CASE STUDY
Unknown Extruder Build-up

PROBLEM
An extruder used for processing polyolefin experienced a build-up of an unknown material.

ANALYTICAL STRATEGY
Due to the critical nature of the application, every effort was made to be certain the techniques involved would identify the issue. For this reason, a variety of techniques were used including, LCMS, PYMS, Headspace GCMS, TGA and PIXE. The mass spectrometry techniques were used to identify the organic composition of the sample, while TGA and PIXE were used to determine the inorganic nature of the sample.

CONCLUSIONS
The major components observed included a series of fatty acids and fatty amide slip agents. Additional components such as olefinic oligomers, a series of diacids, and three polymer antioxidants were detected. The most abundant fatty amides and acids included stearamide, stearic acid and erucylamide. Based upon this analysis, the build-up is most likely due to an excessive application of the mold release agent or lubricant.

Read the following report to see the full analysis.

Final Report

Jordi Labs LLC
Extruder Unknown Identification
Case Study

Date: 11/01/10

Prepared by:
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President
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Report Number: J5169

Jordi Labs LLC Confidential

November 1, 2010

Client Name
Company Name
Address

Dear Valued Client:

Please find enclosed the test results for your samples described as:

1. *Unknown Extruder Build-up*

The following tests were performed:

1. Optical Microscopy (OM)
2. Fourier Transform Infrared Spectroscopy (FTIR)
3. Pyrolysis Mass Spectrometry (PYMS)
4. Quadrupole Time of Flight Liquid Chromatography Mass Spectroscopy (QTOF-LCMS)
5. Headspace Gas Chromatography Mass Spectroscopy (HS-GCMS)
6. Thermogravimetric Analysis (TGA)
7. Proton Induced X-ray Emission (PIXE)

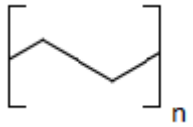
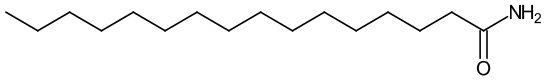
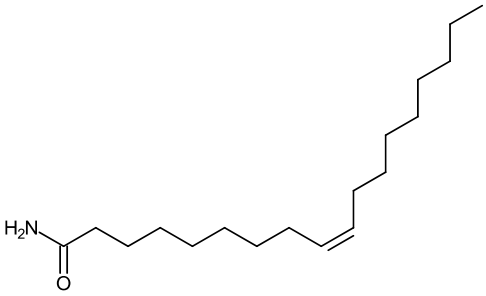
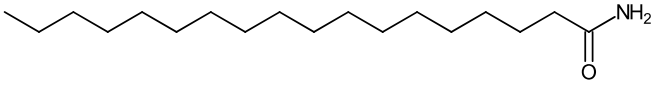
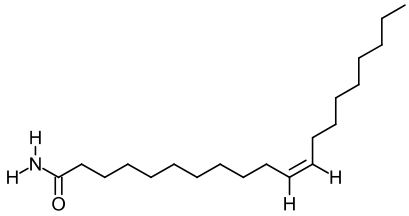
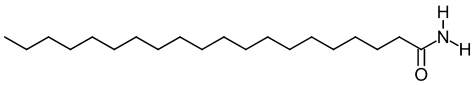
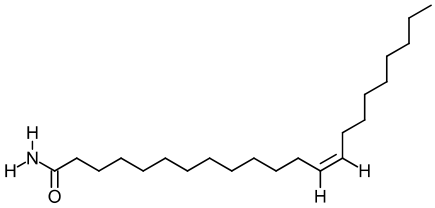
Objectives

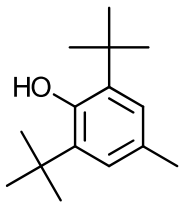
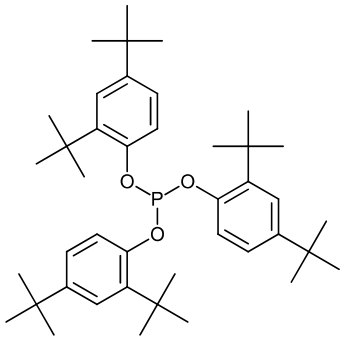
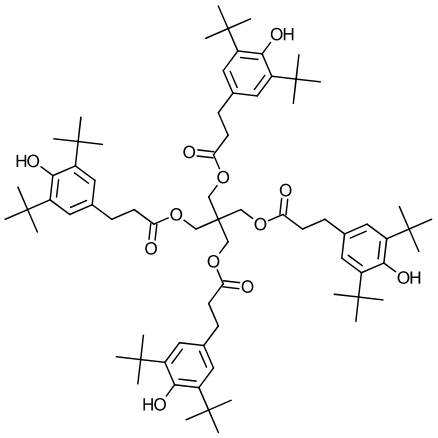
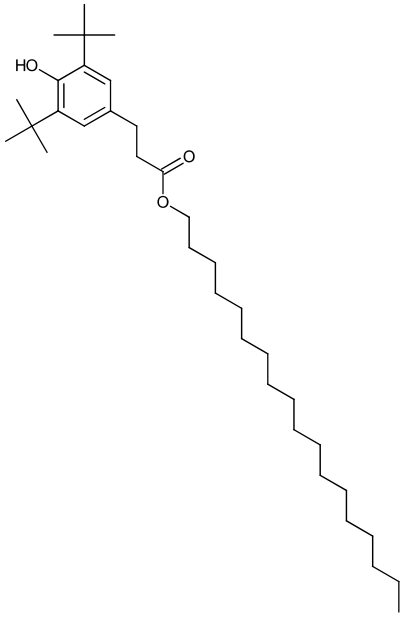
The purpose of this work was to characterize and identify the chemical composition of an unknown sample reported to be a buildup on an extruder used for polyolefin processing.

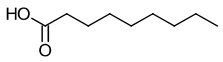
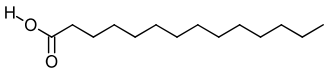
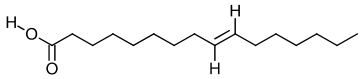
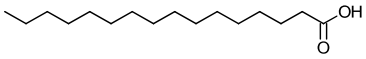
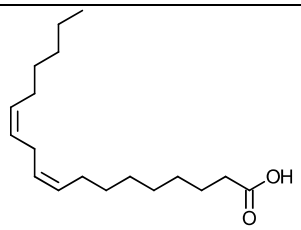
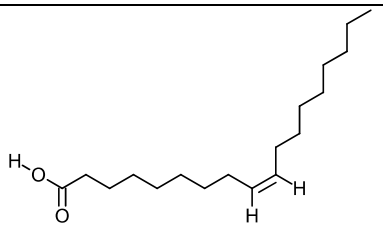
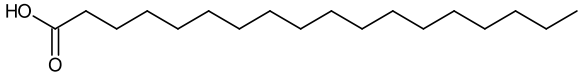
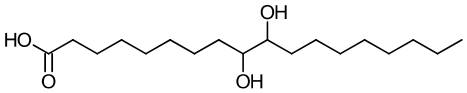
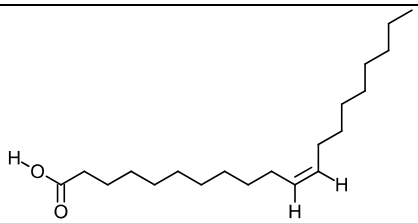
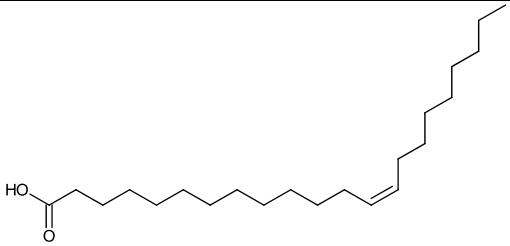
Summary of Results

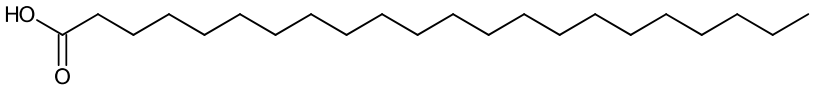
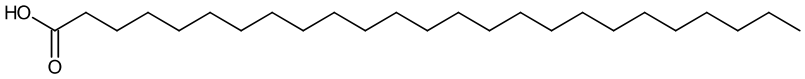
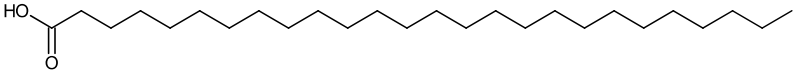
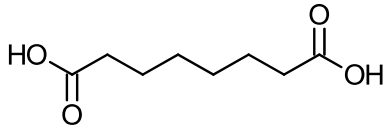
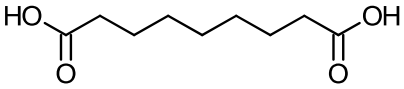
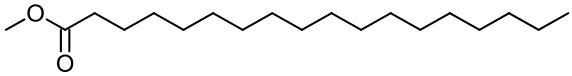
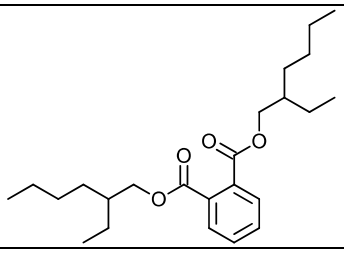
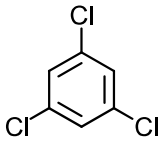
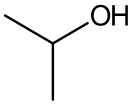
The major components observed included a series of fatty acids and fatty amide slip agents. Additional components such as olefinic oligomers, a series of diacids, and three polymer antioxidants were detected. The most abundant fatty amides and acids included stearamide, stearic acid and erucylamide. Based upon this analysis, the build-up is most likely due to an excessive application of the mold release agent or lubricant. **Table 1** lists the best matching identifications for each observed component.

Table 1: Components Constituting the Unknown Sample

Component	Formula	Structure
Olefinic Oligomers	varies*	
Palmitamide	C16 H33 N O	
Oleamide	C18 H35 N O	
Stearamide	C18 H37 N O	
Eicosenamide	C20 H39 N O	
Eicosanamide	C20 H41 N O	
Erucylamide	C22 H43 N O	

BHT	C15 H24 O	
Irgafos 168	C42 H63 O3 P	
Irganox 1010	C73 H108 O12	
Irganox 1076	C35 H62 O3	

Nonanoic Acid	C9 H18 O2	
Tetradecanoic Acid	C14 H26 O2	
Hexadecenoic Acid	C16 H33 N O	
Palmitic Acid	C16 H32 O2	
Linoleic Acid	C18 H32 O2	
Oleic Acid	C18 H34 O2	
Stearic Acid	C18 H36 O2	
Dihydroxyoctadecanoic Acid	C18 H36 O4	
Eicosenoic Acid	C20 H38 O2	
Erucic Acid	C22 H42 O2	

Docosanoic Acid	C22 H44 O2	
Pentacosanoic Acid	C25 H50 O2	
Hexacosanoic Acid	C26 H52 O2	
Suberic Acid	C8 H14 O4	
Azelaic Acid	C9 H16 O4	
Methyl Stearate	C19 H38 O2	
Bis(2-Ethylhexyl phthalate)	C24 H38 O4	
Trichlorobenzene	C6 H3 Cl3	
Tetrahydrofuran	C4 H8 O	
Isopropanol	C3 H8 O	

*A number of trace level components were identified in the samples which have not been included in the above list for brevity.

Individual Test Results

A summary of the individual test results is provided below. All accompanying data, including spectra, has been mailed in the data section of this report.

Optical Microscopy – Optical microscopy was performed to document the nature of the unknown sample as-received. A small portion (approximately 10mg) was analyzed with an Olympus UC30 camera attached to an Olympus SZ61 microscope. The image can be seen in **Figure 1** below. The material had a white, slightly translucent waxy nature.

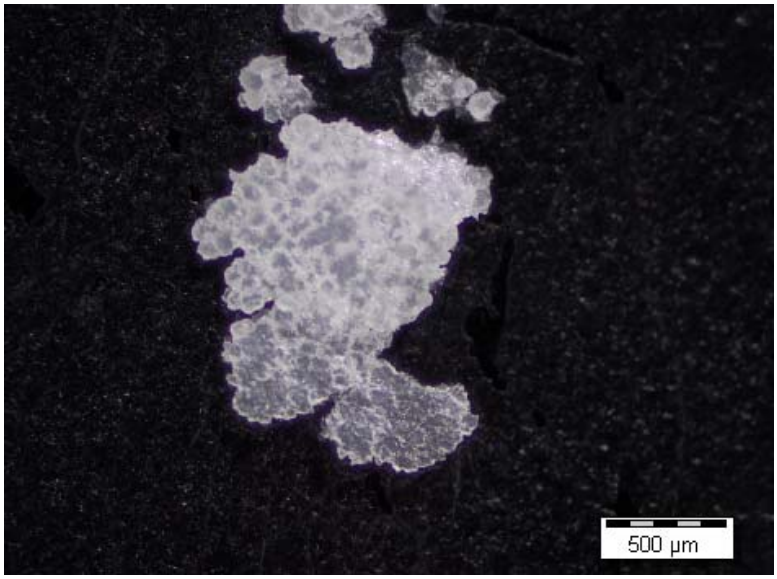


Figure 1: Optical microscopic image of the unknown sample as-received

FTIR – An FTIR spectrum of the sample was found to be most consistent with a fatty amide. Comparison of the sample spectra with a reference library of over 20,000 spectra produced a best match to Kemamide S, a commercial stearamide polymer additive. The match quality was 720 of 1000.

Figure 2 shows an overlay of the unknown and Kemamide S spectra. Peaks associated with amide groups and hydrocarbon chains can be observed. Functional group assignments for each major peak can be found in **Table 2**.

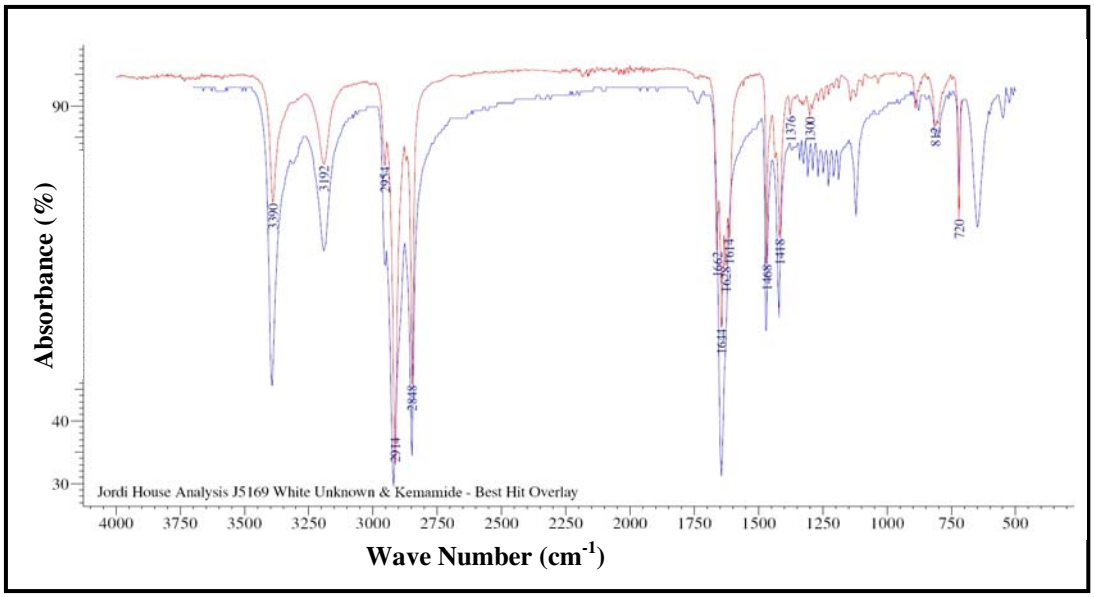


Figure 2: Overlay of FTIR spectrum for the sample (red) as well as the best database match, Kemamide S (blue)

Table 2: FTIR peak identification

IR Frequency	Functional Group
3390/3192 cm^{-1}	NH_2 stretch
2914/2848 cm^{-1}	CH stretch
1644 cm^{-1}	C=O stretch
1468 cm^{-1}	CH_2 bend
1418 cm^{-1}	Amide mixed mode vibration
720 cm^{-1}	Long-chain CH_2 wag

FTIR is a nondestructive technique that requires very little sample (< 1mg). It is important to note that this more basic technique allowed for identification of one of the major components of the sample but provided little indication that the sample is in fact a very complex mixture of components.

PYMS – Analysis by PYMS is conducted using a double shot technique. The 1st pass analysis consists of heating the sample to desorb volatile components followed by GCMS analysis. The remaining portion of the sample is then heated to pyrolysis temperatures and components are cryotrapped and analyzed by GCMS. Sample peaks were compared with over 197,000 reference compounds using the NIST/EPA/NIH mass spectral search program.

The three most prominent peaks found by PYMS had a best match of Stearamide (The component identified by FTIR), erucylamide, and hexadecane (olefin). Butylated hydroxytoluene (BHT) and Irganox 1076 (a BHT-containing acid ester) were also identified in the sample. Hindered phenolic compounds are used as antioxidants in many polymer systems.

A phosphorus-containing compound was observed at 16.7 minutes with a molecular ion at 646 amu. A comparison of the PYMS and LCMS data allows for positive identification of this compound as the antioxidant Irgafos 168.

We did not observe indications of any significant polymeric materials in the sample.

Table 3 lists all of the compounds observed by PYMS and their retention times and match scores. Components are listed in order of peak intensity, beginning with the strongest observed peak.

Table 3: Components Observed by PYMS

Best Matching Component	Retention Time	Match Score
Stearamide*	13.470	747
Erucylamide	14.789	802
Hexadecane	10.125	948
Methyl stearate	12.453	880
Nonadecanamide	14.171	857
Butylated hydroxytoluene (BHT)	9.529	922
Nonadecane	16.123	900
Irganox 1076	17.253	757
Heptadecane, 2,6,10,14-tetramethyl-	11.993	864
Irganox 168*	16.781	651
Heptacosane	18.519	530
Decane, 6-ethyl-2-methyl-	11.112	832

* An alternate match was reported based on manually compared mass spectra
Peaks are listed from highest to lowest intensity.
Retention times are in minutes.

Each peak spectrum and their best matching database entry can be seen in the data section at the end of the report.

HS-GCMS – Approximately 10mg of the sample was hermetically sealed into a headspace sampling vial. The sample is then gently heated (70°C), increasing the concentration of volatile components in the gasses trapped above the sample. A precise aliquot of the headspace gasses are then transferred to the GCMS and analyzed.

The major components detected by HS-GCMS were three solvents isopropanol, tetrahydrofuran (THF), and trichlorobenzene. In headspace analysis, compounds which are more volatile are preferentially evolved due to the lower temperature and thus are easier to identify. These components were not observed during the PYMS analysis. Also detected by HS-GCMS were a few of the shorter chain aliphatic compounds, as well as dichlorobenzene. A list of observed components can be found in **Table 4**.

Table 4: Components Observed by HS-GCMS

Best Matching Component	Retention Time	Match Score
Isopropanol	1.455	857
Tetrahydrofuran	2.026	933
Methyl-nonane	6.673, 6.810	895
Decane	7.187	952
Dichlorobenzene	7.598	897
Trichlorobenzene	9.025	954

QTOF-LCMS –An extract of the sample (~4 mg) was dissolved and filtered before being analyzed by QTOF-LCMS. Both positive and negative *electrospray ionization (ESI)* modes were used in conjunction with two different column types, an Agilent Poroshell 120 and an Agilent C18 Rapid Resolution (RR). Two columns were used to facilitate the broad range of component polarity observed in the sample.

Molecular Feature Extraction (MFE) was performed on the resulting data to find the mass of the unknowns in the sample. Identifications were obtained using *Molecular Formula Generation (MFG) and* a Jordi proprietary database. Each mass as well as the most probable formula can been seen in **Table 5**. Where a match score is provided, the formula was found using MFG.

This method provided a much greater level of detail about the fatty amides as compared with PYMS. Additionally, many different fatty acids, diacids, and several antioxidants were observed as shown in **Table 5**.

QTOF-LCMS has a significant advantage over other mass spectrometric methods due to its high mass accuracy. This technique provides the mass to four decimal places allowing for very reliable identification. The limitation of this method is that the components must be ionizable.

Table 5: Unique sample mass features and their best matching formula as determined by QTOF-LCMS

Best Match	RT	Mass	Formula (DB)	Score (DB)	Formula (MFG)	Score (MFG)	Height	Mode
suberic acid	2.039	174.089	C8 H14 O4	86.63	C8 H14 O4	86.74	18128	ESI -
azelaic acid	2.467	188.1043	C9 H16 O4	85.13	C9 H16 O4	85.35	14779	ESI -
nonanoic acid	16.775	158.1304	C9 H18 O2	87.05	C9 H18 O2	87.11	13140	ESI -
dihydroxy octadecanoic acid	21.508	316.2606	C18 H36 O4	82.73	C18 H36 O4	84.42	13737	ESI -
tetradecenoic acid	26.491	226.1927	C14 H26 O2	85.8	C14 H26 O2	85.81	3873	ESI -
palmitamide	28.373	255.256	C16 H33 N O	99.56			415556	ESI +
oleamide	29.168	281.2716	C18 H35 N O	99.23			737389	ESI +
hexadecenoic acid	29.181	254.2241	C16 H30 O2	85.63	C16 H30 O2	85.87	44165	ESI -
linoleic acid	29.673	280.2394	C18 H32 O2	83.79	C18 H32 O2	82.83	15218	ESI -
palmitic acid	30.553	256.2407	C16 H32 O2	95.74	C16 H32 O2	97.99	859930	ESI -
Stearamide	30.682	283.2872	C18 H37 N O	99.3			1439191	ESI +
eicosenamide	31.12	309.3021	C20 H39 N O	92.31			132920	ESI +
oleic acid	31.133	282.2555	C18 H34 O2	97.84	C18 H34 O2	85.62	69587	ESI -
bis(2-Ethylhexyl) phthalate	32.185	390.2766	C24 H38 O4	97.21	C24 H38 O4	81.28	702745	ESI +
Stearic acid	32.346	284.2723	C18 H36 O2	95.32	C18 H36 O2	96.03	616482	ESI -
eicosanamide	32.392	311.3179	C20 H41 N O	94.02			97832	ESI +
erucylamide	32.626	337.3341	C22 H43 N O	98.89			773870	ESI +
eicosenoic acid	32.666	310.2861	C20 H38 O2	92.19	C20 H38 O2	69.04	3547	ESI -
methyl stearate	33.056	298.2859	C19 H38 O2	88.62	C19 H38 O2	79.26	4149	ESI -
	33.162	312.1757			C16 H28 O4 Si	76.32	130751	ESI -
erucic Acid	33.861	338.3184	C22 H42 O2	99.97	C22 H42 O2	97.58	413449	ESI -
docosanoic acid	34.855	340.3331	C22 H44 O2	93.59	C22 H44 O2	96.42	36119	ESI -
irganox 1010	35.054	1176.7806	C73 H108 O12	84.74			1031	ESI -
	35.193	326.1916			C17 H30 O4 Si	92.95	287065	ESI -
pentacosanoic acid	36.649	382.3798	C25 H50 O2	90.98	C25 H50 O2	79.2	14563	ESI -
	37.212	340.2069			C18 H32 O4 Si	76.28	93099	ESI -
hexacosanoic acid	37.427	396.3959	C26 H52 O2	96.32	C26 H52 O2	95.67	15933	ESI -
Irganox 1076	37.493	530.469	C35 H62 O3	96.9	C32 H67 Cl O Si	49.28	11919	ESI -/+
Oxidized Irgafos 168	37.76	662.4452	C42 H63 O4 P	96.01	C47 H59 Na O	67.57	57065	ESI +

DB = Database
MFG = Molecular Formula Generation

In the unknown sample, a component of mass 284.2948 m/z had the largest peak area. As an example of the power of this method, we closely examined the exact mass, isotope distribution and MS/MS data for this component. **Figure 3** shows the observed mass spectral pattern for the unknown. Three peaks were determined to be related to this one compound. These masses were identified as the M+H (compound with a hydrogen ion), as well as two dimer peaks (2M+H, 2M+Na).

Based upon these mass features, the calculated exact mass of the unknown was determined to be *283.2873 amu*. Molecular formula generation was performed on this mass and the results are shown in **Table 6**.The best matching elemental composition was $C_{18}H_{37}NO$. This formula has a mass of *283.2875 amu* and is consistent with the compound stearamide.

We further compared the isotope distribution patterns for the unknown and the expected pattern for stearamide as shown in **Figure 3**. The isotope distribution for the formula $C_{18}H_{37}NO$ (purple outlines) matches nearly perfectly with the measured values. Lastly, HPLC retention times and MS/MS data was acquired for the unknown and compared to Jordi's proprietary reference library for confirmation of the chemical structure. The resulting MS/MS data is shown in **Figure 4** and is an excellent match.

