PINELLAS COUNTY FORENSIC LABORATORY

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FAX Transmission Cover Sheet

Date: 1/08/99

Sent to FAX number: (615) 741-4787

Please Deliver to: Phil Freere

From: Reta Newman

There is a total of 6 **pages, including this cover sheet.**

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Methods File - GHB

Pinellas County Forensic Laboratory

Procedure and Method Validation for the Analysis of Suspected GHB Cases in Aqueous Samples

1. Evaporate 1ml of sample to dryness.
2. Dissolve residue in EtOH.
3. Run GHB.M (on GC-MS-IRD) or GHB2.M (on GC.MS) method on GC-MS
4. If GBL is present in data then continue to Step 5. If GBL is not indicated then your sample does not contain GBL or GHB. Run a routine drug screen.
5. Extract 5ml of the original solution with excessive amount of CH2Cl2 to remove GBL.
6. Extract remaining solution one more time with equal volume of CH2Cl2 and concentrate the CH2Cl2 portion (save the aqueous solution for Step 7) of extract and analyze by GC-MS to confirm that all the GBL has been effectively removed. If substantial GBL is present repeat Steps 5-6.
7. Evaporate the extracted sample to dryness with low heat (not to exceed 150C). Higher temperatures may convert the GHB to GBL. Do not char. Re-crystallize with acetone. Decant the acetone to remove any residual GBL. Air dry sample. Water will inhibit the derivatizing agents, continue to dry sample using low heat and acetone as necessary.
8. Immediately, place a 25 mg sample of the dried sample in a test tube.
9. Add 0.5ml BSTFA and 0.5ml of TMCS to the dried sample, then add 2 ml of acetonitrile. SAFETY GLASSES, GLOVES, LAB COATS AND A FUME HOOD ARE REQUIRED!!!!!
10. Place the beaker on a 60-80C hot plate **DO NOT** exceed 80C. If solution boils – it is too hot. Allow to react for 15-20 minutes.
11. Analyze by GC-MSD or GC-MSD-IRD (standard data attached).

Case jackets require a minimum of:

1. The initial GC-MS data from the screen in Step 3.
2. The GC-MS data from the extract in Step 6 to confirm the removal of GBL.
3. The GC-MS and GC-IRD data from the derivatized sample.
4. Color tests on the dry extracted sample. (Marquis – NC, CoSCN –SnCl2 – NC, Ferric Chloride – Rust precipitate).
5. pH of the original liquid sample.

Method was validated November, 1998. 7 unknown samples were prepared by Joan Ring and analyzed by R. Newman. The sample preparation is attached. All the unknowns were properly identified using the derivitization technique.

References:

Johnson, Ruth and Bussey, Janet. FDA Laboratory Information Bulletin, No. 3532.
Andrews, K. Personal Communication, DEA Western Regional Laboratory.
Andrews, K. Presentation Notes: The Analysis of GHB and its Butyrolactone.

Date: December 2, 1998

To: Reta Newman
From: Joan Ring

Re.: GHB Sample Information for Method Validation

Sample 1: 24 mg GHB + 20 mls Gatorade
Sample 2: 32 mg GHB + 20 mls distilled water
Sample 3: 31.8 mg GBL + 20mls Gatorade
Sample 4: 24 mg GBL + 20 mls water
Sample 5: 20mls Gatorade
Sample 6: 20mls distilled water
Sample 7: 33.8 mg GHB + 20.2 mg GBL + 20mls Gatorade

's was published in the November 25, 1998 FAW.

ection IV

ergency Rules

epartment of Legal Affairs
ULE TITLE: ..... RULE NO.:
dition of Gamma-Hydroxybutyric Acid (GHB) to Schedule II, Subsection 893.03(2)(a), F.S. 2ER98-1
folio.pci/2448.nfo/query=[JUMP:'2ER98!2D1']/doc/{@1}?firsthit>
PECIFIC REASONS FOR FINDING AN IMMEDIATE DANGER TO THE PUBLIC HEALTH, SAFETY, AND WELFARE: written findings published on the date this emergency rule was filed with the Secretary of State's Office, Attorney eneral Butterworth has found that a deficiency in the current nomenclature (Gamma-hydroxy-butyrate) currently used in 893.03(2)(a)6., Florida Statutes, for the controlled substance commonly known as GHB - when considered in light of the ontinuing problem with the abuse of GHB by Florida's children and young adults presents an immediate danger to the ublic health, safety and welfare, which requires emergency action. In addition, the Attorney General has found that the ubstance gamma-hydroxybutyric acid meets the statutory criteria for placement as a controlled substance in Schedule II, ubsection 893.03(2)(a), Florida Statutes.
EASONS FOR CONCLUDING THAT THE PROCEDURE USED IS FAIR UNDER THE CIRCUMSTANCES: During the 997 regular session, the Florida Legislature designated "Gamma-hydroxy-butyrate (GHB)" a controlled substance in chedule II of the Florida Comprehensive Drug Abuse Prevention and Control Act, Chapter 893, Florida Statutes. (See hapter 97-1, Laws of Florida.) However, the statutory nomenclature describing GHB has resulted in significant problems the prosecution of criminal cases involving this substance. In medical and scientific evaluations submitted to the ttorney General pursuant to section 893.035, Florida Statutes, both the Florida Department of Health and the Florida epartment of Law Enforcement have recommended that gamma-hydroxybutyric acid be designated a Schedule II ontrolled substance. A copy of the Attorney General's findings in support of this emergency rule may be obtained by ontacting the Office of the Attorney General, PL-01, The Capitol, Tallahassee, Florida 32399-1050, (850)487-1963.
UMMARY OF THE RULE: Under the authority of section 893.035, Florida Statutes, the substance gamma-ydroxybutyric acid (GHB) is being added as an enumerated controlled substance in Schedule II, subsection 893.03(2). lorida Statutes.
HE PERSON TO BE CONTACTED REGARDING THE EMERGENCY RULE IS: Marty Moore, Deputy General Counsel, L-01, The Capitol, Tallahassee, Florida 32399-1050
HE FULL TEXT OF THE EMERGENCY RULE IS:
ER98-1 Addition of Gamma-Hydroxybutyric Acid (GHB) to Schedule II, Subsection 893.03(2)(a), F.S.
Under the authority of section 893.035, F.S., the substance gamma-hydroxybutyric acid, any salt, compound, derivative preparation of gamma-hydroxybutyric acid, including any isomers, esters, ethers, and salts of isomers, esters and thers of gamma-hydroxybutyric acid, whenever the existence of such isomers, esters, ethers and salts is possible within he specific chemical designation, is hereby a controlled substance added to Schedule II, subsection 893.03(2)(a), F.S. 2) All provisions of Chapter 893, F.S., applicable to controlled substances listed in Schedule II, subsection 893.03(2)(a), F.S., shall be applicable to gamma-hydroxybutyric acid, any salt, compound, derivative or preparation of gamma-ydroxybutyric acid, including any isomers, esters, ethers, and salts of isomers, esters and ethers of gamma-ydroxybutyric acid, whenever the existence of such isomers, esters, ethers and salts is possible within the specific hemical designation.
Specific Authority 893.035 FS. Law Implemented 893.035 FS. History-New 11-9-98.
THIS RULE TAKES EFFECT IMMEDIATELY UPON BEING FILED WITH THE DEPARTMENT OF STATE.
EFFECTIVE DATE: November 9, 1998

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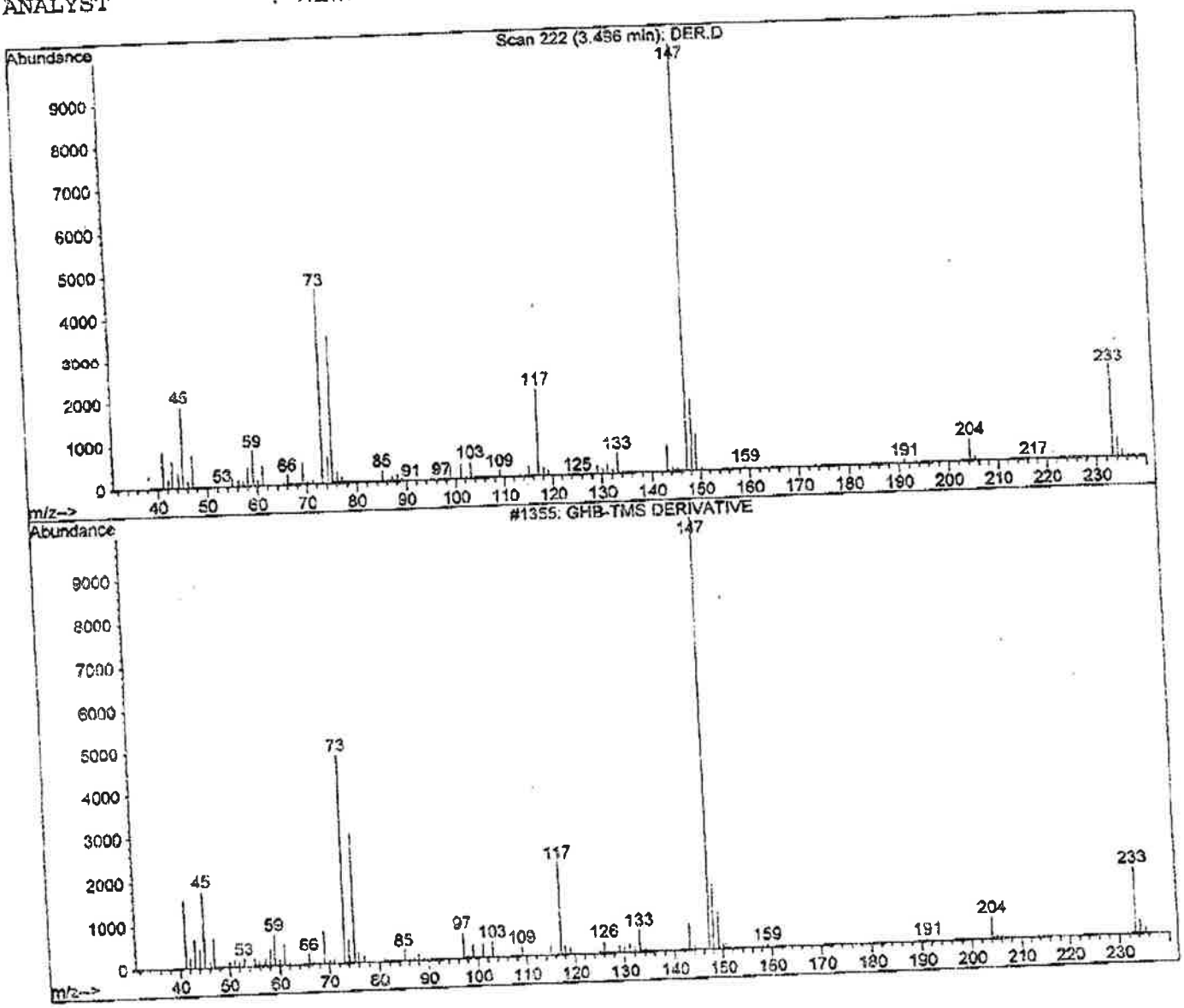
ID : GHB-TMS DERIVATIVE
ANALYST : NEWMAN



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ANALYST= NEWMAN
Mask Used = None

Text Search = Yes
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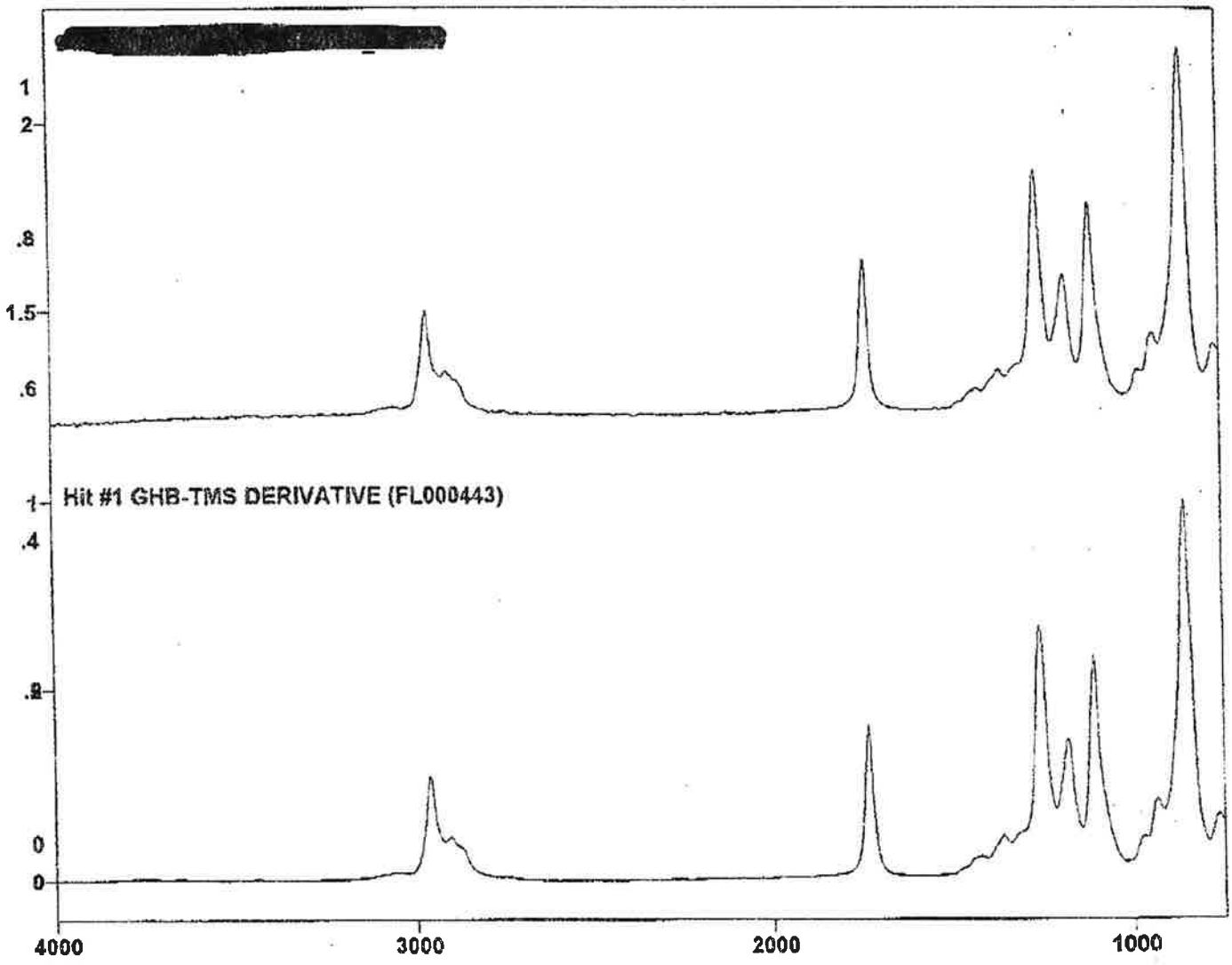


Hit List

Wavenumber (cm-1)

Library	Hit Quality	Number	SPC Identification
C:\DATABASE\IRD\DRUG	.12125	1	GHB-TMS DERIVATIVE

IDENTIFICATION OF THE POTASSIUM SALT OF GAMMA-HYDROXYBUTYRATE (GHB-K^+)

LARA WALKER, CRIMINALIST
California Department of Justice-Freedom Laboratory
440 Airport Blvd., Bldg. A
Watsonville, CA 95076

Recently, our laboratory was asked to analyze a suspected gamma-hydroxybutyrate (GHB) lab that was found in Santa Clara County. The lab site consisted of glassware, clear liquids, and a cooperative suspect that identified the unlabeled chemicals as "lactone" and "potassium hydroxide." The amount of liquid GHB present was the equivalent of over 1,000 doses.

This laboratory encountered two problems with the analysis. The first was the extreme hygroscopic nature of the dried sample, which was inconsistent with the hygroscopic qualities observed with the sodium salt of GHB. It was evident that the sodium salt dried in the oven faster and did not absorb moisture from the air as readily as the potassium salt. The second problem arrived when the infrared spectrum was inconsistent with reference spectra previously published or seen with laboratory standards of the sodium salt of GHB (Fig. 2). Based on the suspect's admission, the potassium salt of GHB was suspected, and it was speculated that the IR spectrum (Fig. 3) and consistency of the drug may be different than previously encountered with the sodium salt of GHB.

As of January 1999, a literature reference and a traceable standard of the potassium salt of GHB were not located. A GHB-K^+ standard was prepared by combining 5 mls of (GBL) gamma-hydroxybutyric acid lactone (Fig. 1) with 5 mls of a 50% aqueous potassium hydroxide solution. To confirm that GHB synthesis was successful, the mass spectra of GHB-K^+ was compared with the mass spectra of a known GHB-Na^+ standard (Figs. 4 and 5). Both samples were prepared by drying and silylating them with BSTFA and 1%TCMS. Forensic Chemist Kathleen Andrews of DEA's Western Laboratory performed additional confirmation of GHB-K^+ using NMR.

Following analysis, it was confirmed that the potassium and sodium salts of GHB have different IR spectra. It was also determined that the potassium salt of GHB needs a much longer drying time than the sodium salt.

Due to the rapid conversion of GBL to GHB by peripheral lactonases in the body, the Department of Justice-Bureau of Forensic Services has recently adopted the position that GBL is an analog of GHB as defined in Section 11401 of the California Health and Safety Code. To be deemed an analog, the chemical structure of the substance must be substantially similar to the chemical structure of the controlled substance OR "...the substance has, is represented as having, or is intended to have a stimulant, depressant, or hallucinogenic effect on the central nervous system that is substantially similar to, or greater than, the stimulant, depressant, or hallucinogenic effect on the central nervous system of a controlled substance classified in Section 11054 or 11055."

The following is the Freedom Laboratory's method for GHB analysis.

Appearance

Clear or colored liquid with possible flavoring, white powder that is hygroscopic, white paste-like material, or white waxy/soap-like chunks.

Odor

The liquid GHB and GBL have slight "burnt plastic" odors

Solubility

GHB is soluble in water and alcohols. It is slightly soluble in acetonitrile and ethyl acetate. It is insoluble in petroleum ether, ethyl ether, chloroform, and methylene chloride.

GBL is soluble in chloroform, methylene chloride, alcohols, ether, acetone, and benzene.

Color Screening Tests

Two color tests are available that indicate the possible presence of GHB (*see Frommold and Busby*). GBL alone does not react with these color tests. However, KOH and NaOH alone will. This laboratory uses them after other common color tests have shown no reaction.

1. **5% Ferric Chloride solution:** Dissolve 5 g of ferric chloride in 100 ml of distilled water. (Reddish-orange is positive with any alkaline solution) **Note:** Works well with liquid or solid samples
2. **1% Cobalt Nitrate solution:** Dissolve 3.9 g of Cobaltous Nitrate in 500 ml of methanol (Light purple is positive. KOH and NaOH give a gray color.)

Note: This color test does not work very well with liquid samples. Dry a couple of drops in 58°C oven if necessary prior to color test.

GC/MS (as a screen)

Methanol extracts of dry samples can be injected. GHB converts to the butyrolactone upon injection so a confirmation is still required.

Acidic extracts in chloroform convert liquid GHB samples back to the GBL and can be injected in trace amounts prior to drying.

Parameters

This method is set up to identify Rohyponol as well and can be shortened if necessary.

GC/MS system used is a HP 5890 GC with a HP 5970 MSD. The column is a HP-1, nominal 12 M, 0.18 to 0.23mm ID coated with 100% crosslinked methyl silicone.

Initial oven temp: ..... 40°C
Ramp: ..... 30.0°C/minute
Final temp: ..... 280°C, hold 5 min
Injector temp: ..... 25°C
Detector and transfer line: ..... 280°C
30:1 split ratio

Confirmation

FTIR (recommended)

Dry liquid or solid samples completely prior to analysis. The Freedom Lab uses a 58°C oven overnight. Incorporate the dry sample into a KBr pellet for analysis. The drying time may increase for the potassium salt of GHB.

Sample Preparation To Remove Impurities

If the sample is liquid, excess butyrolactone can be removed by extracting it with chloroform. Save the organic layer.

Note: Sample preparation, other than drying, has not been necessary on the samples seen in this laboratory thus far.

Derivatizing Samples For GC/MS Confirmation

The molecular ion is *m/z* 248, but is not always seen at lower concentrations. Ions common to TCMS derivatives include *m/z* 73, 147, and 243. For identification, ions *m/z* 117(10%), 133(2%), 159(1%), 204(5%), and 233(10%) need to be present at the proper retention time. At high enough concentrations, the molecular ion, *m/z* 248(1%) may be present.

Sample Prep

- ✓ Add 100 µl of BSTFA with 1% TCMS in an autosampler vial to approximately 0.5 mg of dried sample.
- ✓ Mix well and add 100µl of acetonitrile.
- ✓ Cap and heat in a 45°C water bath for 10 minutes.
- ✓ Inject 0.5 ml to 1 ml

Parameters

- ✓ Initial oven temp: ..... 70°C
- ✓ Ramp: ..... 30°C/minute
- ✓ Final temp: ..... 280°C, hold 3.00 minutes
- ✓ 30:1 split ratio

Steps Taken To Identify GHB At This Lab

- ✓ Both color screening tests. Dry liquid samples if they are too diluted.
- ✓ GC/MS as a screening tool. If butyrolactone is present then proceed, otherwise stop.
- ✓ Dry sample completely for FTIR confirmation
- ✓ GC/MS confirmation using the derivatizing agents when FTIR is insufficient or unavailable.

REFERENCES

1. Andollo, Wilmo, Metro Dade County Medical Examiner- "Toxicology Department Methods" (Handout received at "Date Rape Drugs Seminar" April 1998-Santa Clara).
2. Andrews, Kathy, DEA Western Laboratory, *Personal Communication*, December 1997-January 1999.
3. Blackledge, R.D., and Miller, M.D., "The Identification of GHB," *Microgram*, XXIV, No. 7, 172-179, July 1991.
4. Bommarito, C., "Analytical Profile of Gamma-Hydroxybutyric Acid," *Journal of the Clandestine Laboratory Investigating Chemists*, Vol. 3, p. 10, July 1993
5. Dyer, Jo Ellen, Poisoning and Drug Overdose 3rd Edition, A Large Clinical Manual, Olson KR Editor, Appleton & Lange Norwalk, CT, 1998. DRAFT
6. Frommold, S., and Busby, C., "GHB," Southwest Association of Forensic Scientists Journal, 18-2, p. 5-15, Fall, 1996

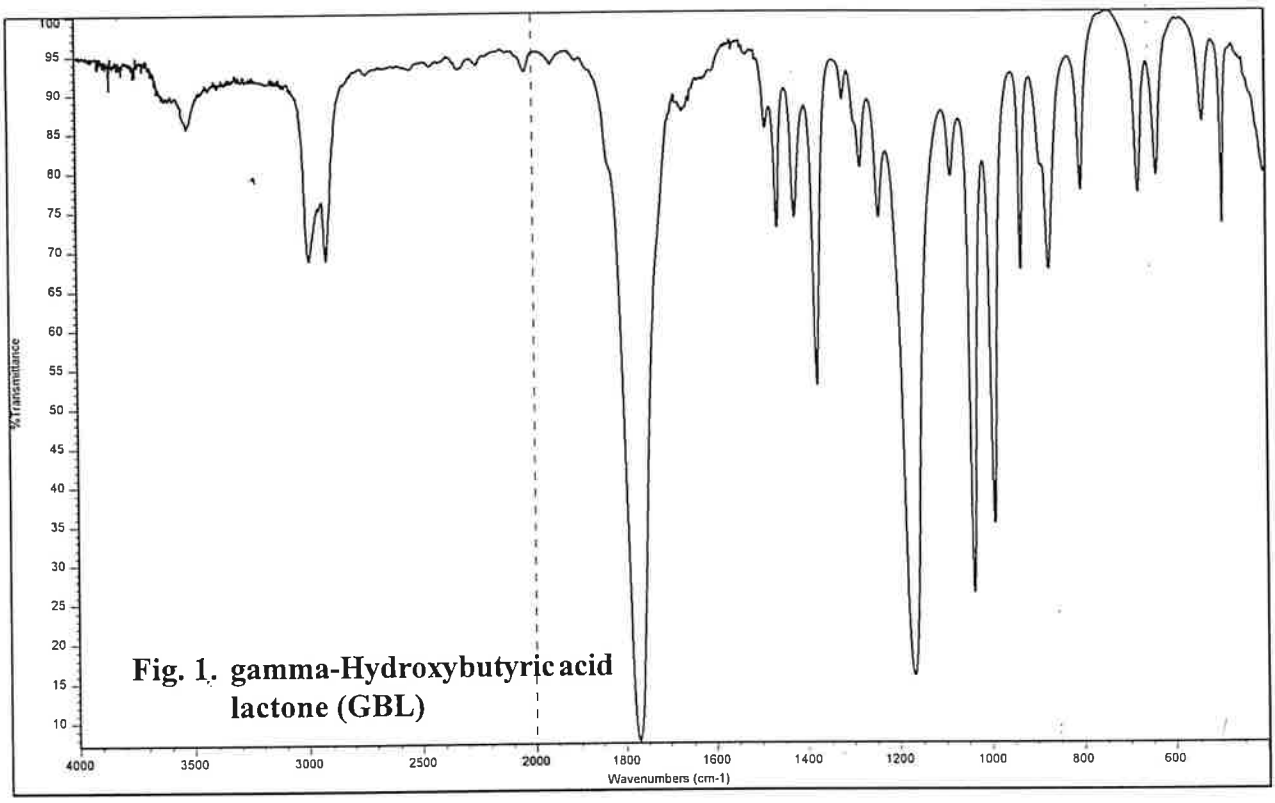


Fig. 1. gamma-Hydroxybutyric acid lactone (GBL)

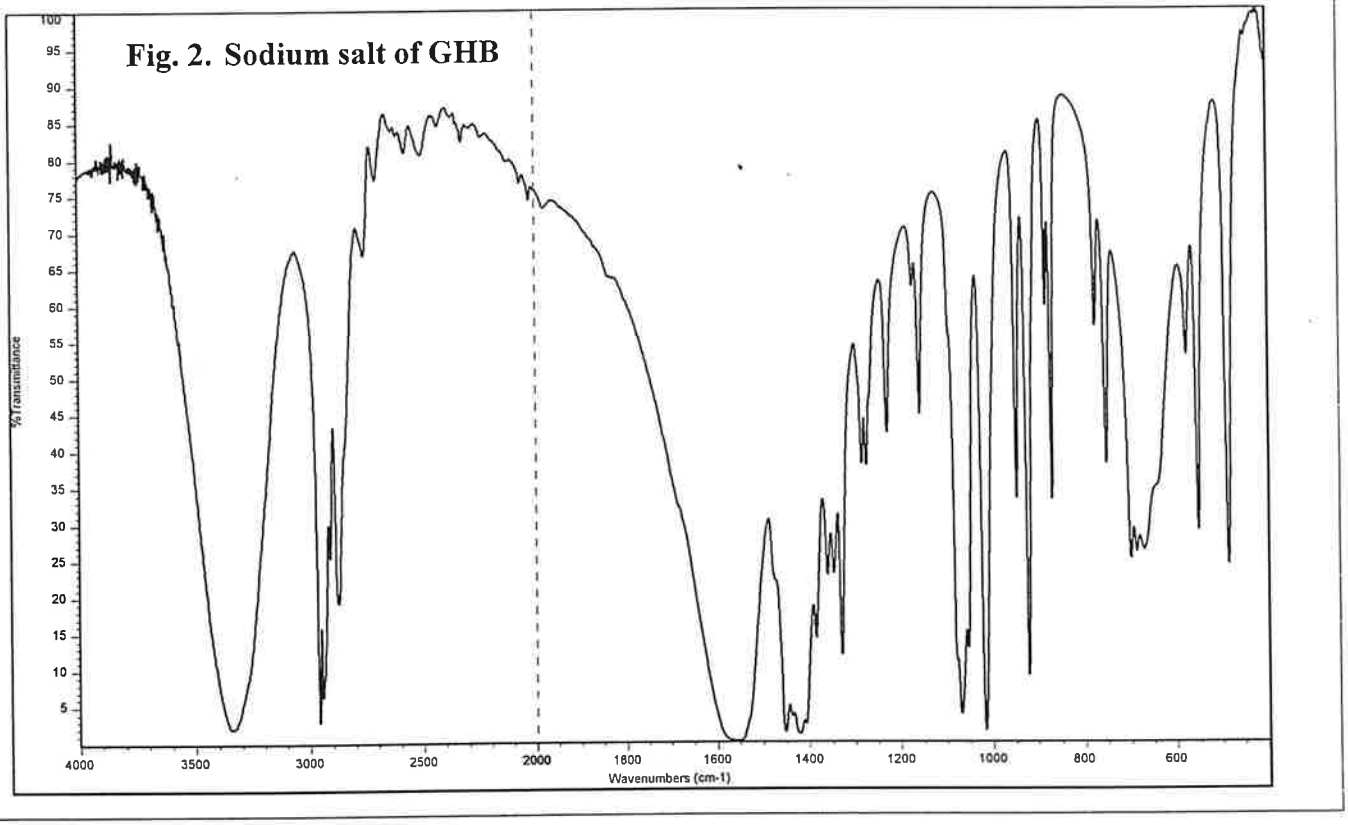


Fig. 2. Sodium salt of GHB

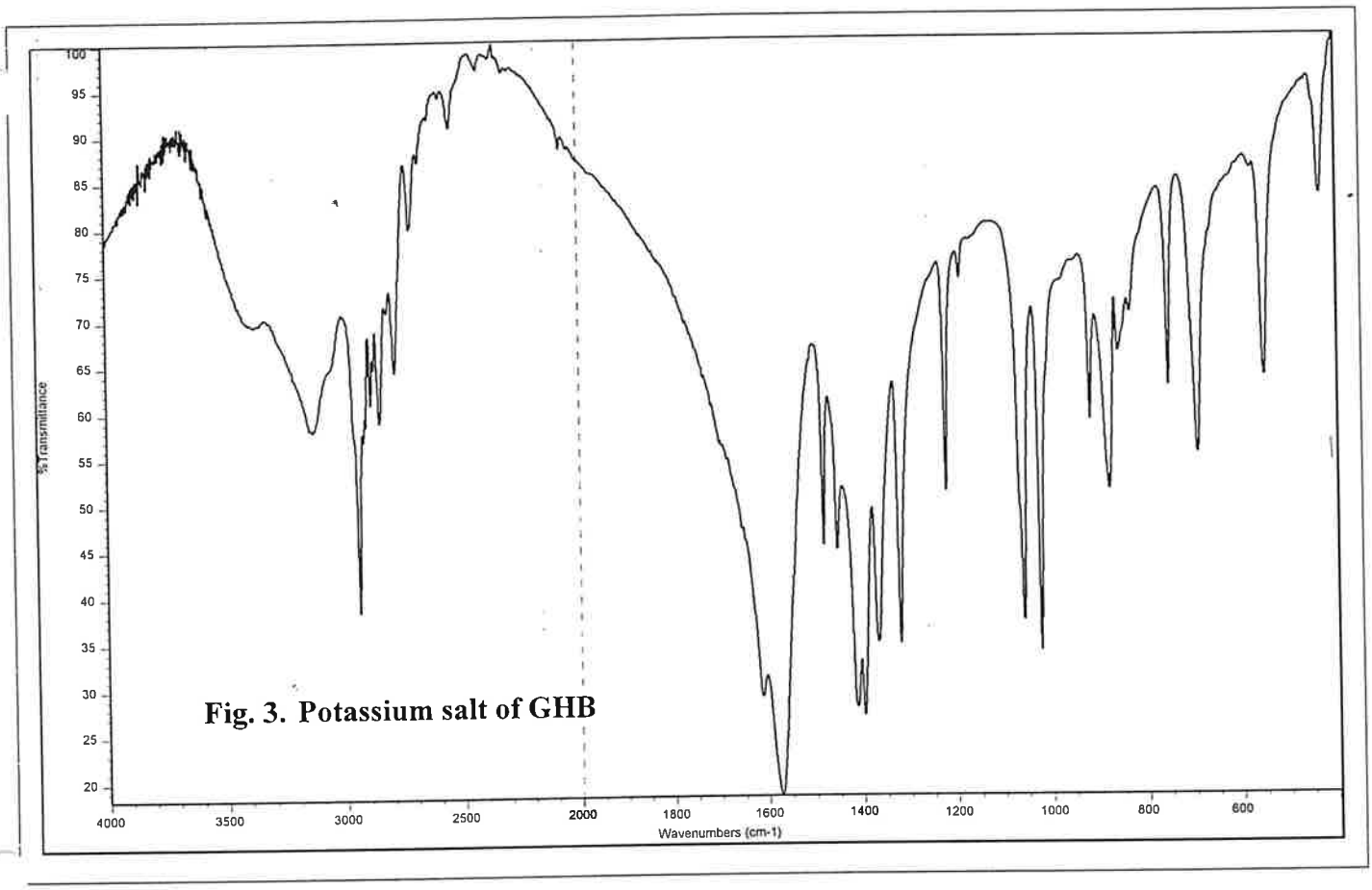


Fig. 3. Potassium salt of GHB

Fig. 4. BSTFA w/ 1% TCMS derivative of GHB-K+

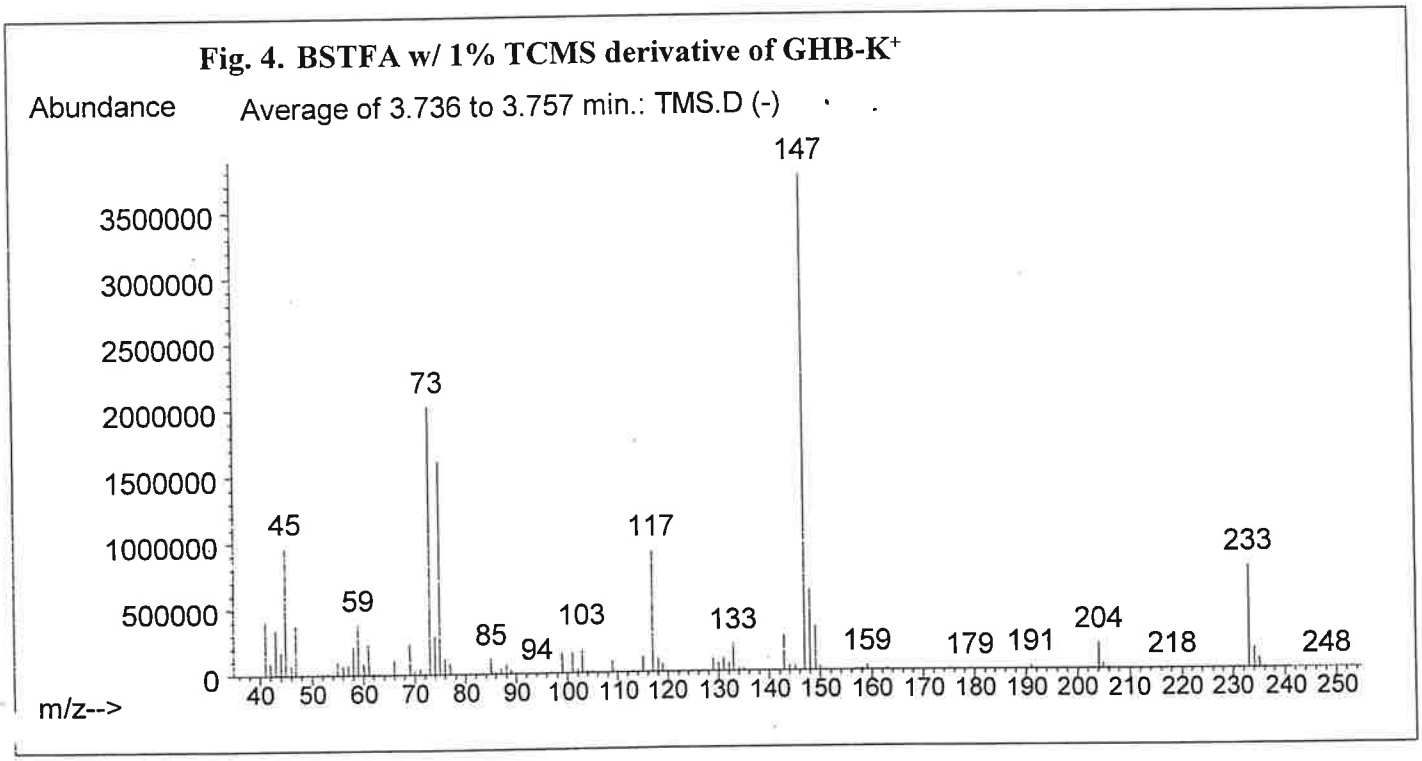
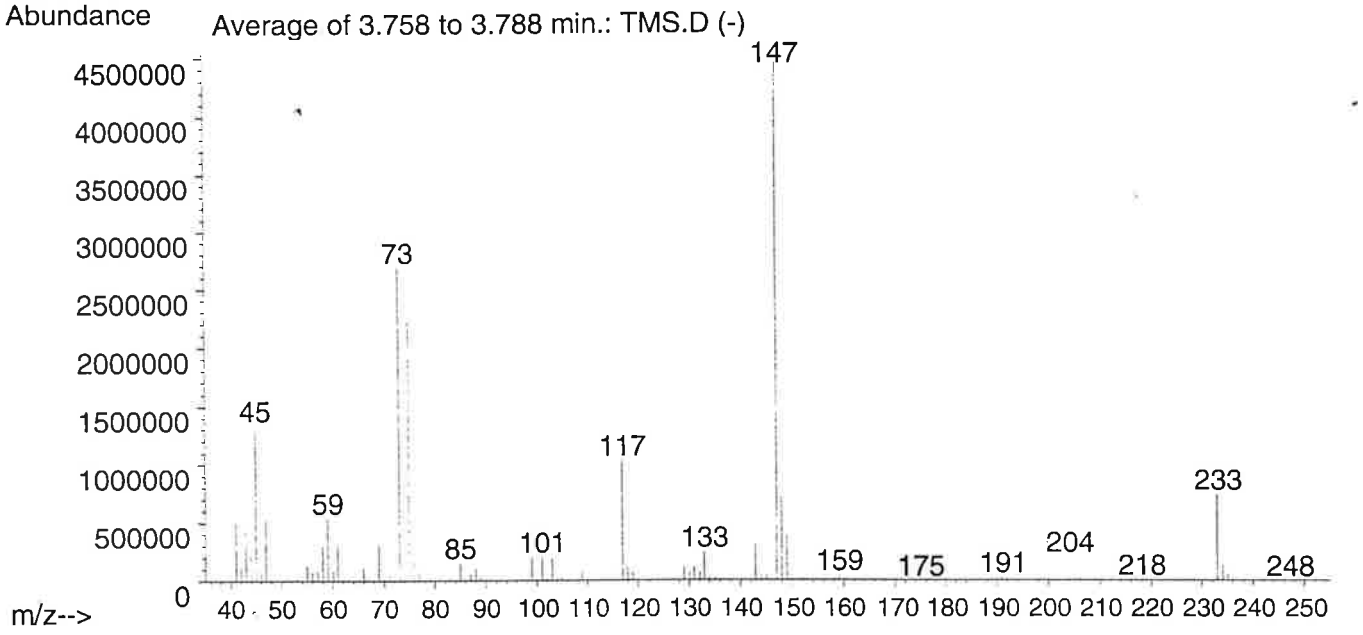


Fig. 5. BSTFA w/ 1% TCMS derivative of GHB-Na$^+$



DEPARTMENT OF JUSTICE
DRUG ENFORCEMENT ADMINISTRATION

FAX TRANSMITTAL SHEET

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TRANSMITTED TO	FAX FTS NO.	FAX COMMERCIAL NO.
		615-741-4787

NAME: William Stanton

ORGANIZATION: Tennessee Crime Lab

BUILDING, ROOM NO., etc.	TELEPHONE/EXTENSION

TRANSMITTED FROM	FAX FTS NO.	FAX COMMERCIAL NO.
		415-744-7055

NAME: Kathy Andrews

ORGANIZATION: JG ENFORCEMENT ADMINISTRATION, WESTERN LABORATORY

BUILDING, ROOM NO., etc.	TELEPHONE/EXTENSION
390 MAIN STREET, ROOM 700, SAN FRANCISCO, CALIFORNIA 94105	(415) 744-7051

COMMENTS:

William:

Sorry I didn't get this out to you earlier. Hope this answers your questions.

If you have any prblems, give me a call. I know that identifying GHB in Coca-Cola is difficult.

APPROVED BY (If applicable)	NAME:

TRANSMITTED BY (Name):	DATE:	TIME:

Suggested Analysis

I. Screens (liquid samples)

a. Acid extraction into ether or chloroform,

- This will convert any GHB into the lactone which will be soluble in the organic layer.

b. Followed by lactone i.d. on FTIR or GC-MS at high injection port temps.

- GHB or lactone originally present in sample.

Post-screen Analysis

Don't alter pH of original sample!

I. Lactone I.D.:

Extraction into ether or chloroform, then FTIR or GC-MS.

II. GHB I.D.:

Air dry liquid down to ppt., followed by

1. FTIR
2. NMR in D_2O
3. Silylation or Alkylation GC work.

Silylation of GHB

FDA Laboratory Information Bulletin No. 3532

20 mg of sample

excess BSTFA(500ul)

excess TMCS (500ul)

2.0 ml acetonitrile

2.0 ml of n-C_{11} as internal stnd

Heated at 80°C for 30 minutes prior to analysis.

Alternative Silylation Method

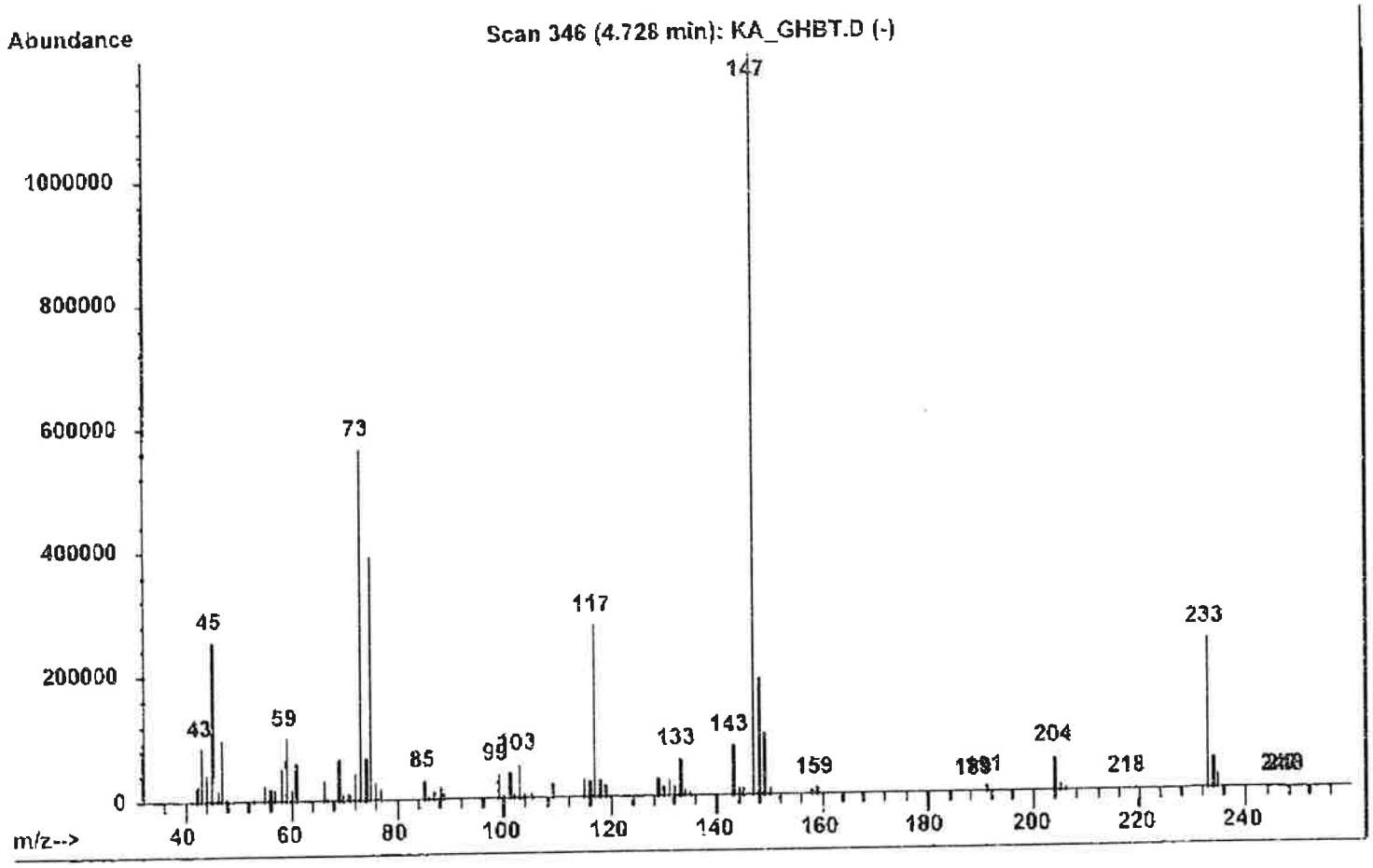
Hexamethyldisilazane (HMDS)

1. To 10mg of sample, add 1ml of HMDS and 1ml of pyridine.
2. Heat mixture to boiling for 30 minutes.
3. Analyze on GC or GC-MS

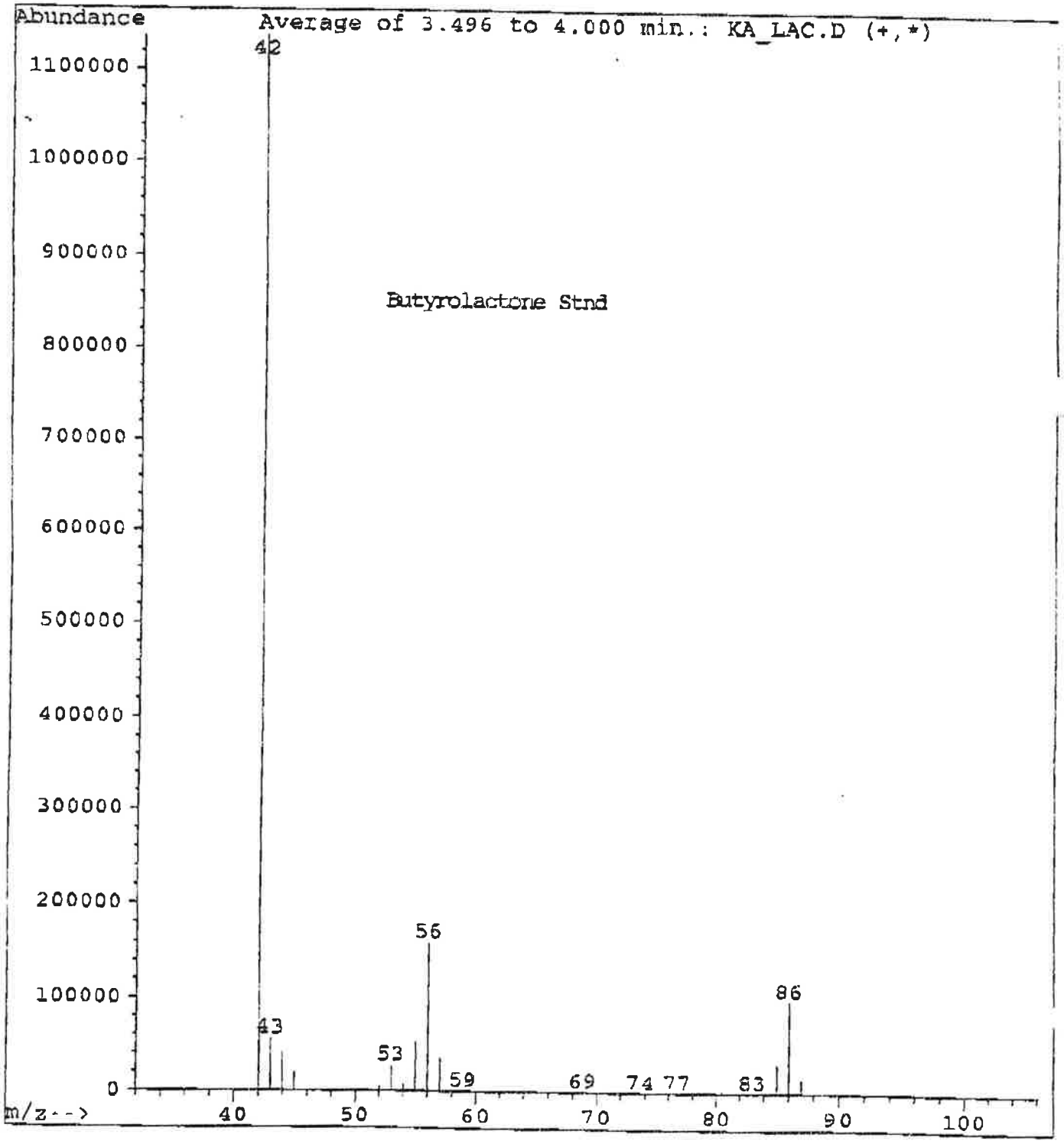
Ref: Alltech GC Reagent Instructions, Cat. No. 18003

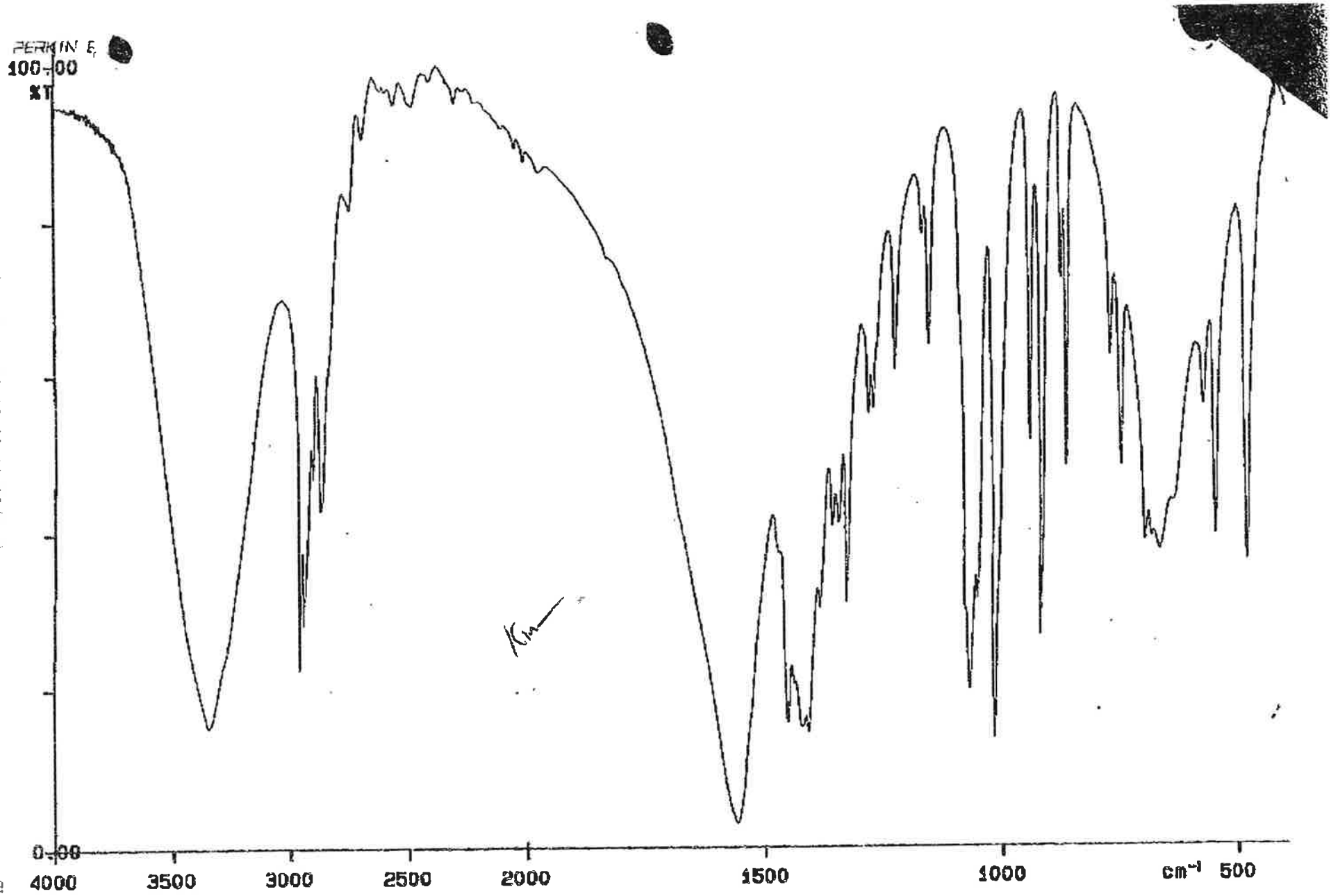
Mass Spectra of GHB-TMS

$$(CH_3)_3\text{-Si-O-}CH_2CH_2CH_2C\text{-O-Si-}(CH_3)_3$$



File : C:\HPCHEM\1\DATA\KA_LAC.D
Operator : KATHY ANDREWS
Acquired : 10 Apr 96 11:48 am using AcqMethod PRM40
Instrument : Mustang
Sample Name: gamma-Butyrolactone stnd
Misc Info : HP-5, 12.5M x 0.20mm x 0.3um
Vial Number: 1





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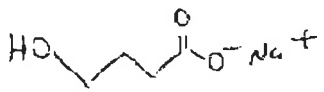

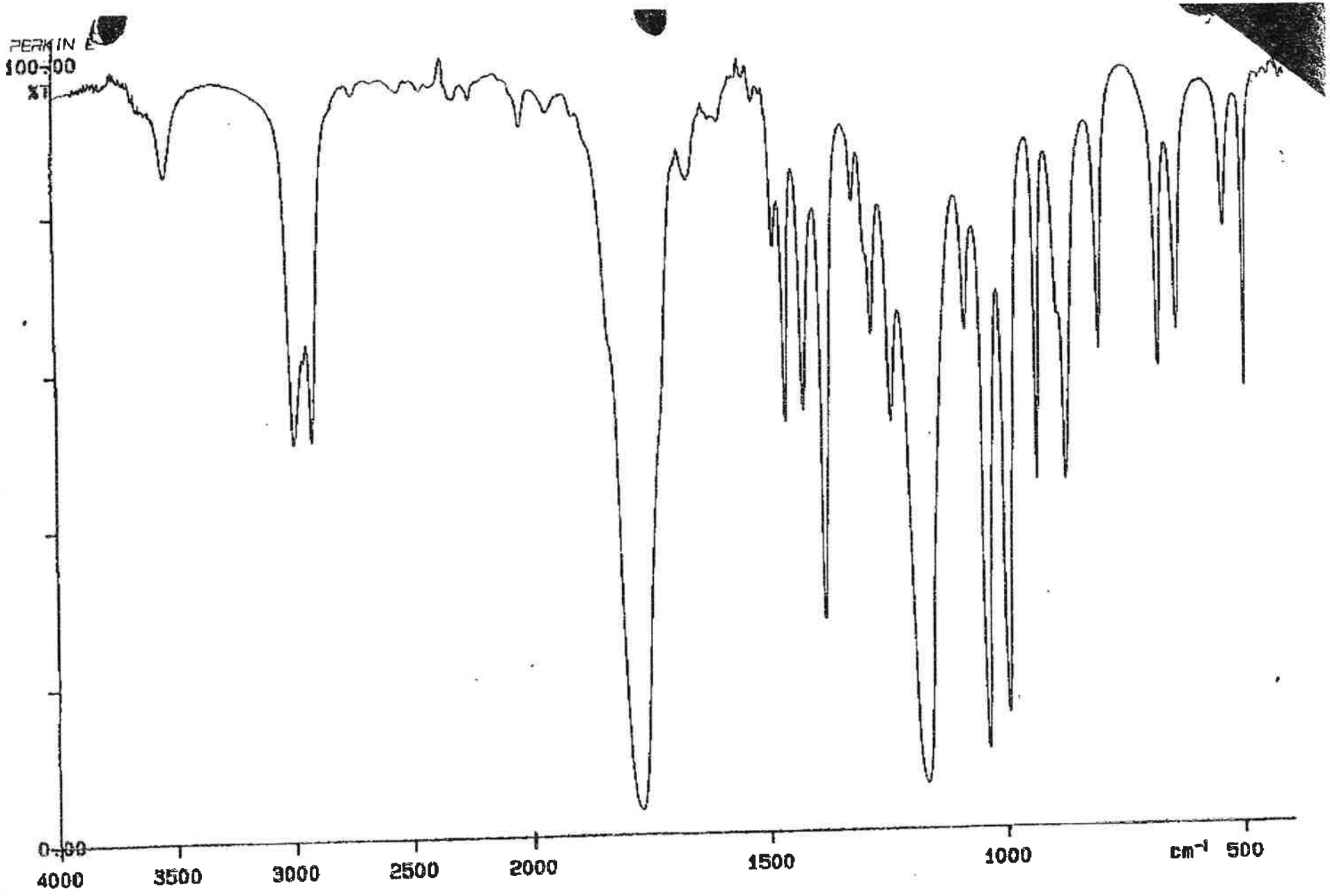

96/04/19 14:35
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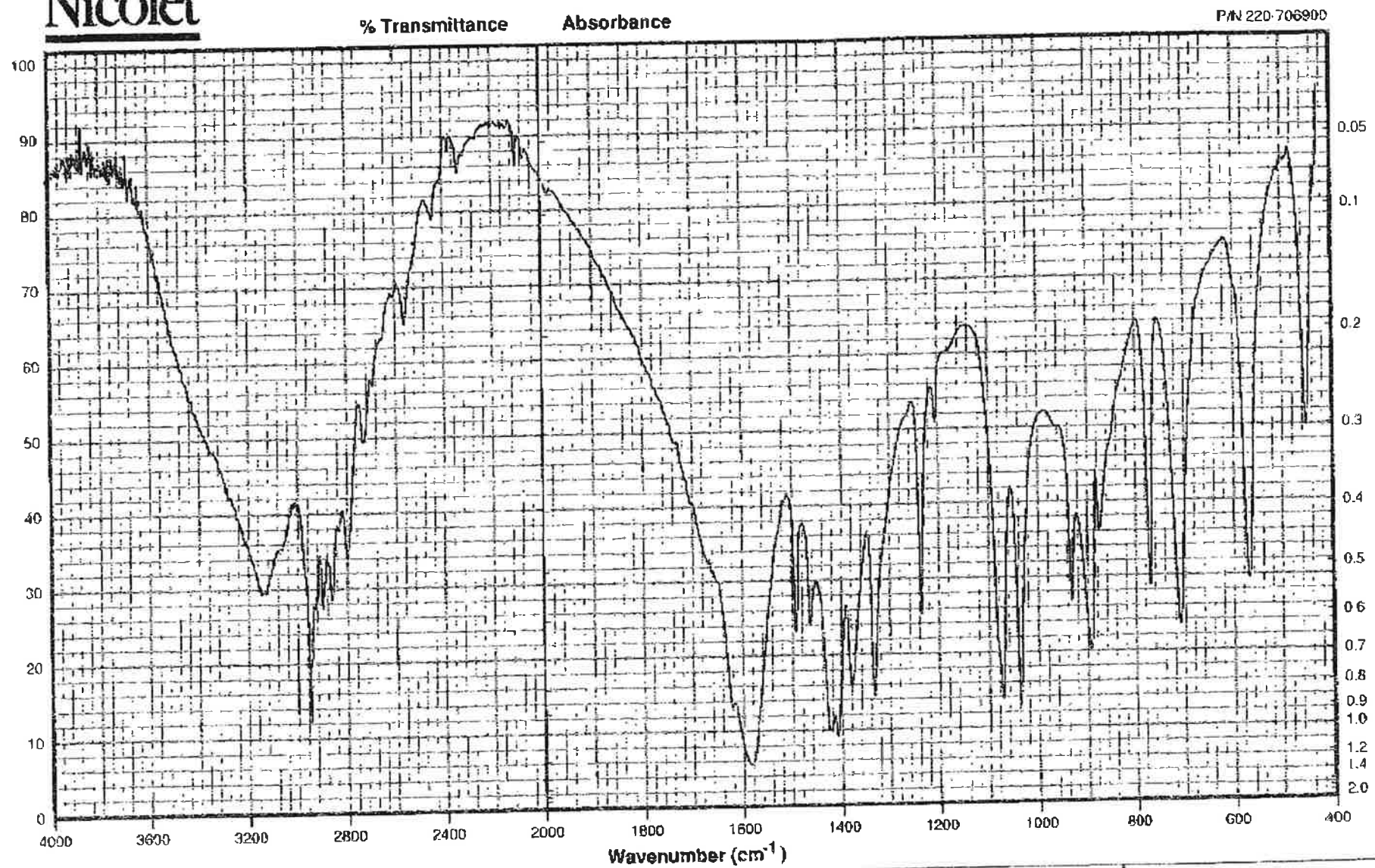
Nicolet

% Transmittance Absorbance

P/N 220-706900



Title: *BUTYROLACTONE + KOH (DRIED) KBr KMA	Calibration: %T, cm-1, 25, 50, ±5, ±25, ±50	Data Processing/Comment: Number of sample scans: 16; Number of background scans: 16; Resolution: 4.000; Sample gain: 1.0; GHB, K+	Resolution
Filename: Collection time: Fri Jan 08 14:00:55 1999			No. Scans
Date / Time			Operator: KMA 31/03/99

The Headaches of Analyzing a GHB Sample

Kathleen A. Andrews
DEA – Western Laboratory
San Francisco, CA, USA

In recent years, GHB has rapidly gained nationwide attention as a date rape drug that is frequently abused by MDMA users, rave partygoers, and body builders. The sudden increase in the drug's abuse within the last two years has prompted at least 15 states to introduce legislative bills seeking control actions on GHB. To date, a total of 12 states have scheduled GHB as a controlled substance. Consequently, as GHB samples are confiscated by law enforcement, crime laboratories are being tasked with having to develop analytical procedures for properly indentifying GHB.

Unfortunately, there is a grey area to the GHB analysis wherein some identification techniques that may be appropriate for a number of street samples are not adequate for others. GHB is illicitly sold in powder form, but is commonly dissolved in a liquid, primarily due to the hygroscopic nature of the drug. Identification of solid GHB samples is easily accomplished by standard FTIR techniques. However the liquid samples require some sample preparation prior to utilizing any analytical techniques. In some cases these sample preps cause additional problems for the analyst. Because GHB lies in equilibrium with its lactone form, namely gamma-butyrolactone, the analysis of both these components can prove to be difficult. The analyst needs to be aware that simple extraction procedures or standard GC screen techniques can cause the equilibrium to shift producing both false negatives and false positives. Qualitative techniques utilizing silylation of the GHB prior to GC analysis can prevent this equilibrium shift and enable the analyst to differentiate correctly between GHB and its lactone. A problem that arises is that these silylation techniques are not always adequate for some of the more commonly seized GHB liquid street samples.

Some suggested techniques for screening and identifying GHB in street samples will be addressed including results of performance evaluations of field test kits and alternative silylation methodology. A brief discussion regarding potential problems encountered by the analyst in court will be presented as well.

GHB Color Test

David J. Koppenhaver
Indiana State Police Laboratory
Fort Wayne, IN 46804

INTRODUCTION

It has been reported, the usual color tests performed in the laboratory yield no results for GHB[1]. If the laboratory's normal operating procedure is to stop analysis after negative results on color tests, UV, and TLC, then a sample of GHB would go undetected.

There is an oxidation reaction that can be used to distinguish between primary and secondary alcohols from tertiary alcohols and most other compounds except aldehydes. Chromium(VI)Oxide in aqueous Sulfuric acid will change from orange to green. The color test will work for GHB sodium salt and also if it is dissolved in water.

EXPERIMENTAL

Chromic Acid reagents were prepared with different concentrations of H_2SO_4 and CrO_3 : Reagent 1; 5M H_2SO_4 saturated with CrO_3 , Reagent 2; 50% H_2SO_4 and 0.25% CrO_3 , Reagent 3; 50% H_2SO_4 , 0.25% CrO_3 , and 0.25% $AgNO_3$. Solutions of 10,000, 14,000, 20,000, and 40,000 ppm of GHB (.75, 1, 2, and 3.5 teaspoons/8 oz. H_2O respectively) were prepared. The GHB sodium salt and the dilutions were placed in a spot plate. Next, two or three drops of reagent were added and the color developed.

DISCUSSION

All reagents performed well with GHB sodium salt. Reagent 1 worked best for the solutions of GHB, followed by Reagent 2, and finally Reagent 3. The time for the color to develop was related to the concentration of the reagent. Silver nitrate in Reagent 3 was to act as a catalyst, but appeared to have little effect on time for the color to develop.

There are many false positives such as alcohols, aldehydes, and common cutting agents i.e., sucrose, lactose. Furthermore, KCr_2O_7 will also give the same results.

REFERENCES

1. Blackledge, R.D., Miller, M.D., "The Identification of GHB", Microgram, Vol. XXIV, No. 7, p. 172-179.

FAX/MAIL ORDER FORM

(850) 436-4879

DIDI
DRUG IDENTIFICATION INC.

P.O. Box 9125 Pensacola, FL 32513-9125 PHONE or FAX (850) 436-4879

Price list for Gamma Hydroxybutyrate Test Kits

Quantity	Product #	Description	Cost / Unit	Total
	GHB001	25 test kits @ $2.50 ea.	$62.50	
	GHB002	50 test kits @ $2.30 ea.	$115.00	
	GHB003	100 test kits @ 2.10 ea.	$210.00	
	GHB004	500 test kits @ 2.00 ea.	$1000.00	
			Shipping & Handling 6%	
Tax Exempt #			or 7% Tax	
			Total	

Customer #: _____
Your P.O. #: _____
Name: _____
Phone: _____
Fax #: _____

Signature: _____

Billing Address:

_____
_____
_____
_____

Shipping Address:

_____
_____
_____
_____

Thank You for Your Order!

Quantitating gamma-Hydroxybutyric Acid By GLC

Prepared by Kathleen Andrews, DEA Western Laboratory

Note: This procedure indirectly quants the GHB thru an acid conversion to its GBL form.

Sample Prep:

1. An alliquot of aqueous sample is washed three times with chloroform, methylene chloride, or ether to extract out any residual GBL in the sample.
2. The aqueous layer is made acidic with concentrated HCl or H_2SO_4. This step will convert the GHB into GBL. I allow the solution to sit for 20 minutes to allow the conversion to occur.
3. The aqueous acidic solution is extracted three times into chloroform, methylene chloride, or ether and further diluted to make an approximately 8-10 mg/ml concentration.
4. To 5.0 ml of this solution is added 5.0 ml of a 0.5 - 0.75 mg/ml solution of n-undecane (n-C_{11}) as an internal standard.

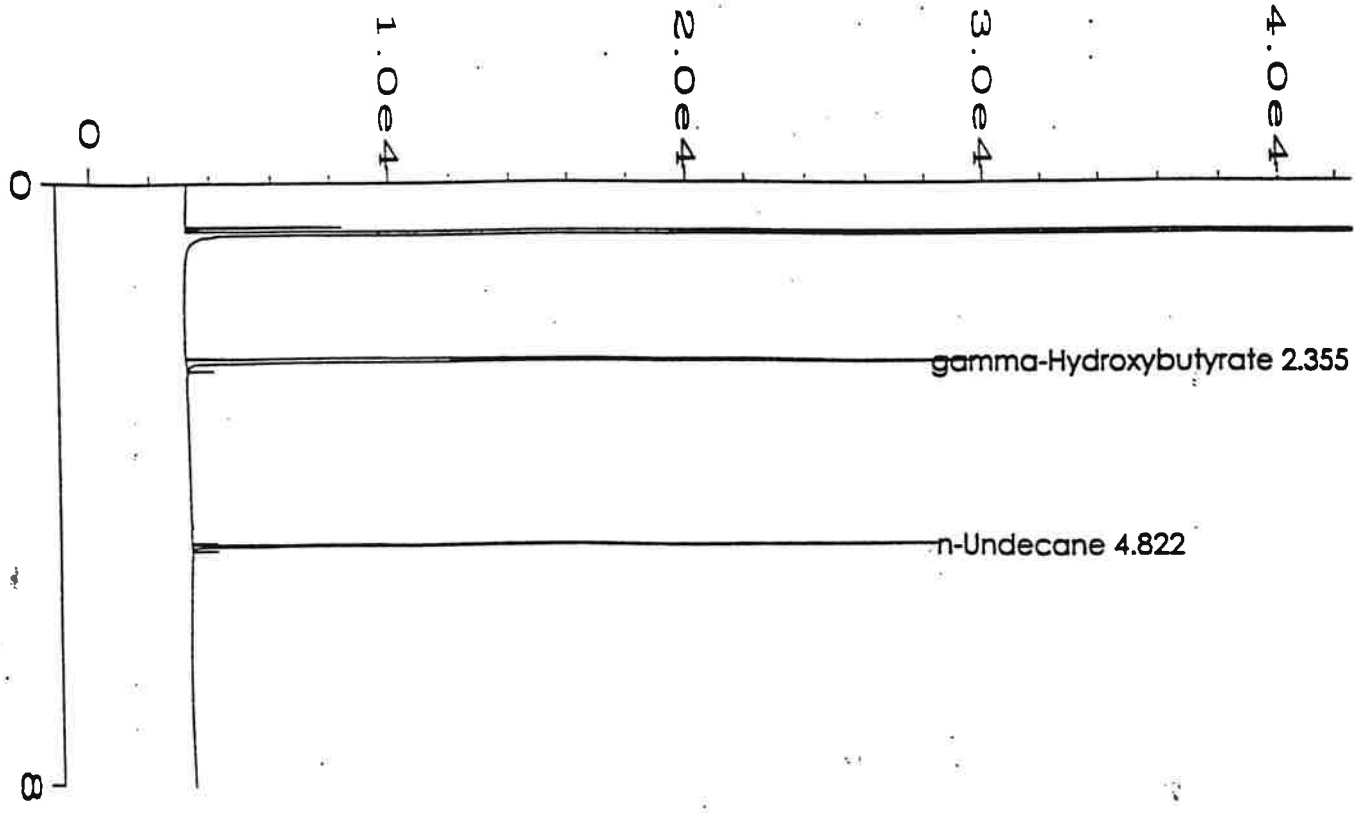
A standard GHB solution was prepared and extracted just like the sample.

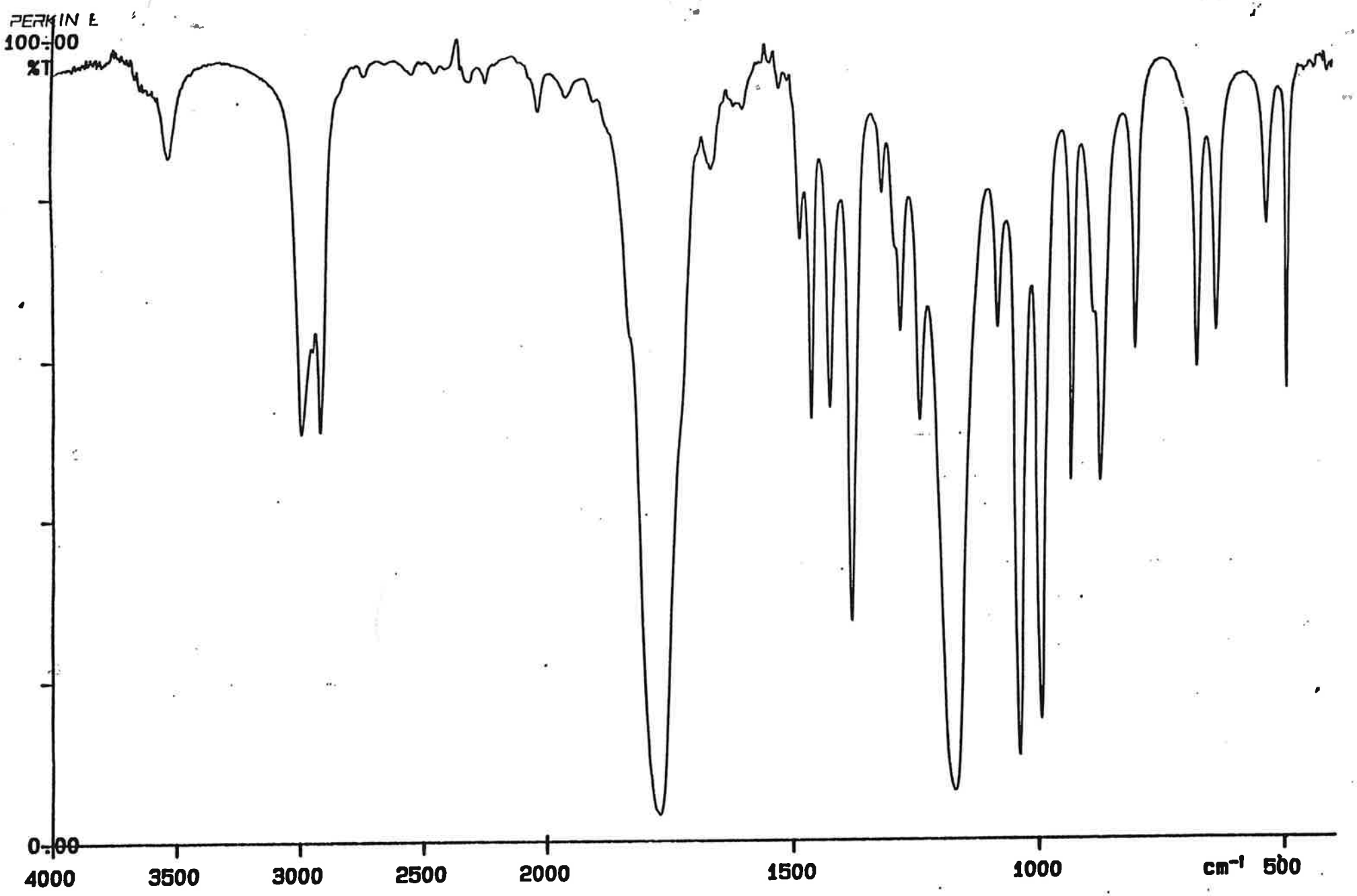
GC Conditions:

Column: HP-1 (12M length, 0.20mm i.d., 0.33um film thickness)

Oven Temperature Program:

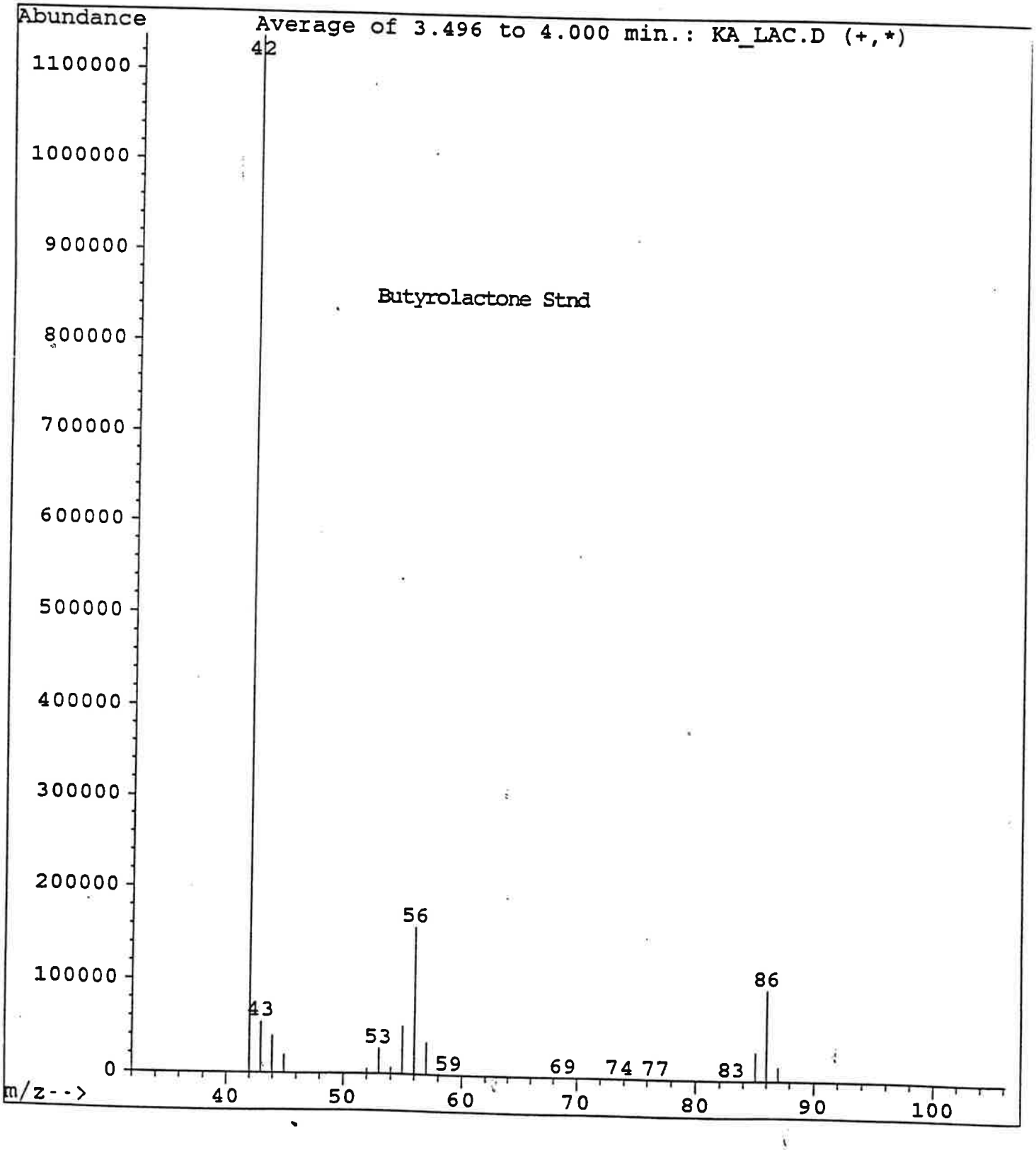
60°C initial temperature, 2.0 min initial hold; 20°C /min rate to 120°C final temperature, 3.0 min hold.

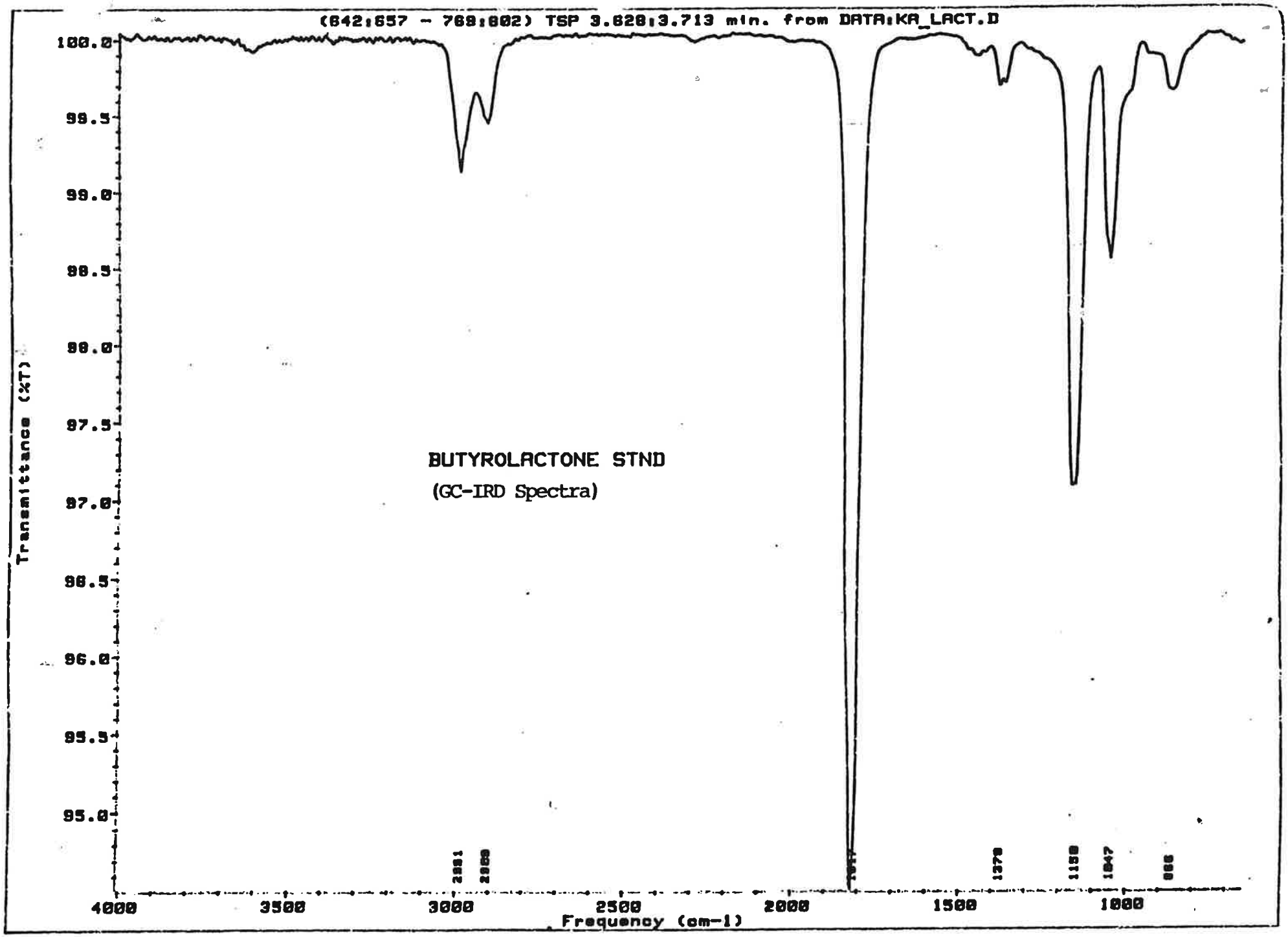




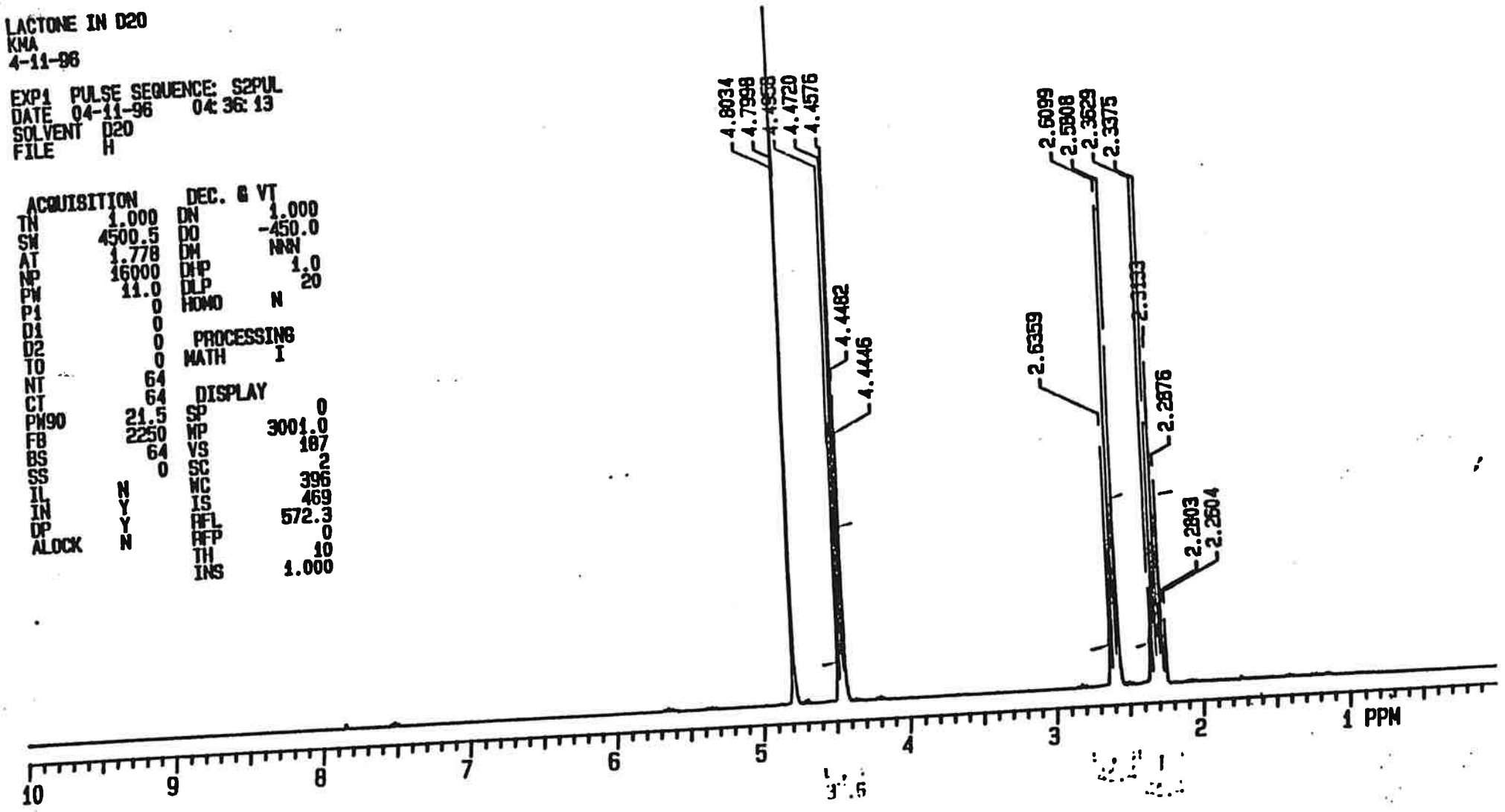
96/04/19 14:35
X: 4 scans, 4.0cm-1, flat, abex
gamma-HYDROXYBUTYROLACTONE (neat)

File : C:\HPCHEM\1\DATA\KA_LAC.D
Operator : KATHY ANDREWS
Acquired : 10 Apr 96 11:48 am using AcqMethod PRM40
Instrument : Mustang
Sample Name: gamma-Butyrolactone stnd
Misc Info : HP-5, 12.5M x 0.20mm x 0.3um
Vial Number: 1

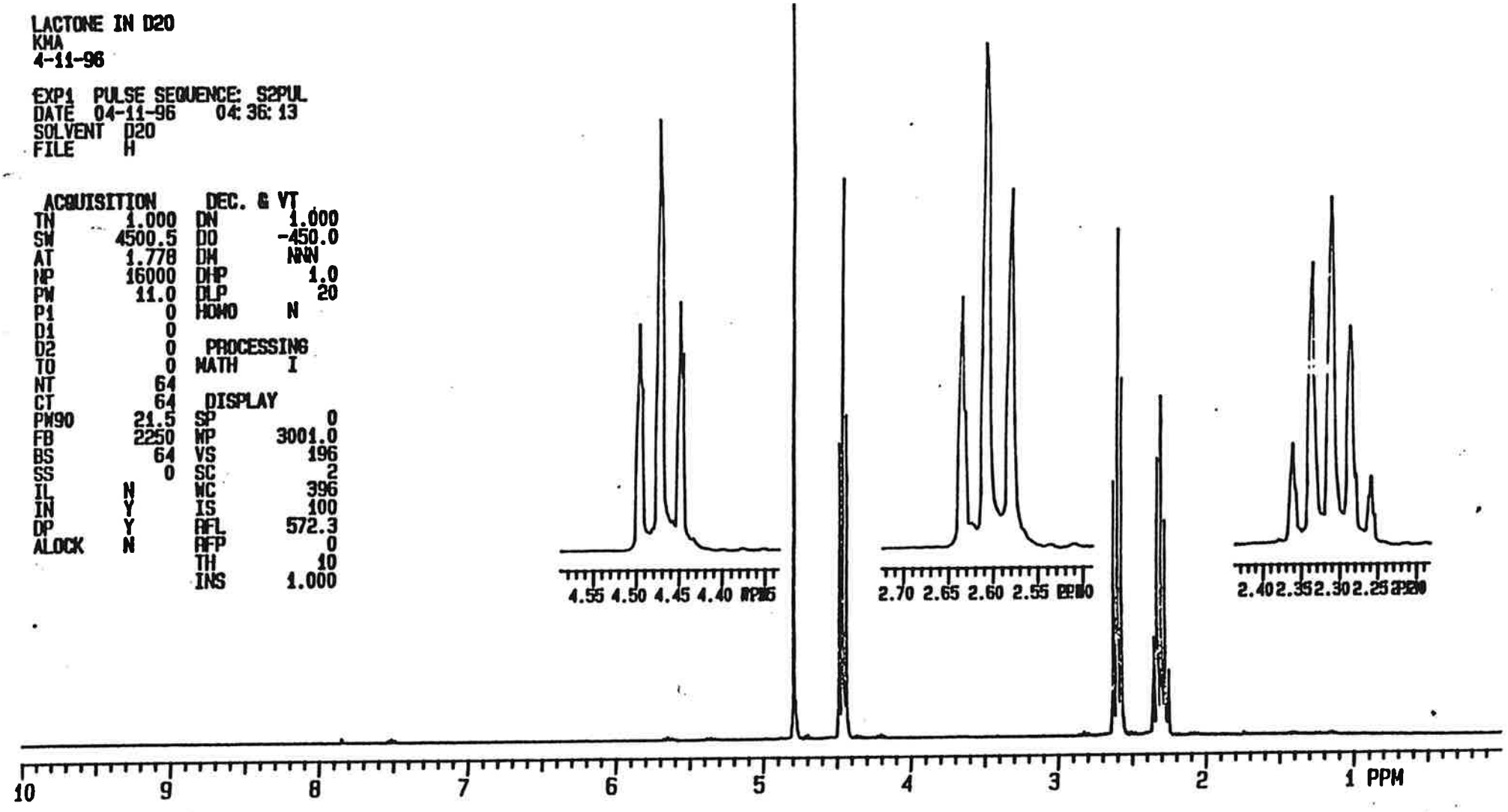



(642:657 - 768:802) TSP 3.628:3.713 min. from DATA:KA_LACT.D
100.0
99.5
99.0
98.5
98.0
97.5
97.0
96.5
96.0
95.5
95.0
Transmittance (%T)
BUTYROLACTONE STND
(GC-IRD Spectra)
2981
2939
1774
1379
1158
1047
888
4000
3500
3000
2500
2000
1500
1000
Frequency (cm-1)

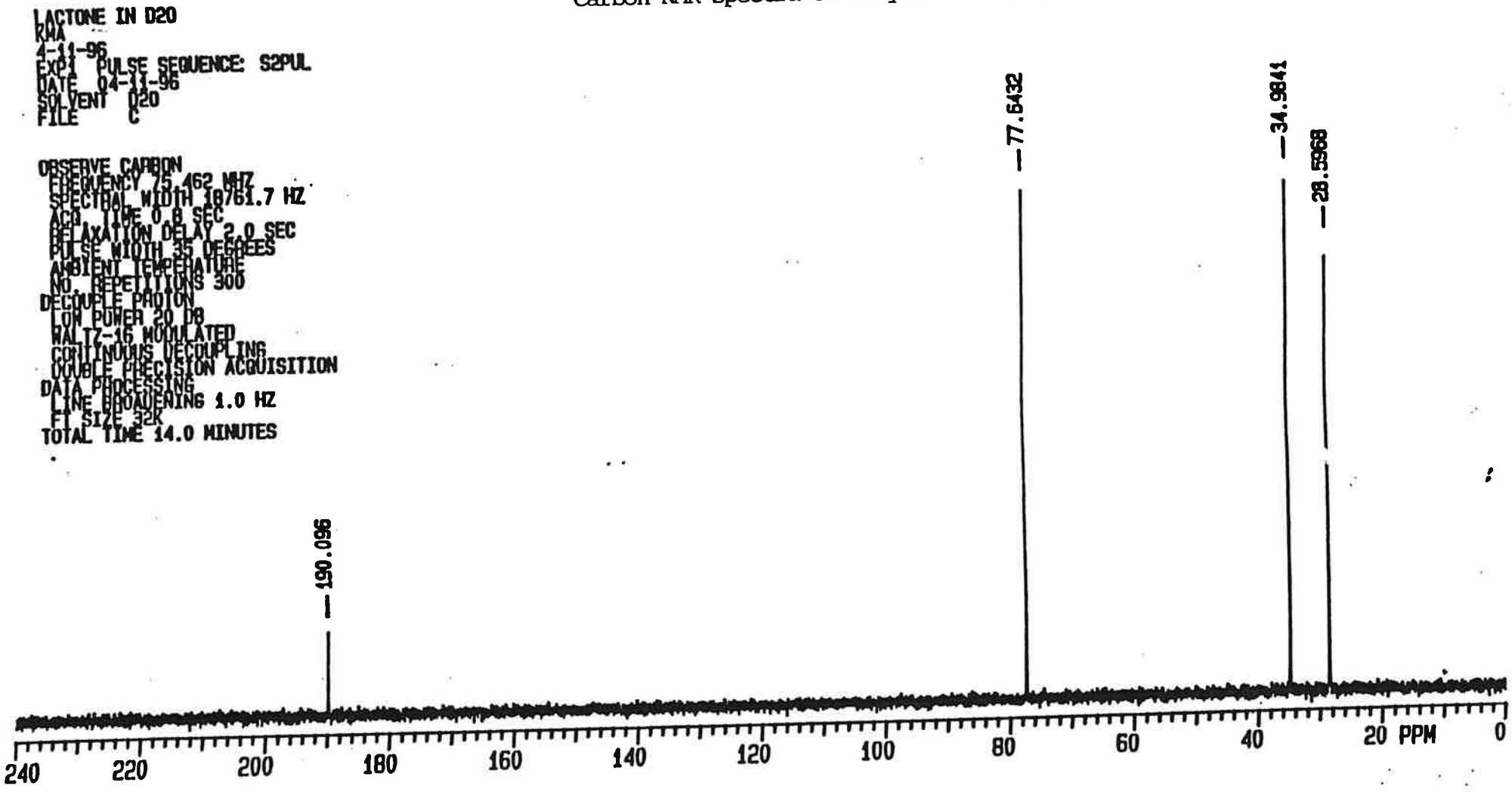
Proton NMR Spectra of Butyrolactone in D20

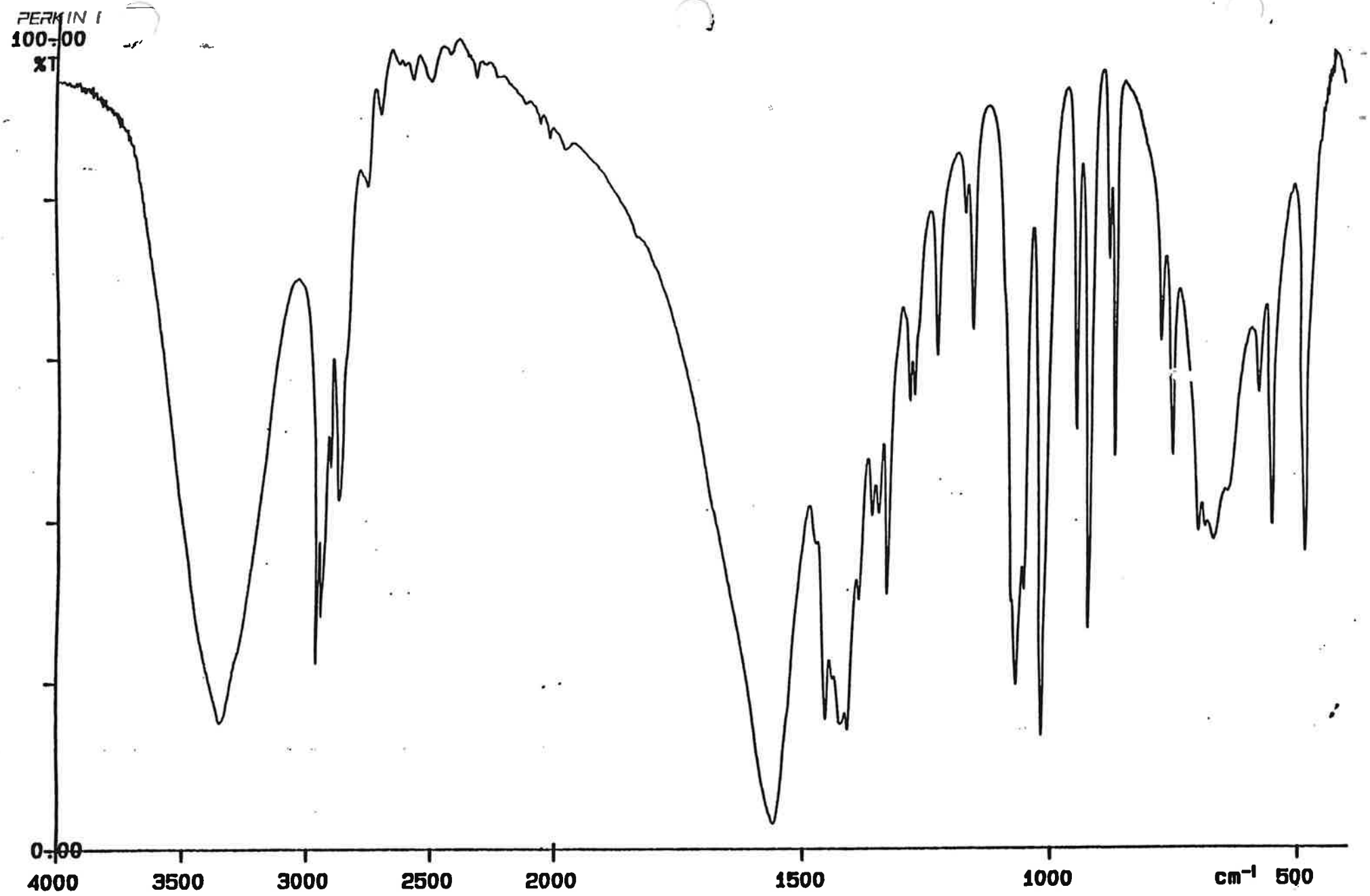


Proton NMR Spectra of Butyrolactone in D20 (expanded peaks)



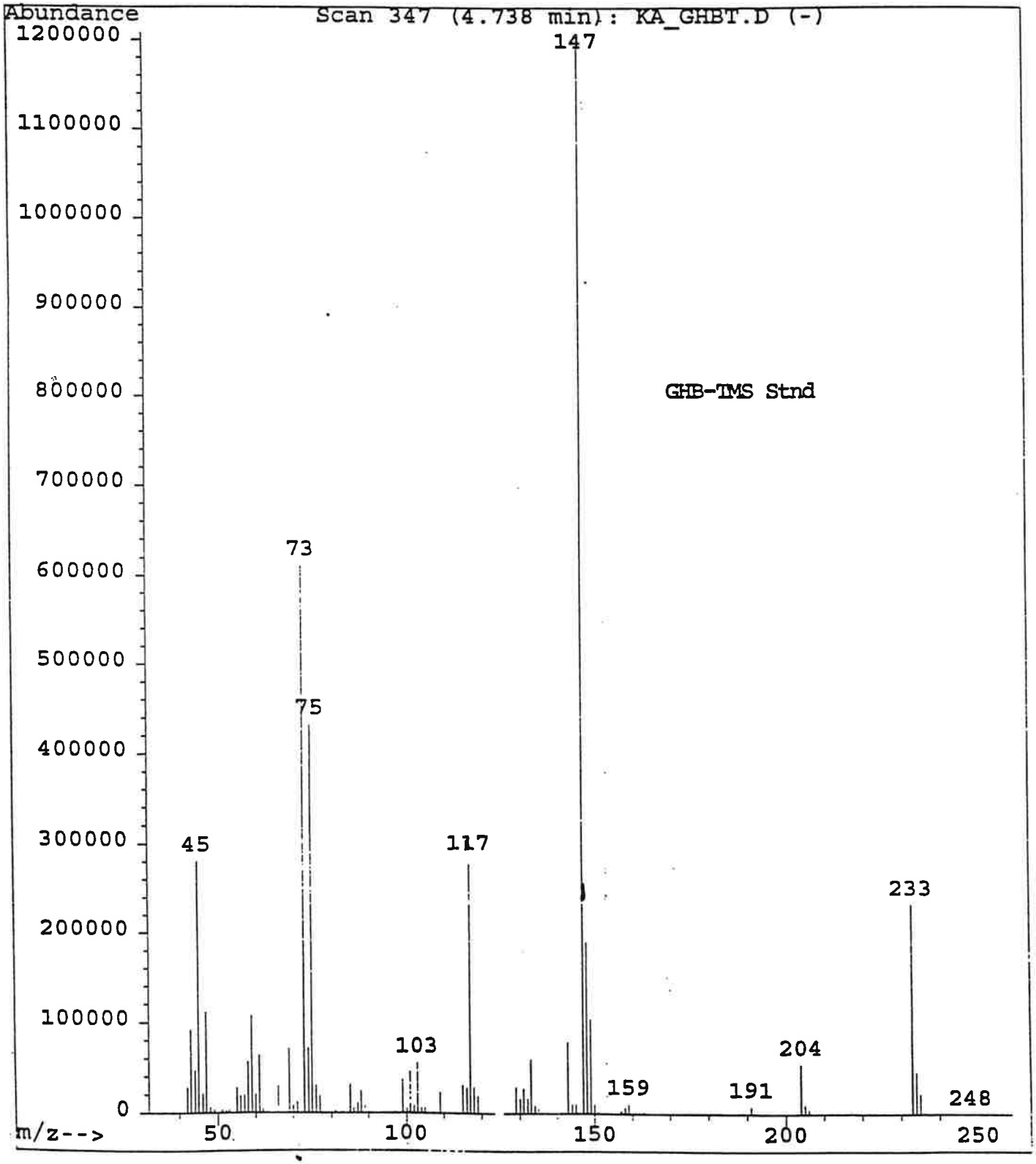
Carbon NMR Spectra of Butyrolactone in D20





96/04/19 14:23
X: 4 scans, 4.0cm-1, flat, abex
gamma-HYDROXYBUTYRATE, Na+ salt

File : C:\HPCHEM\1\DATA\KA_GHBT.D
Operator : KATHY ANDREWS
Acquired : 10 Apr 96 4:45 pm using AcqMethod PRM_40RE
Instrument : Max <
Sample Name: GHB-TMS STND
Misc Info : DB-5: 15m x 0.32mm x 0.25um [19mg:2mL]
Vial Number: 1

