

ATTACHMENT 3

X-ray Fluorescence (XRF) Elemental Analysis



ISO 17025



Testing Cert. #2797.01

**X-RAY FLUORESCENCE (XRF)
ANALYSIS REPORT
21 Mar 2013**

**JOB NUMBER C0DHY160
PO NUMBER 4500000443**

for

Keith Waldrop
Electric Power Research Institute

Prepared by:

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XRF ANALYSIS REPORT

Requester:
Job Number:
Analysis Date:

Keith Waldrop
C0DHY160
21 Mar 2013

Purpose:

The purpose of this analysis was to determine the compositions of particulate matter collected on various substrates. Three samples were analyzed along with clean duplicates as controls (six samples total). The materials analyzed included polyester filters identified in this report as "Filter Used" and "Filter Control"; Scotch-Brite pads (Used and Control); and thin polypropylene or polyethylene filters associated with SaltSmart analyzers (SaltSmart Used and SaltSmart Control).

Summary:

The results are summarized in Table 1 and Table 2. Table 2 has the thin film residues from the two filters quantified separately from the filter media, and also provide an estimated areal density for the coverages.

Experimental:

X-ray Fluorescence (XRF) is a non-destructive technique that can identify and quantify the elemental constituents of a sample using the secondary fluorescence signal produced by irradiation with high energy x-rays. This analysis utilized a wavelength dispersive spectrometer (WDXRF) that is capable of detecting elements from atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range up to 100% by weight.

Analytical Parameters

Instrument	Rigaku Primus II WDXRF
X-ray source	Rhodium x-ray tube
Atmosphere	Vacuum
Analysis area	10mm diameter

Quantification was performed using the Fundamental Parameters (FP) standardless quantification software associated with the system. The fundamental parameters approach uses x-ray physics coupled with established sensitivity factors for pure elements. Relative accuracy by this method usually ranges from better than 5% up to ~20% for major elements.

Results and Interpretations:

Spectra are included in the attached figures. Sample or area names are provided on the spectra. The results are summarized in Table 1.

Data from the two Scotch-Brite samples showed very little difference in composition. These data suggest that any residue filtered through the Scotch-Brite pads either was not retained, or was imbedded too deeply to be detected. Both samples were principally composed of C, N, O and Al, which is consistent with XRD results (reported separately) that found corundum and aluminum nitride associated with the base polymer matrix.

The polyester filter (labeled as #3) had considerable levels of Fe, Cr, Ni and Mn, which might indicate stainless steel residue. The sample also had notable levels of Zn and Ca, along with lower levels of several additional species not found on the Control.

Table 2 provides a more accurate quantification of the residue on the filter, since it removes the contribution of the filter matrix and quantifies the results as a thin film instead of a bulk solid. The data from the filters shown in Table 1 serve only as comparative results between Used sample and Control.

The SaltSmart filter also had metallic species present, although the relative levels were lower than what was seen on the polyester filter. Additionally, the relative levels of the Fe, Cr, Ni and Zn are different from that observed on the polyester filter.

The major species detected on the SaltSmart filter were O, Si, Fe and Ca, along with lower but still significant levels of Mg, Al, K, Ti and Zn. Additional species were detected at trace levels. As with the polyester filter, the results in Table 1 provide a general comparison between Used sample and Control, while Table 2 provides a more accurate representation of the quantities of residue detected.

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Table 1. Sample Compositions (in Wt%)^a

Element	Scotch-Brite Used	Scotch-Brite Control	Filter Used ^b	Filter Control	SaltSmart Used ^b	SaltSmart Control
C	54.9	56.1	58.4	59.9	80.6	98.6
N	8.1	7.9	-	-	-	-
O	29.1	27.6	40.2	39.9	16.2	1.4
Na	0.047	0.045	0.008	-	0.10	-
Mg	0.082	0.055	0.028	-	0.27	-
Al	6.84	7.41	0.066	0.003	0.39	0.005
Si	0.24	0.15	0.18	0.003	1.38	0.012
P	0.002	0.002	0.008	0.004	0.046	0.0057
S	0.020	0.010	0.028	-	0.070	0.003
Cl	0.007	0.005	-	-	0.023	-
K	0.093	0.083	0.037	0.006	0.096	-
Ca	0.097	0.096	0.14	-	0.69	0.0021
Ti	0.37	0.41	0.19	0.16	0.021	-
Cr	0.006	0.004	0.15	-	0.002	-
Mn	0.009	0.011	0.016	-	0.003	-
Fe	0.050	0.036	0.48	0.002	0.083	0.0006
Ni	0.002	0.002	0.032	0.001	0.0005	-
Cu	-	-	0.002	-	-	-
Zn	-	-	0.004	-	0.005	-
Sr	0.011	0.016	-	-	-	-
Y	0.002	0.003	-	-	-	-
Zr	0.041	0.057	-	-	0.0001	-
Sb	-	-	0.002	0.002	-	-
Th	0.001	0.002	-	-	-	-

^a The results are normalized to 100% of the measured and detected elements

^b Residue and filter media were quantified as a homogeneous volume which is not an accurate representation. Table 2 gives results from the residue modeled as films on the filter media, which is a better representation of the actual quantities present.

Table 2. Areal Density of Residue (in $\mu\text{g}/\text{cm}^2$) and Composition (in Wt%)^a

	Filter Used	SaltSmart Used
Areal Density ($\mu\text{g}/\text{cm}^2$)	155	120
Element	Wt% of Elements	
O	- ^b	13.4
Na	0.021	0.4
Mg	0.077	1.16
Al	0.24	2.31
Si	0.84	11.2
P	0.049	0.49
S	0.22	0.97
Cl	-	0.43
K	0.85	4.03
Ca	4.30	38.6
Ti	- ^c	1.95
Cr	13.9	0.26
Mn	1.88	0.67
Fe	69.5	21.3
Ni	6.60	0.20
Cu	0.39	-
Zn	1.12	2.86
Zr	-	0.17

^a Table 2 provides the total areal density of the residue on the filter surface and a compositional breakdown of the residue in weight percent.

^b Oxygen is likely present in the residue as one or more metal oxides; however, it cannot be quantified due to the high O content in the PET filter.

^c Titanium appears to be present in the residue form this sample; however, it cannot be quantified due to the presence of Ti in the filter itself.

Wavelength Dispersive X-ray Fluorescence Spectroscopy (WDXRF) Description Appendix

In XRF photons from an x-ray tube irradiate a sample causing the ejection of inner shell electrons from the excitation volume of the sample, creating inner shell vacancies. In order to reestablish a stable electron configuration, electrons from outer shells fill the inner shell vacancies. In this process fluorescent photons are produced to balance the energy difference between the outer and inner shells. These fluorescent x-rays are the source of the signal in x-ray fluorescence spectroscopy, and their energies are characteristic of the atoms from which they originate. Therefore the fluorescent signal can determine the elements present in the sample matrix and, from the relative intensities, the concentrations. By using an appropriate elemental and matrix reference standard, or fundamental parameter algorithms when standards are unavailable, accurate quantification of the elemental make-up of the sample can be obtained. With appropriate standards accuracies can be better than 1% relative; while using the Fundamental Parameters method typically yields accuracies of better than 5% to ~20% relative for major elements. Long term measurement reproducibility is ~2% at the 95% confidence limit.

In a wavelength dispersive XRF spectrometer (WDXRF) the fluorescence signals from the sample are collimated, after which they impinge upon one or more crystals. Each signal is diffracted at a specific angle based on the lattice spacing of the crystal and the fluorescent photon energy, following Bragg's law. Wavelength dispersive systems are generally operated by sequentially scanning the detectors over the full dispersion range of one or more crystals to collect the elemental signals. The relative intensities of the signals are a function of the concentration of the element, matrix effects, and factors attributable to the primary x-ray radiation. The system used in this analysis is capable of detecting elements of atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range to 100% by weight.

The excitation volume for XRF is both element and matrix dependent. It can range from the micrometer range for light elements in dense metallic materials to a centimeter or more for heavier elements in light element matrices such as polymers.

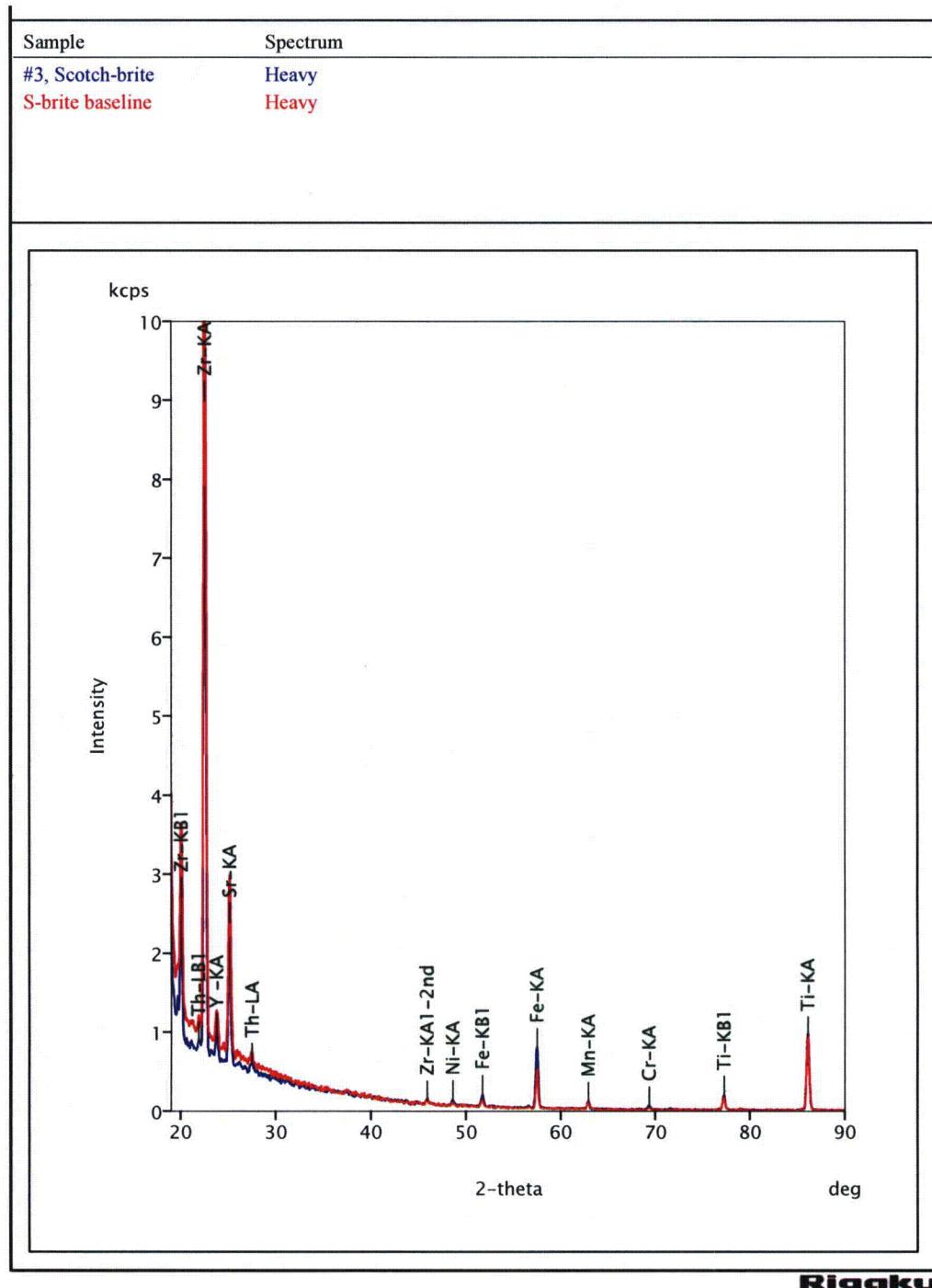


Figure 1

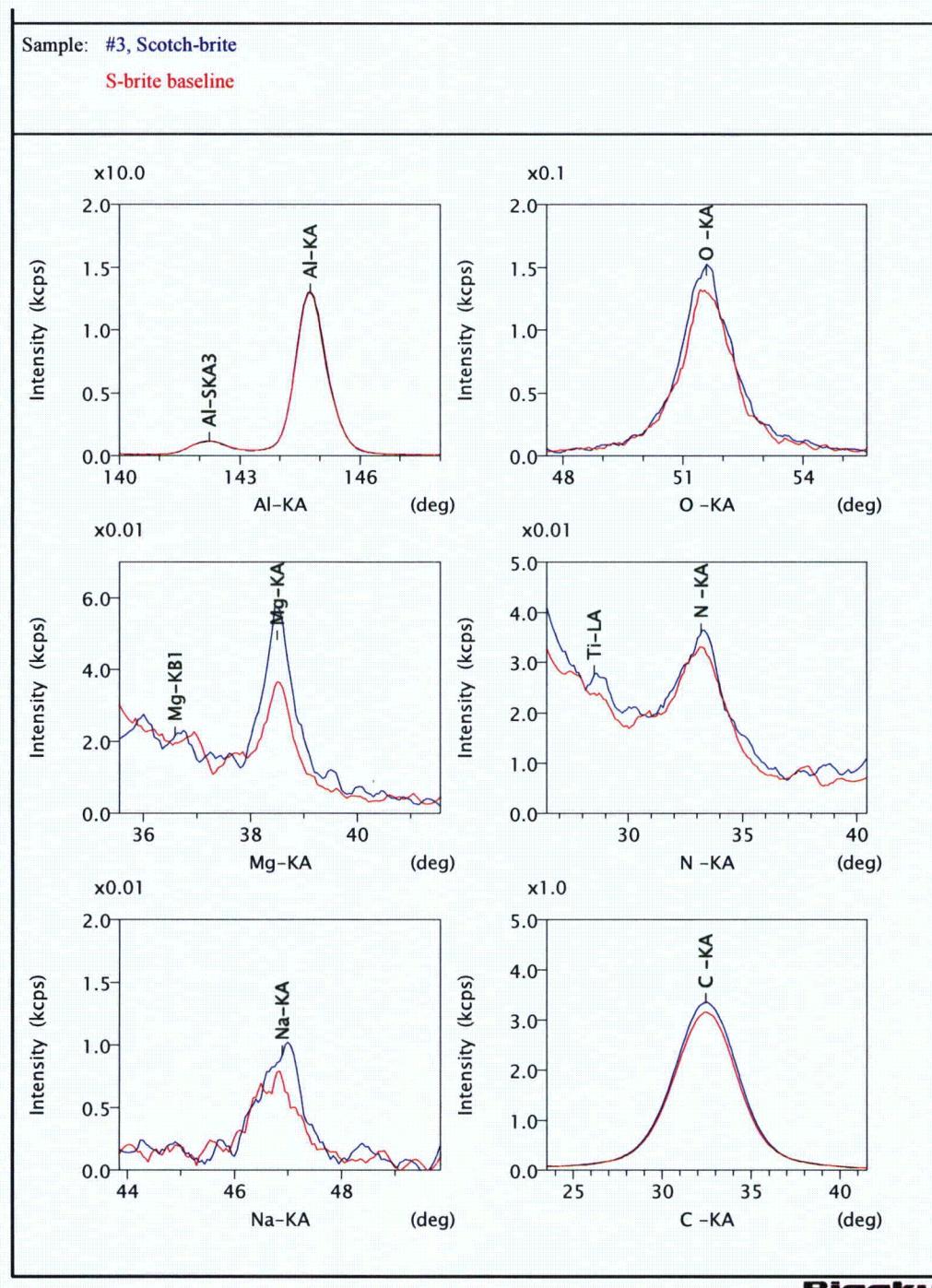
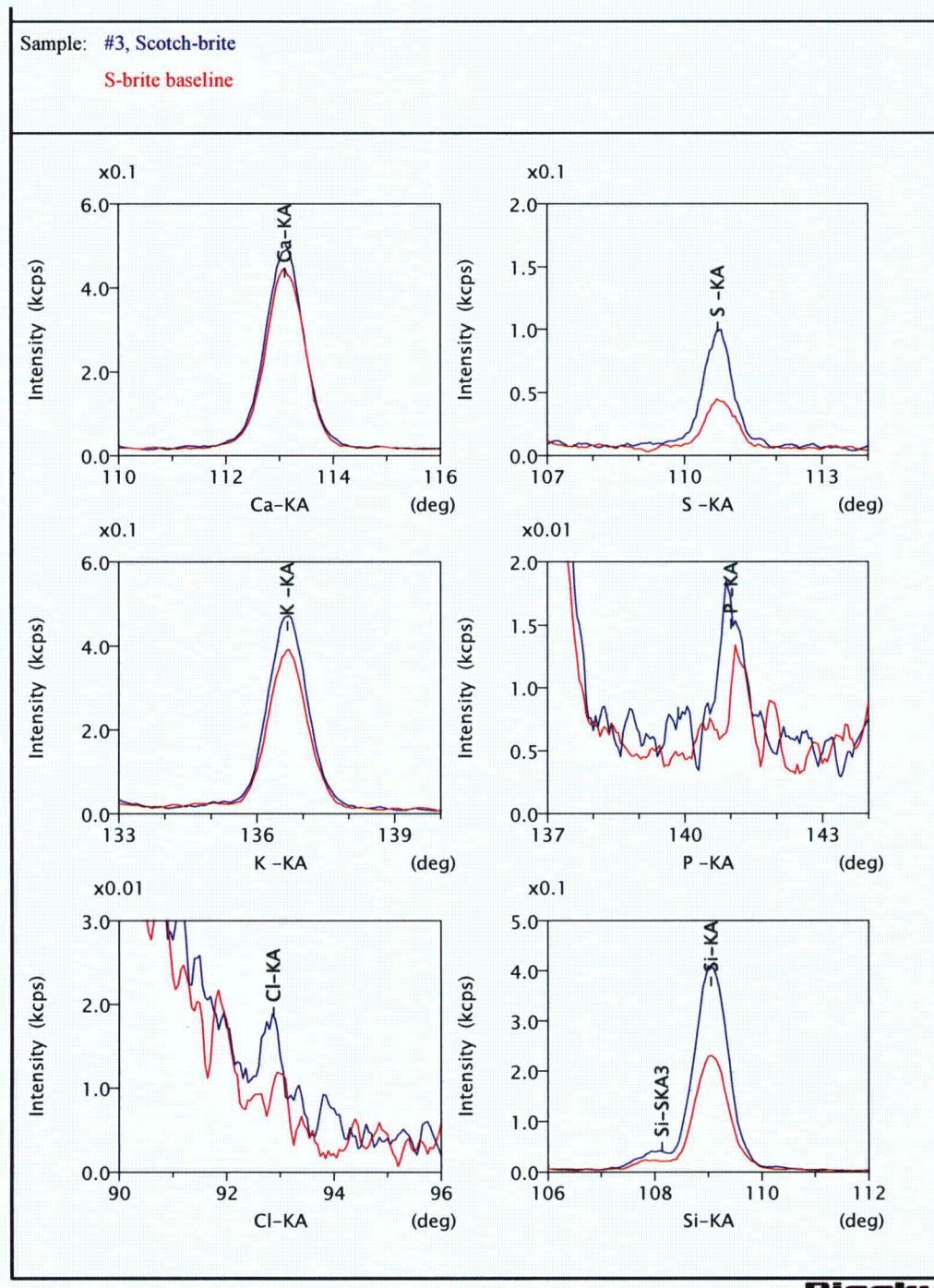


Figure 2



Rigaku

Figure 3

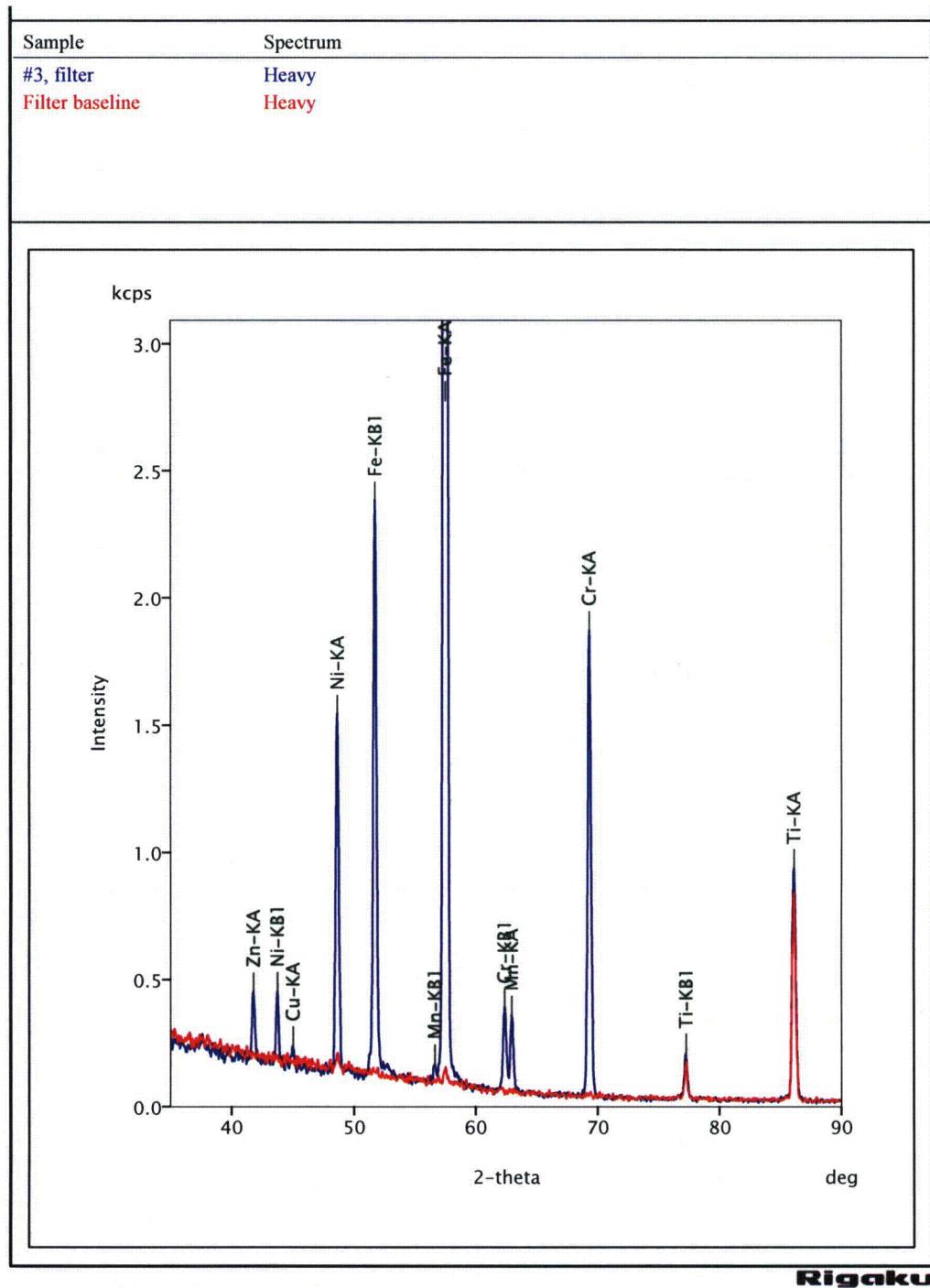
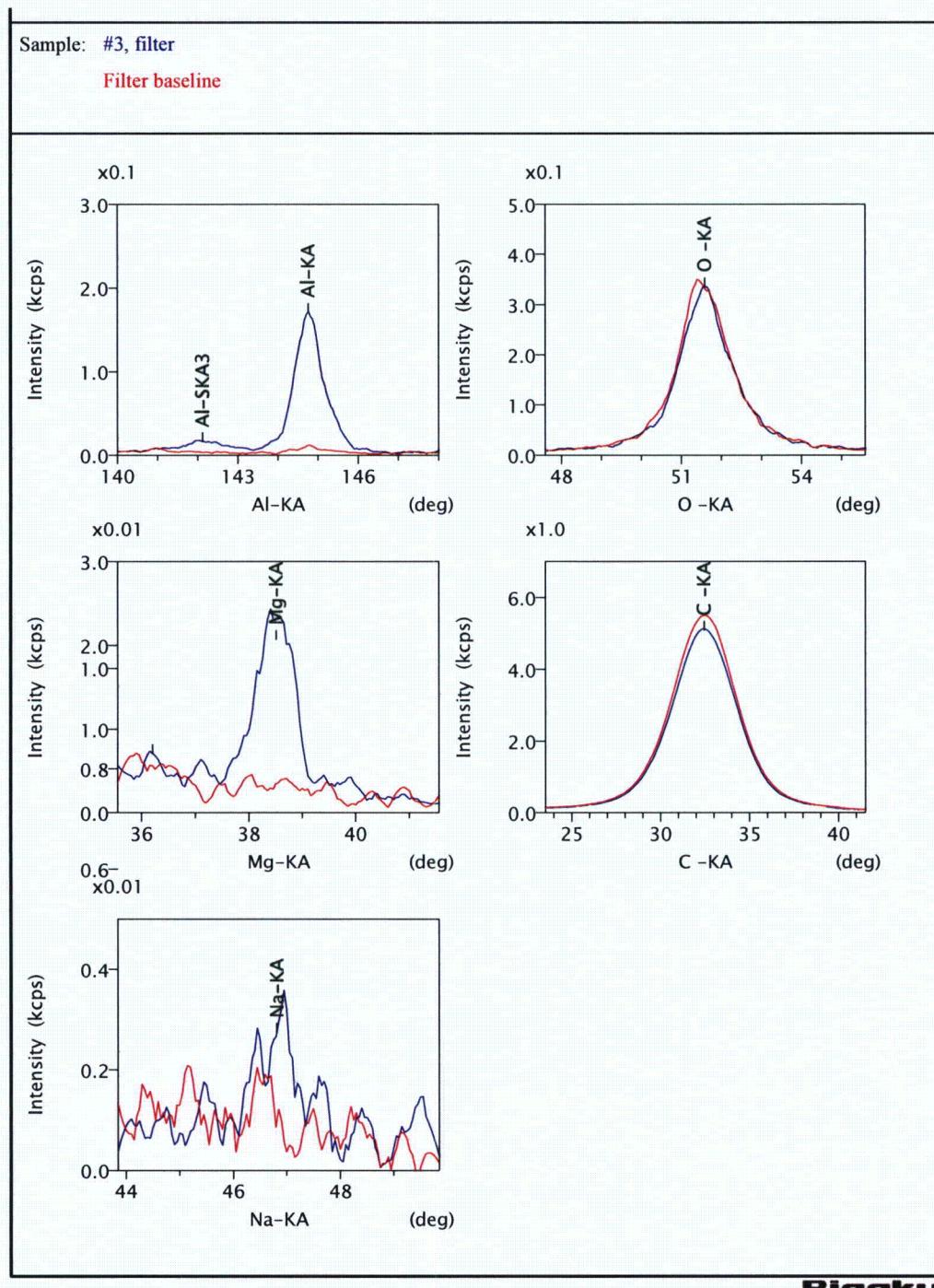


Figure 4



Rigaku

Figure 5

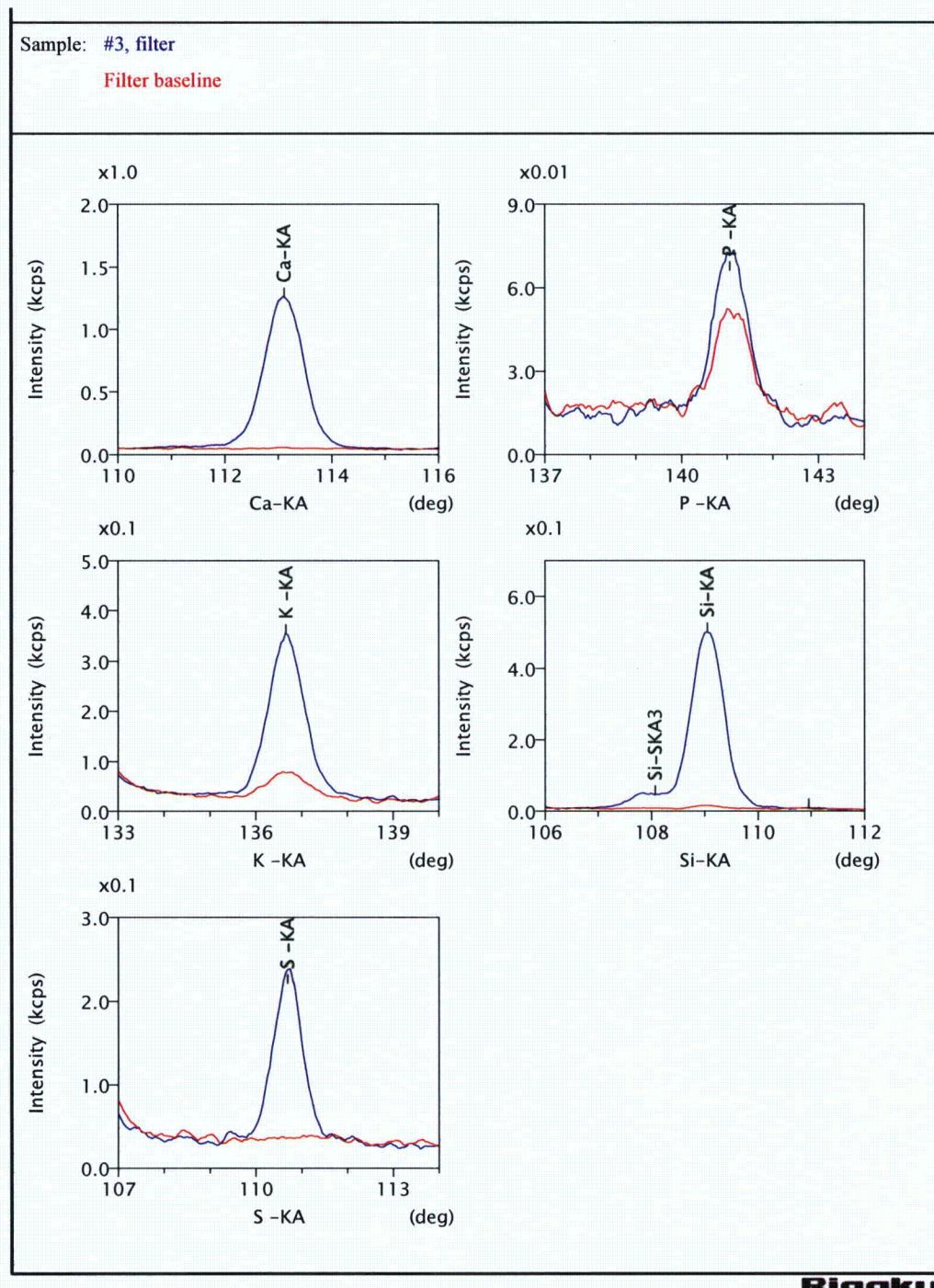


Figure 6

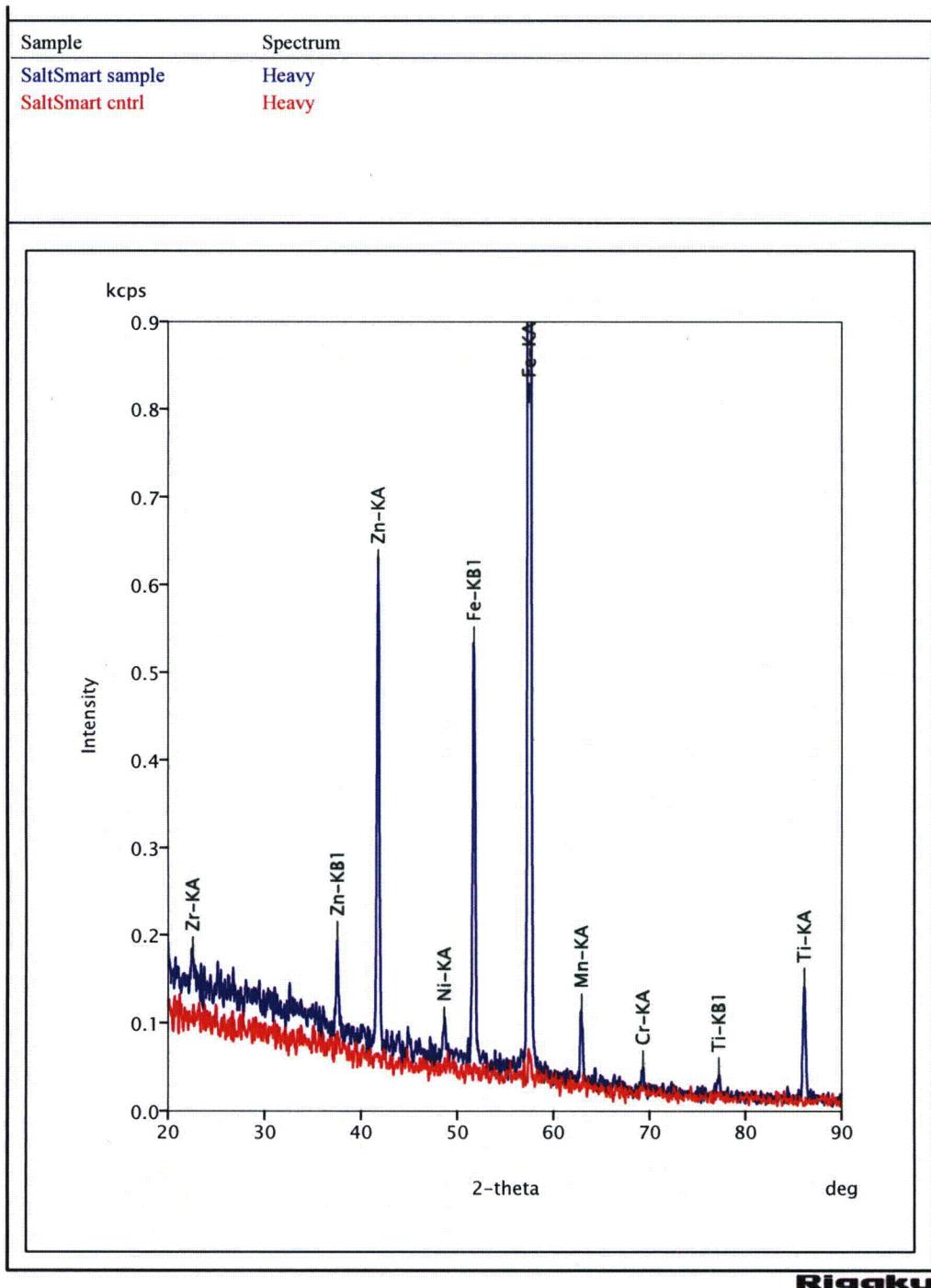
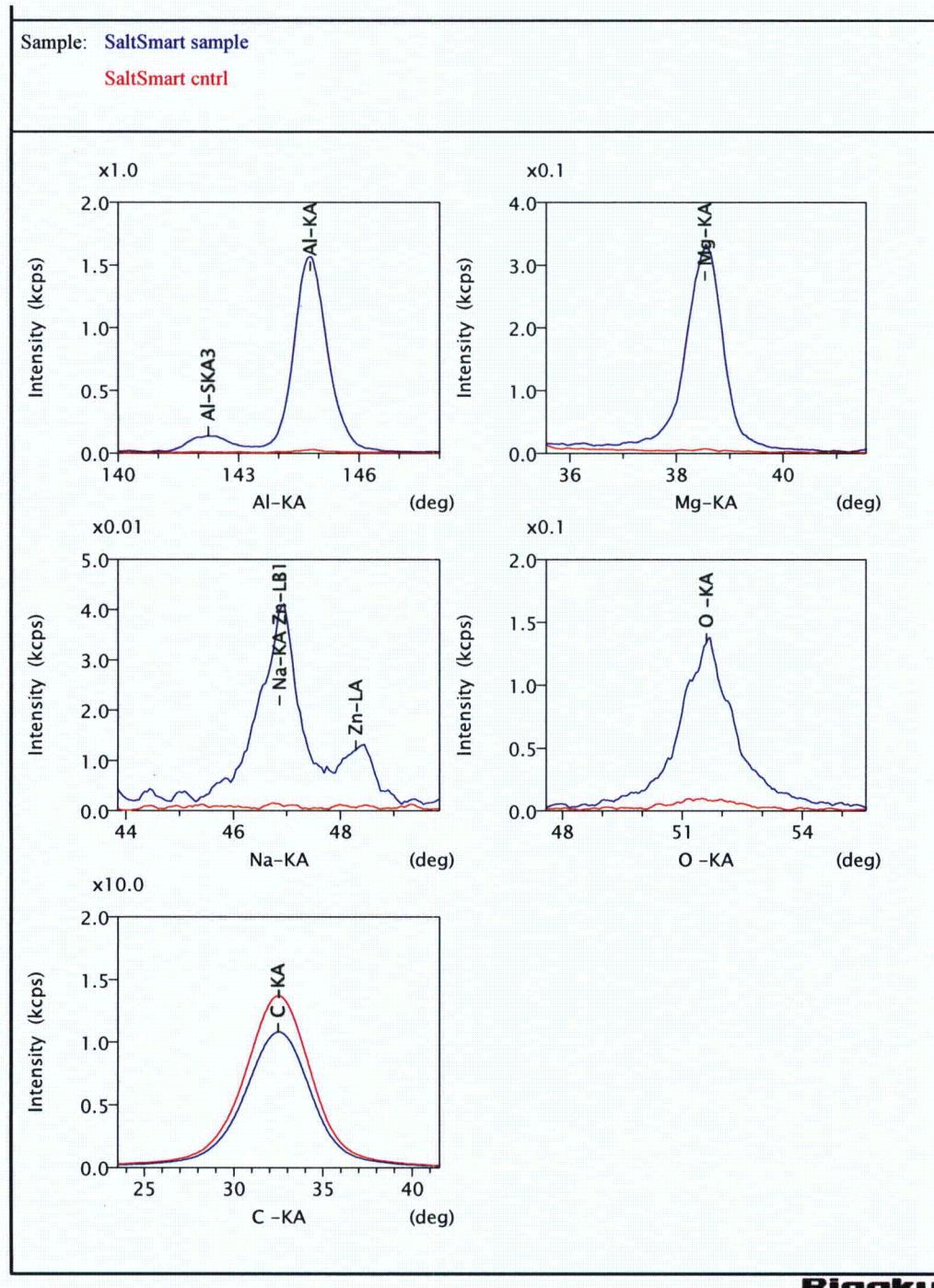


Figure 7



Rigaku

Figure 8

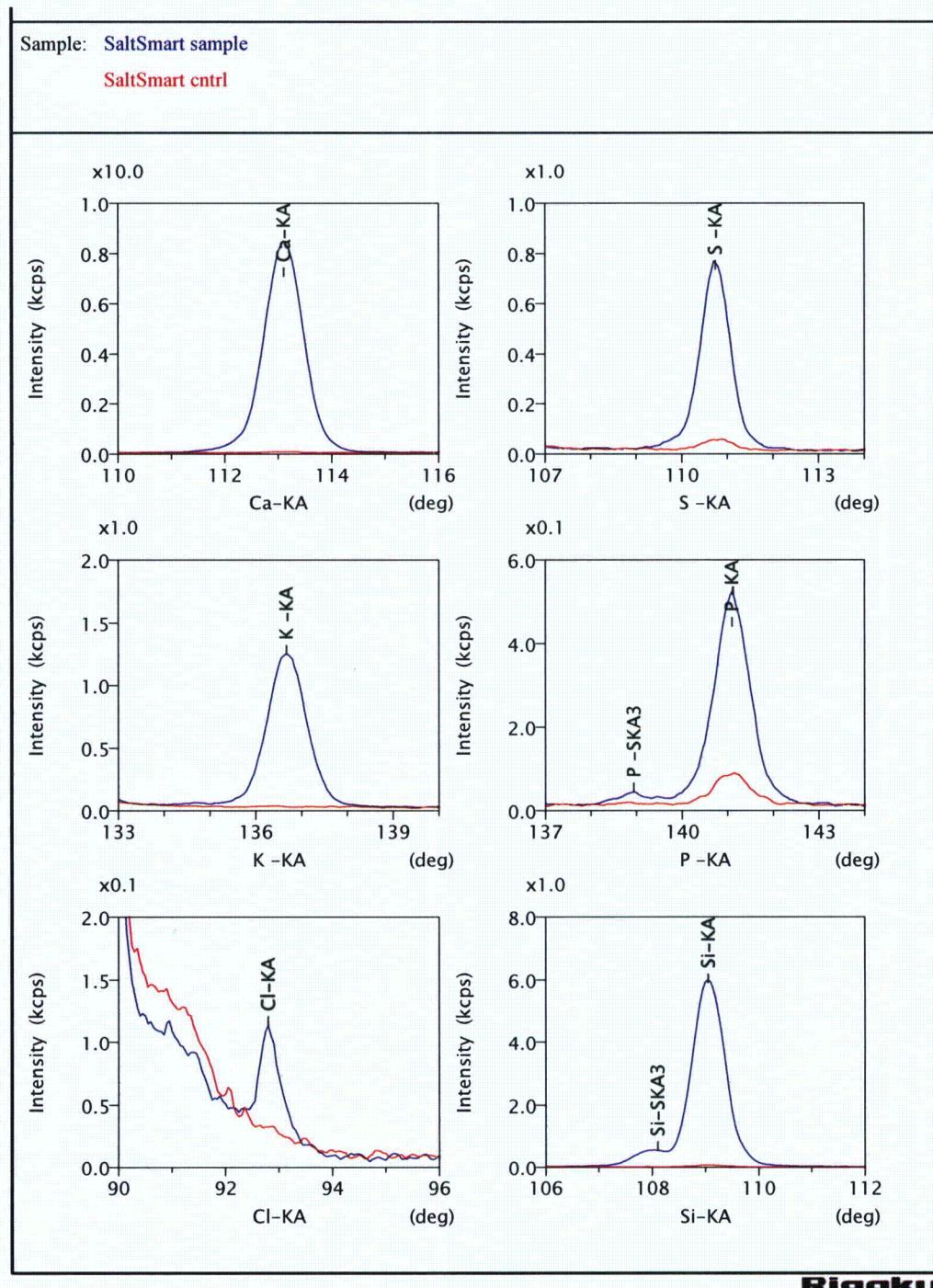


Figure 9

ATTACHMENT 4

**Gas Chromatography Mass Spectrometry (GCMS)
Volatile and Semi-volatile Materials**



Testing Cert. #2797.01

GAS CHROMATOGRAPHY MASS SPECTROMETRY (GC-MS)
ANALYSIS REPORT
21 Mar 2013

**JOB NUMBER C0DHY160
PO NUMBER 4500000443**

for

Keith Waldrop
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GC-MS ANALYSIS REPORT

Requester:
Job Number:
Analysis Date:

Keith Waldrop
C0DHY160
21 Mar 2013

Purpose:

The purpose of the analysis was to identify the contaminant collected by a Scotch-Brite with a filter and a SaltSmart. A reference control of the Scotch-Brite, the filter and the SaltSmart were provided for baseline comparison.

