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Date: **January 10, 2013**

To: **Alan Humphrey, Work Assignment Manager, ERT**

From: **J. Syslo, Analytical Support Chemist, SERAS**

Subject: **Results of Oil Fingerprint Analysis of Product & Sediment Samples using *Draft* SERAS GC/MS SOP# 1803**

Project: **Oil Spill Response Support Site: Enbridge Oil: WA# SER00017**

This document contains the analytical results and report for the following samples:

Chain(s) of Custody #s:	SERAS-017-02/01/12-0001, SERAS-017-02/08/12-0002 SERAS-017-02/21/12-0003, SERAS-017-02/23/12-0004 No. CRA 25320 (Conestoga-Rovers & Associates)
Analyses:	Oil Fingerprint Analysis & Evaluation
No. of Samples:	Seven
Matrix:	2 River bottom sediments, 4 product samples, and 1 sample of product that was recovered from the water fraction of a sediment sample.

This data package contains the results of six samples received by SERAS between 02/01/12 through 03/01/12 for Oil Fingerprint Analysis & Evaluation Draft SERAS GC/MS Method 1803. The 2 sediment samples contained both a sediment and water fraction. The seventh sample was generated at SERAS by skimming visible product from the water and identified as SERAS-017-0000. Preliminary results of the oil fingerprint evaluation were issued to the client as in a Summary of Work technical memo on 03/12/12. The fingerprint evaluation presented in the 03/12/12 memo was modified and submitted as the final fingerprint results in a technical report submitted to the client on 10/18/12. This oil fingerprint data package represents all the analytical work performed to support the results of the 10/18/12 technical memo.

ec A. Humphrey, M. Sprenger, V. Kansal, D. Miller, D. Killeen, and T.F. Miller.

cc Analyst: John Syslo
Central File: G. DePasquale

Case Narrative

Date: January 09, 2013
Project: Oil Spill Response Support Site: Enbridge Oil WA# SER0017
Subject: Results of Oil Fingerprint Analysis of Product & Sediment Samples using *Draft* SERAS GC/MS SOP# 1803

This report contains the analytical results and report for the following samples:

Chain(s) of Custody #s:	SERAS-017-02/01/12-0001, SERAS-017-02/08/12-0002 SERAS-017-02/21/12-0003, SERAS-017-02/23/12-0004 No. CRA 25320 (Conestoga-Rovers & Associates)
Analyses:	Oil Fingerprint Analysis & Evaluation
No. of Samples:	Seven
Matrix:	2 River bottom sediments, 4 product samples, and 1 sample of product that was recovered from the water fraction of a sediment sample.

This data package contains the results of six samples received by SERAS between 02/01/12 through 03/01/12 for Oil Fingerprint Analysis & Evaluation using procedures in draft SERAS SOP 1803, *Oil Characterization, Extraction and Analysis*. The 2 sediment samples contained both a sediment and water fraction. The seventh sample was generated at SERAS by skimming visible product from the water and identified as SERAS-017-0000. Preliminary results of the oil fingerprint evaluation were issued to the client in a Summary of Work technical memo on 03/12/12. The fingerprint evaluation presented in the 03/12/12 memo was modified and submitted as the final fingerprint results in a technical report submitted to the client on 10/18/12. This oil fingerprint data package represents all the analytical work performed to support the results of the 10/18/12 technical memo.

The documentation section of this report contains an Excel spreadsheet (Attachment 01) that lists a summary of all the samples received and work performed for this project. This data package represents the analytical work of the oil fingerprint analysis of the six samples received between 2/01/12 and 3/01/12

The two sediment samples were processed and dried by the Task Leader and submitted to the SERAS central lab and extracted according to the procedures listed in SOP 1803. At the request of the Task Leader, only a blank was required with each sample. The four product samples were extracted "as is" using the Waste Dilution method. Surrogates were added to the sample when it was weighed – before diluting with solvent. A waste dilution blank and duplicate was extracted/weighed with the product samples. This was done to satisfy the fingerprint QC as outlined in ASTM Method D-5739.

All the product samples were extracted and analyzed following the procedures outlined in SOP 1803. The SERAS method applies the ASTM D 5739 criteria for checking the resolution & mass discrimination, analysis of a reference oil, extracting a duplicate with each batch of sample, and bracketing each 12 hour sequence with a duplicate sample. The two sediment samples were initially extracted and analyzed for TPH, and fingerprint evaluation was requested after the TPH analysis. The GC/MS analytical procedures to analyze sample extracts for TPH and oil fingerprints are identical; the only difference is how the data is processed. The fingerprint specific quality control (QC) criteria (from ASTM D 5739) could not be applied to the sediment samples because the standards used to check mass discrimination, reference oil, and duplicate analysis were not analyzed with the sediment extracts.

The results of the fingerprint evaluation of the samples are presented in the results section of this report. The results and conclusions were driven by site-specific goals not listed in this analytical report. The fingerprints and other data used to support the conclusions are part of the results section.

The GC/MS system was calibrated on 05/16/11 using #2 Diesel fuel and the ICAL verified using a second source initial calibration verification (ICV) standard. The ICAL was used to quantitate the surrogate recoveries for the sediment extracts and the waste dilution extraction of the product.

The QC section of this report contains the results of the Resolution & Mass Discrimination check (Table 1.0), the tabulated surrogate recoveries and internal standard areas (Table 2.0). The results of the ICV for the ICAL are in Table 3.0.

All electronic tables and documents for this report are located on the SERAS network at the following location:
I:\Organics\SERAS_After_11-02-2009\Projects\0017 EnbridgeOil\ENO_fingerprintReport2012

J. Syslo - 01/11/13

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Summary of Analytical Method & Procedures for Oil Fingerprinting & TPH Analysis

This method outlines the analytical procedures to prepare samples for oil fingerprinting/oil characterization analysis using gas chromatography/mass spectrometry (GC/MS). The objective of oil characterization is to obtain diagnostic information about the composition of oil from careful examination and evaluation of the ion fingerprints of selected intrinsic hydrocarbon compounds present in the oil. Some common target analytes for fingerprinting are the saturated hydrocarbons, isoprenoids, terpanes, two- to six-ring Polynuclear Aromatic Hydrocarbons (PAH) and sulfur-heterocyclic hydrocarbons and their respective alkyl-substituted homologues, and with biomarkers classified as hopane and sterane compounds. When used correctly, the fingerprints can be used to discern and identify multiple and mixed products through diagnostic hydrocarbon ratio analysis to ascertain the source, or monitor transport, alteration or degradation of petroleum in the environment. The effective use of chemical fingerprinting as an interpretative tool to adequately identify and differentiate contaminant sources is a complex procedure, relying both on the experience of the analytical chemist and on the application of the observed or measured ratios between certain discriminating compounds. At SERAS, a GC/MS method (draft SERAS SOP 1803: *Oil Characterization, Extraction and Analysis*) has been routinely applied as an interpretative tool for oil fingerprint analysis and characterization or to monitor and quantify the effects of bioremediation of specific PAH compounds and Total Petroleum Hydrocarbon (TPH) as a whole.

Extraction Procedure: Waste Dilution Method

Samples which are received as product (oil), or soil and water samples which exhibit oil that is distinctly separate, or can be easily separated from the soil or water matrix are extracted by weighing 0.1 grams of oil and diluting to a 10mL final volume with dichloromethane. If the sample is received as an emulsion, or an oil/water emulsion floating on water, approximately 5.0 mL of the emulsion/water sample is centrifuged for approximately 30 minutes at 3500 rpm to separate the oil from the water. The oil is then weighed and extracted using the waste dilution process. Prior to GC/MS analysis, 20 ul of a 500 ppm solution of internal standard mix is added to 1.0 mL of extract before injection /analysis. Usually fingerprinting does not require the accurate quantitative analysis of specific concentrations of target analytes, or actual TPH concentration, and surrogate compounds are not added to samples processed by the waste dilution method for oil analysis. Concurrently, quality control (QC) samples such as, laboratory control samples (LCS) and matrix spike (MS) samples, are not necessary or relevant since the focus of fingerprint analysis is on qualitative pattern recognition and evaluation of the observed differences or similarities of the fingerprint patterns.

Extraction Procedure: Soil Samples

Thirty grams of sample is mixed with sufficient anhydrous sodium sulfate, and Soxhlet or Soxtherm extracted and concentrated to a final volume of 5 or 10 mL. Prior to extraction, each sample is spiked with 500 ul of a 400 ug/ml surrogate mixture containing anthracene-d_{10}, 5a-androstane, and triacontane-d62. Before GC/MS analysis, 1.0 mL of extract is spiked with 20 ul of a 500 ppm solution of an internal standard mix containing naphthalene-d_8, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}, tetradecane-d_{30}, hexatriacontane- d_{74} , and tetracosane-d_{50}. Although compound quantitation is not necessary, the surrogate recoveries are not used to validate the extraction process, but visually monitored as a diagnostic tool to assure that the oil sample was not altered or lost during the extraction process. LCS, or MS samples, may be added as an extra measure of quality assurance to monitor any changes that may affect the fingerprints from the extraction process.

Extraction Procedure: Water Samples

Water samples are first examined to determine if any visible product can be isolated and skimmed off the water and treated as waste dilution. Water samples with no apparent product, or product that is not removed by physical separation, are extracted using standard separatory funnel techniques. Prior to extraction, each sample is spiked with 500 ul of a 400 ug/ml surrogate mixture containing anthracene-d_{10}, 5a-androstane, and triacontane-d62. A sufficient amount of sample is extracted using a separatory funnel with methylene chloride and then concentrated to a final volume of 1.0 mL. Prior to analysis, 20 ul of a 500 ppm solution of internal standard mix is added to 1.0 mL of extract used for the analysis. Again, the surrogate recoveries are not recorded or used to validate the extraction process, and LCS or MS samples are optional to determine if the extraction procedure alters or has any observable effects on the fingerprints that may affect or hinder the evaluation process.

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GC/MS OPERATING PARAMETERS:

A Hewlett Packard 6890 with a 5975 GC/MS system was used in the selective ion mode for this fingerprint analysis. The instrument conditions are below:

Column	Zebron, ZB-5, 30 m X 0.25mm ID, 0.5 micron film
Initial temp.	40°C, hold for 1.0 min
Temp program	10°C/min to 310°, hold 30 min
Injection port temp.	300° C
Injection mode	Pulsed Split (8:1) , 1 ul injection Pulse Pressure 20.0 psi for 0.5 min. Gas saver ON at 0.6min., 20mL/min.
Transfer line temp.	325° C
Flow	1.2 mL/min EPC constant flow
Post Run	3.0 min with 3.0mL/min @325° oven,
SIM Mode	Samples analyzed at +150 EV above tune.
Three Ion Groups	(3.0 min. to 19.1 min.) 20 ions @dwell 20 (19.1 min. to 25.4 min.) 22 ions @dwell 20 (25.4 min. to 59.0 min.) 20 ions @dwell 20 Scan delay 3.0 min
Source temp.	280° C
Quad temp.	190° C

Traditional fingerprint analysis does not require a calibration range, only a standard to demonstrate that the analytical parameters are sufficient to completely separate C16, C17, and C18. Routinely, the GC/MS system analyzes a standard that consists of ~21 or more PAH combined with a series of n-alkanes ranging from C8 thru C40 with complete separation of all the saturated hydrocarbons (SHC) compounds.

When quantitation is required, an initial calibration range was run at 1.0, 5.0, 10.0, 25.0 and 50.0 ug/mL for the SHC, PAH, and surrogate compounds. The calibration range is used to quantitate surrogate compounds added during the water and soil extraction process, and to quantify the PAH, and SHC compounds during bioremediation studies or when accurate PAH and SHC information is requested or included in the scope of analytical work.

An analysis batch begins by injecting a 50 ug/ml DFTPP standard in linear scan mode, (the system tuned with PFTBA) so that spectra produced meets or passes the acceptable criteria detailed in the method. Then a calibration check standard of 10 µg/mL of the SHC and PAH compounds is analyzed and the responses or behavior of individual compounds evaluated to monitor the instrument stability. The compound responses of the calibration check (CC) are evaluated by comparing the CC response factors to the average response factors of the calibration curve. The calibration check analysis is not a necessary procedure or requirement for fingerprinting analysis, but is analyzed at SERAS to assure that the response for all the SHC and the PAH compounds remain consistent from day to day so that the most accurate fingerprints can be generated by the GC/MS system and repeated over a period of time. The percent difference (%D) and area responses are checked to determine if any analytical parameters are significantly altered when compared to the initial calibration. A %D of $\leq \pm 25\%$ is generally used as the limit to monitor stability, and corrective actions such as maintenance and/or recalibration required if it is determined that the frequency or number of compounds that fail to fall within the 25% will affect the GC/MS performance and ability to reproduce known or predictable fingerprints. Quantitative compound concentrations are not necessary for an oil fingerprint analysis, just evaluation of selected fingerprints which are relevant to the samples being evaluated.

SERAS defines an instrumental batch to be 12 hours after injection of DFTPP, when most laboratories adhere to a 24 hour time period. Samples analyzed outside the 12 hour clock only need to be re-injected if accurate quantitative data is required. Fingerprint samples that are analyzed past the 12 hour time period are not re-injected nor considered to be invalid, or flagged as non-compliant. Samples will be re-injected only if there is distinct and significant evidence that indicates that the GC/MS was not functioning properly, and performance compromised. The DFTPP and calibration checks ensure that the operating conditions remain consistent throughout the analysis so that the physical appearance and relative intensity of fingerprints can be repeated on a daily, or any given period of

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time.

In addition to the QC requirements mandated by both SERAS and NELAC, additional QC requirements listed in ASTM Method D-5739, *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry,* are applied to oil fingerprinting. The additional requirements included performing a resolution check and a mass discrimination mixture. The resolution and mass discrimination checks are used to monitor the analytical column performance by checking the resolution of the both compound pairs: n-C17/pristane and n-C18/phytane, and the ratio of three PAH's: napthalene, fluoranthene, and benzo(g,h,i) perylene. The exact details of these checks are listed in sections 7.3 through 7.4.6 in ASTM Method D-5739. The original ASTM procedures for the mass discrimination check require that the PAH mixture be analyzed in the linear scan mode, and that the nC17/pristane + nC18/phytane mixture be analyzed as a separate mixture. In order to comply with the method and to save time, SERAS has combined both QC checks by obtaining the necessary information from the 10ppm SHC/PAH calibration check, which contains all of the compounds of interest for both checks. The mass discrimination areas are taken from the Selective Ion Measurement (SIM) areas of the 10ppm SHC+PAH daily check. The information for both the resolution checks and mass discrimination (RESMD) are reported in a table which includes the results of several RESMD checks that were collected from other fingerprint projects and/or from other GC/MS systems used for oil fingerprinting at SERAS. The ongoing collective RESMD table assures that the SERAS GC/MS systems are performing and set up to produce data/fingerprints that are reproducible during a current project, and/or match data from previous or future projects.

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APPENDIX A: Oil Target Compound List, Quantitation Ion, and Quantitation Limits
Compounds Arranged with Corresponding Internal Standards
Soil Quantitation Limits Based on Wet Weight

Compound Name	Quant Ion	Retention Time	Quantitation Limits Soil (ug/Kg)	Water (ug/L)
d8-Naphthalene	136	12.56		
Naphthalene	128	12.62	330	10
2-Methylnaphthalene	142	14.30	330	10
C1N Isomers	142	(14-15)	330	10
C2N Isomers	156	(15-17)	330	10
C3N Isomers	170	(16-19)	330	10
C4N Isomers	184	(17.1-20.1)	330	10
Acenaphthylene	152	16.51	330	10
Acenaphthene	154	16.94	330	10
d10-Phenanthrene	188	20.53		
Fluorene	166	18.22	330	10
C1F Isomers	180	(19-20.5)	330	10
C2F Isomers	194	(20-22)	330	10
C3F Isomers	208	(21-21.3)	330	10
Dibenzothiophene	184	20.27	330	10
C1D Isomers	198	(20.5-22.5)	330	10
C2D Isomers	212	(21.5-24)	330	10
C3D Isomers	226	(22-26)	330	10
Phenanthrene	178	20.58	330	10
d10-Anthracene {surr}	188	20.67	330	10
Anthracene	178	20.71	330	10
d14-o-Terphenyl {surr}	244	21.46	330	10
2-Methylanthracene	192	21.96	330	10
C1P/A Isomers	192	(21-23)	330	10
C2P/A Isomers	206	(22-24.5)	330	10
C3P/A Isomers	220	(23-26)	330	10
5a Androstane {surr}	260	23.03	330	10
d12-Chrysene	240	27.06		
Fluoranthene	202	23.52	330	10
Pyrene	202	24.07	330	10
C1F/P Isomers	216	(24-26)	330	10
C2F/P Isomers	230	(25-28)	330	10
Benzo(a)Anthracene	228	27.03	330	10
Chrysene	228	27.12	330	10
C1C Isomers	242	(28-29.2)	330	10
C2C Isomers	256	(28-30)	330	10
d12-Perylene	264	30.35		
Benzo(b)fluoranthene	252	29.50	330	10
Benzo(k)fluoranthene	252	29.56	330	10
Benzo(e)pyrene	252	30.10	330	10

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Benzo(a)pyrene	252	30.22	330	10
Perylene	252	30.42	330	10
Indeno(1,2,3-cd)pyrene	276	33.25	330	10
Dibenzo(a,h)anthracene	278	33.31	330	10
Benzo(g,h,i)perylene	276	34.11	330	10
d30-Tetradecane	66	15.20		
Octane C8	85	5.12	330	10
Nonane C9	85	7.24	330	10
Decane C10	85	9.20	330	10
Undecane C11	85	10.98	330	10
Dodecane C12	85	12.60	330	10
Tridecane C13	85	14.09	330	10
Tetradecane C14	85	15.50	330	10
Pentadecane C15	85	16.82	330	10
Hexadecane C16	85	18.07	330	10
Heptadecane C17	85	19.25	330	10
Pristane	85	19.28	330	10
Octadecane C18	85	20.36	330	10
Phytane	85	20.43	330	10
d50-Tetracosane	66	25.72		
Nonadecane C19	85	21.42	330	10
Eicosane C20	85	22.44	330	10
Heneicosane C21	85	23.41	330	10
Docosane C22	85	24.34	330	10
Tricosane C23	85	25.22	330	10
Tetracosane C24	85	26.07	330	10
Pentacosane C25	85	26.90	330	10
Hexacosane C26	85	27.68	330	10
Heptacosane C27	85	28.45	330	10
Octacosane C28	85	29.18	330	10
Nonocosane C29	85	29.89	330	10
d62-Triacontane {surr}	66	30.23	330	10
d74-Hexatriacontane	66	37.67		
Triacontane C30	85	30.68	330	10
Hentriacontane C31	85	31.57	330	10
Dotriacontane C32	85	32.62	330	10
Tritriacontane C33	85	33.83	330	10
Tetratriacontane C34	85	35.29	330	10
Pentatriacontane C35	85	37.03	330	10
Hexatriacontane C36	85	39.10	330	10
Heptatriacontane C37	85	41.62	330	10
Octatriacontane C38	85	44.63	330	10
Tetracontane C40	85	52.69	330	10
d12-Chrysene				
C27 Baa-cholestane	217	32.30	330	10
d12-Perylene				
C30 17B(H),21a(H) hopane	191	32.73	330	10

TPH response factor is calculated using the sum of three internal standards: d30-Tetradecane, d50-Tetracosane and d74-Hexatriacontane

TPH	83 + 85	(5-55)	330 mg/Kg	10 mg/L

Fingerprints not quantitated

SHC's	85	(3-55)

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Naphthenes	83	(3-55)
SHC and isoprenoids	113	(3-30)
Acyclic Isoprenoids	183	(3-30)
Hopanes	191	(27-44)
Steranes	217	(27-44)
Gasoline Range Aromatics	91	(3-20)
	105	(3-20)

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INSTRUMENT CONTROL PARAMETERS: Slick2

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Tue Dec 06 18:18:47 2011

Control Information

Sample Inlet	GC
Injection Source	GC ALS
Mass Spectrometer	Enabled

Oven	
Equilibration Time	0.5 min
Oven Program	On
40 °C for 1 min	
then 10 °C/min to 310 °C for 25 min	
Run Time	53 min

Front Injector	
Syringe Size	10 µL
Injection Volume	1 µL
Solvent A Washes (PreInj)	2
Solvent A Washes (PostInj)	2
Solvent A Volume	8 µL
Solvent B Washes (PreInj)	2
Solvent B Washes (PostInj)	2
Solvent B Volume	8 µL
Sample Washes	1
Sample Wash Volume	8 µL
Sample Pumps	2
Dwell Time (PreInj)	0 min
Dwell Time (PostInj)	0 min
Solvent Wash Draw Speed	300 µL/min
Solvent Wash Dispense Speed	6000 µL/min
Sample Wash Draw Speed	300 µL/min
Sample Wash Dispense Speed	6000 µL/min
Injection Dispense Speed	6000 µL/min
Viscosity Delay	0 sec
Sample Depth	Disabled

Front SS Inlet He	
Mode	Pulsed Split
Heater	On 300 °C
Pressure	On 9.8343 psi
Total Flow	On 13.8 mL/min
Septum Purge Flow	On 3 mL/min
Gas Saver	On 20 mL/min After 0.6 min
Split Ratio	8 :1
Split Flow	9.6 mL/min
Injection Pulse Pressure	20 psi Until 0.5 min

Thermal Aux 2 {MSD Transfer Line}	
Heater	On
Temperature Program	On
325 °C for 0 min	
Run Time	53 min

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Column #1
Zebron, ZB-5Zebron, ZB-5
360 °C: 33 m x 250 µm x 0.5 µm
In: Front SS Inlet He
Out: Vacuum

(Initial)	40 °C
Pressure	9.8343 psi
Flow	1.2 mL/min
Average Velocity	38.765 cm/sec
Holdup Time	1.4188 min
Flow Program	Off
1.2 mL/min for 60 min	
Run Time	53 min

MS ACQUISITION PARAMETERS

General Information

Tune File	: Xtune.u
Acquistion Mode	: SIM

MS Information

Solvent Delay	: 3.00 min
EMV Mode	: Relative
Relative Voltage	: 200
Resulting EM Voltage	: 1718

[Sim Parameters]

GROUP 1

Group ID	: GRO/DRO						
Resolution	: High						
Plot 1 Ion	: 85.00						
Ions/Dwell In Group	(Mass,	Dwell)	(Mass,	Dwell)	(Mass,	Dwell)	
	(66.00,	20)	(78.00,	20)	(83.00,	20)	
	(85.00,	20)	(91.00,	20)	(105.00,	20)	
	(113.00,	20)	(123.00,	20)	(128.00,	20)	
	(136.00,	20)	(142.00,	20)	(152.00,	20)	
	(154.00,	20)	(156.00,	20)	(166.00,	20)	
	(170.00,	20)	(180.00,	20)	(183.00,	20)	
	(184.00,	20)	(193.00,	20)			

GROUP 2

Group ID	: DRO						
Resolution	: High						
Group Start Time	: 18.40						
Plot 1 Ion	: 85.00						
Ions/Dwell In Group	(Mass,	Dwell)	(Mass,	Dwell)	(Mass,	Dwell)	
	(66.00,	20)	(83.00,	20)	(85.00,	20)	
	(105.00,	20)	(113.00,	20)	(123.00,	20)	
	(178.00,	20)	(180.00,	20)	(183.00,	20)	
	(184.00,	20)	(188.00,	20)	(192.00,	20)	
	(194.00,	20)	(198.00,	20)	(202.00,	20)	
	(206.00,	20)	(208.00,	20)	(212.00,	20)	
	(216.00,	20)	(220.00,	20)	(226.00,	20)	
	(230.00,	20)	(244.00,	20)	(260.00,	20)	

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GROUP 3
Group ID                : DRO/UCM
Resolution              : High
Group Start Time        : 24.40
Plot 1 Ion              : 85.00
Ions/Dwell In Group     (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,  Dwell)
                        ( 66.00,     20) ( 83.00,     20) ( 85.00,     20)
                        (105.00,     20) (113.00,     20) (123.00,     20)
                        (177.00,     20) (183.00,     20) (191.00,     50)
                        (205.00,     20) (217.00,     20) (218.00,     20)
                        (228.00,     20) (230.00,     20) (231.00,     20)
                        (240.00,     20) (242.00,     20) (252.00,     20)
                        (256.00,     20) (264.00,     20) (276.00,     20)
                        (278.00,     20)

[MSZones]

MS Source               : 280 C   maximum 300 C
MS Quad                 : 190 C   maximum 200 C

                    END OF MS ACQUISITION PARAMETERS

                     TUNE PARAMETERS for SN: US90432092
                  ----------------------------

 Trace Ion Detection is OFF.

 EMISSION     :      34.610
 ENERGY       :      69.922
 REPELLER     :      25.432
 IONFOCUS     :      80.667
 ENTRANCE_LE  :       0.000
 EMVOLTS      :    1517.647
                             Actual EMV  :    1717.65
                             GAIN FACTOR :       2.57
 AMUGAIN      :    1857.000
 AMUOFFSET    :     123.313
 FILAMENT     :       1.000
 DCPOLARITY   :       0.000
 ENTLENSOFFS  :     14.000@  3    18.000@ 50    15.800@ 69    14.200@131
15.500@219    15.800@414    14.800@502    17.000@1049
 MASSGAIN     :    -629.000
 MASSOFFSET   :     -36.000

                    END OF TUNE PARAMETERS
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                END OF INSTRUMENT CONTROL PARAMETERS
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Results of the GC/MS Oil Fingerprint Analysis on Enbridge Oil Sediments, Product and Biodegradation Studies

The samples and fingerprints were evaluated by direct visual comparison of selected fingerprints and backed with information provided in the following document: Chun Yang, Zhendi Wang, Zeyu Yang, Bruce Hollebone, Carl E. Brown, Mike Landriault , Ben Fieldhouse, *Chemical Fingerprints of Alberta Oil Sands and Related Petroleum Products*, environmental forensics, 12:2, 173-188 (2011) .

Based on the evaluation of the fingerprints the following conclusions can be made from examining the fingerprints:

- **Exhibit EX01** shows the hopane fingerprints for samples 000, 003, 004, 005 and 006. Using the ion 191 hopane fingerprints, all the product samples originate from a common source.
- **Exhibit EX02** shows the hopane fingerprint for sample 000 compared to the fingerprints from sediment samples 001 and 002. This confirms the presence of 000 in the extracted sediment. The anomalous peaks in the two sediment fingerprints are due to naturally occurring hopanes in the sediment.
- **Exhibit EX03** shows the total TPH fingerprint for the non-sediment samples. Samples 003, and 006 are exact matches to each other, while 005 shows a slight difference in the nC17/pristane and nC18/phytane ratios. Sample 004 is a mechanically/evaporative weathered version of 003 to 006, and sample 000 is a match of 004 that has undergone biodegradation.
- **Exhibits EX04:** Shows the total methyl- and dimethyl-chrysene fingerprints for sample 004 and sediment sample 001. The sediments were contaminated with a second source of PAH that masked *all* of the PAH and alkyl-PAH fingerprints from the spilled oil and could not be used.
- **Exhibit EX05** shows the total TPH of sample 003, 004 and 000 which illustrate the fresh unweathered crude, mechanically weathered crude and the mechanically and biodegraded form of the crude oil.
- Sediment samples 001 and 002 contain a mixture of sulfur and TPH that is identified as naturally occurring background TPH. The hopane fingerprint of **EX02** supplies the only conclusive evidence that oil from samples 0000, or any of the crude oil is present.

I was asked to supply evidence of the "degradability" of the Enbridge Oil sample 0000. Based on my experience with degradation studies of similar oils I've provided fingerprints that show day 0 and day 84 of an oil mixture that is similar in composition to sample 0000. **Exhibits EX06A** show how a similar oil was degraded in an 84 day biodegradation study. **Exhibit EX06B** shows sample 0000. The shape and overall appearance of the unresolved complex mixture (UCM) changed from day 0 to day 84 and we would expect the same results and degradation effects on the chromatograms of **EX06B.**

- **Exhibit EX07** show the TPH fingerprints for the two sediment extracts and source oil samples 017-0000 and 017-0004. It is difficult to determine whether either of the source oils is present using just the TPH fingerprint and that was determined by examining trace biomarkers fingerprints within the source oils and finding those fingerprints within the oil extracted from the river sediments. This set of fingerprints was used to illustrate that the TPH fingerprint of the oil extracted from the sediments indicate that this "oil" is composed mostly of naturally occurring background hydrocarbons derived from plants and other organic material.
- **Exhibits EX08a & EX08b** show the fingerprints of sample 0004 before and after processing and that baking (autoclaving) samples 0003 and 0004 had little effect on the actual TPH fingerprint.
- **Exhibit EX09** shows the TPH fingerprint of sample 0000 and the effects of the 14 and 28 day biodegradation studies performed on the processed oil from sample 0004. The biodegradation effectively "cleaned up" the oil to expose and highlight just the highly resistant biomarker compounds.

Fingerprint Conclusion: Samples 0003 and 0006 are exact matches of each other and exhibit very little weathering. Sample 0005 is a very close match to 0003 and 0006, and shows a slight variation in the nC17/pristane and nC18/phytane ratios when compared to 0003 and 0006. This change is due to the loss of some of the n-alkanes which are the first compounds to be affected by biodegradation. Sample 0004 has a similar nC17/pristane and nC18/phytane ratio that matches 0005 and has been determined to be a mechanically weathered version of sample 0005, and 0005 a slightly weathered version of samples 0003 & 0006. Sample 0000 retains just the pristane/phytane peaks and ratio that is observed in sample 0005. Several distinct biomarker compounds that are present samples 0000 and 0004 are observed in the TPH that was extracted from the sediment samples 0001 and 0002.

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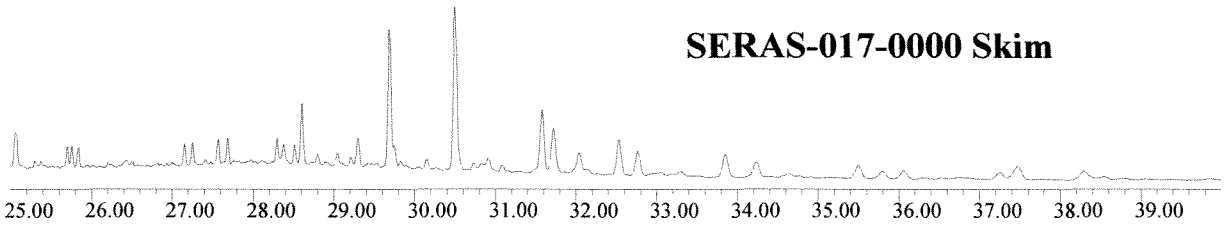
Fingerprint Exhibits

US EPA ARCHIVE DOCUMENT

EX01: Ion 191 Hopane Fingerprints

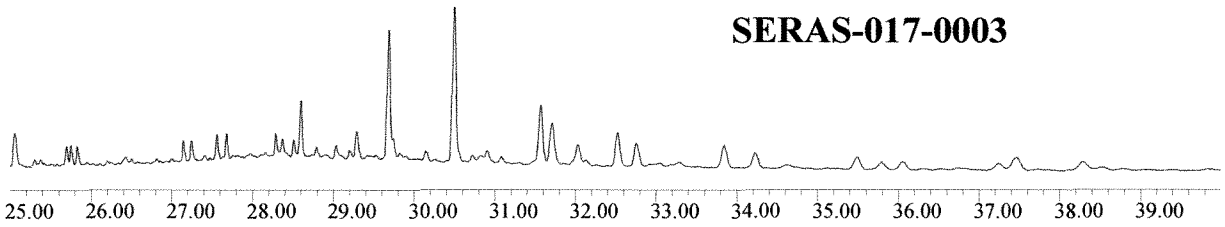
Ion 191.00 (190.70 to 191.70): SL02878.D\data.ms (*)

SERAS-017-0000 Skim



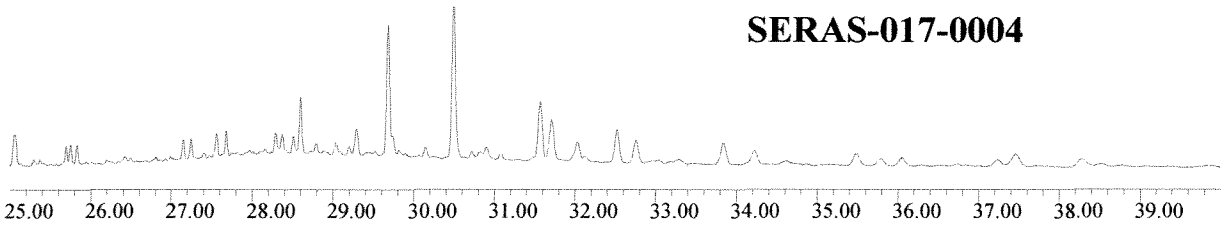
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SERAS-017-0003



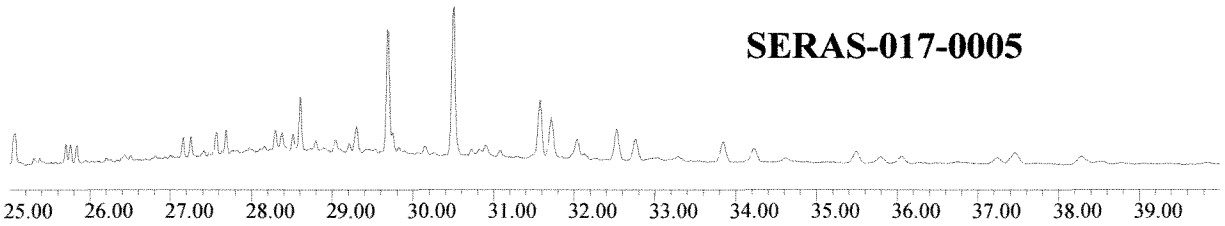
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SERAS-017-0004



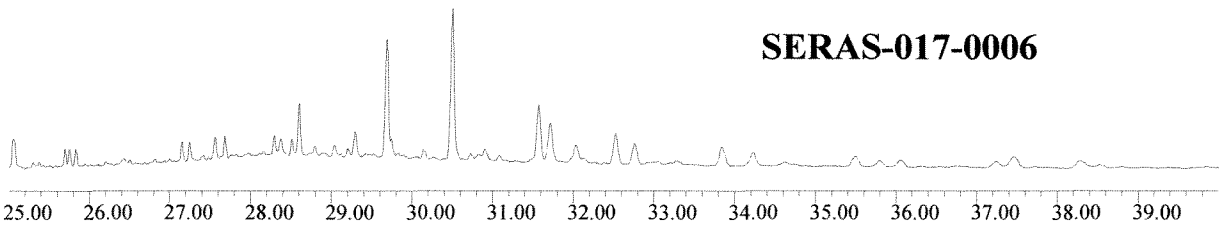
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SERAS-017-0005



Ion 191.00 (190.70 to 191.70): SL02892.D\data.ms (*)

SERAS-017-0006

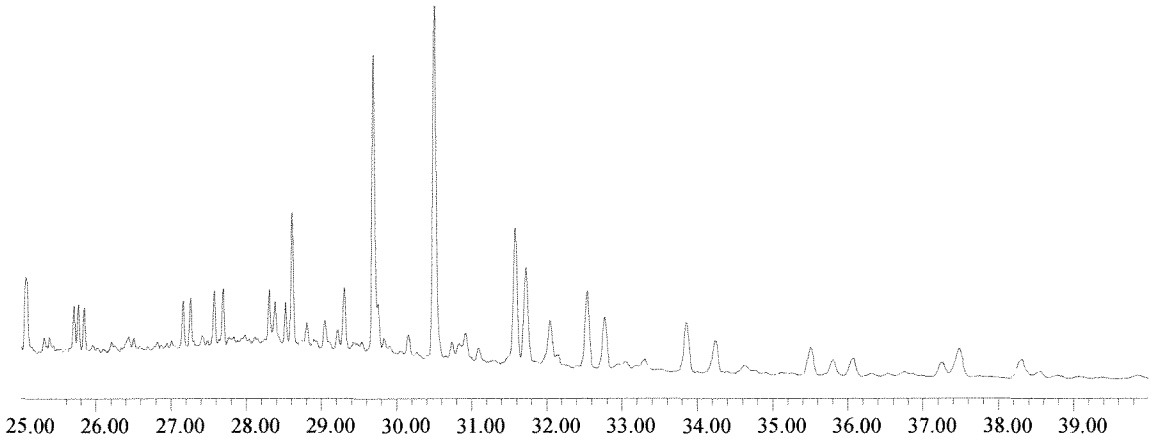


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EX02: Ion 191 Hopane Fingerprints

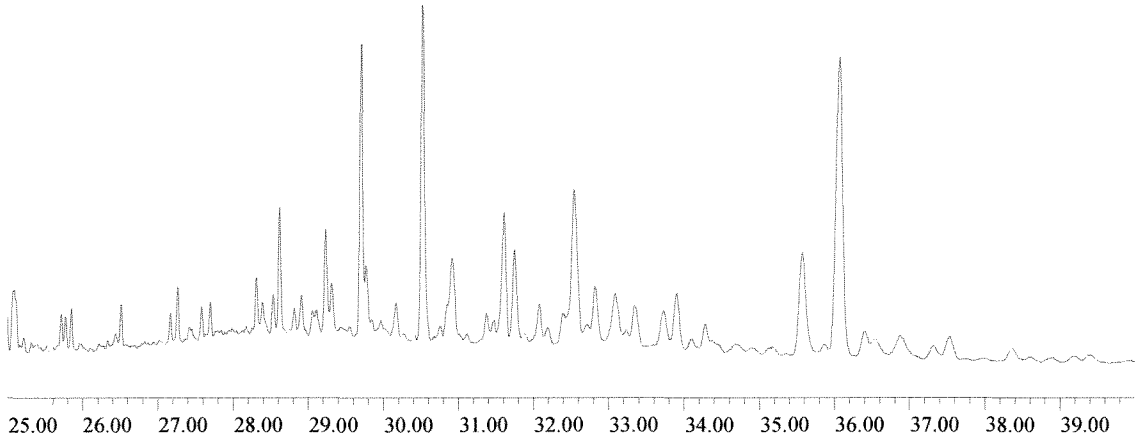
SERAS-017-0000 Skim

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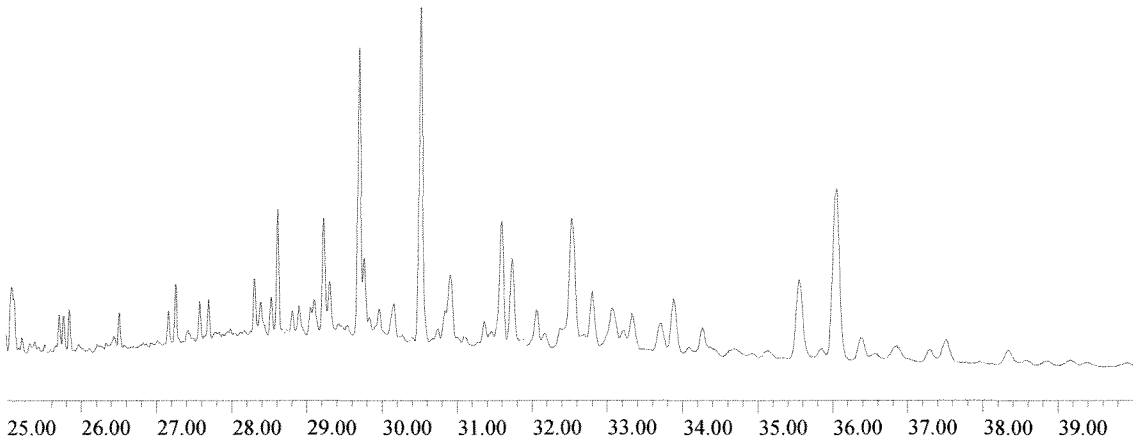
SERAS-017-0001 Sediment

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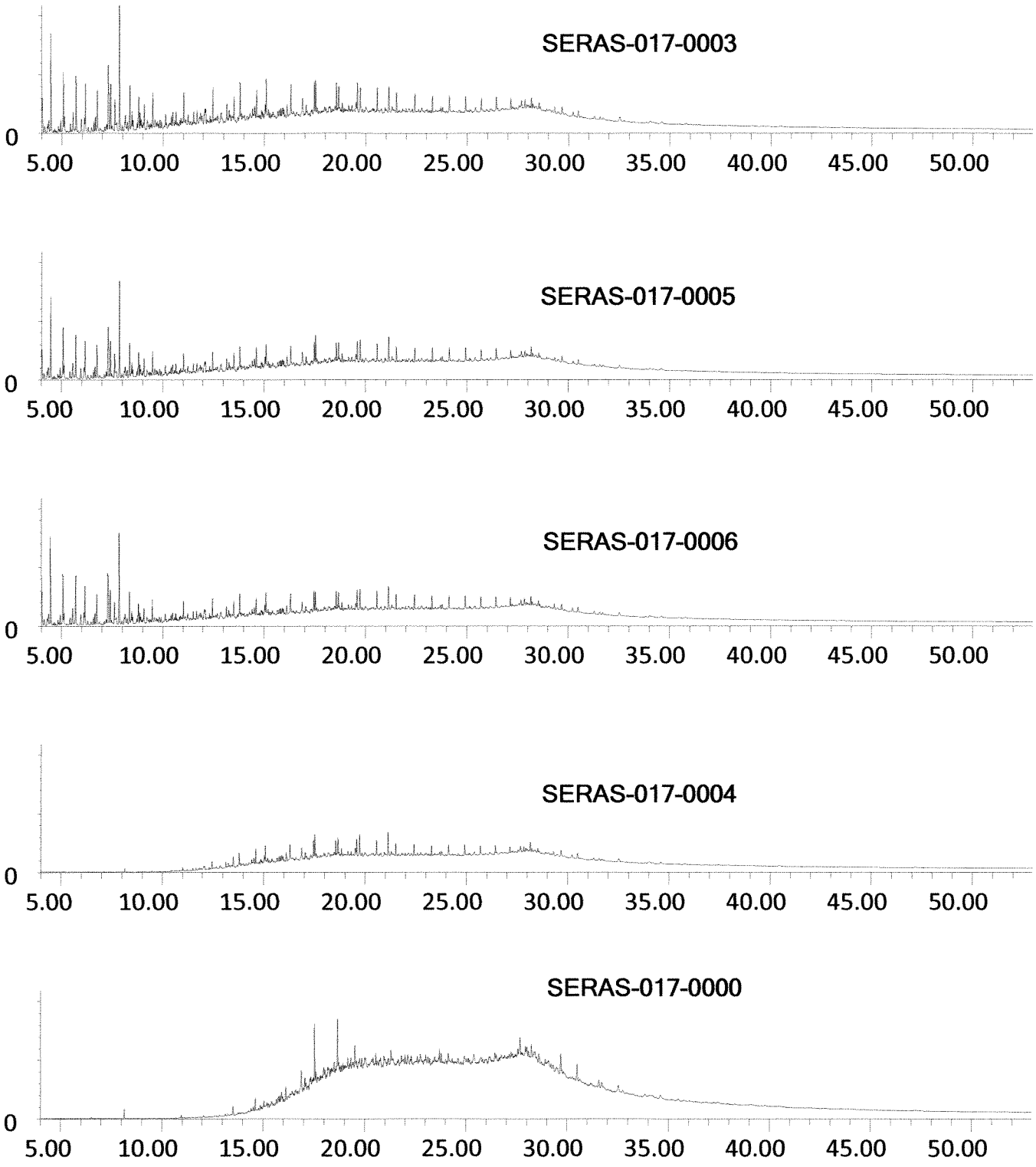
SERAS-017-0002 Sediment

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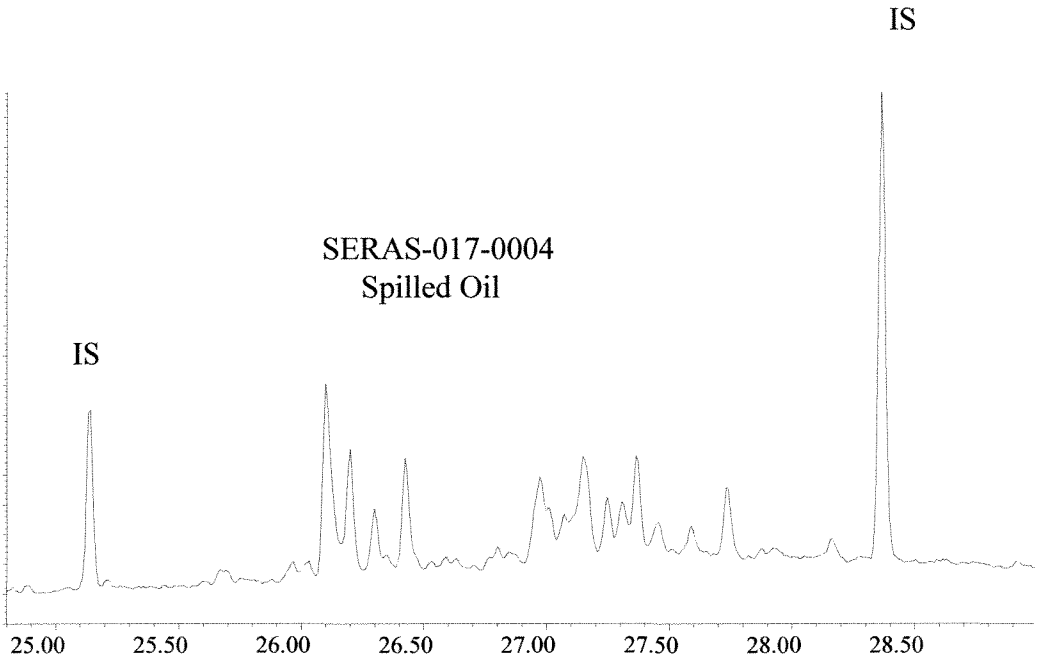
US EPA ARCHIVE DOCUMENT

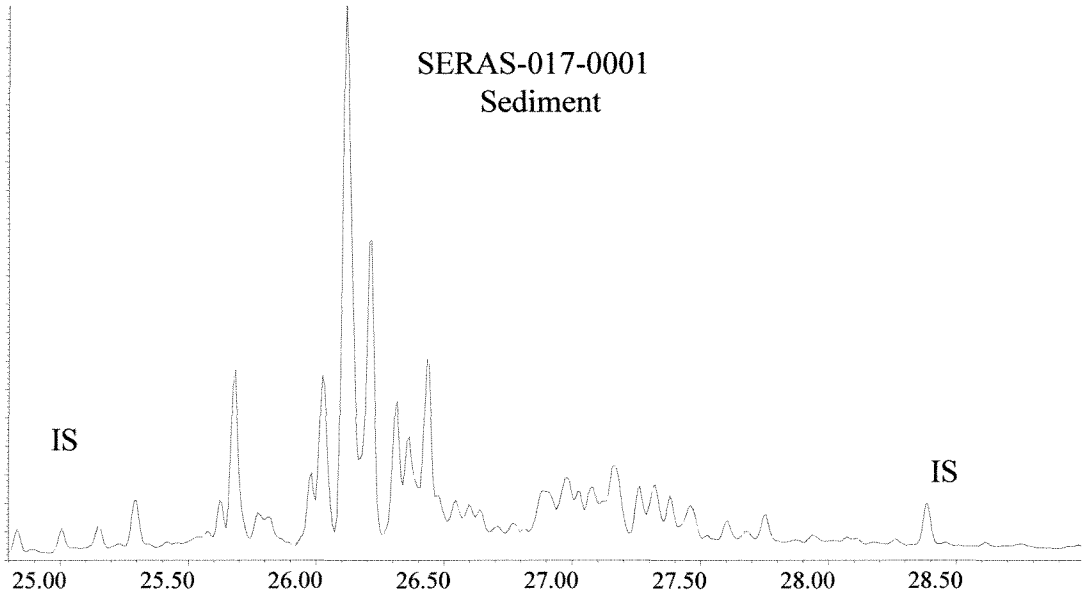
EX03: TPH Fingerprints



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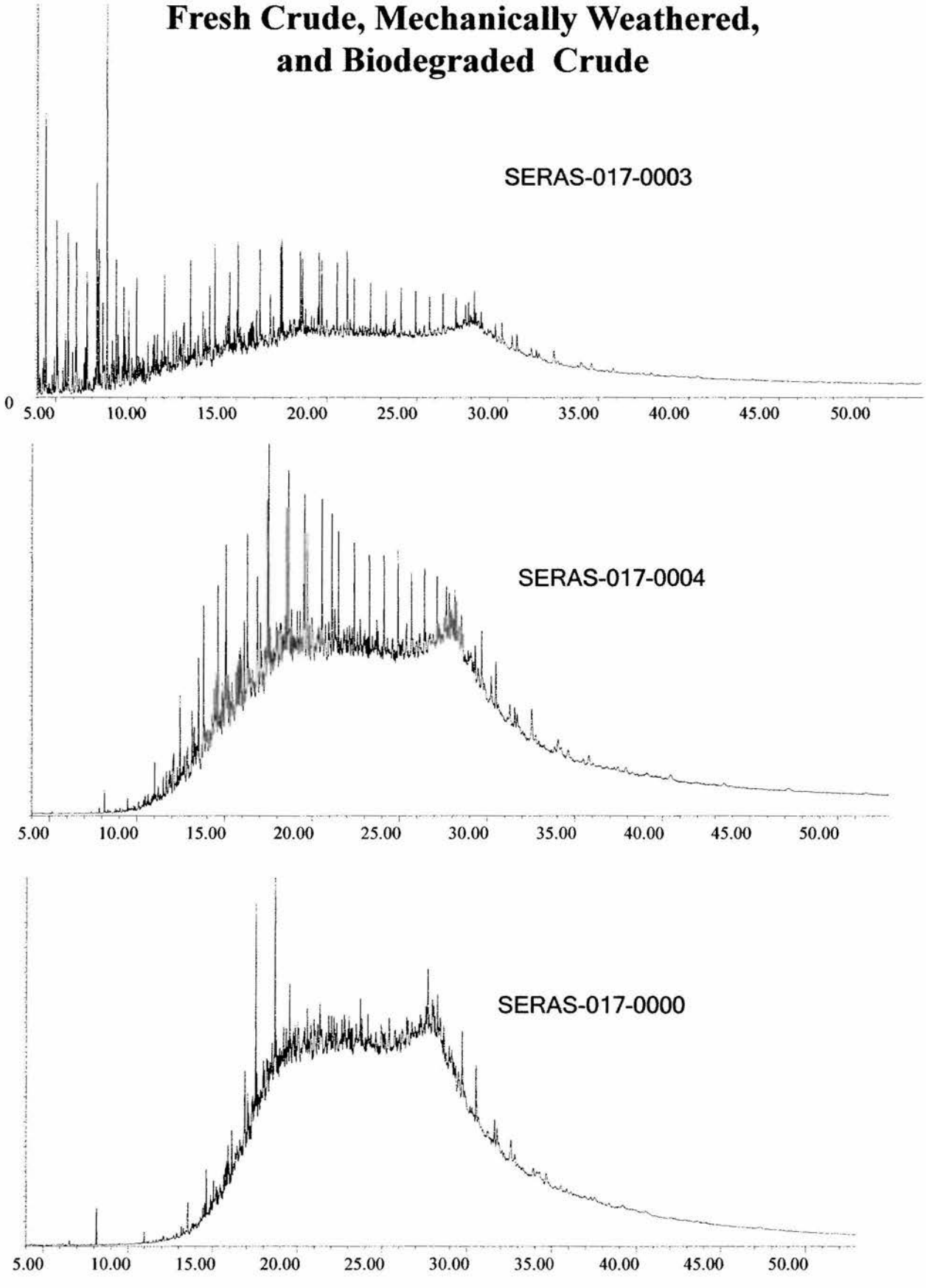
EX04: Total methyl- & dimethyl-Chrysene Fingerprints





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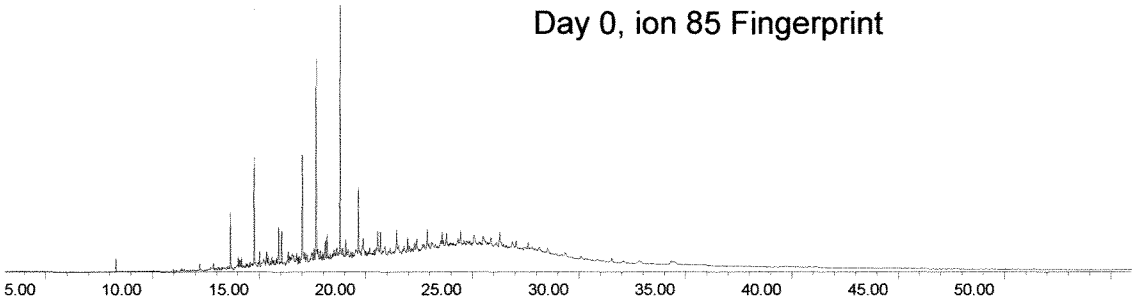
EX05: TPH Fingerprints Illustrating Fresh Crude, Mechanically Weathered, and Biodegraded Crude



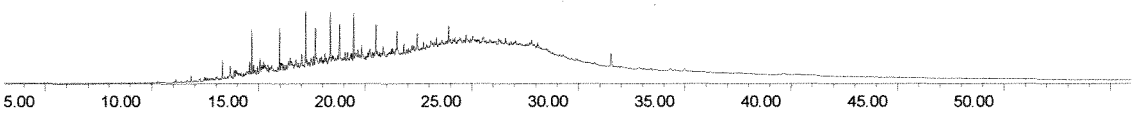
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EX06A: Biodegradation of Ion 85 & ion 83 Compounds During an 84 Day Degradation Study

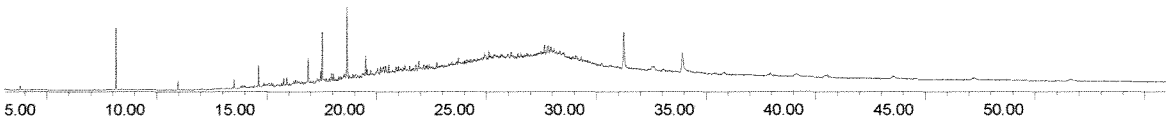
Day 0, ion 85 Fingerprint



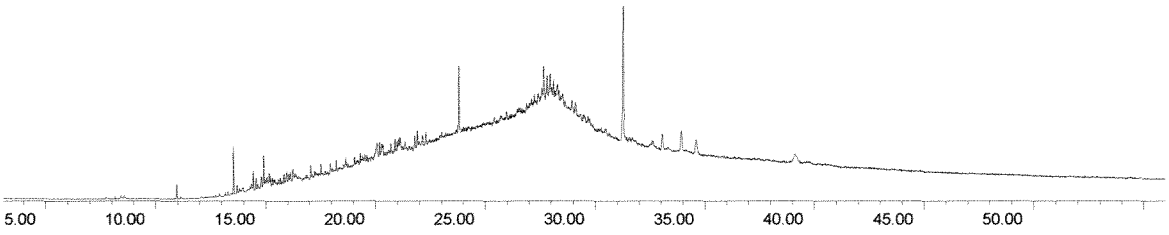
Day 0, ion 83 Fingerprint



Day 84, ion 85 Fingerprint



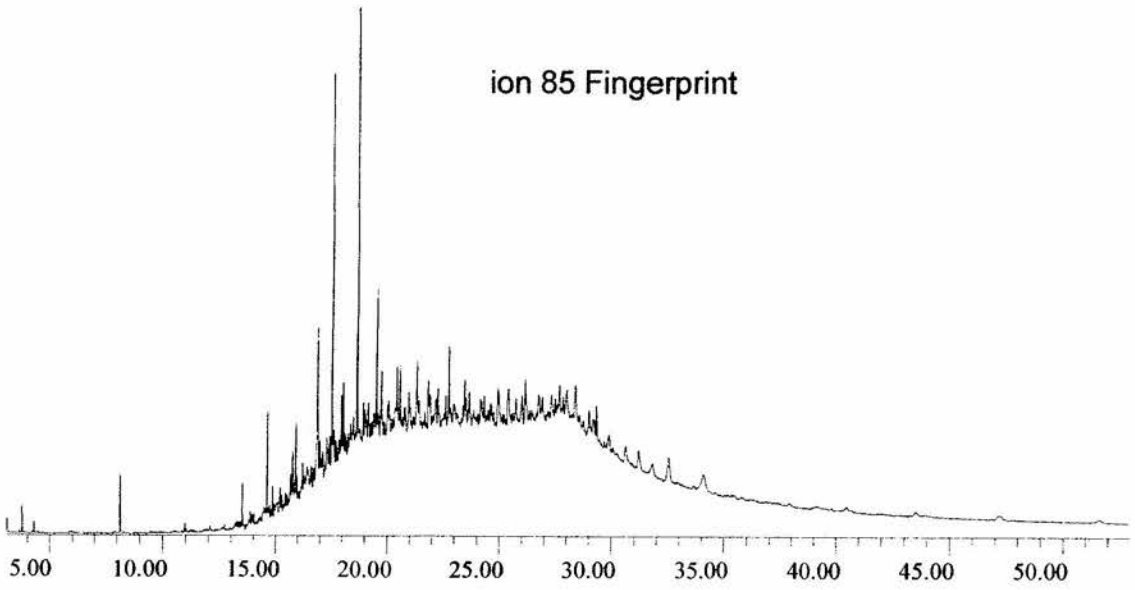
Day 84, ion 83 Fingerprint



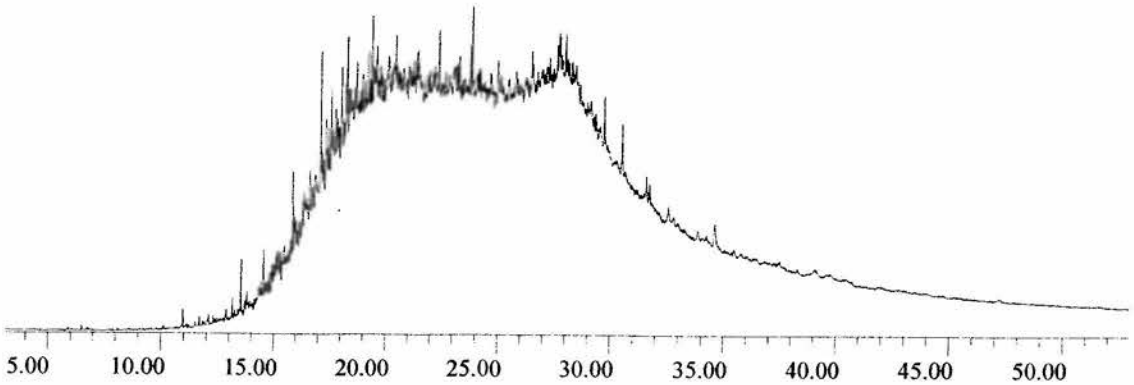
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EX06B: Sample 017-0000 Ions 85 & 83 Fingerprints

ion 85 Fingerprint

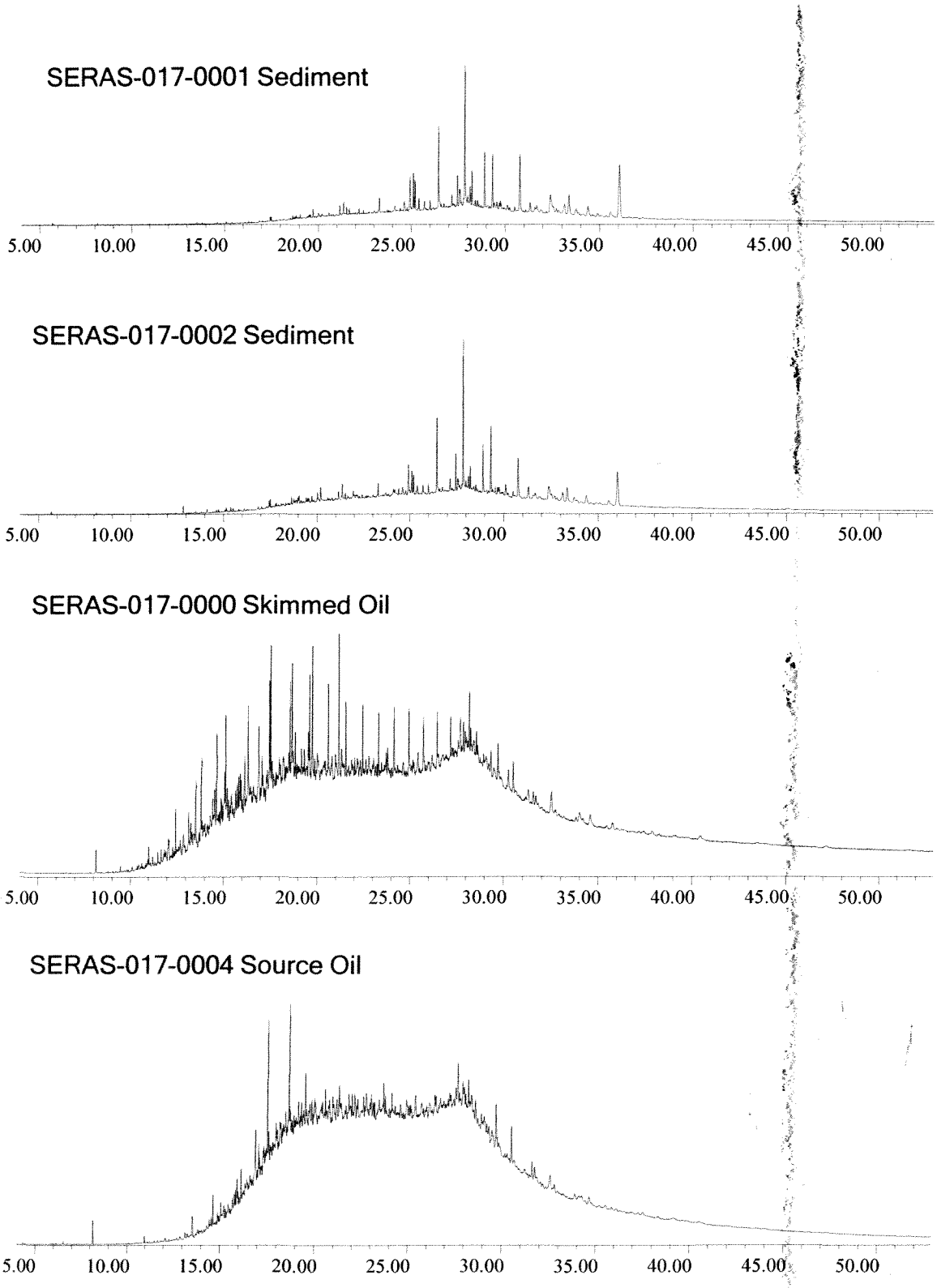


ion 83 Fingerprint



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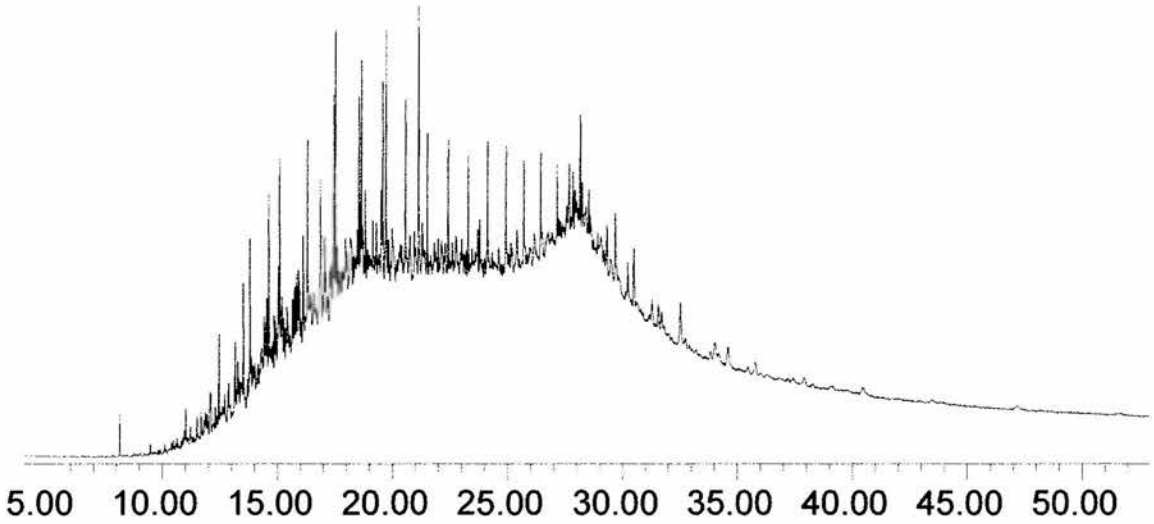
EX07: TPH Fingerprints of the two Sediments Samples Compared to Spilled Oil Samples Illustrating Natural or Introduced "Background Organics"



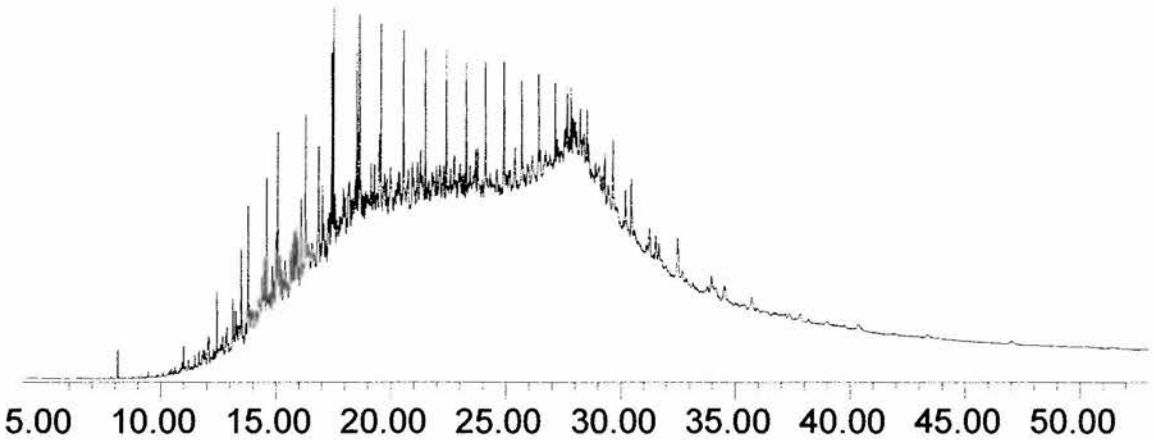
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EX08A: Illustrates Fingerprint of Unprocessed Spilled Oil and Fingerprint of Processed and Autoclaved Oil: Sample 017-0004

Sample 017-0004; Unprocessed Sample



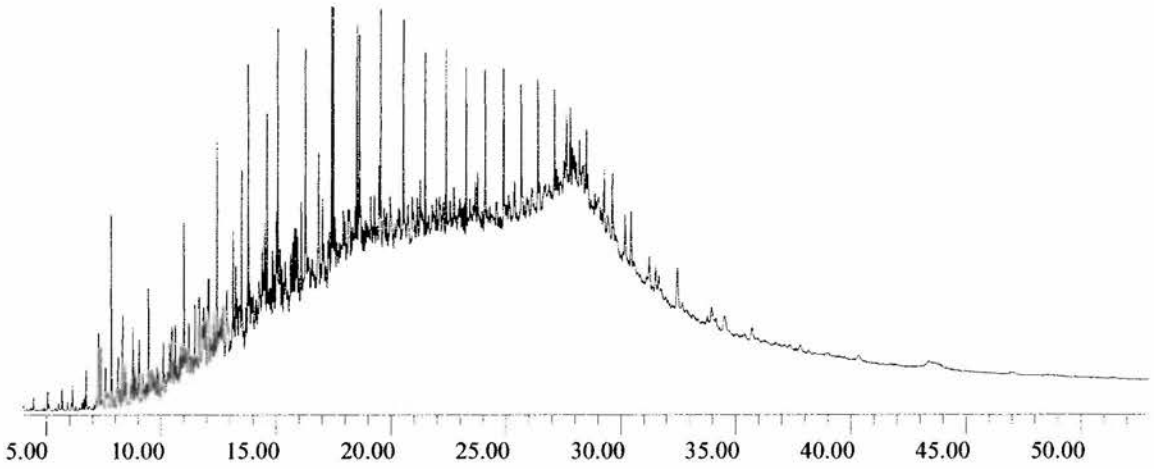
Sample 017-0004; Processed and Autoclaved



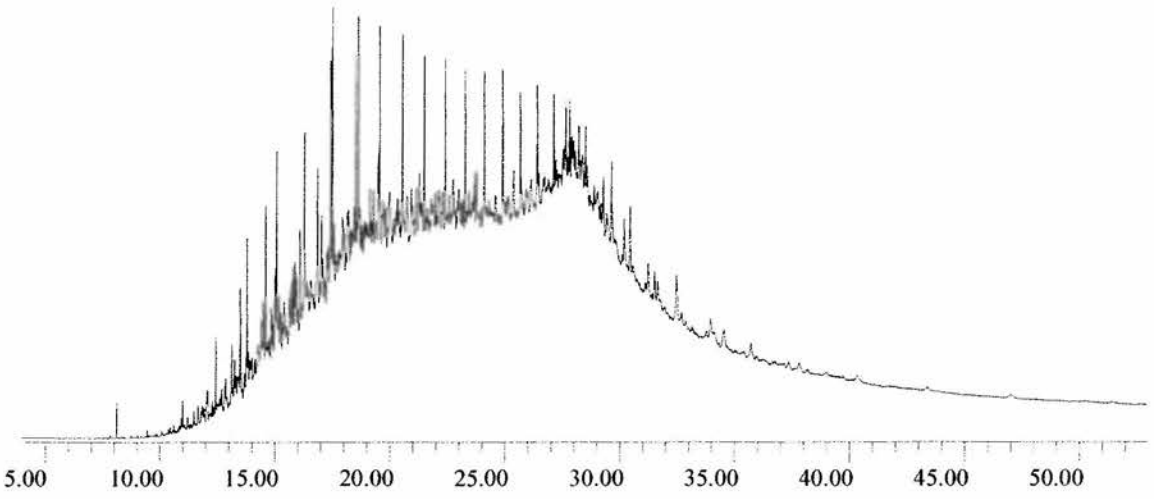
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EX08B: Illustrates Fingerprint of Unprocessed Fresh Crude Oil and Fingerprint of Processed and Autoclaved Oil: Sample 017-0003

Sample 017-0003: Unprocessed Sample

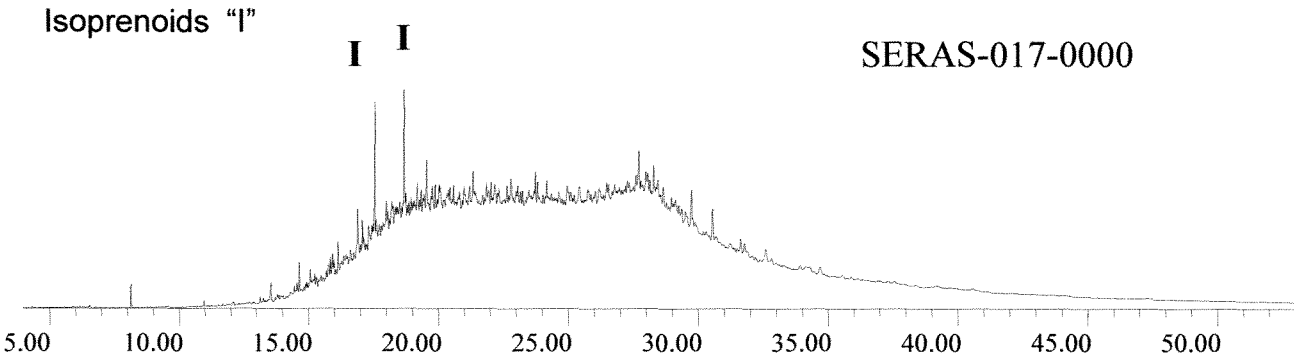


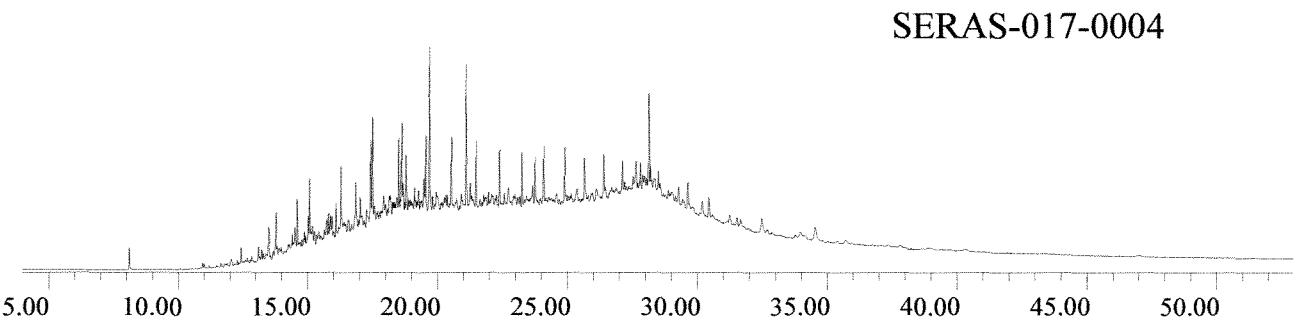
Sample 017-0003: Processed & Autoclaved

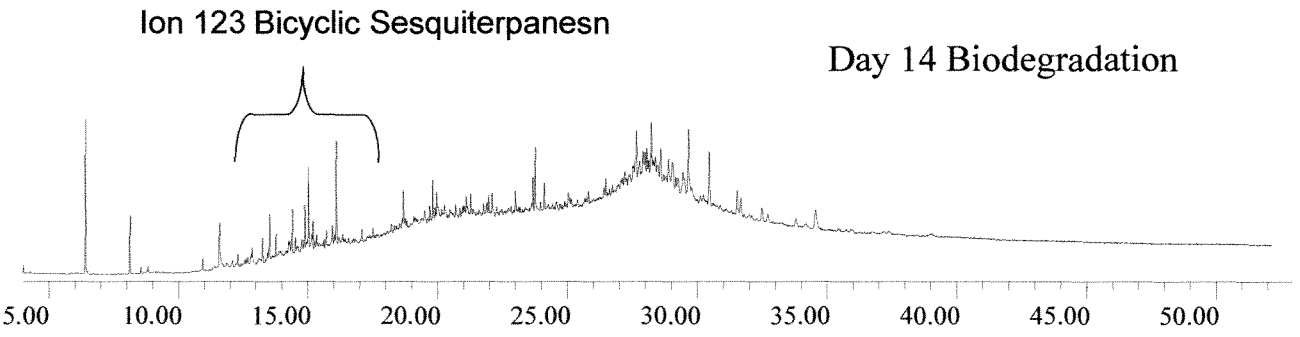


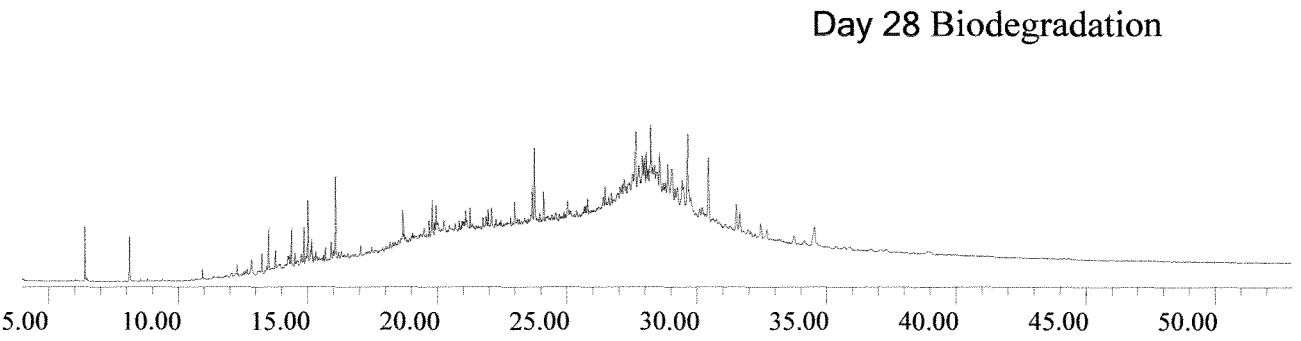
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EX09: TPH Fingerprints









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Table 1.0: Results of ASTM D-5739 Resolution and Mass Discrimination Performance Checks Applied to *draft* SERAS GC/MS Method 1803: Oil Fingerprinting, Characterization and TPH Analysis

Enbridge Oil: Fingerprinting 2012: WA# 0-017

Method: *draft* SERAS SOP 1803

		Resolution Check[1]			Mass Discrimination Checks							Resolution Check: Sysio/SERAS Benzo(b)/Benzo(k) fluoranthene ratio
Date Checked	GC/MS File	C17/pristane % Resolution	C18/phytane % Resolution	Acceptable % Resolution	Naphthalene area	Fluoranthene area	Benzo(g,h,i)perylene area	Nap/Fluo Ratio area/area	B(ghi)/Fluo Ratio area/area	Acceptable Ratios Nap/Fluo	B(ghi)/Fluo	
Enbridge Oil TPH/Fingerprinting: WA# 0-017												
2/23/2012	SL02855[3]	99.8%	99.9%	> 50% v/v	1100101	1358009	1167661	0.81	0.86	<= 2.0	>= 0.20	68.70%
2/24/2012	SL02855[2]	98.5%	99.7%	> 50% v/v	454731	543693	439965	0.84	0.81	<= 2.0	>= 0.20	68.43%
2/27/2012	SL02872[2]	98.5%	99.7%	> 50% v/v	507043	611080	485460	0.83	0.79	<= 2.0	>= 0.20	65.80%
3/1/2012	SL02883[2]	99.0%	99.4%	> 50% v/v	506865	655953	569653	0.77	0.87	<= 2.0	>= 0.20	63.55%
Lab Maintenance Samples: 0-011												
5/23/2011	SL01907	97.94	98.85	> 50% v/v	487005	429769	381358	1.13	0.89	<= 2.0	>= 0.20	62.83
5/24/2011	SL01920	99.04	99.87	> 50% v/v	545444	544337	421740	1.00	0.77	<= 2.0	>= 0.20	61.02
DRO-TPH Soil MDL Study August 11, 2011 on Slick2 - After moving instrument to new lab.												
8/11/2011	SL02081	99.05	99.65	> 50% v/v	504208	637067	494345	0.79	0.78	<= 2.0	>= 0.20	61.40
Imperial Oil fingerprints: November - December 2011 on Slick 2												
11/14/2011	SL02274[2]	98.92	99.85	> 50% v/v	891148	1110386	936738	0.80	0.84	<= 2.0	>= 0.20	63.93
12/1/2011	SL02332[2]	98.14	100.28	> 50% v/v	158336	197959	135116	0.80	0.68	<= 2.0	>= 0.20	59.12
12/2/2011	SL02339[2]	99.01	99.82	> 50% v/v	1666437	2018693	1619360	0.83	0.80	<= 2.0	>= 0.20	67.52
12/3/2011	SL02364[2]	99.06	99.88	> 50% v/v	1246967	1470915	1193650	0.85	0.81	<= 2.0	>= 0.20	68.84
12/5/2011	SL02367[2]	99.04	99.82	> 50% v/v	1281093	1527311	1134178	0.84	0.74	<= 2.0	>= 0.20	69.13
12/6/2011	SL02382[2]	98.87	99.87	> 50% v/v	1152427	1408258	1191425	0.82	0.85	<= 2.0	>= 0.20	67.25
CUC Oil fingerprints: December 2011 on Slick 2												
12/7/2011	SL02403[2]	99.93	99.73	> 50% v/v	1323413	1547091	1278611	0.86	0.83	<= 2.0	>= 0.20	68.49
12/9/2011	SL02419[2]	99.08	99.71	> 50% v/v	1202251	1425463	1171714	0.84	0.82	<= 2.0	>= 0.20	67.22
12/13/2011	SL02441[2]	98.92	99.76	> 50% v/v	1686396	2010249	1665781	0.84	0.83	<= 2.0	>= 0.20	66.62
12/14/2011	SL02456[2]	99.11	99.76	> 50% v/v	1180708	1397756	1127432	0.84	0.81	<= 2.0	>= 0.20	64.46

All resolution and mass discrimination checks are performed using a 10ug/mL PAH & SHC standard unless otherwise noted.

(1) The resolution check is determined using the Enviroquant ChromEval option in the Qedit mode.
(2) Resolution check performed using 10ppm SHC/PAH mixture in SIM mode.
(3) Resolution check performed using 25ppm SHC/PAH mixture in SIM mode.

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TABLE 2.1: Results of the Internal Standard Areas & Surrogate Recoveries

for Oil Fingerprint Analysis of Product Samples using Draft SERAS GC/MS Method 1803/1841

Enbridge Oil: WA# 0=017

SERAS draft TPH SOP# 1841
and SERAS draft Fingerprint SOP# 1803

Internal Standard Areas

Sample Number	GC/MS File	d30-Tetradecane	d50-Tetracosane	d74-Hexatriacontane
Analysis Date: 02/27/12				
1000ppm TPH ICAL Std. CCV	*SL02871*	*165200*	*202882*	*161132*
BLK022412WD	SL02874	100642	116775	65767
SERAS-017-0003	SL02875	99120	125874	118393
SERAS-017-0003 dup	SL02876	103998	130631	119796
SERAS-017-0004	SL02877	96470	122101	113788
SERAS-017-0000	SL02878	227918	283369	257581
SERAS-017-0003 EOS	SL02879	101381	126388	115104
Analysis Date: 03/01/12				
1000ppm TPH ICAL Std. CCV	*SL02885*	*109515*	*150757*	*134865*
BLK030112WD	SL02886	73201	95670	87506
SERAS-017-0005	SL02890	97344	131670	115625
SERAS-017-0005 dup	SL02891	94462	129000	116518
SERAS-017-0006	SL02892	94522	131524	117275

Surrogate Recoveries

Sample Number	d10-Anthracene	o-Terphenyl-d14	5a-Androstane	d62-Triacontane
BLK022412WD	88	99	108	108
SERAS-017-0003	87	96	104	112
SERAS-017-0003 dup	80	88	95	102
SERAS-017-0004	80	88	101	104
SERAS-017-0000	NS*	NS*	NS*	NS*
SERAS-017-0003 EOS	87	95	109	109
BLK030112WD	89	96	107	114
SERAS-017-0005	93	88	102	103
SERAS-017-0005 dup	74	80	87	96
SERAS-017-0006	78	83	91	99

NS* = "Not Spiked" : Sample comsisted of recovered produt floating on water. Required concentration < 1.0mL for analysis.

Table 3.0: Results of Initial Calibration Verificaiton (ICV) for DRO-TPH as #2 Diesel

REAC Method 1803: GC/MS Oil Characterization & TPH

Calibration Date: 05/16/11 - GC/MS ID-File DROTPH051611.M

ICV Prepared at 1000 µg/mL ; GC/MS File SL01878.D

Method: SERAS draft SOP#1841

Compound	Target Conc. µg/mL	ICV Conc. µg/mL	%Recovery	Tentative %Rec.Limits
DRO-TPH as #2 Diesel Fuel	1000	946	95	50-150 %

ICV LIMS# = RUE0049; 1000ppm #2 Diesel from 2nd Source Standard

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ASTM D 5739 Sample Analysis Batch Requirement

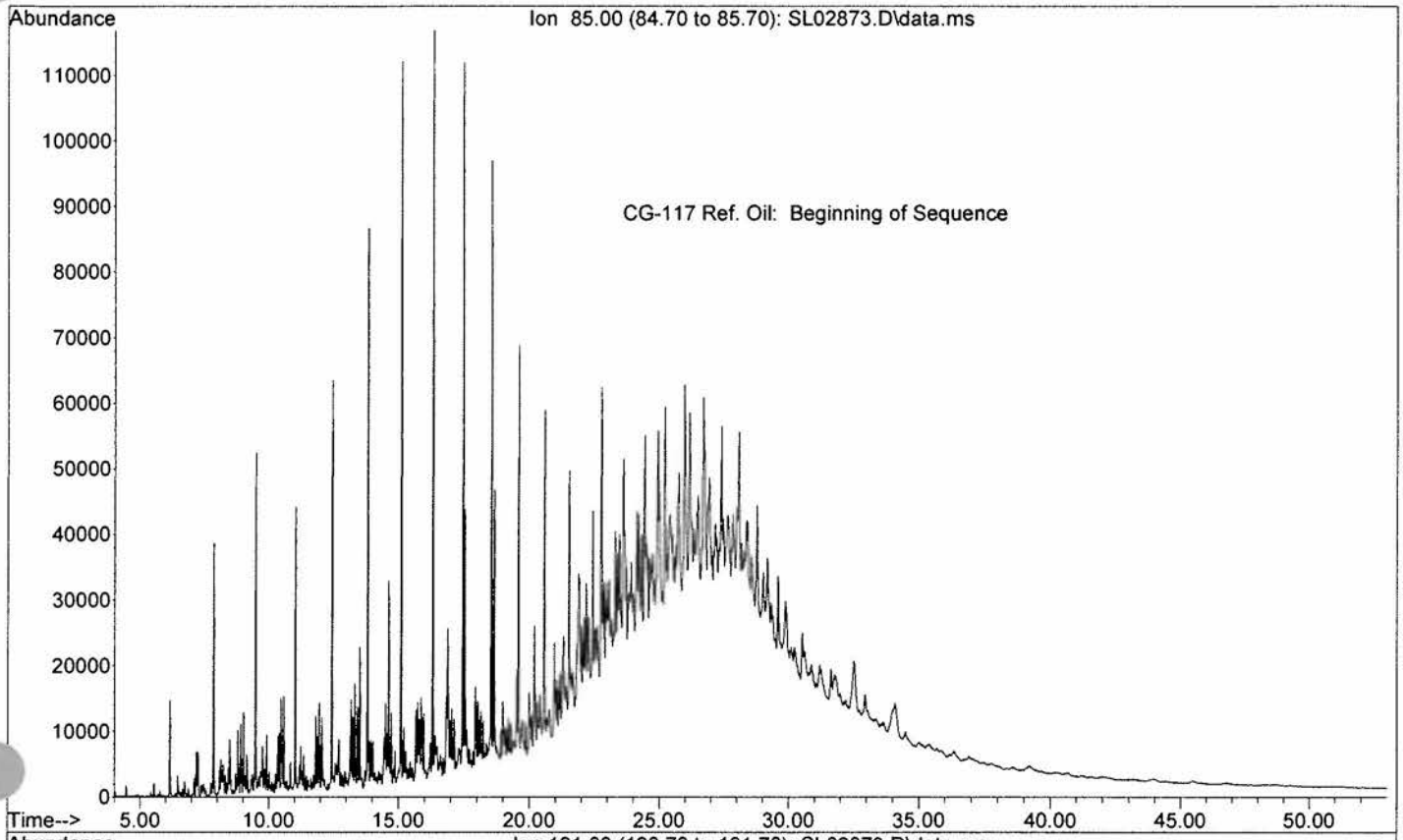
Sequence Bracket : Duplicate or (

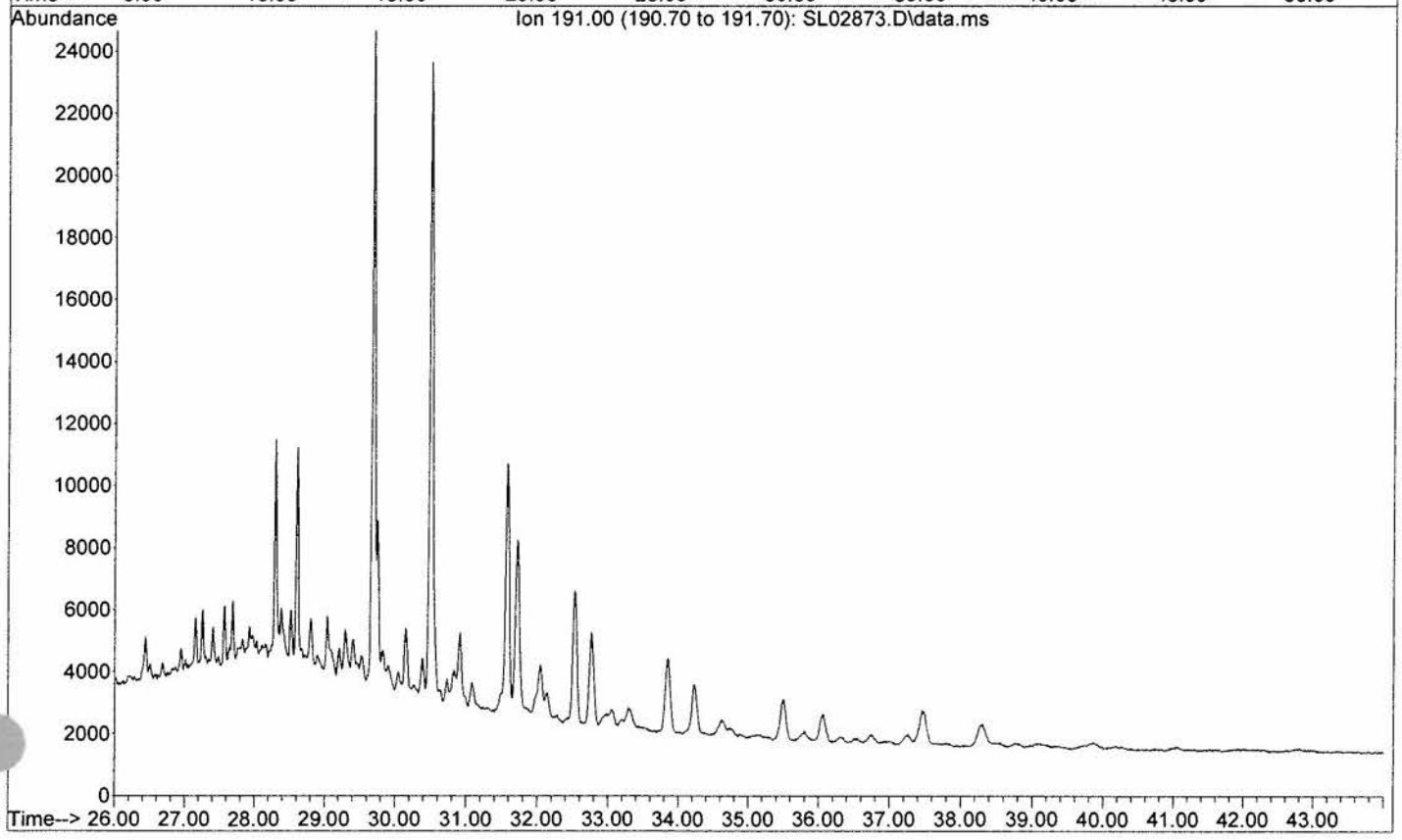
CG-117

For samples run 2/27/12 + 3/1/12

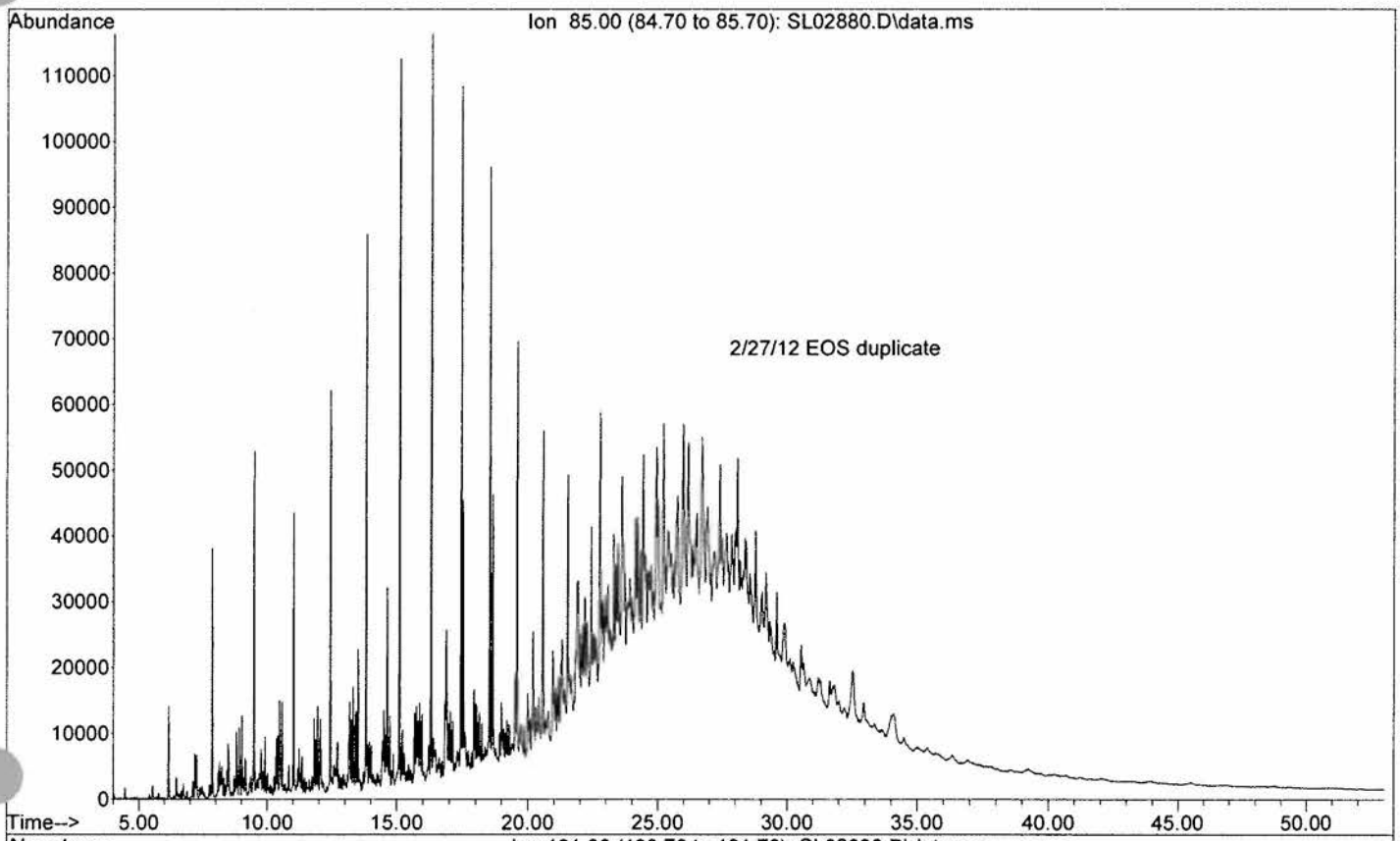
US EPA ARCHIVE DOCUMENT

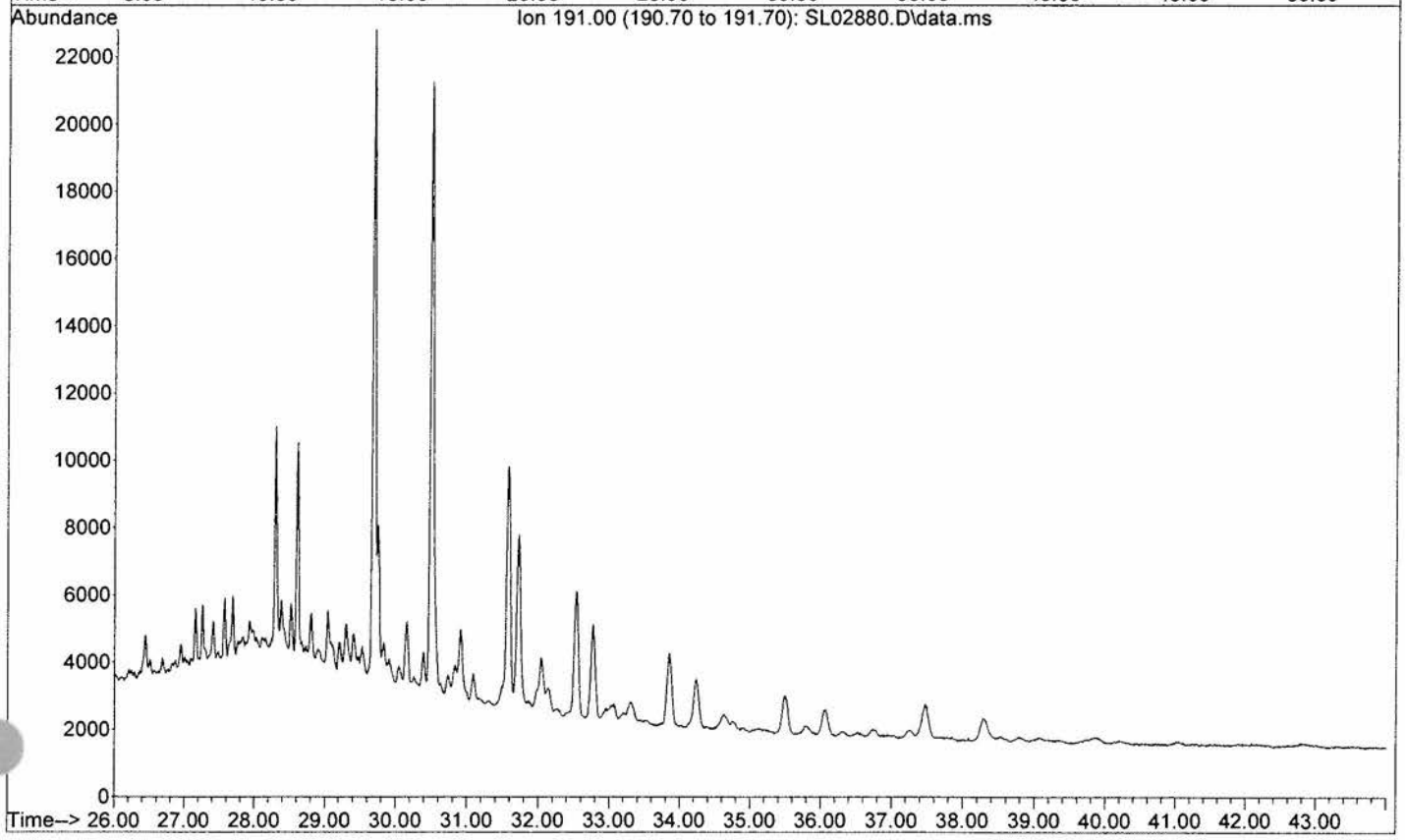
File :C:\msdchem\1\DATA\022712\SL02873.D
Operator : Syslo
Acquired : 27 Feb 2012 17:34 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003
Vial Number: 73



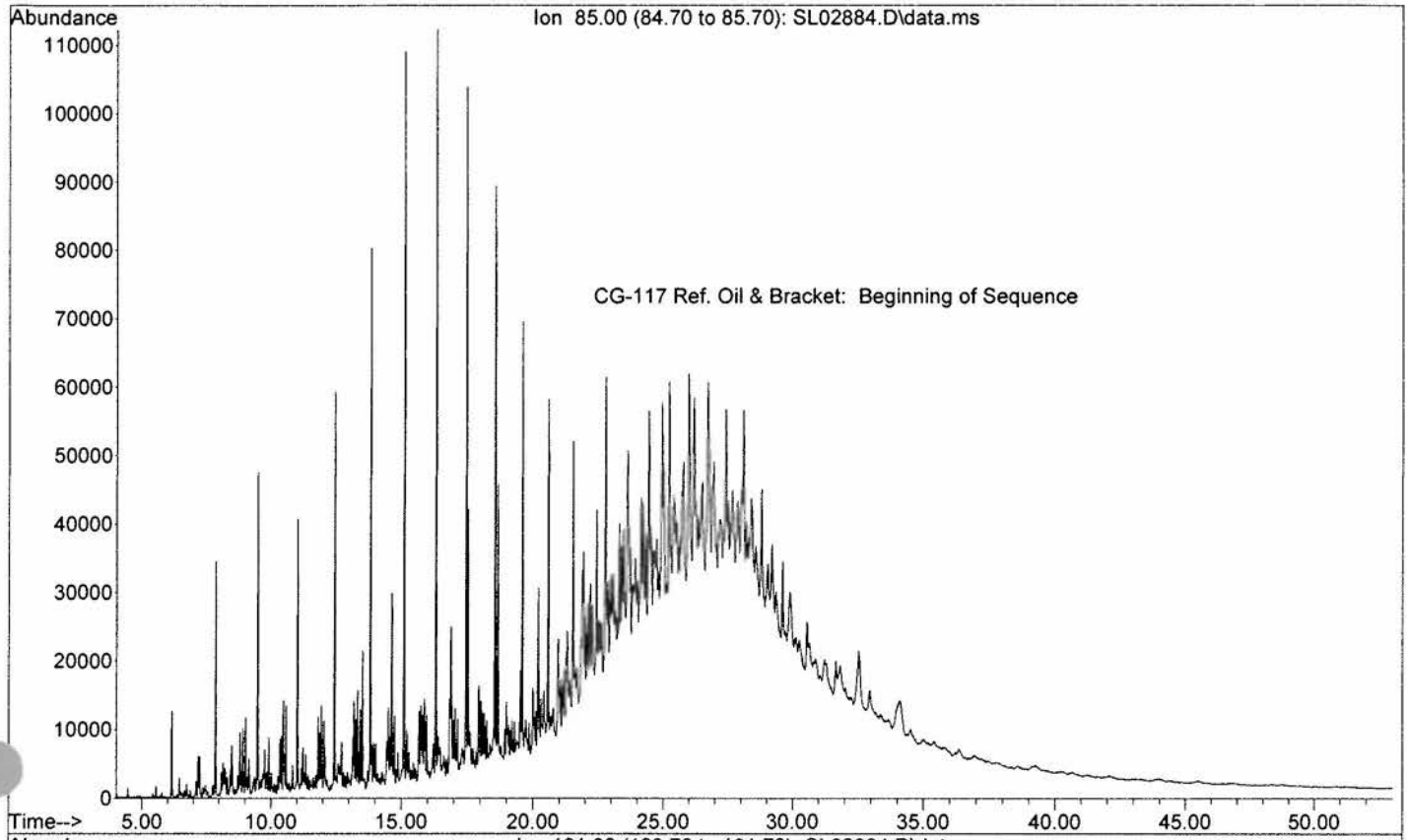


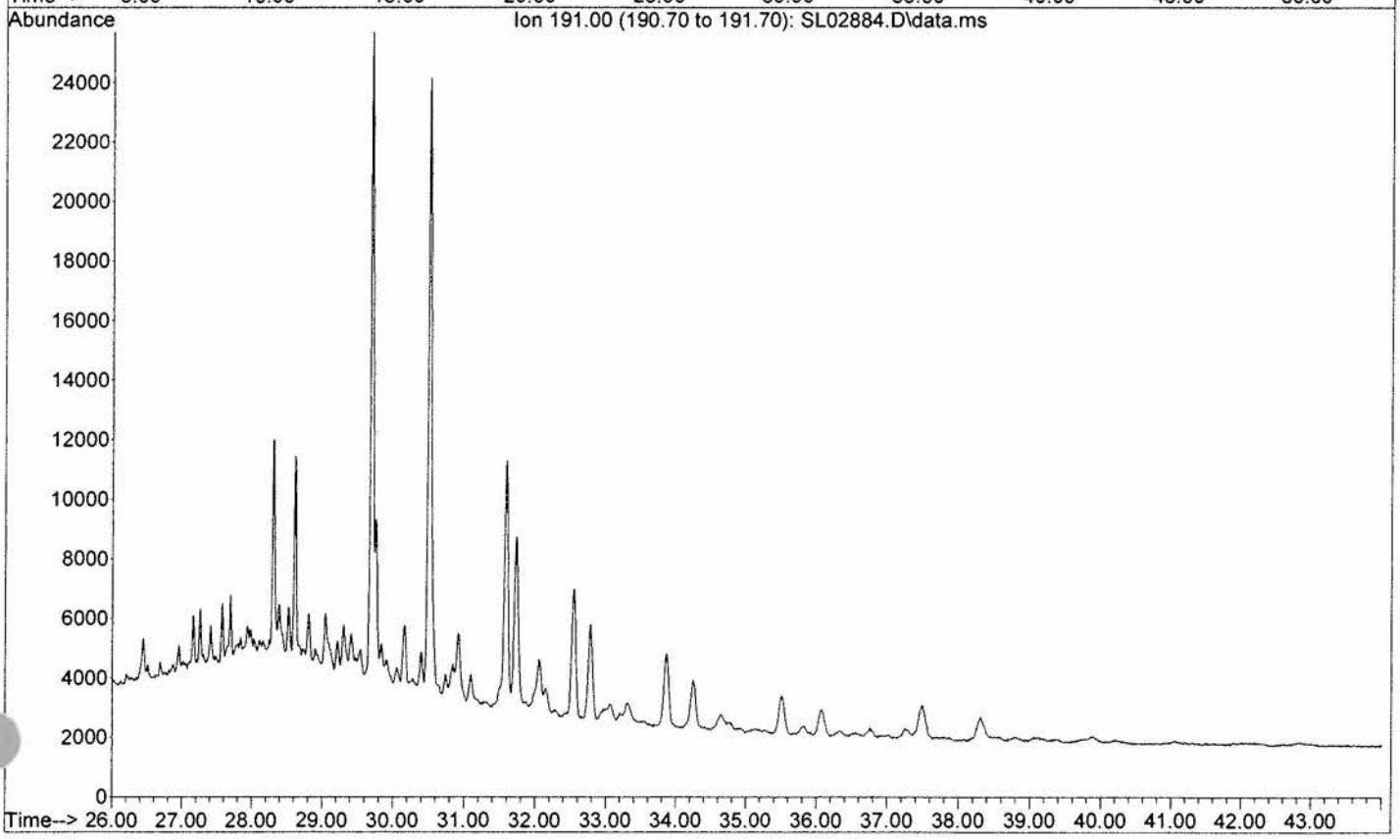
File :C:\msdchem\1\DATA\022712\SL02880.D
Operator : Syslo
Acquired : 28 Feb 2012 00:41 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003 EOS Duplicate
Vial Number: 7



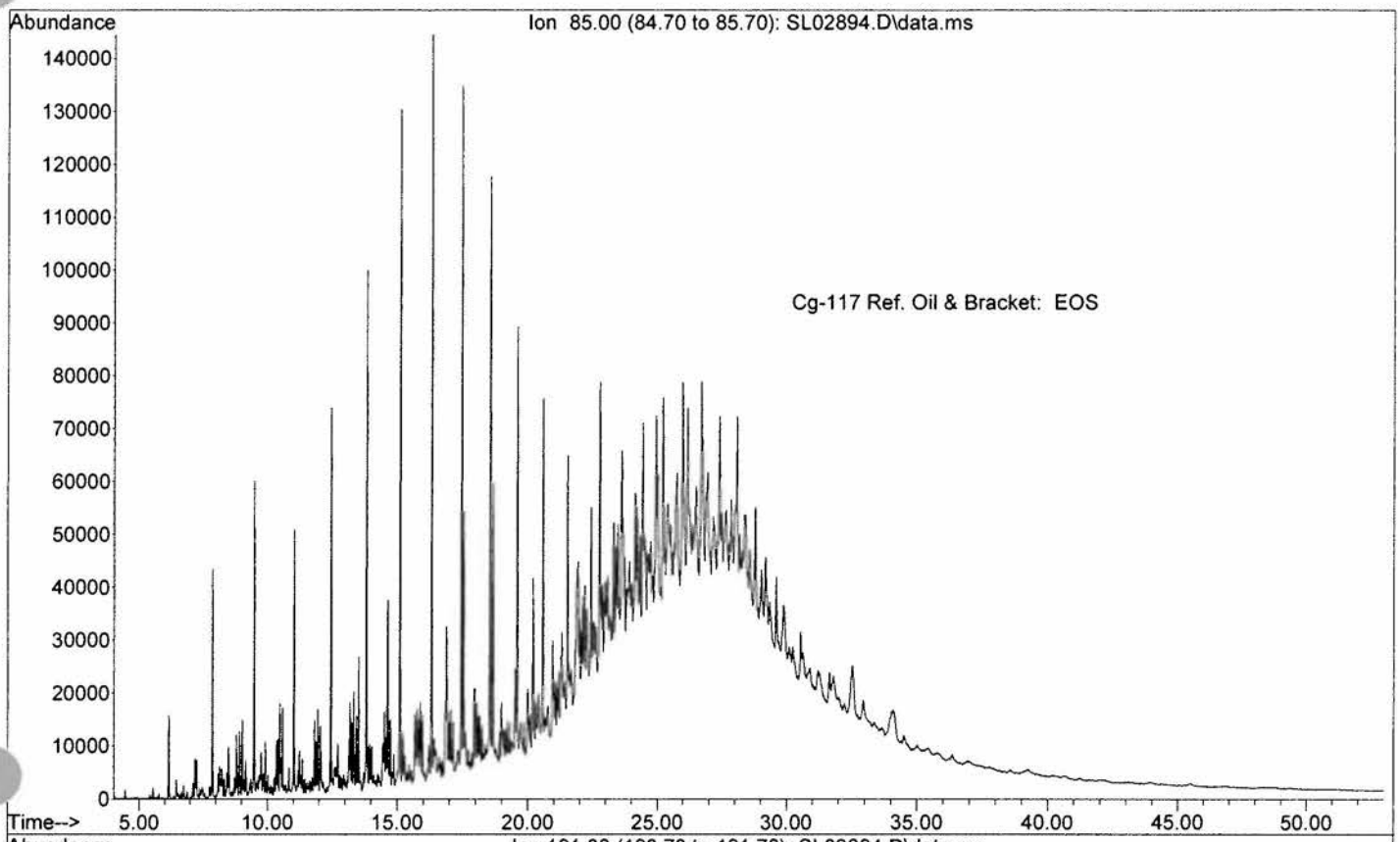


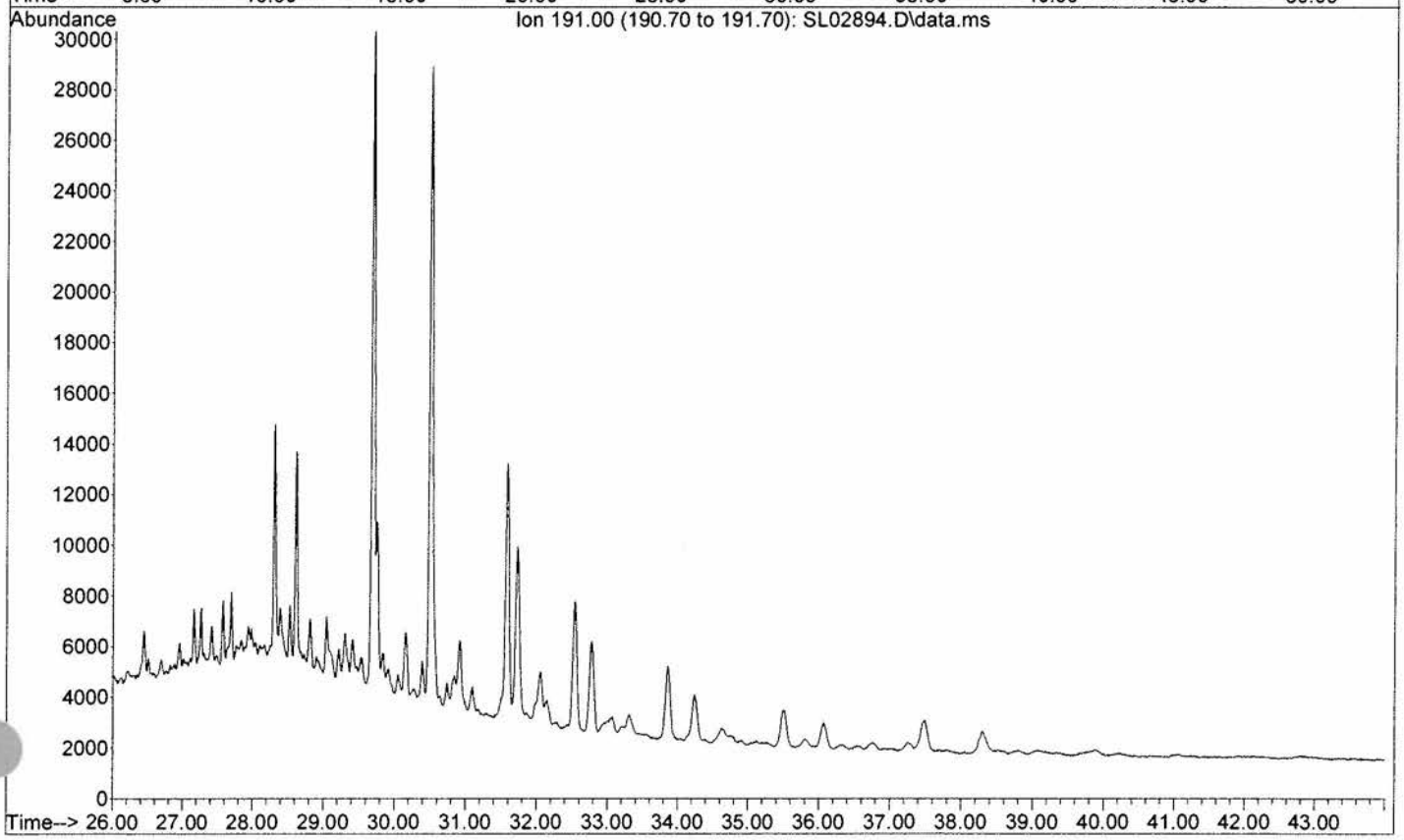
File :C:\msdchem\1\DATA\030112\SL02884.D
Operator : Syslo
Acquired : 1 Mar 2012 13:51 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003
Vial Number: 72





File :C:\msdchem\1\DATA\030112\SL02894.D
Operator : Syslo
Acquired : 2 Mar 2012 00:40 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003 EOS Reference
Vial Number: 10





#2 Diesel TPH Calibration Range - Using SERAS GC/MS Method 1841
TPH Calibration Range of 05/16/11
GC/MS System "Slick2" S/N#'s US10915004/US90432092

DRO-TPH as #2 Diesel Fuel	TPH/Oil Concentration 10000 ug/ml	5000 ug/ml	1000 ug/ml	500 ug/ml	100 ug/ml	50 ug/ml
GC/MS TPH Response (m/z 85+83+105+113+123+183)	86697279	40295375	9385180	4381273	981077	430368
d30-tetracosane response	156450	142438	157056	146200	145897	144045
d50-tetracosane response	176876	151428	182621	153167	134713	128364
d74-Hexatriacontane response	79529	60526	79109	63224	55469	53519
Sum of d30, d50 & d74-IS	412855	354392	418786	362591	336079	325928

IS Conc. (total ng) = 30

	TPH/Oil Concentration 10000 ug/ml	5000 ug/ml	1000 ug/ml	500 ug/ml	100 ug/ml	50 ug/ml	Average RF	% RSD
Response Factor from sum of ions 85+83+105+113+123+183	0.62998	0.68222	0.67231	0.72499	0.87576	0.79226	0.72959	12.38

Using DROTPH051611.M, a clone of OilsimS2.M: SERAS Oil Characterization Method ; with surrogates added to TPH range.

TPH response based on the sum of all peaks in the DRO (m/z 85 + 83 + 105 + 113 + 123 + 183) pattern from ~8.1 min to 28min.
%RSD Denotes Relative Standard Deviation.

Stock Oil Standard = RSJ0102: 50,000pm AccuStandard Stock Diesel Calibration Composite Mix
Calibration range preparation: 05/16/11 by J. Syslo; Oil Surrogates were spiked into the stock diesel standard.

THIS WAS USED ONLY FOR SURROGATE RECOVERIES FOR THE TWO Bucket Sediments

US EPA ARCHIVE DOCUMENT

Response Factor Report Slick2

Method Path : C:\MSDCHEM\1\METHODS\
Method File : DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11
Last Update : Tue May 17 18:20:58 2011
Response Via : Initial Calibration

ICAL - Surrogate

Calibration Files
10 =SL01871.D 100 =SL01872.D 50 =SL01874.D 5 =SL01873.D 1 =SL01875.D

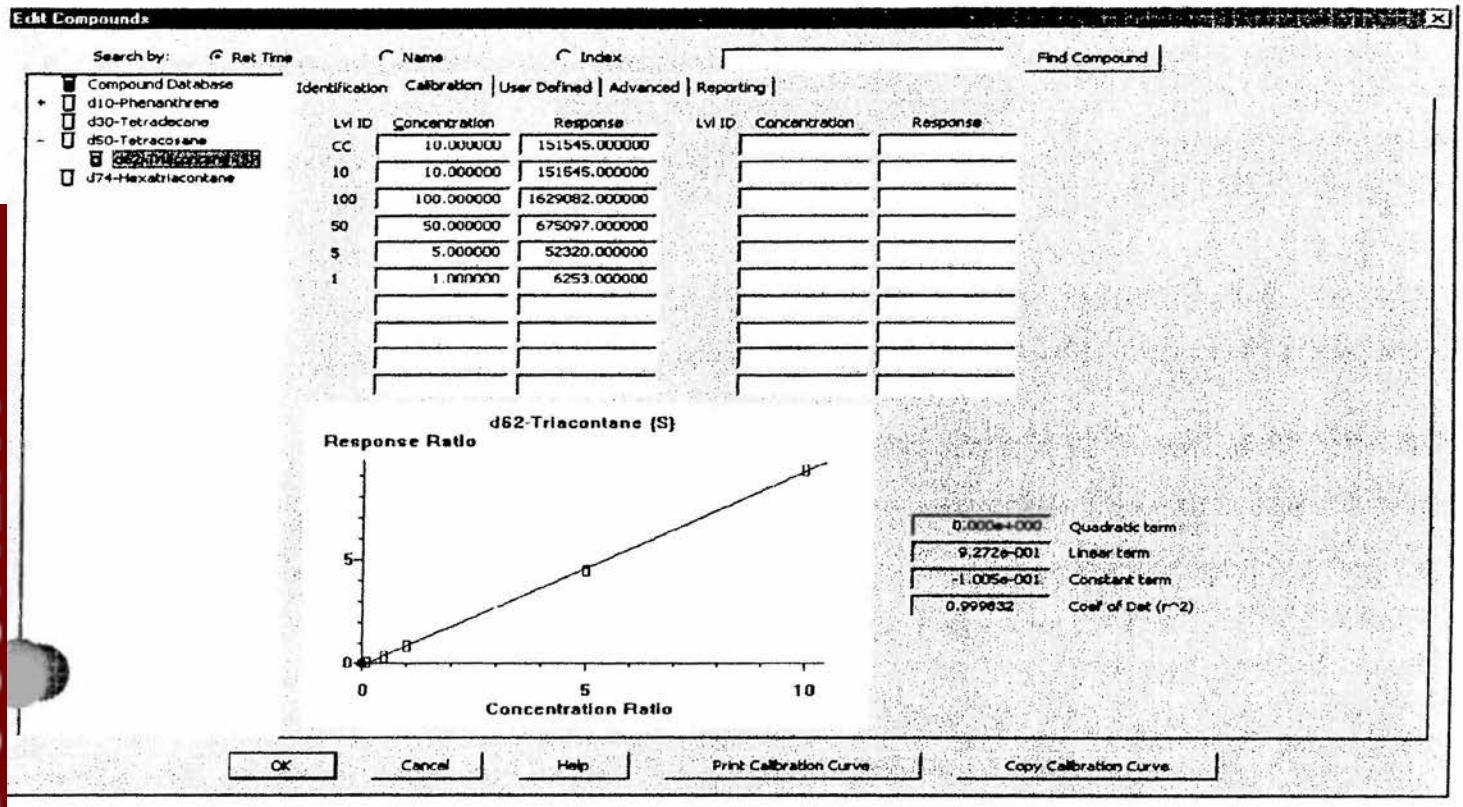
		Compound	10	100	50	5	1	Avg	%RSD
1)	I	d10-Phenanthrene	ISTD						
2)	S	d10-Anthracene {S}	1.078	0.995	1.043	1.018	1.088	1.044	3.78
3)	S	d14-o-Terphenyl {S}	0.498	0.453	0.464	0.482	0.472	0.474	3.65
4)	S	5a-Androstane {S}	0.062	0.059	0.058	0.059	0.055	0.059	4.16
5)	I	d30-Tetradecane	ISTD						
6)	I	d50-Tetracosane	ISTD						
7)	S	d62-Triacontane {S}	0.830	0.921	0.892	0.683	0.464	0.758	24.82
8)	I	d74-Hexatriacontane	ISTD						

LR
0.999832

(#) = Out of Range

DROTPH051611.M Tue May 17 18:21:06 2011

Linear Regression Compounds: DROTPH051611 on Slick II



US EPA ARCHIVE DOCUMENT

Compound List Report Slick2

Method Path : C:\MSDCHEM\1\METHODS\
Method File : DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11
Last Update : Tue May 17 18:20:58 2011
Response Via : Initial Calibration

Total Cpnds : 8

PK#		Compound Name	QIon	Exp_RT	Rel_RT	Cal	#Qual	A/H	ID
1	I	d10-Phenanthrene	188	18.921	1.000	A	0	A	R
2	S	d10-Anthracene {S}	188	19.038	1.006	A	0	A	B
3	S	d14-o-Terphenyl {S}	244	19.941	1.054	A	0	A	B
4	S	5a-Androstane {S}	260	21.357	1.129	A	0	A	B
5	I	d30-Tetradecane	66	13.724	1.000	A	0	A	B
6	I	d50-Tetracosane	66	23.974	1.000	A	0	A	B
7	S	d62-Triacontane {S}	66	28.360	1.183	L	0	A	B
8	I	d74-Hexatriacontane	66	35.131	1.000	A	0	A	B

Cal A = Average L = Linear LO = Linear w/origin Q = Quad QO = Quad w/origin
#Qual = number of qualifiers
A/H = Area or Height
ID R = R.T. B = R.T. & Q Q = Qvalue L = Largest A = All

DROTPH051611.M Tue May 17 18:22:02 2011

Calibration Status Report Slick2

Method Path : C:\MSDCHEM\1\METHODS\
Method File : DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11
Last Update : Tue May 17 18:20:58 2011
Response Via : Initial Calibration

#	ID	Conc	ISTD Conc	Path\File
1	CC	10	10	C:\msdchem\1\DATA\051611\SL01871.D
2	10	10	10	C:\msdchem\1\DATA\051611\SL01871.D
3	100	10	10	C:\msdchem\1\DATA\051611\SL01872.D
4	50	10	10	C:\msdchem\1\DATA\051611\SL01874.D
5	5	10	10	C:\msdchem\1\DATA\051611\SL01873.D
6	1	10	10	C:\msdchem\1\DATA\051611\SL01875.D

#	ID	Update Time	Quant Time	Acquisition Time
1	CC	May 16 18:51 2011	May 16 18:42 2011	16 May 2011 18:09
2	10	May 16 18:51 2011	May 16 18:42 2011	16 May 2011 18:09
3	100	May 17 17:56 2011	May 16 19:30 2011	16 May 2011 18:57
4	50	May 17 18:08 2011	May 16 20:56 2011	16 May 2011 20:23
5	5	May 17 18:08 2011	May 16 20:13 2011	16 May 2011 19:40
6	1	May 17 18:12 2011	May 17 18:11 2011	16 May 2011 21:06

DROTPH051611.M Tue May 17 18:22:10 2011

Sequence Name: C:\msdchem\1\sequence\Xchop.s
Comment: TPH Stds. check
Operator: Syslo
Data Path: C:\MSDCHEM\1\DATA\051611\
Instrument Control Pre-Seq Cmd:
Data Analysis Pre-Seq Cmd:

Instrument Control Post-Seq Cmd:
Data Analysis Post-Seq Cmd:

Method Sections To Run
(X) Full Method
( ) Reprocessing Only

On A Barcode Mismatch
(X) Inject Anyway
( ) Don't Inject

Line		Sample Name/Misc Info
1) Sample	99	SL01867 DFT8270D 50ppm DFTPP
2) Sample	1	Blank + IS
Datafile		SL01868
Method		DROTPH102110
3) Sample	71	1.0K TPH-DRO Std.
Datafile		SL01871
Method		DROTPH051611
4) Sample	72	10K TPH-DRO Std.
Datafile		SL01872
Method		DROTPH051611
5) Sample	73	500ppm TPH-DRO Std.
Datafile		SL01873
Method		DROTPH051611
6) Sample	74	5.0K TPH-DRO Std.
Datafile		SL01874
Method		DROTPH051611
7) Sample	75	100ppm TPH-DRO Std.
Datafile		SL01875
Method		DROTPH051611
8) Sample	76	50ppm TPH-DRO Std.
Datafile		SL01876
Method		DROTPH051611
9) Sample	78	1.0K TPH-DRO ICV
Datafile		SL01878
Method		DROTPH051611

US EPA ARCHIVE DOCUMENT

Injection Log

Data Directory: C:\msdchem\1\DATA\051611\

SampleName	MiscInfo	Vial	Multiplier	Injection Time
1) SL01867.D				
50ppm DFTPP	RUA0001	99	1.000	16 May 2011 14:28
2) SL01868.D				
Blank + IS	DCM/Hex + 20µL RUE00	1	1.000	16 May 2011 15:18
3) SL01869.D				
2000ppm TPH/DRO	10xd of old 20K RTJ0	2	1.000	16 May 2011 16:03
4) SL01870.D				
10ppm SHC/PAH	Mass/Res Check {RTL0	3	1.000	16 May 2011 17:09
5) SL01871.D				
1.0K TPH-DRO Std.	RUE0044	71	1.000	16 May 2011 18:09
6) SL01872.D				
10K TPH-DRO Std.	RUE0042	72	1.000	16 May 2011 18:57
7) SL01873.D				
500ppm TPH-DRO Std.	RUE0045	73	1.000	16 May 2011 19:40
8) SL01874.D				
5.0K TPH-DRO Std.	RUE0043	74	1.000	16 May 2011 20:23
9) SL01875.D				
100ppm TPH-DRO Std.	RUE0046	75	1.000	16 May 2011 21:06
SL01876.D				
ppm TPH-DRO Std.	RUE0047	76	1.000	16 May 2011 21:49
11) SL01877.D				
50ppm TPH-DRO Std.	RUE0048 {alt. prep}	77	1.000	16 May 2011 22:32
12) SL01878.D				
1.0K TPH-DRO ICV	RUE0049 from 2nd sou	78	1.000	16 May 2011 23:14
13) SL01879.D				
10K TPH-DRO old	RTJ0112 prep'd 10/21	79	1.000	16 May 2011 23:57
14) SL01880.D				
5.0K TPH-DRO old	RTJ0113 prep'd 10/21	80	1.000	17 May 2011 00:40
15) SL01881.D				
Surrogate Check 2xd	40ppm RUE0038 dil. 2	81	1.000	17 May 2011 1:23
16) SL01882.D				
400ppm MDL spike che	RUE0039 MDL spike ch	82	1.000	17 May 2011 2:06
17) SL01883.D				
LCS spike check 2xd	2000ppm RUE0040 dil.	83	1.000	17 May 2011 2:49
18) SL01884.D				
5K 75% "w" #2 Diesel	RTK0024 Vendor "weat	84	1.000	17 May 2011 3:32
SL01885.D				
10W40 Oil	RUE0050 Motor Oil	85	1.000	17 May 2011 4:14
SL01886.D				
4.0K 10W40 Oil	RTK0060 Motor Oil	86	1.000	17 May 2011 5:14
21) SL01887.D				

TPH ICAL : DROTPH05161.M

Logbook # SERAS-I-0121

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SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: TPH MDL, DOC in Soil + ICAL — Method File: DFTPP/ DROTPH05161.M

Work Assign.#: 0-011 — Inj. Volume: 1ul 1ul

Analysis/Date: 5/16/11 — Analyst: J. Sybl

ALS	FILE ID	SAMPLE ID		REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL01860	50ppm DFTPP		RUA0001	5/13/11	14:59	PASS
71	SL01861	1000ppm TPH		RUA00102		15:20	exp 9/16/11 17.6%
2	SL01862	10 ppm Oil Surr check		RUE0038		16:08	4ul of RUE0038
3	SL01863	400ppm MDL Check		RUE0039		16:53	6
4	SL01864	20ppm Oil Surr Check		RUE0038			2nd not Run
5	SL01865	1000ppm LCS Check		RUE0040		17:37	
6	SL01866	20ppm Oil Surr Check		RUE0038		18:27	
	BL	end of Std Check 5/13/11					2 Syl
99	SL01867	50ppm DFTPP		RUA0001	5/16/11	14:38	PASS 1446 - 1455
1	SL01868	Blank + IS		RUE0037		15:18	+ 20ul RUE0037
2	SL01869	2000 ppm DRO/TPH		RTJ0110		16:03	FROM RTJ0110
3	SL01870	10 ppm AAH/SHC	Surr	RTL0035		17:09	Oil(JIM-SE.M (FIA Re)
71	SL01871	1000 ppm TPH Cal	10ppm	RUE0044		18:09	TPH as DRO
72	SL01872	10,000 ppm TPH Cal	100	RUE0042		18:57	
73	SL01873	500 ppm TPH Cal	5ppm	RUE0045		19:40	
74	SL01874	5000 ppm TPH Cal	50	RUE0043		20:23	
75	SL01875	100 ppm TPH Cal	1.0	RUE0046		21:06	
76	SL01876	50 ppm TPH Cal	0.5	RUE0047		21:49	TPH as DRO
77	SL01877	5 ppm TPH Cal	0.5	RUE0048		22:32	all peak of 50
78	SL01878	1000 ppm DROTPH ICV	-	RUE0049		23:14	from 2nd Sma
79	SL01879	10K DRO-TPH	10c	RTJ0112	5/16/11	23:57	OLD Std
80	SL01880	5K DRO-TPH	50	RTJ0113	5/17/11	00:40	OLD Std
81	SL01881	Surrogate check 2nd		RUE0038		1:23	2x of 40 ppm (20ppm)
82	SL01882	Addtnl MDL spike check		RUE0039		2:06	
83	SL01883	LCS spike check 2x		RUE0040		2:49	Out of 12 hour clock
84	SL01884	5000ppm 75% Weathered Diesel		RTK0124		3:32	Just Diagnostic Run
85	SL01885	10K 10 Wax		RUE0050		4:14	No Lcr
86	SL01886	4K 10W40 Oil		RTK0040		5:14	No Lcr
1	SL01887	Blank + IS		RUE0037	5/17/11	6:14	No Lcr

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RUA0001	DFTPP	7/2/11	50 ppm		
2	RUE0037 RUE0044	Calibration Check	11/13/11	1000ppm		
3	RUE0037	Internal Standard	11/13/11	500 ppm		
4						
5						
6						

Reviewed By: [signature] — Date Checked: 05-17-11

Analyzed by: J Sybl 5/16/11

US EPA ARCHIVE DOCUMENT

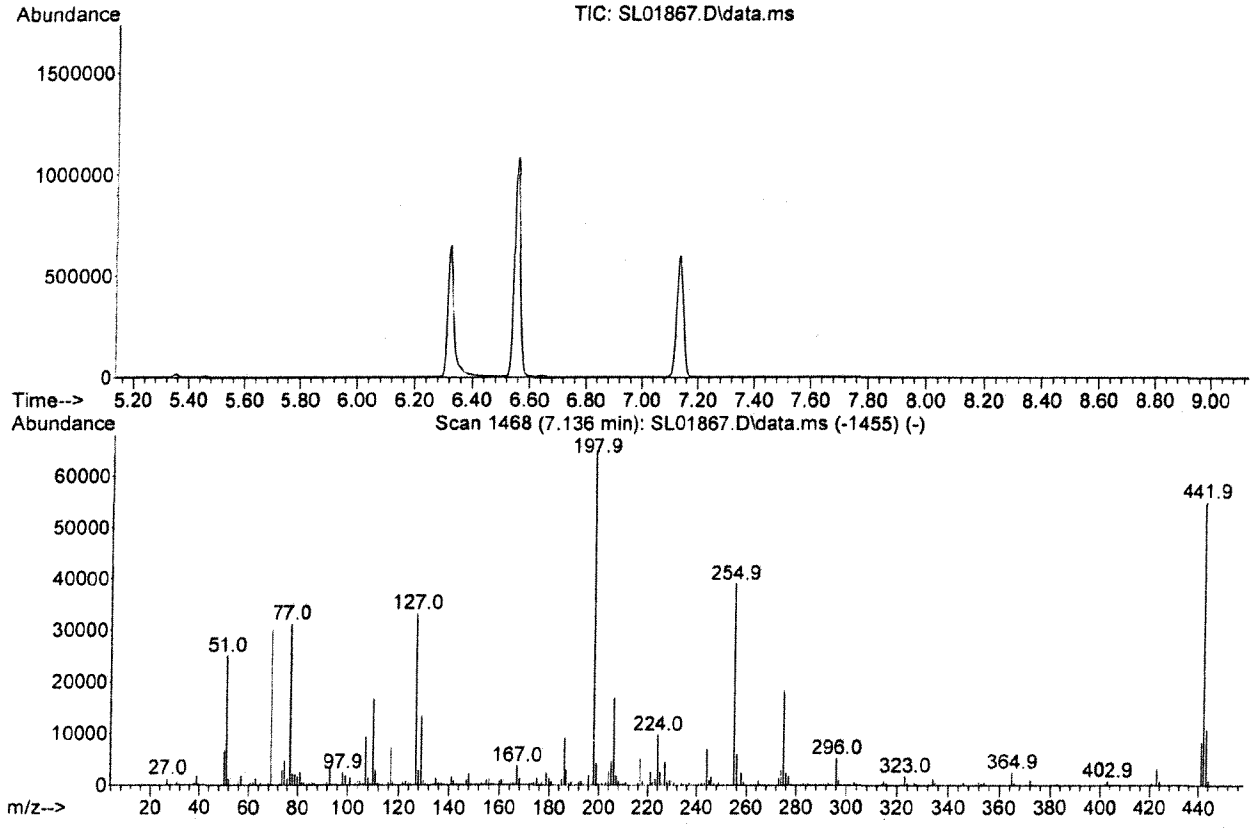
DFTPP - 8270D

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01867.D
Acq On : 16 May 2011 14:28
Operator : Syslo
Sample : 50ppm DFTPP
Misc : RUA0001
ALS Vial : 99 Sample Multiplier: 1

Tailing: Fcp = 1.51
Benzidine = 1.72
% DPT Deg = 1.57%

Integration File: rteint.p

Method : C:\msdchem\1\METHODS\DFT8270d.M
Title : DFTPP Method with 8270D criteria: 8/20/10
Last Update : Tue Aug 24 16:22:45 2010



Spectrum Information: Scan 1468

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	38.5	24981	PASS
68	69	0.00	2	1.4	409	PASS
69	198	0.00	100	46.3	30040	PASS
70	69	0.00	2	0.0	0	PASS
127	198	10	80	51.2	33191	PASS
197	198	0.00	2	0.0	0	PASS
198	198	100	100	100.0	64823	PASS
199	198	5	9	6.6	4251	PASS
275	198	10	60	27.9	18096	PASS
365	198	1	100	3.6	2346	PASS
441	442	0.01	24	15.2	8364	PASS
442	198	50	100	84.8	54954	PASS
443	442	15	24	19.4	10662	PASS

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01867.D
Acq On : 16 May 2011 14:28
Operator : Syslo
Sample : 50ppm DFTPP
Misc : RUA0001
ALS Vial : 99 Sample Multiplier: 1

Quant Time: May 16 14:40:30 2011
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Tue Aug 24 16:22:46 2010
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) Phenanthrene-d10	6.555	188	622865	40.00	ug/mL	0.00
4) Chrysene-d12	12.159	240	543842	40.00	ug/mL	-0.01
Target Compounds						Qvalue
2) Pentachlorophenol	6.323	266	112061	46.31	ug/mL	99
3) DFTPP	7.136	198	111412	40.28	ug/mL	95
5) Benzidine	10.017	184	454349	54.30	ug/mL	98
6) DDT	11.565	235	231180	47.35	ug/mL	90

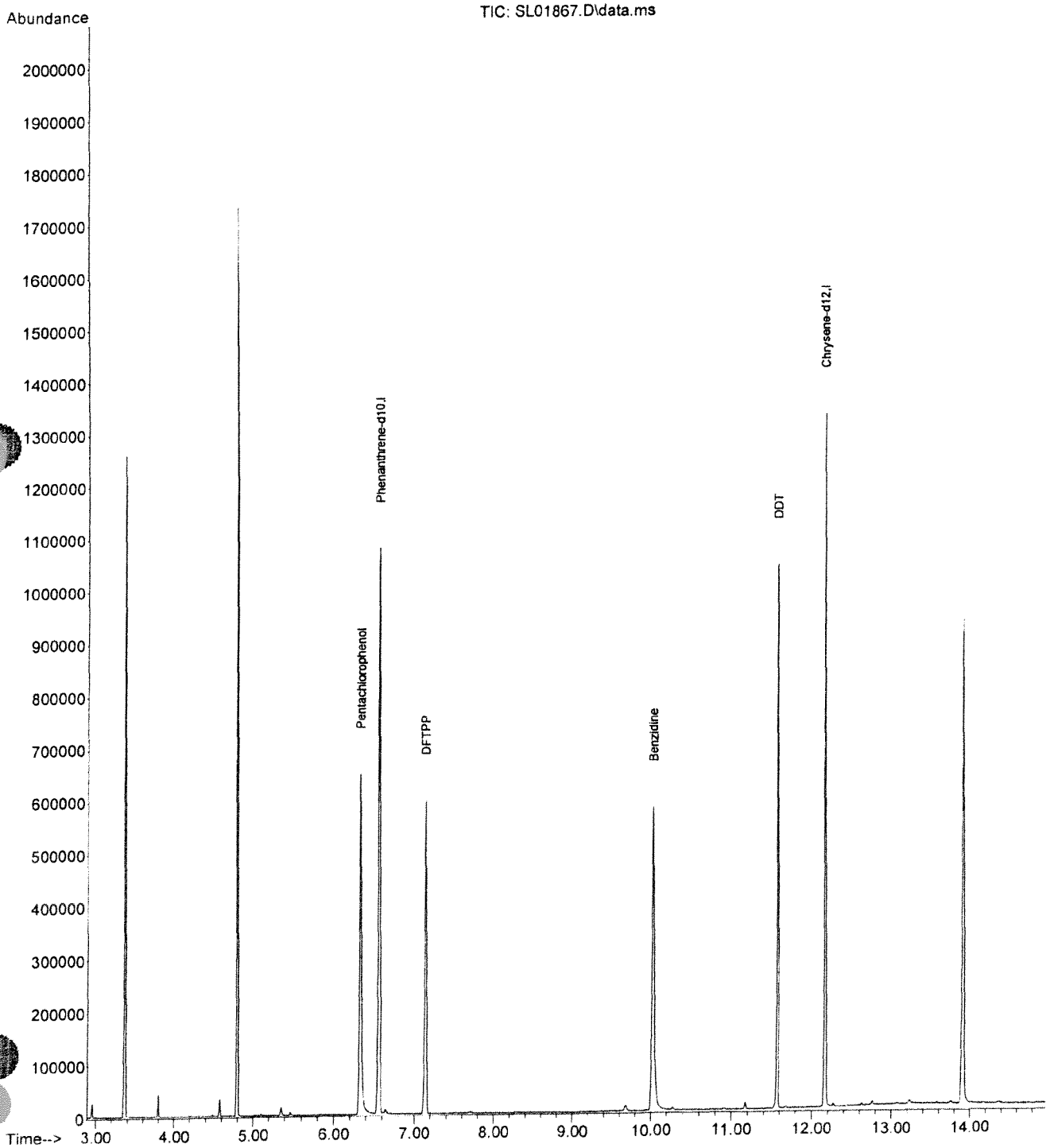
(#) = qualifier out of range (m) = manual integration (+) = signals summed

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01867.D
Acq On : 16 May 2011 14:28
Operator : Syslo
Sample : 50ppm DFTPP
Misc : RUA0001
ALS Vial : 99 Sample Multiplier: 1

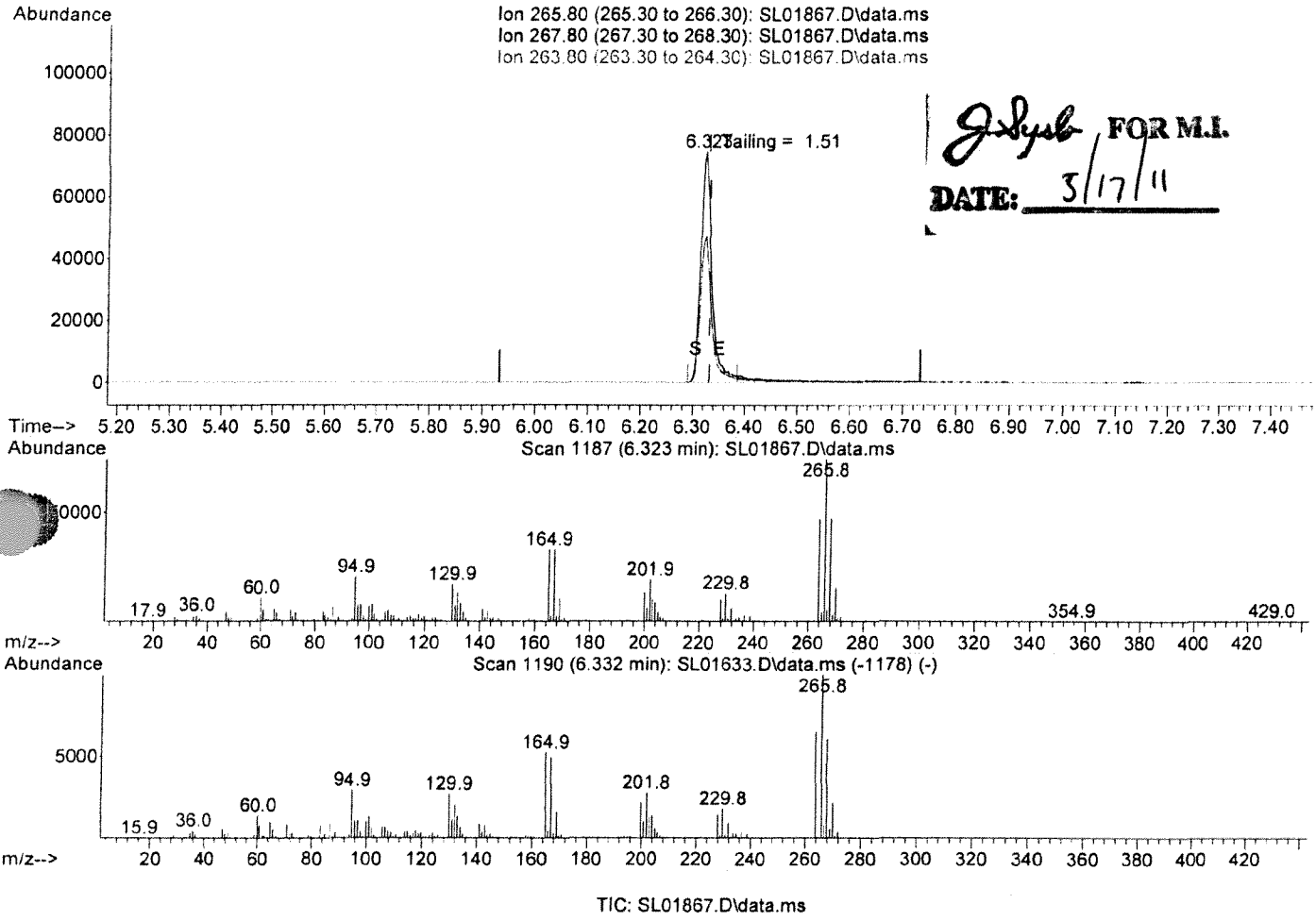
Quant Time: May 16 14:40:30 2011
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Tue Aug 24 16:22:46 2010
Response via : Initial Calibration



Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01867.D
Acq On : 16 May 2011 14:28
Operator : Syslo
Sample : 50ppm DFTPP
Misc : RUA0001
ALS Vial : 99 Sample Multiplier: 1

Quant Time: May 16 14:40:30 2011
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Tue Aug 24 16:22:46 2010
Response via : Initial Calibration



TIC: SL01867.D\data.ms

FOR M.I.
DATE: 5/17/11

(2) Pentachlorophenol

6.323min (-0.009) 46.31 ug/mL

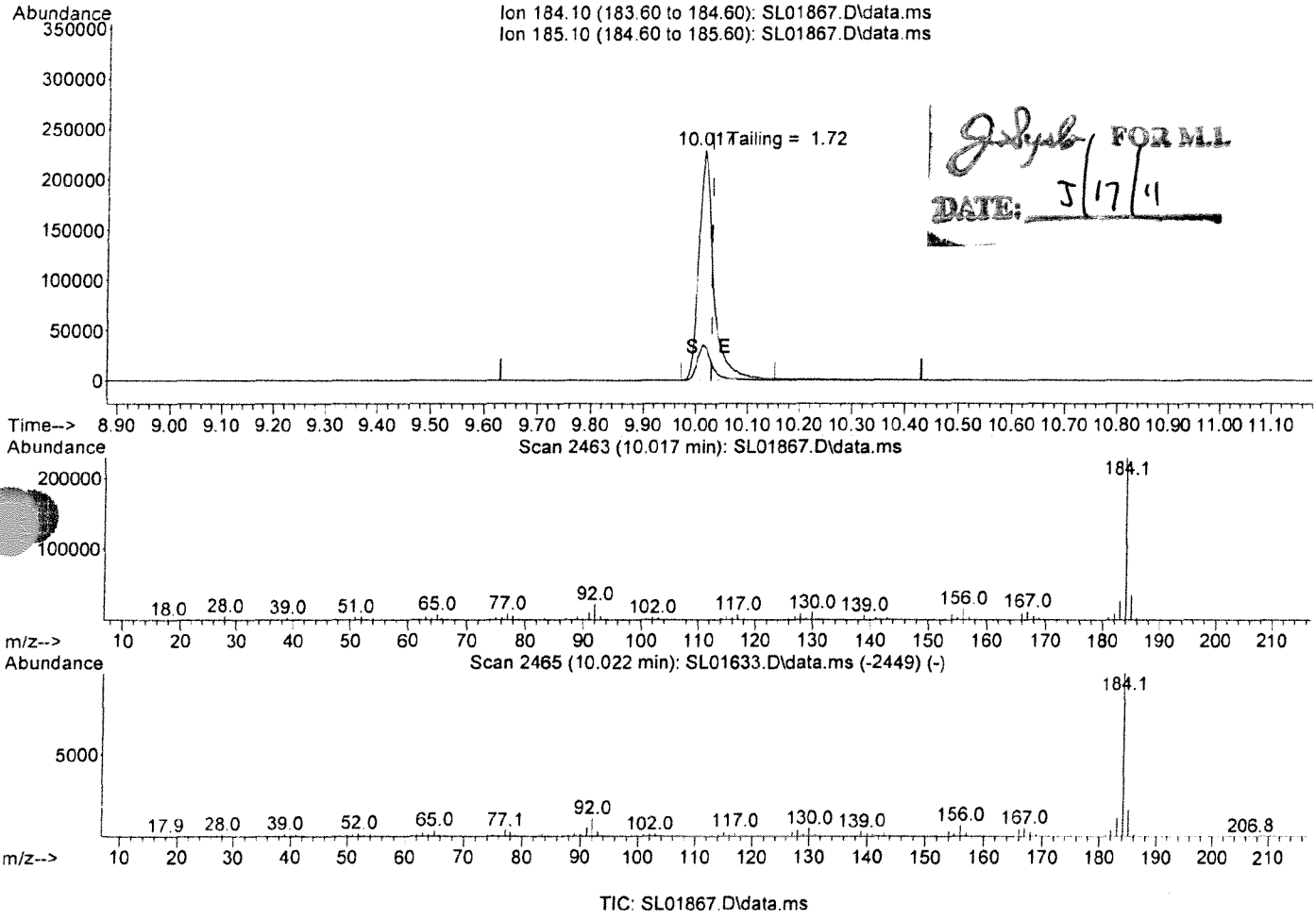
response 112061

Ion	Exp%	Act%
265.80	100	100
267.80	64.50	64.30
263.80	63.60	62.78
0.00	0.00	0.00

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01867.D
Acq On : 16 May 2011 14:28
Operator : Syslo
Sample : 50ppm DFTPP
Misc : RUA0001
ALS Vial : 99 Sample Multiplier: 1

Quant Time: May 16 14:40:30 2011
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Tue Aug 24 16:22:46 2010
Response via : Initial Calibration



TIC: SL01867.D\data.ms

FOR M.L
DATE: 5/17/11

(5) Benzidine

10.017min (-0.014) 54.30 ug/mL

response 454349

Ion	Exp%	Act%
184.10	100	100
185.10	14.60	15.43
0.00	0.00	0.00
0.00	0.00	0.00

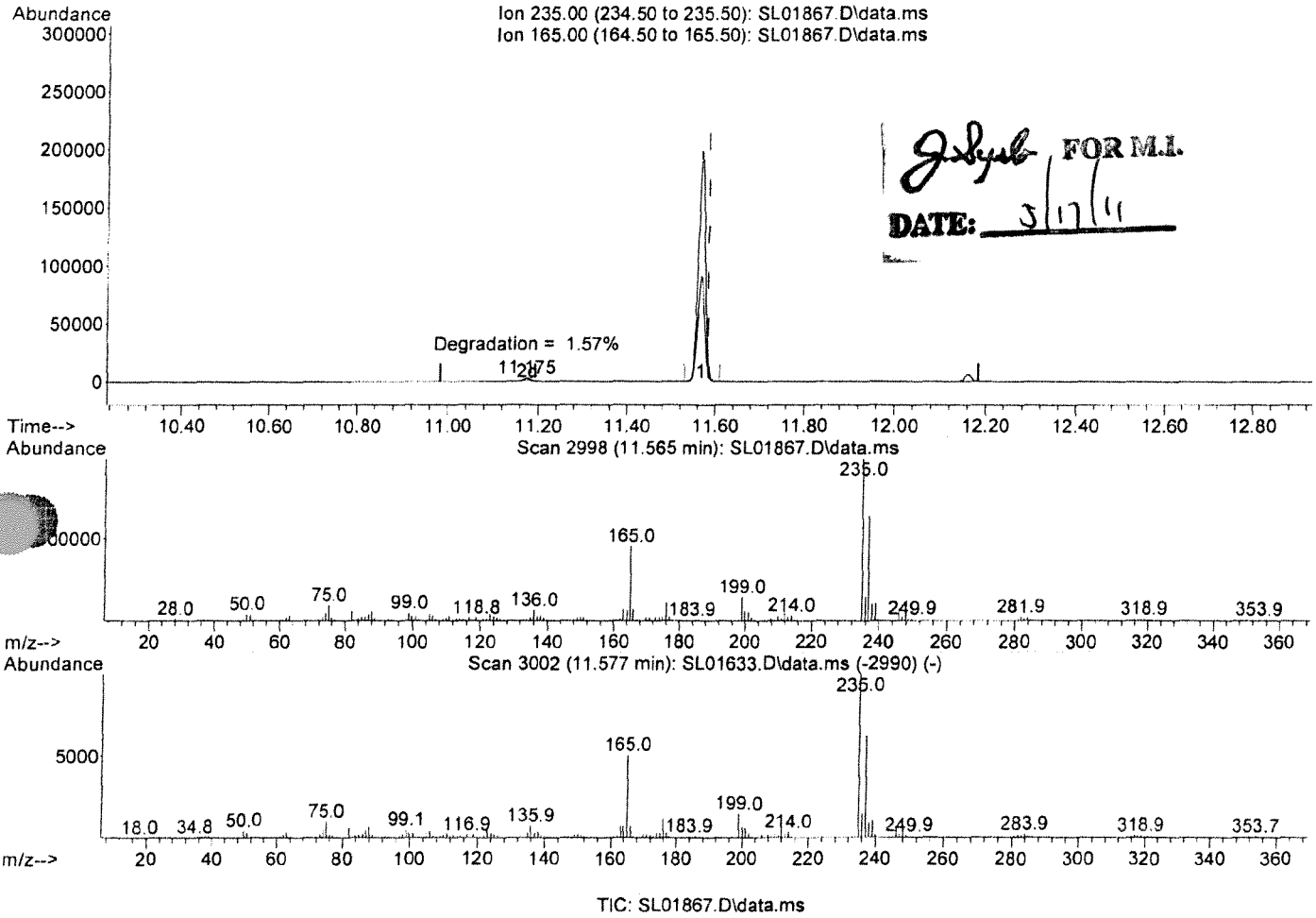
SERAS-017-DTM-011413_3

045

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01867.D
Acq On : 16 May 2011 14:28
Operator : Syslo
Sample : 50ppm DFTPP
Misc : RUA0001
ALS Vial : 99 Sample Multiplier: 1

Quant Time: May 16 14:40:30 2011
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Tue Aug 24 16:22:46 2010
Response via : Initial Calibration



FOR M.I.
DATE: 5/17/11

TIC: SL01867.D\data.ms

(6) DDT

11.565min (-0.017) 47.35 ug/mL

response 231180

Ion	Exp%	Act%
235.00	100	100
165.00	51.80	44.80
0.00	0.00	0.00
0.00	0.00	0.00

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01868.D
Acq On : 16 May 2011 15:18
Operator : Syslo
Sample : Blank + IS
Misc : DCM/Hex + 20µL RUE0037
ALS Vial : 1 Sample Multiplier: 1

Quant Time: May 16 15:58:35 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH102110.m
Quant Title : DRO/TPH ICAL: SIM 10/21/10
QLast Update : Mon Nov 22 17:17:41 2010
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	477227	10.00	ug/mL	0.00
5) d30-Tetradecane	13.732	66	148411	10.00	ug/mL	0.00
6) d50-Tetracosane	23.983	66	162479	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.139	66	86388	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	0.000	188	0	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
3) d14-o-Terphenyl {S}	19.968	244	5	0.00	ug/mL	0.02
Spiked Amount 20.000			Recovery =	0.00%		
4) 5a-Androstane {S}	21.384	260	2	0.00	ug/mL	0.02
Spiked Amount 20.000			Recovery =	0.00%		
7) d62-Triacontane {S}	0.000	66	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

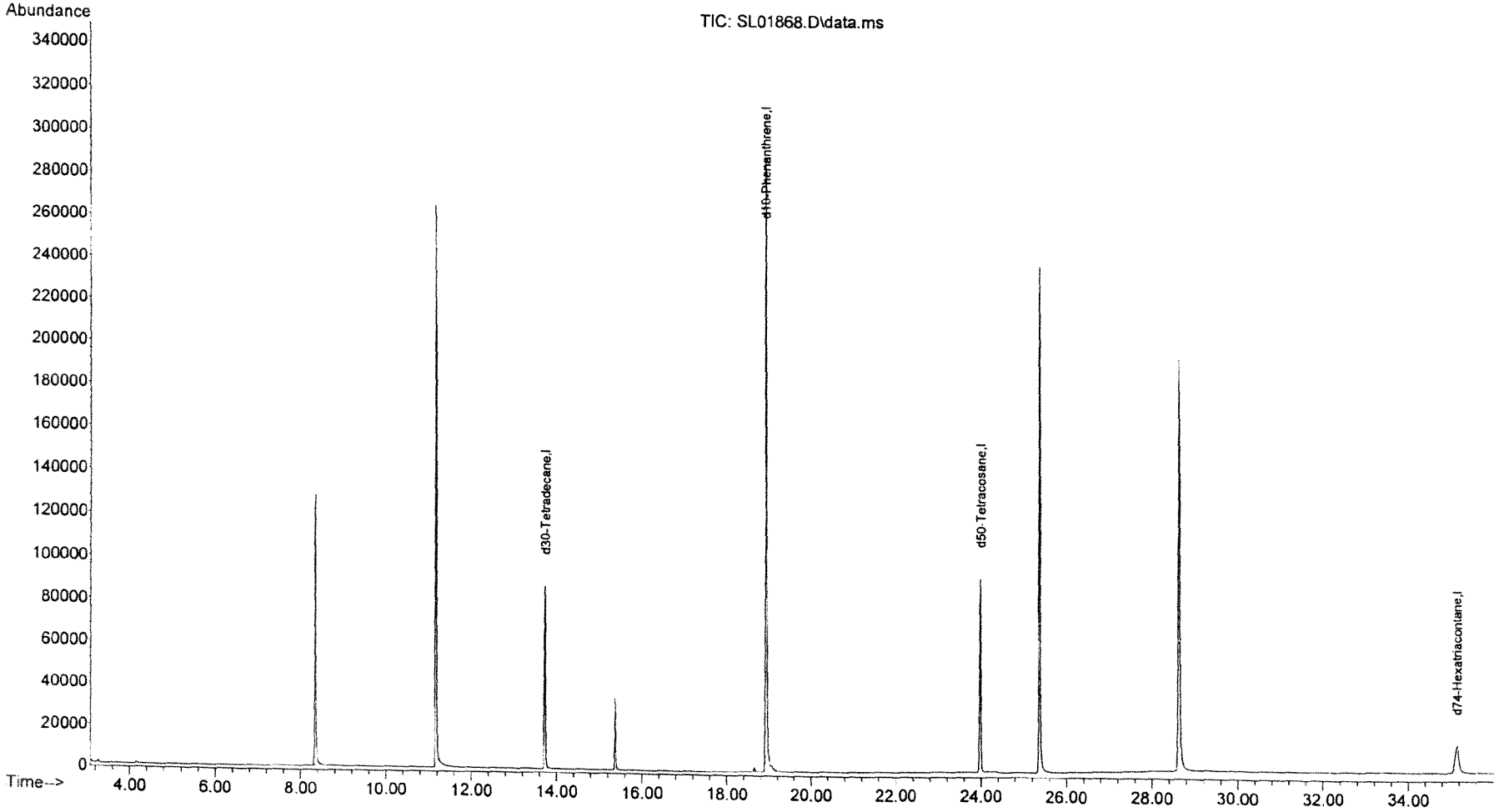
US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01868.D
Acq On : 16 May 2011 15:18
Operator : Syslo
Sample : Blank + IS
Misc : DCM/Hex + 20µL RUE0037
ALS Vial : 1 Sample Multiplier: 1

Quant Time: May 16 15:58:35 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH102110.m
Quant Title : DRO/TPH ICAL: SIM 10/21/10
QLast Update : Mon Nov 22 17:17:41 2010
Response via : Initial Calibration



DROTPH102110.m Tue May 17 17:47:49 2011

048

5.16.11

TPH as DRO + Surr.

ICAL DATA

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01871.D
Acq On : 16 May 2011 18:09
Operator : Syslo
Sample : 1.0K TPH-DRO Std.
Misc : RUE0044
ALS Vial : 71 Sample Multiplier: 1

Quant Time: May 16 18:42:58 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Nov 22 17:17:41 2010
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	481334	10.00	ug/mL	0.00
5) d30-Tetradecane	13.724	66	157056	10.00	ug/mL	0.00
6) d50-Tetracosane	23.974	66	182621	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.131	66	79109	10.00	ug/mL	-0.02
System Monitoring Compounds						
2) d10-Anthracene {S}	19.038	188	518982	12.04	ug/mL	0.00
Spiked Amount 20.000			Recovery =	60.20%		
3) d14-o-Terphenyl {S}	19.941	244	239939	12.48	ug/mL	0.00
Spiked Amount 20.000			Recovery =	62.40%		
4) 5a-Androstane {S}	21.357	260	29860	12.41	ug/mL	0.00
Spiked Amount 20.000			Recovery =	62.05%		
7) d62-Triacontane {S}	28.360	66	151545	10.89	ug/mL	0.00
Spiked Amount 20.000			Recovery =	54.45%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

Σ_{TPH} = 9385180

≈ 1070 ppm on 10/22/11
cwm.

050

SERAS-017-DTM-011413_3

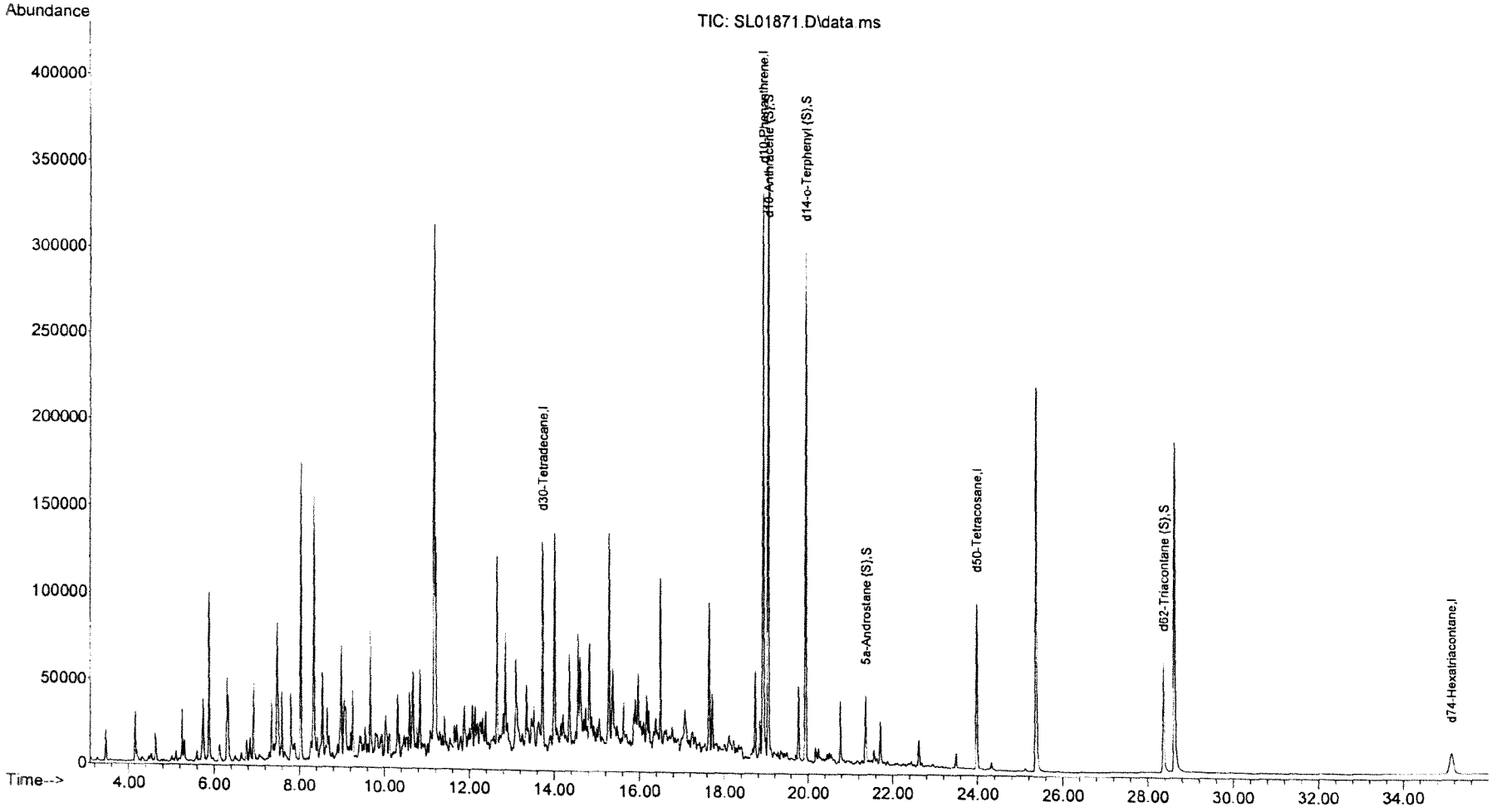
US EPA ARCHIVE DOCUMENT

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SERAS-017-DTM-011413_3

Quantitation Report (QT Rev ed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01871.D
Acq On : 16 May 2011 18:09
Operator : Syslo
Sample : 1.0K TPH-DRO Std.
Misc : RUE0044
ALS Vial : 71 Sample Multiplier: 1

Quant Time: May 16 18:42:58 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Nov 22 17:17:41 2010
Response via : Initial Calibration



DROTPH051611.M Mon May 16 18:45:19 2011

051

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01871.D
Acq On : 16 May 2011 18:09
Operator : Syslo
Sample : 1.0K TPH-DRO Std.
Misc : RUE0044
ALS Vial : 71 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01871.D\data.ms

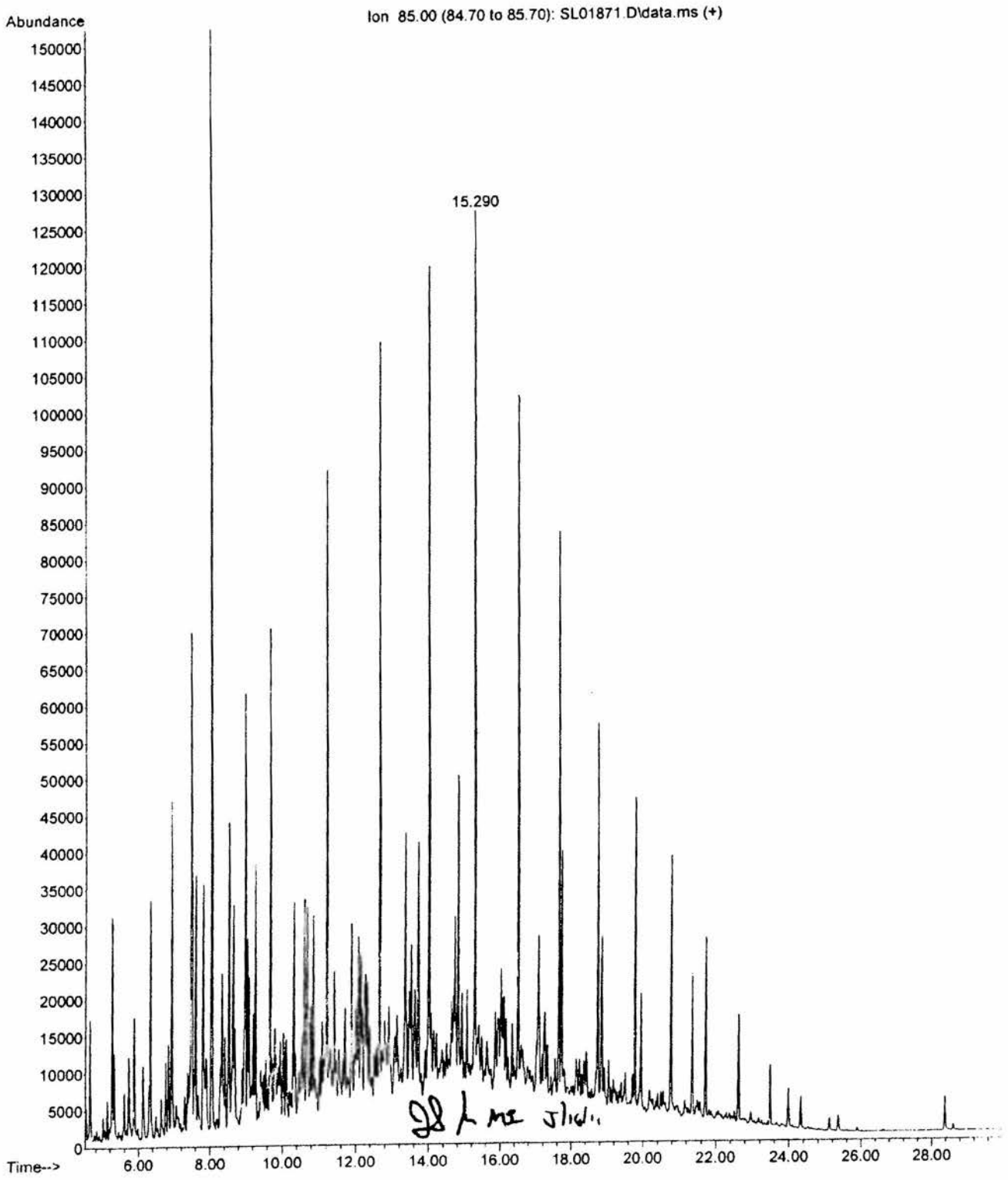
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	15.290	629	1528	2777	rM 9	126231	9385180	100.00%	100.000%

Sum of corrected areas: 9385180

MF 5/16/11

TPH051611.M Mon May 16 18:46:25 2011

File :C:\msdchem\1\DATA\051611\SL01871.D
Operator : Syslo
Acquired : 16 May 2011 18:09 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 1.0K TPH-DRO Std.
Misc Info : RUE0044
Vial Number: 71



US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01872.D
Acq On : 16 May 2011 18:57
Operator : Syslo
Sample : 10K TPH-DRO Std.
Misc : RUE0042
ALS Vial : 72 Sample Multiplier: 1

Quant Time: May 16 19:30:15 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon May 16 18:51:22 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	460599	10.00	ug/mL	0.00
5) d30-Tetradecane	13.732	66	156450	10.00	ug/mL	0.00
6) d50-Tetracosane	23.983	66	176876	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.130	66	79529	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	19.047	188	4582777	106.97	ug/mL	0.00
Spiked Amount 20.000			Recovery = 534.85%			
3) d14-o-Terphenyl {S}	19.949	244	2087742	107.92	ug/mL	0.00
Spiked Amount 20.000			Recovery = 539.60%			
4) 5a-Androstane {S}	21.366	260	272472	112.94	ug/mL	0.00
Spiked Amount 20.000			Recovery = 564.70%			
7) d62-Triacontane {S}	28.369	66	1629082	108.65	ug/mL	0.00
Spiked Amount 20.000			Recovery = 543.25%			
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

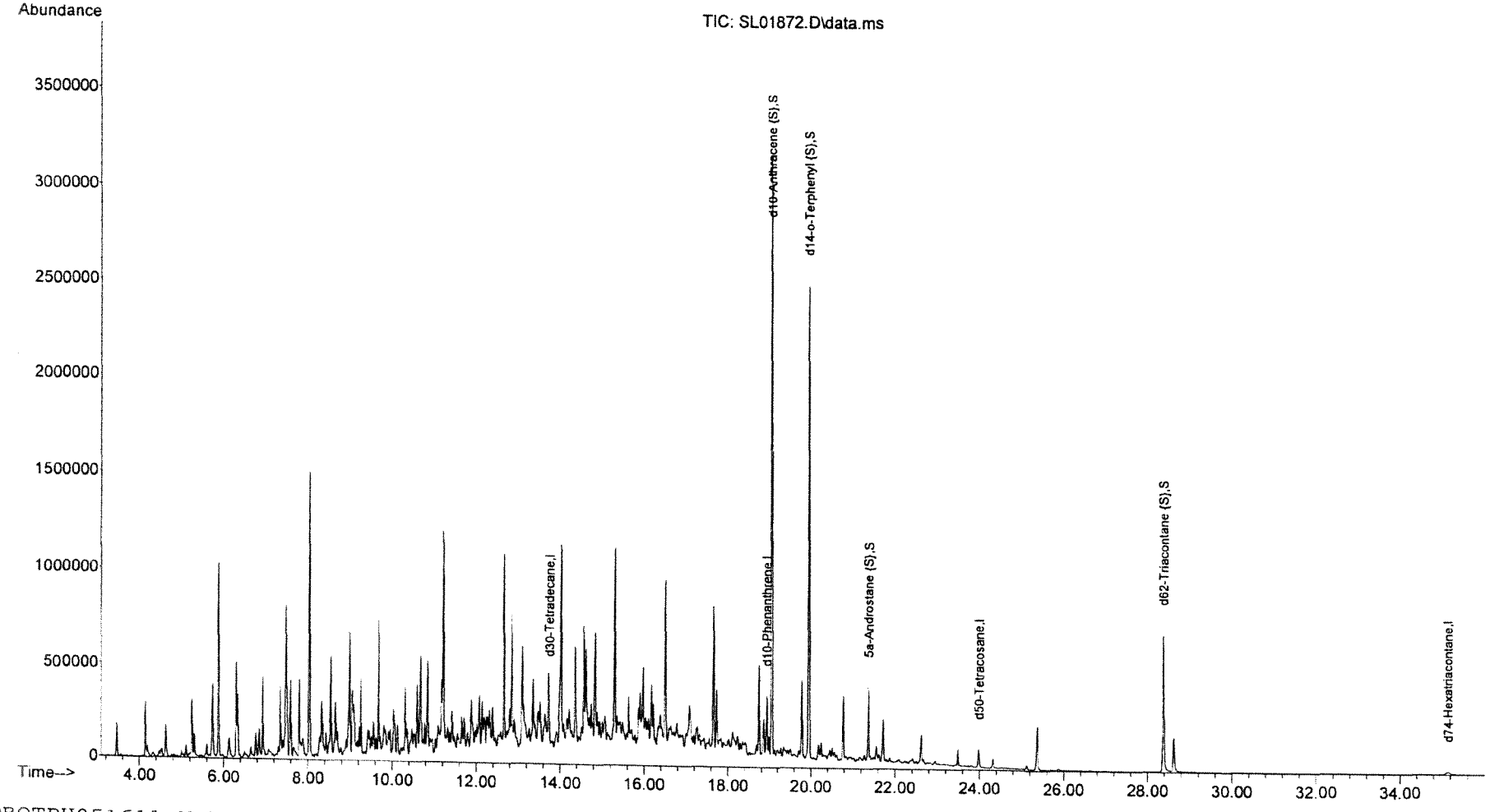
Σ_{TPH} = 86697279

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SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01872.D
Acq On : 16 May 2011 18:57
Operator : Syslo
Sample : 10K TPH-DRO Std.
Misc : RUE0042
ALS Vial : 72 Sample Multiplier: 1

Quant Time: May 16 19:30:15 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon May 16 18:51:22 2011
Response via : Initial Calibration



DROTPH051611.M Tue May 17 17:57:15 2011

055

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01872.D
Acq On : 16 May 2011 18:57
Operator : Syslo
Sample : 10K TPH-DRO Std.
Misc : RUE0042
ALS Vial : 72 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01872.D\data.ms

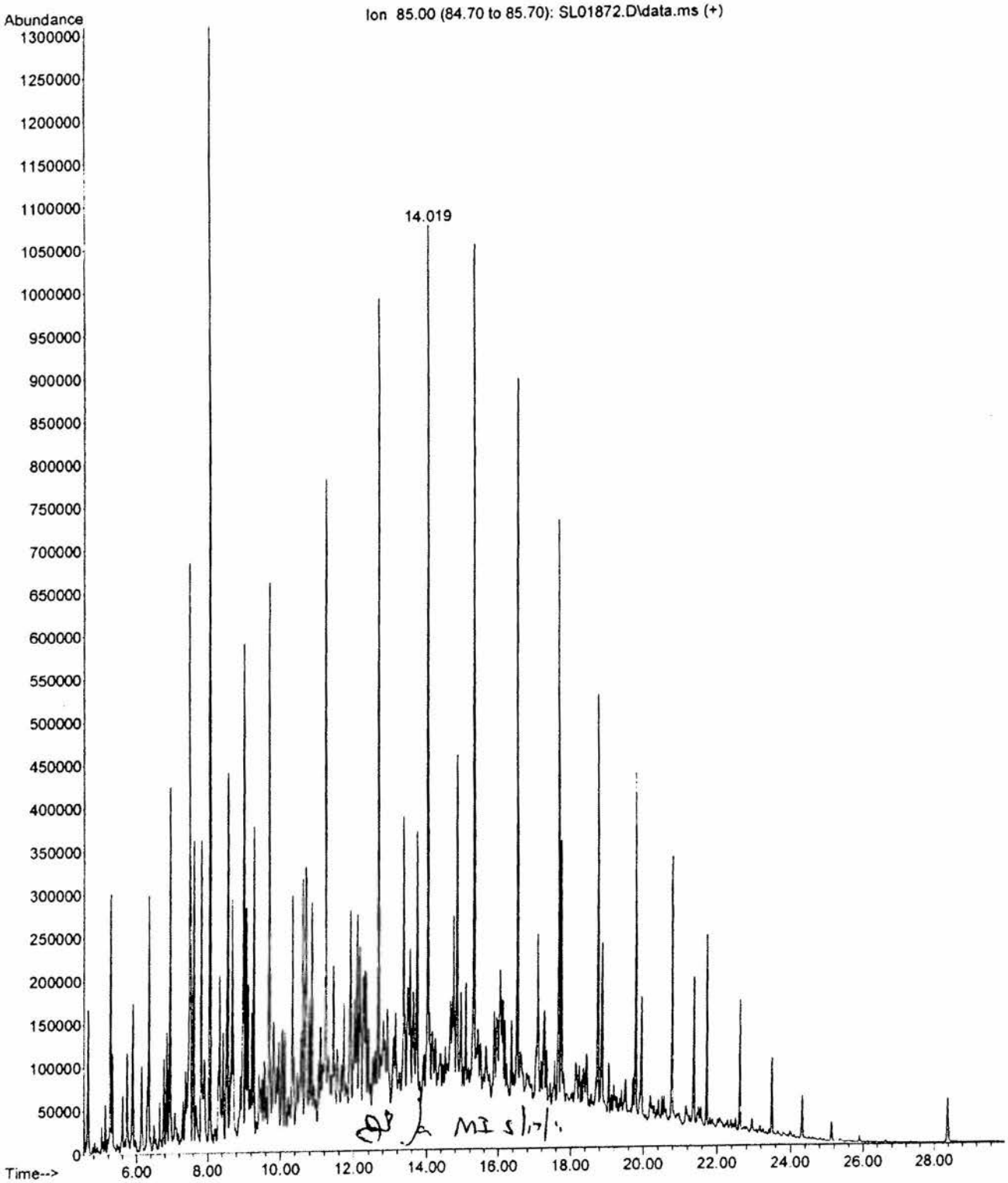
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	14.019	636	1369	2976	rM	7 1073264	86697279	100.00%	100.000%

Sum of corrected areas: 86697279

TPH051611.M Tue May 17 17:58:46 2011

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\051611\SL01872.D
Operator : Syslo
Acquired : 16 May 2011 18:57 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 10K TPH-DRO Std.
Misc Info : RUE0042
Vial Number: 72



US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01873.D
Acq On : 16 May 2011 19:40
Operator : Syslo
Sample : 500ppm TPH-DRO Std.
Misc : RUE0045
ALS Vial : 73 Sample Multiplier: 1

Quant Time: May 16 20:13:26 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon May 16 18:51:22 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	434315	10.00	ug/mL	0.00
5) d30-Tetradecane	13.724	66	146200	10.00	ug/mL	0.00
6) d50-Tetracosane	23.974	66	153167	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.130	66	63224	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	19.038	188	220965	5.47	ug/mL	0.00
Spiked Amount 20.000			Recovery =	27.35%		
3) d14-o-Terphenyl {S}	19.940	244	104564	5.73	ug/mL	0.00
Spiked Amount 20.000			Recovery =	28.65%		
4) 5a-Androstane {S}	21.357	260	12842	5.65	ug/mL	0.00
Spiked Amount 20.000			Recovery =	28.25%		
7) d62-Triacontane {S}	28.360	66	52320	4.71	ug/mL	0.00
Spiked Amount 20.000			Recovery =	23.55%		

Target Compounds Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

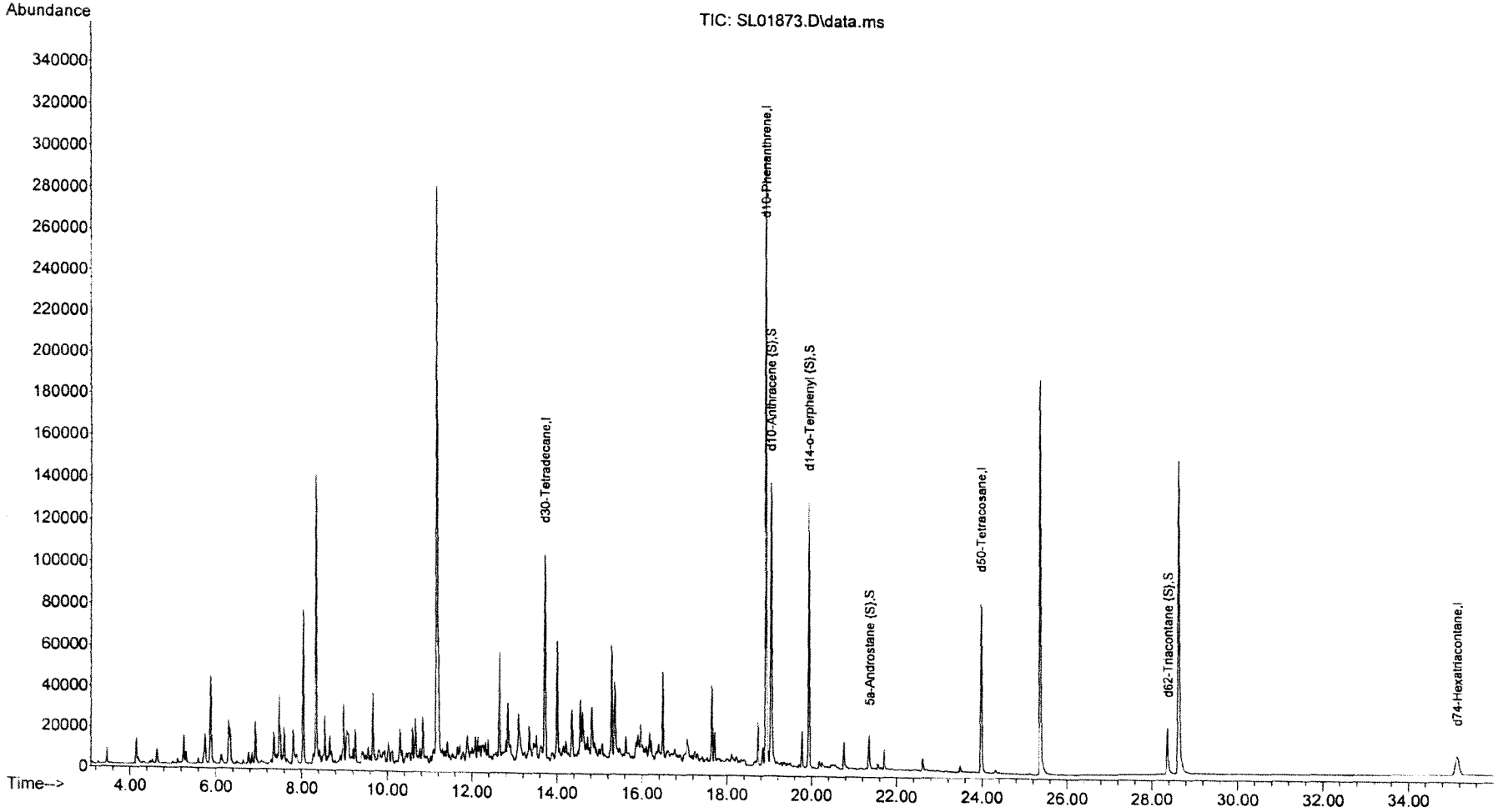
Sph: 4381273

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
059

Quantitation Report (QT Rev ed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01873.D
Acq On : 16 May 2011 19:40
Operator : Syslo
Sample : 500ppm TPH-DRO Std.
Misc : RUE0045
ALS Vial : 73 Sample Multiplier: 1

Quant Time: May 16 20:13:26 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon May 16 18:51:22 2011
Response via : Initial Calibration



DROTPH051611.M Tue May 17 18:02:32 2011

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01873.D
Acq On : 16 May 2011 19:40
Operator : Syslo
Sample : 500ppm TPH-DRO Std.
Misc : RUE0045
ALS Vial : 73 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01873.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	15.290	645	1528	2717	rM 6	57665	4381273	100.00%	100.000%

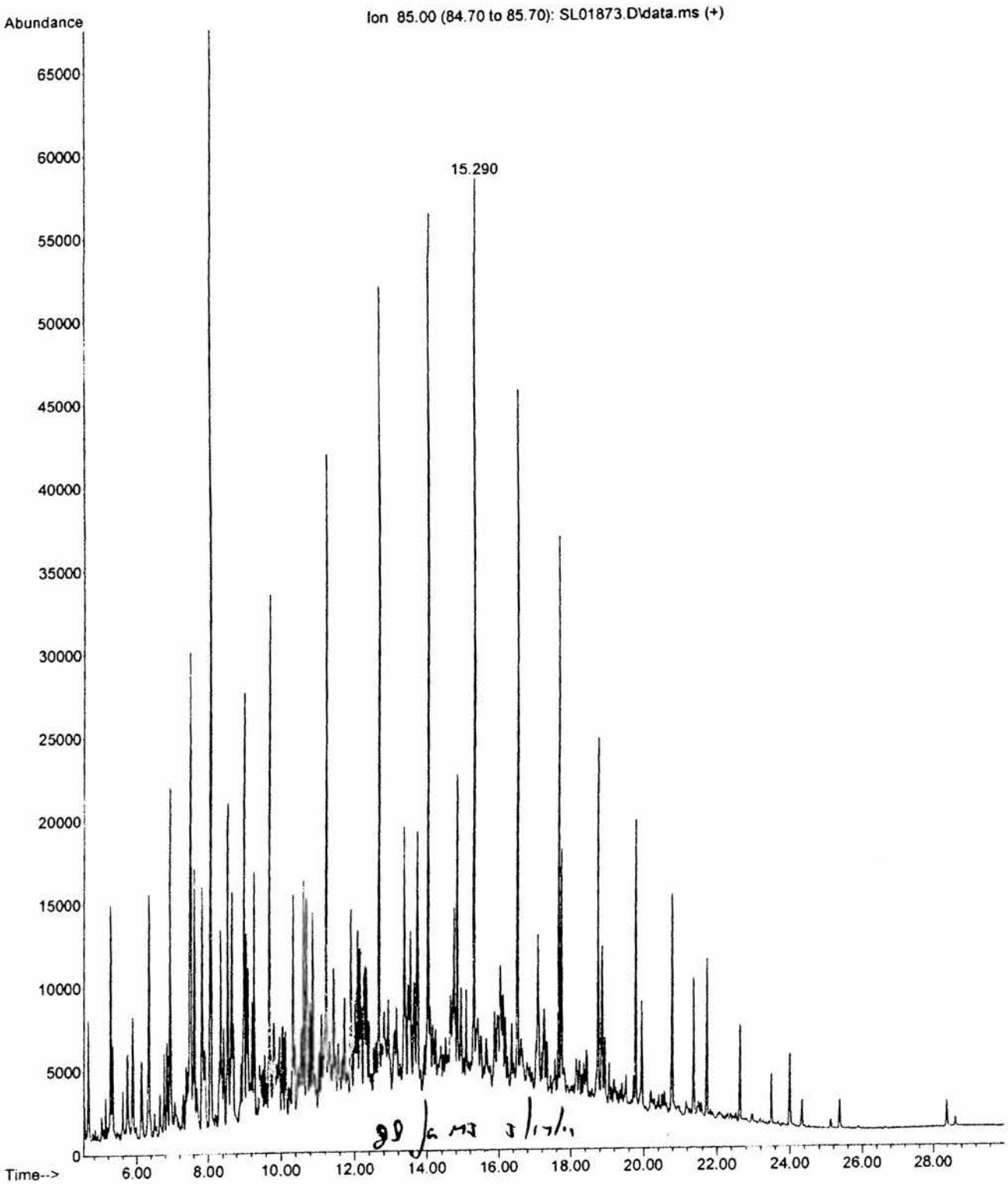
Sum of corrected areas: 4381273

PH051611.M Tue May 17 18:03:26 2011

5/17/11

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\051611\SL01873.D
Operator : Syslo
Acquired : 16 May 2011 19:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 500ppm TPH-DRO Std.
Misc Info : RUE0045
Vial Number: 73



US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01874.D
Acq On : 16 May 2011 20:23
Operator : Syslo
Sample : 5.0K TPH-DRO Std.
Misc : RUE0043
ALS Vial : 74 Sample Multiplier: 1

Quant Time: May 16 20:56:26 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon May 16 20:16:54 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	411935	10.00	ug/mL	0.00
5) d30-Tetradecane	13.724	66	142438	10.00	ug/mL	0.00
6) d50-Tetracosane	23.974	66	151428	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.130	66	60526	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	19.038	188	2148482	53.87	ug/mL	0.00
Spiked Amount 20.000			Recovery =	269.35%		
3) d14-o-Terphenyl {S}	19.940	244	955201	51.94	ug/mL	0.00
Spiked Amount 20.000			Recovery =	259.70%		
4) 5a-Androstane {S}	21.357	260	120381	52.62	ug/mL	0.00
Spiked Amount 20.000			Recovery =	263.10%		
7) d62-Triacontane {S}	28.360	66	675097	60.54	ug/mL	0.00
Spiked Amount 20.000			Recovery =	302.70%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

$\Sigma_{TPH} = 40295375$

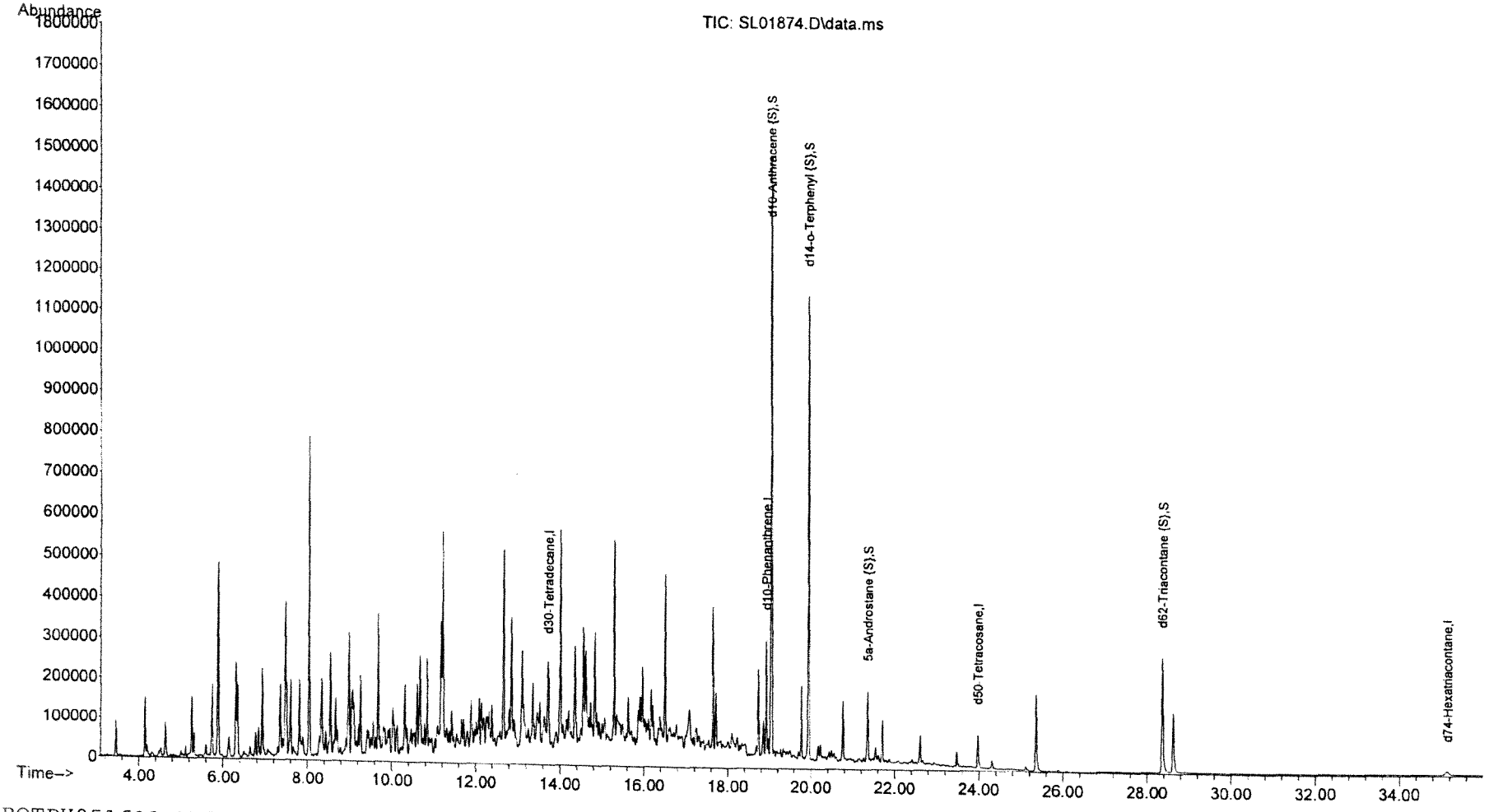
US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01874.D
Acq On : 16 May 2011 20:23
Operator : Syslo
Sample : 5.0K TPH-DRO Std.
Misc : RUE0043
ALS Vial : 74 Sample Multiplier: 1

Quant Time: May 16 20:56:26 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon May 16 20:16:54 2011
Response via : Initial Calibration



DROTPH051611.M Tue May 17 18:05:16 2011

063

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01874.D
Acq On : 16 May 2011 20:23
Operator : Syslo
Sample : 5.0K TPH-DRO Std.
Misc : RUE0043
ALS Vial : 74 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01874.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	14.011	624	1368	2831	rM	8 530728	40295375	100.00%	100.000%

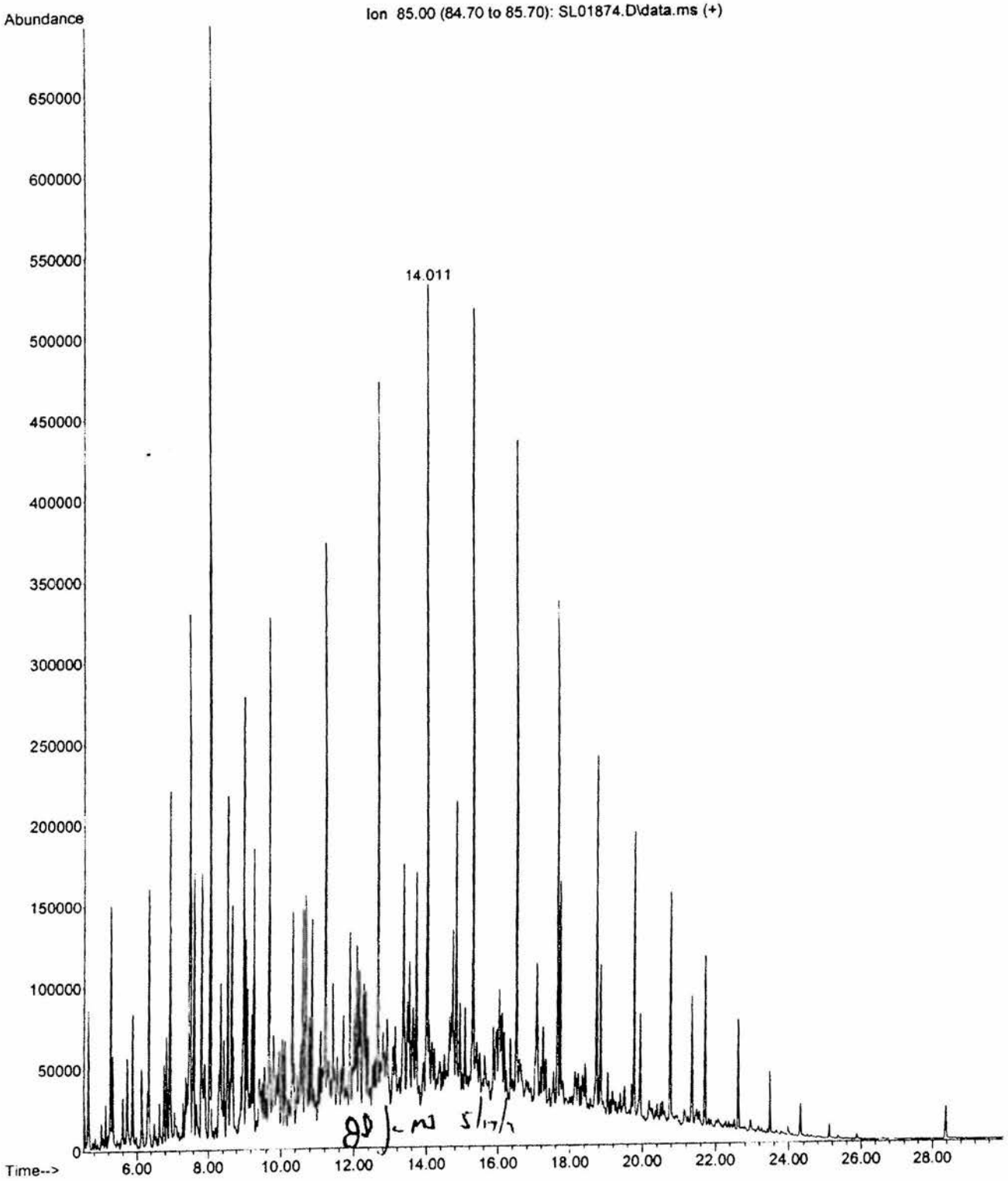
Sum of corrected areas: 40295375

DH/a MS 5/17/11

PH051611.M Tue May 17 18:06:33 2011

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\051611\SL01874.D
Operator : Syslo
Acquired : 16 May 2011 20:23 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 5.0K TPH-DRO Std.
Misc Info : RUE0043
Vial Number: 74



SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01875.D
Acq On : 16 May 2011 21:06
Operator : Syslo
Sample : 100ppm TPH-DRO Std.
Misc : RUE0046
ALS Vial : 75 Sample Multiplier: 1

Quant Time: May 17 18:11:25 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Tue May 17 18:08:56 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	417749	10.00	ug/mL	0.00
5) d30-Tetradecane	13.723	66	145897	10.00	ug/mL	0.00
6) d50-Tetracosane	23.974	66	134713	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.130	66	55469	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	19.056	188	45457	1.09	ug/mL	0.02
Spiked Amount 20.000			Recovery =	5.45%		
3) d14-o-Terphenyl {S}	19.940	244	19720	1.03	ug/mL	0.00
Spiked Amount 20.000			Recovery =	5.15%		
4) 5a-Androstane {S}	21.357	260	2305	0.97	ug/mL	0.00
Spiked Amount 20.000			Recovery =	4.85%		
7) d62-Triacontane {S}	28.360	66	6253	1.62	ug/mL	0.00
Spiked Amount 20.000			Recovery =	8.10%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

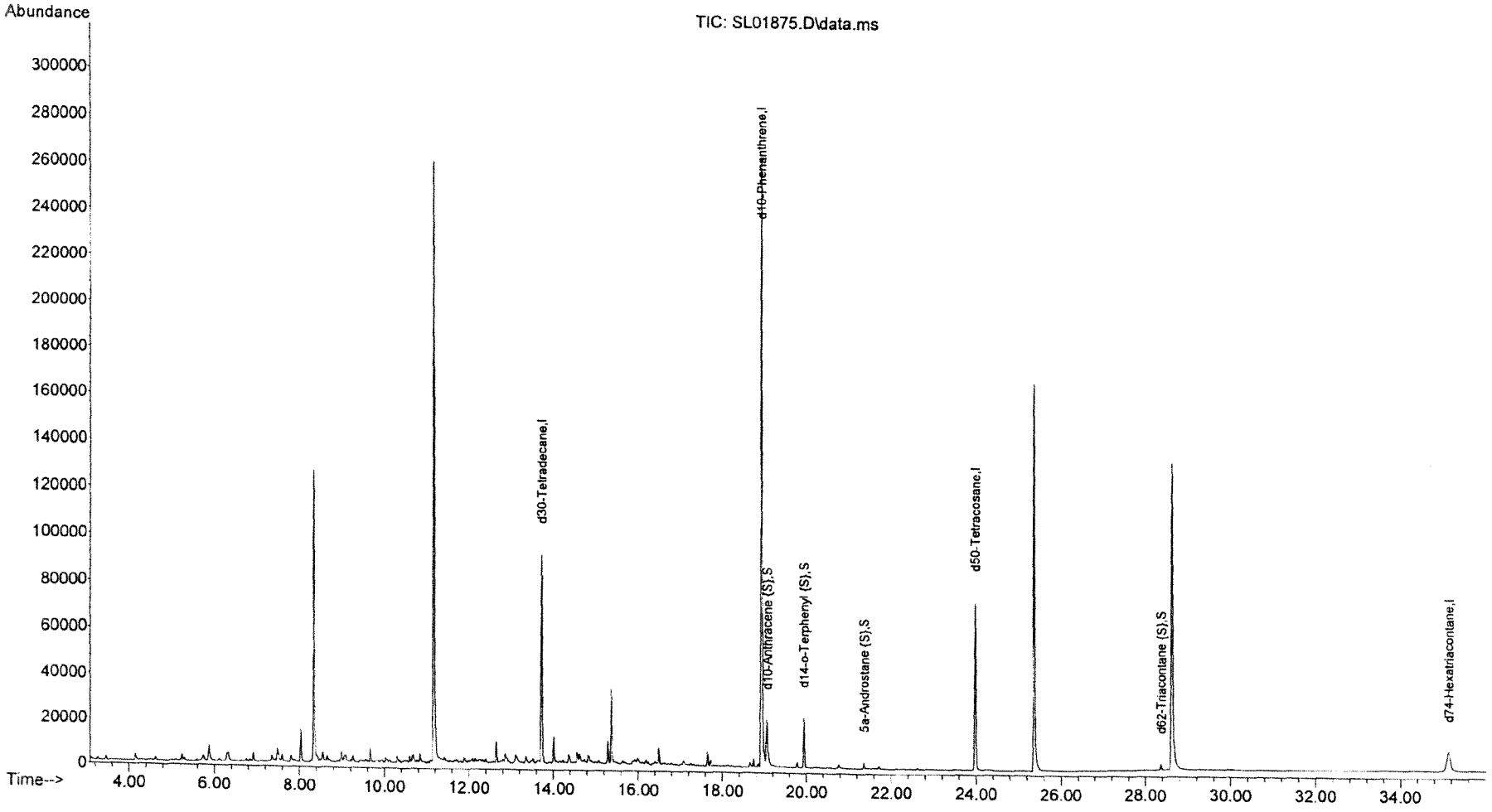
Σ_{TPH} = 98/077

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01875.D
Acq On : 16 May 2011 21:06
Operator : Syslo
Sample : 100ppm TPH-DRO Std.
Misc : RUE0046
ALS Vial : 75 Sample Multiplier: 1

Quant Time: May 17 18:11:25 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Tue May 17 18:08:56 2011
Response via : Initial Calibration



DROTPH051611.M Tue May 17 18:12:27 2011

067

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01875.D
Acq On : 16 May 2011 21:06
Operator : Syslo
Sample : 100ppm TPH-DRO Std.
Misc : RUE0046
ALS Vial : 75 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01875.D\data.ms

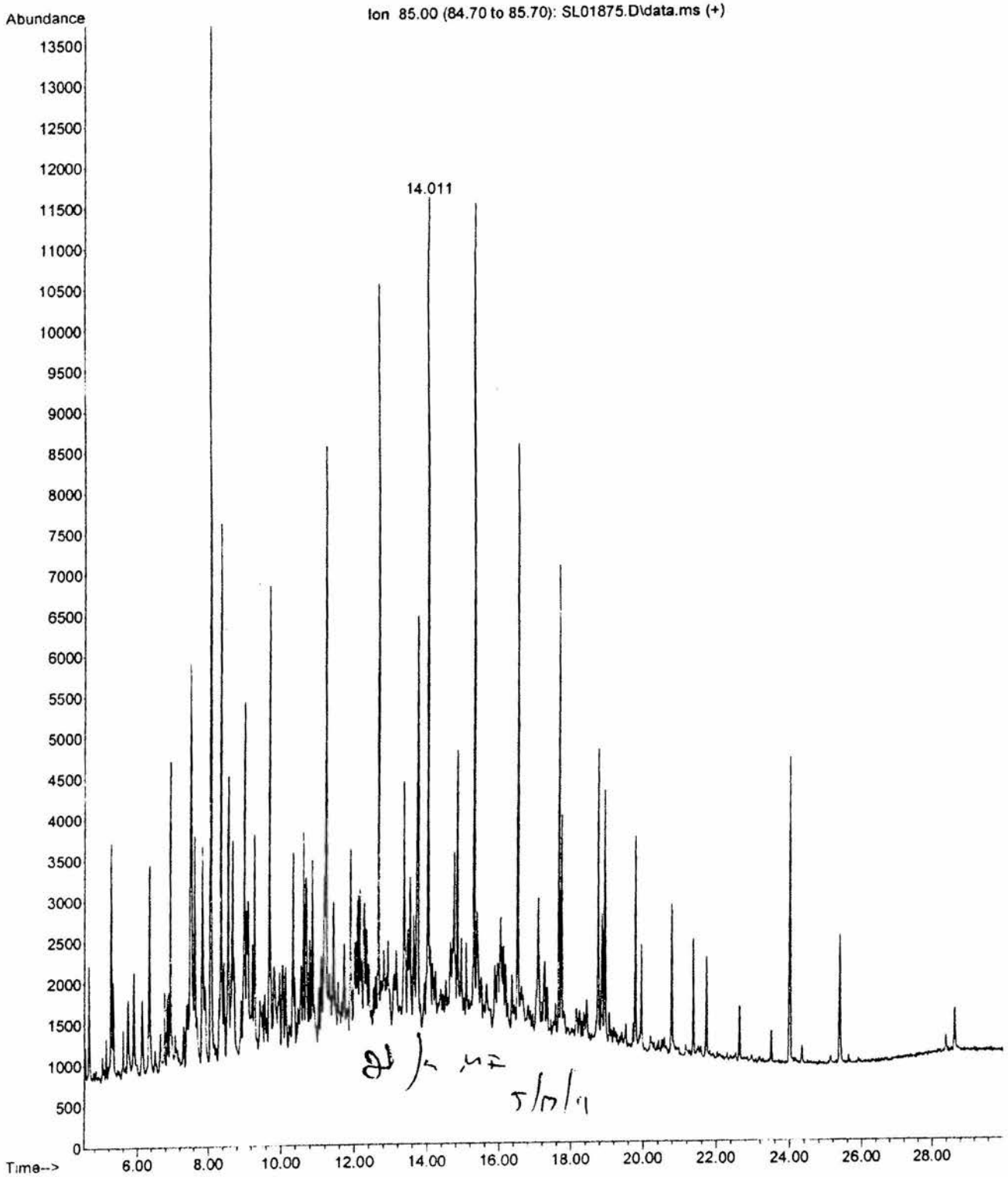
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	14.011	633	1368	2559	rM	4 10898	981077	100.00%	100.000%

