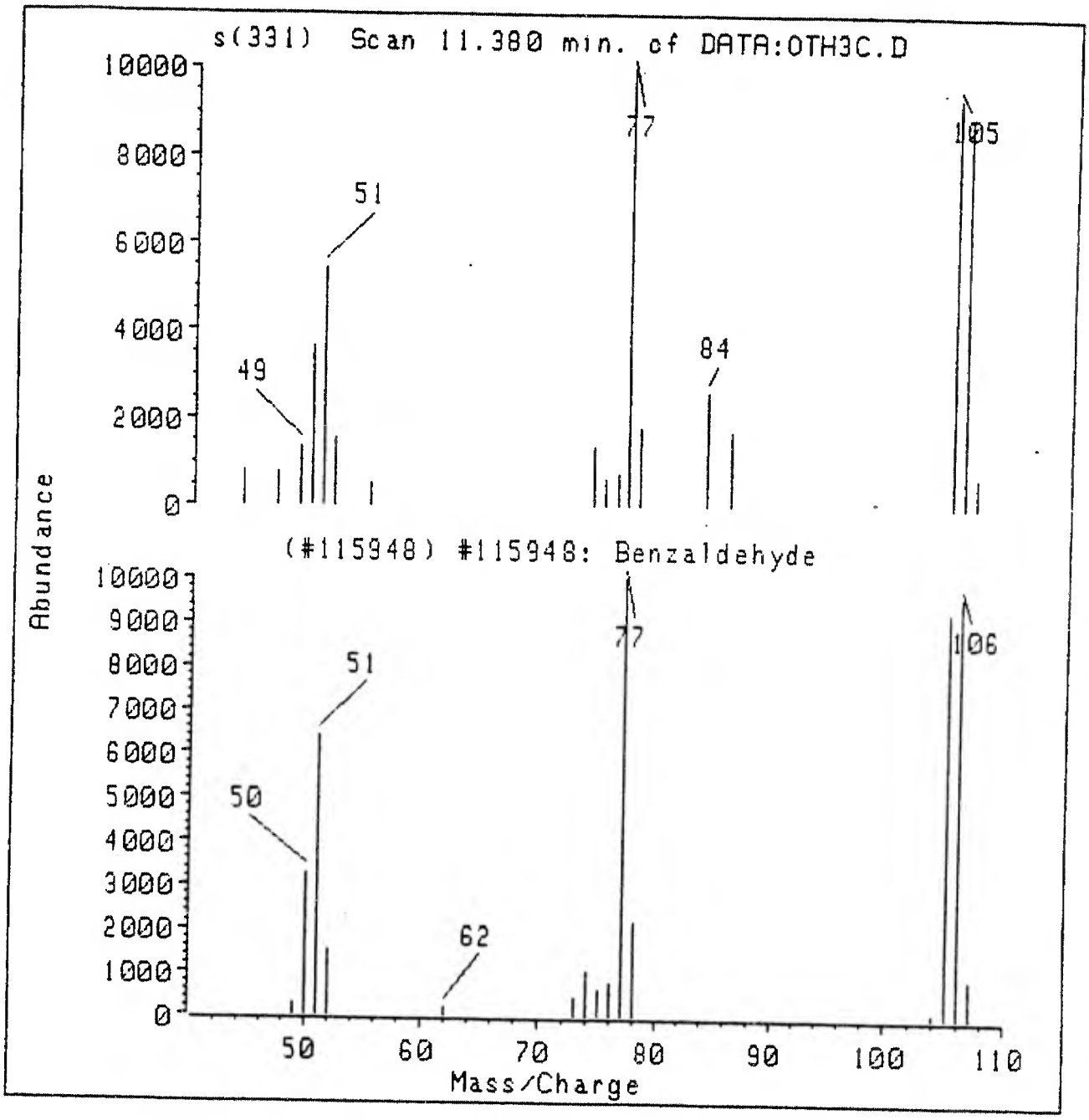


Sample OTH-193-3c

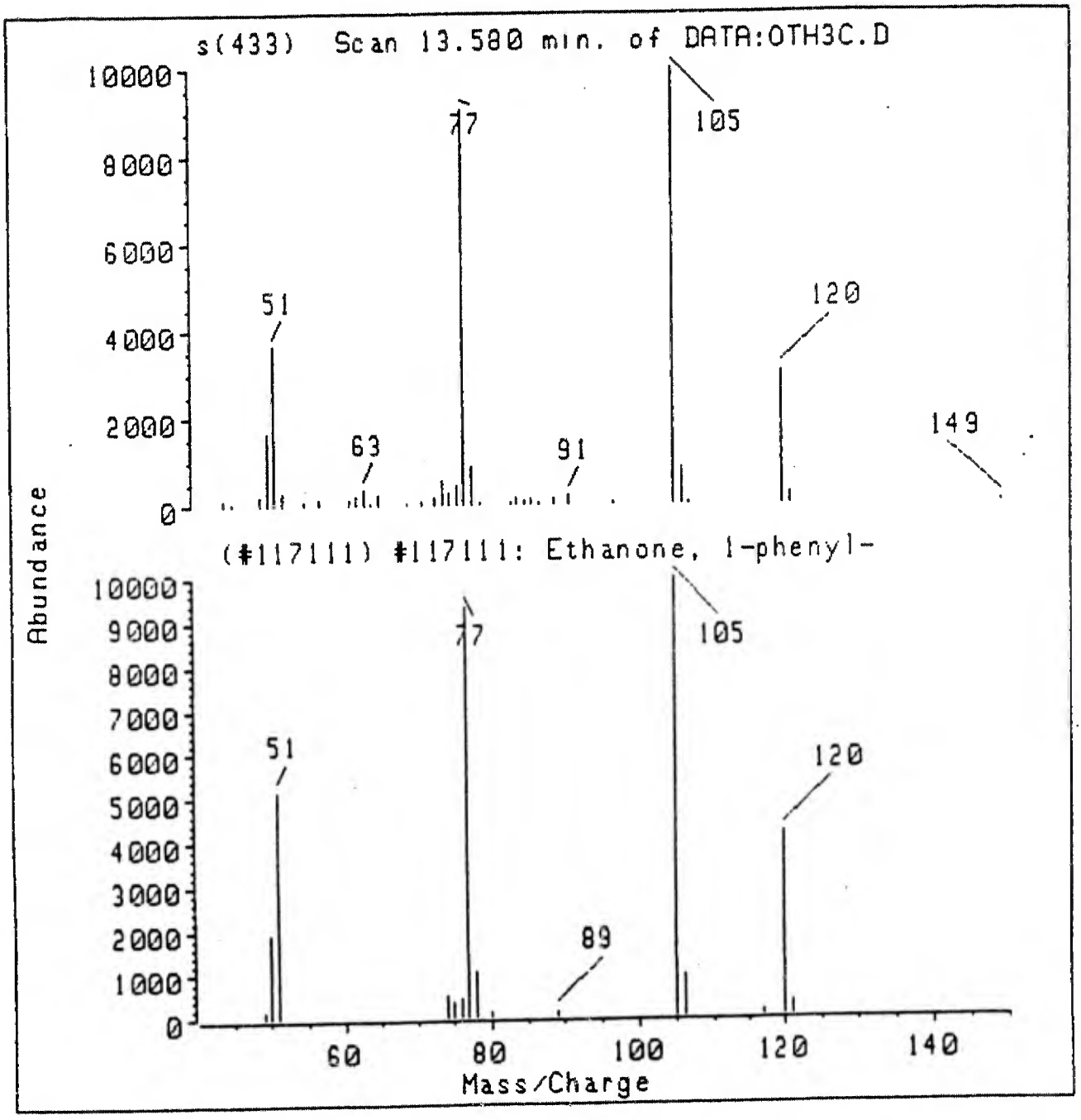


Sample OTH-193-3c

1. Benzaldehyde
2. Acetophenone
3. Chloroacetophenone


s(331) Scan 11.380 min. of DATA:OTH3C.D
10000
8000
6000
4000
2000
0
51
77
105
49
84
(#115948) #115948: Benzaldehyde
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
Abundance
51
77
106
50
62
50
60
70
80
90
100
110
Mass/Charge

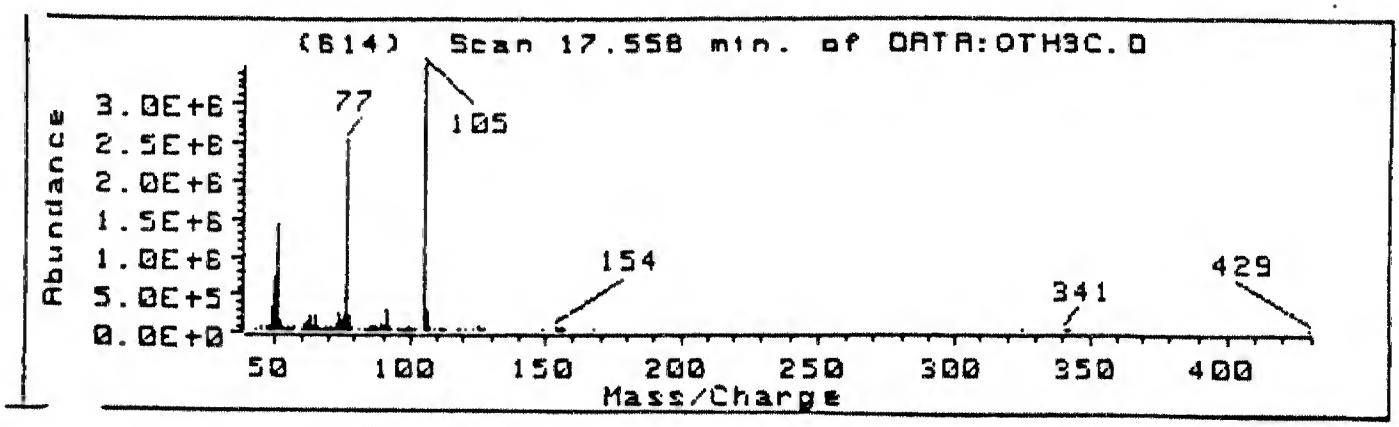
OTH-193-3c



OTH-193-3c

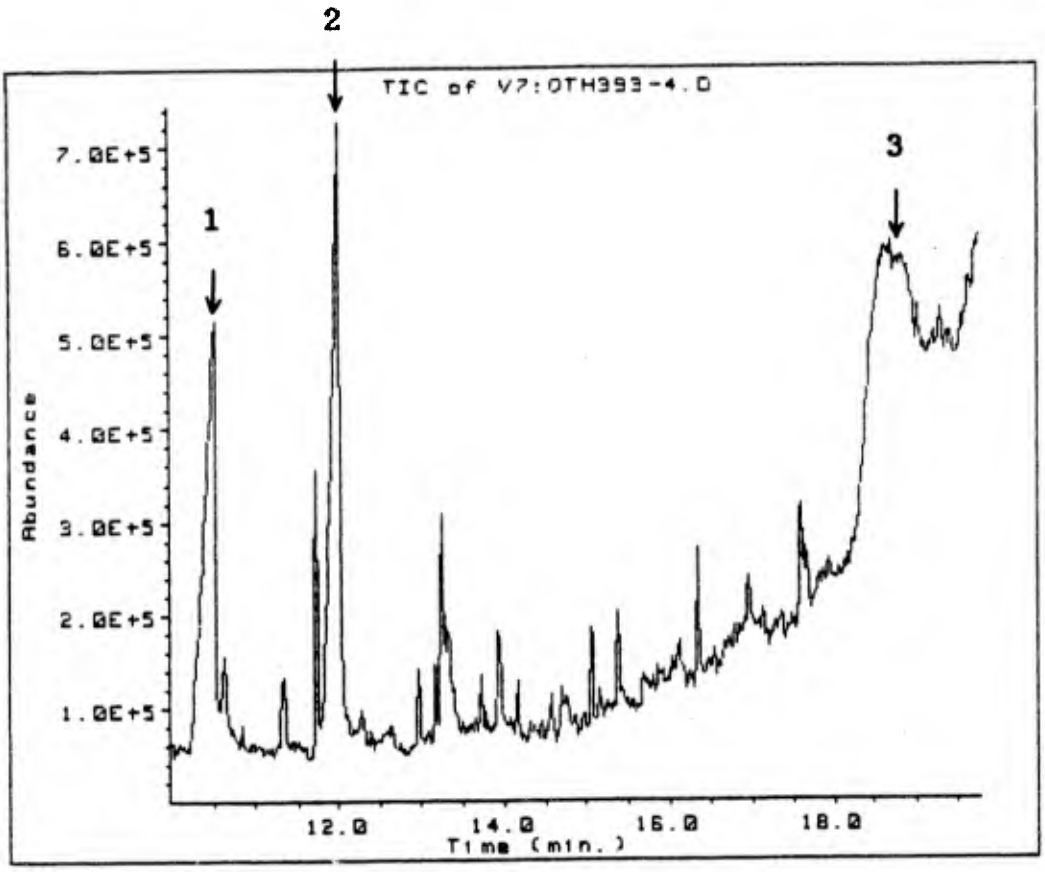
Acetophenone

OTH-193-3c

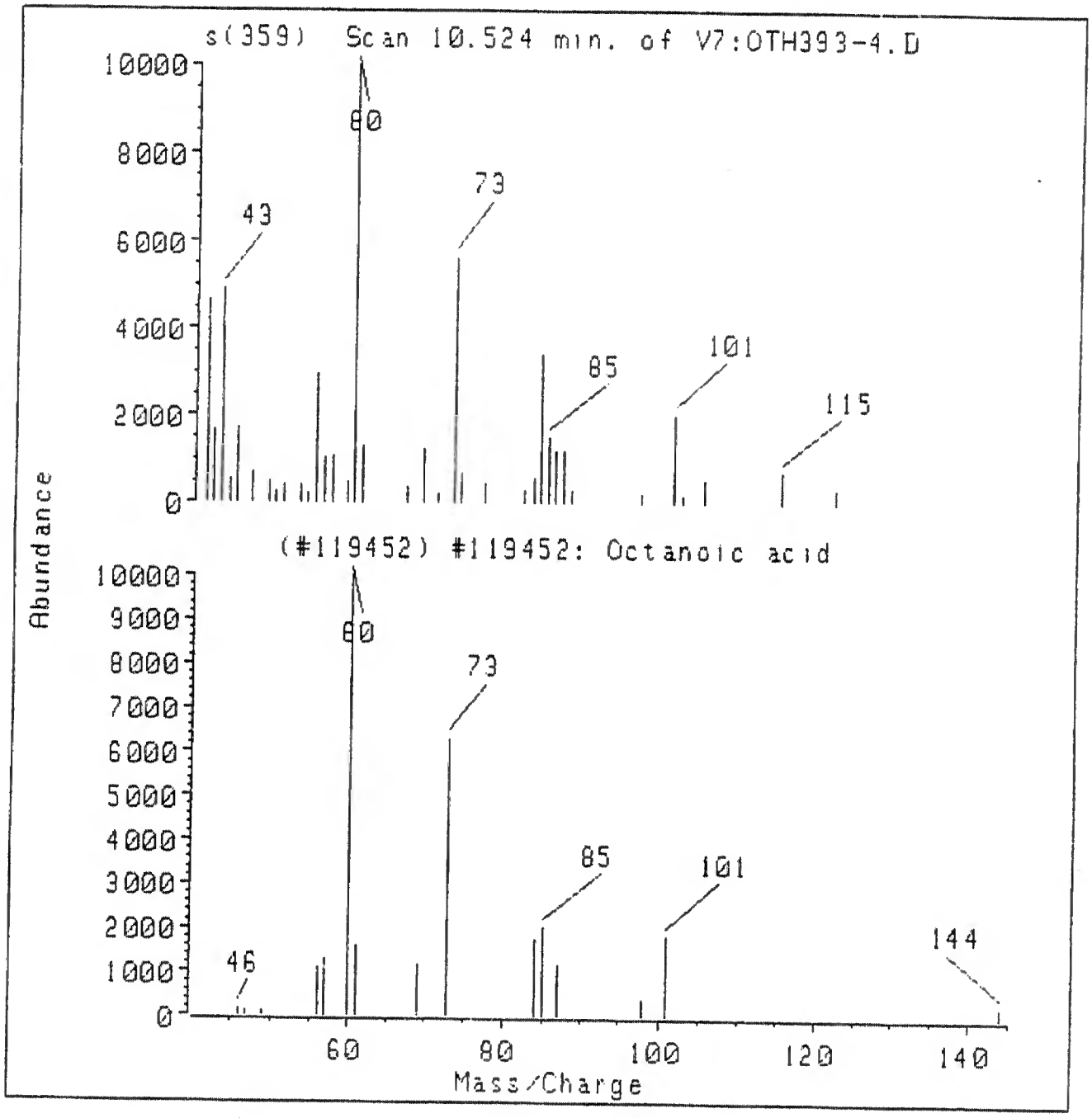


Chloroacetophenone

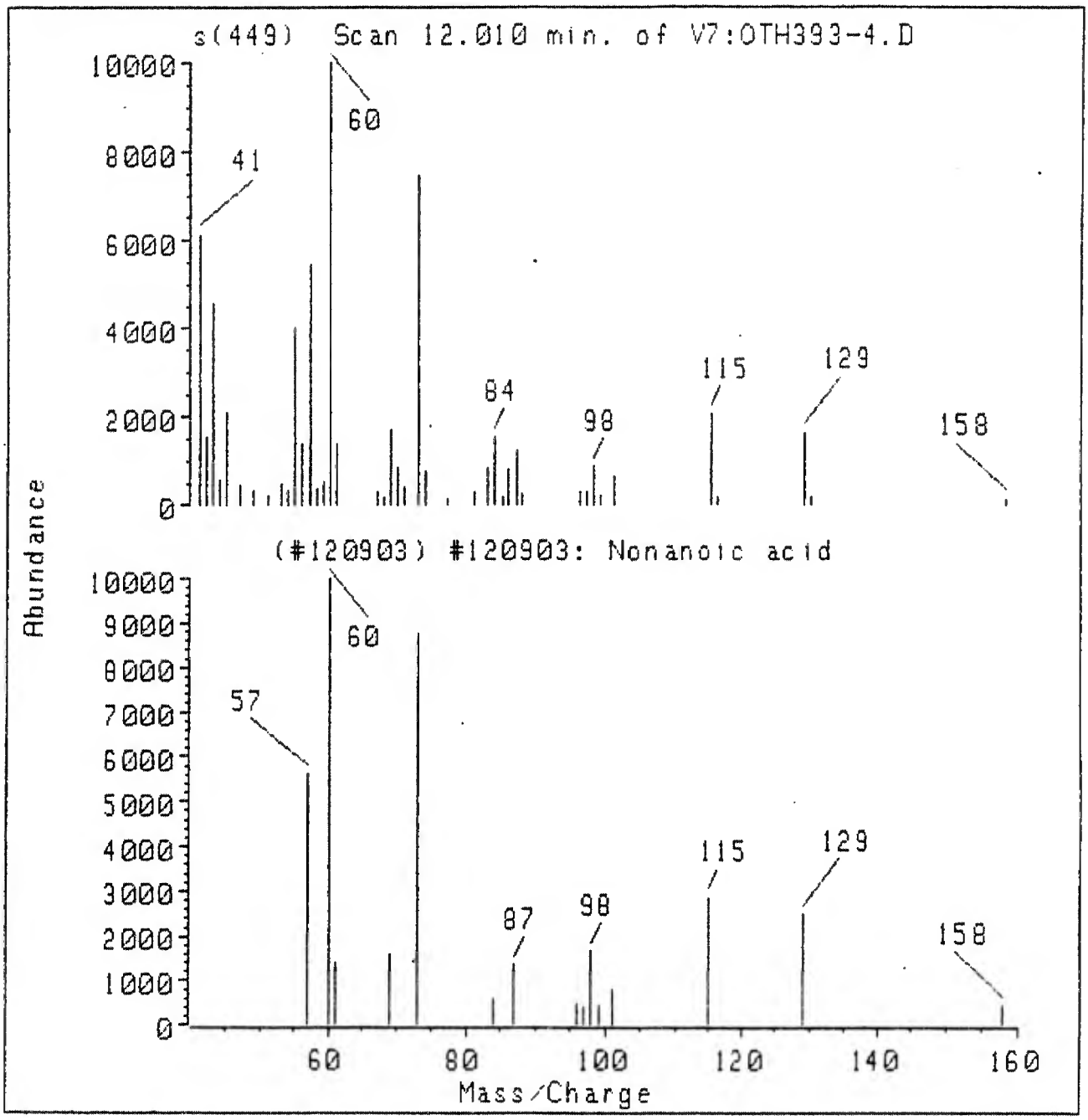
Sample OTH-393-4



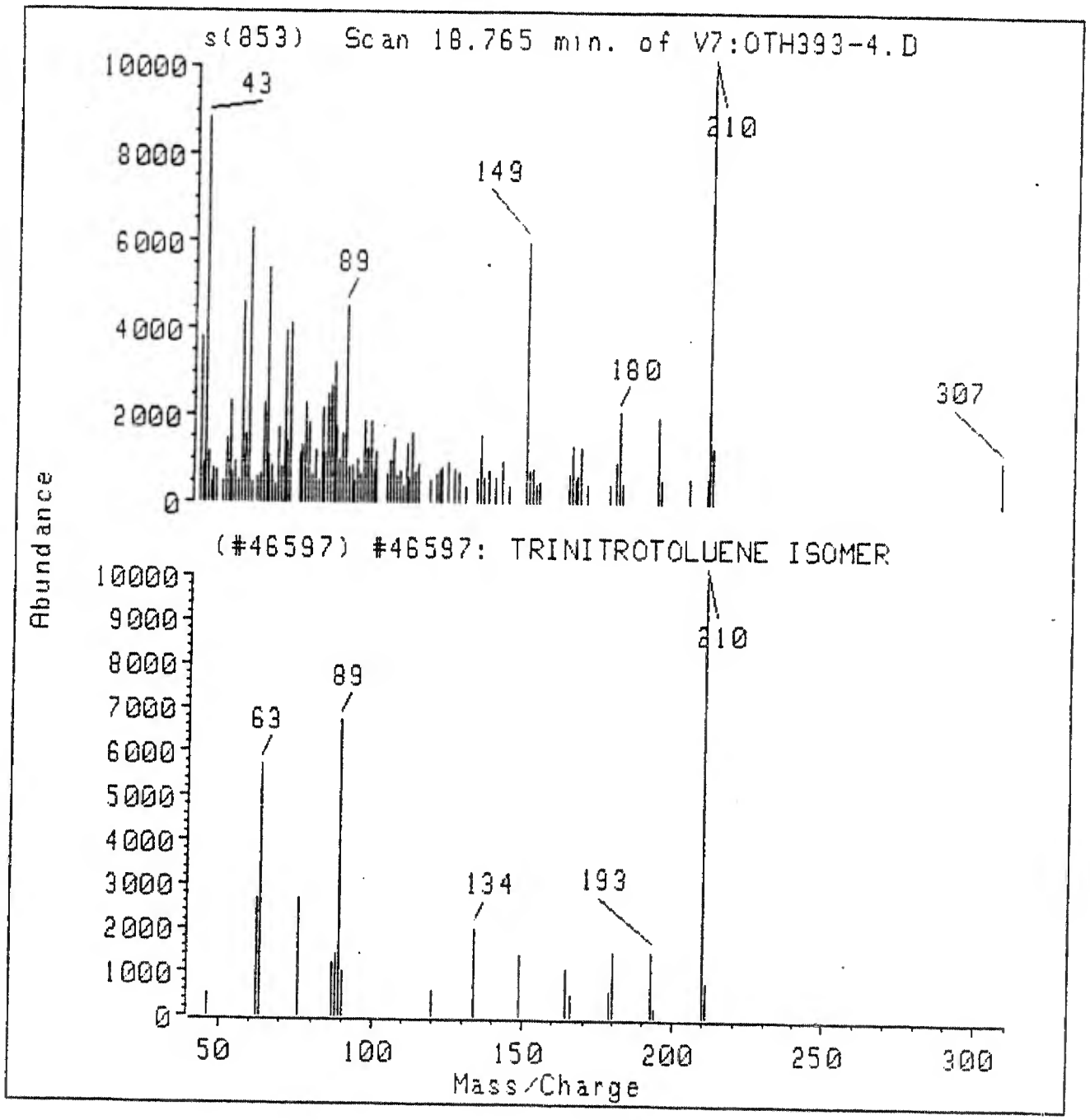
1. Octanoic acid
2. Nonanoic acid
3. TNT


s(359) Scan 10.524 min. of V7:OTH393-4.D
10000
8000
6000
4000
2000
0
60
73
43
85
101
115
(#119452) #119452: Octanoic acid
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
60
73
85
101
46
144
60
80
100
120
140
Abundance
Mass/Charge

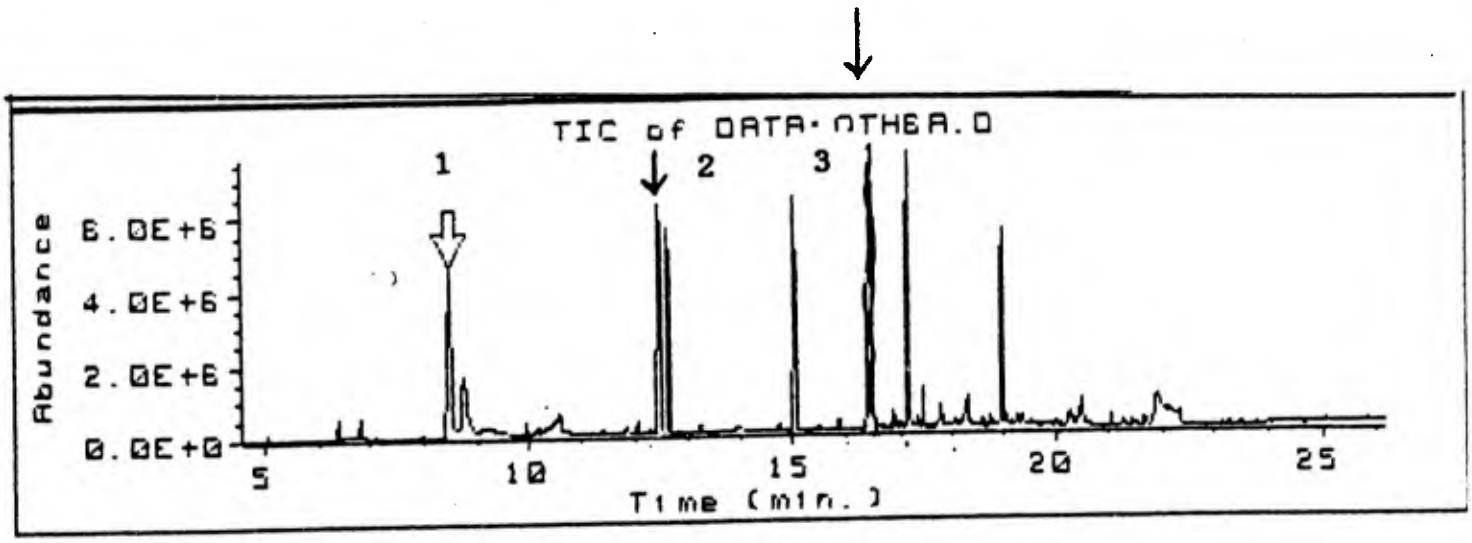
OTH-393-4


s(449) Scan 12.010 min. of V7:OTH393-4.D
10000
8000
6000
4000
2000
0
41
60
84
98
115
129
158
(#120903) #120903: Nonanoic acid
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
57
60
87
98
115
129
158
Abundance
60
80
100
120
140
160
Mass/Charge

OTH-393-4

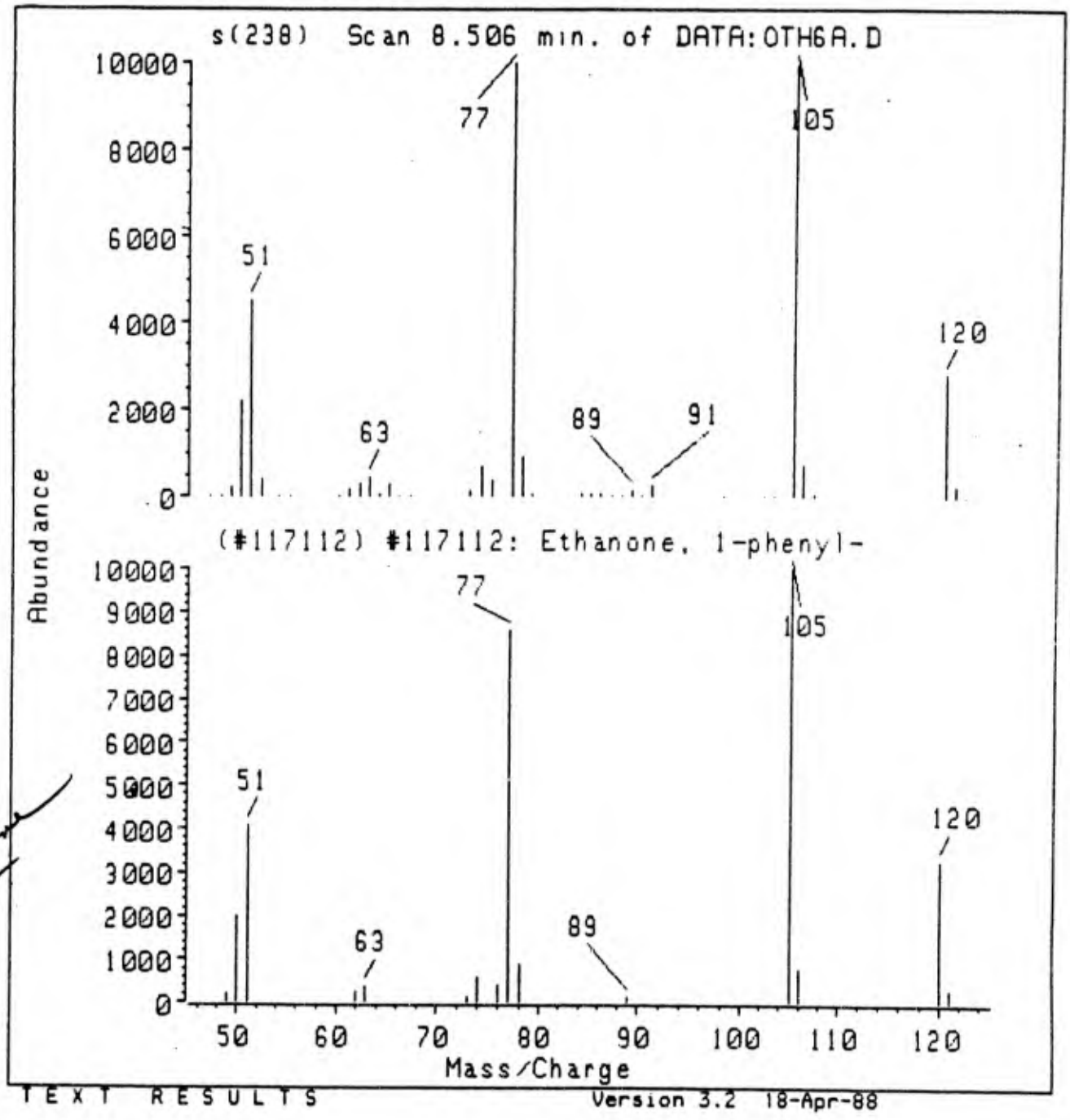

s(853) Scan 18.765 min. of V7:OTH393-4.D
10000
8000
6000
4000
2000
0
43
89
149
180
210
307
(#46597) #46597: TRINITROTOLUENE ISOMER
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
63
89
134
193
210
Abundance
50
100
150
200
250
300
Mass/Charge

Sample OTH-393-6a



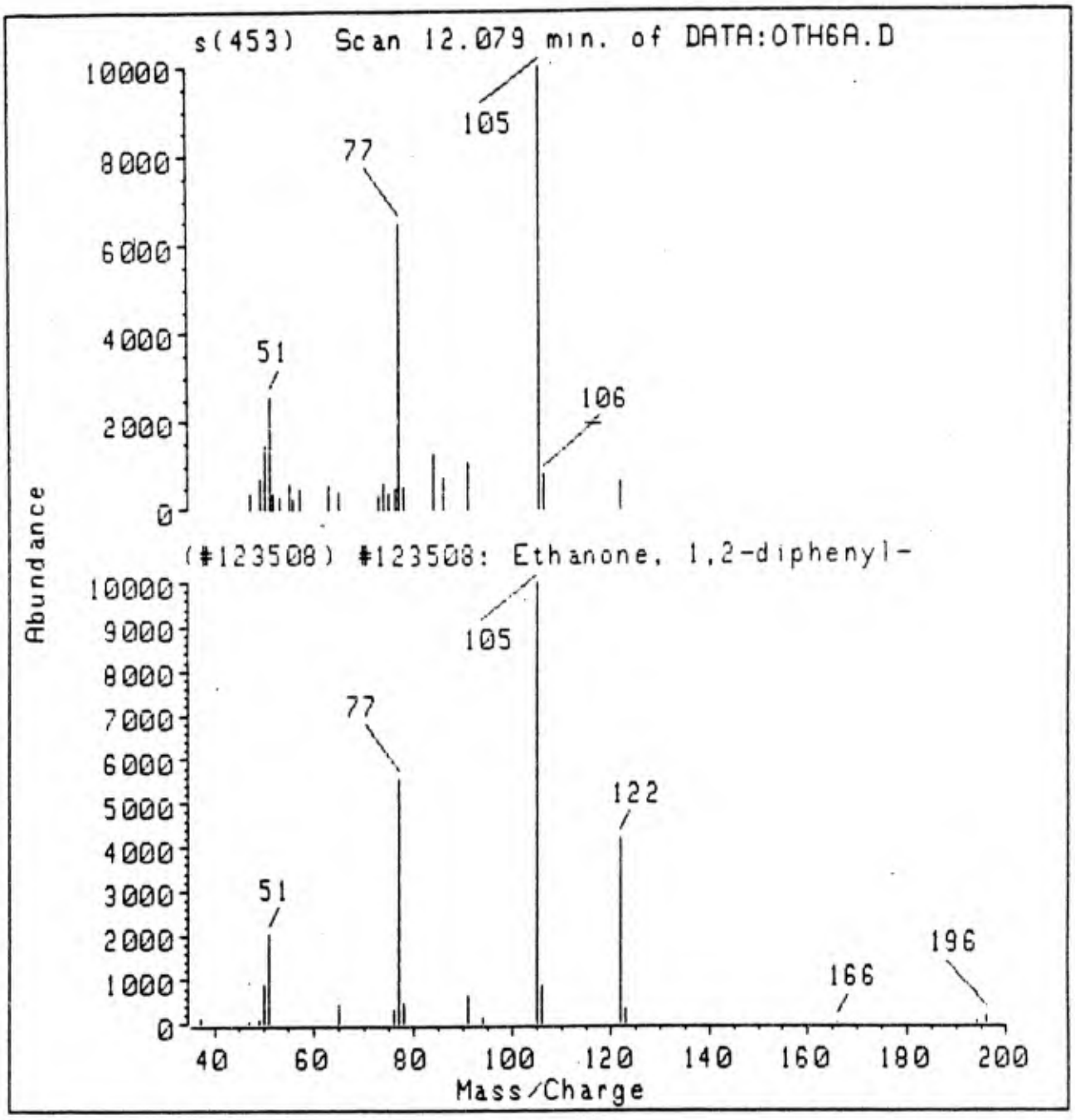
Sample OTH-393-6a

1. Acetophenone
2. 1,2-diphenyl ethanone
3. Diphenyl sulfide



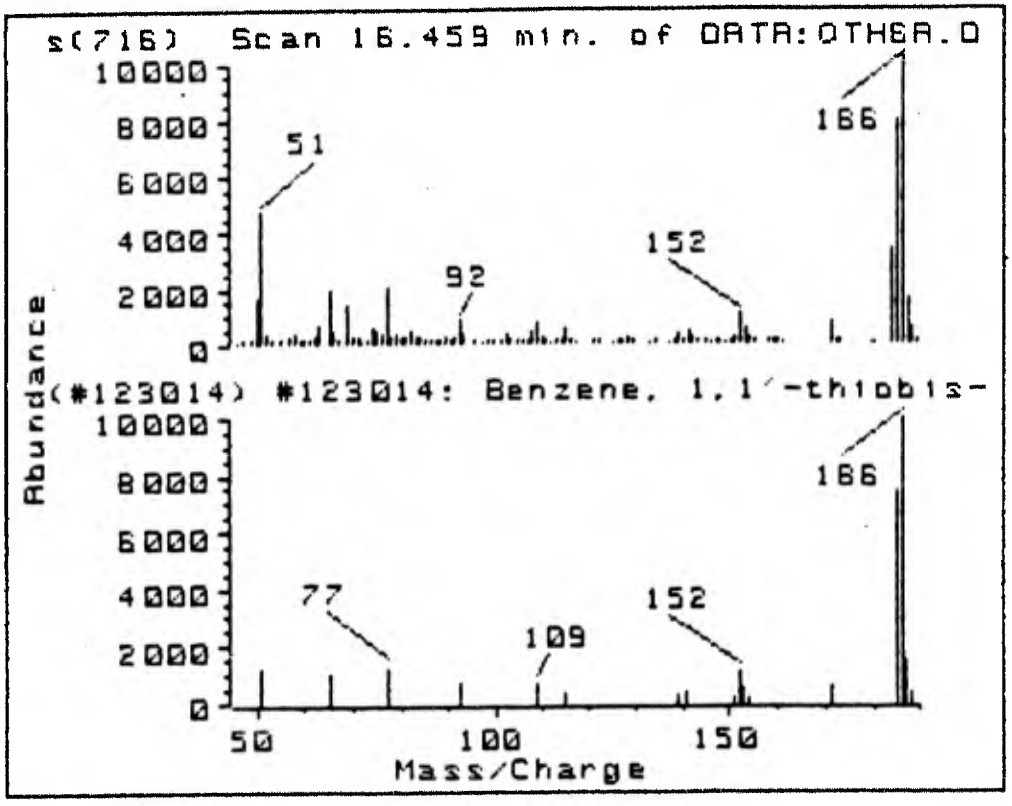
acetophenone

Ethanone, 1-phenyl-	Entry# = 117112	CAS Num = 000098-86-2
000098-86-2 97	Company ID Num = WILEY 1/88	
Ethanone, 1-phenyl-	Mol.Weight = 120.057	Retention Index = 0.000
000098-86-2 97	Melting Point = 0C	Boiling Point = 0C
Ethanone, 1-phenyl-	Name =	
000098-86-2 91	Ethanone, 1-phenyl-	
Ethanone, 1-phenyl-		
000098-86-2 91	**Acetophenone**	
Ethanone, 1-phenyl-		
000098-86-2 91	Mol. Formula =	
Ethanone, 1-phenyl-	C8H8O	
000098-86-2 90		
Ethanone, 1-phenyl-	Misc. Info. =	
000098-86-2 76	1VR	
Benzeneacetic acid,		
015206-55-0 72		
PHENYL-GLYOXYLIC ACI		
72		


s(453) Scan 12.079 min. of DATA:OTH6A.D
10000
8000
6000
4000
2000
0
105
77
51
106
Abundance
(#123508) #123508: Ethanone, 1,2-diphenyl-
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
105
77
122
51
196
166
40
60
80
100
120
140
160
180
200
Mass/Charge

OTH-393-6a

OTH-393-6a



Diphenyl sulfide

Sample OTH-293-9c

P R E P A R E T O I N J E C T 14 Jan 93 10:45 am PARAM:MSPARAM.A

Data file : DATA:OTH9C.D

Words available in file are 334080

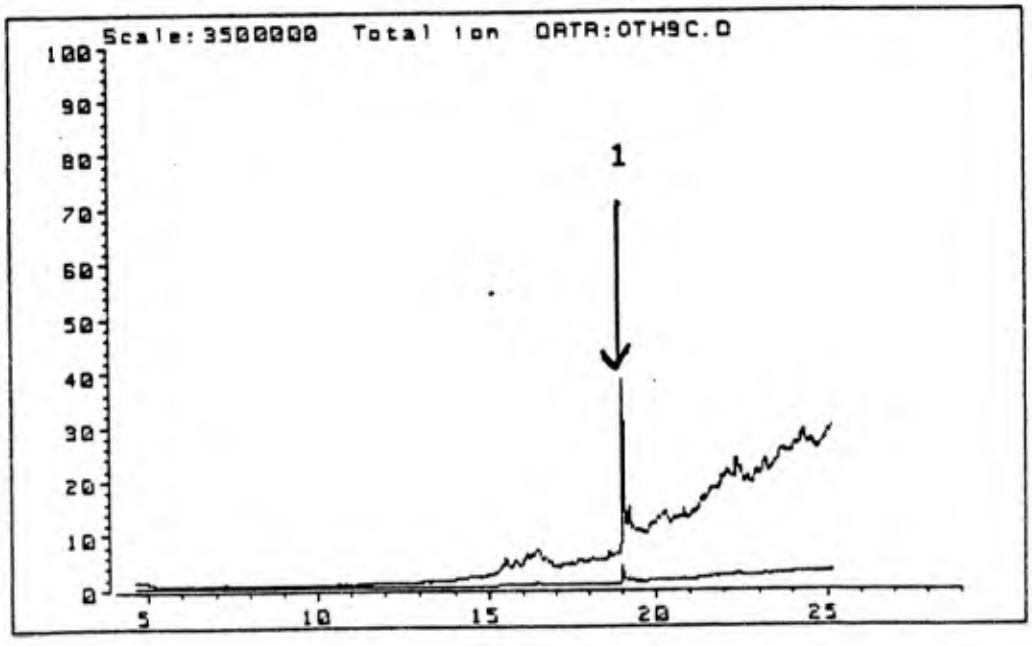
Operator : J. REEDER

sample Name : OTH293-9C CDCL3 CRYOFOCUS

misc. Info :

ZONE	ACTUAL	SETPOINT	ZONE	ACTUAL	SETPOINT
Oven	60	60	Inj Port A	0	OFF
Inj Port B	250	250	Transfer Line	250	250
Detector A	0	OFF			

Ready to inject

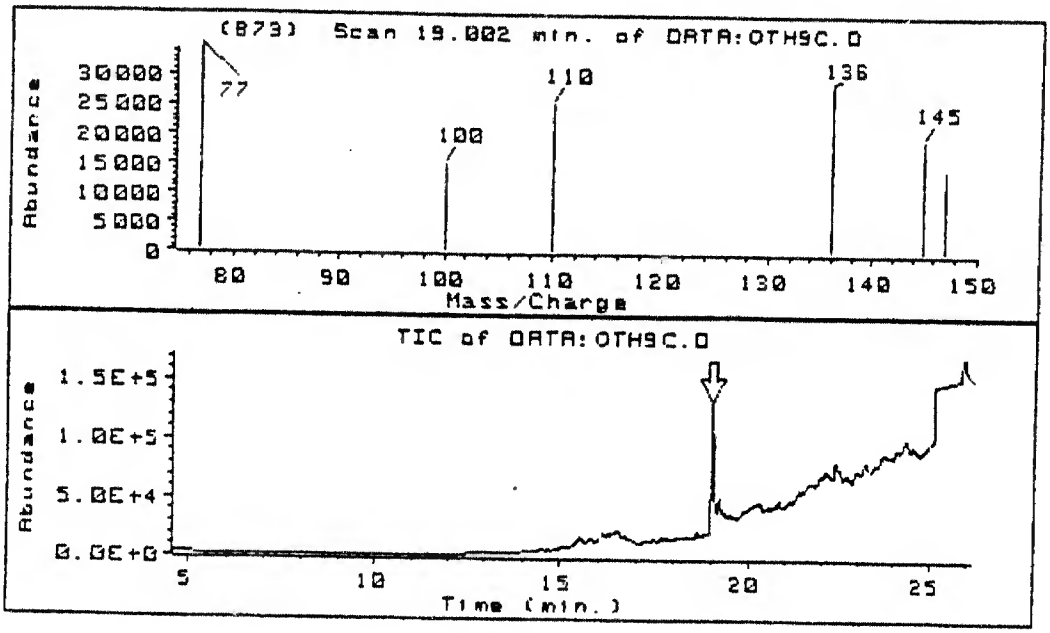


Sample OTH-293-9c

PRCE

1. L-3

OTH-293-9c


(873) Scan 19.002 min. of DATA:OTH9C.D
Abundance
30000
25000
20000
15000
10000
5000
0
77
100
110
136
145
80
90
100
110
120
130
140
150
Mass/Charge
TIC of DATA:OTH9C.D
Abundance
1.5E+5
1.0E+5
5.0E+4
0.0E+0
5
10
15
20
25
Time (min.)