QTOF-LCMS is one of the most powerful and definitive methods for compound identification available.

Table 6

Formula (M)	Score	Mass	Calc Mass	Diff (ppm)	Abund Match	Spacing Match	Mass Match
C18 H37 N O	99.13	283.2873	283.2875	0.6	98.13	99.05	99.78

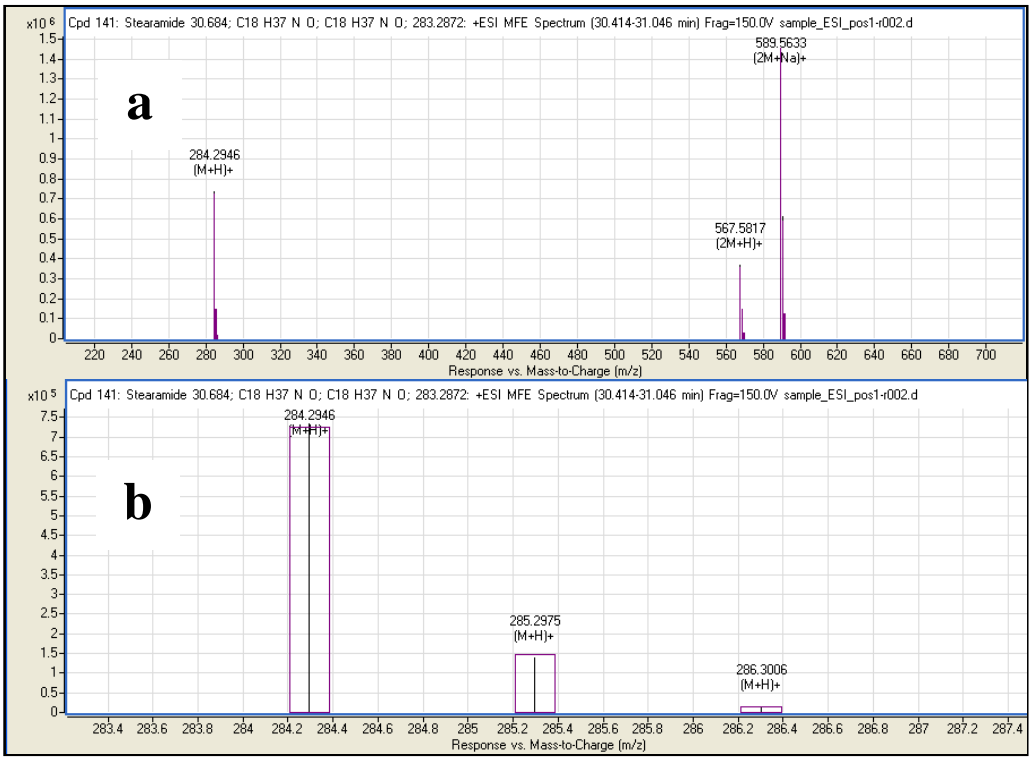


Figure 3: Mass spectrum for unknown (a) and isotope distribution for the unknown compared with a formula of $C_{18}H_{37}NO$ (b)

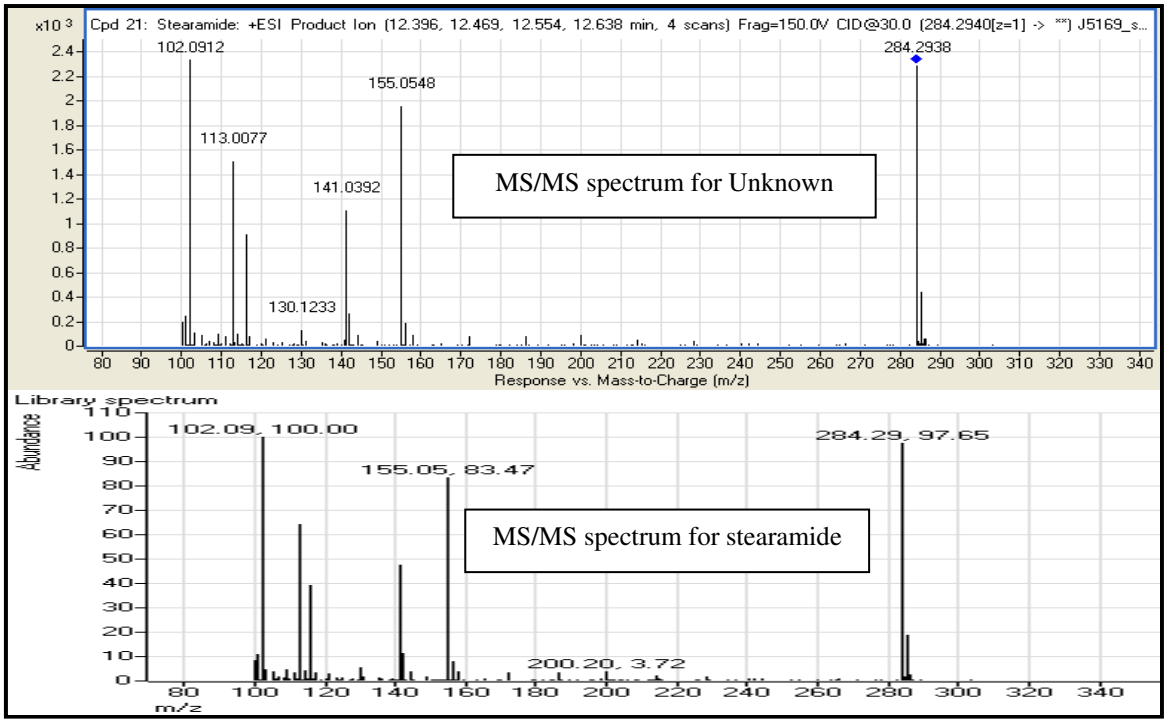


Figure 4: Comparison of MS/MS spectrum for the unknown and stearamide.

PIXE: The sample was analyzed by PIXE to identify any inorganic species present. **Table 7** shows a list of the elements detected and their respective concentrations. Trichlorobenzene and Irgafos 168 were identified by LCMS and HS-GCMS and would explain the higher concentrations of chlorine and phosphorus. All other elements are present at trace levels.

Table 7: Inorganic Elemental concentration as detected by PIXE analysis

PIXE	
Element	**Amount**
Chlorine	0.233%
Phosphorus	123.998 ppm
Calcium	19.460 ppm
Lead	10.890 ppm
Potassium	10.050 ppm
Iron	7.916 ppm
Bromine	6.408 ppm
Zinc	5.487 ppm
Copper	1.165 ppm

TGA – The sample was subjected to TGA analysis from 20 to 1000ºC. The sample was analyzed under nitrogen in duplicate, consuming approximately 20mg of sample. The first sample was run immediately after weighing while the second sample remained in the run queue for approximately 1.5 hours. It was observed that the second sample lost some portion of the volatile material (likely residual solvent) during this time. This indicates that the contaminant is at least partially volatile under ambient conditions.

Analysis results can be seen in **Table 8**. It was noted that the samples burned nearly completely indicating that the buildup has only trace levels of inorganics. It was further noted that the material degraded predominantly under 300°C. This generally indicates that the material is of lower molecular weight. The presence of multiple weight loss steps is consistent with a multi-component chemical system.

Table 8: TGA results for the unknown material

	Run 1	**Run 2**
Total Weight Loss	98.9%	100%
Major Volatization Peaks[1]	40.2 ºC	41.5 ºC
	75.2 ºC	87.3 ºC
	139.7 ºC	131.8 ºC
	259.9 ºC	257.5 ºC
	335.6 ºC	339.8 ºC

1 - Volatization peaks were identified using a first derivative analysis.

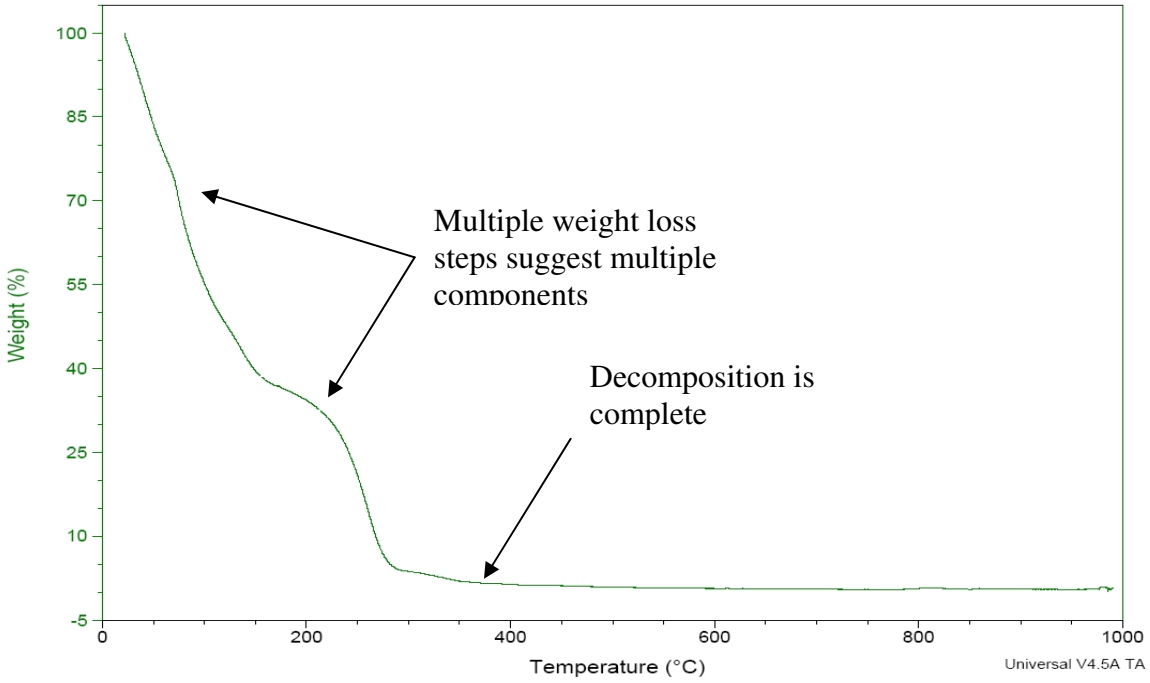


Figure 5: TGA decomposition profile for the Extruder Buildup Sample.

Analysis Conditions

This section of a Jordi report provides information on the methods used including instrument type, temperatures, solvents, sample preparation, etc. The specific conditions have been removed for this case study.

Closing Comments

Deformulation of an unknown material is intended to provide a best estimate of the chemical nature of the sample. All chemical structures are supported by the evidence presented but are subject to revision upon receipt of additional evidence. Additional factors such as material processing conditions may also affect final material properties.

Jordi Labs specializes in polymer testing and has 25 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

Mark Jordi

Mark Jordi, Ph. D.
President
Jordi Labs LLC

Appendix

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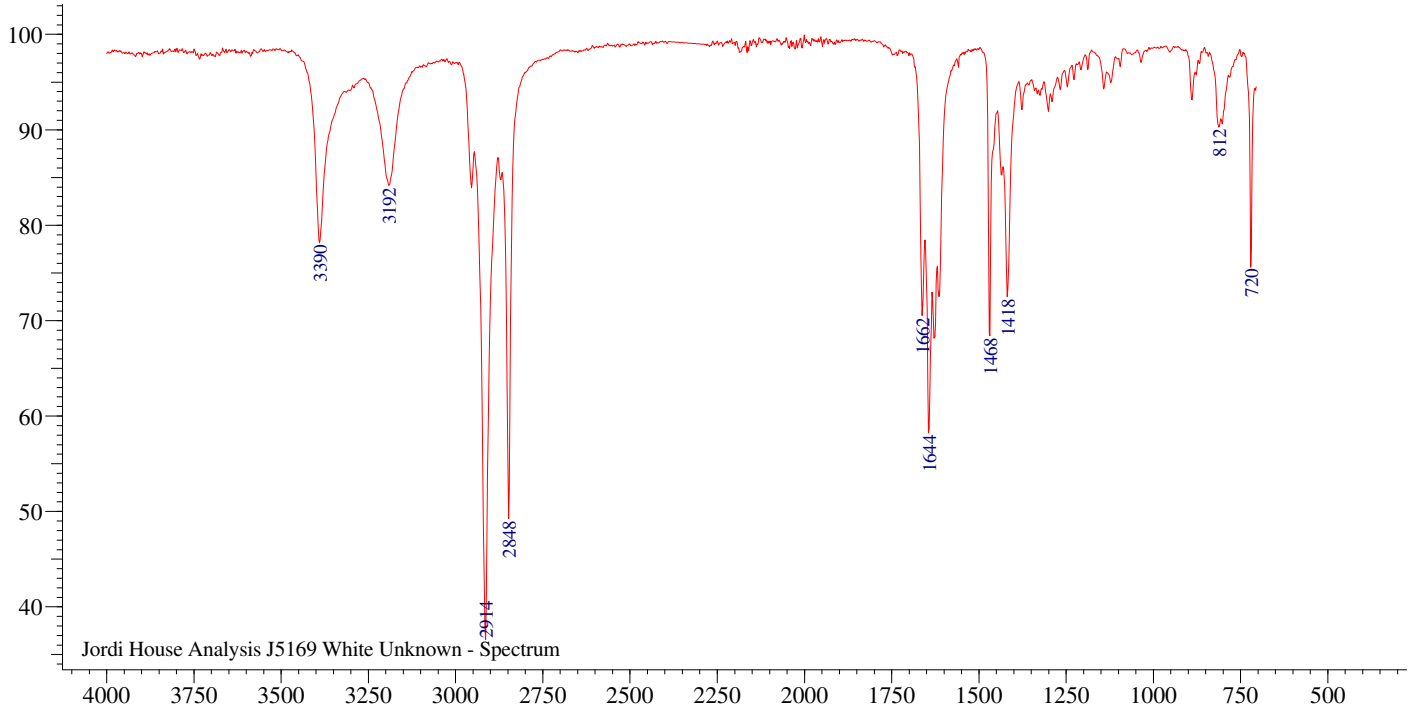
FTIR RESULTS


100
90
80
70
60
50
40
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3192
2914
2848
1662
1644
1468
1418
812
720
Jordi House Analysis J5169 White Unknown - Spectrum
4000
3750
3500
3250
3000
2750
2500
2250
2000
1750
1500
1250
1000
750
500

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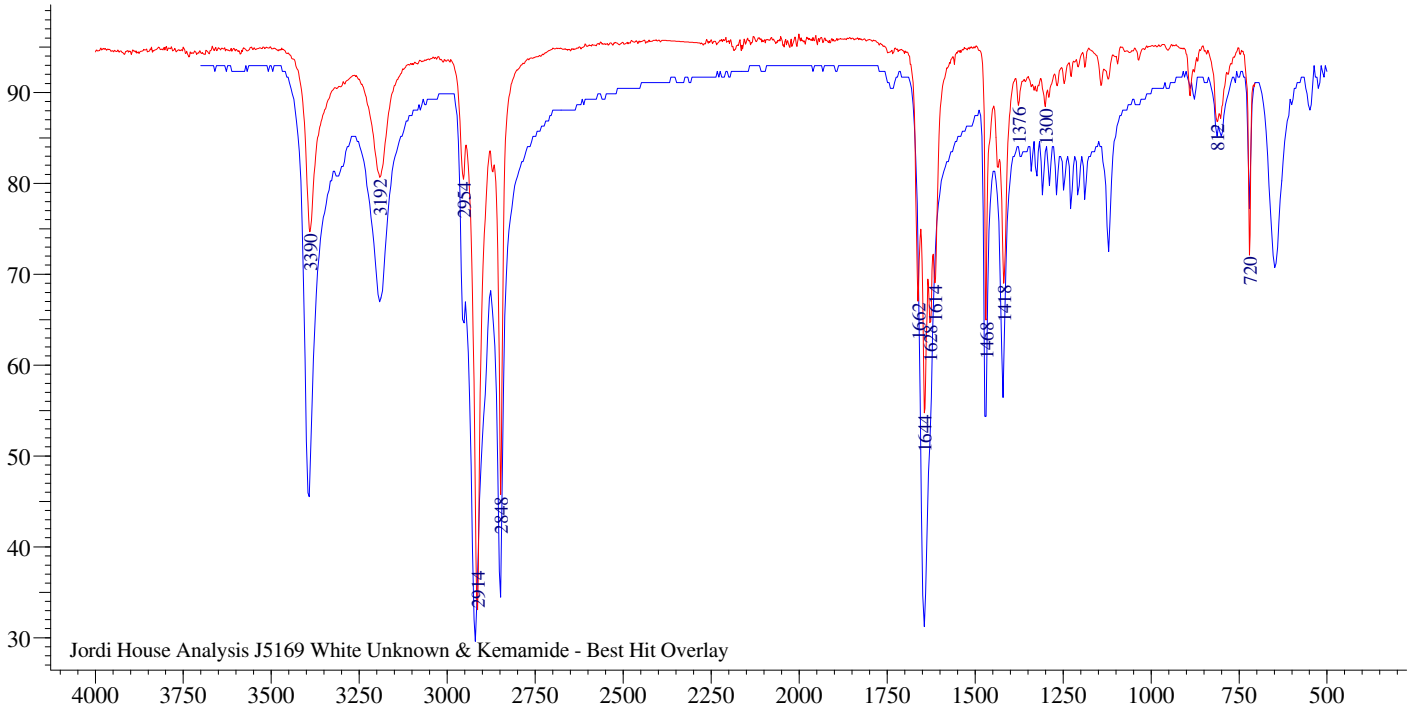

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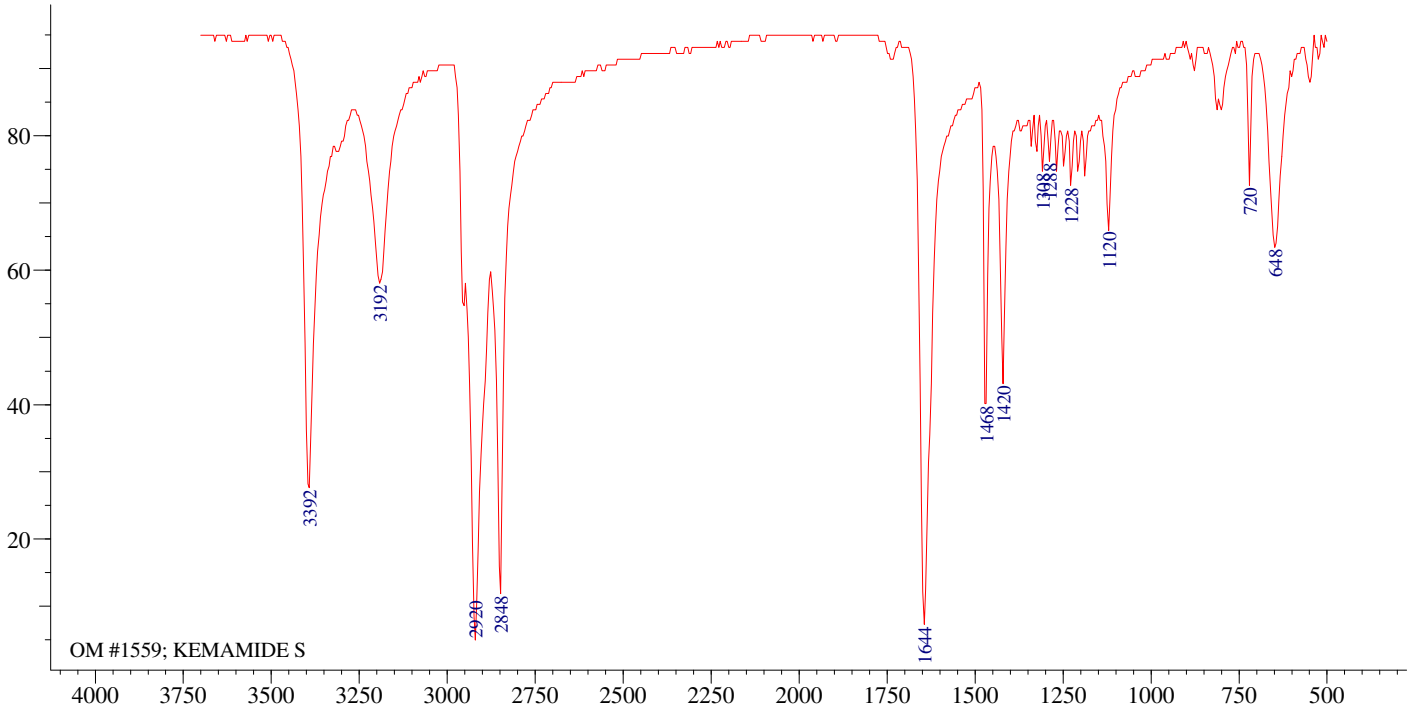
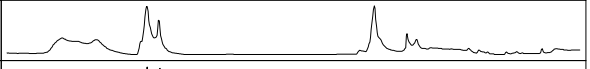


Name(s):	KEMAMIDE S
Source Of Sample:	WITCO CHEMICAL CORPORATION, HUMKO CHEMICAL DIVISION
Technique:	FILM (CAST FROM o-DICHLOROBENZENE)
Density:	(130C) 0.809 G/ML
Flash Point:	(COC) 474.8F
Molecular Weight:	278
Classification:	Polymers: UREAS, AMIDES, CYANURATES
Solution Data:	Acid Number: 4.0 MAX
Values:	Iodine Value: 3.0 MAX
Viscosity Data:	(130C) 5.8 CPS
Molecular Formula:	C.18.H.37.N.1.O.1.
Comments:	Chemical Description: STEARAMIDE

Index	HQI	Chemical Name	Spectrum
OM #1559	719.64	KEMAMIDE S	
CL #570	700.16	AMPHITOL 86B AMPHOTERIC*N-A...	
CL #585	694.78	VELVETEX BST AMPHOTERIC*STE...	
CL #236	672.64	KEMAMIDE 1288*5-HYDROXYSTEA...	
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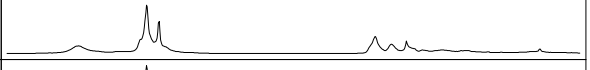
OM #1509	652.48	RHENOFIT NC ACCELERATOR	
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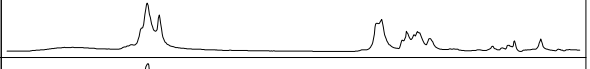
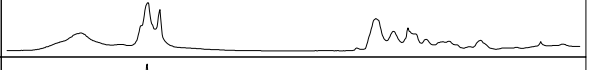
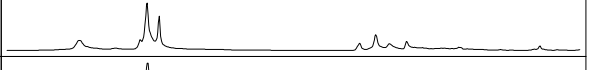
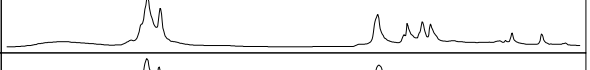
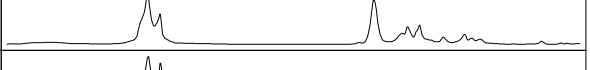
CL #582	640.97	PRODUCT ODN AMPHOTERIC*N-ST...	
CL #481	637.93	ONYXSAN H CATIONIC*ALKYL IM...	
OM #1554	634.56	CARNAPOL 77X	
PL #1320	632.17	ETHYLAN LMP : COCONUT MONOI...	
CL #579	631.22	ANFOTERICO LB AMPHOTERIC*LA...	
HS #6215	630.95	WARCOBASE A	
PL #1440	627.34	CARNAPOL 77X : FATTY DIAMIDE	
CL #587	624.58	CULVERAM TDG AMPHOTERIC BIO...	
PL #637	620.99	LONZAINE 18S : STEARYL BETAINE	
CL #586	620.99	LONZAINE 18S AMPHOTERIC*STE...	
HS #6214	620.46	HALLCOMID M-12	
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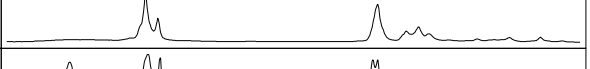
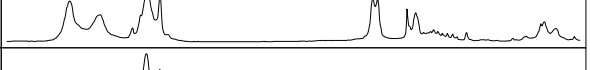
CL #571	617.67	AMPHITOL 24B AMPHOTERIC*N-A...	
PL #1450	614.27	RAM : NITROGENOUS ALIPHATIC...	
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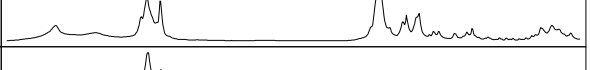
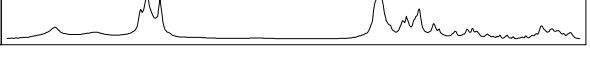
CL #339	612.81	HAMPOSYL M-30*SODIUM MYRIST...	
CL #326	612.11	ADVAWET PB CATIONIC*AMIDO-A...	
CL #340	610.94	HAMPOSYL L-95*SODIUM LAUROY...	