Sum of corrected areas: 981077 JMS 5/17/11

PH051611.M Tue May 17 18:13:49 2011

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\051611\SL01875.D
Operator : Syslo
Acquired : 16 May 2011 21:06 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 100ppm TPH-DRO Std.
Misc Info : RUE0046
Vial Number: 75



SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01876.D
Acq On : 16 May 2011 21:49
Operator : Syslo
Sample : 50ppm TPH-DRO Std.
Misc : RUE0047
ALS Vial : 76 Sample Multiplier: 1

Quant Time: May 17 18:14:14 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Tue May 17 18:12:38 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	412580	10.00	ug/mL	0.00
5) d30-Tetradecane	13.724	66	144045	10.00	ug/mL	0.00
6) d50-Tetracosane	23.974	66	128364	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.130	66	53519	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	19.056	188	23904	0.55	ug/mL	0.02
Spiked Amount 20.000			Recovery =	2.75%		
3) d14-o-Terphenyl {S}	19.949	244	8969	0.46	ug/mL	0.00
Spiked Amount 20.000			Recovery =	2.30%		
4) 5a-Androstane {S}	21.357	260	974	0.40	ug/mL	0.00
Spiked Amount 20.000			Recovery =	2.00%		
7) d62-Triacontane {S}	28.360	66	2424	1.30	ug/mL	0.00
Spiked Amount 20.000			Recovery =	6.50%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

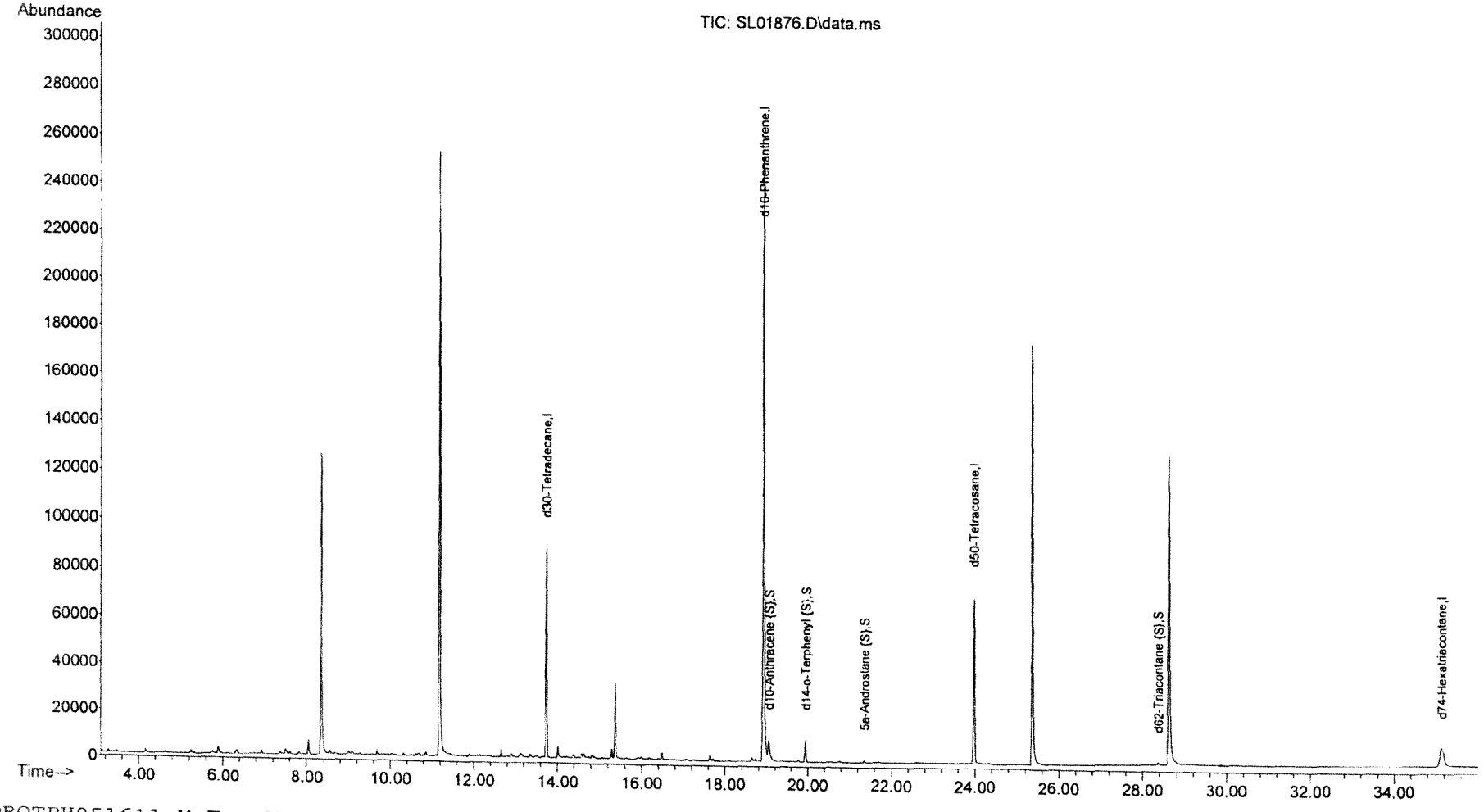
Σ_{TPH} = 430368

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01876.D
Acq On : 16 May 2011 21:49
Operator : Syslo
Sample : 50ppm TPH-DRO Std.
Misc : RUE0047
ALS Vial : 76 Sample Multiplier: 1

Quant Time: May 17 18:14:14 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Tue May 17 18:12:38 2011
Response via : Initial Calibration



DROTPH051611.M Tue May 17 18:15:24 2011

071

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01876.D
Acq On : 16 May 2011 21:49
Operator : Syslo
Sample : 50ppm TPH-DRO Std.
Misc : RUE0047
ALS Vial : 76 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01876.D\data.ms

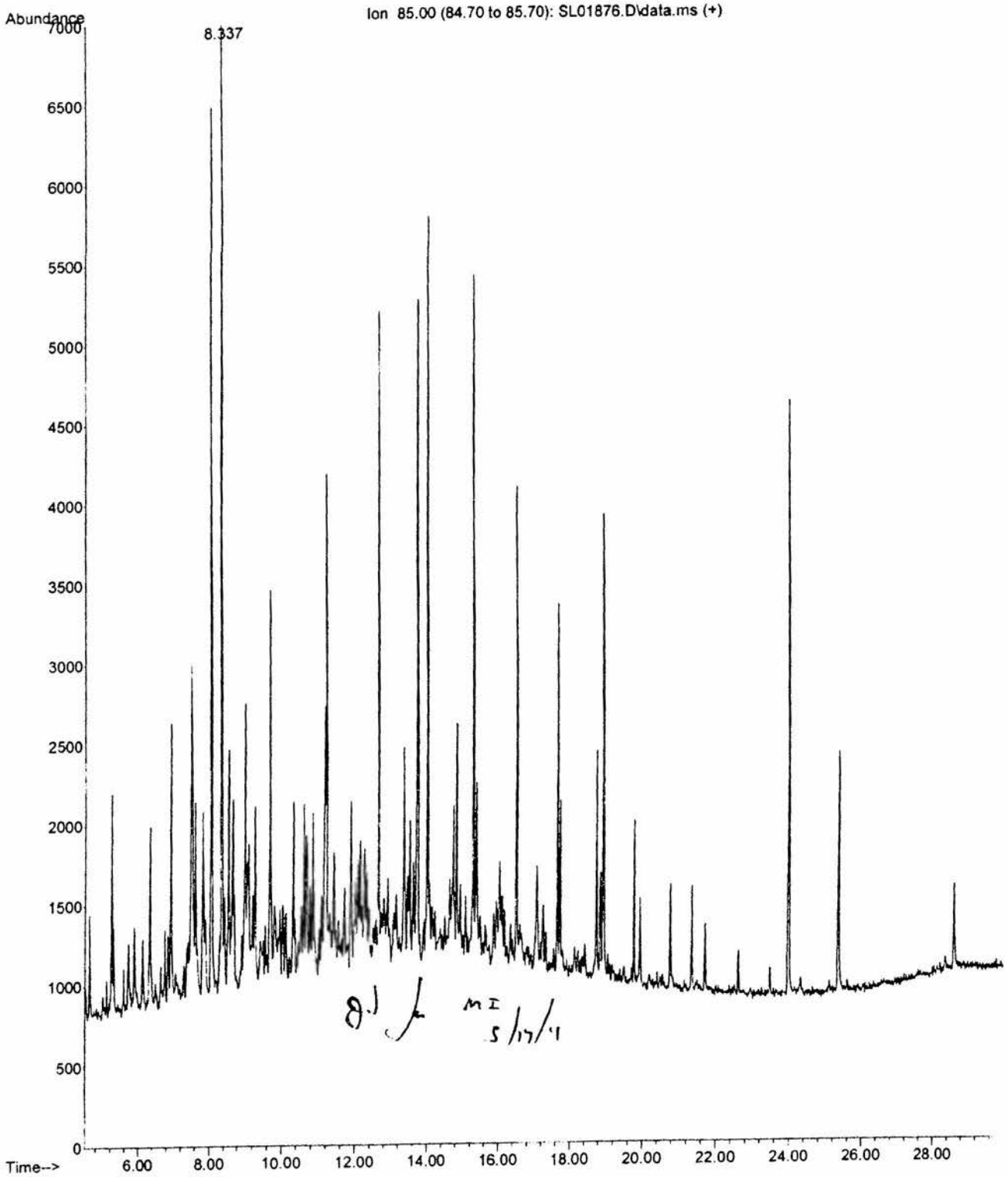
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	8.337	637	658	2405	rM 4	6185	430368	100.00%	100.000%

Sum of corrected areas: 430368

PH051611.M Tue May 17 18:16:27 2011

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\051611\SL01876.D
Operator : Syslo
Acquired : 16 May 2011 21:49 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 50ppm TPH-DRO Std.
Misc Info : RUE0047
Vial Number: 76



US EPA ARCHIVE DOCUMENT

Table X. Results of Initial Calibration Verificaiton (ICV) for DRO-TPH as #2 Diesel
REAC Method 1803: GC/MS Oil Characterization & TPH
Calibration Date: 05/16/11 - GC/MS ID-File DROTPH051611.M
ICV Prepared at 1000 µg/mL ; GC/MS File SL01878.D

Method: SERAS SOP#1803

Compound	Target Conc. µg/mL	ICV Conc. µg/mL	%Recovery	Tentative %Rec.Limits
DRO-TPH as #2 Diesel Fuel	1000	946	95	50-150 %

ICV LIMS# = RUE0049; 1000ppm #2 Diesel from 2nd Source Standard

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01878.D
Acq On : 16 May 2011 23:14
Operator : Syslo
Sample : 1.0K TPH-DRO ICV
Misc : RUE0049 from 2nd source
ALS Vial : 78 Sample Multiplier: 1

Quant Time: May 17 18:18:18 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Tue May 17 18:12:38 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.921	188	389893	10.00	ug/mL	0.00
5) d30-Tetradecane	13.724	66	133926	10.00	ug/mL	0.00
6) d50-Tetracosane	23.974	66	138153	10.00	ug/mL	0.00
8) d74-Hexatriacontane	35.122	66	51687	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	0.000	188	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
3) d14-o-Terphenyl {S}	0.000	244	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
4) 5a-Androstane {S}	0.000	260	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
7) d62-Triacontane {S}	0.000	66	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

Surr

E_{TPH}: 7450171

FCV = 946 / 1000

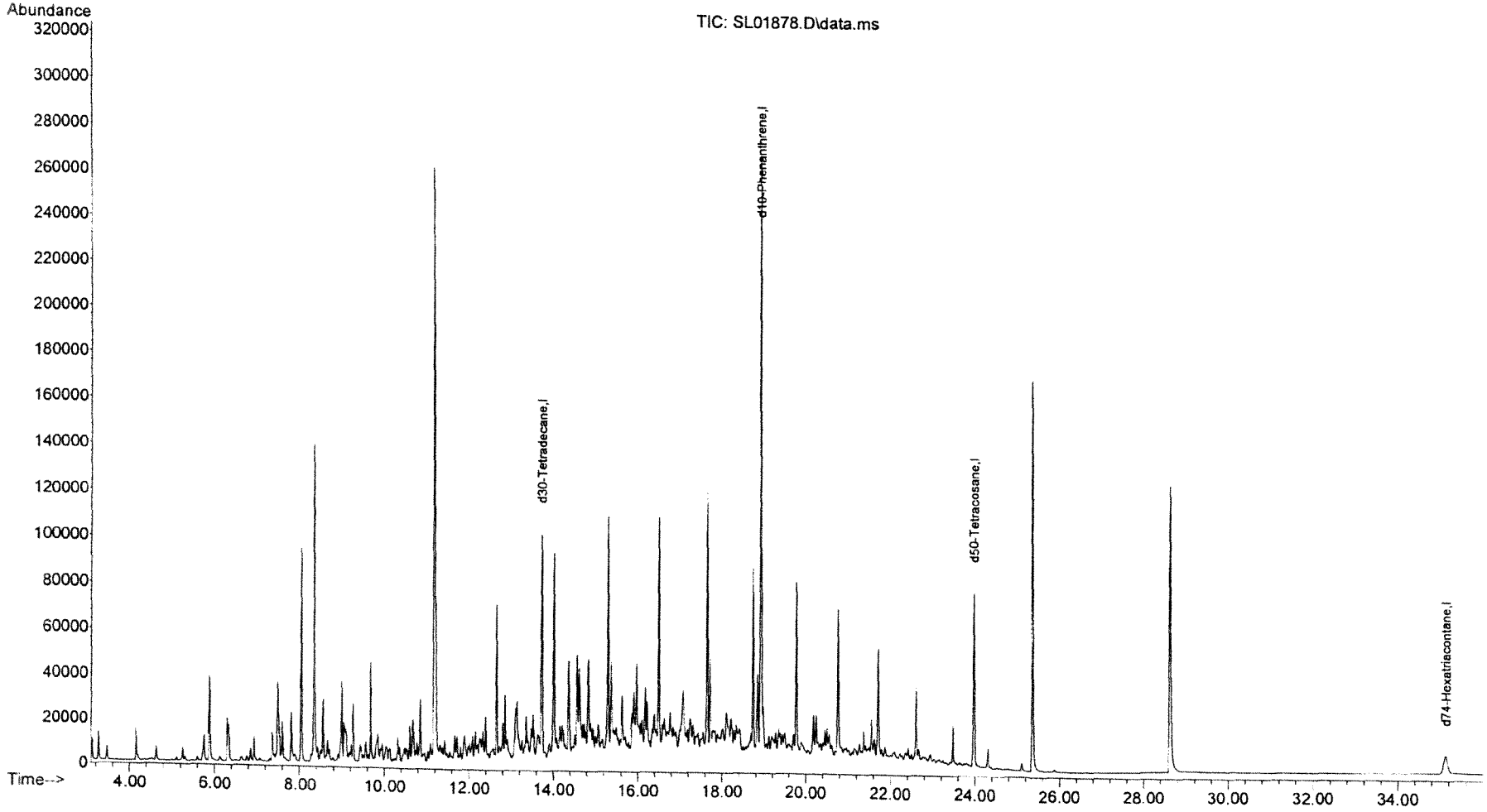
= 5.47

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Quantitation Report (QT Rev ed)

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01878.D
Acq On : 16 May 2011 23:14
Operator : Syslo
Sample : 1.0K TPH-DRO ICV
Misc : RUE0049 from 2nd source
ALS Vial : 78 Sample Multiplier: 1

Quant Time: May 17 18:18:18 2011
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Tue May 17 18:12:38 2011
Response via : Initial Calibration



DROTPH051611.M Tue May 17 18:18:32 2011

076

Area Percent Report

Data Path : C:\msdchem\1\DATA\051611\
Data File : SL01878.D
Acq On : 16 May 2011 23:14
Operator : Syslo
Sample : 1.0K TPH-DRO ICV
Misc : RUE0049 from 2nd source
ALS Vial : 78 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL01878.D\data.ms

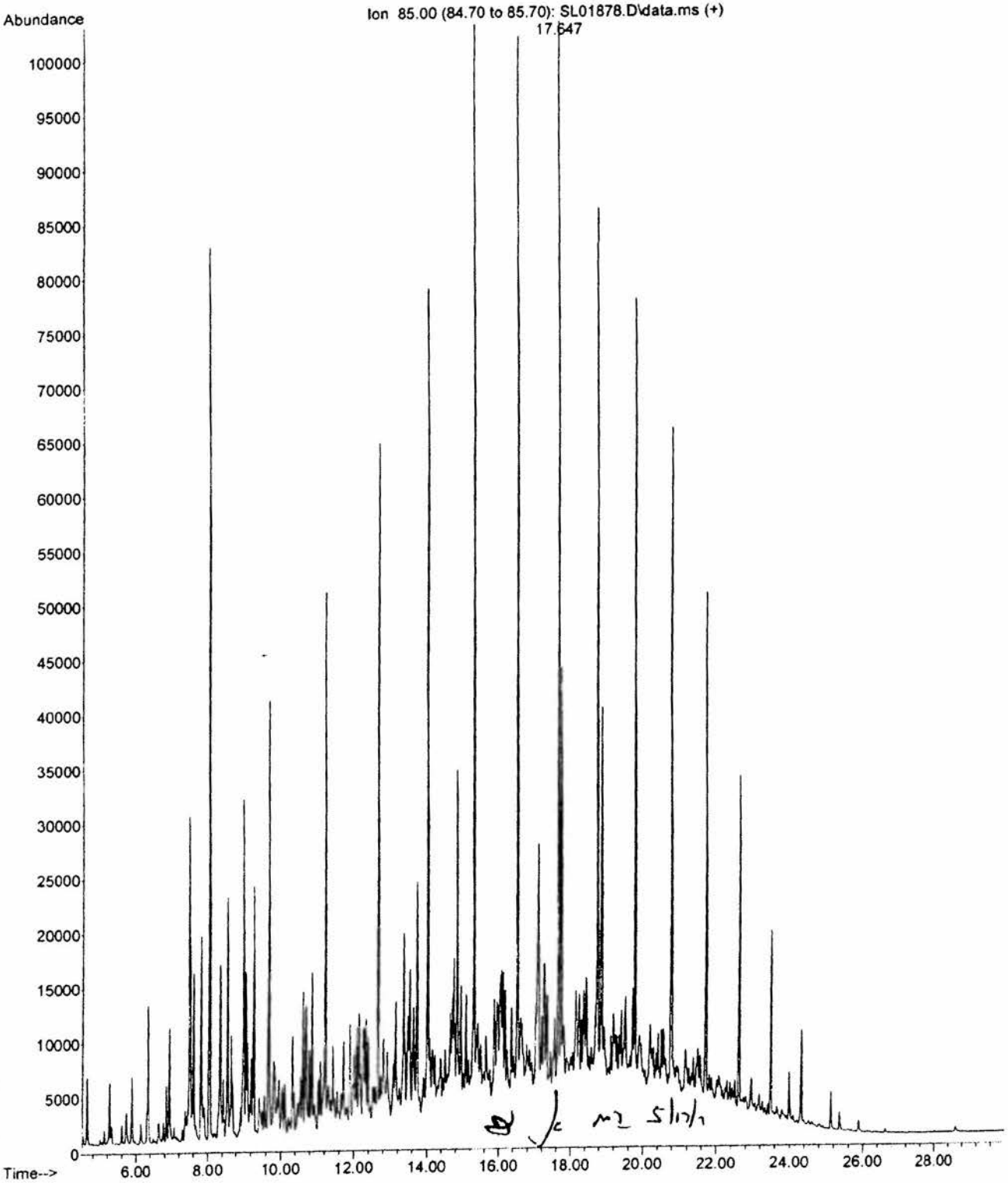
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	17.647	635	1823	2813	rM 5	102642	7450171	100.00%	100.000%

Sum of corrected areas: 7450171

PH051611.M Tue May 17 18:19:05 2011

5/17/11

File :C:\msdchem\1\DATA\051611\SL01878.D
Operator : Syslo
Acquired : 16 May 2011 23:14 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 1.0K TPH-DRO ICV
Misc Info : RUE0049 from 2nd source
Vial Number: 78



US EPA ARCHIVE DOCUMENT

TPH STANDARDS
PREP. LIMS

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0050

Description:	10,000ppm 10W40 Oil	Expires:	11/12/11
Standard Type:	Reagent	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 19:03 by JS
Vendor:	SERAS	Lot Number:	NA

Dilute 20K Stock to 10,oooppm and add IS: No Surrogates

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Motor Oil	NA	10000	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RTK0021	SAE 10W40 Motor Oil 20K	11/12/10	John Syslo	01/30/19	11/19/10 20:42 by JS	0.5
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02

Reviewed By J. Syslo 5/16/11 Date

Analytical Standard Record

ERT/SERAS Laboratory

RUE0049

Description:	1000ppm DRO/TPH ICV	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 18:16 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	B90302060

dilute 2nd Source Stock 50x and add IS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0107	#2 Diesel Fuel LCS 50,000ppm	10/20/09	John Syslo	10/15/19	10/20/09 13:52 by JS	0.02
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02

Reviewed By J. Syslo 5/16/11 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RUE0048

Description:	50ppm DRO + 0.5ppm Surr TPH ICAL	Expires:	11/12/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 18:11 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:
Used 25µL syringe to measure 2.5µL ; not 10µL syringe (RUE0047)

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	0.5	ug/mL
Terphenyl-d14	NA	0.5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	0.5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	0.5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.0025

Reviewed By: J. Syslo 5/16/11
Date

Analytical Standard Record

ERT/SERAS Laboratory

RUE0047

Description:	50ppm DRO + 0.5ppm Surr TPH ICAL	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 17:50 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	0.5	ug/mL
Terphenyl-d14	NA	0.5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	0.5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	0.5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.0025

Reviewed By J. Syslo 5/16/11 Date

SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0046

Description:	100ppm DRO + 1.0ppm Surr TPH ICAL	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 17:49 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1	ug/mL
Terphenyl-d14	NA	1	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		100	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	1	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	1	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.005

Reviewed By _____ Date 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0045

Description:	500ppm DRO + 5.0ppm Surr TPH ICAL	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:20 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	5	ug/mL
Terphenyl-d14	NA	5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.025

Reviewed By 5/16/11 Date

Analytical Standard Record
ERT/SERAS Laboratory
RUE0044

Description:	1000ppm DRO + 10ppm Surr TPH ICAL	Expires:	11/12/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:18 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	10	ug/mL
Terphenyl-d14	NA	10	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.05

Reviewed By _____ Date 5/16/11

SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0043

Description:	5k DRO + 50ppm Surr Cal.	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:19 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	50	ug/mL
Terphenyl-d14	NA	50	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	50	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	50	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.25

Reviewed By _____ Date 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0042

Description:	10k DRO + 100ppm Surr Cal.	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:15 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.5

Reviewed By J. Syslo Date 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0041

Description:	20K DRO & 200ppm Surr. ICAL Stock	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65/35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:04 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	A9070168

Primary Source Diesel + Surrogate mixture for DRO/TPH ICAL. Make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Oil Fingerprint		20000	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
5a-Androstane	438-22-2	200	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0102	#2 Diesel Fuel Mix 50,000 ppm	10/20/09	John Syslo	04/18/19	05/13/11 16:18 by JS	0.8
RUE0036	2000ppm Oil Surrogate Stock	05/13/11	John Syslo	11/13/11	05/13/11 15:13 by JS	0.2

Reviewed By J. Syslo 5/16/11 Date

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0040

Description:	2000ppm TPH (DRO) LCS Spike (2nd)	Expires:	11/13/11
Standard Type:	Analyte Spike	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	20	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 17:09 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	B9030206 (2nd source diesel)

Dilute 50K 2nd source Diesel Standard 25x with DCM/Hexane :
= 800µL Rsj0107 into FV 20.0mL DCM/Hexane (RUC0022) = 2000ppm

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0107	#2 Diesel Fuel LCS 50,000ppm	10/20/09	John Syslo	10/15/19	10/20/09 13:52 by JS	0.8

Reviewed By 5/13/11 Date

Analytical Standard Record

ERT/SERAS Laboratory

RUE0039

Description:	400ppm DRO/TPH MDL Spike	Expires:	11/13/11
Standard Type:	Analyte Spike	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	20	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 16:22 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	A9070168 for DRO

Dilute 160µL of 50,000ppm into 20mL DCM/Hexane

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		400	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0102	#2 Diesel Fuel Mix 50,000 ppm	10/20/09	John Syslo	04/18/19	05/13/11 16:18 by JS	0.16

Reviewed By _____ Date 5/13/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RUE0038

Description:	40ppm Oil Surrogate	Expires:	11/13/11
Standard Type:	Surrogate Spike	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	CENTRAL
Vials:	1	Last Edit:	05/13/11 15:42 by JS
Vendor:	SERAS	Lot Number:	RUC00226

Dilute 2000ppm RUE0036 (500µL in 25mL) with DCM/Hexane
Spike 500 µL per sample for FV of 1.0 mL

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
5a-Androstane	438-22-2	40	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0036	2000ppm Oil Surrogate Stock	05/13/11	John Syslo	11/13/11	05/13/11 15:13 by JS	0.5

Reviewed By 5/13/11 Date

SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0037

Description:	500ppm Oil IS Mix	Expires:	11/13/11
Standard Type:	Internal Standar	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 15:33 by JS
Vendor:	SERAS	Lot Number:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RUE0035) and 2000ppm BNA IS (RTF0070) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RTF0070	Stock SVOC Internal Std. Mix,	2006/16/10	** Vendor **	11/05/11	05/05/11 12:51 by JS	0.5
RUE0035	TPH IS Mix: 2000ppm : 3 Compo	05/13/11	John Syslo	11/13/11	05/13/11 15:10 by JS	0.5

Reviewed By 5/13/11 Date

US EPA ARCHIVE DOCUMENT

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Analytical Standard Record

ERT/SERAS Laboratory

RUE0036

Description:	2000ppm Oil Surrogate Stock	Expires:	11/13/11
Standard Type:	Surrogate Spike	Prepared:	05/13/11
Solvent:	DCM/CS2 75:25	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 15:13 by JS
Vendor:	REAC	Lot Number:	REAC

Mix of 4 neat surrogates for Oil Surrogate Stock. 0.1g each

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2000	ug/mL
Terphenyl-d14	NA	2000	ug/mL
Anthracene-d10	1719-06-8	2000	ug/mL
5a-Androstane	438-22-2	2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0090	Neat Triacontane-d62	06/29/09	John Syslo	06/26/19	06/30/09 13:27 by JS	0.1
RSF0092	Neat o-Terphenyl-d14	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0093	Neat Anthracene-d10	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0122	Neat 5a-Androstane	06/30/09	John Syslo	12/27/19	06/30/09 13:25 by JS	0.1

Reviewed By 5/13/11 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0035

Description:	TPH IS Mix: 2000ppm : 3 Compounds	Expires:	11/13/11
Standard Type:	Internal Standar	Prepared:	05/13/11
Solvent:	DCM/CS2 4:1	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 15:10 by JS
Vendor:	NA	Lot Number:	NA

2000ppm 3 Compound IS Mix for TPH. To be mixed with BNA IS

Analyte	CAS Number	Concentration	Units
n-Tetradecane-d30		2000	mg/Kg
n-Tetracosane-d50	16416-32-3	2000	mg/Kg
n-Hexatriacontane-d74	16416-34-5	2000	mg/Kg

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0123	Neat D50-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 14:26 by JS	0.1
RSF0124	Neat D74-n-Hexatriacontane	06/30/09	John Syslo	12/27/19	06/30/09 14:33 by JS	0.1
RSF0126	Neat D30-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 13:56 by JS	0.1

Reviewed By J Syslo 5/13/11 Date

Attachment 01: Summary of Enbridge Oil Samples Received: 02/01/12 - 03/01/12

COC#	Sample ID	Date Received	Analysis Requested	Date Analyzed	Matrix / Sample Description
SERAS-017-02/01/12-0001	SERAS-017-0001	2/1/2012	TPH	2/2/2012	This was one of two 5 gallon buckets recieved that contained water and rever sediment. The bucket was 3/4 full and the aqueous/sediment layer was approximately equal. The dried sediment was extracted and analyzed on 2/2/12 for TPH.
	SERAS-017-0000	*This sample is referred to as the "skimmed" oil. I took a one liter beaker and grabbed a subsample of this bucket which resulted in ~300mL of sediment and bottom debris topped with ~500mL water. I skimmed a small quantity of the visible product that floated to the surface and placed it in 1.0mL of DCM/hexane and analyzed this for fingerprint analysis. I did not obtain a weight for the "skimmed" product. This sample is referred to as either the "skimmed" product or sample 00X in some of the chromatograms since it was not officially logged in on a COC.*				
SERAS-017-02/08/12-0002	SERAS-017-0002	2/1/2012	TPH	2/15/2012	The sediment of this sample was extracted on 2/14/12 and analyzed for TPH on 2/15/12. The fingerprints of this sample and the TPH were compared to SERAS-017-0001
SERAS-017-02/21/12-0003	SERAS-017-0003	2/21/2012	TPH* fingerprint	2/27/2012	Pure product: Called "product of recovered oil in tank". Mobile black liquid with characteristic odor of "crude" oil. Was not analyzed for TPH since it was product. Analyzed 2/27/12 for fingerprinting.
SERAS-017-02/23/12-0004	SERAS-017-0004**	2/23/2012	TPH* fingerprint	2/27/2012	The matrix indicates "pure product" but the sample was an emulsion of thick black product and fine silt. When the product was separated from the water/silt, it was a thick-black-viscous product. It was analyzed on 2/27 for fingerprinting, but not TPH since the TPH/silt was not the issue. Also referred to as "tar from excavation"
072223 Conestoga-Rovers & Associates	SERAS-017-0005 SERAS-017-0006	3/1/2012	fingerprint	3/1/2012	These two samples were recieved as duplicates in 40mL VOA vials on the Conestoga-Rovers chain of custody. The SERAS numbers were assigned to the sample numbers listed on the COC and information is provided below:
	SERAS-017-0005	is Sample No. WCS-6B-072223-092910-JPS-KA-002-20. two bottles recieved with custody seal # 701795. This is a mobile-black crude oil sample.			
	SERAS-017-0006	is Sample No. CL-6B-072223-092710-JPS-KA-001-33. two bottles recieved with custody seal # 701688. This is a mobile-black crude oil sample. Both of these samples were analyzed on 3/1/12 for fingerprint analysis.			

* Chain of custody indicates TPH analysis. These were samples contained pure product and fingerprint analysis was performed.
** This oil from this sample was processed and used to perform the biodegradation studies.

Summary of Additional Analytical Work Performed: Biodegradatation Studies : 03/01/12 - 04/18/12

On 03/07/2012 a portion of sample SERAS-017-0004 was processed to remove all silt, debris, water, and baked at 125 for 1 hour degrees to simulate autoclaving. Approzximately 6.5 grams was delivered to the SERAS microbiology lab to perform degradation studies. A GC/MS TPH calibration range was performed on 03/22/2012 using the processed oil, and TPH results calculated using the "site" oil.

COC#	# of Samples	Date Received	Analysis Requested	Date Analyzed	Comments
SERAS-017-03/15/12-0005	7	3/19/2012	TPH ***	3/23/2012	**Day 0 Samples** of biodegradation study. Results in **Table 1.0.**
SERAS-017-03/22/12-0006	6	3/22/2012	TPH ***	3/23/2012	**Day 14 Samples** of biodegradation study. Results in **Table 2.0.** *After reviewing the Day 0 and Day14 TPH results, gravimetric analysis was perfomed on 2 select Day 0 and Day 14 samples to obtain information concerning the asphaltene content,* ***Table 2.1*** *shows the TPH as calculated using the GC/MS system and TPH calculated by gravimetric analysis of the TPH extract for the two selected samples.*
SERAS-017-04/06/12-0007	7	4/12/2012	TPH ***	4/18/2012	**Day 28 Samples** of biodegradation study. Results in **Table 3.0**

*** TPH analysis was performed using SERAS GC/MS TPH Method #1841 and additional fingerprint chromatograms were submitted to supply visual evidence of degradation.

COC + Work Order

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-02/01/12-0001

Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R202001

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
01	SERAS-017-0001	275-44-21	TPH-DRO	River Sediment	1/31/2012	1	68-oz HDPE	None	Y

TFM 2/1/12

RECEIVED
FEB 2 2012

Special Instructions:

SAMPLES TRANSFERRED FROM CHAIN OF CUSTODY #

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
1/Analysis	T. F. Miller	2/1/12	[signature]	2/1/12	11:00	All/Analysis	[signature]	2/2/12	[signature]	2/2/12	10:45

E

WORK ORDER

Printed: 6/1/2012 4:37:43PM

R202001

ERT/SERAS Laboratory

Bucket Sed

Client: US EPA/ERT (Edison)
Project: Oil Spill Response Support

Project Manager: Vinod Kansal
Project Number: SERAS-017

Report To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6747
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Alan Humphrey
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6748
Fax: (732) 321-6724

TPH + Fing

Date Due: 02/22/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Logged In By: Lawrence Martin

Date Received: 02/01/12 12:35
Date Logged In: 02/01/12 12:35

Samples Received at: 23°C
Custody Seals No Received On Ice No
Containers Intact Yes
COC/Labels Agree Yes
Preservation Confir No

Analysis	Due	TAT	Expires	Comments
R202001-01 SERAS-017-0001 [Soil] Sampled 01/31/12 00:00 Eastern				**275-44-21**
Fingerprint	02/01/12 16:00	8	02/07/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0101

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-02/08/12-0002
Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R202008

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
01	SERAS-017-0002	275-45-34	TPH-DRO	River Sediment	2/8/2012	1	68-oz HDPE	None	Y

TFm 2/8/12

RECEIVED
FEB 14 2012

Special Instructions:	SAMPLES TRANSFERRED FROM CHAIN OF CUSTODY #

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
1/analysis	T. F. Miller	2/8/12	[signature]	2/8/12	15:00	All Analysis	[signature]	2/13/12	[signature]	2/13/12	14:15

E

WORK ORDER

R202008

ERT/SERAS Laboratory

Printed: 6/1/2012 4:40:03PM

Bucket Jed

Client: US EPA/ERT (Edison)
Project: Oil Spill Response Support

Project Manager: Vinod Kansal
Project Number: SERAS-017

Report To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6747
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Alan Humphrey
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6748
Fax: (732) 321-6724

Fingerprint + TPH

Date Due: 02/29/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Logged In By: Lawrence Martin

Date Received: 02/08/12 15:43
Date Logged In: 02/08/12 15:43

Samples Received at: **23°C**

Custody Seals	No	Received On Ice	No
Containers Intact	Yes		
COC/Labels Agree	Yes		
Preservation Confit	No		

Analysis	Due	TAT	Expires	Comments
R202008-01 SERAS-017-0002 [Soil] Sampled 02/08/12 00:00 Eastern				**275-45-34**
Fingerprint	02/08/12 16:00	8	02/15/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-02/21/12-0003
Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R202016

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
01	SERAS-017-0003	275-55-05	TPH-DRO	Pure Oil Source	2/21/2012	1	2 oz amber	4 C	Y

TFM
2/21/12

RECEIVED
FEB 22 2012

Special Instructions: TPH and fingerprint analysis are requested.

SAMPLES TRANSFERRED FROM CHAIN OF CUSTODY #

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
1/analysis	T. F. Miller	2/21/12	[signature]	2/21/12	14:30	All/Analysis	[signature]	2/22/12	[signature]	2/22/12	0905

E

SERAS-017-DTM-011413_3

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-02/23/12-0004

Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R202016

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
02	SERAS-017-0004	275-56-24	TPH-DRO	Pure Oil Source	2/23/2012	1	2 oz amber	4 C	Y

T. F. Miller 2/23/12

RECEIVED FEB 23 2012

Special Instructions: Please analyze the sample for TPH content and fingerprint analysis.

SAMPLES TRANSFERRED FROM CHAIN OF CUSTODY #

Received Room Temp Jay 2/23/12

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
1/analysis	T. F. Miller	2/23/12	[signature]	2/23/12	16:00	All/Analysis	[signature]	2/25/12	[signature]	2/23/12	16:06

0104

E

WORK ORDER

Printed: 6/1/2012 4:37:41PM

R202016

ERT/SERAS Laboratory

Client: US EPA/ERT (Edison)
Project: Oil Spill Response Support

Project Manager: Vinod Kansal
Project Number: SERAS-017

Report To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6747
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Alan Humphrey
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6748
Fax: (732) 321-6724

Date Due: 03/13/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Logged In By: Lawrence Martin

Date Received: 02/21/12 16:07
Date Logged In: 02/21/12 16:07

Samples Received at: **23°C**
Custody Seals No Received On Ice No
Containers Intact Yes
COC/Labels Agree Yes
Preservation Confir No

Analysis	Due	TAT	Expires	Comments
R202016-01 SERAS-017-0003 [Oil] Sampled 02/21/12 00:00 Eastern				**275-55-05**
Oil Fingerprint	02/22/12 16:00	8	02/28/12 00:00	
R202016-02 SERAS-017-0004 [Oil] Sampled 02/23/12 00:00 Eastern				**275-56-24**
Oil Fingerprint	02/22/12 16:00	8	03/01/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413 3
0106

CHAIN OF CUSTODY RECORD

CONESTOGA-ROVERS & ASSOCIATES
2055 Niagara Falls Blvd
Niagara Falls, NY 14304

SHIPPED TO (Laboratory Name):
Raymond Siegener, PhD
Alpha Analytical
320 Forbes Blvd
Mansfield, MA 02048

REFERENCE NUMBER:
Line 6B Sampling
072223

SAMPLER'S SIGNATURE: Christa Nunn PRINTED NAME: Christa Nunn

SEQ. No.	DATE	TIME	SAMPLE No.	SAMPLE TYPE	No. of Containers	PARAMETERS	REMARKS
1 (-3)	2/22/12	1600	WCS-LB3-072223-092910-JPS-KA-002-20	CO	1	(5)	Bottle Custody Seal # 701795
1 (-4)	1	1	CL-LB3-072223-092710-JPS-KA-001-33	1	1	(6)	7016888
							*Sample Petroleum Crude Oil, 3, UN1267, PGII

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MAR 1 2012

ENBRIDGE O&I

(5) = SERAS-017-0005
(6) = SERAS-017-0006

TOTAL NUMBER OF CONTAINERS: 2 HEALTH/CHEMICAL HAZARDS:

RELINQUISHED BY: ① Christa Nunn	DATE: 2/22/12 TIME: 1615	RECEIVED BY: ① FedEx	DATE: TIME:
RELINQUISHED BY: ② FedEx	DATE: 2/24/12 TIME: 1050	RECEIVED BY: ②	DATE: 2/24/12 TIME: 1050
RELINQUISHED BY: ③	DATE: 2/27/12 TIME: 1135	RECEIVED BY: ③ UPS	DATE: TIME:

METHOD OF SHIPMENT: Fed Ex Ground WAY BILL No. 391687133007659 1

White —Fully Executed Copy
Yellow —Receiving Laboratory Copy
Pink —Shipper Copy
Goldenrod —Sampler Copy

SAMPLE TEAM: Christa Nunn

RECEIVED FOR LABORATORY BY:
DATE: 3/01/2012 TIME: 15:50

Nº CRA 25320

1001 (D) APR 28/97(NF) REV. 0 (F-15)

WORK ORDER

Printed: 6/1/2012 4:37:40PM

R203003

ERT/SERAS Laboratory

Client: US EPA/ERT (Edison)
Project: Enbridge Oil Spill

Project Manager: Vinod Kansal
Project Number: SERAS-017

Report To:
US EPA/ERT (Edison)
Alan Humphrey
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6748
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Alan Humphrey
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6748
Fax: (732) 321-6724

Date Due: 03/22/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Date Received: 03/01/12 14:21

Logged In By: Lawrence Martin
Date Logged In: 03/06/12 14:21

Samples Received at: 23°C
Custody Seals No Received On Ice No
Containers Intact No
COC/Labels Agree No
Preservation Confir No

Analysis	Due	TAT	Expires	Comments
R203003-01 Does Not Exist [Other] Sampled 03/05/12 00:00 Eastern				
R203003-02 Does Not Exist [Other] Sampled 03/05/12 00:00 Eastern				
R203003-03 SERAS-017-0005 [Other] Sampled 02/22/12 00:00 Eastern				**WCS-6B-072223-092910-JPS-KA-002-20**
Oil Fingerprint	03/01/12 16:00	8	02/29/12 00:00	
R203003-04 SERAS-017-0006 [Other] Sampled 02/22/12 00:00 Eastern				**CL-6B-072223-092710-JPS-KA-001-33**
Oil Fingerprint	03/01/12 16:00	8	02/29/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

E1/0

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-03/15/12-0005
Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R203011

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
01	SERAS-017-0007	275-78-10	TPH-DRO	Nutrient Medium	3/6/2012	1	4-oz. amber	formaldehyde	N
02	SERAS-017-0008	275-78-11	TPH-DRO	Nutrient Medium	3/6/2012	1	4-oz. amber	formaldehyde	N
03	SERAS-017-0009	275-78-12	TPH-DRO	Nutrient Medium	3/8/2012	1	4-oz. amber	formaldehyde	N
04	SERAS-017-0010	275-78-13	TPH-DRO	Nutrient Medium	3/8/2012	1	4-oz. amber	formaldehyde	N
05	SERAS-017-0011	275-78-14	TPH-DRO	Nutrient Medium	3/8/2012	1	4-oz. amber	formaldehyde	N
06	SERAS-017-0012	275-78-15	TPH-DRO	Nutrient Medium	3/8/2012	1	4-oz. amber	formaldehyde	N
07	SERAS-017-0013	275-78-16	TPH-DRO	Nutrient Medium	3/14/2012	3	4-oz. amber	formaldehyde	Y

TTM
3/15/12

Special Instructions: Each sample contains 65 mL (nutrient medium),10 mL methylene chloride, 6.5 mL 37% v/v formaldehyde and 200 mg TPH. Extract the entire sample. Please use sample SERAS-017-0013 for matrix, matrix spike and matrix spike duplicate analyses.

SAMPLES TRANSFERRED FROM
CHAIN OF CUSTODY #

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
9/analysis	T. Ferrell Miller	3/15/12	[signature]	3/16/12	8:00	All Analysis	[signature]	3/19/12	[signature]	3/19/12	11:20

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SERAS-017-DTM-011413_3

0108

WORK ORDER

Printed: 6/1/2012 4:37:40PM

R203011

ERT/SERAS Laboratory

Client: US EPA/ERT (Edison)
Project: Bioremediation Support for Oil

Project Manager: Vinod Kansal
Project Number: 0-017

Report To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6747
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6747
Fax: (732) 321-6724

Date Due: 04/06/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Logged In By: Lawrence Martin

Date Received: 03/16/12 09:49
Date Logged In: 03/19/12 09:49

Samples Received at: 23°C
Custody Seals No Received On Ice No
Containers Intact Yes
COC/Labels Agree Yes
Preservation Confir No

Analysis	Due	TAT	Expires	Comments
R203011-01 SERAS-017-0007 [Water] Sampled 03/06/12 00:00 Eastern				**275-78-10**
Oil Fingerprint	03/16/12 16:00	8	03/13/12 00:00	
R203011-02 SERAS-017-0008 [Water] Sampled 03/06/12 00:00 Eastern				**275-78-11**
Oil Fingerprint	03/16/12 16:00	8	03/13/12 00:00	
R203011-03 SERAS-017-0009 [Water] Sampled 03/08/12 00:00 Eastern				**275-78-12**
Oil Fingerprint	03/16/12 16:00	8	03/15/12 00:00	
R203011-04 SERAS-017-0010 [Water] Sampled 03/08/12 00:00 Eastern				**275-78-13**
Oil Fingerprint	03/16/12 16:00	8	03/15/12 00:00	
R203011-05 SERAS-017-0011 [Water] Sampled 03/08/12 00:00 Eastern				**275-78-14**
Oil Fingerprint	03/16/12 16:00	8	03/15/12 00:00	
R203011-06 SERAS-017-0012 [Water] Sampled 03/08/12 00:00 Eastern				**275-78-15**
Oil Fingerprint	03/16/12 16:00	8	03/15/12 00:00	
R203011-07 SERAS-017-0013 [Water] Sampled 03/08/12 00:00 Eastern				**275-78-16**
Oil Fingerprint	03/16/12 16:00	8	03/15/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0110

END

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-03/22/12-0006

Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R203015

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
01	SERAS-017-0014	275-82-07	TPH-DRO	Nutrient Medium	3/20/2012	1	4-oz. amber	formaldehyde	N
02	SERAS-017-0015	275-82-08	TPH-DRO	Nutrient Medium	3/20/2012	1	4-oz. amber	formaldehyde	N
03	SERAS-017-0016	275-82-09	TPH-DRO	Nutrient Medium	3/22/2012	1	4-oz. amber	formaldehyde	N
04	SERAS-017-0017	275-82-10	TPH-DRO	Nutrient Medium	3/22/2012	1	4-oz. amber	formaldehyde	N
05	SERAS-017-0018	275-82-11	TPH-DRO	Nutrient Medium	3/22/2012	1	4-oz. amber	formaldehyde	N
06	SERAS-017-0019	275-82-12	TPH-DRO	Nutrient Medium	3/22/2012	1	4-oz. amber	formaldehyde	N

TFM
3/22/12

Special Instructions: Each sample consists of 65 mL water (nutrient medium), 10 mL methylene chloride, 6.5 mL 37% v/v formaldehyde and 200 mg TPH. Samples SERAS-017-0018 and SERAS-017-0019 contain 17.5 mL methylene chloride rather than 10 ml.

SAMPLES TRANSFERRED FROM
CHAIN OF CUSTODY #

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
6/analysis	T.F. Miller	3/22/12	[signature]	3/22/12	15:00	All/Analysis	[signature]	3/22/12	[signature]	3/22/12	15:30

MAR 22 2012

WORK ORDER

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R203015

ERT/SERAS Laboratory

Client: US EPA/ERT (Edison)
Project: Bioremediation Support for Oil

Project Manager: Vinod Kansal
Project Number: 0-017

Report To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6747
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6747
Fax: (732) 321-6724

Date Due: 04/12/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Logged In By: Lawrence Martin

Date Received: 03/22/12 15:28
Date Logged In: 03/22/12 15:28

Samples Received at: 23°C
Custody Seals No
Containers Intact Yes
COC/Labels Agree Yes
Preservation Confir No
Received On Ice No

Analysis	Due	TAT	Expires	Comments
R203015-01 SERAS-017-0014 [Water] Sampled 03/20/12 00:00 Eastern				**275-82-07**
Oil Fingerprint	03/22/12 16:00	8	03/27/12 00:00	
R203015-02 SERAS-017-0015 [Water] Sampled 03/20/12 00:00 Eastern				**275-82-08**
Oil Fingerprint	03/22/12 16:00	8	03/27/12 00:00	
R203015-03 SERAS-017-0016 [Water] Sampled 03/22/12 00:00 Eastern				**275-82-09**
Oil Fingerprint	03/22/12 16:00	8	03/29/12 00:00	
R203015-04 SERAS-017-0017 [Water] Sampled 03/22/12 00:00 Eastern				**275-82-10**
Oil Fingerprint	03/22/12 16:00	8	03/29/12 00:00	
R203015-05 SERAS-017-0018 [Water] Sampled 03/22/12 00:00 Eastern				**275-82-11**
Oil Fingerprint	03/22/12 16:00	8	03/29/12 00:00	
R203015-06 SERAS-017-0019 [Water] Sampled 03/22/12 00:00 Eastern				**275-82-12**
Oil Fingerprint	03/22/12 16:00	8	03/29/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

EPA/ERT
SERAS, Edison, NJ
EPA Contract Number: EP-W-09-031

CHAIN OF CUSTODY RECORD
Site #: SERAS-017
Contact Name: T. Ferrell Miller
Contact Phone: [redacted]

No: SERAS-017-04/06/12-0007
Lab: SERAS Laboratory
Lab Phone: 732-321-4212

WO# R204002

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
01	SERAS-017-0020	275-89-06	TPH-DRO	Nutrient Medium	4/3/2012	1	4-oz. amber	formaldehyde	N
02	SERAS-017-0021	275-89-07	TPH-DRO	Nutrient Medium	4/3/2012	1	4-oz. amber	formaldehyde	N
03	SERAS-017-0022	275-89-08	TPH-DRO	Nutrient Medium	4/5/2012	1	4-oz. amber	formaldehyde	N
04	SERAS-017-0023	275-89-09	TPH-DRO	Nutrient Medium	4/5/2012	1	4-oz. amber	formaldehyde	N
05	SERAS-017-0024	275-89-10	TPH-DRO	Nutrient Medium	4/5/2012	1	4-oz. amber	formaldehyde	N
06	SERAS-017-0025	275-89-11	TPH-DRO	Nutrient Medium	4/5/2012	1	4-oz. amber	formaldehyde	N

TTM
4/6/12

Special Instructions: Each sample contains 65 mL water (nutrient medium), 10 mL methylene chloride, 6.5 mL 37% v/v formaldehyde and 200 mg TPH. SERAS-017-0025 contains 15 mL methylene chloride instead of 10 mL.

SAMPLES TRANSFERRED FROM
CHAIN OF CUSTODY #

Items/Reason	Relinquished by	Date	Received by	Date	Time
6/analysis	T. F. Miller	4/6/12	[signature]	4/6/12	12:00

Items/Reason	Relinquished By	Date	Received by	Date	Time

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APR 18 2012

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R204002

ERT/SERAS Laboratory

Client: US EPA/ERT (Edison)
Project: Bioremediation Support for Oil

Project Manager: Vinod Kansal
Project Number: 0-017

Report To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone: (732) 321-6747
Fax: (732) 321-6724

Invoice To:
US EPA/ERT (Edison)
Harry Allen
2890 Woodbridge Avenue
Edison, NJ 08837
Phone :(732) 321-6747
Fax: (732) 321-6724

Date Due: 04/27/12 17:00 (15 day TAT)

Received By: Lawrence Martin
Logged In By: Lawrence Martin

Date Received: 04/06/12 12:22
Date Logged In: 04/06/12 12:22

Samples Received at: 23°C
Custody Seals No
Containers Intact Yes
COC/Labels Agree Yes
Preservation Confir No
Received On Ice No

Analysis	Due	TAT	Expires	Comments
R204002-01 SERAS-017-0020 [Water] Sampled 04/03/12 00:00 Eastern				**275-89-06**
Oil Fingerprint	04/06/12 16:00	8	04/10/12 00:00	
R204002-02 SERAS-017-0021 [Water] Sampled 04/03/12 00:00 Eastern				**275-89-07**
Oil Fingerprint	04/06/12 16:00	8	04/10/12 00:00	
R204002-03 SERAS-017-0022 [Water] Sampled 04/05/12 00:00 Eastern				**275-89-08**
Oil Fingerprint	04/06/12 16:00	8	04/12/12 00:00	
R204002-04 SERAS-017-0023 [Water] Sampled 04/05/12 00:00 Eastern				**275-89-09**
Oil Fingerprint	04/06/12 16:00	8	04/12/12 00:00	
R204002-05 SERAS-017-0024 [Water] Sampled 04/05/12 00:00 Eastern				**275-89-10**
Oil Fingerprint	04/06/12 16:00	8	04/12/12 00:00	
R204002-06 SERAS-017-0025 [Water] Sampled 04/05/12 00:00 Eastern				**275-89-11**
Oil Fingerprint	04/06/12 16:00	8	04/12/12 00:00	

Reviewed By

Date

US EPA ARCHIVE DOCUMENT

Injection Log

US EPA ARCHIVE DOCUMENT

TPH ICAL : DROTPH05161.M

Logbook # SERAS-I-0121

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SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: TPH MDL, DOC in Sol + ICAL
Work Assign.#: 0-011
Analysis/Date: 5/16/11
Method File: DFTPP/ DROTPH05161.M
Inj. Volume: 1ul 1ul
Analyst: T. Sysl

ALS	FILE ID	SAMPLE ID		REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL01860	50ppm DFTPP		RUA0001	5/13/11	19:59	Pass
71	SL01861	1000ppm TPH		RUA00027		15:20	exp 9/16/11 17-67.
72	SL01862	10ppm Oil Surr Check		RUE0038		16:08	4ul of RUE0038
73	SL01863	40ppm MDL Check		RUE0039		16:53	
74	SL01864	20ppm Oil Surr Check		RUE0028			2nd - not Run
75	SL01865	1000ppm LCS Check		RUE0040		17:37	
76	SL01866	20ppm Oil Surr Check		RUE0038		18:27	
	SL	end of std Check 5/13/11					2 Syst
99	SL01867	50ppm DFTPP		RUA0001	5/16/11	14:28	Pass 1400 - 1455
1	SL01868	Blank + IS		RUE0037		15:18	+ 20ul RUE0037
2	SL01869	2000ppm DRO/TPH		RTJ0110		16:02	FROM RTJ0110
3	SL01870	10ppm PAH/SHC	Surr	RTL0035		17:09	Oil(SIM-SE.M) (TIA Rc)
71	SL01871	1000ppm TPH Cal	10ppm	RUE0044		18:09	TPH as DRO
72	SL01872	10,000ppm TPH Cal	100	RUE0042		18:57	
73	SL01873	500ppm TPH Cal	5ppm	RUE0045		19:40	
74	SL01874	5000ppm TPH Cal	50	RUE0043		20:23	
75	SL01875	100ppm TPH Cal	1.0	RUE0046		21:06	
76	SL01876	50ppm TPH Cal	0.5	RUE0047		21:49	TPH as DRO
77	SL01877	5ppm TPH Cal	0.5	RUE0048		22:32	old prep of 50
78	SL01878	1000ppm DROTPH ICV	-	RUE0049		23:14	from 2nd Source
79	SL01879	10K DRO-TPH	100	RTJ0112	5/16/11	23:57	OLD std
80	SL01880	5K DRO-TPH	50	RTJ0113	5/17/11	00:40	OLD std
81	SL01881	Surrogate Check 2nd		RUE0038		1:23	2x of 40ppm (20ppm)
82	SL01882	AQUATPH MDL spike check		RUE0039		2:06	
83	SL01883	LCS spike check 2x		RUE0040		2:49	out of 12 hour check
84	SL01884	5000ppm TPH, Weathered Diesel		RTK0124		3:32	not diagnostic RUN
85	SL01885	10K 10 Wax		RUE0050		4:14	No Corr
86	SL01886	4K 10 WAX Oil		RTK0040		5:14	No Corr
1	SL01887	Blank + IS		RUE0037	5/17/11	6:14	No Hold

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RUA0001	DFTPP	7/2/11	50 ppm		
2	RUE0037 RUE0044	Calibration Check	11/13/11	1000ppm		
3	RUE0037	Internal Standard	11/13/11	500 ppm		
4						
5						
6						

Reviewed By: [signature]
Date Checked: 05-17-11

Reviewed by: D Sysl 5/16/11

US EPA ARCHIVE DOCUMENT

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: TPH in Soil MDL + LOQ Study — Method File: DFTPP/ DRO_TPH05 1611

Work Assign.#: Q-011 — Inj. Volume: 1µl

Analysis/Date: 5/17/11 — Analyst: [signature]

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL0/888	50 ppm DFTPP	RVA0001	5/17/11	16:46	Tune
99	SL0/889	1000 ppm DRO-TPH CCV	RVE0044		17:07	
100	SL0/890	Blank + IS	—		17:53	DCM/Hex + IS Blank
1	SL0/891	1100058-BLK1	Blank		18:46	
2	SL0/892	-BS1	LOD-1		19:29	
3	SL0/893	-BS2	LOD-2		20:12	
4	SL0/894	-BS3	LOD-3		20:54	Soil TPH
5	SL0/895	-BS4	LOD-4		21:37	95 DRO
6	SL0/896	-BS5	LOD-5		22:20	
7	SL0/897	-BS6	LOD-6		23:02	MDL/LOQ
8	SL0/898	-BS7	LOD-7	5/17/11	23:45	Study
9	SL0/899	-BS8	LOQ	5/18/11	00:27	
10	SL0/900	-BS9	DOC-1		1:10	
11	SL0/901	-BSA	DOC-2		1:52	Soil
12	SL0/902	-BSB	DOC-3		2:35	DOC Study
13	SL0/903	1100058-BSC	DOC-4		3:17	
14	SL0/904	EOS INSTRUMENT Blank	Blank	5/18/11	4:00	DAT NEEDIAGONISTIC
	SL	EOS Dyke	5/18/11			
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVA0001	DFTPP	7/2/11	50ppm	—	
2	RVE0044	Calibration Check	10/13/11	1000ppm	—	
3	RVE0637	Internal Standard	11/13/11	500ppm	—	
4						
5						
6						

Reviewed By: [signature] — Date Checked: 05-20-11

US EPA ARCHIVE DOCUMENT

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: Mariner Marsh — Method File: DFTPP/CS110711.M

Work Assign.#: 0-170 — Inj. Volume: 2ul

Analysis/Date: Carbon Tube VOC 1/27/12 — Analyst: Sysl.

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
20	SL02493	BS#2 1/27/12	—	1/27/12	18:41	
21	SL02494	BSD#2 1/27/12	—		19:03	
22	SL02495	Station #3 Front	201006-11		19:56	AA-003-012612-002 F
23	SL02496	Station #3 Back	201006-11		20:17	" " " " B
24	SL02497	Station #1 Front	" " -08		20:37	AA-001-012612-002F
25	SL02498	Station #1 Back	" " -08		20:58	" " " " B
26	SL02499	Station #2 F BS Front	" " -09		21:33	AA-002-012612-002F
27	SL02500	Station #2 B FD Front	" " -09		21:54	" " " " B
28	SL02501	Station #2 F	" " -10		22:15	AA-002-012612-003F
29	SL02502	Station #2 B	" " -10		22:35	" " " B
30	SL02503	Station #4 F	" " 12		22:56	AA-004-012612-002 F
31	SL02504	" #4 B	" " 12		23:17	" " " B
32	SL02505	Field Blank F	" " 13		23:37	FB-012612 F
33	SL02506	" " B	" " 13	1/27/12	23:58	B
34	SL02507	Trip Blank F	" " 14	1/28/12	00:18	TB-012612 F
35	SL02508	Trip Blank B	201006-14	1/28/12	00:39	B
—	SL	End of sequence DF	1/30/12			
—	SL	Front + back Station were switched and confirmed by re-analysing				
	SL	original extract for 2nd extraction - DF			1/31/12	
99	SL02609	50ppm DFTPP	RVK0084	1/30/12	14:19	Pass - 1st time
99	SL02610	50ppm DFTPP	RVK0084	1/30/12	14:37	Pass 1st time
71	SL02611	1st Bucket Skim	01SkmS2		15:18	Enpridge Oil Qual
72	SL02612	25ppm VOC CCV	RVK0089		16:18	Pass
1	SL02613	Station #2 Front E	201006-09		16:38	AA-002-12612-002 F
2	SL02614	" " " B	" "		16:59	AA-002-12612-002 B
73	SL02615	2nd Bucket Skim	01SkmS2	1/30/12	17:20	ENBRIDGE OIL Qual
	SL					
	SL	End sequence DF 1/30/12				
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVK0084	DFTPP	5/5/12	50ppm		
2	RVK0089	Calibration Check VOC	2/25/12	25ppm		
3	RSP0412	Internal Standard	5/8/12	250ppm		
4	RSC0154	BS/BSD Spike	7/23/12	500ppm		Spike 10ul/tube Batch MKBB0760
5	RVA0052	CS_2 Used for extraction	7/26/12	Neat		
6						

Reviewed By: — Date Checked: 1/30/12

Analysis by: J. Sysl 2/3/12

* re-do

US EPA ARCHIVE DOCUMENT

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: VOC R+D and Enhanced Oil Fingerprint — Method File: DFTPP/ 0.1Scan S2

Work Assign.#: -011 and 0-037 — Inj. Volume: 1µl

Analysis/Date: 1/30/12 — Analyst: SJG

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
3	SL02616	Station #3 E		1/30/12	19:17	
3	SL02617	Station #3 E re-inj			19:37	
4	SL02618	CS_2 Blank			19:58	+ IS
5	SL02619	BS in Solvent			20:18	
4	SL02620	CS_2 Blank			20:38	
6	SL02621	BS 1/30/12			20:58	
7	SL02622	BSD 1/30/12			21:19	
4	SL02623	CS_2 Blank			21:39	
4	SL02624	CS_2 Blank			21:59	
8	SL02625	BSD3 1/30/12			22:19	Expired Lot Nov 2010
4	SL02626	CS_2 Blank			22:39	
74	SL02627	3rd Skin - From 5000 ml		1/30/12	23:01	No IS
75	SL02628	3rd Skin - Combo + Core to 20ml		1/31/12	0:02	5000 Fingerprint + IS
76	SL02629	Blank + Tank		1/31/12	1:02	
	SL	end of R+D sequence		SJG	1/31/12	
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1		DFTPP				
2		Calibration Check				
3		Internal Standard				
4						
5						
6						

Reviewed By: _____ Date Checked: _____

Analyzed by: SJG 2/1/20

Mariner's Marsh : 1816 Enbridge Oil : 1803 Logbook # SERAS-I-0121

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DRQTPH051611.M

Project Name: Mariner Marsh + Enbridge Oil Method File: DFTPP CS110711.M
Work Assign.#: 0-170 0-017 Inj. Volume: 2ul
Analysis/Date: Carbon Gwo VOC TPH Analyst: Syd.

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
21	SL02655	STATION #1 F	20)006-22	2/2/12	00:59	AA-001-013112-004
22	SL 56	#1 B	-22		1:19	-001- -004
23	SL 57	STATION #2 F	-23		1:39	-002- -005
24	SL 58	#2 B	-23		1:59	-002- -005
25	SL 59	STATION #3 F	-24		2:20	-003- -004
26	SL 60	#3 B	-24		2:40	-003- -004
27	SL 61	STATION #4 F	-25		3:00	-004- -004
28	SL 62	#4 B	-25		3:20	-004- -004
29	SL 63	STATION #5 F	-26		3:40	-005 -004
30	SL 64	#5 B	-26		4:00	AA-005-013112-004
31	SL 65	FB-013112 F	-27		4:20	Field Blank
32	SL 66	" B	-27		4:40	
33	SL 67	TB-013112 F	-28		5:00	Trip Blank
34	SL02668	TB-013112 B	20)006-28	2/2/12	5:21	
	SL	end of Mariner Marsh seq			2/02/12	
99	SL02669	50 ppm DFTPP	RVK0164	2/2/12	14:13	Pass
99	SL02670	50 ppm DFTPP	RVK1084		15:44	Pass 1402-1390
71	SL02671	1000 ppm TPH CCV	RVB0003		16:23	Pass - ok
80	SL02672	Soil Blank	120001201 Blk		17:18	
81	SL02673	SERAS-017-0001 5x d	R20201-01		18:43	30g=1ml, 5x Screen
82	SL02674	SERAS-017-0001	" "		19:53	
80	SL02675	Blank-all-IN-element	-Blank		21:53	Dot Washes
82	SL02676	SERAS-017-001 LINEAR	R20201-01	2/2/12	21:54	LINEAR SCAN Screen
	SL	End of Enbridge Oil sequence			2/3/12	
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVK0184	DFTPP	5/5/12	50 ppm		
2	RVK0039	Calibration Check	2/25/12	50 ppm		
3	RSF0112	Internal Standard	5/8/12	2500 ppm		Spike 20ml=1.6ml/10=.5
4	RSC0154	BS/BSD Spike	7/20/12	5000 ppm		Spike 10ml=tube
5	RVA0052	BS Extraction Solv	7/26/12			
6						

Reviewed By: Date Checked: 2/3/12

Analyses by: J Syd 2/3/12

US EPA ARCHIVE DOCUMENT

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: Enbridge Oil — Method File: DFTPP/ DFTPP05161f.m
Work Assign.#: WA-017 — Inj. Volume: 1ul
Analysis/Date: TPH 2/15/12 — Analyst: [illegible]

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02826	50 ppm DFTPP	RUK00084	2/15/12	12:21	Scan 1598-1358
91	SL02827	1000 ppm TPH CCV	RVB0023		12:42	Run
82	SL02828	Soil Blank			13:43	120824-Blk1
83	SL02829	SERAS-017-0002	R20208-01		14:27	
84	SL02830	Toluene Blank	RTJ0030		15:47	In CHC-DCM
85	SL02831	DCM in Toluene 0.1g			16:07	1ul DCM-1.0ml Tol
86	SL02832	RVB0026 in Toluene			16:23	0.1g → 5.0ml Toluene
87	SL02833	Toluene Blank			16:55	
88	SL02834	BEO 20 in Toluene			17:16	re-run 0.1g → 1.0ml
89	SL02835	Tol. Blank			17:42	
90	SL02836	BEO RV 0.1g → 8ml			18:01	Rig Rate Var
90	SL02837	BEO RV 2.5K			18:40	
82	SL02838	Soil Blank		2/15/12	19:41	
	SL	end of 2/15/12	Sequence			91
99	SL02839	50 ppm DFTPP	RVB0025	2/16/12	12:21	
1	SL02840	Toluene Blank			12:39	
1	SL02841	Toluene Blank			12:57	
1	SL02842	Tol Blank			15:34	
2	SL02843	RVB0026 DCM - 4:20			16:50	0.05g → 2ml Tol
1	SL02844	Toluene Blank			17:26	In DCM
3	SL02845	Toluene Blank			18:16	
4	SL02846	BEO 66.7 x4		2/16/12	18:24	
99	SL02847	50 ppm DFTPP	RVB0025	2/17/12	21:45	
75	SL02848	10K Arabian Crude		2/17/12	22:07	In Tol
99	SL02849	50 ppm DFTPP	RVB0025	2/22/12	10:19	
99	SL02850	50 ppm DFTPP	RVB0025		10:37	
98	SL02851	10K BEO Slm	275-5505		10:59	
98	SL02852	10K BEO Slm	"		19:33	Library Scan
99	SL02853	50 ppm DFTPP	RVB0025	2/22/12	20:29	

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RUK00084	DFTPP	5/5/12	50ppm		
2	RVB0023	Calibration Check	8/2/12	1000 ppm		
3	RUK00080	Internal Standard	5/11/12	500ppm		
4						
5						
6						

Reviewed By: J Dyal Scan > ASA — Date Checked: 2/24/12

Logbook # SERAS-I-0121

ENBRIDGE Oil: FINGERPRINTS + TPH

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SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DRDTPH051614.M

Project Name: ENBRIDGE Oil | Method File: DFTPP/ OILSIMS2

Work Assign.#: 0-017 | Inj. Volume: 1µl

Analysis/Date: 2/24/12 | Analyst: SyS/s

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02856	50 ppm DFTPP	RVB0025	2/24/12	11:35	Pass 1397-1385
71	SL02857	1K TPH CCV	RVB0003		11:57	Pass
72	SL02858	10 ppm SHC/PAH Res/Mass	RTL0035		15:46	Res/Mass Disc Test
73	SL02859	10K CG-117 Reference	RUL0003		17:36	Fingerprint Reference
1	SL02860	Method · WD BLANK	RVB0060		18:58	
2	SL02861	SERAS-017-0003	275-55-05		21:18	0.109g →10ml
3	SL02862	SERAS-017-0004	275-56-24	2/24/12	22:20	0.193g →10ml
4	SL02863	SERAS-017-0003 dup	275-55-05	2/25/12	00:29	0.109g →10ml dup
5	SL02864	RLK0224/2 ml	WD Blank	2/25/12	1:32	10ml RUC0022 + Surr
6	SL02865	Method Blank 1.0ml recinj	RVB0060	2/27/12	9:42	Re-inj of Solvent Blk
7	SL02866	SERAS-017-0003			X	D-5738 "bracket dup"
8	SL02867	10K CG-117 Ref/mass	RUC0003		X	D-5739 "bracket bracket"
9	SL02868	BLK022412MD			X	EOS Blk
10	SL02869	SERAS-017-0004A 1.5xd	275-56-24	2/24/12	23:26	re-extract & DCM/MeOH Ace
11	SL02870	SERAS-017-0004A NK	275-56-24	2/27/12	9:42	" " "
	~~SL~~	Sequence halted at 1.32AM		2/25/12		syringe error
	SL X - not	injected				
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/14/12	50ppm		
2	RVB0003	Calibration Check	8/12/12	1000ppm		TPH as DRO
3	RVB0058	Internal Standard + Oil	5/14/12	500ppm		Spike 20µl →1.0ml
4	RUL0003	CG-117 Reference Fingerprint	qual 5/24/12	10,000ppm		FINGERPRINT RF.
5	RTL0035	10ppm SHC/PAH Std	qual	10ppm		MASS Discrimination Chk
6						

Reviewed By: [signature] Date Checked: 03-06-12

ALS # 10, 11 were inserted in sequence after performing re-extraction of sample The FILE ID's are not in sequence with sequential injection/Time stamp Because they had to be run before other

Enbridge Oil + Flemings-burg

Logbook # SERAS-I-0121

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SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DROTPH05(6).M

Project Name: Enbridge Oil | Method File: DFTPP/ OILS/MS2

Work Assign.#: 6-017 | Inj. Volume: 1ul

Analysis/Date: TPH + Fingerprint 3/27/12 | Analyst: Sglc

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02870	50ppm DFTPP	RVB0025	2/27/12	15:07	ok 1396-1382
71	SL02871	1000ppm TPH CCV	RVB0003		15:31	
72	SL02872	10ppm SHC/PAH Re Mix	RTL0035		16:32	MAH DRC ok
73	SL02873	10K CG-117 ref Oil	RVL0003	2/27/12	17:34	
~~1~~	~~SL02874~~	~~BLK022712WD~~	re-injected file	X	X	WATER DIL Blank
1	SL02875	BLK022712WD	Blank		X	ok
1	SL02876	BLK022712WD	Blank		18:44	
2	SL02877	SERAS-017-0003			19:33	
3	SL02876	SERAS-017-0003 dup			20:35	
4	SL02877	SERAS-017-0004			21:37	
5	SL02878	SERAS-017-0004			22:38	SKIM from Bucket
6	SL02879	SERAS-017-0003 dup		2/27/12	23:40	Early dup
7	SL02880	10K CG-117 ref oil	RVL0003	2/28/12	00:41	D-5739 ref
2	SL02881	SERAS-017-0003 dup	dup of	2/28/12	1:42	- Qual
—	SL	only by Dave	3/1/12			
99	SL02882	50ppm DFTPP	3/1/12	RVB0025	12:28	Pass 1385-1283
71	SL02883	10ppm SHC/PAH Re Mix	RTL0035	3/1/12	12:50	
72	SL02884	10K CG-117 ref oil	RVL0003		13:51	
1	SL02885	1000ppm TPH CCV/LCS	RVB0003		14:56	
2	SL02886	RL 030112	Blank		15:41	0.1g → 10ml
3	SL02887	FOR-TRENCH	R202018-01		17:33	0.055g → 5.0ml
4	SL02888	FOR-TRENCH dup	R202018-01 dup		18:34	0.105g → 5.0ml
5	SL02889	FOR-CREEK			19:36	0.148g → 5.0ml
6	SL02890	SERAS-017-0005			20:37	0.104g → 10ml
7	SL02891	SERAS-017-0005 dup			21:37	0.115g → 10ml
8	SL02892	SERAS-017-0006			22:38	0.102g → 10ml
9	SL02893	10K Amt Oil		3/1/12	23:39	Used Motor Oil
10	SL02894	10K CG-117 Reference	RVL0003	3/2/12	00:40	
11	SL02895	CHC REO	CHC Oil	3/2/12	1:40	0.95g → 10ml

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/14/12	50 ppm		
2	RVB0003	Calibration Check	8/12/12	1000 ppm		TPH
3	RVB0058	Internal Standard	5/14/12	500 ppm		Spk 20µL → 5ml
4	RVL0003	CG-117 Ref. Fuel	5/24/12	10 Wt%		D-5739 ref.
5	RTL0035	SHC/PAH Mix	Qual	1000 ppm		Res/MAH DRC
6						

Reviewed By: Jessica Odencrantz | Date Checked: 03-06-12

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DROTAN 05/6H.M

Project Name: Flemingsburg Oil + Enbridge Oil + Cross Creek Method File: DFTPP/ 012SIM52.M

Work Assign.#: 010017 / 017 / 020017 Inj. Volume: 1uL

Analysis/Date: 3/2, 3/5 → 3/7/12 Analyst: JdL

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
12	SL02896	CHC BEO 1390	CHC	3/2/12	2:41	0.995 > 10uL
2	SL02897	EOS Blank		3/2/12	3:32	*
	SL	End of Sequence	QD 3/2/12			
	SL	* Filament blew during this analysis				
99	SL02898	50 ppm DFTPP	RVB0026	3/2/12	4:56	OK check opt. ll
99	SL02899	↓ ↓	↓	3/5/12	12:02	} all good - dayrA
99	SL02900			3/5/12	12:24	
99	SL02901	50 ppm DFTPP	RVB0026	3/6/12	17:29	
72	SL02902	CHC-BEO 5900 ppm		3/7/12	0:55	Baked - 125°C
73	SL02903	ENO - 0004 11,500 ppm		3/7/12	3:58	" "
74	SL02904	ENO - 0003 9900 ppm		3/7/12	4:56	" "
	SL	End of Sequence	for diagnostic test			JdL 3/7/12
	SL					
99	SL02905	50 ppm DFTPP	RVB0026 QD 3/7/12		17:20	Tass 1384 - 1374
71	SL02906	10 ppm Res/Mar Dec Mix	RLB035		15:28	D-5739 QC
72	SL02907	1.0K TPH CCV for Surrogates	RVB003		19:41	
73	SL02908	10K CC-117 Enbridge Oil	RVL0003		21:52	D-5739 QC
1	SL02909	CWD-BLANK 030712	P-Blk	3/7/12	21:59	Water dilution Blank
2	SL02910	ECO-CREEK EAST	R203002-01	3/8/12	2:04	
3	SL02911	ECO-CREEK EAST dup	R203002-01dup		3:06	
4	SL02912	ECO-Well 108018	R203002-02		4:08	
5	SL02913	ECO-Well 102216	R203002-03		5:11	
6	SL02914	ECO-CREEK EAST dup EOS	R203002-01dup		6:13	
7	SL02915	10K CC-117 Ref EOS	RVL0003		7:15	Did not blow in 12hr
1	SL02916	CWD-BLANK - EOS	-Blk	3/8/12	8:17	
	SL					
	SL					
	SL	end of Seq				JdL 3/8/12
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/14/12	50 ppm		
2	RVB0003	Calibration Check	8/12/12	100 ppm		surrogate
3	RVB0058	Internal Standard	5/14/12	500 ppm		
4	RVL0003	CC-117 - BJ Frac	5/24/12	10,000 ppm		} D-5739 QC
5	RLB035	10 ppm STD/Blk Res/Mix	-None	10 ppm		
6						

Reviewed By: James O'Connell Date Checked: 3-19-12

Analyzed by: JdL

US EPA ARCHIVE DOCUMENT

Logbook # SERAS-I-0121

Test extraction/solvent to dilute Enbridge oil products

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SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: Column Maintenance after CHC | Method File: DFTPP/ CHC TPH032012

Work Assign.#: 017 etc | Inj. Volume: 1ul

Analysis/Date: 3/21/12 | Analyst: Lyle

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
	SL02941	50ppm DFTPP	RVB0025	3/21/12	16:04	
	SL02942	EXO120RB END 4x			17:11	Test of Solution/Extraction
	SL 43	End of 44,444			18:01	of Enbridge Oil
	SL 44	5500ppm RVC0056			19:38	
	SL 45	SERAS-185-0075 Linear Scan			20:35	CHC extract : Linear Scan
	SL 46	5000ppm #2 Diesel			21:39	RVE0043
	SL 47				22:39	
	SL 48			3/21/12	23:40	
	SL 49			3/22/12	00:38	
	SL 50	5000ppm #2 Diesel			1:40	RVE0043
	SL 51	DCM/Ace/Meoh/Hex Blank			2:41	Solvent Mix for END
	SL 52	"			3:41	"
	SL 53	Palo Seco 51684 0.1 g			4:42	5000 ppm
	SL 54	Palo Seco 51684 0.1 5.0g			5:42	
	SL02955	Blank		3/22/12	6:43	
	SL	end of seq vers				
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1		DFTPP				
2		Calibration Check				
3		Internal Standard				
4						
5						
6						

Reviewed By: Jack Lyle Column maintenance 3/26/12 | Date Checked: 3/22/12

US EPA ARCHIVE DOCUMENT

TPH ICAL for ENBRIDGE Oil Samples
- uses site oil

Logbook # SERAS-I-0121

TPH ICAL: ENOTPH032212.M Page 90

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: ENBRIDGE Oil ; TPH ICAL + SAMPLES — Method File: DFTPP/ ENOTPH032212

Work Assign.#: 0-017 — Inj. Volume: 1µL

Analysis/Date: TPH in Bioremediation Study — Analyst: [illegible]

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02956	50 ppm DFTPP	RVB0025	3/22/12	14:51	Pass 1385-1376
71	SL02957	Method Blank			15:30	Solvent Blank + Surr
72	SL02958	5000 ppm ENO TPH Std	RVC0059		16:51	
73	SL02959	1000 ppm ENO TPH Std	RVC0062		17:52	
74	SL02960	10,000 ppm ENO TPH Std	RVC0060		18:52	
75	SL02961	50,000 ppm ENO TPH Std	RVC0058		19:52	
76	SL02962	25,000 ppm ENO TPH Std	RVC0061		20:51	
77	SL02963	0.5 ppm ENO TPH Std	RVC0063		21:51	
78	SL02964	50000 ppm End Source CCV	RVC0064		22:51	
99	SL02965	50 ppm DFTPP	RVB0025	3/22/12	23:47	Pk ht Evaporation
72	SL02966	5000 ppm ENO CCC TPH	RVC0059	3/23/12	00:08	Pass Col. Check TPH
1	SL02967	BLANK	120039-Blk1		1:08	1mL FV
2	SL02968	LCS	120039-BS1		2:07	1mL FV
3	SL02969	SERAS-017-0007 4x	R203011-01		3:08	5mL FV then 4X
4	SL02970	-0008	-02		4:08	
5	SL02971	-0009	-03		5:08	
6	SL02972	-0010	-04		6:09	
7	SL02973	-0011	-05		7:09	
8	SL02974	-0012	-06		8:09	
9	SL02975	-0013	R203011-07		9:09	
10	SL02976	-0013 MS	120039-MS1		10:09	
11	SL02977	SERAS-017-0013 MSD 4x	120039-MSD1		11:09	5mL FV then 4X
12	SL02978	COS Blank	Blank	3/23/12	12:08	
	SL	resol'd 3/24/12 Sys				
99	SL02979	50 ppm DFTPP	RVB0025	3/23/12	17:54	Pass 1387-1379
71	SL02980	5000 ppm ENO TPH CCC	RVC0059		18:21	Pass
1	SL02981	Water Blank	120040-Blk1	120040-MB1	19:38	1mL FV
2	SL02982	SERAS-017-0014 4x	R203015-01		20:37	5mL FV then 4X
3	SL02983	SERAS-017-0015 4x	R203015-02	3/23/12	21:58	5mL FV then 4X

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/14/12	50 ppm		
2	RVC0059	Calibration Check TPH	9/15/12	5000 ppm		Site Specific TPH
3	RVC0035	Internal Standard Oil ID	9/15/12	50 ppm		Spike 20uL/1mL
4						
5						
6						

Reviewed By: [signature] — Date Checked: 3/26/12

Analyzed by: JSyl 3/24/12

US EPA ARCHIVE DOCUMENT

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: Enbridge Oil — Method File: DFTPP/ EN1TP14032212

Work Assign.#: 0-019 — Inj. Volume: 1uL

Analysis/Date: TPH - Water/ — Analyst: SJL

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
4	SL02984	SERAS-017-0016 4x	R283015-03	3/23/12	23:00	5mL FV the 4x
5	SL02985	-0017	-04	3/24/12	00:02	
6	SL02986	-0018	-05		1:03	
7	SL02987	-0019	R20305-06		2:05	
8	SL02988	LCS · 120040 BS1			3:07	1mL FV
9	SL02989	LCSD · 120040 BSD1			4:09	1mL FV
10	SL02990	SERAS-017-0007 5x	R203011-01		5:10	5mL FV the 3x
11	SL02991	120039-MS 5x	MS		6:13	5mL FV the 5x
1	SL02992	120040 - Blk1	EQ Blank		7:15	
12	SL02993	Review Program			8:17	Amt Oil 5000ppm
1	SL02994	120040 Blk1			9:18	
99	SL02995	DFTPP 50 ppb 1mL	RVB0025	3/24/12	14:32	DFTPP
	SL	end of 3/23/12 seq				
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/11/12	50ppm		
2	RVC1059	Calibration Check	9/8/12	3000ppm		
3	RVC0036	Internal Standard	9/15/12	5000ppm		
4						
5						
6						

Reviewed By: Analyses By - SJL 3/24/12

Date Checked: 3/26/12

US EPA ARCHIVE DOCUMENT

Logbook # SERAS-I-0121

Day 28 Treatability Study Enbridge

Page 94

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: Enbridge O.I. Method File: DFTPP/ENOTPH032212

Work Assign.#: 0-819 Inj. Volume: 1ul

Analysis/Date: 4/18/12 Analyst: Sydl

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL03046	50ppm DFTPP	RVB0025	4/9/12		
71	SL03047	1000 ppm DRO TPH				DIAGNOSTIC
72	SL03048	1000 ppm DRO TPH				for Pass of
73	SL03049	2000 ppm DRO TPH		4/9/12		TPH SOP
-	SL	EJL - 4/9/12				
99	SL03050	50 ppm DFTPP	RVB0025	4/18/12	15:33	Pass 1585 - 1374
71	SL03051	500ppm END CCC	RVC0059		15:53	OK - Just Lower EMV
71	SL03052	5000ppm END CCC	RVC0059		17:18	Pass all good
1	SL03053	Sol Blank	120055-BLK		18:19	Ind FV
2	SL03054	Sol LCS	" BSD		19:20	"
3	SL03055	Sol LCS dup	120055-BSD1		20:20	Ind FV
4	SL03056	4x SERAS-017-0020			21:21	375-89-06 Sol FV, the 4x
5	SL03057	4x -017-0021			22:22	-89-07
6	SL03058	4x -017-0022		4/18/12	23:23	-89-08
7	SL03059	4x -017-0023		4/19/12	00:23	-89-09
8	SL03060	4x -017-0024			1:24	-89-10
9	SL03061	4x SERAS-017-0025			2:25	375-89-11 Sol FV, the 4x
1	SL03062	SOS Blank		4/19/12	3:26	DIAGNOSTIC
-	SL	SOS EJL 4/19/12				
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
	SL					
.	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/1/12	50ppm		
2	RVC0059	Calibration Check	9/15/12	500ppm		
3	RVC0035	Internal Standard	9/15/12	500ppm		
4						
5						
6						

Reviewed By: Vinod Kansal Date Checked: 6/5/12

Analyzed by: J. Sydl 4/18/12

US EPA ARCHIVE DOCUMENT

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DROTPH42712

Project Name: DRO-TPH DOC STUDIES + ICAL — Method File: DFTPP/~~DROTPH042512~~

Work Assign.#: 0-011 — Inj. Volume: 1µl

Analysis/Date: 4/25/12 4/27/12 — Analyst: Sysl

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
~~99~~	~~SL03063~~	~~50 ppm DFTPP~~	~~RVB0025~~	~~4/25/12~~		~~D-Sys6 - Aldrin~~
99	SL03063	50 ppm DFTPP	RVB0025	4/26/12	17:44	Pass 1354-1357
71	SL03064	1.0K DRO CCV	RVB0003	↓	18:25	0/D Cal Std
72	SL03065	DRO MS Spike 10x	RVD0069	↓	19:22	
73	SL03066	DRO LCS Spike 2.0x	RVD0070	4/26/12	20:08	
	SL	End of 4/26/12 seq wmk - Sys6				
99	SL03067	50 ppm DFTPP	RVB0025	4/27/12	14:52	
99	SL03068	50 ppm DFTPP	RVB0025	4/27/12	16:26	1356-1372 Pass
71	SL03069	DCM/Hex Blank +IS	↓		17:09	Surr ↓
72	SL03070	1000 ppm DROTPH Cal	RVD0074		18:14	10ppm
73	SL03071	10K DRO TPH Cal	RVD0072		19:00	100 ppm
74	SL03072	5.0K DRO TPHCal	RVD0073		19:47	50 ppm
75	SL03073	500 ppb DRO TPH	RVD0075		20:34	5 ppm
76	SL03074	100 ppb DRO TPH	RVD0076		21:21	1 ppm
77	SL03075	50 ppb DRO TPH	RVD0077		22:07	0.5 ppm
78	SL03076	DRO ICV #1 1000 ppm	RVD0078	↓	22:52	
79	SL03077	DRO ICV #2 1000 ppm	RVD0018	4/27/12	23:36	RVD0018 dilute 2x
1	SL 78	Water DOC-1 NB		4/28/12	00:21	1200047-BS1
2	SL 79	DOC-2			1:06 x	-BS2
3	SL 80	DOC-3			1:53 x	-BS3
4	SL 81	DOC-4			2:38 x	-BS4
5	SL03082	Water Blank NB		4/28/12	3:23 x	1200047-BLK1
~~99~~	~~SL03083~~	~~1000 ppm DROTPH CCV~~	~~RVD0074~~	~~Q8 4/27/12~~		
99	SL03083	50 ppm DFTPP	RVB0025	5/4/12	18:14	Pass
71	SL03084	1000 ppm DROTPH CCV	RVD0074		18:35	Pass
6	SL03085	Soil Blank NB			19:20	1200048-BLK1
7	SL03086	" DOC-1			20:02	-BS1
8	SL03087	" DOC-2			20:49	-BS2
9	SL03088	Soil DOC-3 NB		5/4/12	21:26	1200048-BS3

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/14/12	50 ppm		
2	RVD0074	Calibration Check	10/26/12	1000 ppm		
3	RVC0035	Internal Standard	9/15/12	50 ppm		
4						
5						
6						

Reviewed By: [signature] Date Checked: 05-11-12

US EPA ARCHIVE DOCUMENT

Extraction Logs

- Sediments

+

Day 28 Treat

STUDY

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

PREPARATION BENCH SHEET

Batch No. 1200012

Printed: 2/2/2012 2:54:01PM

Lab Number	Sample #	Prepared	Initial (g)	Final (mL)	Surrogate	µl Surrogate	Spike ID	Source ID	µl Spike	Extraction Solvent Vol.	Extraction Comments
1200012-BLK1	Blank	02/02/12 11:39	30	1	RUK0078	500					
R202001-01	SERAS-017-0001	02/02/12 11:39	30	1	RUK0078	500					

Project Name: Oil Spill Response Support
Project No.: SERAS-017

Matrix: Soil
Analysis: Oil Fingerprint

Prepared By: SS
Prepared using: EPA 3545 MS by Soxtherm

0129

Spiking Witnessed By — Date

Preparation Reviewed By — Date 2/2/12

Extracts Received By — Date 2/02/12

SERAS-017-DTM-011413_3

PREPARATION BENCH SHEET

Batch No. 1200024

Printed: 2/14/2012 12:05:53PM

Lab Number	Sample #	Prepared	Initial (g)	Final (mL)	Surrogate	µl Surrogate	Spike ID	Source ID	µl Spike	Extraction Solvent Vol.	Extraction Comments
1200024-BLK1	Blank	02/14/12 12:04	30	1	RVB0021	500					
R202008-01	SERAS-017-0002	02/14/12 12:04	30	1	RVB0021	500					

Project Name: Oil Spill Response Support
Project No.: SERAS-017

Matrix: Soil
Analysis: Oil Fingerprint

Prepared By: SS
Prepared using: EPA 3545 MS by Soxtherm

0130

Spiking Witnessed By Date

Preparation Reviewed By 2/14/12 Date

Extracts Received By 2/14/12 15:25 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

SERAS

PREPARATION BATCH SHEET

ENO Day 6

Batch No. 1200039

Printed: 3/21/2012 3:04:00PM

Lab Number	Sample #	Prepared	Initial (mL)	Final (mL)	Surrogate	µl Surrogate	Spike ID	Source ID	µl Spike	Extraction Solvent Vol.	Extraction Comments
1200039-BLK1	Blank	03/21/12 15:01	200	1	RVB0021	500					
1200039-BS1	LCS	03/21/12 15:01	200	1	RVB0021	500	RVC0053		500		
1200039-MS1	Matrix Spike	03/21/12 15:01	200	5	RVB0021	500	RVC0053	R203011-07	500		
1200039-MSD1	Matrix Spike Dup	03/21/12 15:01	200	1	RVB0021	500	RVC0053	R203011-07	500		
R203011-01	SERAS-017-0007	03/21/12 15:01	200	1	RVB0021	500					
R203011-02	SERAS-017-0008	03/21/12 15:01	200	1	RVB0021	500					
R203011-03	SERAS-017-0009	03/21/12 15:01	200	1	RVB0021	500					
R203011-04	SERAS-017-0010	03/21/12 15:01	200	1	RVB0021	500					
R203011-05	SERAS-017-0011	03/21/12 15:01	200	1	RVB0021	500					
R203011-06	SERAS-017-0012	03/21/12 15:01	200	1	RVB0021	500					
R203011-07	SERAS-017-0013	03/21/12 15:01	200	1	RVB0021	500					

Project Name: Bioremediation Support for Oil
Project No.: 0-017

Matrix: Water
Analysis: Oil Fingerprint

Prepared By: SS
Prepared using: Method 3510C by Separatory Funnel

dilute it all



0131

Spiking Witnessed By Date

Preparation Reviewed By Date

Extracts Received By 3/21/12 Date

SERAS-017-DTM-011413_3

PREPARATION BENCH SHEET

Day 14

Batch No. 1200040

Printed: 3/23/2012 3:35:32PM

Lab Number	Sample #	Prepared	Initial (mL)	Final (mL)	Surrogate	µl Surrogate	Spike ID	Source ID	µl Spike	Extraction Solvent Vol.	Extraction Comments
1200040-BLK1	Blank	03/23/12 09:00	200	1	RVB0021	500					
1200040-BS1	LCS	03/23/12 09:00	200	1	RVB0021	500	RVC0053		500		
1200040-BSD1	LCS Dup	03/23/12 09:00	200	1	RVB0021	500	RVC0053		500		
R203015-01	SERAS-017-0014	03/23/12 09:00	200	5	RVB0021	500					
R203015-02	SERAS-017-0015	03/23/12 09:00	200	5	RVB0021	500					
R203015-03	SERAS-017-0016	03/23/12 09:00	200	5	RVB0021	500					
R203015-04	SERAS-017-0017	03/23/12 09:00	200	5	RVB0021	500					
R203015-05	SERAS-017-0018	03/23/12 09:00	200	5	RVB0021	500					
R203015-06	SERAS-017-0019	03/23/12 09:00	200	5	RVB0021	500					

Project Name: Bioremediation Support for Oil
Project No.: 0-017

Matrix: Water
Analysis: Oil Fingerprint

Prepared By: SS
Prepared using: Method 3510C by Separatory Funnel

MAR 22 2012

0132

Spiking Witnessed By — Date

Preparation Reviewed By — Date 3/23/12

Extracts Received By — Date 3/23/12

US EPA ARCHIVE DOCUMENT

SERAS

PREPARATION BATCH SHEET

Day 2

Batch No. 1200055

Printed: 4/18/2012 2:52:20PM

Lab Number	Sample #	Prepared	Initial (mL)	Final (mL)	Surrogate	µl Surrogate	Spike ID	Source ID	µl Spike	Extraction Solvent Vol.	Extraction Comments
1200055-BLK1	Blank	04/18/12 14:49	200	1	RVD0036	500					
1200055-BS1	LCS	04/18/12 14:49	200	1	RVD0036	500	RVC0053		500		
1200055-BSD1	LCS Dup	04/18/12 14:49	200	1	RVD0036	500	RVC0053		500		
R204002-01	SERAS-017-0020	04/18/12 14:48	200	5	RVD0036	500					
R204002-02	SERAS-017-0021	04/18/12 14:48	200	5	RVD0036	500					
R204002-03	SERAS-017-0022	04/18/12 14:48	200	5	RVD0036	500					
R204002-04	SERAS-017-0023	04/18/12 14:48	200	5	RVD0036	500					
R204002-05	SERAS-017-0024	04/18/12 14:48	200	5	RVD0036	500					
R204002-06	SERAS-017-0025	04/18/12 14:48	200	5	RVD0036	500					

Project Name: Bioremediation Support for Oil
Project No.: 0-017

Matrix: Water
Analysis: Oil Fingerprint

Prepared By: SS
Prepared using: Method 3510C by Separatory Funnel

RECEIVED
APR 18 2012

0133

Spiking Witnessed By — Date

Preparation Reviewed By — Date 4/18/12

Extracts Received By — Date 4/12/12

3/6/12
Tuesday • Going to simulate autoclave of Bulk Extracted CMC oil to determine if Dry gas and charge
• USING RVB0026 - The oil that should contain DCM
• could save time/a meal BEO
• will put in GC oven at 120°C

Vial wt = 4.634g
Vial + oil = 5.234g
5.192
Loss 7.3%

before bake .608g
-.044

Autoclaved BEO - 0.1185g → 2-3ml DCM/Ltr
~62,000 ppm → dilute 1:10x before inject

004 Flask → Lid 17.176
18.446 = 1.27g

Bake in GC Oven at 120 - see what gets

ADD 10ml DCM/Ltr

2nd one — TO test bake

004 Vial = 4.883
= 5.005g — no lid - Put in 2hrs at 125° 4.975 = 0.092g
= 4.966

Weight 003 will check its AB in 2hrs 2.0ml

VIAL = 12.407g
12.614g ok GC 12.585

= 0.178g ~ Equivalent to 7.4

double by 4's

Continued on Page

Read and Understood By

Signed 3/6/12 Date

Signed 3/12/12 Date

US EPA ARCHIVE DOCUMENT

3/7/2012

Wed • Preparing SERAS-017-0004 product for Fenell Miller: he will use for bioremediation study.

• Sample-0004 consists of an oil soaked mud/solid. I tried to separate oil from oil/mud by just centrifuge - but the product still is very viscous and the solid to work with

• Continuing following by heating entire sample in water bath at 80°C for prolonged as only oil and layer / water (Lost to oil) and brake / lost.

• to remove water - I'll check whether Baker I can dry top layer and put in Real Vac - the bake at 125°C for 4 hour in 60/45 - I'll that remove water + leave heavy oil

• calibrating scale - in logbook SERAS-L-0157 - page 51

↳ 20mL Vial weight = 13.920g
- Decided to use 30mL Vial: 20.954
30.234 - Vial
28.916
= 9.25g wet
= 7.972 cooked at 125°C
in oven @ 11:30

DOW

cooled for 1 HY.

Fenell got 6.514g

3/7/12

Continued on Page

Read and Understood By

Signed Date Signed Date

3/7/12 3/12/12

SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

3/7/12 • Received 3x samples on 3/2/12 ✓ by ✓ by overnight
by Courier to WASTE Dilution with Surrogate
- Scale was calibrated earlier ✓ in Envirobydrock

Sample	Wt	Fin Vol	Description Surrogate = RVK0077
WD_Blank080712	0.1g	10mL	- Blank - 10mL solvent + 100µL Surrogate
CCO CREEK EAST (R20218-03A)	0.155g	→3mL	Samples RICK with thick Sulfur Some of sample seen on inside of ziplock bag
Note → The dilution ok - try for a 5000 → 10,000 ppm product/travel			
CCO CREEK EAST dup (R20218-03B)	0.17g	→9.0mL	- added 30µL RVK0077, dilute 10x for analysis - Sulfur + tarry remains
CCO-WELL-108018 (R20218-06)	*	→10mL	added 10µL surrogate, 06 was a H_2S sulfur smelling water with fine gray silt. There was some product visible on lid - I couldn't weigh - had to gather as much as possible on side of pipette tip and wash pipette with ~1.0mL DCM/hexane - By color ~ 1st wash ~ 2x dilute
CCO-WELL-102266 (R20218-05)			- will run this No weight
CCO-WELL-102266 (R20218-05)	0.245g	→3.0mL	- has gravel - stinky like the East creek

3.7.12

Continued on Page

Read and Understood By

Signed — Date 3/7/12 — Signed — Date 3/12/12

US EPA ARCHIVE DOCUMENT

Extraction / Waste Dilution
Prep

+

Gravimetric Procedure

US EPA ARCHIVE DOCUMENT

2/24/12

FRIDAY → Need to make WASTE dilution of 2 greatest samples for TPH + Oil Fingerprinting

2 Samples:

COC	Sample #	LOCATION
SERAS-017-02/21/12-0003	SERAS-017-0003	275-55-05
SERAS-017-02/23/12-0004	SERAS-017-0004	275-50-24

↳ Extract 1 Blank - Waste Dilution
1 Duplicate - Sample SERAS-017-0003

↳ Sample prep: weigh 0.100 g product, add 100µl of 2000 ppm oil surrogate, dilute to 10mL volume with DCM/Hexane 65:35

↳ Waste Dilution Extraction / Prep LOG
• Checked/calibrated OHAUS scale: SN# 1356
with TROEMNER weights: SN# 000897
• Recorded on page 5 of SERAS-L-0157 logbook

Sample	wt.	Final Vol	Surr Added
BLK022412WD	—	10.0mL	100µl of RUK0077
SERAS-017-0004	0.193g	10.0mL	100µl " "
SERAS-017-0003	0.103g	10.0mL	100µl " "
SERAS-017-0003d	0.189g	10.0mL	100µl " "
SERAS-017-0004A	1.029g	10.0mL	100µl RUK0077

↳ used DCM/Hex 65:35 + 20% Acetone to extract + Also - a beaker had washed out of dust + 1 small rock

SERAS-017-0004 - chunks of oily soil - visibly oil soaked debris - wood. Formed soil swirl balls like Vortex - will do again with DCM/Hex + 10% Acetone

2/24/12

Continued on Page

Read and Understood By

Signed 2/24/12 Date

Signed 3-5-12 Date

2/27/12 Monday
- Sequence that was started 2/24/12 extracts halted - Data not usable according to D-5225
- Centrifuging sample today before weighing -

Sample	wt.	Final Vol	Surr. Added
BLK022712	0.1g	10 ml	100 µl of RVK0077
SERAS-017-0003	0.116g	10ml	100 ul
SERAS-017-0003 dup	0.111g	10ml	100 ul
SERAS-017-004	0.029g	2.0ml	20 ul RVK0077
RVB0026	0.121g	10ml	100ul CUC Sample
AMTSTOCK	0.100g	10ml	NO Surr
CUC-B80	0.508g	10ml	NO Surr
SERAS-017-004	0.825		cont 7.664
CUC-1590			cont

3/1/2012 Thursday
- Extracting Pure Product for Flemingsburg Ky

Sample	wt.	Fin Vol	Surr Added
BLK030112	0.1g	10ml	100 µl RVK0077

Centrifuged sample FOR-TRENCH + FOR-CREEK from COC# in AES: LM 2/28/12

R202018-01	0.055g	5.0ml	50 µl RVK0077
R202018-01 dup	0.105g	5.0ml	50 µl RVK0077
R202018-02	0.148g	5.0ml	50 µl RVK0077

↗ Big Emulsion - Could use more centrifuge!

SERAS-017-0005	0.104g	10 ml	100 µl RVK0027
SERAS-017-0006	0.102g	10 ml	100 ul
SERAS-017-0005 dup	0.115g	10 ml	100 µl RVK0027

Continued on Page

Read and Understood By

Signed 3/1/12 Date

Signed 3-5-12 Date

US EPA ARCHIVE DOCUMENT

3/6/12
Tuesday - Going to simulate autoclave of Bulk Extracted CUC oil & determine if Dry mat change
- USING BVB0026 - The oil that will contain BCH
- could save time & need BEO
- will put in GC oven at 120°C

Vial wt = 4.634g
Vial + oil = 5.234g
5.192
Loss 7.3%

Before bake .602g
+.044

Autoclaved BEO - 0.1188g → 10ml DCM/Hex
= 62,000 ppm → dilute 1:10x before inject

004 Flask + lid 17.174
18.446 - 1.272g

Bake in GC oven at 120 - use with gauze

ADD 10ml DCM/Hex

2nd one - TO test bake

004 Vial = 4.883
= 5.005g
= 4.96g

Oil - Put in 2Hg at 125° 4.975 = 0.092g

Weigh 003 will cook its AM to Hex 2.0ml

VIAL = 12.407
12.614g 6ch 12.585

= 0.178g ~ Adjust to 3/4 use as CCV for Extends 0.1 ECV

divide by 4's

Continued on Page

Read and Understood By

Joel Dyste 3/6/12
Signed Date

Signed 3/12/12
Date

3/7/2012
Wed. • Preparing SERAS-017-0004 product for Fenell Miller: he will use for biodegradation study.
• Sample-0004 consists of an oil soaked mud/solid. Tried to separate oil from solid/mud by just centrifuge - but the product with is very viscous and difficult to work with
• Centrifuging followed by heating entire sample in water bath at 50°C for prolonged period only released larger water beads and broke final bond.
• To remove water - I'll place whatever Basket can of top layer and put in Petri Dish - Take bake at 125°C for 16 hour in GC/AS - Let that remove water - Clean Glassy
• Calibrating SCALE - in logbook SERAS-L-0157 - page 51

↳ 20mL Vial weight = 13.920g
- Decided to use 30mL Vial Vial: 20.984g
In Oven @ 11:30 30.234g - Vial ID
DOW 28.916
= 9.25g wet
= 7.932 cooke at 125

control 14%
Fenell 6.514g

3/7/12

Continued on Page

Read and Understood By

Signed — Date — Signed — Date

3/12/12

US EPA ARCHIVE DOCUMENT

3/20/12

- Making EO TPH ICAL from cleaned + autoclaved SERAS-017-0004 oil
  - weighed 0.503g → 10mL DCM/hexane for 50,000ppm STOCK
  - * Note this oil has that unique "non sticky" physical property — it sticks to itself ~ but not to the object
    - this is like "sample 0000" from Suncor
  - ⇒ Experiment - will take ~ 1.0g oil - Dilute 1:1 DCM
    - Bake off DCM ~ see if "stuff" settles out - Maybe solid
    - See if "stuff" STICKY

EX032012A: 1.267g → ~5mL DCM
- CENTRIFUGE 3.0mL ↑
  - a layer of scum with came to top ~ 50µL
  - → pipetting out 2.0mL of DCM layer & Tared 20mL Vial that — will evap off DCM
    - Vial weight 13.900g
    - 16.339g with DCM + EVO
    - after some DCM bake 14.385
    - after 125° Autoclave 14.378
- ⇒ have 0.178g of AUTOCLAVED 017-0004 oil
  - adding 5.0mL hexane → want to tap and clean
    - = 35,600 ppm
    - = dilute 140µL → 1mL = 5000 ppm
- * It did dissolve in hexane: little black specks everywhere. It formed an emulsion that settles to bottom
  - Added 1mL CS_2 ~ still emulsion
  - Adding 1mL DCM ⇒ got rid of 90% of specks
  - One more mL DCM
    - most specks gone + ok ~ keeping glob
- 8mL Total: Dilute ~ 22,300ppm → dilute 4x DCM/hex
  - = EX032012B: 8mL dilute 4x

Continued on Page

Read and Understood By

SERAS-017-DTM-011413_3

Signed 3/21/12 Date — Vivek Tanwar 0142 Signed 5/2/12 Date

3/21/12 ~ 80 mg/kg
Ex032112A: Took 1.0 ml of the 8 ml extract and added 200 µl of acetone

Ex032112B: Took 1.0 ml of 8 ml extract and added 200 µl of Methanol

- New AcroClave 30 ml + Cap = ~~25.476 g~~ 25.988 g
25.796 g Lost mass at 130°C

Ex032112C
Got 0.330 g → dilute with 6.0 ml DCM / Hexane / Acetone / MeOH
60 30 5 5
It seems like it worked - no top globs or residual
= 55,000 µg/ml ~ all went into solution

Dlynch 3/21/12 →

3/24/12
Saturday Acid cleaning some oils to check effects on PAH's + maybe hopane
Ex032412A: 10,000 ppm Amit Oil → used motor oil
0.1 g → 10 mL Hexane
Ex032412B = 10 ml Hexane blank + 100 µl RVC0032
Ex032412C = Acid cleaned Ex032412B
Used 1 ml extract with 2 ml H_2SO_4 50%
Ex032412D = Acid cleaned Ex032412A
- put brown extract on K_2SO_4 to remove traces of H_2SO_4

Continued on Page

Read and Understood By

Dlynch 4/5/12 Signed Date

4/6/12 Signed Date

Continued From Page

4/19/12

Day 0 Samples

SERAS-017-0007 0003 oil

SERAS-017-0013 0004 oil

Day 14 Samples

SERAS-017-0014 0002 oil

SERAS-017-0019 0004 oil

- Centrifuging @ 3500 rpm ~2.5 ml of each oil extract then ~2 ml each + evaporate to final 4.0 ml vials

VIALS + CAP + CHIPS	OHAUS	EMPTY Mettler 240		CVV Extract 2.0 ml	Mg oil
0007 = A:	6.112	6.1114 g	8.698	6.1844 g	
0013 = B:	6.100	6.1029 g	8.632	6.1756 g	
0014 = C:	6.152	6.1566 g	8.681	6.2108 g	
0019 = D:	6.148	6.1485 g	8.770	6.2076 g	

* Checking METTLER AE 240 with Troemner weights Serial # 004597

WT 10g = 10.0000 1g = 1.0000 0.1g = 0.1001
0.020g = 0.0200 0.010g = 0.0100g

Continued on Page

Read and Understood By

4/19/12 Date

Signed Date 5/2/12

US EPA ARCHIVE DOCUMENT

4/19/12 Thursday
- Predicted that Fugell needs to combine 3 soils from CUC to produce the oil mixture used from CUC Day 0 → Day 84 20N mix and mailed the bulk extracted oil RV 3002C.

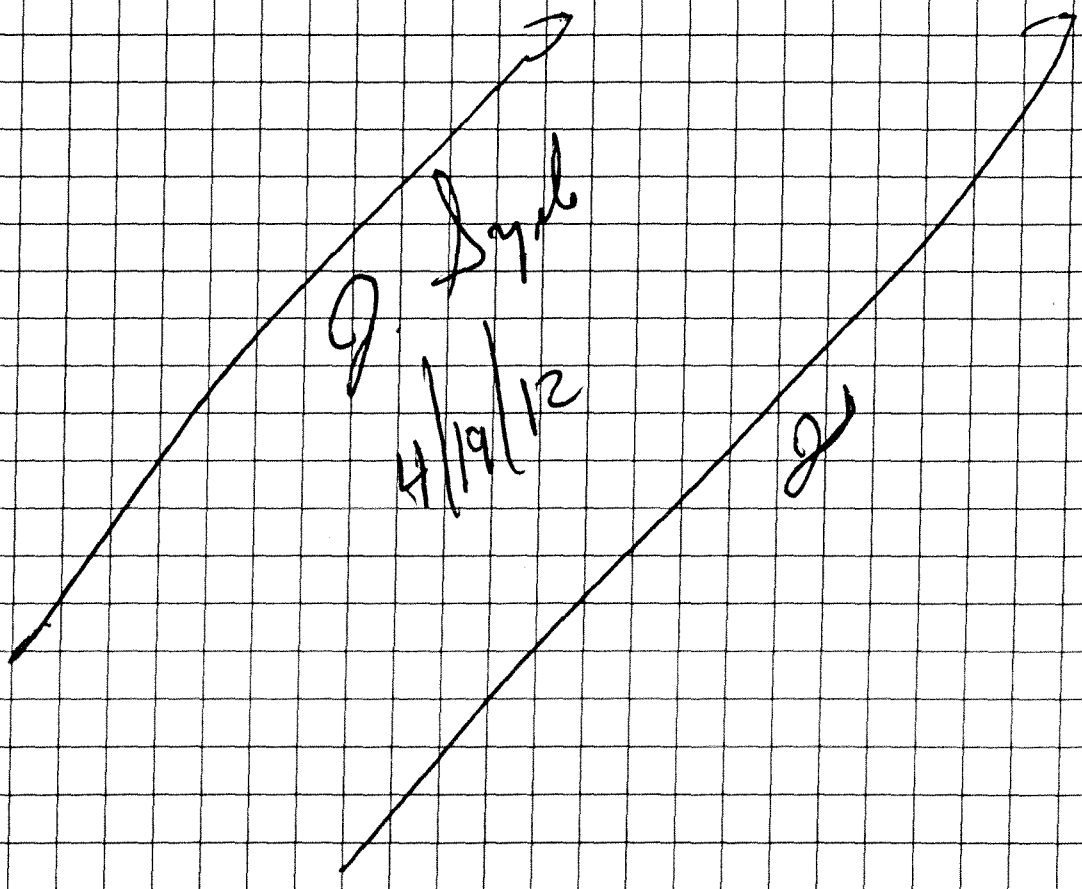
Combine equal weight CUC-01, CUC-03, CUC-06 } Sub : T.F.H gave me sample

I weighed 8.535g ; started with DCM + MeOH 10ml+1ml 2x
3ml+2ml 1x

8.535 = 27 ml total

centrifuging 15min @ 3000 rpm

*Total dil = 100x for original - 01, 03, 06 10g → 40ml the 10x
combined - can 10g → 10ml × 2x = 200x
for 3000 Grant #2
8.535 → 27 ~ 3.1 - Should be OK



Continued on Page

Read and Understood By

J. Fyfe — Signed — 4/19/12 — Date

Kirsten Faucett — Signed — 5/2/12 — Date

STANDARDS PREP.

LIMS LOGS

FOR ONLY 6 Samples

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

0147

Analytical Standard Record
ERT/SERAS Laboratory
RVE0033

Description:	CUC BEO 05/18/12 Neat Product	Expires:	11/15/22 0
Standard Type:	Reagent	Prepared:	05/19/12 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/19/12 08:06 by JS
Vendor:	SERAS	LotNumber:	Extracted from CUC soil

Extracted from soil sample: SERAS-135-0083 on COC# SERAS-135-05/16/12-0015
Gave Ferrell 9.6g, I kept 2g for inventory/standards. J. Syslo

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVE0033

Description:	CUC BEO 05/18/12 Neat Product	Expires:	11/15/22 0
Standard Type:	Reagent	Prepared:	05/19/12 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/19/12 08:06 by JS
Vendor:	SERAS	LotNumber:	Extracted from CUC soil

Extracted from soil sample: SERAS-135-0083 on COC# SERAS-135-05/16/12-0015
Gave Ferrell 9.6g, I kept 2g for inventory/standards. J. Syslo

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By 5/19/12 J Syslo Date

Page 1 of 1

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVD0079

Description:	2nd Source DRO/TPH LCS Spike	Expires:	10/24/12 0
Standard Type:	Analyte Spike	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 20:29 by JS
Vendor:	--	LotNumber:	RVC0018

I made 50ml and delivered 25mL to Central Lab; 4/9/12
Diluted 2x, called ICV #2 for DRo ICAL: JS

Analyte	CAS Number	Concentration	Units
Diesel	NA	2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVD0017	Diesel Fuel - Neat Sandard	04/09/12	John Syslo	10/06/22	04/09/12 17:47 by JS	0.05

Reviewed By 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0078

Description:	DROTPH ICV 1000ppm	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 20:26 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	B2120083

50x dilution of 2nd source DRO-TPH standard + OilIS: Called ICV #1

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0057	#2 Diesel (2nd source) Mix 50,000	04/25/12	John Syslo	08/10/22	04/26/12 18:34 by JS	0.02

Reviewed By 5/19/12 Date

US EPA ARCHIVE DOCUMENT

SERAS-0177-OTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVD0077

Description:	50ppb DROTPH + 0.5ppm Surr.	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	*	LotNumber:	RVC0018

Made serial dilution of 20K DRO stock in 1.0mL solvent: added 20µL OilIS RVC0035

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	0.5	ug/mL
Terphenyl-d14	NA	0.5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	0.5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	0.5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0071	20K DRO & 200ppm Surr. ICAL S	04/27/12	John Syslo	10/27/12	04/27/12 17:23 by JS	0.0025

Reviewed By J Syslo 5/19/12 Date

0149

Analytical Standard Record
ERT/SERAS Laboratory
RVD0076

Description:	100ppb DROTPH + 1.0ppm Surr.	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	*	LotNumber:	RVC0018

Made serial dilution of 20K DRO stock in 1.0mL solvent: added 20µL OilIS RVC0035

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1	ug/mL
Terphenyl-d14	NA	1	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		100	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	1	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	1	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0071	20K DRO & 200ppm Surr. ICAL S	04/27/12	John Syslo	10/27/12	04/27/12 17:23 by JS	0.005

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVD0075

Description:	500ppb DROTPH + 5.0ppm Surr.	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	*	LotNumber:	RVC0018

Made serial dilution of 20K DRO stock in 1.0mL solvent: added 20µL OilIS RVC0035

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	5	ug/mL
Terphenyl-d14	NA	5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0071	20K DRO & 200ppm Surr. ICAL S	04/27/12	John Syslo	10/27/12	04/27/12 17:23 by JS	0.025

Reviewed By _____ 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0074

Description:	1.0k DROTPH + 10ppm Surr.	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	*	LotNumber:	RVC0018

Made serial dilution of 20K DRO stock in 1.0mL solvent: added 20µL OilIS RVC0035

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	10	ug/mL
Terphenyl-d14	NA	10	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0071	20K DRO & 200ppm Surr. ICAL S	04/27/12	John Syslo	10/27/12	04/27/12 17:23 by JS	0.05

Reviewed By _____ 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0151

Analytical Standard Record
ERT/SERAS Laboratory
RVD0073

Description:	5.0k DROTPH + 50ppm Surr.	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	*	LotNumber:	RVC0018

Made serial dilution of 20K DRO stock in 1.0mL solvent: added 20µL OilIS RVC0035

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	50	ug/mL
Terphenyl-d14	NA	50	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	50	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	50	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0071	20K DRO & 200ppm Surr. ICAL S	04/27/12	John Syslo	10/27/12	04/27/12 17:23 by JS	0.25

Reviewed By J. Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVD0072

Description:	10k DROTPH + 100ppm Surr.	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	*	LotNumber:	RVC0018

Made serial dilution of 20K DRO stock in 1.0mL solvent: added 20µL OilIS RVC0035

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVD0071	20K DRO & 200ppm Surr. ICAL S	04/27/12	John Syslo	10/27/12	04/27/12 17:23 by JS	0.5

Reviewed By J. Syslo 5/19/12 Date

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US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RVD0071

Description:	20K DRO & 200ppm Surr. ICAL Stock	Expires:	10/27/12 0
Standard Type:	Calibration Stan	Prepared:	04/27/12 0
Solvent:	DCM/Hexane 65/35 (RVC0018)	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 17:23 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	211051030

Primary Source Diesel + Surrogate mixture for DRO/TPH ICAL. Make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Oil Fingerprint		20000	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
5a-Androstane	438-22-2	200	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.2
RVD0052	#2 Diesel Fuel Mix 50,000 ppm	04/25/12	John Syslo	05/05/21	04/26/12 18:32 by JS	0.8

Reviewed By 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVD0060

Description:	#2 Diesel (2nd source) Mix 50,000 ppm	Expires:	08/10/22 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 21:35 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	B2120083

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By 5/19/12 Date

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SERAS-017-DTM-011413 3
0153

Analytical Standard Record
ERT/SERAS Laboratory
RVD0059

Description:	#2 Diesel (2nd source) Mix 50,000 ppm	Expires:	08/10/22 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 21:34 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	B2120083

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0058

Description:	#2 Diesel (2nd source) Mix 50,000 ppm	Expires:	08/10/22 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 21:34 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	B2120083

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0154

Analytical Standard Record
ERT/SERAS Laboratory
RVD0057

Description:	#2 Diesel (2nd source) Mix 50,000 ppm	Expires:	08/10/22 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/26/12 18:34 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	B2120083

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0056

Description:	#2 Diesel (2nd source) Mix 50,000 ppm	Expires:	08/10/22 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/26/12 18:33 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	B2120083

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0155

Analytical Standard Record
ERT/SERAS Laboratory
RVD0055

Description:	#2 Diesel Fuel Mix 50,000 ppm	Expires:	05/05/21 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 21:33 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	211051030

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J. Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0054

Description:	#2 Diesel Fuel Mix 50,000 ppm	Expires:	05/05/21 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 21:33 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	211051030

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J. Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVD0053

Description:	#2 Diesel Fuel Mix 50,000 ppm	Expires:	05/05/21 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/27/12 21:33 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	211051030

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J. Syl 5/19/12 Date

Page 1 of 1

Analytical Standard Record

ERT/SERAS Laboratory

RVD0052

Description:	#2 Diesel Fuel Mix 50,000 ppm	Expires:	05/05/21 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/26/12 18:32 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	211051030

Cat# DRO-AK-102-DCS-10X-R1-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By J. Syl 5/19/12 Date

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US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RVD0051

Description:	#2 Diesel Fuel Mix 50,000 ppm	Expires:	05/05/21 0
Standard Type:	Calibration Stan	Prepared:	04/25/12 0
Solvent:	Dichloromethane	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/26/12 18:32 by JS
Vendor:	AccuStandard.Inc.	LotNumber:	211051030

Cat# DRO-AK-102-DCS-10X-RI-PAK Stock Diesel Calibration Composite Mix.

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Reviewed By JS 5/19/12 Date

Page 1 of 1

Analytical Standard Record
ERT/SERAS Laboratory
RVD0049

Description:	CUC BEO 04/23/12 Neat Product	Expires:	10/20/22 0
Standard Type:	Reagent	Prepared:	04/23/12 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/23/12 18:05 by JS
Vendor:	SERAS	LotNumber:	Extracted from CUC soil

extracted from combined soil sample: SERAS-135-0079 on COC# SERAS-135-04/18/12-0013
Got 48g of oil, gave Ferrell 45g, I kept 3g for inventory/standards. J. Syslo

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By JS 5/19/12 Date

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0157

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RVD0016

Description:	CUC BEO 04/05/12	Expires:	10/02/17 0
Standard Type:	Reagent	Prepared:	04/05/12 0
Solvent:	Dichloromethane (RVC0017)	Prepared By:	John Syslo
Final Volume (mls):	3.024	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/06/12 18:37 by JS
Vendor:	SERAS	LotNumber:	Extracted from CUC soil

See page 23 + 24, SERAS-L-0146: Processed Bulk Extracted Oil, & Weg-Bake at 150 degrees. 3.024g recovered; DCM free. JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0017

Description:	Diesel Fuel - Neat Sandard	Expires:	10/06/22 0
Standard Type:	Reagent	Prepared:	04/09/12 0
Solvent:	Neat standard	Prepared By:	John Syslo
Final Volume (mls):	100	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/09/12 17:47 by JS
Vendor:	--	LotNumber:	---

Logging in 100mL of neat 2nd source Diesel Fuel - Unknown source: J. Syslo

Analyte	CAS Number	Concentration	Units
Diesel	NA	1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT

0159

Analytical Standard Record
ERT/SERAS Laboratory
RVD0015

Description:	CUC BEO 04/05/12	Expires:	10/02/12 0
Standard Type:	Reagent	Prepared:	04/05/12 0
Solvent:	Dichloromethane (RVC0017)	Prepared By:	John Syslo
Final Volume (mls):	40	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	04/05/12 15:20 by JS
Vendor:	SERAS	LotNumber:	Extracted fro CUC soil

40mL of diluted mixed product that was extracted & reclaimed on 2/13/12 , reclaimed again 04/05/12 from CUC-0135 soil : Concentration approx 25% oil; reprocess this to obtain NEAT product: See page 23, SERAS-L-0146, Syslo

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVD0018

Description:	2nd Source DRO/TPH LCS Spike	Expires:	10/06/12 0
Standard Type:	Analyte Spike	Prepared:	04/09/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	CENTRAL
Vials:	1	Last Edit:	04/11/12 15:07 by SS
Vendor:	--	LotNumber:	RVC0018

I made 50ml and delivered 25mL to Central Lab; 4/9/12

Analyte	CAS Number	Concentration	Units
Diesel	NA	2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVD0017	Diesel Fuel - Neat Sandard	04/09/12	John Syslo	10/06/22	04/09/12 17:47 by JS	0.05

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVC0066

Description:	5000ppm 2nd Source CCV foe Enbridge C	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 18:30 by JS
Vendor:	SERAS	LotNumber:	RVC0018

Diluted the baked crude oil standard 6x and use as 2nd source CCV for Enbridge Oil TPH ICAL; Syslo

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		4954.4	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0065	30,000ppm TPH 2nd Source Enbri	03/22/12	John Syslo	09/18/12	03/22/12 18:26 by JS	0.167

Reviewed By J Syslo 5/19/12 Date

0160

Analytical Standard Record
ERT/SERAS Laboratory
RVC0065

Description:	30,000ppm TPH 2nd Source Enbridge Oil	Expires:	09/18/12 0
Standard Type:	Reagent	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	6	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 18:26 by JS
Vendor:	SERAS	LotNumber:	RVC0018

Cleaned SERAS-017-0003 crude, then baked at 125 degrees C for 1 hour to simulate autoclave. Looks like -0004 now. JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		29667	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0064	ENBRIDGE TPH 2nd Source Stoc	03/22/12	John Syslo	09/18/12	03/22/12 18:22 by JS	0.178

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0161

Analytical Standard Record
ERT/SERAS Laboratory
RVC0064

Description:	ENBRIDGE TPH 2nd Source Stock NEAT	Expires:	09/18/12 0
Standard Type:	Reagent	Prepared:	03/22/12 0
Solvent:	Fresh but Baked Enbridge Crude	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 18:22 by JS
Vendor:	SERAS	LotNumber:	Cleaned & baked samnle 0003

2.0 Grams of Enbridge Oil product: From Sample SERAS-017-0003. Centrifuged & autoclaved. 3/06/2012; page 18 of SERAS-L-0146

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0063

Description:	0.5K EO TPH ICAI Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:22 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2	ug/mL
Terphenyl-d14	NA	2	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	2	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	2	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.01

Reviewed By J Syslo 5/19/12 Date

SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVC0062

Description:	1.0K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:16 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	4	ug/mL
Terphenyl-d14	NA	4	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	4	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	4	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.02

Reviewed By J Syslo 5/19/12 Date

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0162

Analytical Standard Record
ERT/SERAS Laboratory
RVC0061

Description:	25K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:17 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		25000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.5

Reviewed By J Syslo 5/19/12 Date

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US EPA ARCHIVE DOCUMENT

0163

Analytical Standard Record
ERT/SERAS Laboratory
RVC0060

Description:	10K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:15 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	40	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.2

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0059

Description:	5.0K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 15:08 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057L; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.1

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVC0058

Description:	50K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 15:07 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057L; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	200	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	1

Reviewed By J Syslo 5/19/12 Date

0164

Analytical Standard Record
ERT/SERAS Laboratory
RVC0057

Description:	50K EO TPH Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2.2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 15:04 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Has surrogates - do serial dilutions for ICAL; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Oil Fingerprint		50000	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
5a-Androstane	438-22-2	200	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.22
RVC0056	Enbridge TPH Stock	03/21/12	John Syslo	09/17/12	03/21/12 17:06 by JS	2

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RVC0056

Description:	Enbridge TPH Stock	Expires:	09/17/12 0
Standard Type:	Reagent	Prepared:	03/21/12 0
Solvent:	DCM/Hexane/Acetone/Methanol (	Prepared By:	John Syslo
Final Volume (mls):	6	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/21/12 17:06 by JS
Vendor:	SERAS	LotNumber:	NA

This TPH standard is prepared from SERAS-017-0004 oil. The solvent mixture was necessary because of disolution problems. J. Syslo

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		55000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0050	ENBRIDGE TPH Stock NEAT	03/20/12	John Syslo	09/16/22	03/20/12 15:26 by JS	0.33

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0054

Description:	5,000 ppm CCV for CUC TPH	Expires:	09/15/12 0
Standard Type:	Reagent	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	0.5	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 16:38 by JS
Vendor:	SERAS	LotNumber:	LIMS # RVC0018

Using Enbridge Oil Product: SERAS-017-0004: Surrogates added ; Also Check of 5000ppm EO TPH Spike for central

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.005
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.01
RVC0053	5000ppm TPH Spike for Enbridge	03/20/12	John Syslo	09/20/12	03/20/12 15:36 by JS	0.5

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVC0052

Description:	25K EO-TPH & 100ppm Surr. ICAL Stocl	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:31 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using Enbridge Oil Sample SERAS-017-0004. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Oil Fingerprint		25000	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
5a-Androstane	438-22-2	100	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.1
RVC0051	50,000 ppm EO TPH Std	03/20/12	John Syslo	09/16/12	03/20/12 15:28 by JS	1

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0051

Description:	50,000 ppm EO TPH Std	Expires:	09/16/12 0
Standard Type:	Reagent	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:28 by JS
Vendor:	SERAS	LotNumber:	LIMS # RVC0018

From Enbridge Oil Sample SERAS-017-0004: This is to make LCS/MS spikes and ICAL from site extracted oil: No Surrogates

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0050	ENBRIDGE TPH Stock NEAT	03/20/12	John Syslo	09/16/22	03/20/12 15:26 by JS	0.5

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0050

Description:	ENBRIDGE TPH Stock NEAT	Expires:	09/16/22 0
Standard Type:	Reagent	Prepared:	03/20/12 0
Solvent:	Weathered Enbridge Crude	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:26 by JS
Vendor:	SERAS	LotNumber:	Cleaned & baked sample 0004

2.0 Grams of Enbridge Oil product: From Sample SERAS-017-0004. Centrifuged & autoclaved. 3/07/2012

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/21/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0049

Description:	0.25K TPH & 1.0ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1	ug/mL
Terphenyl-d14	NA	1	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		250	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	1	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	1	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 15:20 by JS	0.01

Reviewed By J Syslo 5/21/12 Date

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US EPA ARCHIVE DOCUMENT
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Analytical Standard Record
ERT/SERAS Laboratory
RVC0048

Description:	0.5 K TPH & 2ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2	ug/mL
Terphenyl-d14	NA	2	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	2	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	2	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 15:20 by JS	0.02

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0046

Description:	10 K TPH & 40ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	40	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 15:20 by JS	0.4

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0045

Description:	1.0 K TPH & 4ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	4	ug/mL
Terphenyl-d14	NA	4	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	4	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	4	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 15:20 by JS	0.04

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0044

Description:	5.0 K TPH & 20ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 15:20 by JS	0.2

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0043

Description:	25K TPH & 100ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		25000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 15:20 by JS	1

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0042

Description:	25K TPH & 100ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Oil Fingerprint		25000	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
5a-Androstane	438-22-2	100	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.1
RVC0040	50,000 ppm CUC TPH Std	03/19/12	John Syslo	09/15/12	03/20/12 15:20 by JS	1

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0041

Description:	SV Internal Std. Mix, 2000 ppm	Expires:	09/16/12 0
Standard Type:	Internal Standar	Prepared:	03/20/12 0
Solvent:	DCM	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	4	Last Edit:	03/20/12 11:46 by JS
Vendor:	Restek	LotNumber:	A082566

CAT#31206 : This is one of 5 vials that I am opening up for use.

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	2000	ug/mL
Perylene-d12	NA	2000	ug/mL
Naphthalene-d8	NA	2000	ug/mL
Chrysene-d12	NA	2000	ug/mL
Acenaphthene-d10	NA	2000	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	2000	ug/mL

Reviewed By J Syd 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0040

Description:	50,000 ppm CUC TPH Std	Expires:	09/15/12 0
Standard Type:	Reagent	Prepared:	03/19/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	LIMS # RVC0018

From bulk extracted oil: This is to make LCS/MS spikes and ICAL from site extracted oil: No Surrogates

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0038	CUC-BEO 2/22/12	03/19/12	John Syslo	09/15/22	03/20/12 15:20 by JS	0.5

Reviewed By J Syd 5/19/12 Date

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SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVC0038

Description:	CUC-BEO 2/22/12	Expires:	09/15/22 0
Standard Type:	Reagent	Prepared:	03/19/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	3	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	Bulk Extracted Oil from Soil

1.0 grams of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : Methylene chloride baked off on 2/17/12: This is portion of the extra sample that I had and gave to T.F.M on 03/02/12: JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVC0035

Description:	500ppm Oil IS Mix	Expires:	09/15/12 0
Standard Type:	Internal Standar	Prepared:	03/19/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 11:48 by JS
Vendor:	SERAS	LotNumber:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RCv0034) and 2000ppm BNA IS (RUK0081) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0034	TPH IS Mix: 2000ppm : 3 Compo	03/19/12	John Syslo	09/15/12	03/19/12 11:31 by JS	0.5
RVC0041	SV Internal Std. Mix, 2000 ppm	03/20/12	John Syslo	09/16/12	03/20/12 11:46 by JS	0.5

Reviewed By J Syslo 5/19/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0034

Description:	TPH IS Mix: 2000ppm : 3 Compounds	Expires:	09/15/12 0
Standard Type:	Internal Standar	Prepared:	03/19/12 0
Solvent:	DCM/CS2 4:1	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/19/12 11:31 by JS
Vendor:	NA	LotNumber:	NA

2000ppm 3 Compound IS Mix for TPH. To be mixed with BNA IS
DCM = RCV0017; Cs2 = RVA0051

Analyte	CAS Number	Concentration	Units
n-Tetradecane-d30		2000	mg/Kg
n-Tetracosane-d50	16416-32-3	2000	mg/Kg
n-Hexatriacontane-d74	16416-34-5	2000	mg/Kg

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0123	Neat D50-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 14:26 by JS	0.05
RSF0124	Neat D74-n-Hexatriacontane	06/30/09	John Syslo	12/27/19	06/30/09 14:33 by JS	0.05
RSF0126	Neat D30-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 13:56 by JS	0.05

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record

ERT/SERAS Laboratory

RVC0032

Description:	2000ppm Oil Surrogate Stock	Expires:	09/15/12 0
Standard Type:	Surrogate Spike	Prepared:	03/19/12 0
Solvent:	DCM/CS2 75:25	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/19/12 10:07 by JS
Vendor:	REAC	LotNumber:	REAC

Mix of 4 neat surrogates for Oil Surrogate Stock. 0.1g each
DCM = RCV0017, and CS2 = RVA0051

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2000	ug/mL
Terphenyl-d14	NA	2000	ug/mL
Anthracene-d10	1719-06-8	2000	ug/mL
5a-Androstane	438-22-2	2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0090	Neat Triacontane-d62	06/29/09	John Syslo	06/26/19	06/30/09 13:27 by JS	0.1
RSF0092	Neat o-Terphenyl-d14	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0093	Neat Anthracene-d10	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0122	Neat 5a-Androstane	06/30/09	John Syslo	12/27/19	06/30/09 13:25 by JS	0.1

Reviewed By J Syslo 5/19/12 Date

SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RVC0018

Description:	DCM/Hexane Mix 65/35	Expires:	03/07/13 0
Standard Type:	Reagent	Prepared:	03/07/12 0
Solvent:	DCM/DE507 & Hexane/H49E38	Prepared By:	John Syslo
Final Volume (mls):	1000	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/07/12 18:28 by JS
Vendor:	SERAS	LotNumber:	See Solvent/Solvent Lot

DCM/Hexane solvent mixture used to prepare BNA ICAL standards,: mix 650mL DCM + 350mL Hexane
JS

Analyte	CAS Number	Concentration	Units
Hexane	110-54-3	332500	ug/mL
Dichloromethane	75-09-2	650000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUC0021	Hexanes (95% n-hexane)	03/10/11	John Syslo	03/10/21	03/07/12 18:22 by JS	350
RVC0017	Dichloromethane 99.9% Solvent	03/07/12	John Syslo	03/07/22	03/07/12 17:01 by JS	650

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVC0017

Description:	Dichloromethane 99.9% Solvent	Expires:	03/07/22 0
Standard Type:	Reagent	Prepared:	03/07/12 0
Solvent:	Dichloromethane / DE507	Prepared By:	John Syslo
Final Volume (mls):	4000	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/07/12 17:01 by JS
Vendor:	Burdick & Jackson	LotNumber:	DB241

Solvent Dichloromethane . Honeywell/B&J Solvent. Cat. GC299-4
Logged into LIMS 03/07/12. J. Syslo
Burdick & Jackson listed as Vendor because Honeywell is misspelled.

Analyte	CAS Number	Concentration	Units
Dichloromethane	75-09-2	1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3
0175

Analytical Standard Record
ERT/SERAS Laboratory
RVC0006

Description:	CUC-BEO 12/22/12	Expires:	03/02/19 0
Standard Type:	Reagent	Prepared:	03/01/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	3	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/02/12 12:49 by JS
Vendor:	SERAS	LotNumber:	Bulk Extracted Oil from Soil

2.8 grams of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : Methylene chloride baked off on 2/17/12: This is extra sample that I had and gave to T.F.M on 03/02/12: JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/19/12 Date

Analytical Standard Record
ERT/SERAS Laboratory
RVB0060

Description:	Method/Waste Dilution Blank	Expires:	05/12/12 0
Standard Type:	Reagent	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 19:18 by JS
Vendor:	SERAS	LotNumber:	RUC0022

Added 10µL 2000ppm Surrogate (RUK0077) and 20µ 500ppm IS (RVB0058) to Solvent Blank. A waste dilution blank.

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUK0077	2000ppm Oil Surrogate Stock	11/14/11	John Syslo	05/12/12	11/14/11 16:50 by JS	0.01
RVB0058	500ppm Oil IS Mix	02/24/12	John Syslo	05/14/12	02/24/12 15:13 by JS	0.02

Reviewed By J Syslo 5/8/12 Date

SERAS-017-DTM-011413_3

0176

Analytical Standard Record
ERT/SERAS Laboratory
RVB0057

Description:	CUC-BEO 12/22/12	Expires:	08/20/29 0
Standard Type:	Reagent	Prepared:	02/22/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/22/12 09:20 by JS
Vendor:	SERAS	LotNumber:	Bulk Extracted Oil from Soil

9.25 grams of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : Methylene chloride baked off on 2/17/12: JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 5/15/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RVB0058

Description:	500ppm Oil IS Mix	Expires:	05/14/12 0
Standard Type:	Internal Standar	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 15:13 by JS
Vendor:	SERAS	LotNumber:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RUK0079) and 2000ppm BNA IS (RUK0081) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUK0079	TPH IS Mix: 2000ppm : 3 Compou	11/14/11	John Syslo	05/14/12	11/14/11 17:00 by JS	0.5
RUK0081	Stock SVOC Internal Std. Mix, 2(	11/15/11	John Syslo	05/15/12	11/15/11 15:06 by JS	0.5

Reviewed By J Syslo 5/15/12 Date

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SERAS-017-DTM-011413_3

Analytical Standard Record

ERT/SERAS Laboratory

RUL0003

Description:	10K CG-117 Fingerprint Reference	Expires:	05/24/12 0
Standard Type:	Reagent	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 19:13 by JS
Vendor:	-	LotNumber:	NA

Diluting neat product to obtain 10,000ppm standard. adding IS for SIM method.

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUL0002	10K CG-117 Fingerprint Reference	12/01/11	John Syslo	05/29/12	12/01/11 16:25 by JS	1
RVB0058	500ppm Oil IS Mix	02/24/12	John Syslo	05/14/12	02/24/12 15:13 by JS	0.02

Reviewed By JSyslo 5/19/17 Date

0177

DFTPP + Neat Standard

SERAS-017-DTM-011413_3

0178

Analytical Standard Record
ERT/SERAS Laboratory
RVB0025

Description:	50 ppm DFTPP + ISTD	Expires:	08/12/12 0
Standard Type:	Calibration Stan	Prepared:	02/14/12 0
Solvent:	DE507	Prepared By:	Ben Beauchaine
Final Volume (mls):	20	Department:	SVOCGCMS
Vials:	1	Last Edit:	04/19/12 09:45 by GA
Vendor:	--	LotNumber:	-

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	40	ug/mL
Perylene-d12	NA	40	ug/mL
Pentachlorophenol	87-86-5	50	ug/mL
Naphthalene-d8	NA	40	ug/mL
Decafluorotriphenylphosphine	NA	50	ug/mL
Chrysene-d12	NA	40	ug/mL
Benzidine	92-87-5	50	ug/mL
Acenaphthene-d10	NA	40	ug/mL
4,4'-DDT	50-29-3	50	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	40	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUA0036	GC/MS Tuning Mix (DFTPP), Sto	01/14/11	Ben Beauchaine	07/01/13	06/02/11 14:45 by JS	1
RUJ0028	SV Internal Std. Mix, 2000 ppm	04/12/12	Ben Beauchaine	10/12/12	04/19/12 09:45 by GA	0.4

Reviewed By _____ Date 5/19/12

Analytical Standard Record
ERT/SERAS Laboratory
RUA0036

Description:	GC/MS Tuning Mix (DFTPP), Stock, 100	Expires:	07/01/13 0
Standard Type:	MS Tune Soluti	Prepared:	01/14/11 0
Solvent:	Methylene Chloride	Prepared By:	Ben Beauchaine
Final Volume (mls):	1	Department:	SVOCGCMS
Vials:	5	Last Edit:	06/02/11 14:45 by JS
Vendor:	Restek	LotNumber:	A075699

Catalog # 31615; Mix of 4 compounds for 8270 DFTPP; dilute to 50ppm before use.
BB

Analyte	CAS Number	Concentration	Units
Pentachlorophenol	87-86-5	1000	ug/mL
Decafluorotriphenylphosphine	NA	1000	ug/mL
Benzidine	92-87-5	1000	ug/mL
4,4'-DDT	50-29-3	1000	ug/mL

Reviewed By _____ Date 5/19/12

US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

0179

Analytical Standard Record
ERT/SERAS Laboratory
RUJ0028

Description:	SV Internal Std. Mix, 2000 ppm	Expires:	10/12/12 0
Standard Type:	Internal Standar	Prepared:	04/12/12 0
Solvent:	DCM	Prepared By:	Ben Beauchaine
Final Volume (mls):	1	Department:	SVOCGCMS
Vials:	4	Last Edit:	04/19/12 09:45 by GA
Vendor:	Restek	LotNumber:	A082566

CAT#31206 :

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	2000	ug/mL
Perylene-d12	NA	2000	ug/mL
Naphthalene-d8	NA	2000	ug/mL
Chrysene-d12	NA	2000	ug/mL
Acenaphthene-d10	NA	2000	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	2000	ug/mL

Reviewed By _____ 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RSF0126

Description:	Neat D30-n-Tetradecane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:56 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	PR-16109/04285TD1

1 gram of neat Oil IS compound; CAT# DLM-670-1

Analyte	CAS Number	Concentration	Units
n-Tetradecane-d30		1000000	mg/Kg

Reviewed By _____ 5/19/12 Date

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Analytical Standard Record
ERT/SERAS Laboratory
RSF0124

Description:	Neat D74-n-Hexatriacontane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 14:33 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	P-7449

1.0 grams of Neat Oil IS cmpd. CAT# DLM-2634-1

Analyte	CAS Number	Concentration	Units
n-Hexatriacontane-d74	16416-34-5	1000000	mg/Kg

Reviewed By _____ Date 5/19/17

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Analytical Standard Record
ERT/SERAS Laboratory
RSF0123

Description:	Neat D50-n-Tetradecane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 14:26 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	PR-17753/09216TC1

0.5 grams of neat Oil IS compound CAT# DLM-2209-0.5

Analyte	CAS Number	Concentration	Units
n-Tetracosane-d50	16416-32-3	1000000	mg/Kg

Reviewed By _____ Date 5/17/17

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US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Analytical Standard Record
ERT/SERAS Laboratory
RSF0122

Description:	Neat 5a-Androstane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:25 by JS
Vendor:	Sigma	LotNumber:	098K4035

1 gram of neat Oil Surrogate cmpd. CAT# A0887-1G

Analyte	CAS Number	Concentration	Units
5a-Androstane	438-22-2	1000000	mg/Kg

Reviewed By: 5/19/12 Date

0181

Analytical Standard Record
ERT/SERAS Laboratory
RSF0093

Description:	Neat Anthracene-d10	Expires:	12/26/19 0
Standard Type:	Reagent	Prepared:	06/29/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:28 by JS
Vendor:	Isotec	LotNumber:	IY0212

1 gram of neat oil surrogate compound CAT# 176591-1G

Analyte	CAS Number	Concentration	Units
Anthracene-d10	1719-06-8	1000000	ug/mL

Reviewed By: 5/19/12 Date

US EPA ARCHIVE DOCUMENT

SERAS-172-DTM-011413

Analytical Standard Record
ERT/SERAS Laboratory
RSF0092

Description:	Neat o-Terphenyl-d14	Expires:	12/26/19 0
Standard Type:	Reagent	Prepared:	06/29/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:28 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	PR-19171

1 gram, neat oil surrogate compound, cat# DLM-450-1

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	NA	1000000	mg/Kg

Reviewed By _____ Date 5/15/12

0182

Analytical Standard Record
ERT/SERAS Laboratory
RSF0090

Description:	Neat Triacontane-d62	Expires:	06/26/19 0
Standard Type:	Reagent	Prepared:	06/29/09 0
Solvent:	neat	Prepared By:	John Syslo
Final Volume (mls):	0.5	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:27 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	P-8790

Neat TPH/Oil surrogate, 0.5grams, Cat# DLM-2210-0.5

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1000000	mg/Kg

Reviewed By _____ Date 5/15/12

US EXPORT CONTROLLED

Analytical Standard Record

ERT/SERAS Laboratory

RVC0066

Description:	5000ppm 2nd Source CCV foe Enbridge O	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 18:30 by JS
Vendor:	SERAS	LotNumber:	RVC0018

Diluted the baked crude oil standard 6x and use as 2nd source CCV for Enbridge Oil TPH ICAL; Syslo

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		4954.4	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0065	30,000ppm TPH 2nd Source Enbri	03/22/12	John Syslo	09/18/12	03/22/12 18:26 by JS	0.167

Reviewed By J Syslo 3/22/12 Date

0184

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVC0065

Description:	30,000ppm TPH 2nd Source Enbridge Oil	Expires:	09/18/12 0
Standard Type:	Reagent	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	6	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 18:26 by JS
Vendor:	SERAS	LotNumber:	RVC0018

Cleaned SERAS-017-0003 crude, then baked at 125 degrees C for 1 hour to simulate autoclave. Looks like -0004 now. JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		29667	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0064	ENBRIDGE TPH 2nd Source Stock	03/22/12	John Syslo	09/18/12	03/22/12 18:22 by JS	0.178

Reviewed By J Syslo 3/22/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVC0064

Description:	ENBRIDGE TPH 2nd Source Stock NEAT	Expires:	09/18/12 0
Standard Type:	Reagent	Prepared:	03/22/12 0
Solvent:	Fresh but Baked Enbridge Crude	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 18:22 by JS
Vendor:	SERAS	LotNumber:	Cleaned & baked sample 0003

2.0 Grams of Enbridge Oil product: From Sample SERAS-017-0003. Centrifuged & autoclaved. 3/06/2012; page 18 of SERAS-L-0146

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By 3/22/12 Date

SERAS-017-DTM-011413_3

0186

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVC0063

Description:	0.5K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:22 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2	ug/mL
Terphenyl-d14	NA	2	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	2	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	2	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.01

Reviewed By 3/22/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVC0062

Description:	1.0K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:16 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	4	ug/mL
Terphenyl-d14	NA	4	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	4	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	4	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.02

Reviewed By 3/22/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVC0061

Description:	25K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:17 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		25000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.5

Reviewed By J. Syslo 3/22/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0060

Description:	10K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 16:15 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	40	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.2

Reviewed By D. Syslo 3/22/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0059

Description:	5.0K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 15:08 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057L; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	0.1

J Syslo 3/22/12

Reviewed By Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0058

Description:	50K EO TPH ICAl Std.	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 15:07 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Serial dilution of RVC0057L; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	200	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0057	50K EO TPH Stock	03/22/12	John Syslo	09/15/12	03/22/12 15:04 by JS	1

Reviewed By 3/22/12 Date

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RVC0057

Description:	50K EO TPH Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/22/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2.2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/22/12 15:04 by JS
Vendor:	SERAS	LotNumber:	RVC0038

Site Specific Enbridge Oil-TPH. Has surrogates - do serial dilutions for ICAL; J. Syslo

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Oil Fingerprint		50000	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
5a-Androstane	438-22-2	200	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.22
RVC0056	Enbridge TPH Stock	03/21/12	John Syslo	09/17/12	03/21/12 17:06 by JS	2

Reviewed By J Syslo 3/22/12 Date

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RVC0056

Description:	Enbridge TPH Stock	Expires:	09/17/12 0
Standard Type:	Reagent	Prepared:	03/21/12 0
Solvent:	DCM/Hexane/Acetone/Methanol (	Prepared By:	John Syslo
Final Volume (mls):	6	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/21/12 17:06 by JS
Vendor:	SERAS	LotNumber:	NA

This TPH standard is prepared from SERAS-017-0004 oil. The solvent mixture was necessary because of disolution problems J. Syslo

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		55000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0050	ENBRIDGE TPH Stock NEAT	03/20/12	John Syslo	09/16/22	03/20/12 15:26 by JS	0.33

Placed ~ 200mg of baked K_2SO_4 in the standard to encourage water oil + prevent emulsional behavior. J Syslo

Reviewed By J Syslo 3/21/12 Date

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ERT/SERAS Laboratory

RVC0054

Description:	5,000 ppm CCV for CUC TPH	Expires:	09/15/12 0
Standard Type:	Reagent	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	0.5	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 16:38 by JS
Vendor:	SERAS	LotNumber:	LIMS # RVC0018

Using Enbridge Oil Product: SERAS-017-0004: Surrogates added ; Also Check of 5000ppm EO TPH Spike for central

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.005
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.01
RVC0053	5000ppm TPH Spike for Enbridge	03/20/12	John Syslo	09/20/12	03/20/12 15:36 by JS	0.5

Reviewed By J Syslo 3/20/12 Date

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RVC0053

Description:	5000ppm TPH Spike for Enbridge Oil	Expires:	09/20/12 0
Standard Type:	Analyte Spike	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	CENTRAL
Vials:	1	Last Edit:	03/20/12 15:36 by JS
Vendor:	SERAS	LotNumber:	Lims # RVC0018

Dilute 50K 10x DCM/Hexane : Using Oil extracted from EO Sample SERAS-017-0004

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		5000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0051	50,000 ppm EO TPH Std	03/20/12	John Syslo	09/16/12	03/20/12 15:28 by JS	1

Reviewed By 3/20/12 Date

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ERT/SERAS Laboratory

RVC0051

Description:	50,000 ppm EO TPH Std	Expires:	09/16/12 0
Standard Type:	Reagent	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:28 by JS
Vendor:	SERAS	LotNumber:	LIMS # RVC0018

From Enbridge Oil Sample SERAS-017-0004: This is to make LCS/MS spikes and ICAL from site extracted oil: No Surrogates

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0050	ENBRIDGE TPH Stock NEAT	03/20/12	John Syslo	09/16/22	03/20/12 15:26 by JS	0.5

Reviewed By 3/20/12 Date

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RVC0050

Description:	ENBRIDGE TPH Stock NEAT	Expires:	09/16/22 0
Standard Type:	Reagent	Prepared:	03/20/12 0
Solvent:	Weathered Enbridge Crude	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:26 by JS
Vendor:	SERAS	LotNumber:	Cleaned & baked sample 0004

2.0 Grams of Enbridge Oil product: From Sample SERAS-017-0004. Centrifuged & autoclaved. 3/07/2012

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 3/20/12 Date

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RVC0049

Description:	0.25K TPH & 1.0ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 14:46 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1	ug/mL
Terphenyl-d14	NA	1	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		250	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	1	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	1	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 12:28 by JS	0.01

Reviewed By J Syslo Date 3/20/12

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ERT/SERAS Laboratory

RVC0048

Description:	0.5 K TPH & 2ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 14:45 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2	ug/mL
Terphenyl-d14	NA	2	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	2	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	2	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 12:28 by JS	0.02

Reviewed By J. Syslo 3/20/12 Date

0200

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ERT/SERAS Laboratory

RVC0046

Description:	10 K TPH & 40ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 12:51 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	40	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 12:28 by JS	0.4

Reviewed By 3/20/12 Date

0201

SERAS-017-DTM-011413_3

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0045

Description:	1.0 K TPH & 4ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 12:31 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	4	ug/mL
Terphenyl-d14	NA	4	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	4	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	4	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 12:28 by JS	0.04

Reviewed By 3/20/12

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0044

Description:	5.0 K TPH & 20ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 12:29 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 12:28 by JS	0.2

Reviewed By J Syslo 3/20/12
Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0043

Description:	25K TPH & 100ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 12:28 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		25000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0035	500ppm Oil IS Mix	03/19/12	John Syslo	09/15/12	03/20/12 11:48 by JS	0.02
RVC0042	25K TPH & 100ppm Surr. ICAL S	03/20/12	John Syslo	09/15/12	03/20/12 12:28 by JS	1

Reviewed By: [signature] Date: 3/20/12

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0042

Description:	25K TPH & 100ppm Surr. ICAL Stock	Expires:	09/15/12 0
Standard Type:	Calibration Stan	Prepared:	03/20/12 0
Solvent:	DCM/Hexane 65/35 (RCV0018)	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 12:28 by JS
Vendor:	SERAS	LotNumber:	--

Weathered ORO TPH ICAL using CUC bulk extracted oil. Use this to make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Oil Fingerprint		25000	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
5a-Androstane	438-22-2	100	ug/mL

Parent Standards used in this standard

tandard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.1
RVC0040	50,000 ppm CUC TPH Std	03/19/12	John Syslo	09/15/12	03/19/12 16:12 by JS	1

Reviewed By J Syslo Date 3/20/12

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ERT/SERAS Laboratory

RVC0041

Description:	SV Internal Std. Mix, 2000 ppm	Expires:	09/16/12 0
Standard Type:	Internal Standar	Prepared:	03/20/12 0
Solvent:	DCM	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	4	Last Edit:	03/20/12 11:46 by JS
Vendor:	Restek	LotNumber:	A082566

CAT#31206 : This is one of 5 vials that I am opening up for use.

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	2000	ug/mL
Perylene-d12	NA	2000	ug/mL
Naphthalene-d8	NA	2000	ug/mL
Chrysene-d12	NA	2000	ug/mL
Acenaphthene-d10	NA	2000	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	2000	ug/mL

Reviewed By 3/20/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0040

Description:	50,000 ppm CUC TPH Std	Expires:	09/15/12 0
Standard Type:	Reagent	Prepared:	03/19/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	LIMS # RVC0018

From bulk extracted oil: This is to make LCS/MS spikes and ICAL from site extracted oil: No Surrogates

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		50000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0038	CUC-BEO 2/22/12	03/19/12	John Syslo	09/15/22	03/20/12 15:20 by JS	0.5

Reviewed By 3/20/12 Date

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ERT/SERAS Laboratory

RVC0039

Description:	5000ppm TPH Spike for CUC Oil	Expires:	09/15/12 0
Standard Type:	Analyte Spike	Prepared:	03/19/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	CENTRAL
Vials:	1	Last Edit:	03/20/12 15:37 by JS
Vendor:	SERAS	LotNumber:	Lims # RVC0018

Dilute 50K CUC site specific oil 10x with DCM/Hexane :

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		5000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0040	50,000 ppm CUC TPH Std	03/19/12	John Syslo	09/15/12	03/20/12 15:20 by JS	2.5

Reviewed By J Syslo Date 3/20/12

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ERT/SERAS Laboratory

RVC0038

Description:	CUC-BEO 2/22/12	Expires:	09/15/22 0
Standard Type:	Reagent	Prepared:	03/19/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	3	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 15:20 by JS
Vendor:	SERAS	LotNumber:	Bulk Extracted Oil from Soil

1.0 grams of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : Methylene chloride baked off on 2/17/12: This is portion of the extra sample that I had and gave to T.F.M on 03/02/12: JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By J Syslo 3/20/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0035

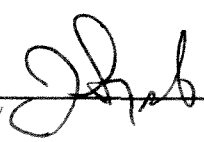
Description:	500ppm Oil IS Mix	Expires:	09/15/12 0
Standard Type:	Internal Standar	Prepared:	03/19/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/20/12 11:48 by JS
Vendor:	SERAS	LotNumber:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RCv0034) and 2000ppm BNA IS (RUK0081) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RVC0034	TPH IS Mix: 2000ppm : 3 Compo	03/19/12	John Syslo	09/15/12	03/19/12 11:31 by JS	0.5
RVC0041	SV Internal Std. Mix, 2000 ppm	03/20/12	John Syslo	09/16/12	03/20/12 11:46 by JS	0.5

Reviewed By  Date 3/20/12

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0034

Description:	TPH IS Mix: 2000ppm : 3 Compounds	Expires:	09/15/12 0
Standard Type:	Internal Standar	Prepared:	03/19/12 0
Solvent:	DCM/CS2 4:1	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/19/12 11:31 by JS
Vendor:	NA	LotNumber:	NA

2000ppm 3 Compound IS Mix for TPH. To be mixed with BNA IS
DCM = RCV0017; Cs2 = RVA0051

Analyte	CAS Number	Concentration	Units
n-Tetradecane-d30		2000	mg/Kg
n-Tetracosane-d50	16416-32-3	2000	mg/Kg
n-Hexatriacontane-d74	16416-34-5	2000	mg/Kg

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0123	Neat D50-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 14:26 by JS	0.05
RSF0124	Neat D74-n-Hexatriacontane	06/30/09	John Syslo	12/27/19	06/30/09 14:33 by JS	0.05
RSF0126	Neat D30-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 13:56 by JS	0.05

Reviewed By J. Syslo Date 3/19/12

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0033

Description:	40 ppm Oil Surrogate - 4 Cmpds.	Expires:	09/15/12 0
Standard Type:	Surrogate Spike	Prepared:	03/19/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	CENTRAL
Vials:	1	Last Edit:	03/20/12 08:05 by JS
Vendor:	SERAS	LotNumber:	RVC0018 for DCM/Hexane

Dilute 2000ppm RVC0032 (500µL in 25mL) with DCM/Hexane
Spike 500 µL per sample for FV of 1.0 mL

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
5a-Androstane	438-22-2	40	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
VC0032	2000ppm Oil Surrogate Stock	03/19/12	John Syslo	09/15/12	03/19/12 10:07 by JS	0.5

Reviewed By J Syslo 3/20/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0032

Description:	2000ppm Oil Surrogate Stock ✓	Expires:	09/15/12 0
Standard Type:	Surrogate Spike	Prepared:	03/19/12 0
Solvent:	DCM/CS2 75:25	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/19/12 10:07 by JS
Vendor:	REAC	LotNumber:	REAC

Mix of 4 neat surrogates for Oil Surrogate Stock. 0.1g each
DCM = RCV0017, and CS2 = RVA0051

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2000	ug/mL
Terphenyl-d14	NA	2000	ug/mL
Anthracene-d10	1719-06-8	2000	ug/mL
5a-Androstane	438-22-2	2000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0090	Neat Triacontane-d62	06/29/09	John Syslo	06/26/19	06/30/09 13:27 by JS	0.1
RSF0092	Neat o-Terphenyl-d14	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0093	Neat Anthracene-d10	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0122	Neat 5a-Androstane	06/30/09	John Syslo	12/27/19	06/30/09 13:25 by JS	0.1

Reviewed By J Syslo 3/19/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0018

Description:	DCM/Hexane Mix 65/35	Expires:	03/07/13 0
Standard Type:	Reagent	Prepared:	03/07/12 0
Solvent:	DCM/DE507 & Hexane/H49E38	Prepared By:	John Syslo
Final Volume (mls):	1000	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/07/12 18:28 by JS
Vendor:	SERAS	LotNumber:	See Solvent/Solvent Lot

DCM/Hexane solvent mixture used to prepare BNA ICAL standards: mix 650mL DCM + 350mL Hexane
JS

Analyte	CAS Number	Concentration	Units
Hexane	110-54-3	332500	ug/mL
Dichloromethane	75-09-2	650000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUC0021	Hexanes (95% n-hexane)	03/10/11	John Syslo	03/10/21	03/07/12 18:22 by JS	350
RVC0017	Dichloromethane 99.9% Solvent	03/07/12	John Syslo	03/07/22	03/07/12 17:01 by JS	650

Reviewed By 3/19/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0017

Description:	Dichloromethane 99.9% Solvent	Expires:	03/07/22 0
Standard Type:	Reagent	Prepared:	03/07/12 0
Solvent:	Dichloromethane / DE507	Prepared By:	John Syslo
Final Volume (mls):	4000	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/07/12 17:01 by JS
Vendor:	Burdick & Jackson	LotNumber:	DB241

Solvent Dichloromethane . Honeywell/B&J Solvent. Cat. GC299-4
Logged into LIMS 03/07/12. J. Syslo
Burdick & Jackson listed as Vendor because Honeywell is misspelled

Analyte	CAS Number	Concentration	Units
Dichloromethane	75-09-2	1000000	ug/mL

Reviewed By 3/19/12 Date

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Analytical Standard Record

ERT/SERAS Laboratory

RVC0006

Description:	CUC-BEO 12/22/12	Expires:	03/02/19 0
Standard Type:	Reagent	Prepared:	03/01/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	3	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/02/12 12:49 by JS
Vendor:	SERAS	LotNumber:	Bulk Extracted Oil from Soil

2.8 grams of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : Methylene chloride baked off on 2/17/12: This is extra sample that I had and gave to T.F.M on 03/02/12: JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By 3/1/12 Date

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ERT/SERAS Laboratory

RVB0060

Description:	Method/Waste Dilution Blank	Expires:	05/12/12 0
Standard Type:	Reagent	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 19:18 by JS
Vendor:	SERAS	LotNumber:	RUC0022

Added 10µL 2000ppm Surrogate (RUK0077) and 20µ 500ppm IS (RVB0058) to Solvent Blank. A waste dilution blank.

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	20	ug/mL
Terphenyl-d14	NA	20	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	20	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	20	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUK0077	2000ppm Oil Surrogate Stock	11/14/11	John Syslo	05/12/12	11/14/11 16:50 by JS	0.01
RVB0058	500ppm Oil IS Mix	02/24/12	John Syslo	05/14/12	02/24/12 15:13 by JS	0.02

Reviewed By: [signature] Date: 2/24/12

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ERT/SERAS Laboratory

RVB0058

Description:	500ppm Oil IS Mix	Expires:	05/14/12 0
Standard Type:	Internal Standar	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 15:13 by JS
Vendor:	SERAS	LotNumber:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RUK0079) and 2000ppm BNA IS (RUK0081) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUK0079	TPH IS Mix: 2000ppm : 3 Compoi	11/14/11	John Syslo	05/14/12	11/14/11 17:00 by JS	0.5
RUK0081	Stock SVOC Internal Std. Mix, 2(	11/15/11	John Syslo	05/15/12	11/15/11 15:06 by JS	0.5

Reviewed By D Syslo 2/24/12 Date

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ERT/SERAS Laboratory

RVB0057

Description:	CUC-BEO 12/22/12	Expires:	08/20/29 0
Standard Type:	Reagent	Prepared:	02/22/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	10	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/22/12 09:20 by JS
Vendor:	SERAS	LotNumber:	Bulk Extracted Oil from Soil

9.25 grams of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : Methylene chloride baked off on 2/17/12: JS

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By 2/22/12 Date

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ERT/SERAS Laboratory

RVB0026

Description:	CUC BEO 12/14/12	Expires:	08/12/22 0
Standard Type:	Reagent	Prepared:	02/14/12 0
Solvent:	Neat Oil Mixture	Prepared By:	John Syslo
Final Volume (mls):	20	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/14/12 12:44 by JS
Vendor:	SERAS	LotNumber:	Extracted from Soil

20mL of mixed product that was extracted & reclaimed on 2/13/12 from CUC-0135 soil : J. Syslo
Logging in the neat standard

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		1000000	ug/mL

Reviewed By _____ Date 2/14/12

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Analytical Standard Record

ERT/SERAS Laboratory

RVB0020

Description:	40 ppm Oil Surrogate - 4 Cmpds.	Expires:	05/12/12 0
Standard Type:	Surrogate Spike	Prepared:	02/13/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/13/12 15:55 by JS
Vendor:	SERAS	LotNumber:	RUC0022 for DCM/Hexane

Dilute 2000ppm RUK077 (500µL in 25mL) with DCM/Hexane
Spike 500 µL per sample for FV of 1.0 mL

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
5a-Androstane	438-22-2	40	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
UK0077	2000ppm Oil Surrogate Stock	11/14/11	John Syslo	05/12/12	11/14/11 16:50 by JS	0.5

Reviewed By 2/17/12 Date

Analytical Standard Record

ERT/SERAS Laboratory

RUL0003

Description:	10K CG-117 Fingerprint Reference	Expires:	05/24/12 0
Standard Type:	Reagent	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 19:13 by JS
Vendor:	-	LotNumber:	NA

Diluting neat product to obtain 10,000ppm standard. adding IS for SIM method.

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUL0002	10K CG-117 Fingerprint Reference	12/01/11	John Syslo	05/29/12	12/01/11 16:25 by JS	1
RVB0058	500ppm Oil IS Mix	02/24/12	John Syslo	05/14/12	02/24/12 15:13 by JS	0.02

Reviewed By _____ Date 2/24/12

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Analytical Standard Record

ERT/SERAS Laboratory

RUL0003

Description:	10K CG-117 Fingerprint Reference	Expires:	05/24/12 0
Standard Type:	Reagent	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 19:13 by JS
Vendor:	-	LotNumber:	NA

Diluting neat product to obtain 10,000ppm standard. adding IS for SIM method.

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUL0002	10K CG-117 Fingerprint Reference	12/01/11	John Syslo	05/29/12	12/01/11 16:25 by JS	1
RVB0058	500ppm Oil IS Mix	02/24/12	John Syslo	05/14/12	02/24/12 15:13 by JS	0.02

Reviewed By 2/24/12 Date

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US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUC0021

Description:	Hexanes (95% n-hexane)	Expires:	03/10/21 0
Standard Type:	Reagent	Prepared:	03/10/11 0
Solvent:	Hexane	Prepared By:	John Syslo
Final Volume (mls):	4000	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/07/12 18:22 by JS
Vendor:	J.T. Baker	LotNumber:	H49E38

Cat# 9262-03

Analyte	CAS Number	Concentration	Units
Hexane	110-54-3	950000	ug/mL

Reviewed By

3/9/12
Date

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Analytical Standard Record

ERT/SERAS Laboratory

RSF0126

Description:	Neat D30-n-Tetradecane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:56 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	PR-16109/04285TD1

1 gram of neat Oil IS compound; CAT# DLM-670-1

Analyte	CAS Number	Concentration	Units
n-Tetradecane-d30		1000000	mg/Kg

Reviewed By J Syslo 3/19/12 Date

SERAS-017-DTM-011413_3

0225

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RSF0124

Description:	Neat D74-n-Hexatriacontane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 14:33 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	P-7449

1.0 grams of Neat Oil IS cmpd. CAT# DLM-2634-1

Analyte	CAS Number	Concentration	Units
n-Hexatriacontane-d74	16416-34-5	1000000	mg/Kg

Reviewed By D Syslo Date 3/19/12

SERAS-017-DTM-011413_3

0226

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RSF0123

Description:	Neat D50-n-Tetradecane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 14:26 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	PR-17753/09216TC1

0.5 grams of neat Oil IS compound; CAT# DLM-2209-0.5

Analyte	CAS Number	Concentration	Units
n-Tetracosane-d50	16416-32-3	1000000	mg/Kg

Reviewed By: 8/19/12 Date

SERAS-017-DTM-011413_3

0227

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RSF0122

Description:	Neat 5a-Androstane	Expires:	12/27/19 0
Standard Type:	Reagent	Prepared:	06/30/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:25 by JS
Vendor:	Sigma	LotNumber:	098K4035

1 gram of neat Oil Surrogate cmpd. CAT# A0887-1G

Analyte	CAS Number	Concentration	Units
5a-Androstane	438-22-2	1000000	mg/Kg

Reviewed By

Date

SERAS-017-DTM-011413_3

0228

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RSF0093

Description:	Neat Anthracene-d10	Expires:	12/26/19 0
Standard Type:	Reagent	Prepared:	06/29/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:28 by JS
Vendor:	Isotec	LotNumber:	IY0212

1 gram of neat oil surrogate compound, CAT# 176591-1G

Analyte	CAS Number	Concentration	Units
Anthracene-d10	1719-06-8	1000000	ug/mL

Reviewed By 3/19/12 Date

SERAS-017-DTM-011413_3

0229

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RSF0092

Description:	Neat o-Terphenyl-d14	Expires:	12/26/19 0
Standard Type:	Reagent	Prepared:	06/29/09 0
Solvent:	Neat	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:28 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	PR-19171

1 gram, neat oil surrogate compound, cat# DLM-450-1

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	NA	1000000	mg/Kg

Reviewed By 3/19/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RSF0090

Description:	Neat Triacontane-d62	Expires:	06/26/19 0
Standard Type:	Reagent	Prepared:	06/29/09 0
Solvent:	neat	Prepared By:	John Syslo
Final Volume (mls):	0.5	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/30/09 13:27 by JS
Vendor:	Cambridge Isotope Labs	LotNumber:	P-8790

Neat TPH/Oil surrogate, 0.5grams, Cat# DLM-2210-0.5

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1000000	mg/Kg

Reviewed By 3/19/13 Date

SERAS-017-DTM-011413_3

0231

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVA0051

Description:	Carbon Disulfide - Solvent	Expires:	07/25/12 0
Standard Type:	Reagent	Prepared:	01/27/12 0
Solvent:	Carbon Disulfide 99.9%	Prepared By:	John Syslo
Final Volume (mls):	100	Department:	Carbon/XAD
Vials:	1	Last Edit:	03/19/12 10:08 by JS
Vendor:	Sigma Aldrich	LotNumber:	MKBB0780

This bottle has 1.34ppm toluene!!!

Carbon Disulfide for carbon tube extraction. COA not available. This is one of about 20 bottles that were recieved 7/2009 , kept in

Analyte	CAS Number	Concentration	Units
Carbon Disulfide	75-15-0	1000000	ug/mL

Reviewed By _____ Date 3/20/12

SERAS-017-DTM-011413_3

0232

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FINGERPRINTS

Stds. used

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RVB0058

Description:	500ppm Oil IS Mix	Expires:	05/14/12 0
Standard Type:	Internal Standar	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 15:13 by JS
Vendor:	SERAS	LotNumber:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RUK0079) and 2000ppm BNA IS (RUK0081) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUK0079	TPH IS Mix: 2000ppm : 3 Compo	11/14/11	John Syslo	05/14/12	11/14/11 17:00 by JS	0.5
RUK0081	Stock SVOC Internal Std. Mix, 2	011/15/11	John Syslo	05/15/12	11/15/11 15:06 by JS	0.5

Reviewed By 3/13/12 Date

SERAS-017-DTM-011413_3

0234

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RVB0025

Description:	50 ppm DFTPP + ISTD	Expires:	08/12/12 0
Standard Type:	Calibration Stan	Prepared:	02/14/12 0
Solvent:	DE507	Prepared By:	Ben Beauchaine
Final Volume (mls):	20	Department:	SVOCGCMS
Vials:	1	Last Edit:	02/14/12 11:55 by bb
Vendor:	--	LotNumber:	-

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	40	ug/mL
Perylene-d12	NA	40	ug/mL
Pentachlorophenol	87-86-5	50	ug/mL
Naphthalene-d8	NA	40	ug/mL
Decafluorotriphenylphosphine	NA	50	ug/mL
Chrysene-d12	NA	40	ug/mL
Benzidine	92-87-5	50	ug/mL
Acenaphthene-d10	NA	40	ug/mL
4,4'-DDT	50-29-3	50	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	40	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUA0036	GC/MS Tuning Mix (DFTPP), Sto	01/14/11	Ben Beauchaine	07/01/13	06/02/11 14:45 by JS	1
RUJ0028	SV Internal Std. Mix, 2000 ppm	10/27/11	Ben Beauchaine	07/01/18	10/27/11 17:16 by bb	0.4

Reviewed By 3/13/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RVB0003

Description:	1000ppm DRO + 10ppm Surr TPH ICAL	Expires:	08/02/12 0
Standard Type:	Calibration Stan	Prepared:	02/02/12 0
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/02/12 15:04 by JS
Vendor:	SERAS	LotNumber:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	10	ug/mL
Terphenyl-d14	NA	10	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0103	#2 Diesel Fuel Mix 50,000 ppm	10/20/09	John Syslo	04/18/19	02/02/12 14:52 by JS	0.02
RUK0077	2000ppm Oil Surrogate Stock	11/14/11	John Syslo	05/12/12	11/14/11 16:50 by JS	0.005
RUK0080	500ppm Oil IS Mix	11/14/11	John Syslo	02/14/12	11/14/11 17:04 by JS	0.02

Reviewed By 3/14/12 Date

Analytical Standard Record

ERT/SERAS Laboratory

RUL0003

Description:	10K CG-117 Fingerprint Reference	Expires:	05/24/12 0
Standard Type:	Reagent	Prepared:	02/24/12 0
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	02/24/12 19:13 by JS
Vendor:	-	LotNumber:	NA

Diluting neat product to obtain 10,000ppm standard. adding IS for SIM method.

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUL0002	10K CG-117 Fingerprint Reference	12/01/11	John Syslo	05/29/12	12/01/11 16:25 by JS	1
RVB0058	500ppm Oil IS Mix	02/24/12	John Syslo	05/14/12	02/24/12 15:13 by JS	0.02

Reviewed By 3/13/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUK0080

Description:	500ppm Oil IS Mix	Expires:	02/14/12 0
Standard Type:	Internal Standar	Prepared:	11/14/11 0
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	11/14/11 17:04 by JS
Vendor:	SERAS	LotNumber:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RUK0079) and 2000ppm BNA IS (RUH0041) to 2.0mL
spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUH0041	SV Internal Std. Mix, 2000 ppm	08/18/11	John Syslo	02/14/12	09/15/11 15:15 by JS	0.5
RUK0079	TPH IS Mix: 2000ppm : 3 Compo	11/14/11	John Syslo	05/14/12	11/14/11 17:00 by JS	0.5

Reviewed By 3/13/12 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUK0077

Description:	2000ppm Oil Surrogate Stock	Expires:	05/12/12 0
Standard Type:	Surrogate Spike	Prepared:	11/14/11 0
Solvent:	DCM/CS2 75:25	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	11/14/11 16:50 by JS
Vendor:	REAC	LotNumber:	REAC

Mix of 4 neat surrogates for Oil Surrogate Stock. 0.1g each

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2000	ug/mL
Terphenyl-d14	NA	2000	ug/mL
Anthracene-d10	1719-06-8	2000	ug/mL
5a-Androstane	438-22-2	2000	ug/mL

Parent Standards used in this standard

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0090	Neat Triacontane-d62	06/29/09	John Syslo	06/26/19	06/30/09 13:27 by JS	0.1
RSF0092	Neat o-Terphenyl-d14	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0093	Neat Anthracene-d10	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0122	Neat 5a-Androstane	06/30/09	John Syslo	12/27/19	06/30/09 13:25 by JS	0.1

Reviewed By J Syslo 3/13/12 Date

Analytical Standard Record

ERT/SERAS Laboratory

RUC0022

Description:	DCM/Hexane Mix 65/35	Expires:	03/11/21 0
Standard Type:	Reagent	Prepared:	03/11/11 0
Solvent:	DCM/DB241 & Hexane/H49E38	Prepared By:	John Syslo
Final Volume (mls):	1000	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	03/07/12 18:23 by JS
Vendor:	SERAS	LotNumber:	See Solvent/Solvent Lot

DCM/Hexane solvent mixture used to prepare BNA ICAL standards: mix 650mL DCM + 350mL Hexane
JS

Analyte	CAS Number	Concentration	Units
Hexane	110-54-3	332500	ug/mL
Dichloromethane	75-09-2	650000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUC0021	Hexanes (95% n-hexane)	03/10/11	John Syslo	03/10/21	03/07/12 18:22 by JS	350
RUC0026	Dichloromethane 99.9% Solvent	03/11/11	John Syslo	03/11/21	03/11/11 14:54 by JS	650

Reviewed By: J Syslo Date: 3/13/12

US EPA ARCHIVE DOCUMENT

Analytical Standard Record
ERT/SERAS Laboratory
RTL0035

Description:	10ppm SHC/PAH Std.	Expires:	04/18/11 0
Standard Type:	Calibration Stan	Prepared:	12/17/10 0
Solvent:	DCM/Hexane 65/35	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	06/01/11 19:02 by JS
Vendor:	SERAS	LotNumber:	RTI0056

Dilute RTL0034 10x and add 20µL of RTK0041 Oil IS; For Characterization / PAH ICAL

Analyte	CAS Number	Concentration	Units
n-Dotriacontane	544-85-4	10	ug/mL
Dibenzothiophene	132-65-0	10	ug/mL
Fluoranthene	206-44-0	10	ug/mL
Fluorene	86-73-7	10	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	10	ug/mL
n-Decane	124-18-5	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL
n-Dodecane	112-40-3	10	ug/mL
Chrysene	218-01-9	10	ug/mL
n-Eicosane	112-95-8	10	ug/mL
n-Heneicosane	629-94-7	10	ug/mL
n-Hentriacontane	630-04-6	10	ug/mL
n-Heptacosane	593-49-7	10	ug/mL
n-Heptadecane	629-78-7	10	ug/mL
n-Heptatriacontane	7194-84-5	10	ug/mL
n-Docosane	629-97-0	10	ug/mL
Benzo (g,h,i) perylene	191-24-2	10	ug/mL
2-methylanthracene	613-12-7	10	ug/mL
2-Methylnaphthalene	91-57-6	10	ug/mL
5a-Androstane	438-22-2	10	ug/mL
Acenaphthene	83-32-9	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
Acenaphthylene	208-96-8	10	ug/mL
Dibenz(a,h)anthracene	53-70-3	10	ug/mL
Anthracene-d10	1719-06-8	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Benzo (k) fluoranthene	207-08-9	10	ug/mL
Benzo [e] pyrene	192-97-2	10	ug/mL
Benzo(a)anthracene	56-55-3	10	ug/mL

Reviewed By 3/12/12 Date

US EPA ARCHIVE DOCUMENT

Benzo(a)pyrene	50-32-8	10	ug/mL
Benzo(b)fluoranthene	205-99-2	10	ug/mL
n-Hexatriacontane	630-06-8	10	ug/mL
Anthracene	120-12-7	10	ug/mL
Perylene-d12	NA	10	ug/mL
n-Hexacosane	630-01-3	10	ug/mL
n-Tricosane	638-67-5	10	ug/mL
n-Tridecane	629-50-5	10	ug/mL
n-Tritriacontane	630-05-7	10	ug/mL
n-Undecane	1120-21-4	10	ug/mL
Naphthalene	91-20-3	10	ug/mL
n-Tetratriacontane	14167-59-0	10	ug/mL
Perylene	198-55-0	10	ug/mL
n-Tetradecane-d30		10	ug/mL
Phenanthrene	85-01-8	10	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Phytane	638-36-8	10	ug/mL
Pristane	1921-70-6	10	ug/mL
Pyrene	129-00-0	10	ug/mL
Terphenyl-d14	NA	10	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Octatriacontane	7194-85-6	10	ug/mL
Triacontane-d62	93952-07-9	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
n-Nonacosane	630-03-5	10	ug/mL
n-Nonadecane	629-92-5	10	ug/mL
n-Nonane	111-84-2	10	ug/mL
n-Octacosane	630-02-4	10	ug/mL
n-Triacontane	638-68-6	10	ug/mL
n-Octane	111-65-9	10	ug/mL
n-Hexadecane	544-76-3	10	ug/mL
n-Pentacosane	629-99-2	10	ug/mL
n-Pentadecane	629-62-9	10	ug/mL
n-Pentatriacontane	630-07-9	10	ug/mL
n-Tetracosane	646-61-1	10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Tetradecane	629-59-4	10	ug/mL
n-Octadecane	593-45-3	10	ug/mL

Reviewed By _____ Date 3/12/12

US EPA ARCHIVE DOCUMENT

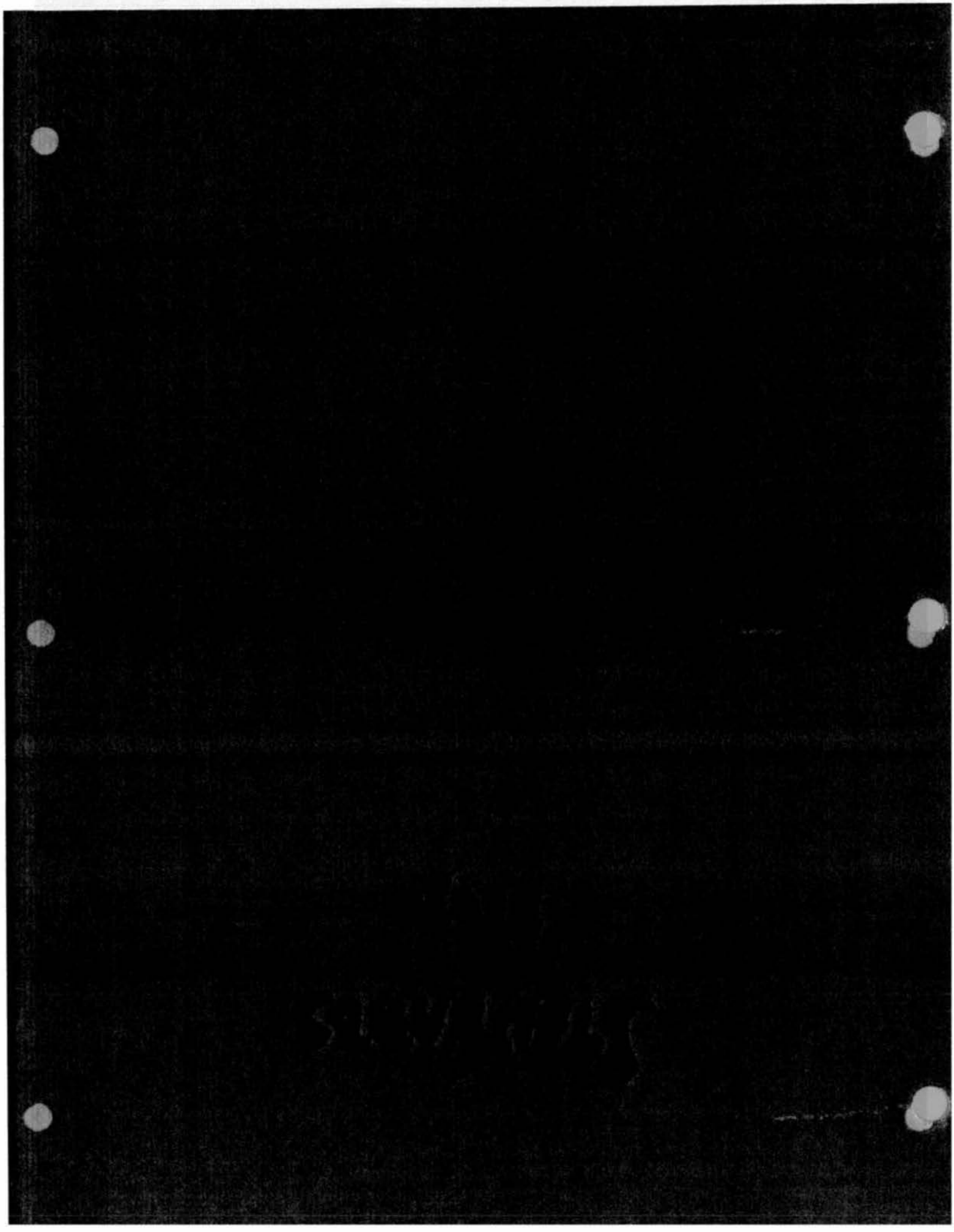
Analytical Standard Record
ERT/SERAS Laboratory
RTL0035

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RTK0041	500ppm Oil IS Mix	11/16/10	John Syslo	04/18/11	06/01/11 19:02 by JS	0.02
RTL0034	100ppm SHC/PAH Stock	12/17/10	John Syslo	04/18/11	12/17/10 18:42 by JS	0.1

US EPA ARCHIVE DOCUMENT

Reviewed By _____ Date 3/12/12



For ICAL

Surrogate

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0050

Description:	10,000ppm 10W40 Oil	Expires:	11/12/11
Standard Type:	Reagent	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 19:03 by JS
Vendor:	SERAS	Lot Number:	NA

Dilute 20K Stock to 10,oooppm and add IS: No Surrogates

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Motor Oil	NA	10000	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RTK0021	SAE 10W40 Motor Oil 20K	11/12/10	John Syslo	01/30/19	11/19/10 20:42 by JS	0.5
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02

Reviewed By J. Syslo 5/16/11 Date

0246

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0049

Description:	1000ppm DRO/TPH ICV	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 18:16 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	B90302060

dilute 2nd Source Stock 50x and add IS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0107	#2 Diesel Fuel LCS 50,000ppm	10/20/09	John Syslo	10/15/19	10/20/09 13:52 by JS	0.02
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02

Reviewed By J. Syslo Date 5/16/11

0247

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0048

Description:	50ppm DRO + 0.5ppm Surr TPH ICAL	Expires:	11/12/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 18:11 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:
Used 25µL syringe to measure 2.5µL ; not 10µL syringe (RUE0047)

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	0.5	ug/mL
Terphenyl-d14	NA	0.5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	0.5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	0.5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.0025

Reviewed By: _____ Date: 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0047

Description:	50ppm DRO + 0.5ppm Surr TPH ICAL	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 17:50 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	0.5	ug/mL
Terphenyl-d14	NA	0.5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		50	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	0.5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	0.5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.0025

Reviewed By J. Syslo 5/16/11 Date

0249

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0046

Description:	100ppm DRO + 1.0ppm Surr TPH ICAL	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 17:49 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	1	ug/mL
Terphenyl-d14	NA	1	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		100	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	1	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	1	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.005

Reviewed By _____ Date 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0045

Description:	500ppm DRO + 5.0ppm Surr TPH ICAL	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:20 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	5	ug/mL
Terphenyl-d14	NA	5	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		500	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	5	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	5	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.025

Reviewed By 5/16/11 Date

0251

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0044

Description:	1000ppm DRO + 10ppm Surr TPH ICAL	Expires:	11/12/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:18 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	10	ug/mL
Terphenyl-d14	NA	10	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		1000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	10	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	10	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.05

Reviewed By _____ Date 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0043

Description:	5k DRO + 50ppm Surr Cal.	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:19 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	50	ug/mL
Terphenyl-d14	NA	50	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		5000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	50	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	50	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.25

Reviewed By: [signature] Date: 5/16/11

0253

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0042

Description:	10k DRO + 100ppm Surr Cal.	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	1	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:15 by JS
Vendor:	SERAS	Lot Number:	NA

Serial dilution of RUE0041 + 20µL IS (RUE0037) TPH ICAL:

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	100	ug/mL
Terphenyl-d14	NA	100	ug/mL
Phenanthrene-d10	NA	10	ug/mL
Perylene-d12	NA	10	ug/mL
Oil Fingerprint		10000	ug/mL
Naphthalene-d8	NA	10	ug/mL
n-Tetradecane-d30		10	ug/mL
n-Tetracosane-d50	16416-32-3	10	ug/mL
n-Hexatriacontane-d74	16416-34-5	10	ug/mL
Chrysene-d12	NA	10	ug/mL
Anthracene-d10	1719-06-8	100	ug/mL
Acenaphthene-d10	NA	10	ug/mL
5a-Androstane	438-22-2	100	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0037	500ppm Oil IS Mix	05/13/11	John Syslo	11/13/11	05/13/11 15:33 by JS	0.02
RUE0041	20K DRO & 200ppm Surr. ICAL S	05/16/11	John Syslo	11/13/11	05/16/11 16:04 by JS	0.5

Reviewed By J. Syslo Date 5/16/11

0254

SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0041

Description:	20K DRO & 200ppm Surr. ICAL Stock	Expires:	11/13/11
Standard Type:	Calibration Stan	Prepared:	05/16/11
Solvent:	DCM/Hexane 65/35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/16/11 16:04 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	A9070168

Primary Source Diesel + Surrogate mixture for DRO/TPH ICAL. Make serial dilutions and add IS: JS

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	200	ug/mL
Terphenyl-d14	NA	200	ug/mL
Oil Fingerprint		20000	ug/mL
Anthracene-d10	1719-06-8	200	ug/mL
5a-Androstane	438-22-2	200	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0102	#2 Diesel Fuel Mix 50,000 ppm	10/20/09	John Syslo	04/18/19	05/13/11 16:18 by JS	0.8
RUE0036	2000ppm Oil Surrogate Stock	05/13/11	John Syslo	11/13/11	05/13/11 15:13 by JS	0.2

Reviewed By J. Syslo Date 5/16/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0040

Description:	2000ppm TPH (DRO) LCS Spike (2nd)	Expires:	11/13/11
Standard Type:	Analyte Spike	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35 (RUC0022)	Prepared By:	John Syslo
Final Volume (mls):	20	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 17:09 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	B9030206 (2nd source diesel)

Dilute 50K 2nd source Diesel Standard 25x with DCM/Hexane :
= 800µL Rsj0107 into FV 20.0mL DCM/Hexane (RUC0022) = 2000ppm

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0107	#2 Diesel Fuel LCS 50,000ppm	10/20/09	John Syslo	10/15/19	10/20/09 13:52 by JS	0.8

Reviewed By 5/13/11 Date

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0039

Description:	400ppm DRO/TPH MDL Spike	Expires:	11/13/11
Standard Type:	Analyte Spike	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	20	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 16:22 by JS
Vendor:	AccuStandard,Inc.	Lot Number:	A9070168 for DRO

Dilute 160µL of 50,000ppm into 20mL DCM/Hexane

Analyte	CAS Number	Concentration	Units
Oil Fingerprint		400	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSJ0102	#2 Diesel Fuel Mix 50,000 ppm	10/20/09	John Syslo	04/18/19	05/13/11 16:18 by JS	0.16

Reviewed By _____ Date 5/13/11

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0038

Description:	40ppm Oil Surrogate	Expires:	11/13/11
Standard Type:	Surrogate Spike	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	25	Department:	CENTRAL
Vials:	1	Last Edit:	05/13/11 15:42 by JS
Vendor:	SERAS	Lot Number:	RUC00226

Dilute 2000ppm RUE0036 (500µL in 25mL) with DCM/Hexane
Spike 500 µL per sample for FV of 1.0 mL

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	40	ug/mL
Terphenyl-d14	NA	40	ug/mL
Anthracene-d10	1719-06-8	40	ug/mL
5a-Androstane	438-22-2	40	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RUE0036	2000ppm Oil Surrogate Stock	05/13/11	John Syslo	11/13/11	05/13/11 15:13 by JS	0.5

Reviewed By _____ Date 5/13/11

0258

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0037

Description:	500ppm Oil IS Mix	Expires:	11/13/11
Standard Type:	Internal Standar	Prepared:	05/13/11
Solvent:	DCM/Hexane 65:35	Prepared By:	John Syslo
Final Volume (mls):	2	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 15:33 by JS
Vendor:	SERAS	Lot Number:	DCM/Hex = LIMS# RUC0022

Dilute 2000ppm TPH IS (RUE0035) and 2000ppm BNA IS (RTF0070) to 2.0mL spike 20µL per 1.0mL extract. JS

Analyte	CAS Number	Concentration	Units
Phenanthrene-d10	NA	500	ug/mL
Perylene-d12	NA	500	ug/mL
Naphthalene-d8	NA	500	ug/mL
n-Tetradecane-d30		500	ug/mL
n-Tetracosane-d50	16416-32-3	500	ug/mL
n-Hexatriacontane-d74	16416-34-5	500	ug/mL
Chrysene-d12	NA	500	ug/mL
Acenaphthene-d10	NA	500	ug/mL
1,4-Dichlorobenzene-d4	3855-82-1	500	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RTF0070	Stock SVOC Internal Std. Mix, 2	06/16/10	** Vendor **	11/05/11	05/05/11 12:51 by JS	0.5
RUE0035	TPH IS Mix: 2000ppm : 3 Compo	05/13/11	John Syslo	11/13/11	05/13/11 15:10 by JS	0.5

Reviewed By 5/13/11 Date

0259

US EPA ARCHIVE DOCUMENT

Analytical Standard Record

ERT/SERAS Laboratory

RUE0036

Description:	2000ppm Oil Surrogate Stock	Expires:	11/13/11
Standard Type:	Surrogate Spike	Prepared:	05/13/11
Solvent:	DCM/CS2 75:25	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 15:13 by JS
Vendor:	REAC	Lot Number:	REAC

Mix of 4 neat surrogates for Oil Surrogate Stock. 0.1g each

Analyte	CAS Number	Concentration	Units
Triacontane-d62	93952-07-9	2000	ug/mL
Terphenyl-d14	NA	2000	ug/mL
Anthracene-d10	1719-06-8	2000	ug/mL
5a-Androstane	438-22-2	2000	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0090	Neat Triacontane-d62	06/29/09	John Syslo	06/26/19	06/30/09 13:27 by JS	0.1
RSF0092	Neat o-Terphenyl-d14	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0093	Neat Anthracene-d10	06/29/09	John Syslo	12/26/19	06/30/09 13:28 by JS	0.1
RSF0122	Neat 5a-Androstane	06/30/09	John Syslo	12/27/19	06/30/09 13:25 by JS	0.1

Reviewed By 5/13/11 Date

0260

Analytical Standard Record

ERT/SERAS Laboratory

RUE0035

Description:	TPH IS Mix: 2000ppm : 3 Compounds	Expires:	11/13/11
Standard Type:	Internal Standar	Prepared:	05/13/11
Solvent:	DCM/CS2 4:1	Prepared By:	John Syslo
Final Volume (mls):	50	Department:	Oil/Fingerprint Lab
Vials:	1	Last Edit:	05/13/11 15:10 by JS
Vendor:	NA	Lot Number:	NA

2000ppm 3 Compound IS Mix for TPH. To be mixed with BNA IS

Analyte	CAS Number	Concentration	Units
n-Tetradecane-d30		2000	mg/Kg
n-Tetracosane-d50	16416-32-3	2000	mg/Kg
n-Hexatriacontane-d74	16416-34-5	2000	mg/Kg

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
RSF0123	Neat D50-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 14:26 by JS	0.1
RSF0124	Neat D74-n-Hexatriacontane	06/30/09	John Syslo	12/27/19	06/30/09 14:33 by JS	0.1
RSF0126	Neat D30-n-Tetradecane	06/30/09	John Syslo	12/27/19	06/30/09 13:56 by JS	0.1

Reviewed By J Syslo Date 5/13/11

US EPA ARCHIVE DOCUMENT

Mariner's Marsh: 1816 Enbridge Oil: 1803 Logbook # SERAS-I-0121

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 ARQTPH05716II.M

Project Name: Mariner Marsh + Enbridge Oil Method File: DFTPP CS110711.M

Work Assign.#: 9-170 9-017 Inj. Volume: 2ul

Analysis/Date: Carbon Cube VOC TPH Analyst: [illegible]

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
21	SL02655	STATION #1 F	20)006-22	2/3/12	00:59	AA-004-013112-004
22	SL 56	#1 B	-22		1:19	1-001- -004
23	SL 57	STATION #2 F	-23		1:39	-002- -005
24	SL 58	#2 B	-23		1:59	-002- -005
25	SL 59	STATION #3 F	-24		2:20	-003- -004
26	SL 60	#3 B	-24		2:40	-003- -004
27	SL 61	STATION #4 F	-25		3:00	-004- -004
28	SL 62	#4 B	-25		3:20	-004- -004
29	SL	STATION #5 F	-26		3:40	-005 -004
30	SL 64	#5 B	-26		4:00	AA-005-013112-004
31	SL 65	FB-013112 F	-27		4:20	Field Blank
32	SL 66	" B	-27		4:40	
33	SL 67	FB-013112 F	-28		5:00	Field Blank
[illegible]		FB-013112 B		2/3/12	5:40	
[illegible]	SL	— end of Mariner Marsh on 2/2/12 —			2/02/12	
[illegible]						
69	SL02670	50ppm DFTPP	RVK0084		15:49	PASS DM2-1590
71	SL02671	100ppm TPH CCV	RVB0003		16:23	Pass - ok
80	SL02672	Soil Blank	12 0003 Blk		17:18	
[illegible]	SL02673	[illegible]	[illegible]		[illegible]	[illegible]
82	SL02674	SEAN-017-0001	" "		19:53	
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]
	SL	— End of Enbridge Oil on 2/3/12 —			2/3/12	
	SL					
	SL					
	SL					
	SL					
	SL					

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVK0084	DFTPP	5/5/12	50 ppm		
2	RVK0039	Calibration Check	2/25/12	50 ppm		
3	RSP0112	Internal Standard	3/8/12	0.50 ppm		Spike 20ml + 1.0ml / 10+.5
4	RSC0154	BS/MSD Spike	7/20/12	5000 ppm		Spike 10ml + tube
5	RVA0052	BS Extraction Sol	7/26/12			
6						

Reviewed By: [signature] Date Checked: 2/3/12

Analyses by: [signature] 2/3/12

US EPA ARCHIVE DOCUMENT

Sequence Name: C:\msdchem\1\sequence\020212.s
Comment: Enbridge Oil
Operator: Syslo
Data Path: C:\MSDCHEM\1\DATA\020212\
Instrument Control Pre-Seq Cmd:
Data Analysis Pre-Seq Cmd:

Instrument Control Post-Seq Cmd:
Data Analysis Post-Seq Cmd:

Method Sections To Run
(X) Full Method
( ) Reprocessing Only

On A Barcode Mismatch
(X) Inject Anyway
( ) Don't Inject

Line		Sample Name/Misc Info
1) Sample	99	~~SL02669 DFT8270D 50 DFTPP~~
2) Sample	99	SL02670 DFT8270D 50 DFTPP
3) Sample	71	1.0k TPH CCV
Datafile		SL02671
Method		DROTPH051611
4) Sample	80	Blank
Datafile		SL02672
Method		DROTPH051611
5) Sample	81	~~SERAS-017-0001 5xd~~
Datafile		SL02673
Method		DROTPH051611
6) Sample	82	SERAS-017-0001
Datafile		SL02674
Method		DROTPH051611
7) Sample	80	Blank
Datafile		SL02675
Method		DROTPH051611
8) Sample	81	SL02676 OILLINS2 SERAS-017-0001 {L}

For Is check + Surr

not need

GC/MS QA-QC Check Report 2/02/12

Tune File : C:\msdchem\1\DATA\020212\SL02670.D
Tune Time : 2 Feb 2012 15:44

Daily Calibration File : C:\msdchem\1\DATA\020212\SL02671.D

File	Sample	Surrogate Recovery %				Internal Standard Responses		
SL02671.D								
	1.0k TPH C	51*	55*	59* 153567	58*	505006	175782	209958
SL02672.D								
	Blank	63*	70*	75* 110992	93*	385841	133055	149646
SL02674.D								
	SERAS-017-	76*	82*	87* 117011	98*	363912	124892	142419

Created: Wed Jan 02 12:07:11 2013 Slick2

FS area ok
-Surrogate rejected in
TPH report

Injection Log

Data Directory: C:\msdchem\1\DATA\020212\

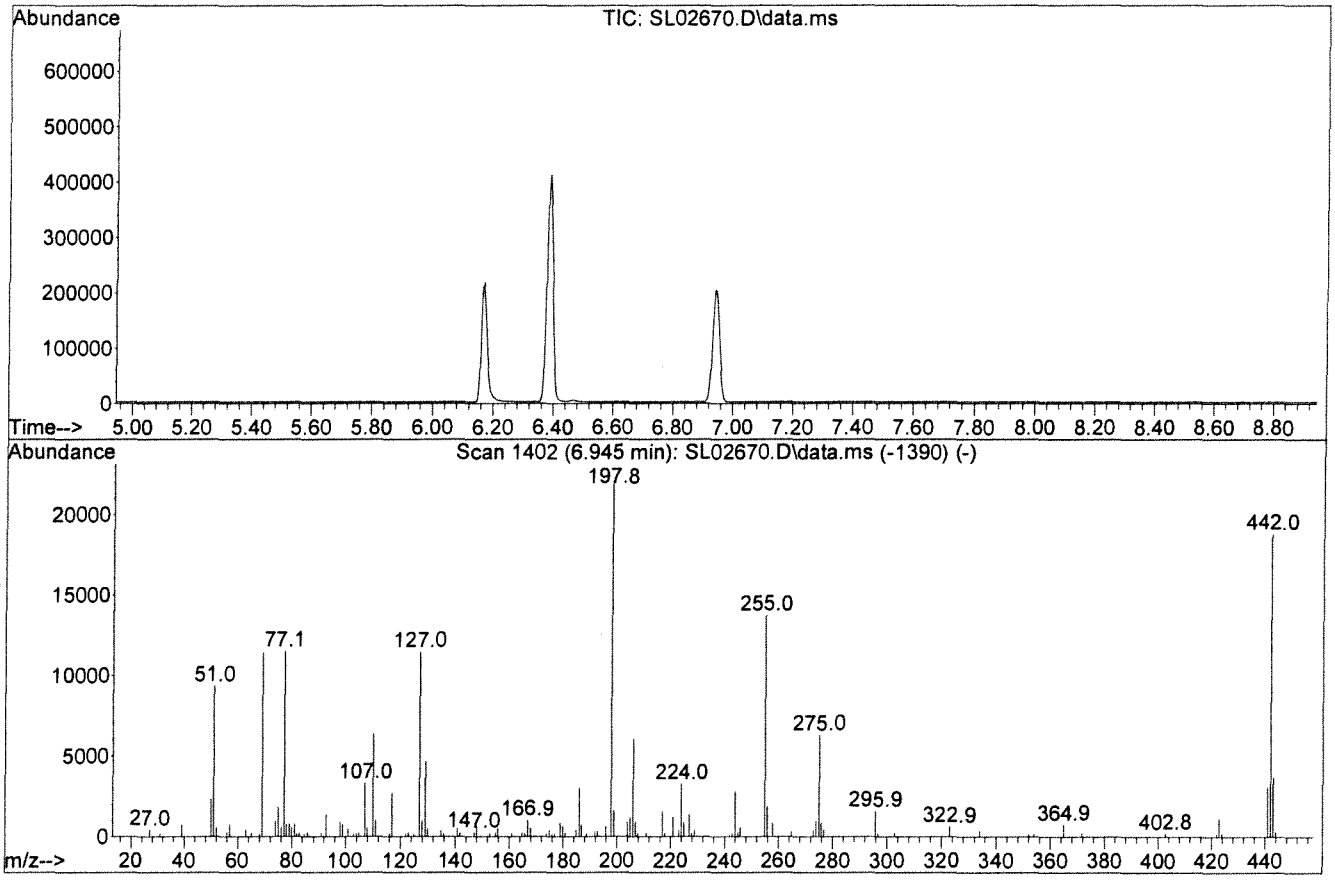
SampleName	MiscInfo	Vial	Multiplier	Injection Time
1) SL02669.D				
50 DFTPP	RUK0084	99	1.000	2 Feb 2012 14:13
2) SL02670.D				
50 DFTPP	RUK0084	99	1.000	2 Feb 2012 15:44
3) SL02671.D				
1.0k TPH CCV	RVB0003	71	1.000	2 Feb 2012 16:23
4) SL02672.D				
Blank	1200012-Blk1	80	1.000	2 Feb 2012 17:18
5) SL02673.D				
SERAS-017-0001 5xd	30g to 1.0mL, then 5	81	1.000	2 Feb 2012 18:43
6) SL02674.D				
SERAS-017-0001	30g to 1.0mL	82	1.000	2 Feb 2012 19:53
7) SL02675.D				
Blank	1200012-Blk1	80	1.000	2 Feb 2012 20:53
8) SL02676.D				
SERAS-017-0001 {L}	Linear Scan 30g to 1	82	1.000	2 Feb 2012 21:54

Tailing: PCP : 1.29
Benzidine: 1.20
2-FBT B.D: 3.20

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02670.D
Acq On : 2 Feb 2012 15:44
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Integration File: rteint.p

Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Title : DFTPP Method with 8270D criteria: 8/20/10
Last Update : Thu Jan 26 14:45:44 2012



Spectrum Information: Scan 1402

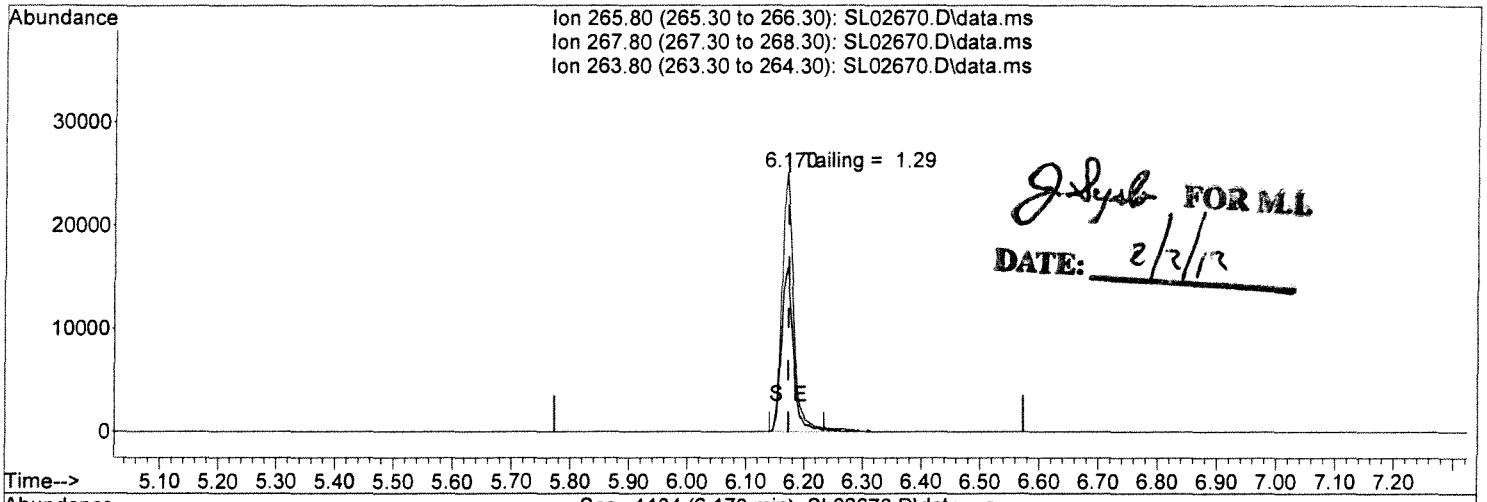
Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	42.4	9351	PASS
68	69	0.00	2	1.7	194	PASS
69	198	0.00	100	51.8	11430	PASS
70	69	0.00	2	0.0	0	PASS
127	198	10	80	52.0	11474	PASS
197	198	0.00	2	0.0	0	PASS
198	198	100	100	100.0	22063	PASS
199	198	5	9	7.2	1586	PASS
275	198	10	60	28.6	6307	PASS
365	198	1	100	3.4	752	PASS
441	442	0.01	24	16.3	3074	PASS
442	198	50	100	85.4	18832	PASS
443	442	15	24	19.5	3679	PASS

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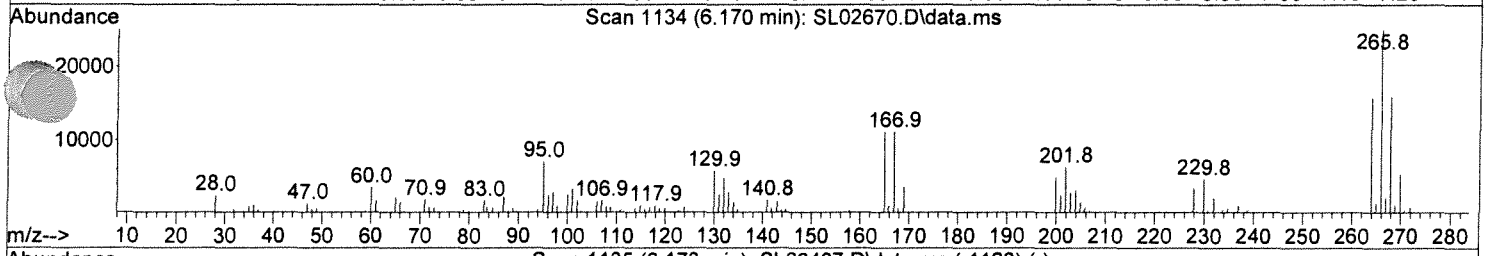
Quantitation Report (Qedit)