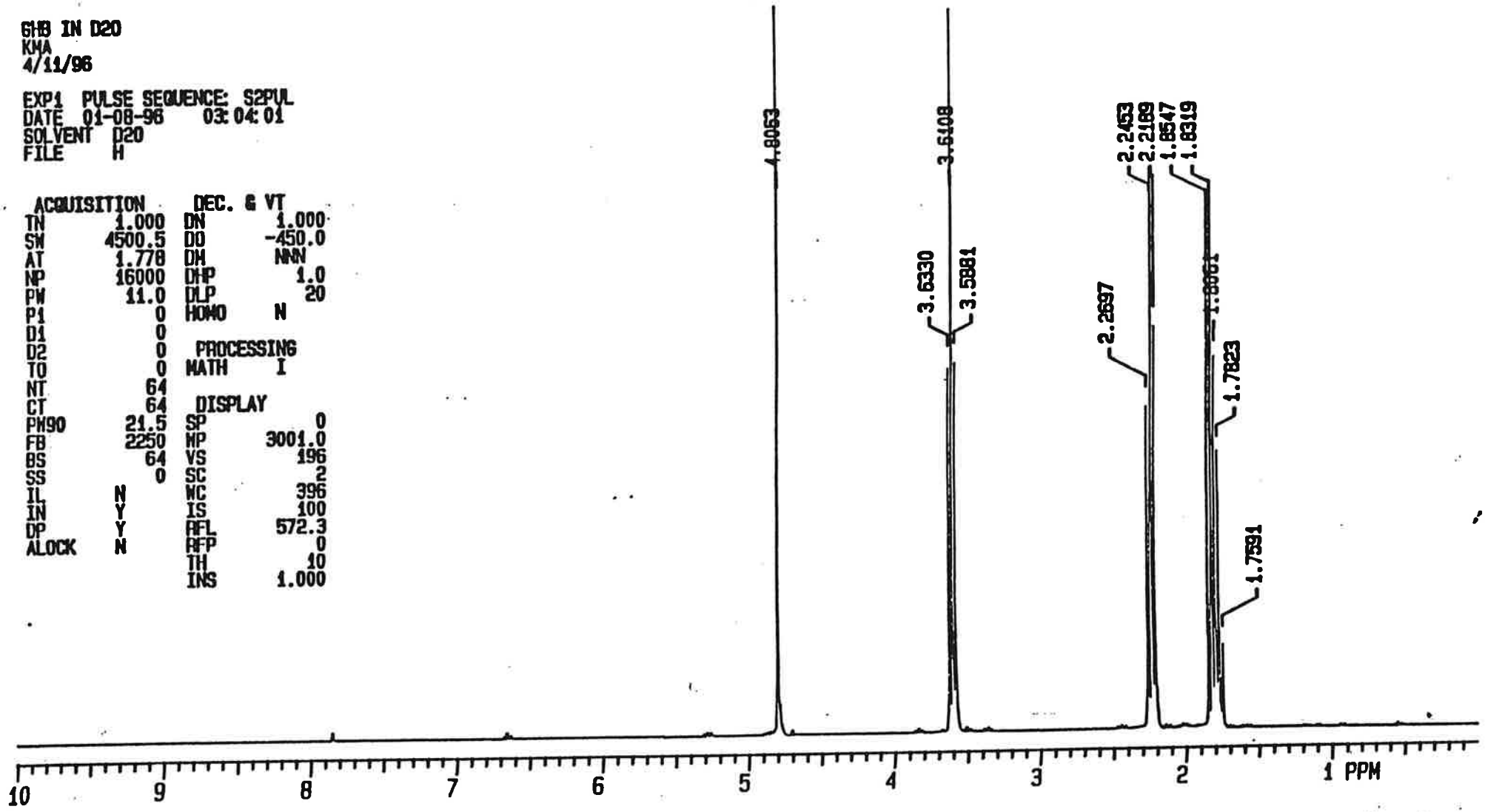


Proton NMR Spectra of GHB in D20

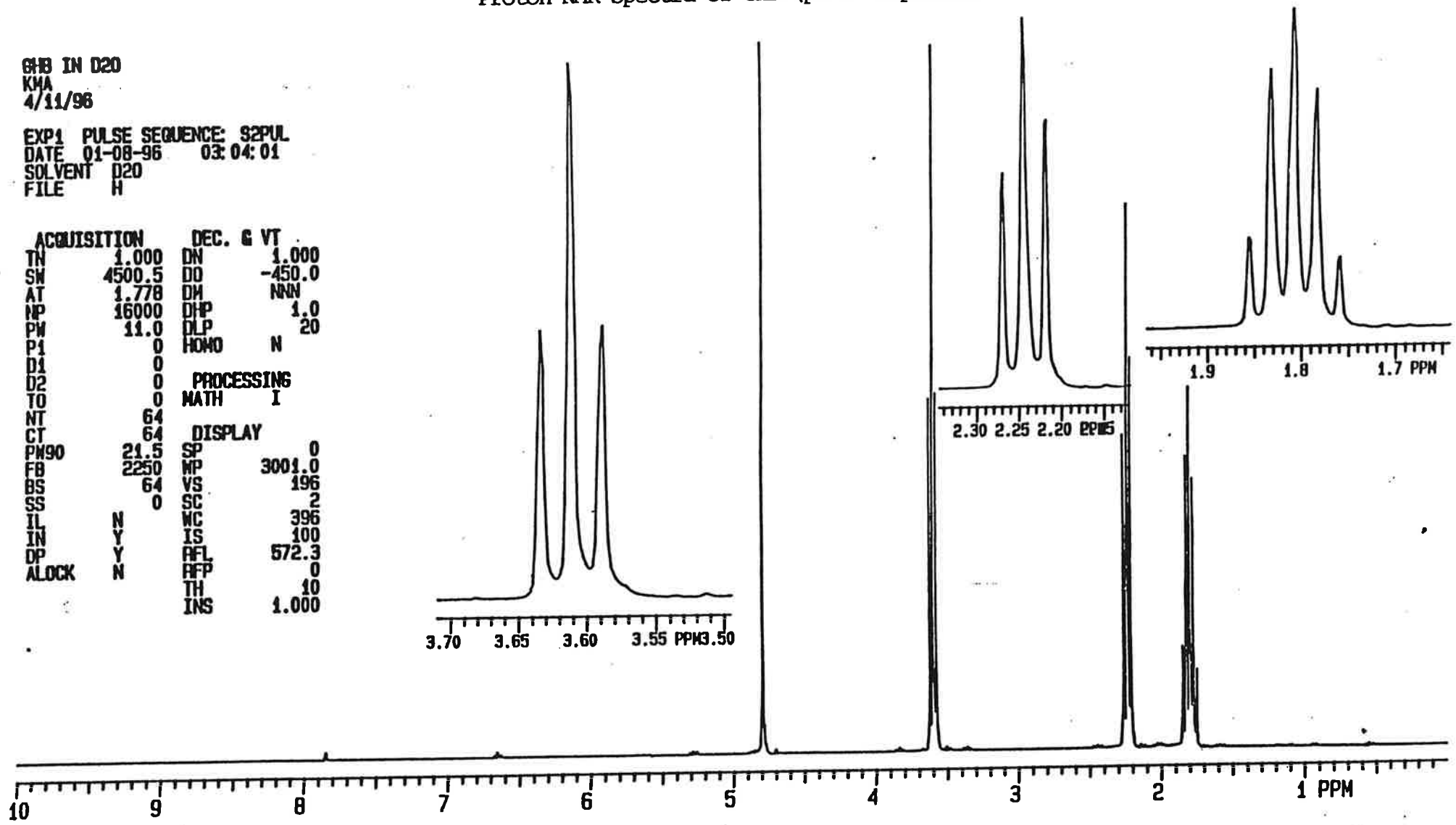
GHB IN D20
KMA
4/11/96

EXP1 PULSE SEQUENCE: S2PUL
DATE 01-08-96 03:04:01
SOLVENT D20
FILE H

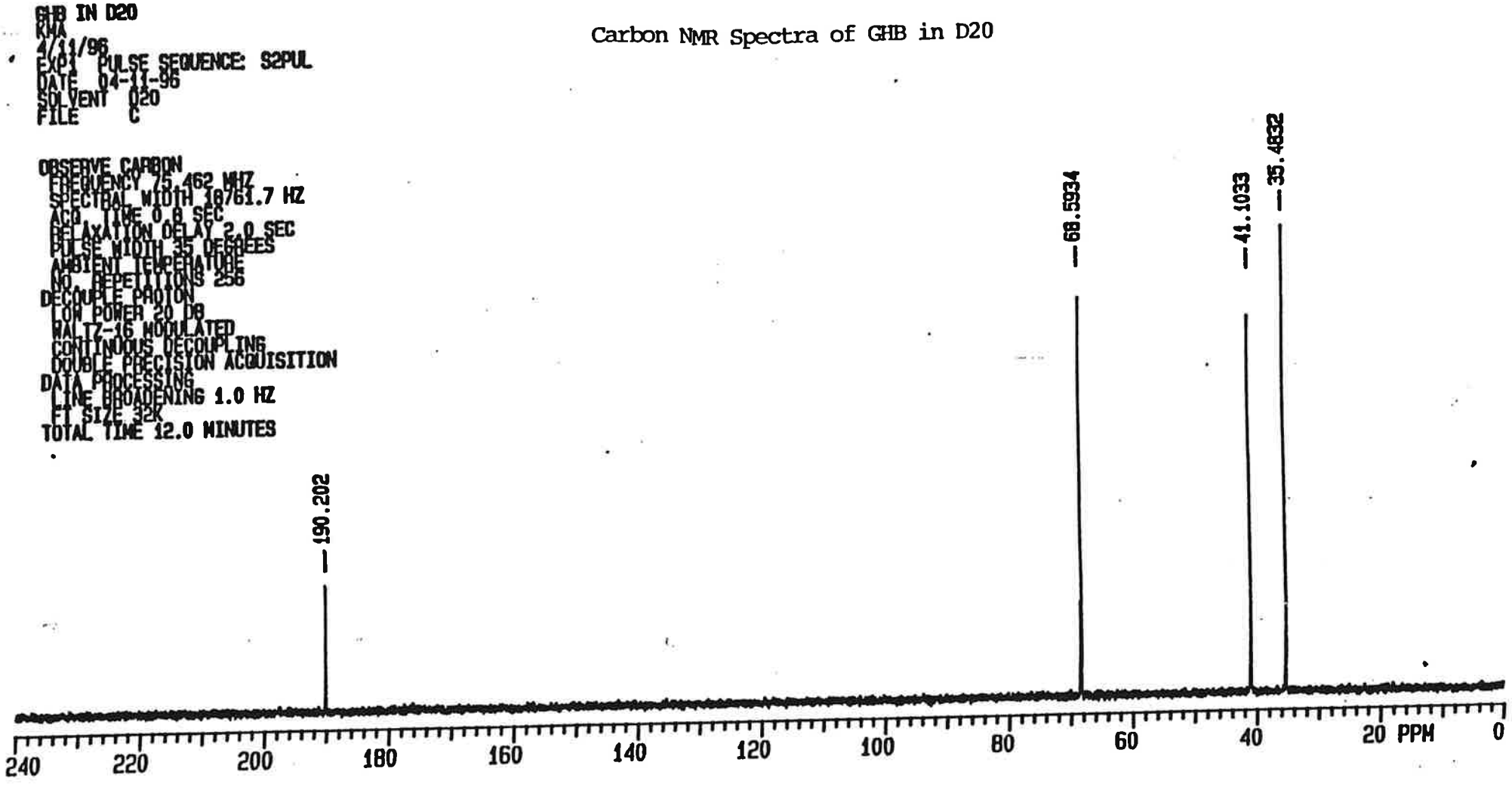
ACQUISITION		DEC. & VT	
TN	1.000	DN	1.000
SW	4500.5	DO	-450.0
AT	1.778	DH	NNN
NP	16000	DHP	1.0
PW	11.0	DLP	20
P1	0	HOMO	N
D1	0		
D2	0	PROCESSING	
TO	0	MATH	I
NT	64		
CT	64	DISPLAY	
PW90	21.5	SP	0
FB	2250	WP	3001.0
BS	64	VS	196
SS	0	SC	2
IL	N	WC	396
IN	Y	IS	100
DP	Y	RFL	572.3
ALOCK	N	RFP	0
		TH	10
		INS	1.000



Proton NMR Spectra of GHB (peaks expanded)



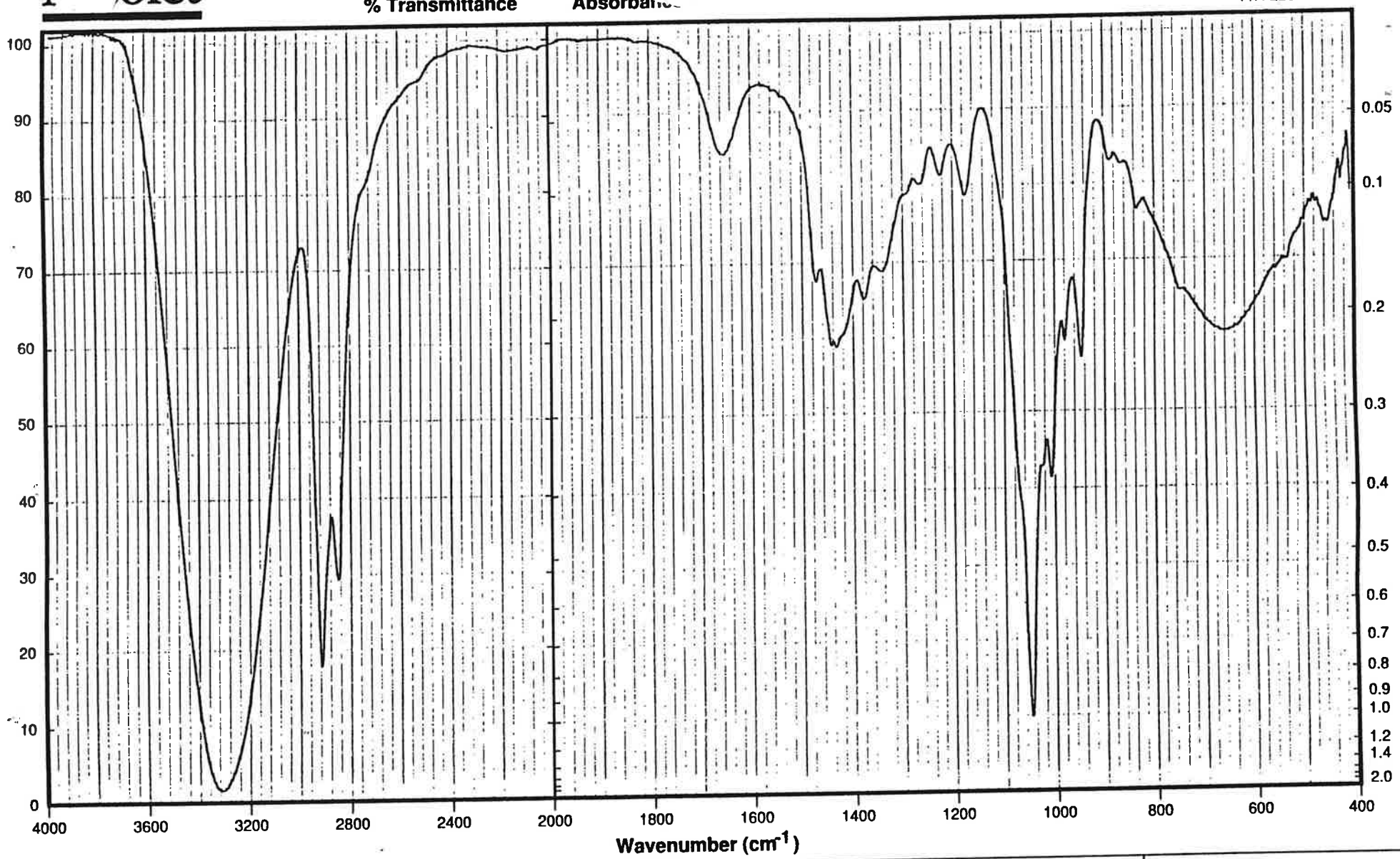
Carbon NMR Spectra of GHB in D20



Nicolet

% Transmittance Absorbance

P/N 220-706900

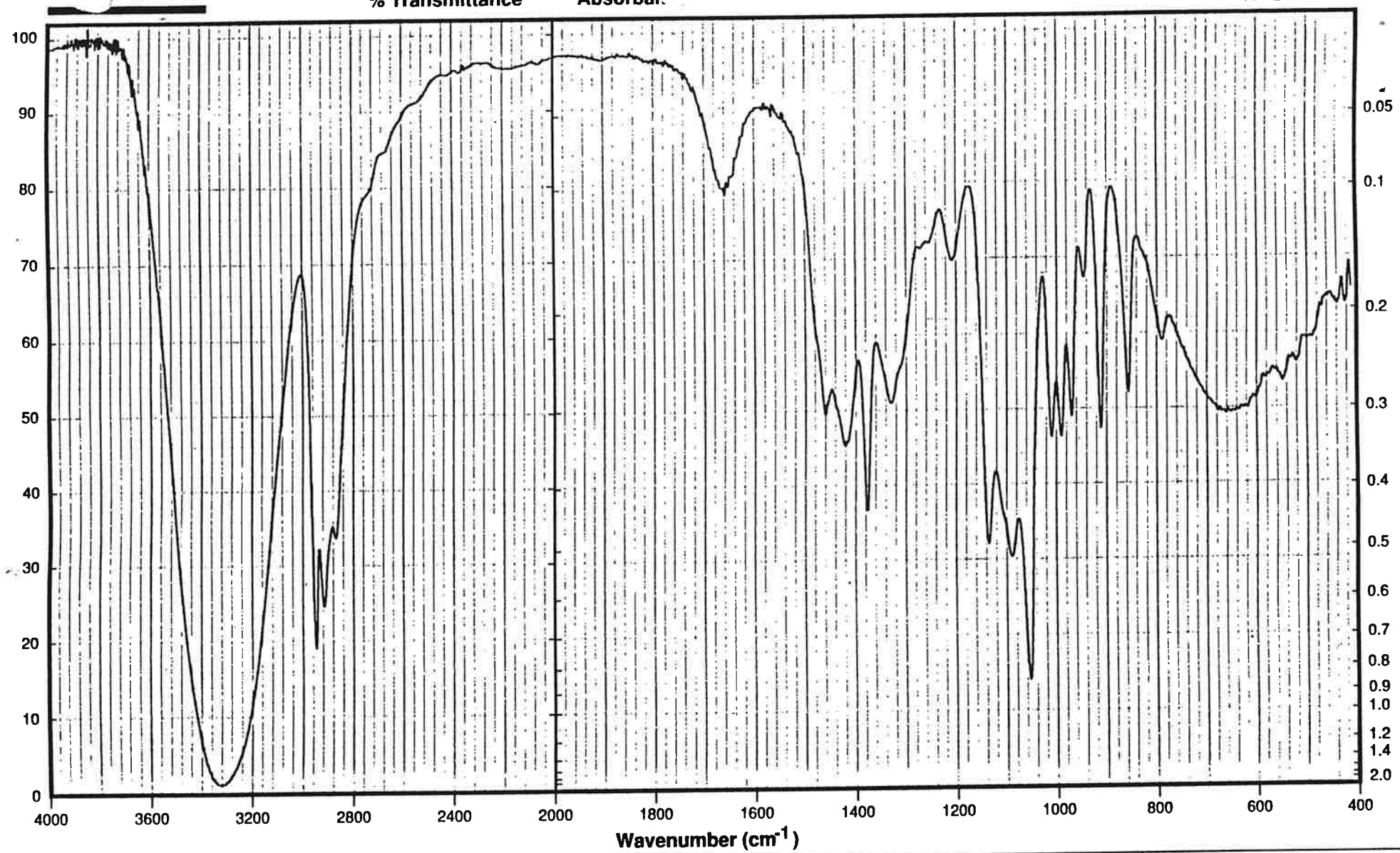


Title *1,4-Butanediol Stnd, (Neat on KCl) (Fluka# 18960)	Calibrator™ %T -5 cm-1 -25 +25 2000/400 -50 +50 4000/2000 +5	Data Processing/Comment Number of sample scans: 16 Number of background scans: 16 Resolution: 4.000 Sample gain: 1.0	Resolution
Filename Collection time: Mon Aug 31 12:04:50 1998			No. Scans
Date / Time			Operator K. Andrews DEA Western Laoratory

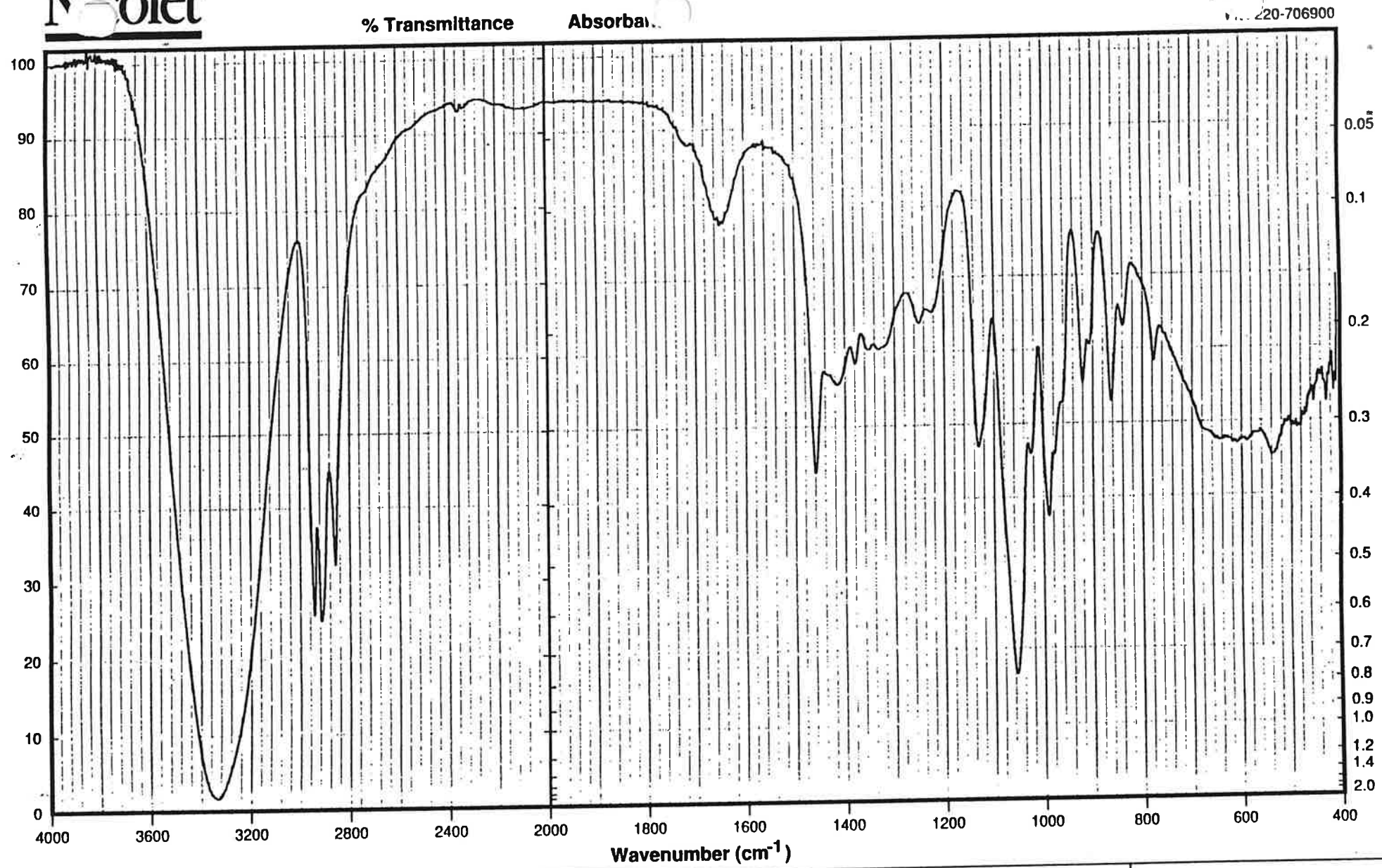
Nicolet

% Transmittance Absorba...

P/N 220-706900

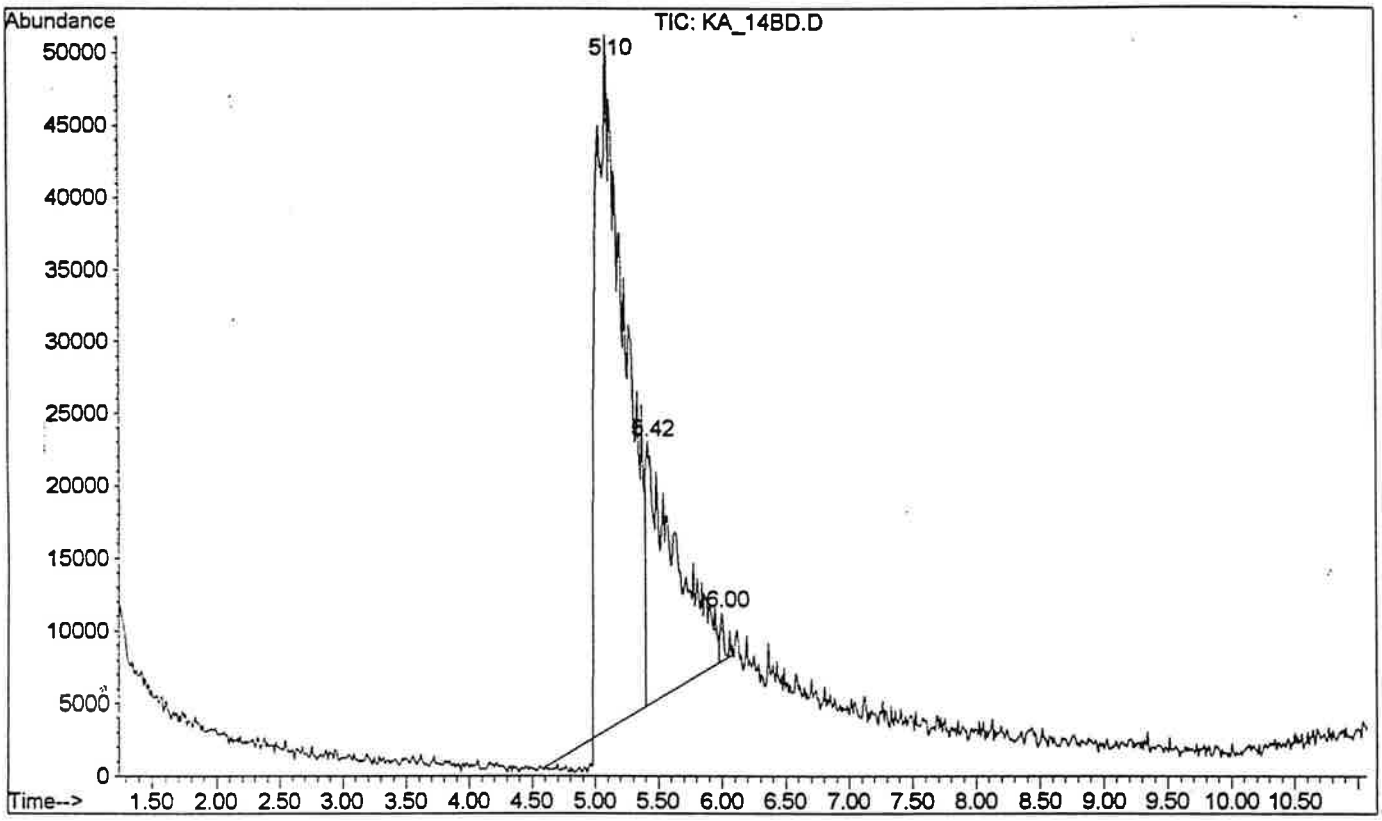


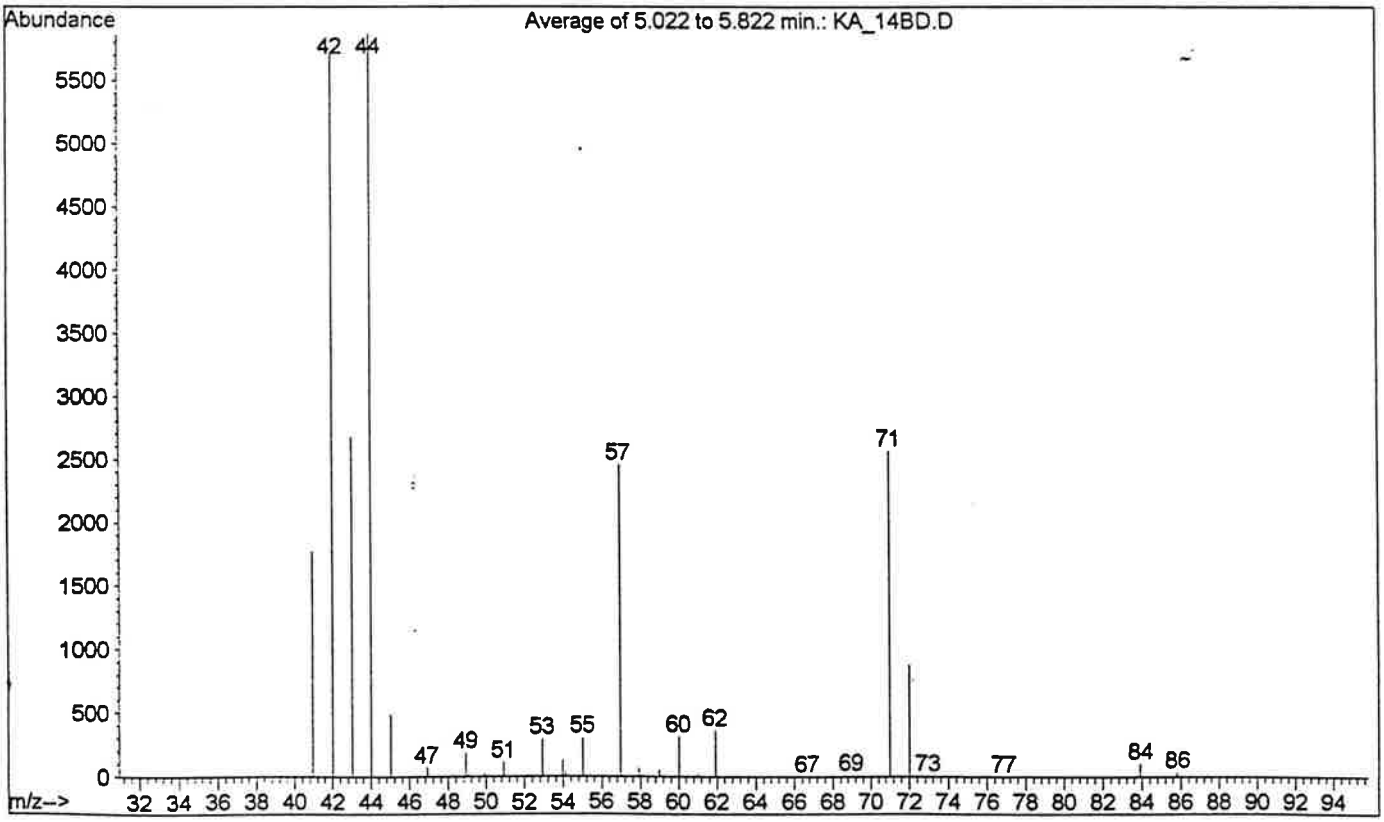
Title *1,3-Butanediol Stnd (Neat on KCl) (Fluka# 18937)	Calibrator™ %T -5 cm-1 -25 +25 2000/400 -50 +50 4000/2000 +5	Data Processing/Comment Number of sample scans: 16 Number of background scans: 16 Resolution: 4.000 Sample gain: 1.0	Resolution
Filename Collection time: Mon Aug 31 12:12:12 1998			No. Scans
Date / Time			Operator K. Andrews DEA Western Laboratory



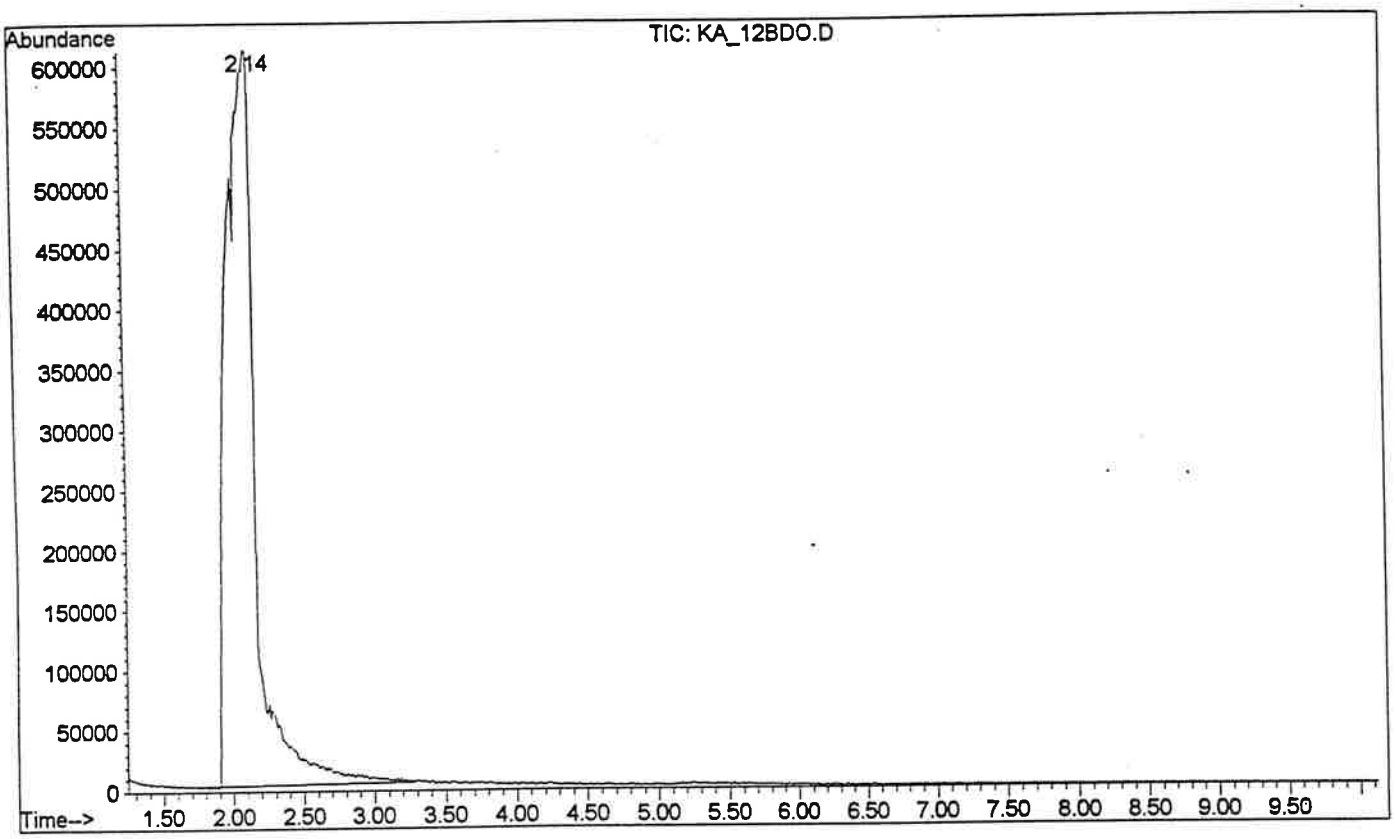
Title *1,2-Butanediol Stnd (Neat on KCl) (Fluka # 18930)	Calibrator™ %T -5 cm-1 -25 +25 2000/400 -50 +50 4000/2000 +5	Data Processing/Comment Number of sample scans: 16 Number of background scans: 16 Resolution: 4.000 Sample gain: 1.0	Resolution
Filename Collection time: Mon Aug 31 12:26:01 1998			No. Scans
Date / Time			Operator K. Andrews DEA Western Laboratory

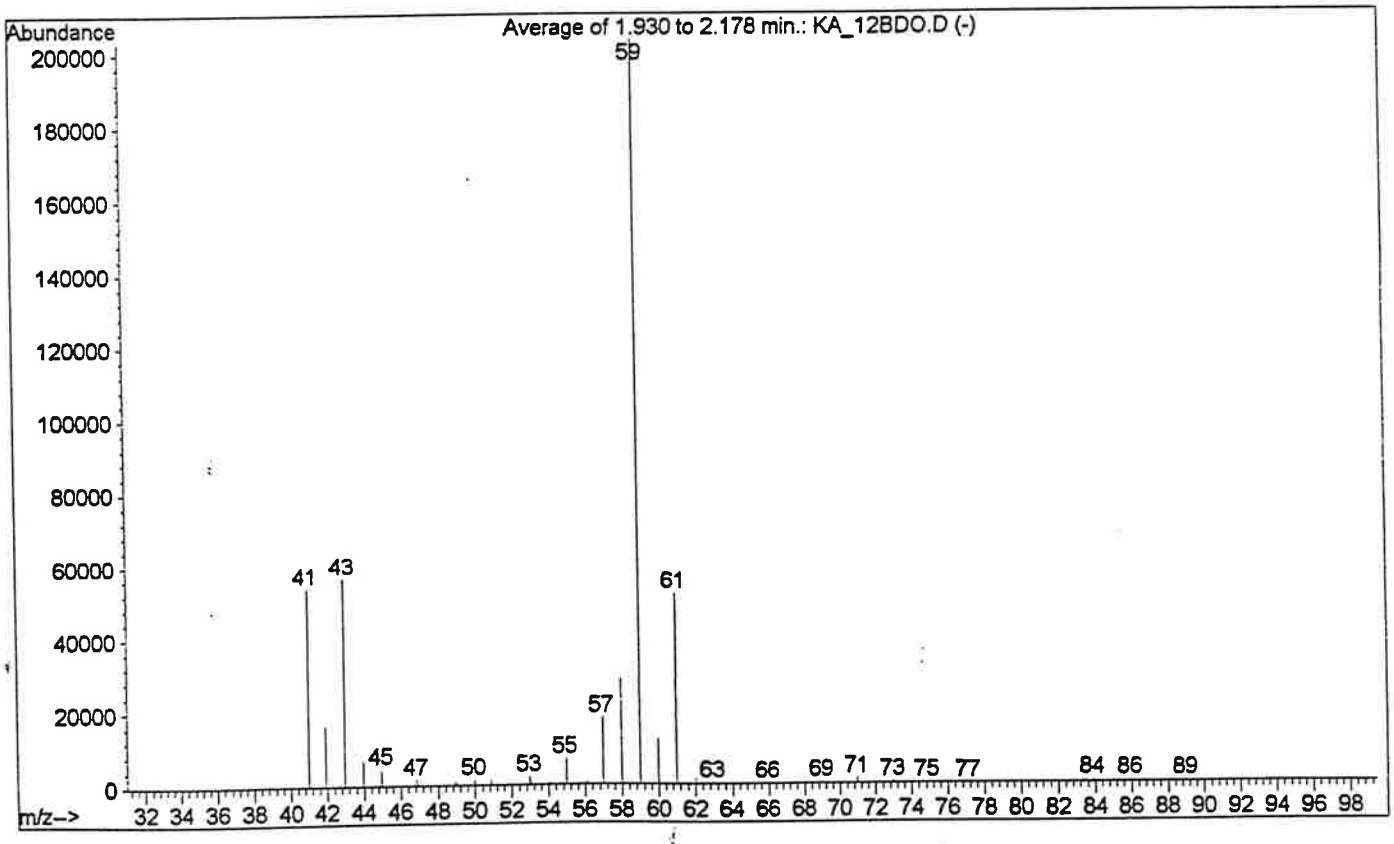
File : C:\HPCHEM\1\DATA\KA_14BD.D
Operator : KATHY ANDREWS
Acquired : 1 Sep 98 9:44 using AcqMethod GHB
Instrument : MUSTANG
Sample Name: 1,4-Butanediol Stnd, in MeCl2: MeOH
Misc Info : HP-1: 12m x 0.20mm x 0.33um
Vial Number: 1



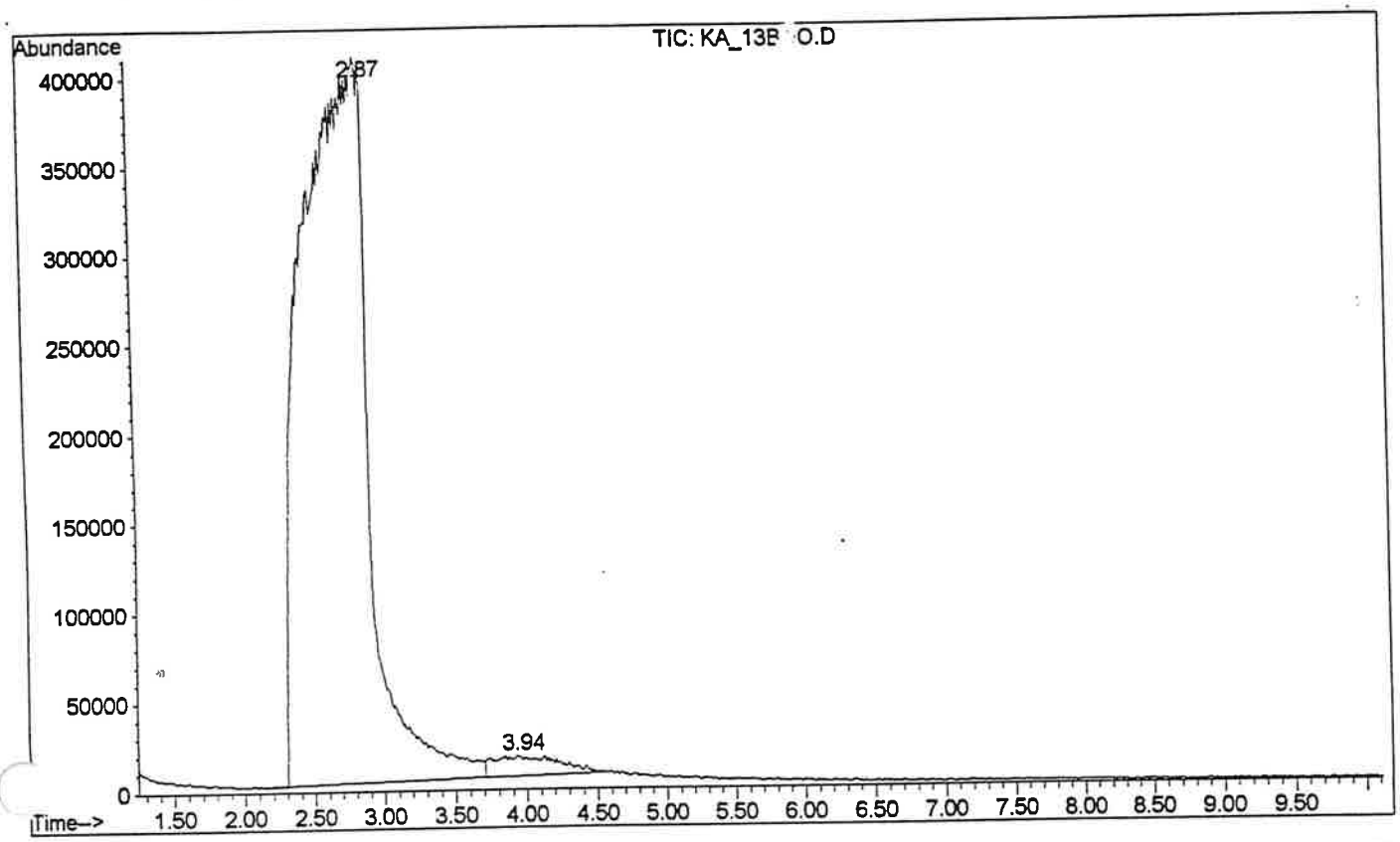


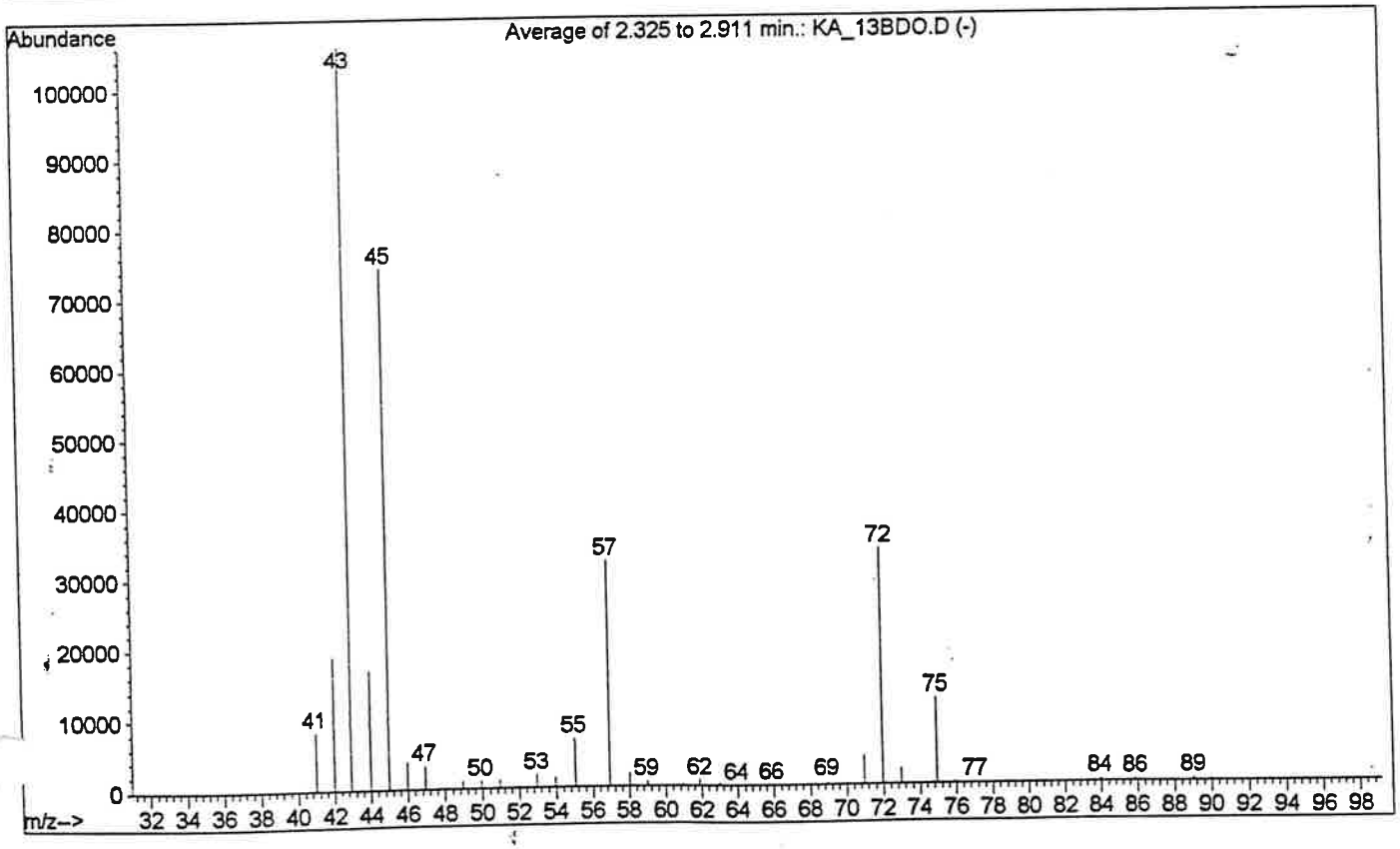
File : C:\HPCHEM\1\DATA\KA_12BDO.D
Operator : KATHY ANDREWS
Acquired : 31 Aug 98 14:39 using AcqMethod GHB
Instrument : MUSTANG
Sample Name: 1,2-Butanediol Stnd, 6mg/ml in MeCL2: MeOH
Misc Info : HP-1: 12m x 0.20mm x 0.33um
Vial Number: 1

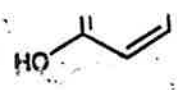




File : C:\HPCHEM\1\DATA\KA_13BDO.D
Operator : KATHY ANDREWS
Acquired : 31 Aug 98 14:05 using AcqMethod GHB
Instrument : MUSTANG
Sample Name: 1,3-Butanediol Stnd, 8mg/ml in MeCL2: MeOH
Misc Info : HP-1: 12m x 0.20mm x 0.33um
ial Number: 1







ıls (rosettes) from benzene, mp 125-126°. Sol in lcohol, chloroform, ethyl acetate.

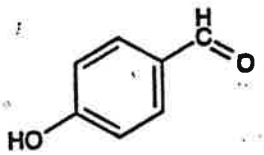
$C_9H_{14}INO$, stout prisms, mp 155°. Freely sol in l, acetone.

ide, $C_9H_{14}ClNO$, crystals from HCl, mp 171- ol ... water, alcohol. Practically insol in ether.

obromide, $C_9H_{14}BrNO$, crystals. Freely sol in lcohol, acetone. Sold as Paredrine Hydrobromide s—a 1% aq soln made isotonic with sodium chloride :served with sodium ethylmercuri thiosalicylate. d as Paredrine Hydrobromide Ophthalmic 1%, with id—a 1% aq soln made tear-isotonic with 2% boric l preserved with sodium ethylmercuri thiosalicylate.

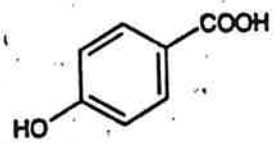
P CAT: Adrenergic (ophthalmic); mydriatic.

. *p*-Hydroxybenzaldehyde. 4-Formylphenol. C_7 nol wt 122.12. C 68.85%, H 4.95%, O 26.20%. distributed in plants in very small amounts. Ob- as a byproduct from the Reimer-Tiemann reaction :ylaldehyde from phenol: L. F. Fieser, M. Fieser, *Chemistry* (Reinhold, New York, 3rd ed., 1956) p Reimer, Tiemann, *Ber.* **9**, 824 (1876); Herzfeld, n, *Ber.* **10**, 64 (1877).



m water. Slight, agreeable, aromatic odor, mp ublimes at atmospheric pressure without decomposi- Reacts like a weak monobasic acid: Ka at 25° =)$^{-8}$. Dipole moment: 4.19. Sparingly sol in cold 1.38 g/100 ml H_2O at 30.5°; more sol in hot water. sol in alc, ether. Slightly sol in benzene: 3.68 g/100 I_6 at 65°.

7. *p*-Hydroxybenzoic Acid. $C_7H_6O_3$; mol wt C 60.87%, H 4.38%, O 34.75%. Prepn from *p*- phenol: Gilinan, Arntzen, *J. Am. Chem. Soc.* **69**, 1947); from *p*-hydroxybenzaldehyde: Pearl, *J. Org.* **12**, 85 (1947); from phenol + potassium ethyl car- : Jones, *Chem. & Ind. (London)* **1958**, 228; from um phenolate + CO_2: Brit. pat. 942,418 (1963 to a). A metabolic product of *Penicillium patulum*: baum, Bassett, *Biochim. Biophys. Acta* **28**, 21 (1958).



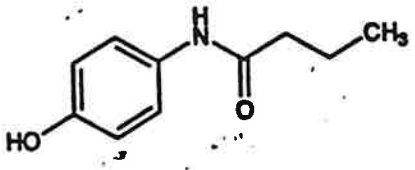
stals, mp 213-214°. d 1.46. Soluble in about 125 ... freely in alcohol, slightly in chloroform; sol in

Leaflets from butanol, mp 110°. Freely sol in water, alcohol; also sol in benzene. Practically insol in anhydr ether.

Sodium salt, *Phos, Phoselit, Phosilite.* Very freely sol in water. The pH is on the acid side, but aq solns are suitable for injection.

THERAP CAT: Sodium salt as nutrient.

4859. 4'-Hydroxybutyranilide. *N-(4-Hydroxyphenyl)-butanamide; p*-hydroxybutyranilide; *N*-butyroyl-*p*-aminophenol; Suconox-4. $C_{10}H_{13}NO_2$; mol wt 179.22. C 67.02%, H 7.31%, N 7.82%, O 17.85%. Prepn: Kuhn *et al.*, *Z. Physiol. Chem.* **247**, 197 (1937); Fierz-David, Kuster, *Helv. Chim. Acta* **22**, 82 (1939); Rohmann, Friedrich, *Arch. Pharm.* **278**, 456 (1940).



Needles from water, mp 139-140°. Slightly sol in cold water, more sol in hot water, sol in alcohol.

USE: As antioxidant: Young, Cottle, U.S. pat. 2,654,722 (1953 to Standard Oil).

4860. γ-Hydroxybutyrate. *4-Hydroxybutanoic acid;* γ-hydroxybutyric acid; 4-hydroxybutyrate; GHB. $C_4H_8O_3$; mol wt 104.11. C 46.15%, H 7.75%, O 46.11%. $HOCH_2CH_2CH_2COOH$. Naturally occurring metabolite of GABA; thought to function as a neurotransmitter or neuromodulator. The highest conc in humans is found in fetal cerebellum and adult hypothalamus. Prepn of salts: A. Saytzeff, *Ann.* **171**, 258 (1874); C. S. Marvel, E. R. Birkhimer, *J. Am. Chem. Soc.* **51**, 260 (1929). Clinical pharmacokinetics: S. D. Ferrara *et al.*, *Brit. J. Clin. Pharmacol.* **34**, 231 (1992). GC/MS determn in biological fluids: K. M. Gibson *et al.*, *Biomed. Environ. Mass Spectrom.* **19**, 89 (1990). Acute toxicity: B. Bruguerolle *et al.*, *Thérapie* **32**, 375 (1977). Clinical studies: L. Scrima *et al.*, *Sleep* **13**, 479 (1990); L. Gallimberti *et al.*, *Neuropsychopharmacology* **9**, 77 (1993). Review of biochemistry and pharmacology: G. Tunnicliff, *Gen. Pharmacol.* **23**, 1027-1034 (1992). Review in treatment of alcoholism: G. Biggio *et al.*, in *Adv. Biochem. Psychopharmacol.* vol. 47 (Raven Press, New York, 1992) pp 281-288. *Review:* M. Mamelak, *Neurosci. Biobehav. Rev.* **13**, 187-198 (1989). Brief overview of public health issues: *J. Am. Med. Assoc.* **265**, 447-448 (1991).

Sodium salt, $C_4H_7NaO_3$, *γ-OH, sodium oxybate, sodium* γ-*oxybutyrate, Somatomax PM, Wy-3478, NSC-84223, Somsanit, Gamma-OH.* Crystals from alcohol. LD_{50} in male, female rats (mg/kg): 2,000, 1,650 i.p. (Bruguerolle).

THERAP CAT: Anesthetic (intravenous). In treatment of narcolepsy; in treatment of alcoholism.

4861. β-Hydroxybutyric Acid. *3-Hydroxybutanoic acid.* $C_4H_8O_3$; mol wt 104.11. C 46.15%, H 7.75%, O 46.11%. $CH_3CHOHCH_2COOH$.

d-Form, prepd by the action of *Aspergillus griseus* on the *dl*-form: McKenzie, Harden, *J. Chem. Soc.* **83**, 430 (1903). Crystals. $[\alpha]_D^{20}$ +24.3° (c = 2.226). Sol in water, alcohol, ether.

l-Form, found in the urine of diabetics (as much as 30 g per day). Isoln: Fischer, Scheibler, *Ber.* **42**, 1221 (1909);

The Headaches of Analyzing a GHB Sample

Presented By Kathleen Andrews
DEA Forensic Chemist
San Francisco, CA

GHB: Not Just a Date Rape Drug

The Abusers

I. Bodybuilders
- sedative
- euphoria high
- steroid enhancer (theory)

II. MDMA/LSD (Psychedelic) Crowd
- rave parties
- abused with LSD, Ketamine, etc.

More GHB Abusers

III. Methamphetamine Crowd
- relief from stimulant effects
- alternative profit maker

IV. Lone GHBers
- no other drug abuse
- produce/sell strictly for profit or personal use

The GHB Sample

Solid

Powder

- waxy or soap-like appearance
- over time, powder will absorb moisture and become slush/sludge
- most likely will not observe in tablet form
- sold in bottles or gel caps

very hygroscopic

The GHB Sample

Liquid

GHB is most commonly observed dissolved in a liquid

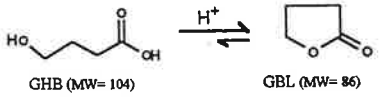
* Water - food coloring, flavoring added
* Alcohol
* Juices, Kool-aid, carbonated drinks, etc.

Conveniently disguised in water bottles!

Seized GHB Samples

1. Florida (1996) -thick, red syrupy liquid with cinammon-like odor
2. Houston, TX (1996) -clear greenish liquid with sweet odor
3. Rhode Island (1995) -GHB dissolved in "Coke" filled bottles and stored in refrig.

The Analysis of GHB



GHB (MW= 104) GBL (MW= 86)

pH Dependent!

- Presence of acid will cause laconization.
  * higher the pH, less lactone observed
- Lactone + base produces GHB.

Suggested Analysis

I. Screens (liquid samples)

a. Acid extraction into ether or chloroform,
- This will convert any GHB into the lactone which will be soluble in the organic layer.

b. Followed by lactone i.d. on FTIR or GC-MS at high injection port temps.
- GHB or lactone originally present in sample.

Post-screen Analysis

Don't alter pH of original sample!

I. Lactone I.D.:

Extraction into ether or chloroform, then FTIR or GC-MS.

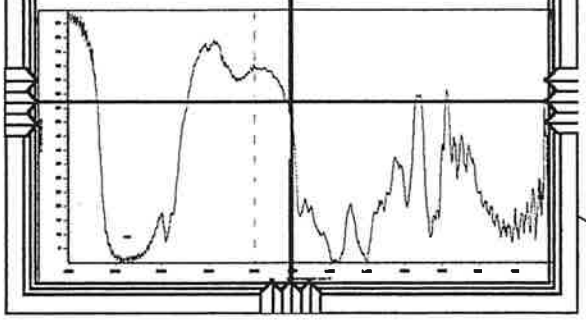
II. GHB I.D.:

Air dry liquid down to ppt., followed by
1. FTIR
2. NMR in D_2O
3. Silylation or Alkylation GC work.

Direct FTIR

1. Chloroform Ext.
   - identifying butyrolactone
2. Air dry liquid samples over low heat until sludge or paste formed.
   - Direct FTIR
   - Alternative: Raman Spectroscopy

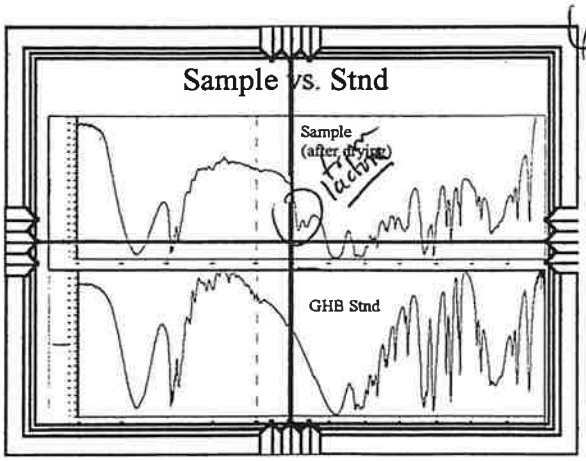
FTIR of Street Sample



hydrated GHB

Same sample after drying in oven ~ 20min

Sample vs. Stnd



GC-MS Analysis

GHB does not chromatograph!

- GHB converts to lactone in injection ports at high temps. over 200°C.
  - conversion not observed at 100°C
  - requires derivatization
    - silylation
    - alkylation

must derivatize to do MS.

Silylation of GHB

FDA Laboratory Information Bulletin No. 3532

20 mg of sample
excess BSTFA(500ul)
excess TMCS (500ul)
2.0 ml acetonitrile
2.0 ml of n-C_{11} as internal stnd

Heated at 80°C for 30 minutes prior to analysis.

absorbs H_2O over time must be kept fresh. because H_2O will interfere w/ silylation

must be dry sample

Alternative Silylation Method

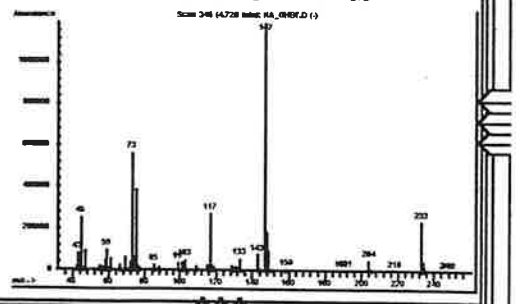
Hexamethyldisilazane (HMDS)

1. To 10mg of sample, add 1ml of HMDS and 1ml of pyridine.
2. Heat mixture to boiling for 30 minutes.
3. Analyze on GC or GC-MS

Ref: Alltech GC Reagent Instructions, Cat. No. 18003

Mass Spectra of GHB-TMS

$(CH_3)_3$-Si-O-$CH_2CH_2CH_2$-C(=O)-O-Si-$(CH_3)_3$



HPLC Analysis

Methodology developed by Mesmer and Satzger of the FDA.

"Determination of Gamma-Hydroxybutyrate (GHB) and Gamma-Butyrolactone (GBL) by HPLC/UV-VIS Spectrophotometry and HPLC/Thermospray Mass Spectrometry"

J. Forensic Sci 1998; 43(3): 489-492

probably the last was to do analysis

NMR Analysis

Samples evaporated to near dryness, then analyzed in D_2O.

I. Little or no sample decomposition
II. Does not require derivatization
III. GHB and GBL detected

takes about 10 min

Color Tests for GHB

I. Chromic Acid/ Sulfuric Acid

Reagent: 5M H_2SO_4 saturated with CrO_3

- changes from orange to green-blue color
- does not react with GBL

Ref: R.L. Shriner, R.C. Fuson, D.Y.Curtin, and T.C.Morrill, The Systemic Identification of Organic Compounds, 6th ed. (Wiley, Toronto, 1980), pages 149-150.

The Chromic Acid/Sulfuric Acid Color Test

CAUTION! **This test will give many false positives**

Reagent will oxidize OH group on CH, CH_2, and CH_3

* positive rxn with ethanol and any 1° and 2° alcohol
* positive rxn with sugars such as sucrose, lactose, etc.

EtOH will give pos. rxn with virtually

Color Tests for GHB

II. Aqueous Ferric Chloride

Reagent: 5% aq. ferric chloride

- will change from yellow to red-brown ppt.
- does not react with GBL or 1,4-Butanediol

CAUTION! **This test will give false positives**

-Ferric chloride will produce a red-brown ppt in presence of any base.

hits w bases

Performance of Field Test Kit

Marketed by Drug Identification, Inc. (Florida)

- a positive test will yield a red-brown ppt
- supposedly will test for GHB
- will not react with GBL

Caution! This test will give positive results in the presence of any alkaline solution.

Ferric chloride reagent

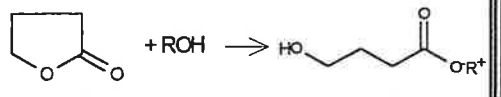
The GHB Field Test Evaluation

	Untainted	with GHB (300 mg/ml)
I. Coca-Cola	no rxn (pH= 3)	positive (pH= 7)
II. Orange Soda	no rxn (pH= 3-4)	positive (pH= 7)
III. Orange Juice	no rxn (pH= 4)	positive (pH= 7)
IV. Ethanol (200pf)	no rxn (pH= 6-7)	positive (pH= 9)
V. Kool-aid (Cherry)	no rxn (pH= acid)	positive (pH= neutral)
VI. 5% aq. NaOH	strong positive reaction	-------

Coca-Cola + thin soln. will not give pos.

Street samples 200mg - 800mg/ml GHB neutralizes soln. when added. w/o pH.

Clandestine Manufacture



GBL pH= 3 - 4 (acidic)

GHB pH= 6-14 (alkaline)

muriatic acid (HCl) added to adjust pH to safe level, (6 - 8).

can obtain yield of 70% of the GBL weight, depending on pH

legitimate uses for GBL : wood cleaner, in paint removers, textiles

must be careful

Making GHB

I. Liquid GHB:

The butyrolactone is added to aqueous hydroxide.

- "ready-to serve" product under an hour.

II. Powdered GHB:

The butyrolactone is added to alcoholic hydroxide.

- ppt forms over 1-2 hours.

HEAT IS NOT NECESSARY

gives powder GHB
ppt. out

THE GHB Clandestine Lab

The Average Level to Sophisticated Labs may have:

1. pH papers/meters
2. inorganic hydroxide, perhaps pharmaceutical grade
3. Temperature controlled environments
4. Dehumidifiers
5. Alcohol and/or acetone for drying and purifying GHB
6. Drying ovens of some sort

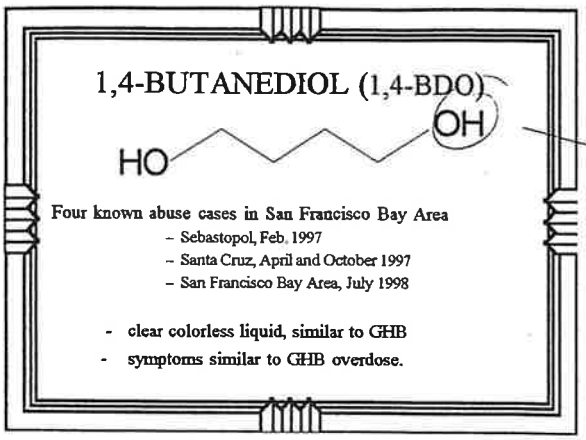
can use KOH or NaOH may have will get gary salt forms

New Year's Eve, 1996

31 young people become violently ill at a rave party in Los Angeles after ingesting a new "herbal drink" labeled as "FX".

- at least 4 victims stopped breathing and one teenager had a heartattack.
- "FX" label listed kava-kava as primary ingredient; however, tests revealed product contained caffeine and a drug called **1,4-butanediol.**

17 yrs. old



analog of GHB

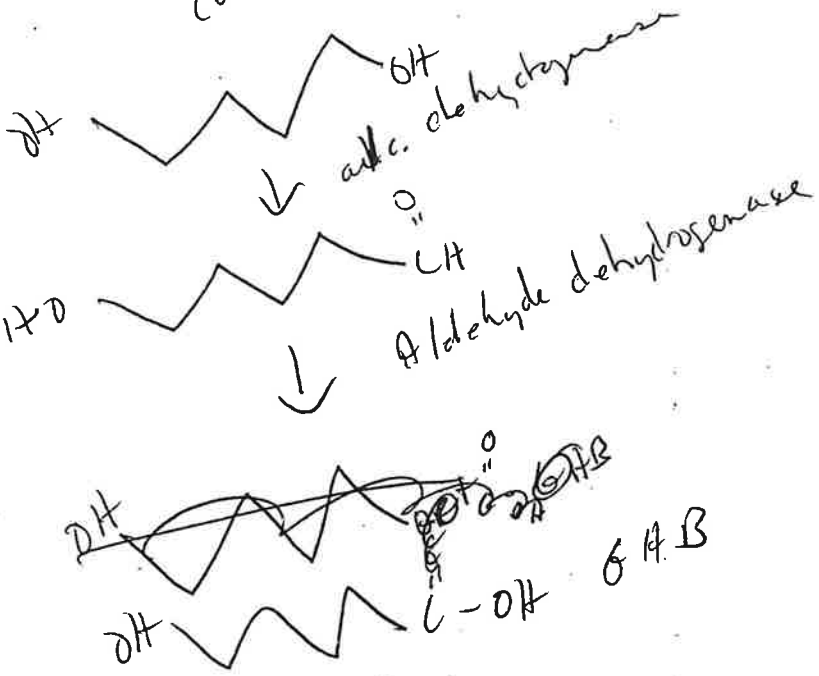
only difference

can be bought over internet.

↓ "

"Biocopia" brand name

contents to GHB in body



GAMMA HYDROXYBUTYRATE (GHB)

Current Status: GHB is a CNS depressant that is not approved for medical use in the United States. In 1990, the FDA issued an advisory declaring GHB unsafe and illicit, except under FDA-approved physician-supervised protocols. GHB is currently not a controlled substance under the federal Controlled Substances Act. To date however, eighteen states have controlled GHB: CI: Georgia, Rhode Island, Hawaii, Illinois, Louisiana, Nevada, Wisconsin, Michigan, Delaware, Idaho, Oklahoma; CII: Florida, California, Indiana, New Hampshire; CIV: Tennessee, Alaska, and North Carolina; two states (Texas, New Jersey) criminalized the sale and possession of GHB and placed it in the same penalty group as LSD and marijuana. The DEA has completed its evaluation and submitted it to the Department of Health and Human Services for a scientific and medical evaluation and scheduling recommendation.

Bills in Congress: (Note: There has been no action on any of these bills.)
Rep. Jackson-Lee (D-TX) On 5/5/97 placing GHB in Schedule I and ketamine in Schedule II.
Rep. Stupak (D-MI) and others on 5/21/97 placing GHB and ketamine in Schedule III.
Rep. Furse (D-OR) on 5/22/97 placing GHB in Schedule I and ketamine in Schedule II.
Rep. Stupak (D-MI) on 6/5/97 placing GHB and ketamine in Schedule III.

Licit Uses in the United States: None. GHB is currently under investigation for use in treating narcolepsy under the FDA's Orphan Drug program.

Illicit Uses: In recent years the abuse of GHB has increased. GHB (street names: liquid X, Georgia Home Boy, Goop, Gamma-OH, Grievous Bodily Harm) is abused for its ability to produce euphoric and hallucinatory states, and its alleged role as a growth hormone releasing agent to stimulate muscle growth. GHB can produce drowsiness, dizziness, nausea, visual disturbances, unconsciousness, seizures, severe respiratory depression and coma. Overdoses usually require emergency medical treatment, including intensive care for respiratory depression and coma. DEA has documented over 1,000 overdoses and law enforcement encounters with GHB and 26 GHB-related deaths.

GHB is produced in illicit laboratories using a relatively simple synthesis and available and inexpensive starting materials (gamma butyrolactone - GBL and sodium hydroxide). Information, kits containing the non-regulated precursor chemicals and "recipes" for making GHB are available in websites on the Internet.

GHB is most commonly found in liquid form in vials or small bottles, or sometimes as powdered material. Plastic sports bottles or "spring water" bottles are most often associated with its use. At bars or "rave" parties GHB is sold for $10 per capful or "swig". The typical dose is 1-5 grams, with an onset of effects of 15-30 minutes, and lasting 3-6 hours.

User Population: GHB is popular with high school and college students, and rave party attendees who use the drug for purposes of intoxication, body builders also abuse GHB for its purported anabolic effects, and by those using the drug to commit sexual assault. GHB is taken orally usually in combination with alcohol. DAWN statistics (1992-1996) confirm that the GHB user is white individuals (84%) and male (64%). GHB-related episodes most often involved 20-24 year olds (39%) and 25-29 year olds (30%); an additional 8% involved 10-19 year olds. In most GHB episodes, the motivation for taking GHB was for recreational abuse (79%).

Drug and Chemical Evaluation Section, DEA (202) 307-7183, 08/14/98

Gamma Hydroxybutyrate: Pharmacology, History and Current Patterns of Abuse

Christine A. Sannerud, Ph.D.
DEA – Drug & Chemical Evaluation Section
Alexandria, VA, USA

Gamma Hydroxybutyrate (GHB) is a CNS depressant abused for its ability to produce euphoric and hallucinatory states and its alleged role as a growth hormone releasing agent to stimulate muscle growth. In the 1980's GHB was available as a "natural" food supplement and sold in health food stores, but the medical community soon became aware of overdoses and other problems caused by its abuse. GHB can produce drowsiness, dizziness, nausea, visual disturbances, unconsciousness, hypotension, bradycardia, petit mal epilepsy, seizures, severe respiratory depression and coma. Overdoses usually require emergency medical treatment including intensive care for respiratory depression and coma. In 1990, the FDA issued an advisory declaring GHB unsafe and illicit, except under FDA-approved physician-supervised protocols. GHB has not been approved by the FDA or marketing, but it is currently under investigation for use in the treatment of narcolepsy under the FDA's Orphan Drug program.

Although banned, the abuse of the drug for its euphoric effects has increased. It is produced in illicit laboratories using a relatively simple synthesis and available and inexpensive starting materials. In illicit traffic, GHB is most commonly found in liquid form in vials or small bottles, or sometimes as powdered material GHB is taken orally usually in combination with alcohol. GHB is popular with a wide range of abusers in the US, including high school and college students and rave party attendees who use GHB for intoxication. Body builders also abuse GHB for its alleged anabolic effects. There are reports of individuals who use GHB to incapacitate women for purposes of committing sexual assault.

Over 1,000 encounters with GHB have been documented by information gathered from law enforcement, poison control centers, and hospitals. There have been many overdose cases attributed to GHB abuse. The DEA has also documented twenty-six cases in which GHB was found in the biological fluids of deceased individuals.

GHB is currently not a controlled substance under t the federal Controlled Substances Act. To date however, seventeen states have controlled GHB: CI – GA, RI, HI, IL, LA, NV, WI, MI, DE, ID; CII – FL, CA, IN, NH; CIV – TN, AL, NC. Two additional states (Texas and New Jersey) have criminalized the sale and possession of GHB and placed it in the same penalty group as LSD and marijuana. In September 997, the DEA completed its evaluation and submitted it to DHHS for a scientific and medical evaluation and scheduling recommendation.

send data about GHB
~~to~~
Fax 202-307-8570

Drug Enforcement Administration
Office of Diversion Control
Drug & Chemical Evaluation Section
Washington, DC 20537

Gamma Hydroxybutyrate (GHB) Briefing Book

July 1998

GAMMA HYDROXYBUTYRATE (GHB)

- GHB is a CNS depressant.

- GHB is abused:
  - as an intoxicant for its euphoric and hallucinatory effects
  - for its alleged role as a growth hormone releasing agent to stimulate muscle growth, and
  - to incapacitate individuals for sexual assault.

- Since 1990, GHB has been banned by the Food and Drug Administration, except under FDA-approved physician-supervised protocols.

- GHB has never been approved by the FDA for medical use.

- GHB is clandestinely manufactured using simple methods and available starting materials.

- Although banned, abuse of GHB continues to dramatically increase:
  - DEA has documented over 1000 cases of overdose, abuse and trafficking encounters in 36 states
  - DEA has documented 26 deaths associated with GHB abuse
  - DEA has documented 9 sexual assault cases, involving 19 victims, under the influence of GHB.

- GHB is not currently controlled under the federal Controlled Substances Act.
  - GHB is controlled in seventeen US states.
  - GHB possession and sale is criminalized in two US states

ABUSE OF GHB

- Abuse of GHB has increased since 1993.

- GHB is abused by:
  - High school and college students, young adults, rave and nightclub attendees
  - Body builders
  - Individuals intent on committing sexual assault or other criminal activity

- GHB is taken orally as a liquid or as a powder mixed in a liquid (water, juice or alcohol), rarely in the form of capsules.

- GHB is abused as an intoxicant at nightclubs and rave parties
  - To get high
  - To produce more profound effect with alcohol
  - To counter the effects of stimulants
  - To regulate the effects of hallucinogens
  - To alleviate withdrawal effects from alcohol

- GHB is abused by bodybuilders
  - For its alleged role as a growth hormone releaser
  - As a diet aid
  - To counter the effects of stimulants
  - To sleep after workouts

- GHB is used to facilitate sexual assault
  - To physically and mentally incapacitate individuals to target for sexual assault
  - "Date-rape" drug
  - Cases documented in Florida, Texas, Maryland, Louisiana, California, Michigan, Wisconsin

HISTORY OF GHB USE

History of Legal GHB use

- GHB was developed in France in the 1950's as an anesthetic.
- Studies of GHB in the 1960's evaluated its effects as an anesthetic. During this time an Investigational New Drug application (IND) for use as an anesthetic was submitted to FDA and then withdrawn.
- In the 1960's-80's GHB was used as a seizure model in animals to study the effects of antiepileptic medications.
- In the mid 1980's FDA-approved IND was submitted for the use of GHB in the treatment of narcolepsy. There is currently no FDA approved medical use of GHB.

History of Illegal GHB use

- In the late 1980's GHB was used as a steroid substitute. It was used by bodybuilders and sold in health food stores and gymnasiums.
- In 1990, FDA issued a health alert and banned the use of GHB.
- Since the early 1990's, GHB has been sold at nightclubs, rave parties, and bars. Kits for making GHB "home-brew" are sold through magazines, via the Internet. GHB has been made in small quantities using home-brew kits on college campuses, and in larger scale clandestine laboratories.
- Use of GHB has resulted in substantial numbers of overdoses, toxic reactions, calls to poison control centers, emergency room visits, and sexual assaults.

ACTUAL ABUSE OF GHB

liquid, powder 1 tsp. (2.5 grams) 1 capful occasionally in capsules.

- PATTERN OF ABUSE

GHB is a powder, however, it is often sold and taken in solution

- The dose is 1-5 grams
- There is an onset of effects within 15 -30 minutes
- The effects last 3 to 6 hours, depending upon the dose

- GHB potentiates the CNS depressant effects of alcohol and other depressants

•ADVERSE EFFECTS:

- GHB dose-dependently produces adverse effects:

  - Drowsiness
  - Dizziness
  - Agitation
  - Confusion
  - Inebriation
  - Stupor
  - Nausea and Vomiting
  - Reduction of Gag Reflex
  - Hypothermia
  - Reduced Muscle Tone
  - Reduced Blood Pressure
  - Reduced Heart Rate
  - Decreased Respiration
  - Seizures
  - Coma
  - Death

Consider female viagra known aphrodisiac

DEPENDENCE ON GHB

- Chronic use of GHB produces physical and psychological dependence
  - Physical Dependence
    - Withdrawal
    - Tolerance
  - Psychological Dependence
    - Escalation of GHB Dose
    - Increased Frequency of Use
    - Continued Use Despite Adverse Consequences
    - Unsuccessful Efforts to Decrease or Stop Use

drug is metabolized quickly.

ADVERSE REACTIONS AND TOXICITY

- DEA has documented over 700 adverse reaction and overdoses cases from many sources, including:

  - Poison Control Center Data from US and from Several States
    California, New York, Texas, Oklahoma, Georgia, and Florida

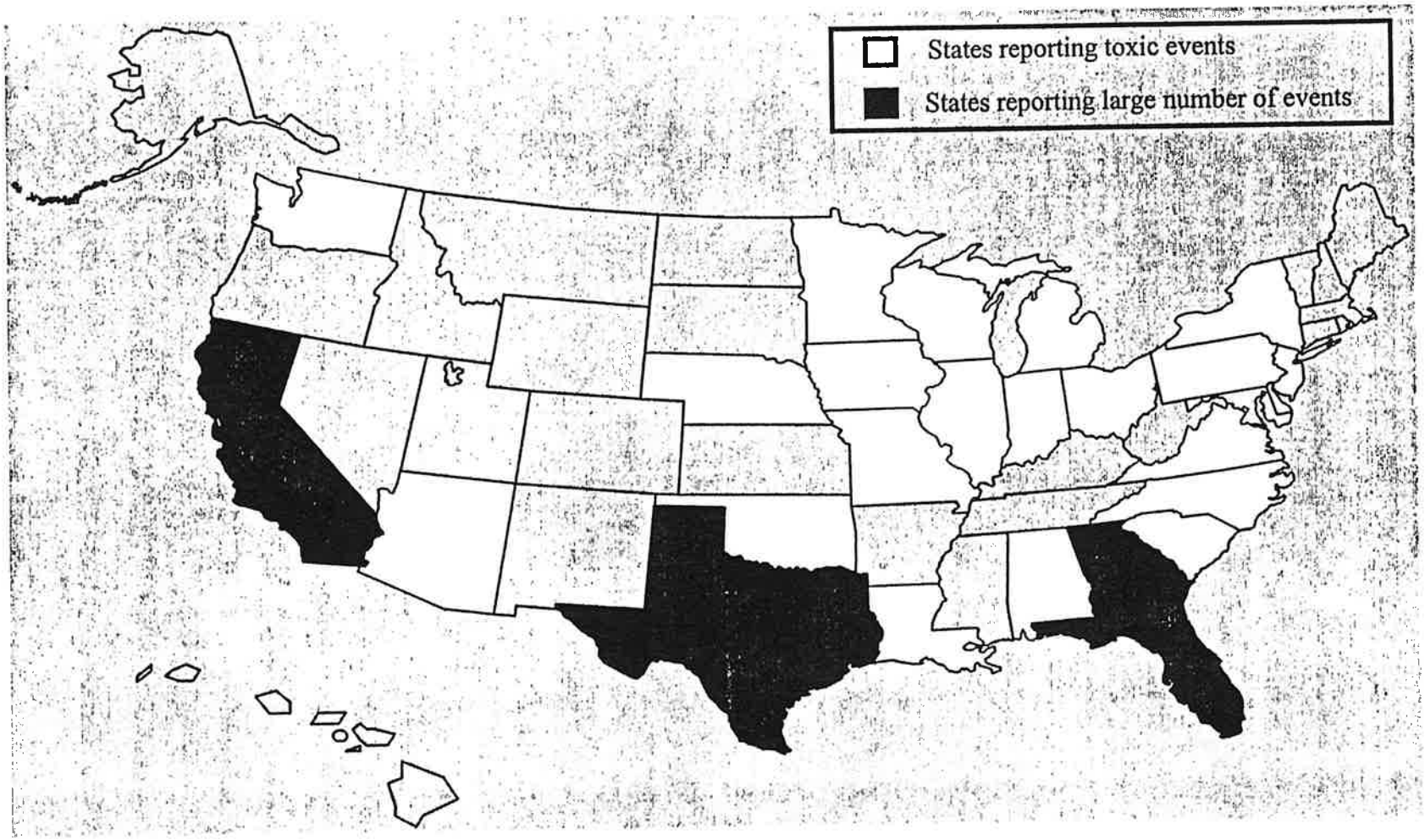
  - Hospital Emergency Room Reports, Emergency Medical Services Reports, College and University Security/Incident Reports, Medical Examiners Autopsy and Toxicology Reports, Coroner's Office Reports

  - Medical Literature
    Case reports and clinical trials

  - Law Enforcement Cases
    DEA, State and Local Cases, including DUI, Drunk and Disorderly Conduct, Overdose, Undercover Operations at Bars/Clubs

  - Drug Abuse Warning Network (DAWN)

GHB Related Toxic Reactions / Overdoses



*Numerous Sources 1990 - 1998

GHB RELATED DEATHS

- There have been 26 GHB Related Deaths Since 1990

Year	Number
1990	1
1993	1
1995	2
1996	12
1997	8
1998	2

- These deaths were reported from:

State	Number
California	7
Florida	6
Texas	4
Georgia	2
Illinois	1
Maryland	1
Michigan	1
Nebraska	1
North Carolina	1
Ohio	1

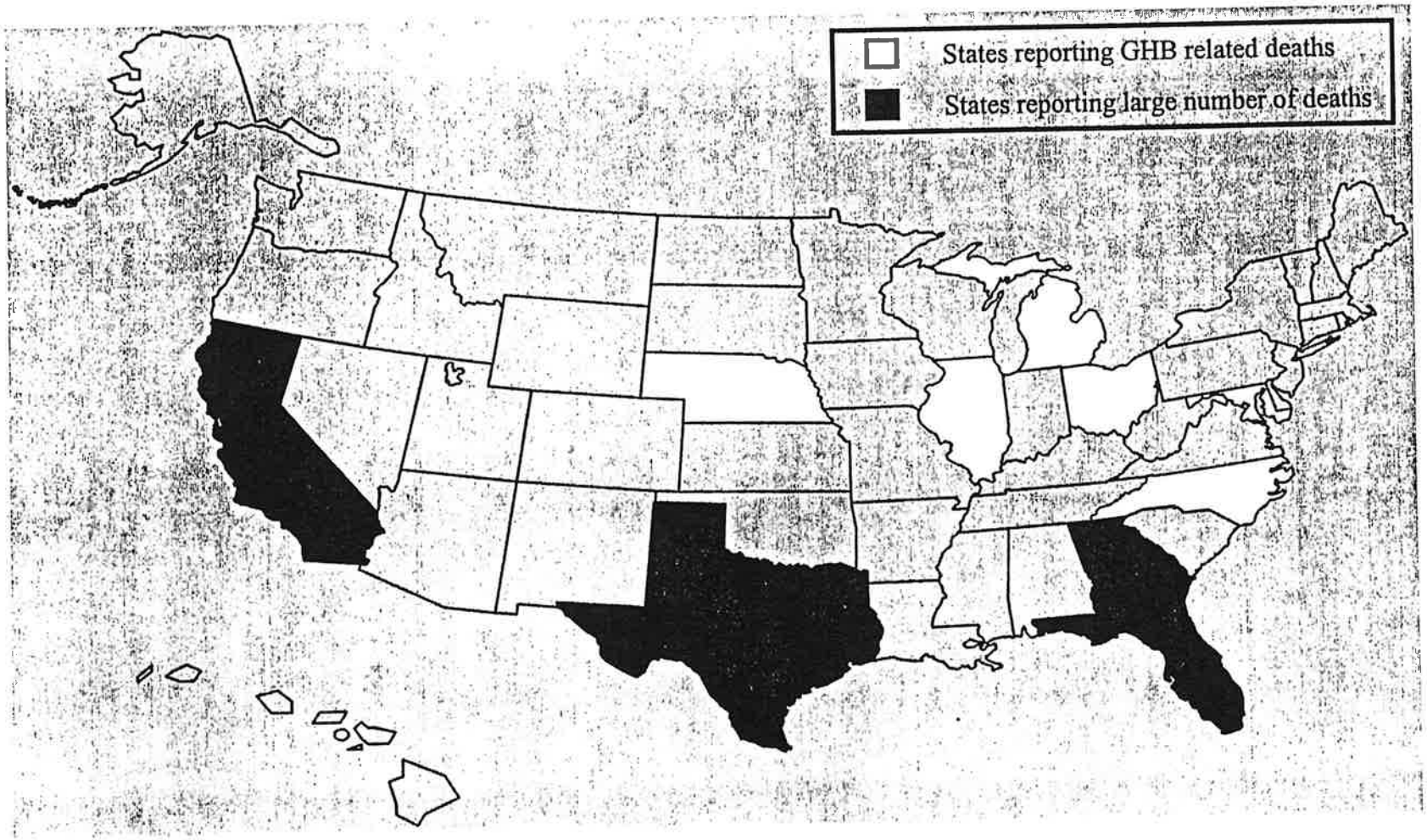
- Ages of decedents

	Number	Percent
10-19 years	3	11%
20-29 years	13	50%
30-39 years	6	23%
40-49 years	2	8%
50-59 years	1	4%
70-79 years	1	4%

- Gender of decedents

		Percent
Male	19	73%
Female	7	27%

GHB Related Deaths in the U.S.



*Numerous Sources 1990 - 1998

Drug Abuse Warning Network (DAWN)
GHB Emergency Room Episodes

- 411 Emergency Room Episodes (1991-1996)

- Demographics:
  - Male (64%)
  - White (84%)
  - Ages:
    - 10-19 years (8%)
    - 20-24 years (39%)
    - 25-29 years (30%)
    - 30-34 years (15%)
    - 35-39 years (8%)

- Reasons for Abusing GHB

Recreational Abuse	79%
Dependence	9%
Psychedelic Effects	7%
Suicide	6%

- GHB Used Alone: 27% of episodes

- GHB Used in Combination With:

Alcohol	86%
Stimulants	20%
Hallucinogens	18%
Sedatives	3%

GHB Clandestine Laboratory Activity

Clandestine Laboratory Synthesis

Precursors:

- Gamma-butyrolactone + Sodium Hydroxide
- Kits and recipes are found on the Internet

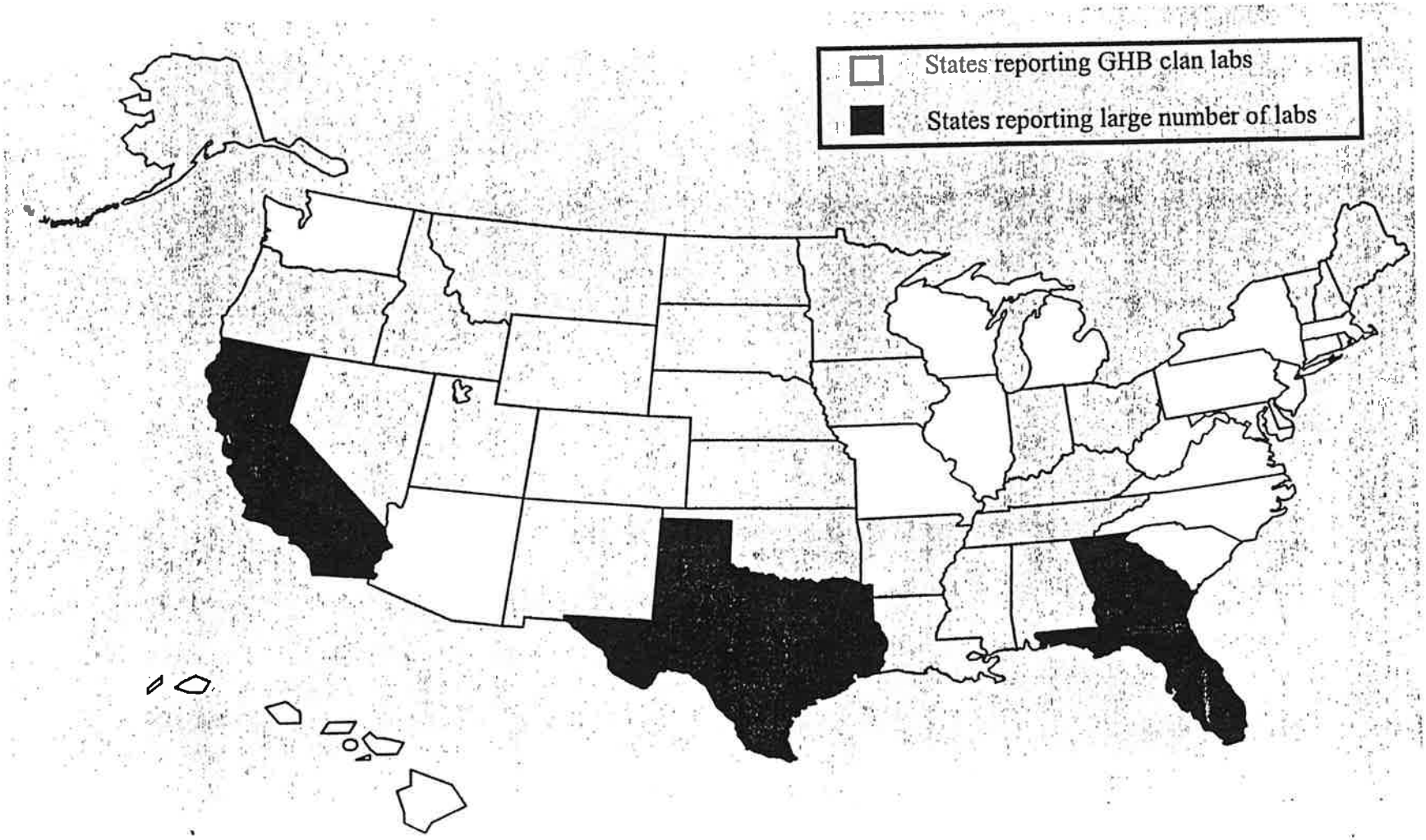
Method:

- "One pot" stove top method
- Doesn't require heat

Year	Number of encounters
1990	1
1991	0
1992	1
1993	6
1994	3
1995	8
1996	7
1997	20
July 1998	23
Total	69

Most of the clandestine laboratory activity was reported from California, Georgia, Arizona, Texas, Florida, North Carolina, Rhode Island, New York, Washington, Michigan, Illinois

GHB Clandestine Laboratory Activity



*Numerous Sources 1990 - 1998

LAW ENFORCEMENT ACTIVITY

- **Since GHB is not a federally controlled substance, it is not a target or priority of DEA law enforcement activity. For these reasons, the abuse and trafficking of GHB is severely underreported.**

- To date, GHB has been encountered in 36 States on 300 occasions

- The forms and amounts of GHB seized ranges from:

Form	Amount
Powder:	0.37g to 1 kg
Liquid:	0.001 ml to 6688 ml
Capsules:	812

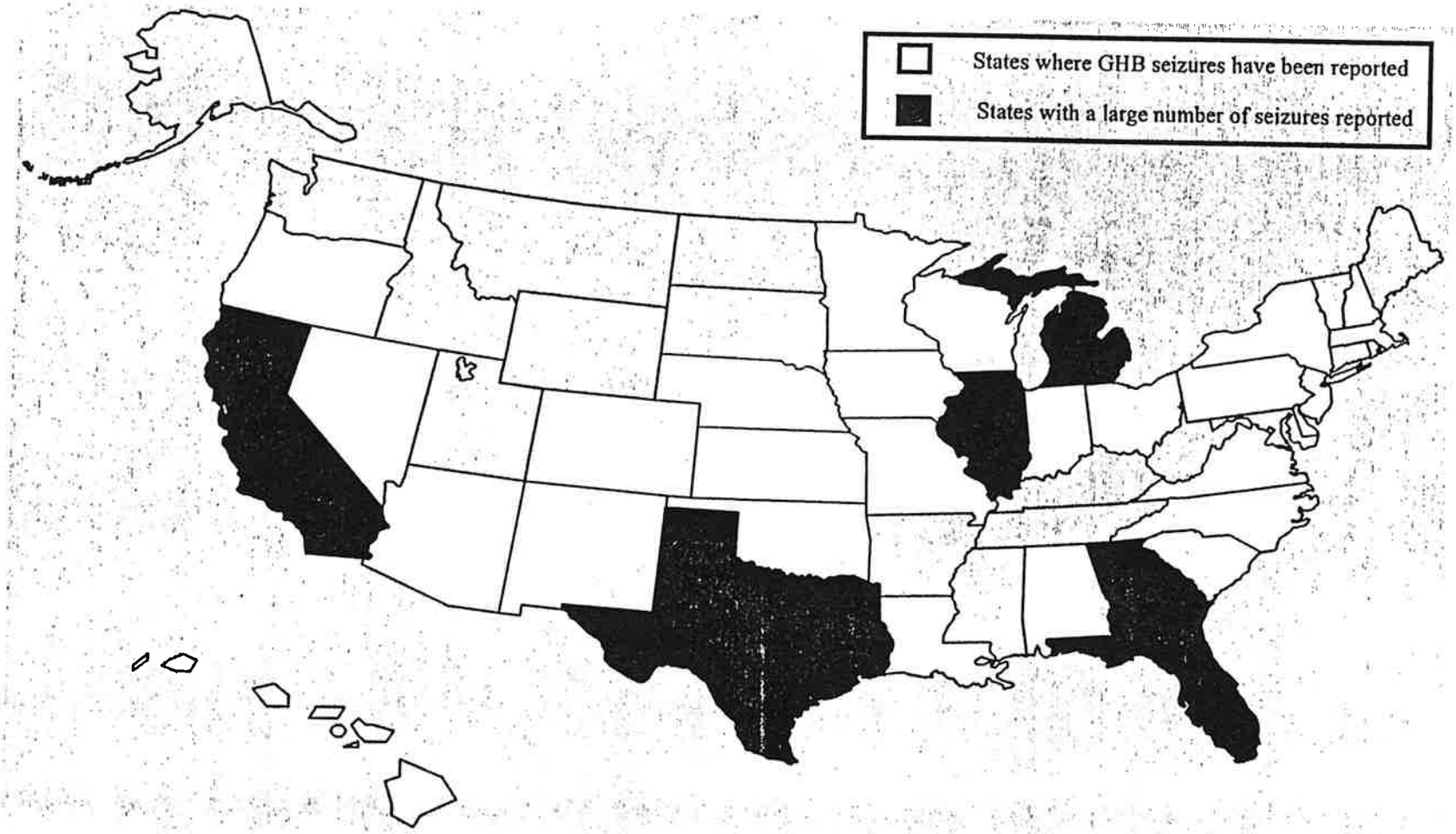
- GHB is encountered in a variety of containers, including water bottles, plastic bags, vials, gallon milk containers, buckets and drums

- GHB is currently controlled under state laws in seventeen states

  - Schedule I: Georgia, Rhode Island, Hawaii, Illinois, Louisiana, Nevada, Wisconsin, Michigan, Delaware, Idaho
  - Schedule II: Florida, California, Indiana, New Hampshire
  - Schedule IV: Tennessee, Alaska, North Carolina

- GHB possession, manufacture and trafficking is criminalized in two states (Texas, New Jersey) and placed in the same penalty group as LSD and marijuana

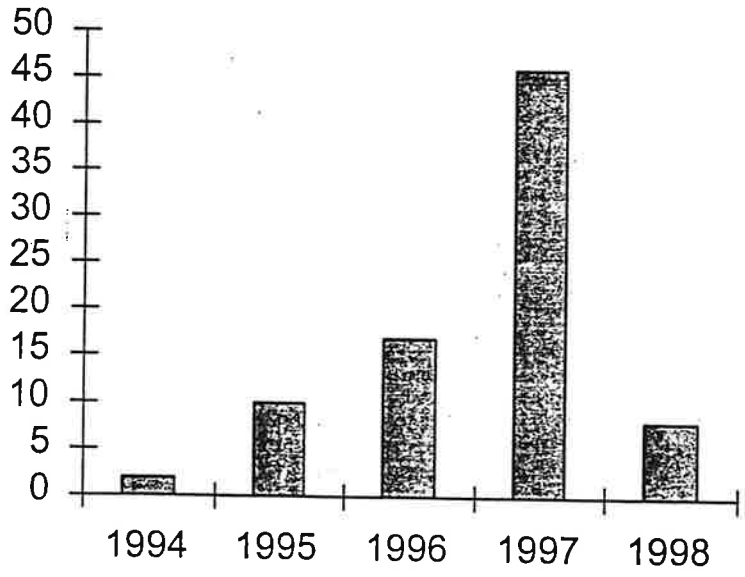
GHB Trafficking and Distribution



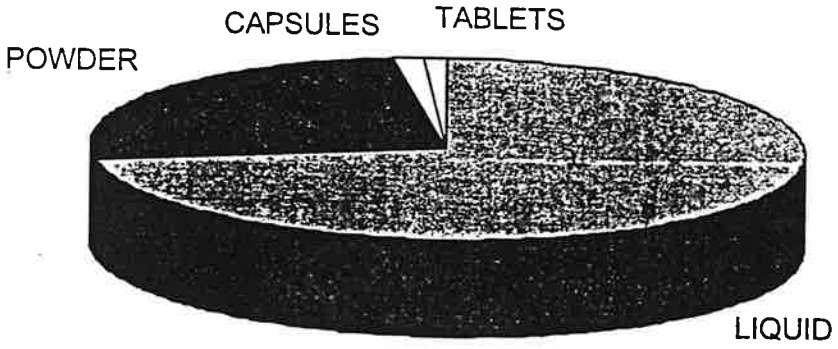
*Numerous Sources 1990 - 1998

GHB Forensic Analysis

Number of Exhibits



Form of Exhibits



GHB Forensic Analysis

- **Some of the GHB samples seized have been analyzed in DEA forensic laboratories and included in the DEA STRIDE database.**

Yearly GHB cases from the DEA STRIDE database (8/94-7/98)

Year	Number of exhibits
1994	2
1995	10
1996	17
1997	46
1998	8
Total	**83 (43 total cases)**

Form of Exhibits in STRIDE Cases (8/94-7/98)

Liquid	60
Powdered material	21
Capsules	1
Tablets	1
TOTAL	**83**

GHB Forensic Analysis

- **In non-DEA cases, GHB has been analyzed by state and local forensic laboratories**

Yearly Non DEA Forensic Analyses (1993-7/98)

Year	Number of exhibits
1993	3
1994	4
1995	2
1996	9
1997	32
July 1998	22
Total	**72**

Form Submitted in Non-DEA Cases (1993-7/98)

Liquid	42
Powdered material	8
Capsules	1
Unknown	21
Total	**72**

CURRENT STATUS OF GHB CONTROL

- Abuse, trafficking and diversion of GHB is increasing. DEA has documented over 1,000 encounters of GHB, including 26 deaths associated with GHB abuse. This information is gathered from law enforcement, poison control centers, and hospitals.

- GHB is not controlled federally under the Controlled Substances Act
  - GHB is not the target of most law enforcement efforts
  - DEA continues to collect and analyze data from many sources
  - DEA has completed an evaluation of GHB
  - This review was sent to the Department of Health and Human Services for a scientific and medical evaluation.

must also take Gamma Butyrolactone (GBL) into consideration.
GBL ⇒ converts to GHB in body.

Law must include all salts, isomers, + esters etc.

if make Na salt only can have problem if K salt, etc.

VERIFICATION OF THE IDENTIFICATION OF GHB IN A LIQUID BY SILYLATION

OVERVIEW

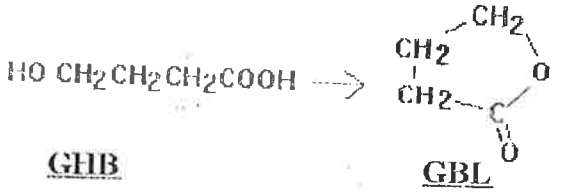
Gamma-hydroxybutyrate, GHB, is often referred to as a date rape drug, but it is also widely abused by body builders and nightclub attendees.

$$HO\text{-}CH_2CH_2CH_2COOH$$

GHB

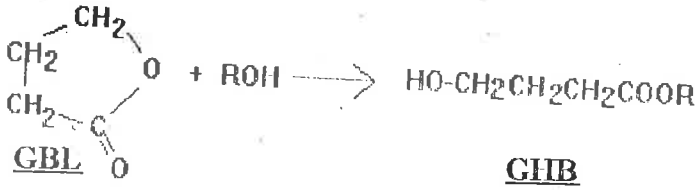
Because the use of GHB is on the rise, crime laboratories are being forced to develop analytical procedures to successfully identify GHB.

The powder form of GHB is relatively easy to analyze; however, liquid samples containing GHB require some sample preparation before analysis is possible. Because GHB lies in equilibrium with its lactone, GBL, it is important to determine whether GBL is present in your sample.



GHB **GBL**

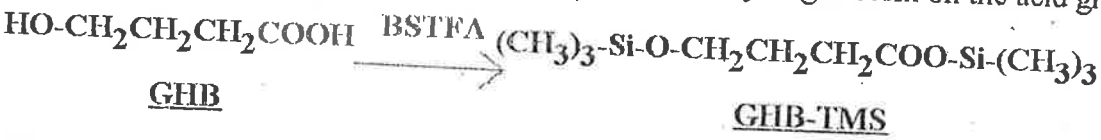
GBL can be used to manufacture GHB in clandestine laboratories and can be easily obtained. GBL is found in wood cleaners, paint removers, and textiles.



GBL **GHB**

Because GBL can be removed from liquids with a chloroform extraction, we can concentrate on determining the presence of GHB in our sample.

Treatment of GHB with BSTFA, a derivatizing agent, will result in a silyl replacement of both the alcohol group hydrogen and the hydrogen atom on the acid group.



GHB

GHB-TMS

Silylation of GHB produces a stable derivative that can be readily analyzed by a GC\MS.

TRIAL PROCEDURE I

A. Take 1-5 mL of your sample and dry it down in a mortar using a hair dryer on low heat. Do not exceed 150 C or GHB may convert to GBL.

B. Transfer your sample from the mortar to a small (10 mL) beaker. To the beaker containing your sample, add 2 mL of acetonitrile, 500 uL of BSTFA, and 500 uL of TCMS.

C. Transfer the entire contents of the beaker to an autosampler vial and cap it. Heat the vial in a 60 C-80 C water bath for 10-15 minutes.

D. Analyze on GC\MS.

RESULTS FROM TRIAL PROCEDURE I

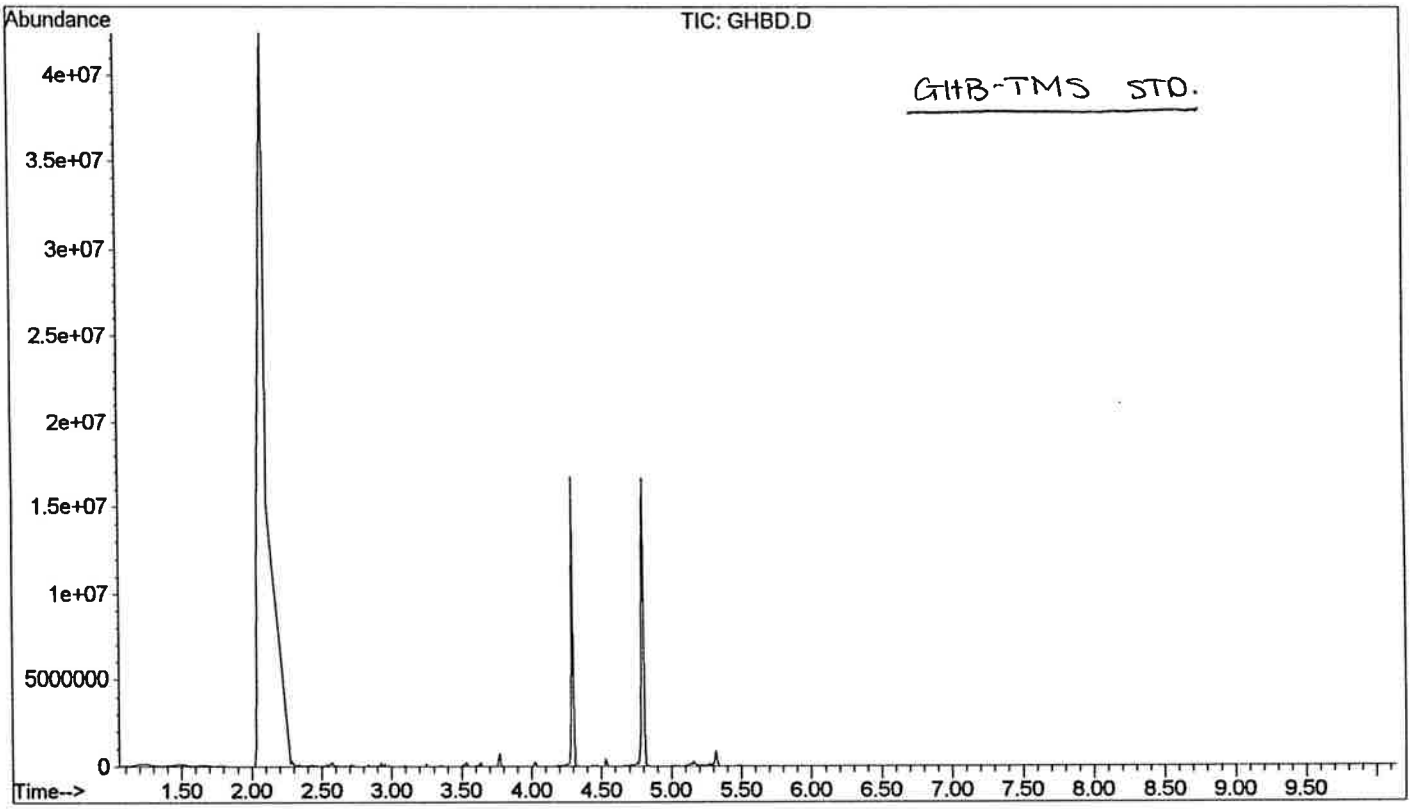
In order to identify GHB-TMS, the GHB derivative, ions 117, 133, 159, 204, and 233 must be present in the mass spectrum. The ions common to TCMS derivatives, 73, 147, and 243, should also be present.

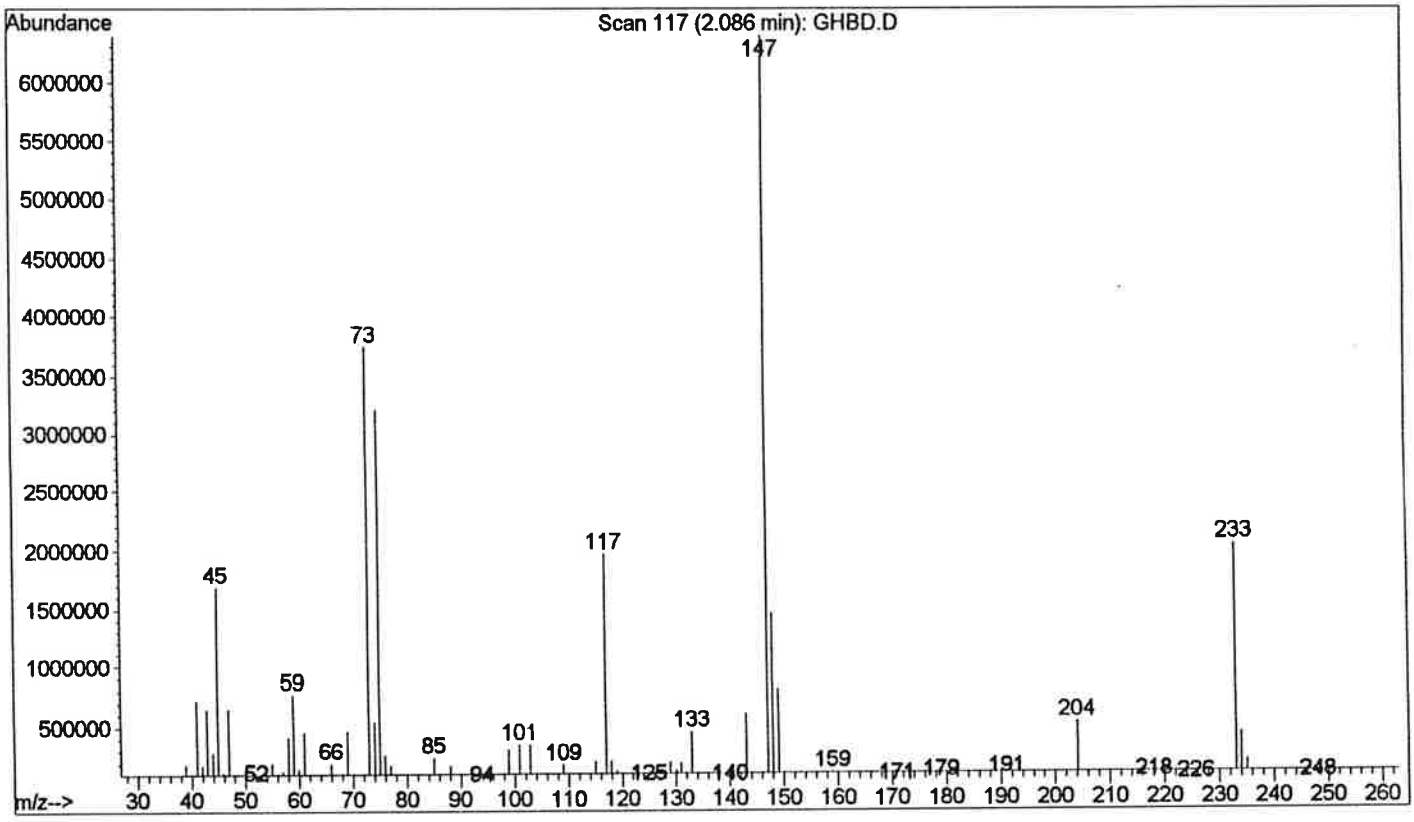
Five samples were prepared following this procedure, including the standard. The standard was prepared by taking 20 mg of GHB and derivatizing it with BSTFA and TCMS. The resulting spectra was consistent with that of known standards for GHB-TMS. The other four samples consisted of 0.2 g of GHB in 10 mL of water, 30 mg of GHB in Gatorade, and 30 mg of GHB in 10 mL of Coca-Cola.

In this procedure, the first step is to dry down your sample. If the sample appeared to be in water, the procedure worked wonderfully; however, if your liquid was in something other than water, the procedure was not very successful. GHB in water will dry down to form a residue that is easily derivatized, but liquids such as Gatorade and Coke only dry down to form a syrup-like substance. This syrup not only contains the GHB, but it also contains a significant amount of water. In order for the derivatizing agents to react with the GHB, the syrup must be introduced to the derivatizing agents, but because water inhibits the derivatizing agents, the syrup contains the solution and the problem.