PYMS RESULTS

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Misc Info  : J5169 Jordi In House Unknown
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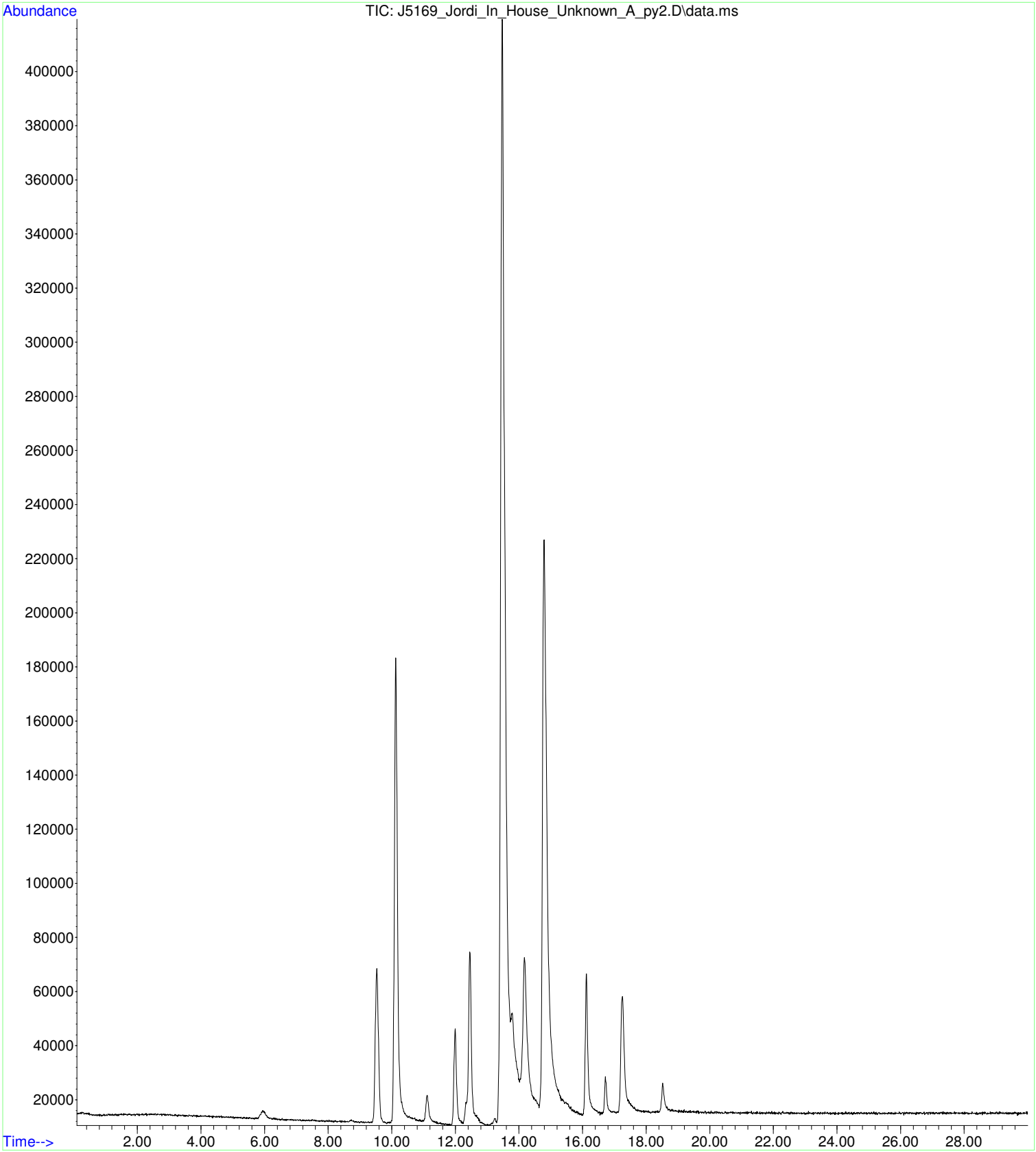

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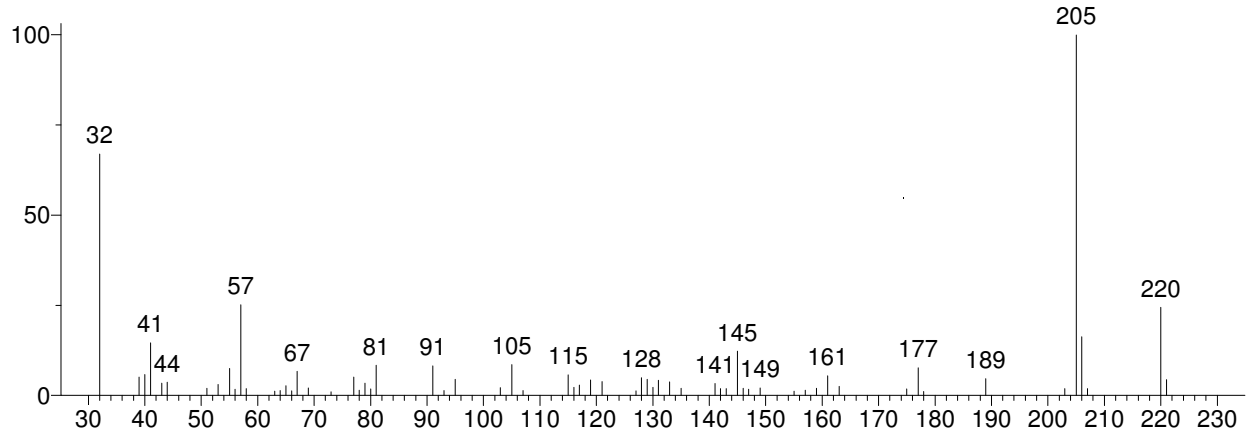

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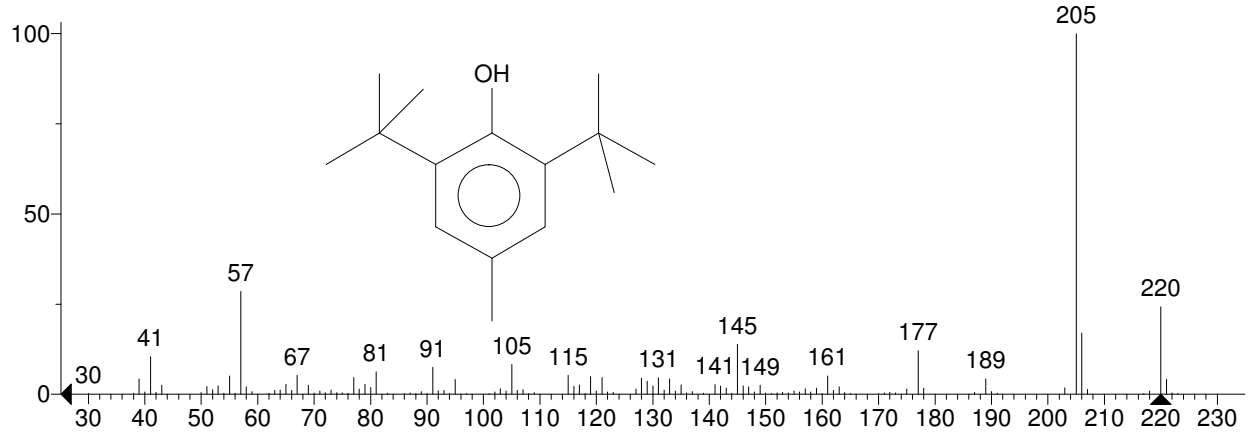

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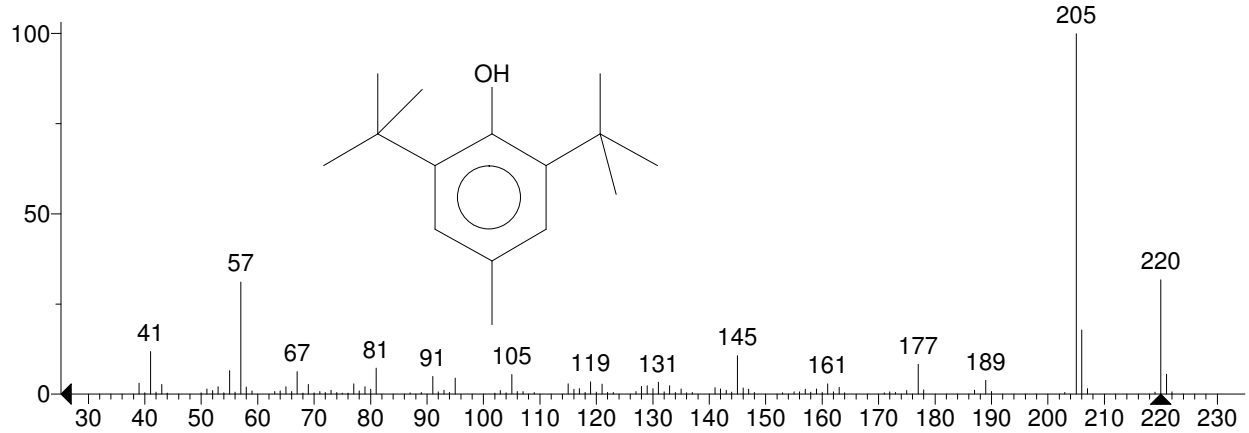

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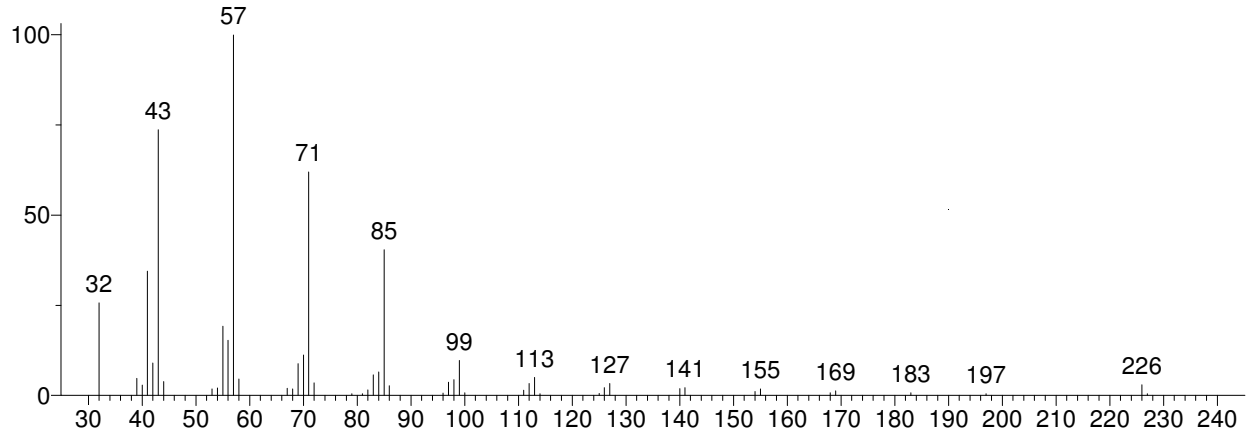

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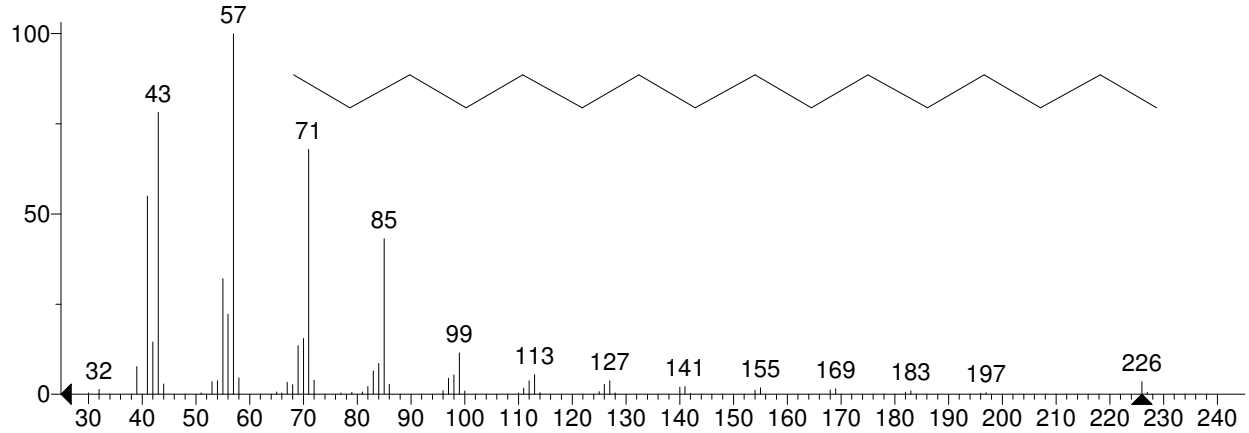

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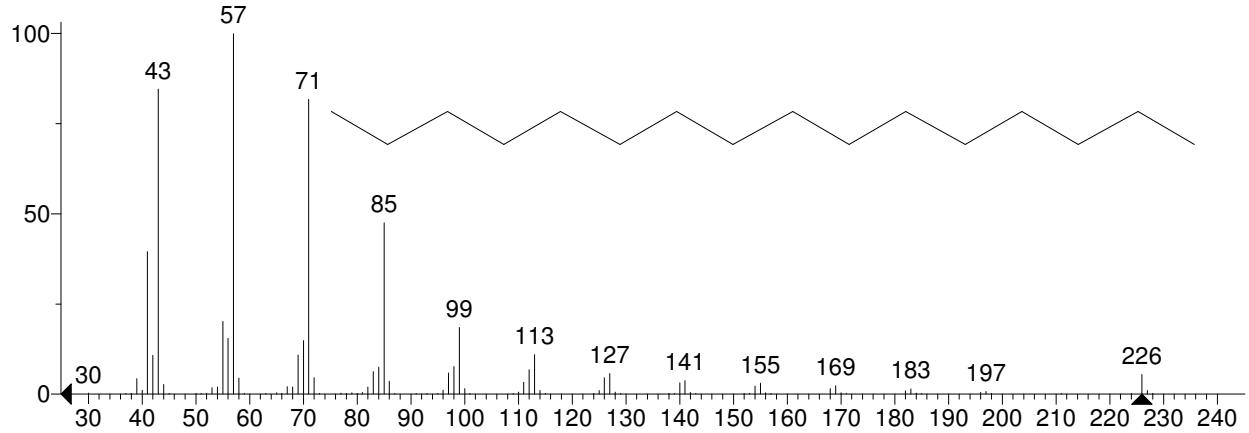

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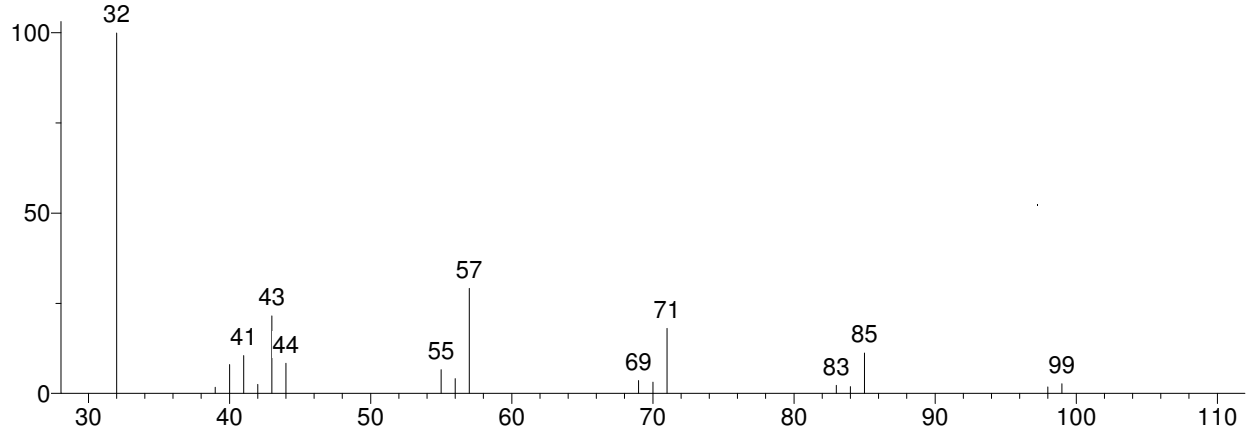

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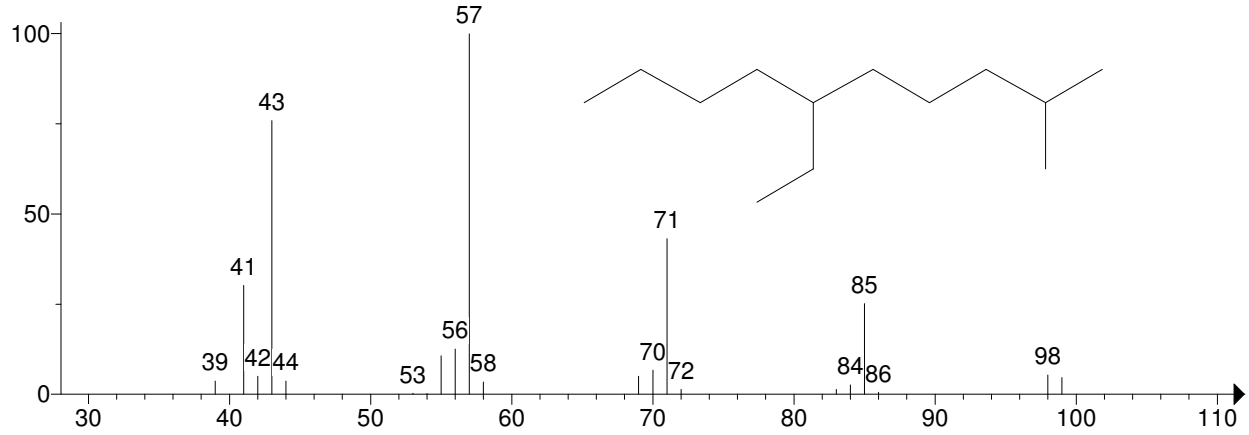

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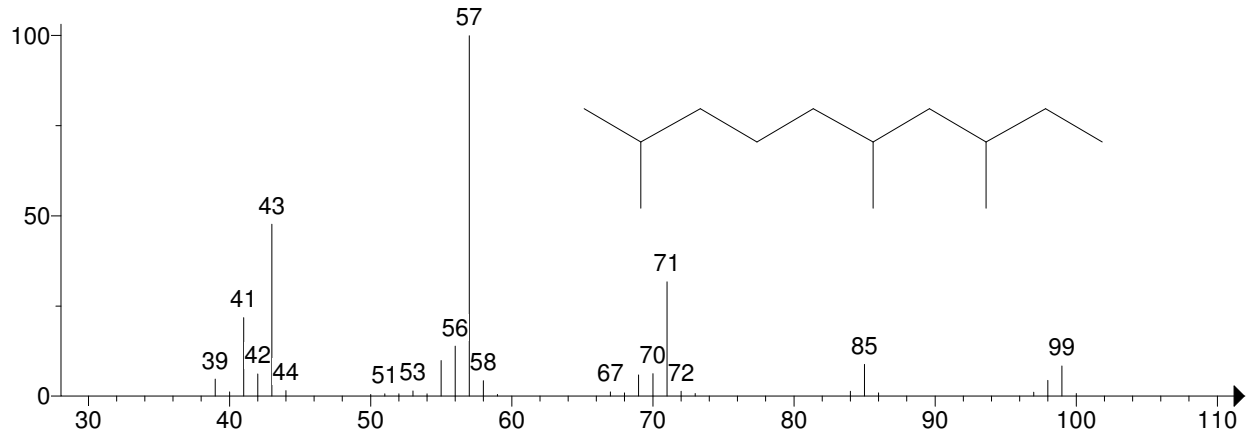

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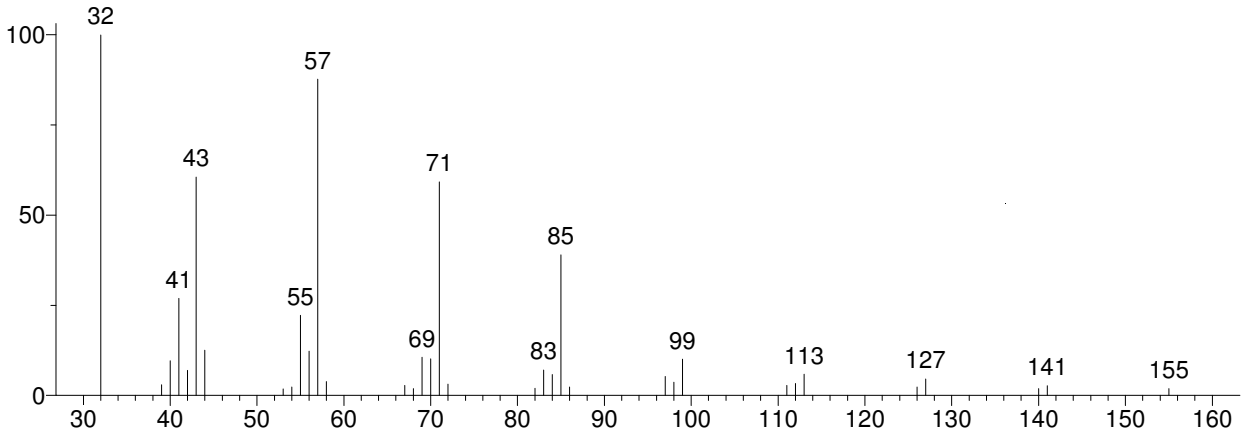

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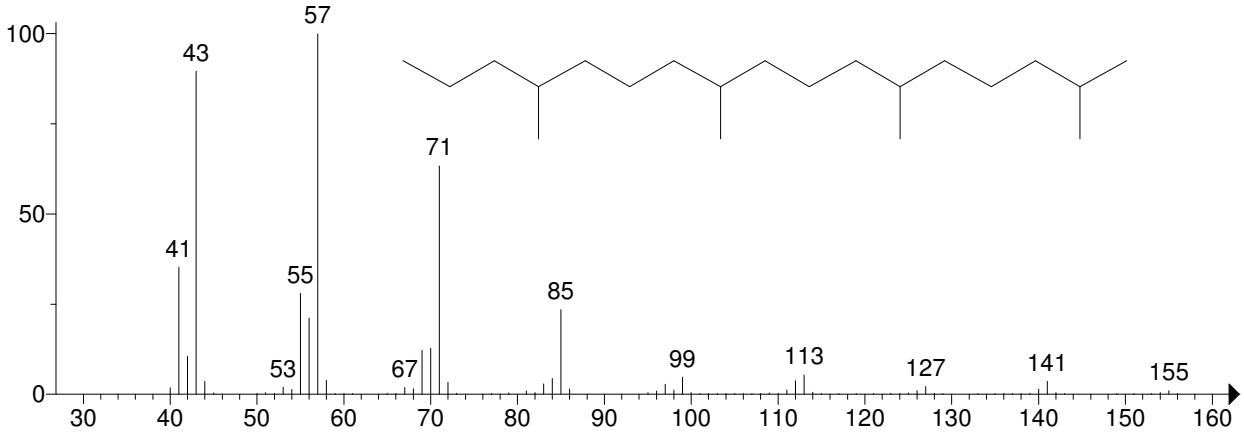

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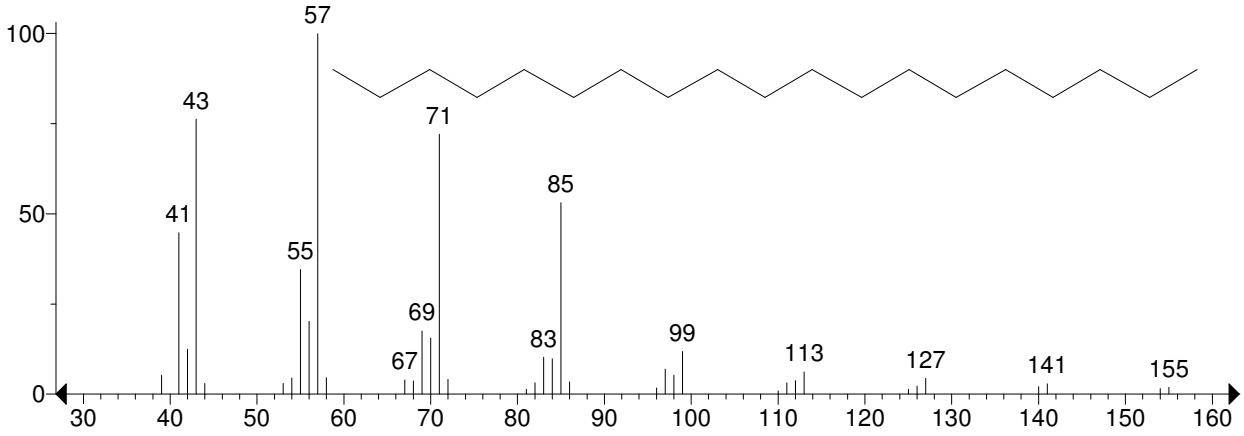

