

Definitive Final Report

Study Title	Extraction and organic analysis of implants
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RESPONSIBLE ANALYST AUTHENTICATION AND COMPLIANCE STATEMENT

Extraction and organic analysis of implants

I, the undersigned, hereby declare that the work described in this final report was performed under my supervision, as Responsible Analyst, and that this final report provides a true and accurate record of the results obtained.

This study was performed in accordance with the agreed protocol, unless otherwise stated, and the study objectives for this phase of the study were achieved. This phase of the study was also performed in accordance with Covance Standard Operating Procedures and in accordance with the current version of the:

United Kingdom GMP Guidelines

The procedures in this phase of the study were also performed to the standards required by the current versions of the:

United Kingdom GLP Regulations

and the:

OECD GLP Principles

[Redacted Signature]

Responsible Analyst
Covance Laboratories Ltd

3/AUG/2010

Date

RESPONSIBLE PERSONNEL

Extraction and organic analysis of implants

The following staff were responsible for key elements of the study:

Analyst

████████████████████ ██████████

████████████████████ ██████████

ARCHIVE STATEMENT

Extraction and organic analysis of implants

All primary data, or authenticated copies thereof, and result reports will be retained in the Covance archives for one year after issue of the final set of results. At this time, the Sponsor will be contacted to determine whether the data should be returned, retained or destroyed on their behalf.

NB archiving of examples of the reference standards and samples, if required, will be the responsibility of the Sponsor.

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SUMMARY

The objective of the study is to develop an extraction procedure for the gels from the supplied implants and analyse the resulting extracts for organic components using gas chromatography with mass selective detection (GC-MSD). General purpose analytical methods will be used to detect any organic components from the extracts.

Head-space GC-MS will be carried out directly on samples of the implant gel to assess volatile organic components.

Detected components, where possible, have been identified by comparison of their mass spectra with those from Wiley7/Nist05 libraries. Limited quantification of individual components from the head-space GC-MS method has been carried out using the internal standard, pentane, as quantification standard.

Details of the GC-MSD methods are provided in Appendix 1.

METHODS

Solvent based extracts

Initially on receipt of the samples, one implant was opened by cutting and removing the implant cover. Samples were taken from the gel and the remainder of the gel stored at controlled room temperature in a sealed container.

Samples of the gel, approx. 2 g were transferred to glass vials and 5 mL of a range of water immiscible solvents were added. The solvents used were: - chloroform, cyclohexane, dichloromethane, ethyl acetate, n-heptane and toluene. The vials were shaken by hand for approx. 10 sec and allowed to stand. The gel was not dispersed by the solvents, so a further 5 mL of solvent was added. No improvement in the dispersion of the gel was seen.

Water (10 mL) was added to all vials and the vials shaken and allowed to stand. Phase separation was obtained from all samples, but the water/solvent mixtures did not fully disperse the gel.

Logbook G867

A second batch of extracts was prepared from the same gel using the following solvents: - chloroform, dichloromethane, ethyl acetate, n-heptane and toluene. Approx. 2 g of gel was added

to 10 mL of solvent. The samples were shaken for 30 minutes on an horizontal shaker at 200 cycles/min. The extracts were allowed to stand and approx. 1 mL was transferred to a GC vial and capped for analysis by GC-MS. Control blanks were assayed for each of the solvents.

Method detail is given in Appendix 1.

The obtained chromatograms were integrated and each peak was compared with Wiley07/Nist05 libraries to obtain, where possible, an identification. Where no suitable match was found the peak was classified as un-identified. The mass to charge ratios (m/z) of the spectral peaks was listed in decreasing order of abundance.

Log book G867 Sequence G20100727B

Direct head-space analysis

Approx. 2 g of gel was transferred to a 20 mL head-space vial and capped securely. The internal standard, pentane dissolved in methanol (1 μ L) was added using a calibrated syringe and needle via the septa. Control blanks were prepared from sealed head-space vials with pentane as above. The vials were transferred to the head-space attachment for GC-MS analysis.

Method detail is given in Appendix 1.

Log book G867 Sequence G20100727A

The obtained chromatograms were integrated and each peak was compared with Wiley07/Nist05 libraries to obtain, where possible, an identification. Where no suitable match was found the peak was classified as un-identified. The mass to charge ratios (m/z) of the spectral peaks was listed in decreasing order of abundance.

RESULTS

The results are given in Table 1 with a list of peak number, retention time, spectra number assessed, peak identification from Wiley7/Nist05 library and comments. Results are given in Tables 1 to 6.

FIGURES

Figure 1 Chloroform extract full scale

File :D:\HPChem Data\8232-400\AA\AA02.D
Operator : ██████████
Acquired : 27 Jul 2010 16:54 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Chloroform extract
Misc Info :
Vial Number: 2

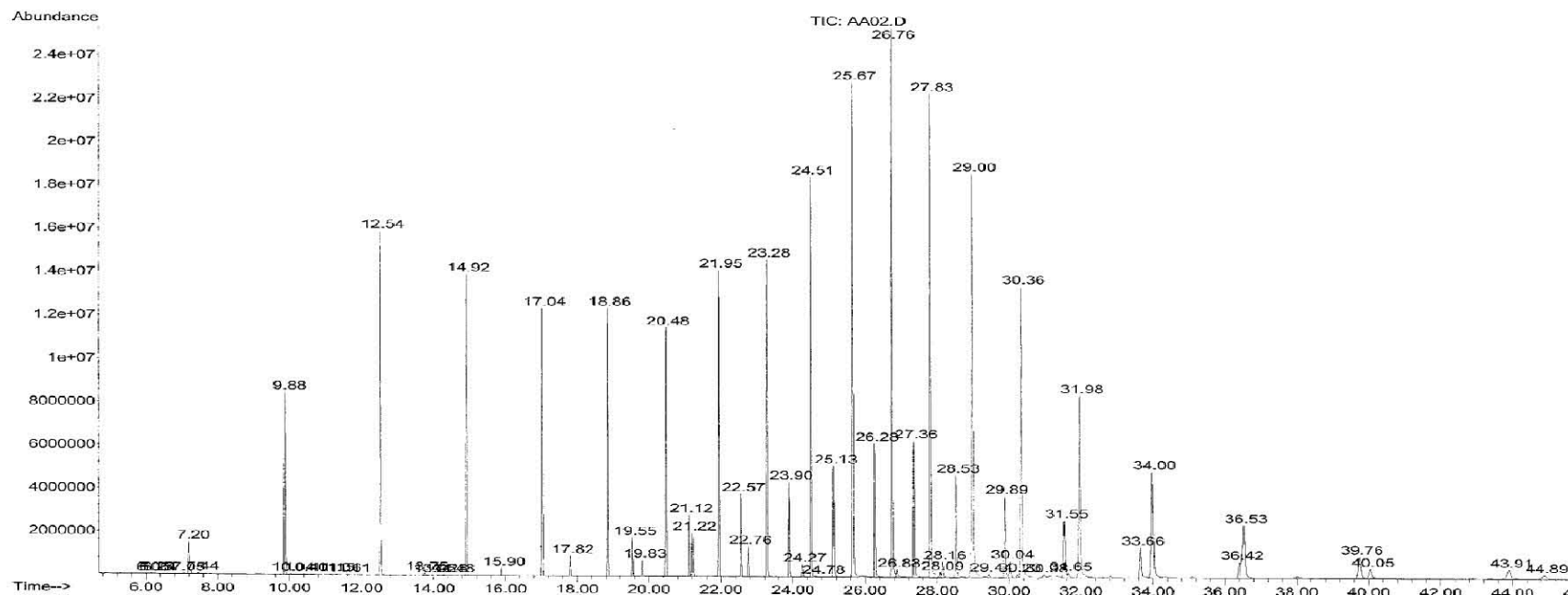


Figure 2 Chloroform extract expanded scale (1)

File :D:\HPChem Data\8232-400\AA\AA02.D
Operator : ██████████
Acquired : 27 Jul 2010 16:54 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Chloroform extract
Misc Info :
Vial Number: 2

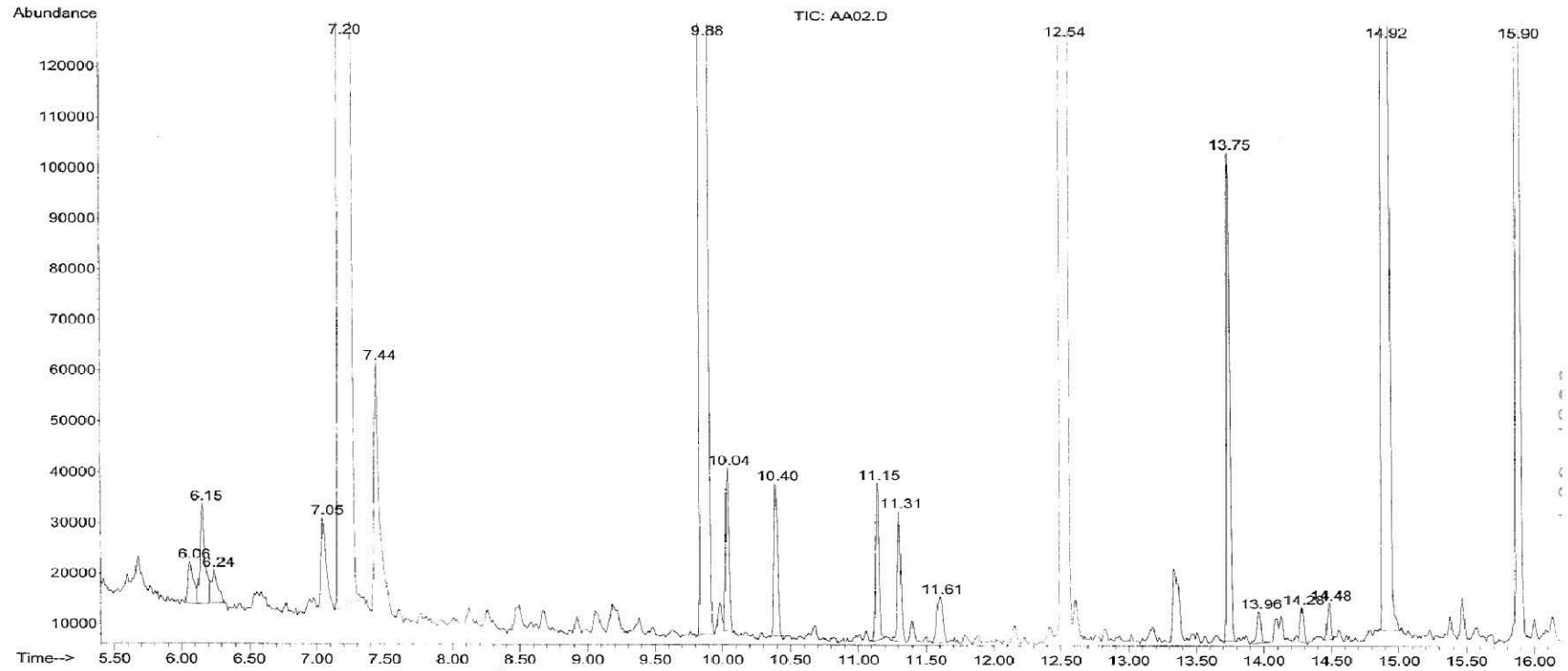


Figure 3 Chloroform extract expanded scale (2)

File :D:\HPCHEM Data\8232-400\AA\AA02.D
Operator : ██████████
Acquired : 27 Jul 2010 16:54 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Chloroform extract
Misc Info :
Vial Number: 2

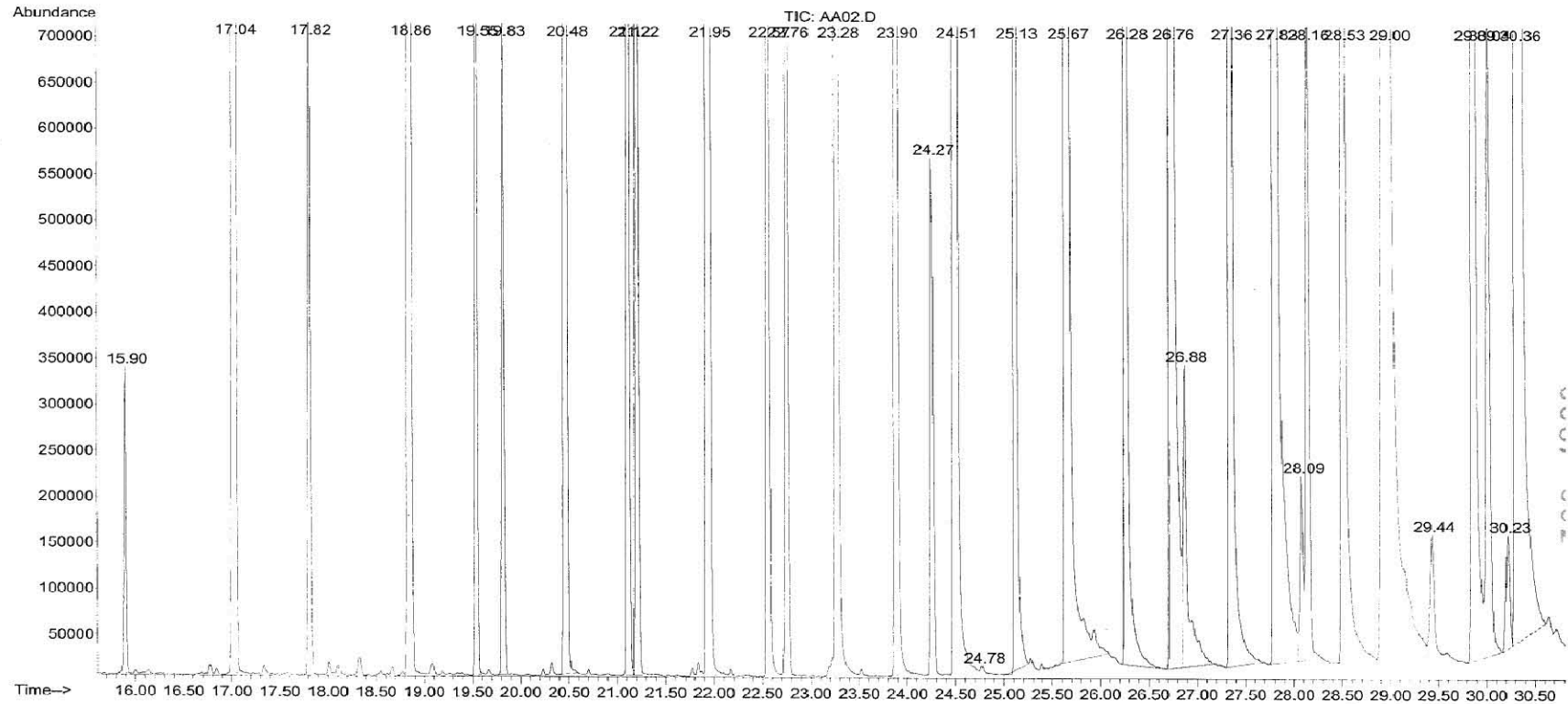


Figure 4 Chloroform extract expanded scale (3)

File :D:\HPChem Data\8232-400\AA\AA02.D
Operator :
Acquired : 27 Jul 2010 16:54 using AcqMethod 8232_400_SCAN.M
Instrument : instrument #1
Sample Name: Chloroform extract
Misc Info :
Vial Number: 2

