



ISO 17025



Testing Cert. #2797.01

**GAS CHROMATOGRAPHY MASS SPECTROMETRY (GC-MS)
ANALYSIS REPORT
21 Jul 2009**

**JOB NUMBER C09Y8961
PO NUMBER Free**

for

Dwain Daniel
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GC-MS ANALYSIS REPORT

Requester: Dwain Daniel
Job Number: C09Y8961
Analysis Date: 14 Jul 2009

Purpose:

The purpose of the analysis was to determine the chemical composition of the vapor/fog that an electronic cigarette generated.

Summary:

Propylene glycol and nicotine were the major components detected from the vapor/fog of a lighted cigarette.

Experimental:

Inhalation/suction

A charcoal tube was attached on to an [air sampling pump](#). The [electronic cigarette](#) is connected with the charcoal tube, as shown in the pictures below. When the sampling pump starts to run, the [cigarette lights](#) and produces vapor/fog, a similar situation as the cigarette is being sucked. The vapor is absorbed by the charcoal tube. After a few minutes, the charcoal tube is disconnected from the sampling pump.

The charcoal tube was cut and the charcoal absorber from the tube was collected in a vial. A 1 ml aliquot of carbon disulfide was added into the vial and sonicated for 20 minutes to extract any organics contained on the charcoal. The carbon disulfide extract was separated from the charcoal and evaporated to dryness under a stream of dry nitrogen without heating to avoid losing any volatile components. A 100 µl aliquot of methylene chloride was added into the extract and 2 µl was injected into GC directly. A second injection was made from a slightly concentrated extract. A blank charcoal tube was processed under the sample condition and analyzed as a reference blank.

Outgassing

The liquid inside the cartridge and the whole set of the electronic cigarette were each placed in one of the 1½" diameter chambers of the dynamic headspace sampling system (CDS 6500 bulk auto-sampler). Prior to sample analysis, a system blank was analyzed. All samples were analyzed on a Hewlett-Packard 6890 Gas Chromatograph/ Hewlett-Packard 5973 Mass Spectrometer.

GC Column: 30m X 0.25mm DB-5MS (J&W scientific) 0.25 μ film thickness
Flow rate: 1.0 ml/min, constant flow mode
Detector: Mass Selective Detector (MSD)
Injection Mode: Split injection (10:1 split ratio), thermal desorption from mixed bed trap at 300 °C for 5 minutes
Outgassing Conditions: 1½" Chamber, purge for two hours at 120°C for the liquid, 50 °C for three hours for the whole cigarette, flow 60 ml/min
Oven temperature: 35°C (5 min.) to 150°C @10°C/min.; to 300°C@15°C/min; hold for 5 min. at 300°C.

Results and Discussion:

[Figure 1](#) is a chromatogram of the blank charcoal after methylene chloride baseline subtraction. Nothing was detected from the reference blank.

[Figure 2](#) is a chromatogram of the volatile organic components detected from the extract of the charcoal tube that attached to the electronic cigarette. Several components were detected. Details are tabulated in the table below.

Table 1
Charcoal extract, Y8961-31

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
2-1	3.82	25.82	Propylene glycol	
2-2	7.74	2.41	Propylene carbonate	
2-3	8.59	0.50	Unknown	Possibly similar to di-silane, trimethyl-
2-4	11.40	17.46	Probably adiminothiourea	Or Benzimidazole
2-5	13.40	49.62	Pyridine, methyl-pyrrolidinyl	Nicotine
2-6	14.69	3.66	Pyridine, methyl-pyrrolyl	Or pyrazole, methyl phenyl
2-7	17.31	0.52	Propenoic acid, methyl ethane-diylbis-oxy-ethanediyl ester	

[Figure 3](#) is a chromatogram of the volatile organic species outgassed from the liquid of the cartridge. Propylene glycol and glycerin were the major components outgassed. Details are tabulated in the table below.

Table 2

Liquid in cartridge, Y8961-21

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
3-1	3.31	1.30	Butyl-amine	
3-2	11.35	57.30	Propylene glycol	
3-3	13.33	3.18	Glycerin	
3-4	13.61-14.44	4.31	Pyridine, methyl pyrrolidinyl	Nicotine, mixed with glycerin
3-5	14.50-17.98	29.98	Glycerin	Mixed with nicotine
3-6	18.67	0.10	Pyridine, methyl pyrrolyl	
3-7	19.19	0.03	Similar to nicotine	
3-8	20.17	1.08	Ethanol, oxy-bis-ethane-diyl-oxy-bis	Tetra-ethylene glycol
3-9	23.09	0.07	Caffeine	
3-10	23.90	0.08	Hexadecanoic acid	

[Figure 4](#) is a chromatogram of the volatile organic species outgassed from the whole cigarette set. Propylene glycol was the major component outgassed. Details are tabulated in the table below.

Table 3
 Cigarette, Y8961-24

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
4-1	4.20	19.14	Similar to methyl-oxiranyl-methanol	
4-2	10.46	49.71	Propylene glycol	
4-3	10.89	2.55	Cyclotetrasiloxane, octamethyl-	Possibly from the cartridge
4-4	12.25	6.40	Unknown	Possibly similar to propenoic acid, methyl methylethyl ester
4-5	13.53	2.21	Cyclopentasiloxane, decamethyl-	Possibly from the cartridge
4-6	15.74	0.77	Hydrocarbon	
4-7	16.04	1.43	Cyclohexasiloxane, dodecamethyl-	Possibly from the cartridge
4-8	17.40	3.85	Biphenyl	
4-9	18.62	0.54	Hydrocarbon	
4-10	19.99	0.21	Hexadecane	

Compound identities were determined with the help of the NIST'08 mass spectral databases. NIST'08 contains 192,000 compounds. The spectra provided at the end of the report include two or three parts. The top spectrum is the actual mass spectrum of a component detected from the analysis. The middle spectrum is a reference spectrum from a NIST library that is similar to the component mass spectrum. The bottom part is the possible structure of the reference spectrum, if the structure is available in NIST'08 library. Isomers of many organic species produce very similar mass spectra.

The reference spectrum provided may not necessarily be the "true" match of the component detected. As the molecular weight increases (with increasing retention time), or if the concentration of a component is too low, the likelihood of getting a good library match decreases. In this case, the uncertainty of the molecular identification increases and the compound identity will be termed as "similar to a certain potential candidate". For spectra produced by novel species, oligomers of polymers, or a GC peak that contains two or more compounds, in which the NIST libraries do not have a good or similar reference spectrum, the peak identities usually are interpreted based on the MS knowledge of the analyst or would be just labeled as "unknown".

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<http://www.eaglabs.com/evaluate.htm?job=C09Y8961GC>

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Appendix 1

Gas Chromatography

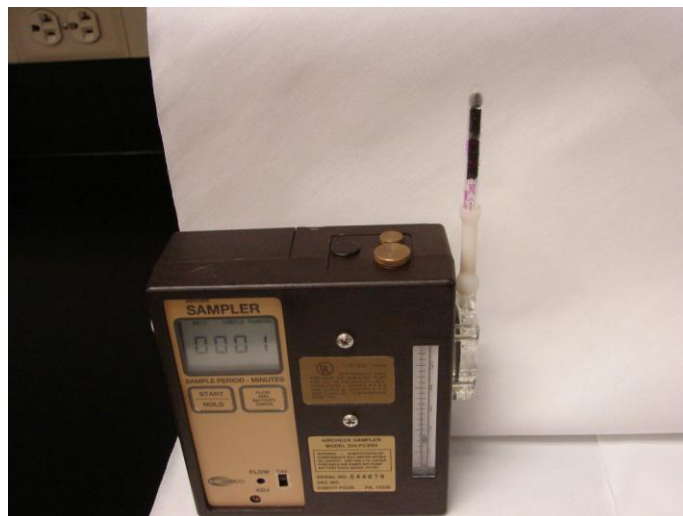
The powerful advantage of the gas chromatograph (GC) is that mixtures of volatile organic components can be separated into individual components for identification. Separation is achieved in a column located in a temperature-controlled oven. A wide variety of column dimensions and stationary phases (non-volatile solvents) are available depending on the application. Separation occurs when the components of a mixture partition between the stationary phase and an inert carrier gas (mobile phase). The separation profile of the sample components depends on their relative affinities for the stationary and mobile phases. Column temperature programming is used to manipulate retention time for the components, increasing the temperature for highly retained components or decreasing the temperature to gain more retention for a low boiling component. The retention time of a component is the time from injection to peak maxima. This is a characteristic property of the component and the liquid phase at a give temperature.

Mass Spectrometry

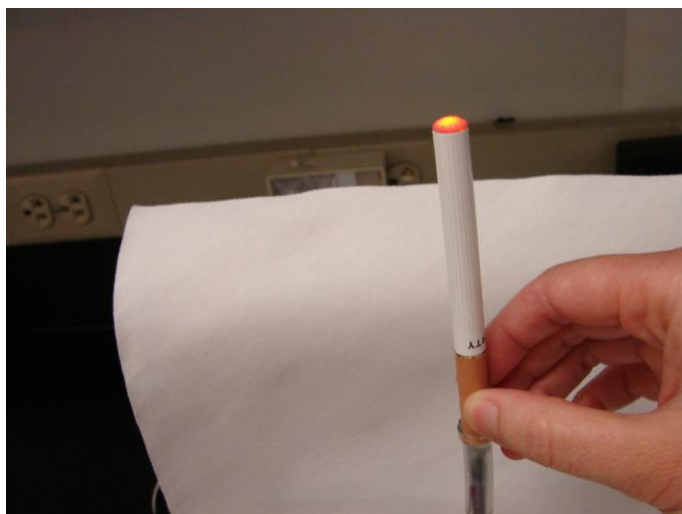
Detection of the eluting components from the gas chromatograph is achieved with the mass spectrometer. In the mass spectrometer, the component molecules are bombarded by a stream of high-energy electrons, converting some of the molecules to ions. The ions are accelerated in an electric field. The accelerated ions enter the mass analyzer, in this case a quadrupole, where the ions are separated according to their mass-to-charge ratios. Finally, the number of ions with a particular mass-to-charge ratio is counted. The result is a mass spectrum of the number of particles detected as a function of mass-to-charge ratio. The primary advantage of utilizing a mass spectrometer is that a mass spectrum can be a unique chemical "fingerprint". It may be possible either to identify an unknown compound or at least to place it within a chemical class.



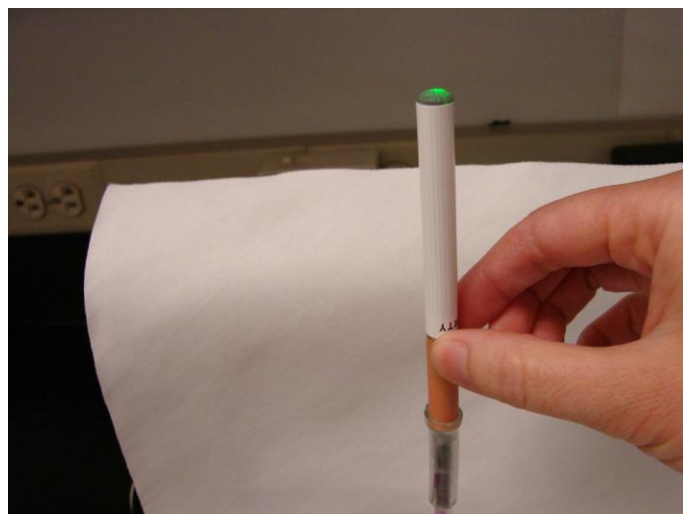
Picture 1



Picture 2



Picture 3



Picture 4

File : E:\JULY09\BSB\Y8961-27.D
Operator : [BSB1]X. CAI
Acquired : 21 Jul 2009 15:41 using AcqMethod STD_10
Instrument : GC/MS Ins
Sample Name: Charcoal blank 1ul/50ul
Misc Info : CDS,
Vial Number: 1

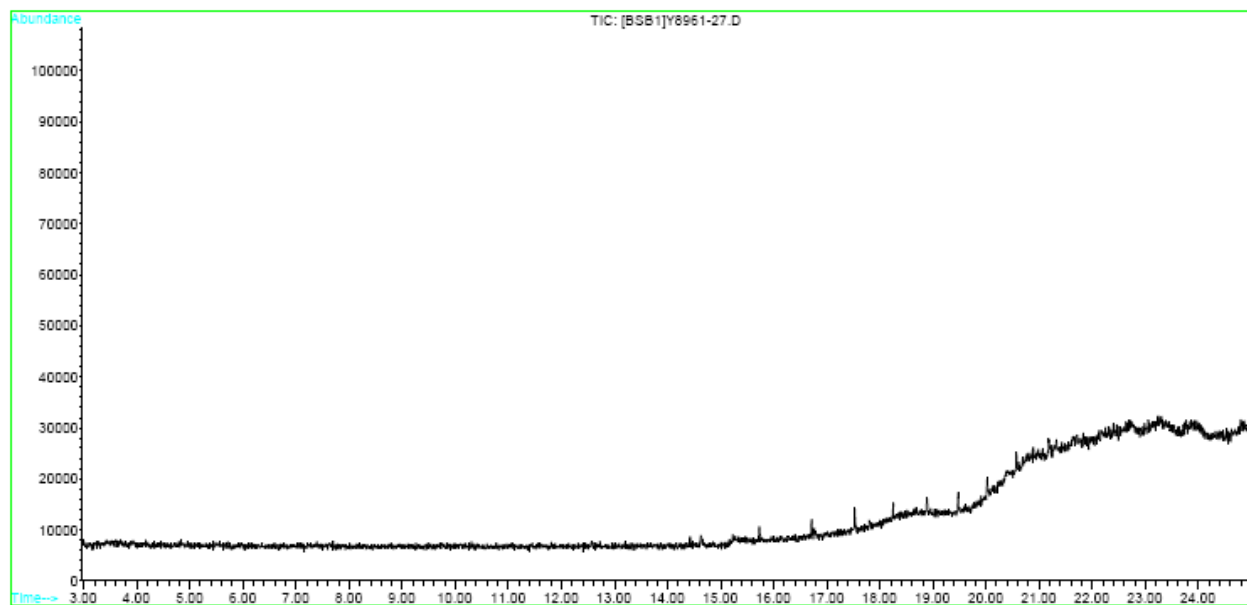


Figure 1

File : E:\JULY09\BSB\Y8961-31.D
Operator : [BSB1]X. CAI
Acquired : 22 Jul 2009 10:32 using AcqMethod STD_10
Instrument : GC/MS Ins
Sample Name: Charcoal extract 2/50ul
Misc Info : CDS,
Vial Number: 1