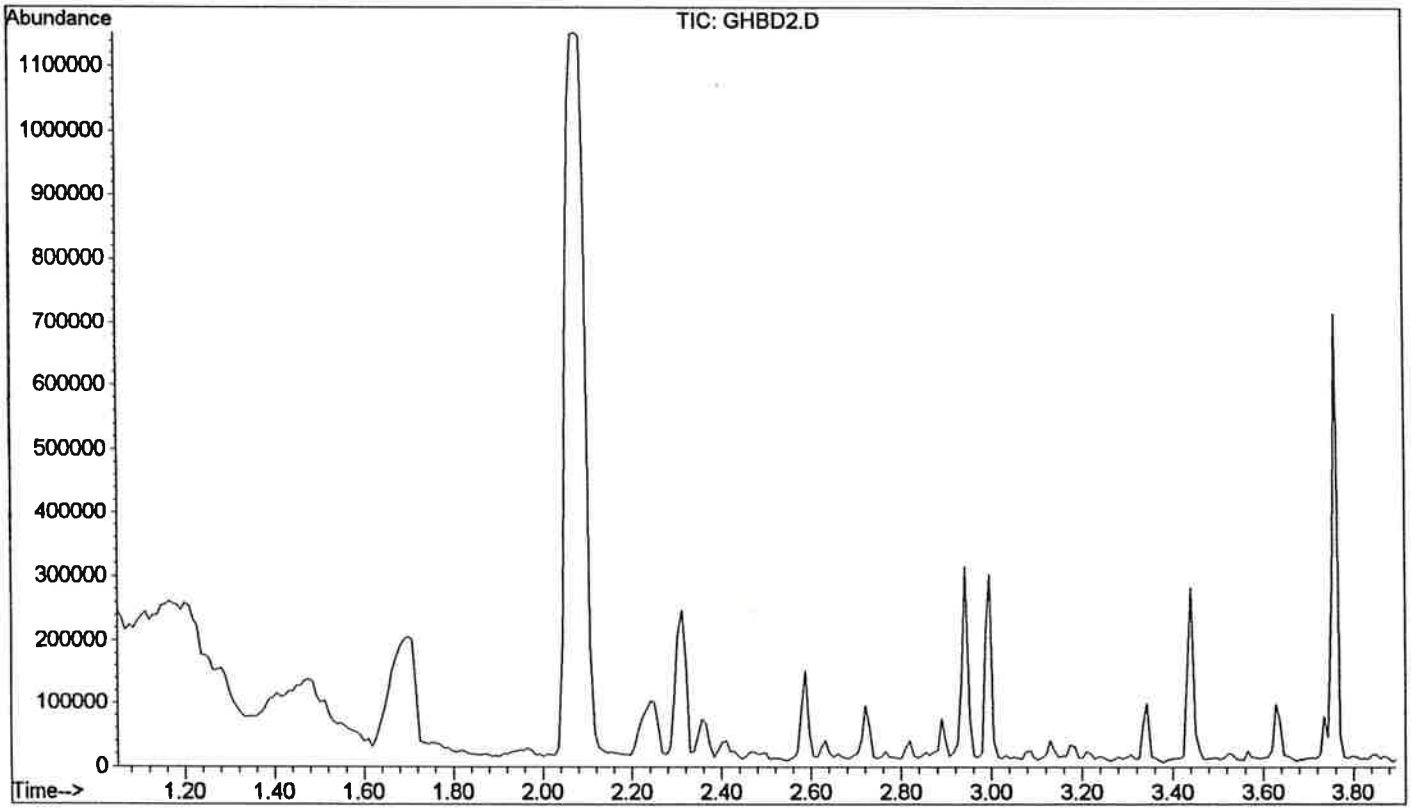
Because water is present in the GHB-Gatorade and GHB-Coke syrups, the GHB-TMS peaks in the resulting spectra are very weak. In addition to the GHB-TMS peak, sugars and acids show up in relatively high concentrations due to the presence of the syrup while derivatizing the GHB. Therefore, the next step in improving the procedure is to find a way to clean up the sample.

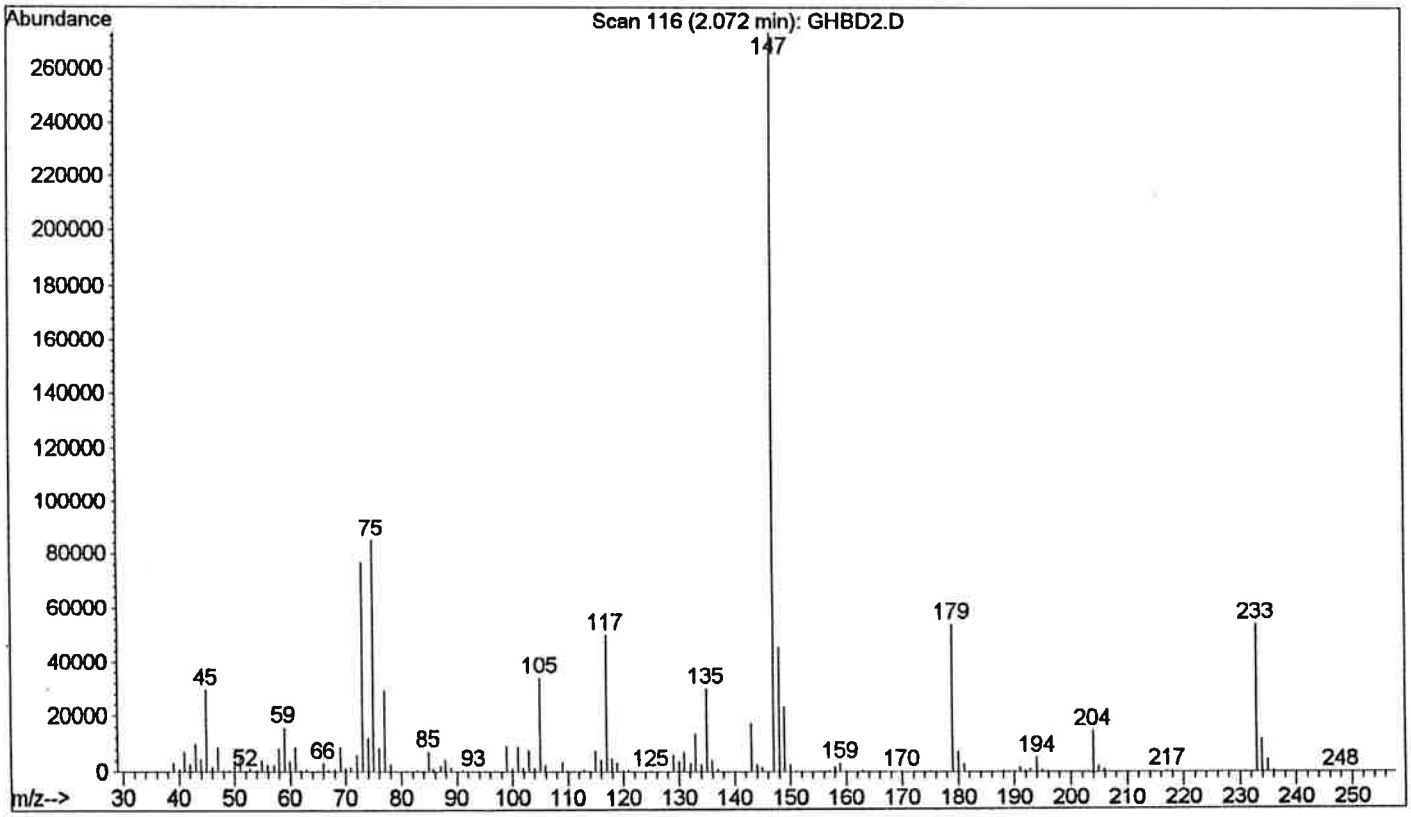
File : C:\HPCHEM\1\DATA\PWF\GHBD.D
Operator : JEJ
Acquired : 14 Jun 1999 10:46 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB-TMS
Misc Info : DERIVATIZING GHB USING TRIAC PROCEDURE I
Vial Number: 1



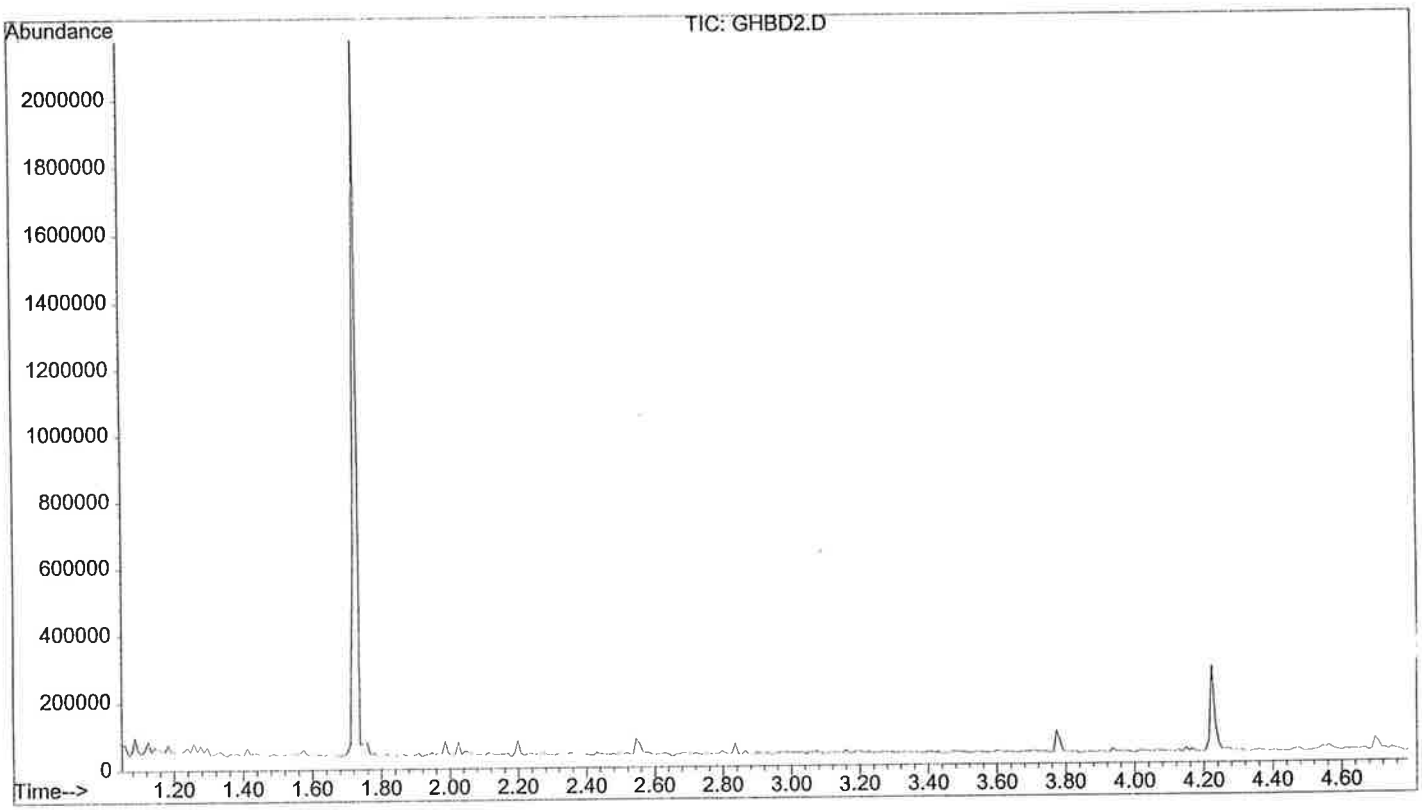


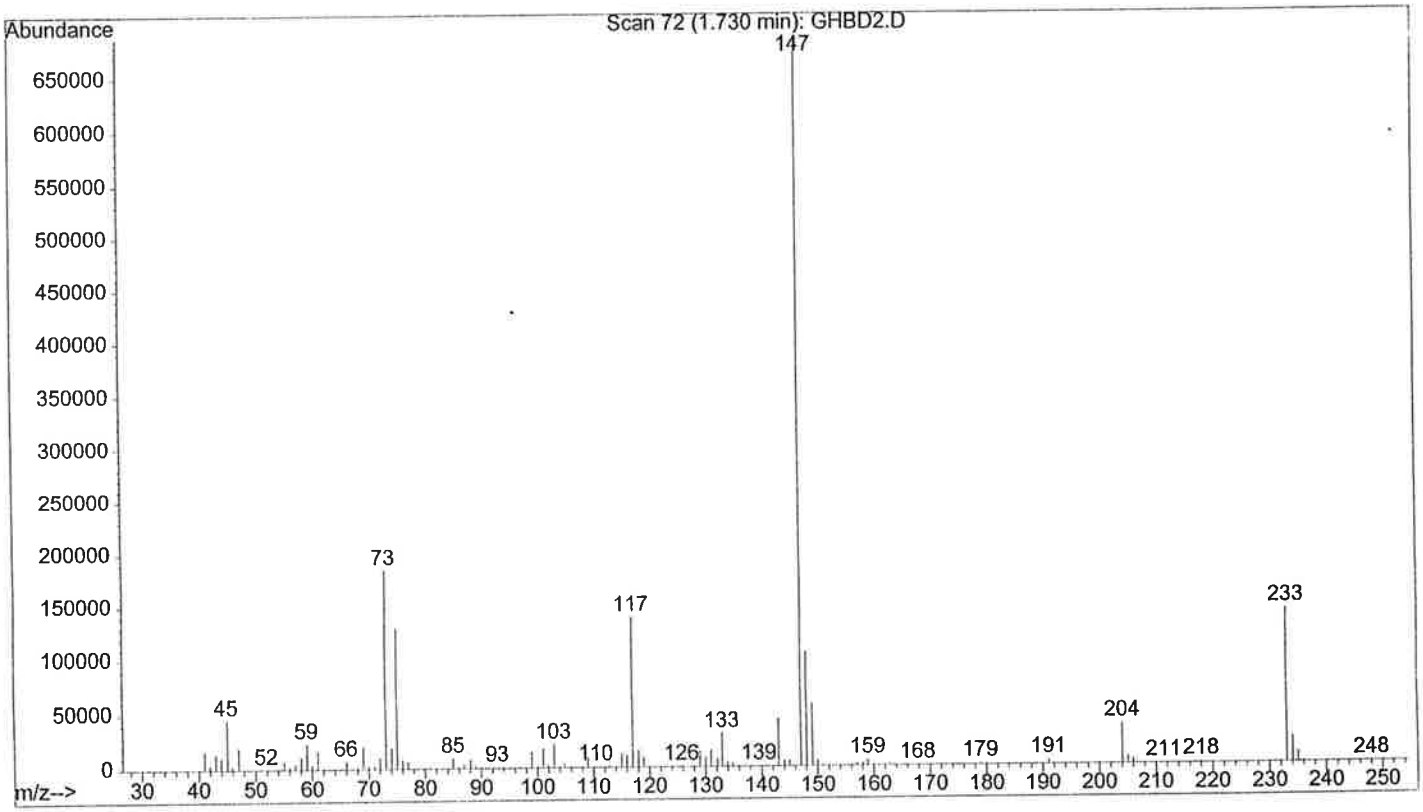
File : C:\HPCHEM\1\DATA\PWF\GHBD2.D
Operator : JEJ
Acquired : 14 Jun 1999 4:04 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: .20 g GHB IN 10 ml H20/DERIV
Misc Info : USING TRIM PROCEDURE I
Vial Number: 1



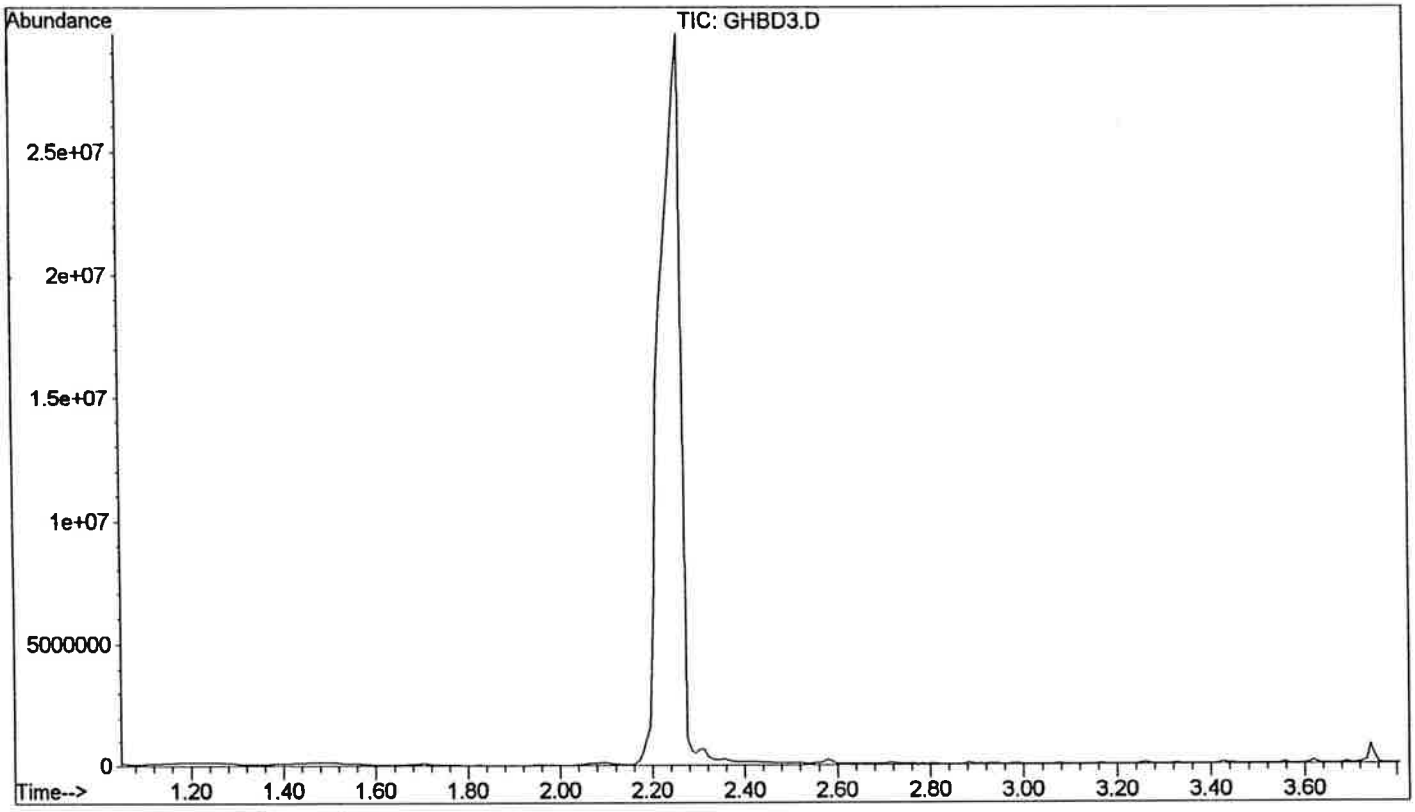


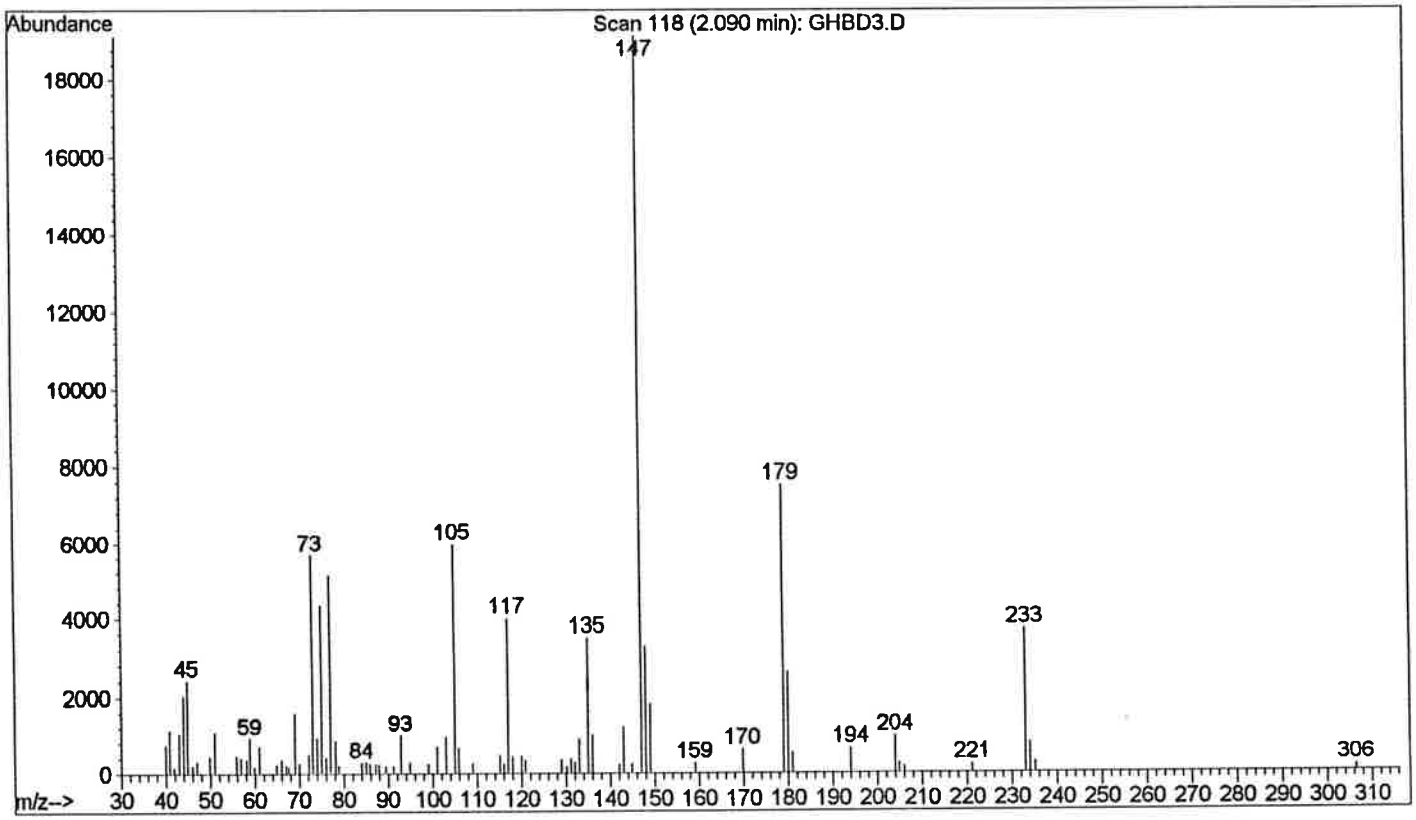
File : C:\HPCHEM\1\DATA\GHBD2.D
Operator : JEJ
Acquired : 15 Jun 1999 8:16 using AcqMethod MSDRUGS
Instrument : Donna
Sample Name: .2 g GHB IN 10 ml H20/DERIV GHB-TMS
Misc Info : USING TRIMC PROCEDURE I
Vial Number: 1



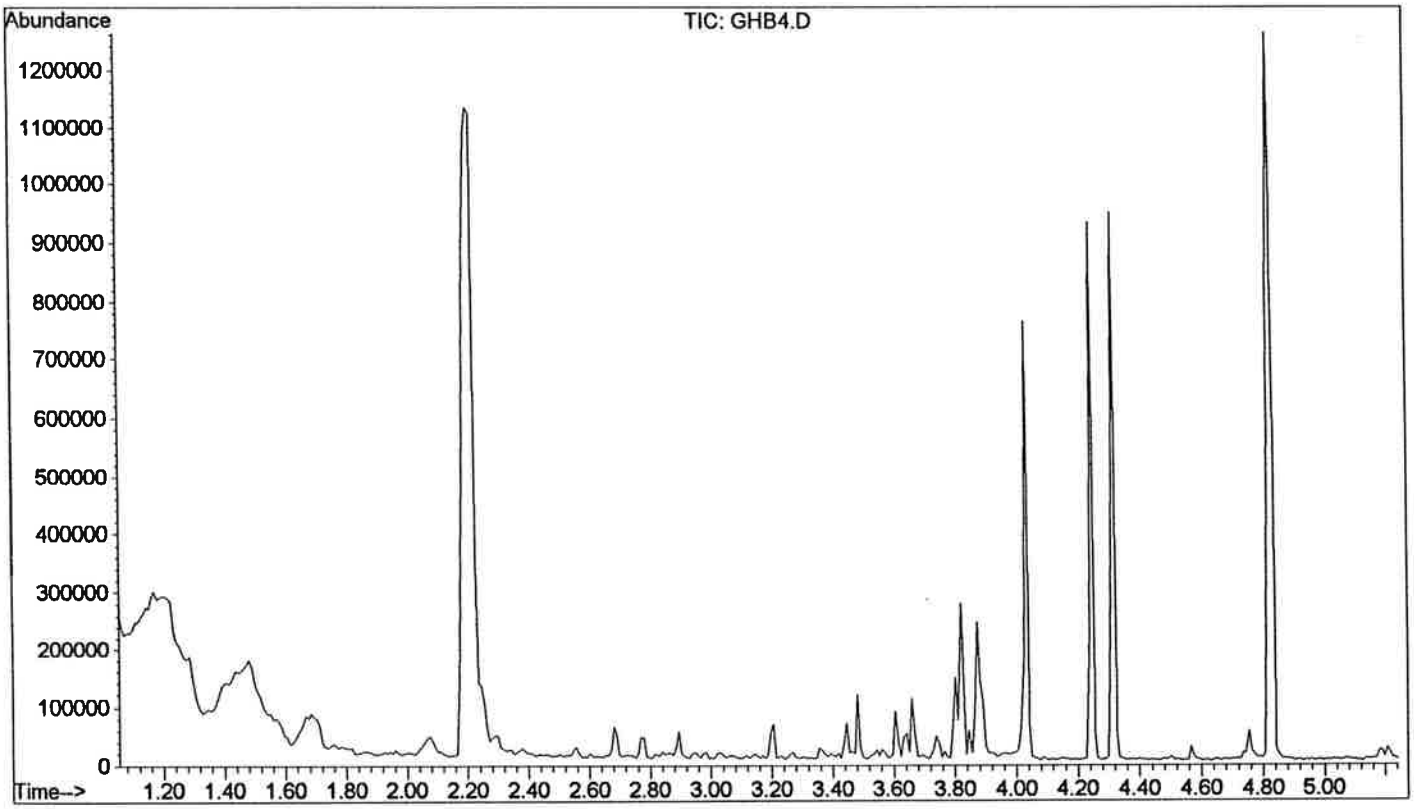


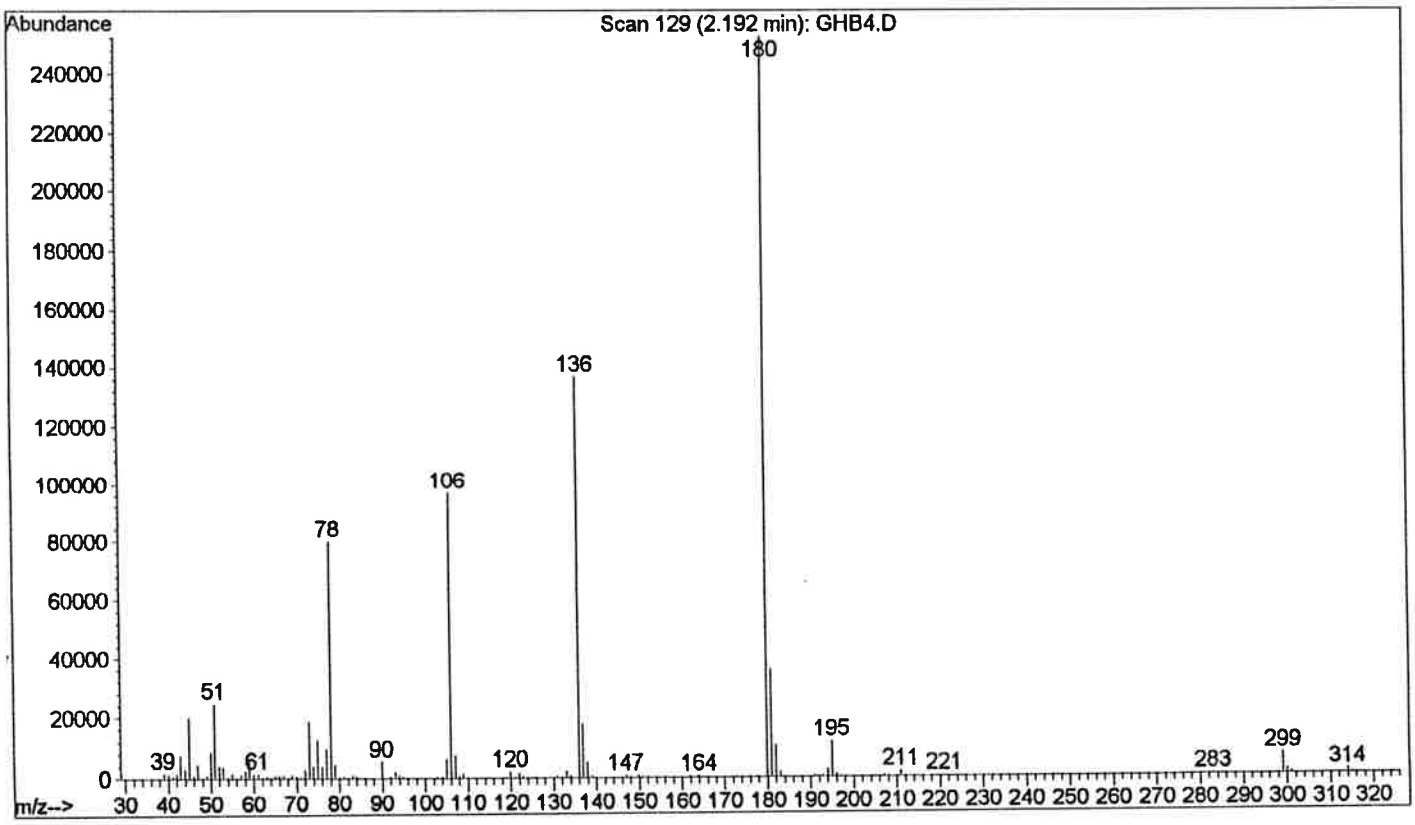
File : C:\HPCHEM\1\DATA\PWF\GHBD3.D
Operator : JEJ
Acquired : 15 Jun 1999 10:23 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: 30 mg GHB IN 10 ml GATORADE
Misc Info : USING TRIAL PROCEDURE I
Vial Number: 1





File : C:\HPCHEM\1\DATA\PWF\GHB4.D
Operator : JES
Acquired : 15 Jun 1999 10:51 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: 30 mg GHB IN 10 ml COKE
Misc Info : USING TRIM PROCEDURE I
Vial Number: 1





TRIAL PROCEDURE II

A. Extract 1-5 mL of your sample with excess $CHCl_3$. Extract once more using an equal amount of $CHCl_3$. This will remove any residual GBL.

B. Take 1-5 mL of your sample and dry it down in a mortar using a hair dryer on low heat. Do not exceed 150 C or GHB may convert to GBL.

C. Transfer your sample from the mortar to a small (10 mL) beaker. To the beaker containing your sample, add 2 mL of acetonitrile, 500 uL of BSTFA, and 500 uL of TCMS.

D. Transfer the entire contents of the beaker to an autosampler vial and cap it. Heat the vial in a 60 C-80 C water bath for 10-15 minutes.

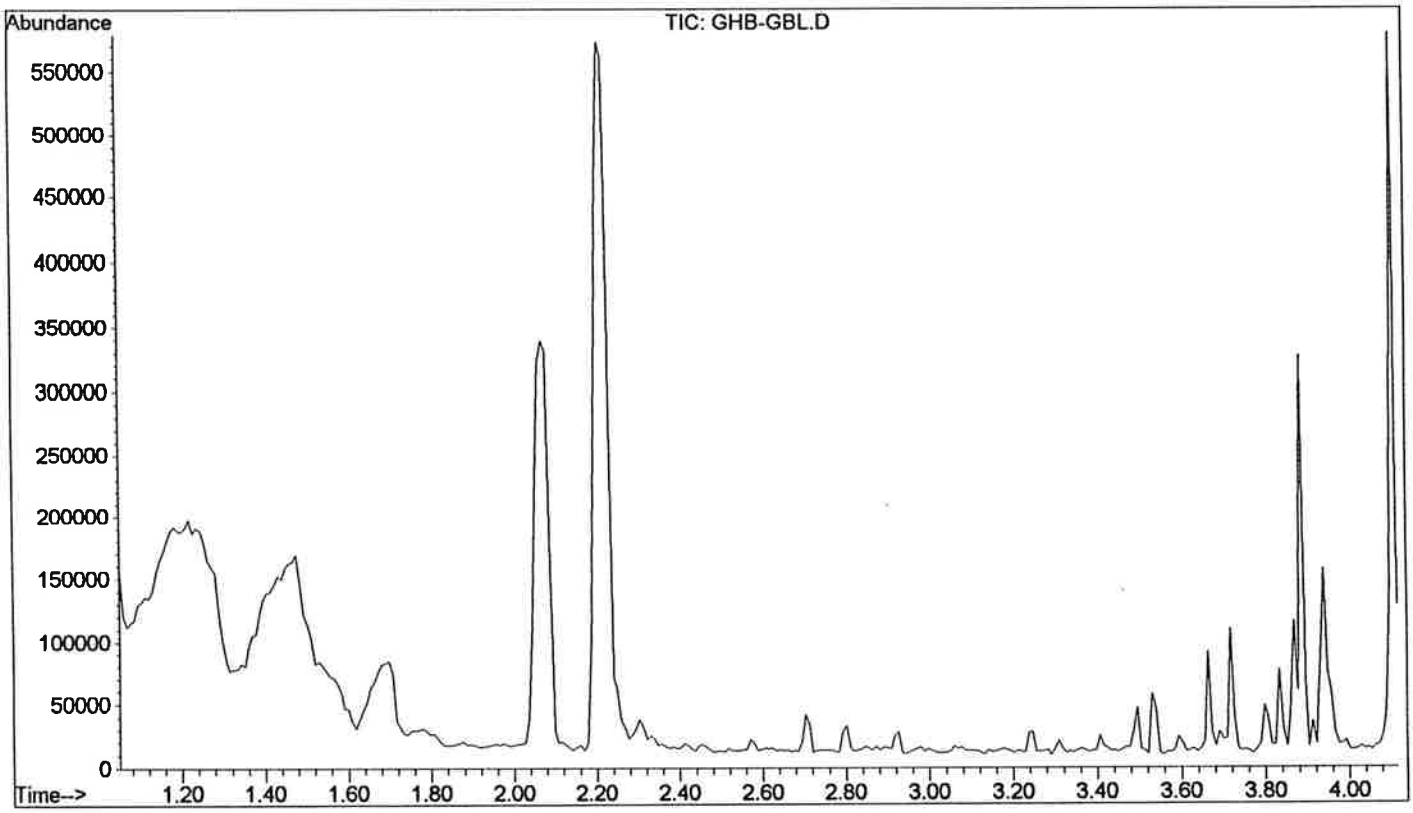
E. Analyze on GC\MS.

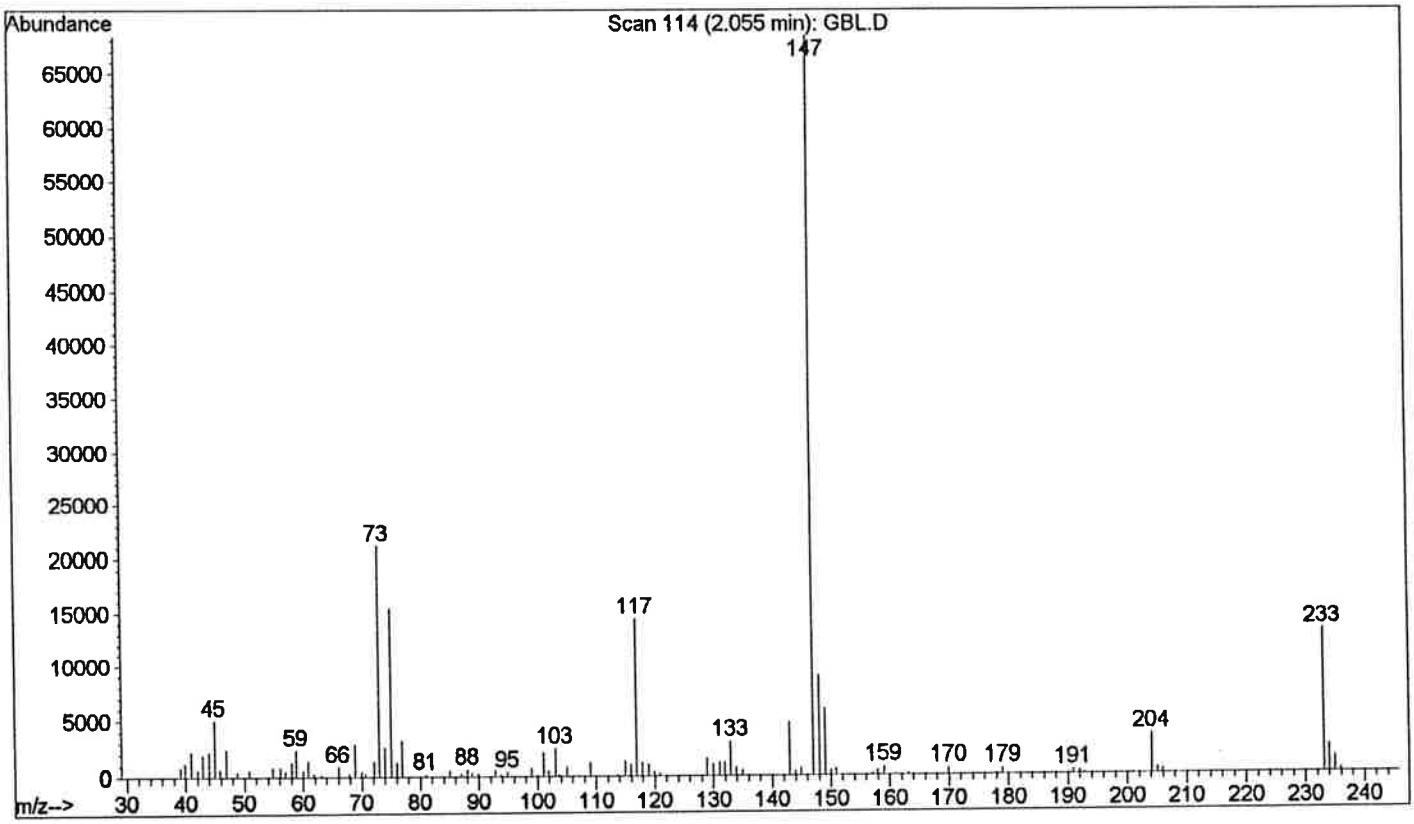
RESULTS FROM TRIAL PROCEDURE II

Three samples were prepared according to this procedure. They consisted of 20 mg of GHB and 20 mg of GBL in 10 mL of Gatorade, 20 mg of GHB and 20 mg of GBL in 10 mL of Coca-Cola, and 20 mg of GHB in 10 mL of Gatorade.

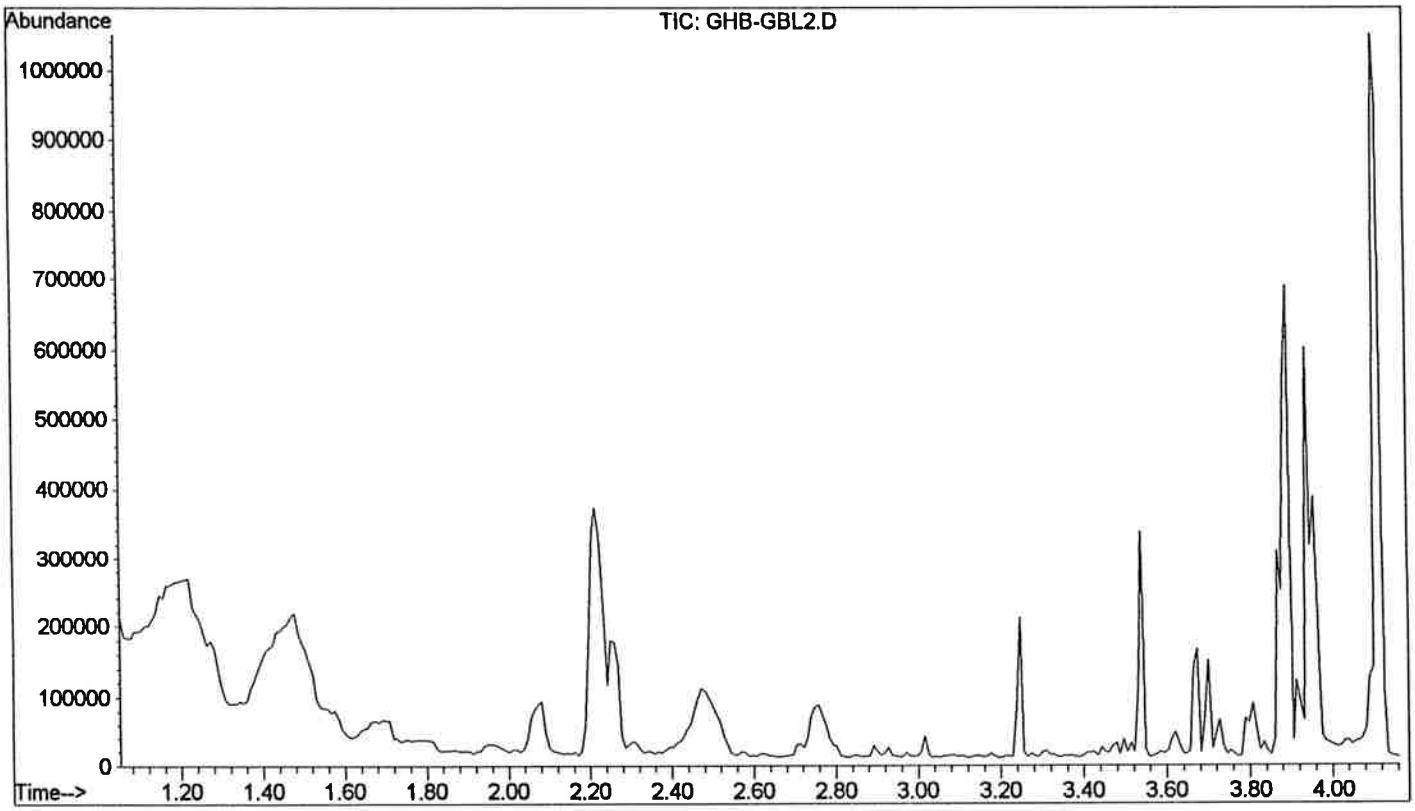
The only difference between trial procedure I and trial procedure II is that the second procedure begins with a chloroform extraction. It was already known that a chloroform extraction would remove any residual GBL, but we also wanted to find out if the extraction could clean up our sample. The samples containing GHB and GBL and the sample containing only GHB produced similar spectra when extracted with chloroform and derivatized; however, the GHB-TMS peaks in the spectra of the extracted samples were still too weak, and there were still too many additional peaks.

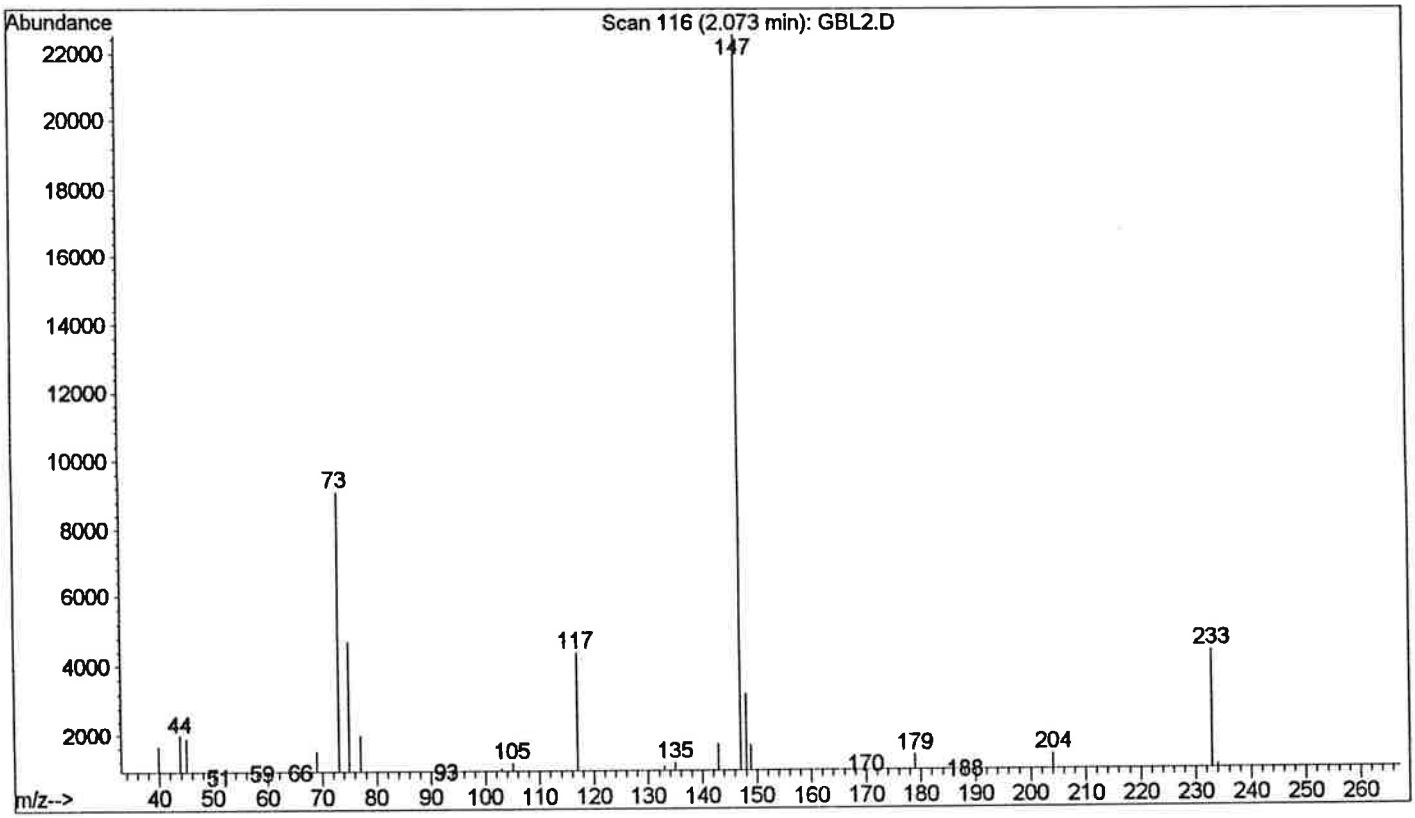
File : C:\HPCHEM\1\DATA\PWF\GHB-GBL.D
Operator : JES
Acquired : 15 Jun 1999 11:05 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: 20 mg GHB + 20 mg GBL + GATORADE
Misc Info : USING TRIAL PROCEDURE II
Vial Number: 1



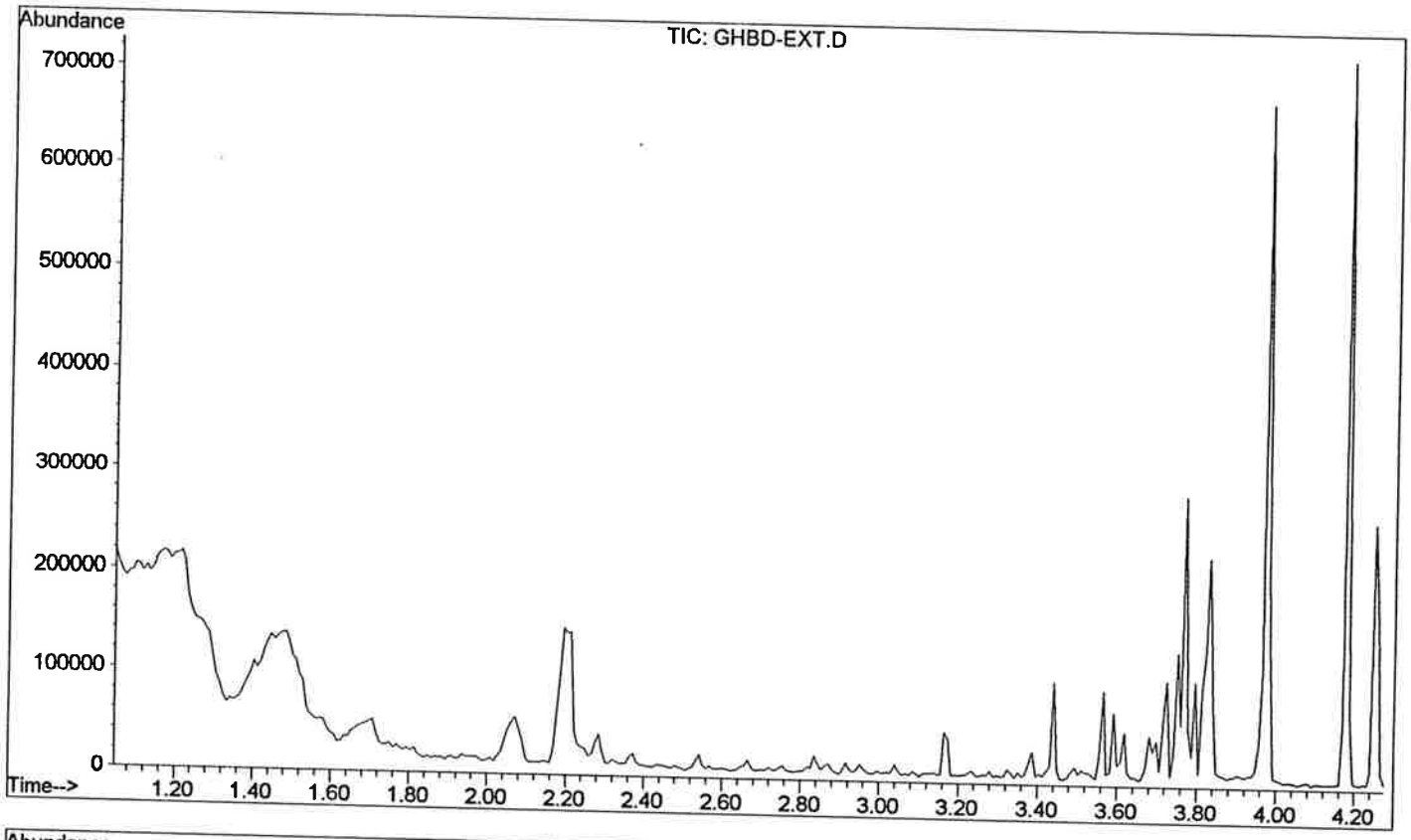


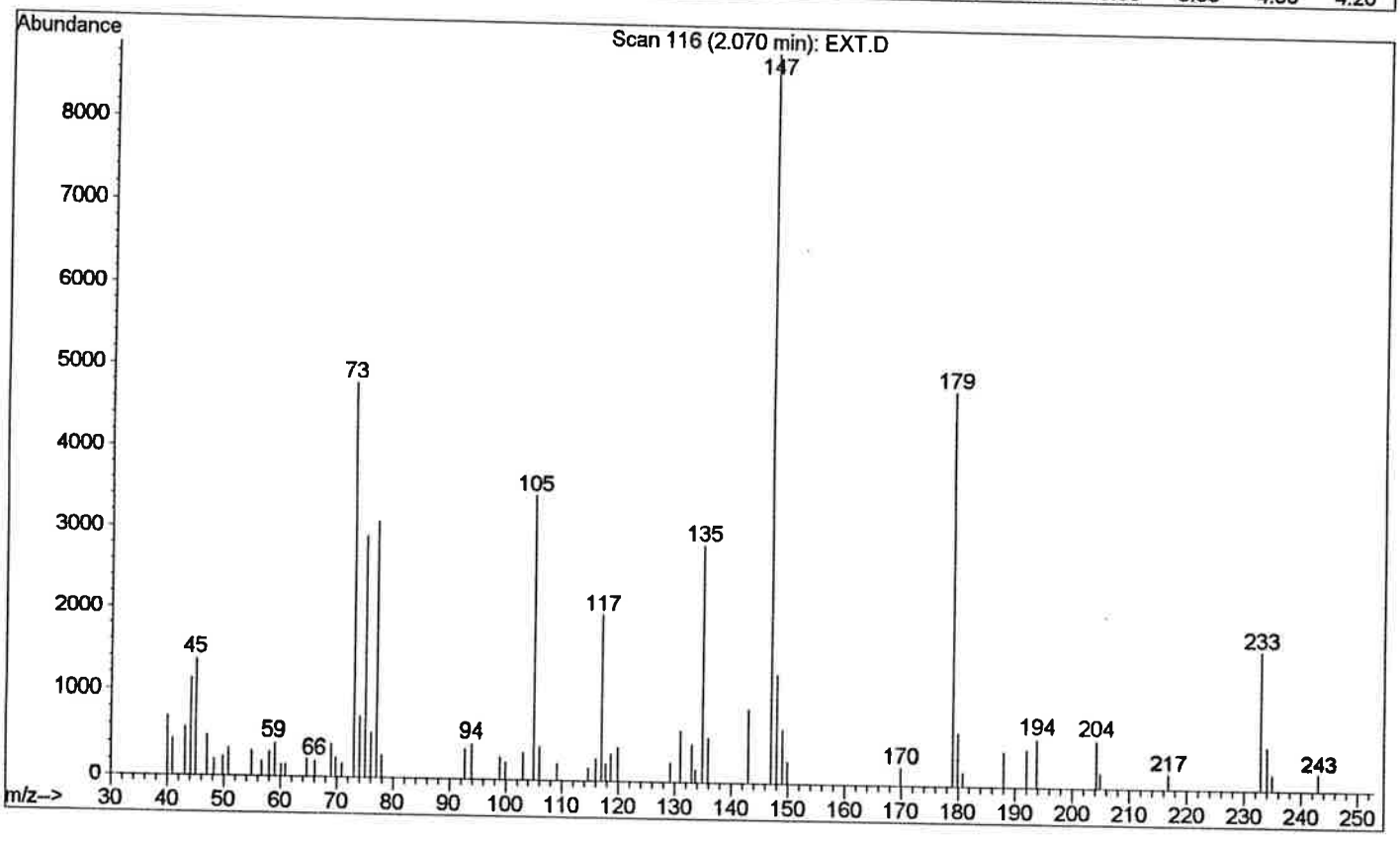
File : C:\HPCHEM\1\DATA\PWF\GHB-GBL2.D
Operator : SES
Acquired : 15 Jun 1999 11:14 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: 20 mg GHB + 20 mg GBL + 10 ml COKE
Misc Info : USING TRIAL PROCEDURE II
Vial Number: 1





File : C:\HPCHEM\1\DATA\GHBD-EXT.D
Operator : JEJ
Acquired : 15 Jun 1999 1:59 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN 10 ml GATORADE/CHCL3 EXT
Misc Info : USING TRIAL PROCEDURE II
Vial Number: 1





TRIAL PROCEDURE III

A. Extract 1-5 mL of your sample with excess $CHCl_3$. Extract once more using an equal amount of $CHCl_3$. This will remove any residual GBL.

B. Take 1-5 mL of your sample and dry it down in a small (10 mL) beaker using a hair dryer on low heat. Do not exceed 150 C or GHB may convert to GBL.

C. Recrystallize sample with acetone.

To recrystallize, add 10 mL of acetone to the beaker containing your sample. Heat the sample to boiling (b.p. of acetone-57 C) on a hot plate with constant stirring. This step should take no longer than five minutes.

D. Dry off the acetone under a hair dryer on low heat.

E. To the beaker containing your sample, add 2 mL of acetonitrile, 500 uL of BSTFA, and 500 uL of TCMS.

F. Transfer the entire contents of the beaker to an autosampler vial and cap it. Heat the vial in a 60 C-80 C water bath for 10-15 minutes.

G. Analyze on GC\MS.

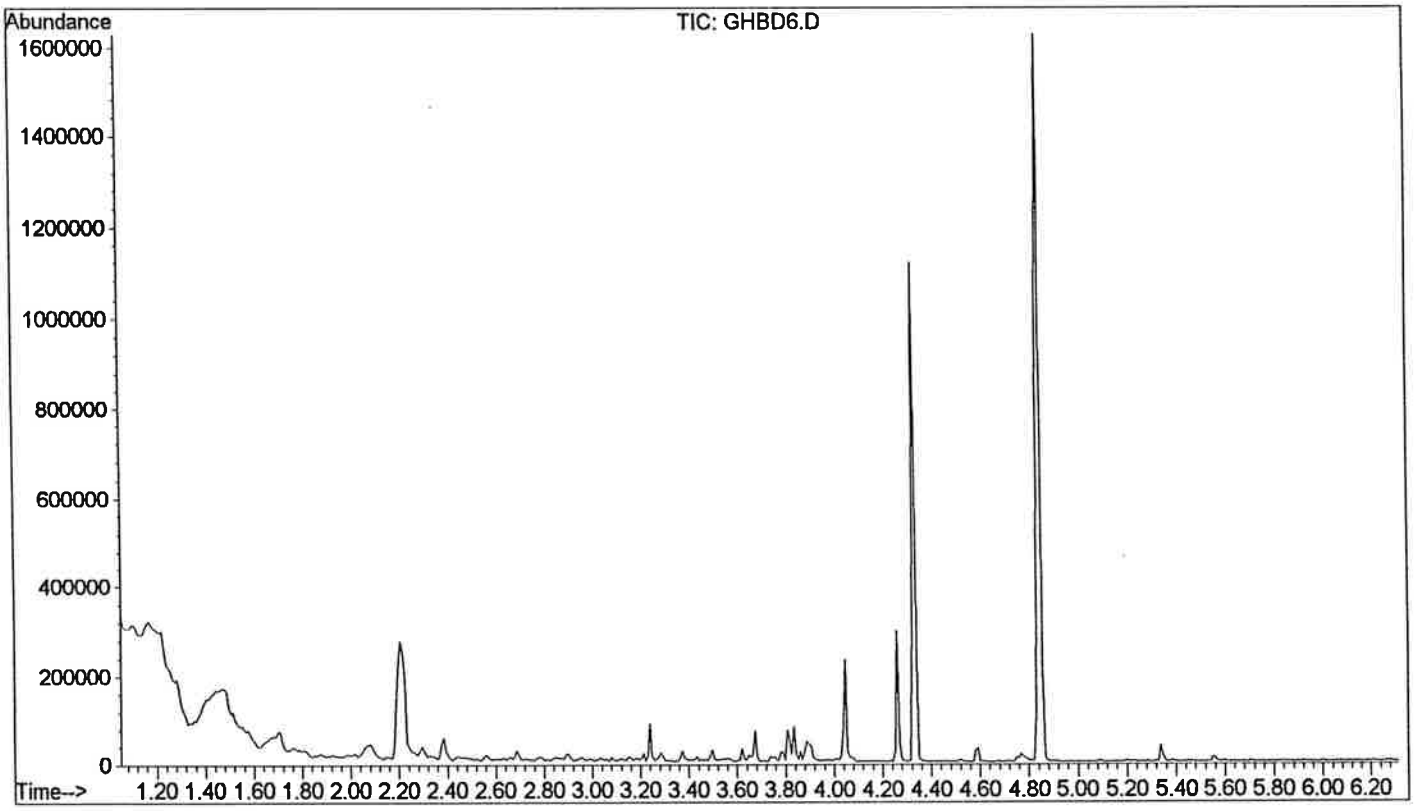
RESULTS FROM TRIAL PROCEDURE III

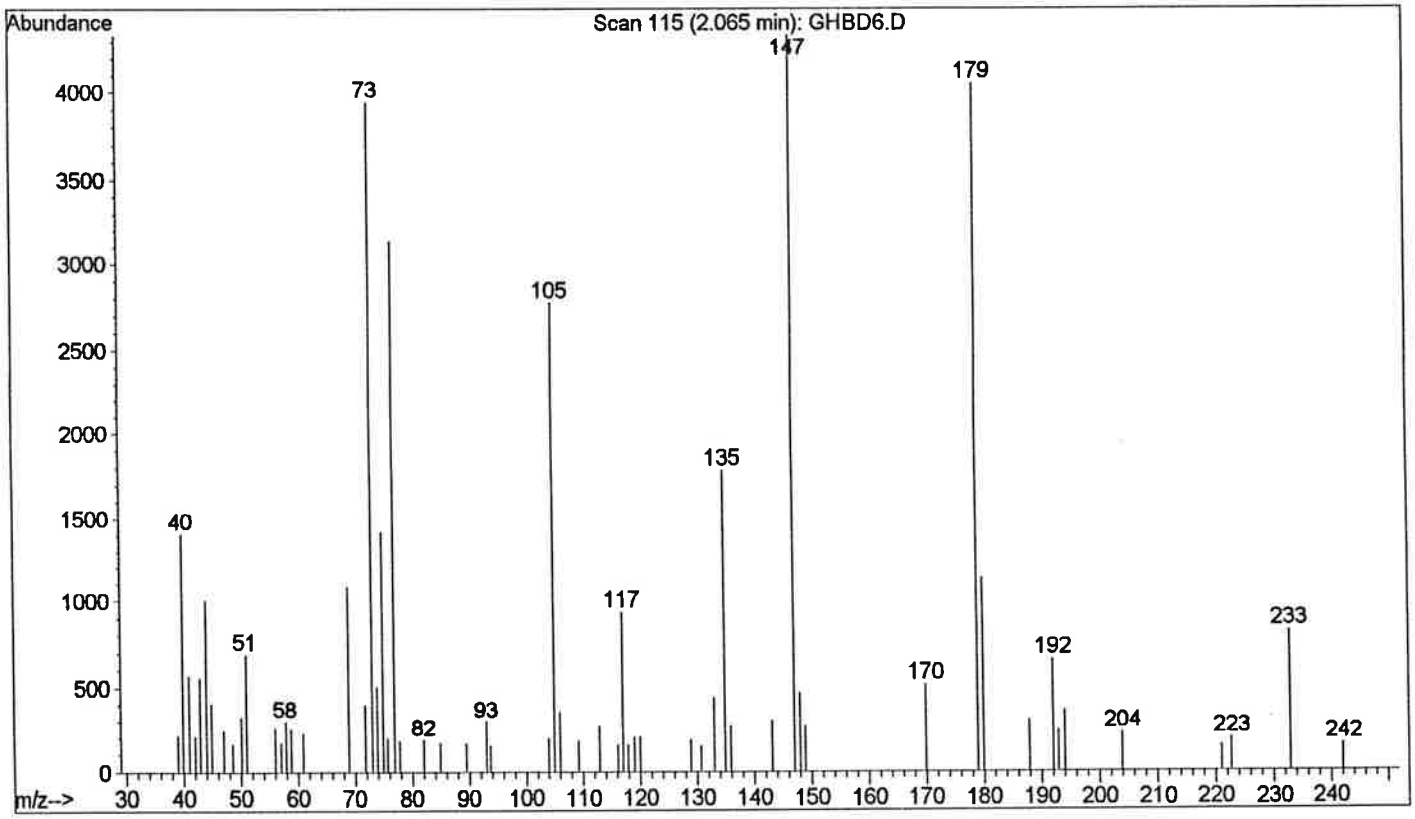
The only difference in trial procedure II and trial procedure III is a recrystallizing step. The object is to find a cold solvent that your impurities are soluble in, while the substance meant to be isolated is only soluble if the solvent is heated. Upon cooling down, impurities do not fit in the pure crystalline structure of the solid. However, because we are starting our recrystallization with a syrup instead of an impure solid, our end product is not pure.

The recrystallization is followed by derivatization, and the solution is then analyzed. Using this procedure, eight samples were analyzed. Seven of the samples were 20 mg of GHB in 10 mL of Gatorade, and the other sample was 20 mg of GHB in 10 mL of Coca-Cola. The GHB-TMS peak was always present, but it was still too weak. The

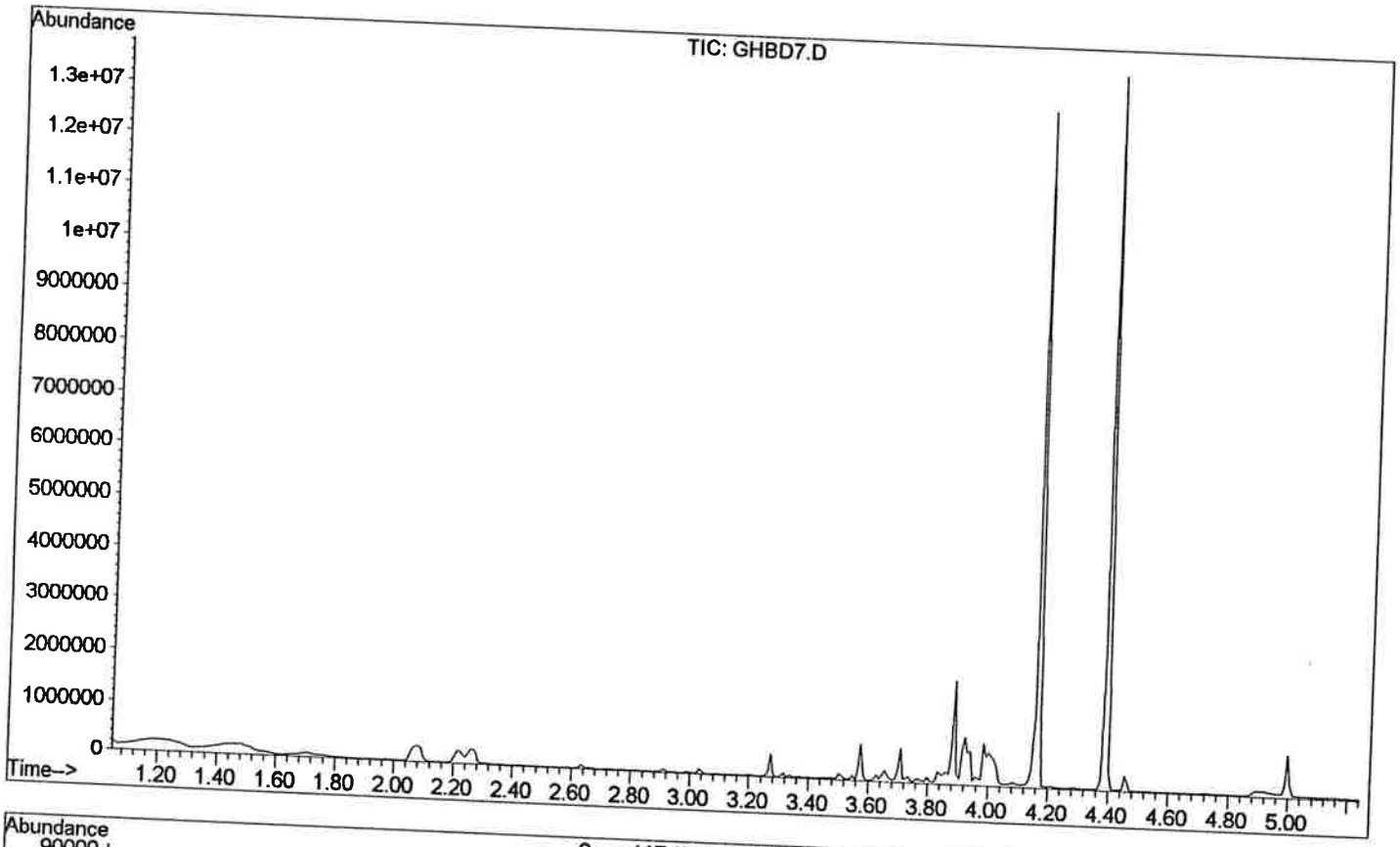
recrystallization step did clean up some of the samples, but it was too inconsistent to be reliable.

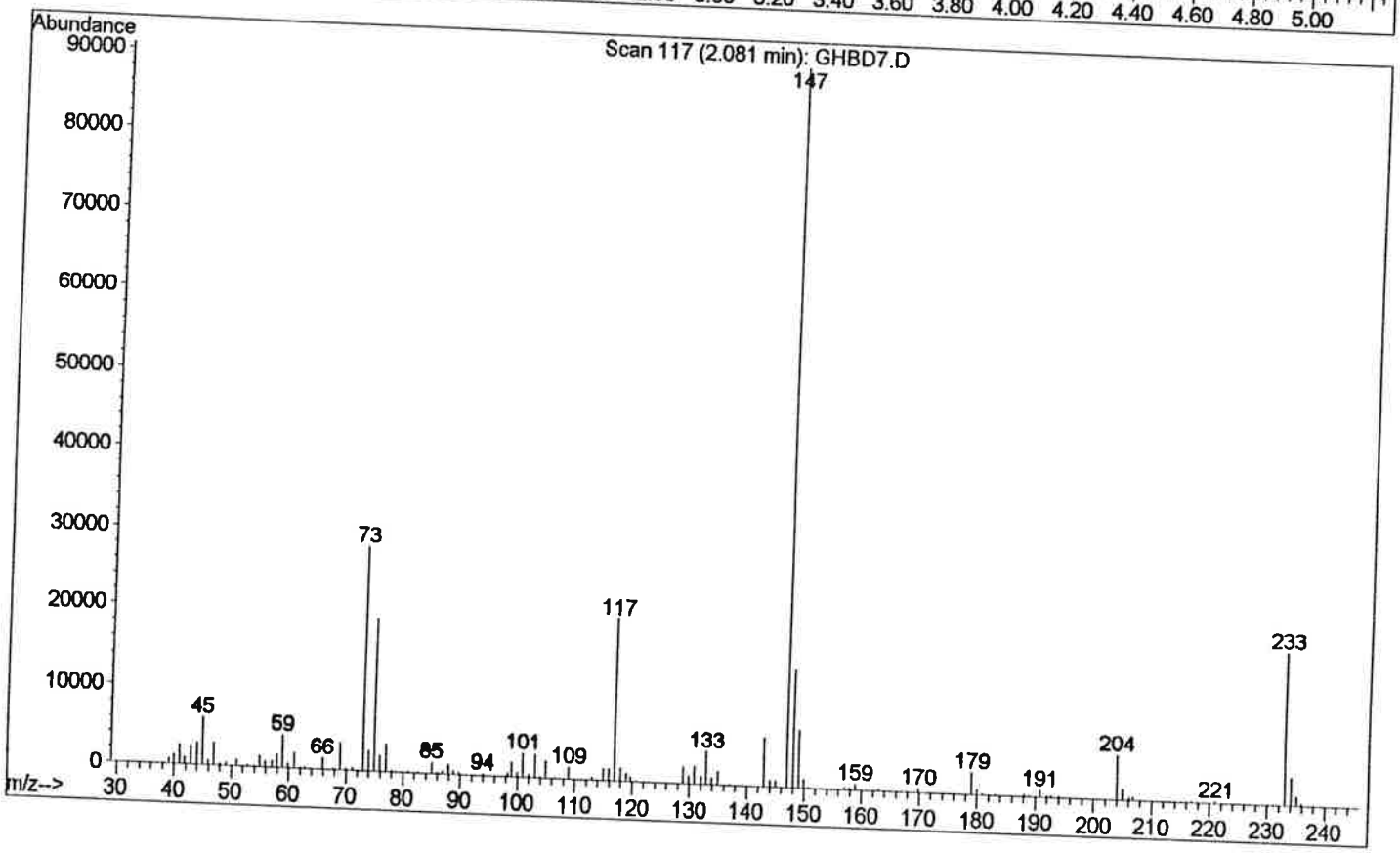
File : C:\HPCHEM\1\DATA\PWF\GHBD6.D
Operator : SES
Acquired : 15 Jun 1999 3:00 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE/CHCL3 EXT ACETONE RECRYST
Misc Info : USING TRIAC PROCEDURE III
Vial Number: 1



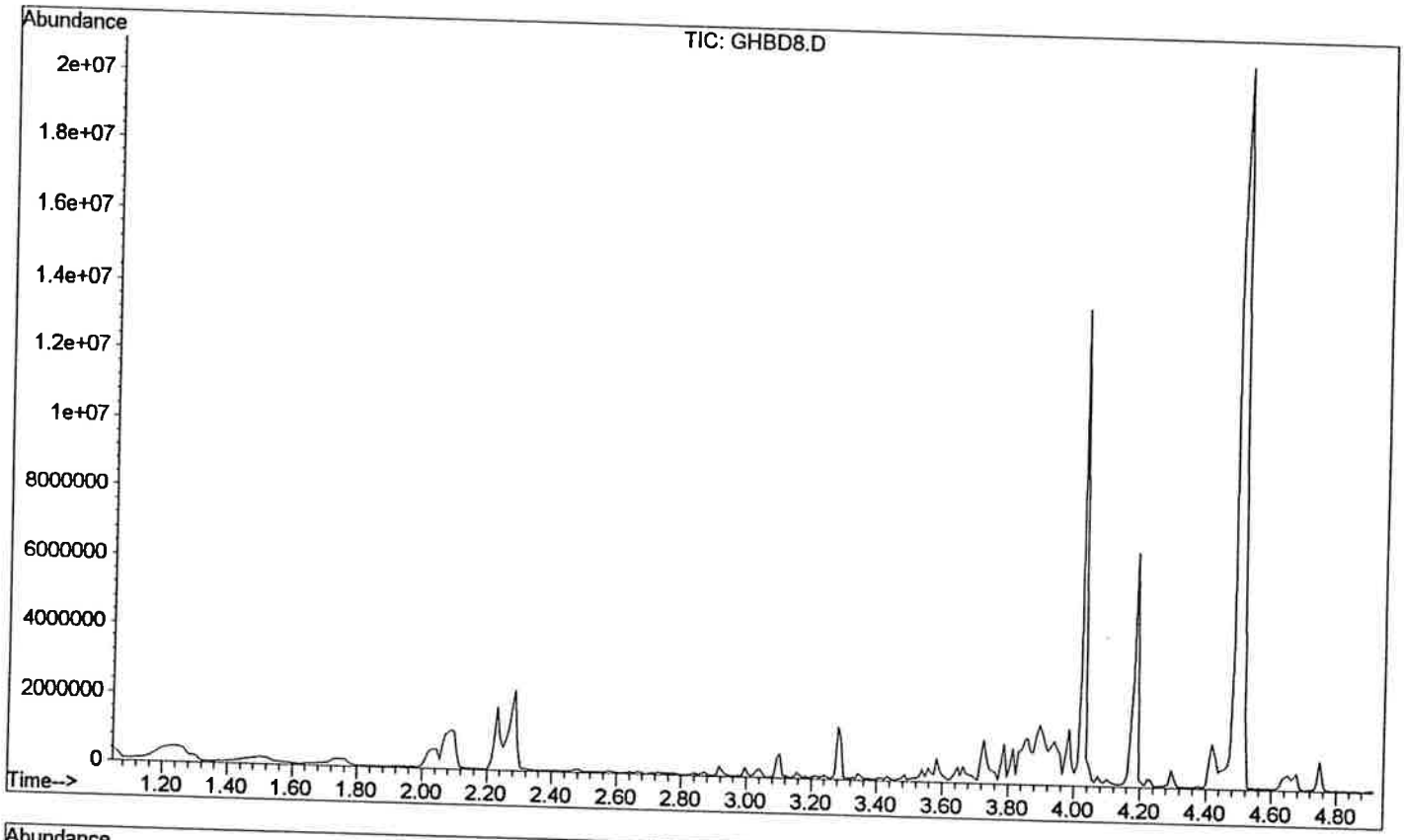


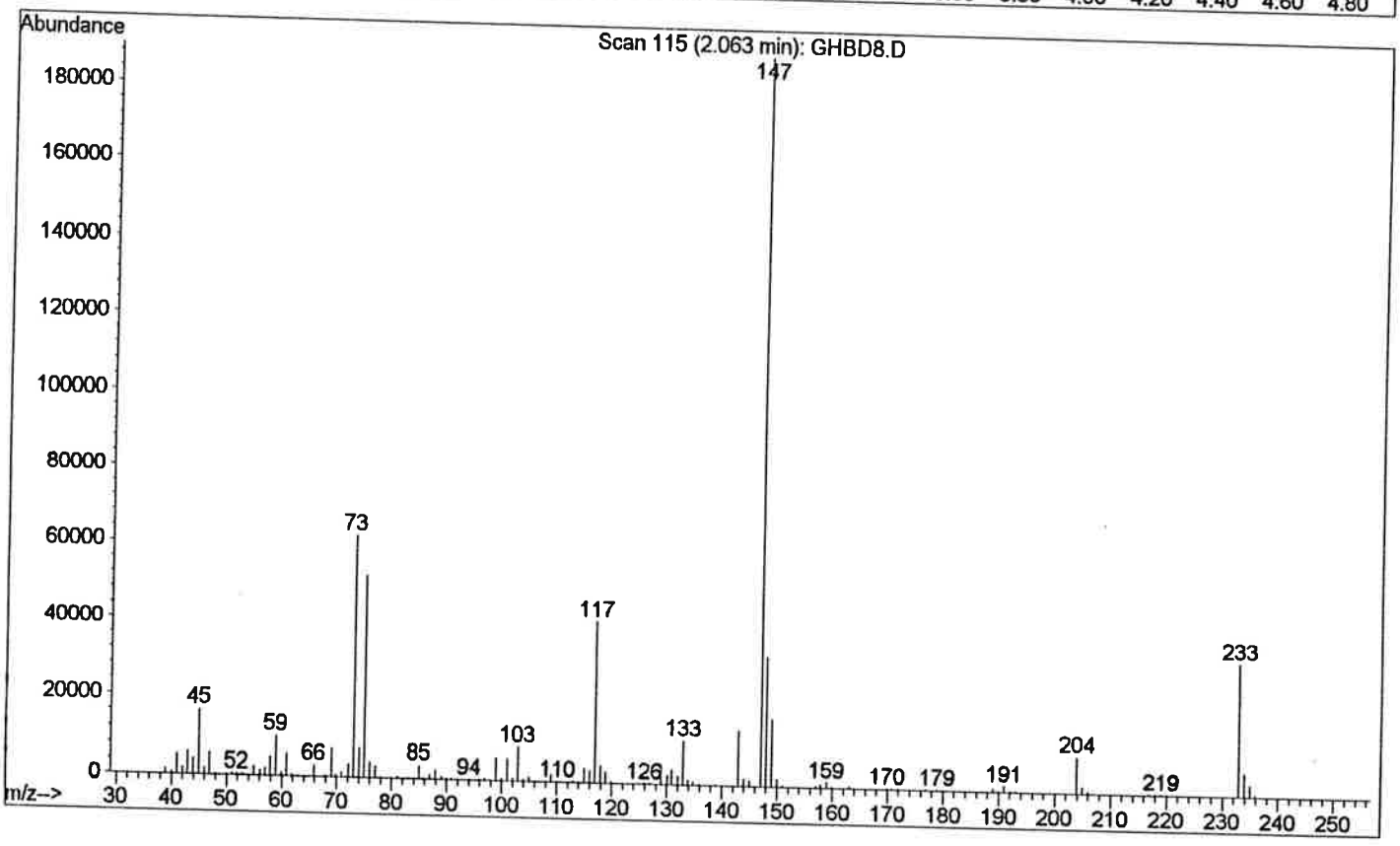
File : C:\HPCHEM\1\DATA\PWF\GHBD7.D
Operator : JEJ
Acquired : 16 Jun 1999 8:06 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE/CHCL3 EXT ACETONE RECRY
Misc Info : USING TRIAL PROCEDURE III
Vial Number: 1



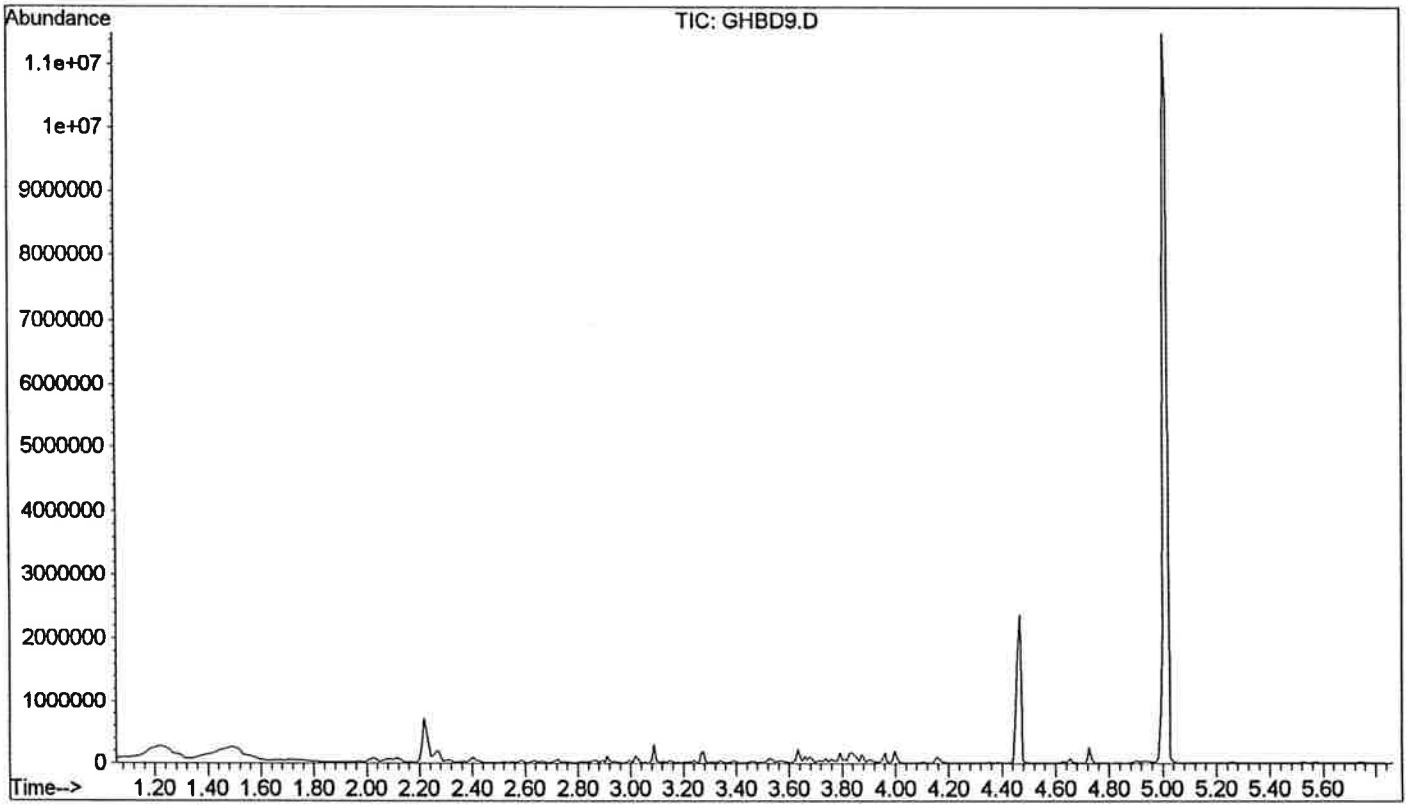


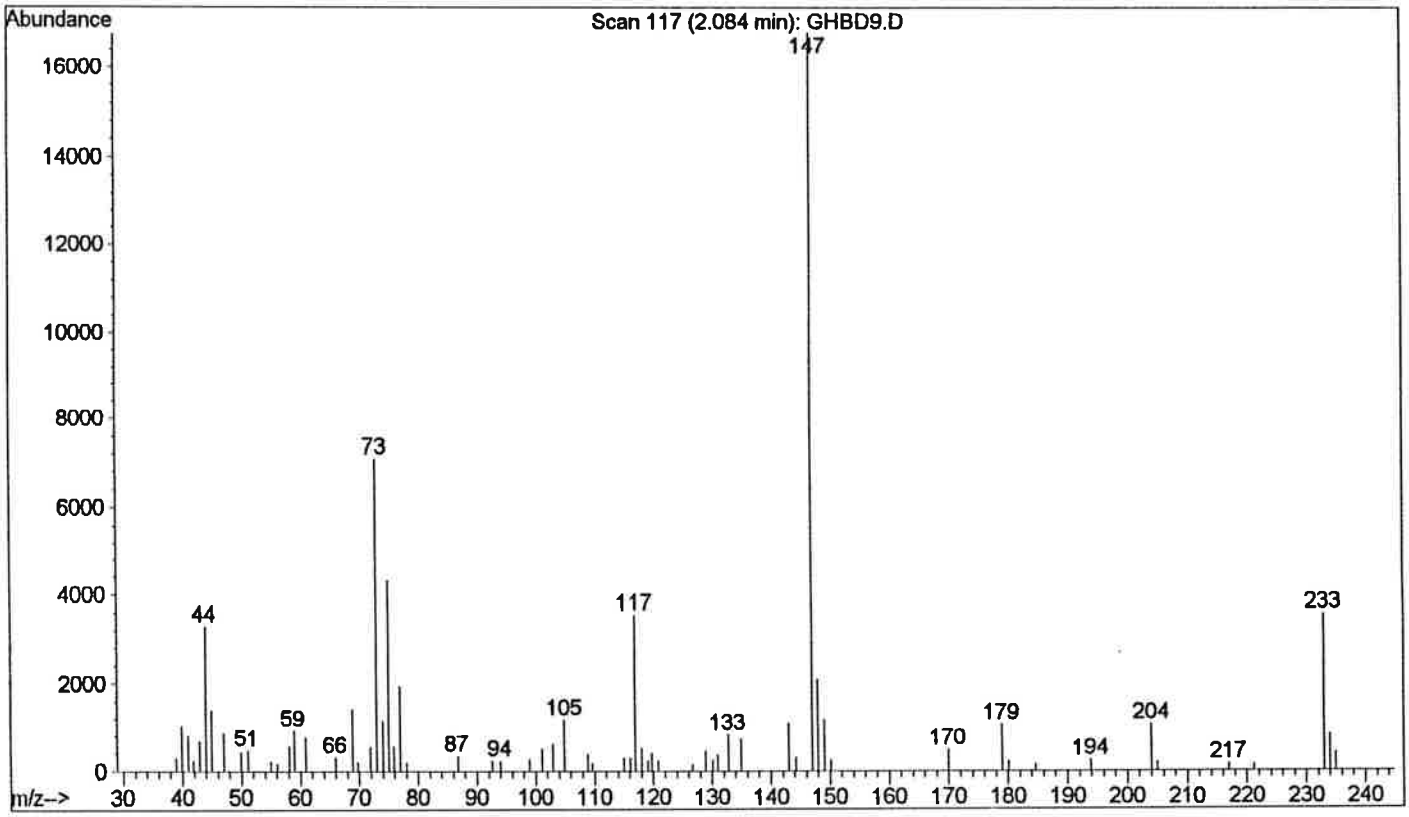
File : C:\HPCHEM\1\DATA\PWF\GHBD8.D
Operator : JEJ
Acquired : 16 Jun 1999 10:17 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAL PROCEDURE III
Vial Number: 1



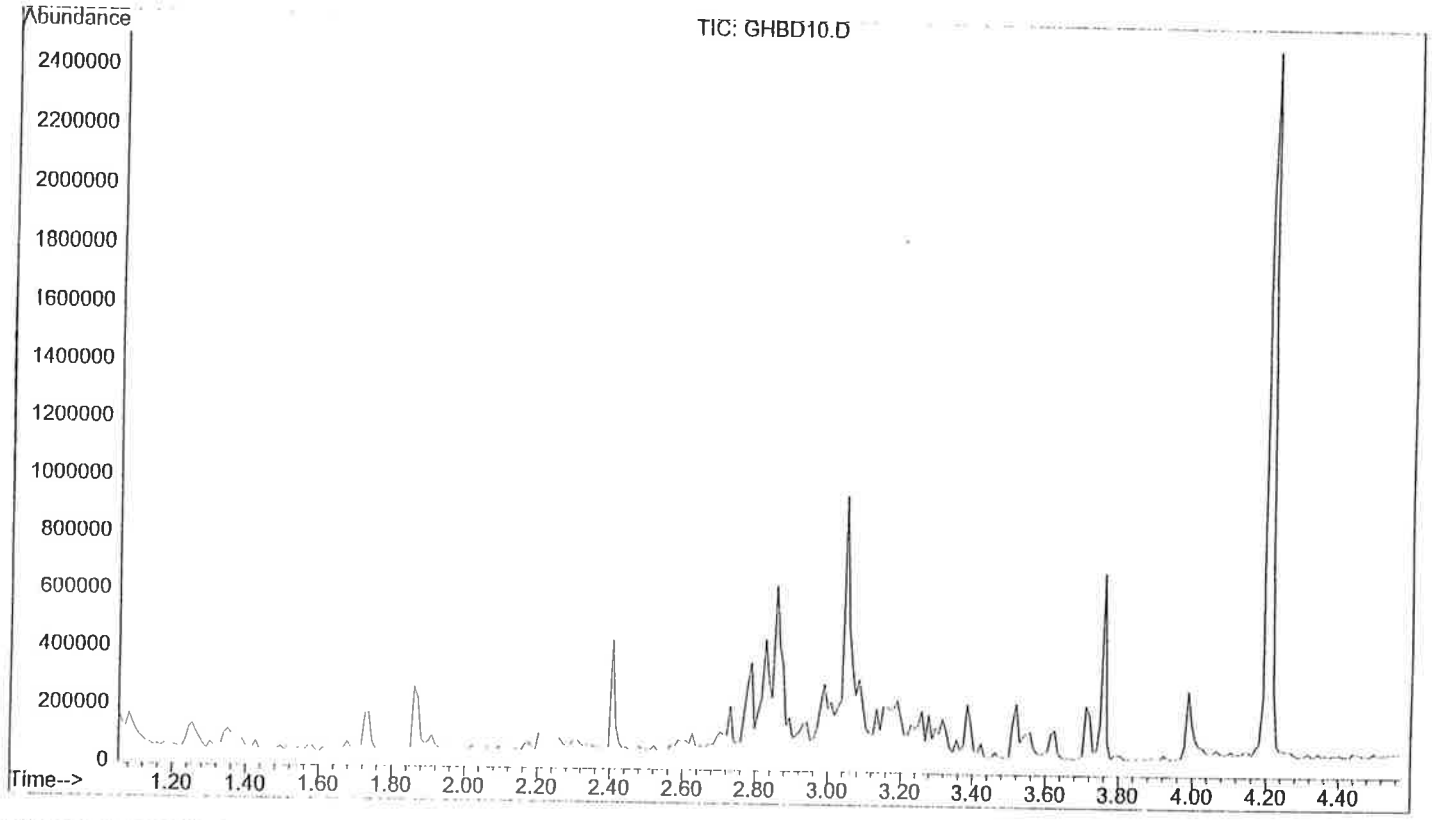


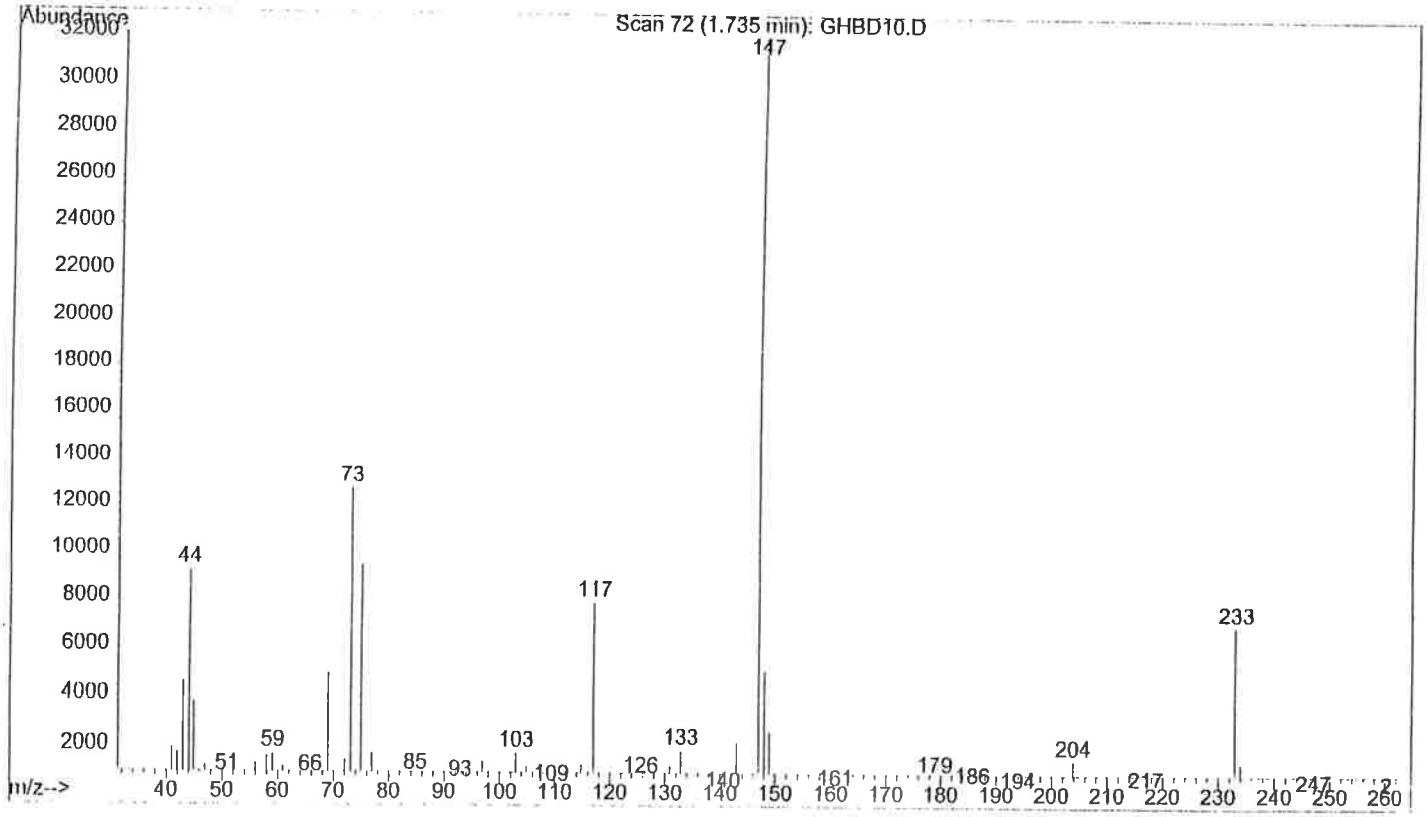
File : C:\HPCHEM\1\DATA\PWF\GHBD9.D
Operator : JES
Acquired : 16 Jun 1999 11:04 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAC PROCEDURE III
Vial Number: 1



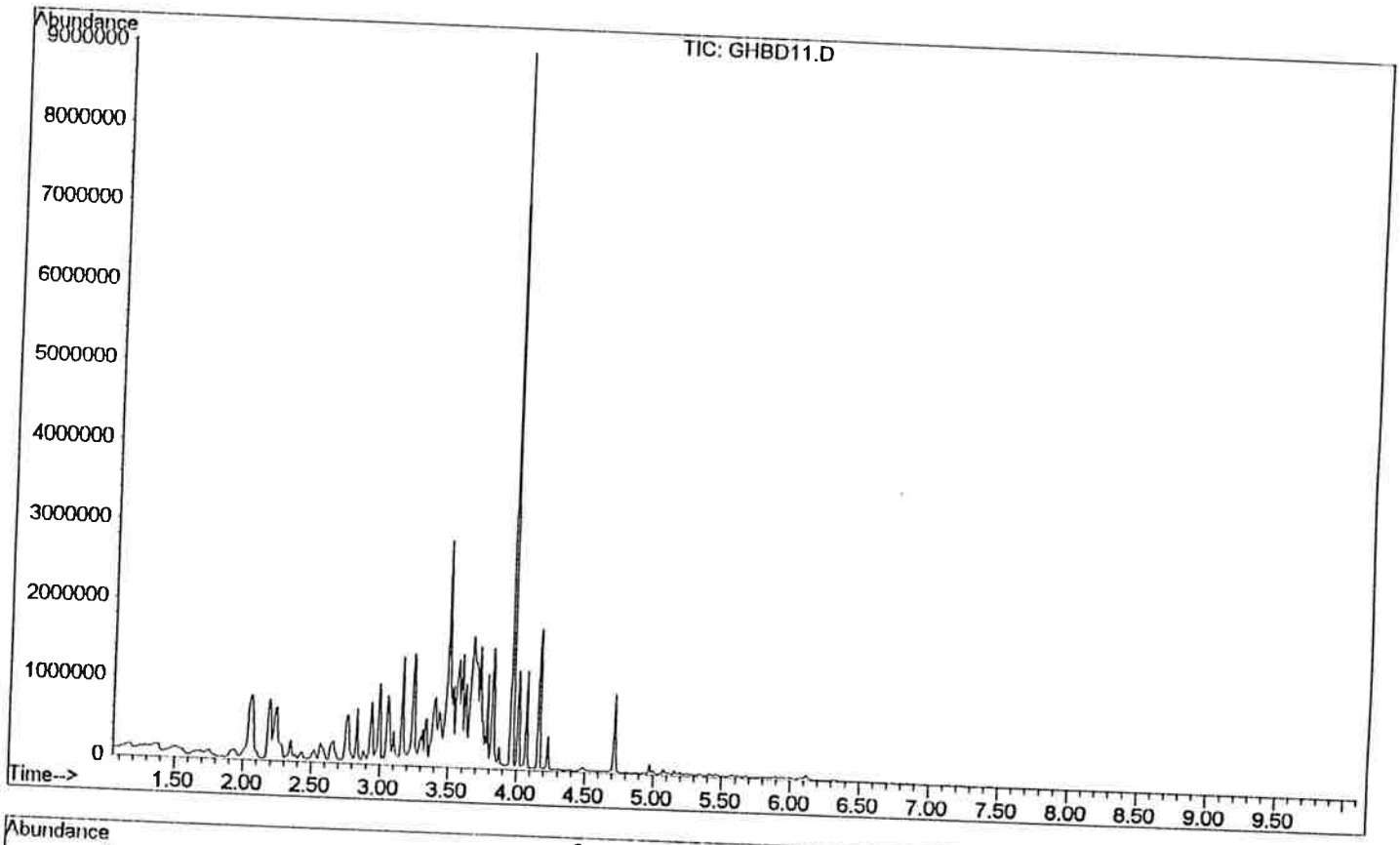


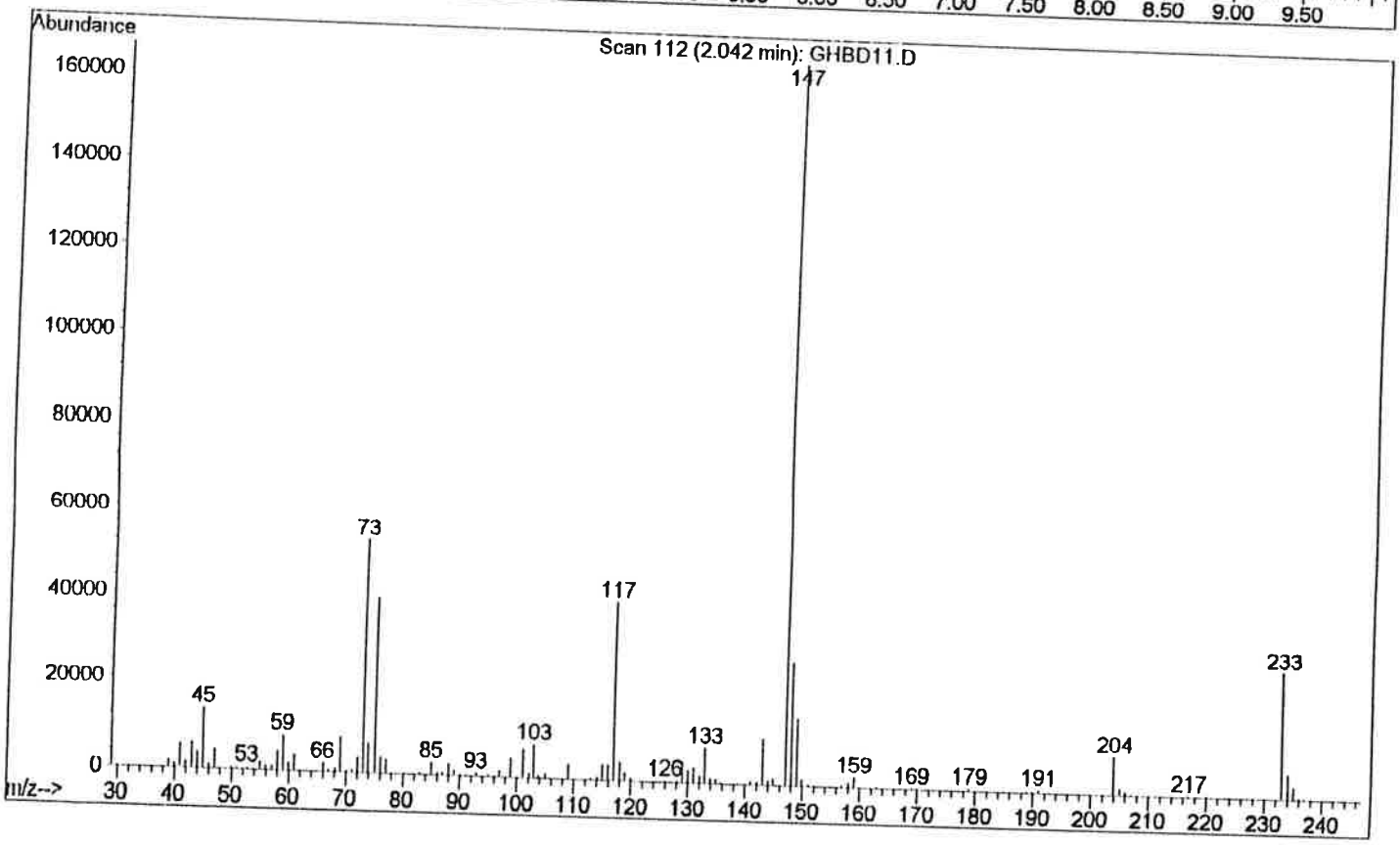
File : C:\HPCHEM\1\DATA\PWF\GHBD10.D
Operator : JEJ
Acquired : 16 Jun 1999 14:39 using AcqMethod MSDRUGS
Instrument : Donna
Sample Name: GHB IN GATORADE
Misc Info : USING TRIM PROCEDURE III
Vial Number: 1



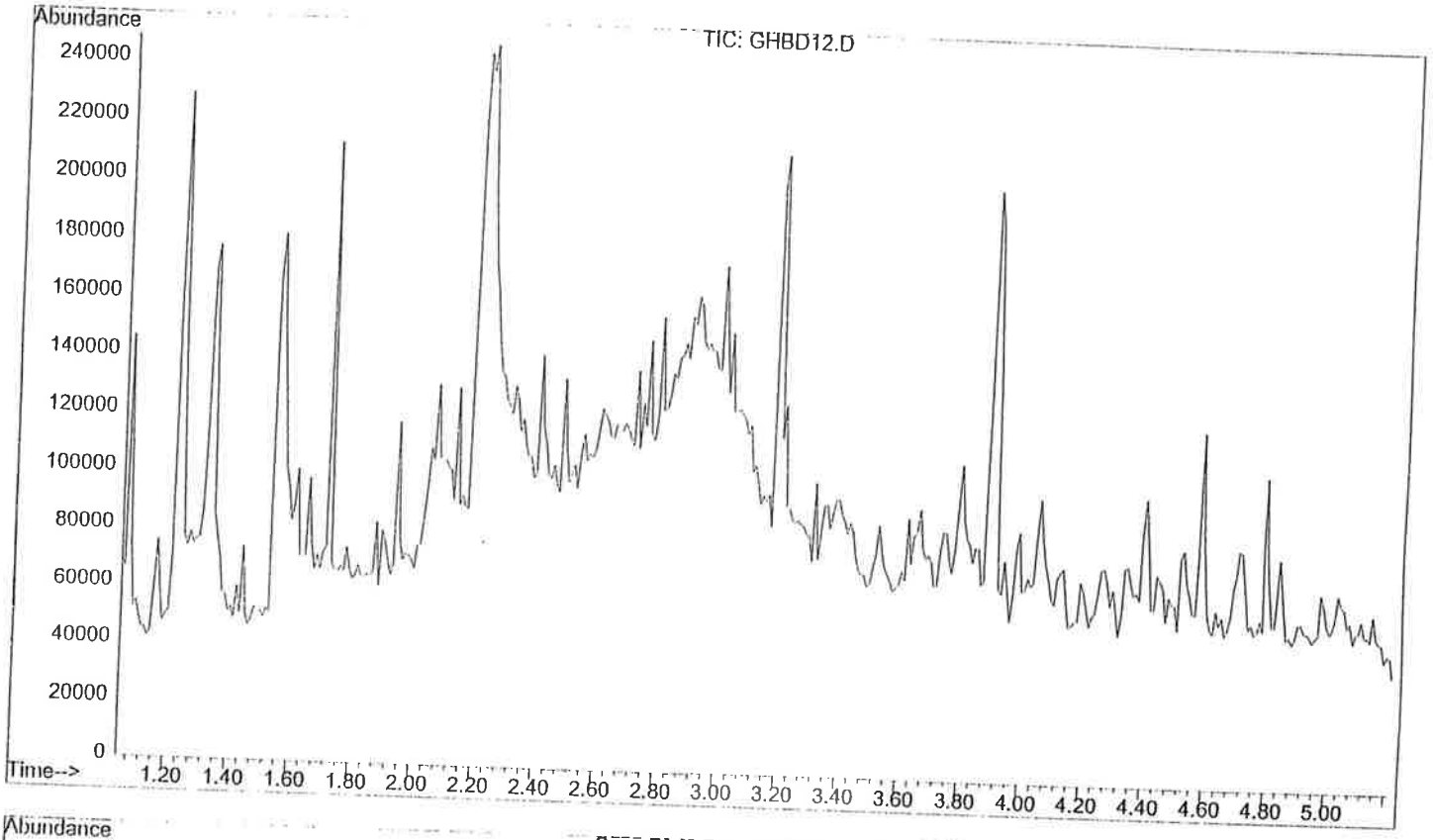


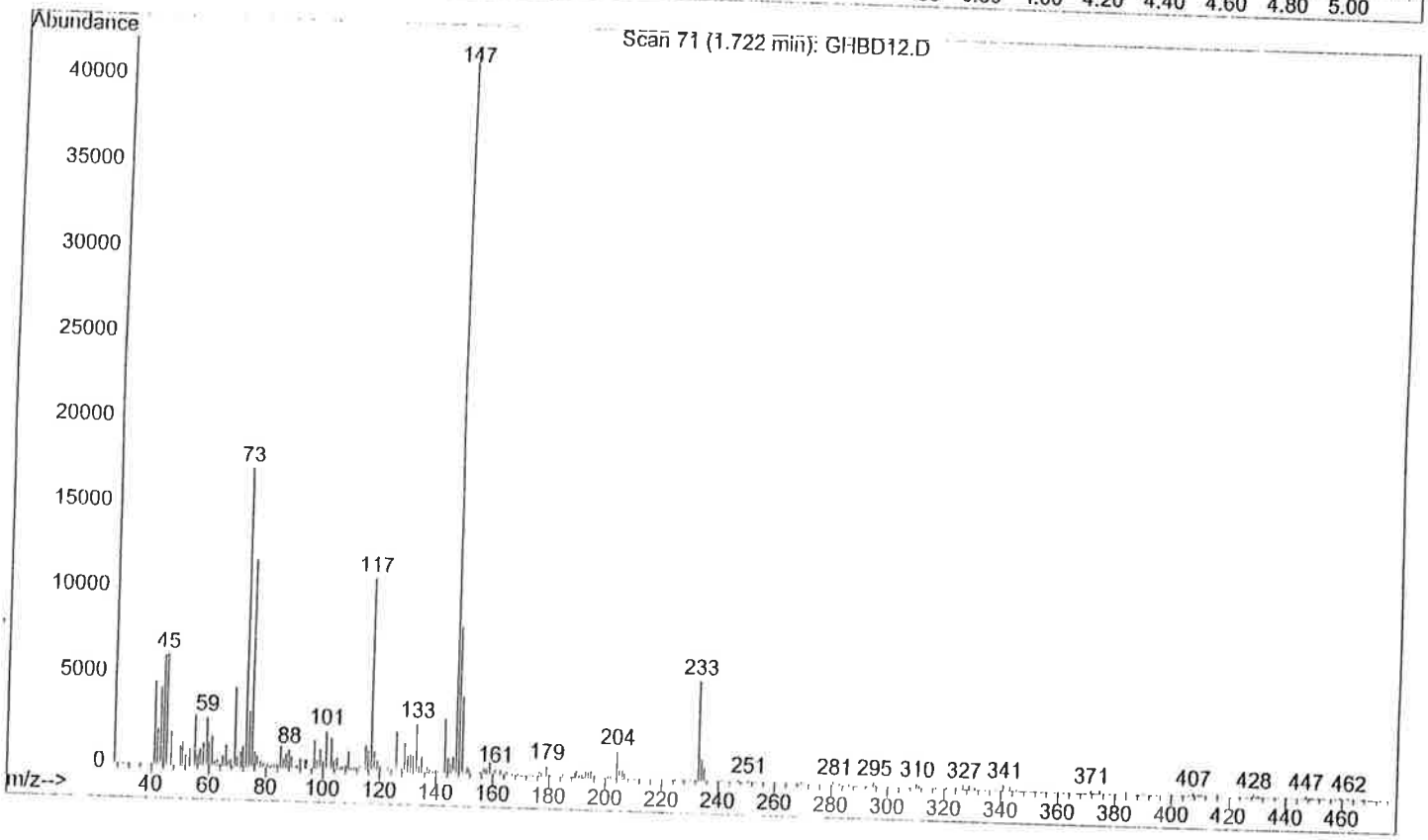
File : C:\HPCHEM\1\DATA\PWF\GHBD11.D
Operator : JET
Acquired : 17 Jun 1999 12:50 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAC PROCEDURE III
Vial Number: 1



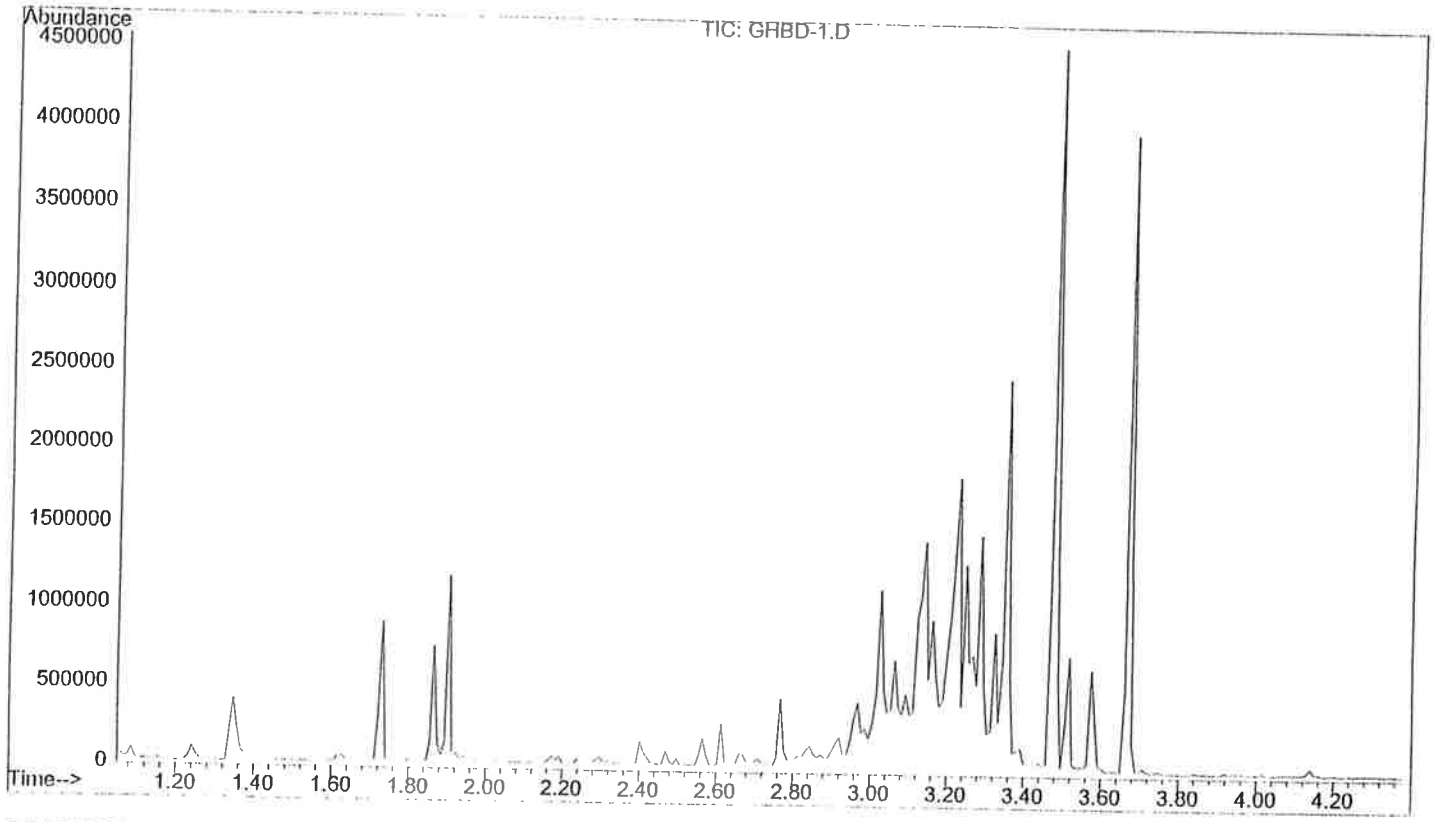


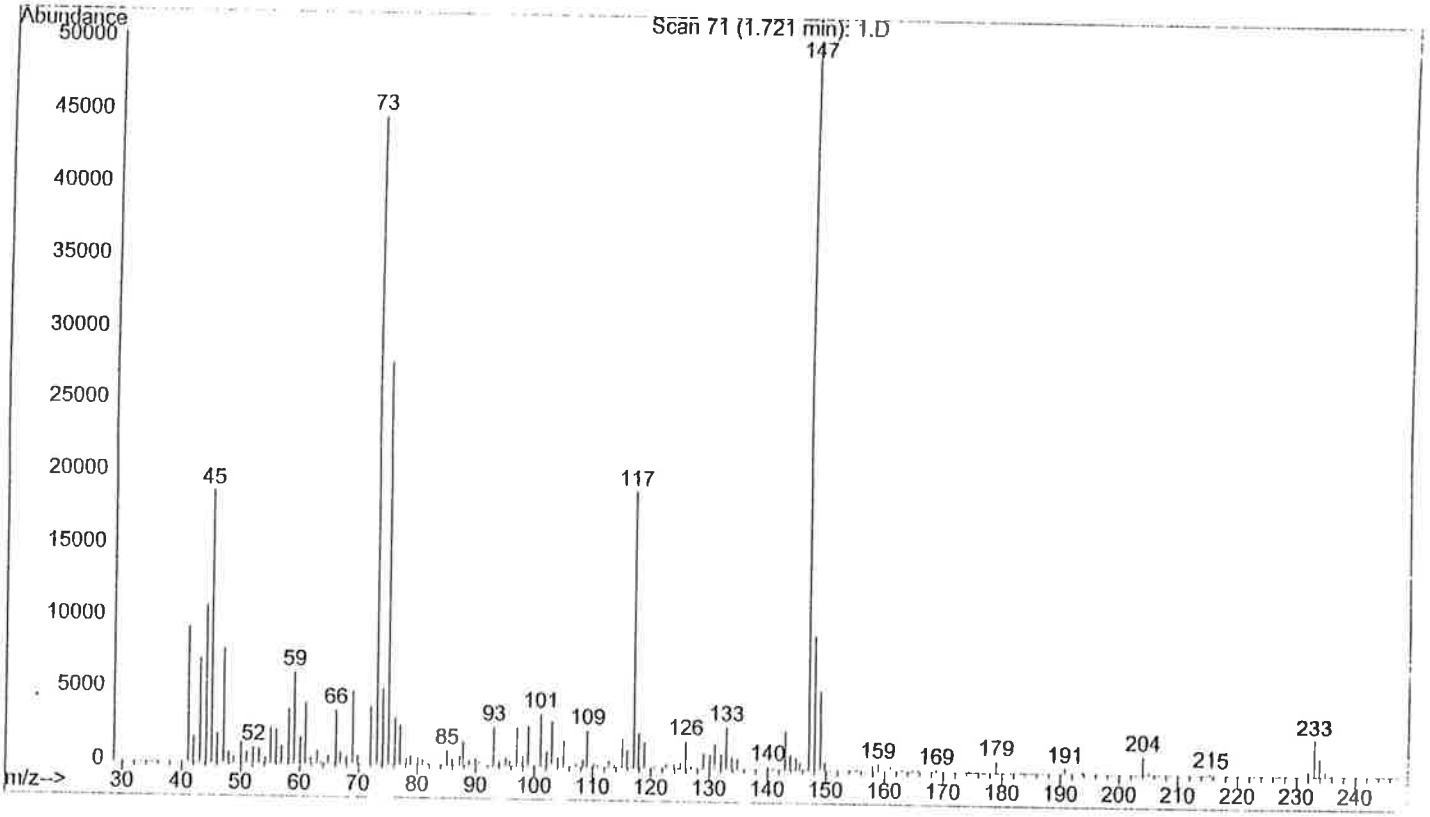
File : C:\HPCHEM\1\DATA\PWF\GHBD12.D
Operator : JES
Acquired : 17 Jun 1999 14:15 using AcqMethod MSDRUGS
Instrument : Donna
Sample Name: GHBD IN COKE
Misc Info : USING TRIMC PROCEDURE III
Vial Number: 1





File : C:\HPCHEM\1\DATA\PWF\GHBD-1.D
Operator : JEJ
Acquired : 21 Jun 1999 10:58 using AcqMethod MSDRUGS
Instrument : Donna
Sample Name: GHB IN GATORADE
Misc Info : USING TRIM PROCEDURE III
Vial Number: 1





TRIAL PROCEDURE IV

A. Extract 1-5 mL of your sample with excess $CHCl_3$. Extract once more using an equal amount of $CHCl_3$. This will remove any residual GBL.