Summary:

Compared with the control filter, additional siloxanes were detected from the used filter. More hydrocarbons were detected from the used SaltSmart. Nothing significant was detected from the used Scotch-Brite, compared with the Scotch-Brite control. The major contaminant may have been inorganic species.

Experimental:

In order to analyze the contaminant collected by three different materials, each reference control and used material was extracted with an organic solvent methylene chloride. The methylene chloride extracts were separated from the sample material and concentrated under a stream of dry nitrogen and analyzed by the GCMS.

Prior to sample analysis, a syringe blank was analyzed. Syringe blanks were analyzed between sample analyses to avoid cross contamination. The samples were analyzed on a Hewlett-Packard 6890 Gas Chromatograph/ Hewlett-Packard 5973 Mass Spectrometer.

GC Column: 30 m 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: Splitless

Injector temperature: 300 °C

Oven temperature: 50°C (2 min.) to 150°C@10°C/min, to 310°C@20°C/min hold for 5 min at 300°C

Results and Interpretations:

Filter Control

Figure 1 is a chromatogram of the volatile organic species detected from the methylene chloride extract of the filter control. Various species were extracted from the base material. Details are tabulated in the table below.

Table 1

Filter control, DHY160-03

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
<u>1-1</u>	15.03	0.49	Diethyl phthalate	
<u>1-2</u>	16.51	0.75	Ethanediyl-bis-oxy bis-benzene	
<u>1-3</u>	17.78	0.83	Oxybenzone	
<u>1-4</u>	18.37	0.60	Hexadecanamide	Possibly from the plastic bag
<u>1-5</u>	19.21	13.30	Octadecenamide	Possibly from the plastic bag
<u>1-6</u>	20.22	2.57	Unknown	Possibly aliphatic ester
<u>1-7</u>	21.70	3.87	Unknown	Possibly aliphatic ester
<u>1-8</u>	22.87	22.62	Aromatic	
<u>1-9</u>	23.93	4.94	Long chain ester	
<u>1-10</u>	27.72	43.81	Aromatic	

Filter Used

Figure 2 is a chromatogram of the volatile organic species detected from the methylene chloride extract of the filter used. Various species were detected. Details are tabulated in the table below.

Table 2

Filter Used, DHY160-08

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
<u>2-1</u>	9.56	0.59	Cyclopentasiloxane, decamethyl-	
<u>2-2</u>	12.04	0.71	Cyclohexasiloxane, dodecamethyl-	
<u>2-3</u>	13.90	1.05	Cycloheptasiloxane, tetradecamethyl-	
<u>2-4</u>	15.20	0.87	Cyclooctasiloxane, hexadecamethyl-	
<u>2-5</u>	16.18	0.53	Cyclononasiloxane, octadecamethyl-	
<u>2-6</u>	18.49	4.81	Benzene dicarboxylic acid, bis-hydroxy-ethyl ester	
<u>2-7</u>	19.19	4.28	Octadecenamide	Possibly from the plastic bag
<u>2-8</u>	20.22	5.04	Unknown	Possibly aliphatic ester
<u>2-9</u>	21.70	6.61	Unknown	Possibly aliphatic ester
<u>2-10</u>	22.87	27.81	Aromatic	
<u>2-11</u>	23.93	6.73	Unknown	Possibly aliphatic ester
<u>2-12</u>	27.72	35.67	Aromatic	

Figure 3 is a chromatogram comparison of the filter used (top in blue) with the filter control (bottom in red). The comparison indicates that more siloxanes were detected from the used filter sample.

Scotch-Brite Control

Figure 4 is a chromatogram of the volatile organic species detected from the methylene chloride extract of the Scotch-Brite control. Various species were extracted from the base material. Details are tabulated in the table below.

Table 3
Scotch-Brite control, DHY160-14

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
<u>4-1</u>	14.67	2.31	Unknown	Probably multi hydroxyl species
<u>4-2</u>	15.03	6.50	Diethyl phthalate	
<u>4-3</u>	16.42	14.25	Probably glycol type	
<u>4-4</u>	16.51	5.76	Ethanediyl-bis-oxy-bis benzene	
<u>4-5</u>	16.99	2.29	Homosalate	
<u>4-6</u>	18.83	4.12	Diazacyclotetradecane-dione	
<u>4-7</u>	20.03	3.03	Probably aliphatic ester	
<u>4-8</u>	22.79	7.13	Probably aliphatic ester	
<u>4-9</u>	24.37	6.33	Probably aliphatic ester	

Scotch-Brite Used

Figure 5 is a chromatogram of the volatile organic species detected from the methylene chloride extract of the Scotch-Brite used. Various species were detected. Details are tabulated in the table below.

Table 4
Scotch-Brite Used, DHY160-10

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
<u>5-1</u>	13.41	1.34	Similar to tetramethyl decyndiol	
<u>5-2</u>	14.36	1.00	Dimethyl ethyl phenol	
<u>5-3</u>	15.03	6.01	Diethyl phthalate	
<u>5-4</u>	16.41	15.17	Probably glycol type	
<u>5-5</u>	16.54	4.01	Probably glycol type	
<u>5-6</u>	16.99	1.45	Homosalate	
<u>5-7</u>	18.82	5.00	Diazacyclotetradecane-dione	
<u>5-8</u>	19.19	7.79	Octadecenamide	
<u>5-9</u>	20.03	3.24	Probably aliphatic ester	

Figure 6 is a chromatogram comparison of the Scotch-Brite used (top in blue) with the Scotch-Brite control (bottom in red). The comparison indicates that the two samples were essentially identical. Tetramethyl decyndiol or similar compound with a peak retention time of 13.41 was higher in the used Scotch-Brite sample.

SaltSmart Control

Figure 7 is a chromatogram of the volatile organic species detected from the methylene chloride extract of the SaltSmart control. Various species were extracted from the base material. Details are tabulated in the table below.

Table 5

SaltSmart Control, DHY160-16

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
<u>7-1</u>	9.56	2.37	Cyclopentasiloxane, decamethyl-	
<u>7-2</u>	12.04	3.54	Cyclohexasiloxane, dodecamethyl	
<u>7-3</u>	13.90	3.16	Cycloheptasiloxane, tetradecamethyl-	
<u>7-4</u>	15.20	2.72	Cyclooctasiloxane, hexadecamethyl-	
<u>7-5</u>	16.18	1.88	Cyclononasiloxane, octadecamethyl-	
<u>7-6</u>	19.16	5.78	Unknown	
<u>7-7</u>	19.31	17.89	Possibly similar to propylene glycol	
<u>7-8</u>	20.15	10.26	Unknown	Possibly crown ether
<u>7-9</u>	20.50	7.93	Probably ethylene glycol type	
<u>7-10</u>	21.14	7.50	Erucamide	Possibly from the packaging bag

SaltSmart Used

Figure 8 is a chromatogram of the volatile organic species detected from the methylene chloride extract of the SaltSmart used. Particles were washed off from the contaminated sample but not dissolved in the methylene chloride extract. This indicated the major contaminant was inorganic. Various species were detected from the extract. Details are tabulated in the table below.

Table 6

SaltSmart Used, DHY160-20

Spectr. No.	RT	% of total area	Compound Identification	Miscellaneous Information
<u>8-1</u>	9.57	1.77	Cyclopentasiloxane, decamethyl-	
<u>8-2</u>	12.04	3.92	Cyclohexasiloxane, dodecamethyl	
<u>8-3</u>	13.41	2.27	Probably tetramethyl decyndiol	
<u>8-4</u>	13.90	4.19	Cycloheptasiloxane, tetradecamethyl-	
<u>8-5</u>	14.36	<1	Dimethyl ethyl Phenol	
<u>8-6</u>	15.20	3.11	Cyclooctasiloxane, hexadecamethyl-	
<u>8-7</u>	15.29	1.91	Unknown	Probably similar to tetramethyl decyndiol
<u>8-8</u>	16.18	2.51	Cyclononasiloxane, octadecamethyl-	
<u>8-9</u>	16.79	2.30	Unknown	Possibly ethylene glycol type
<u>8-10</u>	19.20	4.70	Hydrocarbon	
<u>8-11</u>	19.90	4.59	Hydrocarbon	Mixed with DOP

Figures 9 and 10 are comparisons of figure 8 (top in blue) and figure 7 (bottom in red). The comparisons indicate that dimethyl ethyl phenol, tetramethyl decyndiol and a similar

component were detected from the used SaltSmart. The raised baseline, in the used SaltSmart chromatogram, after retention time 20 minutes indicated presence of hydrocarbons.

The amounts listed in the tables above were based on peak areas only, and are semi-quantitative. No calibration curves were prepared and no attempt was made to correct for response factor differences between species that are structurally/functionally different. Typical reproducibility as determined by statistical process control of the measurement system is about 10% (at 95% confidence level, $k \sim 2$).

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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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.

```
File      :Y:\March13\DHY16003.D
Operator   : X. CAI
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Instrument  : GC/MS Ins
Sample Name: Filter control concentrated
Misc Info  : 1 ul
Vial Number: 1
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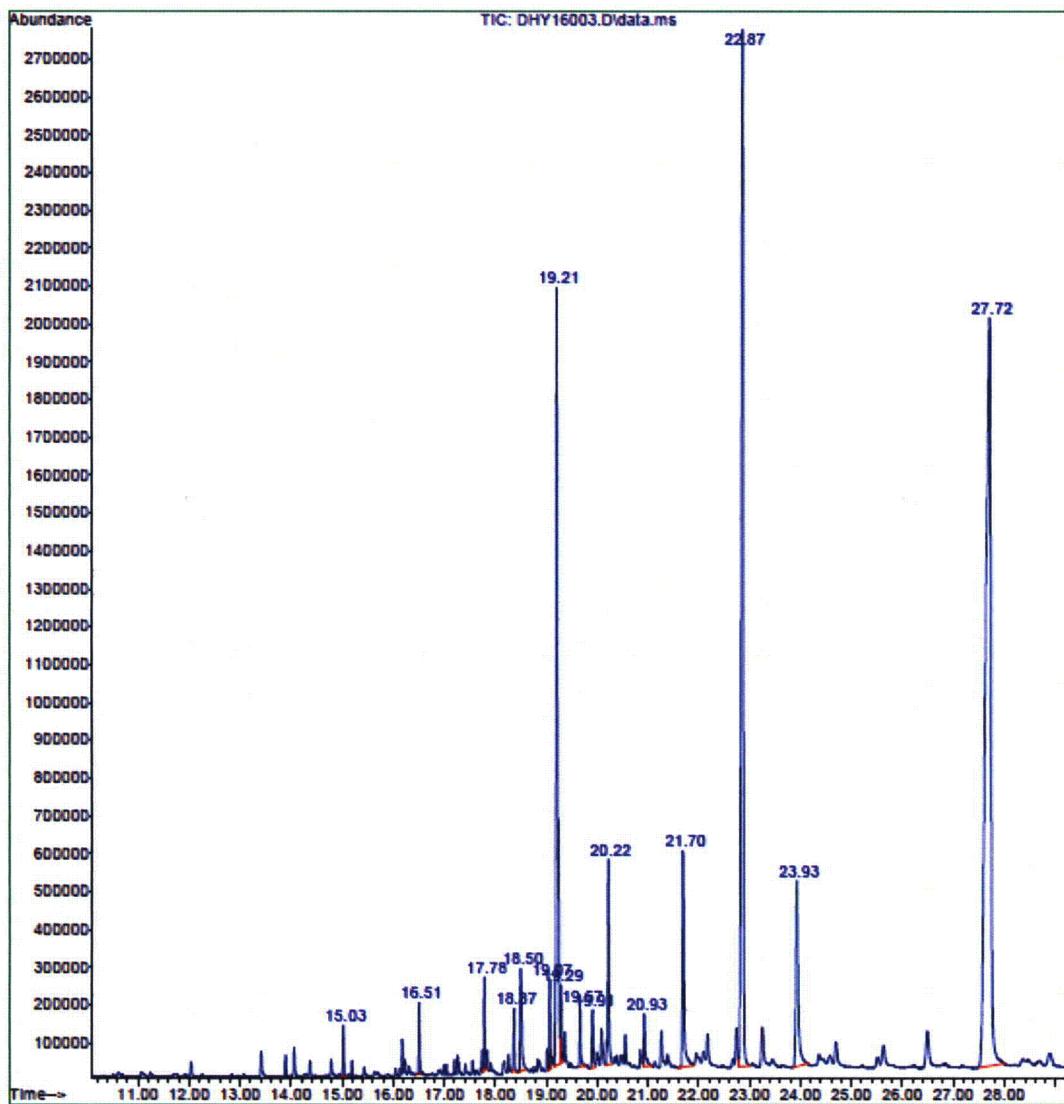
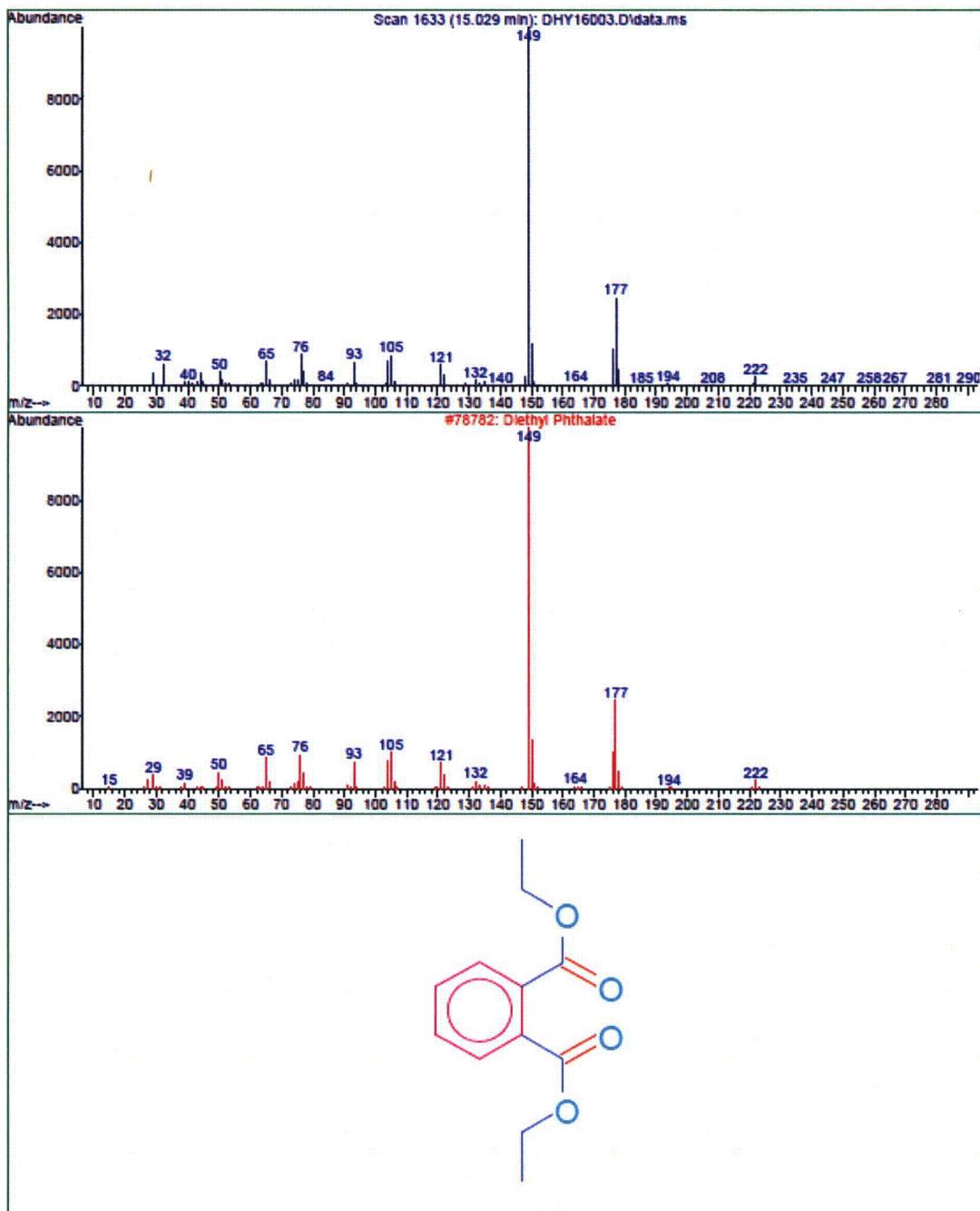


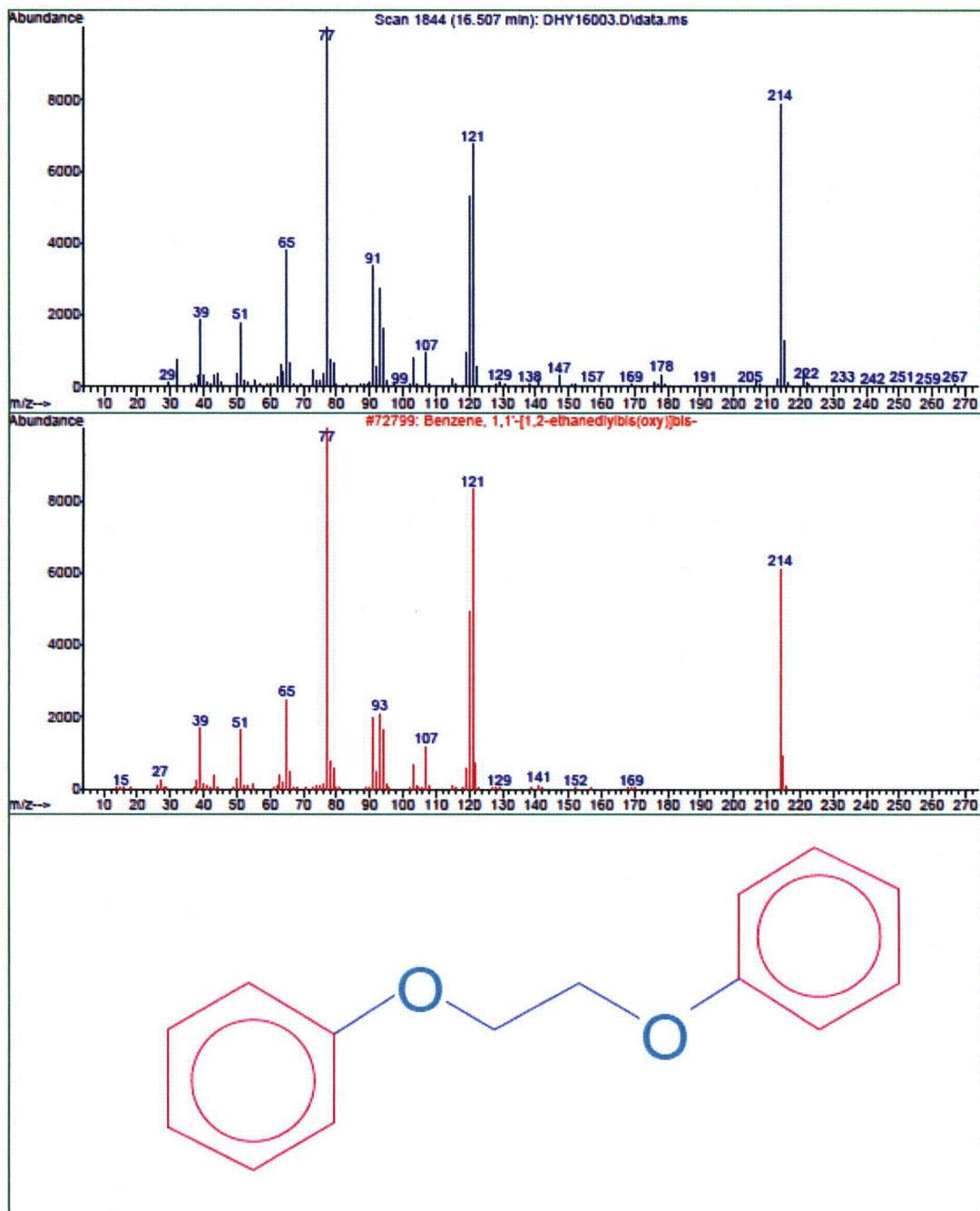
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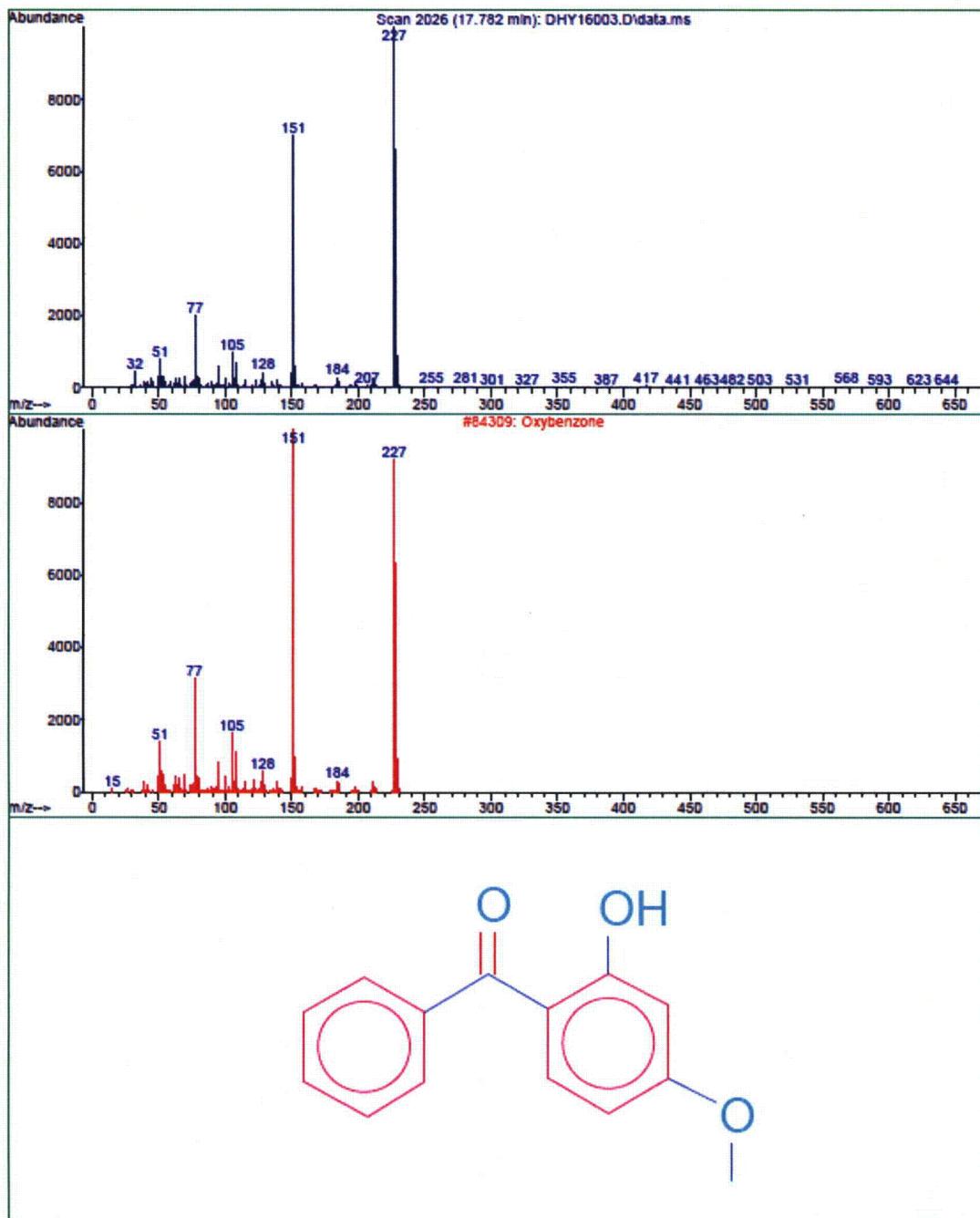
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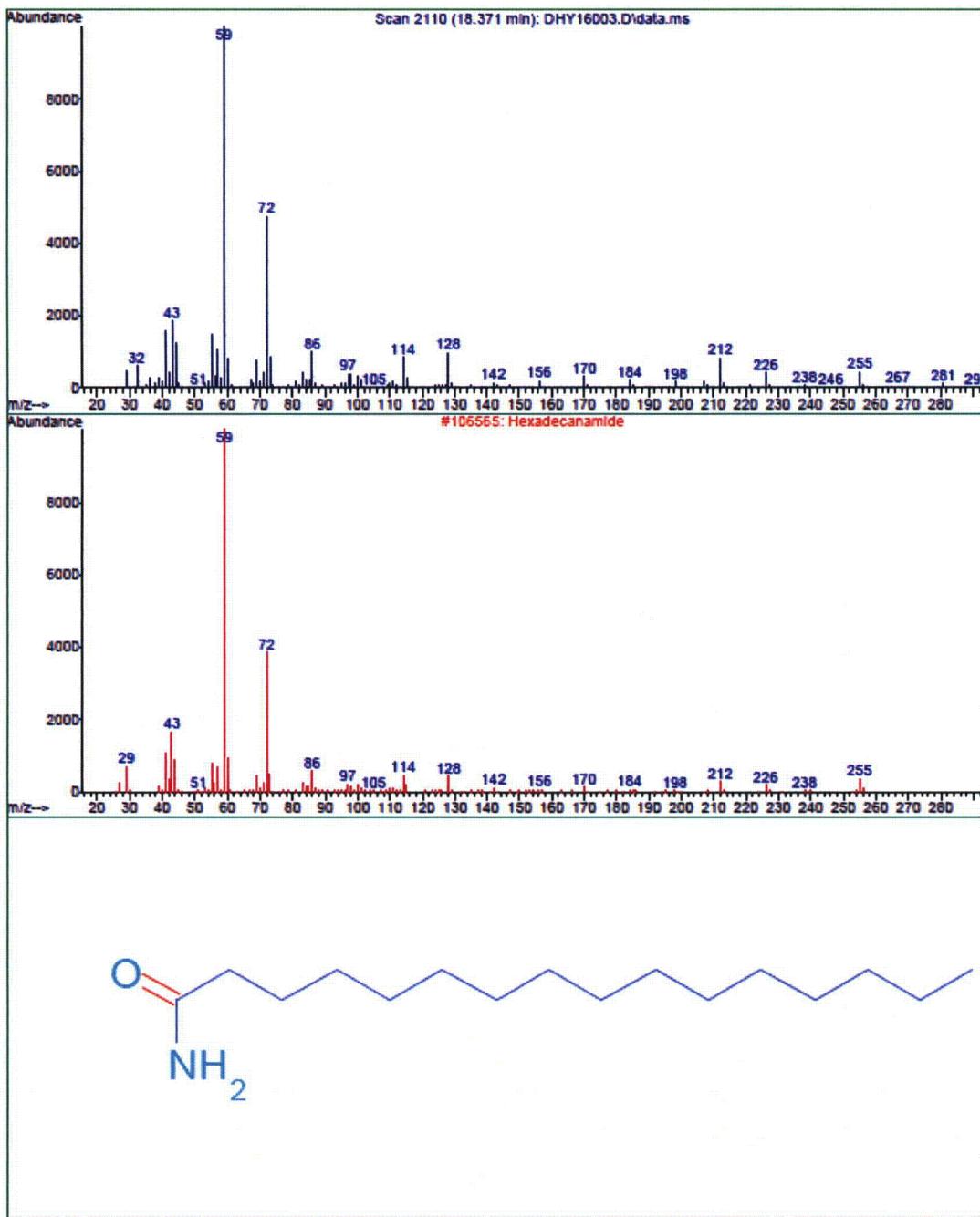
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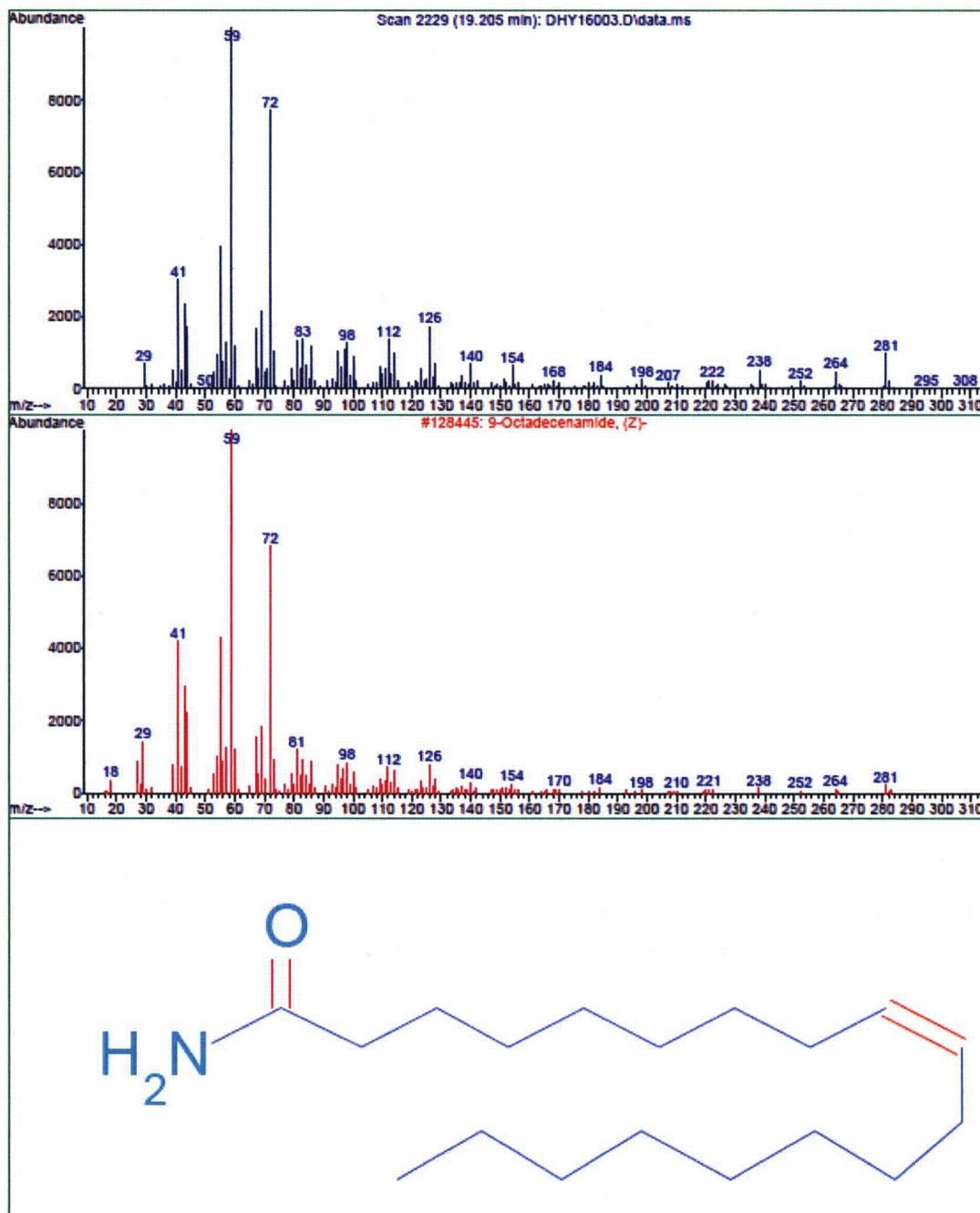
Spectrum 1-3

Library Searched : C:\Database\NIST11.L
Quality : 97
ID : Hexadecanamide



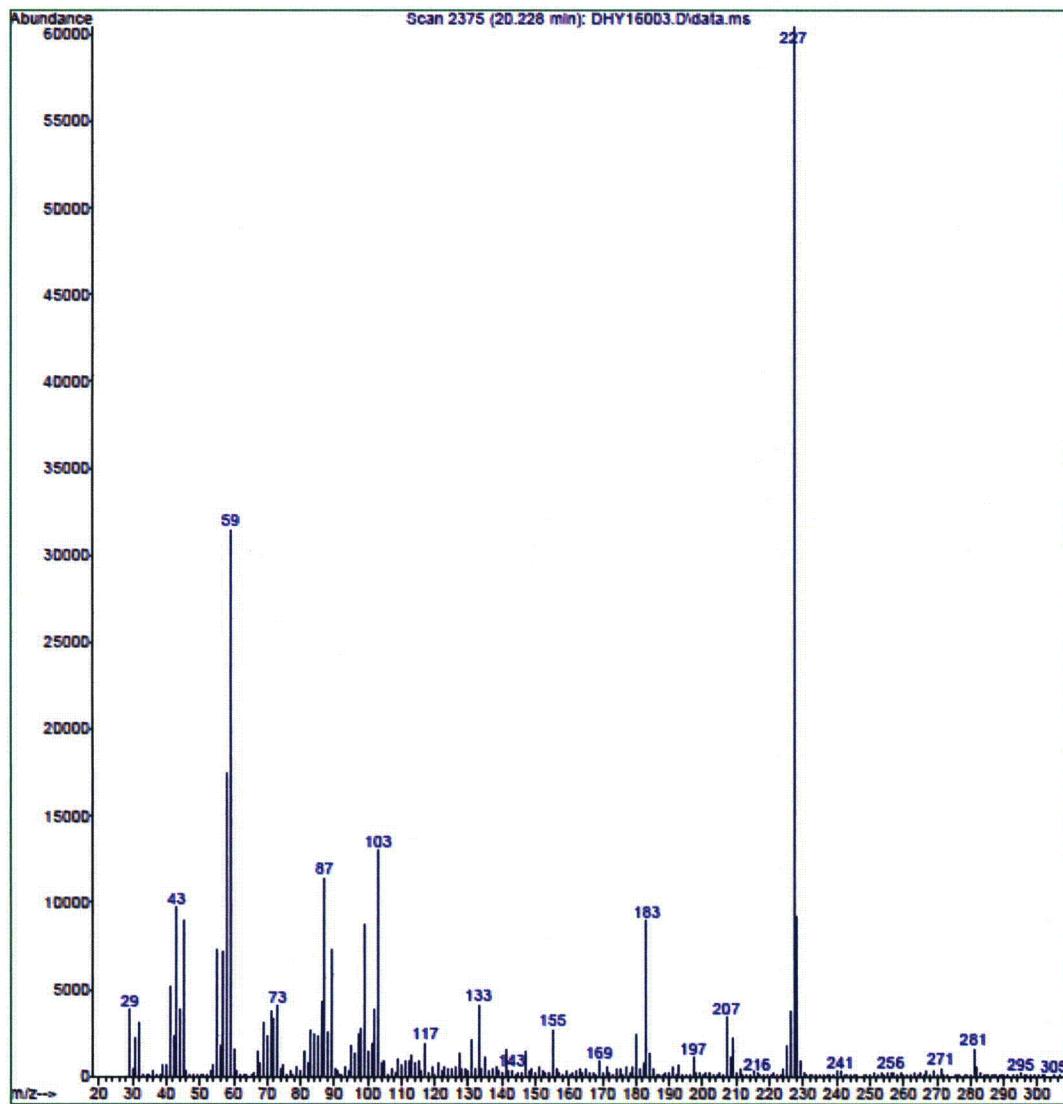
Spectrum 1-4

Library Searched : C:\Database\NIST11.L
Quality : 98
ID : 9-Octadecenamide, (Z)-



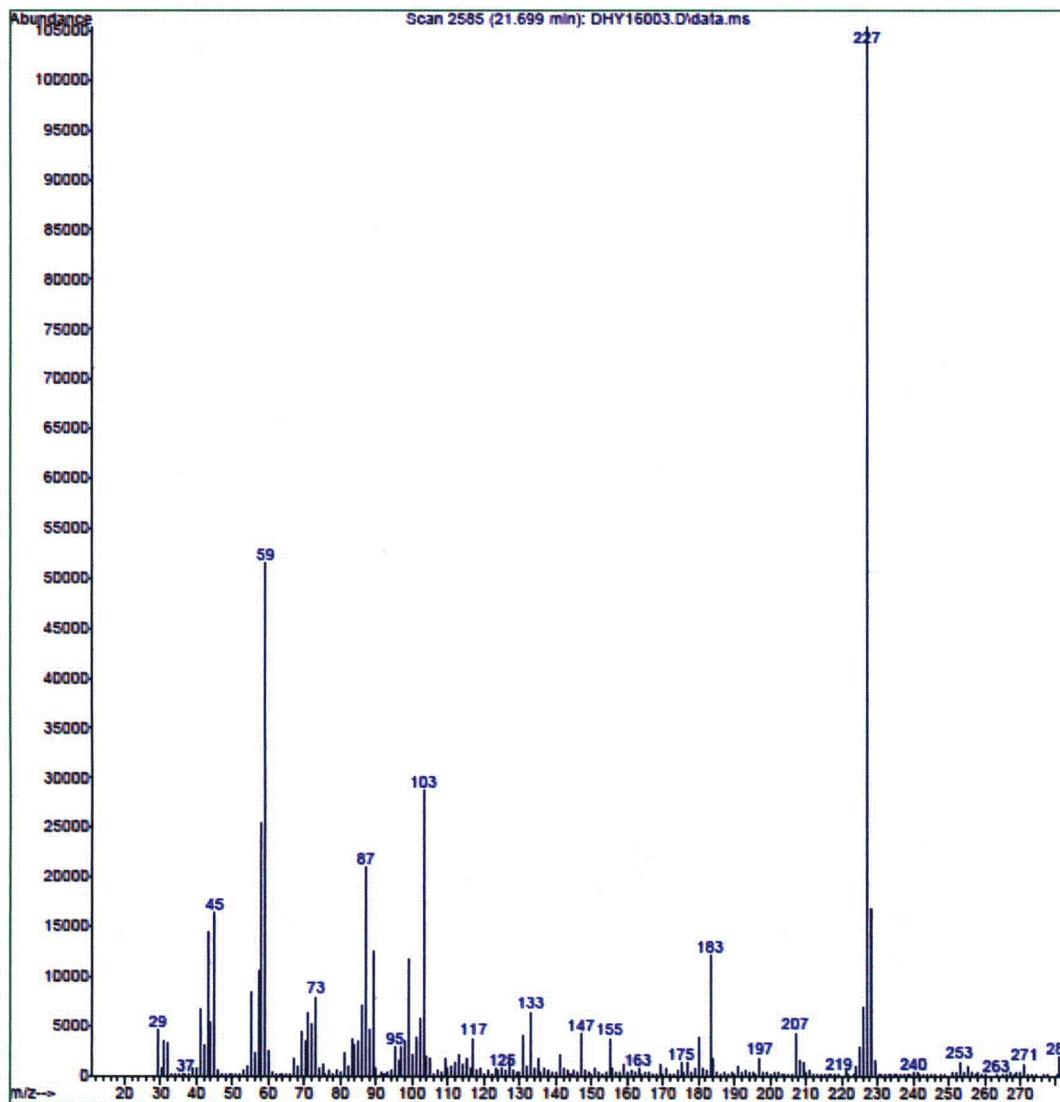
Spectrum 1-5

File :Y:\March13\DHY16003.D
Operator : X. CAI
Acquired : 19 Mar 2013 13:16 using AcqMethod STDSP^L
Instrument : GC/MS Ins
Sample Name: Filter control concentrated
Misc Info : 1 ul
Vial Number: 1



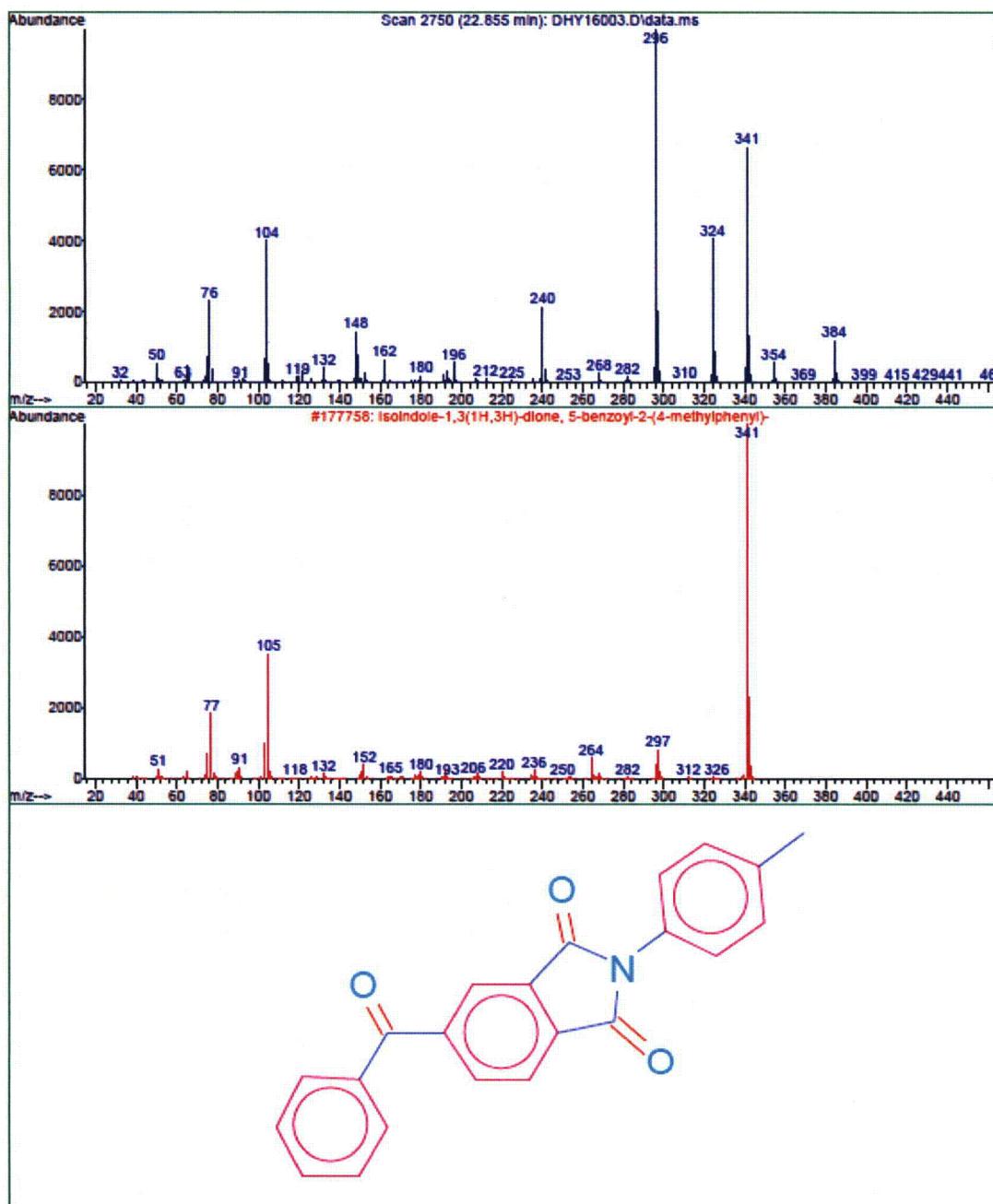
Spectrum 1-6

File : Y:\March13\DHY16003.D
Operator : X. CAI
Acquired : 19 Mar 2013 13:16 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Filter control concentrated
Misc Info : 1 ul
Vial Number: 1



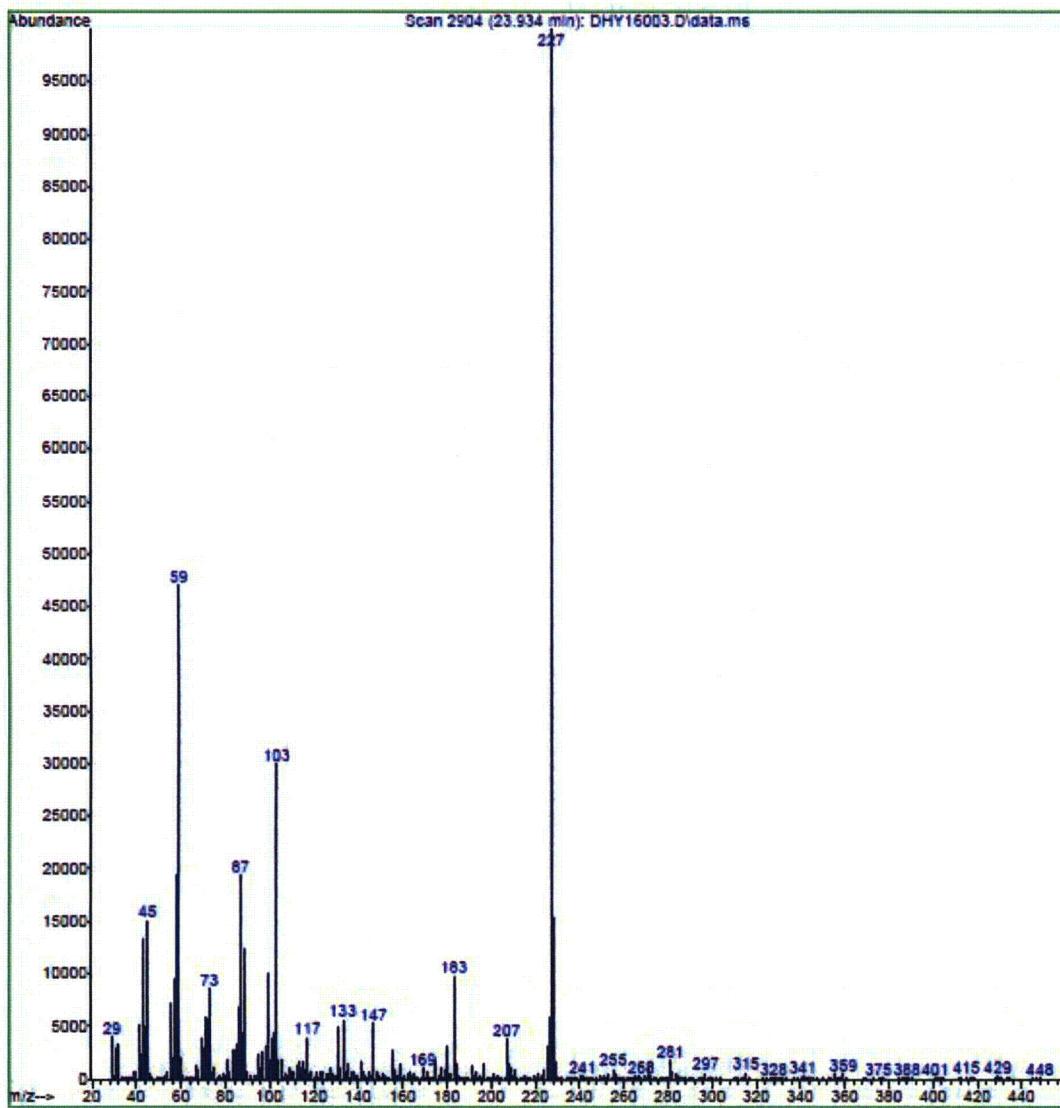
Spectrum 1-7

Library Searched : C:\Database\NIST11.L
Quality : 10
ID : Isoindole-1,3(1H,3H)-dione, 5-benzoyl-2-(4-methylphenyl)-



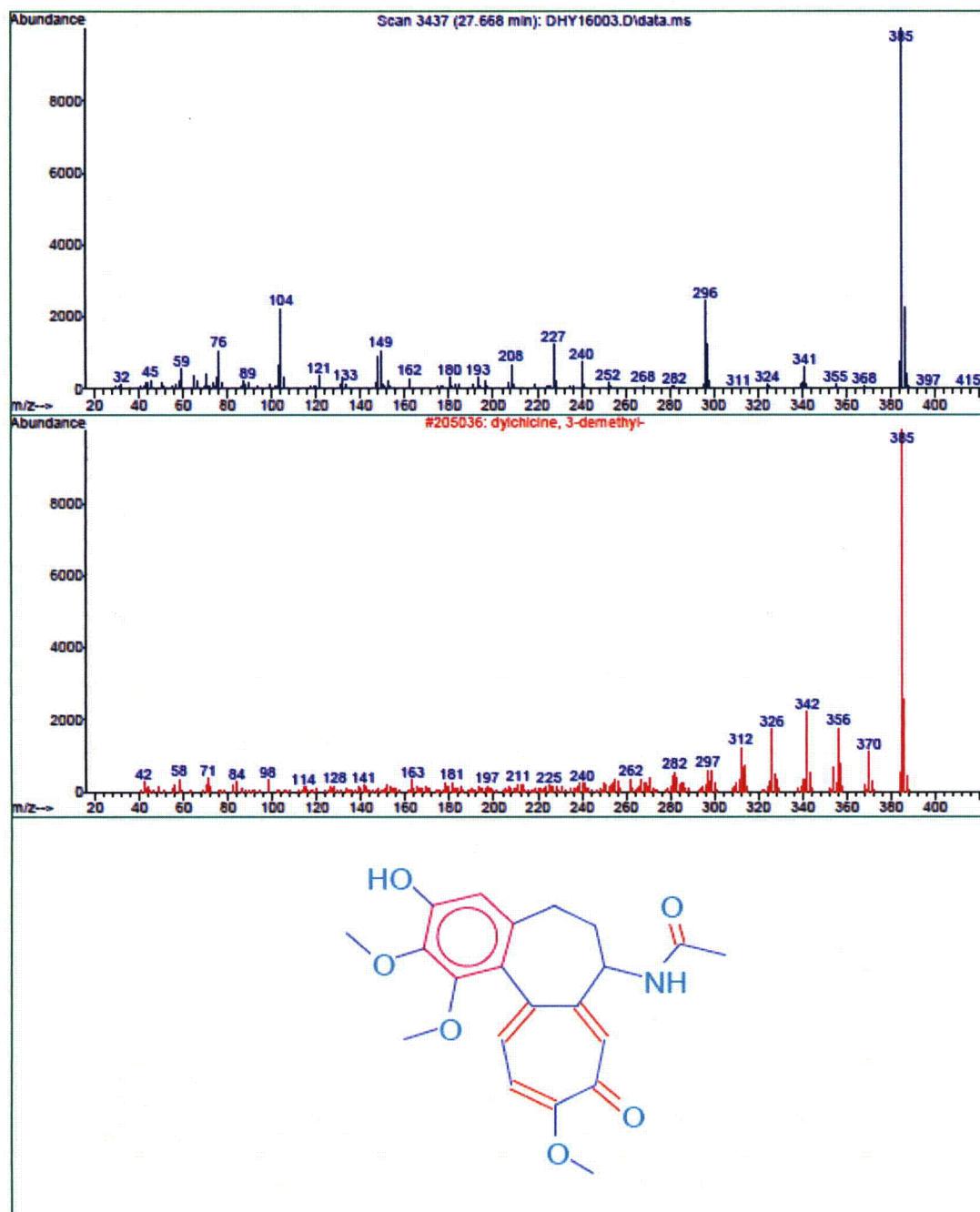
Spectrum 1-8

File : Y:\March13\DHY16003.D
Operator : X. CAI
Acquired : 19 Mar 2013 13:16 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Filter control concentrated
Misc Info : 1 ul
Vial Number: 1



Spectrum 1-9

Library Searched : C:\Database\NIST11.L
Quality : 38
ID : dylchicine, 3-demethyl-



Spectrum 1-10