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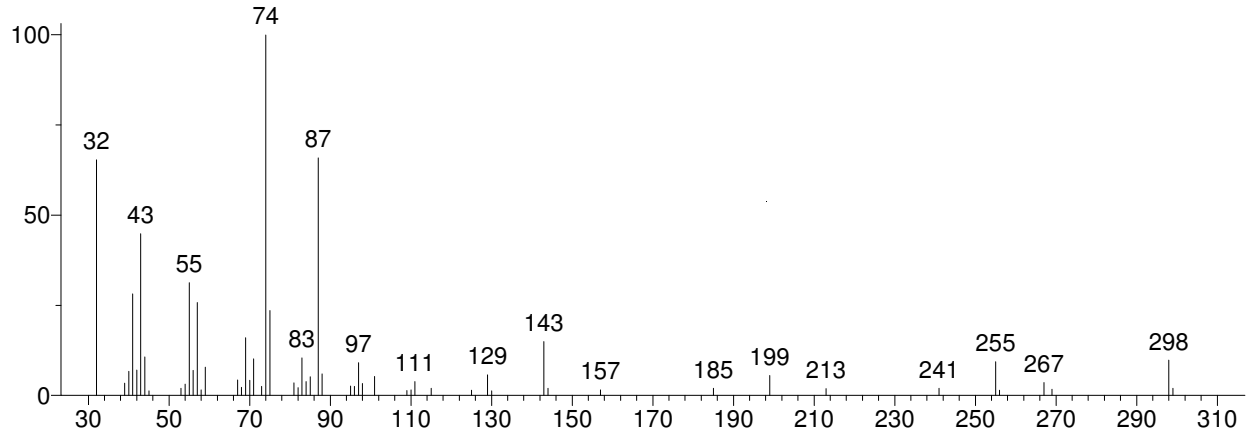

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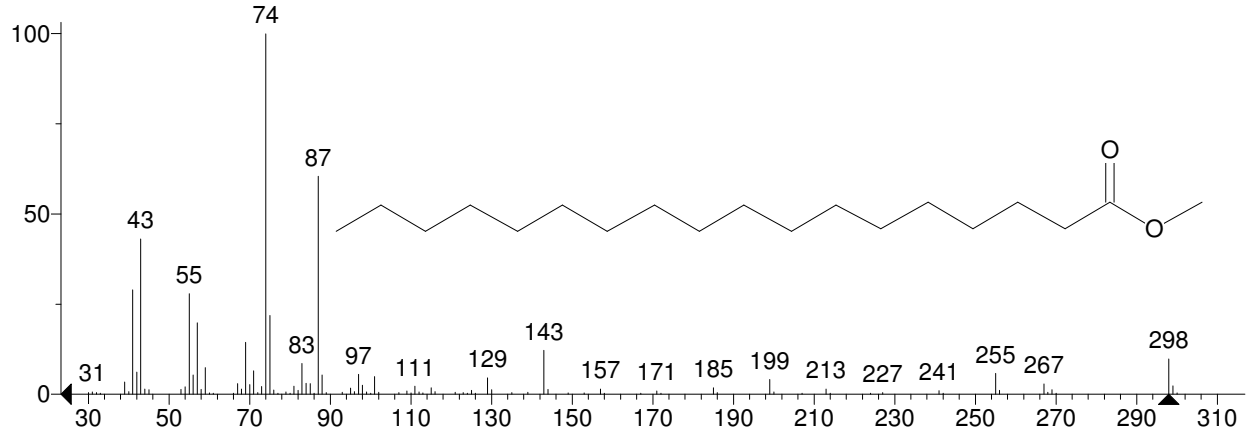

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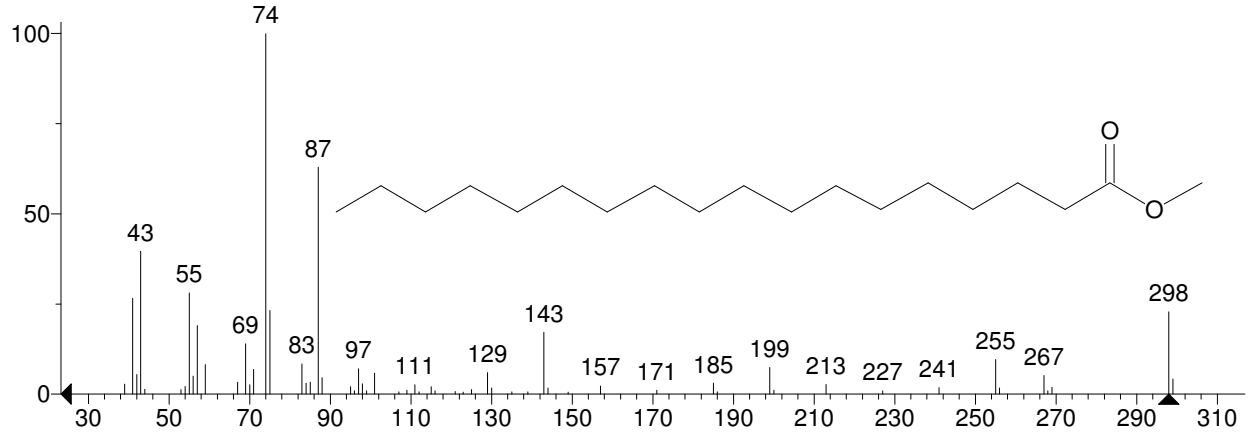

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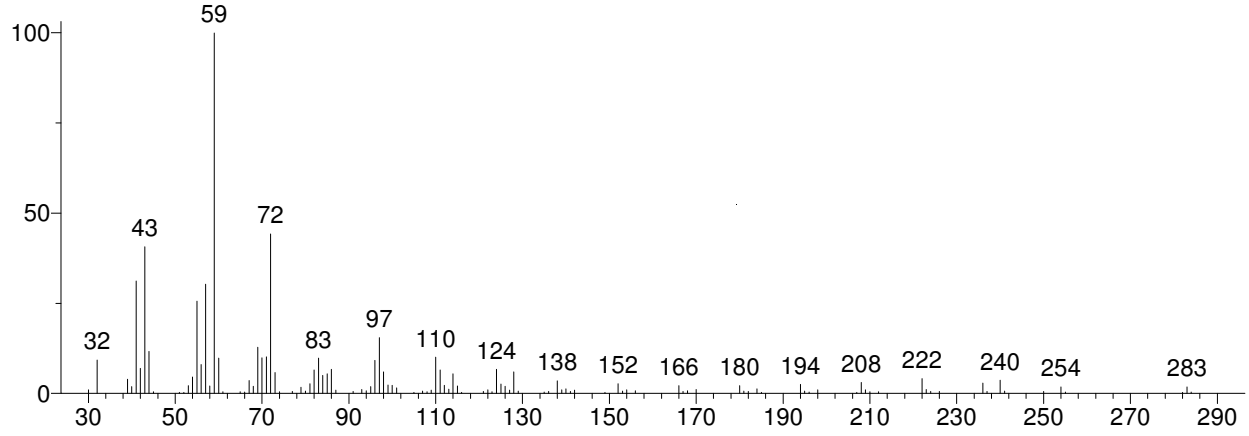

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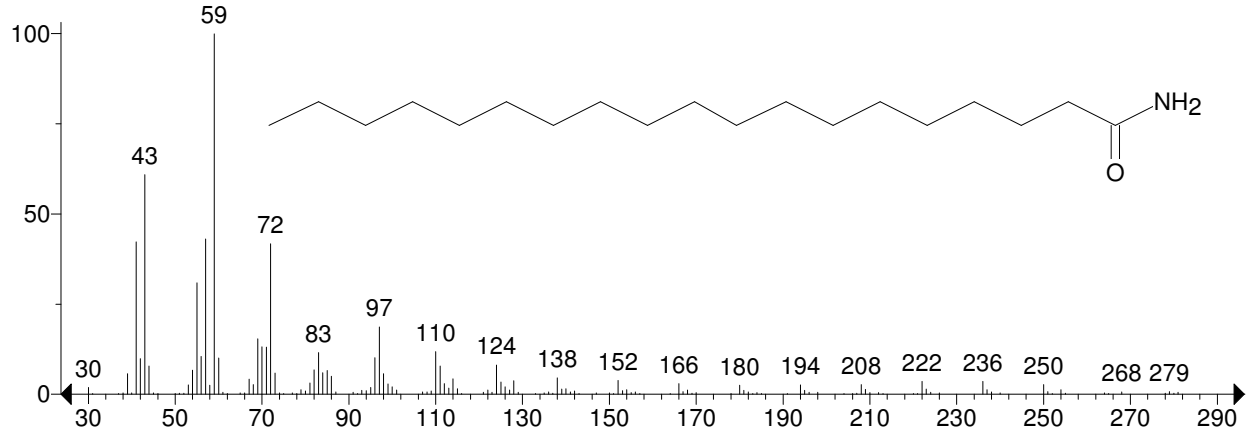

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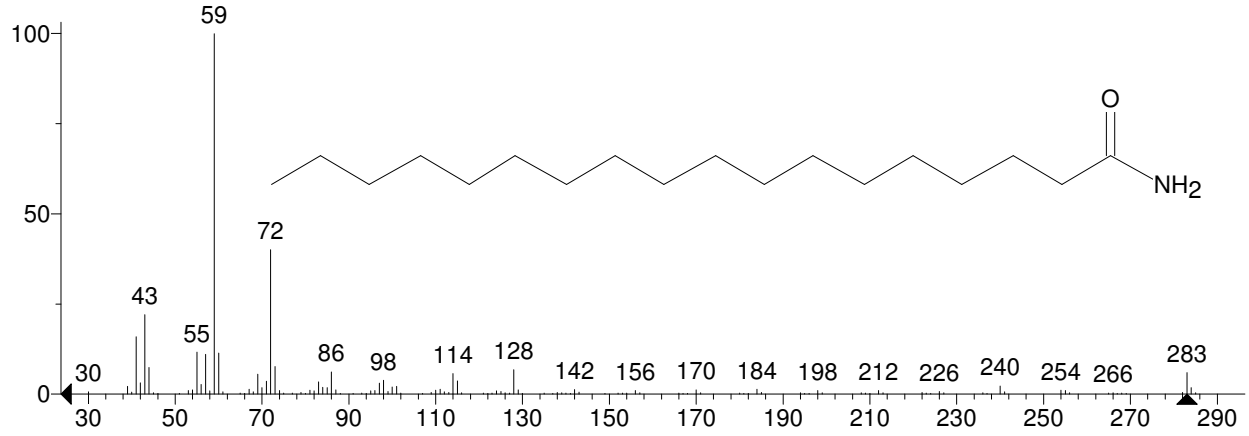

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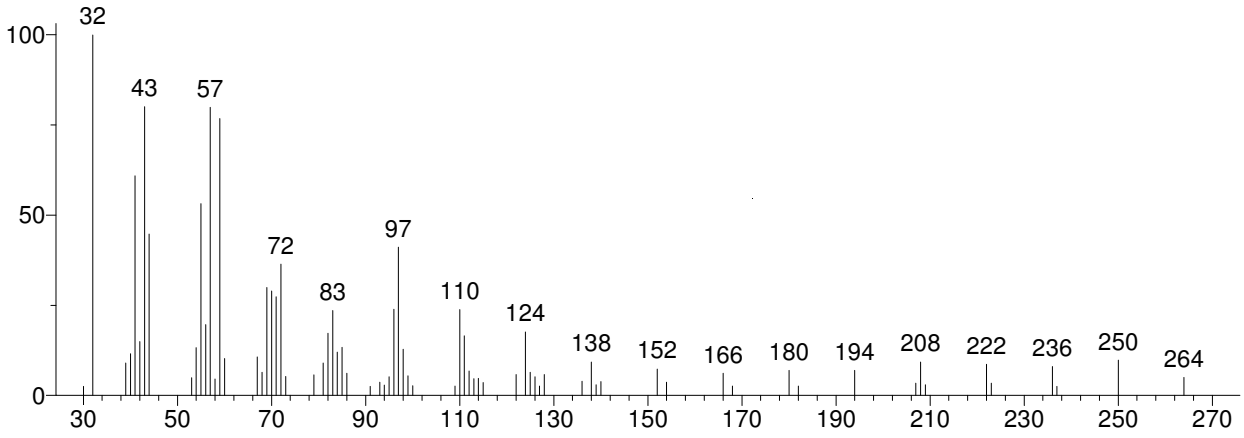
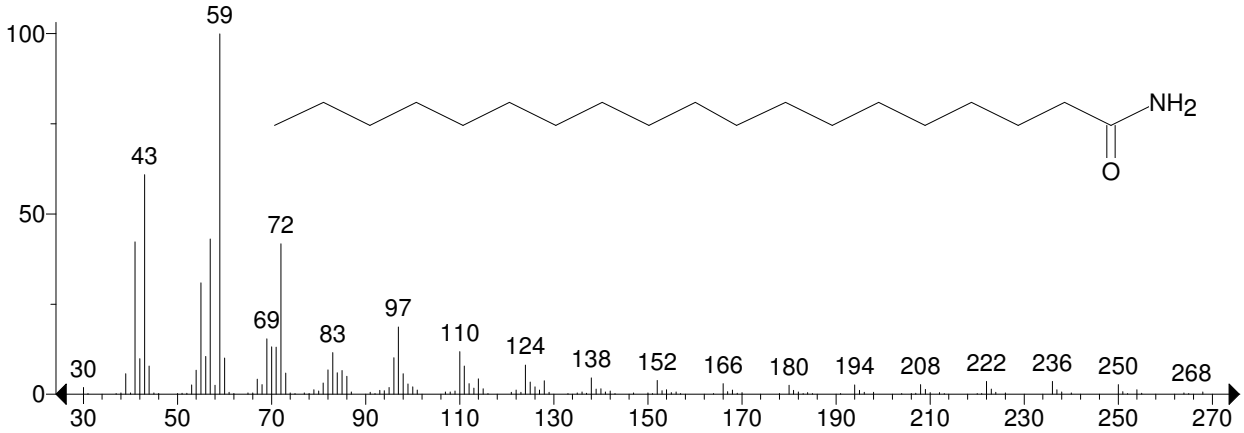
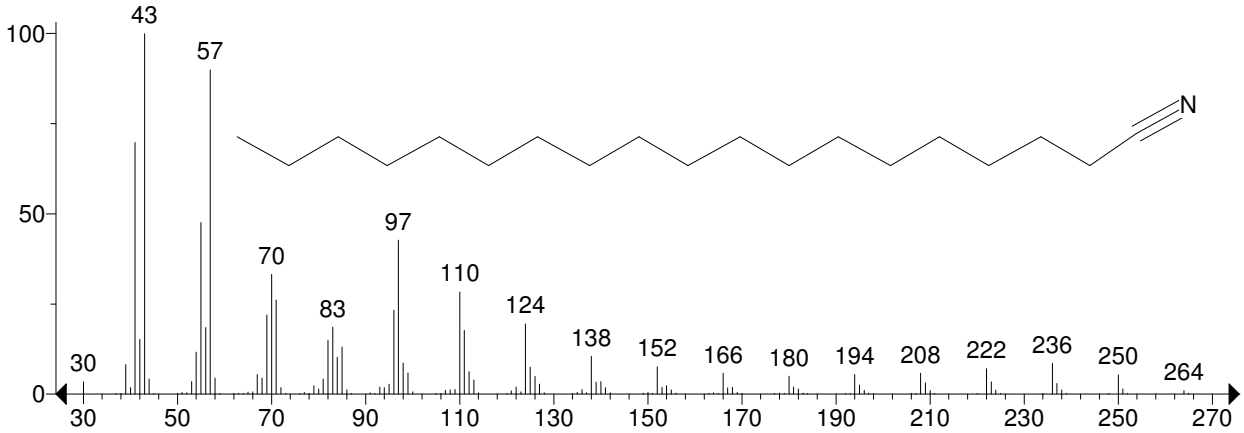
Compound in Library Factor = -197



Hit 1 : Nonadecanamide
C19H39NO; MF: 839; RMF: 857; Prob 60.2%; CAS: 58185-32-3; Lib: mainlib; ID: 24635.

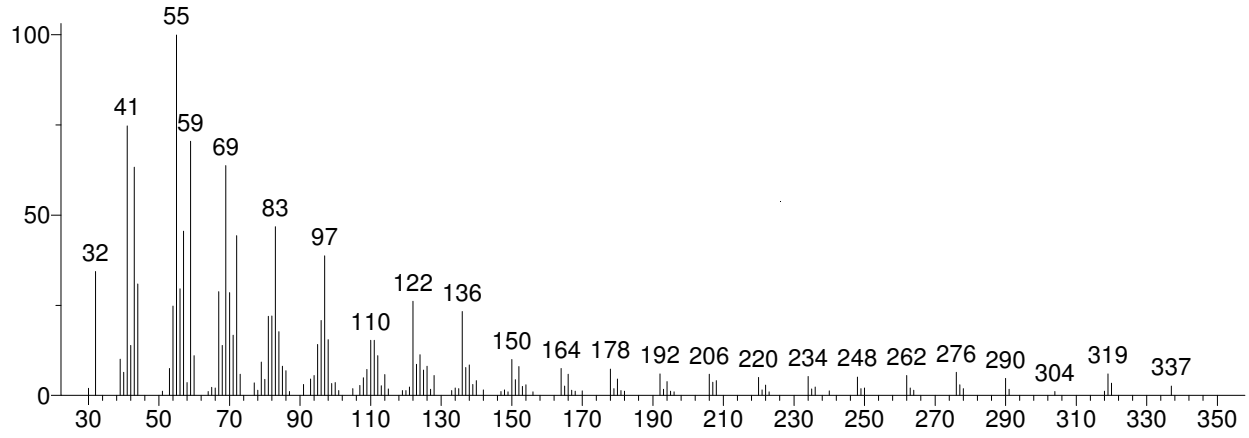


Hit 2 : Nonadecanenitrile
C19H37N; MF: 780; RMF: 839; Prob 10.9%; CAS: 28623-46-3; Lib: mainlib; ID: 6885.

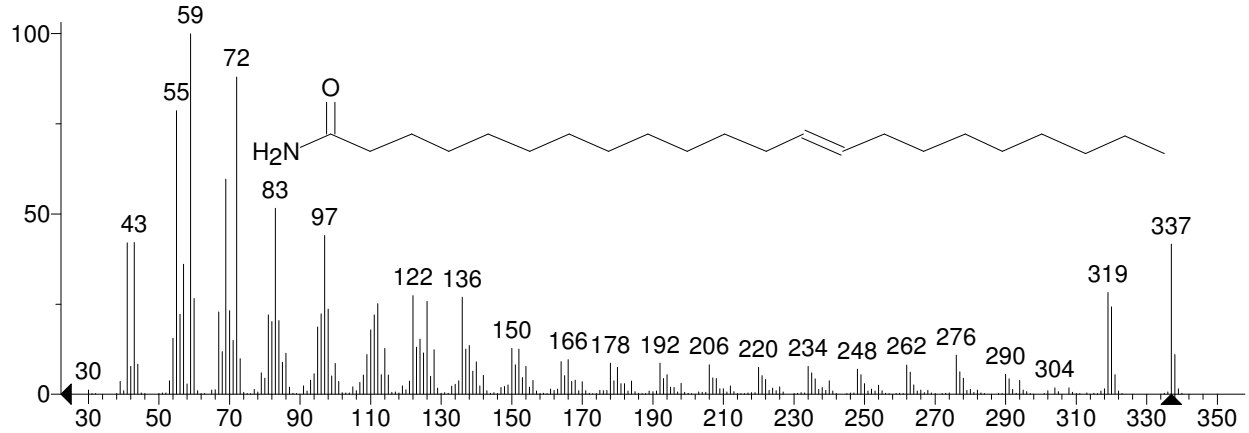


** Search Report Page 1 of 1 **

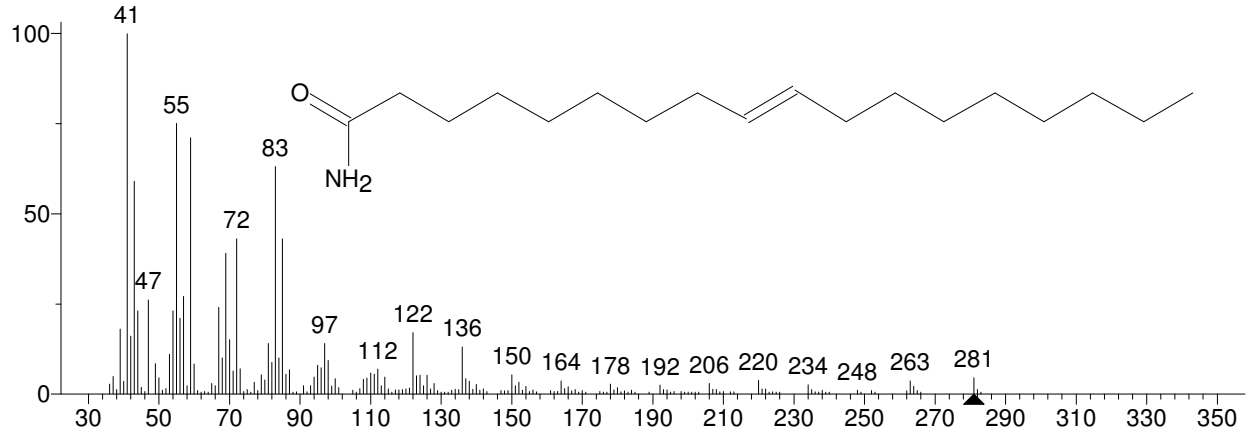
Unknown: Scan 1951 (14.789 min): J5169_Jordi_In_House_Unknown_A_py2.D\data.ms
Compound in Library Factor = -388



Hit 1 : trans-13-Docosenamide
C22H43NO; MF: 801; RMF: 802; Prob 45.6%; CAS: 10436-09-6; Lib: mainlib; ID: 24931.

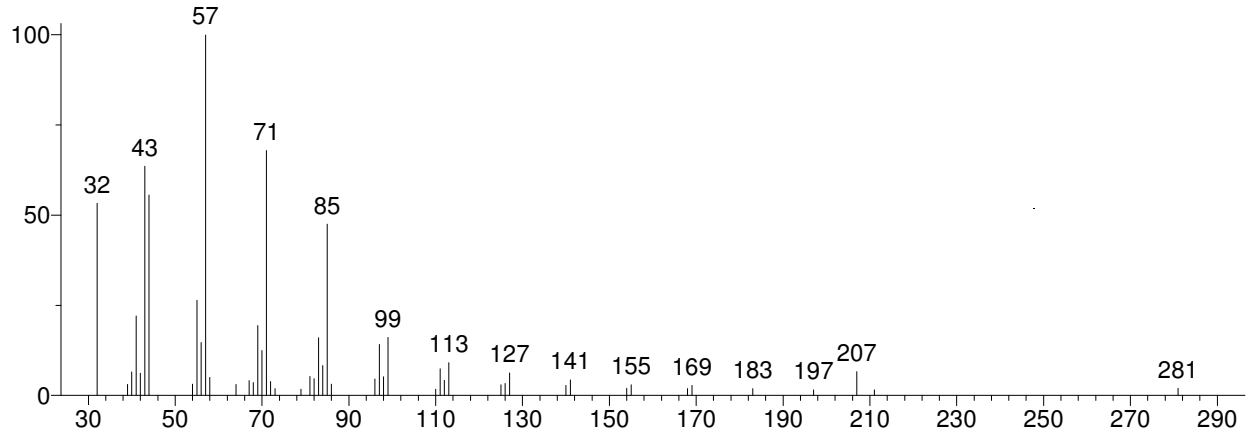


Hit 2 : 9-Octadecenamide
C18H35NO; MF: 782; RMF: 819; Prob 22.2%; CAS: 3322-62-1; Lib: mainlib; ID: 2451.