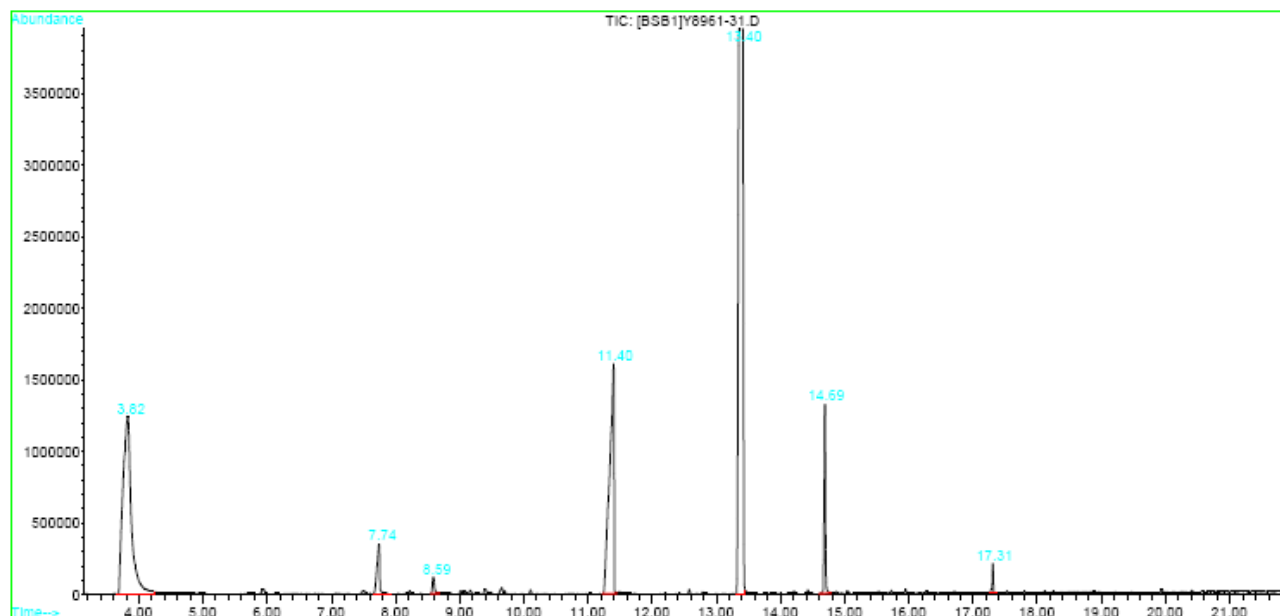
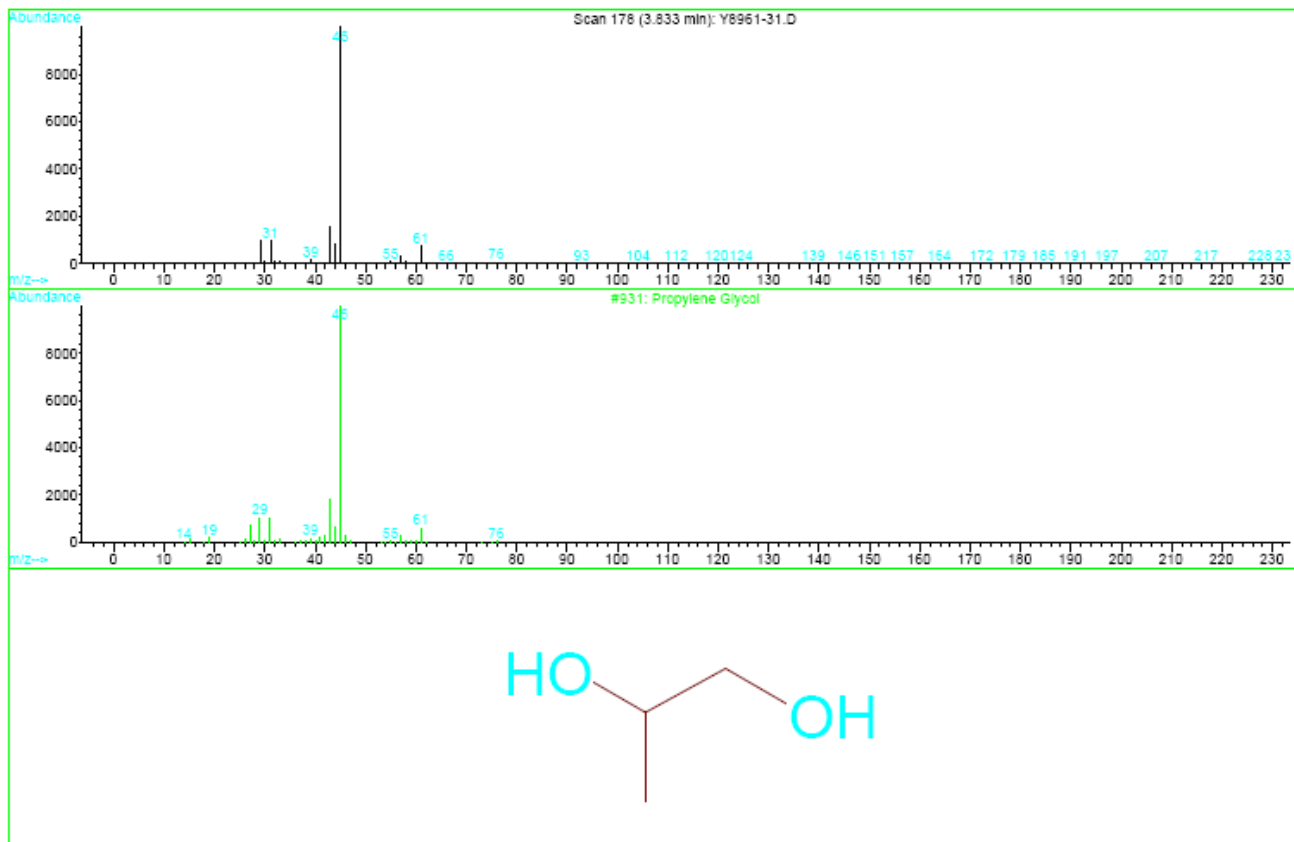


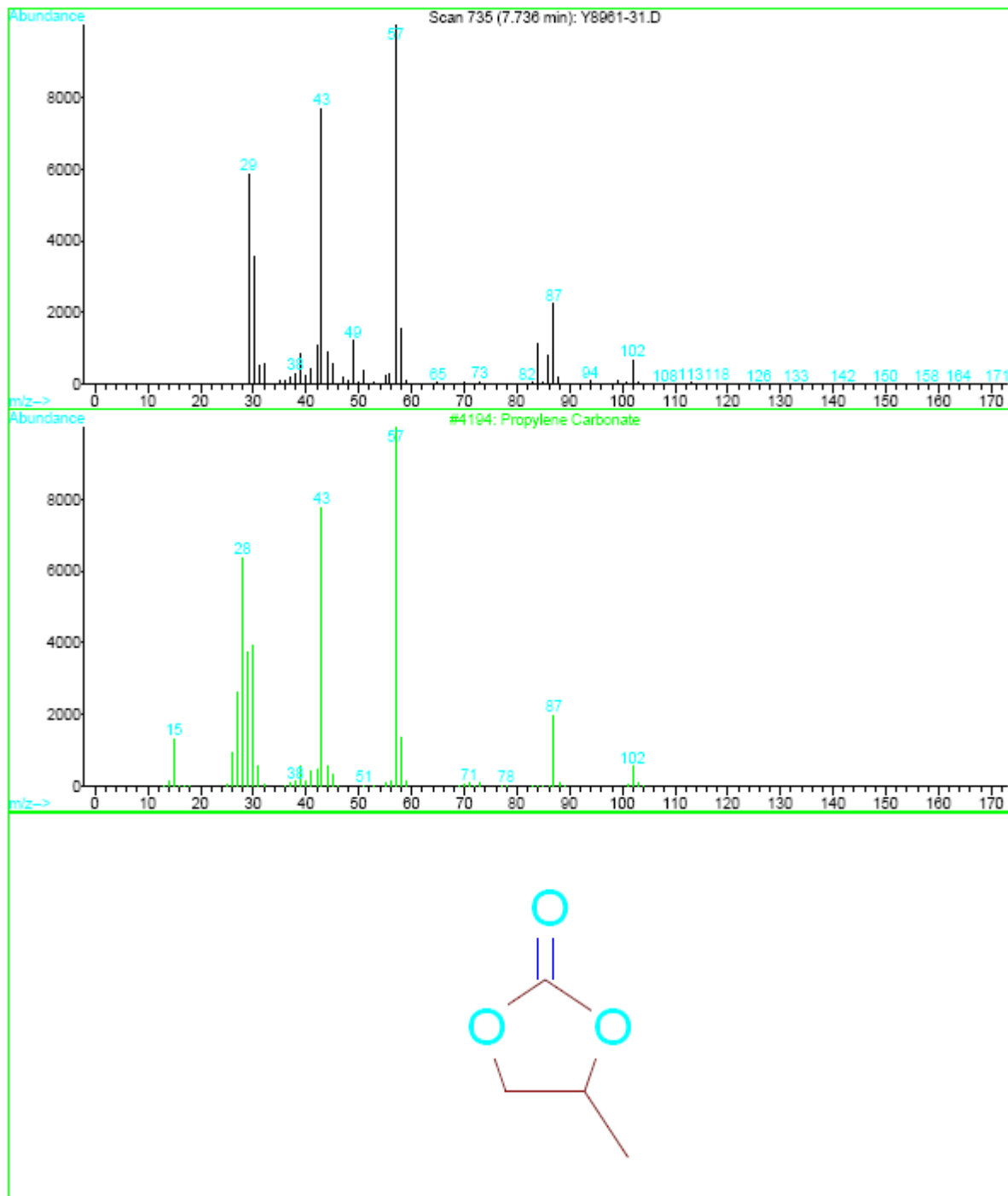
Figure 2

Library Searched : D:\DATABASE\nist08.L
Quality : 90
ID : Propylene Glycol



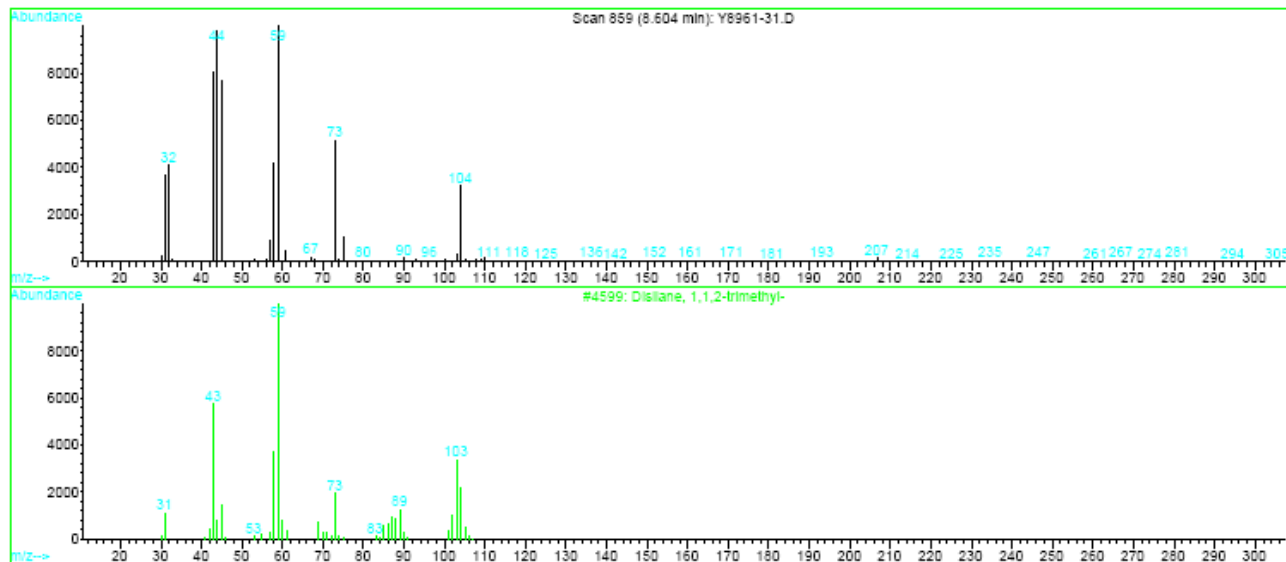
Spectrum 2-1

Library Searched : D:\DATABASE\nist08.L
Quality : 70
ID : Propylene Carbonate



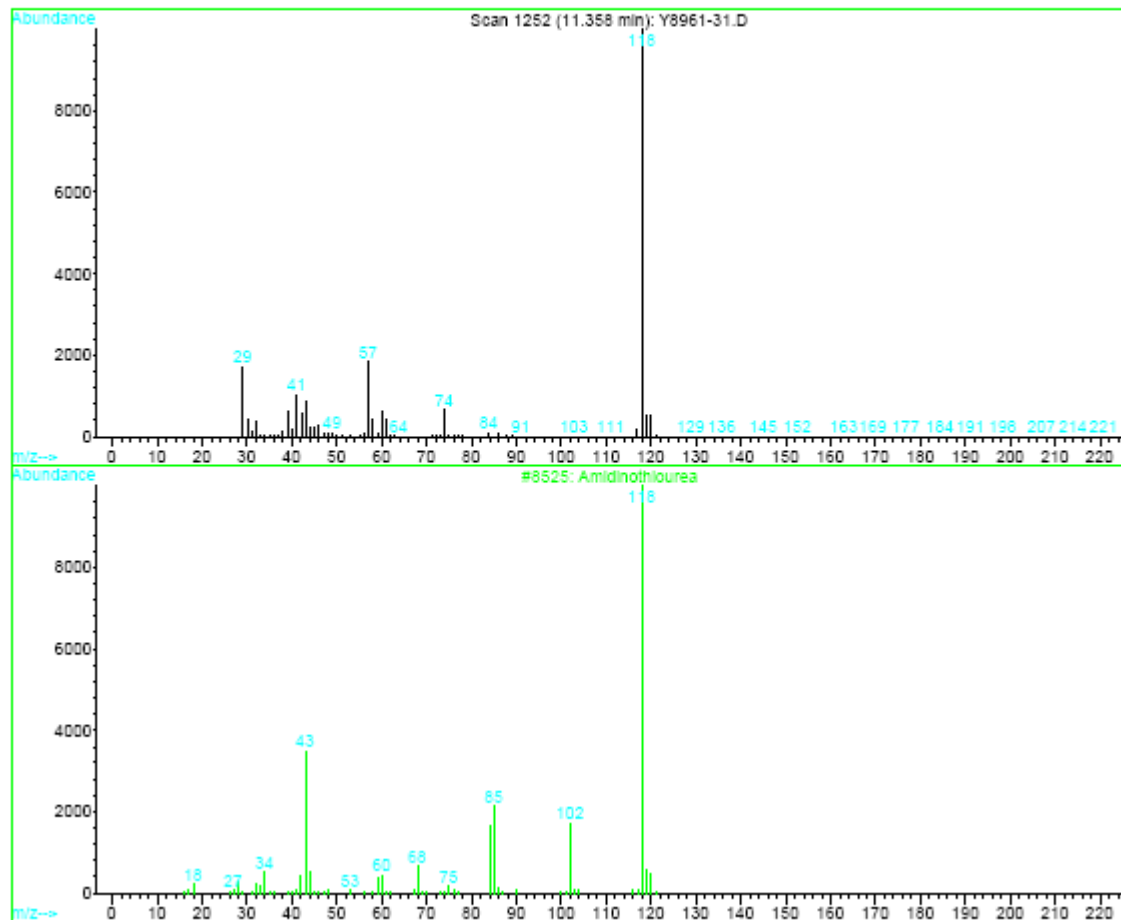
Spectrum 2-2

Library Searched : D:\DATABASE\nist08.L
Quality : 9
ID : Disilane, 1,1,2-trimethyl-