```
File      : Y:\March13\DHY16008.D
Operator   : X. CAI
Acquired   : 20 Mar 2013  9:33      using AcqMethod STDSPL
Instrument  : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info  : 1 ul
Vial Number: 1
```

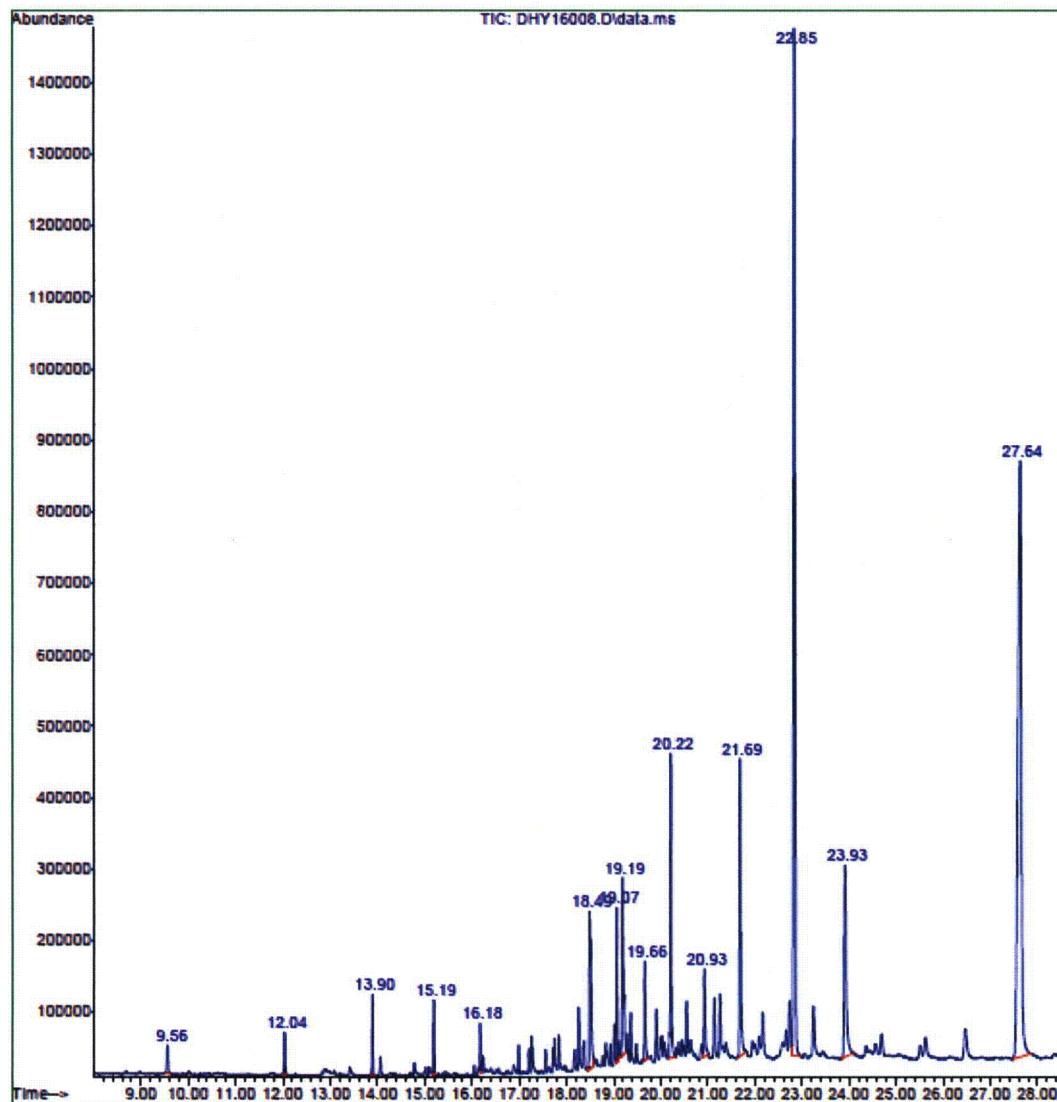
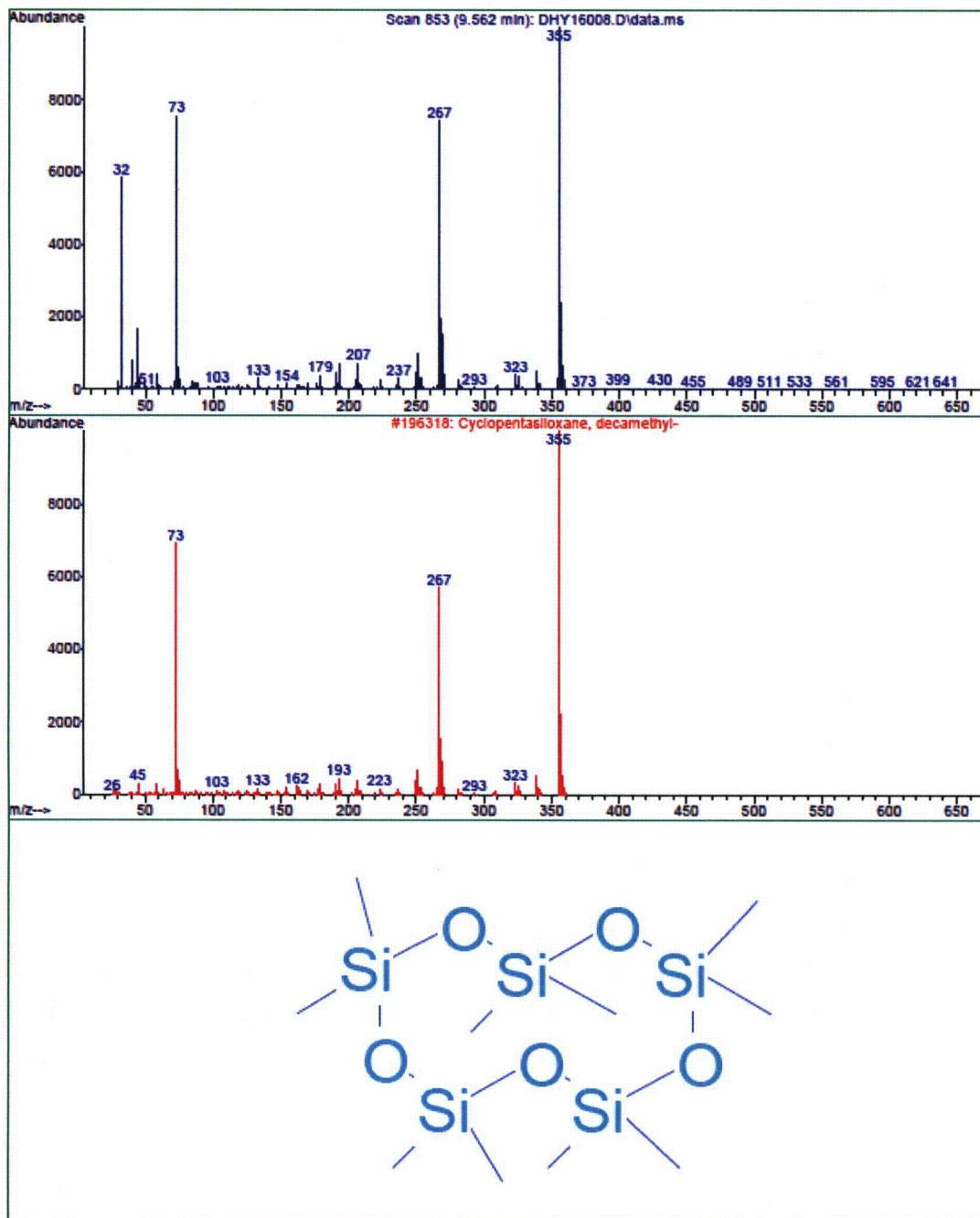


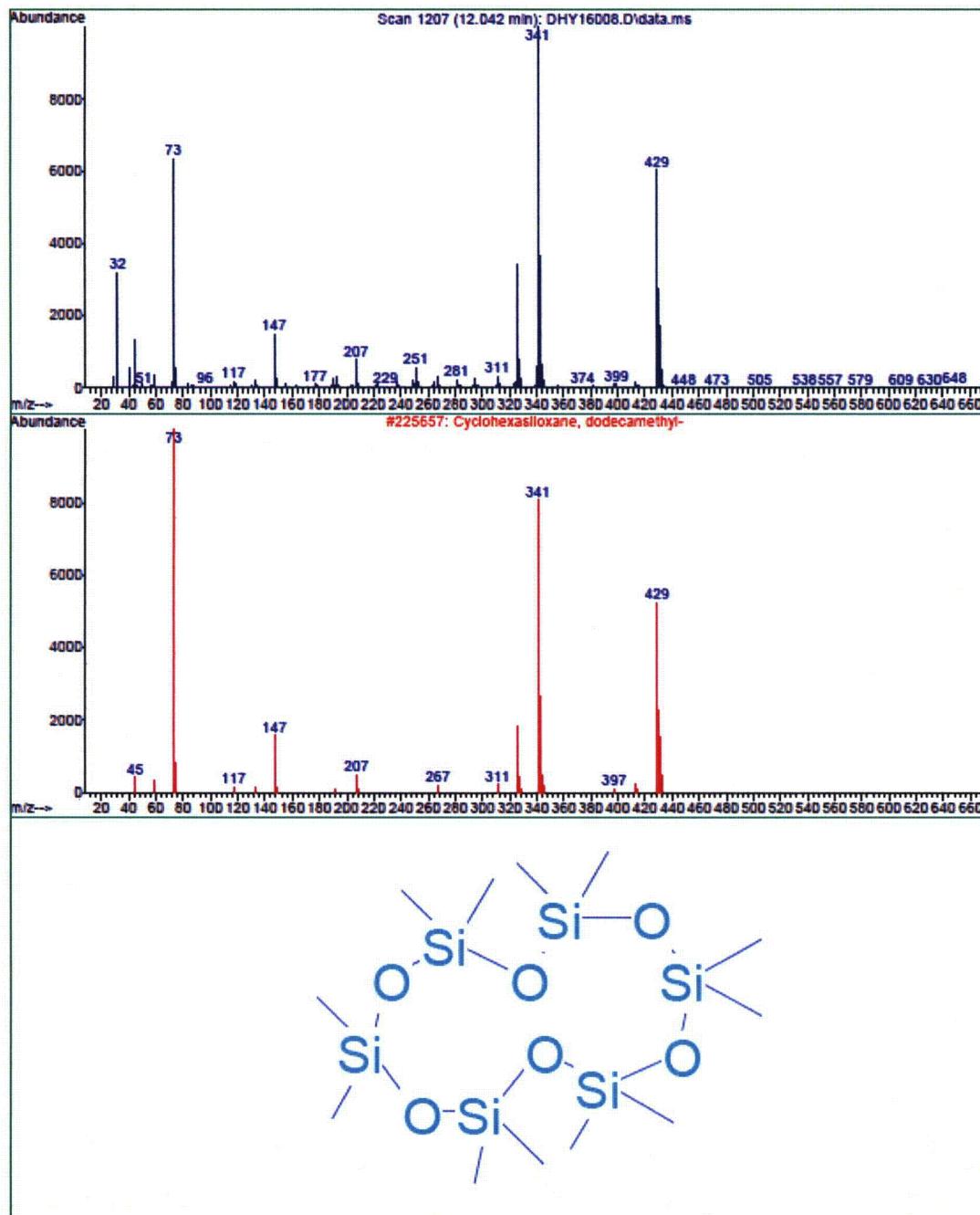
Figure 2

Library Searched : C:\Database\NIST11.L
Quality : 91
ID : Cyclopentasiloxane, decamethyl-

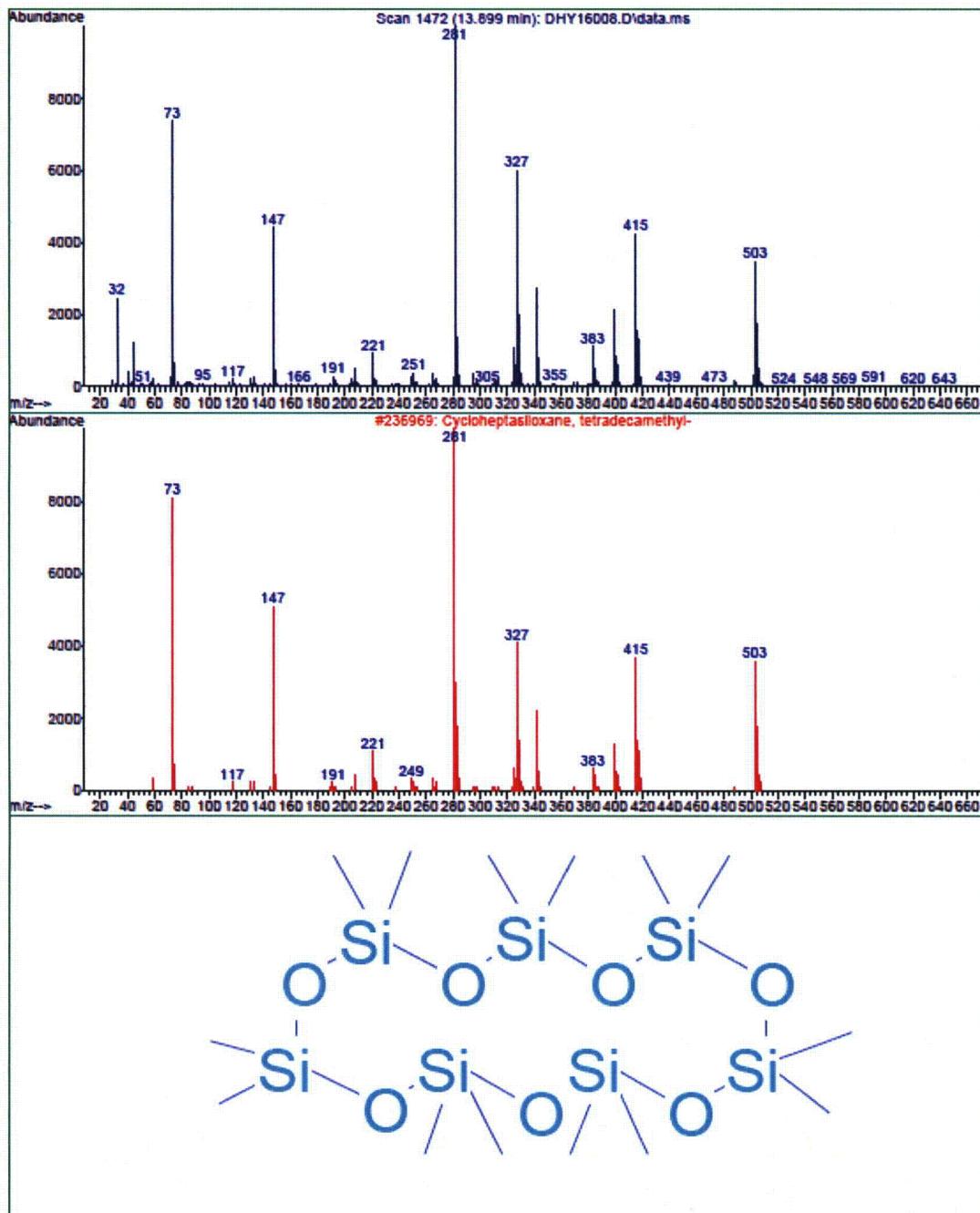


Spectrum 2-1

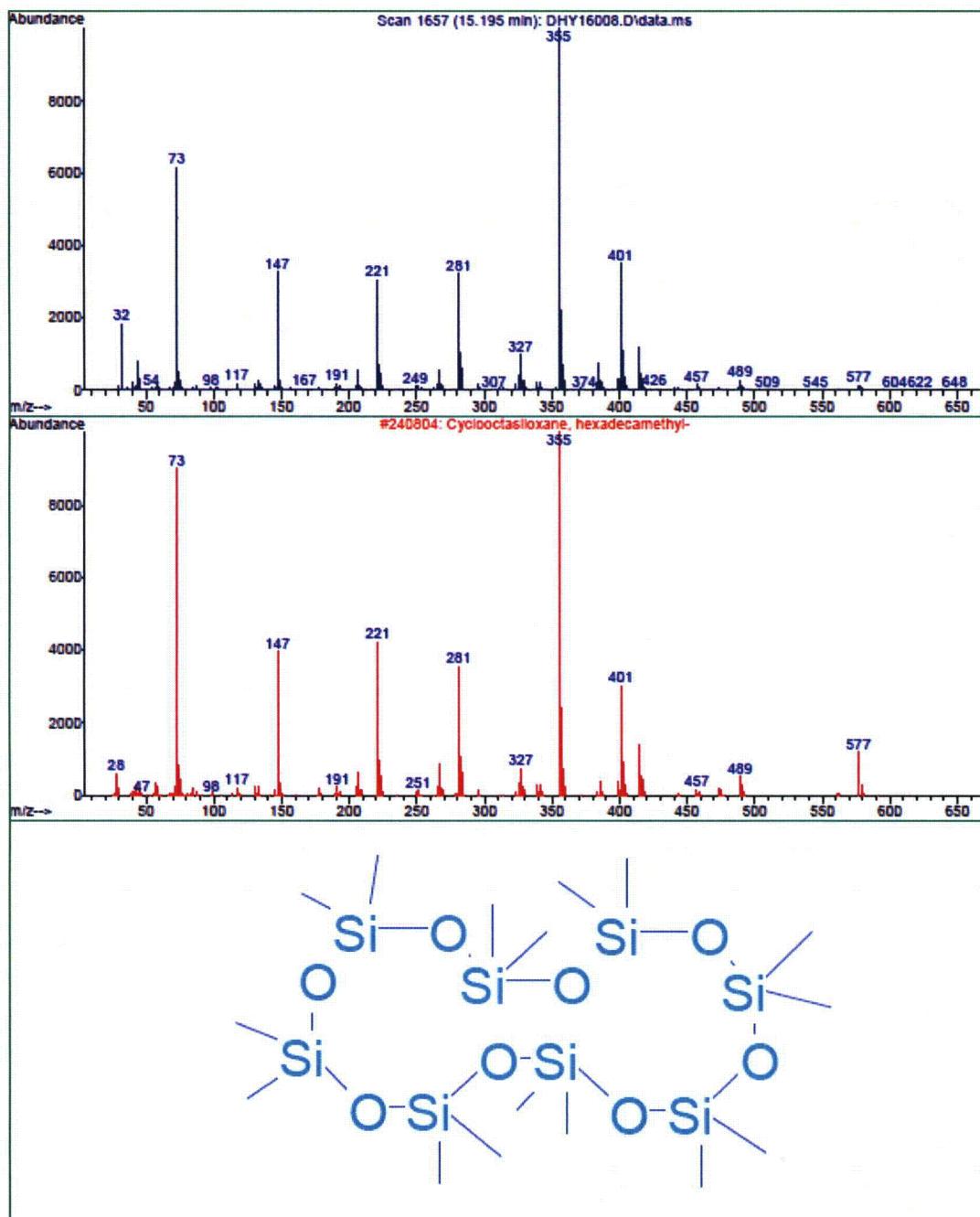
Library Searched : C:\Database\NIST11.L
Quality : 91
ID : Cyclohexasiloxane, dodecamethyl-



Library Searched : C:\Database\NIST11.L
Quality : 93
ID : Cycloheptasiloxane, tetradecamethyl-

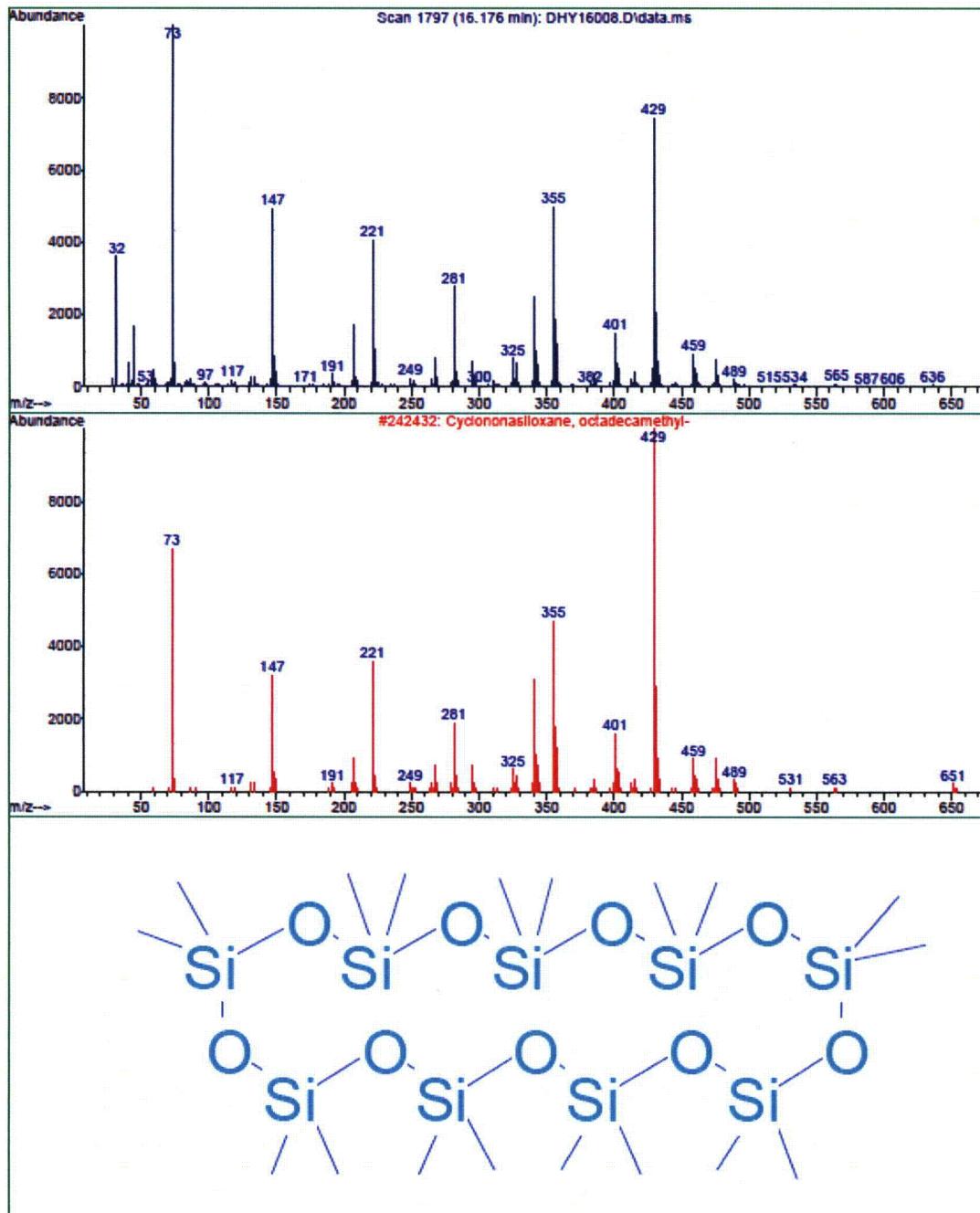


Library Searched : C:\Database\NIST11.L
Quality : 83
ID : Cyclooctasiloxane, hexadecamethyl-



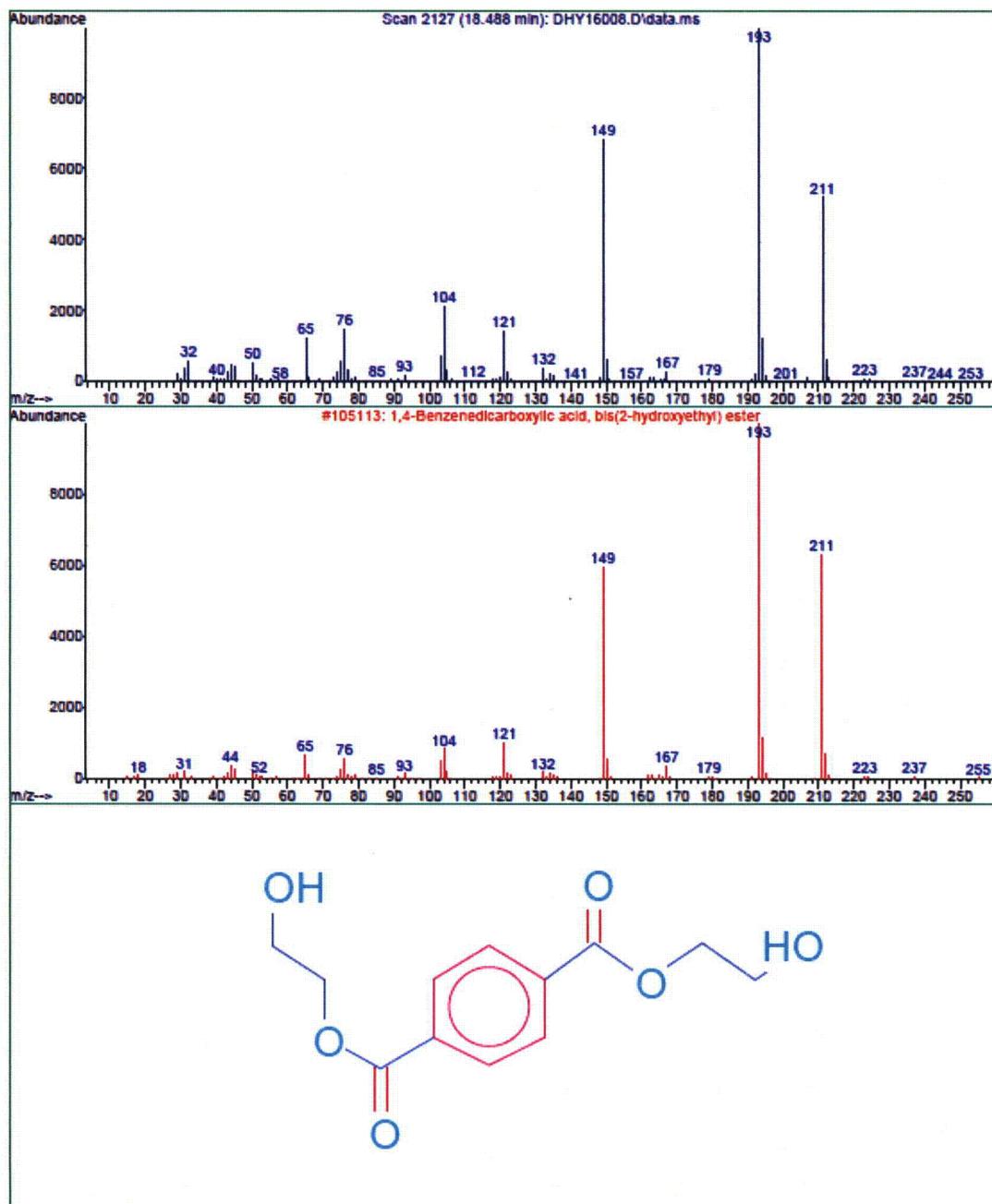
Spectrum 2-4

Library Searched : C:\Database\NIST11.L
Quality : 93
ID : Cyclononasiloxane, octadecamethyl-



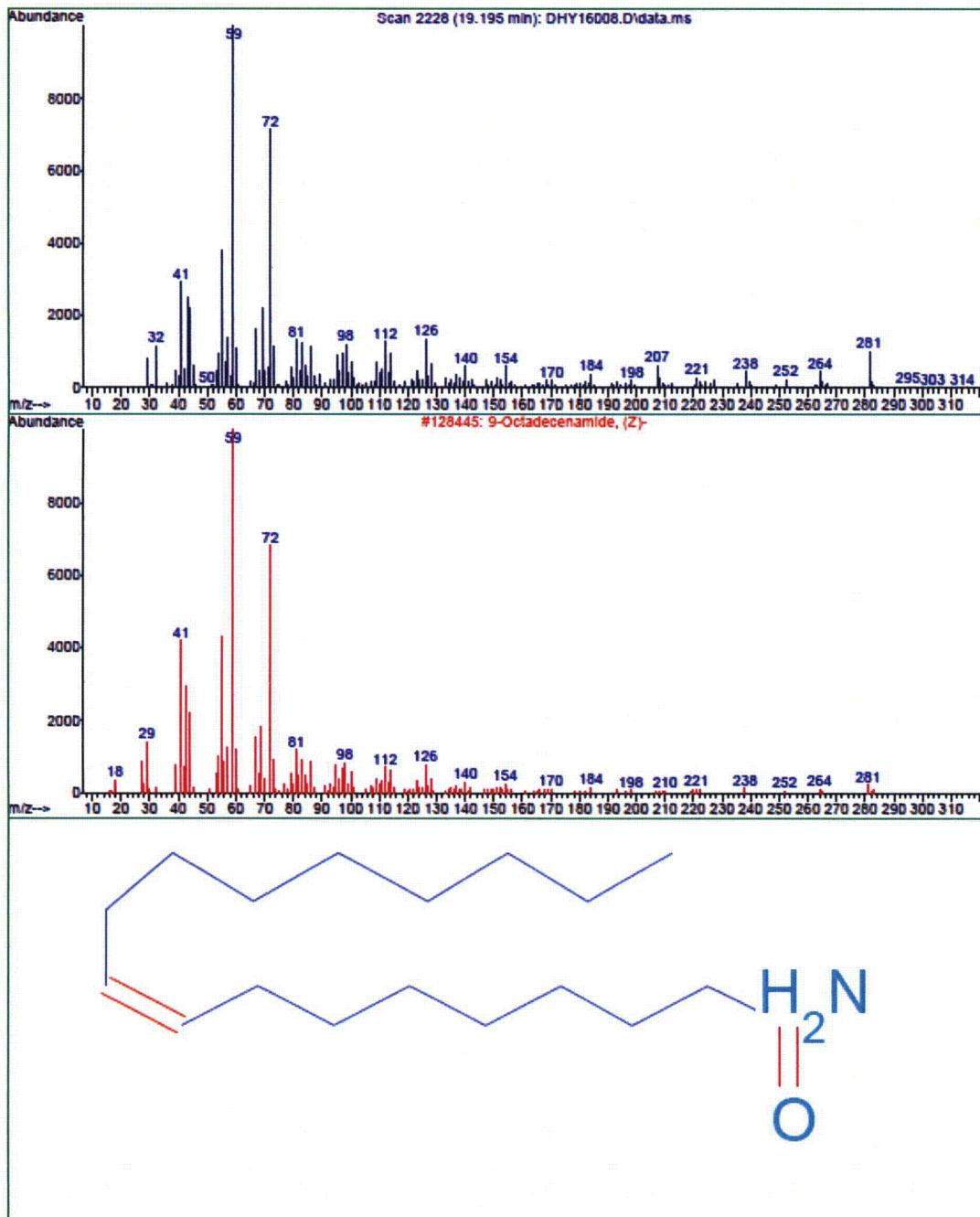
Spectrum 2-5

Library Searched : C:\Database\NIST11.L
Quality : 80
ID : 1,4-Benzenedicarboxylic acid, bis(2-hydroxyethyl) ester

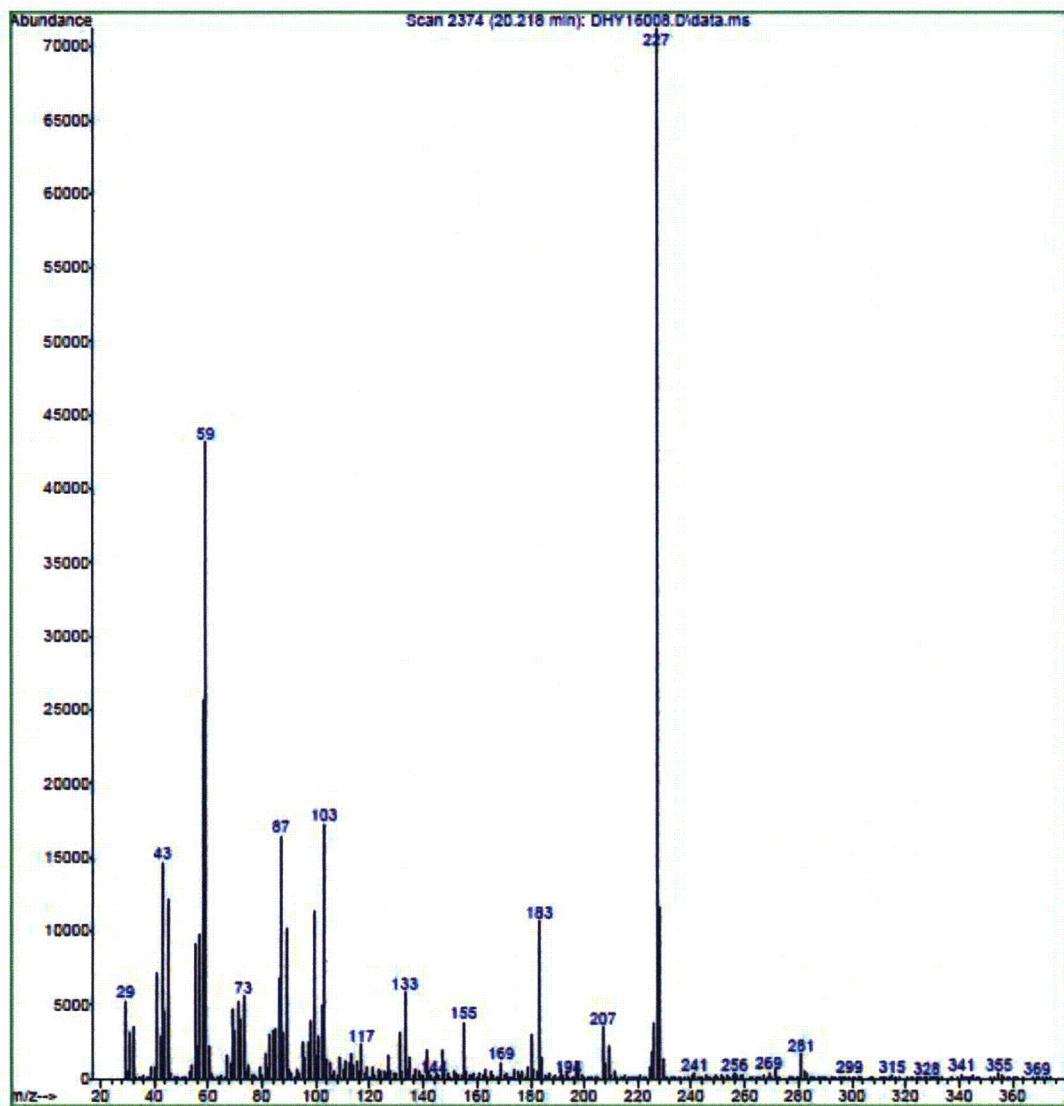


Spectrum 2-6

Library Searched : C:\Database\NIST11.L
Quality : 99
ID : 9-Octadecenamide, (Z)-

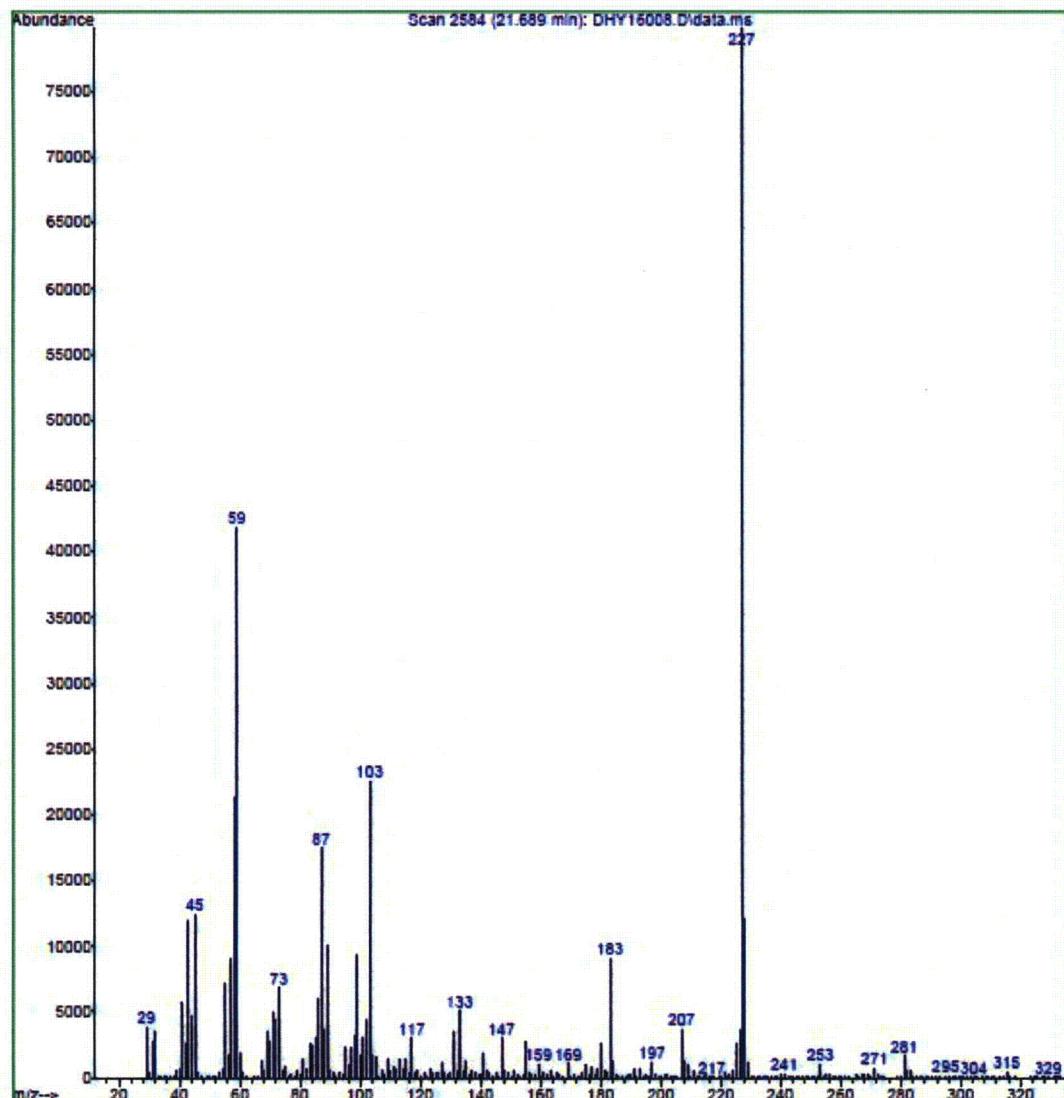


File :Y:\March13\DHY16008.D
Operator : X. CAI
Acquired : 20 Mar 2013 9:33 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info : 1 ul
Vial Number: 1



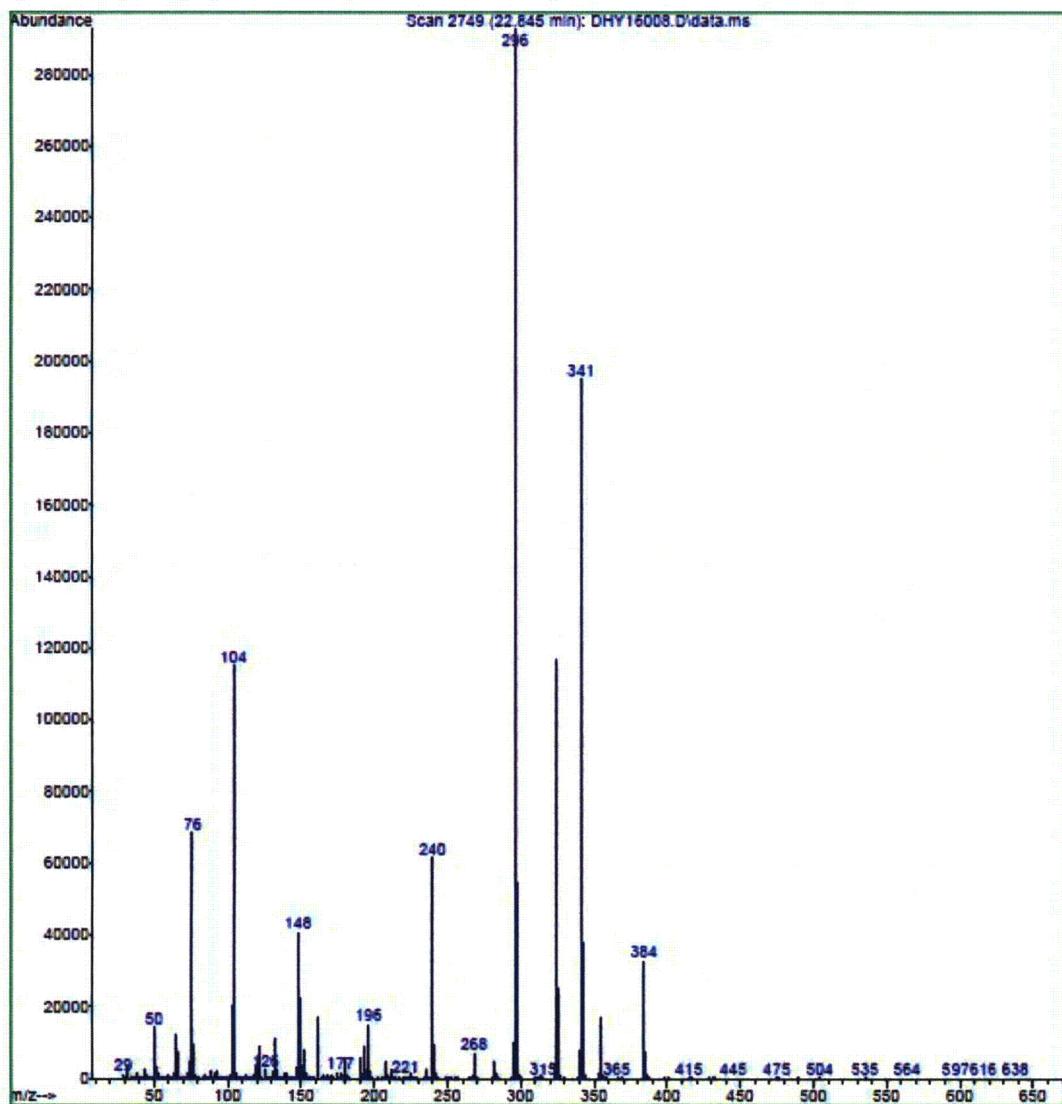
Spectrum 2-8

File : Y:\March13\DHY16008.D
Operator : X. CAI
Acquired : 20 Mar 2013 9:33 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info : 1 ul
Vial Number: 1



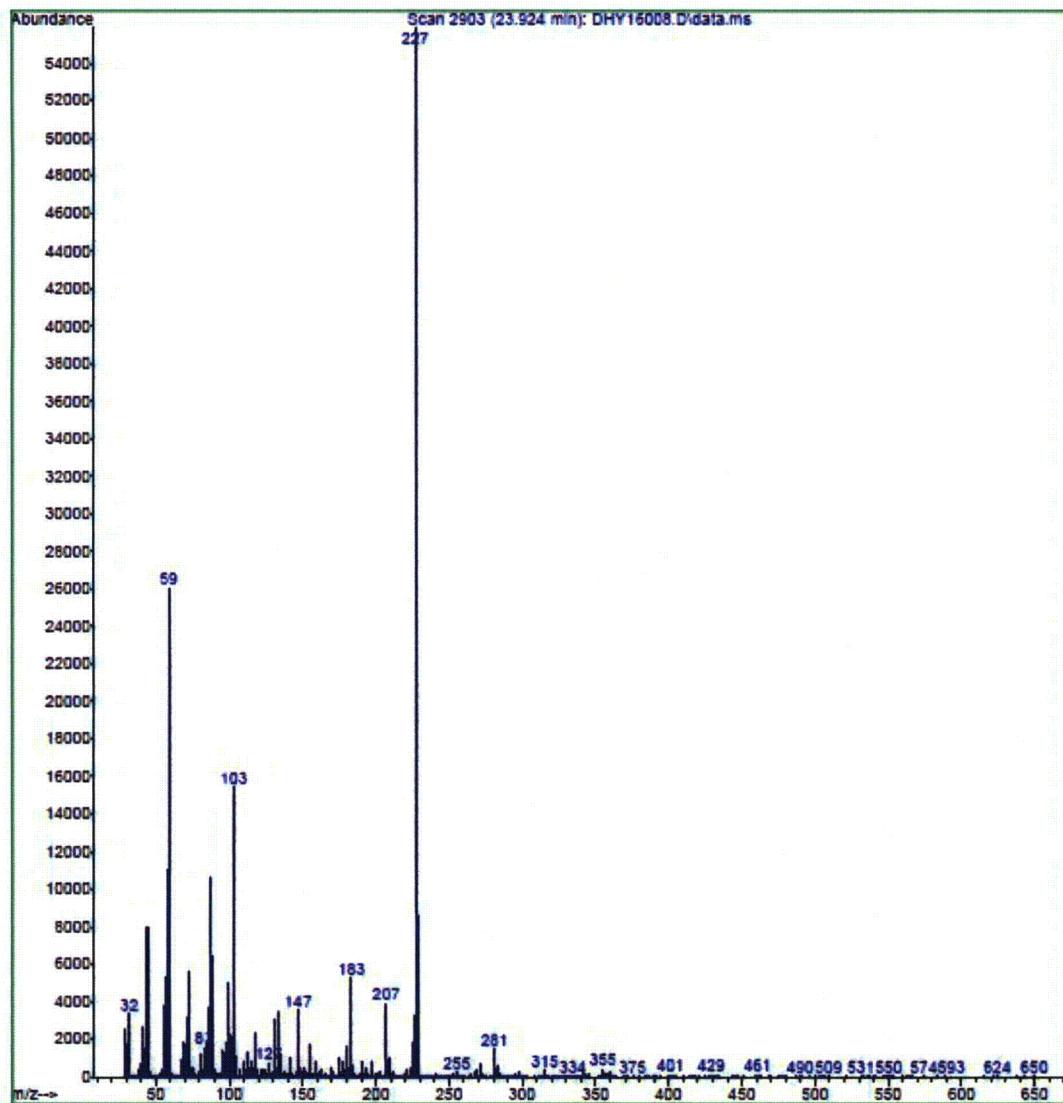
Spectrum 2-9

File : Y:\March13\DHY16008.D
Operator : X. CAI
Acquired : 20 Mar 2013 9:33 using AcqMethod STDSP
Instrument : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info : 1 ul
Vial Number: 1



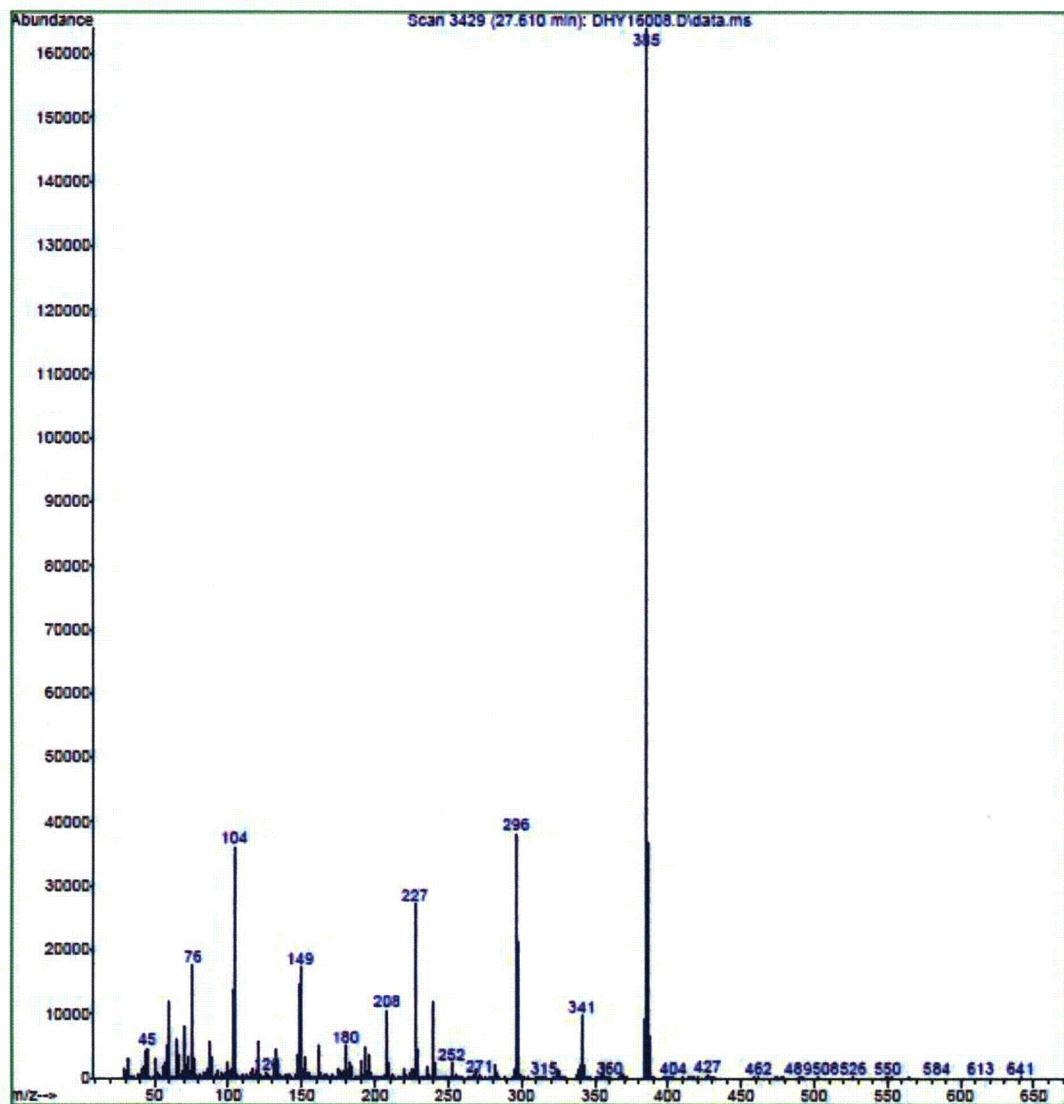
Spectrum 2-10