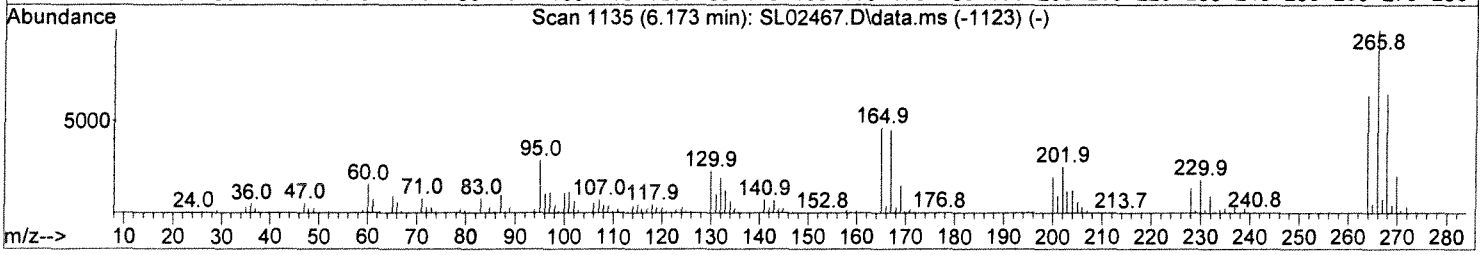
Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02670.D
Acq On : 2 Feb 2012 15:44
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 02 15:57:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



J. Syslo FOR M.L.
DATE: 2/2/12





TIC: SL02670.D\data.ms

(2) Pentachlorophenol

6.170min (-0.003) 45.21 ug/mL

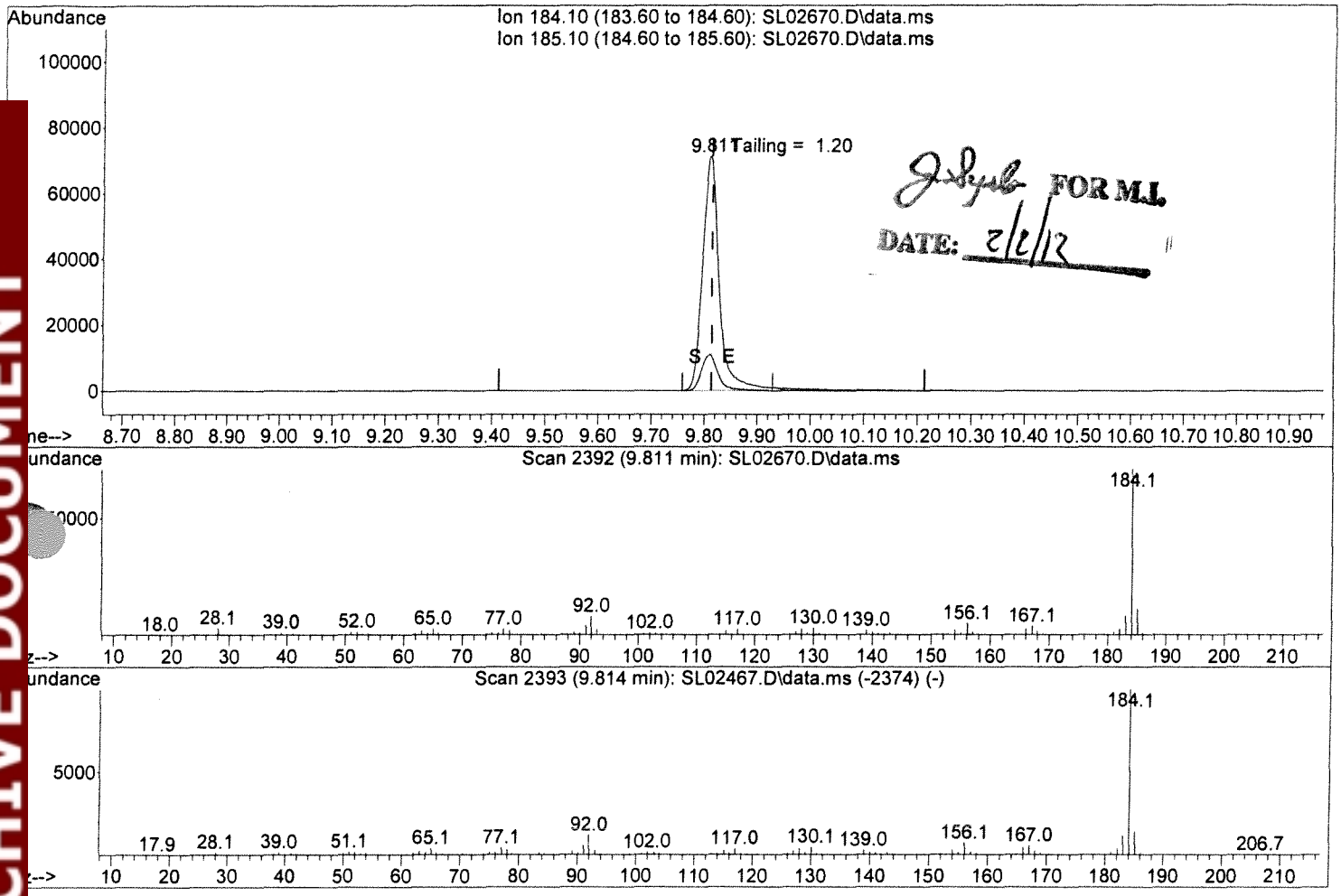
response 34880

Ion	Exp%	Act%
265.80	100	100
267.80	64.50	64.41
263.80	63.60	62.72
0.00	0.00	0.00

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02670.D
Acq On : 2 Feb 2012 15:44
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 02 15:57:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



TIC: SL02670.D\data.ms

(5) Benzidine

9.811min (-0.003) 49.79 ug/mL

response 155668

Ion	Exp%	Act%
184.10	100	100
185.10	14.60	15.12
0.00	0.00	0.00
0.00	0.00	0.00

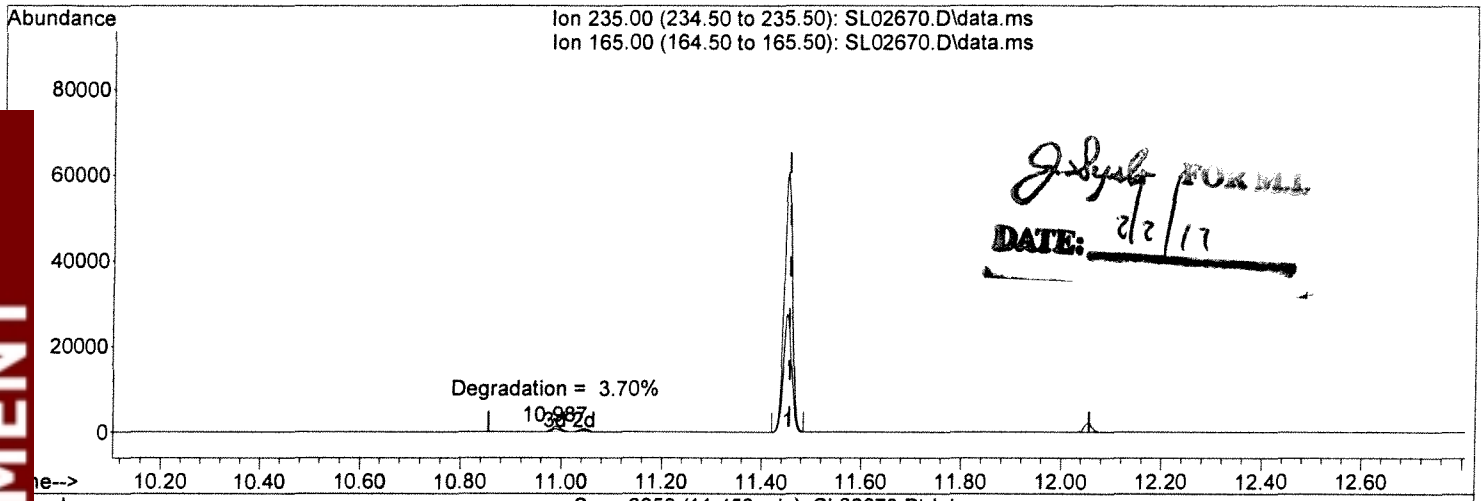
FOR M.L.
DATE: 2/2/12

US EPA ARCHIVE DOCUMENT

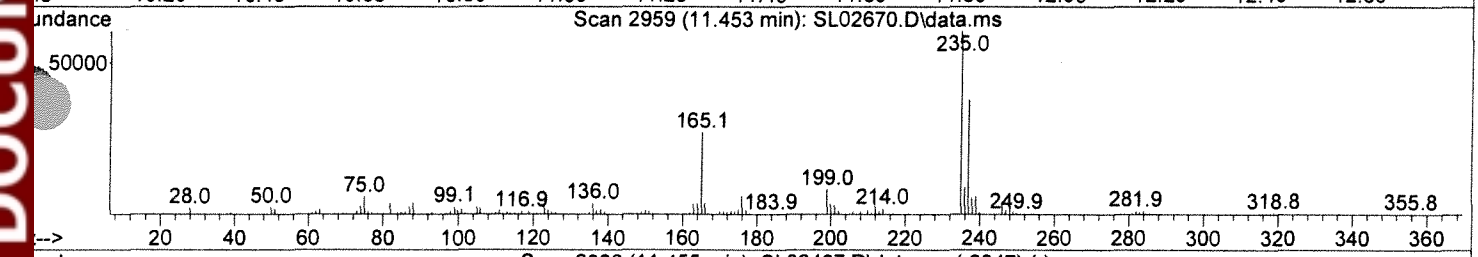
Quantitation Report (Qedit)

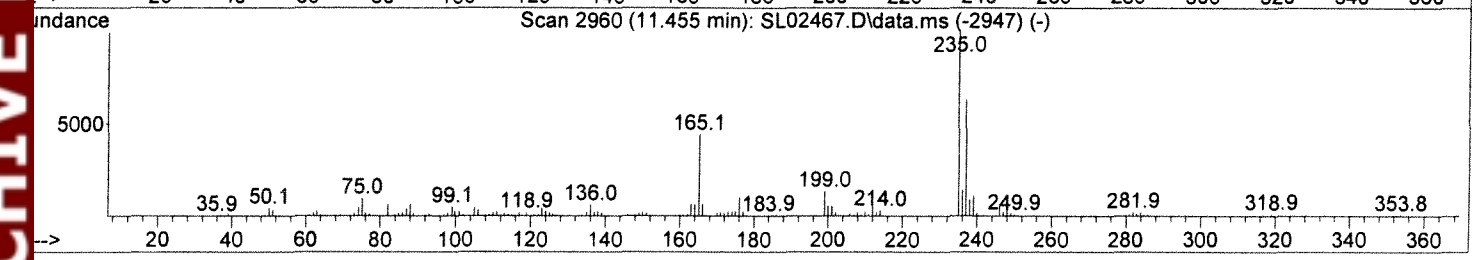
Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02670.D
Acq On : 2 Feb 2012 15:44
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 02 15:57:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



FOR MLL
DATE: 2/2/17





TIC: SL02670.D\data.ms

(6) DDT

11.453min (-0.003) 50.33 ug/mL

response 70001

Ion	Exp%	Act%
235.00	100	100
165.00	51.80	45.18
0.00	0.00	0.00
0.00	0.00	0.00

US EPA ARCHIVE DOCUMENT

TPH Daily Calibration Check Results
#2 Diesel TPH Calibration Range - Using SERAS GC/MS Method 1841

Calibration Check Date	ICAL Date	Project	Wa#	GC/MS Datafile	TPH Response	d30-IS Area	d50-IS Area	d74-IS Area	Cal Check RF	Calibration Average RF	% Diff.
05/17/11	05/16/11	WSoil MDL	0-011	SL01889	7932426	132429	130508	51192	0.75756	0.72959	3.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01942	7860351	128757	120362	11883	0.90348	0.72959	23.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01945	6544592	104943	128978	88104	0.60970	0.72959	16.43
07/08/11	05/16/11	CUC-Power Plant	0-135	SL01970	6848120	109628	127366	77204	0.65387	0.72959	10.38
07/11/11	05/16/11	CUC-Power Plant	0-135	SL01974	7184921	121573	136941	70557	0.65502	0.72959	10.22
07/12/11	05/16/11	CUC-Power Plant	0-135	SL01989	7699927	130026	148583	96240	0.61624	0.72959	15.54
07/27/11	05/16/11	CUC-Power Plant	0-135	SL02012	5526770	90684	107174	65457	0.62968	0.72959	13.69
08/01/11	05/16/11	CUC-Power Plant	0-135	SL02036	7282274	117359	139028	90753	0.62934	0.72959	13.74
08/08/11	05/16/11	Move-Maintenance	0-011	SL02067	20772937	342644	403655	197034	0.66062	0.72959	9.45
08/08/11	05/16/11	Move-Maintenance	0-011	SL02069	9013310	151400	176386	86475	0.65273	0.72959	10.54
08/10/11	05/16/11	Move-Maintenance	0-011	SL02071	8539256	140844	161739	77290	0.67438	0.72959	7.57
08/11/11	05/16/11	TPH Soil MDL	0-011	SL02079	8559600	142237	165518	79165	0.66367	0.72959	9.03
08/12/11	05/16/11	TPH WaterI MDL	0-011	SL02093	8541528	140220	170089	76028	0.66327	0.72959	9.09
08/23/11	05/16/11	CUC-Power Plant	0-135	SL02133	8279388	130044	151992	63182	0.71949	0.72959	1.38
08/24/11	05/16/11	CUC-Power Plant	0-135	SL02166	6757179	110307	122438	62687	0.68617	0.72959	5.95
09/21/11	05/16/11	CUC-Power Plant	0-135	SL02202	11190934	169011	205927	119691	0.67875	0.72959	6.97
09/22/11	05/16/11	CUC-Power Plant	0-135	SL02213	8986654	146134	179078	114918	0.61255	0.72959	16.04
02/02/12	05/16/11	Enbridge Oil	0-017	SL02671	10771363	175782	209958	153567	0.59918	0.72959	17.87
02/15/12	05/16/11	Enbridge Oil	0-017	SL02827	11689228	179024	220786	162003	0.62419	0.72959	14.45
02/24/12	05/16/11	Enbridge Oil	0-017	SL02857	11425492	166909	205857	151555	0.65373	0.72959	10.40
02/27/12	05/16/11	Enbridge Oil	0-017	SL02872	10457816	165200	202882	161132	0.59283	0.72959	18.74
03/01/12	05/16/11	Enbridge Oil	0-017	SL02885	7203855	109515	150757	134865	0.54694	0.72959	25.03

US EPA ARCHIVE DOCUMENT

Evaluate Continuing Calibration Report

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02671.D
Acq On : 2 Feb 2012 16:23
Operator : Syslo
Sample : 1.0k TPH CCV
Misc : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 02 16:56:39 2012
Quant Method : C:\msdchem\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Min. RRF : 0.010 Min. Rel. Area : 50% Max. R.T. Dev 0.50min
Max. RRF Dev : 30% Max. Rel. Area : 200%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	d10-Phenanthrene	1.000	1.000	0.0	105	0.00
2	S	d10-Anthracene {S}	1.063	1.081	-1.7	105	0.00
3	S	d14-o-Terphenyl {S}	0.467	0.510	-9.2	107	0.00
4	S	5a-Androstane {S}	0.057	0.067	-17.5	113	0.00
5	I	d30-Tetradecane	1.000	1.000	0.0	112	0.00
6	I	d50-Tetracosane	1.000	1.000	0.0	115	0.00
7	S	d62-Triacontane {S}	Linear Regression =		-15.0	136	0.00
8	I	d74-Hexatriacontane	1.000	1.000	0.0	194	0.00

(#) = Out of Range SPCC's out = 0 CCC's out = 0

DROTPH051611.M Thu Feb 02 16:57:04 2012 SLICK2

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02671.D
Acq On : 2 Feb 2012 16:23
Operator : Syslo
Sample : 1.0k TPH CCV
Misc : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 02 16:56:39 2012
Quant Method : C:\msdchem\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.722	188	505006	10.00	ug/mL	0.00
5) d30-Tetradecane	13.548	66	175782	10.00	ug/mL	0.00
6) d50-Tetracosane	23.793	66	209958	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.645	66	153567	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.840	188	545660	10.16	ug/mL	0.00
Spiked Amount 20.000			Recovery =	50.80%		
3) d14-o-Terphenyl {S}	19.742	244	257413	10.91	ug/mL	0.00
Spiked Amount 20.000			Recovery =	54.55%		
4) 5a-Androstane {S}	21.159	260	33800	11.77	ug/mL	0.00
Spiked Amount 20.000			Recovery =	58.85%		
7) d62-Triacontane {S}	28.172	66	206090	11.50	ug/mL	0.00
Spiked Amount 20.000			Recovery =	57.50%		
Target Compounds			LR 15%D			Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

Those samples run for TPH
but Fingerprints also requested

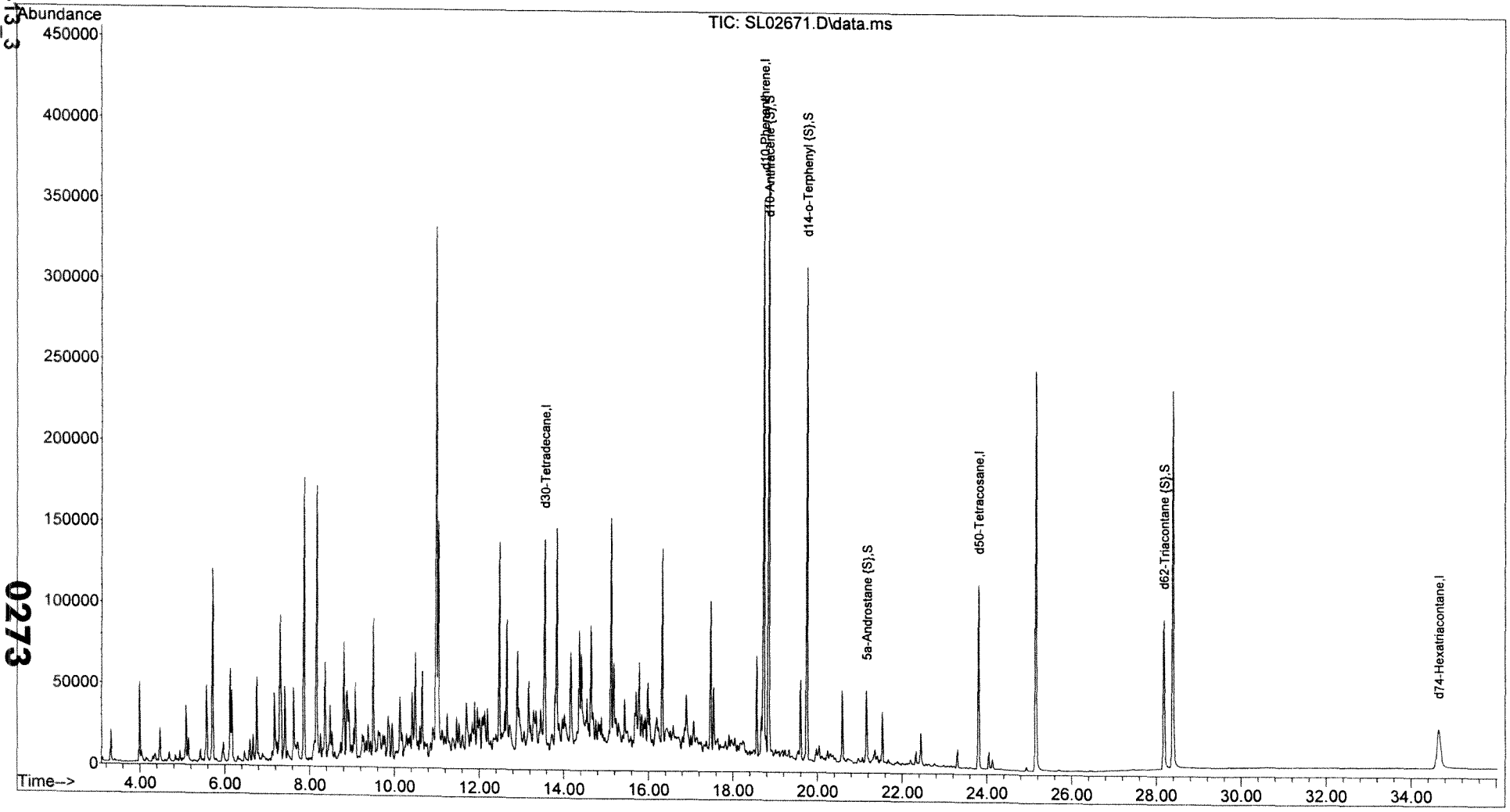
US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02671.D
Acq On : 2 Feb 2012 16:23
Operator : Syslo
Sample : 1.0k TPH CCV
Misc : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 02 16:56:39 2012
Quant Method : C:\msdchem\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration



0273

Area Percent Report

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02671.D
Acq On : 2 Feb 2012 16:23
Operator : Syslo
[illegible]ple : 1.0k TPH CCV
[illegible]c : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 7 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\msdchem\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL02671.D\data.ms

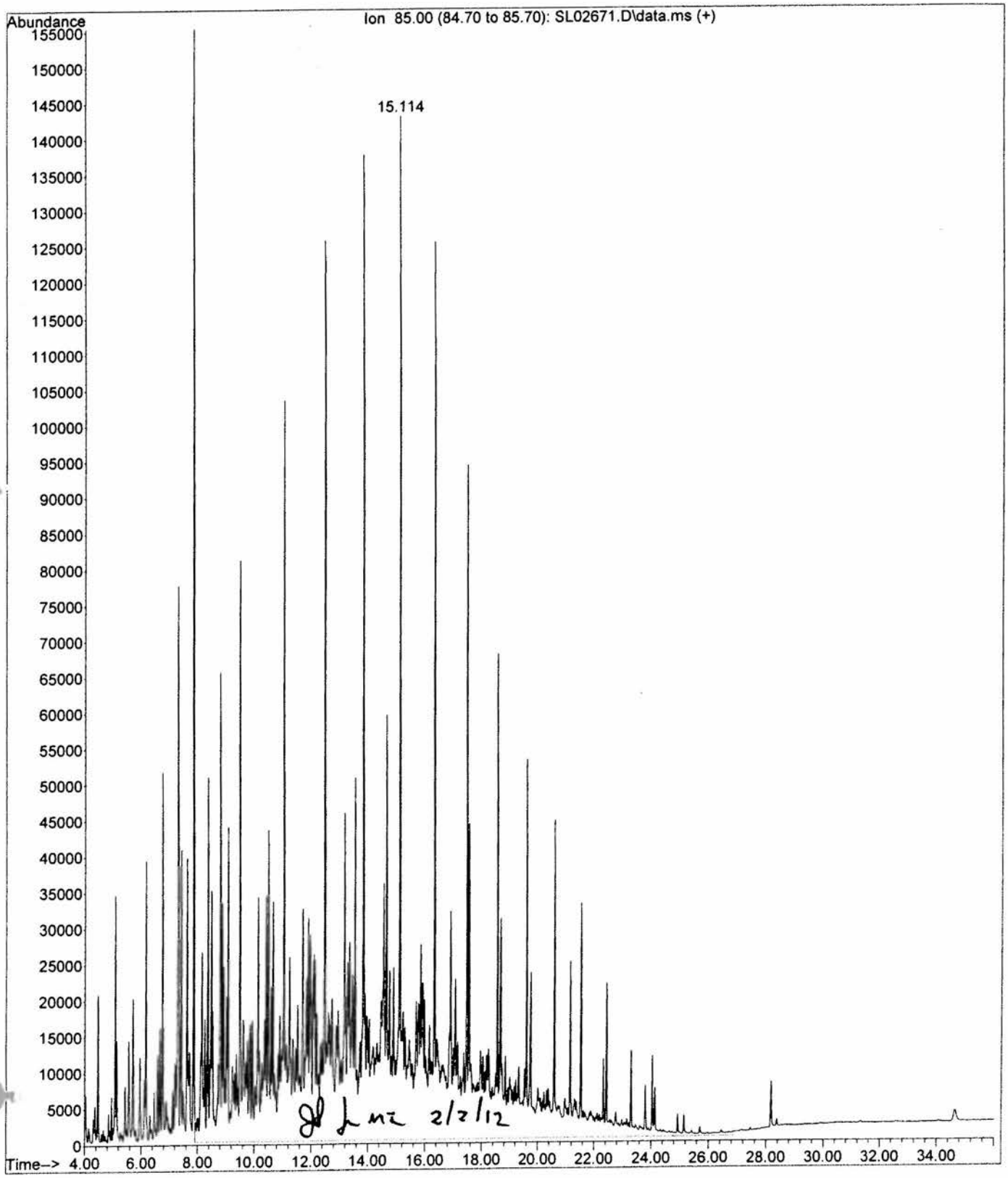
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	15.114	605	1506	2879	rM	10 142688	10771363	100.00%	100.000%

Sum of corrected areas: 10771363

[illegible]PH051611.M Thu Feb 02 17:01:58 2012 SLICK2

Y.D = (7.87)

File :C:\msdchem\1\DATA\020212\SL02671.D
Operator : Syslo
Acquired : 2 Feb 2012 16:23 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 1.0k TPH CCV
Misc Info : RVB0003
Vial Number: 71



JL Lmz 2/2/12

US EPA ARCHIVE DOCUMENT

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02672.D
Acq On : 2 Feb 2012 17:18
Operator : Syslo
Sample : Blank
Misc : 1200012-Blk1
ALS Vial : 80 Sample Multiplier: 1

Quant Time: Feb 02 18:08:13 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.722	188	385841	10.00	ug/mL	0.00
5) d30-Tetradecane	13.548	66	133055	10.00	ug/mL	0.00
6) d50-Tetracosane	23.793	66	149646	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.637	66	110992	10.00	ug/mL	-0.02
System Monitoring Compounds						
2) d10-Anthracene {S}	18.840	188	518432	12.63	ug/mL	0.00
Spiked Amount 20.000			Recovery =	63.15%		
3) d14-o-Terphenyl {S}	19.742	244	251502	13.95	ug/mL	0.00
Spiked Amount 20.000			Recovery =	69.75%		
4) 5a-Androstane {S}	21.159	260	32945	15.02	ug/mL	0.00
Spiked Amount 20.000			Recovery =	75.10%		
7) d62-Triacontane {S}	28.172	66	244793	18.58	ug/mL	0.00
Spiked Amount 20.000			Recovery =	92.90%		

Target Compounds Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

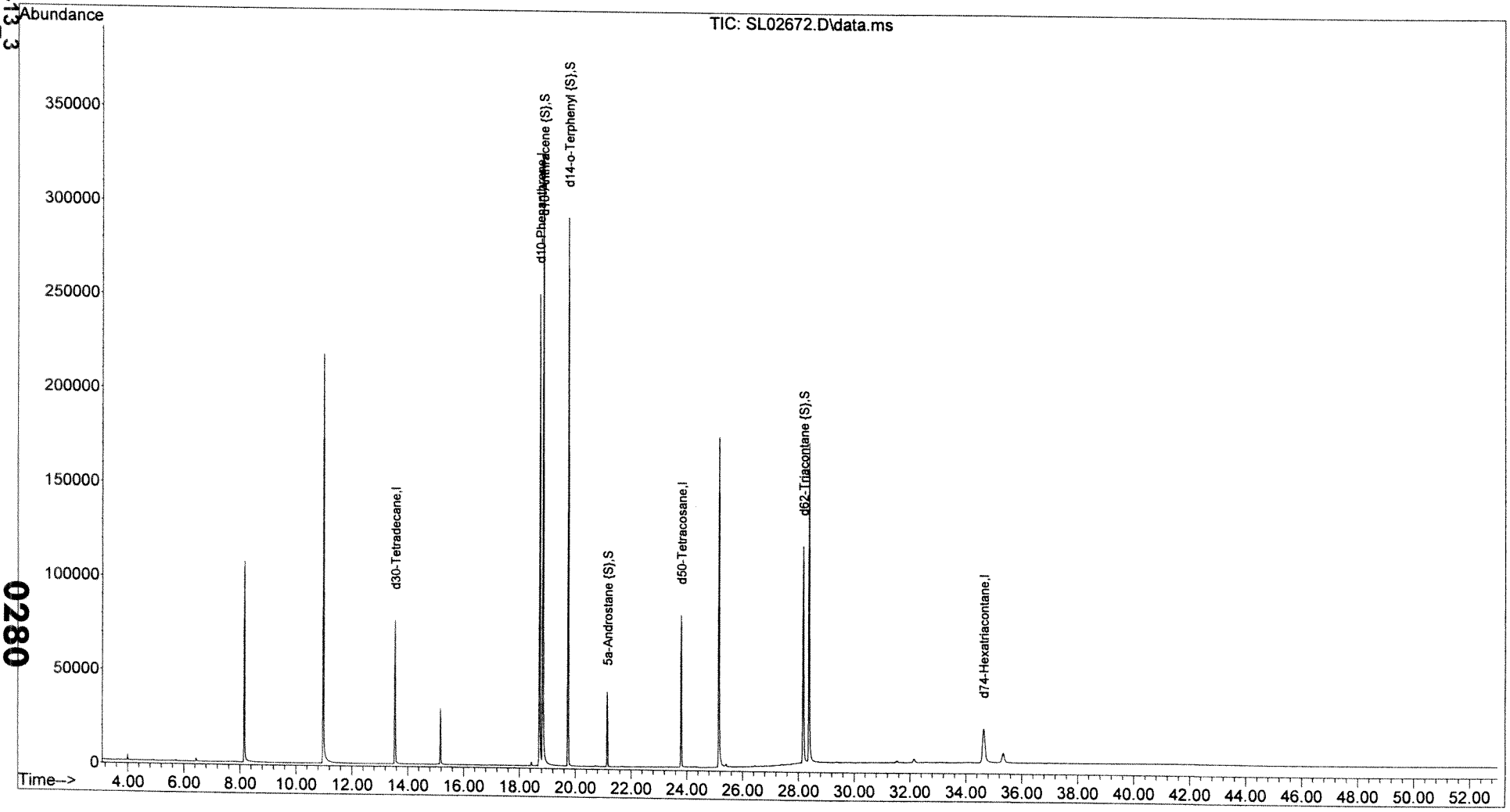
SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Data Pa : C:\msdchem\1\DATA\020212\
Data File : SL02672.D
Acq On : 2 Feb 2012 17:18
Operator : Syslo
Sample : Blank
Misc : 1200012-Blk1
ALS Vial : 80 Sample Multiplier: 1

Quant Time: Feb 02 18:08:13 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0280

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

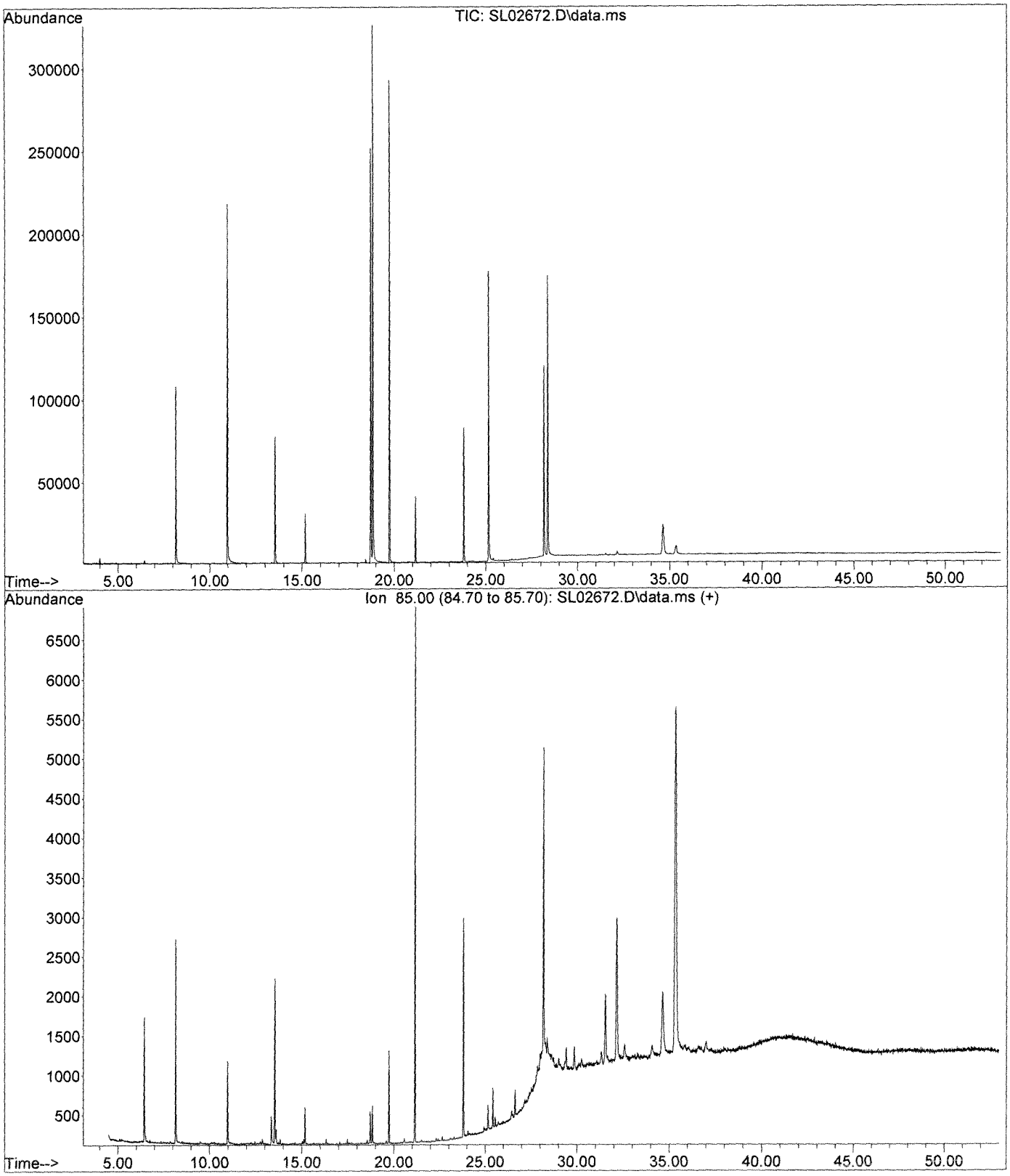
name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

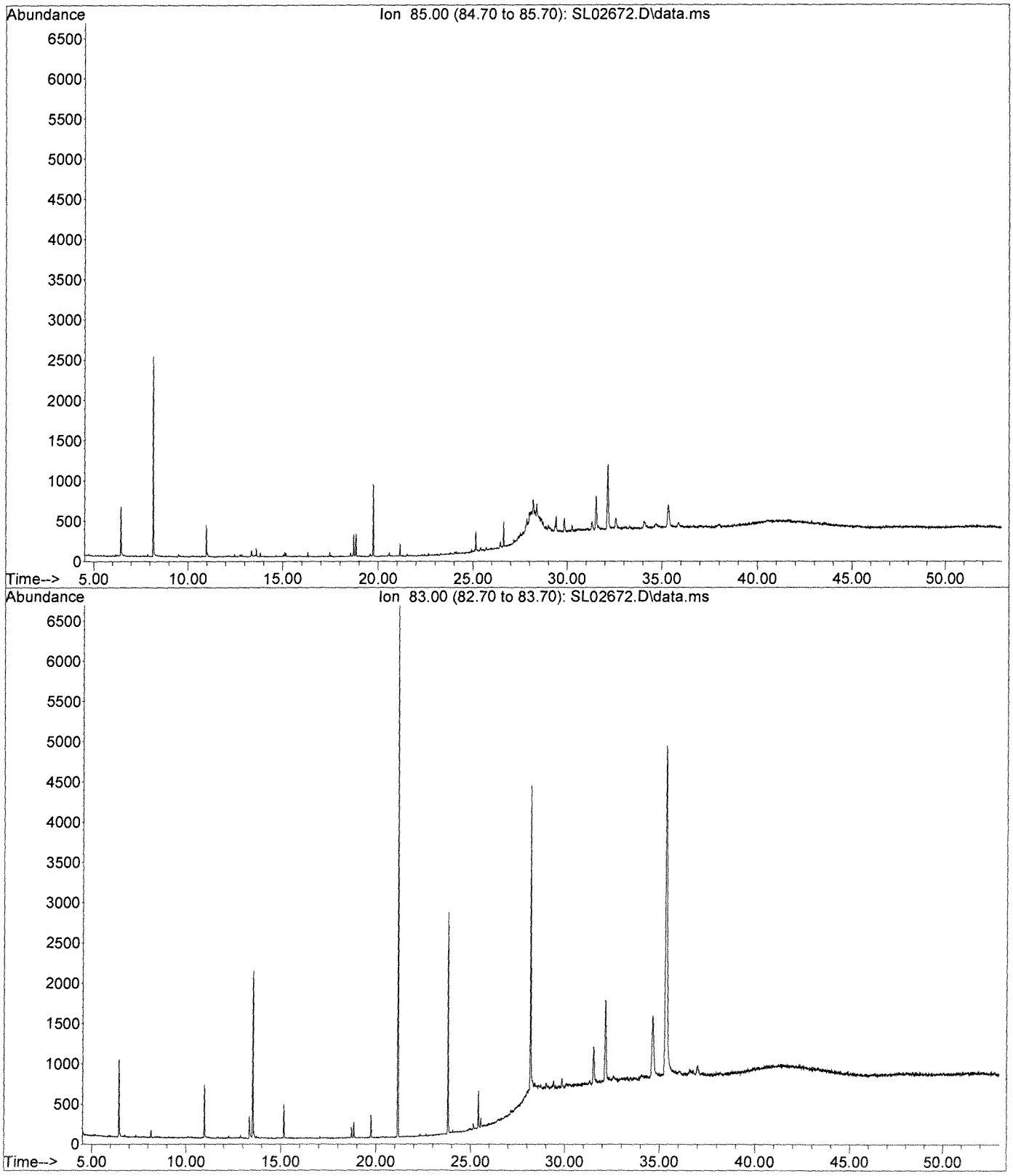
```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012  17:18      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



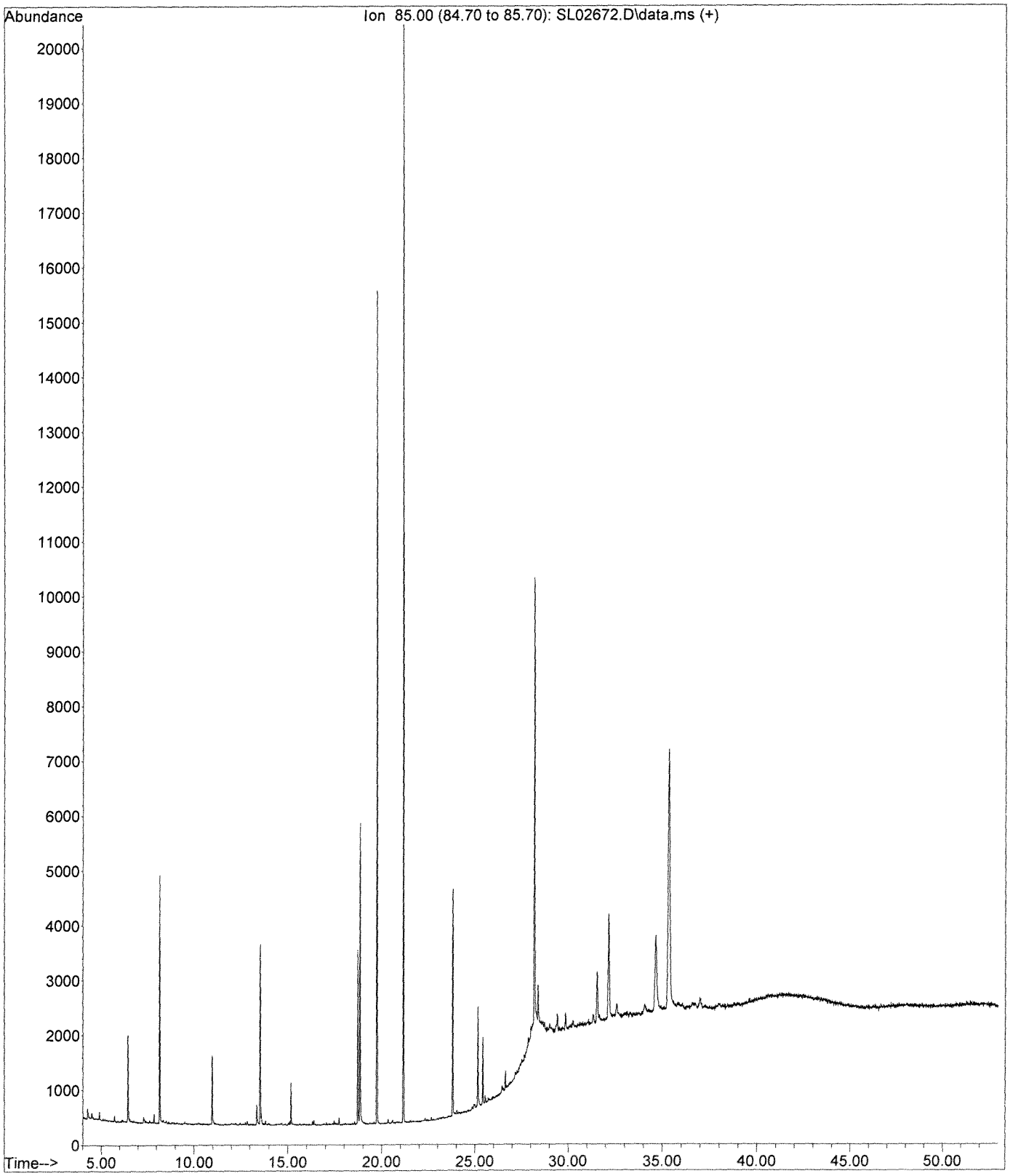
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



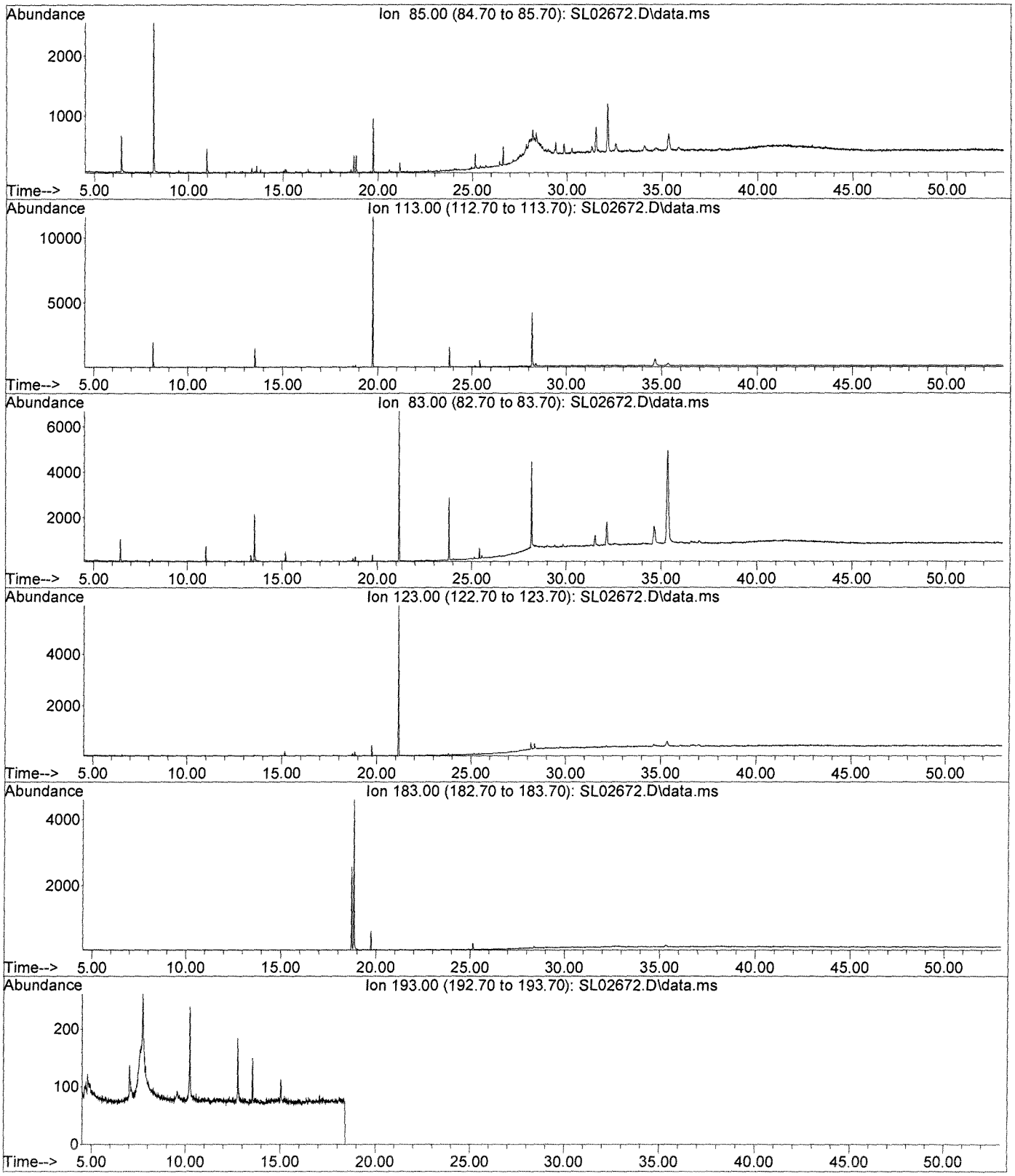
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



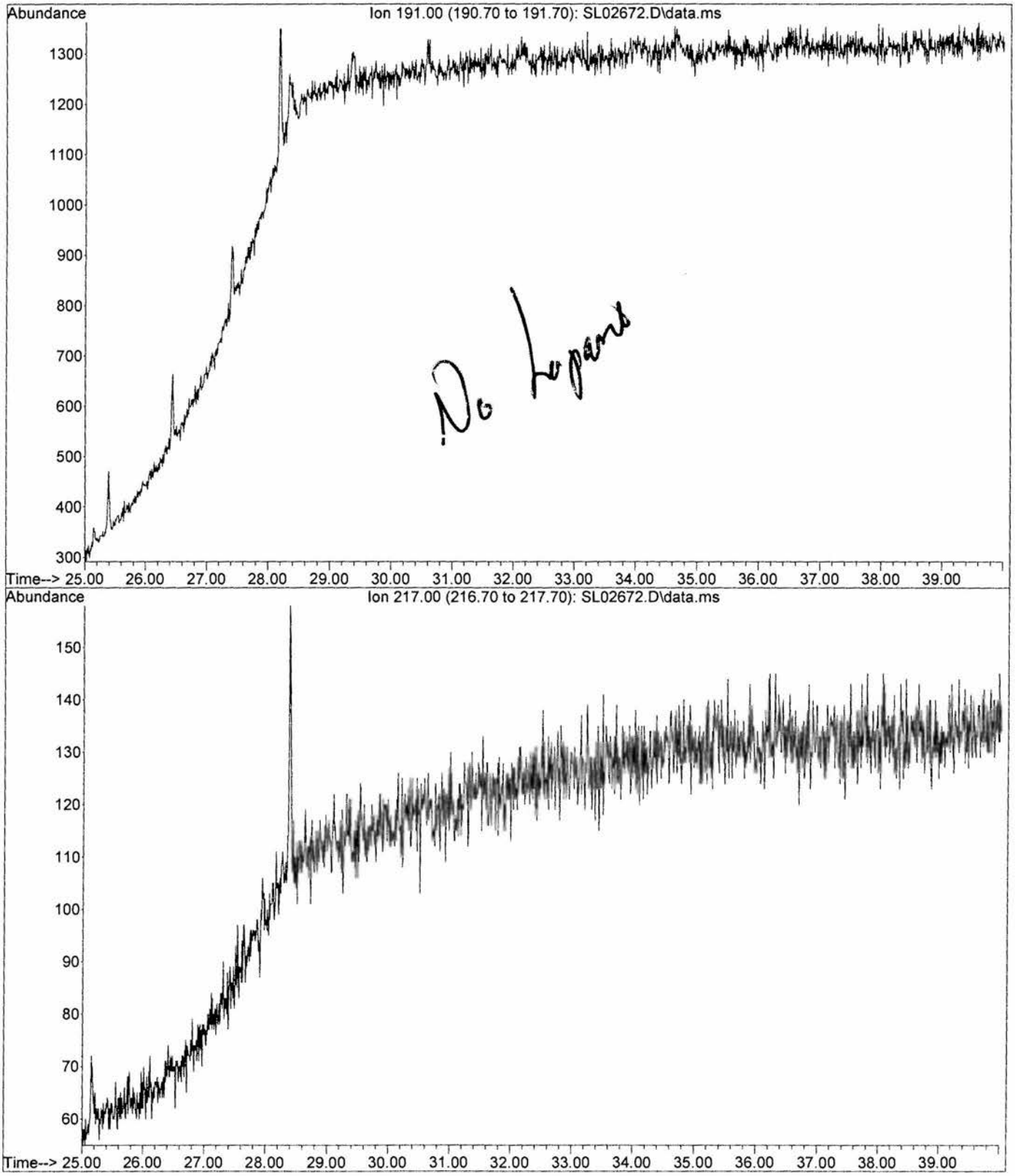
US EPA ARCHIVE DOCUMENT

```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



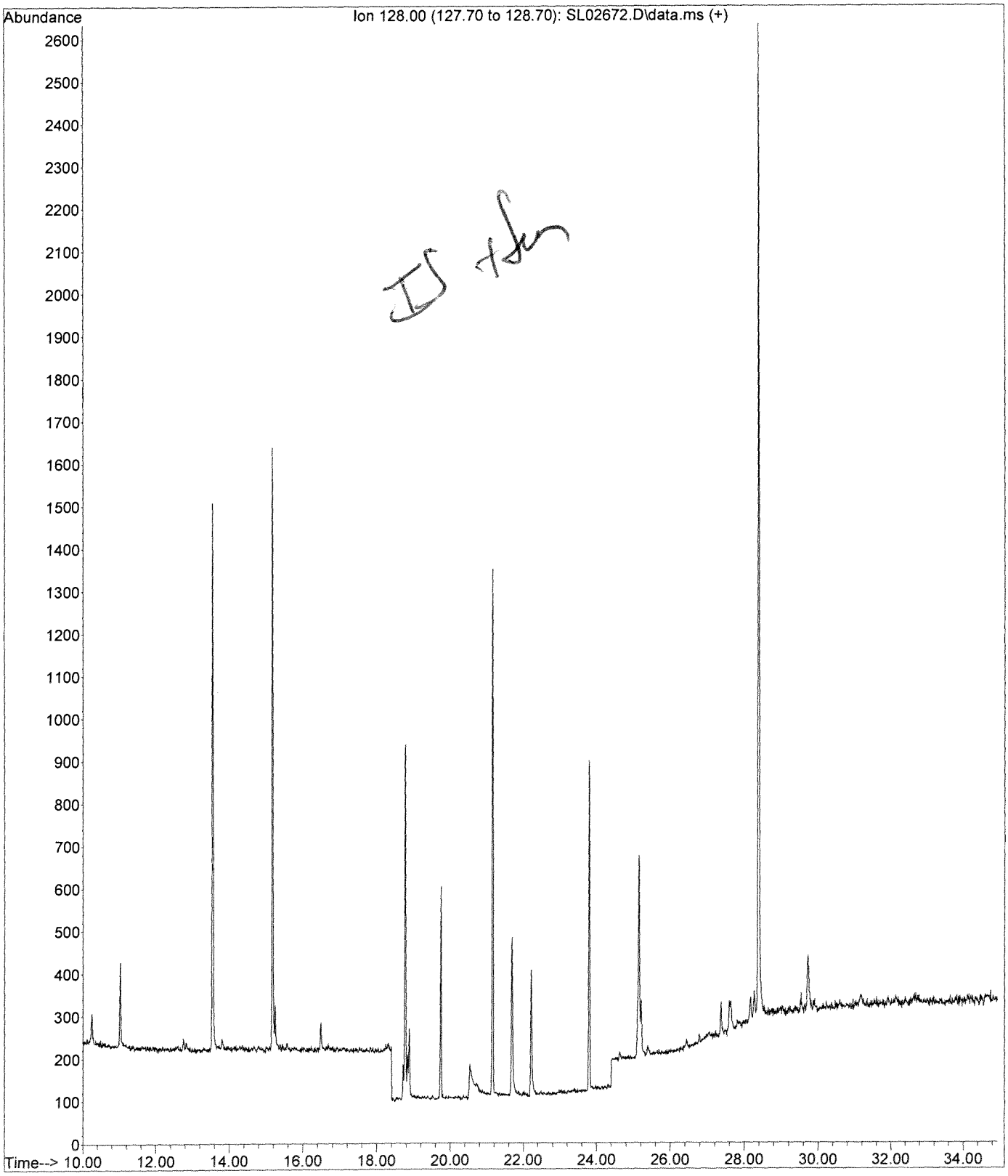
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



IS +Sur

US EPA ARCHIVE DOCUMENT

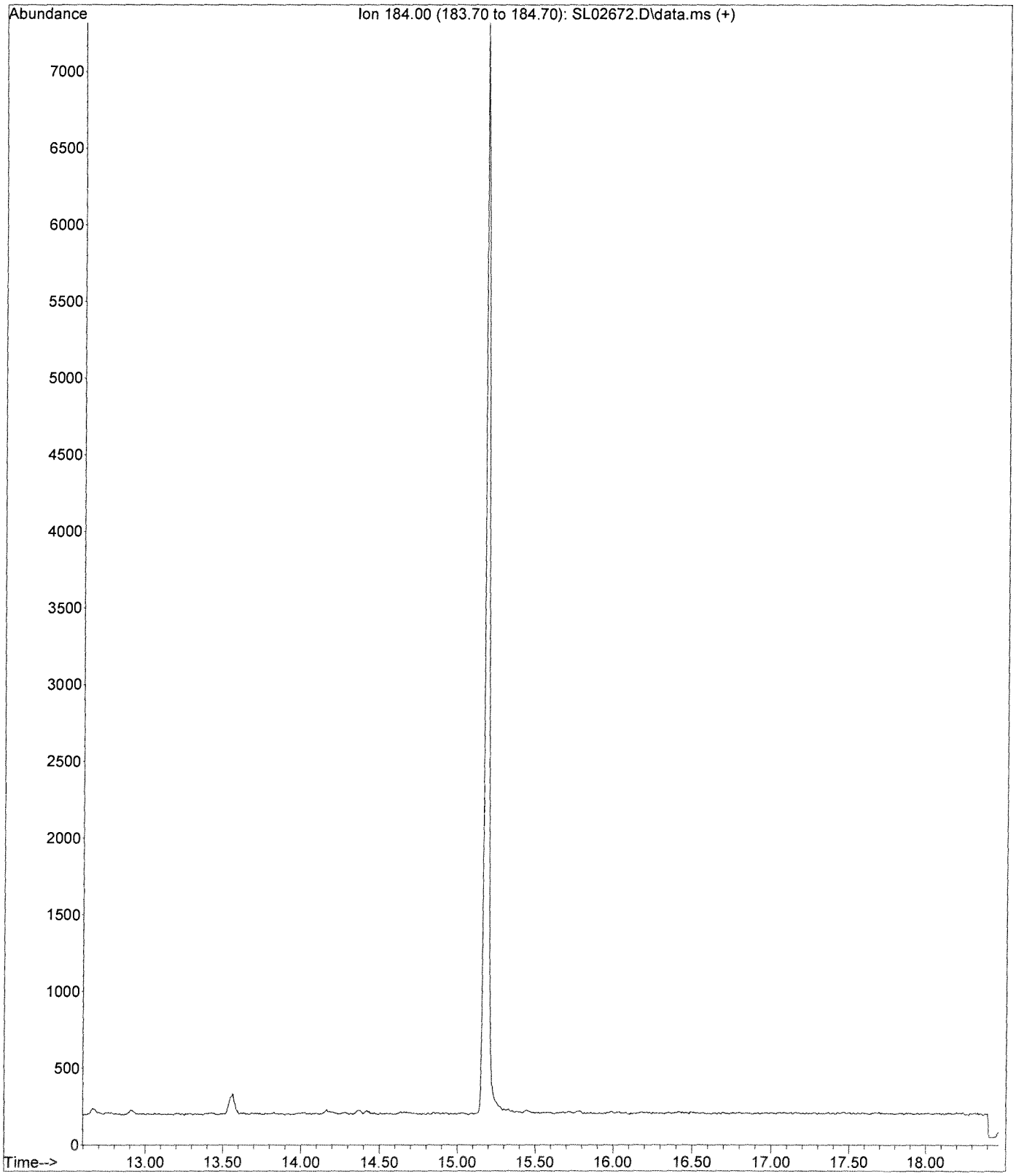
```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

All MP

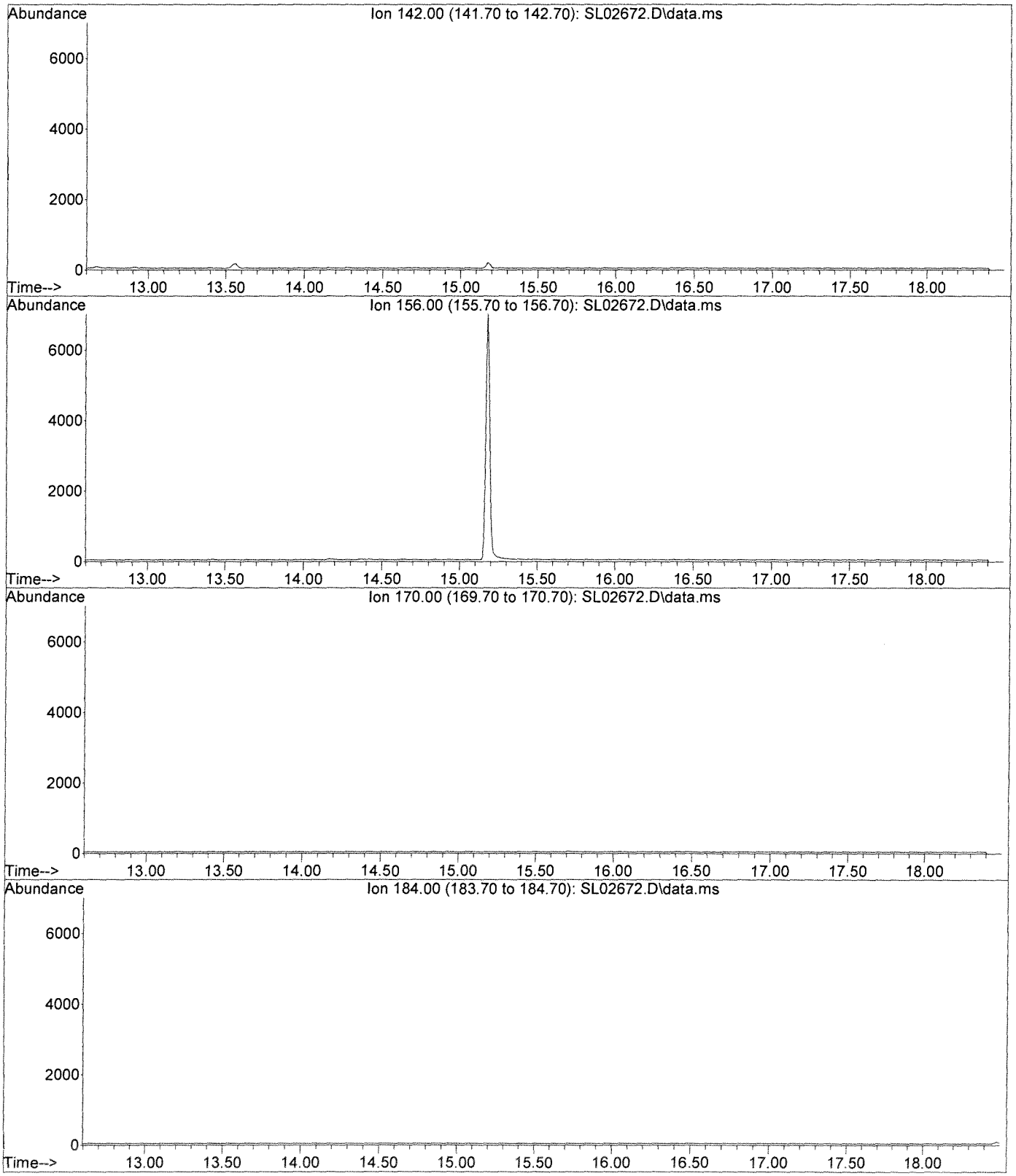
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



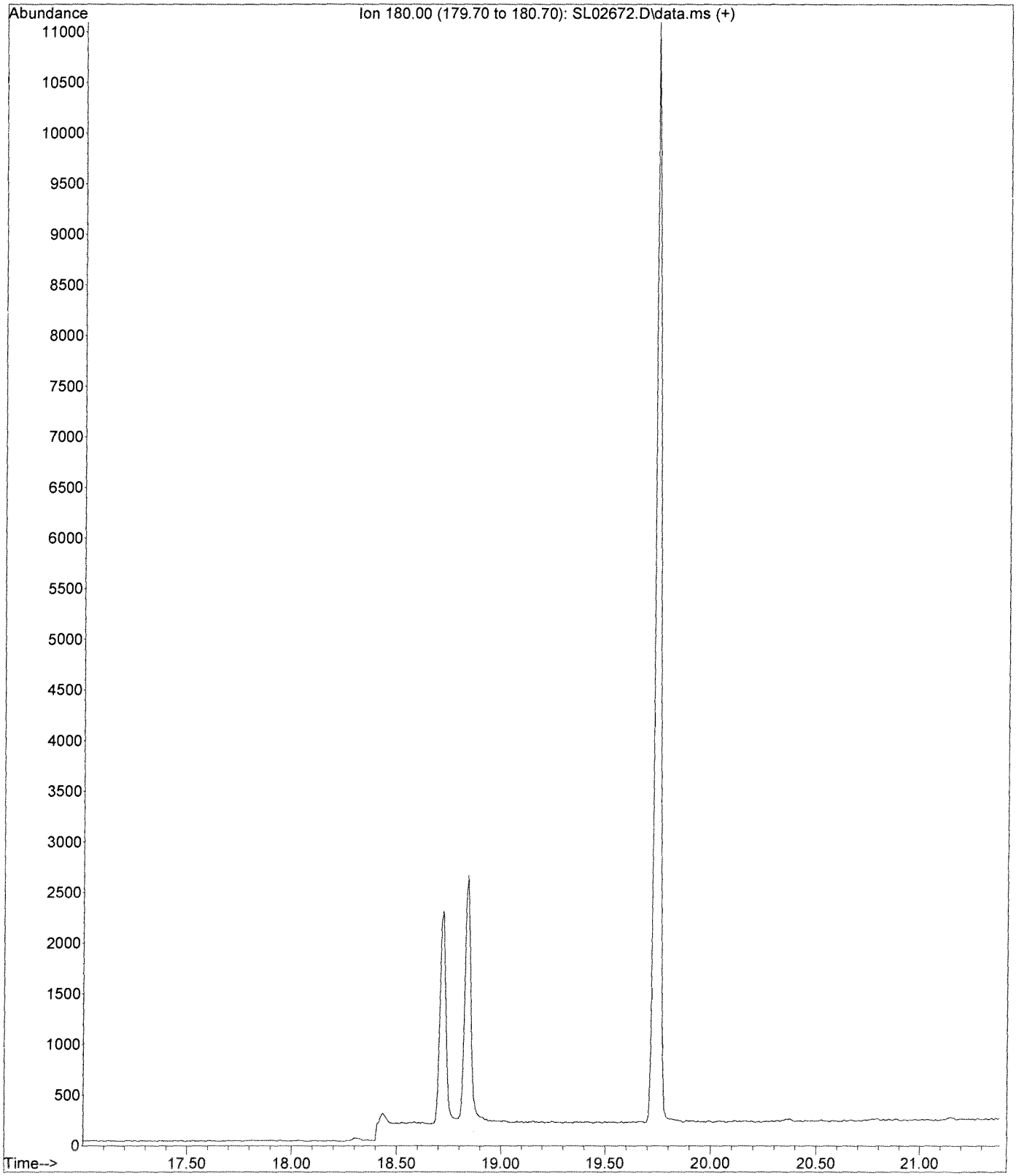
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012  17:18      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



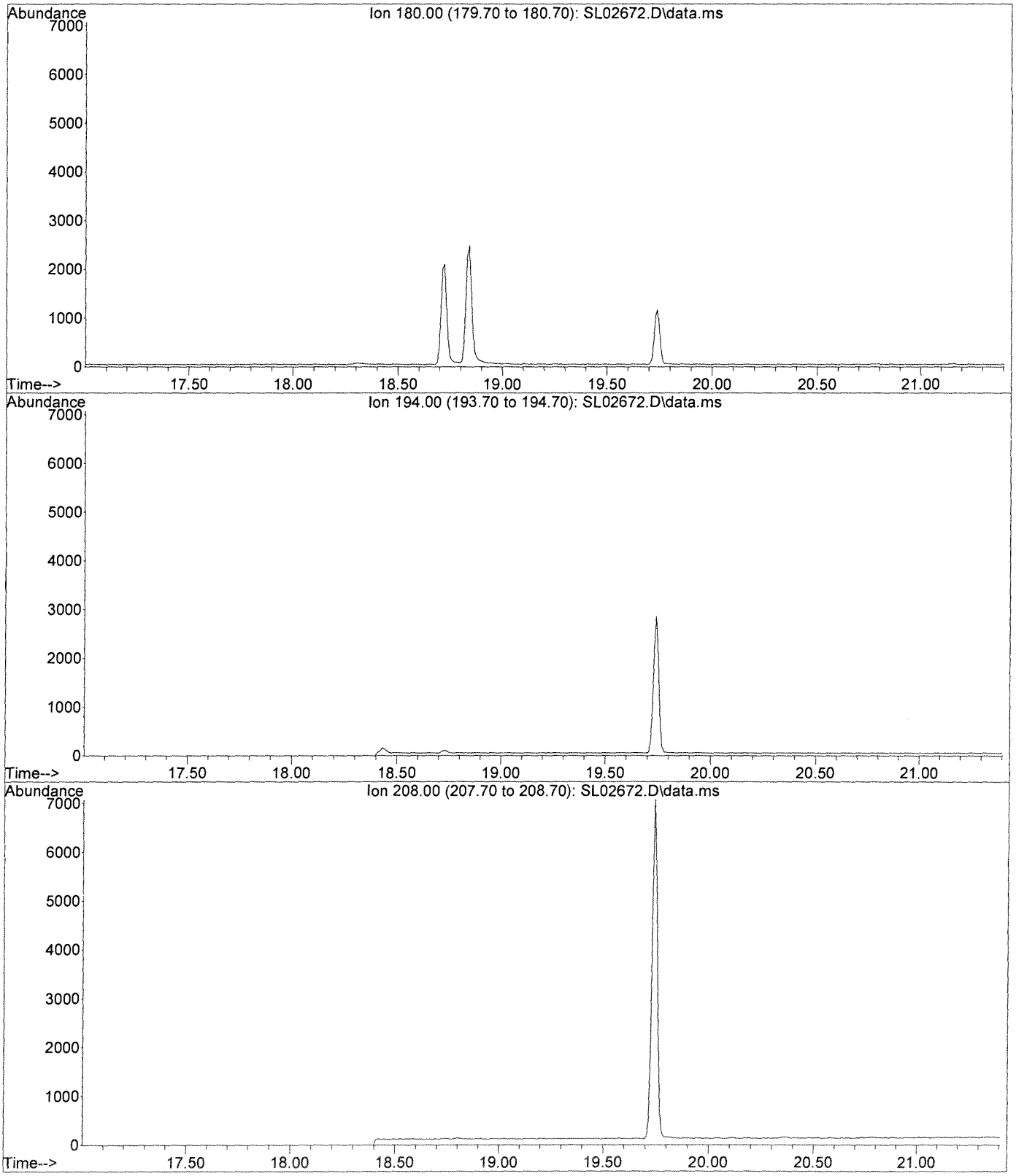
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



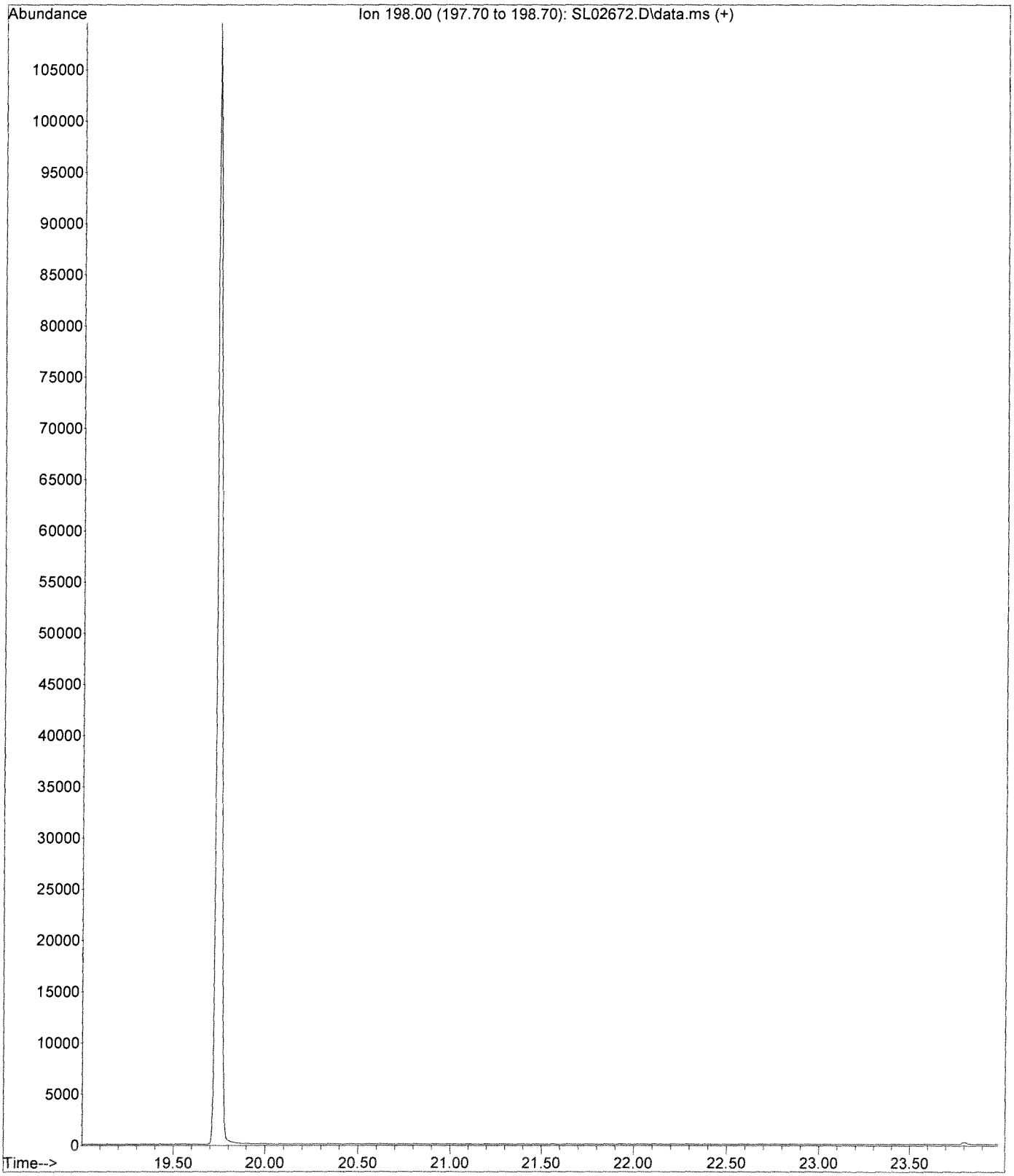
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012  17:18      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



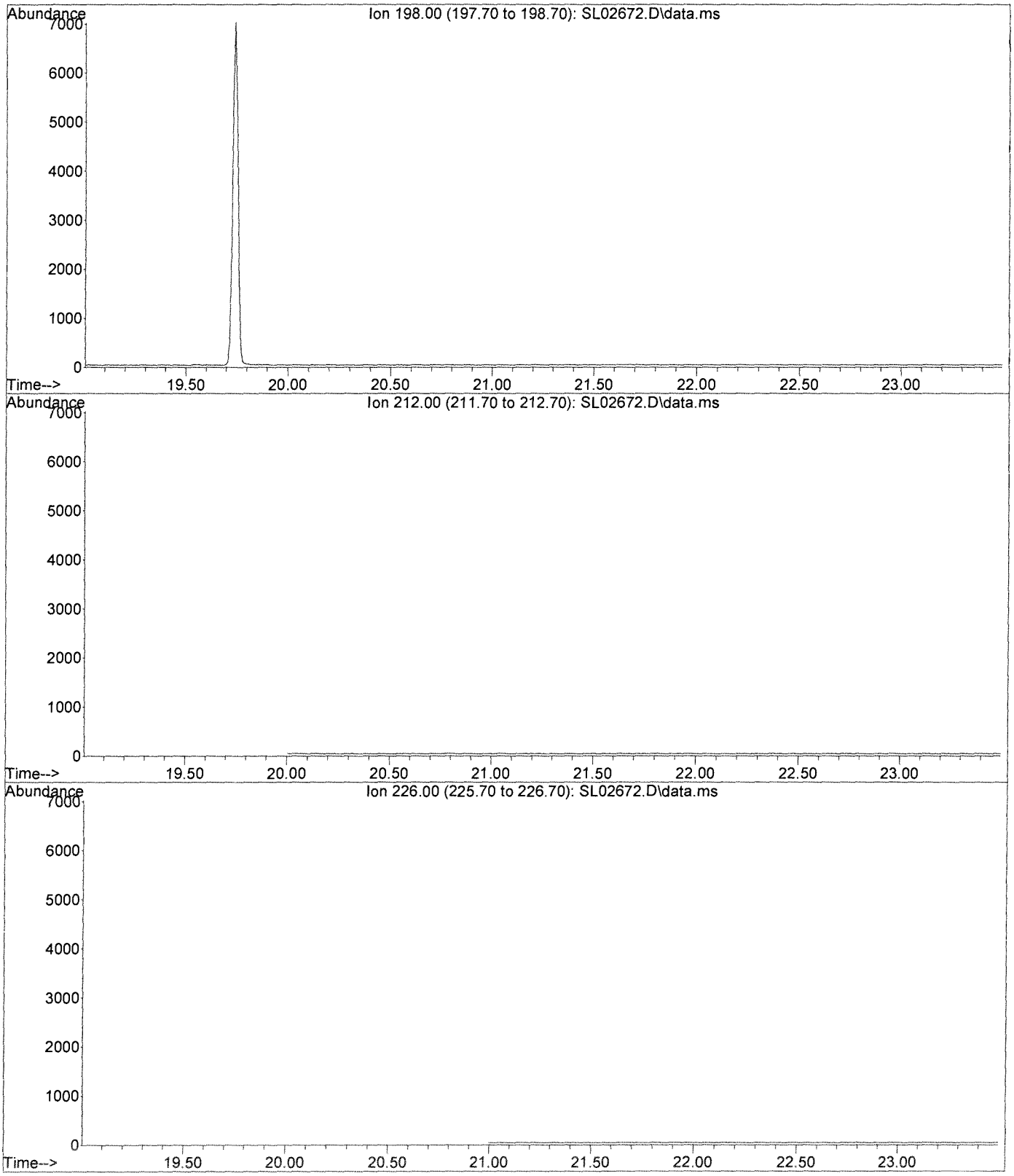
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012  17:18      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



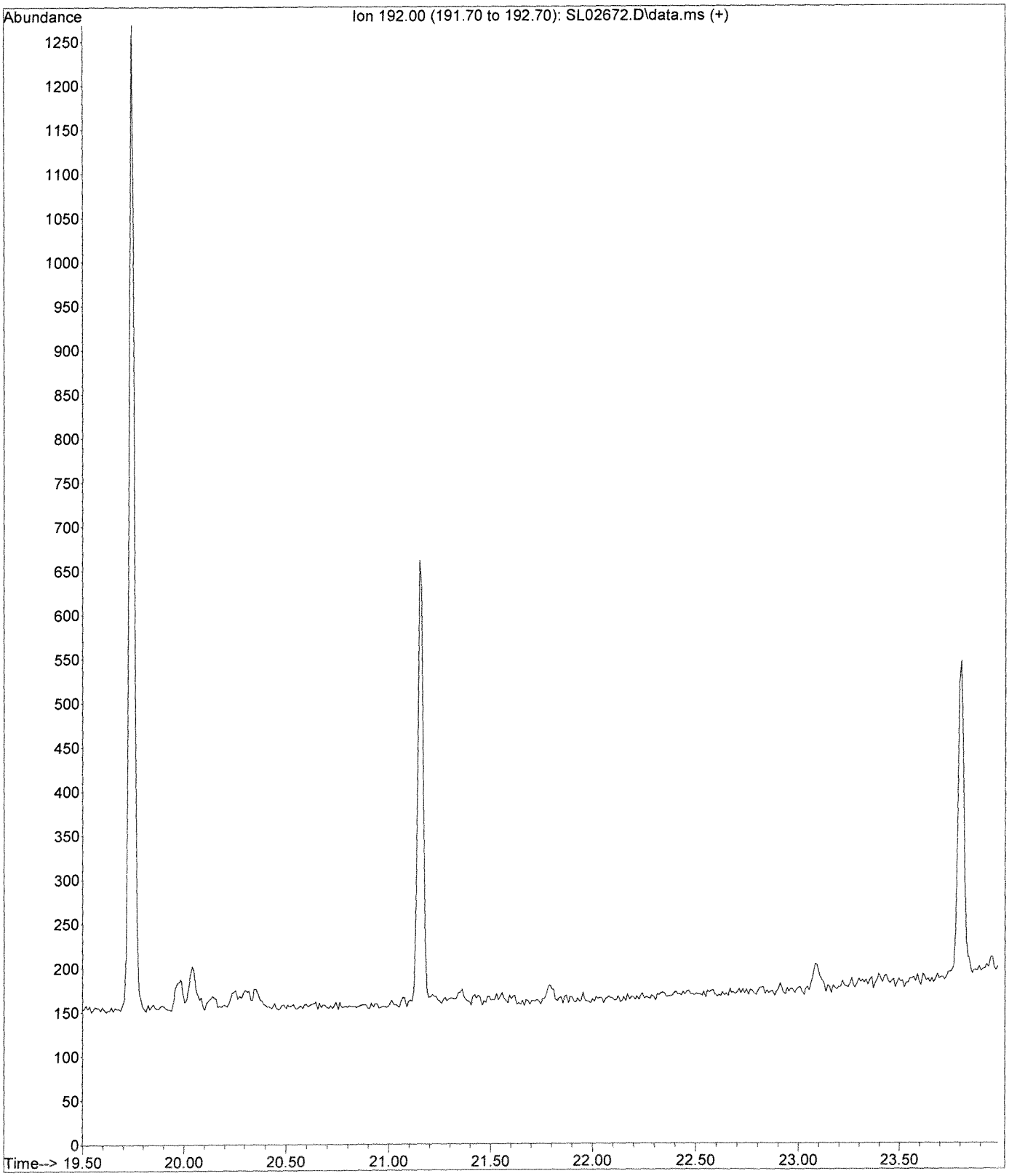
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



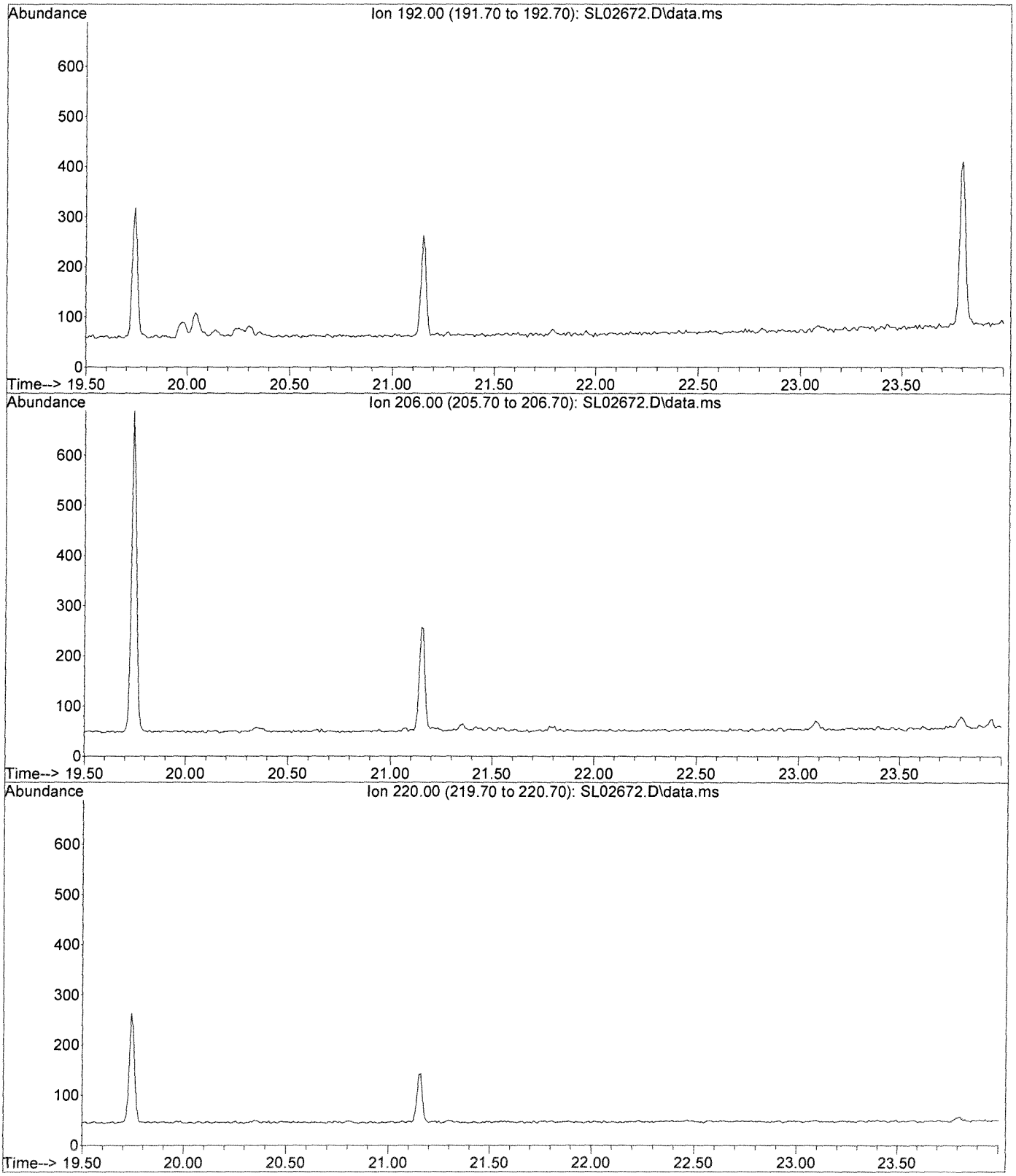
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



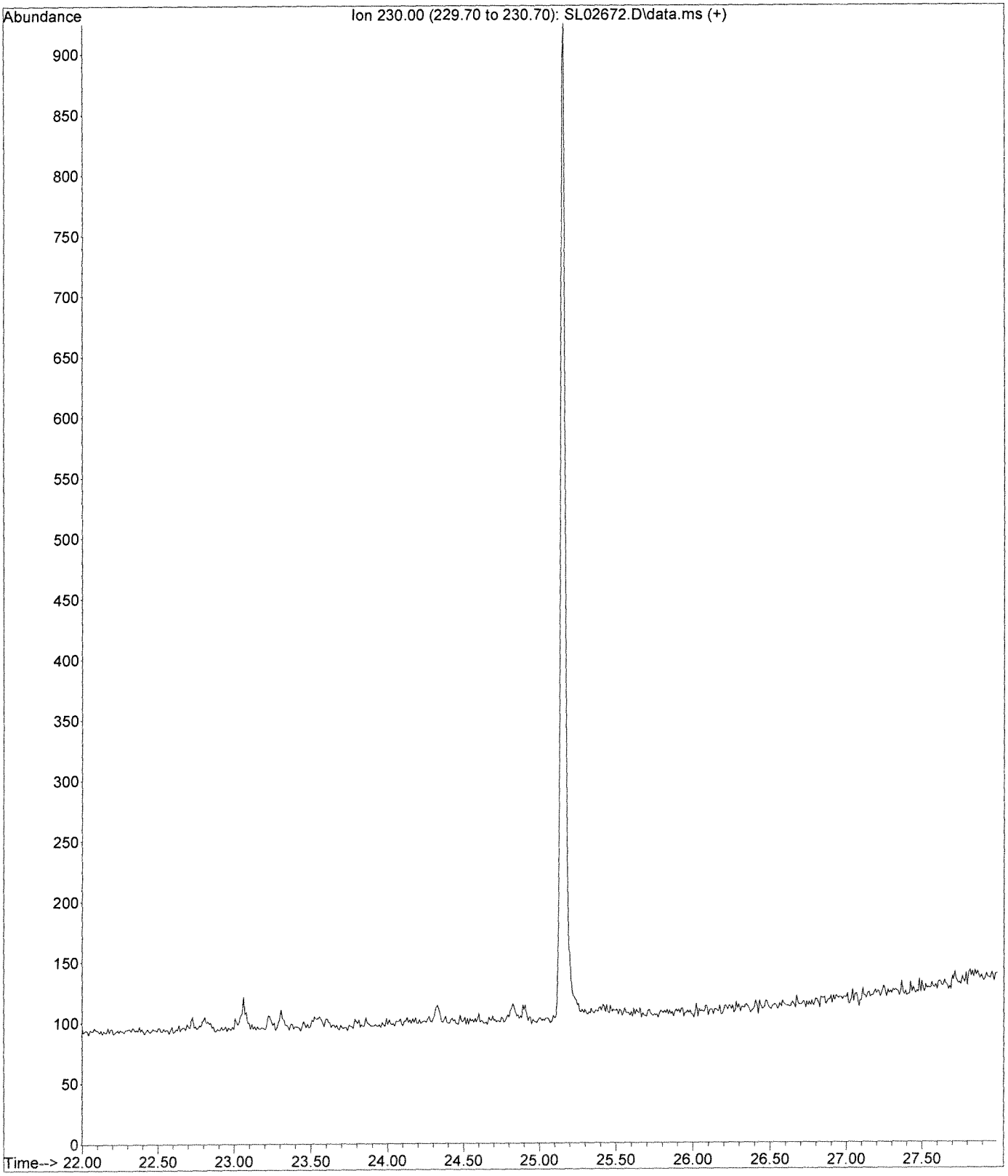
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012  17:18      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



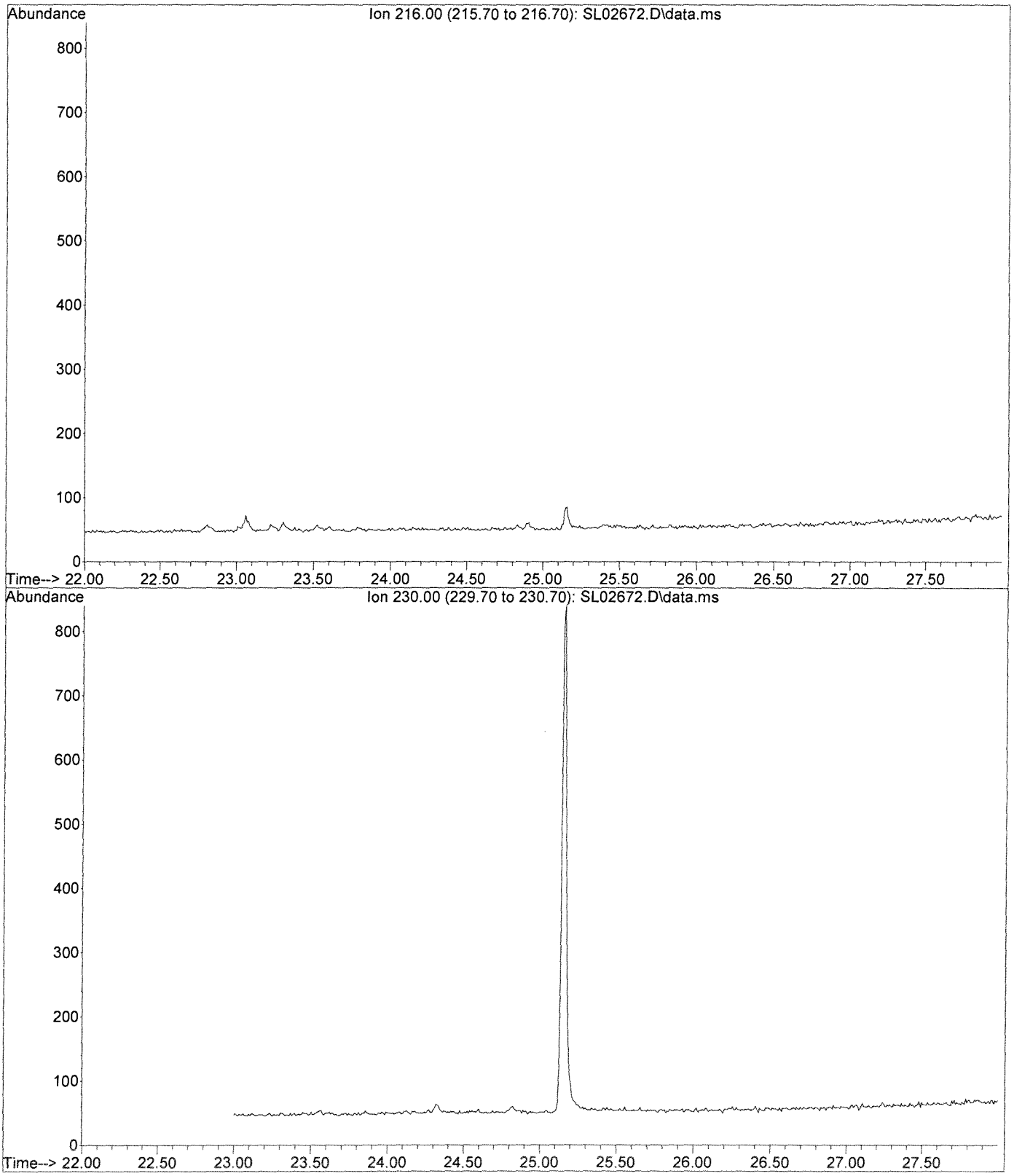
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



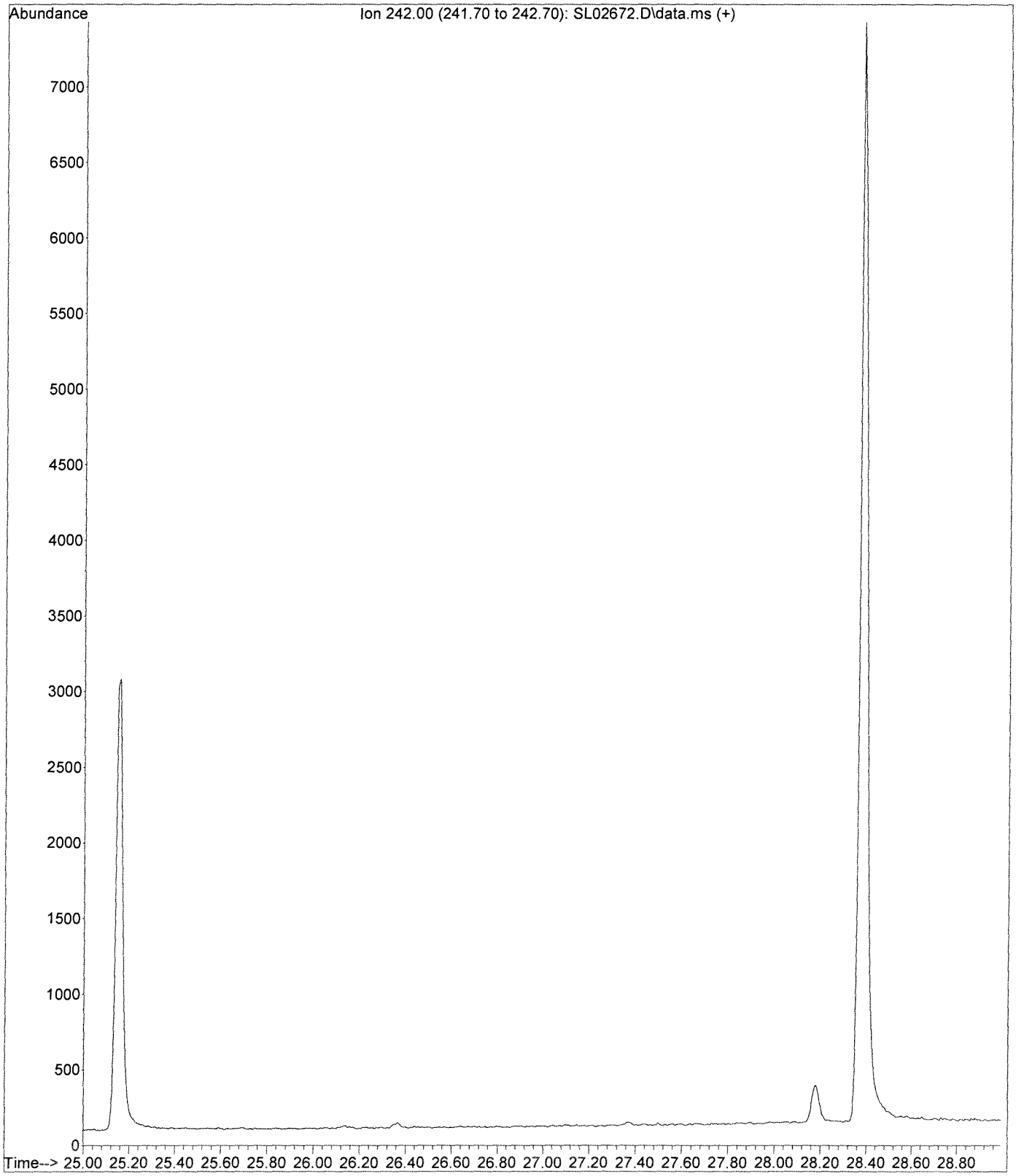
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012   17:18      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



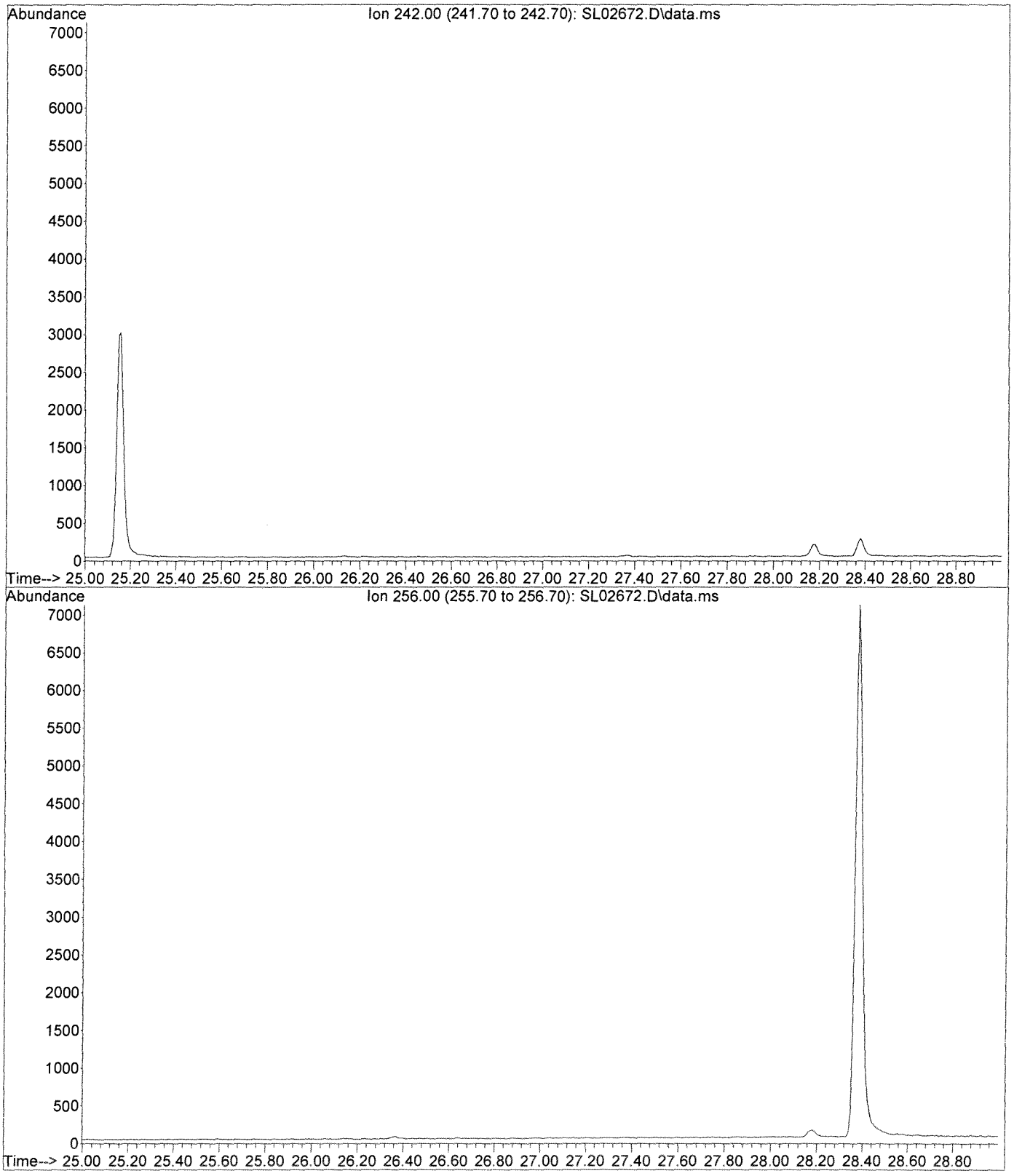
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02672.D
Operator    : Syslo
Acquired    :  2 Feb 2012  17:18      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: Blank
Misc Info   : 1200012-Blk1
Vial Number: 80
```



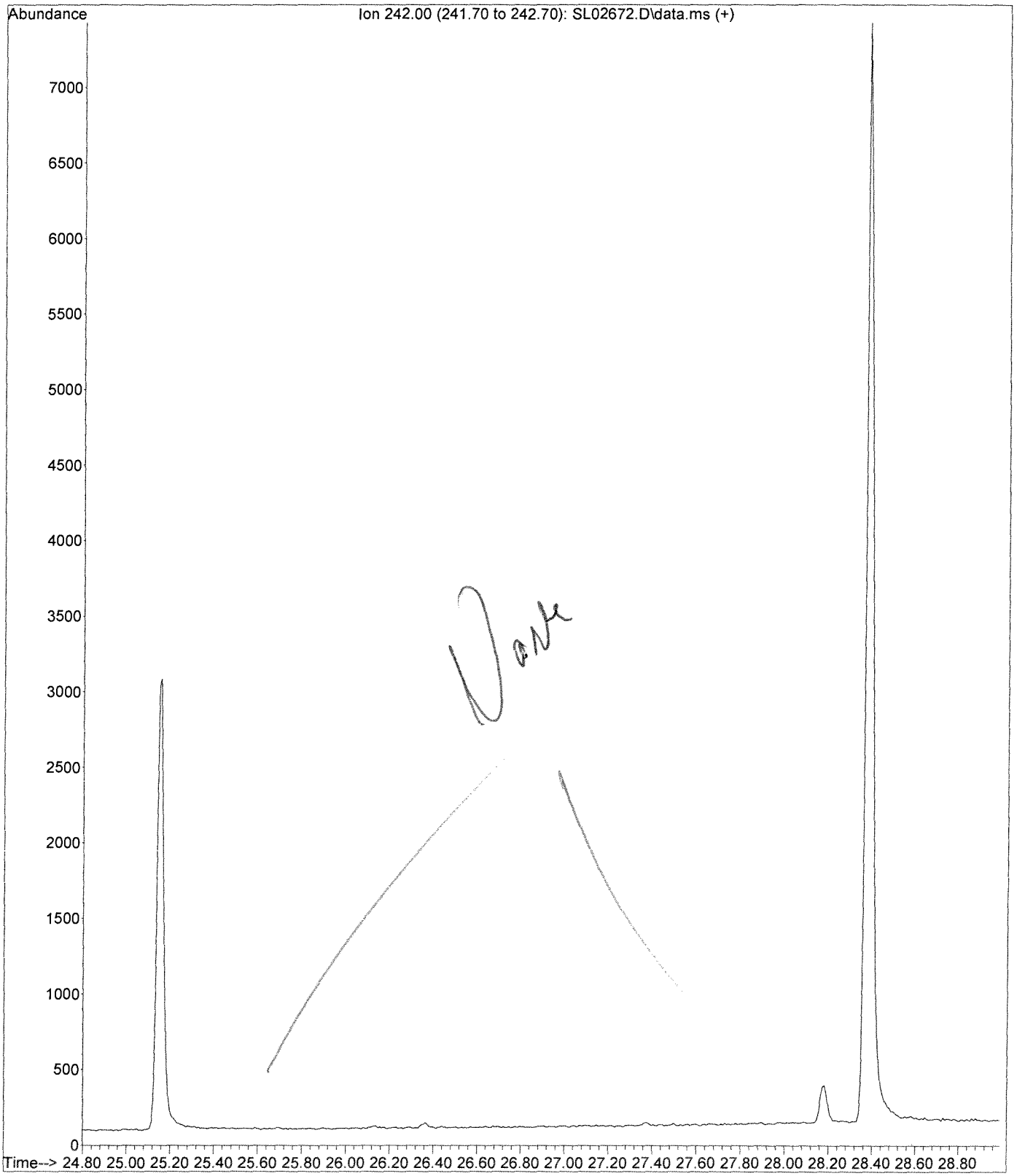
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02672.D
Operator : Syslo
Acquired : 2 Feb 2012 17:18 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Blank
Misc Info : 1200012-Blk1
Vial Number: 80



US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02674.D
Acq On : 2 Feb 2012 19:53
Operator : Syslo
Sample : SERAS-017-0001
Misc : 30g to 1.0mL
ALS Vial : 82 Sample Multiplier: 1

Quant Time: Feb 02 20:43:21 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.713	188	363912	10.00	ug/mL	0.00
5) d30-Tetradecane	13.548	66	124892	10.00	ug/mL	0.00
6) d50-Tetracosane	23.793	66	142419	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.653	66	117011	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.840	188	585933	15.14	ug/mL	0.00
Spiked Amount 20.000			Recovery = 75.70%			
3) d14-o-Terphenyl {S}	19.742	244	278735	16.39	ug/mL	0.00
Spiked Amount 20.000			Recovery = 81.95%			
4) 5a-Androstane {S}	21.158	260	35980	17.39	ug/mL	0.00
Spiked Amount 20.000			Recovery = 86.95%			
7) d62-Triacontane {S}	28.172	66	245317	19.51	ug/mL	0.00
Spiked Amount 20.000			Recovery = 97.55%			
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

am 36521799

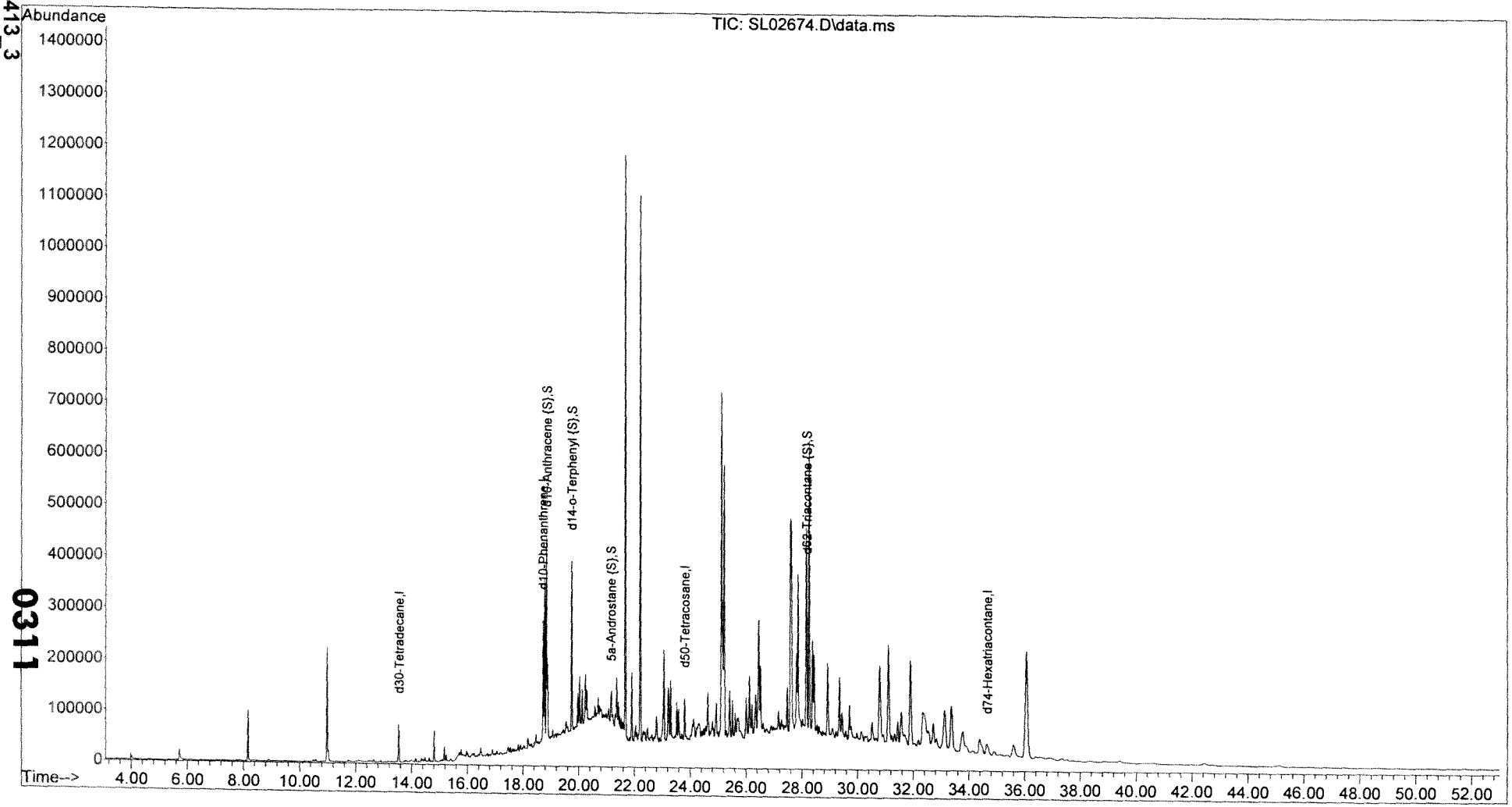
US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02674.D
Acq On : 2 Feb 2012 19:53
Operator : Syslo
Sample : SERAS-017-0001
Misc : 30g to 1.0mL
ALS Vial : 82 Sample Multiplier: 1

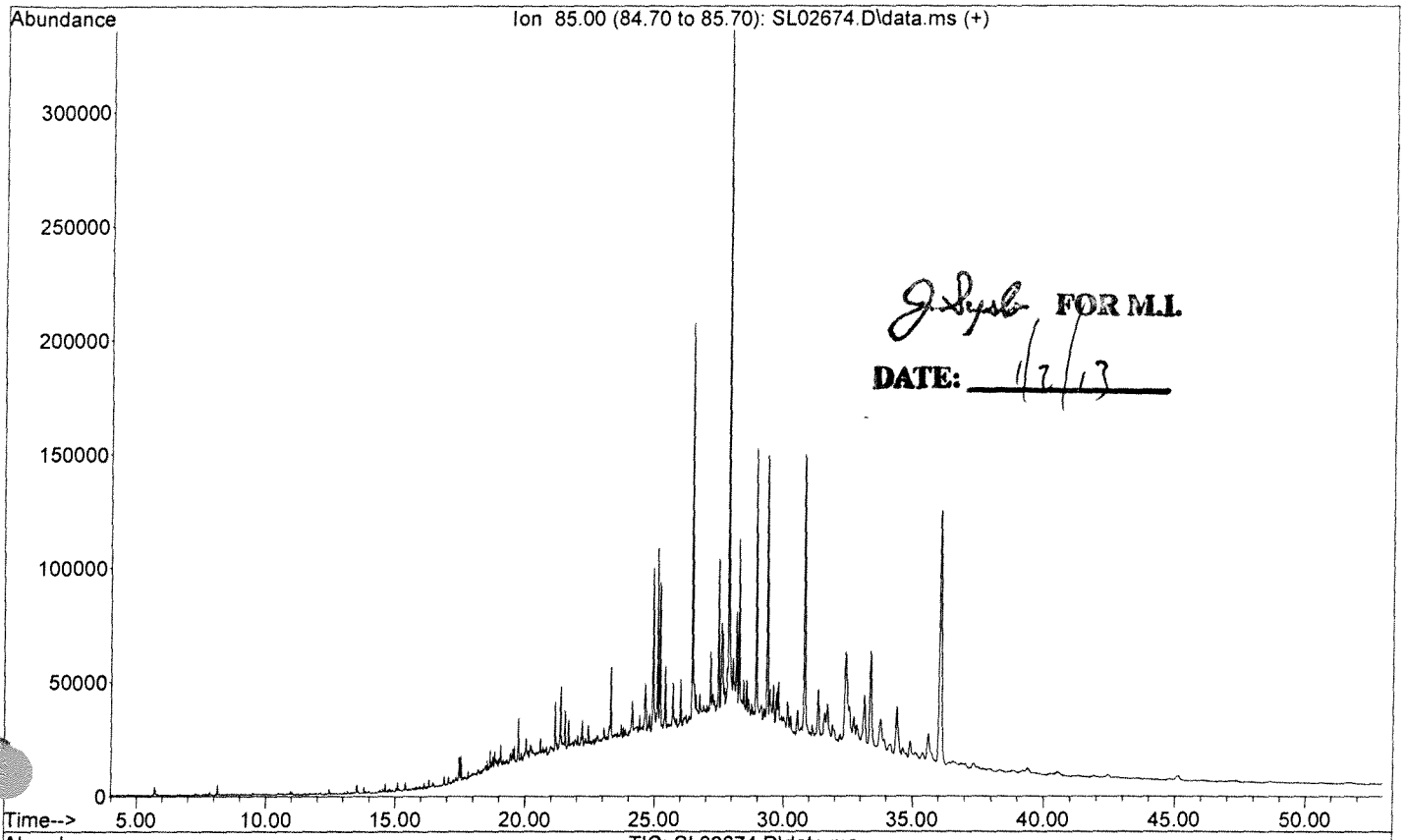
Quant Time: Feb 02 20:43:21 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

SERAS-017-DTM-011413_3

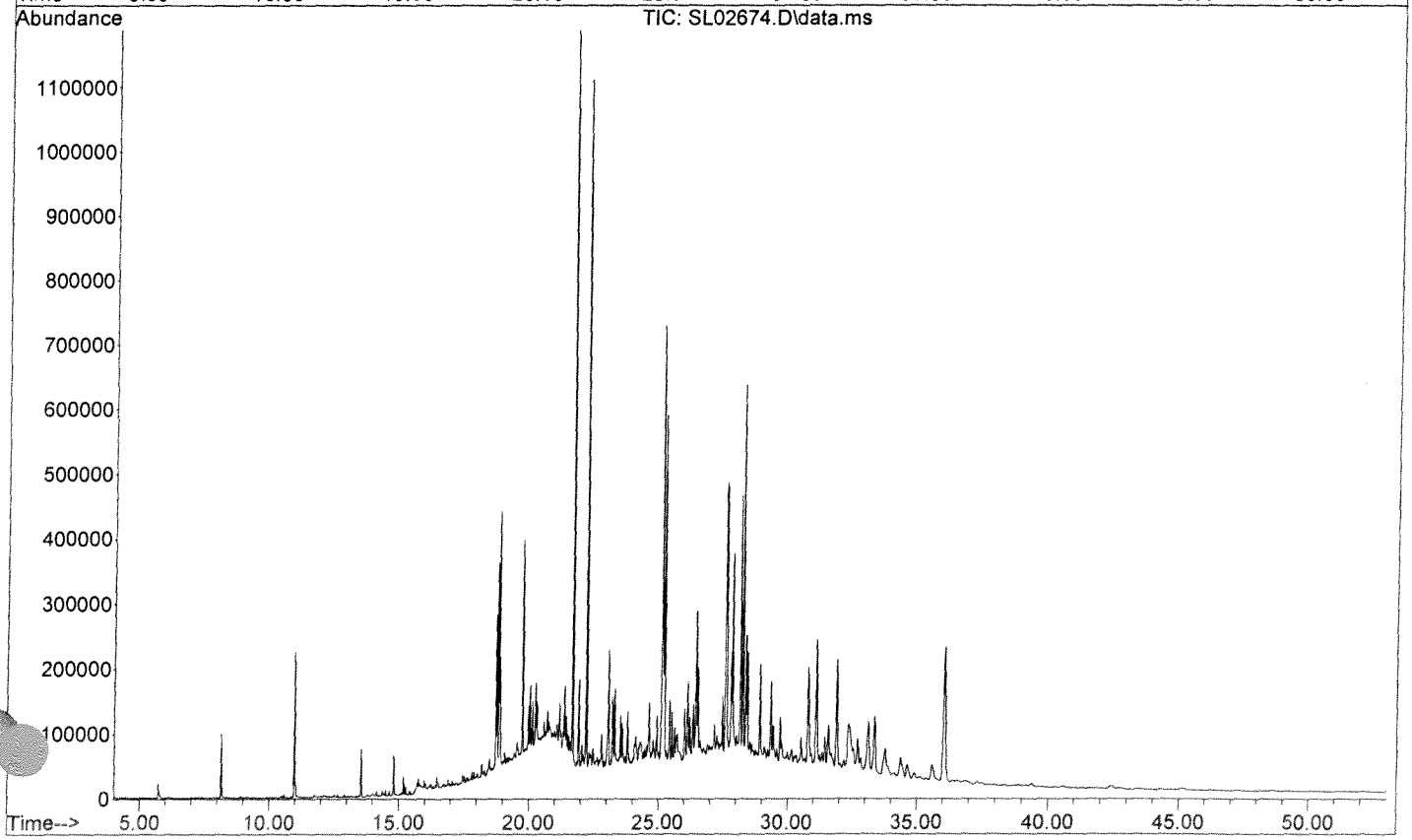


0311

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



J. Syslo FOR M.I.
DATE: 1/7/13



Area Percent Report

Data Path : C:\msdchem\1\DATA\020212\
Data File : SL02674.D
Acq On : 2 Feb 2012 19:53
Operator : Syslo
Sample : SERAS-017-0001
Misc : 30g to 1.0mL
ALS Vial : 82 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 0.5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 5
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\msdchem\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL02674.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	27.856	960	2997	5916	rM	333409	36524299	100.00%	100.000%

Sum of corrected areas: 36524299

DROTPH051611.M Wed Jan 02 12:12:08 2013 SLICK2

J. Syslo FOR M.I.
DATE: 1/2/13

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM.* Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

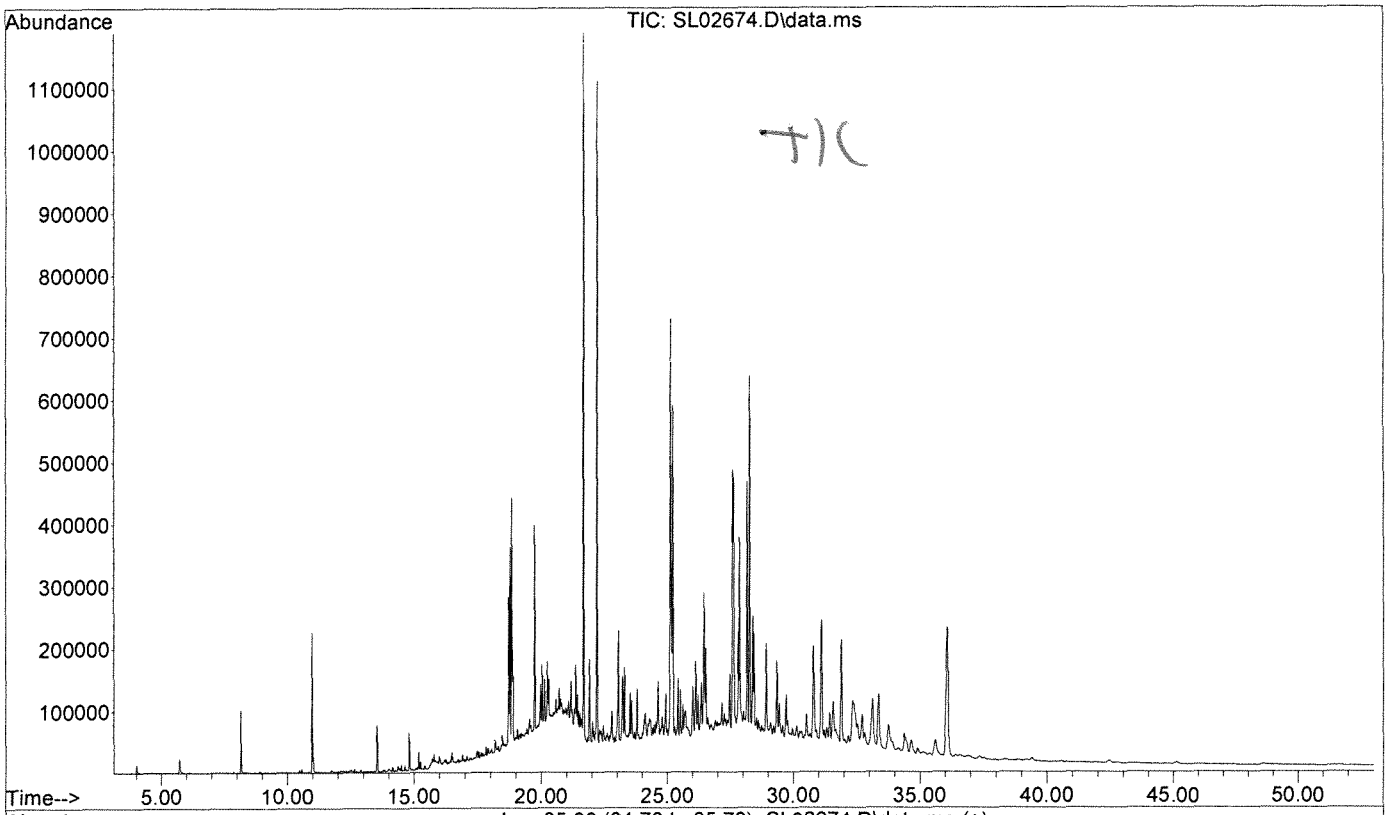
name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

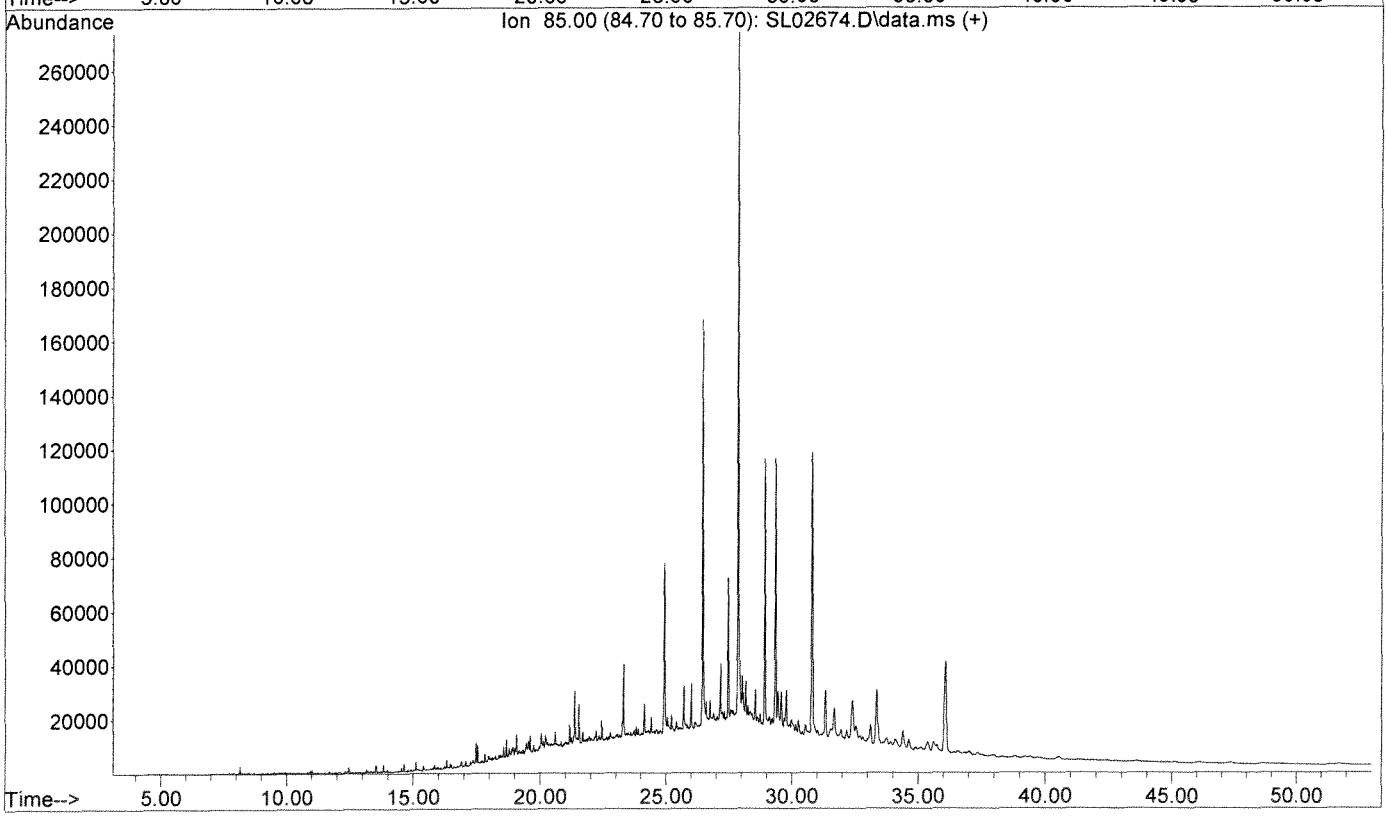
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



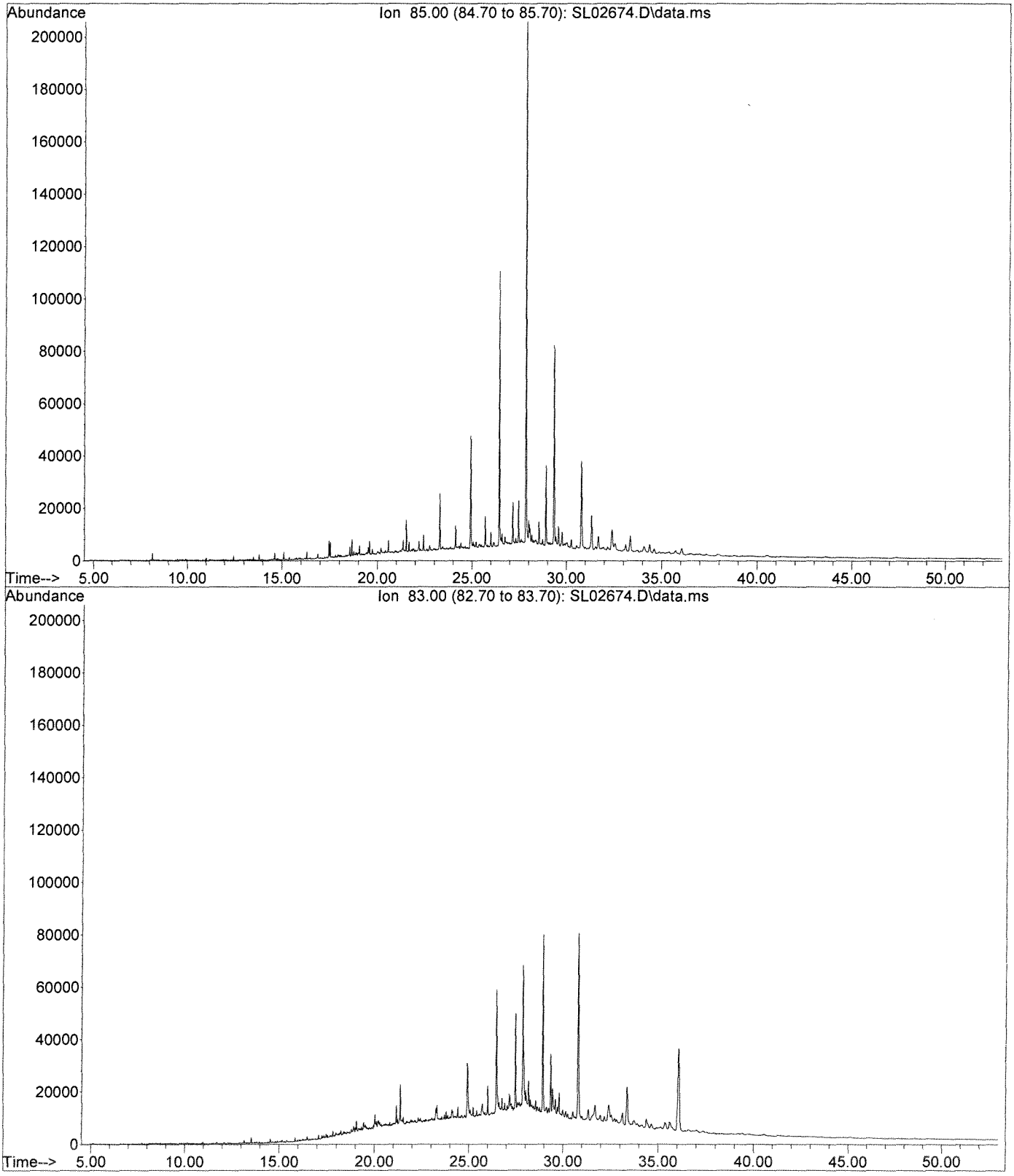


US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



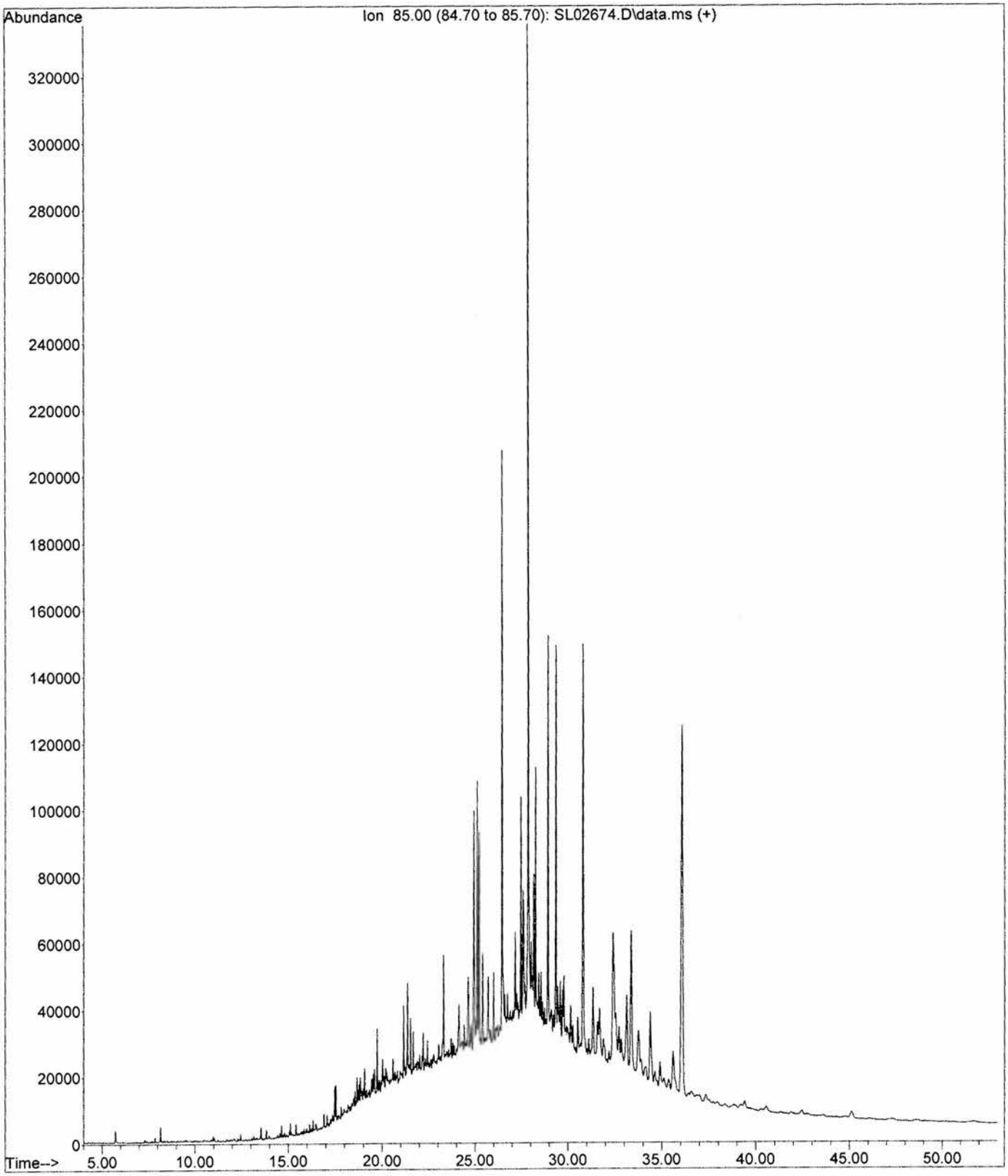
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



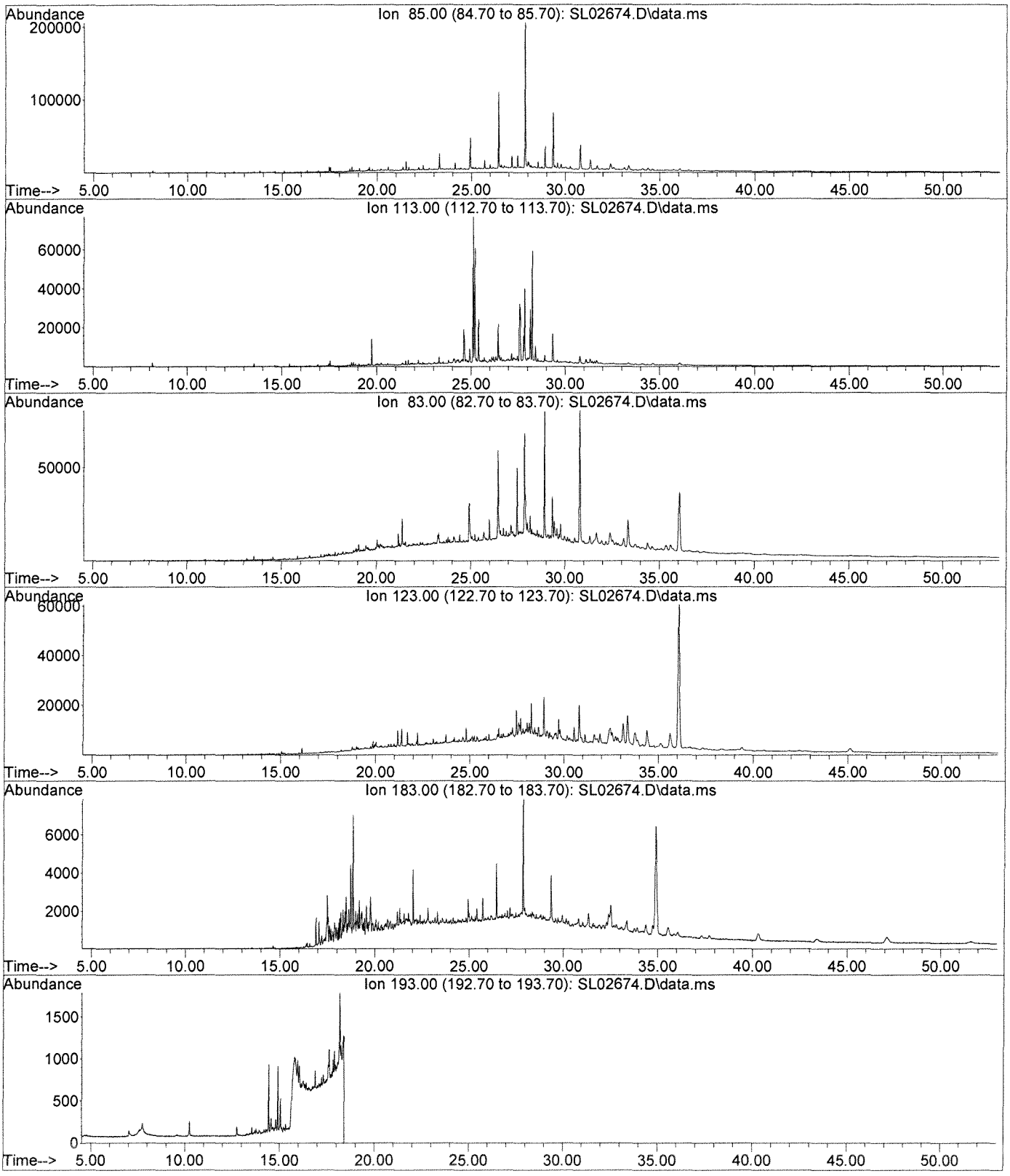
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02674.D
Operator    : Syslo
Acquired    :  2 Feb 2012  19:53      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0001
Misc Info   : 30g to 1.0mL
Vial Number: 82
```



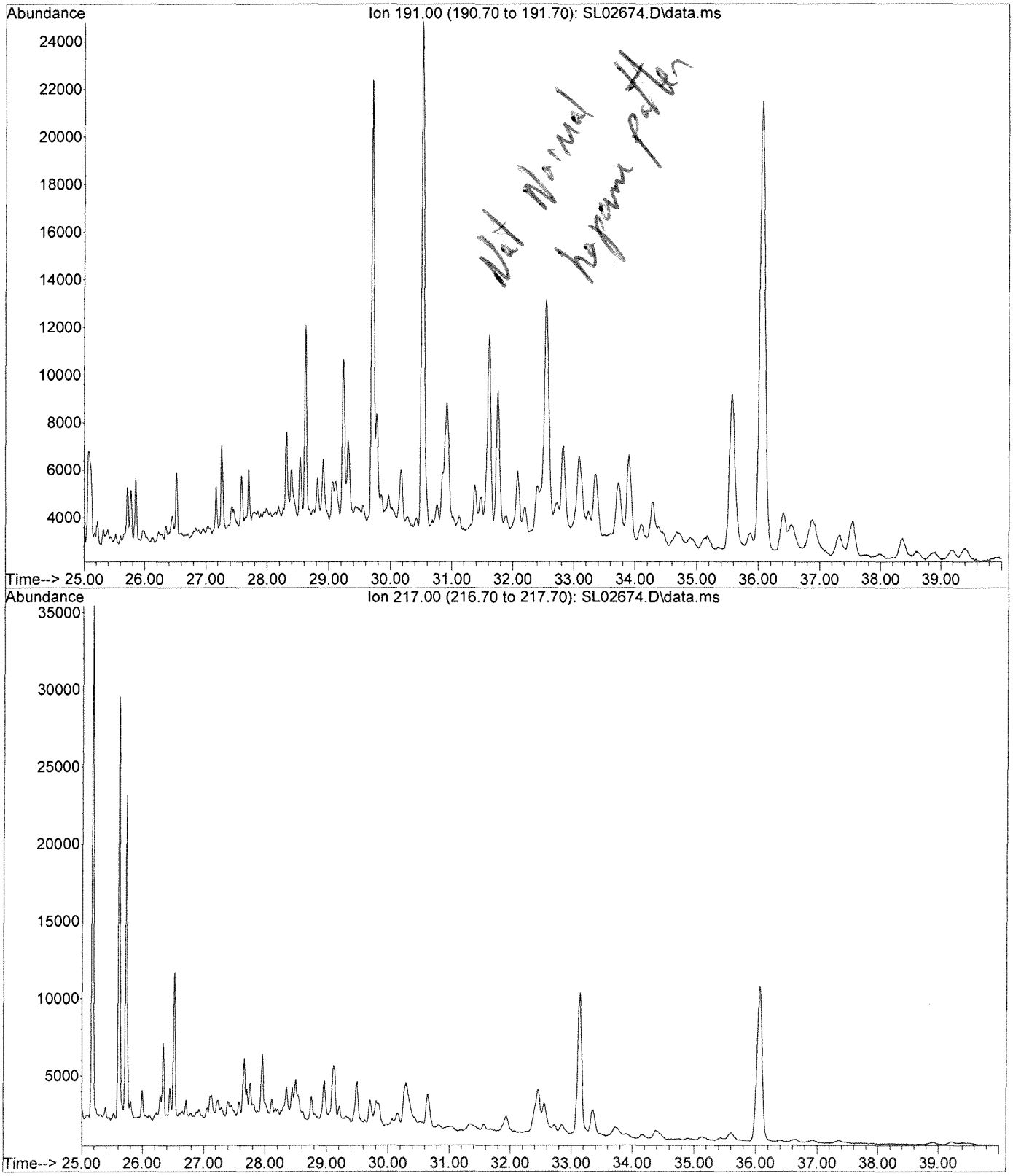
US EPA ARCHIVE DOCUMENT

```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



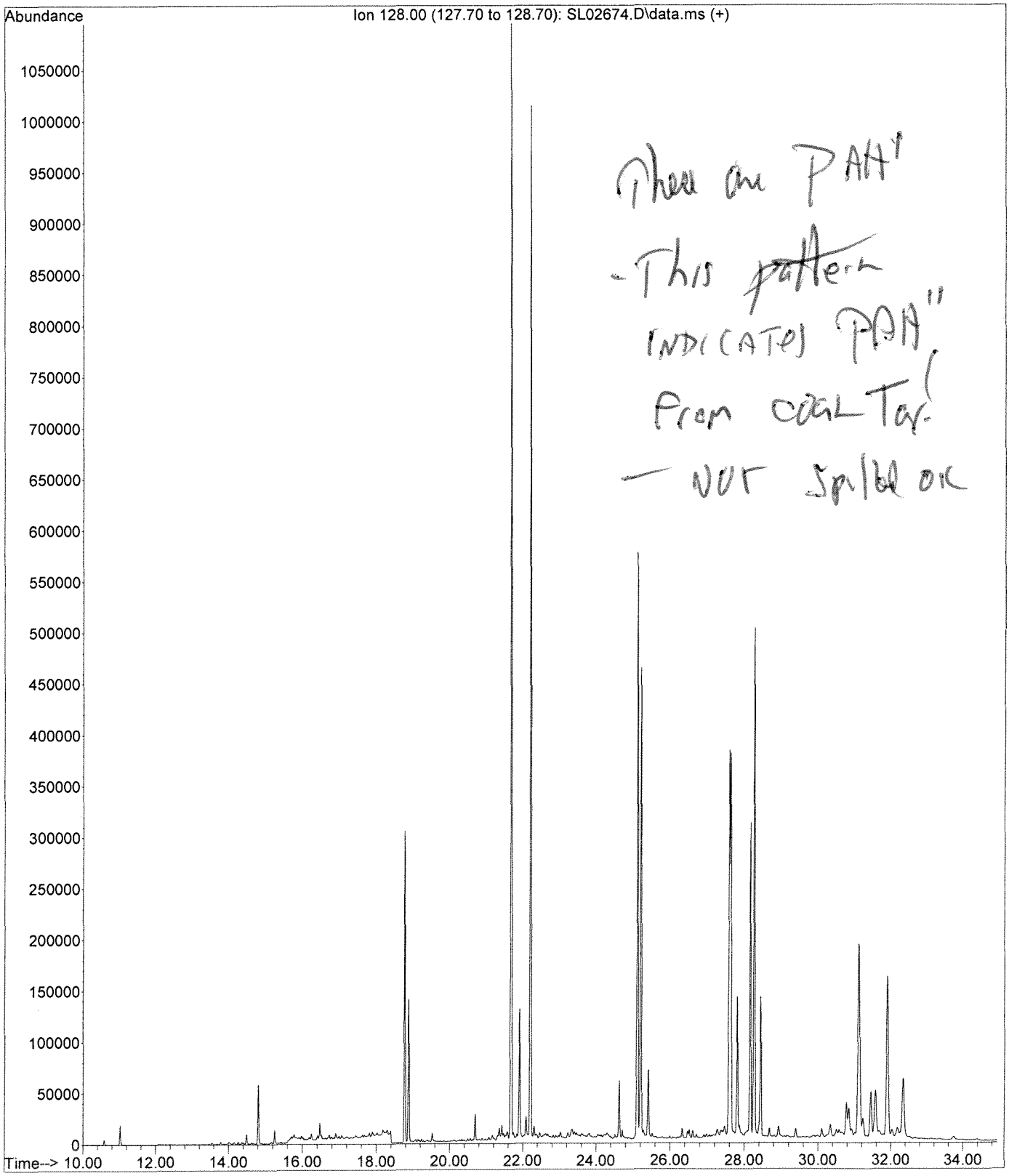
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

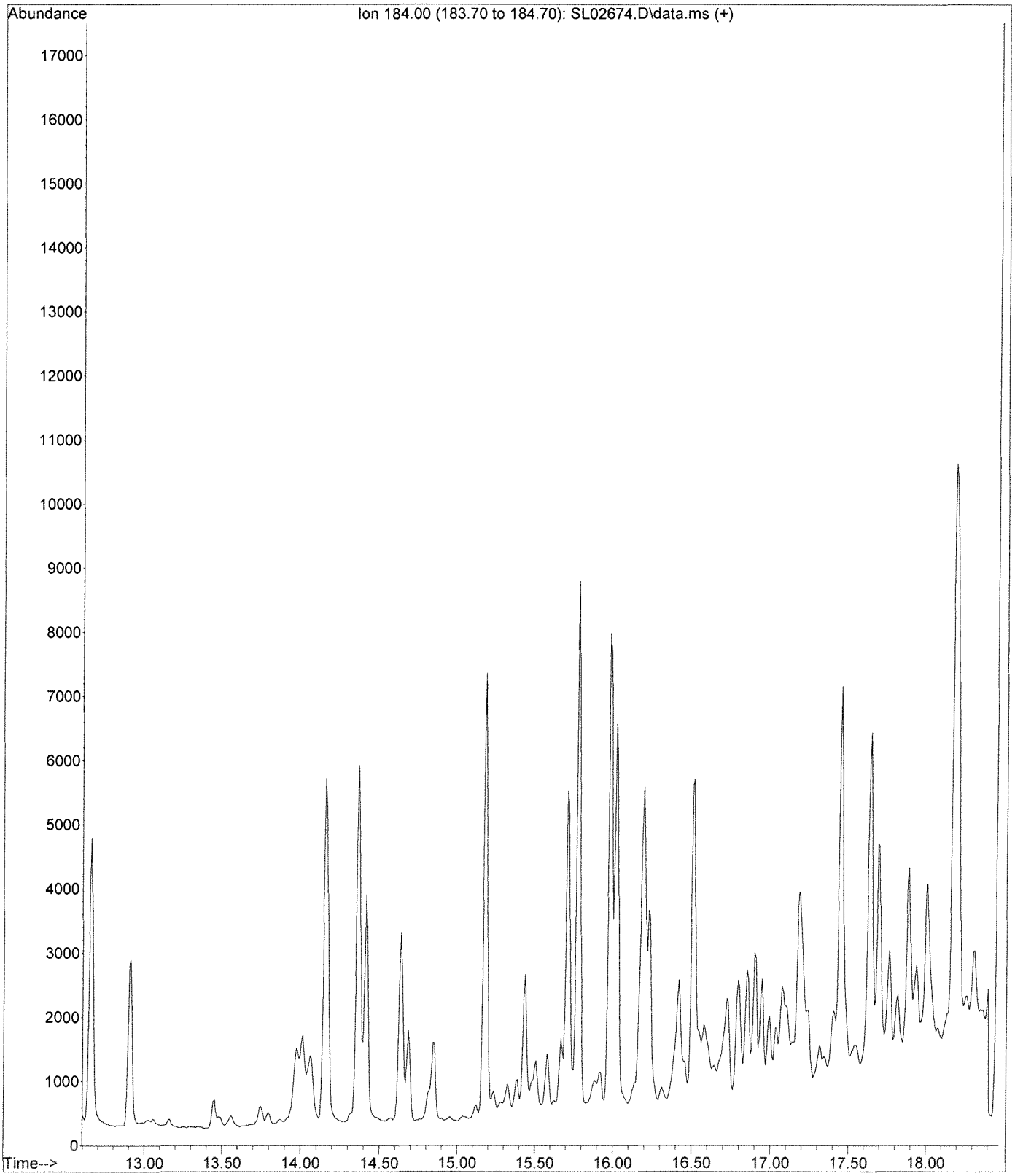
Sample goal

- Can use these fingerprints!

Coal Tar creosote overpowers oil PAH

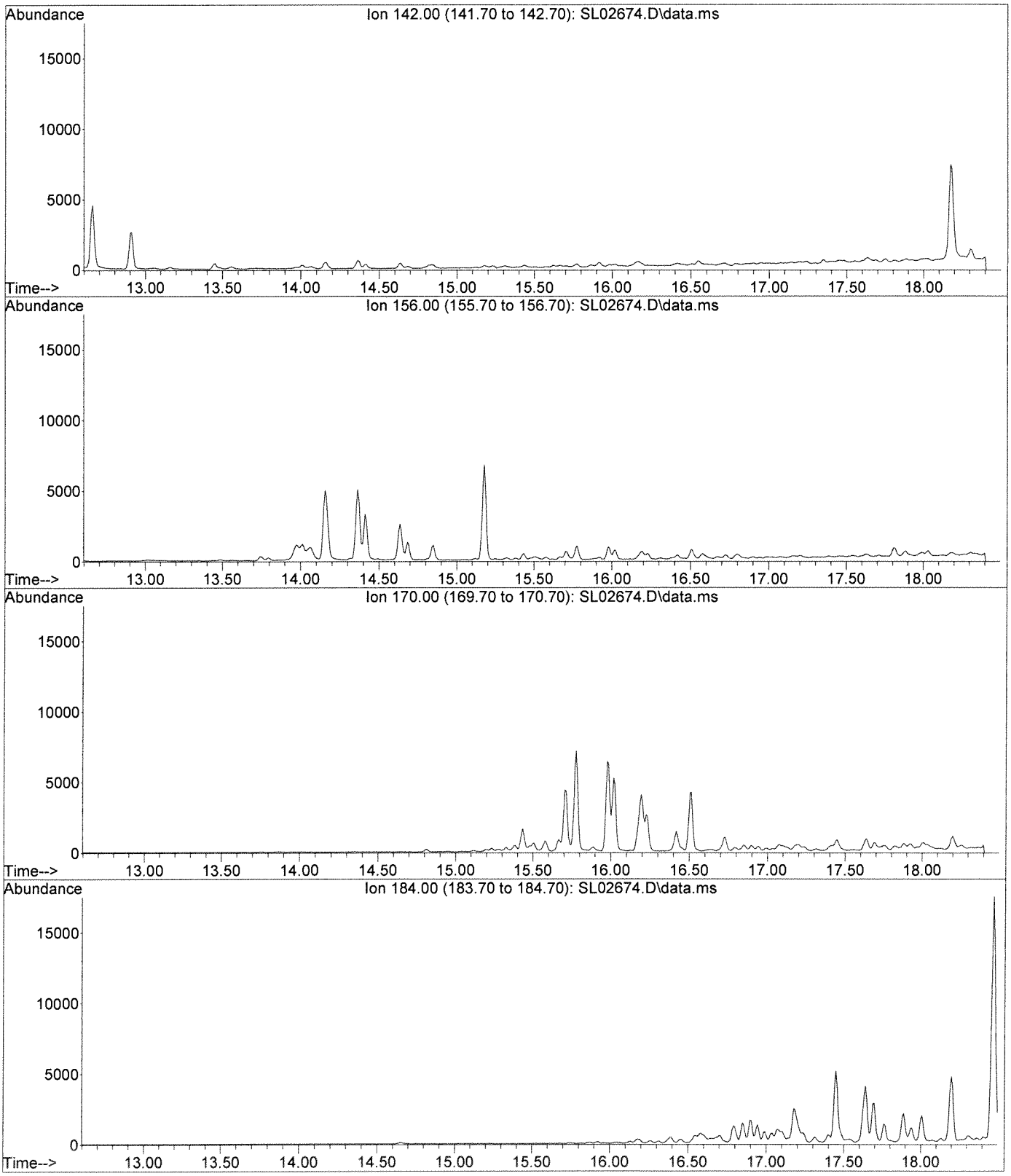
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02674.D
Operator    : Syslo
Acquired    :  2 Feb 2012  19:53      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0001
Misc Info   : 30g to 1.0mL
Vial Number: 82
```



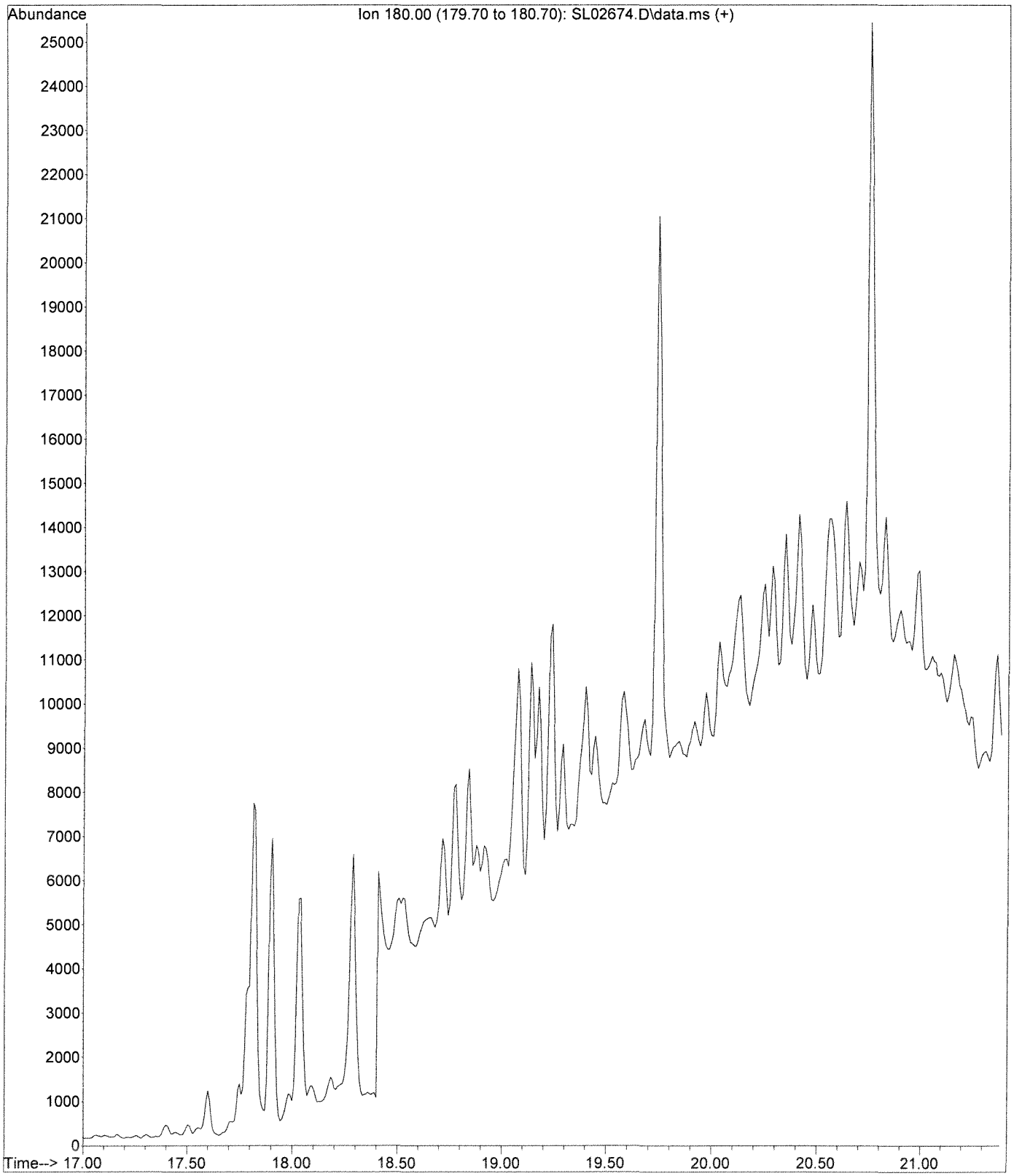
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02674.D
Operator    : Syslo
Acquired    :  2 Feb 2012  19:53      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0001
Misc Info   : 30g to 1.0mL
Vial Number: 82
```



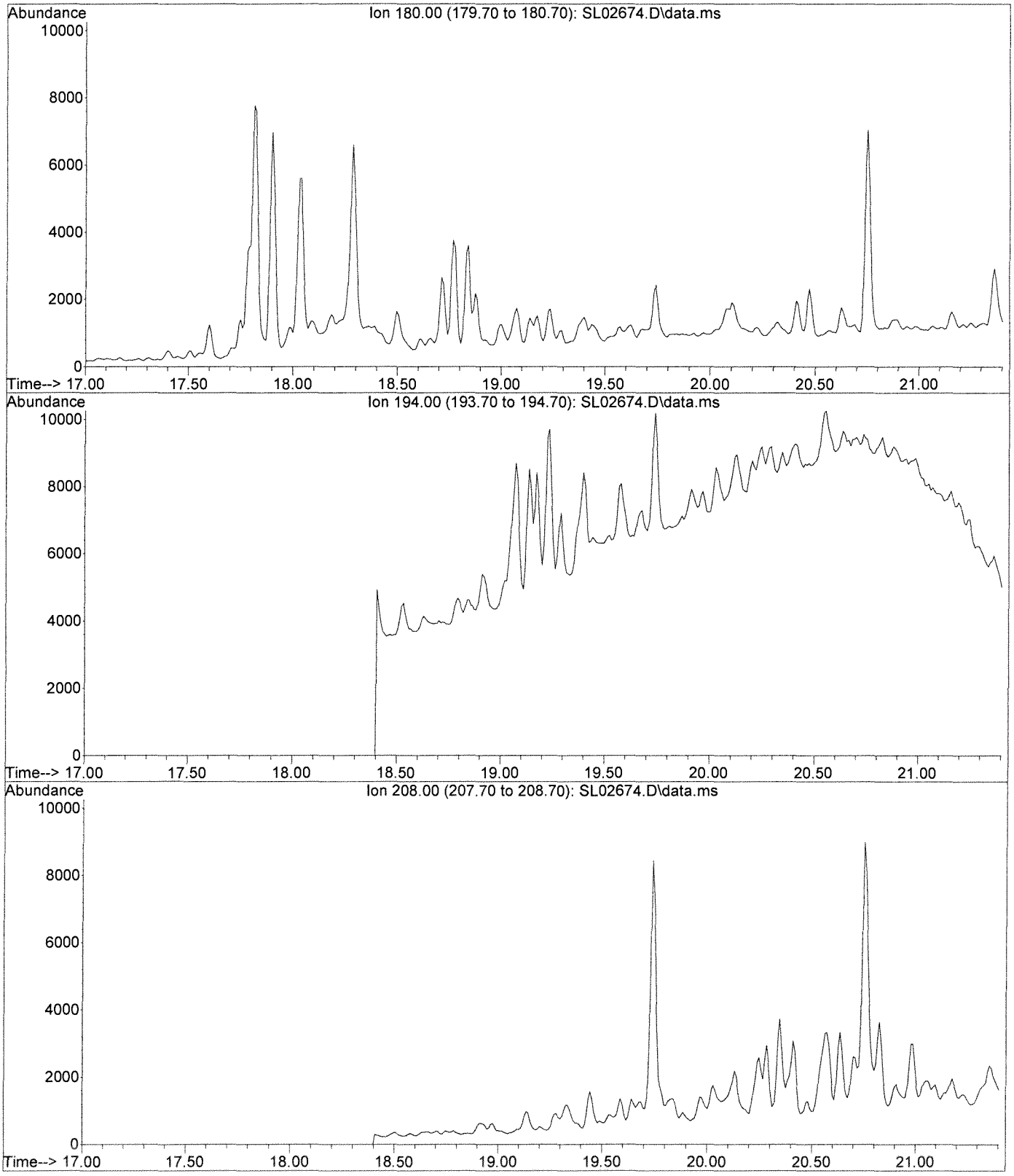
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02674.D
Operator    : Syslo
Acquired    :  2 Feb 2012  19:53      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0001
Misc Info   : 30g to 1.0mL
Vial Number: 82
```



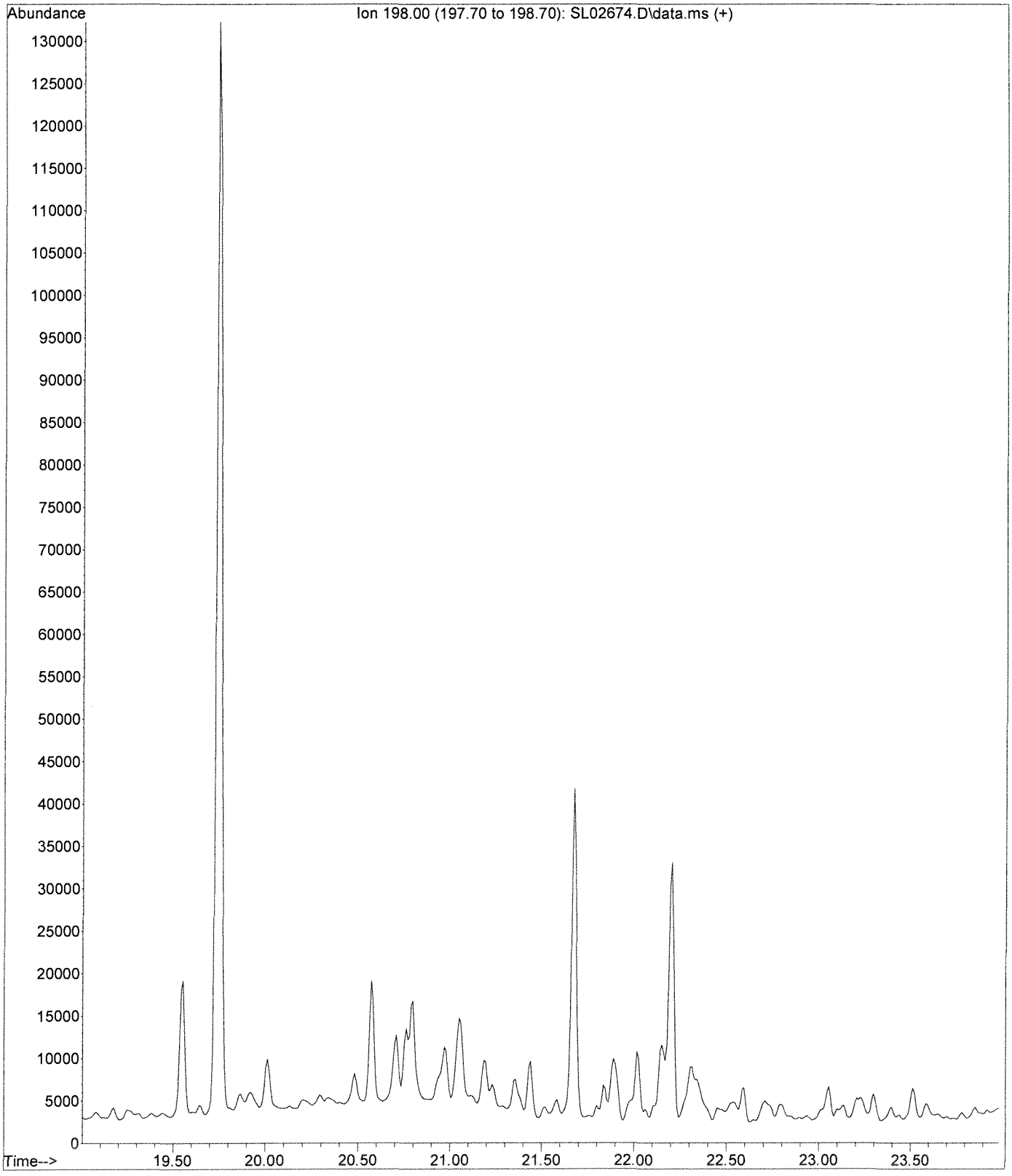
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02674.D
Operator    : Syslo
Acquired    :  2 Feb 2012  19:53      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0001
Misc Info   : 30g to 1.0mL
Vial Number: 82
```



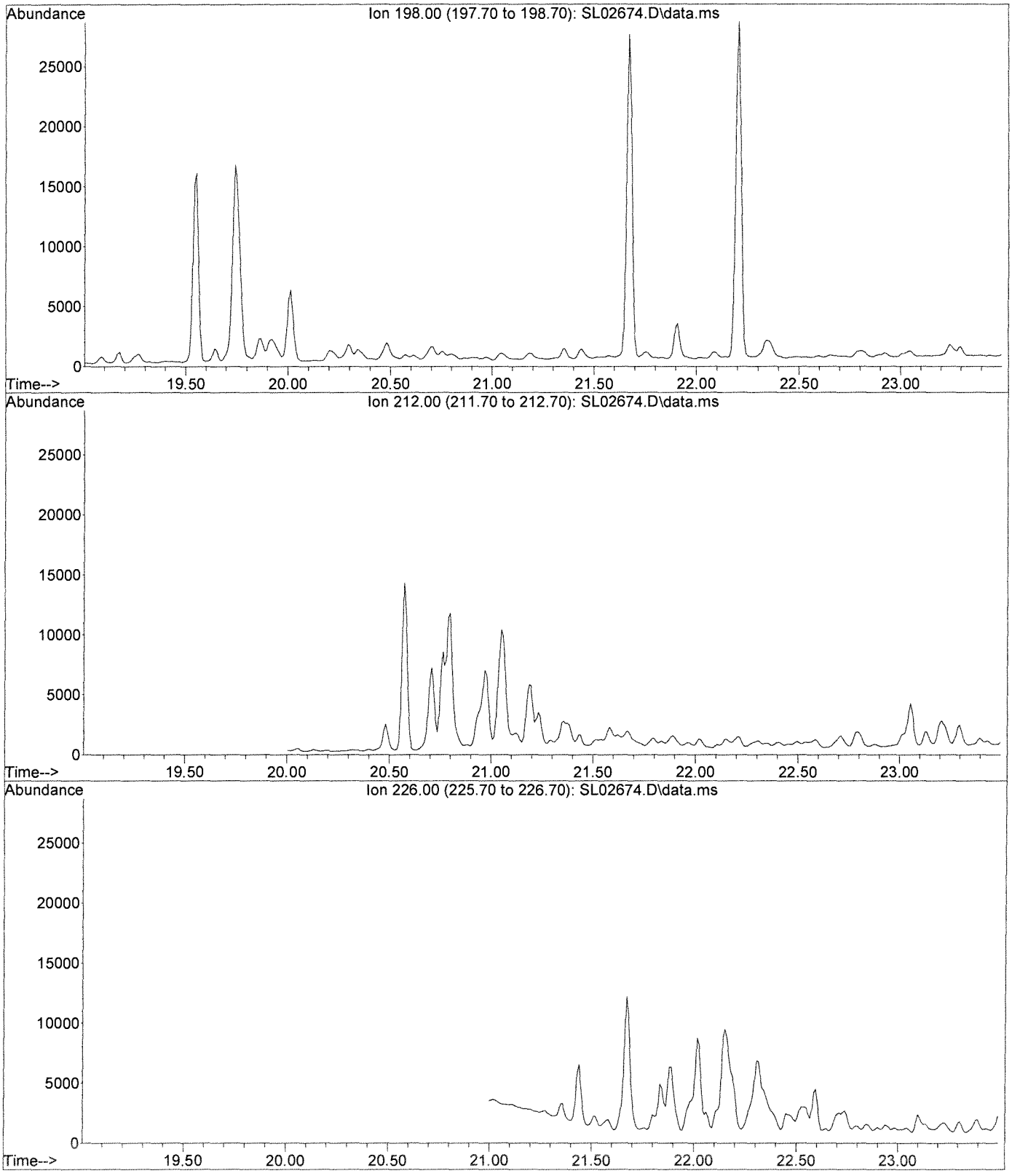
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\020212\SL02674.D
Operator    : Syslo
Acquired    :  2 Feb 2012  19:53      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0001
Misc Info   : 30g to 1.0mL
Vial Number: 82
```



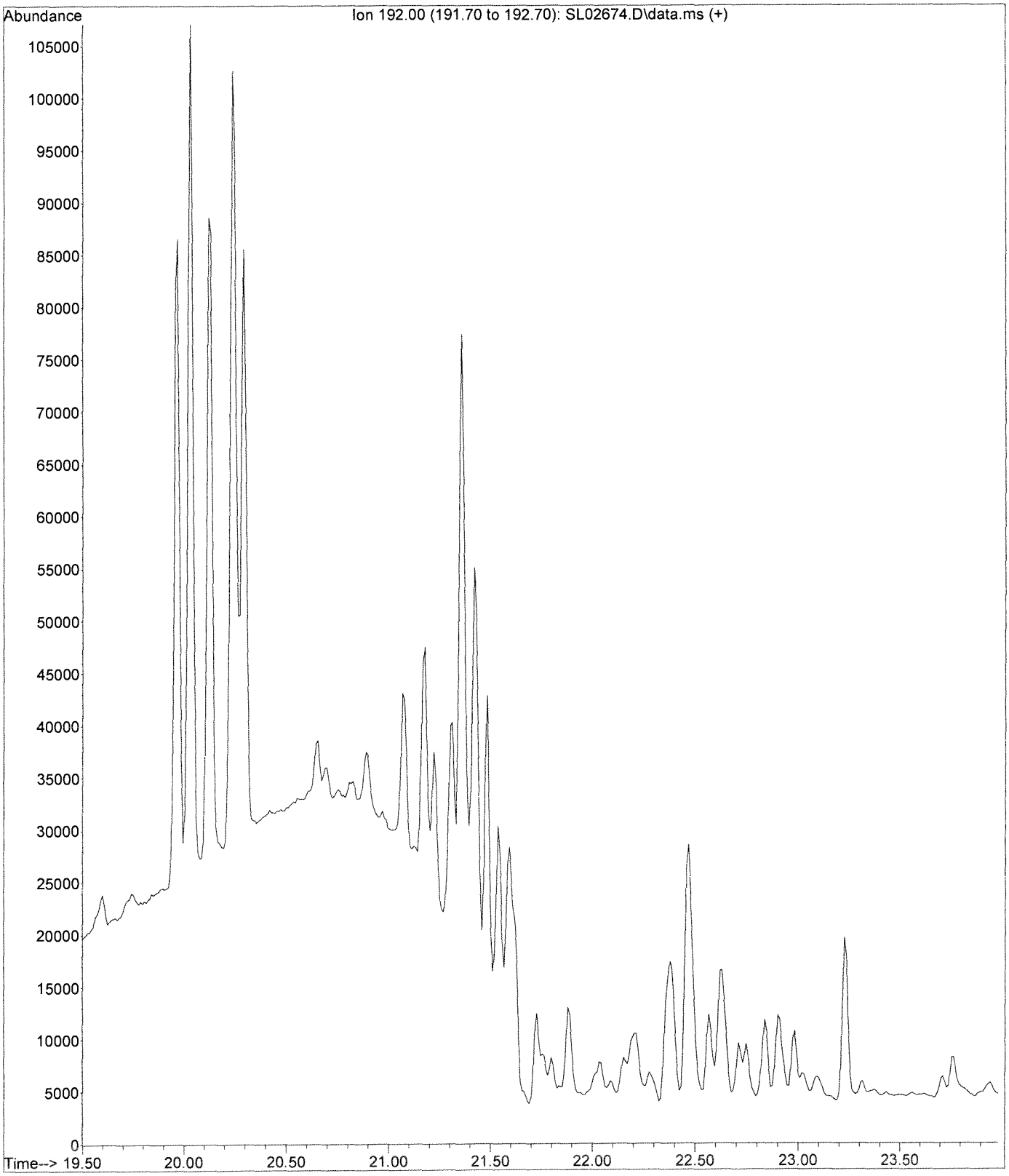
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



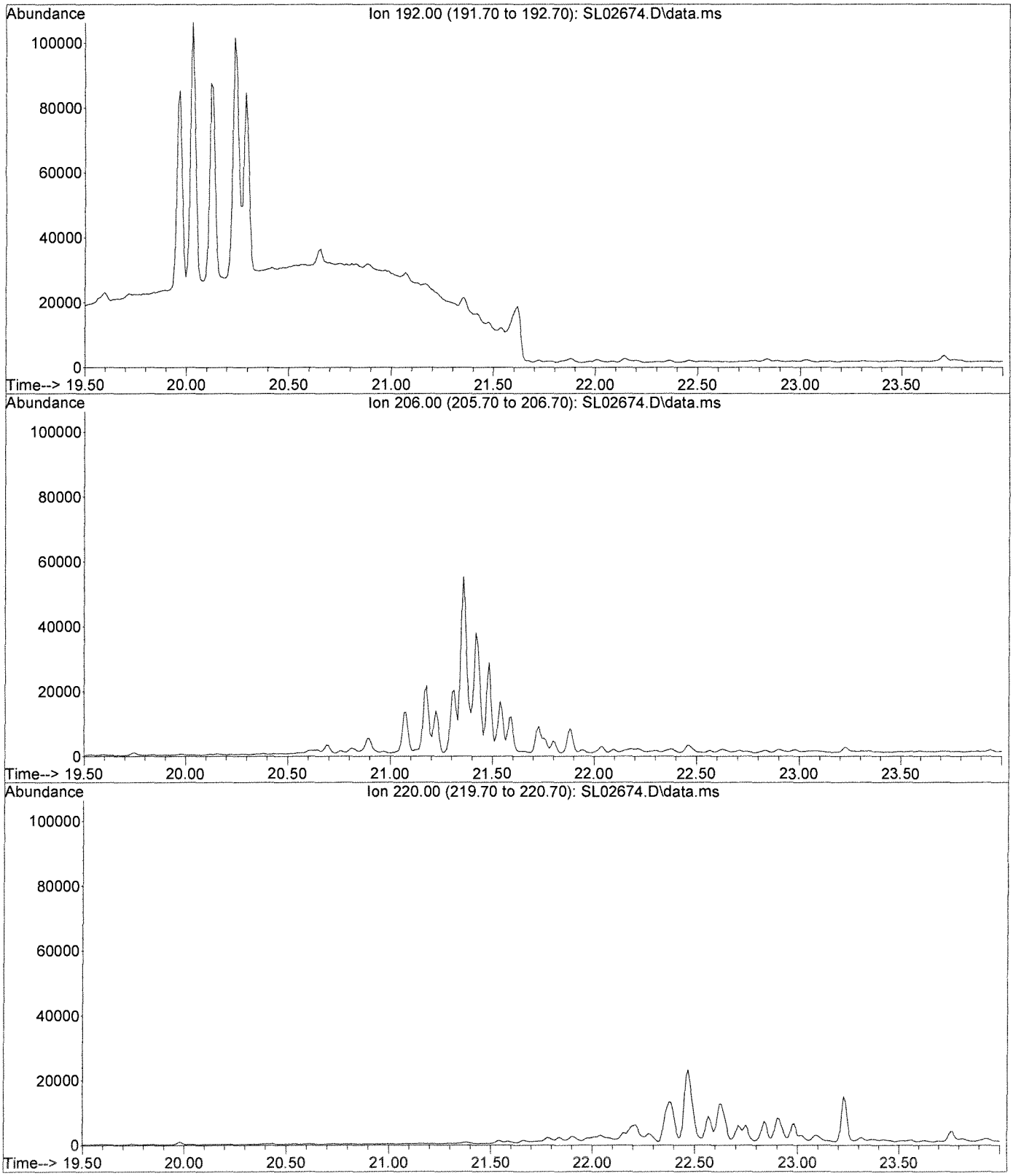
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



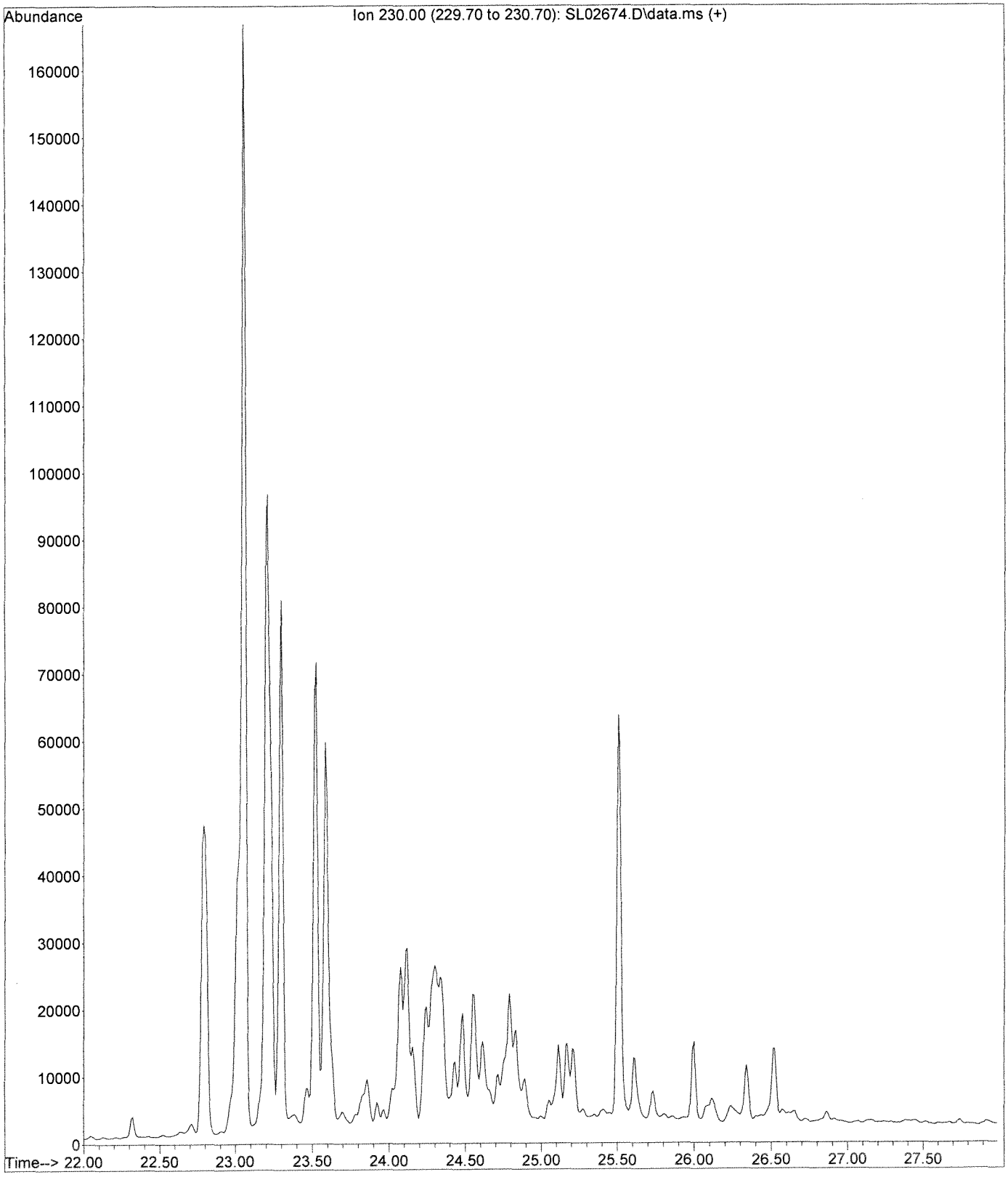
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



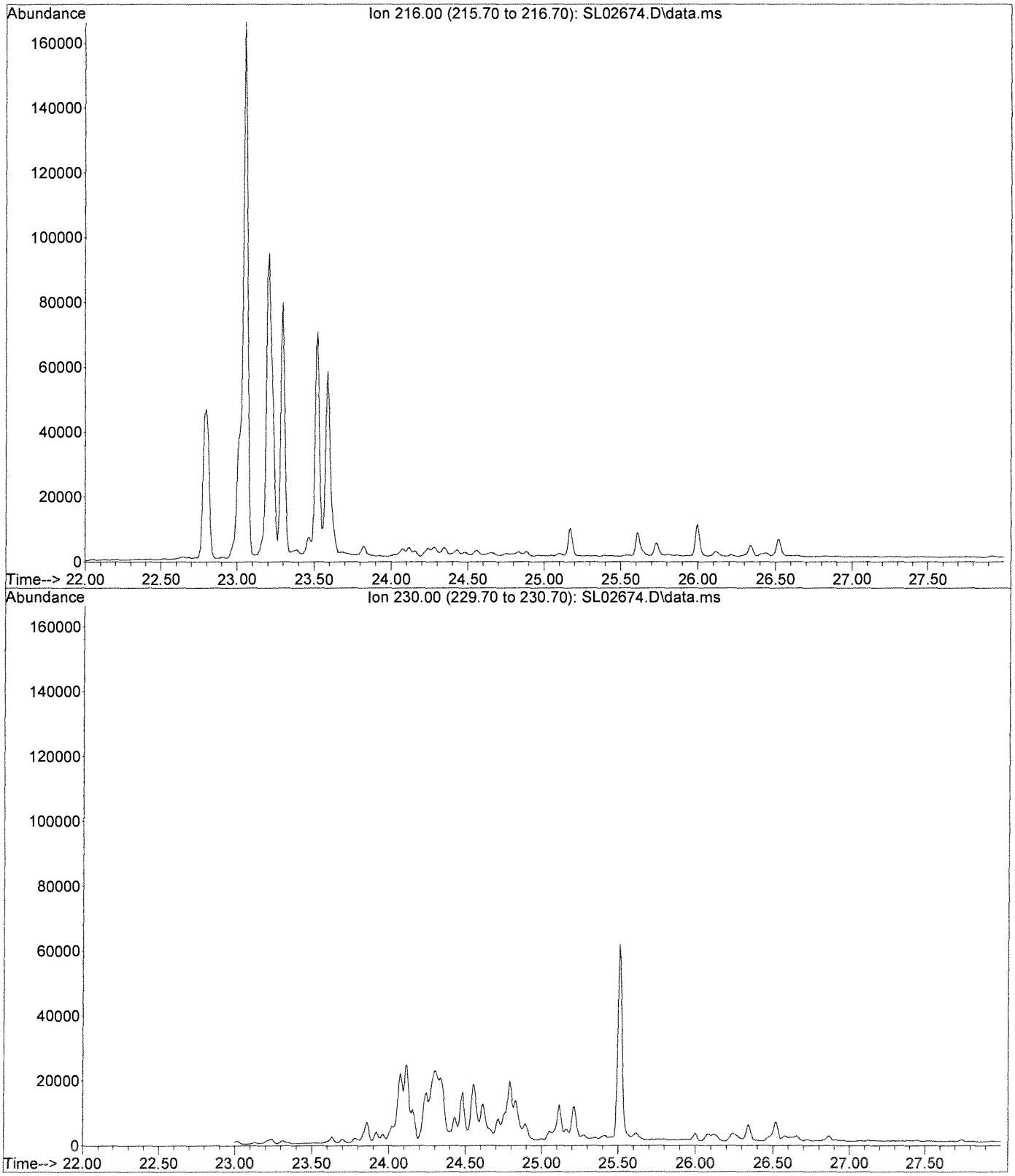
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



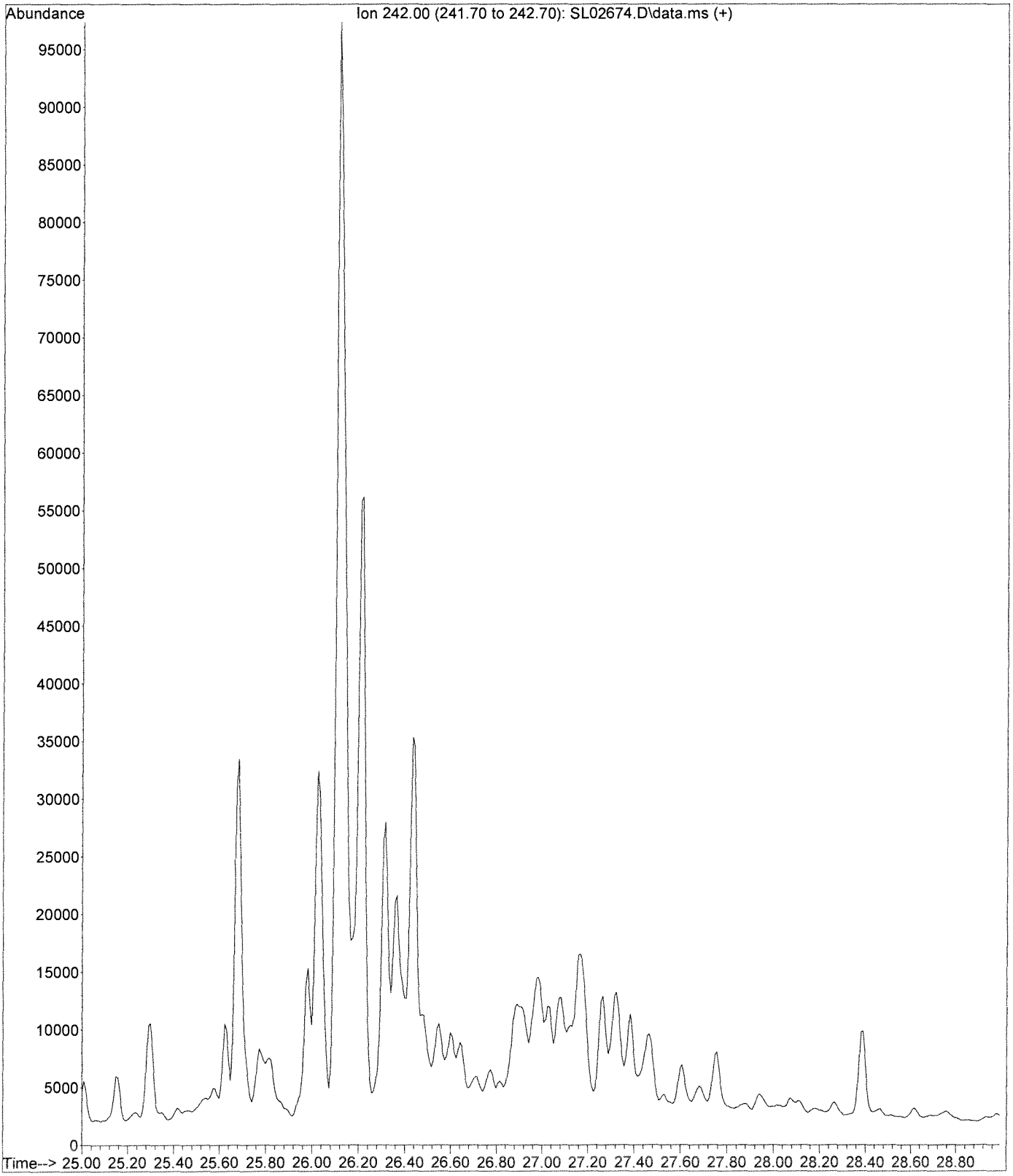
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



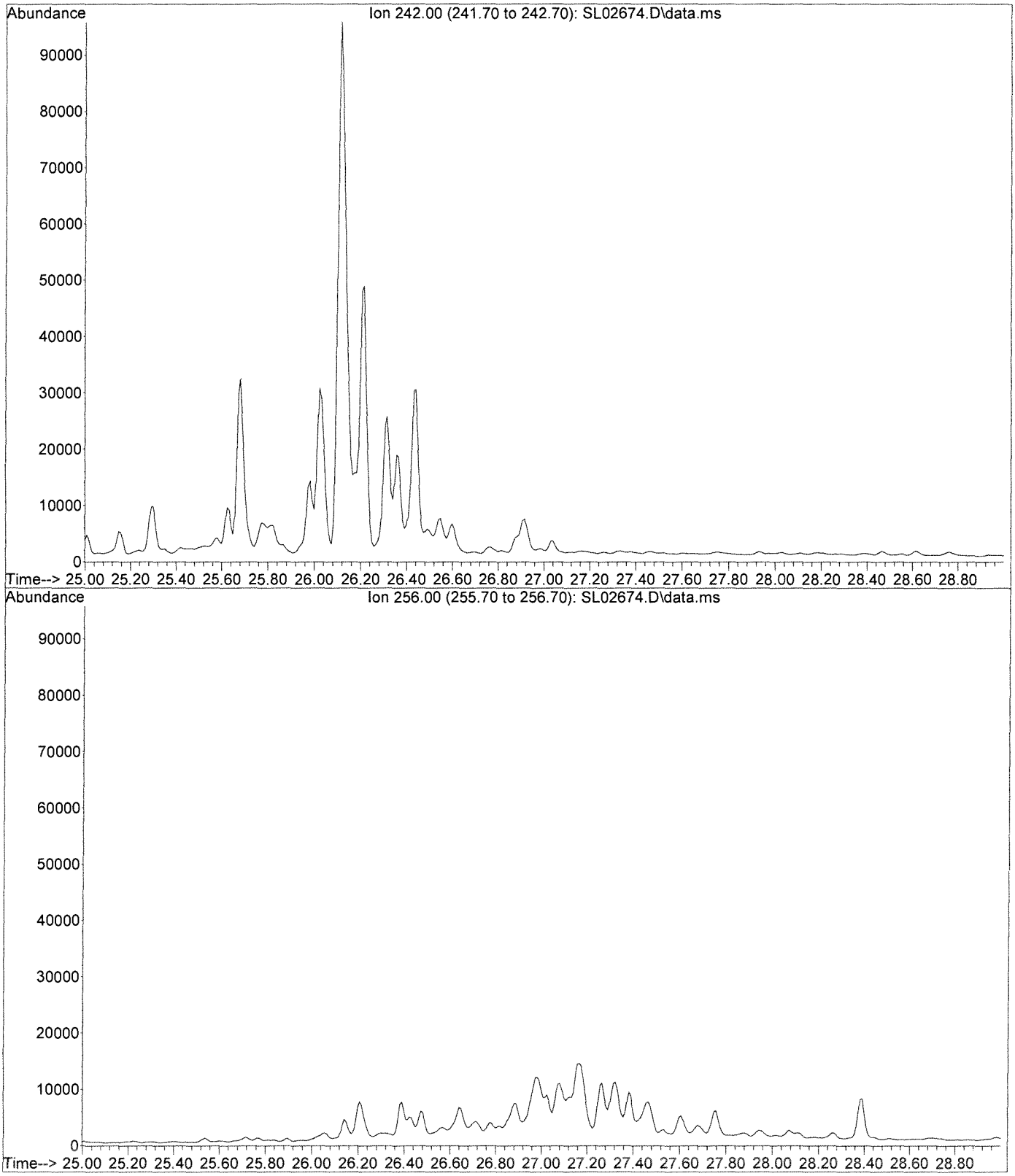
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



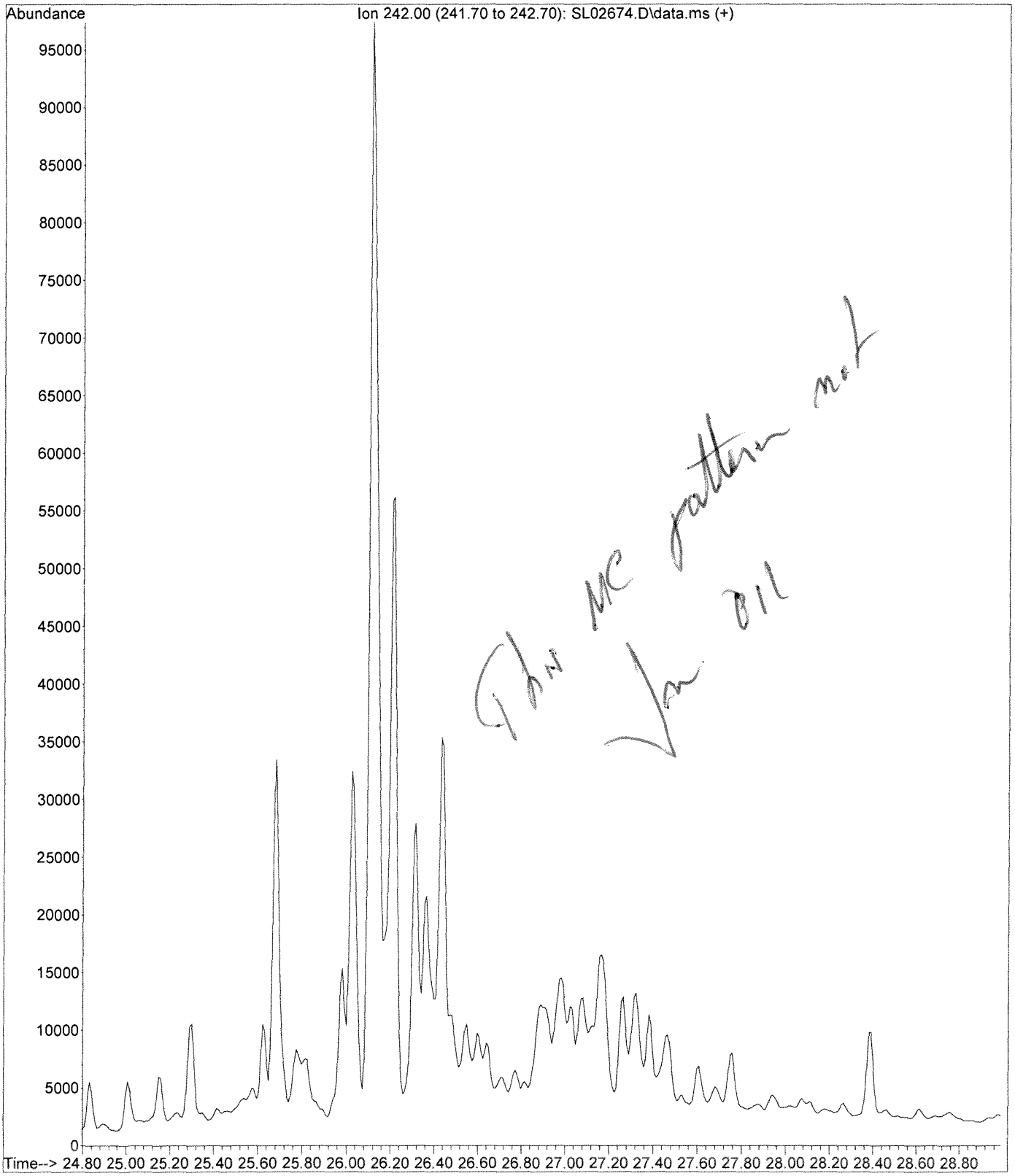
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\020212\SL02674.D
Operator : Syslo
Acquired : 2 Feb 2012 19:53 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0001
Misc Info : 30g to 1.0mL
Vial Number: 82



US EPA ARCHIVE DOCUMENT

TPH DF-0102

Logbook # SERAS-l-0121

Page 84

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092

Project Name: Enbridge Oil | Method File: DFTPP/ DFTPA051611.M

Work Assign.#: WA-017 | Inj. Volume: 1ul

Analysis/Date: TPH 2/15/12 | Analyst: Sygl

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02826	50 ppm DFTPP	RUK0084	2/15/12	12:20	Full 1598-1356
91	SL02827	1000 ppm TPH CCV	RVB0003		12:42	Full
82	SL02828	Soil Blank			13:48	120024-Blk1
83	SL02829	SERAS-017-0002	R202018-01		14:27	
84	SL02830	[illegible]	[illegible]			CCC-DCM
85	SL02831	DCM in Toluene 0.1?			16:07	1ul DCM-1.0ml Tolu
86	SL02832	RVB0026 in Toluene			16:23	0.1g → 5.0ml Toluene
87	SL02833	Toluene Blank			16:55	
88	SL02834	BEO 20 [illegible]			17:16	[illegible] 0.1g → 1.0ml
87	SL02835	Tol. Blank			17:[illegible]	
89	SL02836	BEO [illegible] 0.1g → 8ml			18:01	Rig Rate Var
90	SL02837	BEO [illegible] 2.5K			18:40	
82	SL02838	Soil Blank		2/15/12	19:41	
	SL	end of 2/15/12	Sygnus			DJ
99	SL02839	50 ppm DFTPP	RVB0025	2/16/12	12:21	
1	SL02840	Toluene Blank			12:39	
1	SL02841	Toluene Blank			12:57	
1	SL02842	Tol Blank			13:34	
2	SL02843	RVB0026 DCM - 4ml			16:50	0.05g → 2ml Tol
1	SL02844	Toluene Blank			17:[illegible]	DCM
3	SL02845	Toluene Blank			[illegible]:16	
4	SL02846	BEO [illegible]		2/16/12	18:24	
99	SL02847	50 ppm DFTPP	RVB0025	2/16/12	21:45	
75	SL02848	10K Arabian Crude		2/17/12	22:07	Lo Sal
99	SL02849	50 ppm DFTPP	RVB0025	2/22/12	10:19	
99	SL02850	50 ppm DFTPP	RVB0025		10:37	
98	SL02851	10K ECO SLM	275-05-05		10:59	
98	SL02852	10K BEO Slm			19:33	Liman Acan
99	SL02853	50 ppm DFTP	RVB0025	2/22/12	20:29	

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RUK0084	DFTPP	5/5/12	50ppm		
2	RVB0003	Calibration Check	8/2/12	1000ppm		
3	RVK6080	Internal Standard	5/11/12	500ppm		
4						
5						
6						

Reviewed By: J Pyle screen > ASN Date Checked: 2/24/12

Sediment run for TPA - but extracted Fingerprint info.

US EPA ARCHIVE DOCUMENT

```
   Sequence Name: C:\msdchem\1\sequence\021512.s
         Comment: Enbridge Oil
        Operator: Syslo
       Data Path: C:\MSDCHEM\1\DATA\021512\
Instrument Control  Pre-Seq Cmd:
Data Analysis       Pre-Seq Cmd:

Instrument Control Post-Seq Cmd:
Data Analysis      Post-Seq Cmd:

   Method Sections To Run      On A Barcode Mismatch
    (X) Full Method              (X) Inject Anyway
    ( ) Reprocessing Only        ( ) Don't Inject