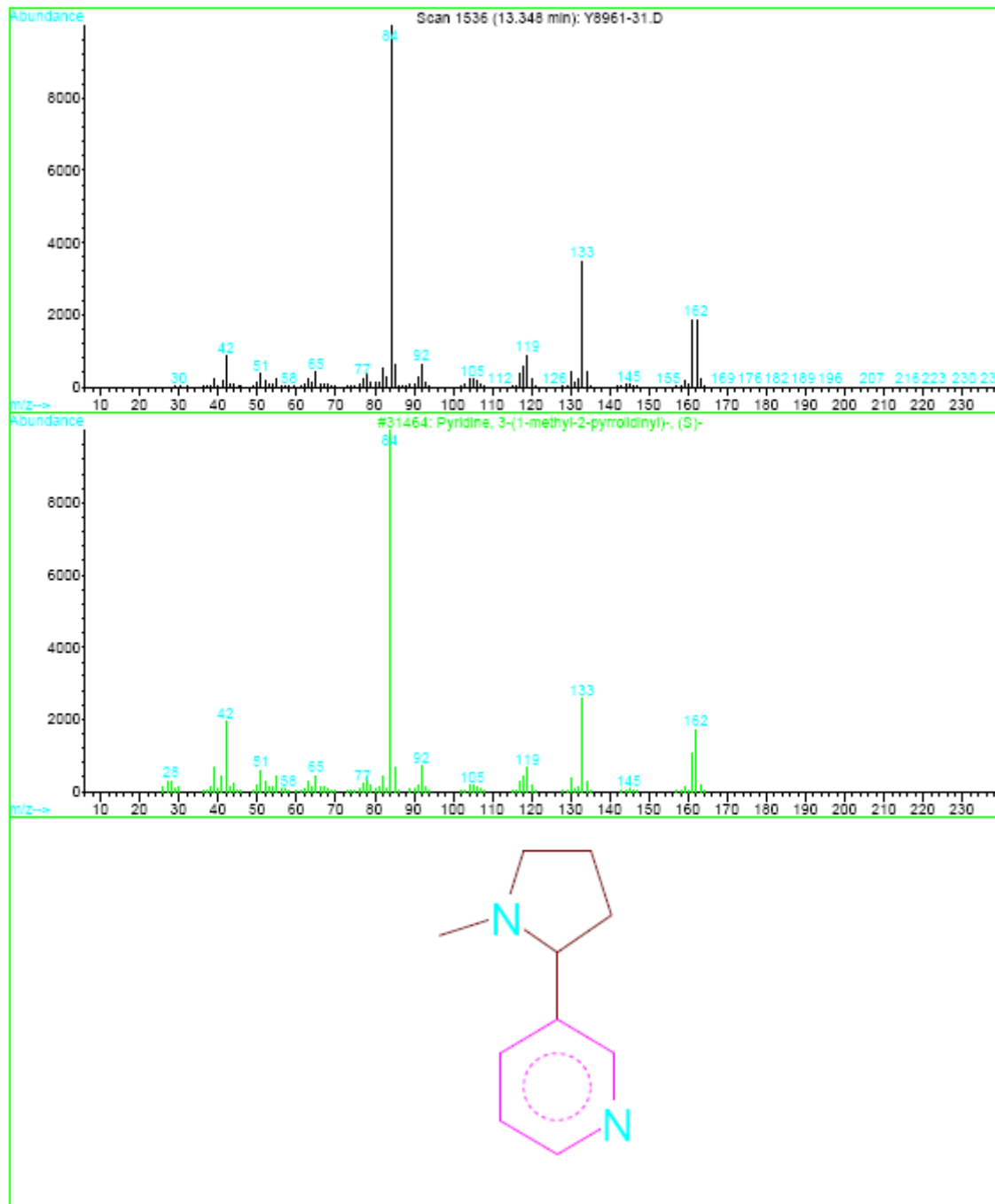
Spectrum 2-3

Library Searched : D:\DATABASE\nist08.L
Quality : 64
ID : Amidinothiourea



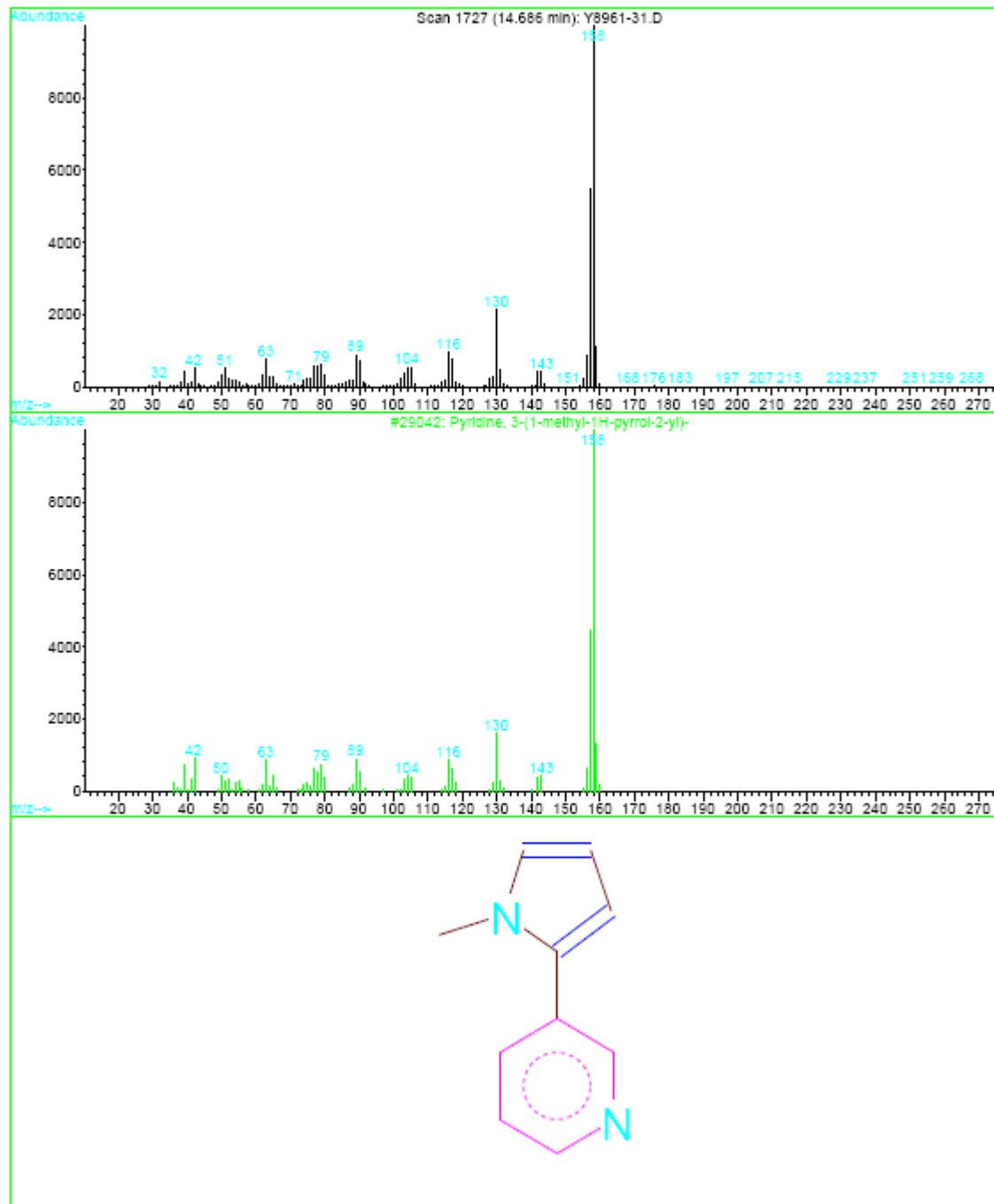
Spectrum 2-4

Library Searched : D:\DATABASE\nist08.L
Quality : 94
ID : Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-



Spectrum 2-5

Library Searched : D:\DATABASE\nist08.L
Quality : 93
ID : Pyridine, 3-(1-methyl-1H-pyrrol-2-yl)-

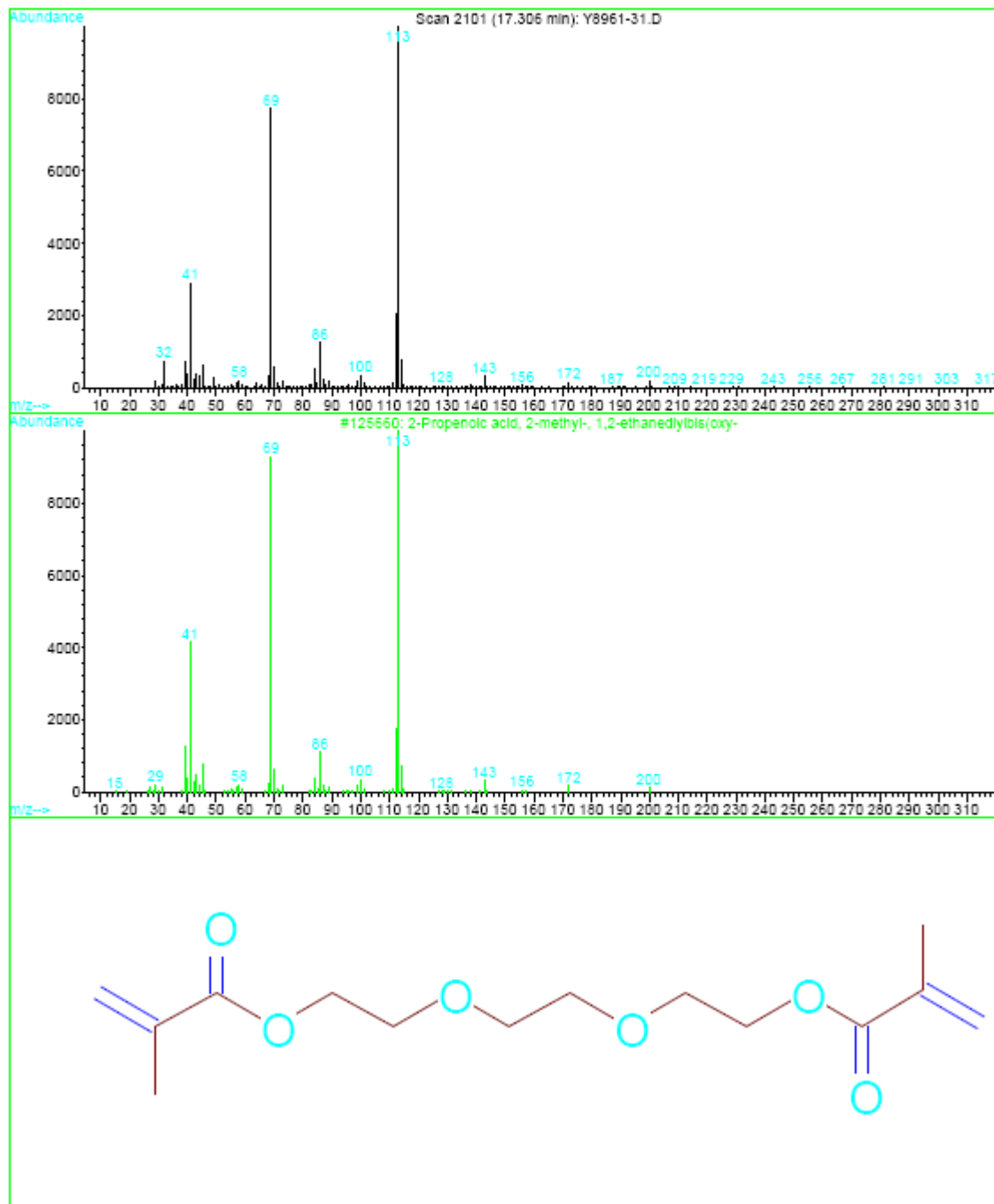


Spectrum 2-6

Library Searched : D:\DATABASE\nist08.L

Quality : 90

ID : 2-Propenoic acid, 2-methyl-, 1,2-ethanediybis(oxy-2,1-ethanediy) ester



Spectrum 2-7

File : E:\JULY09\Y8961-21.D
Operator : X. CAI
Acquired : 14 Jul 2009 11:54 using AcqMethod CDS_10_0
Instrument : GC/MS Ins
Sample Name: Liquid
Misc Info : Ch4, 120C 2 hours
Vial Number: 2

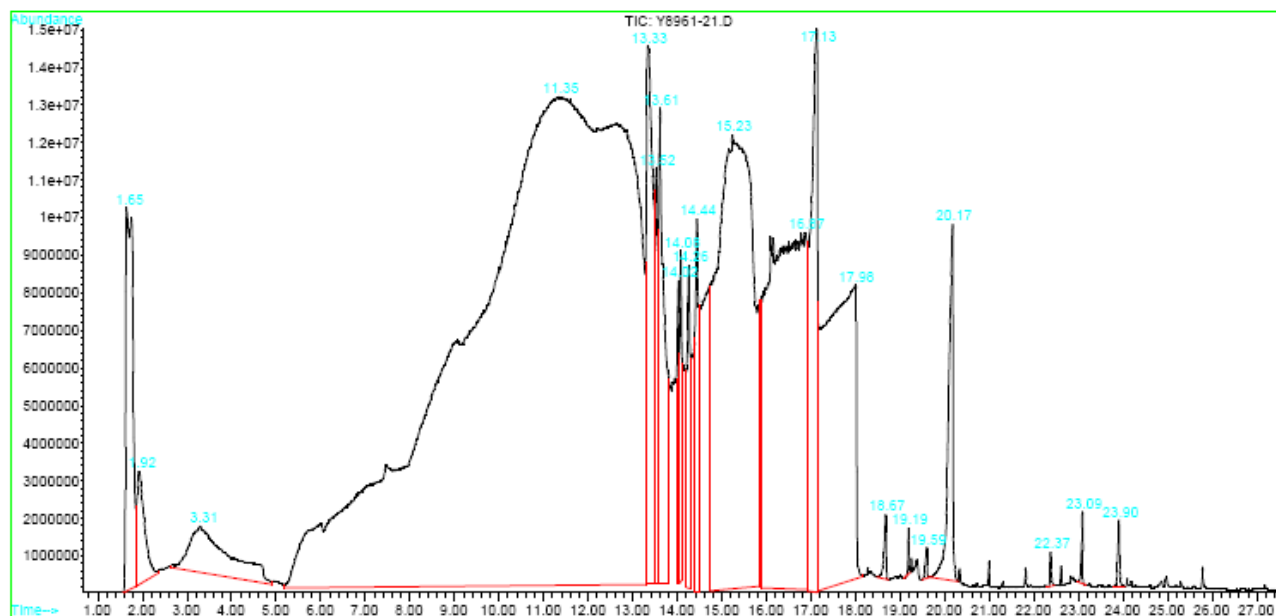
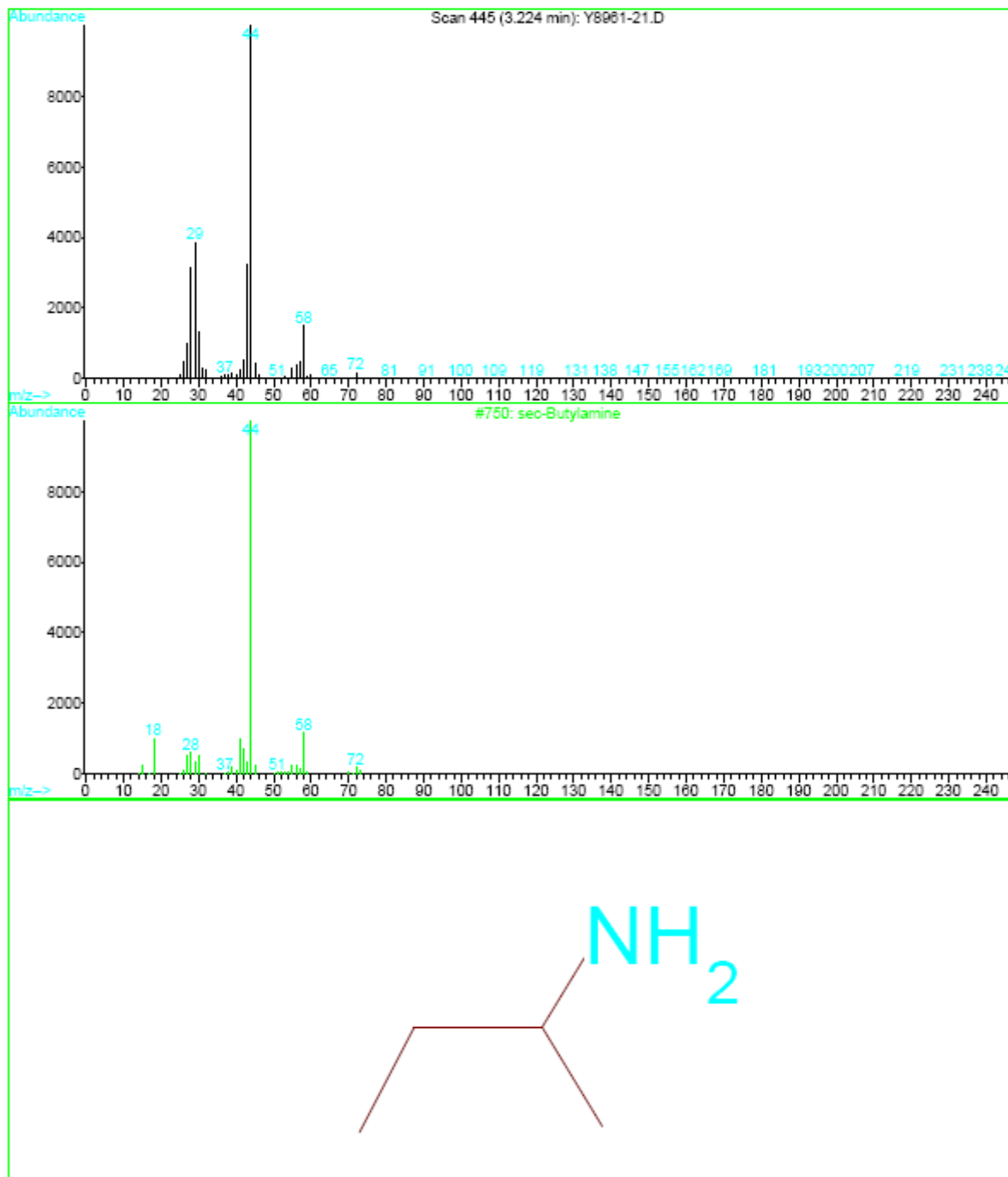