B. Take 1-5 mL of your sample and dry it down in a small (10 mL) beaker using a hair dryer on low heat. Do not exceed 150 C or GHB may convert to GBL.

C. Recrystallize sample with acetone. (There is no need to recrystallize if your sample will dry down)

   To recrystallize, add 10 mL of acetone to the beaker containing your sample. Heat the sample to boiling (b.p. of acetone-57 C) on a hot plate with constant stirring. This step should take no longer than five minutes. Immediately pour the hot solvent into another small beaker leaving the syrup behind.

D. Dry off the acetone under a hair dryer on low heat.

E. To the beaker containing your sample, add 2 mL of acetonitrile, 500 uL of BSTFA, and 500 uL of TCMS.

F. Transfer the entire contents of the beaker to an autosampler vial and cap it. Heat the vial in a 60 C-80 C water bath or heat covered on a hot plate at about 70 C for 10-15 minutes.

G. Analyze on GC\MS.

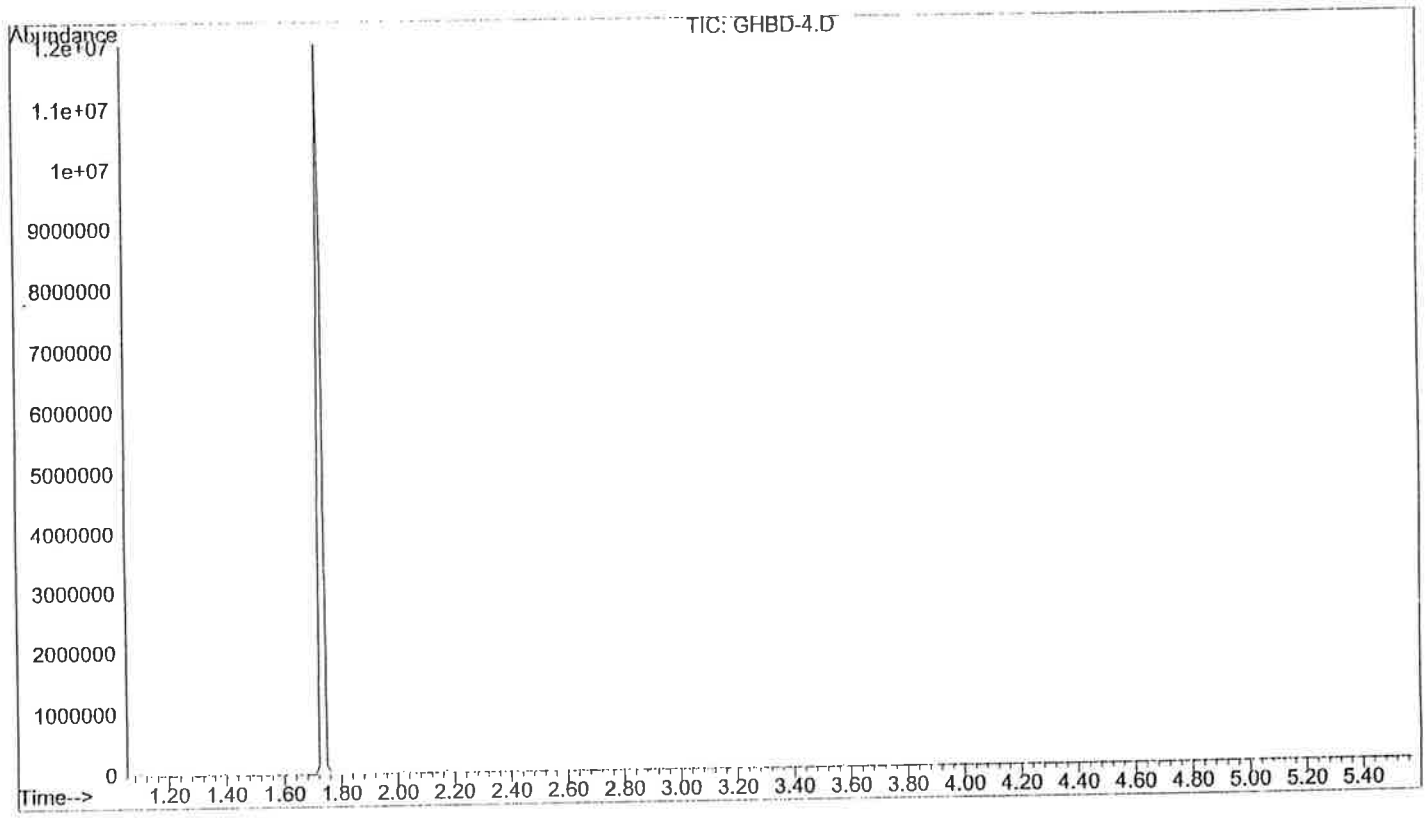
RESULTS FROM TRIAL PROCEDURE IV

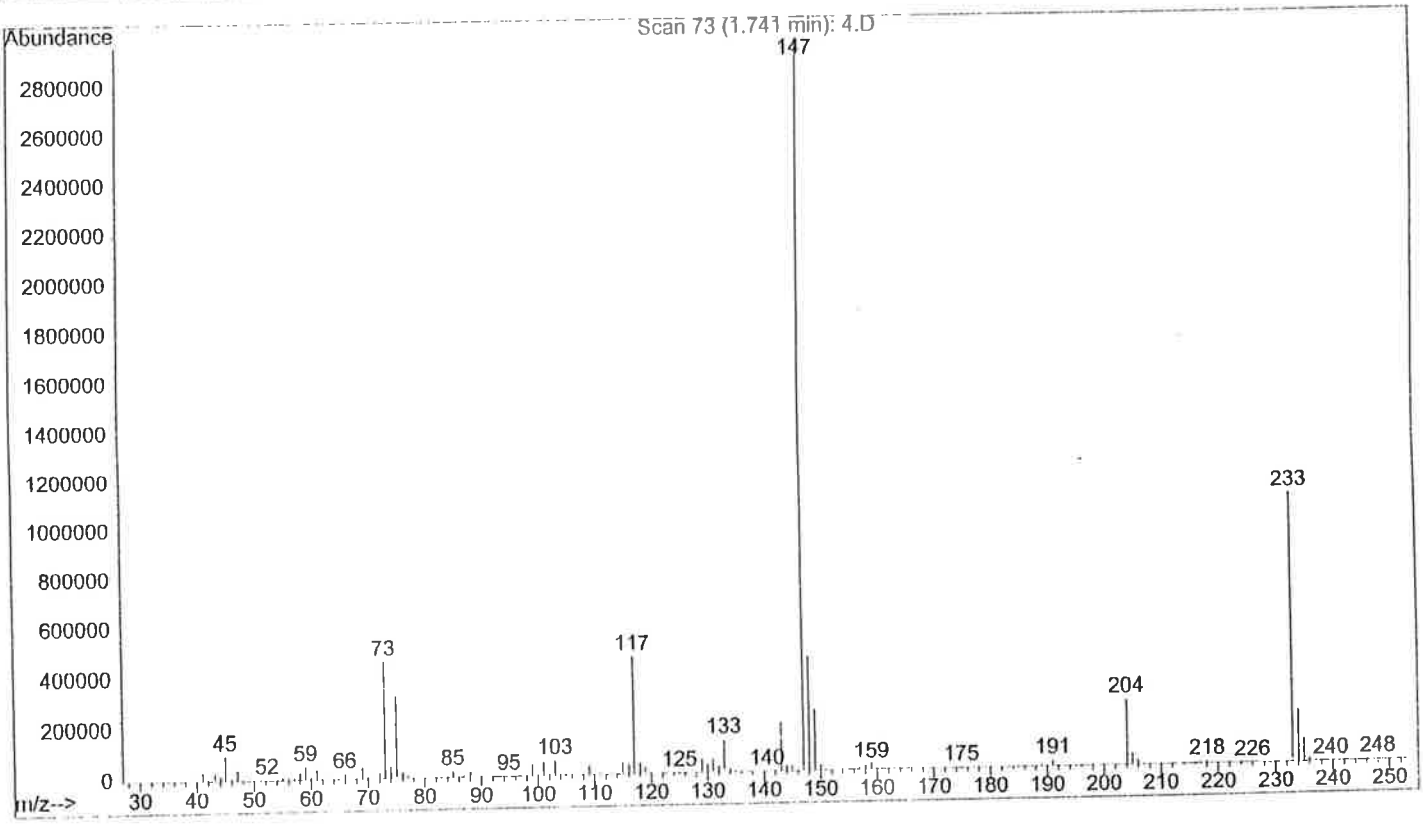
The difference in this procedure is in the recrystallizing step you pour your hot solvent only into another beaker. This leaves some impurities, but it allows more of the GHB to be exposed to the derivatizing agents. The acetone also helps in drying the sample.

Seventeen samples were run using this procedure. One consisted of 20 mg of GHB in 10 mL of water, seven samples were of 20 mg of GHB in 10 mL of Gatorade, six samples were of 20 mg of GHB in 10 mL of Coca-Cola, and three samples were of 20 mg of GHB in 10 mL of a rum and Coke mixture; the mixture was made up of 1 oz. of rum

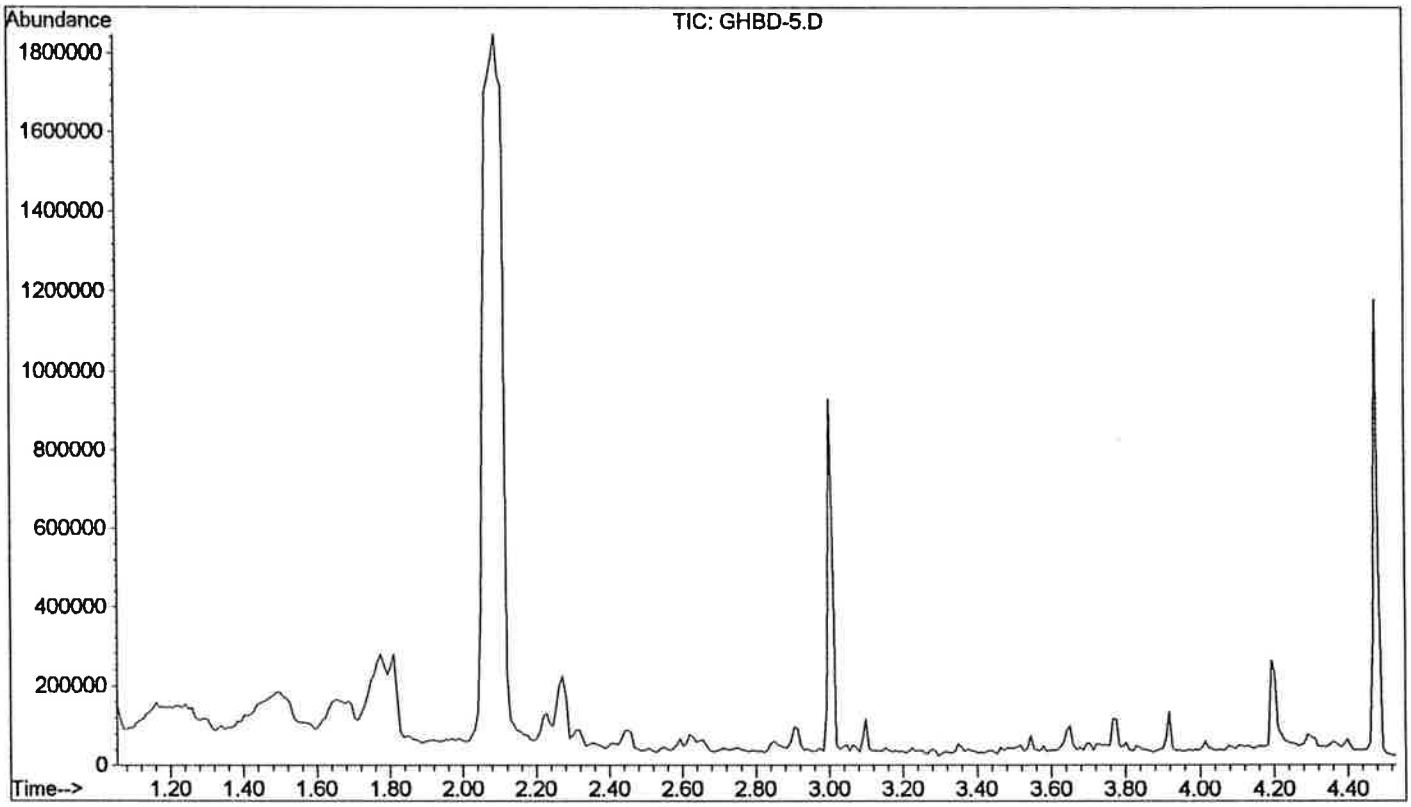
and 200 mL of Coke. All of the samples produced a much stronger GHB-TMS peak and were consistently cleaner.

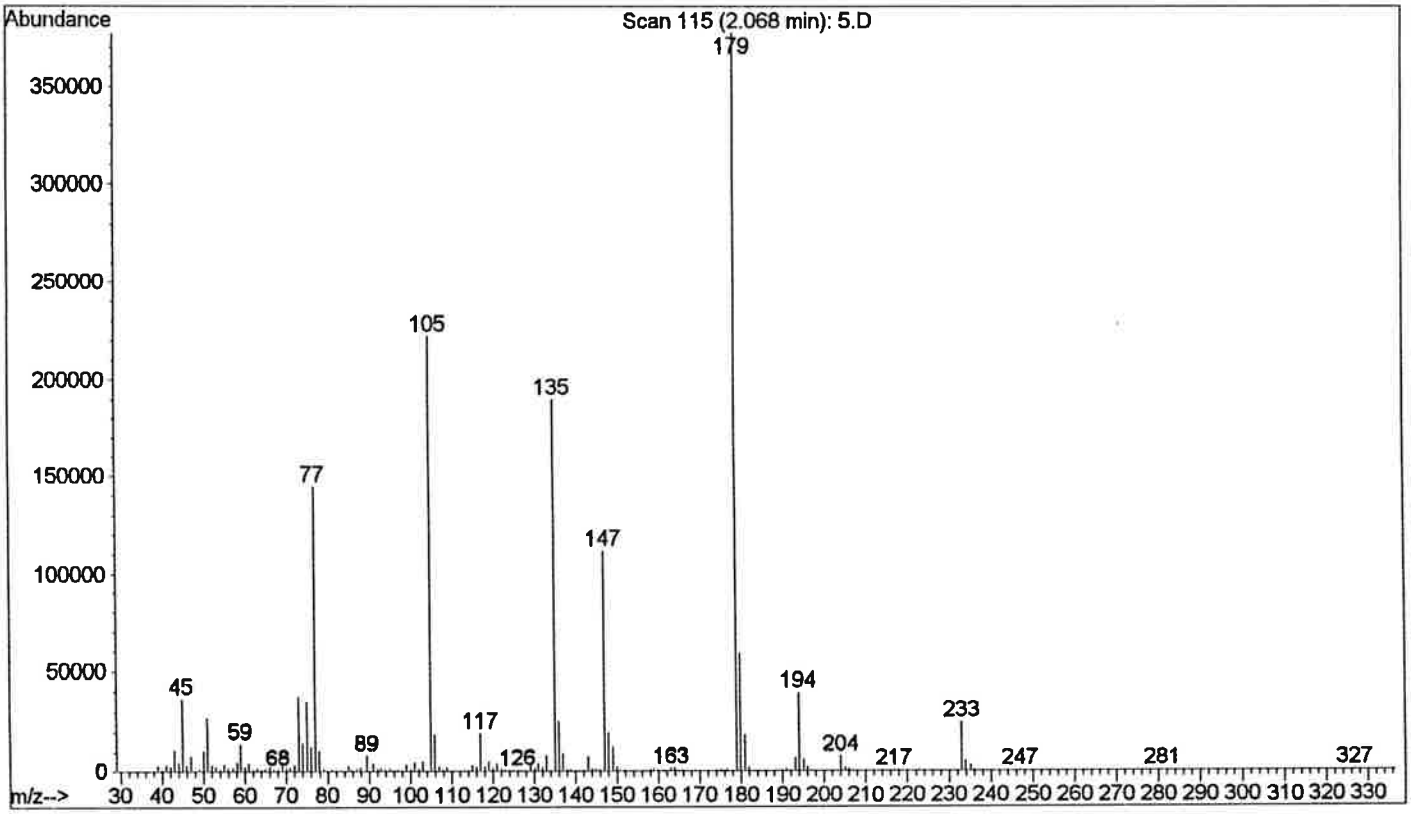
File : C:\HPCHEM\1\DATA\PWF\GHBD-4.D
Operator : JEJ
Acquired : 21 Jun 1999 13:59 using AcqMethod MSDRUGS
Instrument : Donna
Sample Name: GHB IN WATER/DERIVATIVE
Misc Info : USING TRIPLE PROCEDURE IV
Vial Number: 1



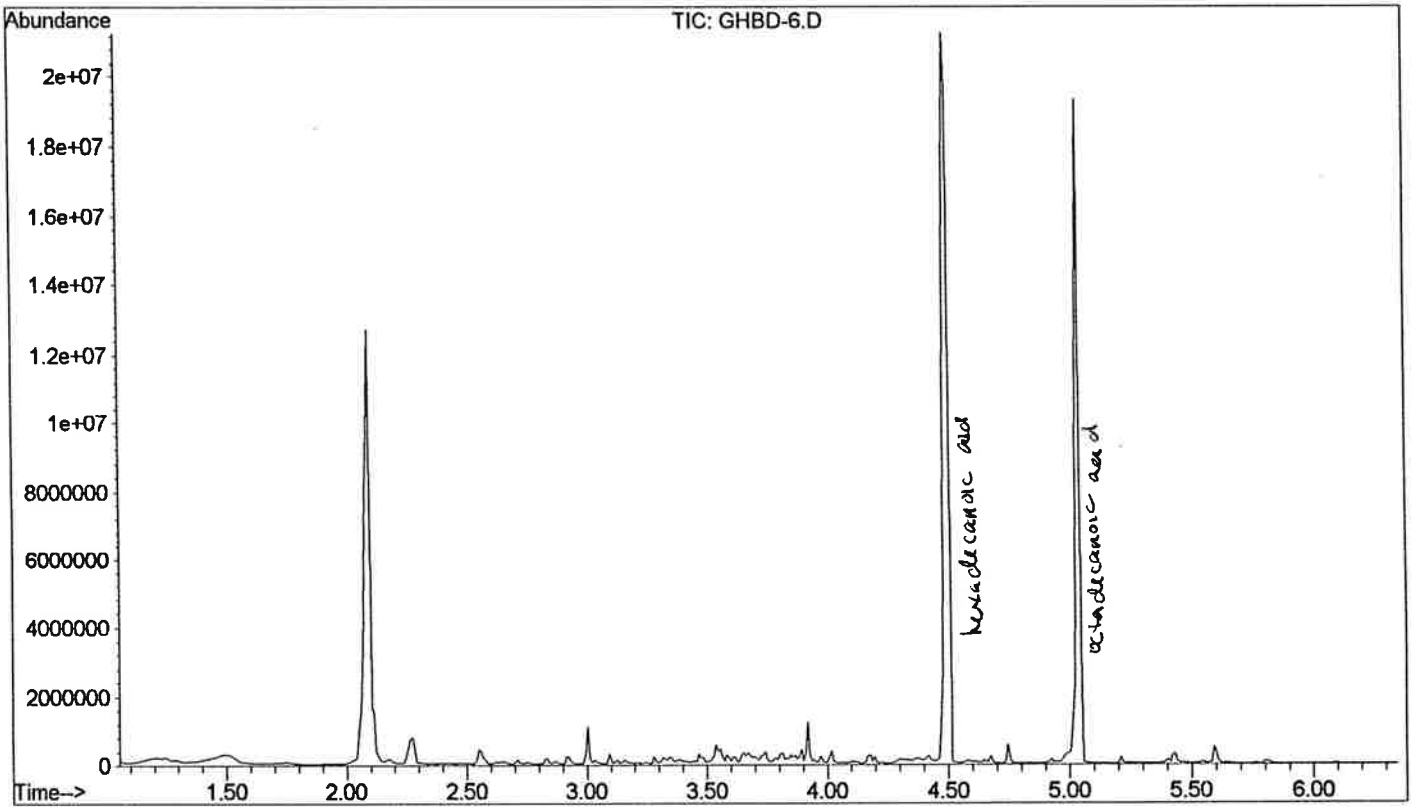


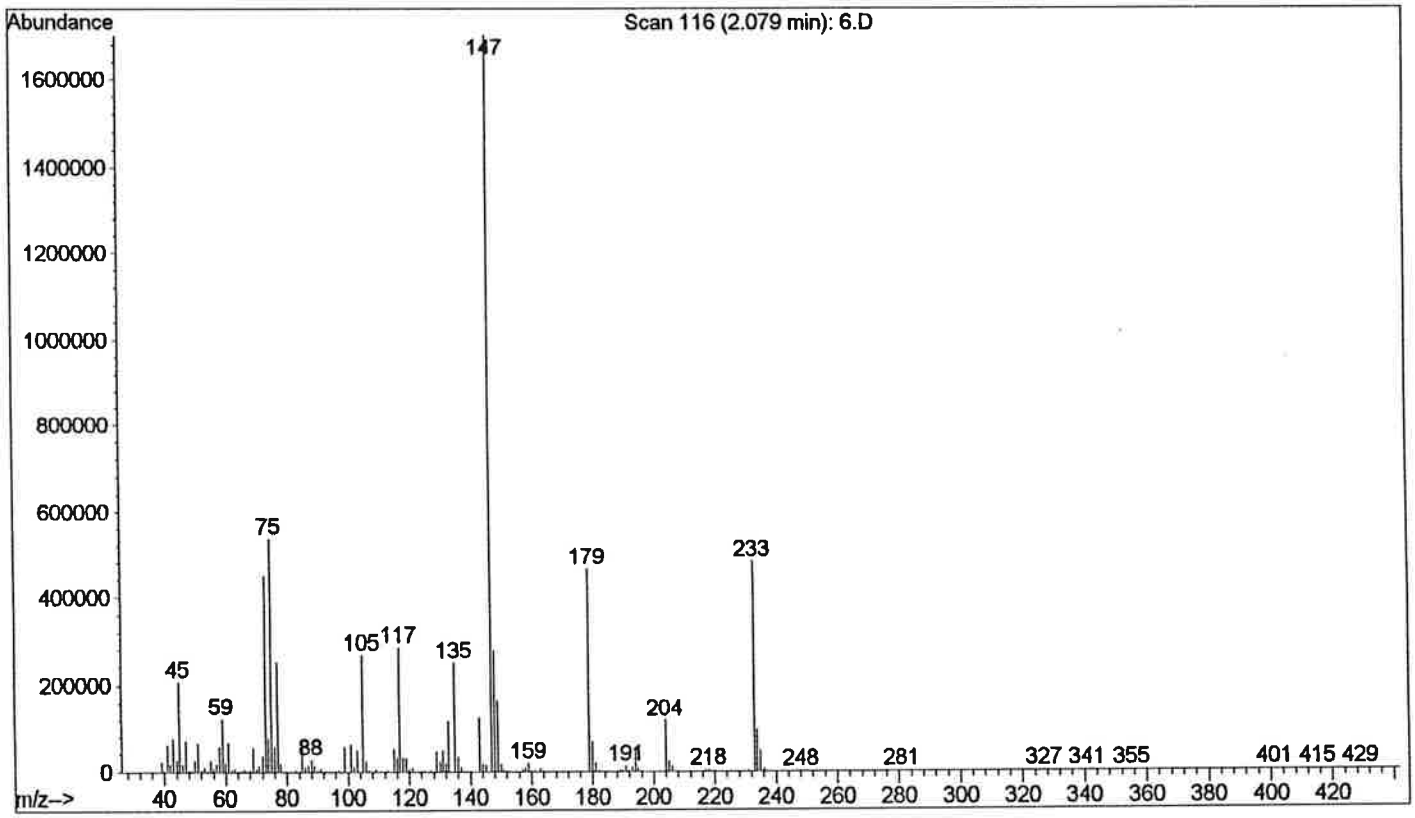
File : C:\HPCHEM\1\DATA\PWF\GHBD-5.D
Operator : JES
Acquired : 21 Jun 1999 4:15 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



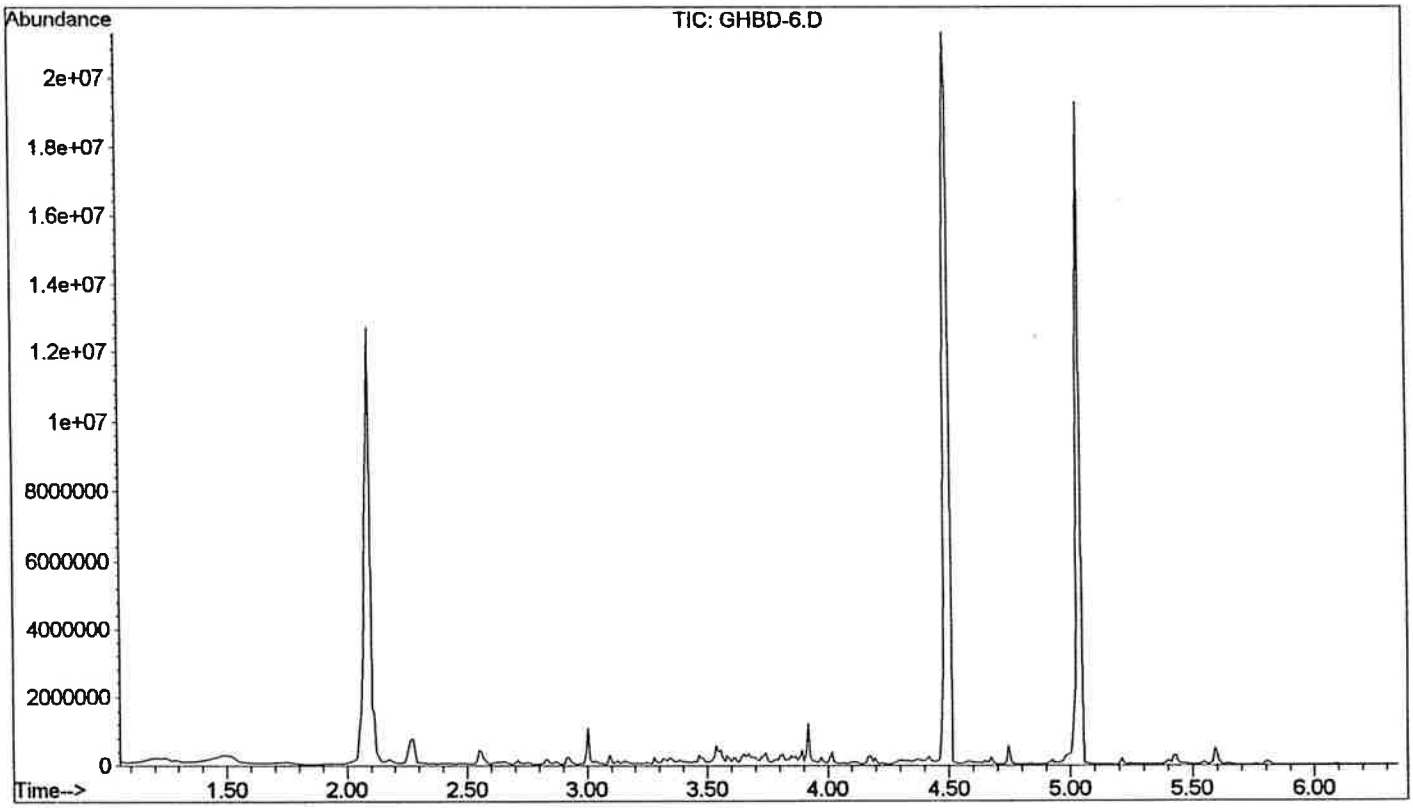


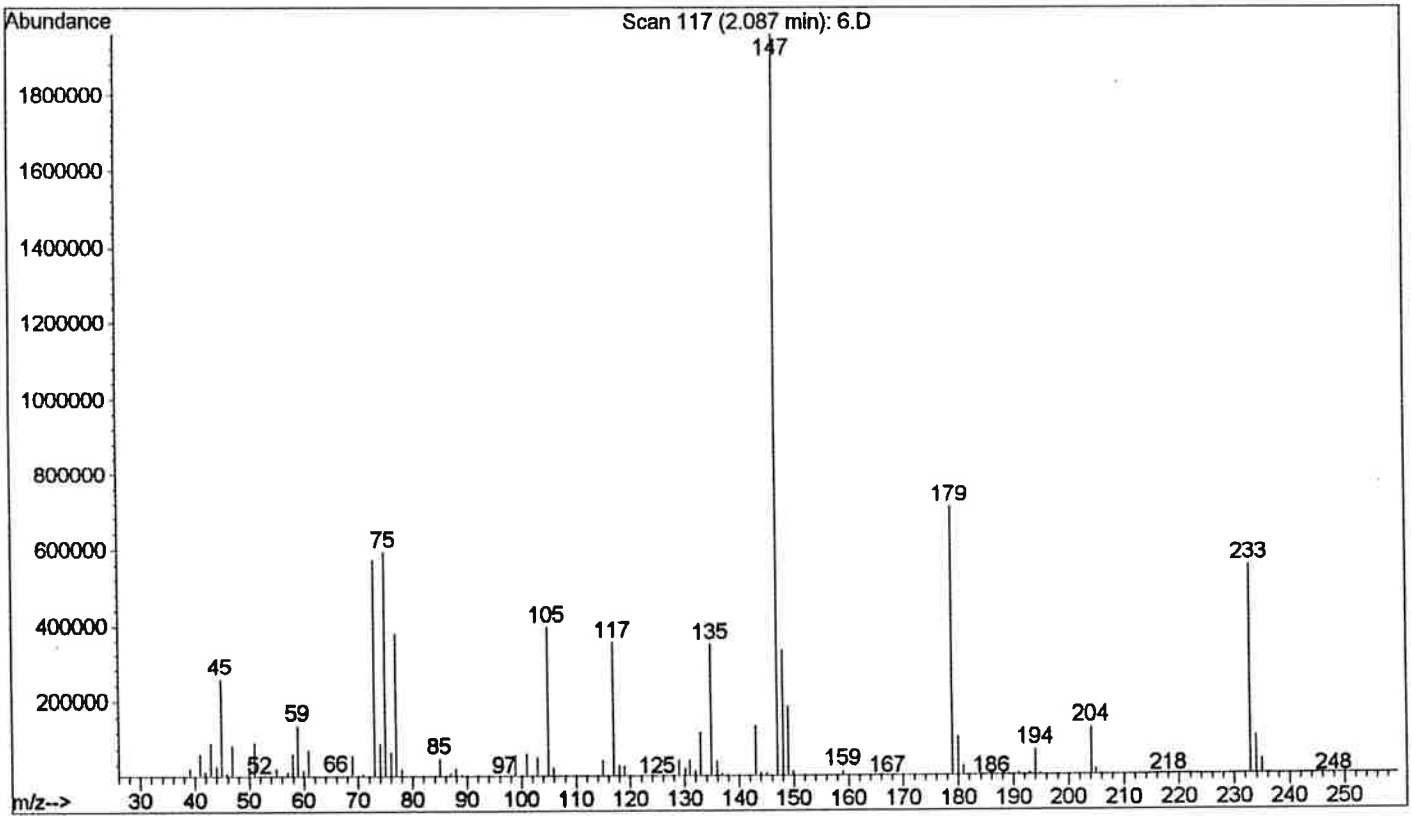
File : C:\HPCHEM\1\DATA\PWF\GHBD-6.D
Operator : JES
Acquired : 22 Jun 1999 1:43 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAC PROCEDURE IV
Vial Number: 1



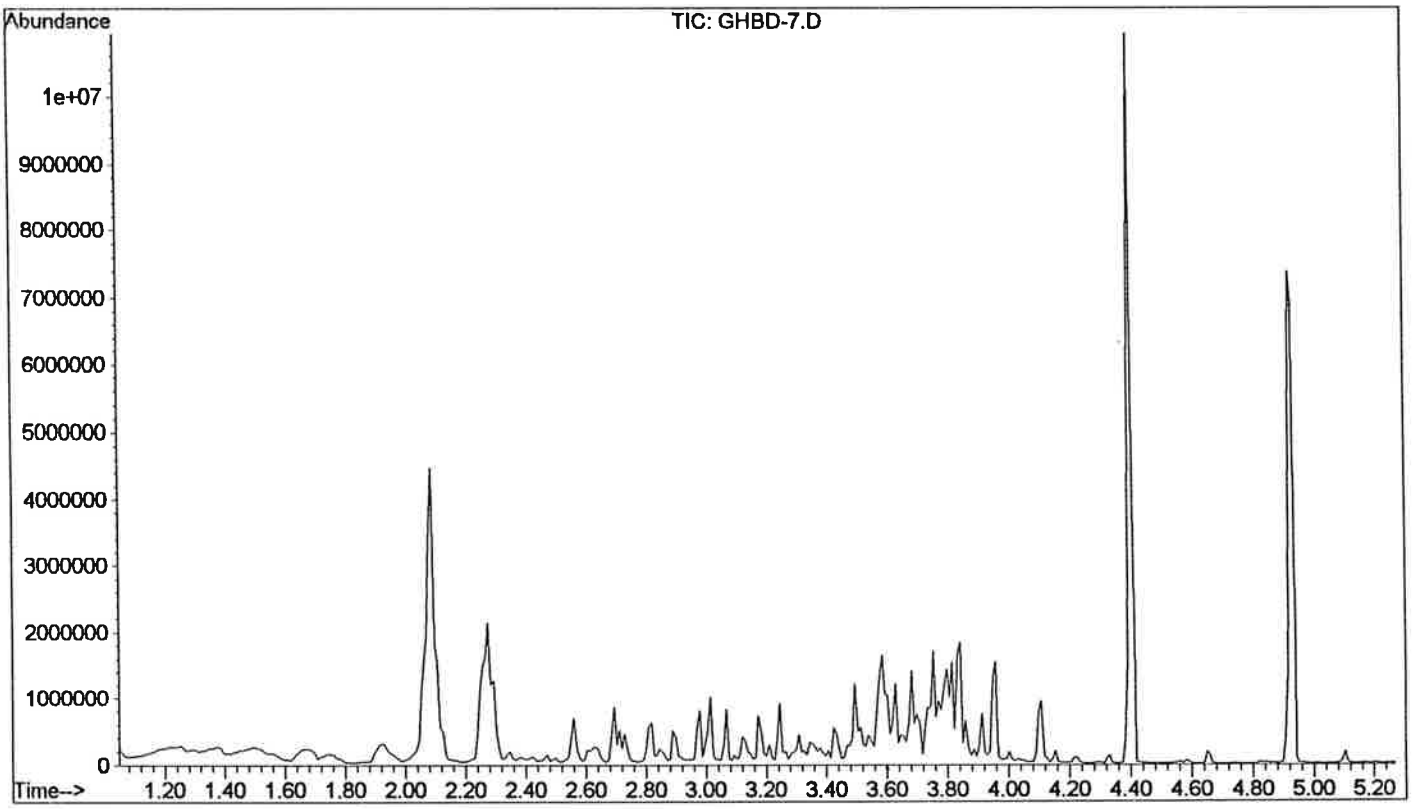


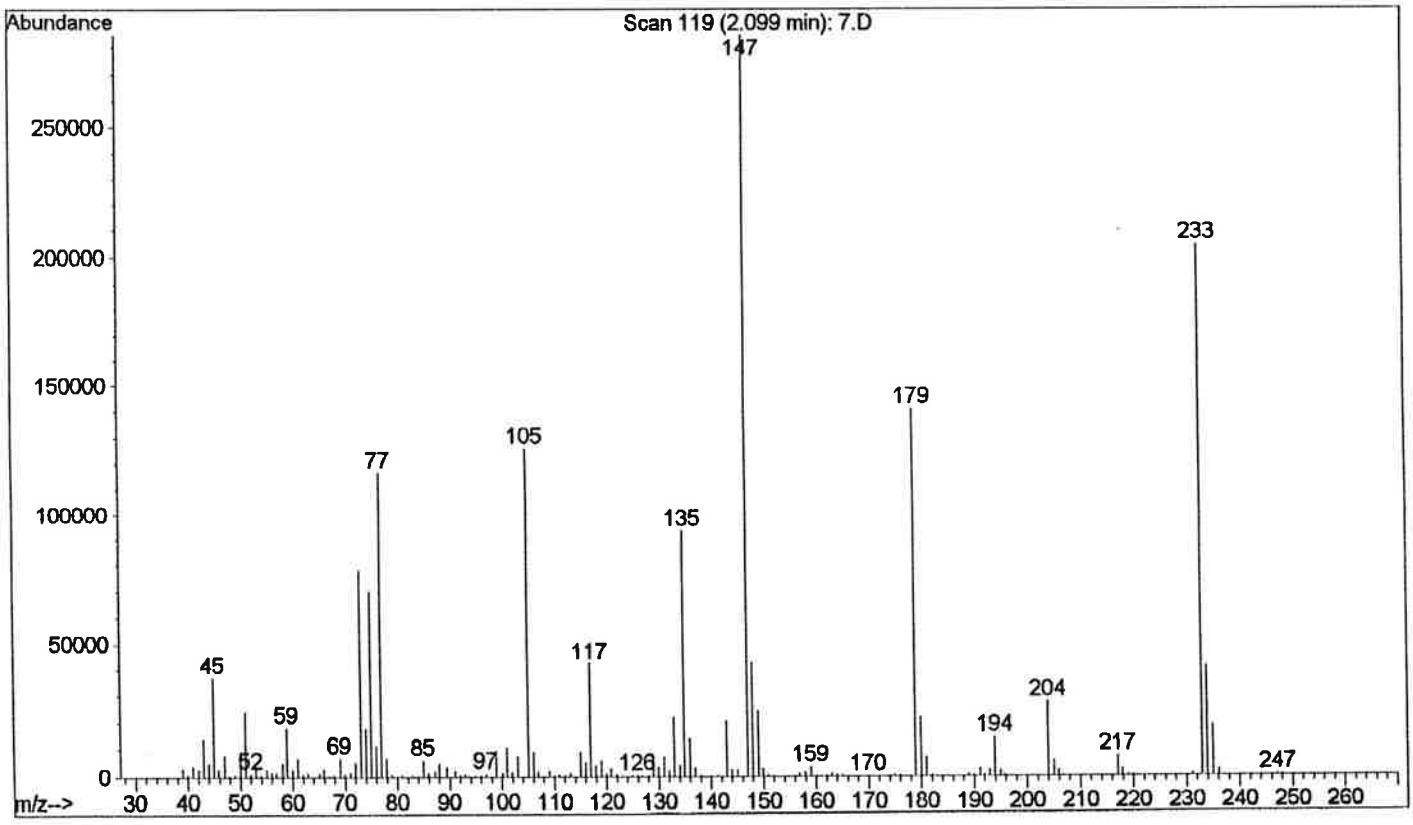
File : C:\HPCHEM\1\DATA\PWF\GHBD-6.D
Operator : JET
Acquired : 22 Jun 1999 1:43 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



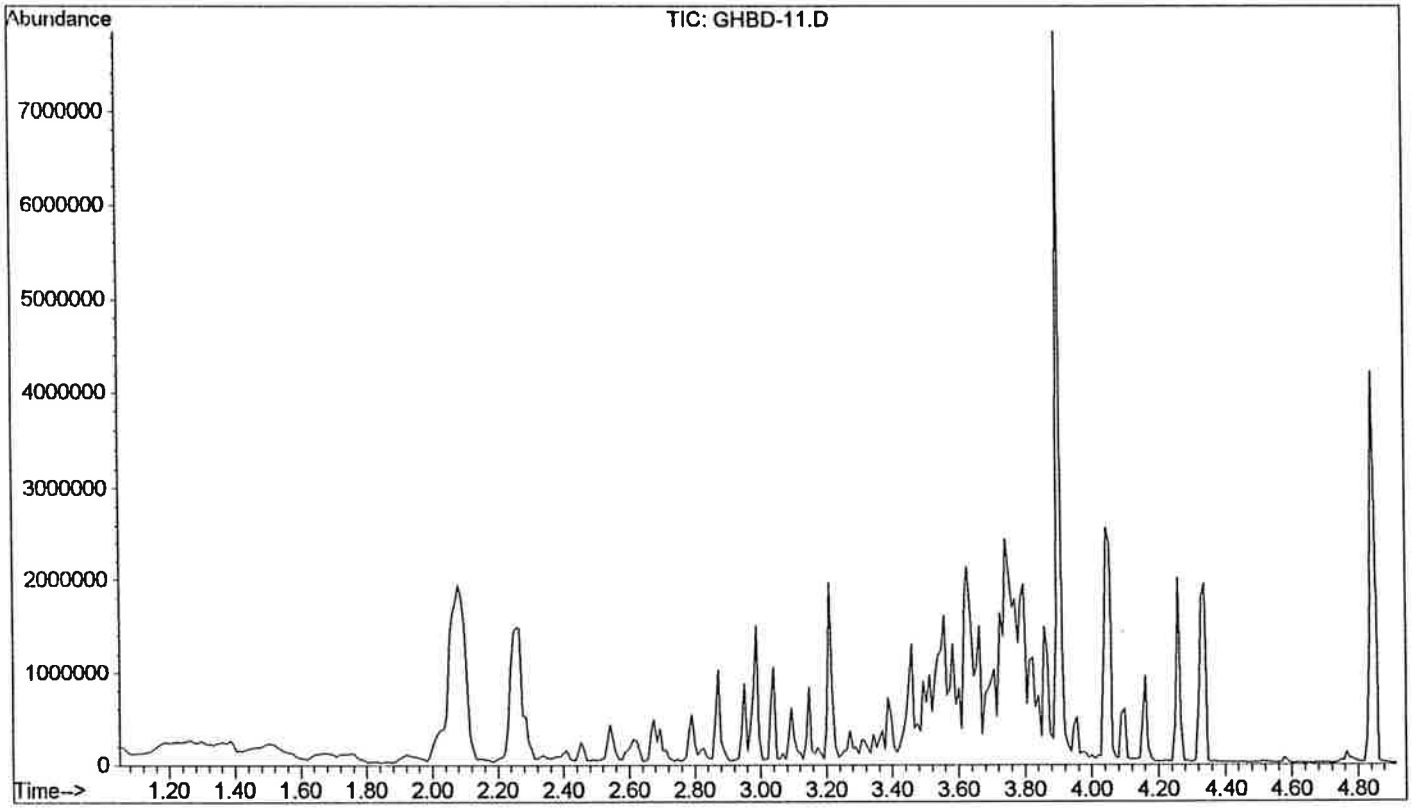


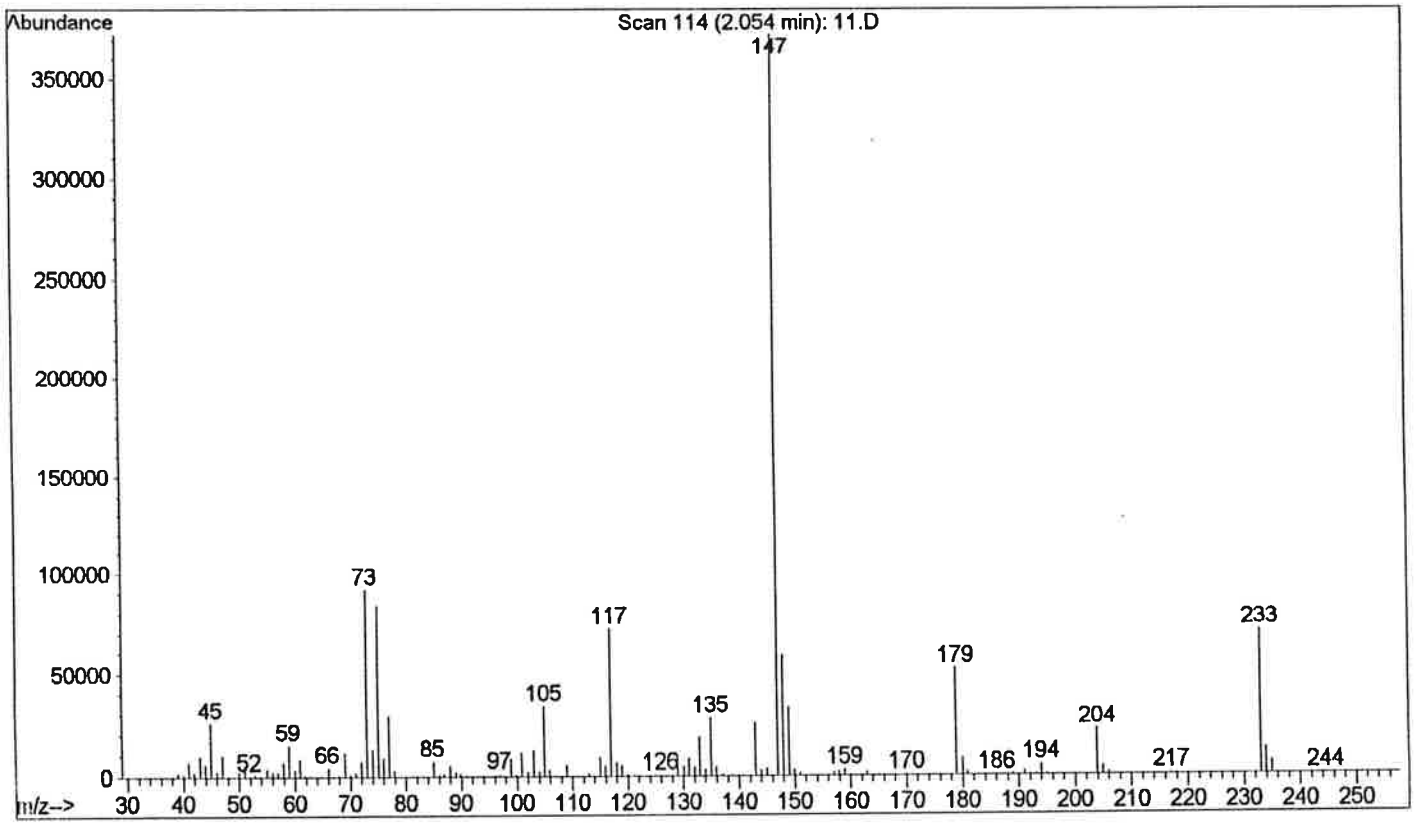
File : C:\HPCHEM\1\DATA\PWF\GHBD-7.D
Operator : JEJ
Acquired : 22 Jun 1999 2:50 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN COKE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



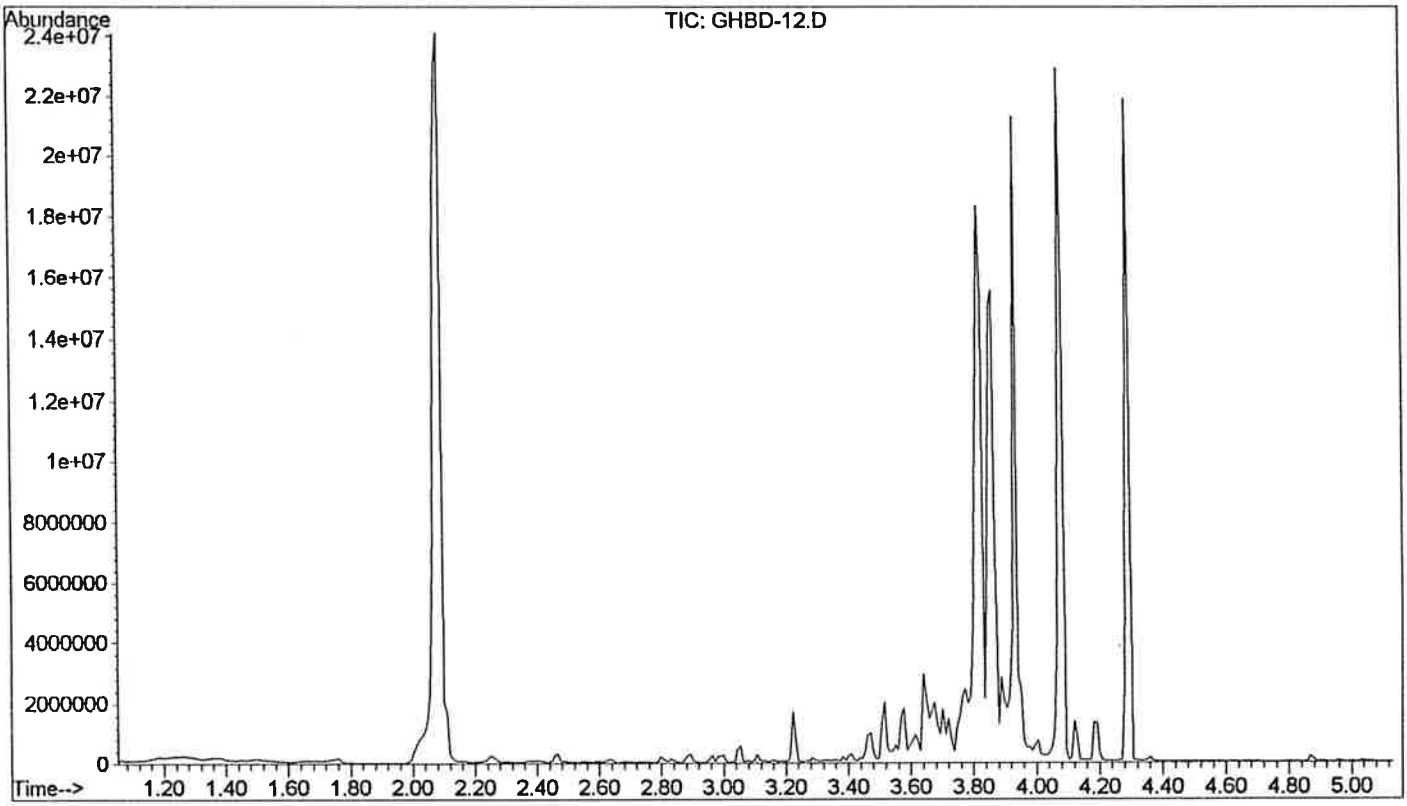


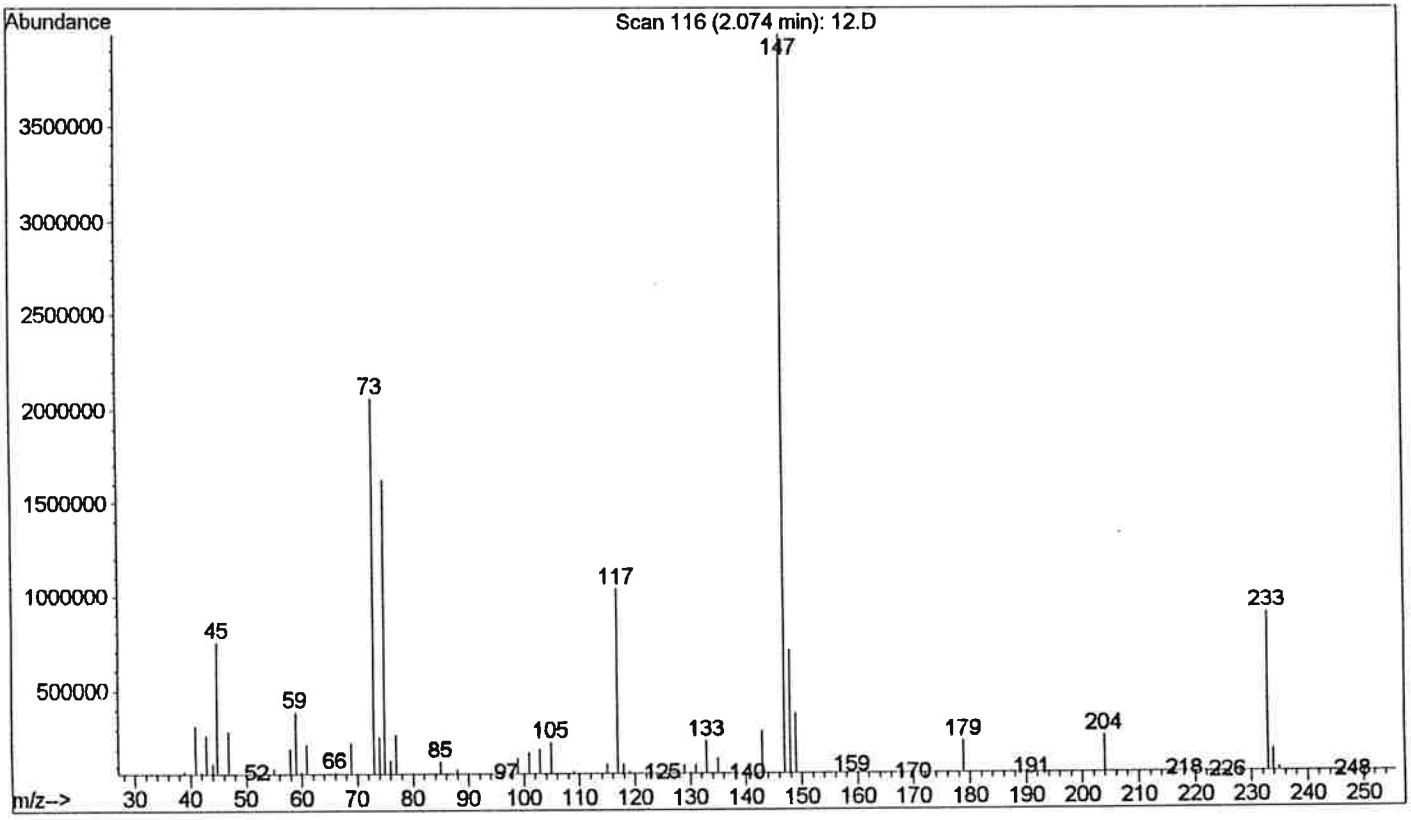
File : C:\HPCHEM\1\DATA\PWF\GHBD-11.D
Operator : JEJ
Acquired : 23 Jun 1999 3:41 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN RUM AND COKE
Misc Info : USING TRIMC PROCEDURE IV
Vial Number: 1



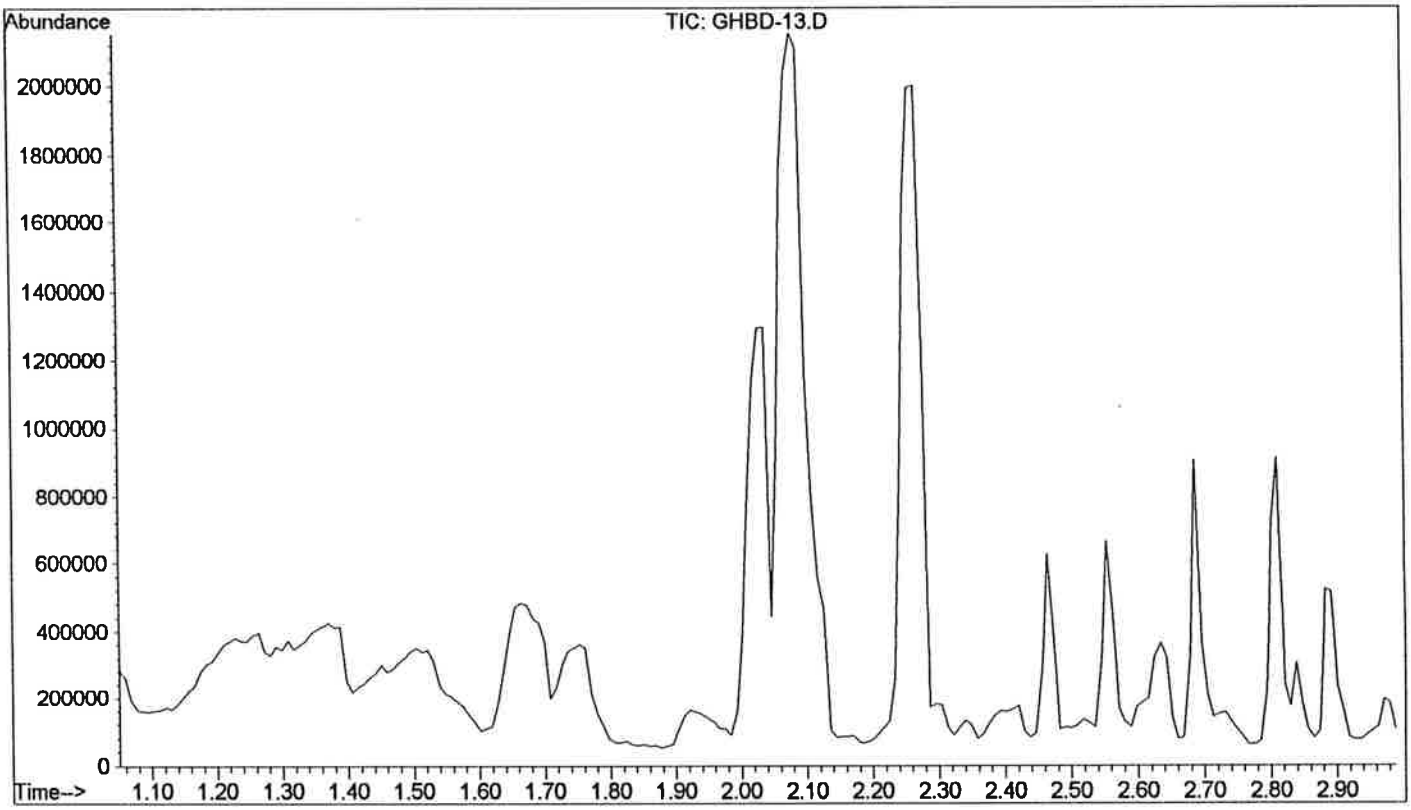


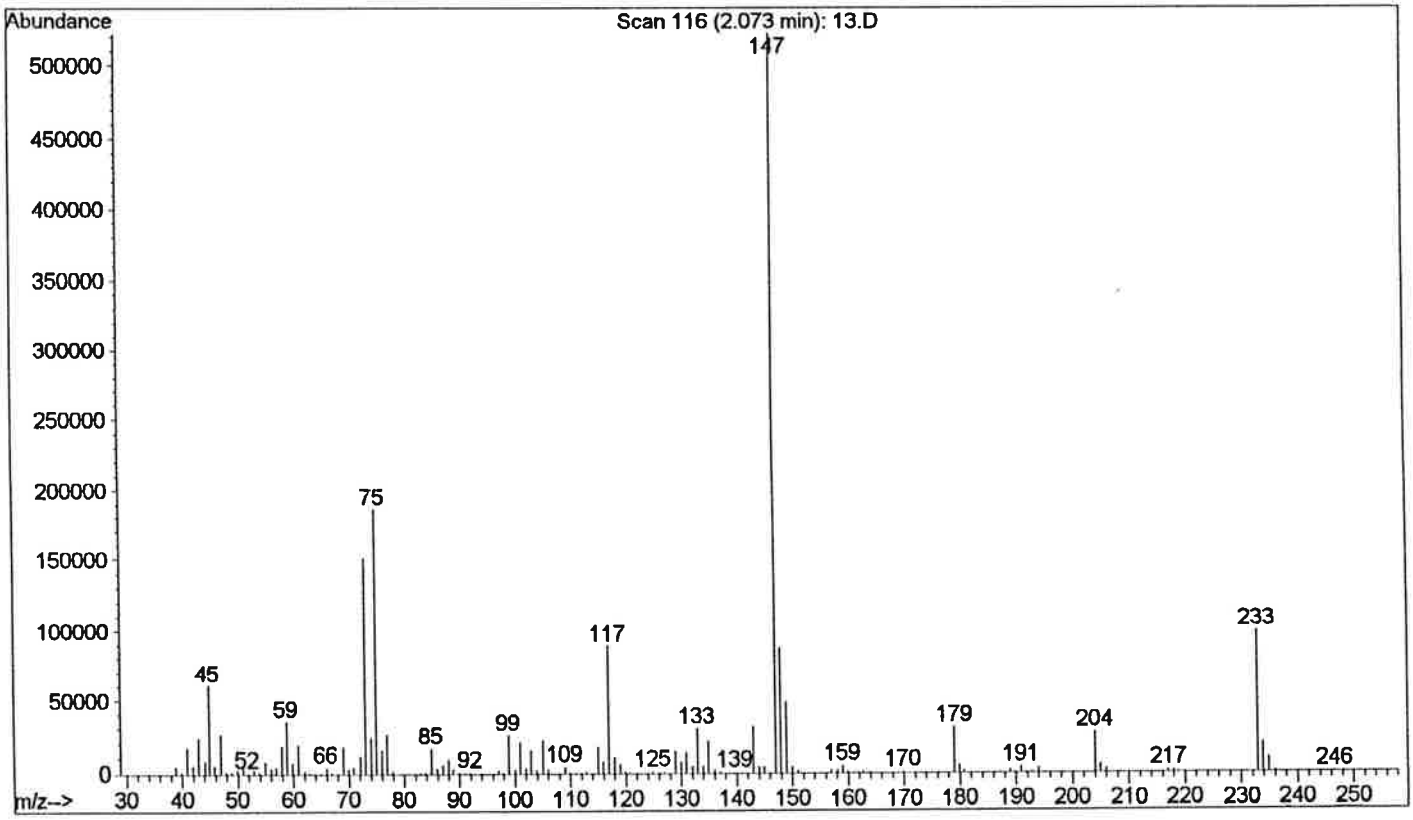
File : C:\HPCHEM\1\DATA\PWF\GHBD-12.D
Operator : JET
Acquired : 28 Jun 1999 10:49 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



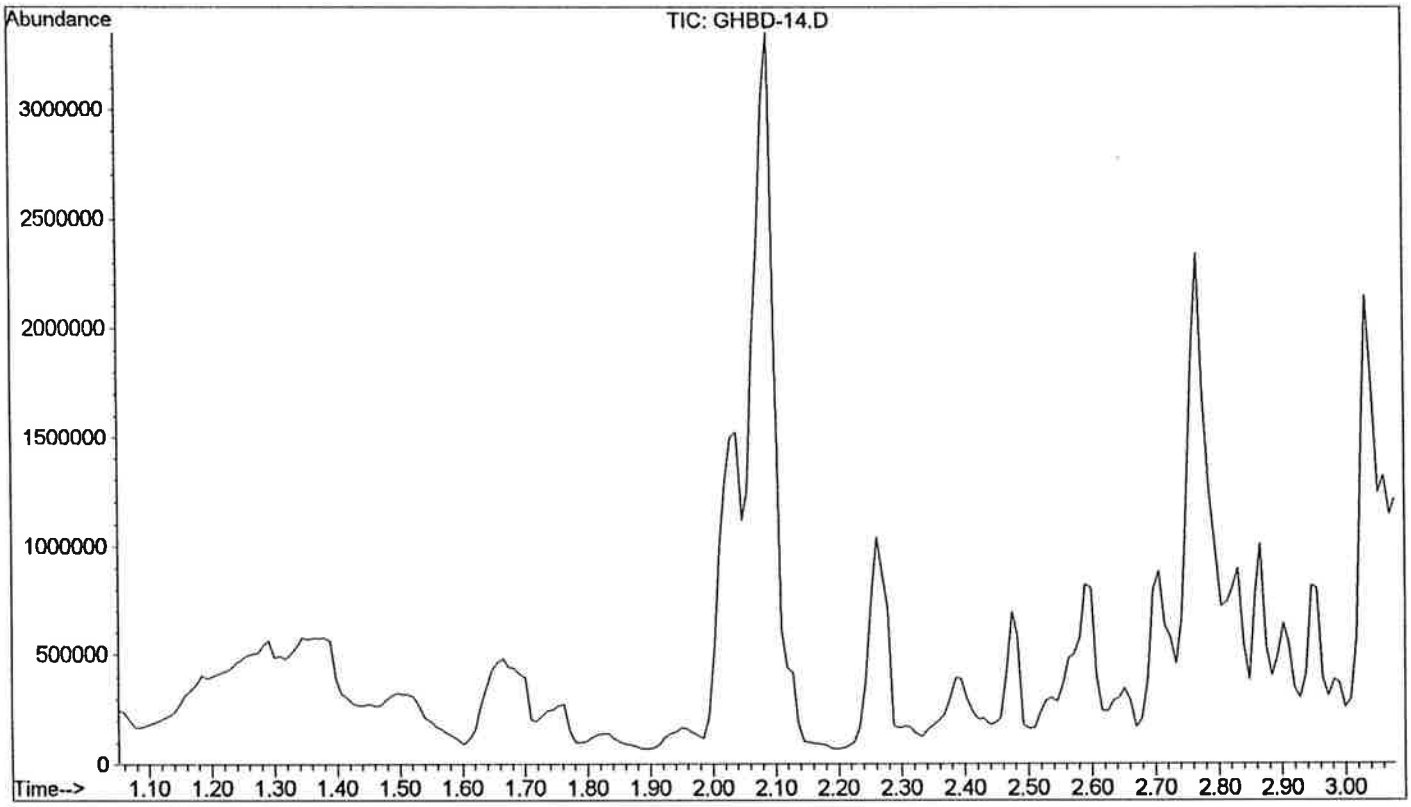


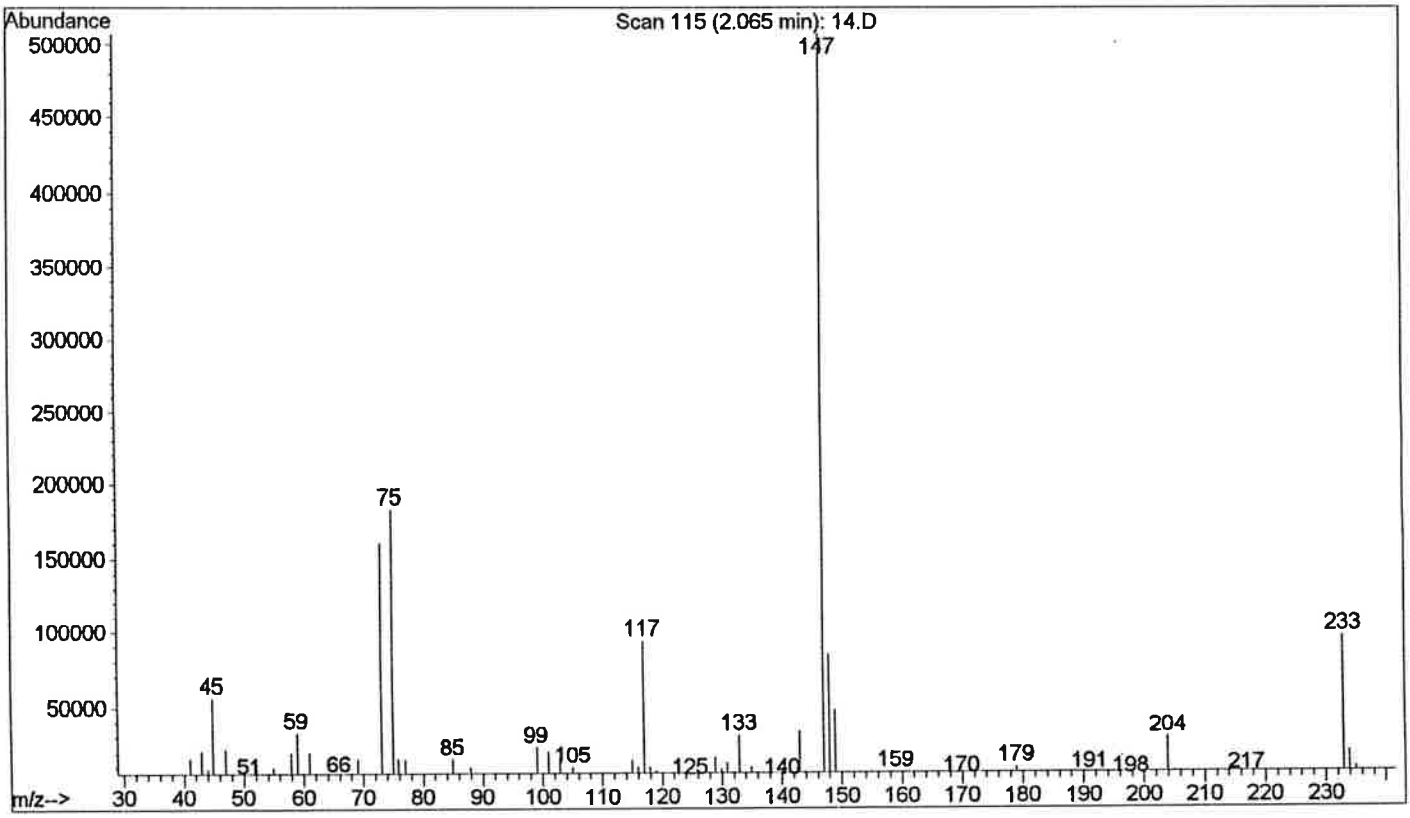
File : C:\HPCHEM\1\DATA\PWF\GHBD-13.D
Operator : JES
Acquired : 28 Jun 1999 10:57 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN RUM AND COKE
Misc Info : USING TRIAK PROCEDURE IV
Vial Number: 1



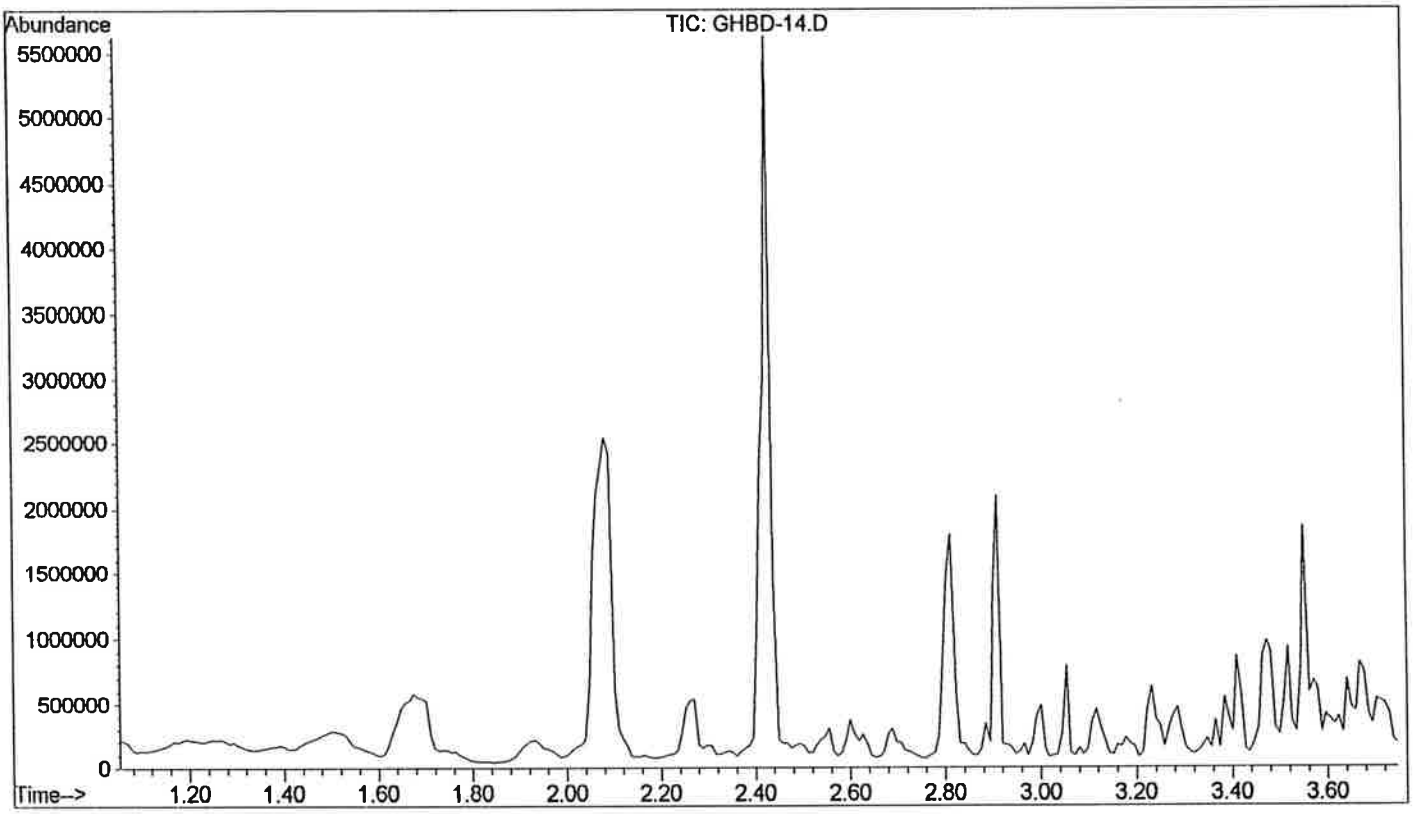


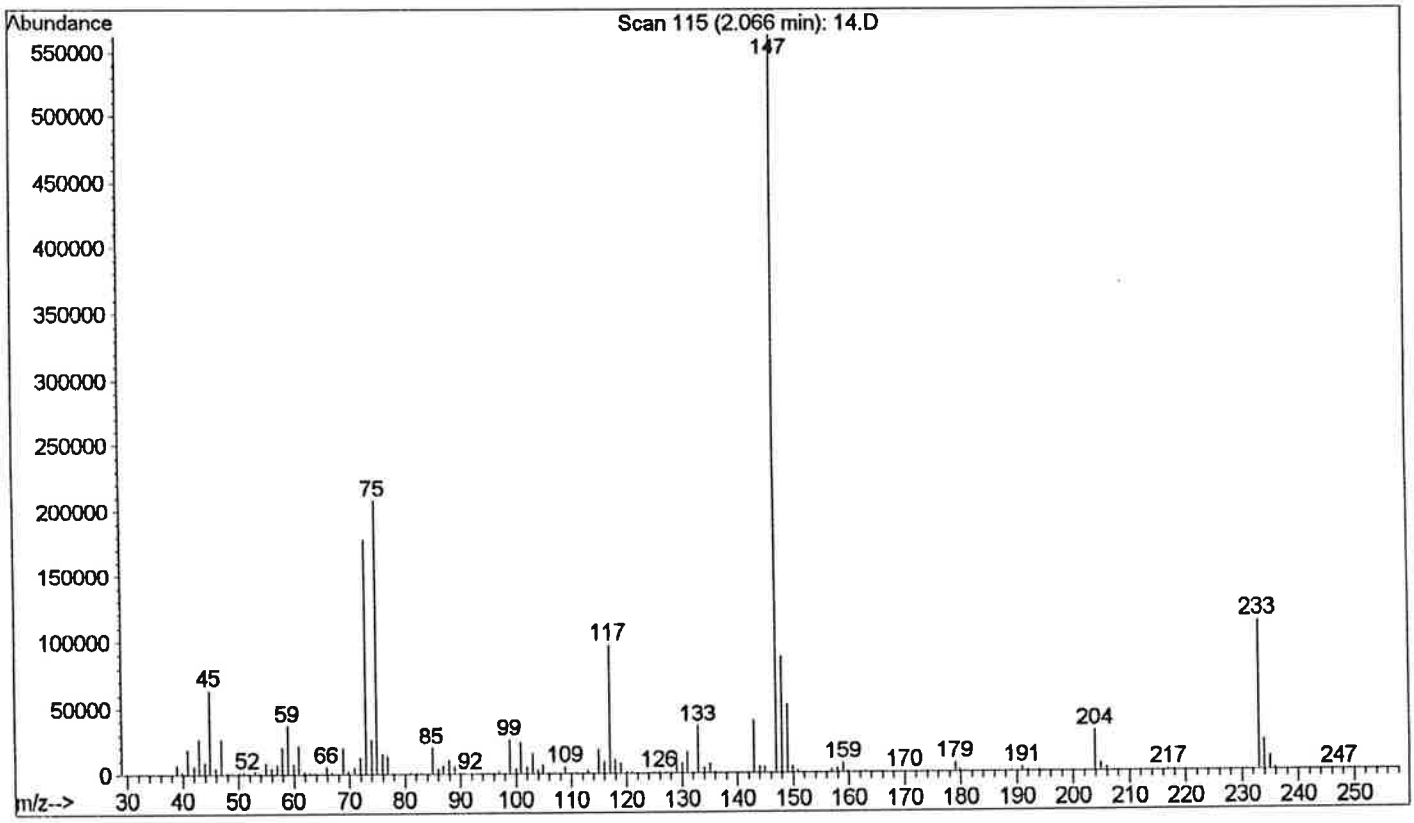
File : C:\HPCHEM\1\DATA\PWF\GHBD-14.D
Operator : JES
Acquired : 28 Jun 1999 11:04 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN COKE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



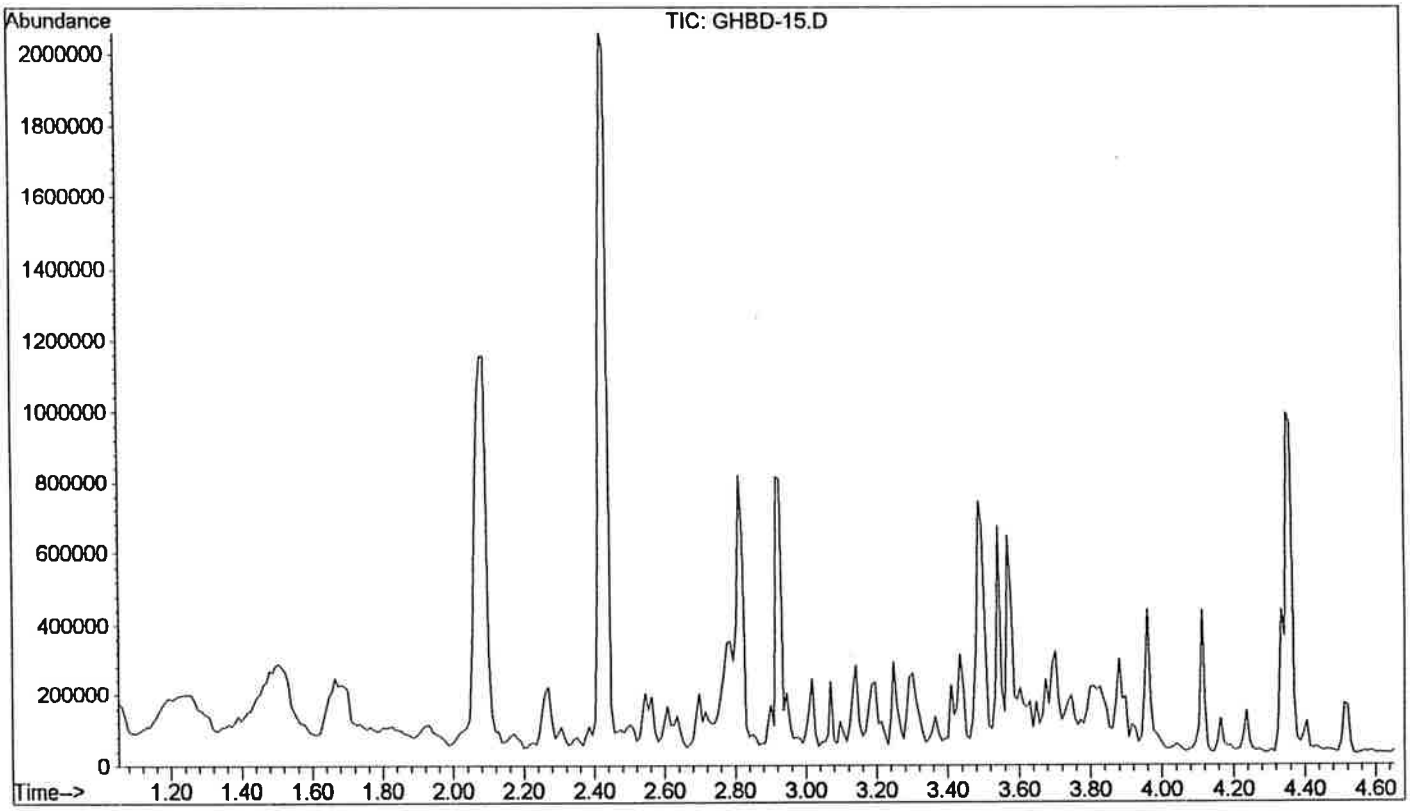


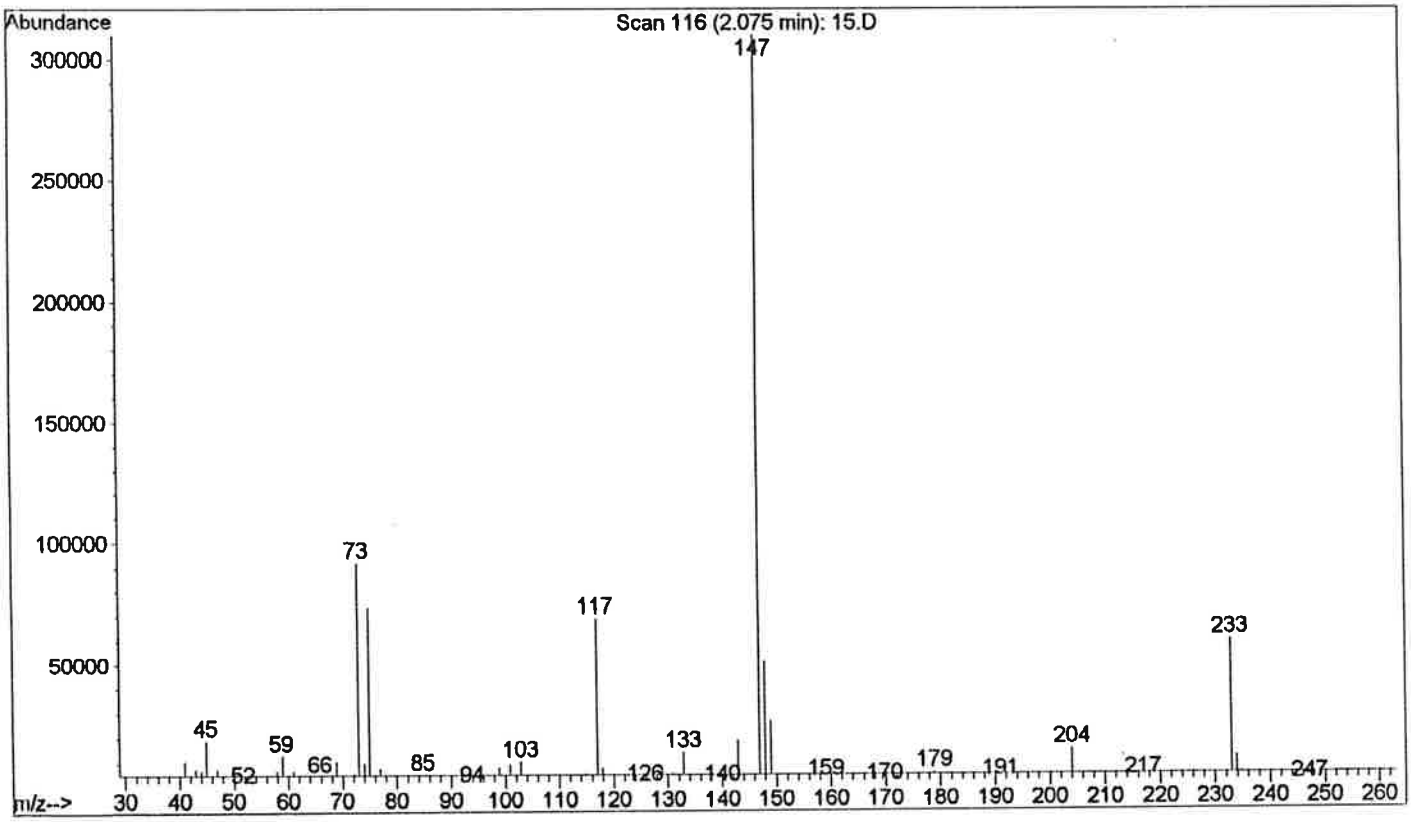
File : C:\HPCHEM\1\DATA\PWF\GHBD-14.D
Operator : JE J
Acquired : 28 Jun 1999 3:09 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN COKE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



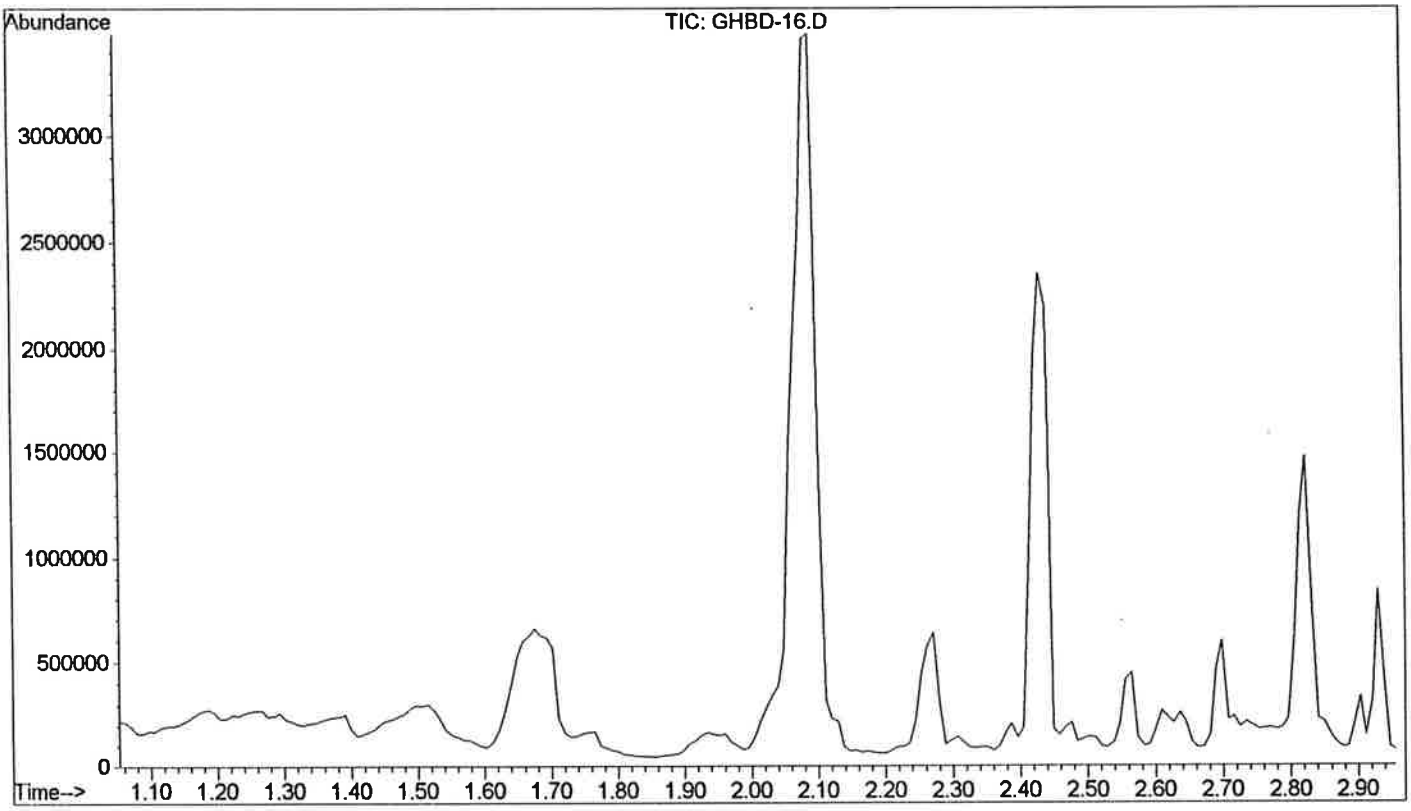


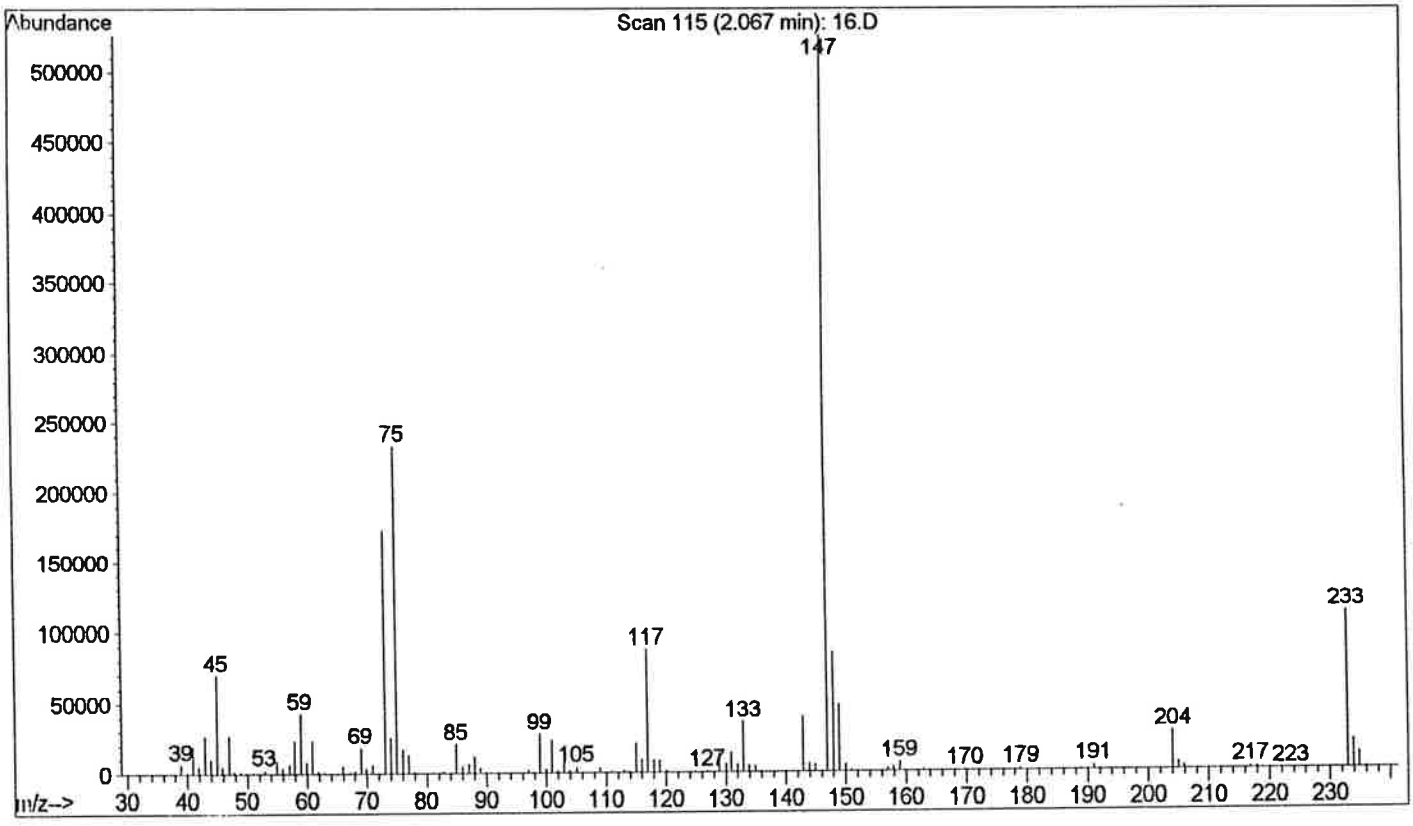
File : C:\HPCHEM\1\DATA\PWF\GHBD-15.D
Operator : JEJ
Acquired : 28 Jun 1999 3:16 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN COKE
Misc Info : USING TRIMC PROCEDURE IV
Vial Number: 1



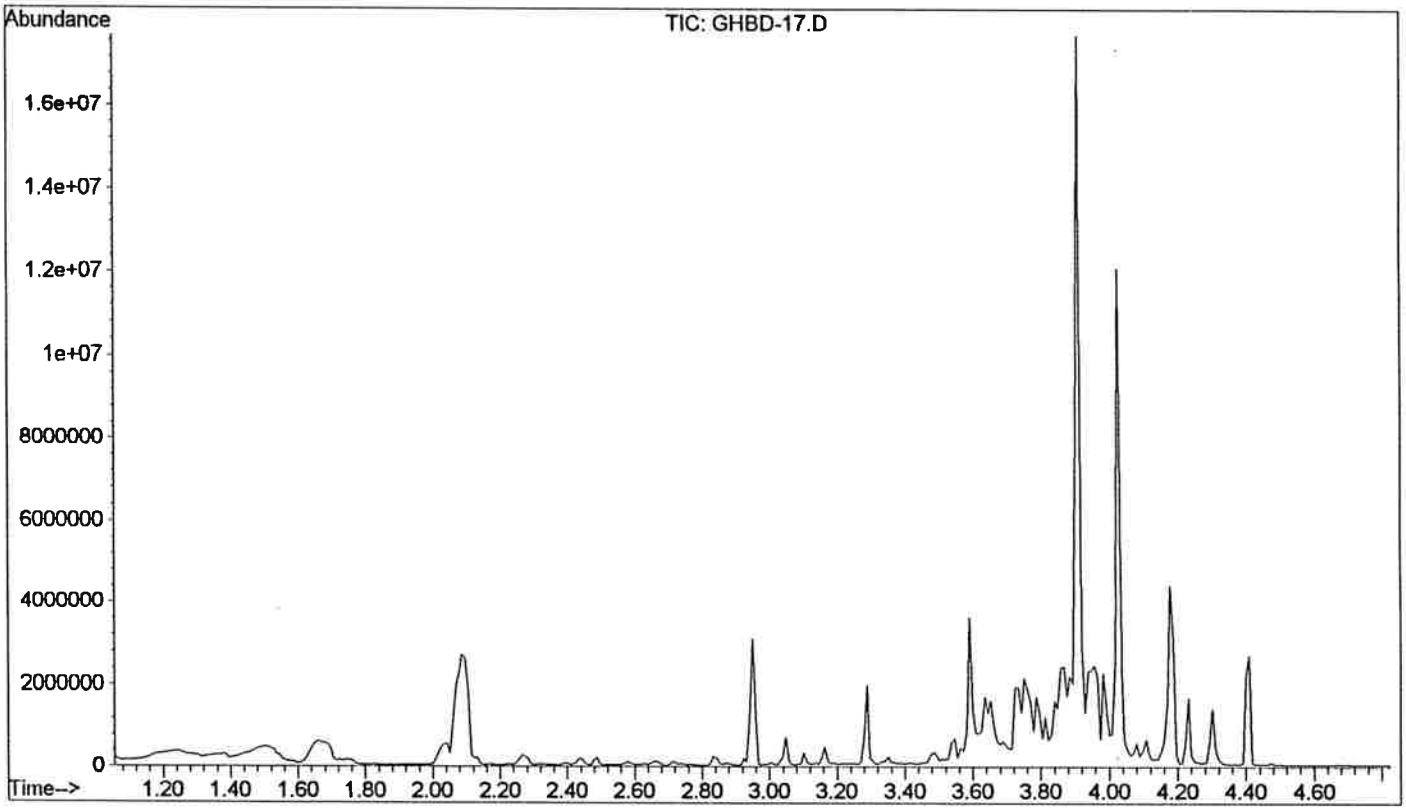


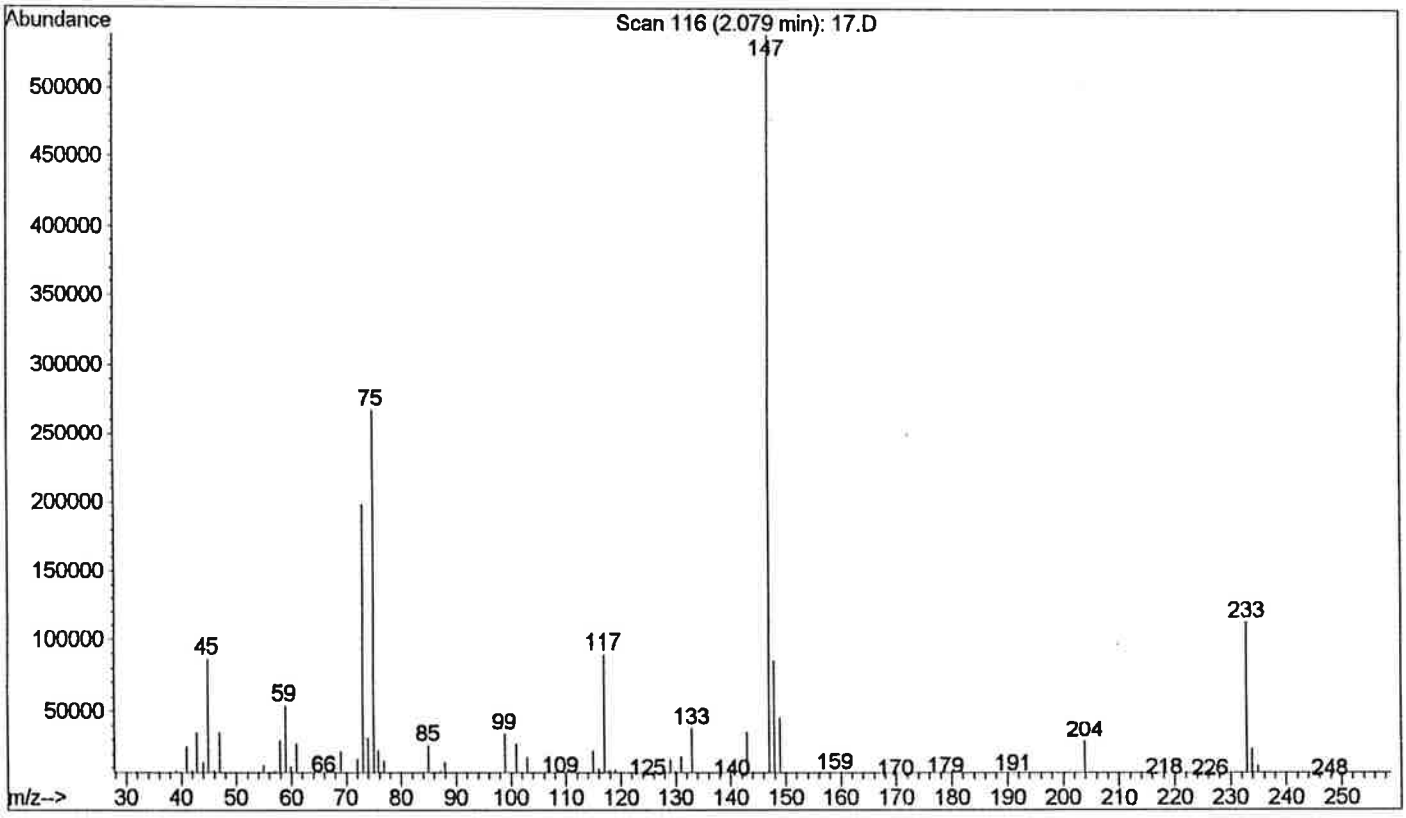
File : C:\HPCHEM\1\DATA\PWF\GHBD-16.D
Operator : JEJ
Acquired : 28 Jun 1999 3:23 pm using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN COKE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