```
File      :Y:\March13\DHY16008.D
Operator   : X. CAI
Acquired   : 20 Mar 2013    9:33      using AcqMethod STDSPL
Instrument  : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info   : 1 ul
Vial Number: 1
```



Spectrum 2-11

```
File      : Y:\March13\DHY16008.D
Operator   : X. CAI
Acquired   : 20 Mar 2013  9:33      using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info  : 1 ul
Vial Number: 1
```



Spectrum 2-12

File : Y:\March13\DHY16008.D
Operator : X. CAI
Acquired : 20 Mar 13 9:33 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Sample filter extract concentrated 2
Misc Info : 1 ul
Vial Number: 1

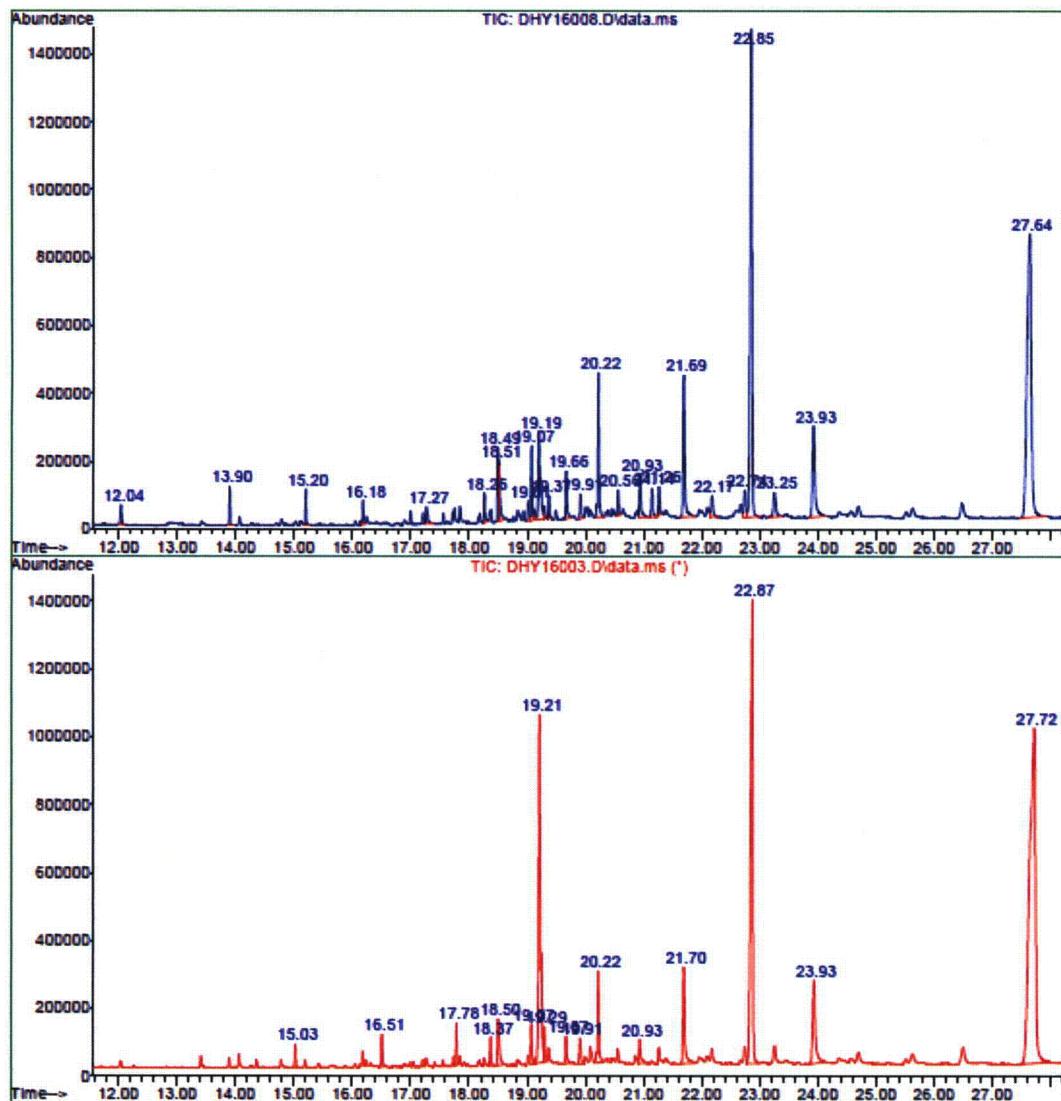


Figure 3

File : Y:\March13\DHY16014.D
Operator : X. CAI
Acquired : 21 Mar 2013 7:51 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch-Brite control concentrated
Misc Info : 1 ul
Vial Number: 1

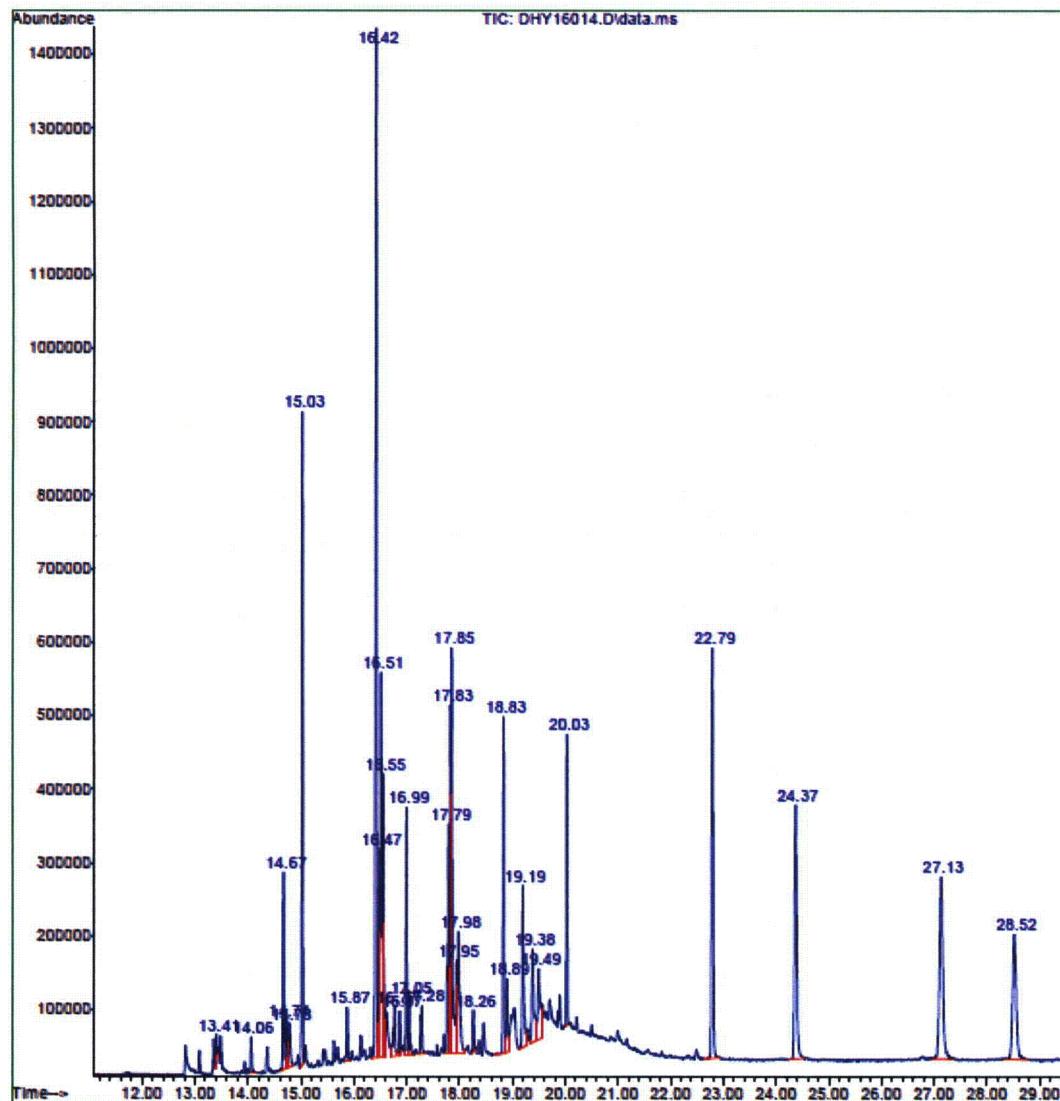
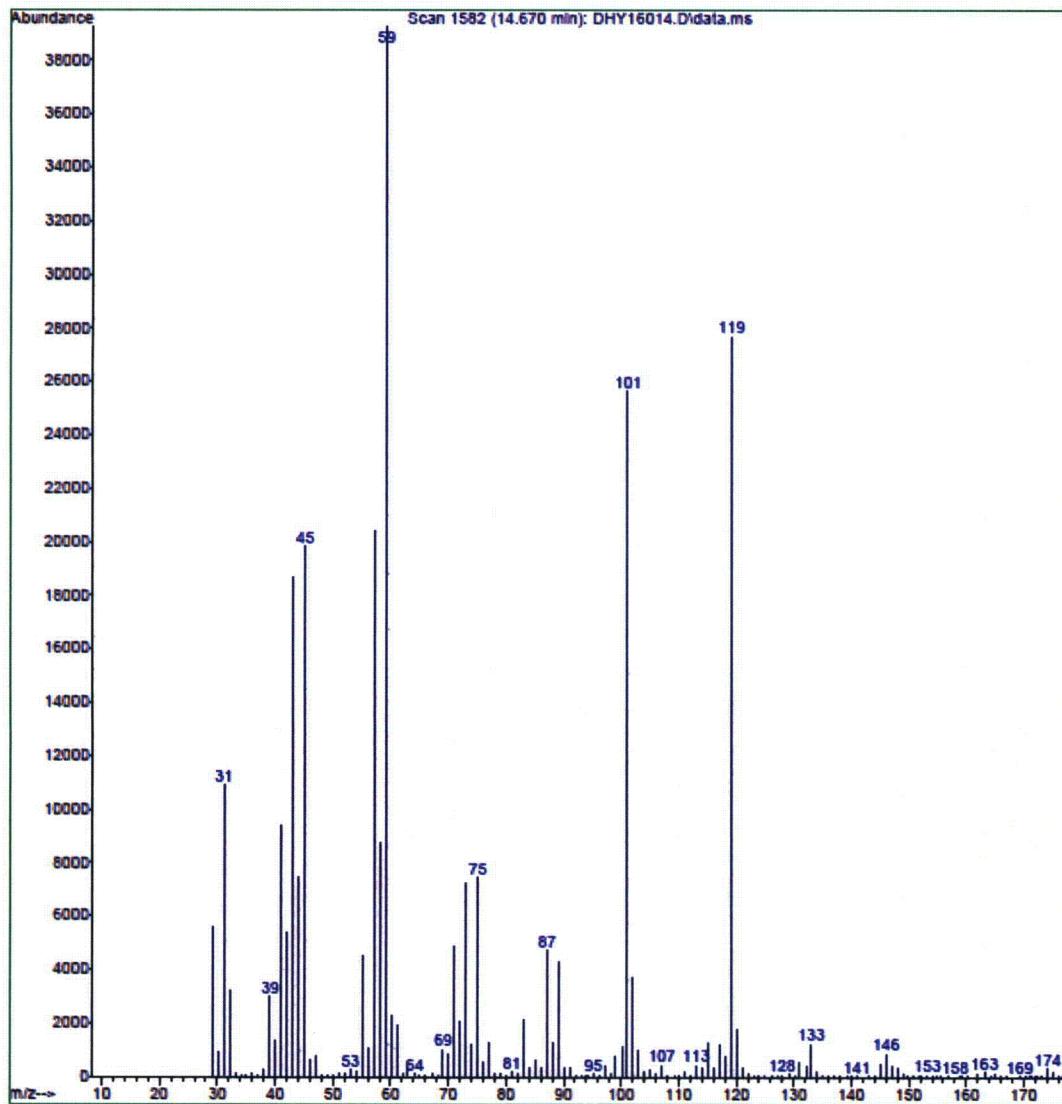


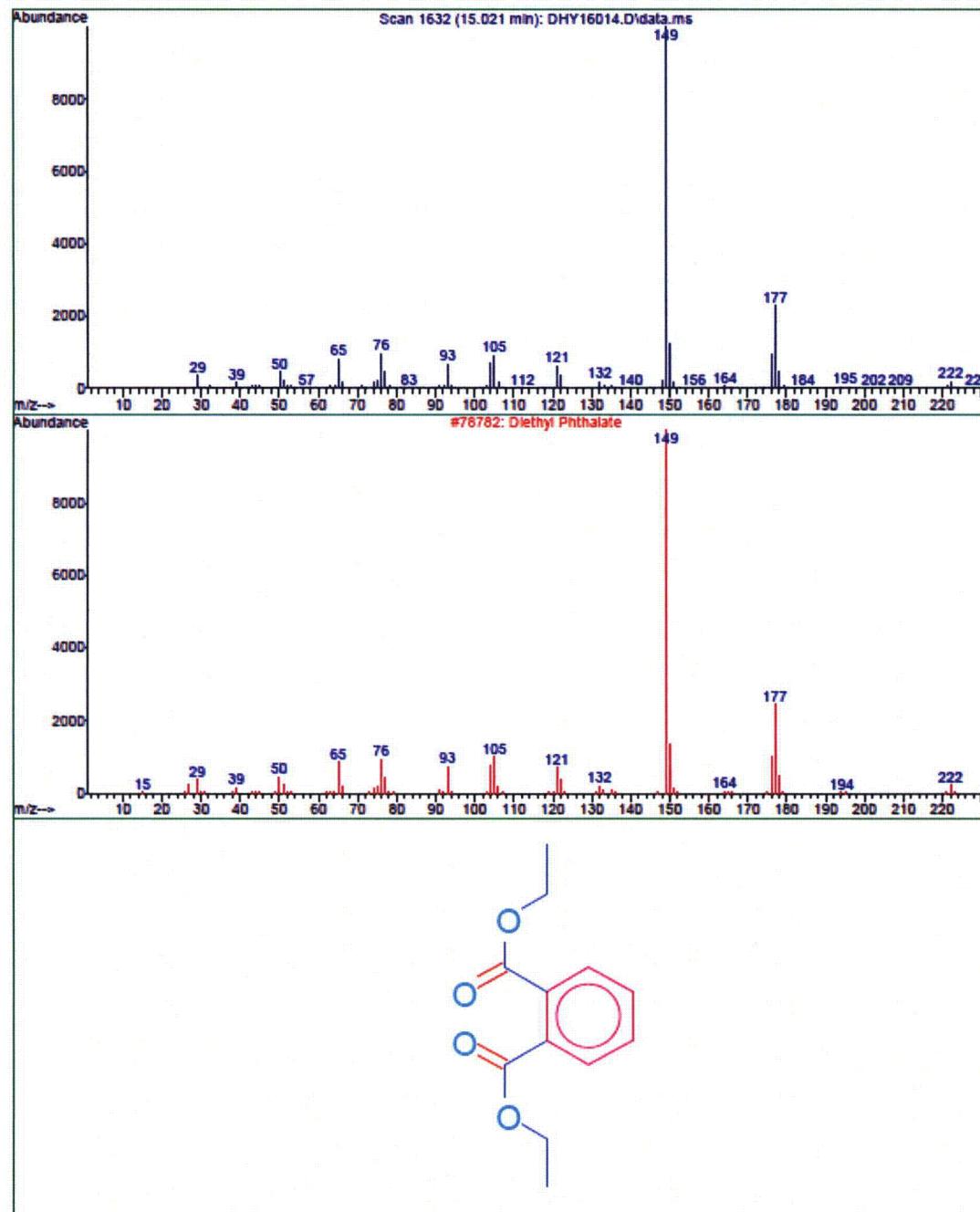
Figure 4

File :Y:\March13\DHY16014.D
Operator : X. CAI
Acquired : 21 Mar 2013 7:51 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch-Brite control concentrated
Misc Info : 1 ul
Vial Number: 1



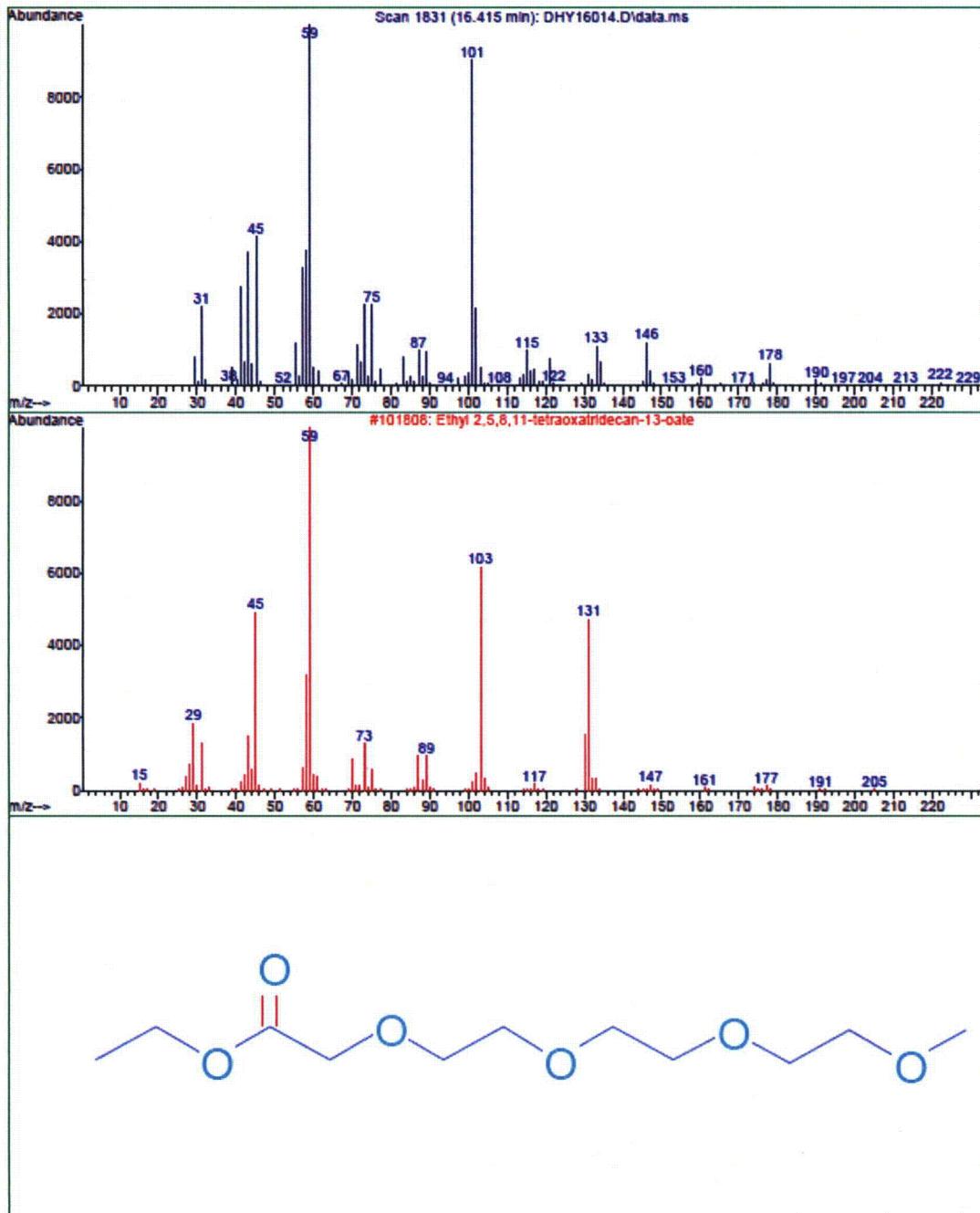
Spectrum 4-1

Library Searched : C:\Database\NIST11.L
Quality : 98
ID : Diethyl Phthalate



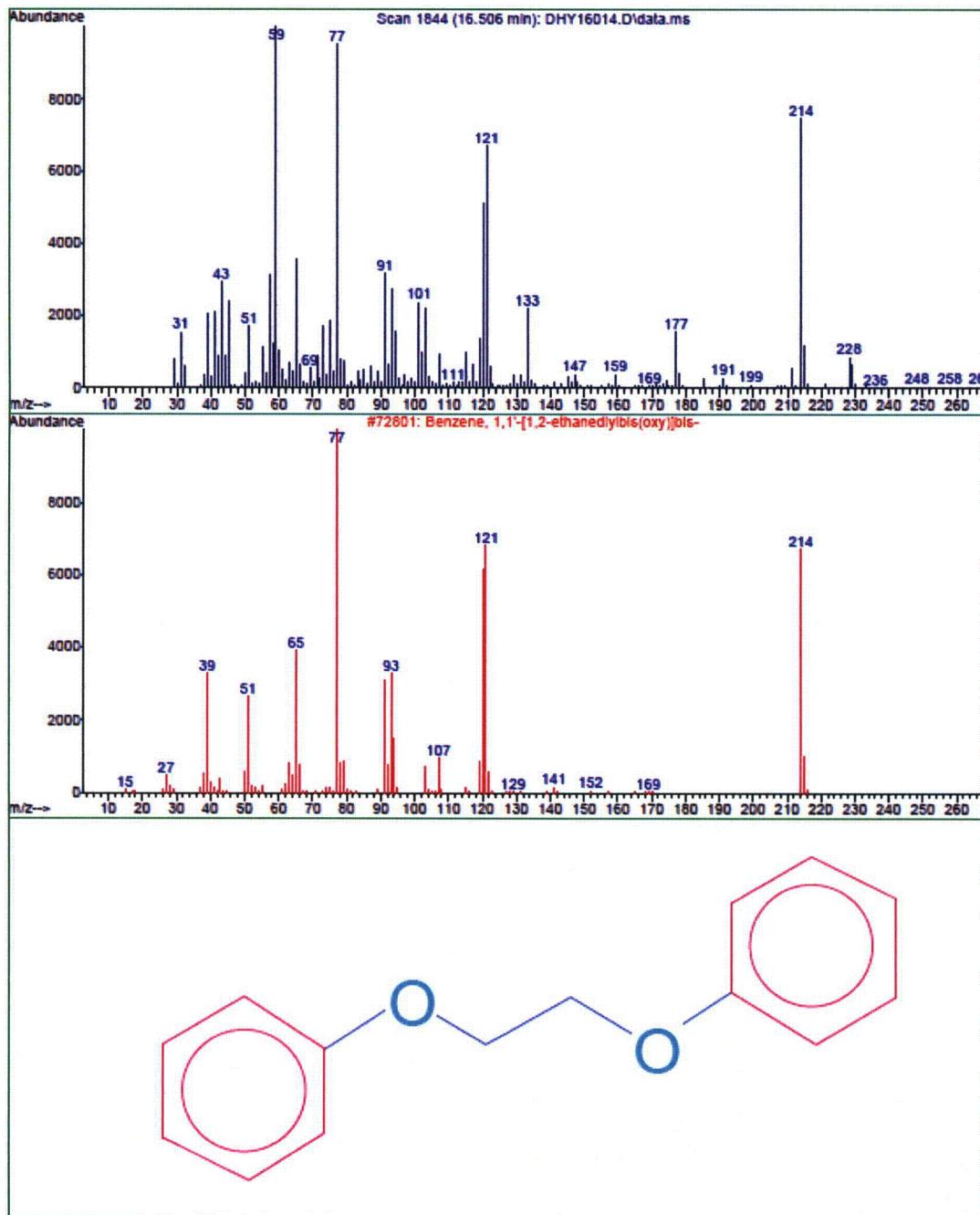
Spectrum 4-2

Library Searched : C:\Database\NIST11.L
Quality : 38
ID : Ethyl 2,5,8,11-tetraoxatridecan-13-oate



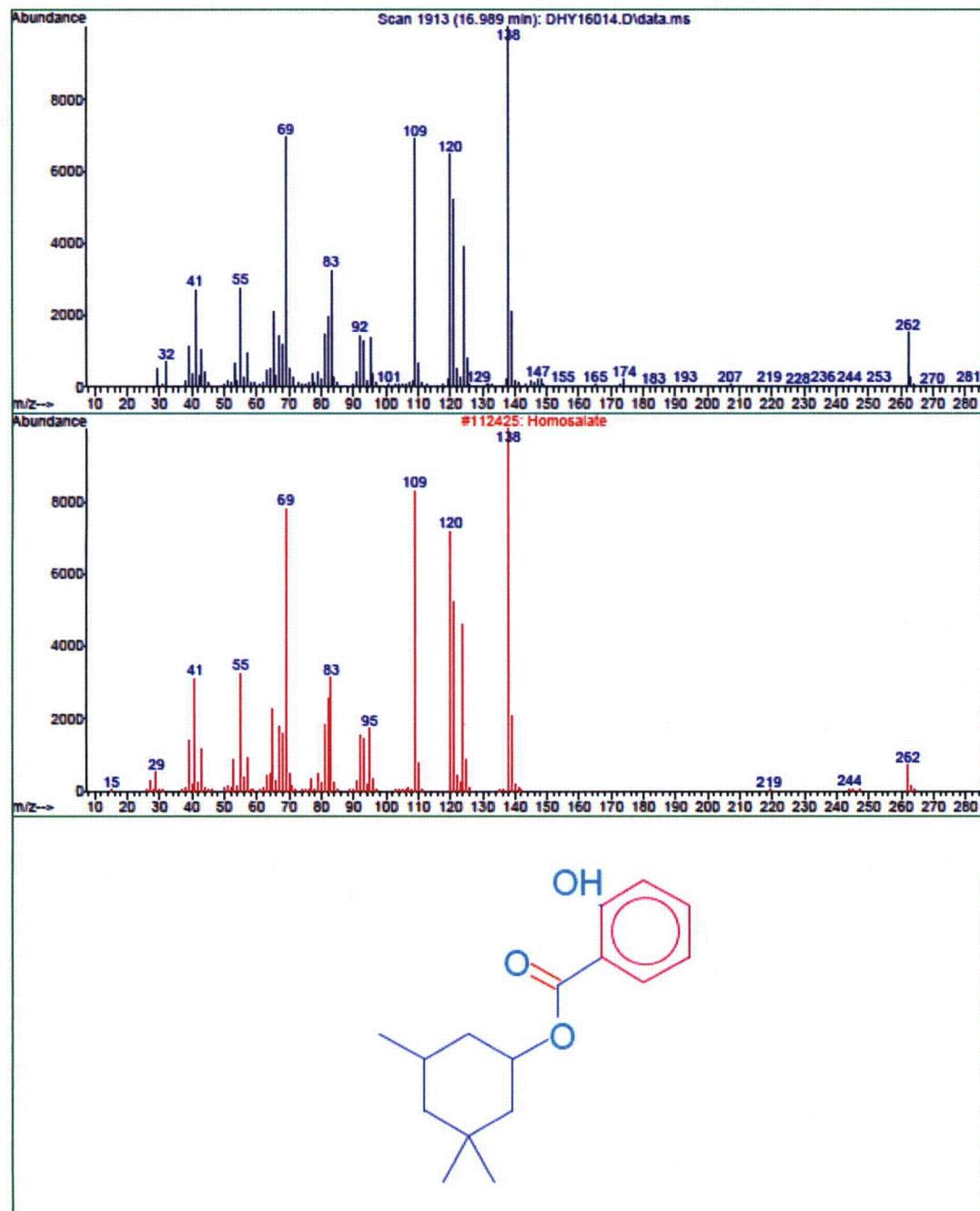
Spectrum 4-3

Library Searched : C:\Database\NIST11.L
Quality : 92
ID : Benzene, 1,1'-[1,2-ethanediylbis(oxy)]bis-



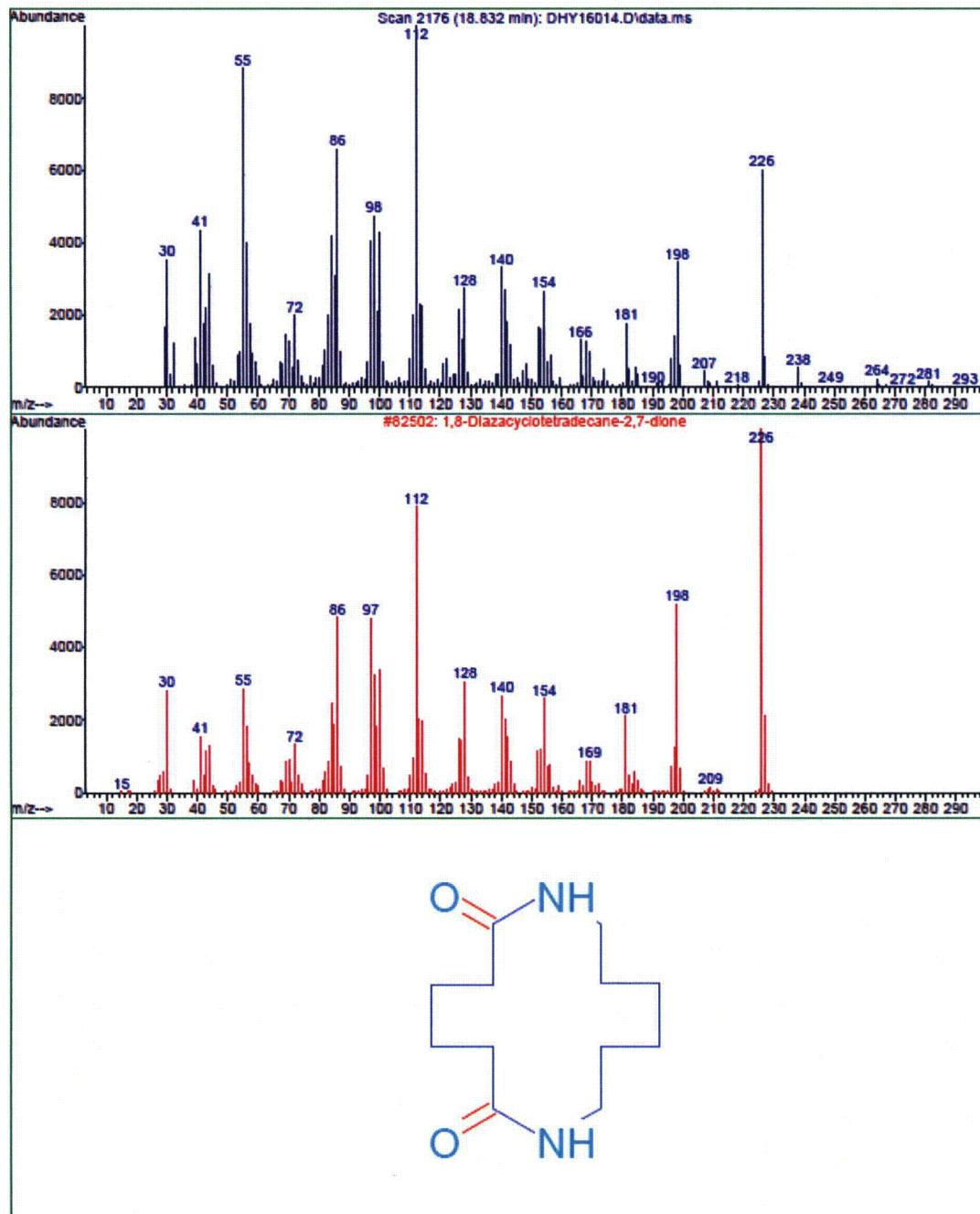
Spectrum 4-4

Library Searched : C:\Database\NIST11.L
Quality : 99
ID : Homosalate



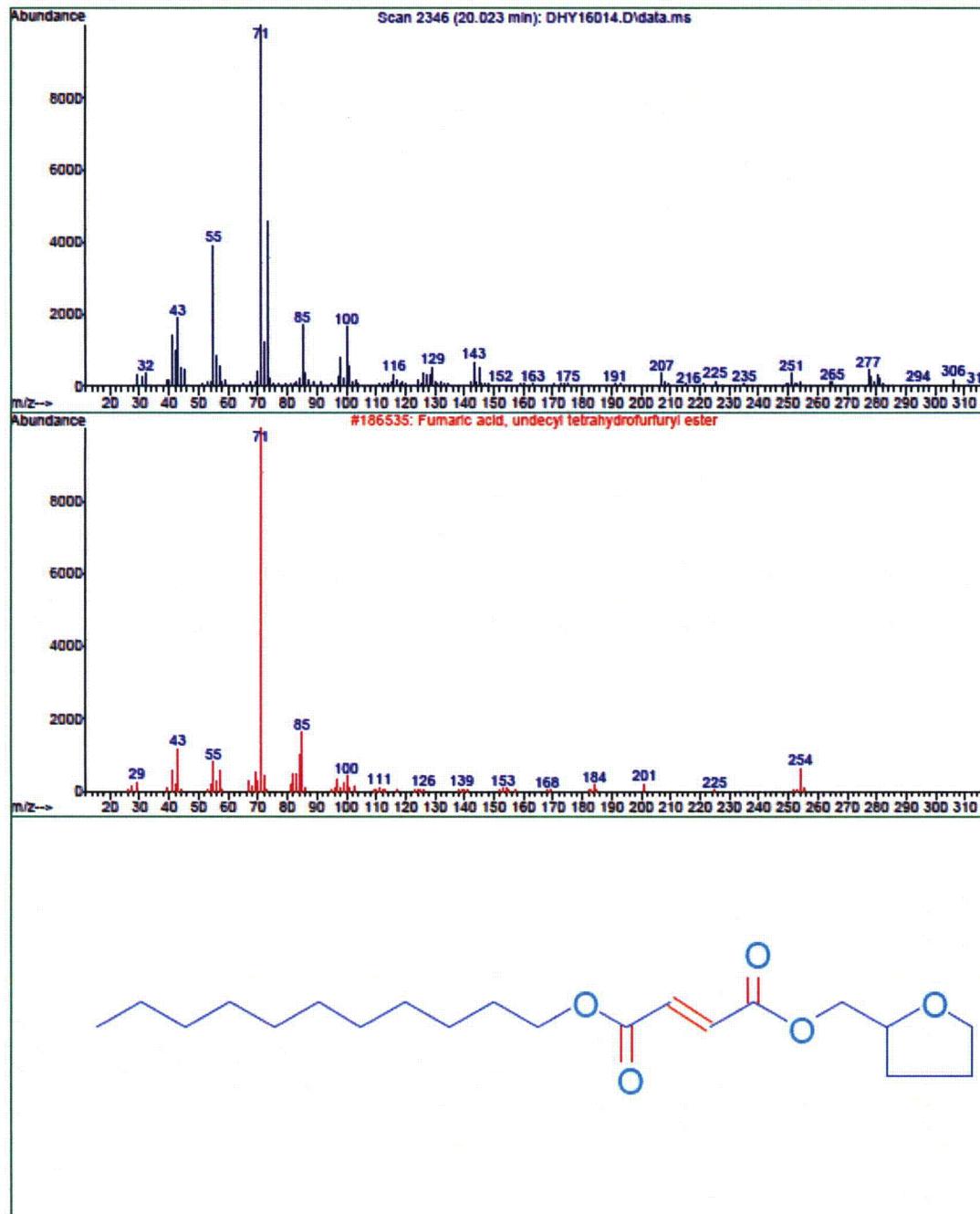
Spectrum 4-5

Library Searched : C:\Database\NIST11.L
Quality : 53
ID : 1,8-Diazacyclotetradecane-2,7-dione



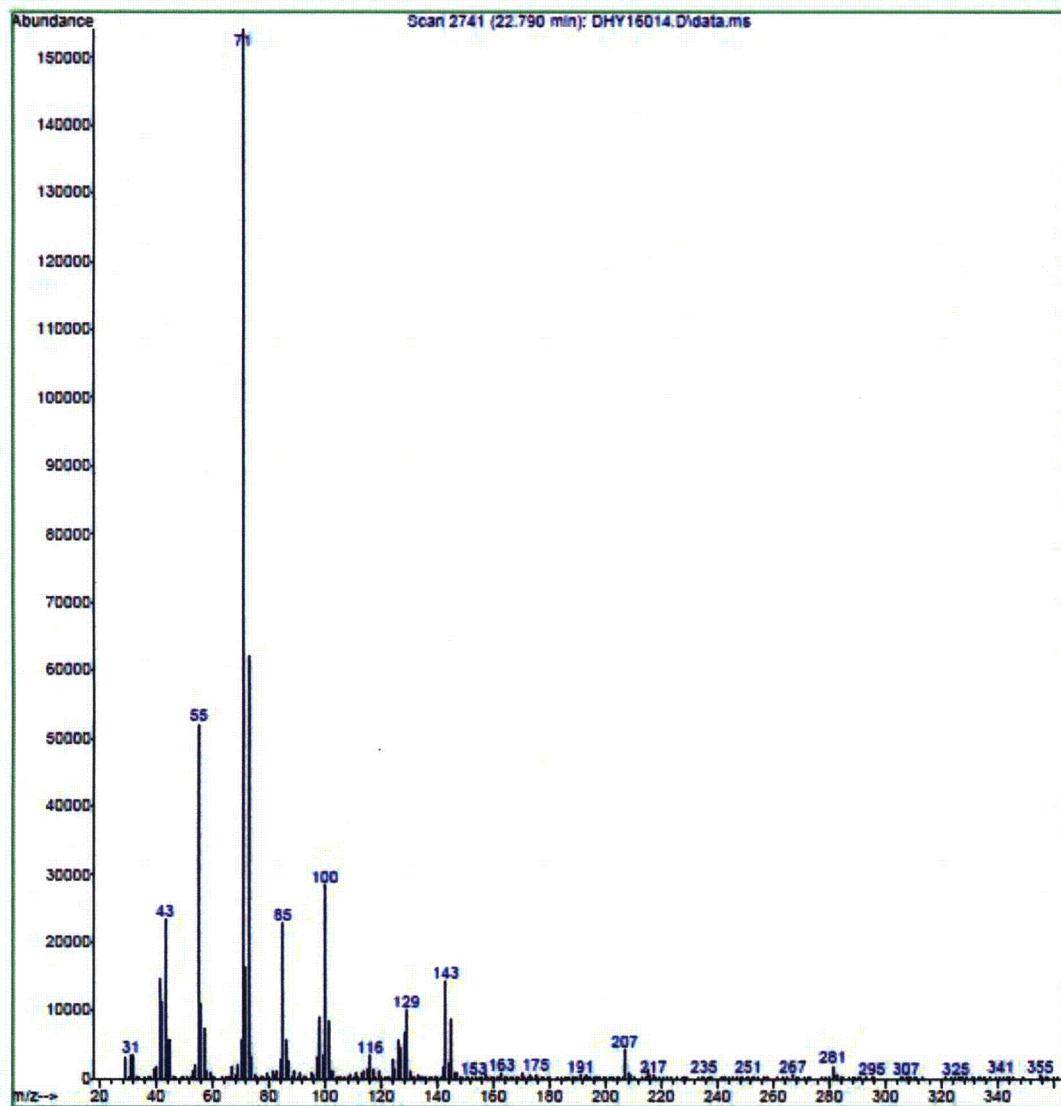
Spectrum 4-6

Library Searched : C:\Database\NIST11.L
Quality : 53
ID : Fumaric acid, undecyl tetrahydrofurfuryl ester



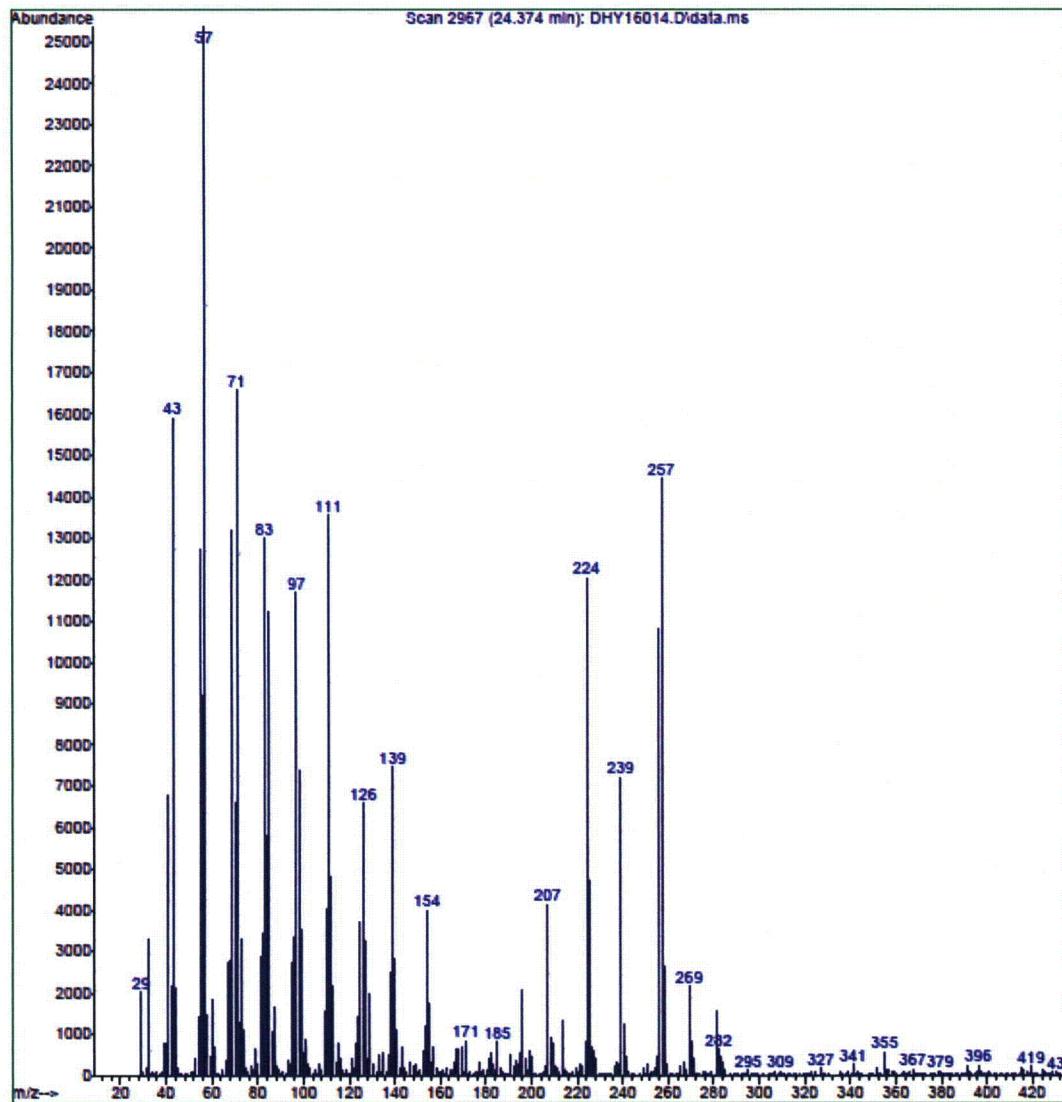
Spectrum 4-7

File : Y:\March13\DHY16014.D
Operator : X. CAI