   ------------------------------------------------------------------------
   Line                     Sample Name/Misc Info
 1)  Sample        99   SL02826  DFT8270D 50 DFTPP
 2)  Sample        71   1.0k TPH CCV
     Datafile           SL02827
     Method             DROTPH051611
 3)  Sample        82   Soil Blank
     Datafile           SL02828
     Method             DROTPH051611
 4)  Sample        83   SERAS-017-0001
     Datafile           SL02829
     Method             DROTPH051611
```

Injection Log

Data Directory: C:\msdchem\1\DATA\021512\

SampleName	MiscInfo	Vial	Multiplier	Injection Time
1) SL02826.D				
50 DFTPP	RUK0084	99	1.000	15 Feb 2012 12:20
2) SL02827.D				
1.0k TPH CCV	RVB0003	71	1.000	15 Feb 2012 12:42
3) SL02828.D				
Soil Blank	1200024-Blk1	82	1.000	15 Feb 2012 13:43
4) SL02829.D				
SERAS-017-0002	30g to 1.0mL	83	1.000	15 Feb 2012 14:27

with 12h

GC/MS QA-QC Check Report: 02/15/12

Tune File : C:\msdchem\1\DATA\021512\SL02826.D
Tune Time : 15 Feb 2012 12:20

Daily Calibration File : C:\msdchem\1\DATA\021512\SL02827.D

File	Sample	Surrogate Recovery %				Internal Standard Responses		
SL02827.D								
	1.0k TPH C	51*	55*	59*	57*	602225	179024	220786
				162003				
SL02828.D								
	Soil Blank	56*	61*	66*	72*	455645	139126	159054
				106564				
SL02829.D								
	SERAS-017-	58*	62*	65*	70*	419430	129686	144537
				130132				

Created: Thu Jan 10 09:40:17 2013 Slick2

oh

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

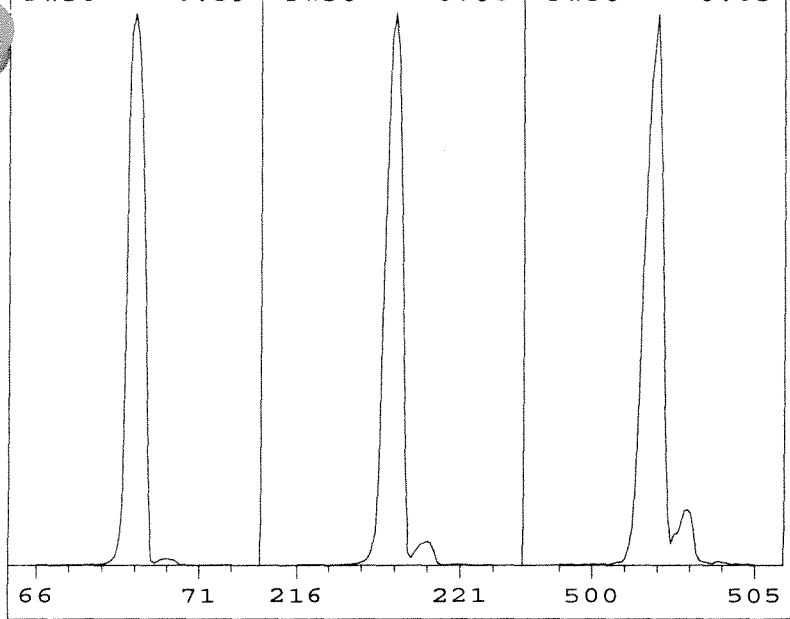
- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

Wed Feb 15 12:13:01 2012 Instrument: Slick2
C:\msdchem\1\5975\Xtune.u US90432092

Mass	69.00	Mass	219.00	Mass	502.00
Ab	100272	Ab	73200	Ab	2171
Pw50	0.59	Pw50	0.60	Pw50	0.65

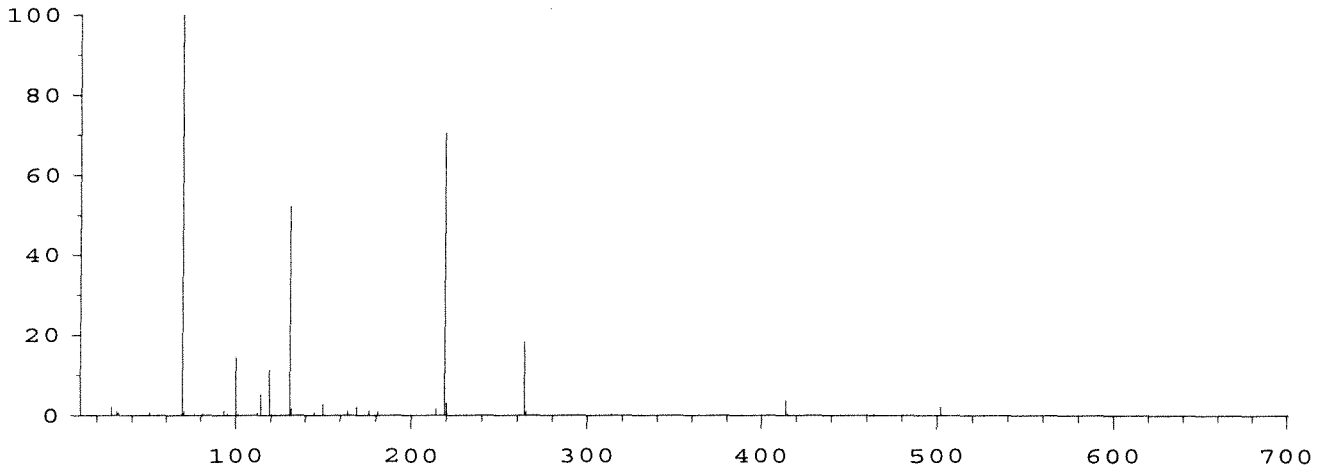


Ion Pol	Pos	MassGain	-629
		MassOffs	-37
Emission	34.6	AmuGain	1857
EIEnrgy	69.9	AmuOffs	123.31
Filament	1	Wid219	-0.018
		DC Pol	Pos
Repeller	25.43		
IonFcus	80.7	HEDEnab	On
EntLens	0.0	EMVolts	1553
EntOffs	Var		
		Samples	8
PFTBA	Open	Averages	5
		Stepsize	0.10

Temperatures and Pressures:

MS Source	280	TurboSpd	100
MS Quad	190	HiVac	1.65e05

Scan: 10.00 - 701.00 Samples: 8 Thresh: 100 Step: 0.10
52 peaks Base: 69.00 Abundance: 98008



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
69.00	98008	100.00	70.00	1075	1.10
219.00	69000	70.40	220.00	3005	4.36
502.00	2069	2.11	502.90	212	10.25

Air/Water Check: H2O~0.14% N2~2.15% O2~0.69% CO2~0.00% N2/H2O~1517.27%

Ramp Criteria:
Ion Focus Maximum 110 volts using ion 502; EM Gain 16016
Repeller Maximum 35 volts using ion 502; Gain Factor 0.16

MassGain Values(Samples): -619(3) -608(2) -591(1) -551(0) -464(FS)

TARGET MASS:	50	69	131	219	414	502	1050
Amu Offset:	123.3	123.3	123.3	123.3	123.3	123.3	123.3
Entrance Lens Offset:	18.0	15.8	14.2	15.5	15.8	14.8	17.0
Target Abund(%):	1.0	100.0	45.0	55.0	3.5	2.0	
Actual Tune Abund(%):	0.7	100.0	52.3	70.4	3.7	2.3	

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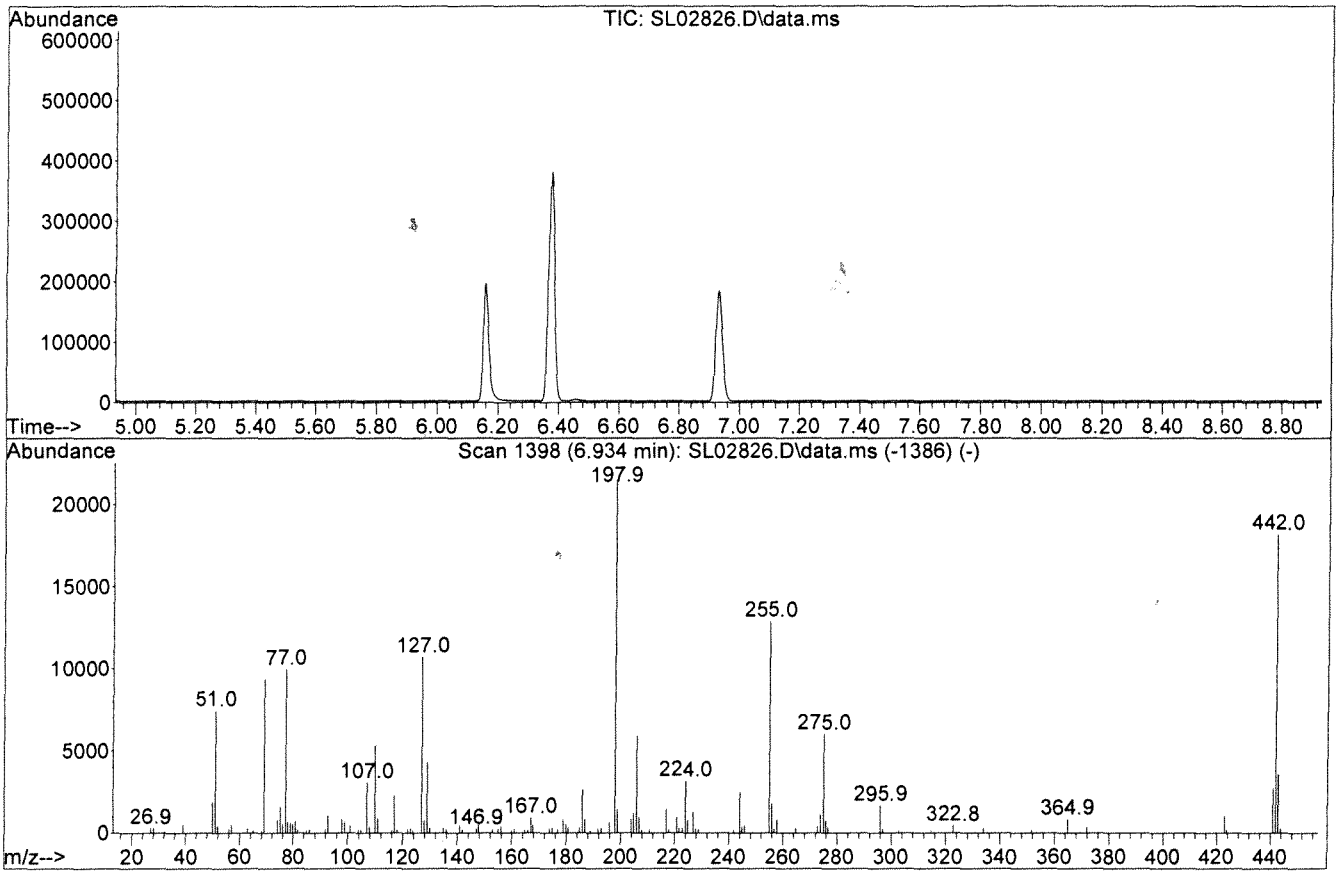
DFTPP - 8270D

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02826.D
Acq On : 15 Feb 2012 12:20
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Tailing PCP 1.31
Benzidine 1.51
DDT deg = 0.63

Integration File: rteint.p

Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Title : DFTPP Method with 8270D criteria: 8/20/10
Last Update : Thu Jan 26 14:45:44 2012



Spectrum Information: Scan 1398

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	34.6	7431	PASS
68	69	0.00	2	1.6	152	PASS
69	198	0.00	100	43.6	9368	PASS
70	69	0.00	2	0.0	0	PASS
127	198	10	80	49.8	10705	PASS
197	198	0.00	2	0.0	0	PASS
198	198	100	100	100.0	21480	PASS
199	198	5	9	6.6	1418	PASS
275	198	10	60	27.9	5996	PASS
365	198	1	100	3.9	829	PASS
441	442	0.01	24	15.0	2737	PASS
442	198	50	100	84.8	18216	PASS
443	442	15	24	19.5	3560	PASS

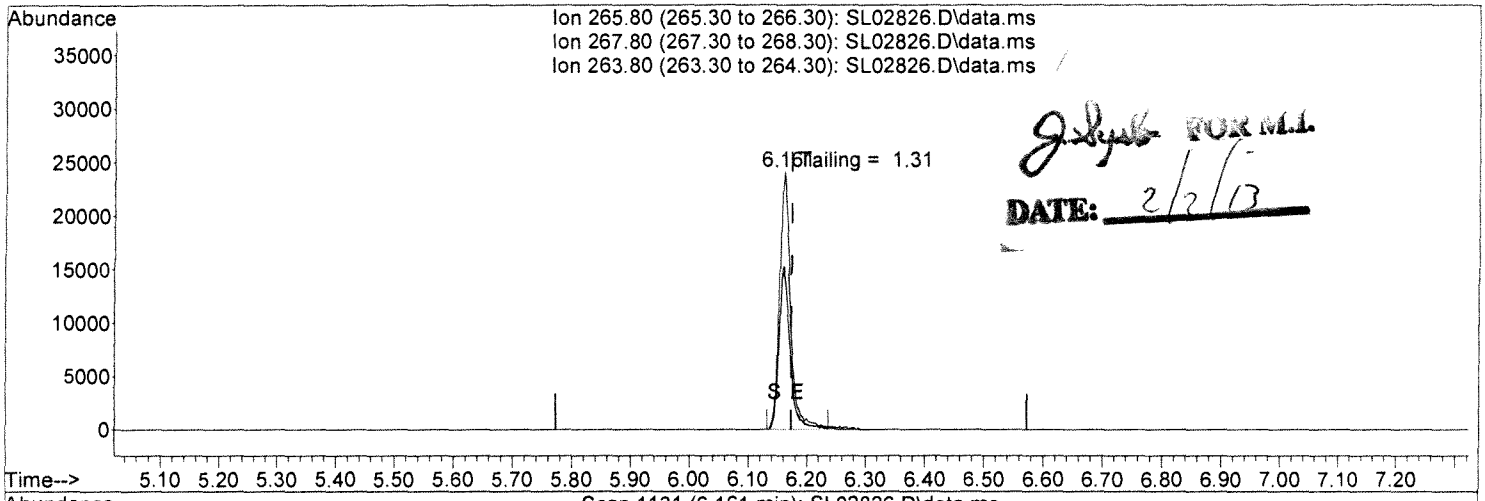
SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

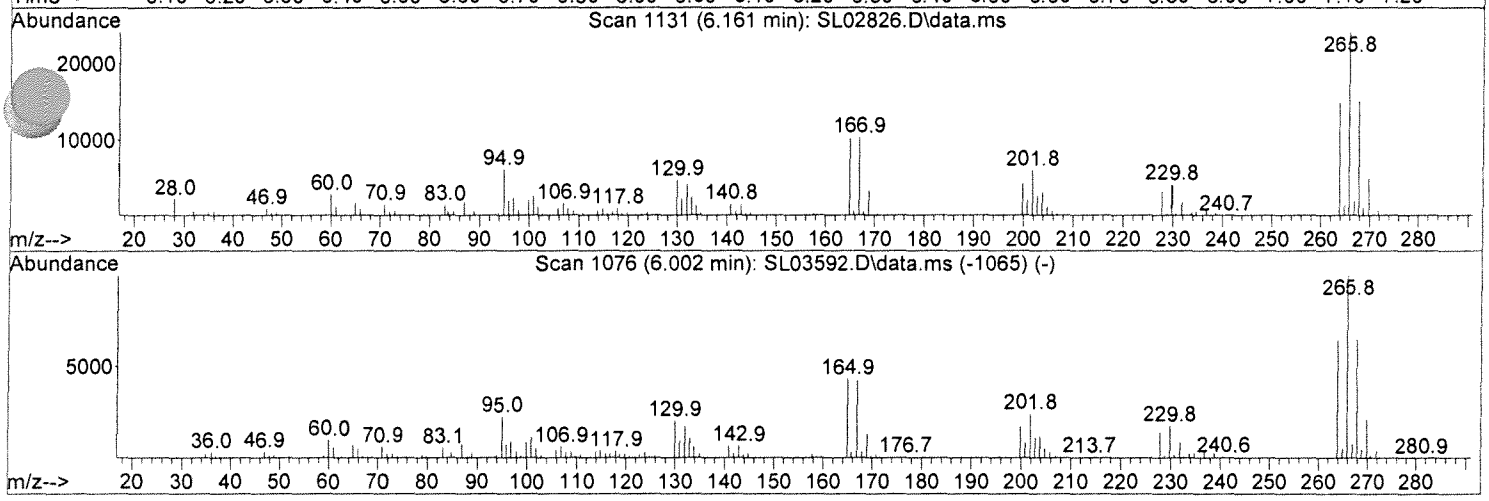
Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02826.D
Acq On : 15 Feb 2012 12:20
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 15 12:32:33 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



FOR M.L.
DATE: 2/2/13



TIC: SL02826.D\data.ms

(2) Pentachlorophenol

6.161min (-0.012) 45.36 ug/mL

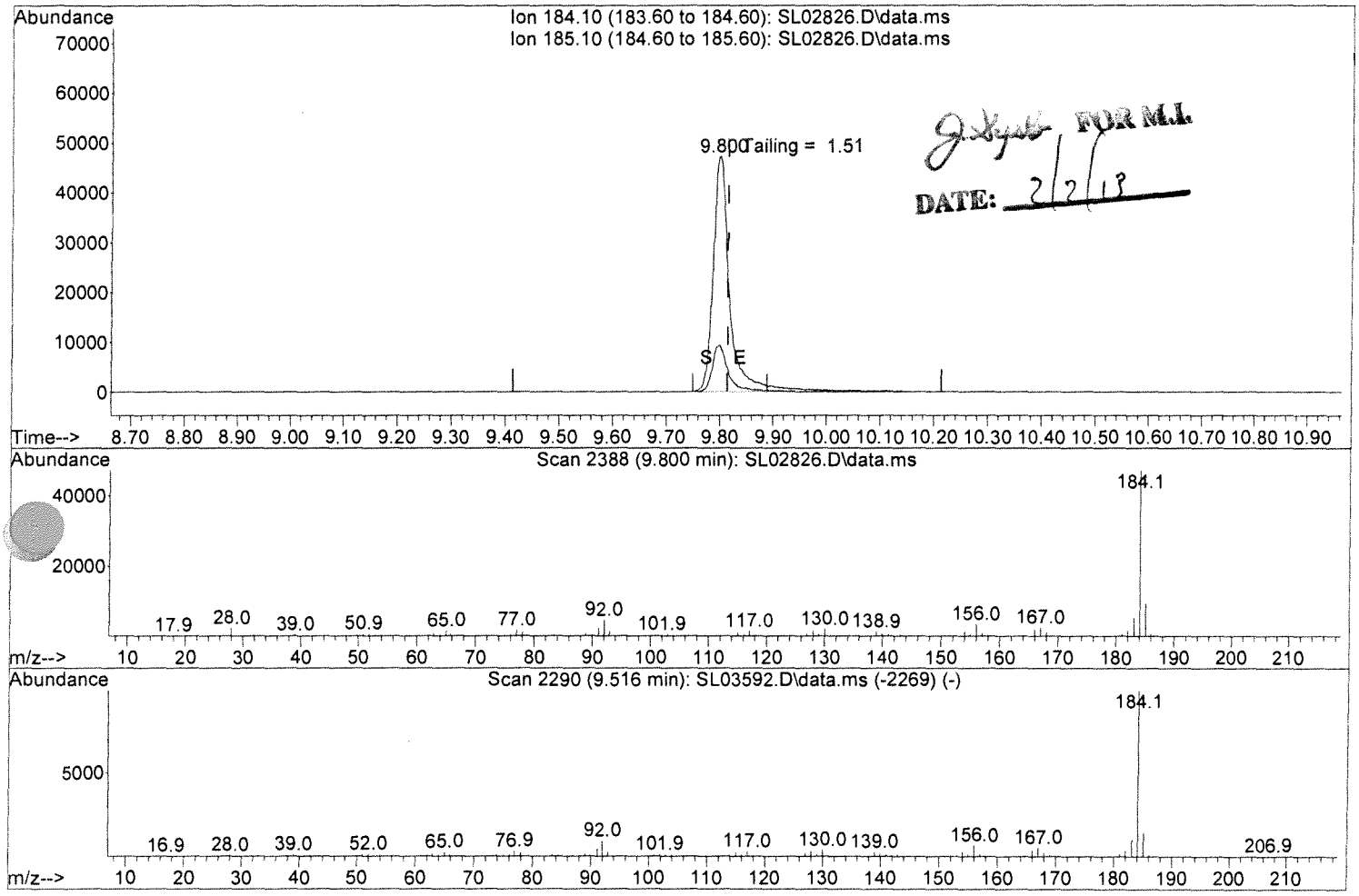
response 32028

Ion	Exp%	Act%
265.80	100	100
267.80	64.50	63.06
263.80	63.60	62.24
0.00	0.00	0.00

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02826.D
Acq On : 15 Feb 2012 12:20
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 15 12:32:33 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



J. Kyle FOR M.I.
DATE: 2/2/13

TIC: SL02826.D\data.ms

(5) Benzidine

9.800min (-0.015) 35.06 ug/mL

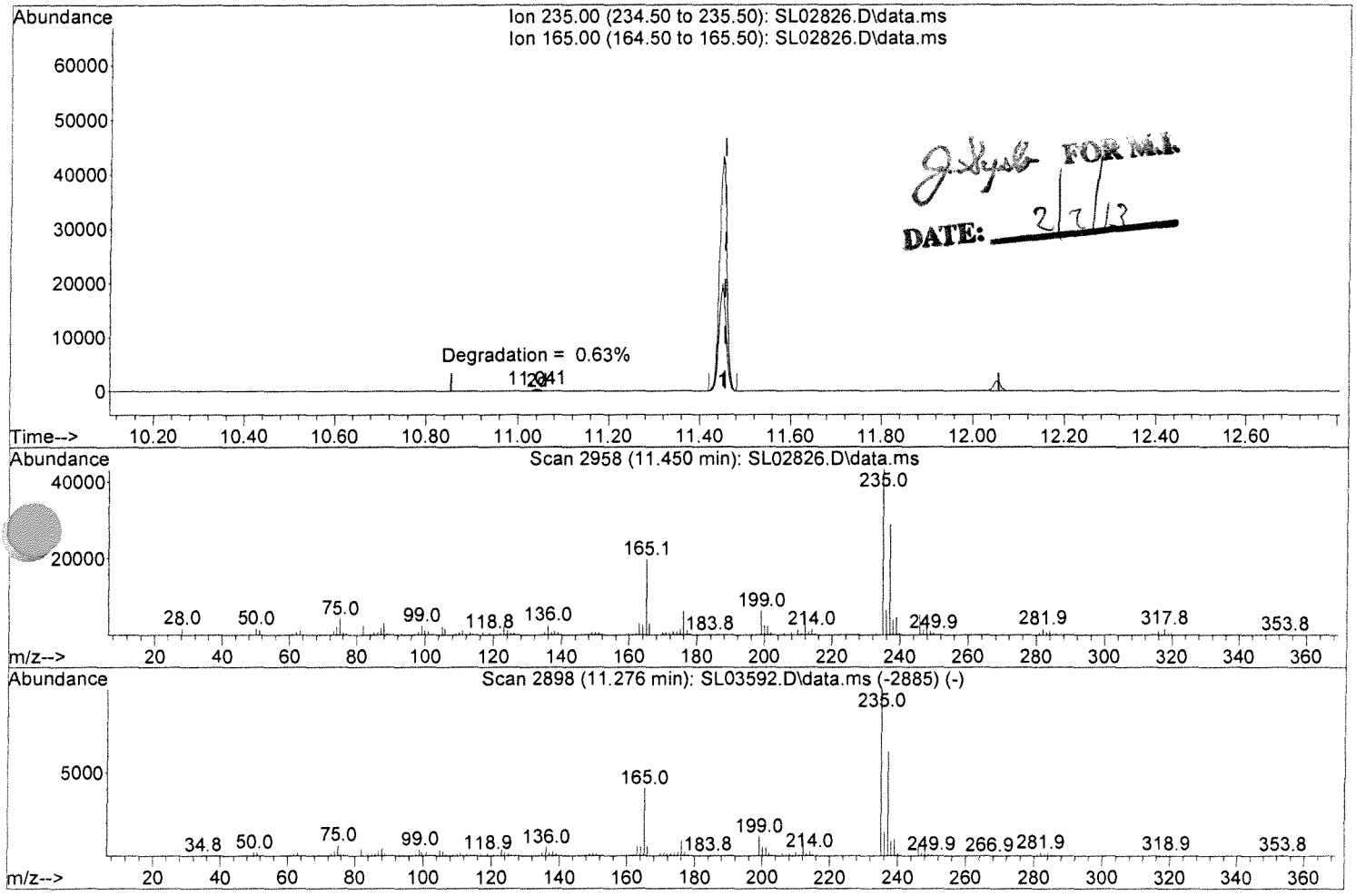
response 98122

Ion	Exp%	Act%
184.10	100	100
185.10	14.60	19.17
0.00	0.00	0.00
0.00	0.00	0.00

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02826.D
Acq On : 15 Feb 2012 12:20
Operator : Syslo
Sample : 50 DFTPP
Misc : RUK0084
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 15 12:32:33 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



J. Syslo FOR M.L.
DATE: 2/7/13

TIC: SL02826.D\data.ms

(6) DDT

11.450min (-0.006) 38.78 ug/mL

response 48284

Ion	Exp%	Act%
235.00	100	100
165.00	51.80	43.93
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

TPH Daily Calibration Check Results
#2 Diesel TPH Calibration Range - Using SERAS GC/MS Method 1803

Calibration Check Date	ICAL Date	Project	Wa#	GC/MS Datafile	TPH Response	d30-IS Area	d50-IS Area	d74-IS Area	Cal Check RF	Calibration Average RF	% Diff.
05/17/11	05/16/11	WSoil MDL	0-011	SL01889	7932426	132429	130508	51192	0.75756	0.72959	3.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01942	7860351	128757	120362	11883	0.90348	0.72959	23.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01945	6544592	104943	128978	88104	0.60970	0.72959	16.43
07/08/11	05/16/11	CUC-Power Plant	0-135	SL01970	6848120	109628	127366	77204	0.65387	0.72959	10.38
07/11/11	05/16/11	CUC-Power Plant	0-135	SL01974	7184921	121573	136941	70557	0.65502	0.72959	10.22
07/12/11	05/16/11	CUC-Power Plant	0-135	SL01989	7699927	130026	148583	96240	0.61624	0.72959	15.54
07/27/11	05/16/11	CUC-Power Plant	0-135	SL02012	5526770	90684	107174	65457	0.62968	0.72959	13.69
08/01/11	05/16/11	CUC-Power Plant	0-135	SL02036	7282274	117359	139028	90753	0.62934	0.72959	13.74
08/08/11	05/16/11	Move-Maintenance	0-011	SL02067	20772937	342644	403655	197034	0.66062	0.72959	9.45
08/08/11	05/16/11	Move-Maintenance	0-011	SL02069	9013310	151400	176386	86475	0.65273	0.72959	10.54
08/10/11	05/16/11	Move-Maintenance	0-011	SL02071	8539256	140844	161739	77290	0.67438	0.72959	7.57
08/11/11	05/16/11	TPH Soil MDL	0-011	SL02079	8559600	142237	165518	79165	0.66367	0.72959	9.03
08/12/11	05/16/11	TPH Waterl MDL	0-011	SL02093	8541528	140220	170089	76028	0.66327	0.72959	9.09
08/23/11	05/16/11	CUC-Power Plant	0-135	SL02133	8279388	130044	151992	63182	0.71949	0.72959	1.38
08/24/11	05/16/11	CUC-Power Plant	0-135	SL02166	6757179	110307	122438	62687	0.68617	0.72959	5.95
09/21/11	05/16/11	CUC-Power Plant	0-135	SL02202	11190934	169011	205927	119691	0.67875	0.72959	6.97
09/22/11	05/16/11	CUC-Power Plant	0-135	SL02213	8986654	146134	179078	114918	0.61255	0.72959	16.04
02/02/12	05/16/11	Enbridge Oil	0-017	SL02671	10771363	175782	209958	153567	0.59918	0.72959	17.87
02/15/12	05/16/11	Enbridge Oil	0-017	SL02827	11689228	179024	220786	162003	0.62419	0.72959	14.45

PH response based on the sum of all peaks in the DRO (m/z 85 + 83 + 105 + 113 + 123 + 183) pattern from ~8min to 28min.

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02827.D
Acq On : 15 Feb 2012 12:42
Operator : Syslo
[illegible]mple : 1.0k TPH CCV
[illegible]sc : RVB0003
[illegible]S Vial : 71 Sample Multiplier: 1

Quant Time: Feb 15 13:33:01 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	602225	10.00	ug/mL	-0.02
5) d30-Tetradecane	13.540	66	179024	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	220786	10.00	ug/mL	-0.02
8) d74-Hexatriacontane	34.612	66	162003	10.00	ug/mL	-0.04
System Monitoring Compounds						
2) d10-Anthracene {S}	18.831	188	649341	10.14	ug/mL	0.00
Spiked Amount 20.000			Recovery = 50.70%			
3) d14-o-Terphenyl {S}	19.733	244	308159	10.95	ug/mL	-0.02
Spiked Amount 20.000			Recovery = 54.75%			
4) 5a-Androstane {S}	21.150	260	40473	11.82	ug/mL	0.00
Spiked Amount 20.000			Recovery = 59.10%			
7) d62-Triacontane {S}	28.164	66	213472	11.34	ug/mL	0.00
Spiked Amount 20.000			Recovery = 56.70%			
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

11689228

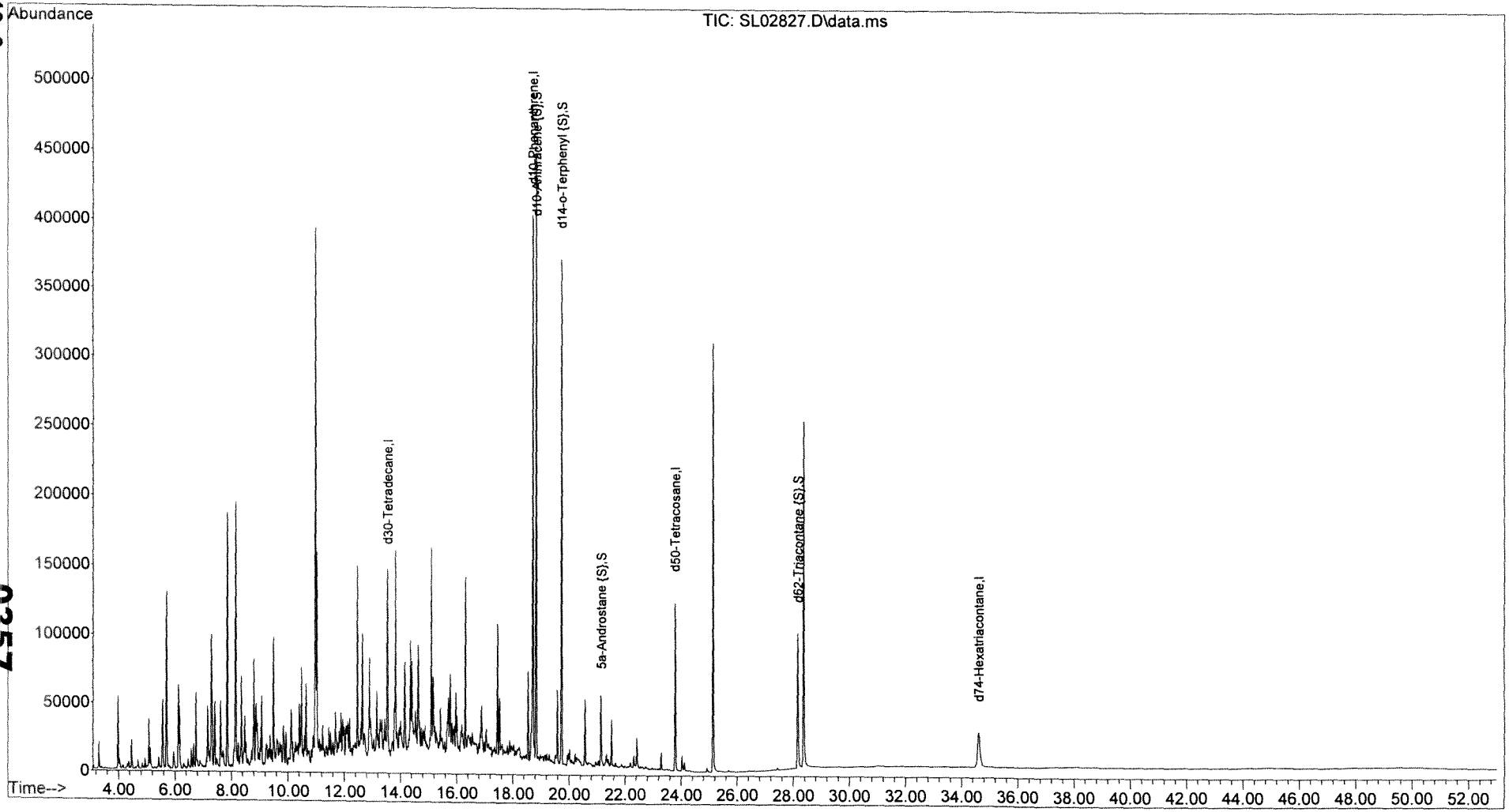
US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02827.D
Acq On : 15 Feb 2012 12:42
Operator : Syslo
Sample : 1.0k TPH CCV
Misc : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 15 13:33:01 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration



0357

Evaluate Continuing Calibration Report

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02827.D
Acq On : 15 Feb 2012 12:42
Operator : Syslo
Sample : 1.0k TPH CCV
Misc : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 15 13:33:01 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

For Surr

Min. RRF : 0.010 Min. Rel. Area : 50% Max. R.T. Dev 0.50min
Max. RRF Dev : 30% Max. Rel. Area : 200%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	d10-Phenanthrene	1.000	1.000	0.0	125	-0.02
2	S	d10-Anthracene {S}	1.063	1.078	-1.4	125	0.00
3	S	d14-o-Terphenyl {S}	0.467	0.512	-9.6	128	-0.02
4	S	5a-Androstane {S}	0.057	0.067	-17.5	136	0.00
5	I	d30-Tetradecane	1.000	1.000	0.0	114	0.00
6	I	d50-Tetracosane	1.000	1.000	0.0	121	-0.02
7	S	d62-Triacontane {S}	0.695	0.967	-13.4	141	0.00
8	I	d74-Hexatriacontane	1.000	1.000	0.0	205	-0.04

(#) = Out of Range SPCC's out = 0 CCC's out = 0

DROTPH051611.M Wed Feb 15 13:52:47 2012 SLICK2

Area Percent Report

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02827.D
Acq On : 15 Feb 2012 12:42
Operator : Syslo
[illegible]ple : 1.0k TPH CCV
[illegible]sc : RVB0003
ALS Vial : 71 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 7 % of largest Peak
Start Thrs: 0.05 Max Peaks: 150
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\msdchem\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL02827.D\data.ms

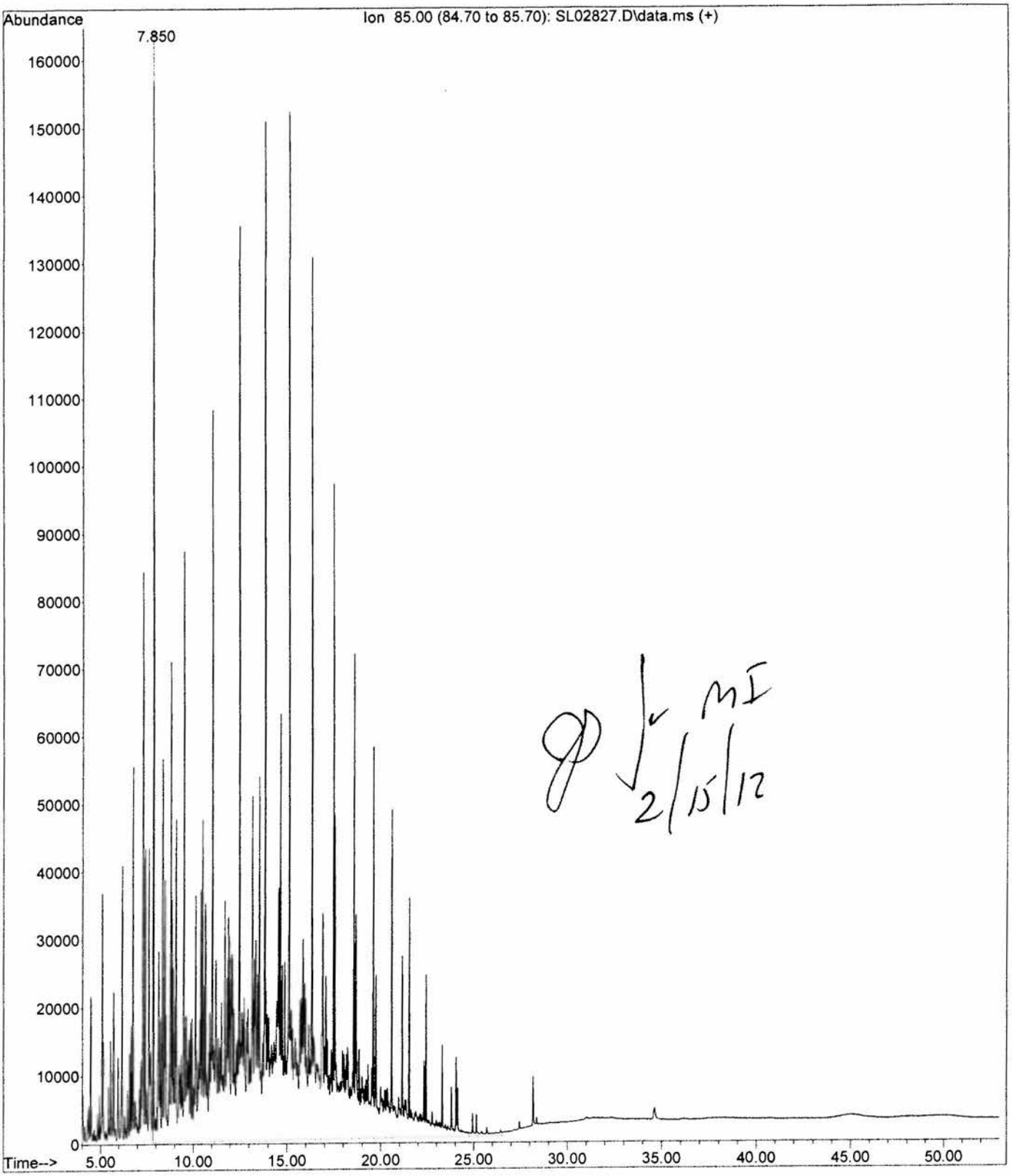
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	7.850	597	597	2838	rM 8	164679	11689228	100.00%	100.000%

Sum of corrected areas: 11689228

)P[illegible]PH051611.M Wed Feb 15 13:56:09 2012 SLICK2

gs J. ME 2/15/12

File :C:\msdchem\1\DATA\021512\SL02827.D
Operator : Syslo
Acquired : 15 Feb 2012 12:42 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 1.0k TPH CCV
Misc Info : RVB0003
Vial Number: 71



MI
2/15/12

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Sal Blank

1200024-BLK

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02828.D
Acq On : 15 Feb 2012 13:43
Operator : Syslo
[illegible]mple : Soil Blank
[illegible]sc : 1200024-Blk1
ALS Vial : 82 Sample Multiplier: 1

Quant Time: Feb 15 16:22:43 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	455645	10.00	ug/mL	-0.02
5) d30-Tetradecane	13.540	66	139126	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	159054	10.00	ug/mL	-0.02
8) d74-Hexatriacontane	34.612	66	106564	10.00	ug/mL	-0.04
System Monitoring Compounds						
2) d10-Anthracene {S}	18.822	188	539411	11.13	ug/mL	-0.02
Spiked Amount 20.000			Recovery =	55.65%		
3) d14-o-Terphenyl {S}	19.733	244	258958	12.16	ug/mL	-0.02
Spiked Amount 20.000			Recovery =	60.80%		
4) 5a-Androstane {S}	21.141	260	34097	13.16	ug/mL	-0.02
Spiked Amount 20.000			Recovery =	65.80%		
7) d62-Triacontane {S}	28.156	66	197748	14.33	ug/mL	-0.02
Spiked Amount 20.000			Recovery =	71.65%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

TPH: ND

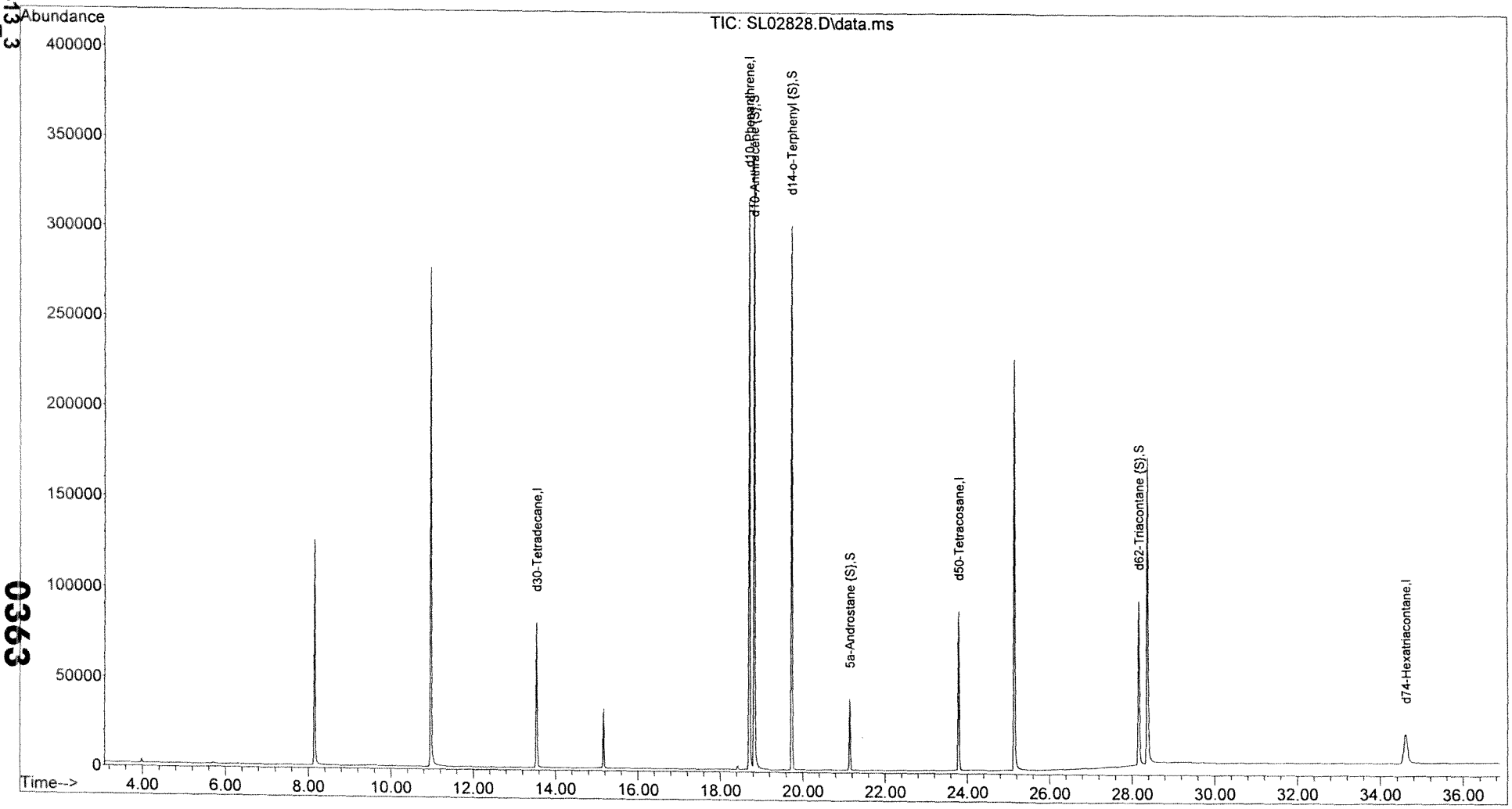
US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

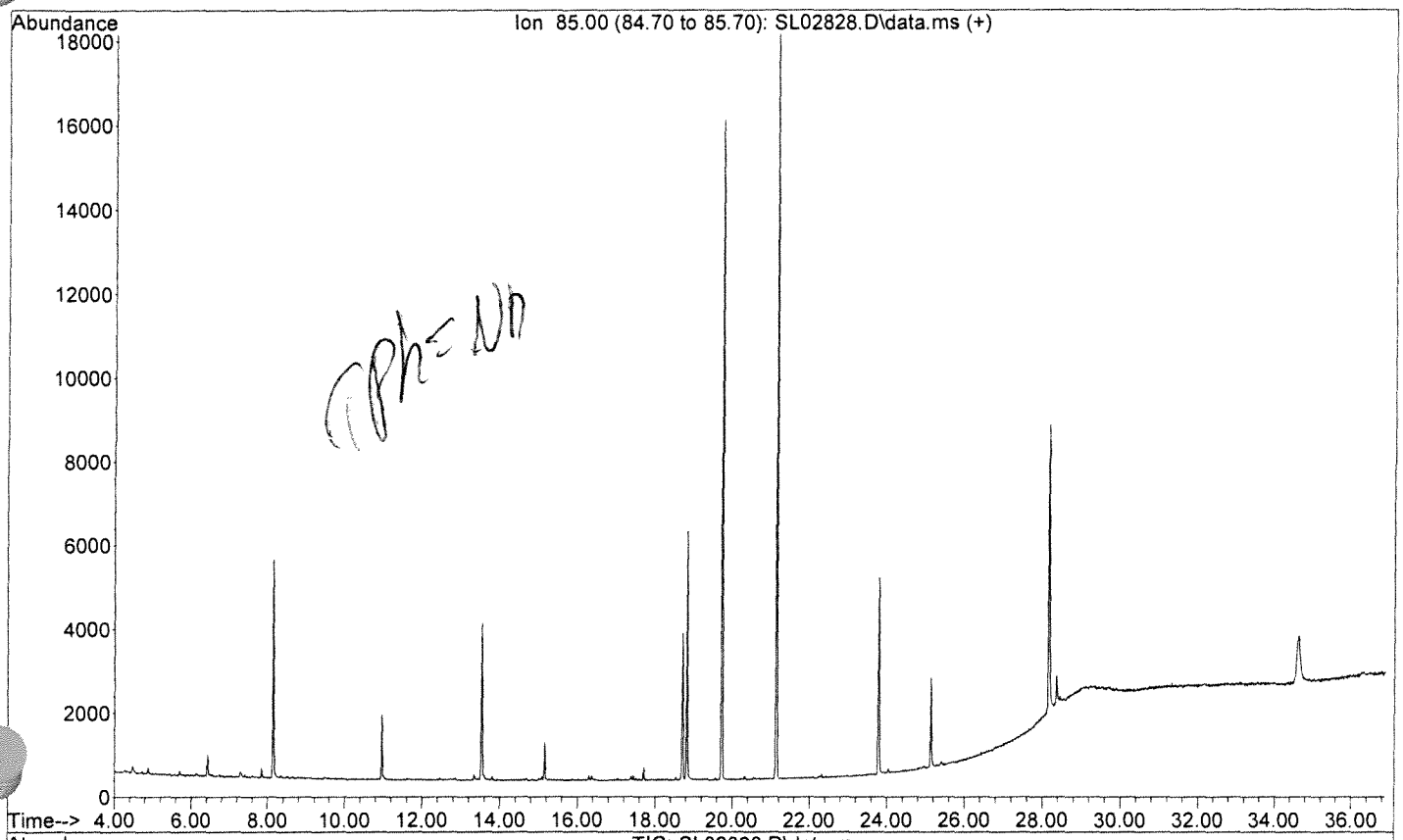
Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02828.D
Acq On : 15 Feb 2012 13:43
Operator : Syslo
Sample : Soil Blank
Misc : 1200024-Blk1
ALS Vial : 82 Sample Multiplier: 1

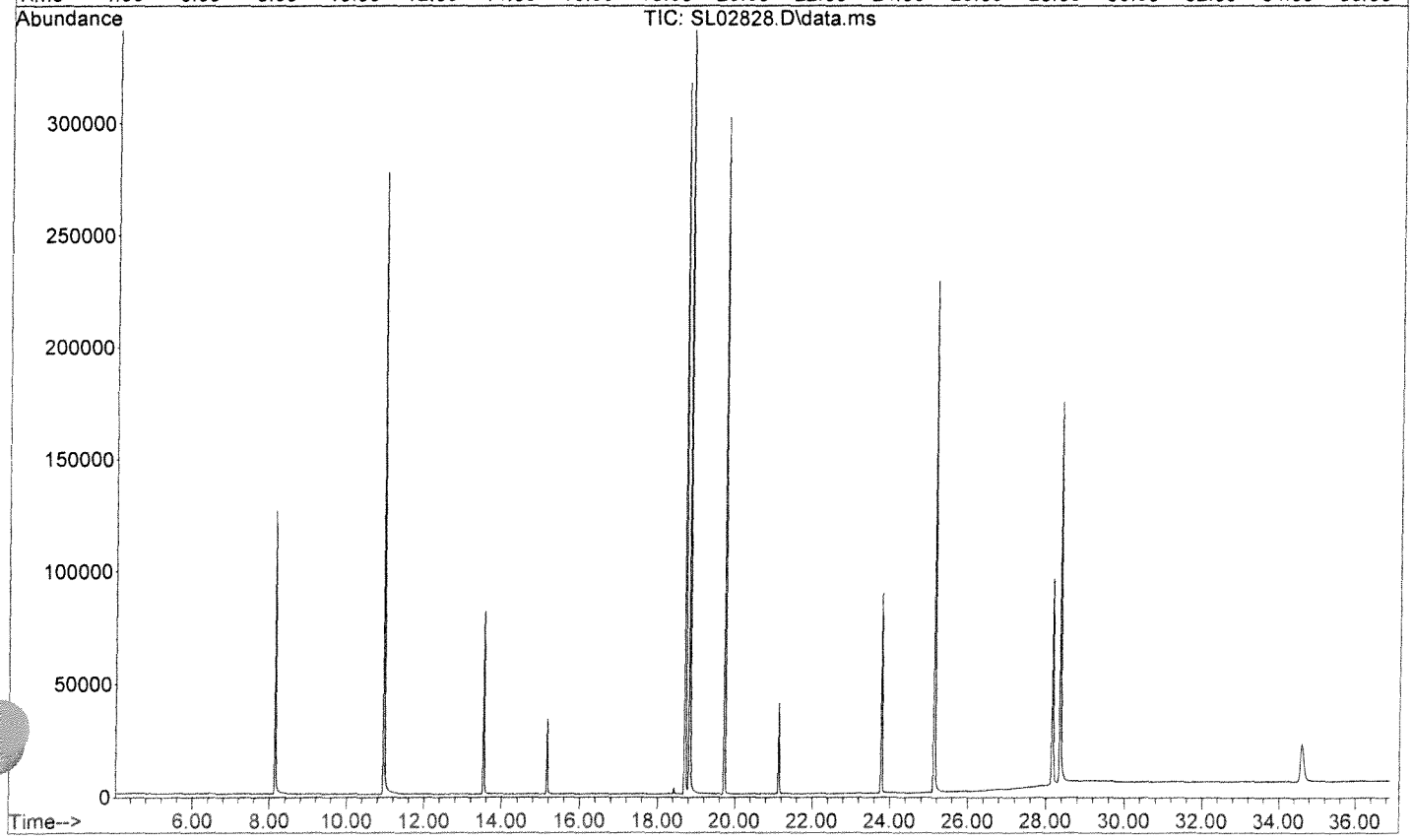
Quant Time: Feb 15 16:22:43 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration



0363

ile :C:\msdchem\1\DATA\021512\SL02828.D
perator : Syslo
cquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
nstrument : Slick2
ample Name: Soil Blank
Info : 1200024-Blk1
Number: 82





```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

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Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM.* Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

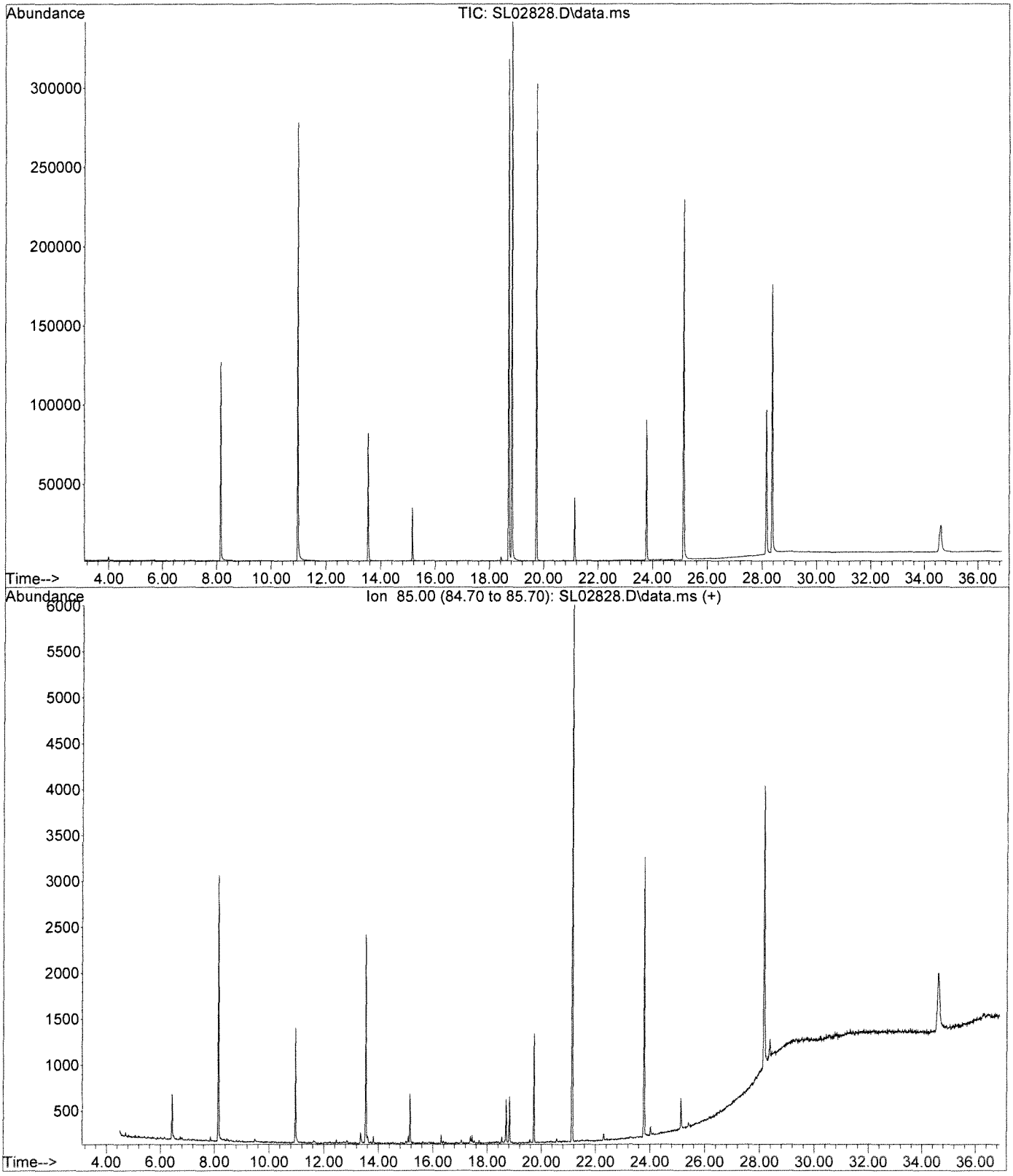
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82

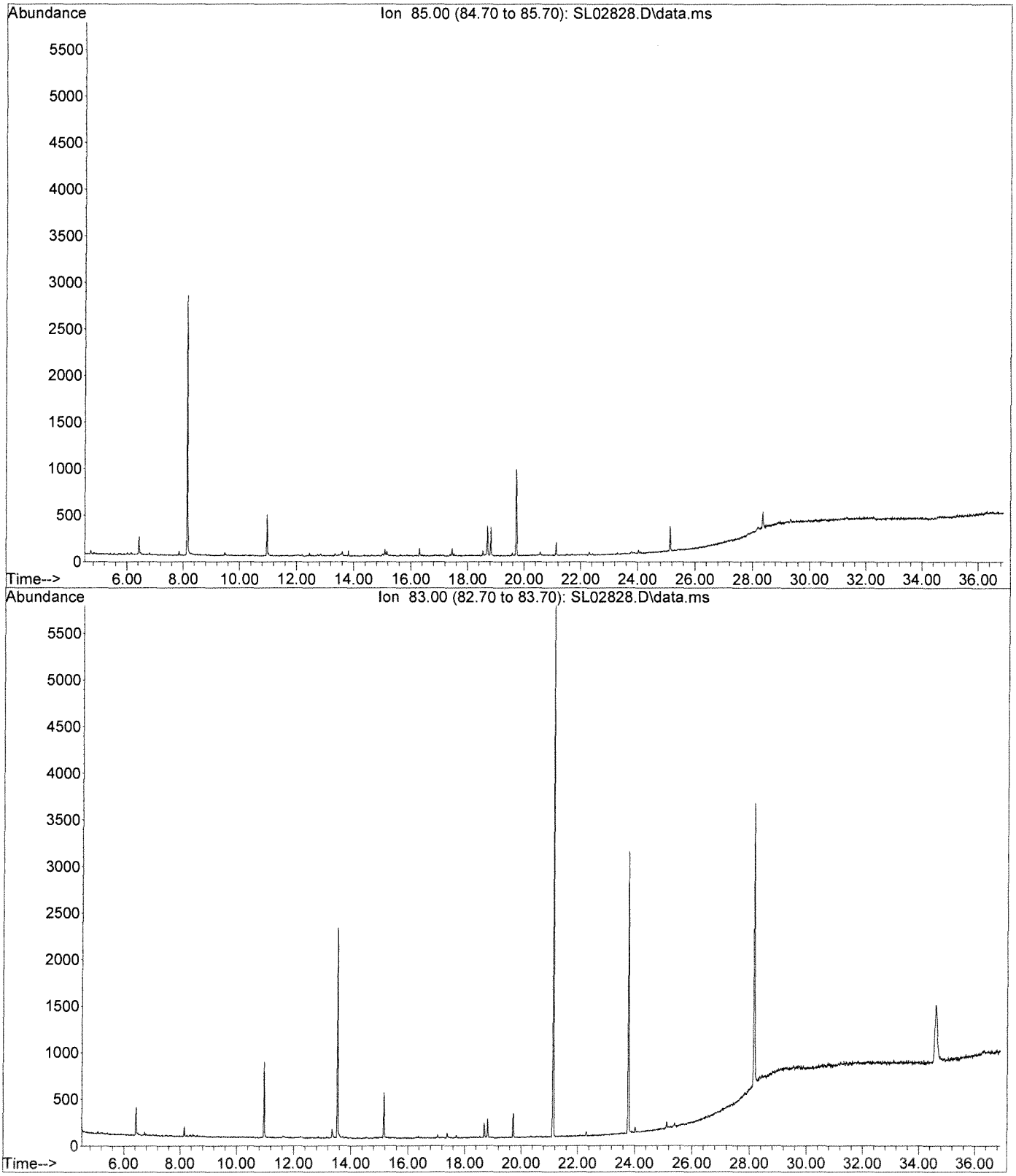


US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



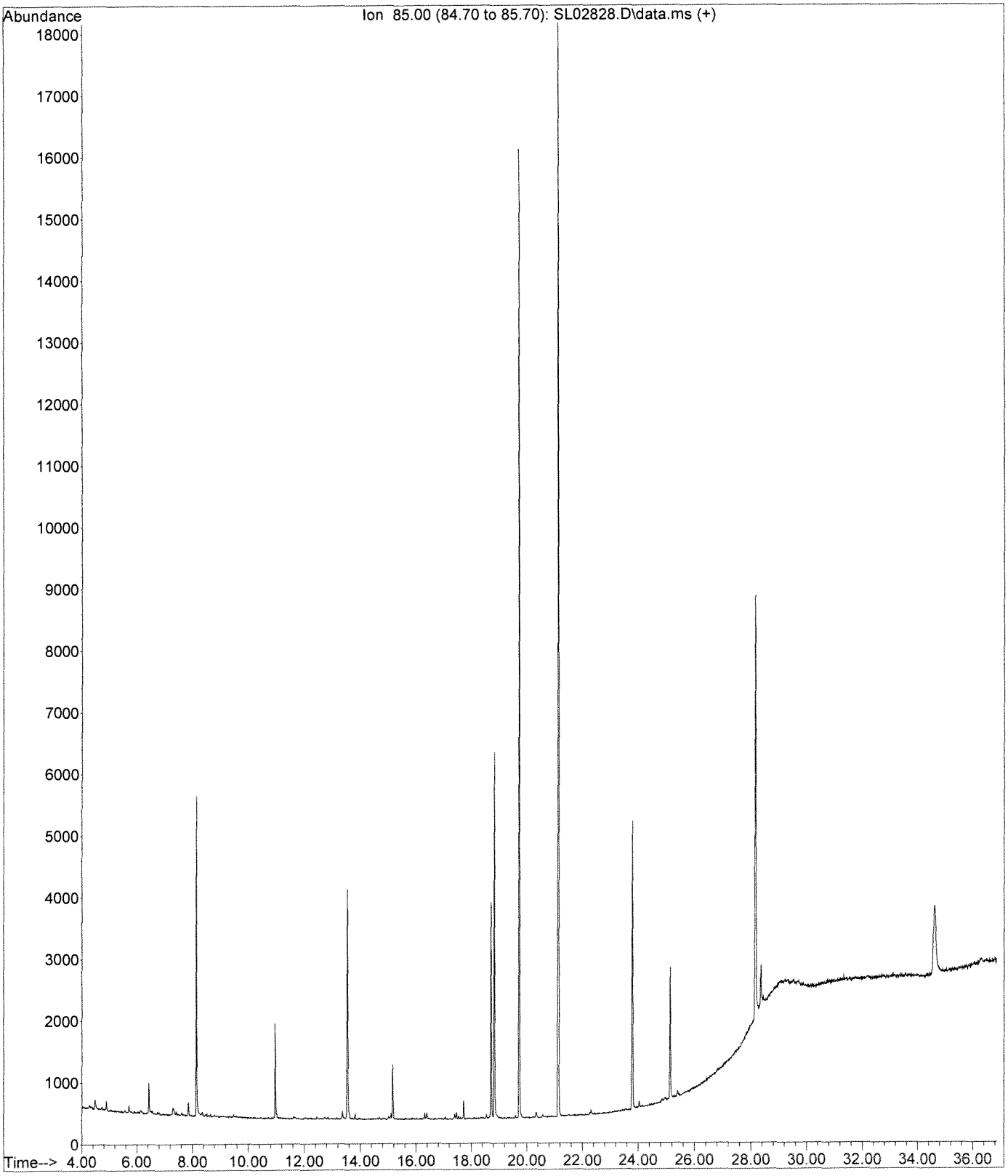
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



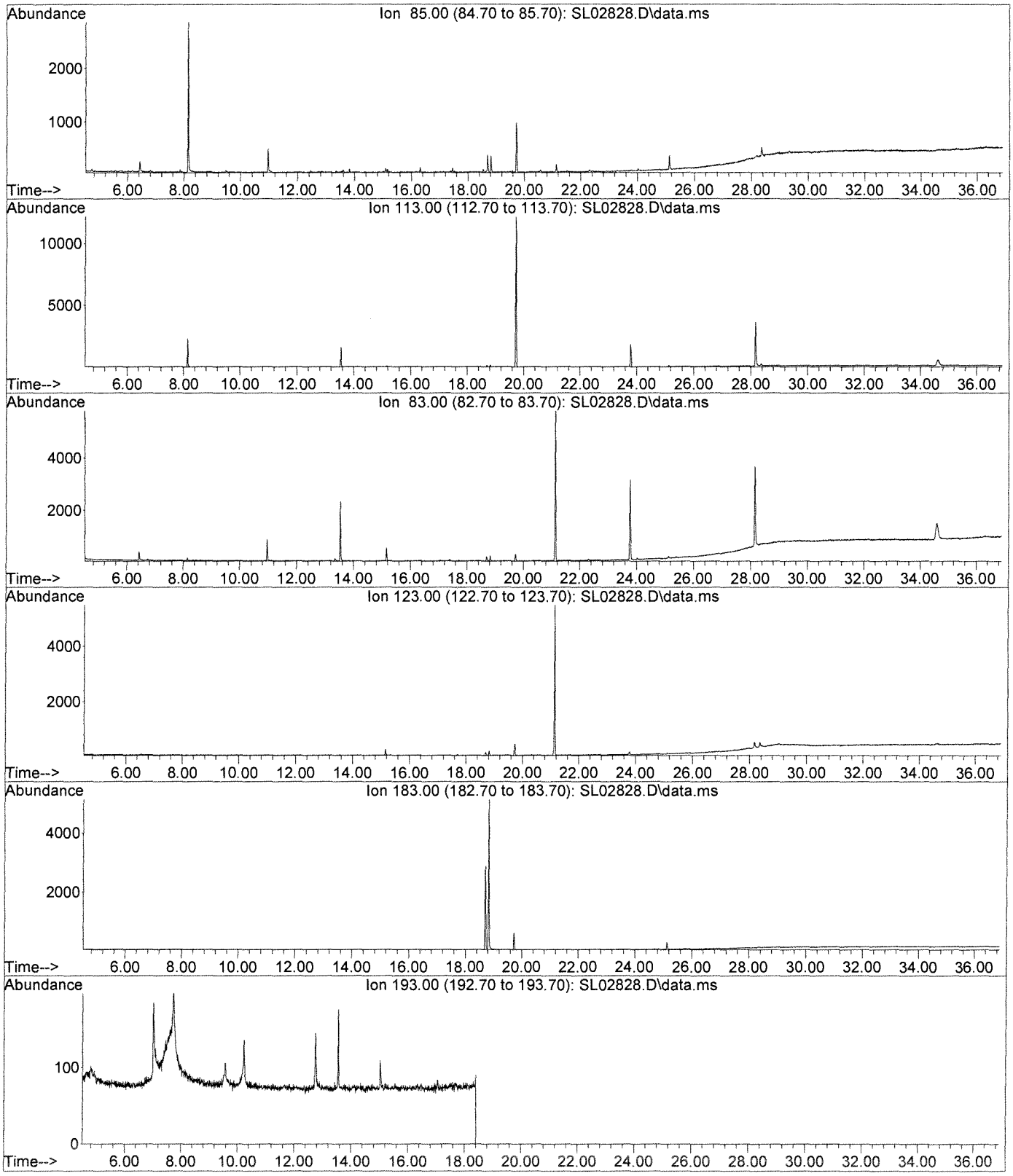
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



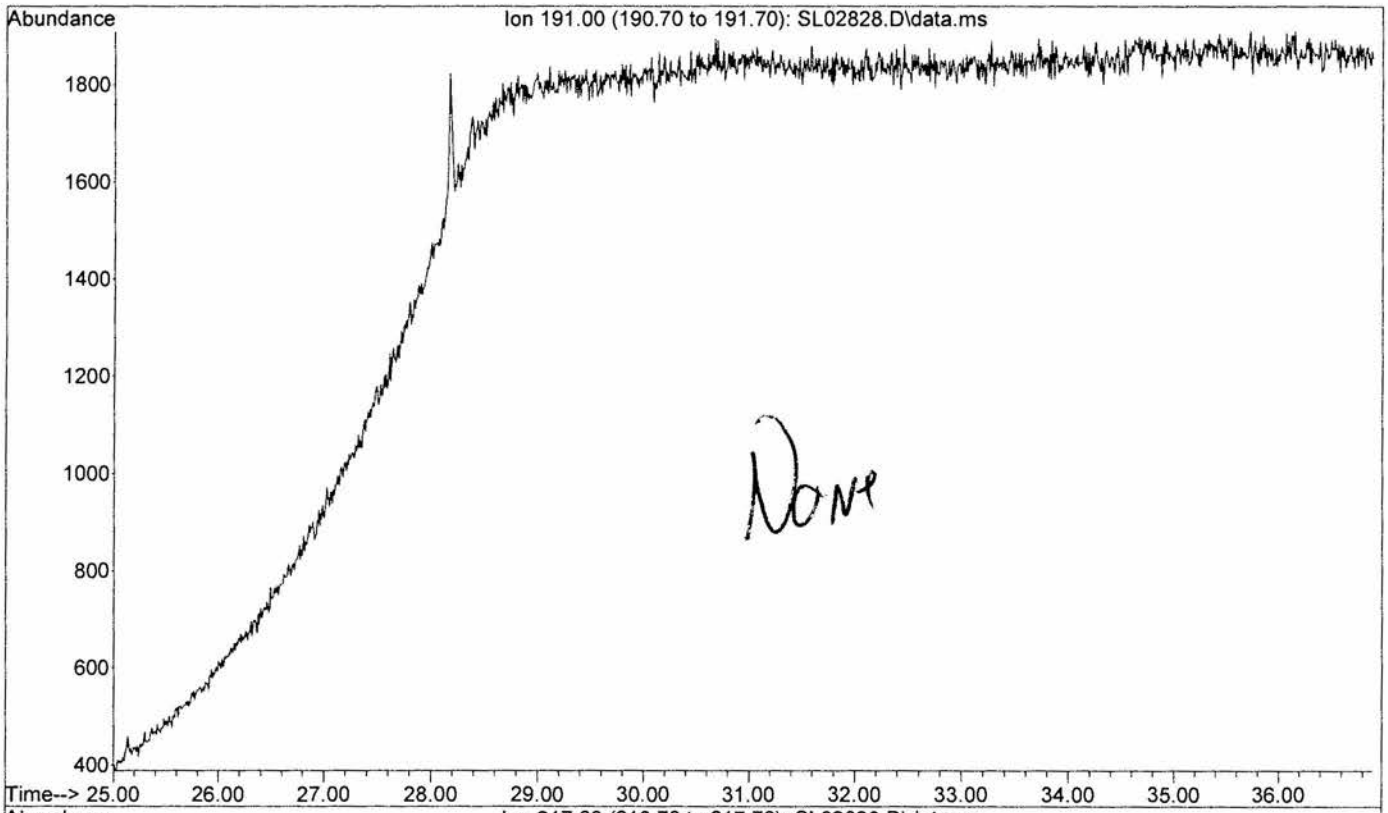
US EPA ARCHIVE DOCUMENT

```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

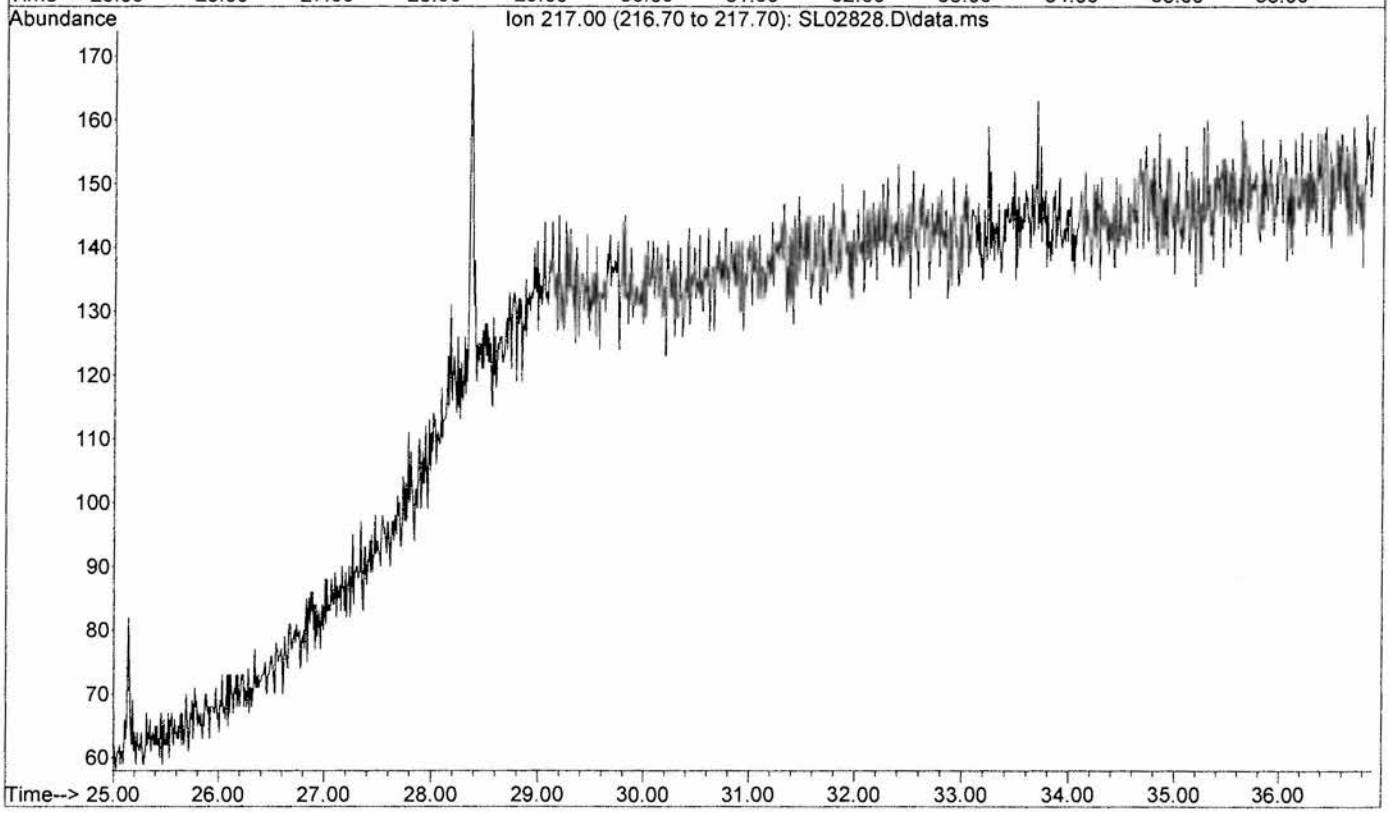
NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



None



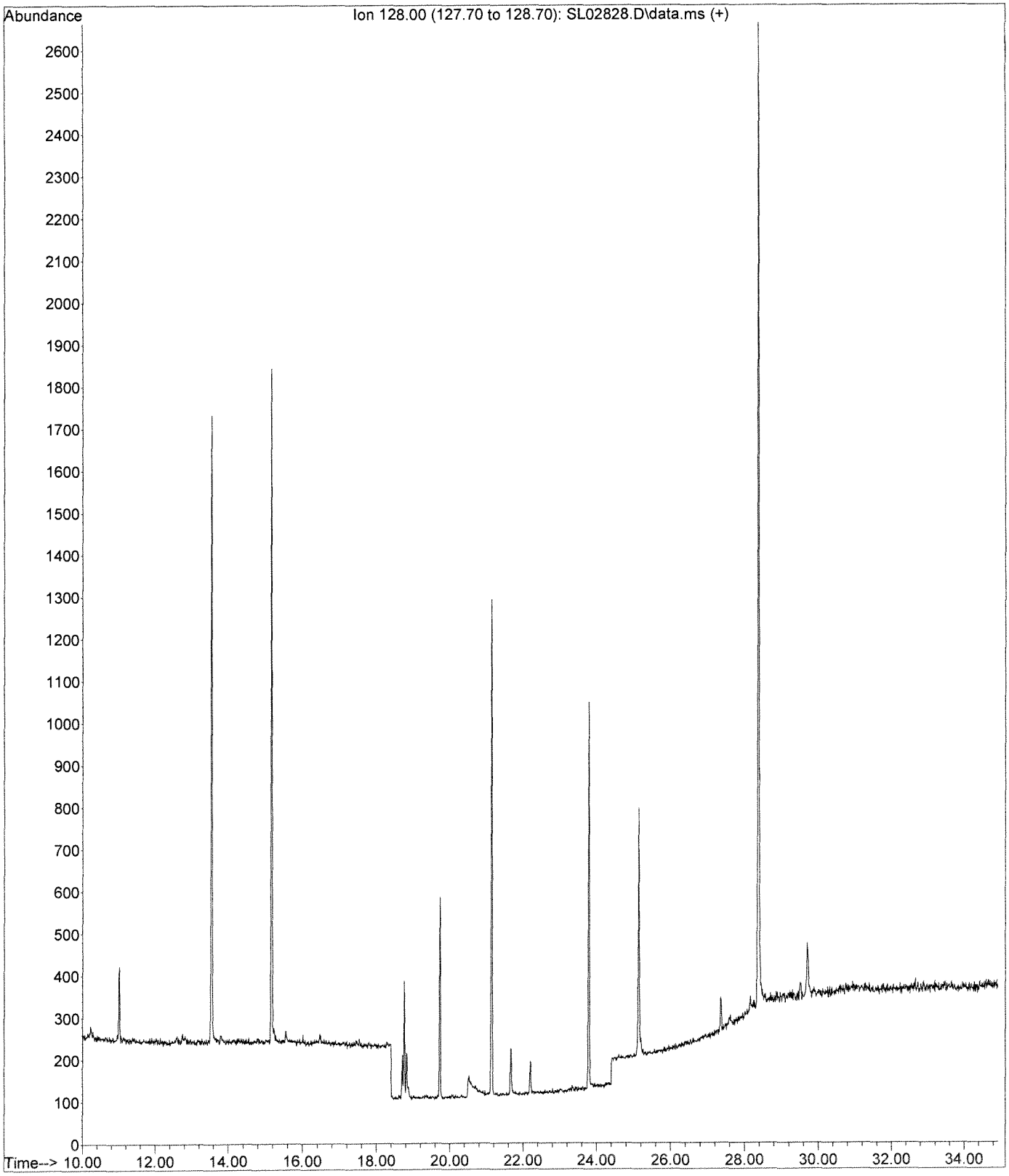
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



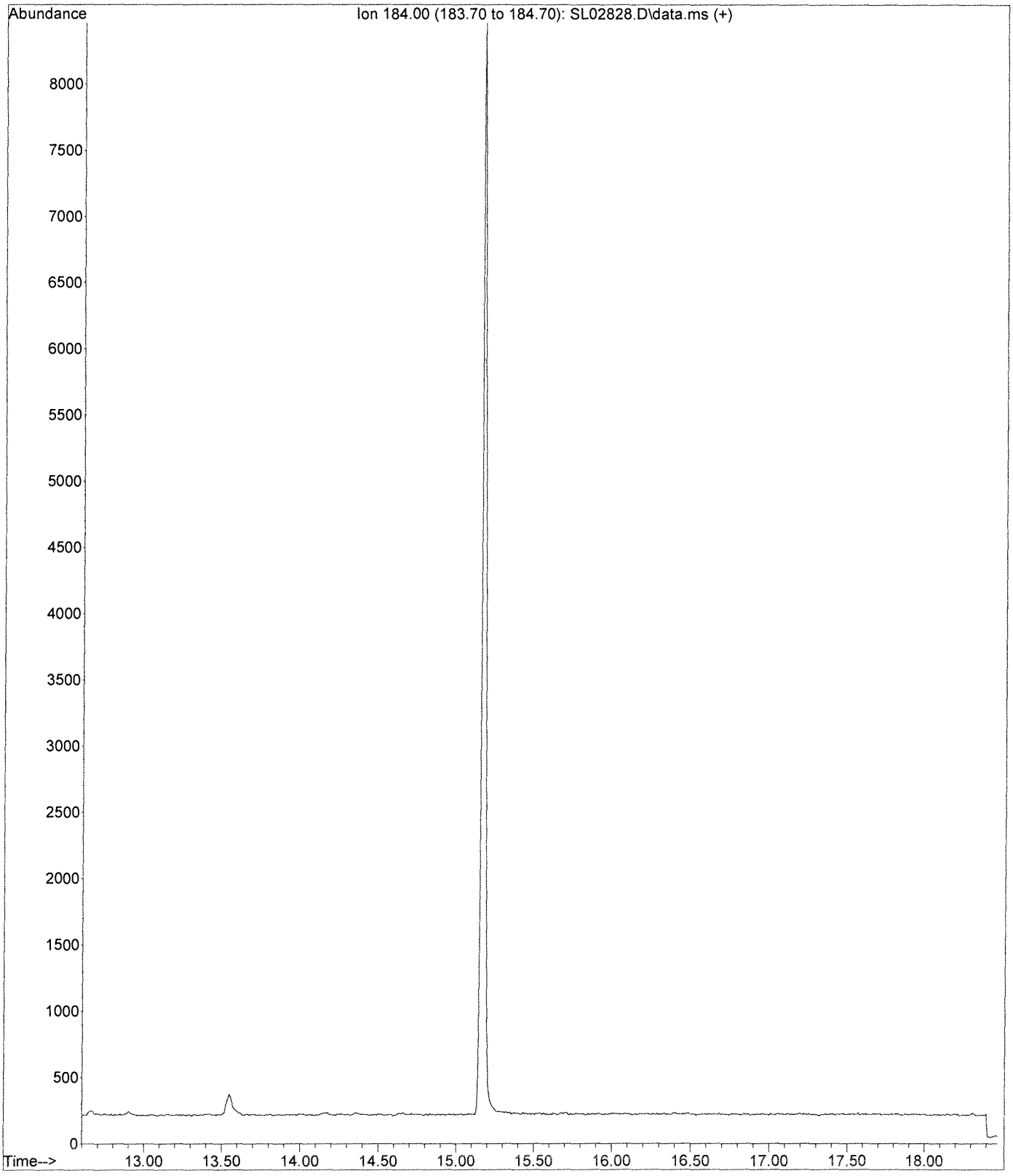
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

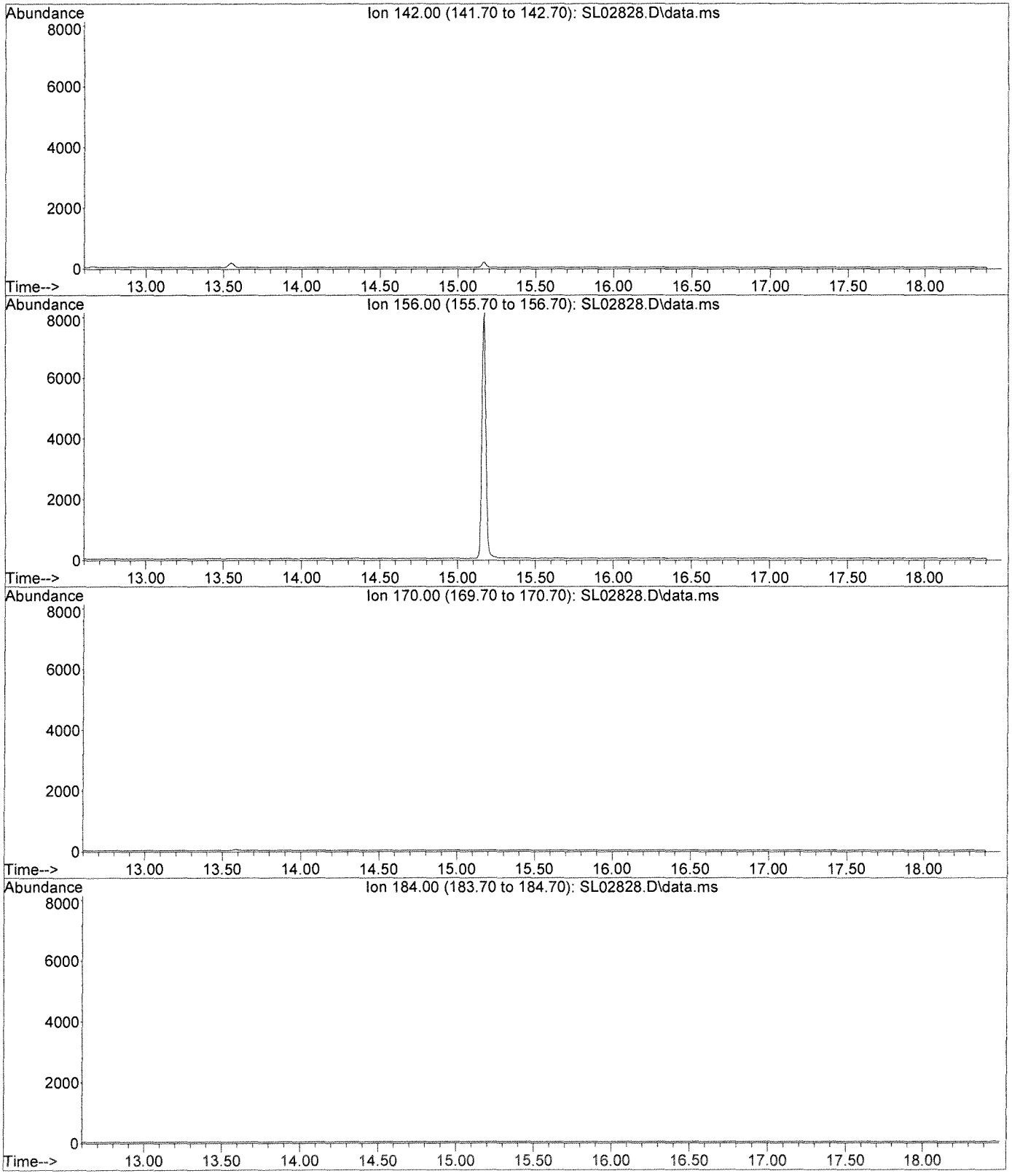
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



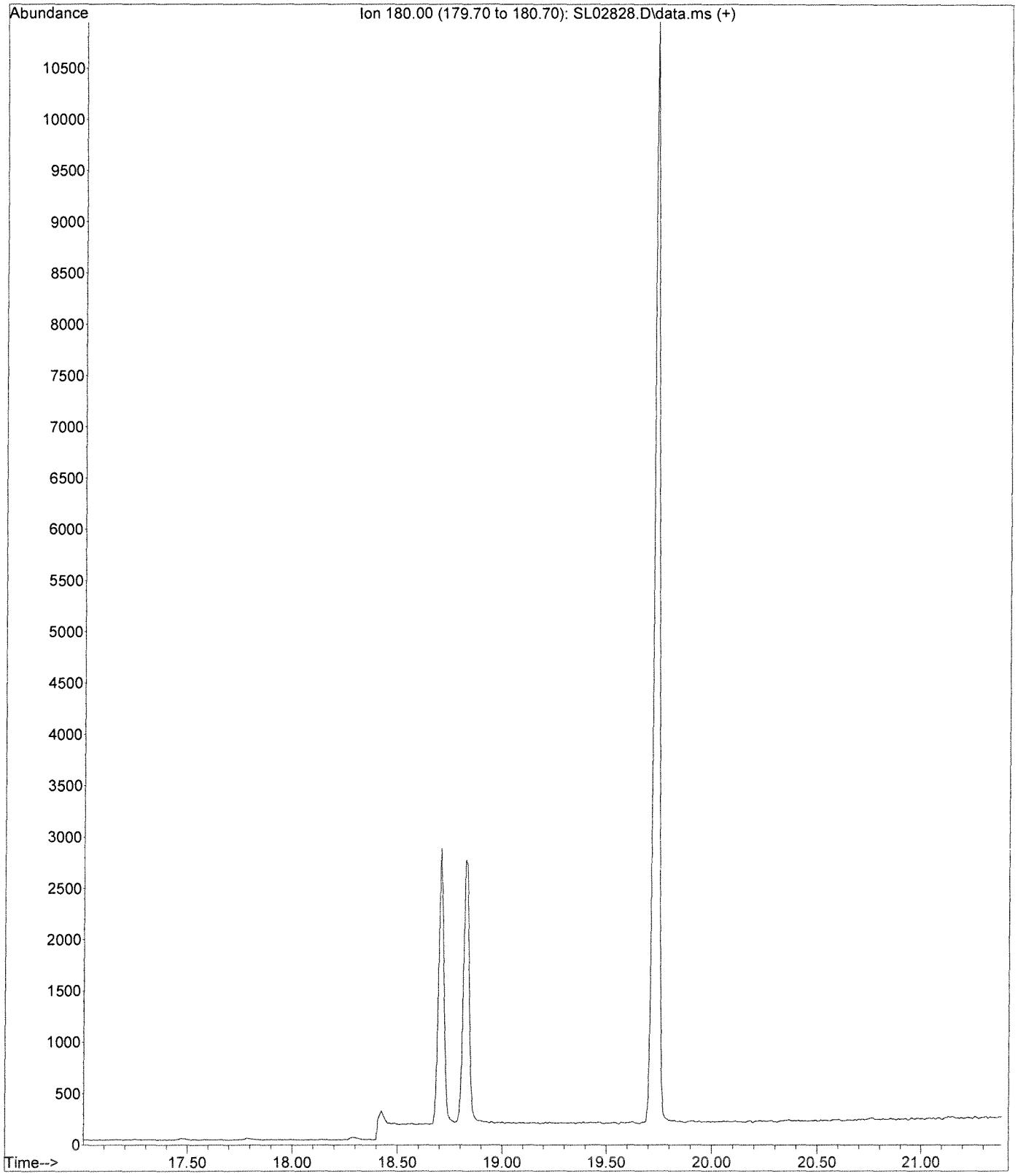
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



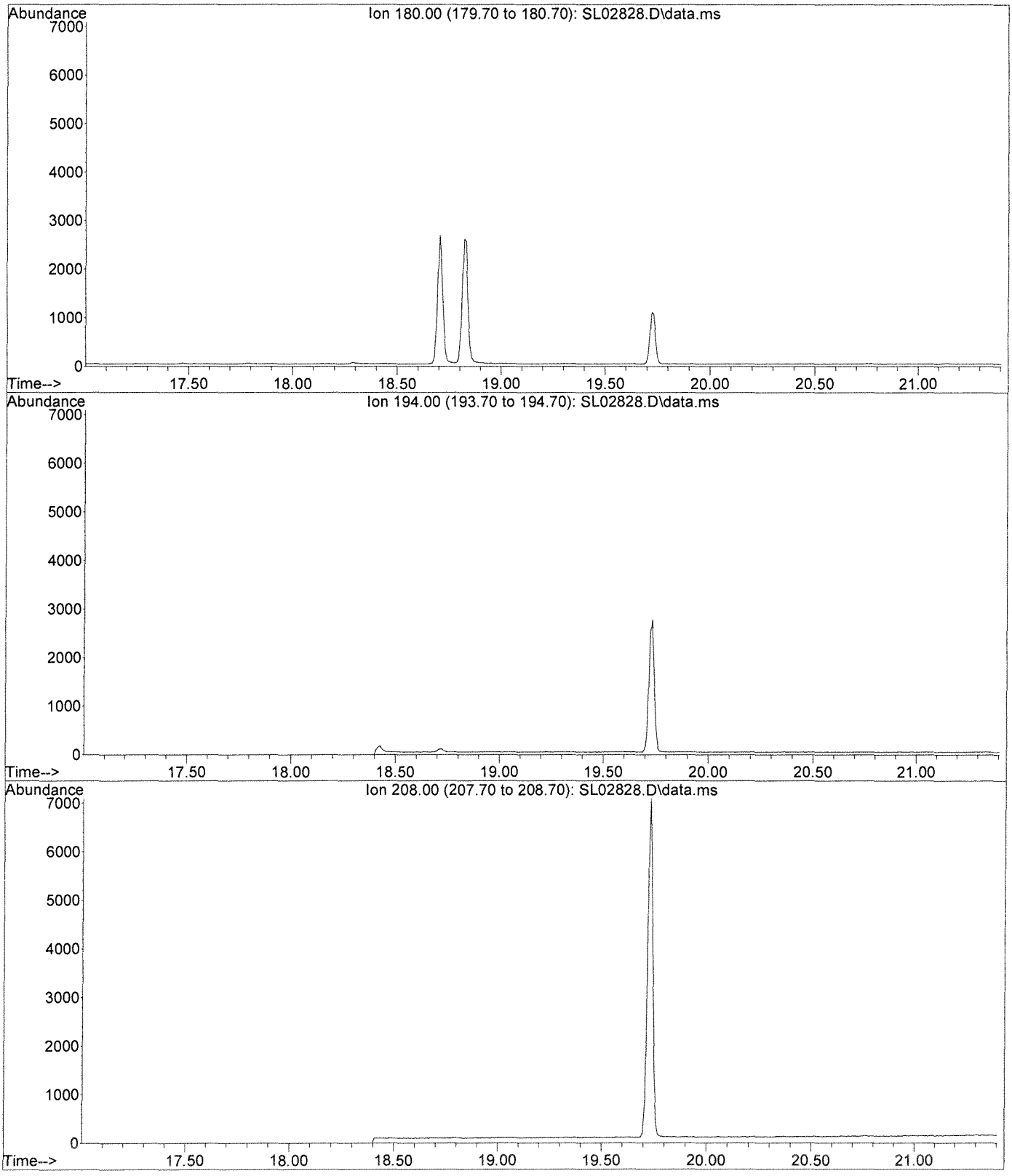
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



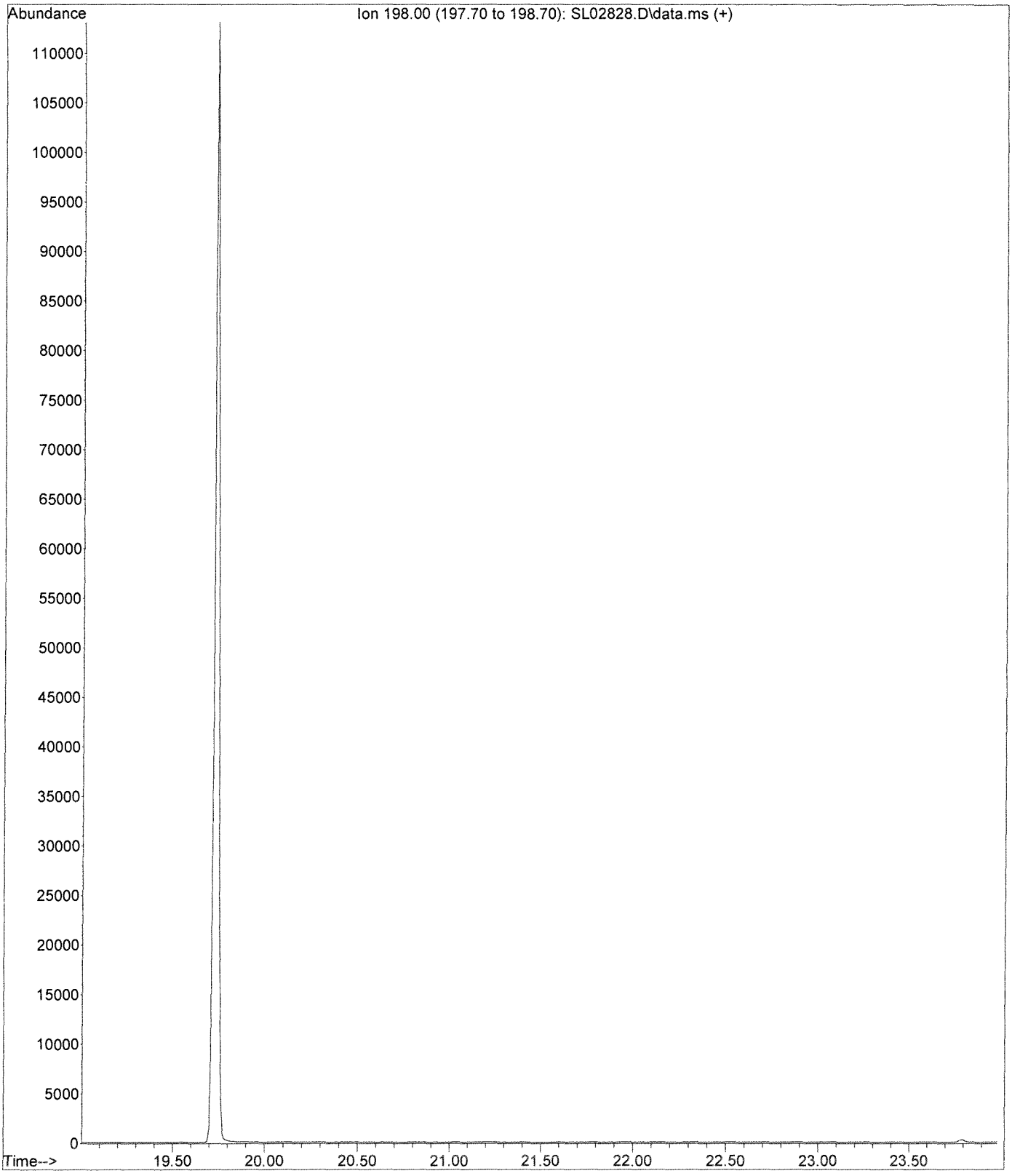
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02828.D
Operator    : Syslo
Acquired    : 15 Feb 2012  13:43      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: Soil Blank
Misc Info   : 1200024-Blk1
Vial Number: 82
```



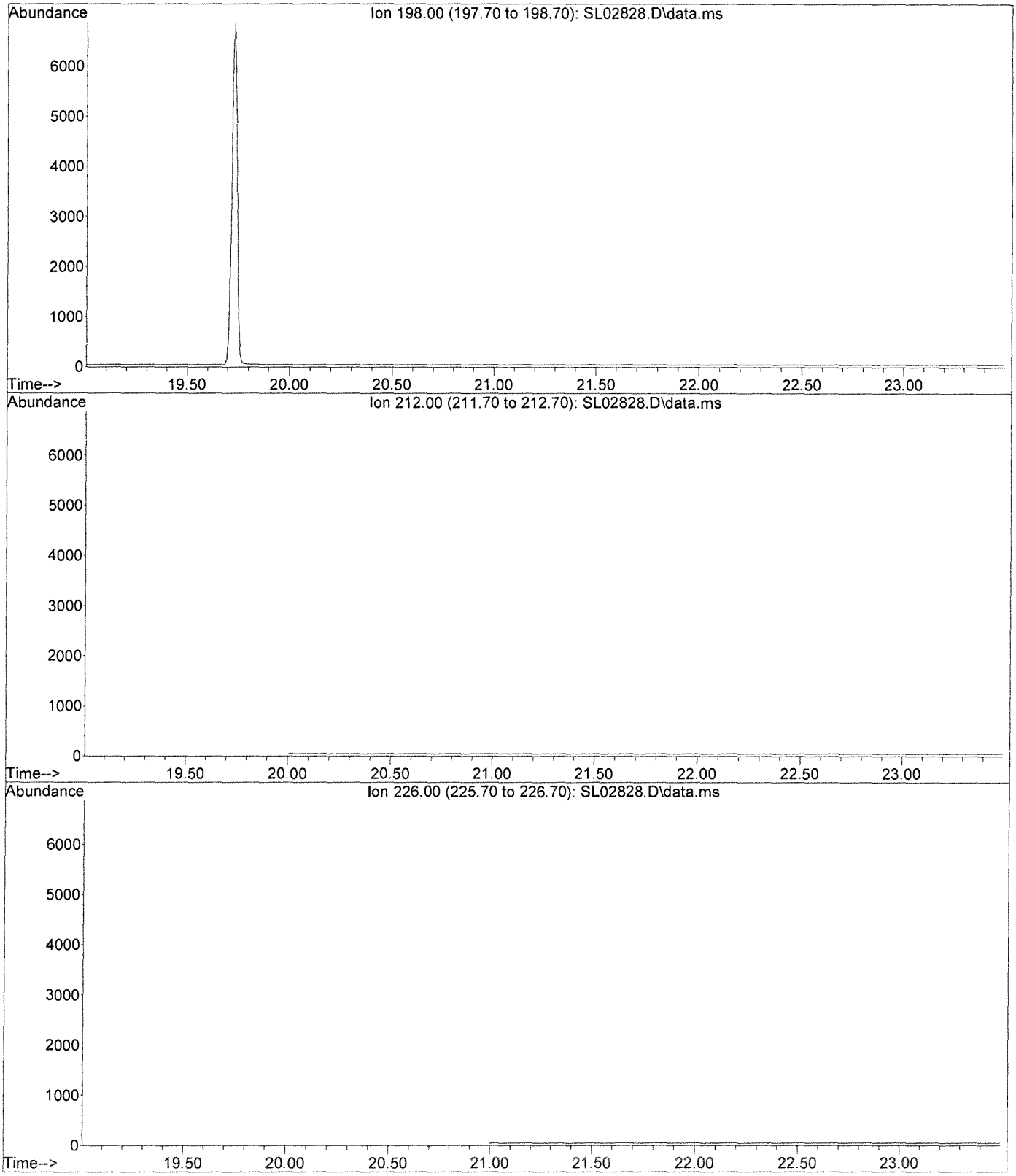
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



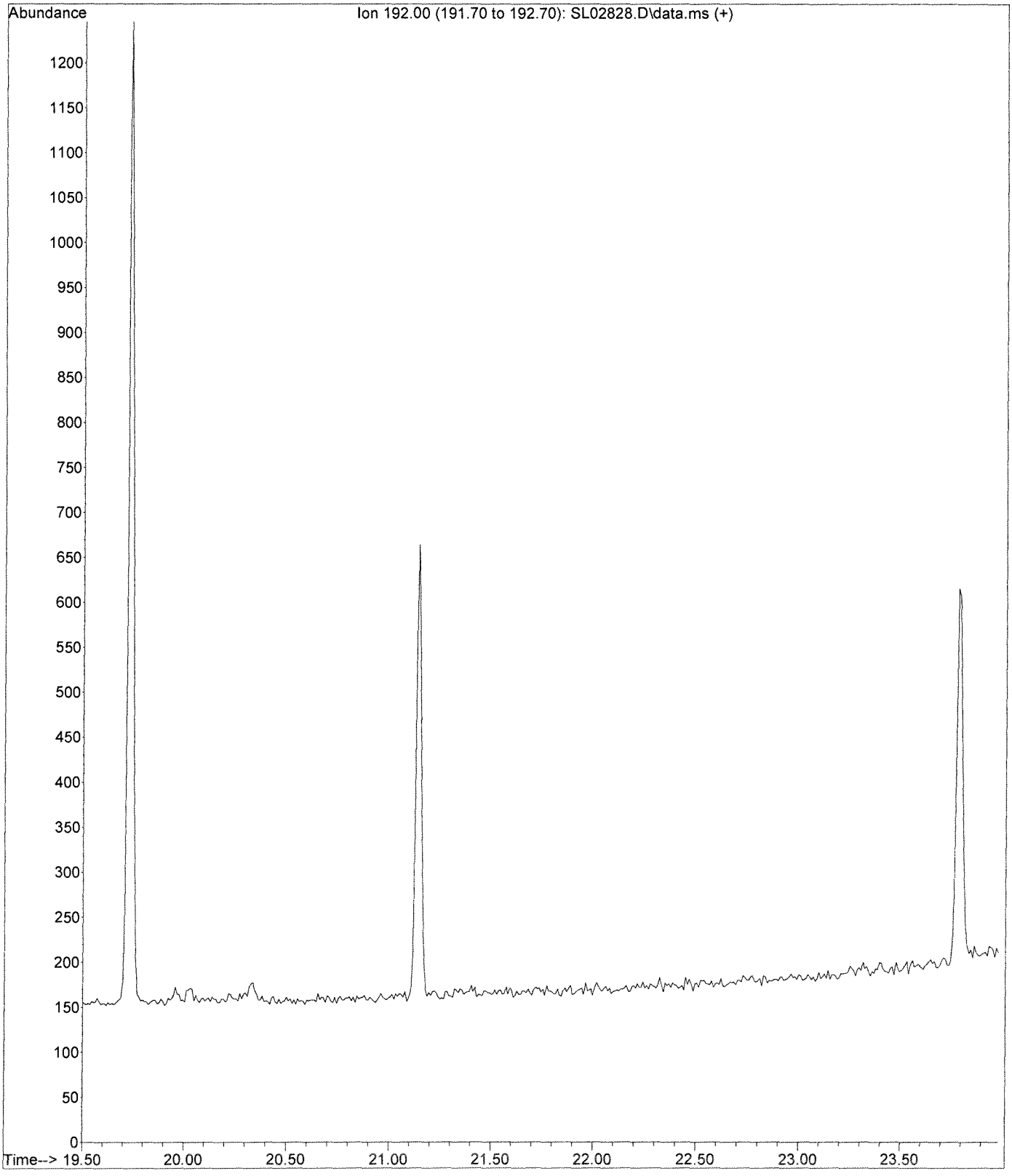
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



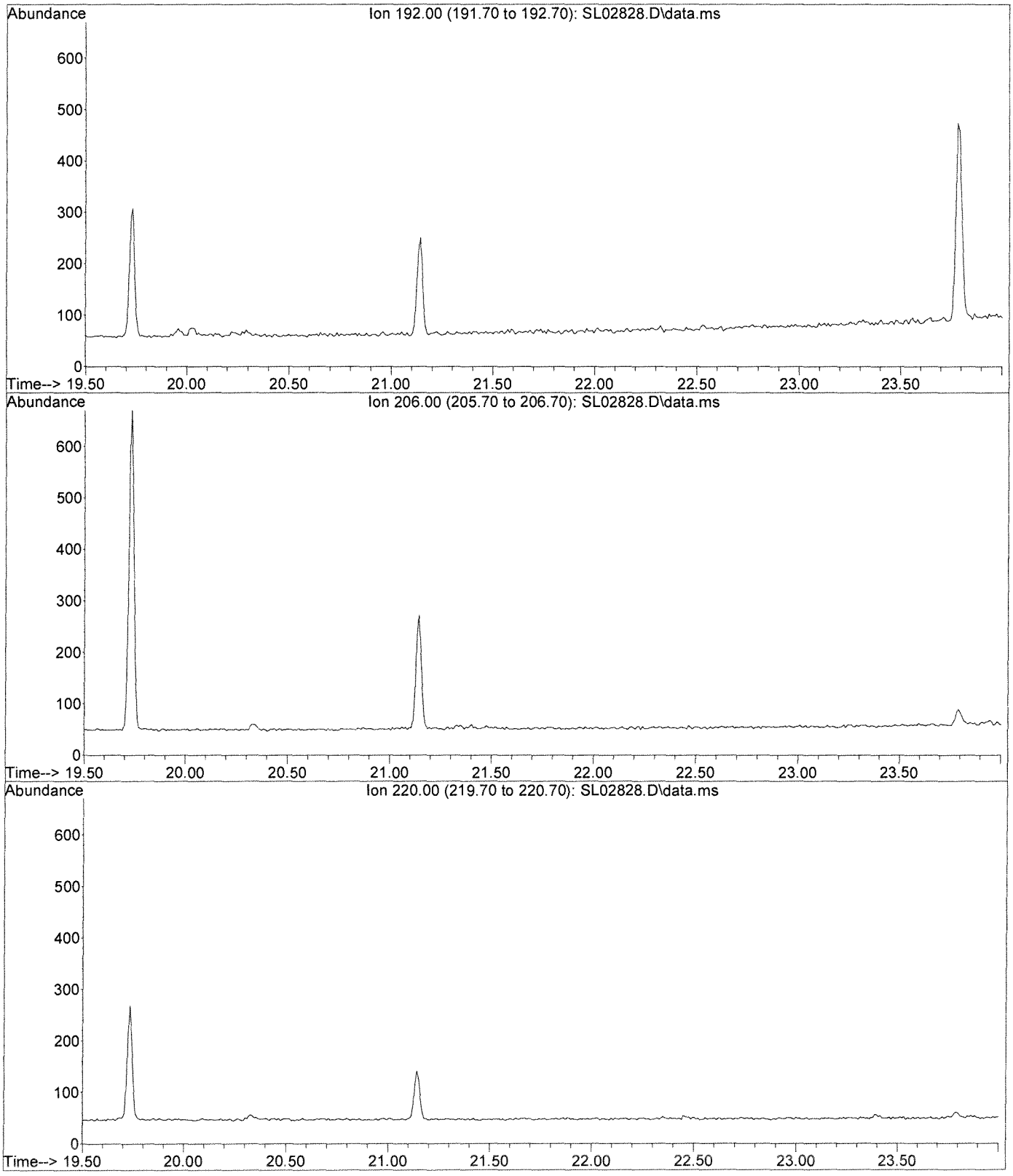
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



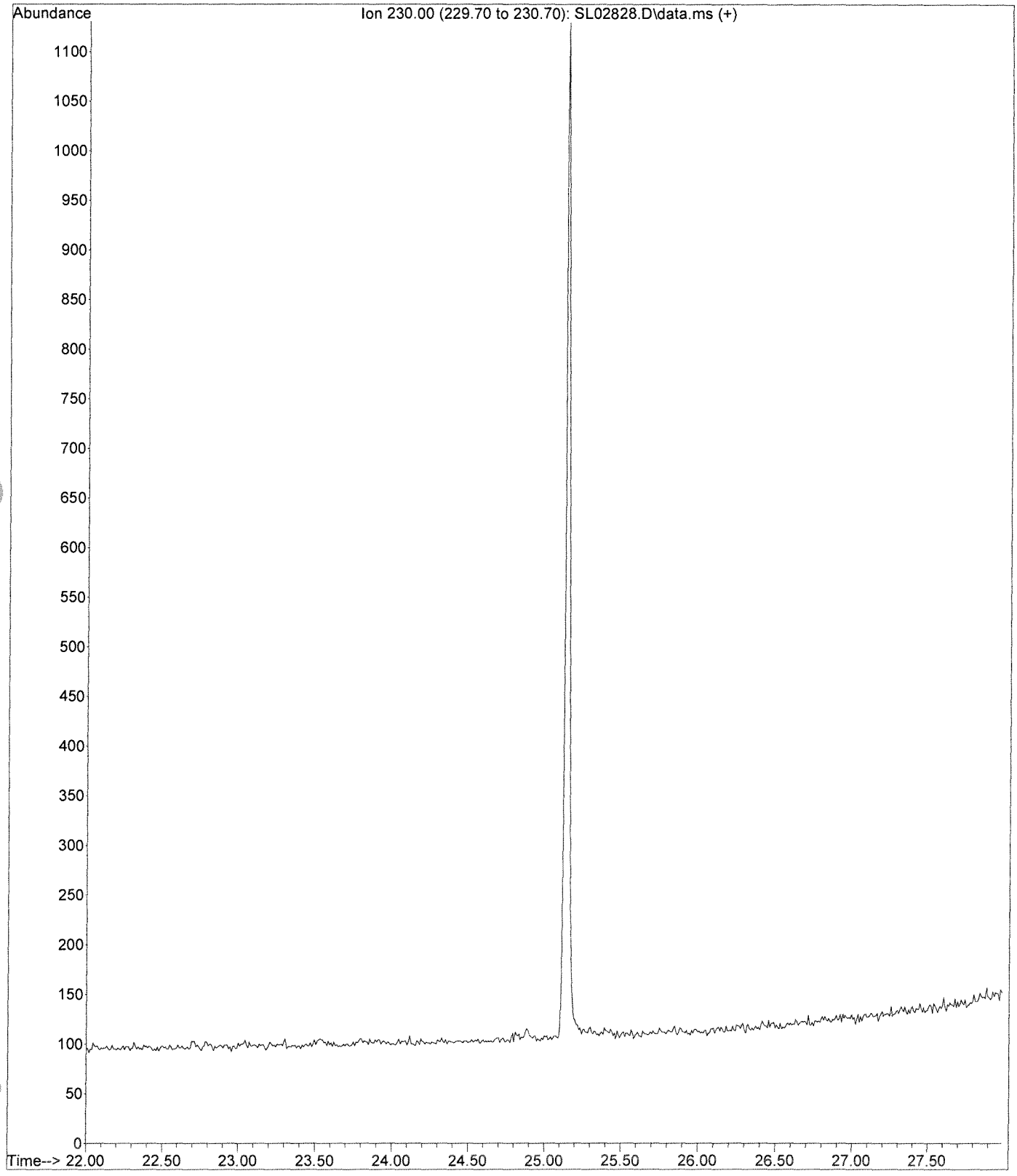
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



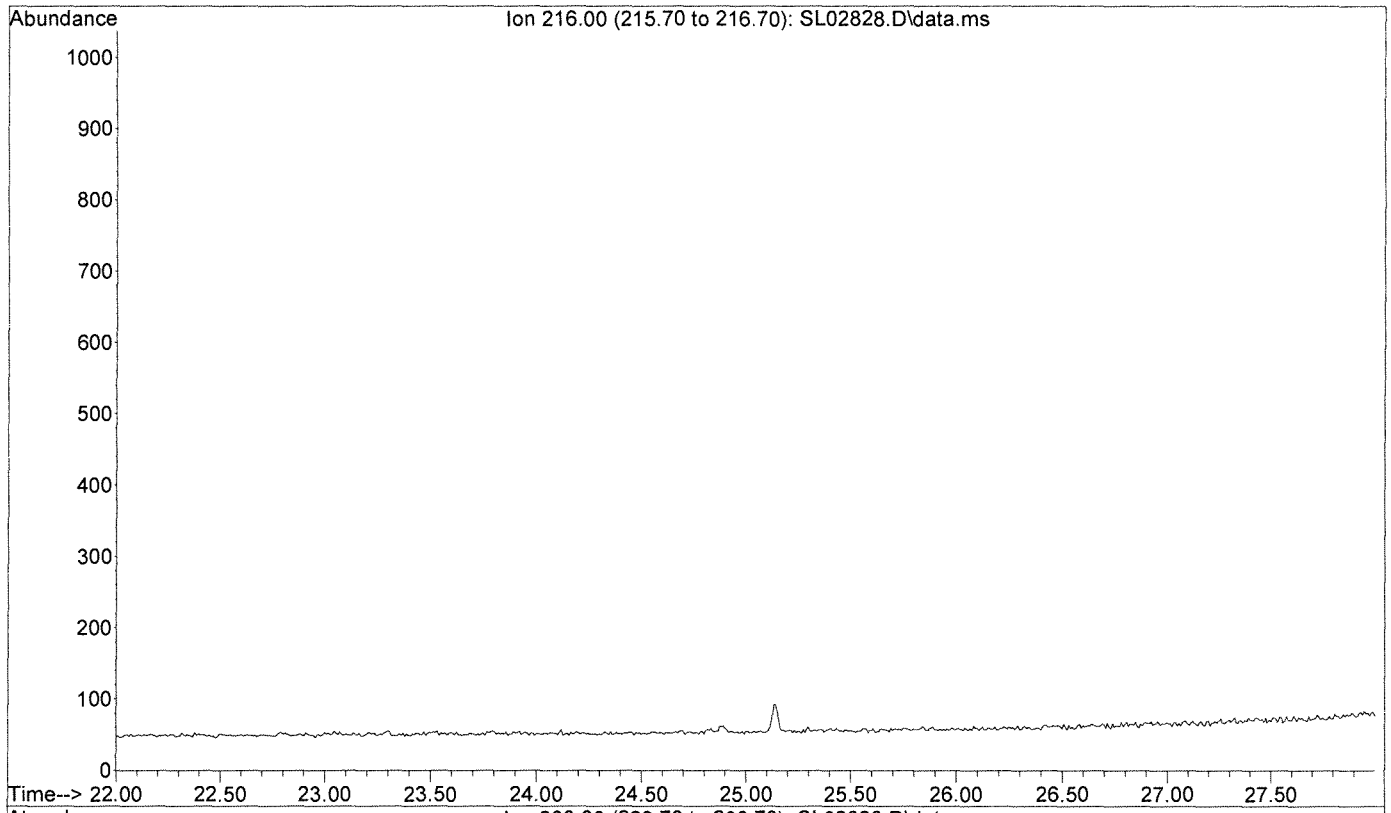
US EPA ARCHIVE DOCUMENT

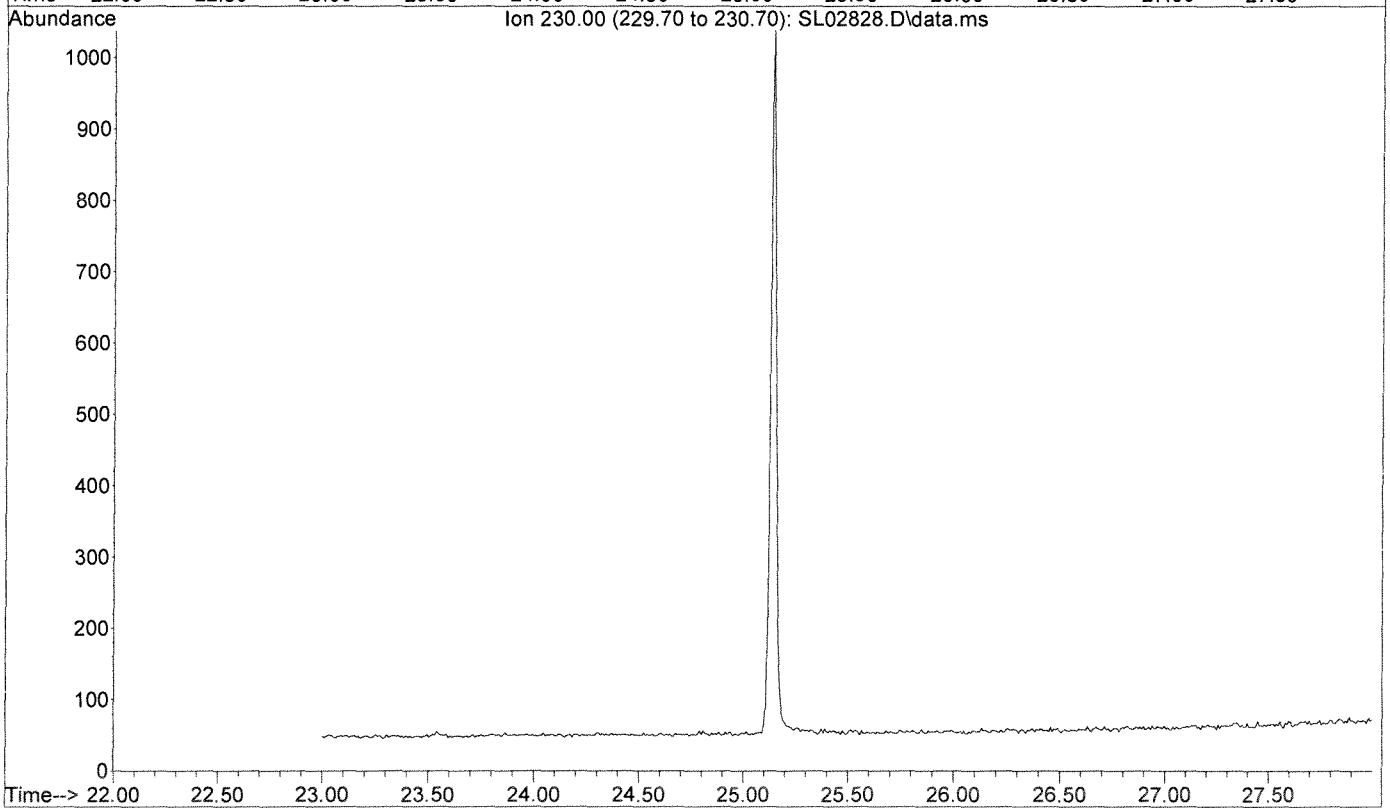
File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



US EPA ARCHIVE DOCUMENT

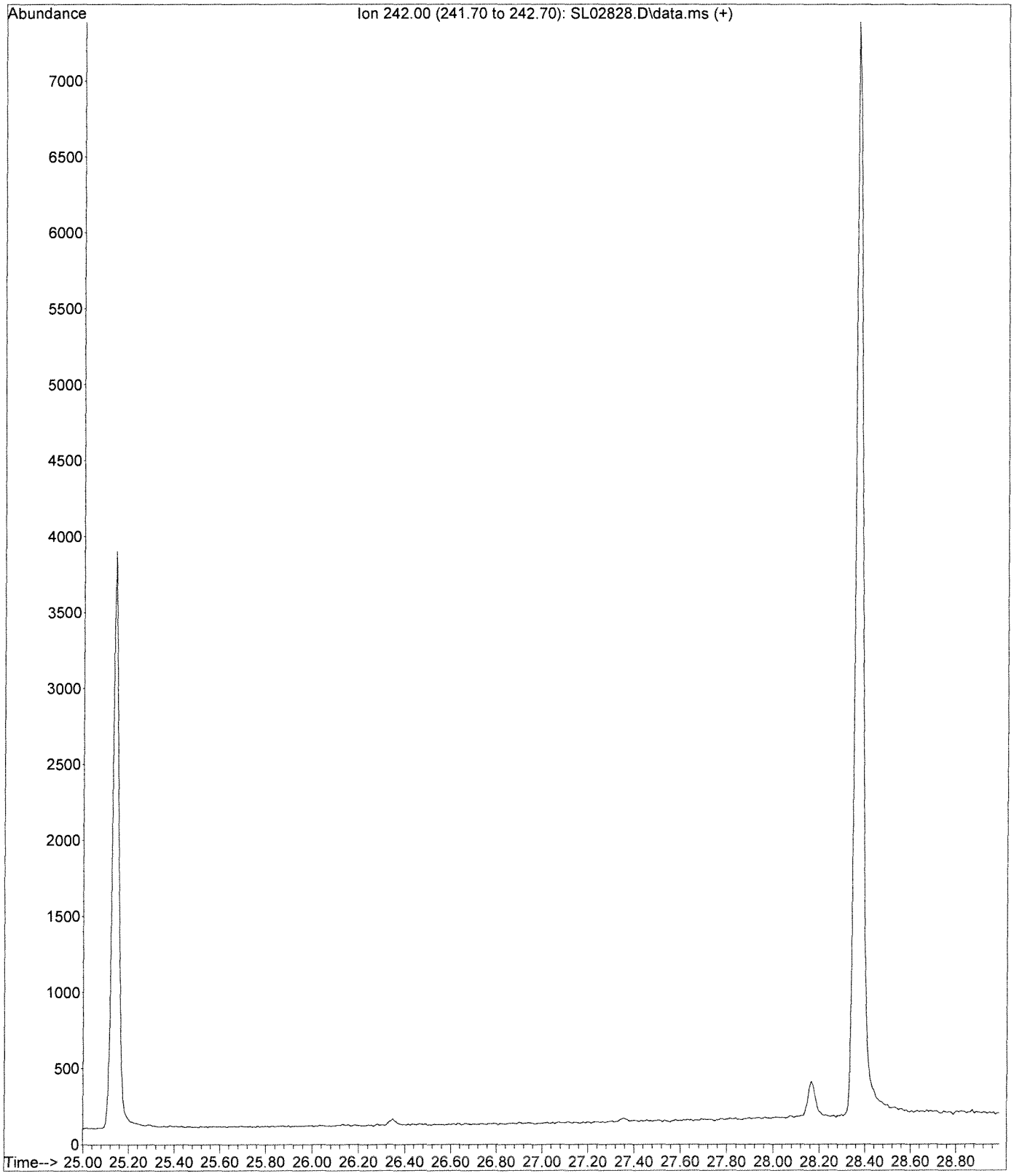
File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82





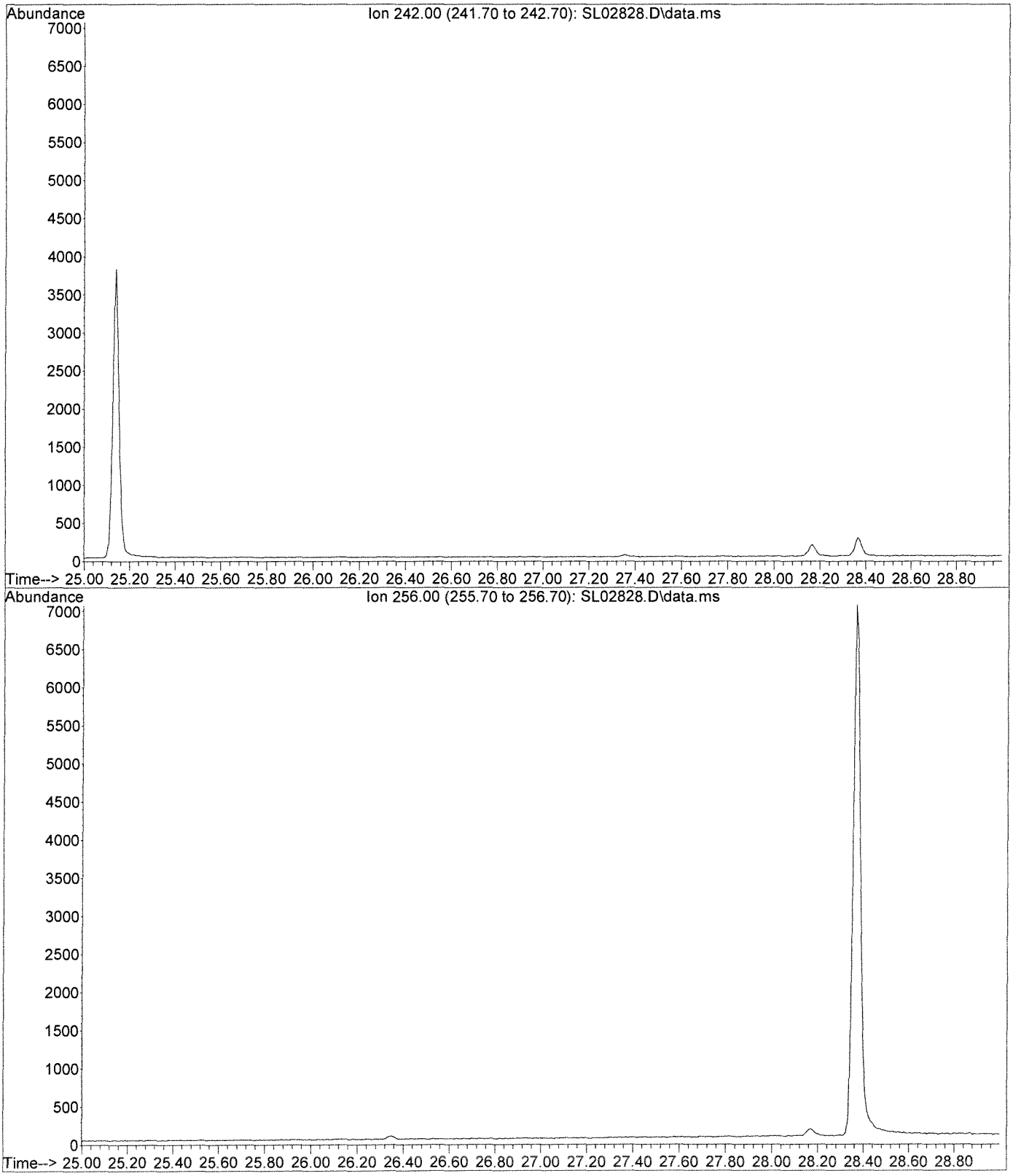
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



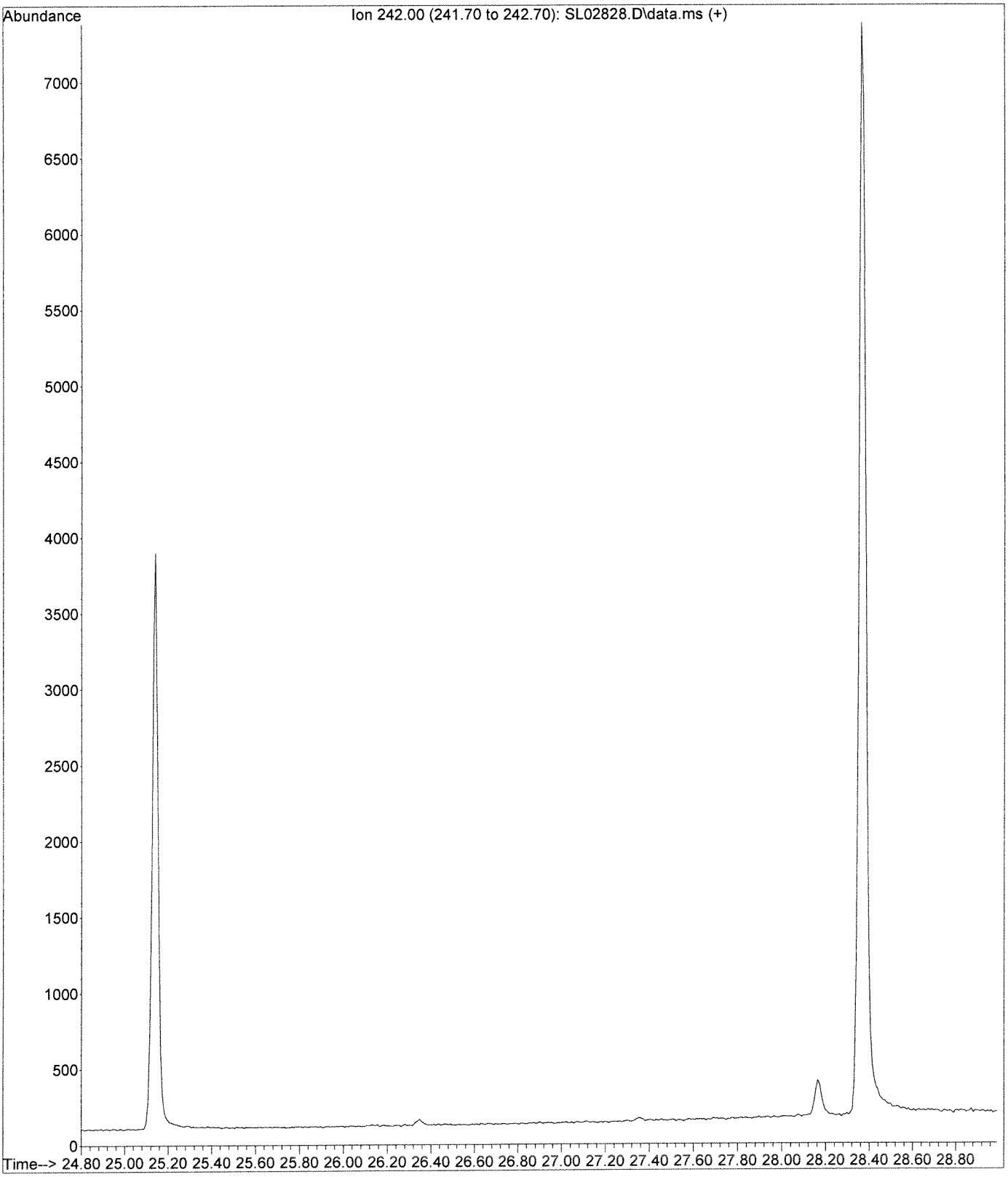
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02828.D
Operator : Syslo
Acquired : 15 Feb 2012 13:43 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: Soil Blank
Misc Info : 1200024-Blk1
Vial Number: 82



US EPA ARCHIVE DOCUMENT

Sample 017-0002

TPH

Characterization

+

TGI+ report

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02829.D
Acq On : 15 Feb 2012 14:27
Operator : Syslo
Sample : SERAS-017-0002
Misc : 30g to 1.0mL
ALS Vial : 83 Sample Multiplier: 1

Quant Time: Feb 15 15:17:56 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	419430	10.00	ug/mL	-0.02
5) d30-Tetradecane	13.532	66	129686	10.00	ug/mL	-0.02
6) d50-Tetracosane	23.784	66	144537	10.00	ug/mL	-0.02
8) d74-Hexatriacontane	34.637	66	130132	10.00	ug/mL	-0.02
System Monitoring Compounds						
2) d10-Anthracene {S}	18.830	188	514605	11.54	ug/mL	0.00
Spiked Amount 20.000			Recovery = 57.70%			
3) d14-o-Terphenyl {S}	19.733	244	243105	12.40	ug/mL	-0.02
Spiked Amount 20.000			Recovery = 62.00%			
4) 5a-Androstane {S}	21.158	260	30879	12.95	ug/mL	0.00
Spiked Amount 20.000			Recovery = 64.75%			
7) d62-Triacontane {S}	28.164	66	176034	14.06	ug/mL	-0.01
Spiked Amount 20.000			Recovery = 70.30%			

Target Compounds Qvalue

#) = qualifier out of range (m) = manual integration (+) = signals summed

TPH = 5723 7733

US EPA ARCHIVE DOCUMENT

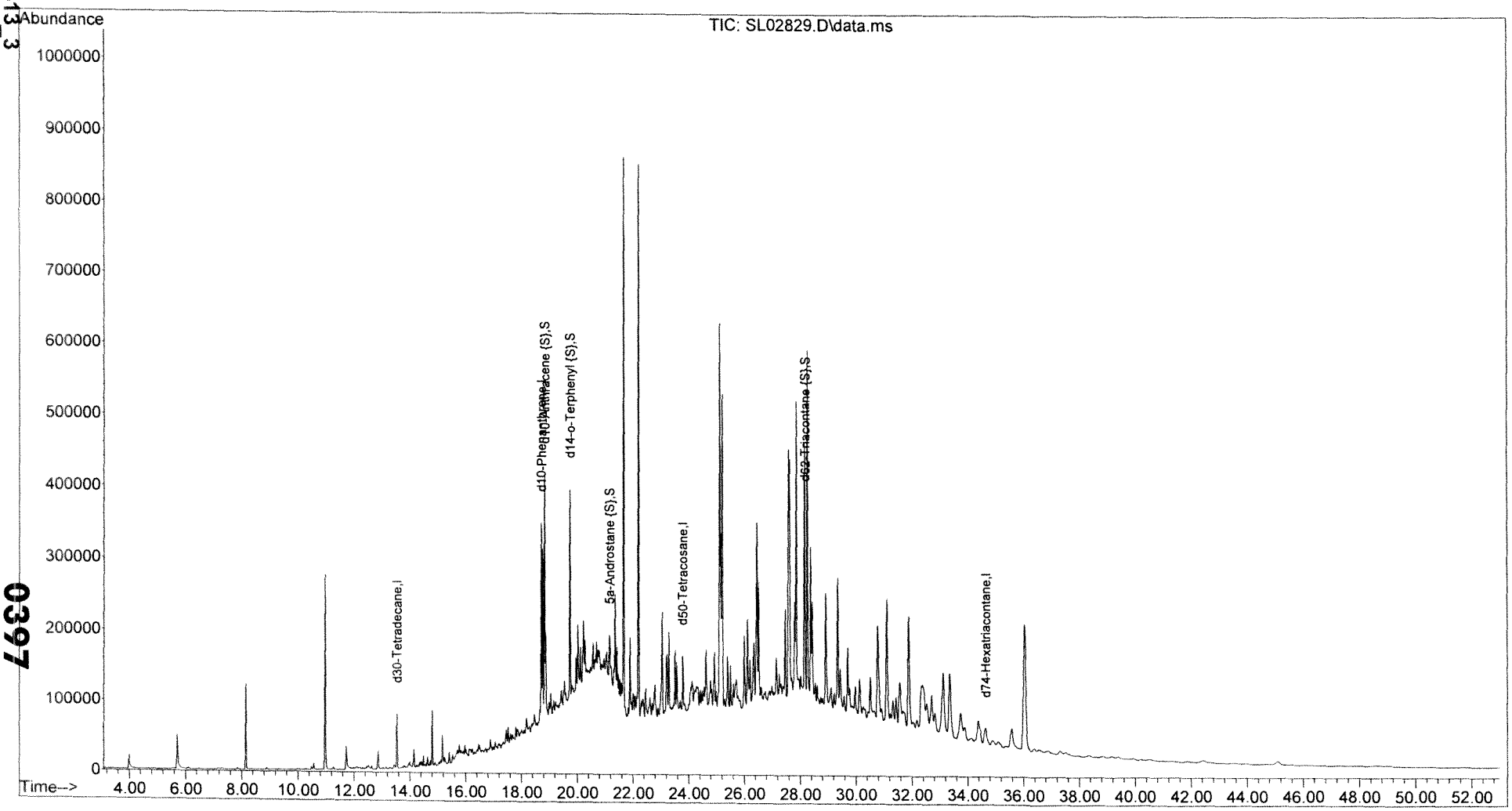
SERAS-017-DTM-011413_3

0397

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02829.D
Acq On : 15 Feb 2012 14:27
Operator : Syslo
Sample : SERAS-017-0002
Misc : 30g to 1.0mL
ALS Vial : 83 Sample Multiplier: 1

Quant Time: Feb 15 15:17:56 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Dec 15 15:26:49 2011
Response via : Initial Calibration



Area Percent Report

Data Path : C:\msdchem\1\DATA\021512\
Data File : SL02829.D
Acq On : 15 Feb 2012 14:27
Operator : Syslo
Sample : SERAS-017-0002
Misc : 30g to 1.0mL
ALS Vial : 83 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 0.5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 5
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Tangent else baseline drop >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL02829.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	27.856	973	2997	5610	rM	459843	57237733	100.00%	100.000%

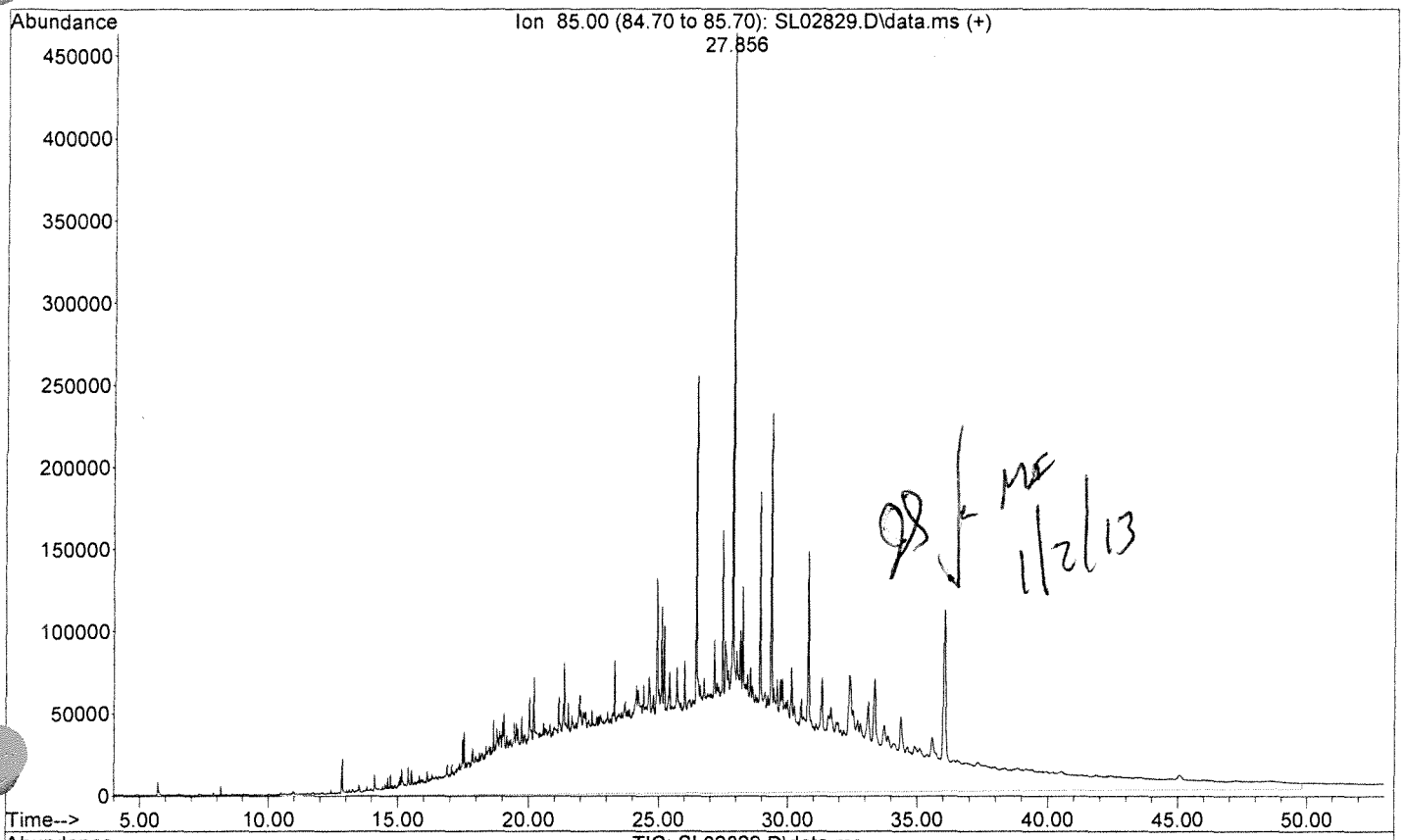
Sum of corrected areas: 57237733

DRO/TPH051611.M Wed Jan 02 16:39:59 2013 SLICK2

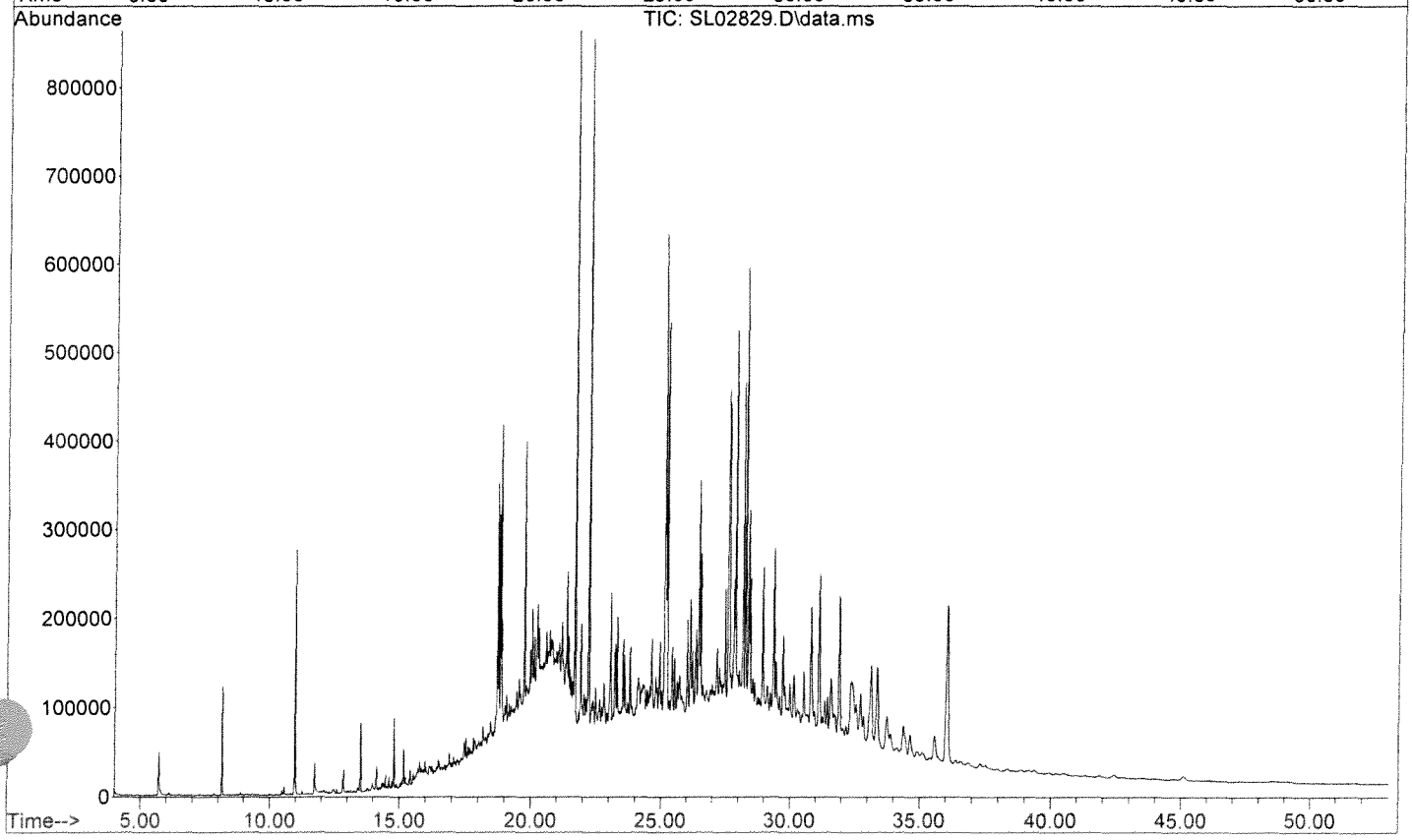
FOR M.I.
DATE: 1/2/13

DATE: 1/2/13

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



98 ✓ MS 1/2/13



```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

US EPA ARCHIVE DOCUMENT

ENOfingFinal.mac: This macro ran the following 8 nested macros to produce VDR's and fingerprints:

TICTPH.mac: Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the TPH fingerprint generated by macro **TPHSERAS.mac**.

TPH8583stacked.mac: A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints.

TPHSERAS.mac: This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183 and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using the *Draft* SERAS GC/MS TPH SOP# 1841. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.

ENOTPHSEPN.mac: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).

Hopster.mac: This is an example of a stacked set of fingerprints of the ion 191 hopane over the ion 217 sterane biomarker compounds. It's a simple VDR with many applications.

ParentPAH.mac: Shows the EIC's for *just* the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for just the parent PAH's ; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.

PAH1803N.mac: Illustrates all the alkylated PAH homologues in a staked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).

MCDET.MAC: Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference. This combined alkyl-PAH fingerprint was supplied in a REAC fingerprint report. Supported by the testimony of the former REAC GC/MS chemist who wrote and developed this method, this unique fingerprint was a crucial piece of evidence used by USEPA attorneys to identify and successfully prosecute the PRP (after 6 years of litigation) who released several million gallons of oil into a river. After reviewing the REAC fingerprint report, the USCG Coil Lab changed their SOP to adopt and implement this former REAC, now SERAS parameter.

The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739:

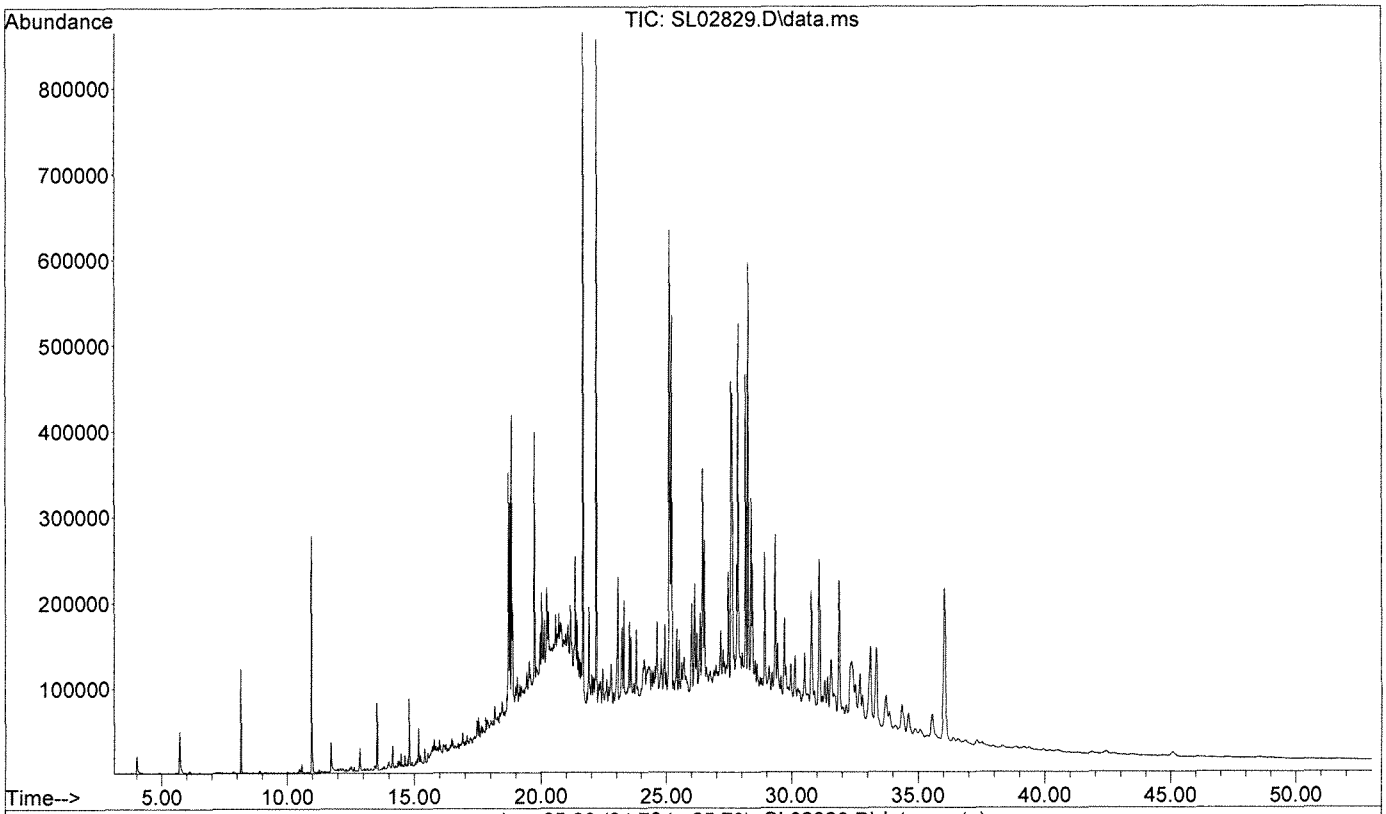
FireF117.mac: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications on unknown site oils.

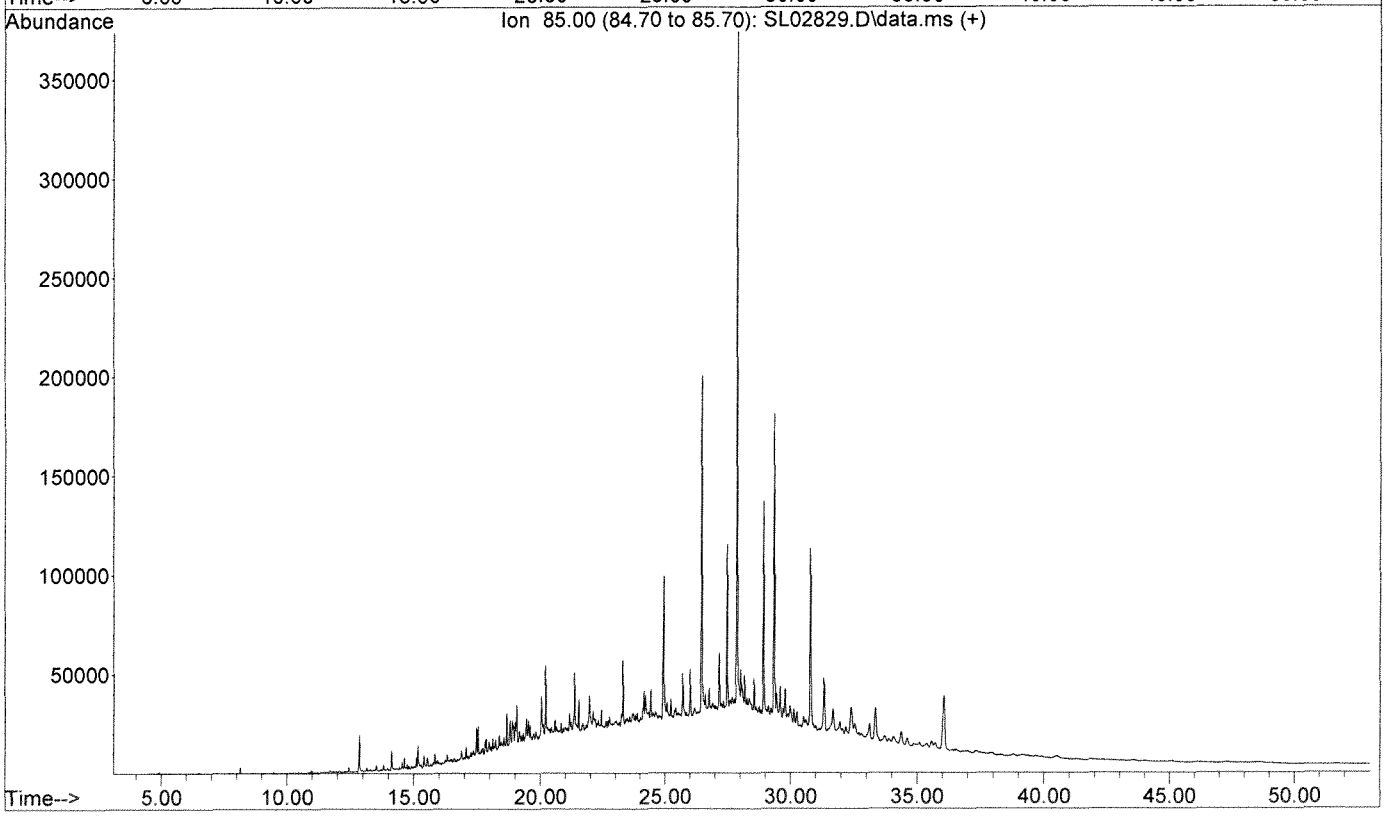
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



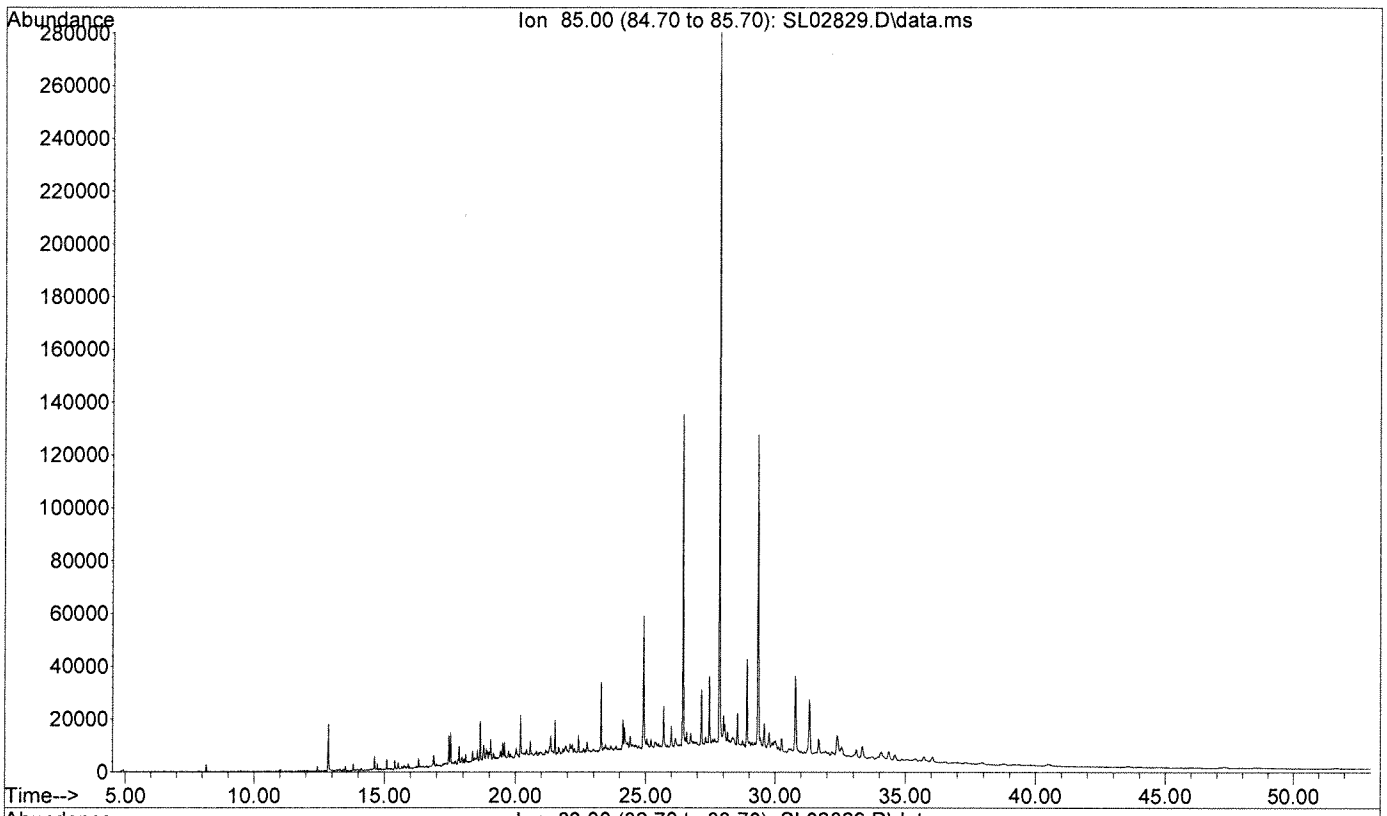


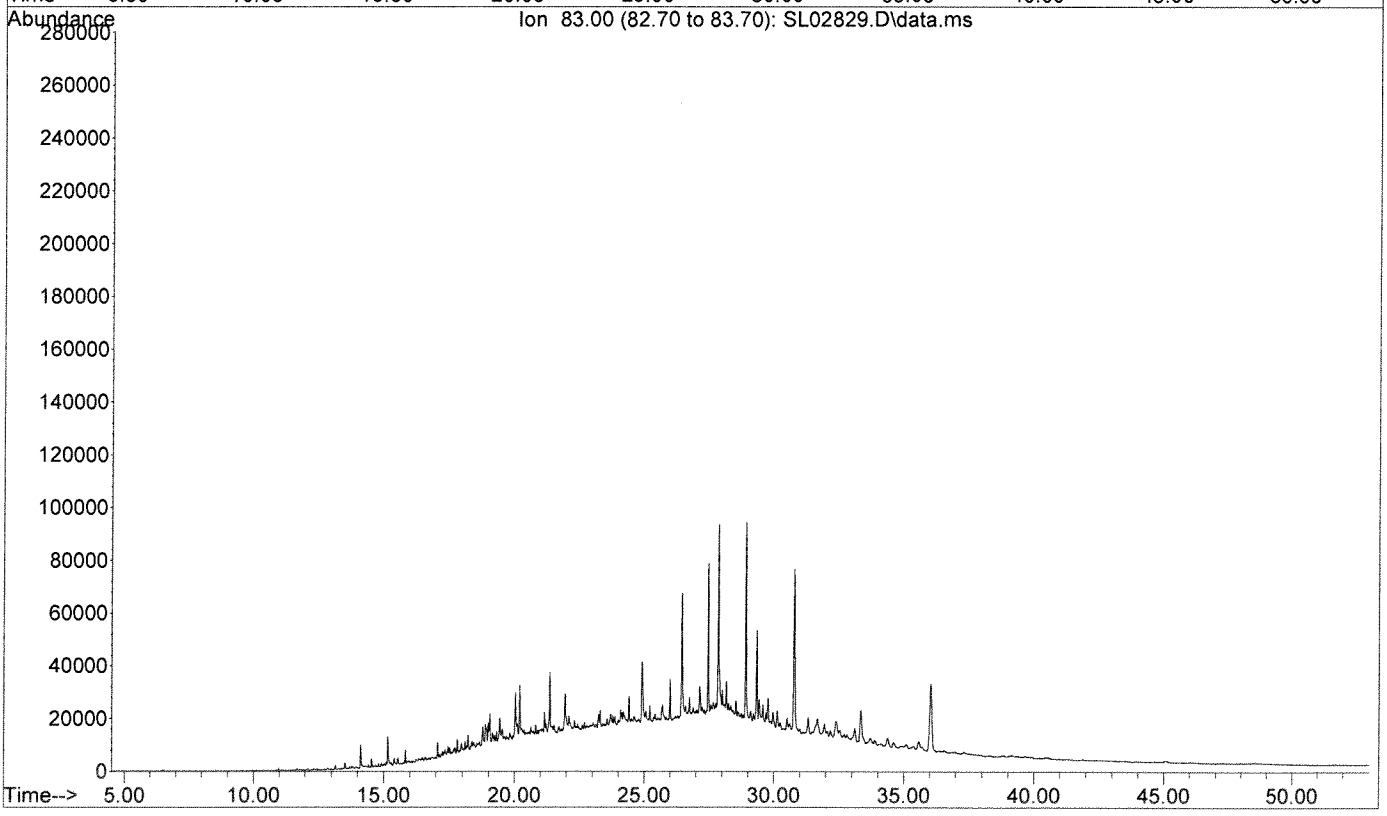
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83





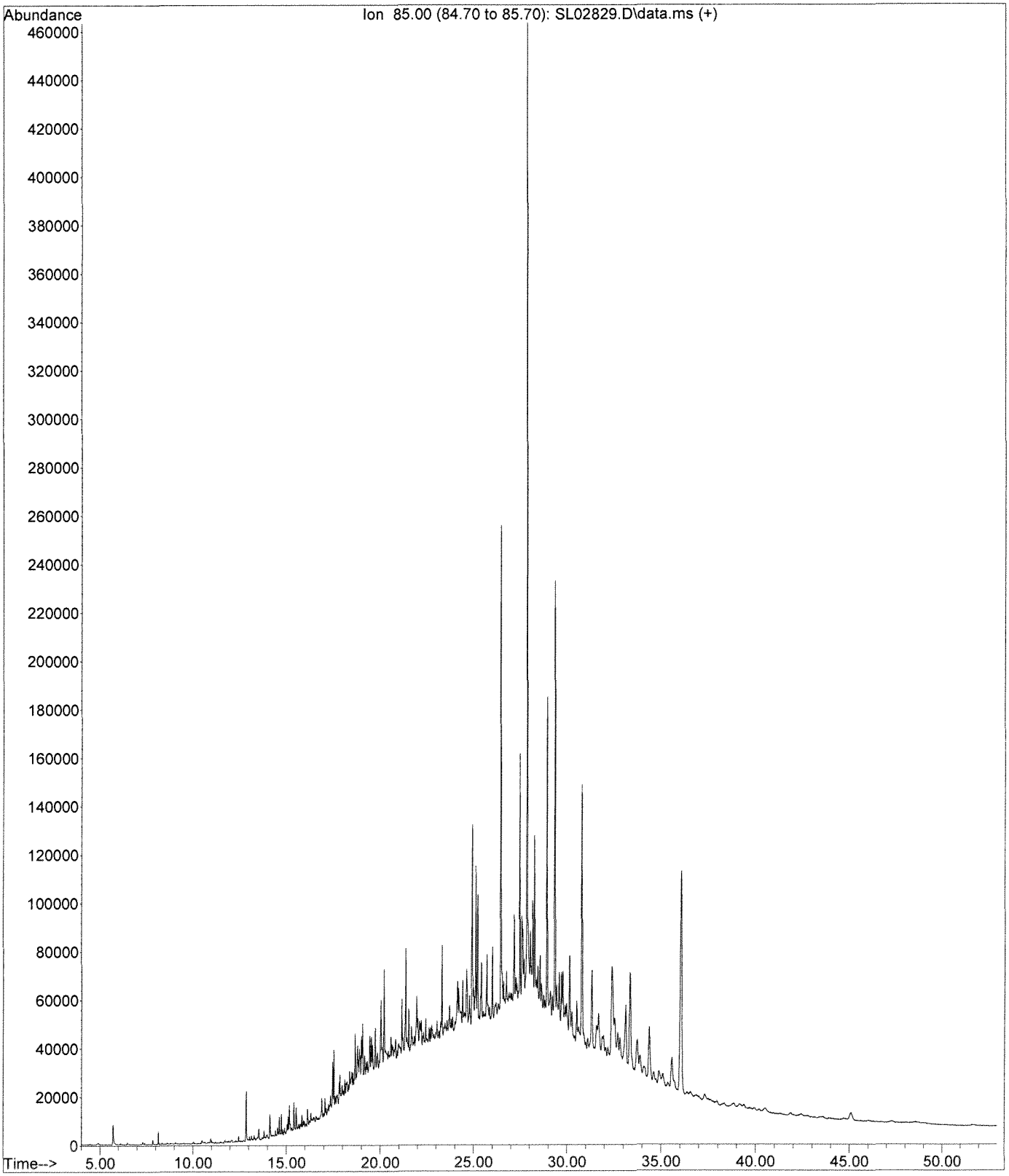
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012   14:27      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



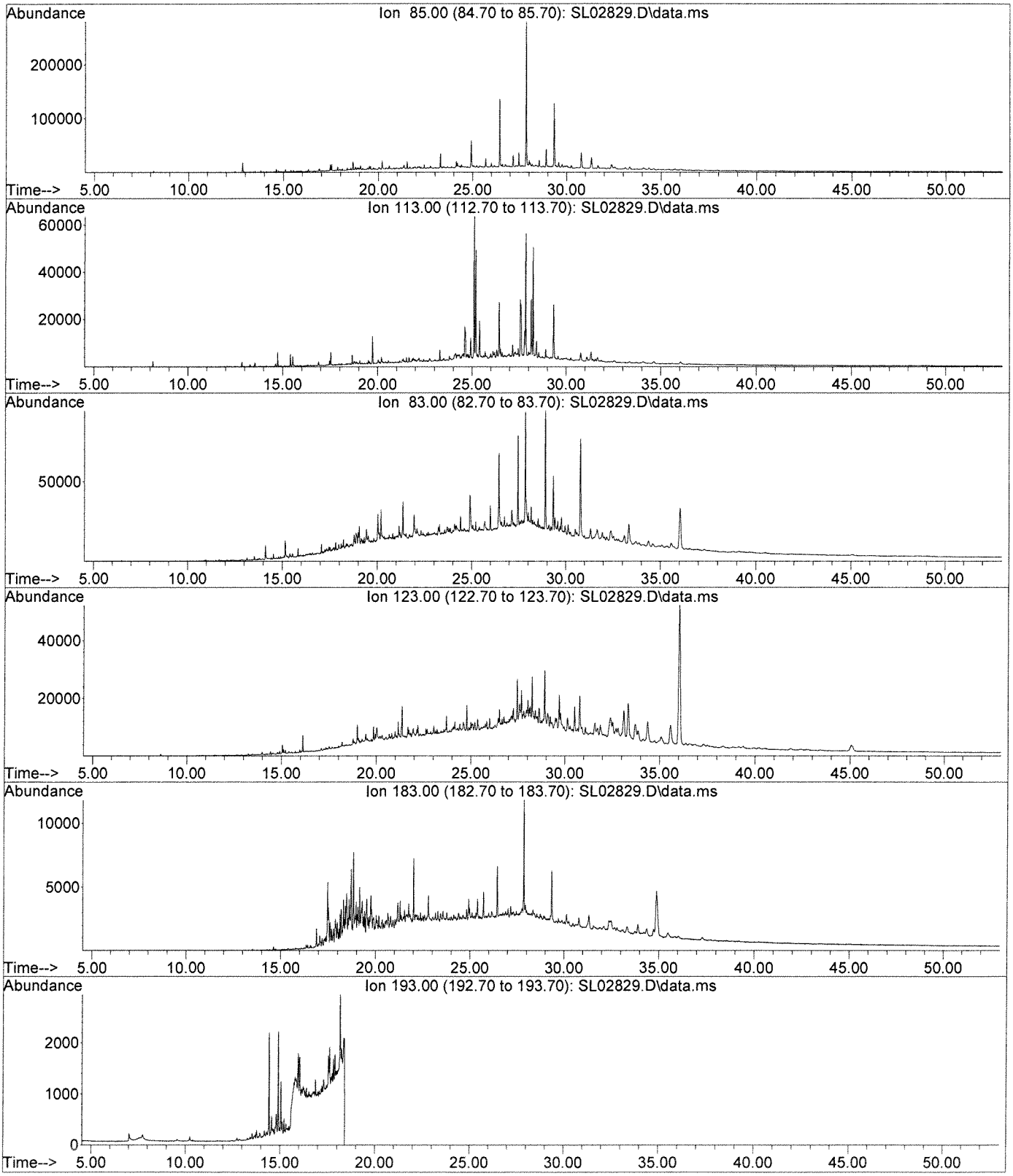
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012  14:27      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



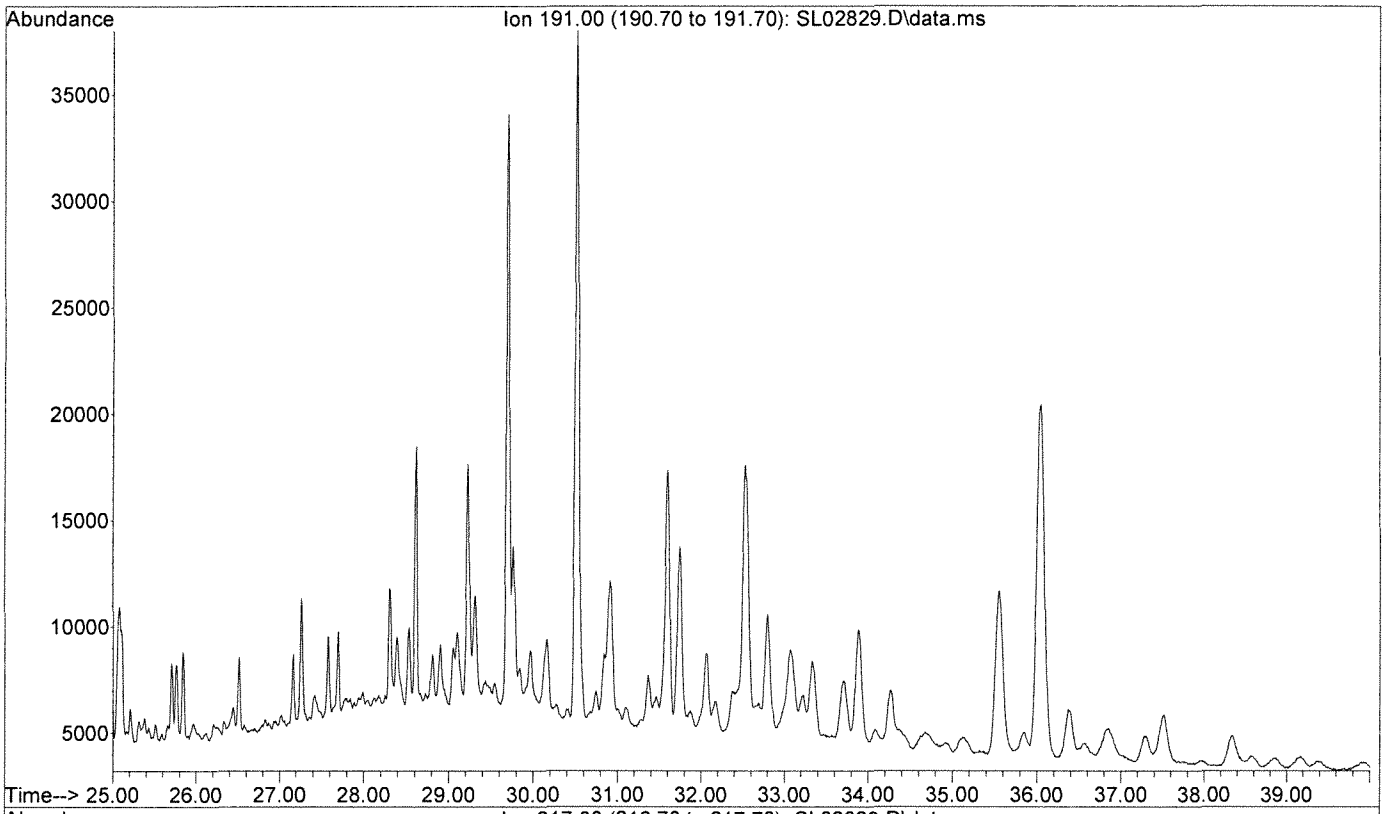
US EPA ARCHIVE DOCUMENT

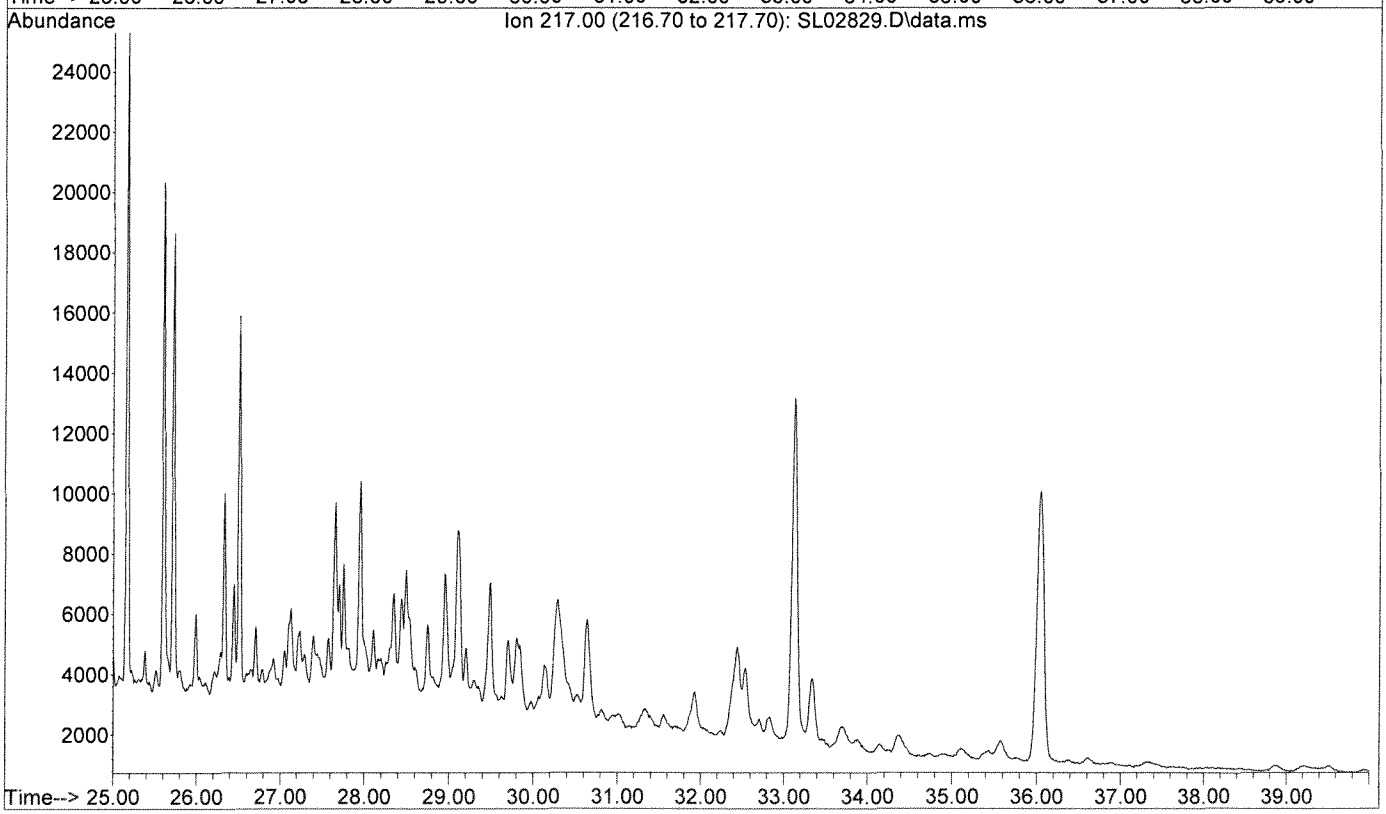
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83





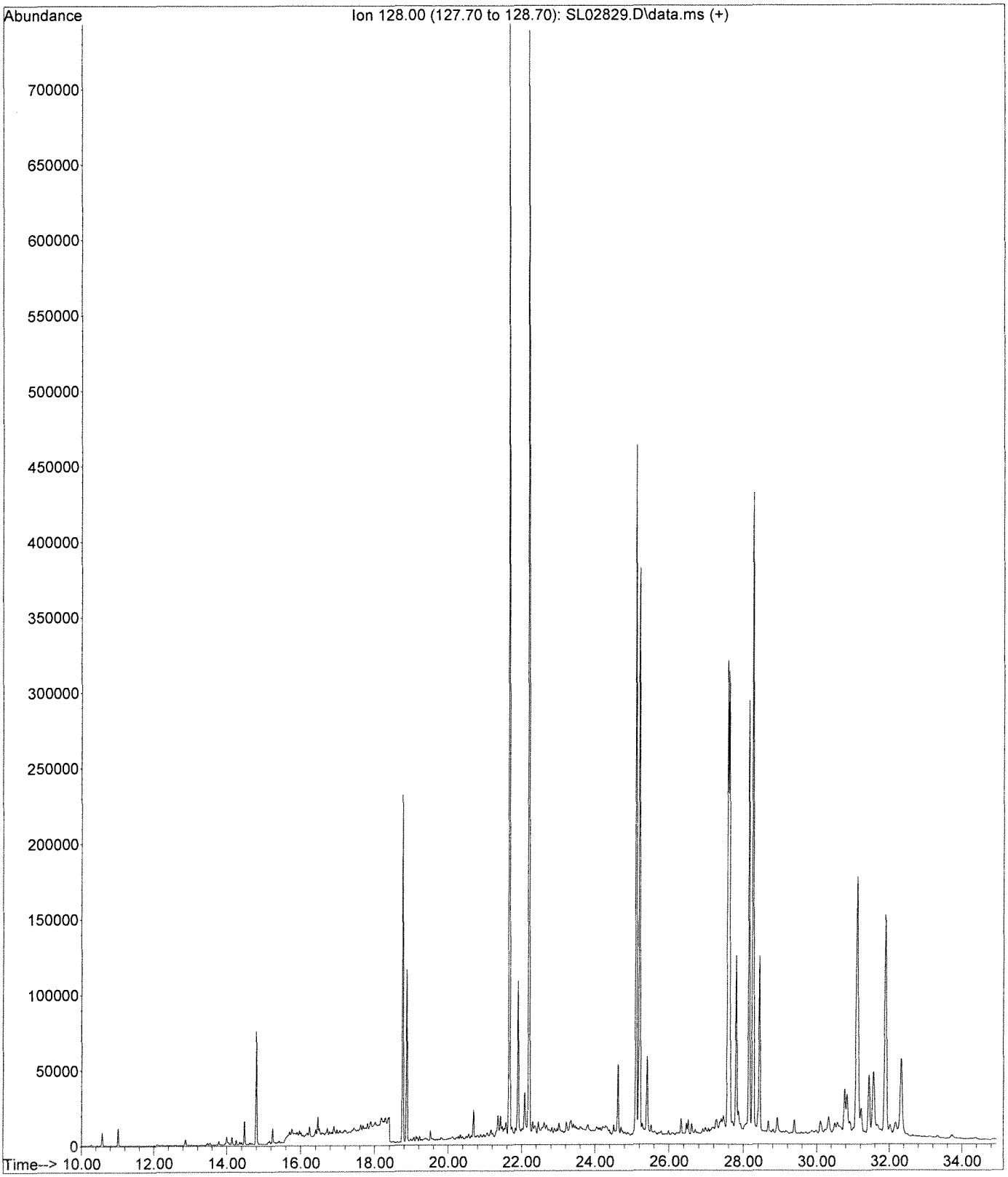
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



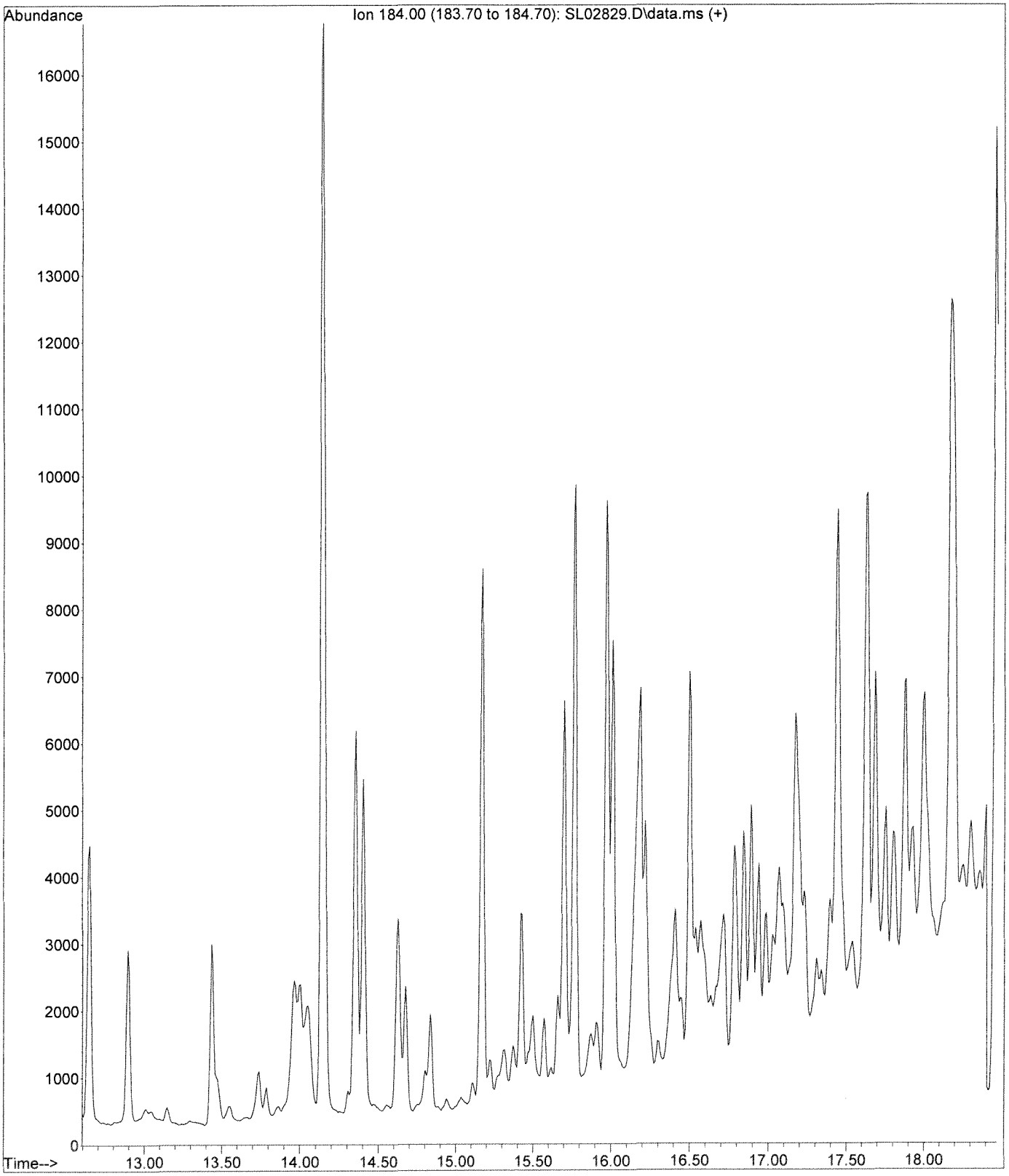
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

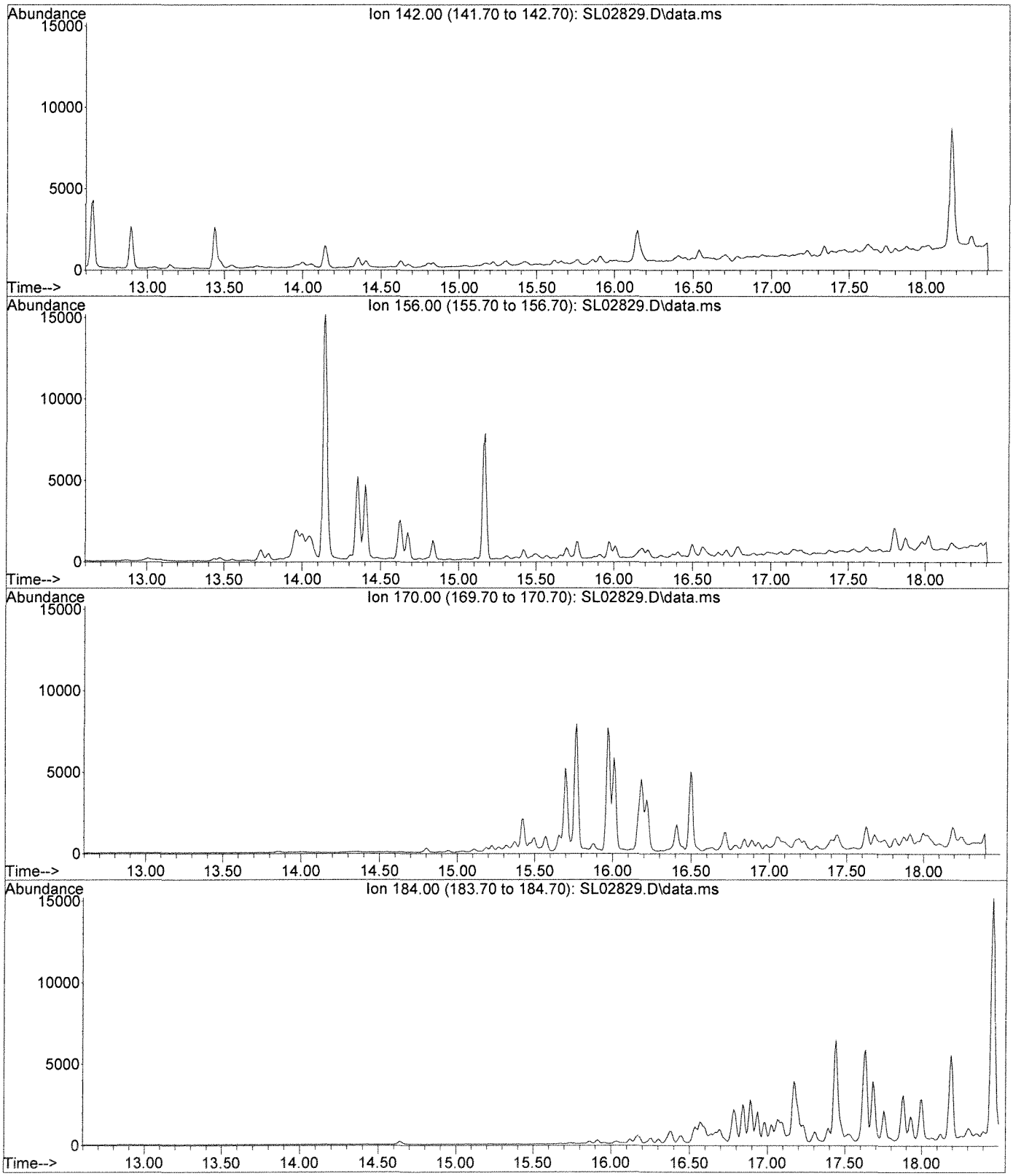
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



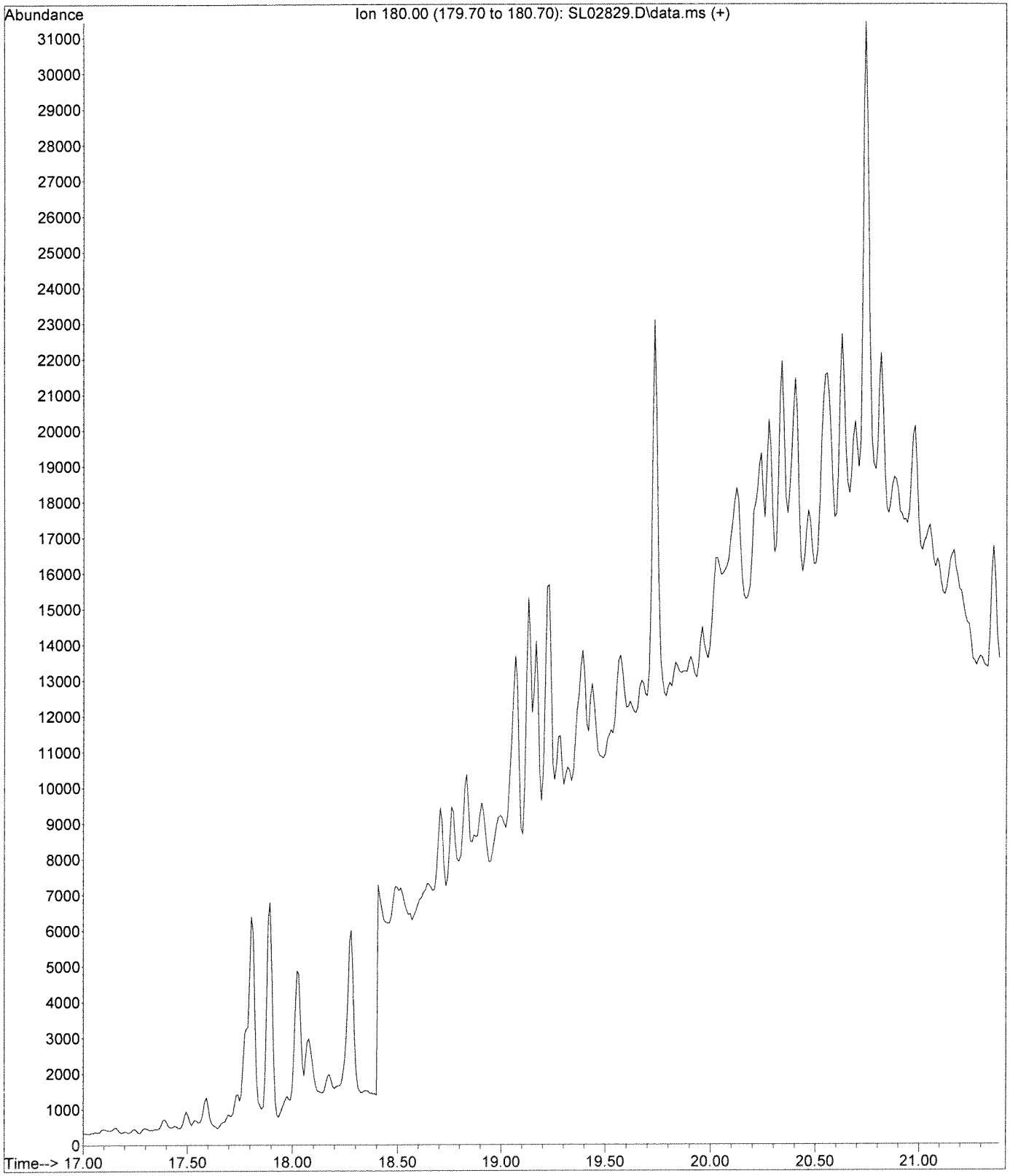
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012   14:27      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



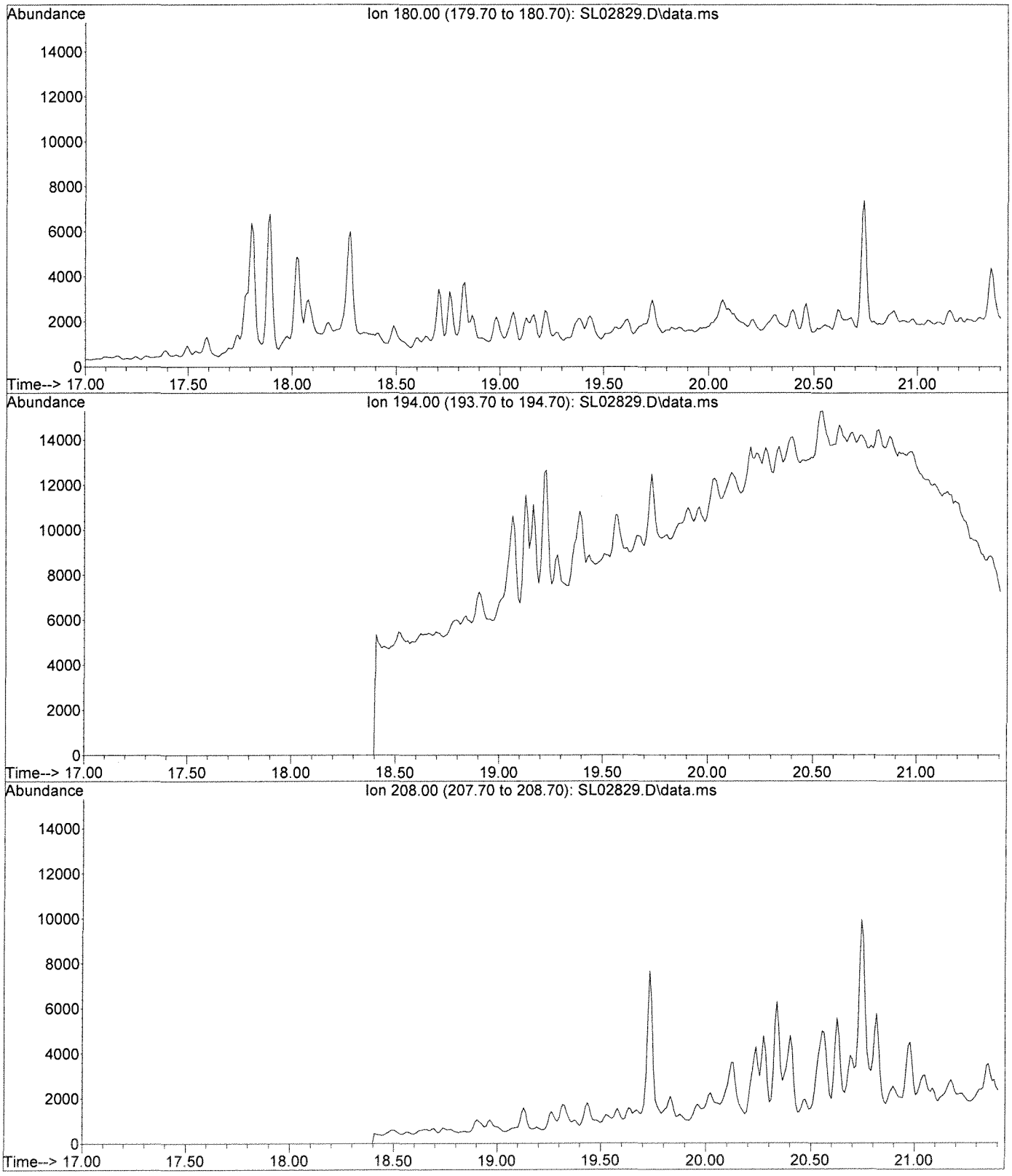
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012  14:27      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



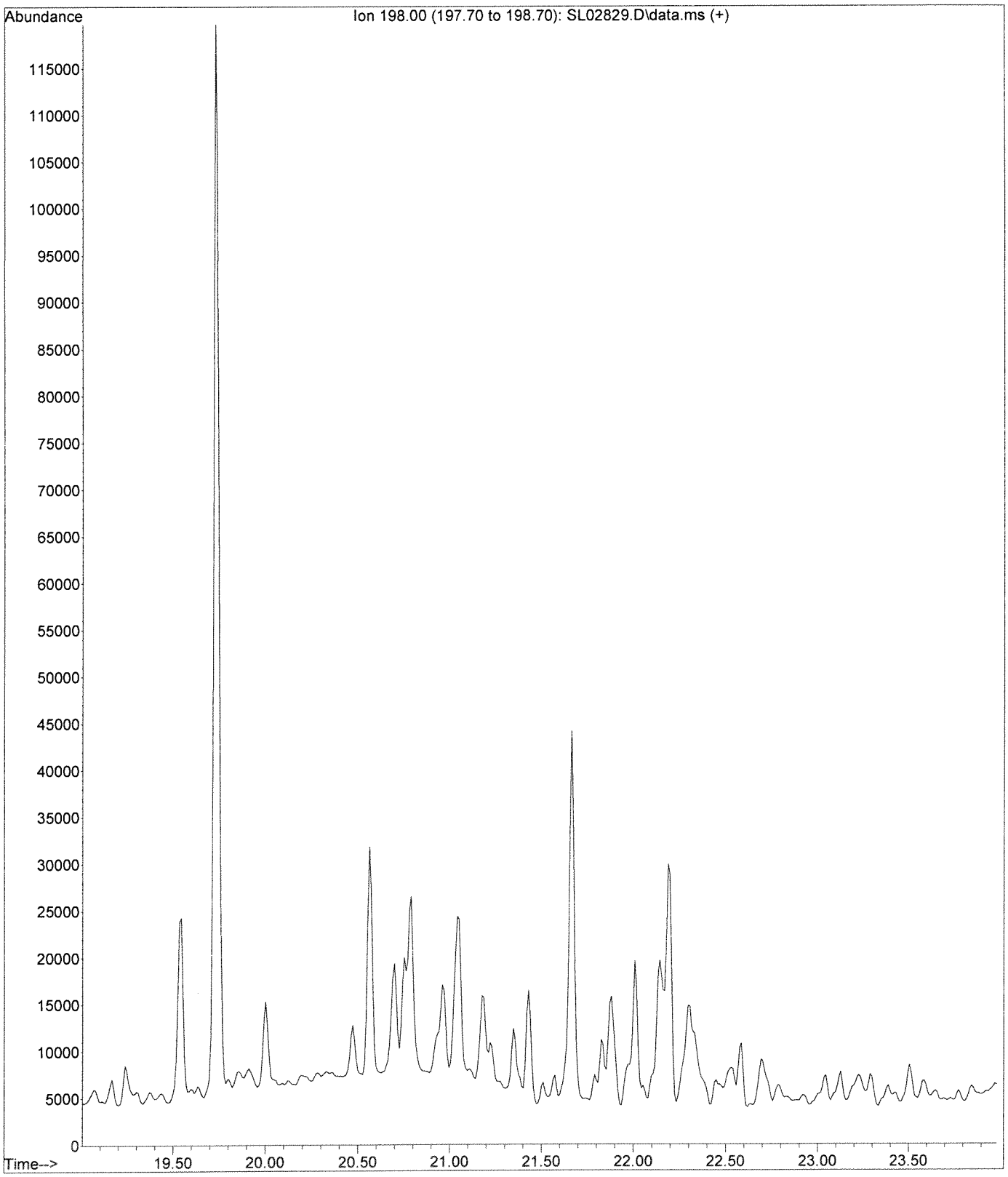
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012  14:27      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



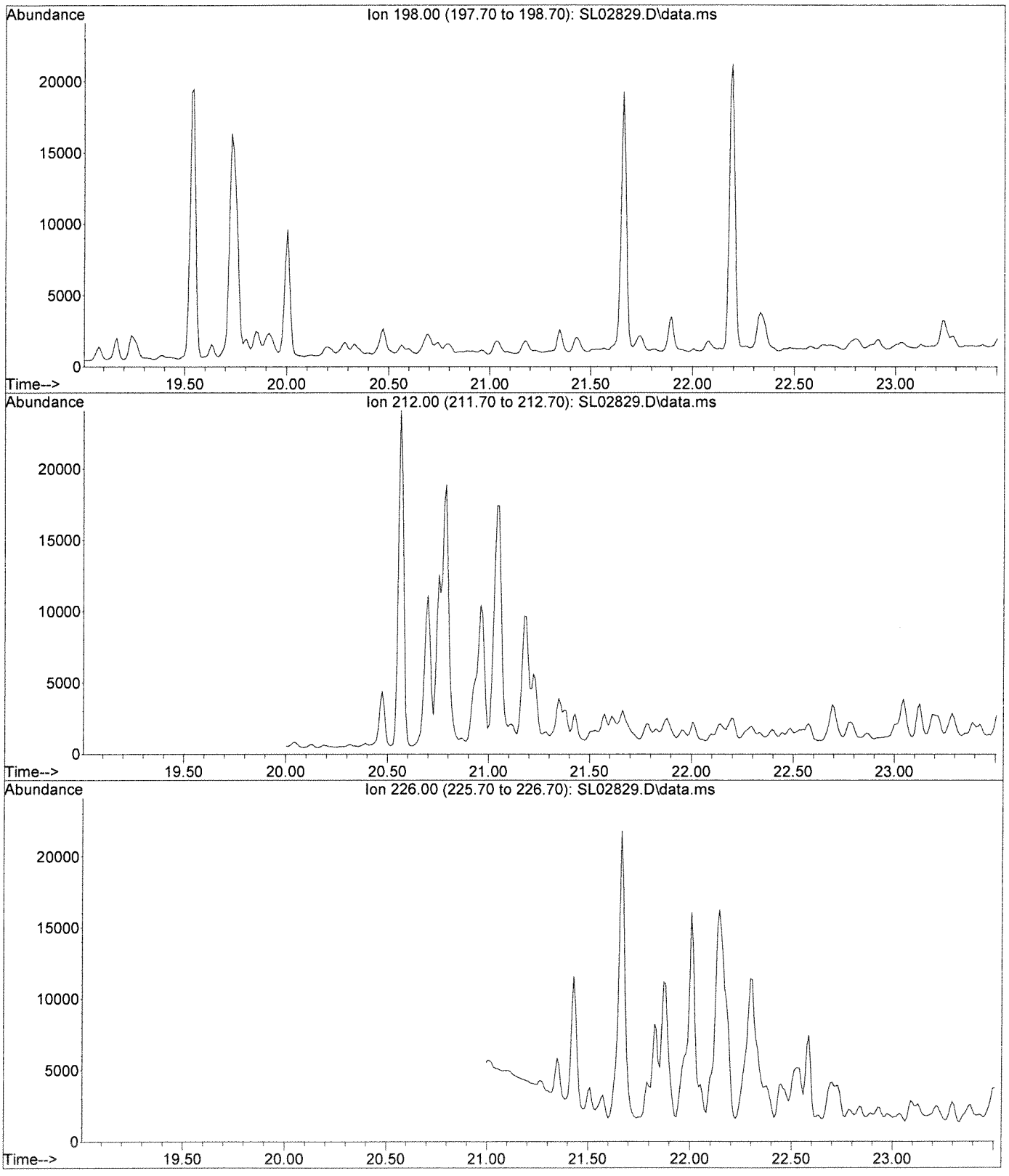
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



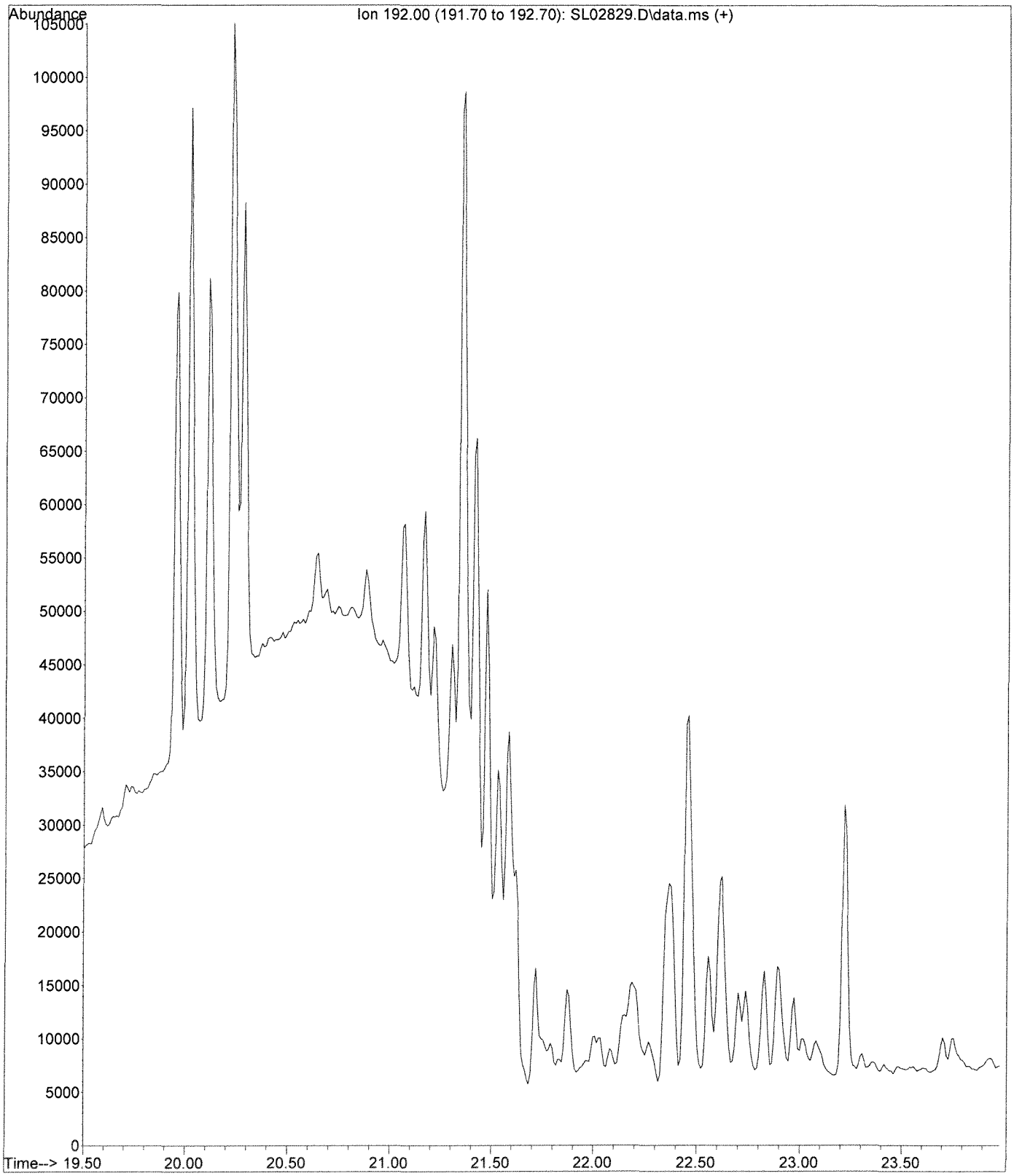
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012  14:27      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



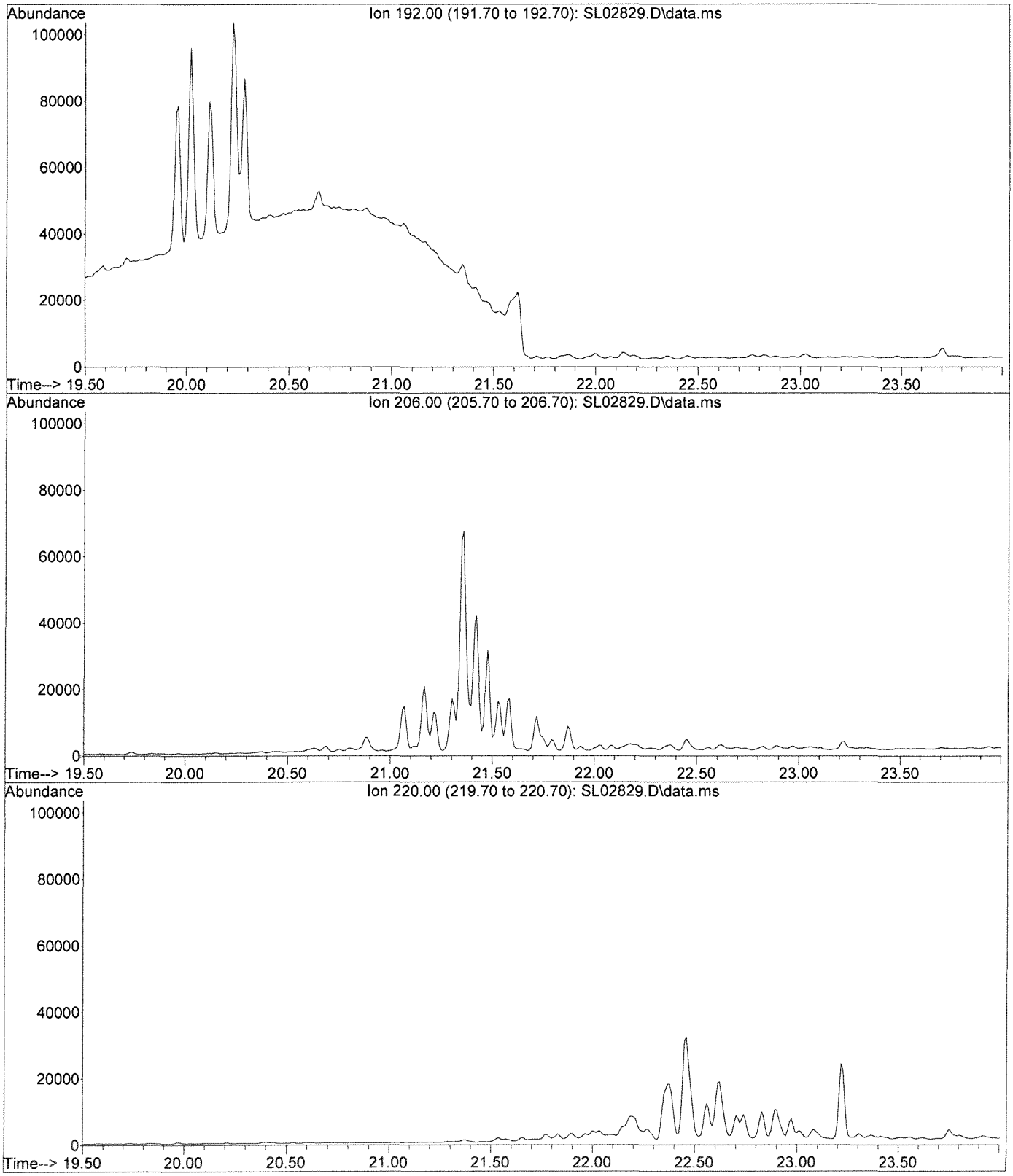
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



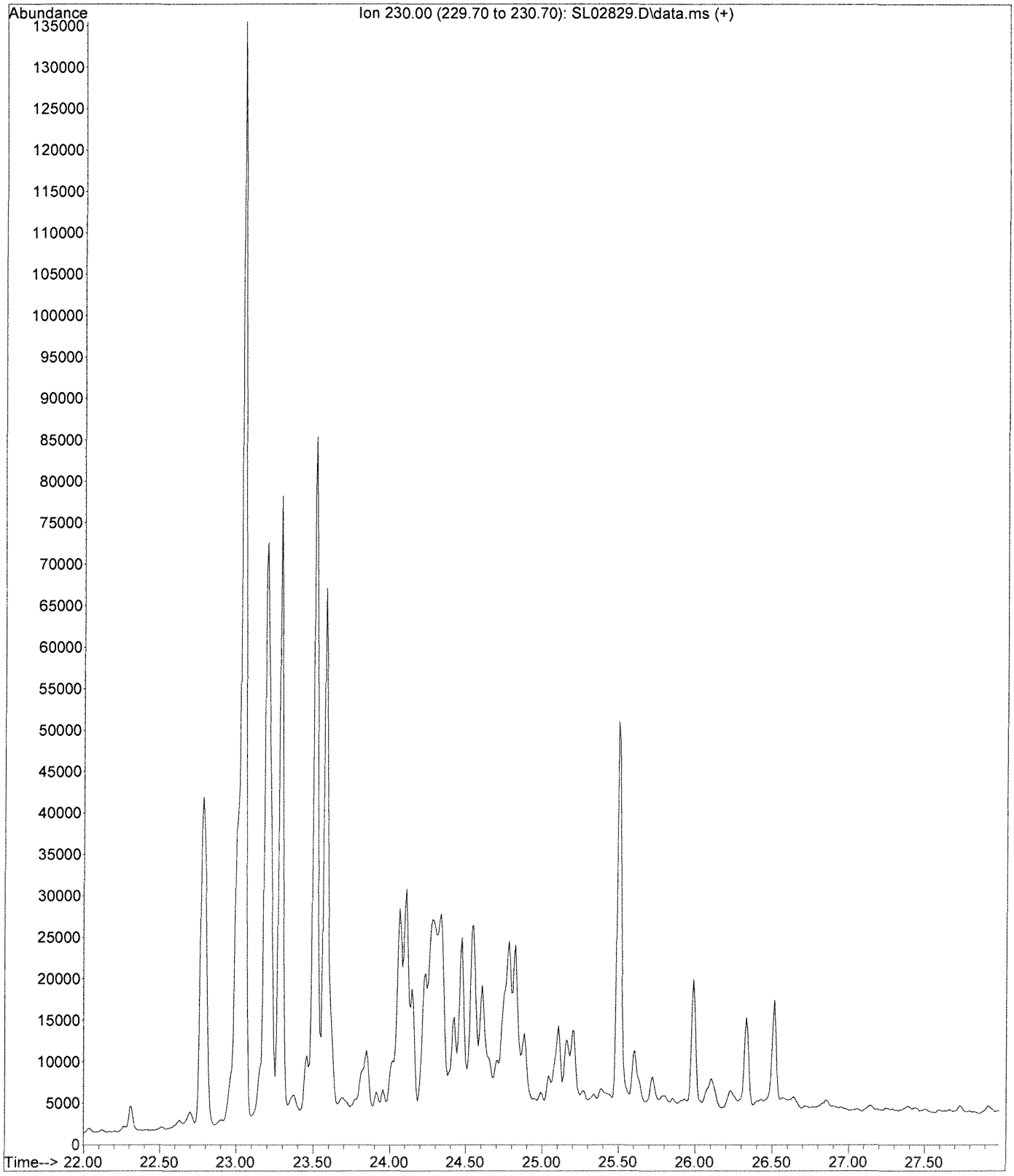
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



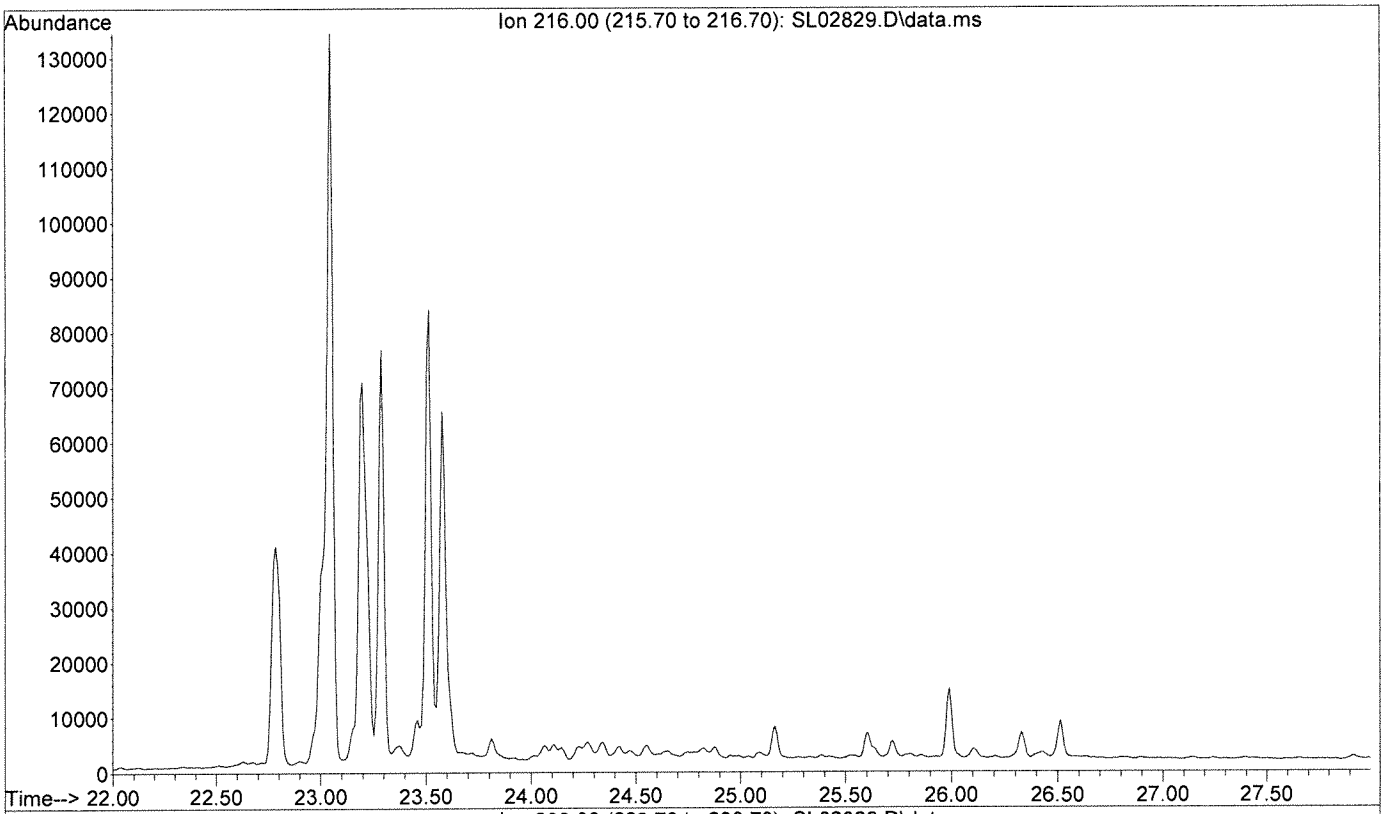
US EPA ARCHIVE DOCUMENT

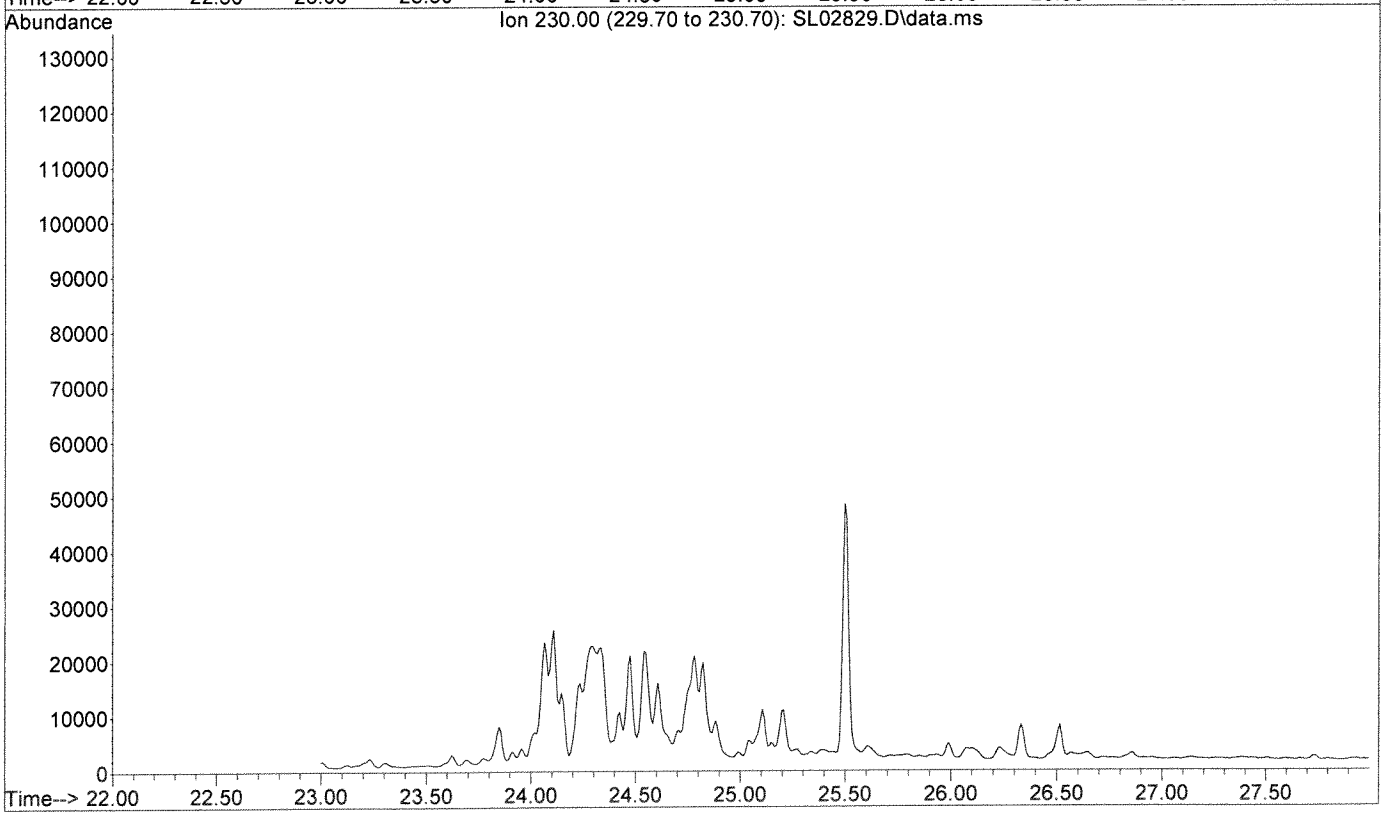
File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



US EPA ARCHIVE DOCUMENT

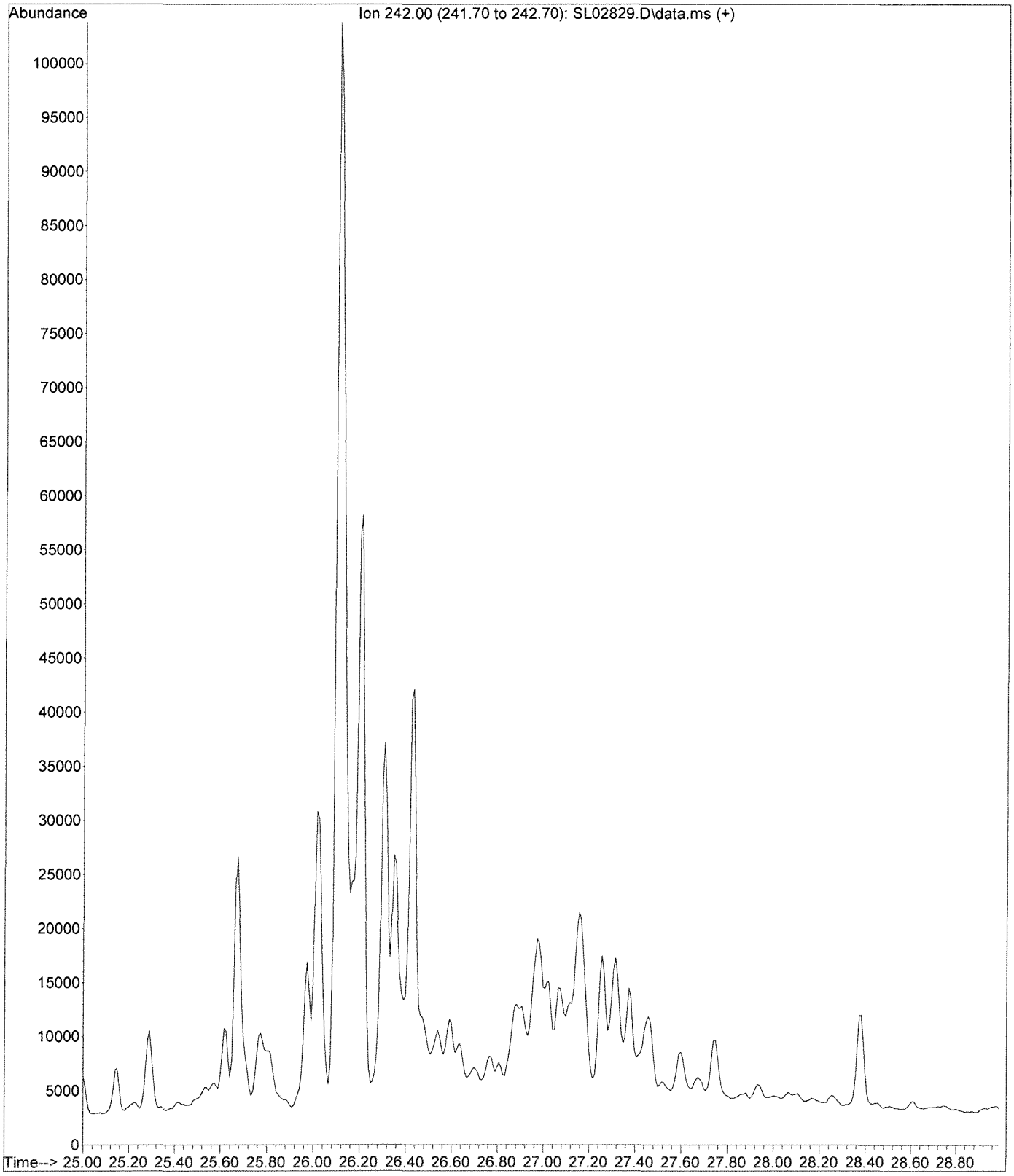
```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012   14:27      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```