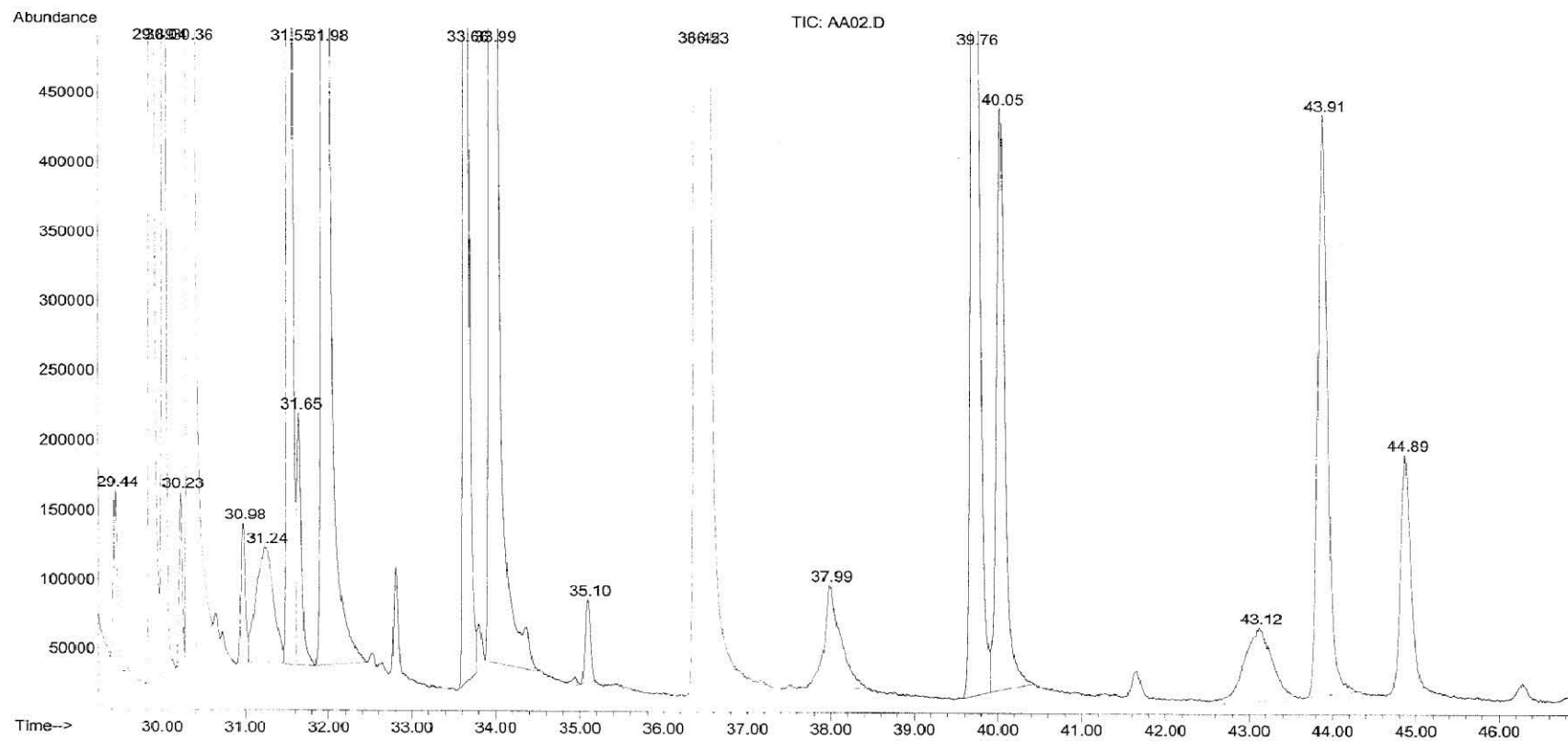


Figure 5 Dichloromethane extract full scale

File :D:\HPChem Data\8232-400\AA\AA04.D
Operator :
Acquired : 27 Jul 2010 18:49 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: DCM extract
Misc Info :
Vial Number: 4

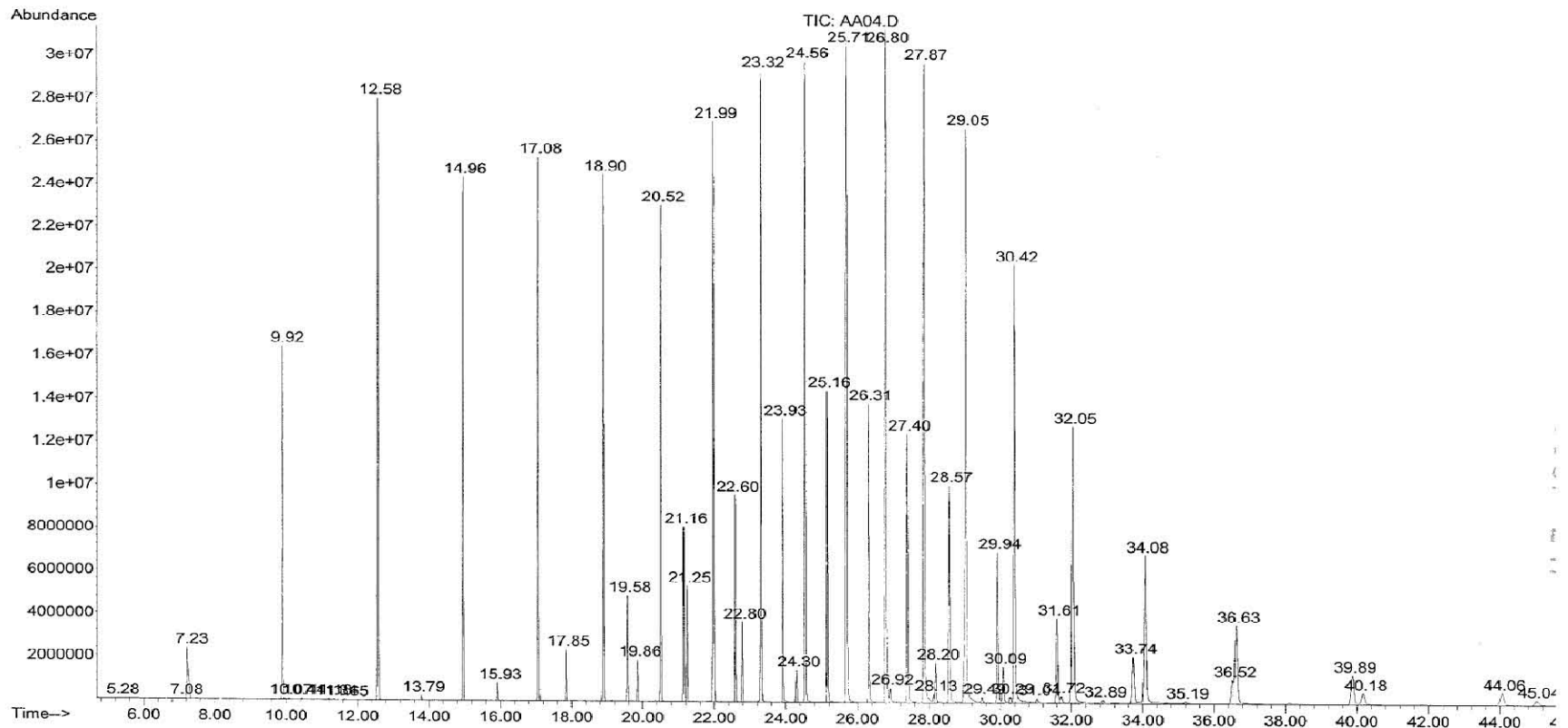


Figure 6 Dichloromethane extract expanded scale (1)

File :D:\HPChem Data\8232-400\AA\AA04.D
Operator : ██████████
Acquired : 27 Jul 2010 18:49 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: DCM extract
Misc Info :
Vial Number: 4

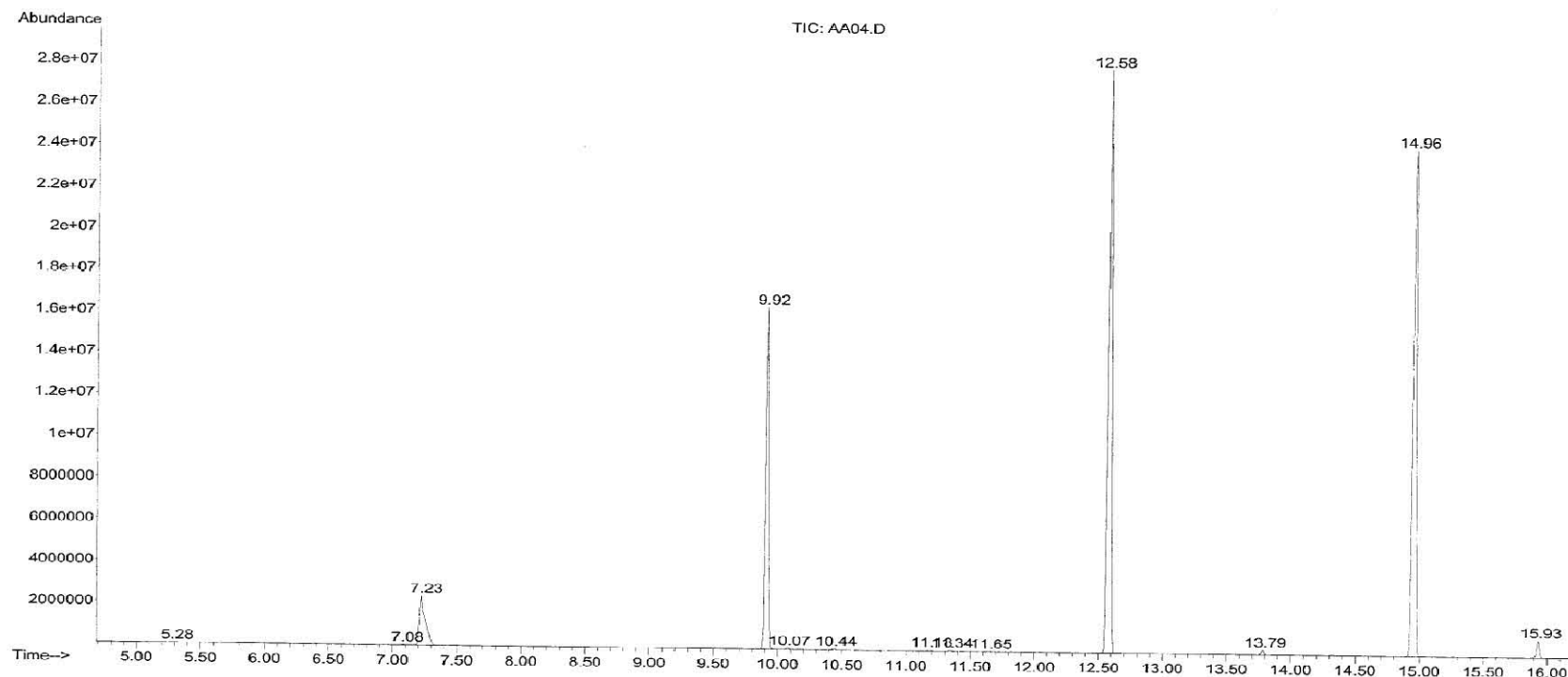


Figure 7 Dichloromethane extract expanded scale (2)

File :D:\HPChem Data\8232-400\AA\AA04.D
Operator :
Acquired : 27 Jul 2010 18:49 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: DCM extract
Misc Info :
Vial Number: 4

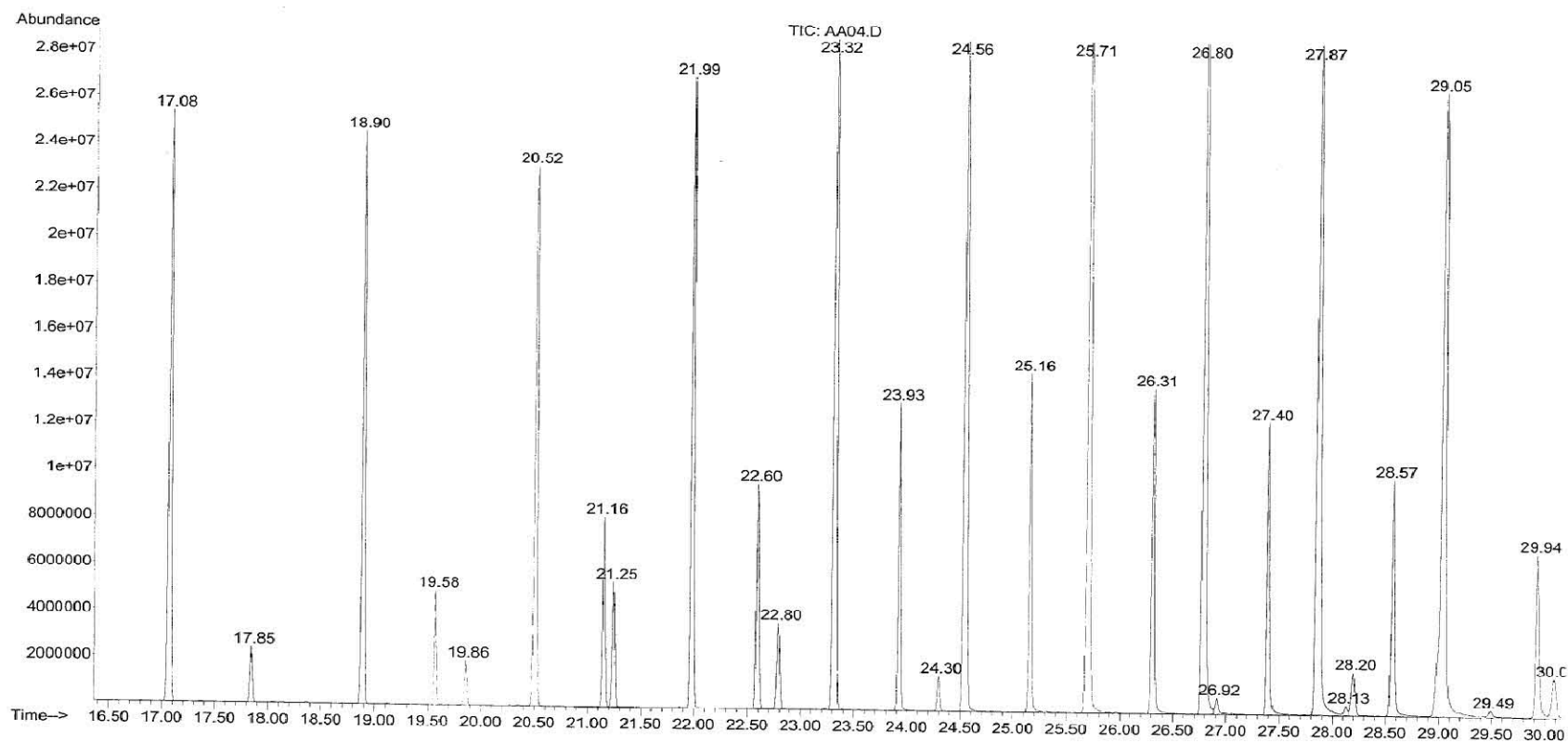


Figure 8 Dichloromethane extract expanded scale (3)

File : D:\HPCHEM>Data\8232-400\AA\AA04.D
Operator :
Acquired : 27 Jul 2010 18:49 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: DCM extract
Misc Info :
Vial Number: 4

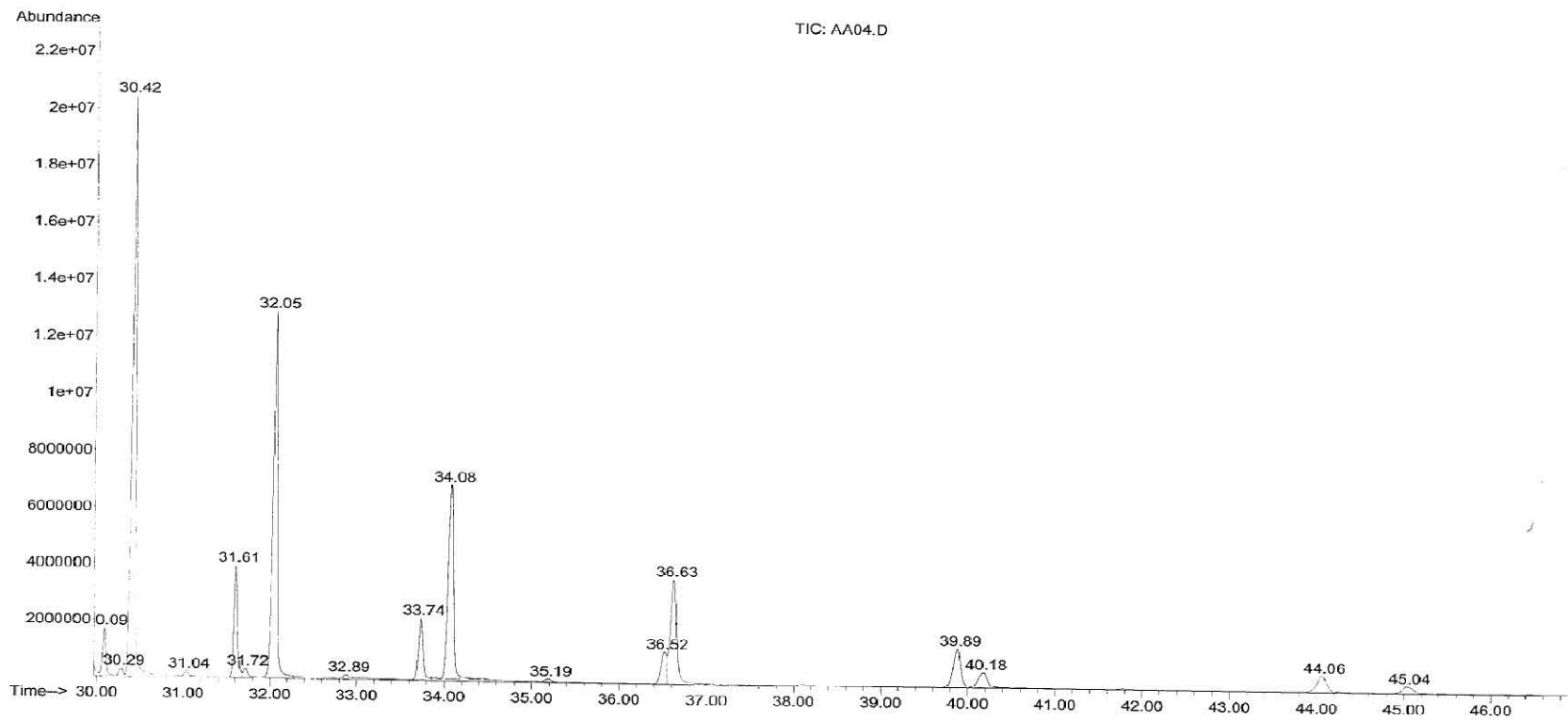


Figure 9 Ethyl acetate extract full scale

File : D:\HPChem Data\8232-400\AA\AA06.D
Operator : ██████████
Acquired : 27 Jul 2010 20:44 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Ethyl acetate extract
Misc Info :
Vial Number: 6