L-3

Sample OTH-293-10c

S I M A C Q U I S I T I O N 14 Jan 93 7:57 am PARAM:MSPARAM.A

solvent delaY 4.50 eM volts 0 relAtive resultıng voltage 2600

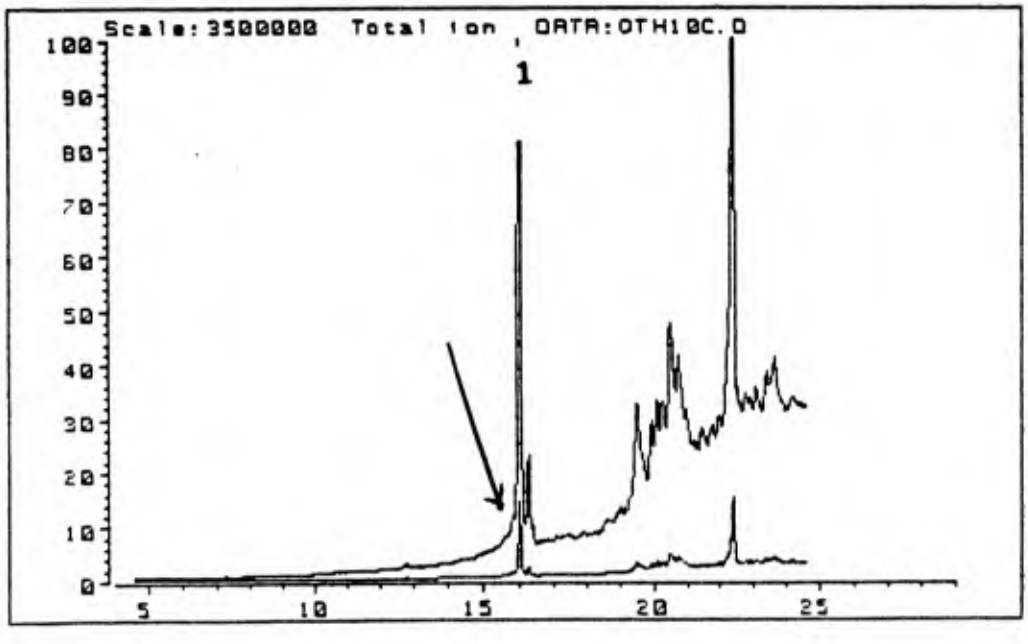
Group	1	2	3	4	5	6	7	8	9	10
# of Ions	6	6	20	20	20	20	20	20	20	20
start Time	4.50	4.50	-----	-----	-----	-----	-----	-----	-----	-----

low mass Resolution NO cycles per second 1.4

ion #	1	2	3	4	5	6
m/Z	77.00	100.00	110.00	136.00	145.00	147.00
Dwell	100	100	100	100	100	100

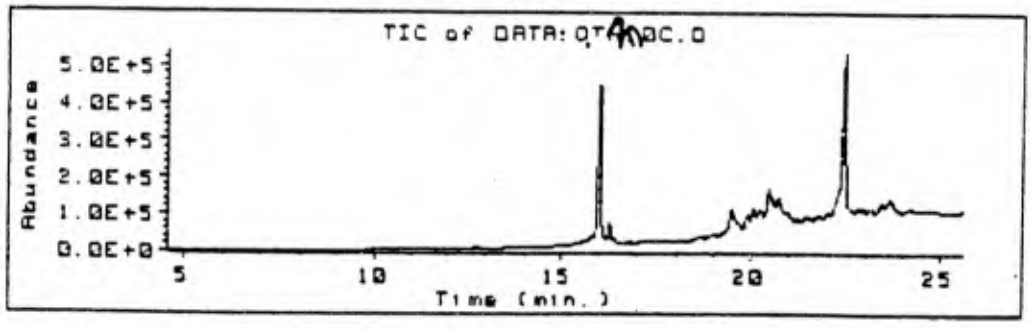
nUmber of plot traces 1 initiaLly ON time Window 25.0

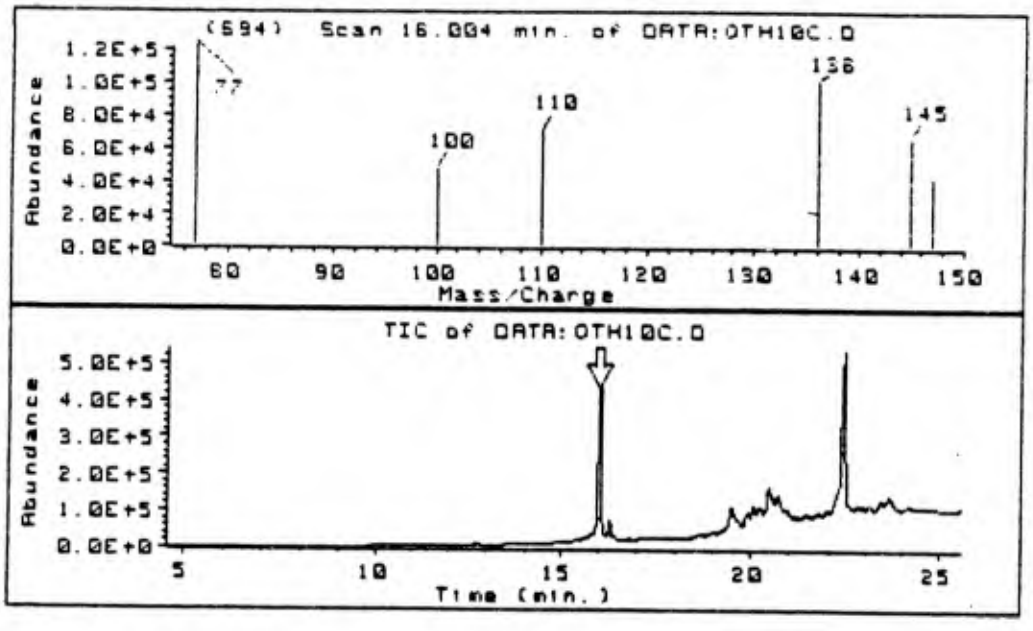
Plot # 1 m/Z TOTAL sCale 3500000



L-3

OTH-293-10c

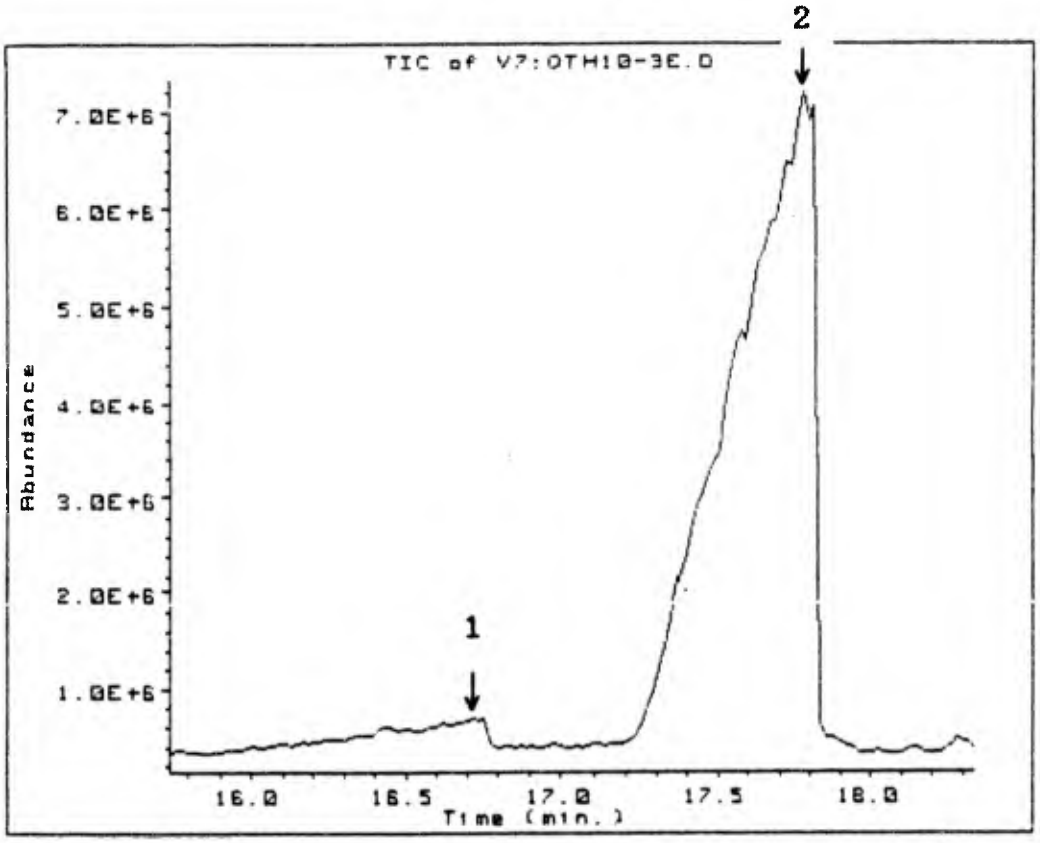




Sample OTH-293-10c

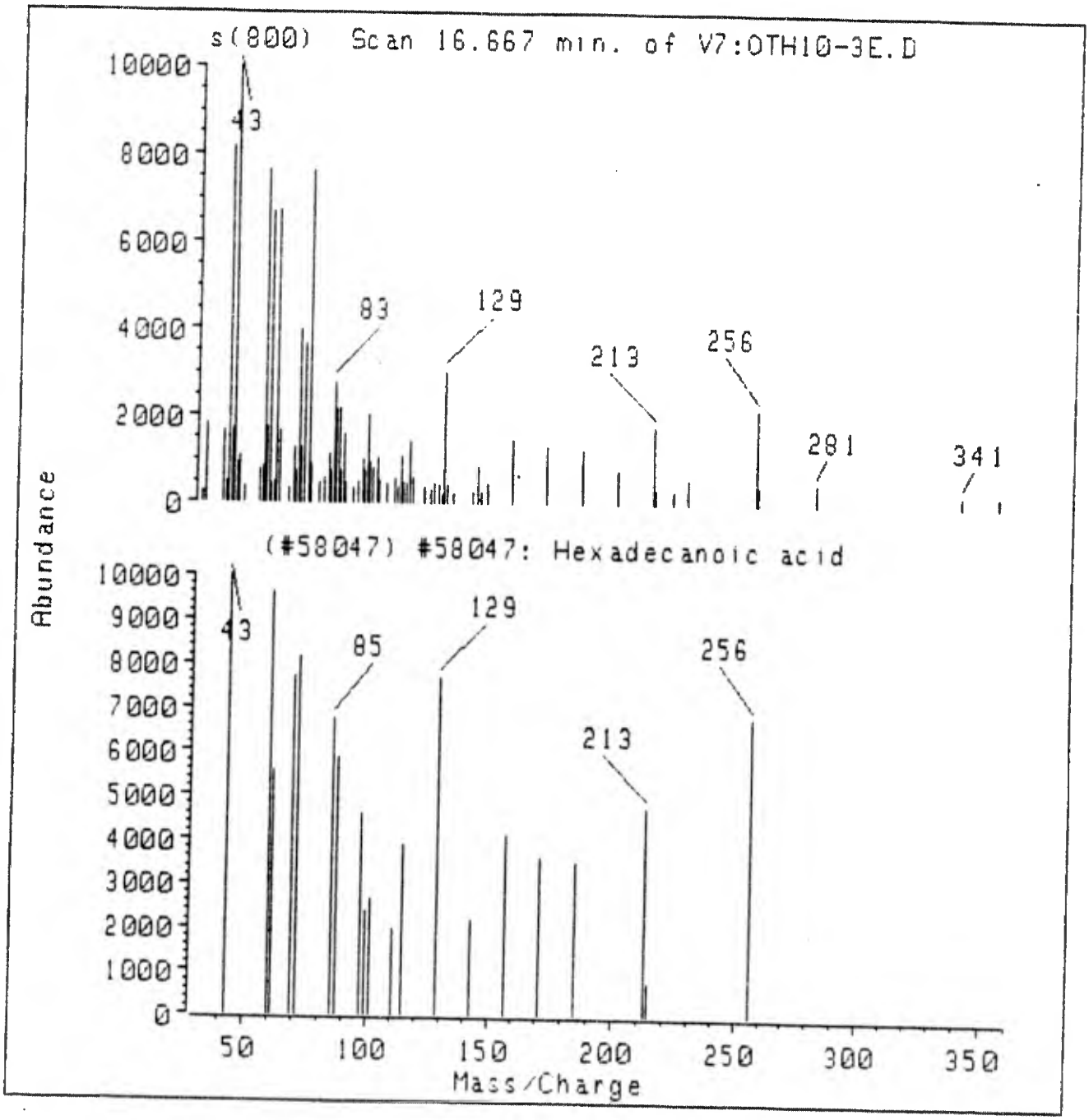
1. **L-3**

Sample OTH-1093-3e

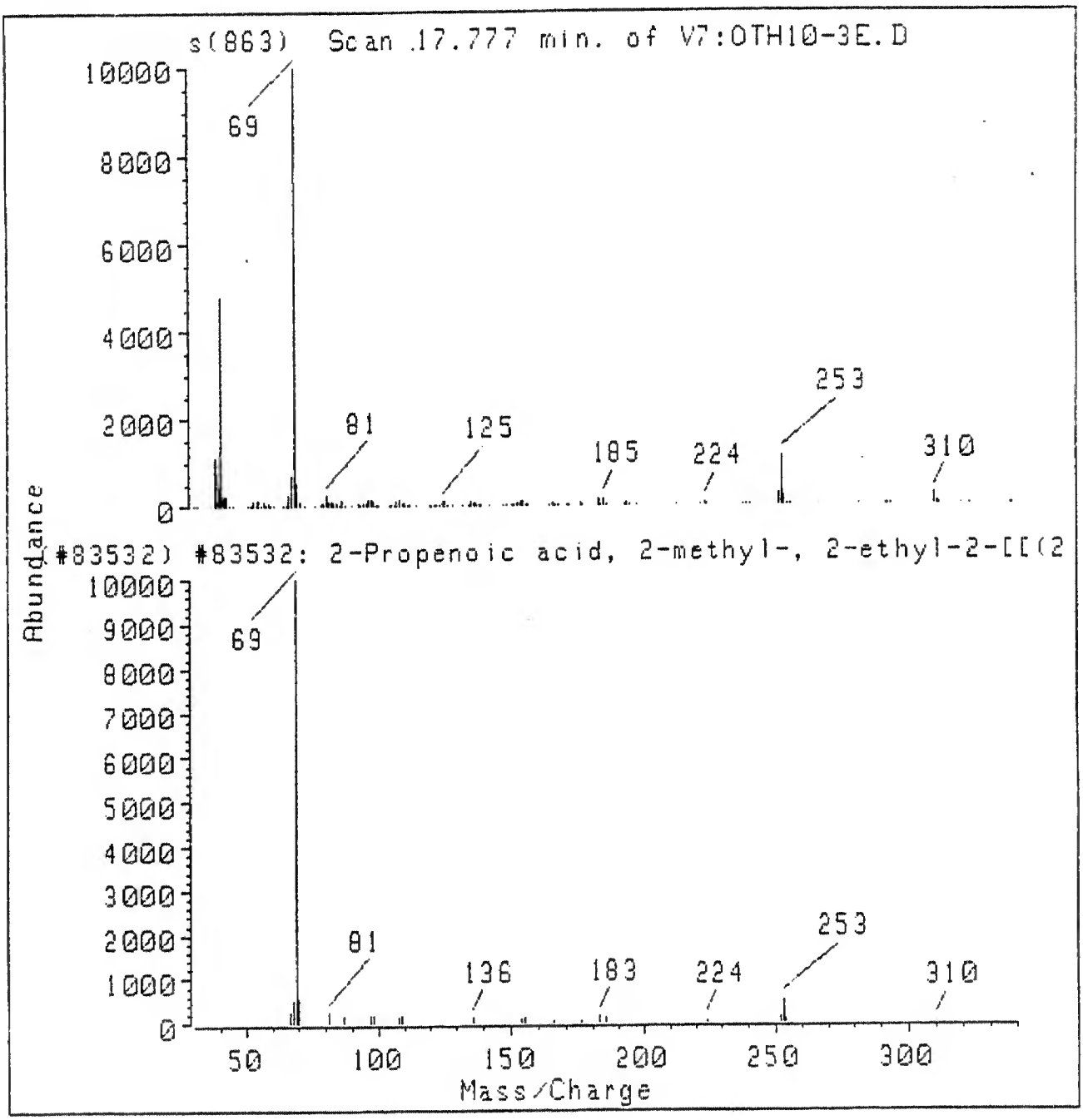


1. Hexadecanoic acid
2. Propenoic acid

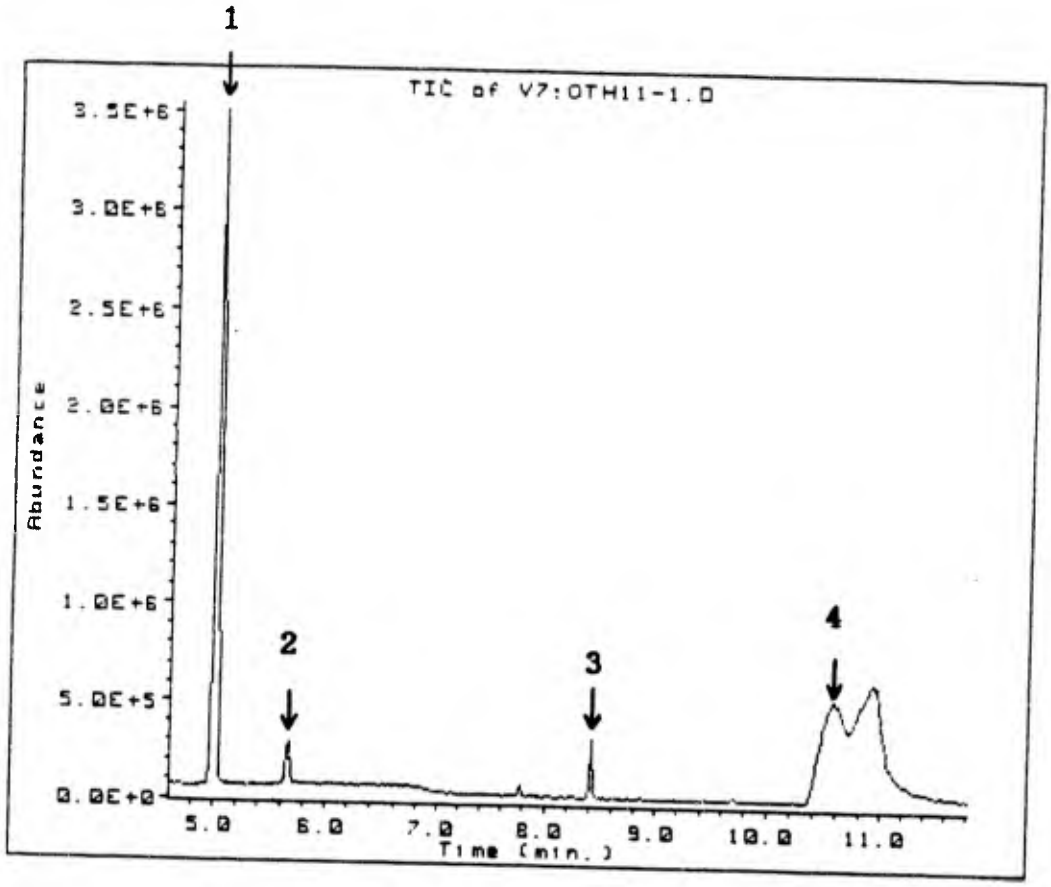
OTH-1093-3e


s(800) Scan 16.667 min. of V7:OTH10-3E.D
10000
8000
6000
4000
2000
0
43
83
129
213
256
281
341
(#58047) #58047: Hexadecanoic acid
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
43
85
129
213
256
Abundance
50
100
150
200
250
300
350
Mass/Charge

OTH-1093-3e

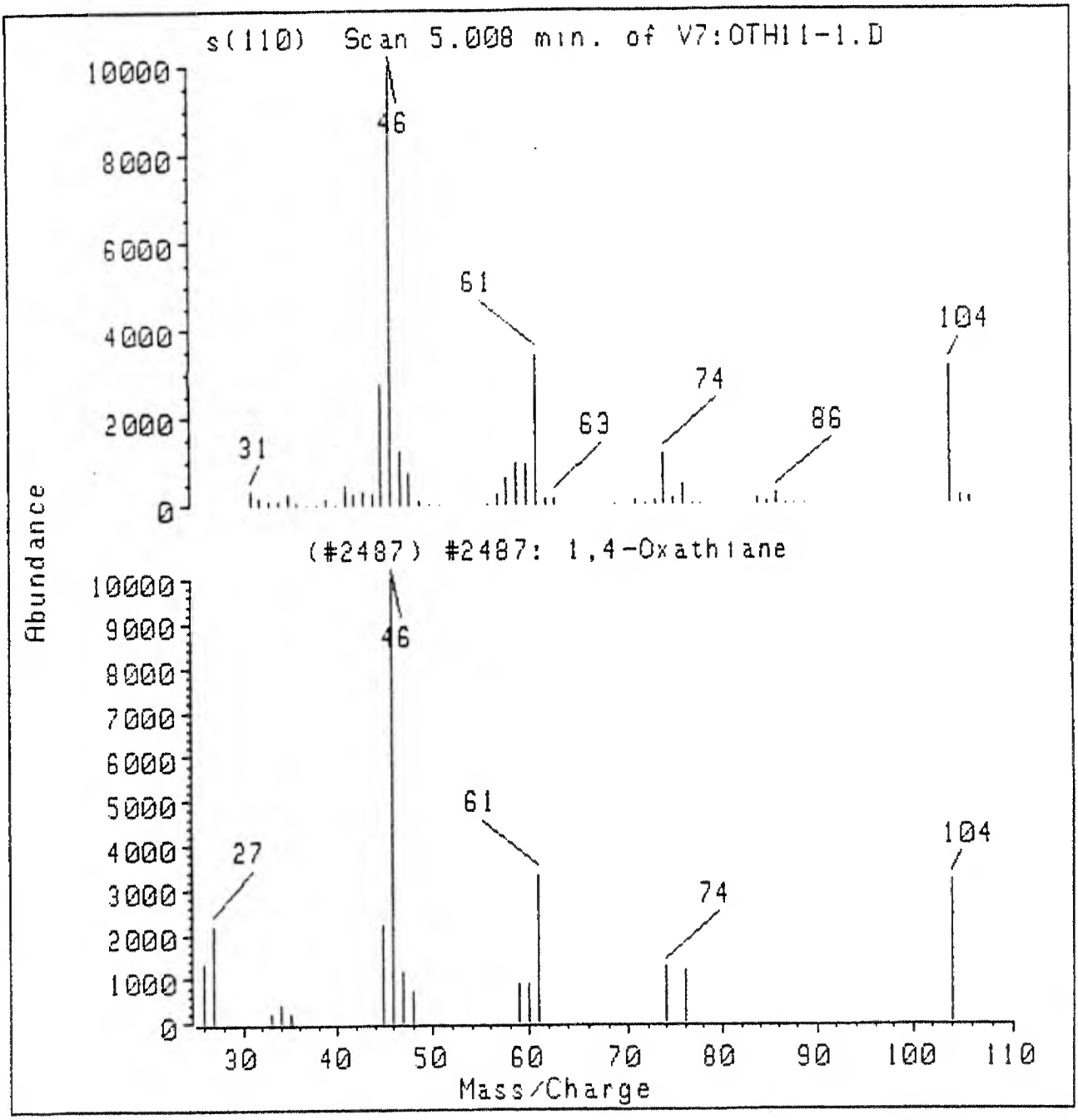

s(863) Scan 17.777 min. of V7:OTH10-3E.D
10000
8000
6000
4000
2000
0
69
81
125
185
224
253
310
#83532) #83532: 2-Propenoic acid, 2-methyl-, 2-ethyl-2-[[(2
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
69
81
136
183
224
253
310
Abundance
50
100
150
200
250
300
Mass/Charge

Sample OTH-1193-1



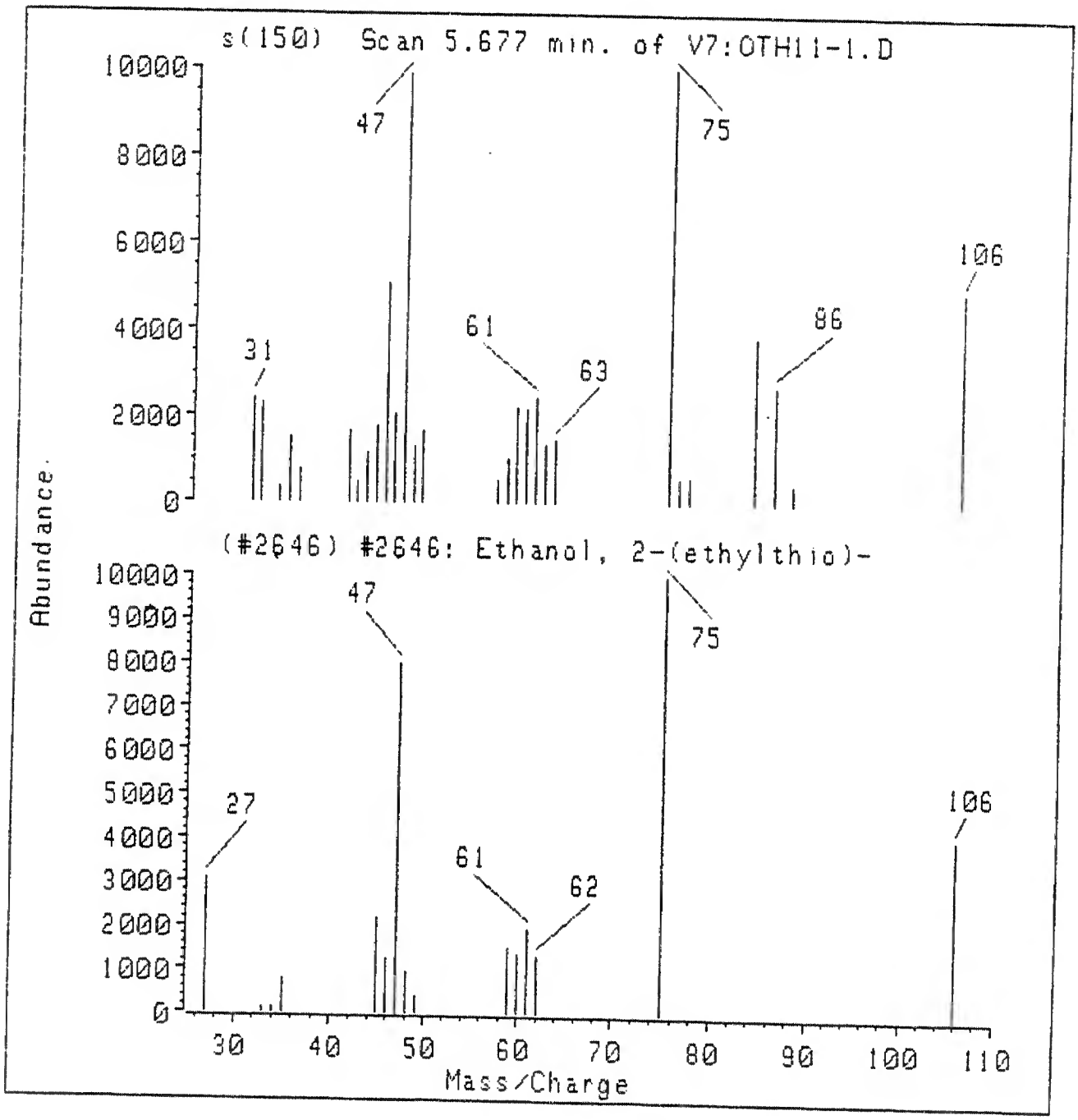
1. 1,4-Oxathiane
2. Ethyl 2-hydroxyethyl sulfide
3. 1,4-Dithiane
4. Thiodiglycol

OTH-1193-1

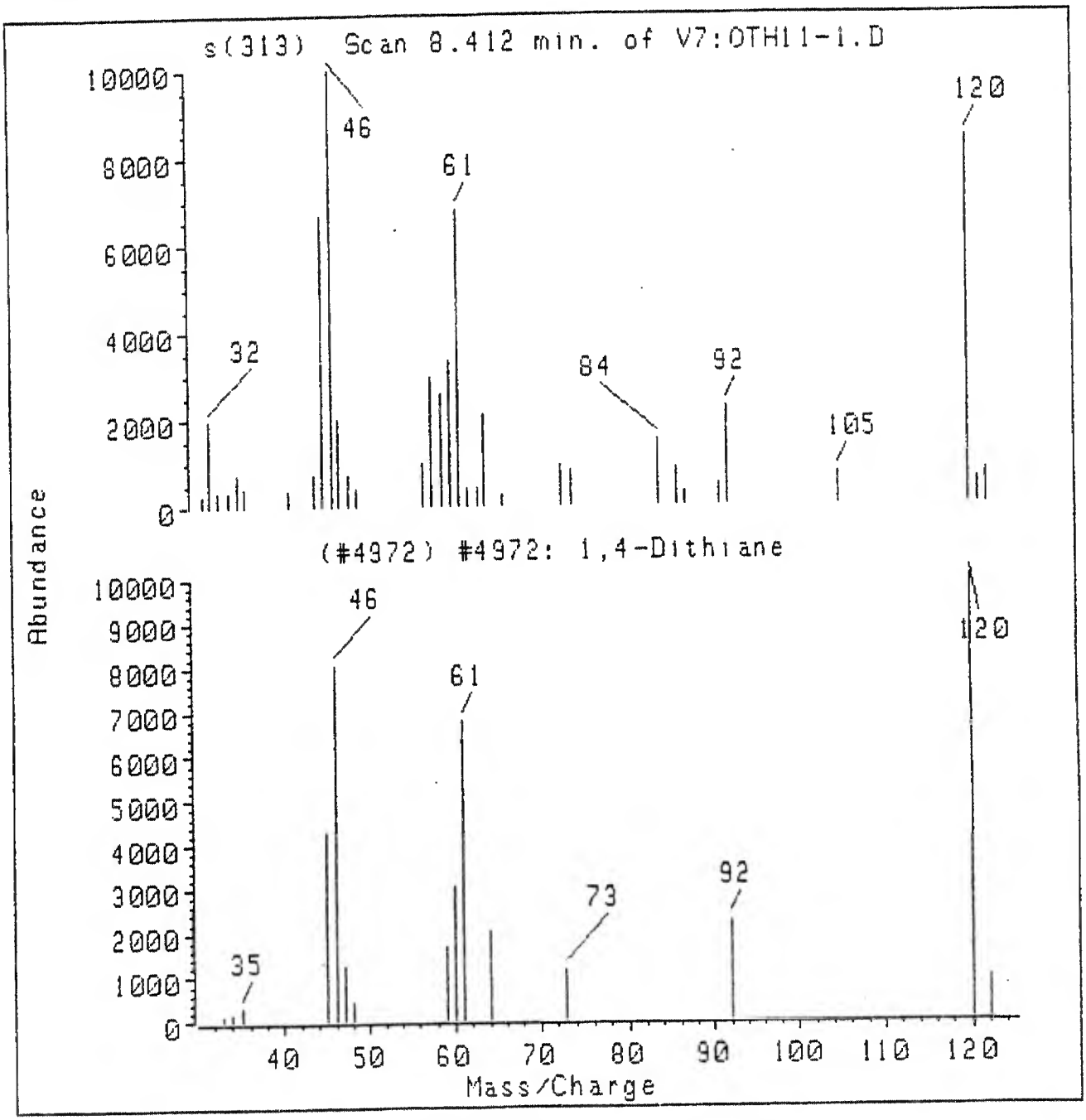

s(110) Scan 5.008 min. of V7:OTH11-1.D
10000
8000
6000
4000
2000
0
46
61
104
74
63
86
31
(#2487) #2487: 1,4-Oxathiane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
46
61
104
27
74
Abundance
30
40
50
60
70
80
90
100
110
Mass/Charge