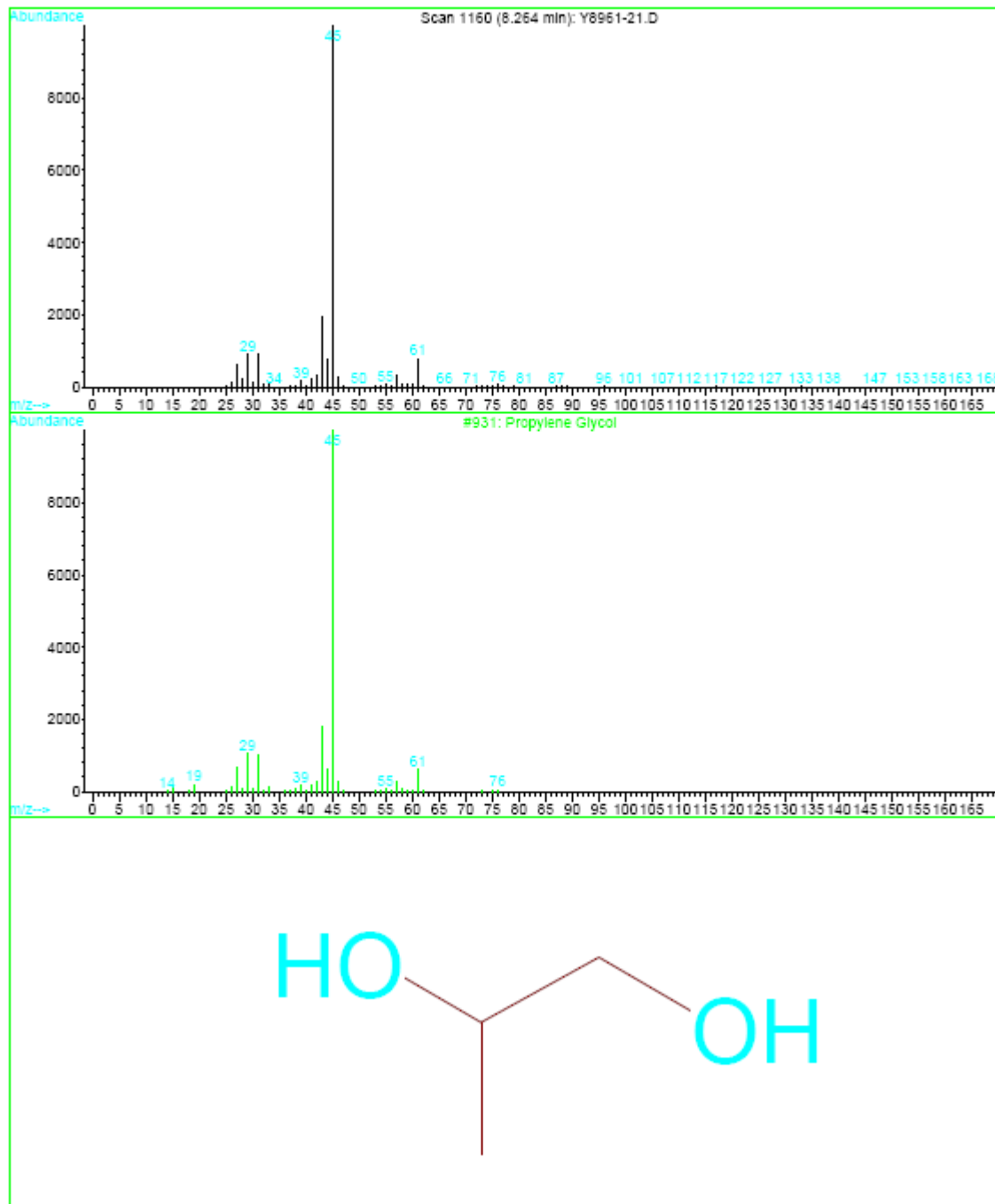
Figure 3

Library Searched : D:\DATABASE\Nist08.L
Quality : 78
ID : sec-Butylamine



Spectrum 3-1

Library Searched : D:\DATABASE\nist08.L
Quality : 90
ID : Propylene Glycol

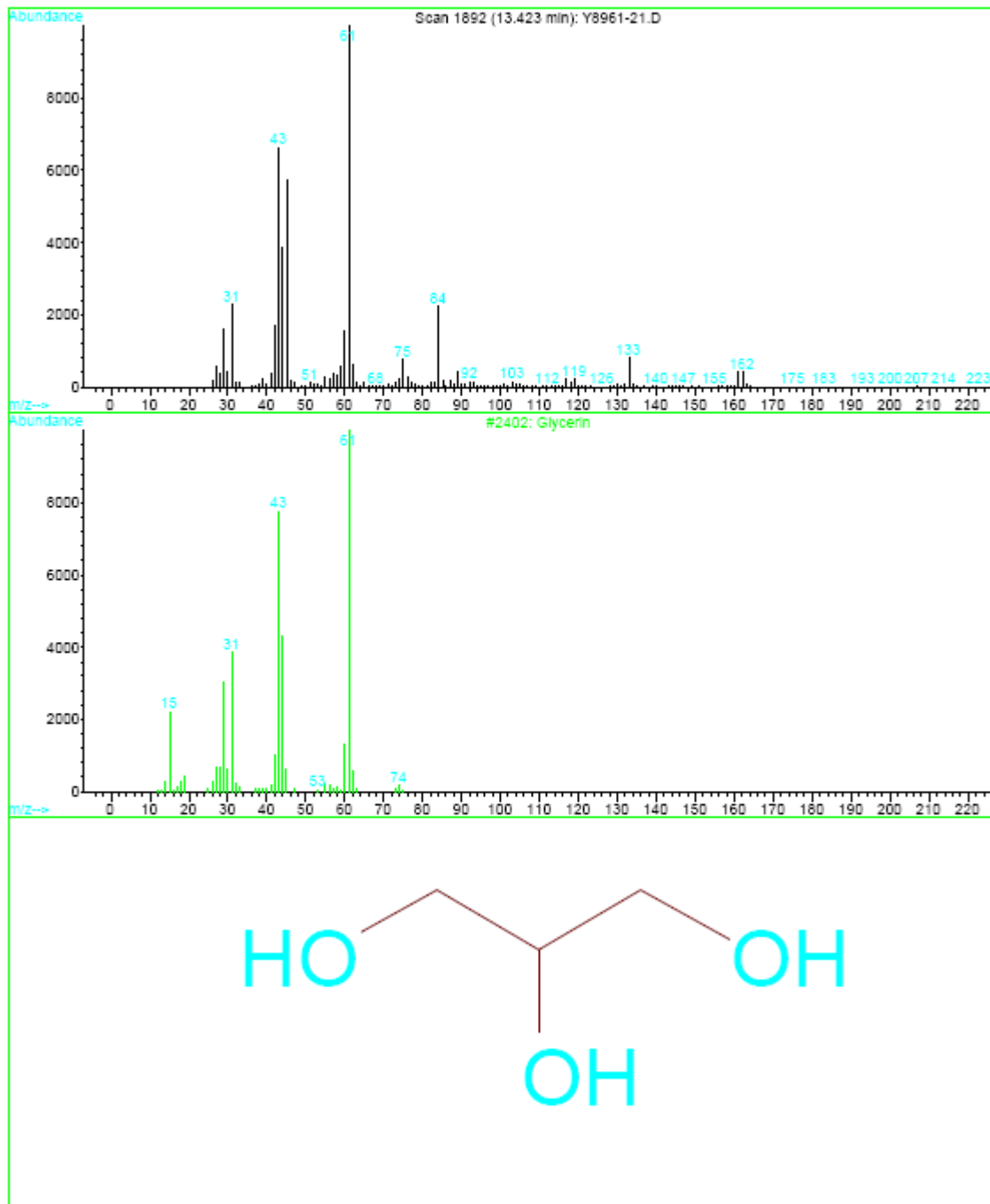


Spectrum 3-2

Library Searched : D:\DATABASE\nist08.L

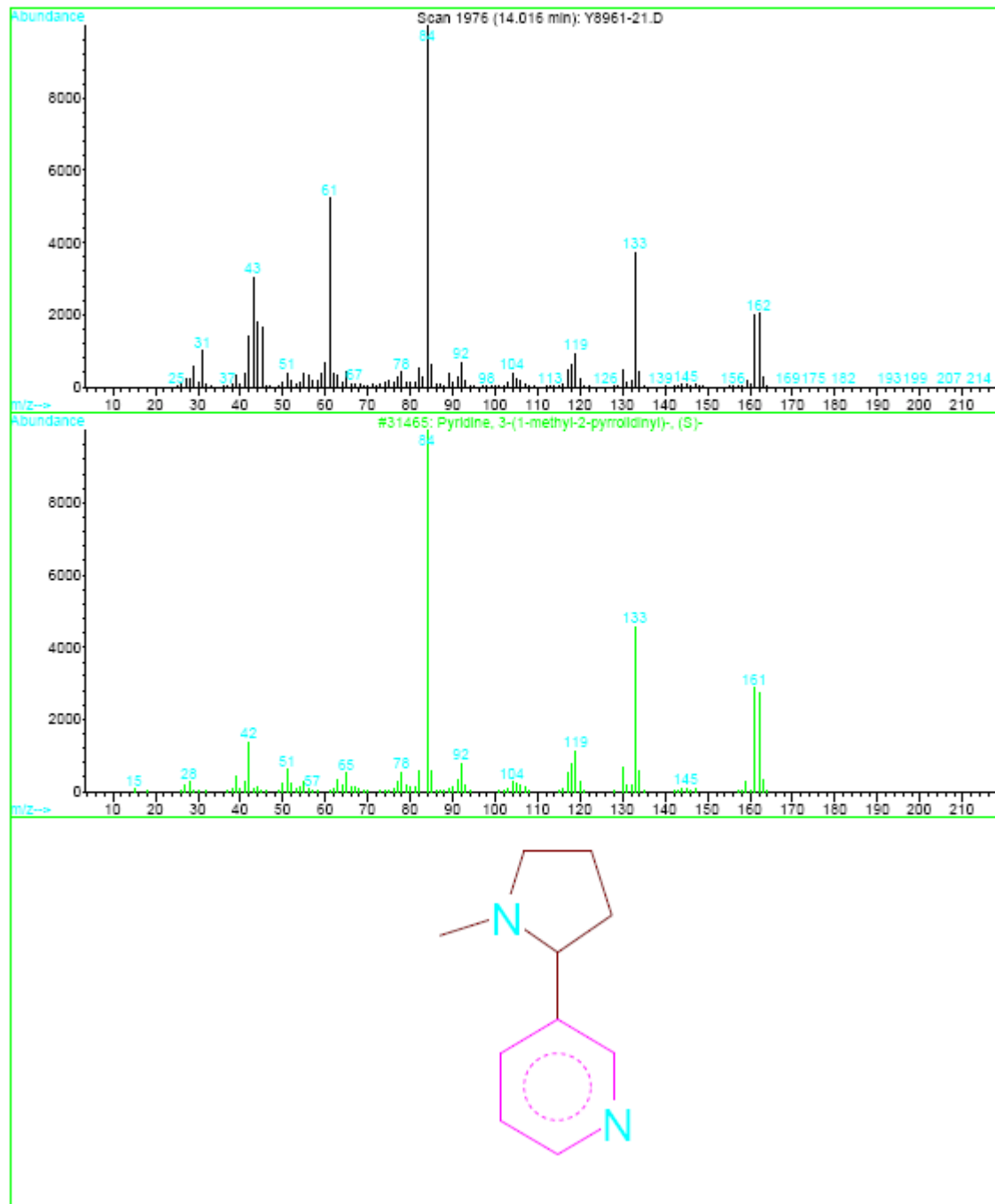
Quality : 53

ID : Glycerin



Spectrum 3-3

Library Searched : D:\DATABASE\nist08.L
Quality : 95
ID : Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-