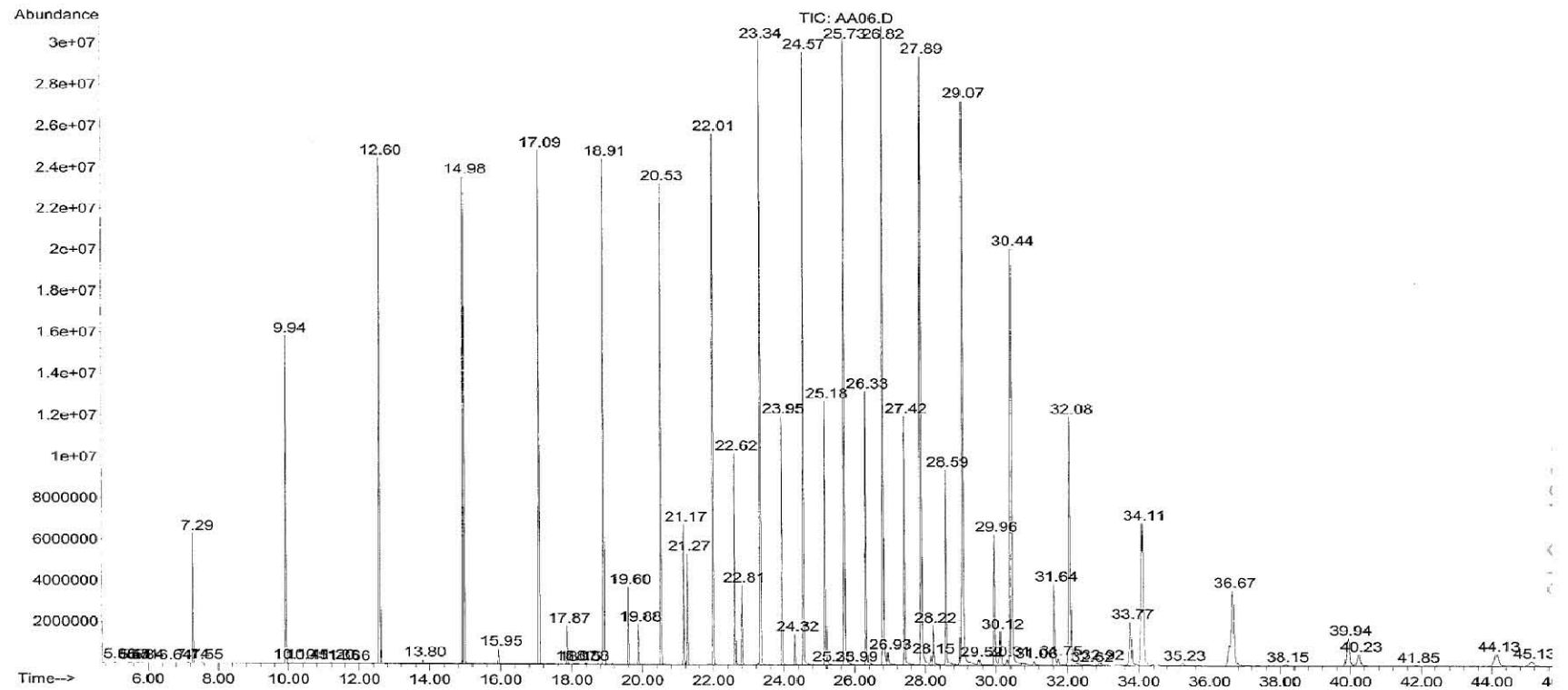


Figure 10 Ethyl acetate extract expanded scale (1)

File :D:\HPCHEM Data\8232-400\AA\AA06.D
Operator : ██████████
Acquired : 27 Jul 2010 20:44 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Ethyl acetate extract
Misc Info :
Vial Number: 6

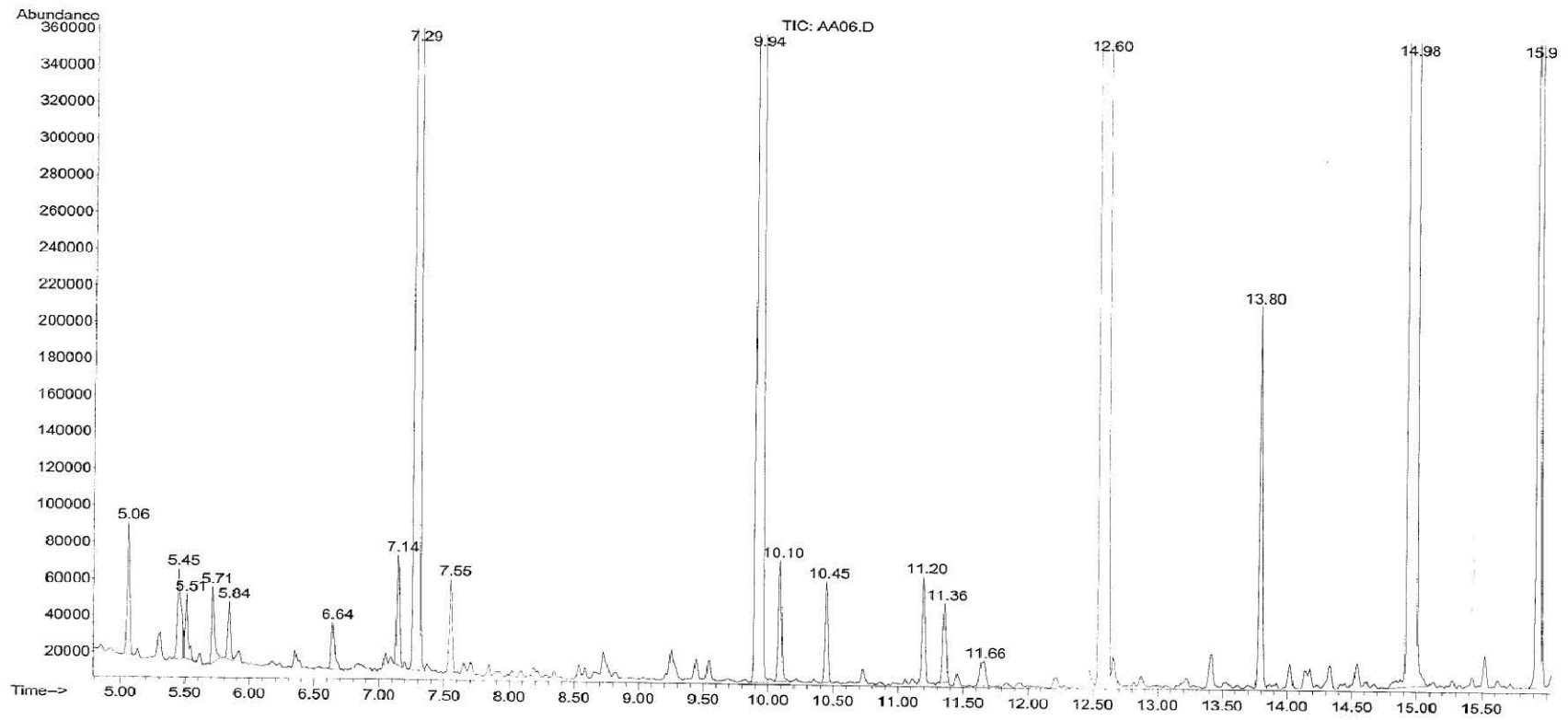


Figure 11 Ethyl acetate extract expanded scale (2)

File :D:\HPChem Data\8232-400\AA\AA06.D
Operator :
Acquired : 27 Jul 2010 20:44 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Ethyl acetate extract
Misc info :
Vial Number: 6

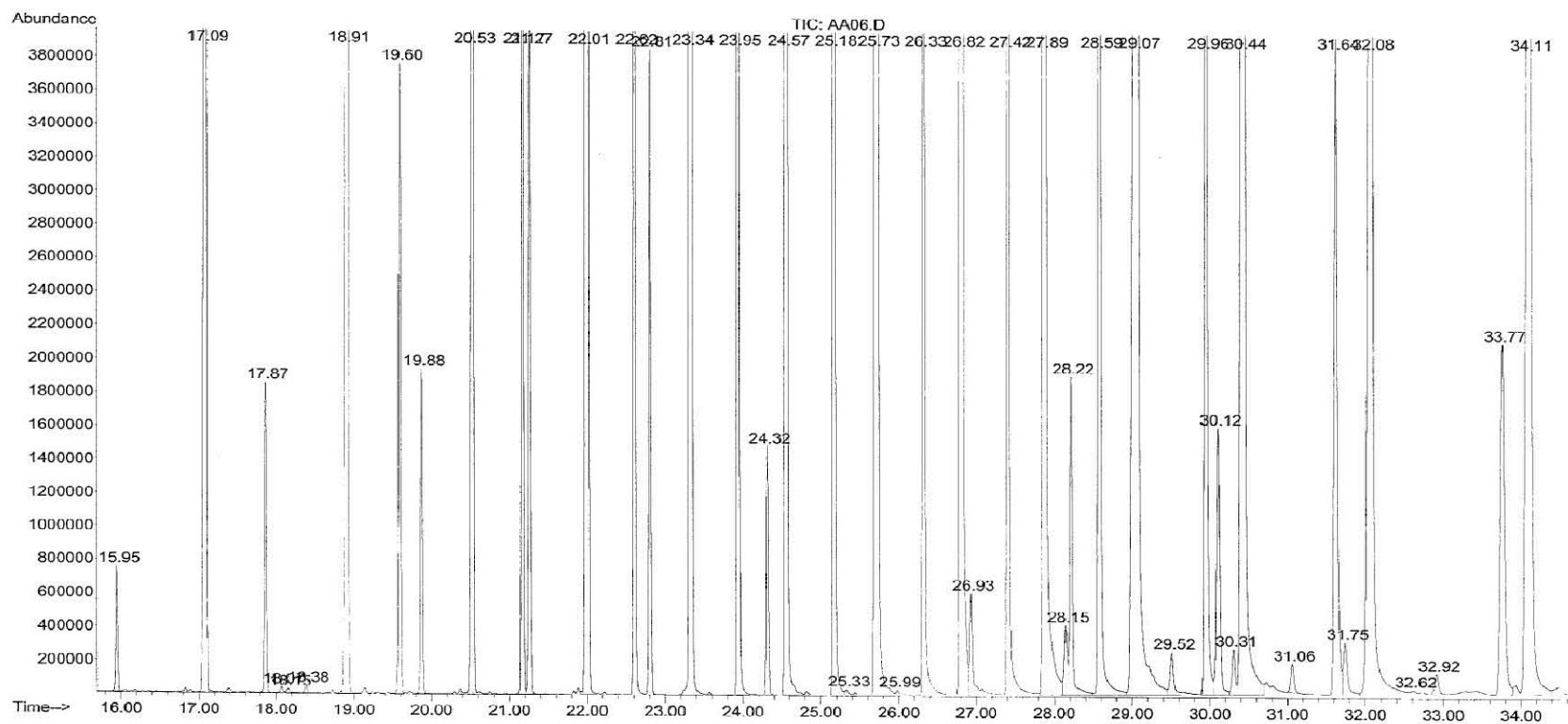


Figure 12 Ethyl acetate extract expanded scale (3)

File :D:\HPChem Data\8232-400\AA\AA06.D
Operator :
Acquired : 27 Jul 2010 20:44 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Ethyl acetate extract
Misc Info :
Vial Number: 6

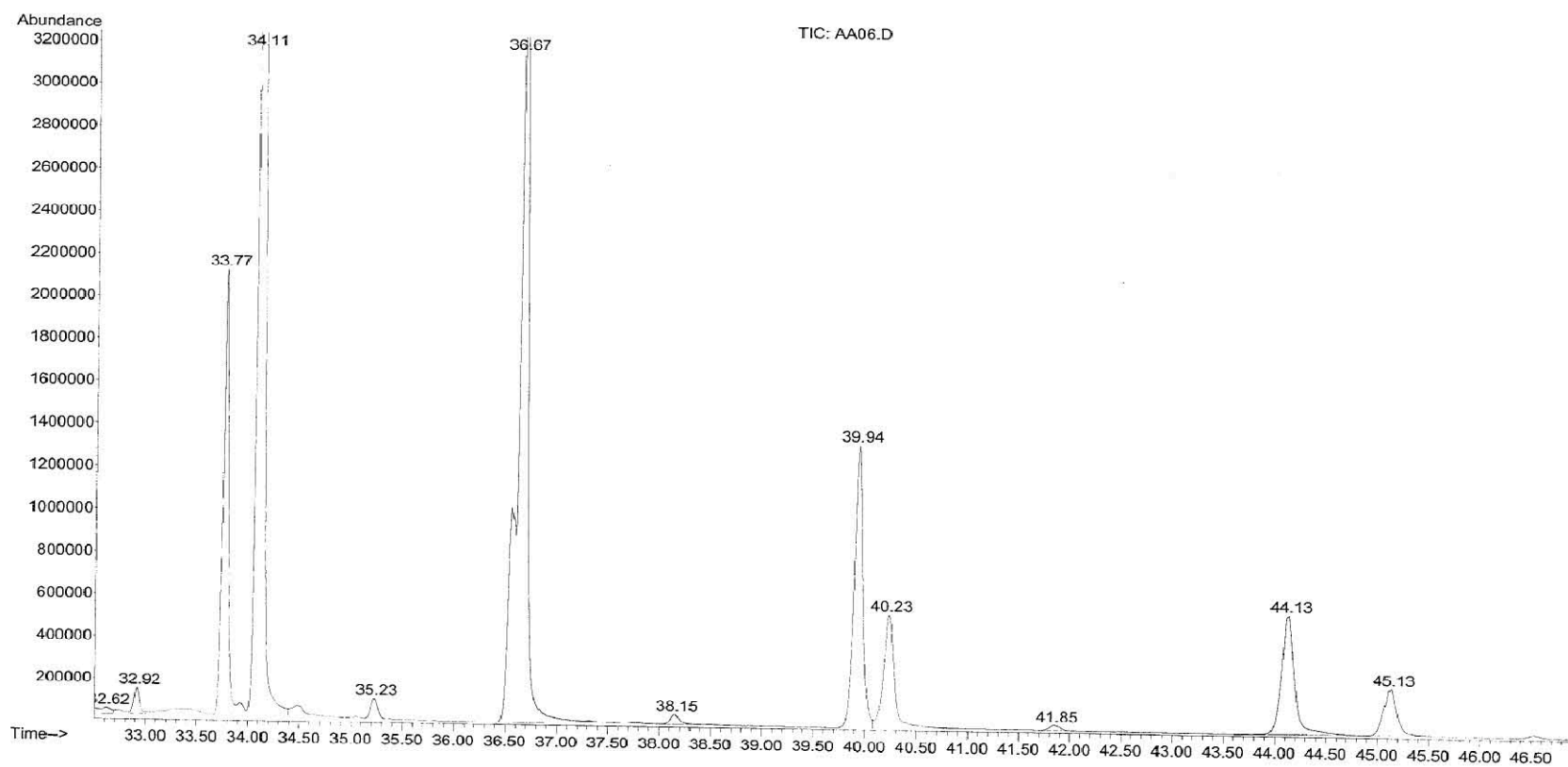


Figure 13 n-Heptane extract full scale

File :D:\HPChem_Data\8232-400\AA\AA08.D
Operator : ██████████
Acquired : 27 Jul 2010 22:40 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: n-heptane extract
Misc Info :
Vial Number: 8