Ethyl 2-hydroxyethyl sulfide

OTH-1193-1

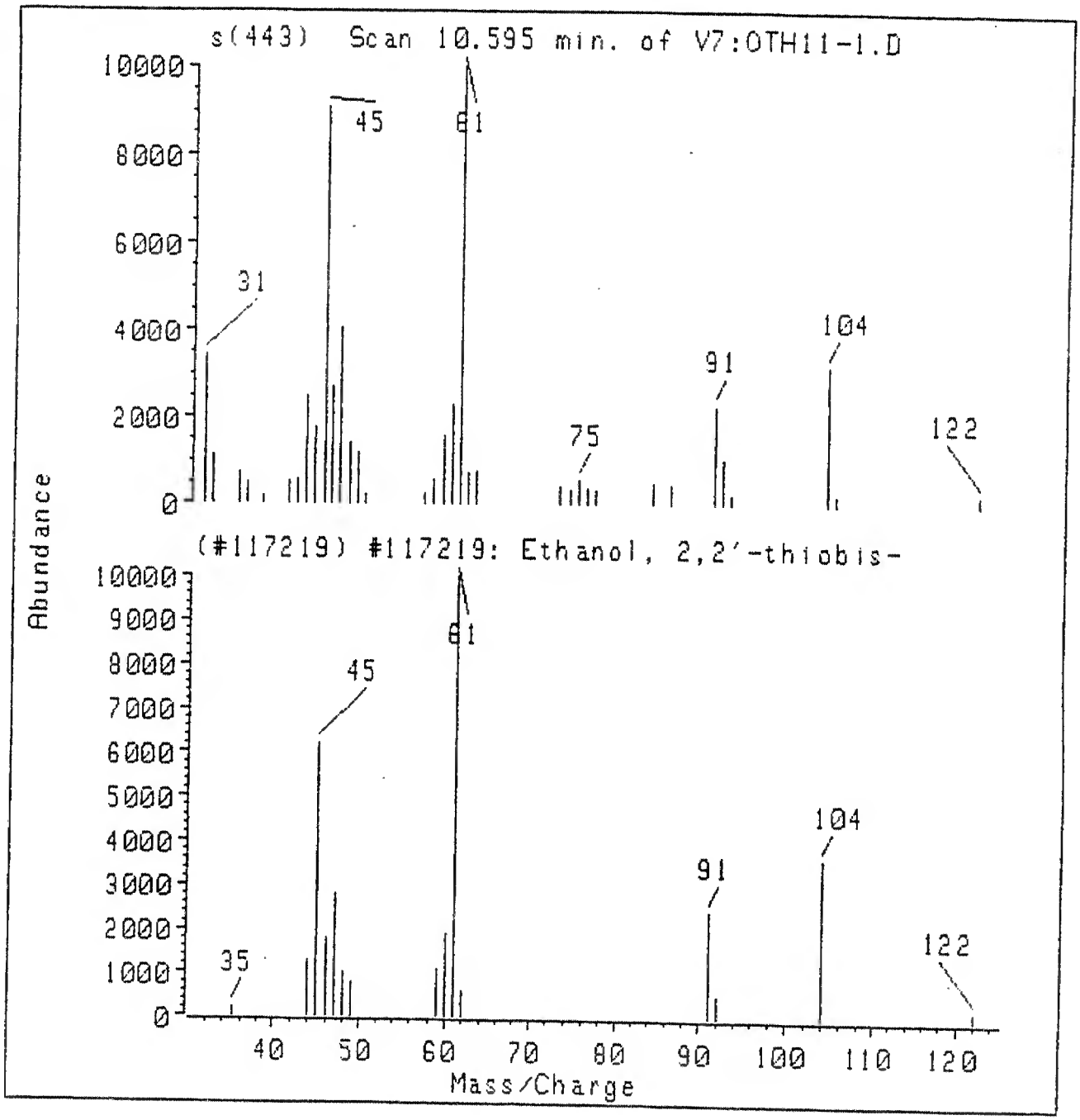


OTH-1193-1

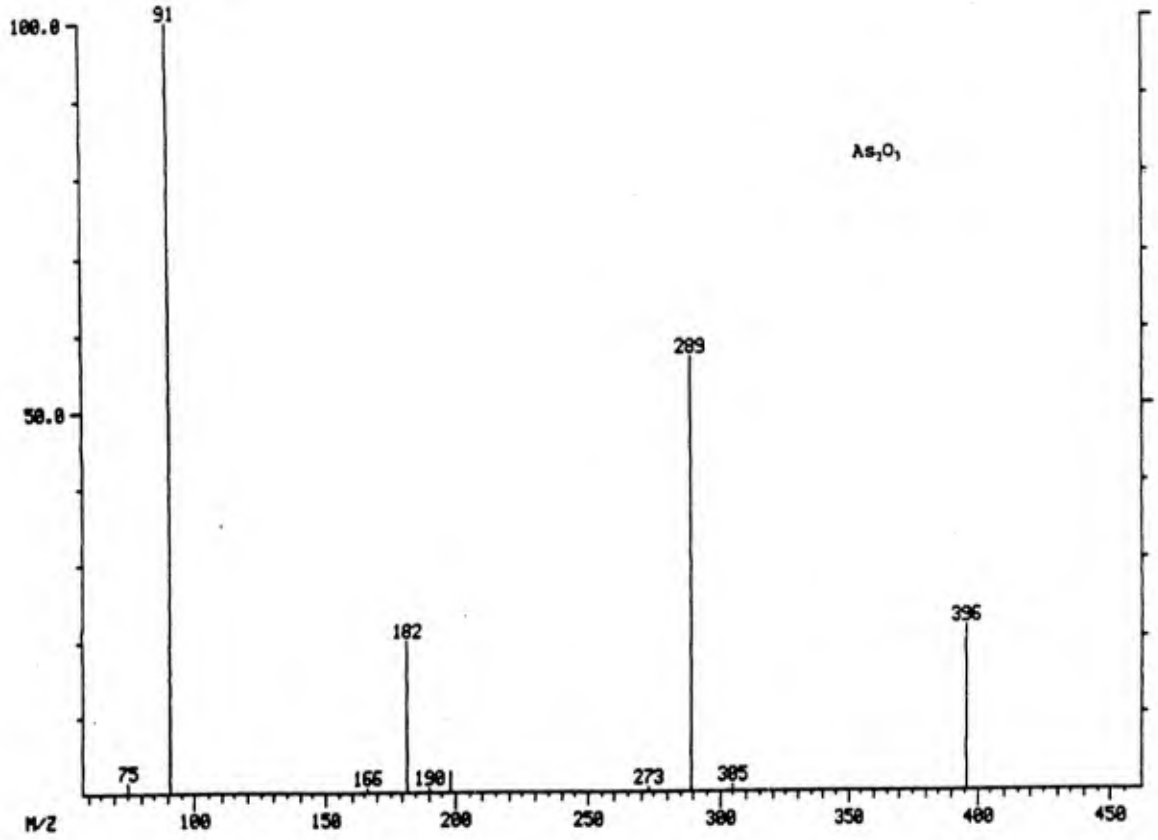

s(313) Scan 8.412 min. of V7:OTH11-1.D
10000
8000
6000
4000
2000
0
46
61
120
32
84
92
105
(#4972) #4972: 1,4-Dithiane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
46
61
120
92
73
35
Abundance
40
50
60
70
80
90
100
110
120
Mass/Charge

Thiodiglycol

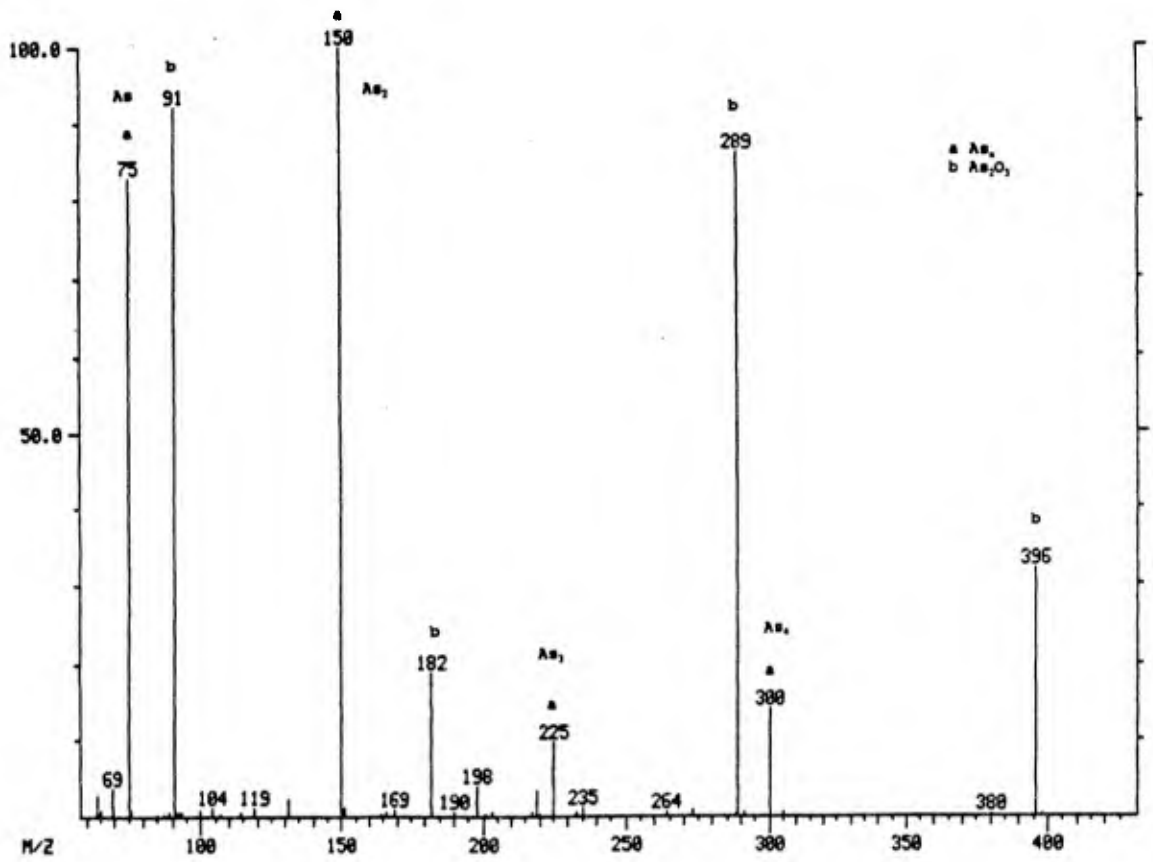
OTH-1193-1



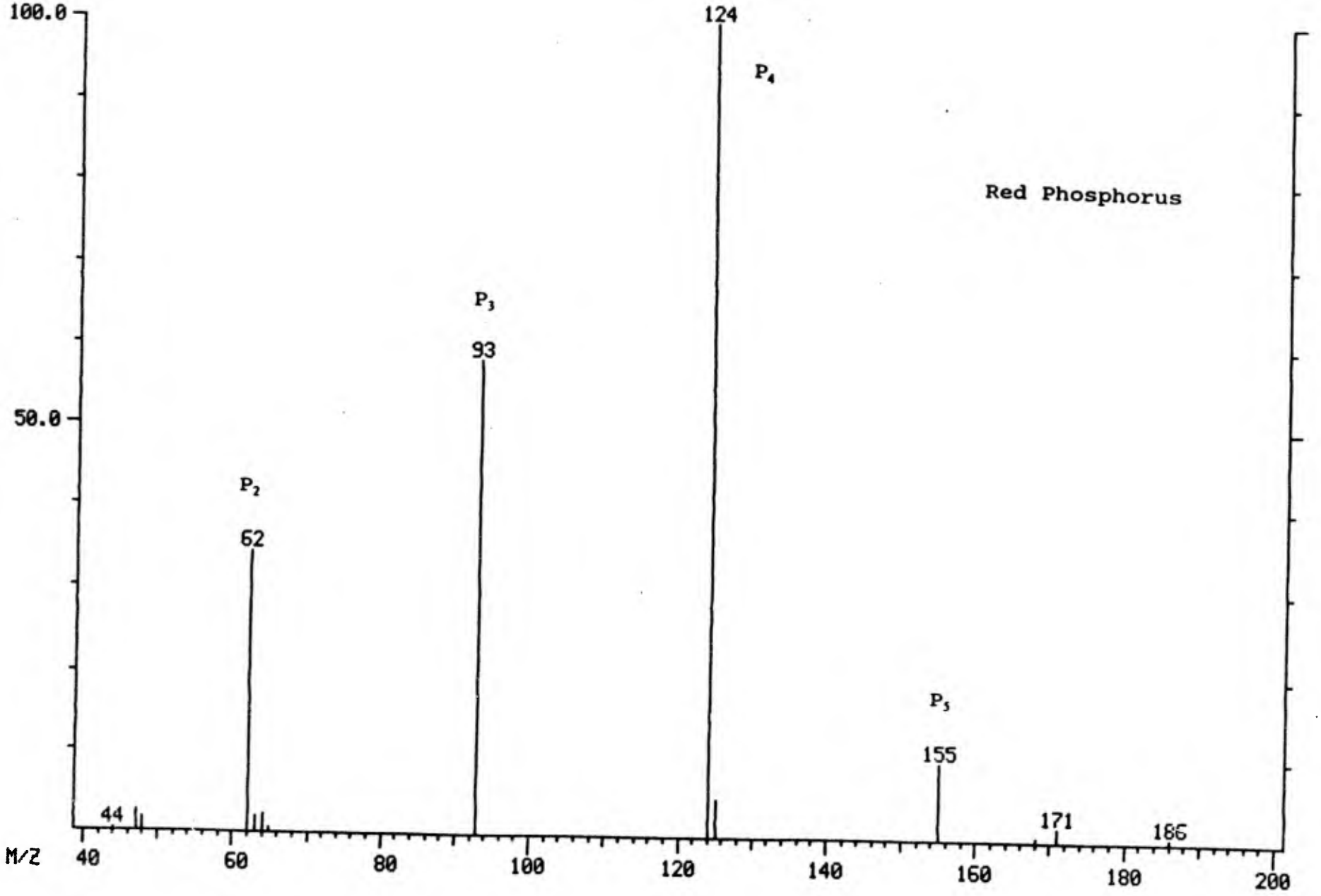
DEP/EI Mass Spectrum of Arsenic Trioxide Reference Standard



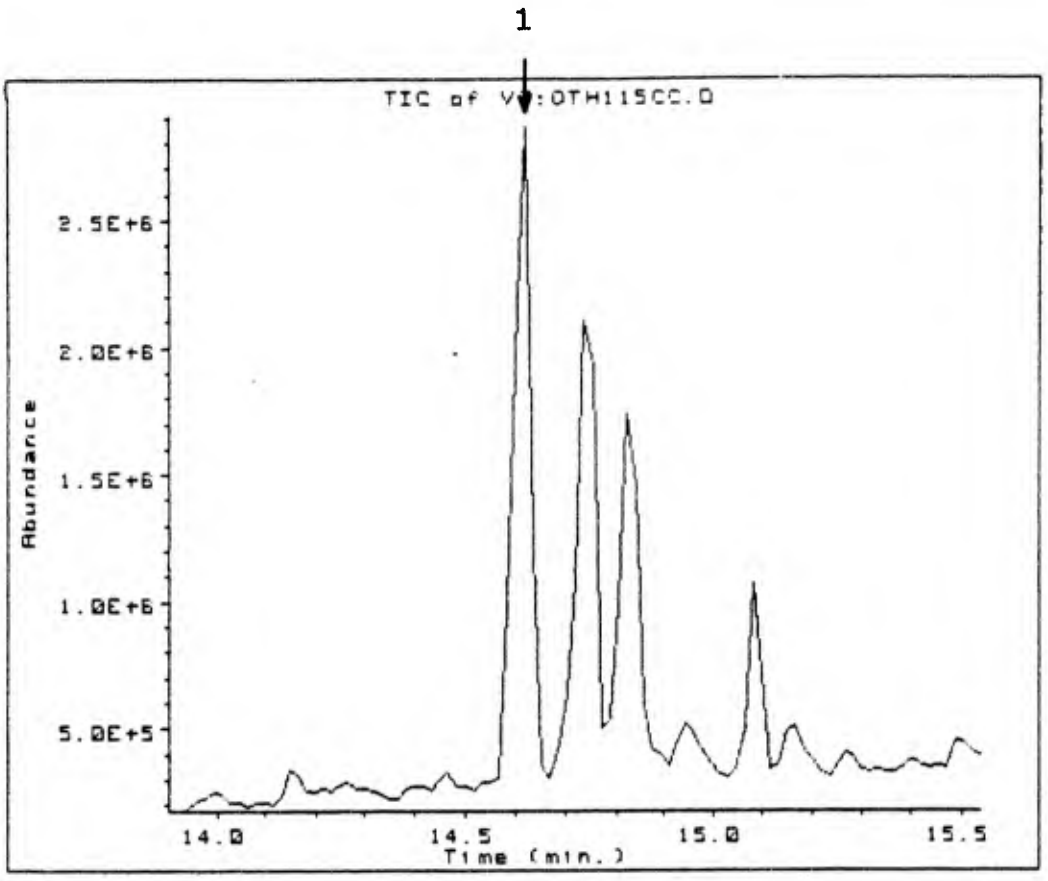
DEP/EI Mass Spectrum of OTH-1193-2c (Pig #18)



DEP/EI Mass Spectrum of OTH-1193-5a (Pig #22)

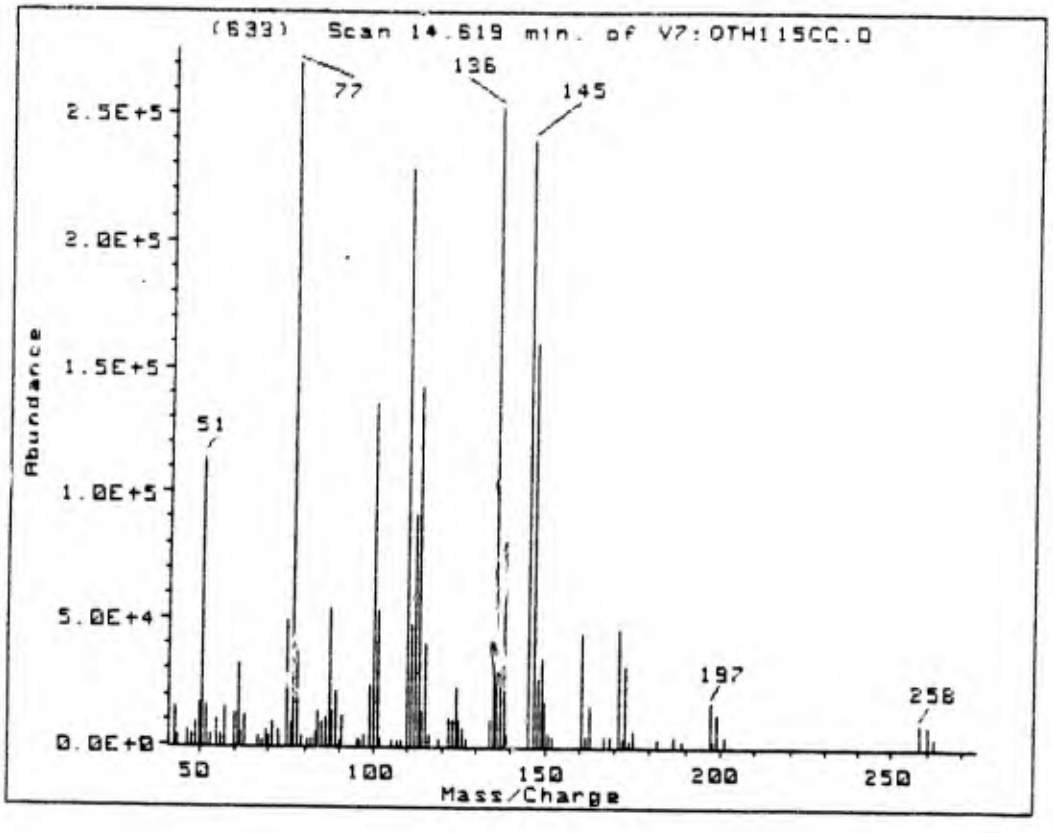


Sample OTH-1193-5c

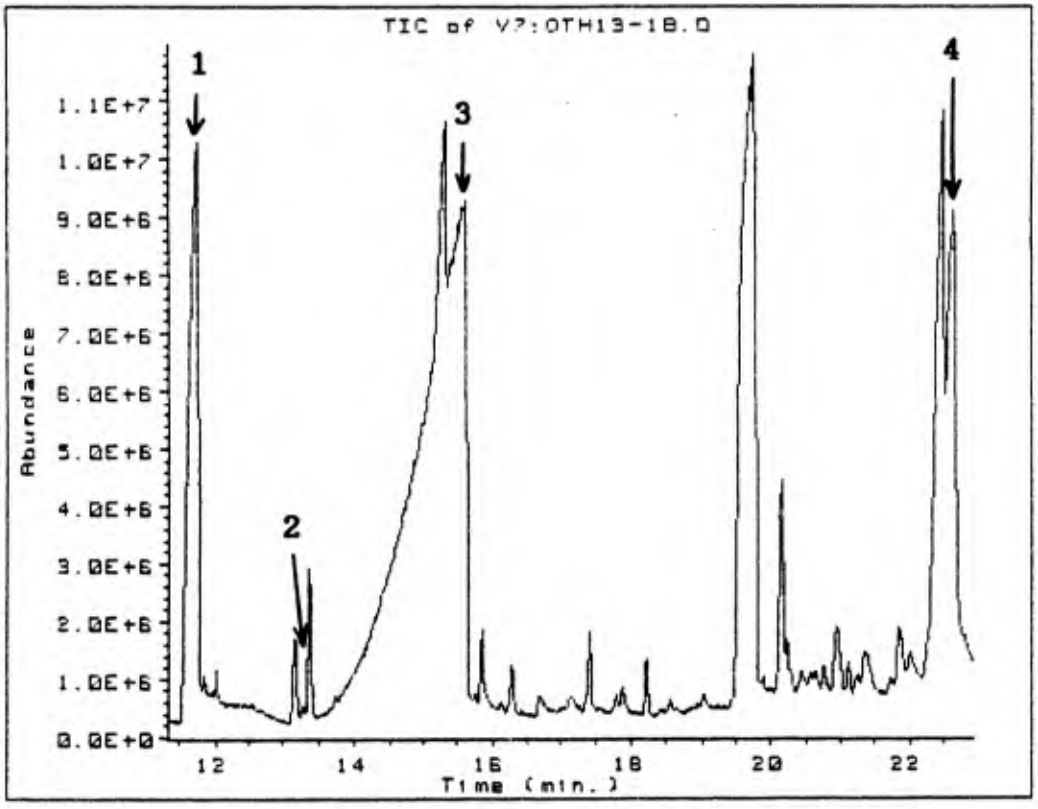


1. L-3

L-3


(633) Scan 14.619 min. of V7:OTH115CC.D
77
136
145
51
197
258
2.5E+5
2.0E+5
1.5E+5
1.0E+5
5.0E+4
0.0E+0
Abundance
50
100
150
200
250
Mass/Charge

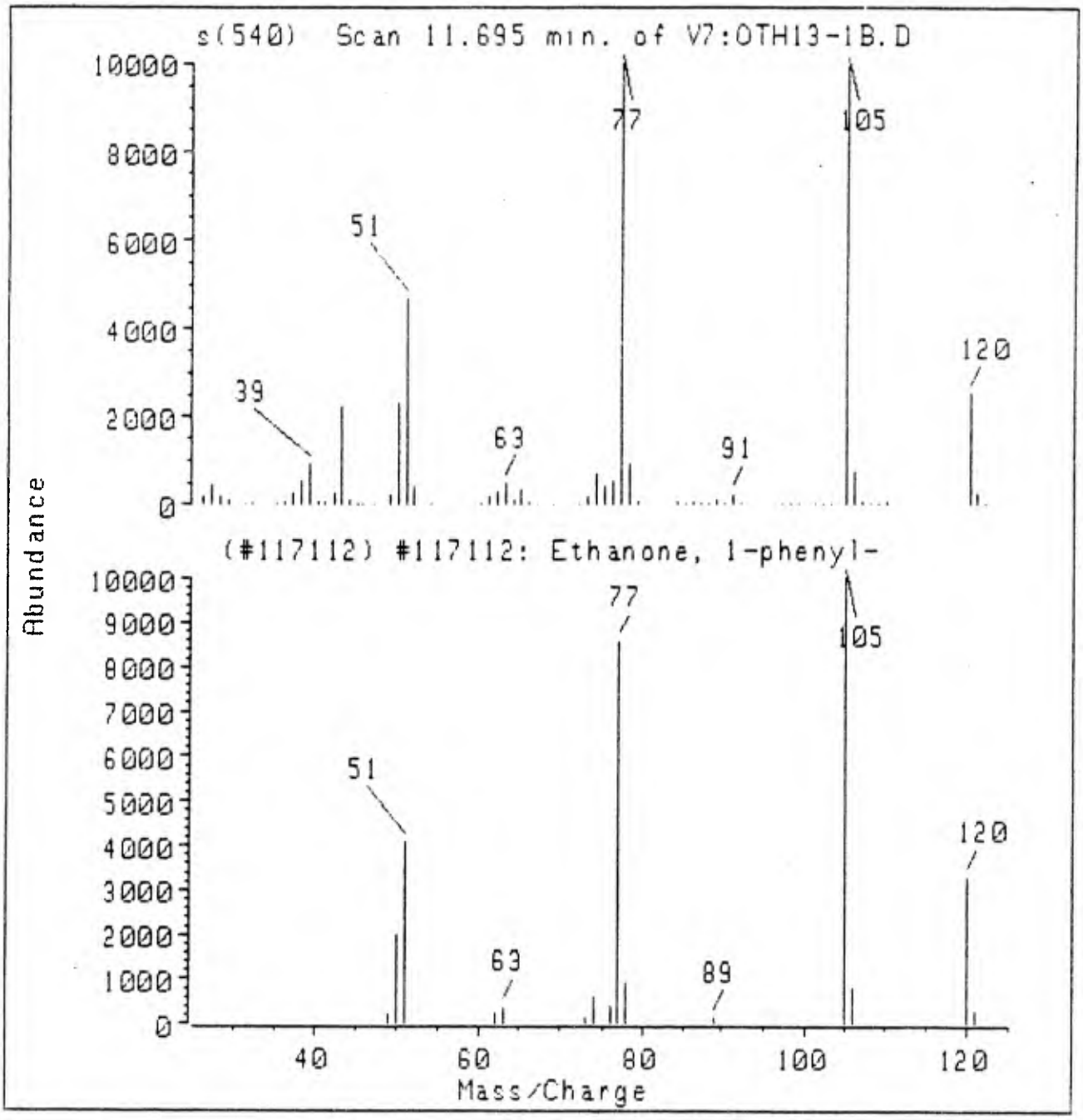
Sample OTH-1393-1b



1. Acetophenone
2. Chloroacetophenone
3. Benzoic acid
4. Diphenyl disulfide

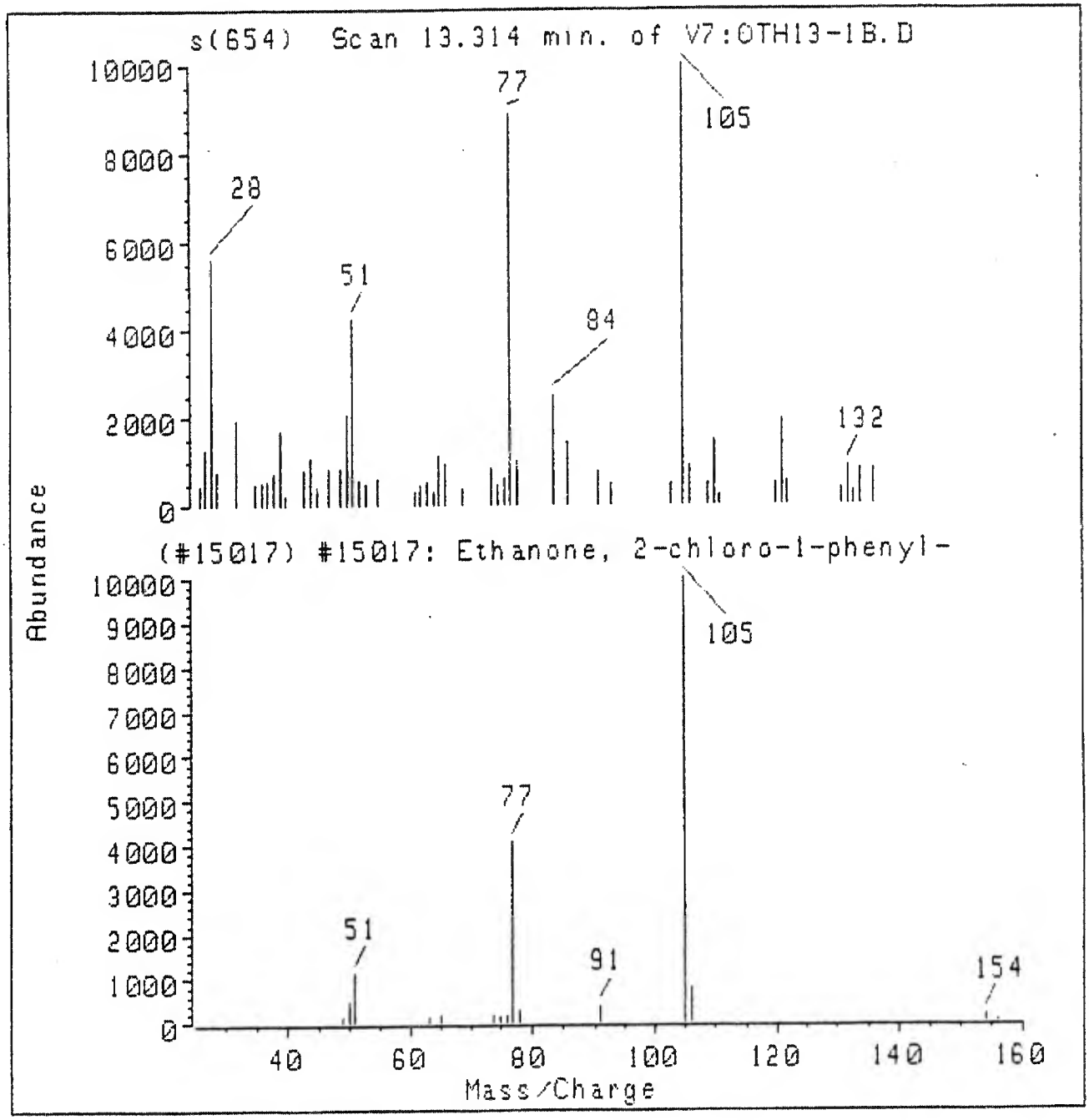
Acetophenone

OTH-1393-1b

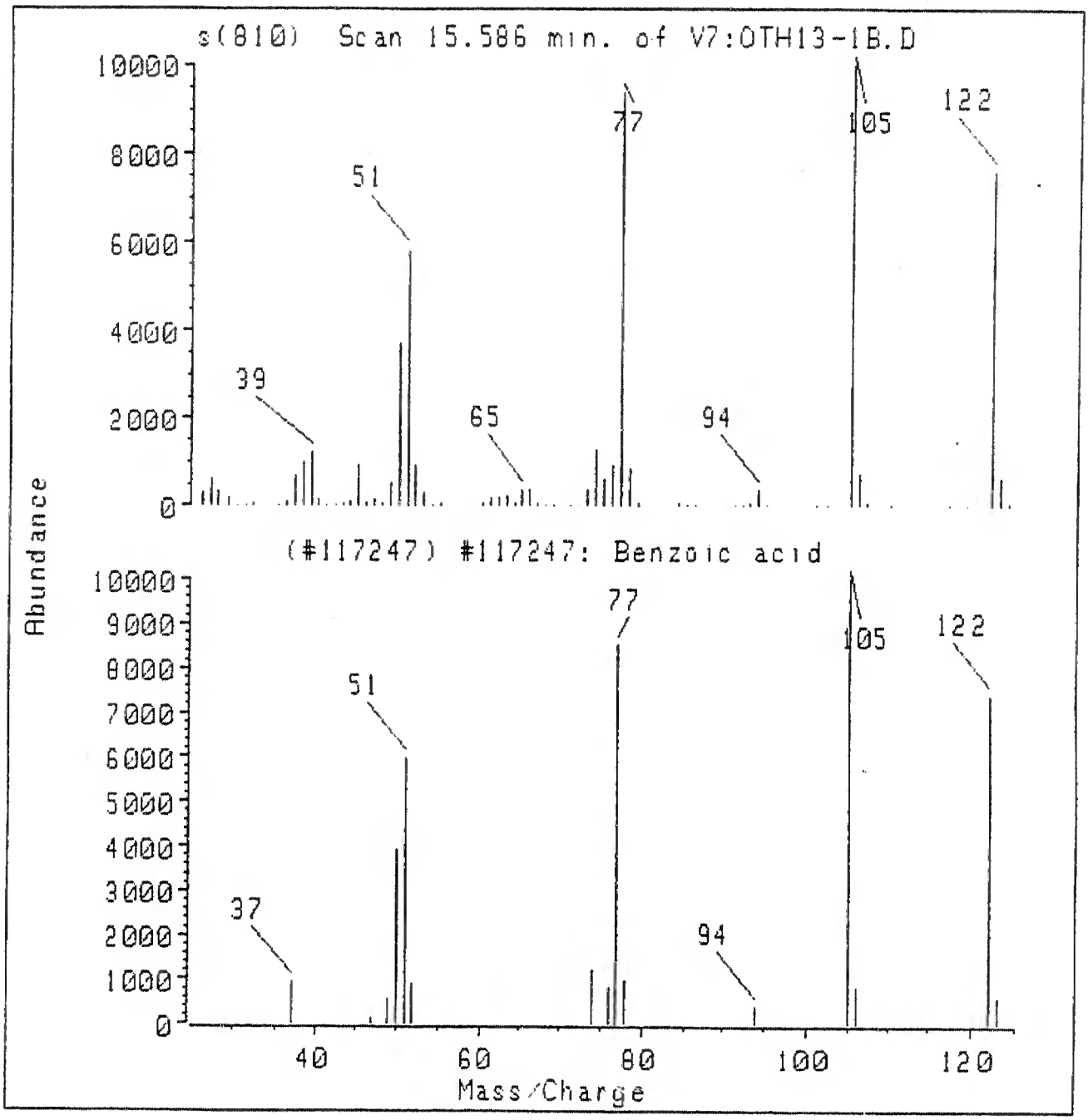


Chloroacetophenone

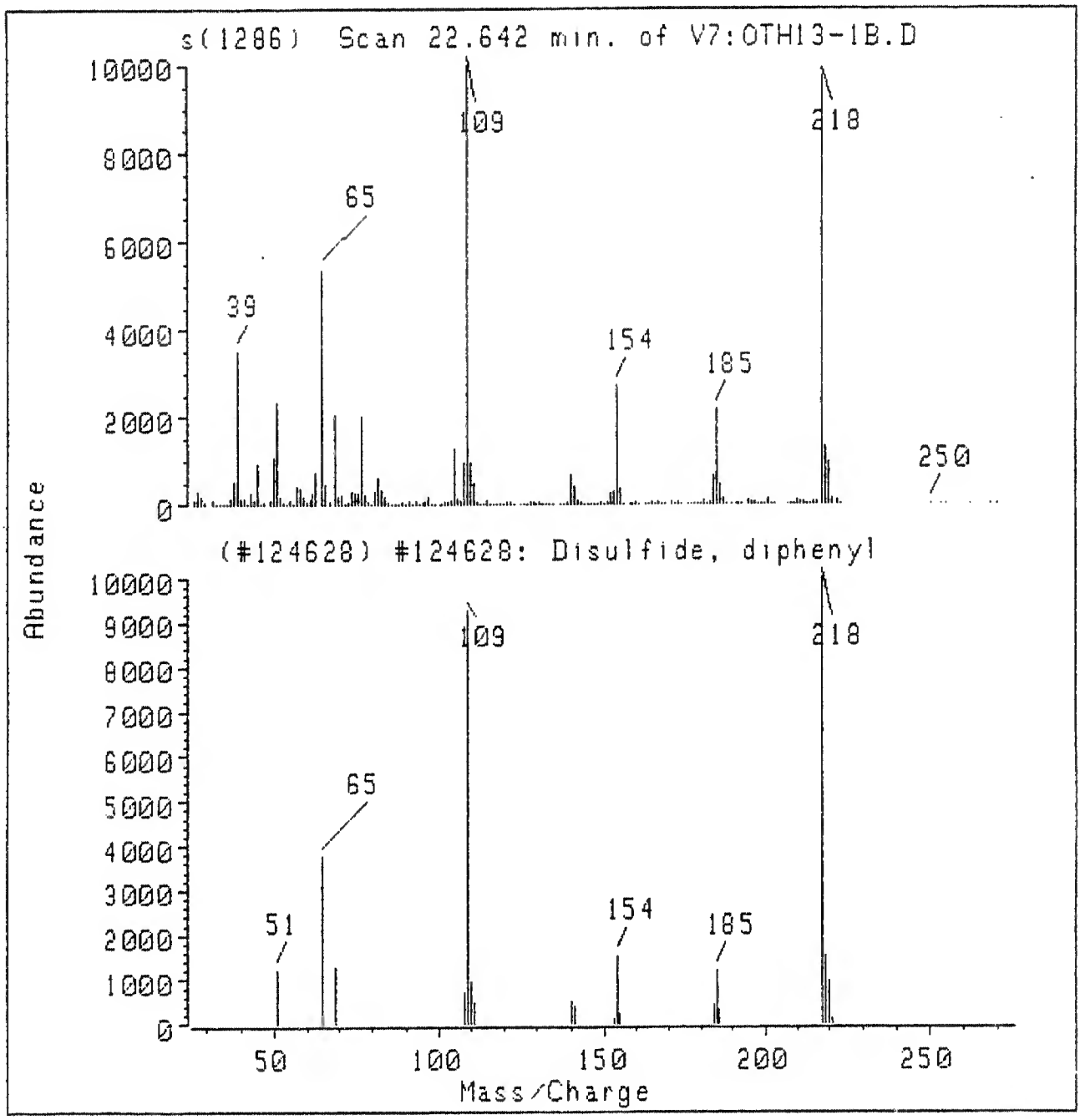
OTH-1393-1b



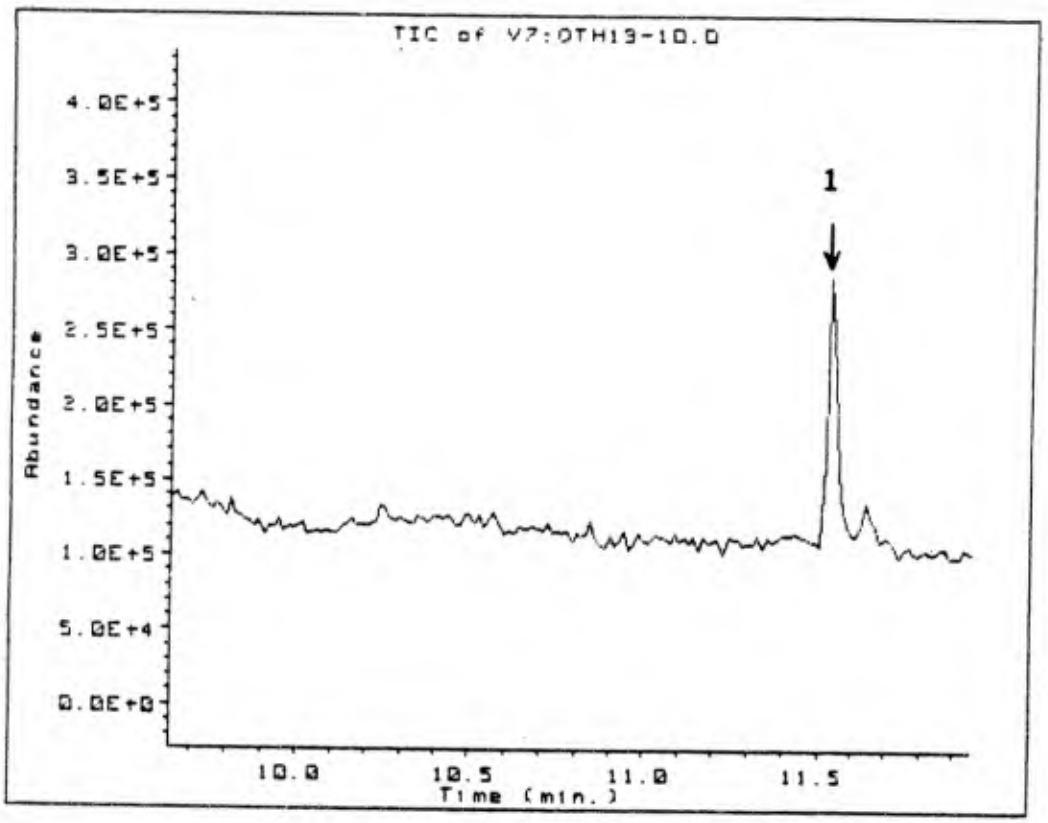
OTH-1393-1b


s(810) Scan 15.586 min. of V7:OTH13-1B.D
10000
8000
6000
4000
2000
0
39
51
65
77
94
105
122
(#117247) #117247: Benzoic acid
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
37
51
77
94
105
122
Abundance
40
60
80
100
120
Mass/Charge