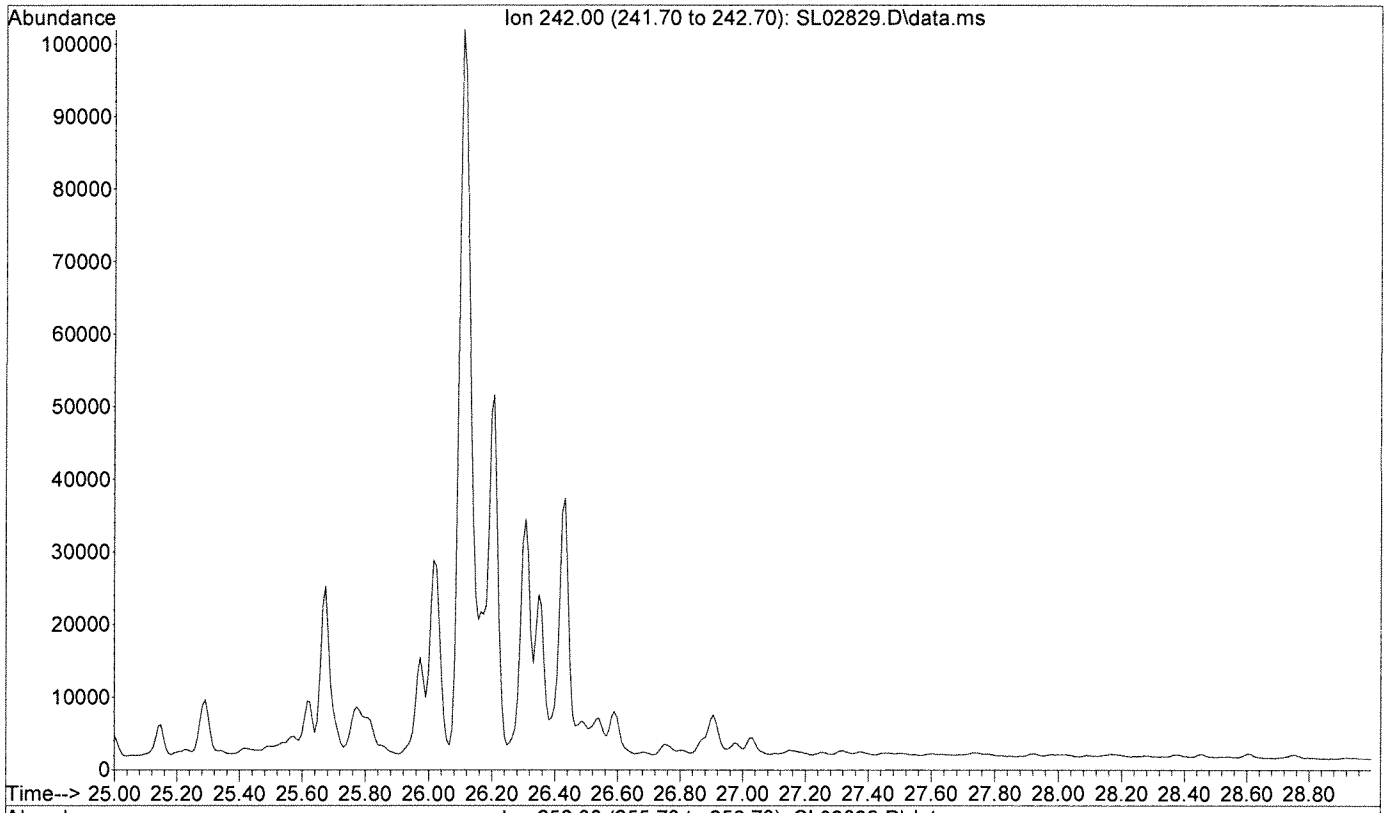
US EPA ARCHIVE DOCUMENT

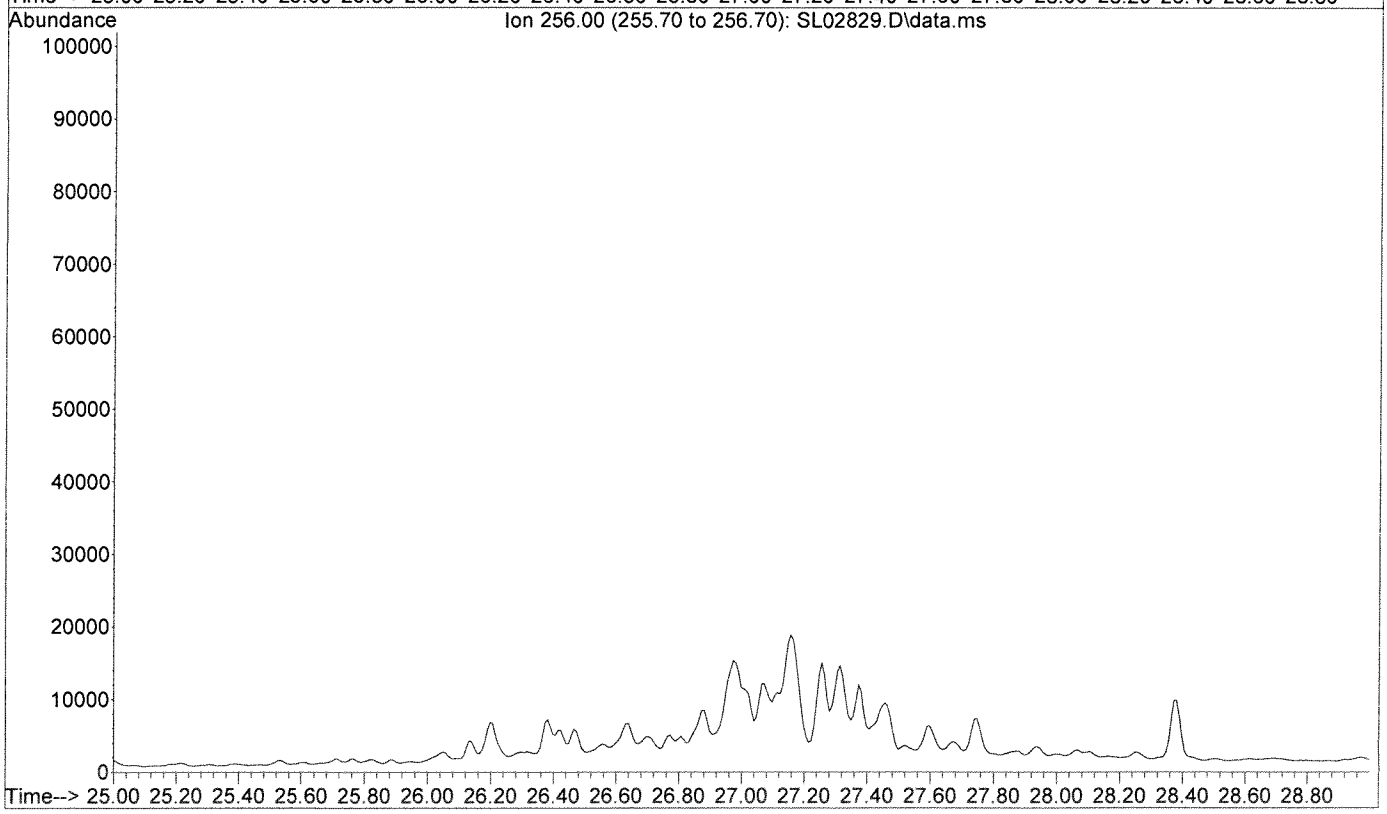
```
File        :C:\msdchem\1\DATA\021512\SL02829.D
Operator    : Syslo
Acquired    : 15 Feb 2012  14:27      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0002
Misc Info   : 30g to 1.0mL
Vial Number: 83
```



US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83





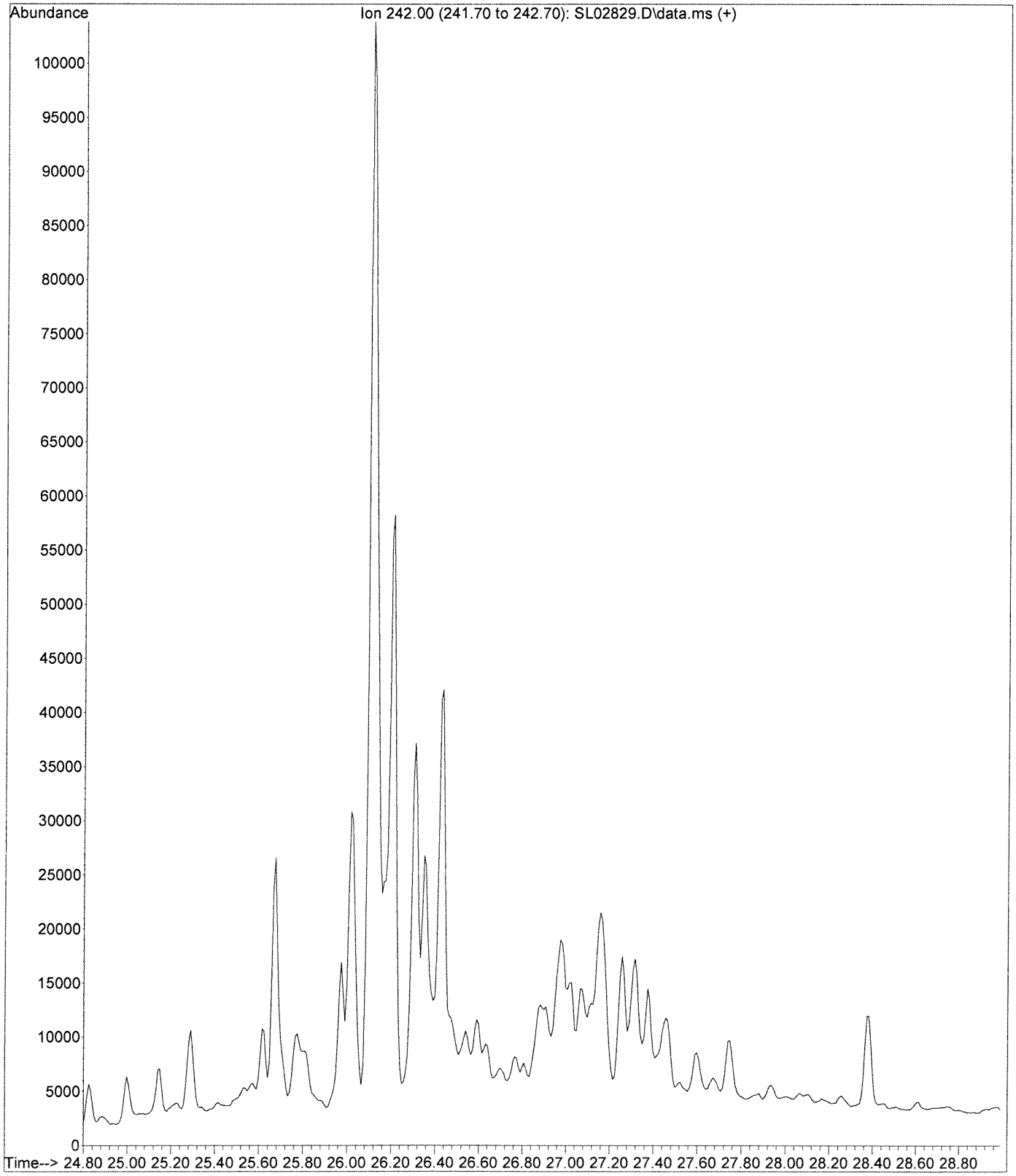
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\021512\SL02829.D
Operator : Syslo
Acquired : 15 Feb 2012 14:27 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0002
Misc Info : 30g to 1.0mL
Vial Number: 83



US EPA ARCHIVE DOCUMENT

Logbook # SERAS-I-0121

Page 86

Enbridge Oil + Flemingsburg

SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DROTPH05(6)1.M

Project Name: Enbridge Oil — Method File: DFTPP/ OILS/MS2

Work Assign.#: 6-017 — Inj. Volume: 1ul

Analysis/Date: TPH + Fingerprint 3/27/12 — Analyst: Split

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02870	50ppm DFTPP	RVB00025	2/27/12	15:07	ok 1396-1382
71	SL02871	1000ppm TPH CCV	RVB0003		15:31	
72	SL02872	10ppm SHC/PAH Resolution Mix	RTL0035		16:32	MALL DEC ok
73	SL02873	10K CG-117 ref oil	RVL0003	2/27/12	17:34	
~~1~~	~~SL02874~~	~~BLK022712WD~~	~~Re-extracted~~		X	~~WATER DIL Blank~~
1	SL02875	BLK022712WD	Blank		X	ok
1	SL02876	BLK022712WD	Blank		18:44	
2	SL02877	SERAS-017-0003			19:38	
3	SL02876	SERAS-017-0003dup			20:35	
4	SL02877	SERAS-017-0004			21:37	
5	SL02878	SERAS-017-0004			22:38	SKIM from Bucket
6	SL02879	SERAS-017-0003dup		2/27/12	23:40	Ear dup
7	SL02880	10K CG-117 ref oil	RVL0003	2/28/12	00:41	D-1779 ref
~~8~~	~~SL02881~~	~~SERAS-017-0003 by Enviro~~	~~Enviro~~	~~2/28/12~~	1:42	~~Qual~~
—	SL	only by DFTPP	3/1/12			
99	SL02882	50ppm DFTPP	3/1/12	RVB0025	8:24	PASS 1395-1283
71	SL02883	10ppm SHC/PAH Resolution Mix	RTL0035	3/1/12	12:50	
72	SL02884	10K CG-117 ref oil	RVL0003		13:51	
1	SL02885	1000ppm TPH CCV/Surr	RVB0003		14:56	
2	SL02886	RL@30/12	Blank		15:41	0.1g → 10ml
3	SL02887	FOR-TRENCH	R202018-01		17:33	0.055g → 5.0ml
4	SL02888	FOR-TRENCH dup	R202018-01dup		18:34	0.105g → 5.0ml
5	SL02889	FOR-CREEK			19:36	0.148g → 5.0ml
6	SL02890	SERAS-017-0005			20:37	0.104g → 10ml
7	SL02891	SERAS-017-0005 dup			21:37	0.115g → 10ml
8	SL02892	SERAS-017-0006			22:38	0.102g → 10ml
9	SL02893	10K Ambient Oil		3/1/12	23:39	Used Motor Oil
10	SL02894	10K CG-117 Reference	RVL0003	3/2/12	00:40	
11	SL02895	CMC REO	CMC 0.1	3/2/12	1:40	0.95g → 10ml

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB00025	DFTPP	8/14/12	50ppm		
2	RVB0003	Calibration Check	8/12/12	1000ppm		TPH
3	RVB00058	Internal Standard	5/14/12	500ppm		Spk 20µL → 5ml
4	RVL0003	CG-117 Ref. Eng	5/24/12	10 Kit		D-1779 ref.
5	RTL0035	SHC/PAH Mix	Gum	1000ppm		Res/Mod Disc
6						

Reviewed By: Jaime Odneal — Date Checked: 03-06-12

US EPA ARCHIVE DOCUMENT

Sequence Name: C:\msdchem\1\sequence\022712.s
Comment: Enbridge Oil
Operator: Syslo
Data Path: C:\MSDCHEM\1\DATA\022712\
Instrument Control Pre-Seq Cmd:
Data Analysis Pre-Seq Cmd:

Instrument Control Post-Seq Cmd:
Data Analysis Post-Seq Cmd:

Method Sections To Run
(X) Full Method
( ) Reprocessing Only

On A Barcode Mismatch
(X) Inject Anyway
( ) Don't Inject

Line		Sample Name/Misc Info
1) Sample	99	SL02870 DFT8270D 50 DFTPP
2) Sample	71	1K DRO/TPH CCV
Datafile		SL02871
Method		DROTPH051611
3) Sample	72	SL02872 OILSIMS2 10ppm SHC/PAH
4) Sample	73	SL02873 OILSIMS2 10K CG-117 Reference
5) Sample	1	BLK022712WD
Datafile		SL02874
Method		DROTPH051611
6) Sample	2	SERAS-017-0003
Datafile		SL02875
Method		DROTPH051611
7) Sample	3	SERAS-017-0003 dup
Datafile		SL02876
Method		DROTPH051611
8) Sample	4	SERAS-017-0004
Datafile		SL02877
Method		DROTPH051611
9) Sample	5	SERAS-017-000x
Datafile		SL02878
Method		DROTPH051611
Sample	6	SERAS-017-0003 EOS
Datafile		SL02879
Method		DROTPH051611
11) Sample	7	SL02880 OILSIMS2 10K CG-117 Reference
~~12) Sample~~	~~2~~	~~SL02881 OILLINS2 SERAS-017-0003 LIN~~

✓ DFTPP
✓ For surrogate
✓ Fing SHC Std.
✓ D5734 Std oil
Blank - Waste dilution
> lay waste
NOT Needed

Samples 0003
0003 dup
0004
and 000 - 3 blank runs

Injection Log

Data Directory: C:\msdchem\1\DATA\022712\

SampleName	MiscInfo	Vial	Multiplier	Injection Time
1) SL02870.D				
50 DFTPP	RVB0025	99	1.000	27 Feb 2012 15:07
2) SL02871.D				
1K DRO/TPH CCV	RVB0003 Exp. 8/12/1	71	1.000	27 Feb 2012 15:31
3) SL02872.D				
10ppm SHC/PAH	RTL0035 RES/MASS DIS	72	1.000	27 Feb 2012 16:32
4) SL02873.D				
10K CG-117 Reference	RUL0003	73	1.000	27 Feb 2012 17:34
5) SL02874.D				
BLK022712WD	10mL RUC0022 + 0.1mL	1	1.000	27 Feb 2012 18:44
6) SL02875.D				
SERAS-017-0003	275-55-05 {0.116g in	2	1.000	27 Feb 2012 19:33
7) SL02876.D				
SERAS-017-0003 dup	275-55-05 {0.111gi n	3	1.000	27 Feb 2012 20:35
8) SL02877.D				
SERAS-017-0004	275-56-24 {0.029g in	4	1.000	27 Feb 2012 21:37
9) SL02878.D				
SERAS-017-000x	Bucket 2 "qual skim"	5	1.000	27 Feb 2012 22:38
10) SL02879.D				
SERAS-017-0003 EOS	275-55-05 EOB Duplic	6	1.000	27 Feb 2012 23:40
11) SL02880.D				
10K CG-117 Reference	RUL0003 EOS Duplica	7	1.000	28 Feb 2012 00:41
12) SL02881.D				
SERAS-017-0003 LIN	Linear Scan of 0003	2	1.000	28 Feb 2012 1:42

GC/MS QA-QC Check Report: 02/27/12

Tune File : C:\msdchem\1\DATA\022712\SL02870.D
Tune Time : 27 Feb 2012 15:07

Daily Calibration File : C:\msdchem\1\DATA\022712\SL02871.D

File	Sample	Surrogate Recovery %				Internal Standard Responses		
SL02871.D								
	1K DRO/TPH	51*	56*	61*	60*	496730	165200	202882
				161132				
No Quant Results for C:\msdchem\1\DATA\022712\SL02872.D								
SL02873.D								
	10K CG-117	0	0	0	0	566477	177045	223004
				204402				
SL02874.D								
	BLK022712W	88*	99*	108*	108*	300975	100642	116776
				95670				
SL02875.D								
	SERAS-017-	87*	96*	104*	112*	306220	99120	125874
-0003				118393				
SL02876.D								
	SERAS-017-	80*	88*	95*	102*	319905	103998	130631
-0003 dup				119796				
SL02877.D								
	SERAS-017-	80*	88*	101*	104*	298775	96470	122101
-0004				113788				
SL02878.D								
	SERAS-017-	0	0	0	0	698465	227918	283369
-0004X				257581				
SL02879.D								
	SERAS-017-	87*	95*	101*	109*	309155	101381	126388
-0003				115104				
SL02880.D								
	10K CG-117	0	0	0	0	558084	174777	210874
				191945				

Created: Thu Jan 10 10:28:46 2013 Slick2

Note- I actually added surrogate to waste dilution

DFTPP

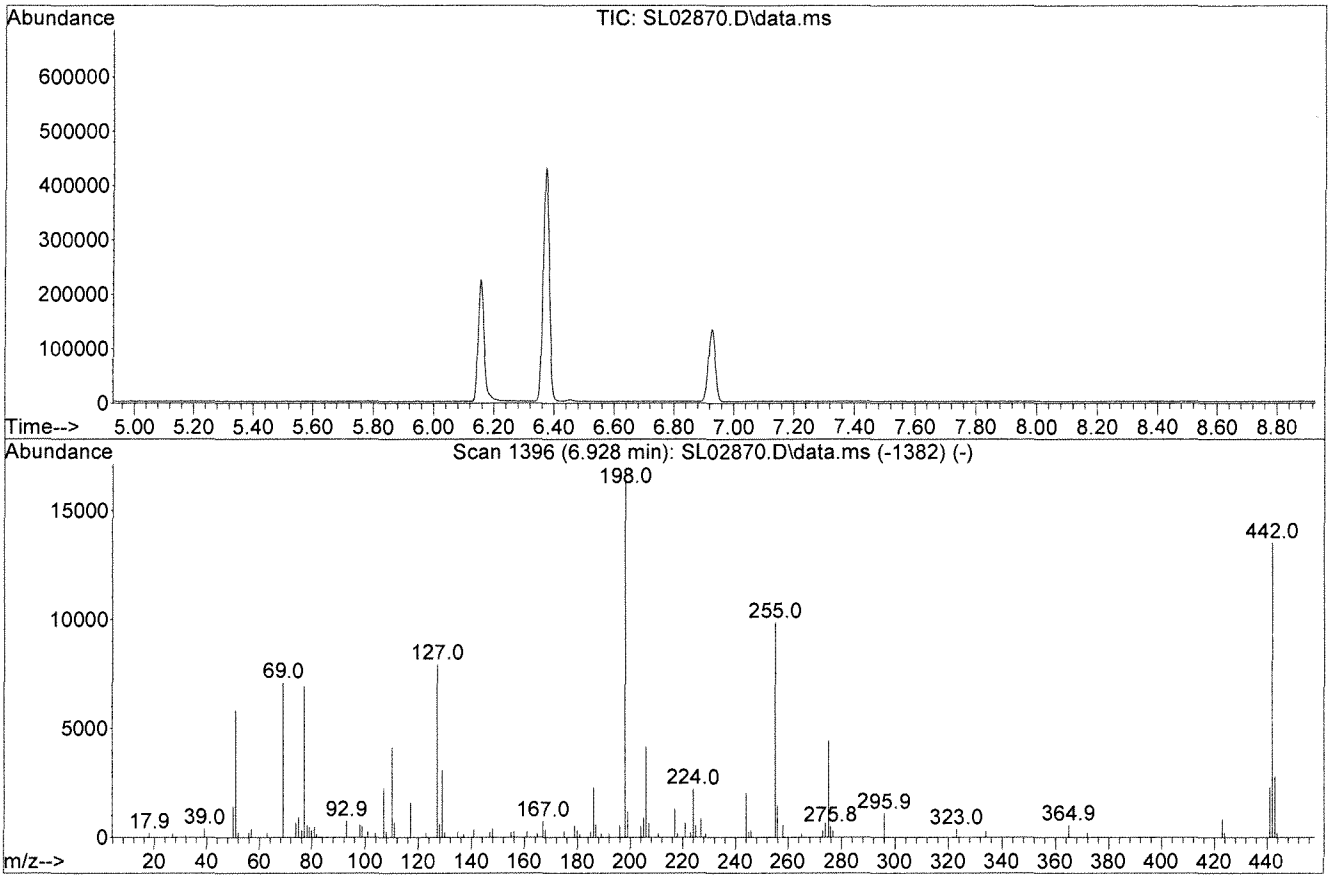
US EPA ARCHIVE DOCUMENT

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02870.D
Acq On : 27 Feb 2012 15:07
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Tailing PCP = 1.43
Benzidine 1.34
4,4'-DDT dev 28.94%

Integration File: rteint.p

Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Title : DFTPP Method with 8270D criteria: 8/20/10
Last Update : Thu Jan 26 14:45:44 2012



Spectrum Information: Scan 1396

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	35.7	5828	PASS
68	69	0.00	2	0.0	0	PASS
69	198	0.00	100	43.5	7105	PASS
70	69	0.00	2	0.0	0	PASS
127	198	10	80	48.7	7957	PASS
197	198	0.00	2	0.0	0	PASS
198	198	100	100	100.0	16336	PASS
199	198	5	9	7.4	1207	PASS
275	198	10	60	27.2	4436	PASS
365	198	1	100	3.5	567	PASS
441	442	0.01	24	17.1	2309	PASS
442	198	50	100	82.8	13520	PASS
443	442	15	24	20.7	2798	PASS

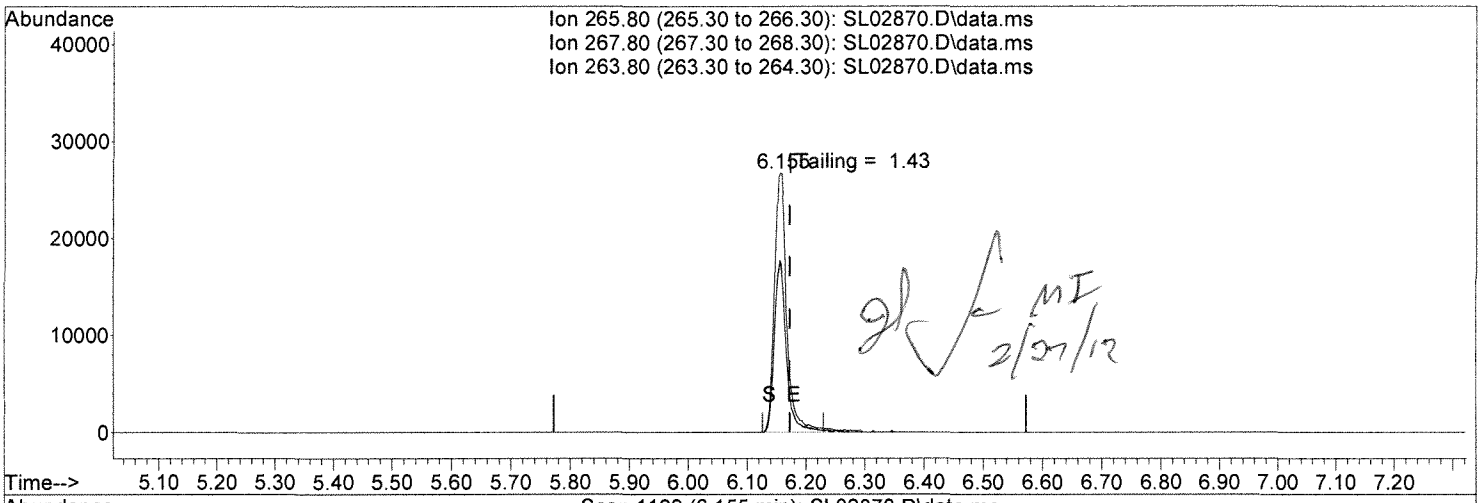
SERAS-017-DTM-011413_3

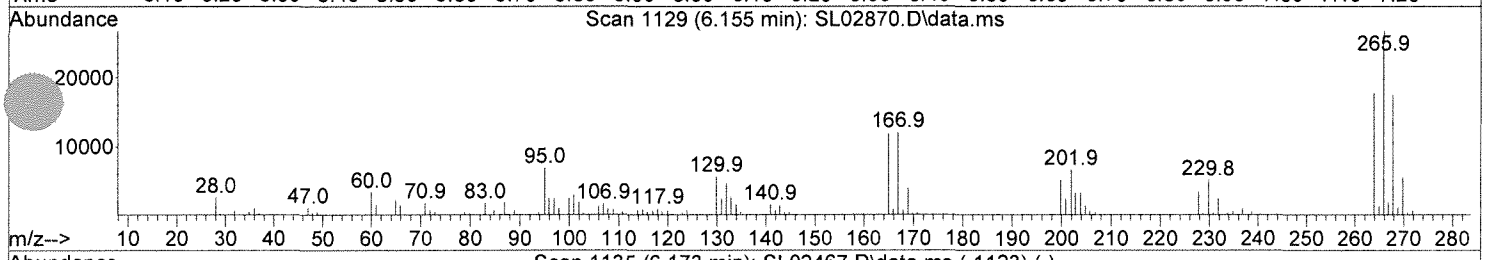
US EPA ARCHIVE DOCUMENT

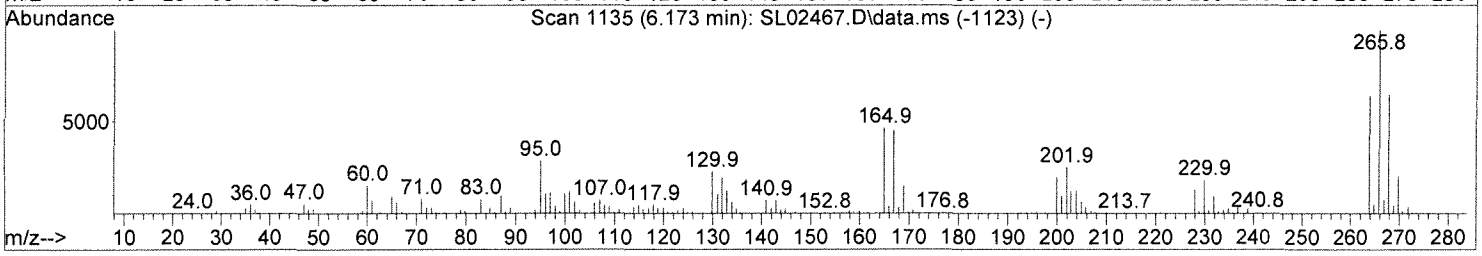
Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02870.D
Acq On : 27 Feb 2012 15:07
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 27 15:19:40 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration







TIC: SL02870.D\data.ms

(2) Pentachlorophenol

6.155min (-0.017) 48.43 ug/mL

response 39301

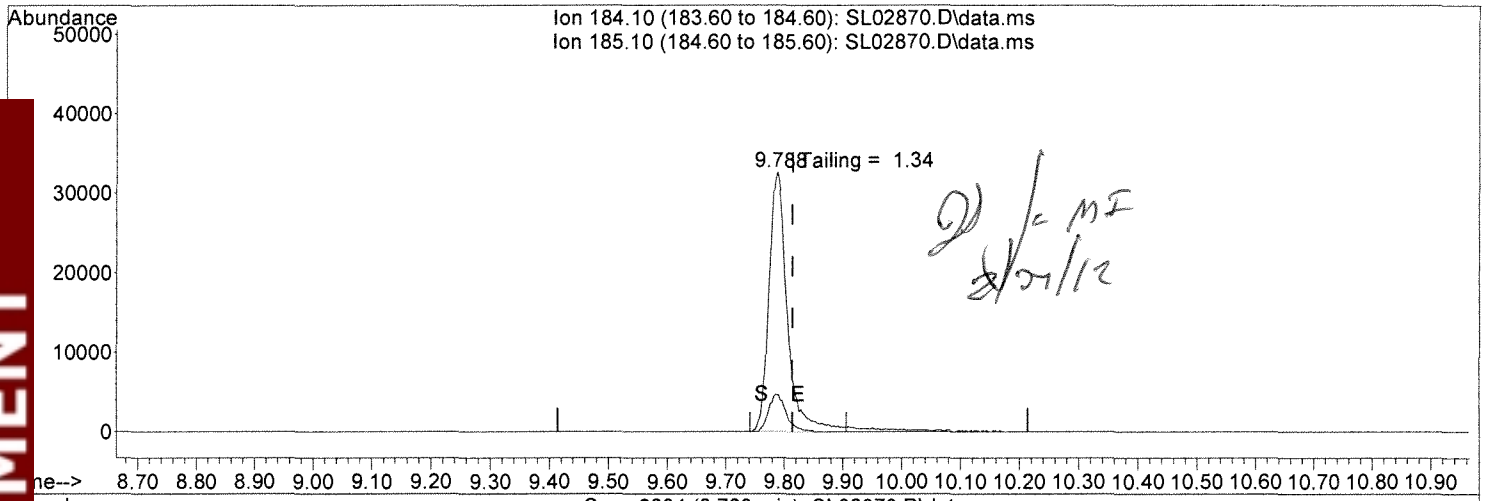
Ion	Exp%	Act%
265.80	100	100
267.80	64.50	62.45
263.80	63.60	64.03
0.00	0.00	0.00

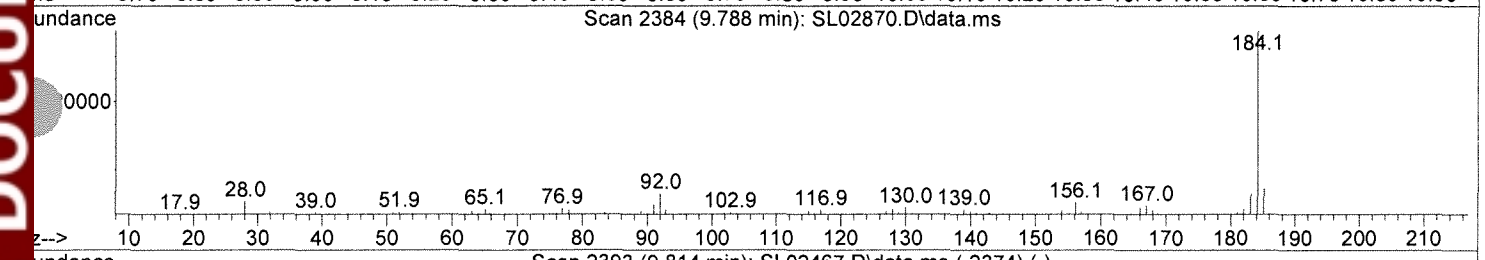
SERAS-017-DTM-011413_3

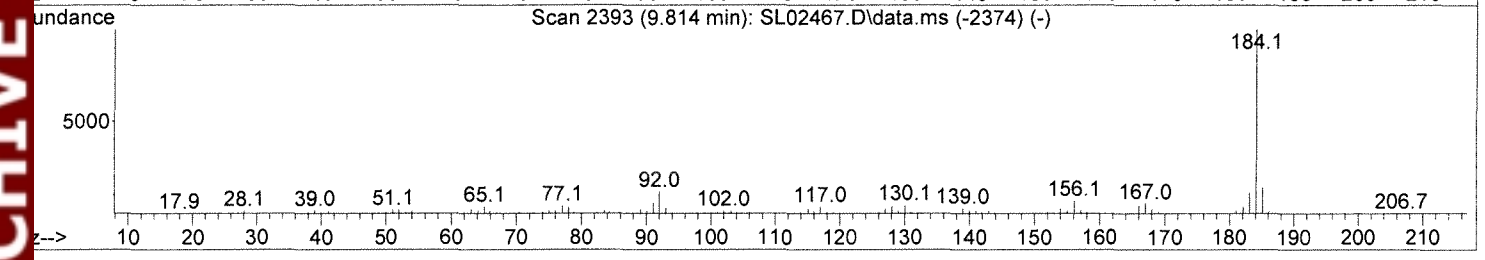
Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02870.D
Acq On : 27 Feb 2012 15:07
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 27 15:19:40 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration







TIC: SL02870.D\data.ms

(5) Benzidine

9.788min (-0.026) 23.34 ug/mL

response 71434

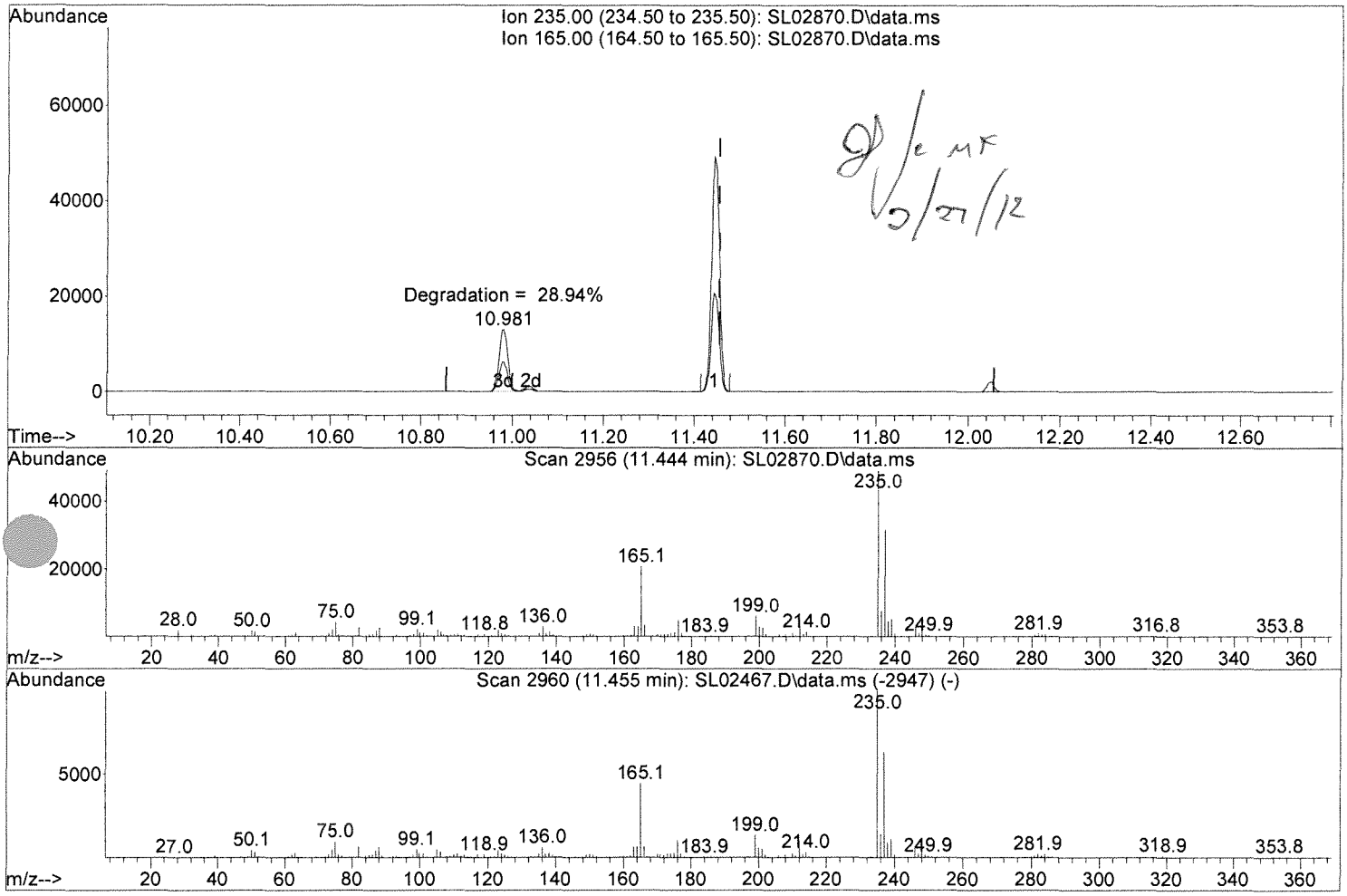
Ion	Exp%	Act%
184.10	100	100
185.10	14.60	13.95
0.00	0.00	0.00
0.00	0.00	0.00

US EPA ARCHIVE DOCUMENT

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02870.D
Acq On : 27 Feb 2012 15:07
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Feb 27 15:19:40 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



TIC: SL02870.D\data.ms

(6) DDT

11.444min (-0.012) 43.92 ug/mL

response 59812

Ion	Exp%	Act%
235.00	100	100
165.00	51.80	42.57
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

1000 ppm DRO CCV

√ on IS7 Surr

US EPA ARCHIVE DOCUMENT

TPH Daily Calibration Check Results
#2 Diesel TPH Calibration Range - Using draft SERAS GC/MS Method 1841

Calibration Check Date	ICAL Date	Project	Wa#	GC/MS Datafile	TPH Response	d30-IS Area	d50-IS Area	d74-IS Area	Cal Check RF	Calibration Average RF	% Diff.
05/17/11	05/16/11	WSoil MDL	0-011	SL01889	7932426	132429	130508	51192	0.75756	0.72959	3.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01942	7860351	128757	120362	11883	0.90348	0.72959	23.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01945	6544592	104943	128978	88104	0.60970	0.72959	16.43
07/08/11	05/16/11	CUC-Power Plant	0-135	SL01970	6848120	109628	127366	77204	0.65387	0.72959	10.38
07/11/11	05/16/11	CUC-Power Plant	0-135	SL01974	7184921	121573	136941	70557	0.65502	0.72959	10.22
07/12/11	05/16/11	CUC-Power Plant	0-135	SL01989	7699927	130026	148583	96240	0.61624	0.72959	15.54
07/27/11	05/16/11	CUC-Power Plant	0-135	SL02012	5526770	90684	107174	65457	0.62968	0.72959	13.69
08/01/11	05/16/11	CUC-Power Plant	0-135	SL02036	7282274	117359	139028	90753	0.62934	0.72959	13.74
08/08/11	05/16/11	Move-Maintenance	0-011	SL02067	20772937	342644	403655	197034	0.66062	0.72959	9.45
08/08/11	05/16/11	Move-Maintenance	0-011	SL02069	9013310	151400	176386	86475	0.65273	0.72959	10.54
08/10/11	05/16/11	Move-Maintenance	0-011	SL02071	8539256	140844	161739	77290	0.67438	0.72959	7.57
08/11/11	05/16/11	TPH Soil MDL	0-011	SL02079	8559600	142237	165518	79165	0.66367	0.72959	9.03
08/12/11	05/16/11	TPH Water MDL	0-011	SL02093	8541528	140220	170089	76028	0.66327	0.72959	9.09
08/23/11	05/16/11	CUC-Power Plant	0-135	SL02133	8279388	130044	151992	63182	0.71949	0.72959	1.38
08/24/11	05/16/11	CUC-Power Plant	0-135	SL02166	6757179	110307	122438	62687	0.68617	0.72959	5.95
09/21/11	05/16/11	CUC-Power Plant	0-135	SL02202	11190934	169011	205927	119691	0.67875	0.72959	6.97
09/22/11	05/16/11	CUC-Power Plant	0-135	SL02213	8986654	146134	179078	114918	0.61255	0.72959	16.04
02/02/12	05/16/11	Enbridge Oil	0-017	SL02671	10771363	175782	209958	153567	0.59918	0.72959	17.87
02/15/12	05/16/11	Enbridge Oil	0-017	SL02827	11689228	179024	220786	162003	0.62419	0.72959	14.45
02/24/12	05/16/11	Enbridge Oil	0-017	SL02857	11425492	166909	205857	151555	0.65373	0.72959	10.40
02/27/12	05/16/11	Enbridge Oil	0-017	SL02872	10457816	165200	202882	161132	0.59283	0.72959	18.74

To check IS areas + Surr

US EPA ARCHIVE DOCUMENT

Evaluate Continuing Calibration Report

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02871.D
Acq On : 27 Feb 2012 15:31
Operator : Syslo
Sample : 1K DRO/TPH CCV
Misc : RVB0003 Exp. 8/12/12
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 27 16:21:23 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Fri Feb 24 16:05:34 2012
Response via : Initial Calibration

Min. RRF : 0.010 Min. Rel. Area : 50% Max. R.T. Dev 0.50min
Max. RRF Dev : 30% Max. Rel. Area : 200%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	d10-Phenanthrene	1.000	1.000	0.0	103	-0.02
2	S	d10-Anthracene {S}	1.063	1.079	-1.5	103	-0.02
3	S	d14-o-Terphenyl {S}	0.467	0.525	-12.4	109	0.00
4	S	5a-Androstane {S}	0.057	0.070	-22.8	116	0.00
5	I	d30-Tetradecane	1.000	1.000	0.0	105	-0.02
6	I	d50-Tetracosane	1.000	1.000	0.0	111	0.00
7	S	d62-Triacontane {S}	0.695	1.024	~~-47.3#~~	137	-0.02
8	I	d74-Hexatriacontane	1.000	1.000	0.0	204#	-0.05

(#) = Out of Range SPCC's out = 0 CCC's out = 0

DROTPH051611.M Thu Jan 10 10:40:44 2013 SLICK2

~~18.74~~ 1/10/13

* uses linear regression
%D = 18.74

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02871.D
Acq On : 27 Feb 2012 15:31
Operator : Syslo
Sample : 1K DRO/TPH CCV
Misc : RVB0003 Exp. 8/12/12
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 27 16:21:23 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Fri Feb 24 16:05:34 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	496730	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	165200	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	202882	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	161132	10.00	ug/mL	-0.02
System Monitoring Compounds						
2) d10-Anthracene {S}	18.821	188	535829	10.14	ug/mL	0.00
Spiked Amount 20.000			Recovery =	50.70%		
3) d14-o-Terphenyl {S}	19.733	244	260666	11.23	ug/mL	0.00
Spiked Amount 20.000			Recovery =	56.15%		
4) 5a-Androstane {S}	21.149	260	34696	12.28	ug/mL	0.00
Spiked Amount 20.000			Recovery =	61.40%		
7) d62-Triacontane {S}	28.156	66	207784	11.96	ug/mL	0.00
Spiked Amount 20.000			Recovery =	59.80%		
Target Compounds						Qvalue

#) = qualifier out of range (m) = manual integration (+) = signals summed

*2nds Linear regression = %Δ = 19.6%.
Ran to quant still.
What's wrong.

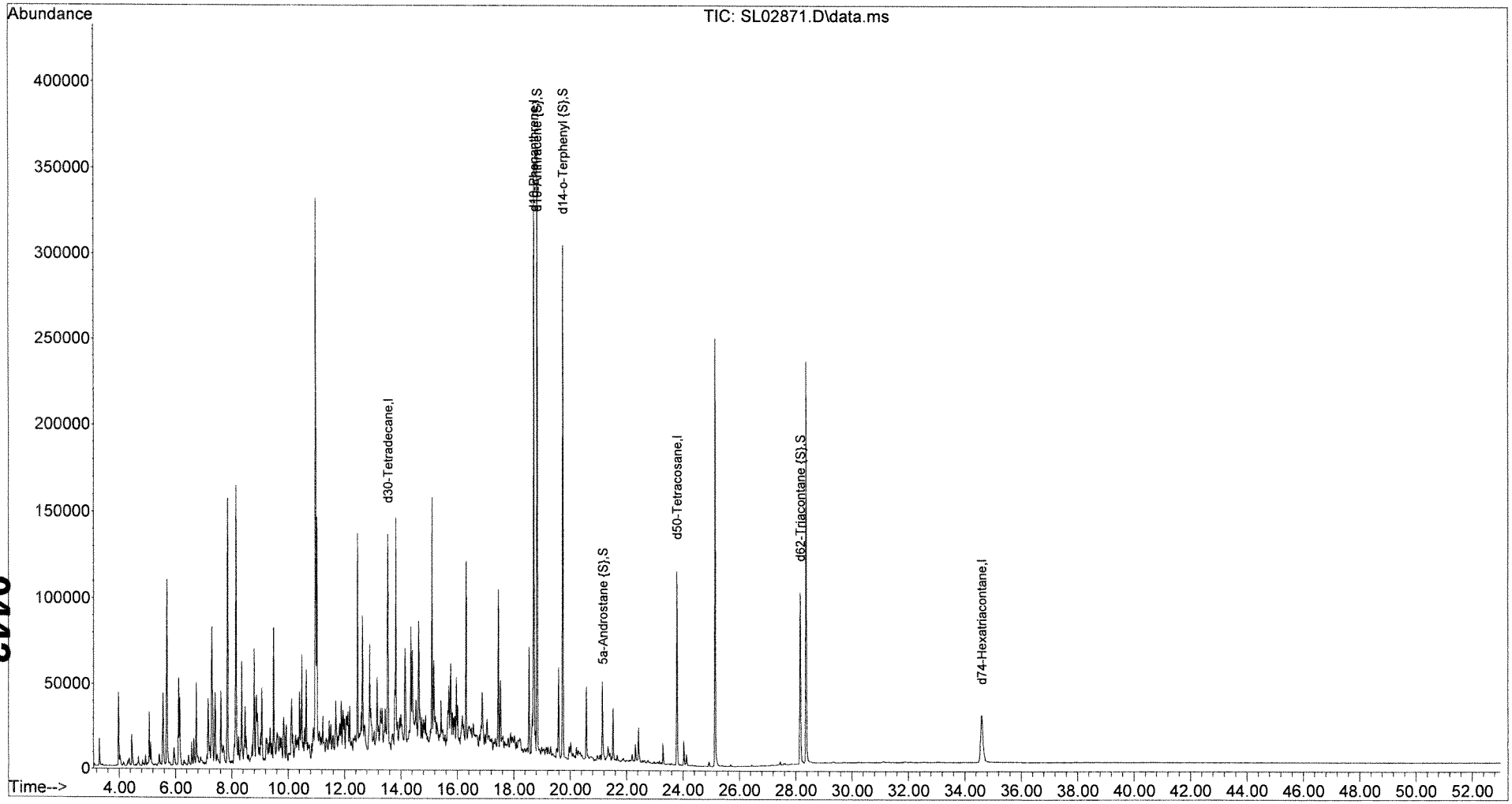
%D for TPH = 58.74

SERAS-017-DTM-011413_3

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02871.D
Acq On : 27 Feb 2012 15:31
Operator : Syslo
Sample : 1K DRO/TPH CCV
Misc : RVB0003 Exp. 8/12/12
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Feb 27 16:21:23 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Fri Feb 24 16:05:34 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0442

Area Percent Report

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02871.D
Acq On : 27 Feb 2012 15:31
Operator : Syslo
Sample : 1K DRO/TPH CCV
Misc : RVB0003 Exp. 8/12/12
ALS Vial : 71 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 0.5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 5
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Baseline drop else tangent >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL02871.D\data.ms

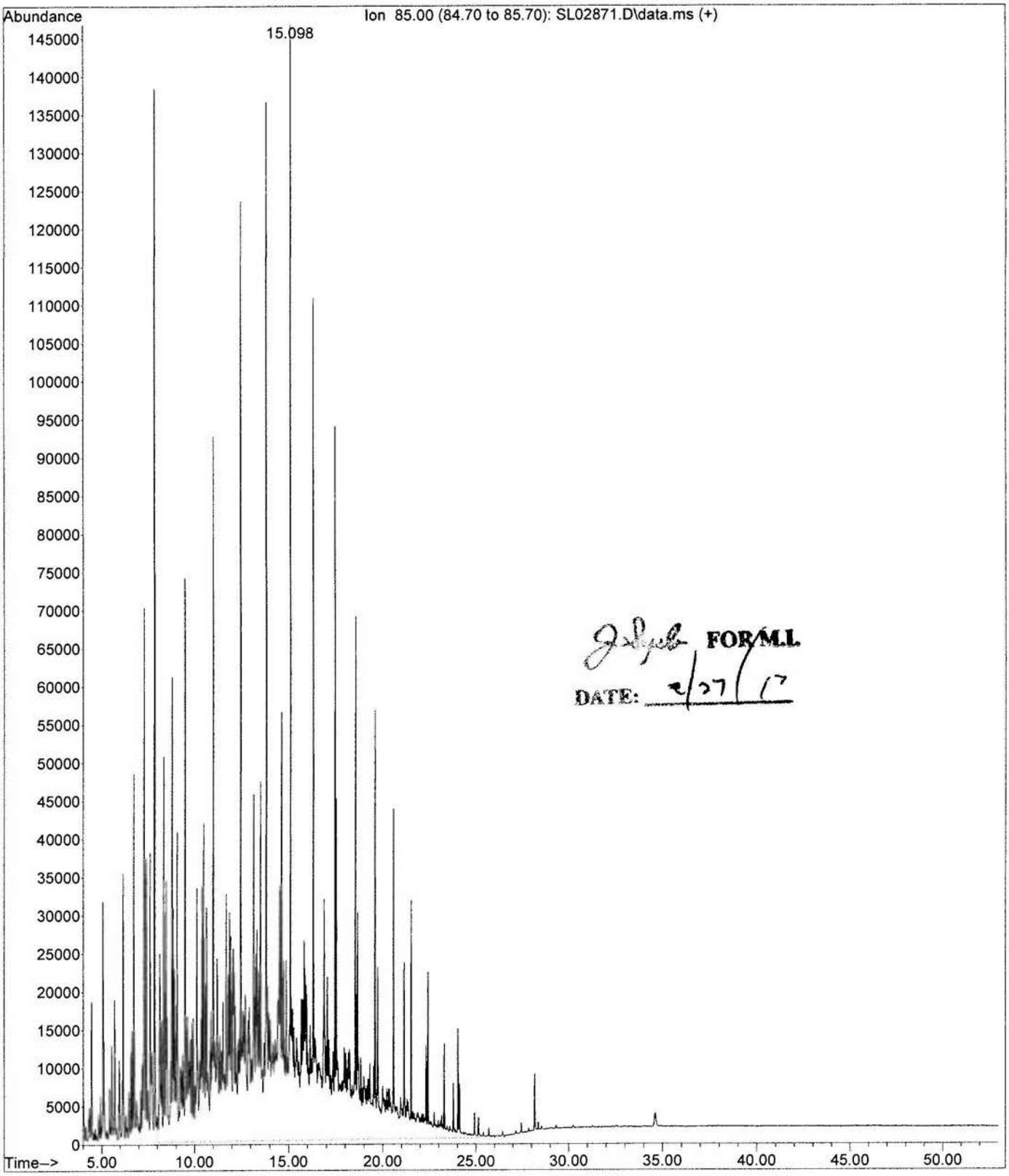
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	15.098	613	1504	2839	rM	146379	10457816	100.00%	100.000%

Sum of corrected areas: 10457816

DROTPH051611.M Mon Feb 27 16:57:26 2012 SLICK2

FOR M.L.
DATE: 2/27/12

File :C:\msdchem\1\DATA\022712\SL02871.D
Operator : Syslo
Acquired : 27 Feb 2012 15:31 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: 1K DRO/TPH CCV
Misc Info : RVB0003 Exp. 8/12/12
Vial Number: 71



FOR M.L.
DATE: 2/27/17

US EPA ARCHIVE DOCUMENT

10 ppm SAC/PAH Mix
For Rec + Mass Discrimination

US EPA ARCHIVE DOCUMENT

Table 1.0: Results of ASTM D-5739 Resolution and Mass Discrimination Performance Checks Applied to *draft* SERAS GC/MS Method 1803: Oil Fingerprinting, Characterization and TPH Analysis

Enbridge Oil: Fingerprinting 2012: WA# 0-017

Method: *draft* SERAS SOP 1803

		Resolution Check[1]			Mass Discrimination Checks							Resolution Check: Svslo/SERAS Benzo(b)/Benzo(k) fluoranthene ratio
Date Checked	GC/MS File	C17/pristane % Resolution	C18/phytane % Resolution	Acceptable % Resolution	Naphthalene area	Fluoranthene area	Benzo(g,h,i)peryiene area	Nap/Fluo Ratio area/area	B(ghi)/Fluo Ratio area/area	Acceptable Ratios Nap/Fluo	Acceptable Ratios B(ghi)/Fluo	
Enbridge Oil TPH/Fingerprinting: WA# 0-017												
2/23/2012	SL02855[3]	99.8%	99.9%	> 50% v/v	1100101	1358009	1167661	0.81	0.86	<= 2.0	>= 0.20	68.70%
2/24/2012	SL02855[2]	98.5%	99.7%	> 50% v/v	454731	543693	439965	0.84	0.81	<= 2.0	>= 0.20	68.43%
2/27/2012	SL02872[2]	98.5%	99.7%	> 50% v/v	507043	611080	485460	0.83	0.79	<= 2.0	>= 0.20	65.80%
3/1/2012	SL02883[2]	99.0%	99.4%	> 50% v/v	506865	655953	569653	0.77	0.87	<= 2.0	>= 0.20	63.55%
Lab Maintenance Samples: 0-011												
5/23/2011	SL01907	97.94	98.85	> 50% v/v	487005	429769	381358	1.13	0.89	<= 2.0	>= 0.20	62.83
5/24/2011	SL01920	99.04	99.87	> 50% v/v	545444	544337	421740	1.00	0.77	<= 2.0	>= 0.20	61.02
DRO-TPH Soil MDL Study August 11, 2011 on Slick2 - After moving instrument to new lab.												
8/11/2011	SL02081	99.05	99.85	> 50% v/v	504208	637067	494345	0.79	0.78	<= 2.0	>= 0.20	61.40
Imperial Oil fingerprints: November - December 2011 on Slick 2												
11/14/2011	SL02274[3]	98.92	99.85	> 50% v/v	891148	1110386	936738	0.80	0.84	<= 2.0	>= 0.20	63.93
12/1/2011	SL02332[3]	98.14	100.28	> 50% v/v	158336	197959	135116	0.80	0.68	<= 2.0	>= 0.20	59.12
12/2/2011	SL02339[3]	99.01	99.82	> 50% v/v	1666437	2018693	1619360	0.83	0.80	<= 2.0	>= 0.20	67.52
12/3/2011	SL02364[3]	99.06	99.88	> 50% v/v	1246967	1470915	1193650	0.85	0.81	<= 2.0	>= 0.20	68.84
12/5/2011	SL02367[3]	99.04	99.82	> 50% v/v	1281093	1527311	1134178	0.84	0.74	<= 2.0	>= 0.20	69.13
12/6/2011	SL02382[3]	98.87	99.87	> 50% v/v	1152427	1408258	1191425	0.82	0.85	<= 2.0	>= 0.20	67.25
CUC Oil fingerprints: December 2011 on Slick 2												
12/7/2011	SL02403[3]	99.93	99.73	> 50% v/v	1323413	1547091	1278611	0.86	0.83	<= 2.0	>= 0.20	68.49
12/9/2011	SL02419[3]	99.08	99.71	> 50% v/v	1202251	1425463	1171714	0.84	0.82	<= 2.0	>= 0.20	67.22
12/13/2011	SL02441[3]	98.92	99.76	> 50% v/v	1686396	2010249	1665781	0.84	0.83	<= 2.0	>= 0.20	66.62
12/14/2011	SL02456[3]	99.11	99.76	> 50% v/v	1180706	1397756	1127432	0.84	0.81	<= 2.0	>= 0.20	64.46

All resolution and mass discrimination checks are performed using a 10ug/mL PAH & SHC standard unless otherwise noted.

(1) The resolution check is determined using the Enviroquant ChromEval option in the Qedit mode.
(2) Resolution check performed using 10ppm SHC/PAH mixture in SIM mode.
(3) Resolution check performed using 25ppm SHC/PAH mixture in SIM mode.

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02872.D
Acq On : 27 Feb 2012 16:32
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 72 Sample Multiplier: 1

For Mass Discrimination + Res Check

Quant Time: Feb 27 17:32:19 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d4-1,4-Dichlorobenzene	8.144	152	140205	10.00	ug/mL	0.00
14) d8-Naphthalene	10.968	136	449498	10.00	ug/mL	0.00
23) d10-Phenanthrene	18.699	188	463945	10.00	ug/mL	0.00
41) d12-Chrysene	25.133	240	444109	10.00	ug/mL	0.00
50) d12-Perylene	28.365	264	416711	10.00	ug/mL	0.00
59) d30-Tetradecane	13.536	66	152140	10.00	ug/mL	0.00
73) d50-Tetracosane	23.783	66	185434	10.00	ug/mL	0.00
86) d74-Hexatriacontane	34.588	66	154527	10.00	ug/mL	-0.03
System Monitoring Compounds						
33) d10-Anthracene {S}	18.821	188	403770	8.66	ug/mL	0.00
Spiked Amount 20.000			Recovery =	43.30%		
35) d14-o-Terphenyl {S}	19.732	244	191308	8.77	ug/mL	0.00
Spiked Amount 20.000			Recovery =	43.85%		
40) 5a-Androstane {S}	21.137	260	25058	9.25	ug/mL	0.00
Spiked Amount 20.000			Recovery =	46.25%		
85) d62-Triacontane {S}	28.157	66	155555	12.01	ug/mL	0.00
Spiked Amount 20.000			Recovery =	60.05%		
Target Compounds						Qvalue
15) Naphthalene	11.011	128	507043	8.85	ug/mL	100
16) 2-Methylnaphthalene	12.641	142	323254	8.81	ug/mL	100
21) Acenaphthylene	14.795	152	521750	9.08	ug/mL	100
22) Acenaphthene	15.239	154	299401	8.80	ug/mL	100
24) Fluorene	16.462	166	357706	9.01	ug/mL	100
28) Dibenzothiophene	18.457	184	515833	8.59	ug/mL	100
32) Phenanthrene	18.751	178	537228	8.80	ug/mL	100
34) Anthracene	18.864	178	511629	8.94	ug/mL	100
36) 2-Methylanthracene	20.114	192	333546	9.14	ug/mL	100
42) Fluoranthene	21.649	202	611080	9.12	ug/mL	100
43) Pyrene	22.187	202	610896	9.07	ug/mL	100
46) Benzo(a)anthracene	25.099	228	535949	9.08	ug/mL	100
47) Chrysene	25.191	228	491718	8.81	ug/mL	100
51) Benzo(b)fluoranthene	27.565	252	539810	9.50	ug/mL	100
52) Benzo(k)fluoranthene	27.615	252	492207	8.49	ug/mL	100
53) Benzo(e)pyrene	28.132	252	531779	8.74	ug/mL	100
54) Benzo(a)pyrene	28.240	252	497338	9.37	ug/mL	100
55) Perylene	28.415	252	480014	8.91	ug/mL	100
56) Indeno(1,2,3-cd)pyrene	31.064	276	576808	9.51	ug/mL	100
57) Dibenzo(a,h)anthracene	31.097	278	457181	9.60	ug/mL	100
58) Benzo(g,h,i)perylene	31.847	276	485460	9.28	ug/mL	100
60) Octane C8	4.455	85	79054	10.25	ug/mL	100
61) Nonane C9	6.150	85	56227	10.13	ug/mL	100
62) Decane C10	7.853	85	47790	10.18	ug/mL	100
63) Undecane C11	9.483	85	53955	10.37	ug/mL	100
64) Dodecane C12	11.019	85	66863	10.18	ug/mL	100
65) Tridecane C13	12.459	85	75480	10.43	ug/mL	100
66) Tetradecane C14	13.820	85	84265	10.48	ug/mL	100
67) Pentadecane C15	15.094	85	91013	10.67	ug/mL	100
68) Hexadecane C16	16.309	85	95077	10.69	ug/mL	100
69) Heptacosane C17	17.451	85	96134	10.86	ug/mL	100
70) Pristane	17.531	85	80599	10.66	ug/mL	100
71) Octacosane C18	18.543	85	105633	11.07	ug/mL	100

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02872.D
Acq On : 27 Feb 2012 16:32
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 72 Sample Multiplier: 1

Quant Time: Feb 27 17:32:19 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
72) Phytane	18.656	85	108037	10.89	ug/mL	100
74) Nonadecane C19	19.576	85	108562	10.29	ug/mL	100
75) Eicosane C20	20.565	85	111425	10.54	ug/mL	100
76) Heneicosane C21	21.510	85	112975	10.64	ug/mL	100
77) Docosane C22	22.421	85	114491	10.76	ug/mL	100
78) Tricosane C23	23.289	85	116358	11.07	ug/mL	100
79) Tetracosane C24	24.122	85	118897	11.03	ug/mL	100
80) Pentacosane C25	24.916	85	118365	11.53	ug/mL	100
81) Hexacosane C26	25.691	85	118493	11.82	ug/mL	100
82) Heptacosane C27	26.432	85	117587	12.24	ug/mL	100
83) Octacosane C28	27.149	85	118086	12.48	ug/mL	100
84) Nonacosane C29	27.840	85	119022	13.07	ug/mL	100
87) Triacontane C30	28.531	85	121785	8.11	ug/mL	100
88) Hentriacontane C31	29.323	85	117472	8.55	ug/mL	100
89) Dotriacontane C32	30.231	85	112371	9.02	ug/mL	100
90) Tritriacontane C33	31.289	85	109459	9.61	ug/mL	100
91) Tetratriacontane C34	32.538	85	107070	10.00	ug/mL	100
92) Pentatriacontane C35	34.021	85	100781	10.59	ug/mL	100
93) Hexatriacontane C36	35.796	85	104075	10.68	ug/mL	100
94) Heptatriacontane C37	37.912	85	95525	11.52	ug/mL	100
95) Octatriacontane C38	40.461	85	94077	12.17	ug/mL	100
97) Tetracontane C40	47.184	85	85454	14.29	ug/mL	100

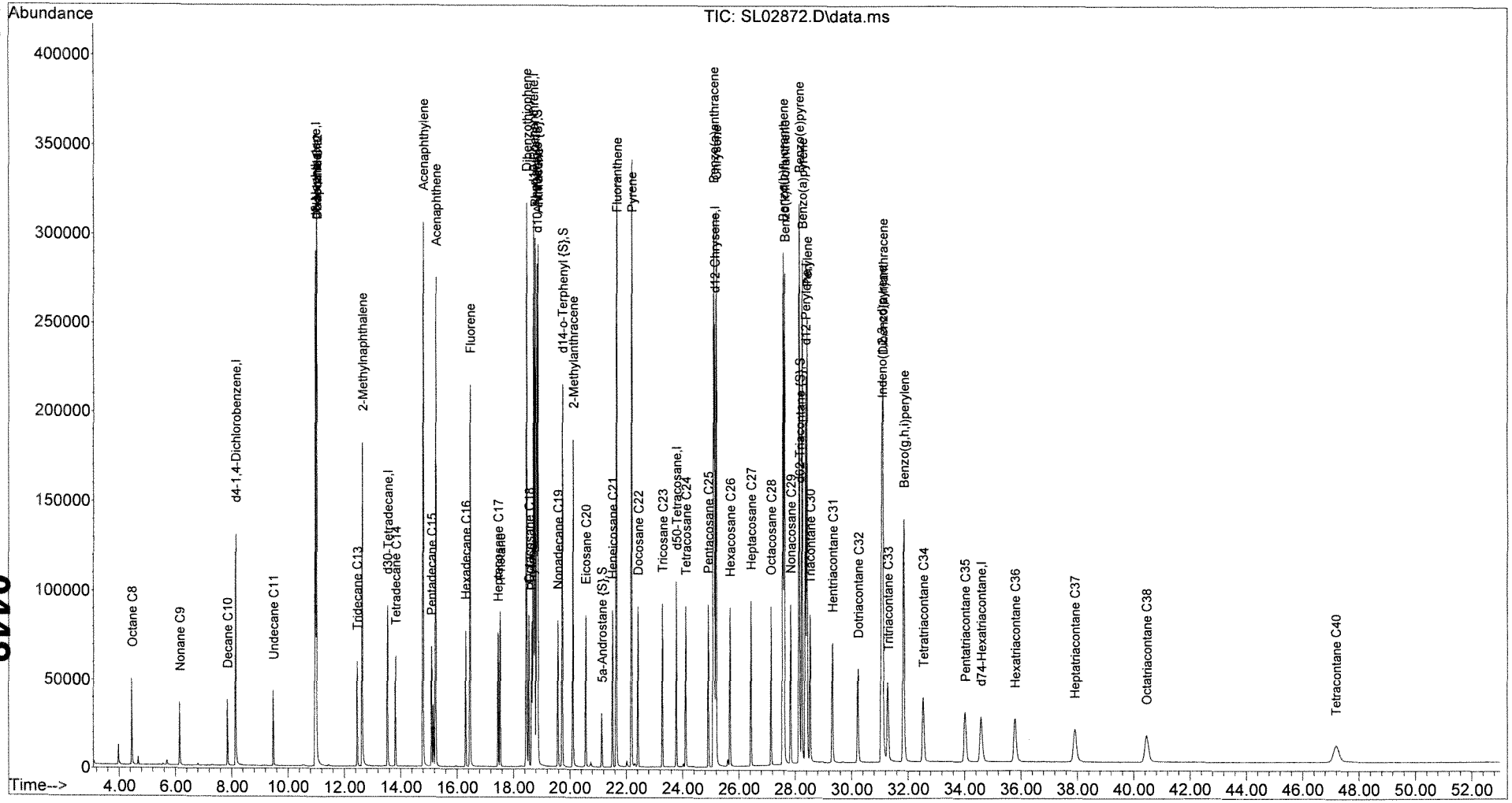
(#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02872.D
Acq On : 27 Feb 2012 16:32
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 72 Sample Multiplier: 1

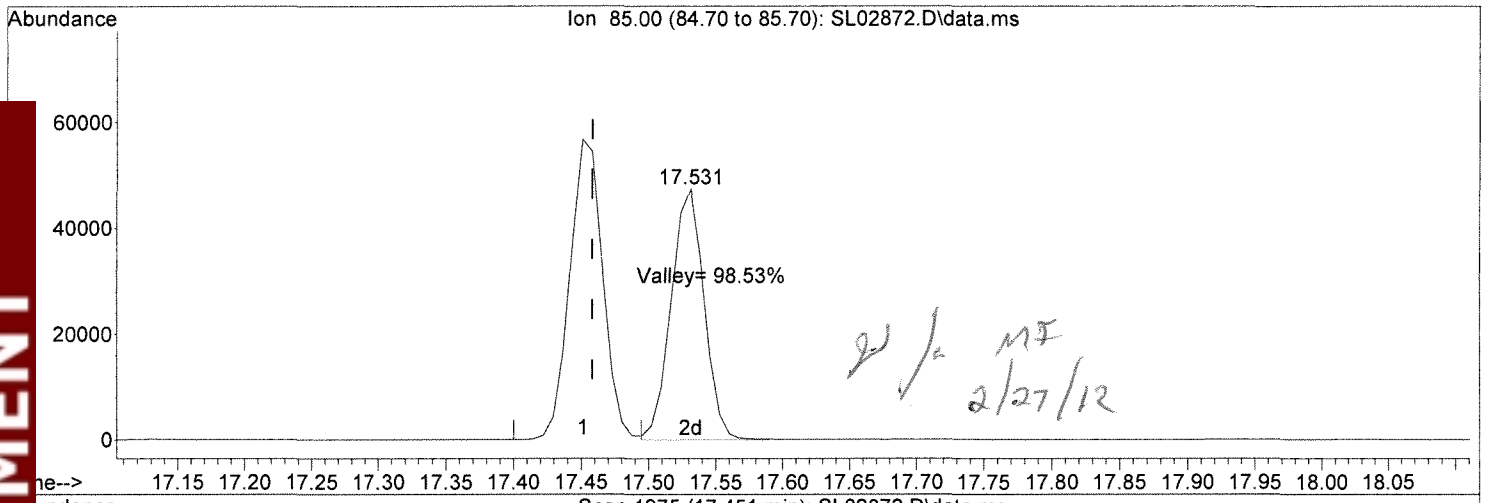
Quant Time: Feb 27 17:32:19 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration

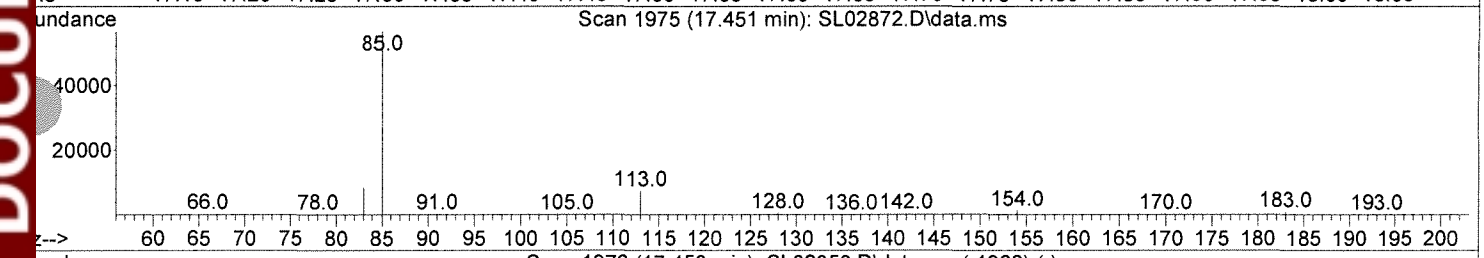


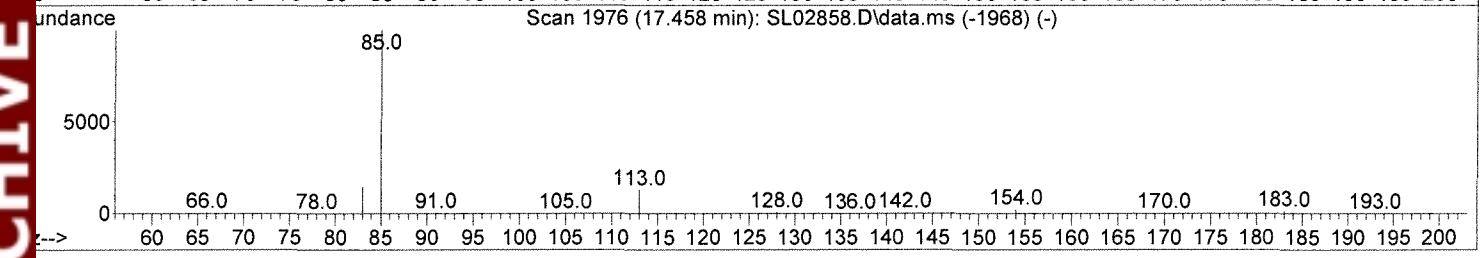
Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02872.D
Acq On : 27 Feb 2012 16:32
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 72 Sample Multiplier: 1

Quant Time: Feb 27 17:22:40 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration







TIC: SL02872.D\data.ms

(69) Heptacosane C17

17.451min (-0.007) 10.86 ug/mL

response 96134

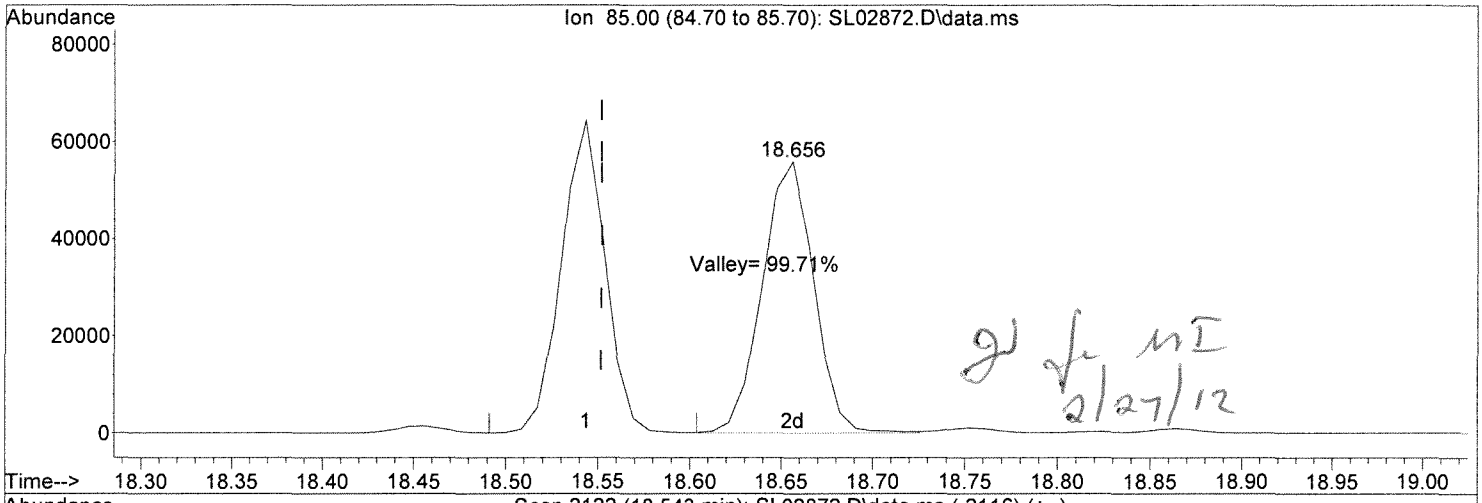
Ion	Exp%	Act%
85.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

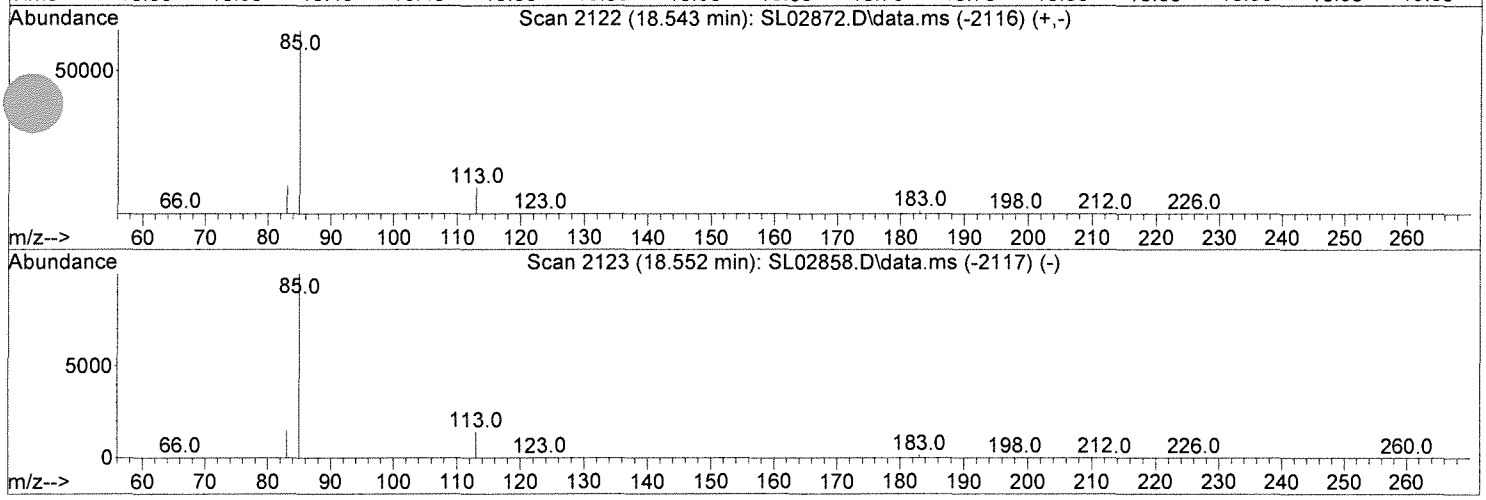
US EPA ARCHIVE DOCUMENT
SERAS-017-DTM-011413_3

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02872.D
Acq On : 27 Feb 2012 16:32
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 72 Sample Multiplier: 1

Quant Time: Feb 27 17:22:40 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration





TIC: SL02872.D\data.ms

(71) Octacosane C18

18.543min (-0.009) 11.07 ug/mL

response 105633

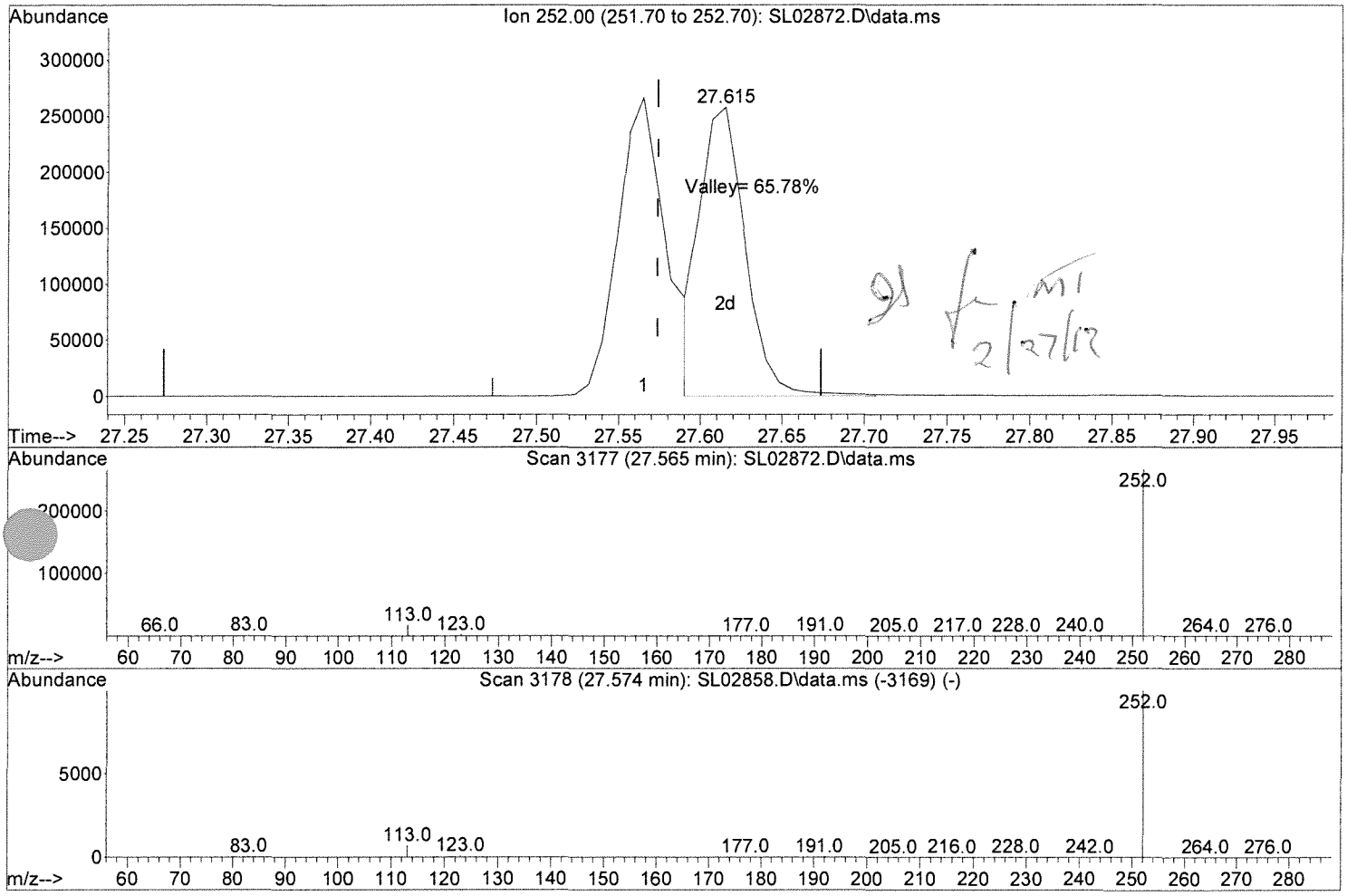
Ion	Exp%	Act%
85.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02872.D
Acq On : 27 Feb 2012 16:32
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 72 Sample Multiplier: 1

Quant Time: Feb 27 17:22:40 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration



TIC: SL02872.D\data.ms

(51) Benzo(b)fluoranthene

27.565min (-0.009) 9.50 ug/mL

response 539810

Ion	Exp%	Act%
252.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

CG-117 re/sesma oil

- ASTM D-5739 QC
  - re/sesma oil and
  - bracket/batch duplicate

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02873.D
Acq On : 27 Feb 2012 17:34
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003
ALS Vial : 73 Sample Multiplier: 1

Quant Time: Mar 01 13:13:20 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:23 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.708	188	566477	10.00	ug/mL	0.00
5) d30-Tetradecane	13.536	66	177045	10.00	ug/mL	0.00
6) d50-Tetracosane	23.783	66	223004	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.605	66	204402	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	0.000	188	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
3) d14-o-Terphenyl {S}	0.000	244	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
4) 5a-Androstane {S}	0.000	260	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
7) d62-Triacontane {S}	0.000	66	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
Target Compounds						Qvalue

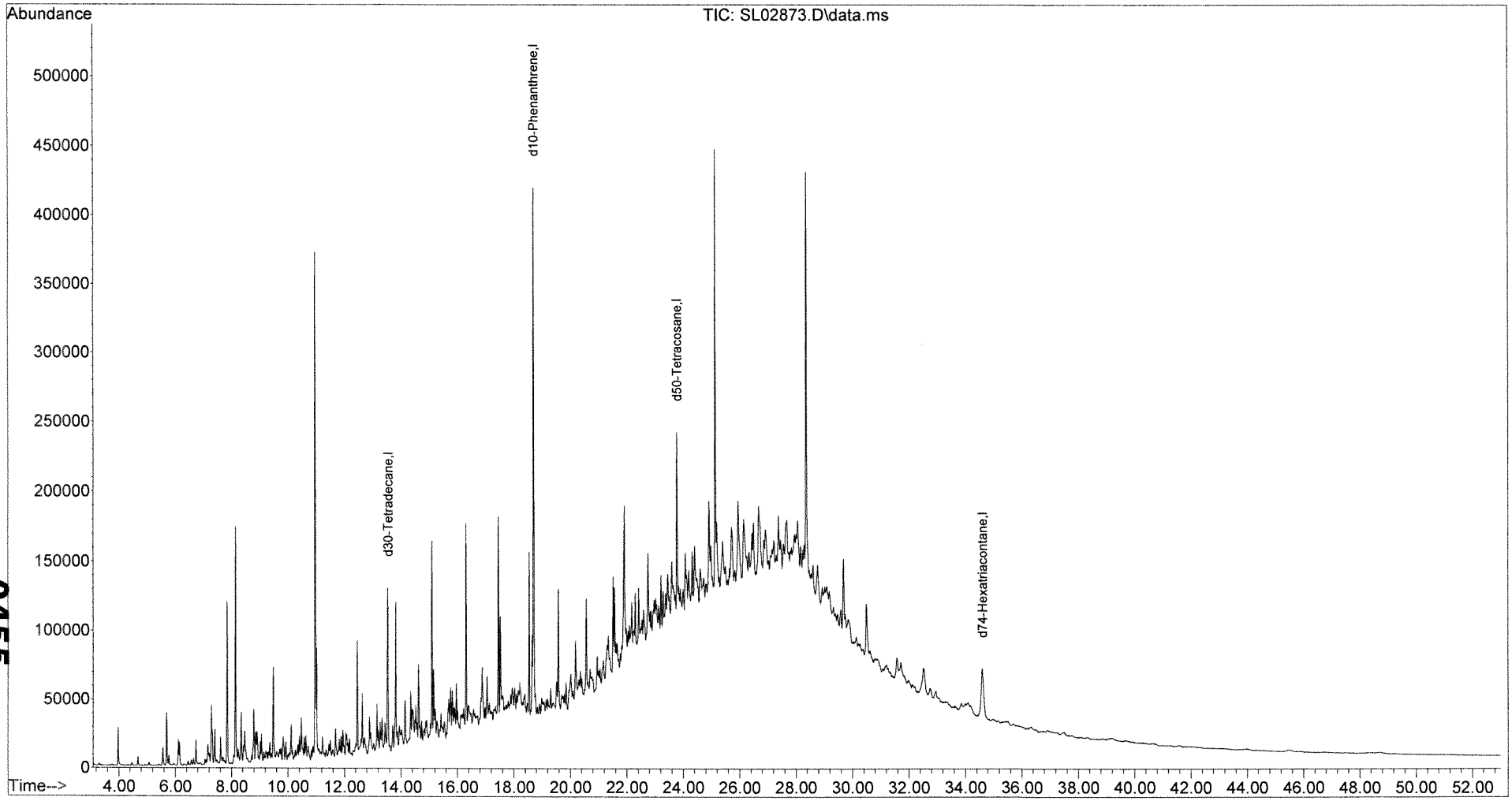
#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02873.D
Acq On : 27 Feb 2012 17:34
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003
ALS Vial : 73 Sample Multiplier: 1

Quant Time: Mar 01 13:13:20 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:23 2012
Response via : Initial Calibration



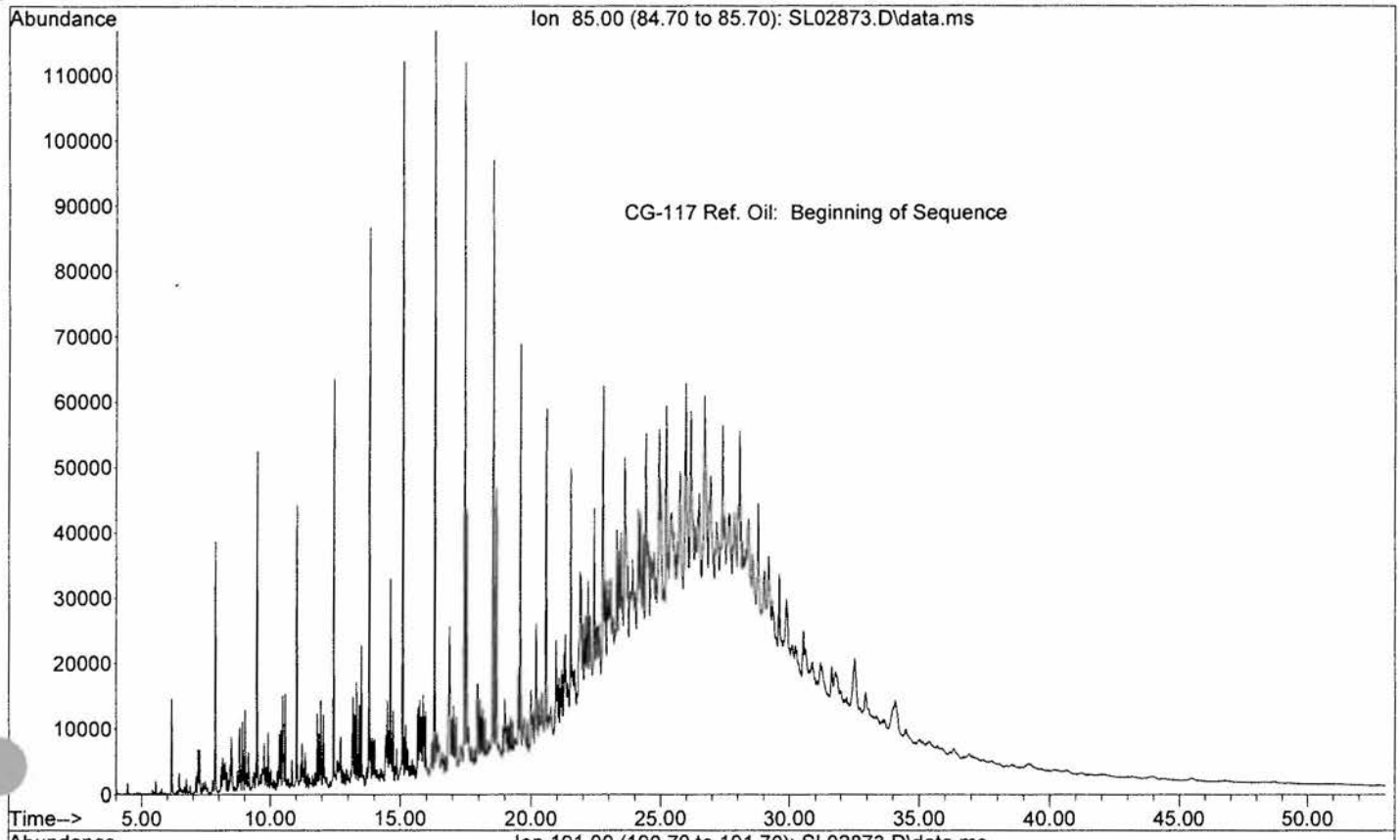
0455

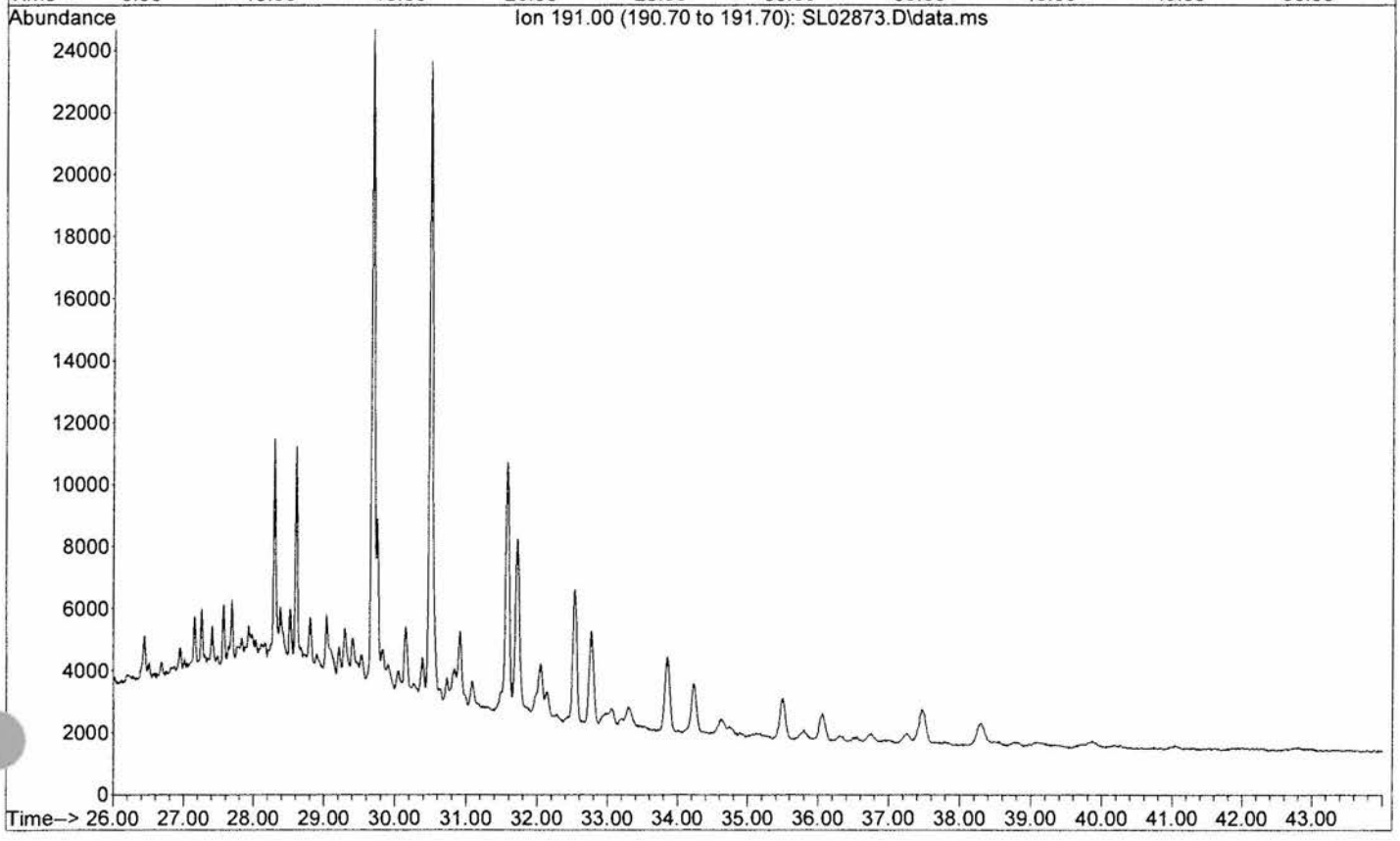
```
! File  : FIREF117.MAC
! J. Syslo MACRO for reference fingerprint using CG117: 11/22/09
!  Hopanes are normalized for easy viewing!
! Draws chromatogram in window 2 and window 1 . .  you have to print "TIC &
Spectrum"
!  I have a lot of old CG-117's like that - better reference"  JS

NAME FIREF117
format separated,,,0
EIC 4:55,85
exchange r0,x
EIC 26:44,191
exchange r0,y
draw 2,y
draw 1,x
REMOVE FIREF117
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02873.D
Operator : Syslo
Acquired : 27 Feb 2012 17:34 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003
Vial Number: 73





Waste Dilution Blank
02/27/12

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02874.D
Acq On : 27 Feb 2012 18:44
Operator : Syslo
Sample : BLK022712WD
Misc : 10mL RUC0022 + 0.1mL RUK0077
ALS Vial : 1 Sample Multiplier: 1

Quant Time: Mar 01 13:06:24 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	300975	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	100642	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	116776	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.587	66	95670	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.821	188	561282	17.54	ug/mL	0.00
Spiked Amount 20.000			Recovery = 87.70%			
3) d14-o-Terphenyl {S}	19.733	244	279001	19.84	ug/mL	0.00
Spiked Amount 20.000			Recovery = 99.20%			
4) 5a-Androstane {S}	21.140	260	36869	21.54	ug/mL	0.00
Spiked Amount 20.000			Recovery = 107.70%			
7) d62-Triacontane {S}	28.156	66	223136	21.55	ug/mL	0.00
Spiked Amount 20.000			Recovery = 107.75%			
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

Blank = ND

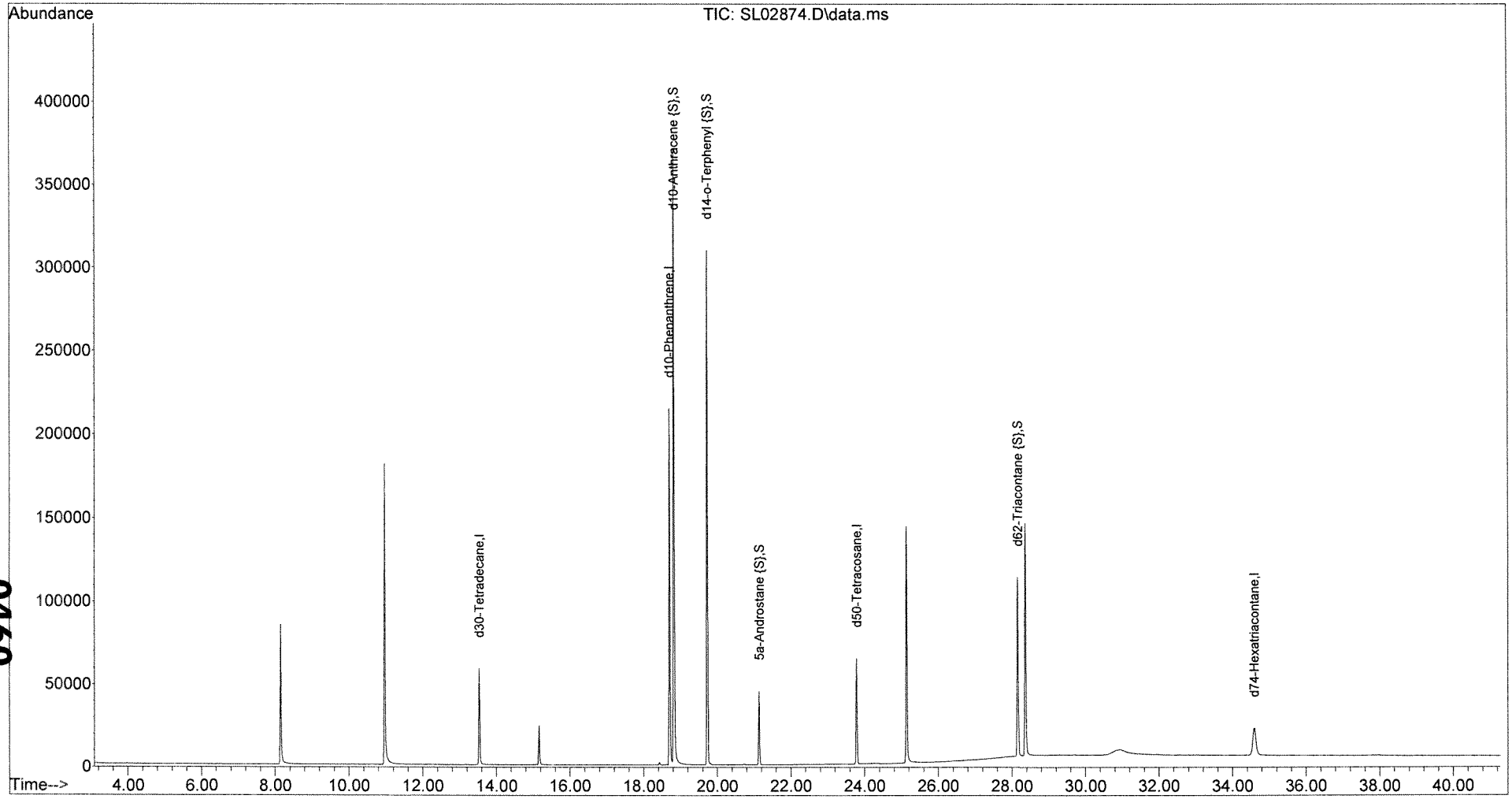
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02874.D
Acq On : 27 Feb 2012 18:44
Operator : Syslo
Sample : BLK022712WD
Misc : 10mL RUC0022 + 0.1mL RUK0077
ALS Vial : 1 Sample Multiplier: 1

Quant Time: Mar 01 13:06:24 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0460

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

US EPA ARCHIVE DOCUMENT

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM.* Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

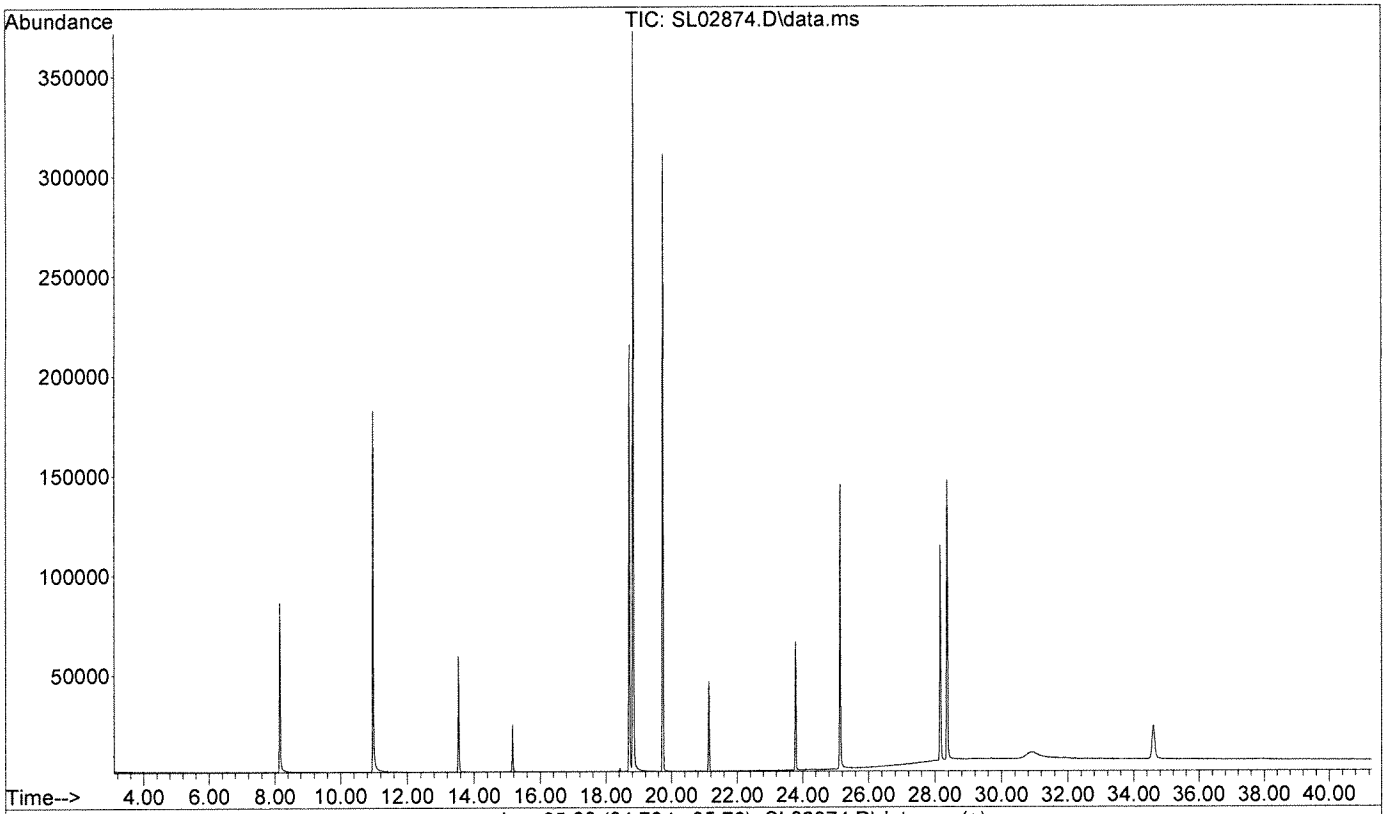
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

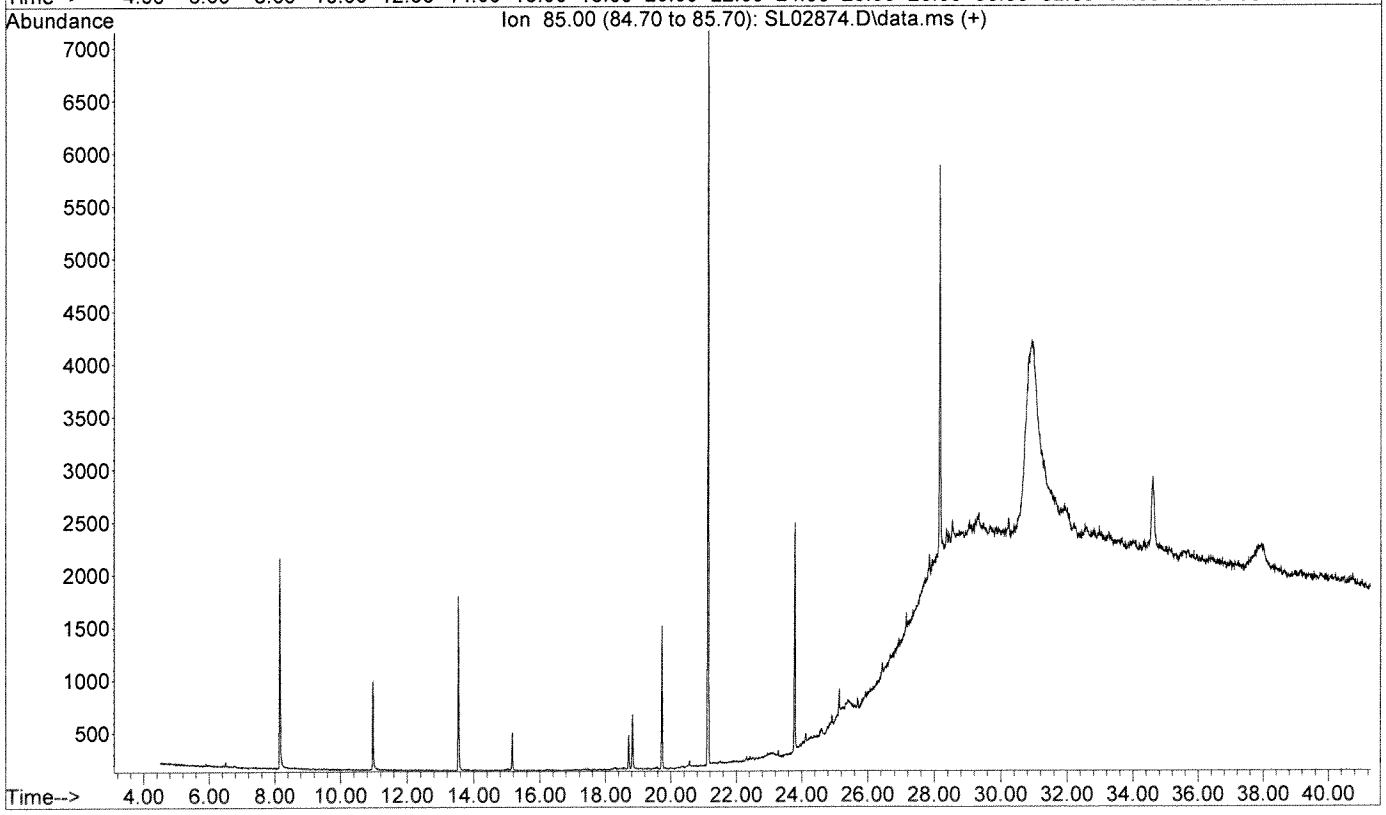
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



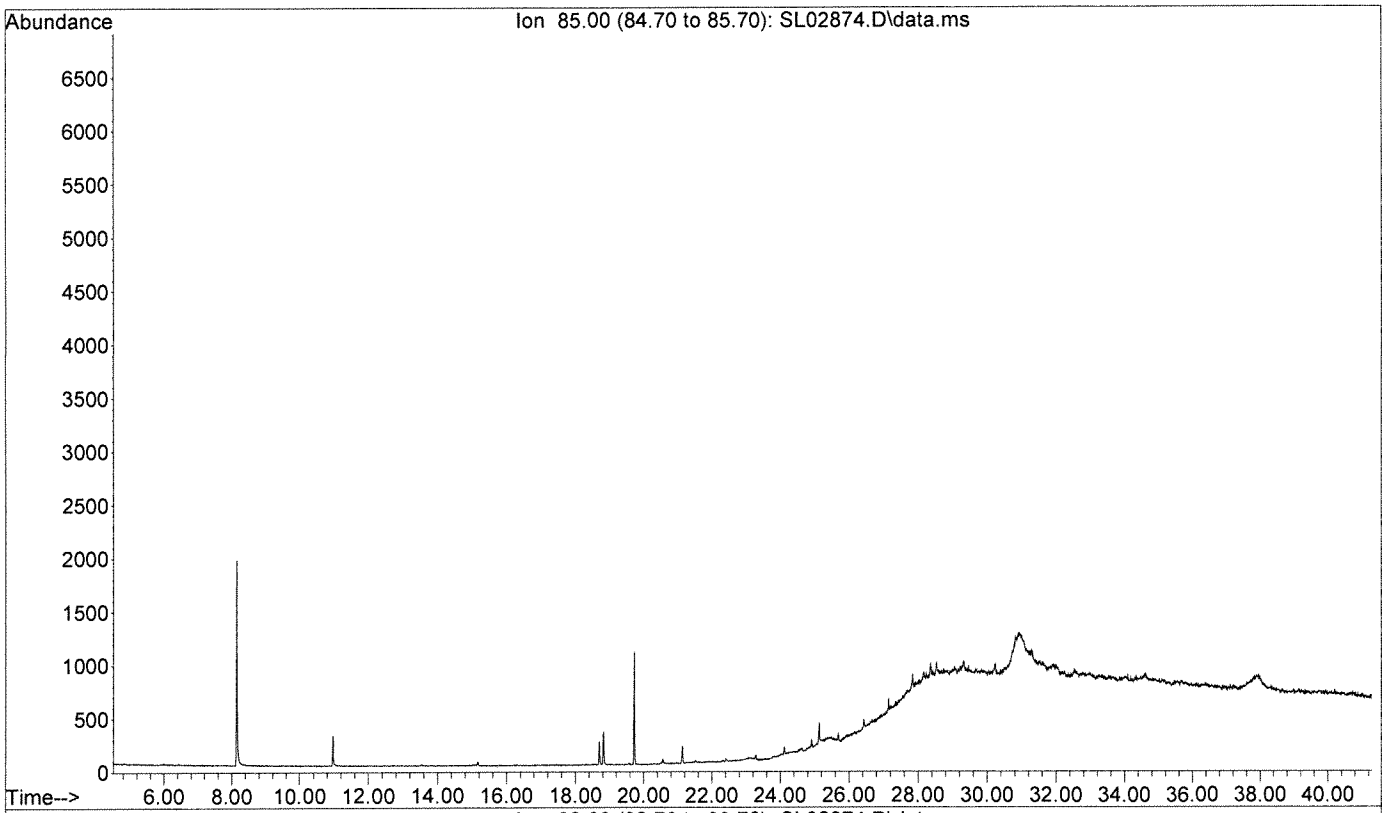


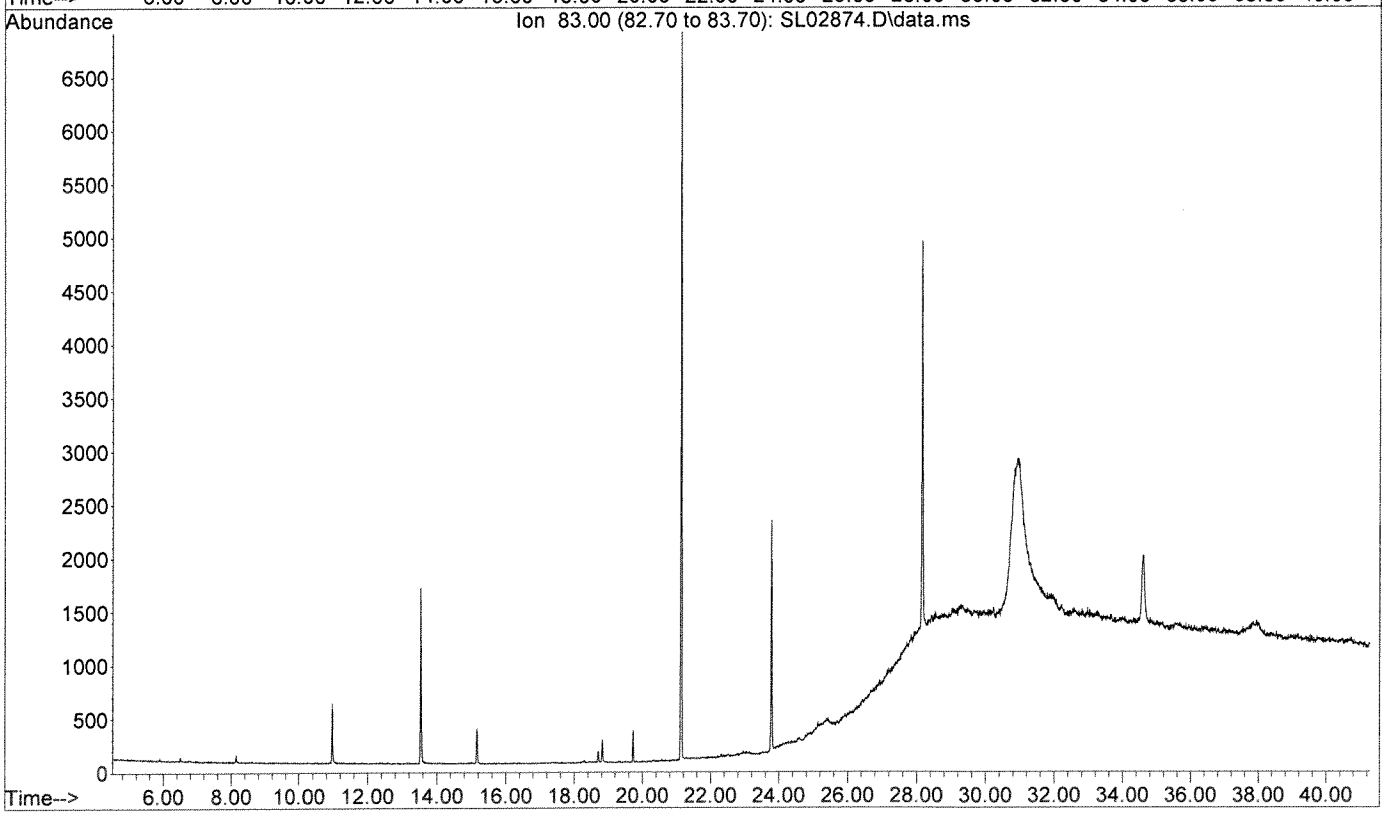
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012   18:44      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```





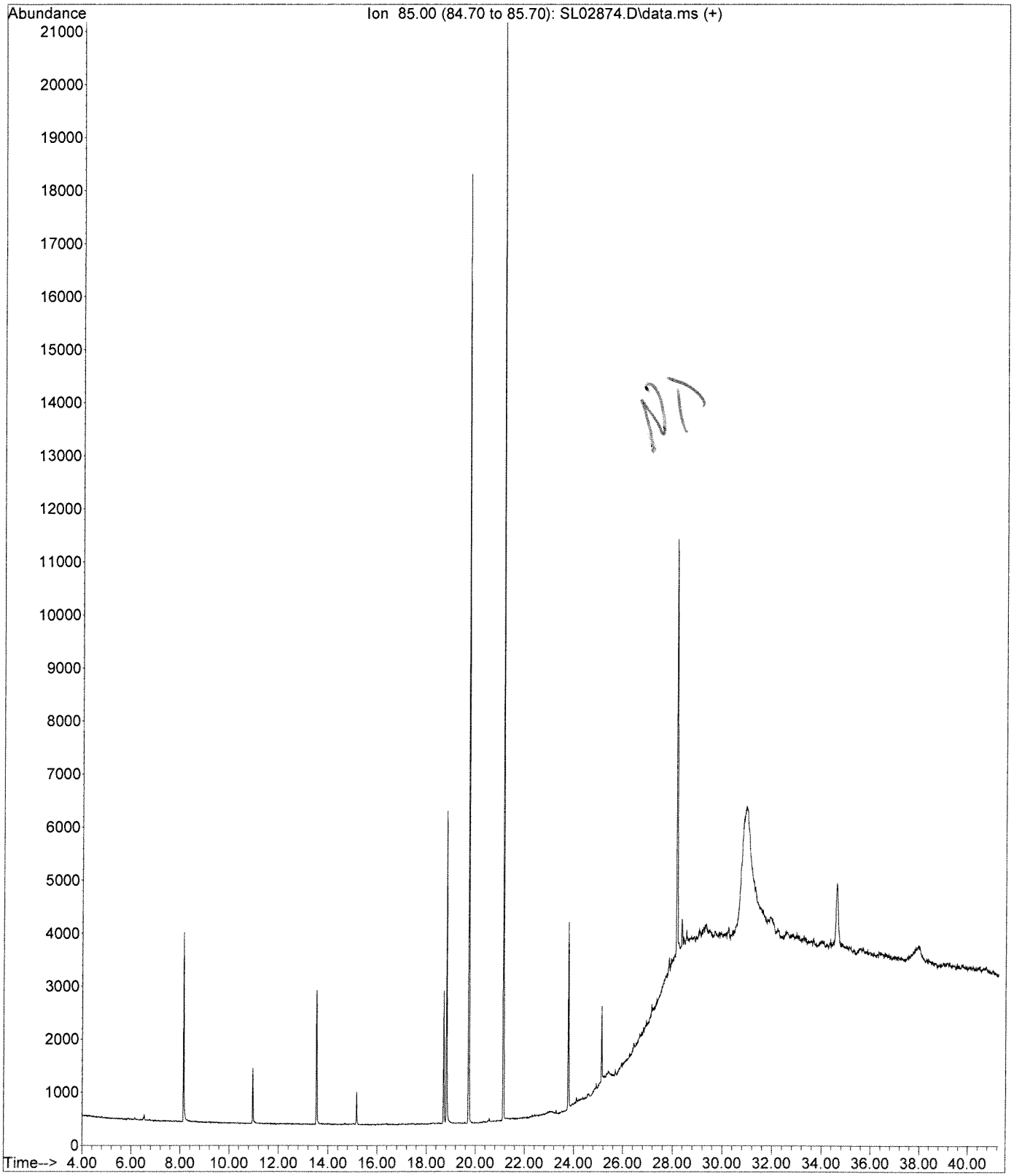
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



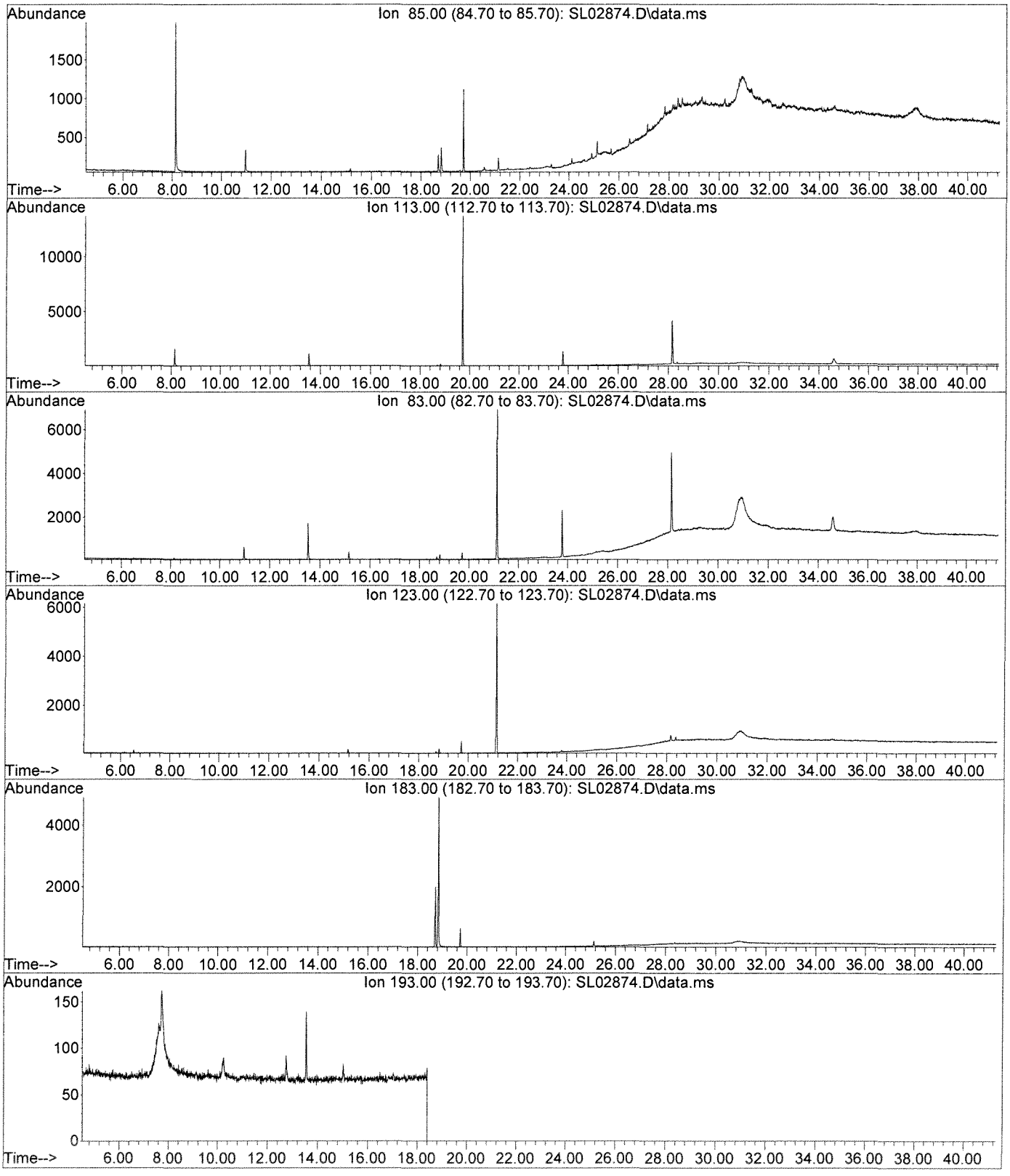
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



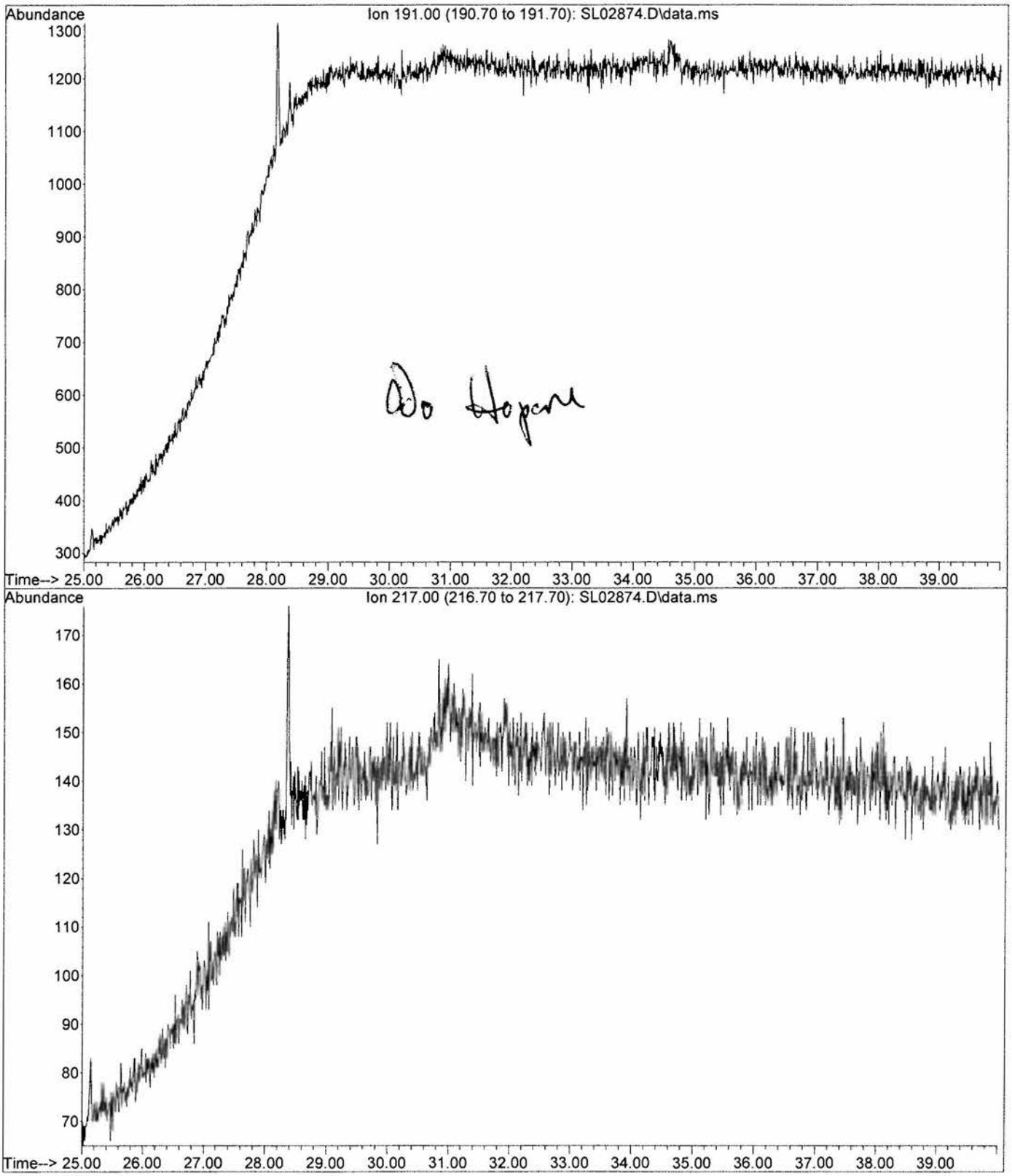
US EPA ARCHIVE DOCUMENT

```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



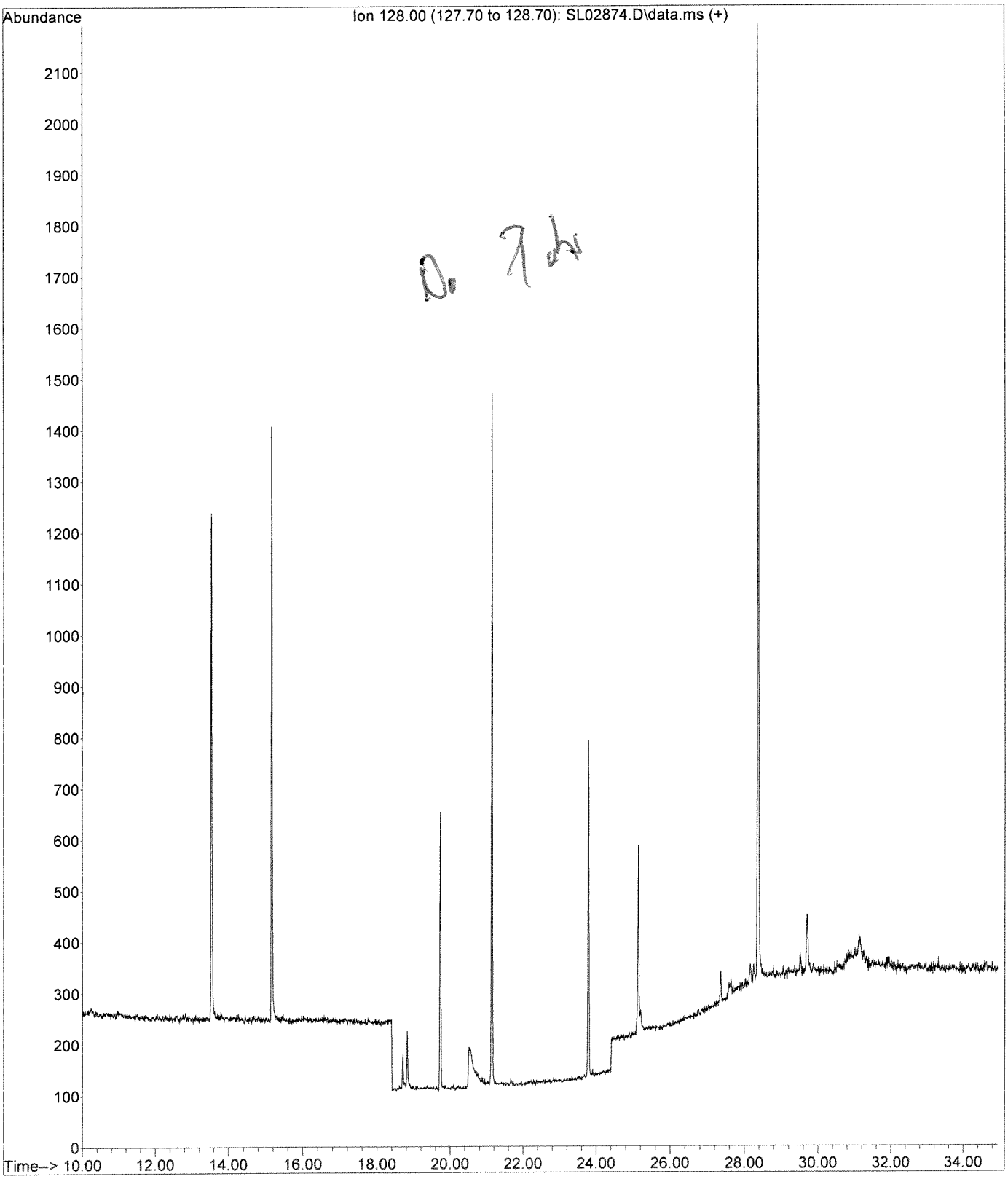
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



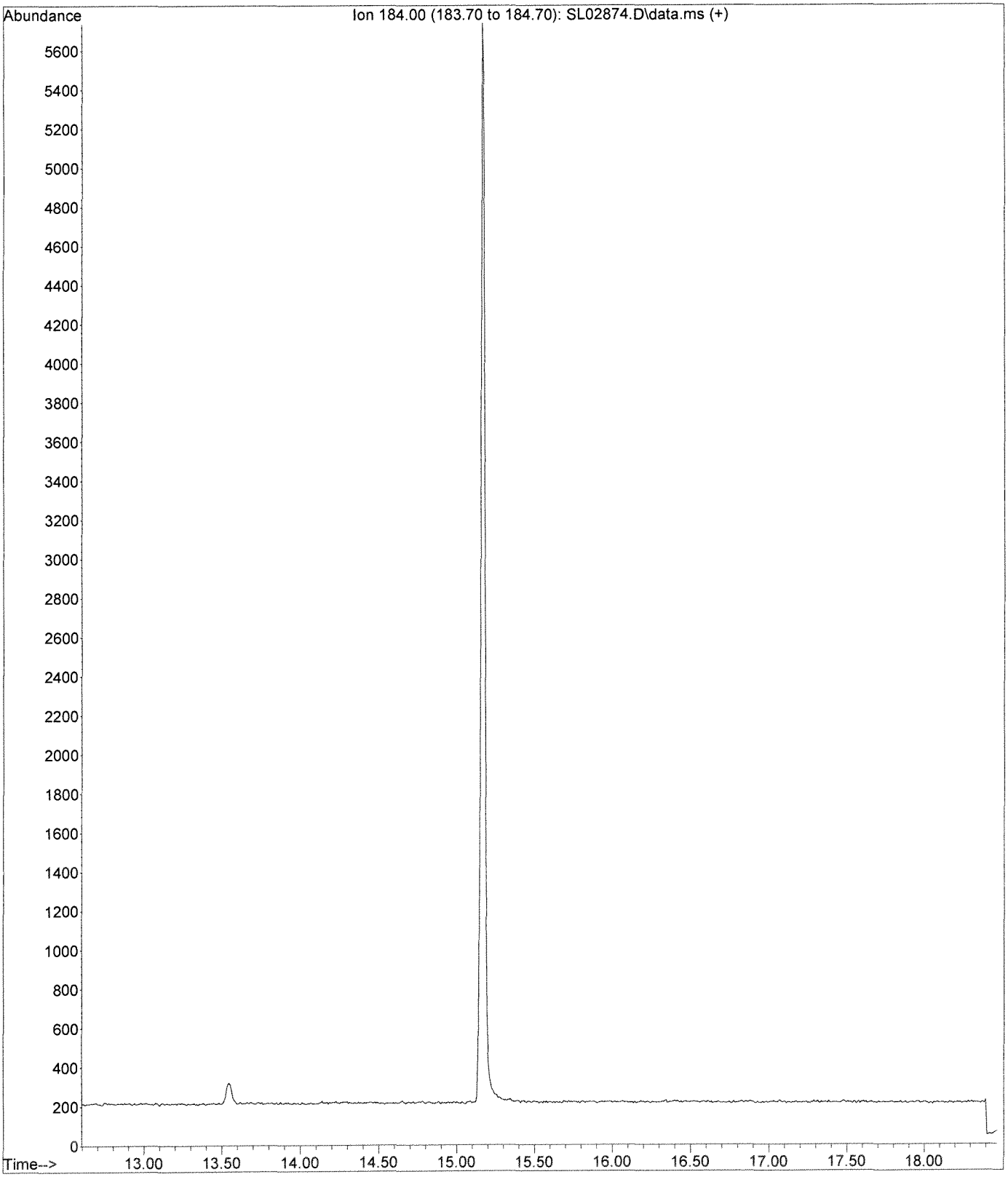
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

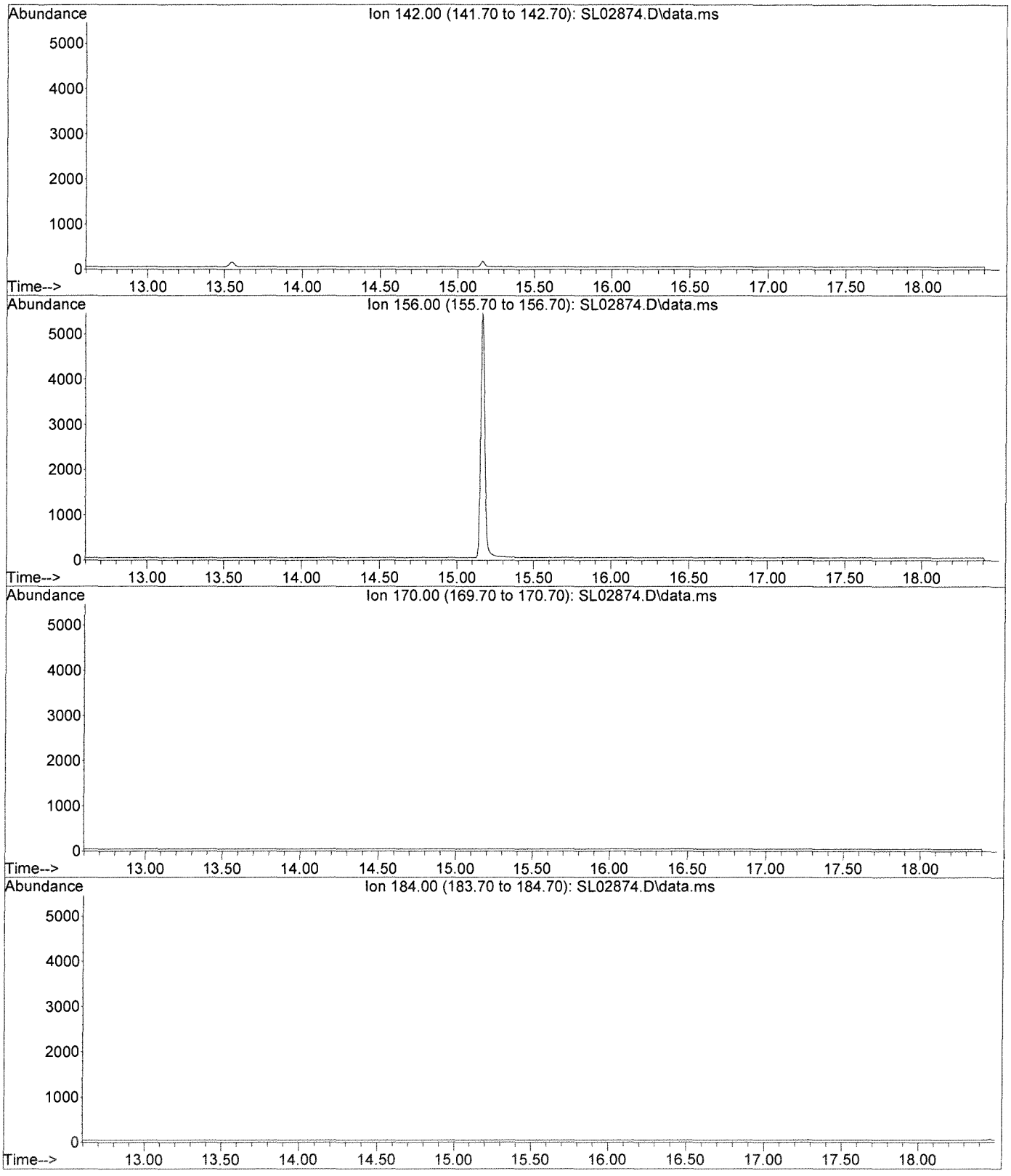
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



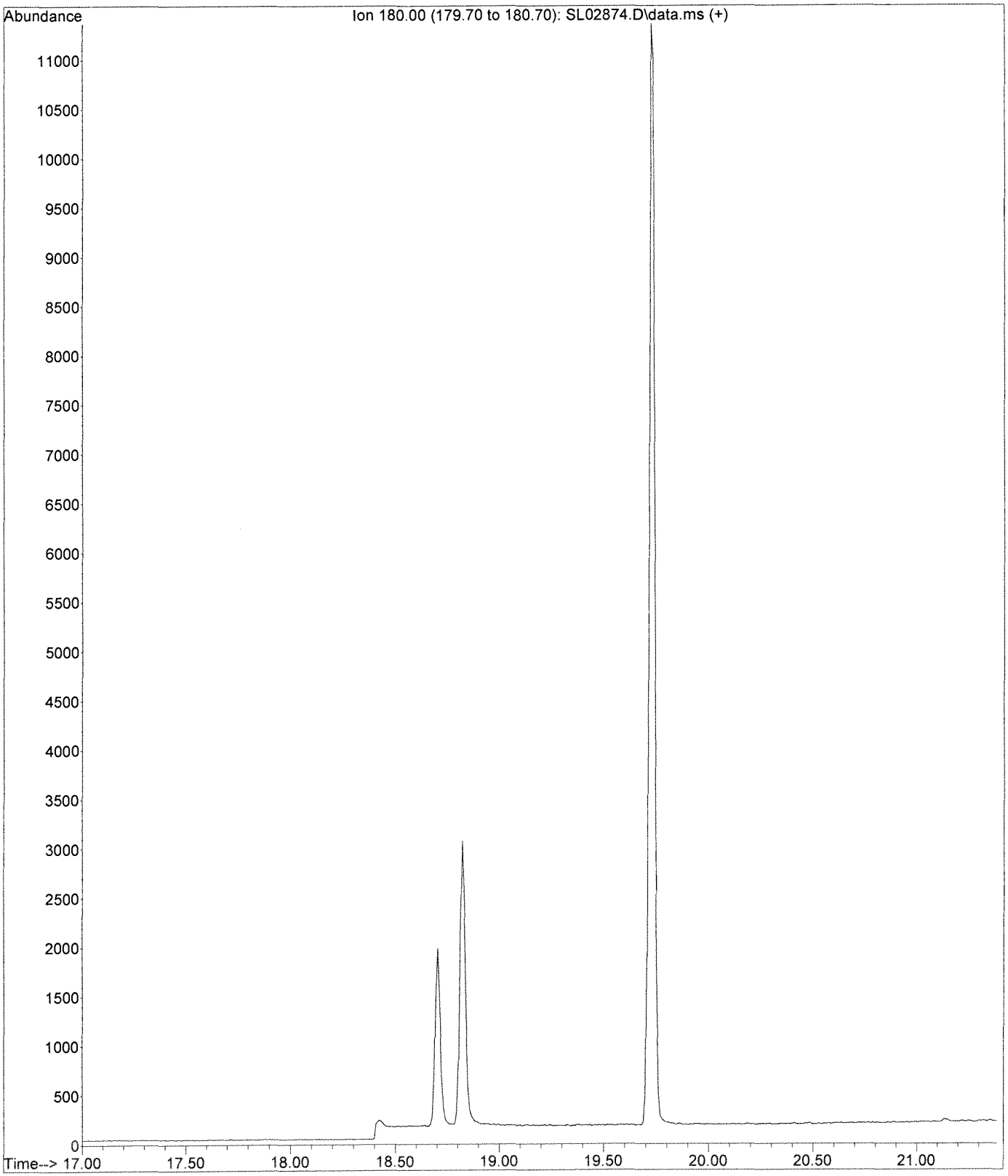
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



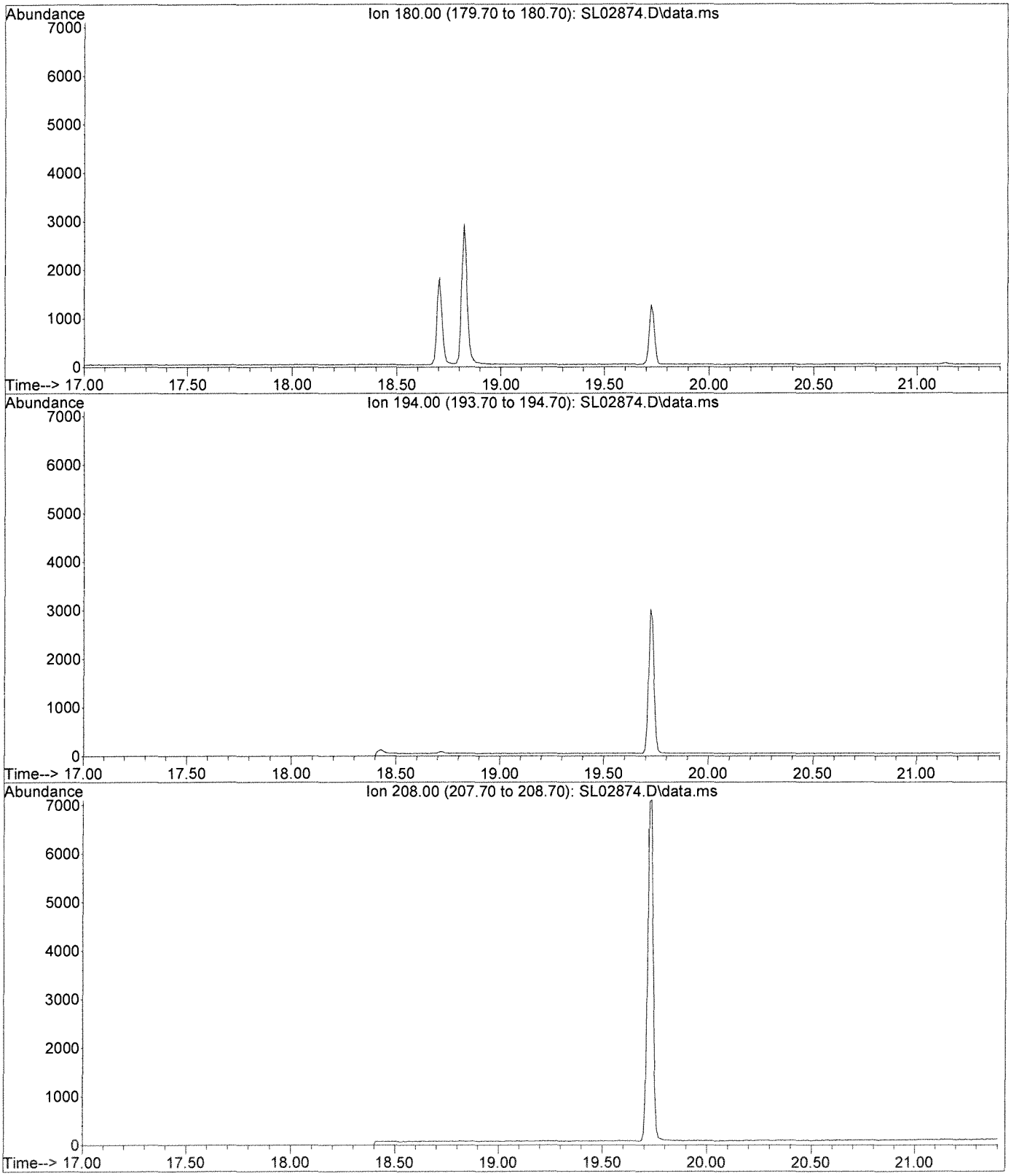
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



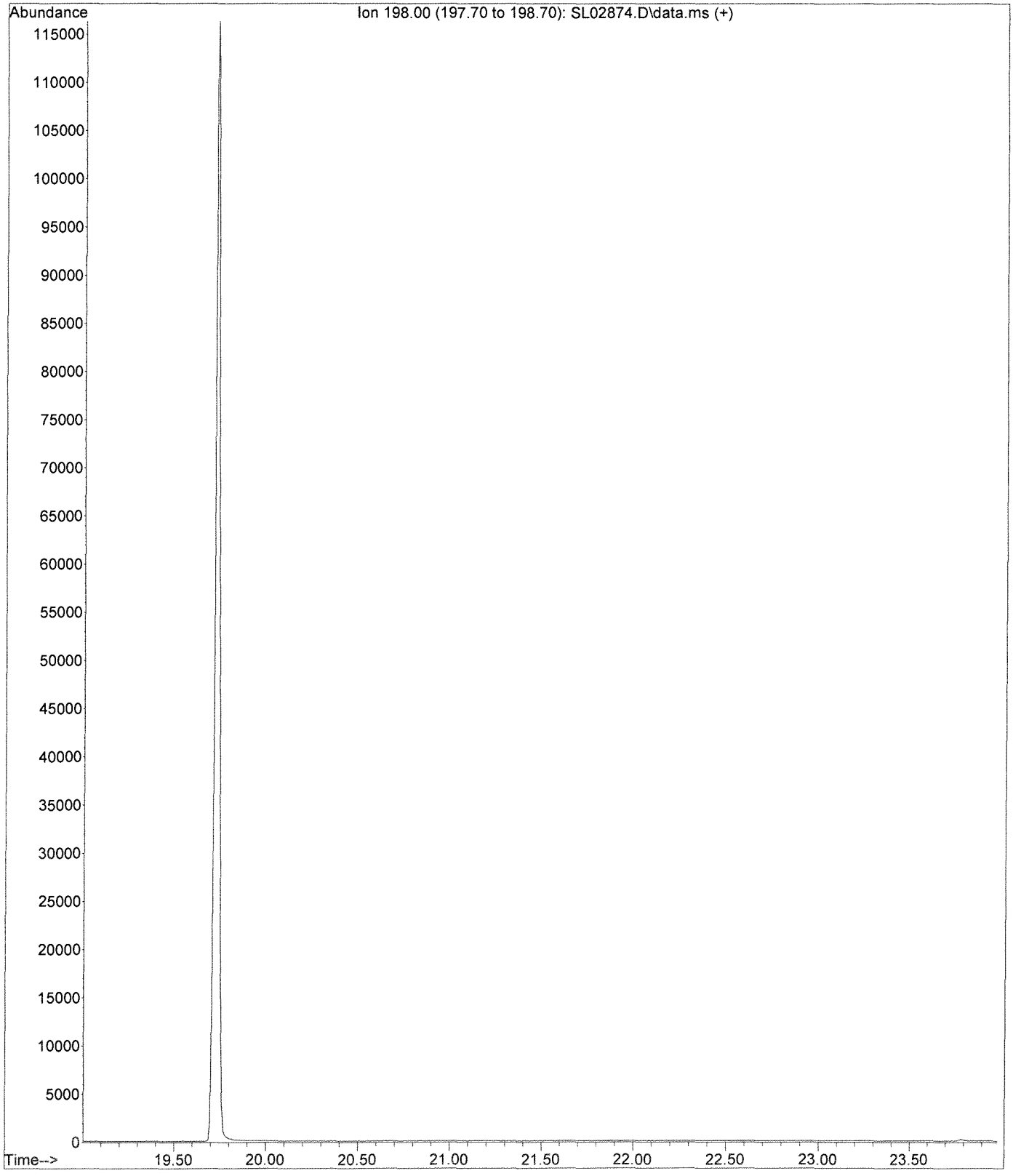
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



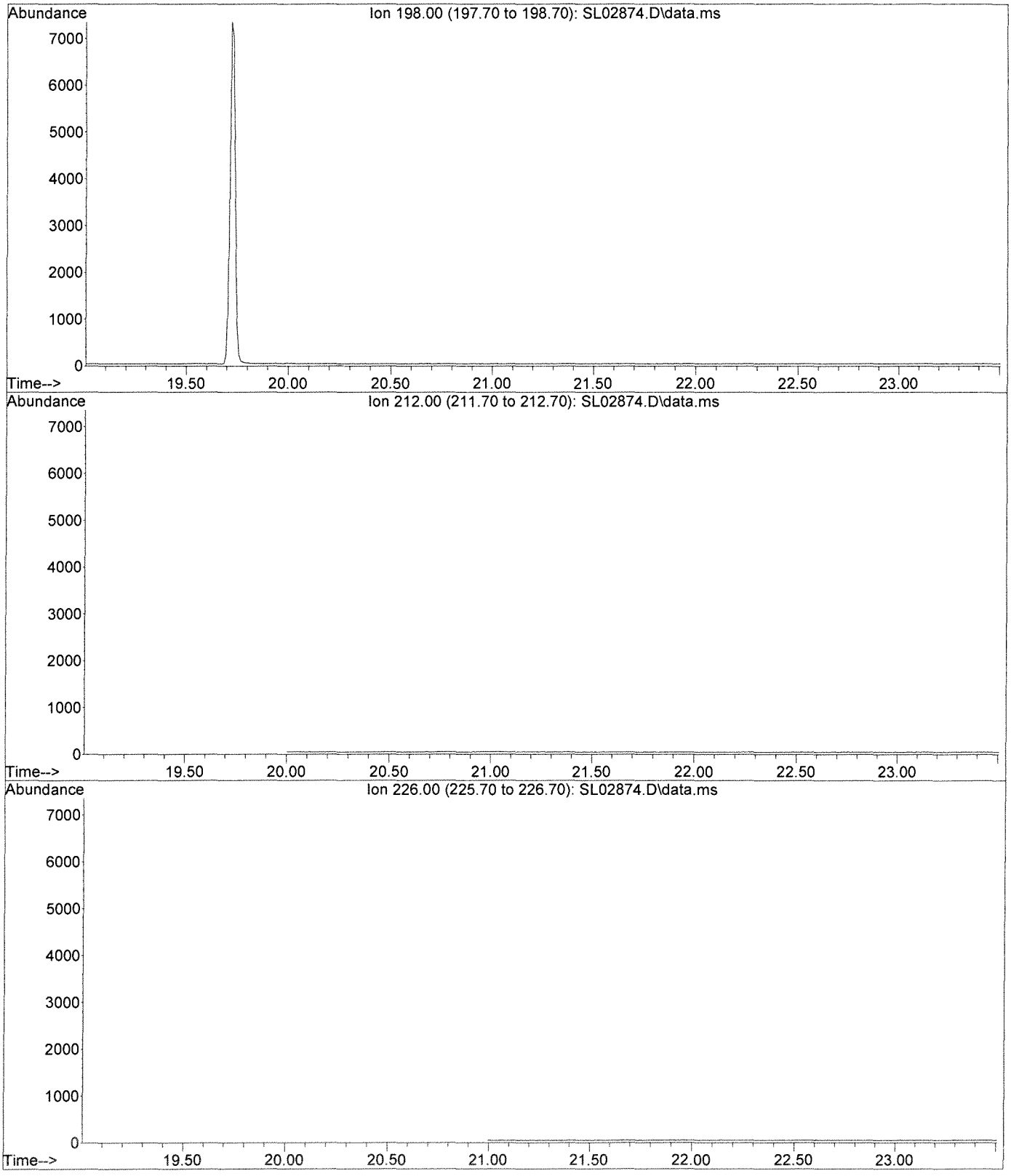
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



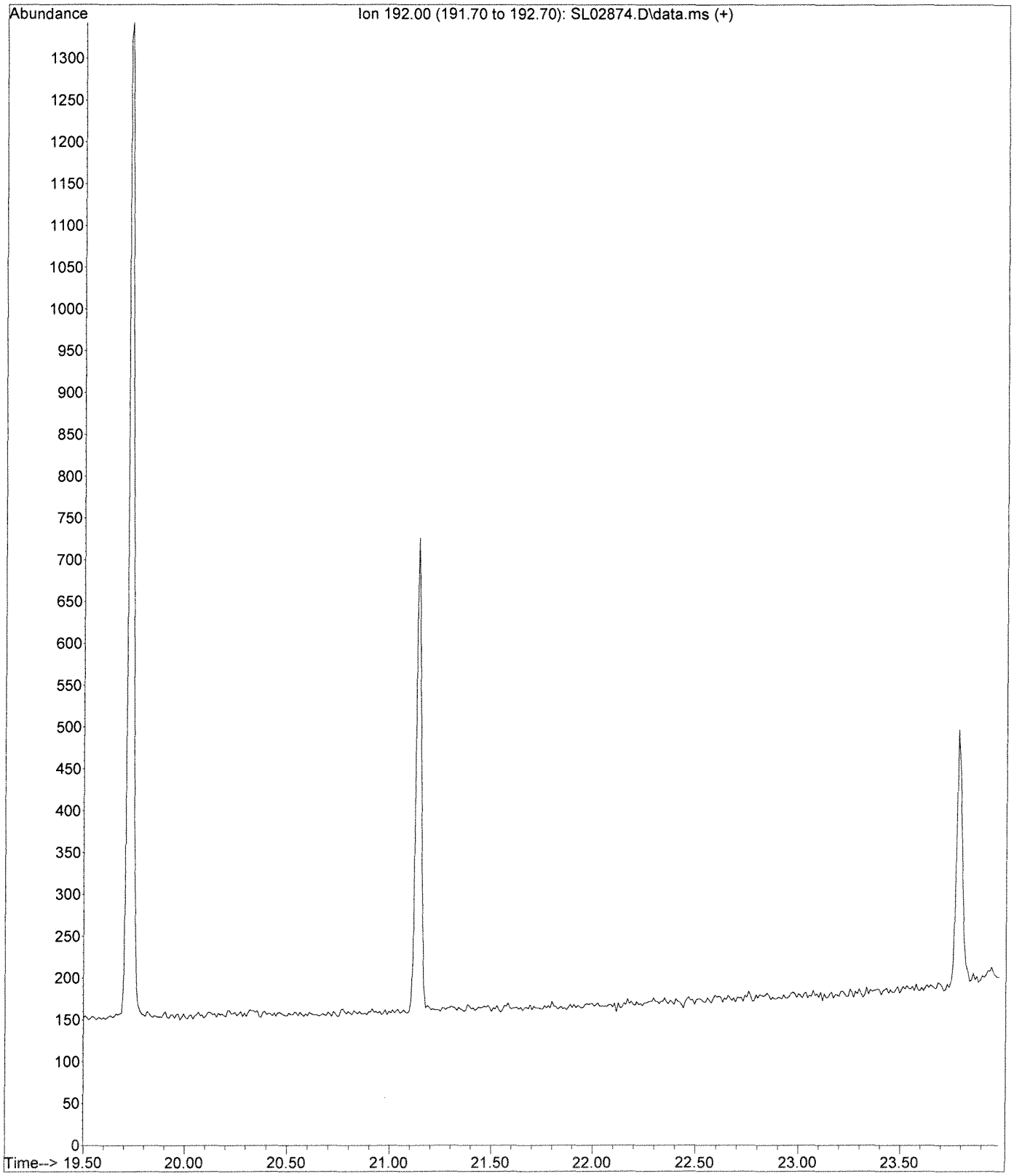
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



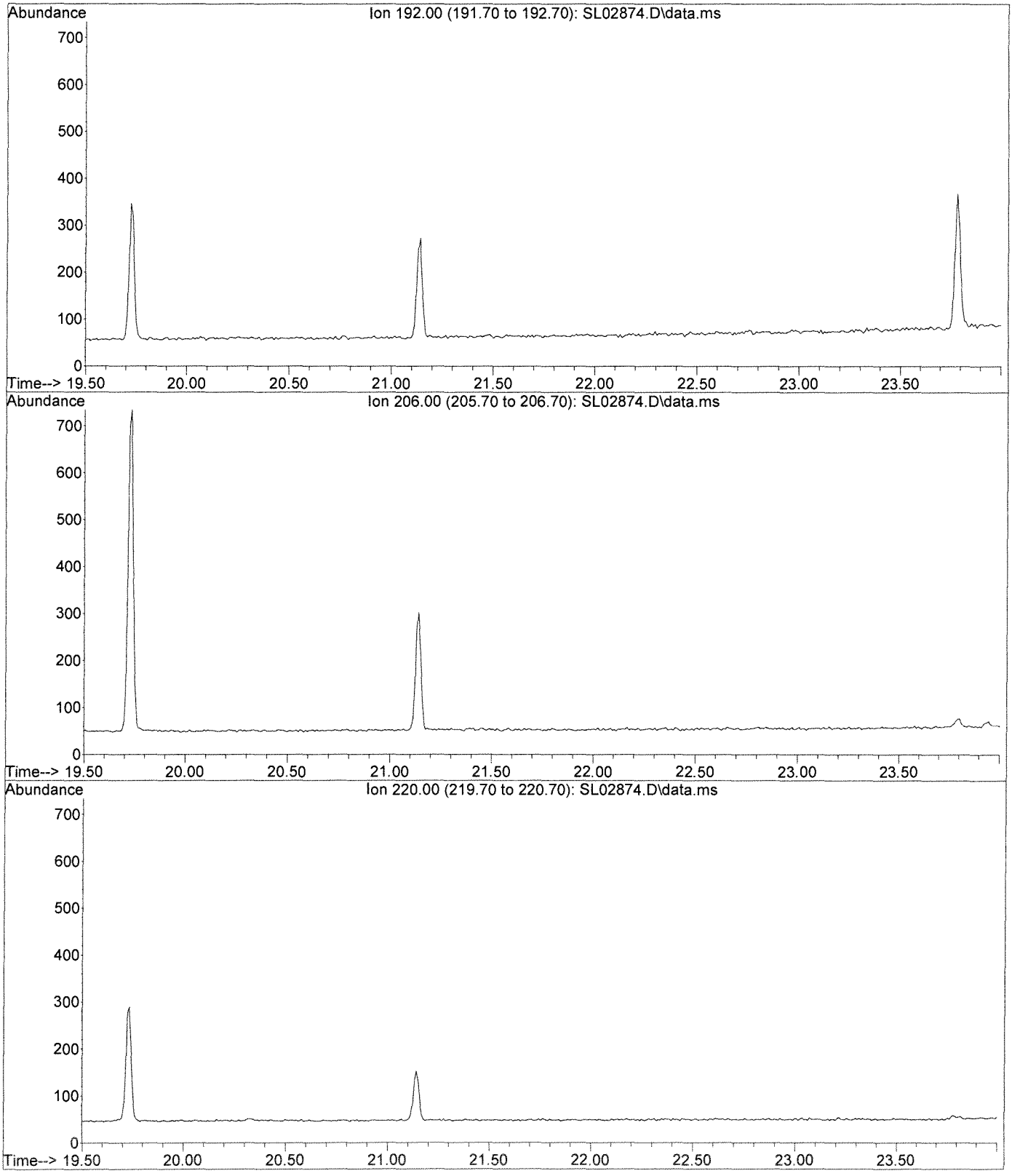
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



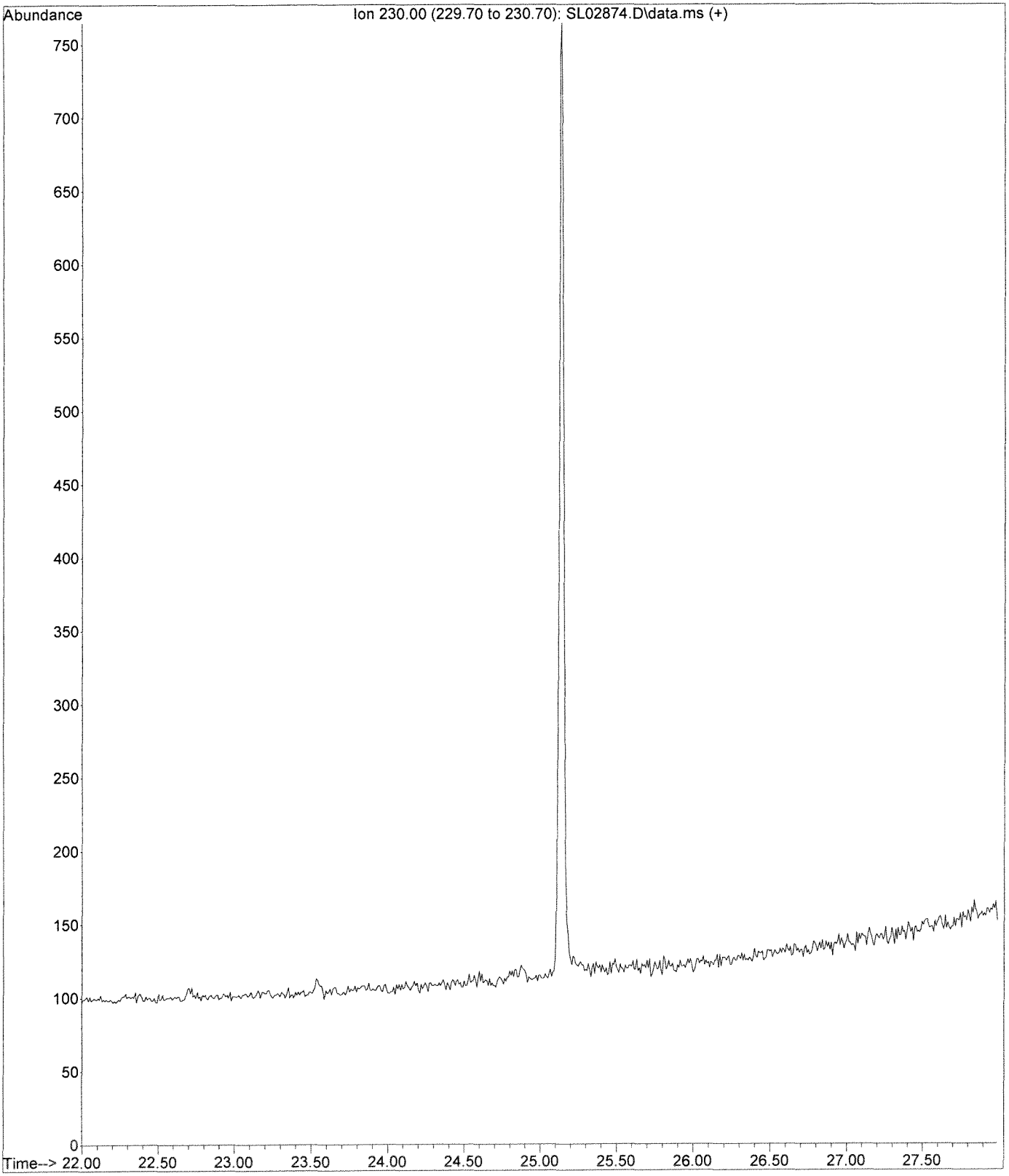
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



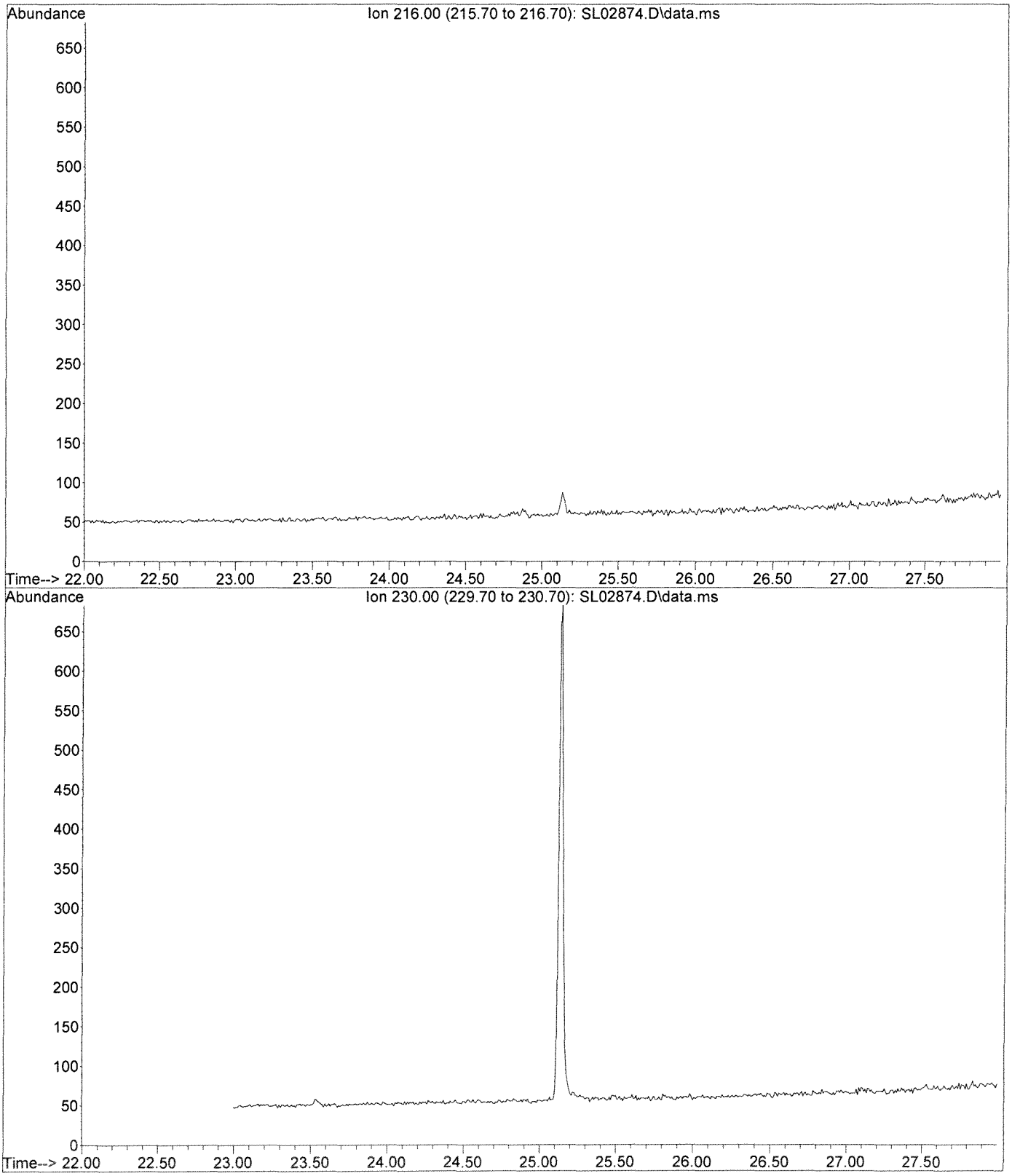
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



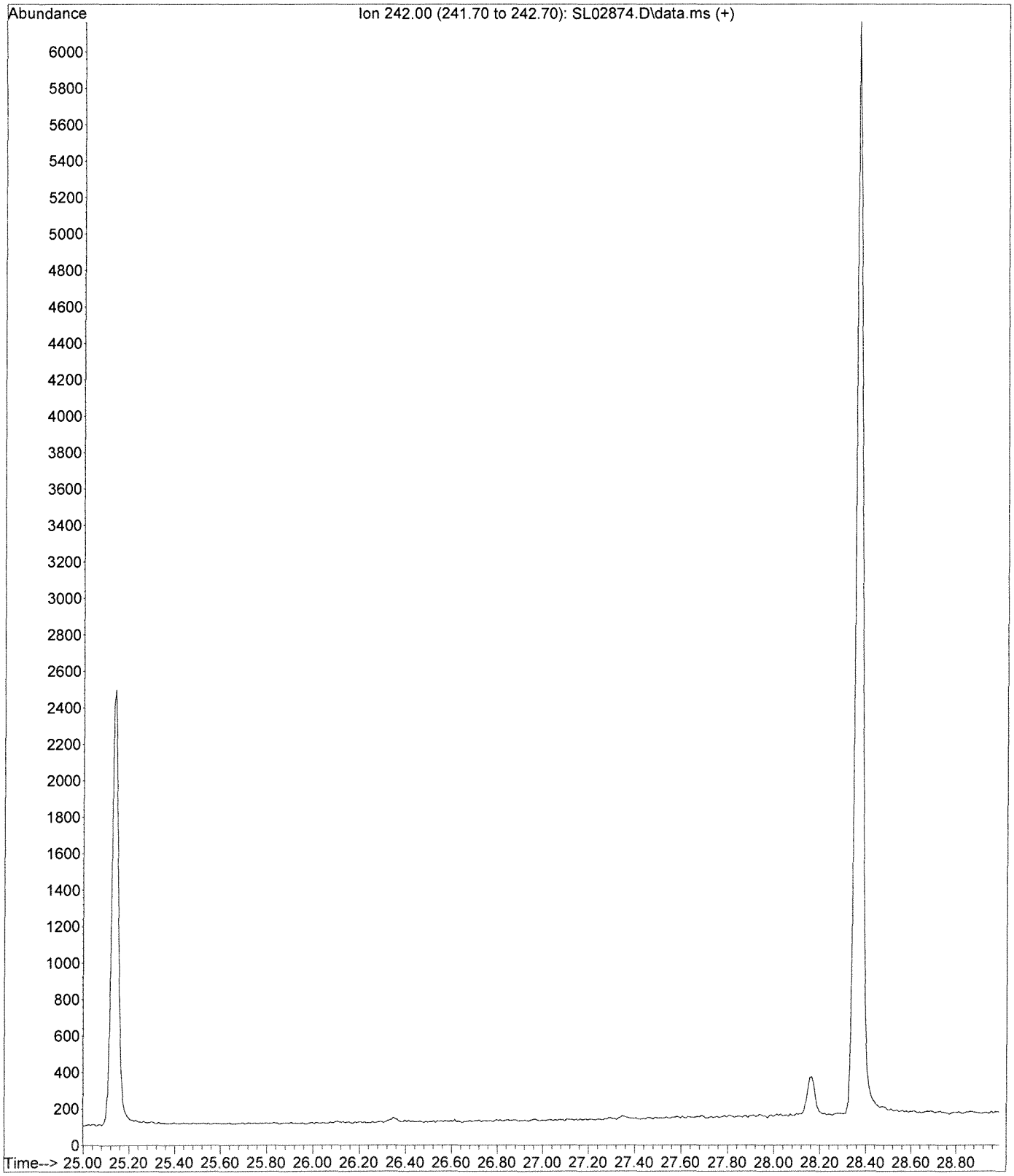
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012   18:44      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



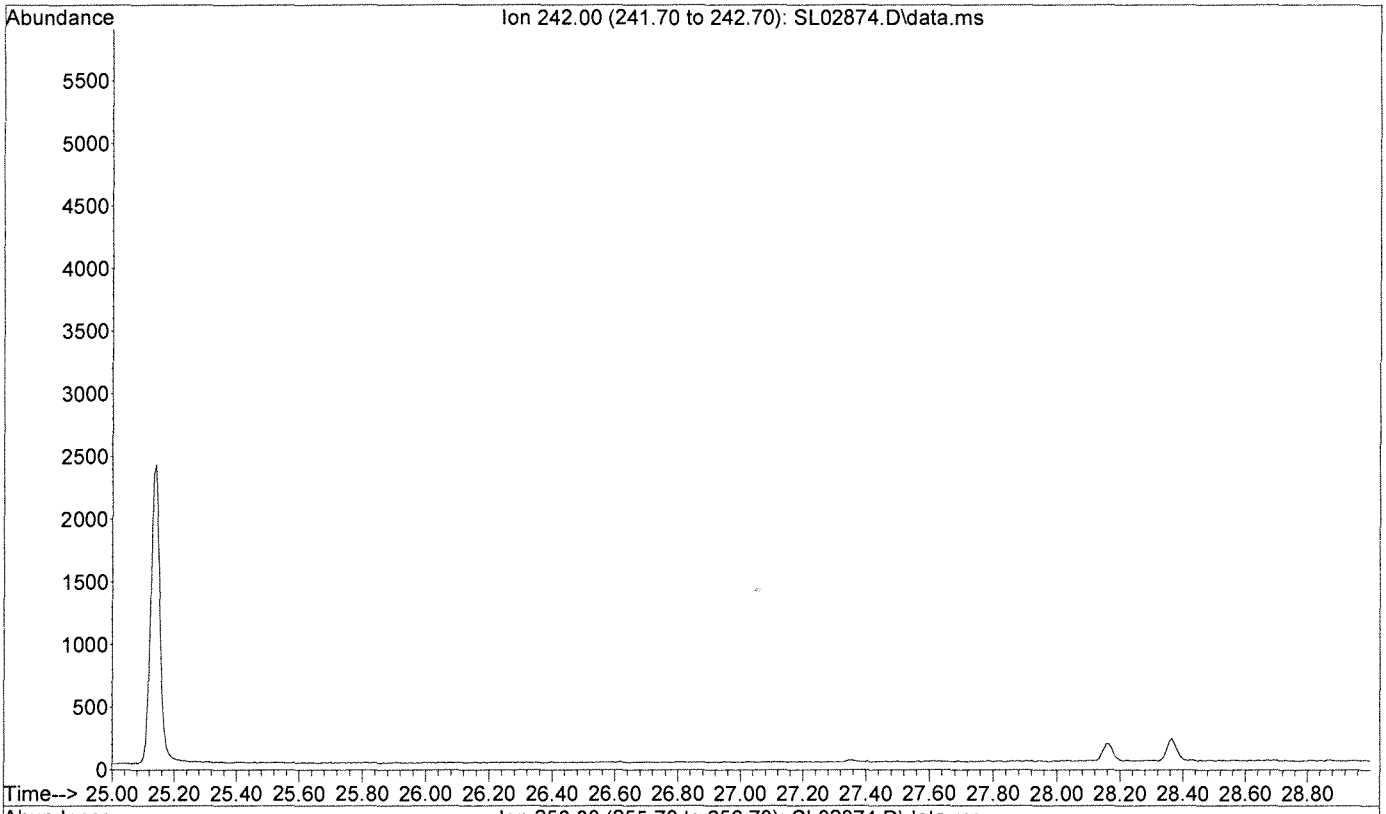
US EPA ARCHIVE DOCUMENT

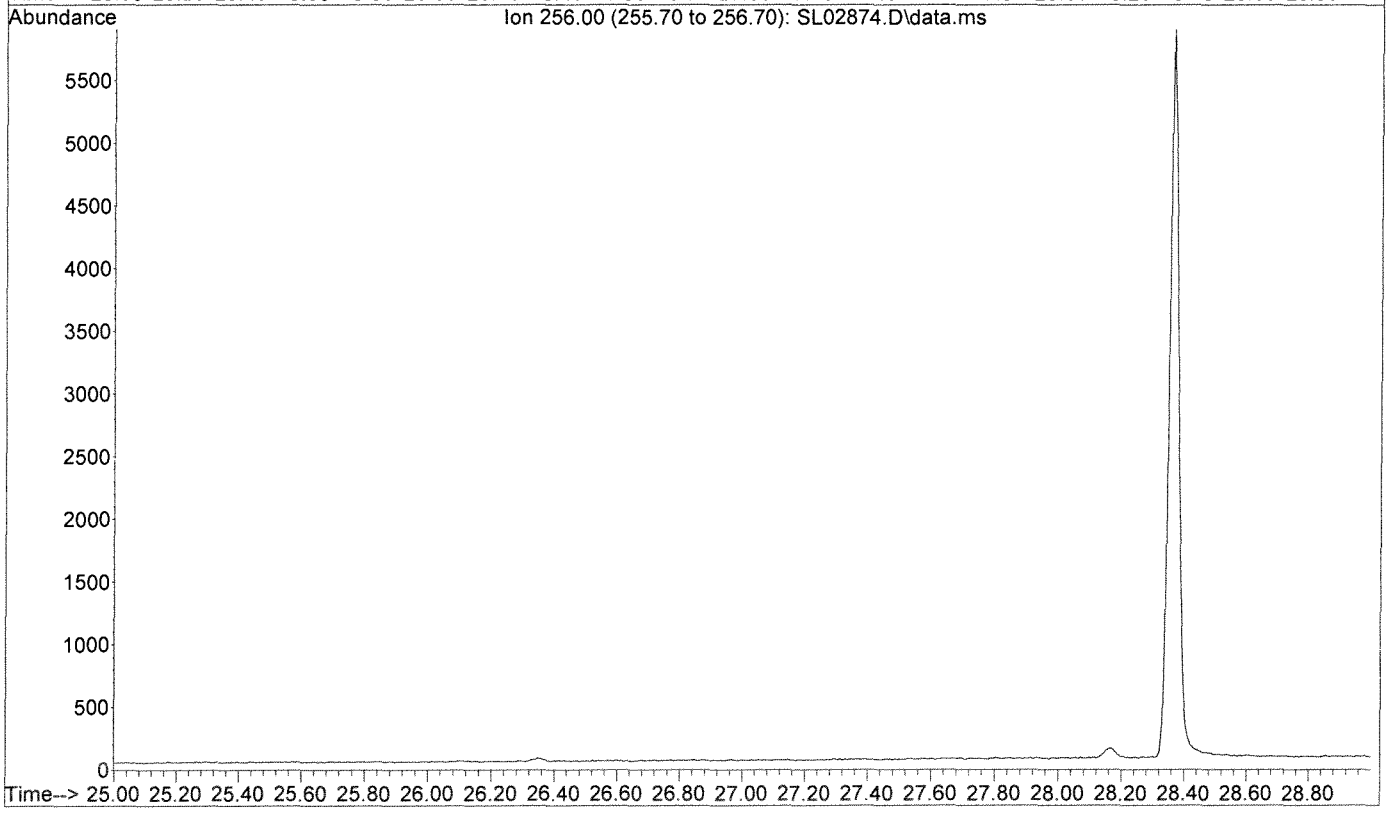
```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02874.D
Operator    : Syslo
Acquired    : 27 Feb 2012  18:44      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: BLK022712WD
Misc Info   : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1
```





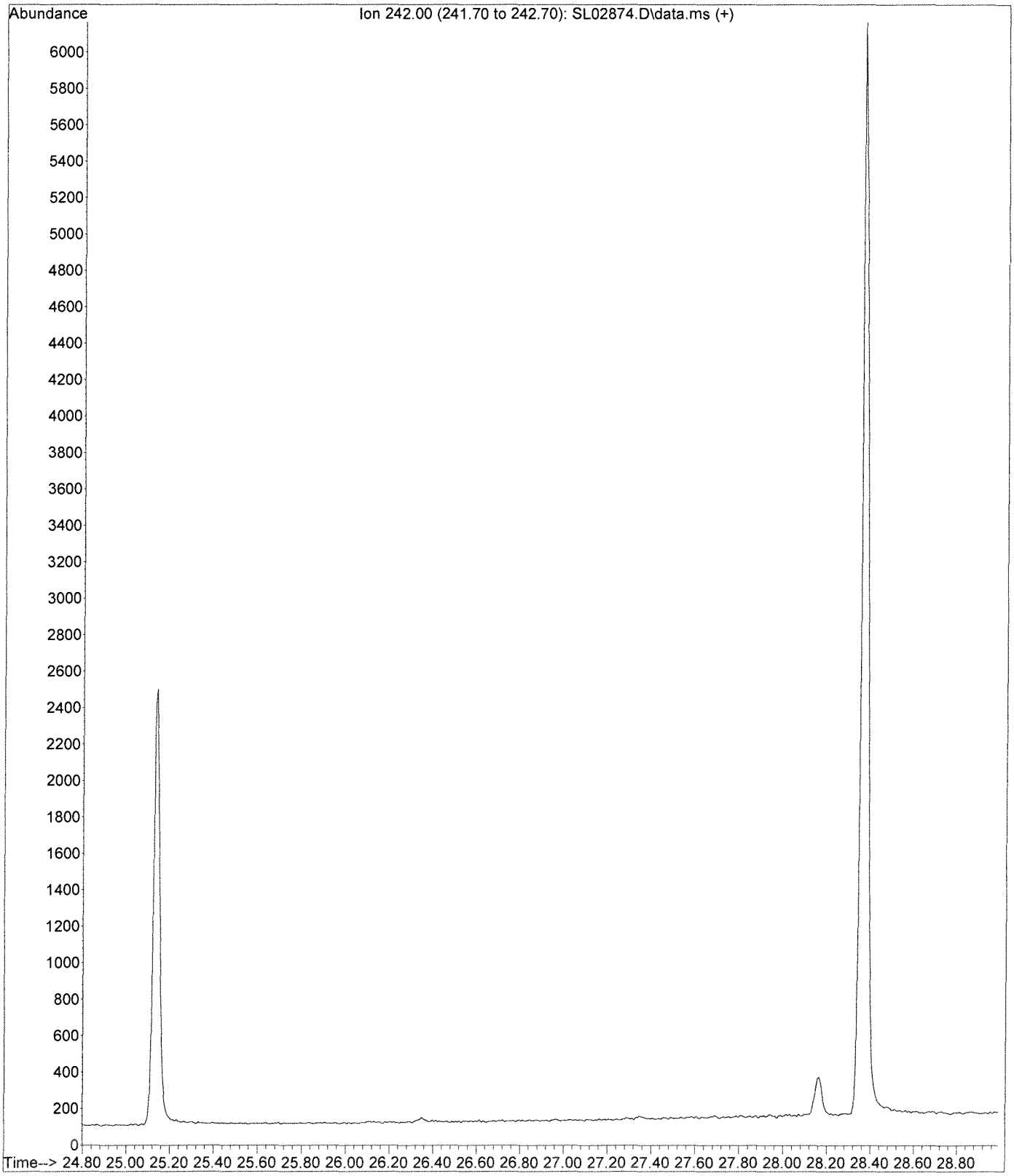
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02874.D
Operator : Syslo
Acquired : 27 Feb 2012 18:44 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: BLK022712WD
Misc Info : 10mL RUC0022 + 0.1mL RUK0077
Vial Number: 1



US EPA ARCHIVE DOCUMENT

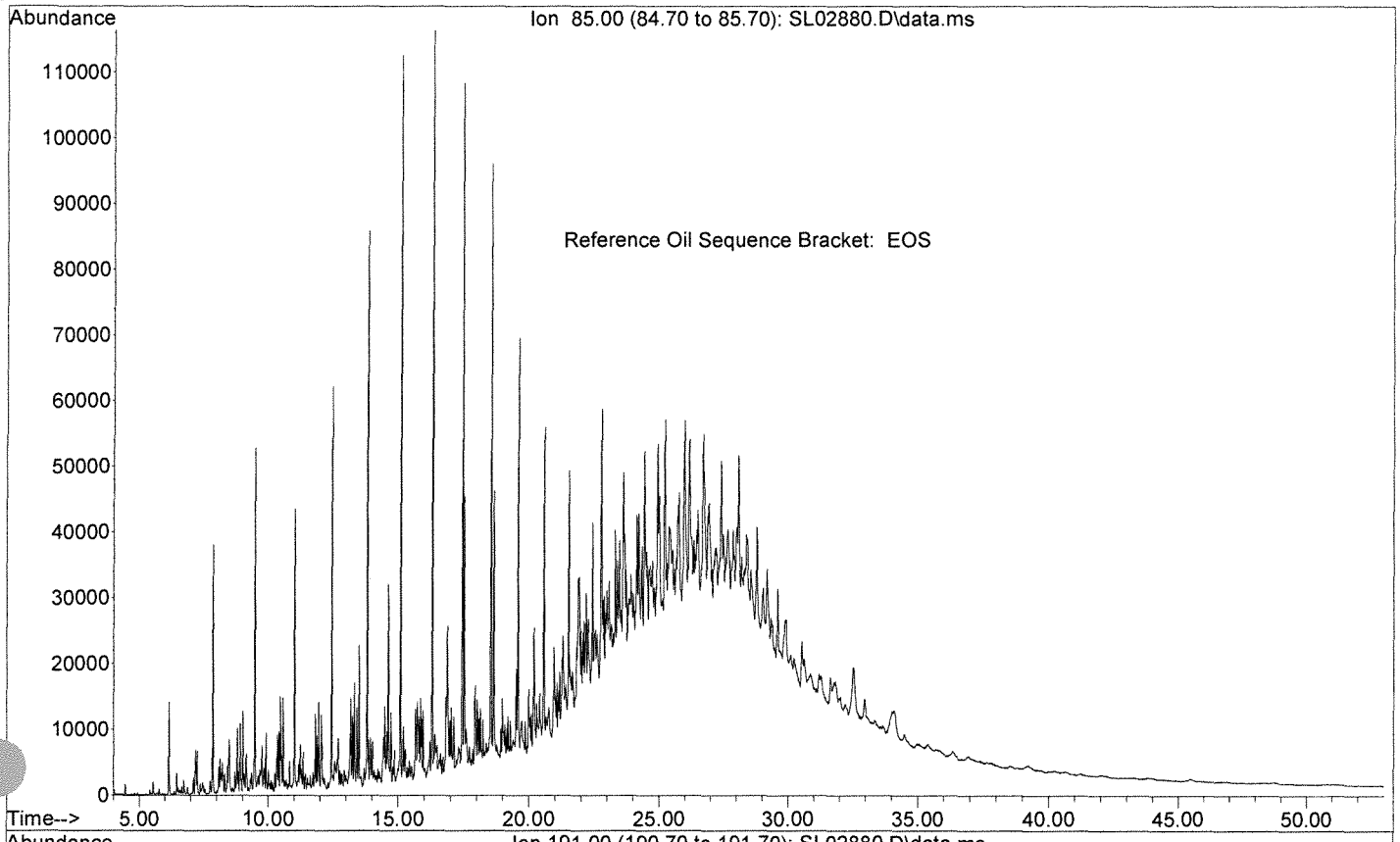
End of Sequence

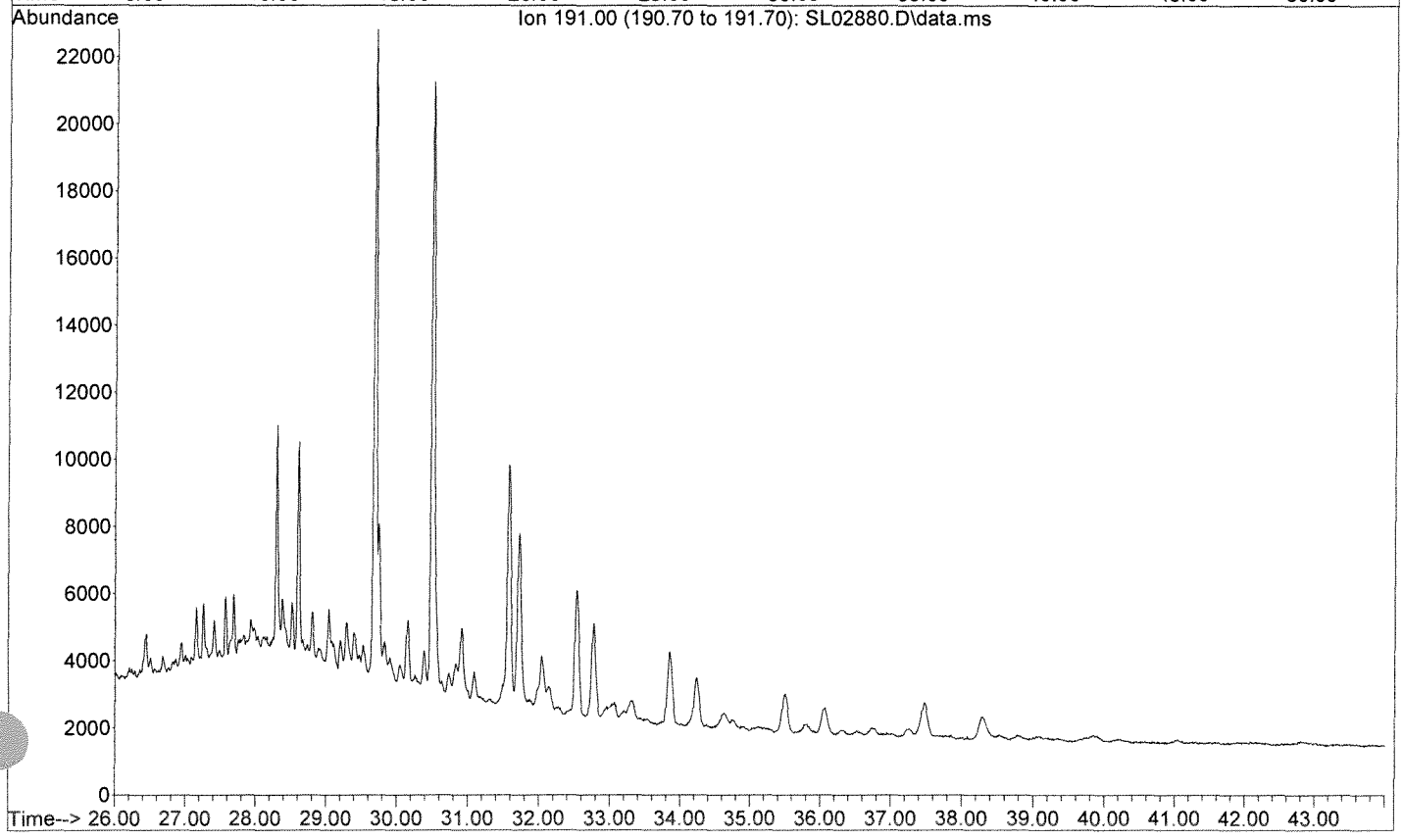
Blue Oil Bucket

CG-117

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02880.D
Operator : Syslo
Acquired : 28 Feb 2012 00:41 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003 EOS Duplicate
Vial Number: 7





Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02880.D
Acq On : 28 Feb 2012 00:41
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003 EOS Duplicate
ALS Vial : 7 Sample Multiplier: 1

Quant Time: Mar 01 13:14:45 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.708	188	558084	10.00	ug/mL	0.00
5) d30-Tetradecane	13.536	66	174777	10.00	ug/mL	0.00
6) d50-Tetracosane	23.783	66	210874	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.605	66	191945	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	0.000	188	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
3) d14-o-Terphenyl {S}	0.000	244	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
4) 5a-Androstane {S}	0.000	260	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
7) d62-Triacontane {S}	0.000	66	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
Target Compounds						Qvalue

(#) = qualifier out of range (m) = manual integration (+) = signals summed

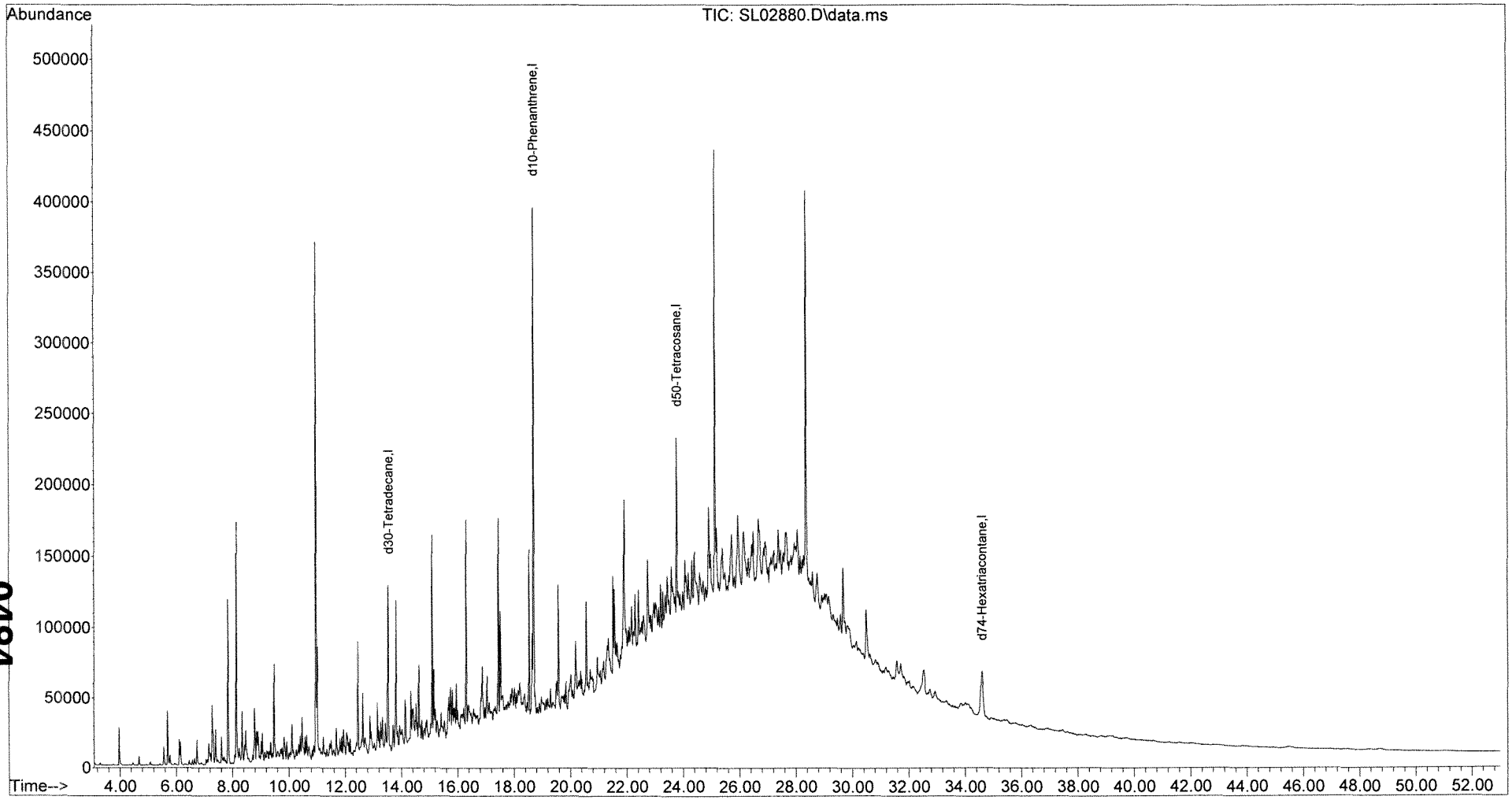
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02880.D
Acq On : 28 Feb 2012 00:41
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003 EOS Duplicate
ALS Vial : 7 Sample Multiplier: 1

Quant Time: Mar 01 13:14:45 2012
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Quant Title : DRO/TPH ICAL + Surr. 05/16/11
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SERAS-017-DTM-011413_3