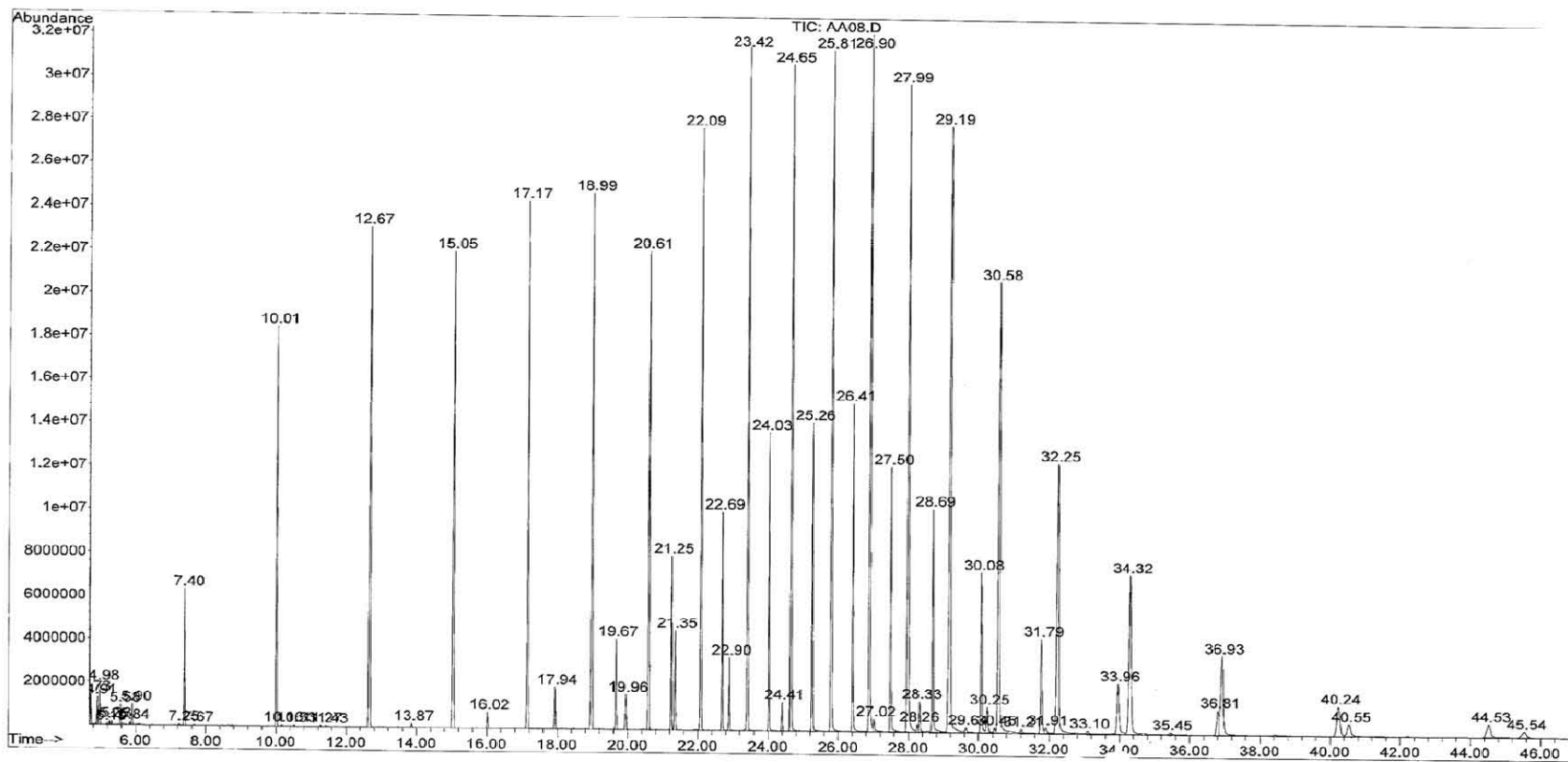


Figure 14 n-Heptane extract expanded scale (1)

File :D:\HPCChem Data\8232-400\AA\AA08.D
Operator : ██████████
Acquired : 27 Jul 2010 22:40 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: n-heptane extract
Misc Info :
Vial Number: 8

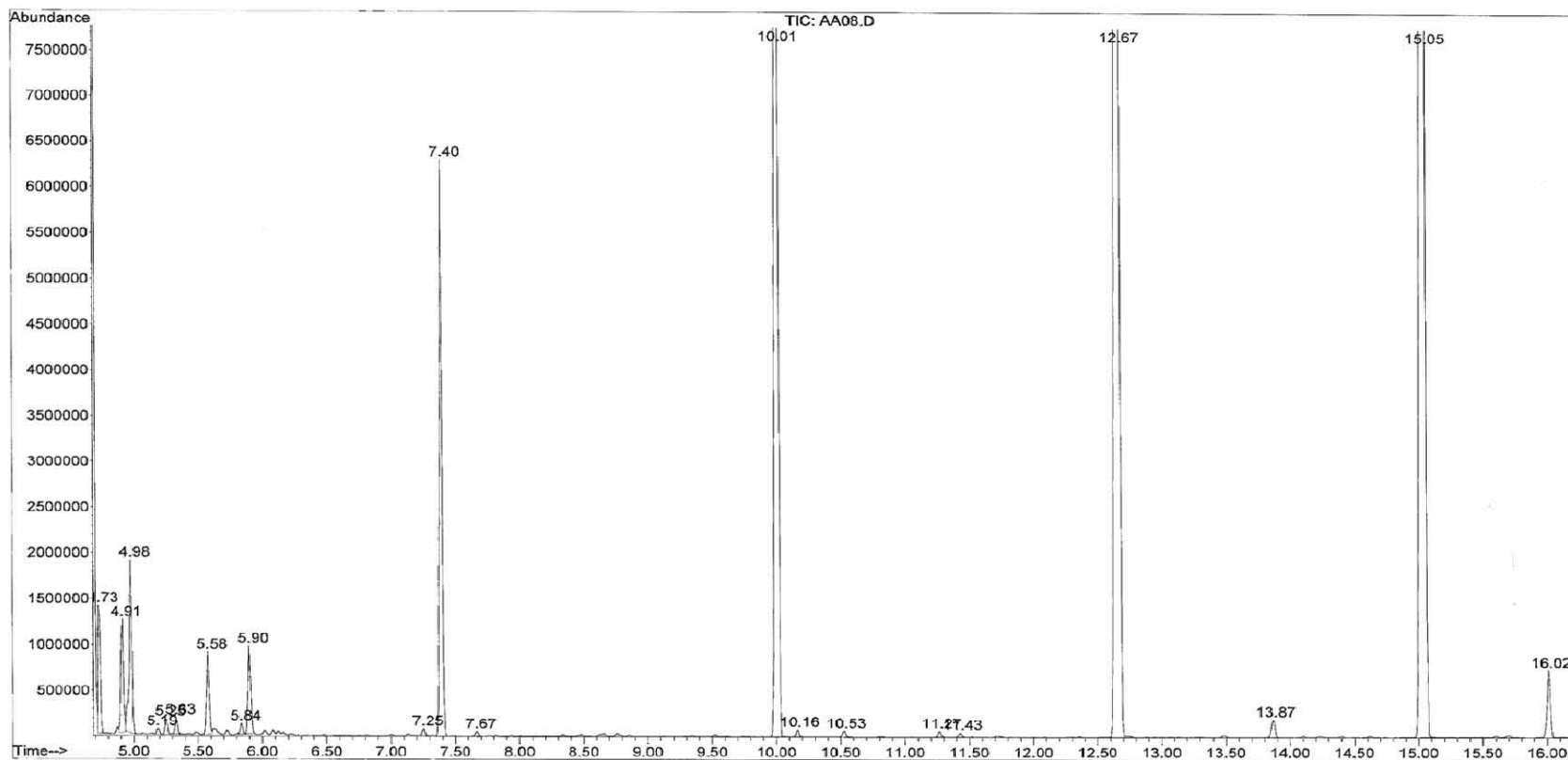


Figure 15 n-Heptane extract expanded scale (2)

File :D:\HPCHEM>Data\8232-400\AA\AA08.D
Operator :
Acquired : 27 Jul 2010 22:40 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: n-heptane extract
Misc Info :
Vial Number: 8

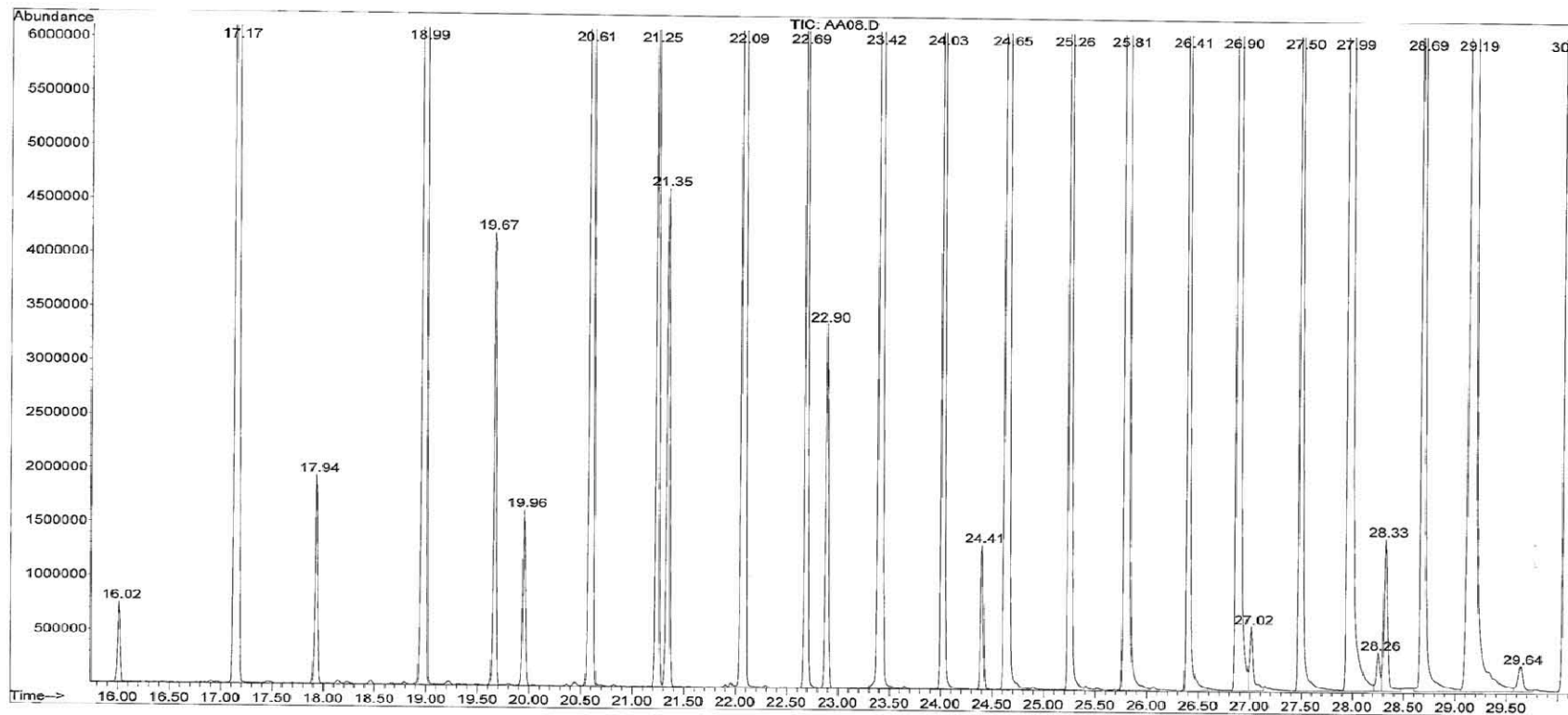


Figure 16 n-Heptane extract expanded scale (3)

File :D:\HPChem Data\8232-400\AA\AA08.D
Operator : ██████████
Acquired : 27 Jul 2010 22:40 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: n-heptane extract
Misc Info :
Vial Number: 8

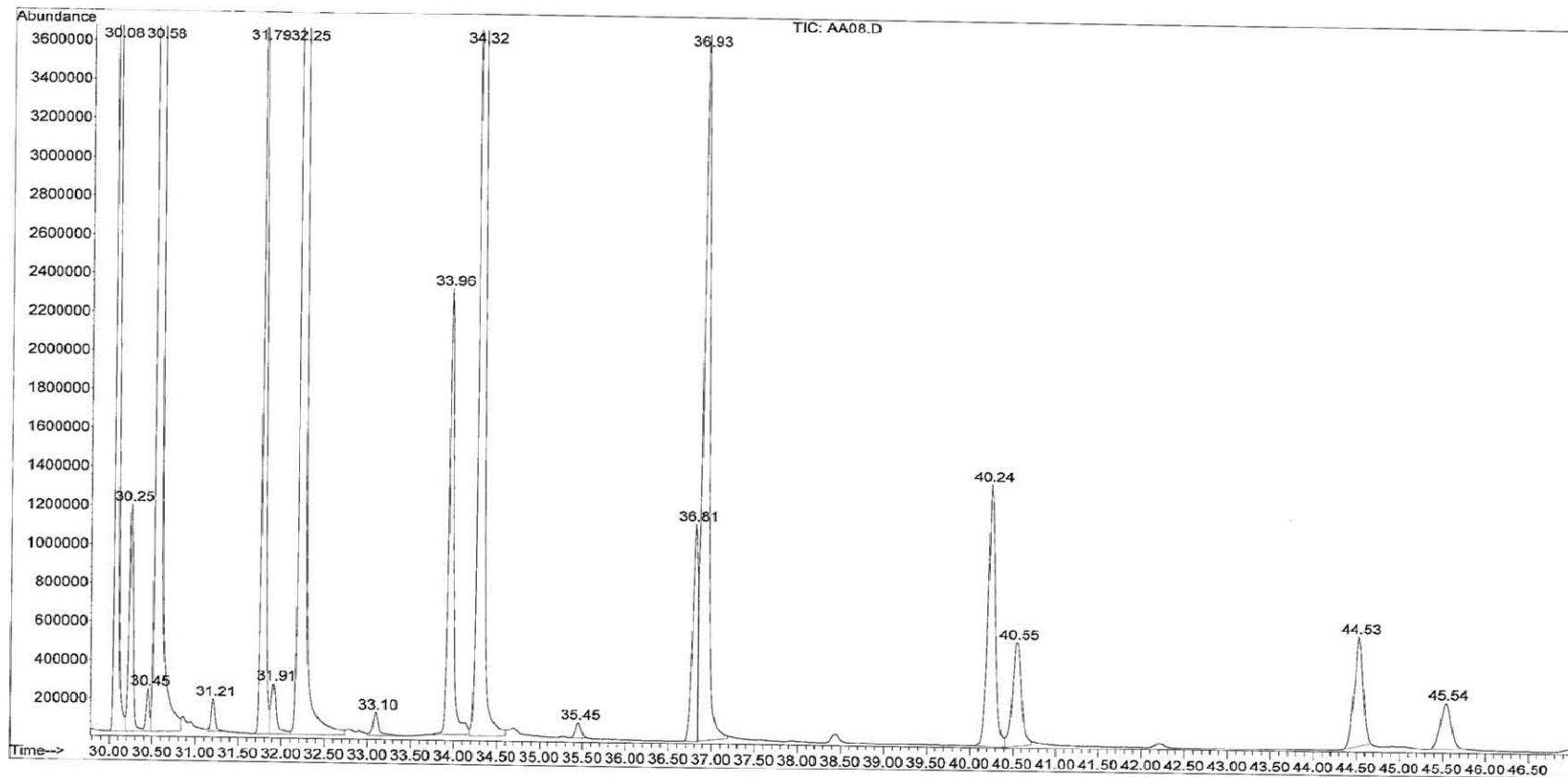


Figure 17 Toluene extract full scale

File :D:\HPChem Data\8232-400\AA\AA10.D
Operator : ██████████
Acquired : 28 Jul 2010 00:35 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Toluene extract
Misc Info :
Vial Number: 10