OTH-1393-1b


s(1286) Scan 22.642 min. of V7:OTH13-1B.D
10000
8000
6000
4000
2000
0
109
218
65
39
154
185
250
(#124628) #124628: Disulfide, diphenyl
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
109
218
65
51
154
185
50
100
150
200
250
Abundance
Mass/Charge

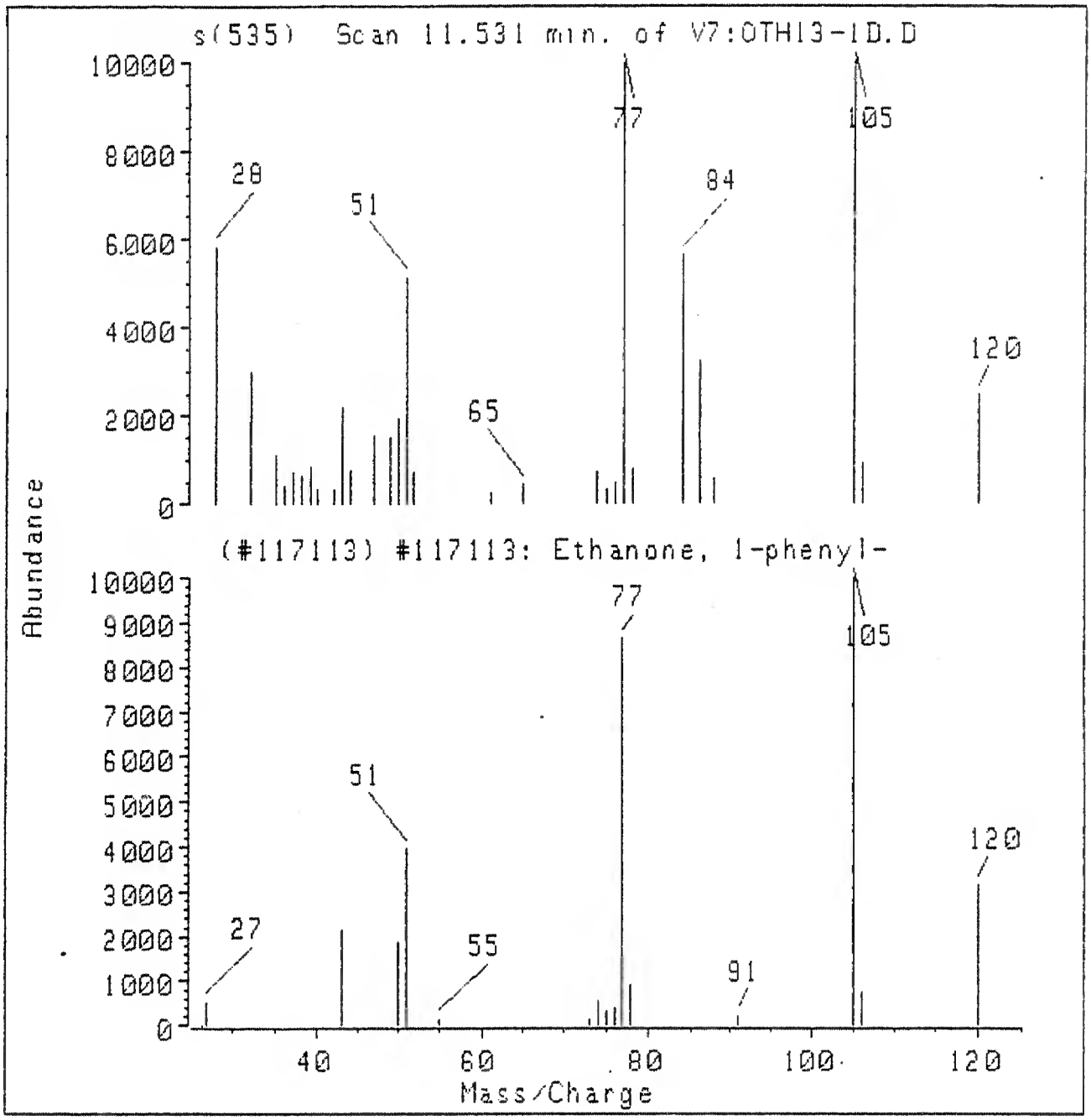
Sample OTH-1393-1d



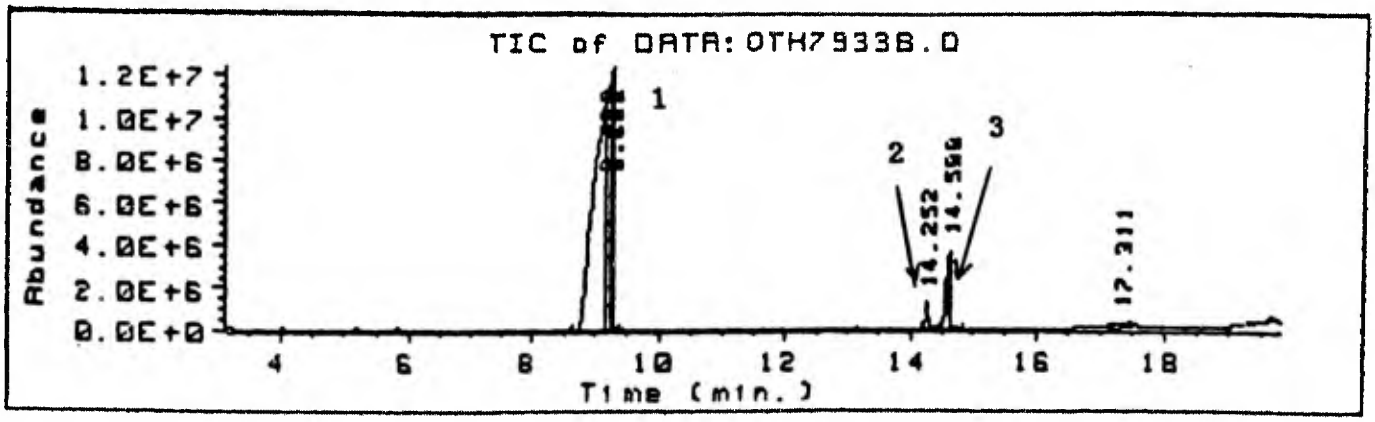
1. Acetophenone

Acetophenone

OTH-1393-1d

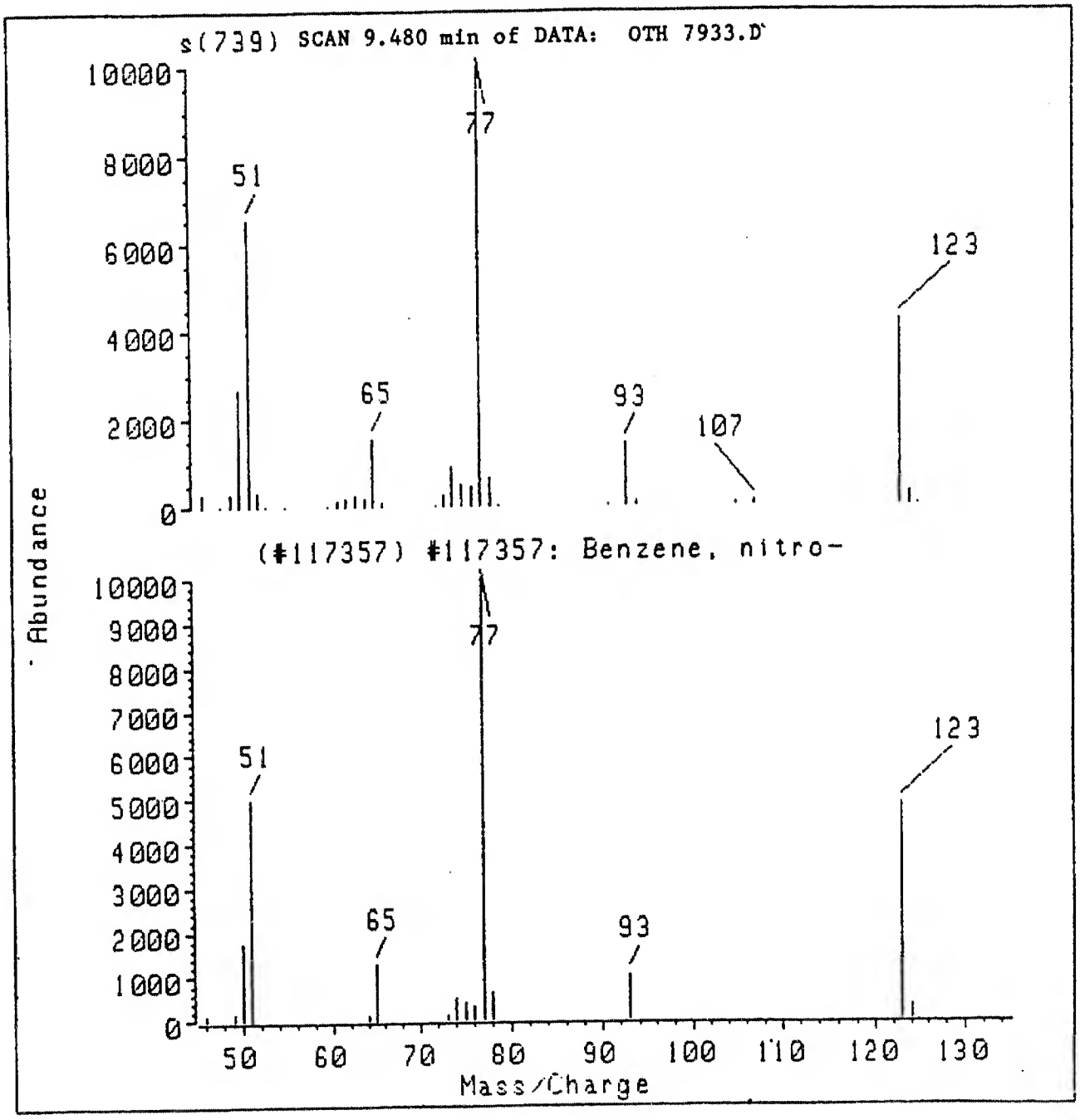


Sample OTH-793-2



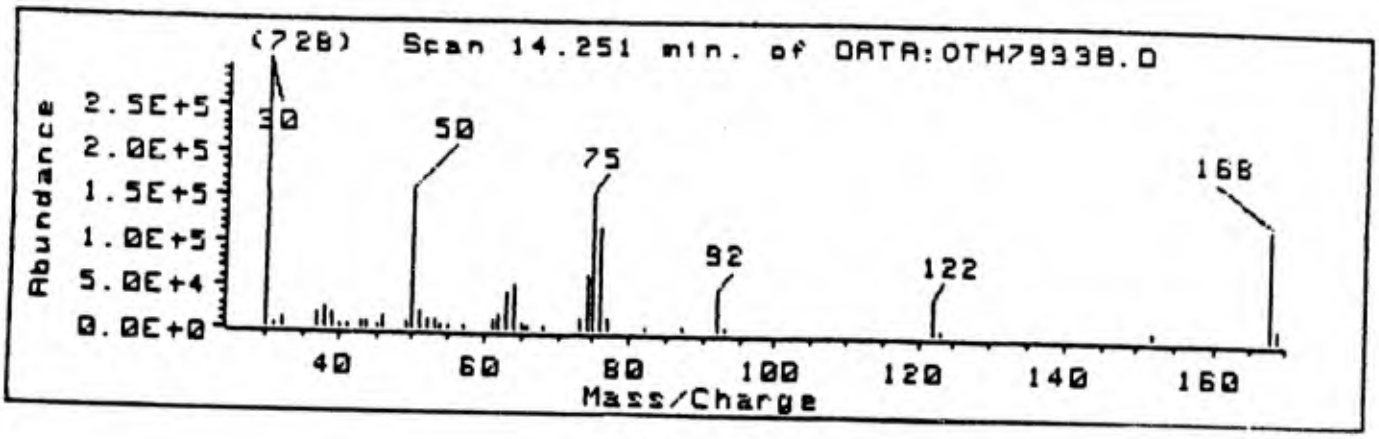
sample Name : ETHER EXTRACT OTH 793-3 DIRECT INJECTION

1. **Nitrobenzene**
2. **1,4-dinitrobenzene**
3. **1,3-dinitrobenzene**


s(739) SCAN 9.480 min of DATA: OTH 7933.D
10000
8000
6000
4000
2000
0
77
51
65
93
107
123
(#117357) #117357: Benzene, nitro-
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
77
51
65
93
123
Abundance
50 60 70 80 90 100 110 120 130
Mass/Charge