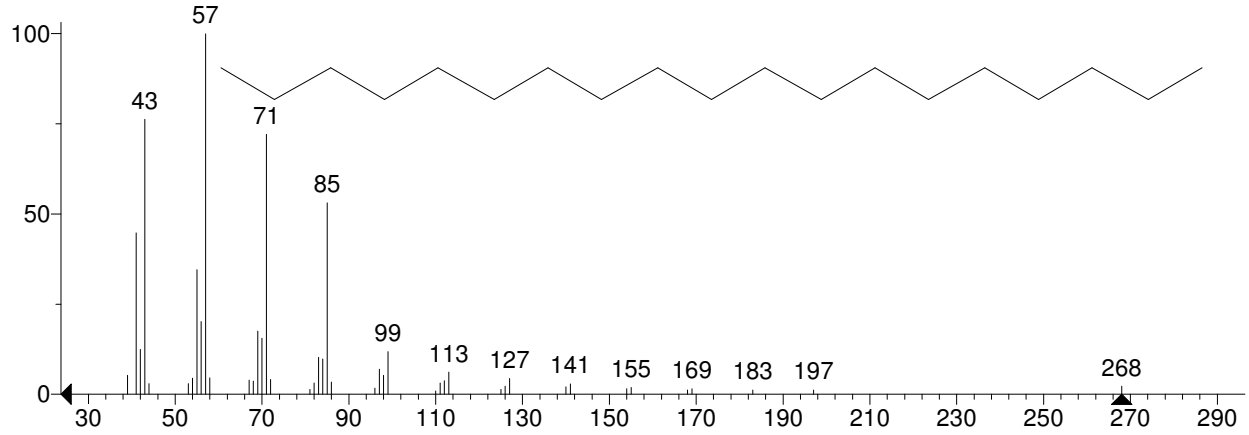


** Search Report Page 1 of 1 **

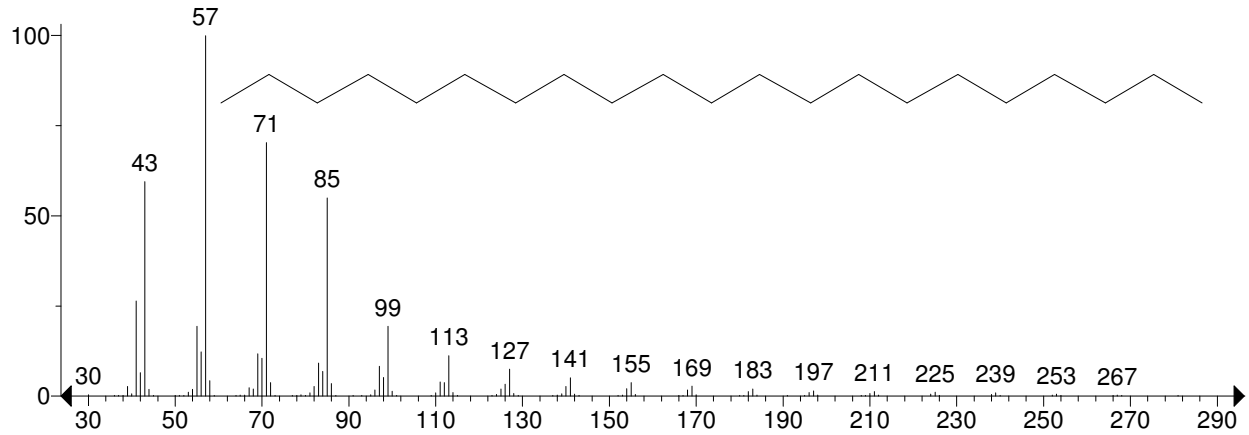
Unknown: Scan 2128 (16.123 min): J5169_Jordi_In_House_Unknown_A_py2.D\data.ms
Compound in Library Factor = -256



Hit 1 : Nonadecane
C19H40; MF: 850; RMF: 900; Prob 18.3%; CAS: 629-92-5; Lib: replib; ID: 5489.

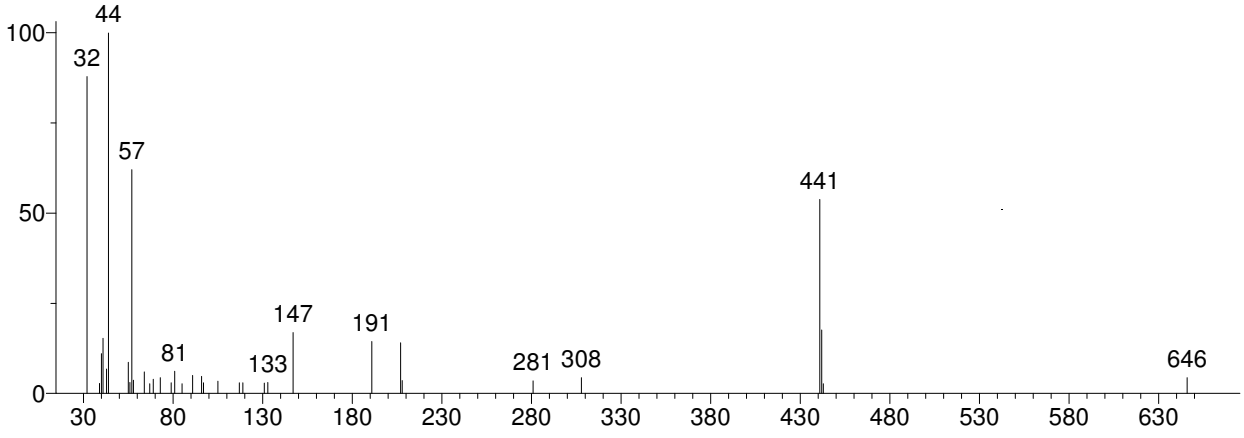


Hit 2 : Heneicosane
C21H44; MF: 818; RMF: 862; Prob 4.89%; CAS: 629-94-7; Lib: replib; ID: 5723.

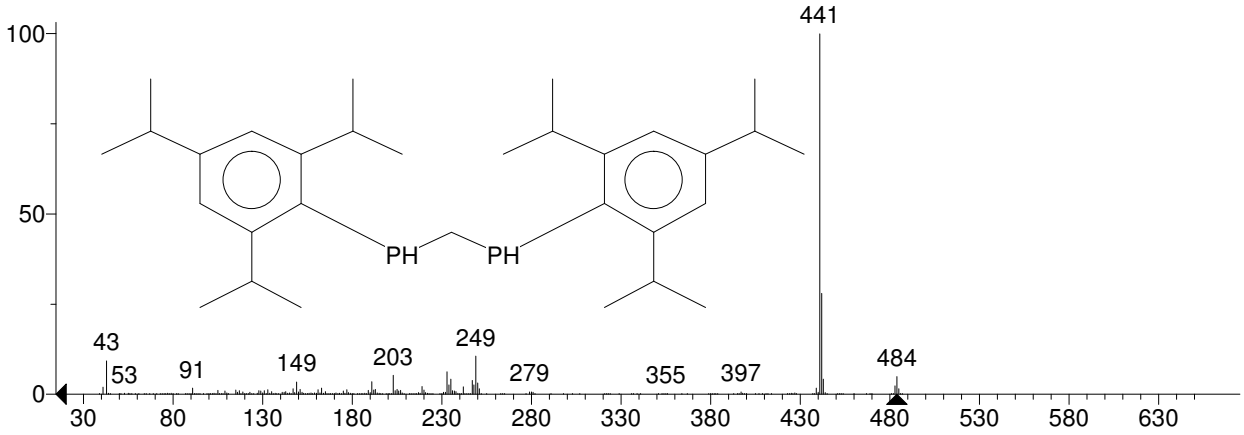


** Search Report Page 1 of 1 **

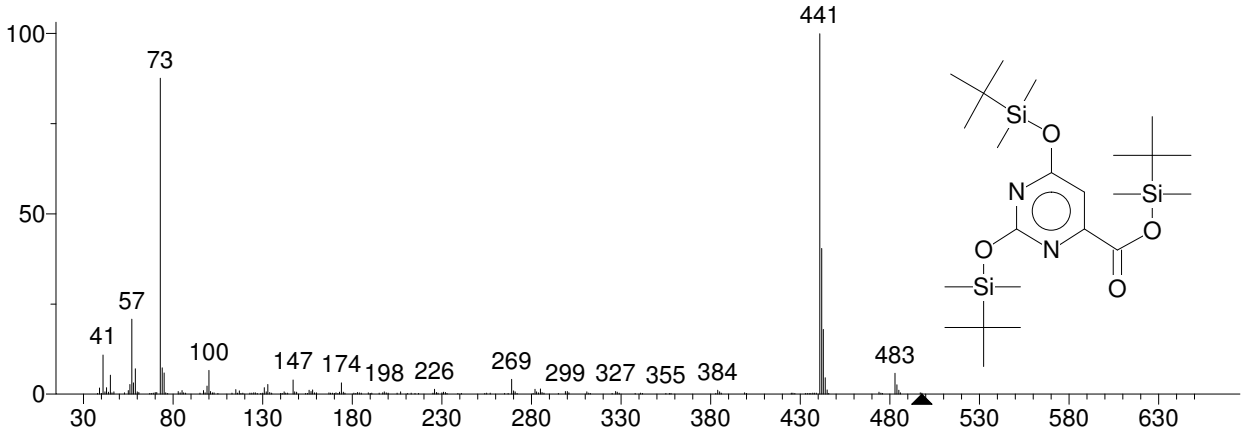
Unknown: Scan 2207 (16.718 min): J5169_Jordi_In_House_Unknown_A_py2.D\data.ms
Compound in Library Factor = -1230



Hit 1 : Methylenebis(2,4,6-triisopropylphenylphosphine)
C31H50P2; MF: 566; RMF: 651; Prob 60.1%; Lib: mainlib; ID: 161561.

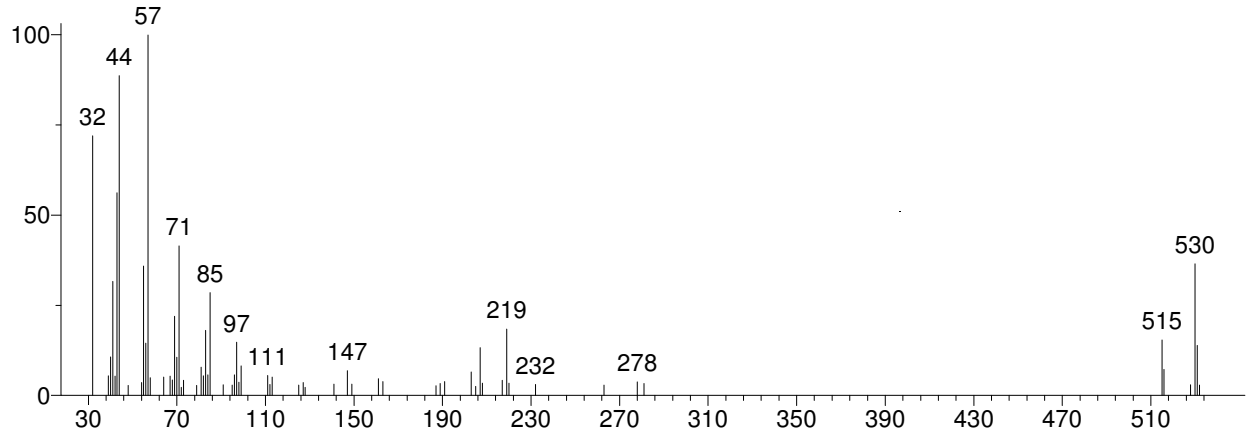


Hit 2 : 4-Pyrimidinecarboxylic acid, 2,6-bis[(tert-butyldimethylsilyl)oxy]-, tert-butyldimethylsilyl ester
C23H46N2O4Si3; MF: 543; RMF: 589; Prob 22.0%; Lib: mainlib; ID: 161552.

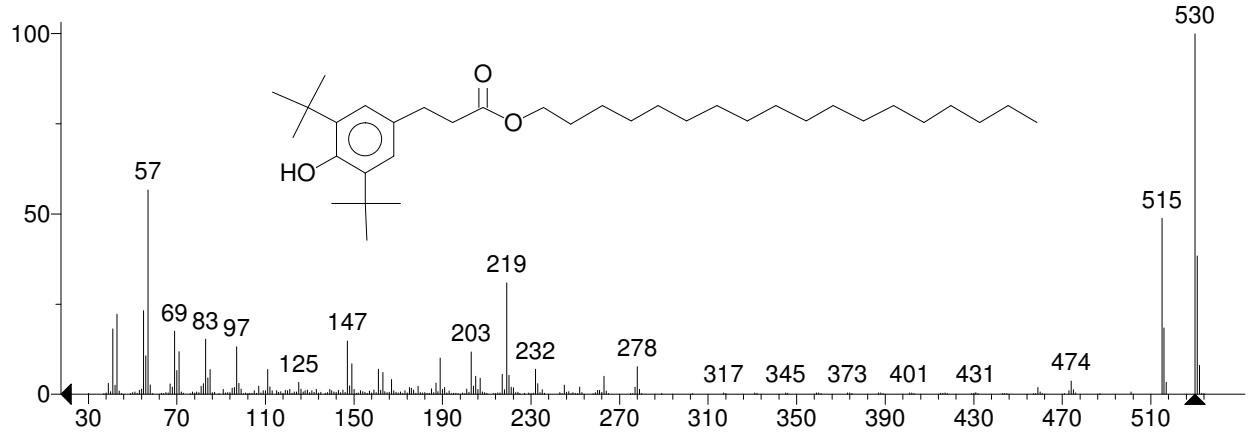


** Search Report Page 1 of 1 **

Unknown: Scan 2278 (17.253 min): J5169_Jordi_In_House_Unknown_A_py2.D\data.ms
Compound in Library Factor = -118



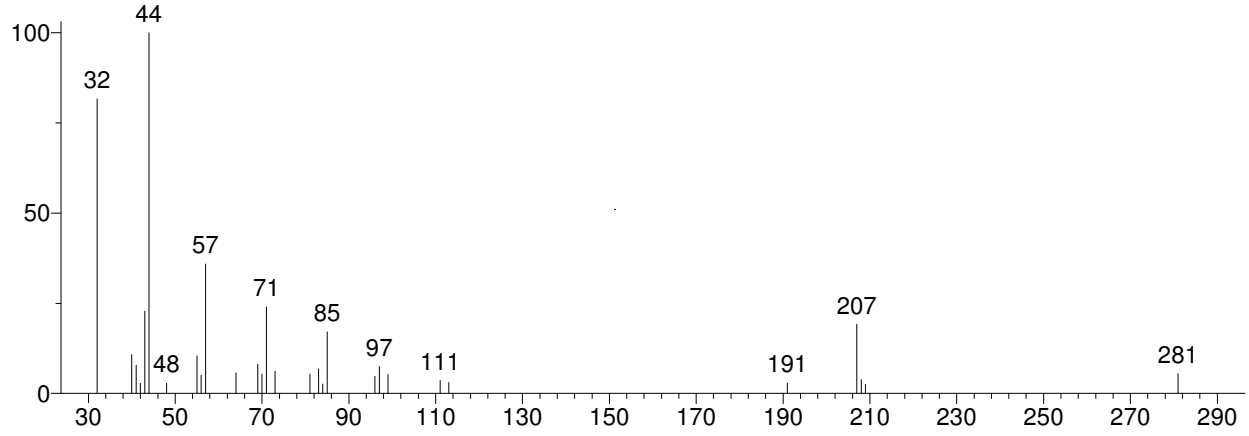
Hit 1 : Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester
C35H62O3; MF: 734; RMF: 757; Prob 80.5%; CAS: 2082-79-3; Lib: mainlib; ID: 162602.



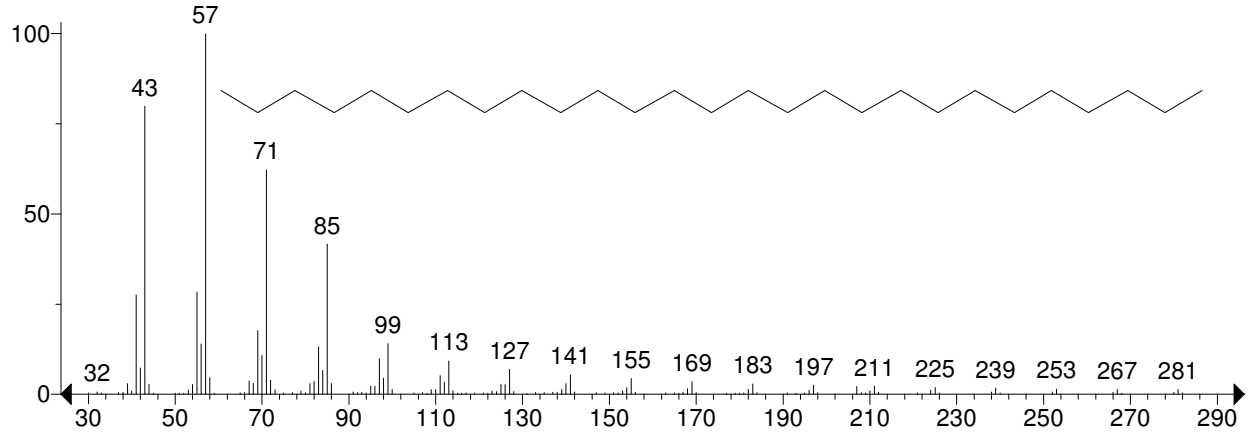
Hit 2 : Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester
C35H62O3; MF: 719; RMF: 728; Prob 80.5%; CAS: 2082-79-3; Lib: replib; ID: 27601.

** Search Report Page 1 of 1 **

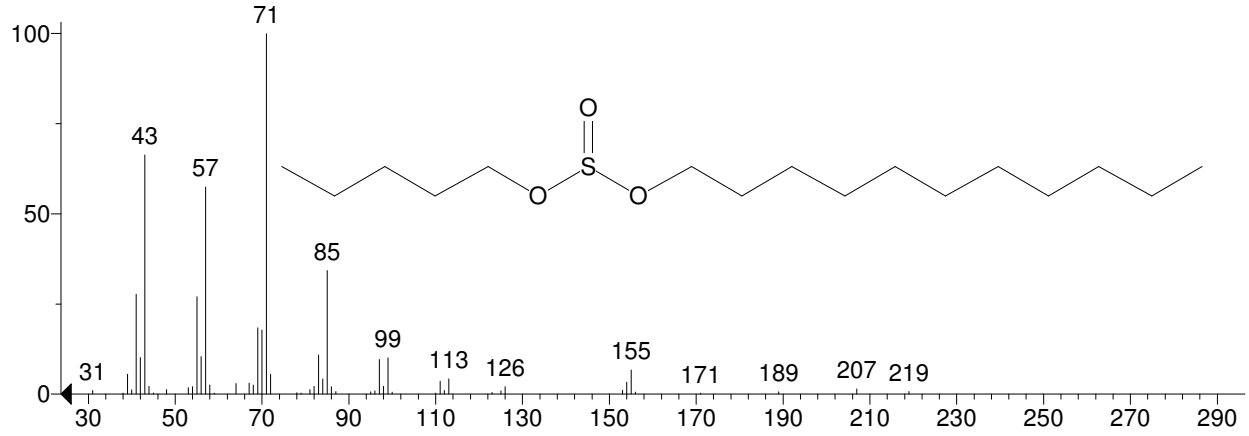
Unknown: Scan 2446 (18.519 min): J5169_Jordi_In_House_Unknown_A_py2.D\data.ms
Compound in Library Factor = -2259



Hit 1 : Heptacosane
C27H56; MF: 525; RMF: 530; Prob 3.58%; CAS: 593-49-7; Lib: replib; ID: 5461.

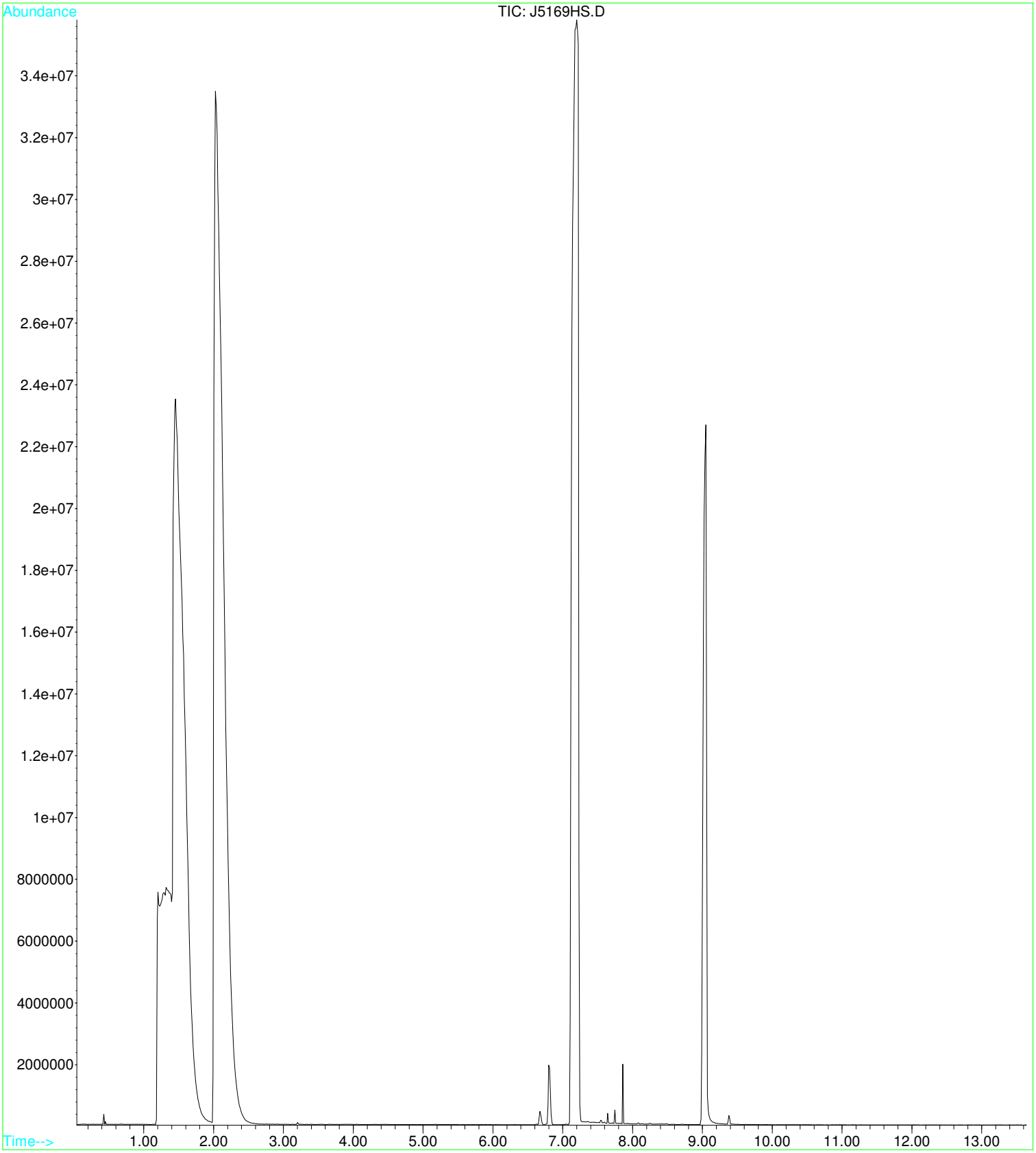


Hit 2 : Sulfurous acid, pentyl undecyl ester
C16H34O3S; MF: 524; RMF: 610; Prob 3.44%; Lib: mainlib; ID: 30897.