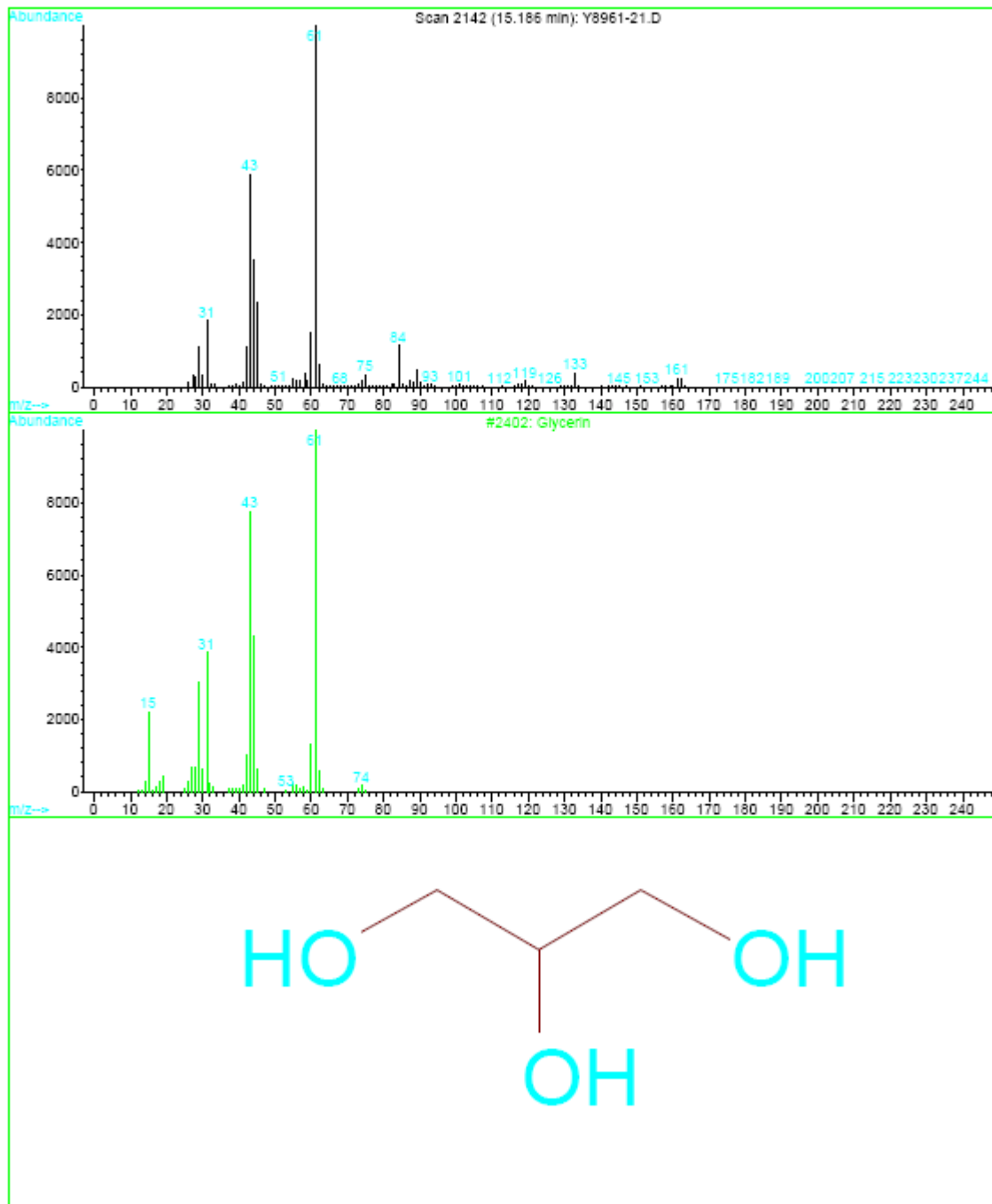


Spectrum 3-4

Library Searched : D:\DATABASE\nist08.L

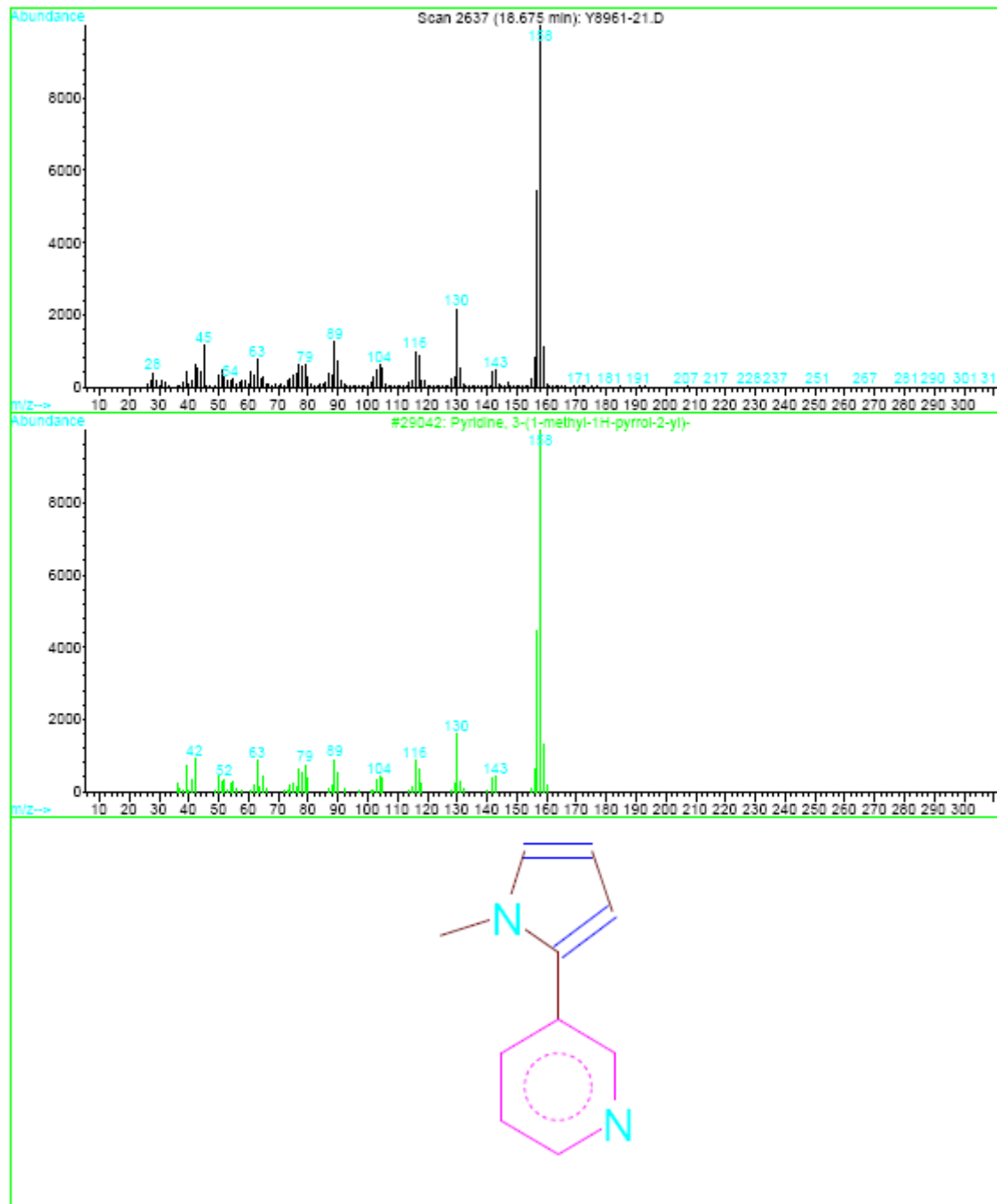
Quality : 64

ID : Glycerin



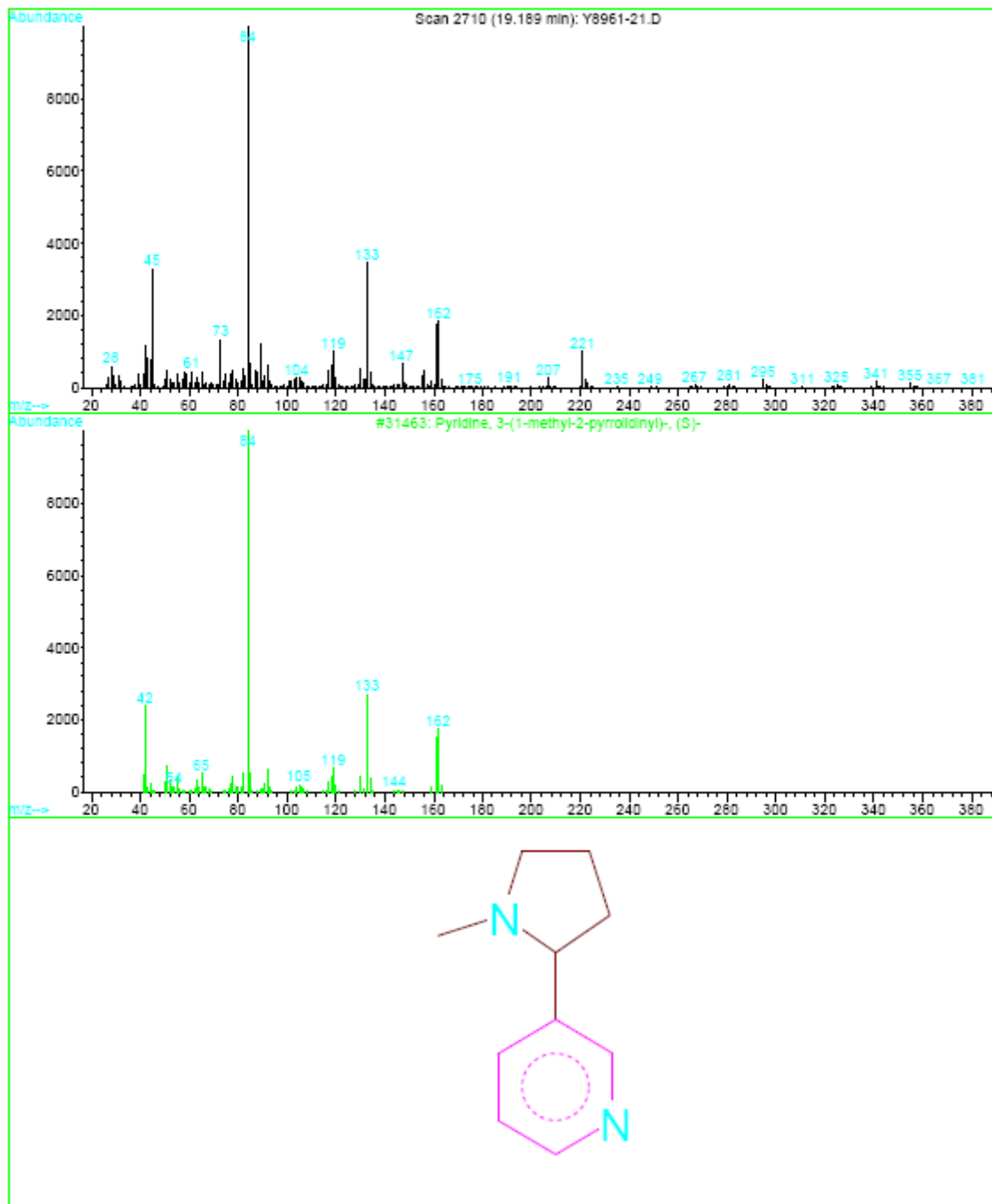
Spectrum 3-5

Library Searched : D:\DATABASE\nist08.L
Quality : 93
ID : Pyridine, 3-(1-methyl-1H-pyrrol-2-yl)-



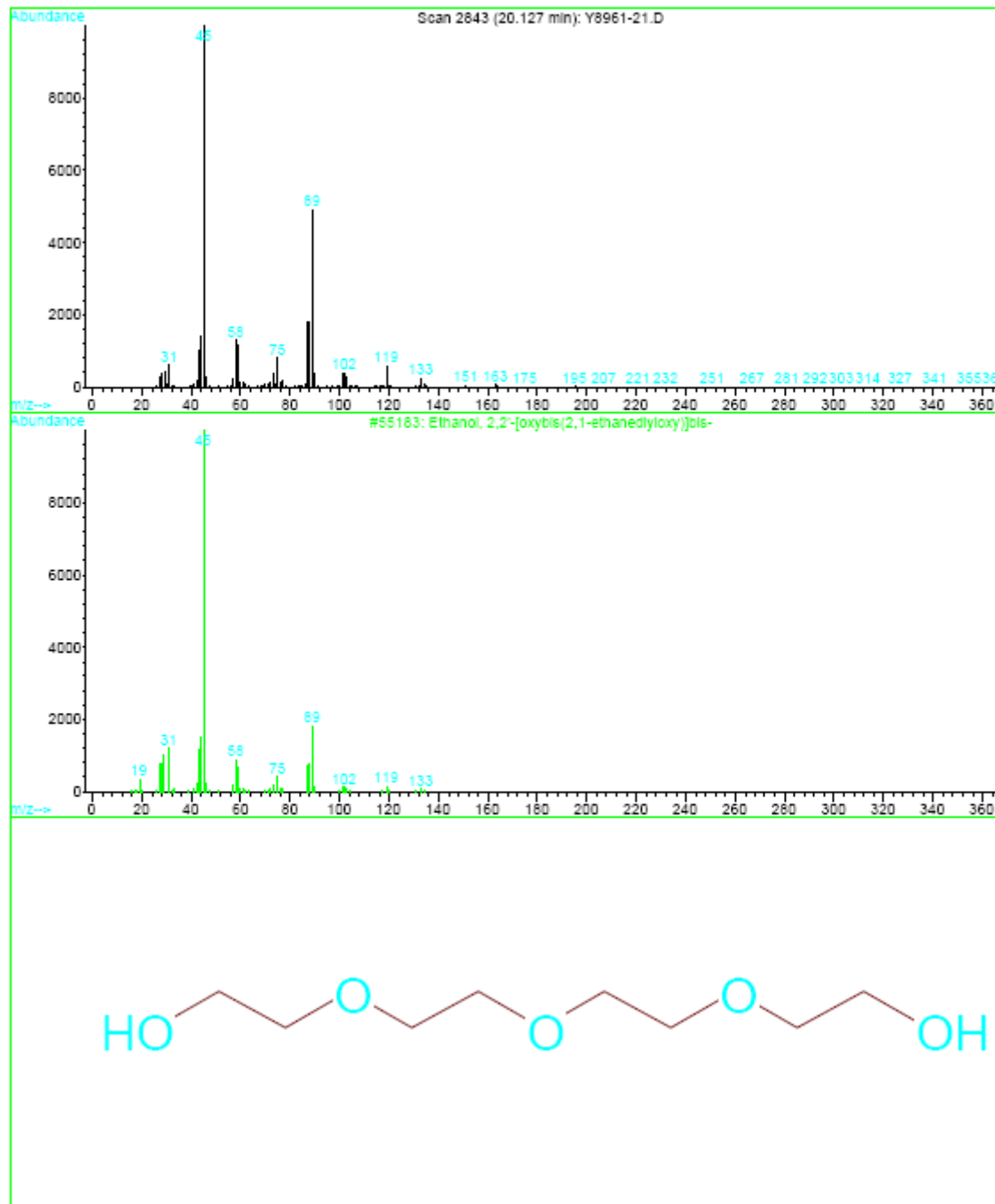
Spectrum 3-6

Library Searched : D:\DATABASE\NIST08.L
Quality : 92
ID : Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-