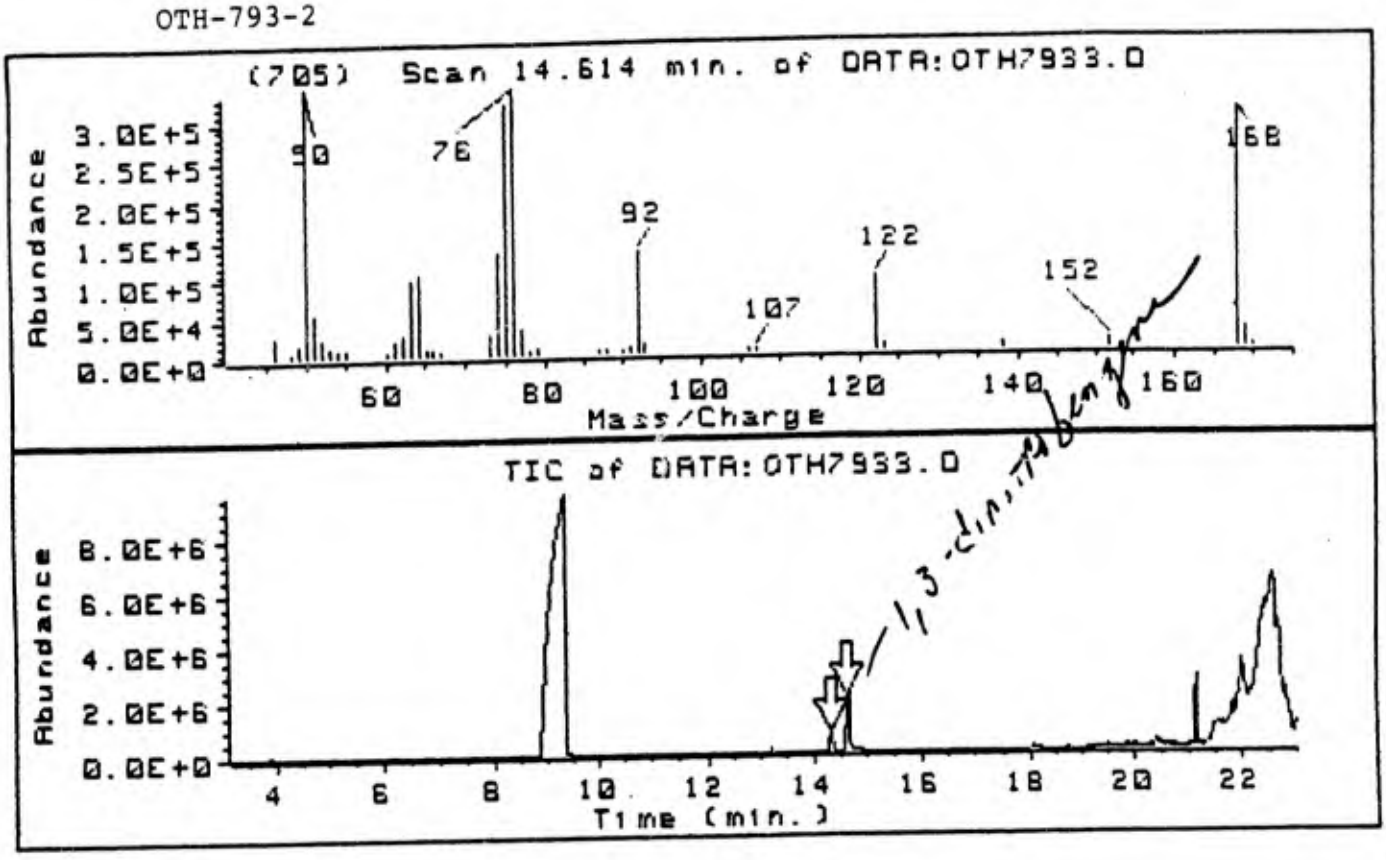
1,4-Dinitrobenzene

Ready to inject OTH-793-2

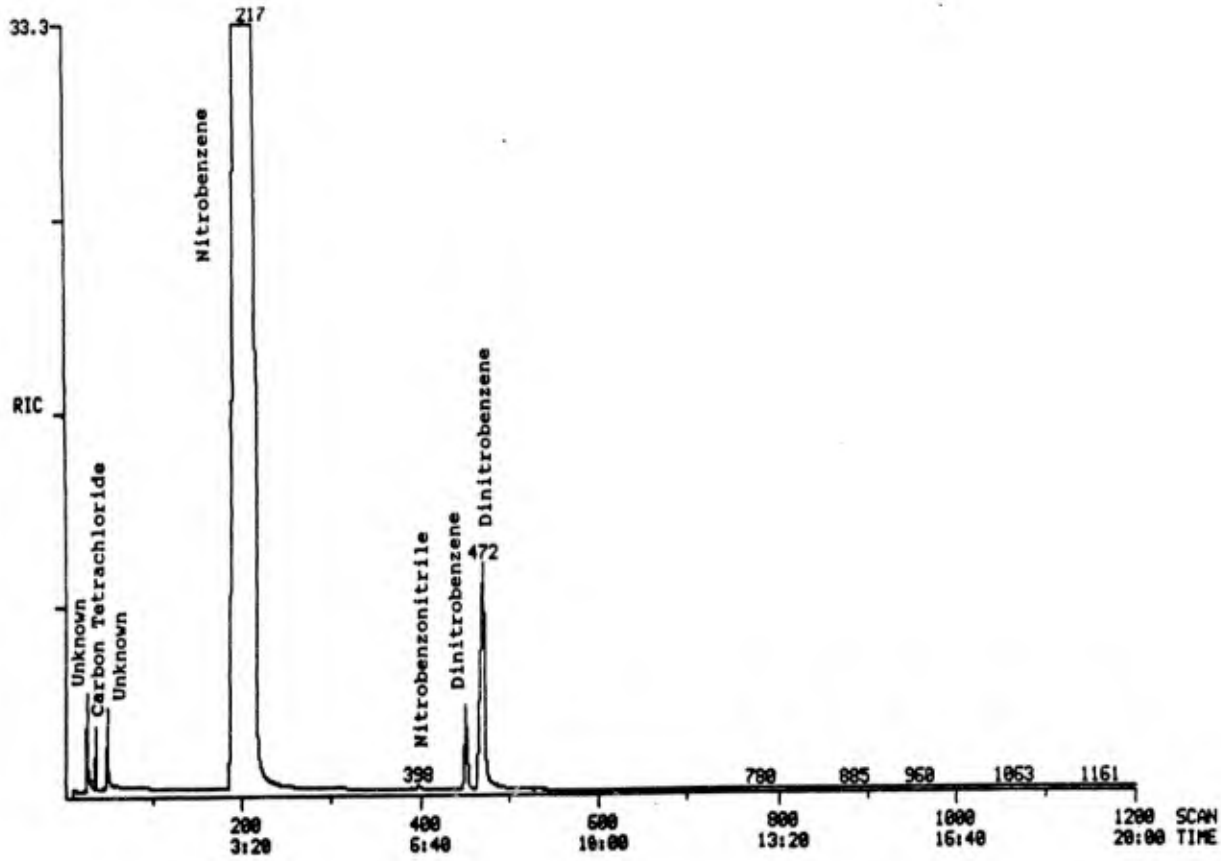


1,3-Dinitrobenzene

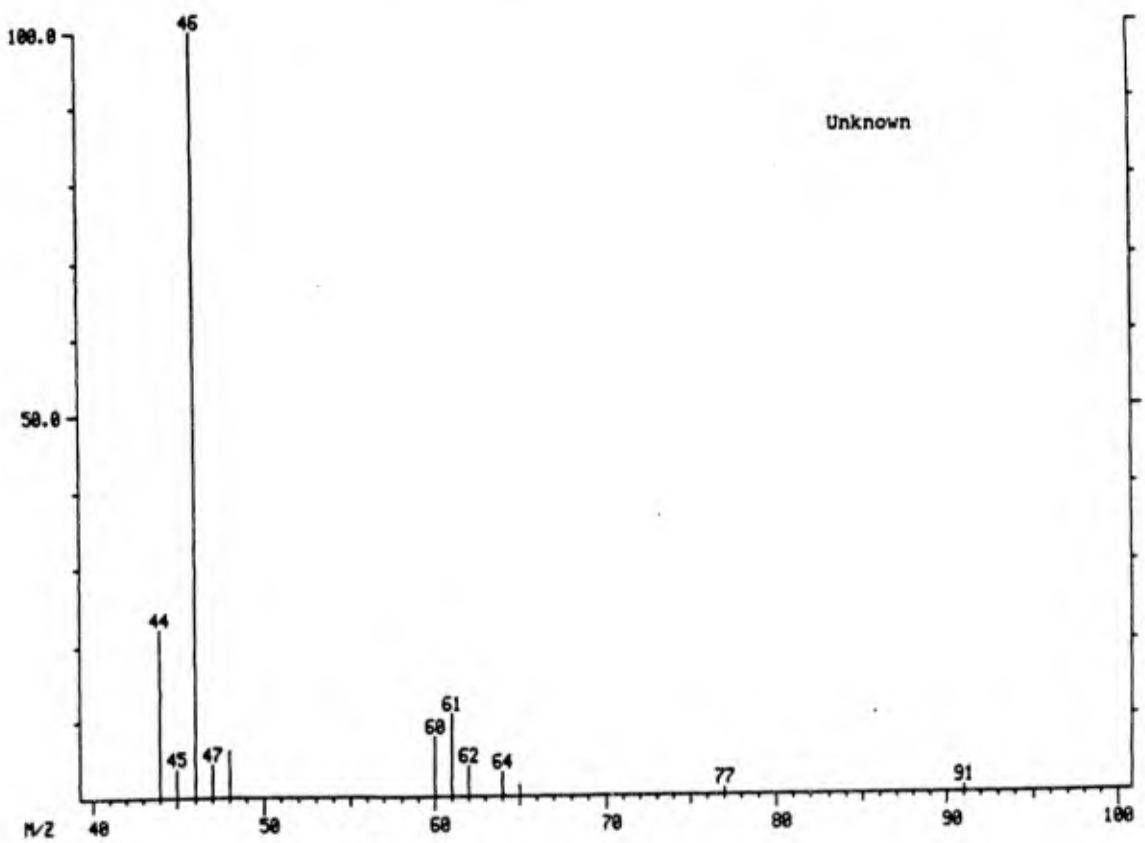
OTH-793-2



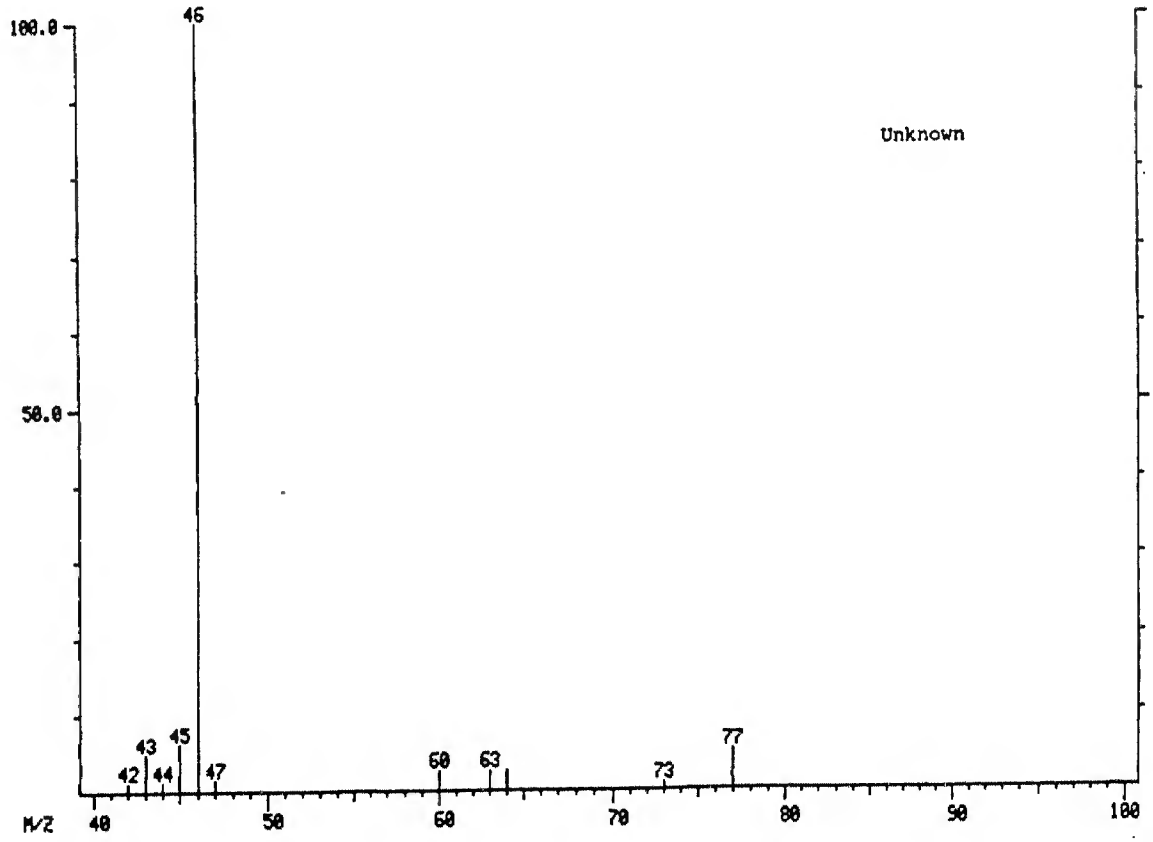
GC/MS/EI Chromatogram of OTH-793-2



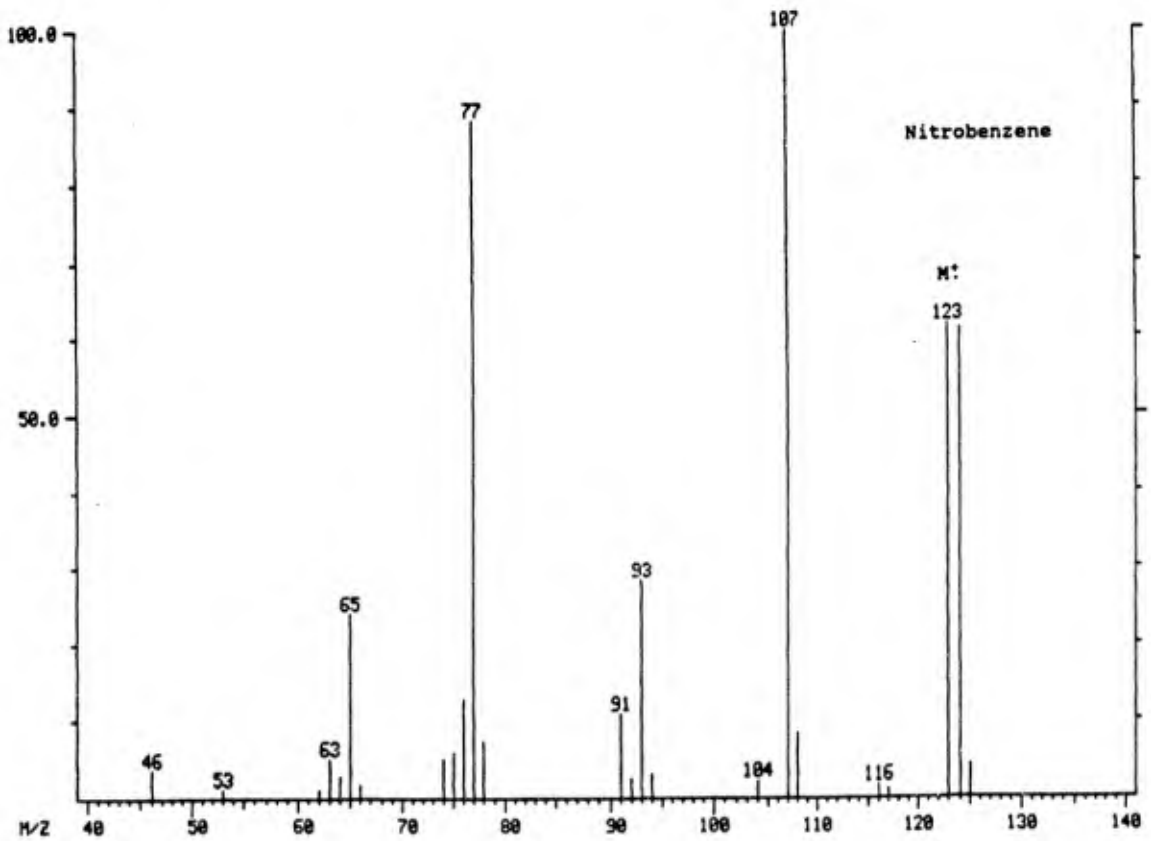
EI Mass Spectrum of OTH-793-2 Scan 26



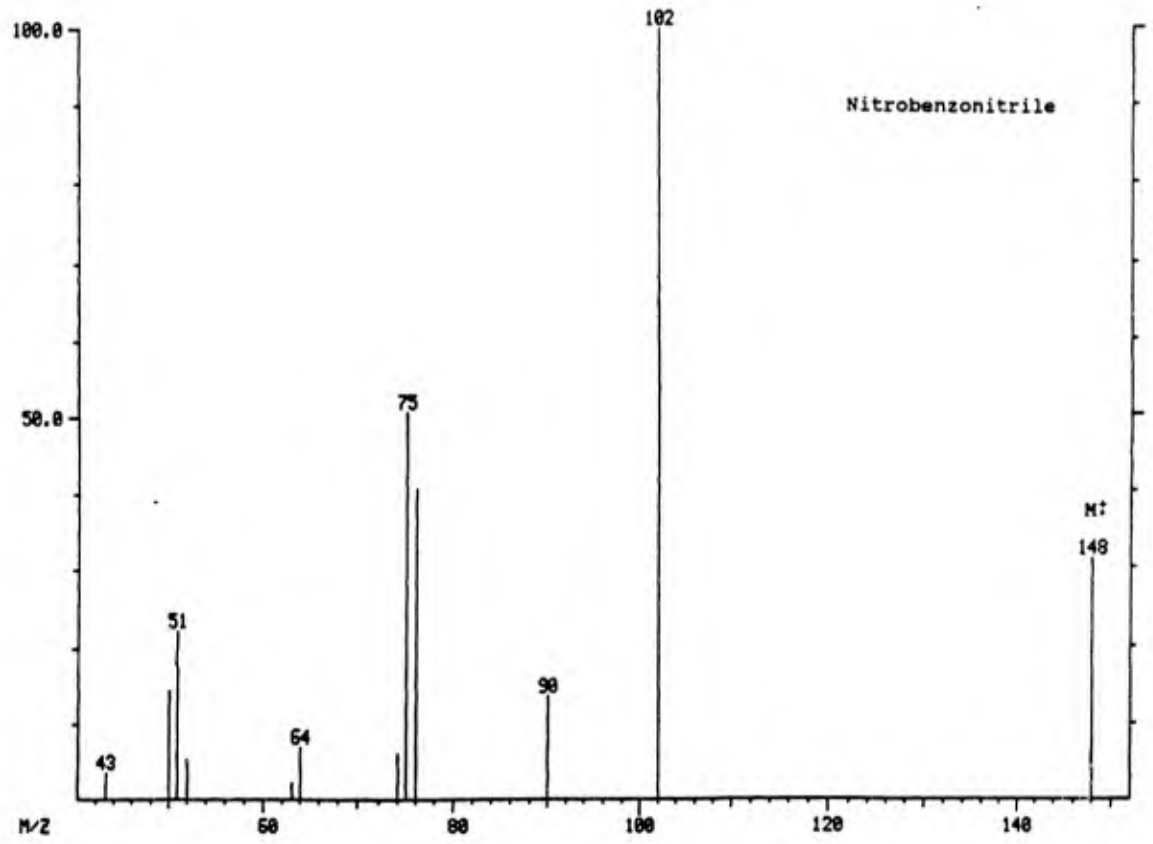
EI Mass Spectrum of OTH-793-2 Scan 49



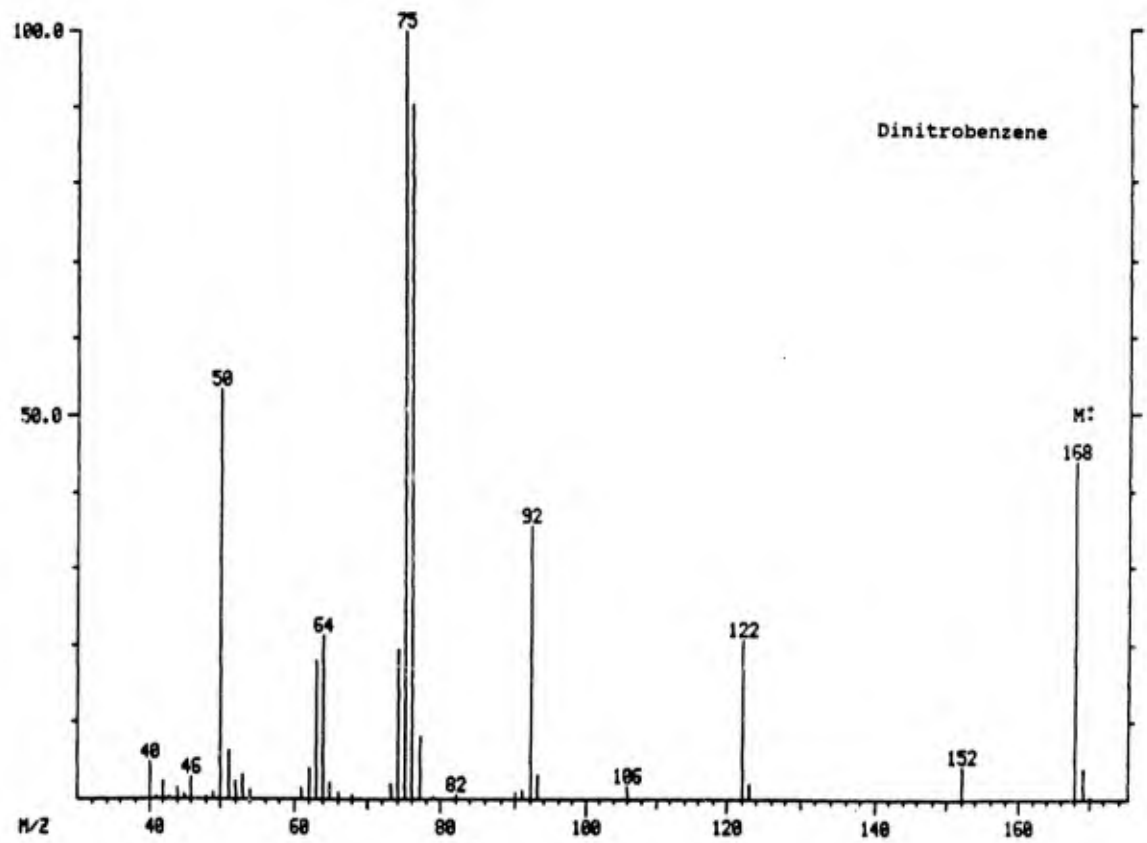
EI Mass Spectrum of OTH-793-2 Scan 217



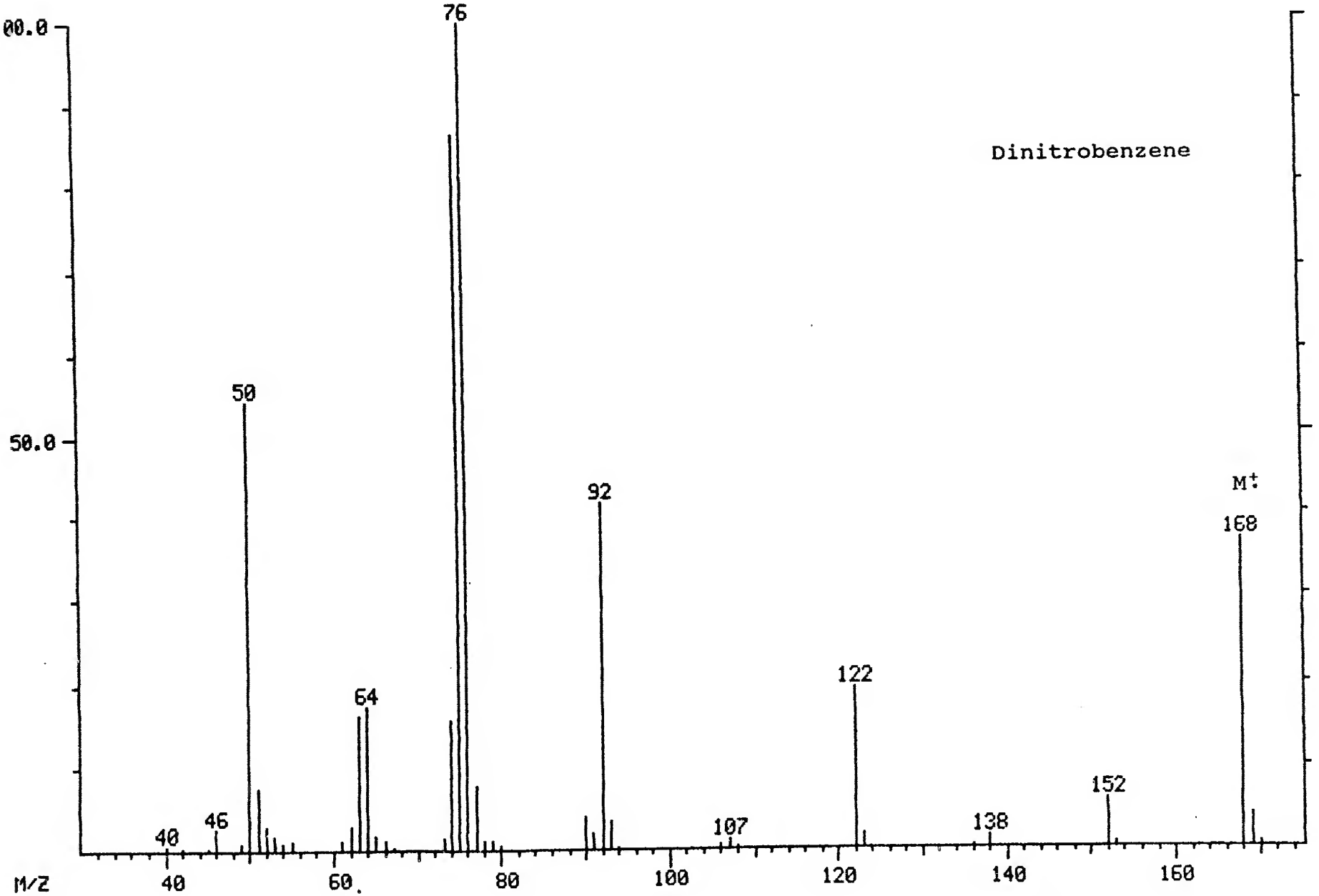
EI Mass Spectrum of OTH-793-2 Scan 398



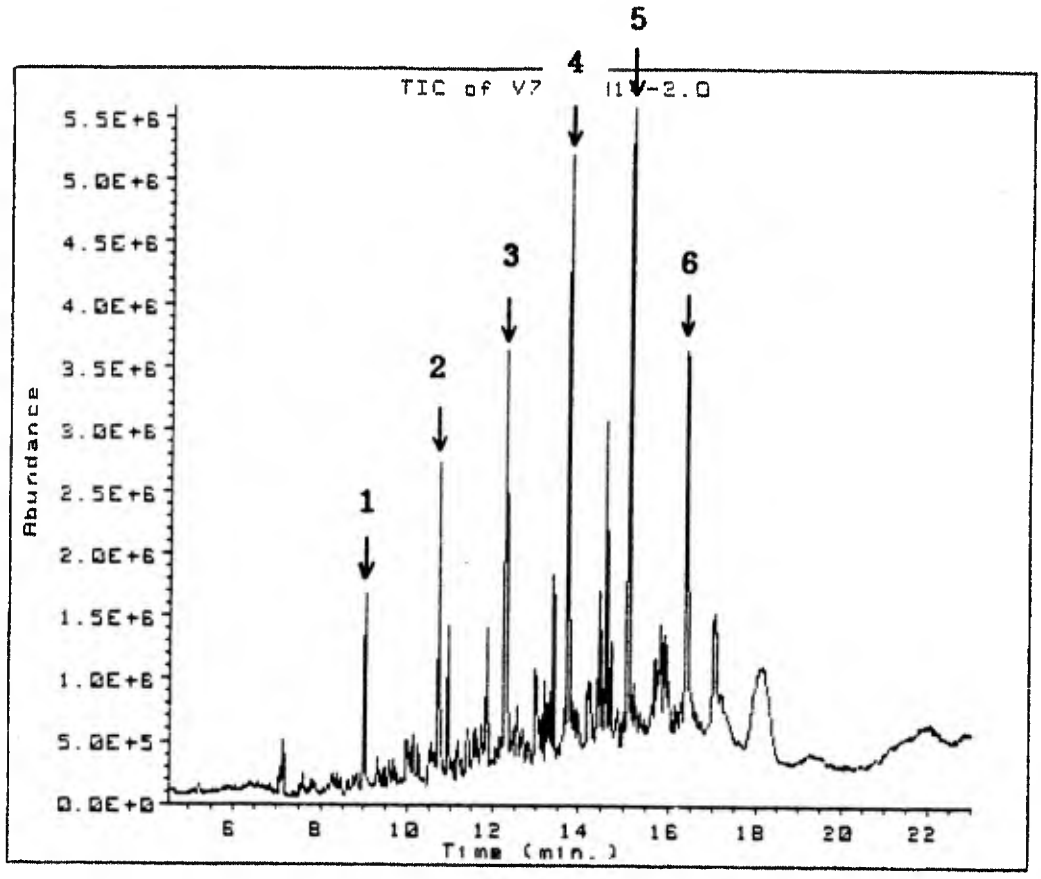
EI Mass Spectrum of OTH-793-2 Scan 452



EI Mass Spectrum of OTH-793-2 Scan 472

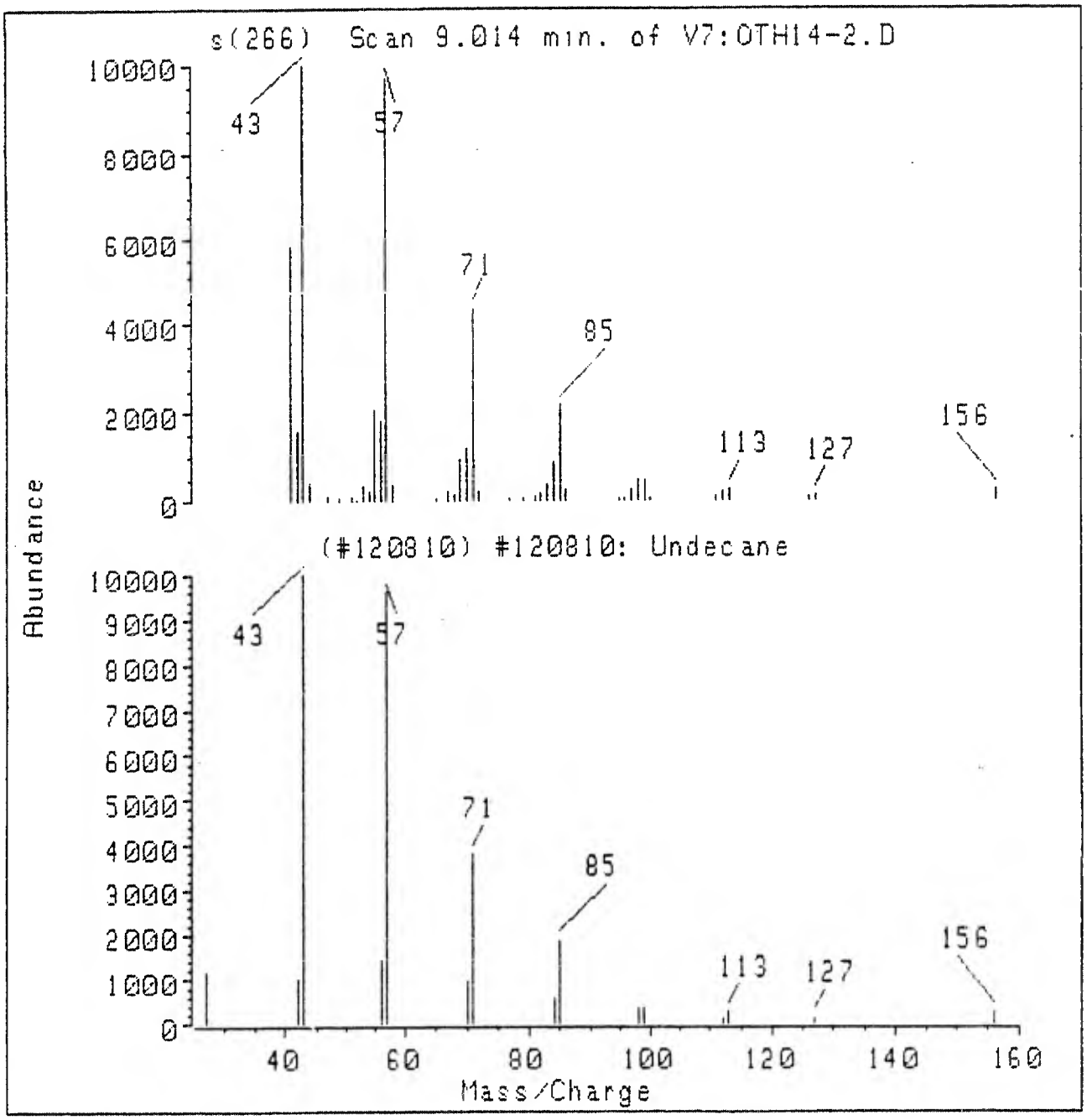


Sample OTH-1493-2

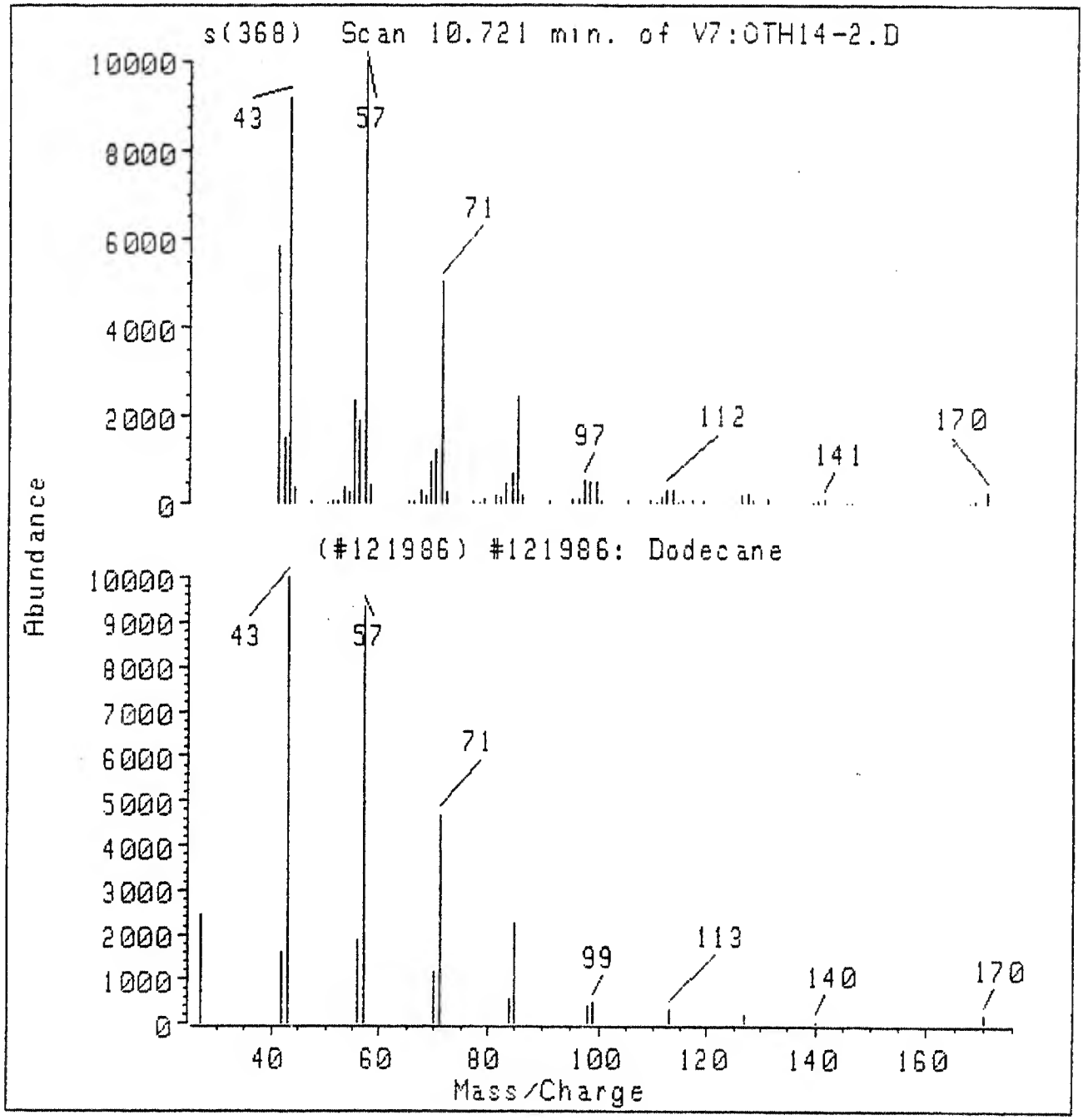


1. Undecane
2. Dodecane
3. Tridecane
4. Tetradecane
5. Pentadecane
6. Hexadecane

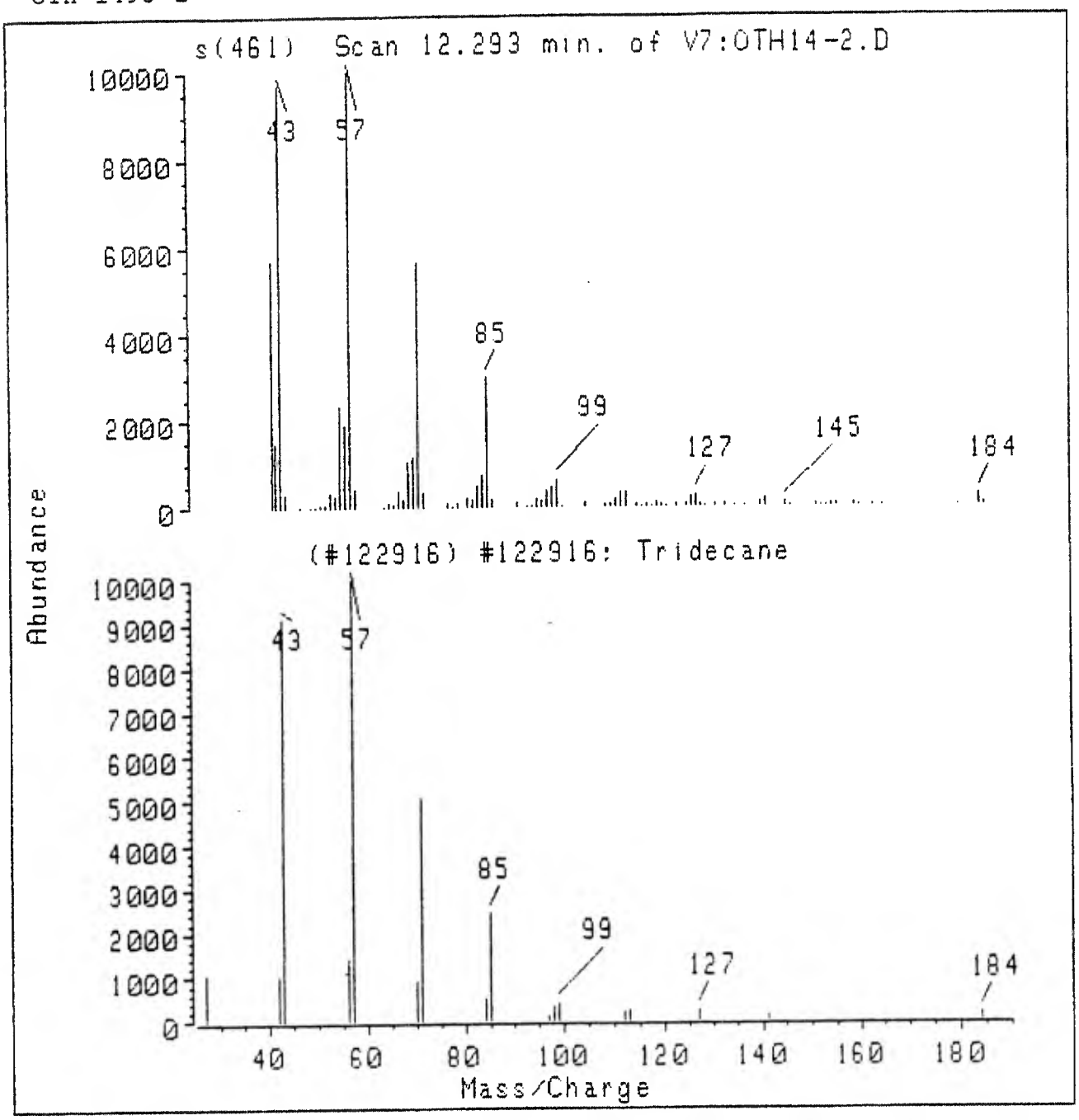
OTH-1493-2


s(266) Scan 9.014 min. of V7:OTH14-2.D
10000
8000
6000
4000
2000
0
43
57
71
85
113
127
156
(#120810) #120810: Undecane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
43
57
71
85
113
127
156
Abundance
40
60
80
100
120
140
160
Mass/Charge

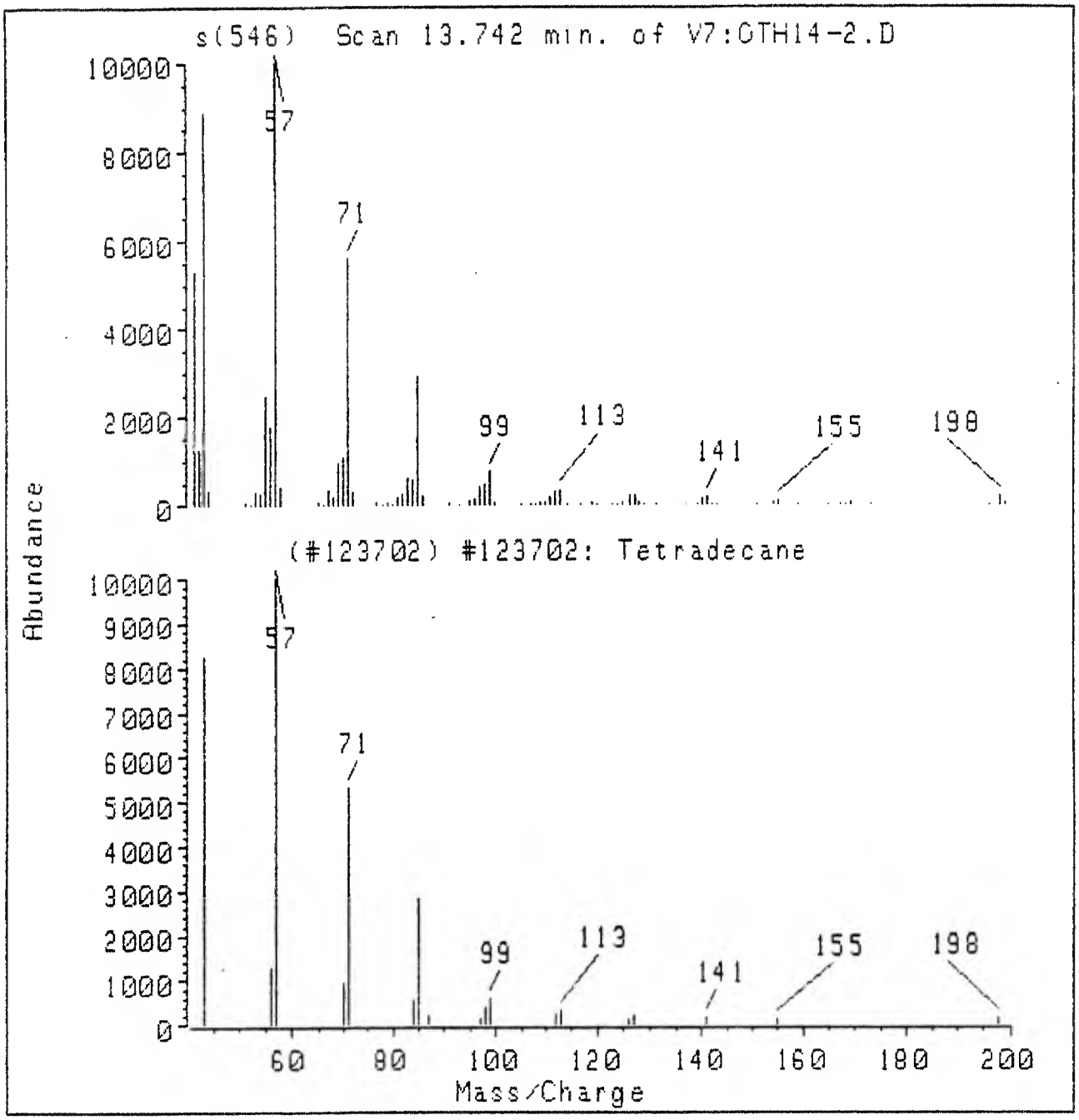
OTH-1493-2


s(368) Scan 10.721 min. of V7:OTH14-2.D
10000
8000
6000
4000
2000
0
43
57
71
97
112
141
170
(#121986) #121986: Dodecane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
43
57
71
99
113
140
170
Abundance
40
60
80
100
120
140
160
Mass/Charge

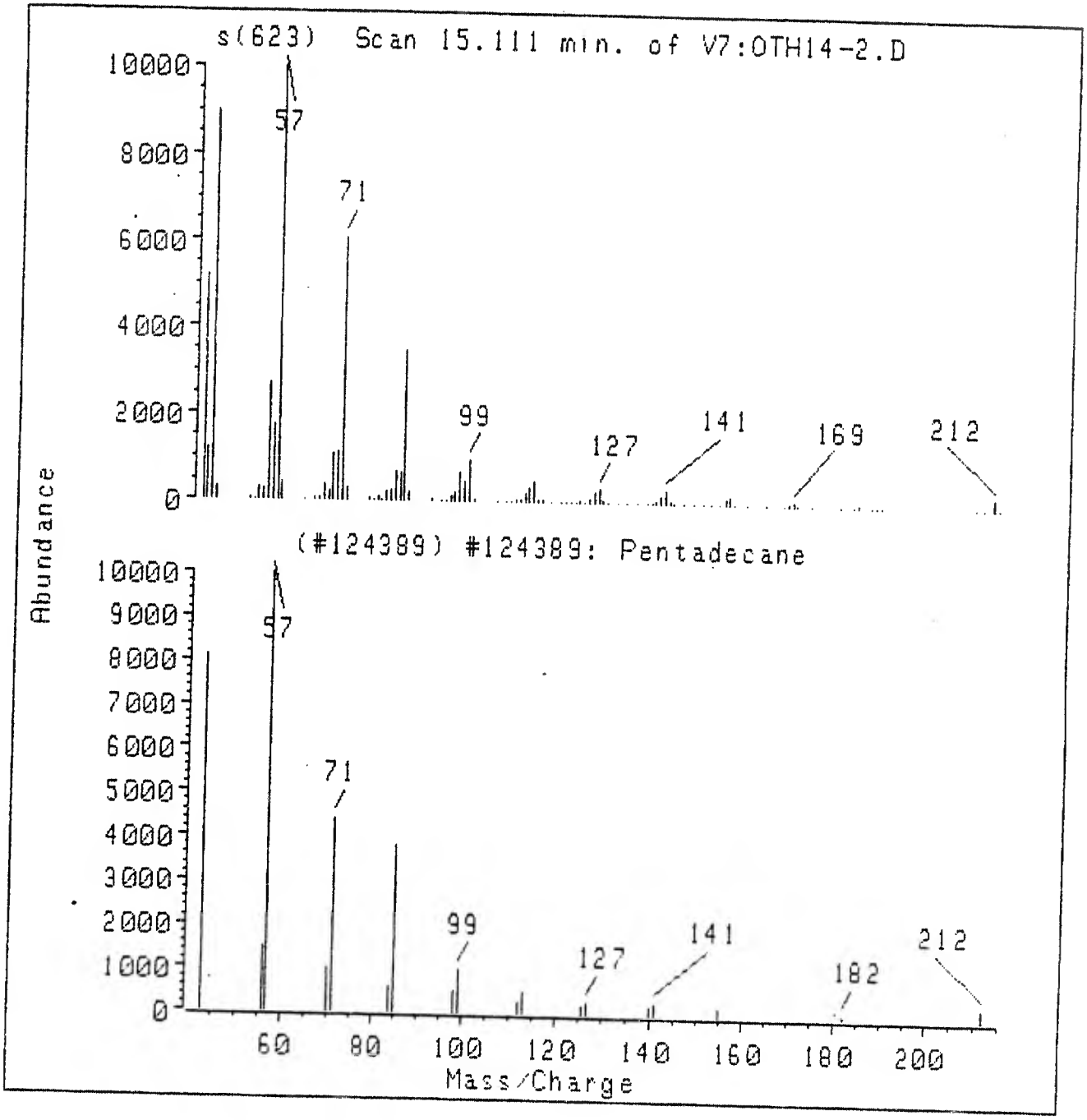
OTH-1493-2


s(461) Scan 12.293 min. of V7:OTH14-2.D
10000
8000
6000
4000
2000
0
43
57
85
99
127
145
184
(#122916) #122916: Tridecane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
43
57
85
99
127
184
Abundance
40
60
80
100
120
140
160
180
Mass/Charge

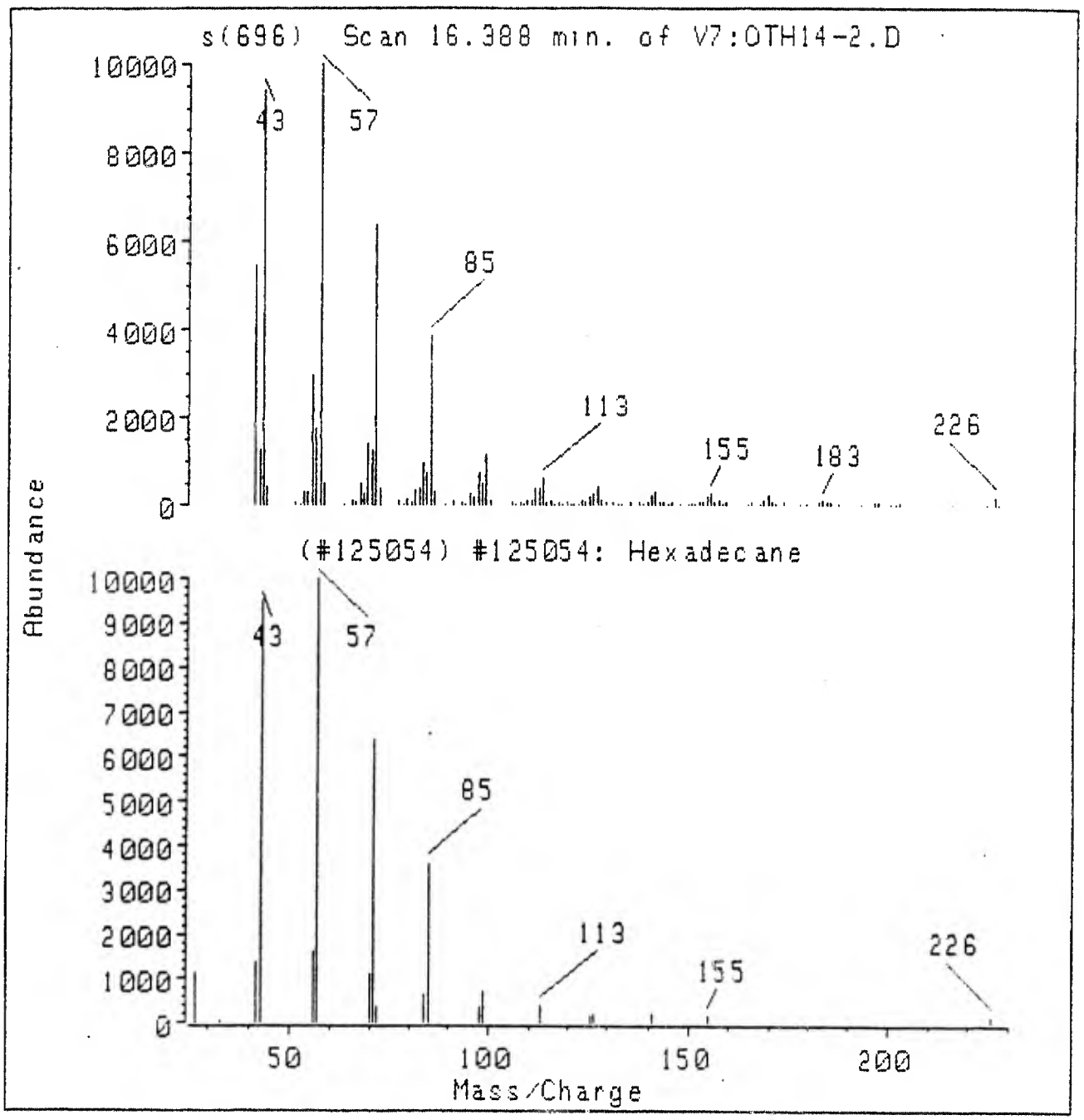
OTH-1493-2


s(546) Scan 13.742 min. of V7:OTH14-2.D
10000
8000
6000
4000
2000
0
57
71
99
113
141
155
198
(#123702) #123702: Tetradecane
Abundance
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
57
71
99
113
141
155
198
60
80
100
120
140
160
180
200
Mass/Charge

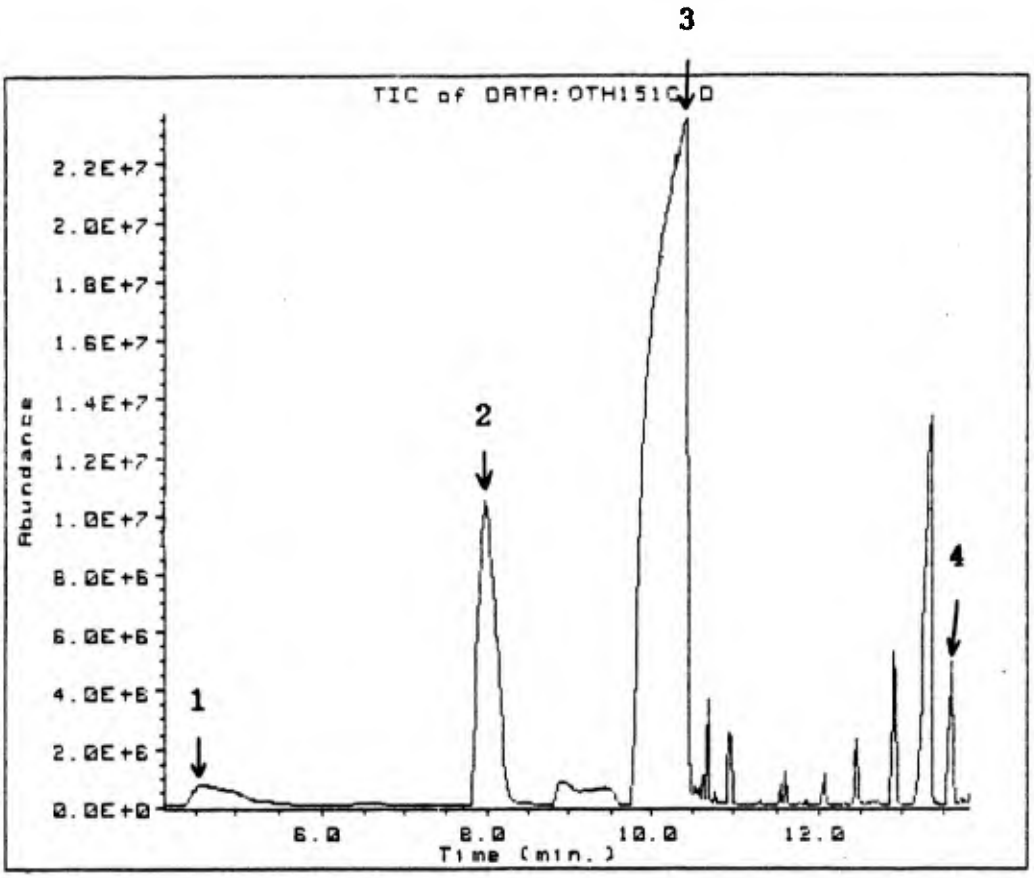
OTH-1493-2


s(623) Scan 15.111 min. of V7:OTH14-2.D
10000
8000
6000
4000
2000
0
57
71
99
127
141
169
212
(#124389) #124389: Pentadecane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
57
71
99
127
141
182
212
Abundance
60
80
100
120
140
160
180
200
Mass/Charge

OTH-1493-2

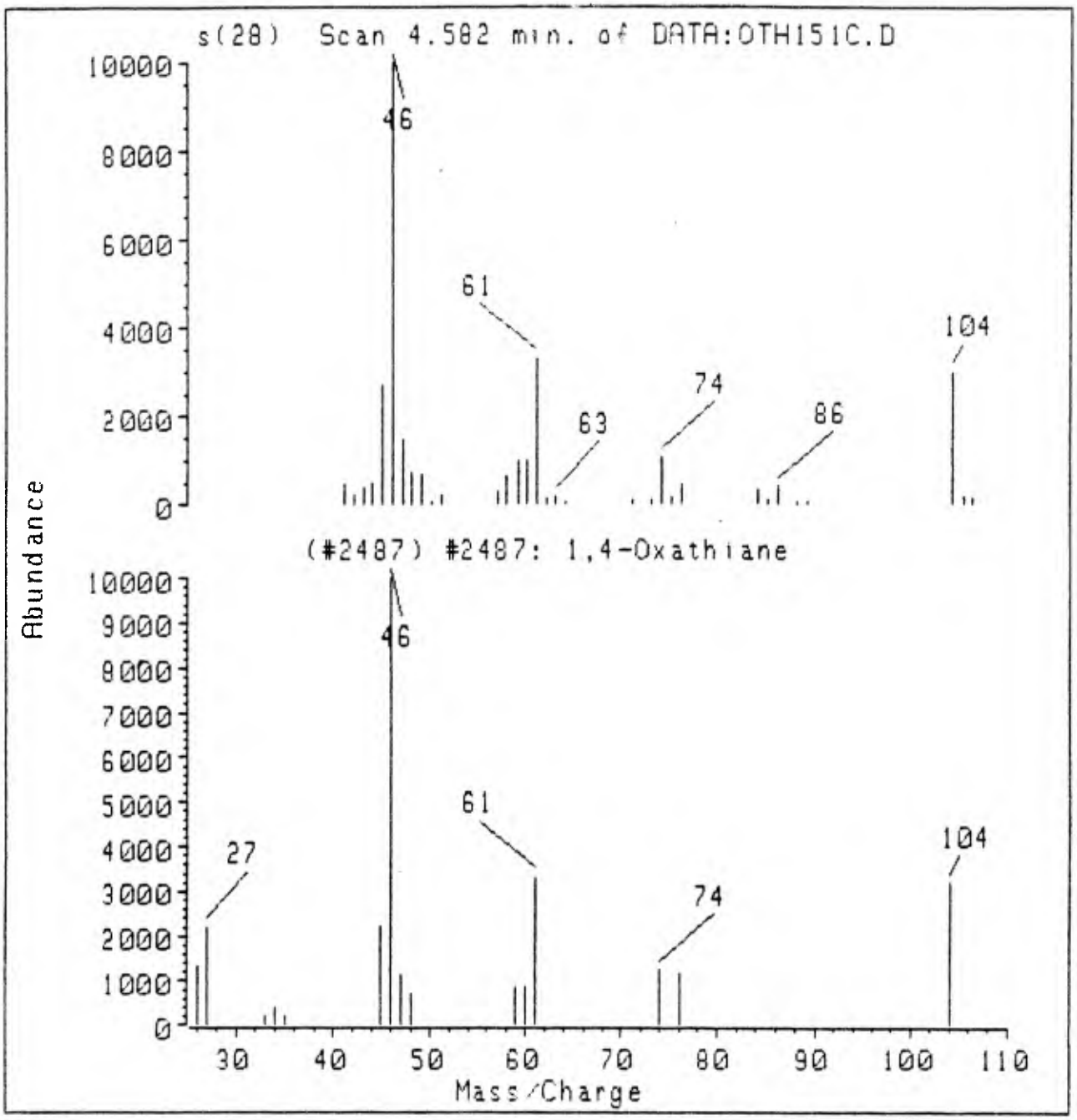

s(696) Scan 16.388 min. of V7:OTH14-2.D
10000
8000
6000
4000
2000
0
43
57
85
113
155
183
226
(#125054) #125054: Hexadecane
Abundance
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
43
57
85
113
155
226
50
100
150
200
Mass/Charge

Sample OTH-1593-1c

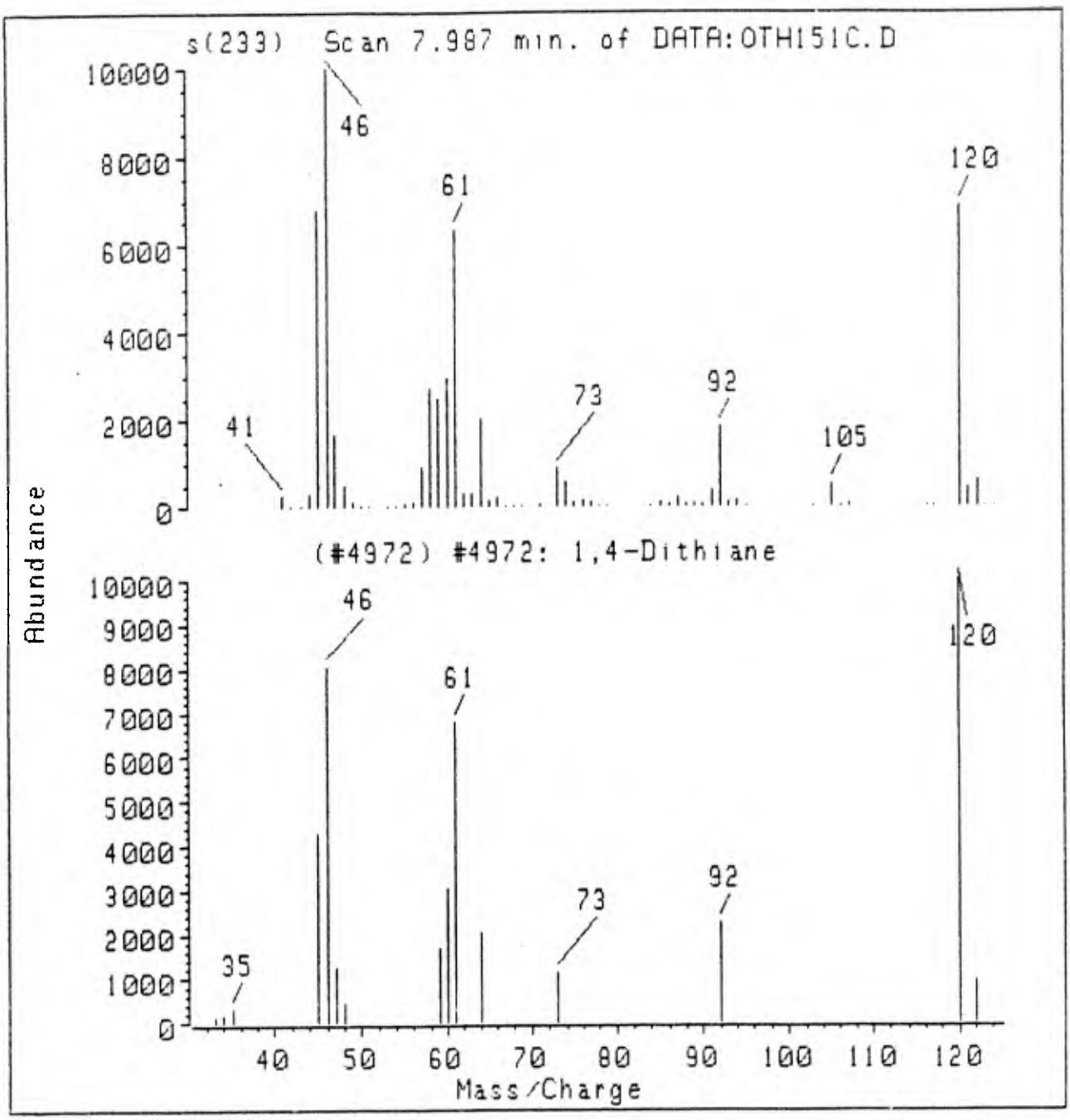


1. 1,4-Oxathiane
2. 1,4-Dithiane
3. HD
4. Thiodiglycol dimer

OTH-1593-1c

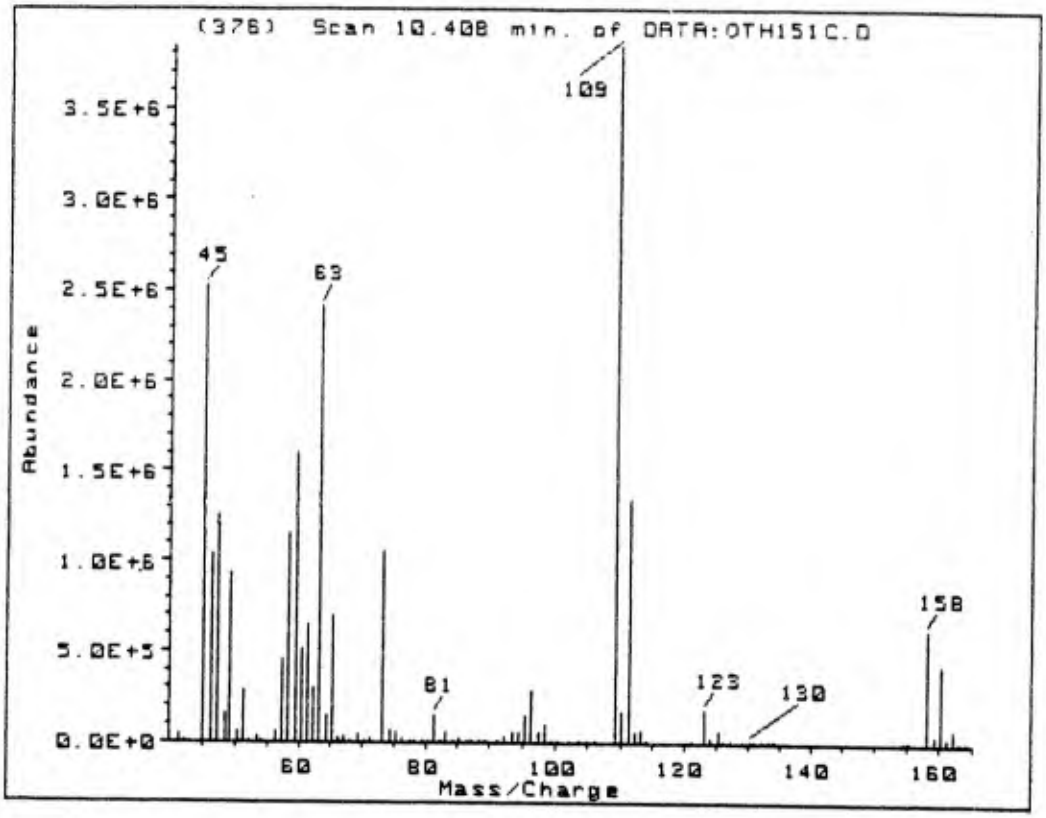

s(28) Scan 4.582 min. of DATA:OTH151C.D
(#2487) #2487: 1,4-Oxathiane
Abundance
Mass/Charge
46
61
63
74
86
104
27