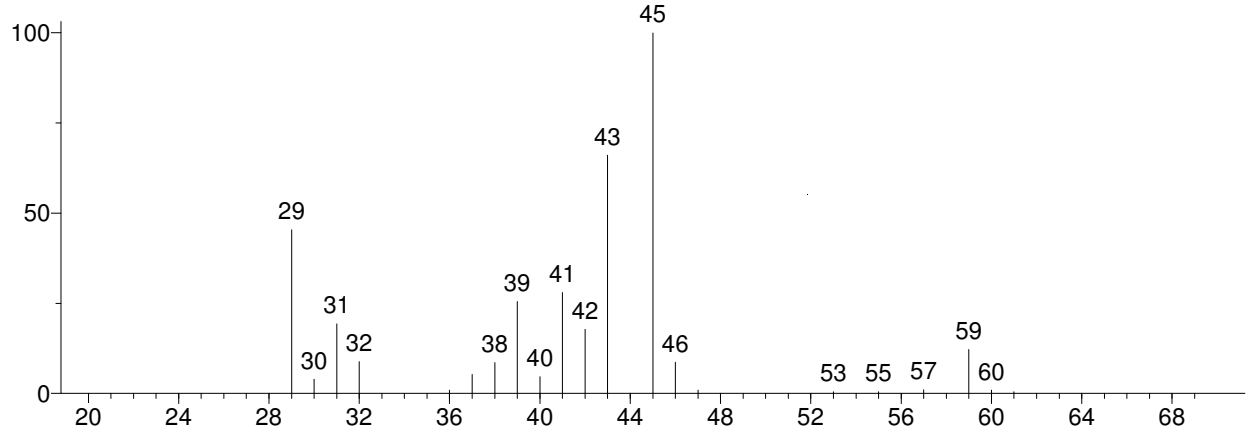
HEADSPACE GCMS RESULTS

```
File       : C:\HPCHEM\1\DATA\2010\JOB2010\J5169JOR\J5169HS.D
Operator   :
Acquired   : 21 Oct 2010  11:44     using AcqMethod HEADSTD2
Instrument :   DMS Instr
Sample Name: J5169 Jordi Unknown
Misc Info  :
Vial Number: 1
```

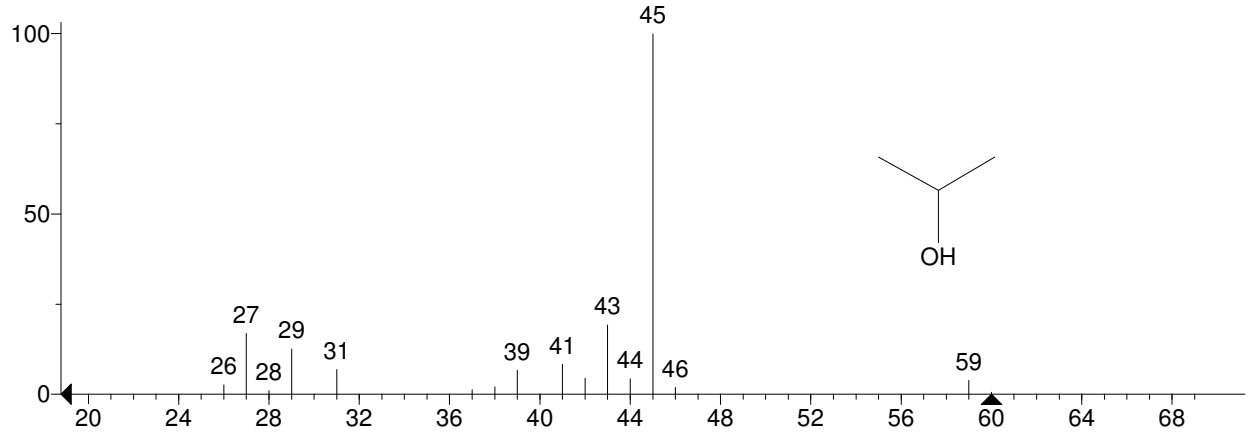


** Search Report Page 1 of 1 **

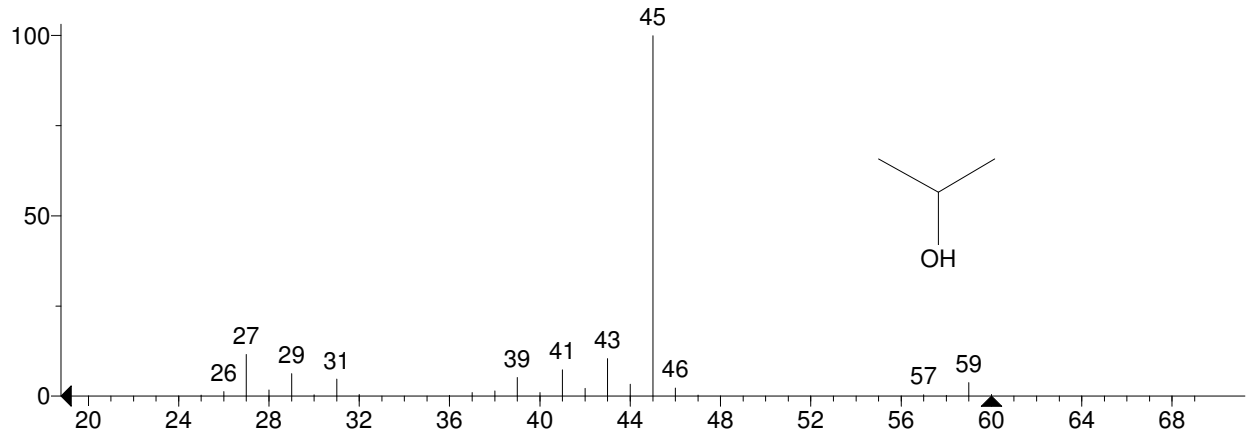
Unknown: Scan 125 (1.455 min): J5169HS.D
Compound in Library Factor = -136



Hit 1 : Isopropyl Alcohol
C3H8O; MF: 826; RMF: 857; Prob 55.5%; CAS: 67-63-0; Lib: replib; ID: 3548.

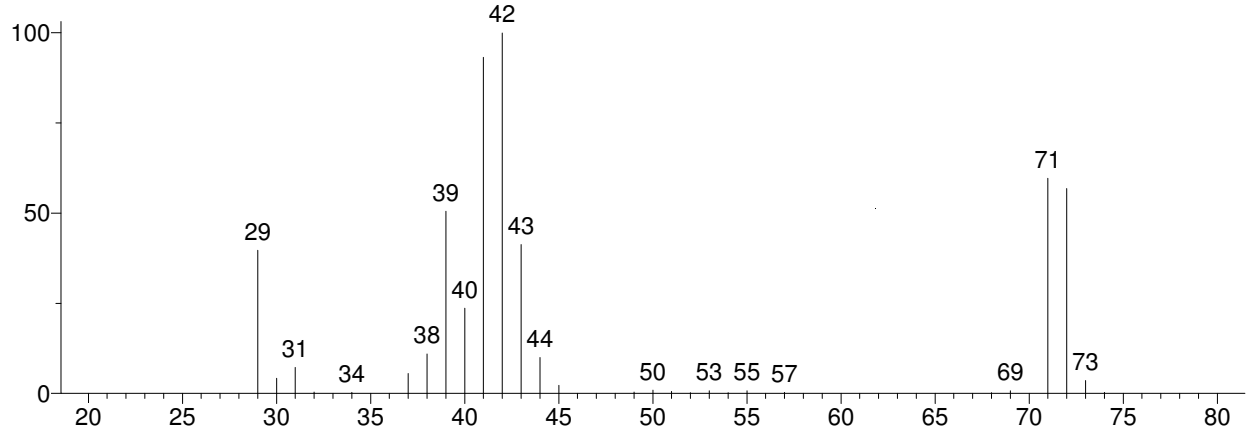


Hit 2 : Isopropyl Alcohol
C3H8O; MF: 802; RMF: 804; Prob 55.5%; CAS: 67-63-0; Lib: mainlib; ID: 14532.

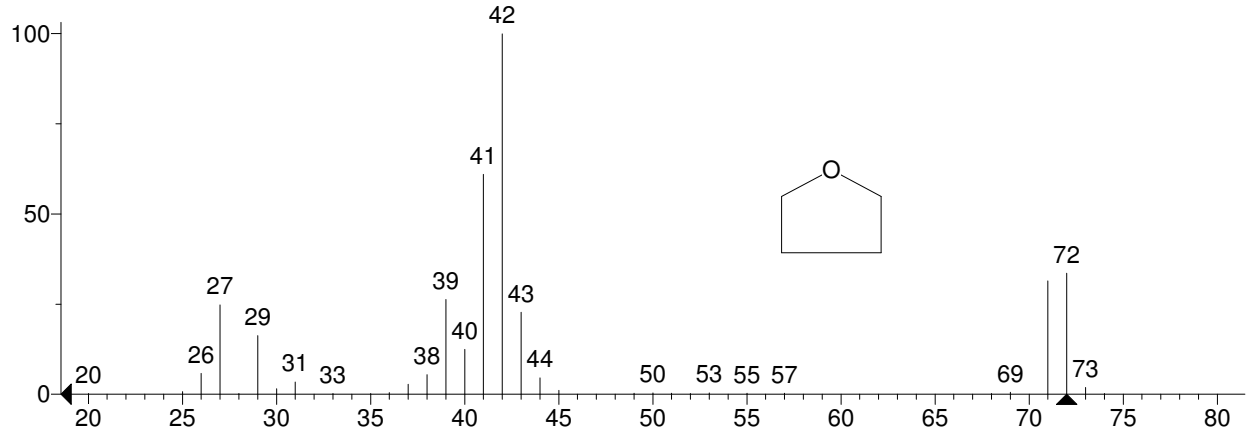


** Search Report Page 1 of 1 **

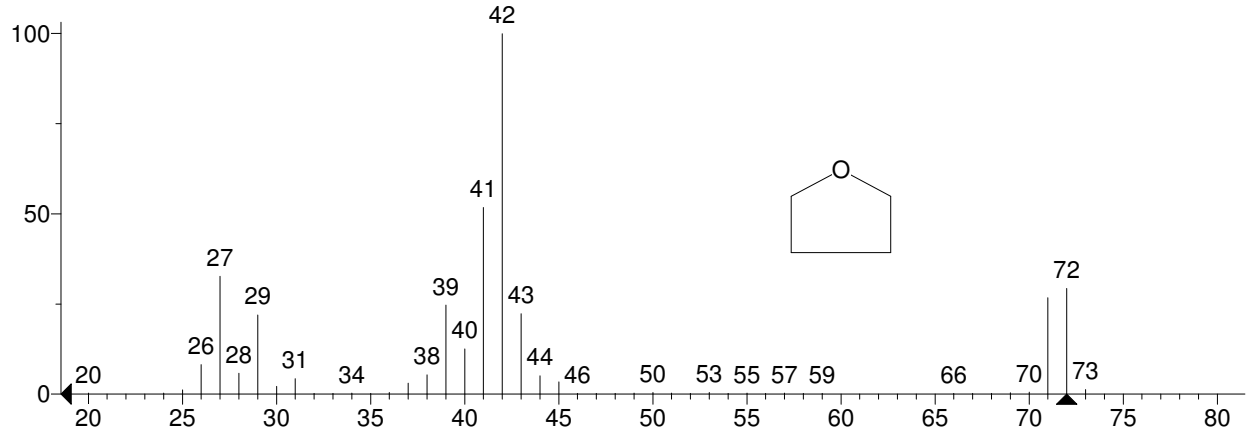
Unknown: Scan 175 (2.026 min): J5169HS.D
Compound in Library Factor = 237



Hit 1 : Furan, tetrahydro-
C4H8O; MF: 933; RMF: 933; Prob 87.4%; CAS: 109-99-9; Lib: replib; ID: 1325.

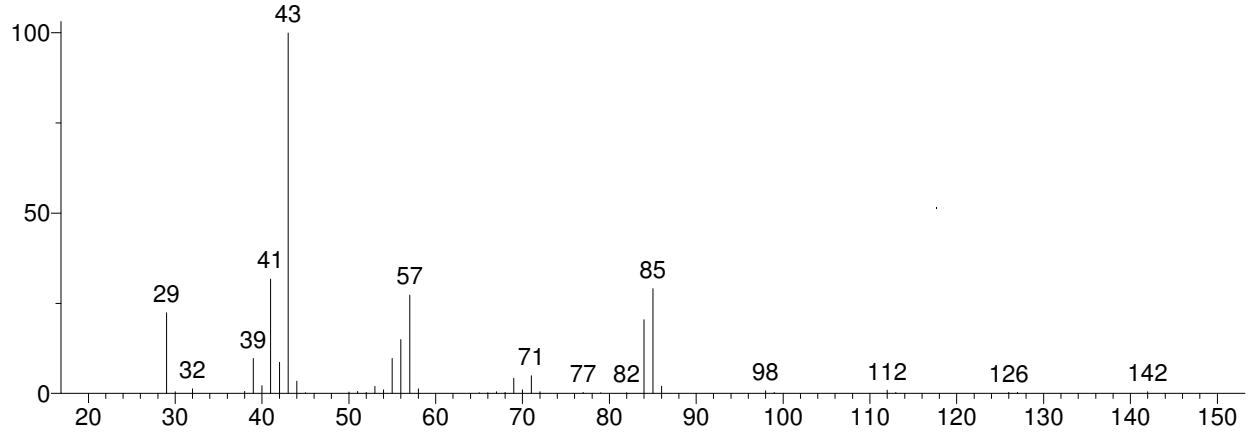


Hit 2 : Furan, tetrahydro-
C4H8O; MF: 893; RMF: 894; Prob 87.4%; CAS: 109-99-9; Lib: replib; ID: 1305.

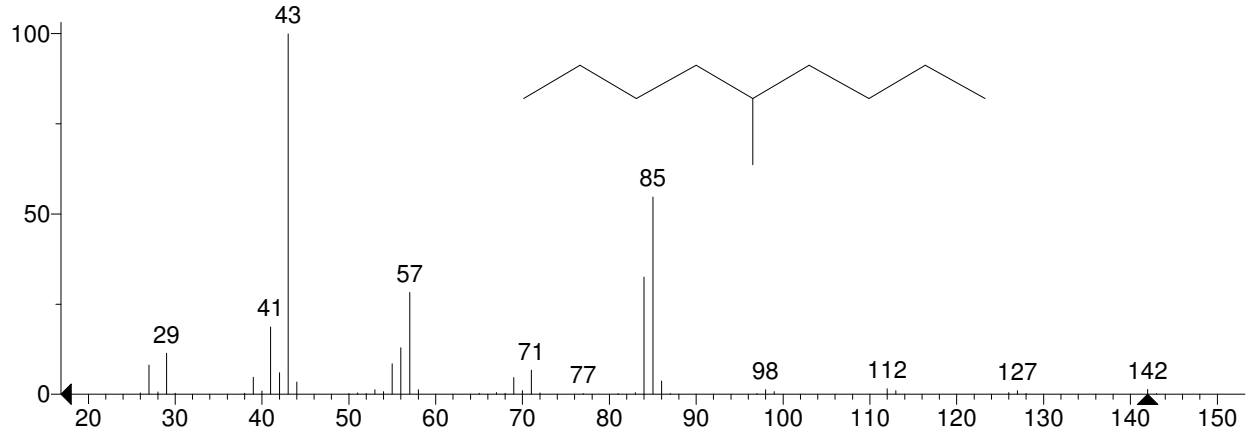


** Search Report Page 1 of 1 **

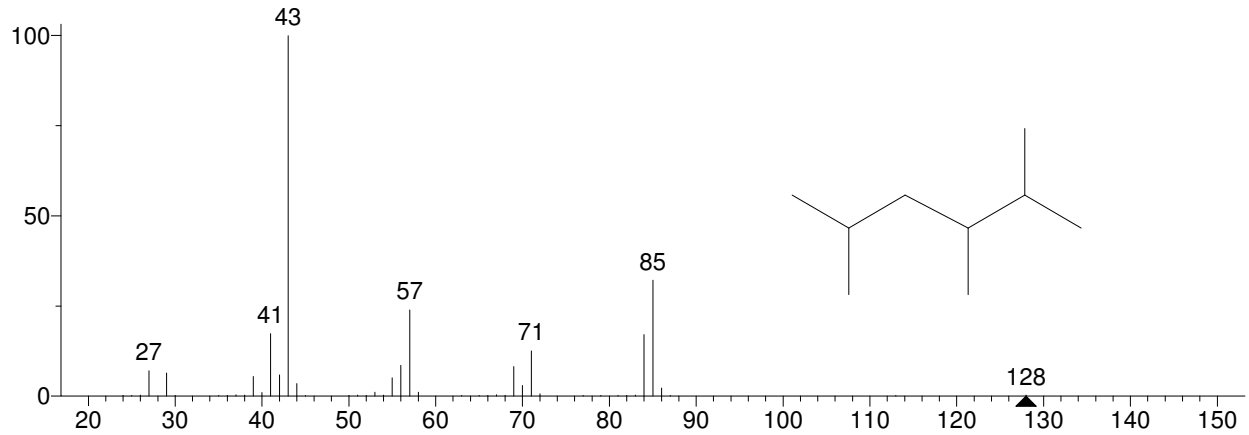
Unknown: Scan 582 (6.673 min): J5169HS.D
Compound in Library Factor = -151



Hit 1 : Nonane, 5-methyl-
C10H22; MF: 894; RMF: 895; Prob 15.0%; CAS: 15869-85-9; Lib: mainlib; ID: 8816.

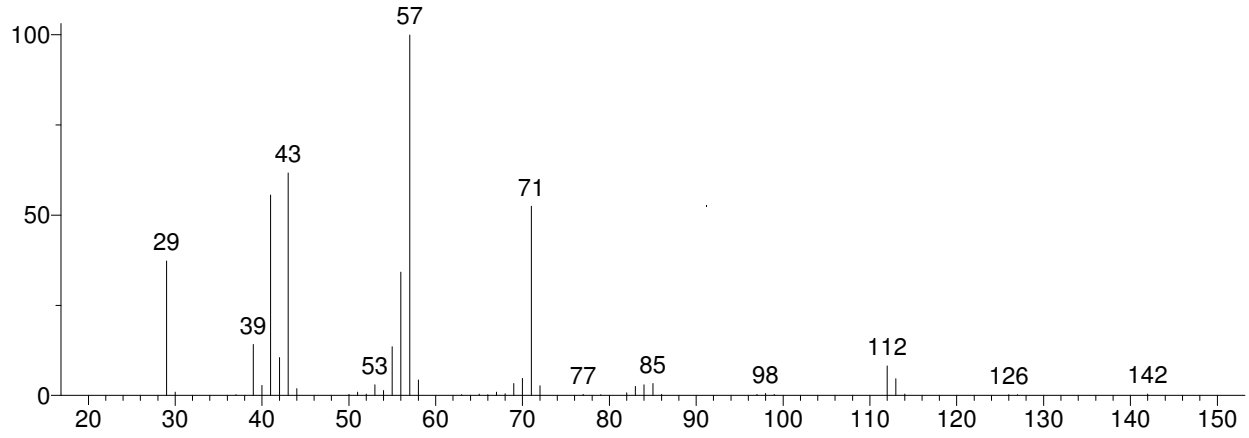


Hit 2 : Hexane, 2,3,5-trimethyl-
C9H20; MF: 890; RMF: 903; Prob 12.7%; CAS: 1069-53-0; Lib: replib; ID: 2635.

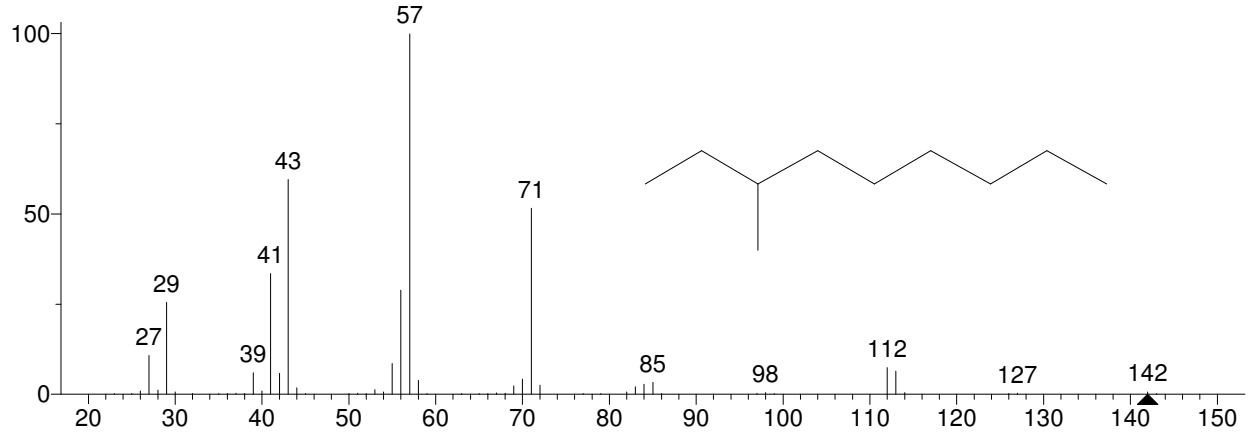


** Search Report Page 1 of 1 **

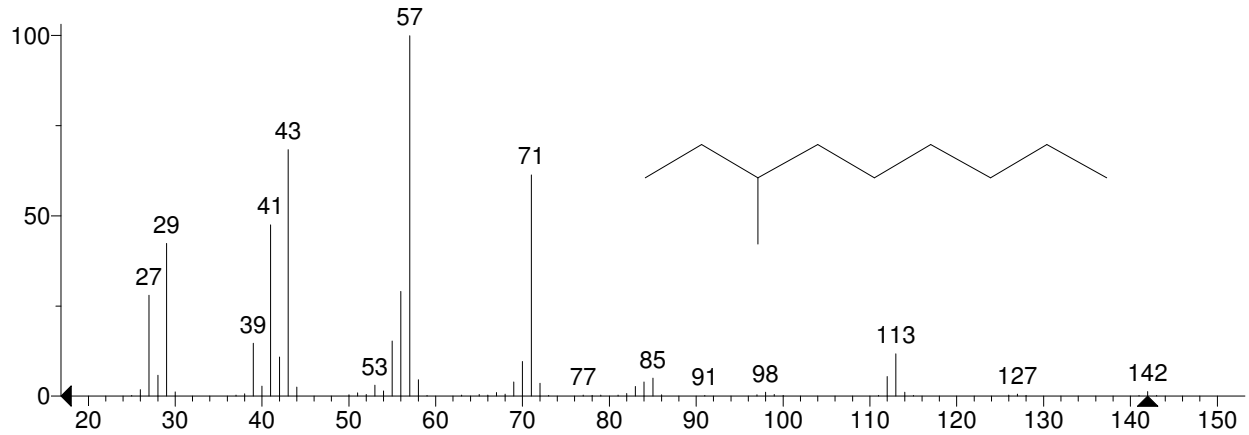
Unknown: Scan 594 (6.810 min): J5169HS.D
Compound in Library Factor = 109



Hit 1 : Nonane, 3-methyl-
C10H22; MF: 925; RMF: 925; Prob 32.1%; CAS: 5911-04-6; Lib: replib; ID: 5419.

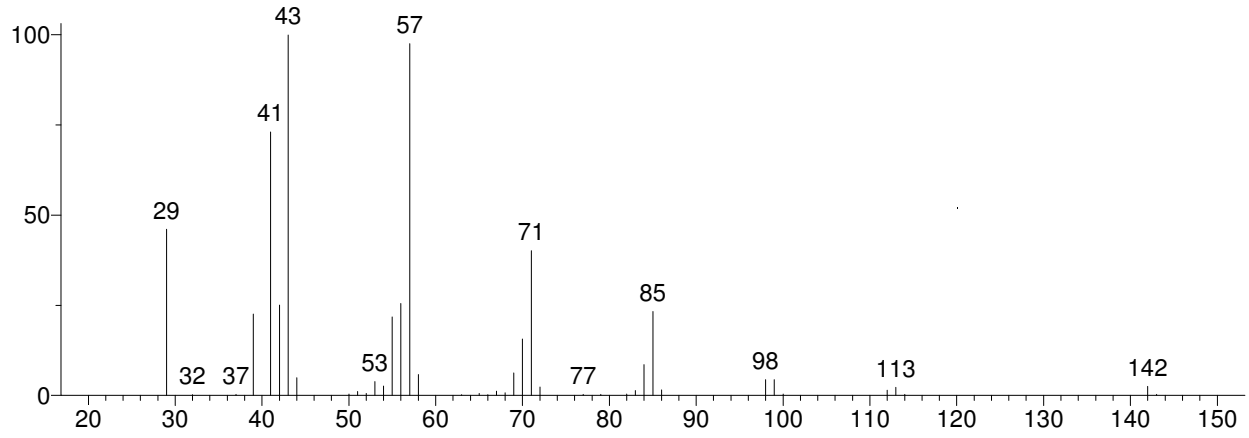


Hit 2 : Nonane, 3-methyl-
C10H22; MF: 917; RMF: 917; Prob 32.1%; CAS: 5911-04-6; Lib: replib; ID: 5418.