Acquired : 21 Mar 2013 7:51 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch-Brite control concentrated
Misc Info : 1 ul
Vial Number: 1



Spectrum 4-8

File :Y:\March13\DHY16014.D
Operator : X. CAI
Acquired : 21 Mar 2013 7:51 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch-Brite control concentrated
Misc Info : 1 ul
Vial Number: 1



Spectrum 4-9

File :Y:\March13\DHY16010.D
Operator : X. CAI
Acquired : 20 Mar 2013 10:48 using AcqMethod STDSP
Instrument : GC/MS Ins
Sample Name: Scotch Brite Used
Misc Info : 1 ul
Vial Number: 1

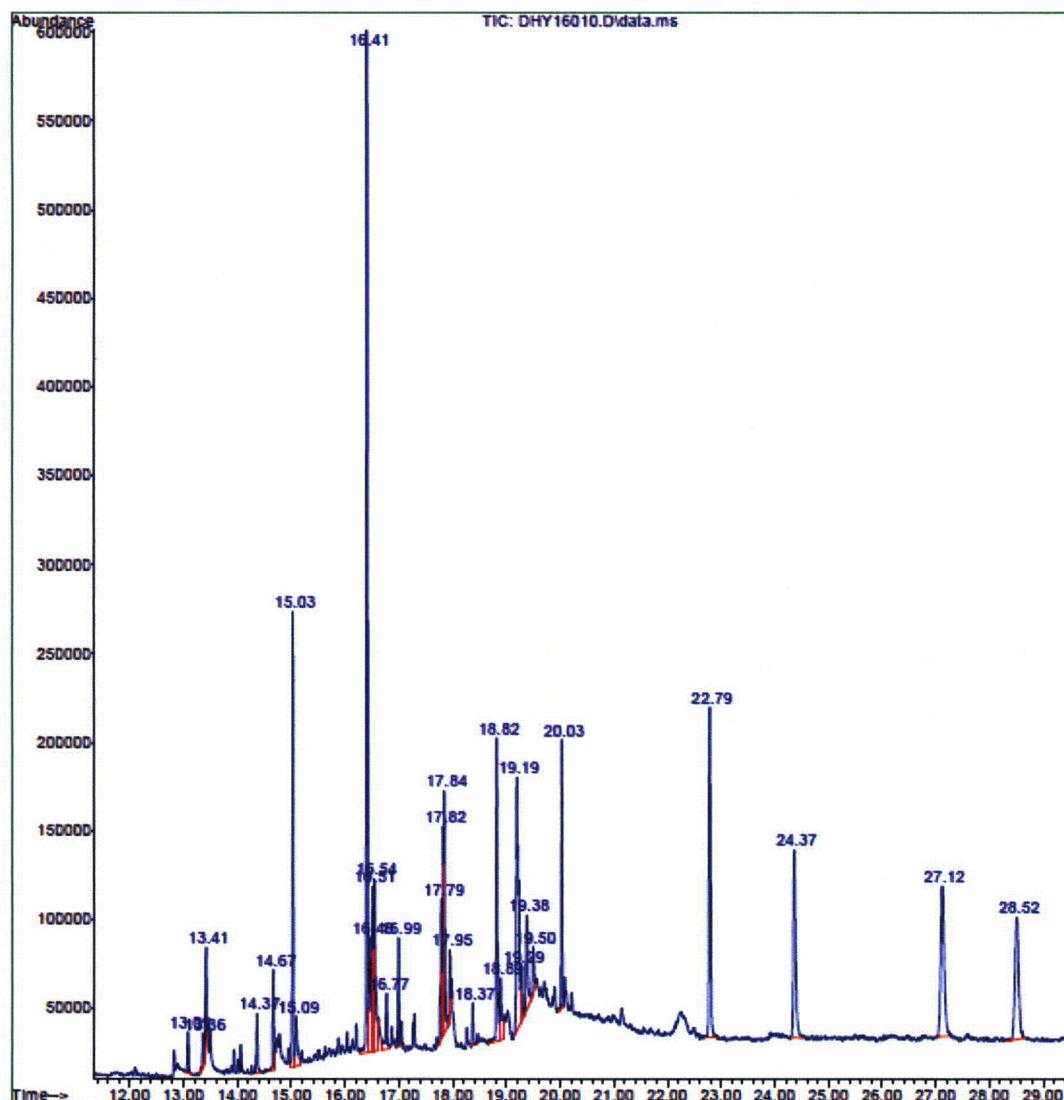
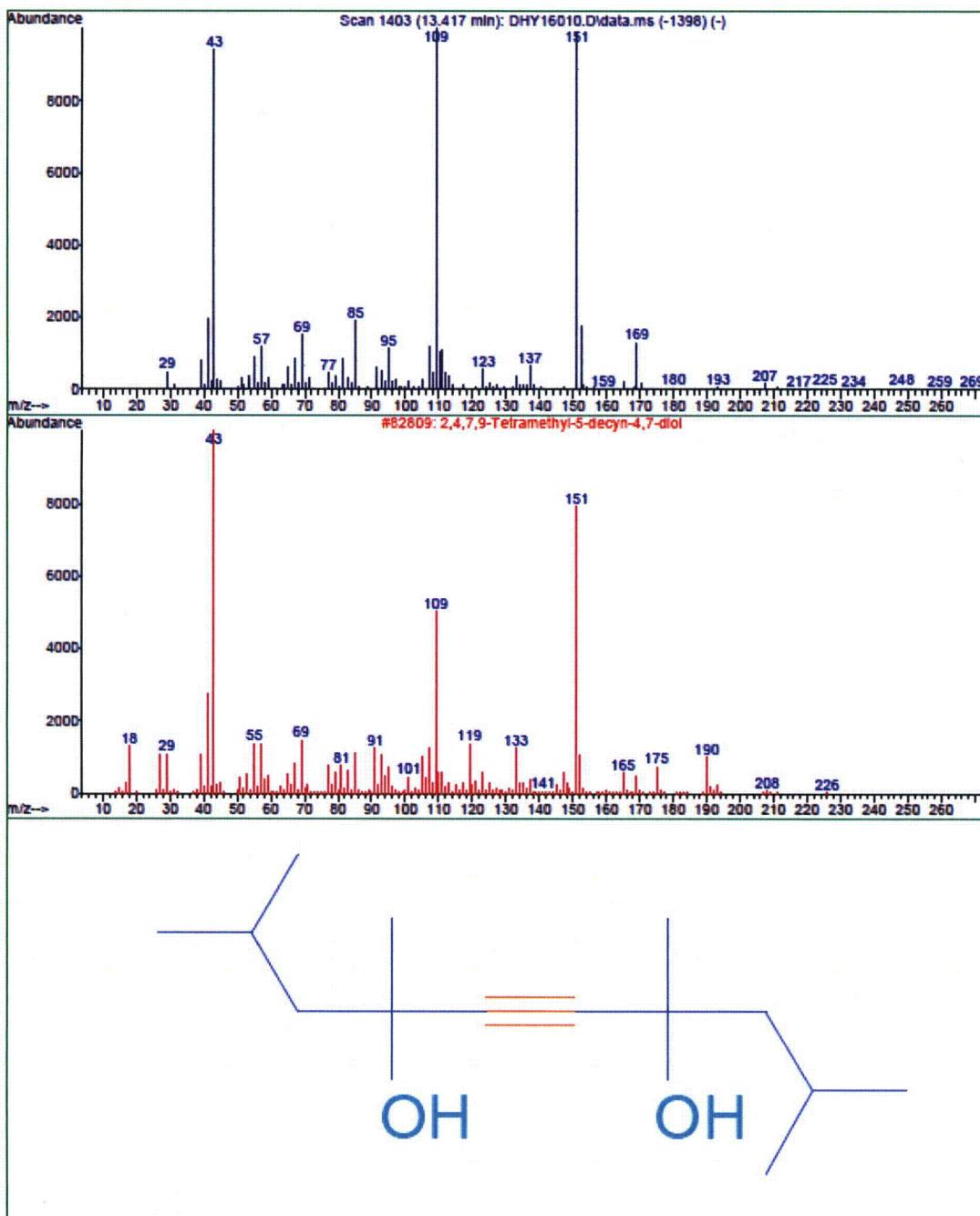


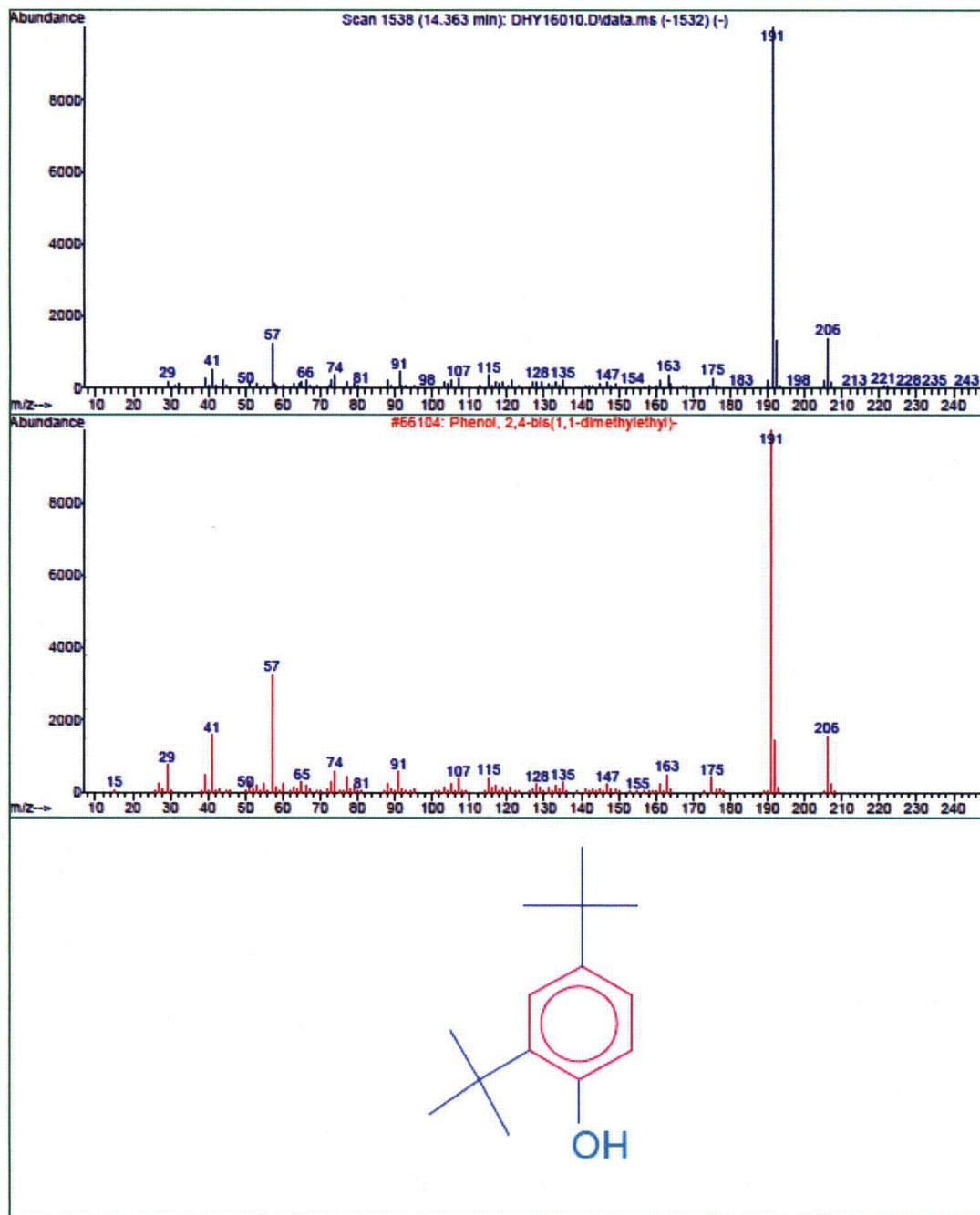
Figure 5

Library Searched : C:\Database\NIST11.L
Quality : 72
ID : 2,4,7,9-Tetramethyl-5-decyn-4,7-diol



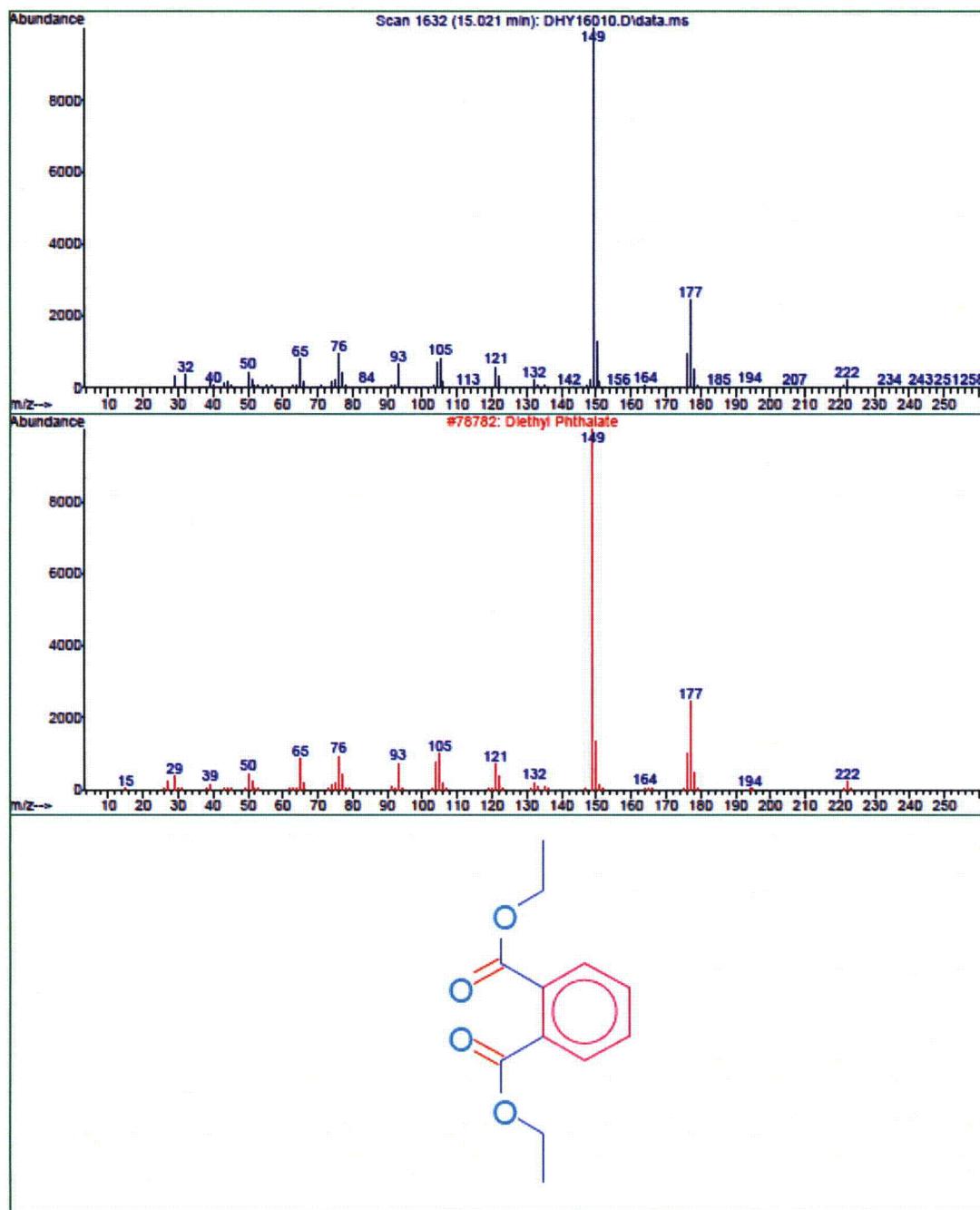
Spectrum 5-1

Library Searched : C:\Database\NIST11.L
Quality : 96
ID : Phenol, 2,4-bis(1,1-dimethylethyl)-



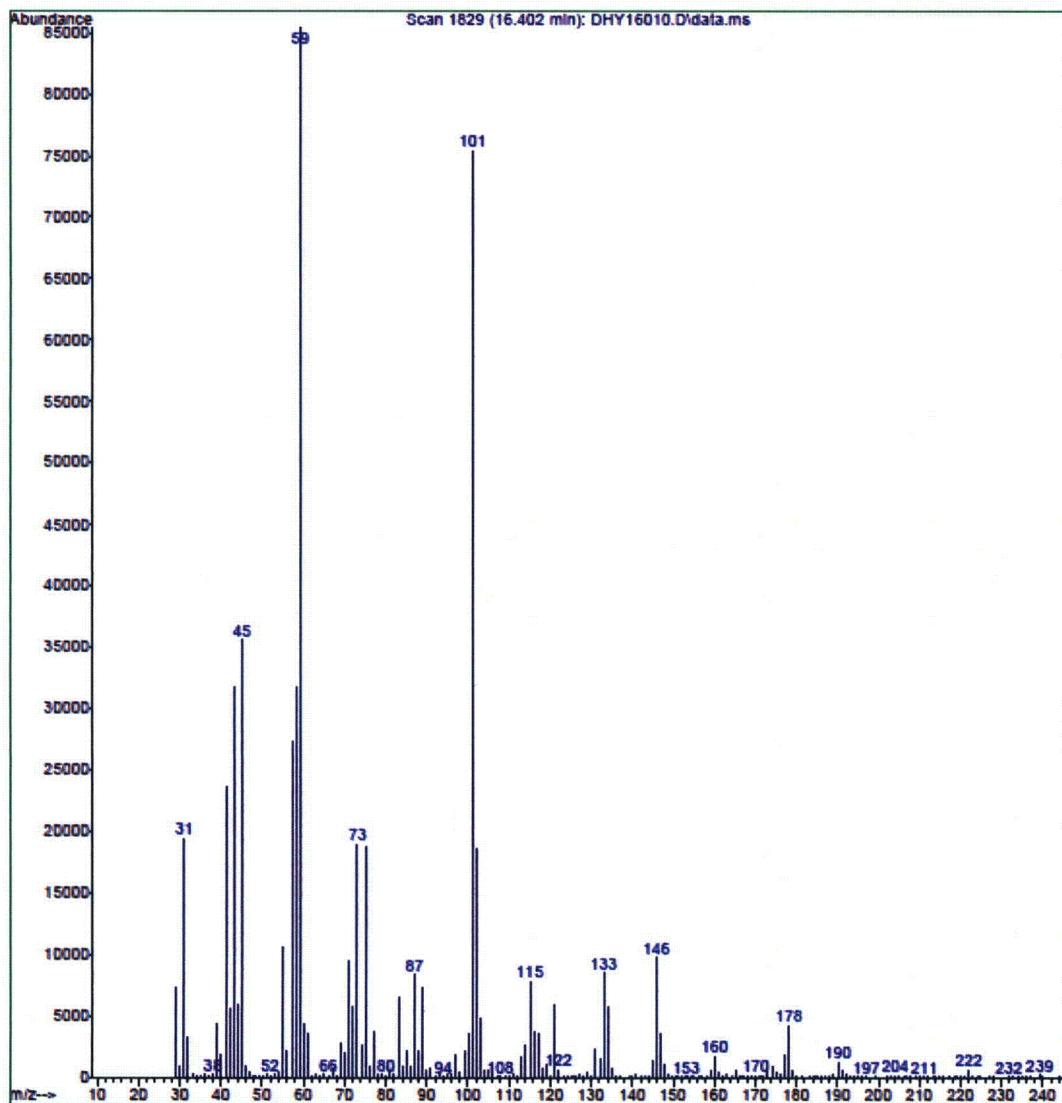
Spectrum 5-2

Library Searched : C:\Database\NIST11.L
Quality : 98
ID : Diethyl Phthalate



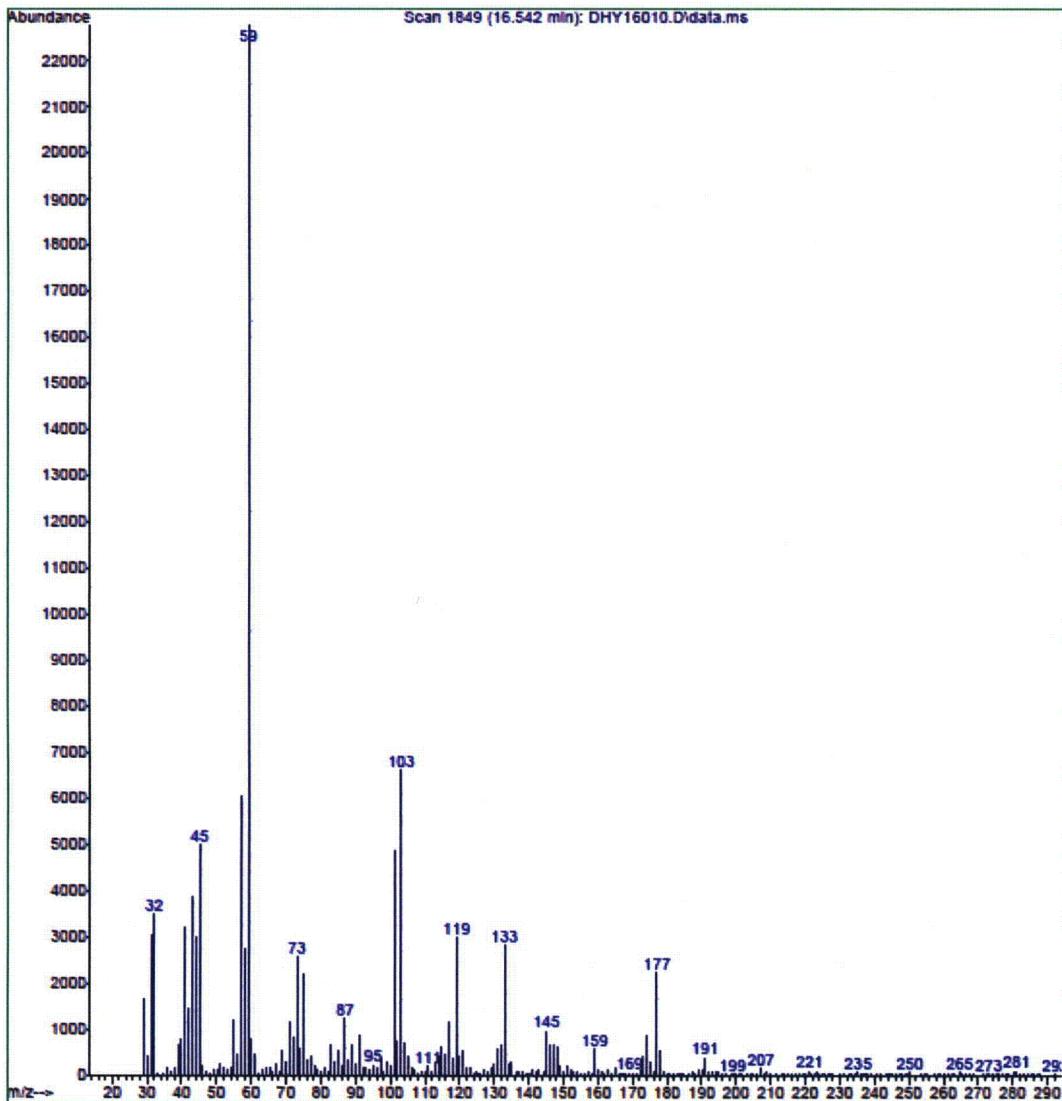
Spectrum 5-3

File : Y:\March13\DHY16010.D
Operator : X. CAI
Acquired : 20 Mar 2013 10:48 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch Brite Used
Misc Info : 1 ul
Vial Number: 1

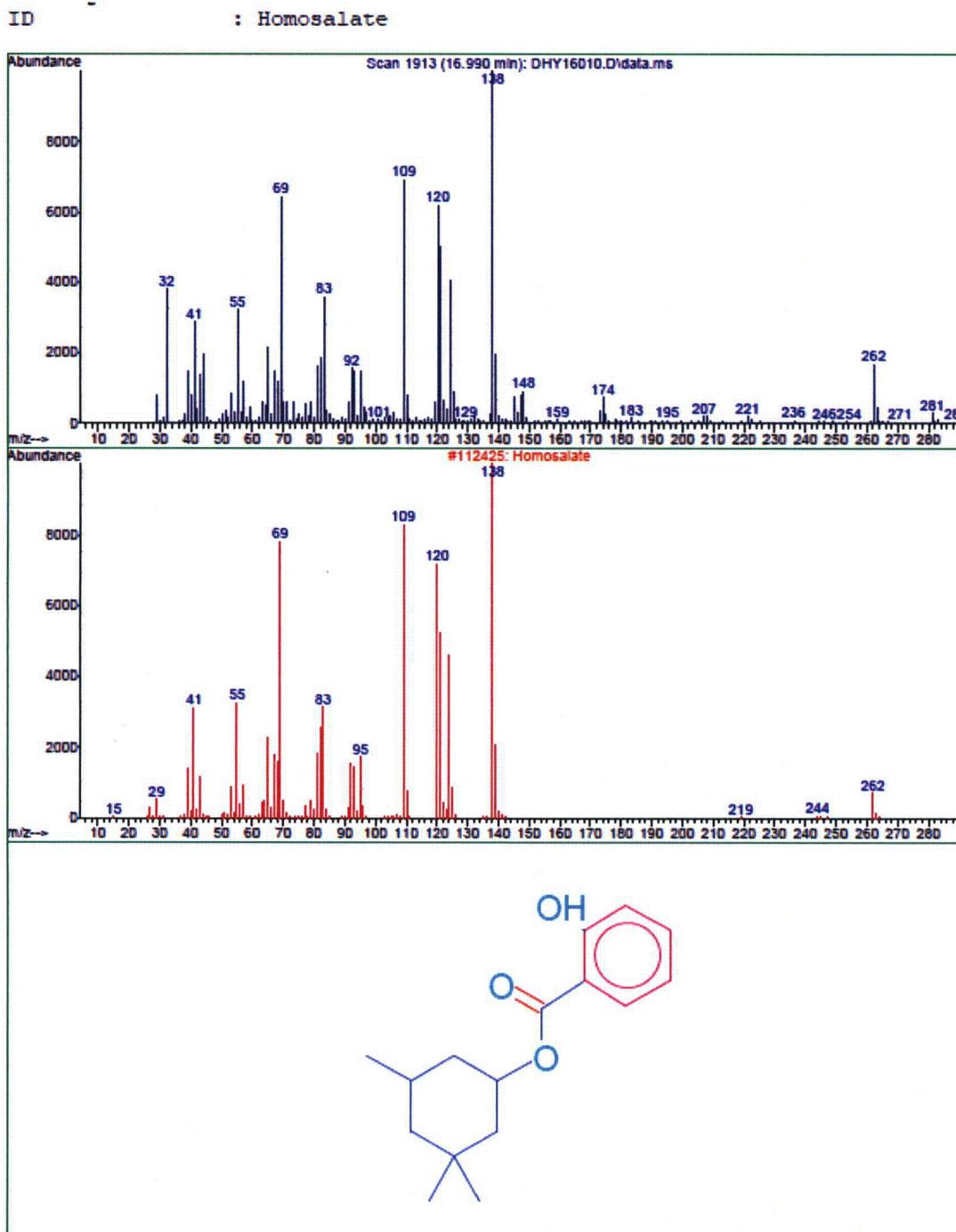


Spectrum 5-4

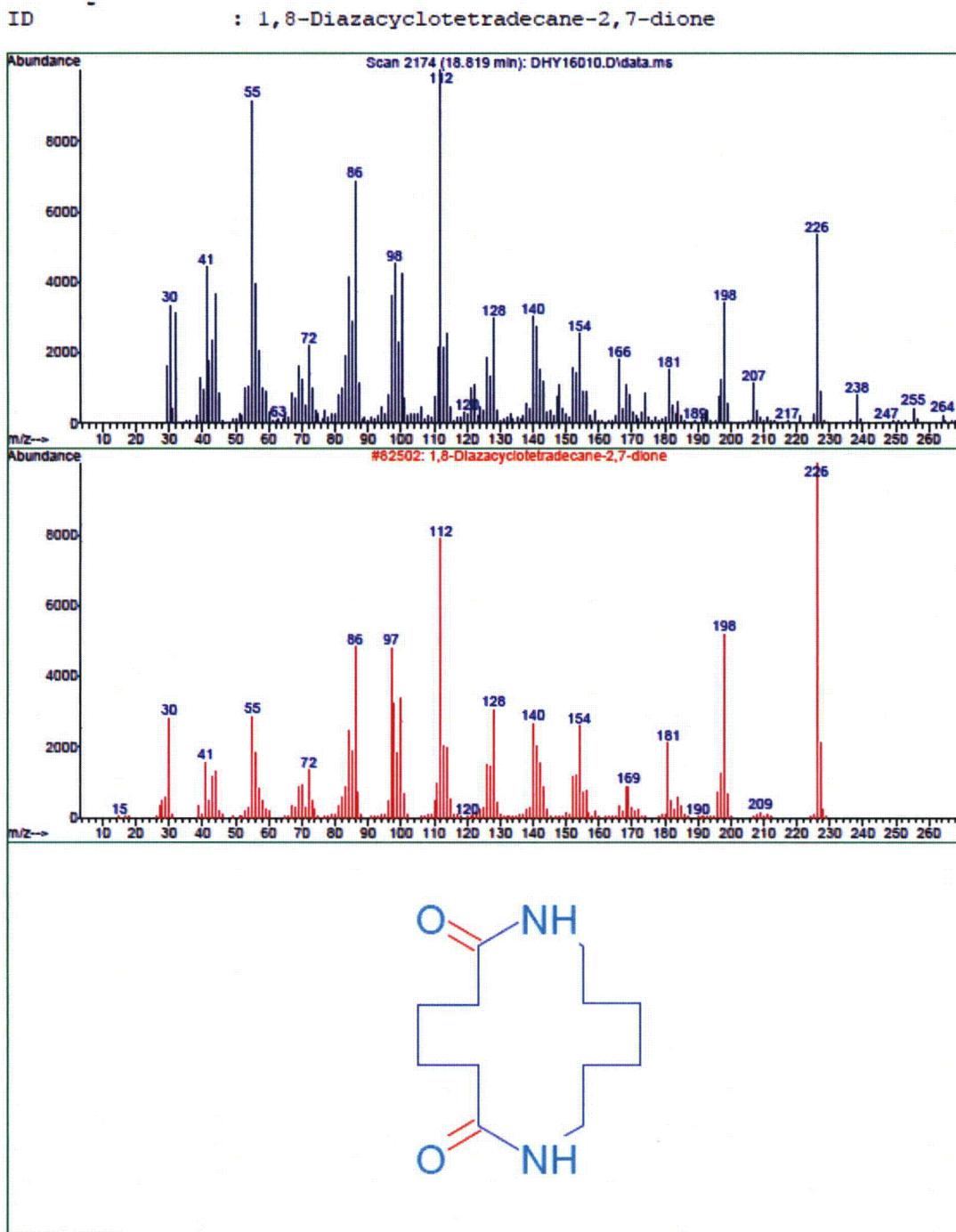
File : Y:\March13\DHY16010.D
Operator : X. CAI
Acquired : 20 Mar 2013 10:48 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch Brite Used
Misc Info : 1 ul
Vial Number: 1



Spectrum 5-5

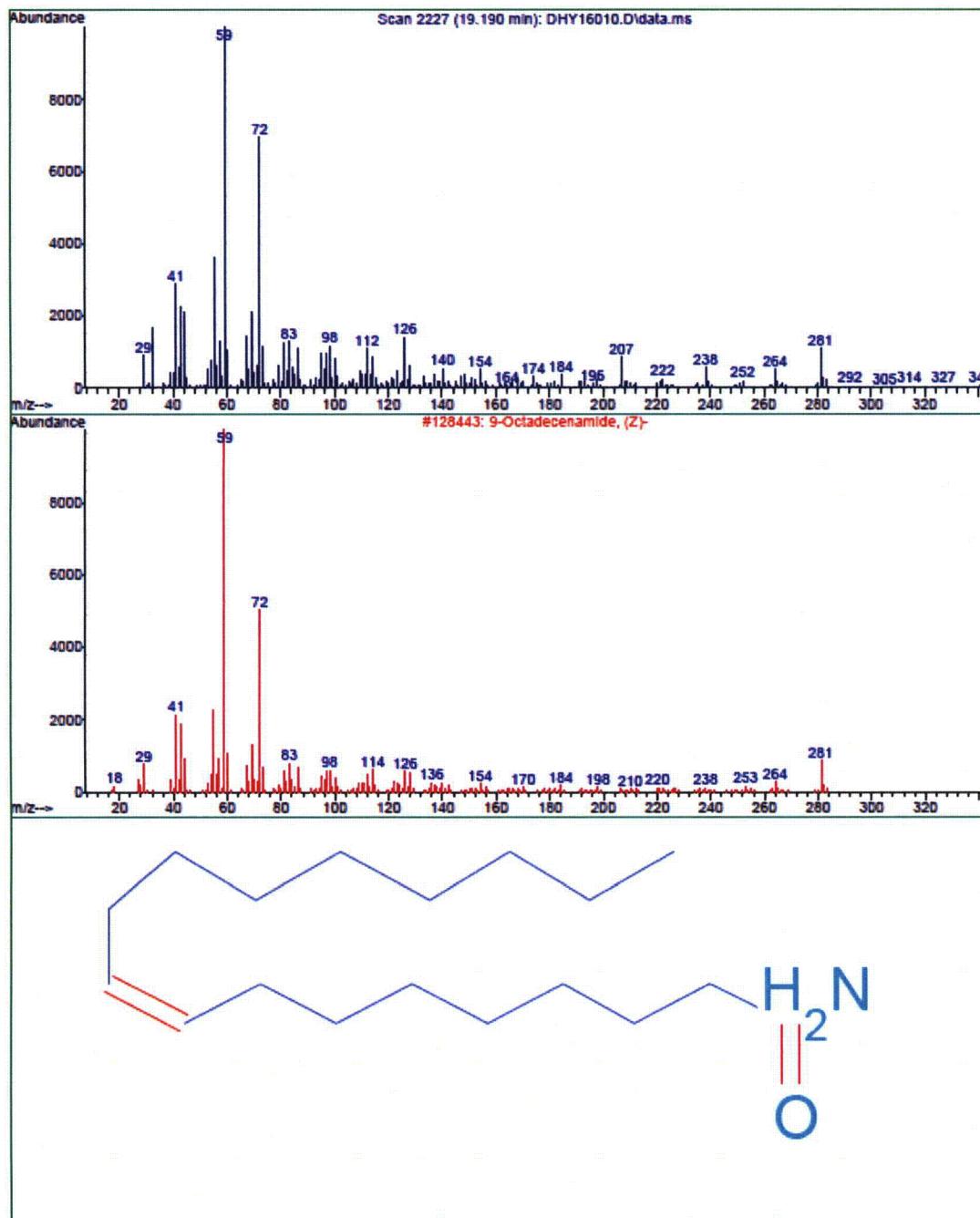


Spectrum 5-6



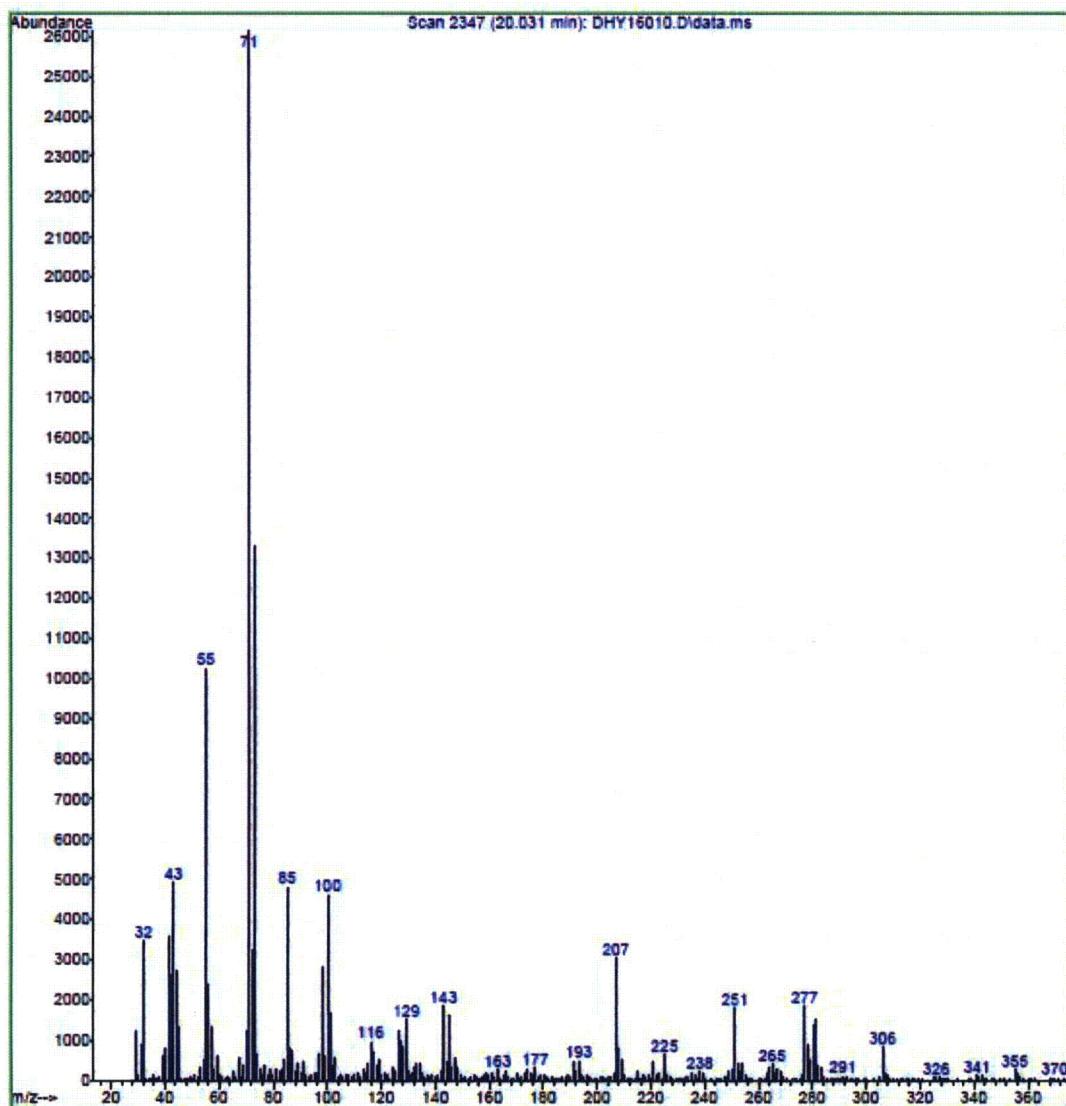
Spectrum 5-7

Library Searched : C:\Database\NIST11.L
Quality : 98
ID : 9-Octadecenamide, (Z)-



Spectrum 5-8

File : Y:\March13\DHY16010.D
Operator : X. CAI
Acquired : 20 Mar 2013 10:48 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch Brite Used
Misc Info : 1 ul
Vial Number: 1



Spectrum 5-9

File : Y:\March13\DHY16010.D
Operator : X. CAI
Acquired : 20 Mar 13 10:48 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: Scotch Brite Used
Misc Info : 1 ul
Vial Number: 1

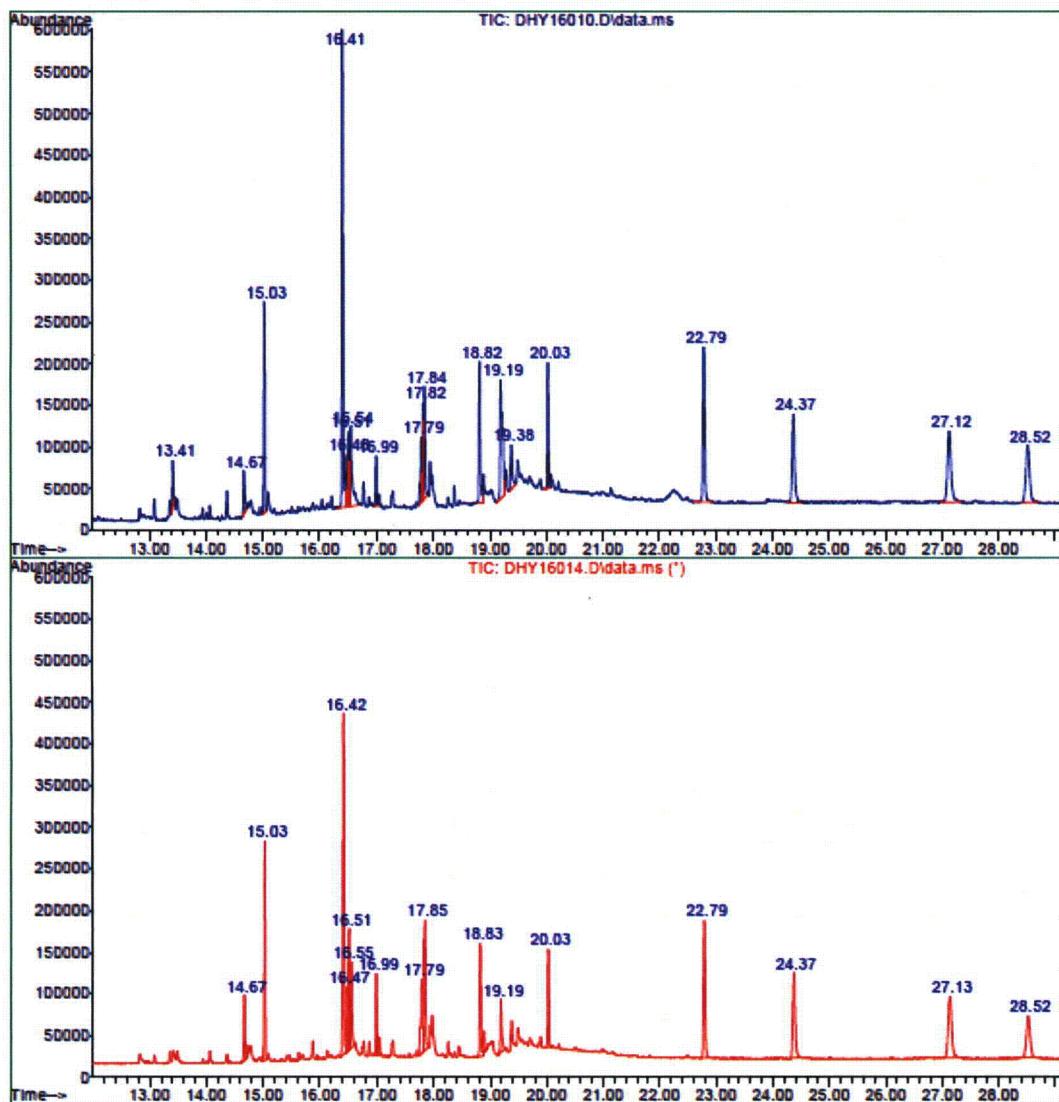


Figure 6