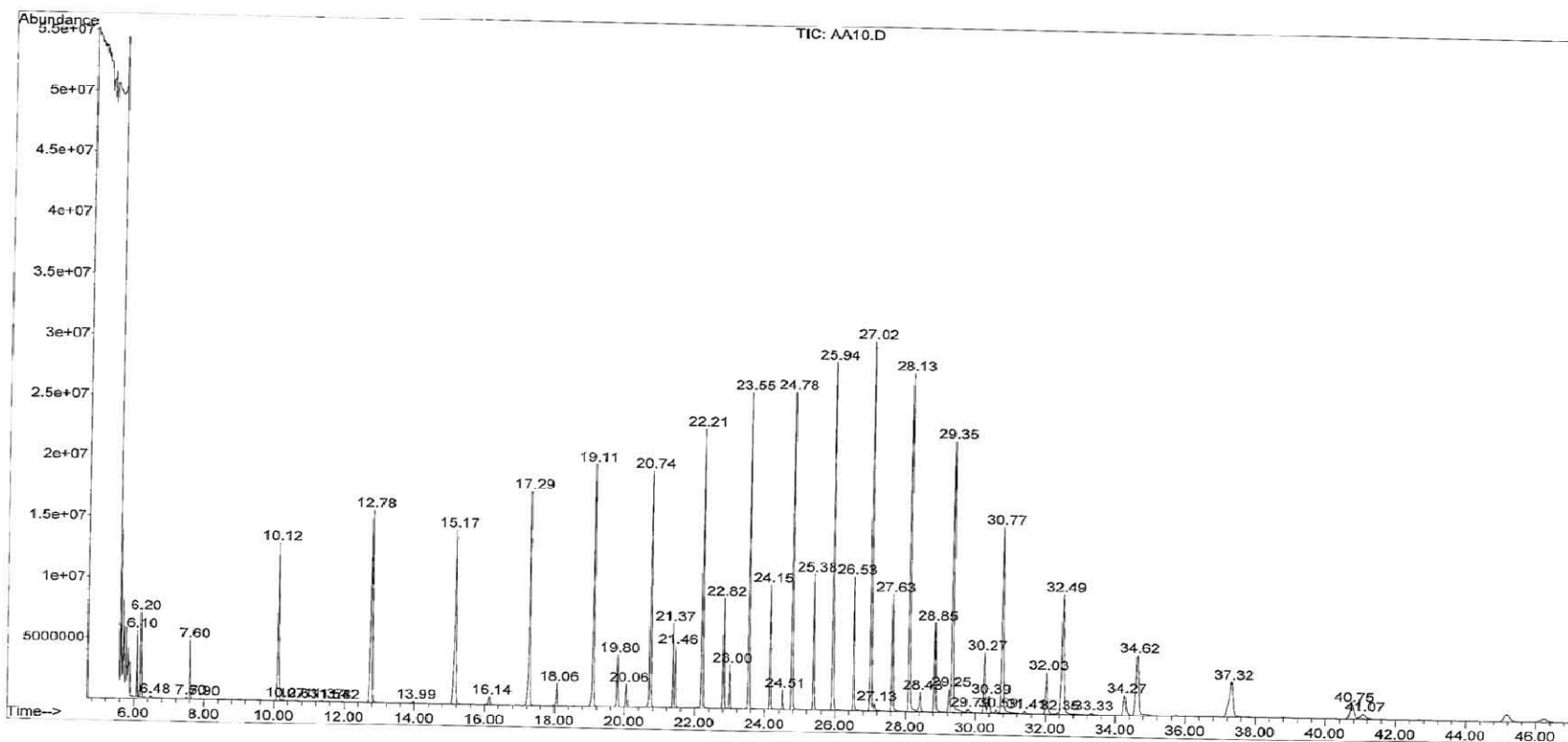


Figure 18 Toluene extract expanded scale (1)

File :D:\HPChem_Data\8232-400\AA\AA10.D
Operator : ██████████
Acquired : 28 Jul 2010 00:35 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Toluene extract
Misc Info :
Vial Number: 10

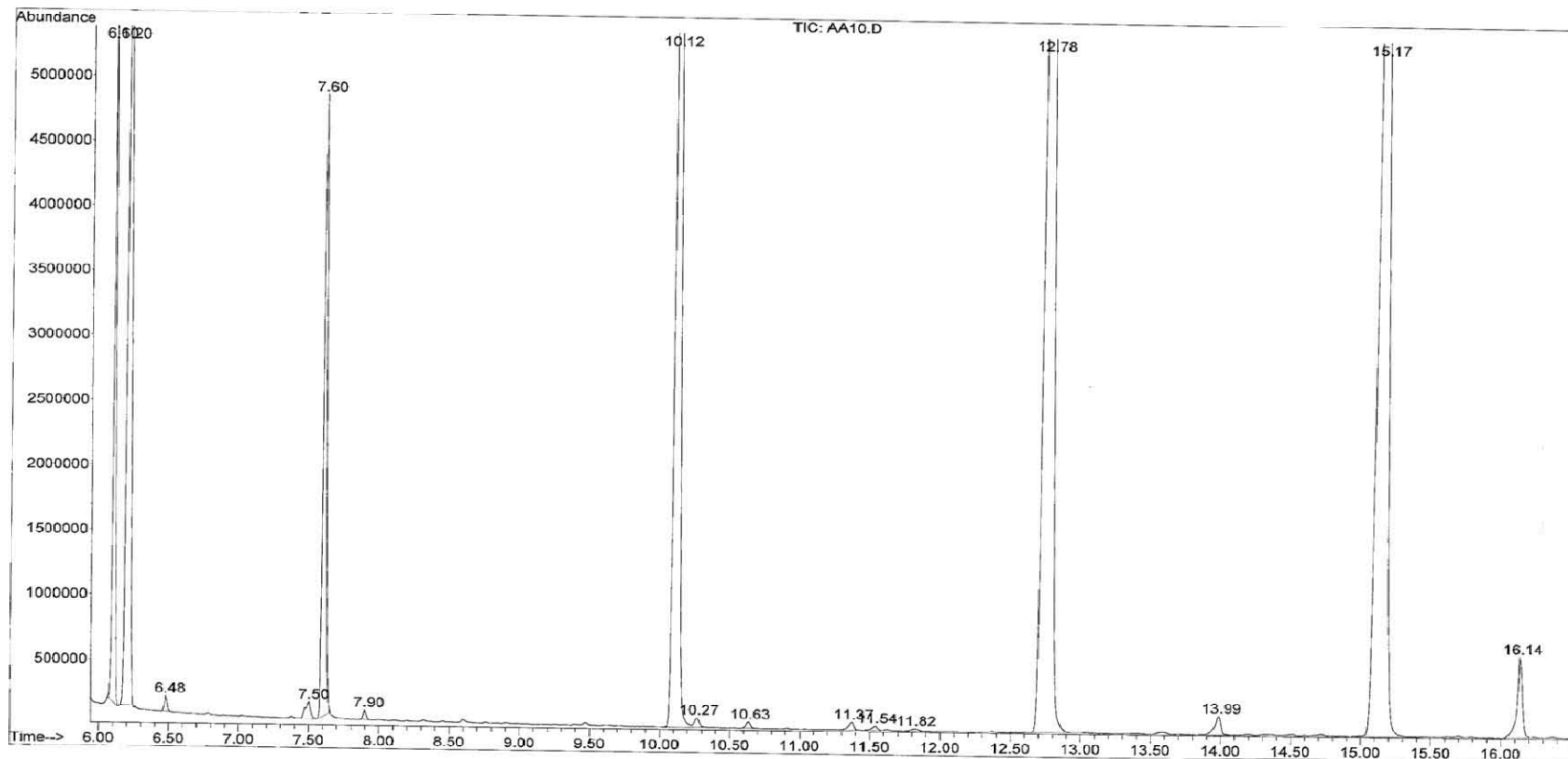


Figure 19 Toluene extract expanded scale (2)

File :D:\HEChem Data\8232-400\AA\AA10.D
Operator :
Acquired : 28 Jul 2010 00:35 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Toluene extract
Misc Info :
Vial Number: 10

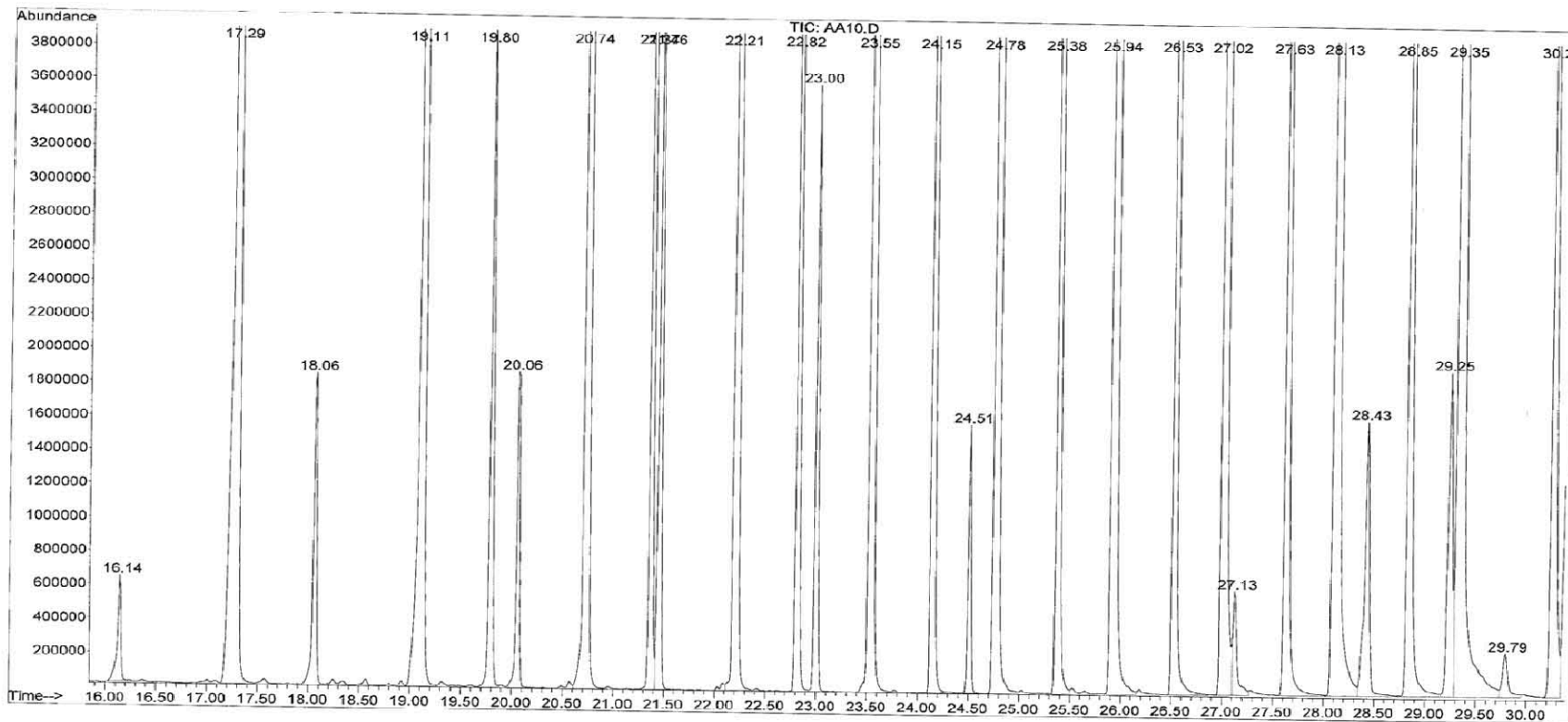


Figure 20 Toluene extract expanded scale (3)

File : D:\HPChem Data\8232-400\AA\AA10.D
Operator : ██████████
Acquired : 28 Jul 2010 00:35 using AcqMethod 8232_400_SCAN.M
Instrument : Instrument #1
Sample Name: Toluene extract
Misc Info :
Vial Number: 10

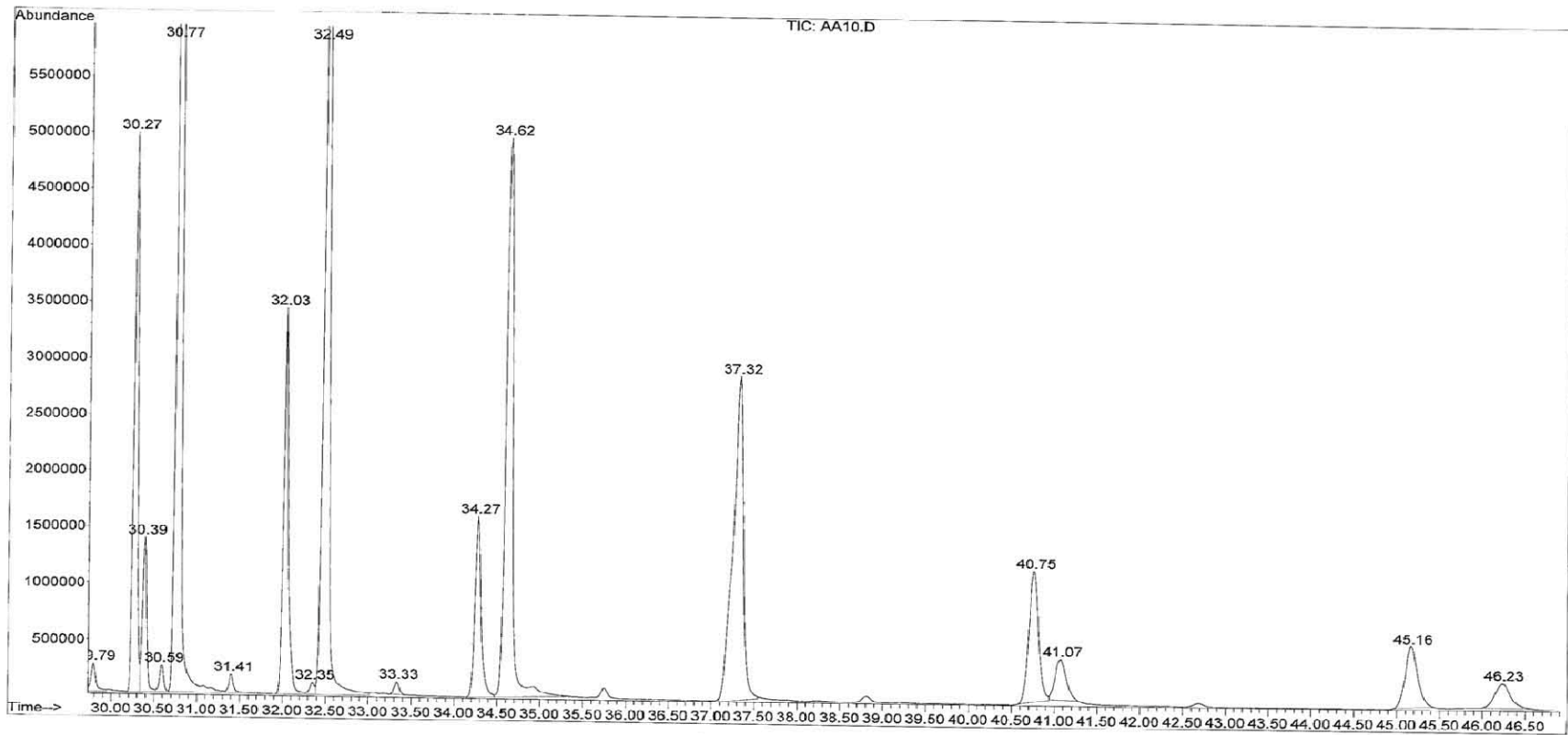


Figure 21 Head-space chromatograms full scale

File :D:\HPCHEM Data\8232-400\Volatiles-2010\AA\AA07.D
Operator : ██████████
Acquired : 27 Jul 2010 23:52 using AcqMethod 8232400.M
Instrument : Instrument #1
Sample Name : HP-52198-1 Prep 1
Misc Info :
Vial Number : 7