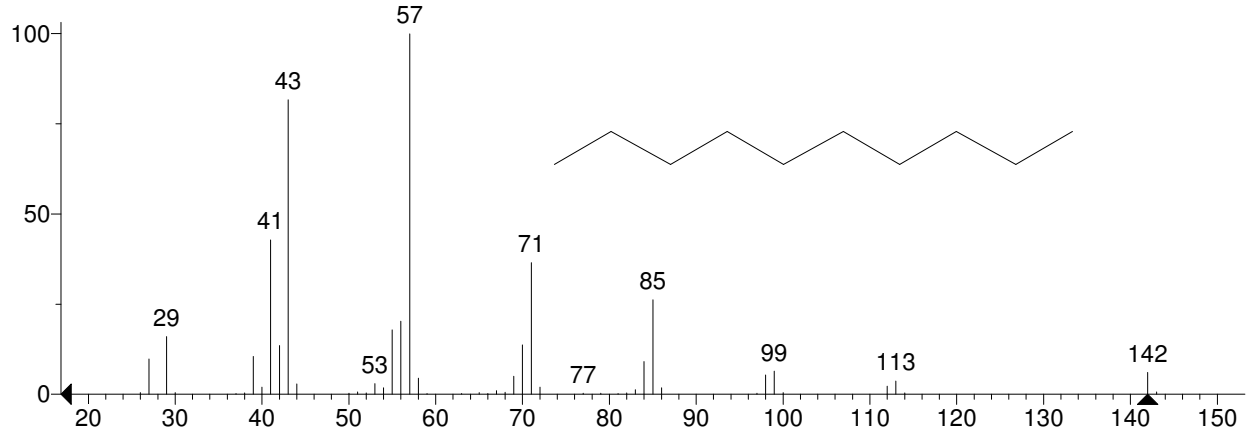


** Search Report Page 1 of 1 **

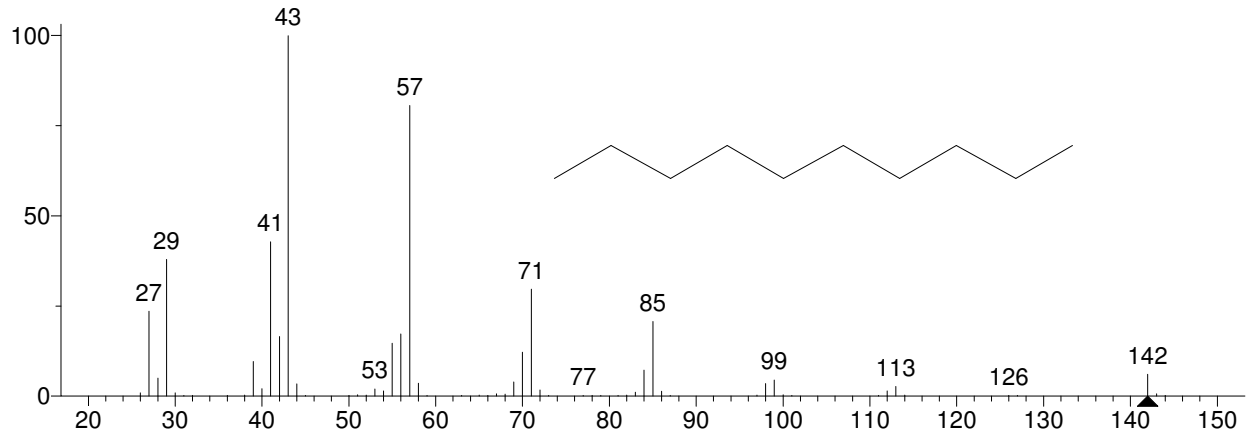
Unknown: Scan 627 (7.187 min): J5169HS.D
Compound in Library Factor = 144



Hit 1 : Decane
C10H22; MF: 952; RMF: 952; Prob 39.6%; CAS: 124-18-5; Lib: replib; ID: 5329.

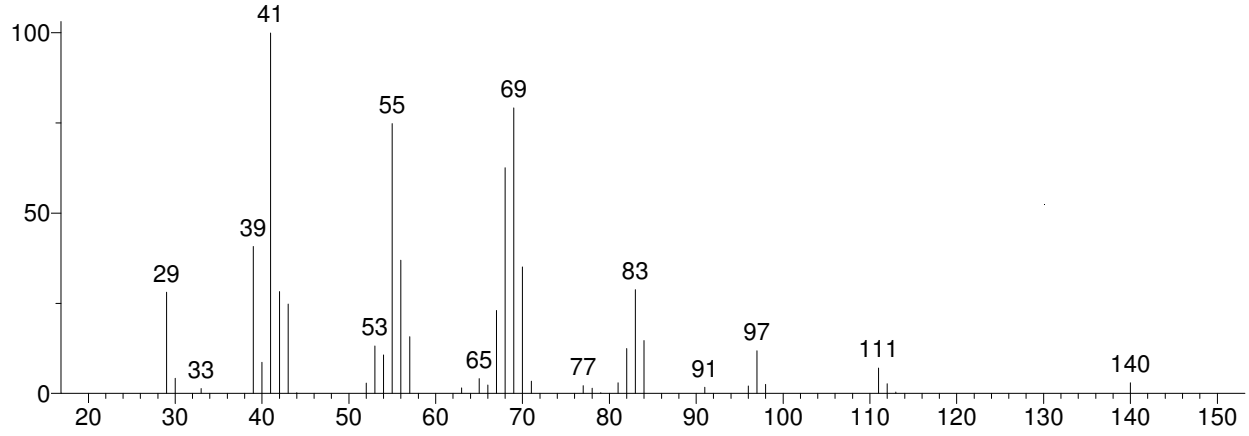


Hit 2 : Decane
C10H22; MF: 940; RMF: 941; Prob 39.6%; CAS: 124-18-5; Lib: mainlib; ID: 6868.

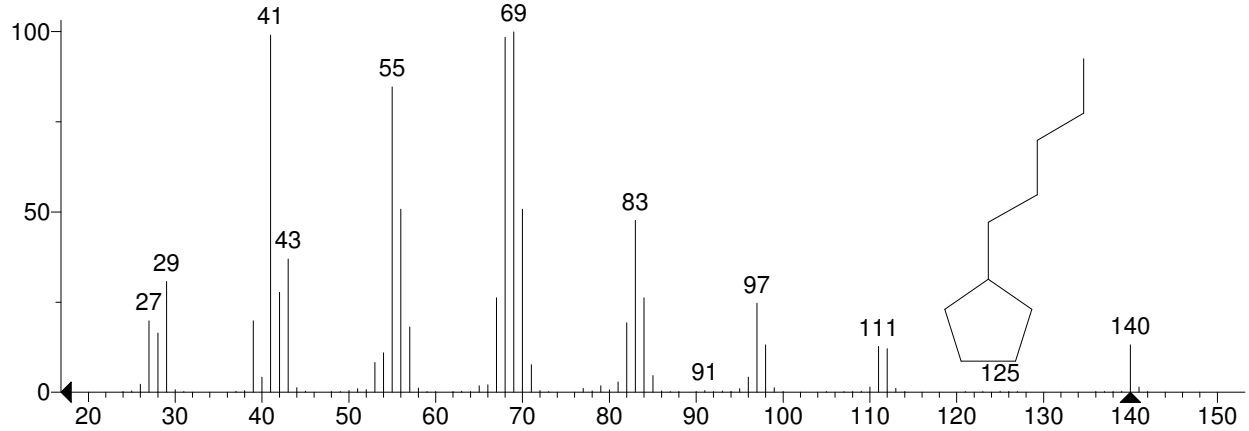


** Search Report Page 1 of 1 **

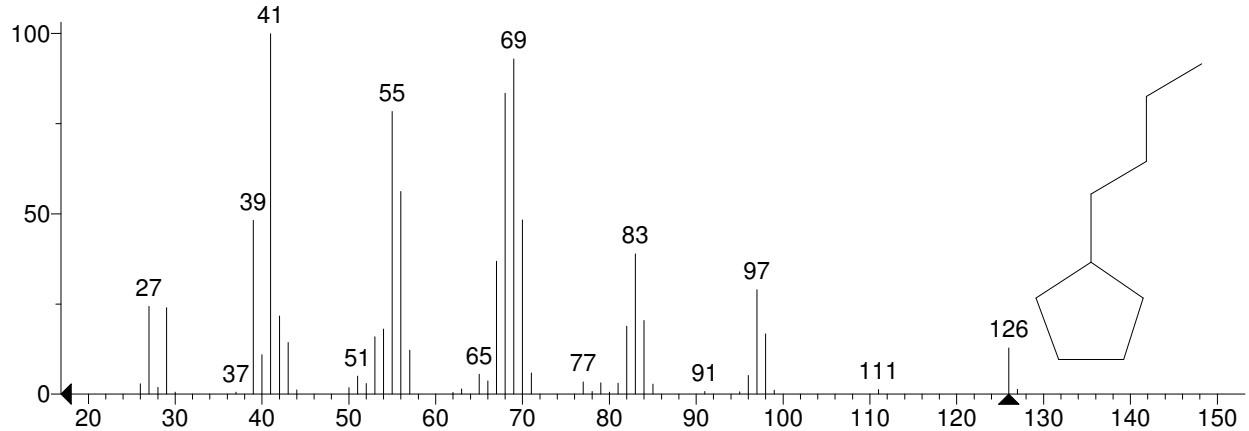
Unknown: Scan 658 (7.541 min): J5169HS.D
Compound in Library Factor = -246



Hit 1 : Cyclopentane, pentyl-
C10H20; MF: 861; RMF: 862; Prob 13.8%; CAS: 3741-00-2; Lib: mainlib; ID: 28159.

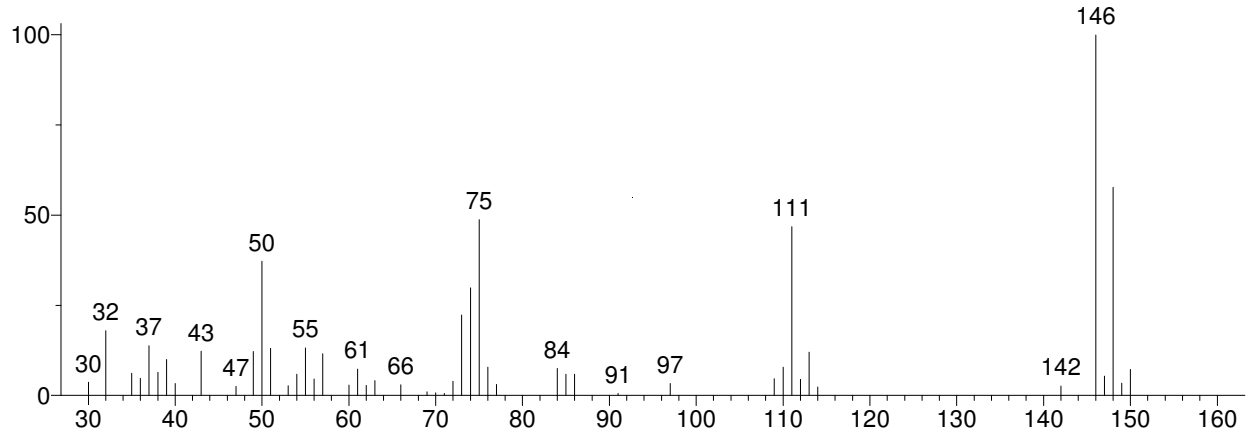


Hit 2 : Cyclopentane, butyl-
C9H18; MF: 855; RMF: 865; Prob 10.9%; CAS: 2040-95-1; Lib: mainlib; ID: 3070.

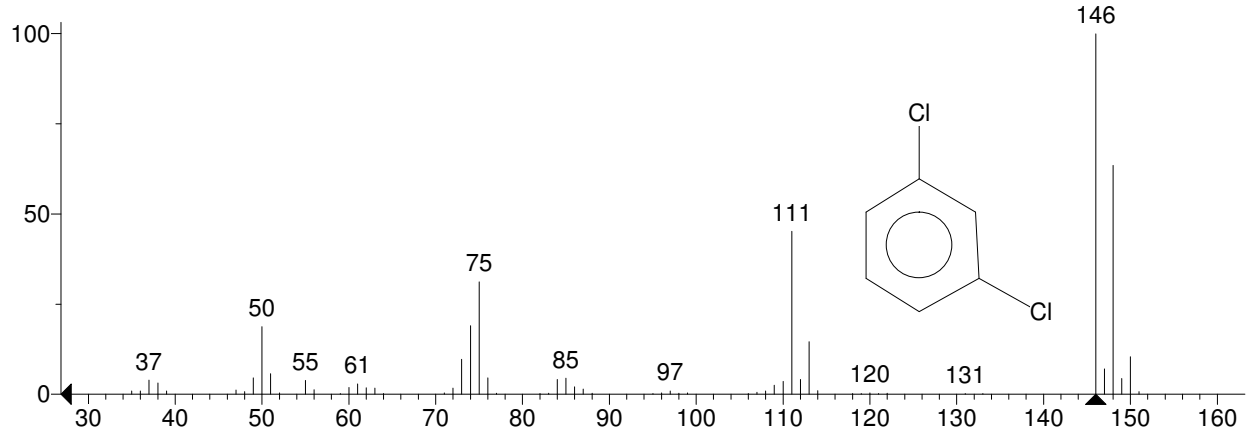


** Search Report Page 1 of 1 **

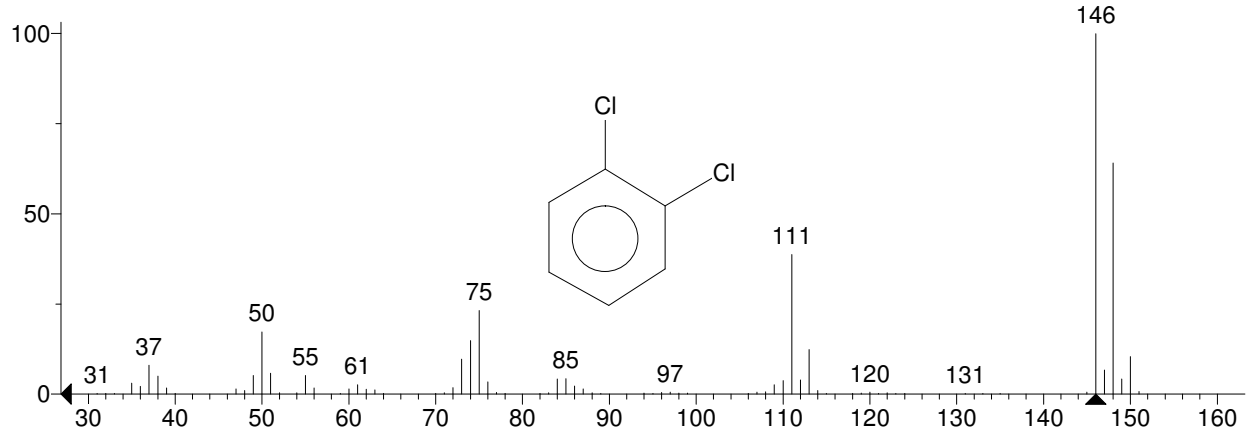
Unknown: Scan 663 (7.598 min): J5169HS.D
Compound in Library Factor = 226



Hit 1 : Benzene, 1,3-dichloro-
C6H4Cl2; MF: 866; RMF: 897; Prob 35.9%; CAS: 541-73-1; Lib: replib; ID: 19431.

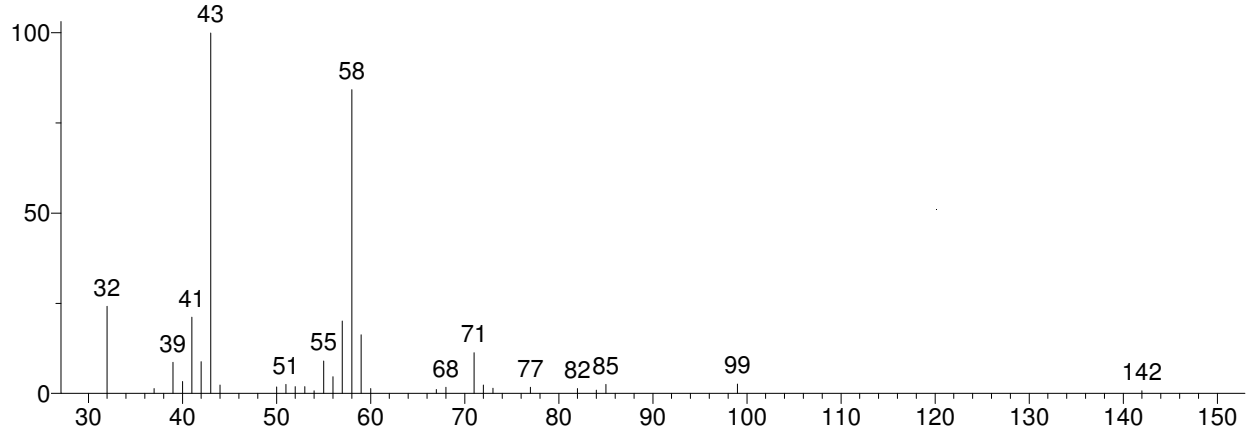


Hit 2 : Benzene, 1,2-dichloro-
C6H4Cl2; MF: 866; RMF: 886; Prob 35.9%; CAS: 95-50-1; Lib: replib; ID: 19426.

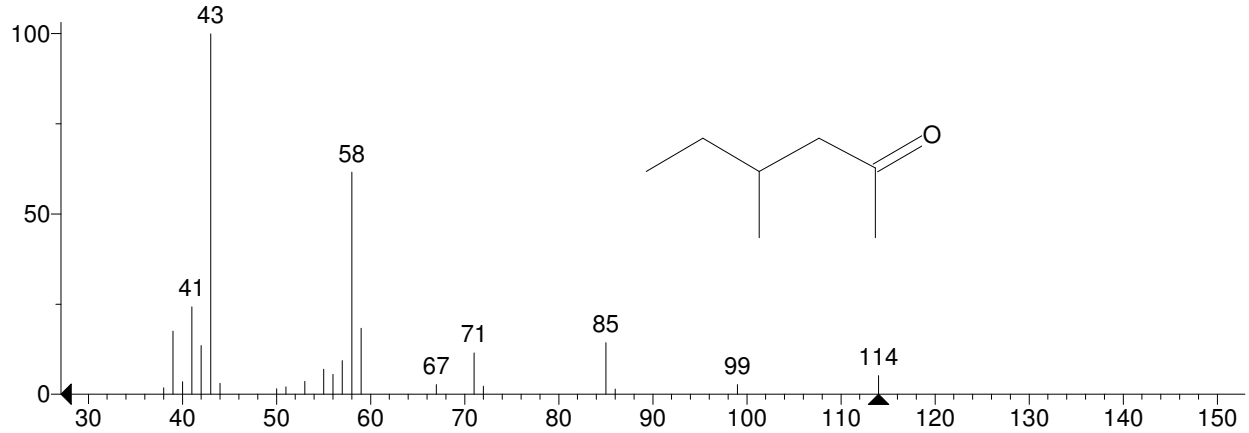


** Search Report Page 1 of 1 **

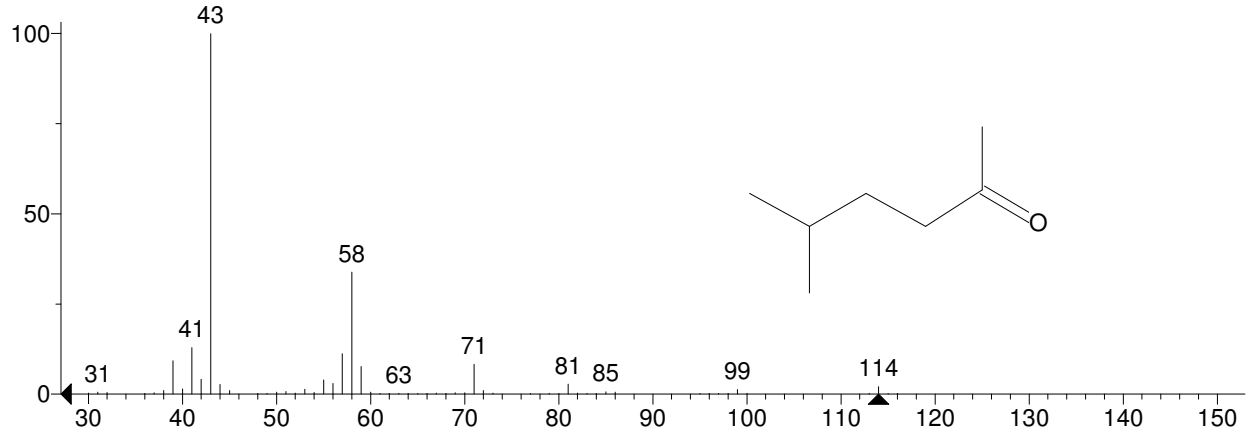
Unknown: Scan 705 (8.077 min): J5169HS.D
Compound in Library Factor = -367



Hit 1 : 2-Hexanone, 4-methyl-
C7H14O; MF: 826; RMF: 879; Prob 24.0%; CAS: 105-42-0; Lib: replib; ID: 2188.

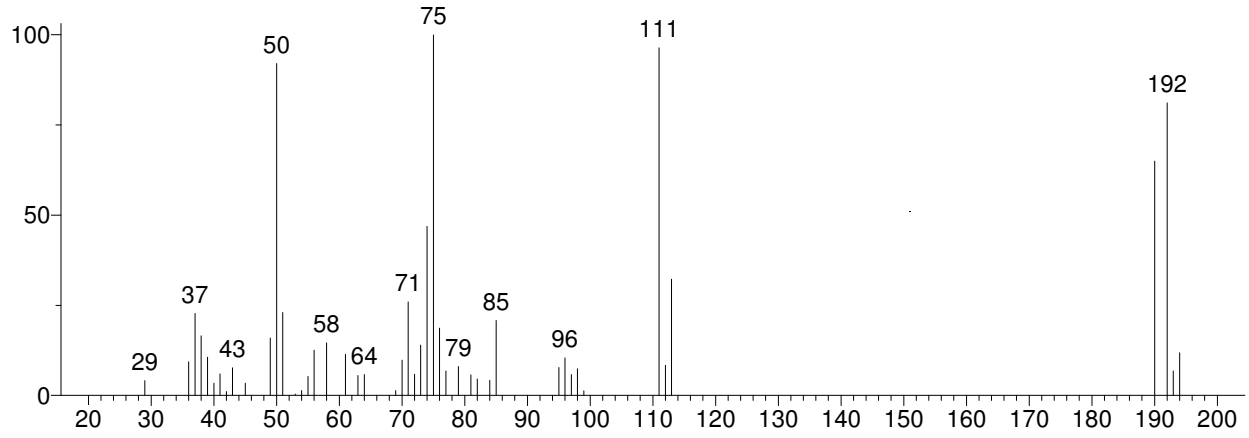


Hit 2 : 2-Hexanone, 5-methyl-
C7H14O; MF: 816; RMF: 828; Prob 16.9%; CAS: 110-12-3; Lib: mainlib; ID: 7217.

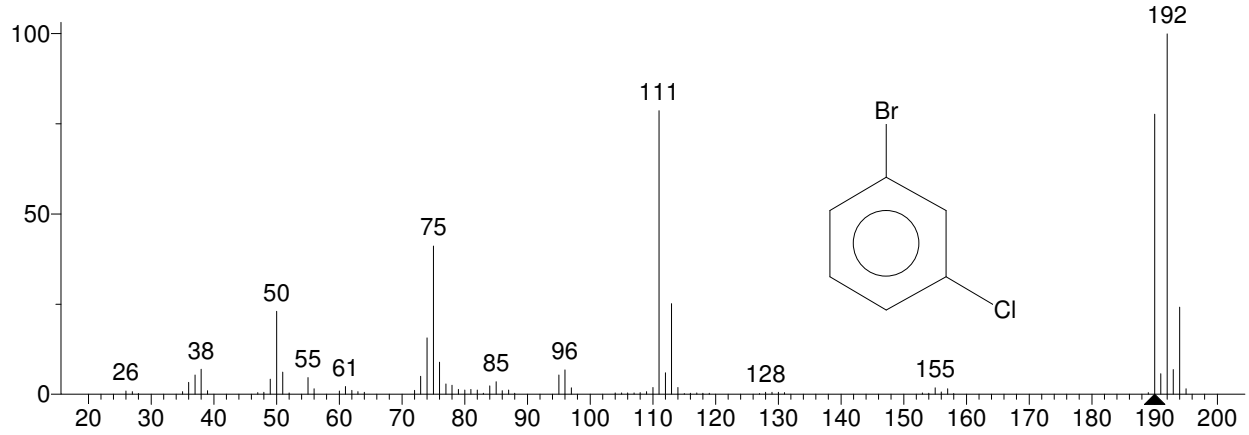


** Search Report Page 1 of 1 **

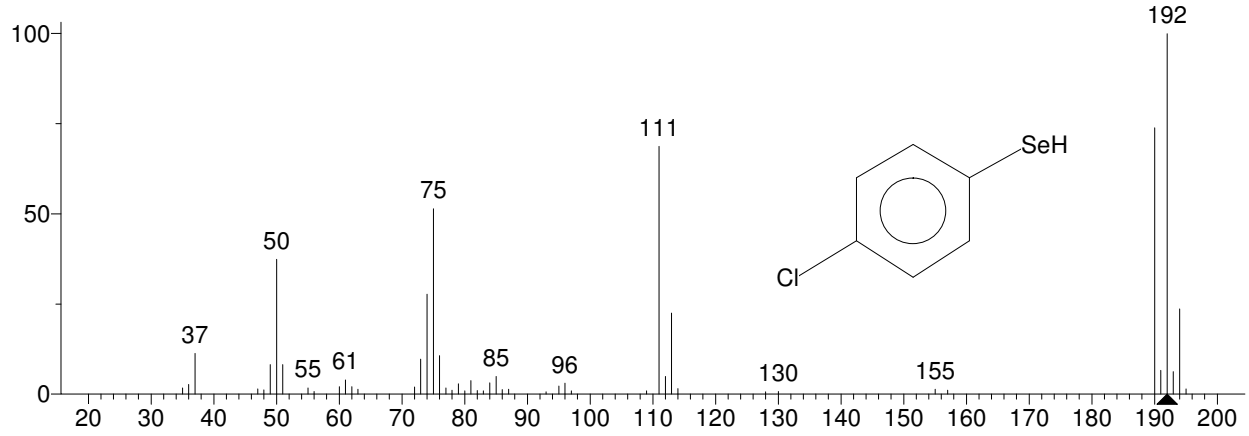
Unknown: Scan 720 (8.249 min): J5169HS.D
Compound in Library Factor = 177



Hit 1 : Benzene, 1-bromo-3-chloro-
C6H4BrCl; MF: 812; RMF: 846; Prob 33.6%; CAS: 108-37-2; Lib: replib; ID: 23330.