```
File      : Y:\March13\BSB\DHY16016.D
Operator   : [BSB1]X. CAI
Acquired  : 21 Mar 2013  9:11      using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Control
Misc Info  : 1 ul
Vial Number: 1
```

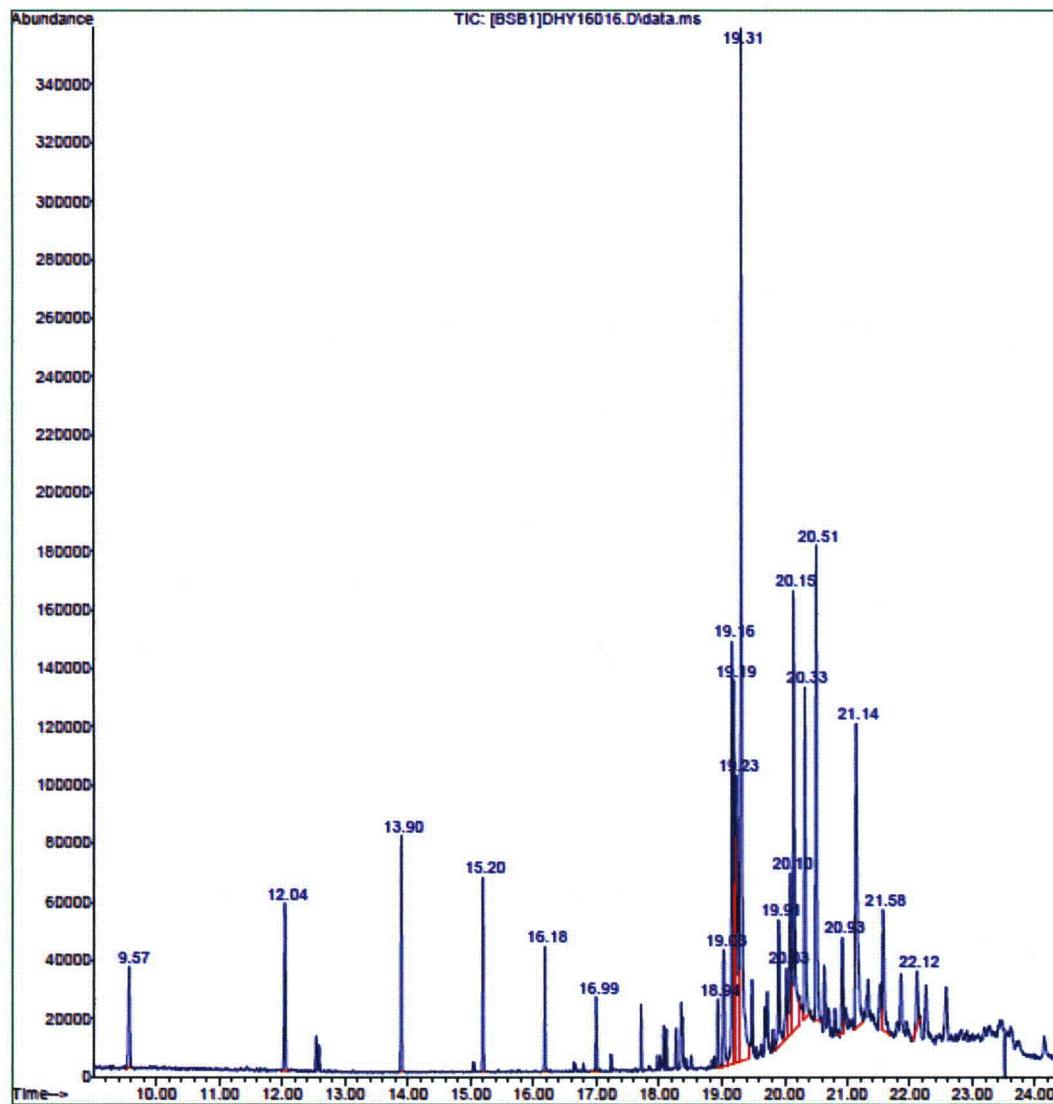
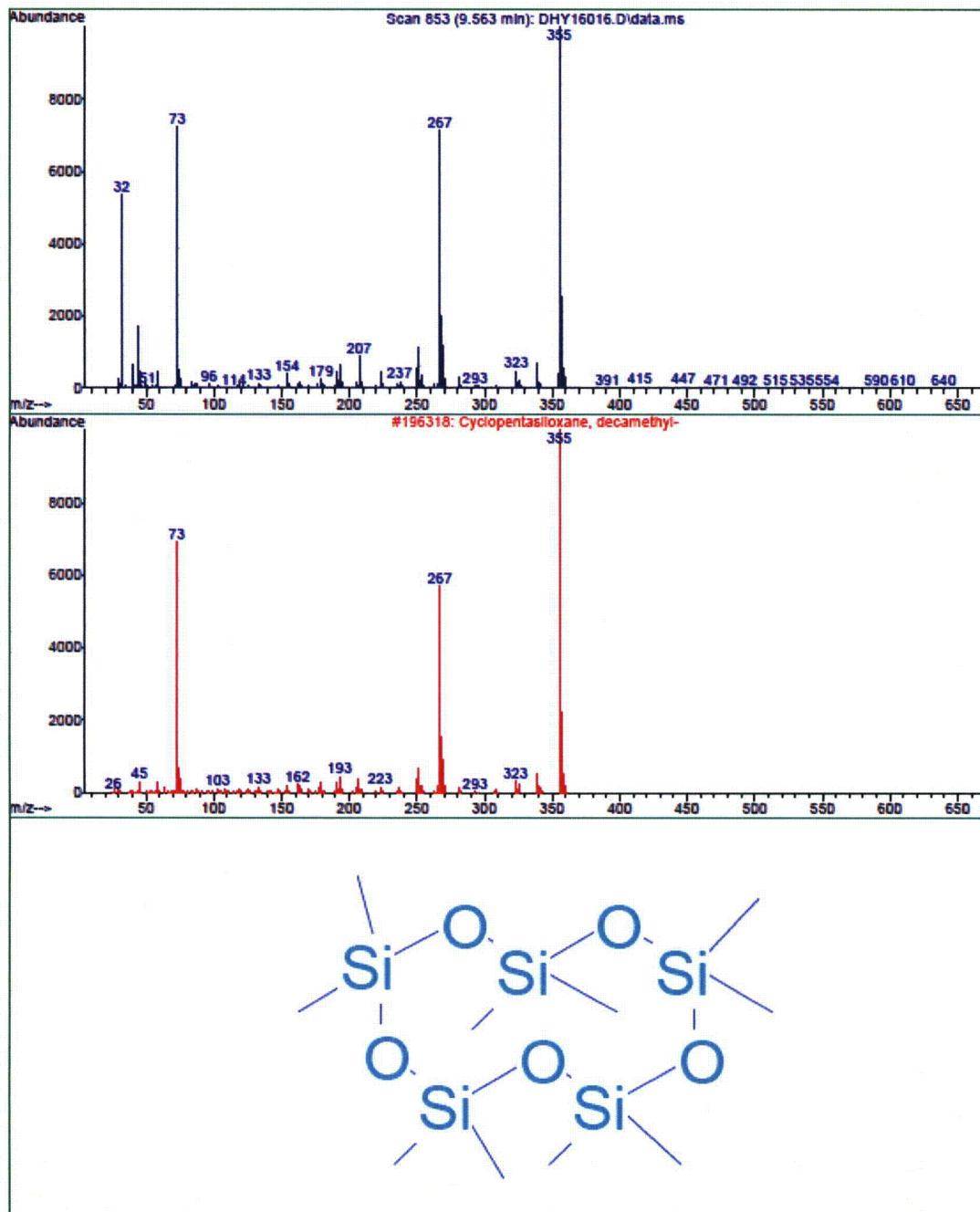


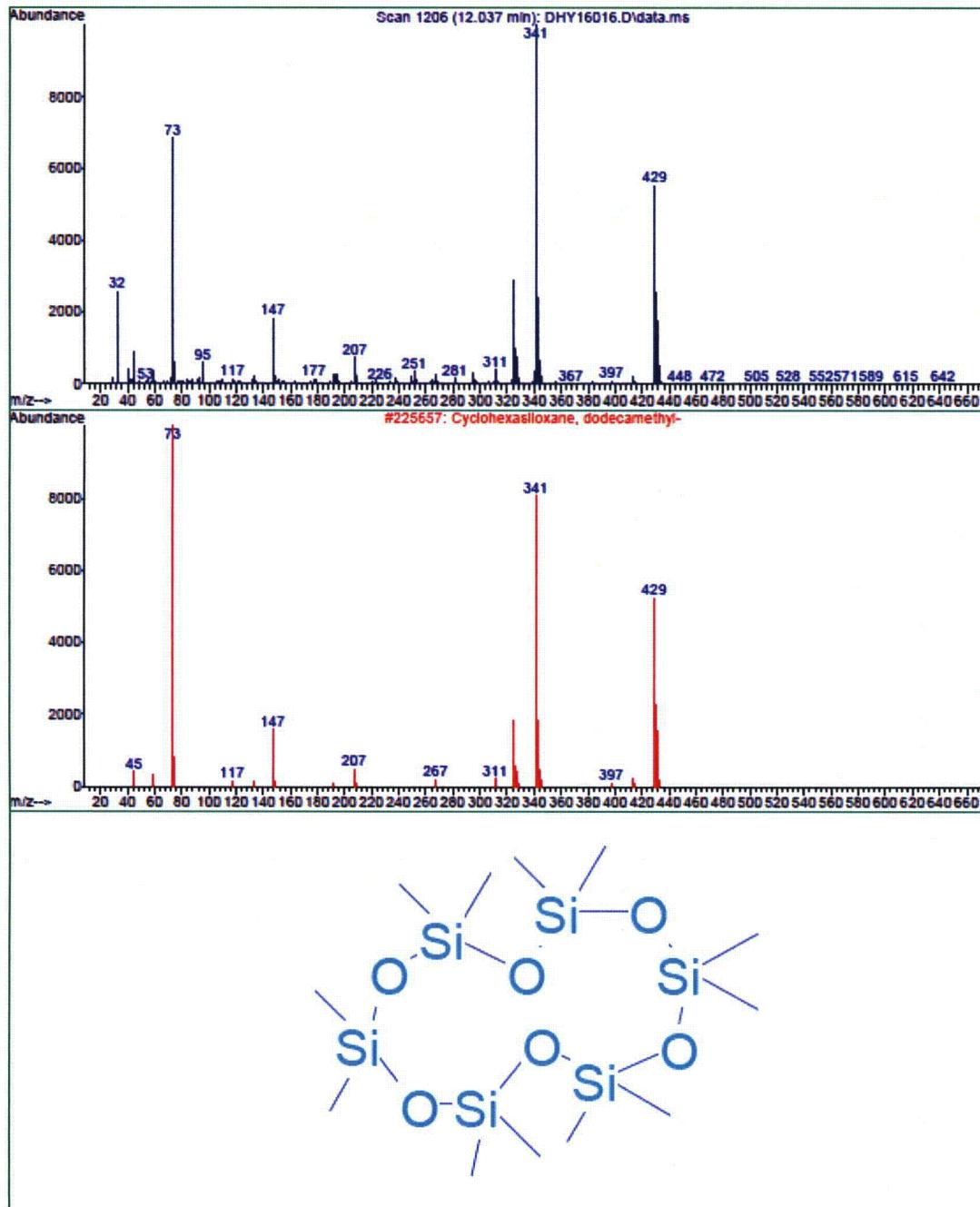
Figure 7

Library Searched : C:\Database\NIST11.L
Quality : 90
ID : Cyclopentasiloxane, decamethyl-



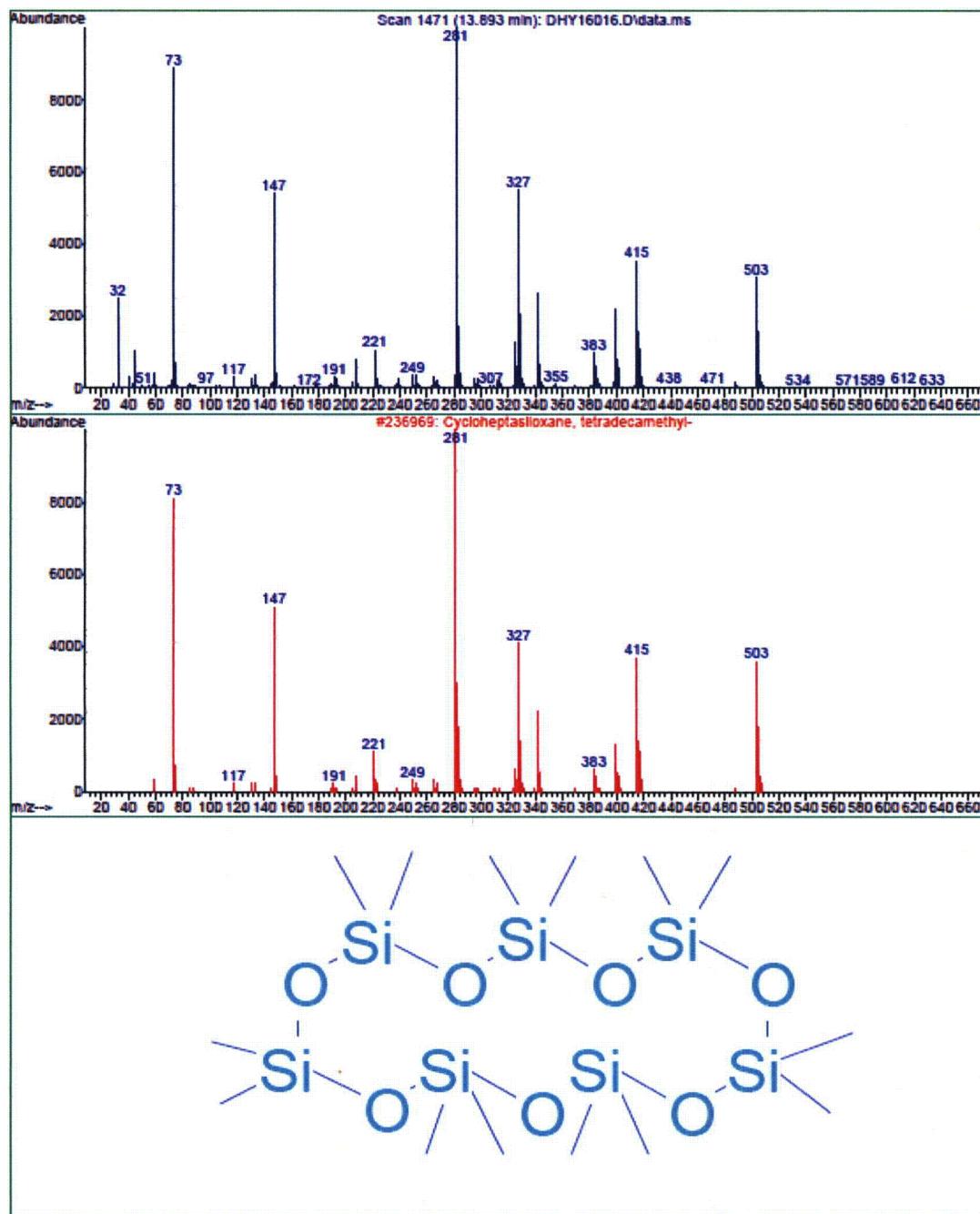
Spectrum 7-1

Library Searched : C:\Database\NIST11.L
Quality : 90
ID : Cyclohexasiloxane, dodecamethyl-



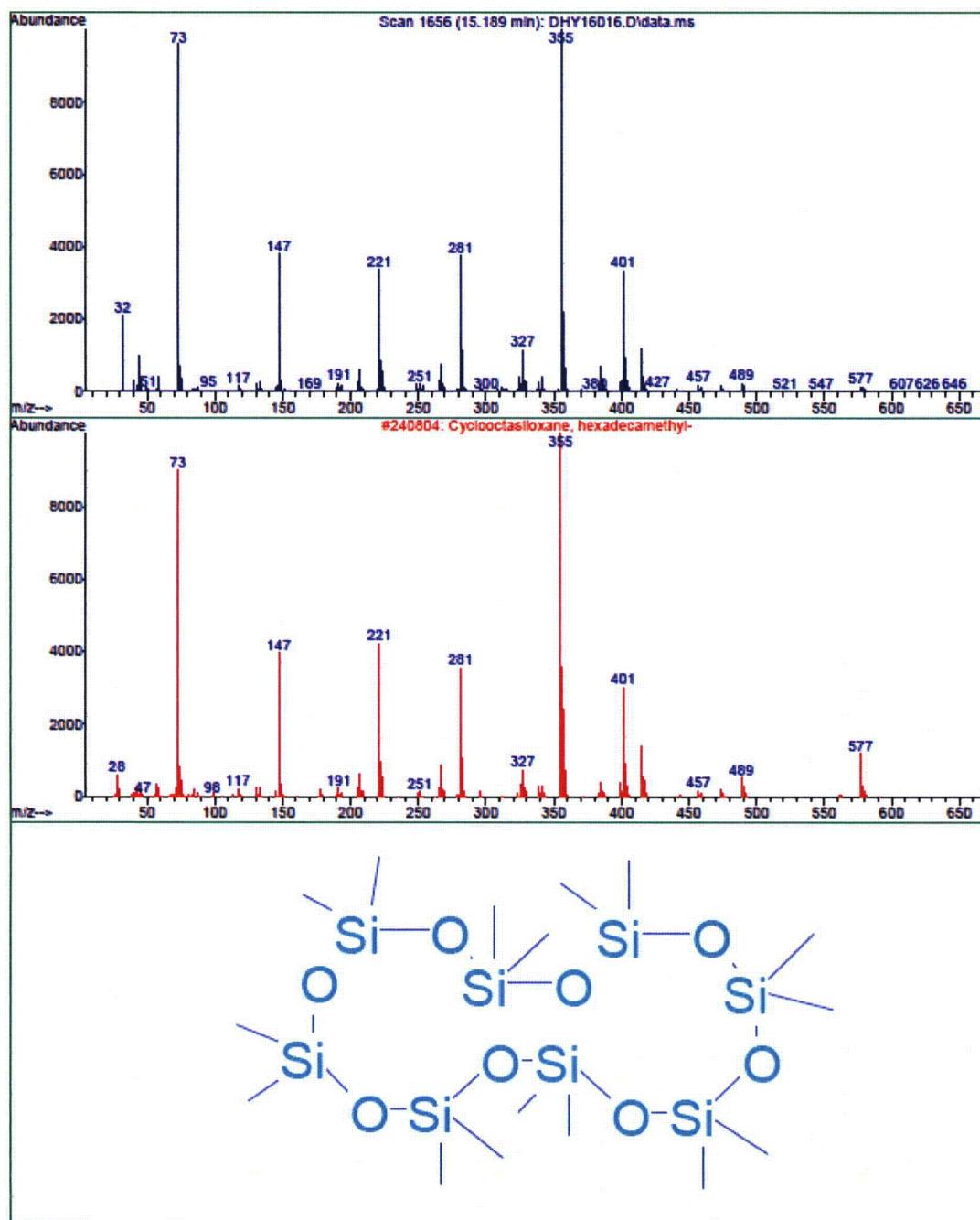
Spectrum 7-2

Library Searched : C:\Database\NIST11.L
Quality : 93
ID : Cycloheptasiloxane, tetradecamethyl-



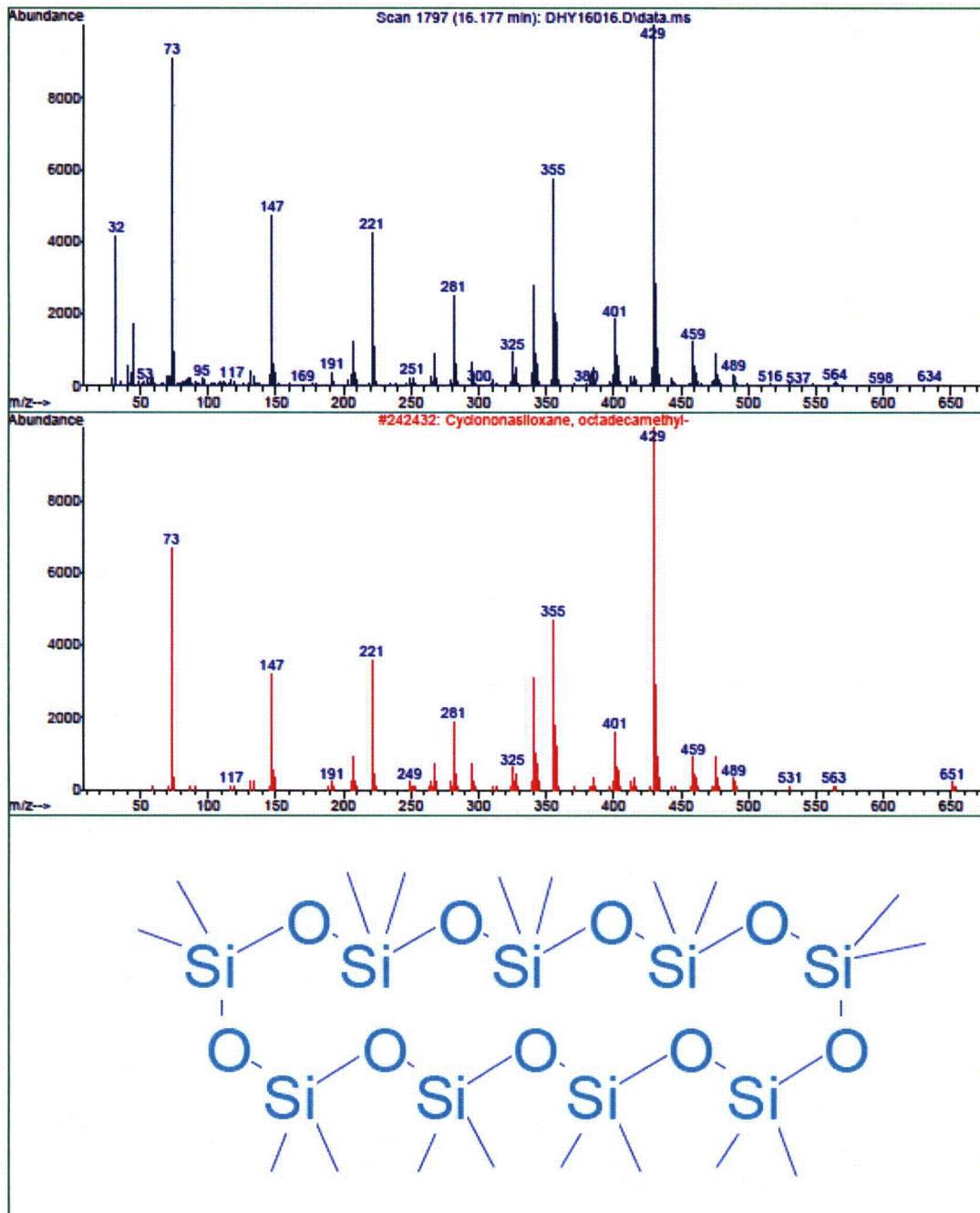
Spectrum 7-3

Library Searched : C:\Database\NIST11.L
Quality : 91
ID : Cyclooctasiloxane, hexadecamethyl-



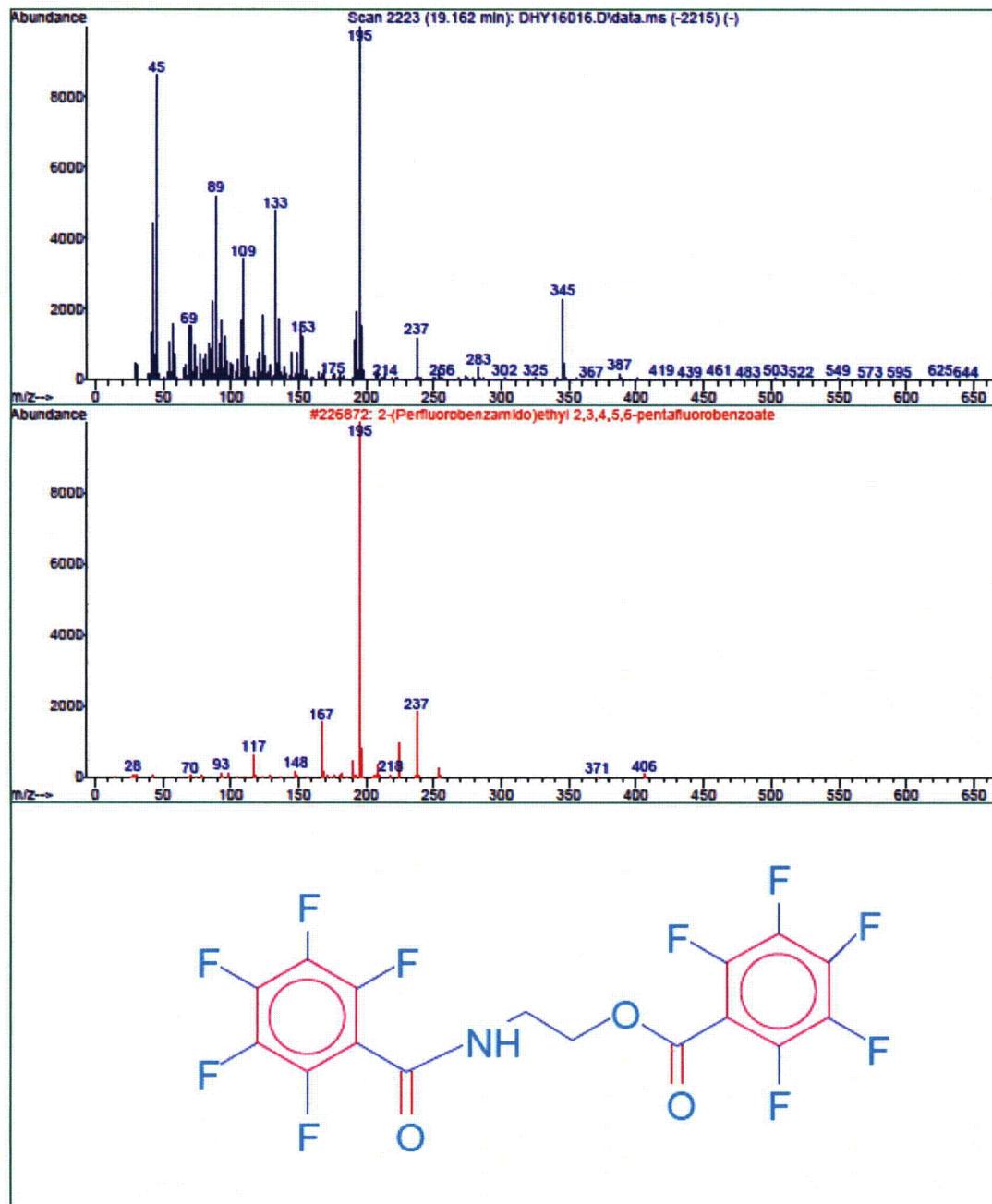
Spectrum 7-4

Library Searched : C:\Database\NIST11.L
Quality : 95
ID : Cyclononasiloxane, octadecamethyl-



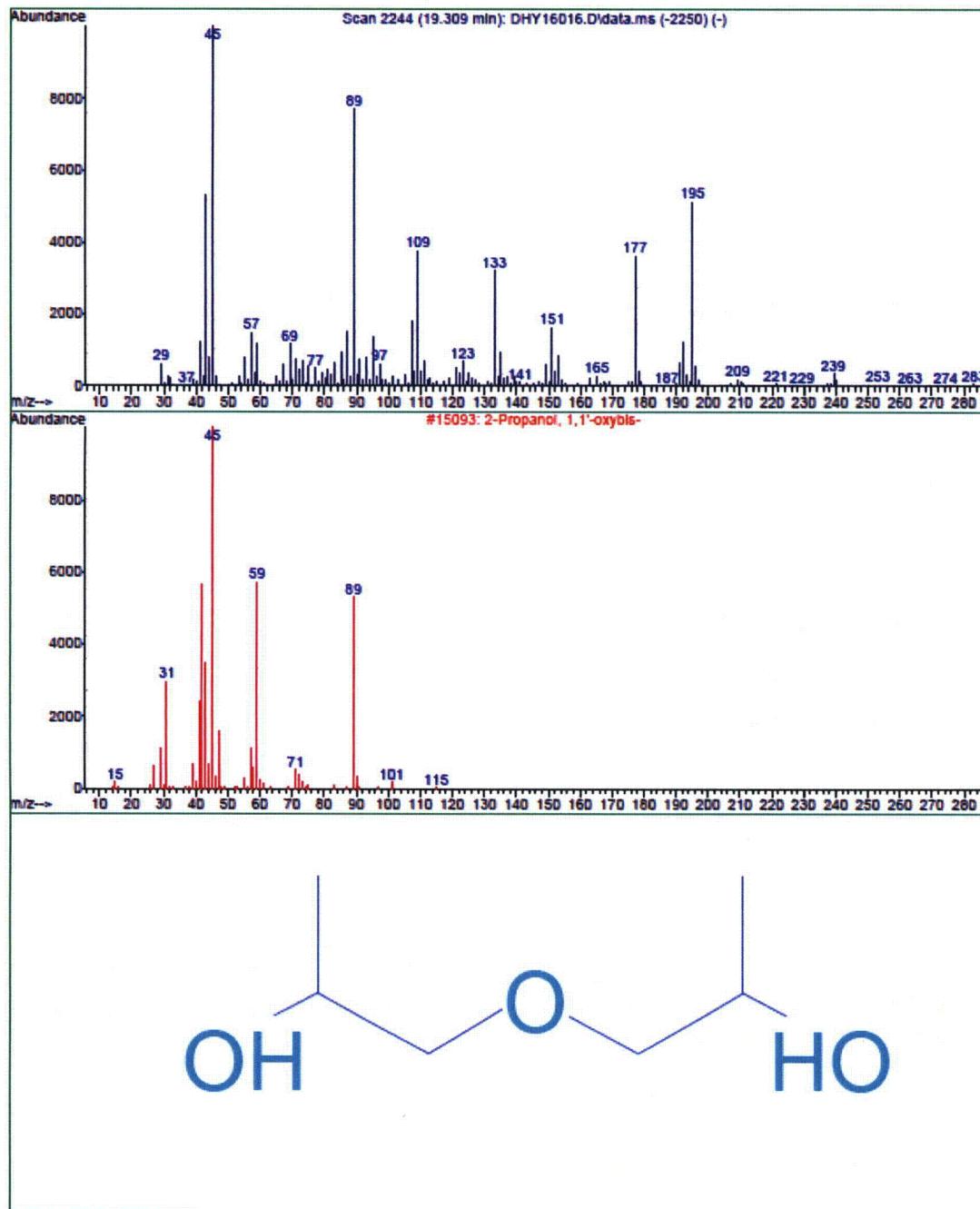
Spectrum 7-5

Library Searched : C:\Database\NIST11.L
Quality : 32
ID : 2-(Perfluorobenzamido)ethyl 2,3,4,5,6-pentafluorobenz
oate

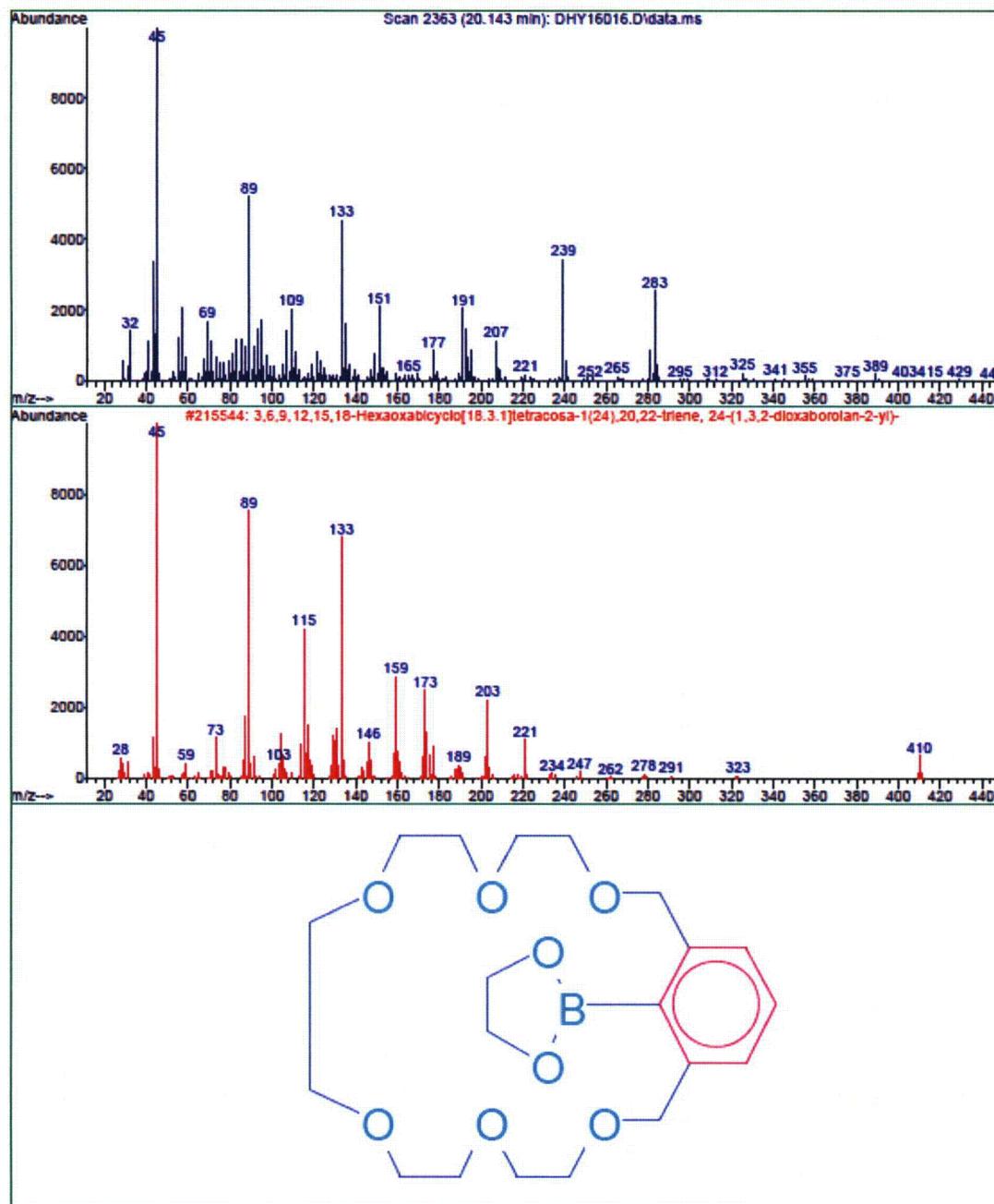


Spectrum 7-6

Library Searched : C:\Database\NIST11.L
Quality : 40
ID : 2-Propanol, 1,1'-oxybis-

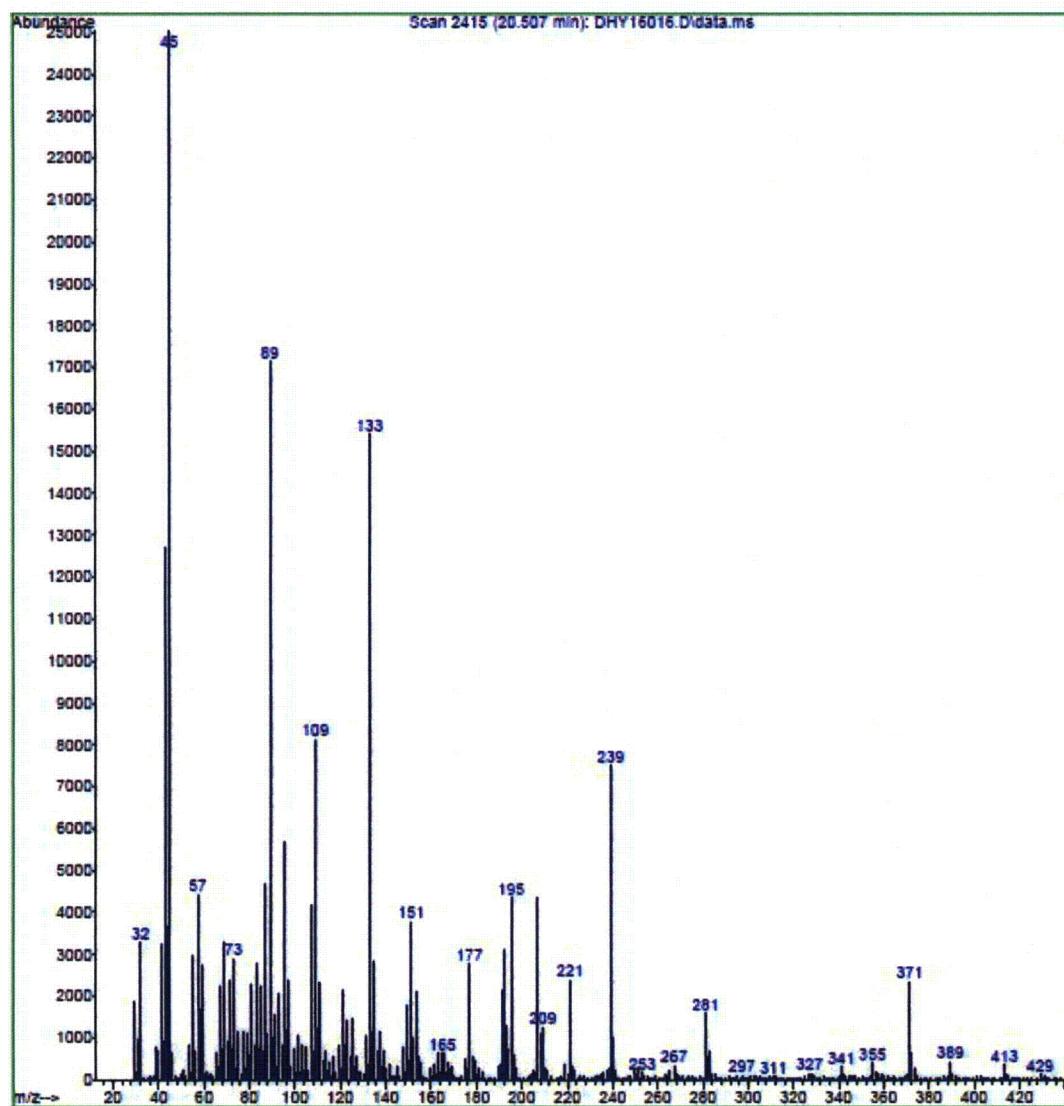


Library Searched : C:\Database\NIST11.L
Quality : 38
ID : 3,6,9,12,15,18-Hexaoxabicyclo[18.3.1]tetracosa-1(24),
20,22-triene, 24-(1,3,2-dioxaborolan-2-yl)-



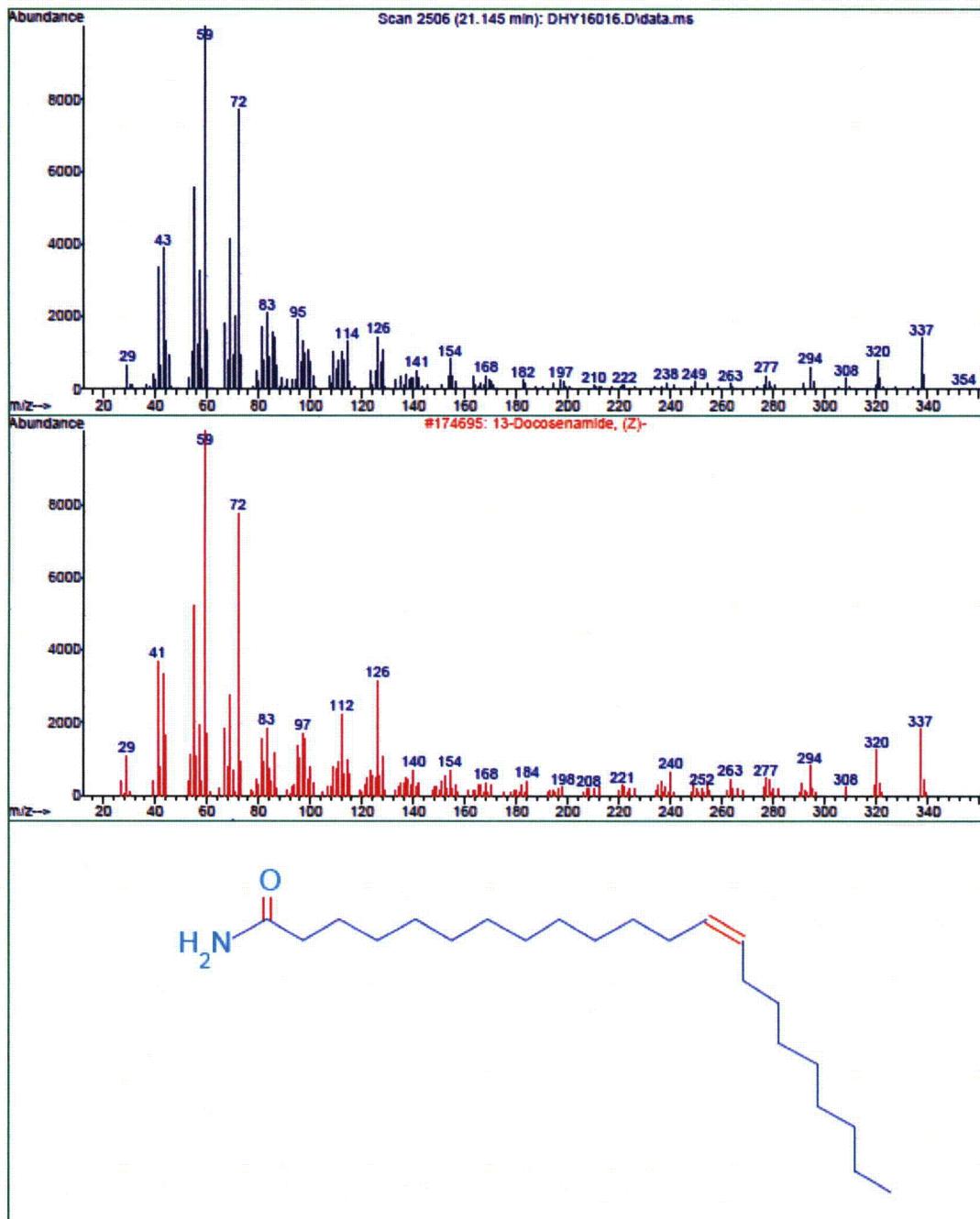
Spectrum 7-8

