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in analytical science*

COMMERCIAL-IN-CONFIDENCE

## **Comparative Analysis of Silicone Implant Samples**



MHRA

October 2012

LGCNW15655



Setting standards  
in analytical science

## Comparative Analysis of Silicone Implant Samples

[Redacted]

Medicines and Healthcare Products Regulatory  
Agency  
Floor 4-Y  
151 Buckingham Palace Road  
LONDON  
SW1W 9SZ

Prepared by:

[Redacted]

[Redacted]

Technical Approval:

[Redacted]

[Redacted]

Quality Approval:

[Redacted]

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Date: October 2012

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## **Executive Summary**

The aim of the study was to investigate potential chemical differences between the filler material present in silicone implant samples by a comparative analysis of a series of different batches of PIP material together with samples from several alternate suppliers of medical grade material.

The results show no significant differences in the bulk composition between the PIP samples and the medical grade materials.

The analysis does show significantly higher levels of extractable siloxane related species in the PIP samples when compared with the medical grade materials. There is no evidence of any other significant organic impurities in any of the samples tested.

The profile of extractable metals shows no significant differences between the PIP and the medical grade materials with the exception of Platinum where higher levels were found to be present in the medical grade material.

## 1. Background

To investigate potential differences between the filler material present in silicone implant samples. To include analysis of a series of different batches of PIP material plus samples from several alternate suppliers of medical grade material.

## 2. Samples Received

Customer Reference	Lot Ref	LGC Reference
PIP_IMGHC-TX-H-270	41609	15655-1
PIP_IMGHC-TX-H-130	02808	15655-2
PIP_IMGHC-TX-S-225	37908	15655-3
PIP_IMGHC-TX-H-270	27609	15655-4
PIP_IMGHC-TX-H-130	13606	15655-5
[REDACTED]		15655-6
[REDACTED]		15655-7
[REDACTED]		15655-8
[REDACTED]	[REDACTED]	15655-9
[REDACTED]	[REDACTED]	15655-10
[REDACTED]	[REDACTED]	15655-11

### 3. General Procedure

Samples of silicone implant material analysed as follows;

#### 3.1 Compositional Profile

##### 3.1.1 FTIR Spectroscopy

FTIR of the implant filler to profile the chemical composition.

##### 3.1.2 GC-MS

Samples of implant filler dissolved in ethyl acetate and analysed using GC-MS. Data screened against the NIST mass spectral library to profile the siloxane chain length distribution and identify any volatile organic additives and impurities by comparison of the experimental and library data.

#### 3.2 Extractable Profile

##### 3.2.1 GC-MS

Samples of implant filler extracted by reflux in hexane and then analysed using GC-MS to identify residual monomers, cyclic and linear oligo-siloxanes and other volatile low molecular weight (<1500 Daltons) organic components.

The following cyclic siloxanes are available as certified reference materials and were used to quantify the levels of each present in the sample extracts;

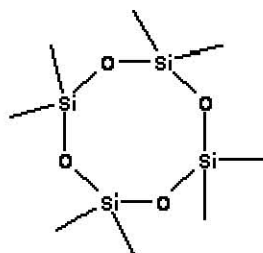
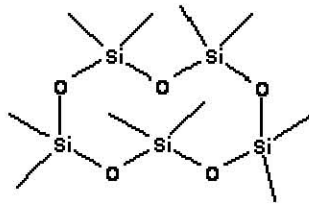
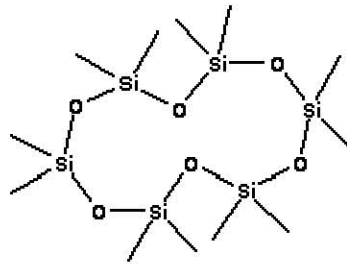


Figure 1 - Cyclotetrasiloxane, octamethyl- [D4]



**Figure 2 - Cyclopentasiloxane, decamethyl- [D5]**



**Figure 3 - Cyclohexasiloxane, dodecamethyl- [D6]**

### 3.2.2 Residue Analysis

The residue from the hexane extraction analysed by FTIR in order to profile the chemical composition.

### 3.2.3 Inorganic Analysis

Samples of implant filler extracted by reflux in 1%v/v nitric acid and then analysed using ICP-MS to quantify the elemental composition.

### 3.3 Abbreviations

FTIR – Fourier Transform Infra Red Spectroscopy

GC-MS – Gas Chromatography Mass Spectrometry

ICP-MS – Inductively Coupled Plasma Mass Spectrometry

NIST Library – Database of Mass Spectra compiled by the National Institute of Standards and Technology (NIST), US Department of Commerce.

The NIST library contains over 190000 reference spectra which are compared to experimental data using an algorithm to produce match factors. As a general guide match factors of greater than 90 is considered an excellent match, 80-90 a good match, 70-80 a reasonable match etc.

Match factors quoted are for information only. Positive confirmation of structure identity carried out by comparison of mass spectral and retention time data with a certified reference material.

## 4. Discussion

Raw data is presented in section 6.3.

### 4.1 FTIR

FTIR spectra of all samples (see figure 4) shows no significant differences between the raw filler material and the residue from the hexane extraction. Spectra are consistent with silicone with no significant evidence of any additional impurity. Spectral overlays (see figures 5 to 7) show no significant differences between the extracts and the residue samples.

### 4.2 GC-MS

See section 6.3.2 (Figures 8 to 34)

GC-MS shows significantly increased levels of low molecular weight siloxane related species in the PIP batches when compared with the medical grade samples (see table 1).

The hexane extract samples in all cases appear to show an increased range of similar siloxane related species when compared to the ethyl acetate extract samples.

The data shows no evidence of any significant additional organic impurities in any of the tested batches. The tables associated with the GC-MS chromatograms (see figures 10 to 31) shows results from a comparison of the mass spectral data with the NIST reference library (see section 3.3 for further detail).



Total levels of extractable siloxane species determined in the implant samples as follows;

Siloxane Levels [all ug/g sample]	LGC Reference						
	15655-1	15655-2	15655-3	15655-4	15655-5	15655-6	15655-9
<b>Total LOWER<sup>1,3</sup></b>	4800	3980	1680	3230	800	70	180
<b>Total UPPER<sup>1,3</sup></b>	13440	11140	4720	9060	2240	190	510
<b>D4<sup>2,4</sup></b>	270	100	270	180	40	20	50
<b>D5<sup>2,4</sup></b>	640	200	210	210	0	10	10
<b>D6<sup>2,4</sup></b>	710	10	170	380	40	20	10

**Table 1 – GC-MS quantitation of siloxane levels**

**Note 1:** Due to varying responses in peak areas for the individual siloxanes, it was not possible to give a definitive total concentration in each extract sample. The lower and upper response factors were taken to calculate the extreme case concentrations Total LOWER and Total UPPER.

**Note 2:** The levels of D4, D5 and D6 only make up a minor proportion of the total amount of siloxane species present in the extract samples. Figures 7 - 16 clearly shows the presence of a significant number of higher molecular weight siloxanes. Each species gives rise to a separate peak in the GC chromatogram (see figures 29 - 31). The identity of D4, D5 and D6 is confirmed by both the library match and the retention time vs. the individual reference standards (see figures 32 to 34).

**Note 3:** It should be noted that this is not a validated extraction method and is based on comparable extractions performed, giving comparable data. This may not equate to the total extractable.

**Note 4:** The levels quoted for the individual Siloxanes D4, D5 and D6 are based on response factors determined from the limited calibration data available.

## 4.3 ICP-MS

See section 6.3.3.

Several common elements such as Mg, K, Ti, Cr, Fe, Mn, Co, Ni, Cu were detected at trace levels (low ppm), see section 6.3.3 for more detail.

There were also some less common elements such as Sn, Cs, Pt, Au, Pb detected at low levels.

Although there were no major differences between PIP and the other samples there were some minor differences that may be significant:-

- Low level Cs (~0.3ppm) detected in all PIP samples but not present in any of the medical grade material.
- Lower Pt levels (~0.1ppm) were found in all PIP samples compared with the medical grade material (~3ppm). This may indicate different manufacturing conditions. If Pt is being used as a catalyst, variation in catalyst grade/quantity may impact the polymerisation process and the distribution of molecular weights in the silicone.
- For the remaining elements the differences in levels are considered to be insignificant in the context of this experiment as it stands.

## 5. Conclusions

FTIR shows no significant differences between the PIP samples and the medical grade materials.

GC-MS shows significantly higher levels (at least an order of magnitude) of extractable siloxane related species in the PIP samples when compared with the medical grade materials. There is no evidence of any other significant organic impurities in any of the samples tested.

ICP-MS shows levels of Pt in all samples with higher levels present in the medical grade material. There are low levels of a number of additional elements but no significant differences between the PIP and the medical grade materials.

## 6. Appendices

### 6.1 Sample / Standard Preparation

#### 6.1.1 GC-MS

1.0g sample extracted as follows;

- 10ml ethyl acetate (Analytical grade ex. Fisher Scientific) ultrasonic bath for 6 hrs.
- 10ml hexane (Pesticide grade ex. Fisher) reflux for 6 hrs.

#### 6.1.2 ICP-MS

0.1 - 0.2g sample + 8ml HNO<sub>3</sub> (trace analysis grade ex. Fisher Scientific) + 0.5ml H<sub>2</sub>O<sub>2</sub> (Aristar grade ex. BDH), microwave digested at 200°C using a CEM Discover/Explorer microwave digestion system.

The cooled solution was made up to 25 ml with water, followed by x10 dilution

#### 6.1.3 FTIR

Samples analysed as received.

### 6.2 Experimental Procedure

#### 6.2.1 GC-MS Method

Instrument	HP6890 /MSD Ref: SS/GCMS/1
Method	PIP_LL.M
Column	30m x 320µm x 0.25µm CP-SIL CD
Column reference	WCOT/350/L/21
Carrier	Helium at 1.0ml/min constant Flow
Oven temp	40°C hold for 2min ramp to 300°C at 20° C/min hold for 30min.
Injection mode	1µl Splitless@250°C
Detector	MSD Scan mode from 50-680 mass units
Data path	C:\MSDCHEM\1\DATA\

**Table 2 – GC-MS Method Parameters**

**Note.** The data is compared to NIST08 library. Match factors quoted are for information only. Positive confirmation of structure identity carried out by comparison of mass spectral and retention time data.

## 6.2.2 ICP-MS

Instrument	Agilent 7500 ICP-MS
Spray chamber	Quartz cooled to 2 deg C
RF power	1550 W
Plasma gas flow	15.0 l min <sup>-1</sup>
Carrier gas flow	1.0 l min <sup>-1</sup>
Make up gas flow	0.1 l min <sup>-1</sup>
Nebuliser type	Glass concentric
Sample uptake	80 s
Rinse time	90 s
Pump rate	0.3 rps (~0.5ml min <sup>-1</sup> )
No of replicates	3
Replicate time	0.3s mass <sup>-1</sup>

**Table 3 – ICP-MS Method Parameters**

## 6.2.3 FTIR

### SPECTROMETER DESCRIPTION:

Spectrometer: Nexus  
Source: IR  
Detector: DTGS TEC  
Smart Accessory ID: 070-12015  
Beamsplitter: KBr  
Sample spacing: 2.0000  
Digitizer bits: 20  
Mirror velocity: 0.6329  
Aperture: 100.00  
Sample gain: 4.0  
High pass filter: 20.0000  
Low pass filter: 11000.0000

### DATA COLLECTION INFORMATION:

Number of sample scans: 250  
Collection length: 185.6 sec  
Resolution: 8.000  
Levels of zero filling: 2  
Number of scan points: 4384  
Number of FFT points: 16384  
Laser frequency: 15798.3 cm<sup>-1</sup>  
Interferogram peak position: 2048  
Apodization: Happ-Genzel  
Phase correction: Mertz  
Number of background scans: 250  
Background gain: 4.0