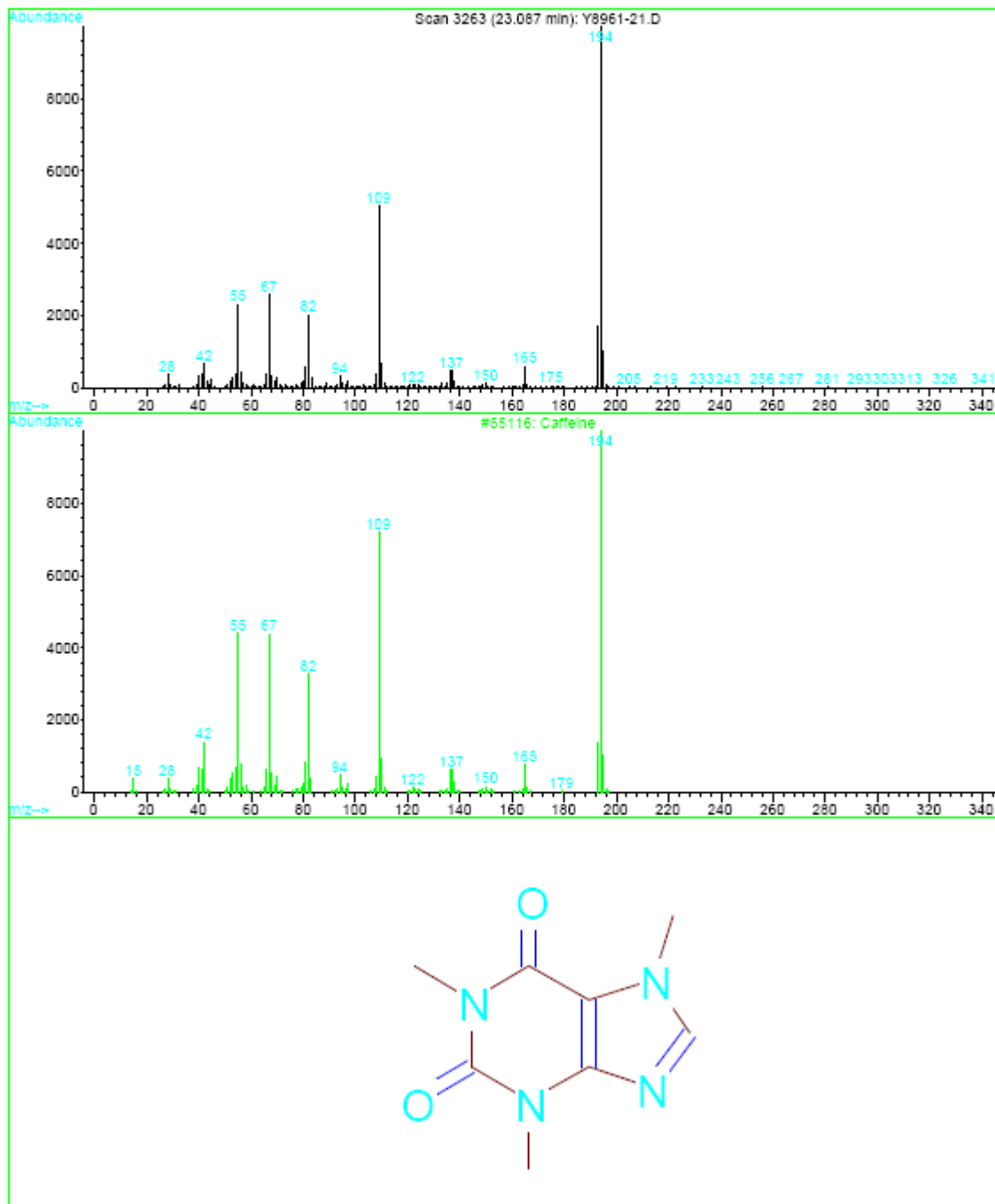
Spectrum 3-7

Library Searched : D:\DATABASE\nist08.L
Quality : 90
ID : Ethanol, 2,2'-[oxybis(2,1-ethanediyoxy)]bis-



Spectrum 3-8

Library Searched : D:\DATABASE\nist08.L
Quality : 97
ID : Caffeine

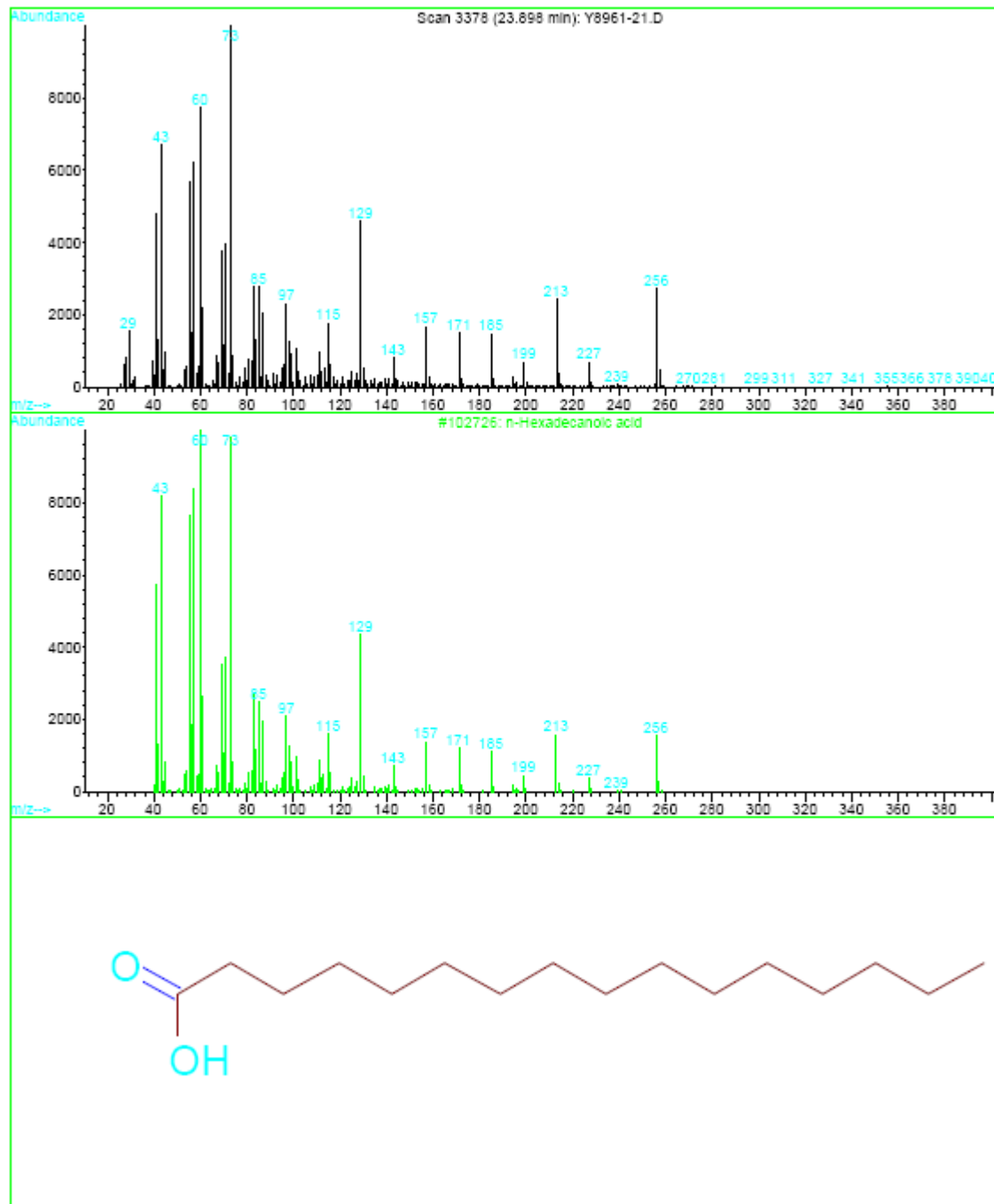


Spectrum 3-9

Library Searched : D:\DATABASE\nist08.L

Quality : 99

ID : n-Hexadecanoic acid



Spectrum 3-10

File : E:\JULY09\Y8961-24.D
Operator : X. CAI
Acquired : 17 Jul 2009 13:11 using AcqMethod CDS_10_0
Instrument : GC/MS Ins
Sample Name: Cigarette
Misc Info : 50C 180 minutes
Vial Number: 3

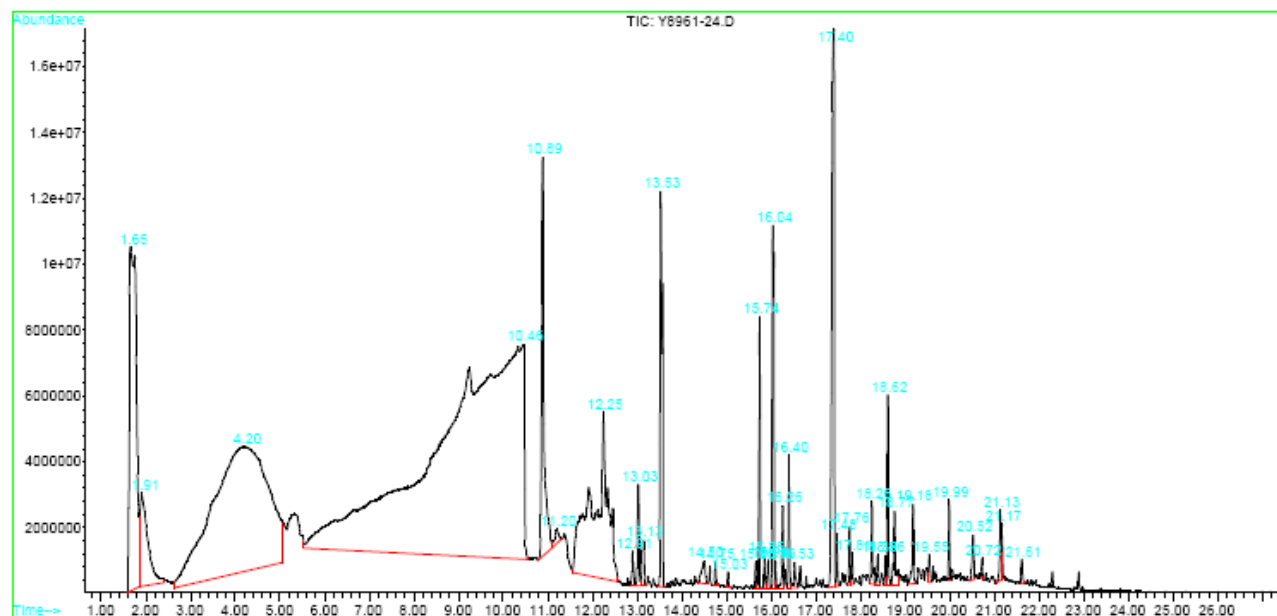
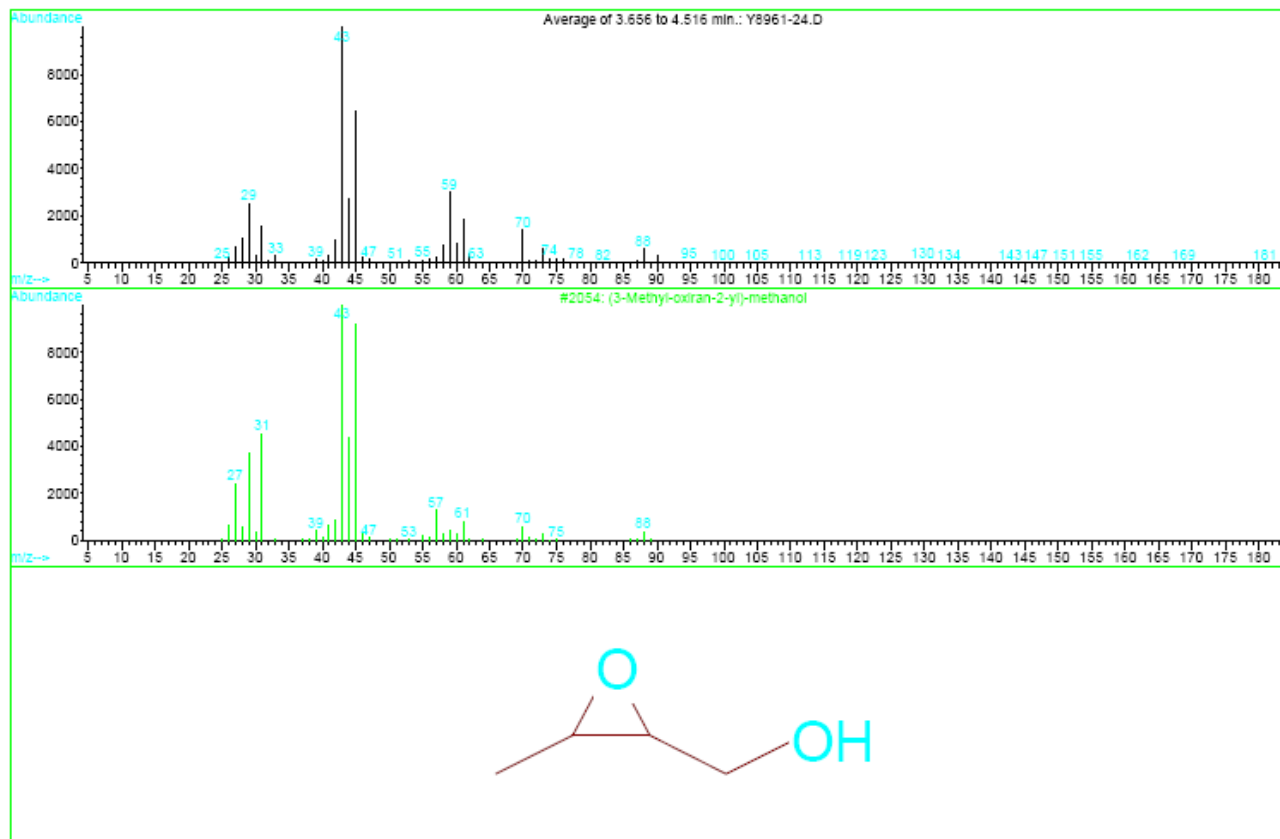