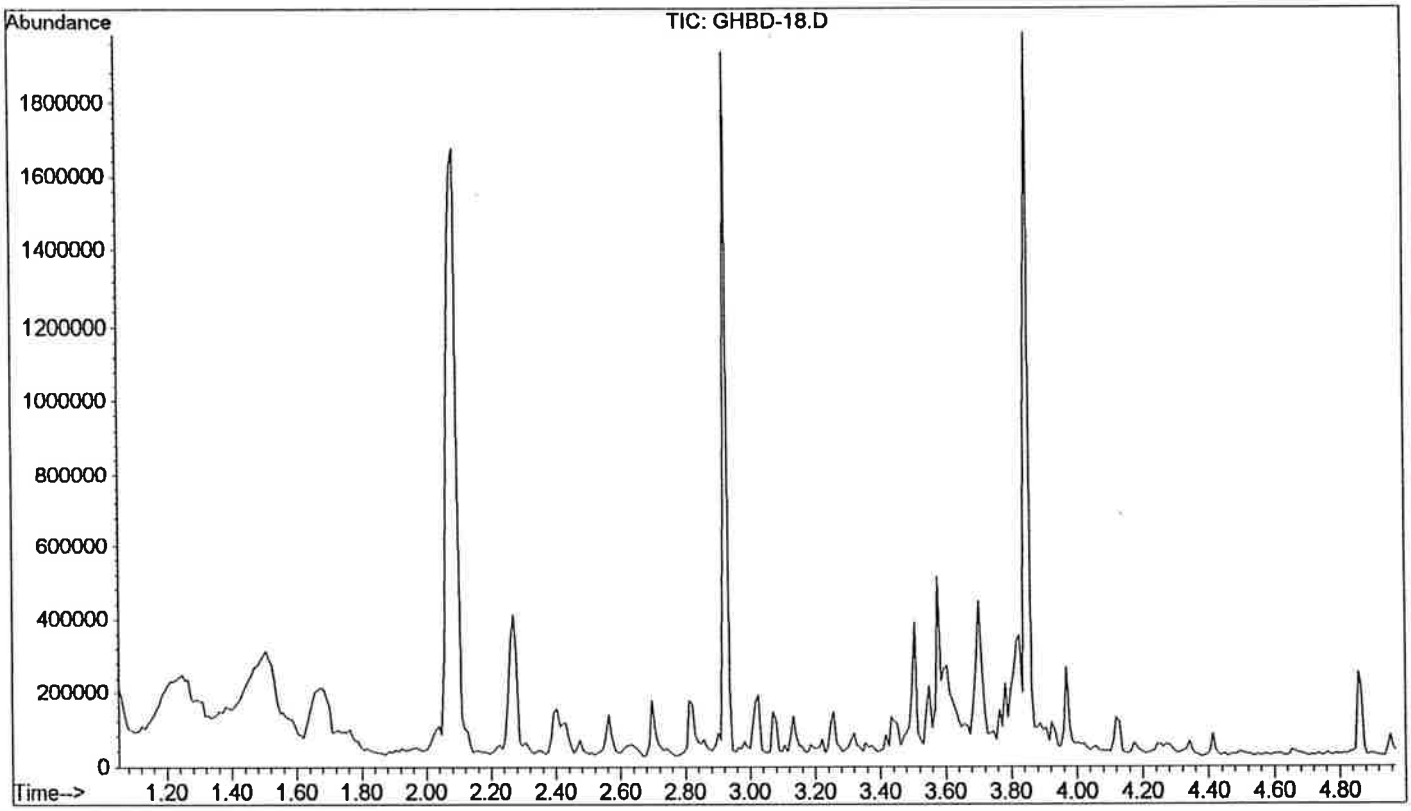


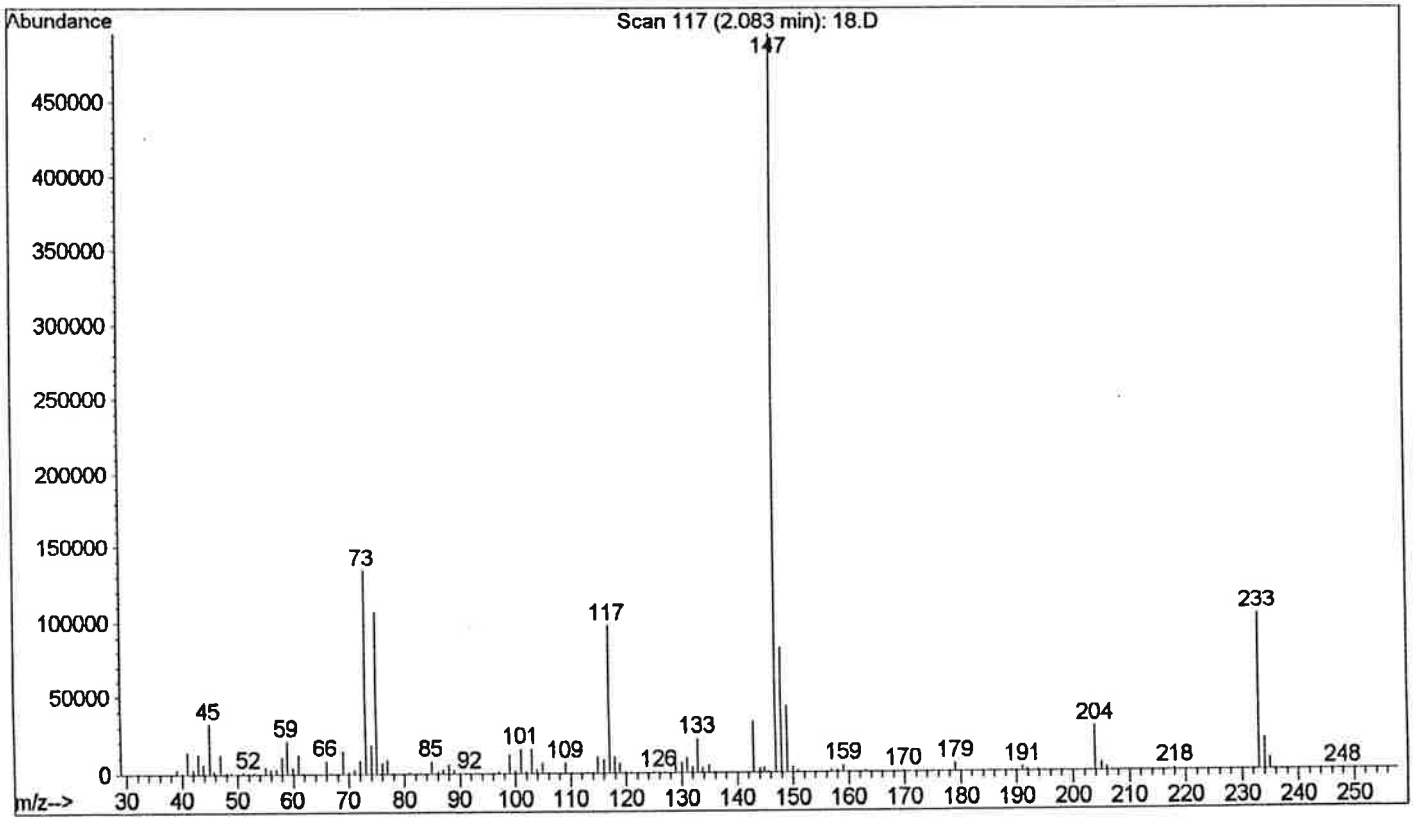
File : C:\HPCHEM\1\DATA\PWF\GHBD-17.D
Operator : JEJ
Acquired : 29 Jun 1999 9:58 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAC PROCEDURE IV
Vial Number: 1



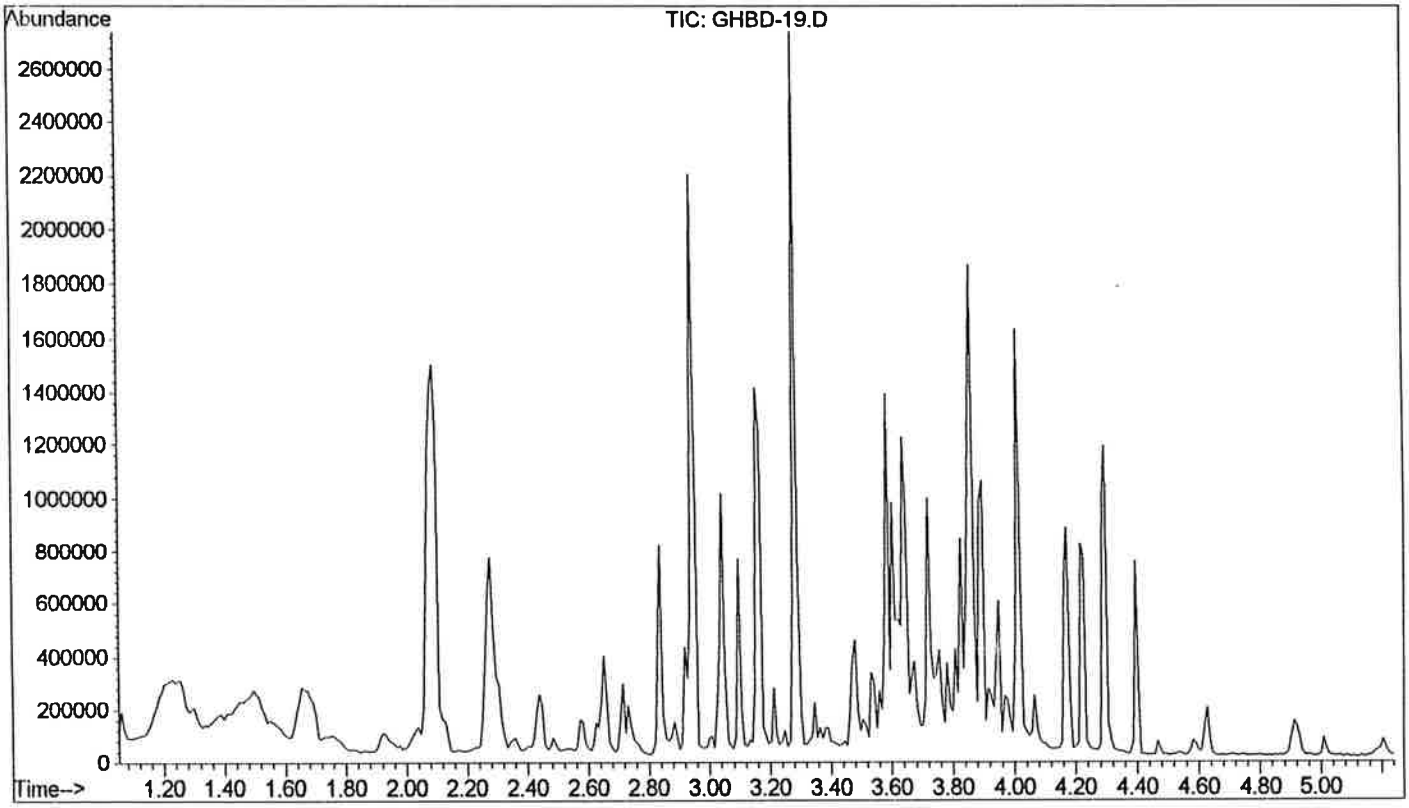


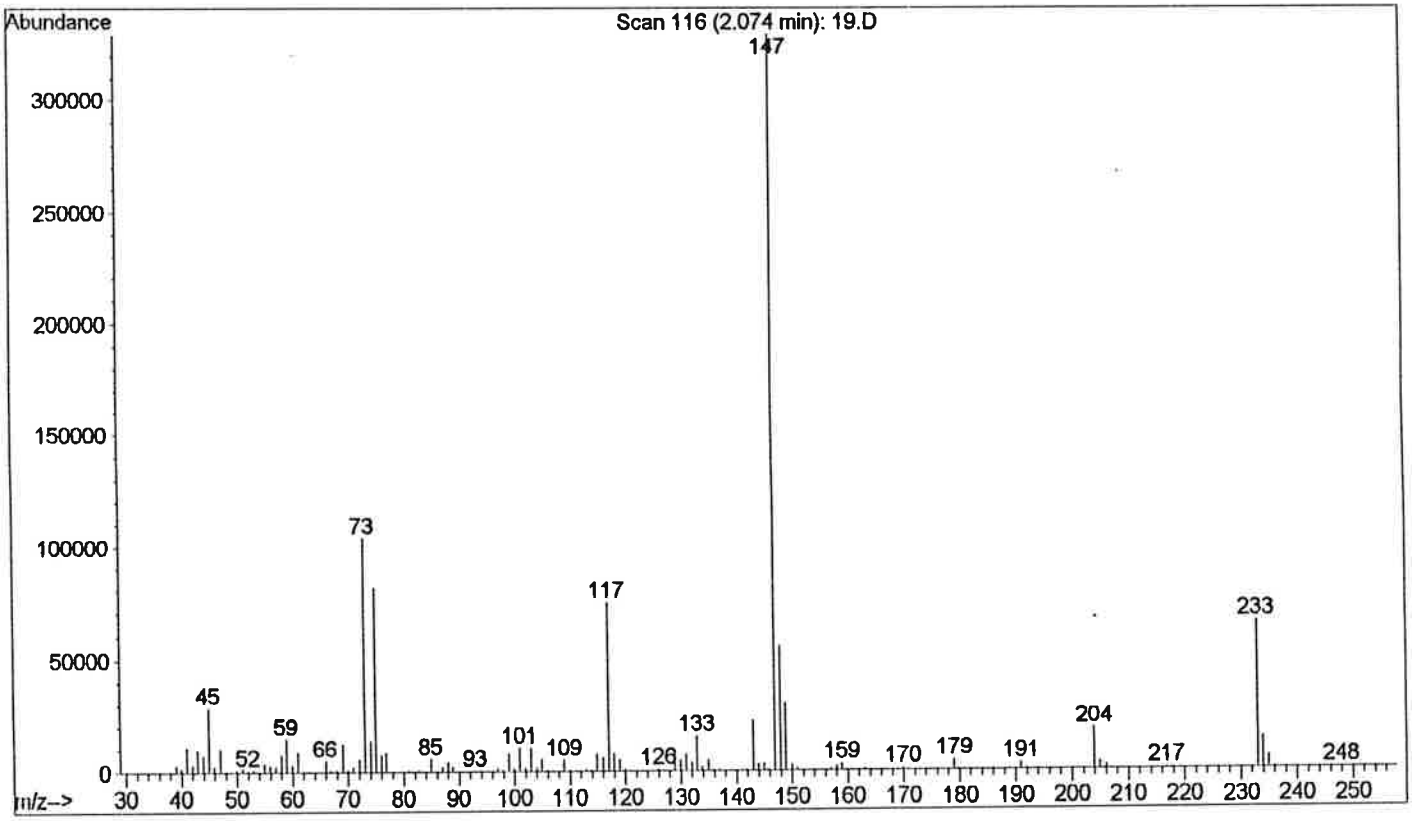
File : C:\HPCHEM\1\DATA\PWF\GHBD-18.D
Operator : SEJ
Acquired : 29 Jun 1999 10:05 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



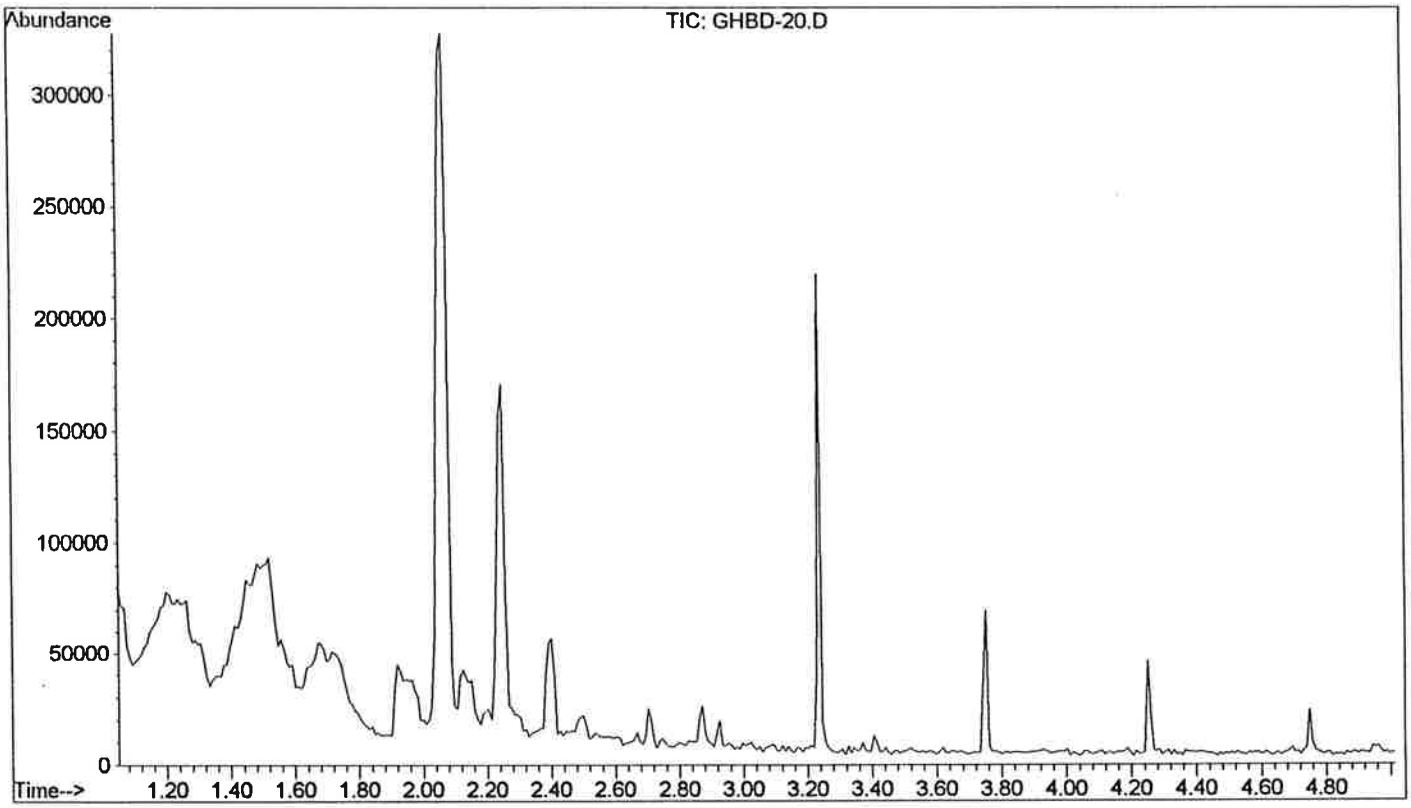


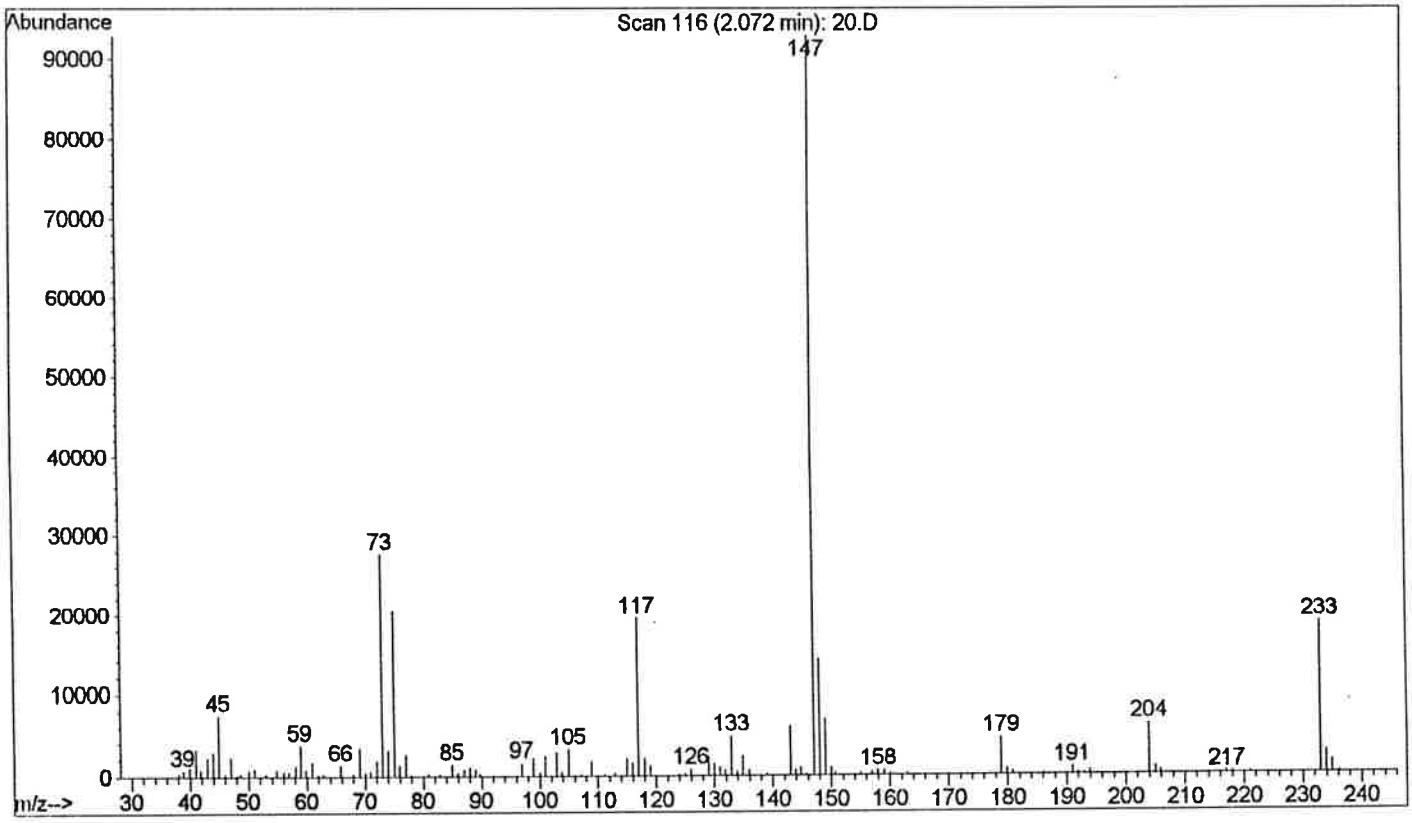
File : C:\HPCHEM\1\DATA\PWF\GHBD-19.D
Operator : JEJ
Acquired : 29 Jun 1999 14:33 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN COKE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



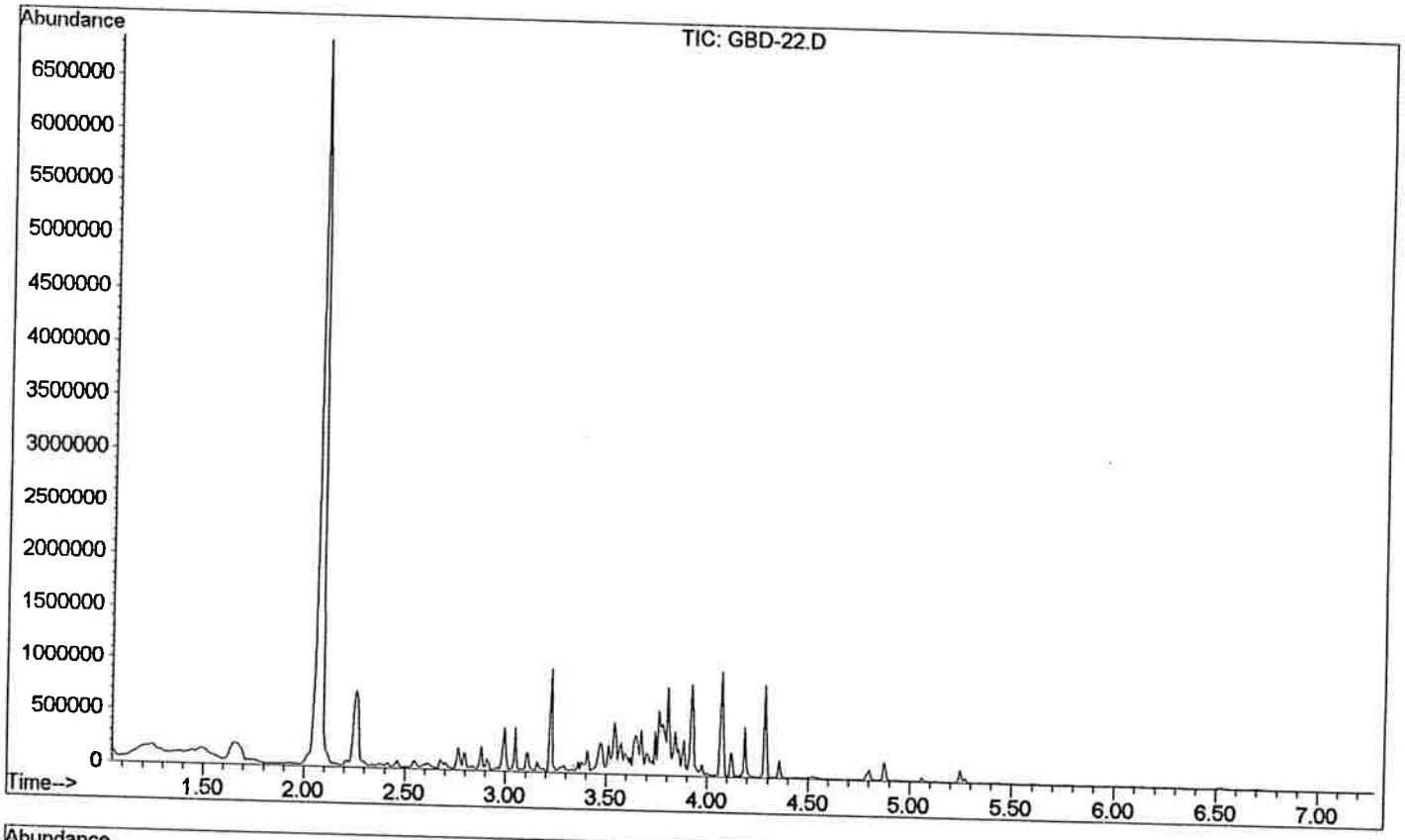


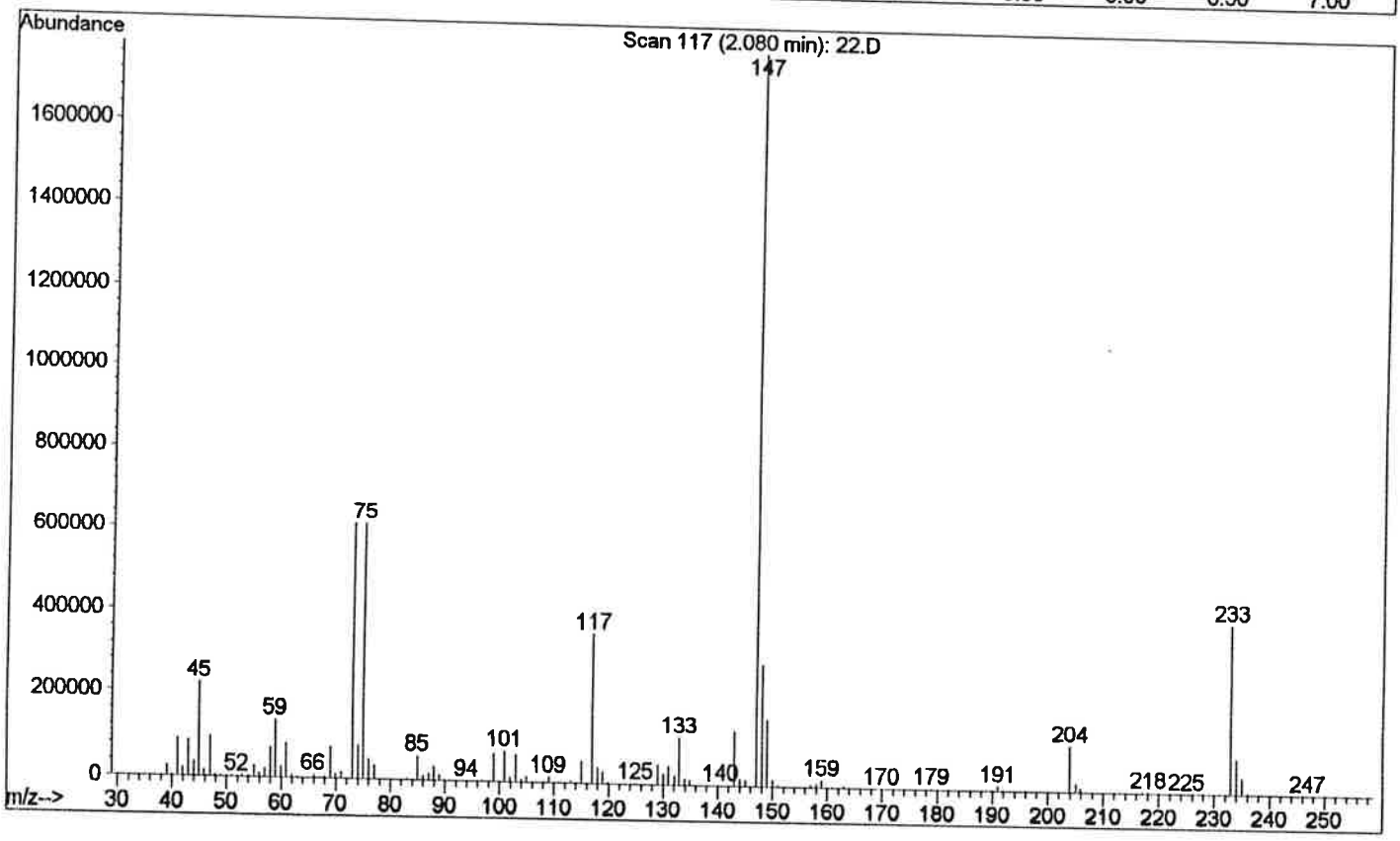
File : C:\HPCHEM\1\DATA\PWF\GHBD-20.D
Operator : JEJ
Acquired : 2 Jul 1999 9:07 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIAL PROCEDURE IV
Vial Number: 1



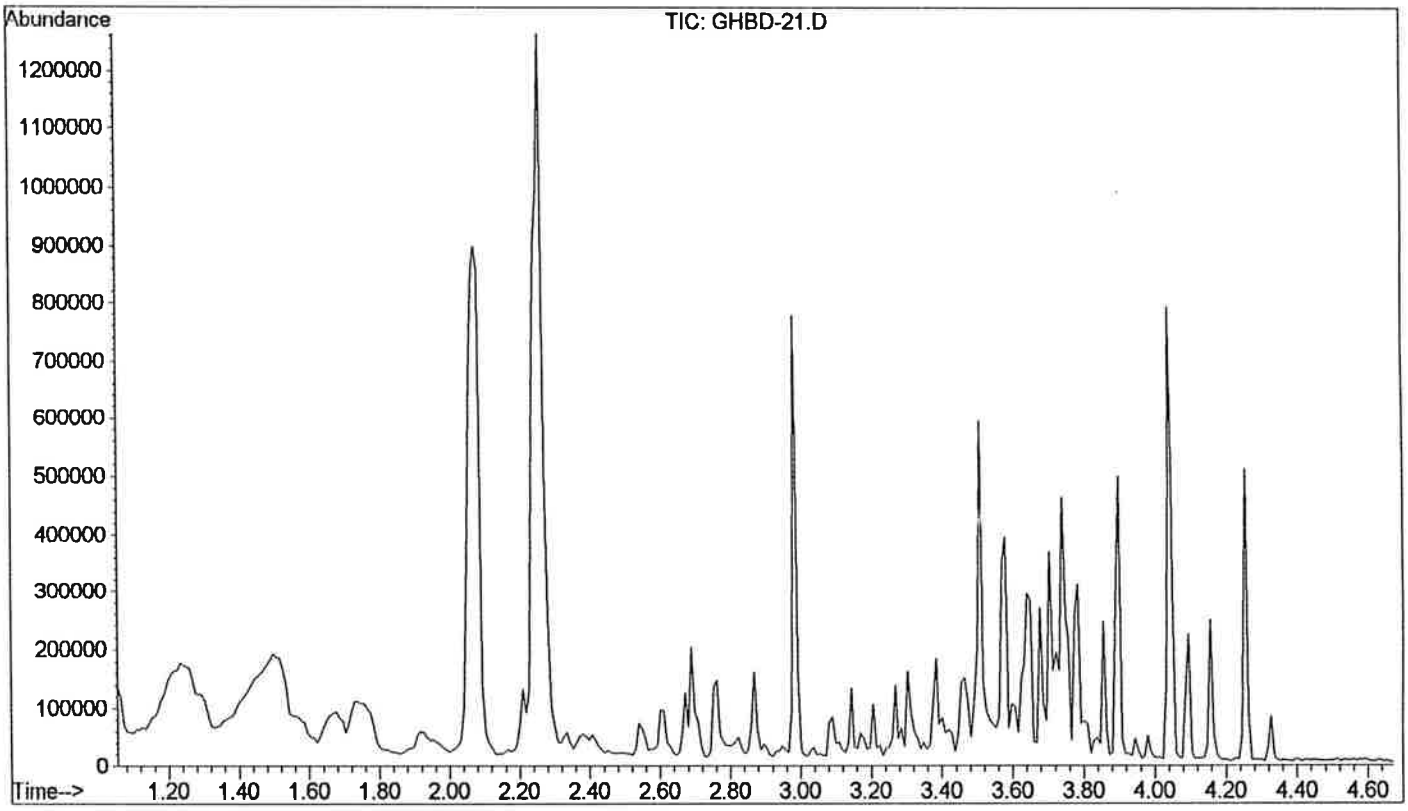


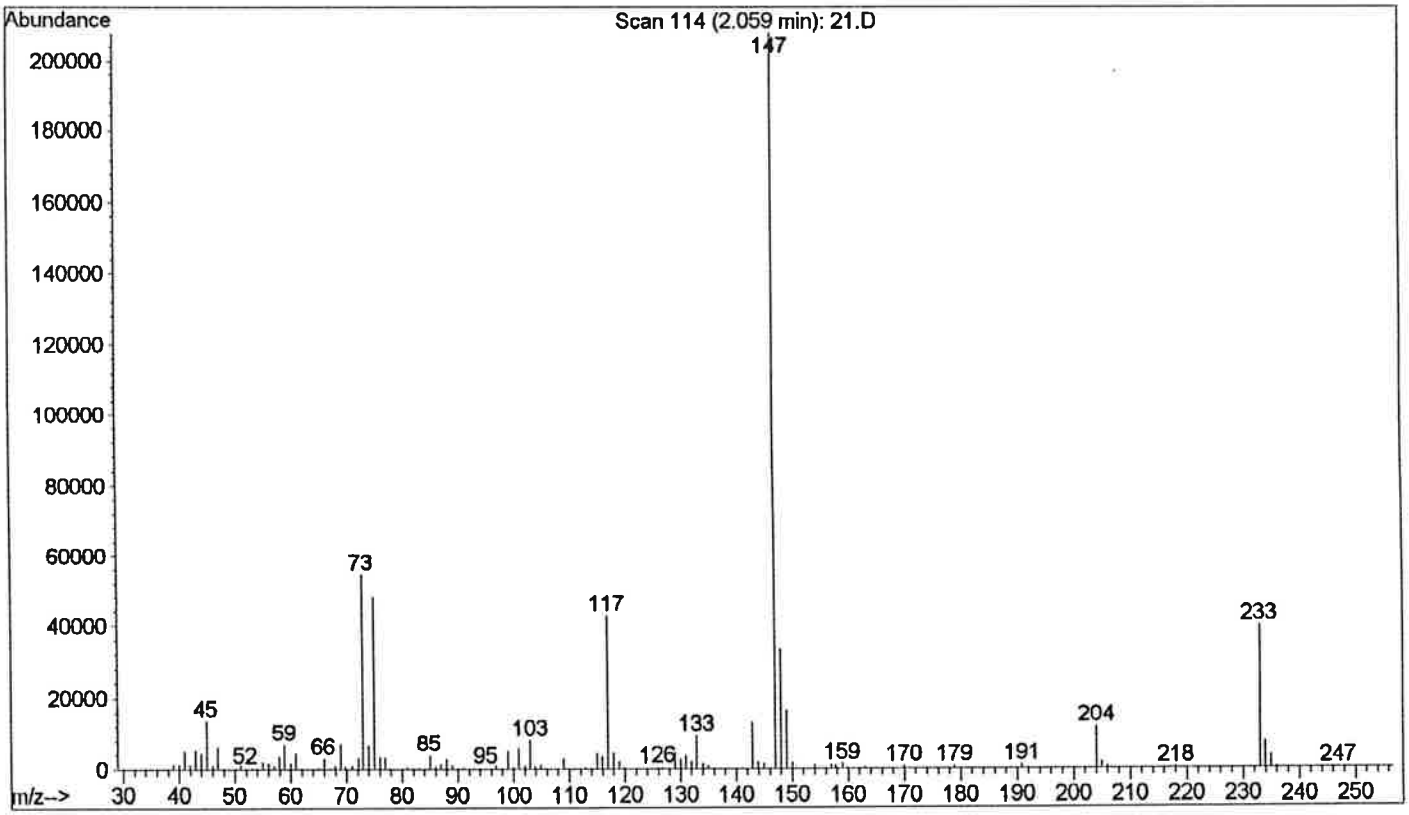
File : C:\HPCHEM\1\DATA\PWF\GBD-22.D
Operator : SEJ
Acquired : 7 Jul 1999 10:47 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN GATORADE
Misc Info : USING TRIM PROCEDURE IV
Vial Number: 1





File : C:\HPCHEM\1\DATA\PWF\GHBD-21.D
Operator : JEJ
Acquired : 7 Jul 1999 10:58 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB IN RUM AND COKE
Misc Info : USING TRIAC PROCEDURE IV
Vial Number: 1





NOTES

In addition to derivatizing GHB, we ran many additional test to ensure accurate results. To be certain that GBL would not interfere with the results, derivatizing GBL with TCMS and BSTFA was attempted. The spectra of GHB-TMS and that of the derivatized GBL were in no way similar. Because GBL will not derivatize the way GHB does, it is not essential to remove all GBL if you are just trying to establish the presence of GHB. An FTIR of GBL was also obtained by smearing some of the GBL on a pellet of KBr. Again, it was simple to distinguish between the IR of GHB and that of GBL.

Because of its similarity with GHB, 1,4-Butanediol was a concern.

$$HO\text{-}CH_2CH_2CH_2CH_2\text{-}OH$$

1,4-BUTANEDIOL

1,4-Butanediol is a clear, colorless solution that converts to GHB in the body. After obtaining a mass spectrum, a UV\VIS, and an FTIR of 1,4-Butanediol, it was clear that 1,4-Butanediol could not be confused with GHB. We also attempted derivatizing 1,4-Butanediol with BSTFA and TCMS and did not see any similarities when compared to the mass spectrum of GHB-TMS.

REFERENCES

A. Andrews, Kathleen A., DEA Western Regional Laboratory, Personal Communication.

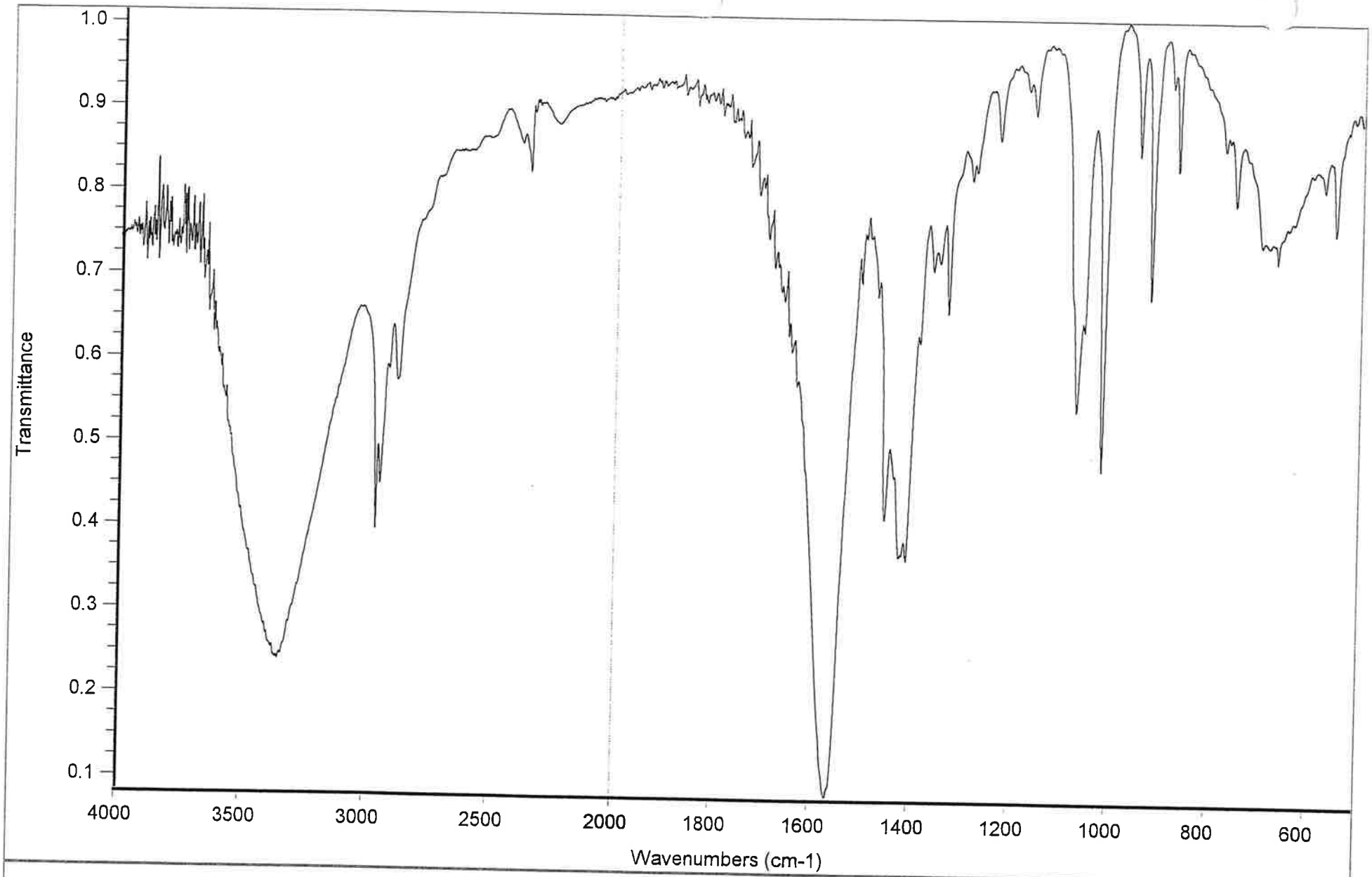
B. Andrews, Kathleen A. Presentation Notes: The Headaches of Analyzing a GHB Sample.

C. Blackledge, Robert D. and Miller, Michael D., Naval Investigative Service Regional Forensic Laboratory. "The Identification of GHB," Microgram, Vol. XXIV, No. 7, July 1991.

D. Newman, Reta, Pinellas County Forensic Laboratory, Personal Communication.

E. Newman, Reta. Procedure and Method Validation for the Analysis of Suspected GHB Cases in Aqueous Solutions.

F. Walker, Lara, California Department of Justice. "Identification of the Potassium Salt of Gamma-Hydroxybutyrate ($GHB\text{-}K^+$)," Journal of the Clandestine Laboratory Investigating Chemists, Vol. 9, p. 17, January 1999.



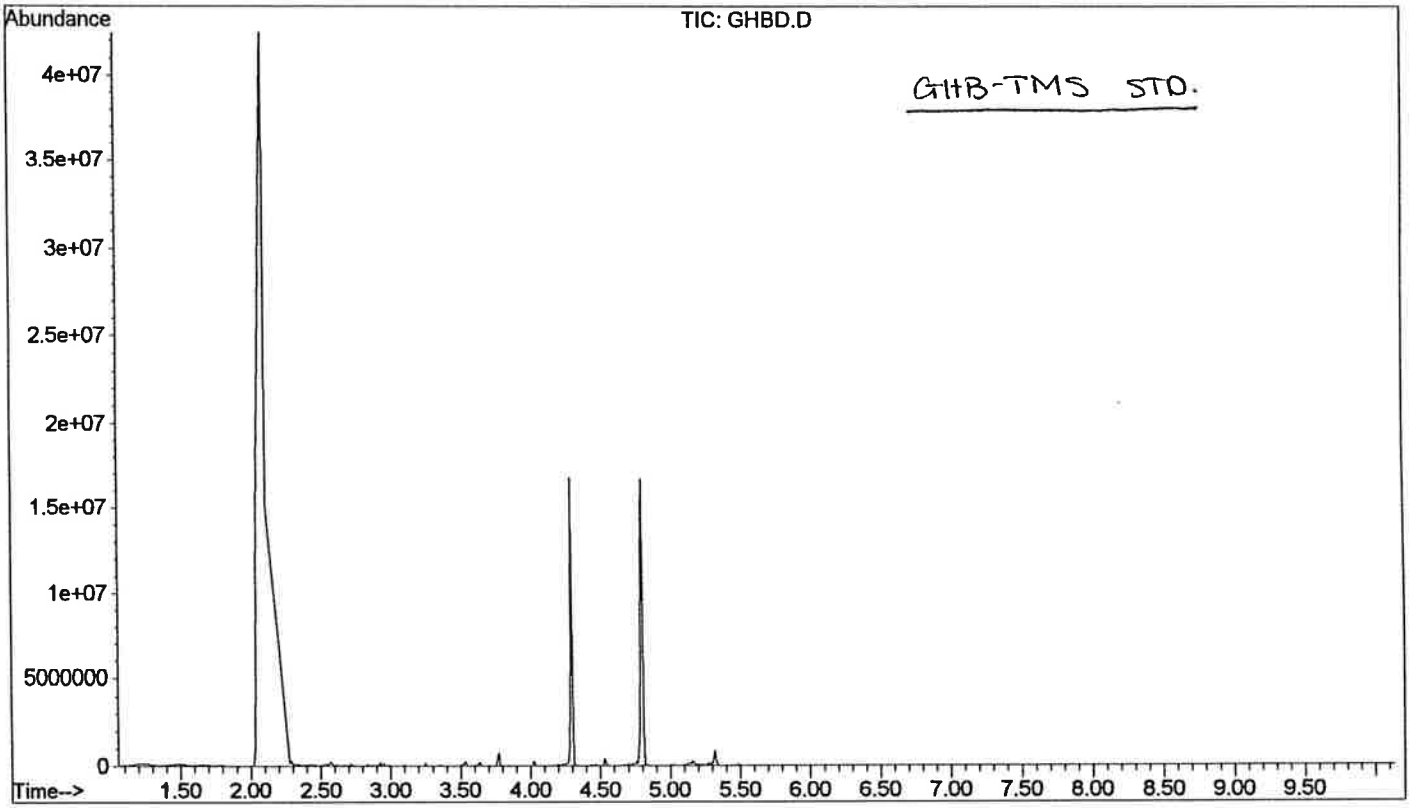
Date: Thu Dec 05 08:51:20 1996

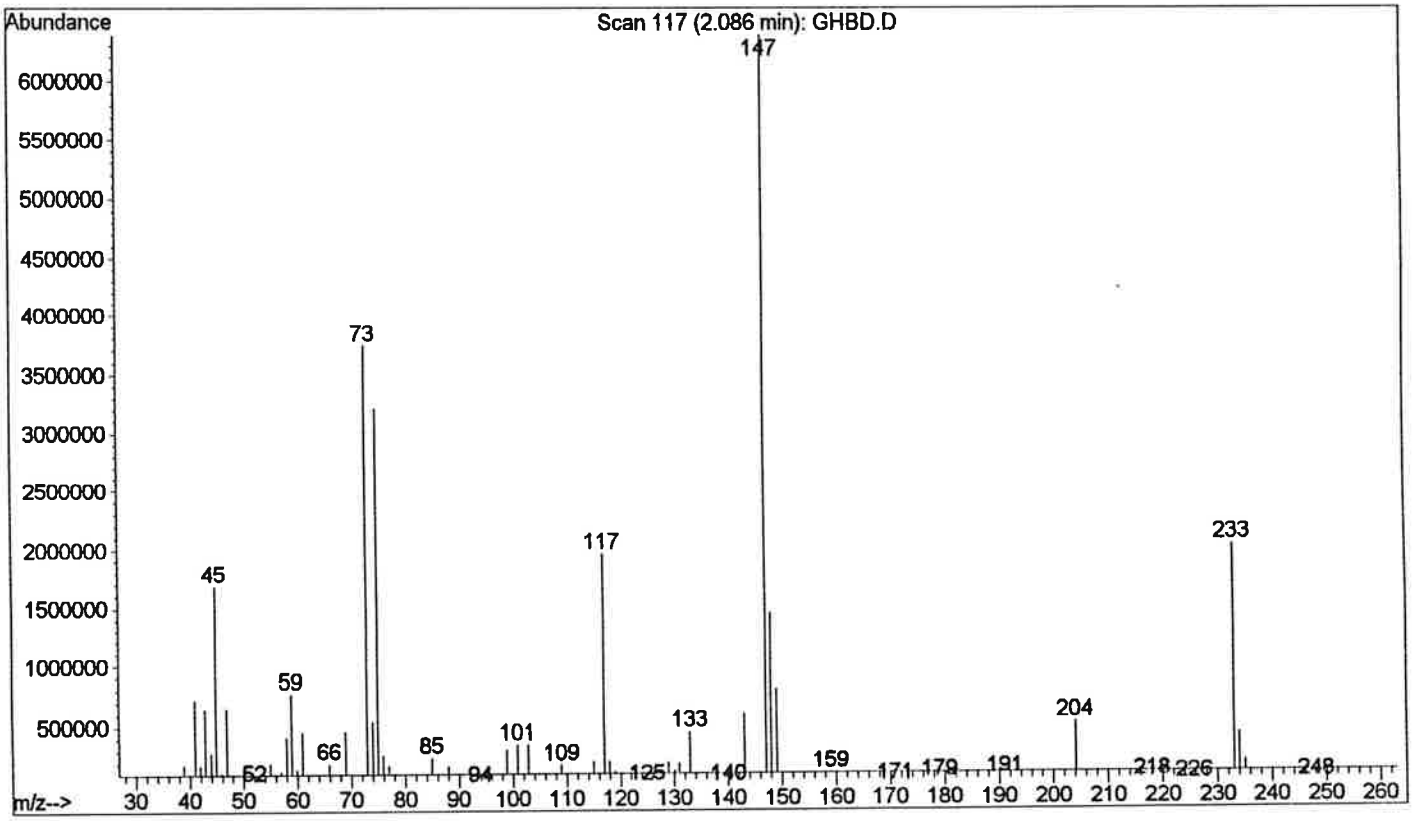
GAMMA-HYDROXYBUTYRIC ACID , SODIUM (GHB) PWF

Scans: 10

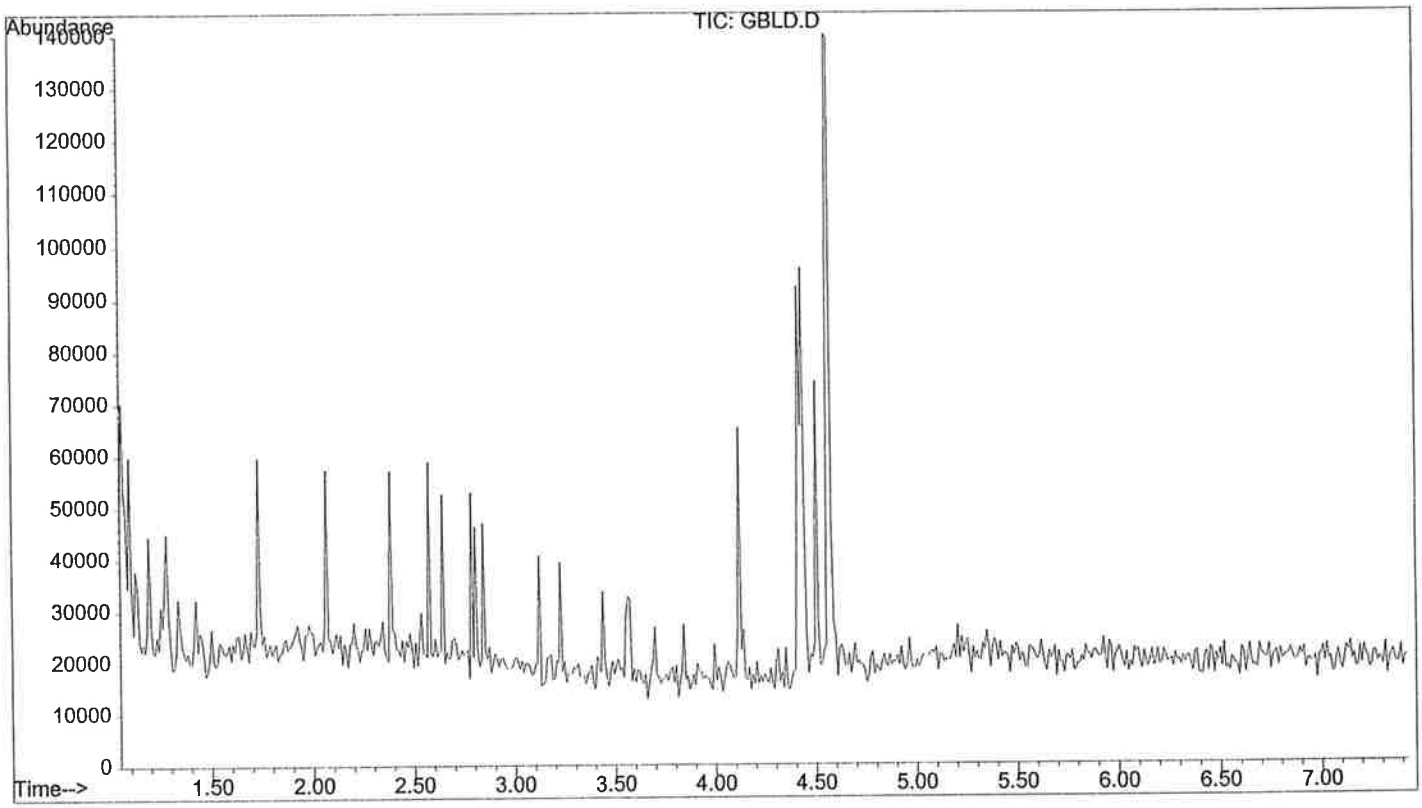
Resolution: Unknown

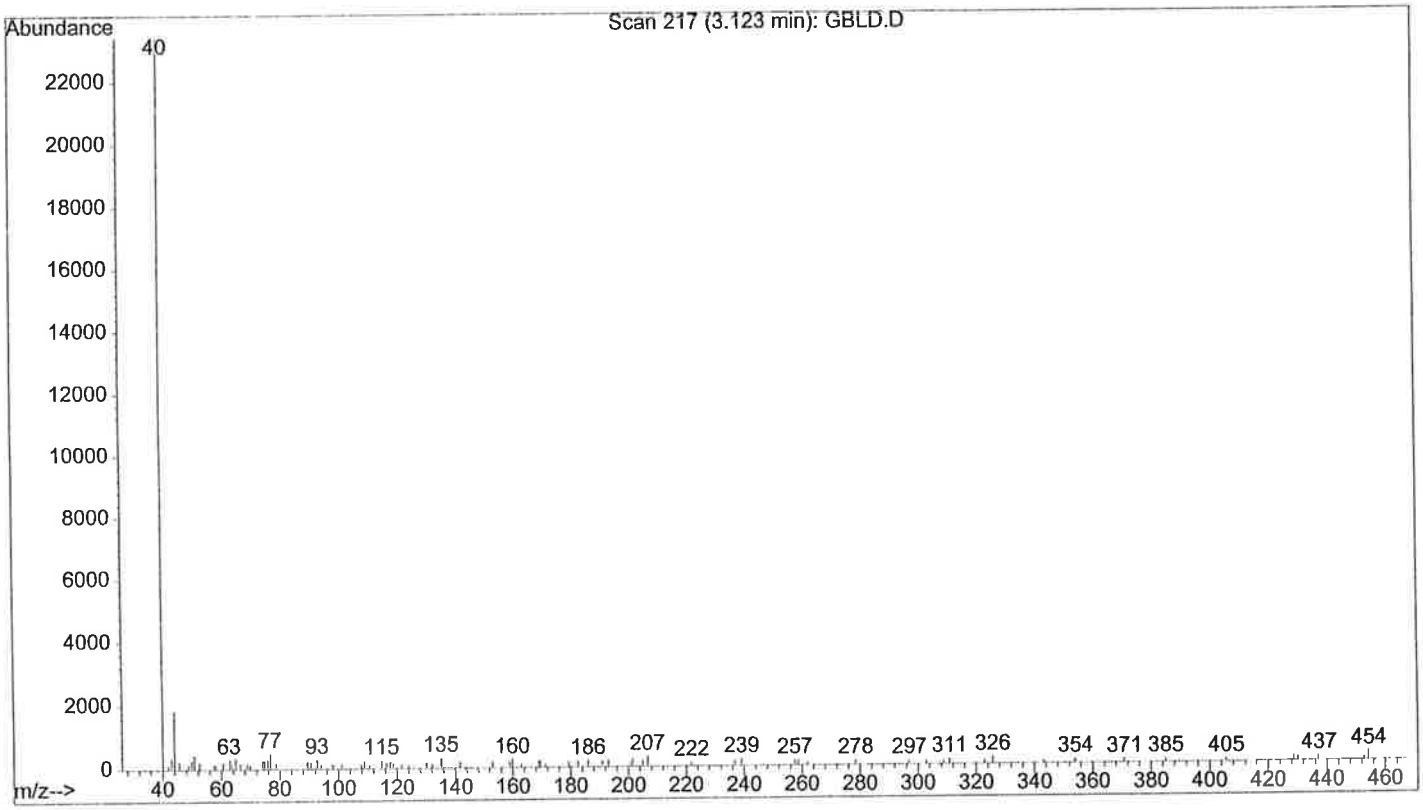
File : C:\HPCHEM\1\DATA\PWF\GHBD.D
Operator : JEJ
Acquired : 14 Jun 1999 10:46 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB-TMS
Misc Info : DERIVATIZING GHB USING TRIAK PROCEDURE I
Vial Number: 1

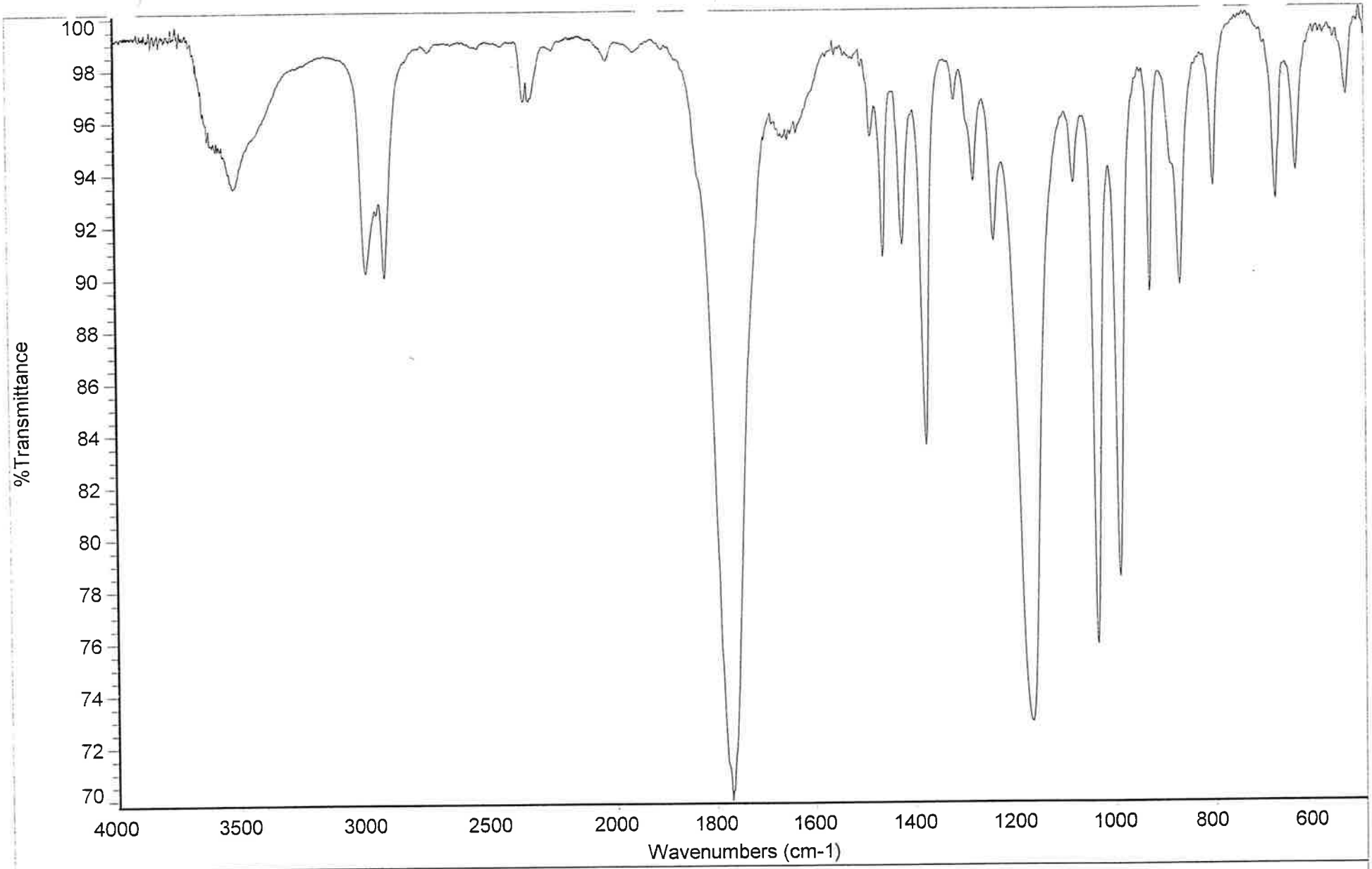




File : C:\HPCHEM\1\DATA\PWF\GBLD.D
Operator : SES
Acquired : 14 Jun 1999 14:43 using AcqMethod MSDRUGS
Instrument : Donna
Sample Name: GBL DERIVATIVE
Misc Info : DERIVATIZING GBL WITH BSTFA AND TCMS
Vial Number: 1





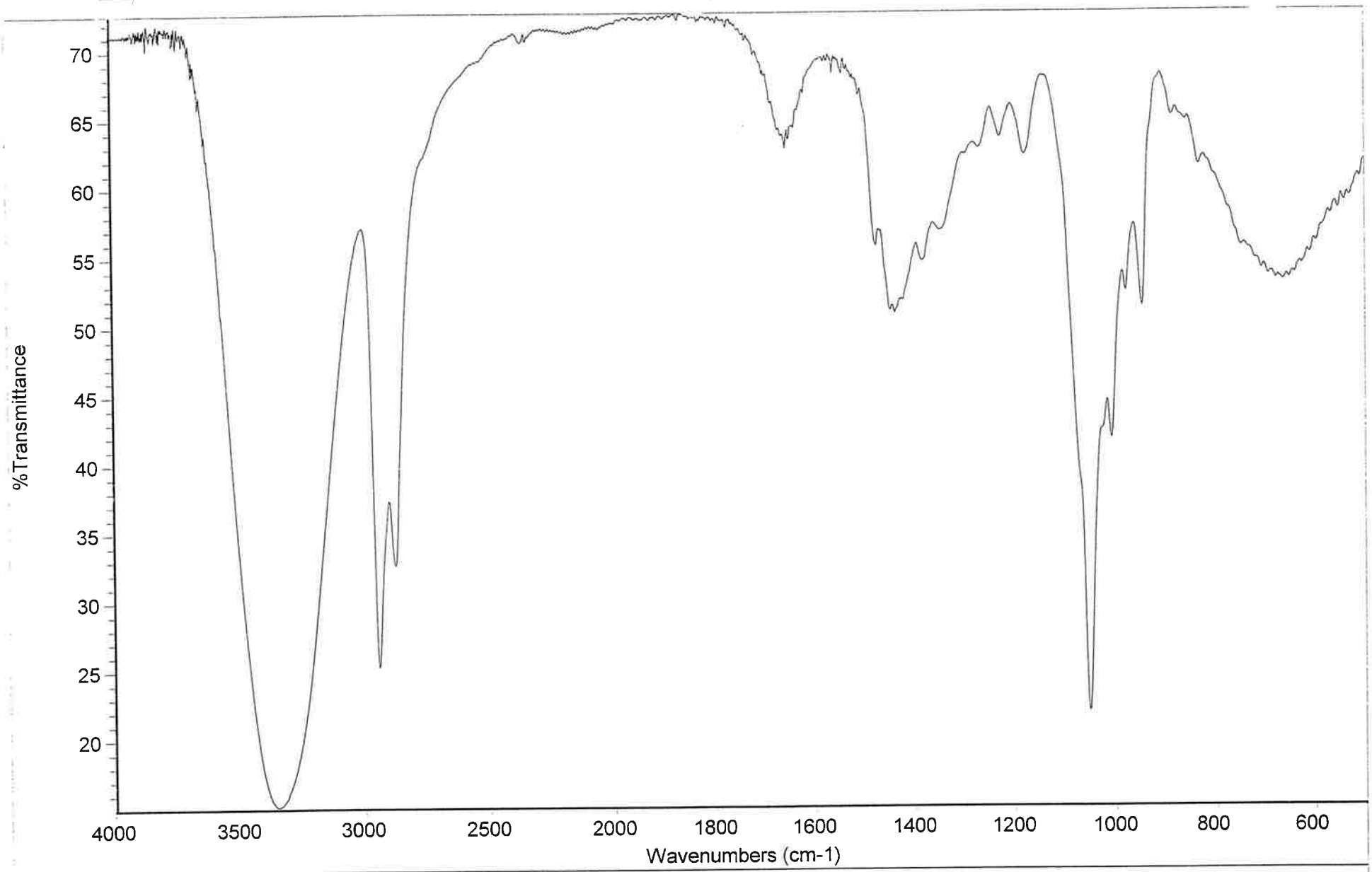


Date: Mon Jul 19 13:43:32 1999

*GAMMA HYDROXYBUTYROLACTONE STD NEAT JES

Scans: 10

Resolution: 4.000



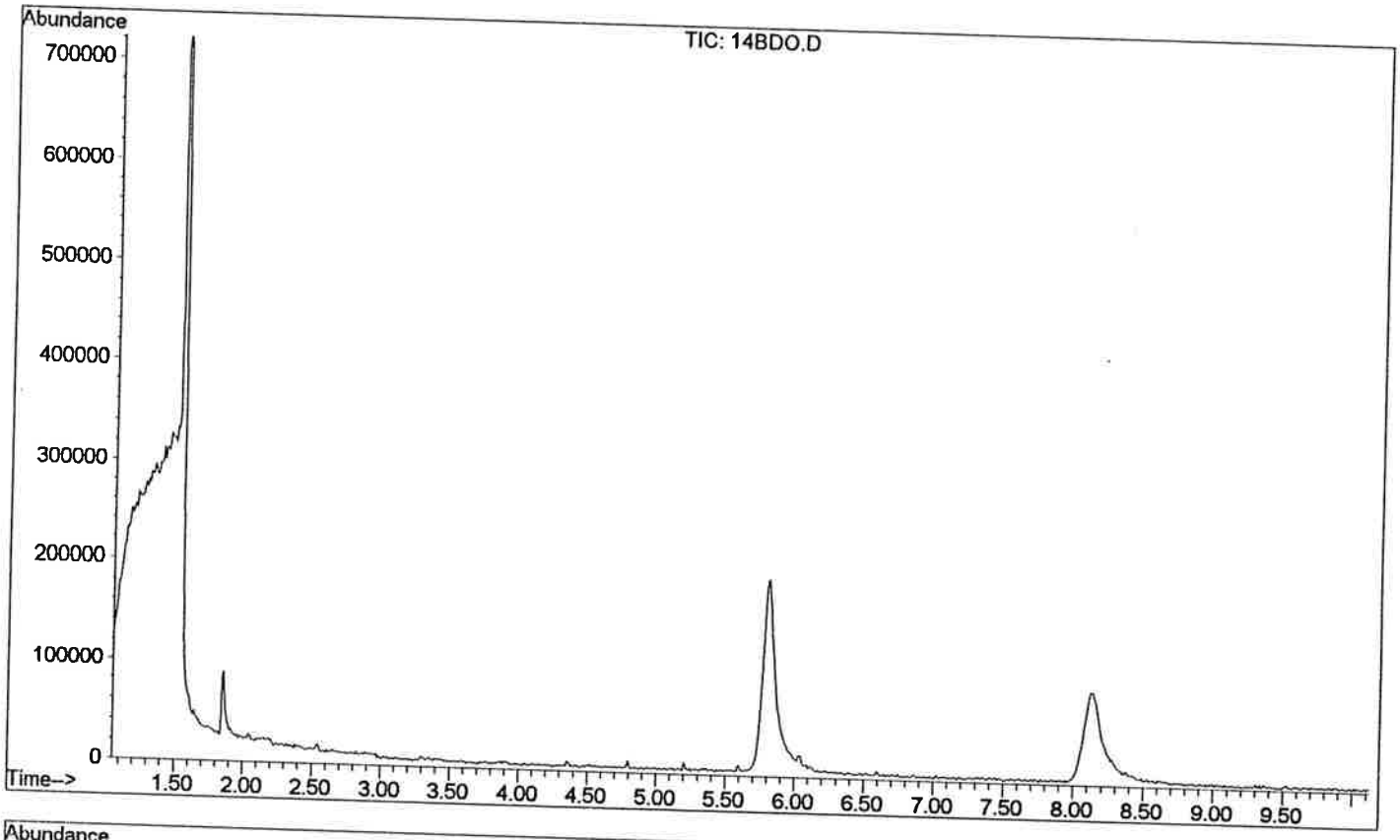
Date: Mon Jul 19 09:18:42 1999

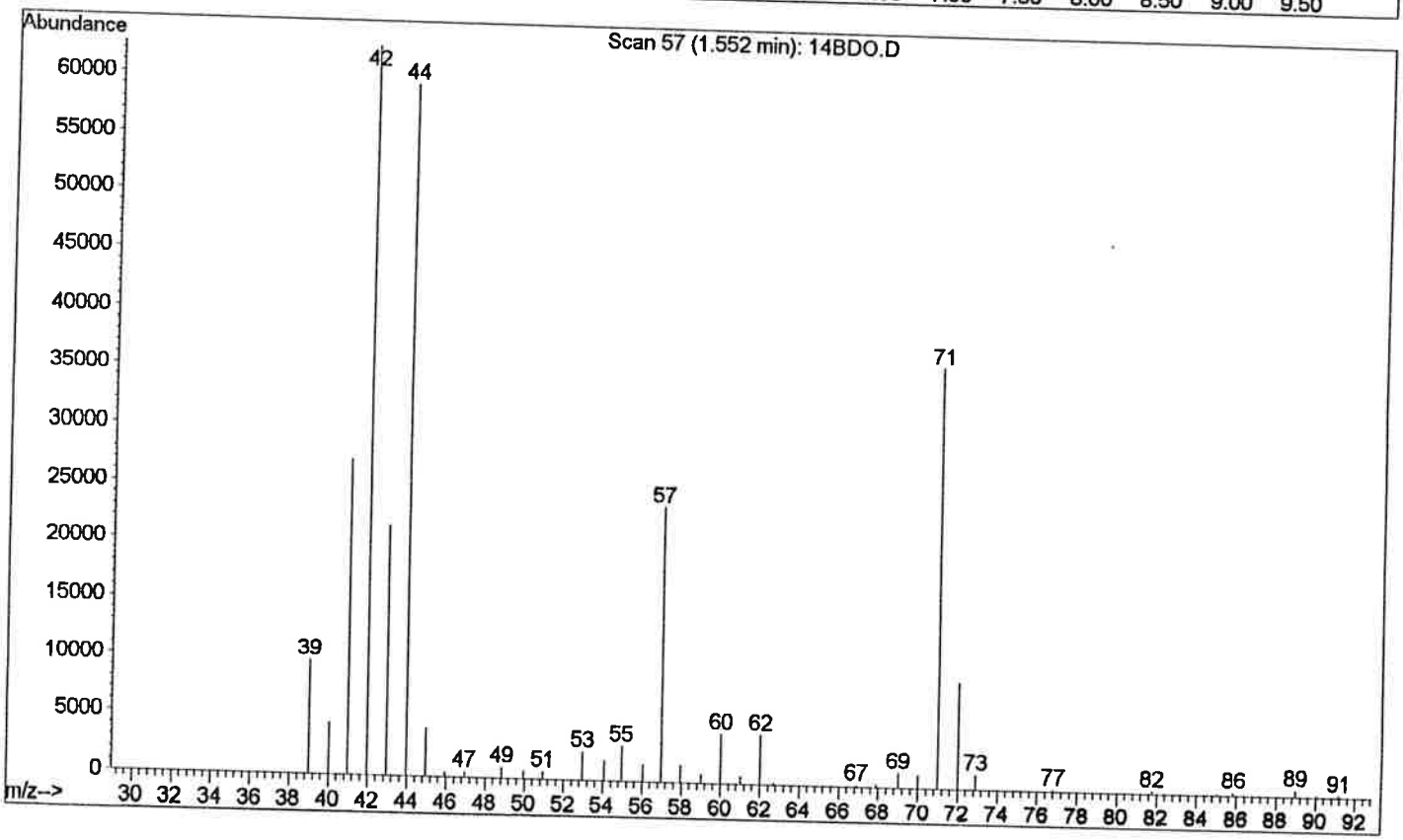
1,4-BUTANEDIOL STD NEAT ON KBR JEJ

Scans: 10

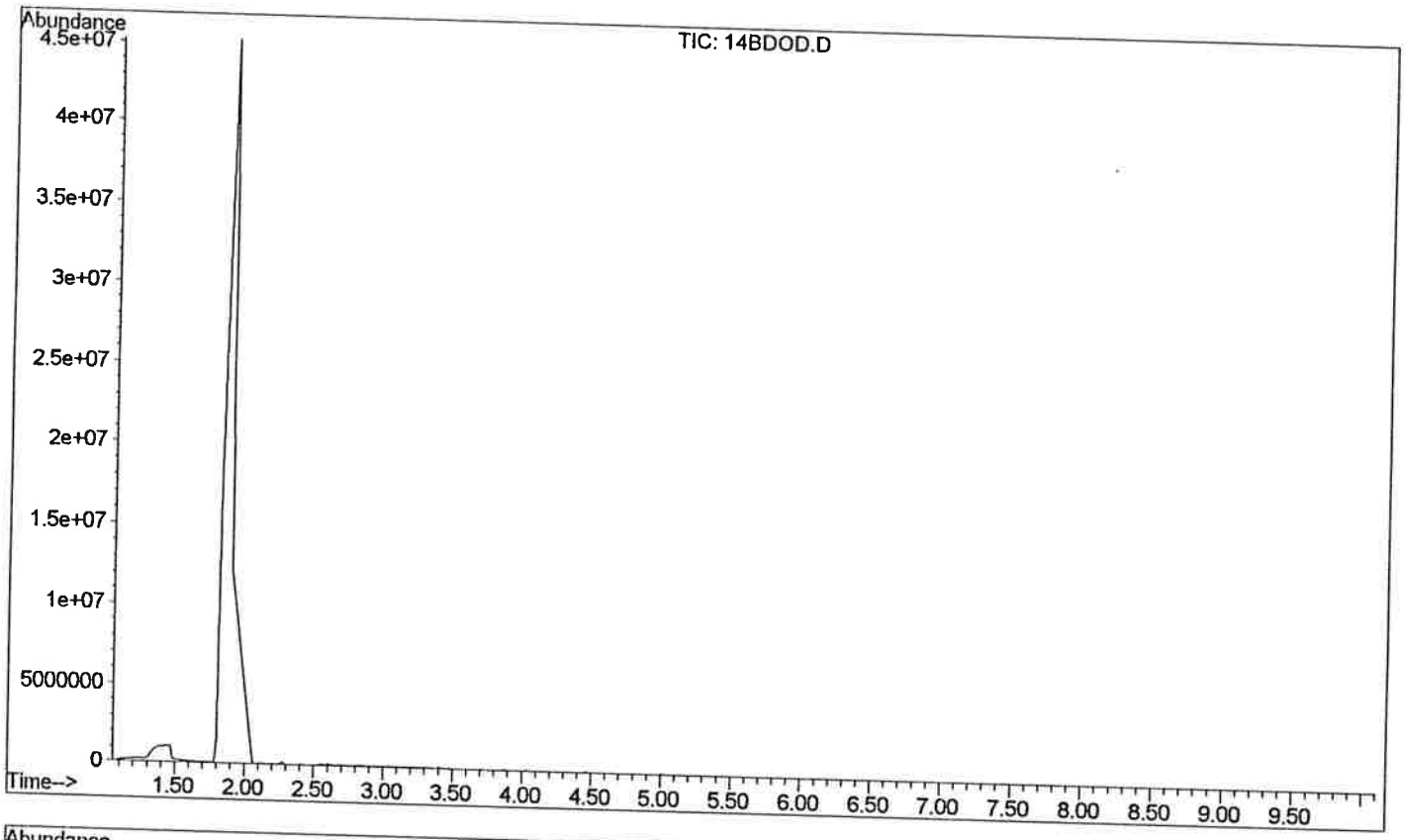
Resolution: 4.000

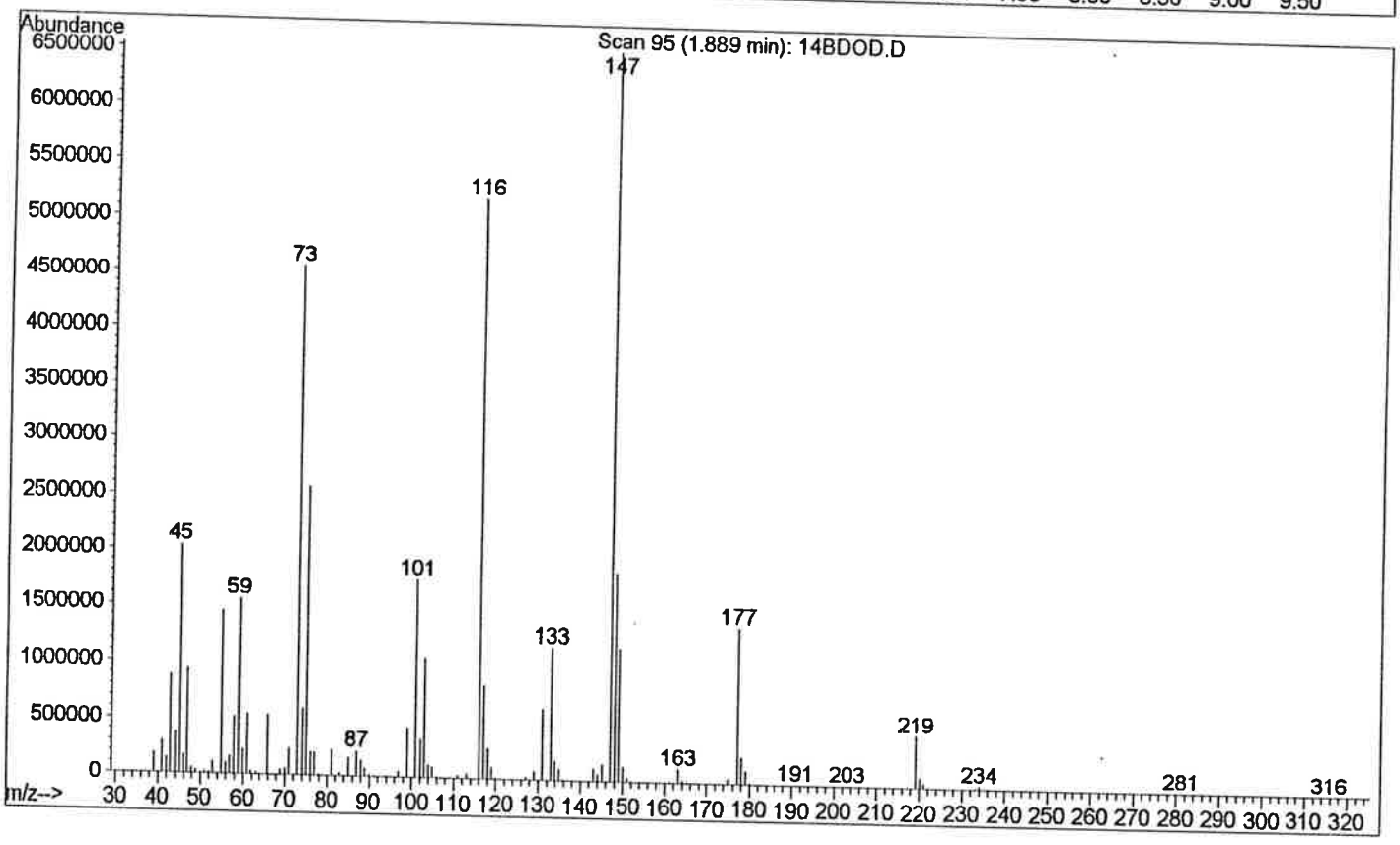
File : C:\HPCHEM\1\DATA\PWF\14BDO.D
Operator : JENNIFER JOHNSON
Acquired : 19 Jul 1999 10:00 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: 1,4-BUTANEDIOL STD
Misc Info : NEAT IN METHANOL
Vial Number: 1



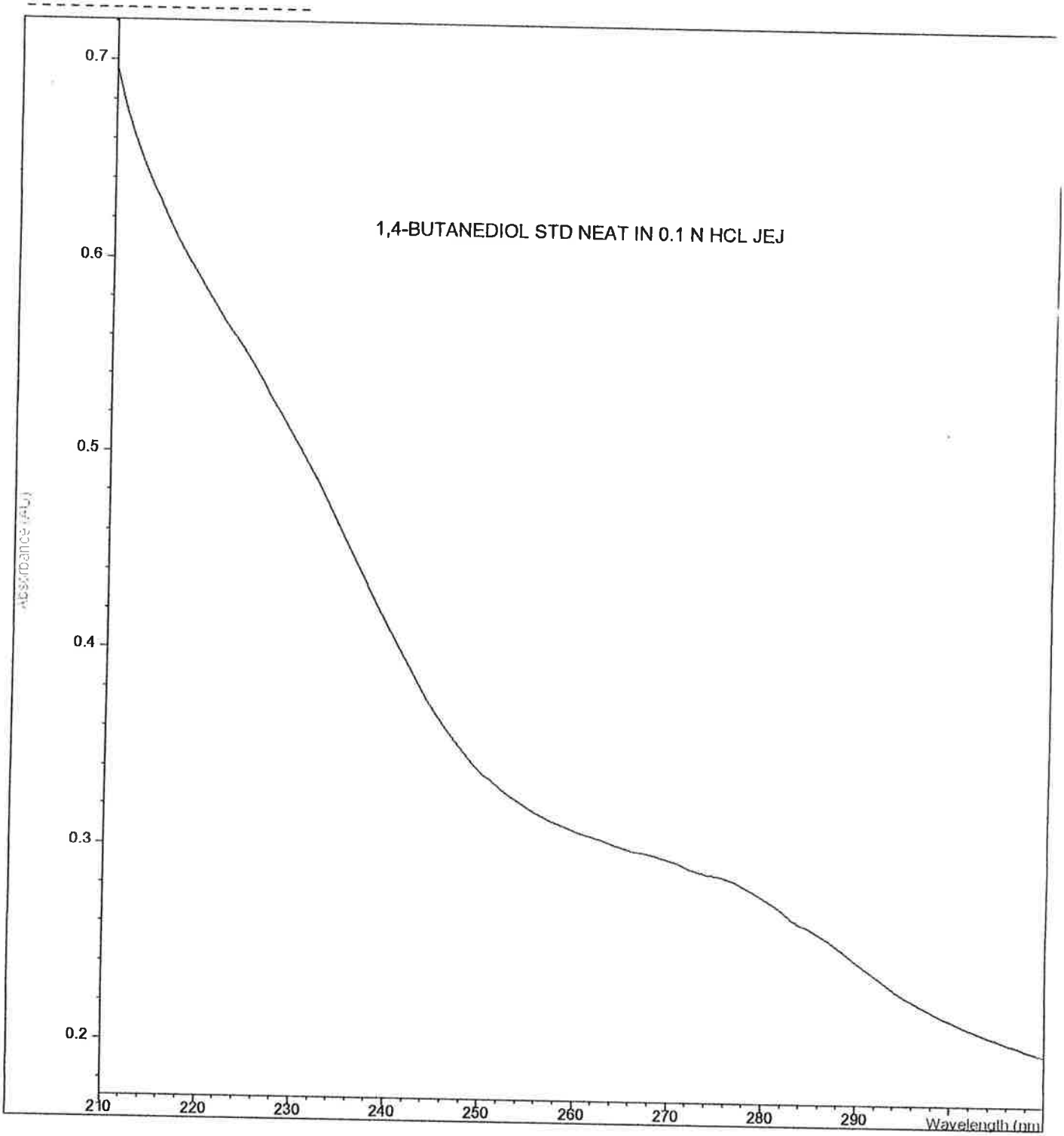


File : C:\HPCHEM\1\DATA\PWF\14BDOD.D
Operator : JEJ
Acquired : 23 Jul 1999 10:41 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: 1,4-BUTANEDIOL DERIVATIVE
Misc Info : DERIVATIZING 1,4- BUTANEDIOL WITH BSTFA AND TCMS
Vial Number: 1





Last Sample Spectrum



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METHOD: IDENTIFICATION OF GHB IN A LIQUID BY SILYLATION

1. SAMPLE REQUIRED

   Unknown Liquid

2. SCIENTIFIC INSTRUMENTS

   A. GC\MS

3. CHEMICALS REQUIRED

   A. Acetone

   B. Acetonitrile

   C. Bis-(Trimethylsilyl) trifluoroacetamide (BSTFA)

   D. Trimethylchlorosilane (TCMS)

4. PROCEDURE

   A. Extract 1-5 mL of your unknown sample with excess $CHCl_3$. ~~Extract once more with an~~ equal amount of $CHCl_3$. This will remove any residual GBL from your sample.

   B. If possible, dry down extracted sample in a small (10 mL) beaker. This can be accomplished by using a hair dryer; however, do not exceed 150 C or the GHB may convert to GBL.

      NOTE: If sample turns syrupy upon drying it down, you will need to recrystallize with acetone. To recrystallize, add 10 mL of acetone to the beaker containing your partially dry sample. Heat on a hot plate at about 70 C (b.p. of acetone-57 C) with stirring until sample boils. This step should take no longer than five minutes. Immediately pour the hot solvent into another small beaker leaving the gummy substance behind. Dry off the acetone using a hair dryer on low heat.

   C. To the beaker containing your dry sample, add 2 mL of acetonitrile, 500 uL of BSTFA, and 500 uL of TCMS. Now, heat the solution on a hot plate at 60 C-80 C covered for 10-15 minutes. Do not exceed 80 C, and do not let the solution boil.

   D. Analyze your product on a GC\MS. GHB-TMS should come off of the column at

about two minutes using MSDRUGS.

5. NOTES

A. When working with derivatizing agents such as BSTFA and TCMS, safety glasses, gloves, lab coat, and a fume hood are required.

B. Water inhibits the derivatizing agents; therefore, it is imperative that your sample is as dry as possible.

C. To identify GHB-TMS, ions 117, 133, 159, 204, and 233 must be present. Ions common to TCMS derivatives include 73, 147, and 243.

6. REFERENCES

A. Andrews, Kathleen A., DEA Western Regional Laboratory, Personal Communication.

B. Andrews, Kathleen A. Presentation Notes: The Headaches of Analyzing a GHB Sample.

C. Blackledge, Robert D. and Miller, Michael D., Naval Investigative Service Regional Forensic Laboratory. "The Identification of GHB," Microgram, Vol. XXIV, No. 7, July 1991.

D. Newman, Reta, Pinellas County Forensic Laboratory, Personal Communication.

E. Newman, Reta. Procedure and Method Validation for the Analysis of Suspected GHB Cases in Aqueous Solutions.

F. Walker, Lara, California Department of Justice. "Identification of the Potassium Salt of Gamma-Hydroxybutyrate ($GHB\text{-}K^{+}$)," Journal of the Clandestine Laboratory Investigating Chemists, Vol. 9, p. 17, January 1999.

VALIDATION UNKNOWNS

Made by Jennifer Johnson on July 20, 1999

1. 22 mg of GHB in 10 mL of water
2. 10 mL of Gatorade
3. 10 mL of rum and Coke mixture
4. 20 mg of GHB in 10 mL of rum and Coke mixture

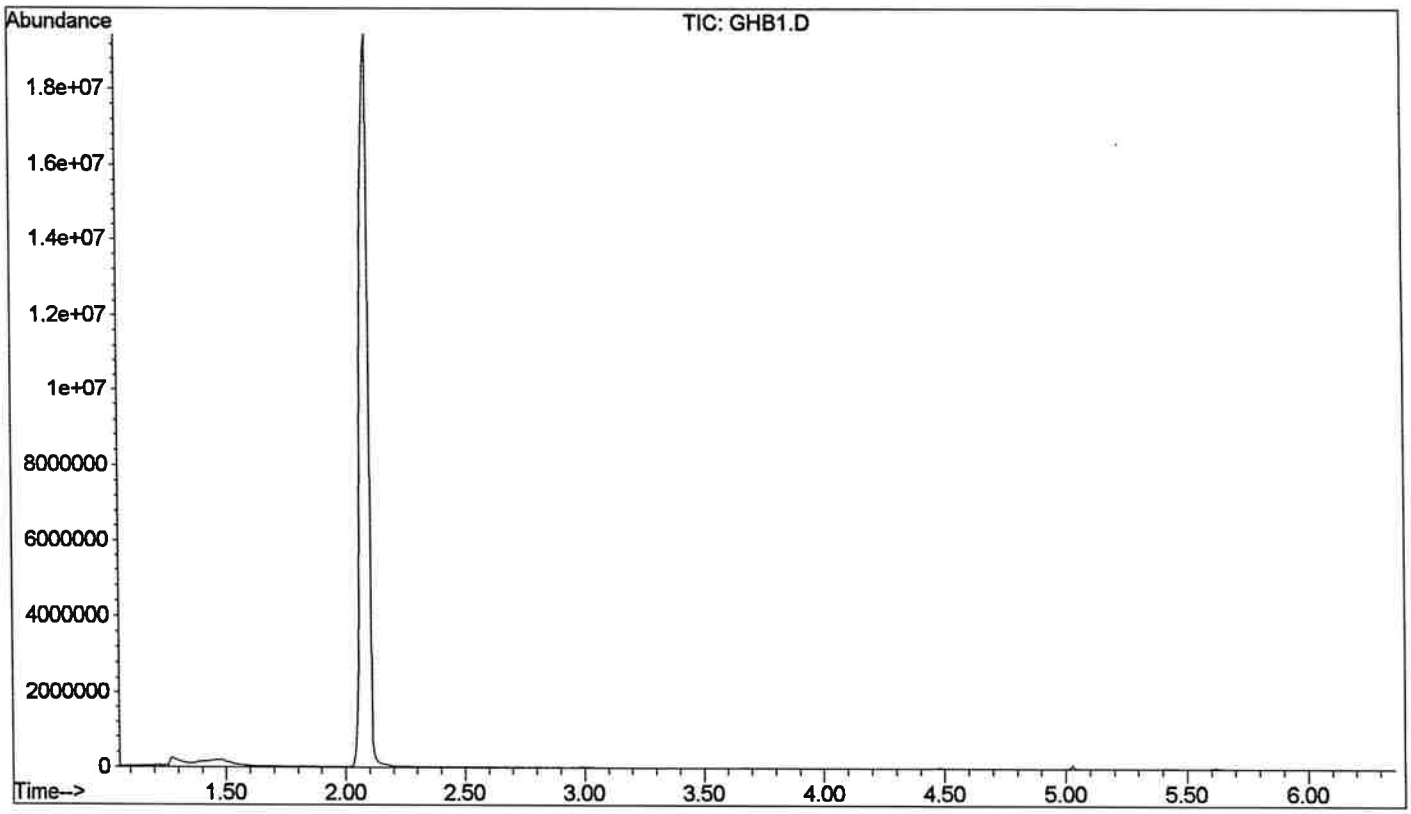
7/20/99 - Unknowns

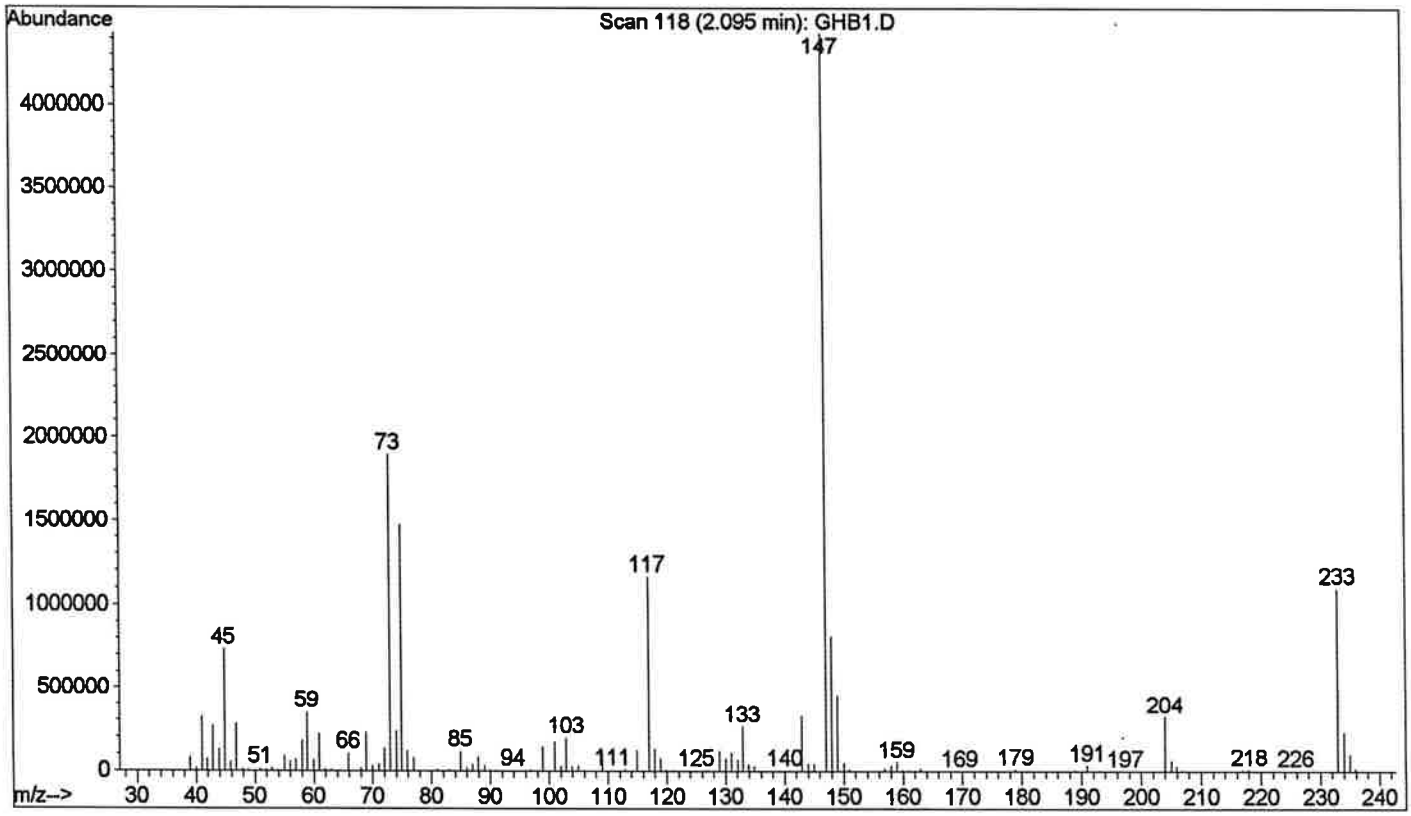
RESULTS:

1. positive for GHB
2. Negative for GHB
3. Negative for GHB
4. positive for GHB

Unknowns analyzed using Silylation of GHB Method on 7/20/99 - by Phillip W. Freeze

File : C:\HPCHEM\1\DATA\PWF\GHB1.D
Operator : PWF
Acquired : 20 Jul 1999 15:36 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB #1
Misc Info :
Vial Number: 1





File : C:\HPCHEM\1\DATA\PWF\GHB2.D
Operator : PWF
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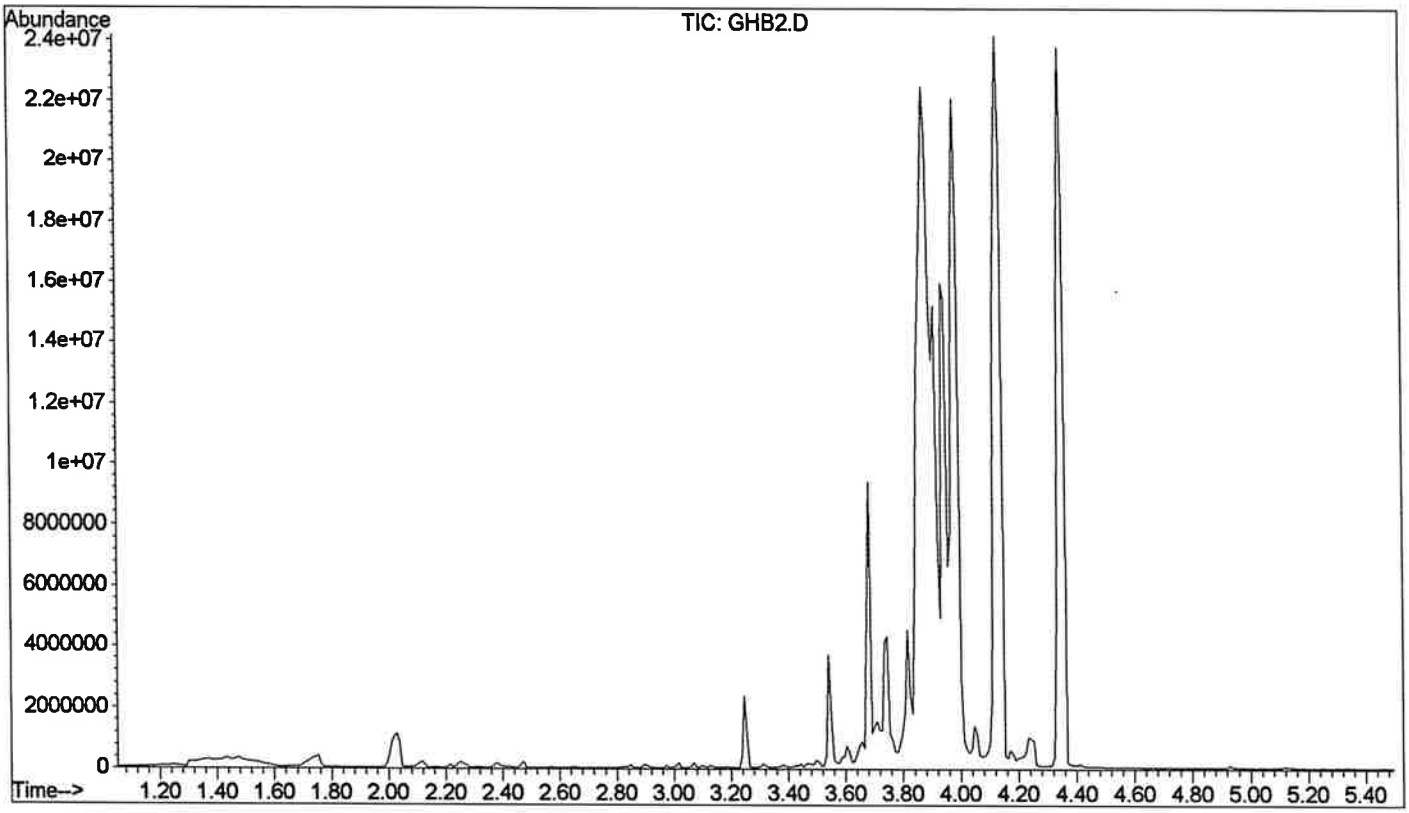
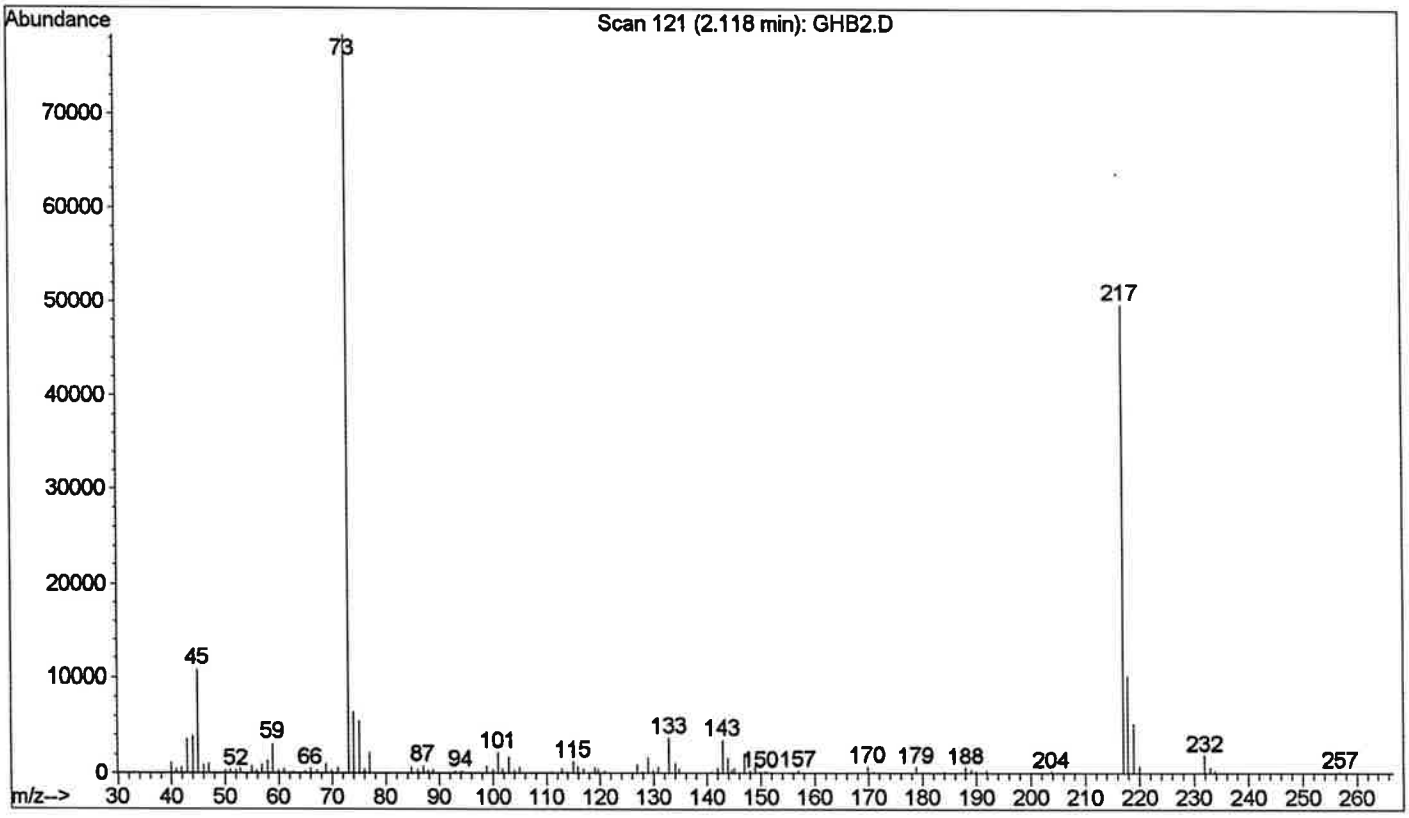
Instrument : Grumpy#1
Sample Name: GHB#2
Misc Info :
Vial Number: 1