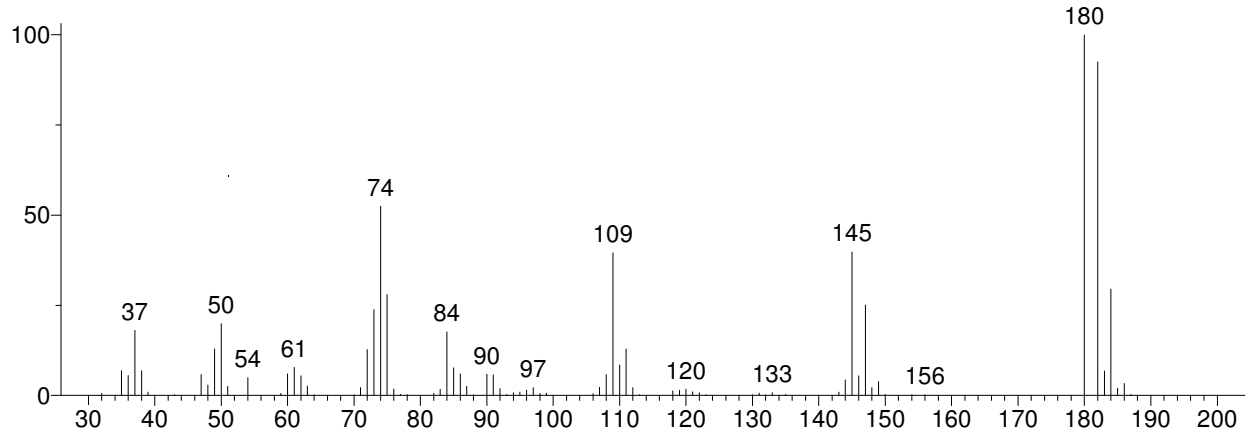


Hit 2 : 4-Chlorophenylselenol
C6H5ClSe; MF: 808; RMF: 853; Prob 28.4%; CAS: 16645-10-6; Lib: mainlib; ID: 120419.

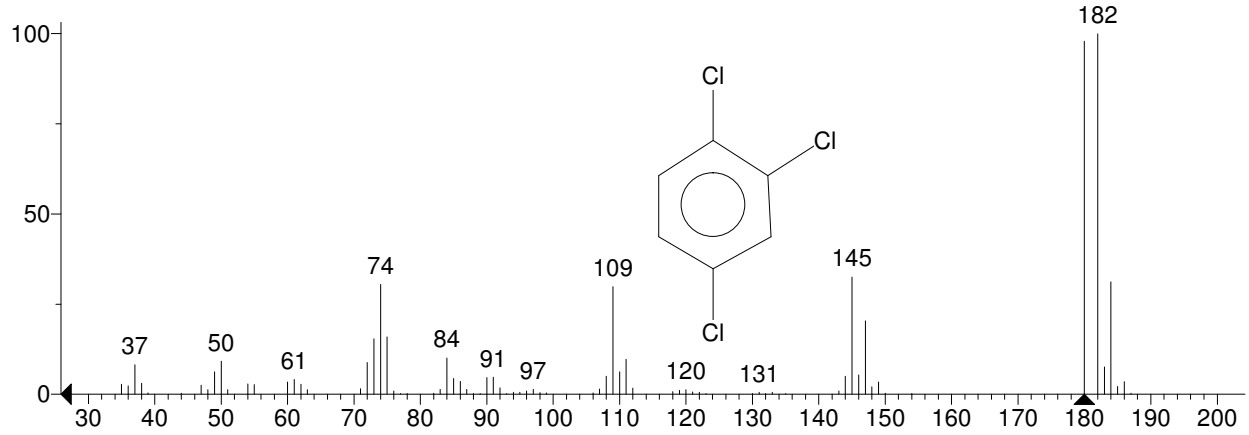


** Search Report Page 1 of 1 **

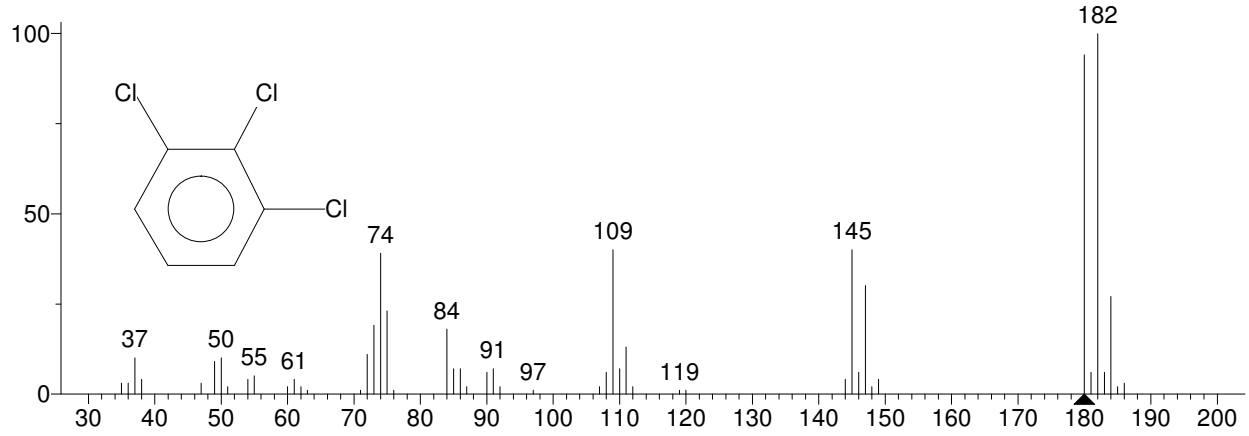
Unknown: Scan 788 (9.025 min): J5169HS.D
Compound in Library Factor = 1033



Hit 1 : Benzene, 1,2,4-trichloro-
C6H3Cl3; MF: 954; RMF: 954; Prob 45.7%; CAS: 120-82-1; Lib: replib; ID: 22722.

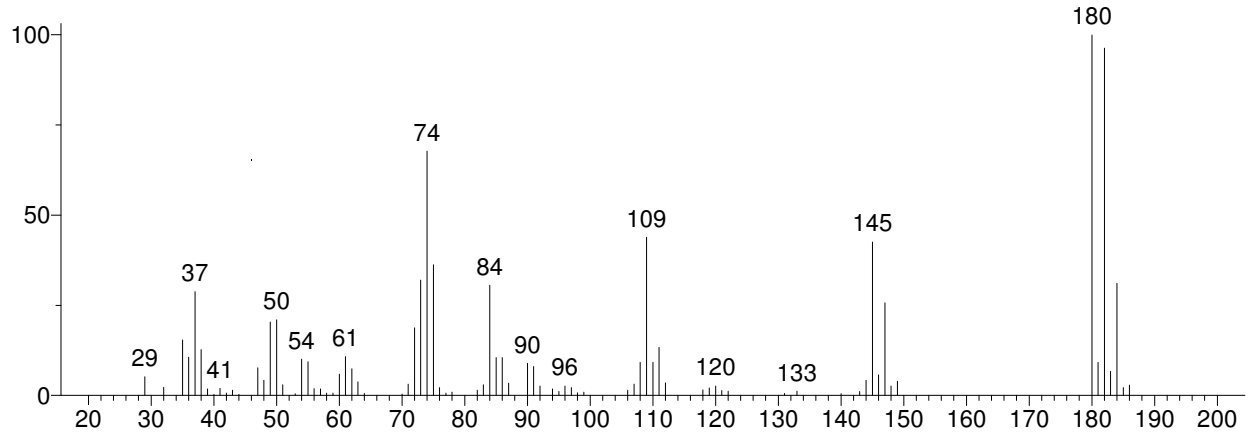


Hit 2 : Benzene, 1,2,3-trichloro-
C6H3Cl3; MF: 942; RMF: 953; Prob 30.4%; CAS: 87-61-6; Lib: replib; ID: 22721.

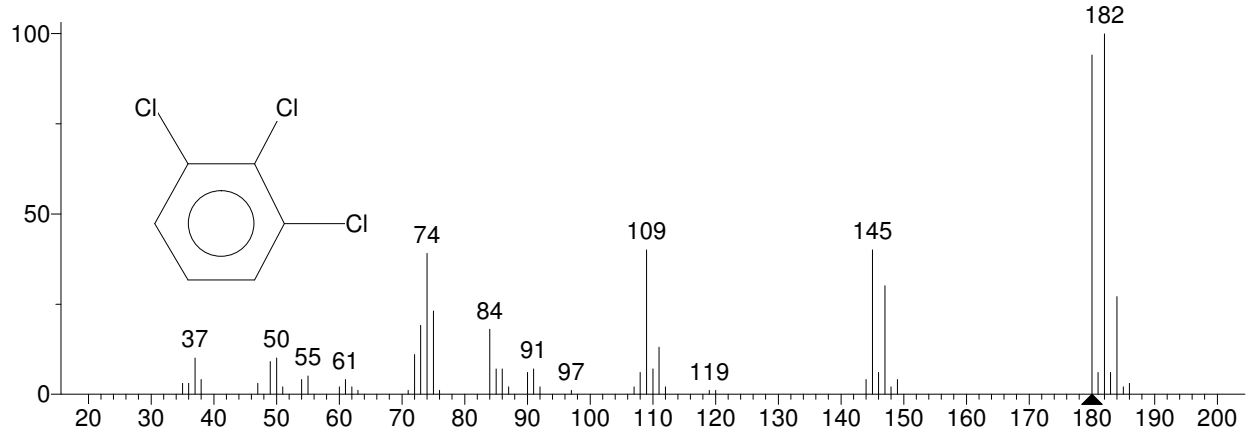


** Search Report Page 1 of 1 **

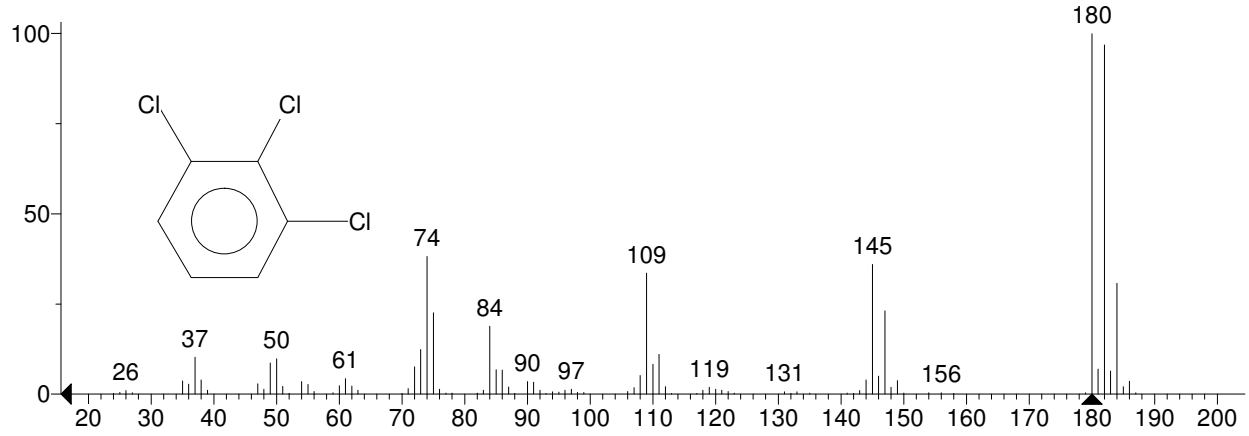
Unknown: Scan 819 (9.379 min): J5169HS.D
Compound in Library Factor = 889



Hit 1 : Benzene, 1,2,3-trichloro-
C6H3Cl3; MF: 933; RMF: 950; Prob 57.1%; CAS: 87-61-6; Lib: replib; ID: 22721.



Hit 2 : Benzene, 1,2,3-trichloro-
C6H3Cl3; MF: 933; RMF: 936; Prob 57.1%; CAS: 87-61-6; Lib: replib; ID: 22558.



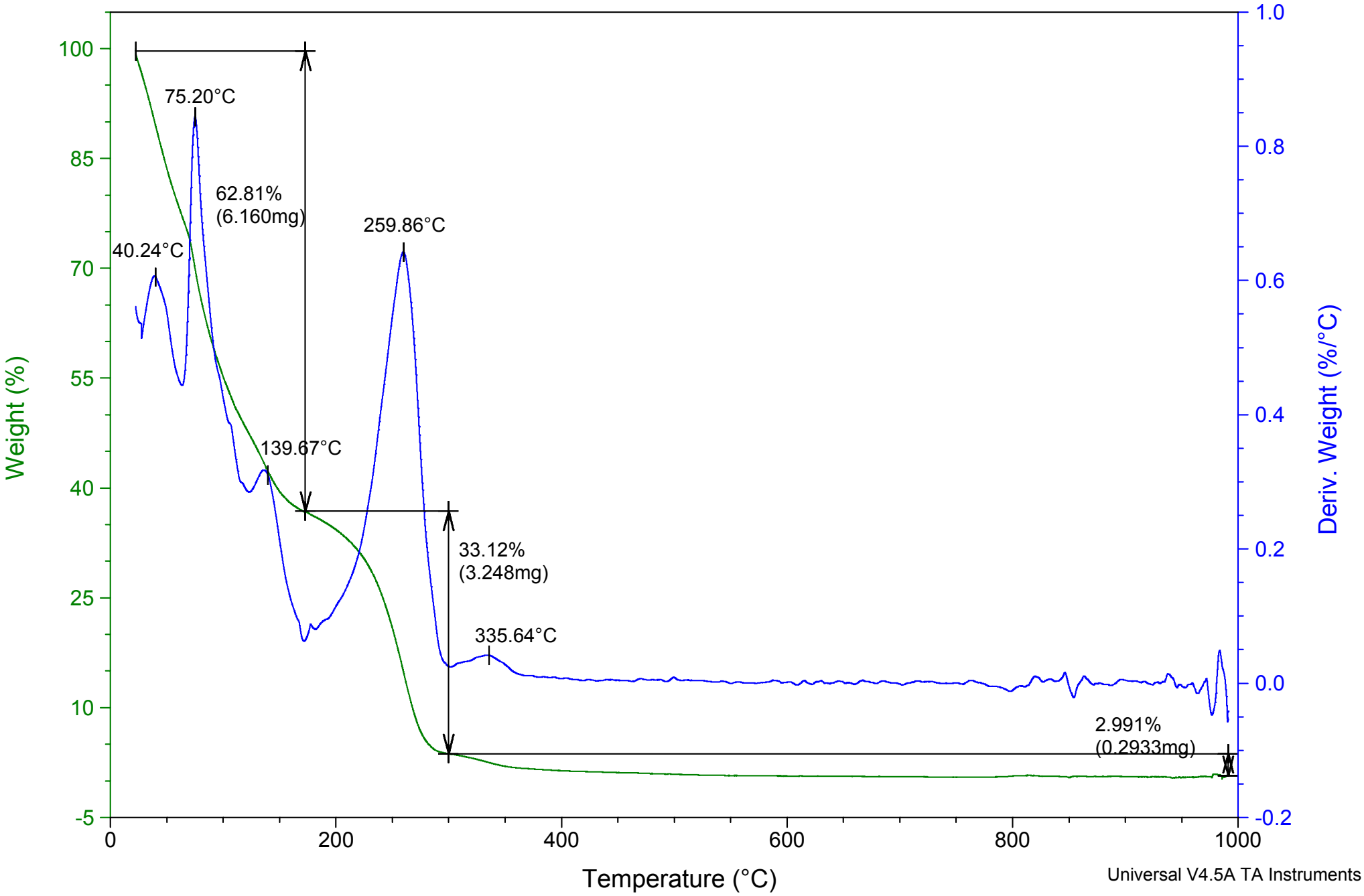
TGA Data

Sample: Jordi Unknown
Size: 9.8080 mg
Method: Custom Ramp

TGA

File: J:...\TGA Results\unknown.001

Run Date: 01-Oct-2010 14:40
Instrument: TGA Q500 V20.8 Build 34

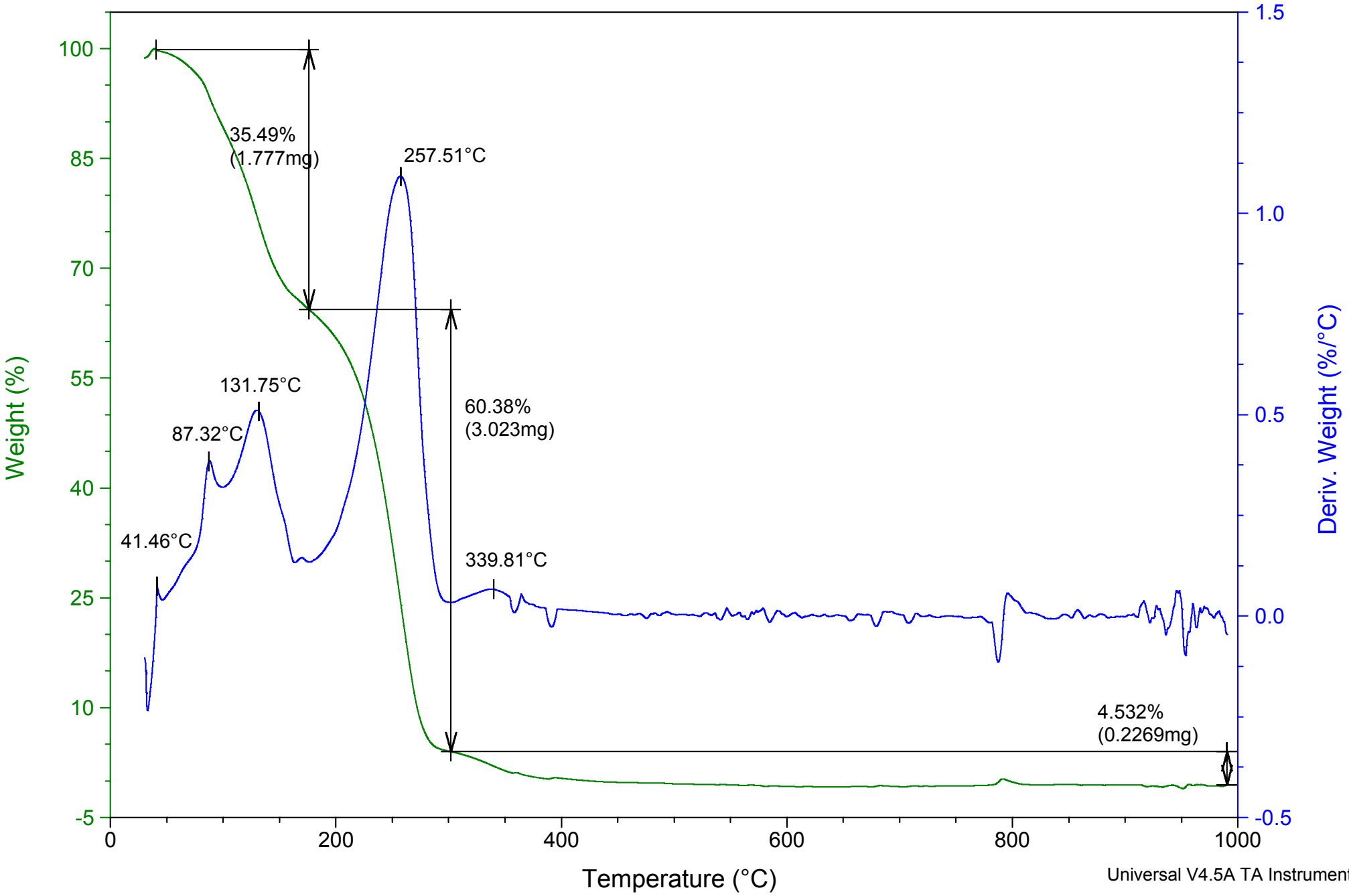


Sample: Jordi Unknown
Size: 5.0070 mg
Method: Custom Ramp

TGA

File: J:...\TGA Results\unknown.002

Run Date: 01-Oct-2010 18:29
Instrument: TGA Q500 V20.8 Build 34



PIXE RESULTS

Date: 10/14/2010
Run: 136
Job: 3628-10
Name: Jordi Labs LLC
Project:
Target: Unknown

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
* H ----------			12.050%	
* C ----------			71.769%	
* O ----------			15.930%	
Na	1.041	37.540 ppm		
Mg	1.254	13.640 ppm		
Al	1.485	7.382 ppm		
Si	1.740	4.675 ppm		
Phosphorus	2.010	5.197 ppm	123.998 ppm	3.348 ppm
S	2.308	3.158 ppm		
Chlorine	2.622	3.644 ppm	0.233%	0.002%
Potassium	3.314	1.827 ppm	10.050 ppm	1.357 ppm
Calcium	3.692	1.934 ppm	19.460 ppm	1.604 ppm
Sc	4.091	1.295 ppm		
Ti	4.511	1.443 ppm		
V	4.952	1.025 ppm		
Cr	5.415	0.679 ppm		
Mn	5.899	0.833 ppm		
Iron	6.405	0.963 ppm	7.916 ppm	0.841 ppm
Co	6.930	0.485 ppm		
Ni	7.478	0.315 ppm		
Copper	8.048	0.433 ppm	1.165 ppm	0.314 ppm
Zinc	8.639	0.559 ppm	5.487 ppm	0.586 ppm
Ga	9.250	0.391 ppm		
Ge	9.887	0.291 ppm		
As	10.544	1.131 ppm		
Se	11.222	0.794 ppm		
Bromine	11.924	1.188 ppm	6.408 ppm	1.089 ppm
Rb	13.395	0.793 ppm		
Sr	14.165	1.812 ppm		
Y	14.959	1.313 ppm		
Zr	15.775	2.732 ppm		
Nb	16.615	2.730 ppm		
Mo	17.480	2.285 ppm		
Tc	18.367	4.623 ppm		
Ru	19.279	4.753 ppm		
Rh	20.216	7.939 ppm		
Pd	21.176	9.207 ppm		
Ag	2.984	5.045 ppm		
Cd	3.133	3.954 ppm		
In	3.286	5.559 ppm		
Sn	3.444	3.911 ppm		
Sb	3.604	5.695 ppm		
Te	3.768	6.631 ppm		
I	3.937	3.410 ppm		
Cs	4.288	3.497 ppm		
Ba	4.466	4.761 ppm		
La	4.648	3.797 ppm		
Ce	4.841	3.000 ppm		
Pr	5.034	3.025 ppm		
Nd	5.230	3.635 ppm		
Pm	5.431	1.933 ppm		
Sm	5.632	2.164 ppm		
Eu	5.841	2.173 ppm		

Date: 10/14/2010
Run: 136
Job: 3628-10
Name: Jordi Labs LLC
Project:
Target: Unknown

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
Gd	6.050	1.951 ppm		
Tb	6.271	2.779 ppm		
Dy	6.492	3.356 ppm		
Ho	6.725	1.152 ppm		
Er	6.945	1.613 ppm		
Tm	7.182	1.160 ppm		
Yb	7.416	1.512 ppm		
Lu	7.655	1.078 ppm		
Hf	7.899	1.018 ppm		
Ta	8.149	1.200 ppm		
W	8.398	1.266 ppm		
Re	8.652	3.317 ppm		
Os	8.911	1.570 ppm		
Ir	9.174	1.128 ppm		
Pt	9.443	1.073 ppm		
Au	9.712	1.002 ppm		
Hg	9.989	1.333 ppm		
Tl	10.267	1.618 ppm		
Lead	10.551	2.946 ppm	10.890 ppm	2.208 ppm
Bi	10.838	1.710 ppm		
Th	12.968	3.235 ppm		
U	13.616	4.558 ppm		