## 6.3 Raw Data

### 6.3.1 FTIR

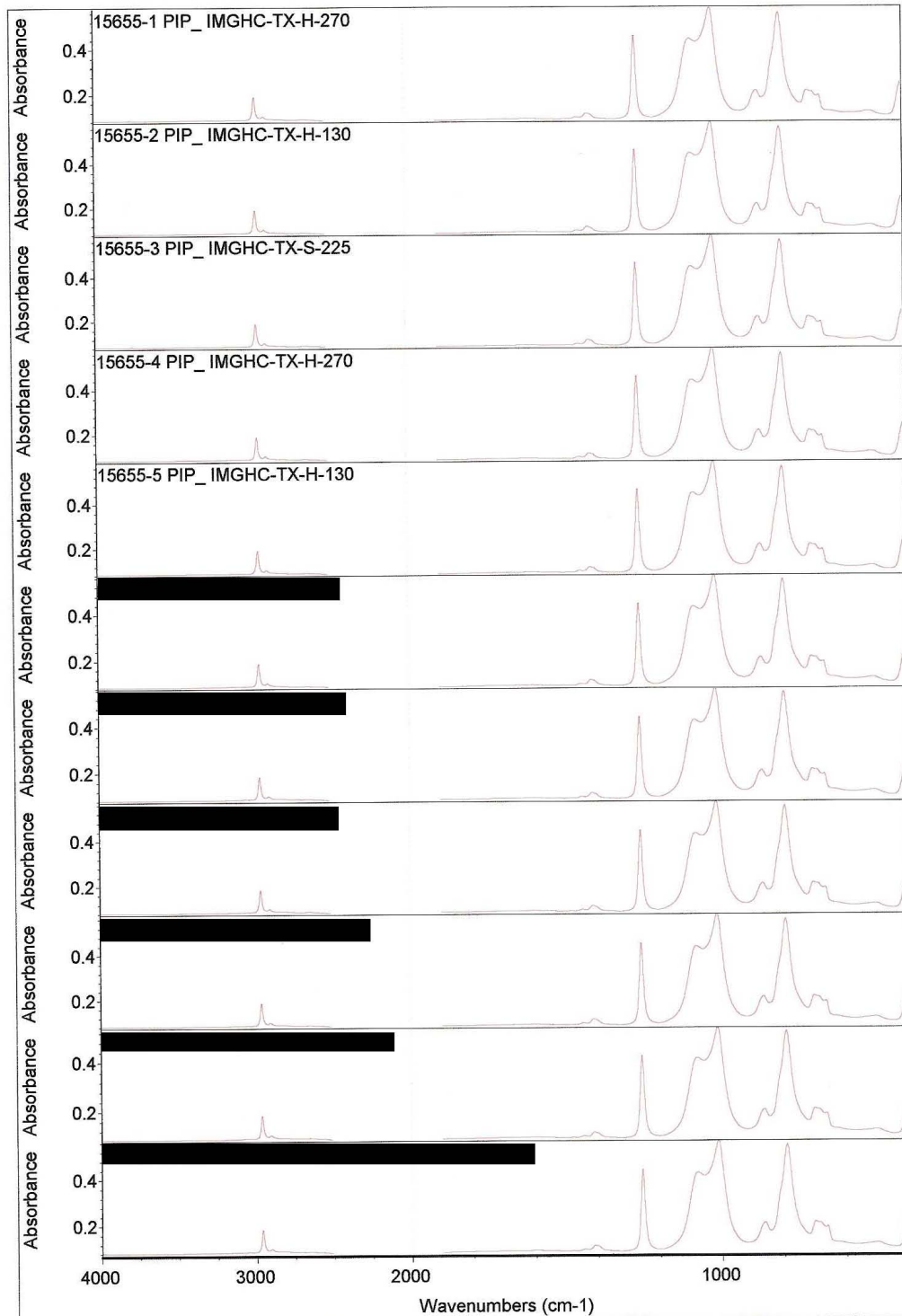


Figure 4 – FTIR spectra of silicone implant material samples

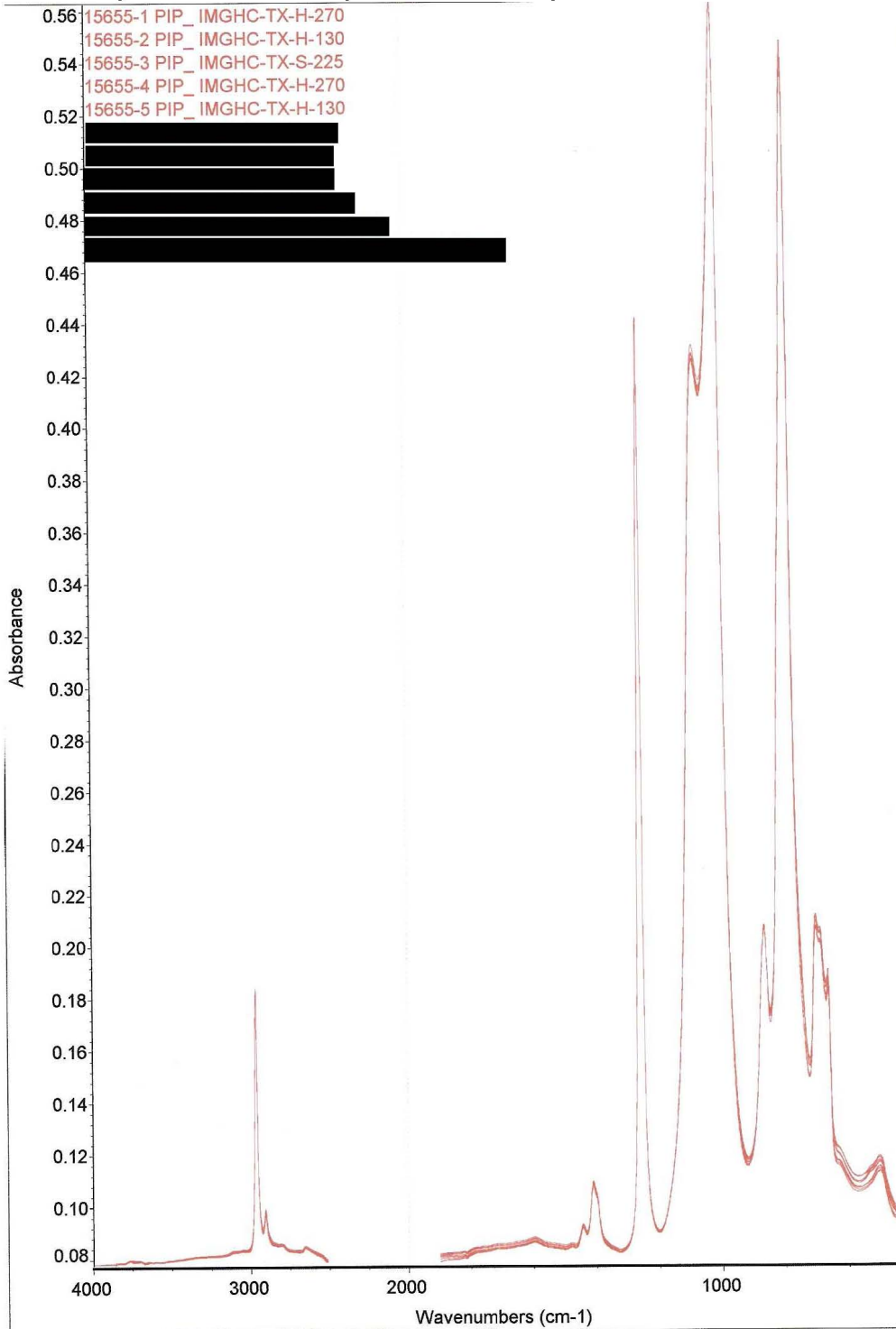
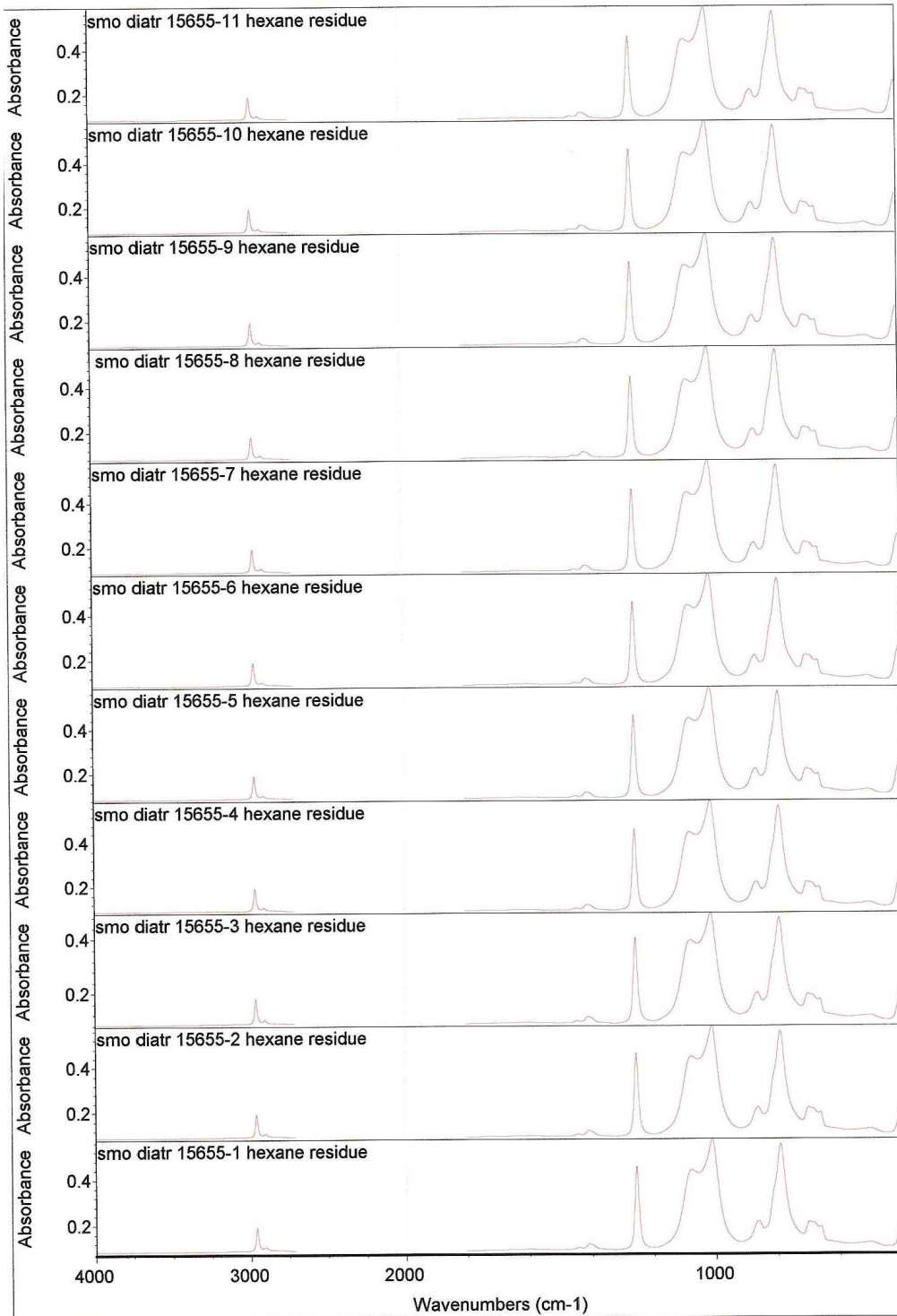
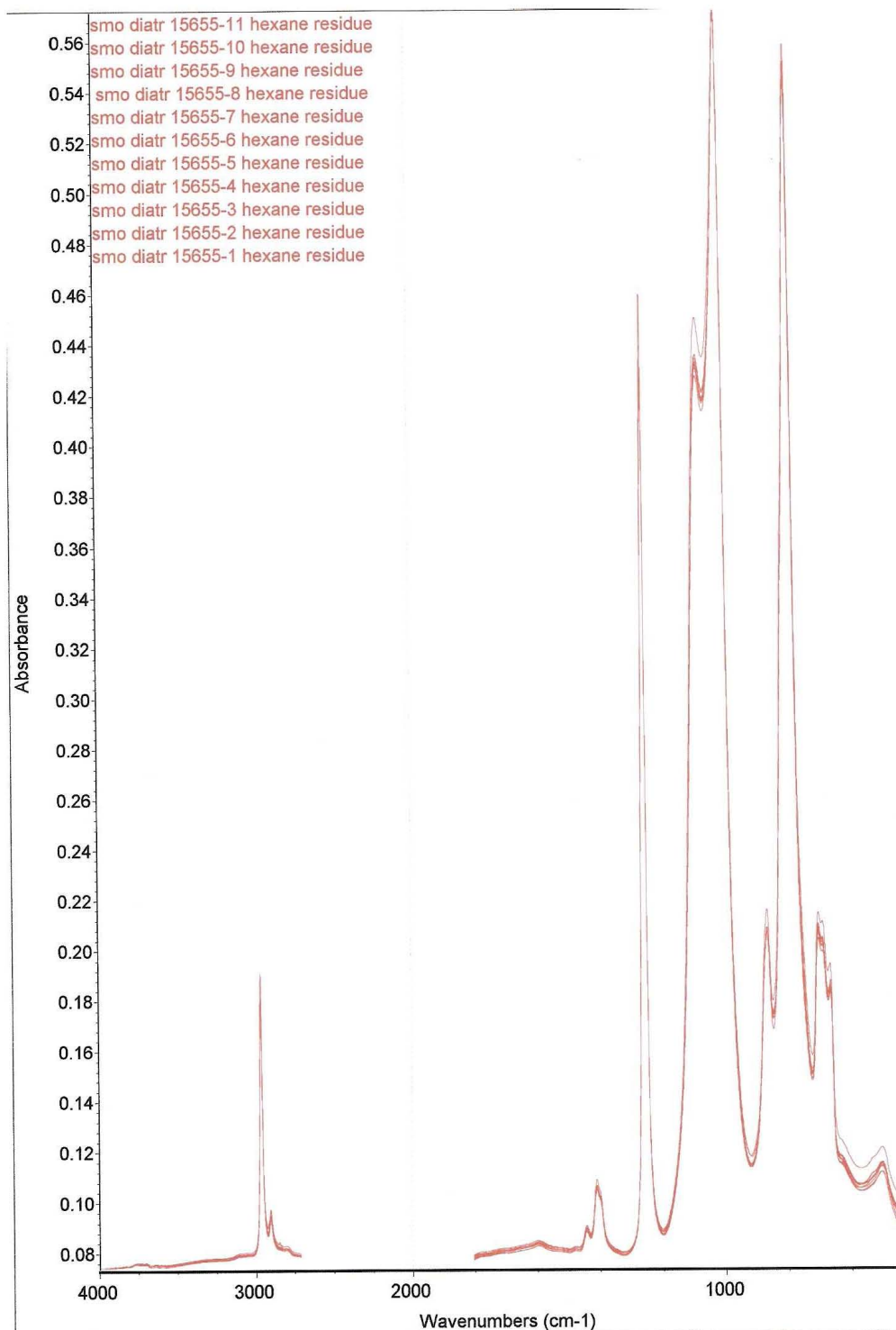


Figure 5 – FTIR spectral overlay of silicone implant material samples (no significant differences evident between any of the implant material samples)



**Figure 6 – FTIR spectra of silicone implant residue following hexane extraction (no significant differences evident between any of the residue samples)**



**Figure 7 – FTIR spectral overlay of silicone implant residue following hexane extraction (no significant differences evident between any of the residue samples)**