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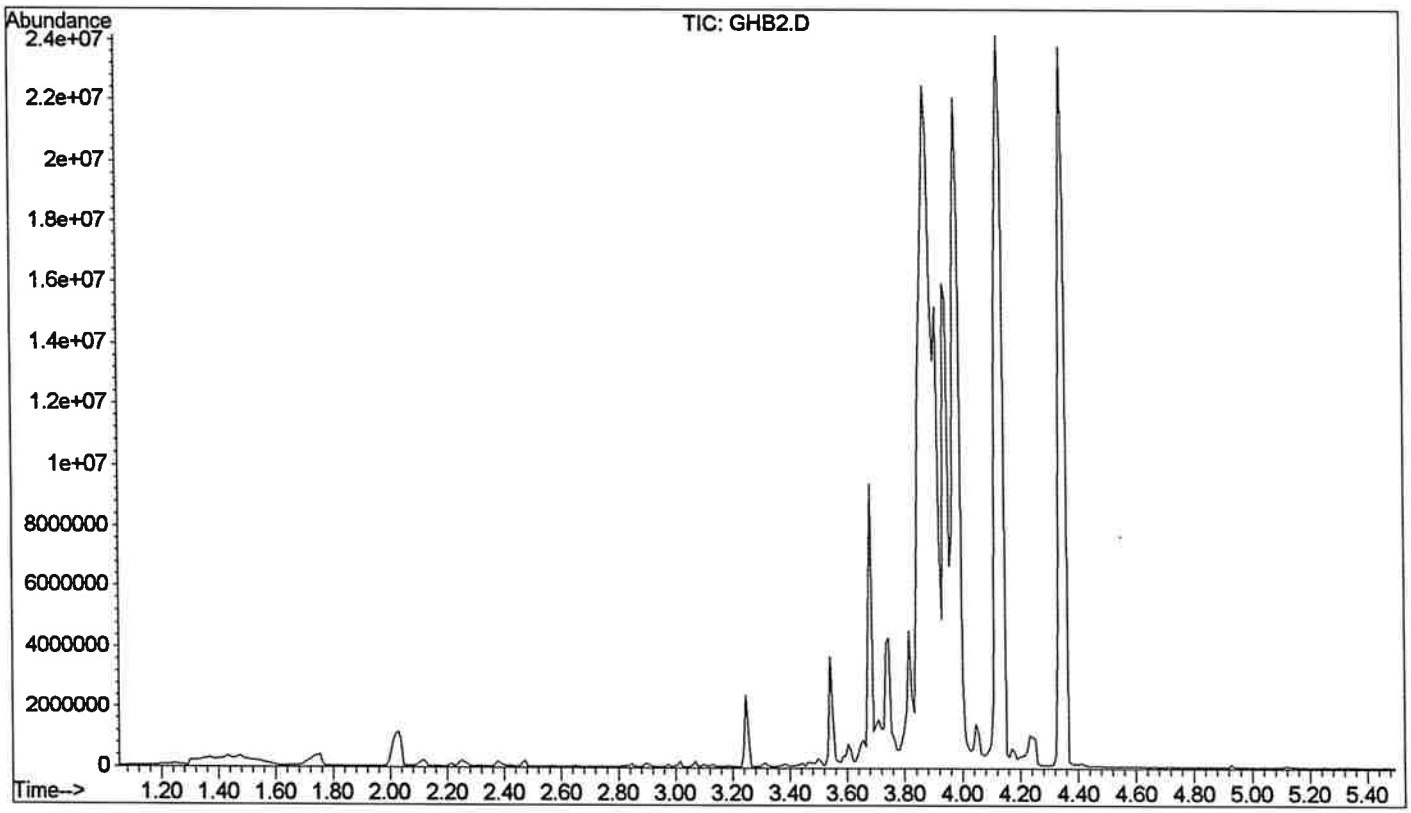
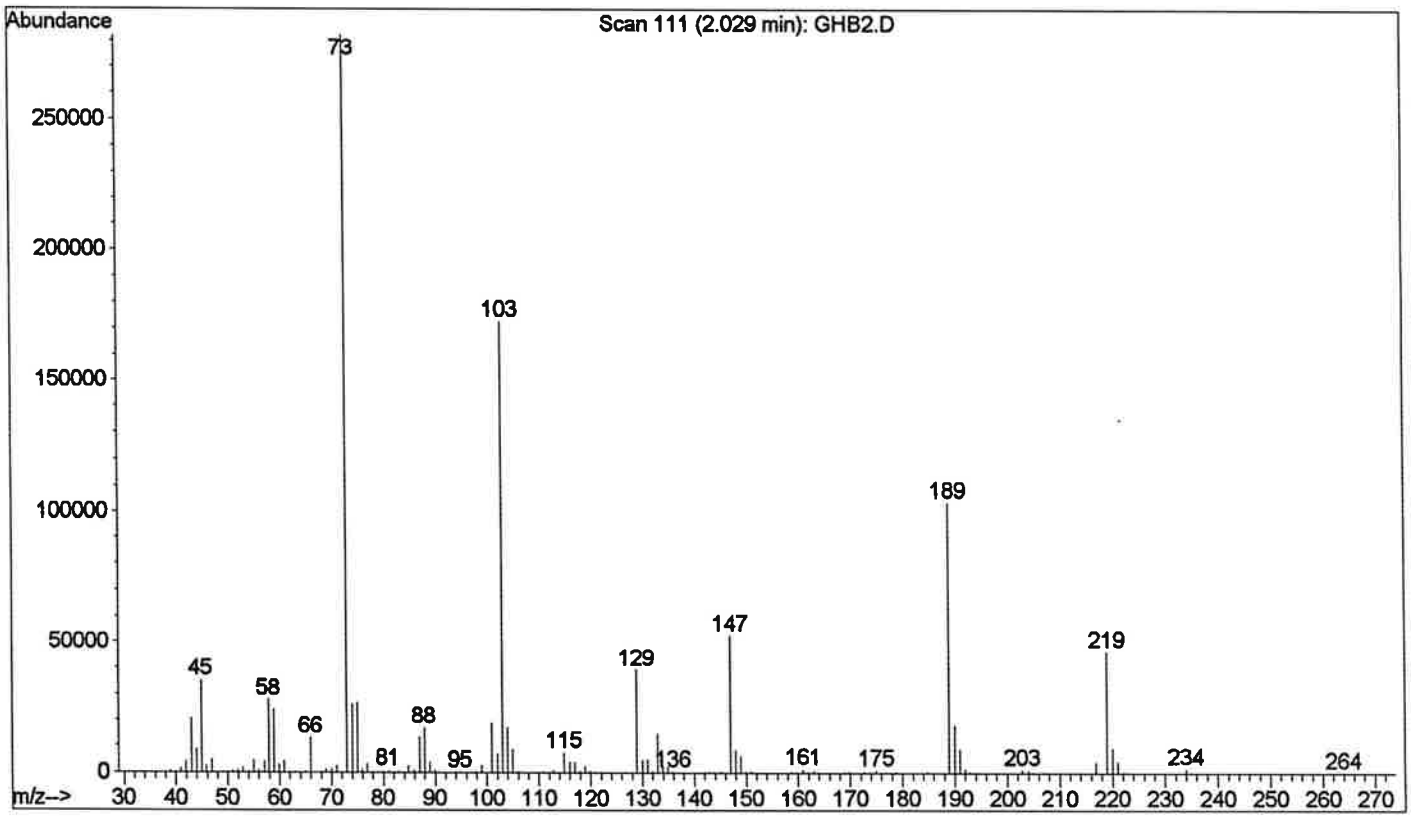
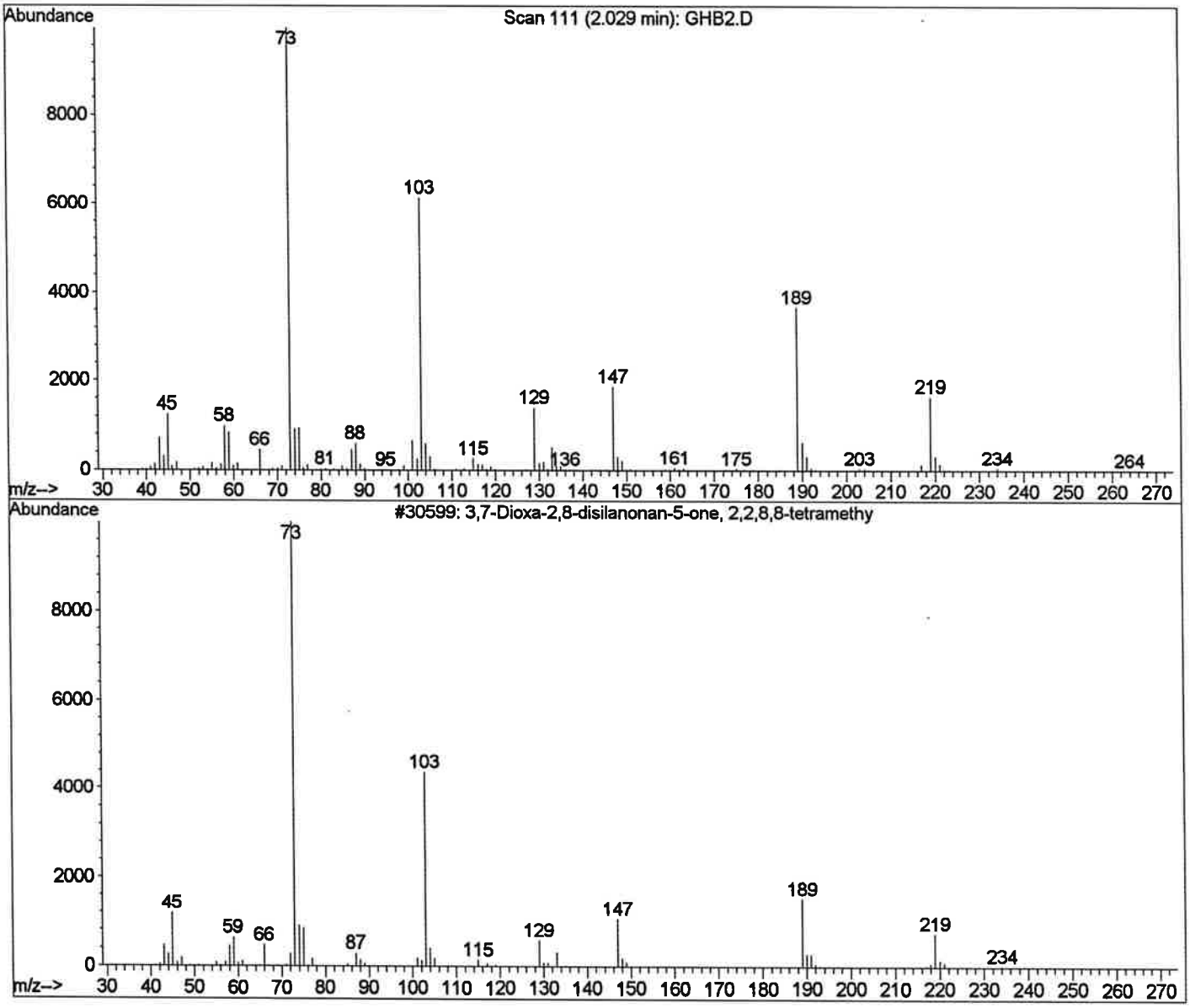
Instrument : Grumpy#1
Sample Name: GHB#2
Misc Info :
Vial Number: 1





Library Searched : C:\DATABASE\NBS75K.L
Quality : 94
ID : 3,7-Dioxa-2,8-disilanonan-5-one, 2,2,8,8-tetramethyl-



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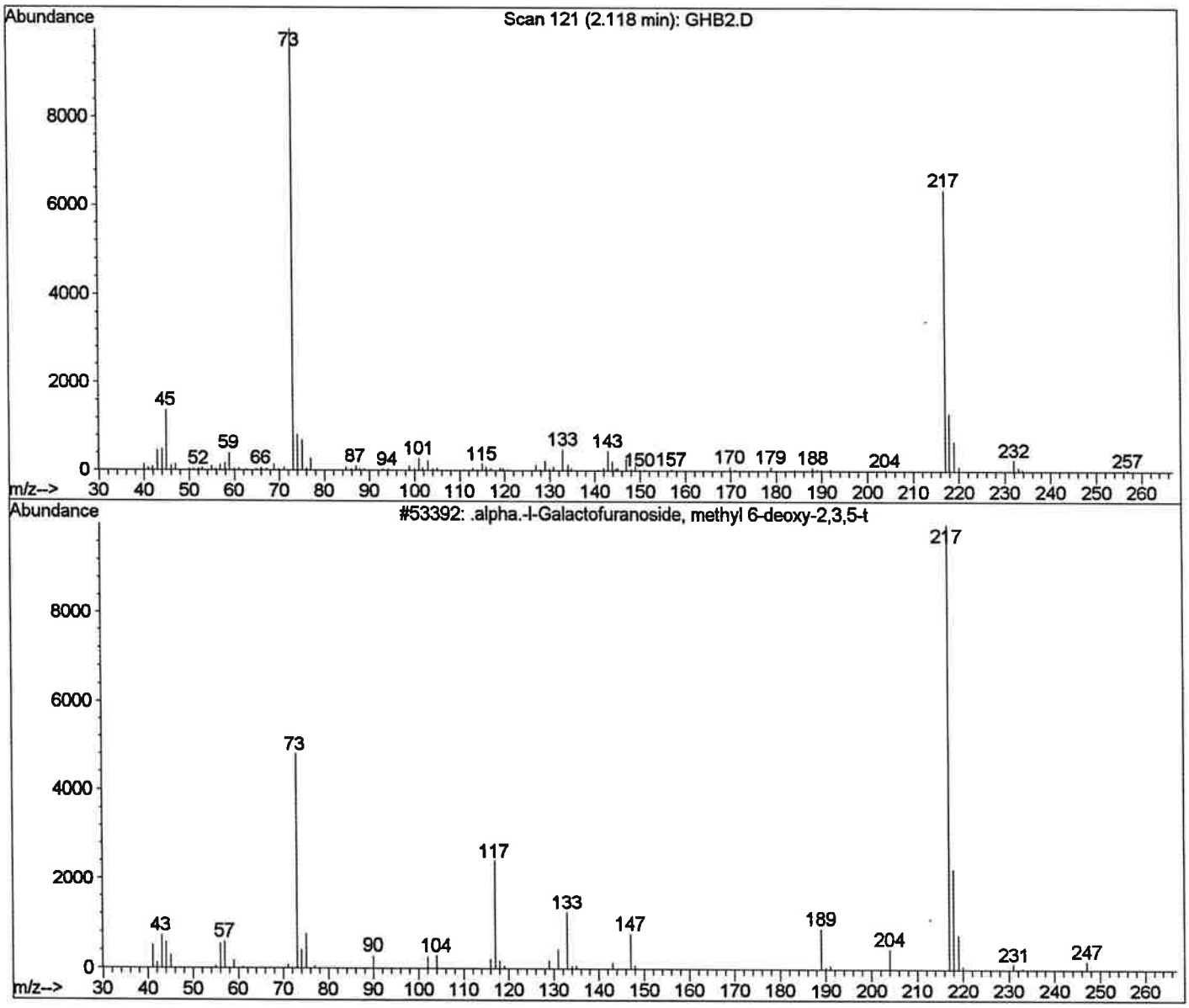
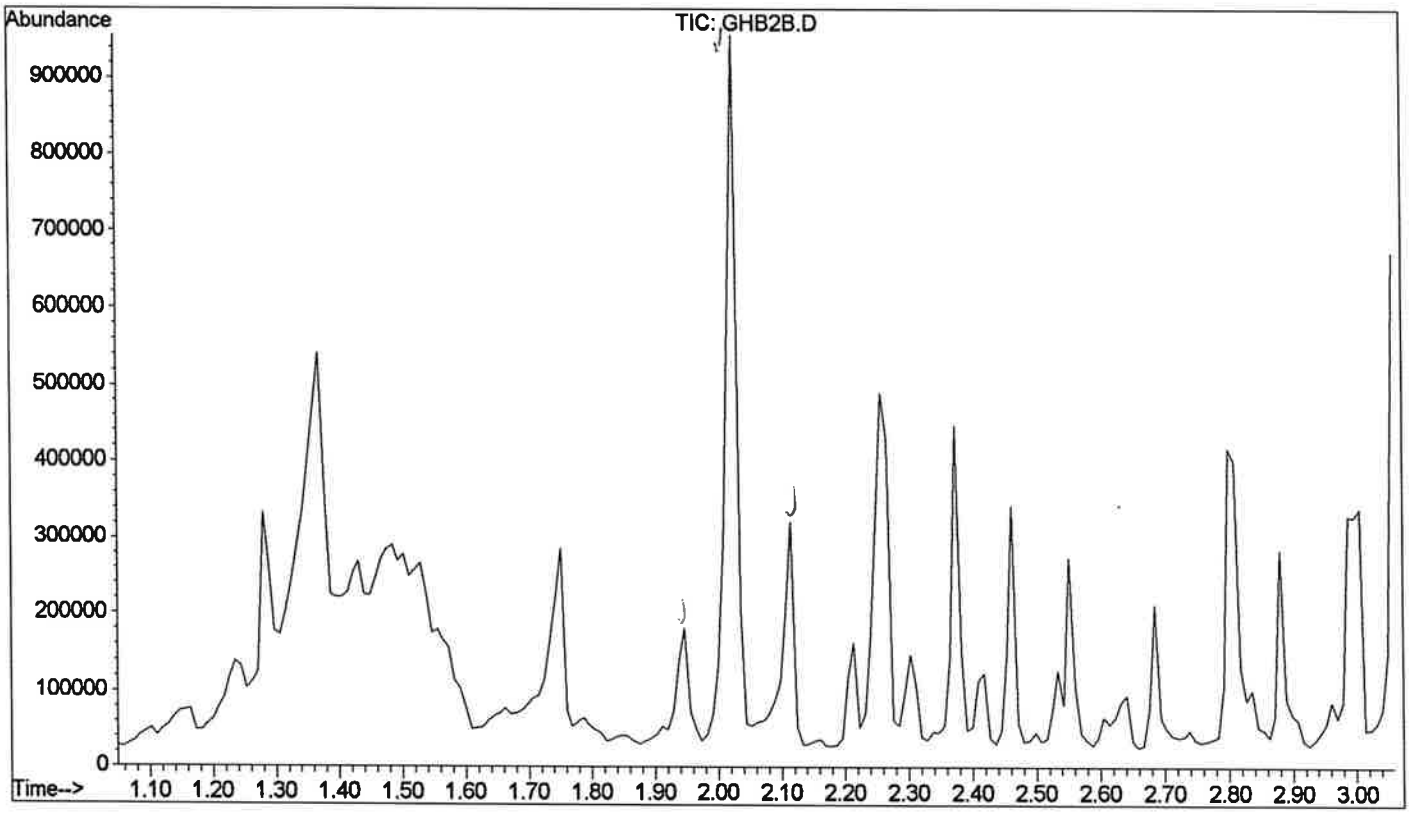
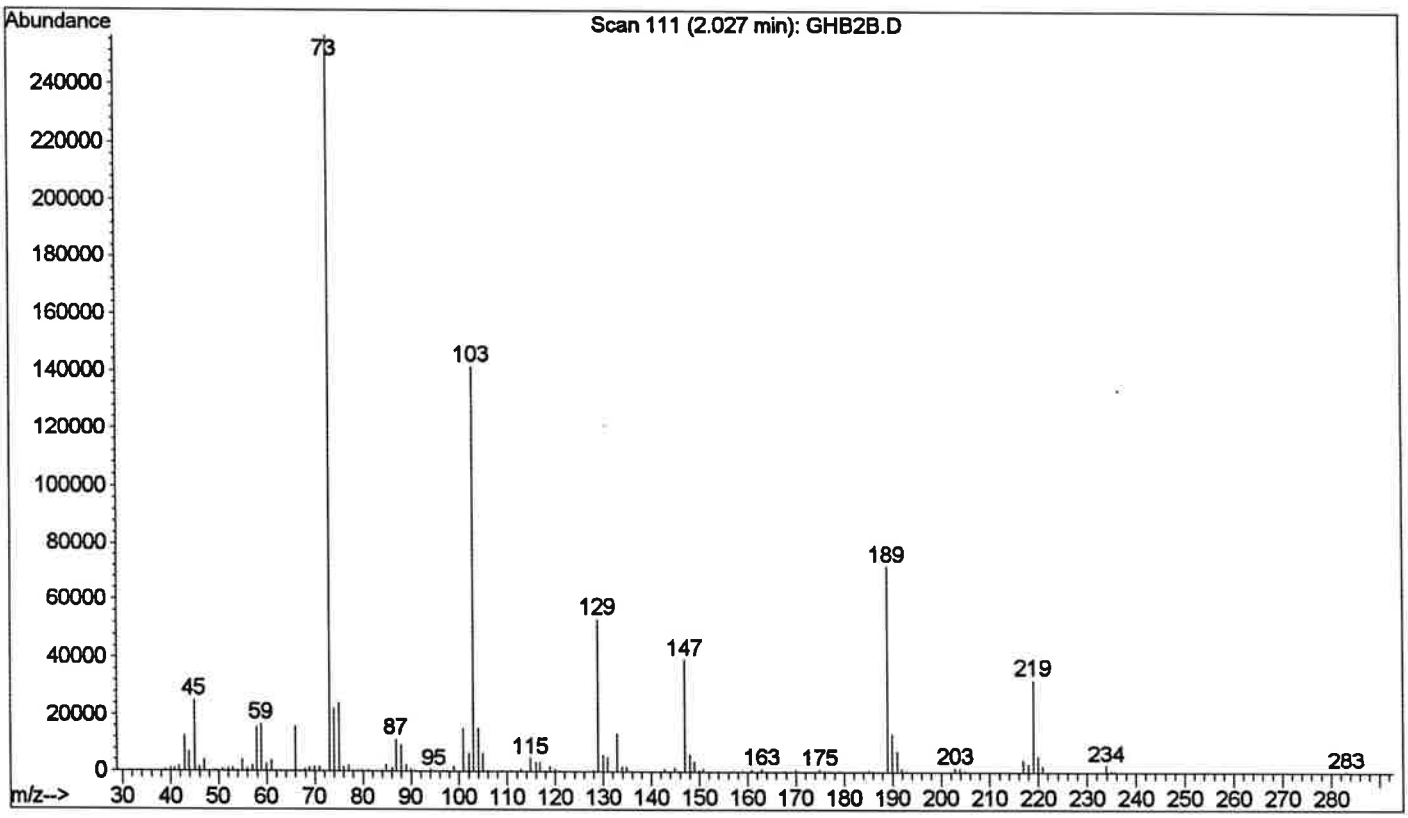
Quality : 64
ID : .alpha.-l-Galactofuranoside, methyl 6-deoxy-2,3,5-tris-O-(trimethylsilyl)-



File : C:\HPCHEM\1\DATA\PWF\GHB2B.D
Operator : PWF
Acquired : 21 Jul 1999 7:31 am using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB #2 SECOND SHOT
Misc Info :
Vial Number: 1





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Quality : 95
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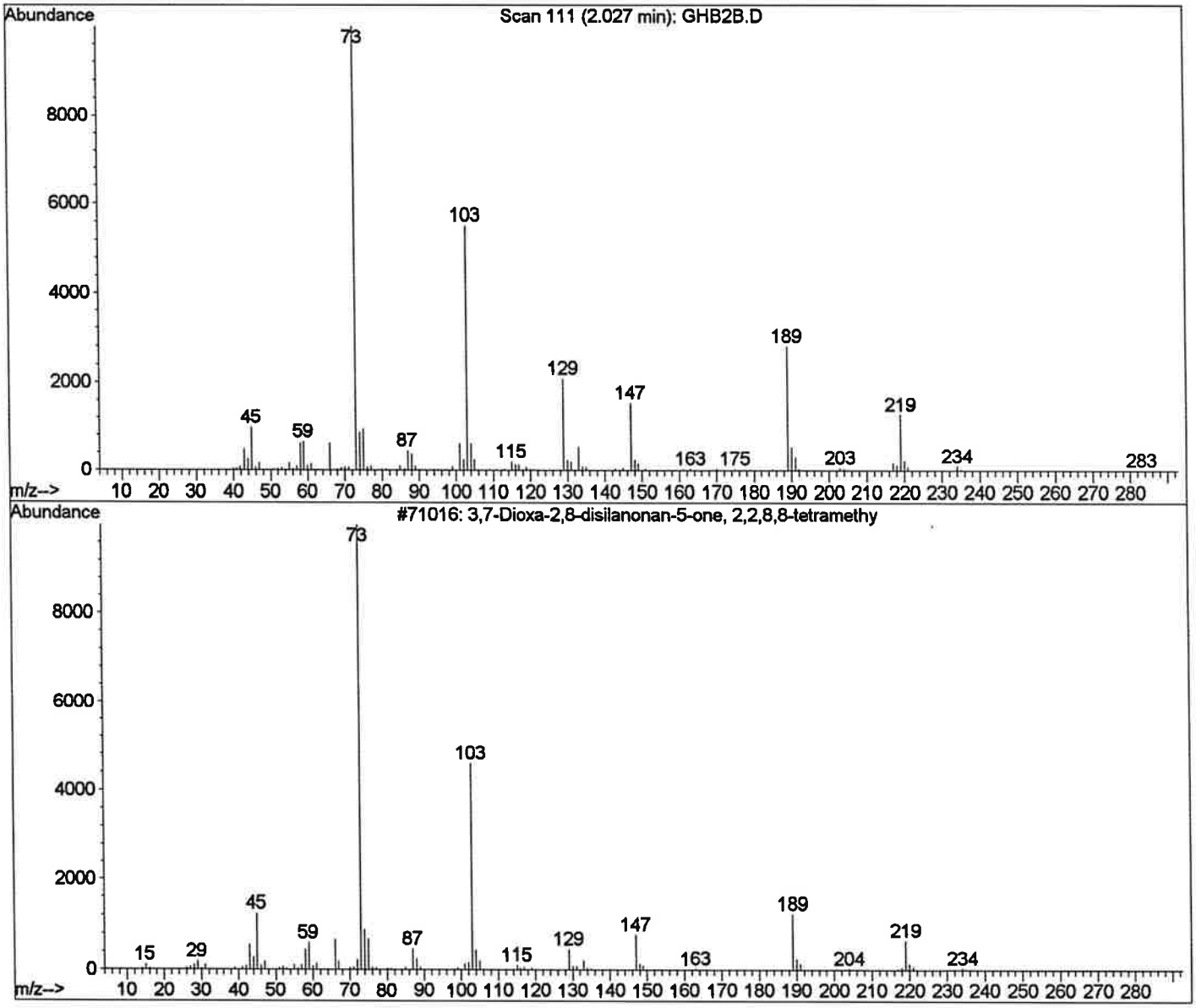
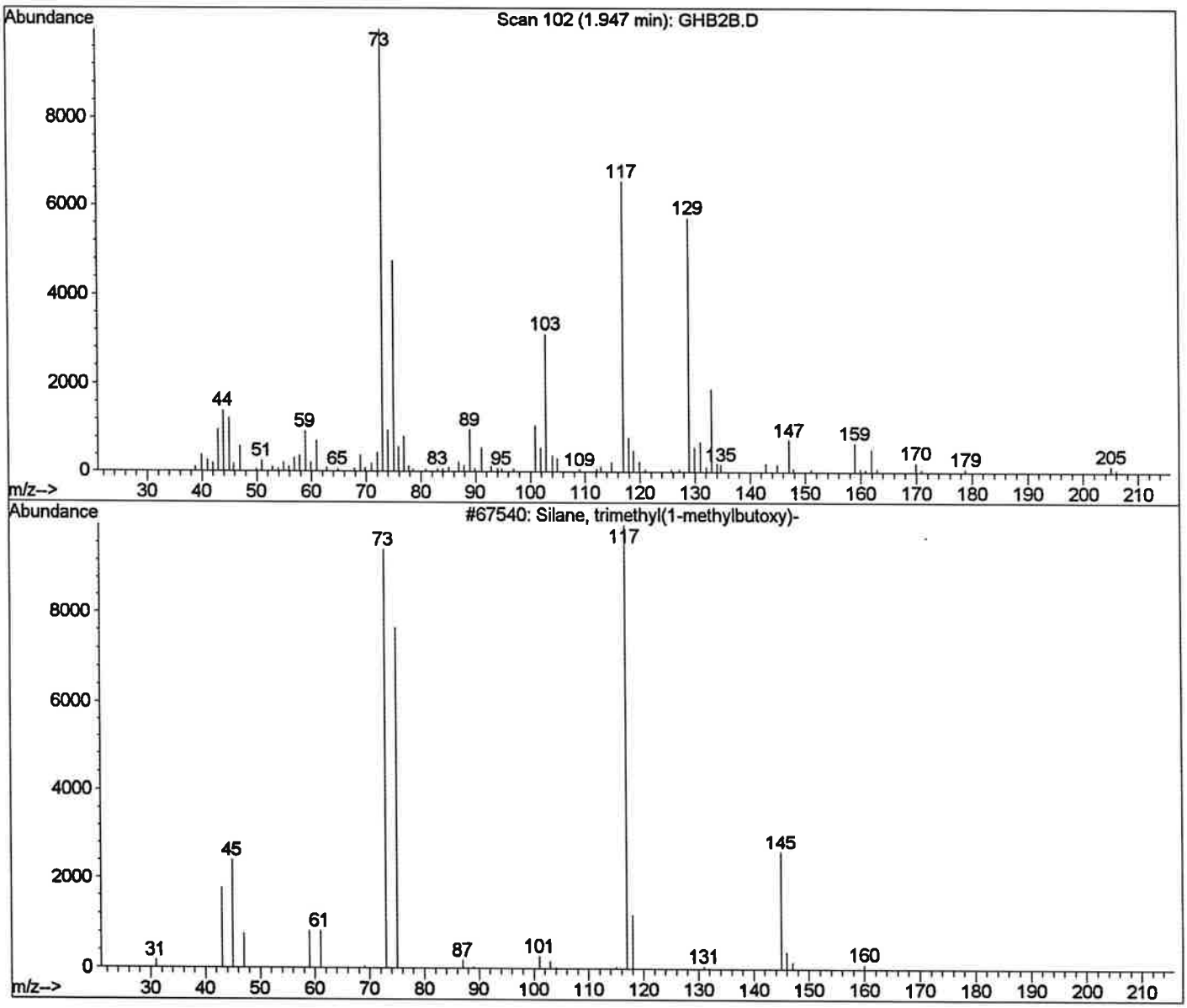


Library Searched : C:\DATABASE\NBS75K.L
Quality : 43
ID : Silane, trimethyl(1-methylbutoxy)-



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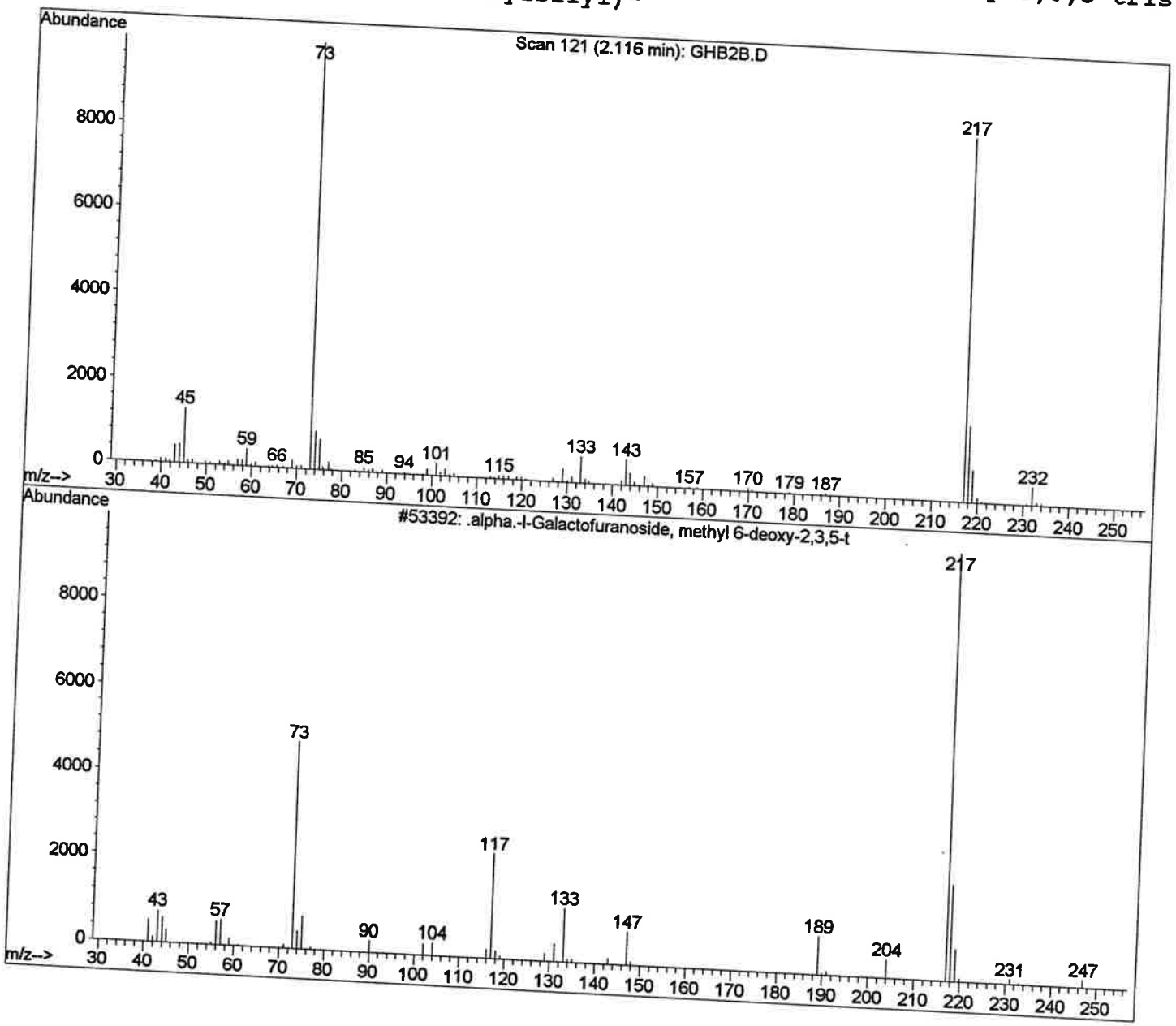
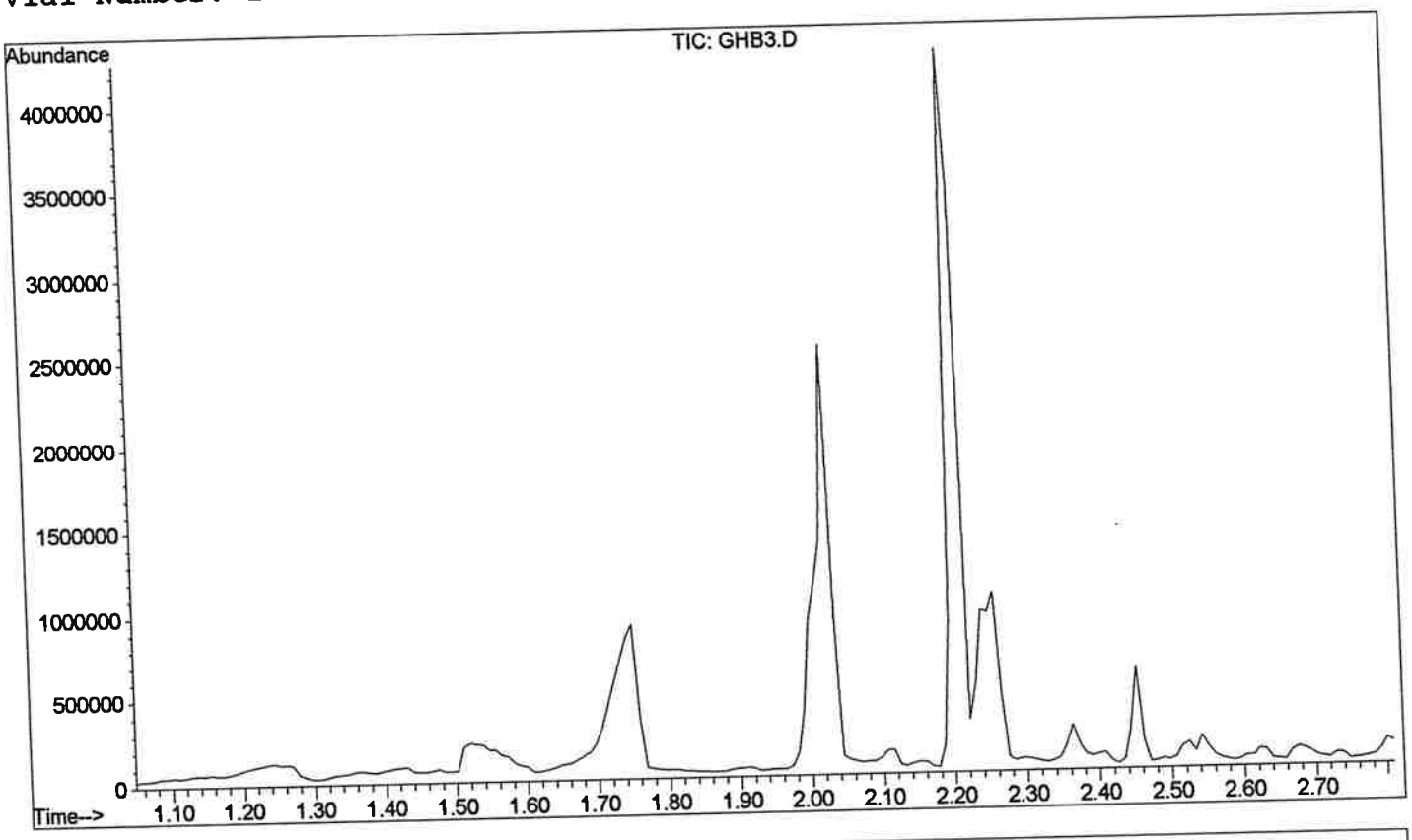
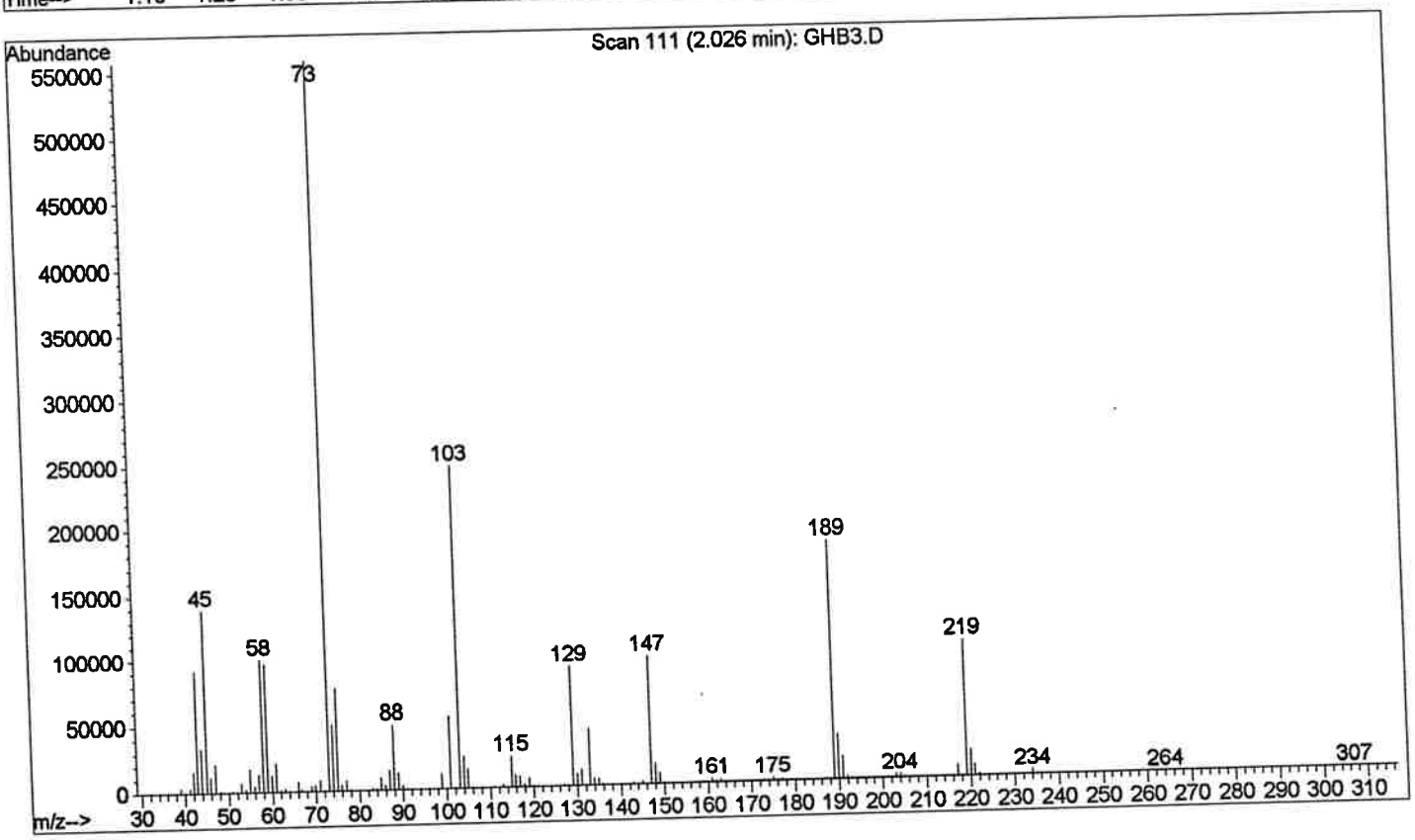
Quality : 64
ID : .alpha.-l-Galactofuranoside, methyl 6-deoxy-2,3,5-tris
-O-(trimethylsilyl)-



File : C:\HPCHEM\1\DATA\PWF\GHB3.D
Operator : PWF
Acquired : 20 Jul 1999 16:13 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB #3
Misc Info :
Vial Number: 1





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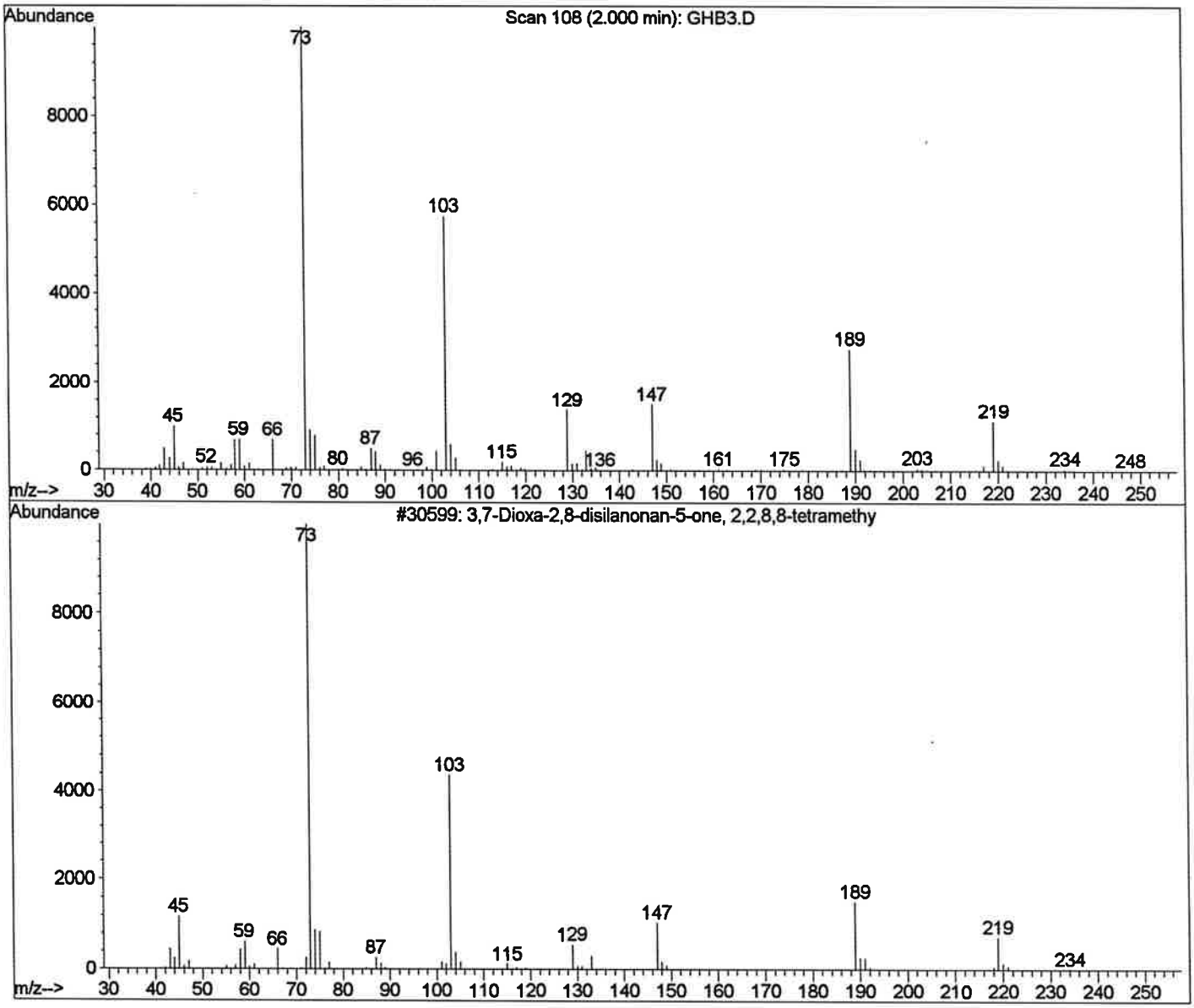

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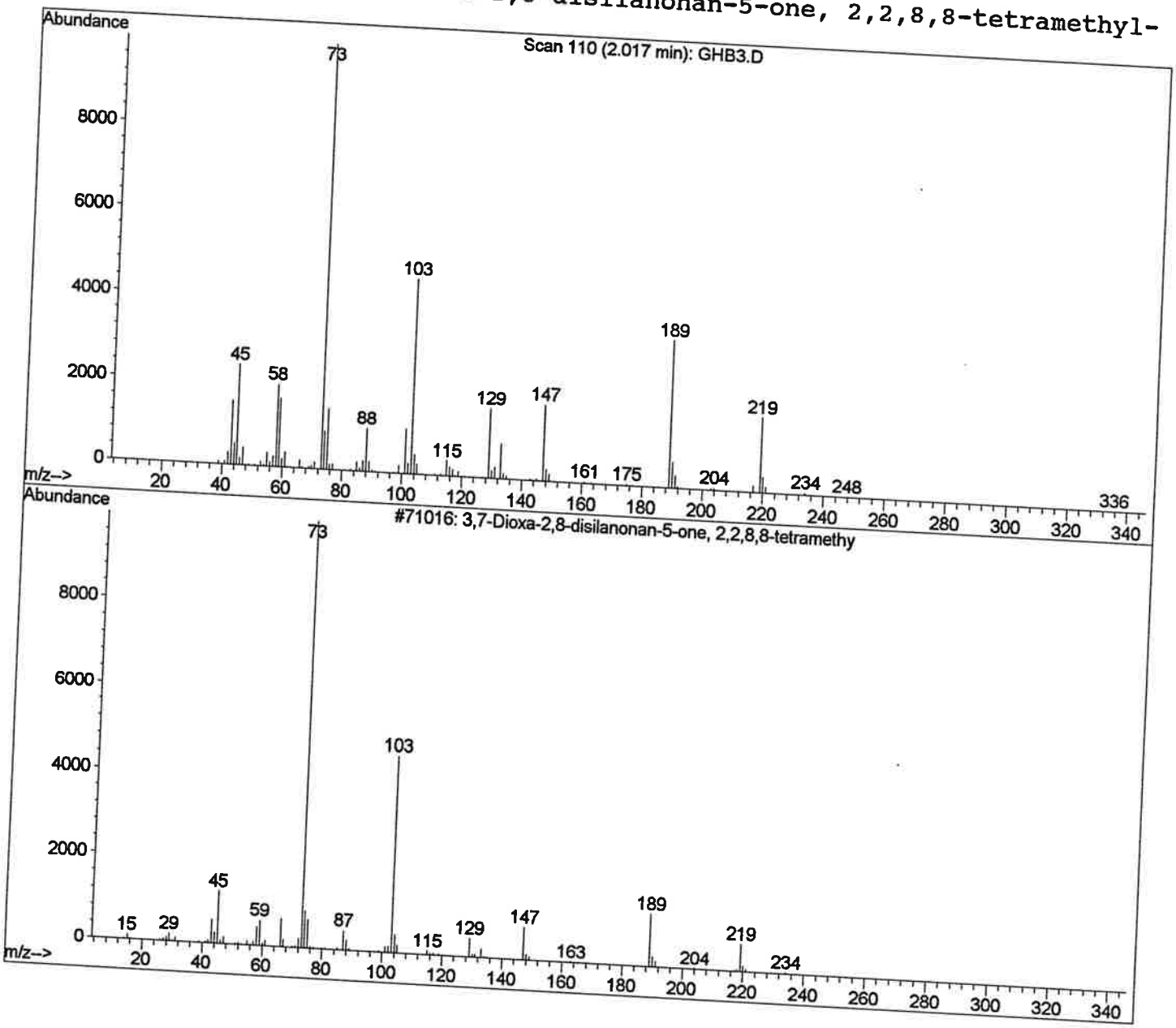

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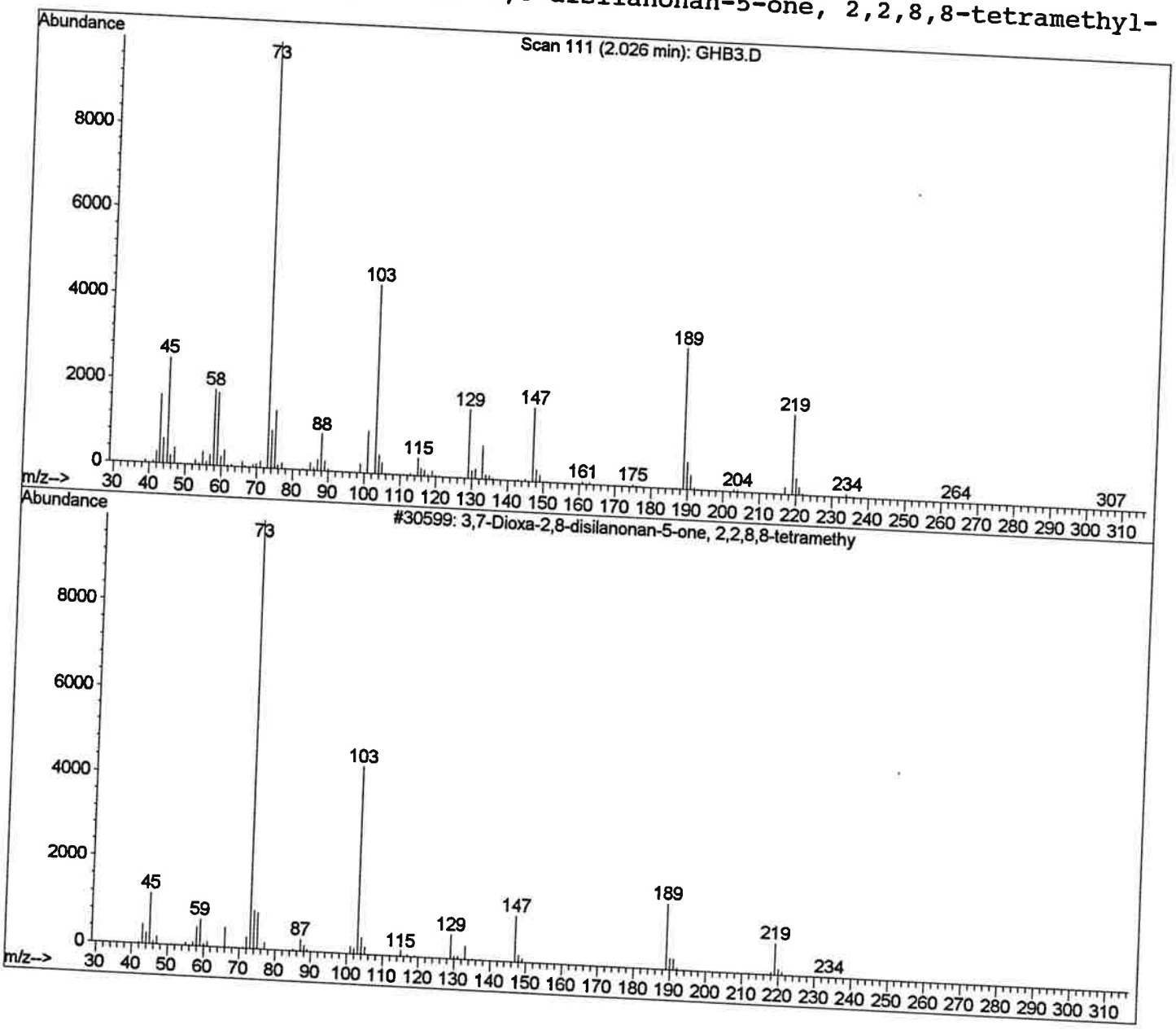
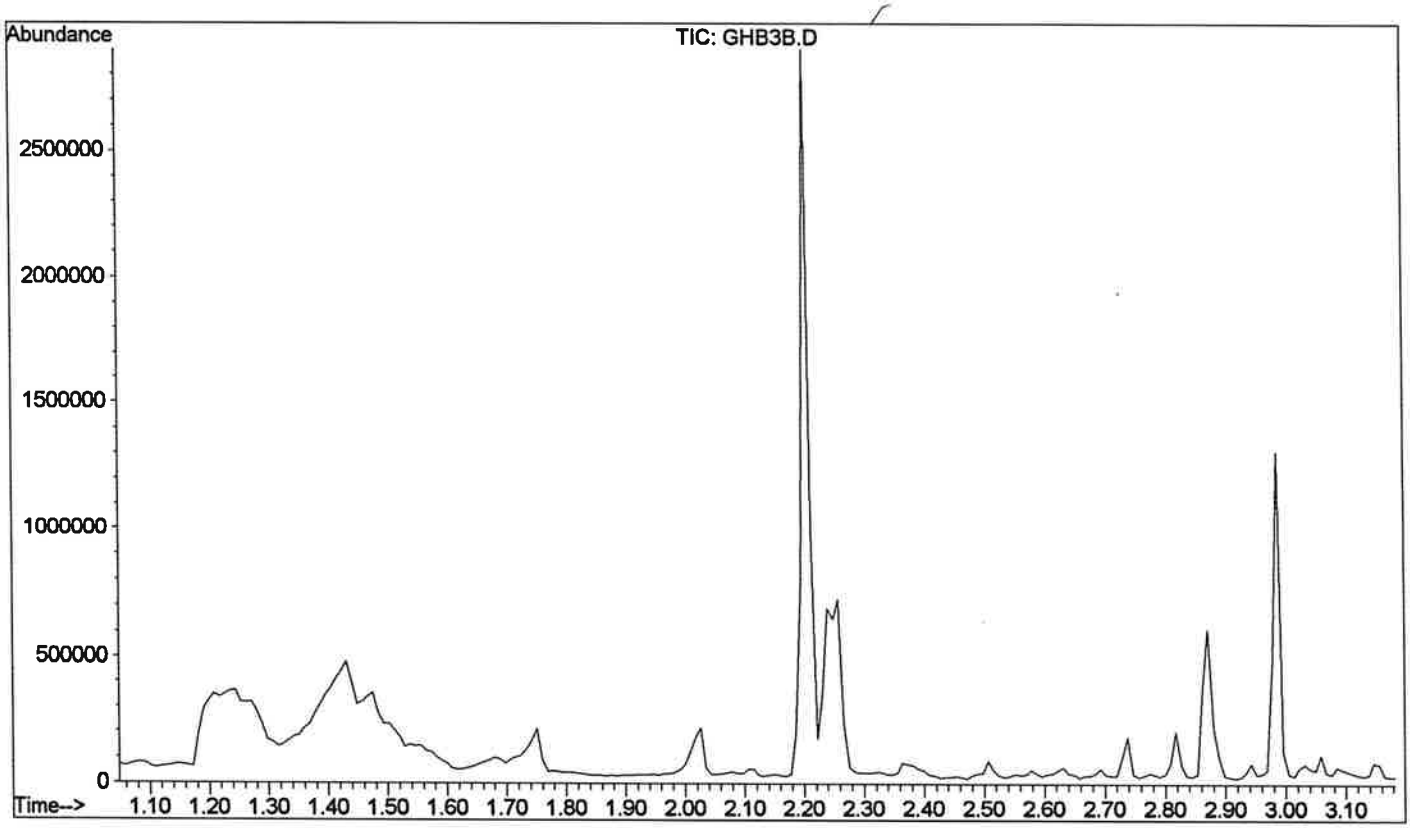
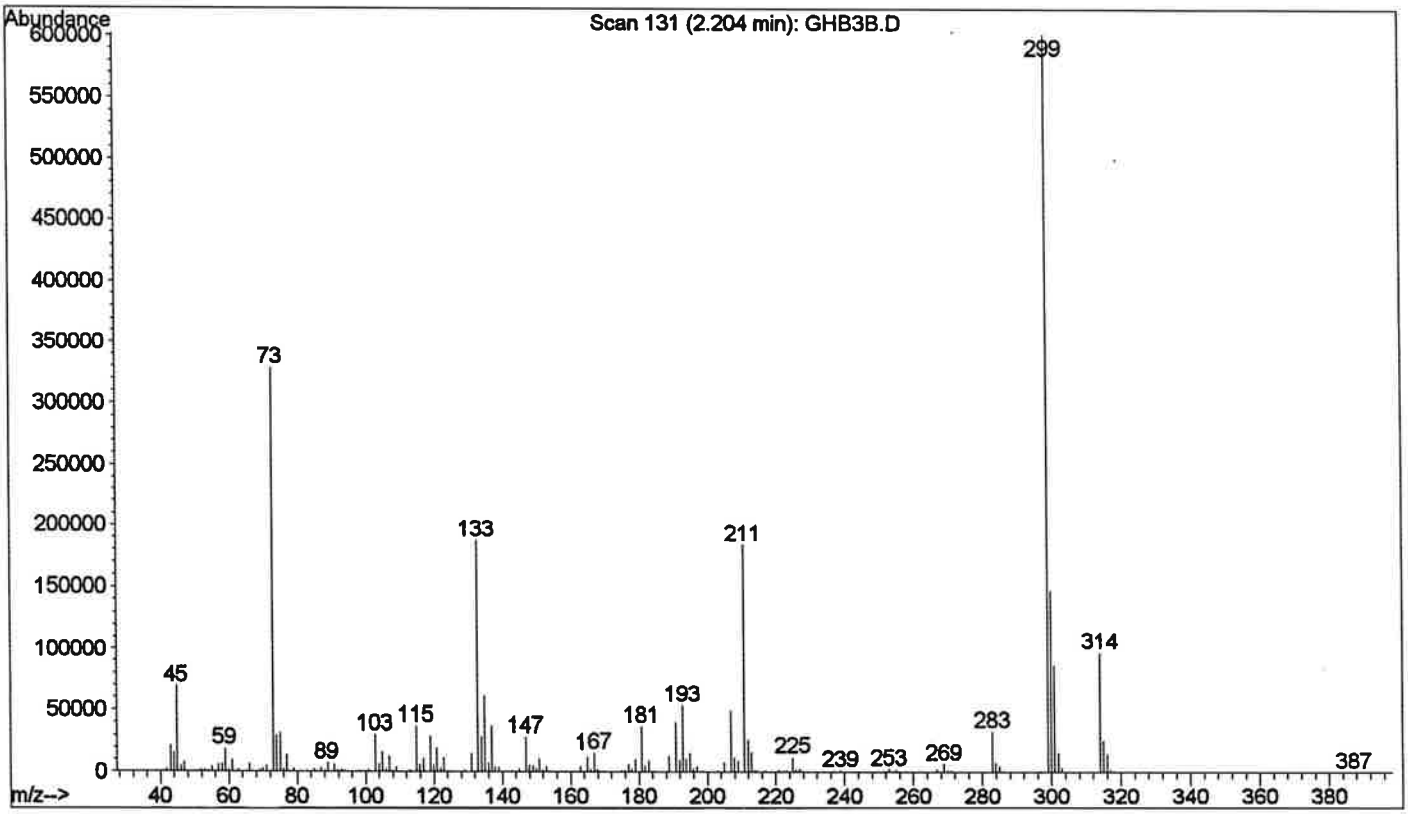


File : C:\HPCHEM\1\DATA\PWF\GHB3B.D
Operator : PWF
Acquired : 21 Jul 1999 7:43 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB 3 SECOND SHOT
Misc Info :
Vial Number: 1





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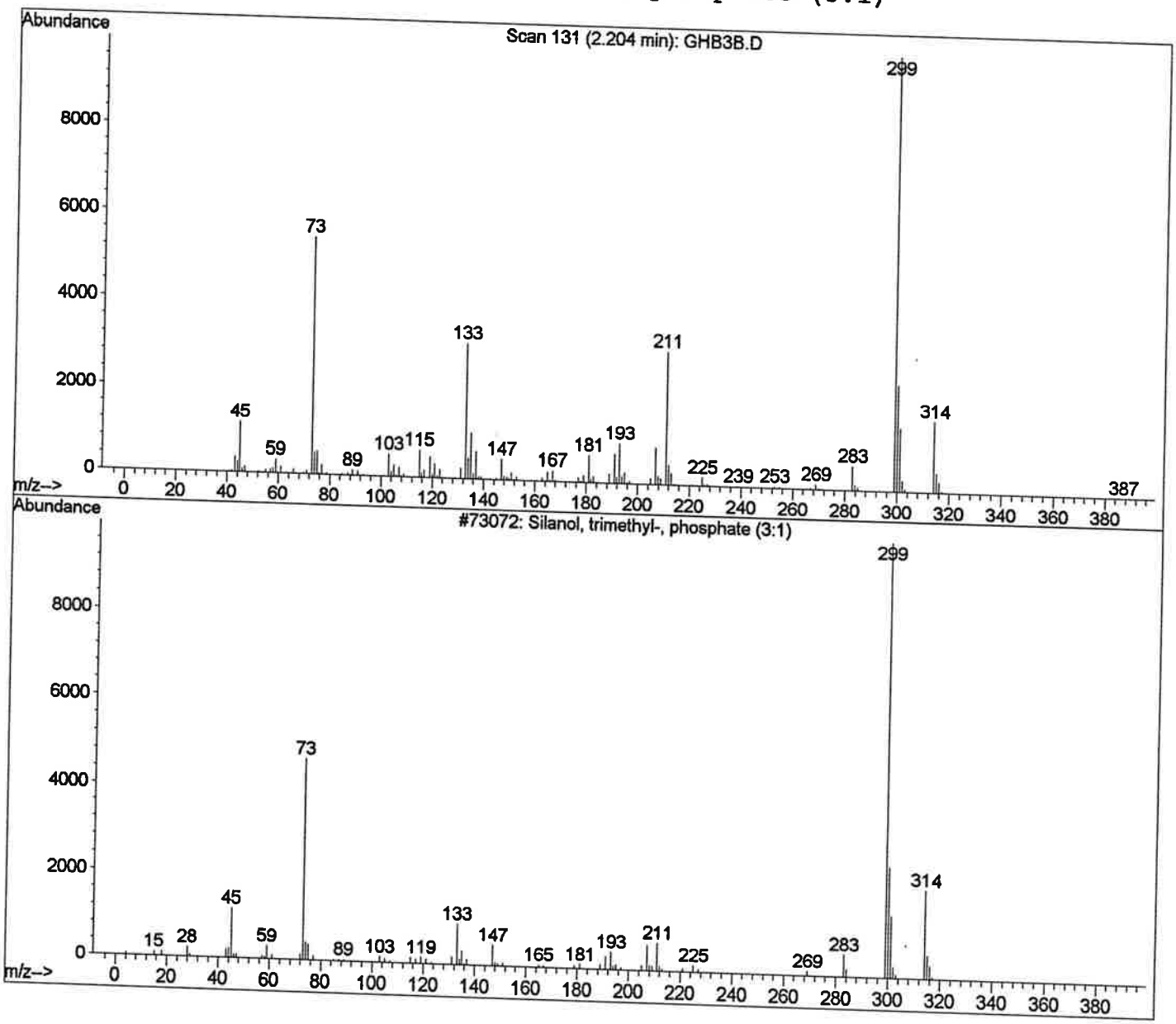
Quality : 97
ID : Silanol, trimethyl-, phosphate (3:1)



Library Searched : C:\DATABASE\NBS75K.L
Quality : 72
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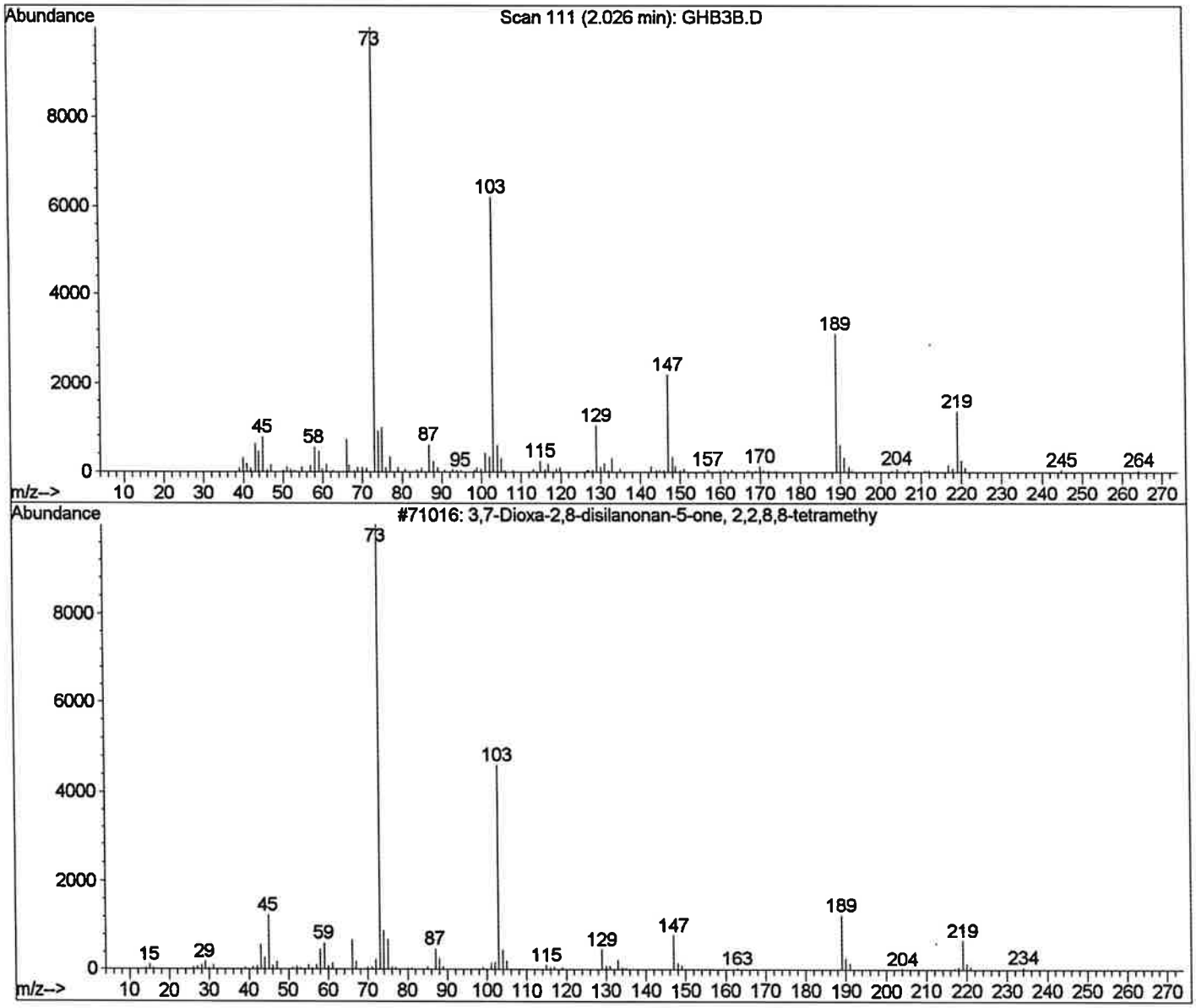

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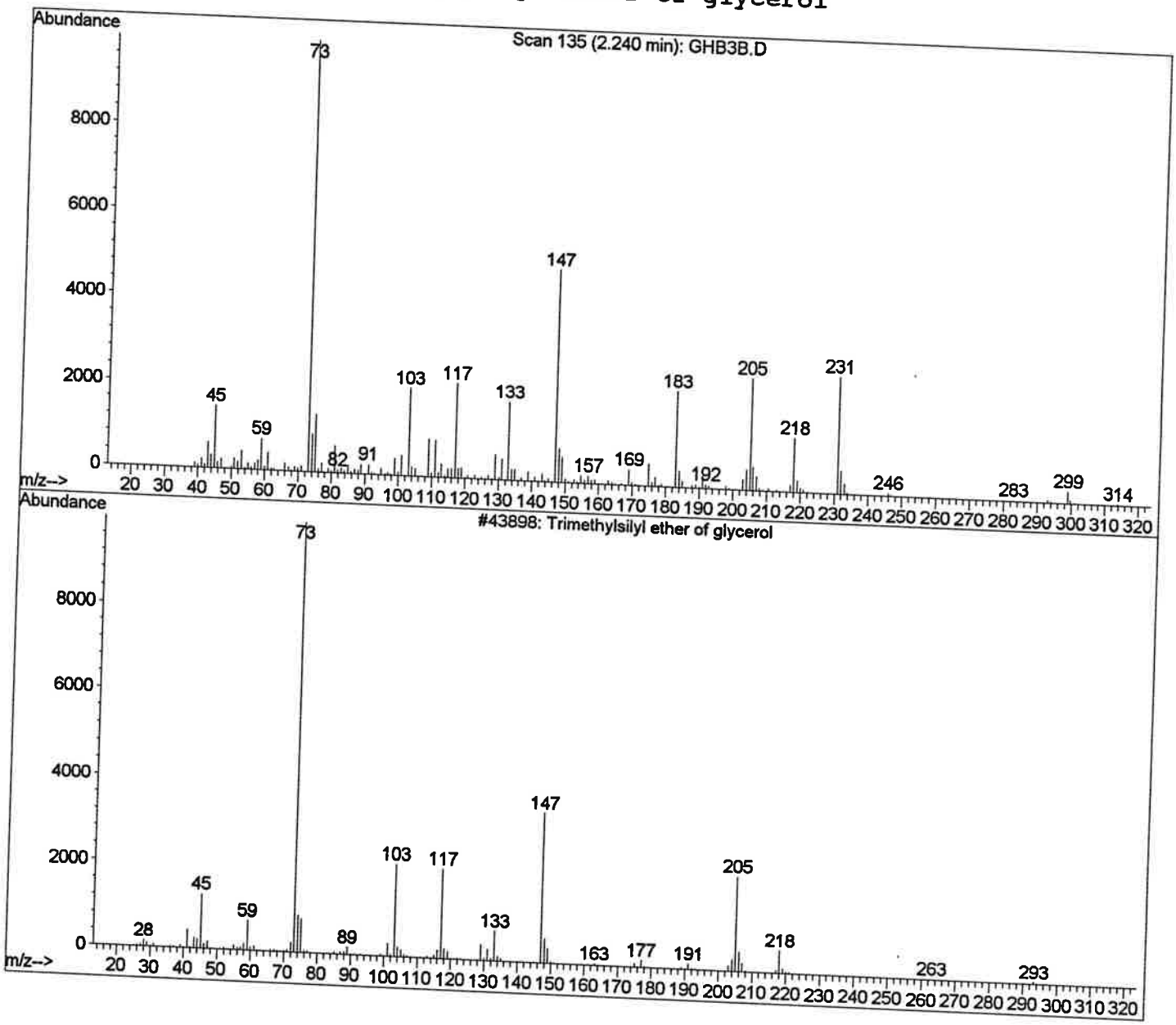
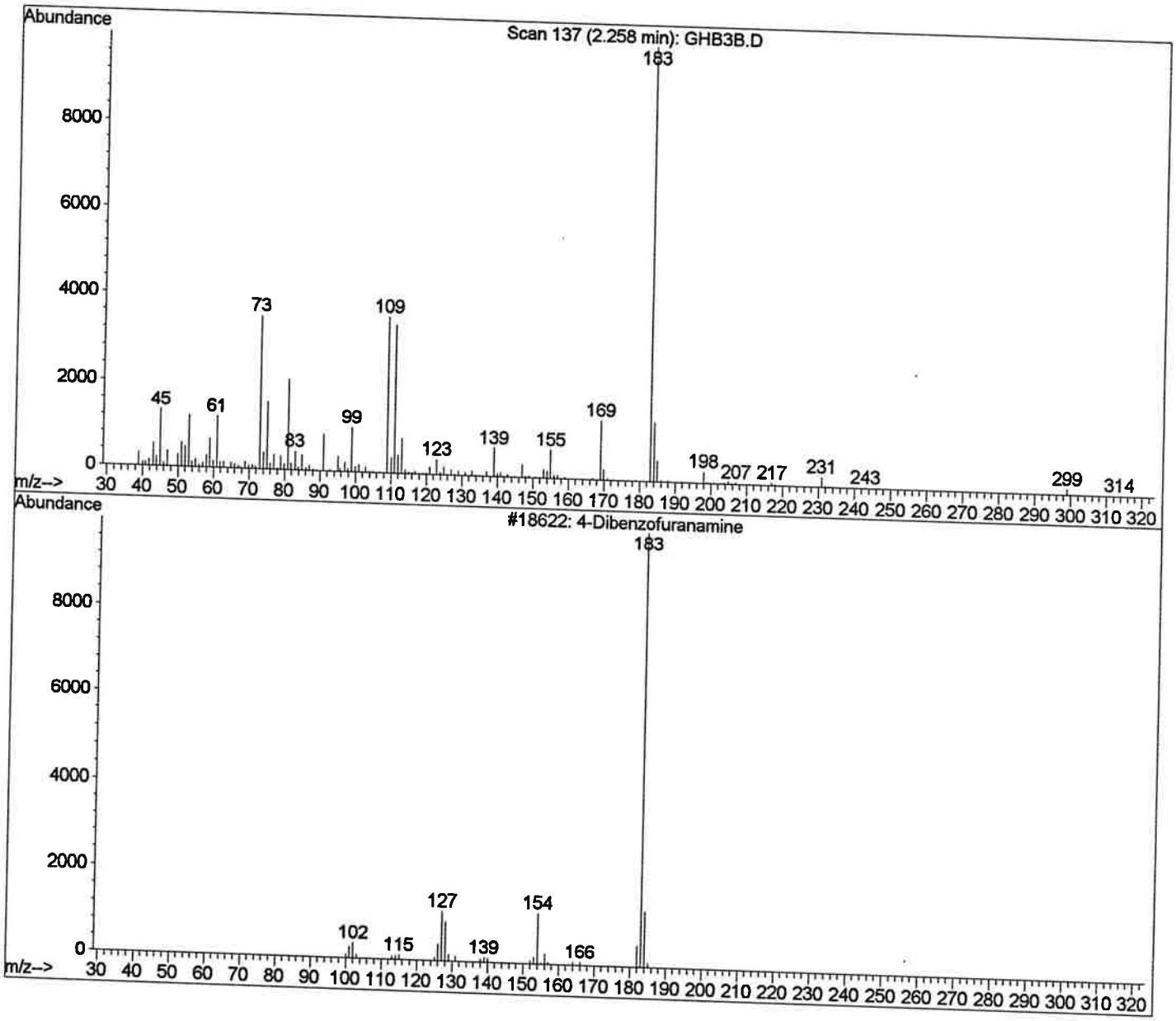
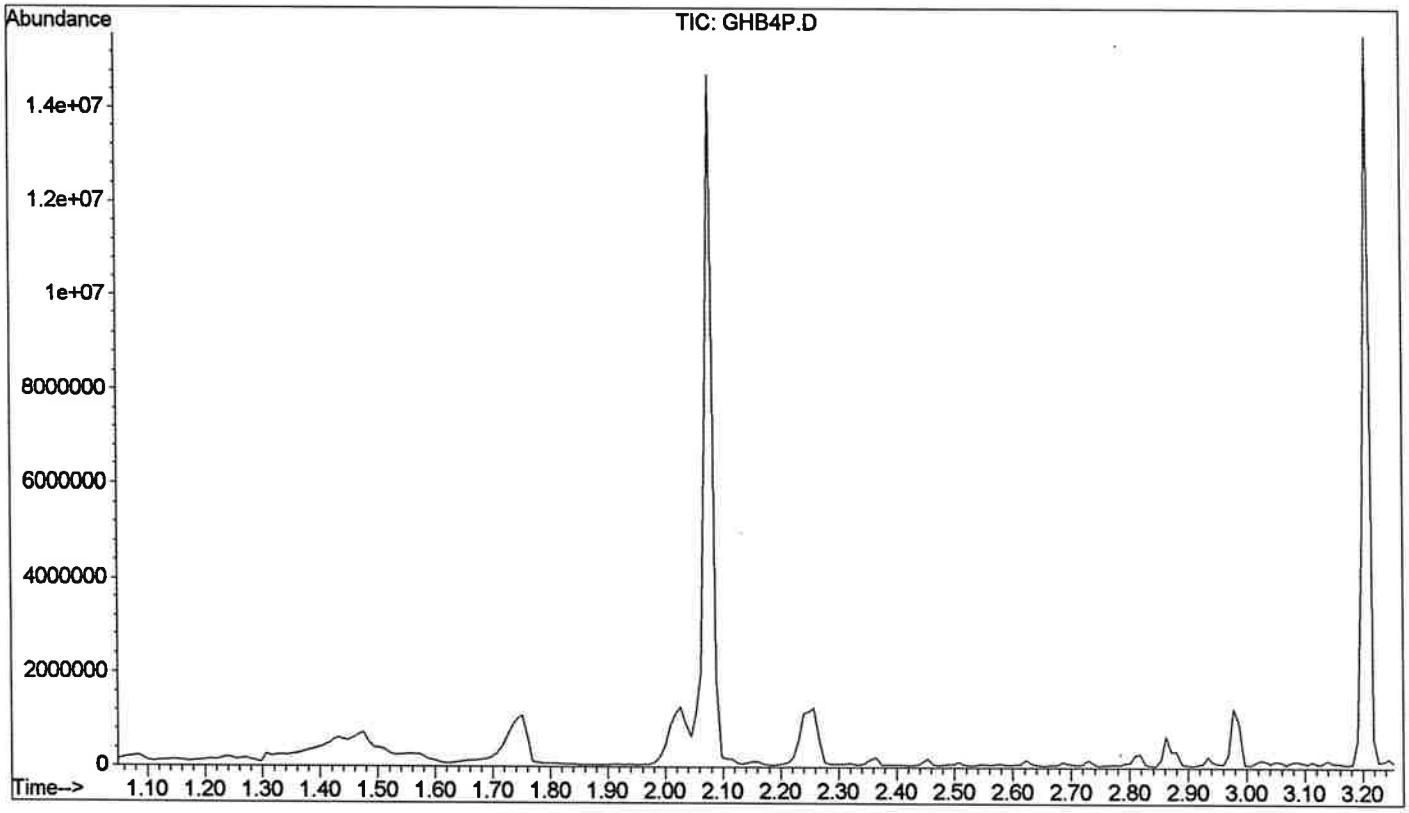
ID : Trimethylsilyl ether of glycerol

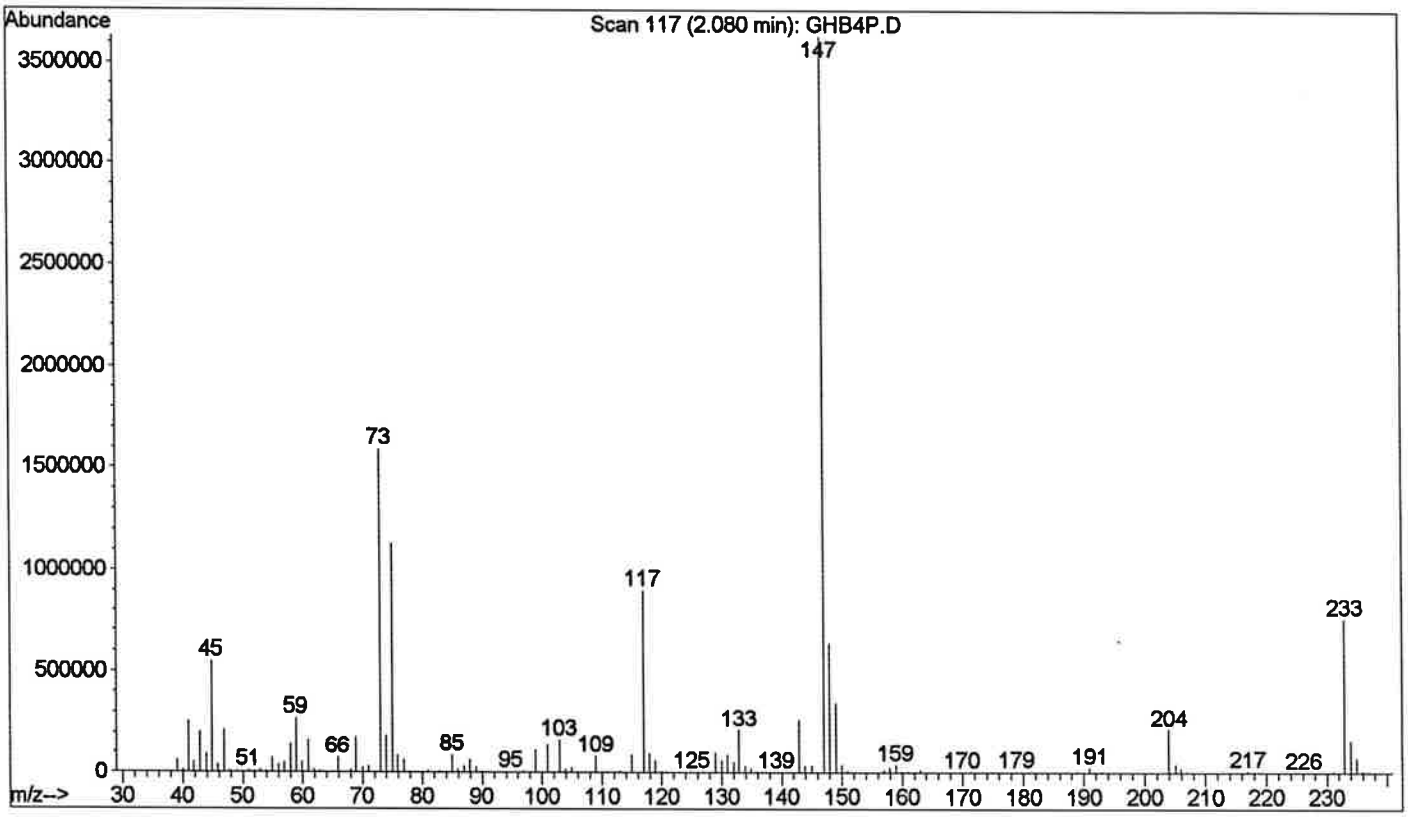


Library Searched : C:\DATABASE\NBS75K.L
Quality : 38
ID : 4-Dibenzofuranamine



File : C:\HPCHEM\1\DATA\PWF\GHB4P.D
Operator : PWF
Acquired : 20 Jul 1999 16:26 using AcqMethod MSDRUGS
Instrument : Grumpy#1
Sample Name: GHB # 4
Misc Info :
Vial Number: 1





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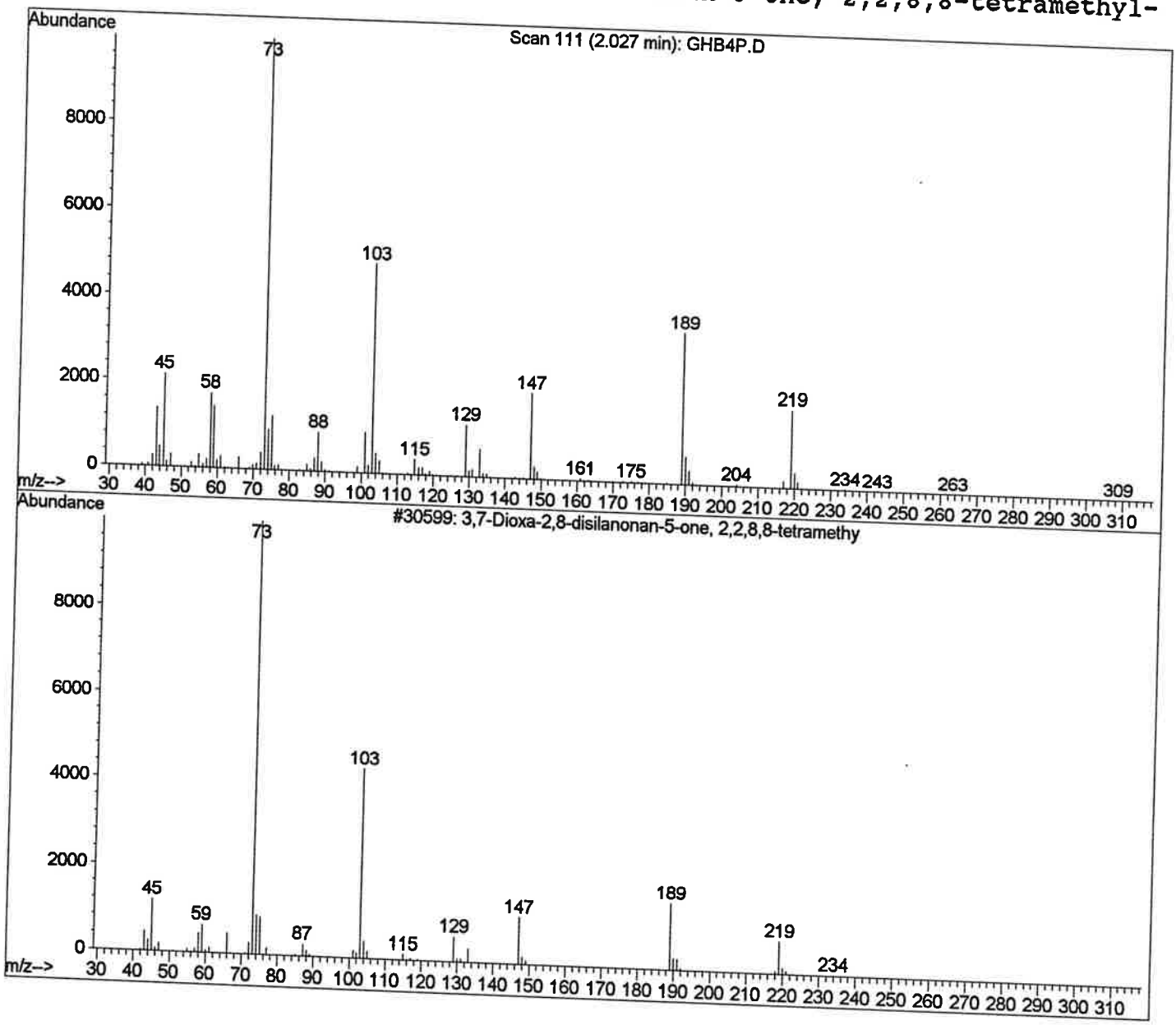
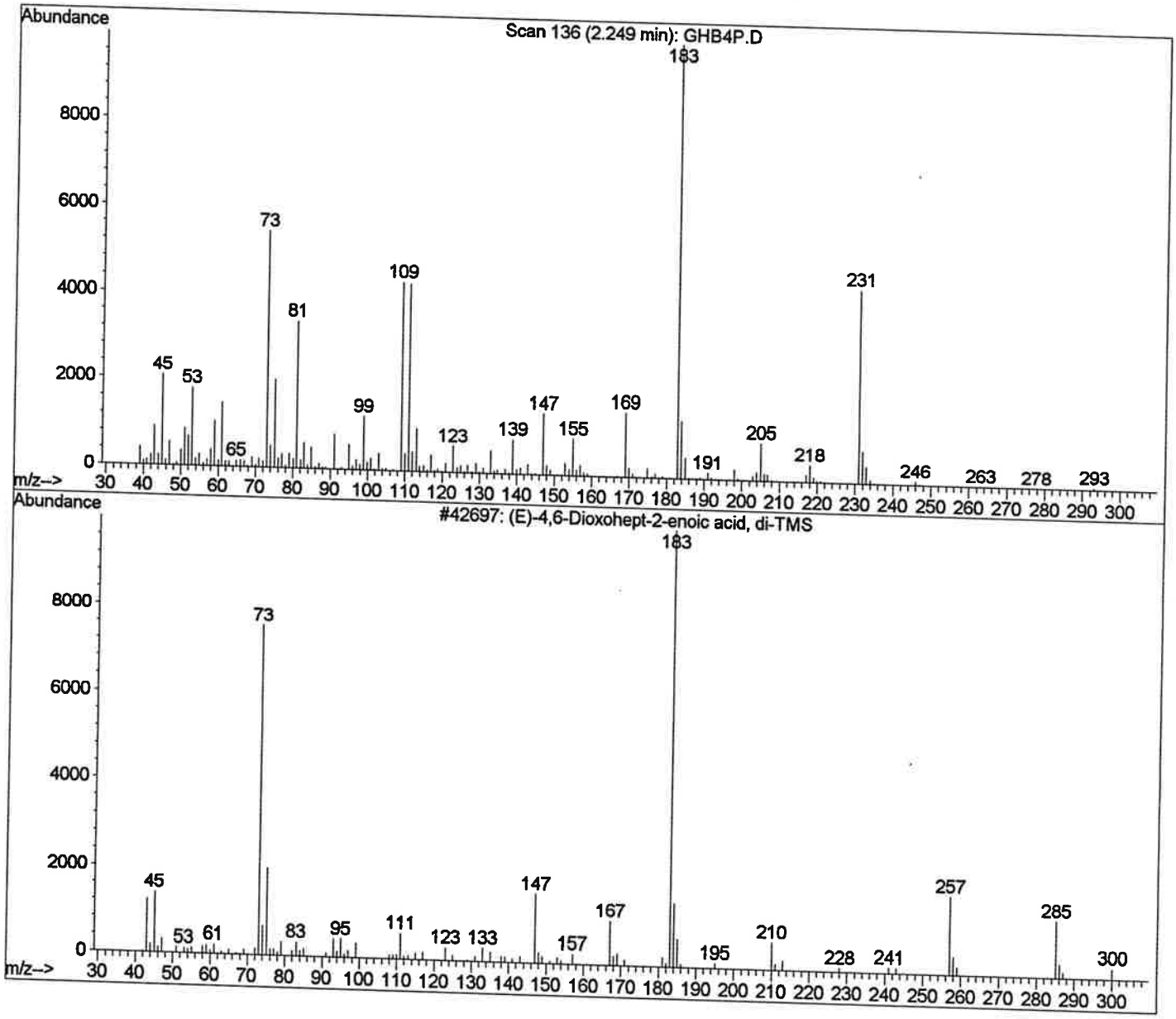


Library Searched : C:\DATABASE\NBS75K.L
Quality : 27
ID : (E)-4,6-Dioxohept-2-enoic acid, di-TMS



Method: Identification of GHB in an Aqueous Liquid by Silylation

1. Sample required

   Aqueous Liquid

2. Scientific instruments

   A. Gas chromatograph/mass spectrometer (GC/MS)

3. Reagents required

   A. Acetone
   B. Acetonitrile
   C. Bis-(Trimethylsilyl) trifluoracetamide (BSTFA)
   D. Trimethylchlorosilane (TCMS)
   E. Chloroform ($CHCl_3$)

4. Procedure

   A. Extract 1-5 ml of your unknown sample three times with excess $CHCl_3$. Extract once more with an equal amount of $CHCl_3$. This will remove any residual GBL from your sample. Analyze the $CHCl_3$ layer on a GC/MS to show that all the GBL has been removed. If GBL is still present, re-extract and analyze the $CHCl_3$ until no GBL is present.

   B. If possible, dry down the extracted sample in a small (10 ml) beaker. This can be accomplished by using a hair dryer; however, do not exceed 150C or the GHB may convert to GBL.
   NOTE: Re-crystallize with acetone by adding 10 ml of acetone to the beaker containing the sample and heating it on a hot plate at about 70 C (b.p. of acetone is 57 C) with stirring until it boils. This should take no longer than five minutes. Immediately pour the hot solvent into another small beaker and dry off the acetone using a hair dryer on low heat.

   C. To the beaker containing the dry sample, add 2 ml of acetonitrile, 500 uL of BSTFA, and 800 uL of TCMS. Cover the beaker and heat the solution on a hot plate at 60 C-80 C for 10-15 minutes. Do not exceed 80 C, and do not let the solution boil.

   D. Analyze the product on a GC/MS. GHB-TMS should come off the column at about two minutes using MSDRUGS.

5. Notes

   A. When working with derivatizing agents such as BSTFA and TCMS, safety glasses, gloves , lab coat, and a fume hood are required.

B. Water inhibits the derivatizing agents; therefore, it is imperative that your sample is as dry as possible.

C. To identify GHB-TMS, ions 117, 133, 459, 204, and 233 must be present. Ions common to TCMS derivatives include 73, 147, and 243.

D. If the sample is nonaqueous perform a FTIR on the liquid by spotting the liquid on a KBr pellet or on a NaCl salt plate. The liquid could be GBL. It can be identified by comparing the IR to a standard spectra of GBL.

6. References

A. Andrews, Kathleen A., DEA Western Regional Laboratory, Personal Communication.

B. Andrews, Kathleen A. Presentation Notes: The Headaches of Analyzing a GHB Sample.

C. Blackledge, Robert D. and Miller, Michael D., Naval Investigative Service Regional Forensic Laboratory. "The Identification of GHB," Microgram, Vol. XXIV, No. 7, July1991.

D. Newman, Reta, Pinellas County Forensic Laboratory, Personal Communication.

E. Newman, Reta. Procedure and Method Validation for the Analysis of Suspected GHB Cases in Aqueous Solutions.

F. Walker, Lara, California Department of Justice. "Identification of the Potassium Salt of Gamma-Hydroxybutyrate (GHB-K^+)," Journal of the Clandestine Laboratory Investigating Chemists, Vol. 9, p. 17, January 1999.

9/28/99 PWD

GBL 1- Standard GBL(2ml) evaporated to dryness, recrystallized with Acetone, derivatized using GHB silyation procedure

* No GHB-TMS present via GC/MS.

GBL 2- Standard GBL (5 drops) derivatized using GHB Silyation Procedure.

* No GHB-TMS present via GC/MS

GBL 3- Standard GBL (1ml) plus 2ml H_2O washed 4 times with $CHCl_3$ (GBL still present via GC/MS).

GHB4- Standard GBL(1ml) plus 2ml H_2O washed 7 times with $CHCl_3$ (No GBL present on GC/MS).

GBL D- Standard GBL(1ml) plus 2ml H_2O washed 7 times with $CHCl_3$ evaporated to dryness, recrystallized with Acetone, derivatized using GHB Silyation procedure

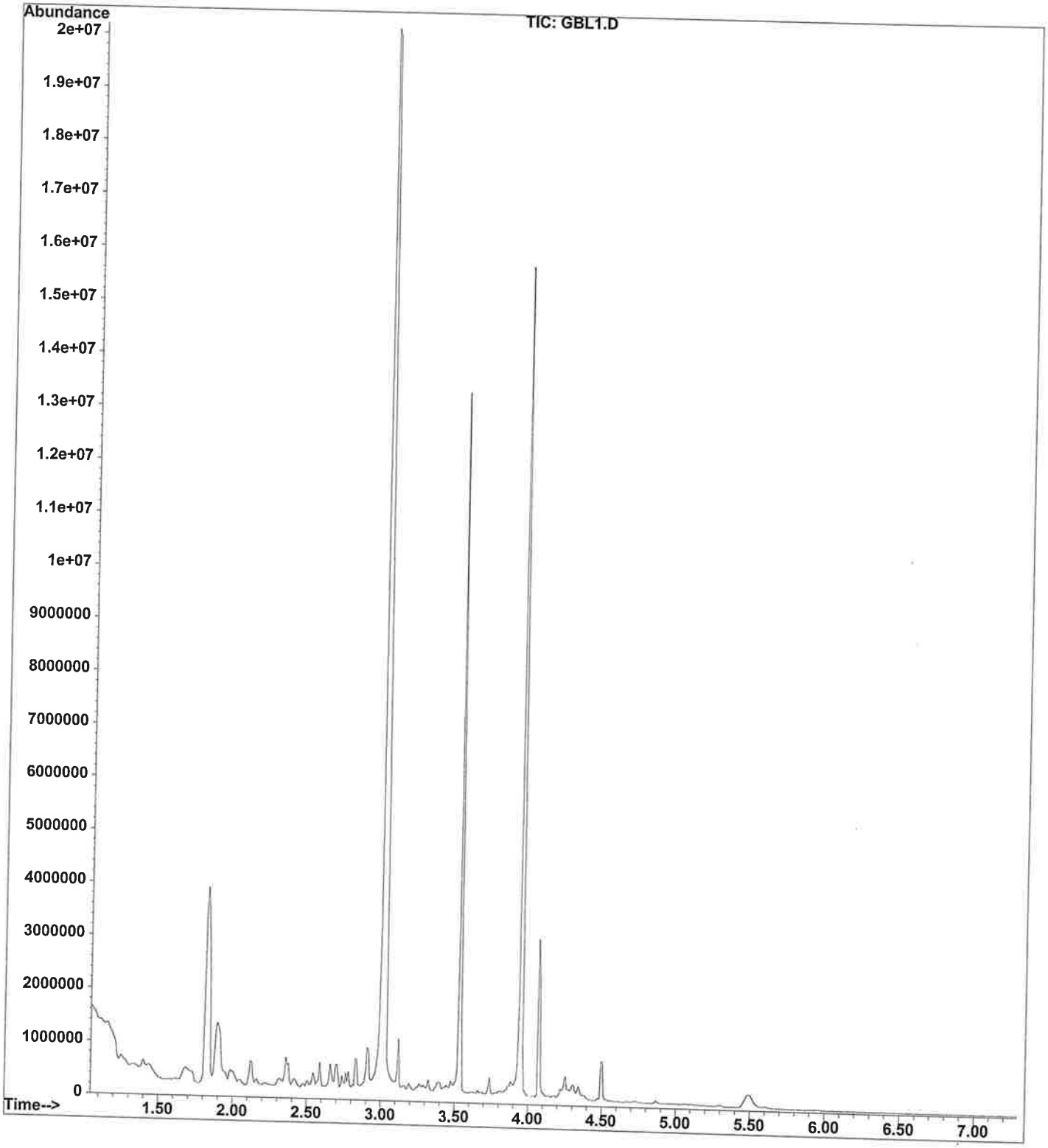
* No GHB-TMS present via GC/MS.

* GBL3, GHB4, GBLD are the same sample Extracted a total of 7 times with $CHCl_3$ & derivatized using GHB silyation Procedure.

GBL D4 Std GBL (5 drops) derivatized using GHB silyation procedure.

* No GHB-TMS present via GC/MS.