OTH-1593-1c


s(233) Scan 7.987 min. of DATA:OTH151C.D
10000
8000
6000
4000
2000
0
46
61
120
73
92
41
105
(#4972) #4972: 1,4-Dithiane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
46
61
120
73
92
35
Abundance
40
50
60
70
80
90
100
110
120
Mass/Charge

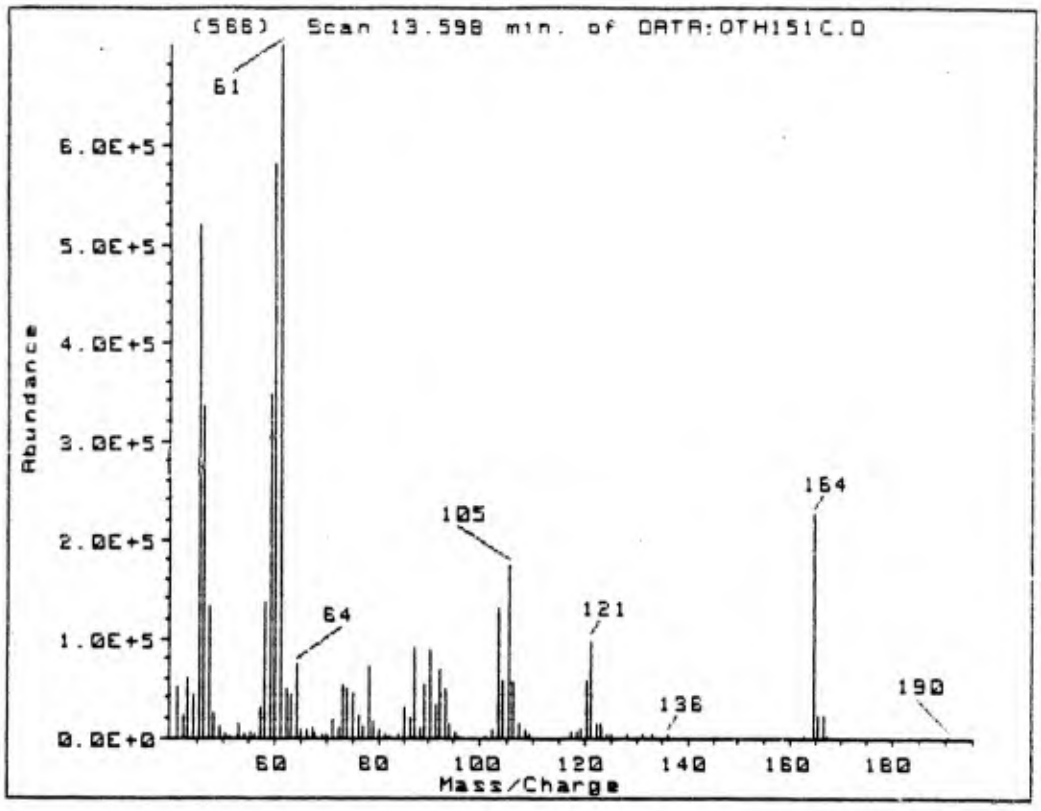
HD

OTH-1593-1c

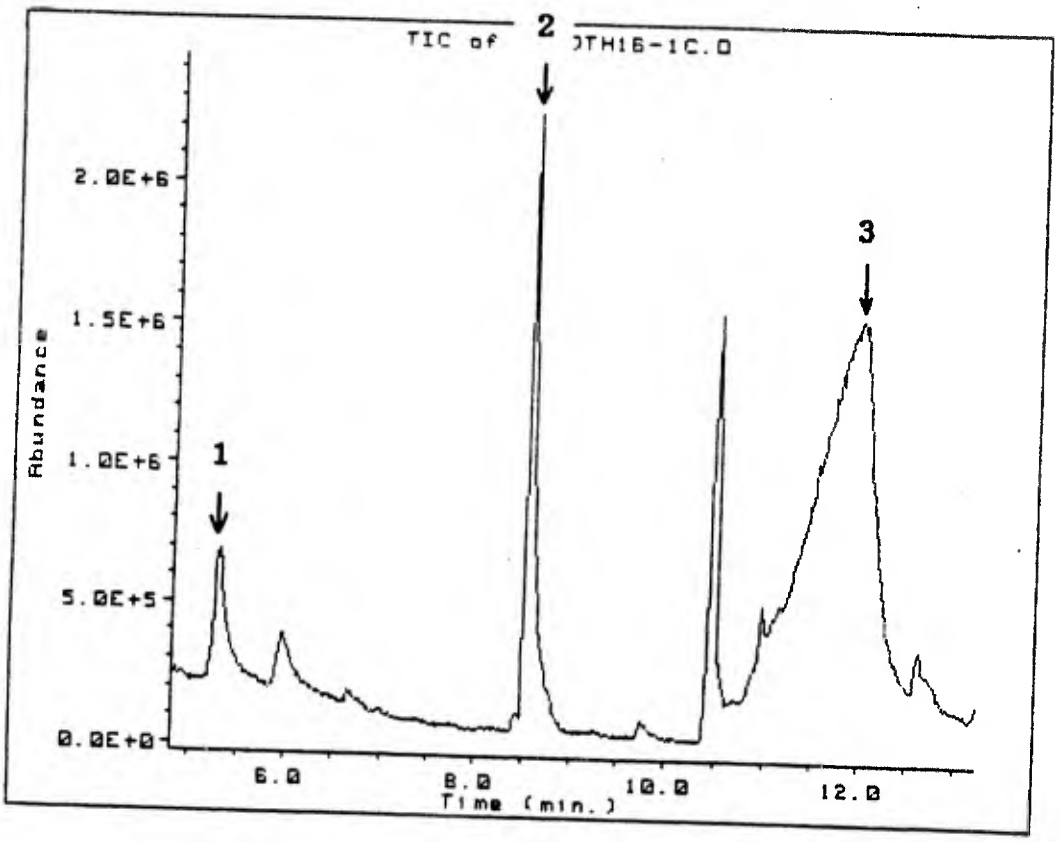


Thiodiglycol dimer

OTH-1593-1c

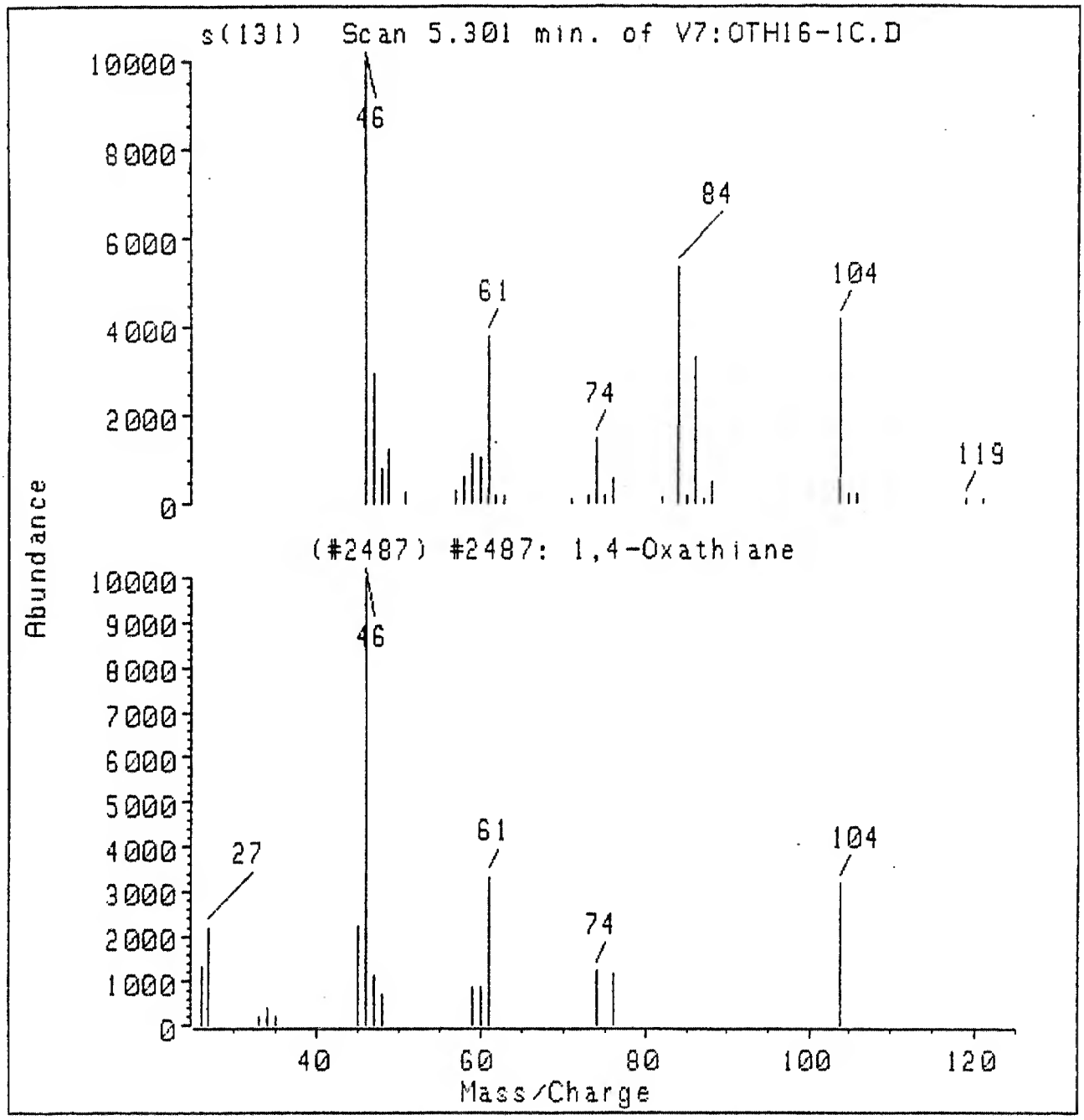


Sample OTH-1693-1c

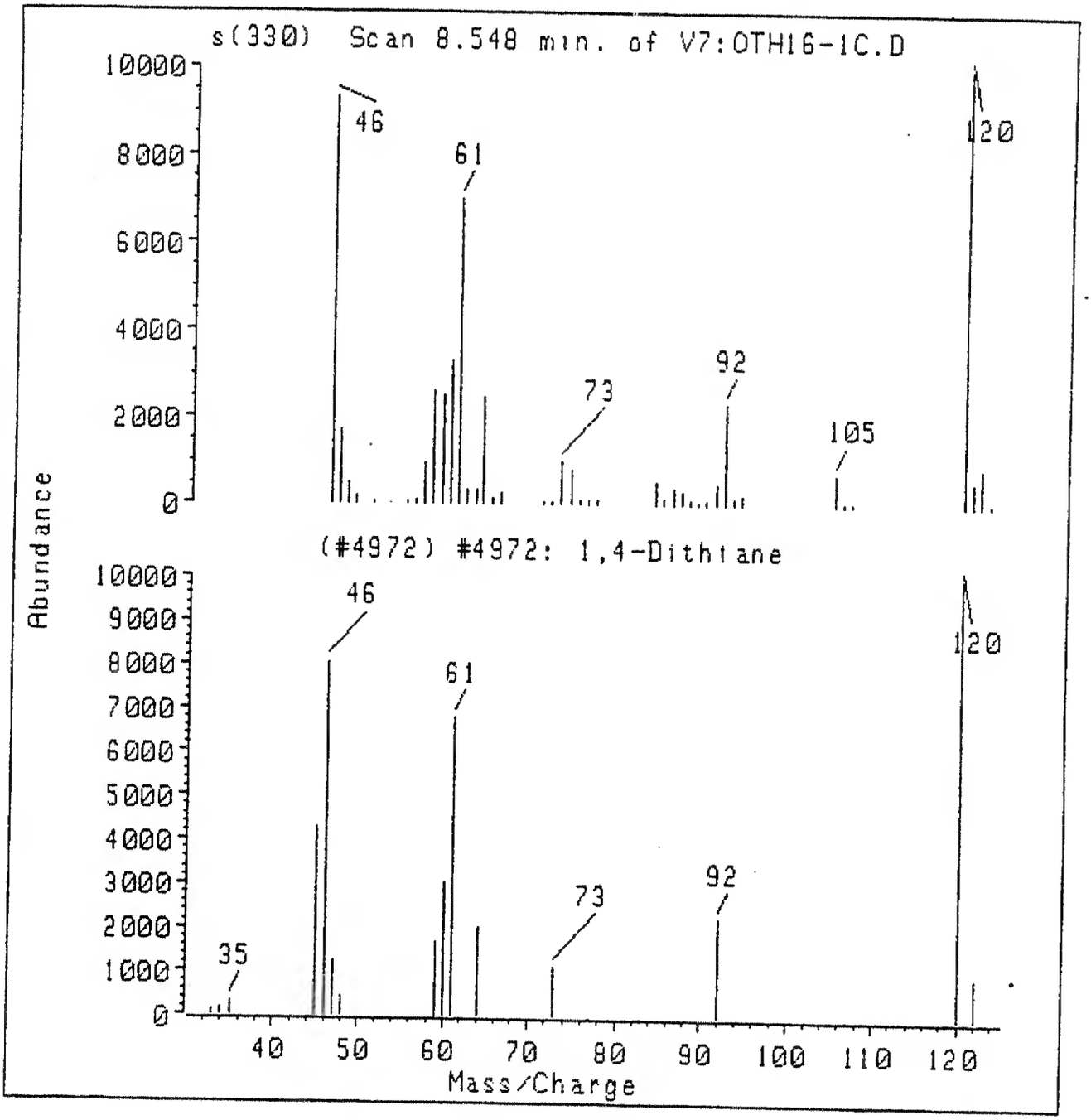


1. 1,4-Oxathiane
2. 1,4-Dithiane
3. Thiodiglycol

OTH-1693-1c

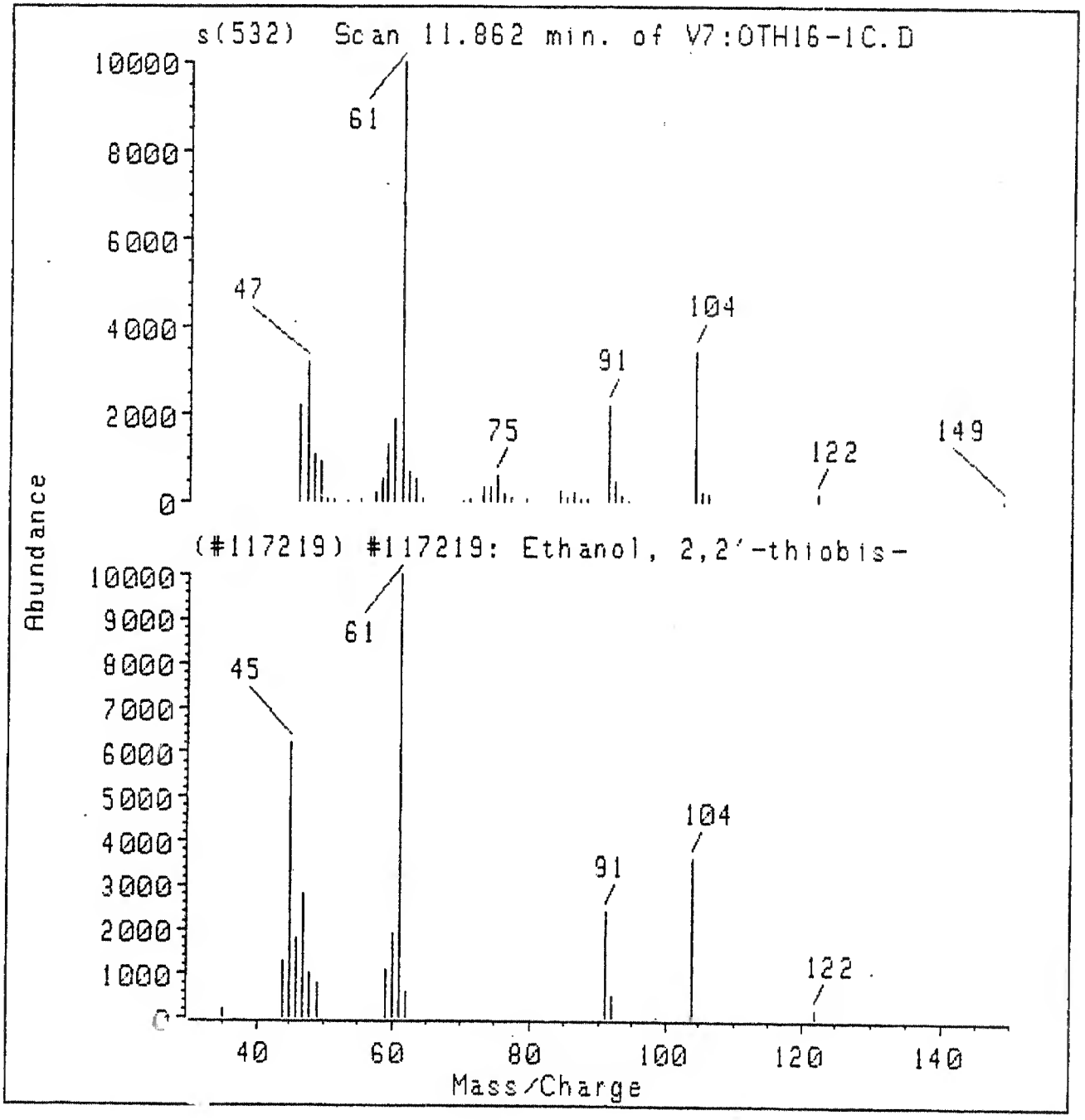

s(131) Scan 5.301 min. of V7:OTH16-1C.D
10000
8000
6000
4000
2000
0
46
84
61
104
74
119
(#2487) #2487: 1,4-Oxathiane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
46
27
61
104
74
Abundance
40
60
80
100
120
Mass/Charge

OTH-1693-1c


s(330) Scan 8.548 min. of V7:OTH16-1C.D
10000
8000
6000
4000
2000
0
46
61
73
92
105
120
(#4972) #4972: 1,4-Dithiane
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
35
46
61
73
92
120
Abundance
40
50
60
70
80
90
100
110
120
Mass/Charge

Thiodiglycol

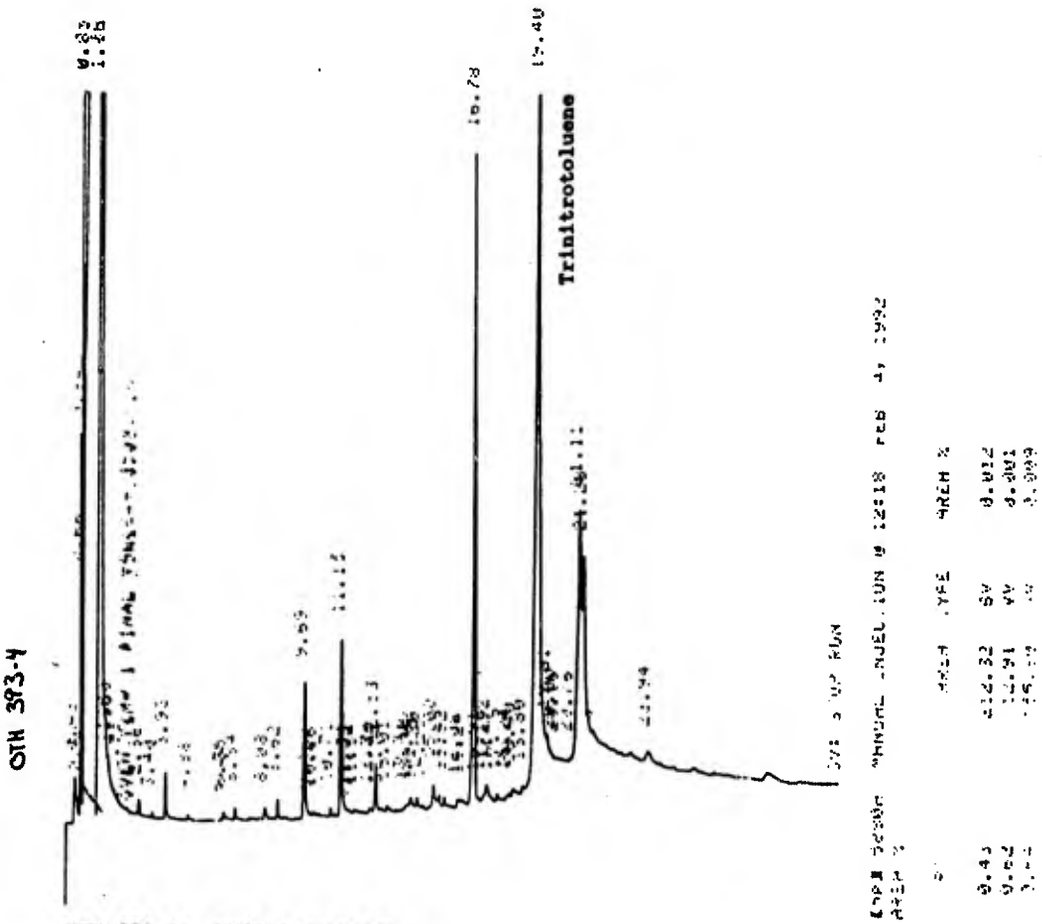
OTH-1693-1c



Blank

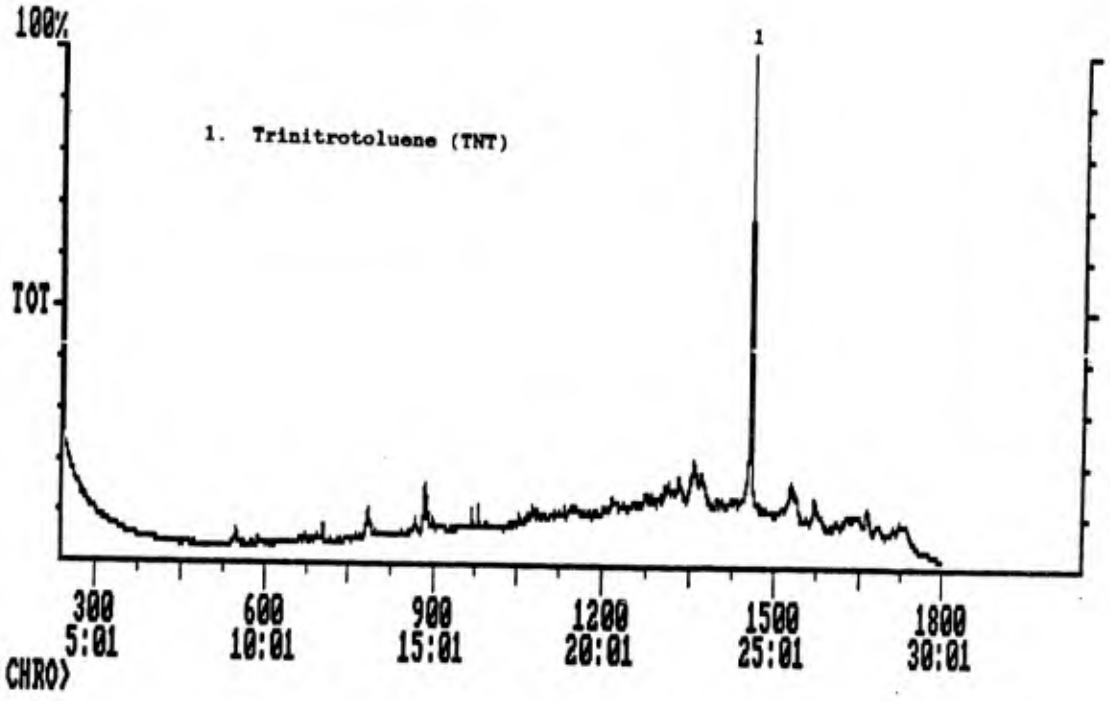
APPENDIX F

GC/FID AND GC/ITD SPECTRA

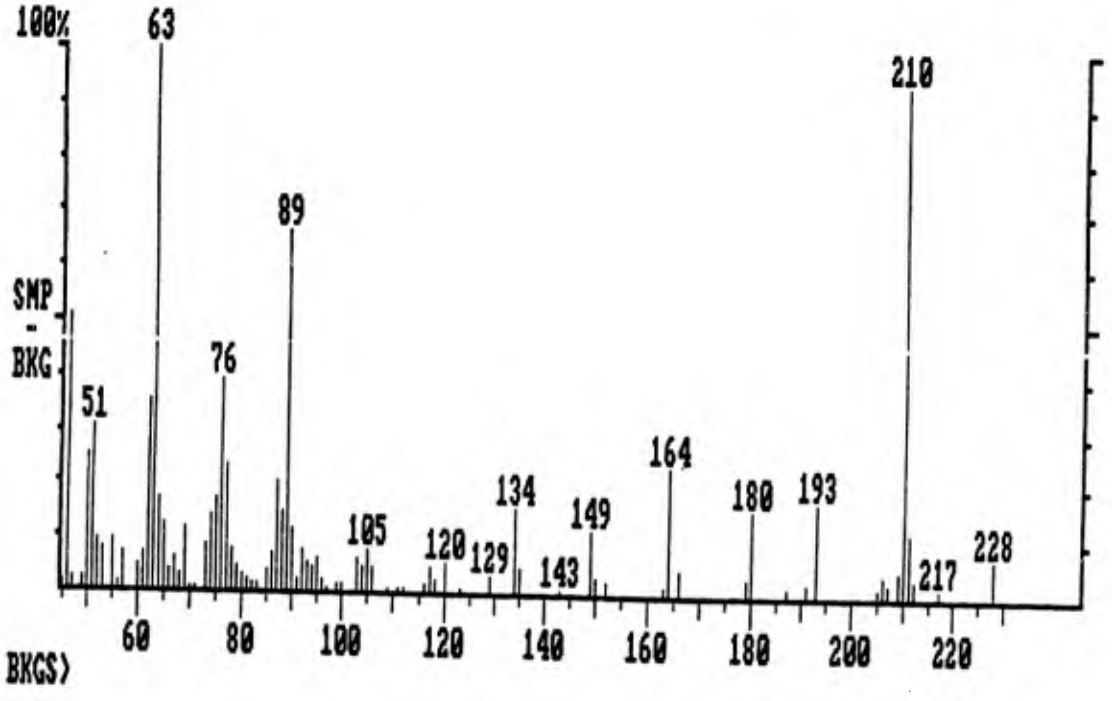


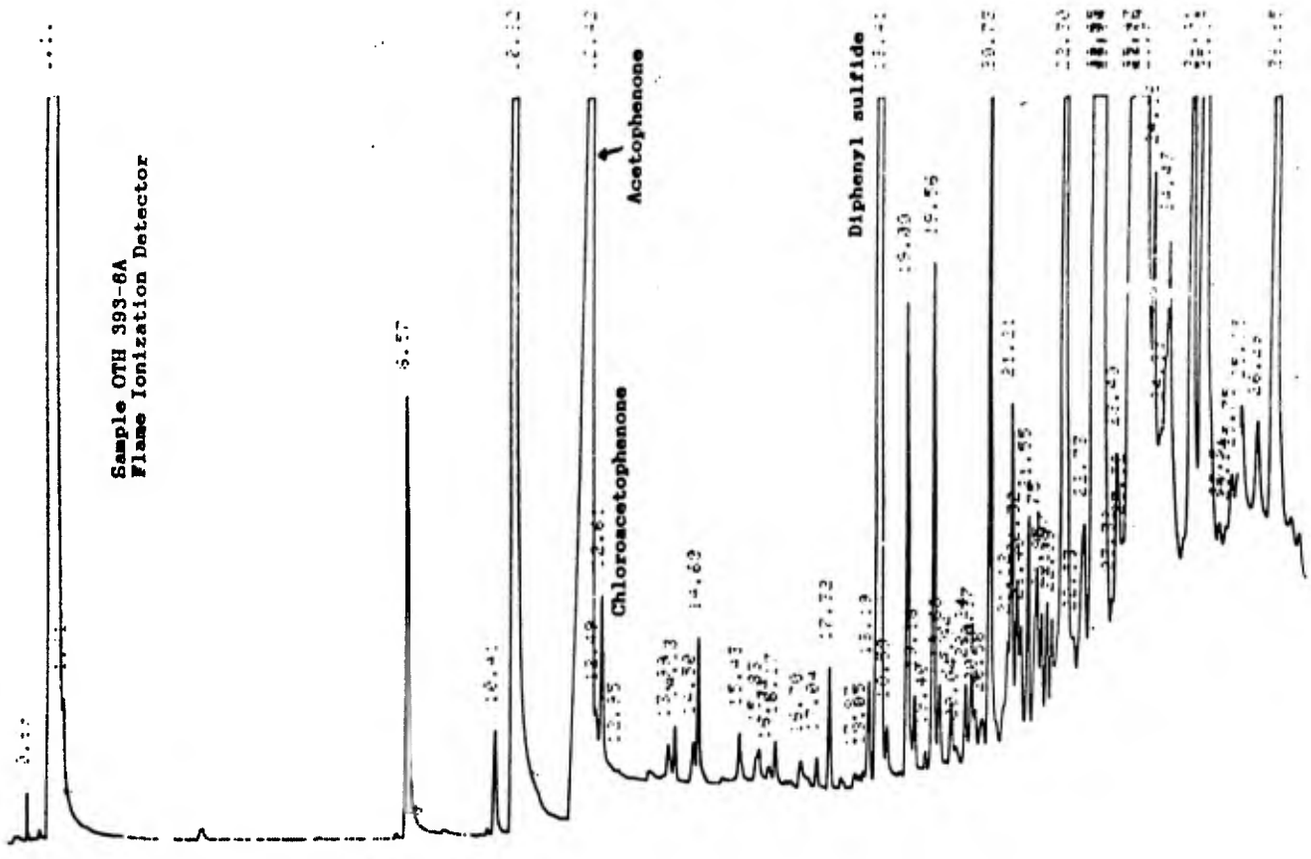
OTH-393-4: GC/FID spectrum.

Sample 393-4: GC/ITD spectrum.



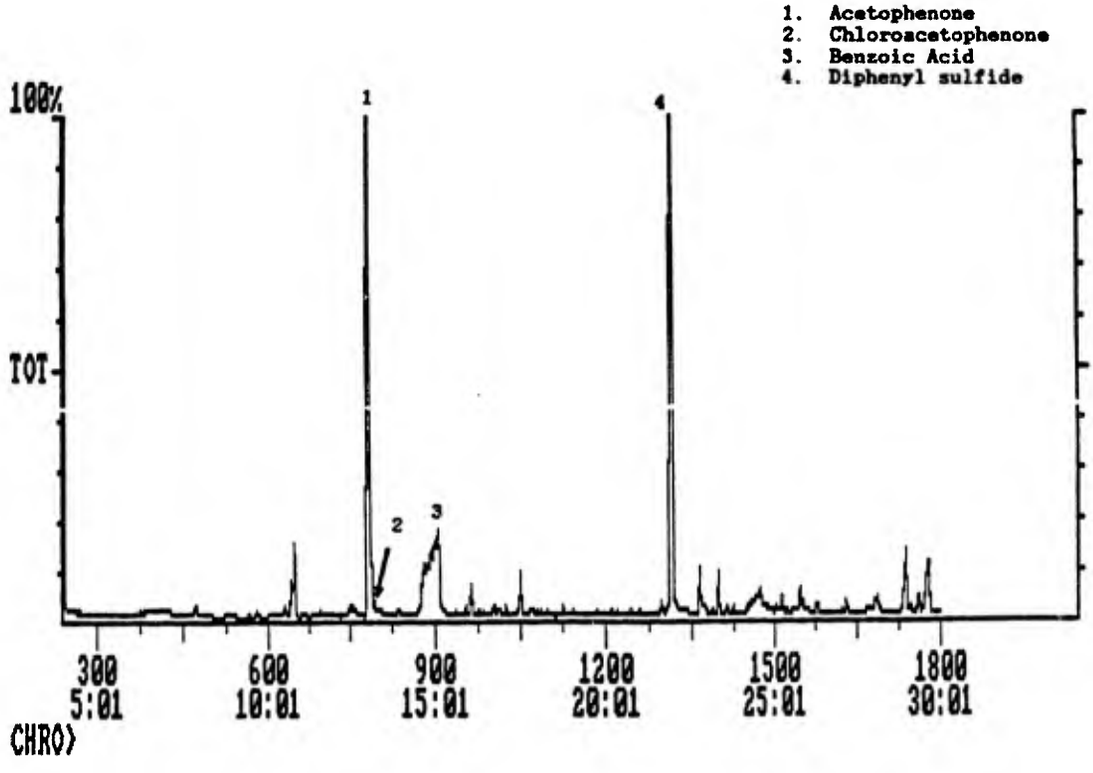
Sample 393-4
Trinitrotoluene (TNT) (1)



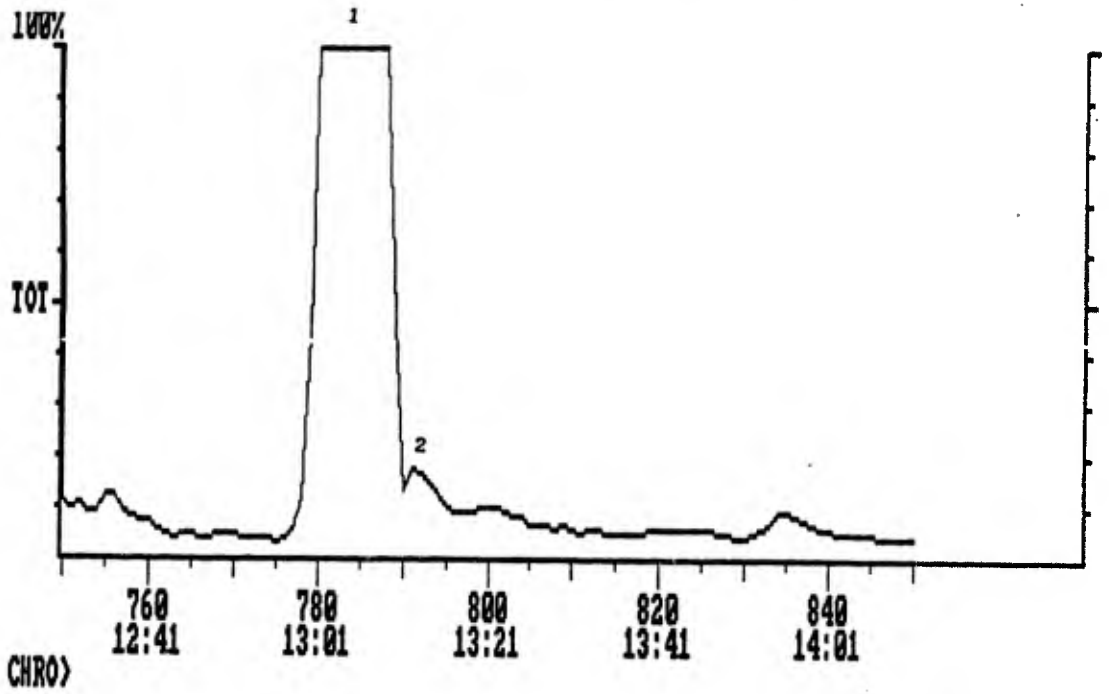


OTH-393-6a: GC/FID spectrum.

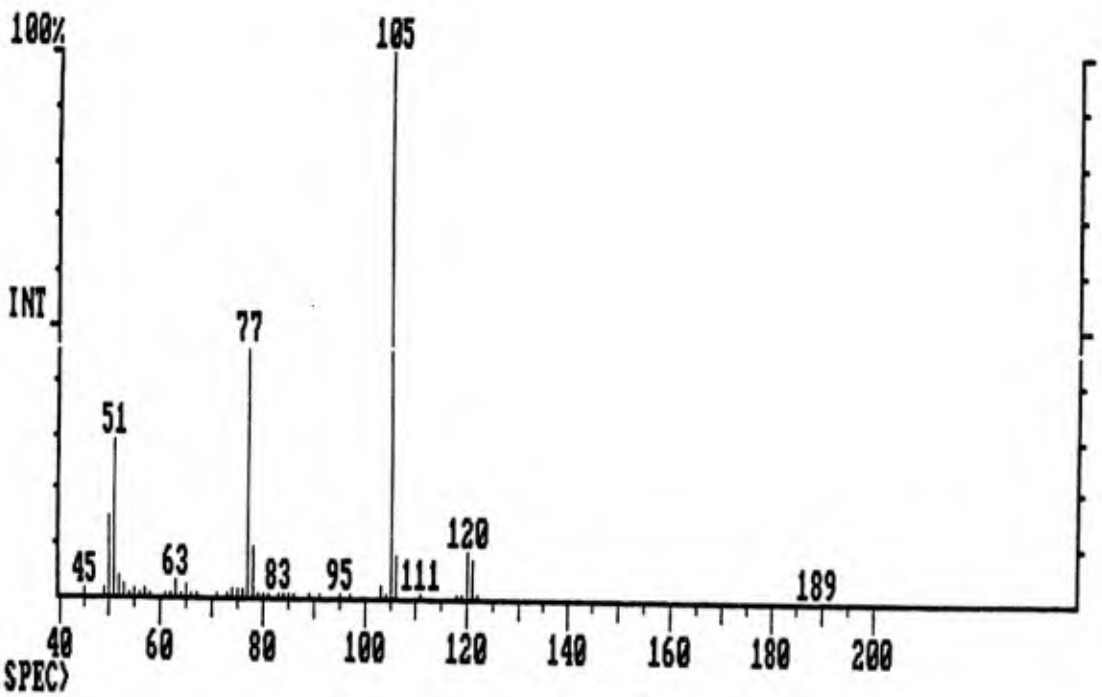
Sample 393-6A : GC/ITD spectrum.