### 6.3.2 GC-MS

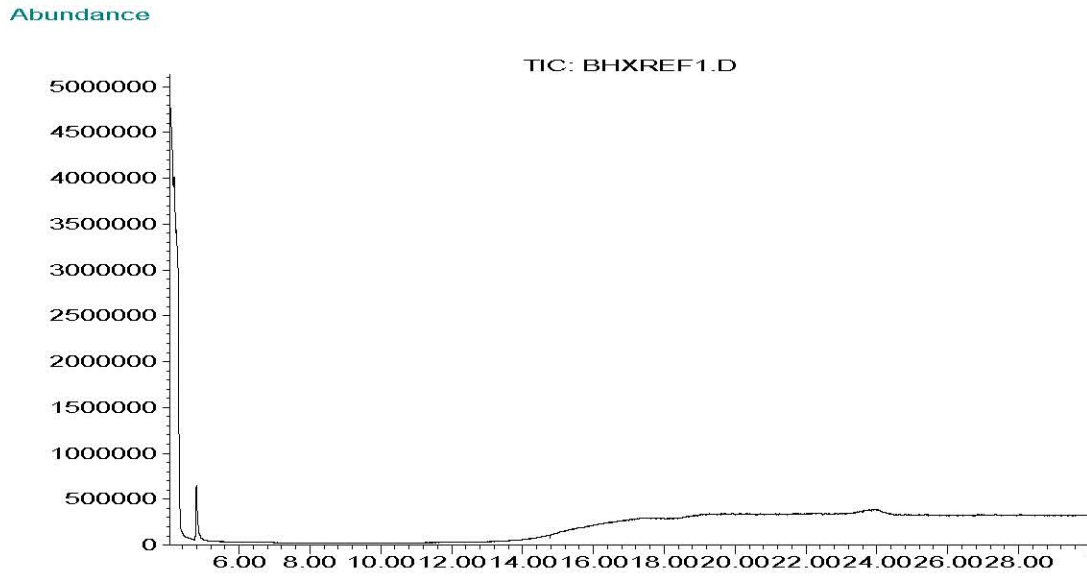


Figure 8 – GC-MS Chromatogram - Hexane Blank

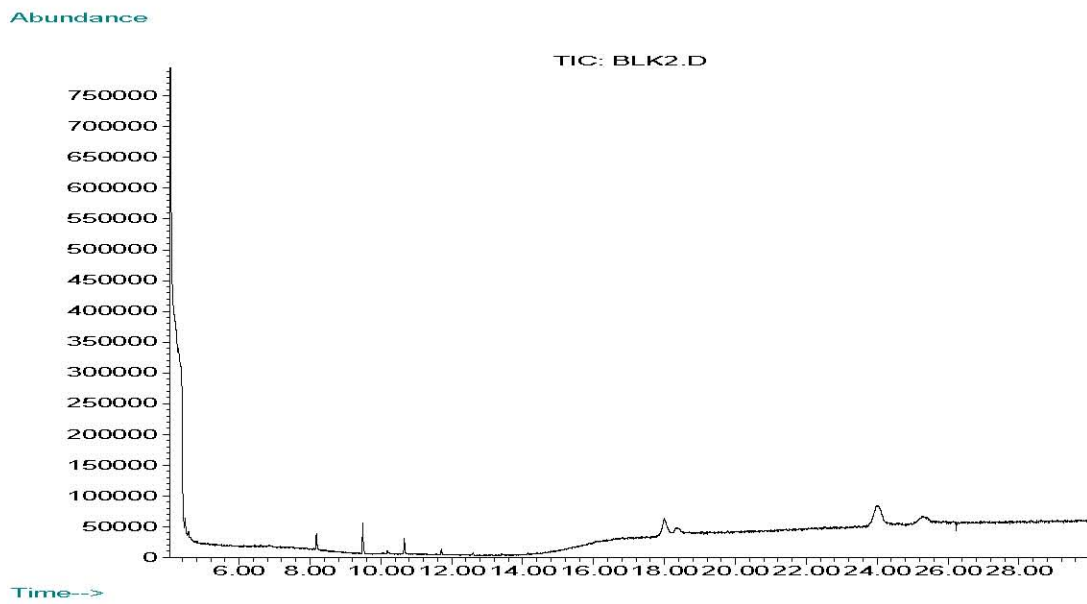
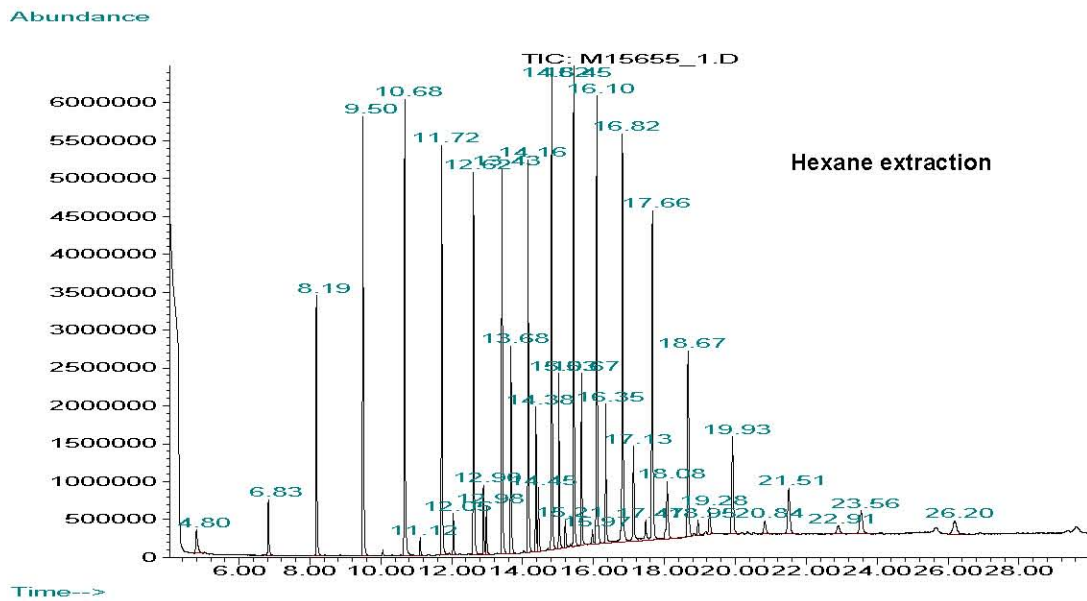
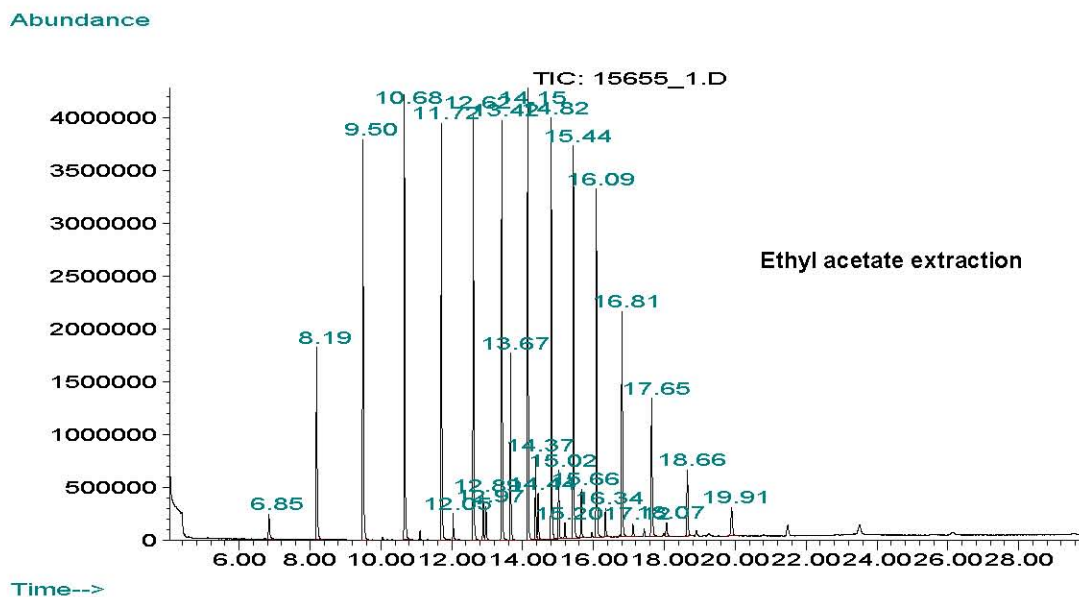


Figure 9 – GC-MS Chromatogram - Ethyl Acetate Blank



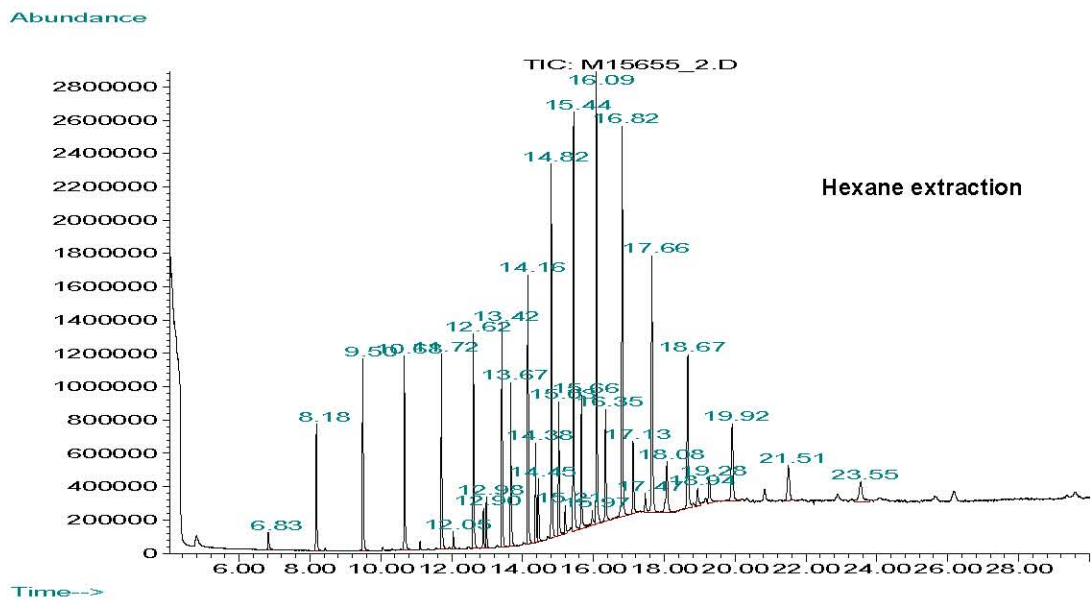
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
4.80	0.63	Octane	000111-65-9	80
6.83	0.81	Cyclotetrasiloxane, octamethyl-	000556-67-2	80
8.18	3.56	Cyclopentasiloxane, decamethyl-	000541-02-6	91
9.50	6.00	Cyclohexasiloxane, dodecamethyl-	000540-97-6	74
10.67	5.87	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
11.11	0.22	Trisiloxane, octamethyl-	000107-51-7	37
11.72	5.23	N-(Trifluoroacetyl)-N,O,O',O"-tet	000000-00-0	38
12.06	0.51	3,6-Dioxa-2,4,5,7-tetraoctane,	004342-25-0	40
12.62	5.14	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	32
12.90	0.91	9,12,15-Octadecatrienoic acid, 2,3	071579-69-6	30
12.99	0.62	Morphinan-6-one, 4,5-epoxy-3,14-di	000465-65-6	9
13.43	5.14	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	25
13.68	2.76	2-Methoxybenzoylformic acid, TMS	000000-00-0	27
14.16	5.46	Benzoic acid, 2,5-bis(trimethylsil	003618-20-0	38
14.38	1.73	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
14.46	0.89	Tricyclo[3.3.1.1 <sup>3,7</sup> ]decane, 2-brom	007314-85-4	25
14.83	5.96	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	33
15.03	2.07	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
15.21	0.39	Silane, (chloromethyl)dimethylphen	001833-51-8	47
15.44	6.68	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	30
15.66	2.09	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	35
15.97	0.24	Thieno[2,3-c]pyridine	000272-12-8	49
16.10	7.10	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	42
16.35	1.97	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
16.83	7.27	1-Monolinoleoylglycerol trimethyls	054284-45-6	30
17.13	1.65	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	25
17.47	0.5	Silanol, trimethyl-, pyrophosphate	018395-45-4	47
17.66	5.96	Ethanedioic acid, bis(trimethylsil	018294-04-7	32
18.09	1.49	Hexasiloxane, tetradecamethyl-	000107-52-8	47
18.67	3.92	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	43
18.95	0.33	1,2-Benzisothiazole-3-propanoic ac	050565-45-2	38
19.28	0.62	Mercaptoacetic acid, bis(trimethyl	006398-62-5	38
19.93	2.52	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
20.84	0.43	Mercaptoacetic acid, bis(trimethyl	006398-62-5	40
21.51	1.56	Silane, [(1-methyl-1,3-propanediyl	056771-47-2	27
23.56	1.02	2-Propenoic acid, 2,3,3-tris(trim	040333-10-6	35
26.20	0.77	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38