File : C:\HPCHEM\1\DATA\PWF\GBL1.D
Operator : PWF
Acquired : 27 Sep 1999 16:14 using AcqMethod MSDRUGS
Instrument : DONNA
Sample Name: STD GBL EVP ACETONE EXT DER
Misc Info :
Vial Number: 1



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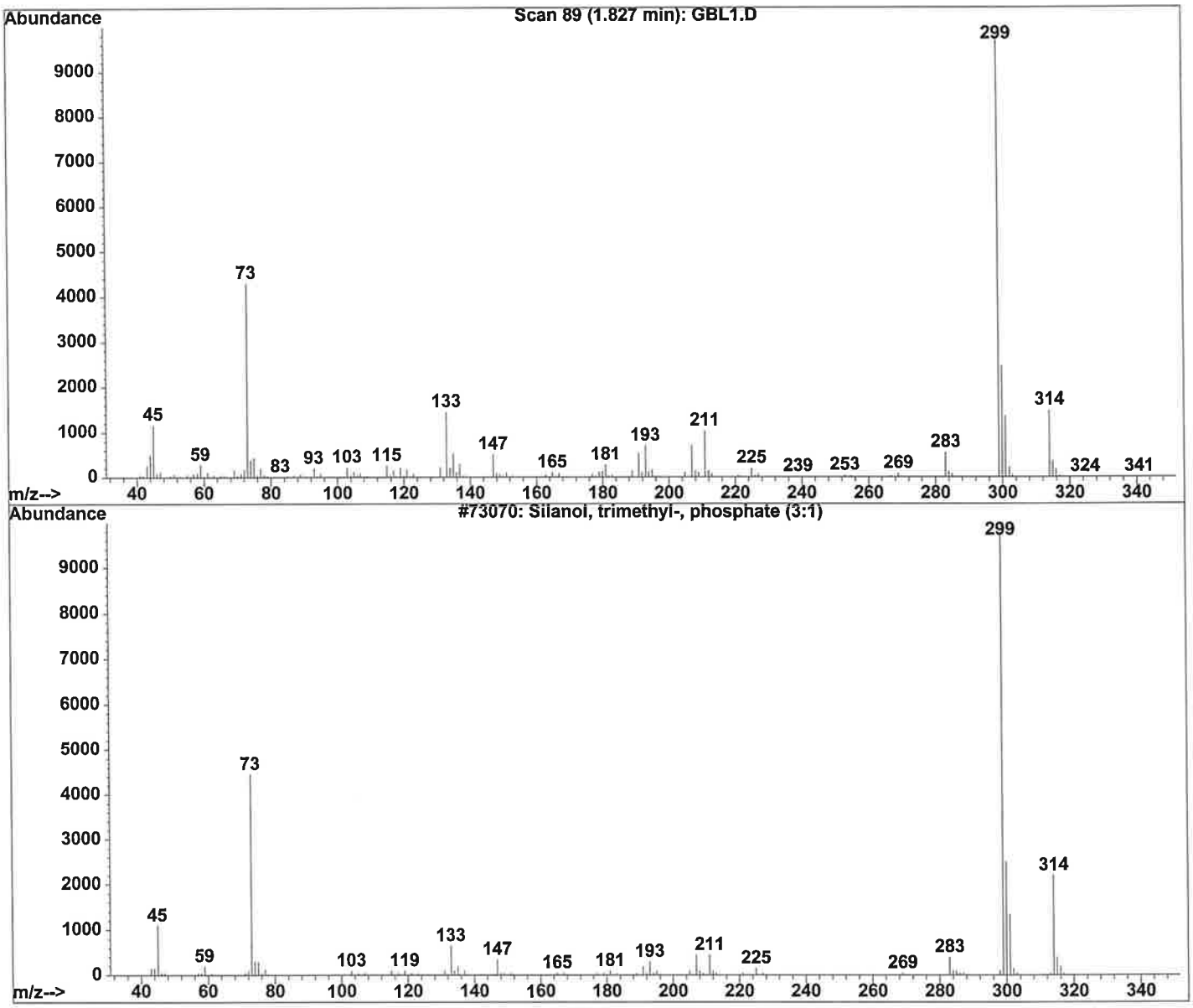
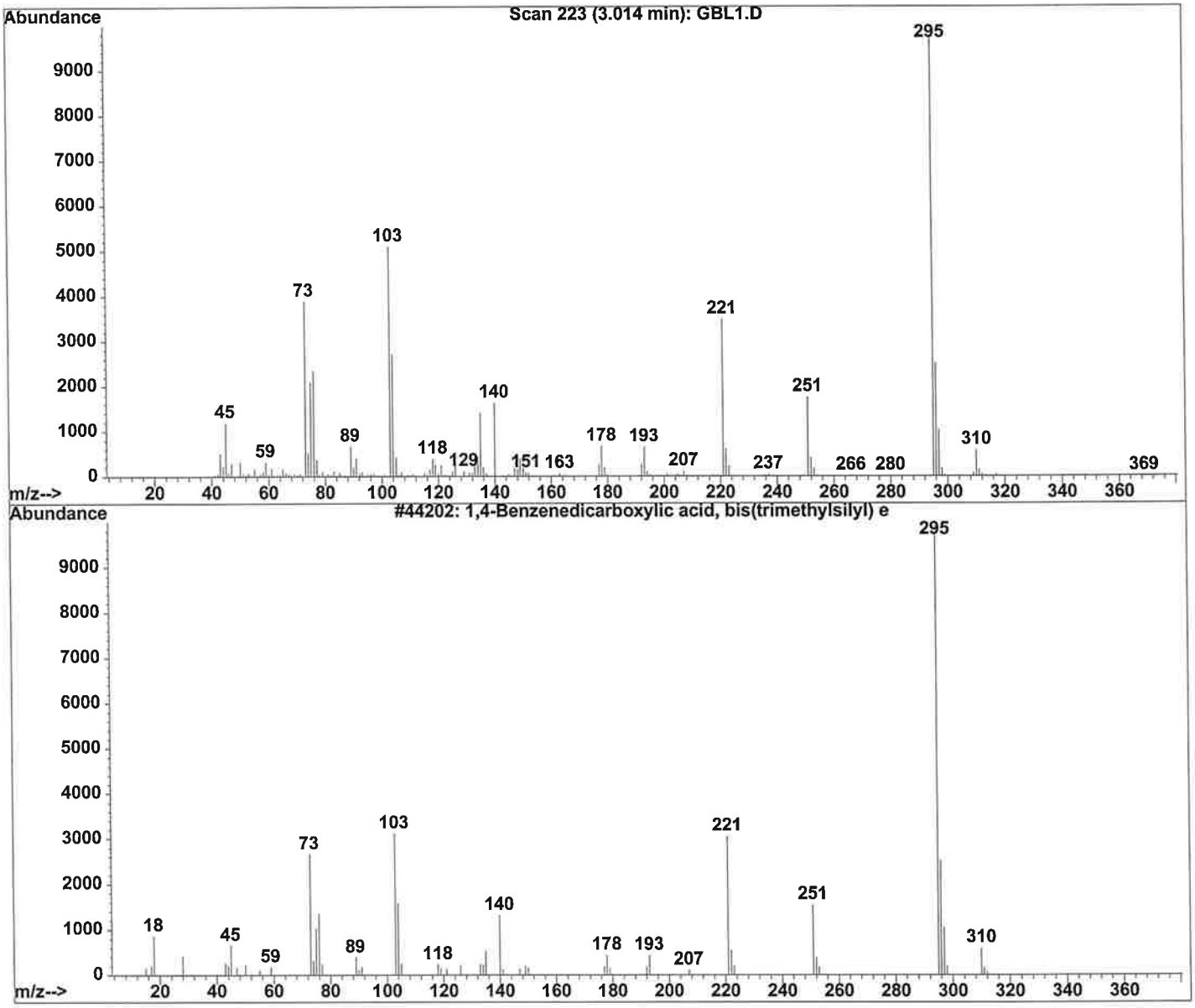
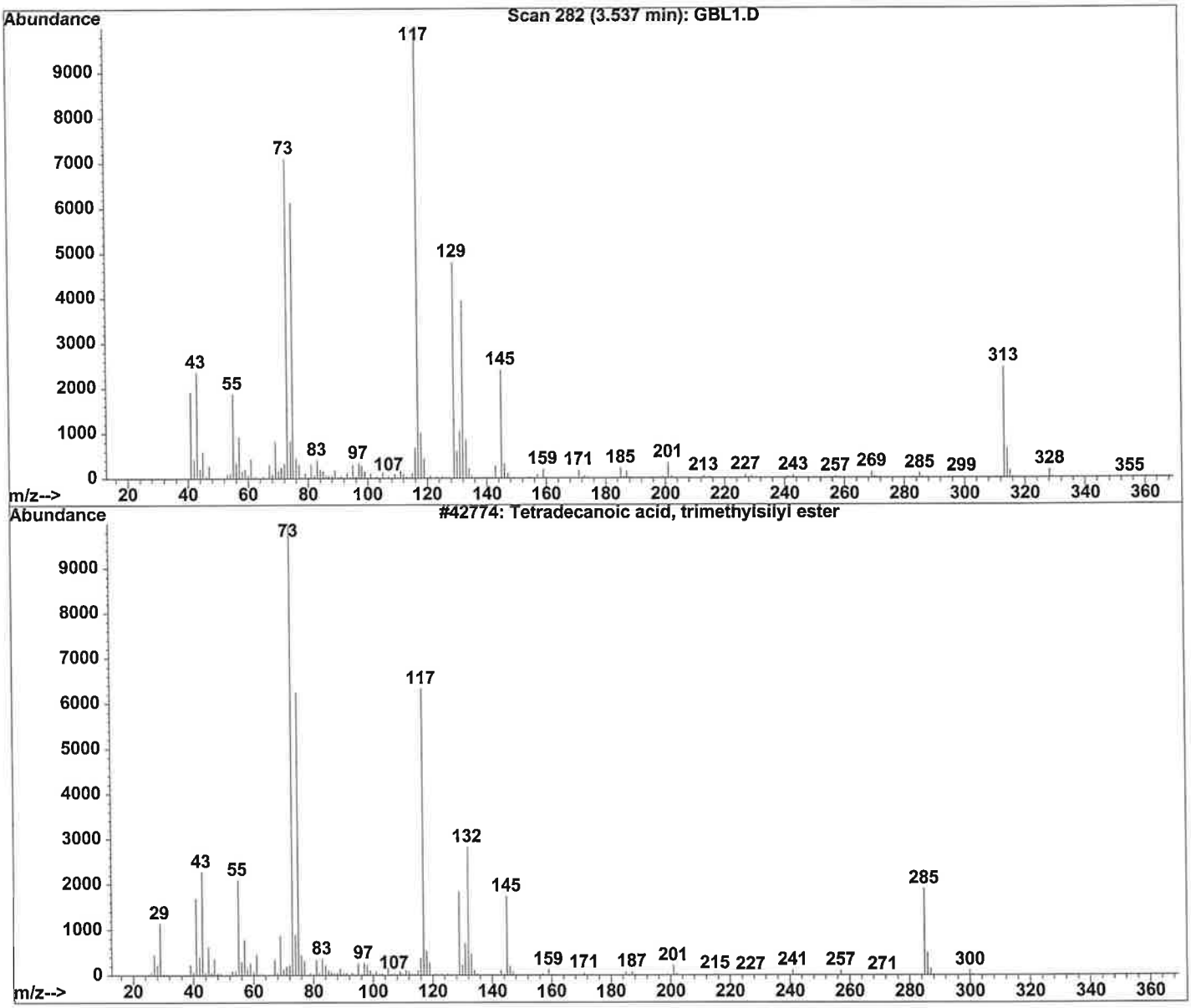
Quality : 97
ID : Silanol, trimethyl-, phosphate (3:1)



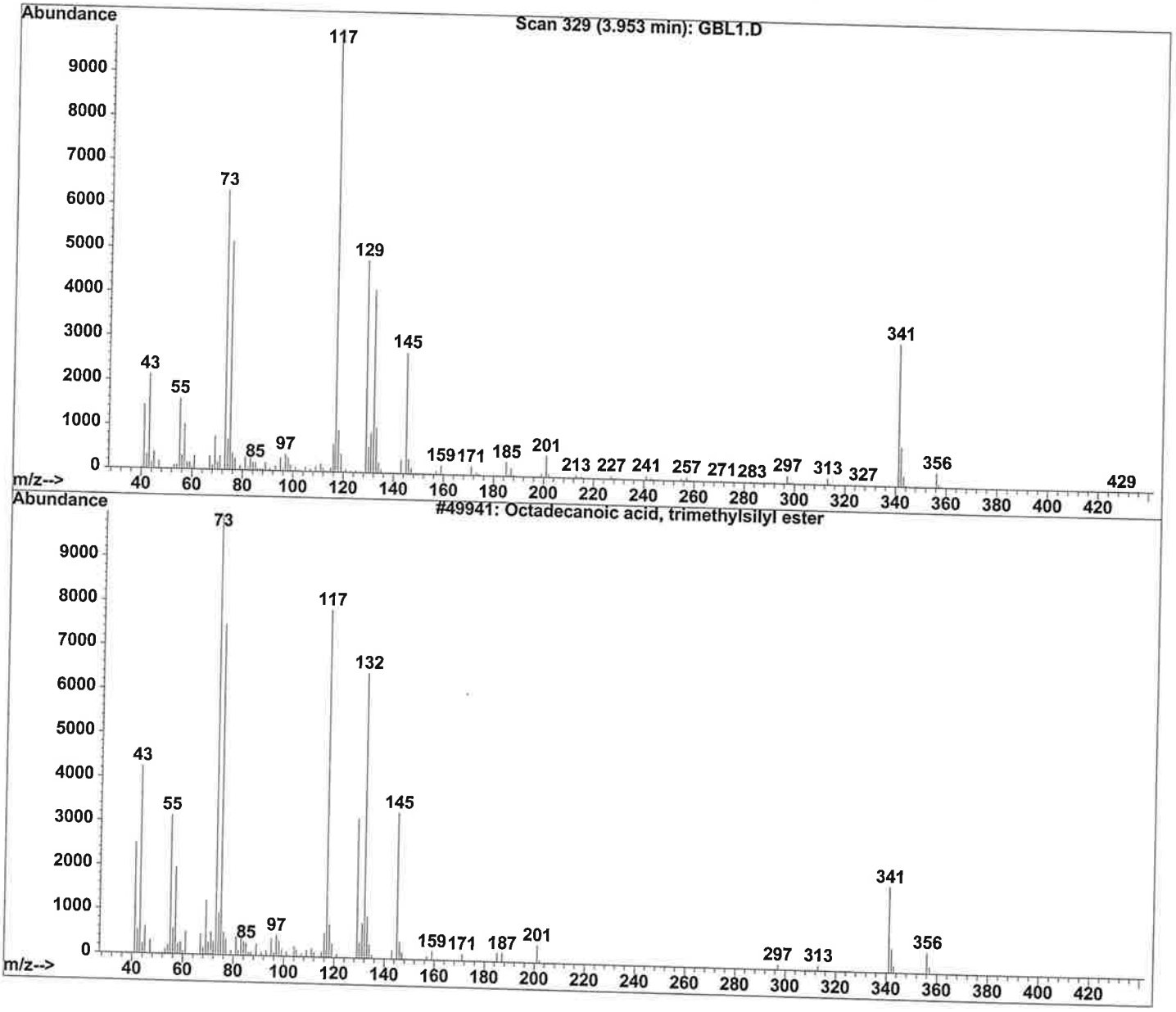
Library Searched : C:\DATABASE\NBS75K.L
Quality : 99
ID : 1,4-Benzenedicarboxylic acid, bis(trimethylsilyl) este
r



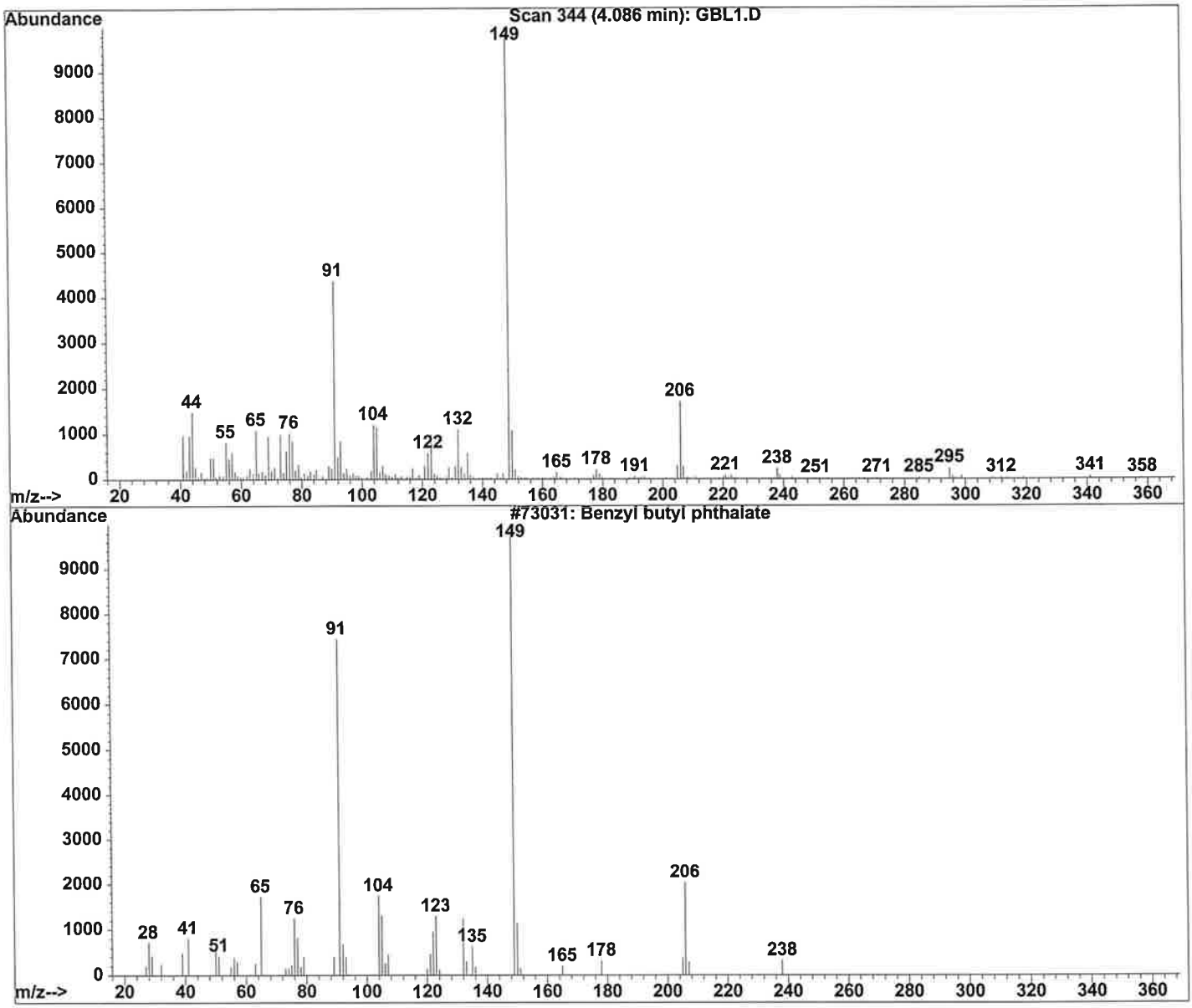
Library Searched : C:\DATABASE\NBS75K.L
Quality : 64
ID : Tetradecanoic acid, trimethylsilyl ester



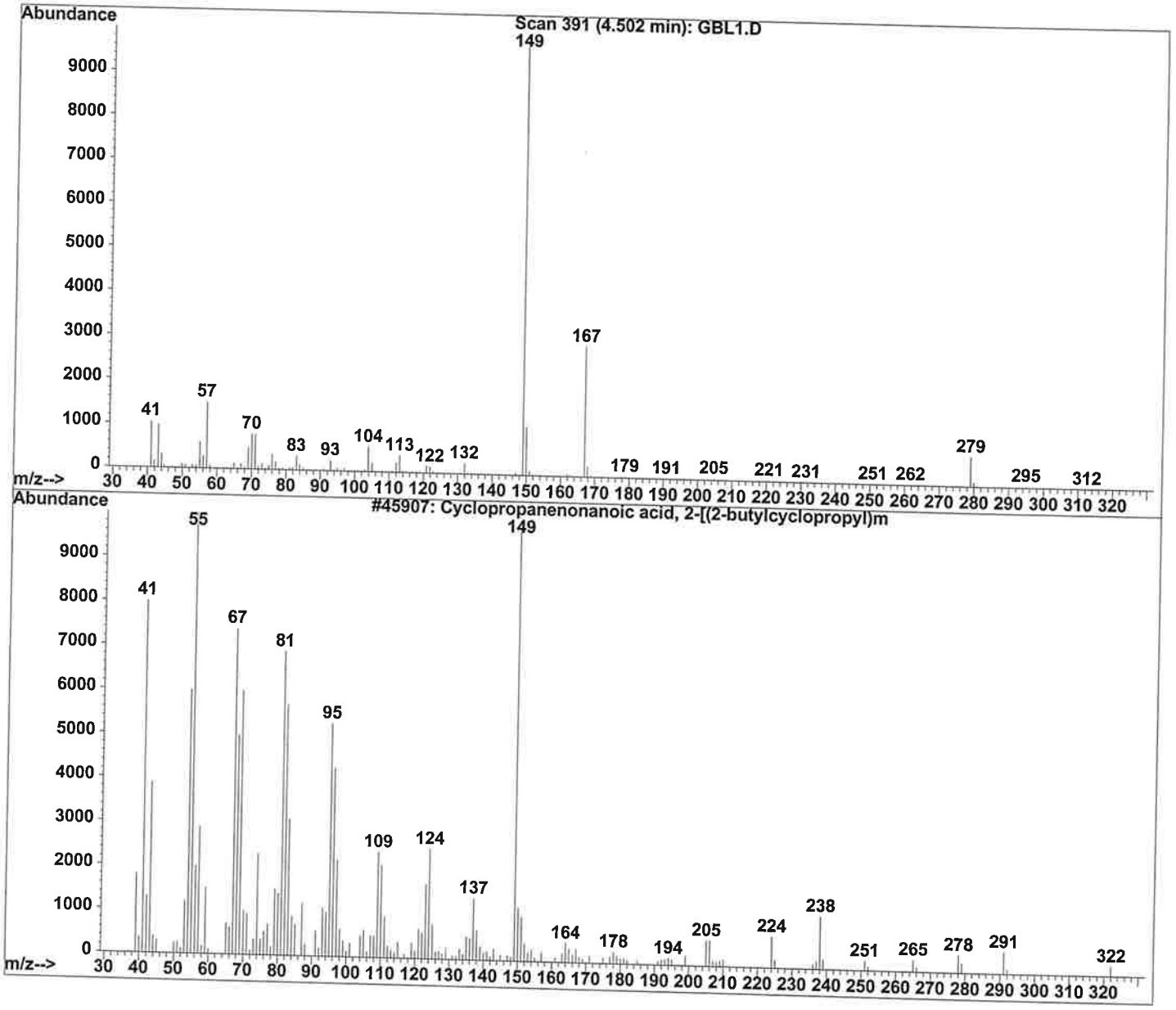
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : Octadecanoic acid, trimethylsilyl ester



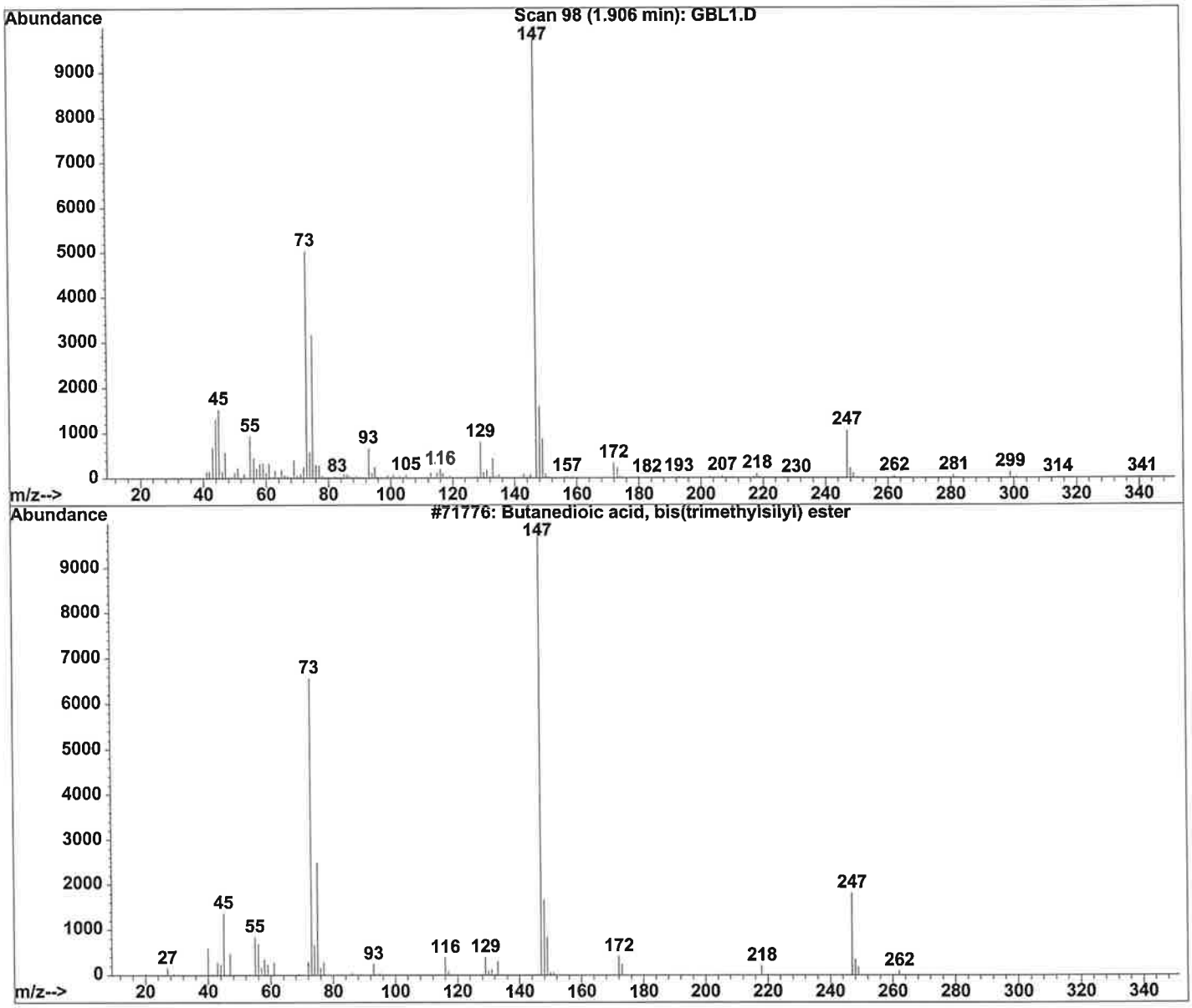
Library Searched : C:\DATABASE\NBS75K.L
Quality : 64
ID : Benzyl butyl phthalate



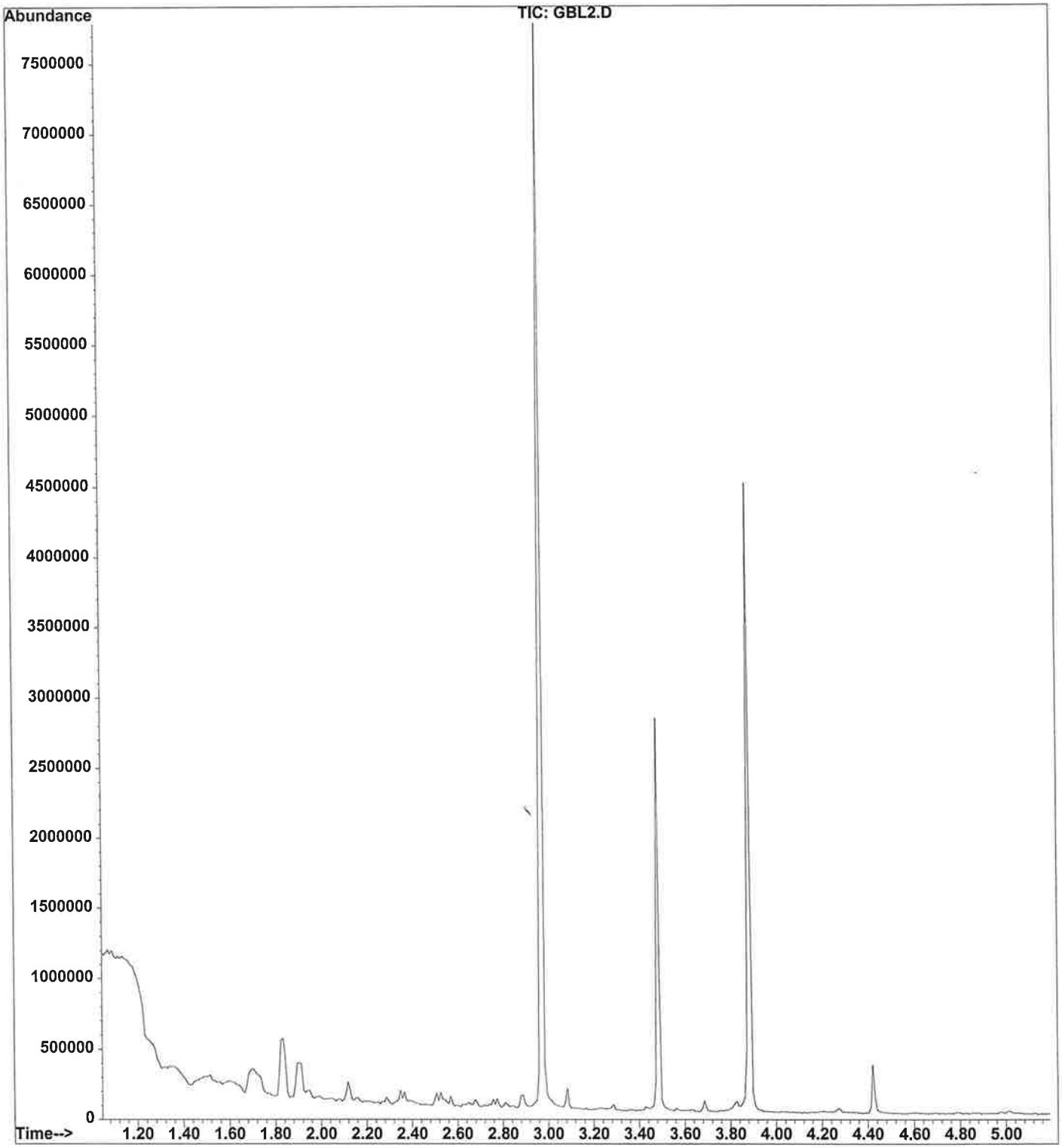
Library Searched : C:\DATABASE\NBS75K.L
Quality : 96
ID : Cyclopropanenonanoic acid, 2-[(2-butylcyclopropyl)methyl]-, methyl ester



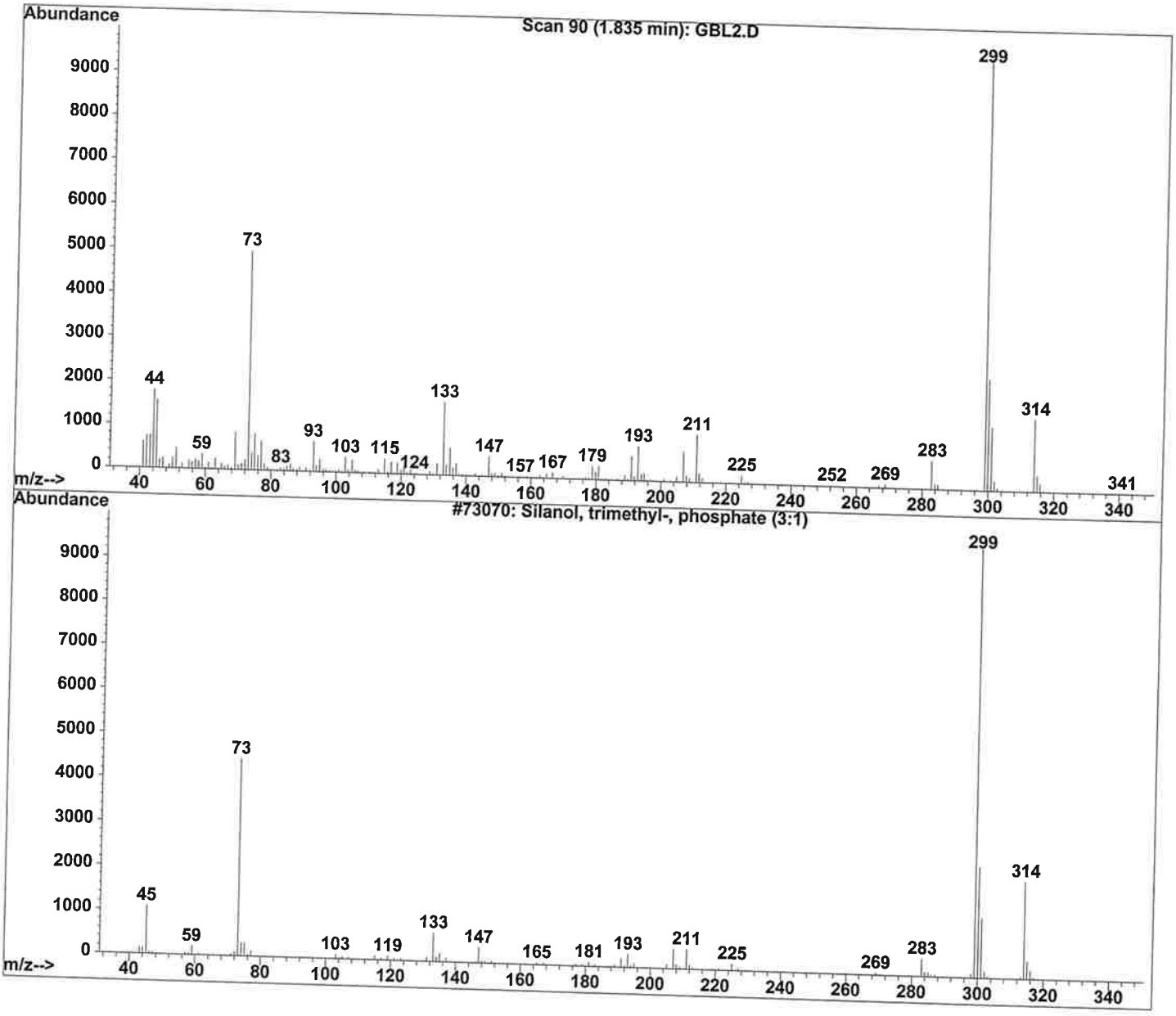
Library Searched : C:\DATABASE\NBS75K.L
Quality : 93
ID : Butanedioic acid, bis(trimethylsilyl) ester



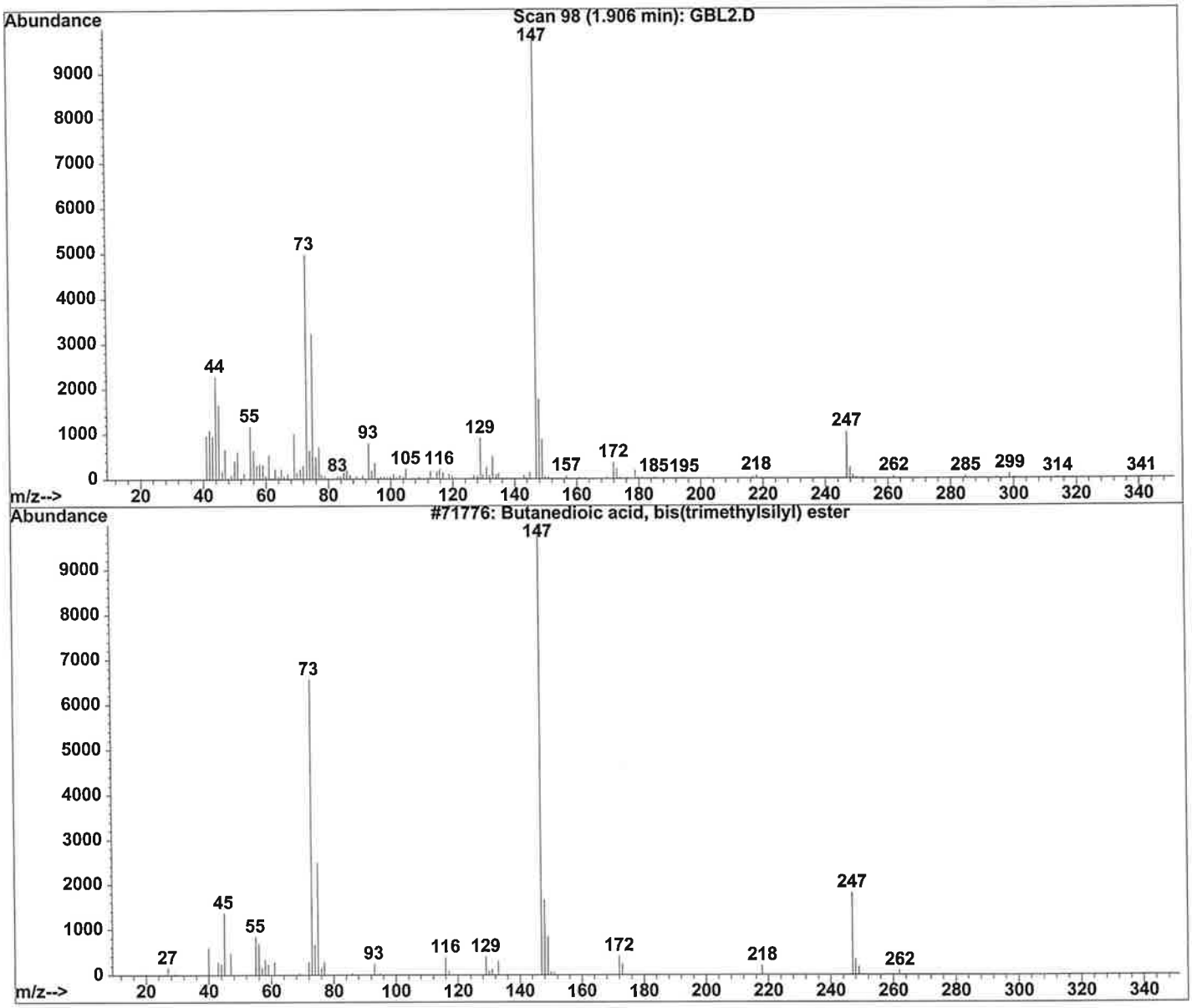
File : C:\HPCHEM\1\DATA\PWF\GBL2.D
Operator : PWF
Acquired : 27 Sep 1999 16:29 using AcqMethod MSDRUGS
Instrument : DONNA
Sample Name: STD GBL NEAT DER
Misc Info :
Vial Number: 1



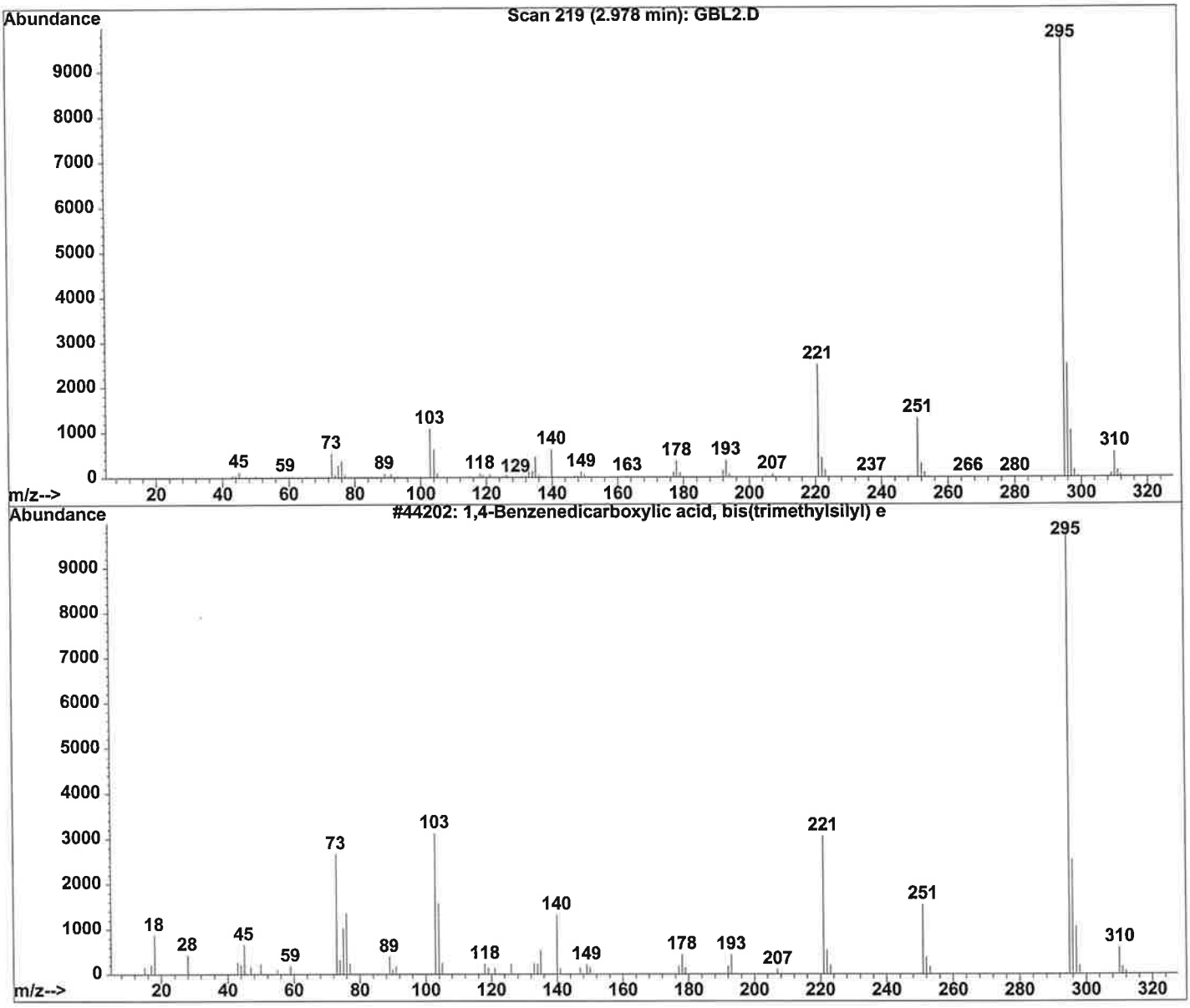
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : Silanol, trimethyl-, phosphate (3:1)



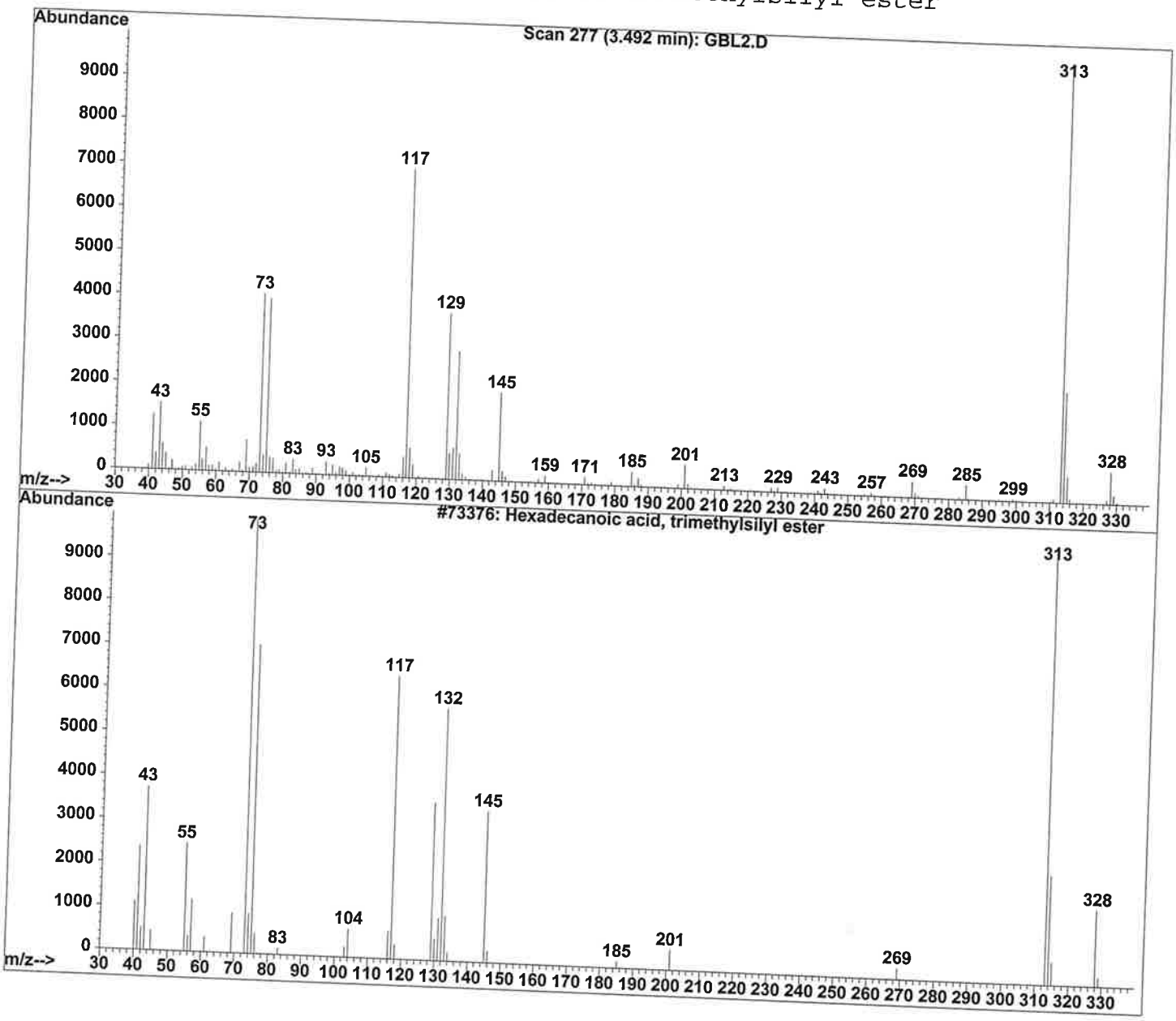
Library Searched : C:\DATABASE\NBS75K.L
Quality : 87
ID : Butanedioic acid, bis(trimethylsilyl) ester



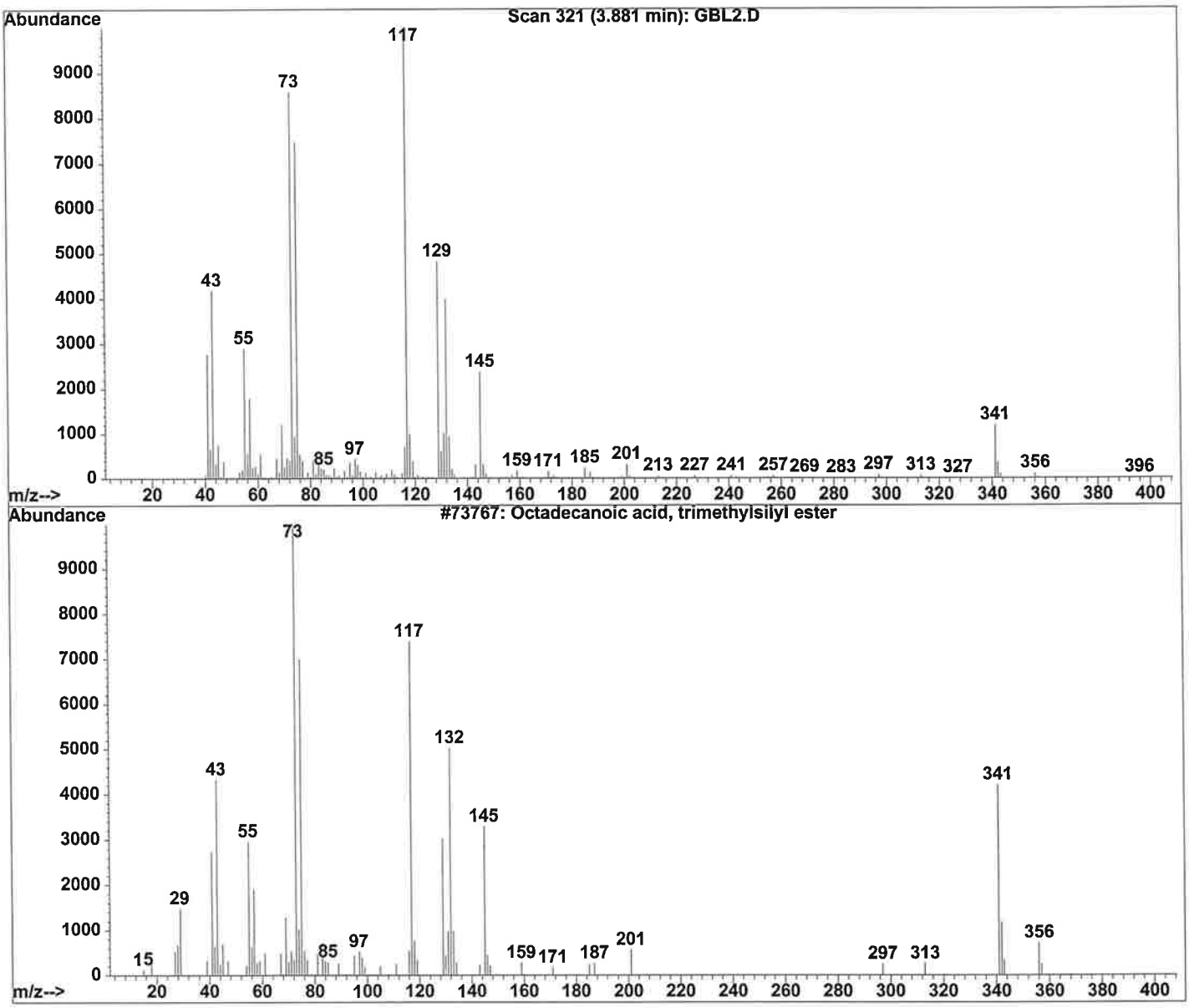
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : 1,4-Benzenedicarboxylic acid, bis(trimethylsilyl) este
r



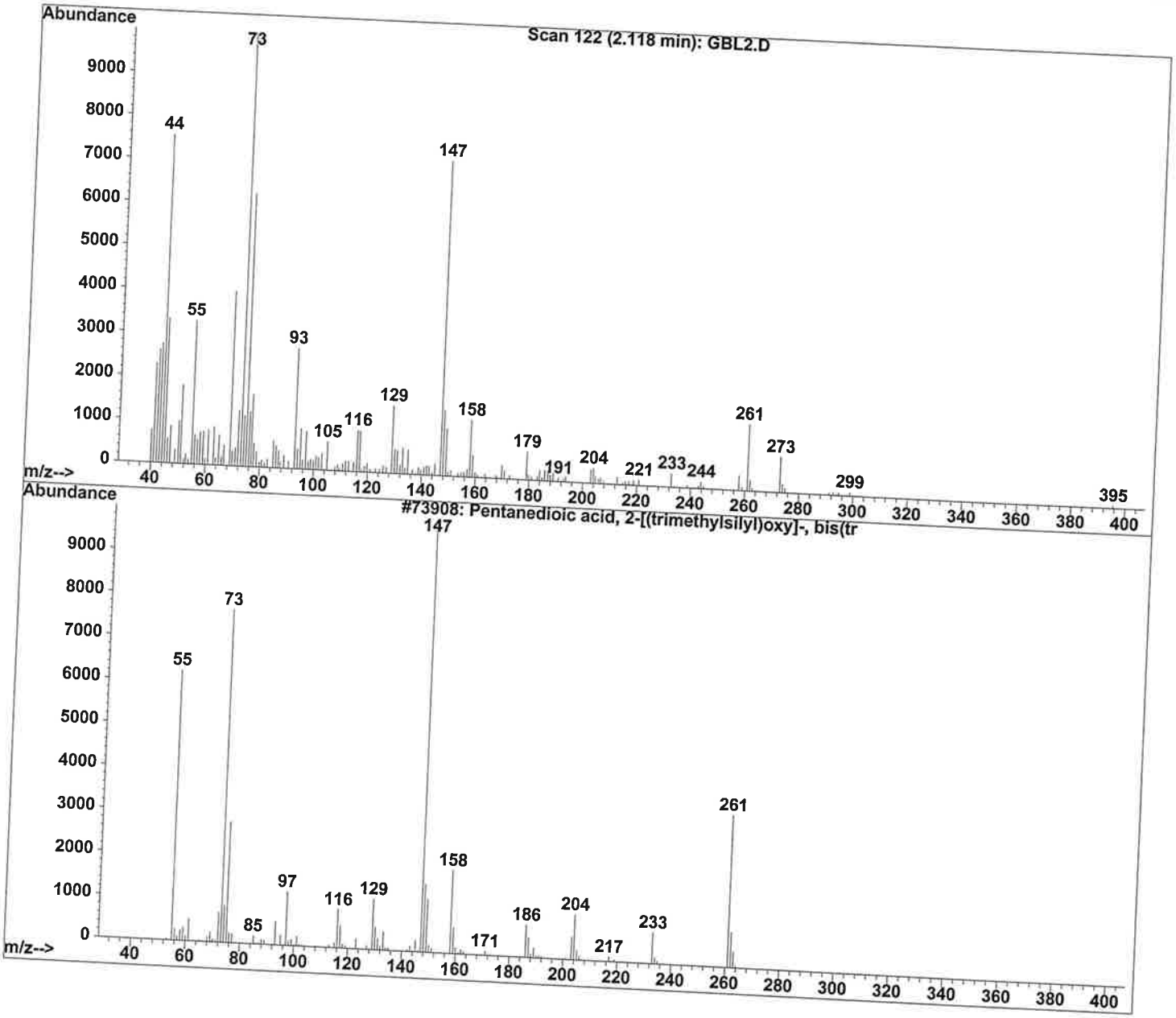
Library Searched : C:\DATABASE\NBS75K.L
Quality : 91
ID : Hexadecanoic acid, trimethylsilyl ester



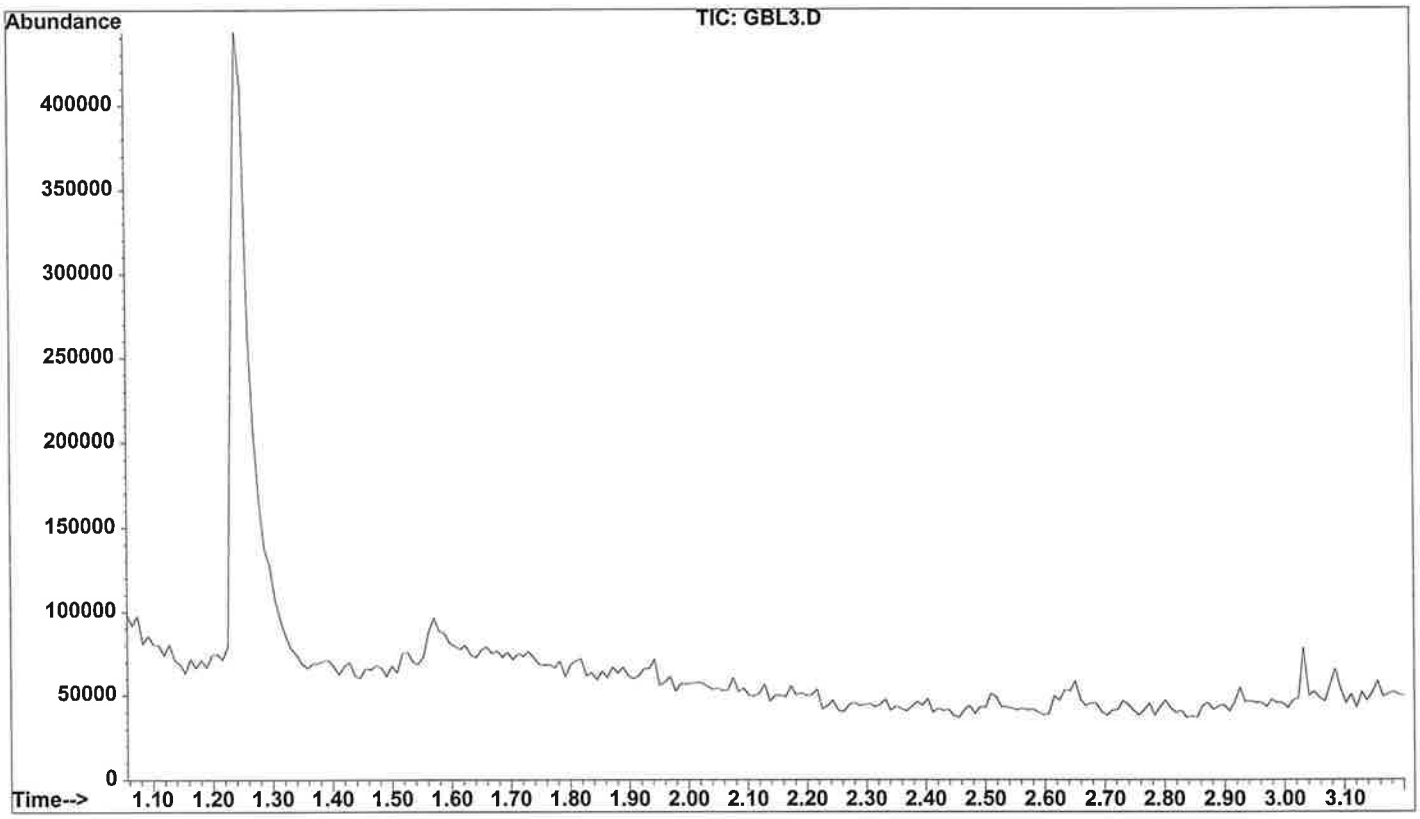
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : Octadecanoic acid, trimethylsilyl ester

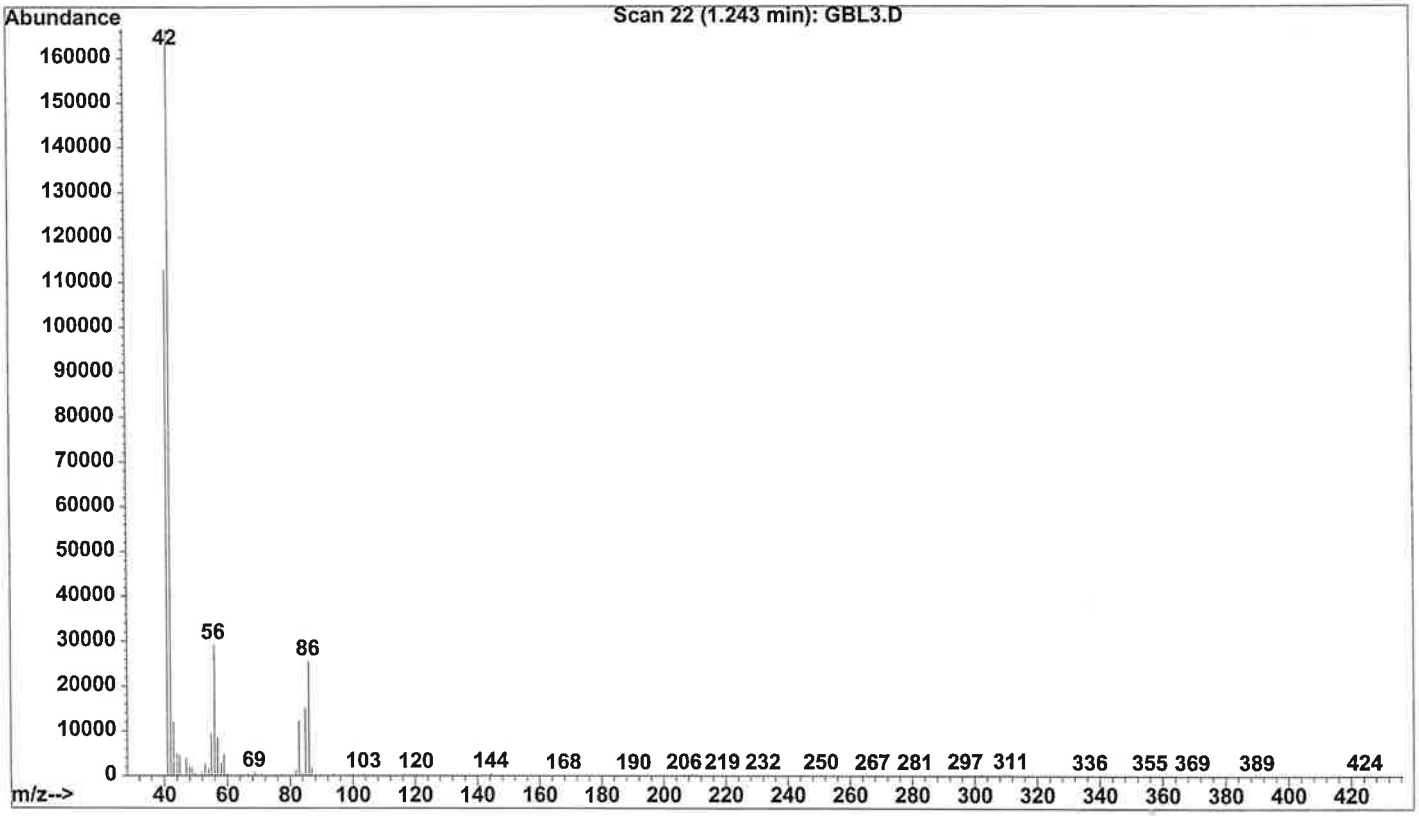


Library Searched : C:\DATABASE\NBS75K.L
Quality : 64
ID : Pentanedioic acid, 2-[(trimethylsilyl)oxy]-, bis(trimethylsilyl) ester

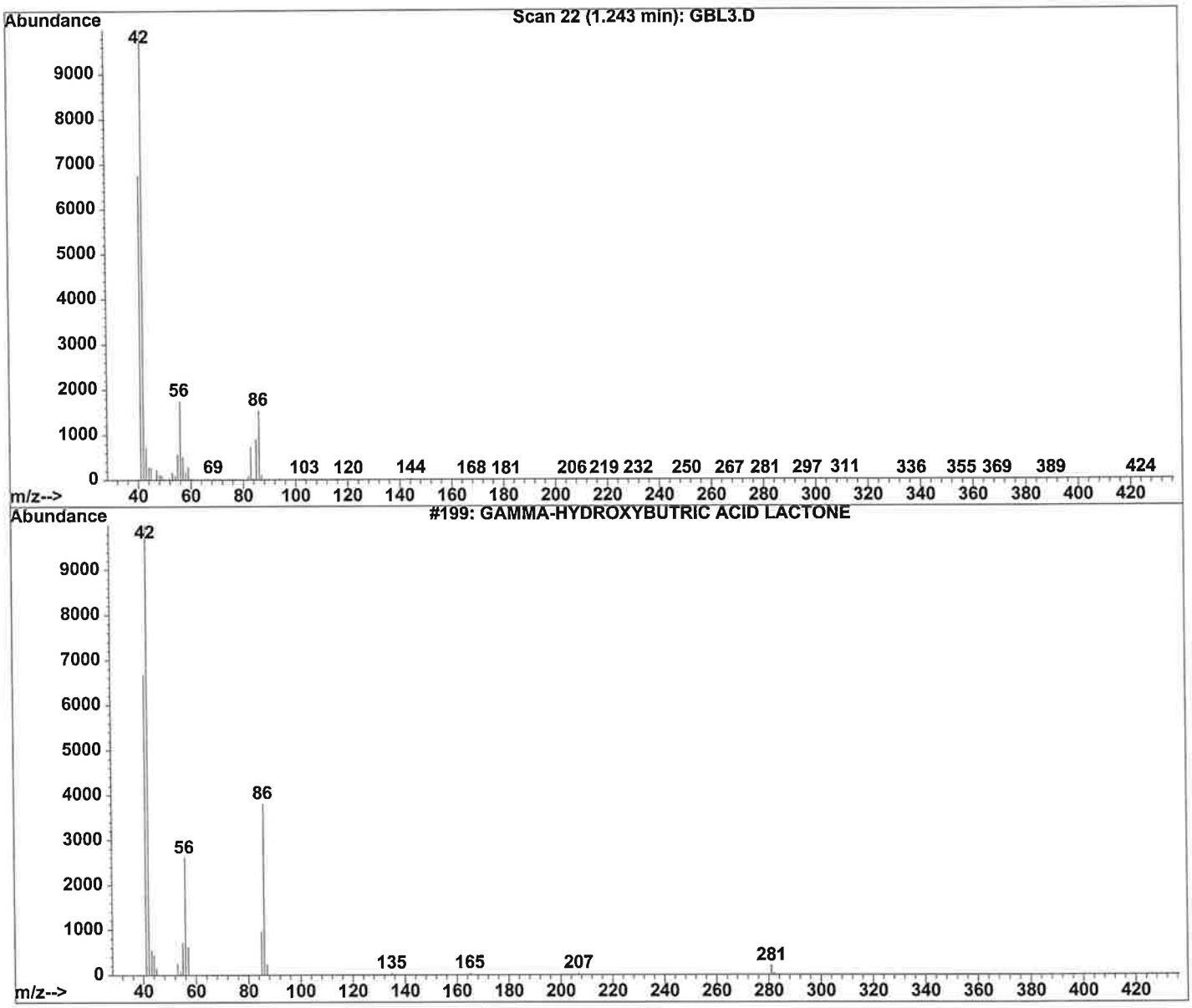


File : C:\HPCHEM\1\DATA\PWF\GBL3.D
Operator : PWF
Acquired : 28 Sep 1999 7:12 using AcqMethod MSDGHB
Instrument : DONNA
Sample Name: STD GBL IN H2O CHCL3 X 4
Misc Info :
Vial Number: 1

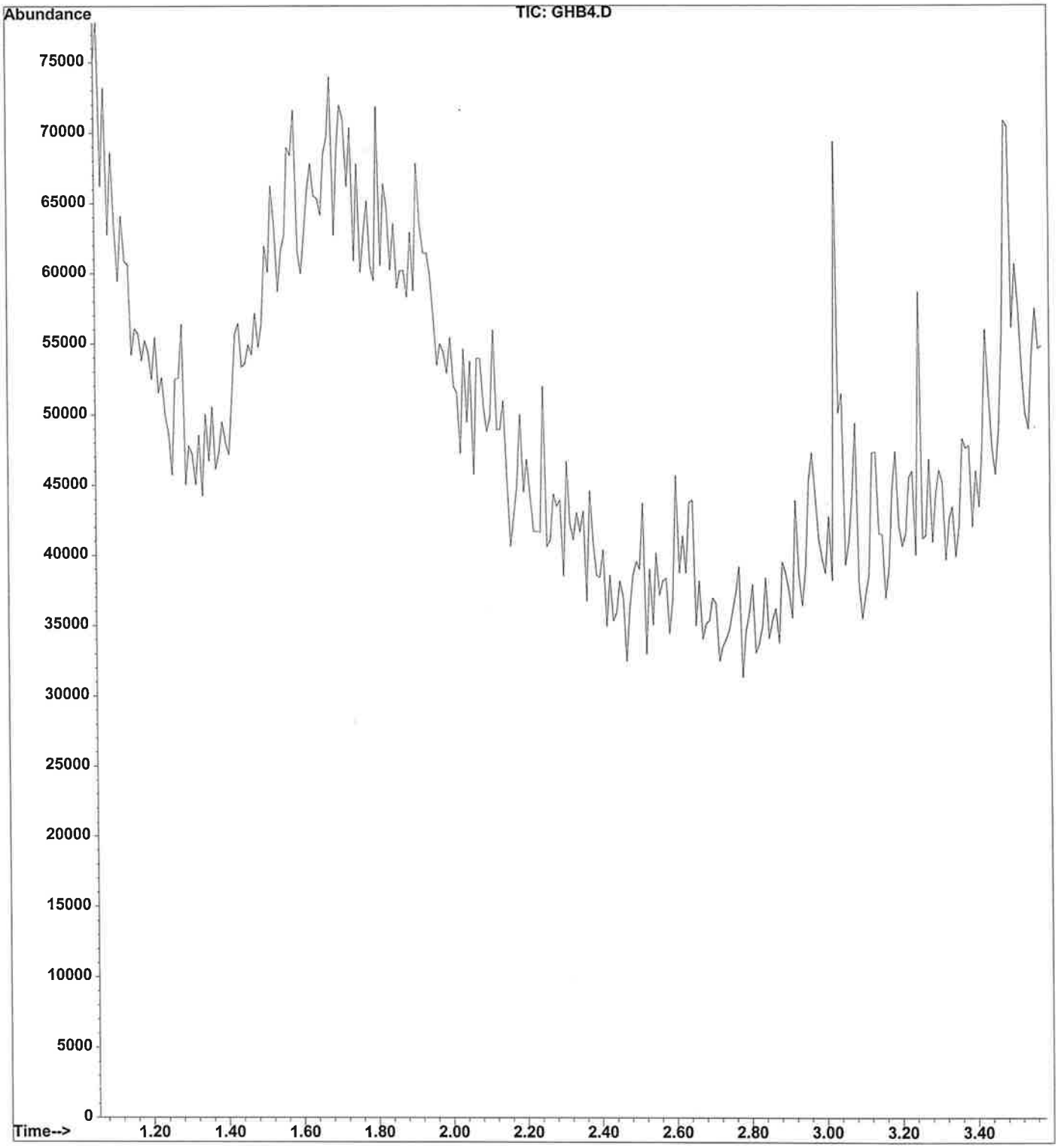




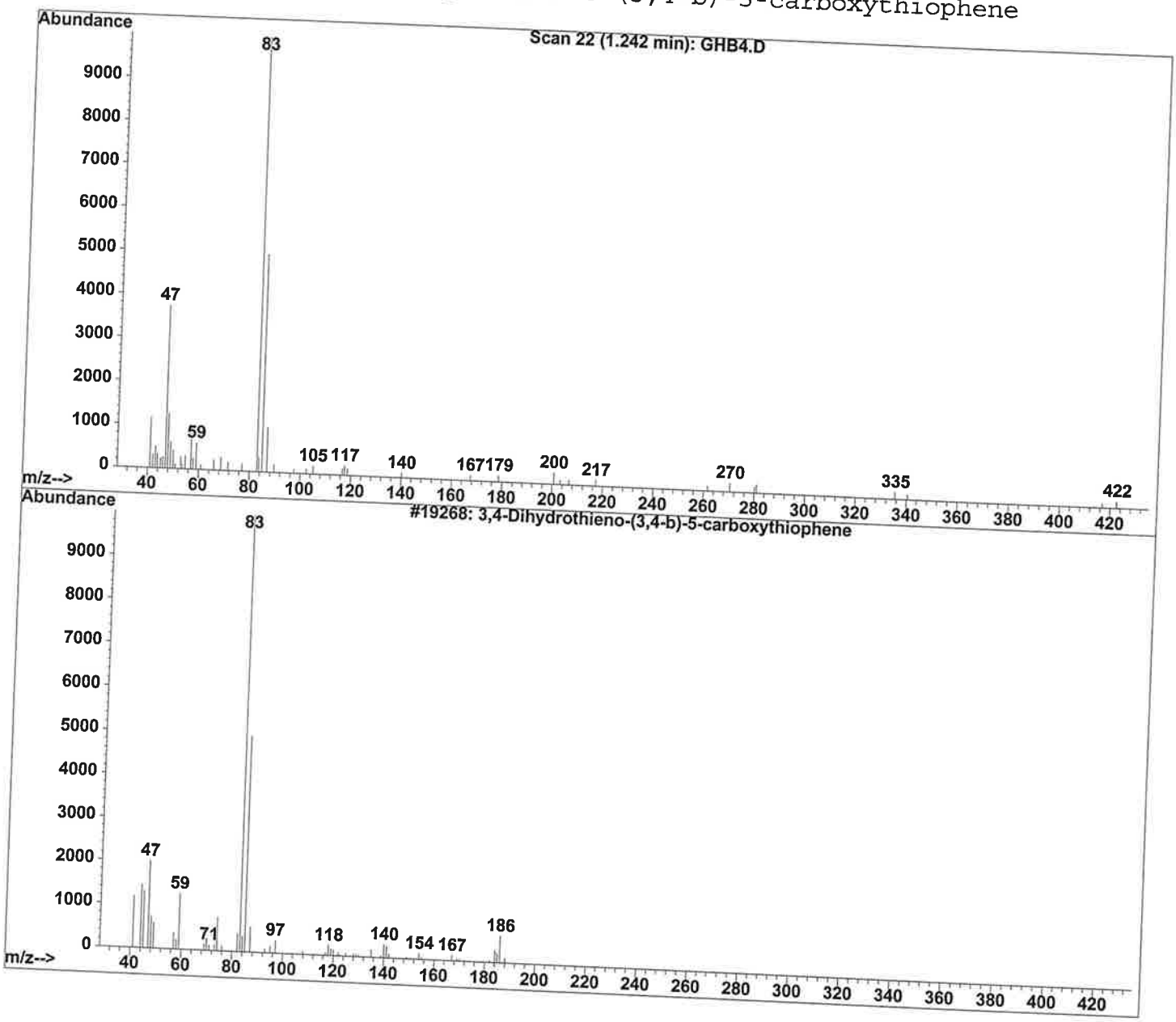
Library Searched : C:\DATABASE\TBI_STD.L
Quality : 64
ID : GAMMA-HYDROXYBUTRIC ACID LACTONE



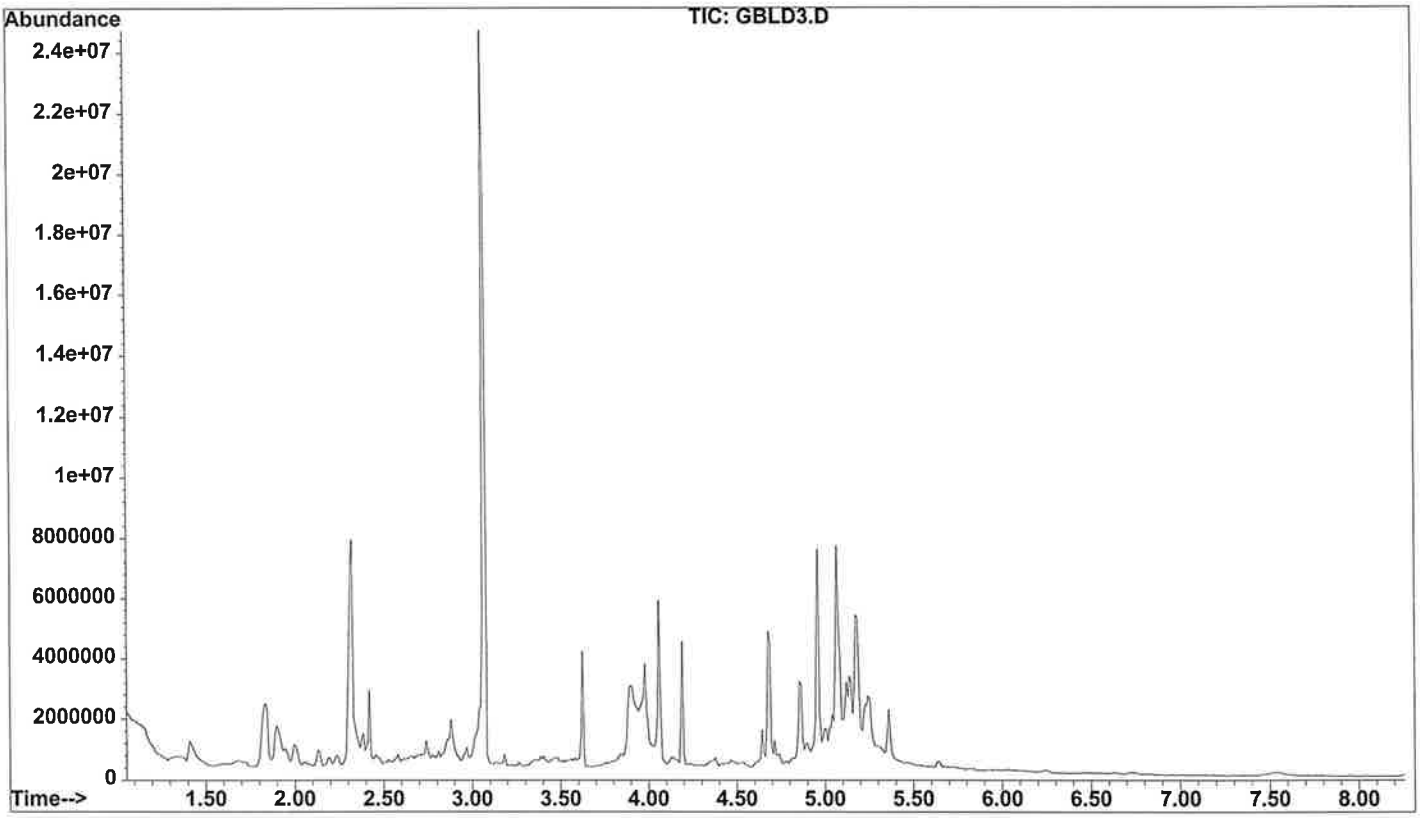
File : C:\HPCHEM\1\DATA\GHB4.D
Operator : PWF
Acquired : 28 Sep 1999 7:33 using AcqMethod MSDGHB
Instrument : DONNA
Sample Name: STD GHB CHCH3 X7
Misc Info :
Vial Number: 1

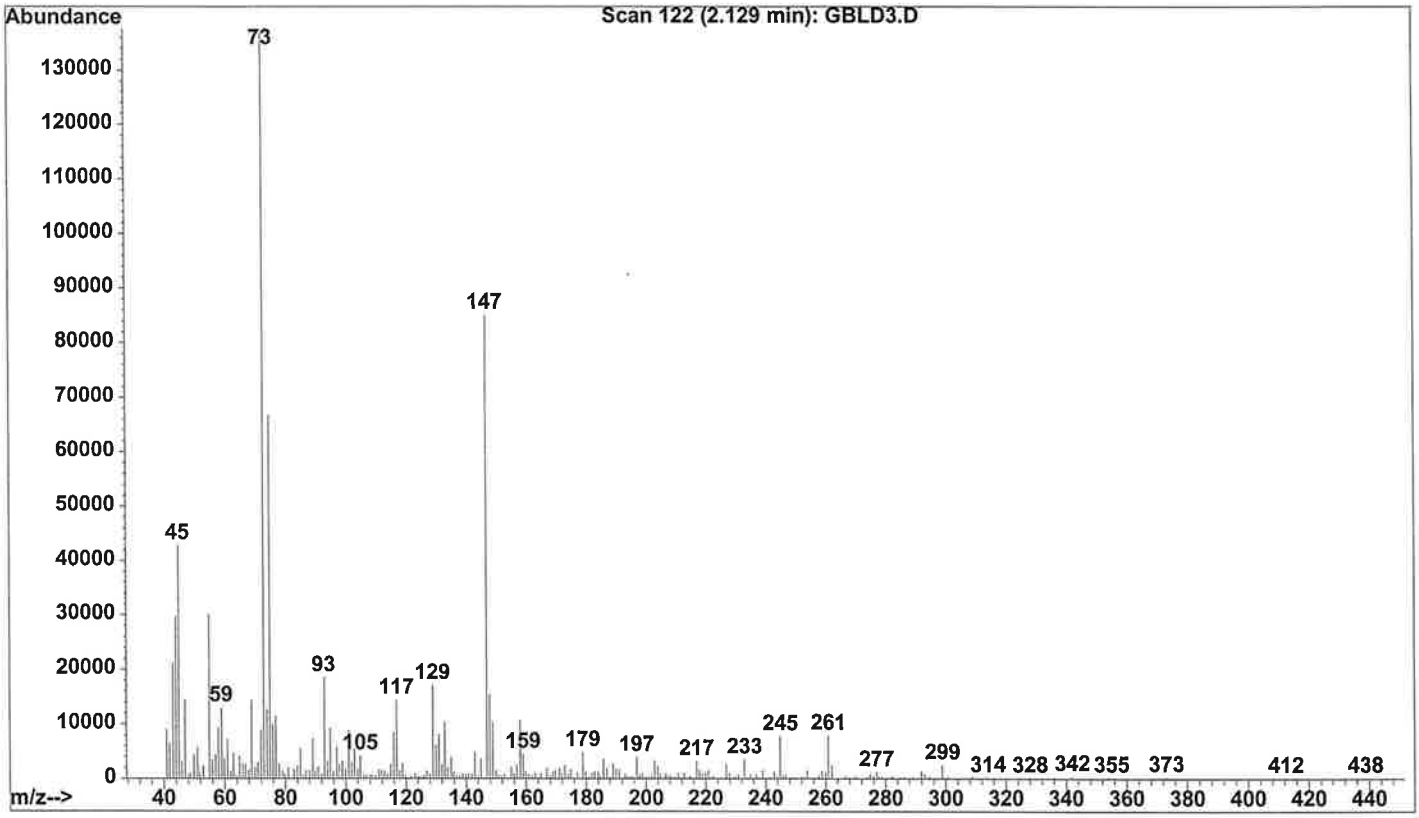


Library Searched : C:\DATABASE\NBS75K.L
Quality : 83
ID : 3,4-Dihydrothieno-(3,4-b)-5-carboxythiophene

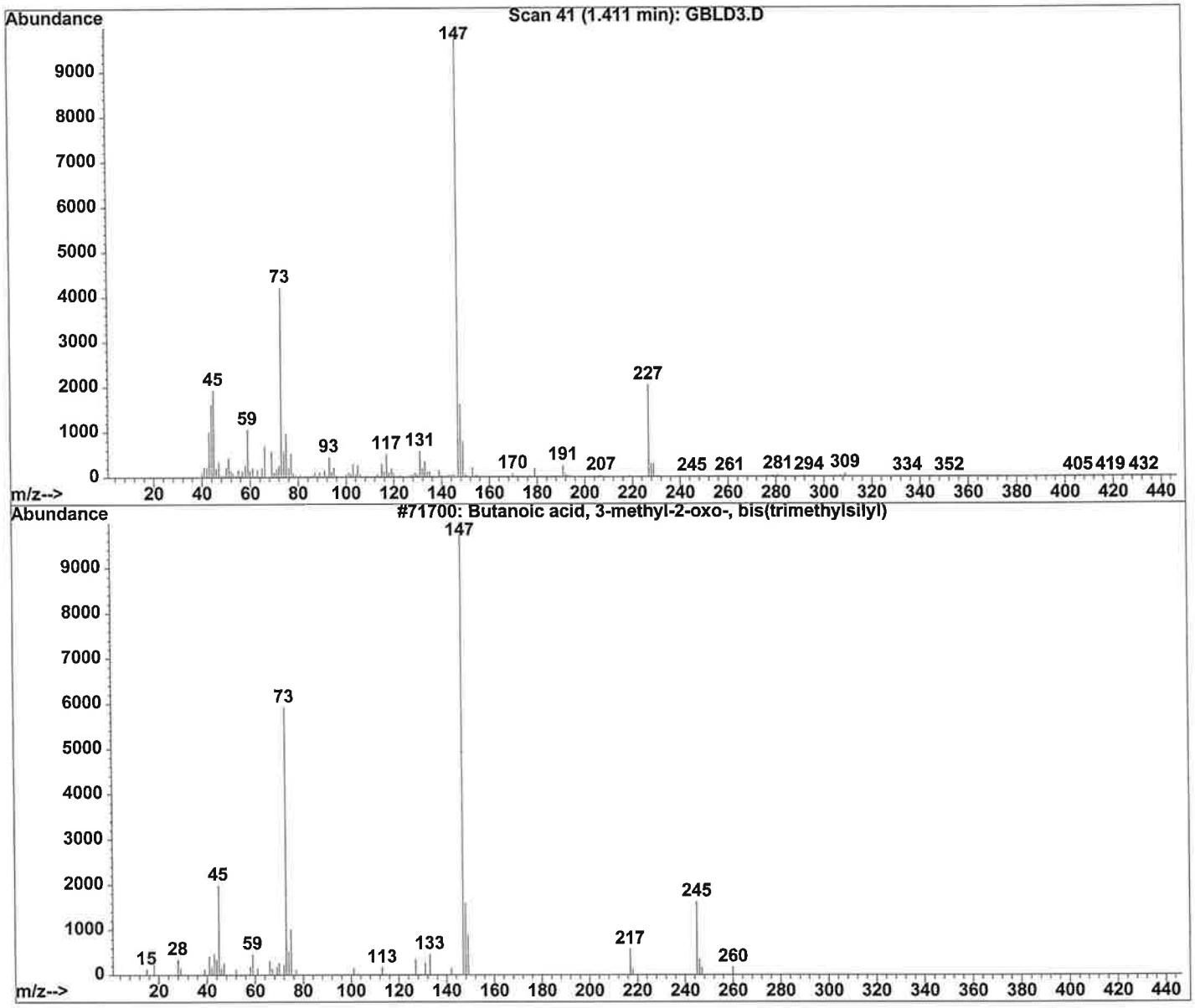


File : C:\HPCHEM\1\DATA\PWF\GBLD3.D
Operator : PWF
Acquired : 28 Sep 1999 9:38 using AcqMethod MSDRUGS
Instrument : DONNA
Sample Name: STD GBL W/H2O DER
Misc Info :
Vial Number: 1

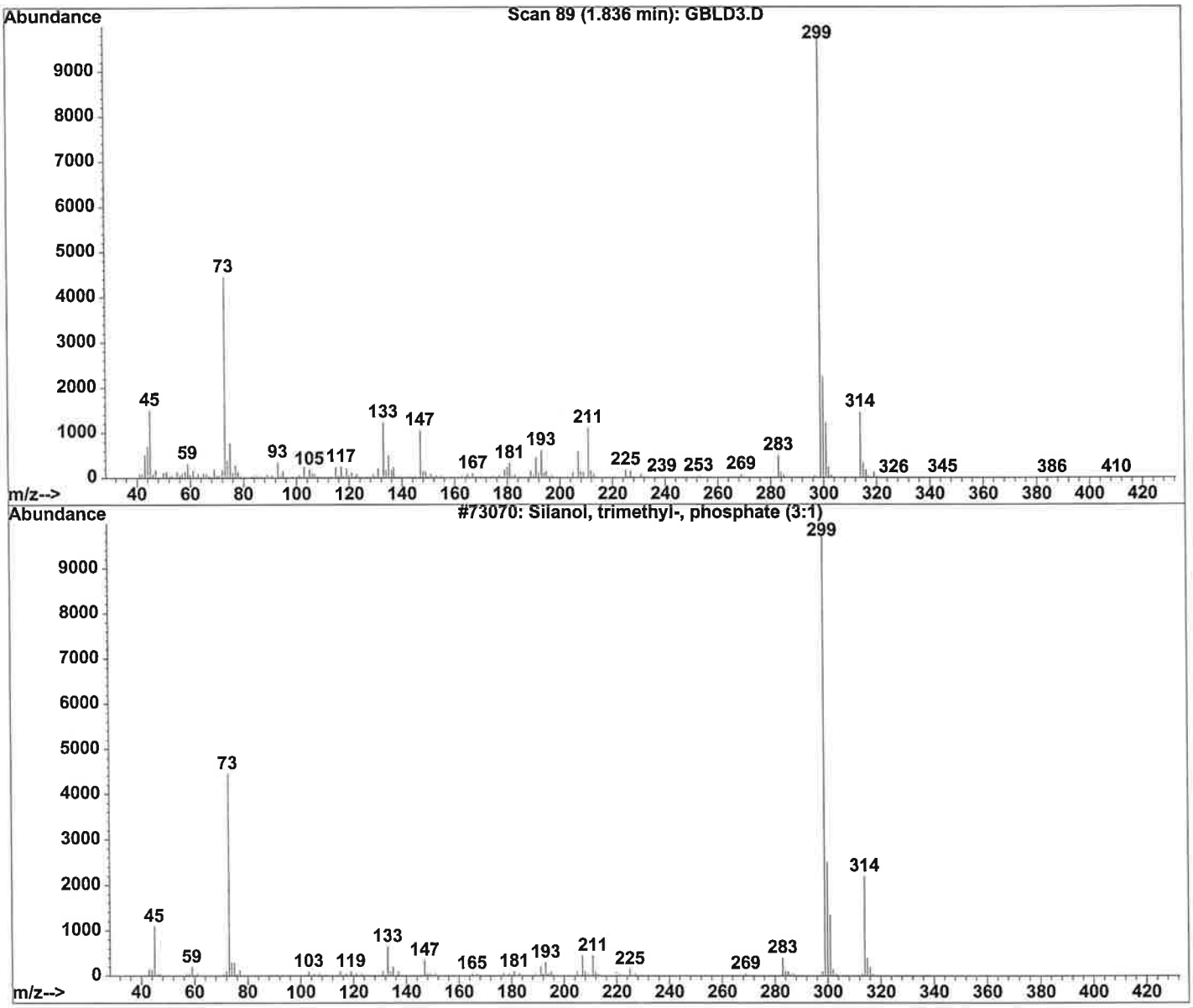




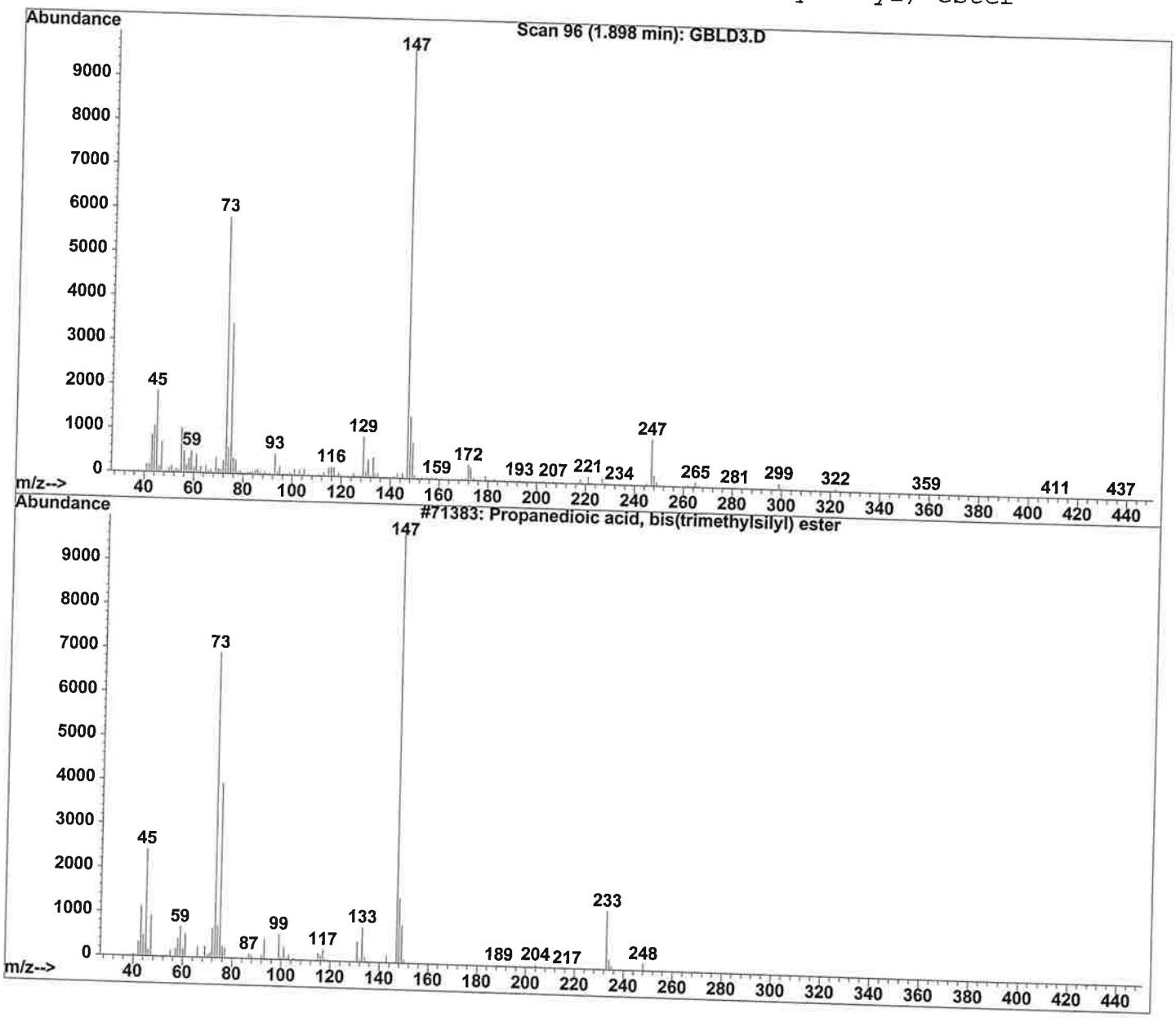
Library Searched : C:\DATABASE\NBS75K.L
Quality : 64
ID : Butanoic acid, 3-methyl-2-oxo-, bis(trimethylsilyl) deriv.



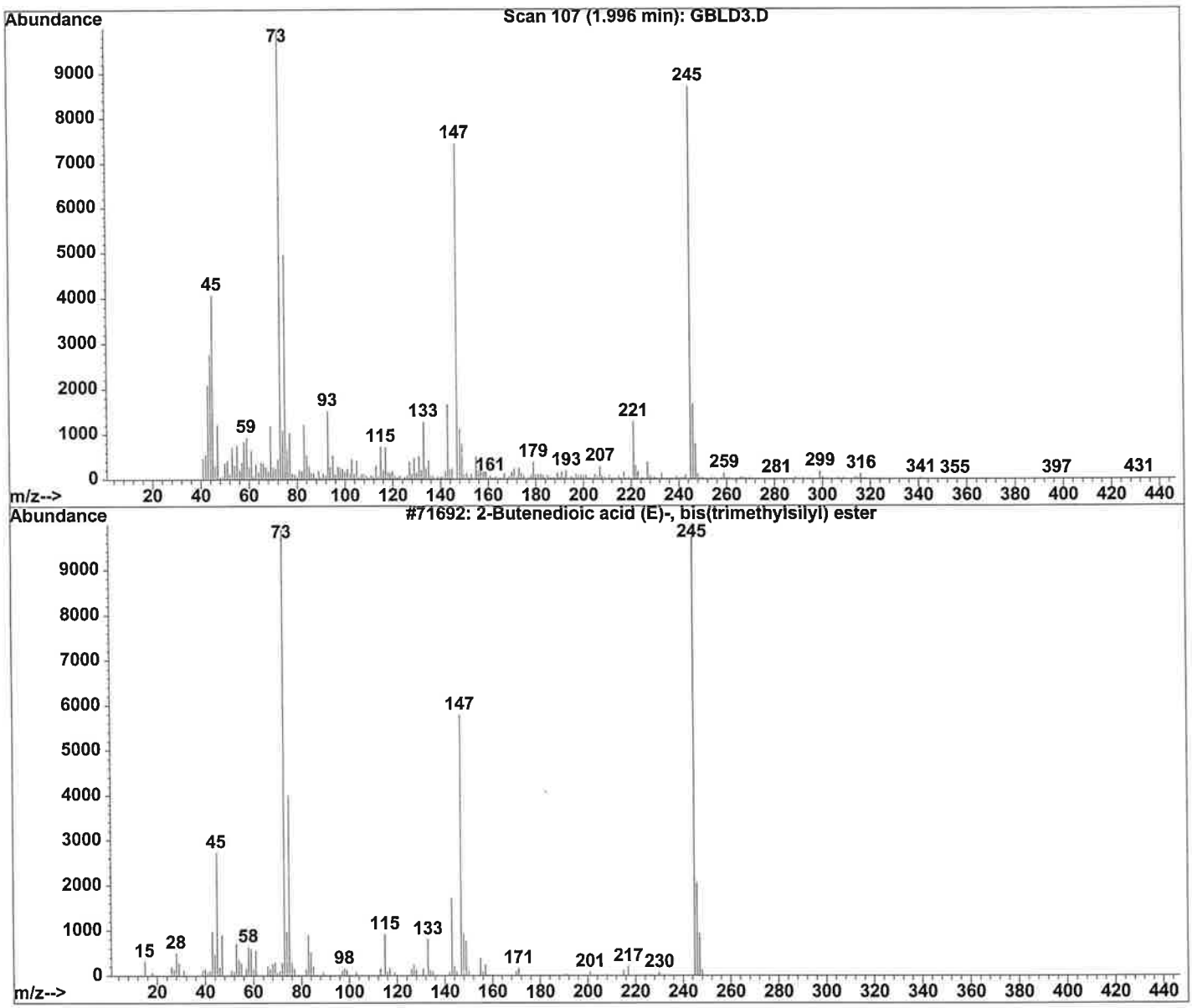
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : Silanol, trimethyl-, phosphate (3:1)



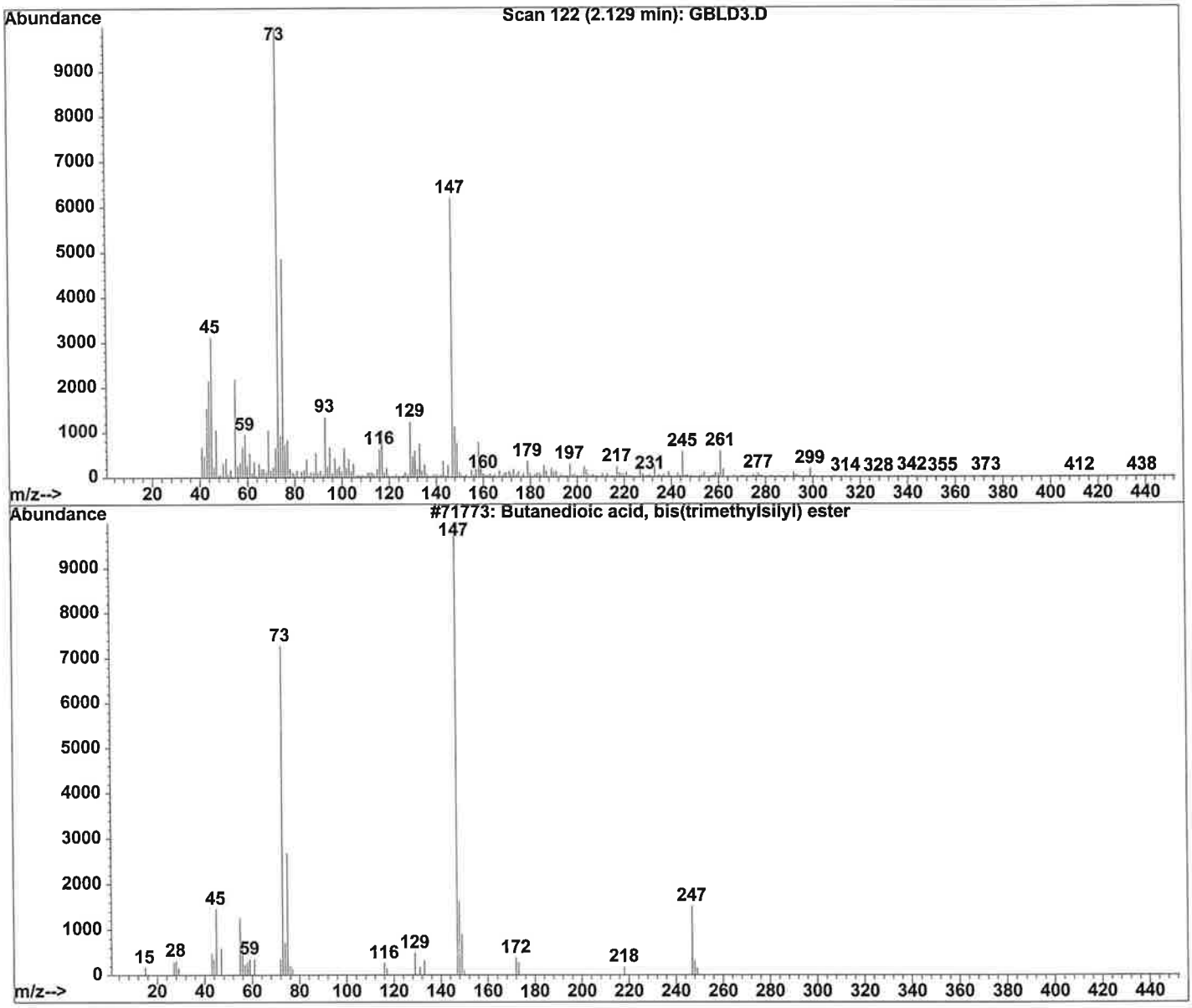
Library Searched : C:\DATABASE\NBS75K.L
Quality : 90
ID : Propanedioic acid, bis(trimethylsilyl) ester



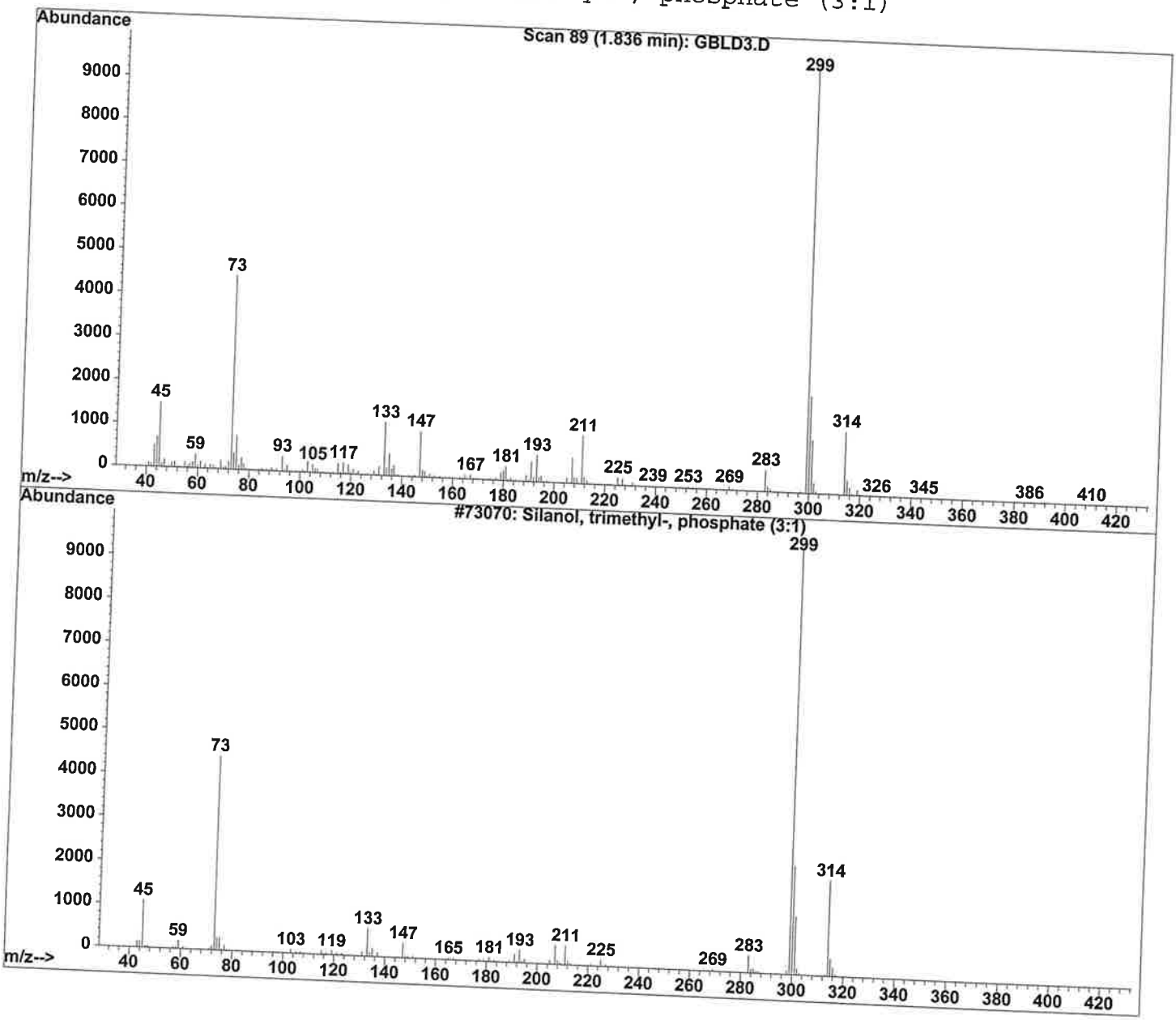
Library Searched : C:\DATABASE\NBS75K.L
Quality : 70
ID : 2-Butenedioic acid (E)-, bis(trimethylsilyl) ester



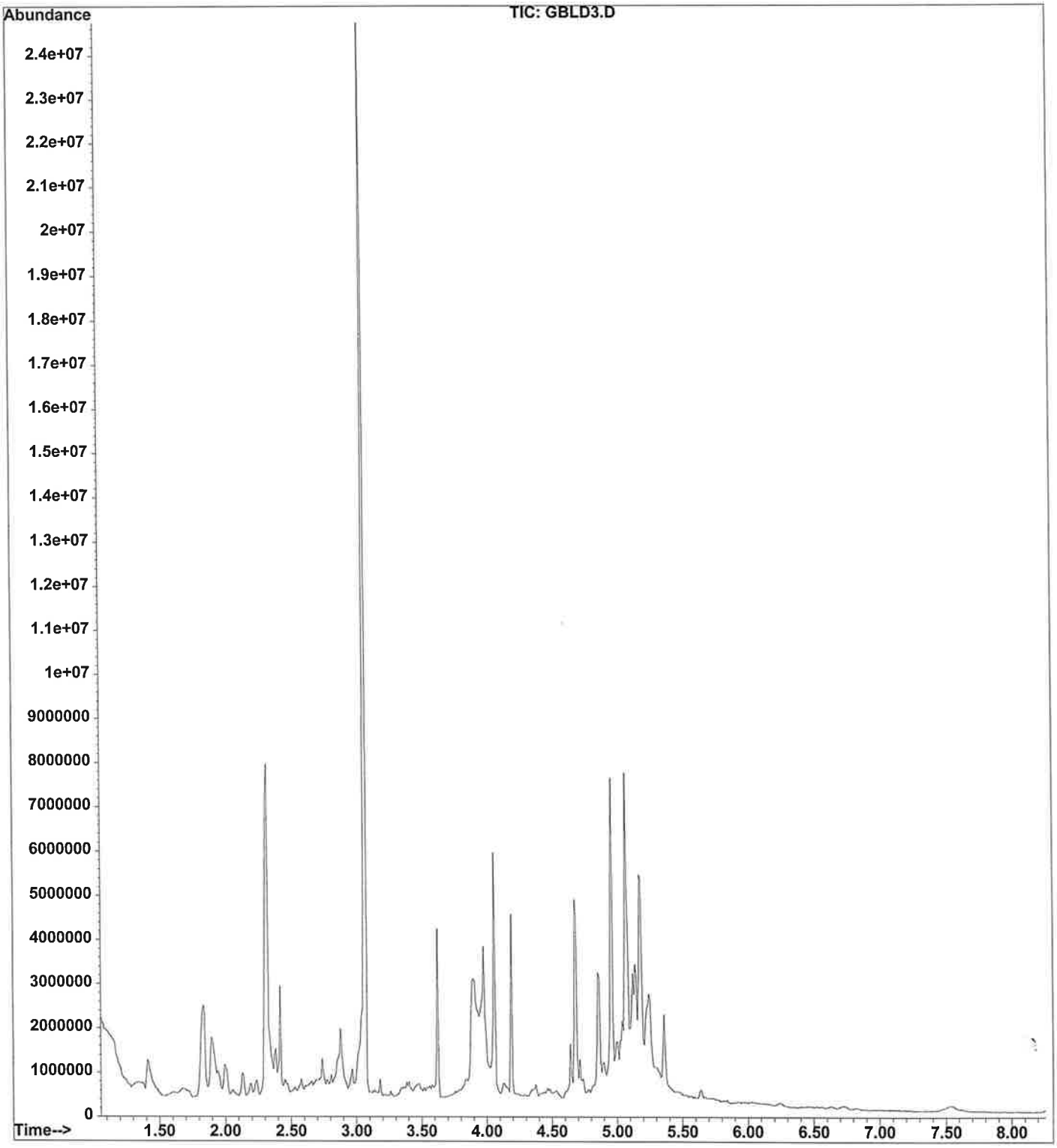
Library Searched : C:\DATABASE\NBS75K.L
Quality : 72
ID : Butanedioic acid, bis(trimethylsilyl) ester



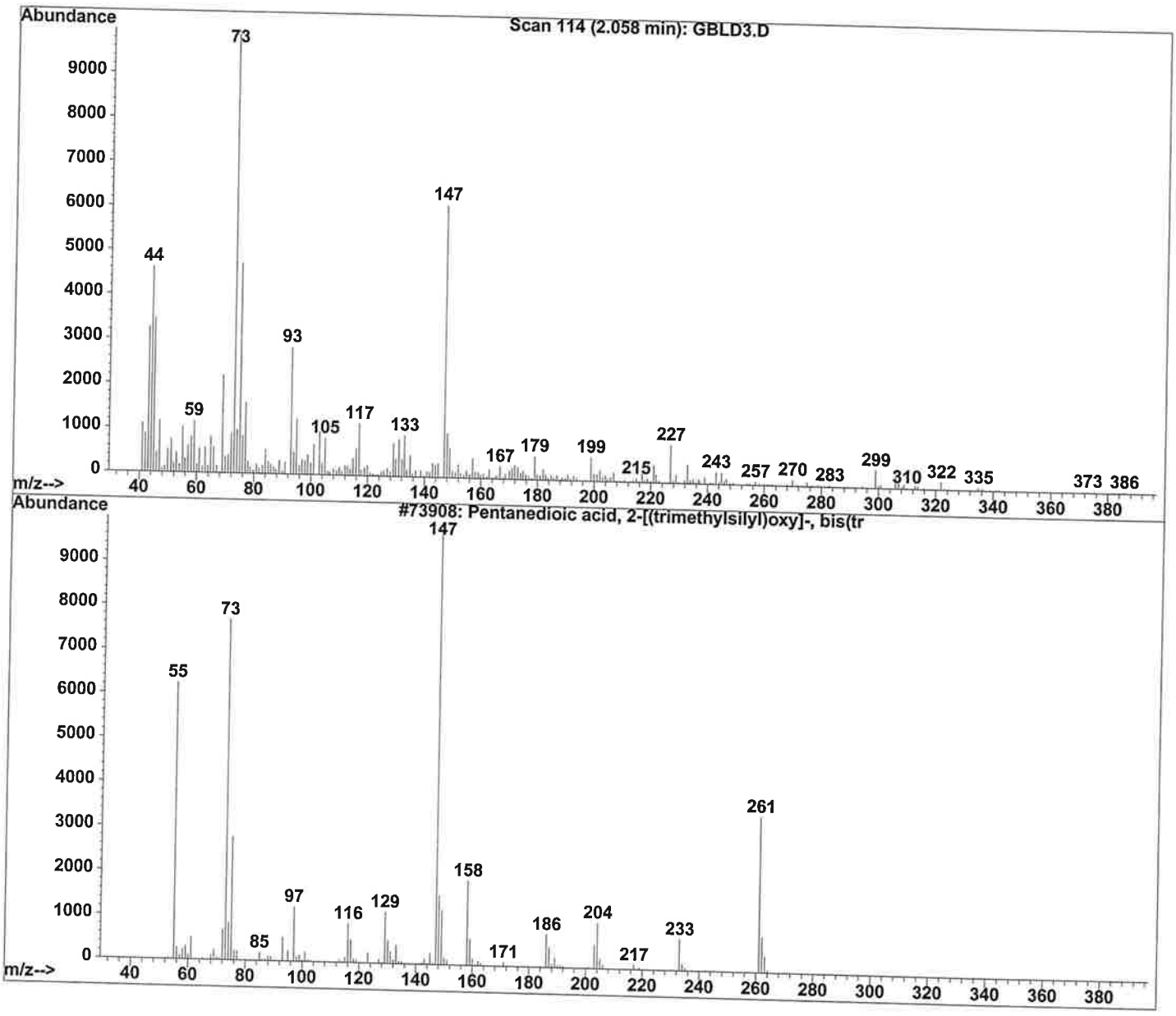
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : Silanol, trimethyl-, phosphate (3:1)



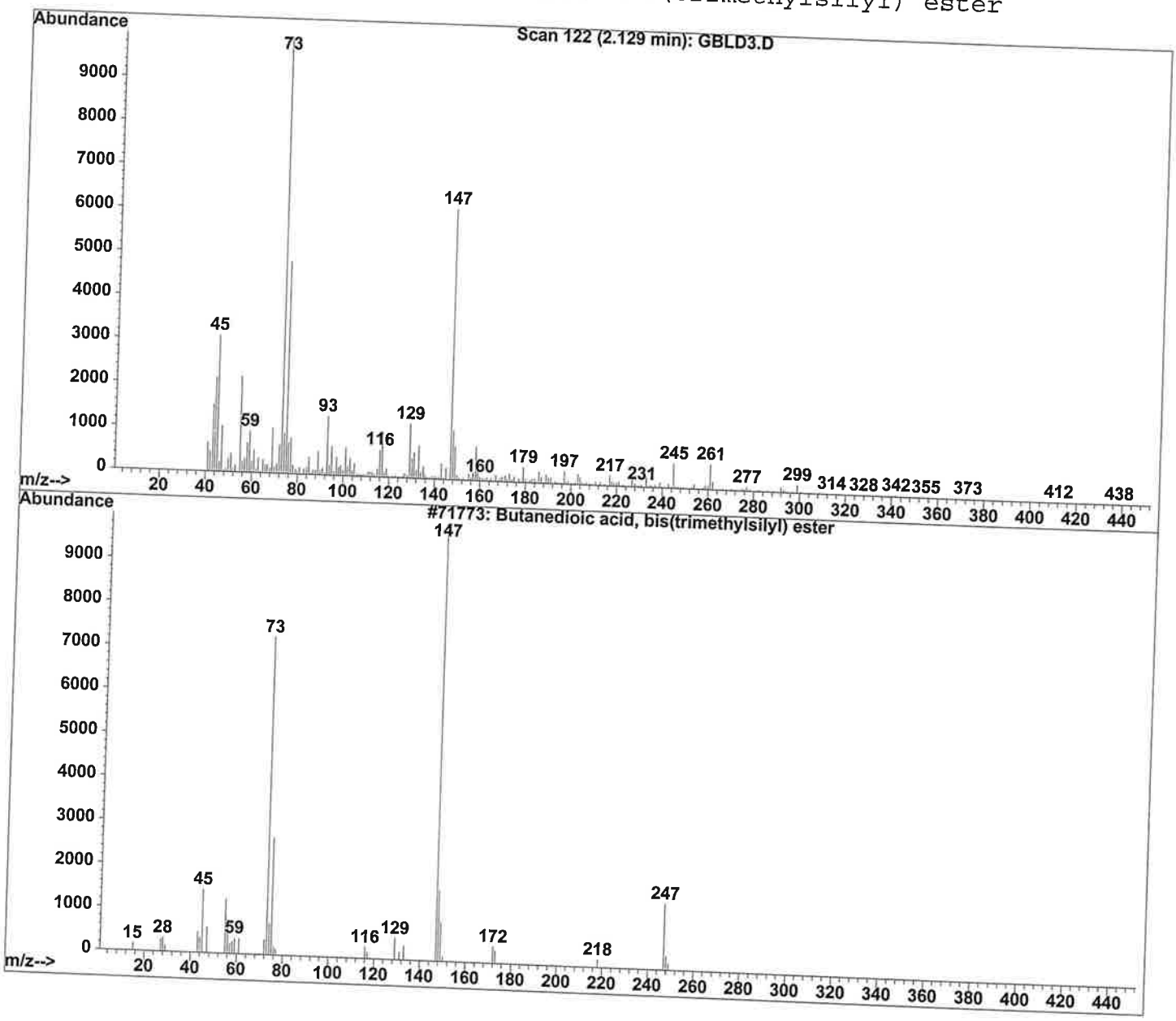
File : C:\HPCHEM\1\DATA\PWF\GBLD3.D
Operator : PWF
Acquired : 28 Sep 1999 9:38 using AcqMethod MSDRUGS
Instrument : DONNA
Sample Name: STD GBL W/H2O DER
Misc Info :
Vial Number: 1