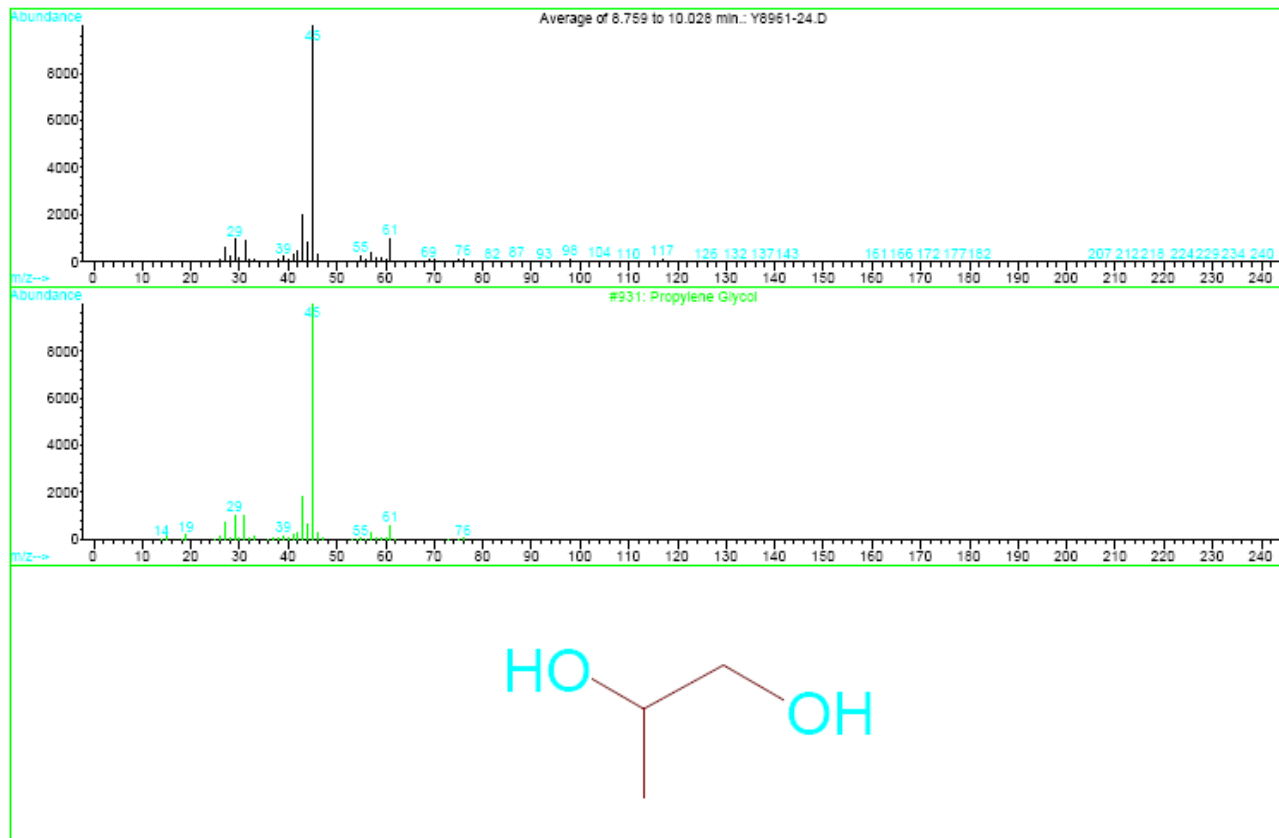
Figure 4

Library Searched : D:\DATABASE\NIST08.L
Quality : 59
ID : (3-Methyl-oxiran-2-yl)-methanol



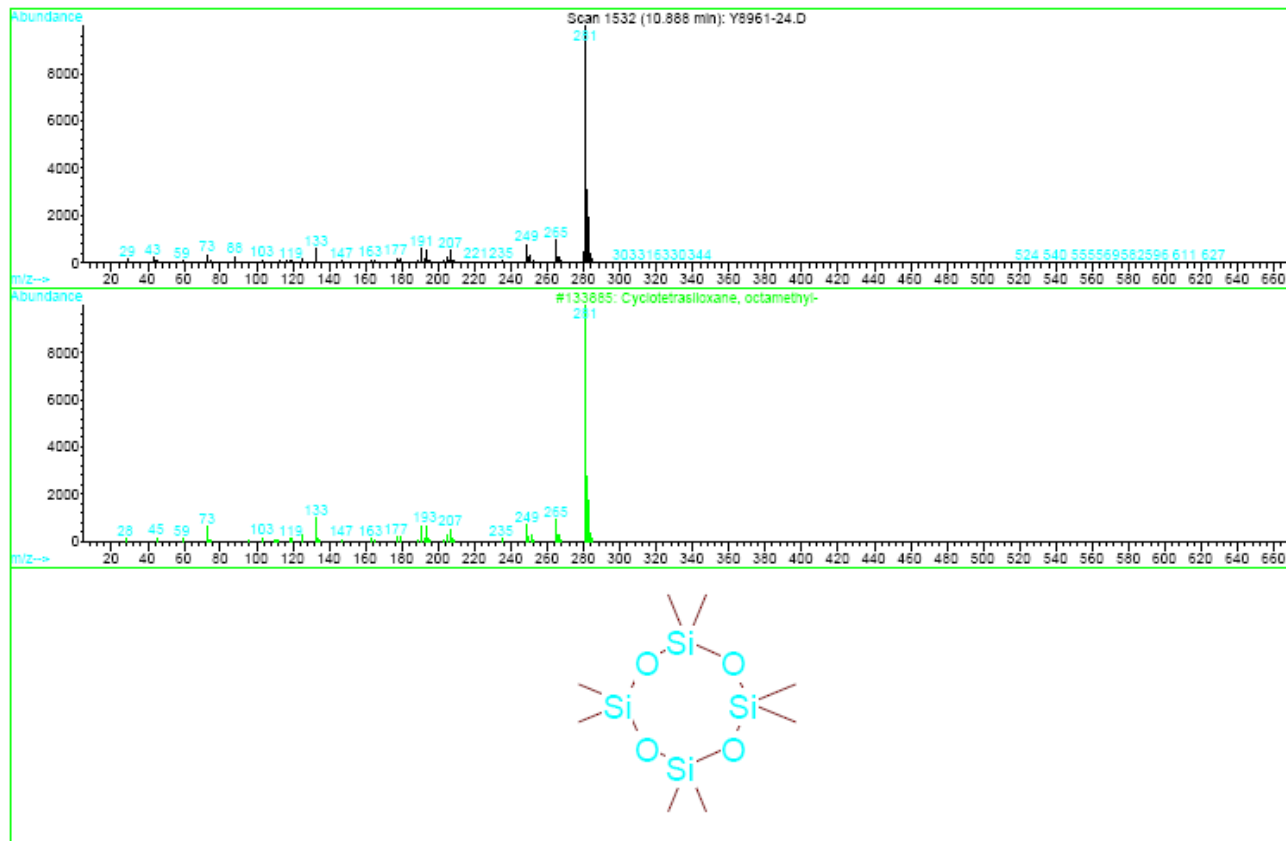
Spectrum 4-1

Library Searched : D:\DATABASE\nist08.L
Quality : 86
ID : Propylene Glycol



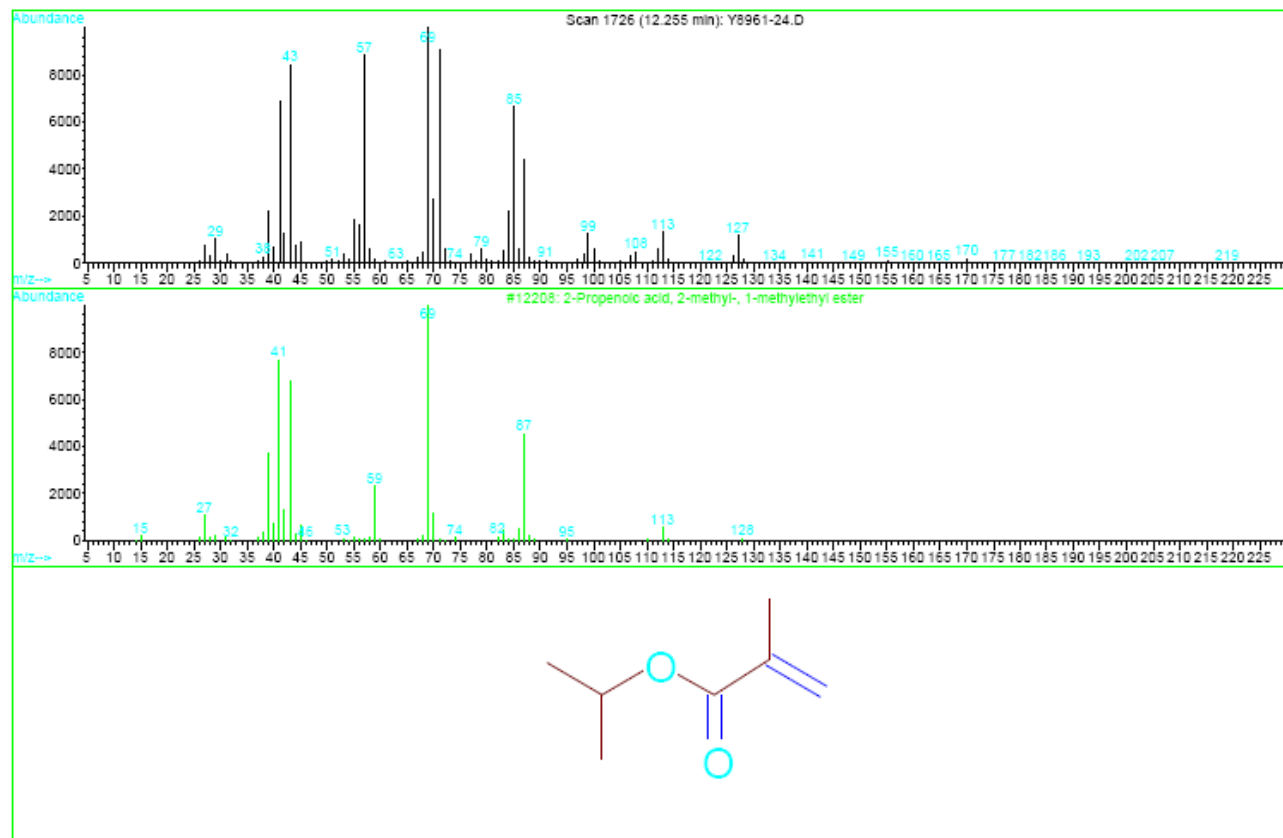
Spectrum 4-2

Library Searched : D:\DATABASE\nist08.L
Quality : 87
ID : Cyclotetrasiloxane, octamethyl-



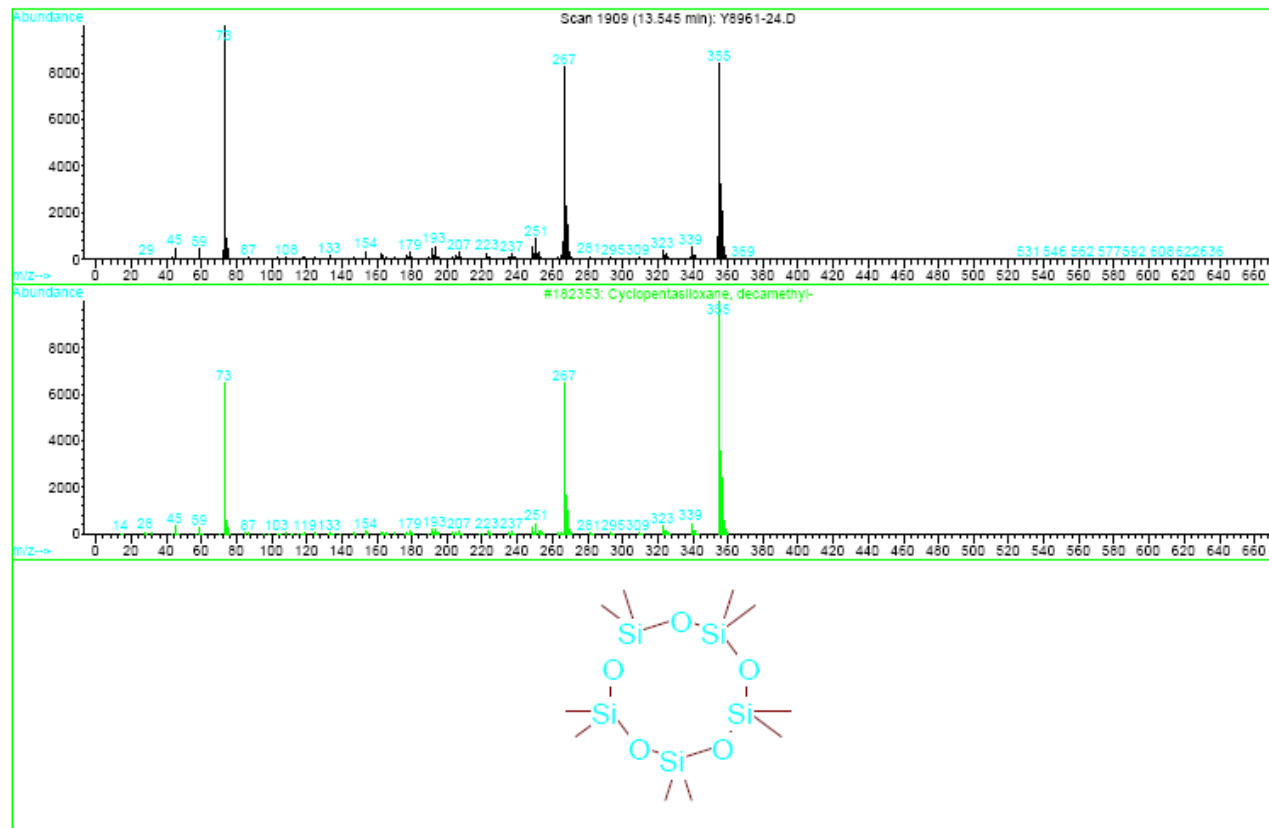
Spectrum 4-3

Library Searched : D:\DATABASE\nist08.L
Quality : 42
ID : 2-Propenoic acid, 2-methyl-, 1-methylethyl ester



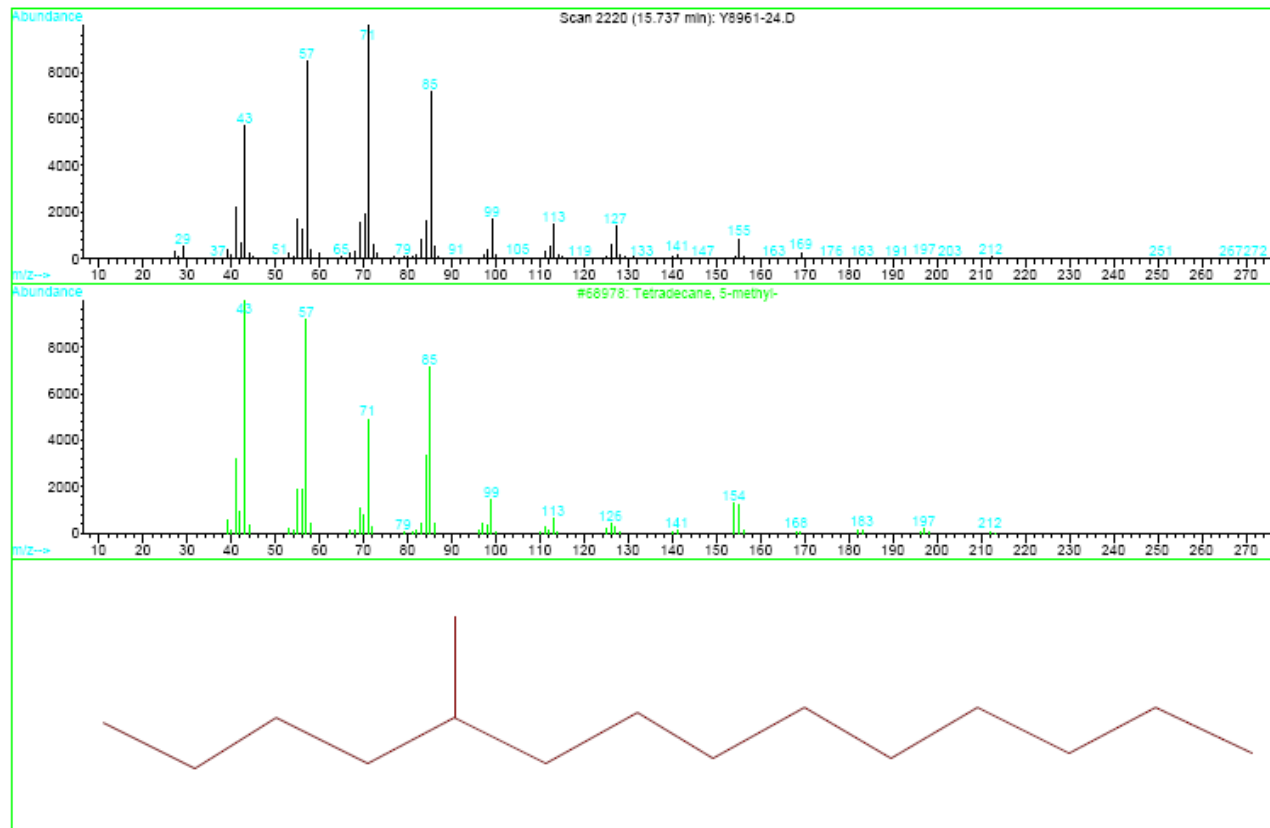
Spectrum 4-4

Library Searched : D:\DATABASE\nist08.L
Quality : 91
ID : Cyclopentasiloxane, decamethyl-