```
File      : Y:\March13\DHY16016.D
Operator   : X. CAI
Acquired  : 21 Mar 2013    9:11      using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Control
Misc Info  : 1 ul
Vial Number: 1
```



Spectrum 7-9

Library Searched : C:\Database\NIST11.L
Quality : 94
ID : 13-Docosenamide, (Z)-



Spectrum 7-10

File : Y:\March13\BSB\DHY16020.D
Operator : [BSB1]X. CAI
Acquired : 21 Mar 2013 11:50 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1

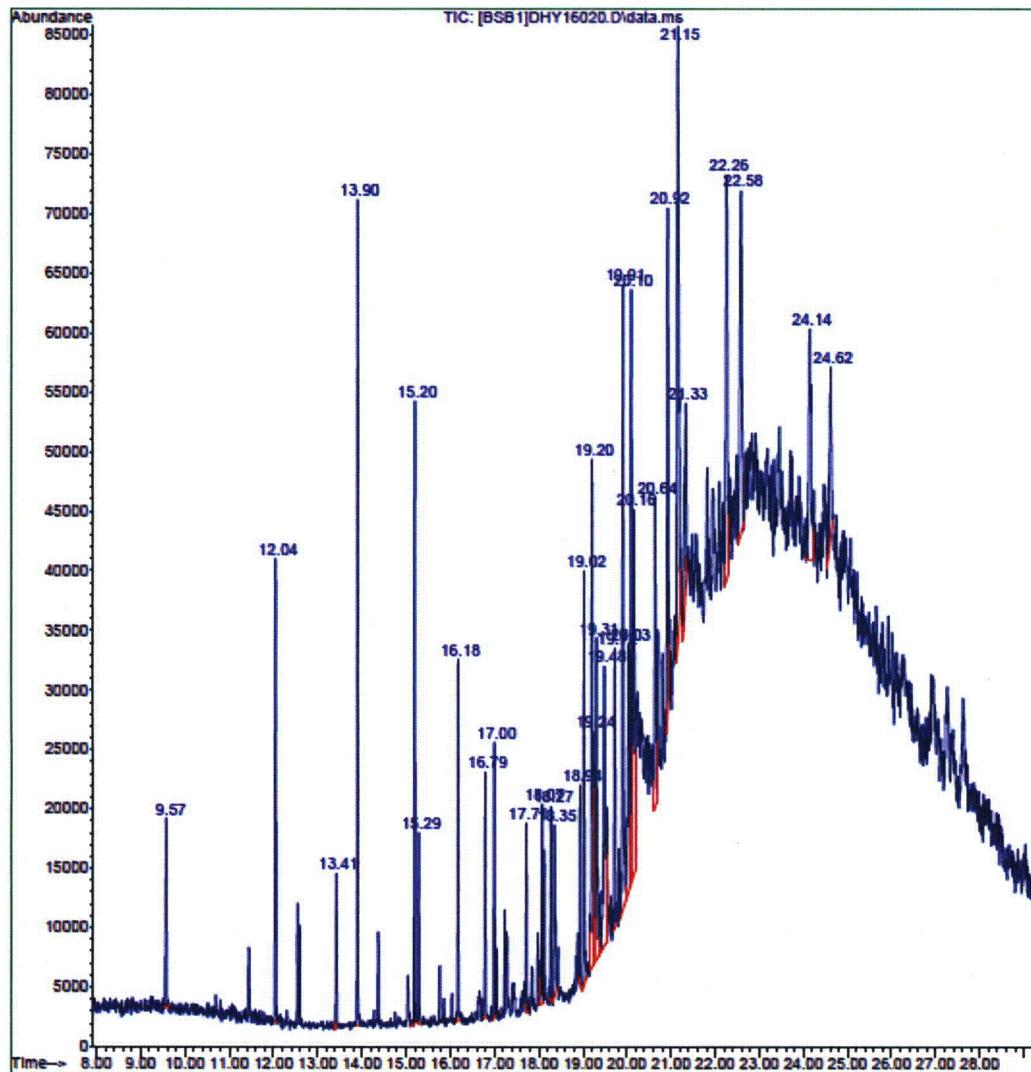
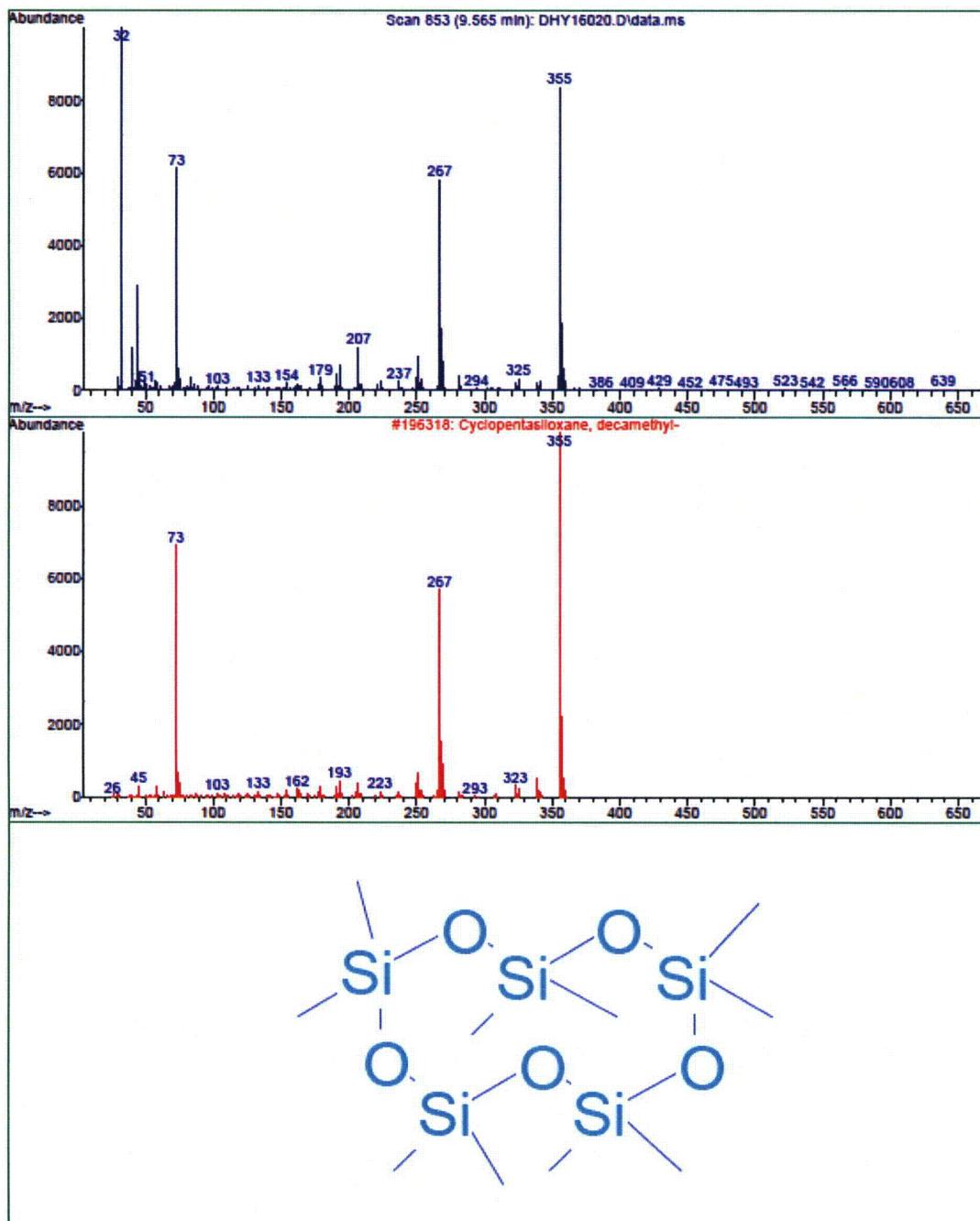


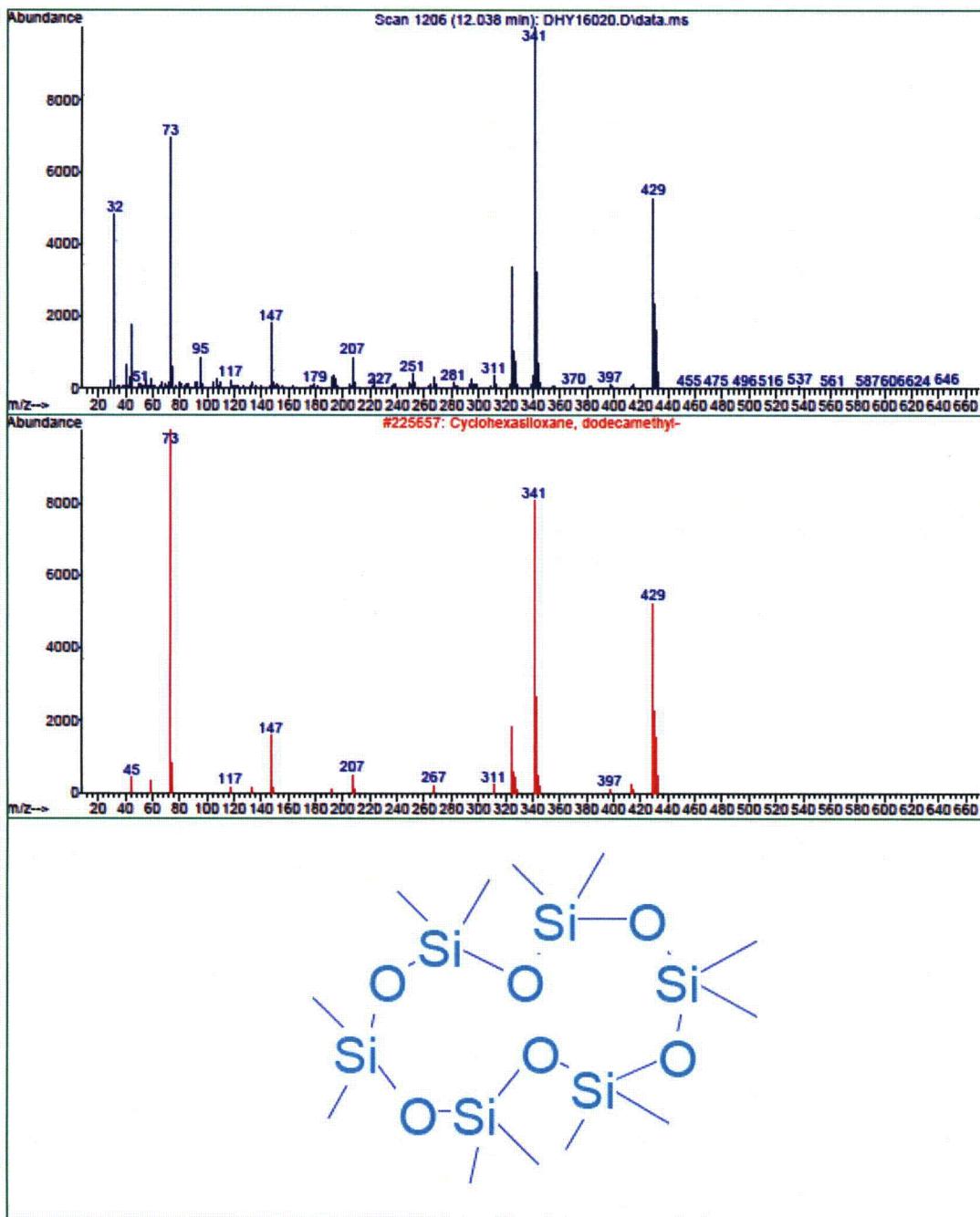
Figure 8

Library Searched : C:\Database\NIST11.L
Quality : 91
ID : Cyclopentasiloxane, decamethyl-



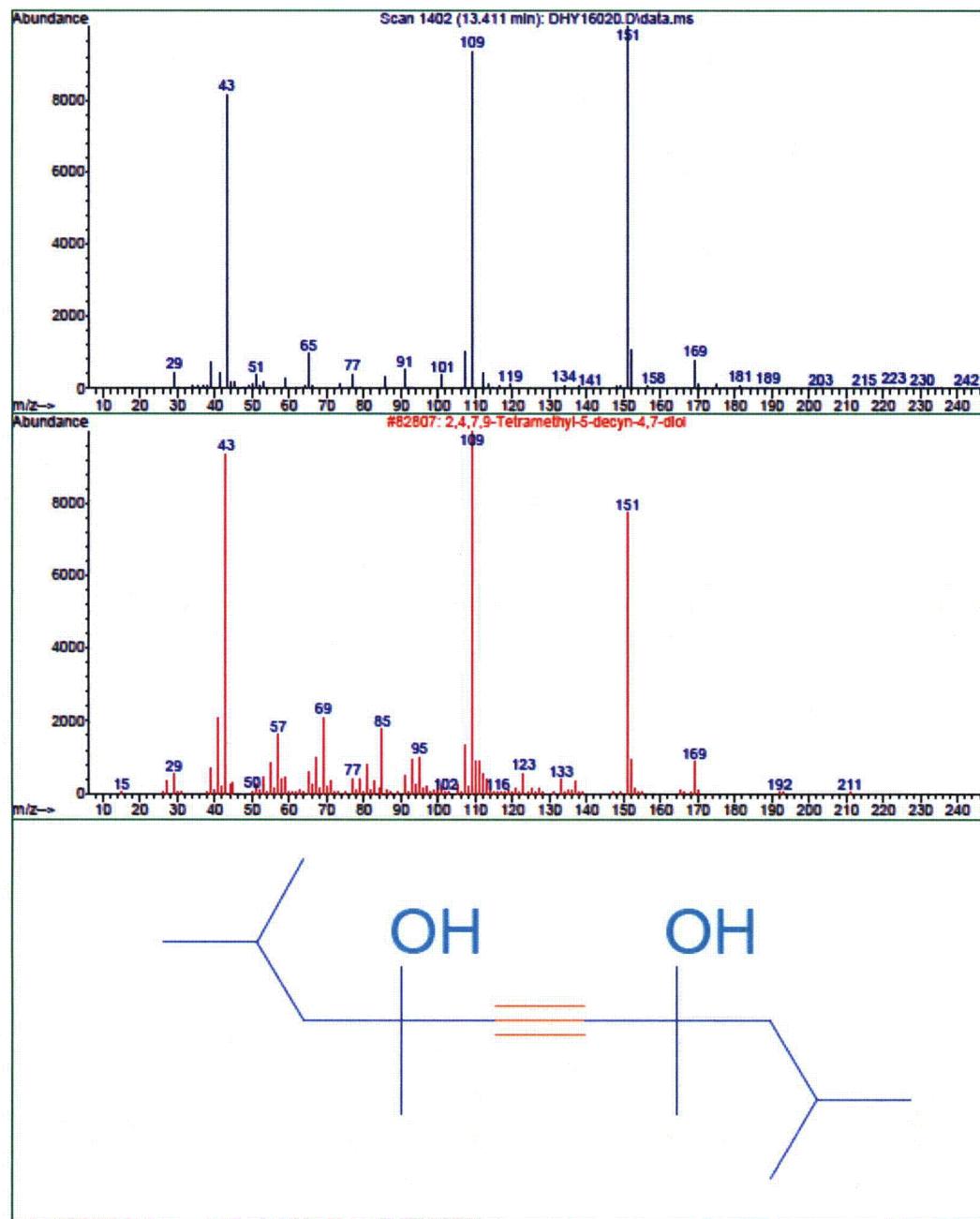
Spectrum 8-1

Library Searched : C:\Database\NIST11.L
Quality : 93
ID : Cyclohexasiloxane, dodecamethyl-



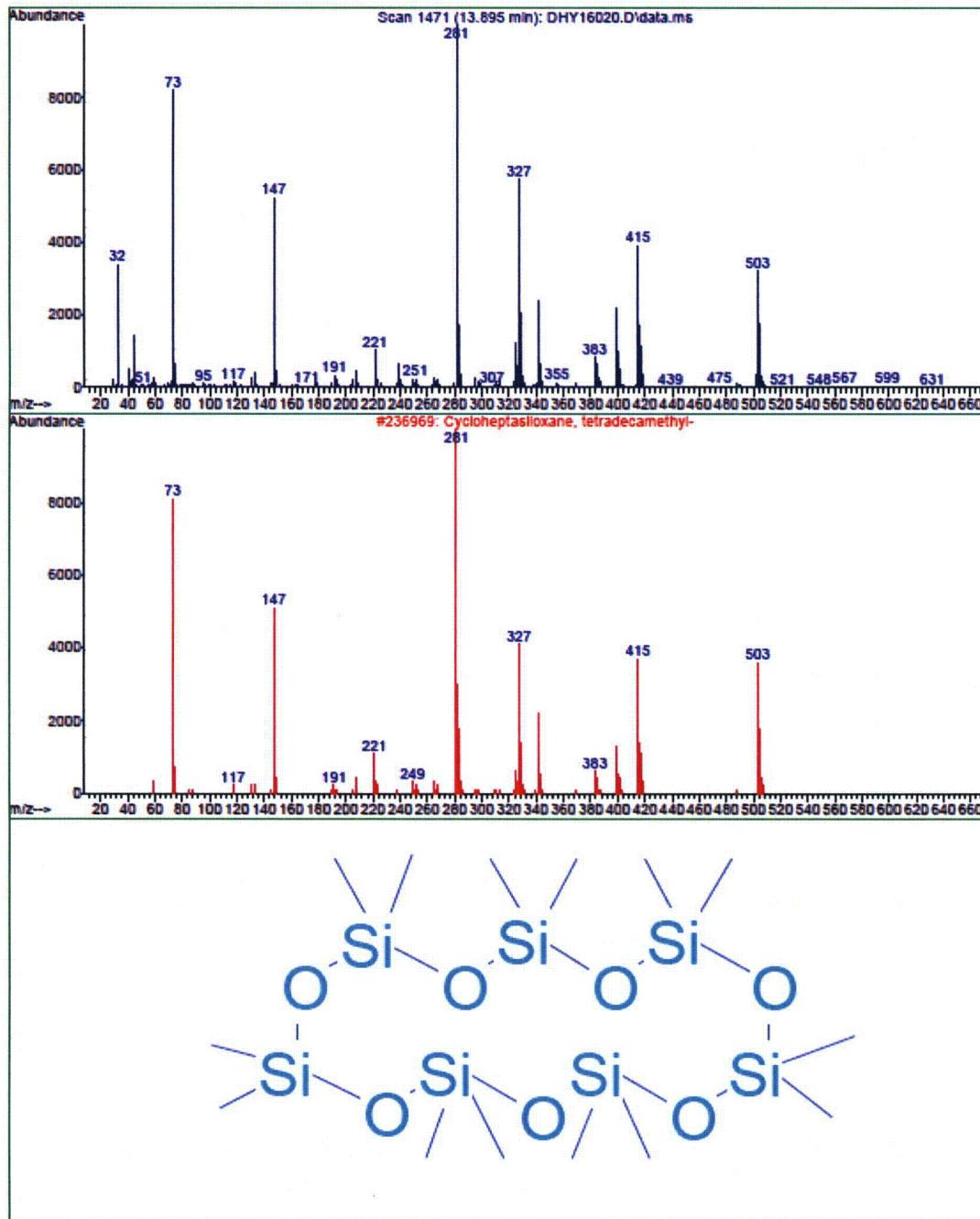
Spectrum 8-2

Library Searched : C:\Database\NIST11.L
Quality : 42
ID : 2,4,7,9-Tetramethyl-5-decyn-4,7-diol



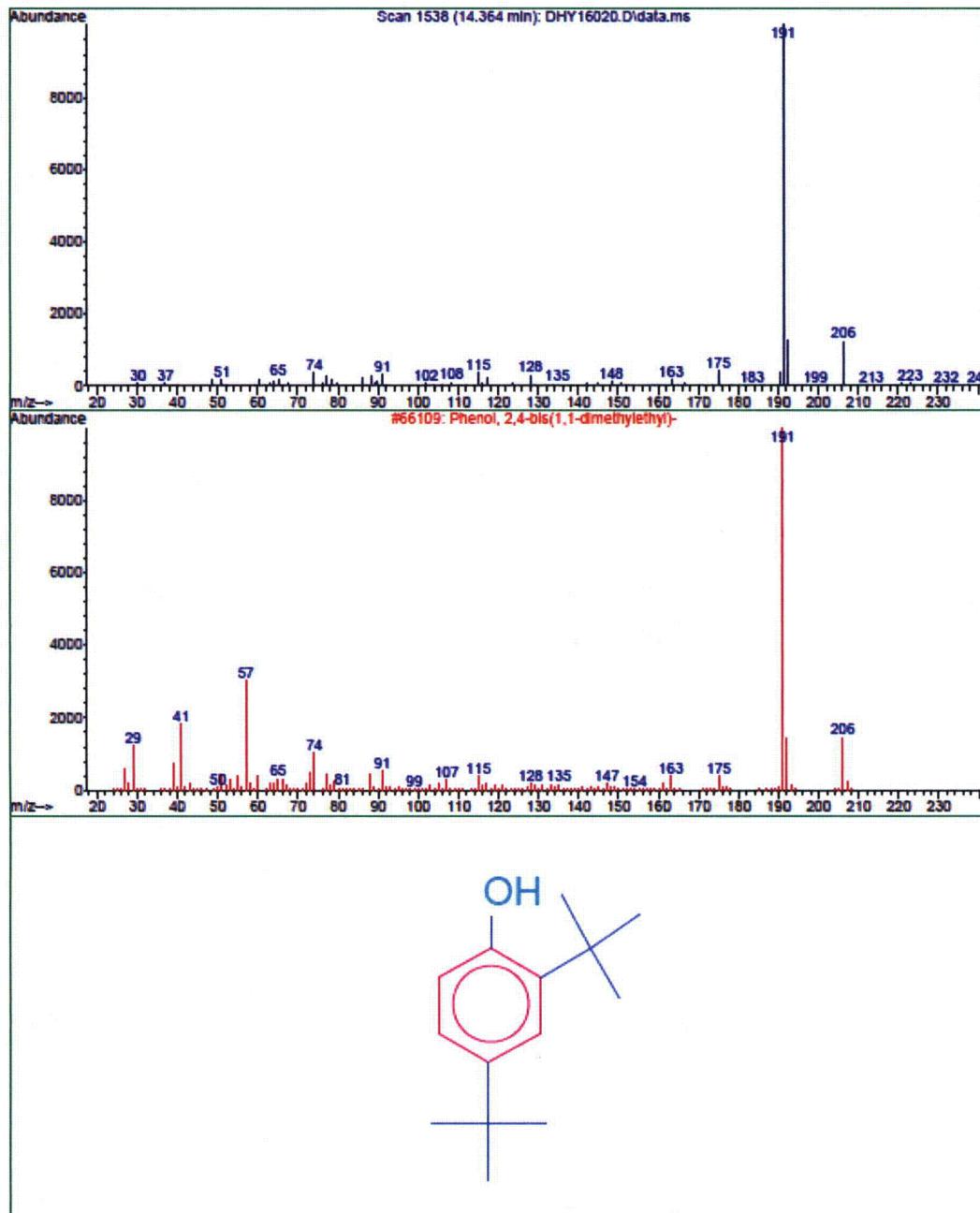
Spectrum 8-3

Library Searched : C:\Database\NIST11.L
Quality : 93
ID : Cycloheptasiloxane, tetradecamethyl-



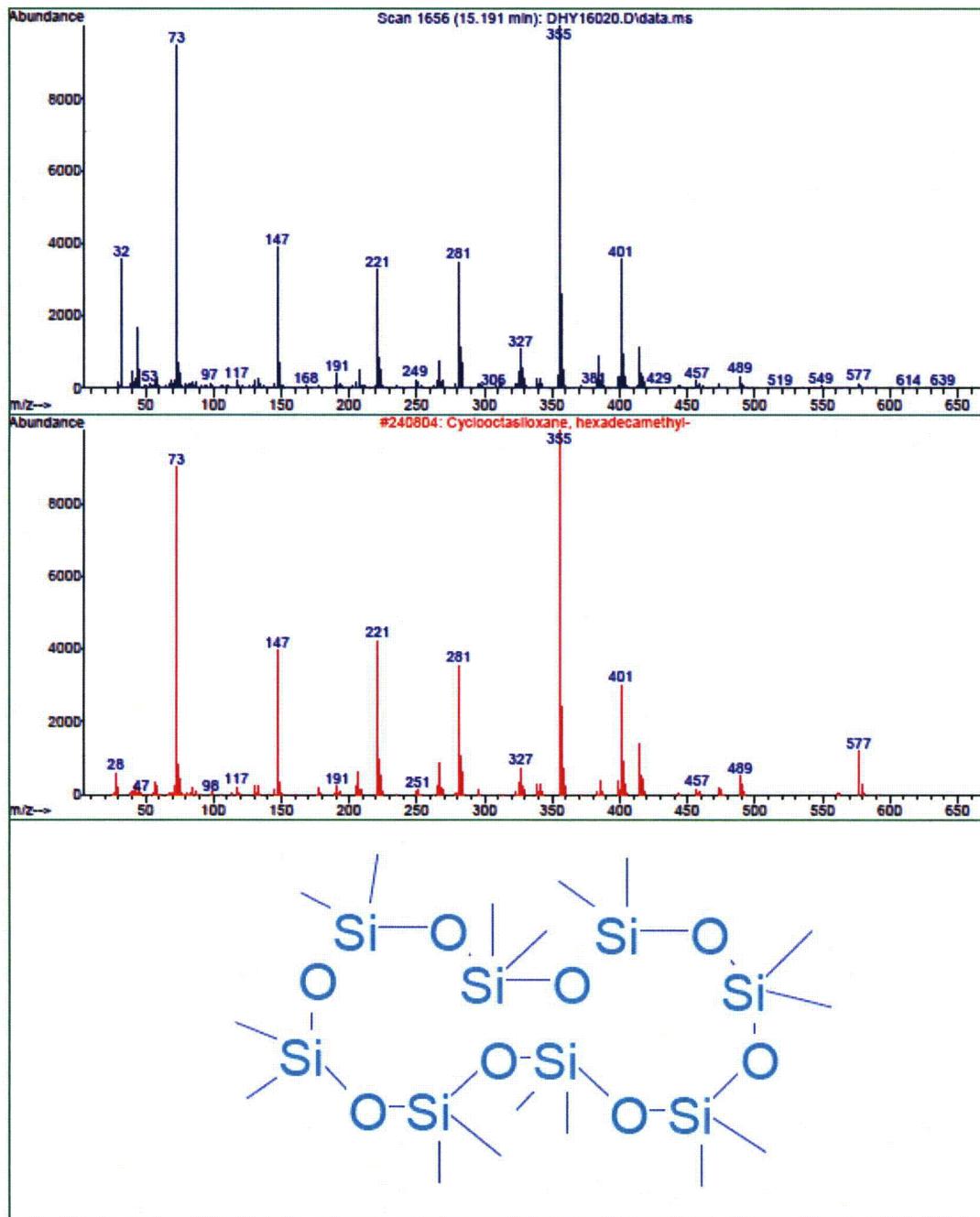
Spectrum 8-4

Library Searched : C:\Database\NIST11.L
Quality : 72
ID : Phenol, 2,4-bis(1,1-dimethylethyl)-



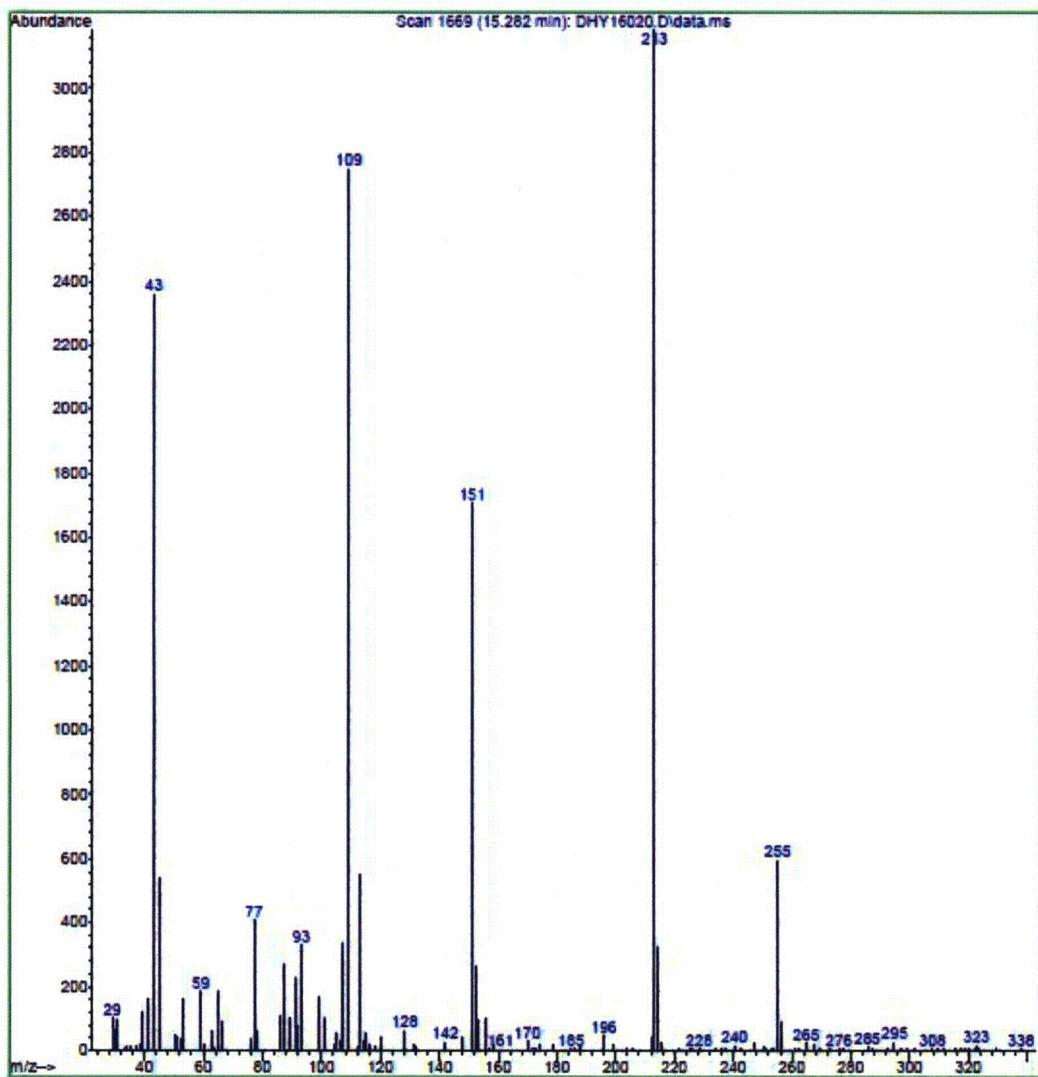
Spectrum 8-5

Library Searched : C:\Database\NIST11.L
Quality : 91
ID : Cyclooctasiloxane, hexadecamethyl-



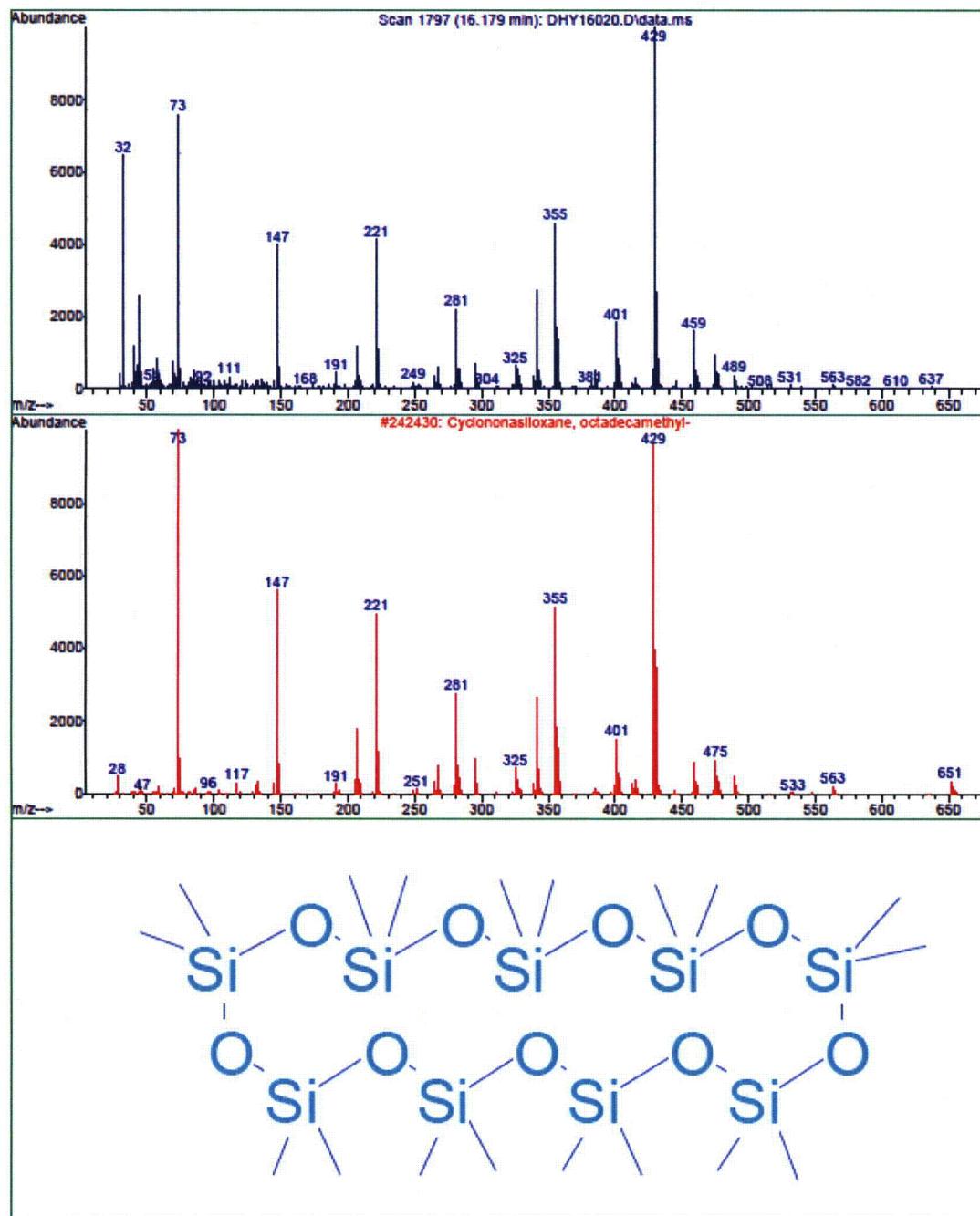
Spectrum 8-6

File : Y:\March13\BSB\DHY16020.D
Operator : [BSB1]X. CAI
Acquired : 21 Mar 13 11:50 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1



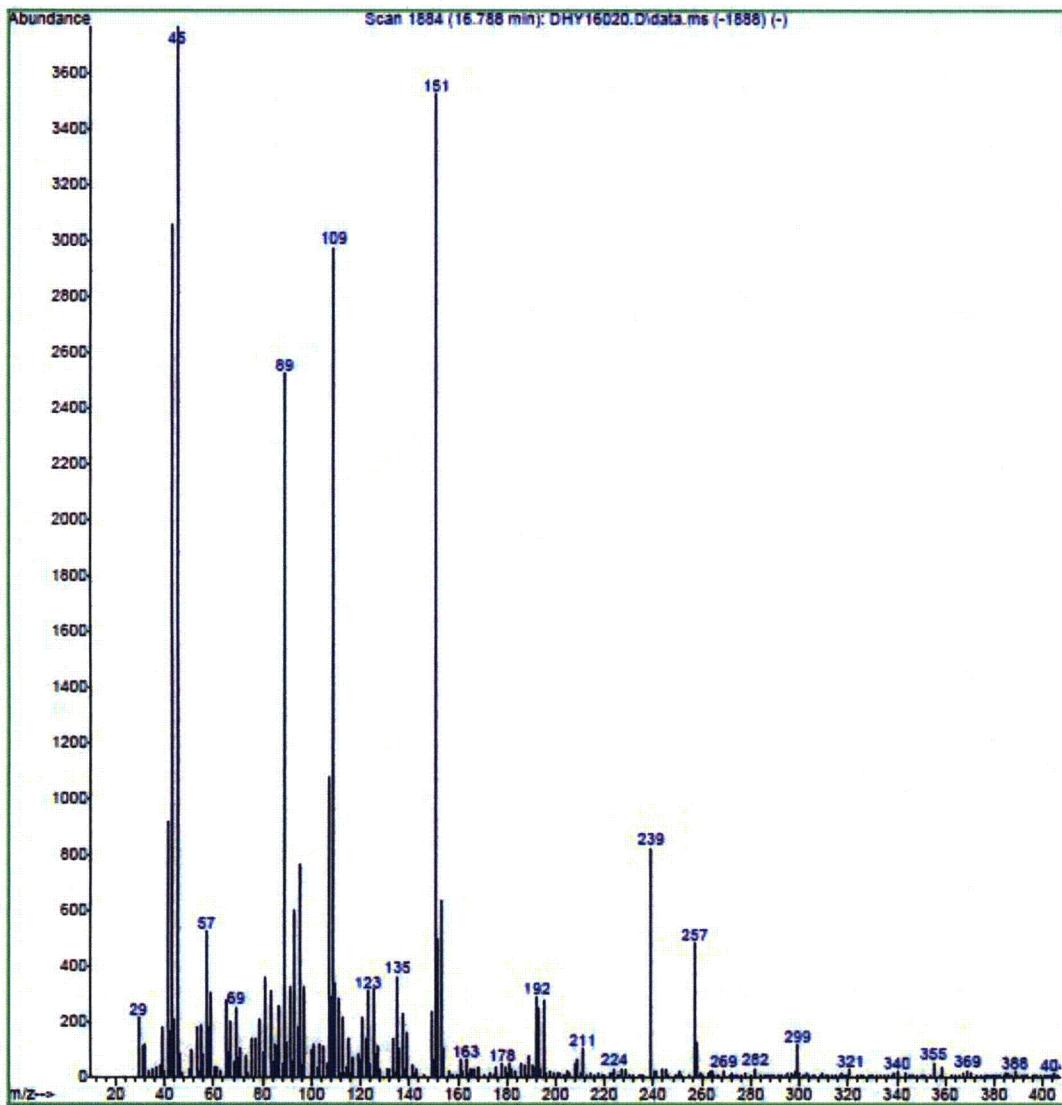
Spectrum 8-7

Library Searched : C:\Database\NIST11.L
Quality : 95
ID : Cyclononasiloxane, octadecamethyl-



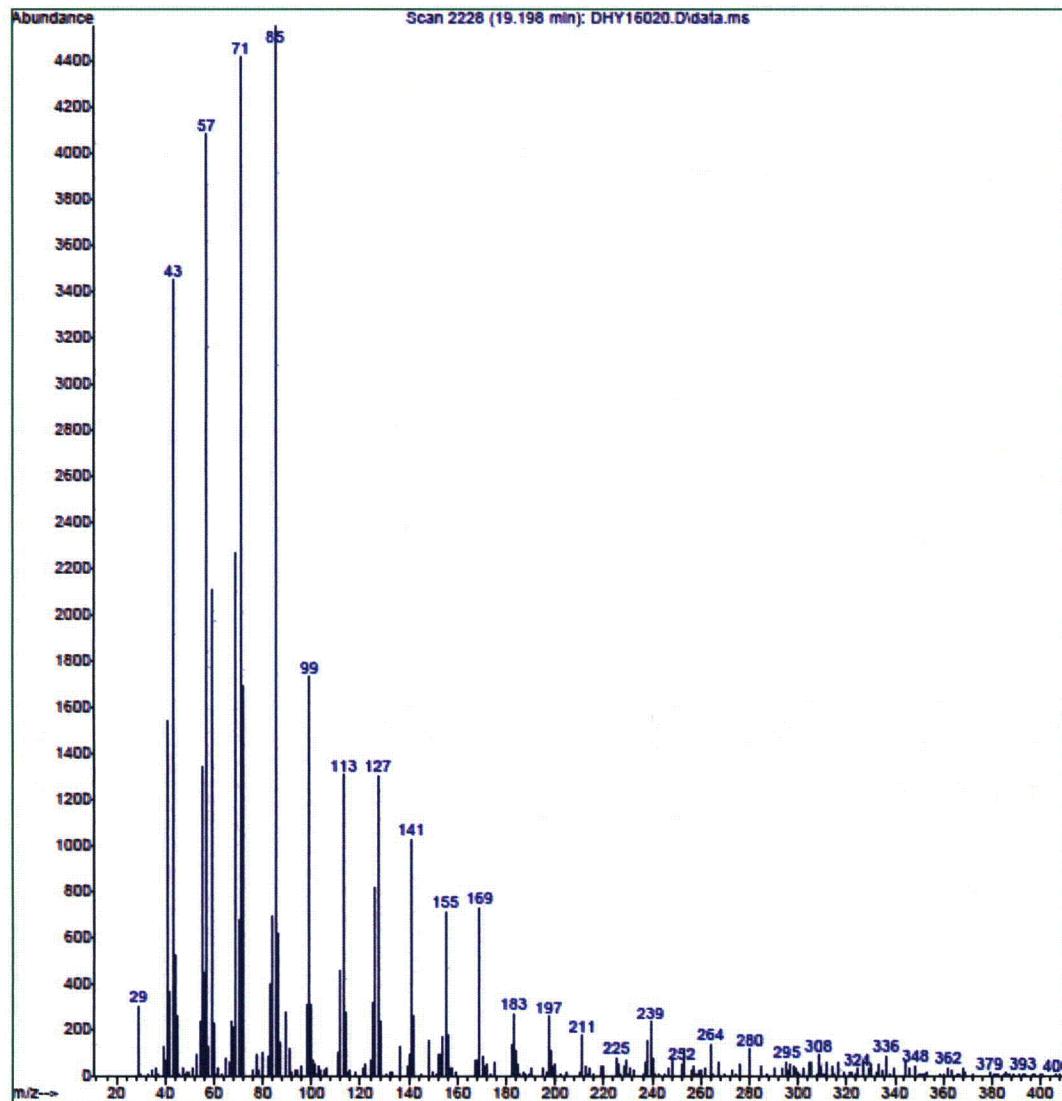
Spectrum 8-8

File : Y:\March13\DHY16020.D
Operator : X. CAI
Acquired : 21 Mar 2013 11:50 using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1



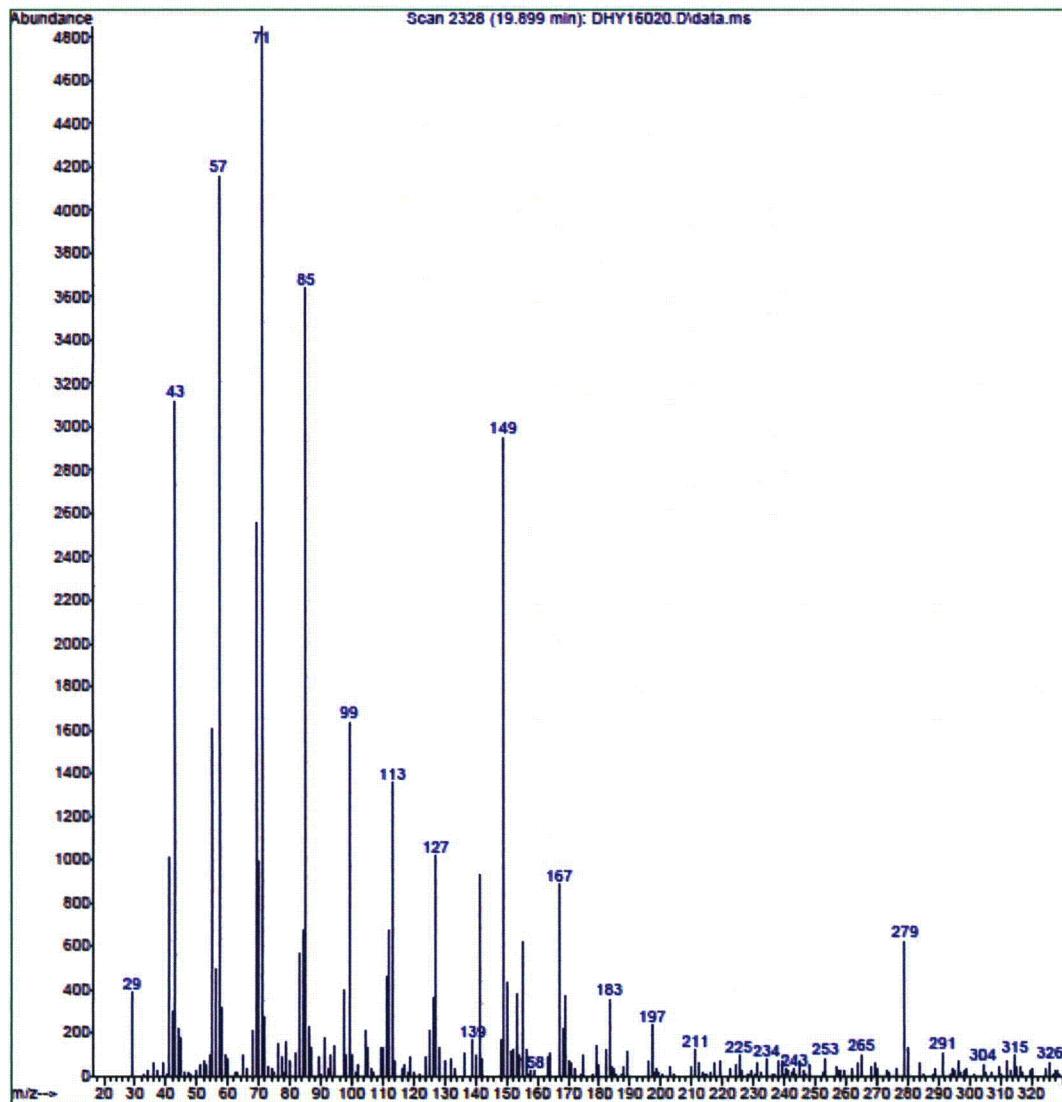
Spectrum 8-9

File :Y:\March13\BSB\DHY16020.D
Operator : [BSB1]X. CAI
Acquired : 21 Mar 13 11:50 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1



Spectrum 8-10

File : Y:\March13\BSB\DHY16020.D
Operator : [BSB1]X. CAI
Acquired : 21 Mar 13 11:50 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1



Spectrum 8-11

File : Y:\March13\BSB\DHY16020.D
Operator : [BSB1]X. CAI
Acquired : 21 Mar 13 11:50 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1

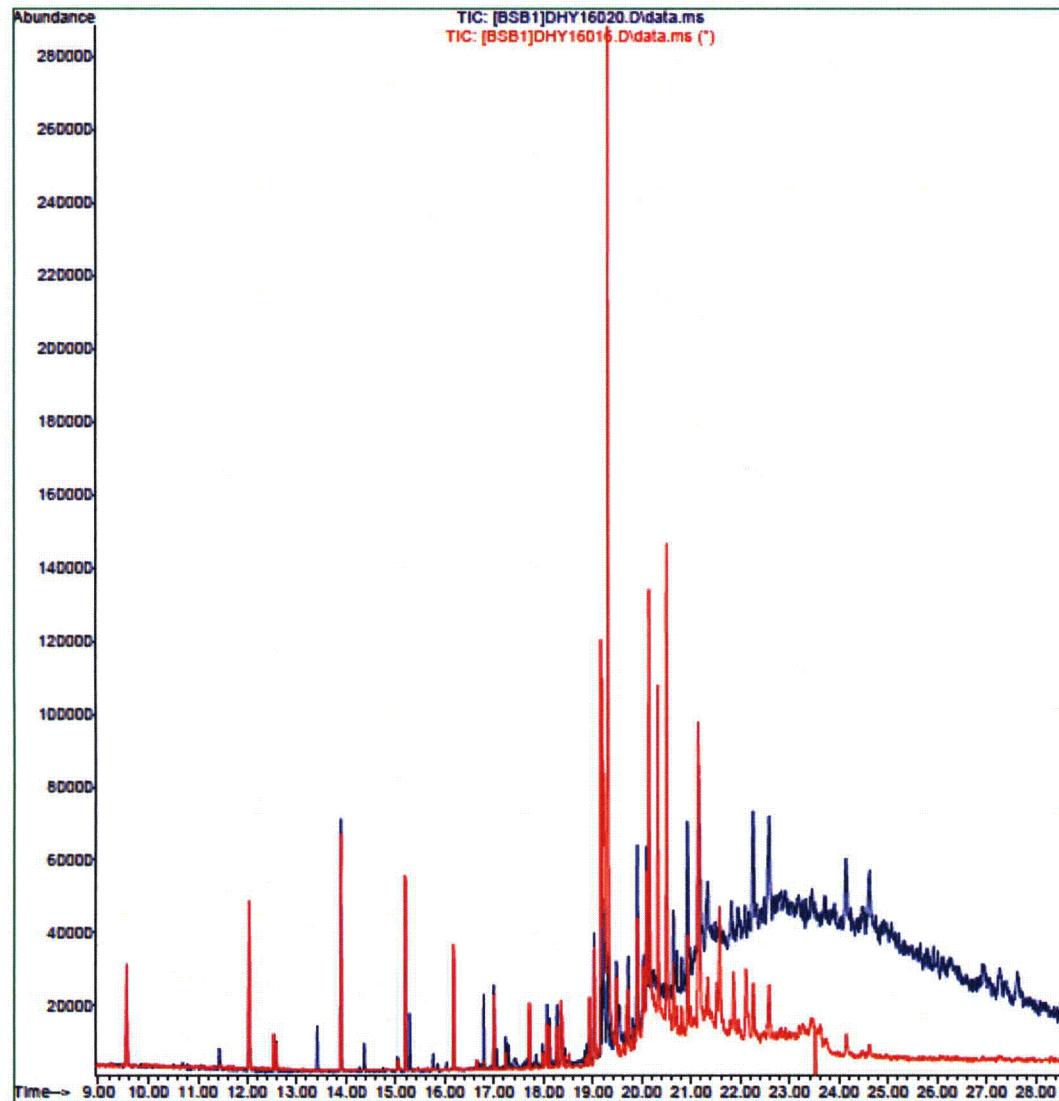


Figure 9

File : Y:\March13\BSB\DHY16020.D
Operator : [BSB1]X. CAI
Acquired : 21 Mar 13 11:50 am using AcqMethod STDSPL
Instrument : GC/MS Ins
Sample Name: SaltSmart Used concentrated
Misc Info : 1 ul
Vial Number: 1

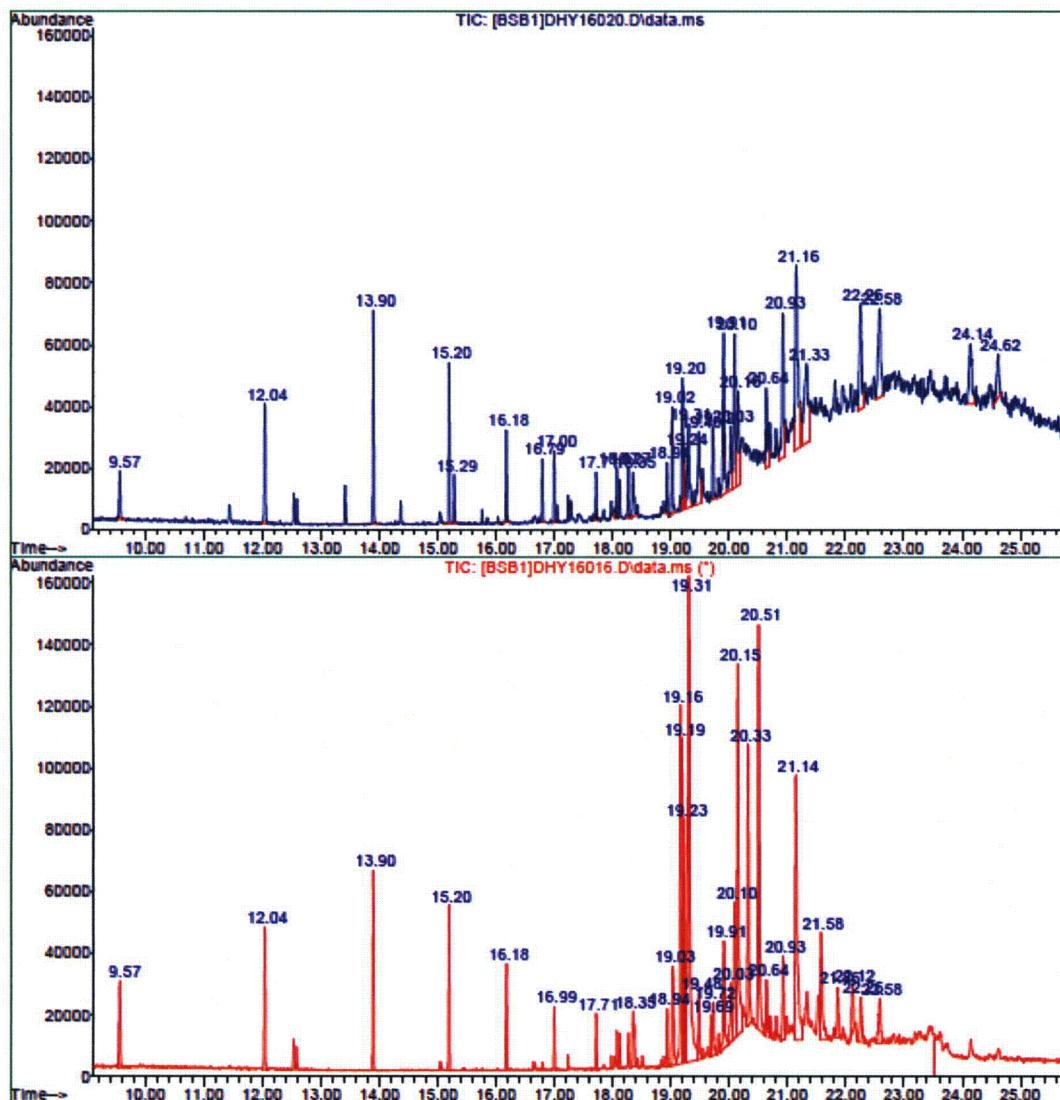


Figure 10

ATTACHMENT 5

Ion Chromatography Cations and Anions



ION CHROMATOGRAPHY ANALYSIS REPORT

JOB NUMBER C0DHY160
PO NUMBER 4500000443

for

Keith Waldrop
Electric Power Research Institute

Prepared by:

A handwritten signature in black ink that appears to read "Xiaohua Cai".

Xiaohua Cai
Senior Scientist GC-MS and FTIR Services
(Tel. 408 530 3616; xcai@eaglabs.com)

Reviewed by:

A handwritten signature in black ink that appears to read "Thomas F. Fister".

Thomas F. Fister, Ph.D.