Figure 10 - 15655-1 GC-MS Chromatogram + NIST Library Search Report



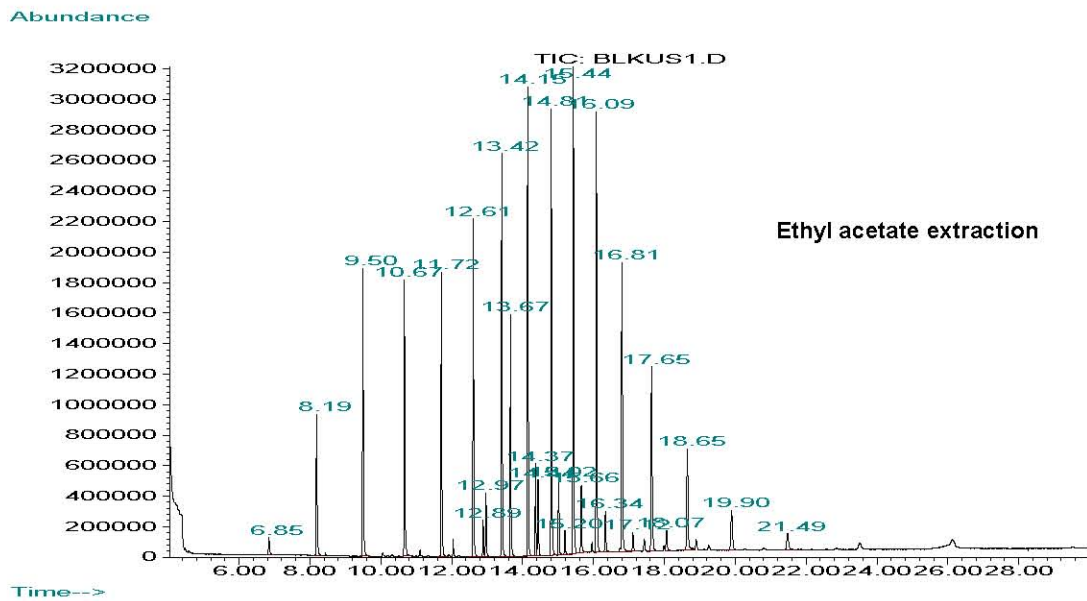
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.85	0.62	Cyclotetrasiloxane, octamethyl-	000556-67-2	87
8.19	4.44	Cyclopentasiloxane, decamethyl-	000541-02-6	90
9.50	8.06	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.68	8.29	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	38
11.72	7.49	N-(Trifluoroacetyl)-N,O,O',O'-tet	000000-00-0	35
12.05	0.45	Ethanedioic acid, bis(trimethylsil	018294-04-7	27
12.61	7.61	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	53
12.90	0.83	9,12,15-Octadecatrienoic acid, 2,3	071579-69-6	25
12.97	0.70	Silane, dimethyl(octadecyloxy)prop	065597-99-1	9
13.43	7.84	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	47
13.68	3.37	2-Methoxybenzoylformic acid, TMS	000000-00-0	25
14.16	8.11	Benzoic acid, 2,4-bis(trimethylsi	010586-16-0	32
14.38	1.37	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
14.44	1.02	Tricyclo[3.3.1.1 <sup>3,7</sup> ]decane, 2-brom	007314-85-4	47
14.81	8.19	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	38
15.02	1.32	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	27
15.20	0.35	Silane, (chloromethyl)dimethylphen	001833-51-8	38
15.44	8.22	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	37
15.66	1.02	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	25
16.09	7.19	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	36
16.34	0.61	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
16.81	5.51	1-Monolinoleoylglycerol trimethyls	054284-45-6	25
17.12	0.33	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	32
17.64	3.59	Ethanedioic acid, bis(trimethylsil	018294-04-7	32
18.07	0.41	1,2-Benzisothiazole-3-propanoic ac	050565-45-2	27
18.65	1.99	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	43
19.91	1.05	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	43

Figure 11 - 15655-1 GC-MS Chromatogram + NIST Library Search Report



Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.83	0.44	Cyclotetrasiloxane, octamethyl-	000556-67-2	80
8.18	2.37	Cyclopentasiloxane, decamethyl-	000541-02-6	91
9.50	3.78	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.67	3.53	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	53
11.11	0.22	Trisiloxane, octamethyl-	000107-51-7	37
11.72	3.43	N-(Trifluoroacetyl)-N,O,O',O"-tet	000000-00-0	38
12.06	0.33	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	27
12.62	3.87	3-Isopropoxy-1,1,1,7,7,7-hexamethy	071579-69-6	43
12.90	0.70	3-Isopropoxy-1,1,1,7,7,7-hexamethy	071579-69-6	28
12.99	4.44	Morphinan-6-one, 4,5-epoxy-3,14-di	000465-65-6	9
13.43	5.14	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
13.68	3.21	2-Methoxybenzoylformic acid, TMS	000000-00-0	38
14.16	5.37	Heptasiloxane, hexadecamethyl-	000541-01-5	32
14.38	1.74	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	27
14.45	1.25	Adenine	000073-24-5	37
14.82	6.47	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	36
15.02	2.28	3,6-Dioxa-2,4,5,7-tetra-sila-octane	004342-25-0	45
15.21	0.67	Silane, trichloro(2-tricyclo[3.3.1	037843-11-1	41
15.44	8.16	3-Isopropoxy-1,1,1,7,7,7-hexamethy	071579-69-6	38
15.66	2.53	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
15.98	0.57	1,2-Benzisothiazole-3-propanoic ac	000272-12-8	38
16.10	8.92	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	40
16.35	2.31	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	30
16.81	8.87	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	64
17.13	1.75	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	32
17.47	0.52	Silanol, trimethyl-, pyrophosphate	018395-45-4	25
17.66	5.96	Ethanedioic acid, bis(trimethylsil	018294-04-7	32
18.08	2.06	Hexasiloxane, tetradecamethyl-	000107-52-8	35
18.66	4.83	Ethanedioic acid, bis(trimethylsil	018294-04-7	38
18.95	0.61	Naphtho[1,2-b]furan-4,5-dione, 2-i	013019-42-6	20
19.28	1.02	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	35
19.91	3.03	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
21.51	1.57	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	43
23.54	1.46	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	27

Figure 12 - 15655-2 GC-MS Chromatogram + NIST Library Search Report

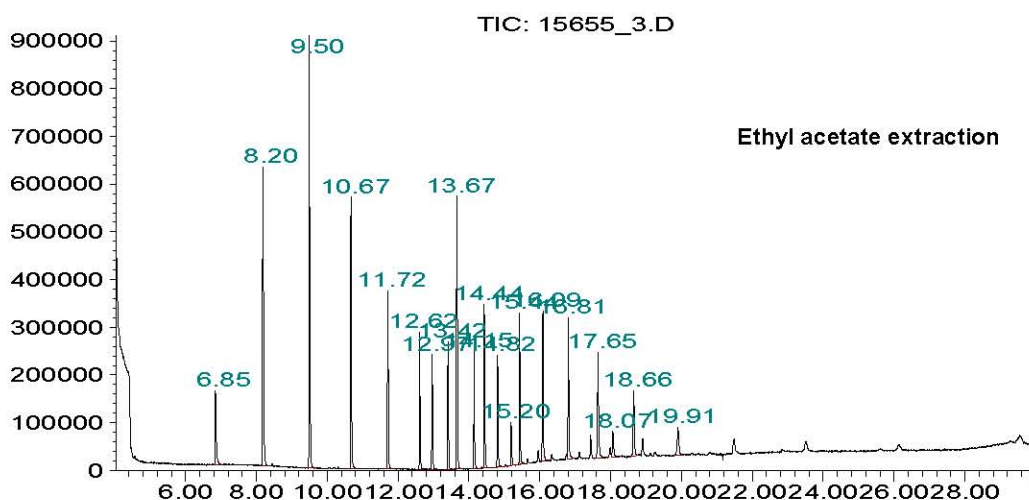


Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.85	0.39	Cyclotetrasiloxane, octamethyl-	000556-67-2	80
8.19	3.16	Cyclopentasiloxane, decamethyl-	000541-02-6	91
9.50	5.47	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.67	5.17	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	25
11.72	5.08	N-(Trifluoroacetyl)-N,O,O',O'-tet	000000-00-0	43
12.61	5.83	Ethanedioic acid, bis(trimethylsil	018294-04-7	43
12.90	0.63	Tris(trimethylsilyl)borate	004325-85-3	32
12.97	1.23	2-(4-(Dimethylamino)-1-naphthyl)na	000000-00-0	9
13.43	6.95	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	27
13.66	4.49	2-Methoxybenzoylformic acid, TMS	000000-00-0	43
14.16	8.04	Benzoic acid, 2,4-bis(trimethylsi	010586-16-0	32
14.38	1.45	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	32
14.44	1.02	Thymol	000089-83-8	28
14.81	8.98	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	37
15.02	1.47	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
15.20	0.51	3,4-Methylenedioxy-N-ethyl-N-methyl	000000-00-0	47
15.44	9.50	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	28
15.66	1.50	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
16.09	9.20	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	40
16.34	0.86	1-Monolinoleoylglycerol trimethyls	054284-45-6	28
16.81	7.20	Ethanedioic acid, bis(trimethylsil	018294-04-7	32
17.13	0.46	1-Monolinoleoylglycerol trimethyls	054284-45-6	64
17.64	4.72	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	56
18.07	0.62	1,2-Benzisothiazole-3-propanoic ac	050565-45-2	16
18.65	3.18	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	43
19.91	1.48	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
21.48	0.76	Tetrasiloxane, decamethyl-	000141-62-8	35

Figure 13 - 15655-2 GC-MS Chromatogram + NIST Library Search Report



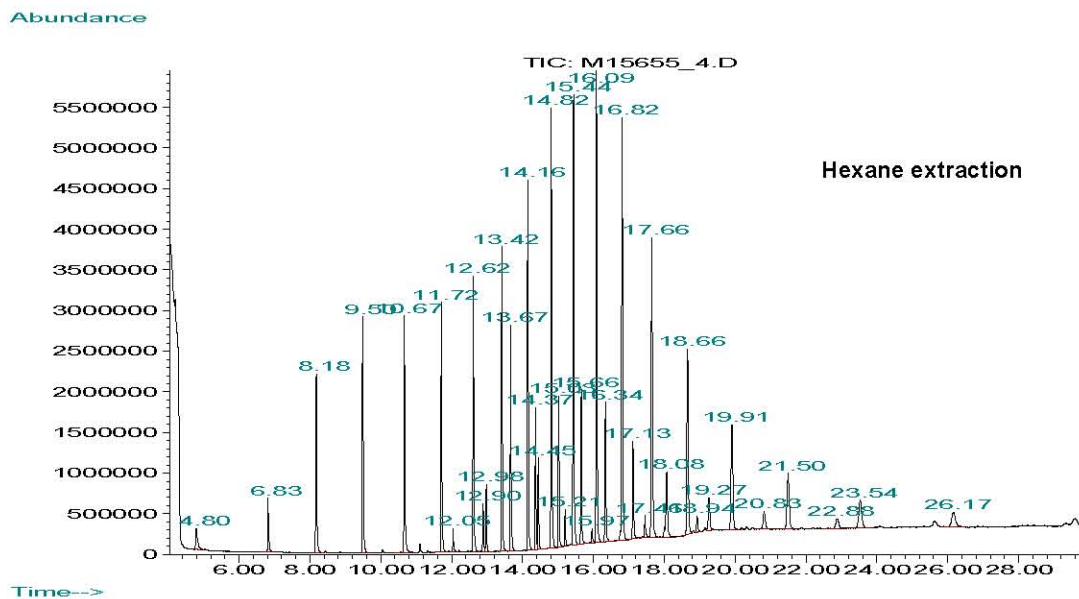
Abundance



Time-->

Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.85	3.14	Cyclotetrasiloxane, octamethyl-	000556-67-2	83
8.19	10.73	Cyclopentasiloxane, decamethyl-	000541-02-6	91
9.50	13.35	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.67	8.51	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	42
11.71	5.20	N-(Trifluoroacetyl)-N,O,O',O'-tet	000000-00-0	37
12.61	3.89	Ethanedioic acid, bis(trimethylsil	018294-04-7	32
12.98	3.19	12,13-Dihydro-12-methyl-13,14-diox	059050-19-0	10
13.41	3.37	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	38
13.67	8.96	Benzothiazole	000095-16-9	32
14.15	3.17	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
14.38	1.45	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	32
14.44	5.10	Thymol	000089-83-8	38
14.82	3.53	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	40
15.02	1.47	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
15.20	1.50	3,4-Methylenedioxy-N-ethyl-N-methyl	000000-00-0	47
15.44	4.47	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	37
15.66	1.50	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
16.09	5.27	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	50
16.34	0.86	1-Monolinoleoylglycerol trimethyls	054284-45-6	28
16.81	5.45	Ethanedioic acid, bis(trimethylsil	018294-04-7	47
17.13	0.46	1-Monolinoleoylglycerol trimethyls	054284-45-6	64
17.65	4.72	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	56
18.07	1.43	1,2-Benzisothiazole-3-propanoic ac	050565-45-2	37
18.66	3.15	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	42
19.91	1.88	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	32

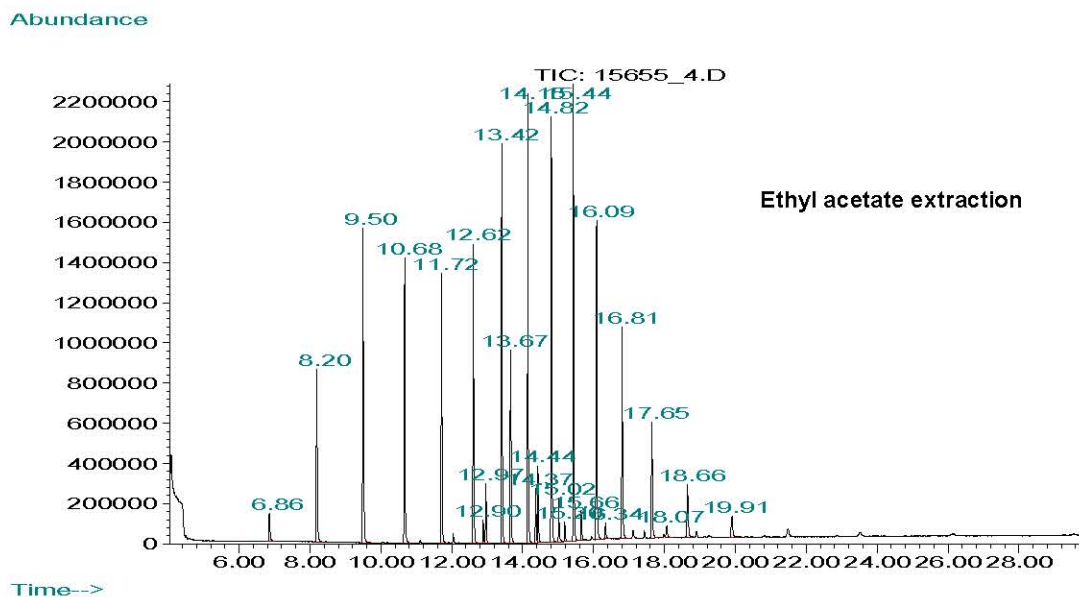
Figure 15 - 15655-3 GC-MS Chromatogram + NIST Library Search Report



Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
4.80	0.67	Octane	000111-65-9	86
6.83	0.89	Cyclotetrasiloxane, octamethyl-	000556-67-2	43
8.18	2.92	Cyclopentasiloxane, decamethyl-	000541-02-6	91
9.50	3.92	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.67	3.72	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	53
11.72	3.52	Benzoic acid, 2,4-bis(trimethylsilyloxy)-	010586-16-0	45
12.06	0.30	3,6-Dioxa-2,4,5,7-tetrasilaoctane, 1,1,1,5,5,5-hexamethyl-	004342-25-0	25
12.62	3.87	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)-	038147-00-1	43
12.90	0.67	9,12,15-Octadecatrienoic acid, 2,3	071579-69-6	27
12.97	0.95	Morphinan-6-one, 4,5-epoxy-3,14-dimethyl-	000465-65-6	9
13.43	4.46	Cholestane, 3-thiocyanato-, (3.alpha.)-	020997-49-3	90
13.68	3.28	2-Methoxybenzoylformic acid, TMS	000000-00-0	32
14.16	5.38	Benzoic acid, 2,5-bis(trimethylsilyloxy)-	003618-20-0	37
14.38	1.76	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	38
14.46	1.27	Glycine, N-(2-methoxybenzoyl)-, methyl ester	027796-49-2	23
14.83	6.44	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)-	038147-00-1	40
15.03	2.27	Hexasiloxane, tetradecamethyl-	000107-52-8	36
15.21	0.52	Benzene, (1-ethoxyethyl)-	003299-05-6	47
15.44	7.41	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)-	038147-00-1	25
15.66	2.34	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	35
15.97	0.28	Thieno[2,3-c]pyridine	000272-12-8	49
16.10	7.90	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)-	038147-00-1	32
16.35	2.13	3,6-Dioxa-2,4,5,7-tetrasilaoctane, 1,1,1,5,5,5-hexamethyl-	004342-25-0	38
16.83	7.79	1-Monolinoleoylglycerol trimethylsilyloxy-	054284-45-6	25
17.13	1.70	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	38
17.47	0.49	Silanol, trimethyl-, pyrophosphate	018395-45-4	47
17.66	6.50	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)-	038147-00-1	42
18.09	1.96	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	35
18.67	4.55	Heptasiloxane, hexadecamethyl-	000541-01-5	27
18.95	0.37	1,2-Benzisothiazole-3-propanoic acid	050565-45-2	35
19.28	0.95	Mercaptoacetic acid, bis(trimethylsilyloxy)-	006398-62-5	38
19.93	3.19	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	38
20.84	0.66	Mercaptoacetic acid, bis(trimethylsilyloxy)-	006398-62-5	22
21.51	2.09	Silane, [(1-methyl-1,3-propanediyl)dimethylsilyloxy]-	056771-47-2	35
22.89	0.47	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	25
23.56	1.36	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)-	038147-00-1	56
26.20	1.03	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	32

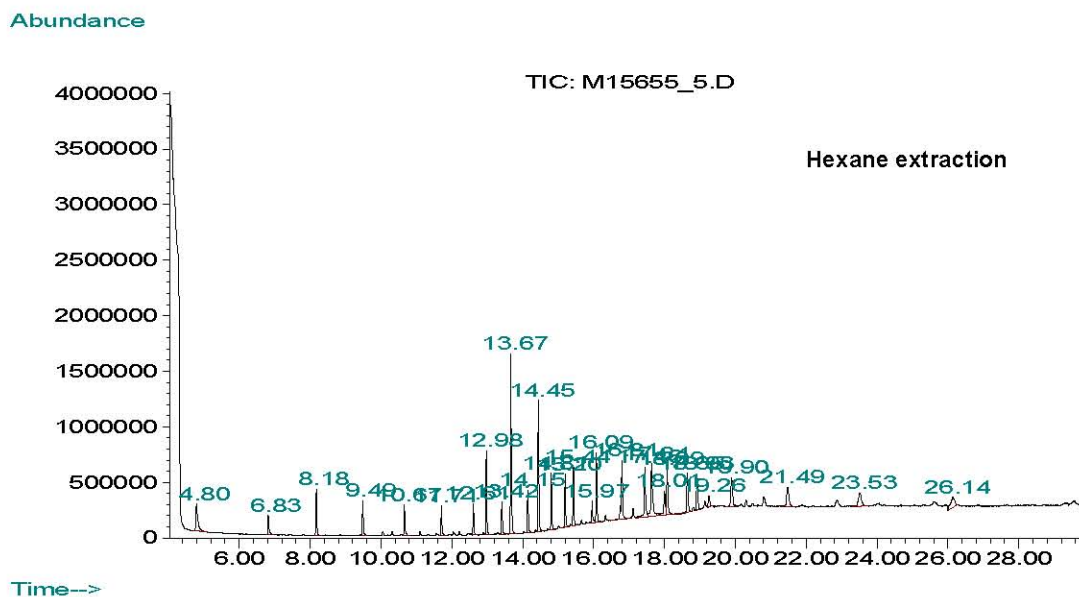
Figure 16 - 15655-4 GC-MS Chromatogram + NIST Library Search Report





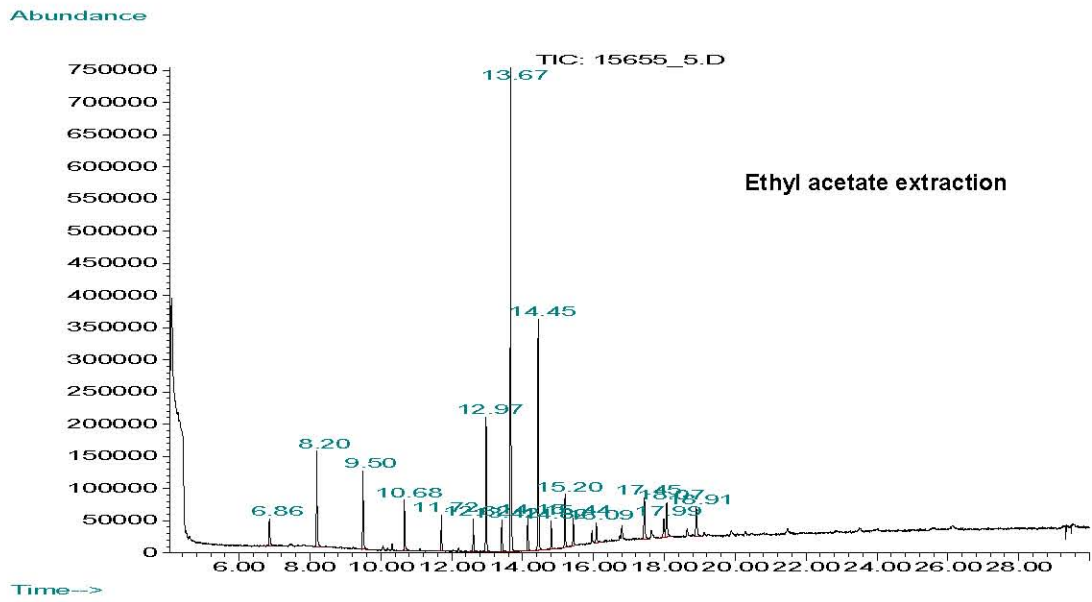
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.86	0.84	Cyclotetrasiloxane, octamethyl-	000556-67-2	86
8.19	4.36	Cyclopentasiloxane, decamethyl-	000541-02-6	90
9.50	7.12	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.67	6.38	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	47
11.71	5.73	Benzoic acid, 2,4-bis[(trimethylsilyl)oxy]phenyl-	73975 010586-16-0 40	40
12.61	6.39	Cyclohexasiloxane, dodecamethyl-	000540-97-6	43
12.90	0.44	Cyclohexasiloxane, dodecamethyl-	000540-97-6	25
12.98	1.25	12,13-Dihydro-12-methyl-13,14-diox	059050-19-0	10
13.41	7.80	Hexasiloxane, tetradecamethyl	000107-52-8	40
13.67	4.71	2-Methoxybenzoylformic acid, TMS	000000-00-0	40
14.15	8.83	1-Monolinoleoylglycerol trimethylsilyl ether	054284-45-6	40
14.37	1.07	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
14.44	1.73	Thymol	000089-83-8	38
14.82	9.78	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)propane	038147-00-1	38
15.03	1.04	1-Monolinoleoylglycerol trimethylsilyl ether	054284-45-6	28
15.20	0.48	Thiocyanic acid, phenyl ester	005285-87-0	38
15.44	9.82	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-1,3-dioxane	071579-69-6	38
15.66	0.76	Tris(trimethylsilyl)borate	004325-85-3	25
16.09	8.38	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsilyloxy)propane	038147-00-1	40
16.34	0.43	Tetrasiloxane, decamethyl-	000141-62-8	35
16.81	5.92	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	47
17.65	3.43	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	47
18.07	0.46	1,2-Benzisothiazole-3-propanoic acid	050565-45-2	38
18.66	1.88	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	38
19.91	0.97	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	37

Figure 17 - 15655-4 GC-MS Chromatogram + NIST Library Search Report



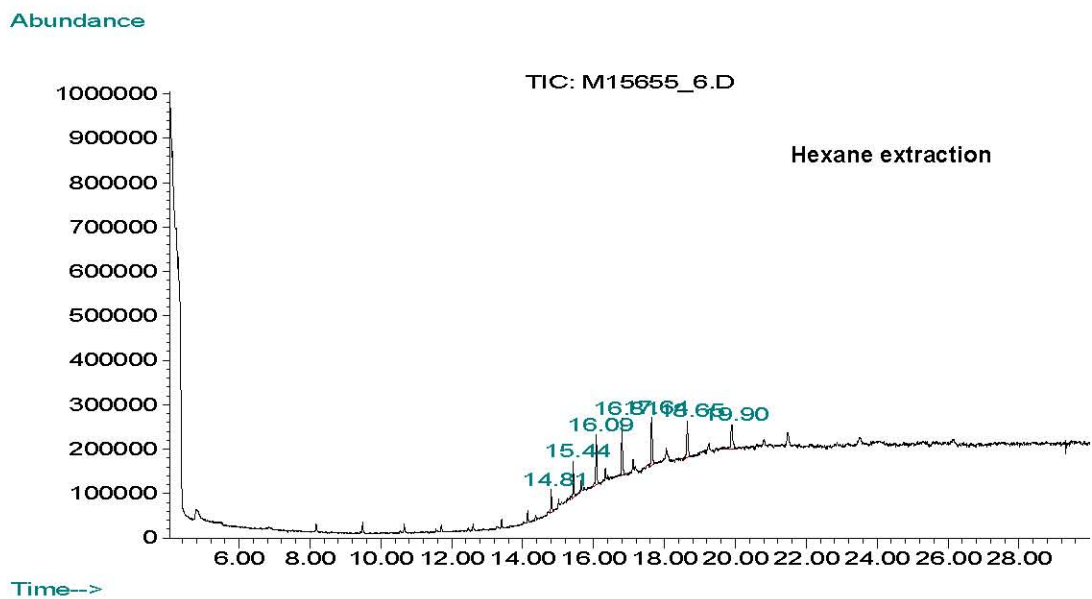
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
4.80	3.60	Octane	000111-65-9	78
6.83	1.45	Cyclotetrasiloxane, octamethyl-	000556-67-2	83
8.18	2.66	Cyclopentasiloxane, decamethyl-	000541-02-6	91
9.50	9.49	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.67	1.74	Tetrasiloxane, 3,5-diethoxy-1,1,1,	072439-78-2	35
11.72	1.60	Benzoic acid, 2,4-bis(trimethylsi	010586-16-0	32
12.62	3.87	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	56
12.90	0.67	9,12,15-Octadecatrienoic acid, 2,3	071579-69-6	27
12.98	4.55	Morphinan-6-one, 4,5-epoxy-3,14-di	000465-65-6	9
13.43	1.93	Ethanedioic acid, bis(trimethylsil	018294-04-7	43
13.68	11.40	2-Methoxybenzoylformic acid, TMS	000000-00-0	49
14.16	2.42	Benzoic acid, 2,5-bis(trimethylsil	003618-20-0	37
14.38	1.76	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
14.46	7.29	Glycine, N-(2-methoxybenzoyl)-, me	027796-49-2	23
14.81	3.10	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	45
15.21	2.93	Benzene, (1-ethoxyethyl)-	003299-05-6	50
15.44	3.75	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	33
15.96	1.52	Adenine	000073-24-5	47
16.08	4.16	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	38
16.81	5.30	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
17.45	4.49	Silanol, trimethyl-, pyrophosphate	018395-45-4	18
17.64	5.91	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
18.01	2.37	2,2'-Bipyridine	000366-18-7	25
18.09	1.96	Benzene, 1,1',1''-[1-(bromomethyl)	055282-37-6	35
18.65	3.27	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	47
18.92	3.23	1H-Pyrazole, 3,4-bis(trimethylsilyl	016037-45-9	38
19.26	1.15	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	22
19.90	3.26	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	37
21.49	2.91	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	42
23.53	3.00	3-Isopropoxy-1,1,1,7,7,7-hexamethy	071579-69-6	32
26.15	2.77	1-Monolinoleoylglycerol trimethyls	054284-45-6	25