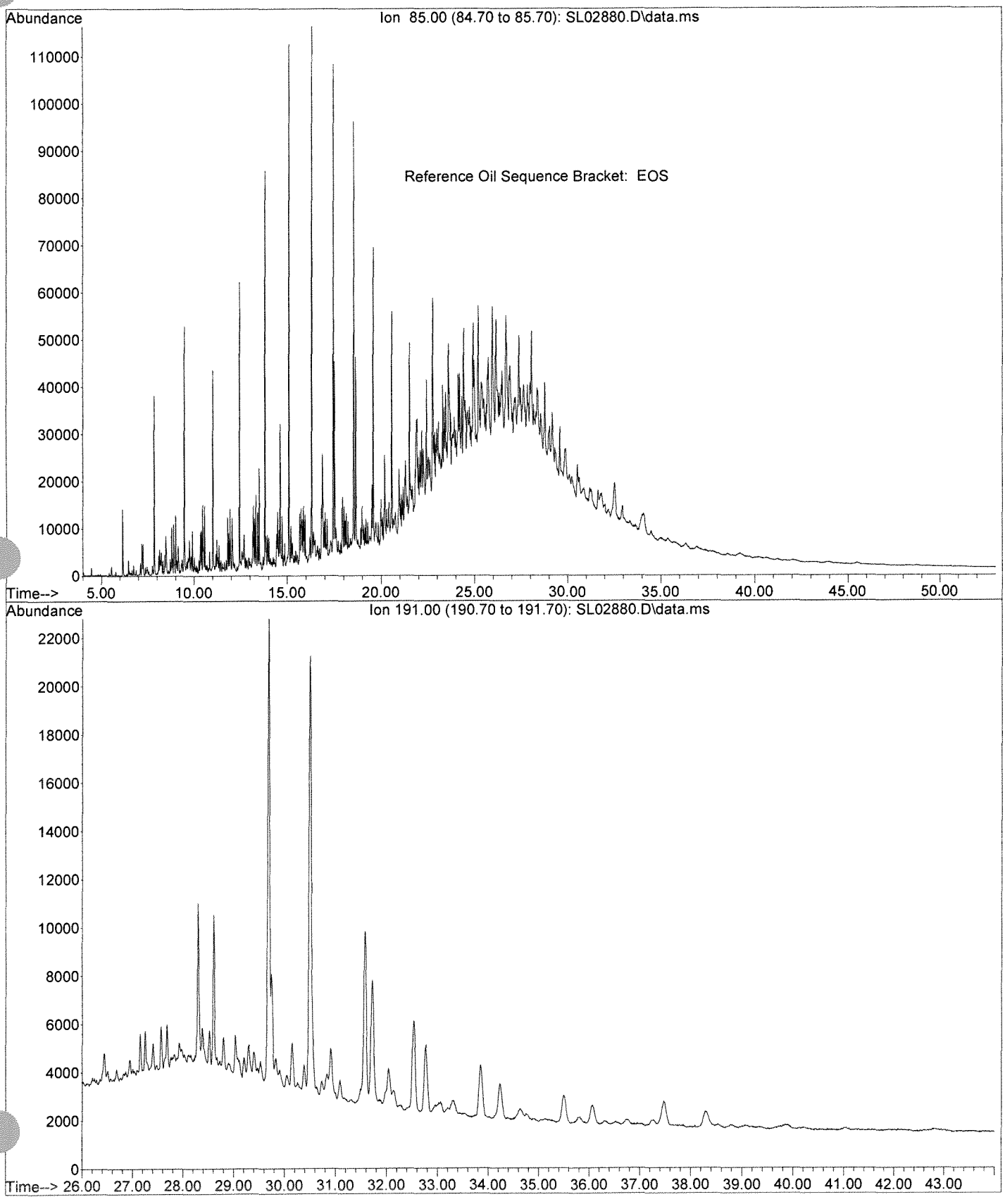
0494

```
! File  : FIREF117.MAC
! J. Syslo MACRO for reference fingerprint using CG117: 11/22/09
!  Hopanes are normalized for easy viewing!
! Draws chromatogram in window 2 and window 1 . .  you have to print "TIC &
Spectrum"
!  I have a lot of old CG-117's like that - better reference"  JS

NAME FIREF117
format separated,,,0
EIC 4:55,85
exchange r0,x
EIC 26:44,191
exchange r0,y
draw 2,y
draw 1,x
REMOVE FIREF117
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02880.D
Operator : Syslo
Acquired : 28 Feb 2012 00:41 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003 EOS Duplicate
Vial Number: 7



SERAS-017-0003
SL02875.D

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02875.D
Acq On : 27 Feb 2012 19:33
Operator : Syslo
Sample : SERAS-017-0003
Misc : 275-55-05 {0.116g in10mL}
ALS Vial : 2 Sample Multiplier: 1

Quant Time: Mar 01 13:06:26 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	306220	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	99120	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	125874	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	118393	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.822	188	566158	17.39	ug/mL	0.00
Spiked Amount 20.000			Recovery =	86.95%		
3) d14-o-Terphenyl {S}	19.733	244	275007	19.22	ug/mL	0.00
Spiked Amount 20.000			Recovery =	96.10%		
4) 5a-Androstane {S}	21.141	260	36068	20.71	ug/mL	0.00
Spiked Amount 20.000			Recovery =	103.55%		
7) d62-Triacontane {S}	28.156	66	250640	22.42	ug/mL	0.00
Spiked Amount 20.000			Recovery =	112.10%		
Target Compounds						Qvalue

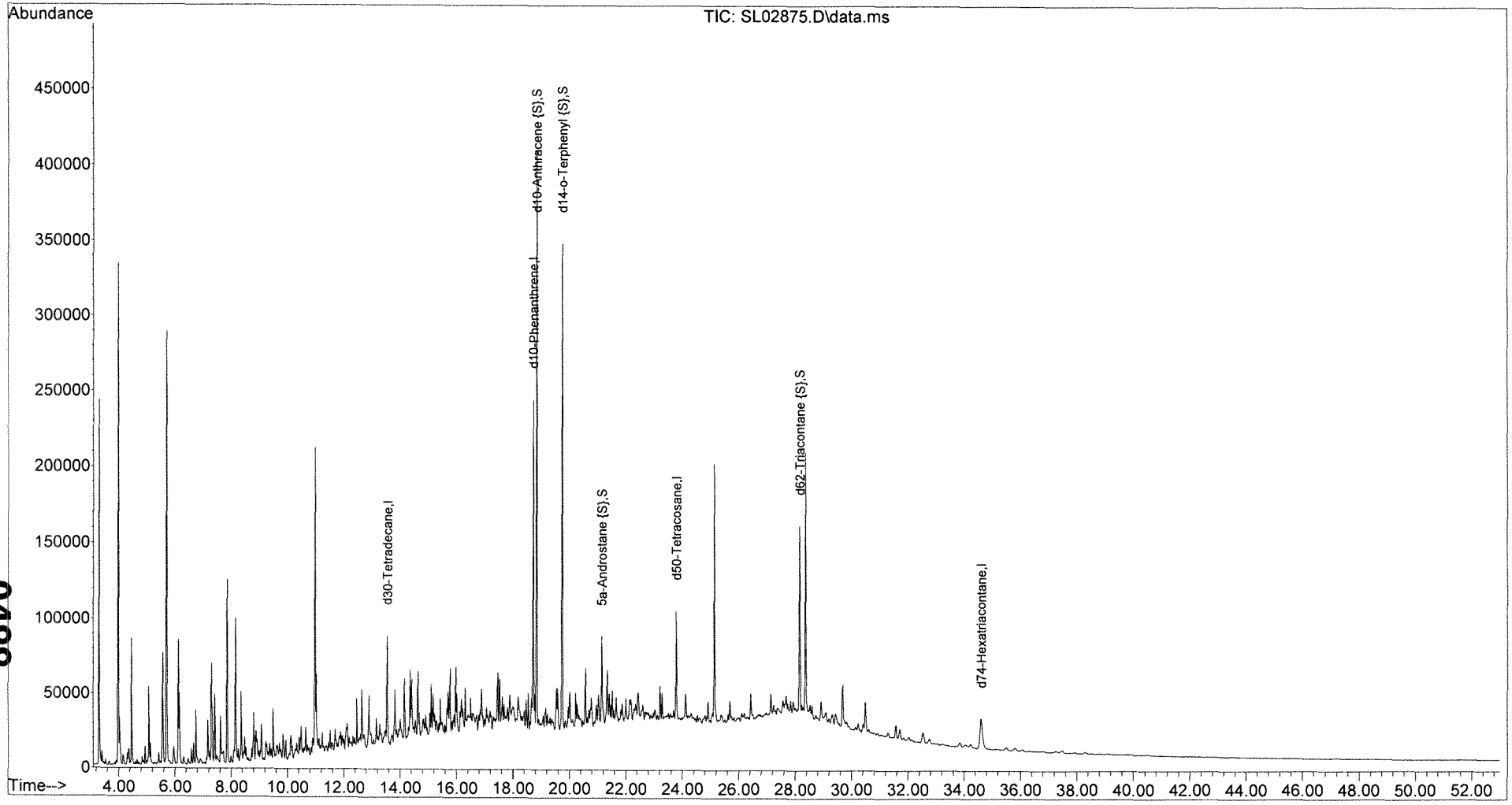
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (QT Reviewed)

Data Pa : C:\msdchem\1\DATA\022712\
Data File : SL02875.D
Acq On : 27 Feb 2012 19:33
Operator : Syslo
Sample : SERAS-017-0003
Misc : 275-55-05 {0.116g in10mL}
ALS Vial : 2 Sample Multiplier: 1

Quant Time: Mar 01 13:06:26 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0499

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

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Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM.* Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

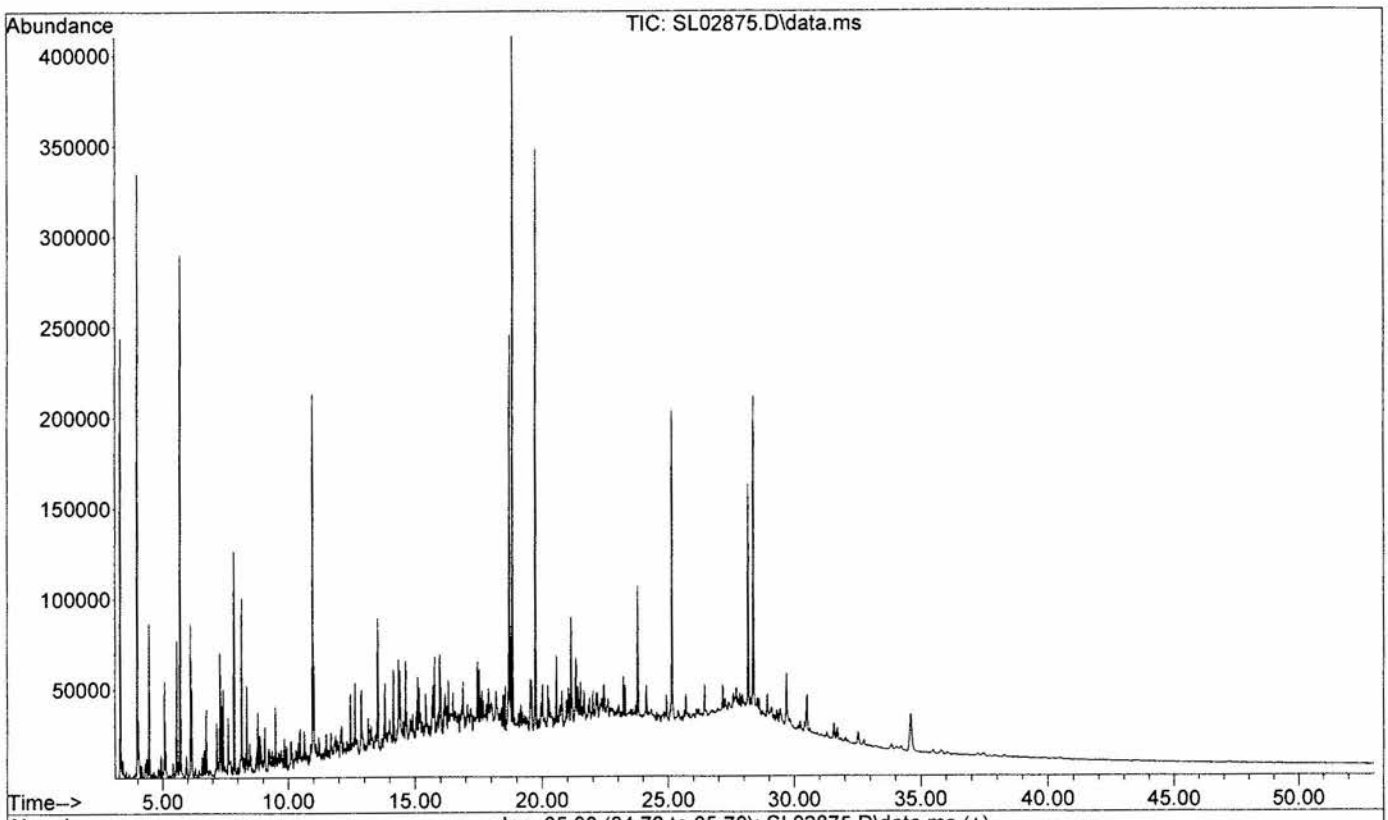
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

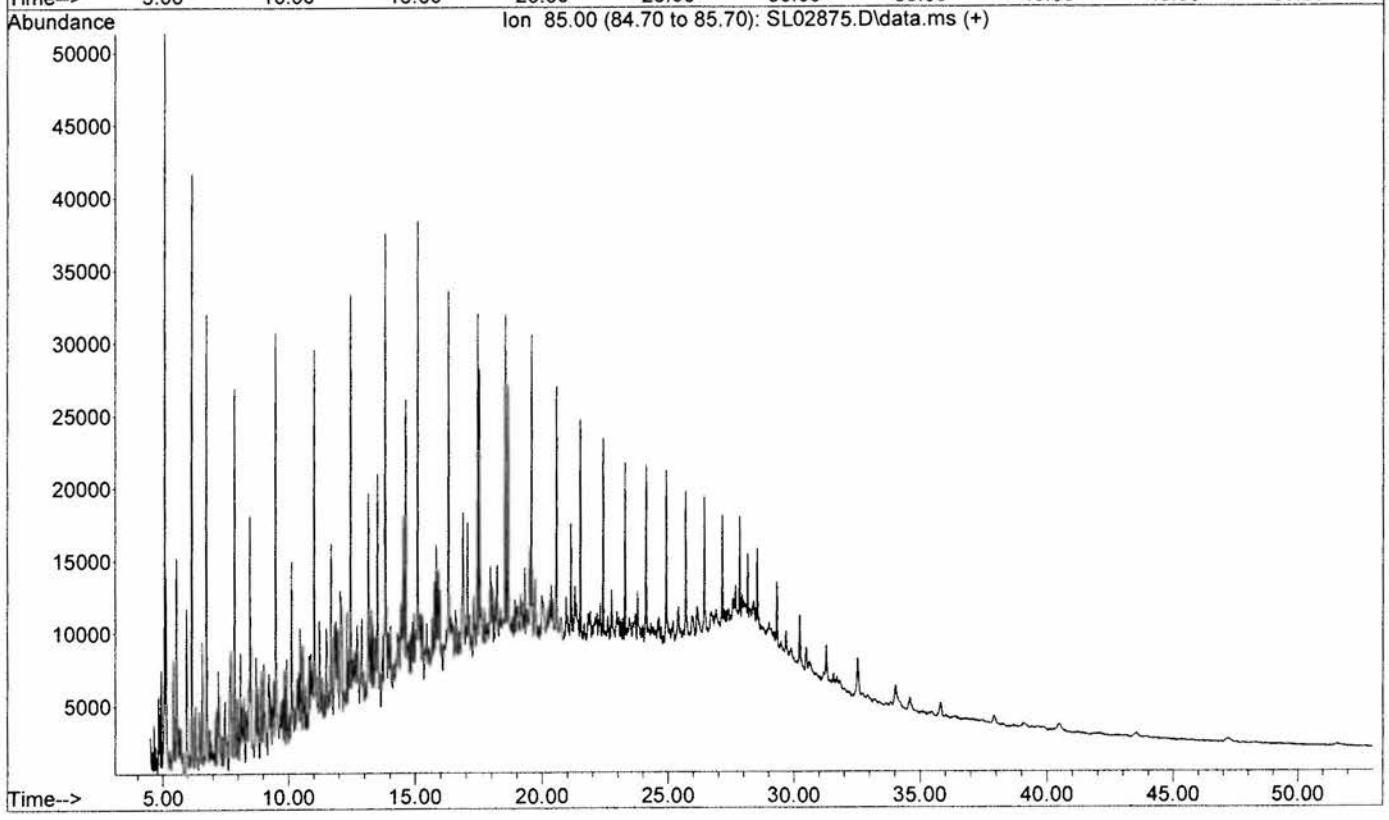
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



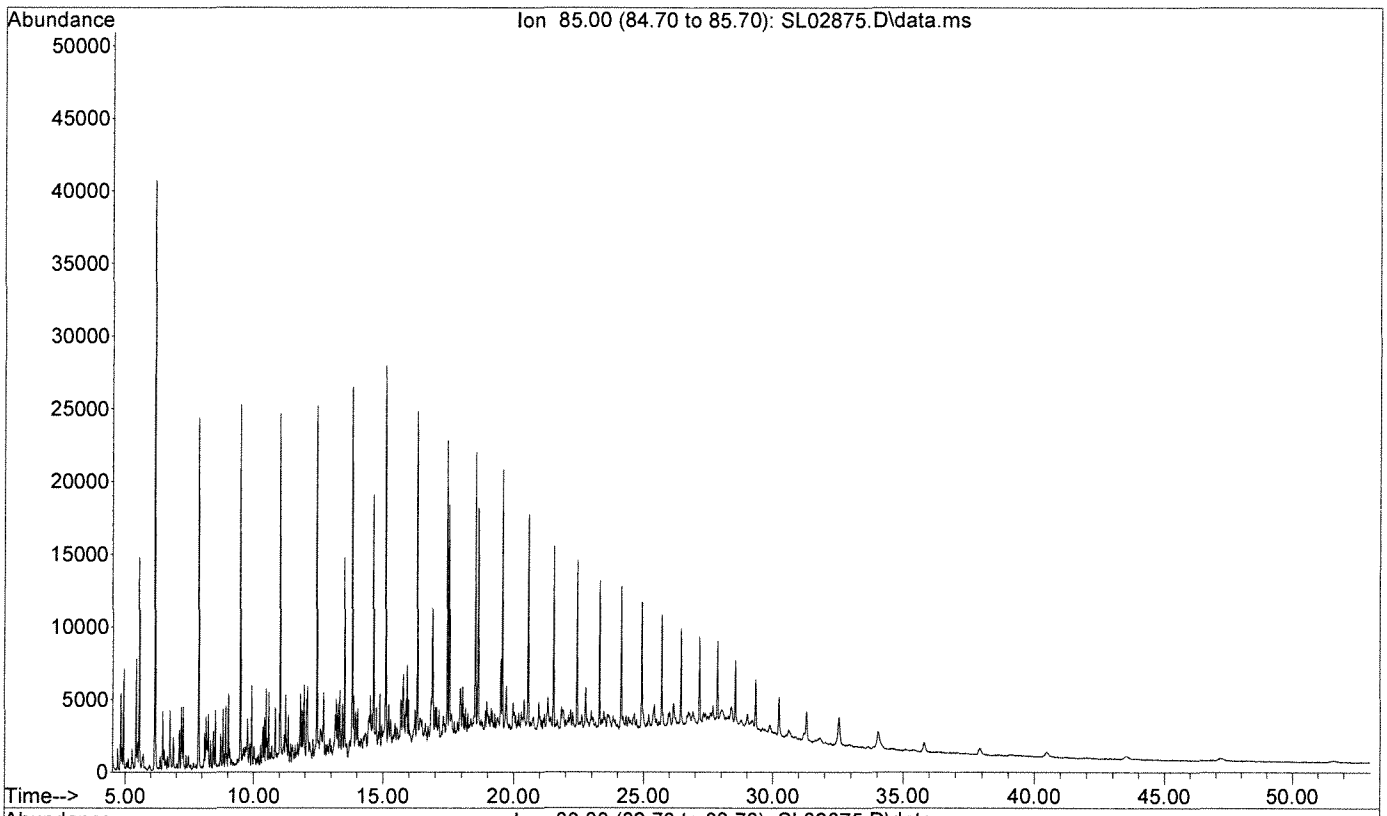


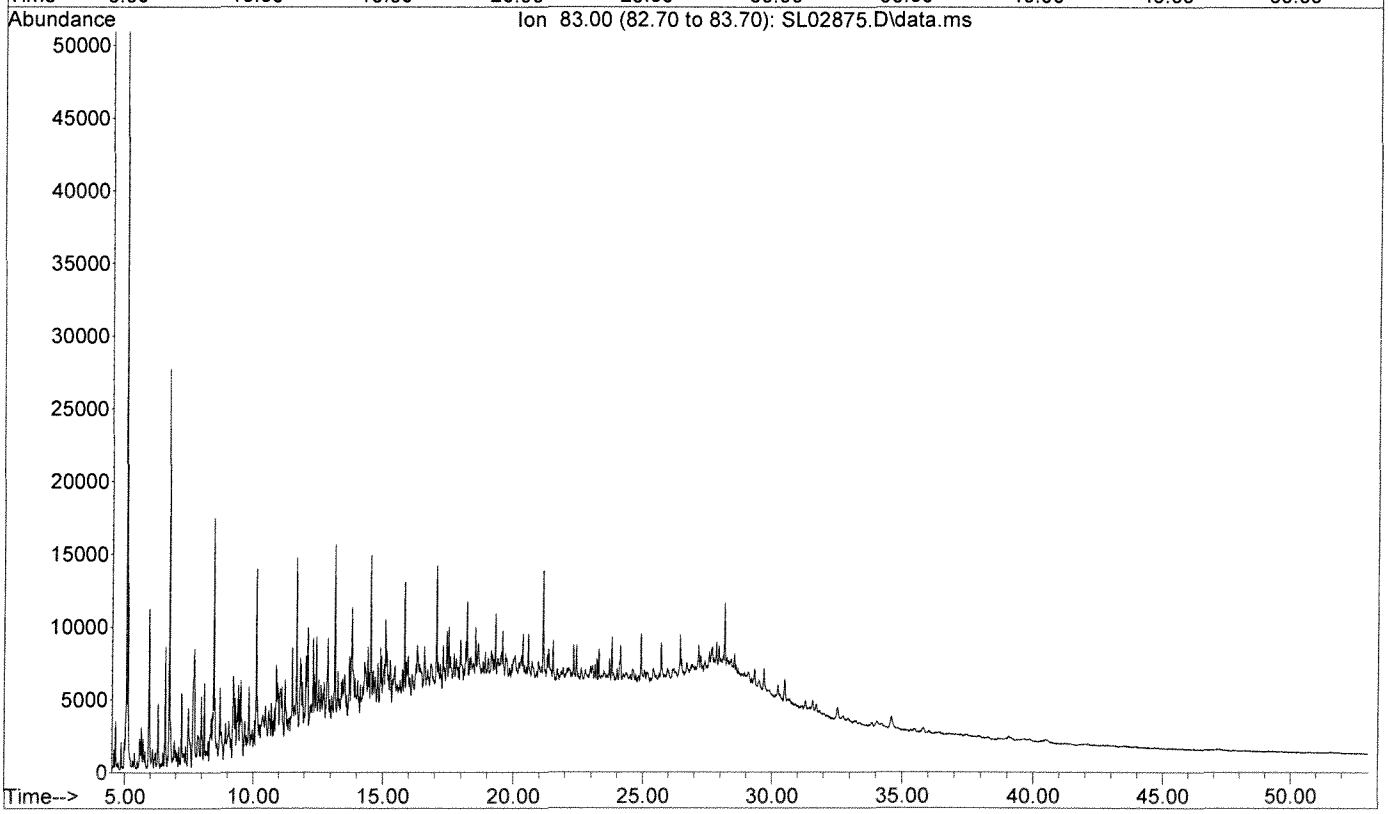
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2





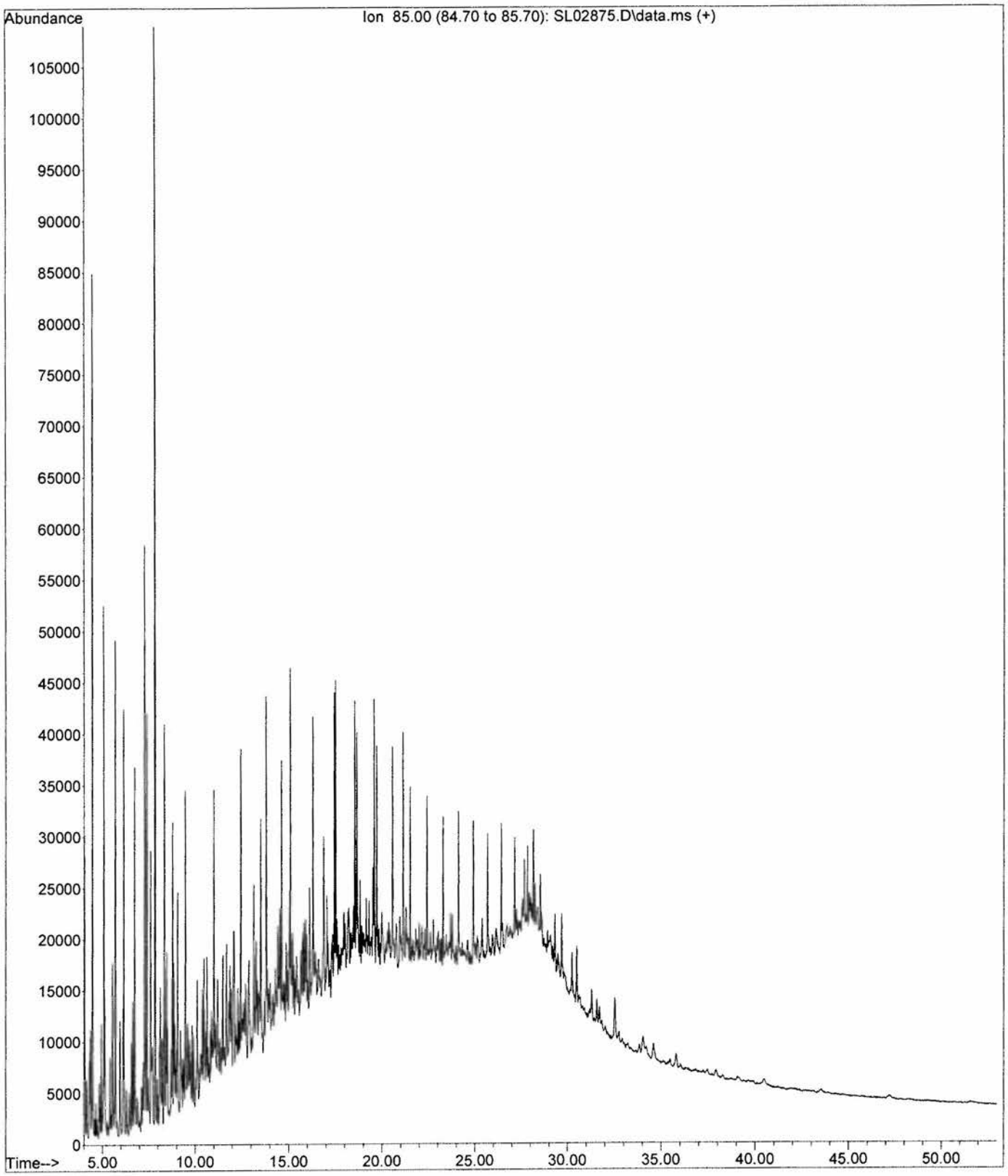
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

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Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



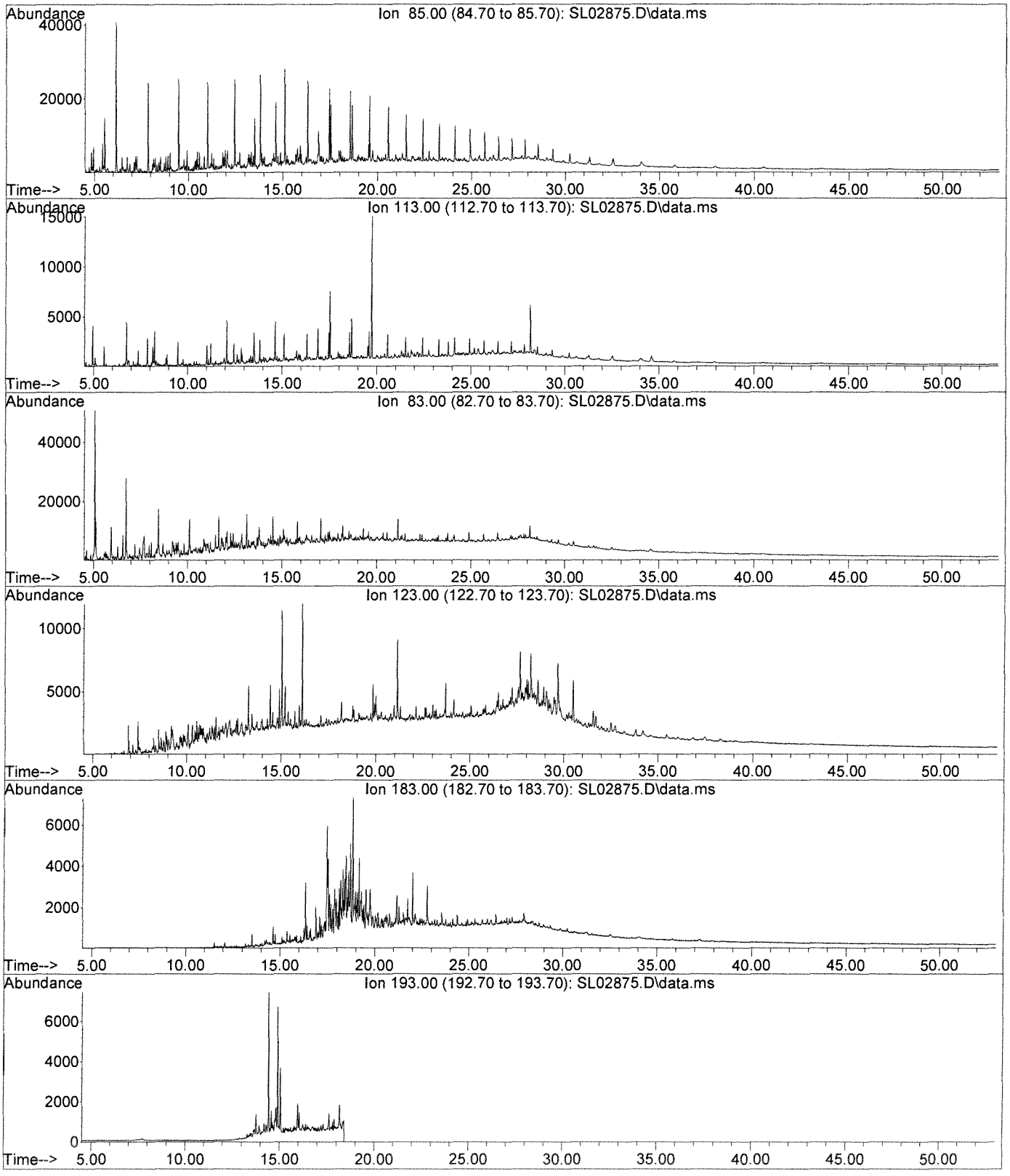
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



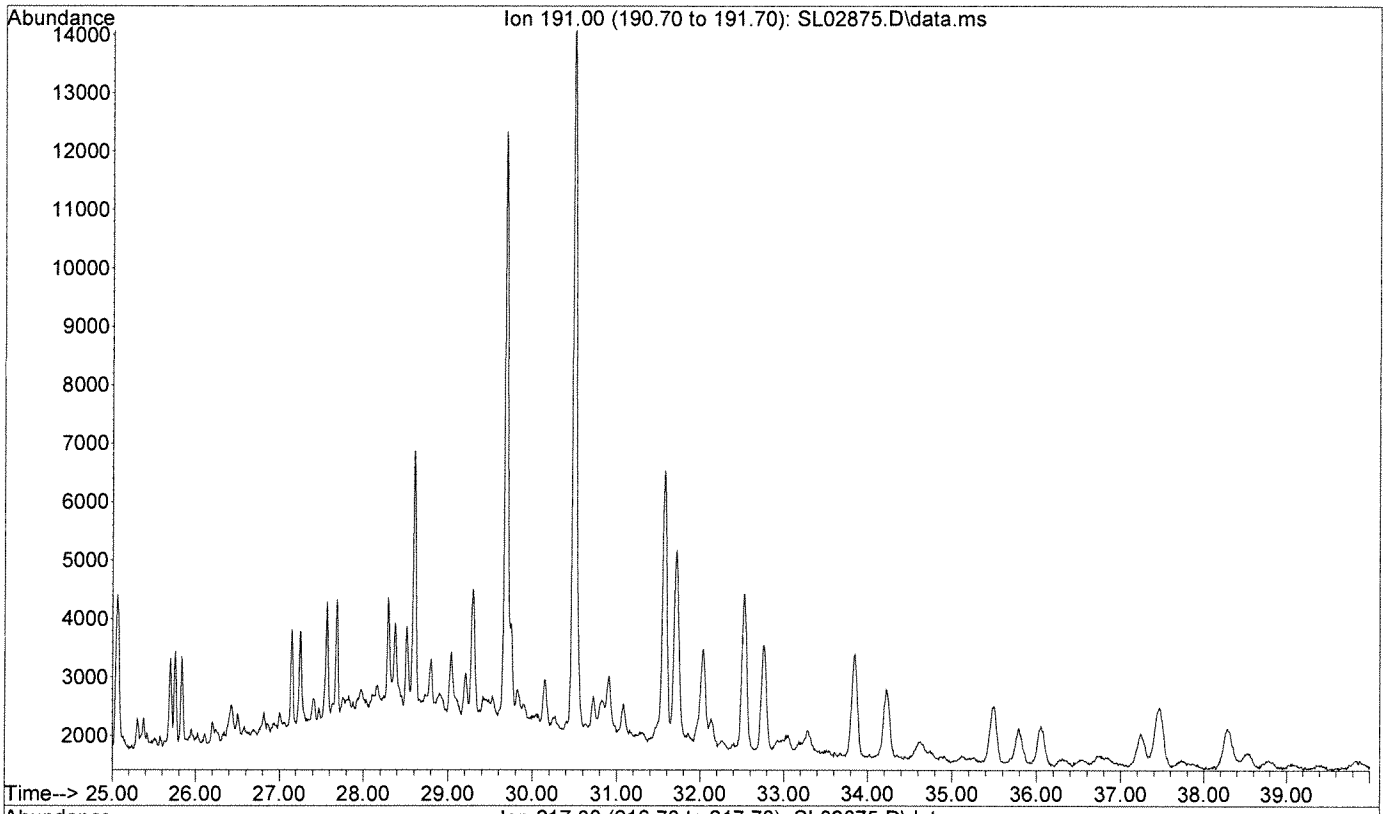
US EPA ARCHIVE DOCUMENT

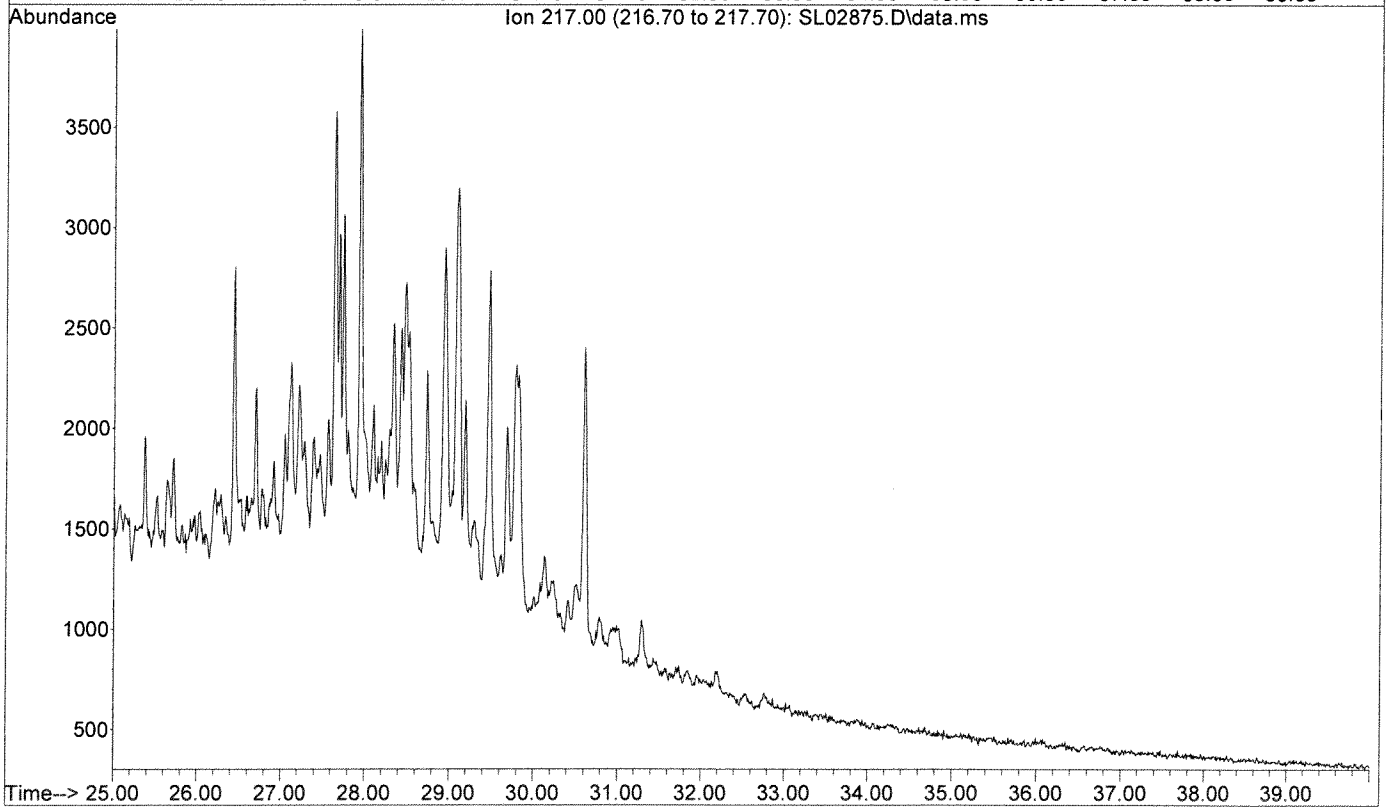
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

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Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2





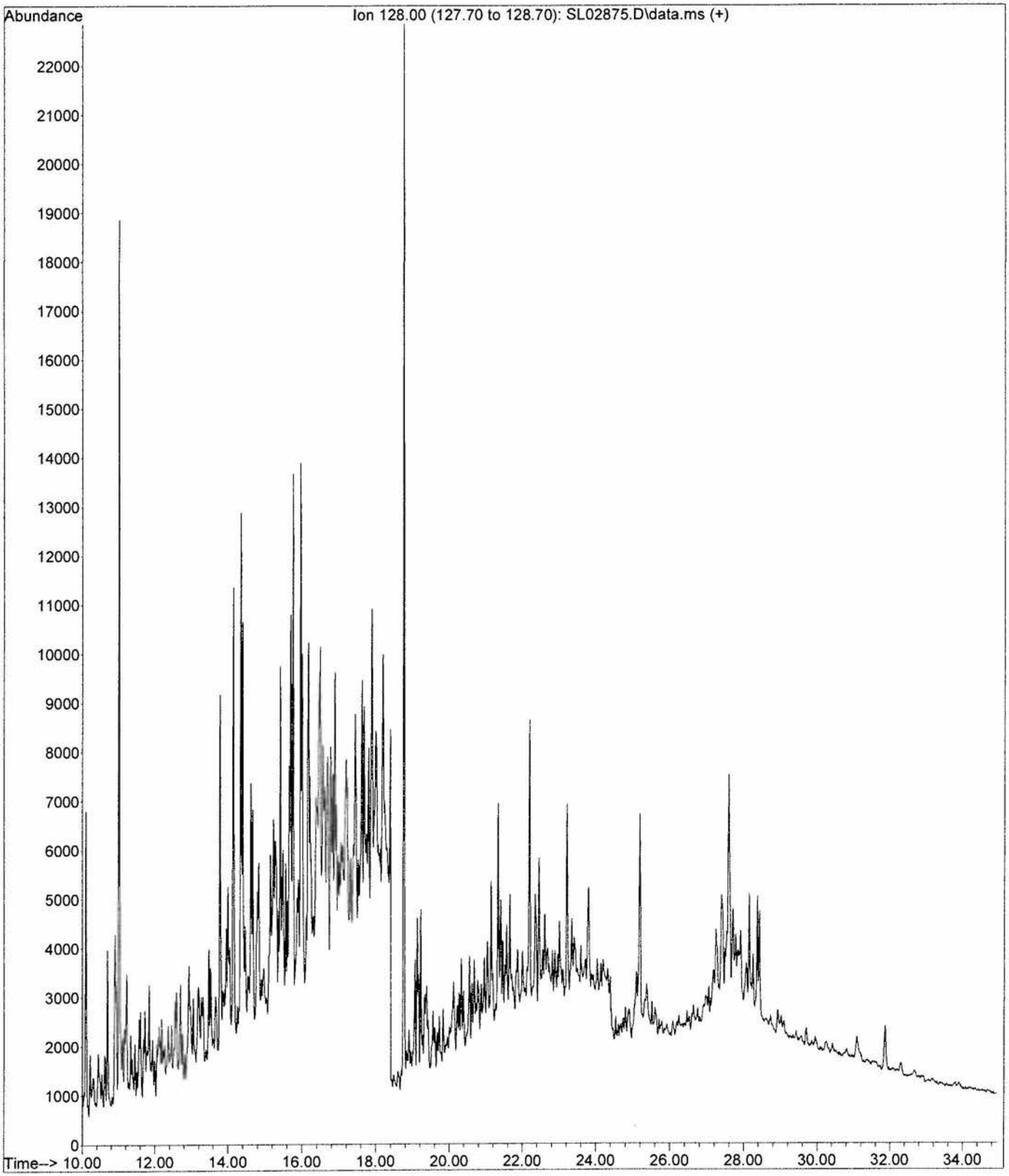
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

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Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



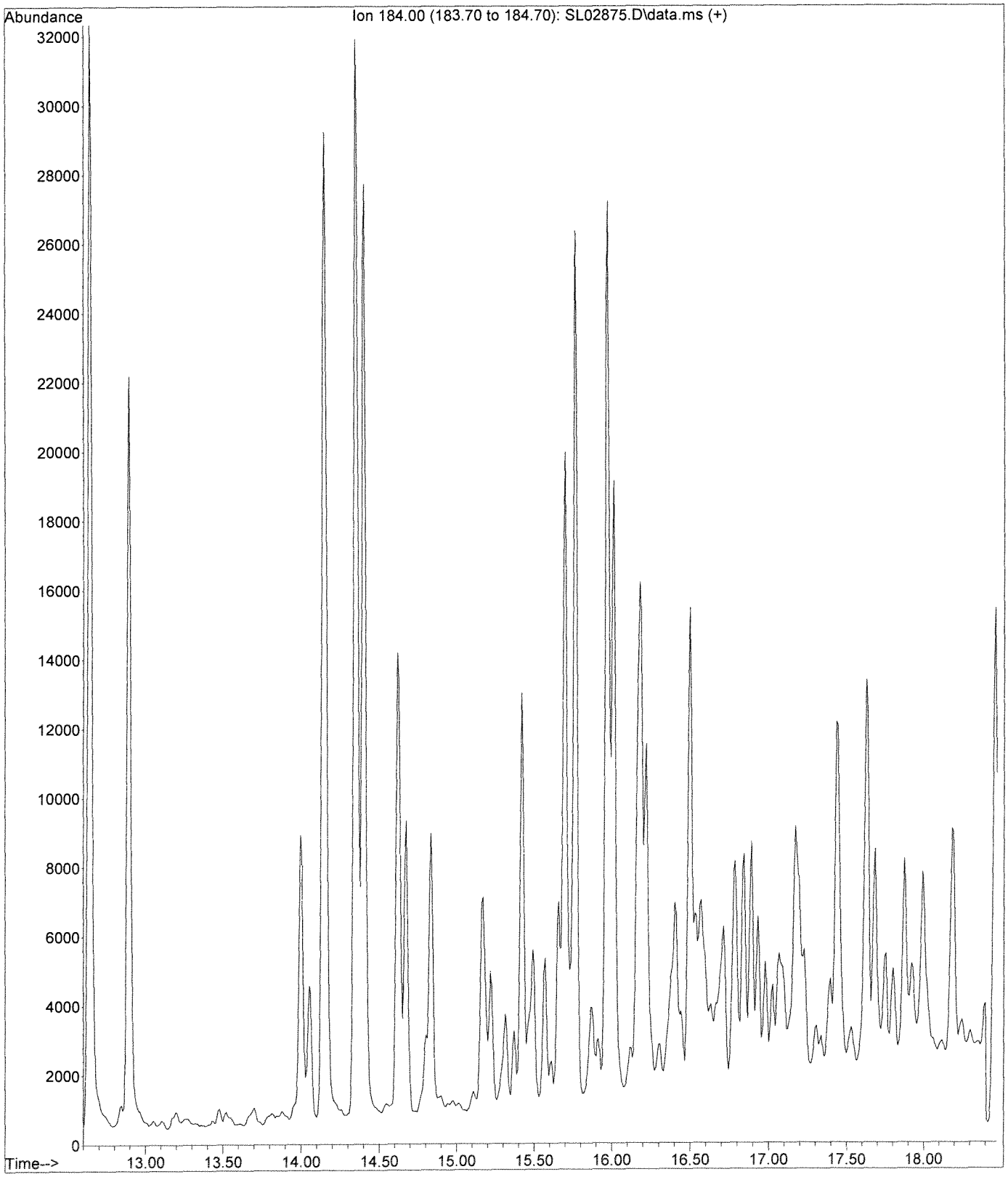
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

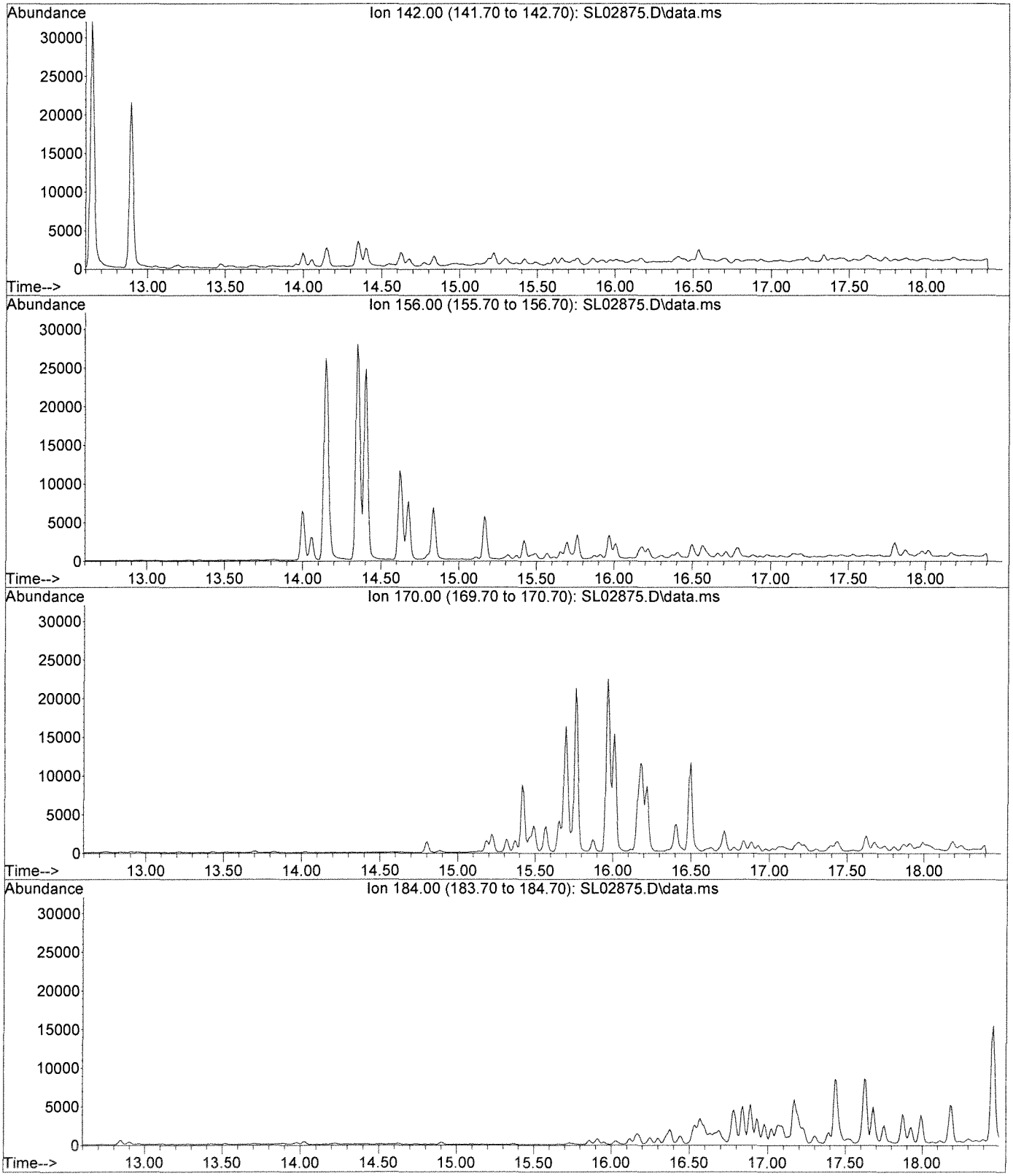
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



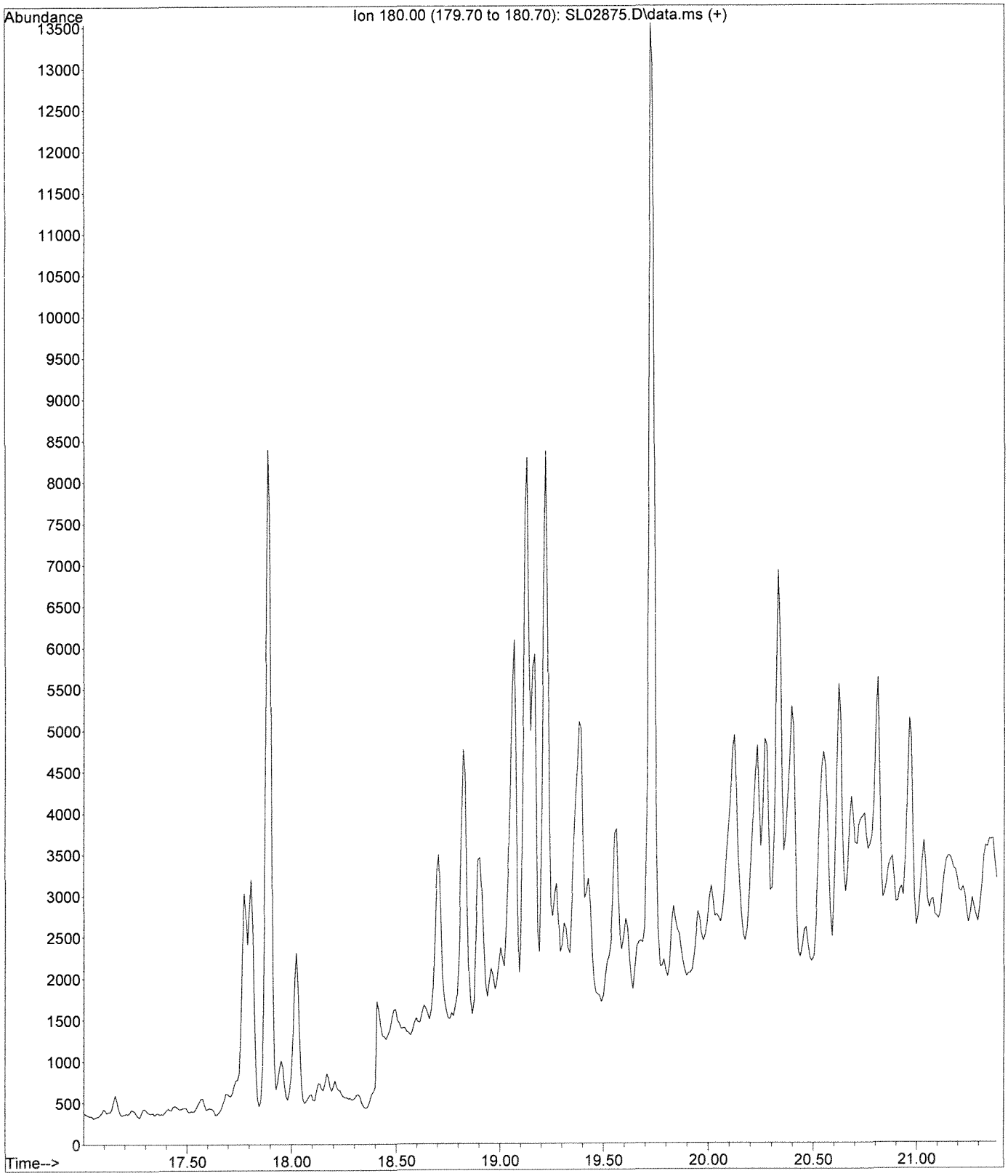
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



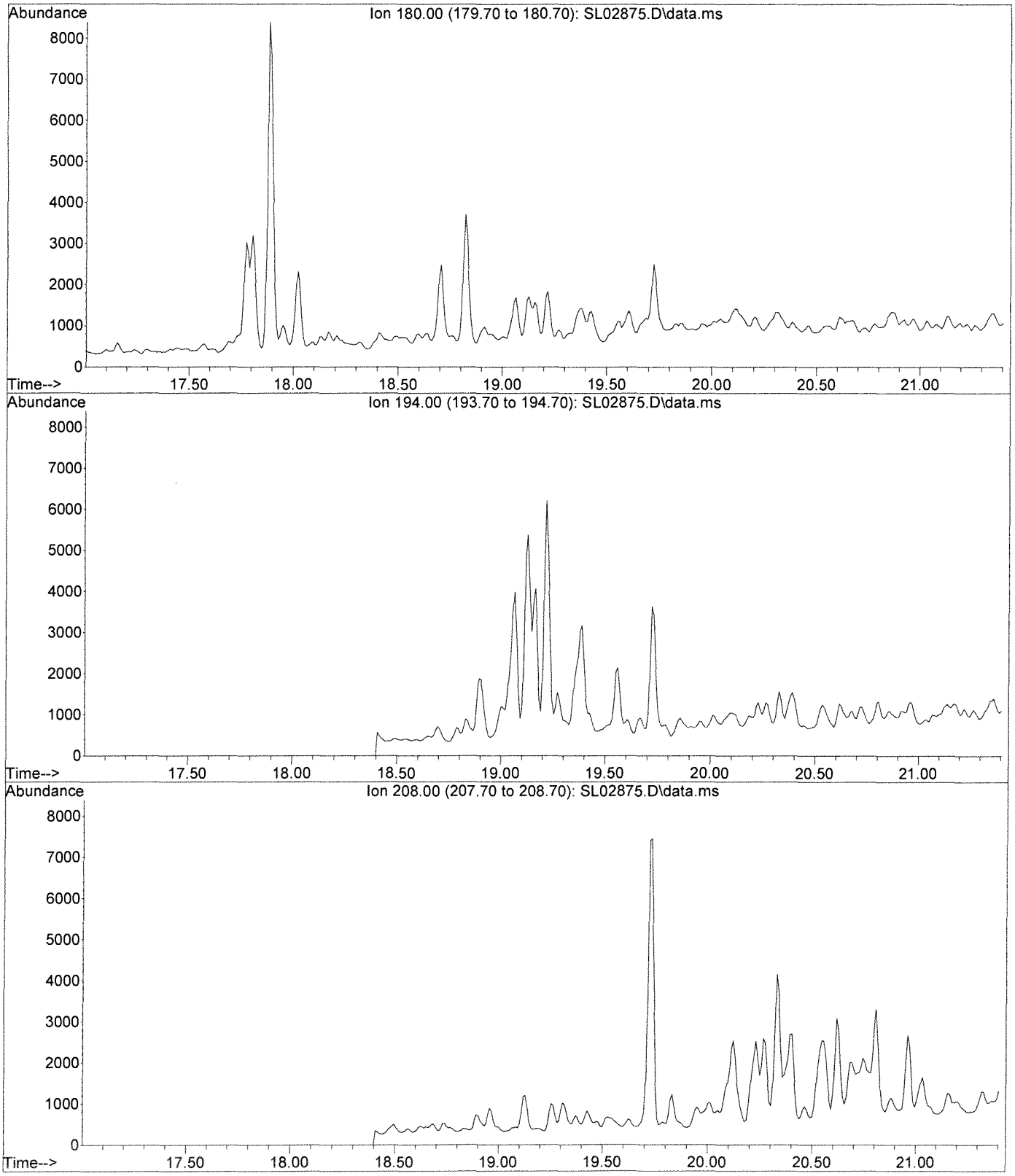
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



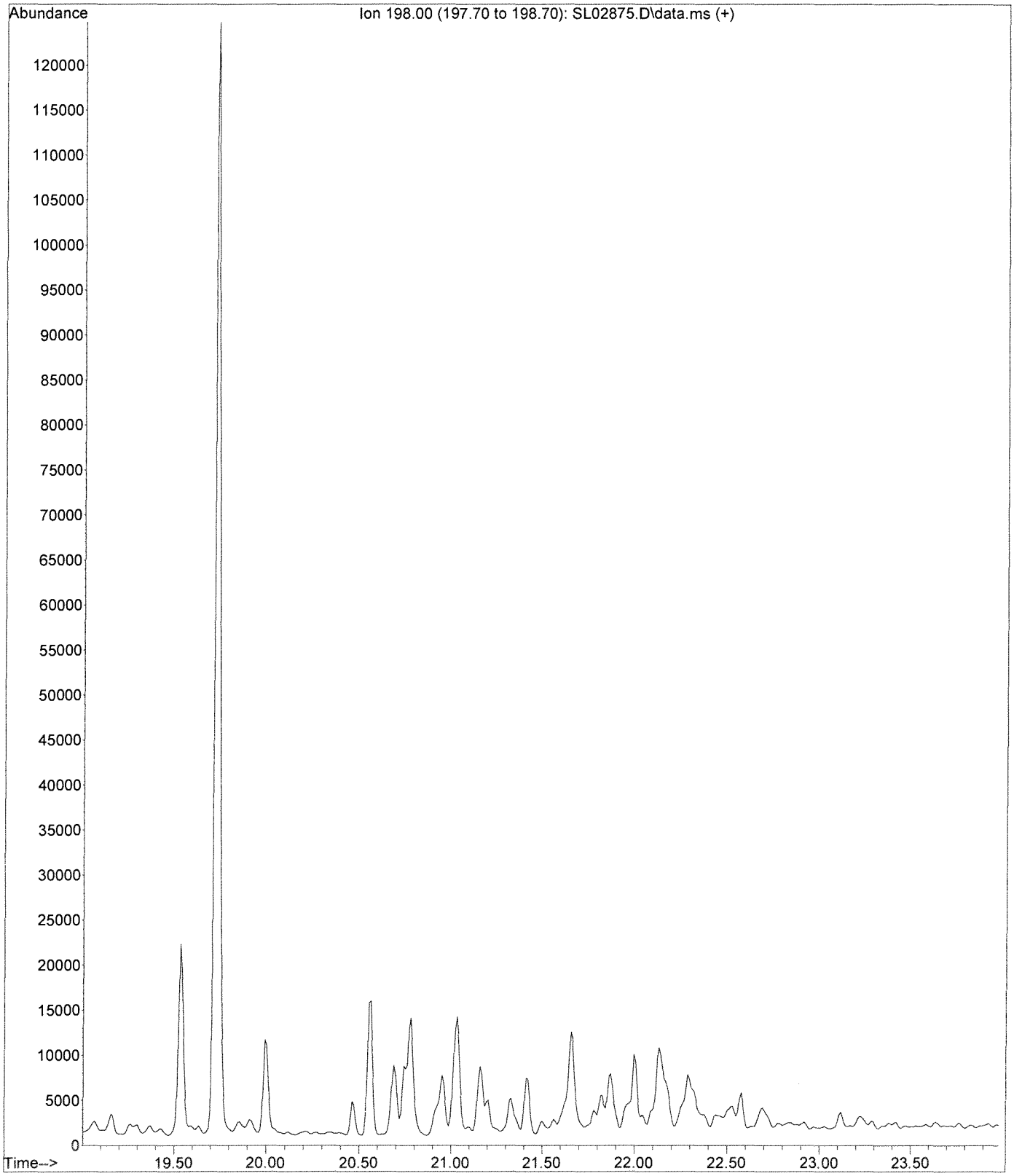
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



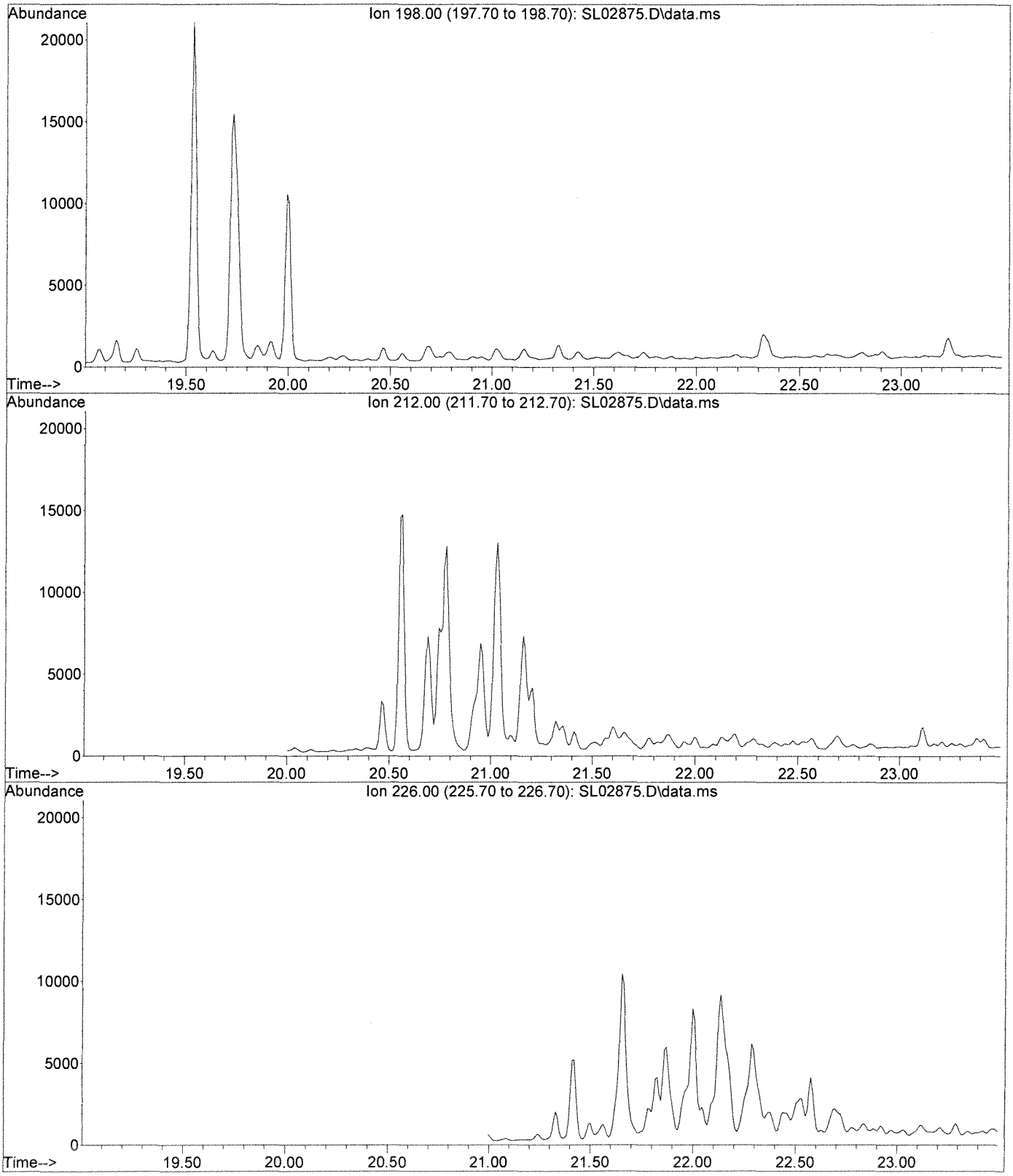
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



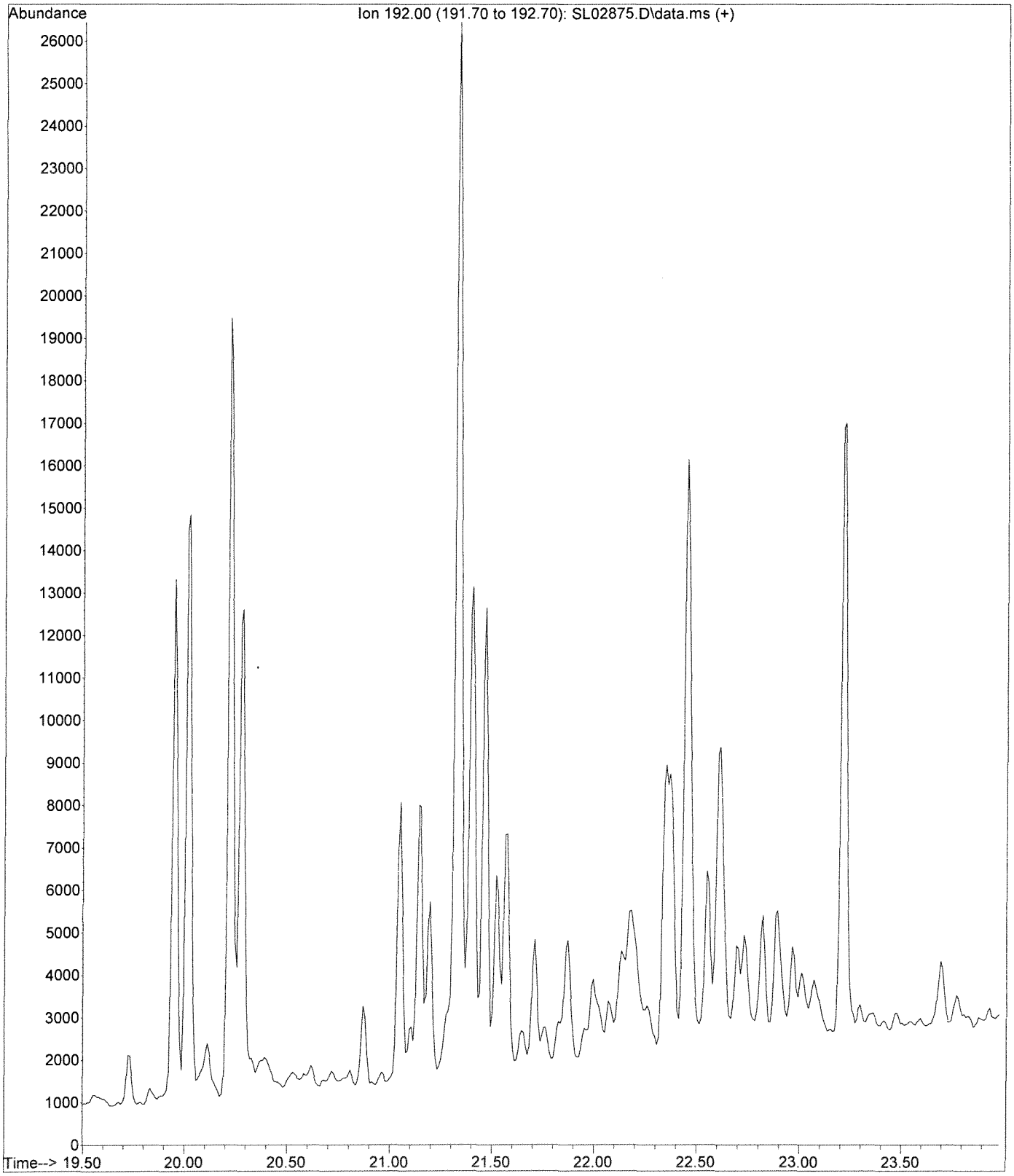
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



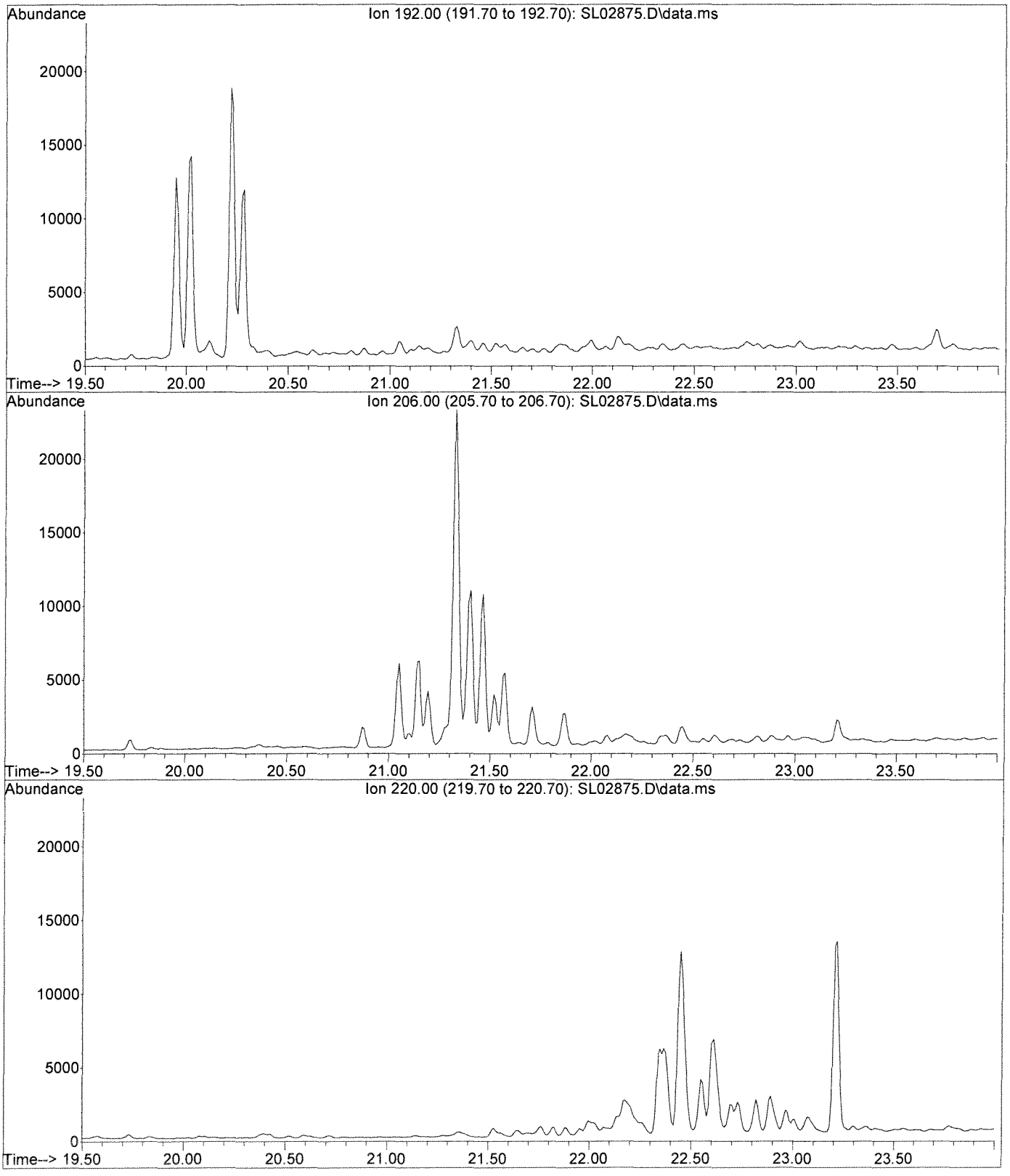
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



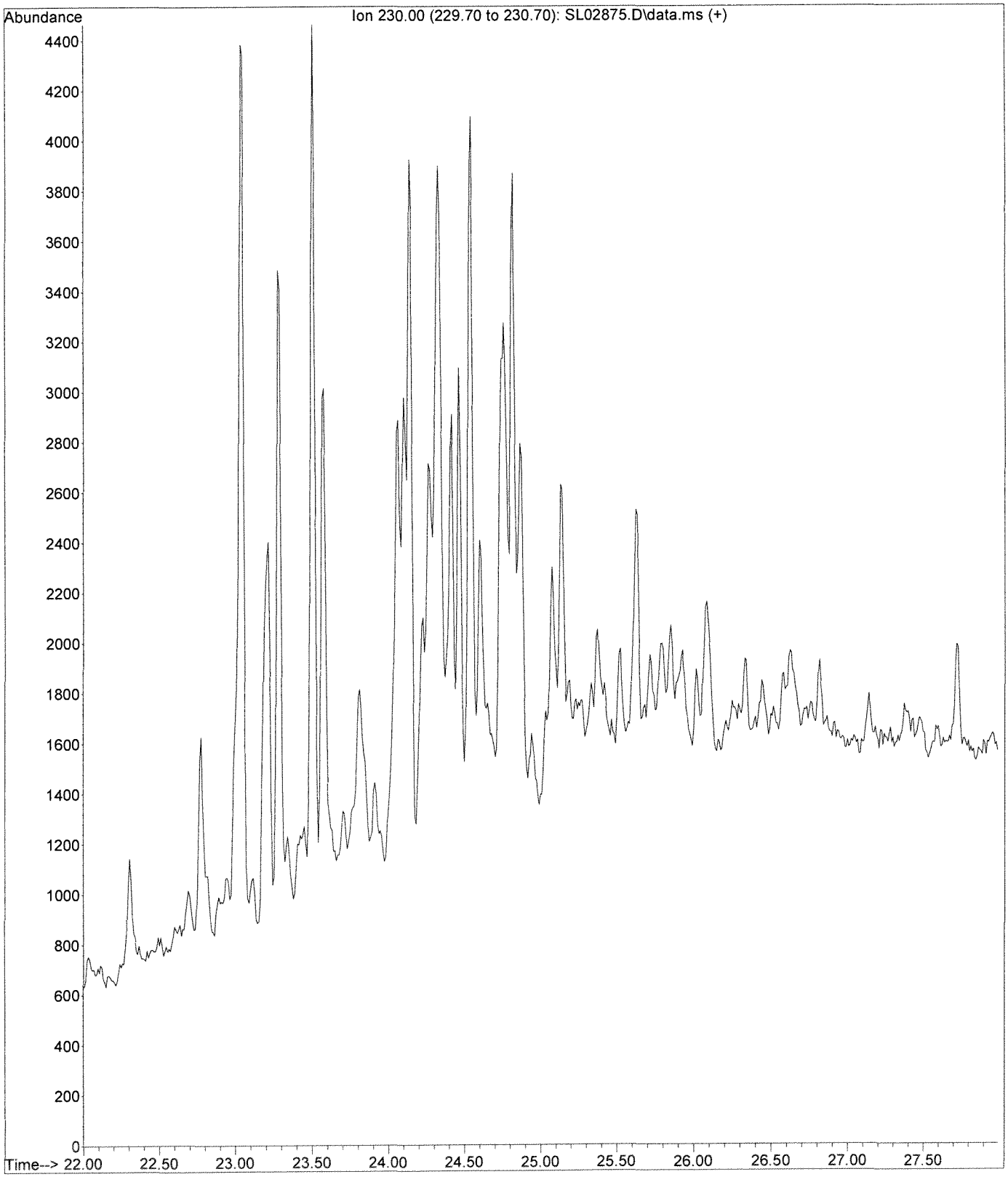
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



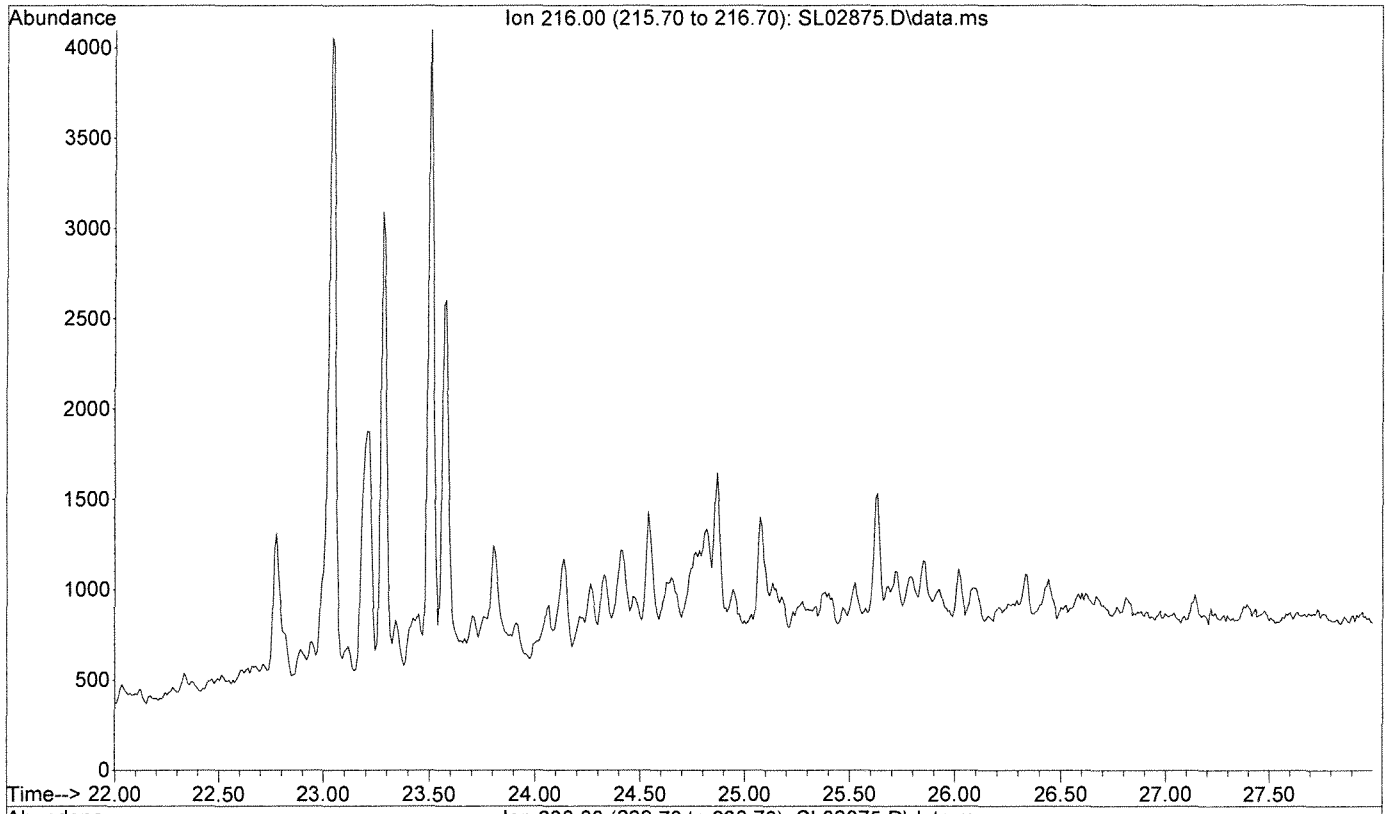
US EPA ARCHIVE DOCUMENT

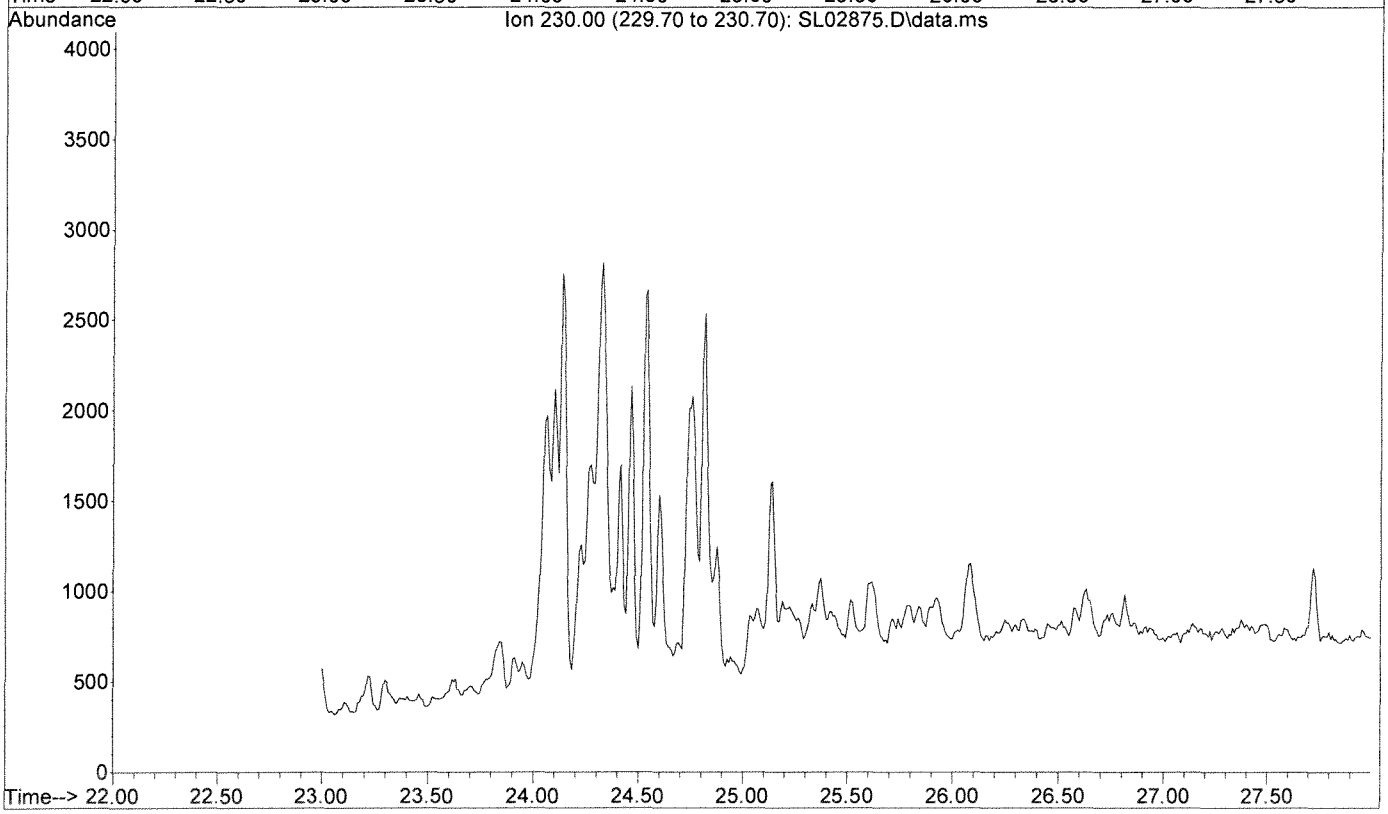
File :C:\msdchem\1\DATA\022712\SL02875.D
Operator : Syslo
Acquired : 27 Feb 2012 19:33 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003
Misc Info : 275-55-05 {0.116g in10mL}
Vial Number: 2



US EPA ARCHIVE DOCUMENT

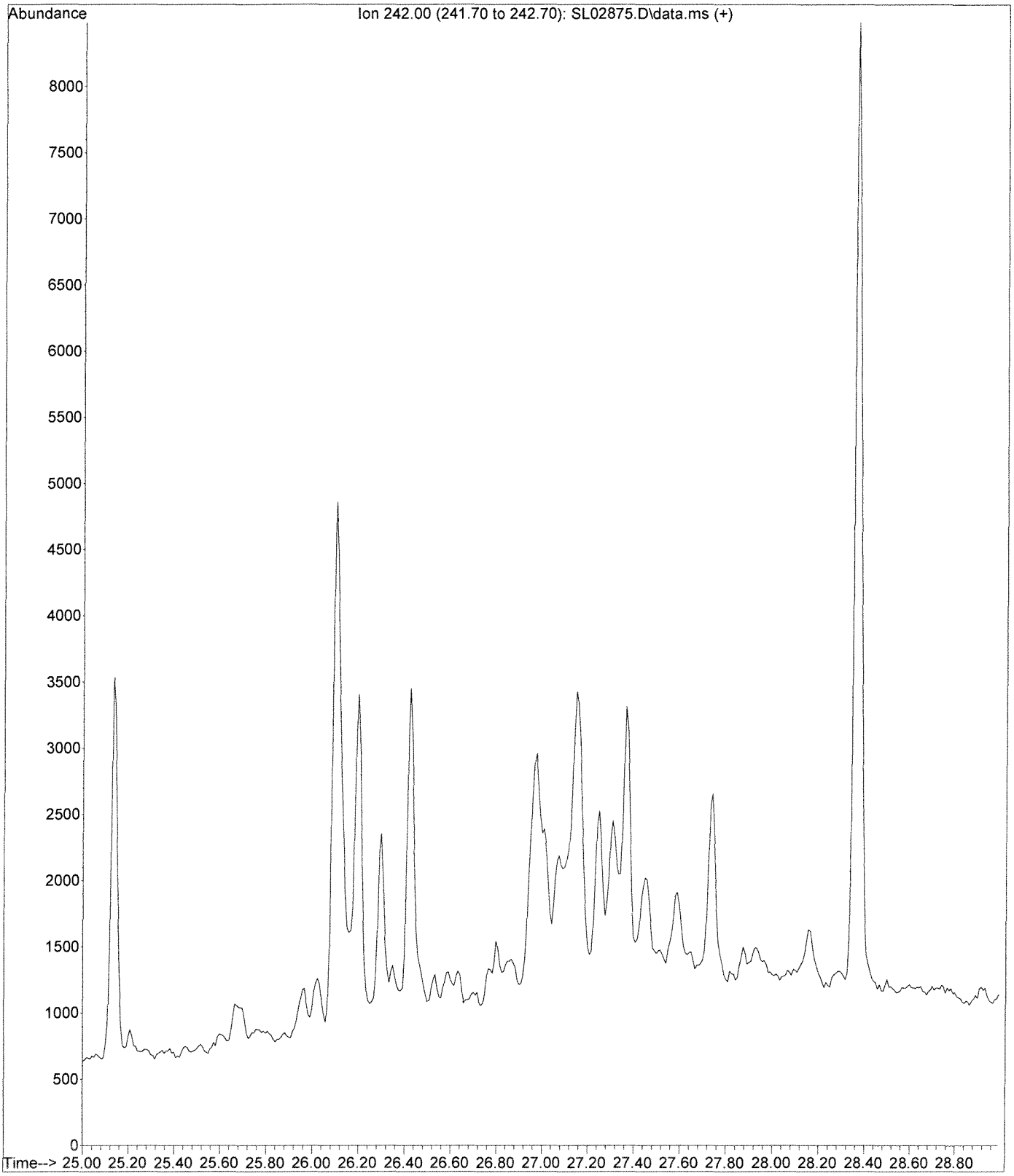
```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```





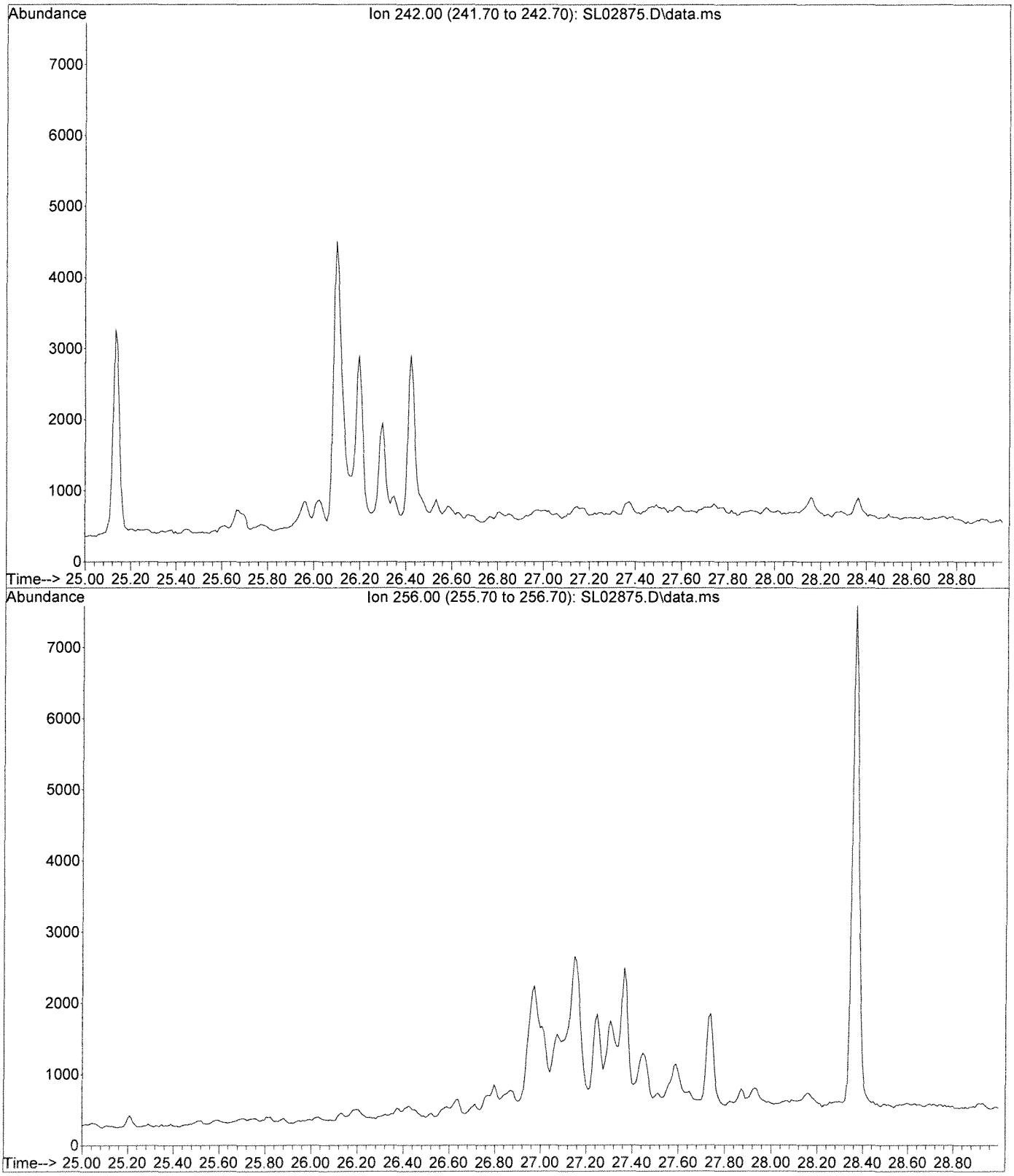
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



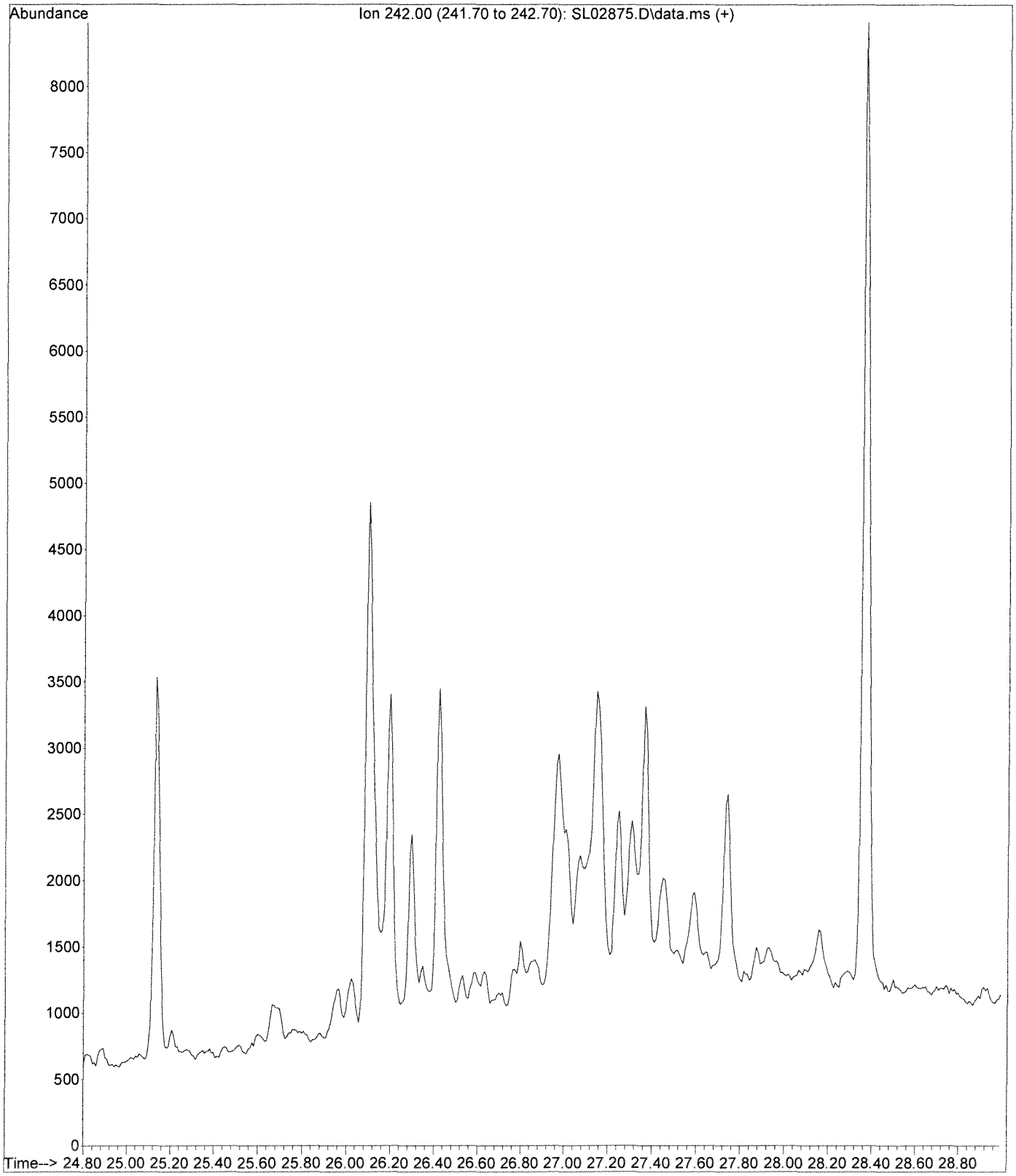
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02875.D
Operator    : Syslo
Acquired    : 27 Feb 2012  19:33      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003
Misc Info   : 275-55-05 {0.116g in10mL}
Vial Number: 2
```



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SERAS-017-0003 dup
SL028761 D

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Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02876.D
Acq On : 27 Feb 2012 20:35
Operator : Syslo
Sample : SERAS-017-0003 dup
Misc : 275-55-05 {0.111gi n10mL}
ALS Vial : 3 Sample Multiplier: 1

Quant Time: Mar 01 13:06:28 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	319905	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	103998	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	130631	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	119796	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.822	188	544414	16.00	ug/mL	0.00
Spiked Amount 20.000			Recovery =	80.00%		
3) d14-o-Terphenyl {S}	19.733	244	261953	17.52	ug/mL	0.00
Spiked Amount 20.000			Recovery =	87.60%		
4) 5a-Androstane {S}	21.140	260	34508	18.97	ug/mL	0.00
Spiked Amount 20.000			Recovery =	94.85%		
7) d62-Triacontane {S}	28.156	66	235375	20.37	ug/mL	0.00
Spiked Amount 20.000			Recovery =	101.85%		
Target Compounds						Qvalue

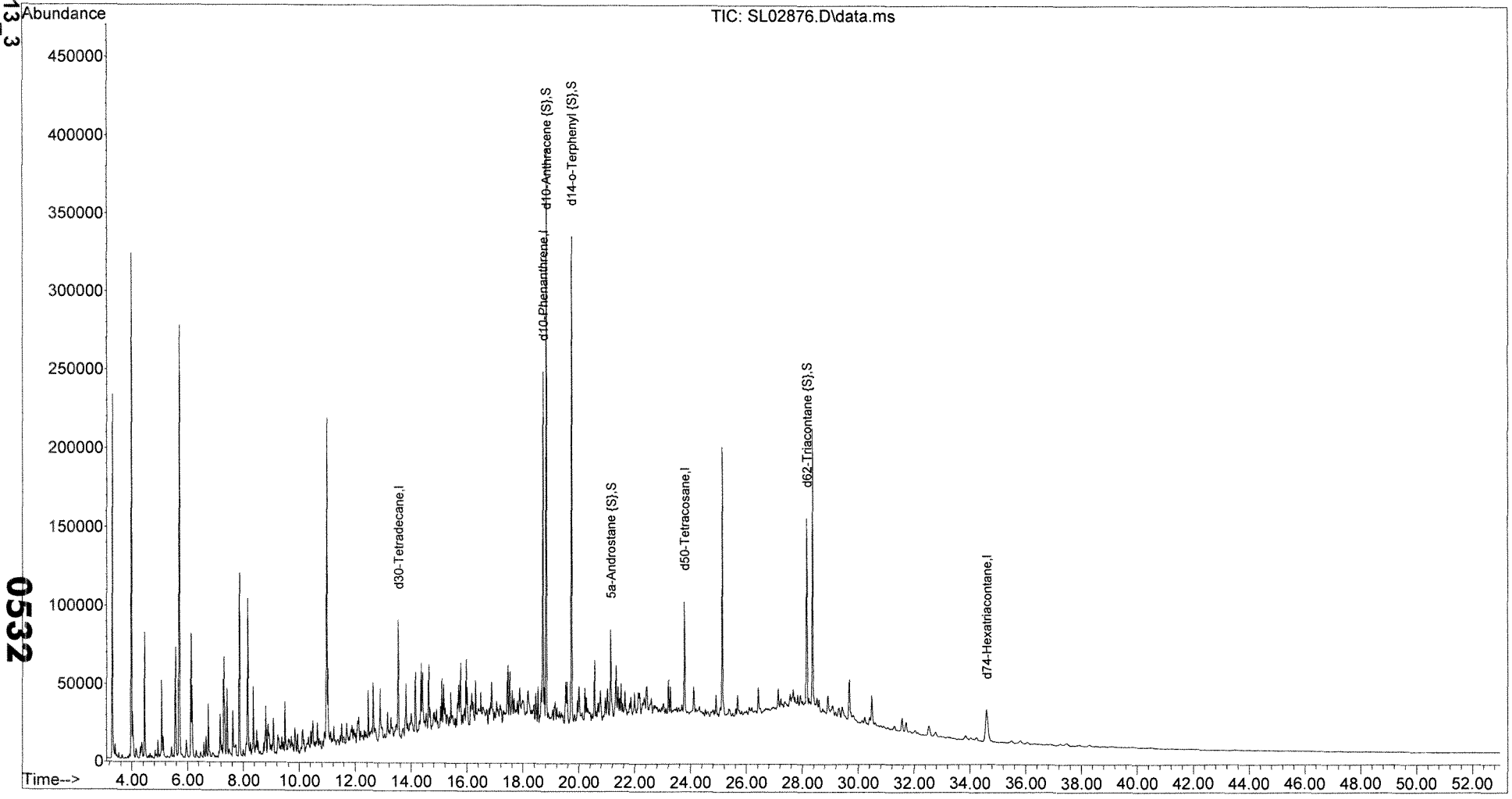
(#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Data Pa : C:\msdchem\1\DATA\022712\
Data File : SL02876.D
Acq On : 27 Feb 2012 20:35
Operator : Syslo
Sample : SERAS-017-0003 dup
Misc : 275-55-05 {0.111gi n10mL}
ALS Vial : 3 Sample Multiplier: 1

Quant Time: Mar 01 13:06:28 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0532

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

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Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM.* Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

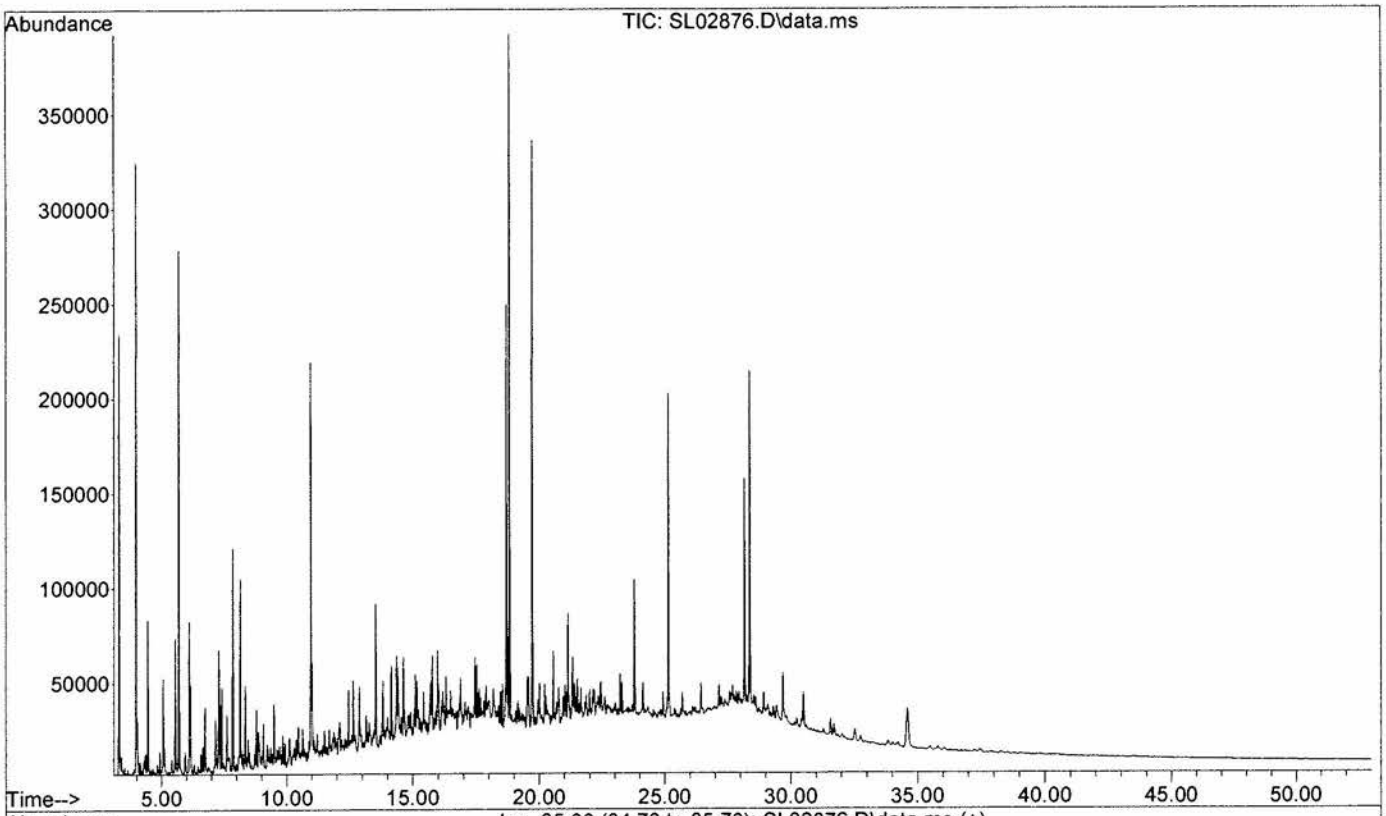
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

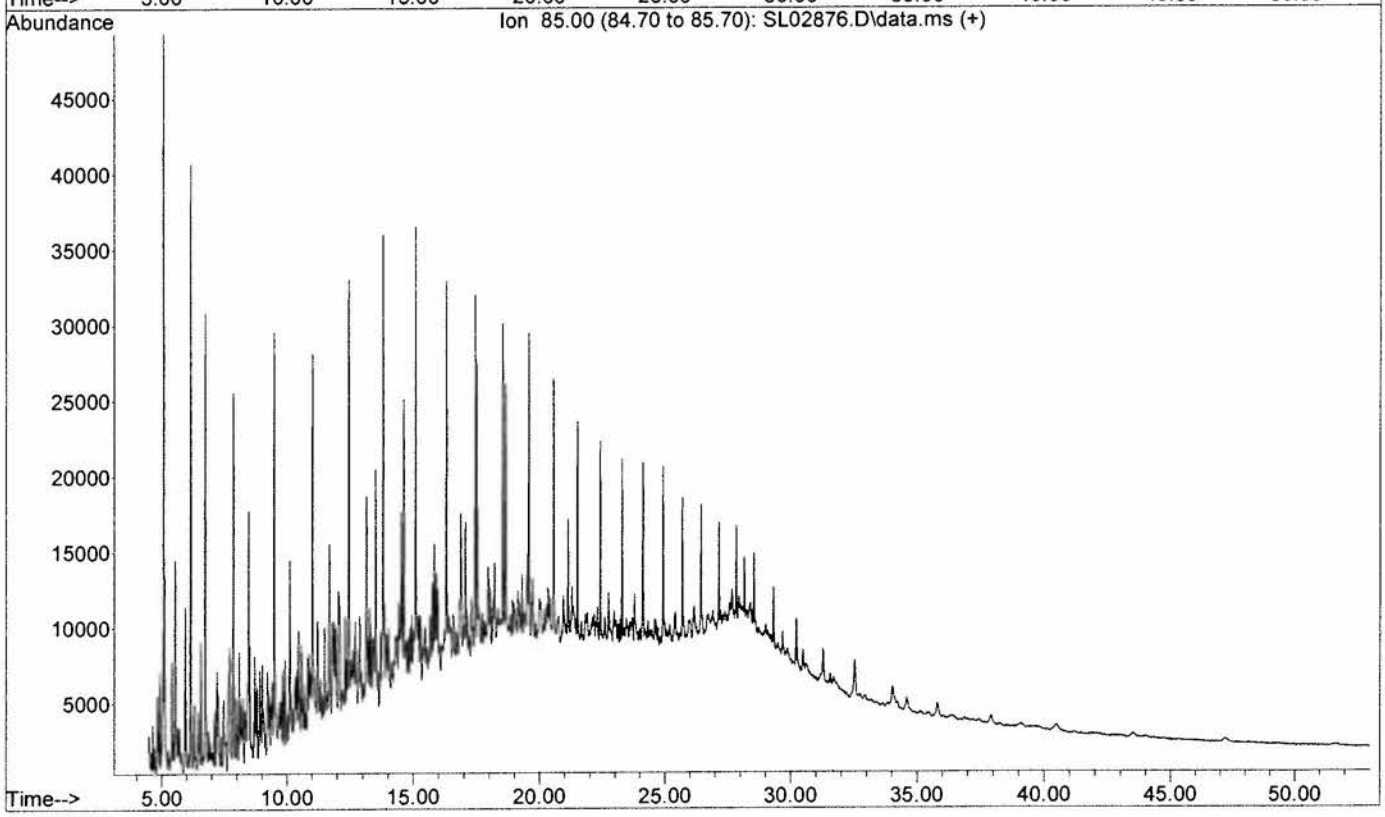
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



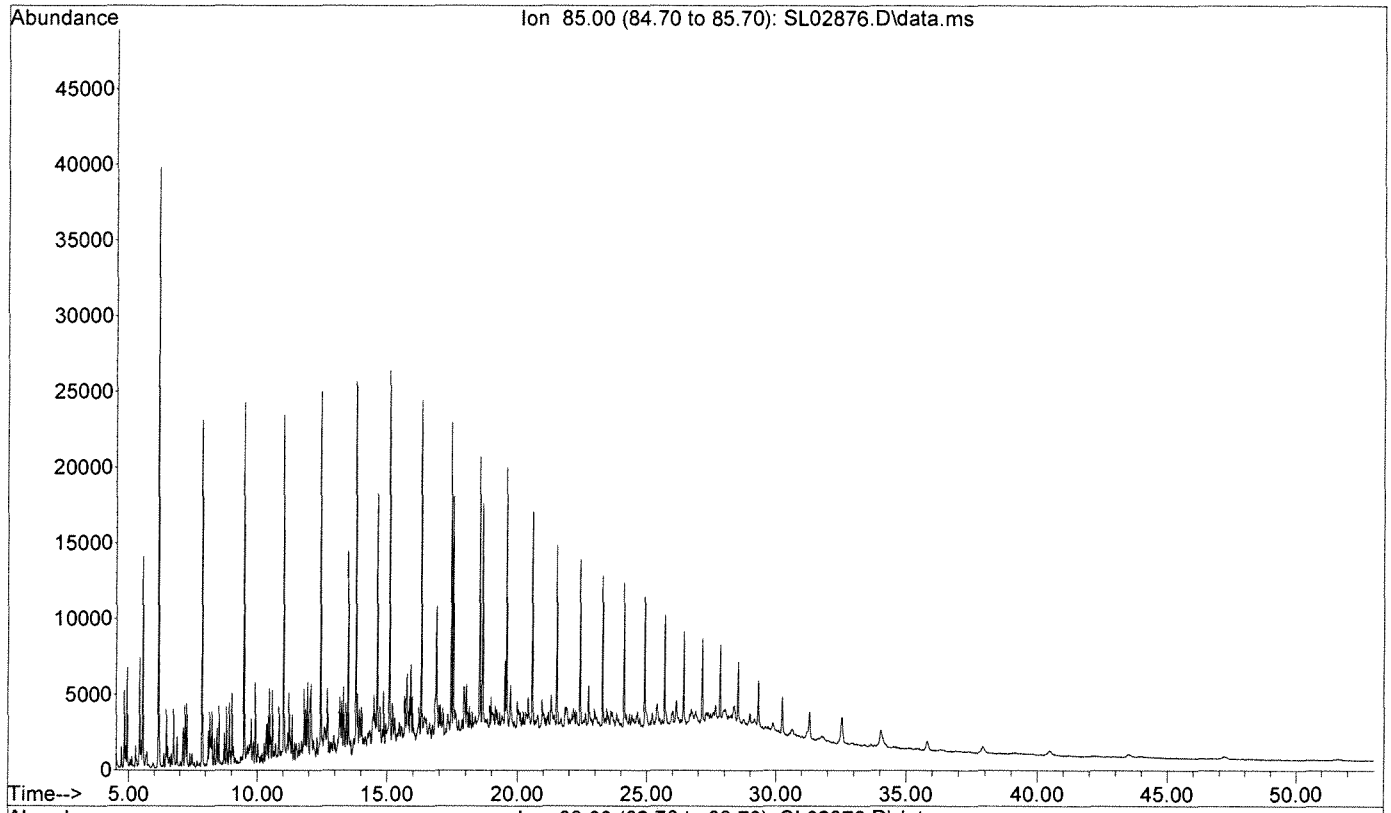


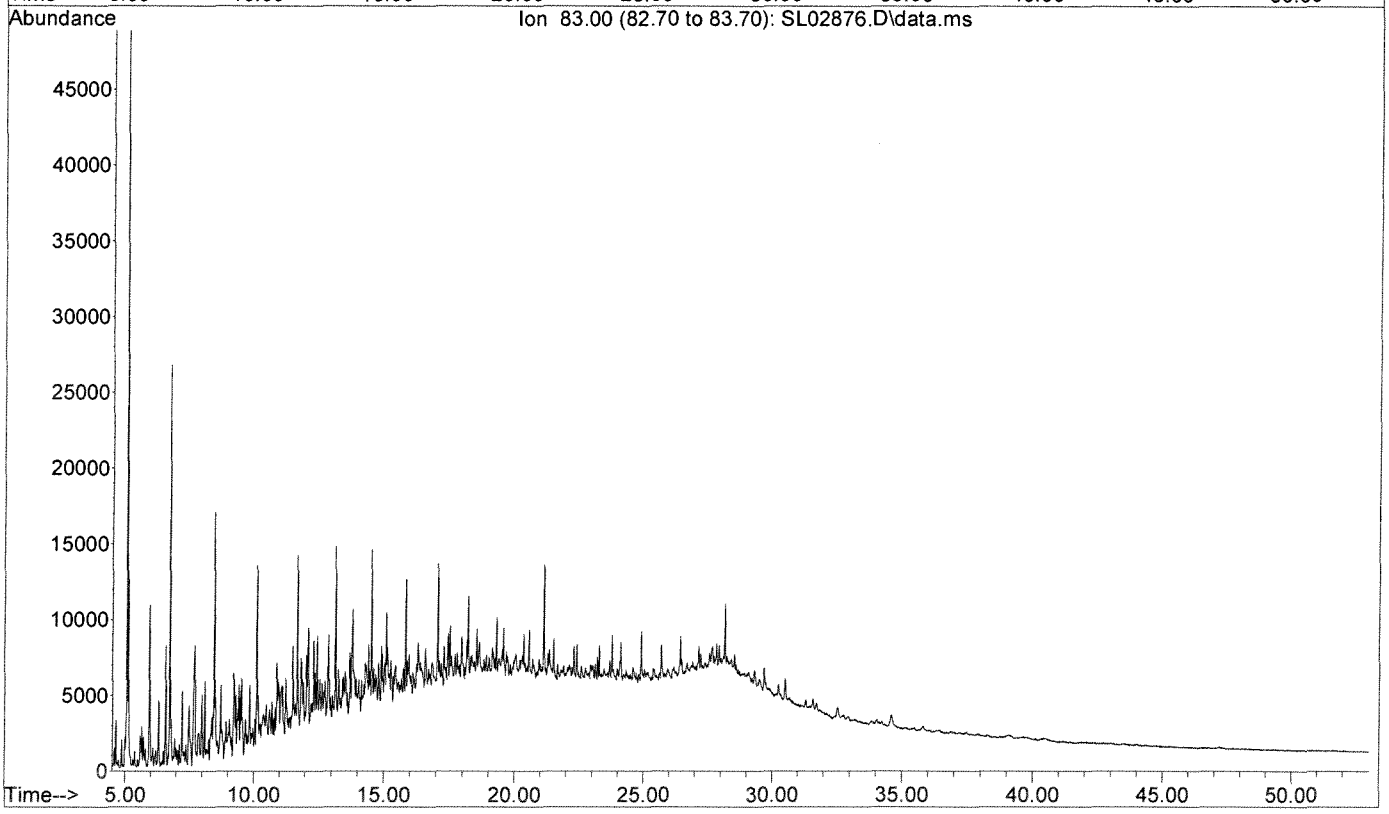
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```





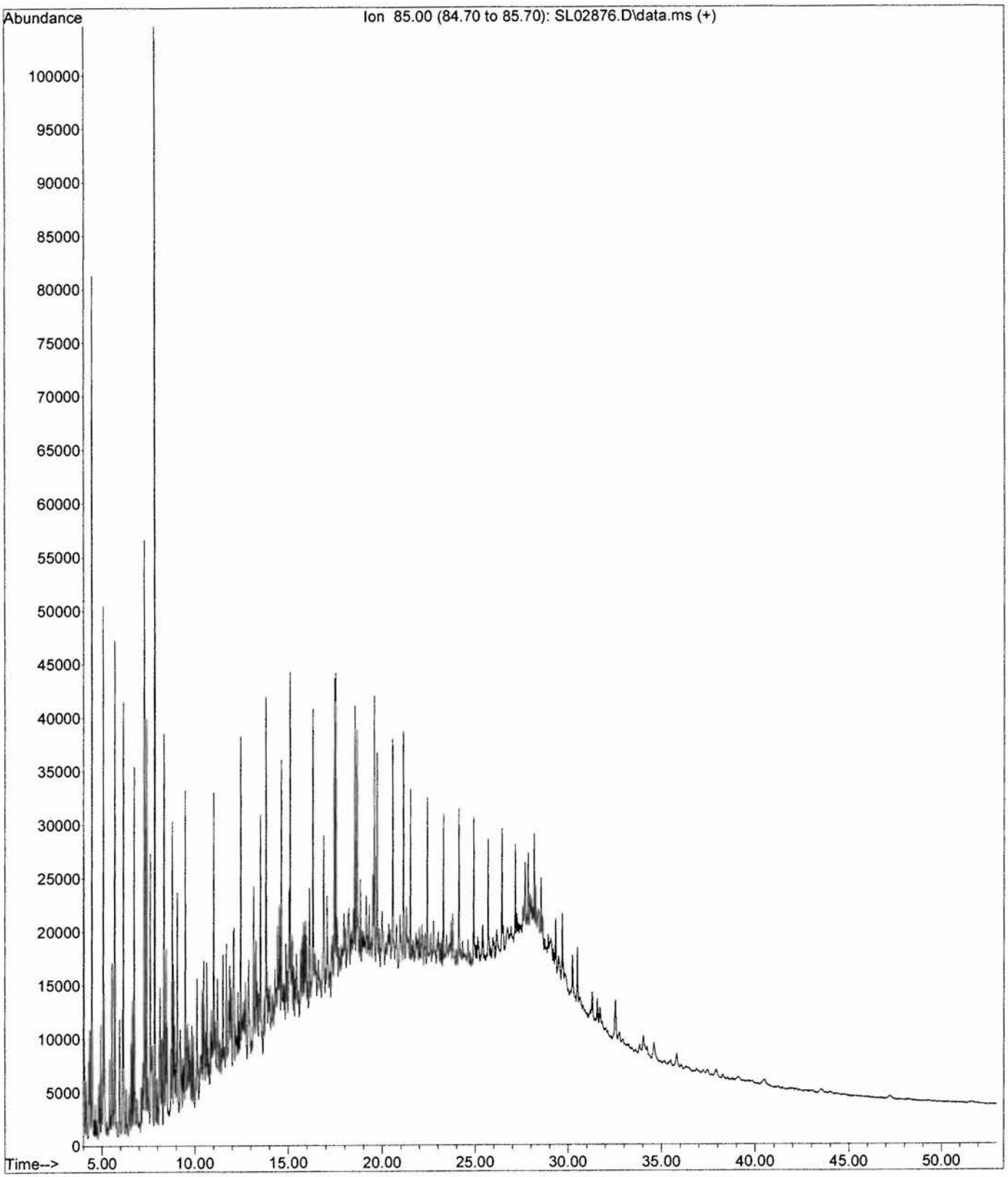
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



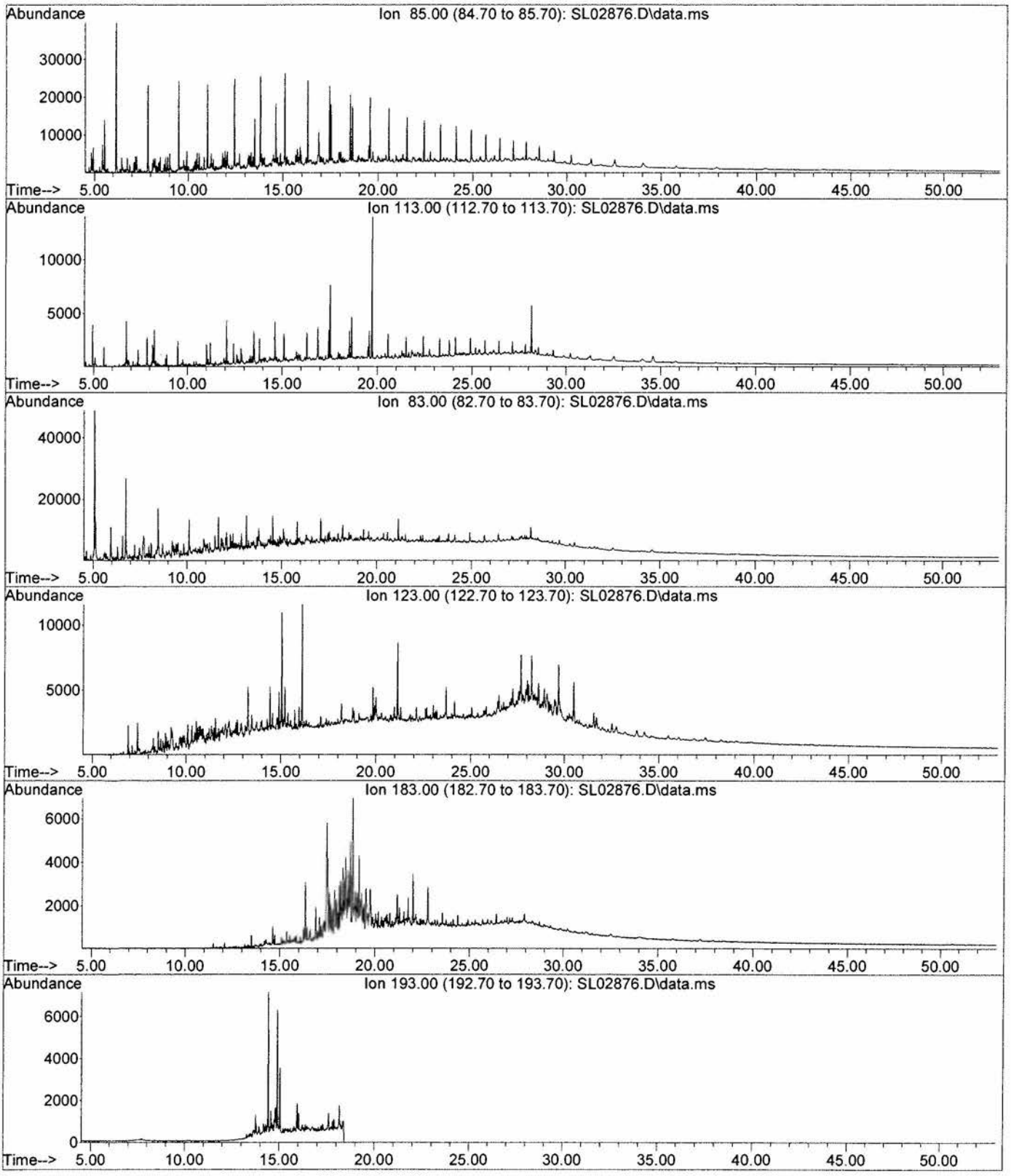
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



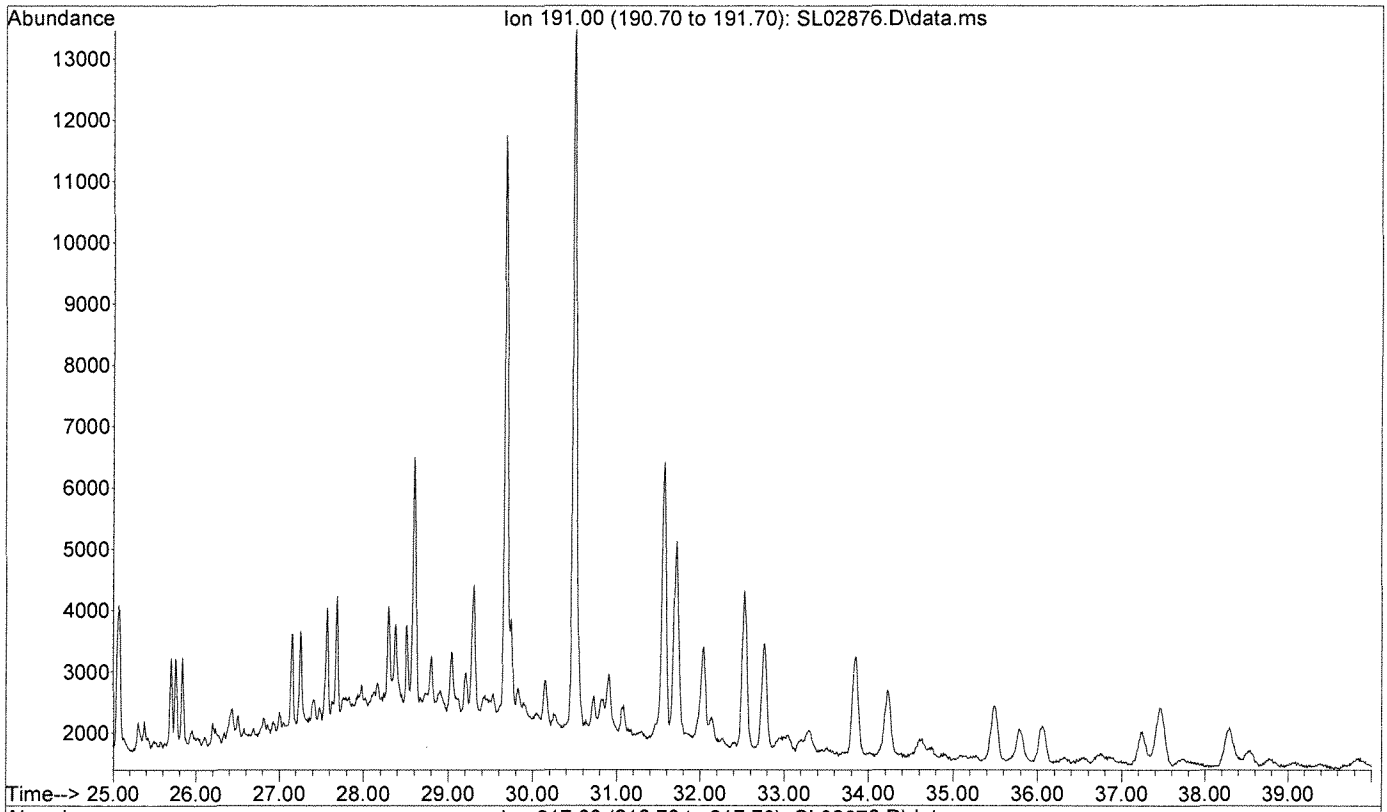
US EPA ARCHIVE DOCUMENT

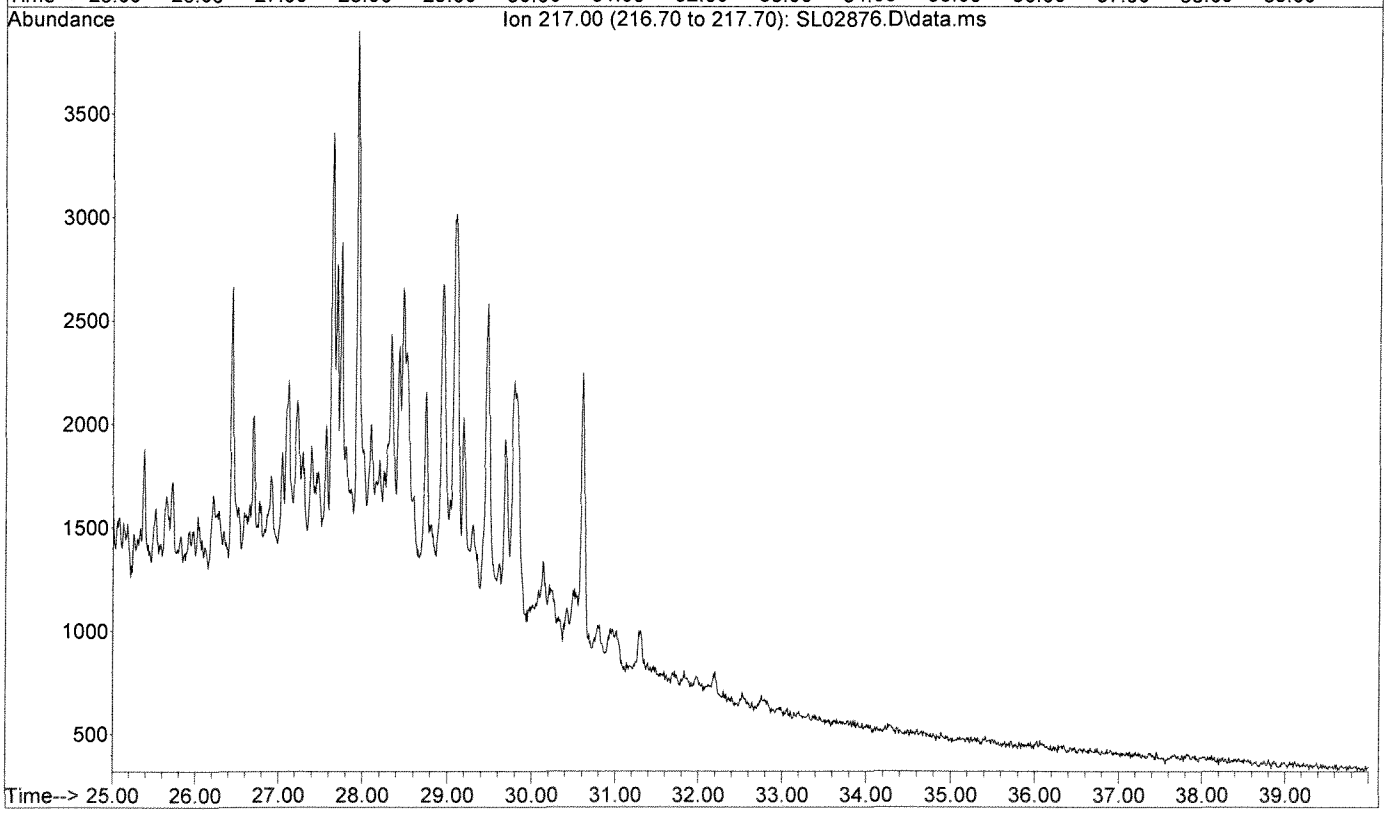
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```





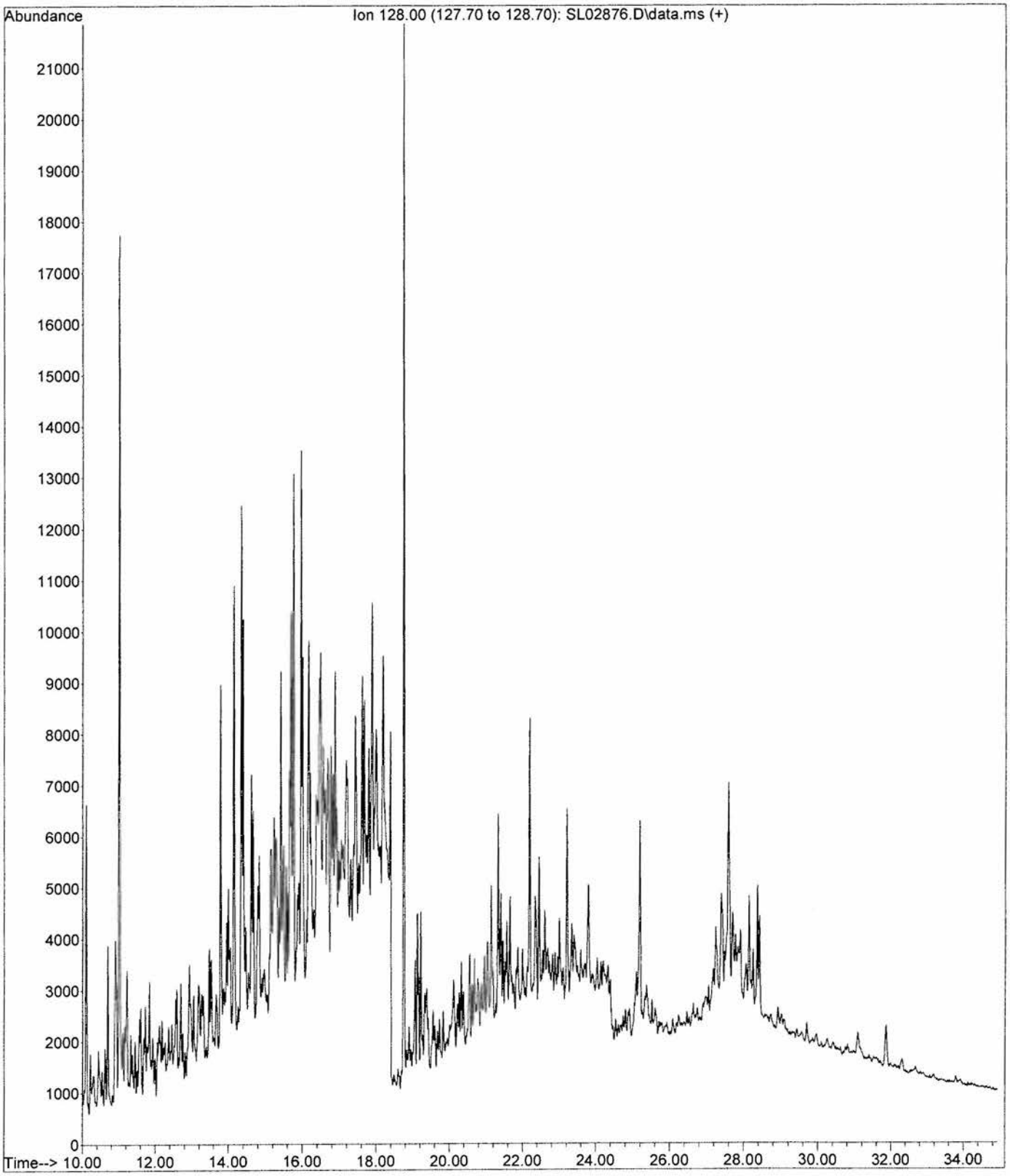
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02876.D
Operator : Syslo
Acquired : 27 Feb 2012 20:35 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 dup
Misc Info : 275-55-05 {0.111gi n10mL}
Vial Number: 3



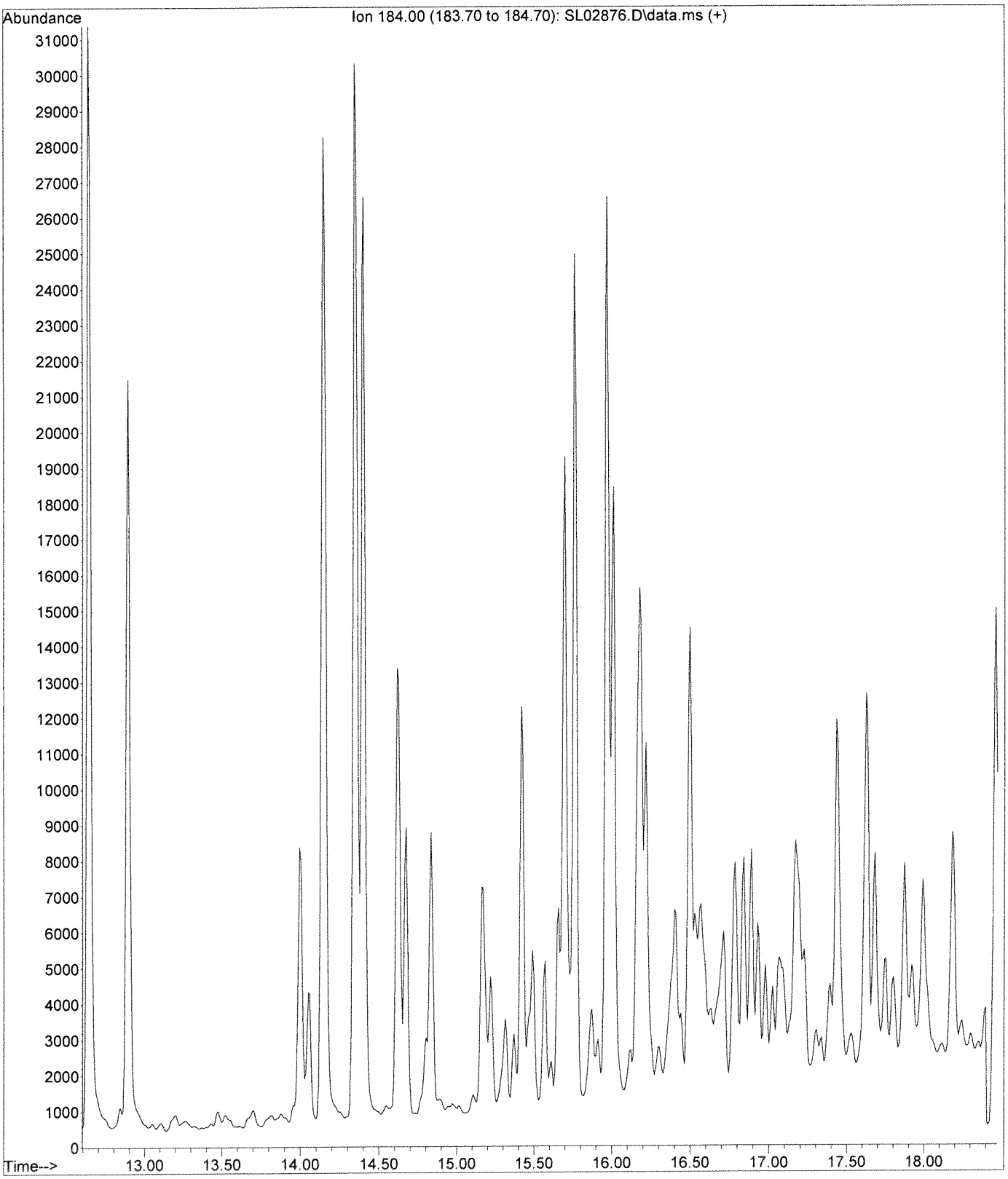
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

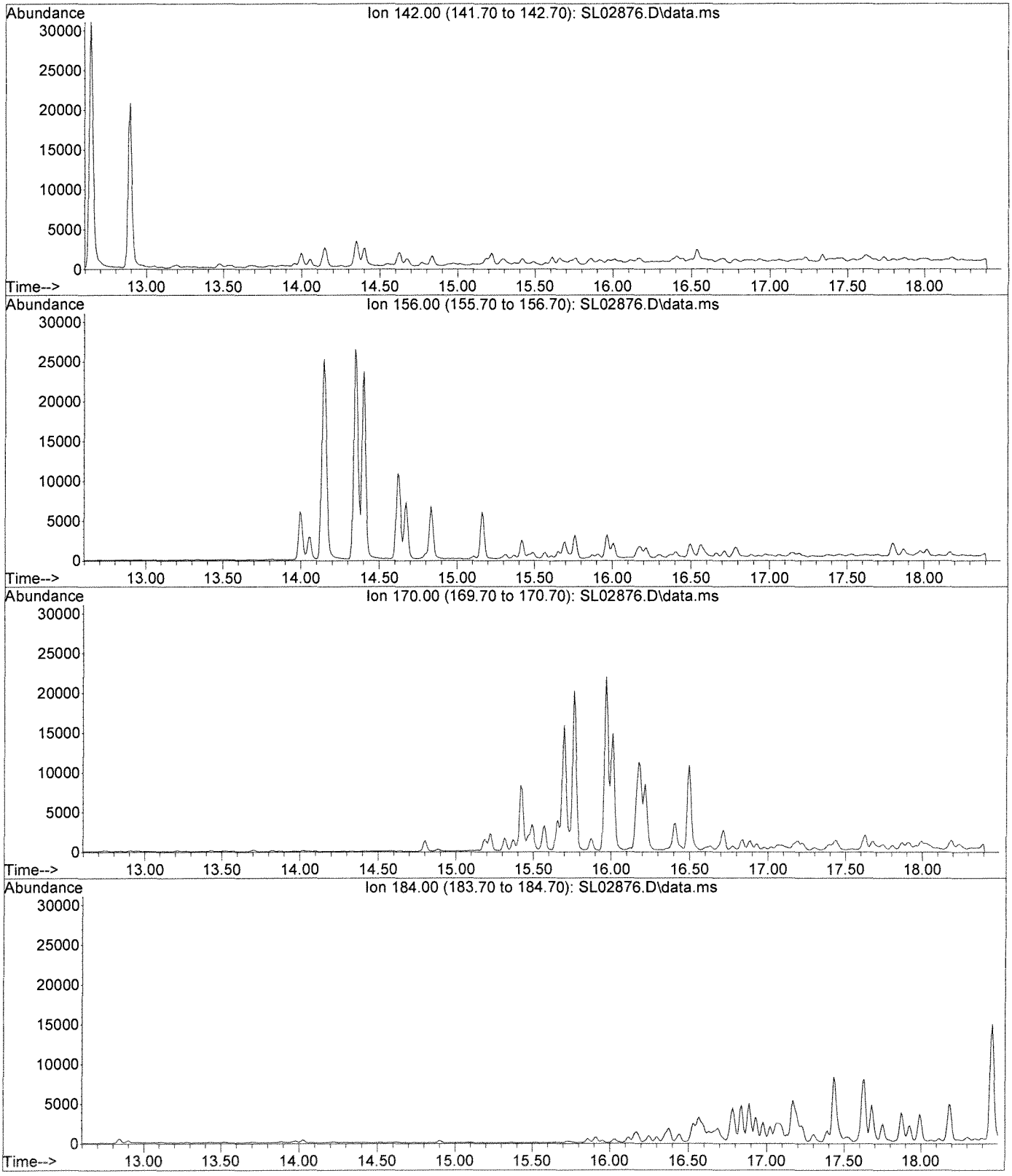
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



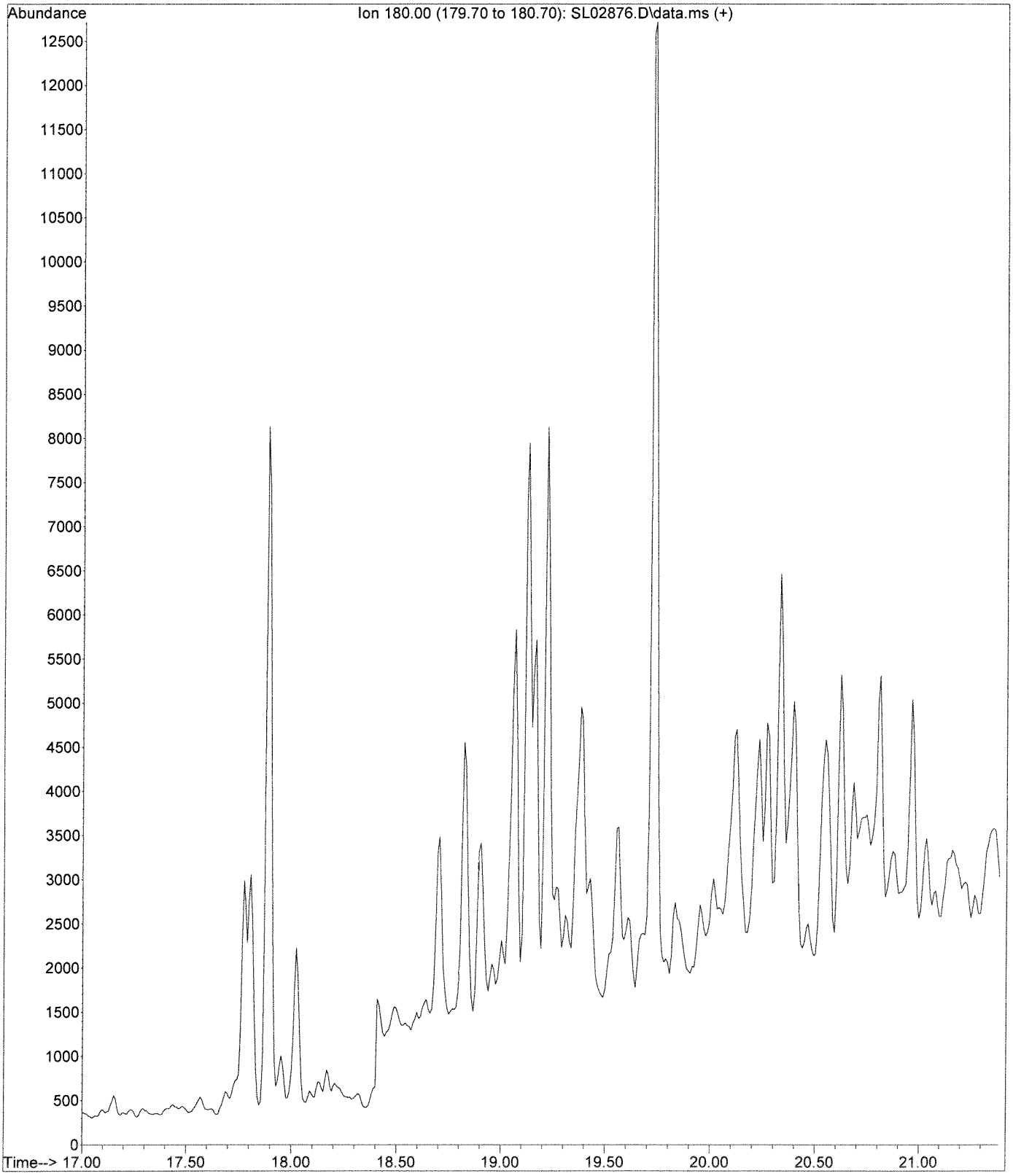
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



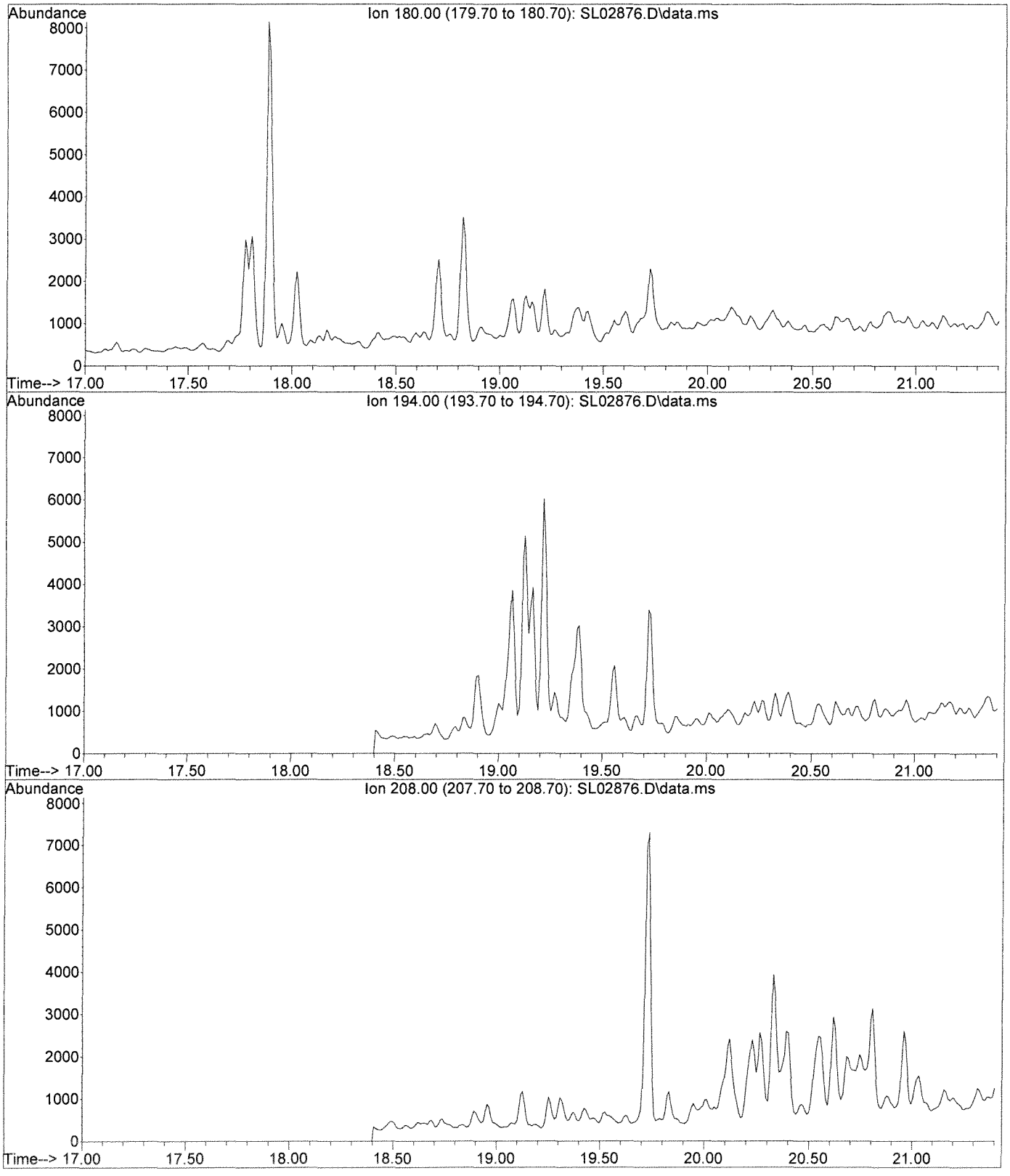
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



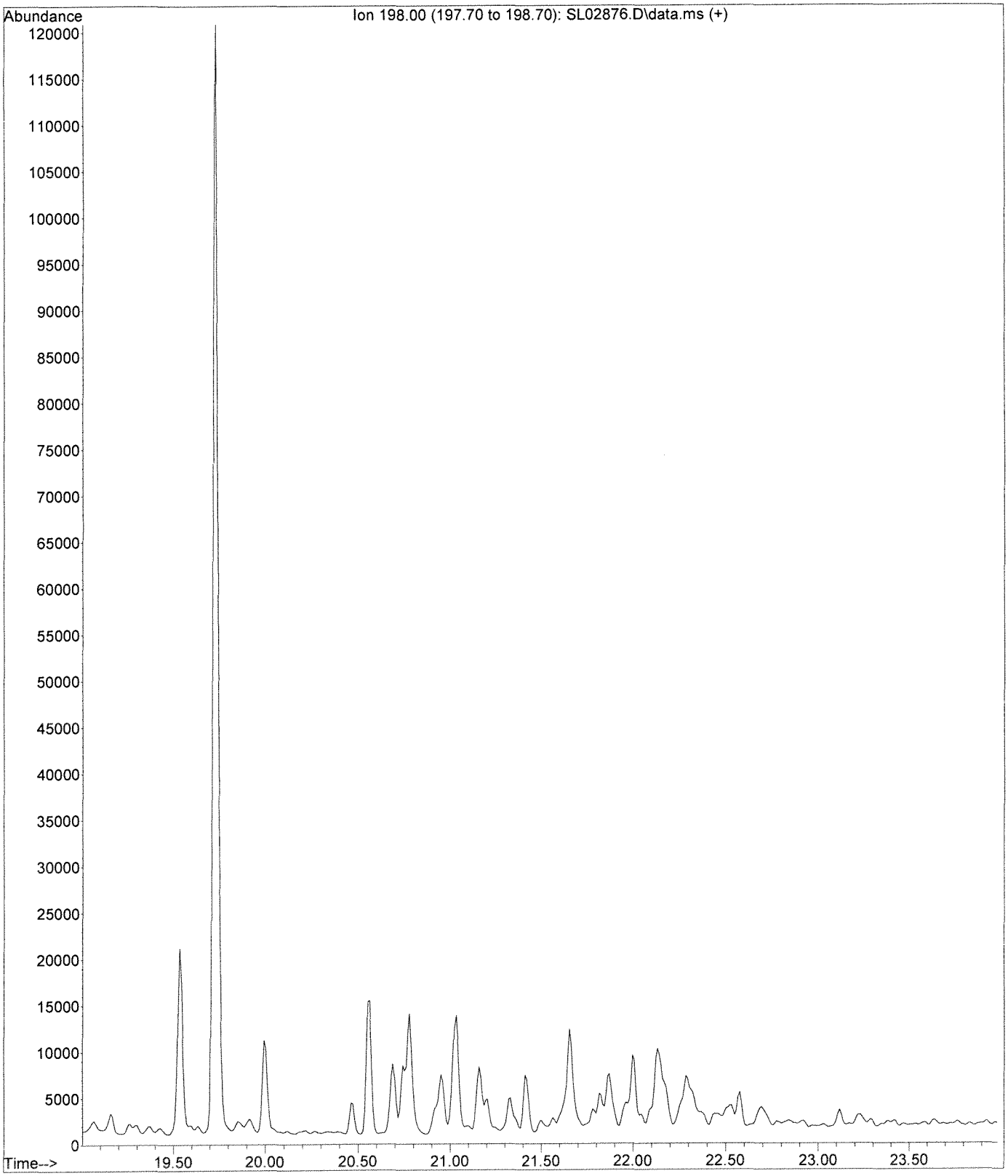
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



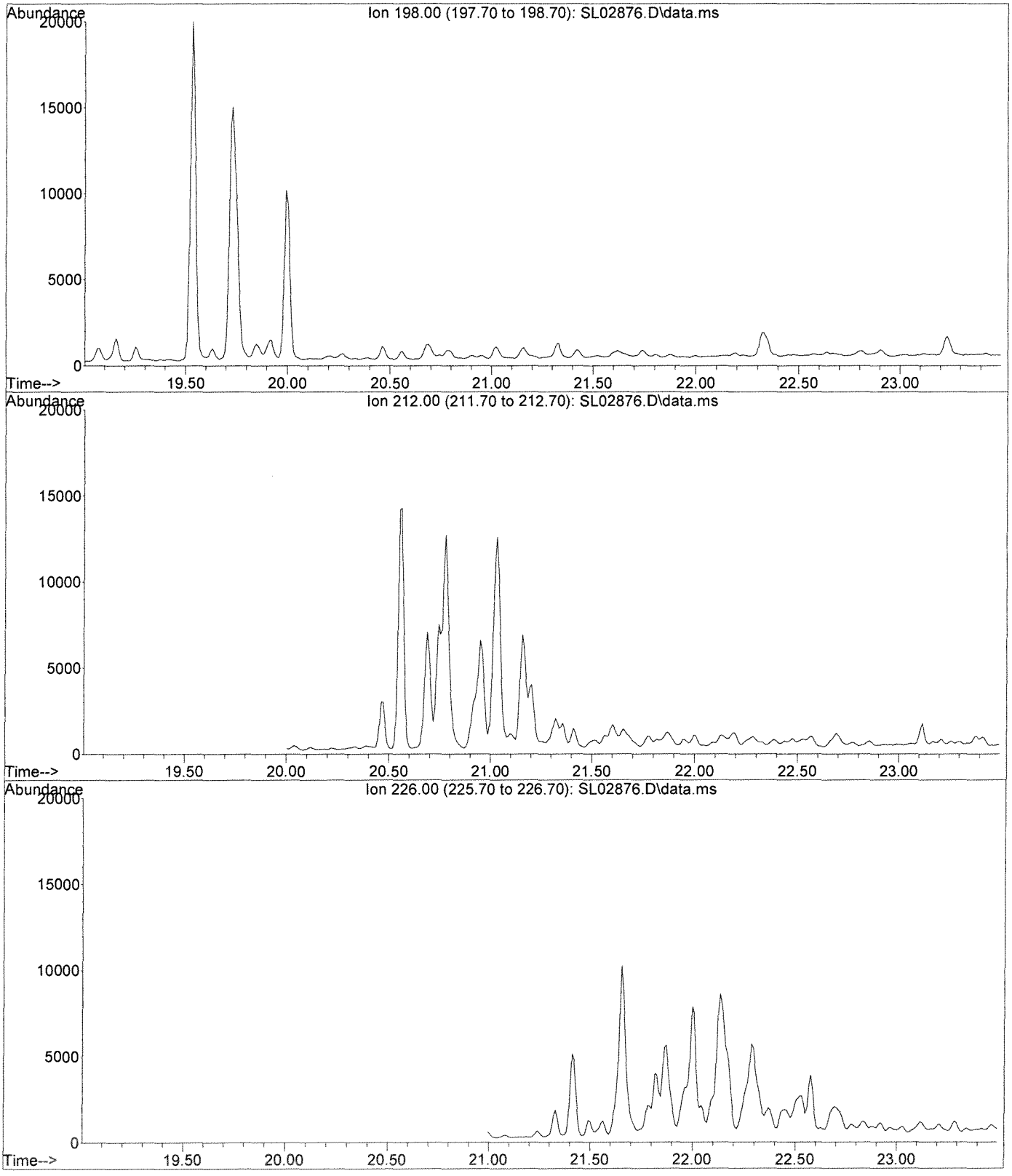
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



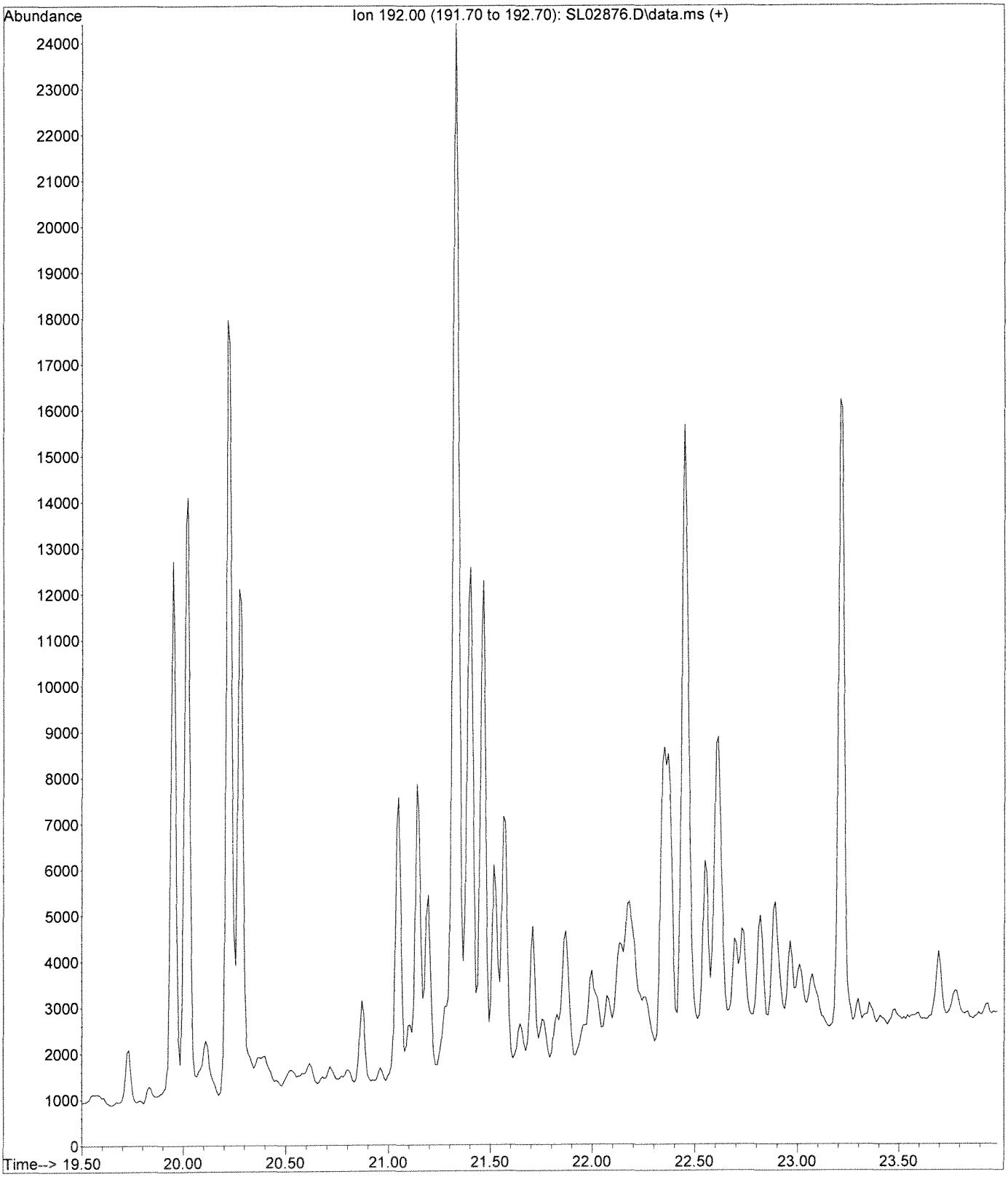
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



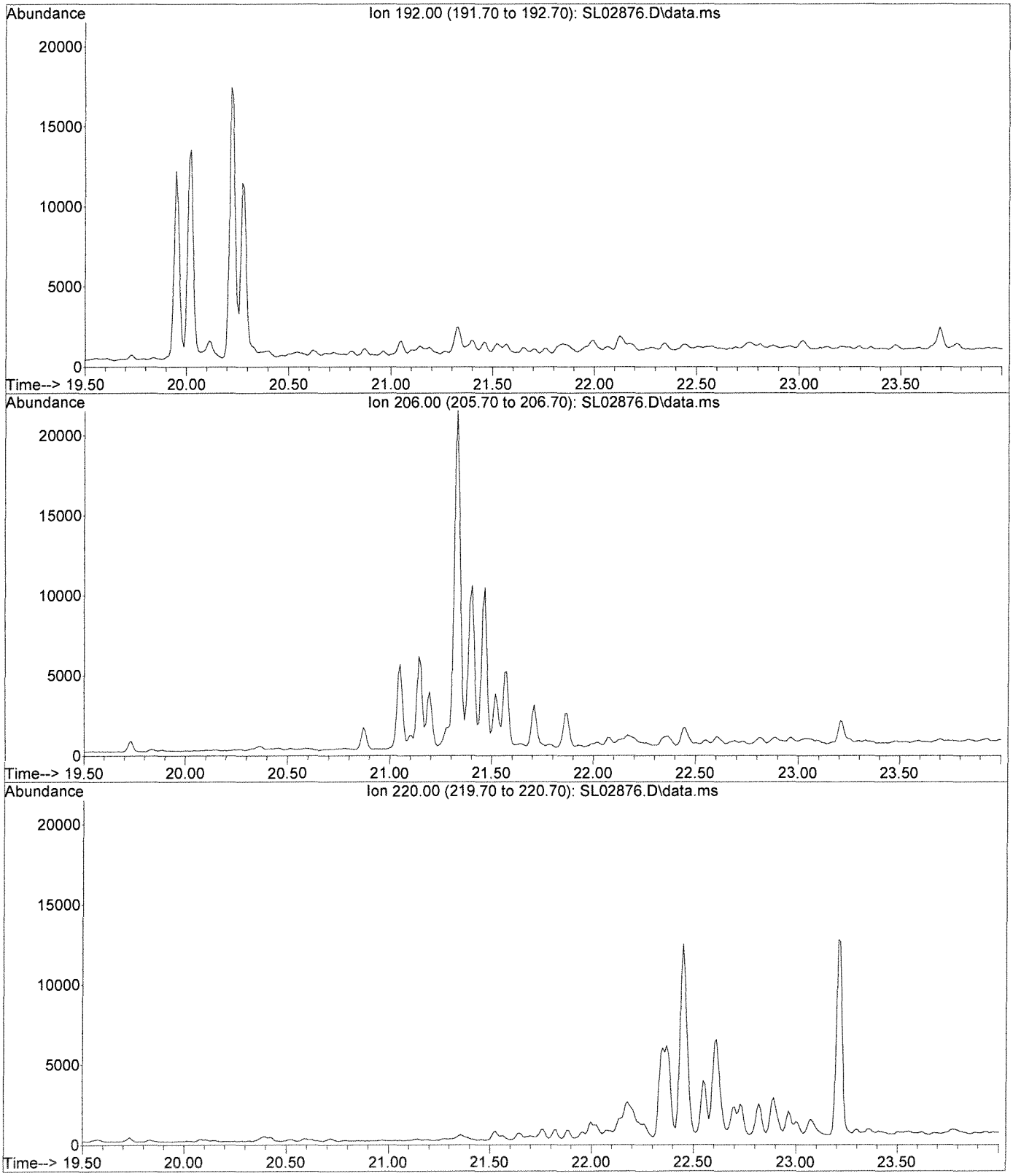
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



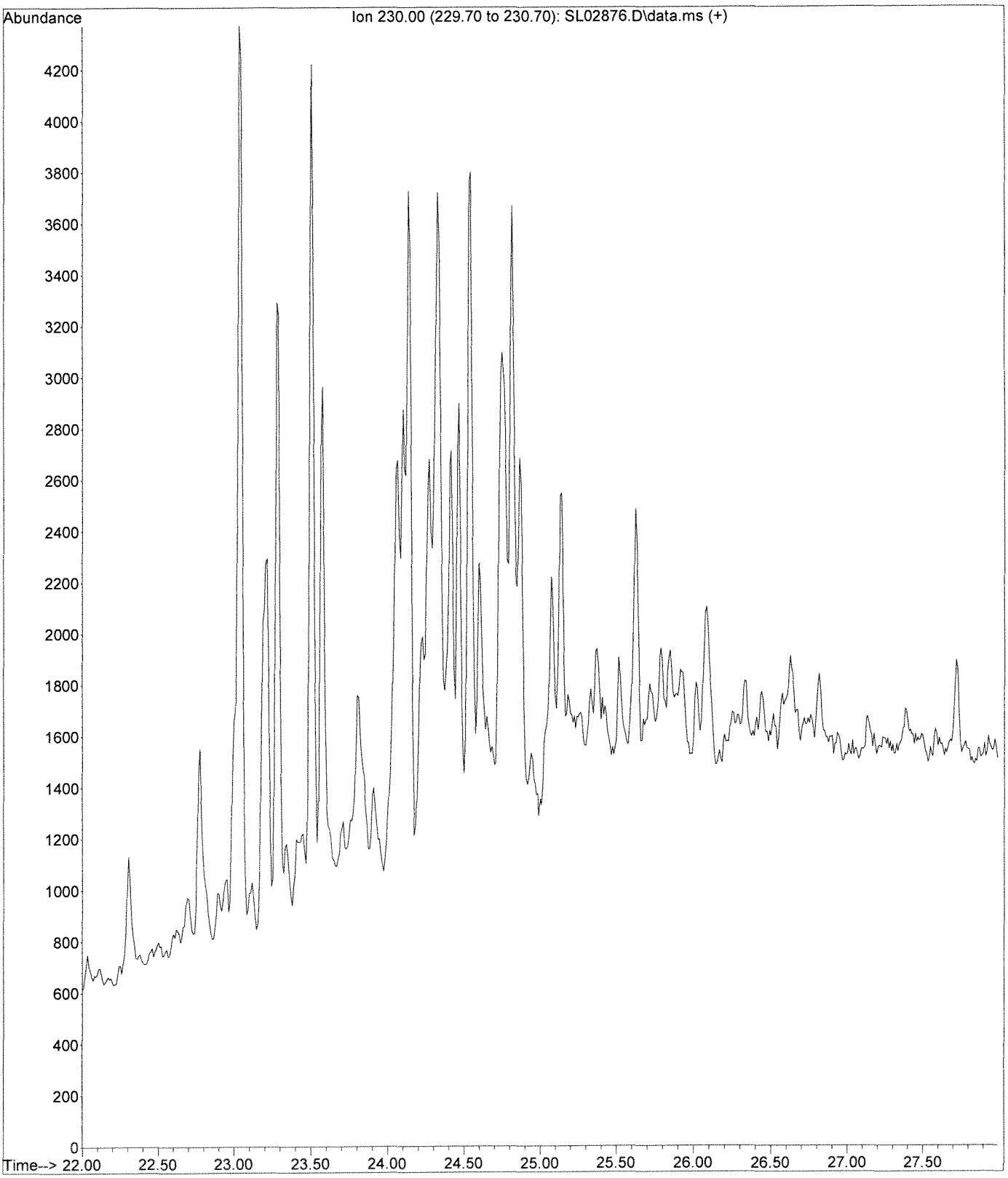
US EPA ARCHIVE DOCUMENT

```
File       :C:\msdchem\1\DATA\022712\SL02876.D
Operator   : Syslo
Acquired   : 27 Feb 2012  20:35     using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info  : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



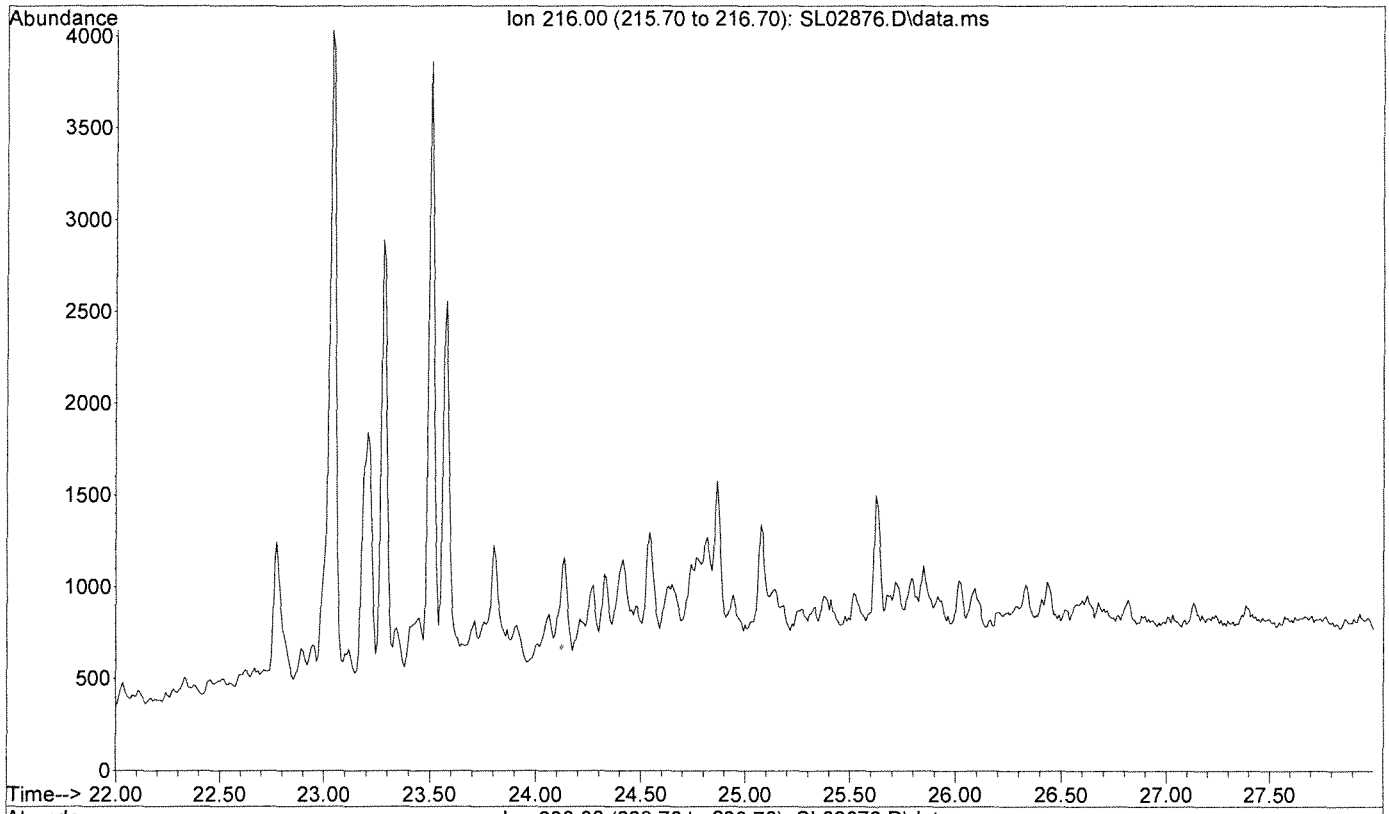
US EPA ARCHIVE DOCUMENT

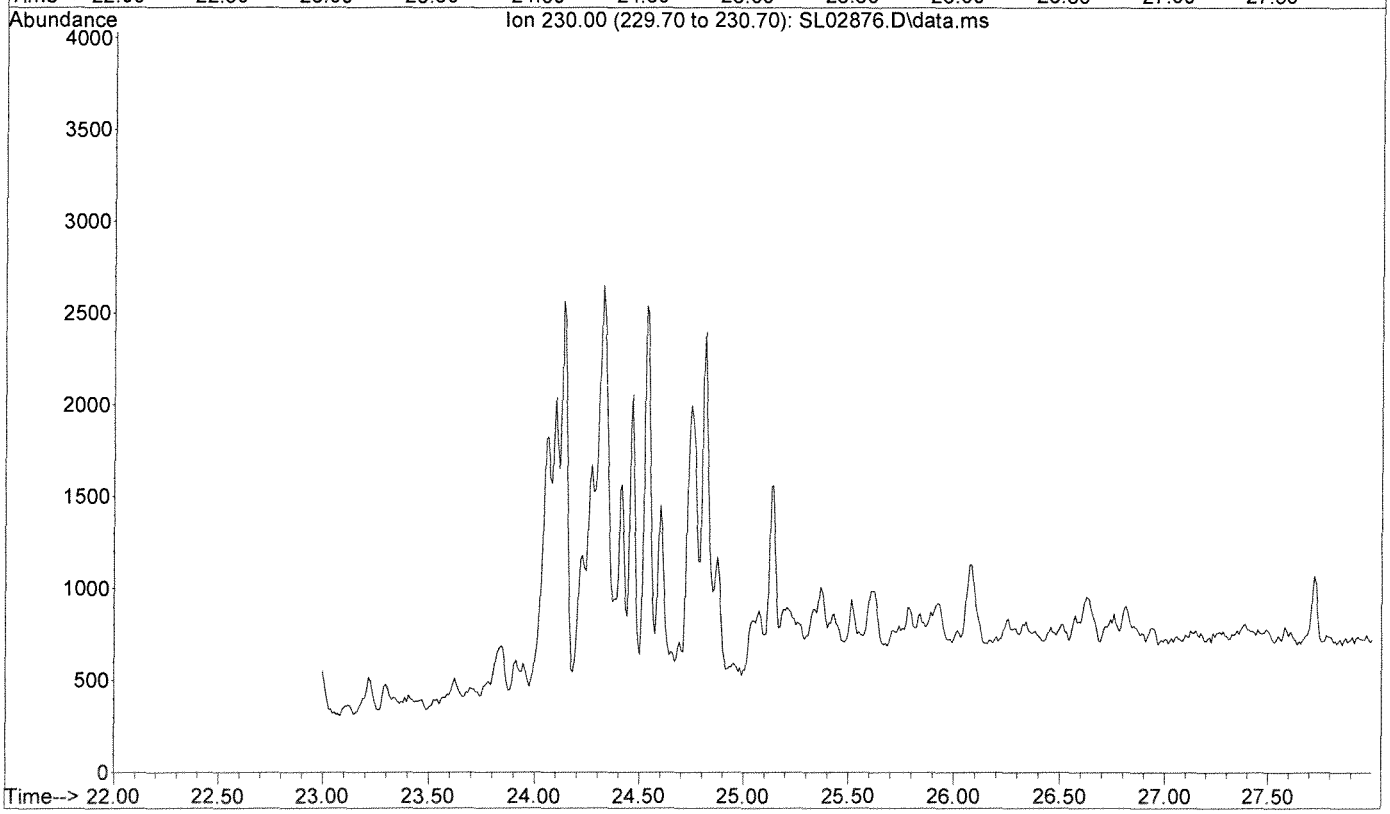
```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



US EPA ARCHIVE DOCUMENT

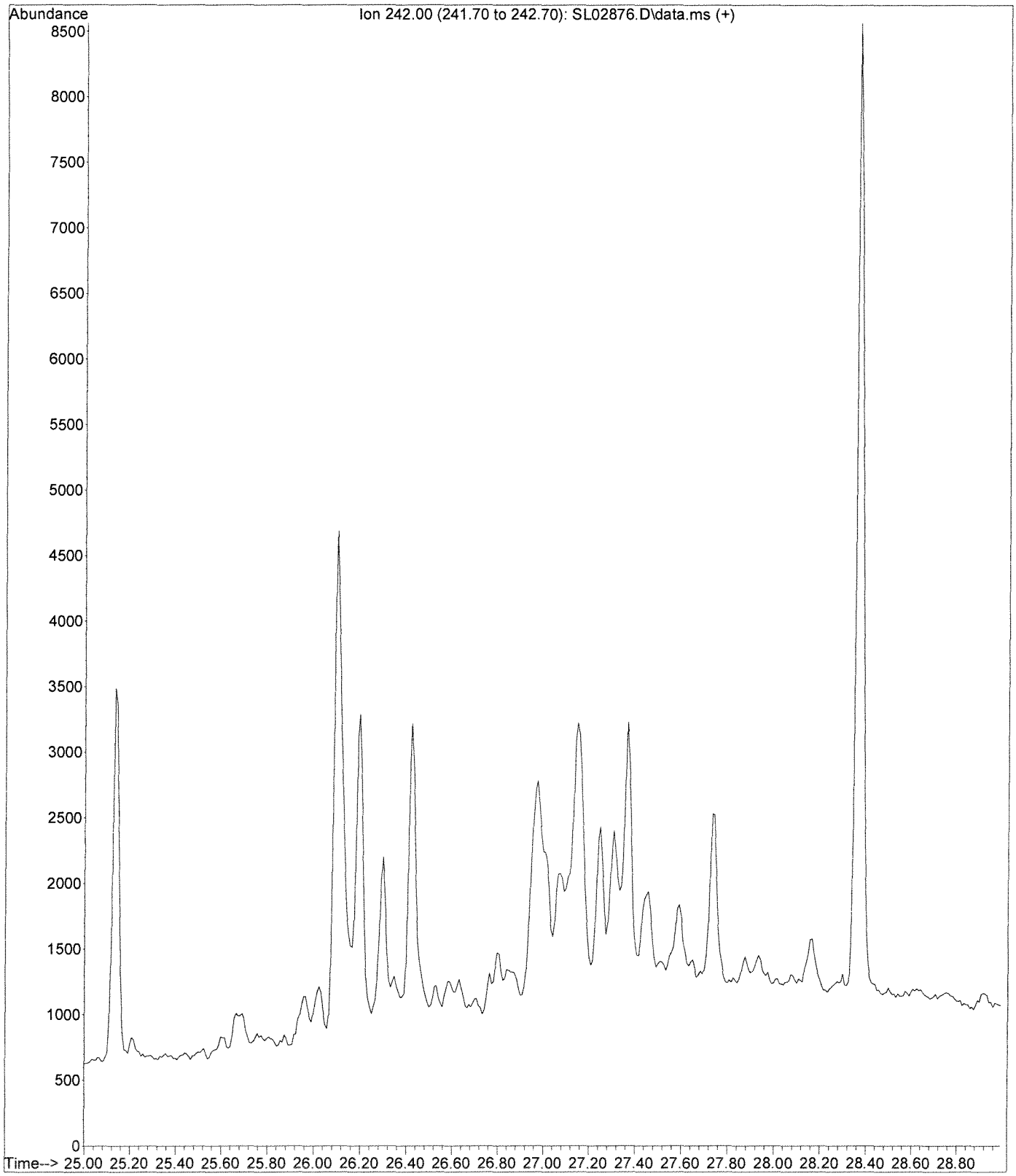
```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```





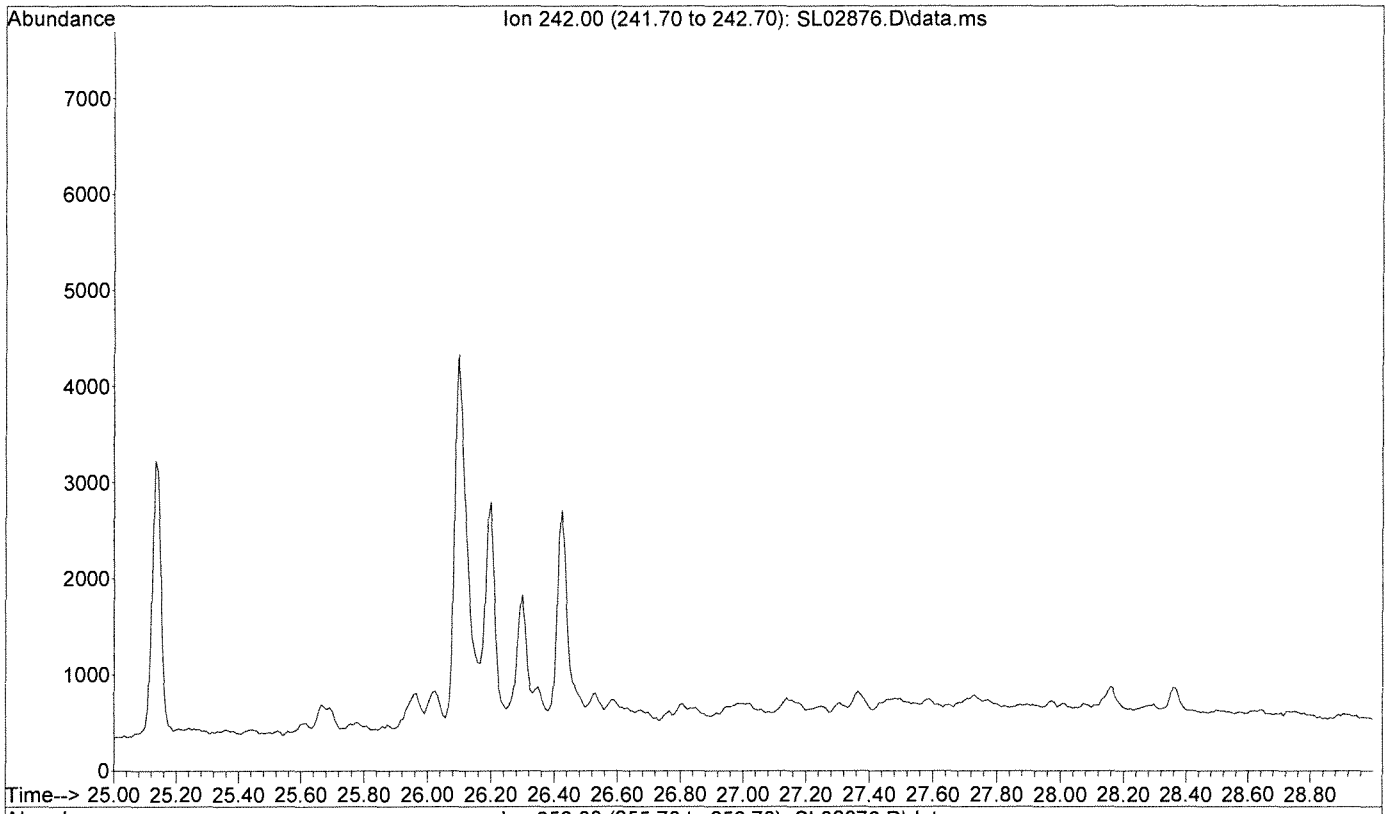
US EPA ARCHIVE DOCUMENT

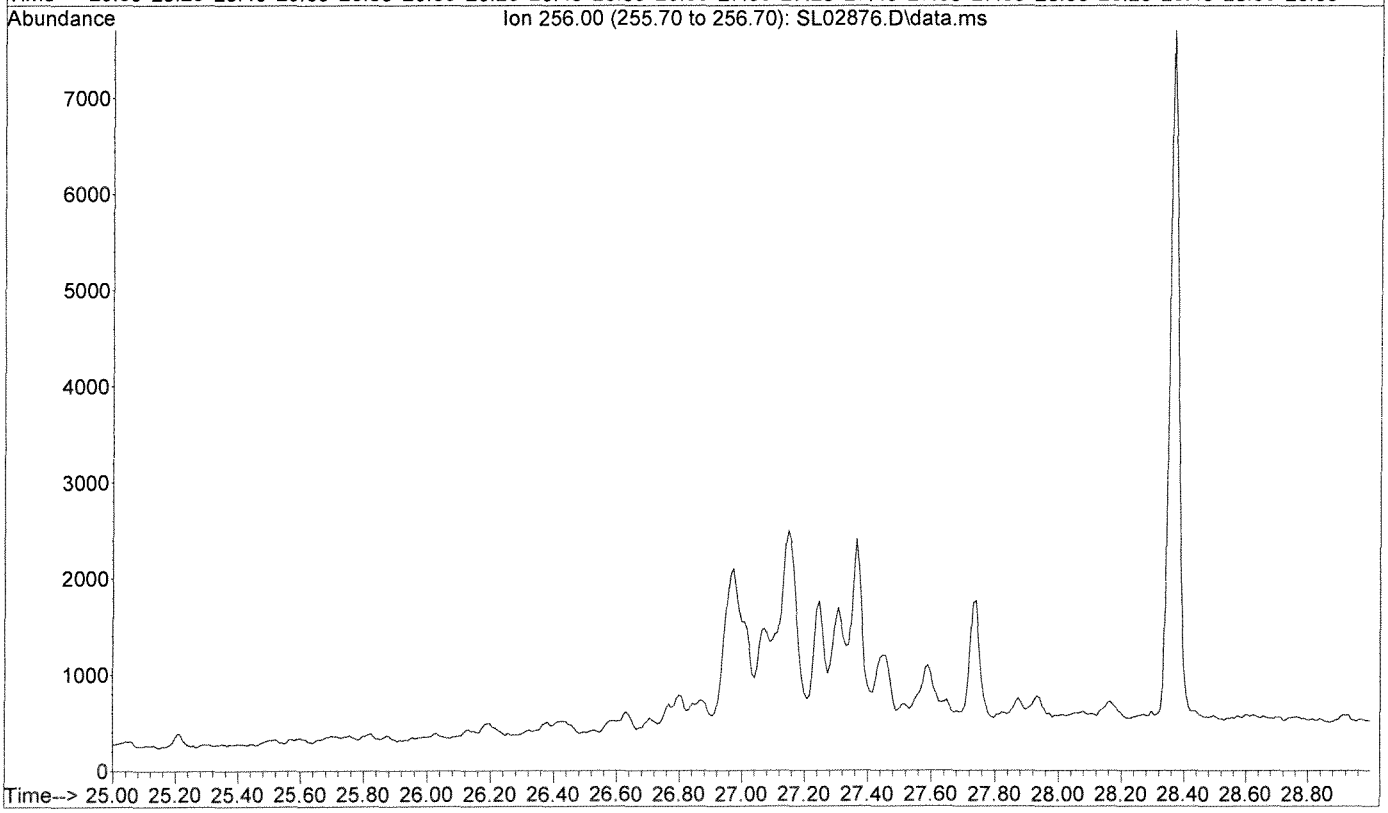
```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```





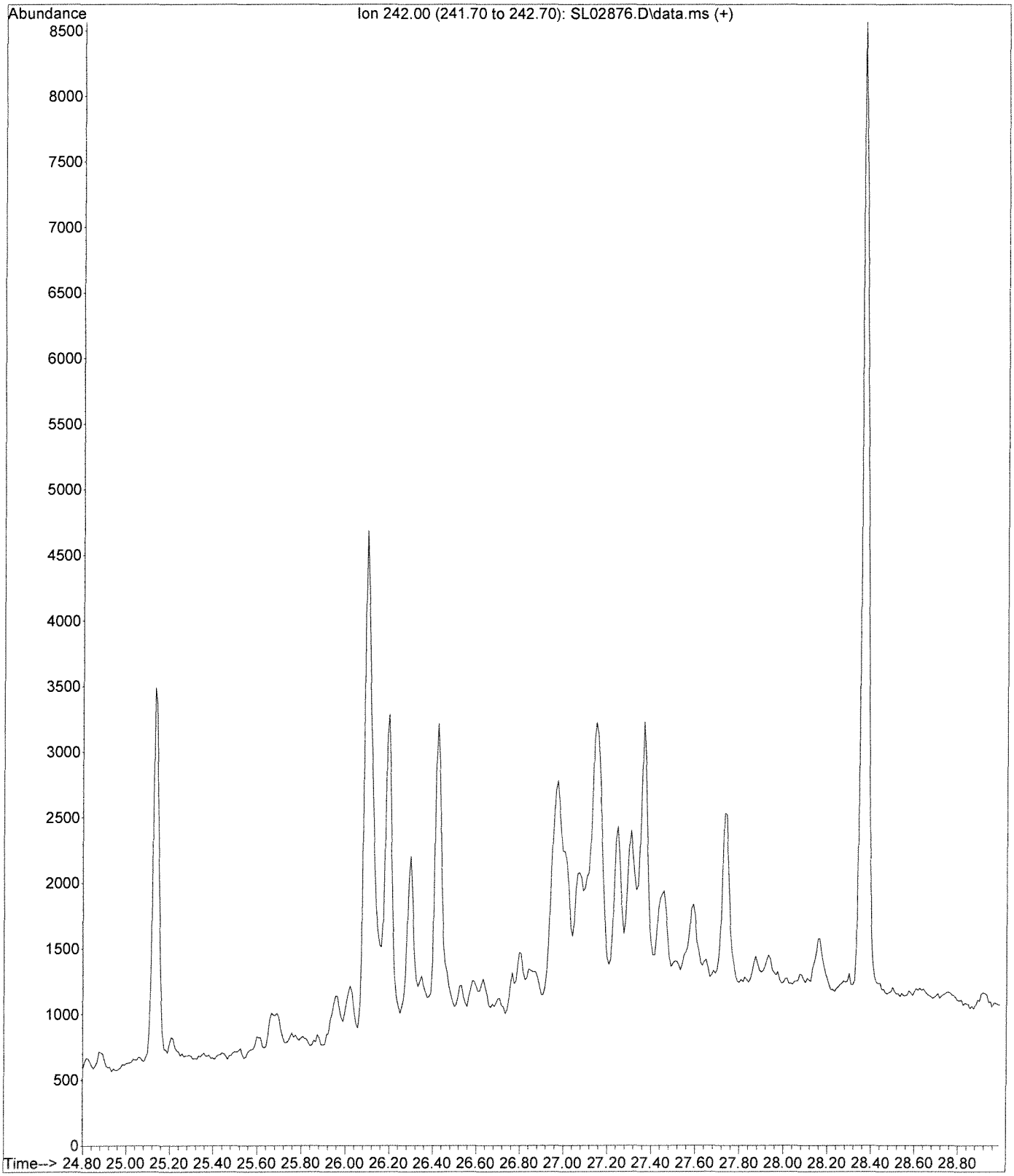
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02876.D
Operator    : Syslo
Acquired    : 27 Feb 2012  20:35      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 dup
Misc Info   : 275-55-05 {0.111gi n10mL}
Vial Number: 3
```



US EPA ARCHIVE DOCUMENT

SERAS-017-0004

SERAS-017-DTM-011413_3

0563

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02877.D
Acq On : 27 Feb 2012 21:37
Operator : Syslo
Sample : SERAS-017-0004
Misc : 275-56-24 {0.029g in 2.0mL}
ALS Vial : 4 Sample Multiplier: 1

Quant Time: Mar 01 13:06:30 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	298775	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	96470	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	122101	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	113788	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.821	188	510238	16.06	ug/mL	0.00
Spiked Amount 20.000			Recovery =	80.30%		
3) d14-o-Terphenyl {S}	19.733	244	246865	17.68	ug/mL	0.00
Spiked Amount 20.000			Recovery =	88.40%		
4) 5a-Androstane {S}	21.140	260	34192	20.13	ug/mL	0.00
Spiked Amount 20.000			Recovery =	100.65%		
7) d62-Triacontane {S}	28.156	66	224406	20.76	ug/mL	0.00
Spiked Amount 20.000			Recovery =	103.80%		
Target Compounds						Qvalue

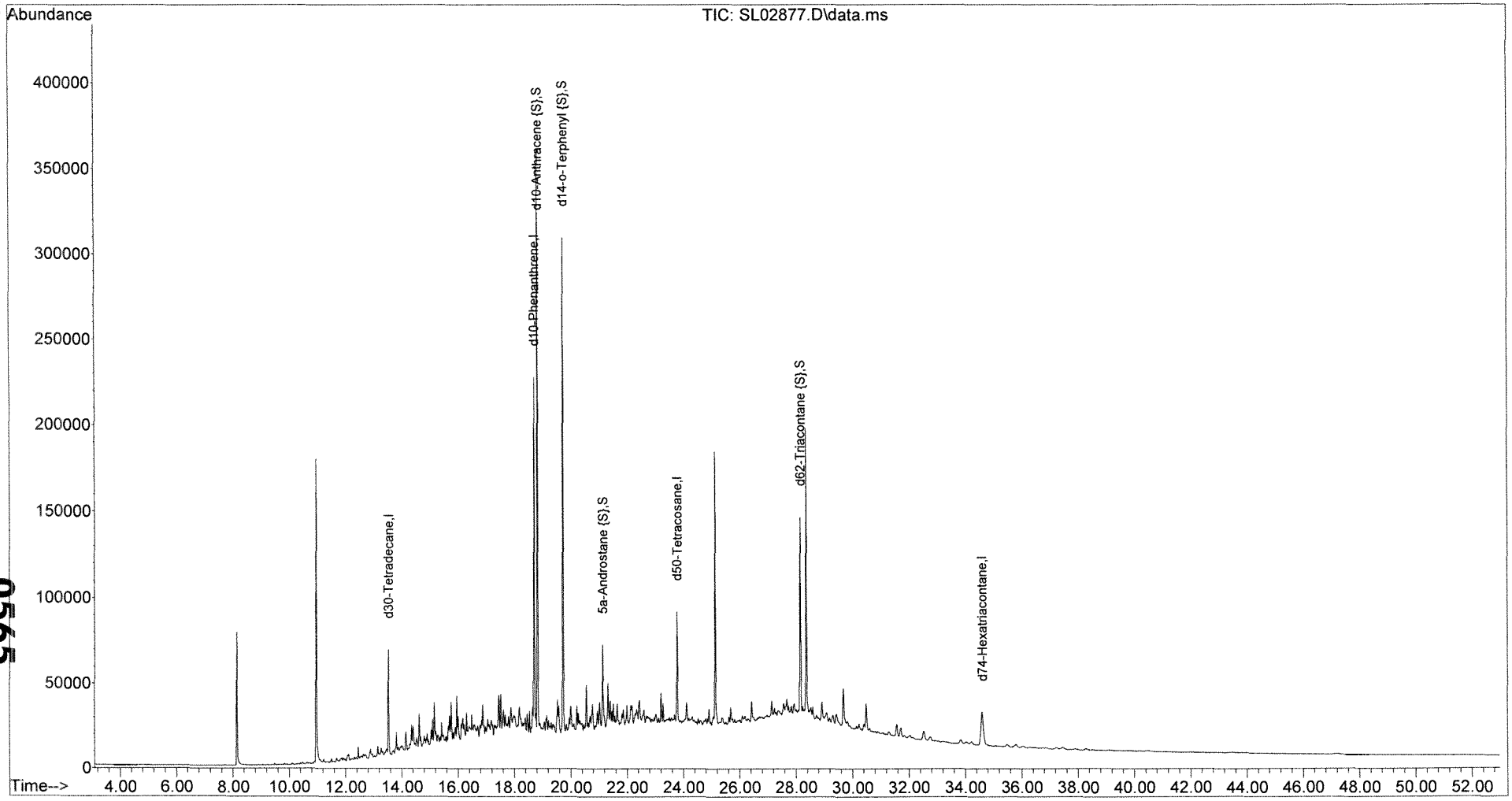
#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02877.D
Acq On : 27 Feb 2012 21:37
Operator : Syslo
Sample : SERAS-017-0004
Misc : 275-56-24 {0.029g in 2.0mL}
ALS Vial : 4 Sample Multiplier: 1

Quant Time: Mar 01 13:06:30 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0565

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

US EPA ARCHIVE DOCUMENT

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

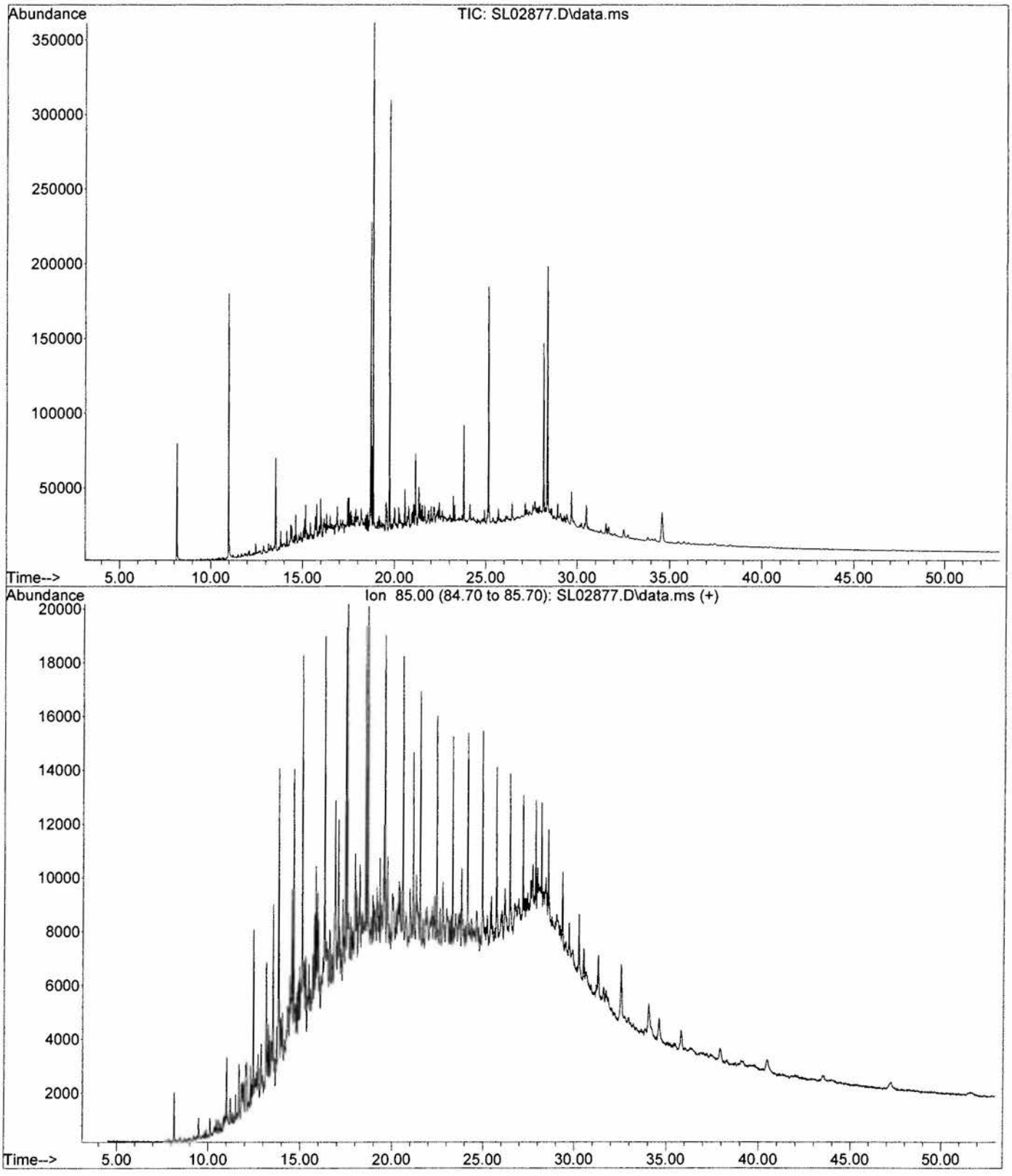
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```

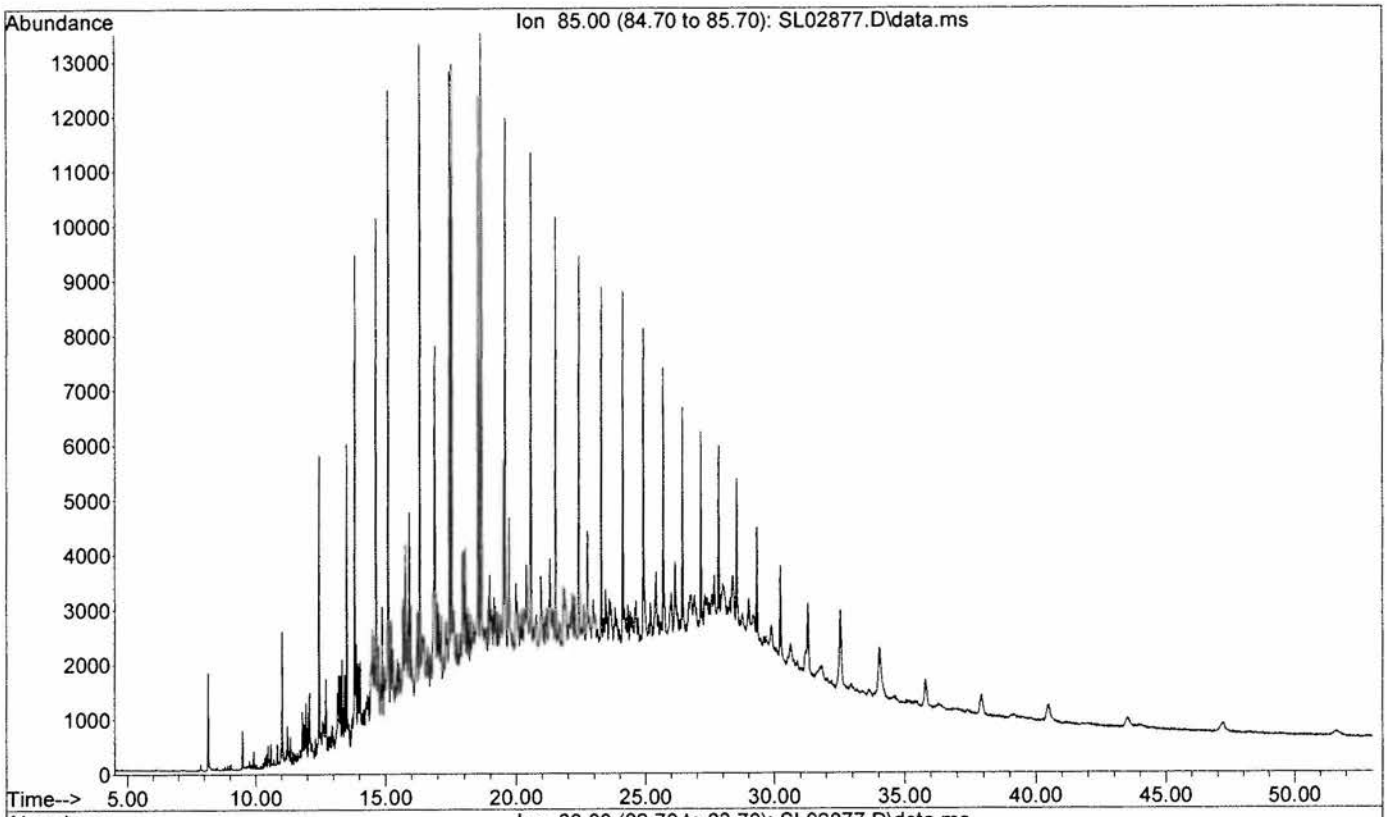


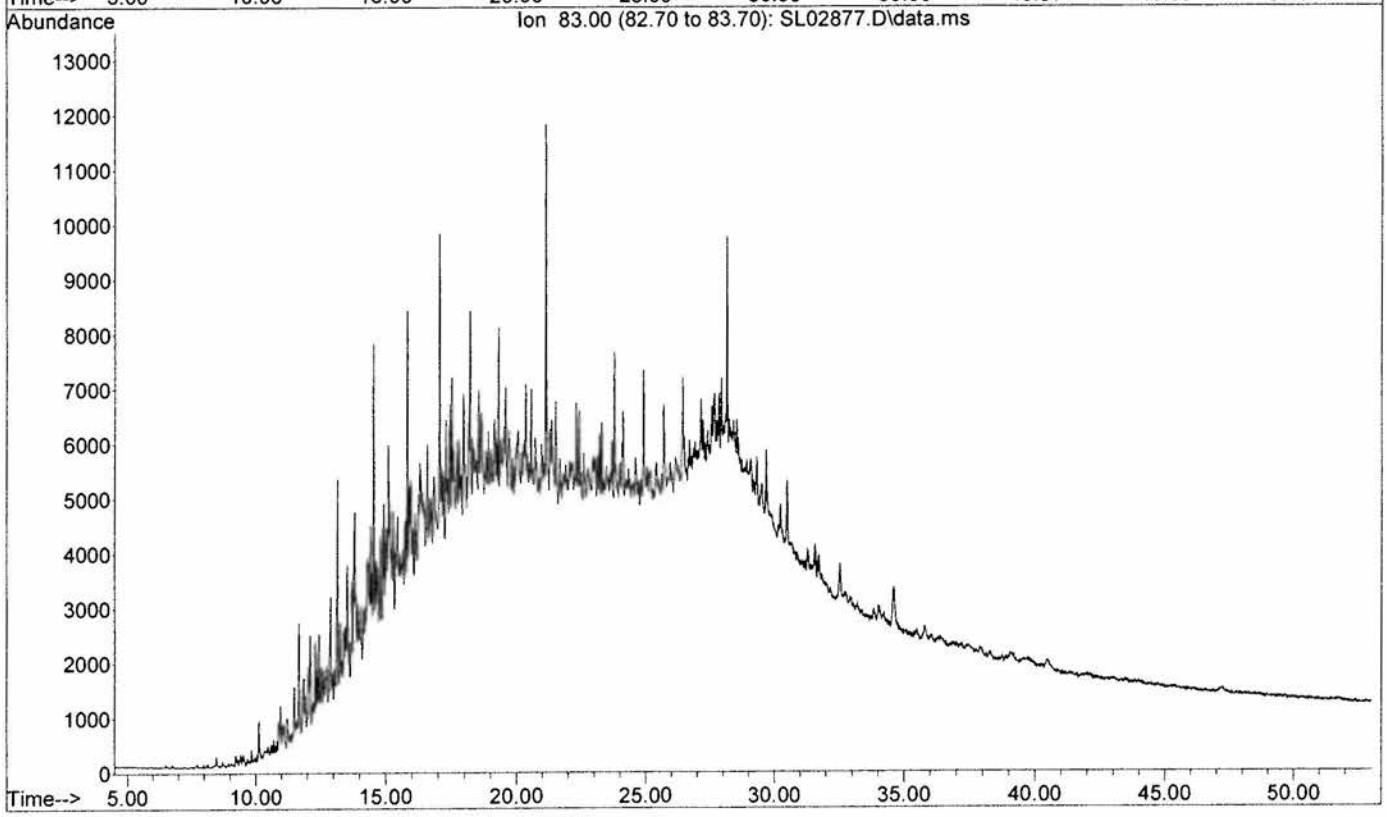
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4





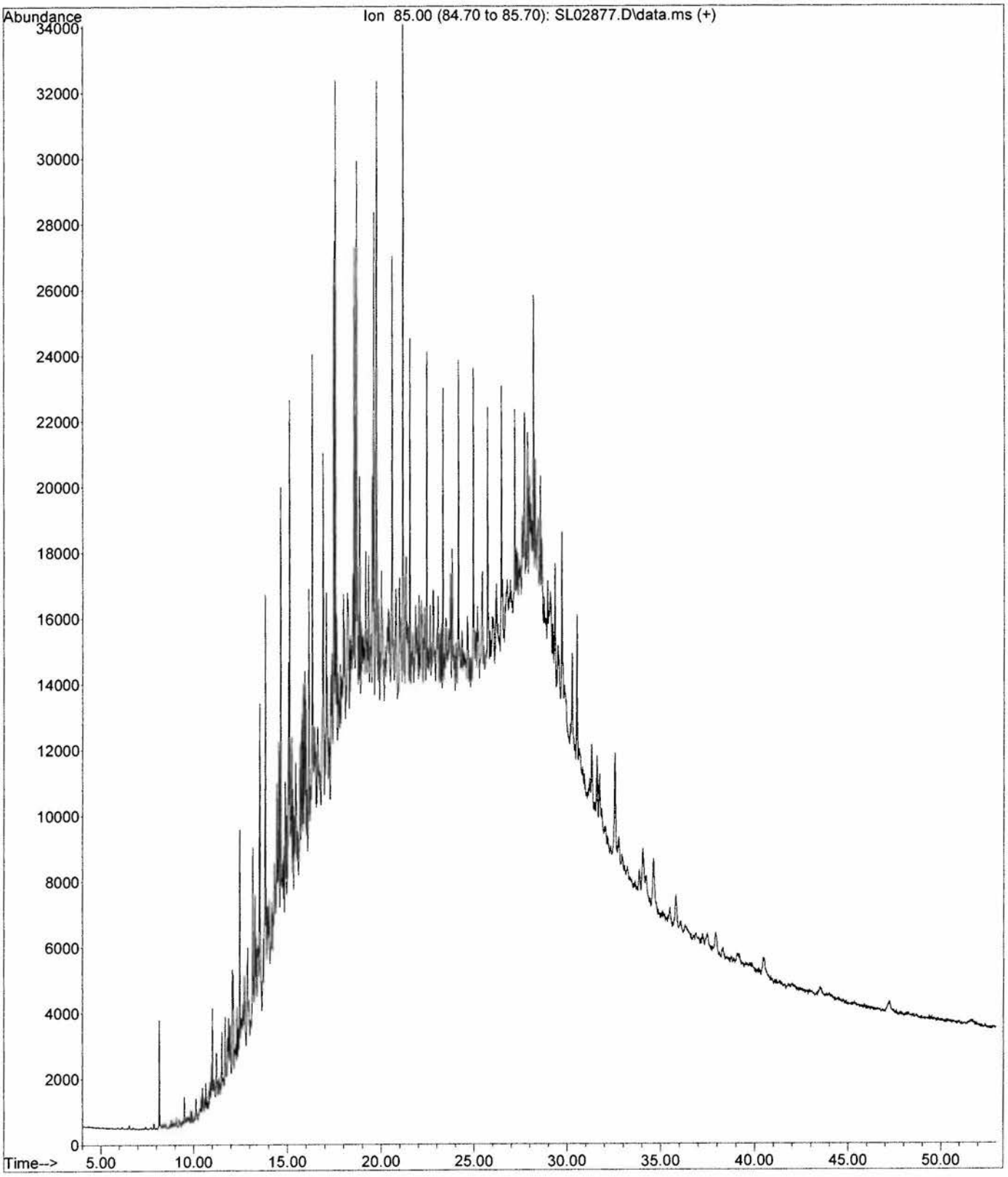
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