Sr. Director of Analytical Services
(Tel. 408-530-3660; tfister@eaglabs.com)

ION CHROMATOGRAPHY ANALYSIS REPORT

Requester:
Job Number:
Analysis Date:

Keith Waldrop
C0DHY160
21 Mar 2013

Purpose:

The purpose of the analysis was to extract six samples with deionized water to determine the concentrations of anions and cations of the extracts by Ion Chromatography.

Summary:

Concentration of ions (ppb in 50 ml DI water solution)

<u>Anions</u>	Filter Control	Filter Used	Scotch-Brite Control	Scotch-Brite Used	SaltSmart Control	SaltSmart Used
Fluoride F ⁻	ND	ND	ND	ND	ND	8.99
Chloride Cl ⁻	1.7	5.7	4.7	12.7	2.7	29.7
Nitrite NO ₂ ⁻	ND	ND	ND	1.41	ND	ND
Bromide Br ⁻	ND	ND	ND	ND	ND	ND
Nitrate NO ₃ ⁻	2.06	24.06	11.06	50.06	ND	ND
Sulfate SO ₄ ²⁻	ND	85.79	8.79	129.79	ND	429.79
Phosphate PO ₄ ³⁻	17	17	ND	5	ND	95
<u>Cations</u>	0	0	0	0	0	0
Lithium Li ⁺	ND	2	90	82	ND	ND
Sodium Na ⁺	3.5	13.5	38.5	62.5	2.5	148.5
Ammonium NH ₄ ⁺	ND	9.9	1.9	5.9	ND	5.9
Potassium K ⁺	55.38	51.38	27.38	57.38	ND	91.38
Magnesium Mg ²⁺	ND	11.82	ND	8.82	ND	77.82
Calcium Ca ²⁺	5.29	92.29	14.29	74.29	6.29	539.29

ND not detected.

Experiment

DI water extraction

Each control sample and used sample with similar sizes was soaked in 50 ml of deionized water and sonicated for 20 minutes. The solid sample materials were then removed from the water extracts. The water extracts were analyzed by ion chromatography. A de-ionized water blank was prepared for IC analysis at the same time.

IC analysis

The samples were analyzed as received on a Dionex DX300 Ion Chromatography System equipped with a gradient pump and conductivity detector. The sample was analyzed using a CS12A column set with methanesulfonic acid isocratic elution. The IC was calibrated with calibration standards made from high purity salts containing target cations. Quality control standards, prepared from NIST or NIST traceable cation standards, were run to verify the instrument calibration before and during sample analysis. The accuracy of each ion in the QC standard was within \pm 10% of the nominal concentration after blank subtraction, or the data were QC-recovery corrected or repeated. Method detection limits were 0.002 ppm for all cations

If questions arise as you review the results of this analysis, the report author or any other member of our technical staff will be available for consultation.

After reviewing this report, you may assess our services using an electronic service evaluation form. This can be done by clicking on the link below, or by pasting it into your internet browser. Your comments and suggestions allow us to determine how to better serve you in the future.
<http://www.eaglabs.com/main-survey.html?job=C0DHY160IC>

This analysis report should not be reproduced except in full, without the written approval of EAG.

ATTACHMENT (3)

REGULATORY COMMITMENT

Calvert Cliffs Nuclear Power Plant, LLC
April 24, 2013

ATTACHMENT (3)
REGULATORY COMMITMENT

The table below lists the action committed to in this submittal. Any other statements in this submittal are provided for information purposes and are not considered to be regulatory commitments.

Regulatory Commitment	Date
Submit the response to RAIs E-1 and E-2 contained in Reference (b) listed on the cover letter.	June 14, 2013