Figure 18 - 15655-5 GC-MS Chromatogram + NIST Library Search Report



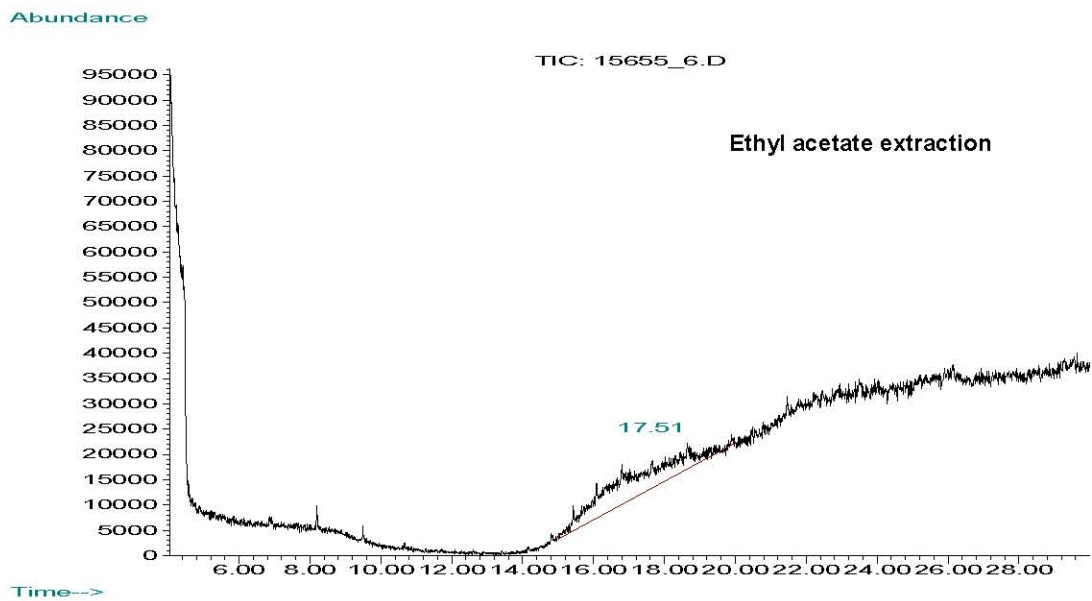
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.86	2.36	Cyclotetrasiloxane, octamethyl-	000556-67-2	59
8.20	6.54	Cyclopentasiloxane, decamethyl-	000541-02-6	83
9.50	4.91	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.68	2.99	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	58
11.72	2.09	Benzoic acid, 2,4-bis[(trimethylsilyl)amino]-	003618-20-0	43
12.61	1.73	4-Cyclohexene-1,2-dicarboxylic acid	106450-29-7	25
12.97	9.29	1,1'-Bicyclopentyl-2,2'-diol bis(trimethylsilyl) ether	073429-75-1	7
13.42	1.89	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-1,3,5-triazine	071579-69-6	56
13.67	28.83	Thieno[2,3-c]pyridine	000272-12-8	37
14.15	2.08	1-Monolinoleoylglycerol trimethylsilyl ether	054284-45-6	25
14.44	15.23	Thymol	000089-83-8	38
14.82	2.08	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-1,3,5-triazine	071579-69-6	50
15.20	0.48	Thiocyanic acid, phenyl ester	005285-87-0	33
15.44	1.97	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-1,3,5-triazine	071579-69-6	38
16.09	1.74	1-Monolinoleoylglycerol trimethylsilyl ether	054284-45-6	56
17.45	3.88	Cyclotrisiloxane, hexamethyl-	000541-05-9	11
18.00	1.88	Cyclotrisiloxane, hexamethyl-	000541-05-9	35
18.07	3.58	1,2-Benzisothiazole-3-propanoic acid	050565-45-2	25
19.91	3.21	1,2-Benzisothiazole-3-propanoic acid	050565-45-2	38

Figure 19 - 15655-5 GC-MS Chromatogram + NIST Library Search Report



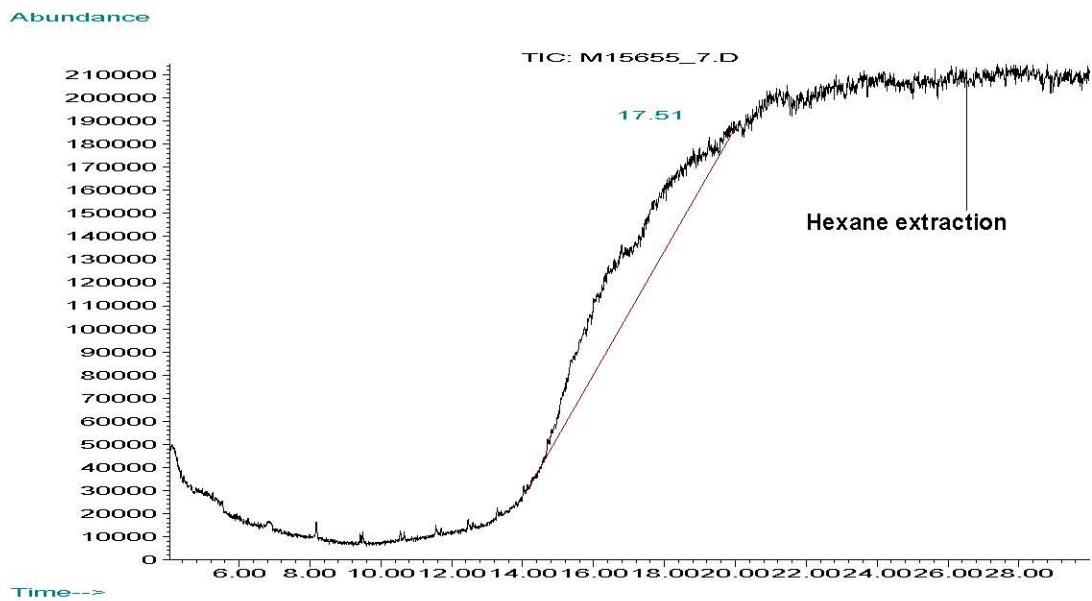
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
14.81	5.65	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	33
15.44	10.06	1,1,1,5,7,7,7-Heptamethyl-3,3-bis	038147-00-1	35
16.08	15.99	1-Monolinoleoylglycerol trimethyls	054284-45-6	28
16.81	18.12	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	23
17.64	18.32	1-Monolinoleoylglycerol trimethyls	054284-45-6	33
18.65	16.28	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	38
19.90	15.59	Trisiloxane, 1,1,1,5,5,5-hexamethy	003555-47-3	22

Figure 20 - 15655-6 GC-MS Chromatogram + NIST Library Search Report



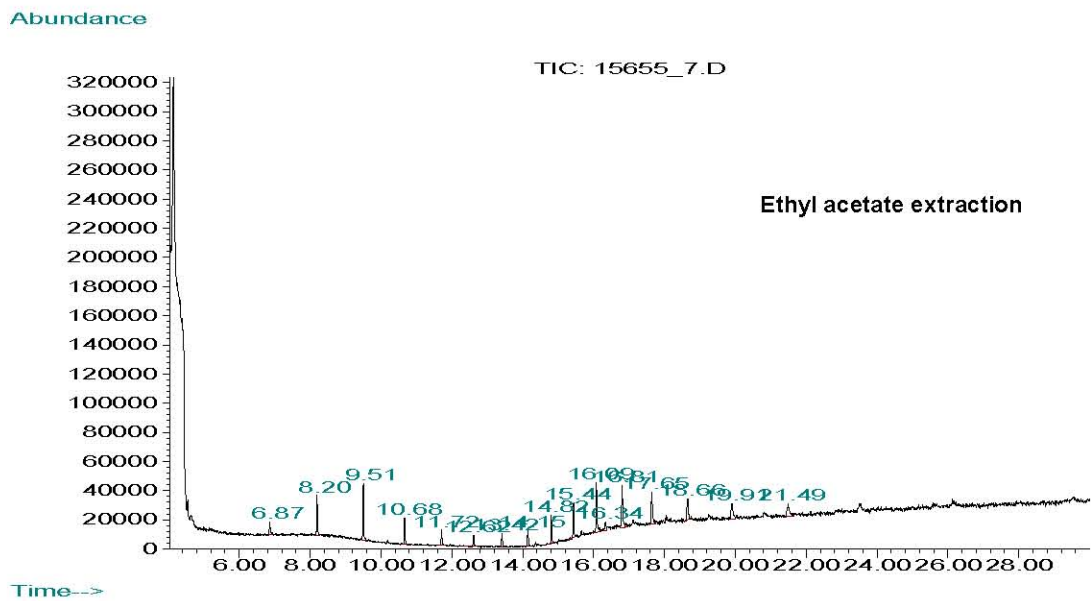
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	50

Figure 21 - 15655-6 GC-MS Chromatogram + NIST Library Search Report



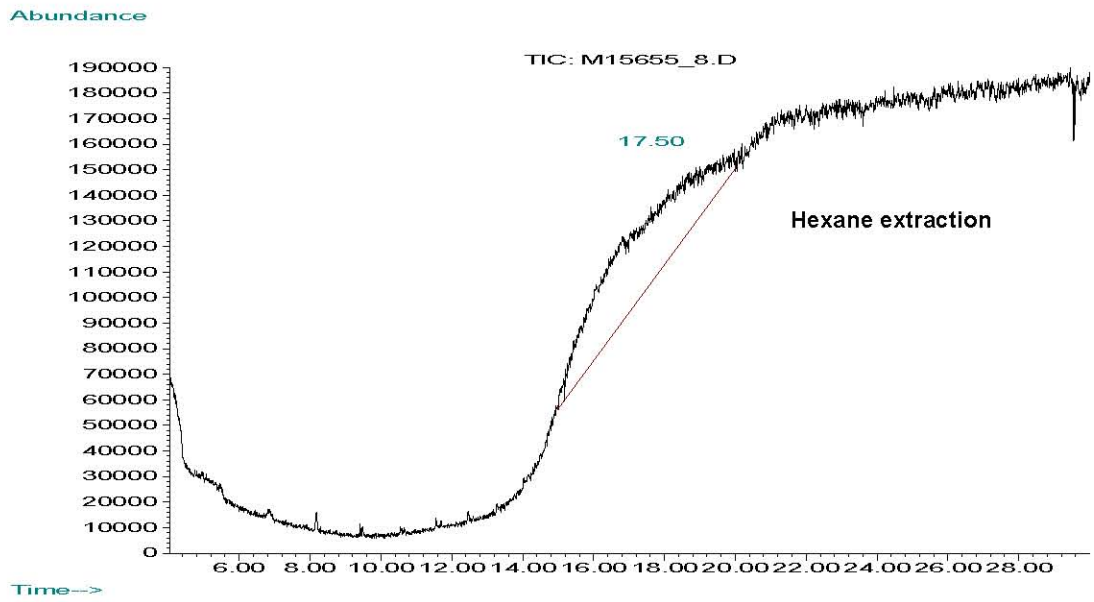
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	35

Figure 22 - 15655-7 GC-MS Chromatogram + NIST Library Search Report



Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.86	2.84	Cyclotetrasiloxane, octamethyl-	000556-67-2	14
8.20	7.39	Cyclopentasiloxane, decamethyl-	000541-02-6	47
9.50	9.77	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.68	4.77	Trisiloxane, 1,1,1,5,5,5-hexamethyl-	003555-47-3	13
11.72	2.72	Noradrenaline, tetra-TMS	000000-00-0	16
12.61	2.32	1,6-Anhydro-.beta.-D-glucofuranose	007425-74-3	9
13.42	2.44	2-Hexenedioic acid, bis(trimethyls	055494-10-5	28
14.15	3.31	2-Hexenedioic acid, bis(trimethyls	055494-10-5	10
14.82	4.33	Tetrasiloxane, decamethyl-	000141-62-8	38
15.44	6.38	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-	071579-69-6	50
16.09	10.61	1-Monolinoleoylglycerol trimethyls	054284-45-6	56
16.35	2.38	Cyclotrisiloxane, hexamethyl-	000541-05-9	47
16.81	11.47	Silane, trimethyl[5-methyl-2-(1-me	055012-80-1	47
17.65	9.21	1,1,1,3,5,5,5-Heptamethyltrisiloxa	001873-88-7	50
18.66	6.88	Tetrasiloxane, decamethyl-	000141-62-8	47
19.91	6.43	Cyclotrisiloxane, hexamethyl-	000541-05-9	50
21.49	3.21	Cyclotrisiloxane, hexamethyl-	000541-05-9	52

Figure 23 - 15655-7 GC-MS Chromatogram + NIST Library Search Report



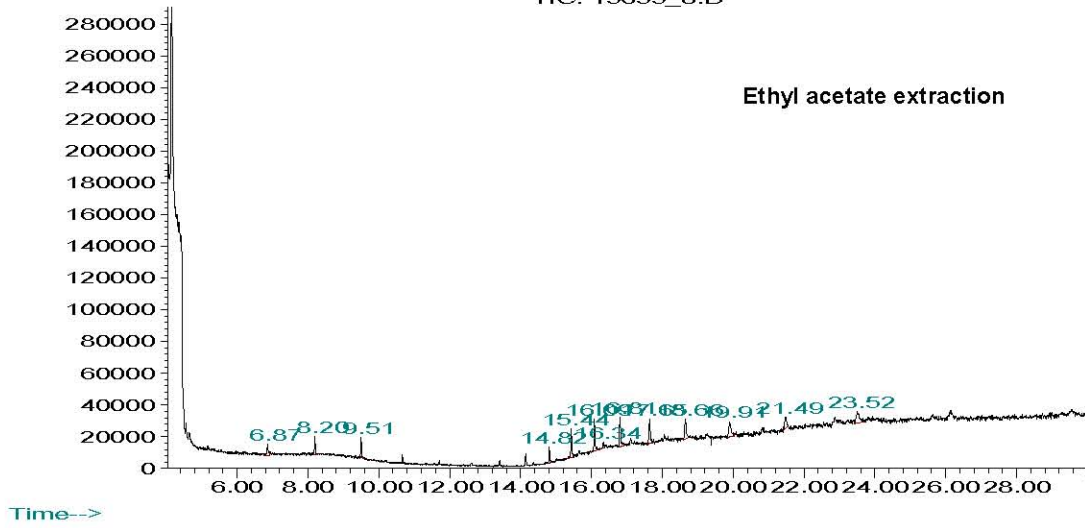
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	47