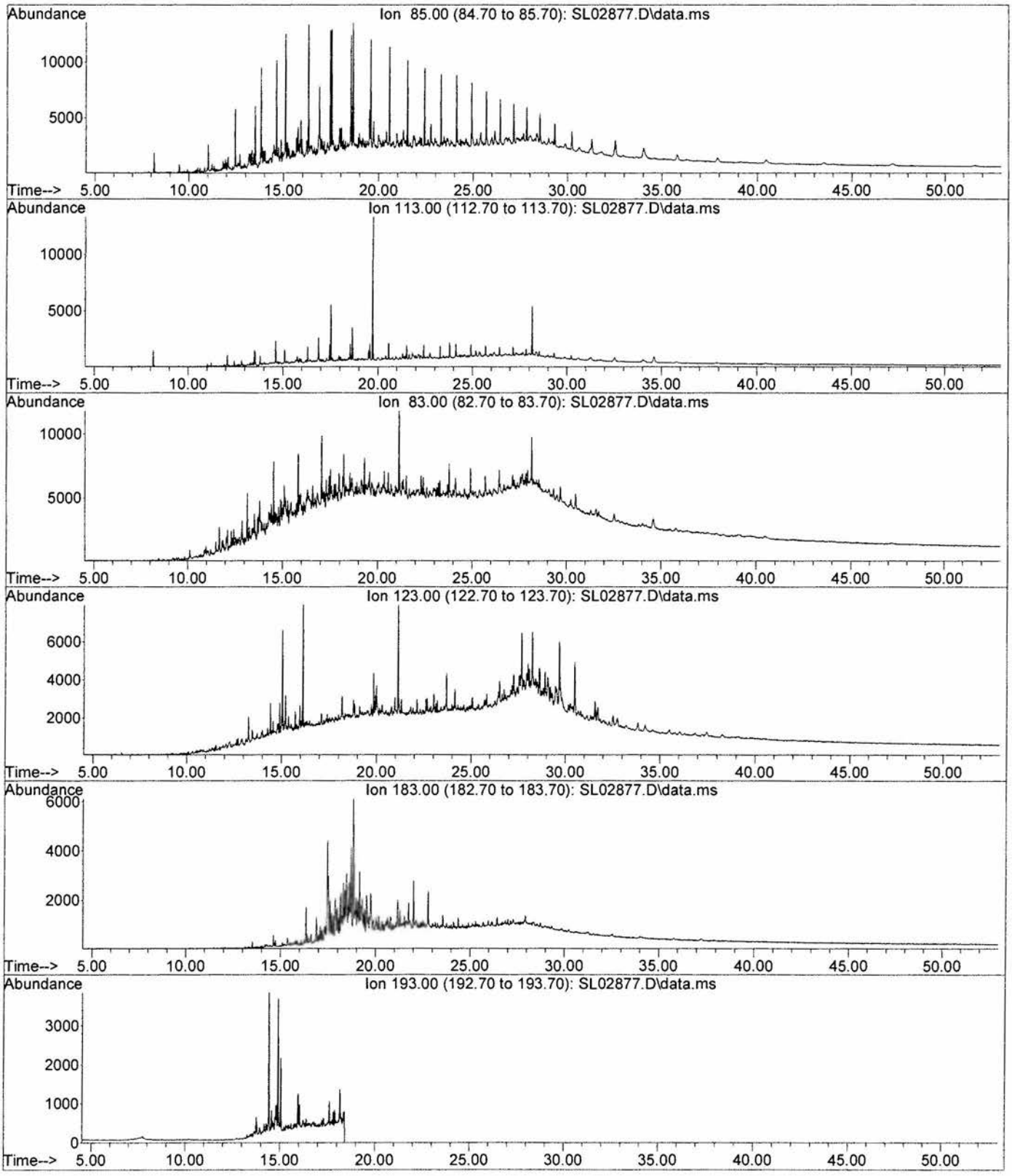
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```



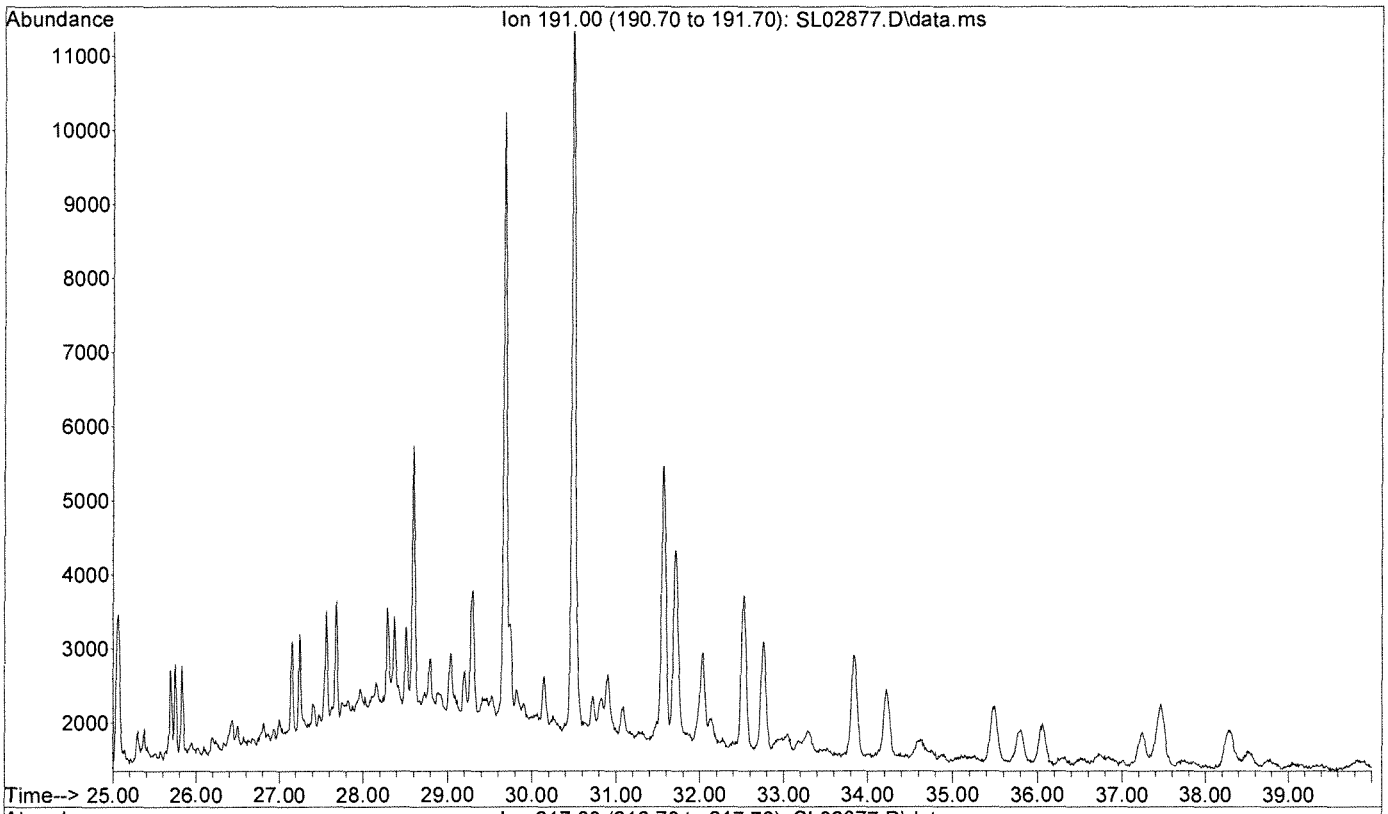
US EPA ARCHIVE DOCUMENT

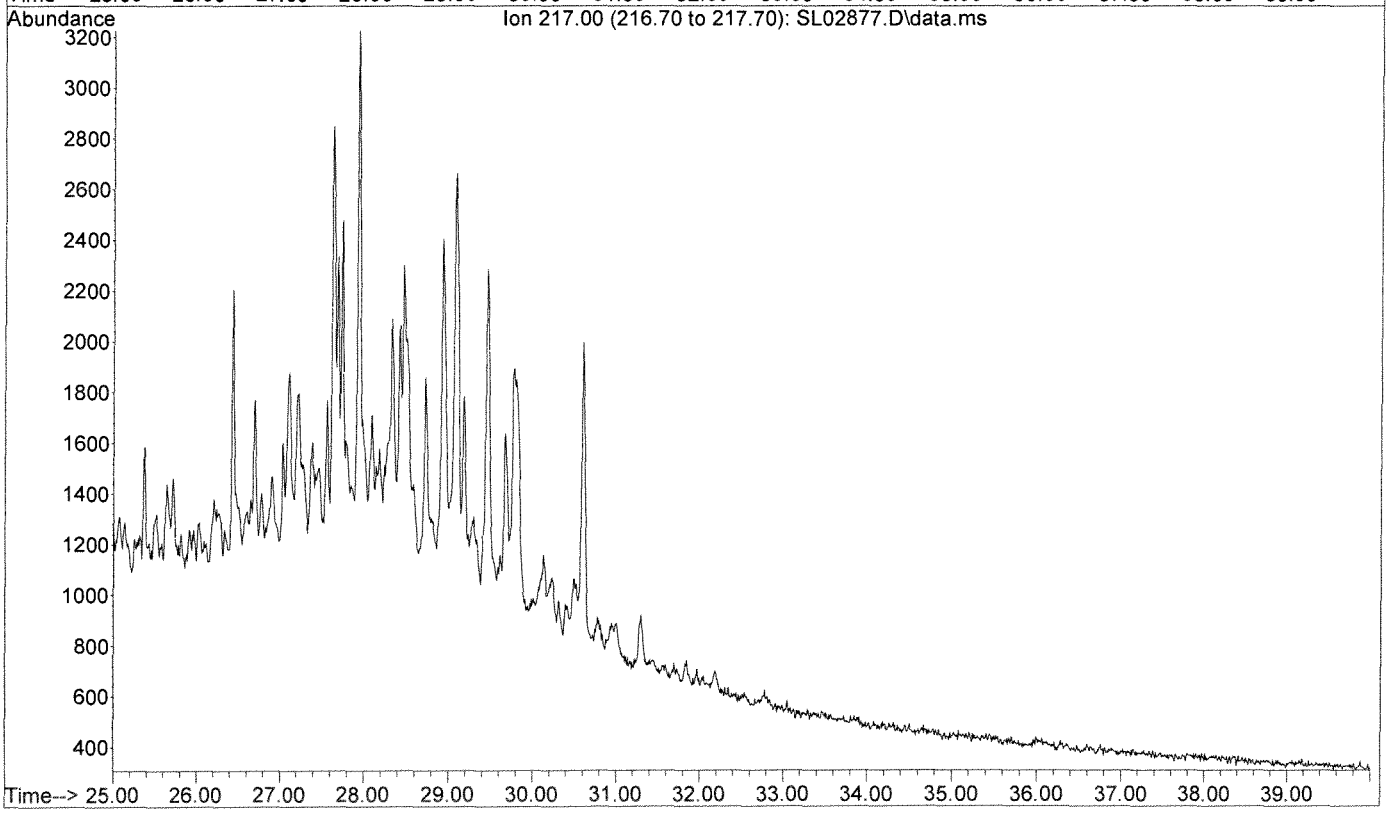
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```





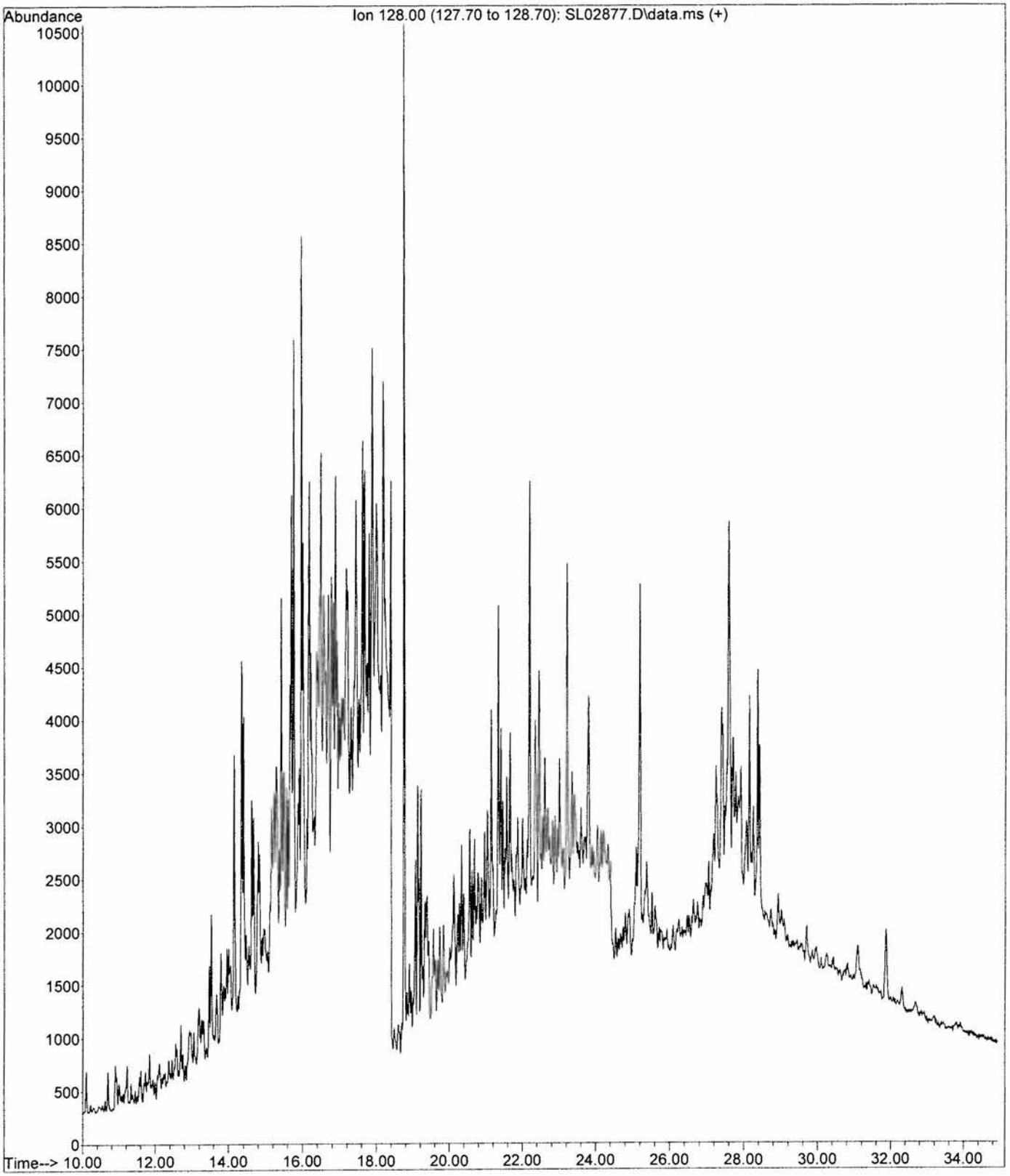
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



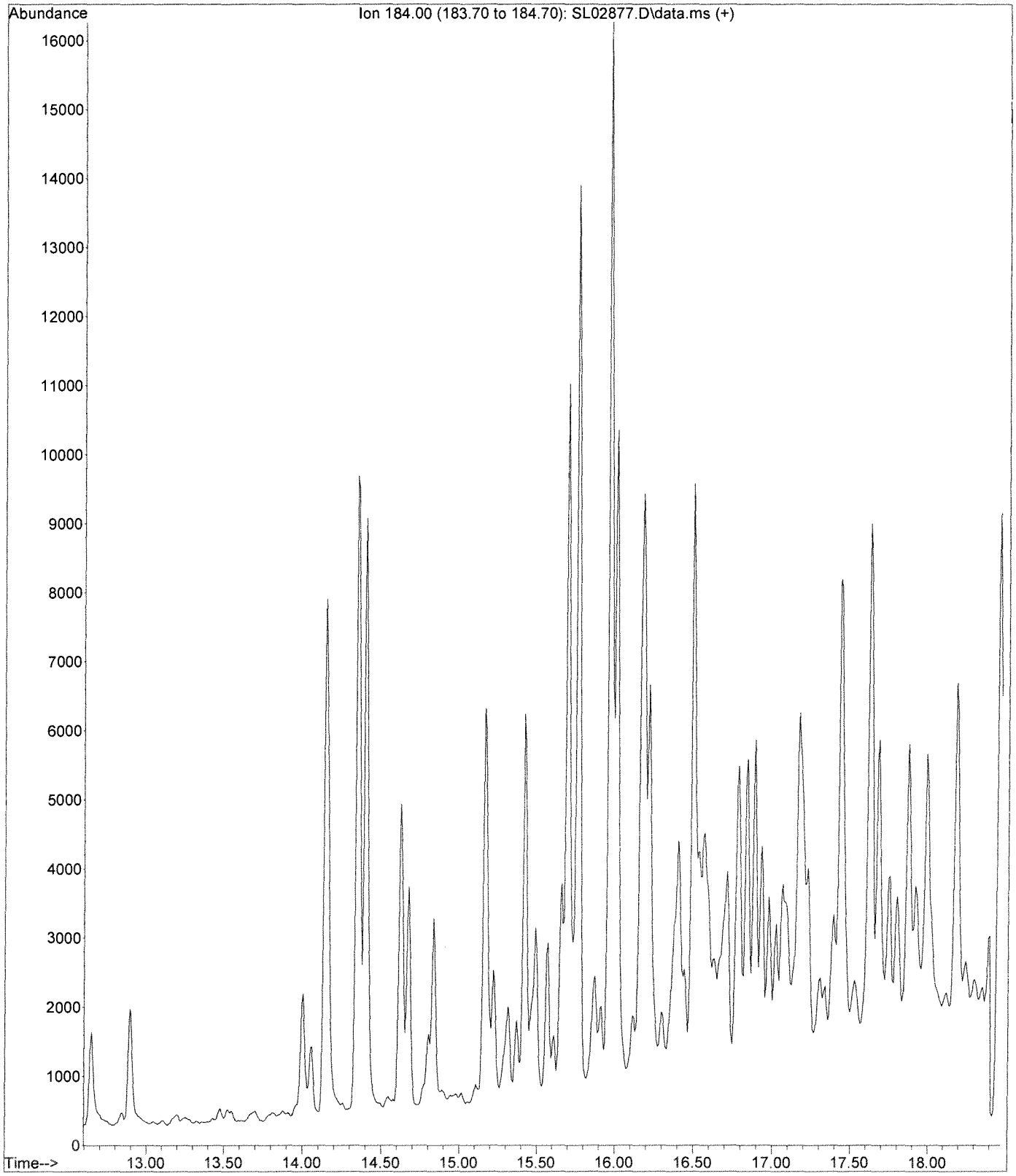
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

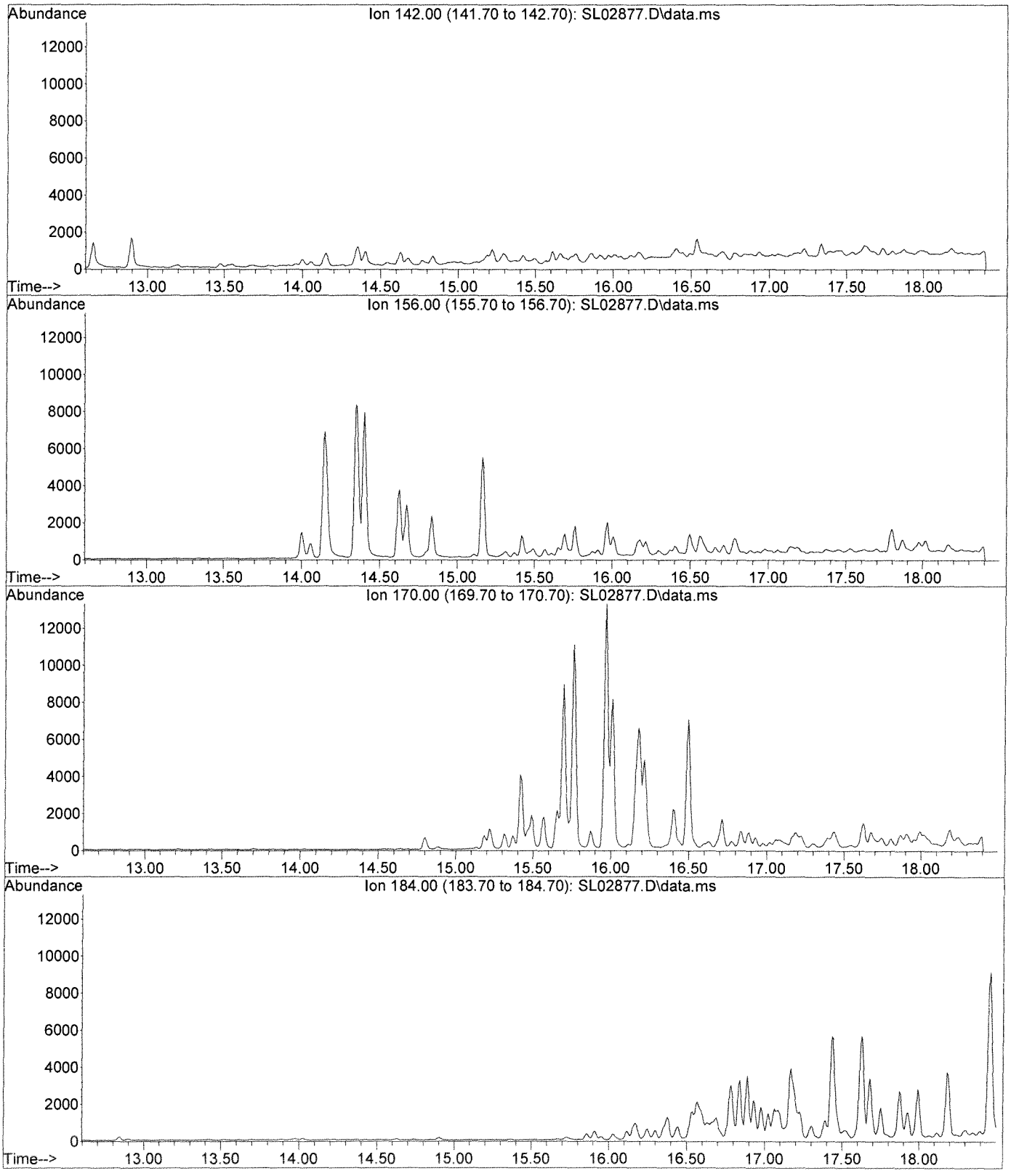
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



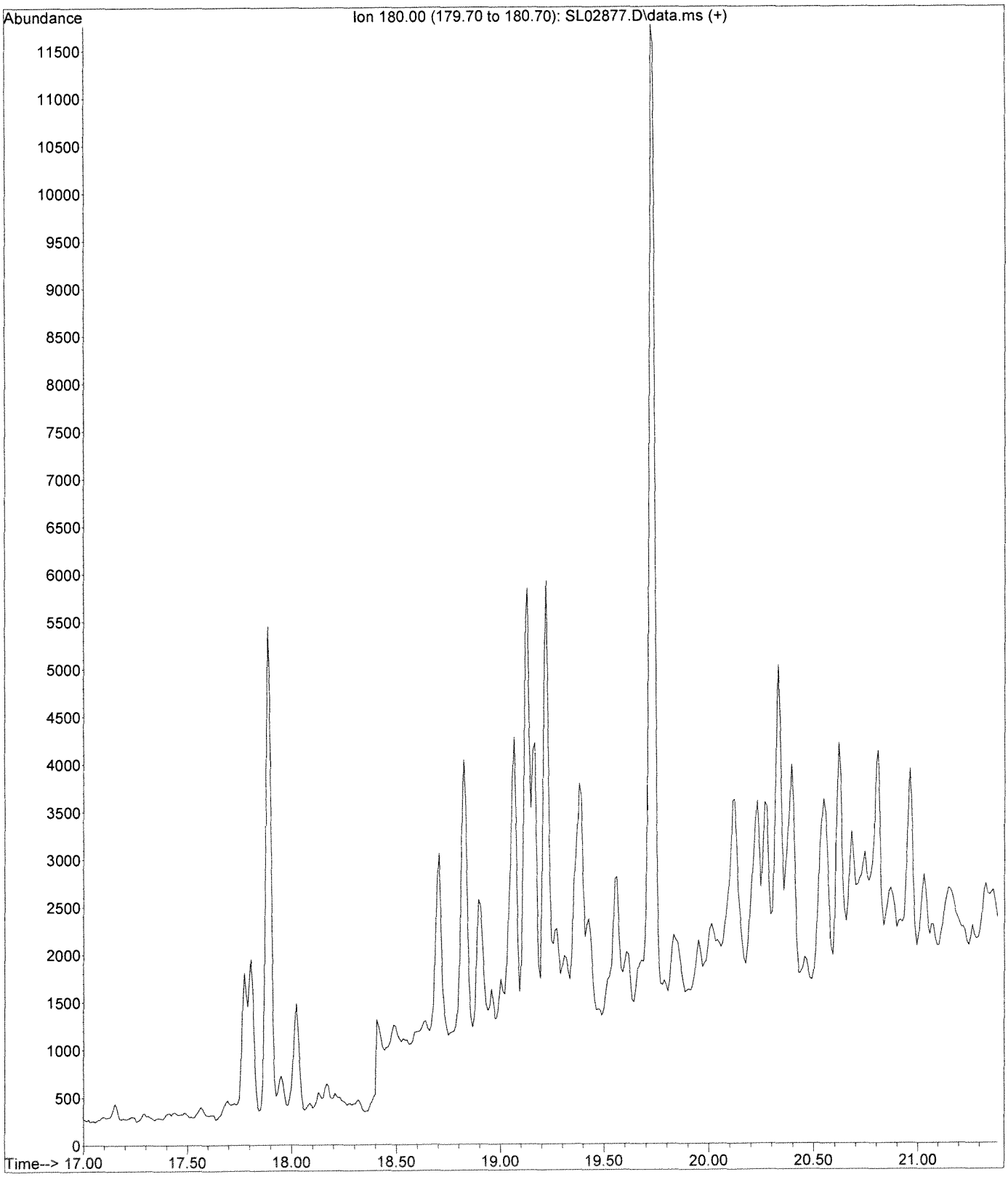
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```



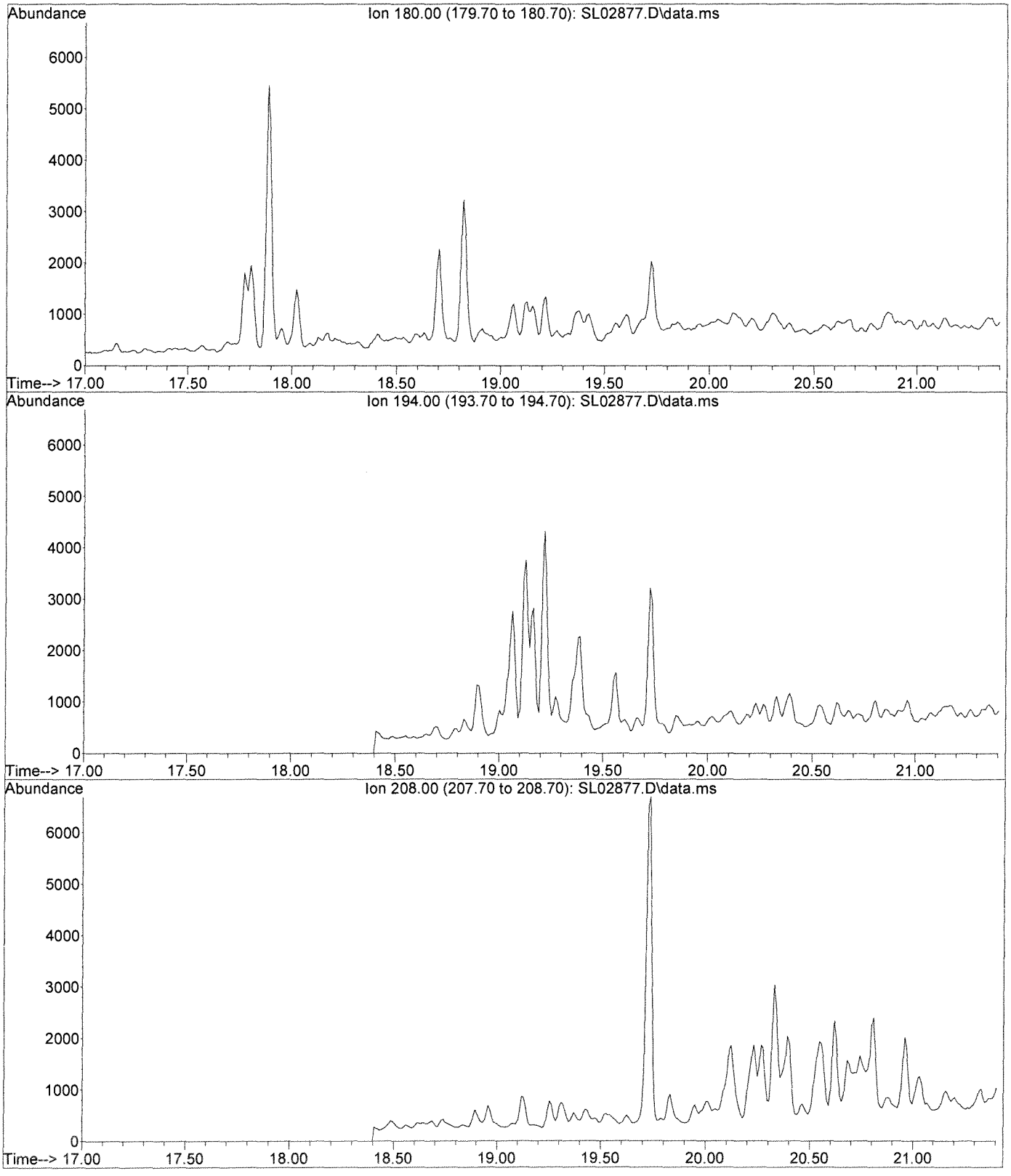
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```



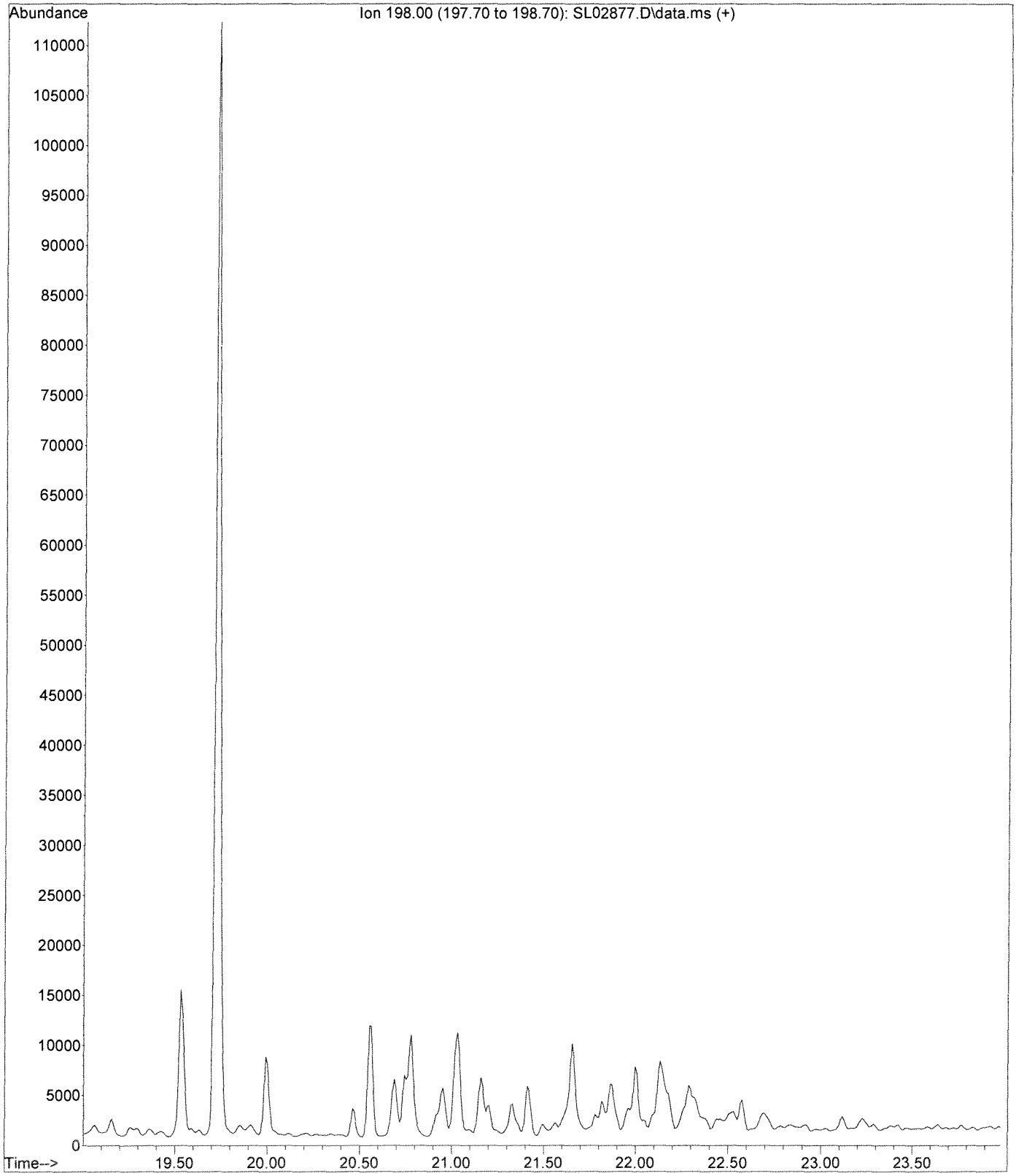
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



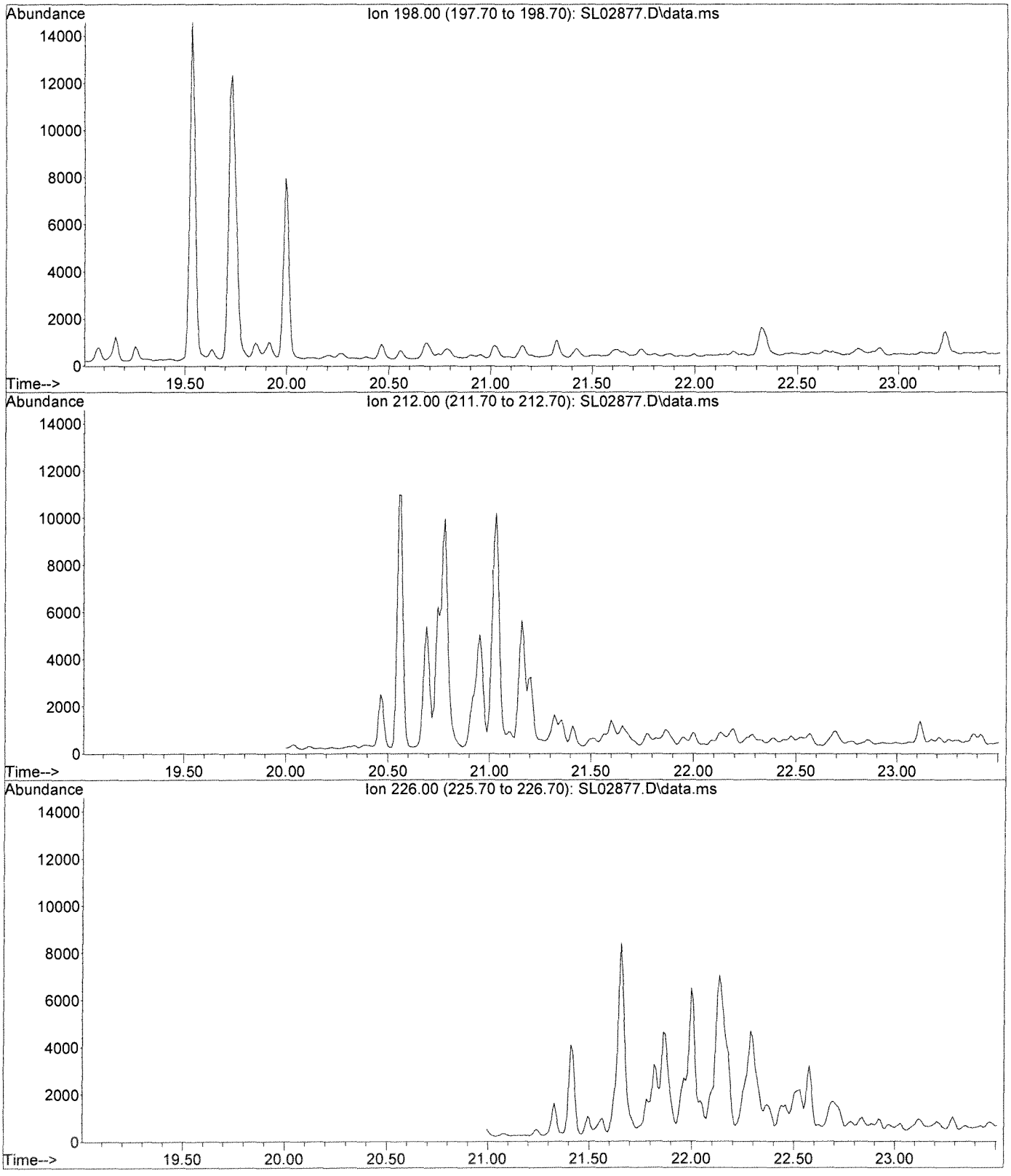
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



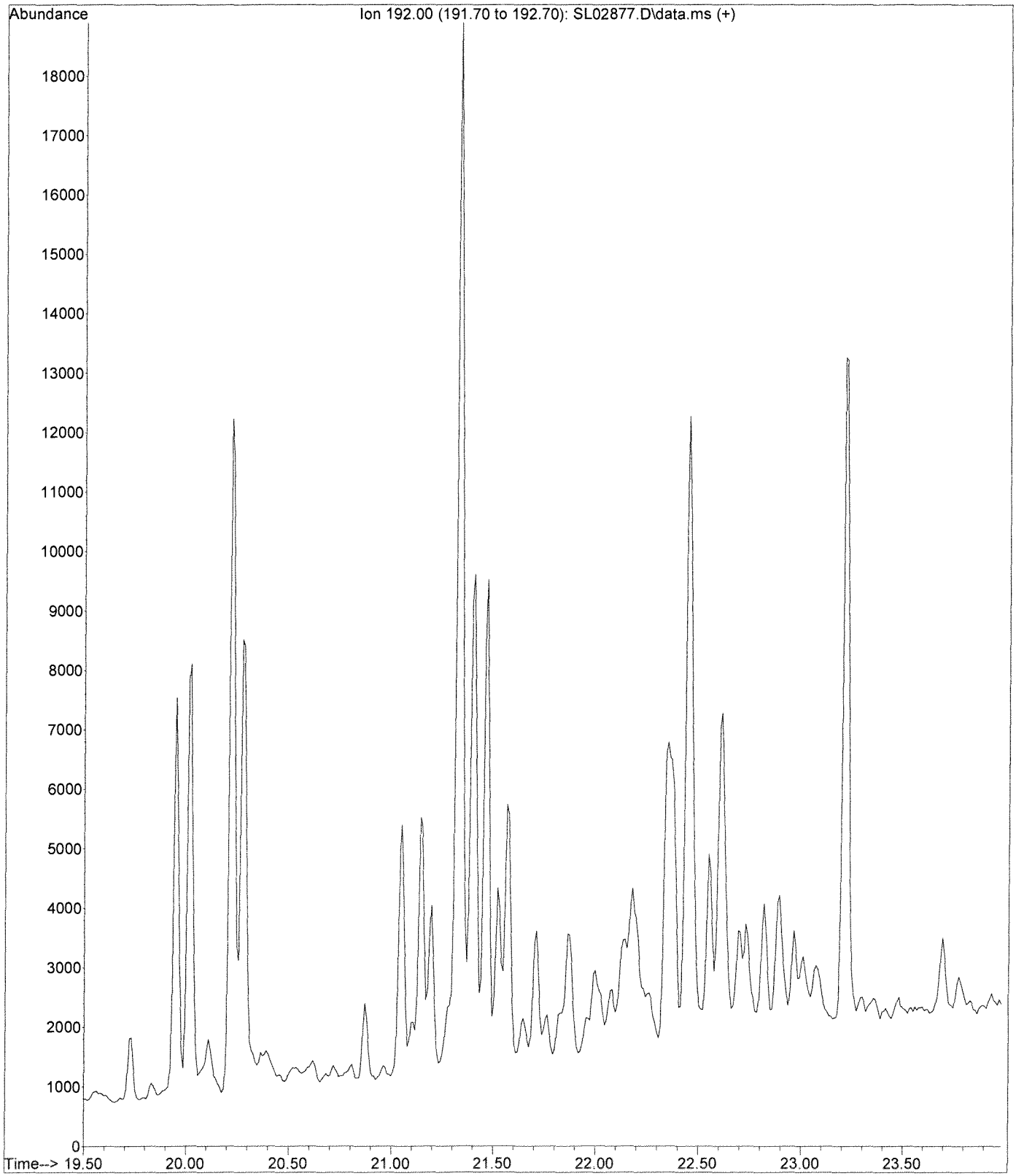
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```



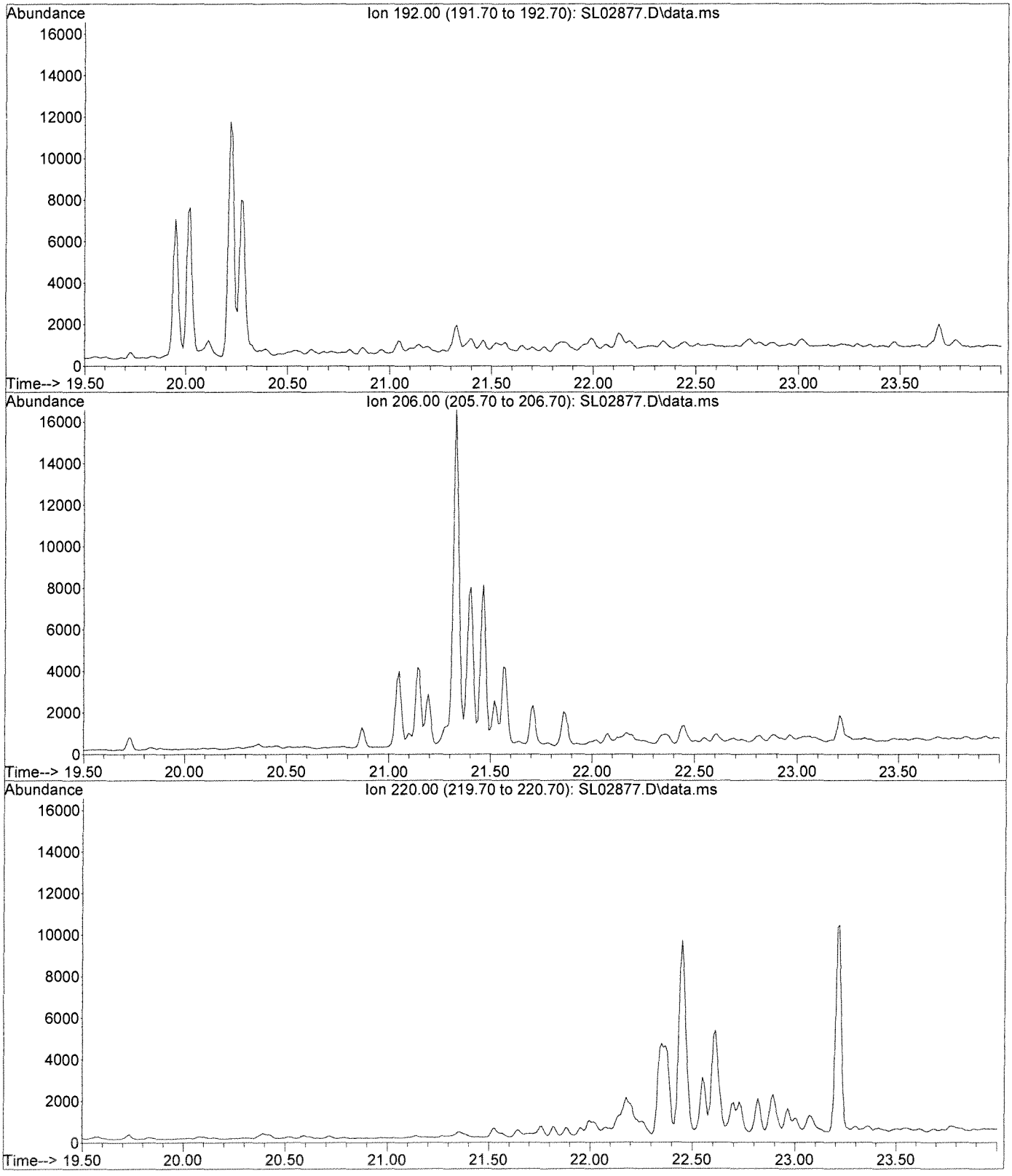
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



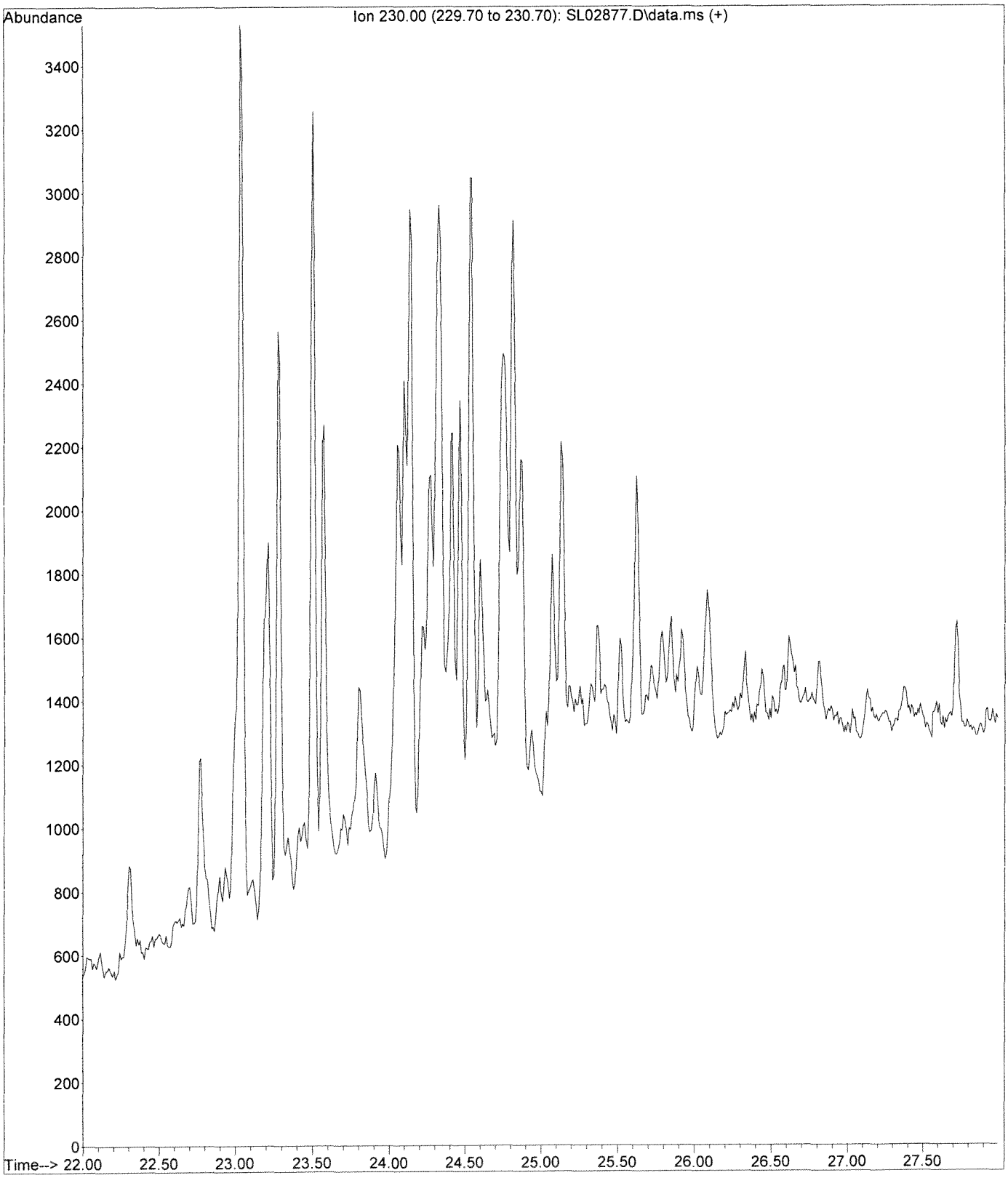
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```



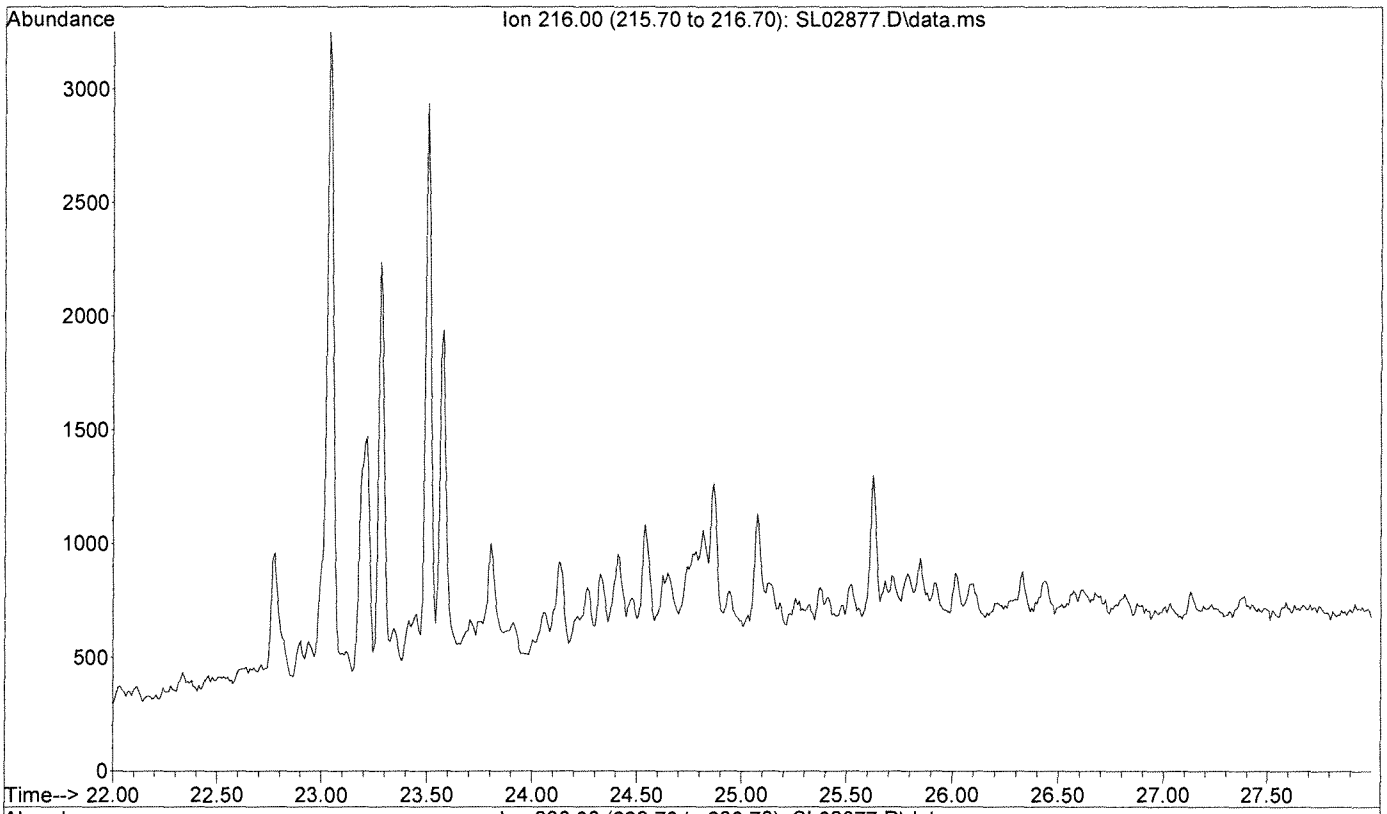
US EPA ARCHIVE DOCUMENT

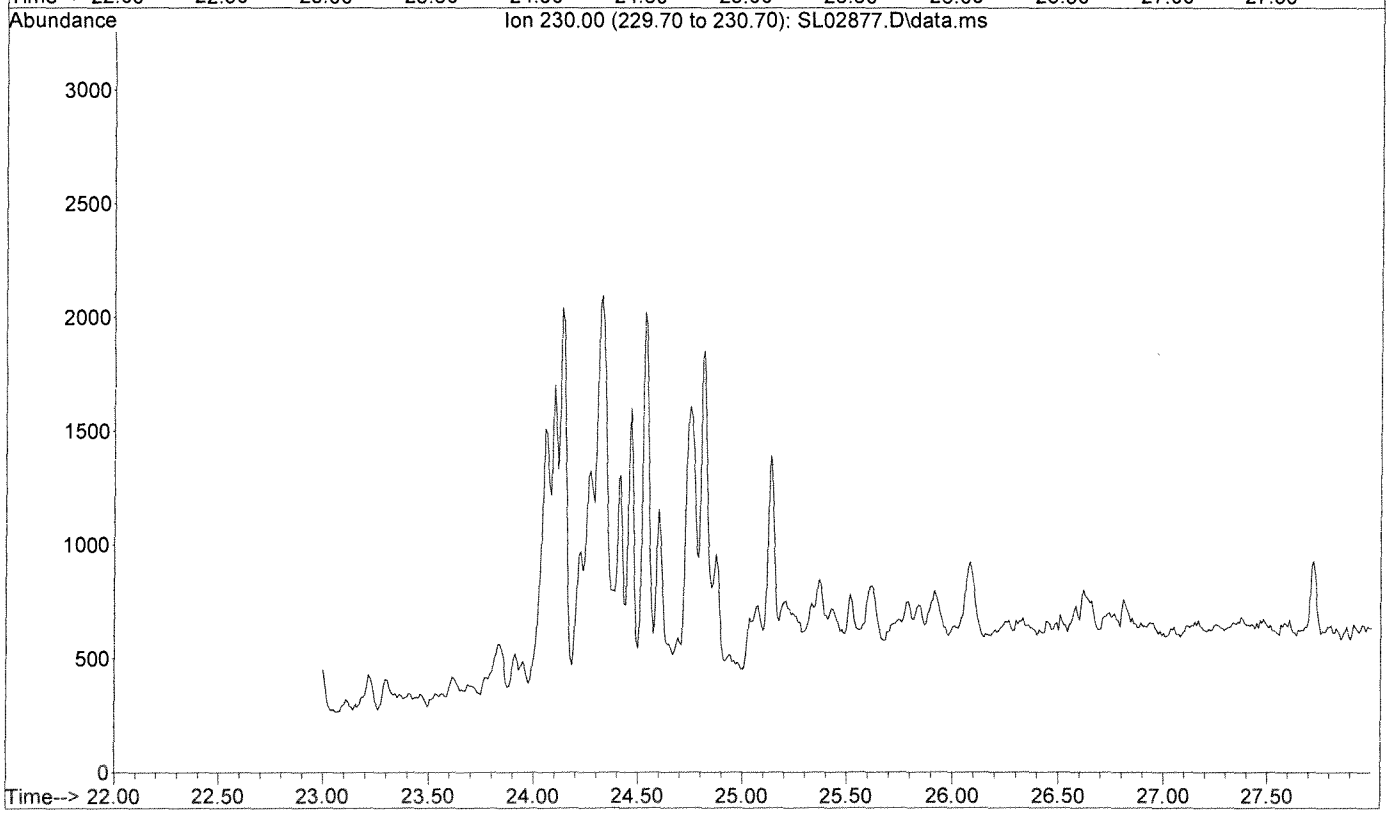
```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```



US EPA ARCHIVE DOCUMENT

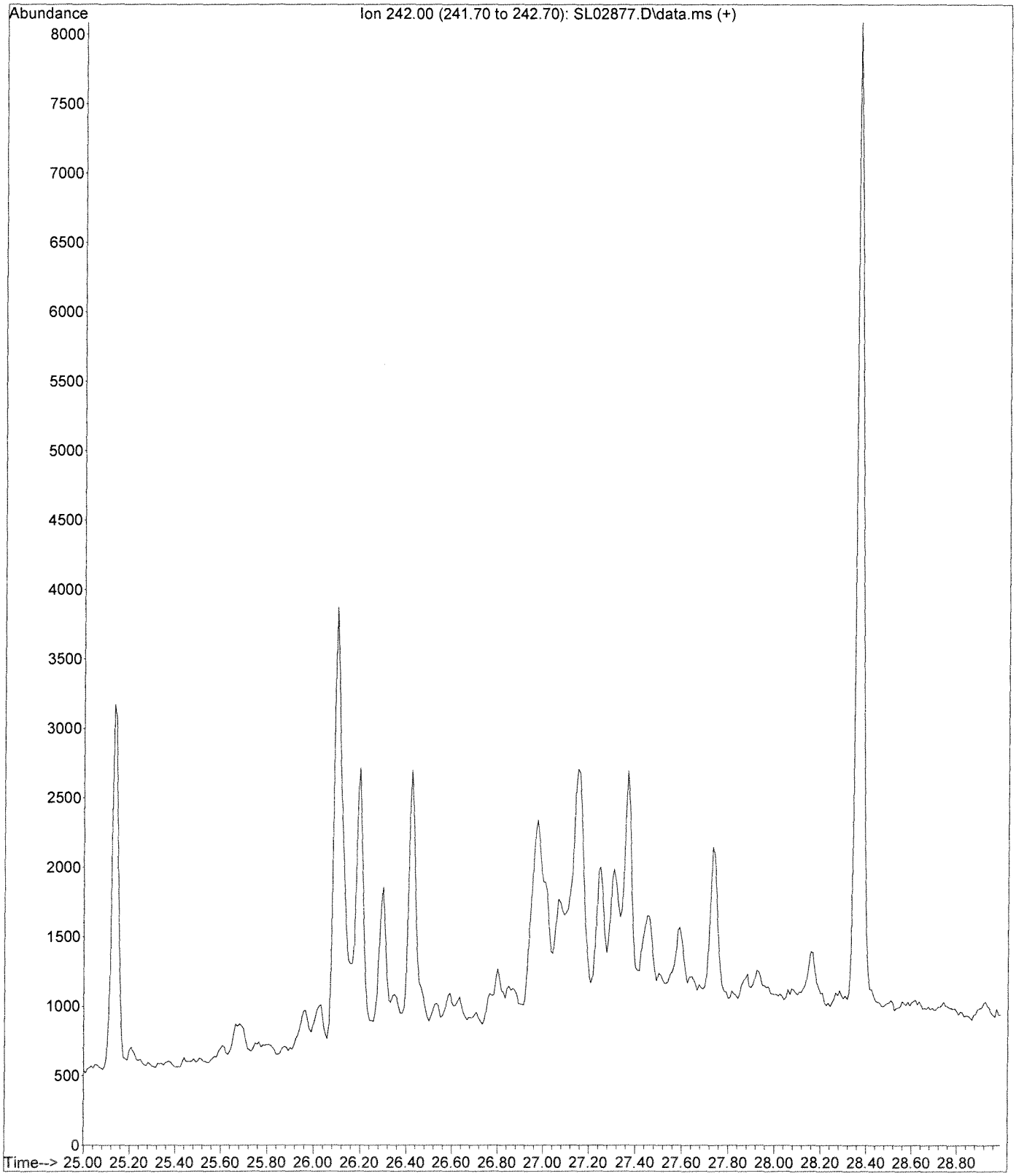
```
File       :C:\msdchem\1\DATA\022712\SL02877.D
Operator   : Syslo
Acquired   : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info  : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```





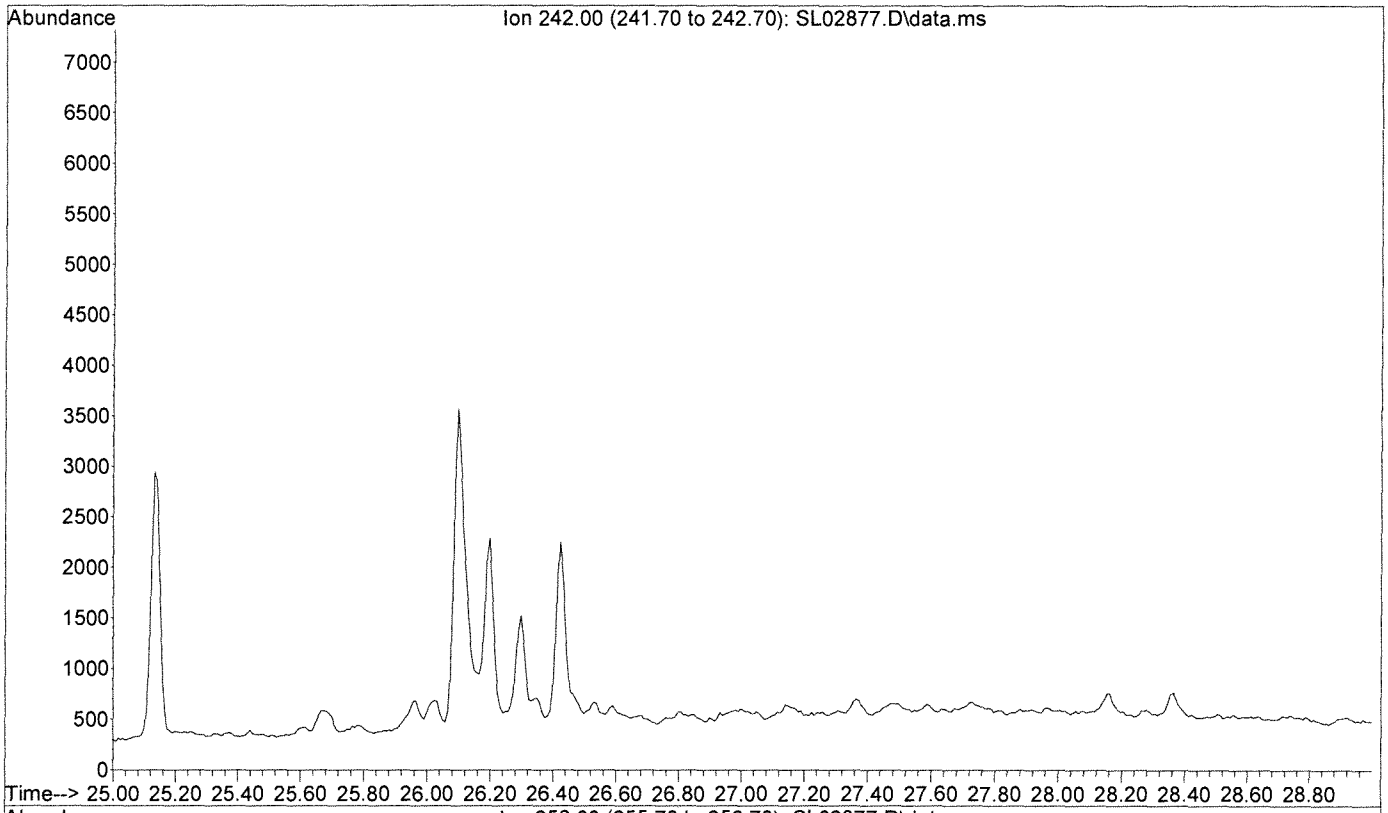
US EPA ARCHIVE DOCUMENT

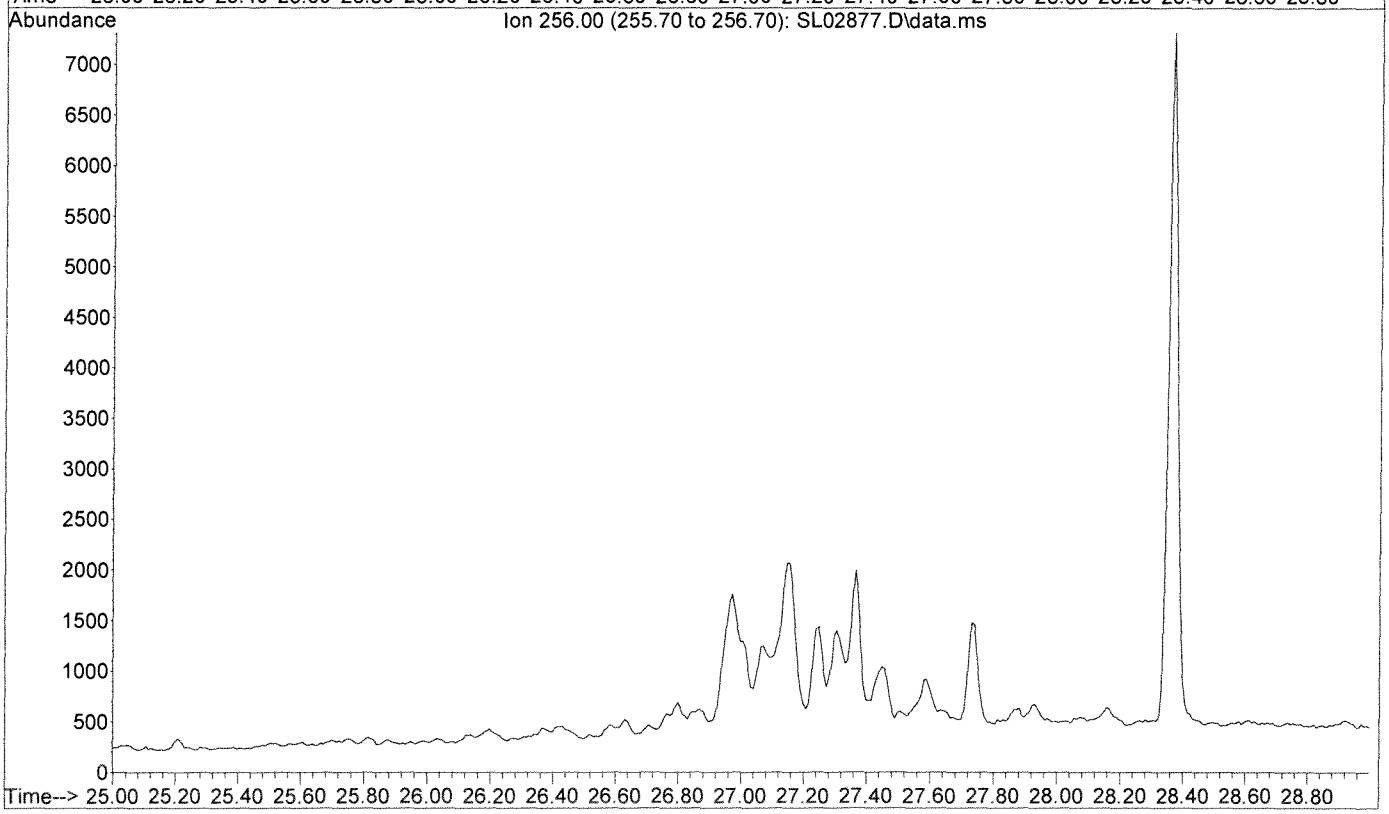
File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02877.D
Operator    : Syslo
Acquired    : 27 Feb 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0004
Misc Info   : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4
```





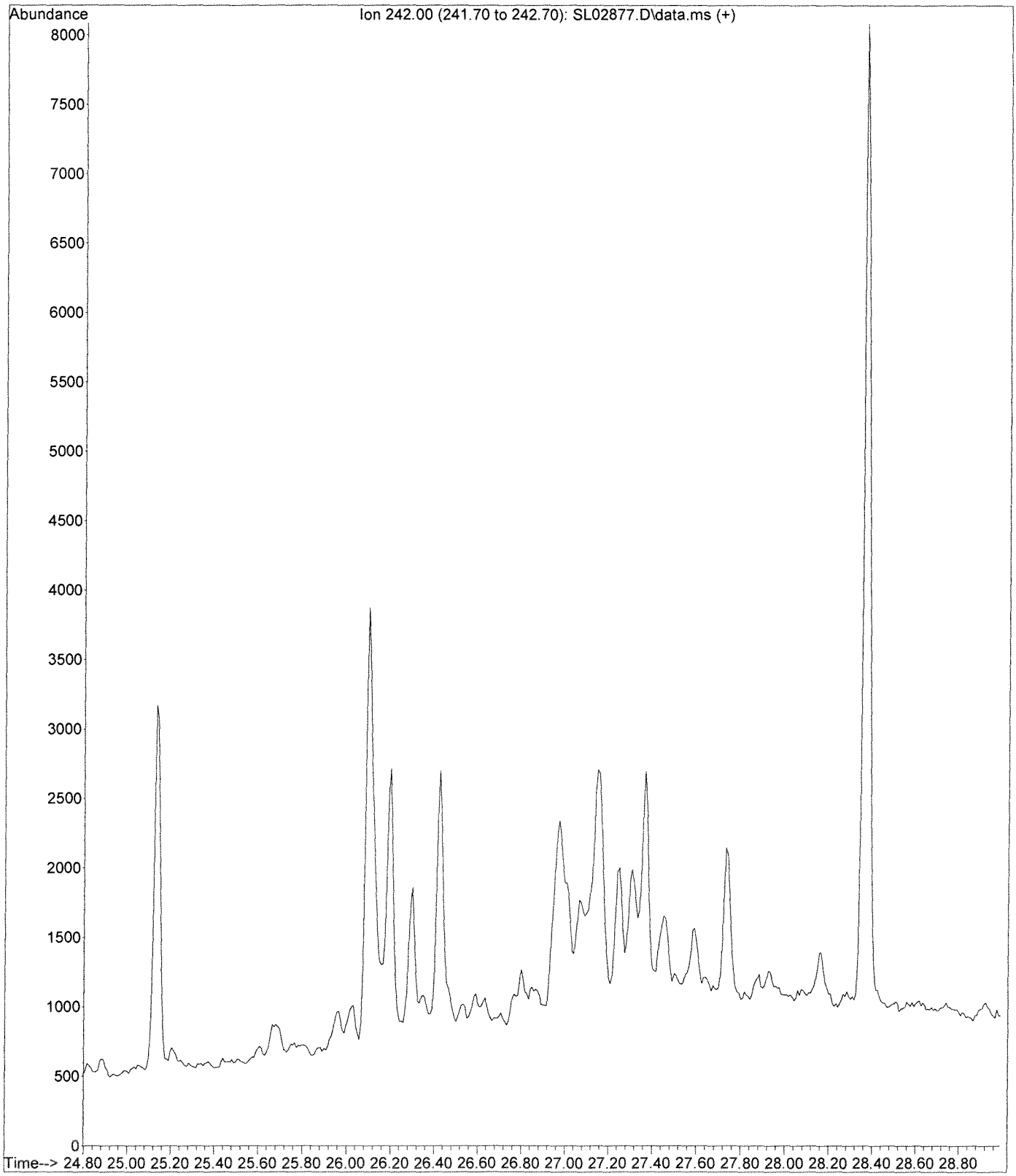
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02877.D
Operator : Syslo
Acquired : 27 Feb 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0004
Misc Info : 275-56-24 {0.029g in 2.0mL}
Vial Number: 4



US EPA ARCHIVE DOCUMENT

GLOBULE FROM Bucket

SERAS-017-000X

SL02878.D

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02878.D
Acq On : 27 Feb 2012 22:38
Operator : Syslo
Sample : SERAS-017-000x
Misc : Bucket 2 "qual skim"
ALS Vial : 5 Sample Multiplier: 1

Quant Time: Mar 01 13:14:15 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	698465	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	227918	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	283369	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.603	66	257581	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	0.000	188	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
3) d14-o-Terphenyl {S}	0.000	244	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
4) 5a-Androstane {S}	0.000	260	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
7) d62-Triacontane {S}	0.000	66	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
Target Compounds						Qvalue

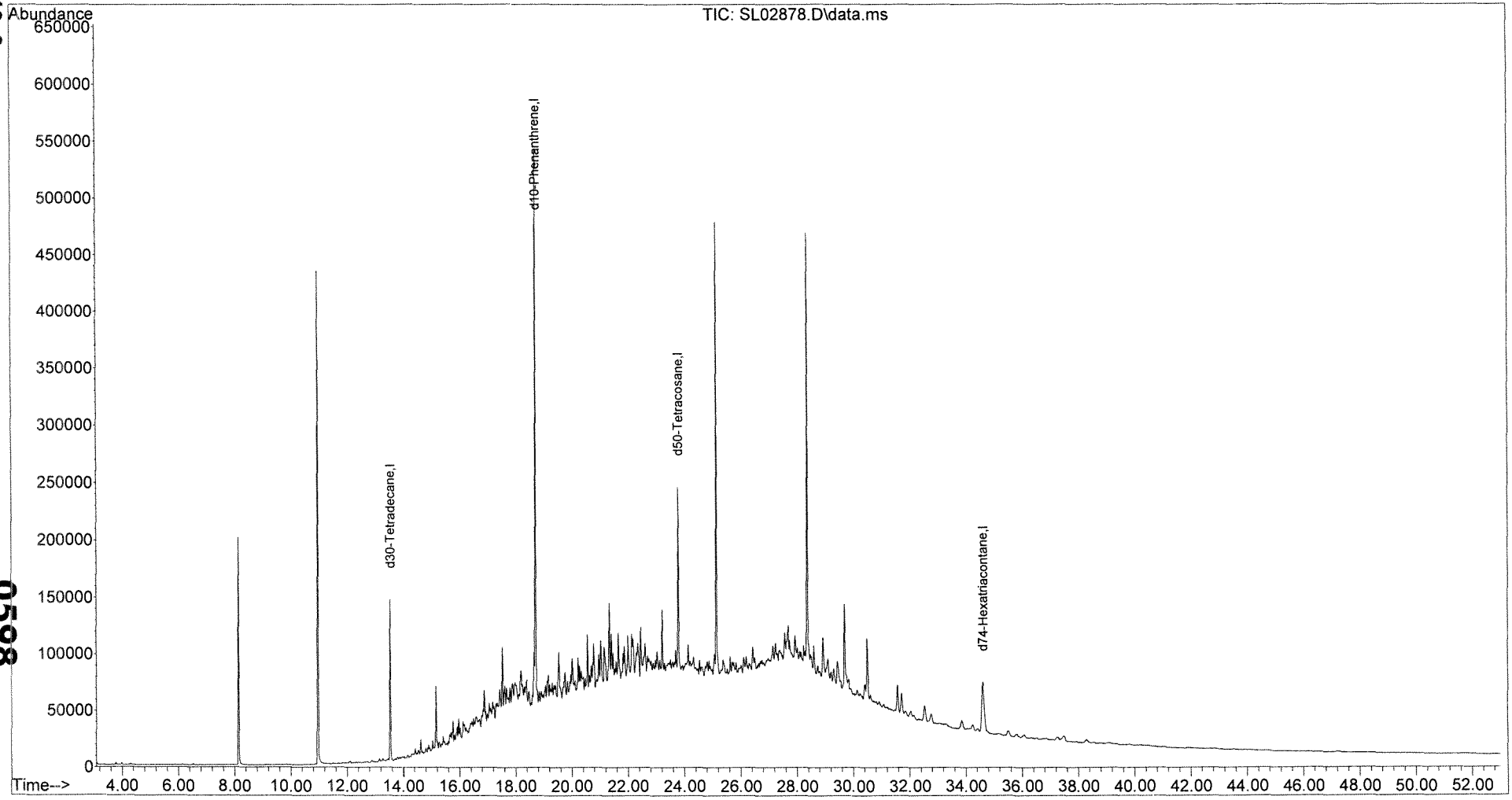
(#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02878.D
Acq On : 27 Feb 2012 22:38
Operator : Syslo
Sample : SERAS-017-000x
Misc : Bucket 2 "qual skim"
ALS Vial : 5 Sample Multiplier: 1

Quant Time: Mar 01 13:14:15 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0598

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

US EPA ARCHIVE DOCUMENT

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM.* Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

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The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

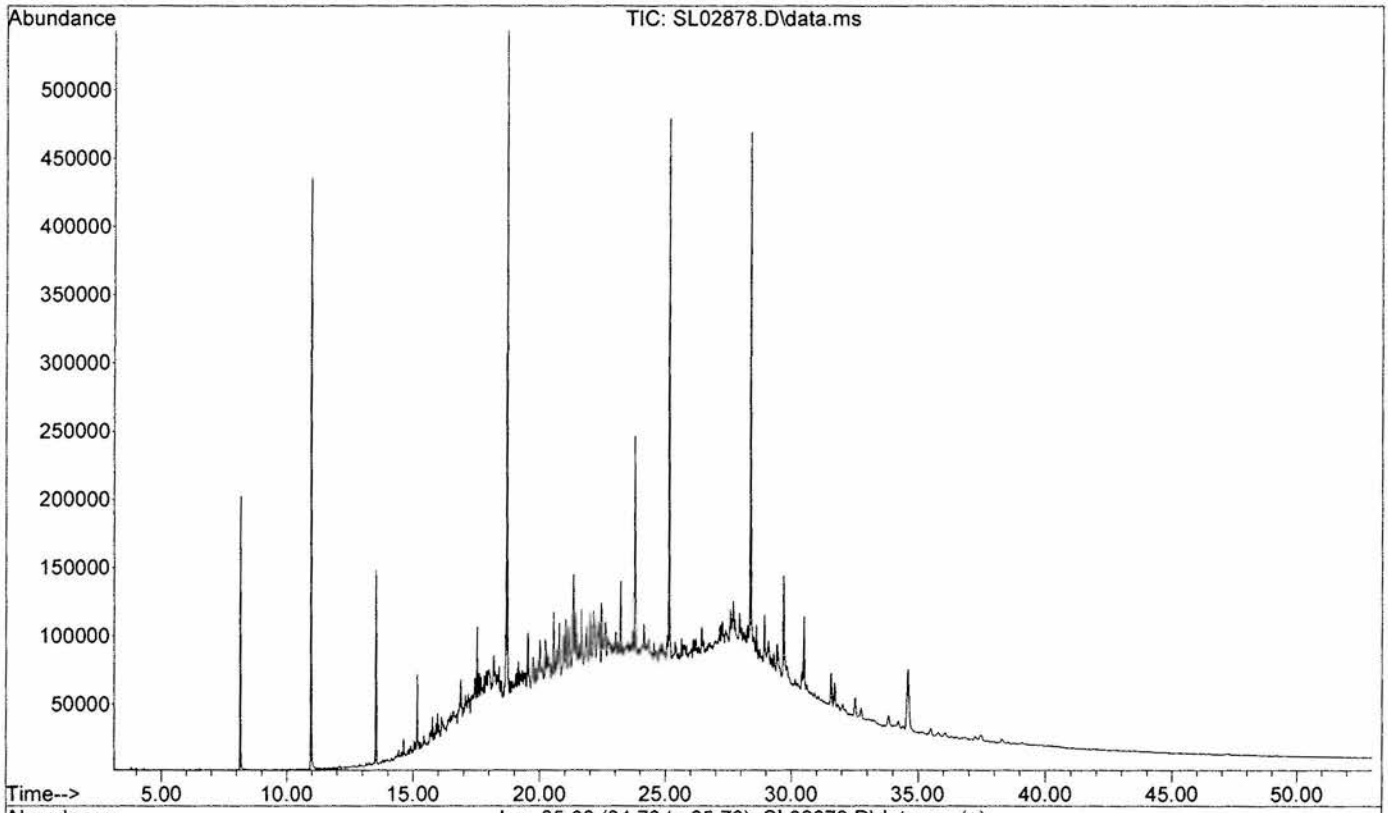
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

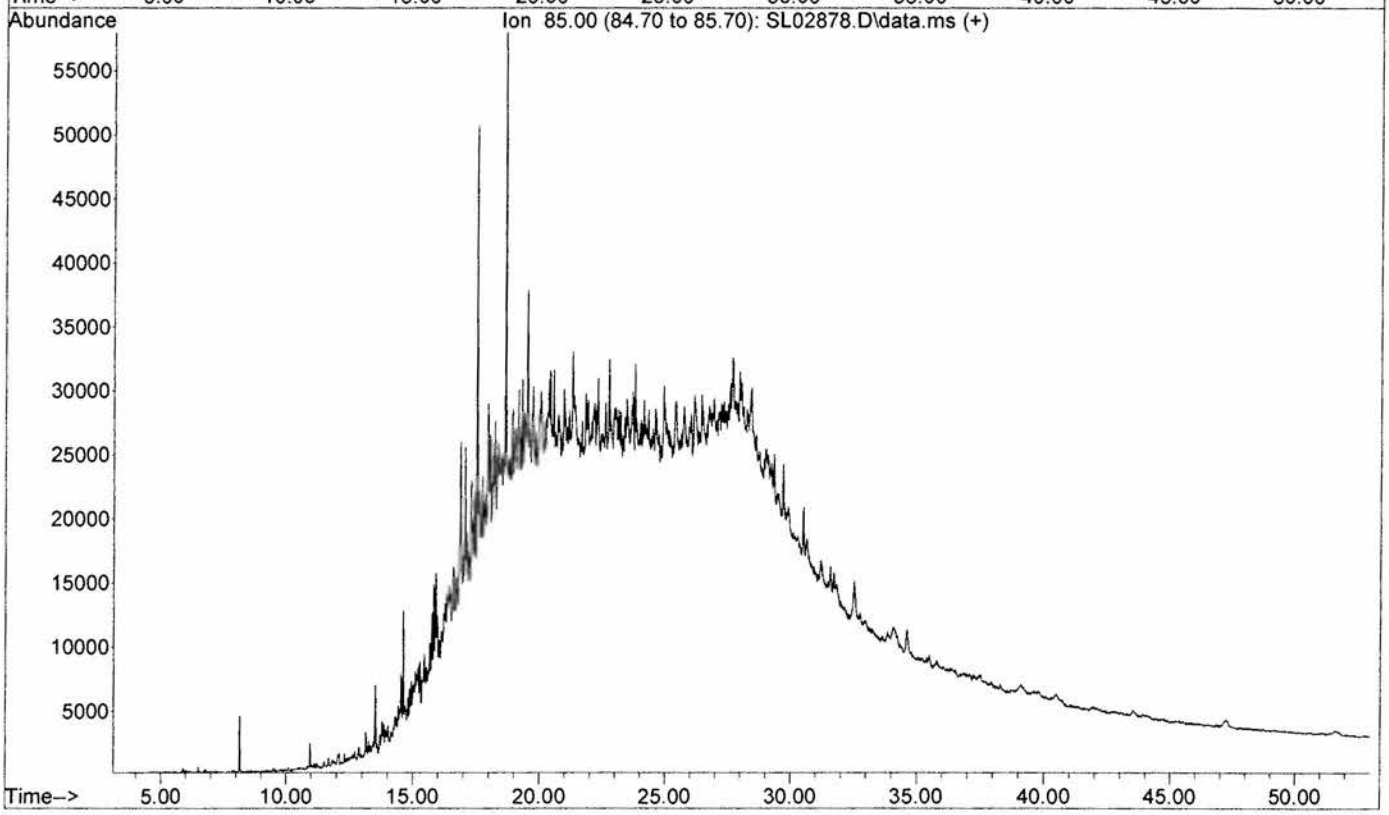
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5

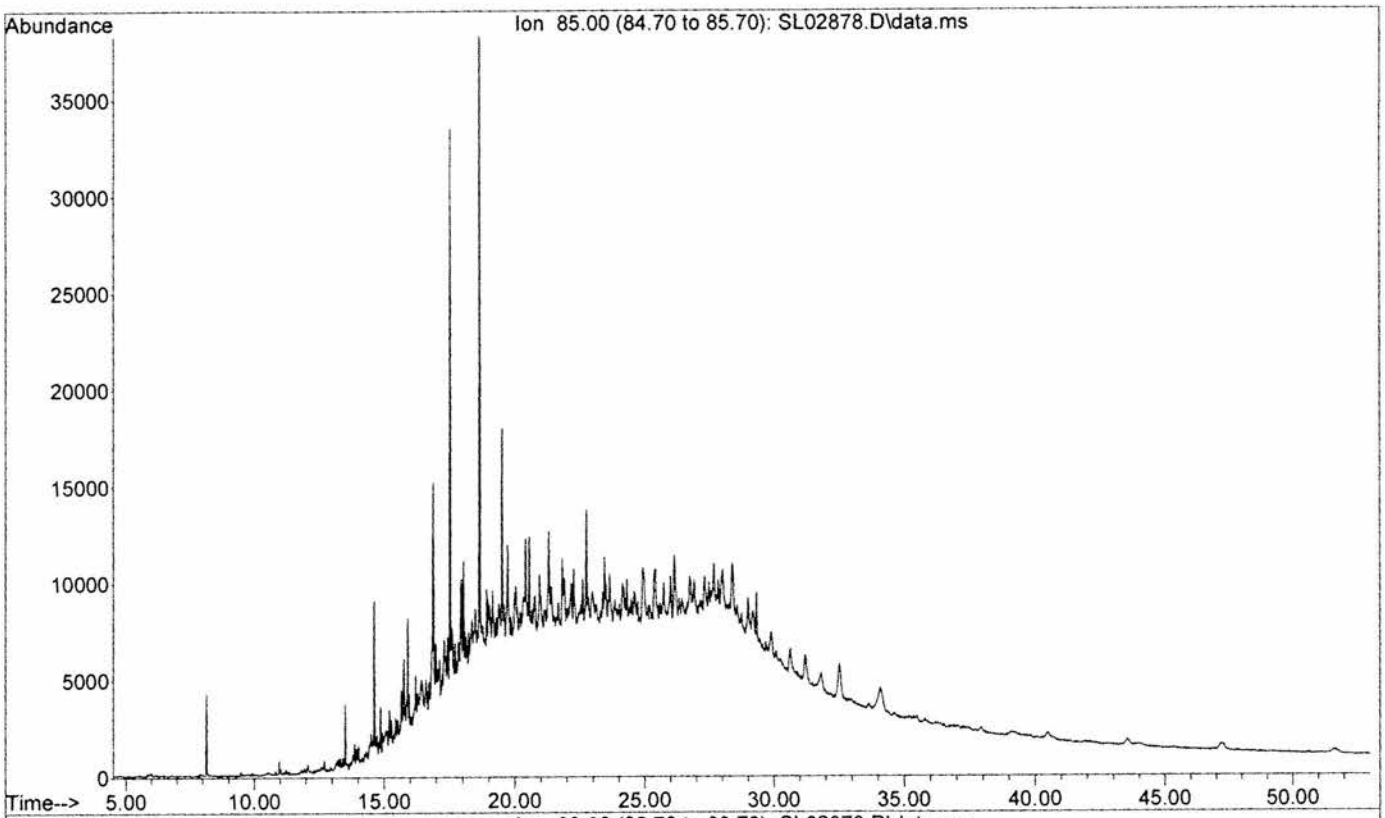


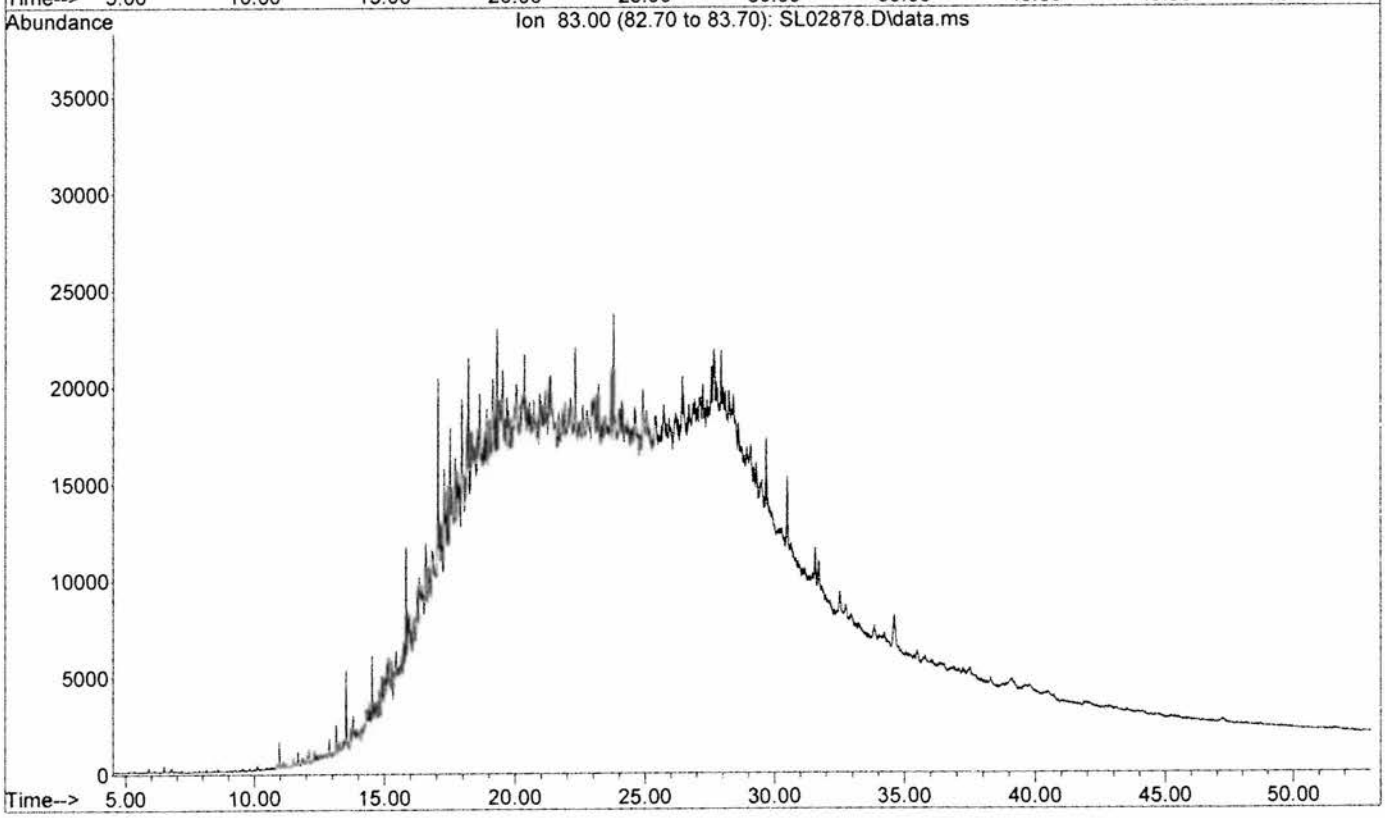


US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```

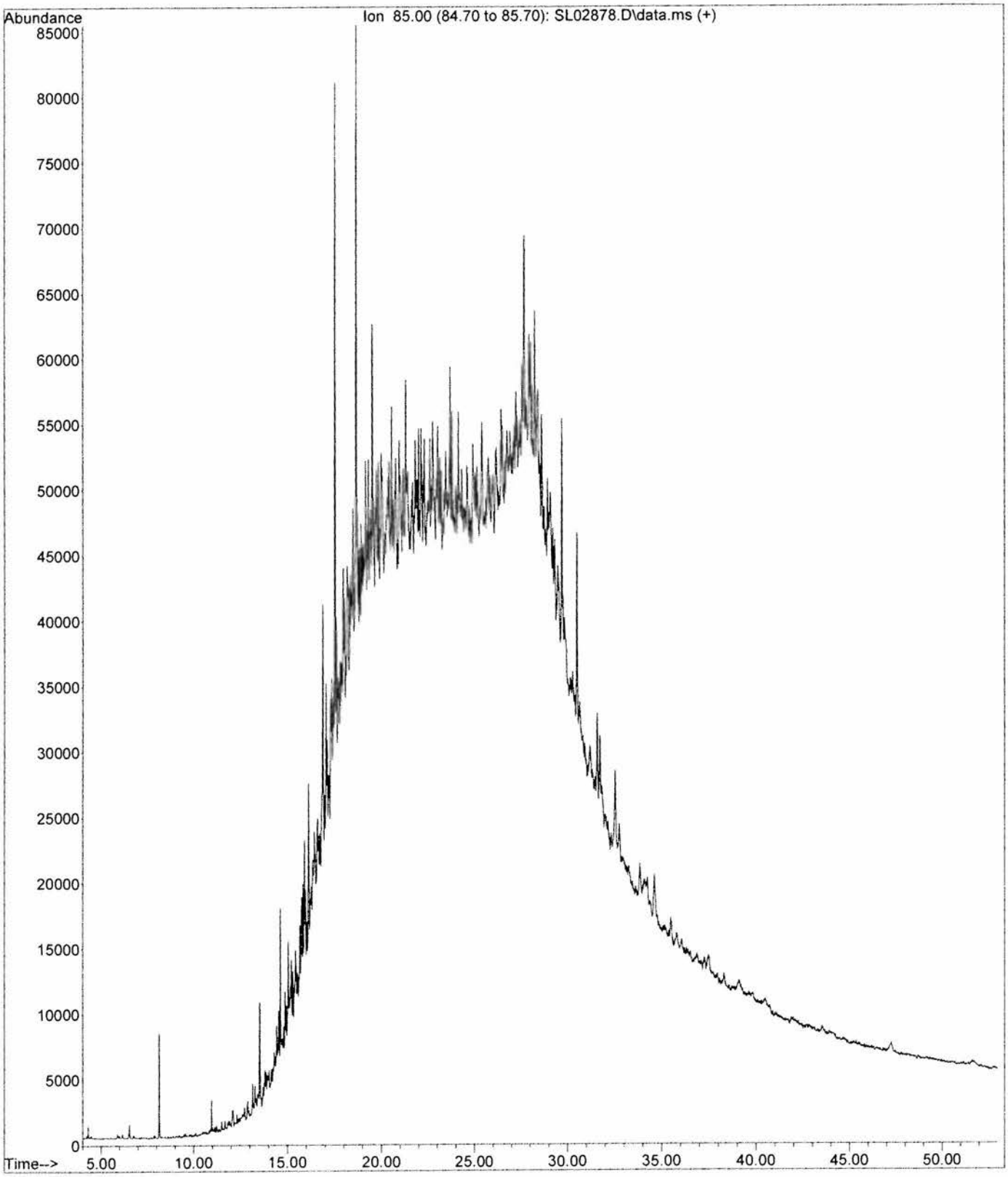




```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5



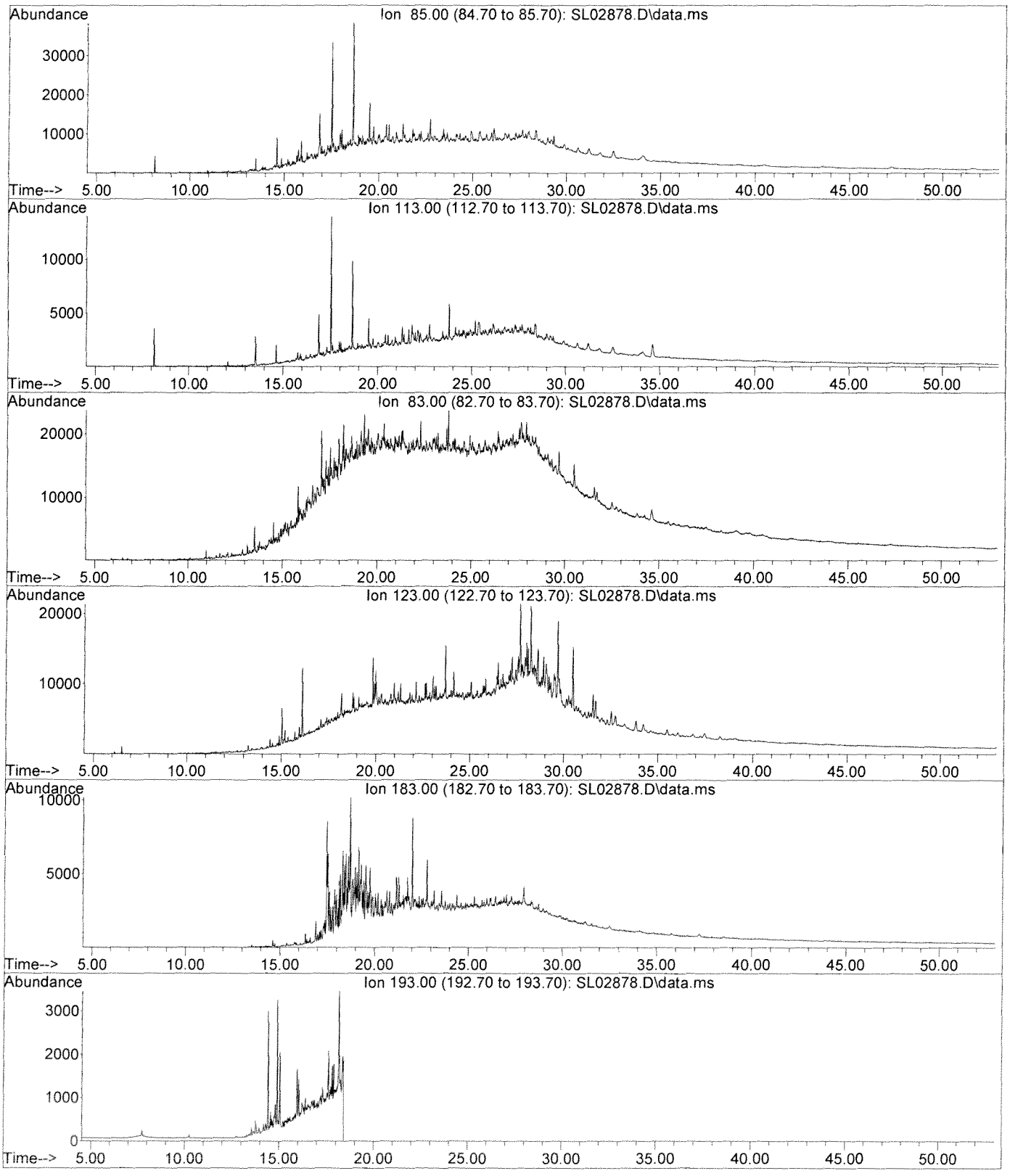
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



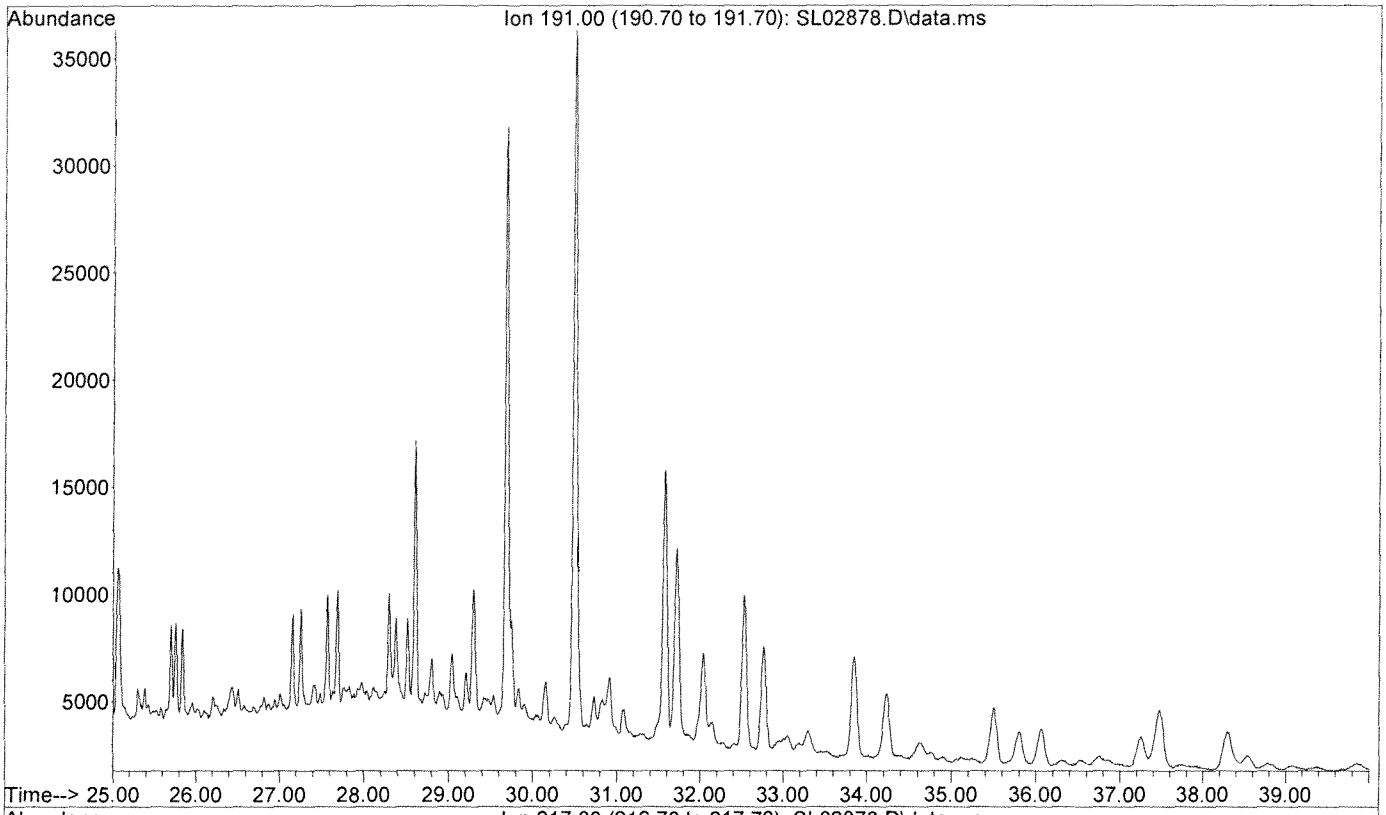
US EPA ARCHIVE DOCUMENT

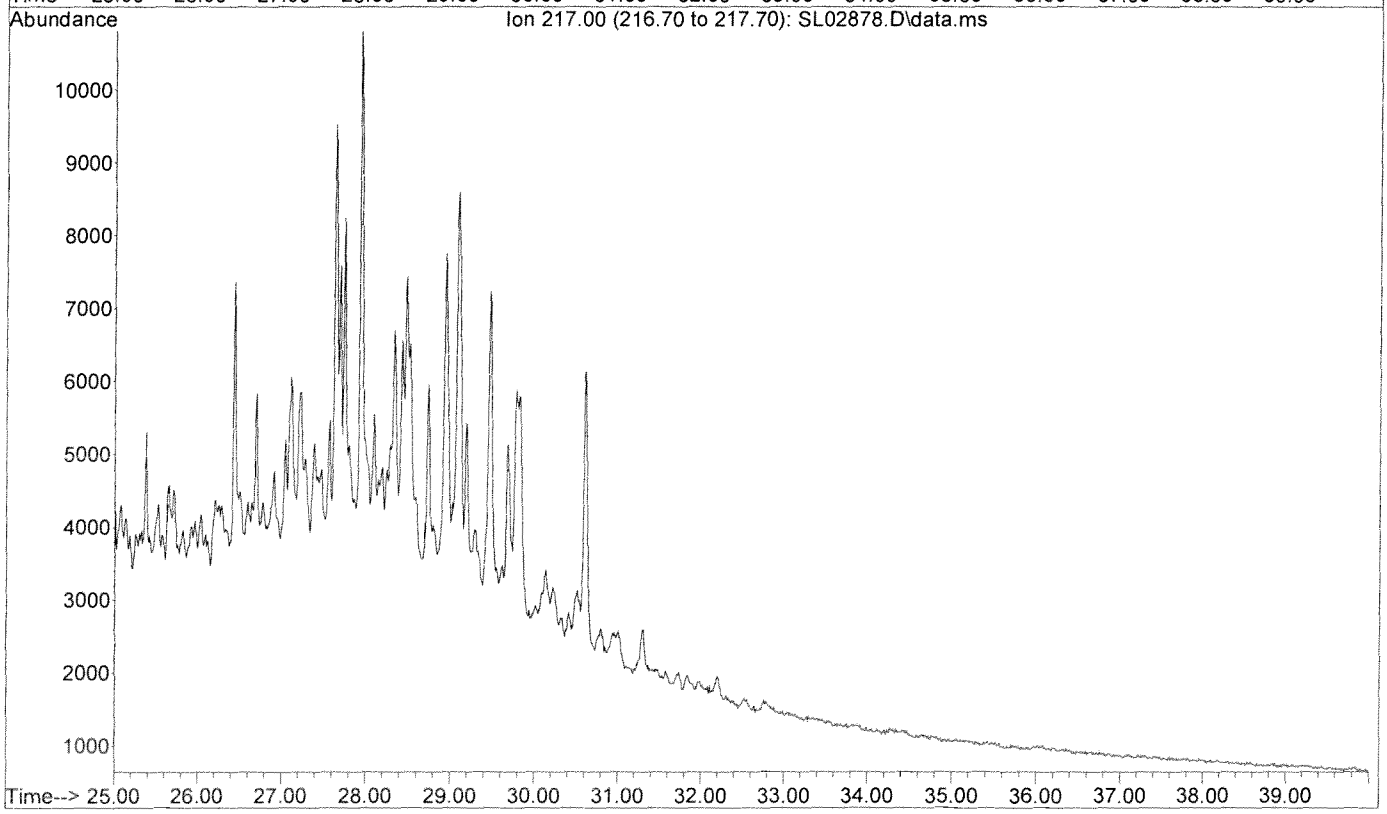
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```





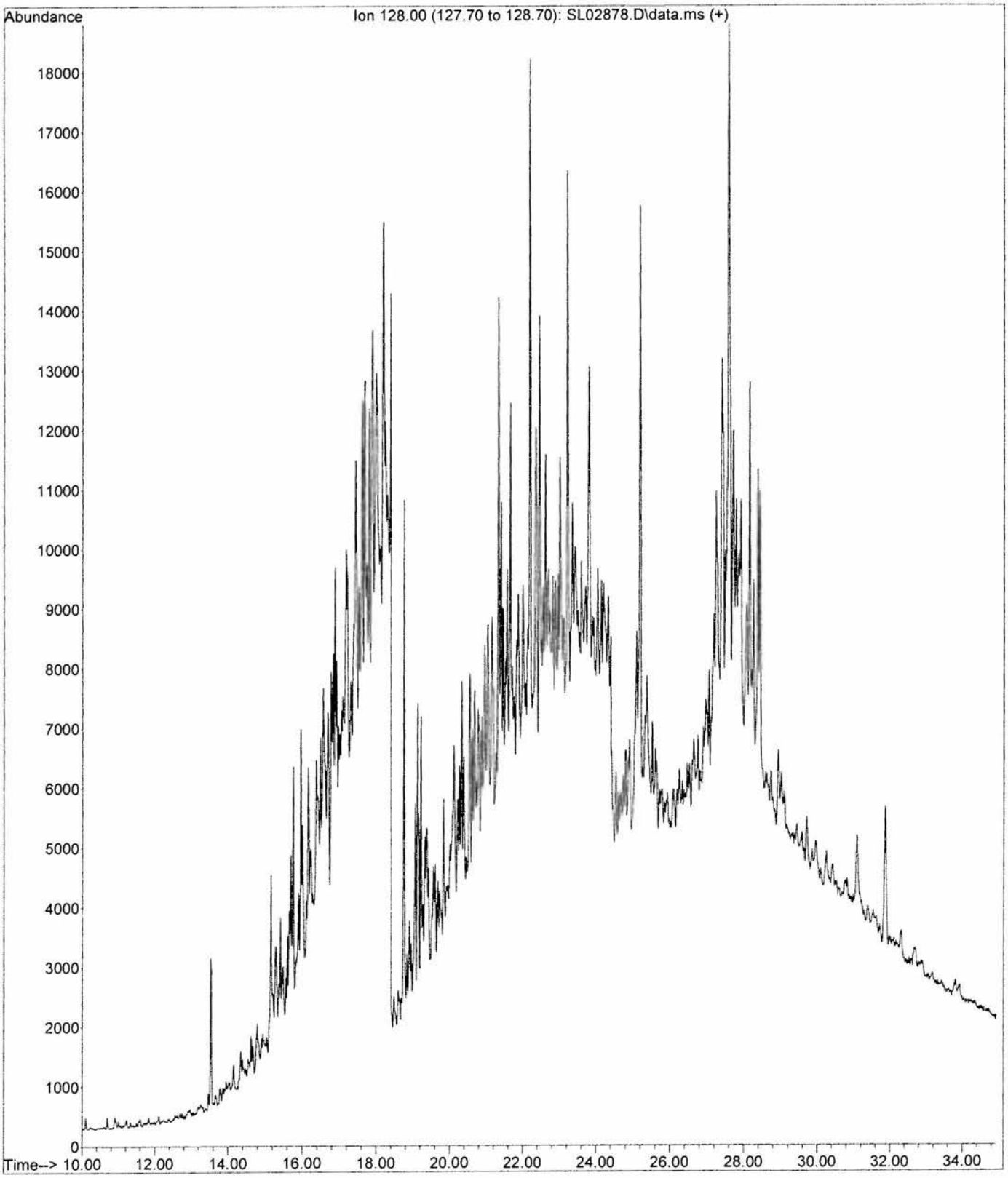
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5



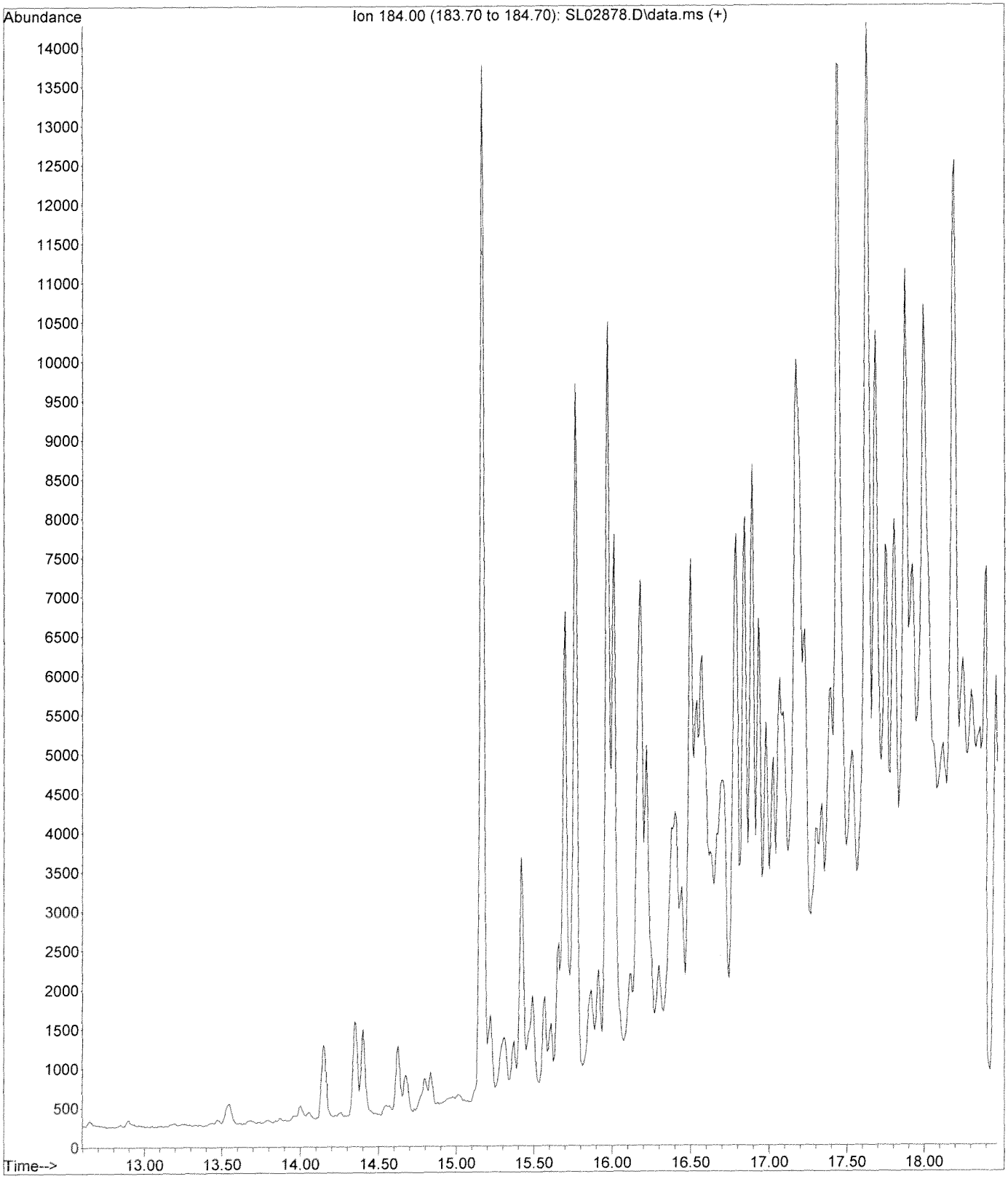
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

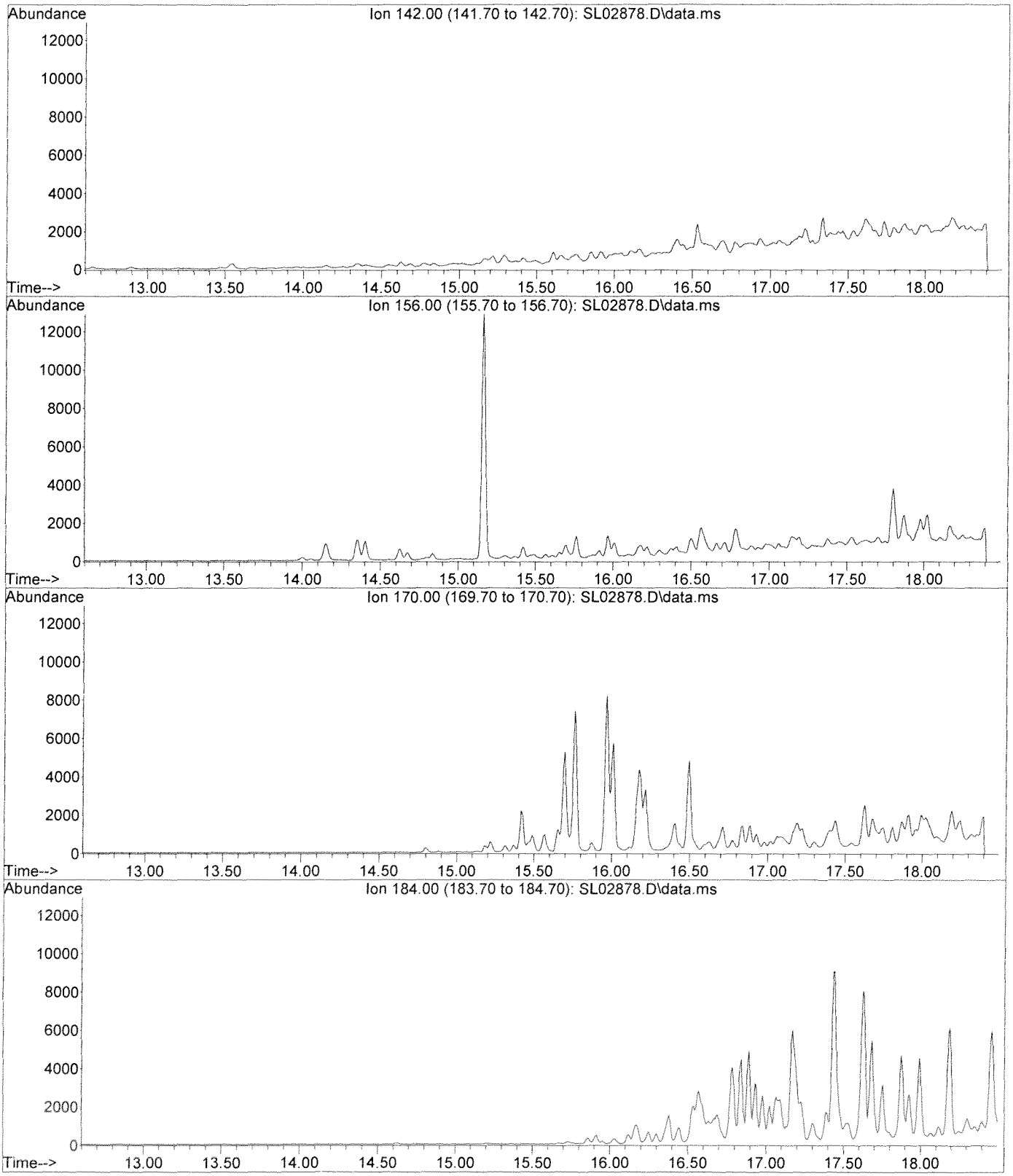
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5



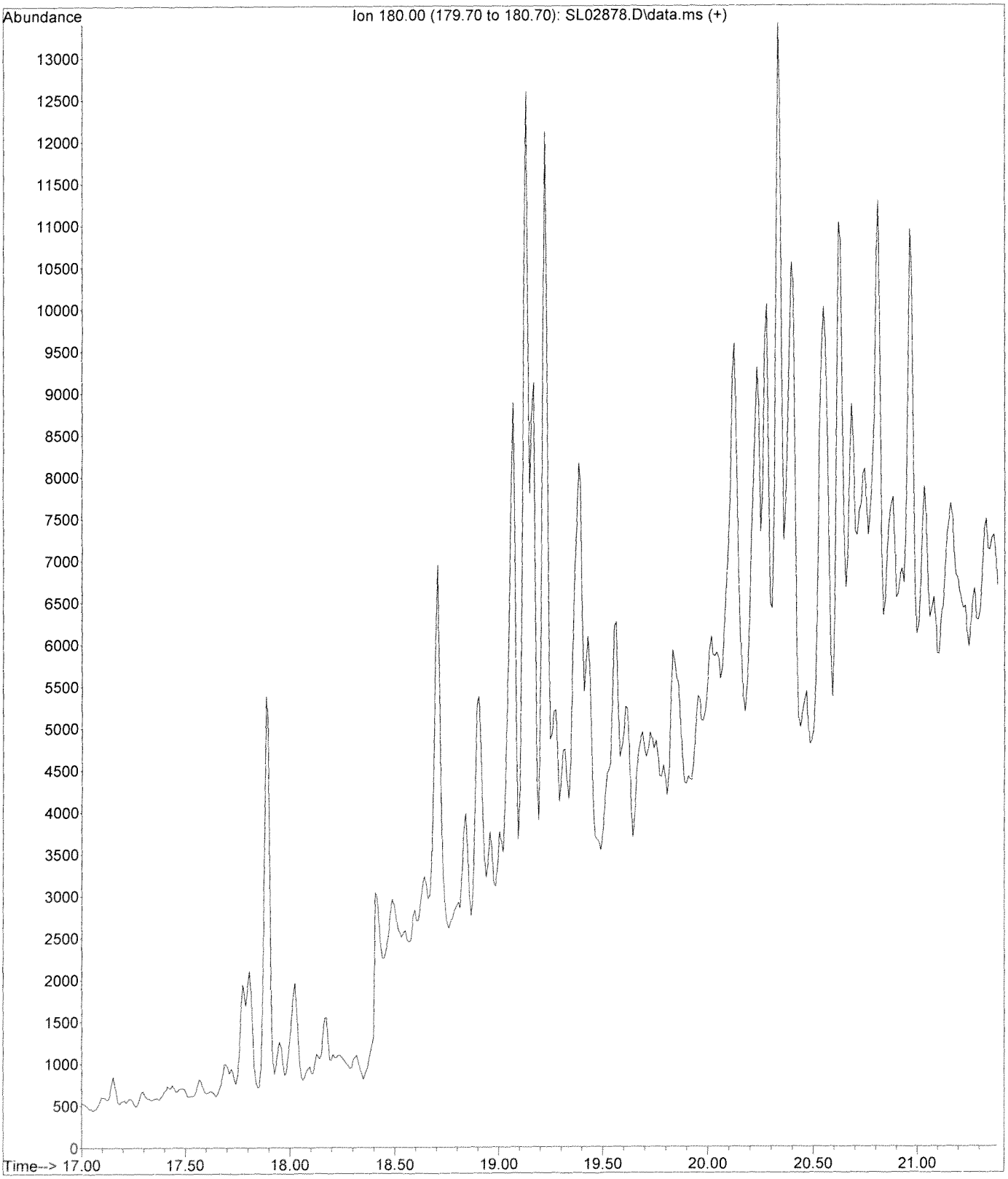
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



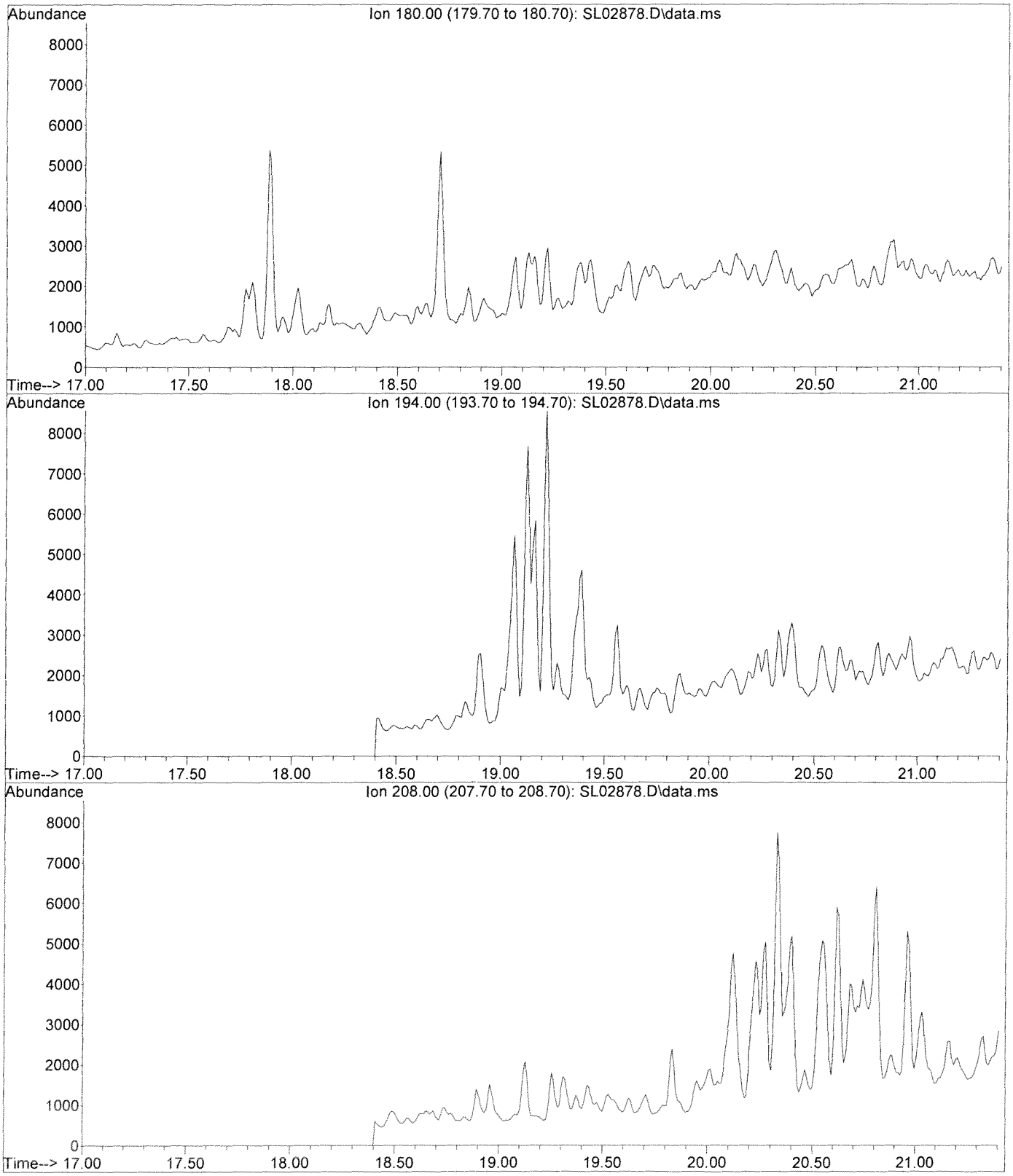
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5



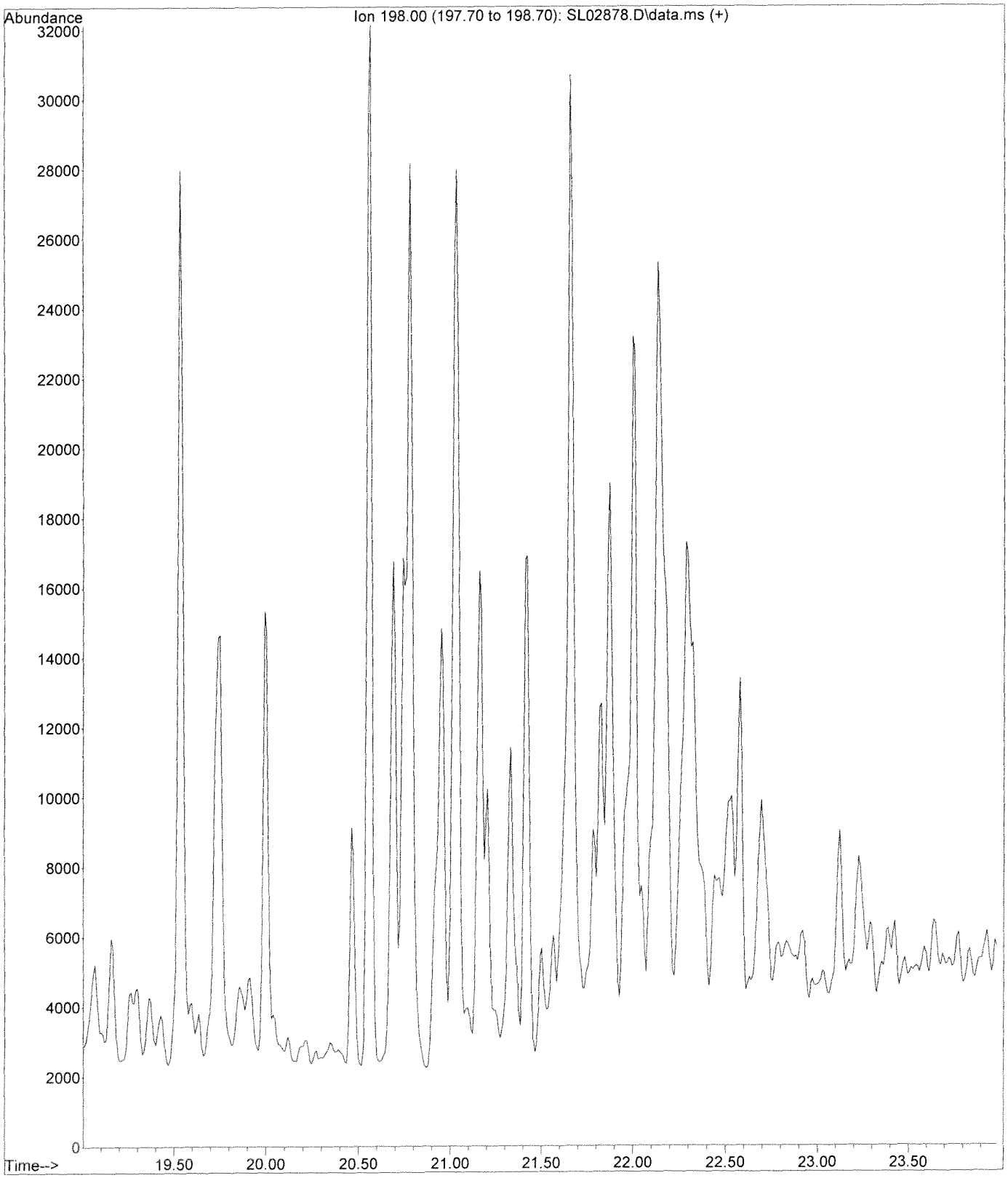
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5



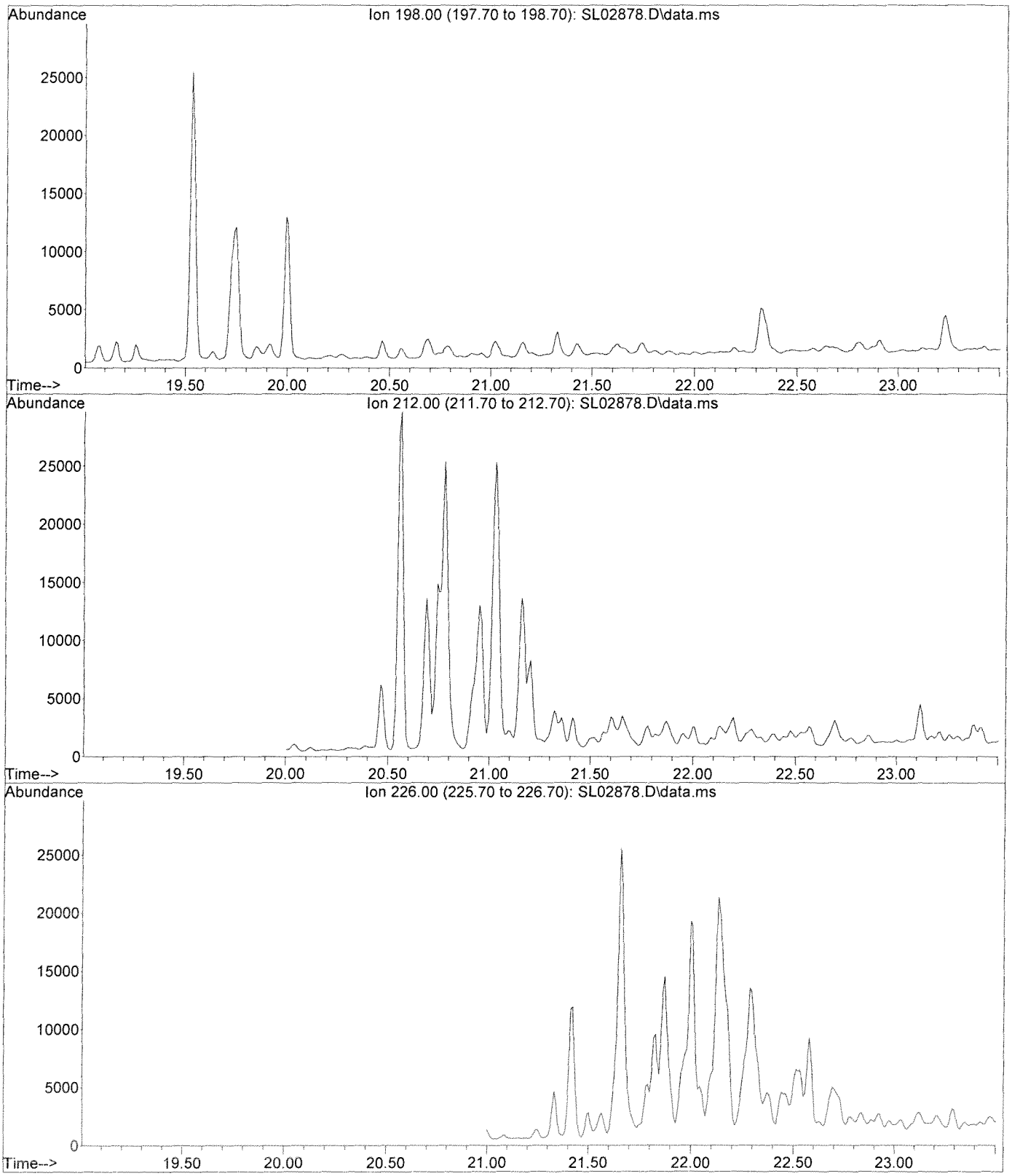
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02878.D
Operator : Syslo
Acquired : 27 Feb 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-000x
Misc Info : Bucket 2 "qual skim"
Vial Number: 5



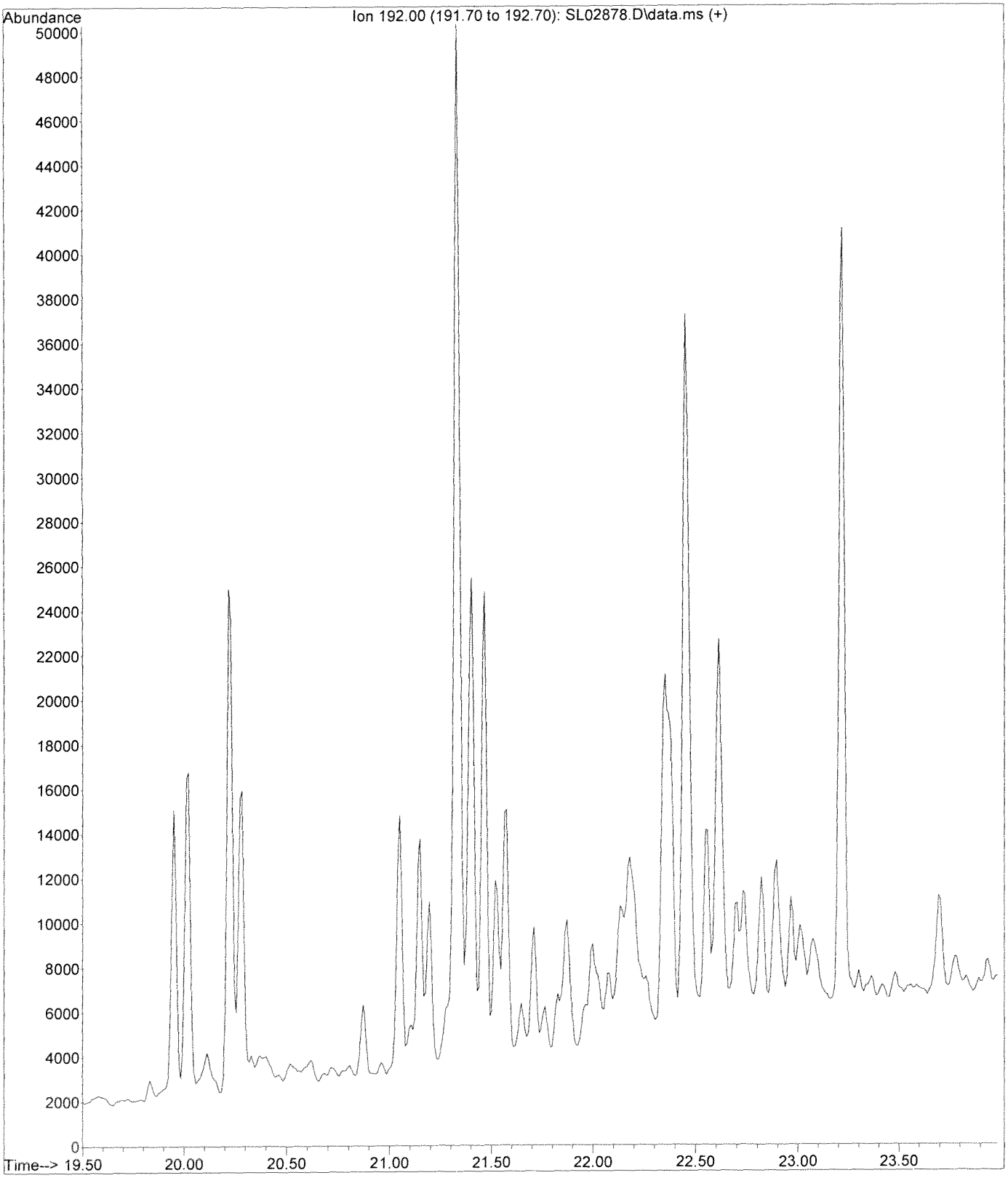
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



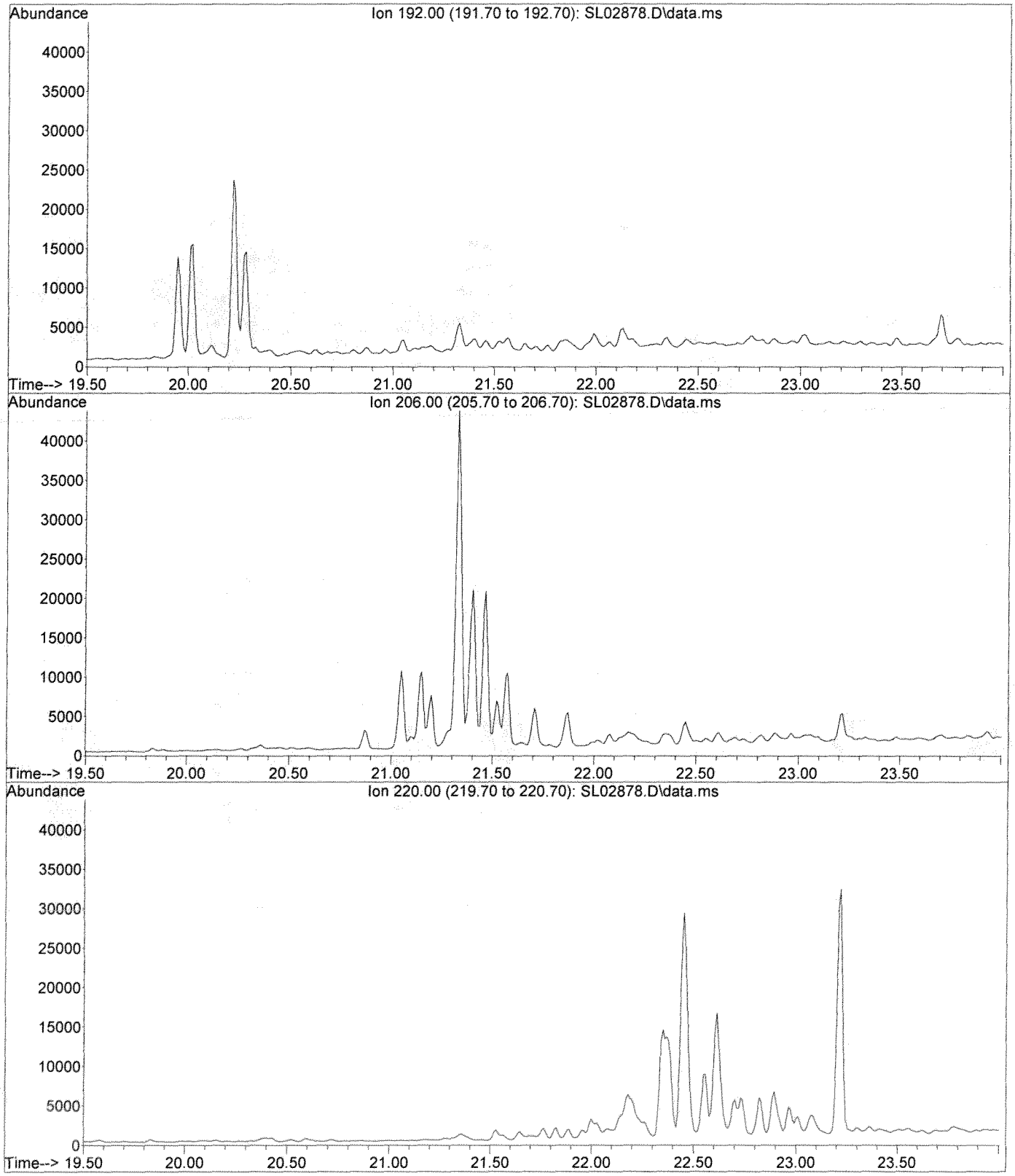
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



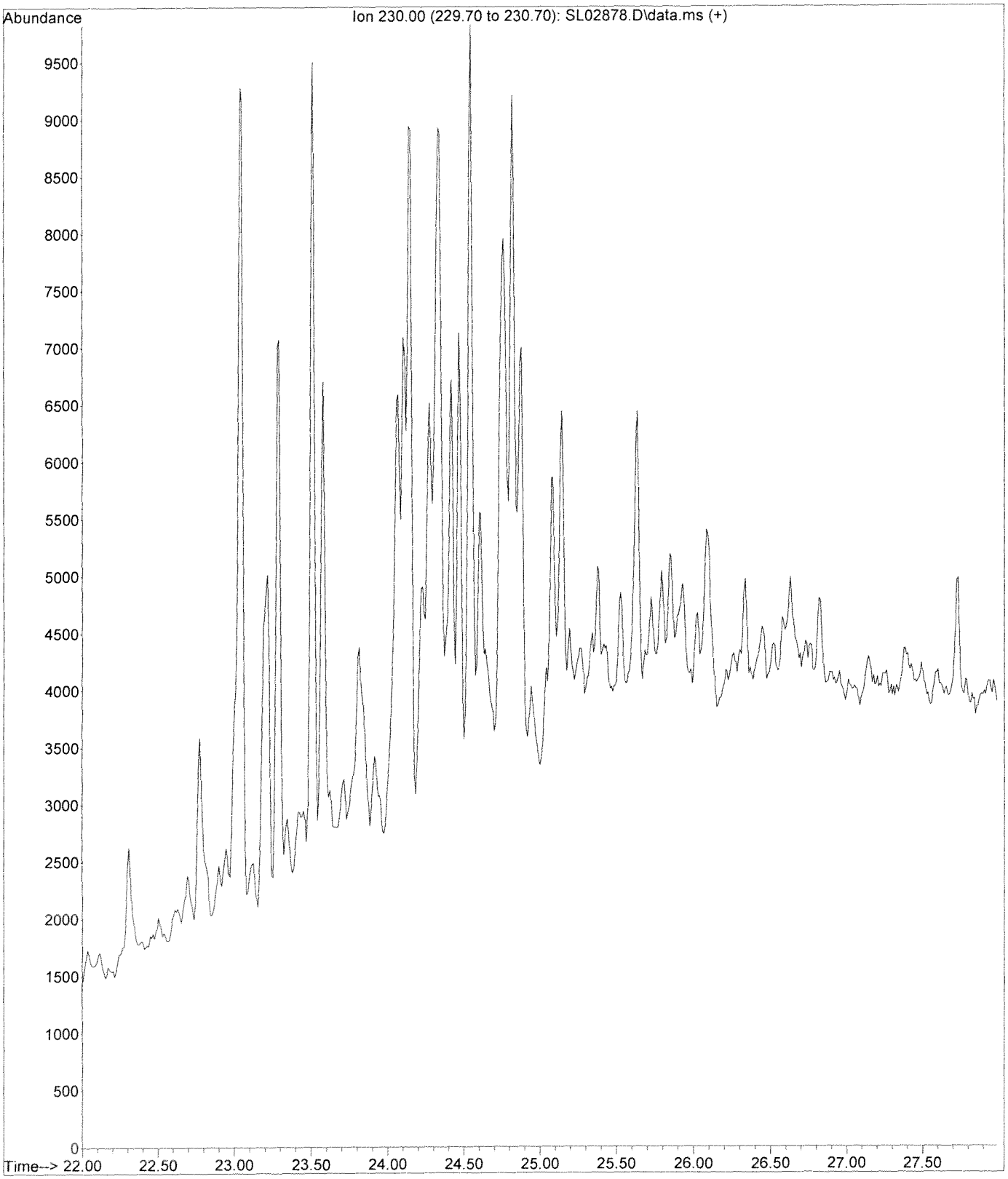
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



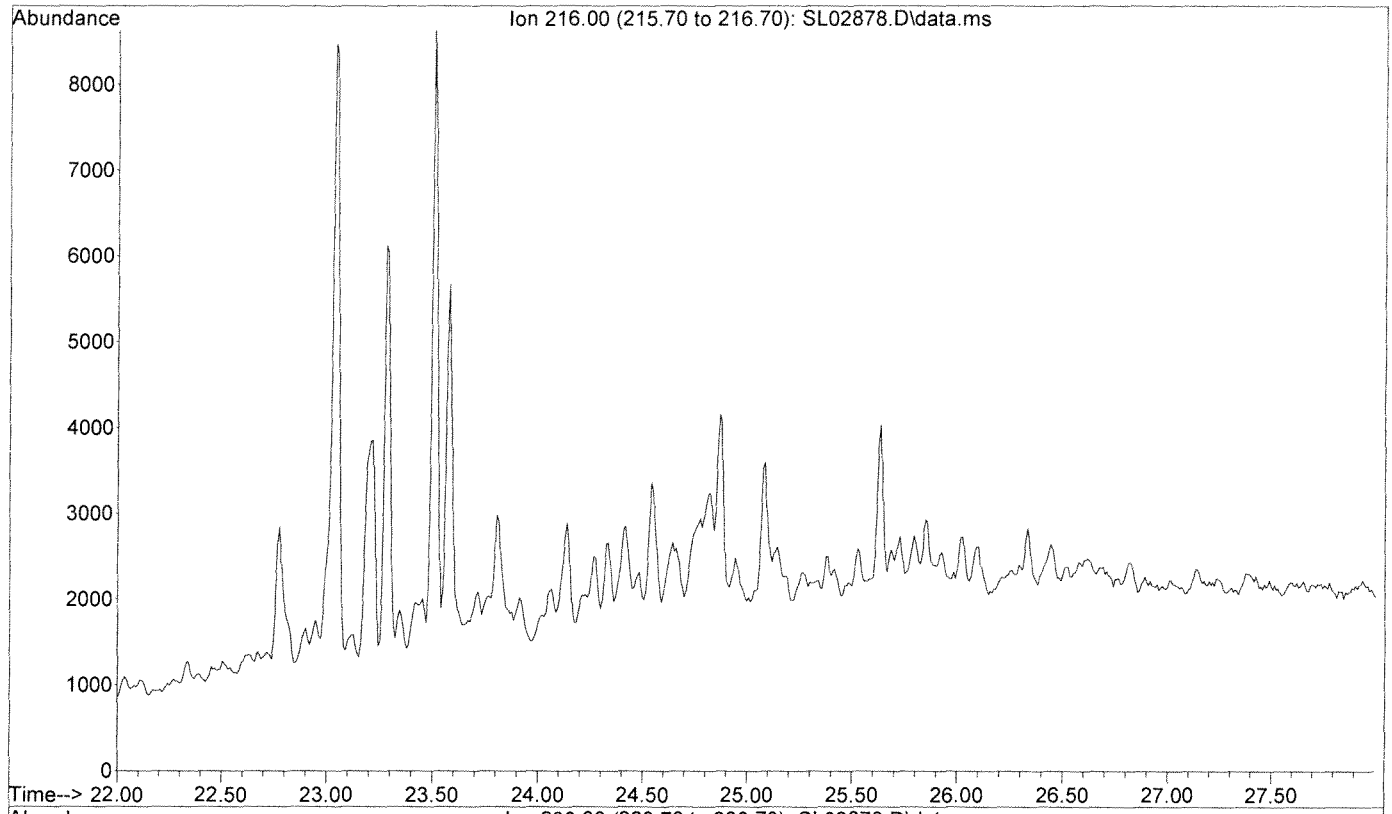
US EPA ARCHIVE DOCUMENT

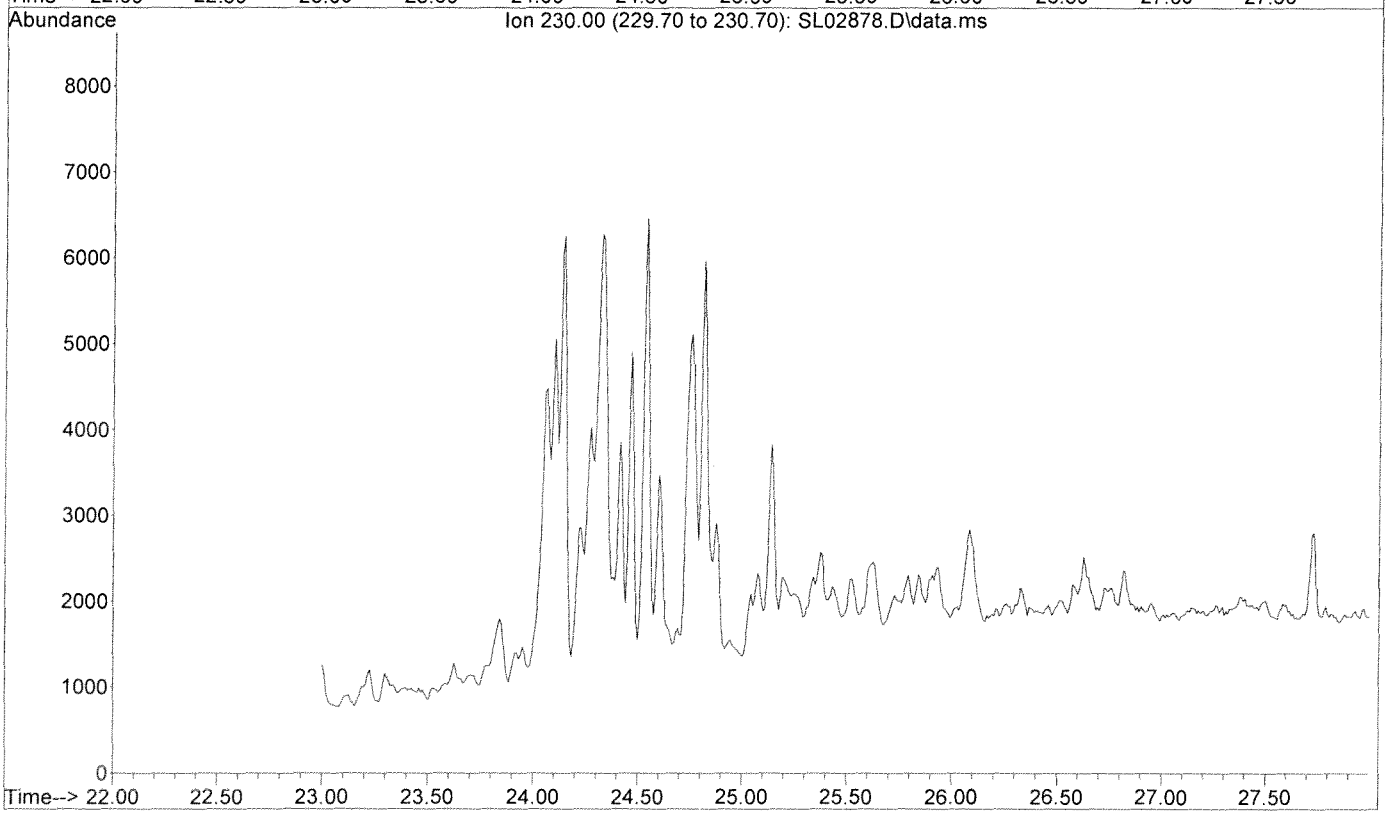
```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



US EPA ARCHIVE DOCUMENT

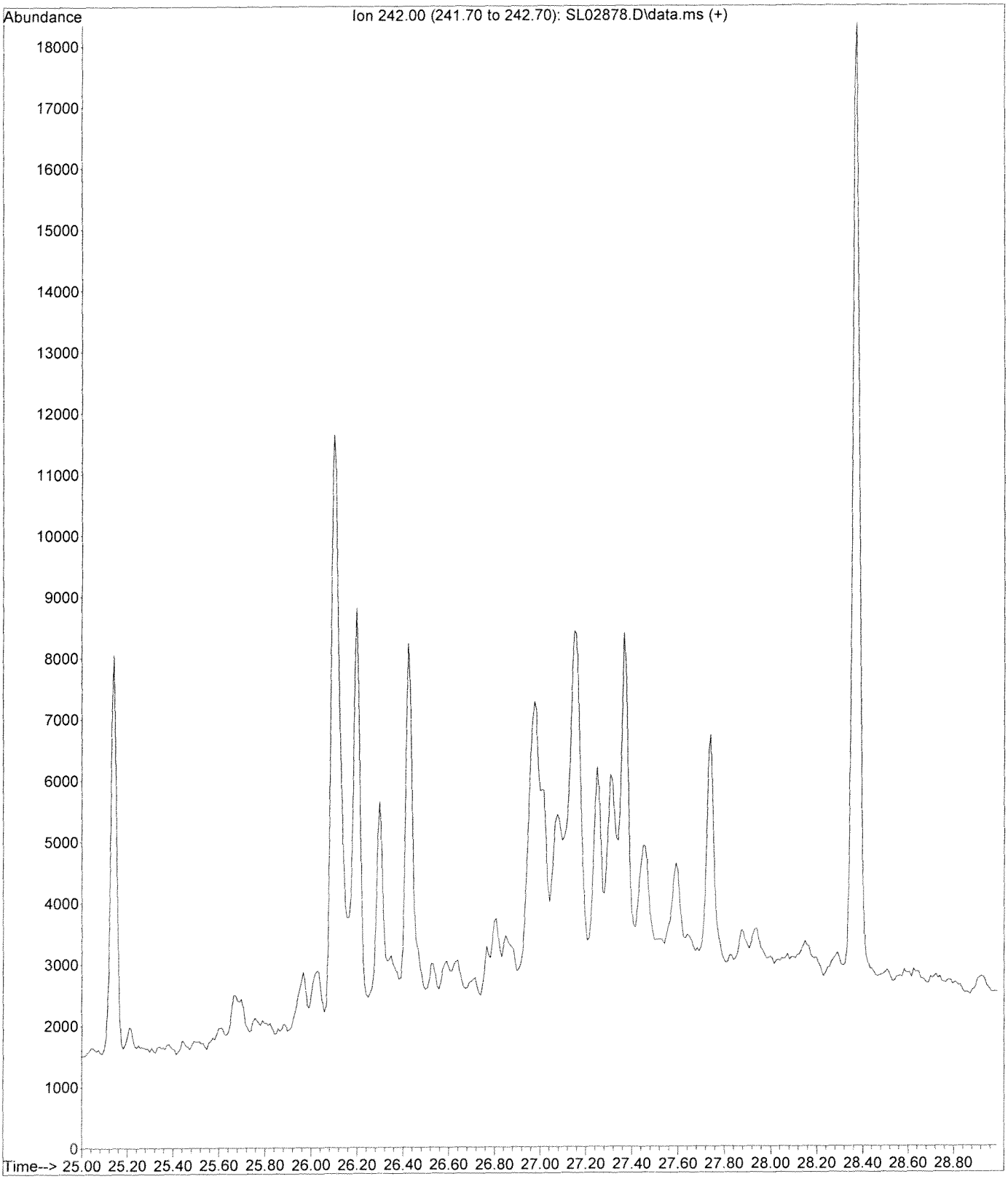
```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012   22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```





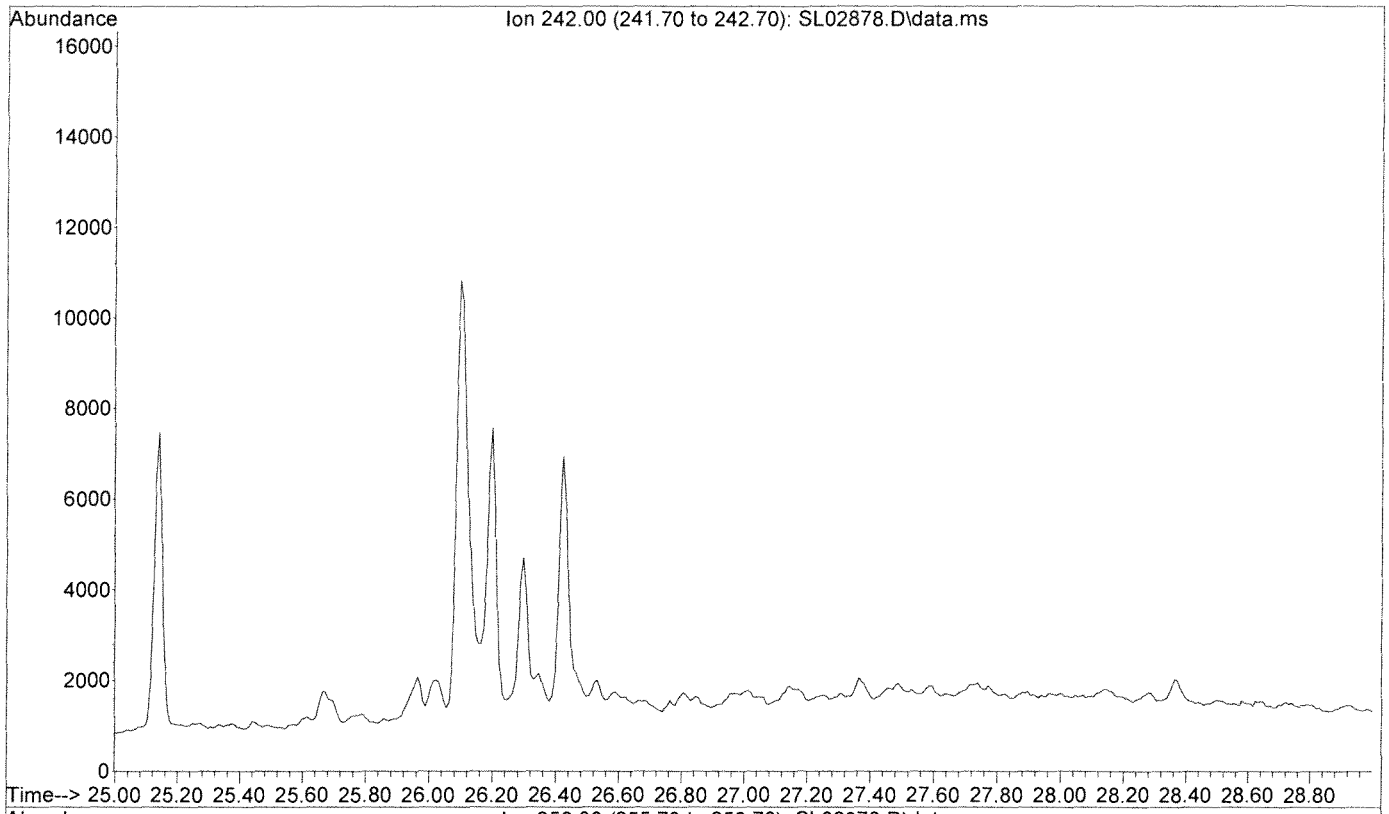
US EPA ARCHIVE DOCUMENT

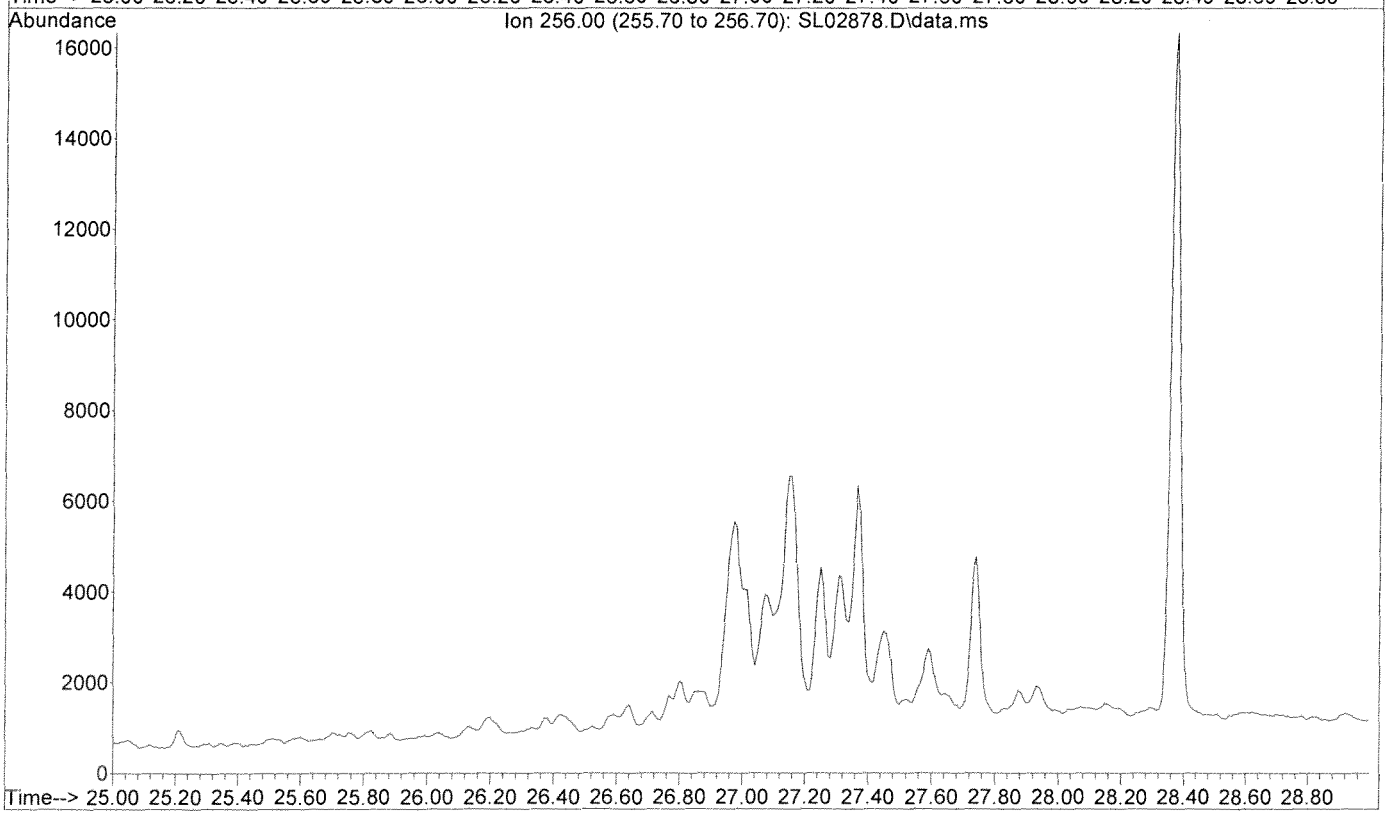
```
File         :C:\msdchem\1\DATA\022712\SL02878.D
Operator     : Syslo
Acquired     : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-000x
Misc Info    : Bucket 2 "qual skim"
Vial Number: 5
```



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```





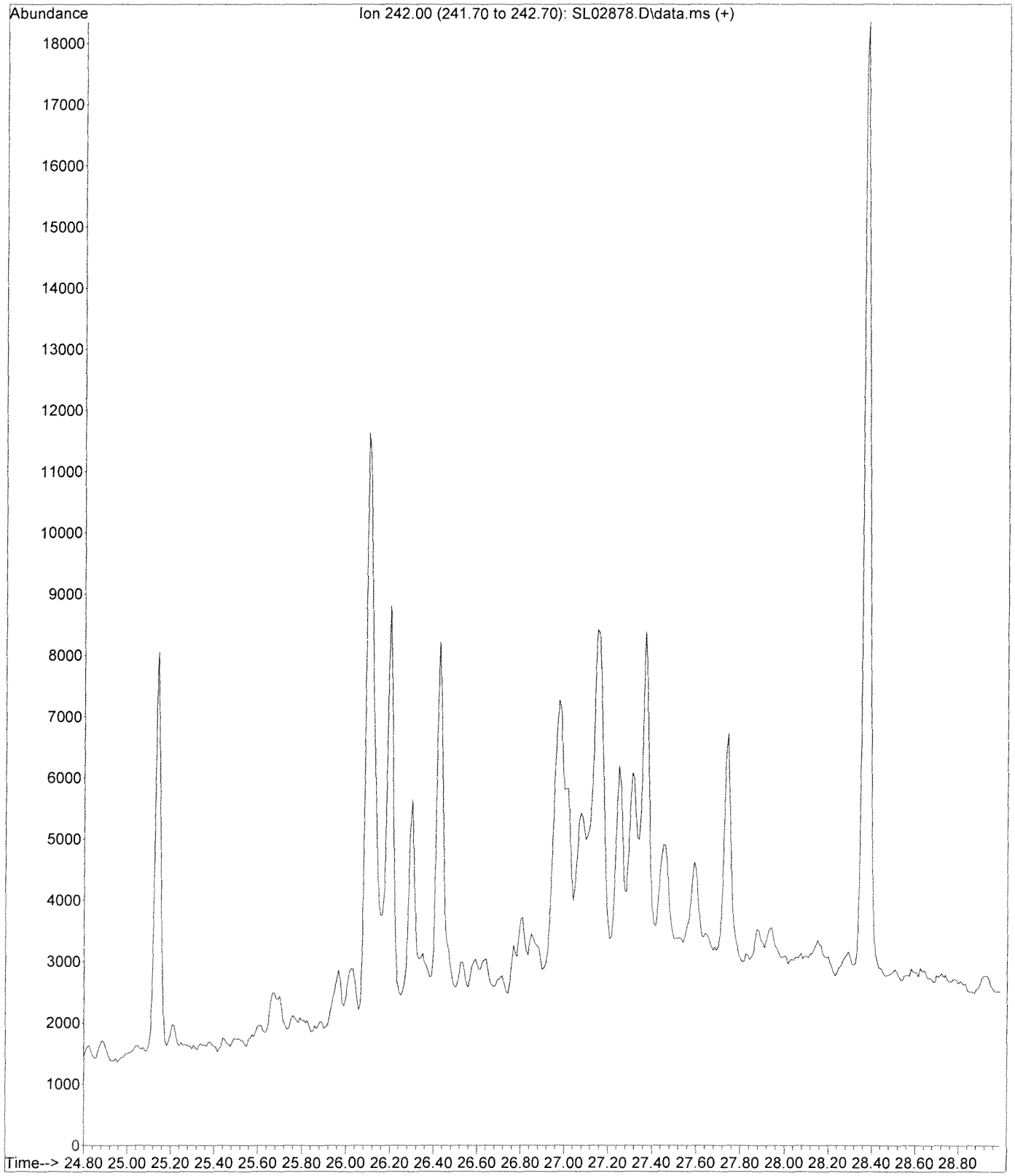
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02878.D
Operator    : Syslo
Acquired    : 27 Feb 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-000x
Misc Info   : Bucket 2 "qual skim"
Vial Number: 5
```



US EPA ARCHIVE DOCUMENT

SERAS-017-0003 EOS
SL02879.1

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02879.D
Acq On : 27 Feb 2012 23:40
Operator : Syslo
Sample : SERAS-017-0003 EOS
Misc : 275-55-05 EOB Duplicate
ALS Vial : 6 Sample Multiplier: 1

Quant Time: Mar 01 13:06:34 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	309155	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	101381	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	126388	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	115104	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.822	188	570170	17.34	ug/mL	0.00
Spiked Amount 20.000			Recovery = 86.70%			
3) d14-o-Terphenyl {S}	19.733	244	273247	18.91	ug/mL	0.00
Spiked Amount 20.000			Recovery = 94.55%			
4) 5a-Androstane {S}	21.140	260	35542	20.22	ug/mL	0.00
Spiked Amount 20.000			Recovery = 101.10%			
7) d62-Triacontane {S}	28.156	66	244580	21.81	ug/mL	0.00
Spiked Amount 20.000			Recovery = 109.05%			
Target Compounds						Qvalue

#) = qualifier out of range (m) = manual integration (+) = signals summed

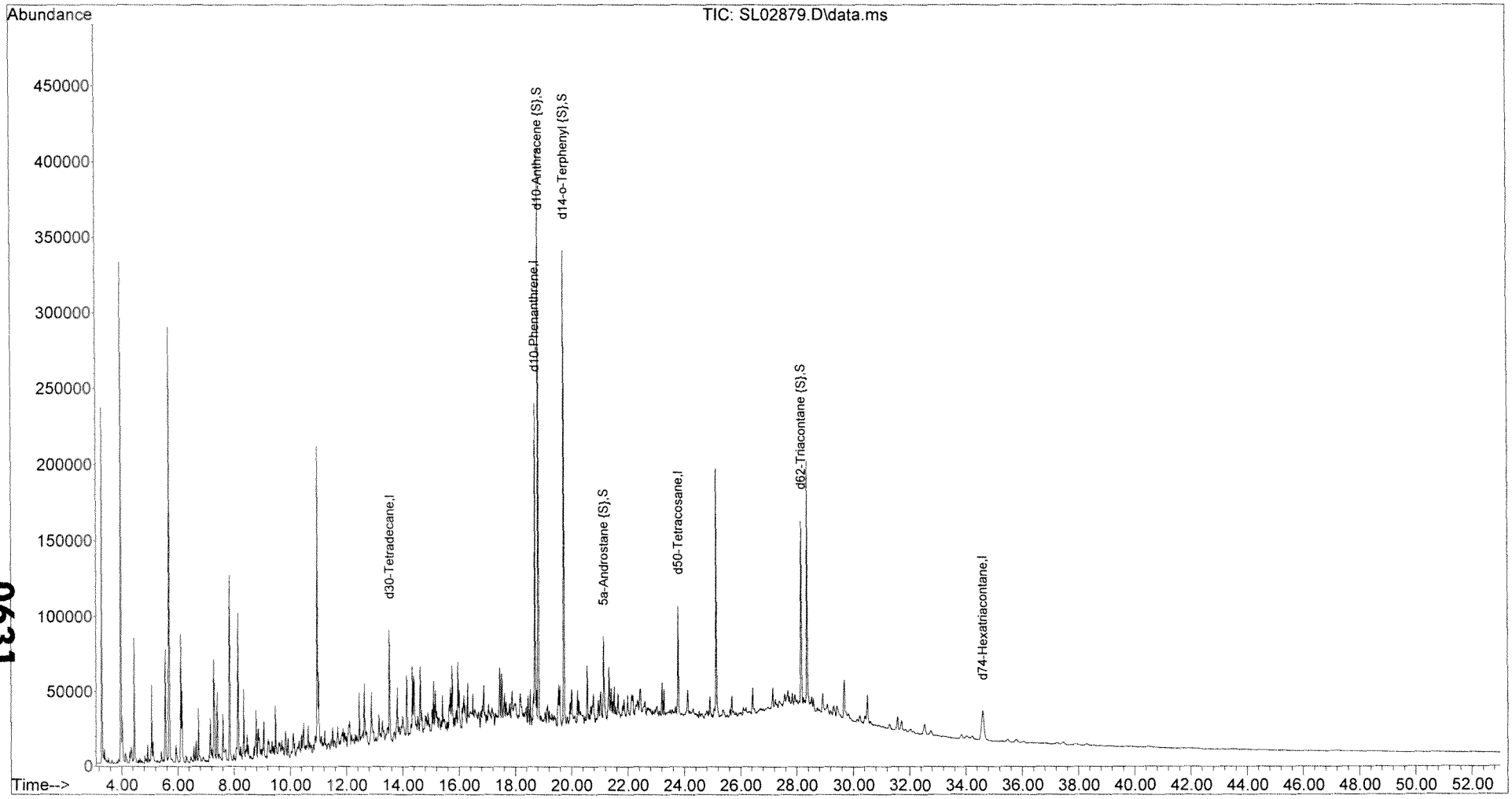
US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\022712\
Data File : SL02879.D
Acq On : 27 Feb 2012 23:40
Operator : Syslo
Sample : SERAS-017-0003 EOS
Misc : 275-55-05 EOB Duplicate
ALS Vial : 6 Sample Multiplier: 1

Quant Time: Mar 01 13:06:34 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0631

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

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Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

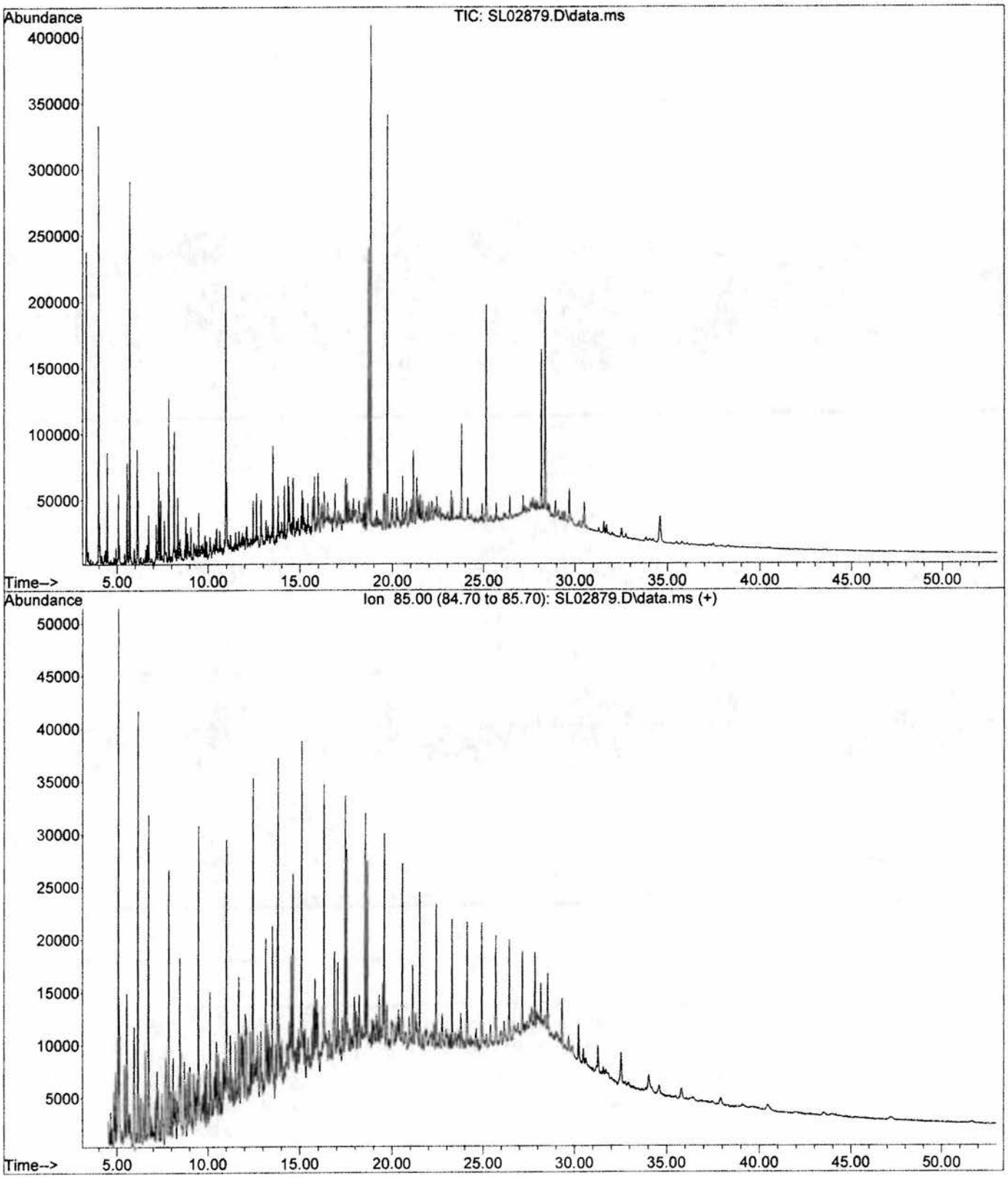
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

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```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6

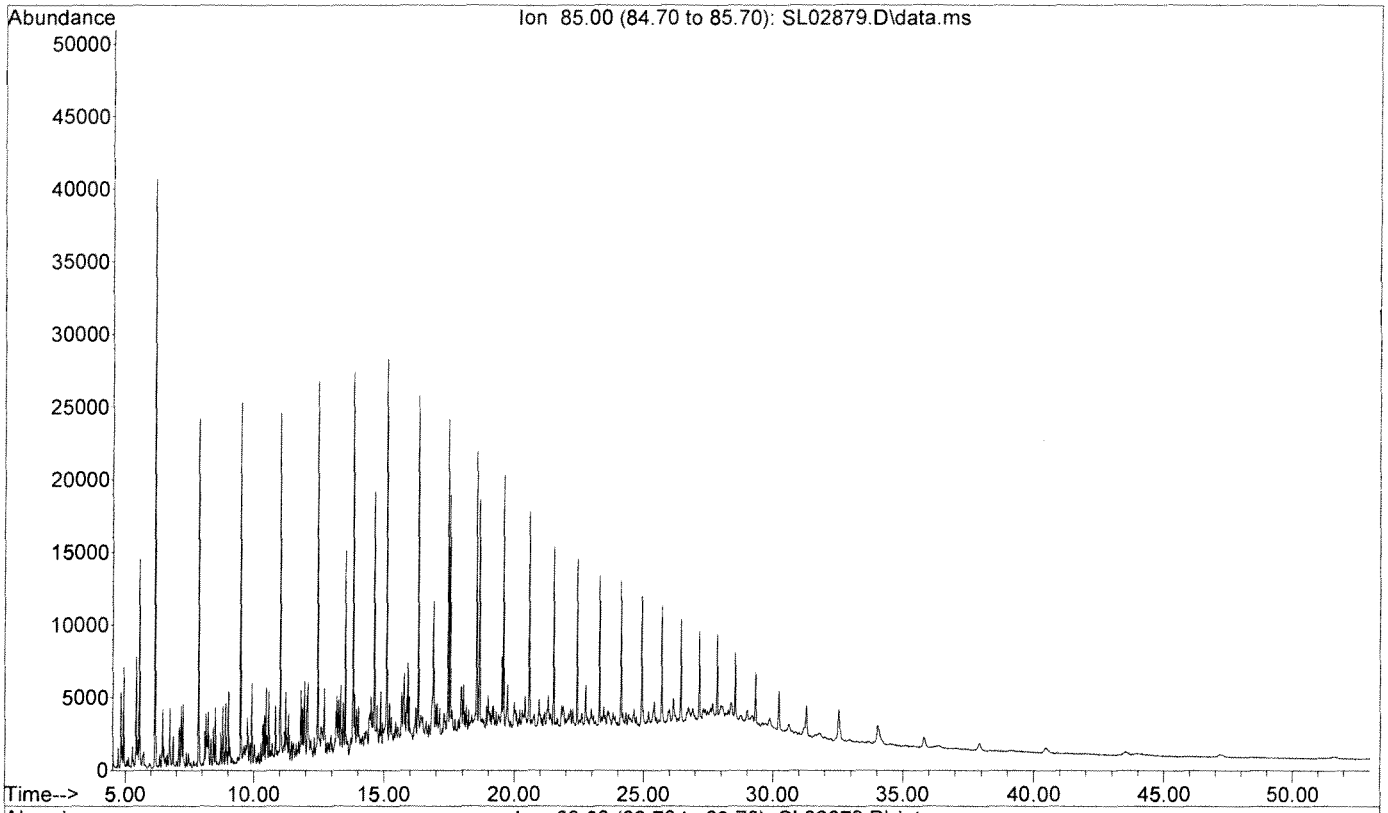


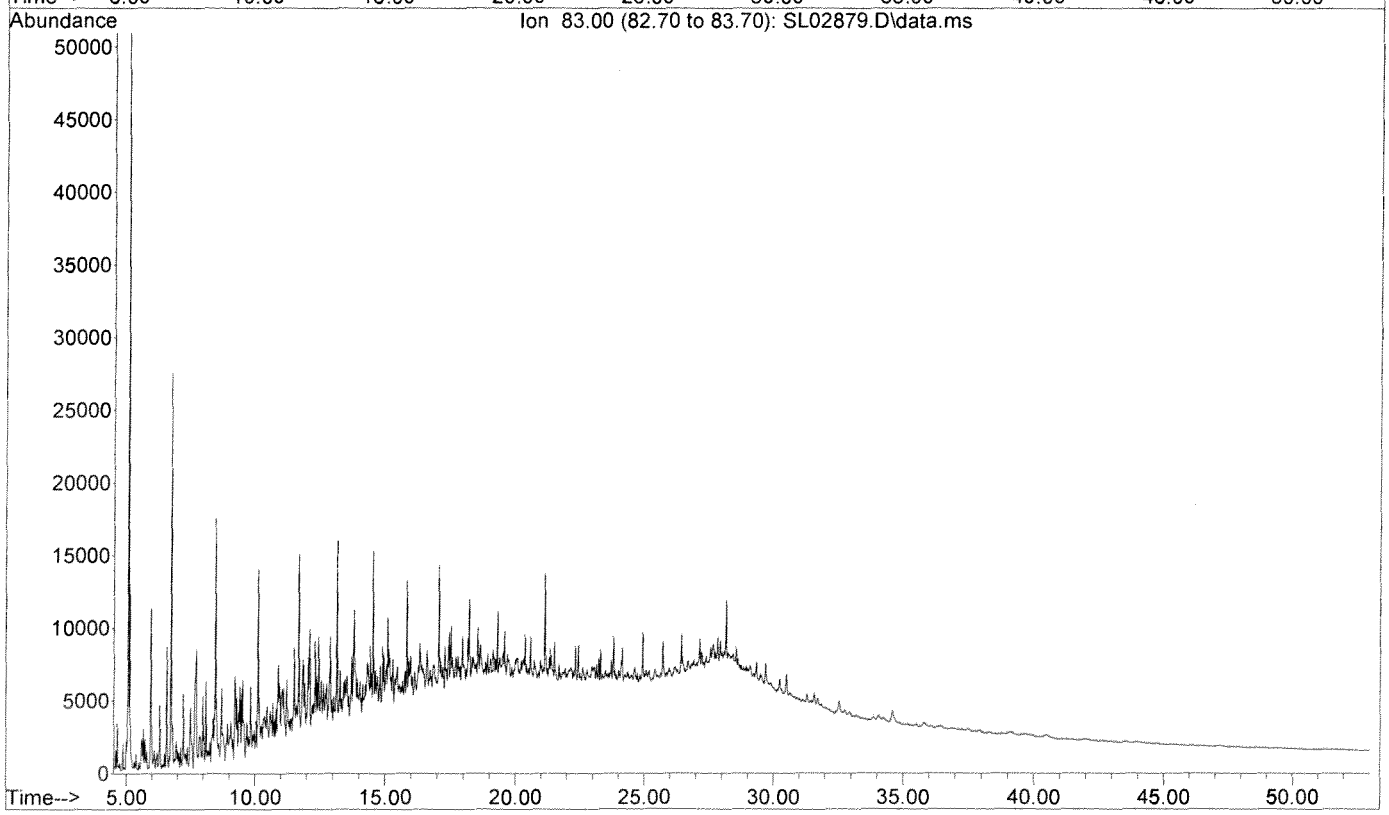
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6





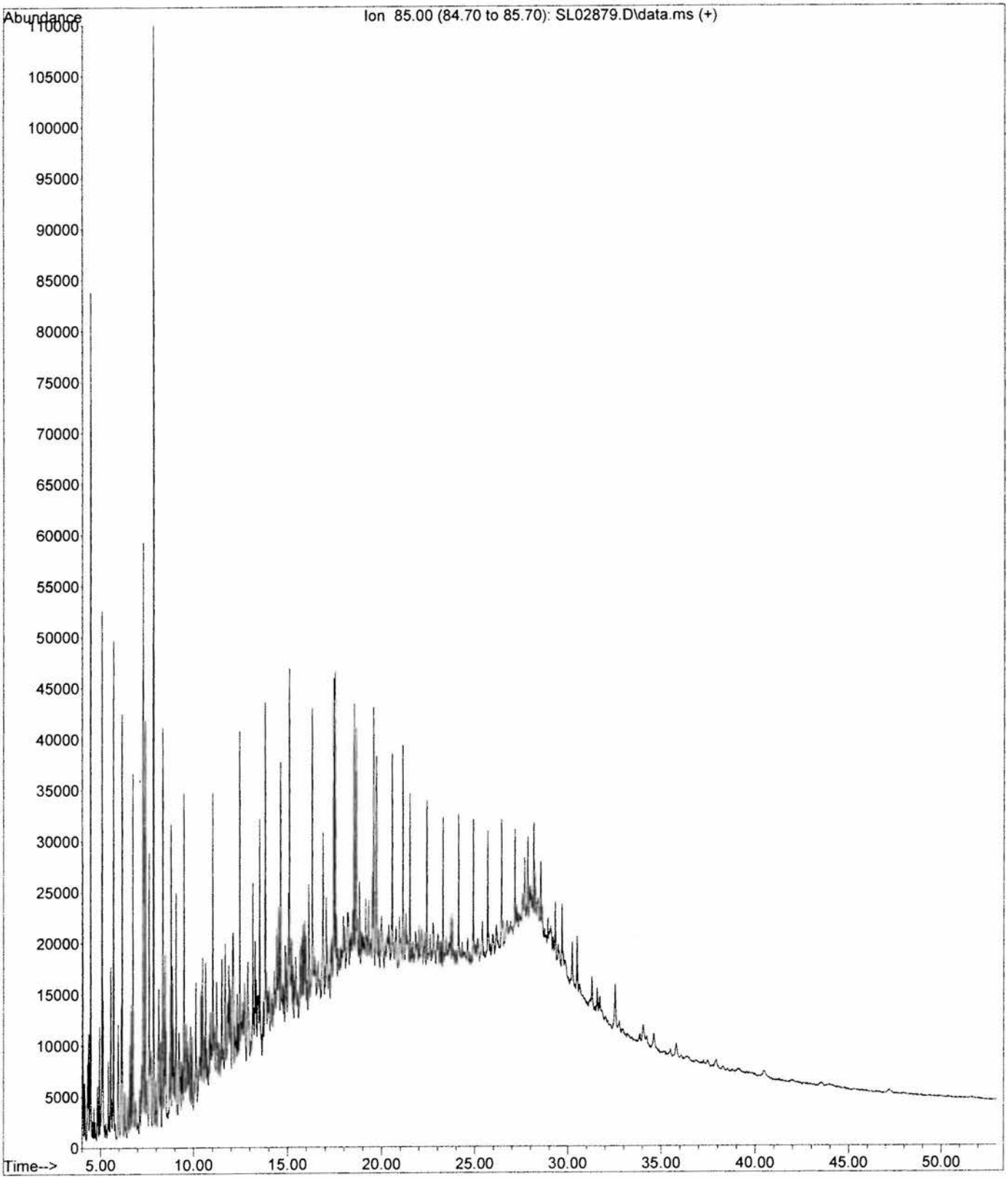
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6



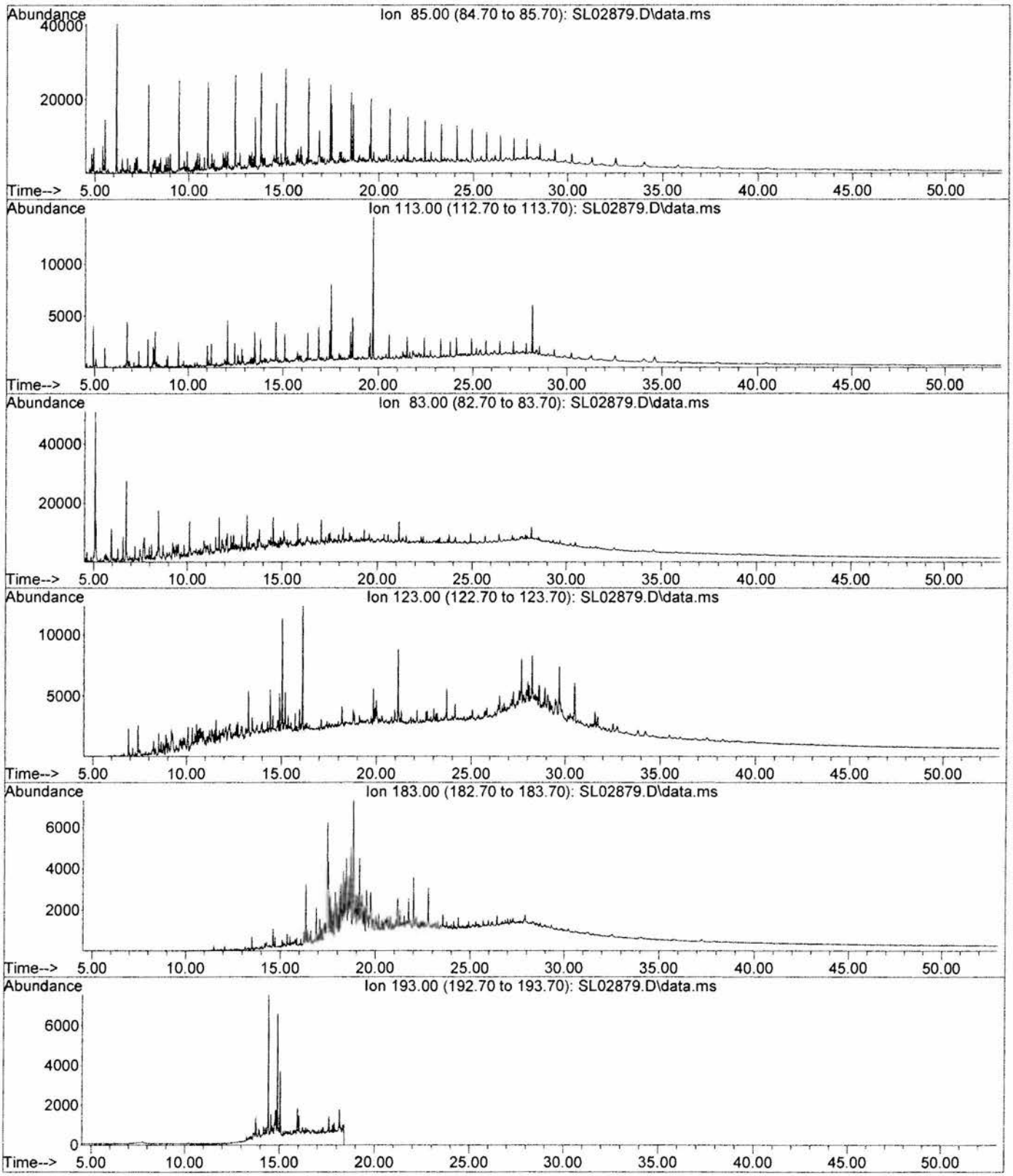
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02879.D
Operator    : Syslo
Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
```



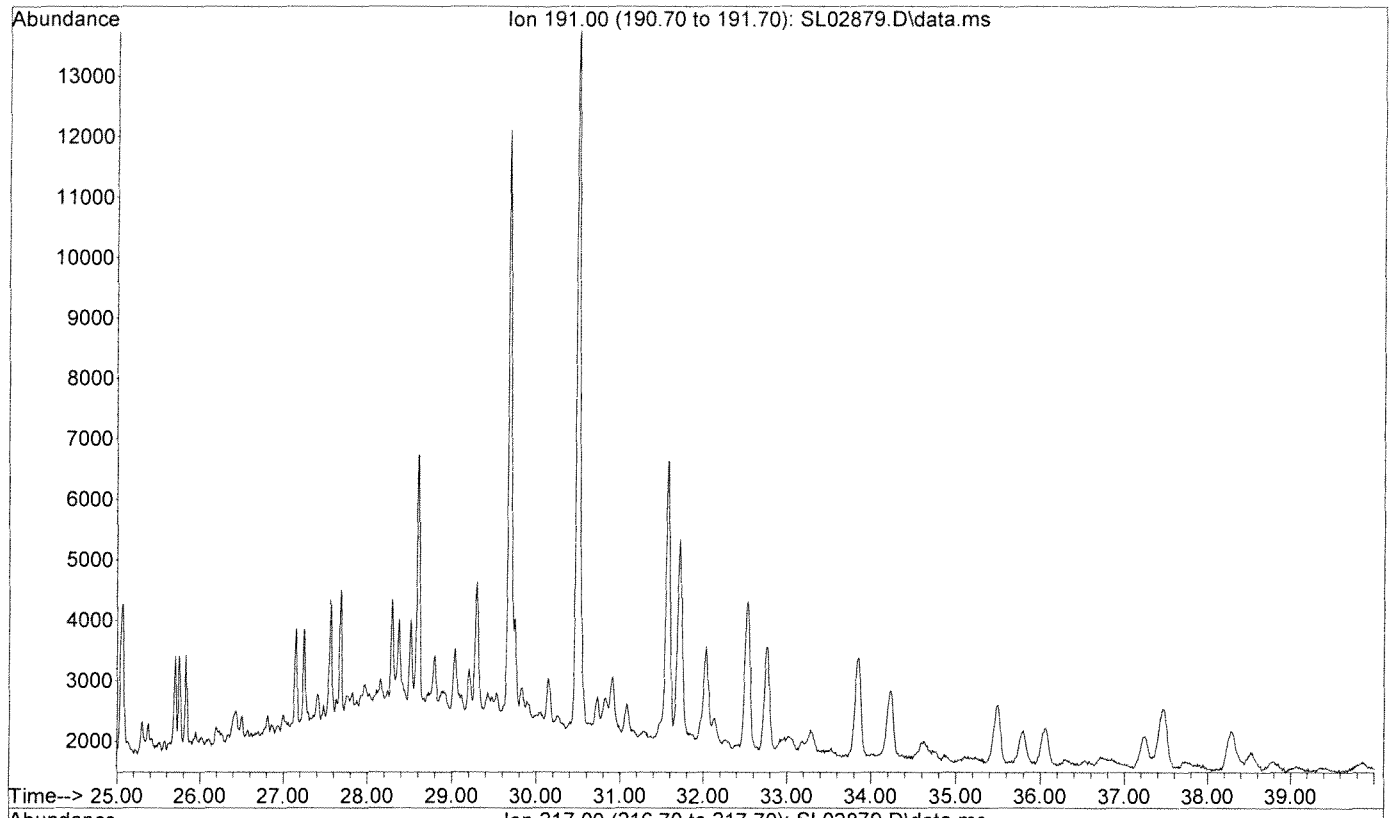
US EPA ARCHIVE DOCUMENT

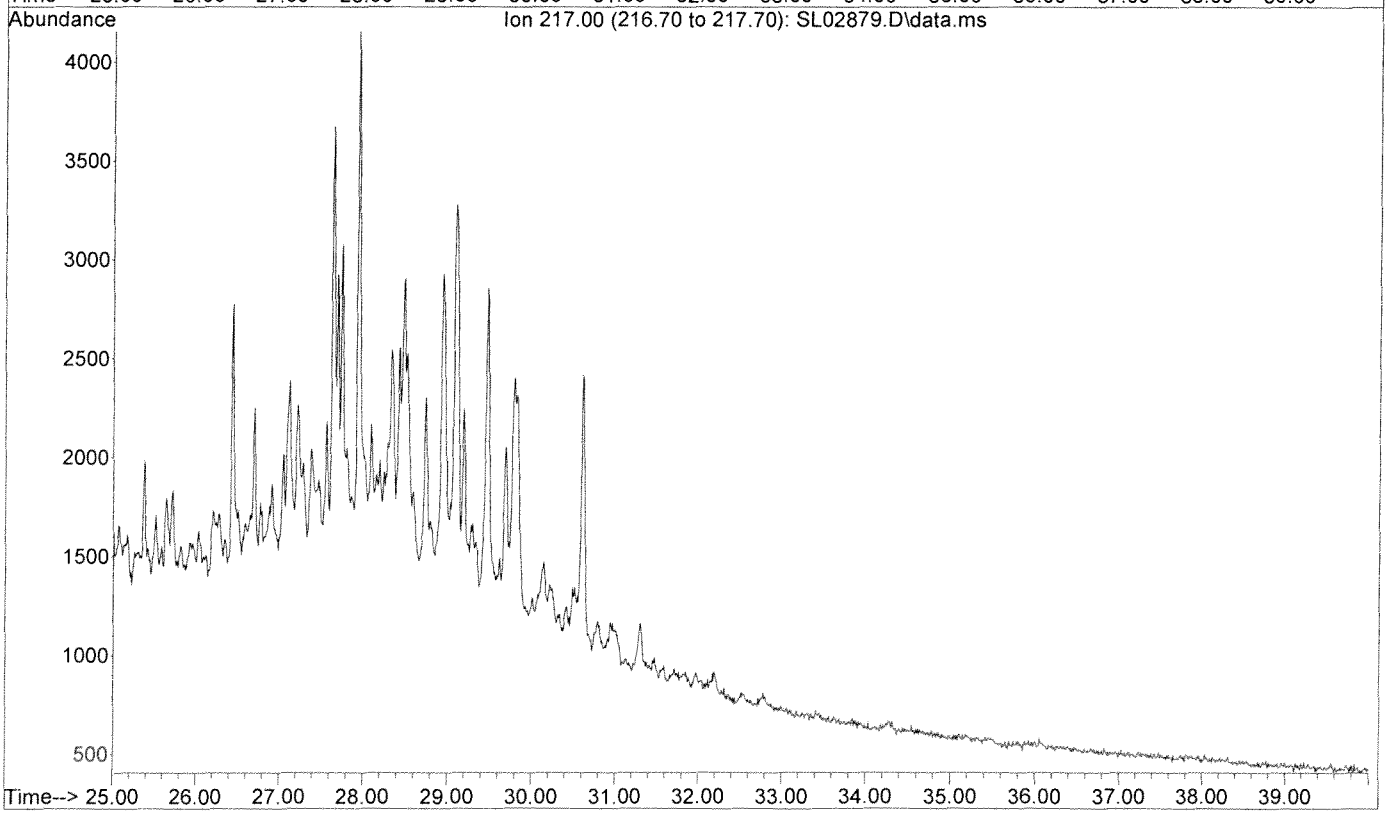
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6





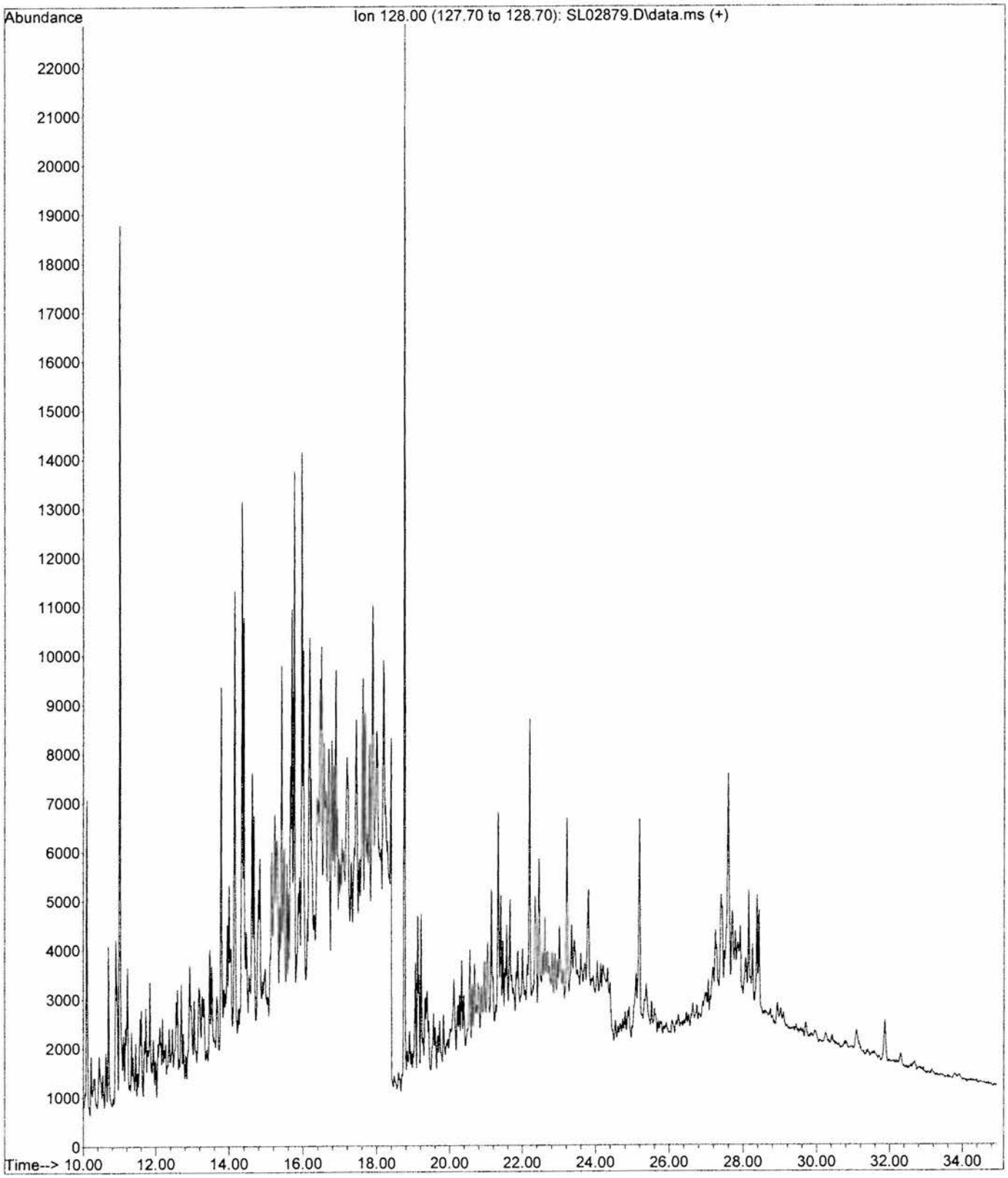
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6



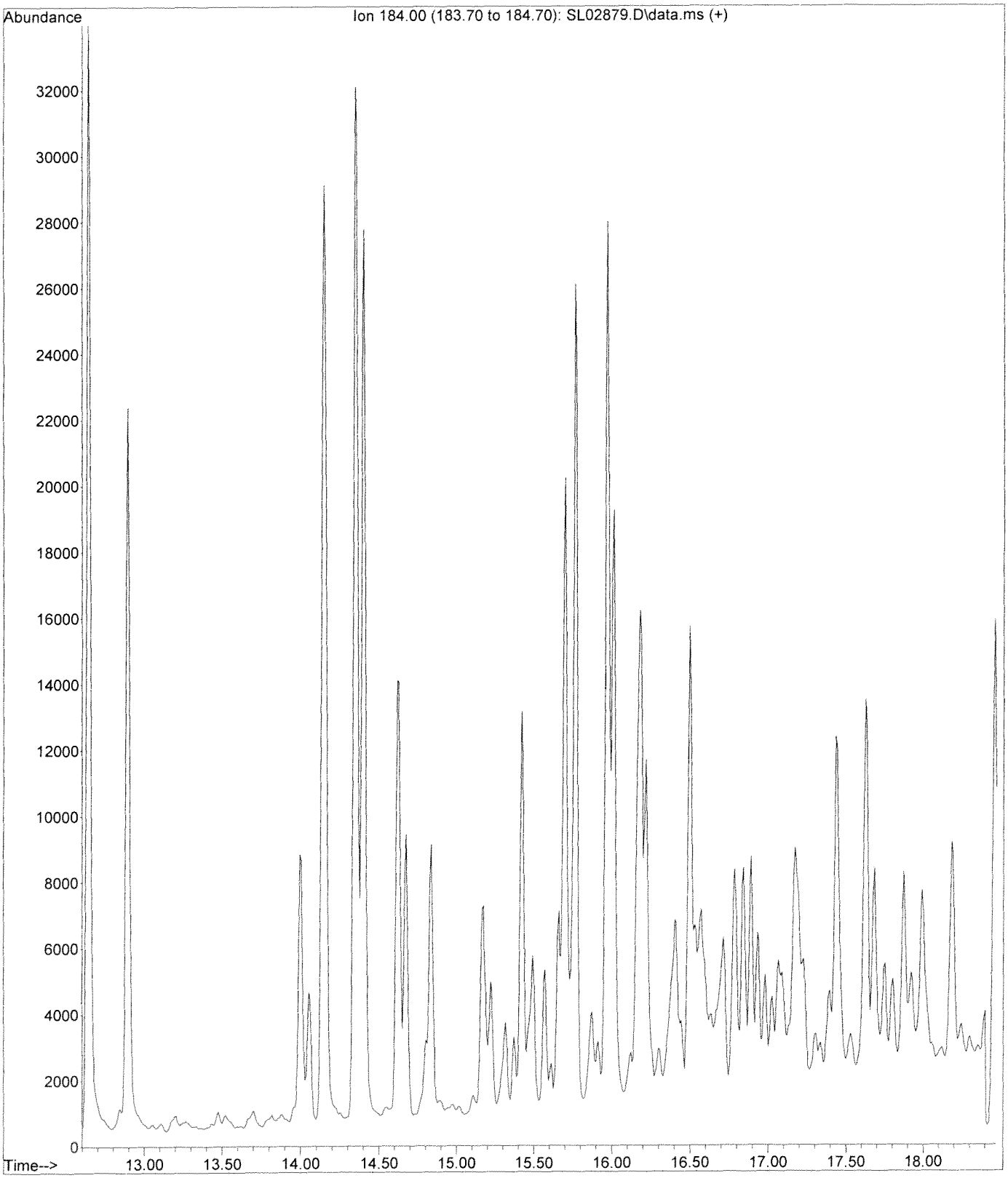
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