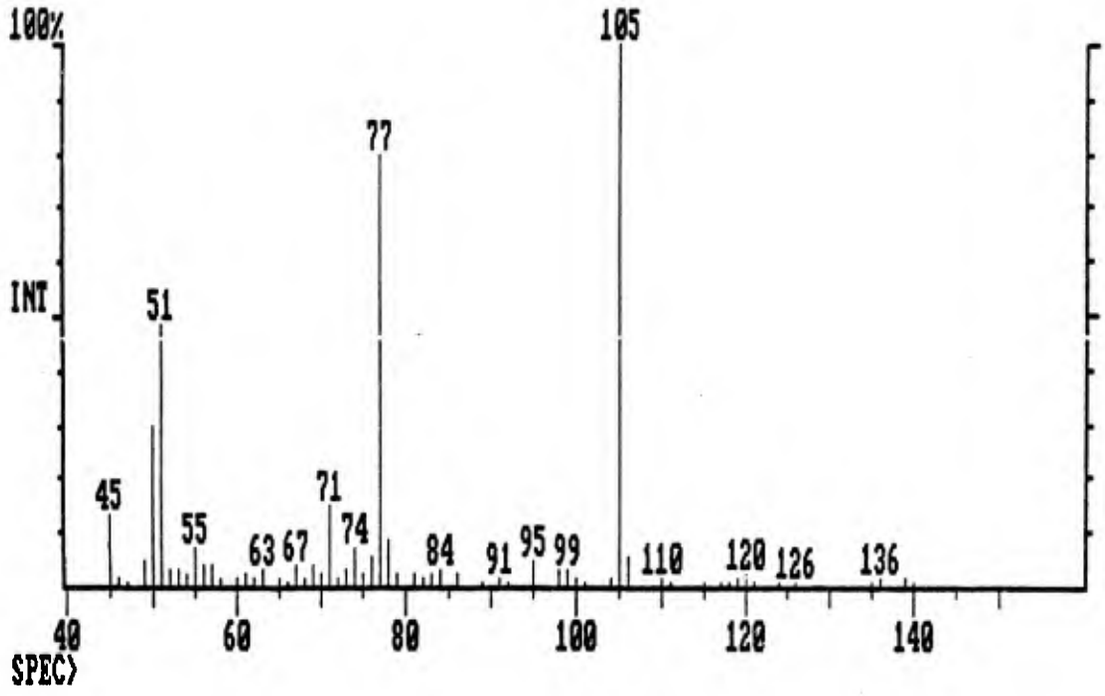
Sample 393-6A
1. Acetophenone
2. Chloroacetophenone



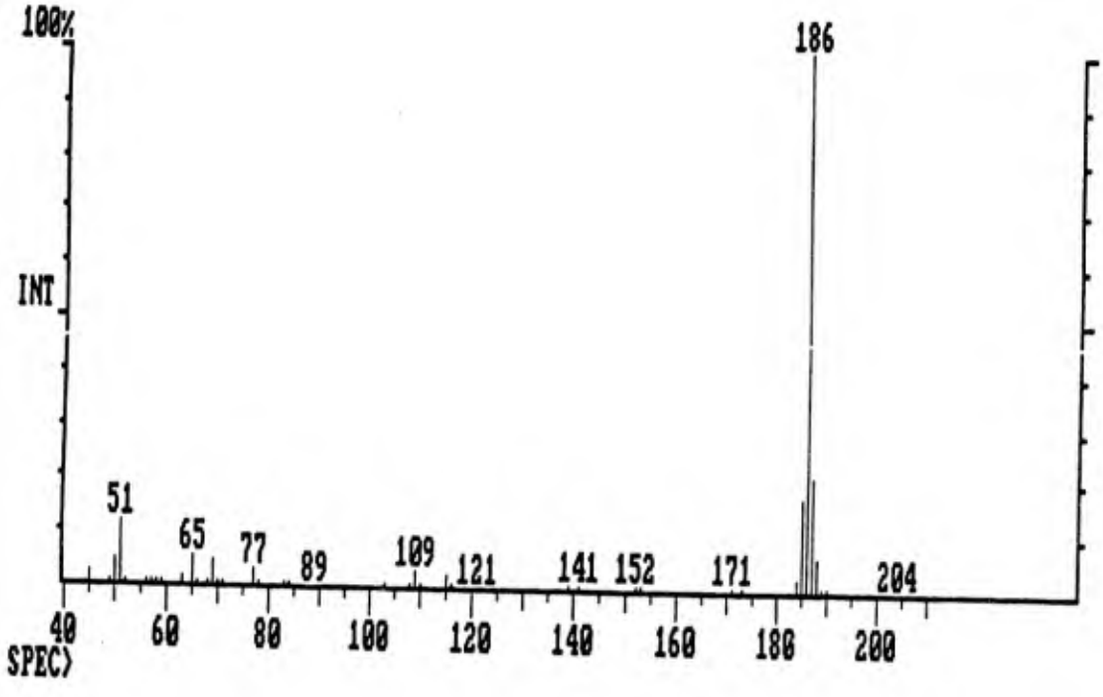
Sample 393-6A
Acetophenone (1)

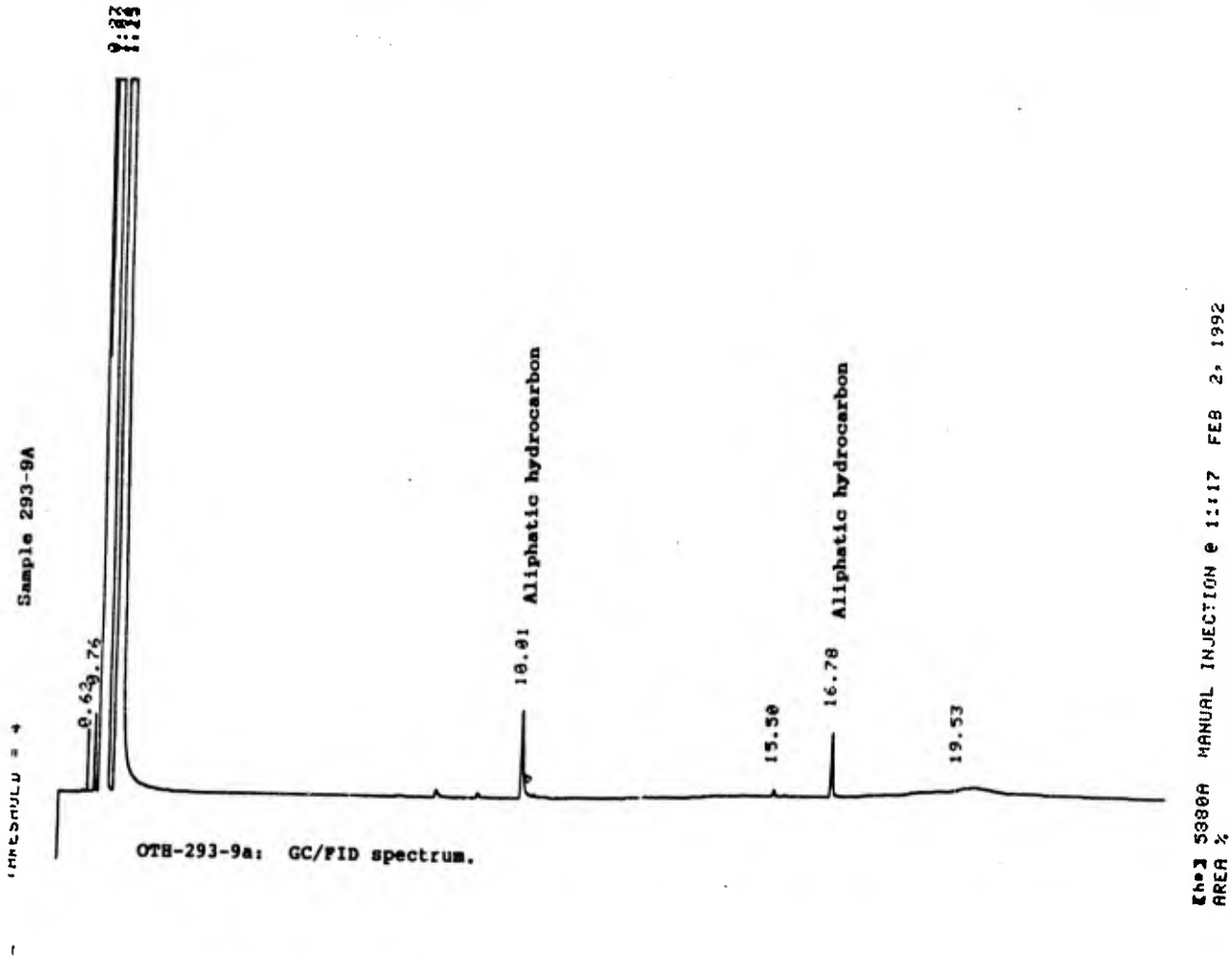


Sample 393-6A
Chloroacetophenone (2)



Sample 393-6A
Diphenyl sulfide (4)

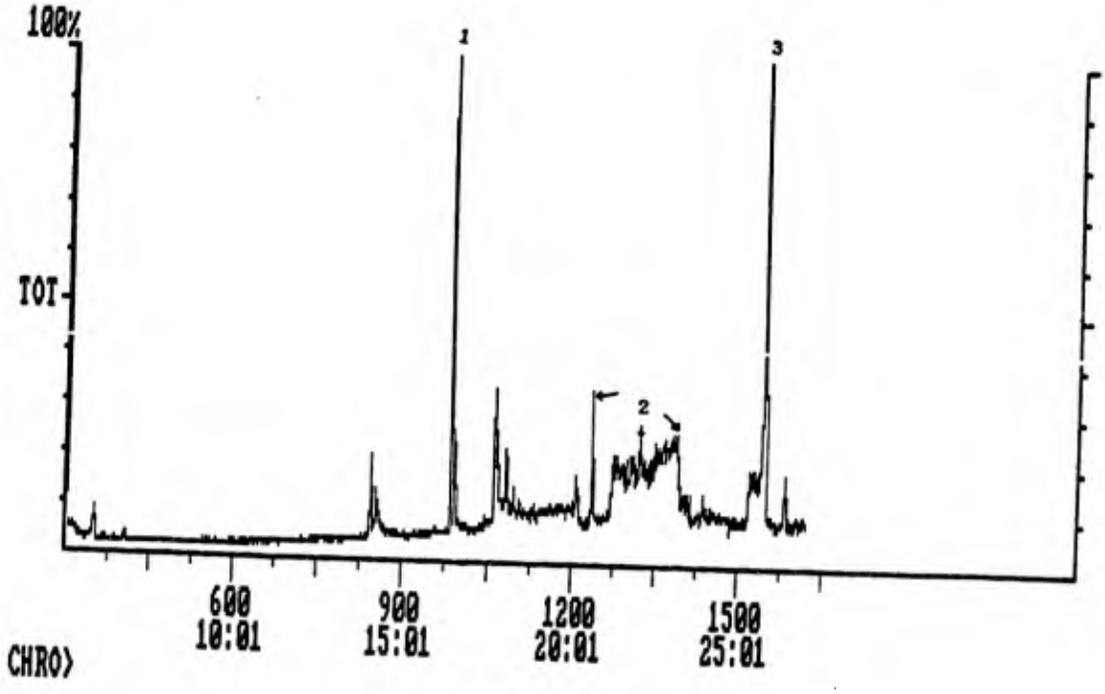




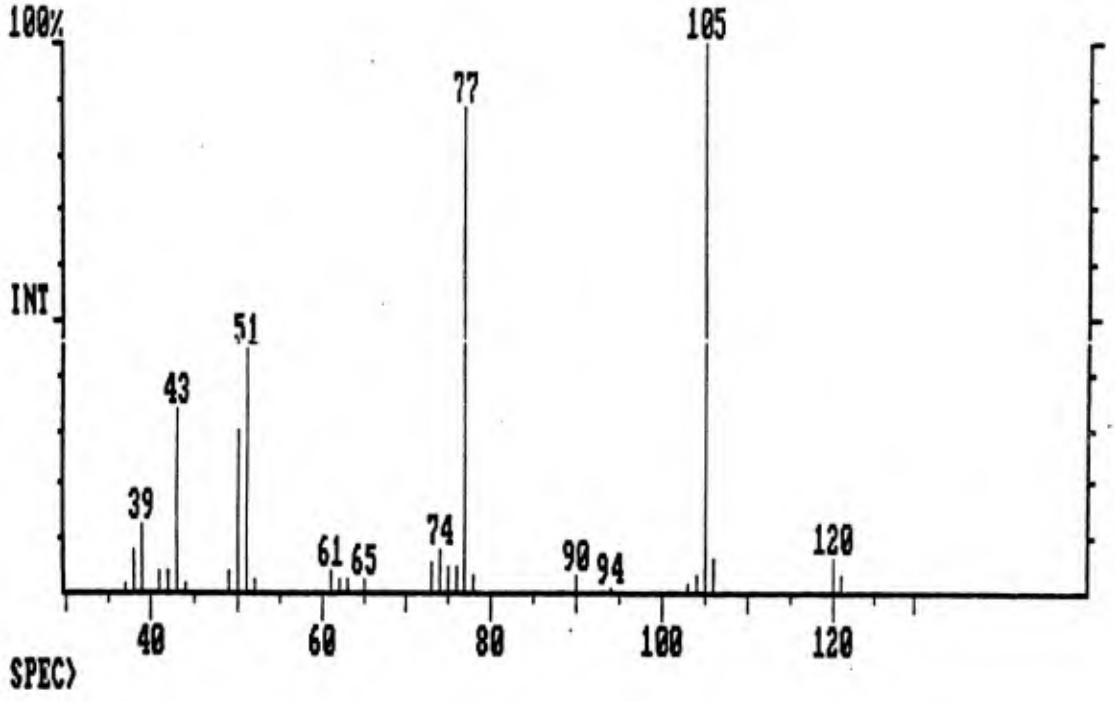
OTH-293-9a: GC/FID spectrum.

Sample 1393-1b: GC/ITD spectrum.

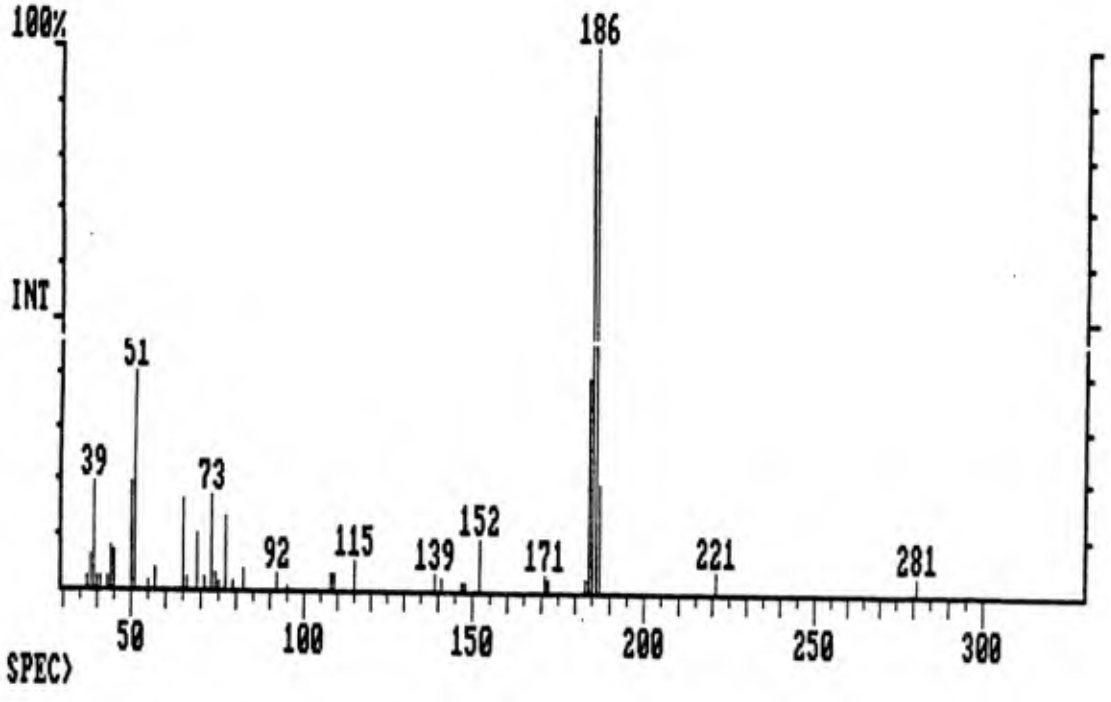
1. Acetophenone
2. Hydrolysis of Chloroacetophenone
3. Diphenyl sulfide



Sample 1393-1b
Acetophenone (1)



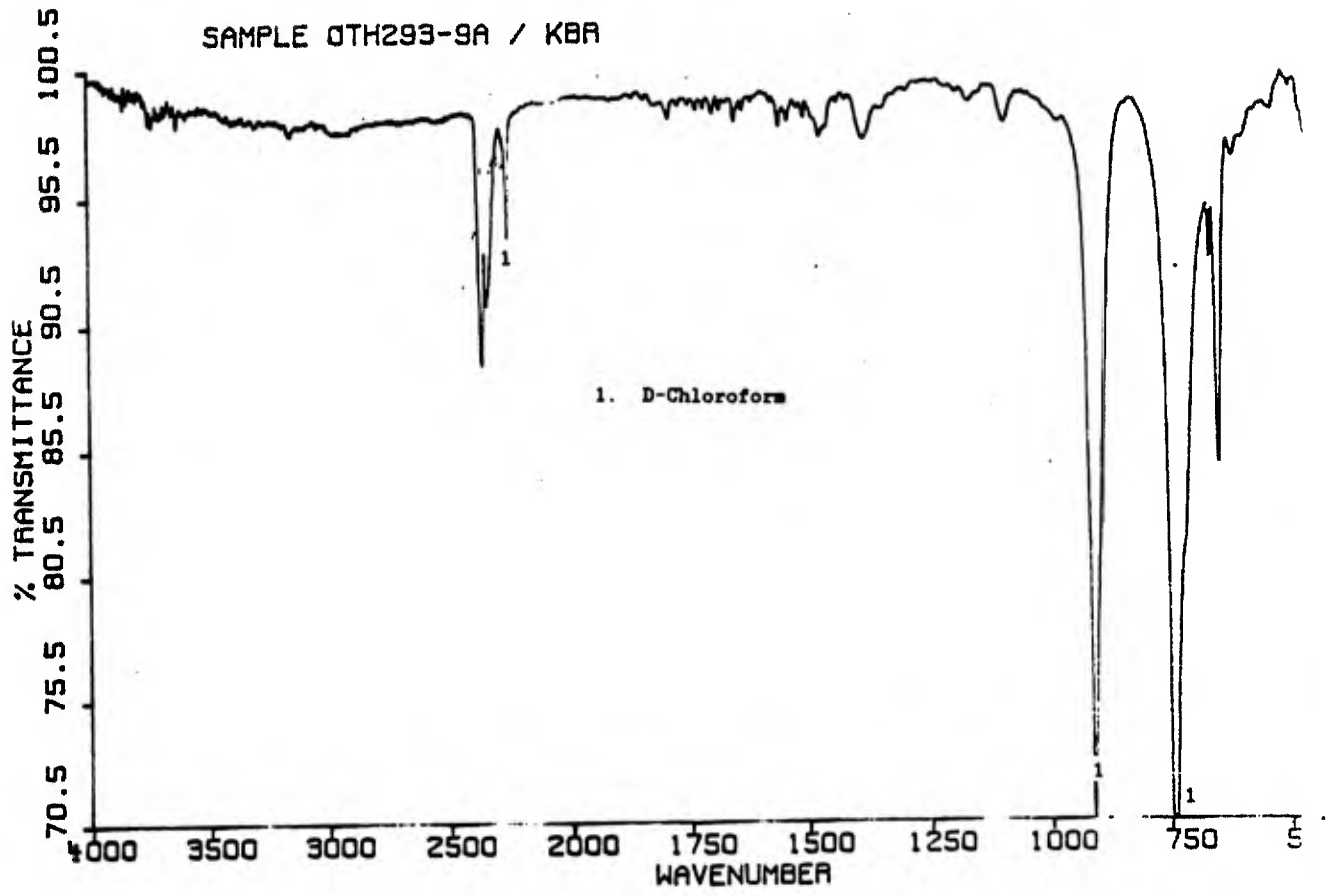
Sample 1393-1b
Diphenyl sulfide (3)

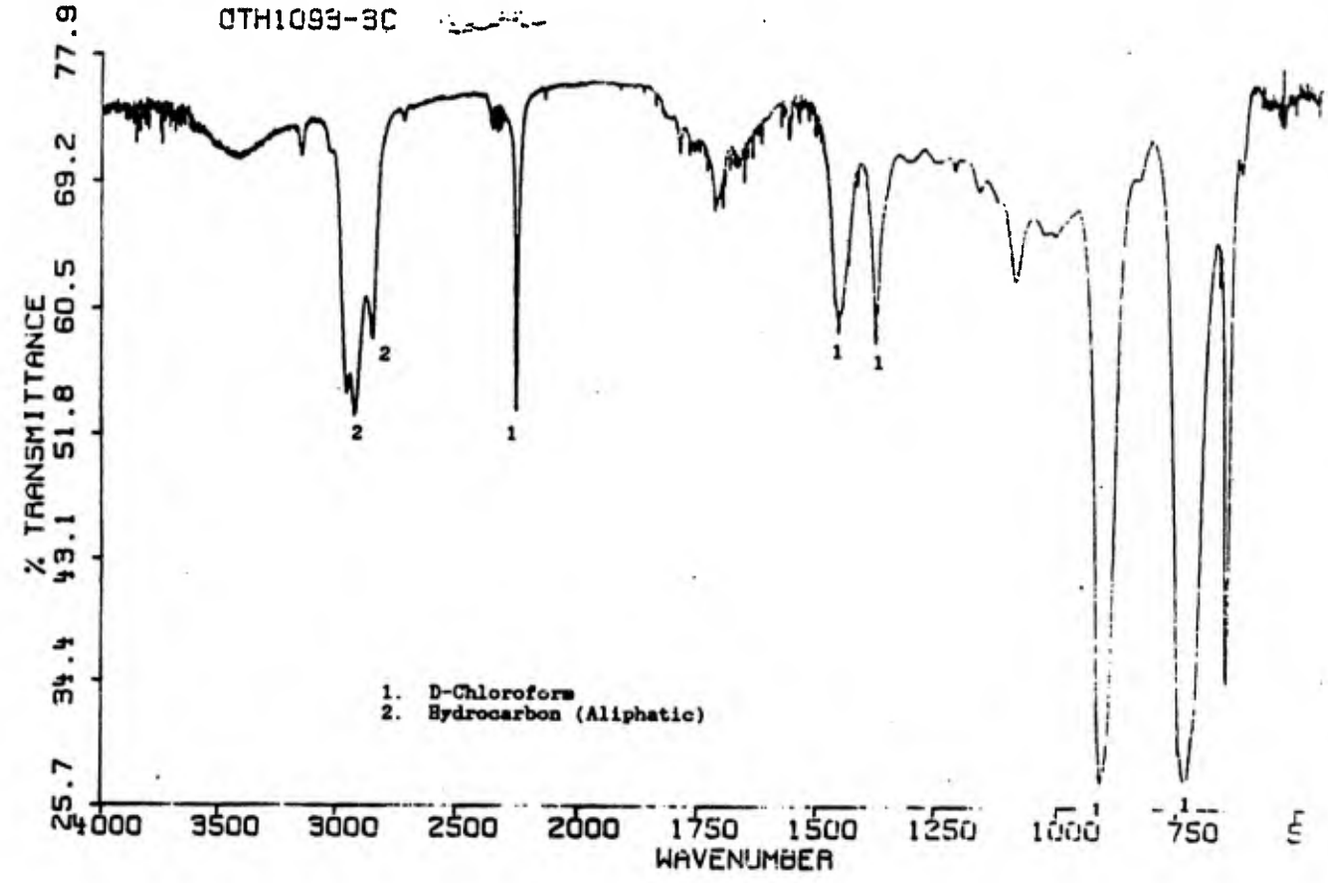


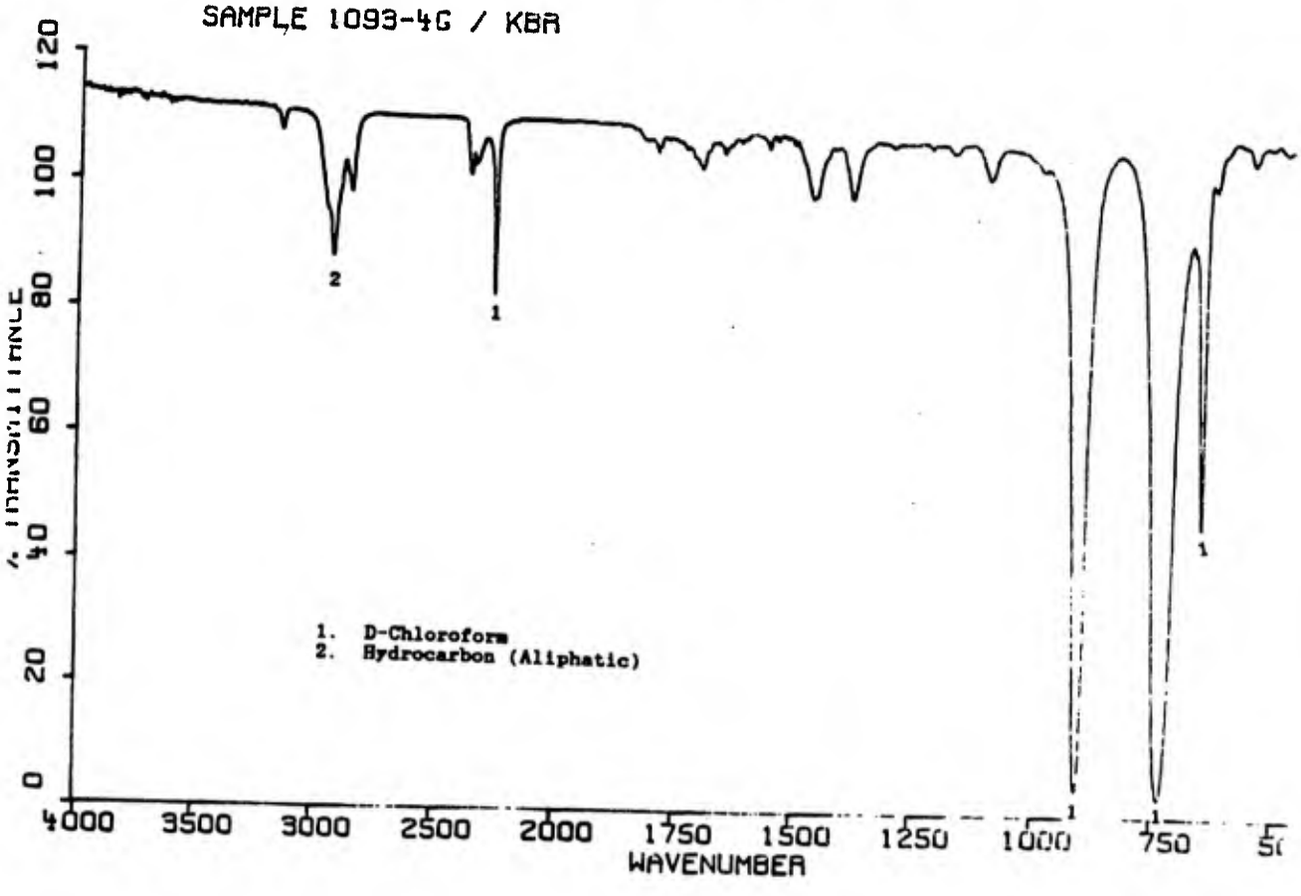
Blank

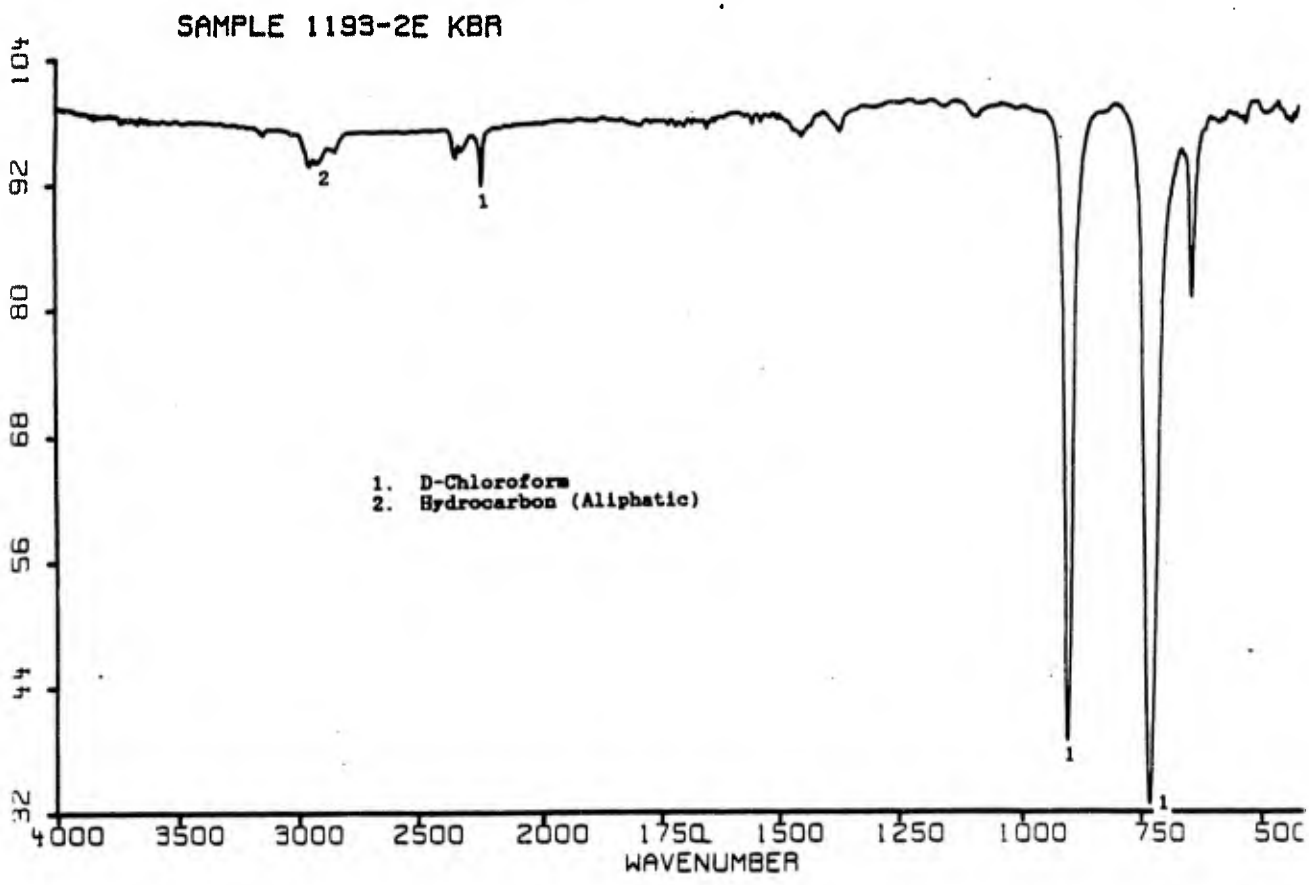
APPENDIX G

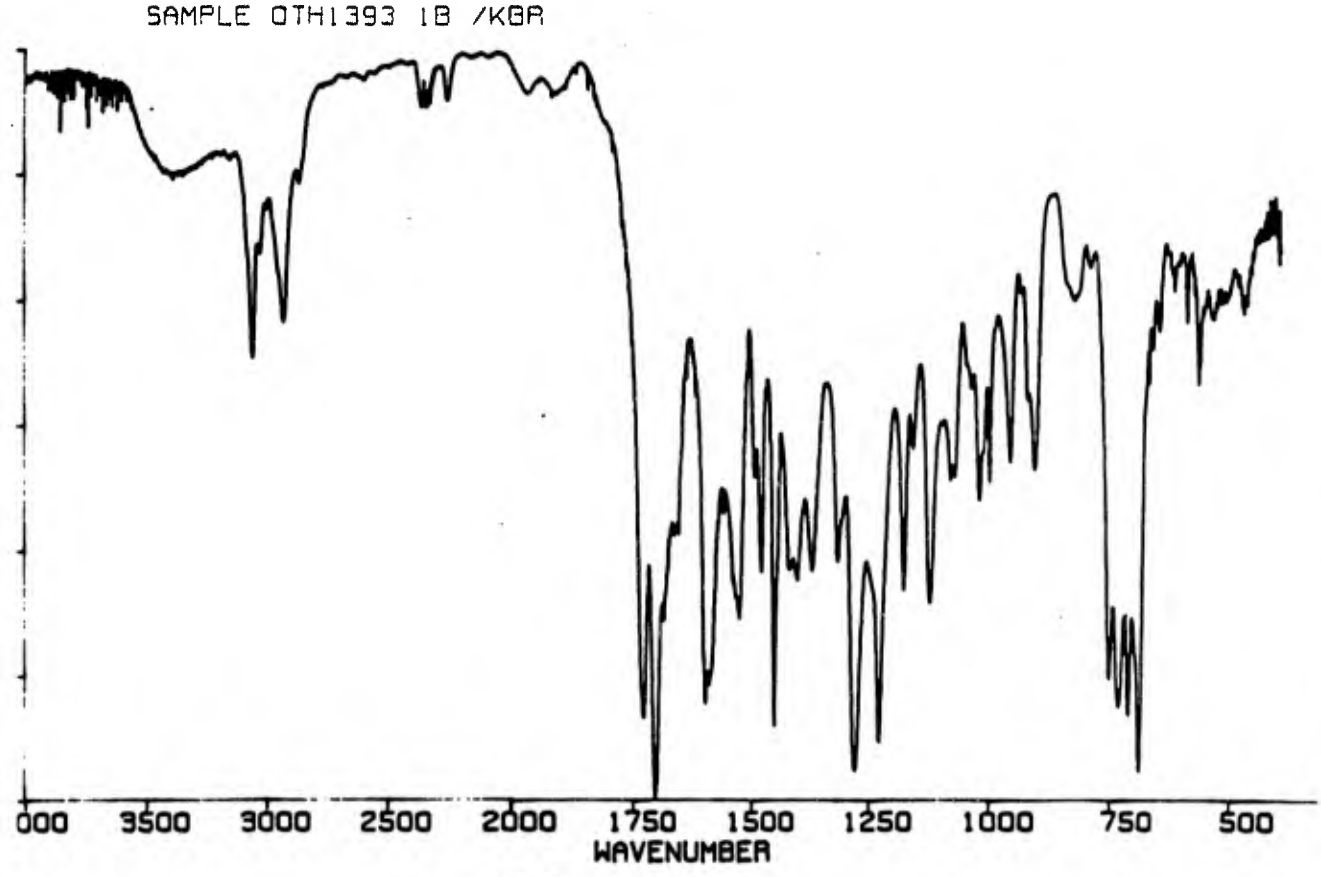
FTIR SPECTRA




OTH1093-3C
% TRANSMITTANCE
77.9
69.2
60.5
51.8
43.1
34.4
25.7
4000
3500
3000
2500
2000
1750
1500
1250
1000
750
WAVENUMBER
1. D-Chloroform
2. Hydrocarbon (Aliphatic)


SAMPLE 1093-4G / KBR
120
100
80
60
40
20
0
4000
3500
3000
2500
2000
1750
1500
1250
1000
750
WAVENUMBER
1. D-Chloroform
2. Hydrocarbon (Aliphatic)

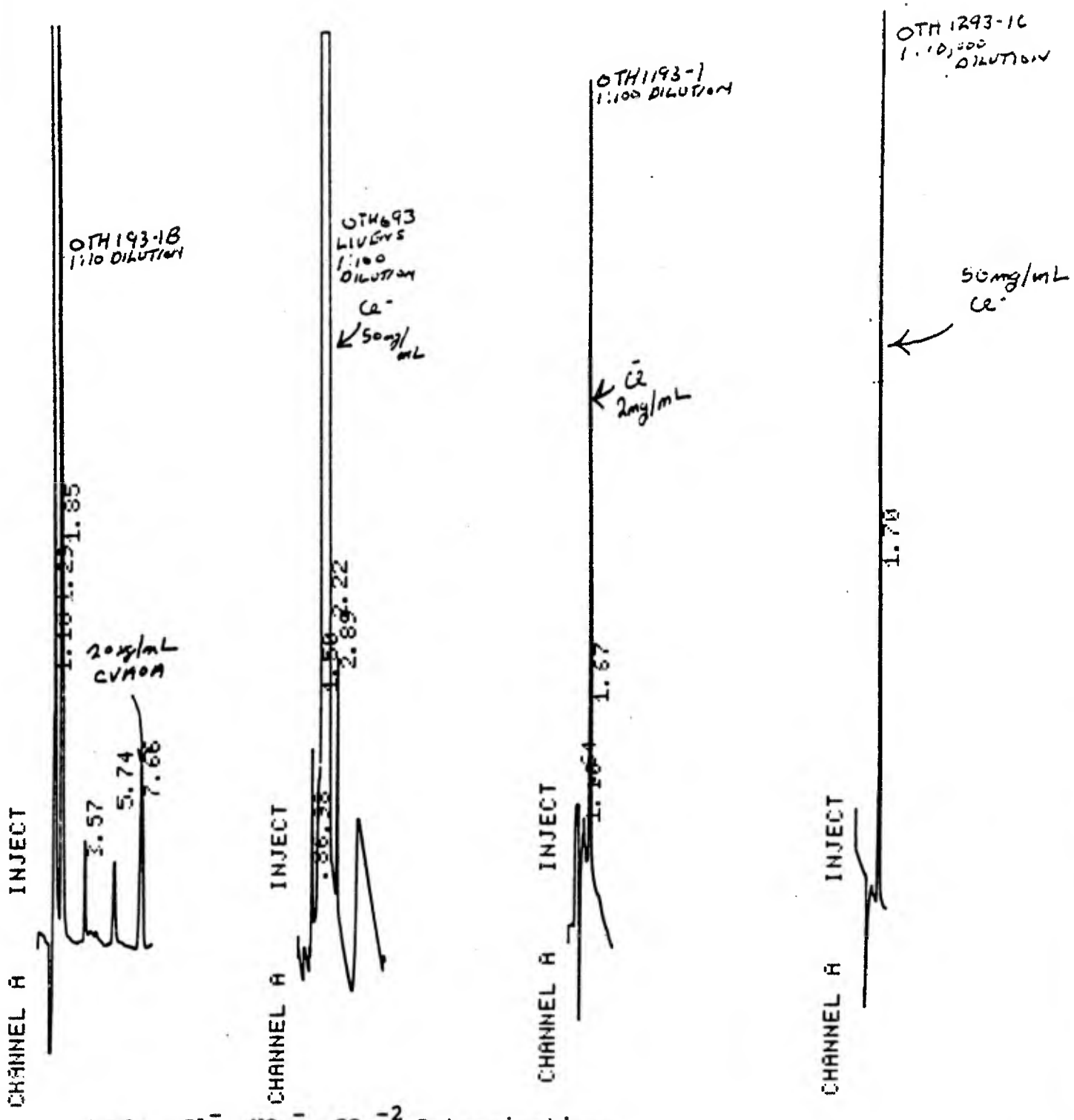

SAMPLE 1193-2E KBR
104
92
80
68
56
44
32
1. D-Chloroform
2. Hydrocarbon (Aliphatic)
4000
3500
3000
2500
2000
1750
1500
1250
1000
750
500
WAVENUMBER