Figure 24 - 15655-8 GC-MS Chromatogram + NIST Library Search Report

Abundance

TIC: 15655\_8.D

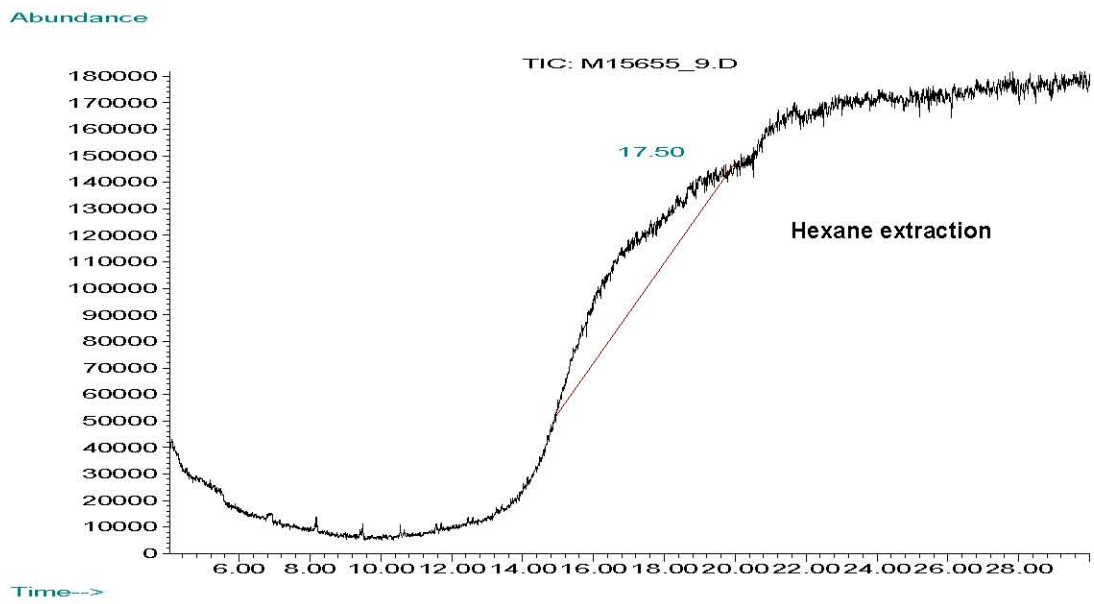
Ethyl acetate extraction



Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
6.86	4.69	Cyclotetrasiloxane, octamethyl-	000556-67-2	22
8.20	4.93	Cyclopentasiloxane, decamethyl-	000541-02-6	35
9.50	5.02	Cyclohexasiloxane, dodecamethyl-	000540-97-6	35
14.82	4.14	Tetrasiloxane, decamethyl-	000141-62-8	37
15.44	7.80	1,1,1,3,5,5,5-Heptamethyltrisiloxa	001873-88-7	28
16.09	10.71	1,1,1,3,5,5,5-Heptamethyltrisiloxa	001873-88-7	40
16.35	2.32	Cyclotrisiloxane, hexamethyl-	000541-05-9	47
16.81	11.84	Tetrasiloxane, decamethyl-	000141-62-8	50
17.65	10.04	Tetrasiloxane, decamethyl-	000141-62-8	47
18.66	9.50	Tetrasiloxane, decamethyl-	000141-62-8	47
19.91	10.07	Cyclotrisiloxane, hexamethyl-	000541-05-9	50
21.48	7.54	Cyclotrisiloxane, hexamethyl-	000541-05-9	50
23.51	11.41	Cyclotrisiloxane, hexamethyl-	000541-05-9	58

Figure 25 - 15655-8 GC-MS Chromatogram + NIST Library Search Report



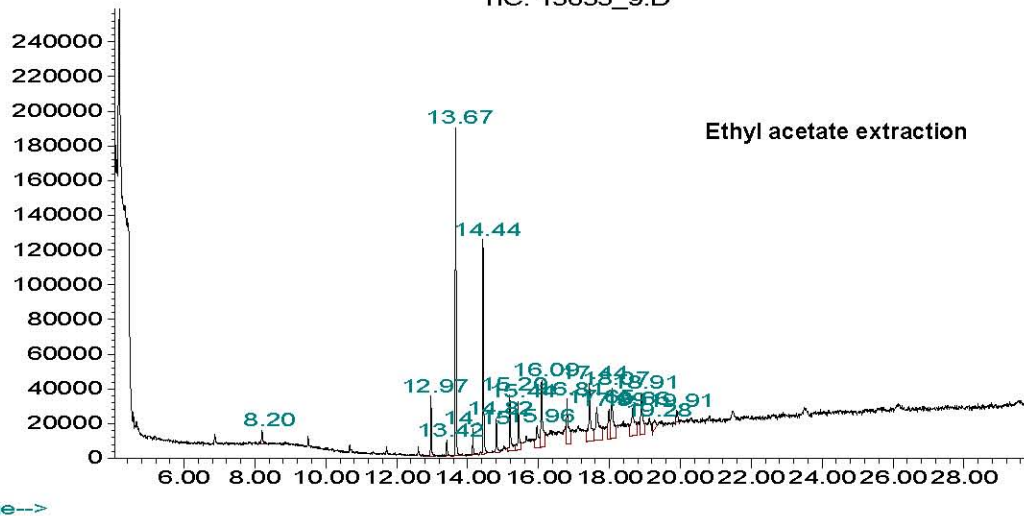


Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	46

Figure 26 - 15655-9 GC-MS Chromatogram + NIST Library Search Report

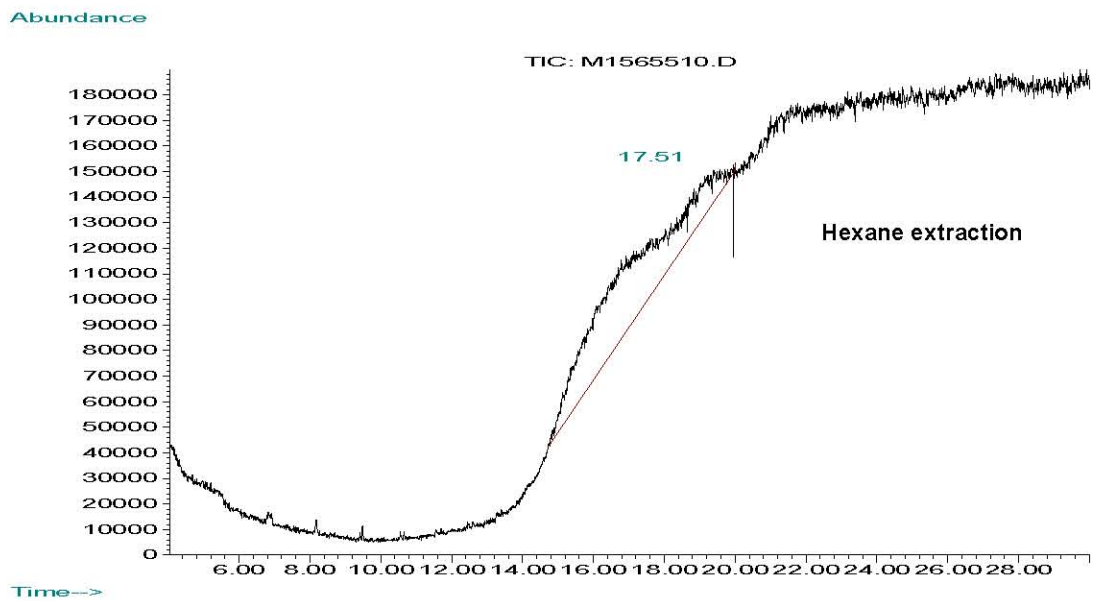
Abundance

TIC: 15655\_9.D



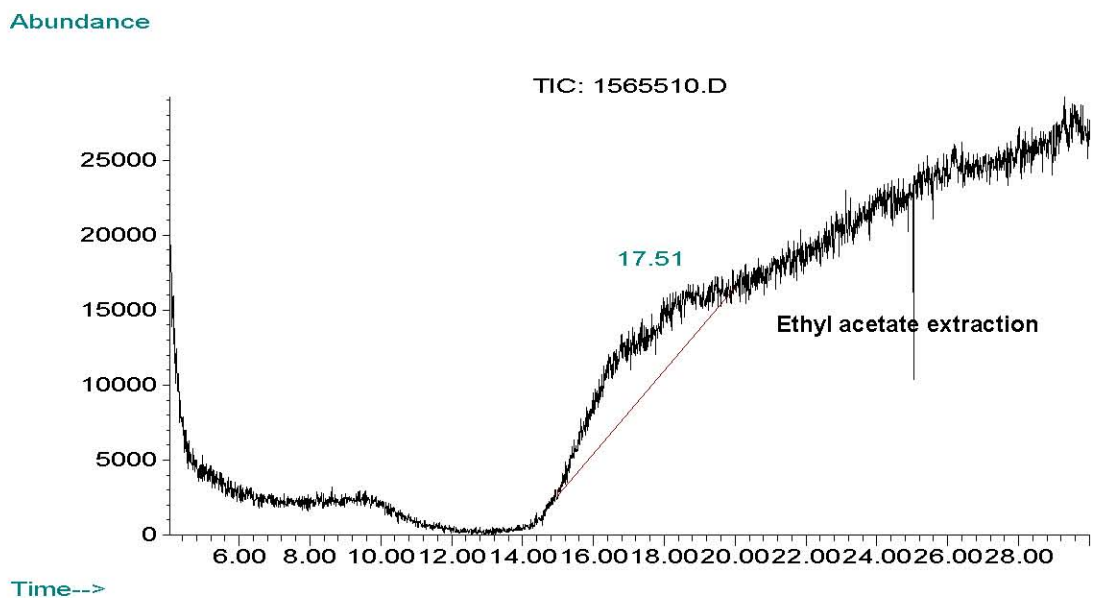
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
8.20	1.11	Cyclopentasiloxane, decamethyl-	000541-02-6	9
12.98	3.02	2,2'-Bipyridine	000366-18-7	9
13.43	0.77	Benzeneacetic acid, .alpha.-oxo-,	055517-36-7	9
13.67	16.83	Glycine, N-(2-methoxybenzoyl)-, me	027796-49-2	28
14.15	1.37	Mercaptoacetic acid, bis(trimethyl	006398-62-5	28
14.45	11.65	Thymol	000089-83-8	40
14.82	2.01	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	47
15.20	4.37	Benzamide, N-(1,1-dimethylethyl)-4	019486-73-8	47
15.44	3.83	1,1,1,3,5,5,5-Heptamethyltrisiloxa	001873-88-7	37
15.96	3.62	Silane, trimethyl[5-methyl-2-(1-me	055012-80-1	37
16.09	10.71	Tetrasiloxane, decamethyl-	000141-62-8	43
16.81	5.71	Tetrasiloxane, decamethyl-	000141-62-8	47
17.45	7.27	1H-Indole, 5-methyl-2-phenyl-	013228-36-9	35
17.65	7.52	Tetrasiloxane, decamethyl-	000141-62-8	47
17.99	3.31	Tetrasiloxane, decamethyl-	000141-62-8	46
18.08	6.82	1,2-Benzisothiazole-3-propanoic ac	050565-45-2	43
18.66	6.18	Tetrasiloxane, decamethyl-	000141-62-8	43
18.91	5.26	1,2-Benzisothiazole-3-propanoic ac	050565-45-2	47
19.28	1.93	Cyclotrisiloxane, hexamethyl-	000541-05-9	53
19.91	10.07	Cyclotrisiloxane, hexamethyl-	000541-05-9	52

Figure 27 - 15655-9 GC-MS Chromatogram + NIST Library Search Report



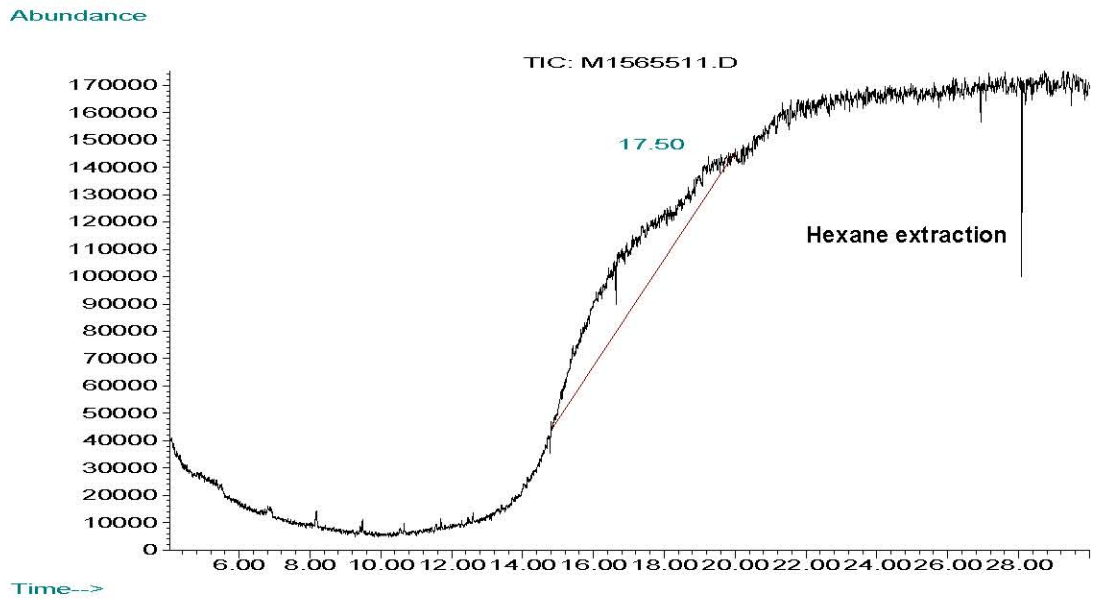
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	49