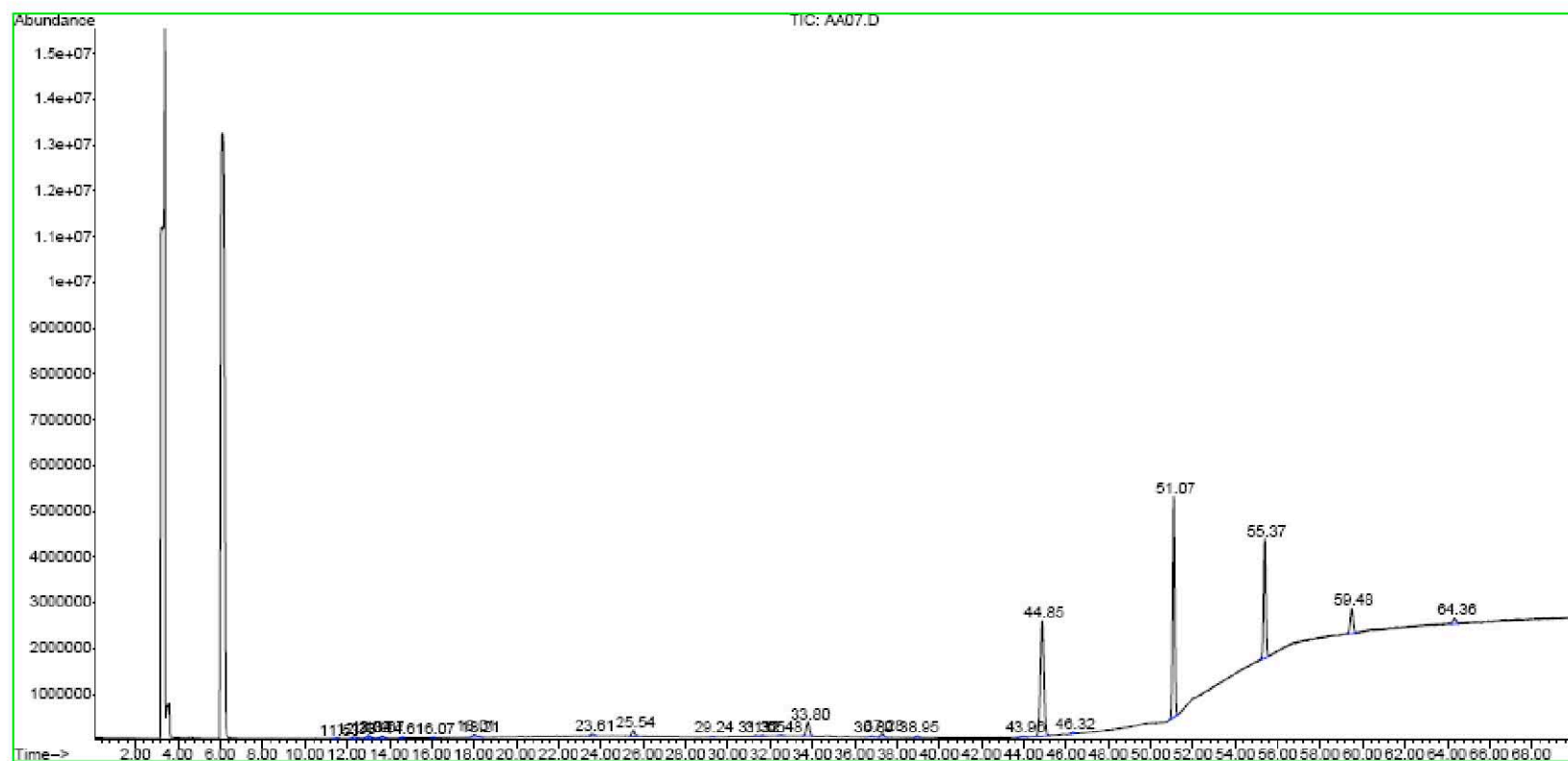


Figure 22 Head-space chromatogram expanded scale (1)

File : D:\HPCHEM_Data\8232-400\Volatiles-2010\AA\AA07.D
Operator : ██████████
Acquired : 27 Jul 2010 23:52 using AcqMethod 8232400.M
Instrument : Instrument #1
Sample Name : HP-52198-1 Prep 1
Misc Info :
Vial Number : 7

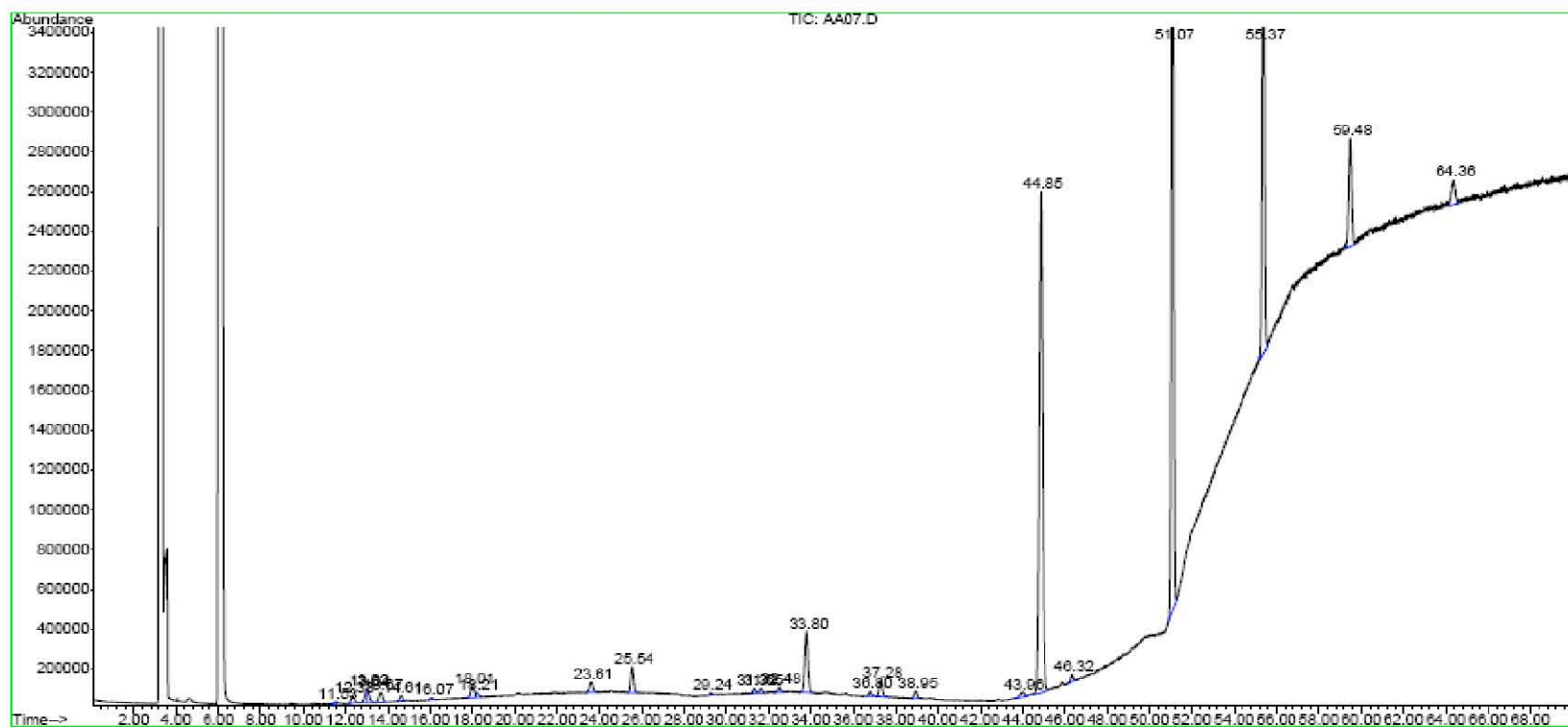


Figure 23 Head-space chromatogram expanded scale (2)

File :D:\HPChem Data\8232-400\Volatiles-2010\AA\AA07.D
Operator : ██████████
Acquired : 27 Jul 2010 23:52 using AcqMethod 8232400.M
Instrument : Instrument #1
Sample Name : HP-52198-1 Prep 1
Misc Info :
Vial Number: 7

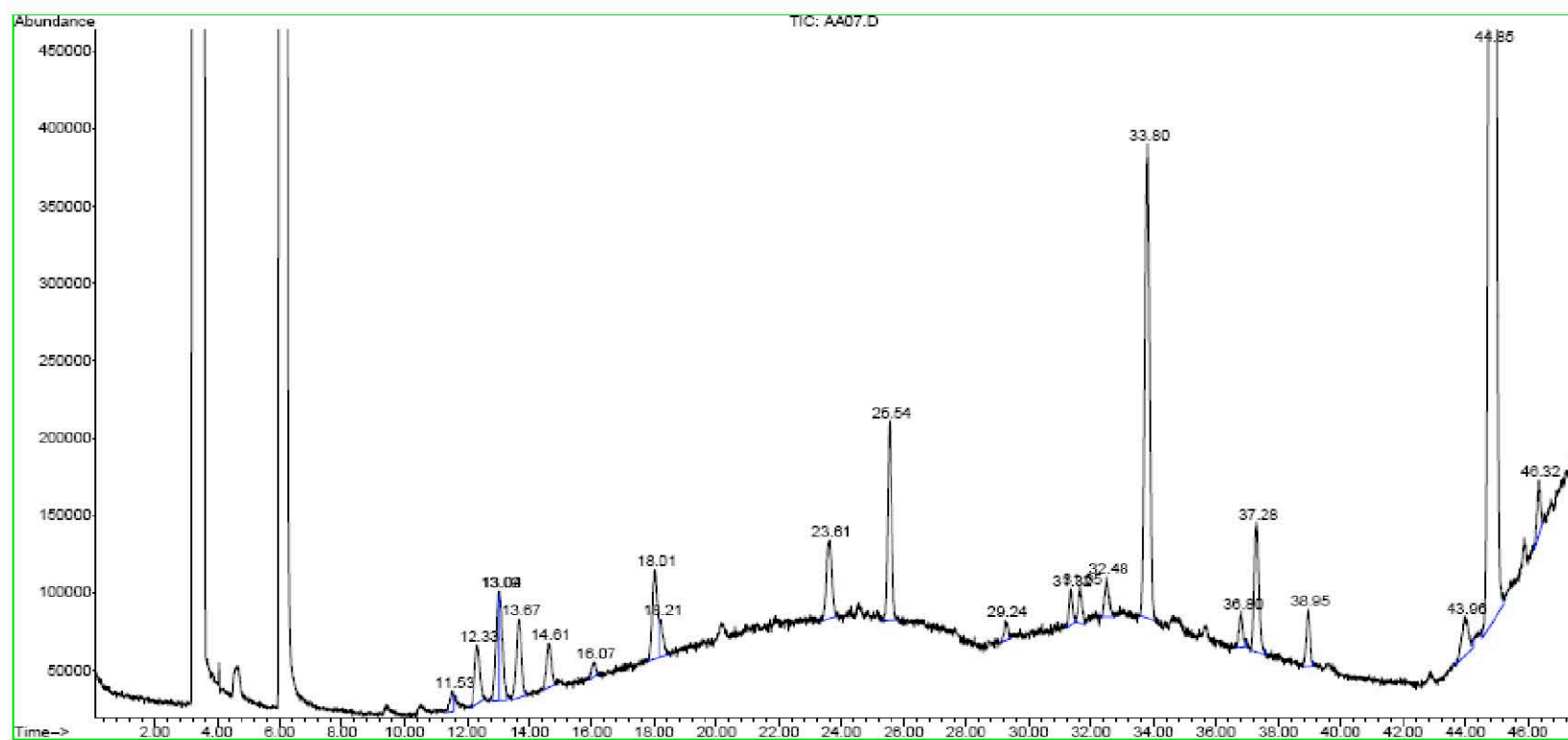
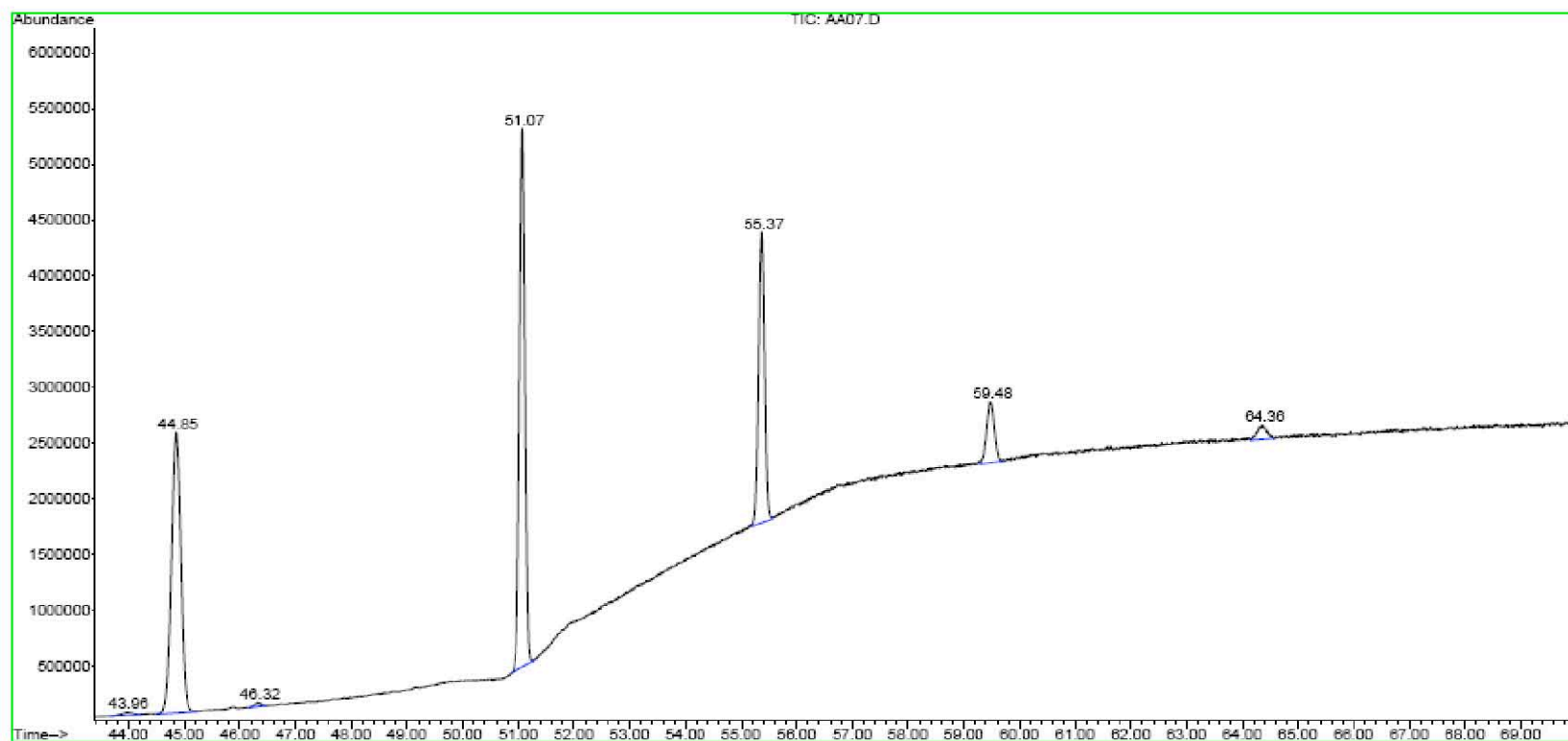


Figure 24 Head-space chromatogram expanded scale (3)

File :D:\HPChem Data\8232-400\Volatiles-2010\AA\AA07.D
Operator :
Acquired : 27 Jul 2010 23:52 using AcqMethod 8232400.M
Instrument : Instrument #1
Sample Name: HP-52198-1 Prep 1
Misc Info :
Vial Number: 7



TABLES

Table 1 Chloroform extract

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
1	6.063	133	Un-identified	-	m/z 83, 207, 151, 133, 179
2	6.156	142	1,1,2,2-tetrachloroethane	95	-
3	6.239	152	Un-identified	-	m/z 83, 207, 193, 281
4	7.047	230	1,1,3,3,5,5,7,7-Octamethyltetrasiloxane	64	-
5	7.193	243	1,1,3,3,5 Octamethylcyclotetrasiloxane	92	-
6	7.441	267	2,2,4,6,6-pentamethylheptane	59	-
7	9.877	502	Decamethyl-cyclopentasiloxane	91	-
8	10.042	517	1,1,3,3,5,5,7,7,9,9-decamethylpentasiloxane	94	-
9	10.395	552	Dodecamethyl-pentasiloxane	91	-
10	11.141	624	Un-identified	-	m/z 73, 327, 341, 207
11	11.307	640	Dodecamethylpentasiloxane	91	Fragmented oligomers
12	11.607	670	Un-identified	-	m/z 193, 207, 73, 327, 341
13	12.540	758	Dodecamethyl-cyclohexasiloxane	91	-
14	13.752	876	Tetradecamethyl-hexasiloxane	72	-
15	13.960	896	Un-identified	-	m/z 207, 105, 94, 44, 119
16	14.281	927	Un-identified	-	m/z 73, 207, 387, 44
17	14.488	947	Un-identified	-	m/z 207, 161, 91
18	14.923	988	Dodecamethyl-pentasiloxane	38	Fragmented oligomers
19	15.898	1083	Hexadecamethyl-heptasiloxane	80	-
20	17.037	1192	Tetracosamethylcyclododecasiloxane	64	-
21	17.815	1267	Un-identified	-	m/z 73, 221, 147, 207
22	18.861	1367	Tetradecamethyl-hexasiloxane	50	Fragmented oligomers