SAMPLE OTH1393 1B /KBR
000
3500
3000
2500
2000
1750
1500
1250
1000
750
500
WAVENUMBER

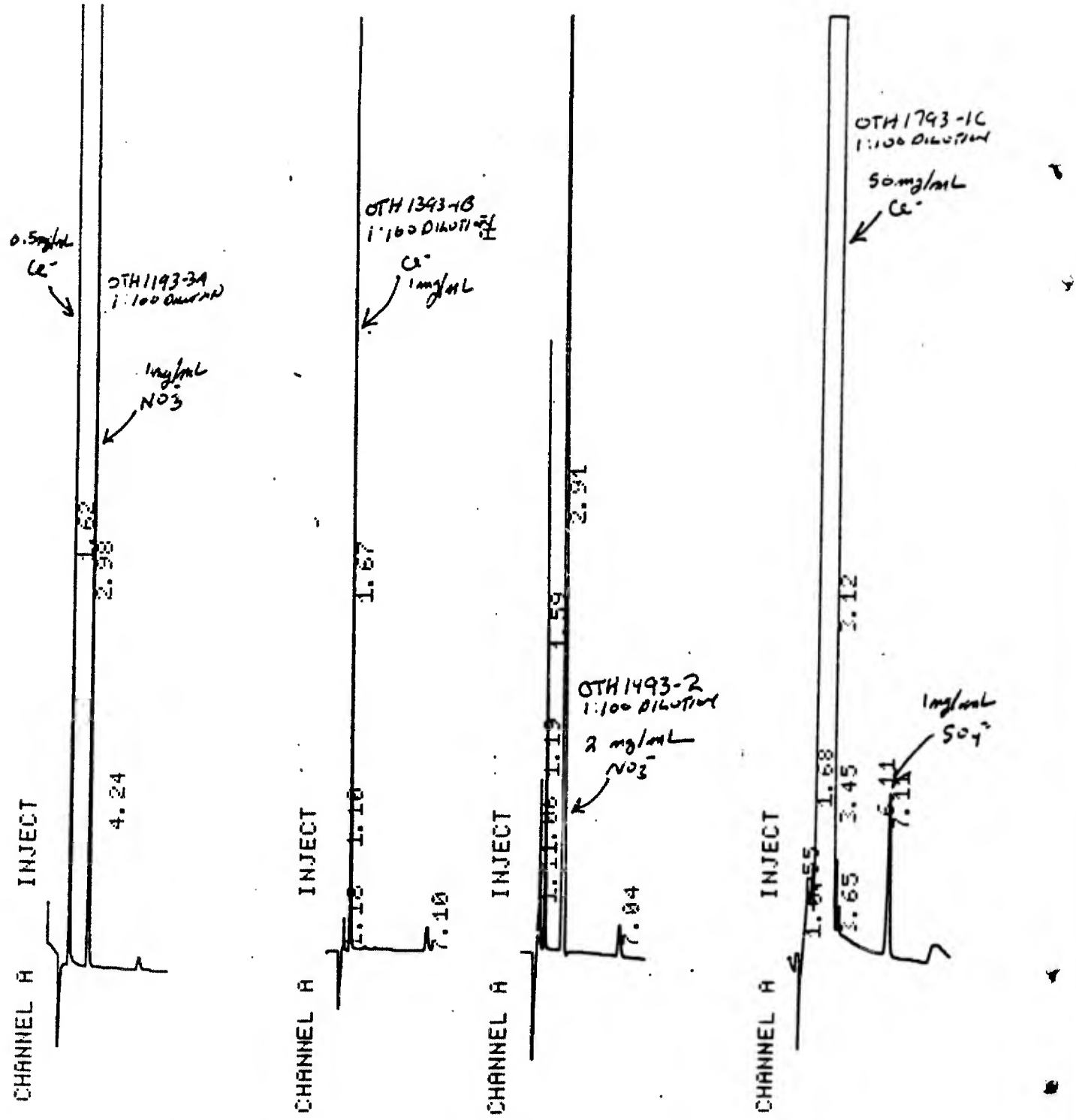
Blank

APPENDIX H

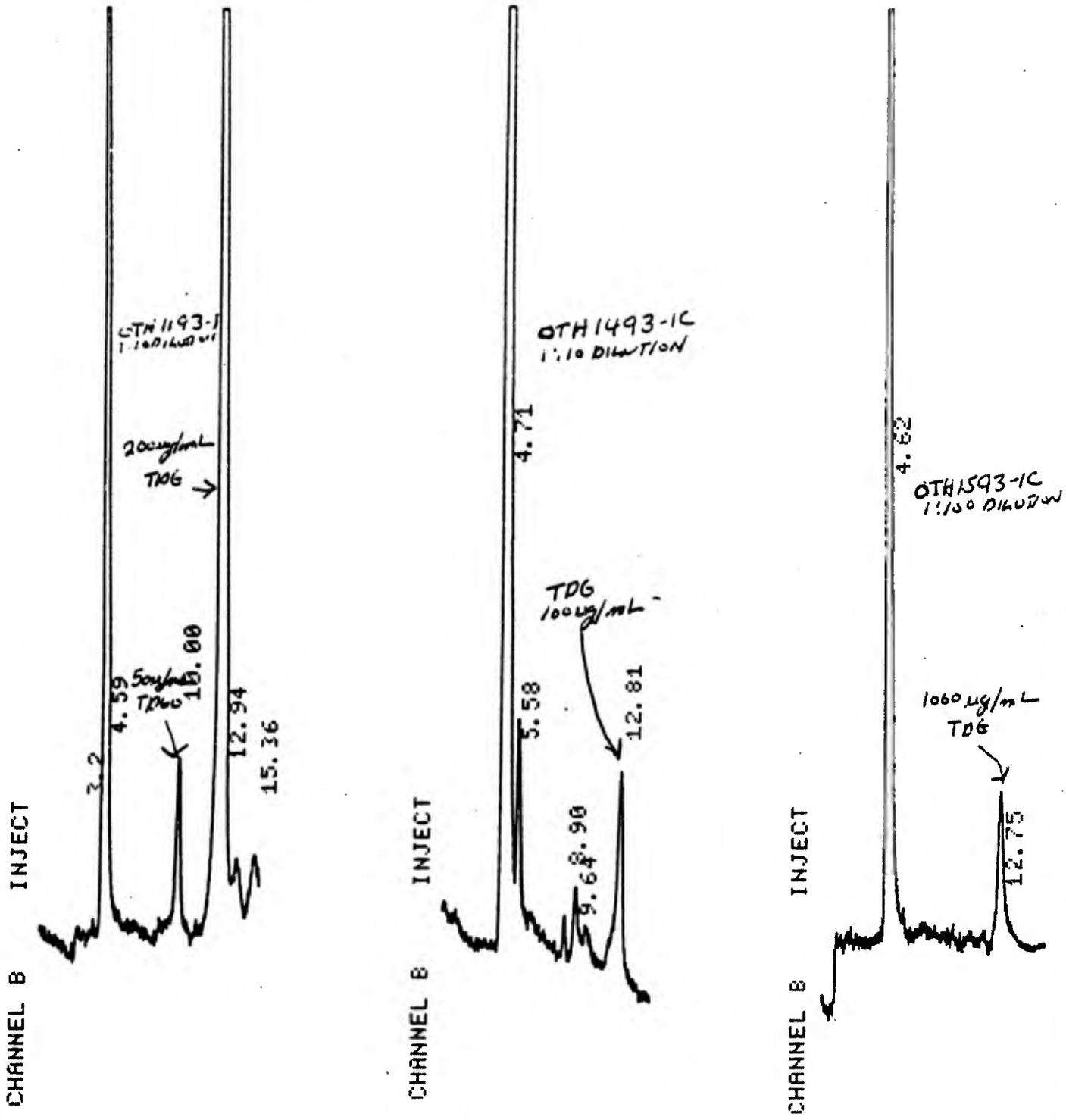
HPLC/IC SPECTRA



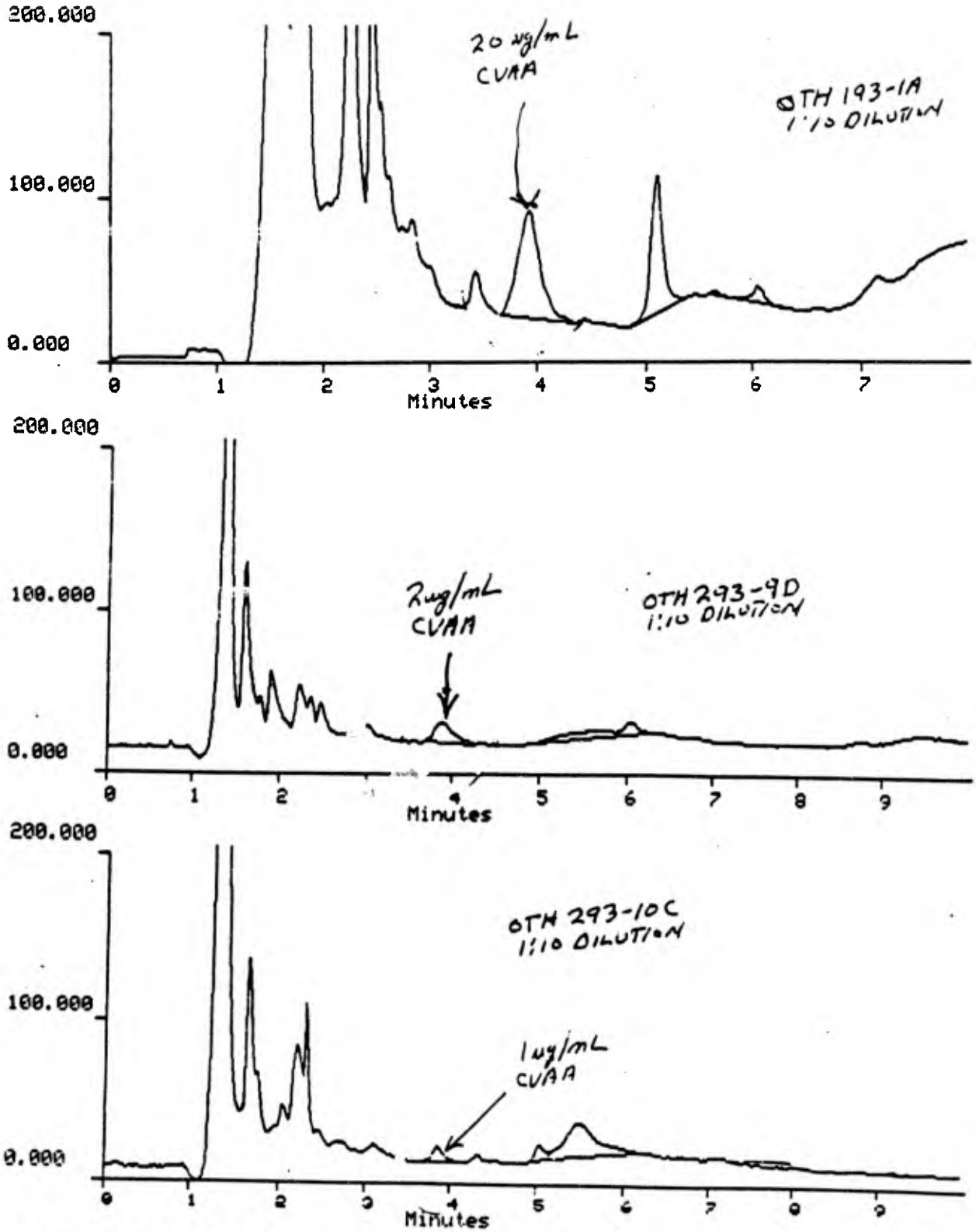
CVAOA, Cl^-, NO_3^-, SO_4^{-2} Determinations.



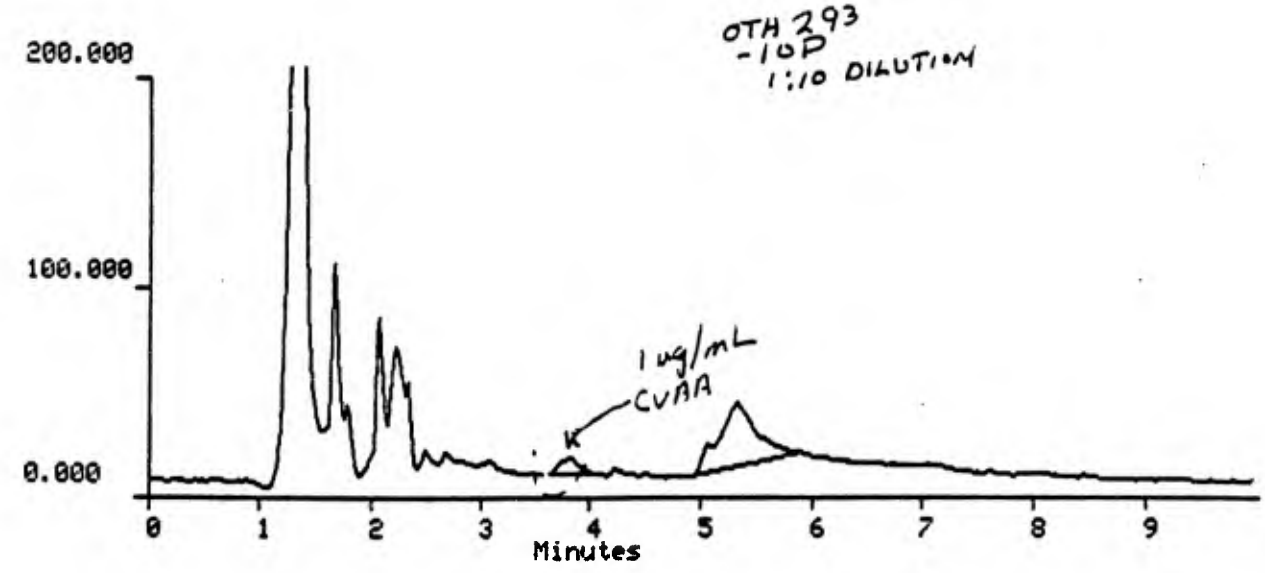
CVAOA, Cl^-, NO_3^-, SO_4^{-2} Determinations.

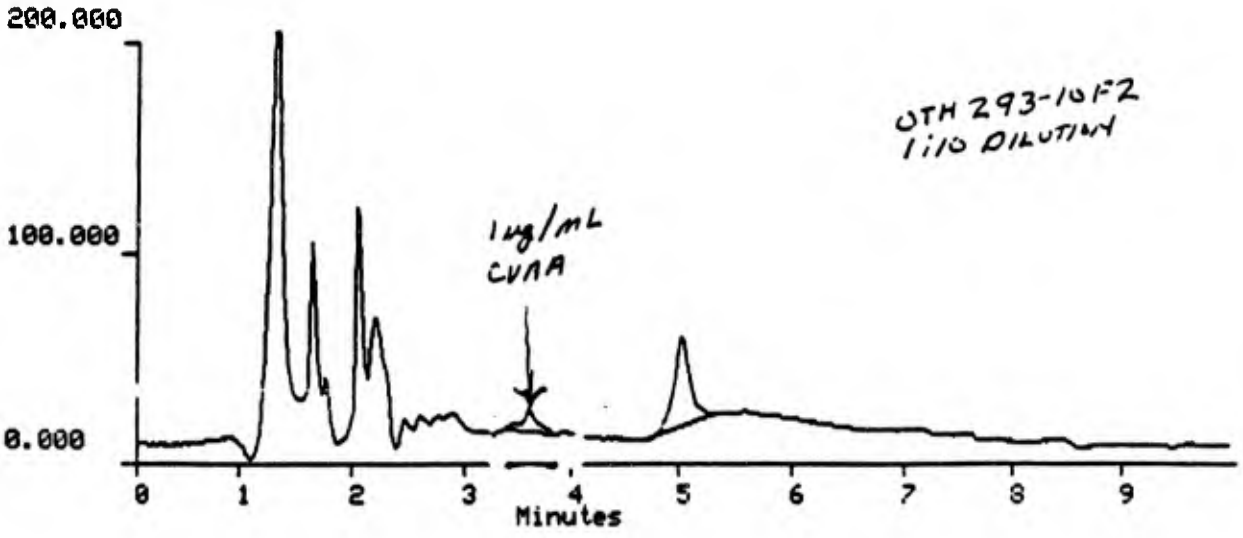


TDG, TDGO, TDGO2, AsO_2^-, AsO_4^{-2} Determinations.

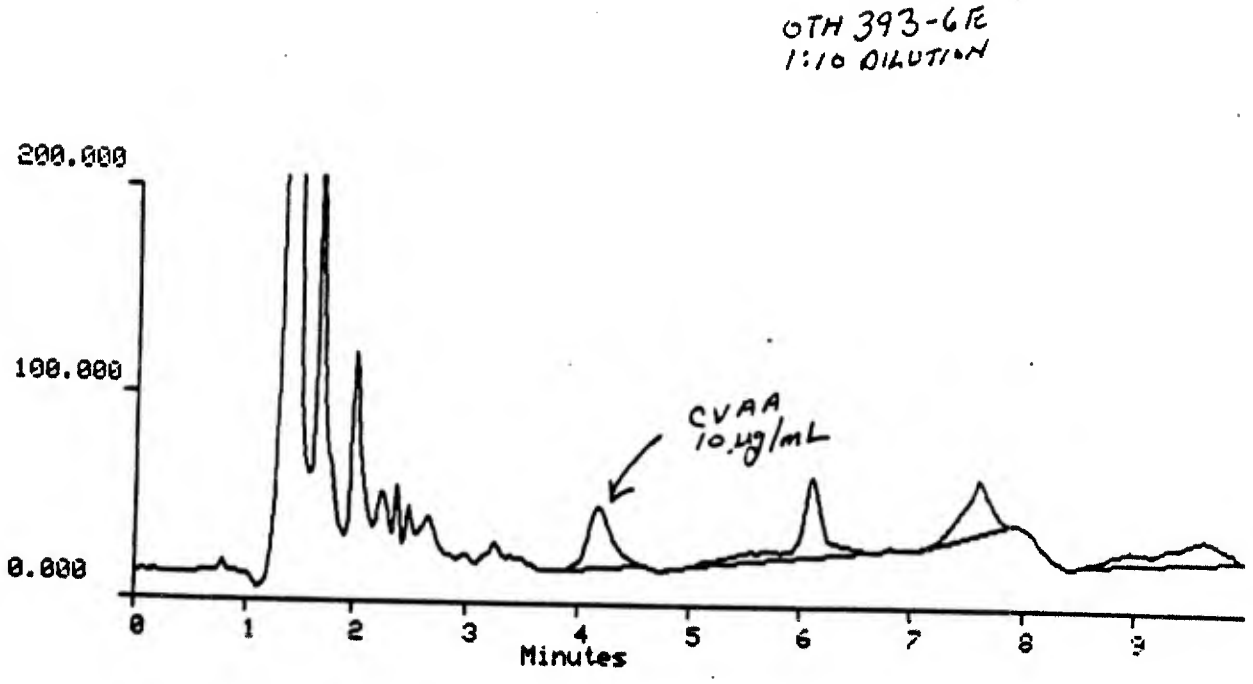


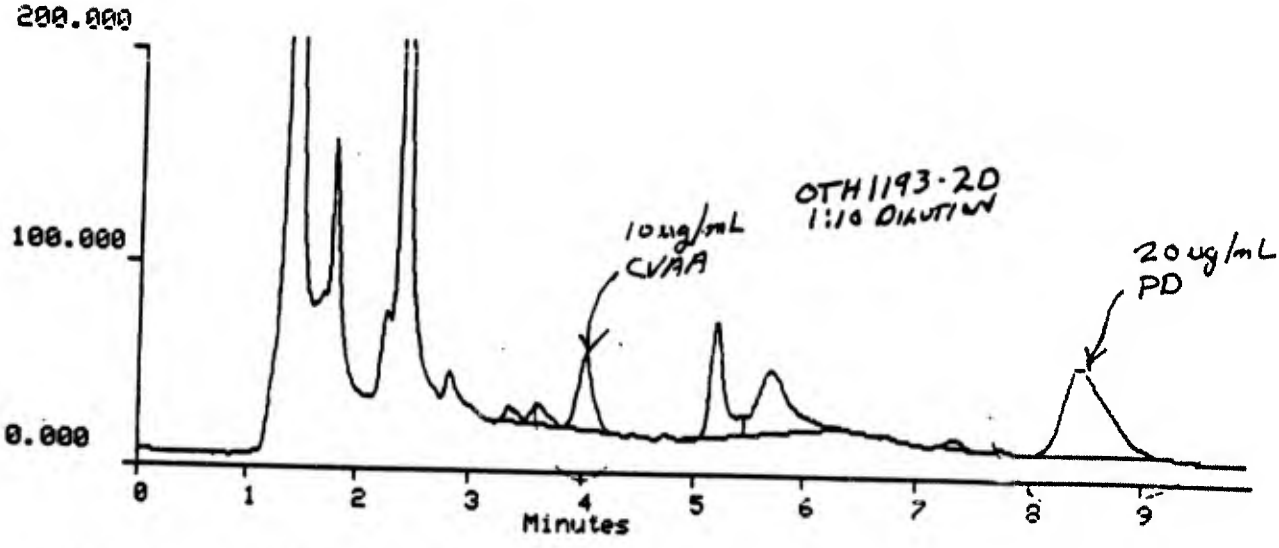
CVAA Determinations.





CVAA Determinations.





CVAA and PD Determinations.

APPENDIX I

DD FORM 1911

CHAIN OF CUSTODY

SAMPLES OTH-193

MATERIEL COURIER RECEIPT

SHIPPER	TEU
DESTINATION	APG - EA, MD
SHIPPER'S CONTROL/DOCUMENT NO.	TEU 93-3009
SUPPLY ACCOUNT NUMBER	N/A
SUPPLY ACCOUNT NUMBER	N/A

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

Transfer	Location of Transfer	Date (YR/MO/DAY)	Recipient's Printed Name (Last, First, M.I.)	Organ. or Account No.	Signature	Social Security Number
FIRST	Dietrick	1/9/93	[illegible]	USA MRIID	Parker, Clemon W.	460-86-2280
SECOND	Ft Dietrick MRIID	1/9/93	Brooks, Mark A.	USA Tech Escort Unit	[signature]	216-56-9147
THIRD	CRDA Bldg [illegible]	1/1/93	Lochner, Joseph M.	SCBRD-RTS	[signature]	212488611
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

Line Number	Quantity	Serial Numbers	Remarks
	1	Box #1	Samples (Spring Valley D.C.)
	1	Box #2	Sample (Spring Valley D.C.)
	1	Box #3	Sample (Spring Valley D.C.)
93-002	1	Box #5	Sample (Spring Valley D.C.)

DD FORM 1911 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-293 (II)

MATERIEL COURIER RECEIPT

SHIPPER	USATEU
DESTINATION	USAMRIID
SHIPPER'S CONTROL/DOCUMENT NO.	#93-003
SUPPLY ACCOUNT NUMBER	
SUPPLY ACCOUNT NUMBER	

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

Transfer	Location of Transfer	Date (YR/MO/DAY)	Recipient's Printed Name (Last, First, M.I.)	Organ. or Account No.	Signature	Social Security Number
FIRST	USAMRIID	93/01/12	[illegible]		[signature]	575-68-0550
SECOND	USAMRIID	93/01/12	Brower, James C	USATEU	[signature]	097-40-9134
THIRD	3330 EA Raymond E Herd 8-45-19	93/1/12	Raymond E Herd	MA	[signature]	304-36-6613
FOURTH	Bldg 3330	93/1/13	Brower, James C	TEU	[signature]	097409134
FIFTH	E 3300	93/01/13	Lochner, Joseph M	SCBRD-RTS	[signature]	212488611

SHIPMENT DESCRIPTION

Line Number	Quantity	Serial Numbers	Remarks
1	2	#9, #10	PIG's w/glassware and solid samples
1 — Nothing Follows			

DD FORM 1911 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-393

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: #93-002

SHIPPER: USATEU

SUPPLY ACCOUNT NUMBER:

DESTINATION: Research Branch, ERDEC

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	Spring Valley, Washington D.C.	93/01/14	D M Rebellion	TEU/TDET	D M Rebellion	SIX-70-4507
SECOND	USAMRIID, Ft. Detrick, MD.	93/01/14	EMTEN, EDWARD M.		Edward M.	423-78-5211
THIRD	USAMRIID, Ft Detrick, MD.	93/01/14	D M Rebellion	TEU/TDET	D M Rebellion	SIX-70-4507
FOURTH	ERDEC, APG, MD	93/01/14	Marguerite Brooks		Marguerite Brooks	115-14-7254
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1	2	#4, #6	Pipe w/ Glassware & Various Solids
1		Nothing Follows	
1	2	#4, #6	Received and transferred back to TEU.
1	2	#4, #6	T. Jenkins - Glassware - Solid

DD FORM 16 MAY 1911 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-493

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBRD-ODX-0-3018

SHIPPER: T.A. BLADES E3832

SUPPLY ACCOUNT NUMBER: 1476

DESTINATION: 3300

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	ERDEC 330	93-1-14	[illegible]		[illegible]	115-14-7254
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1	10 ml	ITEM# 71	LIQUID SAMPLE
2	2EA	TENAX TA 60/80	VAPOR SAMPLES
		NOTHING	FOLLOWS

DD FORM 16 MAY 1911 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-493

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBD-ODO-OTH-1477

SHIPPER: T A BLADES, E3952, X4207

SUPPLY ACCOUNT NUMBER:

DESTINATION: M BROOKS, E3300, X3957

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C. Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	EMEL E3300	93/1/16	Victoria L Brooks			115-16-7256
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	10ml	ITEM# 82 TENAX TA0305	LIQUID SAMPLE
2.	2EA	TENAX GR0206	VAPOR SAMPLES
		NOTHING FOLLOWS	

DD FORM 1911 MAY 86 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 88.

SAMPLES OTH-593

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBD-ODO-OTH-1478

SHIPPER: T A BLADES, E3952, X4207

SUPPLY ACCOUNT NUMBER:

DESTINATION: M BROOKS, E3300, X3957

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C. Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	EMEL E3300					
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	10ml	ITEM# 65 TENAX TA0305	LIQUID SAMPLE
2.	2EA	TENAX GR0206	VAPOR SAMPLE
		NOTHING FOLLOWS	

DD FORM 1911 MAY 86 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 88.

SAMPLES OTH-693

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCLLD-DC-M-3020-14

SHIPPER: T.A Blades, E-132, X4202

SUPPLY ACCOUNT NUMBER:

DESTINATION: M. Lochner, E3300, X3157

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSE(S): To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)
FIRST	E 3300	93/01/20
SECOND		
THIRD		
FOURTH		
FIFTH		

	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	LOCHNER, JOSEPH M	SCLLD-RTS		217-48-6611
SECOND				
THIRD				
FOURTH				
FIFTH				

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	4 lbs	ITEM = 65	Liquid Sample
		NOTHING FOLLOWS	

DD FORM 1911 (OCT 88 MAY) PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 93.

SAMPLES OTH-793

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: 3021-1

SHIPPER: G.L. Smith, EPDC, X4683

SUPPLY ACCOUNT NUMBER:

DESTINATION: M. Lochner, E3300, X3457

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSE(S): To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)
FIRST	E3300 RM 102	93/01/21
SECOND		
THIRD		
FOURTH		
FIFTH		

	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	LOCHNER, JOSEPH M	SCBRD-RTS		217-48-6611
SECOND				
THIRD				
FOURTH				
FIFTH				

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	1 EA WOOD BOX	ENVIRONMENTAL SAMPLE #8 3016-1 PIPE VALLEY, WASH DC	SAMPLE, SPRING
		NOTHING FOLLOWS	

DD FORM 1911 (OCT 88 MAY) PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 93.

SAMPLES OTH-893

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: 93-008

SHIPPER: USATEU

SUPPLY ACCOUNT NUMBER:

DESTINATION: ERDEC

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)
FIRST	Spring Valley, Washington D.C.	93/01/21
SECOND	ERDEC E3300	93/01/21
THIRD		
FOURTH		
FIFTH		

	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	BROWER James C	TE4	James C Brower	097-40-9134
SECOND	[illegible]		[illegible]	115-16-7254
THIRD				
FOURTH				
FIFTH				

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1	1	#9	4 Items, (w/ Hardware)
		Nothing Follows	

DD FORM 1911 MAY 88 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-993

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: TE4 93-22

SHIPPER: USA TEU Sig'd in ORR

SUPPLY ACCOUNT NUMBER:

DESTINATION: ERDEC Edgewood

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)
FIRST	SPRING Valley	93-01-22
SECOND	E3300	93/01/22
THIRD		
FOURTH		
FIFTH		

	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	BROWER James C	TE4	James C Brower	097-40-9134
SECOND	LOCKNER Joseph M	SCRD-RTS	Joseph M Lockner	212-18-6611
THIRD				
FOURTH				
FIFTH				

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1	1	Pg 10	(7) Saddle Kevlars

DD FORM 1911 MAY 88 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1093

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: 92-011

SHIPPER: USATEU — SUPPLY ACCOUNT NUMBER:

DESTINATION: BLDG #5300, APG MD: Spring Valley, Washington DC — SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	Spring Valley, Washington DC	93/01/23				
SECOND	Bldg #5352, APG, MD 21010	93.01.23	HOFFMANN, JOSEPH R.	USATEU		065-36-2859
THIRD	BLD 5352 APG MD 21010	93.01.25	Pechillo, David M	USATEU	David M Pechillo	218-70-4502
FOURTH	BLD E 5352	93/01/27		SFIKD-RTS		217-[illegible]
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1	5	#11, 12, 13, 14, 15	Various samples from [illegible]
			Nothing Follows

DD FORM 1911 PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1193

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: 93-011

SHIPPER: USATEU — SUPPLY ACCOUNT NUMBER:

DESTINATION: Bldg 3300, [illegible] — SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	SPRING VALLEY	93-01-26	BROWER JAMES C.	TEU	James C Brower	097-40-9134
SECOND	Bldg #E5352 APG MD 210N	93/01/26	HOFFMANN, JOSEPH R	USATEU		065-36-2859
THIRD	Bldg # E 5352 APG MD 21010	93/01/27	BANKS, MARK A. MAJ	USATEU	Mark A. Banks MAJ	[redacted]
FOURTH	BLD E 5352 APG MD 21010	93/01/27	Pechillo, David M	USATEU	David M Pechillo	218-70-4502
FIFTH	[illegible]	93/01/27	[illegible]	[illegible]	[illegible]	217-[illegible]-6611

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1	5	#17, 18, 19, [illegible] 52	Soil samples [illegible]
			#5-10
			Hazardous waste
			for [illegible] use in
			Pkg #5-1.
			Nothing Follows

DD FORM 1911 PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1293

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.	93 [illegible] 004-0-5032-MRD
SHIPPER	T.A. BLADES, E5832, X4202
SUPPLY ACCOUNT NUMBER	
DESTINATION	M. BROOKS, E3300, X3957
SUPPLY ACCOUNT NUMBER	

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

Transfer	Location of Transfer	Date (YR/MO/DAY)	Recipient's Printed Name (Last, First, M.I.)	Organ. or Account No.	Signature	Social Security Number
FIRST	E3300 ERDEC	2/93 [illegible]	[illegible]		[illegible]	115-[illegible]-7254
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

Line Number	Quantity	Serial Numbers	Remarks
1.	10ml	ITEM #87	LIQUID SAMPLE
2.	2EA	TENAX TA CTF-[illegible] TENAX GR-CTF-[illegible]	VAPOR SAMPLE
		NOTHING FOLLOWS	

DD FORM 1911, MAY — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1393

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.	93-018
SHIPPER	USATEU
SUPPLY ACCOUNT NUMBER	
DESTINATION	1) USAMRIID, McCaulde 2) [illegible] ERDEC, M. Brooks
SUPPLY ACCOUNT NUMBER	

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

Transfer	Location of Transfer	Date (YR/MO/DAY)	Recipient's Printed Name (Last, First, M.I.)	Organ. or Account No.	Signature	Social Security Number
FIRST	Spring Valley Wash DC	93/01/29	Penhallow, David M	USA TEU	David M Penhallow	218-70-4302
SECOND	USAMRIID	93/01/29	CAUDLE, LETTER C. III	USAMRIID	L.C. Caudle	575-68-0880
THIRD	USAMRIID	93/01/29	Penhallow, David M	USA TEU	David M Penhallow	218-70-4302
FOURTH	Bldg #E5352 APG, MD	930129	HOFFMANN, [illegible] R	USATEU	[illegible]	065 36 0899
FIFTH	Bldg E5352	93-02-1	BROWER JAMES C	USATEU	James C Brower	097409134

SHIPMENT DESCRIPTION

Line Number	Quantity	Serial Numbers	Remarks
1	1	#23	4 items in pig: 1) SOL 97: Broken glass bottle w/ black substance 2) SOX 98: Brown filter [illegible] inside [illegible] #141 3) SOX 99: Lead bolts + filter [illegible] inside [illegible] #151 4) Lead bolts [illegible] inside [illegible] #100
		Nothing Follows	
2	1	Small Silver metal Container	Sample for Dr. Margaret Brooks - ERDEC
3	1	Letter for LTC Bill	BATT
		Nothing Follows	
4	1	LTR FOR LTC BATT GIVEN TO [illegible] (65CDO [illegible]) + LOGGED.	

DD FORM 1911, MAY — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

APPENDIX I

SAMPLES OTH-1493

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBRD-ODC-0-3035-1488

SHIPPER: G.L. Smith, ERDEC, X4683

SUPPLY ACCOUNT NUMBER:

DESTINATION: M. Brooks, E3300 X3957

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	E3300, ERDEC		[illegible]		[illegible]	[illegible]
SECOND	E 3300	93/02/05	Rice Philip C	KDESD / ERDEC	Philip Rice	212-84-3211
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	10 ml	Item #67	Liquid Sample
2.	10 ml	Item #113	Liquid Sample
		NOTHING FOLLOWS	

DD FORM 1911 (OCT MAY)

PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1493

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBRD-ODC-0-3683-1491

SHIPPER: G.L. Smith, E3832, x4382

SUPPLY ACCOUNT NUMBER:

DESTINATION: M. Brooks, E3300, x3957

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	E3300	91/1.	[illegible]		[illegible]	[illegible]
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	1 mg	Item #147	Solid Sample (cotton)
2.	5 ml	Item #67	Liquid Sample
3.	2 mg	Item #67	Solid Sample (metal)
4	2 ml	Item #67	Vapor Sample (Tenax Tube)
		Nothing Follows	

DD FORM 1911 (OCT MAY)

PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTB-1593

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBRD-ODC-O- 3033-1493

SHIPPER: G.L. SMITH E3532 X4302

SUPPLY ACCOUNT NUMBER:

DESTINATION: M. BROOKS, E3330 X3957

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST						
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	2EA	ITEM # 147	TENEX TUBES
		NOTHING FOLLOWS	

DD FORM 1911 (NO MAY BE USED) PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTB-1593

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SCBRD-ODC-O-3033-1493

SHIPPER: G.L. Smith E3532

SUPPLY ACCOUNT NUMBER:

DESTINATION: M Brooks, E3300, x3957

SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	E 3300	930202				
SECOND	E3300	930202	TAYLOR, HARRY C.	EFDC		214566372
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	2 ea	Item # 40	Vapor Sample (Tenax Tub.)
2.	1 w/10ml	Item # 90	Liquid Sample
3.	1 ea	Item # 40	Metal Shavings
		NOTHING FOLLOWS	

DD FORM 1911 (NO MAY BE USED) PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1693

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SC880-00C-0-3033-1484

SHIPPER: G.L. Smith E3832 — SUPPLY ACCOUNT NUMBER:

DESTINATION: M. Brooks, E3300, X3957 — SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	E3300	930202	Brooks			
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	2 ea	Item # 113	Vapor Sample (Tenax Tubes)
2.	1 ea 10 ml	Item # 113	Liquid Sample
3.	1 ea	Item # 113	Metal Shavings
		Nothing Follows	

DD FORM 16 MAY 1911 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.

SAMPLES OTH-1793

MATERIEL COURIER RECEIPT

SHIPPER'S CONTROL/DOCUMENT NO.: SC880-00C-0-3033-1485

SHIPPER: G.L. Smith E3832 — SUPPLY ACCOUNT NUMBER:

DESTINATION: M. Brooks, E3300, X53597 — SUPPLY ACCOUNT NUMBER:

I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.

PRIVACY ACT STATEMENT

AUTHORITY: 5 U.S.C., Sec 552a (PL 93-579)
PRINCIPLE PURPOSES: To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receipting for the material.
ROUTINE USES: To document transfer of material from a shipper to a courier, courier to courier and/or receiver.
DISCLOSURE IS VOLUNTARY: Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.

SHIPMENT TRANSFERS

	LOCATION OF TRANSFER	DATE (YR/MO/DAY)	RECIPIENT'S PRINTED NAME (LAST, FIRST, M.I.)	ORGAN. OR ACCOUNT NO.	SIGNATURE	SOCIAL SECURITY NUMBER
FIRST	E3300	93/02/02	Brooks			
SECOND						
THIRD						
FOURTH						
FIFTH						

SHIPMENT DESCRIPTION

LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
1.	2 ea	Item # 142	Vapor Sample (Tenax Tubes)
2.	1 ea 10 ml	Item # 142	Liquid Sample
3.	1 ea	Item # 142	Metal Shavings
		Nothing Follows	

DD FORM 16 MAY 1911 — PREVIOUS EDITION MAY BE USED UNTIL 31 DEC 82.