Figure 28 - 15655-10 GC-MS Chromatogram + NIST Library Search Report



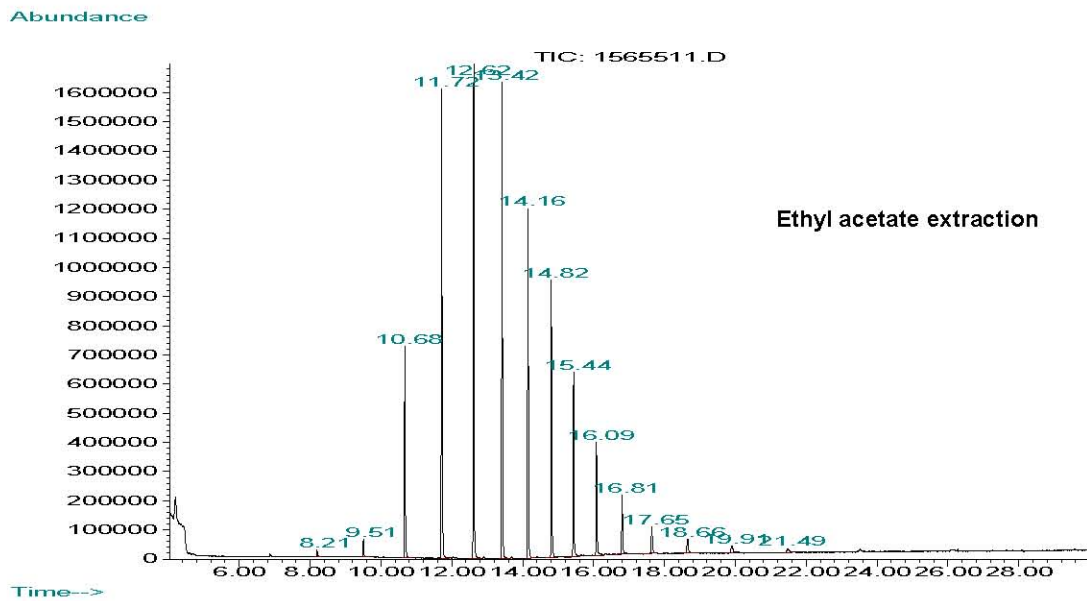
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	53

Figure 29 - 15655-10 GC-MS Chromatogram + NIST Library Search Report



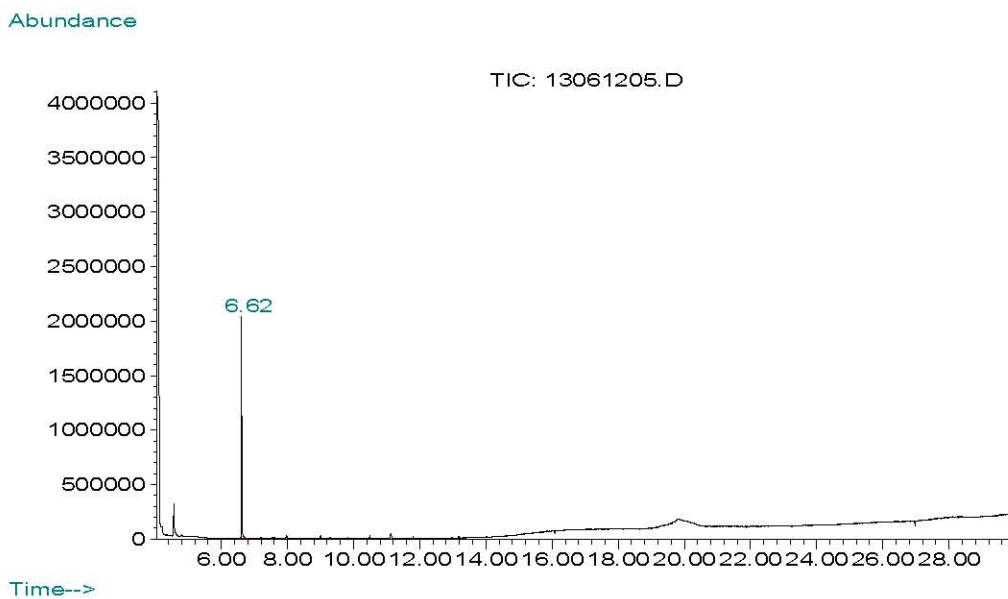
Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
17.51	100	Cyclotrisiloxane, hexamethyl-	000541-05-9	49

Figure 30 - 15655-11 GC-MS Chromatogram+ NIST Library Search Report

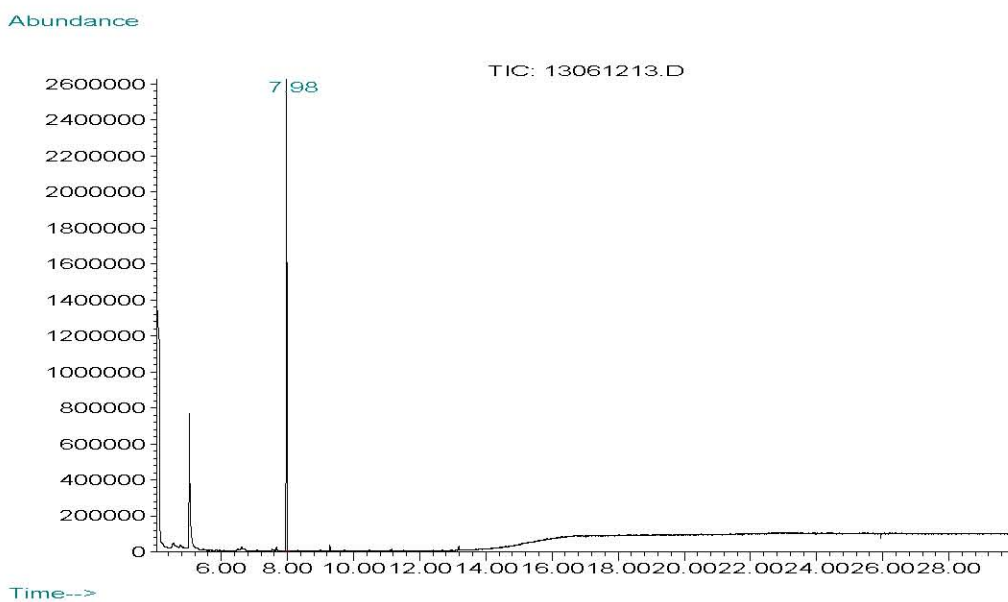


Retention Time (Min)	Area %	NIST Library Match	CAS no	% match
8.21	0.31	Cyclopentasiloxane, decamethyl-	000541-02-6	47
9.51	0.65	Cyclohexasiloxane, dodecamethyl-	000540-97-6	91
10.68	7.68	Trisiloxane, 1,1,1,5,5,5-hexamethyl	003555-47-3	47
11.72	16.58	Tris(trimethylsilyl)borate	004325-85-3	22
12.61	18.18	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	50
13.42	16.28	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	40
14.15	12.79	1-Monolinoleoylglycerol trimethyls	054284-45-6	30
14.82	9.39	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(	038147-00-1	64
15.44	1.97	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	45
16.09	4.78	9,12,15-Octadecatrienoic acid, 2,3	055521-22-7	43
16.81	2.51	3-Isopropoxy-1,1,1,7,7,7-hexamethyl	071579-69-6	45
17.65	1.49	1-Monolinoleoylglycerol trimethyls	054284-45-6	64
18.66	1.814	Tetrasiloxane, decamethyl-	000141-62-8	35
19.91	0.71	Tetrasiloxane, decamethyl-	000141-62-8	43
21.49	3.21	Tetrasiloxane, decamethyl-	000141-62-8	50

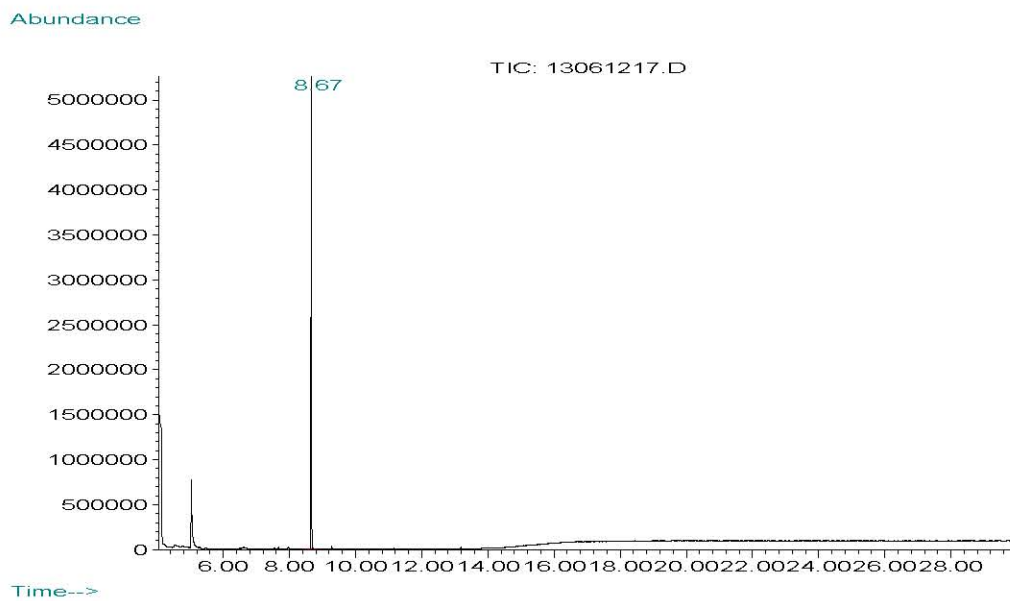
Figure 31 - 15655-11 GC-MS Chromatogram+ NIST Library Search Report



**Figure 32 – GC-MS Chromatogram - Octamethylcyclopentasiloxane [D4] 20ppm Standard**



**Figure 33 – GC-MS Chromatogram - Decamethylcyclopentasiloxane [D5] 20ppm Standard**



**Figure 34 – GC-MS Chromatogram - Dodecamethylpentasiloxane [D6] 20ppm Standard**

### 6.3.3 ICP-MS

#### Summary of Results (ppm)

results < 0.05 ppm not reported

>1 ppm in  
bold

Sample:	15655-1A	15656-1B	15657-2A	15658-2B	15659-3A	15660-3B	15661-4A	15662-4B	15663-5A	15664-5B
	PIP_IMGHC-TX-H-270	PIP_IMGHC-TX-H-270	PIP_IMGHC-TX-H-130	PIP_IMGHC-TX-H-130	PIP_IMGHC-TX-S-225	PIP_IMGHC-TX-S-225	PIP_IMGHC-TX-H-270	PIP_IMGHC-TX-H-270	PIP_IMGHC-TX-H-130	PIP_IMGHC-TX-H-130
Mg										<b>1.31</b>
K										<b>2.36</b>
Ti					0.11	0.08				0.16
Cr				0.18	<b>1.50</b>	<b>1.64</b>	0.26		0.13	0.27
Mn					0.18					
Fe				<b>10.08</b>	<b>52.56</b>	<b>15.71</b>	<b>5.05</b>		<b>14.86</b>	<b>8.24</b>
Co					0.13	0.05	0.10			0.05
Ni	0.09			0.17	<b>1.16</b>	0.62	0.24		0.17	0.31
Cu				0.06	0.15	0.10	0.13			0.06
Ga				0.06						
Se							0.11			
Sn				0.68	0.5	0.35	0.66		0.14	0.45
Cs	0.39	0.37	0.20	0.22	0.11	0.11	0.36			
Ba				0.56	0.30	0.12	0.07			
Pt	0.15	0.16	0.18	0.2	0.07	0.1	0.12	0.13	0.25	0.25
Au	<b>2.16</b>	<b>3.03</b>	<b>2.69</b>	<b>2.02</b>	<b>2.41</b>	<b>1.99</b>	<b>1.82</b>	<b>1.25</b>	<b>2.00</b>	<b>1.61</b>
Pb					0.97		0.06			

Table 4 – ICP-MS Results Summary  
[Note: All other elements < 0.05 ppm]



Sample:	15665-6A	15666-7A	15667-8A	15668-9A	15669-10A	15670-11A
<b>Mg</b>					0.90	
<b>K</b>			<b>3.51</b>	<b>1.40</b>	0.09	<b>14.81</b>
<b>Ti</b>						
<b>Cr</b>	0.42	0.42	0.41	<b>1.10</b>	0.28	0.84
<b>Mn</b>	0.15					0.12
<b>Fe</b>	<b>10.54</b>	<b>11.15</b>	<b>9.71</b>	<b>18.19</b>	<b>10.54</b>	<b>25.20</b>
<b>Co</b>	0.04	0.06		0.05		0.07
<b>Ni</b>	0.08	0.49	0.90	0.38	0.09	<b>1.50</b>
<b>Cu</b>	0.20				0.07	0.21
<b>Ga</b>						
<b>Se</b>						
<b>Sn</b>	<b>1.25</b>	0.45	0.39	0.32	0.27	<b>1.6</b>
<b>Cs</b>						
<b>Ba</b>	0.41	0.16		0.09		0.29
<b>Pt</b>	<b>3.49</b>	<b>3.67</b>	<b>3.76</b>	<b>2.51</b>	0.39	0.79
<b>Au</b>	<b>1.29</b>	<b>1.01</b>	<b>1.01</b>	0.93	0.38	0.58
<b>Pb</b>				0.91		<b>4.29</b>

Table 4 (cont)  
[Note: All other elements <0.05ppm]

## 6.4 Standard Operating Procedures Used

LGC/SOP/VS/NW/10 - Infrared Spectroscopy General Procedures and Interpretation

LGC/SOP/VS/NW/12 - Operation of Nicolet Fourier Transform Infrared Spectrometers

LGC/SOP/VS/NW/18 - Use of Attenuated Total Reflection Units

SOP/CHROM/NW/73 - Operation and use of Hewlett Packard GC/MS

LGC/SOP/EA/NW/80 v6 - Operation of the Agilent 7500ce ICP-MS

LGC/SOP/EA/NW/85 v3 - Semi-Quantitative Analysis using the Agilent 7500ce ICP-MS  
SINS/A1-0014 Procedure for Acid Digestion using Microwave Energy

## 6.5 Records

<b>Analyst name:</b>	<b>Record book:</b>	<b>Technique:</b>
██████████	LGCNW/576	FTIR
██████████	LGCNW/546	GC-MS
██████████	LGCNW/634	ICP-MS