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
23	19.545	1435	Hexadecamethyl-heptasiloxane	80	Fragmented oligomers
24	19.825	1463	Un-identified	-	m/z 327, 405, 156, 343
25	20.478	1524	Tetradecamethyl-hexasiloxane	43	Fragmented oligomers
26	21.121	1587	Hexadecamethyl-heptasiloxane	10	Fragmented oligomers
27	21.214	1596	Un-identified	-	m/z 135, 197, 479, 329, 73
28	21.950	1666	Un-identified	-	m/z 73, 147, 355, 221, 281
29	22.561	1726	Un-identified see Note 1	-	m/z 221, 73, 147, 295, 207
30	22.758	1745	Un-identified	-	m/z 135, 403, 197, 73
31	23.286	1797	Octadecamethylcyclononasiloxane	59	-
32	23.898	1854	Un-identified see Note 1	-	m/z, 221, 73, 147, 295, 207
33	24.271	1890	Bis[di(trimethylsiloxy)phenylsiloxy]trimethylsiloxane	47	-
34	24.509	1913	Tetradecamethyl-hexasiloxane	76	Fragmented oligomers
35	24.779	1940	Un-identified	-	m/z 207, 73, 147, 221, 281
36	25.131	1973	Tetradecamethyl-hexasiloxane	32	Fragmented oligomers
37	25.670	2025	Un-identified see Note 1	-	m/z, 221, 73, 147, 355, 281
38	26.281	2084	Un-identified see Note 1	-	m/z, 221, 73, 147, 295, 207
39	26.758	2131	Un-identified	-	m/z 355, 221, 147, 73, 429
40	26.882	2143	Un-identified see Note 2	-	m/z 135, 73, 147, 221
41	27.359	2190	Un-identified see Note 1	-	m/z, 221, 295, 147, 73, 281
42	27.825	2234	Un-identified	-	m/z 221, 355, 147, 73, 429
43	28.095	2258	Un-identified see Note 2	-	m/z 135, 73, 147, 221
44	28.157	2265	Un-identified	-	m/z 451, 218, 389, 373, 156
45	28.530	2302	Un-identified see Note 1	-	m/ z 221, 295, 147, 73, 369
46	28.996	2346	Un-identified see Note 1	-	m/z 221, 147, 73, 355, 281
47	29.442	2391	Un-identified see Note 2	-	m/z 135, 73, 147, 221, 207
48	29.888	2432	Un-identified see Note 1	-	m/z 221, 147, 73, 295, 281
49	30.033	2446	Un-identified see Note 3	-	m/z 197, 135, 259
50	30.230	2466	Un-identified see Note 3	-	m/z 135, 197, 73, 465, 259
51	30.354	2479	Un-identified see Note 1	-	m/z, 221, 73, 147, 355, 281

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
52	30.976	2538	Un-identified see Note 2	-	m/z 135, 73, 147, 221
53	31.245	2569	Un-identified see Note 1	-	m/z 221, 147, 73, 207, 281
54	31.546	2593	Un-identified see Note 1	-	m/z 221, 147, 295, 73, 281
55	31.649	2603	Un-identified	-	m/z 135, 197, 73, 221
56	31.981	2634	Un-identified see Note 1	-	m/z 221, 147, 73, 355, 281
57	33.660	2795	Un-identified see Note 1	-	m/z 221, 147, 73, 295, 207
58	33.991	2826	Un-identified see Note 1	-	m/z 221, 147, 73, 281, 355
59	35.100	2936	Un-identified see Note 1	-	m/z 73, 135, 207, 147, 221
60	36.427	3062	Un-identified see Note 1	-	m/z 221, 147, 73, 295, 281
61	36.530	3074	Un-identified see Note 1	-	m/z 221, 147, 73, 355, 281
62	37.992	3215	Un-identified see Note 1	-	m/z 221, 147, 73, 207, 135
63	39.753	3384	Un-identified see Note 1	-	m/z 221, 147, 73, 281, 355
64	40.054	3413	Un-identified see Note 1	-	m/z 221, 147, 295, 73, 281
65	43.121	3707	Un-identified see Note 1	-	m/z 221, 207, 147, 281, 73
66	43.909	3785	Un-identified see Note 1	-	m/z 221, 147, 73, 281, 355
67	44.893	3878	Un-identified see Note 1	-	m/z 221, 147, 73, 295, 207

Note 1 Un-identified but likely to be polysiloxane oligomer fragments

Note 2 Un-identified but likely to be polysiloxane oligomer fragments from a different source than Note 1

Note 3 Closely related compounds

Peaks in **bold** text appear in the solvent blank

Table 2 Dichloromethane extract

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
1	5.286	59	p-xylene	78	-
2	7.089	235	1,1,3,3,5,5-hexamethyl-trisiloxane	80	-
3	7.224	249	Octamethyl-cyclotetrasiloxane	91	-
4	9.918	504	Decamethyl-cyclopentasiloxane	91	-
5	10.084	521	1,1,3,3,5,5,7,7,9,9-decamethyl-pentasiloxane	91	-
6	10.436	556	Dodecamethyl-pentasiloxane	91	-
7	11.182	628	Un-identified	-	m/z 73, 327, 341, 401, 59
8	11.338	643	Dodecamethyl-pentasiloxane	87	Silicone oligomers fragment
9	11.649	672	Un-identified	-	m/z 73, 193, 327, 207, 415
10	12.581	763	Dodecamethyl-cyclohexasiloxane	91	-
11	13.783	879	Tetradecamethyl-hexasiloxane	35	-
12	14.965	992	Dodecamethyl-pentasiloxane	25	Silicone oligomers fragment
13	15.929	1086	Un-identified see Note 1	-	m/z 221, 73, 147, 207
14	17.079	1196	Tetracosamethylcyclo-dodecasiloxane	22	-
15	17.846	1271	Un-identified see Note 1	-	m/z 221, 73, 147, 207
16	18.903	1373	Octadecamethyl-cyclononasiloxane	45	-
17	19.576	1438	Hexadecamethyl-heptasiloxane	72	-
18	19.867	1466	Un-identified	-	m/z 327, 405, 156, 343, 253
19	20.519	1527	Tetradecamethyl-hexasiloxane	38	Silicone oligomers fragment
20	21.162	1588	Silicone polymer	53	-
21	21.255	1599	Un-identified see Note 2	-	m/z 135, 197, 73, 479, 329
22	21.991	1670	Tetracosamethylcyclo-dodecasiloxane	58	Silicone oligomers fragment
23	22.602	1729	Un-identified see Note 1	-	m/z 221, 73, 147, 295, 207
24	22.799	1748	Un-identified see Note 2	-	m/z 135, 197, 73, 403, 341
25	23.328	1798	Tetradecamethyl-hexasiloxane	59	Silicone oligomers fragment
26	23.929	1858	Un-identified see Note 1	-	m/z 221, 73, 147, 295, 281

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
27	24.302	1894	Un-identified see Note 2	-	m/z 135, 73, 197, 209, 327
28	24.551	1918	Octadecamethyl-cyclononasiloxane	59	Silicone oligomers fragment
29	25.162	1977	Tetradecamethyl-hexasiloxane	25	Silicone oligomers fragment
30	25.711	2030	Octadecamethyl-cyclononasiloxane	47	Silicone oligomers fragment
31	26.312	2088	Hexadecamethyl-heptasiloxane	33	Silicone oligomers fragment
32	26.799	2133	Octadecamethyl-cyclononasiloxane	83	Silicone oligomers fragment
33	26.913	2146	Nonamethylphenyl-cyclopentasiloxane	38	-
34	27.400	2193	Hexadecamethyl-heptasiloxane	33	Silicone oligomers fragment
35	27.877	2237	Octadecamethyl-cyclononasiloxane	80	Silicone oligomers fragment
36	28.136	2263	Un-identified see Note 2	-	m/z 135, 73, 147, 221
37	28.198	2270	Un-identified	-	m/z 451, 218, 389, 373, 156
38	28.571	2306	Hexadecamethyl-heptasiloxane	25	Silicone oligomers fragment
39	29.059	2351	Tetradecamethyl-hexasiloxane	43	Silicone oligomers fragment
40	29.494	2395	Un-identified see Note 2	-	m/z 135, 73, 147, 221, 355
41	29.939	2437	Un-identified see Note 1	-	m/z 221, 147, 73, 295
42	30.095	2453	Un-identified	-	m/z 197, 135, 259, 391
43	30.292	2471	Un-identified see Note 2	-	m/z 135, 197, 73, 465, 259
44	30.416	2483	Octadecamethyl-cyclononasiloxane	72	Silicone oligomers fragment
45	31.038	2544	Un-identified see Note 2	-	m/z 135, 73, 147, 221, 281
46	31.608	2599	Hexadecamethyl-heptasiloxane	33	Silicone oligomers fragment
47	31.722	2610	Un-identified	-	m/z 135, 197, 73, 465
48	32.053	2642	Tetracosamethylcyclo-dodecasiloxane	72	Silicone oligomers fragment
49	32.883	2722	Un-identified see Note 2	-	m/z 135, 73, 147, 221, 207
50	33.732	2805	Hexadecamethyl-heptasiloxane	53	Silicone oligomers fragment
51	34.085	2835	Octadecamethyl-cyclononasiloxane	86	Silicone oligomers fragment
52	35.183	2945	Un-identified see Note 2	-	m/z 135, 73, 147, 221, 207
53	36.520	3071	Hexadecamethyl-heptasiloxane	59	Silicone oligomers fragment
54	36.624	3083	Octadecamethyl-cyclononasiloxane	64	Silicone oligomers fragment
55	39.888	3397	Octadecamethyl-cyclononasiloxane	80	Silicone oligomers fragment

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
56	40.188	3426	Hexadecamethyl-heptasiloxane	53	Silicone oligomers fragment
57	44.054	3799	Octadecamethyl-cyclononasiloxane	86	Silicone oligomers fragment
58	45.028	3895	Hexadecamethyl-heptasiloxane	45	Silicone oligomers fragment

Note 1 Un-identified but likely to be polysiloxane oligomer fragments

Note 2 Un-identified compounds but related fragments

Peaks in **bold** text appear in the solvent blank

Table 3 Ethyl acetate extract

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
1	5.058	37	Ethyl 2-methyl butyrate	94	-
2	5.452	75	m-xylene	94	-
3	5.514	81	Iso-amylacetate	80	-
4	5.711	100	Un-identified	-	m/z 133, 151, 43, 207
5	5.845	113	p-xylene	95	-
6	6.643	191	Un-identified	-	m/z 193, 209, 43, 97
7	7.141	238	1,1,3,3,5,5,7,7-octamethyl-tetrasiloxane	55	-
8	7.286	251	1,1,3,3,5,5,7,7-octamethyl-cyclotetrasiloxane	91	-
9	7.545	277	2,2,4,6,6-pentamethylheptane	59	-
10	9.939	507	Decamethyl-cyclopentasiloxane	91	-
11	10.094	522	1,1,3,3,5,5,7,7,9,9-decamethyl-pentasiloxane	91	-
12	10.447	557	Dodecamethyl-pentasiloxane	91	-
13	11.203	629	Un-identified	-	m/z 73, 327, 341, 59
14	11.359	645	Dodecamethyl-pentasiloxane	94	Silicone oligomers fragment
15	11.659	672	Un-identified	-	m/z 73, 327, 415, 207, 43
16	12.602	763	Dodecamethyl-cyclohexasiloxane	91	-
17	13.804	880	Tetradecamethyl-hexasiloxane	58	-
18	14.975	992	Dodecamethyl-pentasiloxane	38	Silicone oligomers fragment
19	15.949	1088	Hexadecamethyl-heptasiloxane	56	-
20	17.089	1197	Tetracosamethyl-cyclododecasiloxane	10	-
21	17.867	1272	Un-identified	-	m/z 73, 221, 147, 207, 295
22	18.063	1292	n-heptadecane	86	-
23	18.157	1301	Un-identified	-	m/z 207, 129, 222, 73, 91
24	18.385	1323	2,3-dihydro-1,1,3-trimethyl-3-phenyl-1H-Indene	81	CAS 3910-35-8
25	18.913	1373	Octadecamethyl-cyclononasiloxane	87	-

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
26	19.597	1439	Hexadecamethyl-heptasiloxane	32	Silicone oligomers fragment
27	19.877	1467	Un-identified	-	m/z 327, 405, 156, 253, 343
28	20.530	1530	Tetradecamethyl-hexasiloxane	43	Silicone oligomers fragment
29	21.172	1592	Hexadecamethyl-heptasiloxane	10	Silicone oligomers fragment
30	21.266	1601	Un-identified Note 1	-	m/z 135, 197, 479, 73, 329
31	22.001	1672	Tetracosamethyl-cyclododecasiloxane	58	Silicone oligomers fragment
32	22.613	1731	Hexadecamethyl-heptasiloxane	59	Silicone oligomers fragment
33	22.810	1751	Un-identified Note 1	-	m/z 135,403, 197, 341, 73
34	23.338	1800	Tetracosamethyl-cyclododecasiloxane	25	Silicone oligomers fragment
35	23.950	1860	Hexadecamethyl-heptasiloxane	10	Silicone oligomers fragment
36	24.323	1895	Bis[di(trimethylsiloxy)phenylsiloxy]-trimethylsiloxane	64	-
37	24.571	1919	Octadecamethyl-cyclononasiloxane	53	Silicone oligomers fragment
38	25.183	1978	Hexadecamethyl-heptasiloxane	53	Silicone oligomers fragment
39	25.338	1994	Silicate ion tetramer	53	-
40	25.732	2031	Tetracosamethyl-cyclododecasiloxane	64	Silicone oligomers fragment
41	25.981	2056	Tetradecamethyl-hexasiloxane	43	-
42	26.333	2089	Un-identified	-	m/z 221, 147, 73, 295, 281
43	26.820	2136	Tetracosamethyl-cyclododecasiloxane	38	Silicone oligomers fragment
44	26.934	2148	Bis[di(trimethylsiloxy)phenylsiloxy]-trimethylsiloxane	38	Silicone oligomers fragment
45	27.411	2194	Hexadecamethyl-heptasiloxane	42	Silicone oligomers fragment
46	27.888	2238	Octadecamethyl-cyclononasiloxane	91	Silicone oligomers fragment
47	28.157	2265	Un-identified Note 1	-	m/z 135, 73, 147, 221, 281
48	28.219	2270	Un-identified	-	m/z 218, 451, 389, 156, 373
49	28.592	2308	Hexadecamethyl-heptasiloxane	16	Silicone oligomers fragment
50	29.069	2354	Tetracosamethyl-cyclododecasiloxane	49	Silicone oligomers fragment
51	29.515	2397	Un-identified Note 1	-	m/z 135, 73, 147, 221, 281
52	29.960	2440	Hexadecamethyl-heptasiloxane	25	Silicone oligomers fragment
53	30.116	2455	Un-identified	-	m/z 197, 135, 259, 313, 391
54	30.313	2474	Un-identified	-	m/z 135, 197, 73, 465

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
55	30.437	2485	Octadecamethyl-cyclononasiloxane	72	Silicone oligomers fragment
56	31.059	2545	Un-identified	-	m/z 135, 73, 147, 221, 207
57	31.639	2601	Hexadecamethyl-heptasiloxane	33	Silicone oligomers fragment
58	31.753	2612	Un-identified Note 1	-	m/z 135, 197, 73, 465
59	32.085	2641	Octadecamethyl-cyclononasiloxane	91	Silicone oligomers fragment
60	32.623	2696	Octadecamethyl-cyclononasiloxane	45	Silicone oligomers fragment
61	32.914	2725	Octadecamethyl-cyclononasiloxane	45	Silicone oligomers fragment
62	33.774	2808	Hexadecamethyl-heptasiloxane	33	Silicone oligomers fragment
63	34.116	2842	Octadecamethyl-cyclononasiloxane	86	Silicone oligomers fragment
64	35.225	2948	Un-identified Note 1	-	m/z 135, 73, 147, 207, 221
65	36.665	3088	Tetracosamethyl-cyclododecasiloxane	64	Silicone oligomers fragment
66	38.147	3230	Un-identified Note 1	-	m/z 135, 73, 207, 147, 221
67	39.940	3403	Octadecamethyl-cyclononasiloxane	90	Silicone oligomers fragment
68	40.230	3431	Hexadecamethyl-heptasiloxane	53	Silicone oligomers fragment
69	41.847	3586	Un-identified	-	m/z 207, 73, 135, 147, 281
70	44.137	3807	Tetracosamethyl-cyclododecasiloxane	72	Silicone oligomers fragment
71	45.132	3903	Hexadecamethyl-heptasiloxane	59	Silicone oligomers fragment