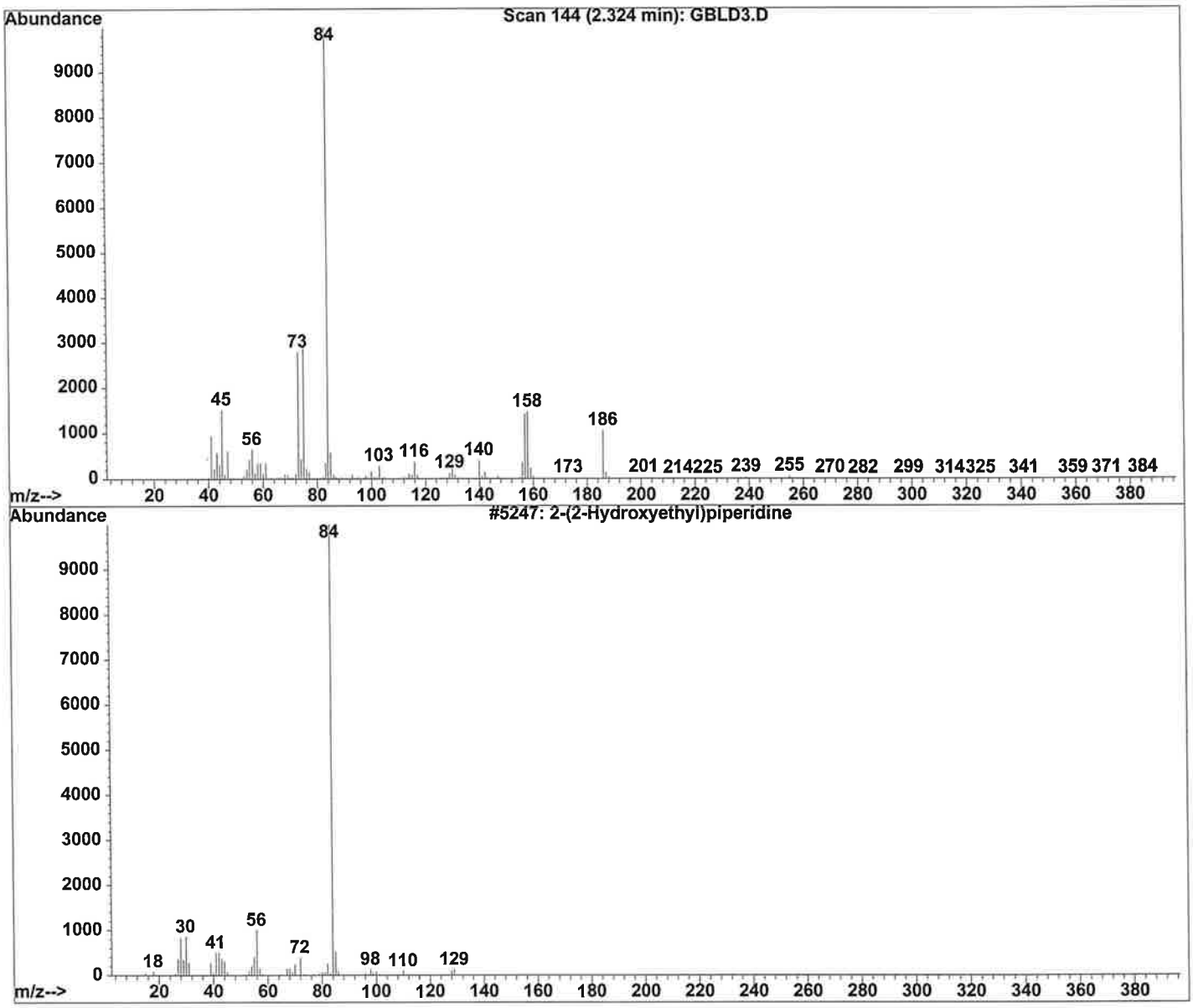
Library Searched : C:\DATABASE\NBS75K.L
Quality : 50
ID : Pentanedioic acid, 2-[(trimethylsilyl)oxy]-, bis(trimethylsilyl) ester



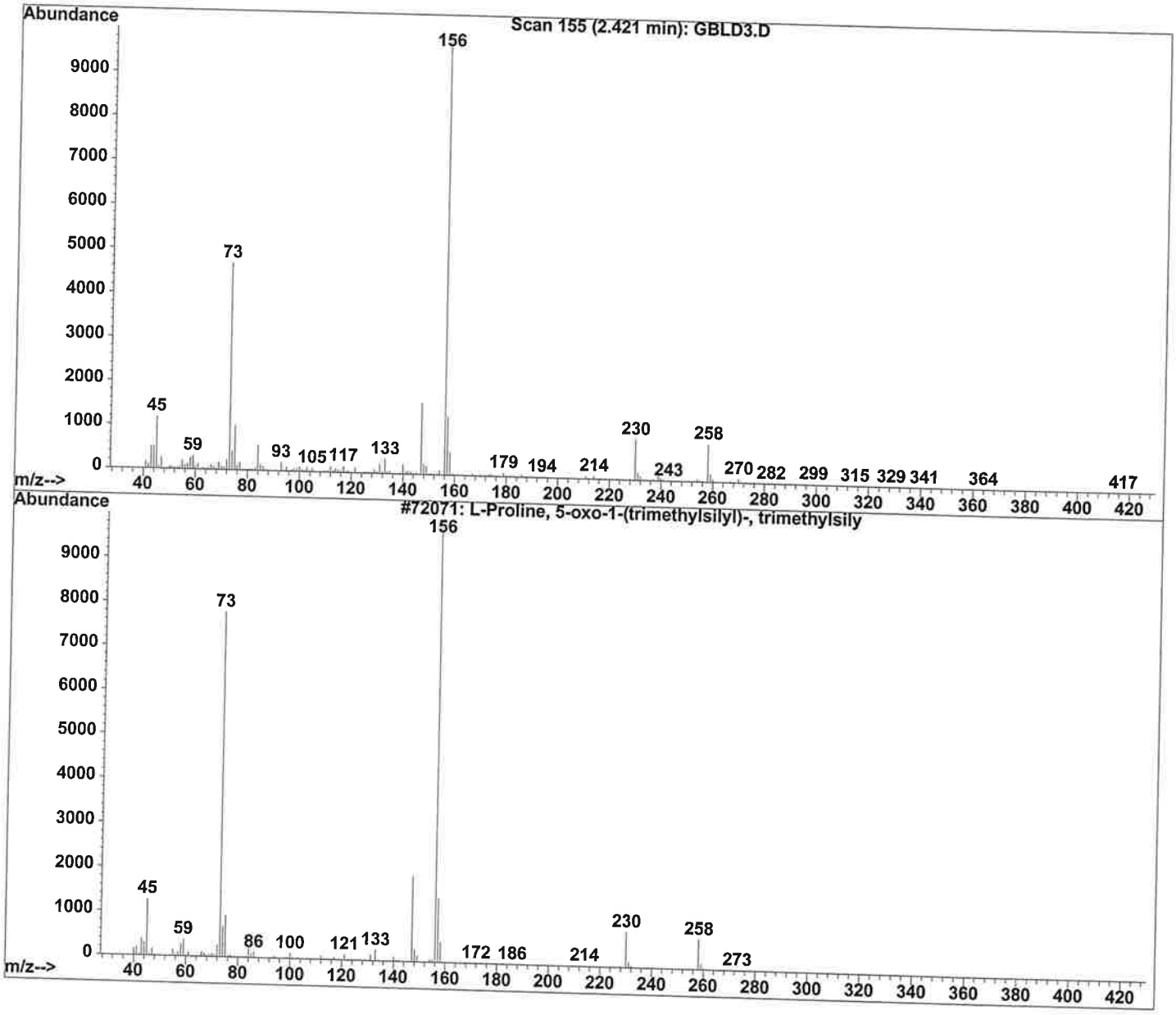
Library Searched : C:\DATABASE\NBS75K.L
Quality : 72
ID : Butanedioic acid, bis(trimethylsilyl) ester



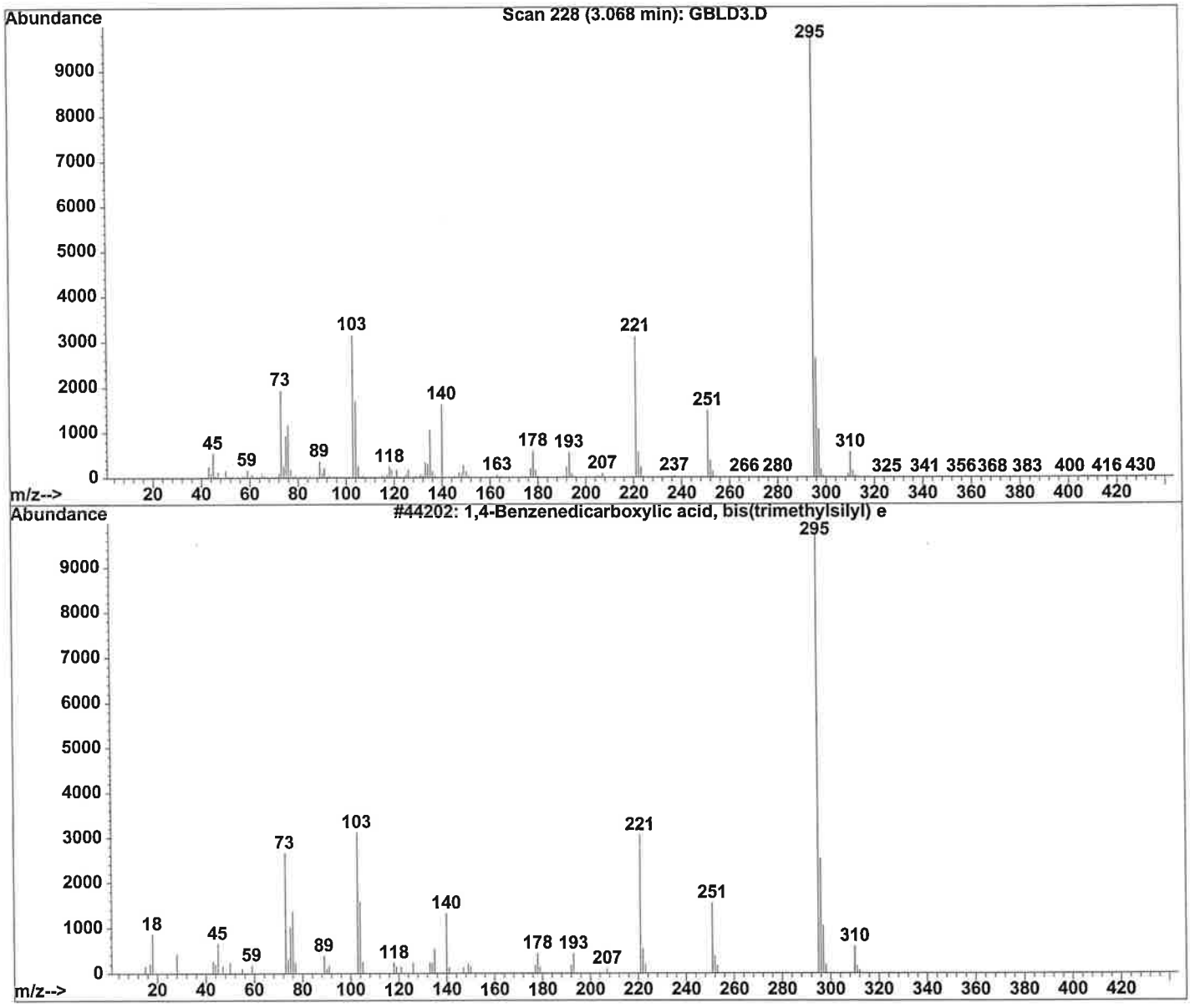
Library Searched : C:\DATABASE\NBS75K.L
Quality : 38
ID : 2-(2-Hydroxyethyl)piperidine



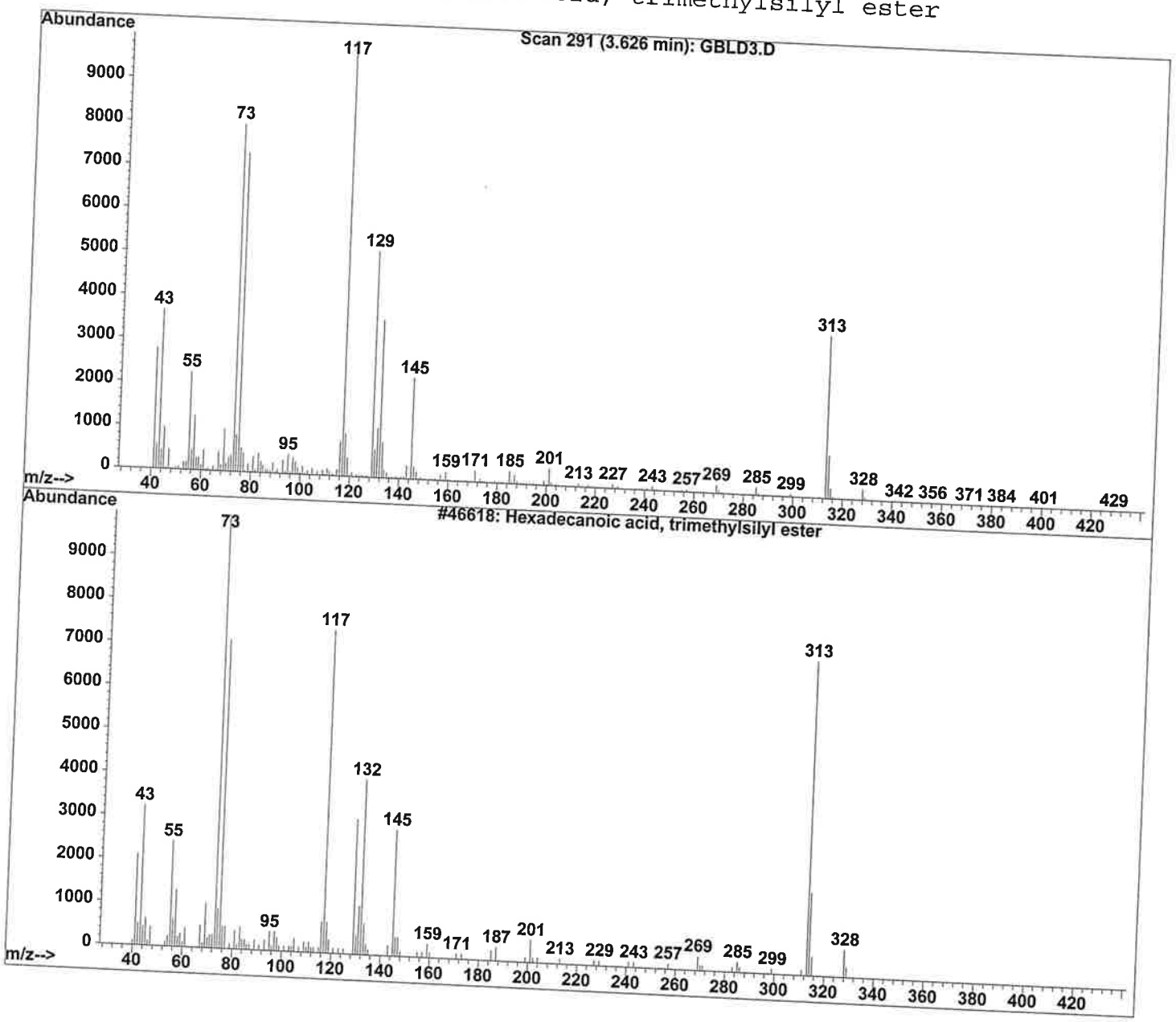
Library Searched : C:\DATABASE\NBS75K.L
Quality : 52
ID : L-Proline, 5-oxo-1-(trimethylsilyl)-, trimethylsilyl ester



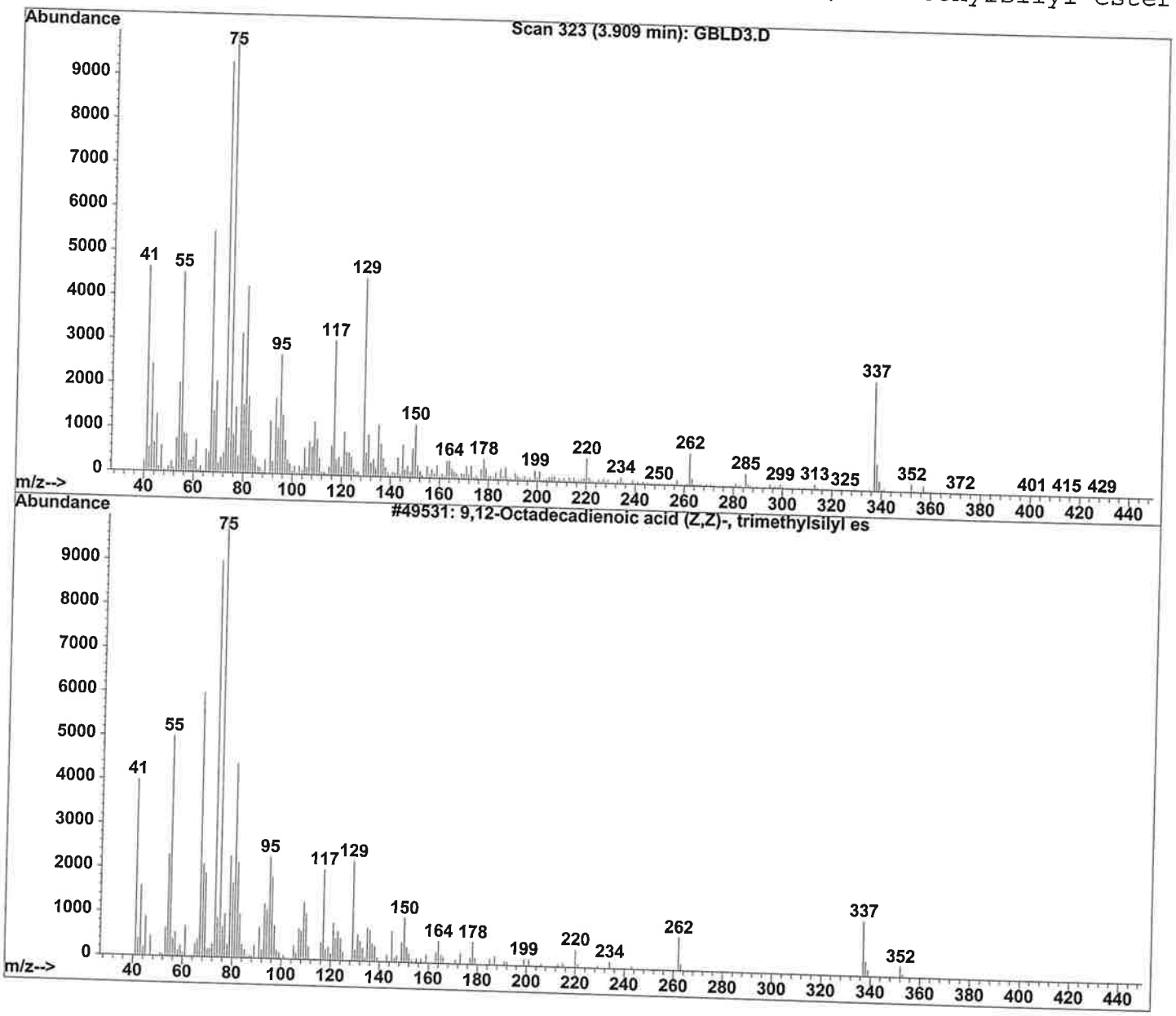
Library Searched : C:\DATABASE\NBS75K.L
Quality : 99
ID : 1,4-Benzenedicarboxylic acid, bis(trimethylsilyl) este
r



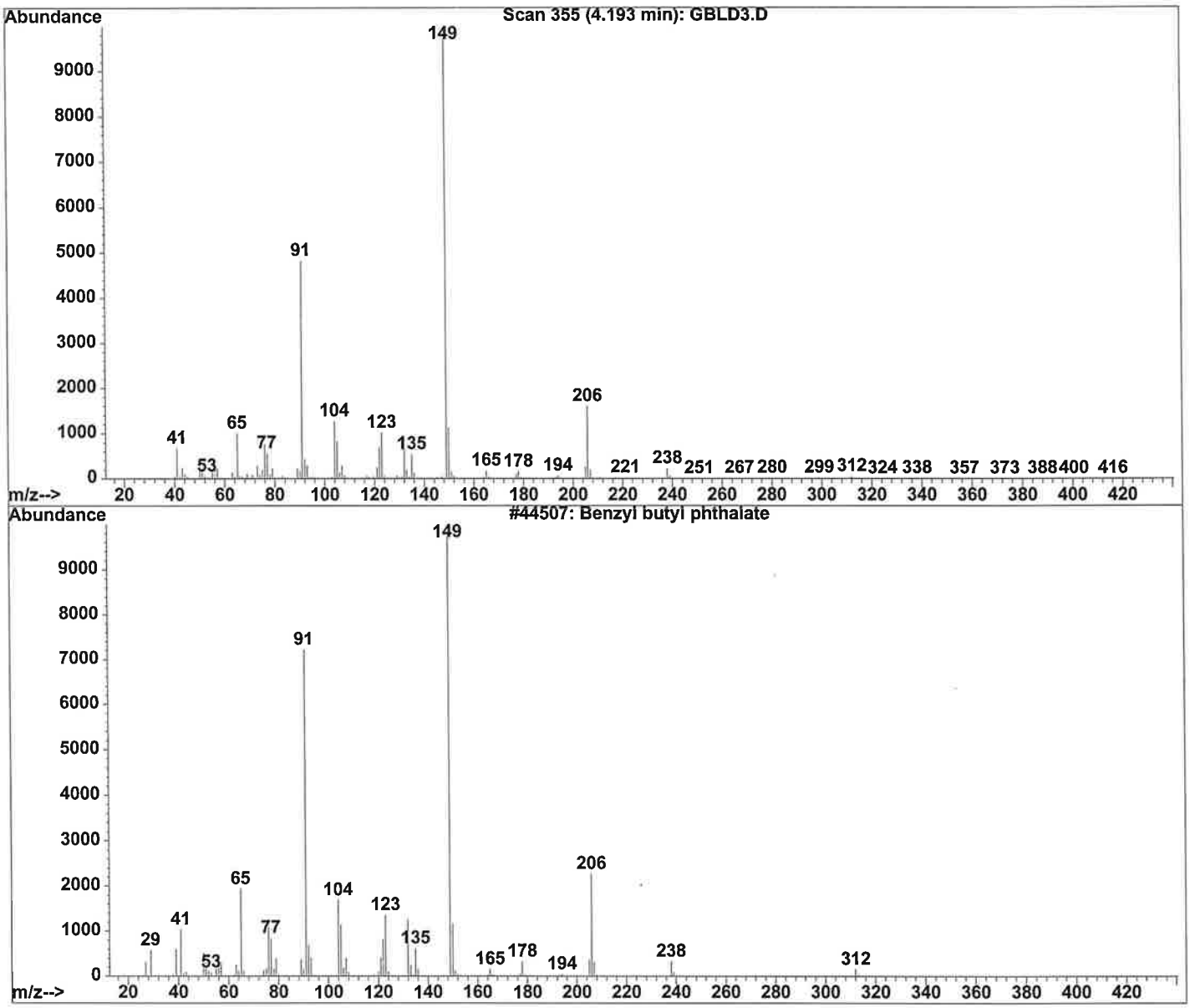
Library Searched : C:\DATABASE\NBS75K.L
Quality : 83
ID : Hexadecanoic acid, trimethylsilyl ester



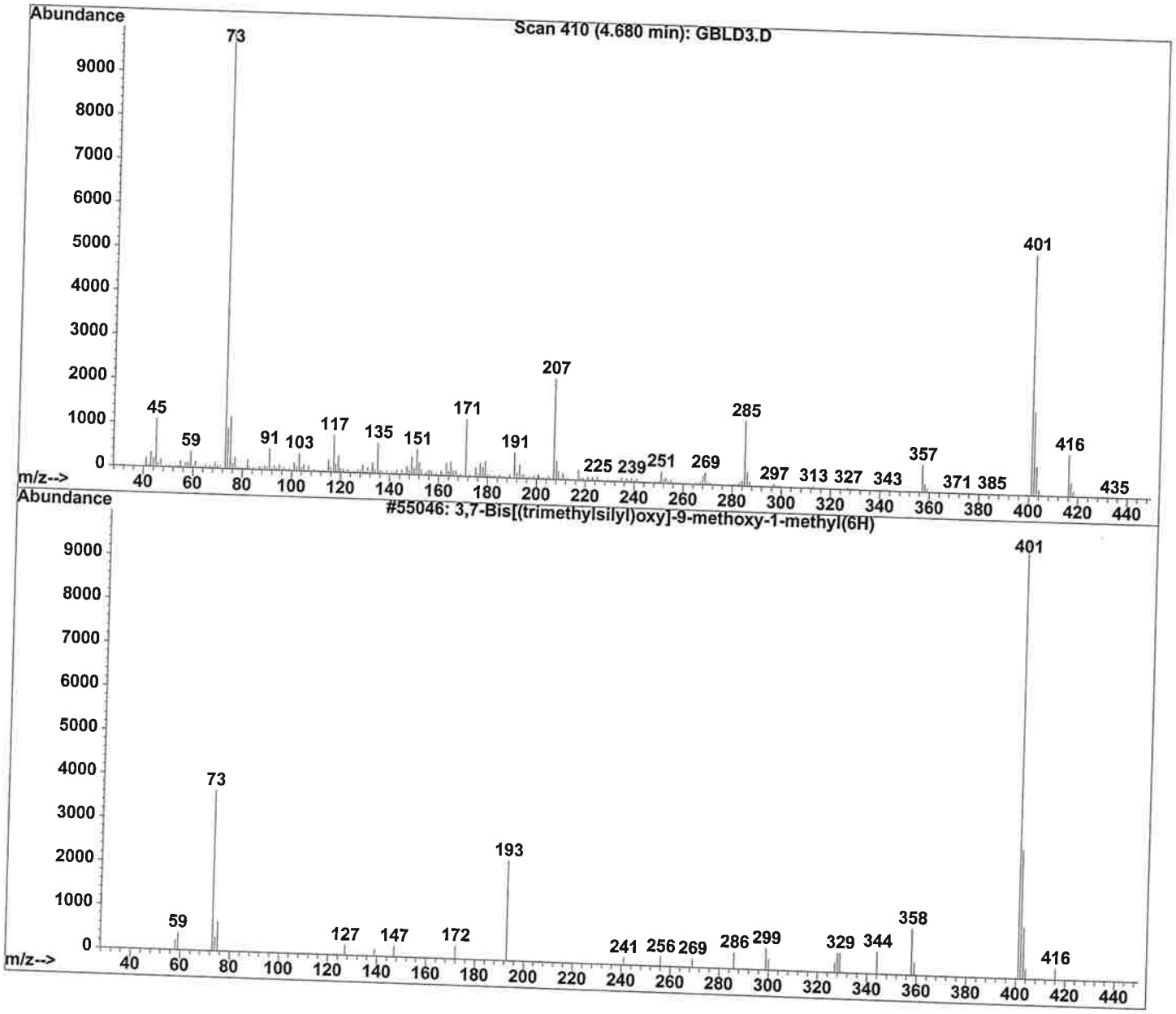
Library Searched : C:\DATABASE\NBS75K.L
Quality : 95
ID : 9,12-Octadecadienoic acid (Z,Z)-, trimethylsilyl ester



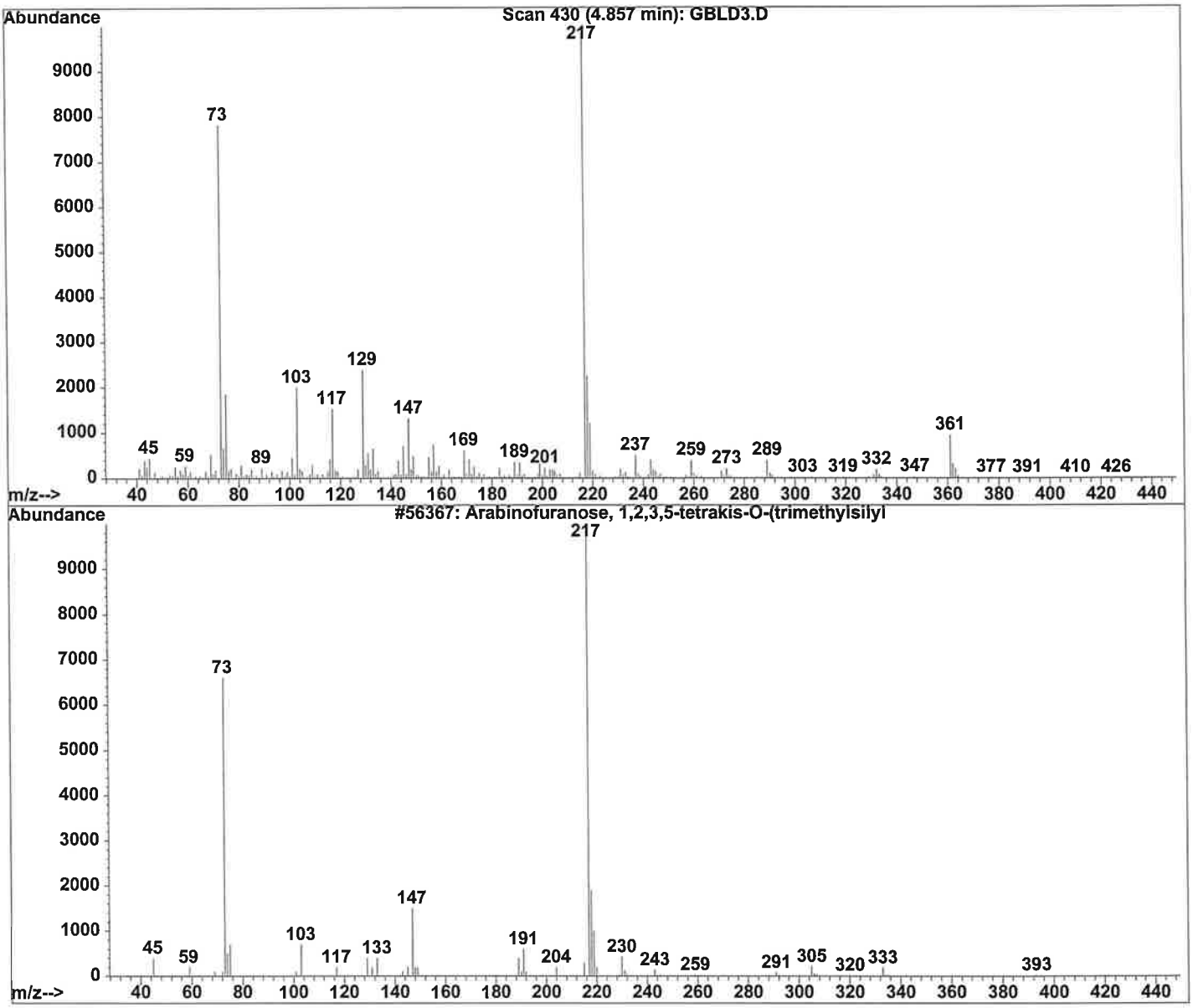
Library Searched : C:\DATABASE\NBS75K.L
Quality : 70
ID : Benzyl butyl phthalate



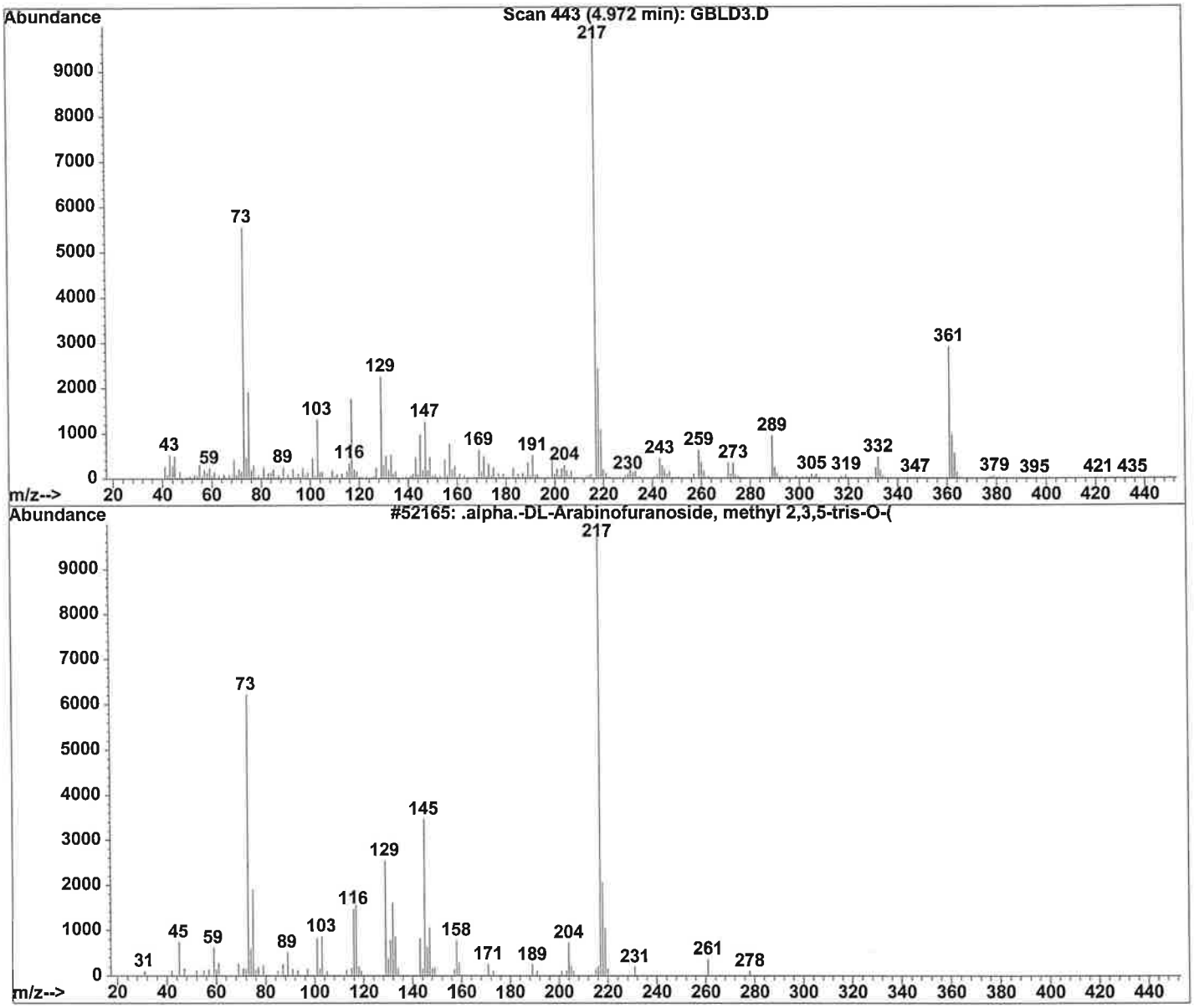
Library Searched : C:\DATABASE\NBS75K.L
Quality : 38
ID : 3,7-Bis[(trimethylsilyl)oxy]-9-methoxy-1-methyl(6H)dibenzo[b,d]pyran-6-one



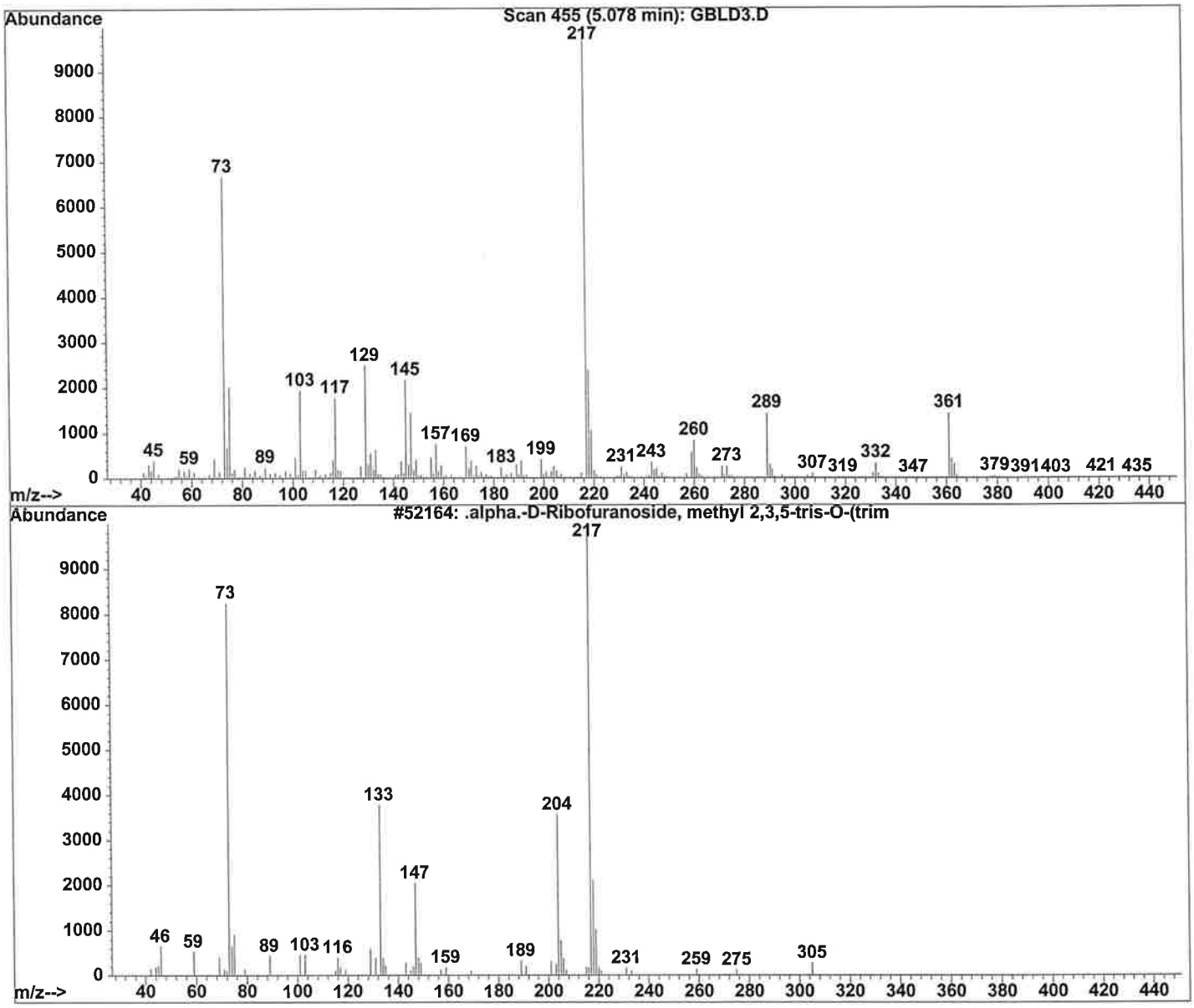
Library Searched : C:\DATABASE\NBS75K.L
Quality : 53
ID : Arabinofuranose, 1,2,3,5-tetrakis-O-(trimethylsilyl)-



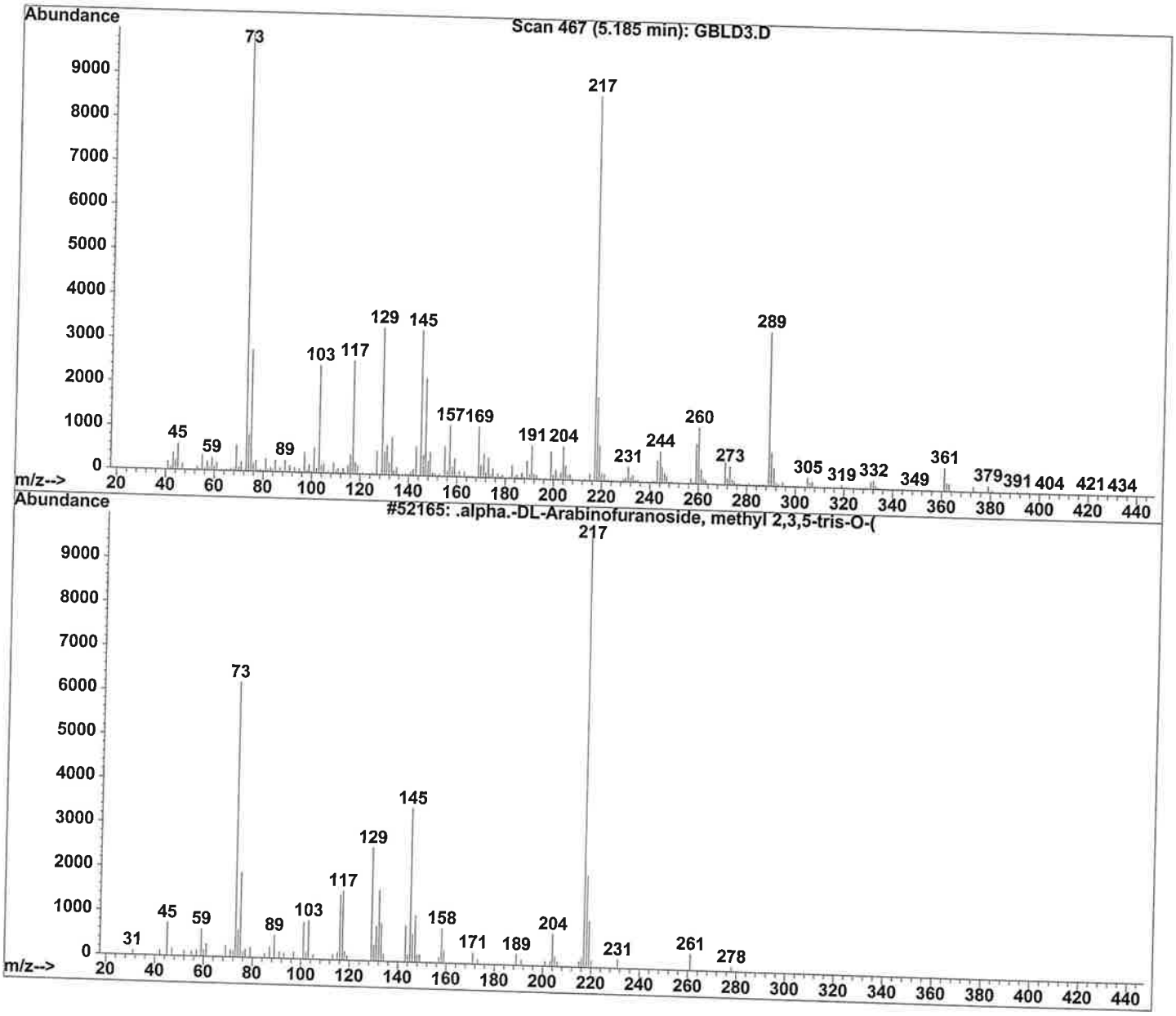
Library Searched : C:\DATABASE\NBS75K.L
Quality : 47
ID : .alpha.-DL-Arabinofuranoside, methyl 2,3,5-tris-O-(trimethylsilyl)-



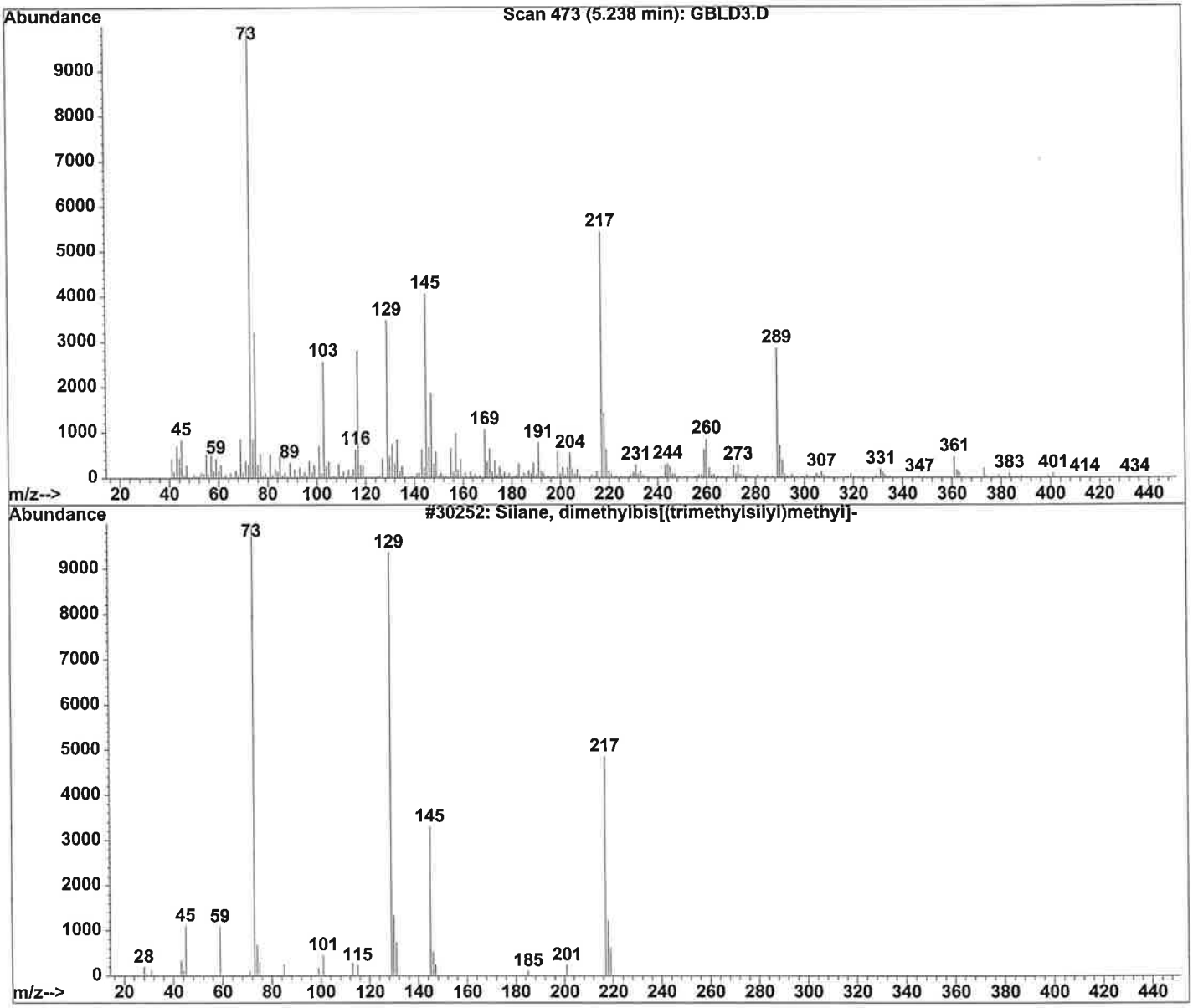
Library Searched : C:\DATABASE\NBS75K.L
Quality : 43
ID : .alpha.-D-Ribofuranoside, methyl 2,3,5-tris-O-(trimethylsilyl)-



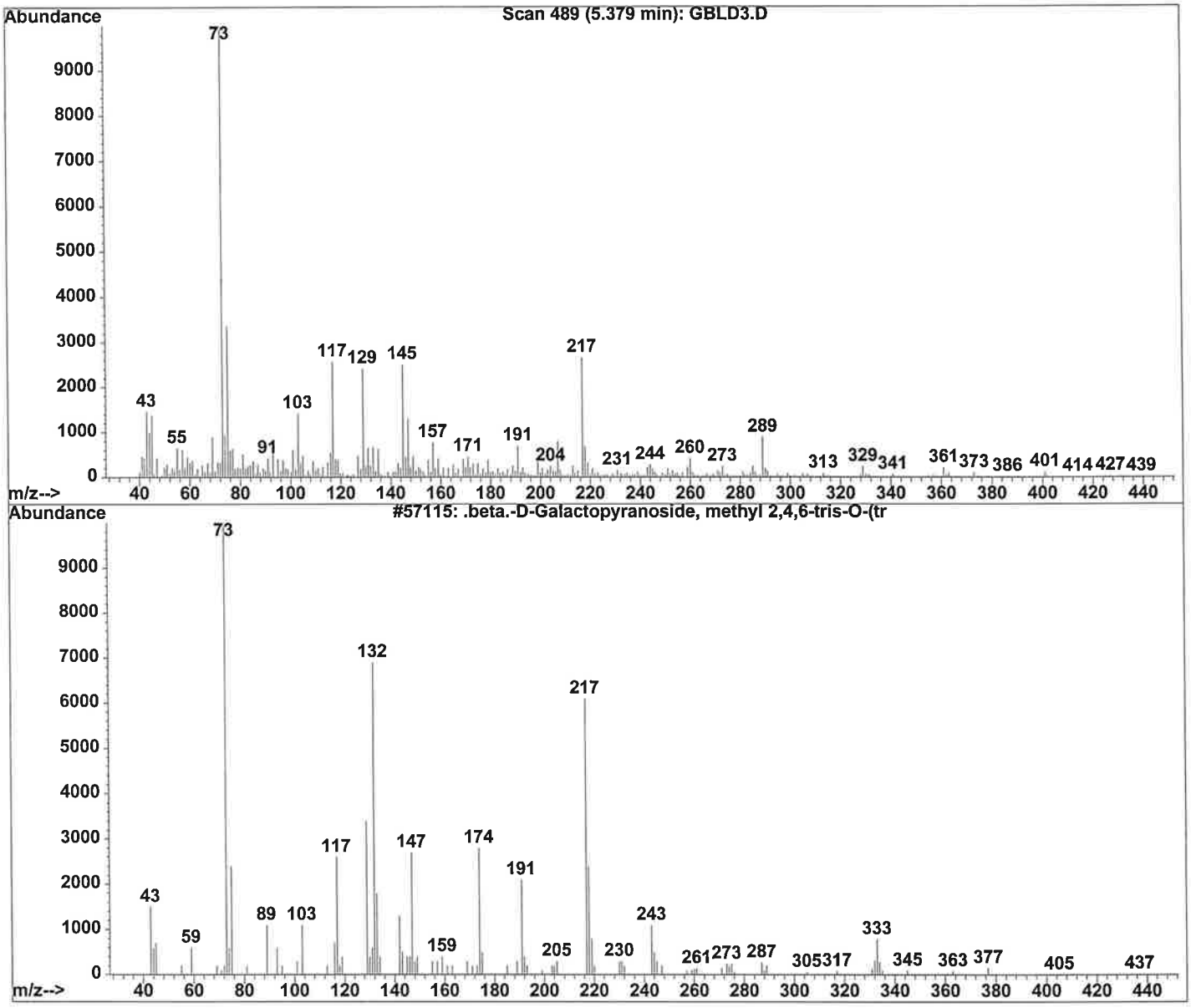
Library Searched : C:\DATABASE\NBS75K.L
Quality : 52
ID : .alpha.-DL-Arabinofuranoside, methyl 2,3,5-tris-O-(trimethylsilyl)-



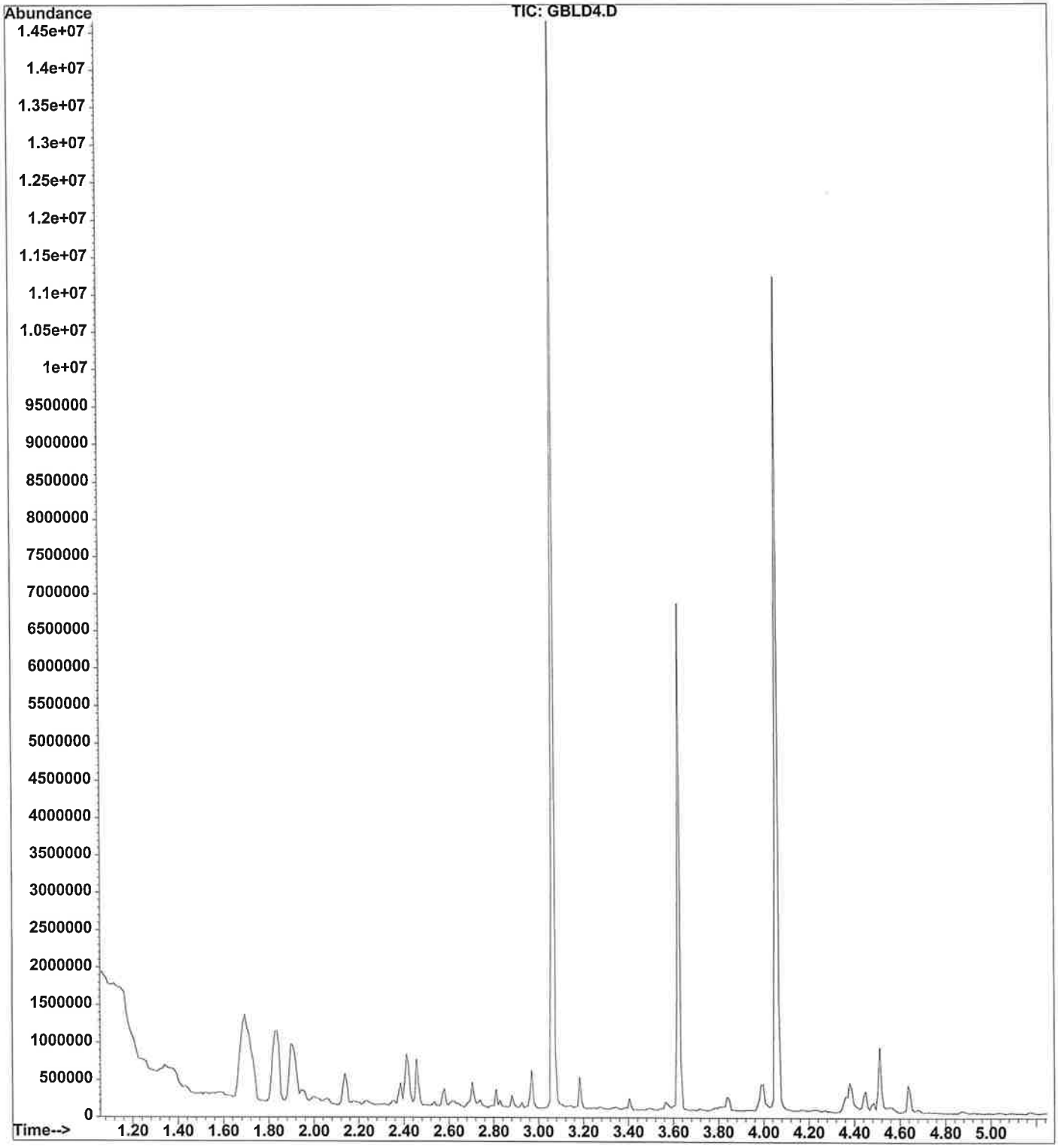
Library Searched : C:\DATABASE\NBS75K.L
Quality : 43
ID : Silane, dimethylbis[(trimethylsilyl)methyl]-



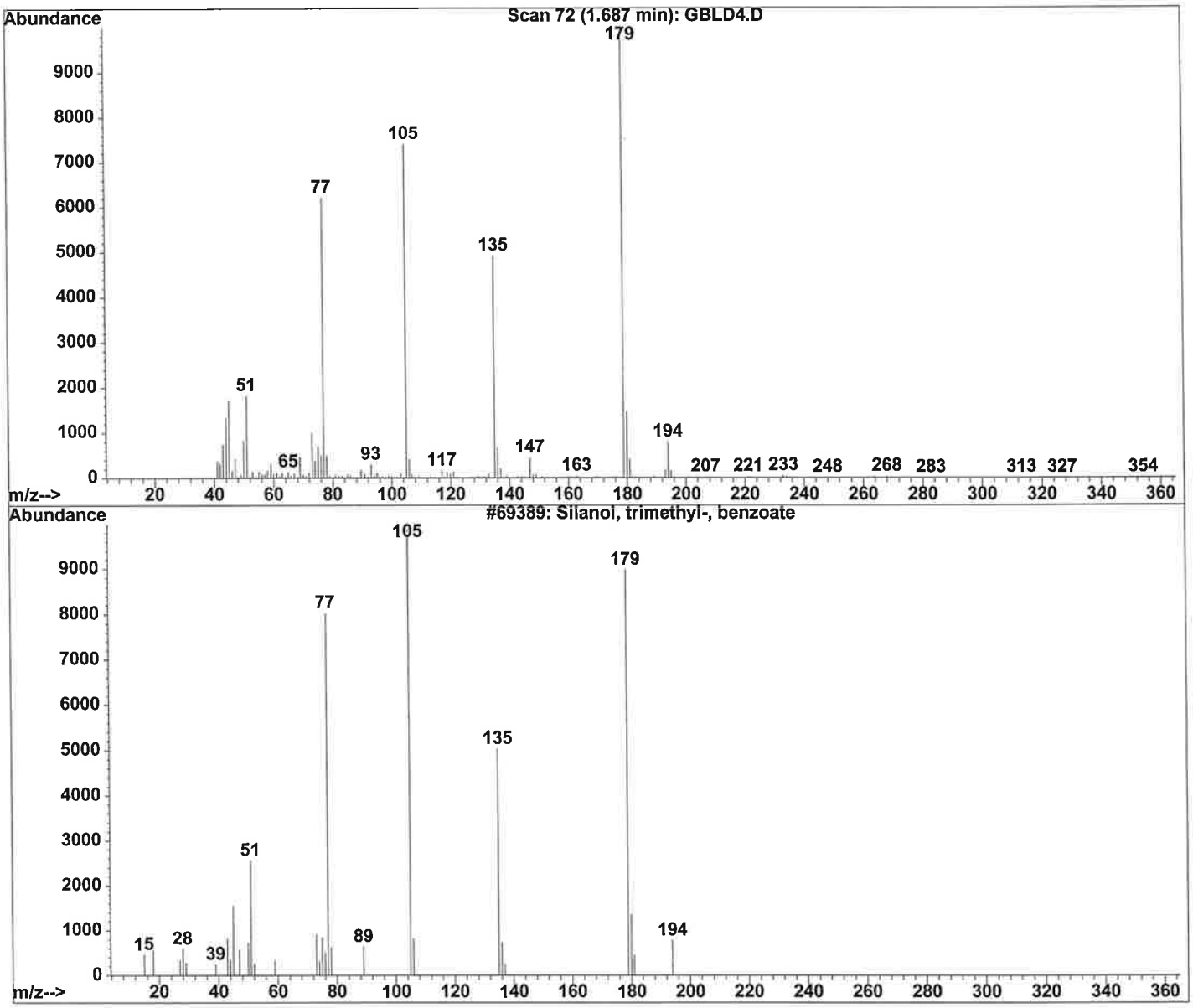
Library Searched : C:\DATABASE\NBS75K.L
Quality : 43
ID : .beta.-D-Galactopyranoside, methyl 2,4,6-tris-O-(trimethylsilyl)-, acetate



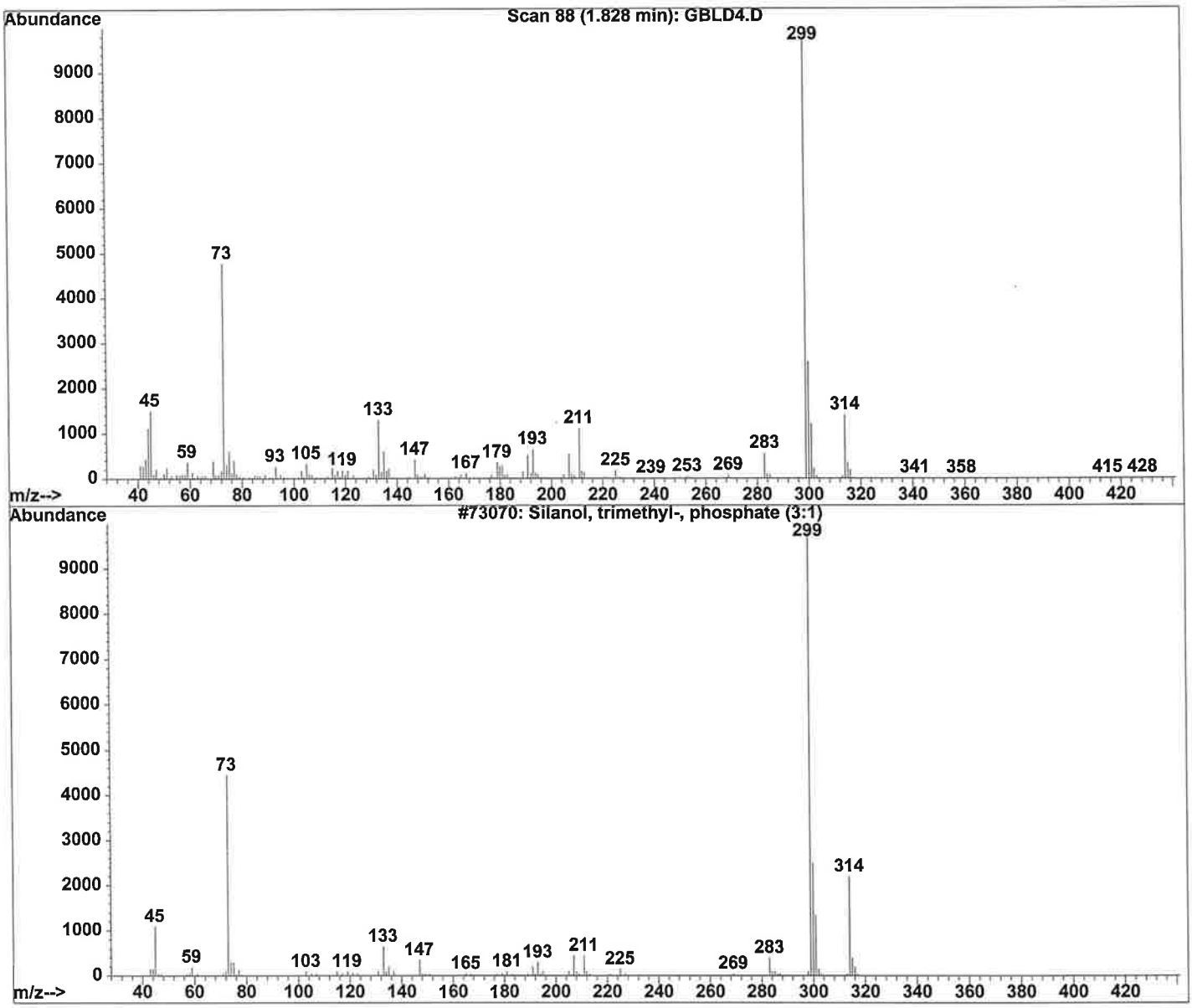
File : C:\HPCHEM\1\DATA\PWF\GBLD4.D
Operator : PWF
Acquired : 29 Sep 1999 12:24 using AcqMethod MSDRUGS
Instrument : DONNA
Sample Name: STD GBL NEAT WITH DER AGENTS
Misc Info :
Vial Number: 1



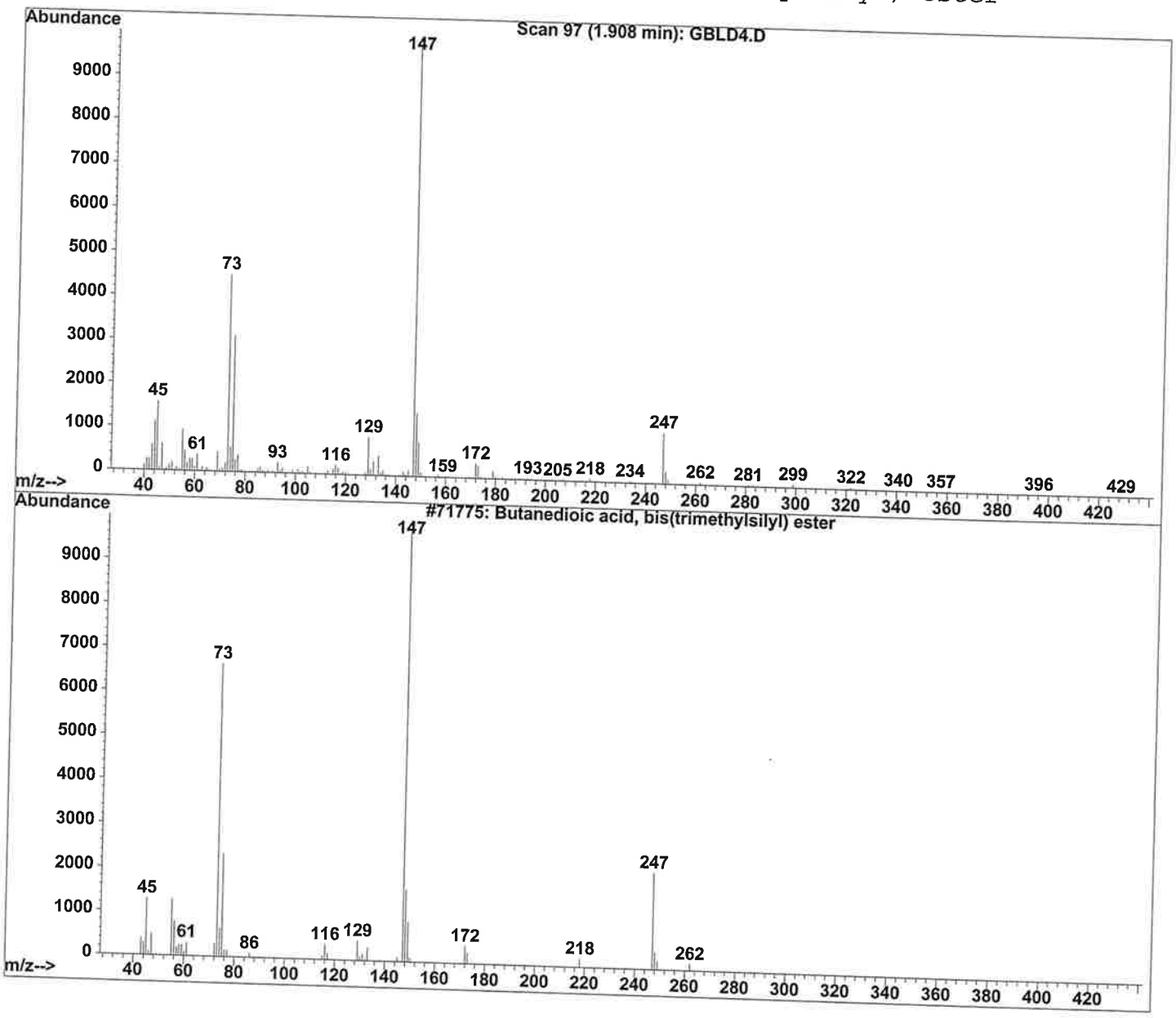
Library Searched : C:\DATABASE\NBS75K.L
Quality : 94
ID : Silanol, trimethyl-, benzoate



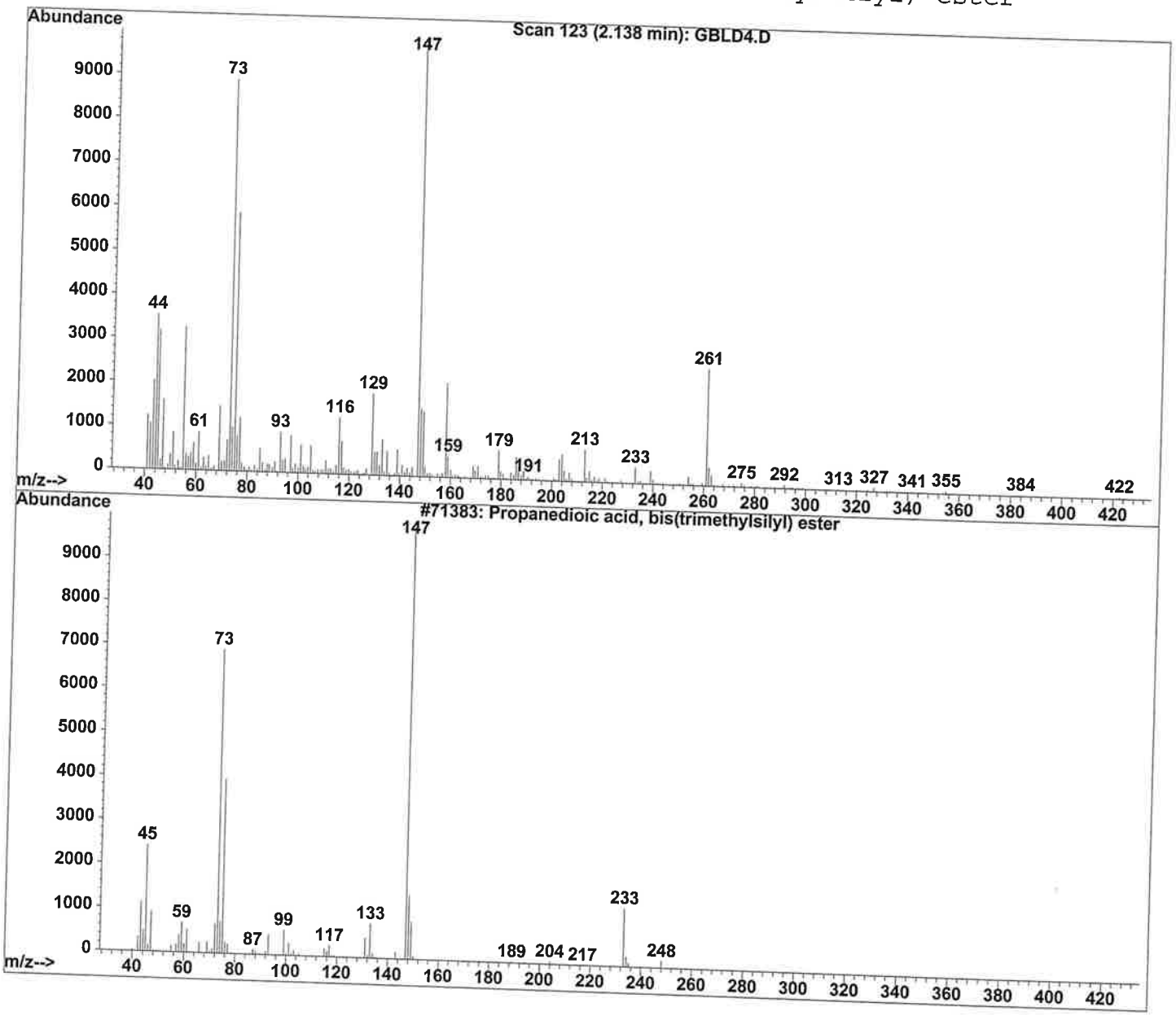
Library Searched : C:\DATABASE\NBS75K.L
Quality : 97
ID : Silanol, trimethyl-, phosphate (3:1)



Library Searched : C:\DATABASE\NBS75K.L
Quality : 90
ID : Butanedioic acid, bis(trimethylsilyl) ester



Library Searched : C:\DATABASE\NBS75K.L
Quality : 64
ID : Propanedioic acid, bis(trimethylsilyl) ester



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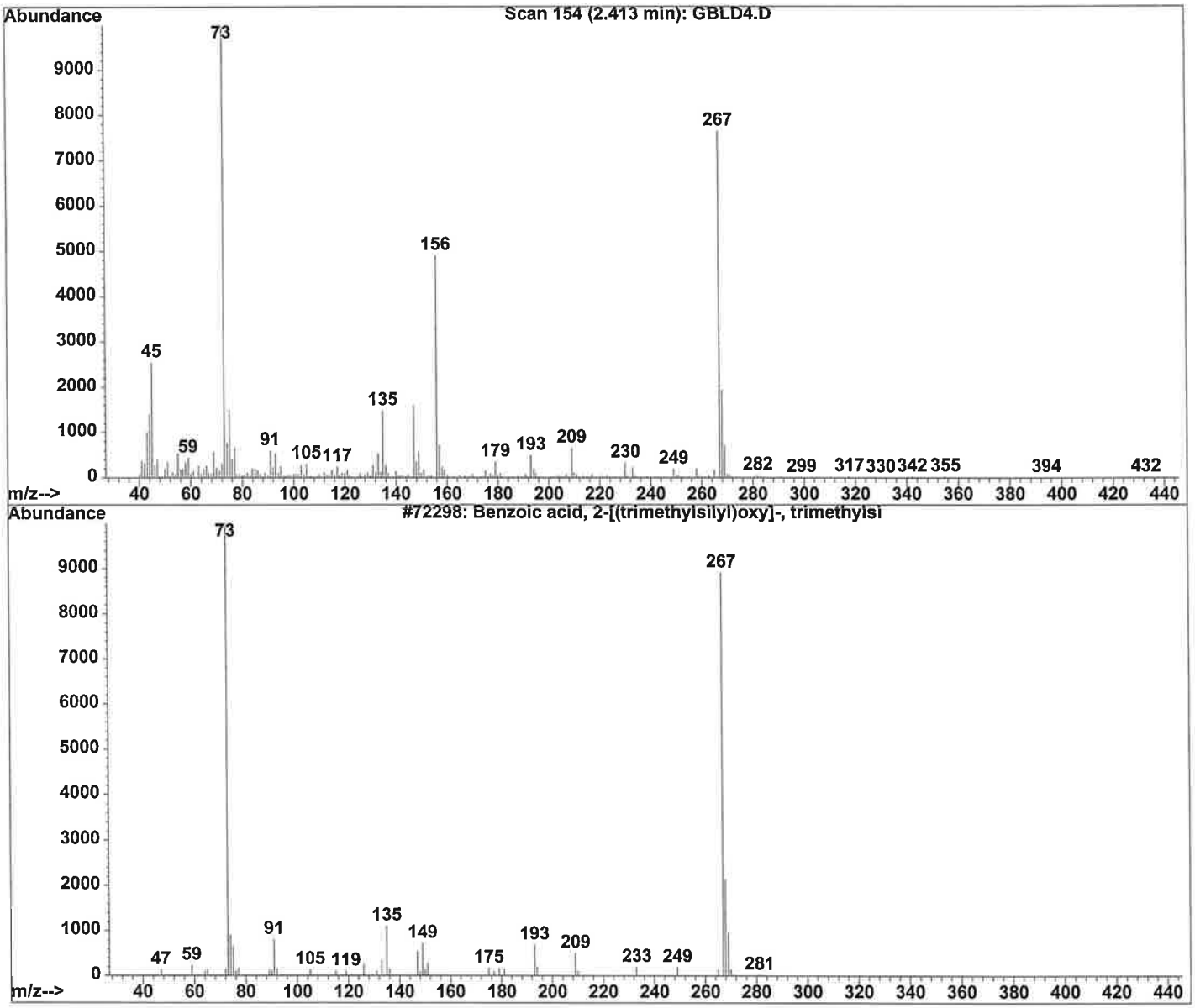
Quality : 76
ID : Benzoic acid, 2-[(trimethylsilyl)oxy]-, trimethylsilyl ester



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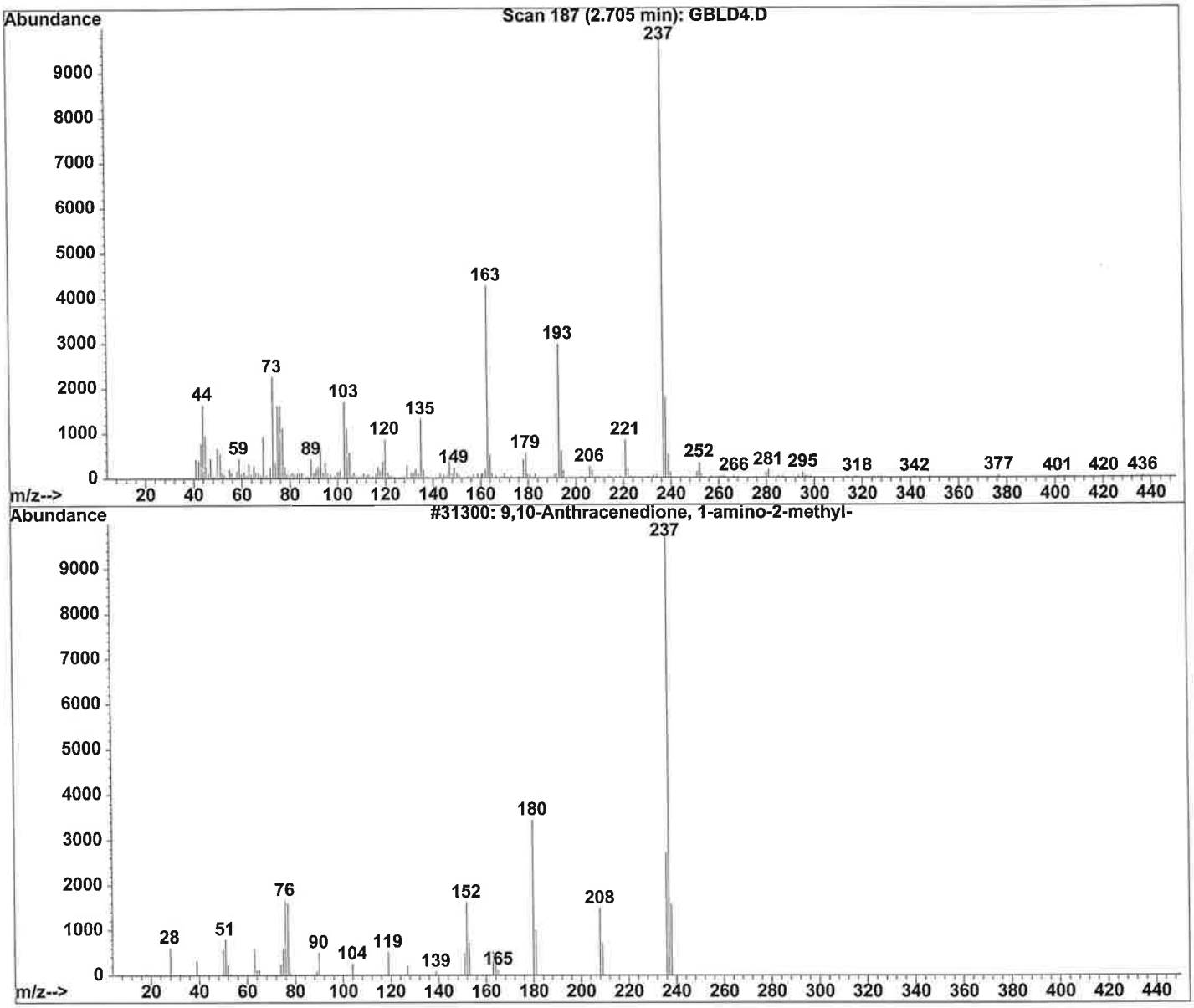
Quality : 35
ID : 9,10-Anthracenedione, 1-amino-2-methyl-



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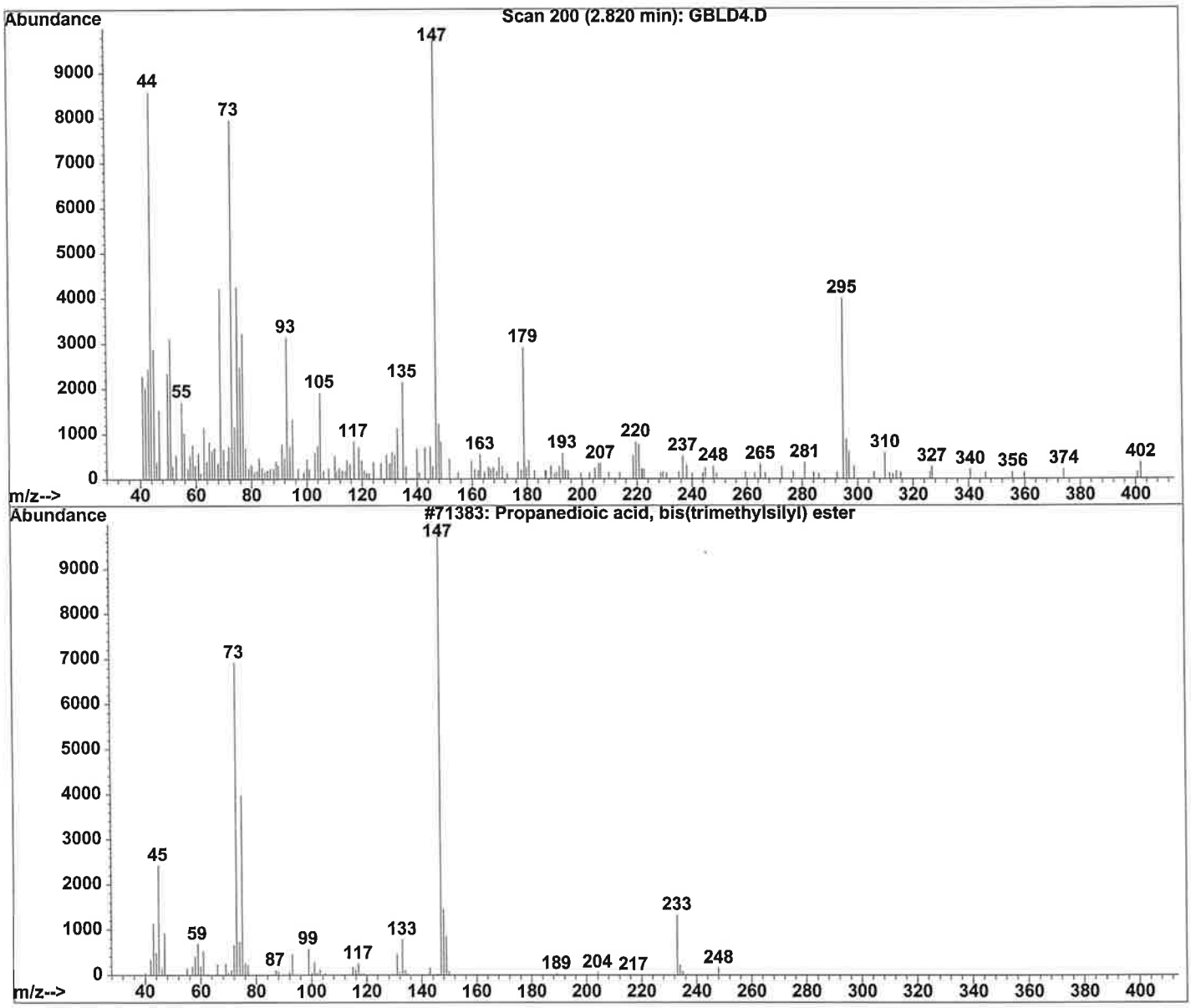
Quality : 66
ID : Propanedioic acid, bis(trimethylsilyl) ester



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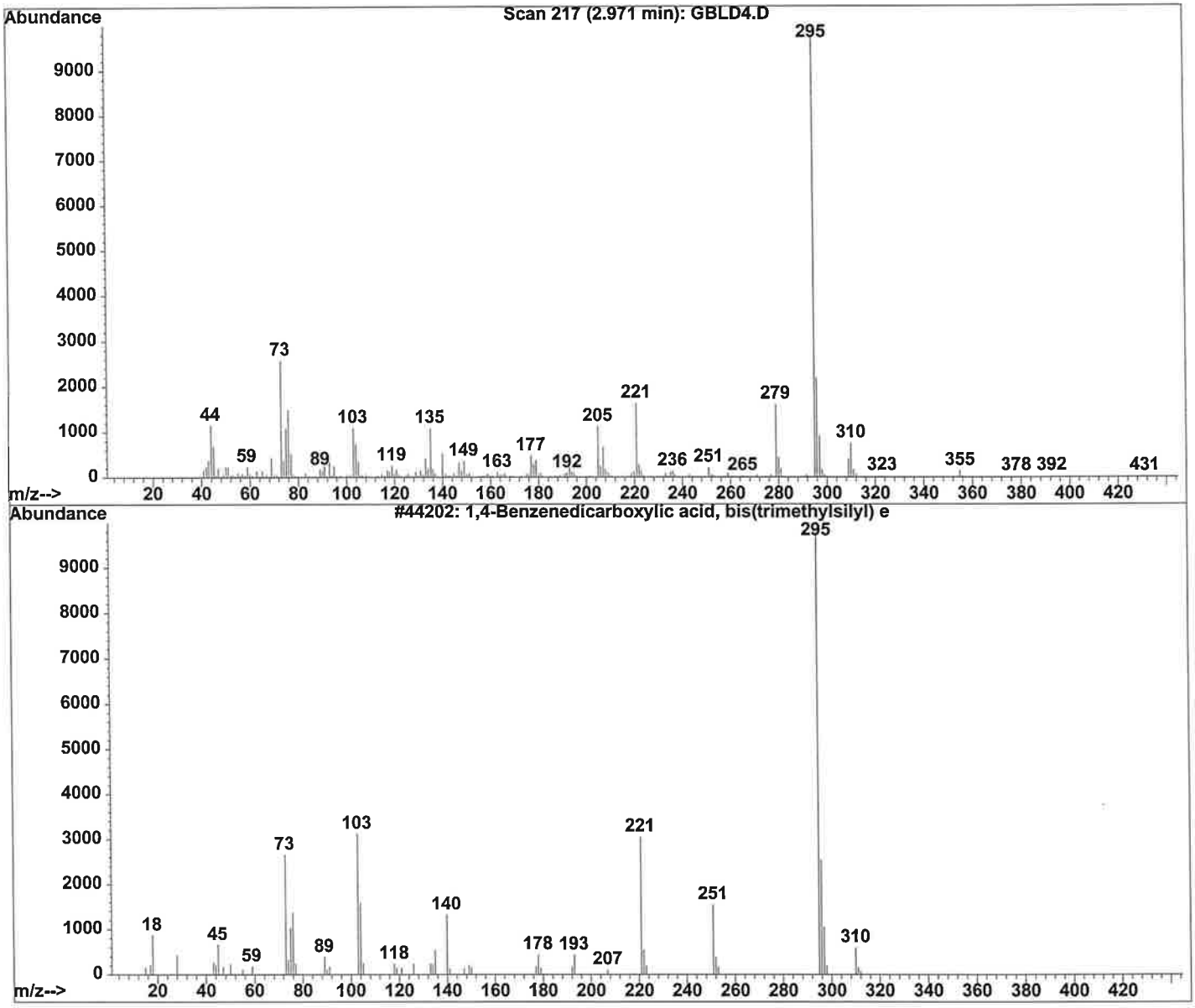
Quality : 72
ID : 1,4-Benzenedicarboxylic acid, bis(trimethylsilyl) este
r



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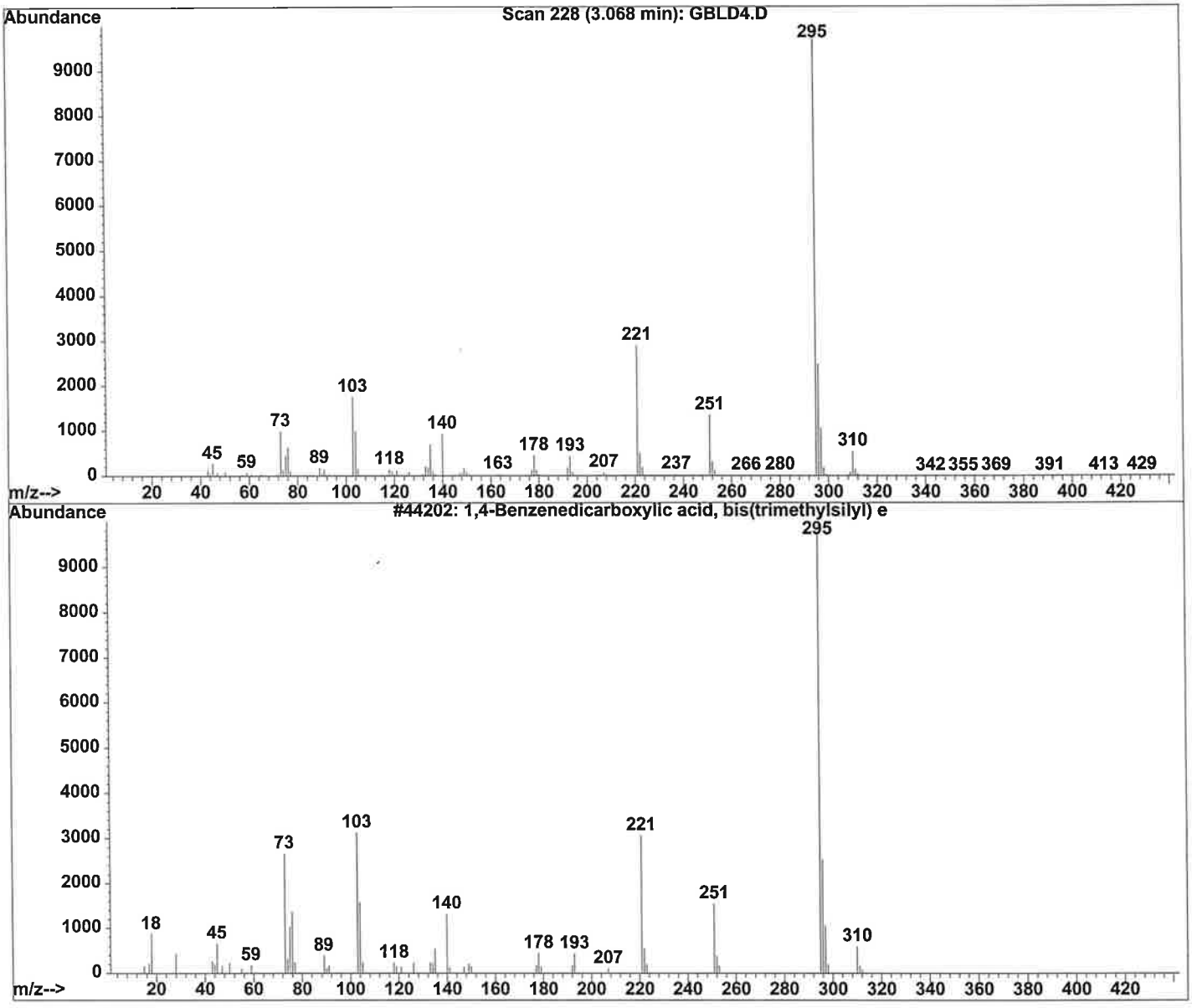
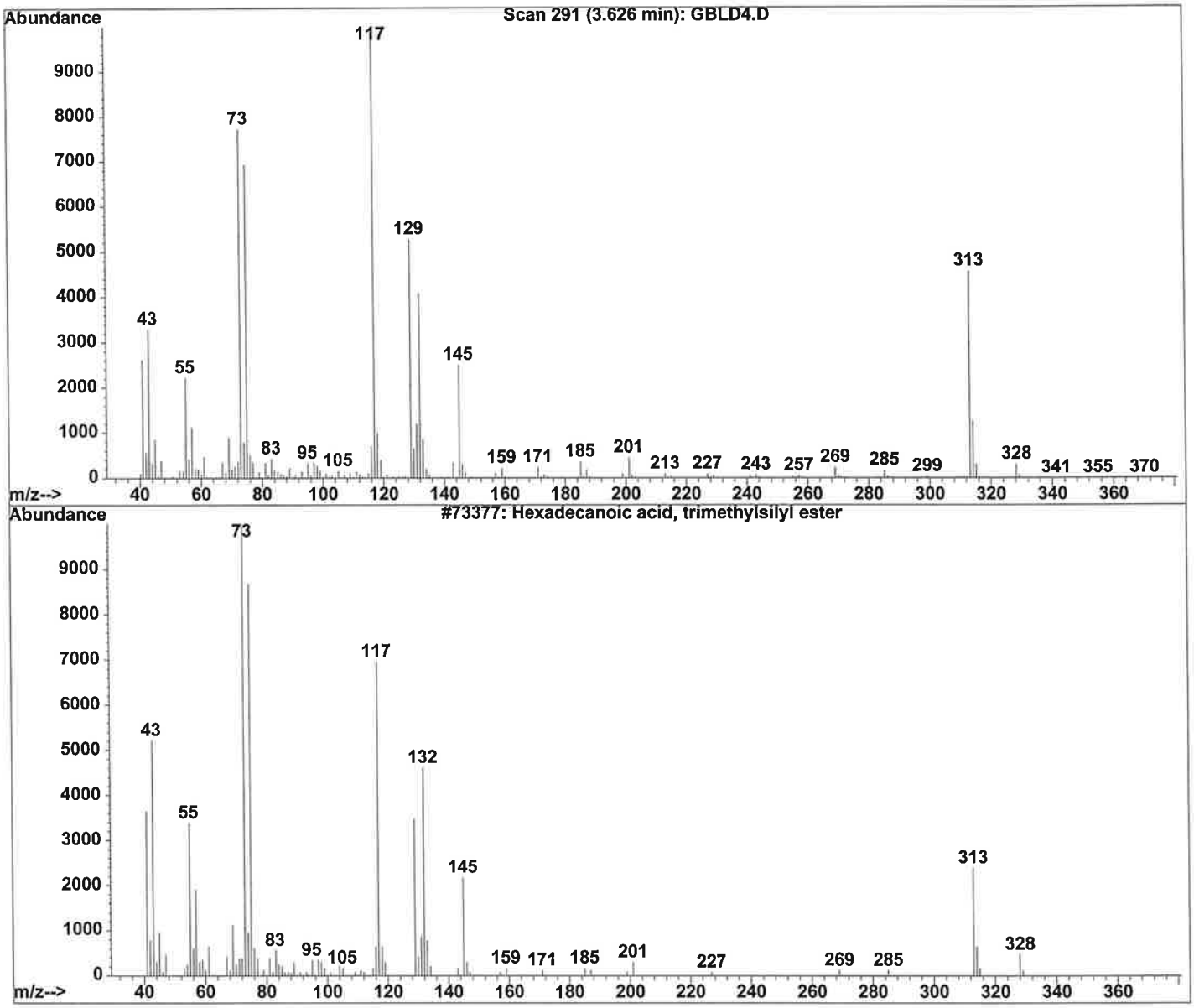
Quality : 98
ID : 1,4-Benzenedicarboxylic acid, bis(trimethylsilyl) este
r



Library Searched : C:\DATABASE\NBS75K.L
Quality : 90
ID : Hexadecanoic acid, trimethylsilyl ester



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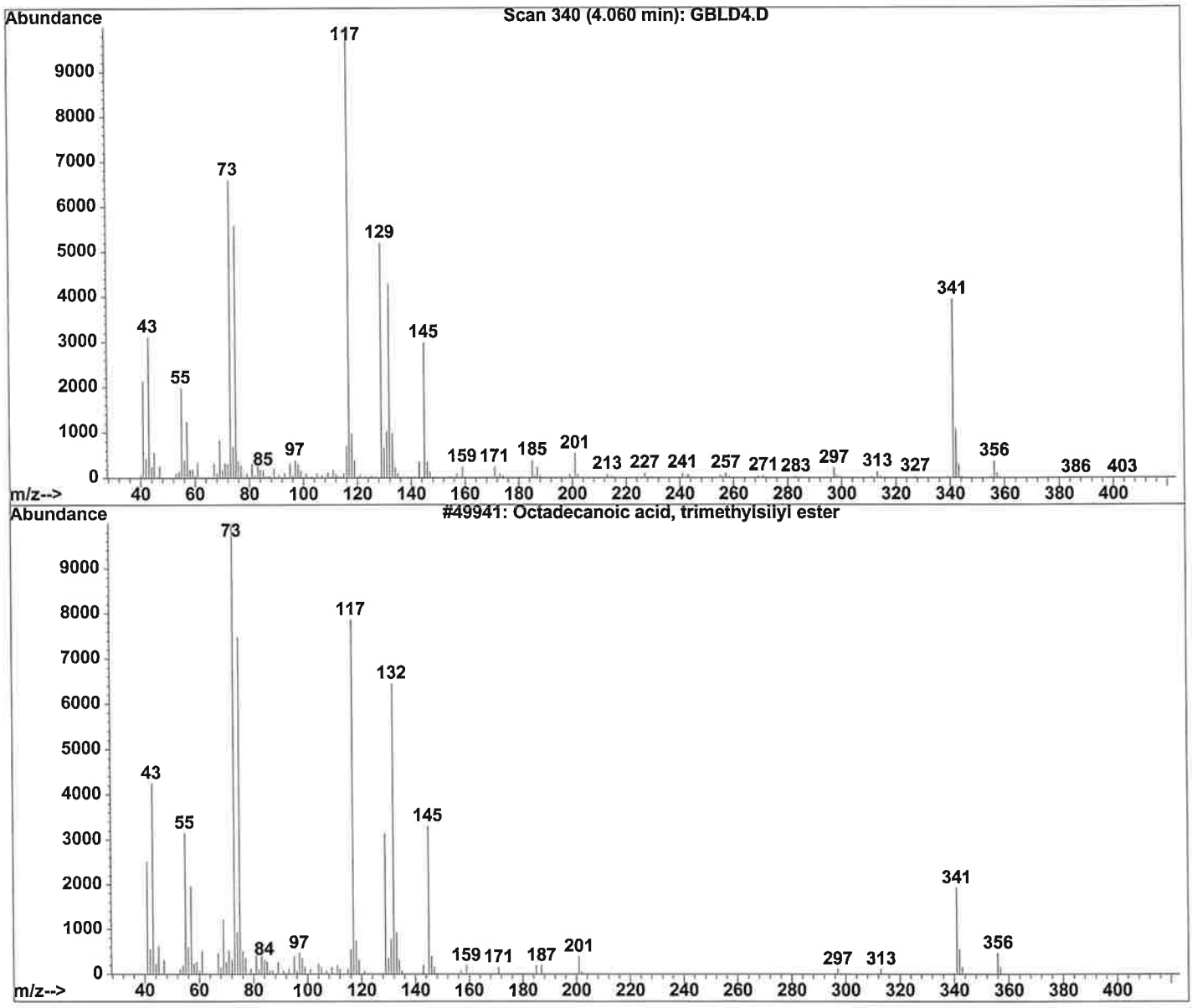
Quality : 98
ID : Octadecanoic acid, trimethylsilyl ester



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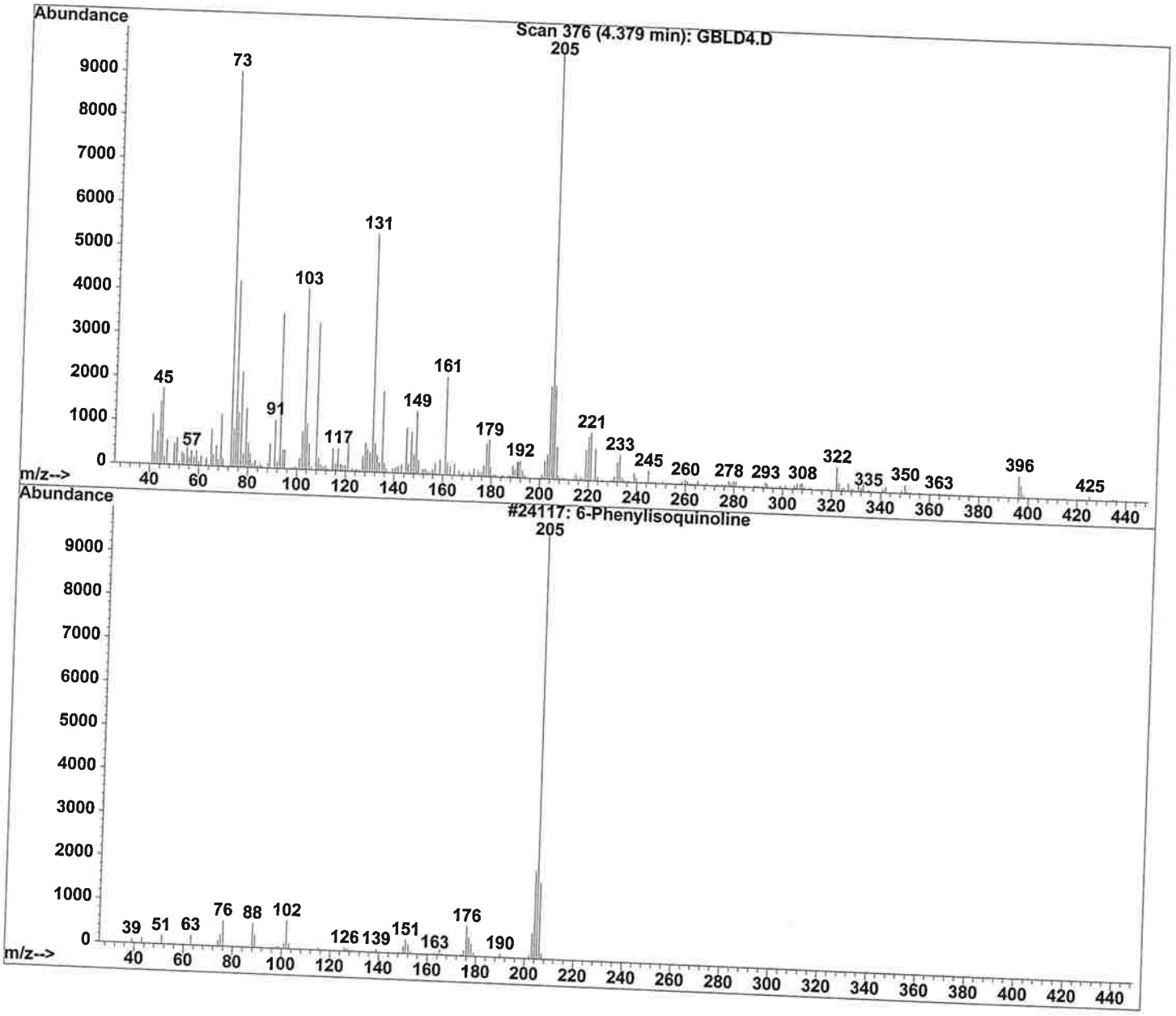
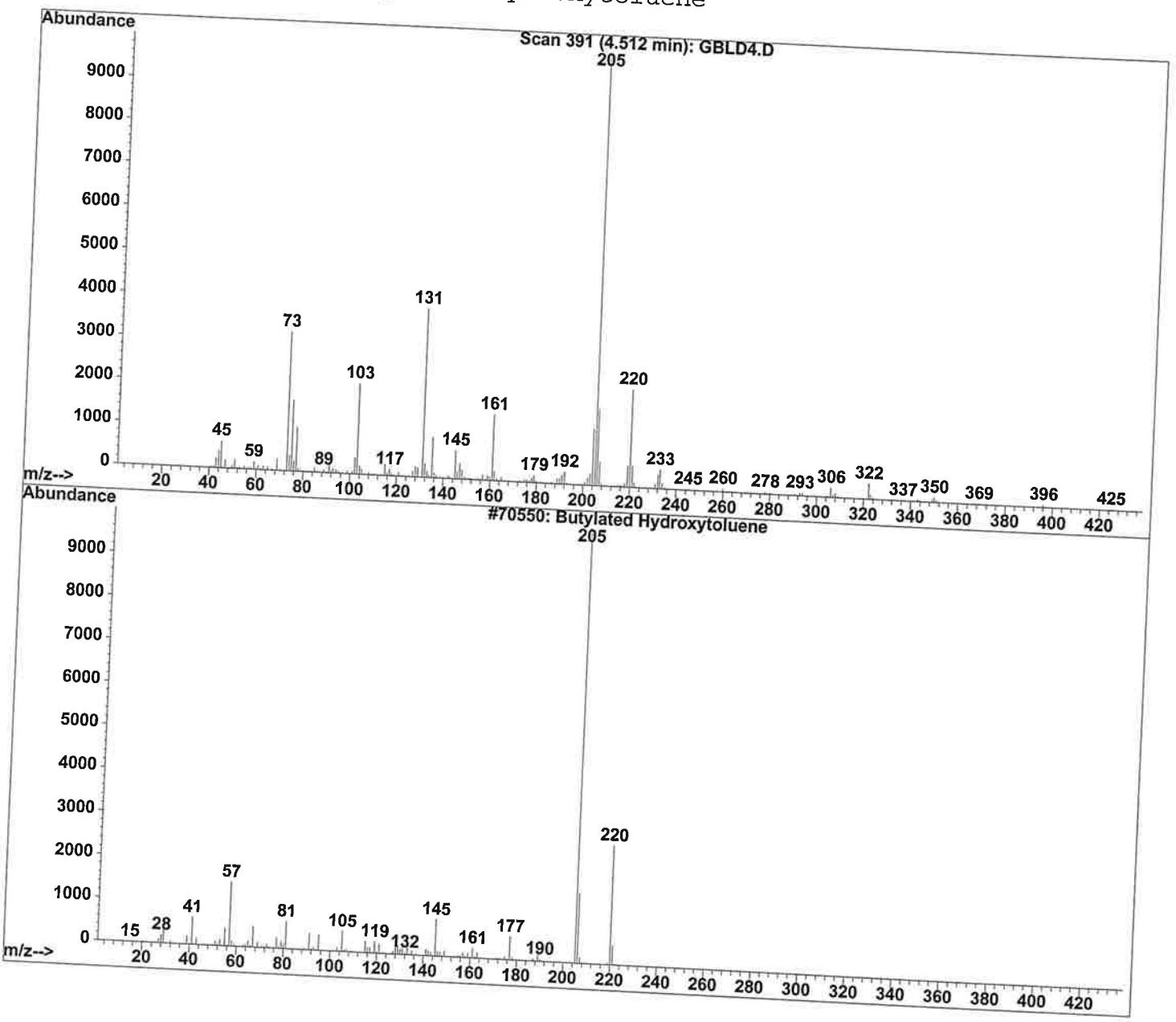
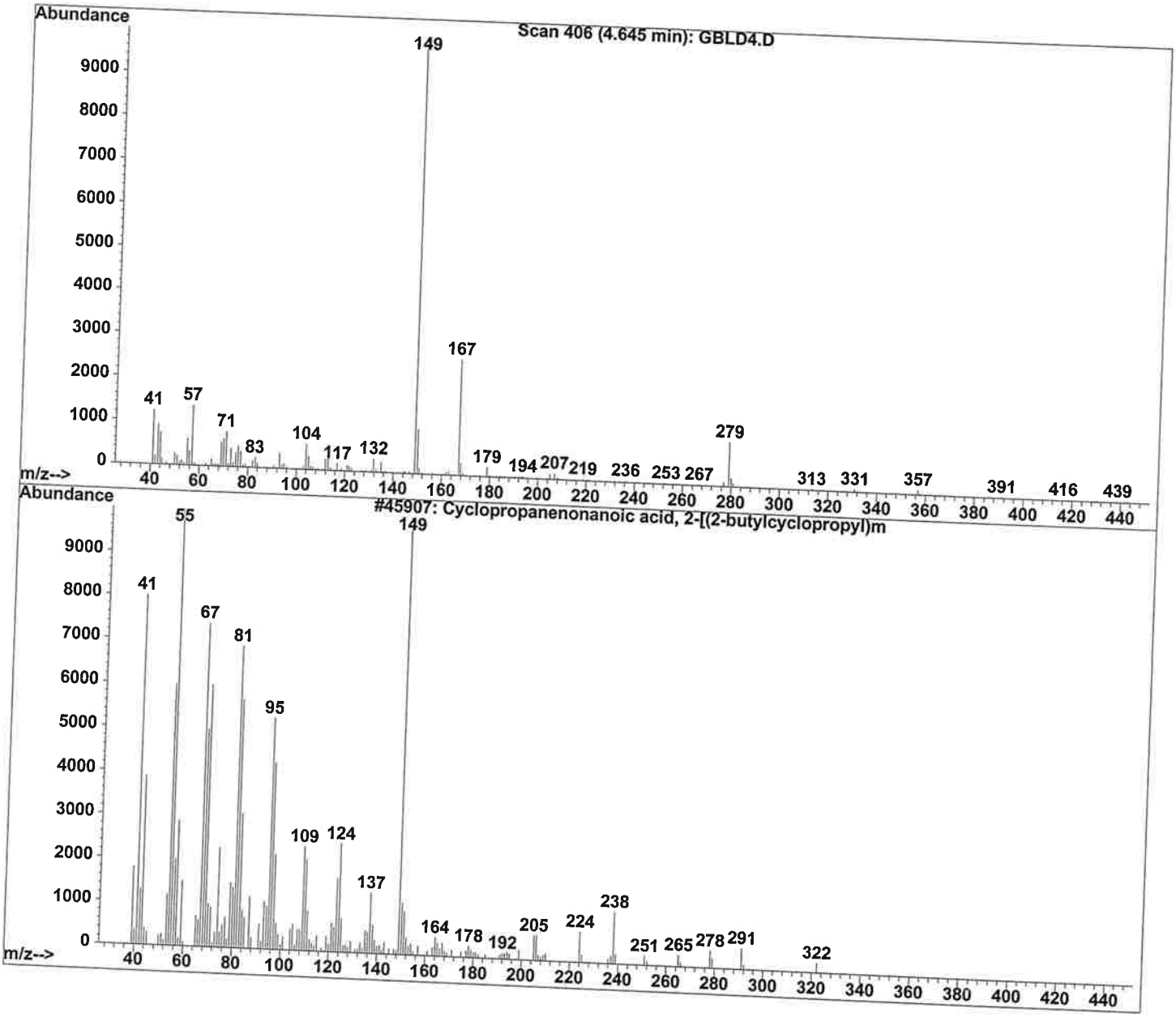
Quality : 27
ID : 6-Phenylisoquinoline



Library Searched : C:\DATABASE\NBS75K.L
Quality : 45
ID : Butylated Hydroxytoluene



Library Searched : C:\DATABASE\NBS75K.L
Quality : 95
ID : Cyclopropanenonanoic acid, 2-[(2-butylcyclopropyl)methyl]-, methyl ester



Library Searched : C:\DATABASE\NBS75K.L
Quality : 64
ID : Propanedioic acid, bis(trimethylsilyl) ester