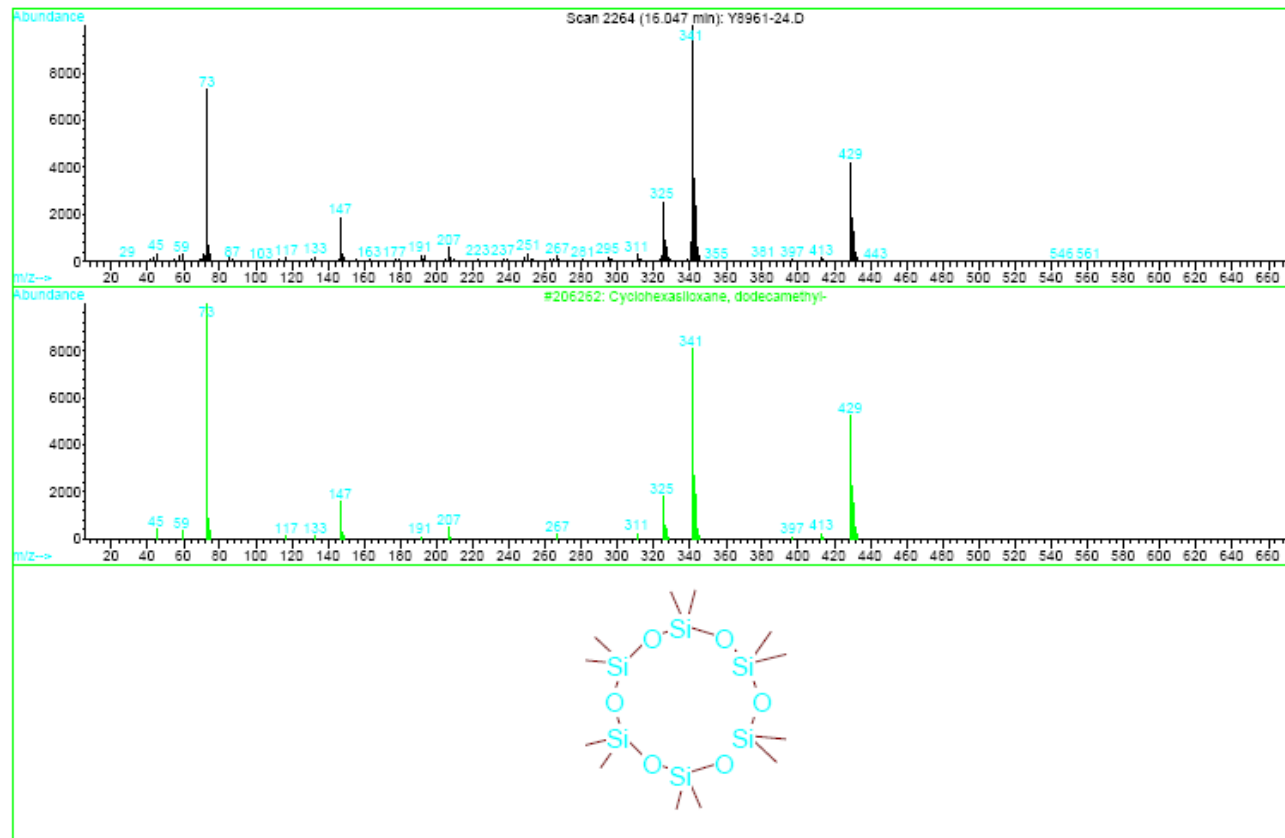
Spectrum 4-5

Library Searched : D:\DATABASE\nist08.L
Quality : 43
ID : Tetradecane, 5-methyl-



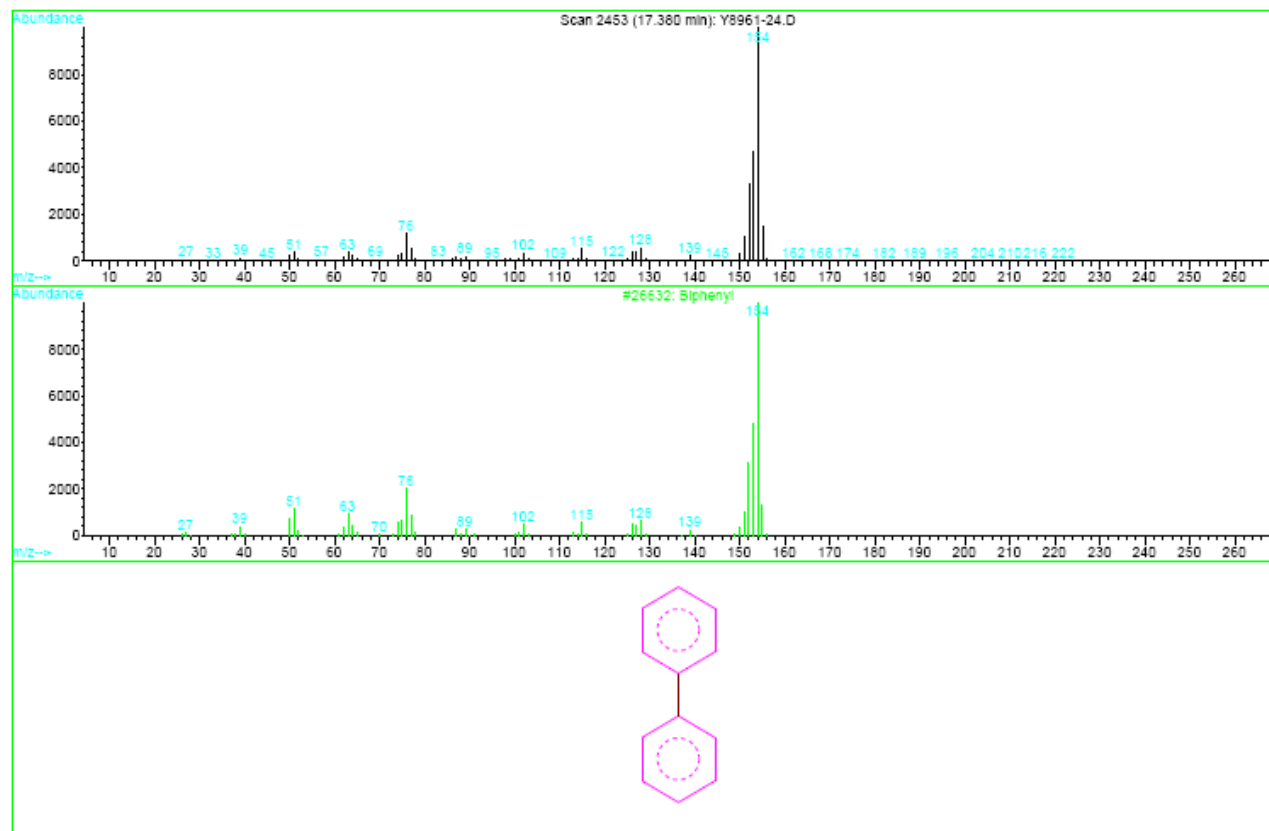
Spectrum 4-6

Library Searched : D:\DATABASE\nist08.L
Quality : 91
ID : Cyclohexasiloxane, dodecamethyl-



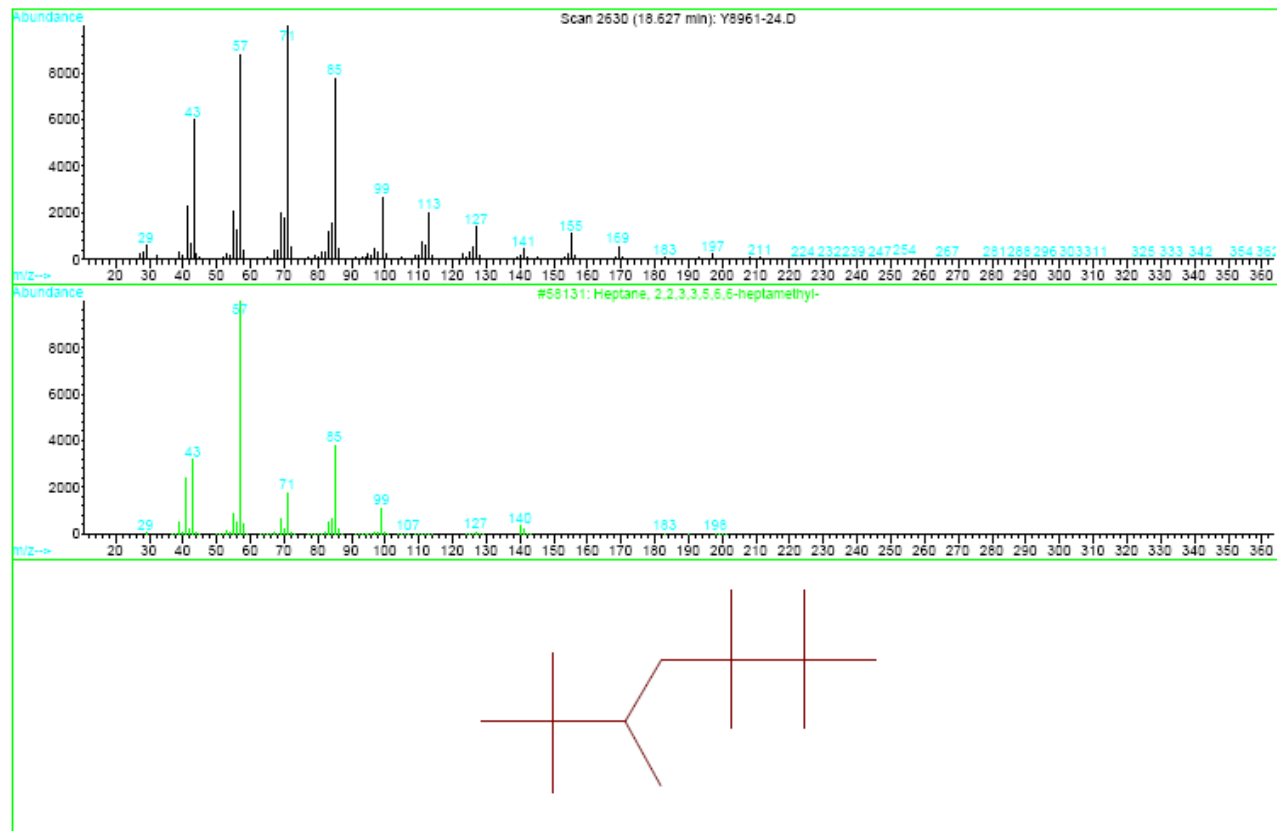
Spectrum 4-7

Library Searched : D:\DATABASE\nist08.L
Quality : 91
ID : Biphenyl



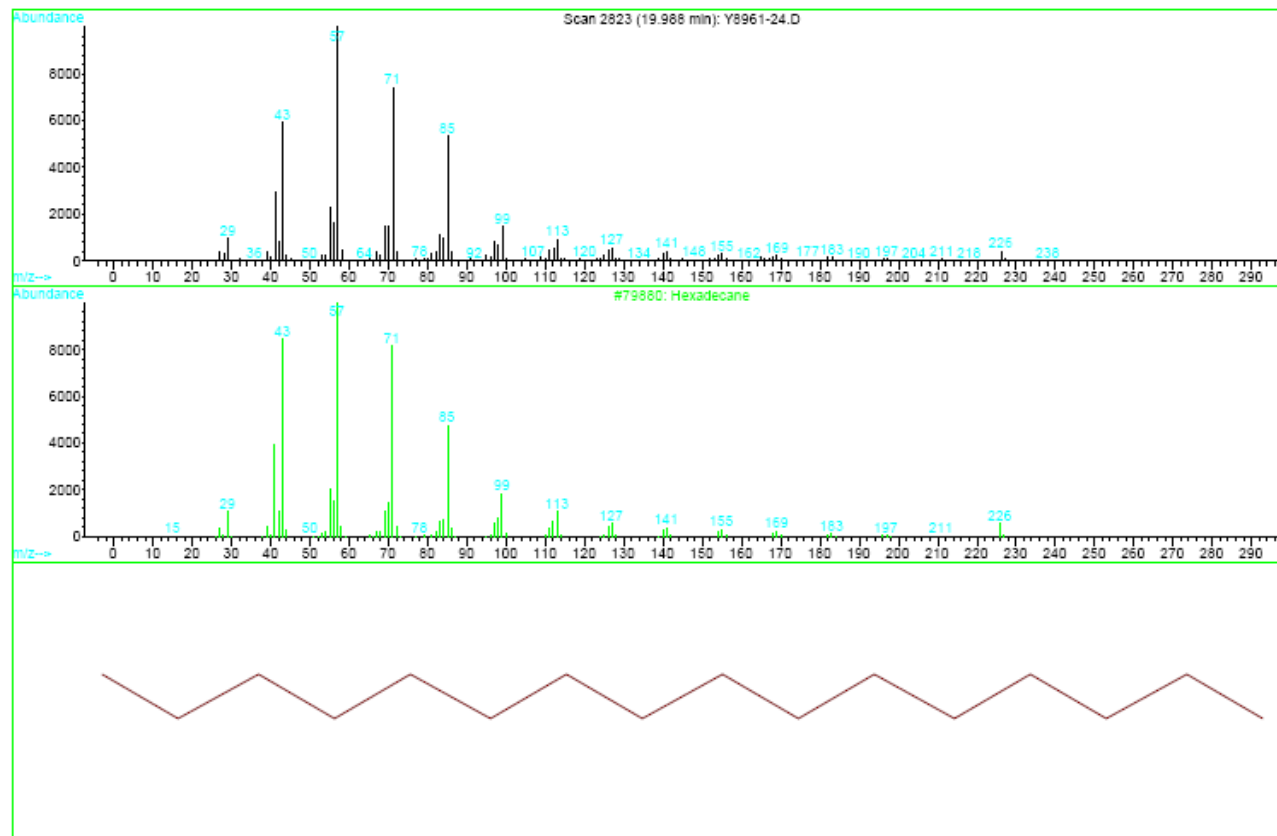
Spectrum 4-8

Library Searched : D:\DATABASE\nist08.L
Quality : 38
ID : Heptane, 2,2,3,3,5,6,6-heptamethyl-



Spectrum 4-9

Library Searched : D:\DATABASE\nist08.L
Quality : 98
ID : Hexadecane



Spectrum 4-10