Note 1 Un-identified compounds but related fragments

Peaks in **bold** text appear in the solvent blank

Table 4 n-heptane extract

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
1	4.726	6	Hexamethyl-cyclotrisiloxane	90	-
2	4.913	23	Un-identified	-	m/z 43, 55, 73, 85
3	4.975	29	Un-identified	-	m/z 43, 55, 73, 85
4	5.182	50	Tetrahydro-2H-Pyran-2-methanol	64	-
5	5.255	56	2-ethyl-3-propyloxirane	53	-
6	5.327	63	Cis-2,3-epoxyheptane	38	-
7	5.576	87	Tetrahydro[2,2']bifuranyl-5-one	78	-
8	5.835	112	3-heptanone	90	-
9	5.897	118	2-heptanone	91	-
10	7.255	249	1,1,3,3,5,5,7,7-octamethyl-tetrasiloxane	93	-
11	7.400	262	Octamethyl-cyclotetrasiloxane	91	-
12	7.669	288	2,2,4,6,6-pentamethylheptane	72	-
13	10.011	514	Decamethyl-cyclopentasiloxane	91	-
14	10.167	530	Un-identified	-	m/z 267, 281, 73, 133, 207
15	10.529	565	Dodecamethyl-pentasiloxane	91	-
16	11.265	636	Un-identified	-	m/z 73, 327, 341, 401
17	11.431	652	Dodecamethyl-pentasiloxane	94	Silicone oligomers fragment
18	12.675	770	Dodecamethyl-cyclohexasiloxane	91	-
19	13.877	888	Tetradecamethyl-hexasiloxane	80	-
20	15.048	999	Dodecamethyl-pentasiloxane	40	Silicone oligomers fragment
21	16.022	1094	Unidentified	-	m/z 221, 73, 147, 207
22	17.162	1204	Tetracosamethyl-cyclododecasiloxane	32	-
23	17.939	1279	Un-identified	-	m/z 73, 221, 147, 207
24	18.986	1380	Octadecamethyl-cyclononasiloxane	83	-
25	19.670	1446	Hexadecamethyl-heptasiloxane	64	-
26	19.960	1474	Un-identified	-	m/z 327, 405, 156, 253, 343
27	20.613	1537	Octadecamethyl-cyclononasiloxane	46	-

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
28	21.245	1599	Un-identified	-	m/z 221, 73, 147,295, 281
29	21.348	1608	Un-identified Note 1	-	m/z 135, 197, 73, 329
30	22.084	1680	Tetracosamethyl-cyclododecasiloxane	49	Silicone oligomers fragment
31	22.696	1736	Tetracosamethyl-cyclododecasiloxane	46	Silicone oligomers fragment
32	22.893	1758	Un-identified Note 1	-	m/z 135, 197, 403, 73
33	23.421	1807	Tetradecamethyl-hexasiloxane	62	Silicone oligomers fragment
34	24.022	1869	Hexadecamethyl-heptasiloxane	42	Silicone oligomers fragment
35	24.406	1904	Bis[di(trimethylsiloxy)phenylsiloxy]-trimethylsiloxane	42	-
36	24.654	1927	Octadecamethyl-cyclononasiloxane	53	Silicone oligomers fragment
37	25.266	1985	Un-identified	-	m/z 221, 73, 147, 295, 207
38	25.815	2039	Tetracosamethyl-cyclododecasiloxane	64	Silicone oligomers fragment
39	26.406	2098	Hexadecamethyl-heptasiloxane	38	Silicone oligomers fragment
40	26.903	2143	Tetracosamethyl-cyclododecasiloxane	49	Silicone oligomers fragment
41	27.017	2156	Un-identified Note 1	-	m/z 135, 73, 147, 221, 209
42	27.504	2203	Hexadecamethyl-heptasiloxane	25	Silicone oligomers fragment
43	27.991	2246	Tetradecamethyl-hexasiloxane	87	Silicone oligomers fragment
44	28.261	2275	Un-identified Note 1	-	m/z 135, 73, 147, 221, 209
45	28.333	2282	Un-identified	-	m/z 451, 218, 389, 373, 156
46	28.696	2318	Hexadecamethyl-heptasiloxane	25	Silicone oligomers fragment
47	29.183	2365	Un-identified	-	m/z 221, 355, 147, 281, 429
48	29.639	2409	Un-identified Note 1	-	m/z 135, 73, 147, 221, 281
49	30.084	2452	Hexadecamethyl-heptasiloxane	25	Silicone oligomers fragment
50	30.250	2468	Un-identified	-	m/z 197, 135, 259
51	30.447	2487	Un-identified Note 1	-	m/z 135, 197, 73, 465
52	30.572	2499	Hexadecamethyl-heptasiloxane	22	Silicone oligomers fragment
53	31.214	2561	Octadecamethyl-cyclononasiloxane	43	Silicone oligomers fragment
54	31.794	2616	Hexadecamethyl-heptasiloxane	40	Silicone oligomers fragment
55	31.919	2628	Un-identified Note 1	-	m/z 135, 197, 73, 221, 465
56	32.250	2661	Octadecamethyl-cyclononasiloxane	87	Silicone oligomers fragment

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
57	33.100	2743	Un-identified Note 1	-	m/z 135, 73, 147, 207, 221
58	33.960	2825	Hexadecamethyl-heptasiloxane	40	Silicone oligomers fragment
59	34.313	2860	Octadecamethyl-cyclononasiloxane	72	Silicone oligomers fragment
60	35.453	2970	Un-identified Note 1	-	m/z 135, 73, 147, 207, 221
61	36.810	3100	Hexadecamethyl-heptasiloxane	53	Silicone oligomers fragment
62	36.934	3111	Octadecamethyl-cyclononasiloxane	86	Silicone oligomers fragment
63	40.240	3433	Octadecamethyl-cyclononasiloxane	90	Silicone oligomers fragment
64	40.551	3461	Hexadecamethyl-heptasiloxane	28	Silicone oligomers fragment
65	44.531	3846	Tetracosamethyl-cyclododecasiloxane	22	Silicone oligomers fragment
66	45.546	3943	Hexadecamethyl-heptasiloxane	39	Silicone oligomers fragment

Note 1 Un-identified compounds but related fragments

Peaks in **bold** text appear in the solvent blank

Table 5 Toluene extract

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
1	6.104	138	Ethylbenzene	95	-
2	6.198	147	1,3-dimethylbenzene	97	-
3	6.477	174	o-xylene	53	-
4	7.503	270	Un-identified	-	m/z 193, 207, 91, 73, 267
5	7.607	283	Octamethyl-cyclotetrasiloxane	91	-
6	7.897	311	2,2,4,6,6-pentamethylheptane	53	-
7	10.125	524	Decamethyl-cyclopentasiloxane	91	-
8	10.270	540	Un-identified	-	m/z 267, 281, 73, 91, 133
9	10.633	575	Dodecamethyl-pentasiloxane	90	-
10	11.369	646	Un-identified	-	m/z 327, 73, 91, 341, 401
11	11.535	662	Dodecamethyl-pentasiloxane	87	-
12	11.814	690	Un-identified	-	m/z 91, 327, 73, 193, 207
13	12.778	781	Dodecamethyl-cyclohexasiloxane	91	-
14	13.991	899	Tetradecamethyl-hexasiloxane	81	-
15	15.172	1011	Dodecamethyl-pentasiloxane	33	Silicone oligomers fragment
16	16.146	1106	Hexadecamethyl-heptasiloxane	50	-
17	17.286	1218	Tetracosamethyl-cyclododecasiloxane	52	-
18	18.063	1292	Tetracosamethyl-cyclododecasiloxane	27	Silicone oligomers fragment
19	19.110	1393	Octadecamethyl-cyclononasiloxane	74	-
20	19.794	1459	Hexadecamethyl-heptasiloxane	86	-
21	20.063	1485	Un-identified	-	m/z 327, 405, 156, 343, 253
22	20.737	1550	1,1,1,5,7,7,7-heptamethyl-3,3-bis(trimethylsiloxy)tetrasiloxane	43	-
23	21.369	1611	Hexadecamethyl-heptasiloxane	59	Silicone oligomers fragment
24	21.452	1619	Un-identified Note 1	-	m/z 135, 197, 479, 329, 73

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
25	22.208	1692	Tetracosamethyl-cyclododecasiloxane	50	Silicone oligomers fragment
26	22.820	1750	Hexadecamethyl-heptasiloxane	59	Silicone oligomers fragment
27	23.006	1768	Un-identified Note 1	-	m/z 135, 197, 73, 403, 341
28	23.545	1820	Un-identified	-	m/z 73, 355, 221, 147, 429
29	24.146	1879	Hexadecamethyl-heptasiloxane	10	Silicone oligomers fragment
30	24.509	1914	Un-identified Note 1	-	m/z 135, 73, 197, 209, 327
31	24.778	1939	Octadecamethyl-cyclononasiloxane	53	Silicone oligomers fragment
32	25.380	1998	Tetracosamethyl-cyclododecasiloxane	22	Silicone oligomers fragment
33	25.939	2051	Tetracosamethyl-cyclododecasiloxane	64	Silicone oligomers fragment
34	26.530	2109	Hexadecamethyl-heptasiloxane	33	Silicone oligomers fragment
35	27.027	2156	Octadecamethyl-cyclononasiloxane	59	Silicone oligomers fragment
36	27.131	2167	Un-identified Note 1	-	m/z 135, 73, 147, 221, 283
37	27.639	2215	Un-identified	-	m/z 221, 147, 73, 295, 369
38	28.126	2262	Octadecamethyl-cyclononasiloxane	72	Silicone oligomers fragment
39	28.437	2292	Un-identified	-	m/z 451, 218, 389, 373, 156
40	28.851	2333	Hexadecamethyl-heptasiloxane	25	Silicone oligomers fragment
41	29.245	2371	Un-identified	-	m/z 197, 135, 259, 451
42	29.349	2381	Tetradecamethyl-hexasiloxane	47	Silicone oligomers fragment
43	29.794	2424	Un-identified Note 1	-	m/z 135, 73, 147, 221, 281
44	30.271	2469	Un-identified	-	m/z 221, 147, 73, 295, 369
45	30.385	2481	Un-identified	-	m/z 197, 135, 259
46	30.592	2501	Un-identified	-	m/z 197, 135, 465, 73, 259
47	30.768	2517	Octadecamethyl-cyclononasiloxane	91	Silicone oligomers fragment
48	31.400	2579	Un-identified Note 1	-	m/z 135, 73, 147, 221, 207
49	32.022	2639	Hexadecamethyl-heptasiloxane	40	Silicone oligomers fragment
50	32.354	2670	Un-identified Note 1	-	m/z 135, 197, 73, 207, 465
51	32.489	2684	Octadecamethyl-cyclononasiloxane	87	Silicone oligomers fragment
52	33.328	2765	Un-identified Note 1	-	m/z 135, 73, 147, 221, 207

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
53	34.271	2856	Hexadecamethyl-heptasiloxane	50	Silicone oligomers fragment
54	34.623	2890	Octadecamethyl-cyclononasiloxane	72	Silicone oligomers fragment
55	37.318	3149	Octadecamethyl-cyclononasiloxane	90	Silicone oligomers fragment
56	40.758	3480	Un-identified	-	m/z 221, 147, 73, 281, 355
57	41.069	3511	Hexadecamethyl-heptasiloxane	38	Silicone oligomers fragment
58	45.163	3907	Octadecamethyl-cyclononasiloxane	72	Silicone oligomers fragment
59	46.230	4013	Hexadecamethyl-heptasiloxane	38	Silicone oligomers fragment

Note 1 un-identified compounds but related fragments

Peaks in **bold** text appear in the solvent blank

Table 6 Direct Headspace Analysis

Peak number	RT (min)	Scan number	Peak ID (Wiley07/Nist5)	% Fit	Comment
1	11.5	1096	Acetone	9	-
2	13.0	1239	Methoxytrimethylsilane	64	-
3	14.6	1392	Dimethylsilanol	78	-
4	16.0	1526	Carbon Disulfide	9	-
5	18.0	1715	Trimethylsilanol	78	-
6	18.2	1736	Methoxytrimethylsilane	78	-
7	20.2	1922	1,1,3,3-tetramethyldisiloxane	81	-
8	23.6	2251	1,1,1,3,3-pentamethyldisiloxane	80	-
9	25.5	2435	Cyclohexane	94	-
10	29.3	2794	2-methoxybenzoic acid methyl ester	43	-
11	31.3	2988	Toluene	76	-
12	31.6	3019	Unidentified	-	m/z 149, 133, 75
13	32.5	3100	1,1,3,3,5,5-hexamethyltrisiloxane	70	-
14	33.8	3224	Hexamethylcyclotrisiloxane	87	-
15	36.8	3511	Ethylbenzene	60	-
16	37.3	3560	1,3-dimethylbenzene (o/m/p-xylene)	95	-
17	38.9	3717	o/m/p-xylene	81	-
18	44.0	4197	1,1,3,3,5,5-hexamethyltrisiloxane	53	-
19	44.9	4286	octamethylcyclotetrasiloxane	91	-
20	46.3	4423	2,2,4,6,6-pentamethylheptane	50	-
21	51.1	4878	2,2,4,4,6,6,8,8,10,10-decamethylcyclopentasiloxane	46	-
22	55.4	5289	Dodecamethylcyclohexasiloxane	91	-
23	59.5	5679	1,1,3,3,5,5,7,7,9,9,11,11-dodecamethylhexasiloxane	40	-
24	64.4	6147	1,1,3,3,5,5,7,7,9,9,11,11,13,13-tetradecamethylheptasiloxane	25	-

Table 7 Quantification of volatile organics

	Peak RT	Peak Response	Peak Concentration (µg/g ppm)	Wiley07/NIST05 Identification
	Prep 1 (AA07)			
1	11.526	1787527	0.107	Acetone
2	16.064	953441	0.057	Carbon Bisulfide
3	25.548	11905608	0.711	Cyclohexane
4	29.240	1029064	0.061	2-methoxybenzoic acid methyl ester
5	31.320	1752667	0.105	Toluene
6	31.645	2017057	0.120	Unidentified
7	36.800	2168678	0.130	Ethylbenzene
8	37.281	8883004	0.530	1,3-dimethylbenzene (o/m/p-xylene)
9	38.944	3643856	0.218	o/m/p-xylene
10	46.326	3150583	0.188	2,2,4,6,6-pentamethylheptane
	Prep 2 (AA08)			
1	11.505	2159824	0.135	Acetone
2	25.548	15579873	0.972	Cyclohexane
3	29.281	1260895	0.079	2-methoxybenzoic acid methyl ester
4	31.331	1542741	0.096	Toluene
5	31.634	2047111	0.128	Unidentified
6	36.800	2484450	0.155	Ethylbenzene
7	37.281	9394702	0.586	1,3-dimethylbenzene (o/m/p-xylene)
8	38.944	4253436	0.265	o/m/p-xylene
9	46.326	2064747	0.129	2,2,4,6,6-pentamethylheptane

	Peak RT	Peak Response	Peak Concentration ($\mu\text{g/g}$ ppm)	Wiley07/NIST05 Identification
	Prep 3 (AA09)			
1	11.546	1388574	0.088	Acetone
2	25.538	15439040	0.978	Cyclohexane
3	29.292	1209338	0.077	2-methoxybenzoic acid methyl ester
4	31.331	979073	0.062	Toluene
5	31.645	2185112	0.138	Unidentified
6	36.789	2068264	0.131	Ethylbenzene
7	37.270	9737593	0.617	1,3-dimethylbenzene (o/m/p-xylene)
8	38.954	3698333	0.234	o/m/p-xylene
9	46.358	1528951	0.097	2,2,4,6,6-pentamethylheptane

APPENDIX 1

Analytical methodology for solvent extraction method

Column (Analytical)	30 m x 0.25 mm x 0.25 μ m J & W DB5-MS column
Flow rate	Helium @ 10 psi (constant)
Liner	Agilent Cat no. 5181-3316
Split/splitless	Splitless mode
Injector temperature	300°C
Injection volume	1 μ L
Purge vent	On @ 50 mL/min @ 0.6 min
Oven programme	50°C for 2 min, then 10°C/min to 300°C hold for 20 min
Transfer line	300°C
MSD settings	
Mass scan range	35 to 500 AMU
Scan rate	1.6 scans/second
Data collection	4.6 to 47 min

Analytical methodology for head-space for GC-MSD

Head-space parameters

Equilibration temperature	85°C
Equilibration time	60 min
Transfer line temperature	110°C
Sample pressure	Adjust vial pressure to >5psi above column head pressure
Pressure time	90 sec
Shaker	On
Needle temperature	110°C
GC cycle time	75 min
Injection time	12 sec
Withdrawal time	30 sec
Vial venting	On

GC parameters

Column (Analytical)	Agilent J&W, DB-1, 60m x 0.32mm, film thickness 5µm with 2 m retention gap
Liner	Silitek 1mm
Carrier gas	Helium @ 13psi constant
Injection type	Split
Split ratio	1:1
Injector temperature	250°C
Oven temperature	35°C for 10 minutes Then 5°C/min to 150°C hold for 10 minutes Then 15°C/min to 250°C hold for 1 minute Then 30°C/min to 280°C hold for 18.33 minutes
Transfer line temperature	250°C
MSD settings	
Mass scan range	35 to 500 AMU
Scan rate	1.6 scans/second
Data collection	4.6 to 37 min