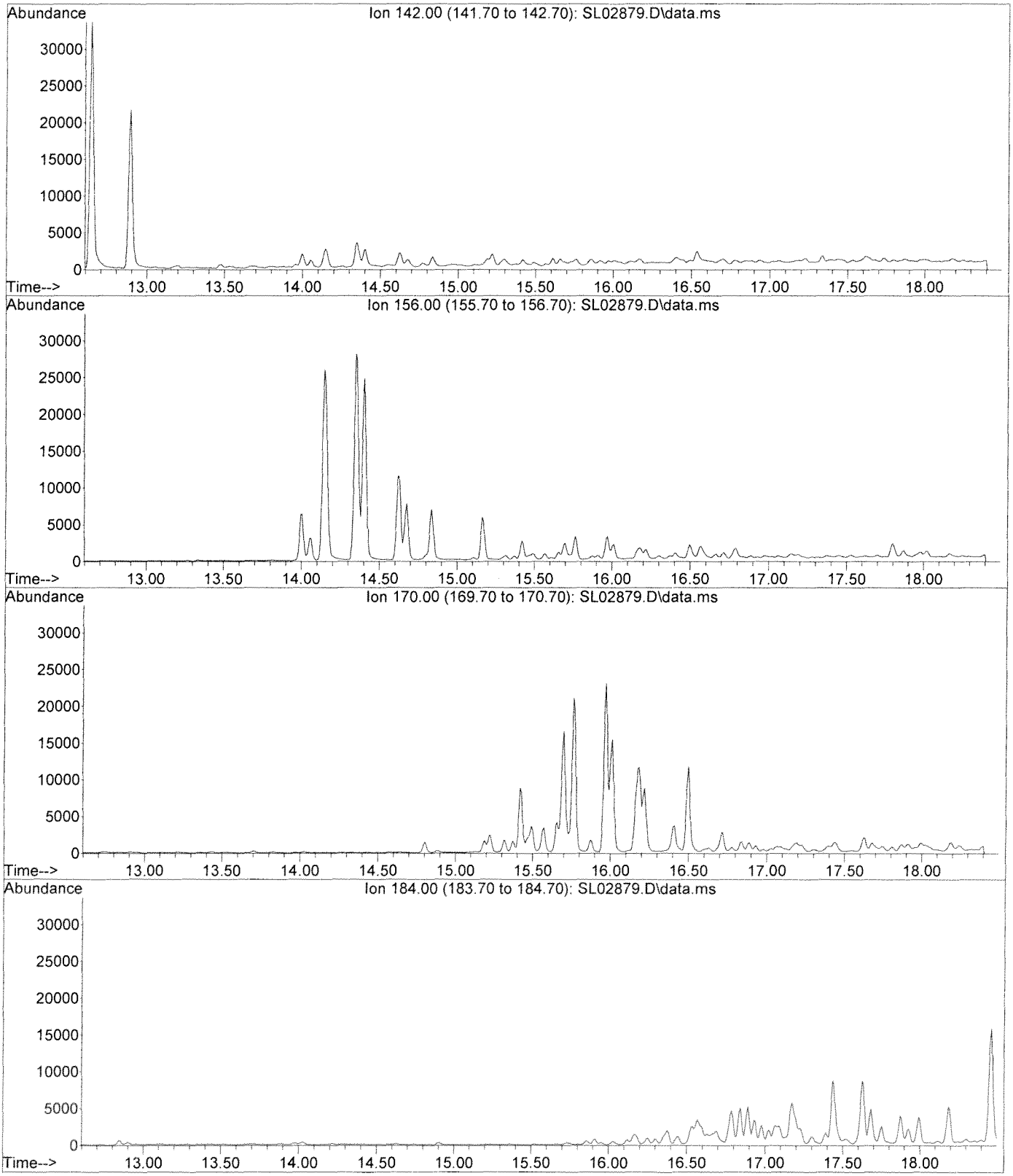
US EPA ARCHIVE DOCUMENT

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File        :C:\msdchem\1\DATA\022712\SL02879.D
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Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
```



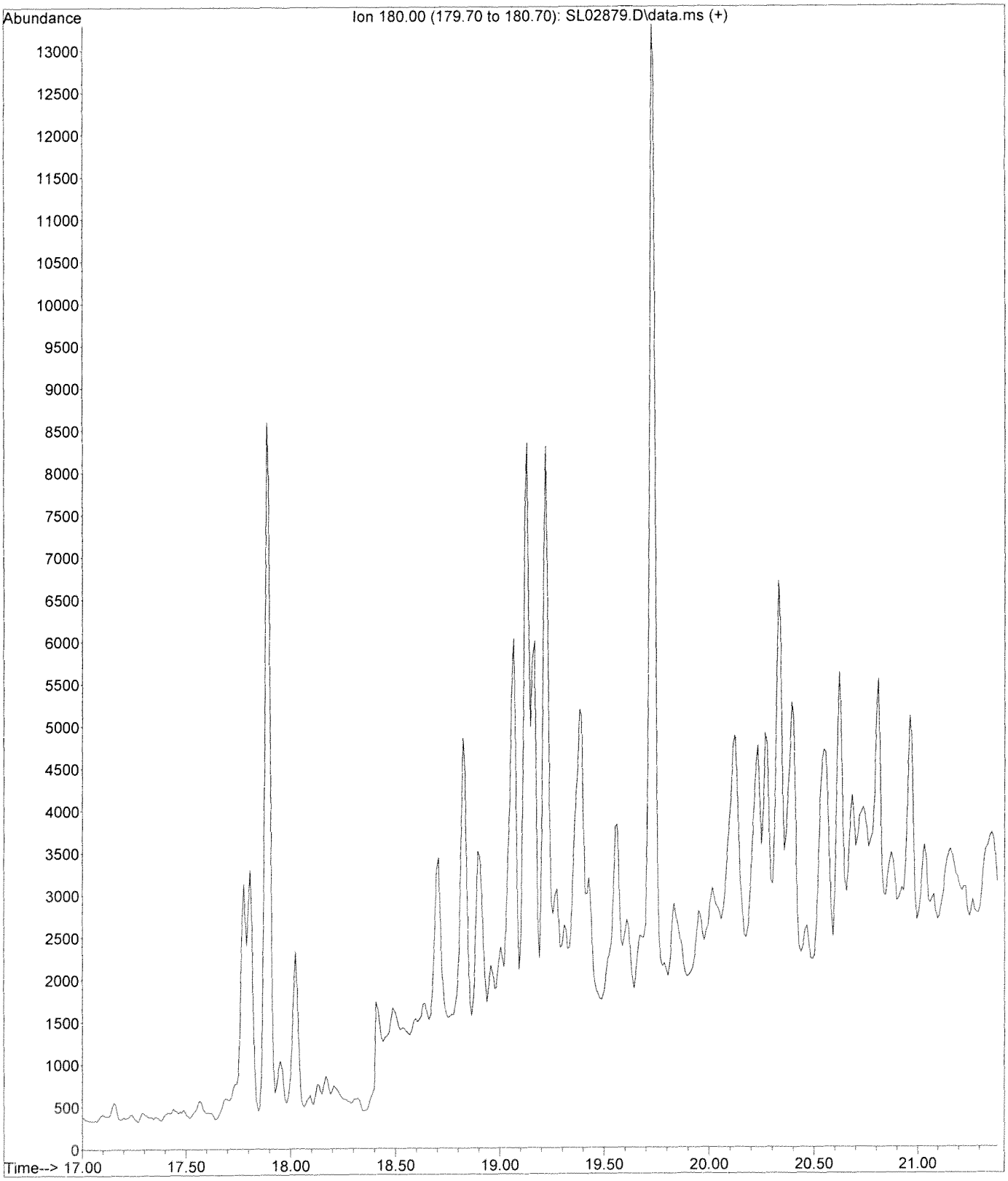
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Instrument :   Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
```



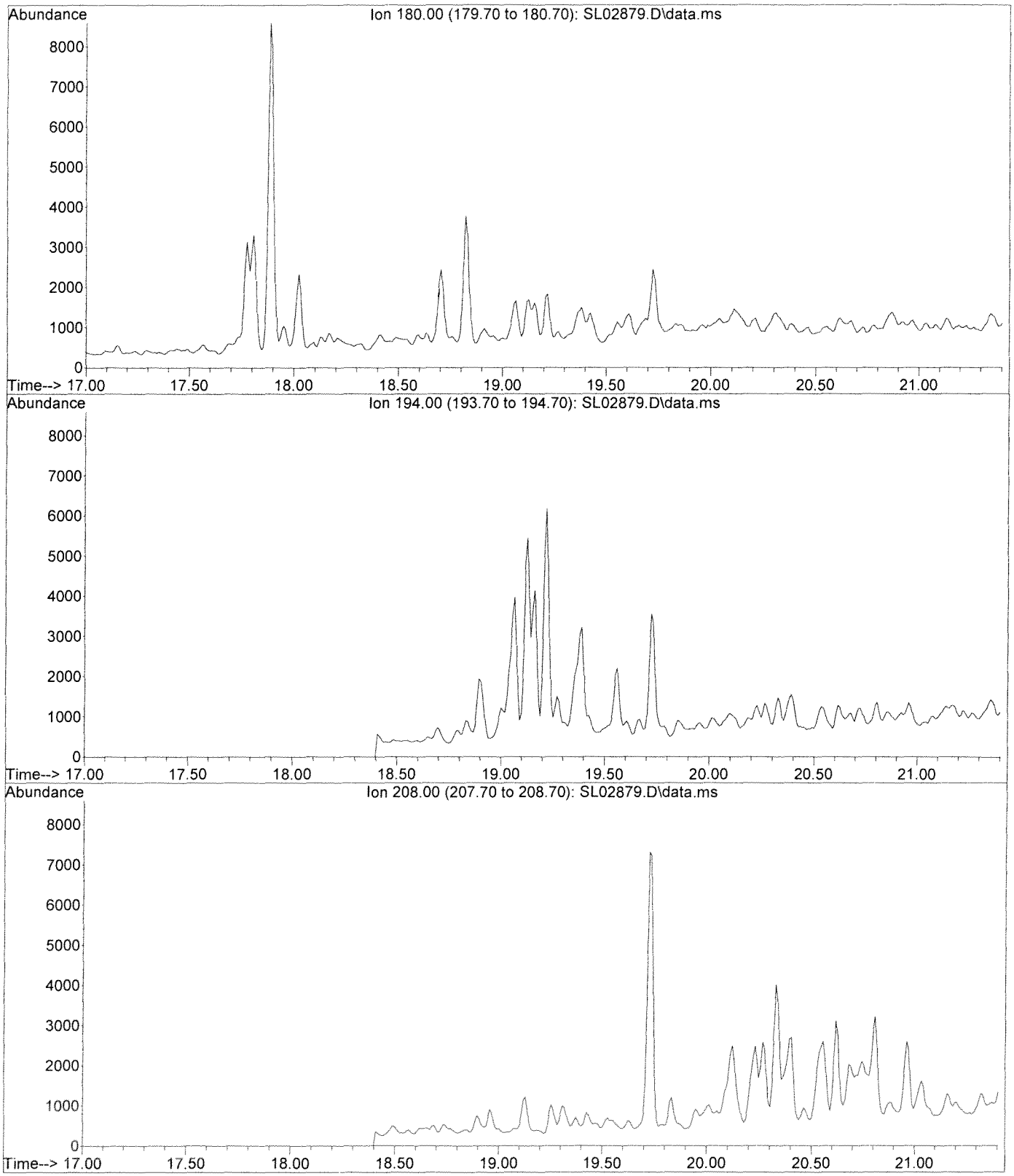
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6



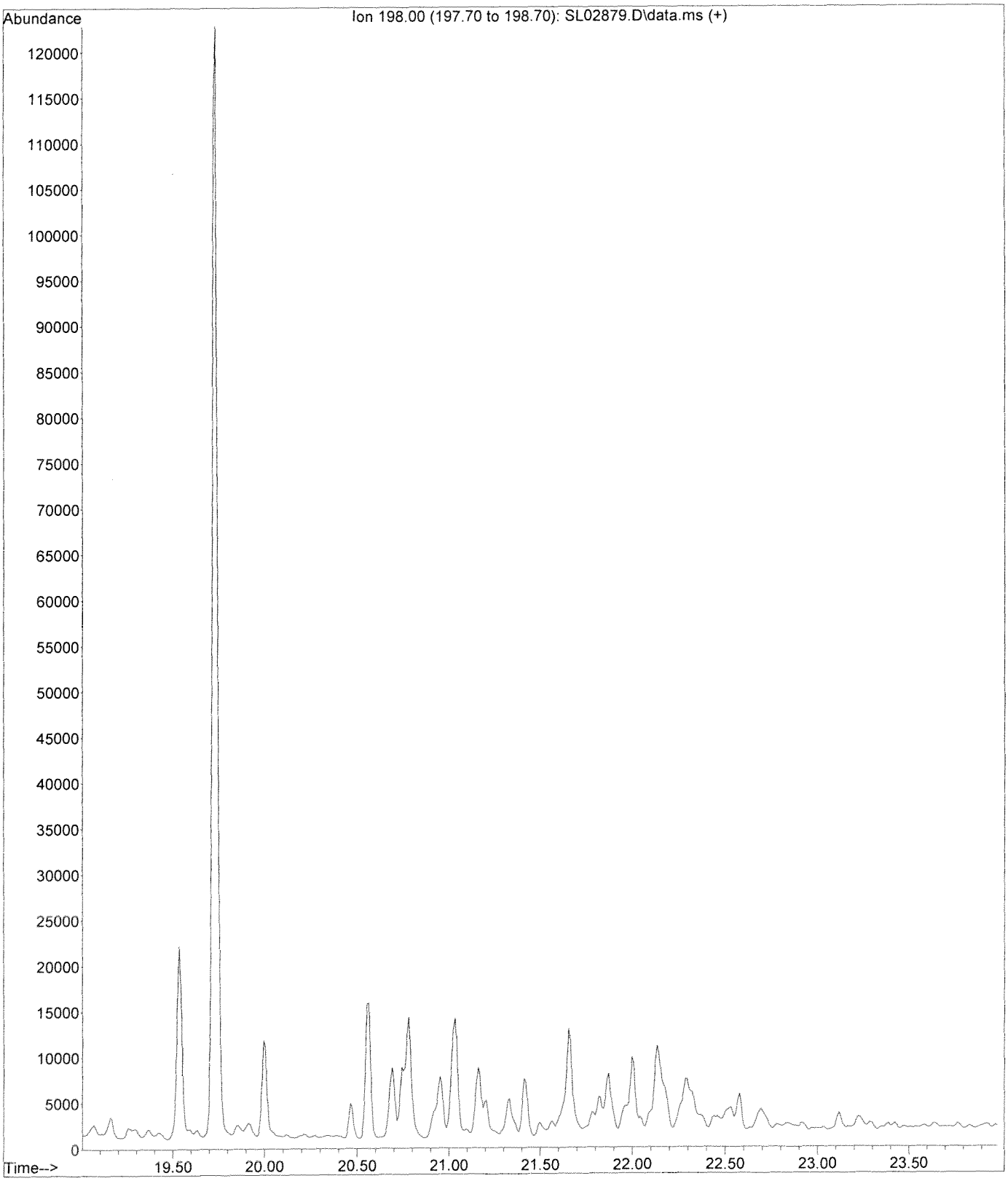
US EPA ARCHIVE DOCUMENT

```
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Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
```



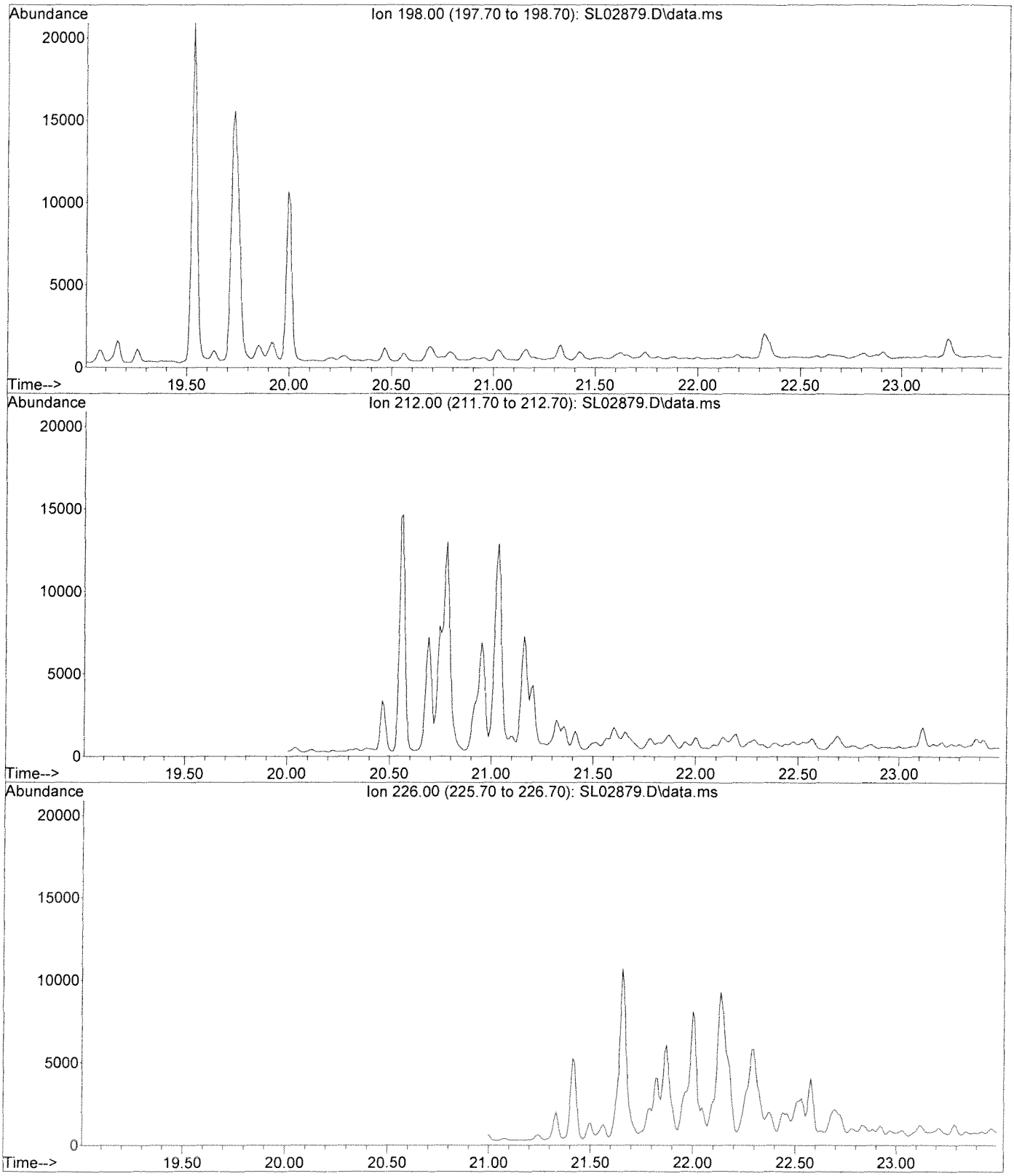
US EPA ARCHIVE DOCUMENT

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Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6



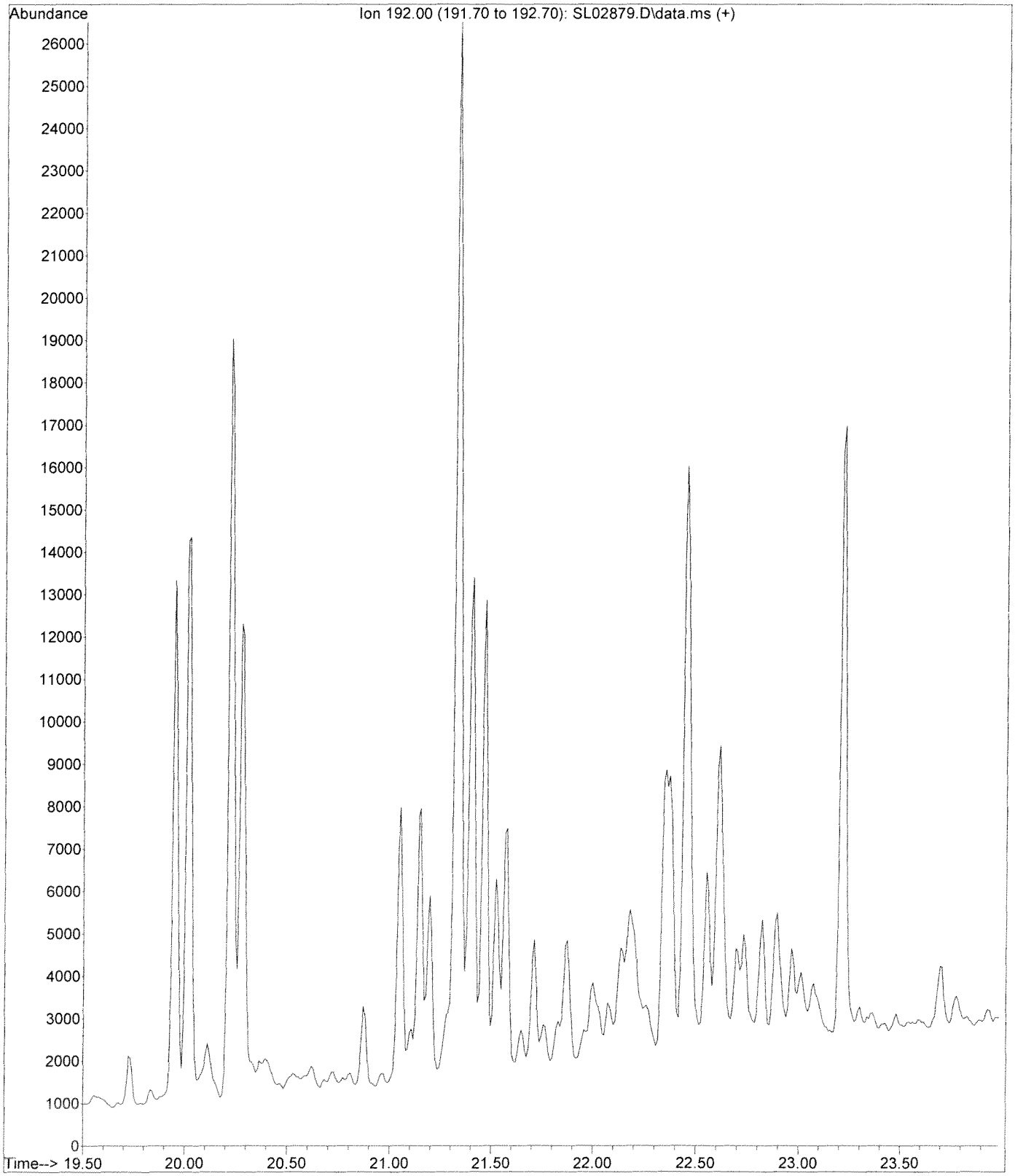
US EPA ARCHIVE DOCUMENT

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Operator    : Syslo
Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
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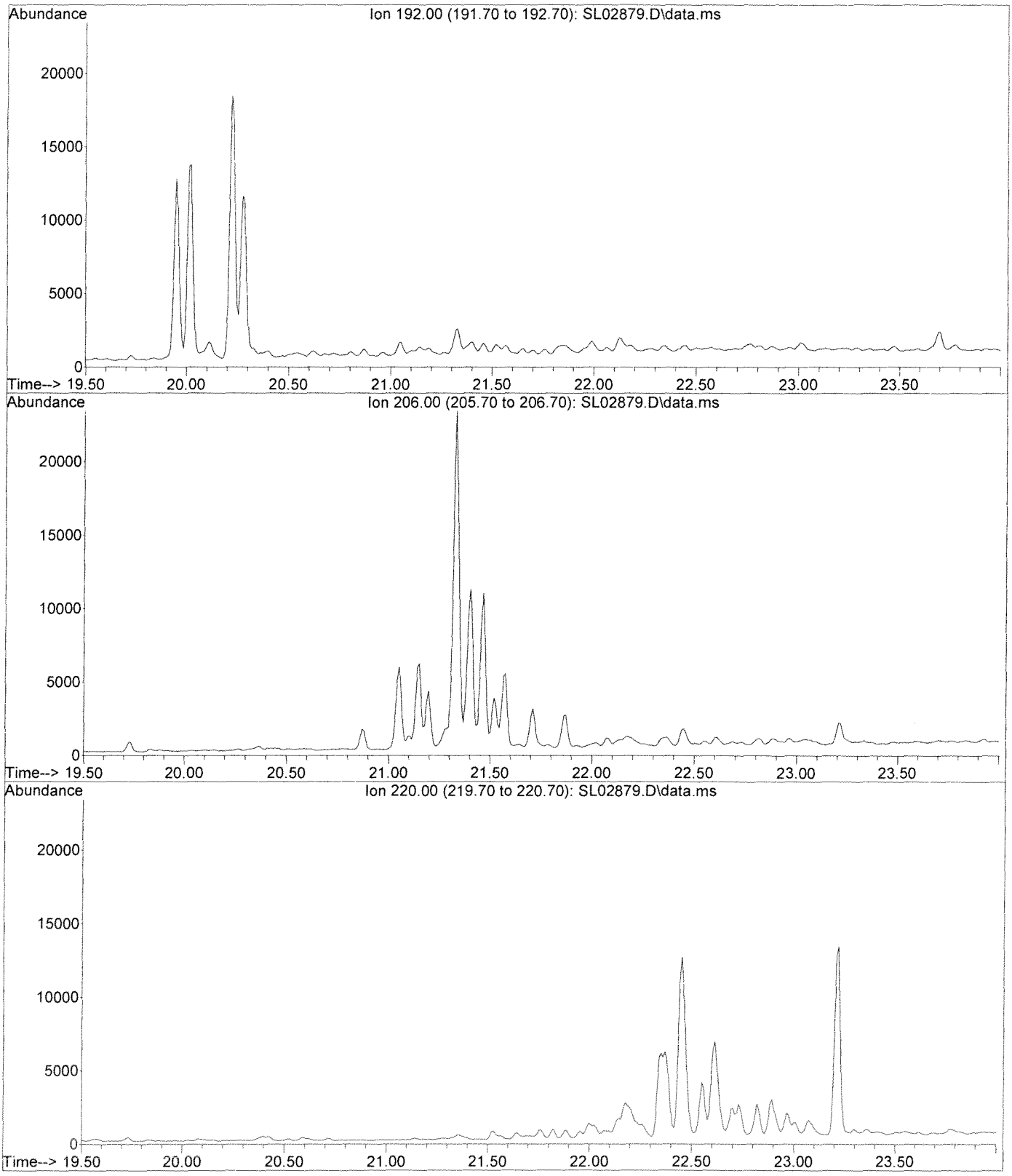
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File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
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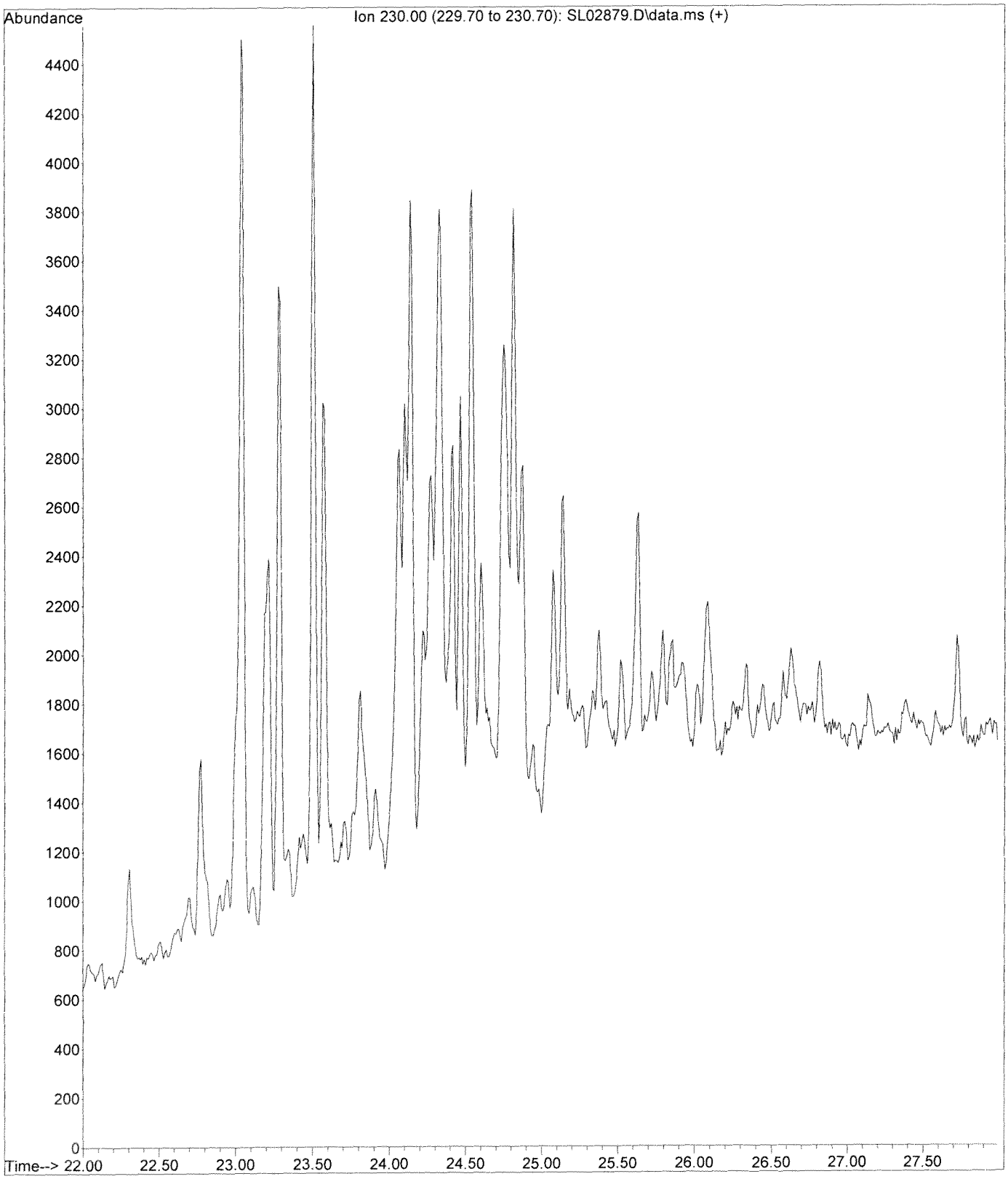
US EPA ARCHIVE DOCUMENT

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File        :C:\msdchem\1\DATA\022712\SL02879.D
Operator    : Syslo
Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 EOS
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Vial Number: 6
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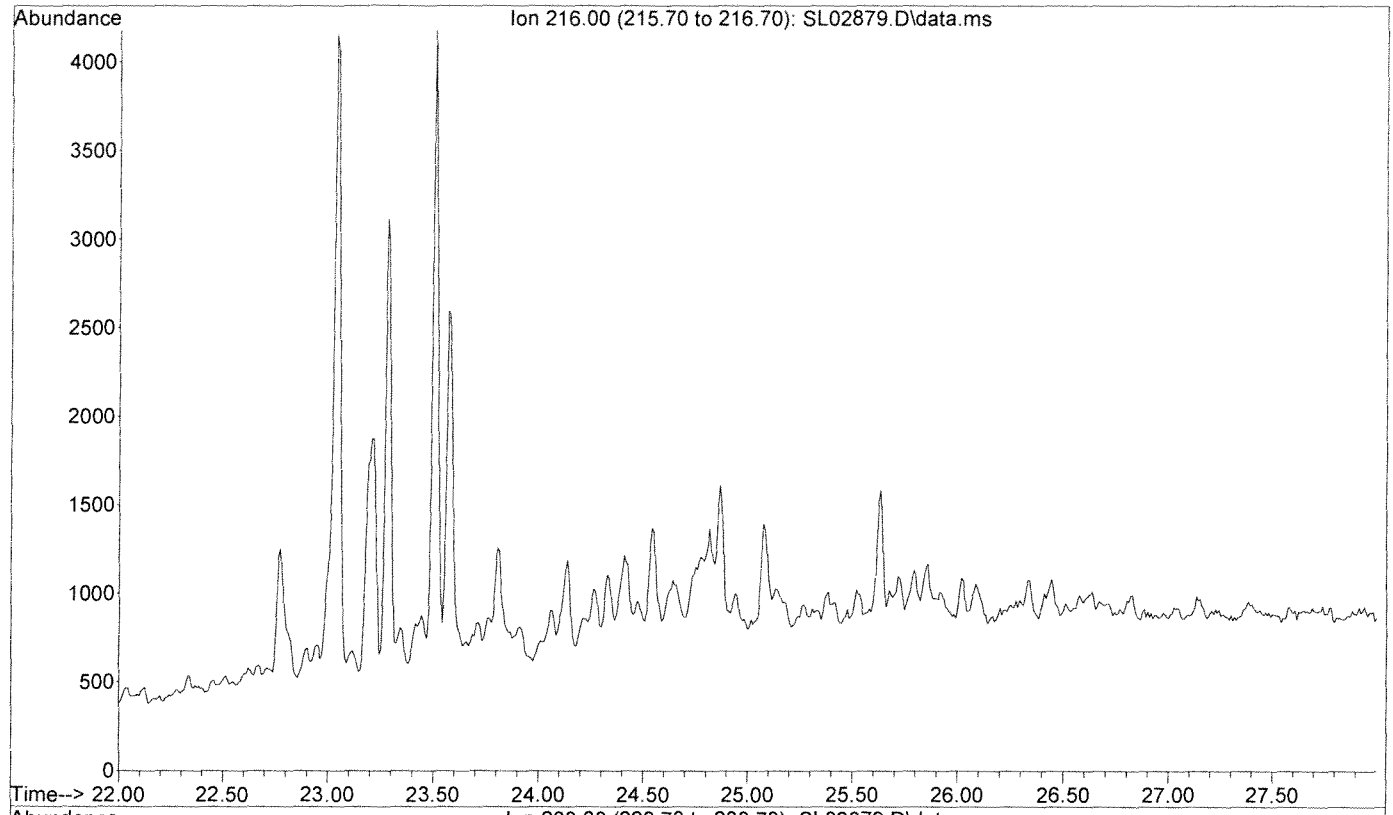
US EPA ARCHIVE DOCUMENT

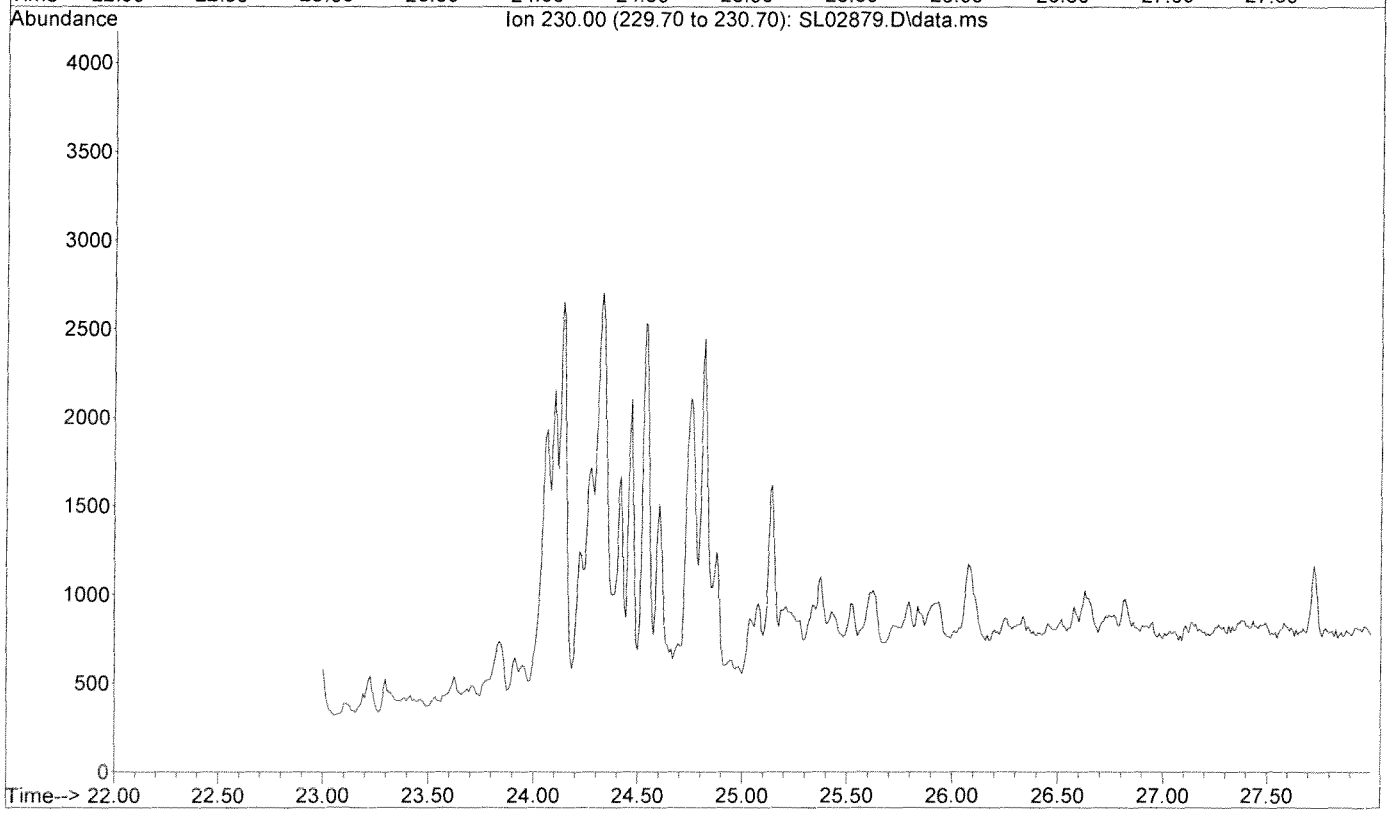
File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6



US EPA ARCHIVE DOCUMENT

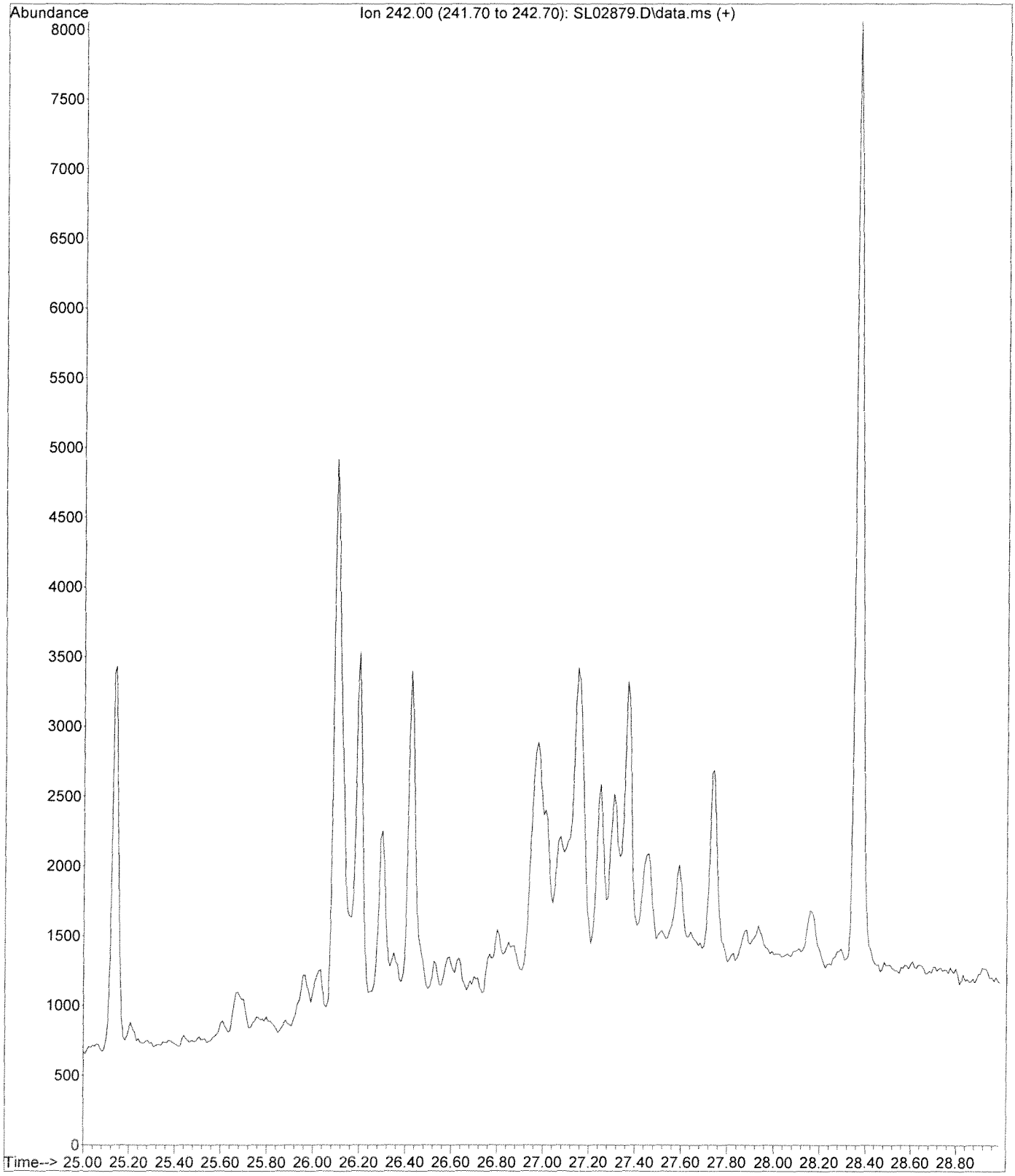
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File        :C:\msdchem\1\DATA\022712\SL02879.D
Operator    : Syslo
Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument :   Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
```





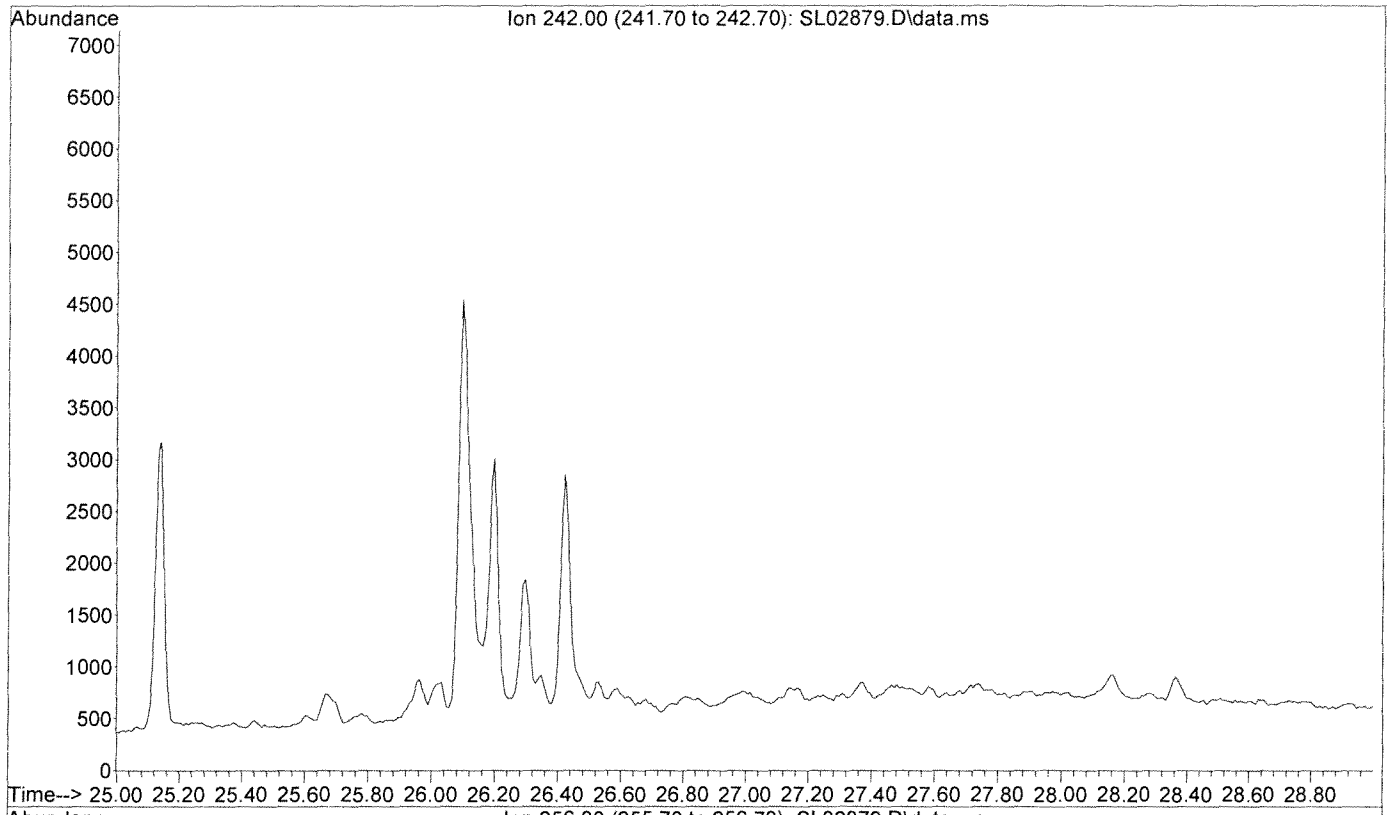
US EPA ARCHIVE DOCUMENT

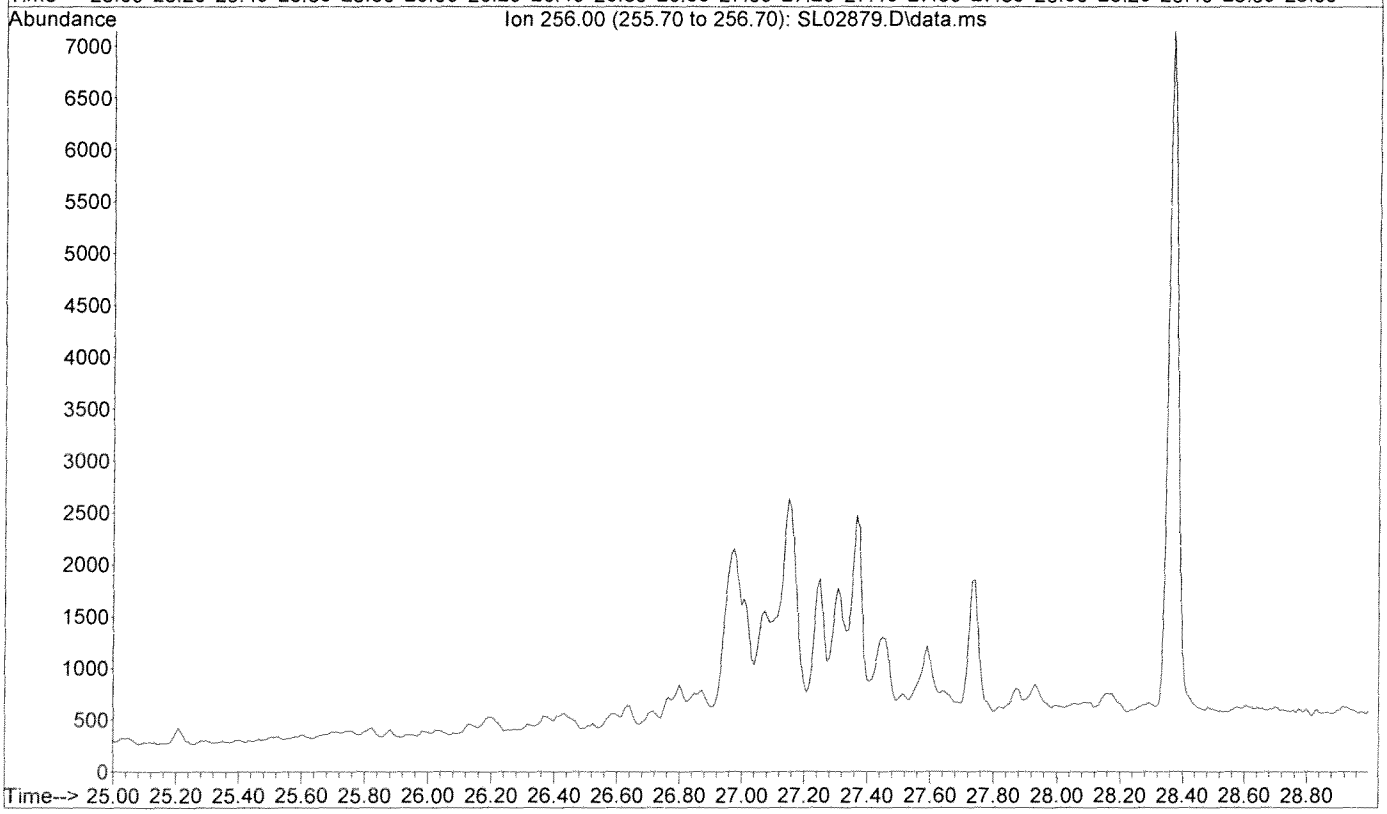
File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6



US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\022712\SL02879.D
Operator : Syslo
Acquired : 27 Feb 2012 23:40 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info : 275-55-05 EOB Duplicate
Vial Number: 6





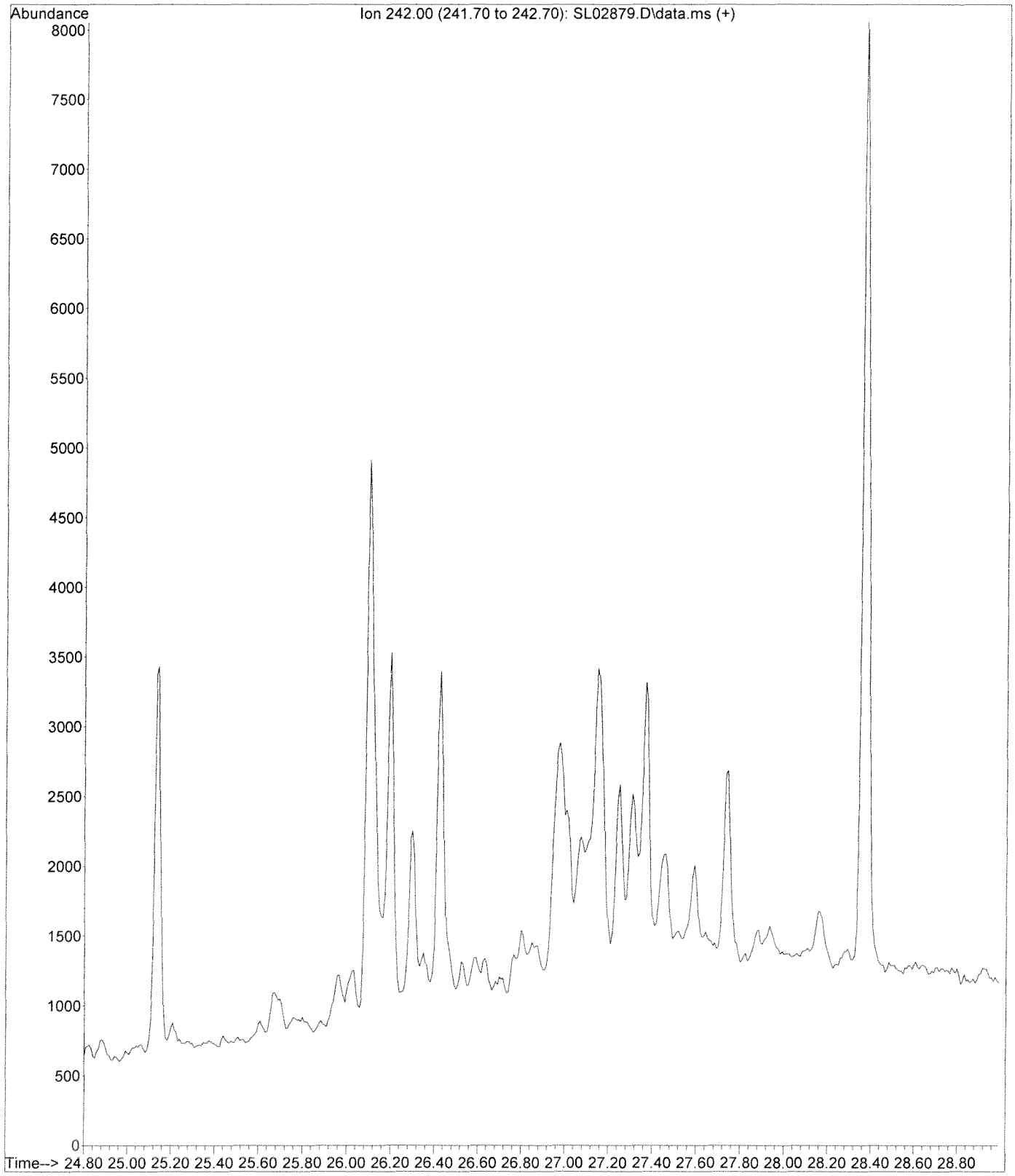
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\022712\SL02879.D
Operator    : Syslo
Acquired    : 27 Feb 2012  23:40      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0003 EOS
Misc Info   : 275-55-05 EOB Duplicate
Vial Number: 6
```



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US EPA ARCHIVE DOCUMENT

3/1/12 Enbridge QL + Hopkinsville

Logbook # SERAS-I-0121

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SERAS, GC/MS Injection Log

GC/MS System: "SLICK II" S/N#'s US10915004/US90432092 DROTPH05(6)1.M

Project Name: Enbridge Oil — Method File: DFTPP/ OILS/MS2

Work Assign.#: 6-017 — Inj. Volume: 1ul

Analysis/Date: TPH + Fingerprint 3/27/12 — Analyst: Sgile

ALS	FILE ID	SAMPLE ID	REAC ID	Date of Analysis	TIME Inj.	COMMENTS
99	SL02870	50ppm DFTPP	RVB0025	2/27/12	15:07	ok 1396-1382
71	SL02871	1000ppm TPH CCV	RVB0003		15:31	
72	SL02872	10ppm SHC/PAH Resolution Mix	RTL0035		16:32	MALL DLC ok
73	SL02873	10K CG-117 Ref Oil	RVL0003	2/27/12	17:34	
~~1~~	~~SL02874~~	~~BLK022712WD~~	~~re-extracted~~		X	~~CONTR ALL Blank~~
1	SL02875	BLK022712WD	Blank		X	ok
1	SL02876	BLK022712WD	Blank		18:44	
2	SL02877	SERAS-017-0003			19:33	
3	SL02878	SERAS-017-0003dup			20:35	
4	SL02879	SERAS-017-0004			21:37	
5	SL02878	SERAS-017-0004			22:38	SKIM from Bucket
6	SL02879	SERAS-017-0003dup		2/27/12	23:40	Early Dup
7	SL02880	10K CG-117 ref oil	RVL0003	2/28/12	00:41	D-1739 ref
8	SL02881	SERAS-017-0004 dup		2/28/12	1:42	Qual
—	SL	only by Dept		3/1/12		
99	SL02882	50ppm DFTPP		3/1/12	RVB0025	8:28 Pass 1395-1383
71	SL02883	10ppm SHC/PAH Res Mix	RTL0035	3/1/12	12:50	
72	SL02884	10K CG-117 ref oil	RVL0003		13:51	
1	SL02885	1000ppm TPH CCV/LCS	RVB0003		14:54	
2	SL02886	BLK030112V	Blank		15:41	0.1g → 10ml
3	SL02887	FOR-TRENCH	R2020118-01		17:33	0.055g → 5.0ml
4	SL02888	FOR-TRENCH dup	R2020118-01d		18:34	0.105g → 5.0ml
5	SL02889	FOR-CREEK			19:36	0.1485g → 5.0ml
6	SL02890	SERAS-017-0005			20:37	0.104g → 10ml
7	SL02891	SERAS-017-0005 dup			21:37	0.115g → 10ml
8	SL02892	SERAS-017-0006			22:38	0.102g → 10ml
9	SL02893	10K Alnt Oil		3/1/12	23:39	Used Natr 0.1
10	SL02894	10K CG-117 Reference	RVL0003	3/2/12	00:40	
11	SL02895	CLC B60	CLC 0.1	3/2/12	1:40	0.95g → 10ml

	REAC ID	Standard Description	Exp.date	Conc.	Ref. P.	Comment
1	RVB0025	DFTPP	8/14/12	50ppm		
2	RVB0003	Calibration Check	8/12/12	1000ppm		TPH
3	RVB0058	Internal Standard	5/14/12	500ppm		Spk 20ul → 5ml
4	RVL0003	CG-117 Ref. Fuel	5/24/12	10 Pct		D-1739 ref.
5	RTL0035	SHC/PAH Mix	Qual	1000ppm		Res/Mat/Diesel
6						

Reviewed By: Jerome Odencrantz — Date Checked: 03-06-12

```
   Sequence Name: C:\msdchem\1\sequence\030112.s
         Comment: Flemingsburg/Enbridge Oil
        Operator: Syslo
       Data Path: C:\MSDCHEM\1\DATA\030112\
Instrument Control  Pre-Seq Cmd:
Data Analysis       Pre-Seq Cmd:

Instrument Control Post-Seq Cmd:
Data Analysis      Post-Seq Cmd:

   Method Sections To Run      On A Barcode Mismatch
    (X) Full Method              (X) Inject Anyway
    ( ) Reprocessing Only        ( ) Don't Inject

   ------------------------------------------------------------------------------
   Line                    Sample Name/Misc Info
 1)  Sample        99  SL02882  DFT8270D 50 DFTPP
 2)  Sample        71  SL02883  OILSIMS2 10ppm SHC/PAH
 3)  Sample        72  SL02884  OILSIMS2 10K CG-117 Reference
 4) Sample          1  1000ppm TPH CCV
     Datafile          SL02885
     Method            DROTPH051611
 5) Sample          2  BLK030112
     Datafile          SL02886
     Method            DROTPH051611
 6) Sample          6  SERAS-017-0005
     Datafile          SL02890
     Method            DROTPH051611
 7) Sample          7  SERAS-017-0005 DUP
     Datafile          SL02891
     Method            DROTPH051611
 8) Sample          8  SERAS-017-0006
     Datafile          SL02892
     Method            DROTPH051611
 9)  Sample        10  SL02894  OILSIMS2 10K CG-117 Reference
```

Injection Log

Data Directory: C:\msdchem\1\DATA\030112\

SampleName	MiscInfo	Vial	Multiplier	Injection Time
1) SL02882.D				
50 DFTPP	RVB0025	99	1.000	1 Mar 2012 12:28
2) SL02883.D				
10ppm SHC/PAH	RTL0035 RES/MASS DIS	71	1.000	1 Mar 2012 12:50
3) SL02884.D				
10K CG-117 Reference	RUL0003	72	1.000	1 Mar 2012 13:51
4) SL02885.D				
1000ppm TPH CCV	RVB0003 for Surr.	1	1.000	1 Mar 2012 14:56
5) SL02886.D				
BLK030112	Waste Dilution Blank	2	1.000	1 Mar 2012 15:41
6) SL02887.D				
R202018-01	FOR-TRENCH .{0.055g	3	1.000	1 Mar 2012 17:33
7) SL02888.D				
R202018-01 dup	FOR-TRENCH .{0.105g	4	1.000	1 Mar 2012 18:34
8) SL02889.D				
R202018-02	FOR-CREEK.{0.148g in	5	1.000	1 Mar 2012 19:36
9) SL02890.D				
SERAS-017-0005	WCS-6B-072223-092910	6	1.000	1 Mar 2012 20:37
10) SL02891.D				
SERAS-017-0005 DUP	WCS-6B-072223-092910	7	1.000	1 Mar 2012 21:37
11) SL02892.D				
SERAS-017-0006	CL-6B-072223-092710-	8	1.000	1 Mar 2012 22:38
12) SL02893.D				
MotOil 10K	10k Used Motor OIL	9	1.000	1 Mar 2012 23:39
13) SL02894.D				
10K CG-117 Reference	RUL0003 EOS Referen	10	1.000	2 Mar 2012 00:40

• Flemingsburg Samples

** This is 28 min over 12hr clock but CG-117 is not a sample.

GC/MS QA-QC Check Report: 03/01/12

Tune File : C:\msdchem\1\DATA\030112\SL02882.D
Tune Time : 1 Mar 2012 12:28

Daily Calibration File : C:\msdchem\1\DATA\030112\SL02885.D

File	Sample	Surrogate Recovery %				Internal Standard Responses		
SL02885.D								
	1000ppm TP	51*	55*	61*	64*	358723	109515	150757
				134865				
SL02886.D								
	BLK030112	89*	96*	107*	114*	244273	73201	95670
				87506				
SL02890.D								
	SERAS-017-	83*	88*	102*	103*	324307	97344	131670
				115625				
SL02891.D								
	SERAS-017-	74*	80*	87*	96*	322263	94462	129000
				116518				
SL02892.D								
	SERAS-017-	78*	83*	91*	99*	321005	94522	131524
				117275				
SL02894.D								
	10K CG-117	0	0	0	0	727287*	207961	281953
				245390				

Created: Thu Jan 10 15:15:14 2013 Slick2

DFTPP

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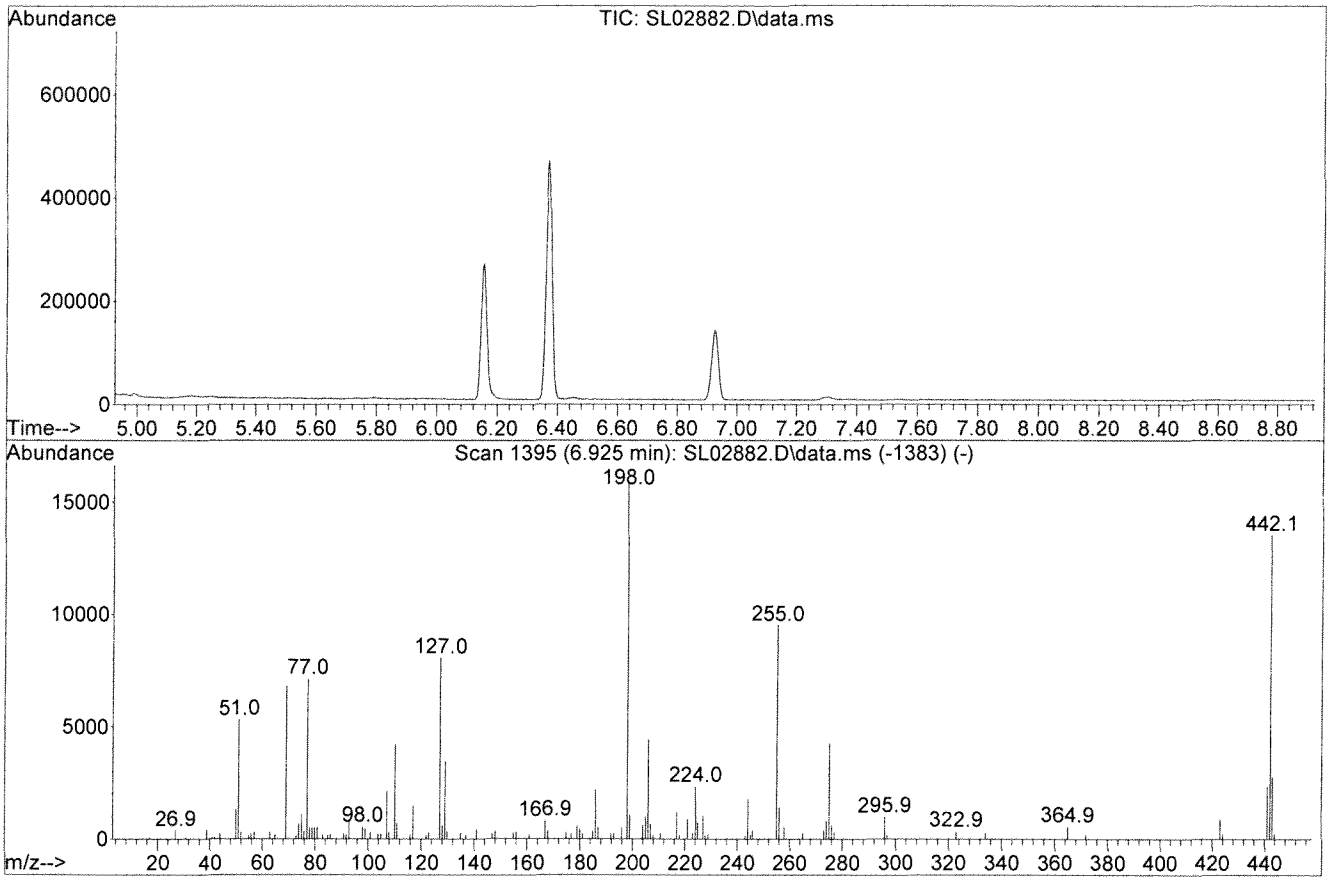
DFTPP - 8270D

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02882.D
Acq On : 1 Mar 2012 12:28
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Tailing: PCP = 1.13
Benzidine = 1.23
3,3'DDT dg = 18:20

Integration File: rteint.p

Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Title : DFTPP Method with 8270D criteria: 8/20/10
Last Update : Thu Dec 20 10:09:10 2012



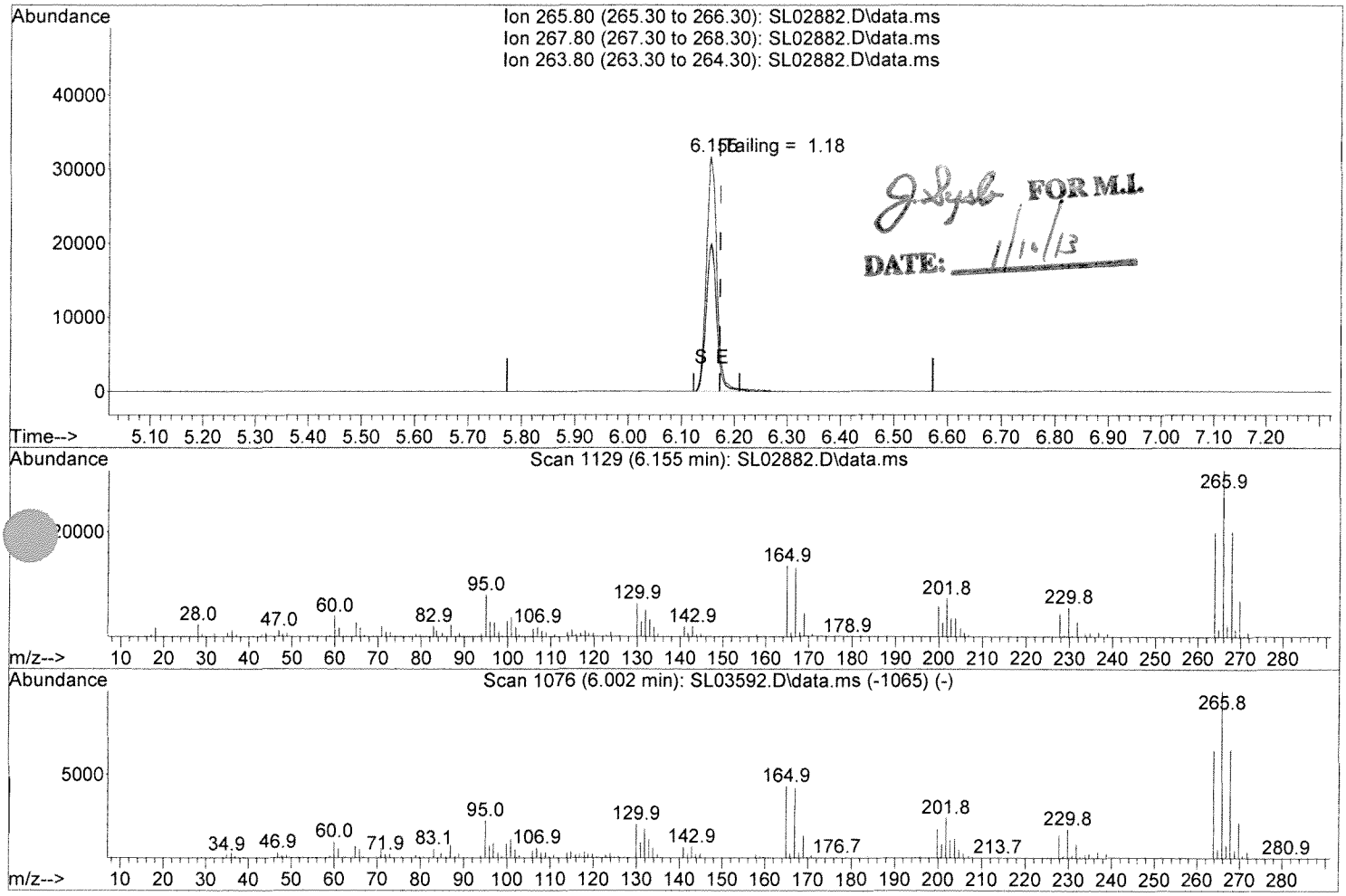
Spectrum Information: Scan 1395

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	33.4	5299	PASS
68	69	0.00	2	0.0	0	PASS
69	198	0.00	100	43.1	6837	PASS
70	69	0.00	2	0.0	0	PASS
127	198	10	80	50.7	8044	PASS
197	198	0.00	2	0.0	0	PASS
198	198	100	100	100.0	15852	PASS
199	198	5	9	6.7	1055	PASS
275	198	10	60	26.9	4267	PASS
365	198	1	100	3.4	541	PASS
441	442	0.01	24	17.2	2322	PASS
442	198	50	100	85.3	13516	PASS
443	442	15	24	20.3	2750	PASS

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02882.D
Acq On : 1 Mar 2012 12:28
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Mar 01 12:41:06 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



TIC: SL02882.D\data.ms

(2) Pentachlorophenol

6.155min (-0.017) 53.09 ug/mL

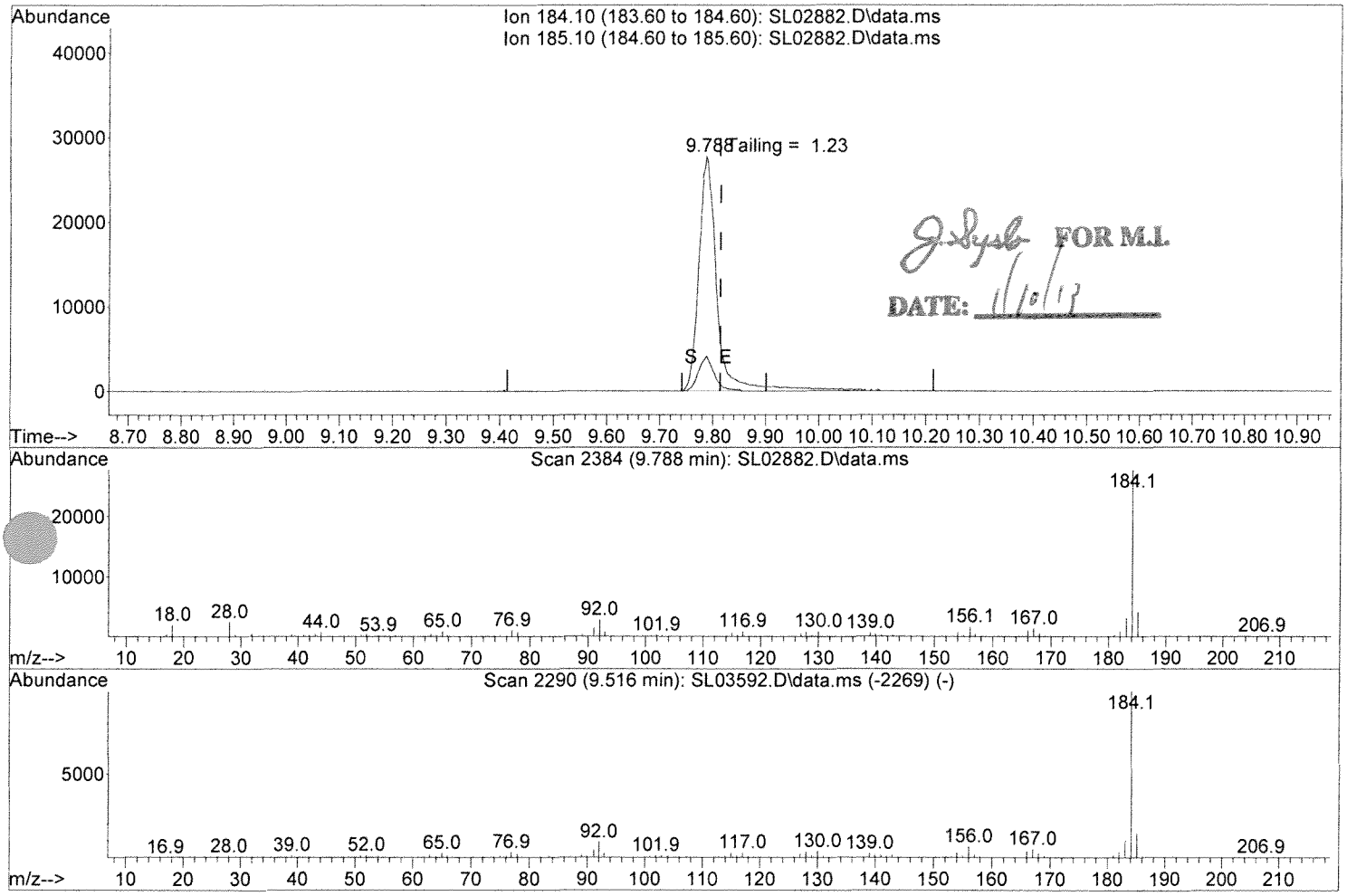
response 43846

Ion	Exp%	Act%
265.80	100	100
267.80	64.50	63.30
263.80	63.60	62.18
0.00	0.00	0.00

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02882.D
Acq On : 1 Mar 2012 12:28
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Mar 01 12:41:06 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



FOR M.I.
DATE: 1/10/13

TIC: SL02882.D\data.ms

(5) Benzidine

9.788min (-0.026) 18.89 ug/mL

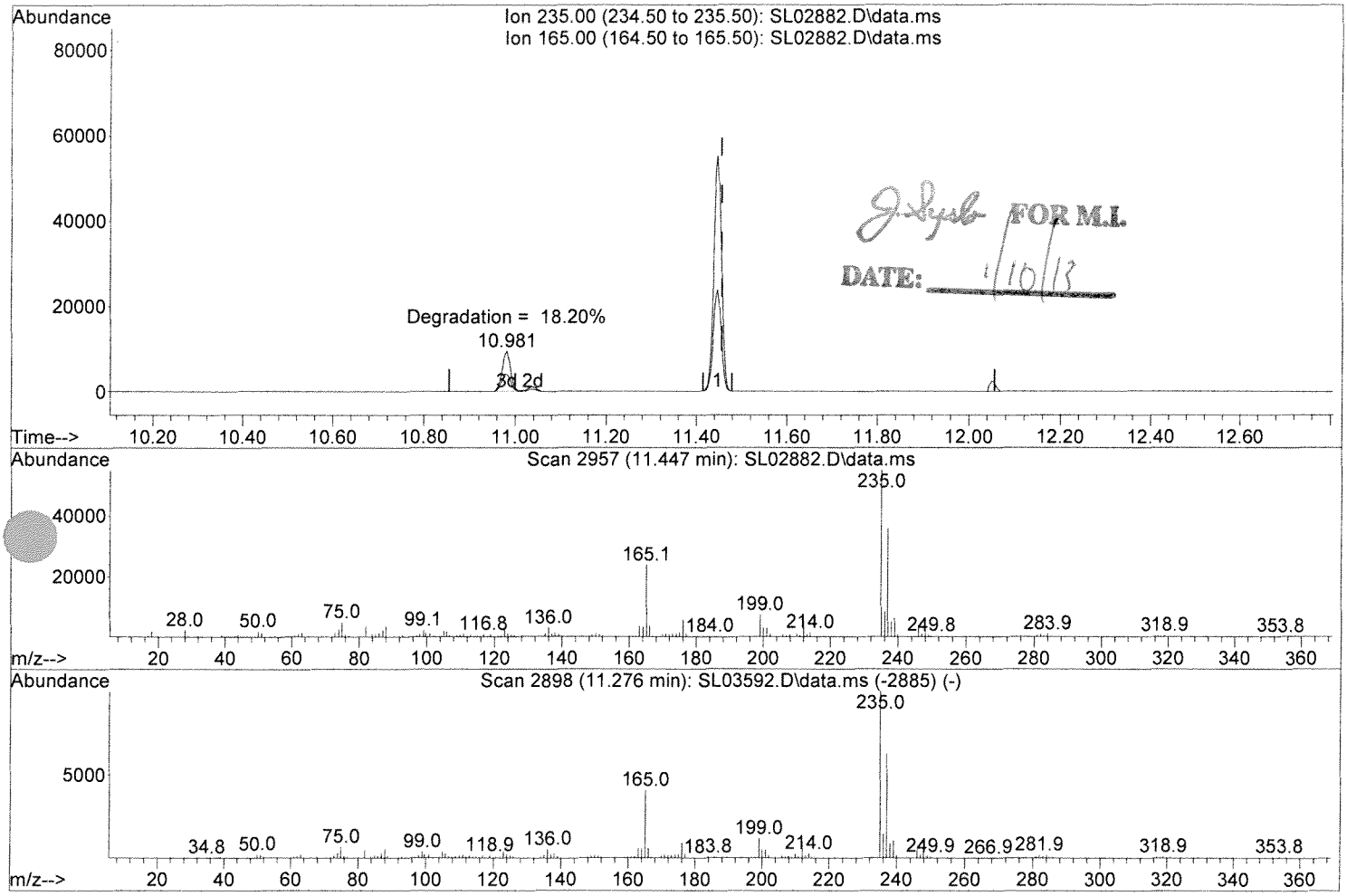
response 62974

Ion	Exp%	Act%
184.10	100	100
185.10	14.60	13.43
0.00	0.00	0.00
0.00	0.00	0.00

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02882.D
Acq On : 1 Mar 2012 12:28
Operator : Syslo
Sample : 50 DFTPP
Misc : RVB0025
ALS Vial : 99 Sample Multiplier: 1

Quant Time: Mar 01 12:41:06 2012
Quant Method : C:\MSDCHEM\1\METHODS\DFT8270D.M
Quant Title : DFTPP Method with 8270D criteria: 8/20/10
QLast Update : Thu Jan 26 14:45:51 2012
Response via : Initial Calibration



J. Lysik FOR M.L.
DATE: 1/10/13

TIC: SL02882.D\data.ms

(6) DDT

11.447min (-0.009) 44.58 ug/mL

response 66123

Ion	Exp%	Act%
235.00	100	100
165.00	51.80	43.32
0.00	0.00	0.00
0.00	0.00	0.00

10 ppm SOC/PAH for Res/Mun
Discrimination

US EPA ARCHIVE DOCUMENT

Table 1.0: Results of ASTM D-5739 Resolution and Mass Discrimination Performance Checks Applied to *draft* SERAS GC/MS Method 1803: Oil Fingerprinting, Characterization and TPH Analysis

Enbridge Oil: Fingerprinting 2012: WA# 0-017

Method: *draft* SERAS SOP 1803

		Resolution Check[1]			Mass Discrimination Checks							Resolution Check: Syslo/SERAS
Date Checked	GC/MS File	C17/pristane % Resolution	C18/phytane % Resolution	Acceptable % Resolution	Naphthalene area	Fluoranthene area	Benzo(g,h,i)perylene area	Nap/Fluo Ratio area/area	B(ghi)/Fluo Ratio area/area	Acceptable Ratios Nap/Fluo	Acceptable Ratios B(ghi)/Fluo	Benzo(b)/Benzo(k) fluoranthene ratio
Enbridge Oil TPH/Fingerprinting: WA# 0-017												
2/23/2012	SL02855[3]	99.8%	99.9%	> 50% v/v	1100101	1358009	1167661	0.81	0.86	<= 2.0	>= 0.20	68.70%
2/24/2012	SL02855[2]	98.5%	99.7%	> 50% v/v	454731	543693	439965	0.84	0.81	<= 2.0	>= 0.20	68.43%
2/27/2012	SL02872[2]	98.5%	99.7%	> 50% v/v	507043	611080	485460	0.83	0.79	<= 2.0	>= 0.20	65.80%
3/1/2012	SL02883[2]	99.0%	99.4%	> 50% v/v	506865	655953	569653	0.77	0.87	<= 2.0	>= 0.20	63.55%
Lab Maintenance Samples: 0-011												
5/23/2011	SL01907	97.94	98.85	> 50% v/v	487005	429769	381358	1.13	0.89	<= 2.0	>= 0.20	62.83
5/24/2011	SL01920	99.04	99.87	> 50% v/v	545444	544337	421740	1.00	0.77	<= 2.0	>= 0.20	61.02
DRO-TPH Soil MDL Study August 11, 2011 on Slick2 - After moving instrument to new lab.												
8/11/2011	SL02081	99.05	99.65	> 50% v/v	504208	637067	494345	0.79	0.78	<= 2.0	>= 0.20	61.40
Imperial Oil fingerprints: November - December 2011 on Slick 2												
11/14/2011	SL02274[2]	98.92	99.85	> 50% v/v	891148	1110386	936738	0.80	0.84	<= 2.0	>= 0.20	63.93
12/1/2011	SL02332[3]	98.14	100.28	> 50% v/v	158336	197959	135116	0.80	0.68	<= 2.0	>= 0.20	59.12
12/2/2011	SL02339[2]	99.01	99.82	> 50% v/v	1666437	2018693	1619360	0.83	0.80	<= 2.0	>= 0.20	67.52
12/3/2011	SL02364[2]	99.06	99.88	> 50% v/v	1246967	1470915	1193650	0.85	0.81	<= 2.0	>= 0.20	68.64
12/5/2011	SL02367[2]	99.04	99.82	> 50% v/v	1281093	1527311	1134178	0.84	0.74	<= 2.0	>= 0.20	69.13
12/6/2011	SL02382[2]	98.87	99.87	> 50% v/v	1152427	1408258	1191425	0.82	0.85	<= 2.0	>= 0.20	67.25
CUC Oil fingerprints: December 2011 on Slick 2												
12/7/2011	SL02403[2]	99.93	99.73	> 50% v/v	1323413	1547091	1278611	0.86	0.83	<= 2.0	>= 0.20	68.49
12/9/2011	SL02419[2]	99.08	99.71	> 50% v/v	1202251	1425483	1171714	0.84	0.82	<= 2.0	>= 0.20	67.22
12/13/2011	SL02441[2]	98.92	99.76	> 50% v/v	1686396	2010249	1665781	0.84	0.83	<= 2.0	>= 0.20	66.62
12/14/2011	SL02456[2]	99.11	99.76	> 50% v/v	1180706	1397756	1127432	0.84	0.81	<= 2.0	>= 0.20	64.46

All resolution and mass discrimination checks are performed using a 10ug/mL PAH & SHC standard unless otherwise noted.

(1) The resolution check is determined using the Enviroquant ChromEval option in the Qedit mode.
(2) Resolution check performed using 10ppm SHC/PAH mixture in SIM mode.
(3) Resolution check performed using 25ppm SHC/PAH mixture in SIM mode.

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02883.D
Acq On : 1 Mar 2012 12:50
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Mar 01 17:20:31 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d4-1,4-Dichlorobenzene	8.144	152	139683	10.00	ug/mL	0.00
14) d8-Naphthalene	10.968	136	447243	10.00	ug/mL	0.00
23) d10-Phenanthrene	18.708	188	485751	10.00	ug/mL	0.00
41) d12-Chrysene	25.141	240	509820	10.00	ug/mL	0.00
50) d12-Perylene	28.373	264	488642	10.00	ug/mL	0.00
59) d30-Tetradecane	13.536	66	152347	10.00	ug/mL	0.00
73) d50-Tetracosane	23.783	66	197232	10.00	ug/mL	0.00
86) d74-Hexatriacontane	34.613	66	174375	10.00	ug/mL	0.00
System Monitoring Compounds						
33) d10-Anthracene {S}	18.829	188	429171	8.80	ug/mL	0.00
Spiked Amount 20.000			Recovery = 44.00%			
35) d14-o-Terphenyl {S}	19.732	244	196735	8.61	ug/mL	0.00
Spiked Amount 20.000			Recovery = 43.05%			
40) 5a-Androstane {S}	21.146	260	26073	9.19	ug/mL	0.00
Spiked Amount 20.000			Recovery = 45.95%			
85) d62-Triacontane {S}	28.165	66	170303	12.36	ug/mL	0.00
Spiked Amount 20.000			Recovery = 61.80%			
Target Compounds						Qvalue
15) Naphthalene	11.011	128	506856	8.89	ug/mL	100
16) 2-Methylnaphthalene	12.641	142	329477	9.03	ug/mL	100
21) Acenaphthylene	14.795	152	533290	9.33	ug/mL	100
22) Acenaphthene	15.239	154	304613	9.00	ug/mL	100
24) Fluorene	16.462	166	371844	8.94	ug/mL	100
28) Dibenzothiophene	18.456	184	544919	8.67	ug/mL	100
32) Phenanthrene	18.760	178	562682	8.80	ug/mL	100
34) Anthracene	18.864	178	538101	8.98	ug/mL	100
36) 2-Methylanthracene	20.113	192	362717	9.49	ug/mL	100
42) Fluoranthene	21.658	202	655953	8.52	ug/mL	100
43) Pyrene	22.187	202	667166	8.63	ug/mL	100
46) Benzo(a)anthracene	25.100	228	627494	9.26	ug/mL	100
47) Chrysene	25.200	228	576088	9.00	ug/mL	100
51) Benzo(b)fluoranthene	27.574	252	590399	8.86	ug/mL	100
52) Benzo(k)fluoranthene	27.615	252	628253	9.24	ug/mL	100
53) Benzo(e)pyrene	28.140	252	617202	8.66	ug/mL	100
54) Benzo(a)pyrene	28.249	252	592346	9.52	ug/mL	100
55) Perylene	28.423	252	562635	8.91	ug/mL	100
56) Indeno(1,2,3-cd)pyrene	31.073	276	691907	9.73	ug/mL	100
57) Dibenzo(a,h)anthracene	31.114	278	562415	10.07	ug/mL	100
58) Benzo(g,h,i)perylene	31.856	276	569653	9.29	ug/mL	100
60) Octane C8	4.455	85	75739	9.81	ug/mL	100
61) Nonane C9	6.158	85	54579	9.82	ug/mL	100
62) Decane C10	7.853	85	47368	10.08	ug/mL	100
63) Undecane C11	9.483	85	53419	10.25	ug/mL	100
64) Dodecane C12	11.018	85	66957	10.18	ug/mL	100
65) Tridecane C13	12.459	85	76024	10.49	ug/mL	100
66) Tetradecane C14	13.820	85	85097	10.57	ug/mL	100
67) Pentadecane C15	15.101	85	92842	10.87	ug/mL	100
68) Hexadecane C16	16.309	85	97737	10.97	ug/mL	100
69) Heptacosane C17	17.458	85	98252	11.08	ug/mL	100
70) Pristane	17.531	85	81850	10.81	ug/mL	100
71) Octacosane C18	18.543	85	109109	11.42	ug/mL	100

US EPA ARCHIVE DOCUMENT

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02883.D
Acq On : 1 Mar 2012 12:50
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Mar 01 17:20:31 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
72) Phytane	18.656	85	110184	11.09	ug/mL	100
74) Nonadecane C19	19.584	85	112504	10.02	ug/mL	100
75) Eicosane C20	20.573	85	116028	10.32	ug/mL	100
76) Heneicosane C21	21.519	85	118891	10.53	ug/mL	100
77) Docosane C22	22.421	85	121301	10.72	ug/mL	100
78) Tricosane C23	23.289	85	124434	11.13	ug/mL	100
79) Tetracosane C24	24.122	85	128648	11.22	ug/mL	100
80) Pentacosane C25	24.925	85	127066	11.64	ug/mL	100
81) Hexacosane C26	25.691	85	128427	12.05	ug/mL	100
82) Heptacosane C27	26.441	85	128446	12.57	ug/mL	100
83) Octacosane C28	27.149	85	130350	12.96	ug/mL	100
84) Nonacosane C29	27.840	85	132460	13.67	ug/mL	100
87) Triacontane C30	28.540	85	136055	8.03	ug/mL	100
88) Hentriacontane C31	29.331	85	131194	8.46	ug/mL	100
89) Dotriacontane C32	30.240	85	125782	8.94	ug/mL	100
90) Tritriacontane C33	31.298	85	124077	9.65	ug/mL	100
91) Tetratriacontane C34	32.547	85	122856	10.17	ug/mL	100
92) Pentatriacontane C35	34.047	85	115097	10.72	ug/mL	100
93) Hexatriacontane C36	35.821	85	118607	10.78	ug/mL	100
94) Heptatriacontane C37	37.937	85	110304	11.75	ug/mL	100
95) Octatriacontane C38	40.478	85	108687	12.42	ug/mL	100
97) Tetracontane C40	47.217	85	98319	14.52	ug/mL	100

(#) = qualifier out of range (m) = manual integration (+) = signals summed

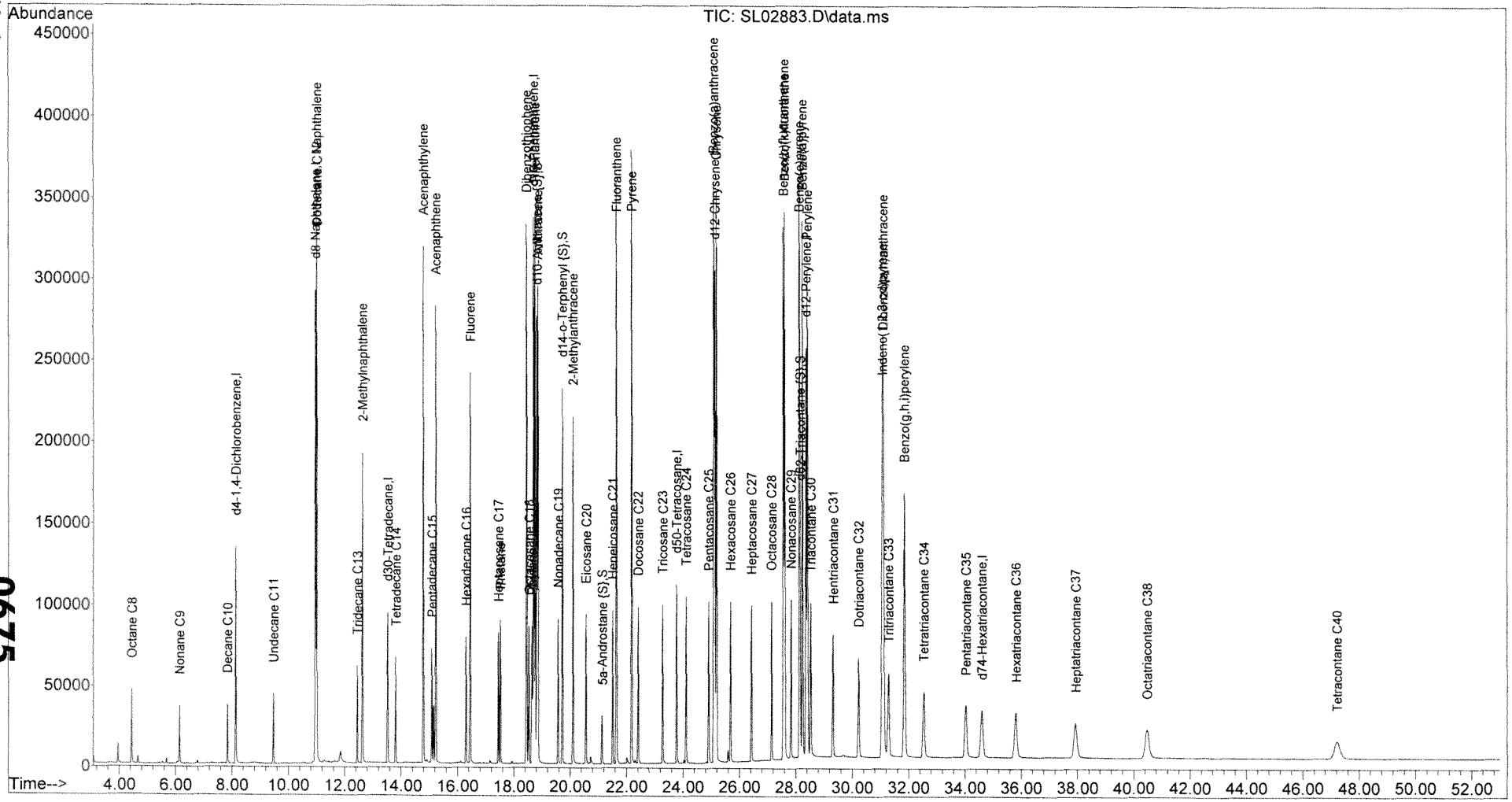
SERAS-017-DTM-011413_3

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02883.D
Acq On : 1 Mar 2012 12:50
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Mar 01 17:20:31 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3

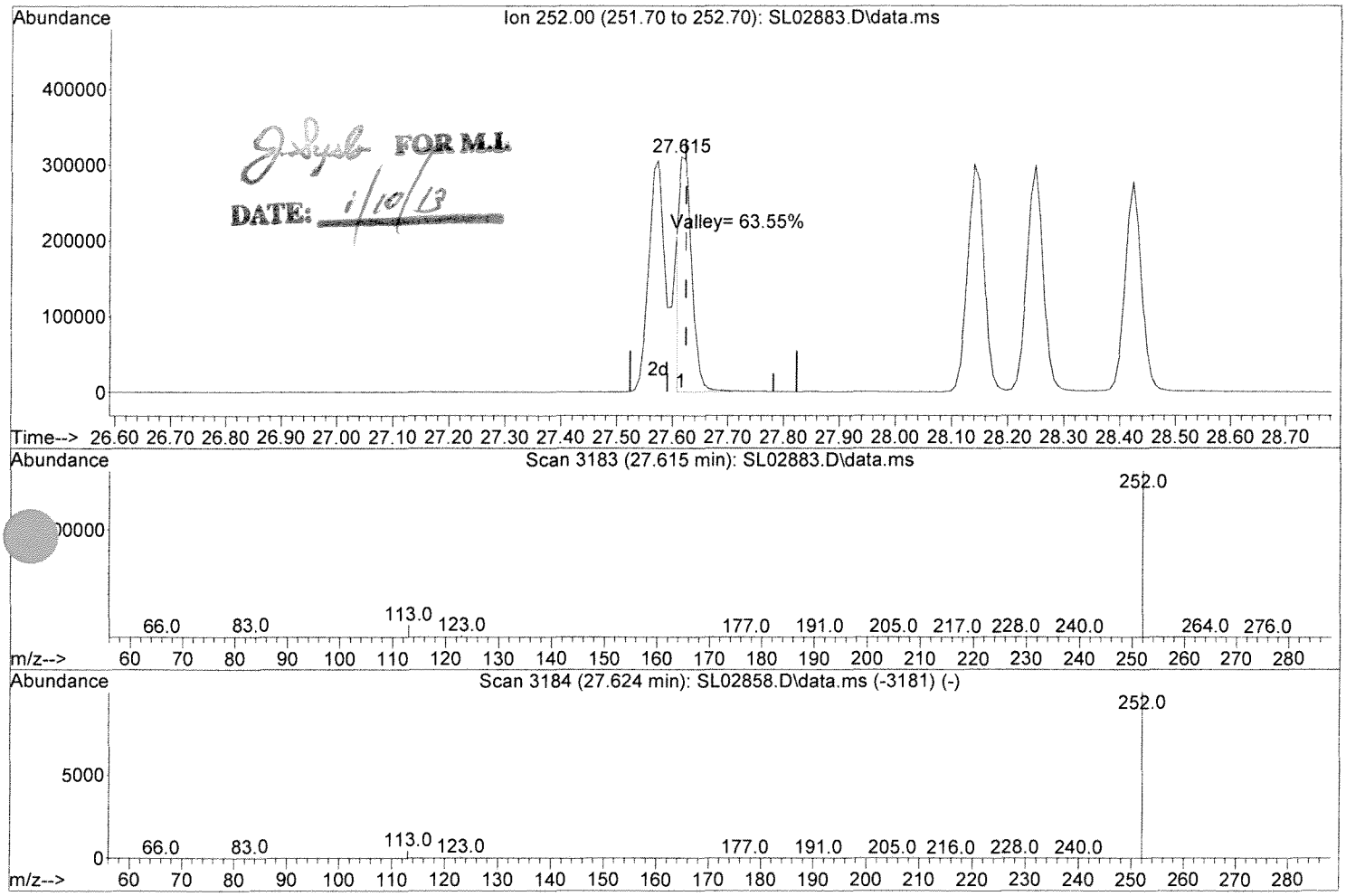
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Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02883.D
Acq On : 1 Mar 2012 12:50
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Mar 01 17:20:31 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration



TIC: SL02883.D\data.ms

(52) Benzo(k)fluoranthene

27.615min (-0.008) 9.24 ug/mL

response 628253

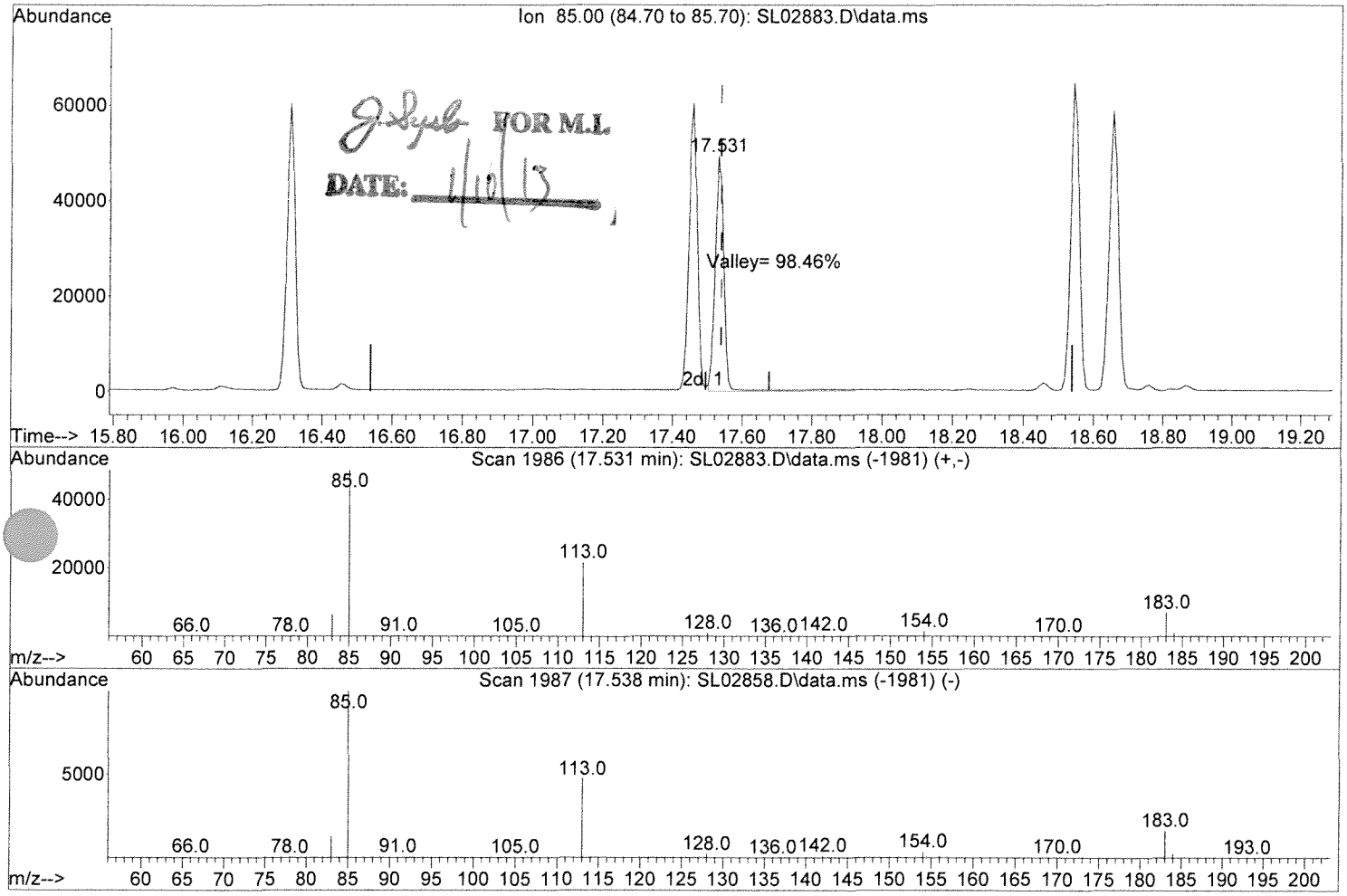
Ion	Exp%	Act%
252.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02883.D
Acq On : 1 Mar 2012 12:50
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Mar 01 17:20:31 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration



FOR M.L.
DATE: 1/10/13

TIC: SL02883.D\data.ms

(70) Pristane

17.531min (-0.007) 10.81 ug/mL

response 81850

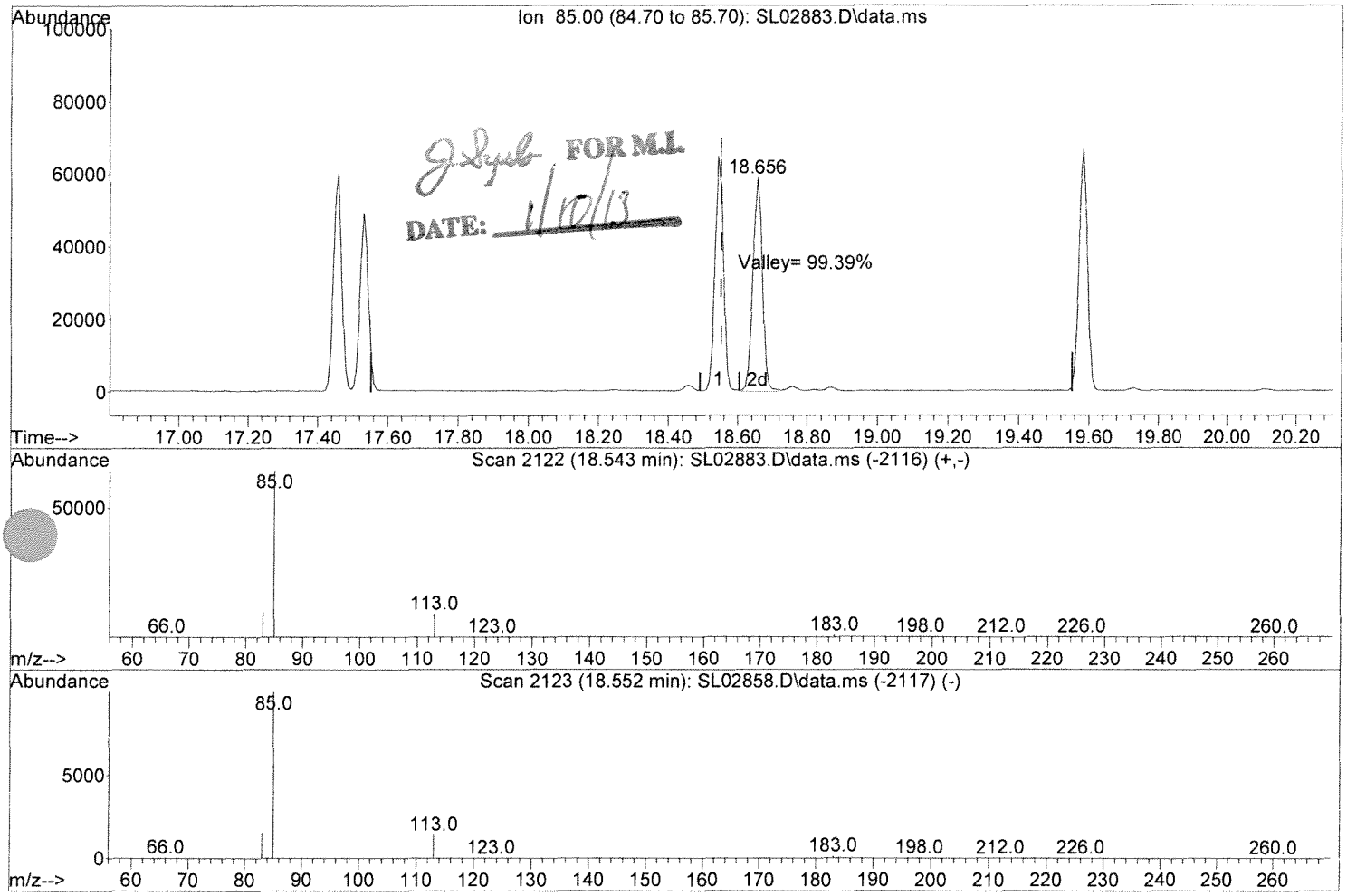
Ion	Exp%	Act%
85.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

Quantitation Report (Qedit)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02883.D
Acq On : 1 Mar 2012 12:50
Operator : Syslo
Sample : 10ppm SHC/PAH
Misc : RTL0035 RES/MASS DISC.
ALS Vial : 71 Sample Multiplier: 1

Quant Time: Mar 01 17:20:31 2012
Quant Method : C:\MSDCHEM\1\METHODS\OILSIMS2.M
Quant Title : 12/21/10 Slick2 PAH/SHC ICAL
QLast Update : Fri Feb 24 18:58:52 2012
Response via : Initial Calibration



FOR M.L.
DATE: 1/10/13

TIC: SL02883.D\data.ms

(71) Octacosane C18

18.543min (-0.009) 11.42 ug/mL

response 109109

Ion	Exp%	Act%
85.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

SERAS-017-DTM-011413_3

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02884.D
Acq On : 1 Mar 2012 13:51
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003
ALS Vial : 72 Sample Multiplier: 1

Quant Time: Mar 01 17:21:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.708	188	561636	10.00	ug/mL	0.00
5) d30-Tetradecane	13.536	66	162366	10.00	ug/mL	0.00
6) d50-Tetracosane	23.792	66	220377	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.613	66	208229	10.00	ug/mL	0.02
System Monitoring Compounds						
2) d10-Anthracene {S}	18.829	188	4726	0.08	ug/mL	0.00
Spiked Amount 20.000			Recovery =	0.40%		
3) d14-o-Terphenyl {S}	19.680	244	840	0.03	ug/mL	-0.05
Spiked Amount 20.000			Recovery =	0.15%		
4) 5a-Androstane {S}	21.146	260	515	0.16	ug/mL	0.00
Spiked Amount 20.000			Recovery =	0.80%		
7) d62-Triacontane {S}	28.190	66	397	0.91	ug/mL	0.03
Spiked Amount 20.000			Recovery =	4.55%		
Target Compounds						Qvalue

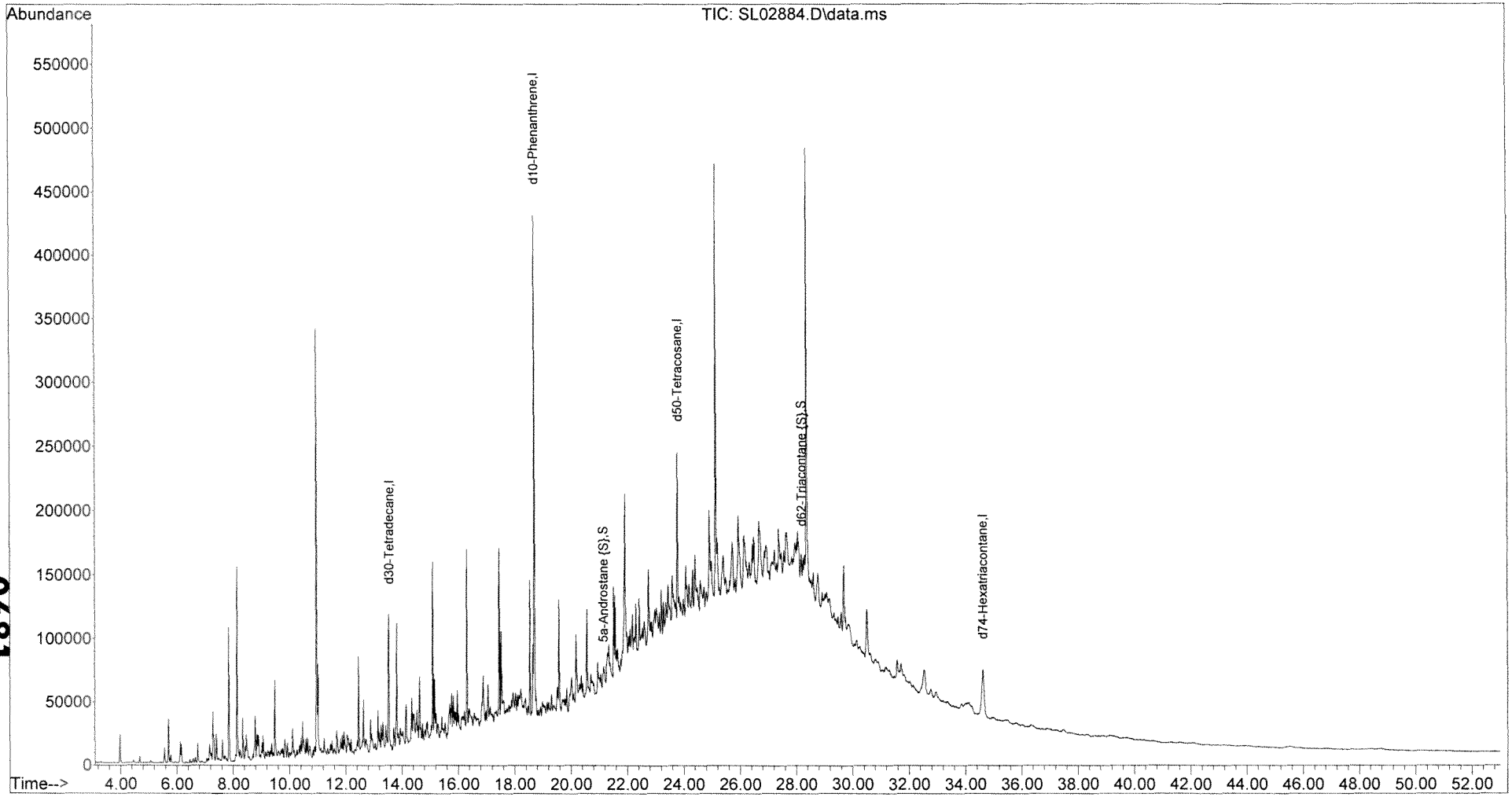
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02884.D
Acq On : 1 Mar 2012 13:51
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003
ALS Vial : 72 Sample Multiplier: 1

Quant Time: Mar 01 17:21:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3

0681

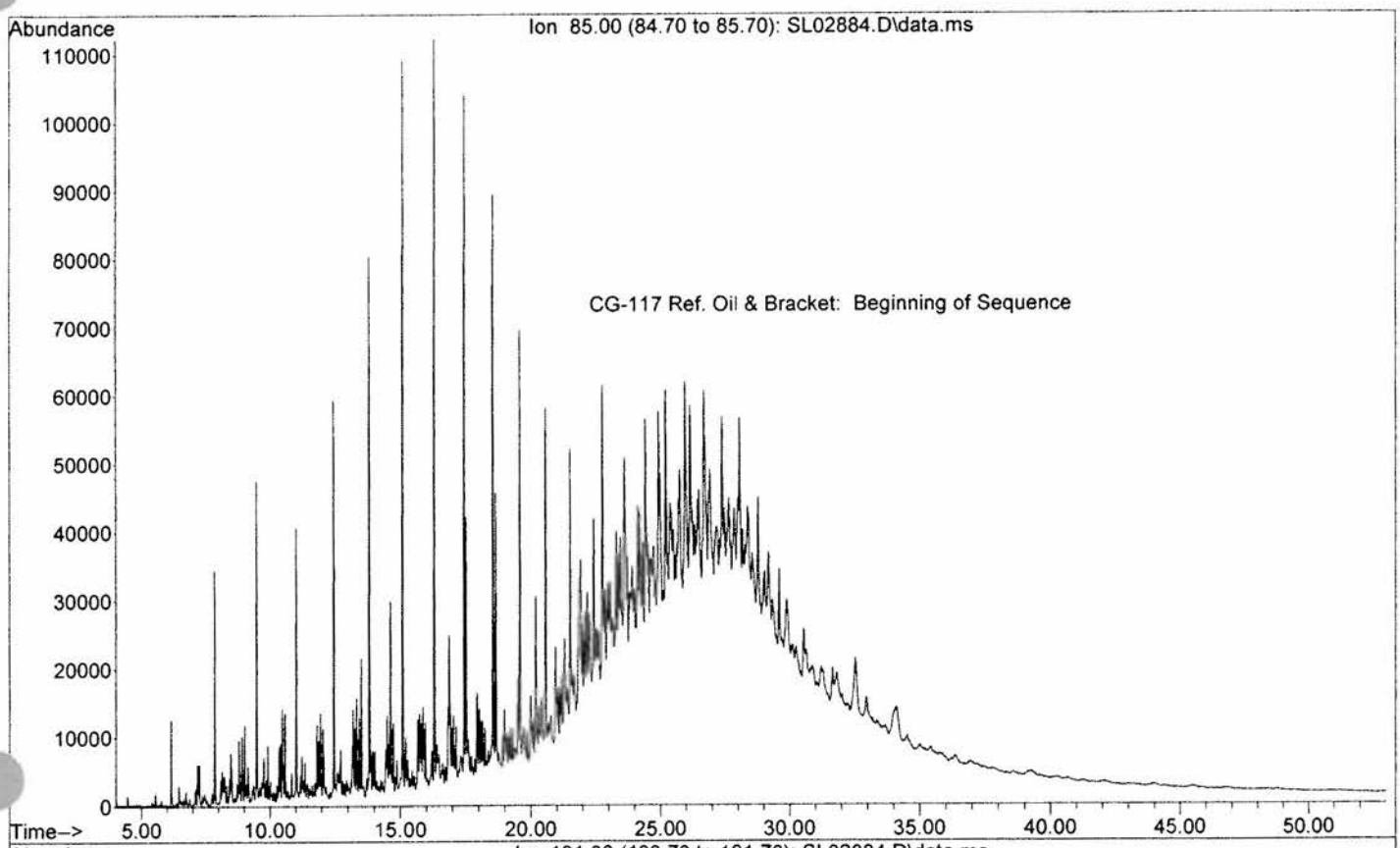


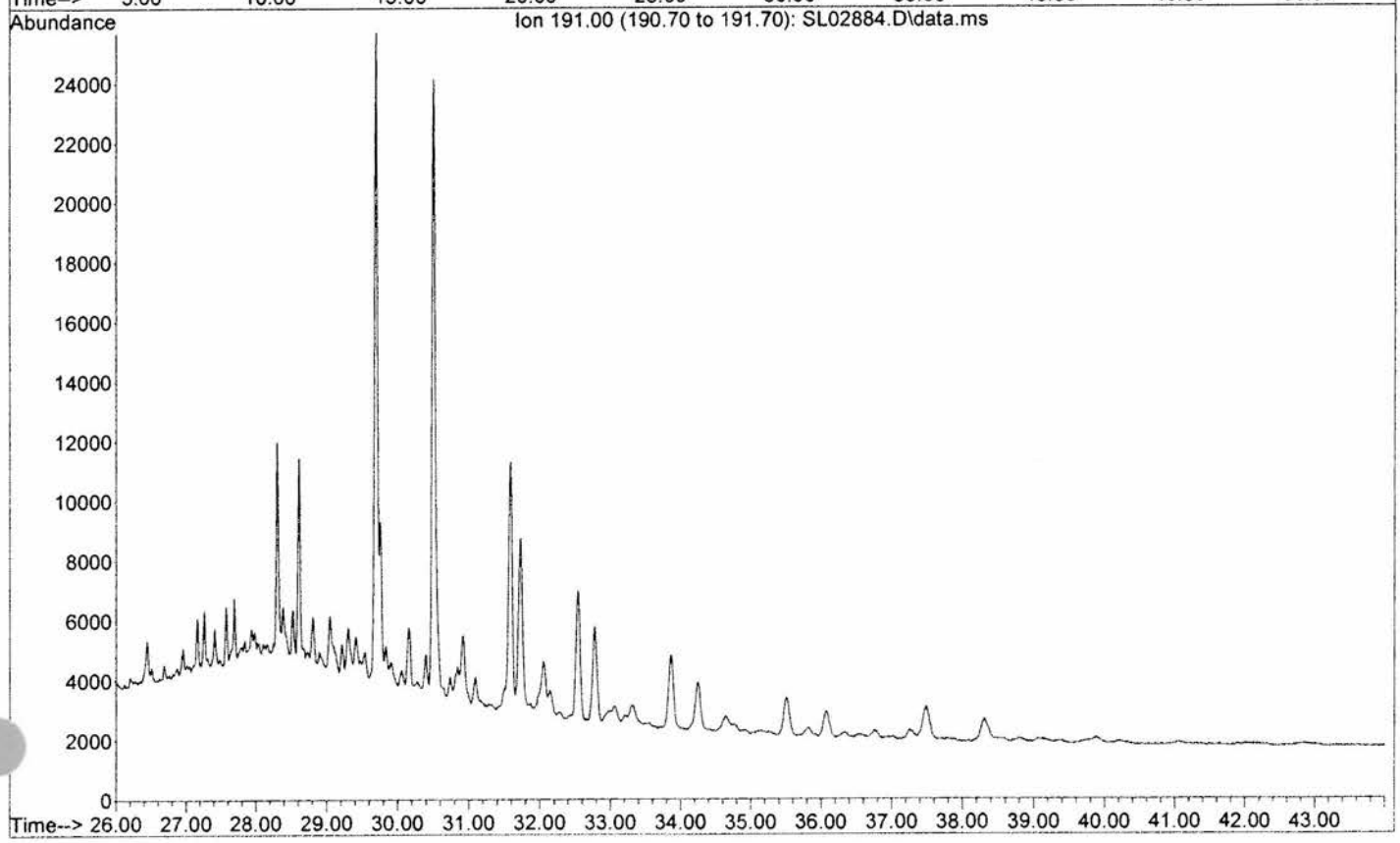
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! File  : FIREF117.MAC
! J. Syslo MACRO for reference fingerprint using CG117: 11/22/09
!  Hopanes are normalized for easy viewing!
! Draws chromatogram in window 2 and window 1 . .  you have to print "TIC &
Spectrum"
!  I have a lot of old CG-117's like that - better reference"  JS

NAME FIREF117
format separated,,,0
EIC 4:55,85
exchange r0,x
EIC 26:44,191
exchange r0,y
draw 2,y
draw 1,x
REMOVE FIREF117
clear
return
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US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02884.D
Operator : Syslo
Acquired : 1 Mar 2012 13:51 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003
Vial Number: 72





1000ppm TPH CCV

w/ IS + Surr.

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

TPH Daily Calibration Check Results
#2 Diesel TPH Calibration Range - Using draft SERAS GC/MS Method 1841

Calibration Check Date	ICAL Date	Project	Wa#	GC/MS Datafile	TPH Response	d30-IS Area	d50-IS Area	d74-IS Area	Cal Check RF	Calibration Average RF	% Diff.
05/17/11	05/16/11	WSoil MDL	0-011	SL01889	7932426	132429	130508	51192	0.75756	0.72959	3.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01942	7860351	128757	120362	11883	0.90348	0.72959	23.83
06/09/11	05/16/11	CUC-Power Plant	0-135	SL01945	6544592	104943	128978	88104	0.60970	0.72959	16.43
07/08/11	05/16/11	CUC-Power Plant	0-135	SL01970	6848120	109628	127366	77204	0.65387	0.72959	10.38
07/11/11	05/16/11	CUC-Power Plant	0-135	SL01974	7184921	121573	136941	70557	0.65502	0.72959	10.22
07/12/11	05/16/11	CUC-Power Plant	0-135	SL01989	7699927	130026	148583	96240	0.61624	0.72959	15.54
07/27/11	05/16/11	CUC-Power Plant	0-135	SL02012	5526770	90684	107174	65457	0.62968	0.72959	13.69
08/01/11	05/16/11	CUC-Power Plant	0-135	SL02036	7282274	117359	139028	90753	0.62934	0.72959	13.74
08/08/11	05/16/11	Move-Maintenance	0-011	SL02067	20772937	342644	403655	197034	0.66062	0.72959	9.45
08/08/11	05/16/11	Move-Maintenance	0-011	SL02069	9013310	151400	176386	86475	0.65273	0.72959	10.54
08/10/11	05/16/11	Move-Maintenance	0-011	SL02071	8539256	140844	161739	77290	0.67438	0.72959	7.57
08/11/11	05/16/11	TPH Soil MDL	0-011	SL02079	8559600	142237	165518	79165	0.66367	0.72959	9.03
08/12/11	05/16/11	TPH Water MDL	0-011	SL02093	8541528	140220	170089	76028	0.66327	0.72959	9.09
08/23/11	05/16/11	CUC-Power Plant	0-135	SL02133	8279388	130044	151992	63182	0.71949	0.72959	1.38
08/24/11	05/16/11	CUC-Power Plant	0-135	SL02166	6757179	110307	122438	62687	0.68617	0.72959	5.95
09/21/11	05/16/11	CUC-Power Plant	0-135	SL02202	11190934	169011	205927	119691	0.67875	0.72959	6.97
09/22/11	05/16/11	CUC-Power Plant	0-135	SL02213	8986654	146134	179078	114918	0.61255	0.72959	16.04
02/02/12	05/16/11	Enbridge Oil	0-017	SL02671	10771363	175782	209958	153567	0.59918	0.72959	17.87
02/15/12	05/16/11	Enbridge Oil	0-017	SL02827	11689228	179024	220786	162003	0.62419	0.72959	14.45
02/24/12	05/16/11	Enbridge Oil	0-017	SL02857	11425492	166909	205857	151555	0.65373	0.72959	10.40
02/27/12	05/16/11	Enbridge Oil	0-017	SL02872	10457816	165200	202882	161132	0.59283	0.72959	18.74
03/01/12	05/16/11	Enbridge Oil	0-017	SL02885	7041868	109515	150757	134865	0.54694	0.72959	25.03

TPH Not Necessary

Evaluate Continuing Calibration Report

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02885.D
Acq On : 1 Mar 2012 14:56
Operator : Syslo
Sample : 1000ppm TPH CCV
Misc : RVB0003 for Surr.
ALS Vial : 1 Sample Multiplier: 1

Quant Time: Mar 01 17:22:25 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Min. RRF : 0.010 Min. Rel. Area : 50% Max. R.T. Dev 0.50min
Max. RRF Dev : 30% Max. Rel. Area : 200%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	d10-Phenanthrene	1.000	1.000	0.0	75	0.00
2	S	d10-Anthracene {S}	1.063	1.089	-2.4	75	0.00
3	S	d14-o-Terphenyl {S}	0.467	0.514	-10.1	77	0.00
4	S	5a-Androstane {S}	0.057	0.069	-21.1	83	0.00
5	I	d30-Tetradecane	1.000	1.000	0.0	70	0.00
6	I	d50-Tetracosane	1.000	1.000	0.0	83	0.00
7	S	d62-Triacontane {S}	0.695	1.097	~~-57.8#~~	109	0.00
8	I	d74-Hexatriacontane	1.000	1.000	0.0	170	0.00

07.4% Lin Reg.

(#) = Out of Range SPCC's out = 0 CCC's out = 0

DROTPH051611.M Thu Jan 10 15:31:31 2013 SLICK2

For Surr

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02885.D
Acq On : 1 Mar 2012 14:56
Operator : Syslo
Sample : 1000ppm TPH CCV
Misc : RVB0003 for Surr.
ALS Vial : 1 Sample Multiplier: 1

Quant Time: Mar 01 17:22:25 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	358723	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	109515	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	150757	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.605	66	134865	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.822	188	390516	10.24	ug/mL	0.00
Spiked Amount 20.000			Recovery =	51.20%		
3) d14-o-Terphenyl {S}	19.733	244	184507	11.01	ug/mL	0.00
Spiked Amount 20.000			Recovery =	55.05%		
4) 5a-Androstane {S}	21.141	260	24901	12.21	ug/mL	0.00
Spiked Amount 20.000			Recovery =	61.05%		
7) d62-Triacontane {S}	28.157	66	165308	12.74	ug/mL	0.00 *
Spiked Amount 20.000			Recovery =	63.70%		

Target Compounds Qvalue

#) = qualifier out of range (m) = manual integration (+) = signals summed

For Surr & IS

* %D = 27.4 by Lin reg.

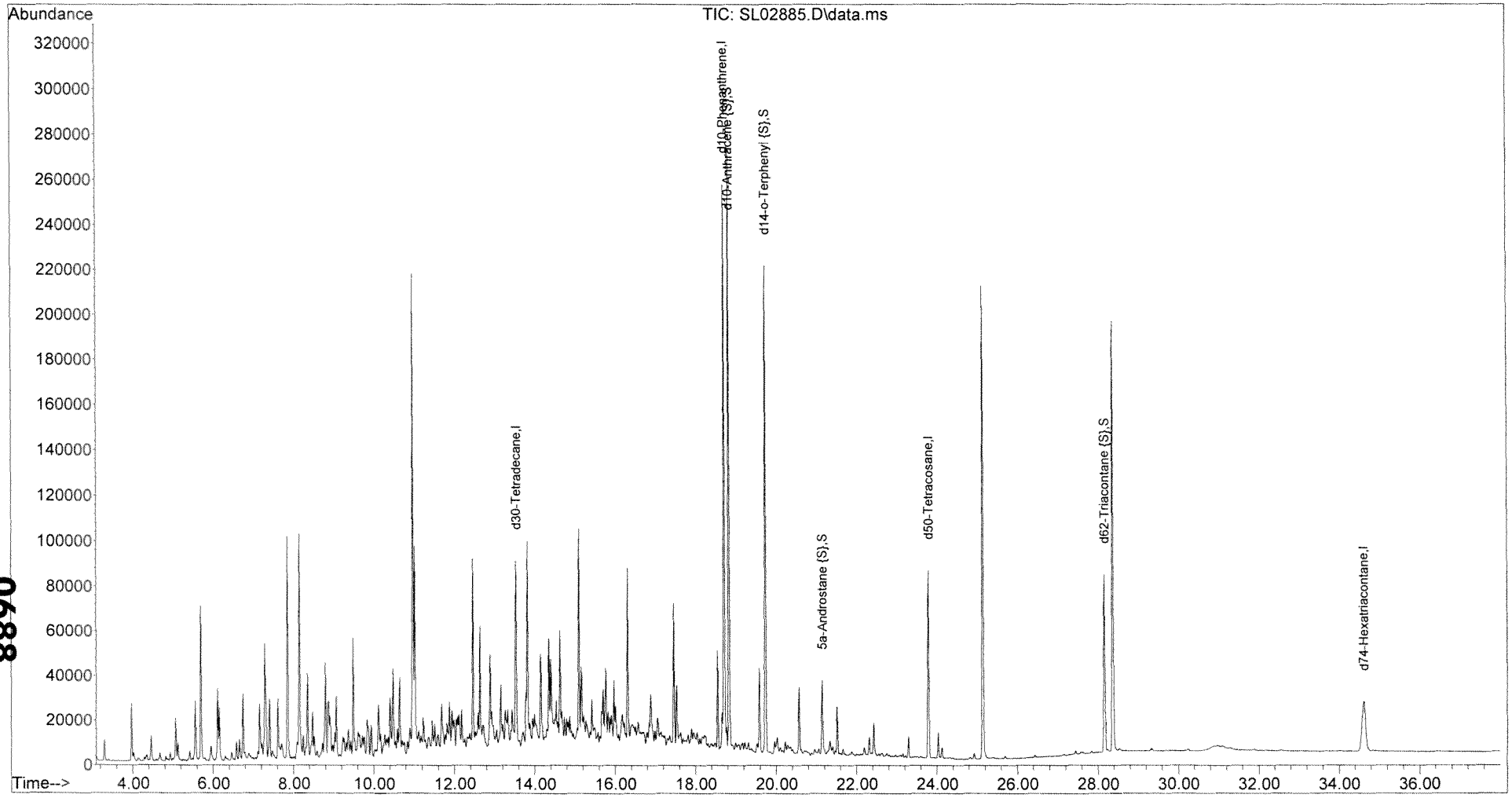
SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02885.D
Acq On : 1 Mar 2012 14:56
Operator : Syslo
Sample : 1000ppm TPH CCV
Misc : RVB0003 for Surr.
ALS Vial : 1 Sample Multiplier: 1

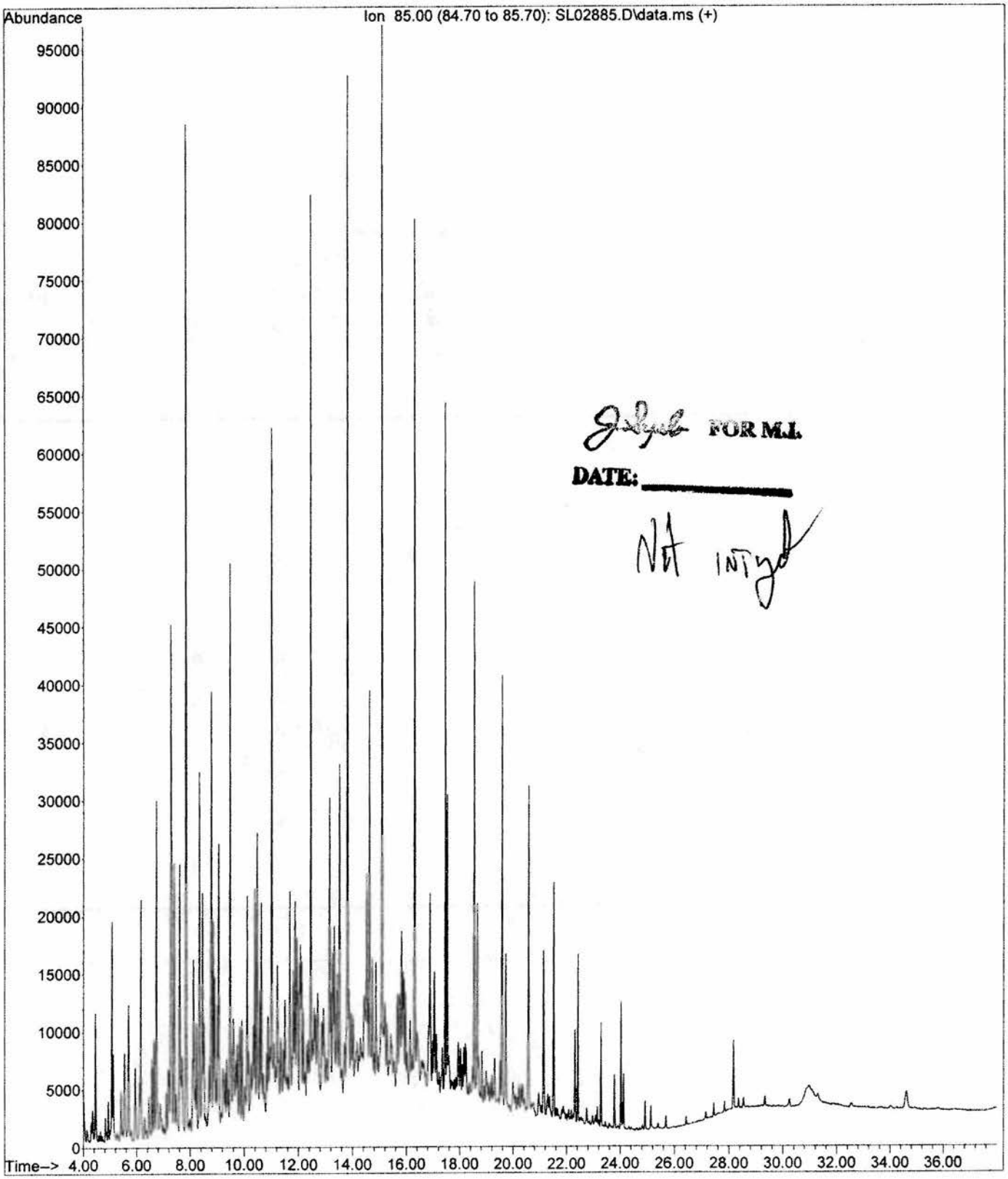
Quant Time: Mar 01 17:22:25 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Mon Feb 27 17:34:25 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3



0688

File :C:\msdchem\1\DATA\030112\SL02885.D
Operator : Syslo
Acquired : 1 Mar 2012 14:56 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: 1000ppm TPH CCV
Misc Info : RVB0003 for Surr.
Vial Number: 1



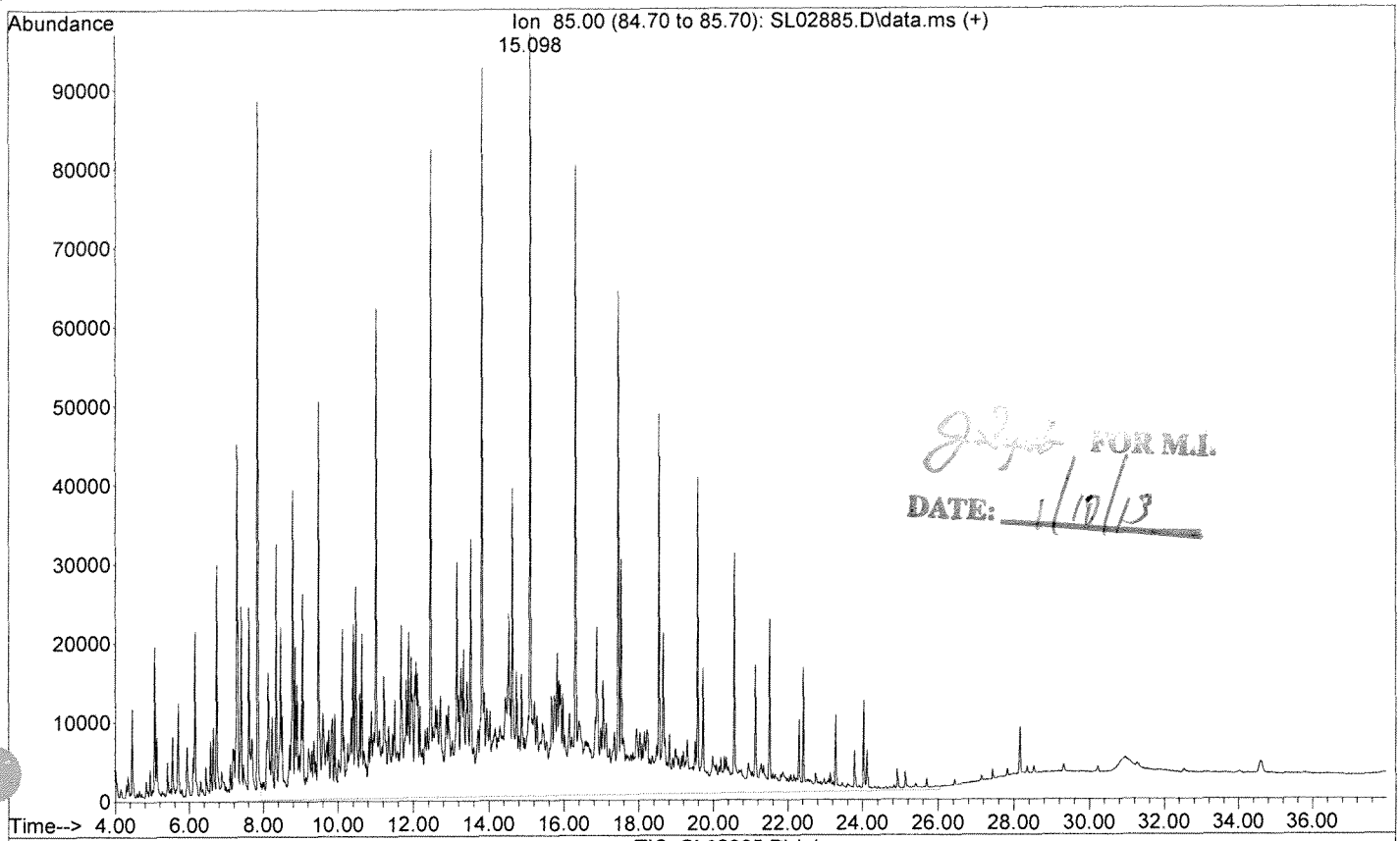
FOR M.I.

DATE: _____

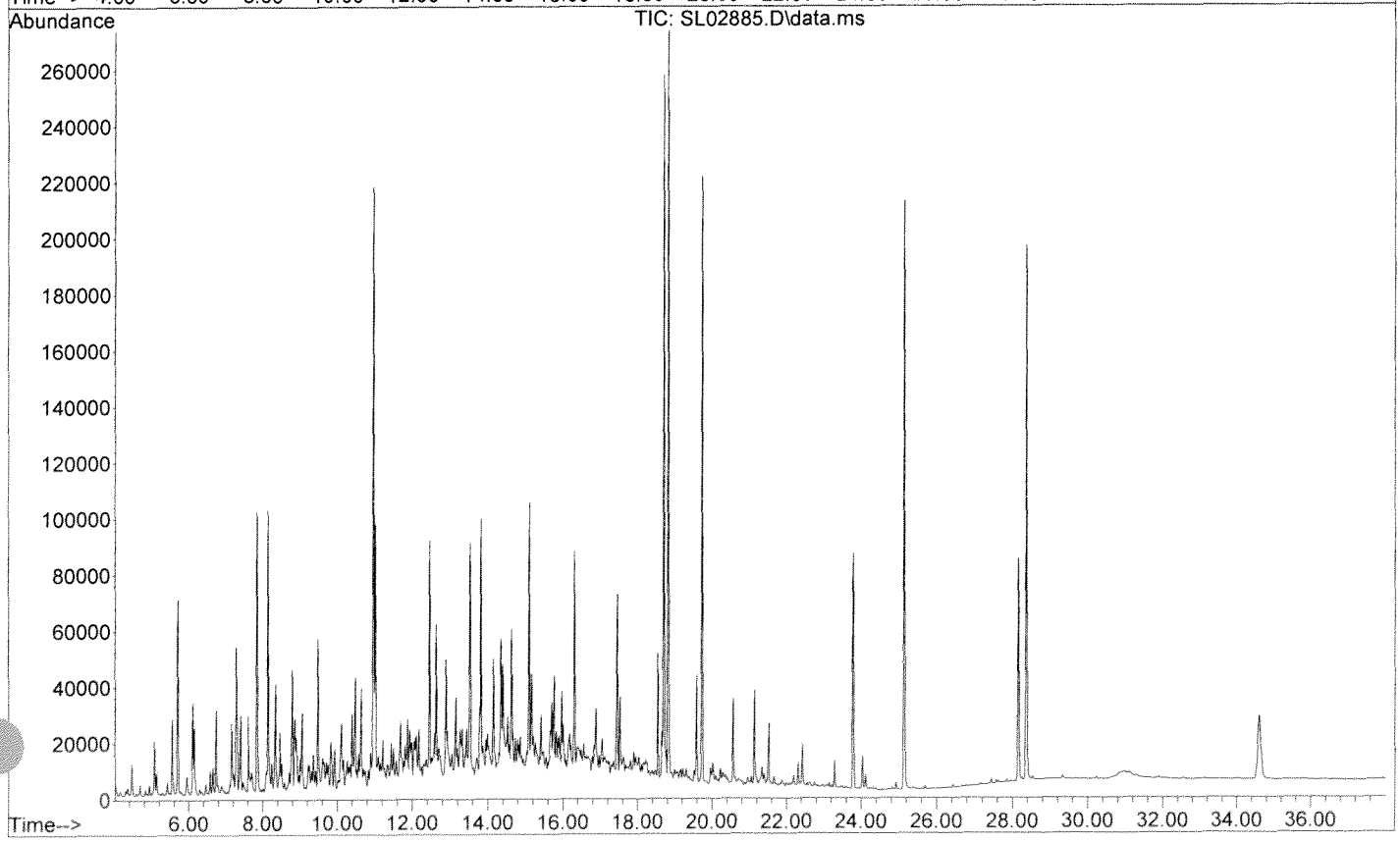
Nat INT'l

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02885.D
Operator : Syslo
Acquired : 1 Mar 2012 14:56 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: 1000ppm TPH CCV
Misc Info : RVB0003 for Surr.
Vial Number: 1



FOR M.I.
DATE: 1/10/13



Area Percent Report

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02885.D
Acq On : 1 Mar 2012 14:56
Operator : Syslo
mple : 1000ppm TPH CCV
sc : RVB0003 for Surr.
ALS Vial : 1 Sample Multiplier: 1

Integration Parameters: Oilint.P
Integrator: RTE
Smoothing : ON Filtering: 5
Sampling : 1 Min Area: 0.5 % of largest Peak
Start Thrs: 0.05 Max Peaks: 5
Stop Thrs : 0.05 Peak Location: TOP

If leading or trailing edge < 50 prefer < Tangent else baseline drop >
Peak separation: 3

Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Title : DRO/TPH ICAL + Surr. 05/16/11

Signal : EIC Ion 85.00 (84.70 to 85.70): SL02885.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	15.098	606	1504	2776	rM 9	96504	7041868	100.00%	100.000%

Sum of corrected areas: 7041868

DROTPH051611.M Thu Jan 10 15:39:59 2013 SLICK2

FOR M.L.
DATE: 1/10/13

BLK030112

VANTE Dilution

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02886.D
Acq On : 1 Mar 2012 15:41
Operator : Syslo
Sample : BLK030112
Misc : Waste Dilution Blank 3/1/2012
ALS Vial : 2 Sample Multiplier: 1

Quant Time: Mar 01 17:24:36 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	244273	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	73201	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	95670	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.596	66	87506	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.821	188	462345	17.80	ug/mL	0.00
Spiked Amount 20.000			Recovery =	89.00%		
3) d14-o-Terphenyl {S}	19.733	244	219441	19.22	ug/mL	0.00
Spiked Amount 20.000			Recovery =	96.10%		
4) 5a-Androstane {S}	21.140	260	29588	21.30	ug/mL	0.00
Spiked Amount 20.000			Recovery =	106.50%		
7) d62-Triacontane {S}	28.157	66	193321	22.74	ug/mL	0.00
Spiked Amount 20.000			Recovery =	113.70%		
Target Compounds						Qvalue

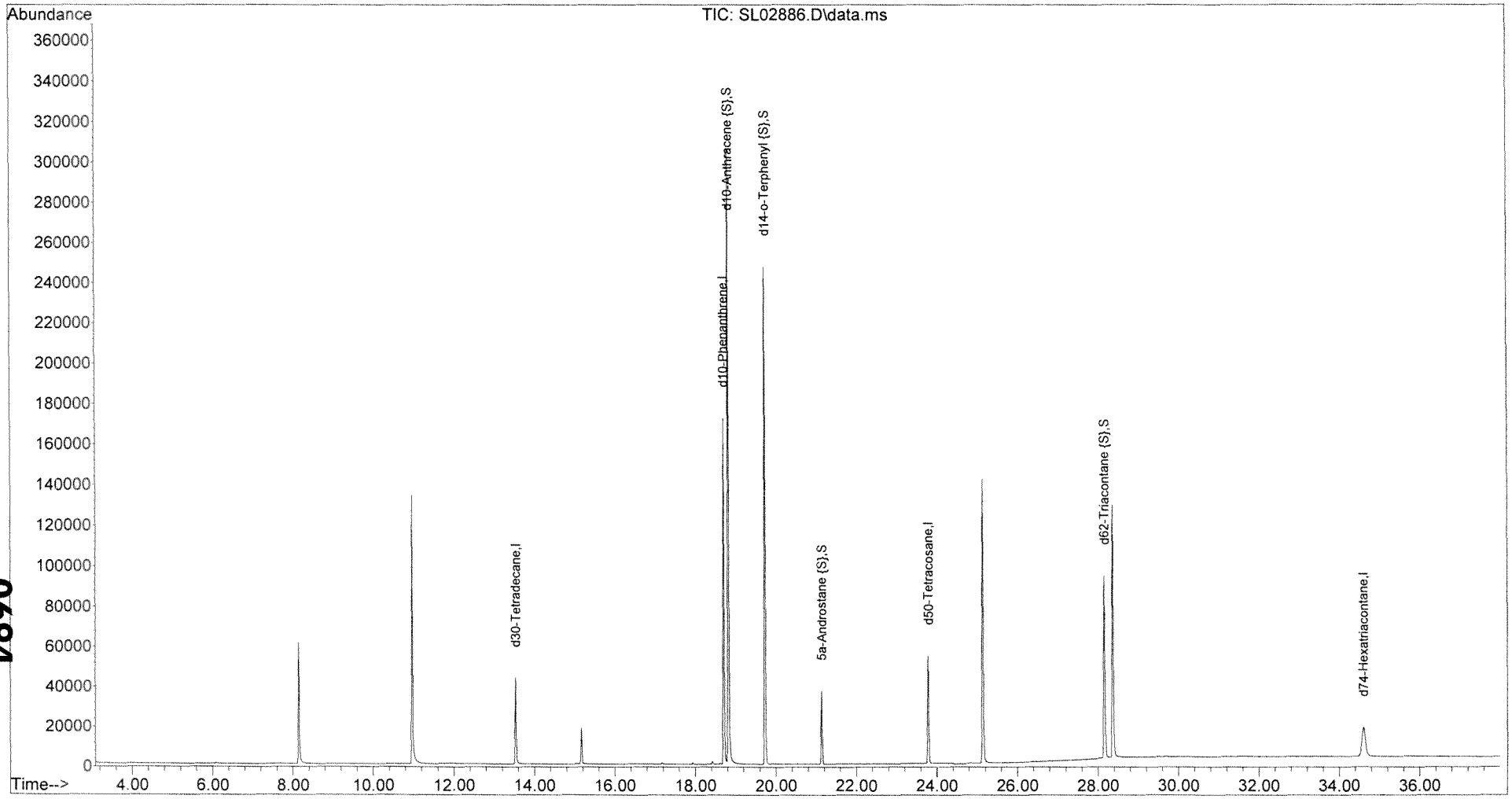
#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02886.D
Acq On : 1 Mar 2012 15:41
Operator : Syslo
Sample : BLK030112
Misc : Waste Dilution Blank 3/1/2012
ALS Vial : 2 Sample Multiplier: 1

Quant Time: Mar 01 17:24:36 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration



0694

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

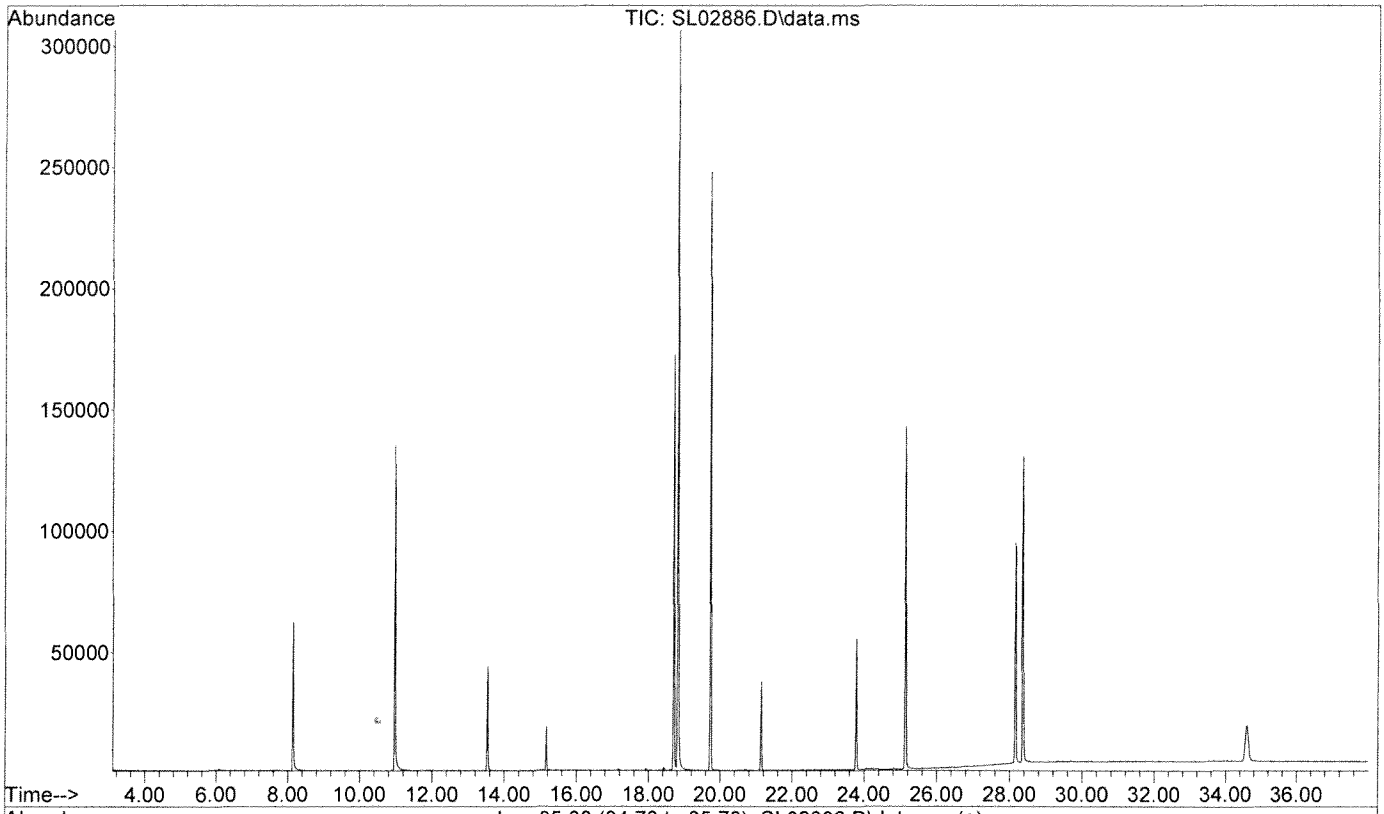
name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

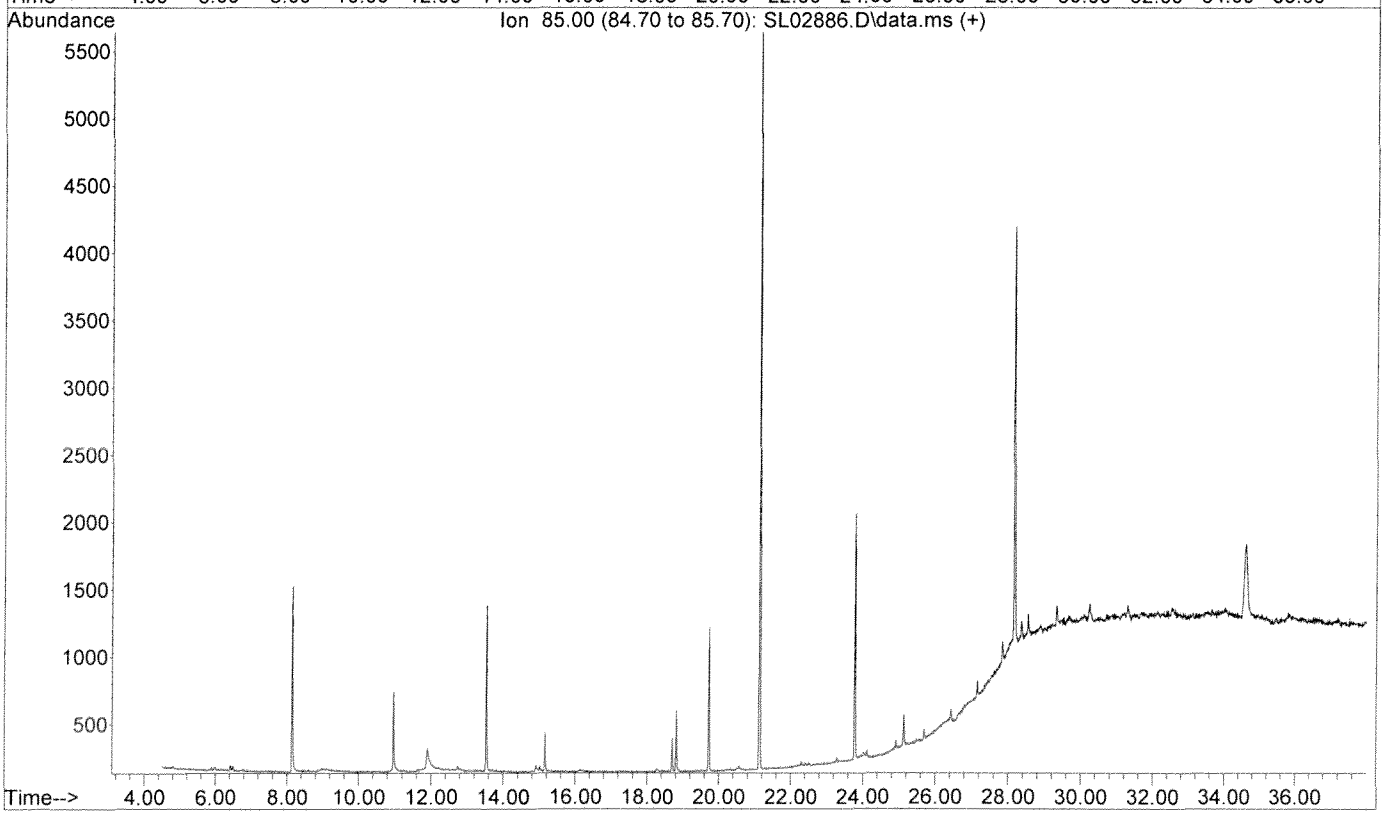
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



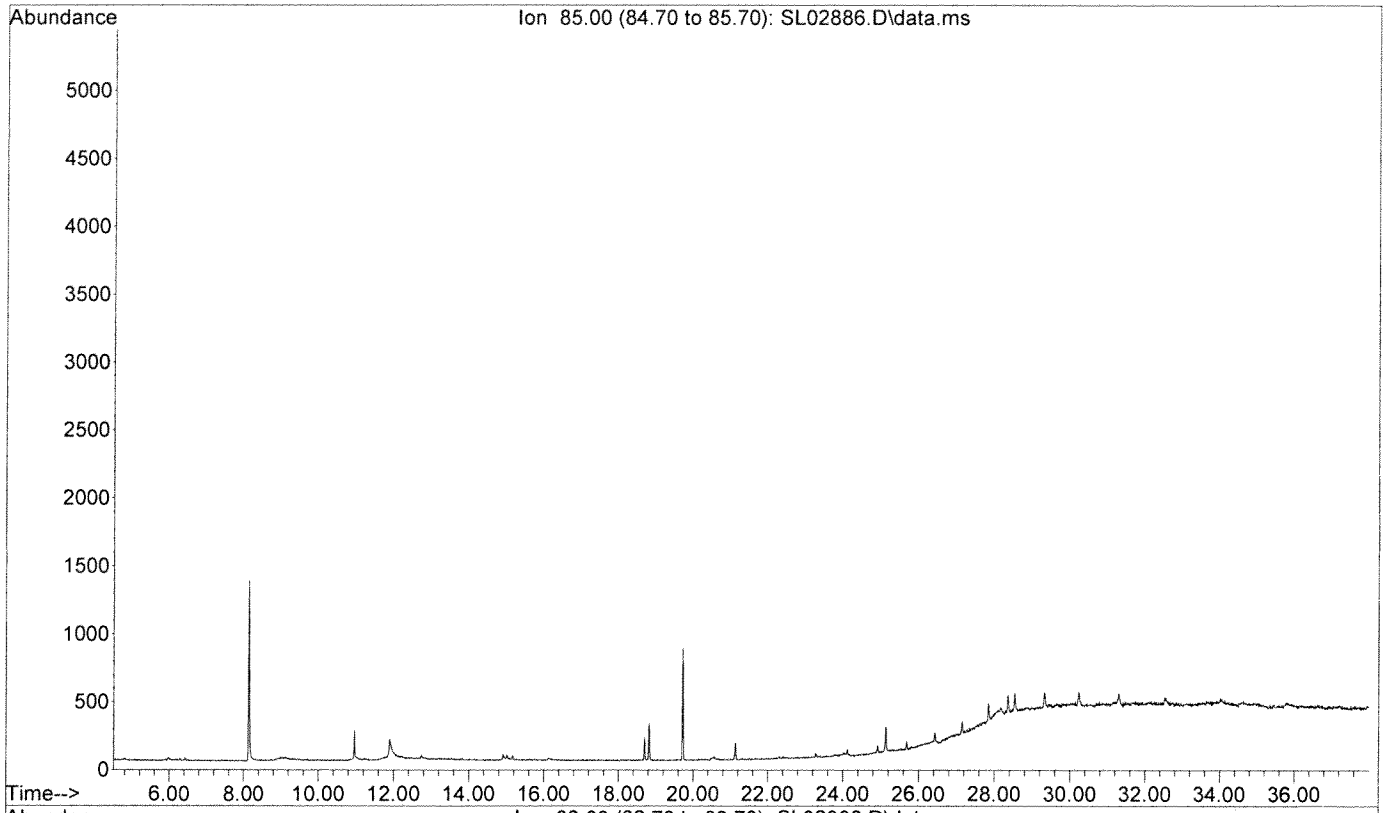


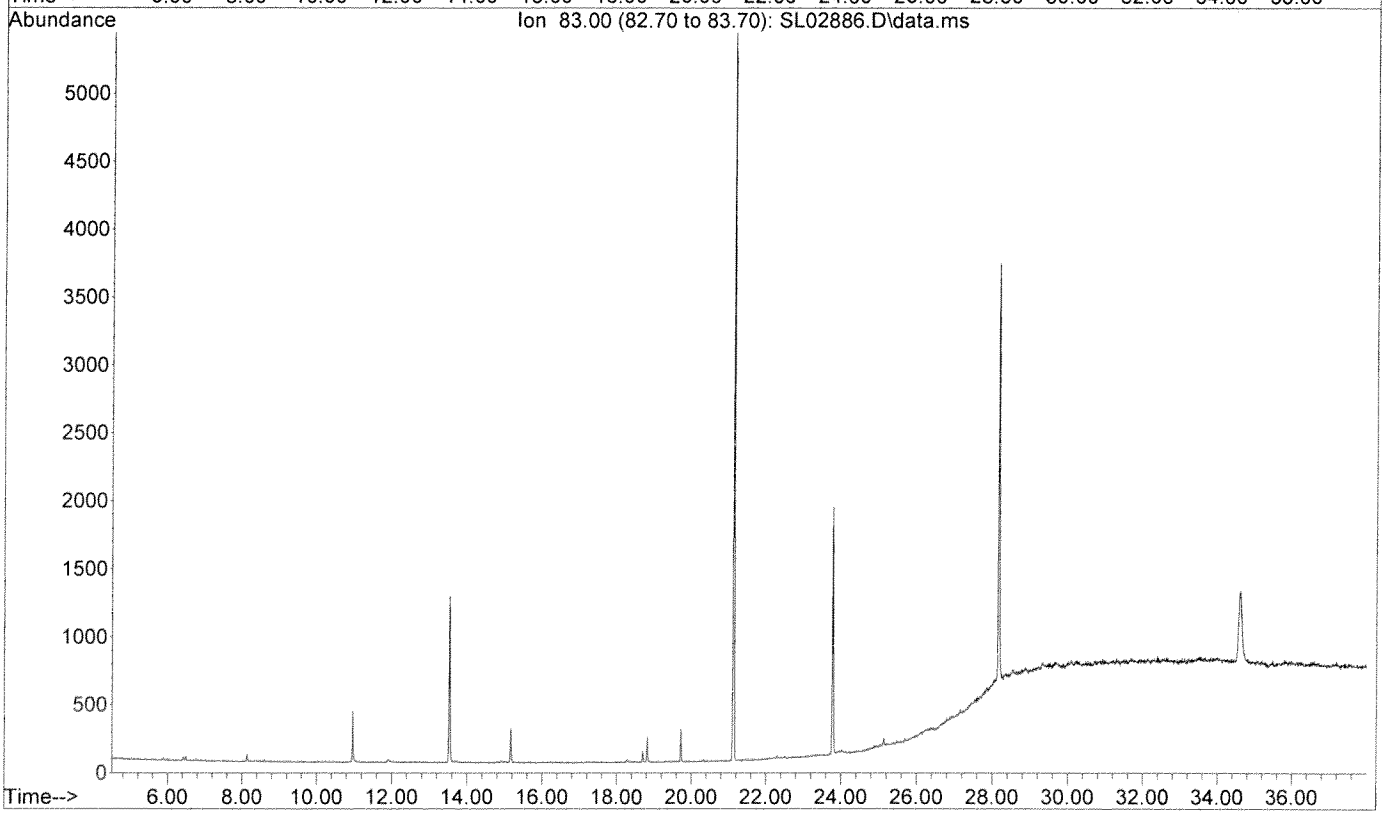
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2





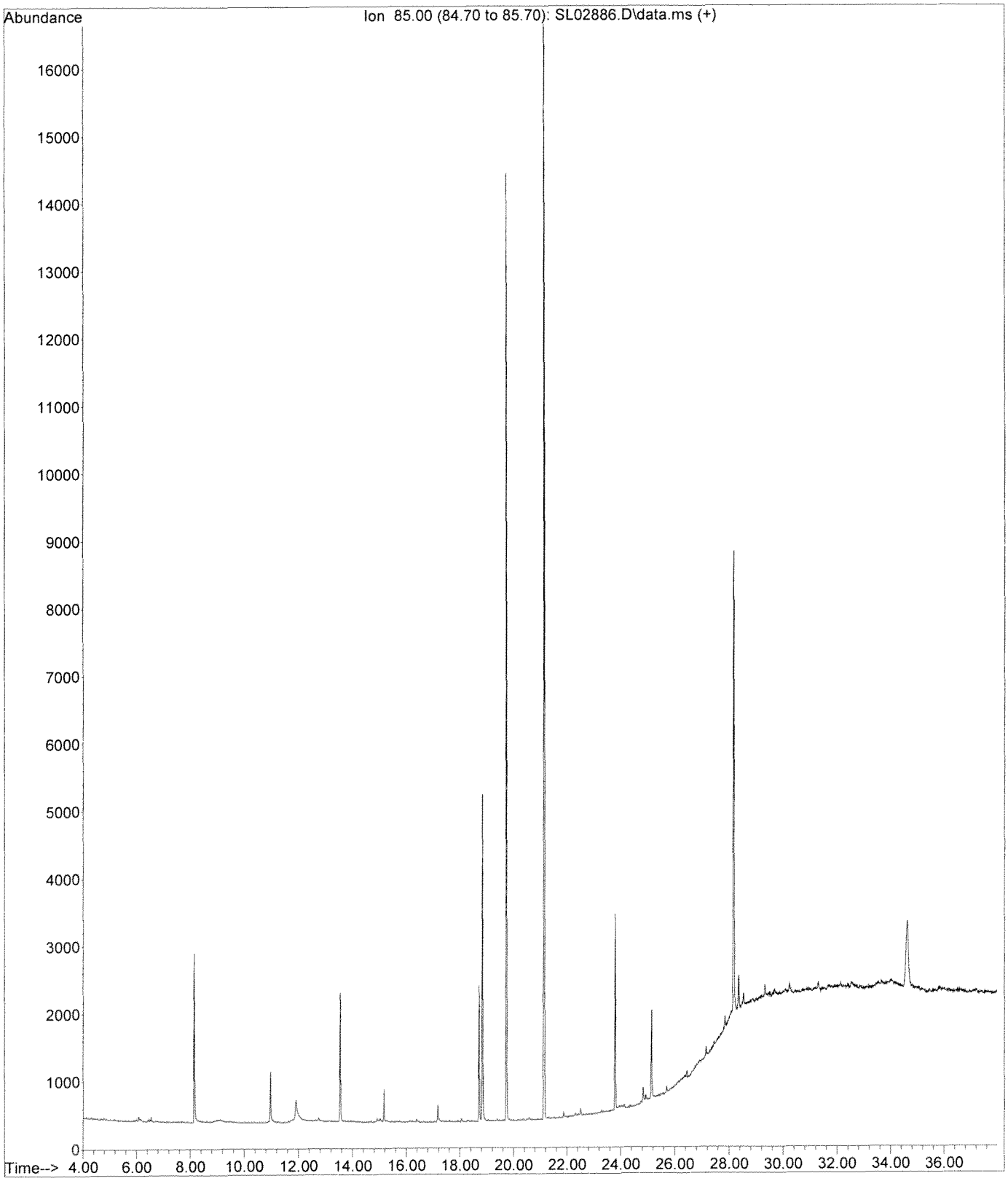
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



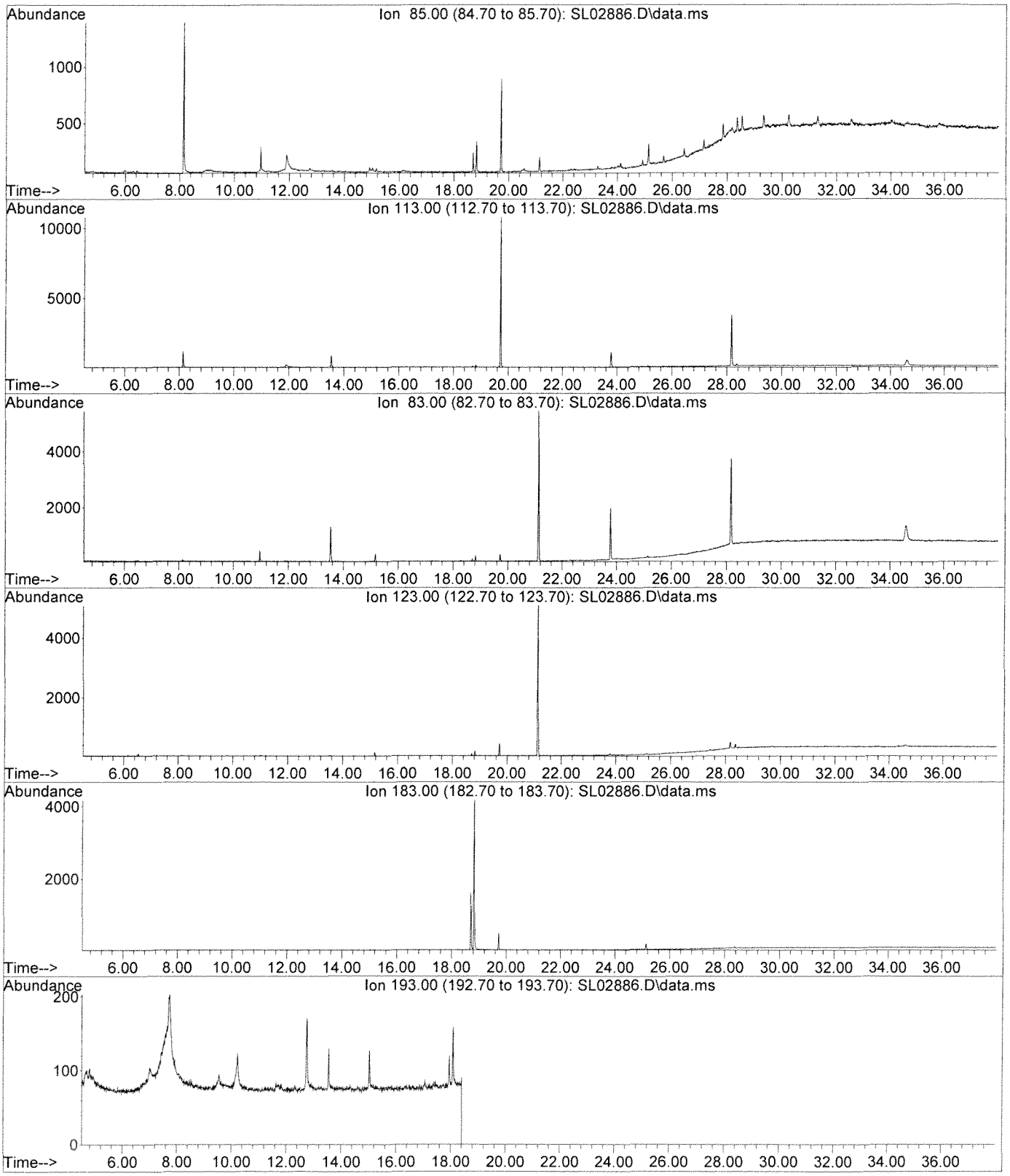
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



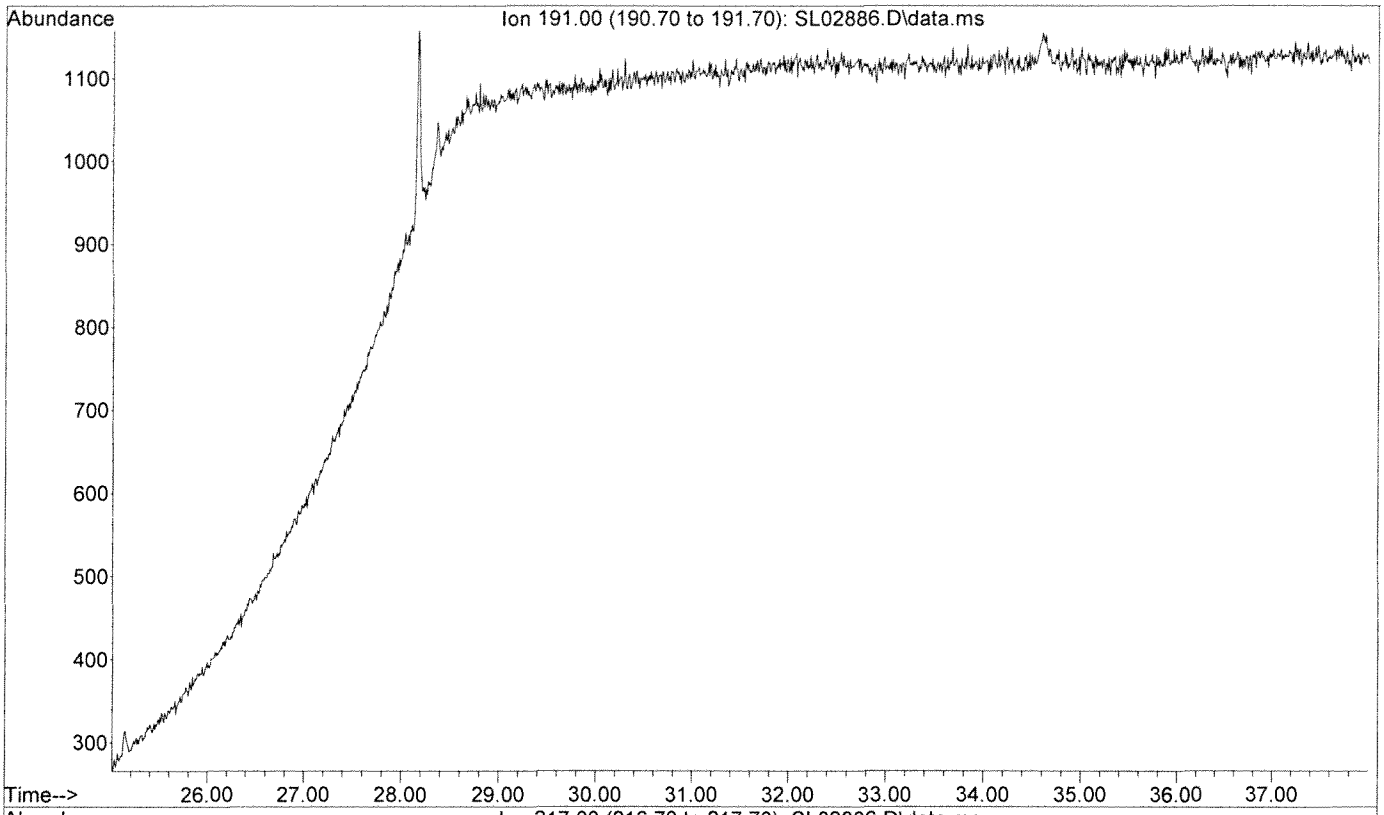
US EPA ARCHIVE DOCUMENT

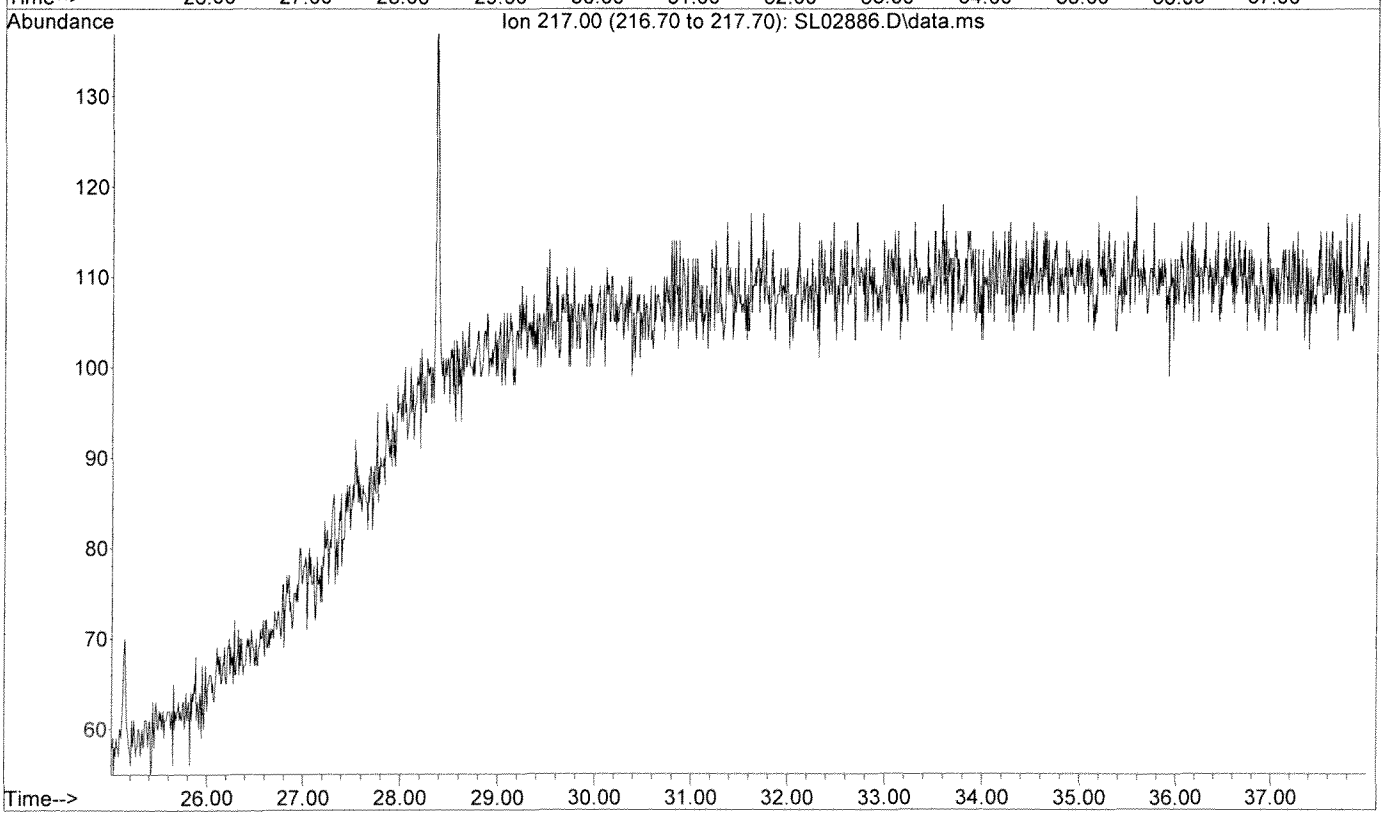
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02886.D
Operator    : Syslo
Acquired    :  1 Mar 2012  15:41      using AcqMethod DROTPHX51611.M
Instrument  :   Slick2
Sample Name: BLK030112
Misc Info   : Waste Dilution Blank 3/1/2012
Vial Number: 2
```





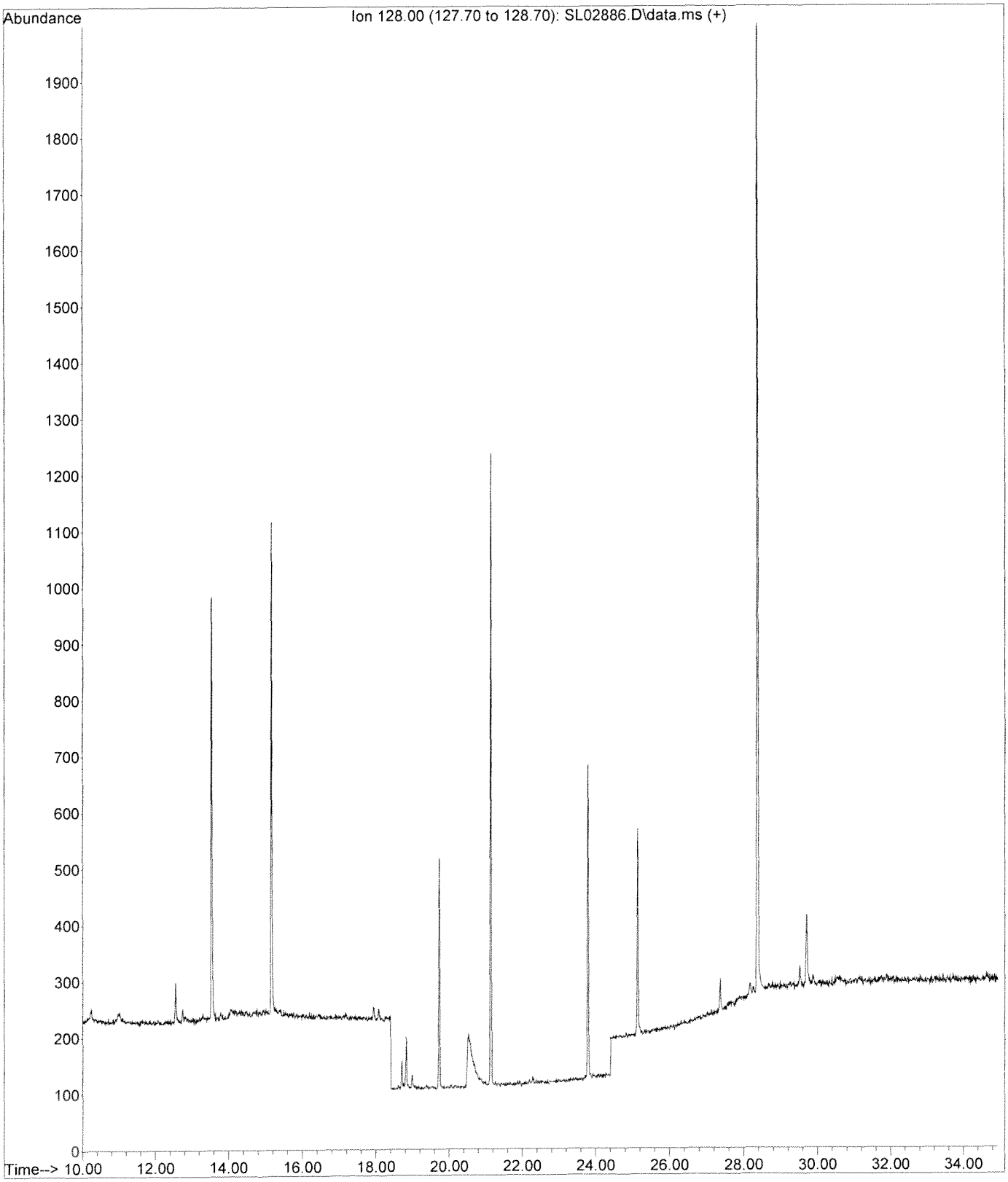
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



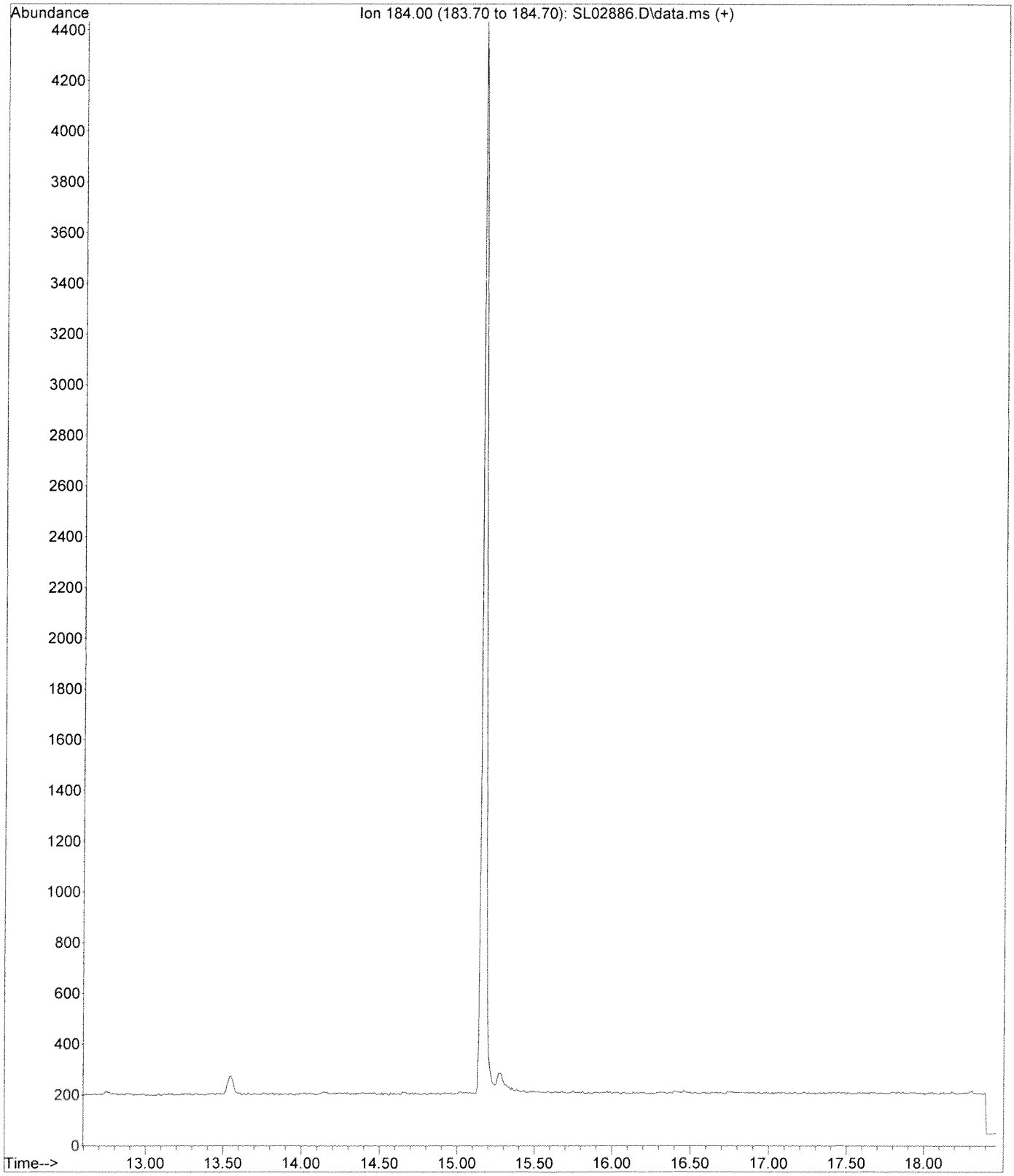
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

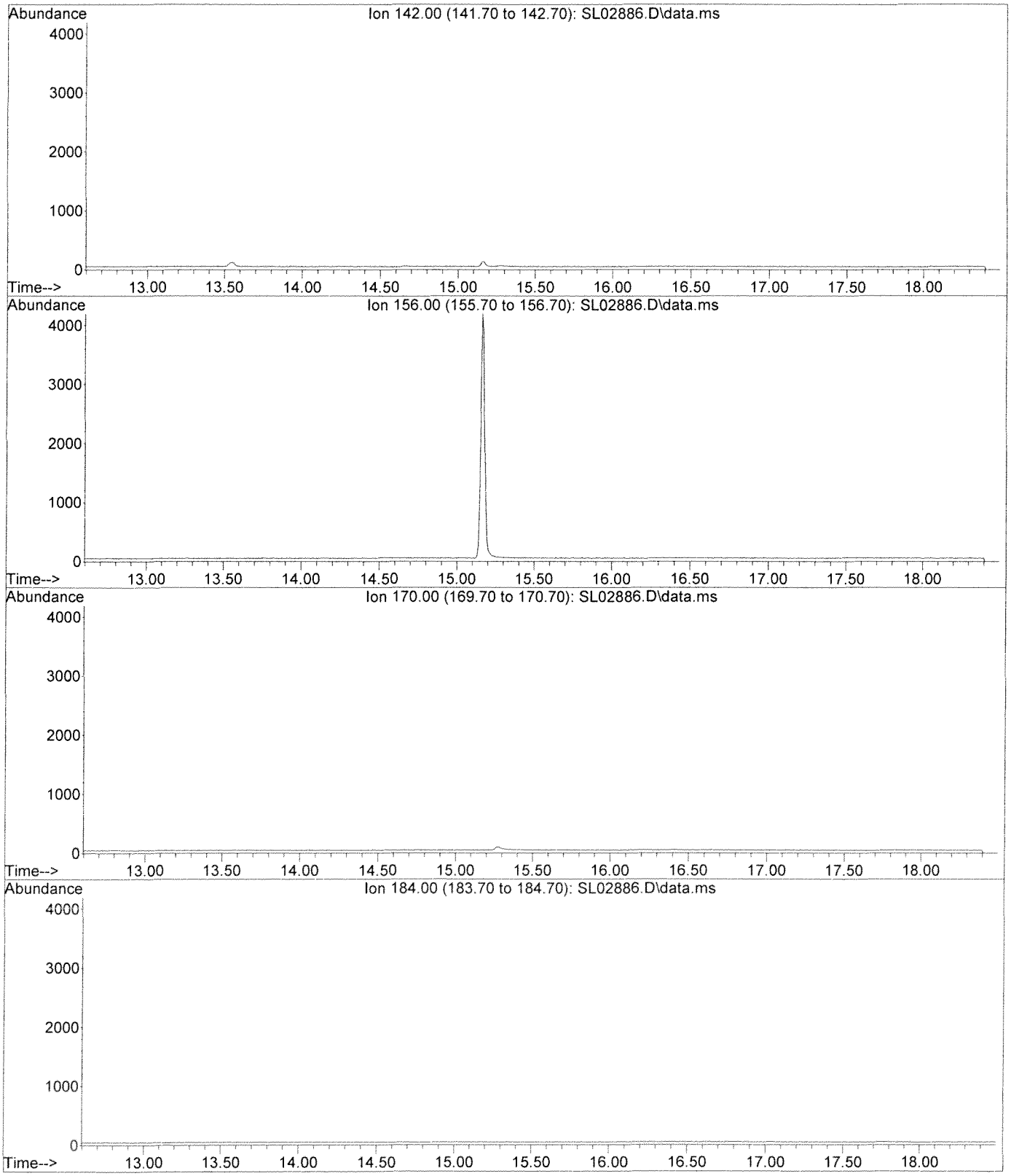
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



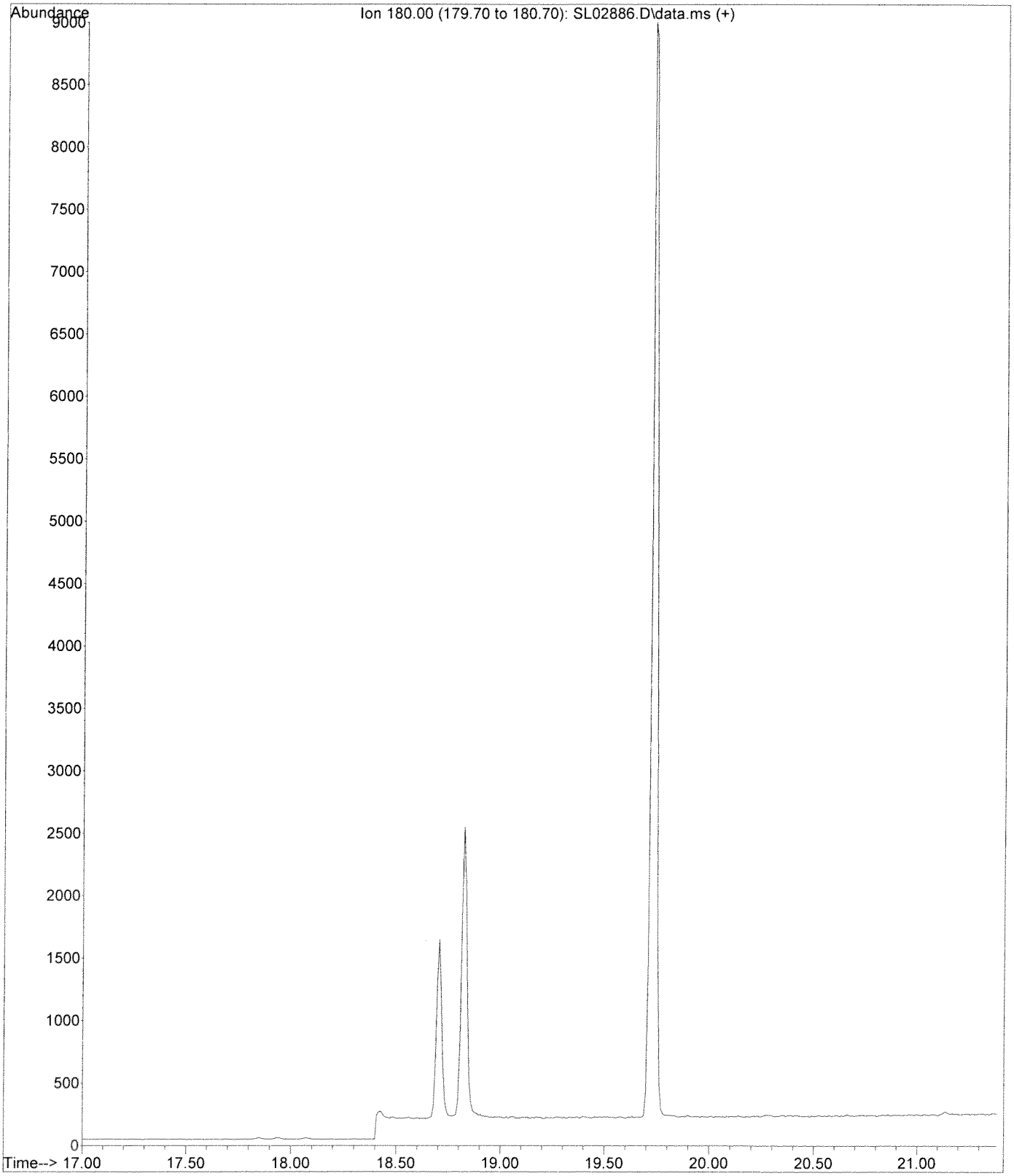
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



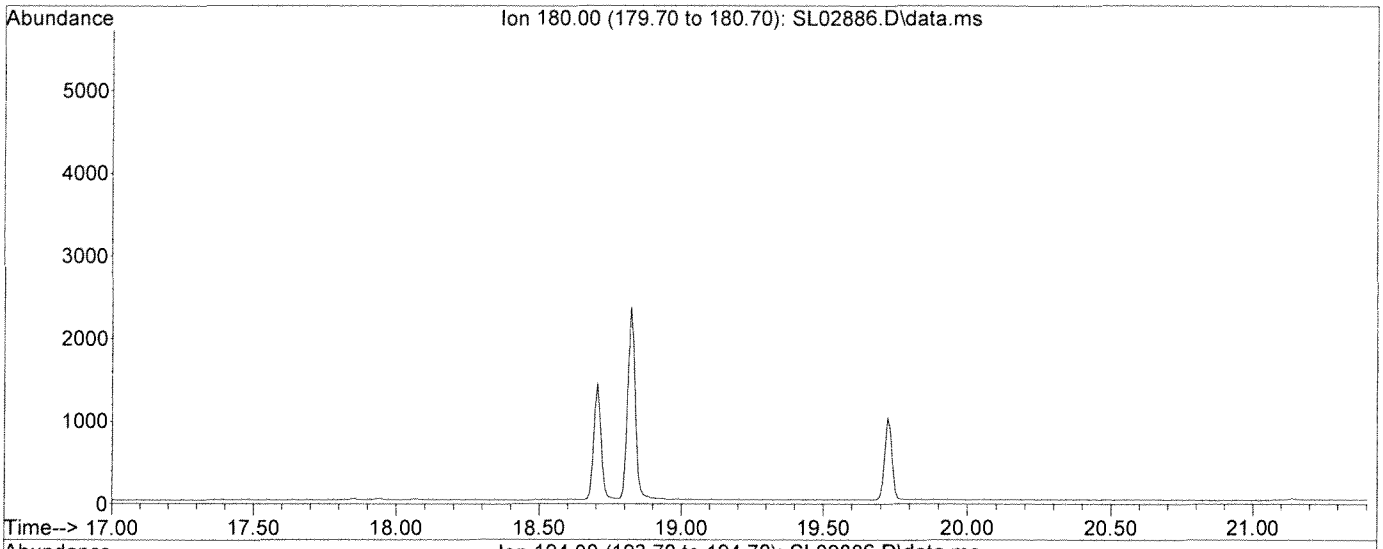
US EPA ARCHIVE DOCUMENT

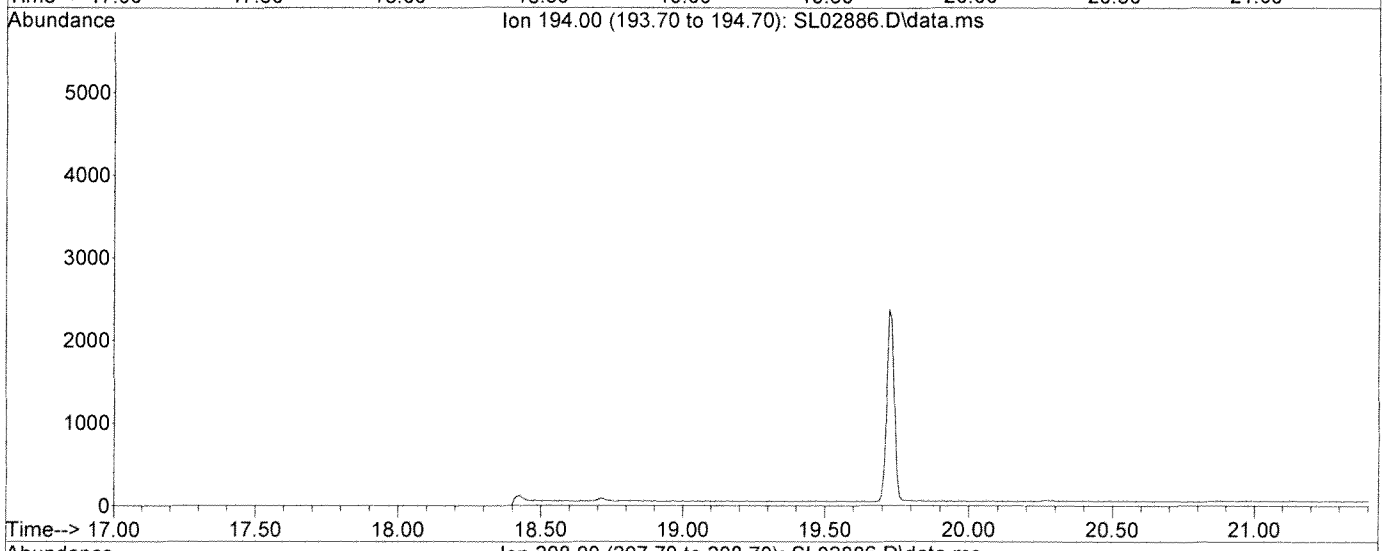
File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2

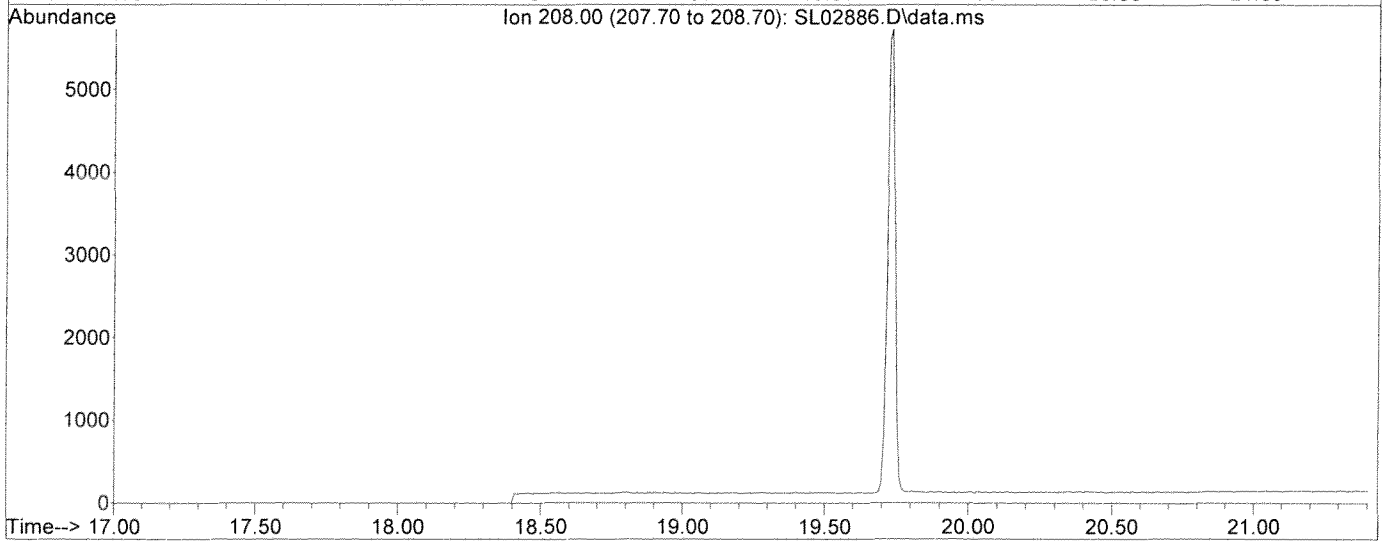


US EPA ARCHIVE DOCUMENT

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File        :C:\msdchem\1\DATA\030112\SL02886.D
Operator    : Syslo
Acquired    :  1 Mar 2012  15:41      using AcqMethod DROTPHX51611.M
Instrument  :   Slick2
Sample Name: BLK030112
Misc Info   : Waste Dilution Blank 3/1/2012
Vial Number: 2
```

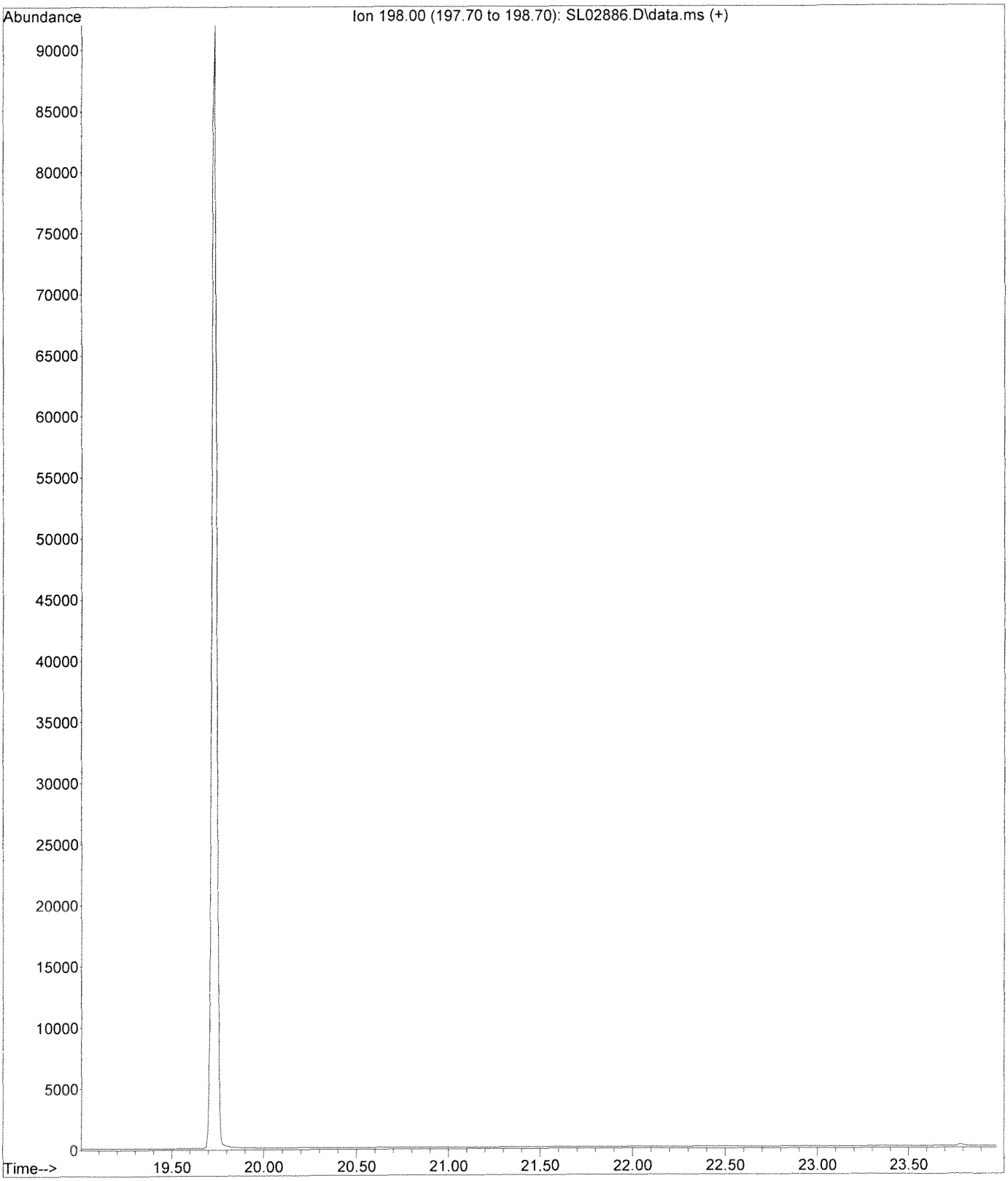






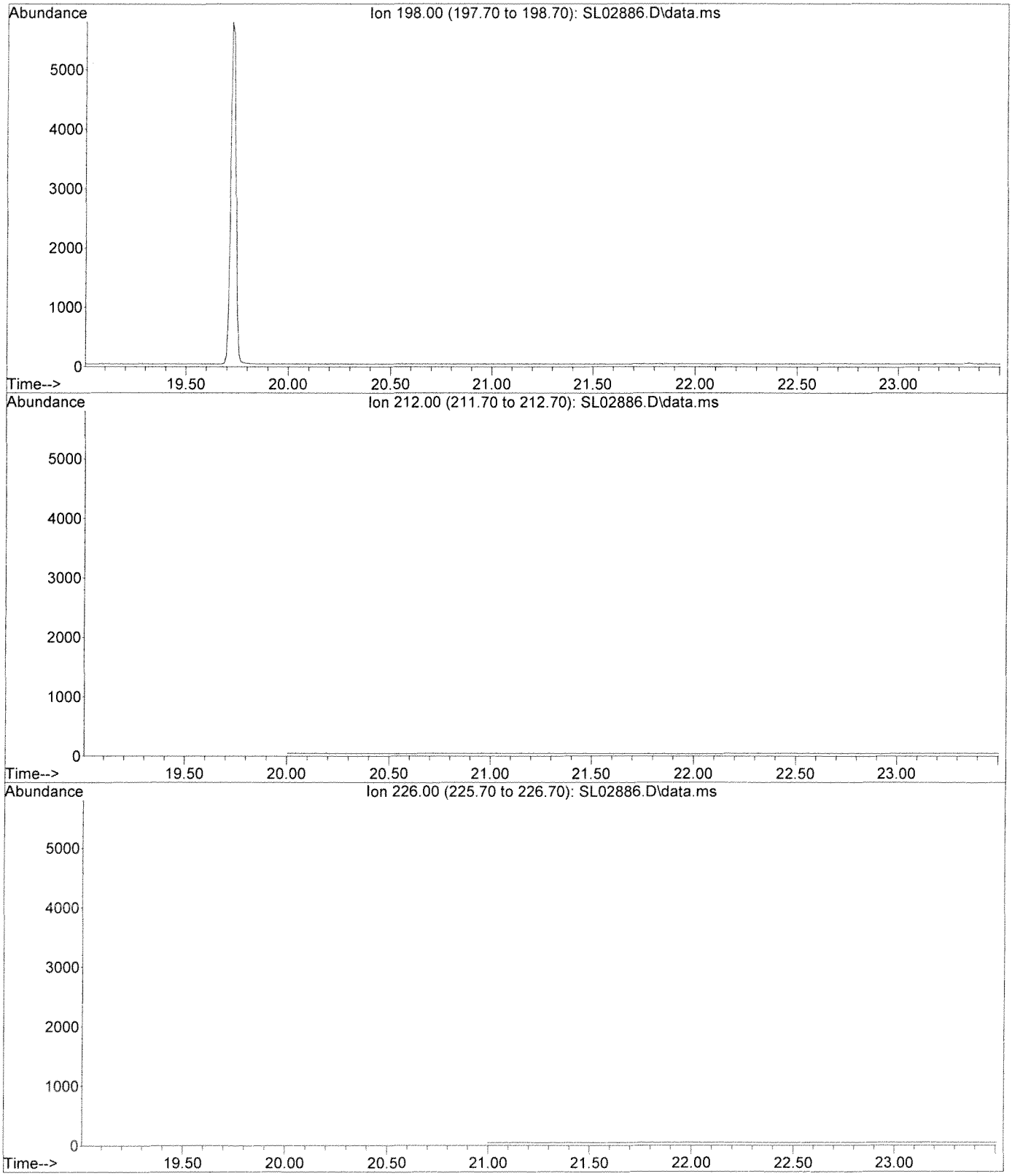
US EPA ARCHIVE DOCUMENT

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File        :C:\msdchem\1\DATA\030112\SL02886.D
Operator    : Syslo
Acquired    :  1 Mar 2012  15:41      using AcqMethod DROTPHX51611.M
Instrument  :   Slick2
Sample Name: BLK030112
Misc Info   : Waste Dilution Blank 3/1/2012
Vial Number: 2
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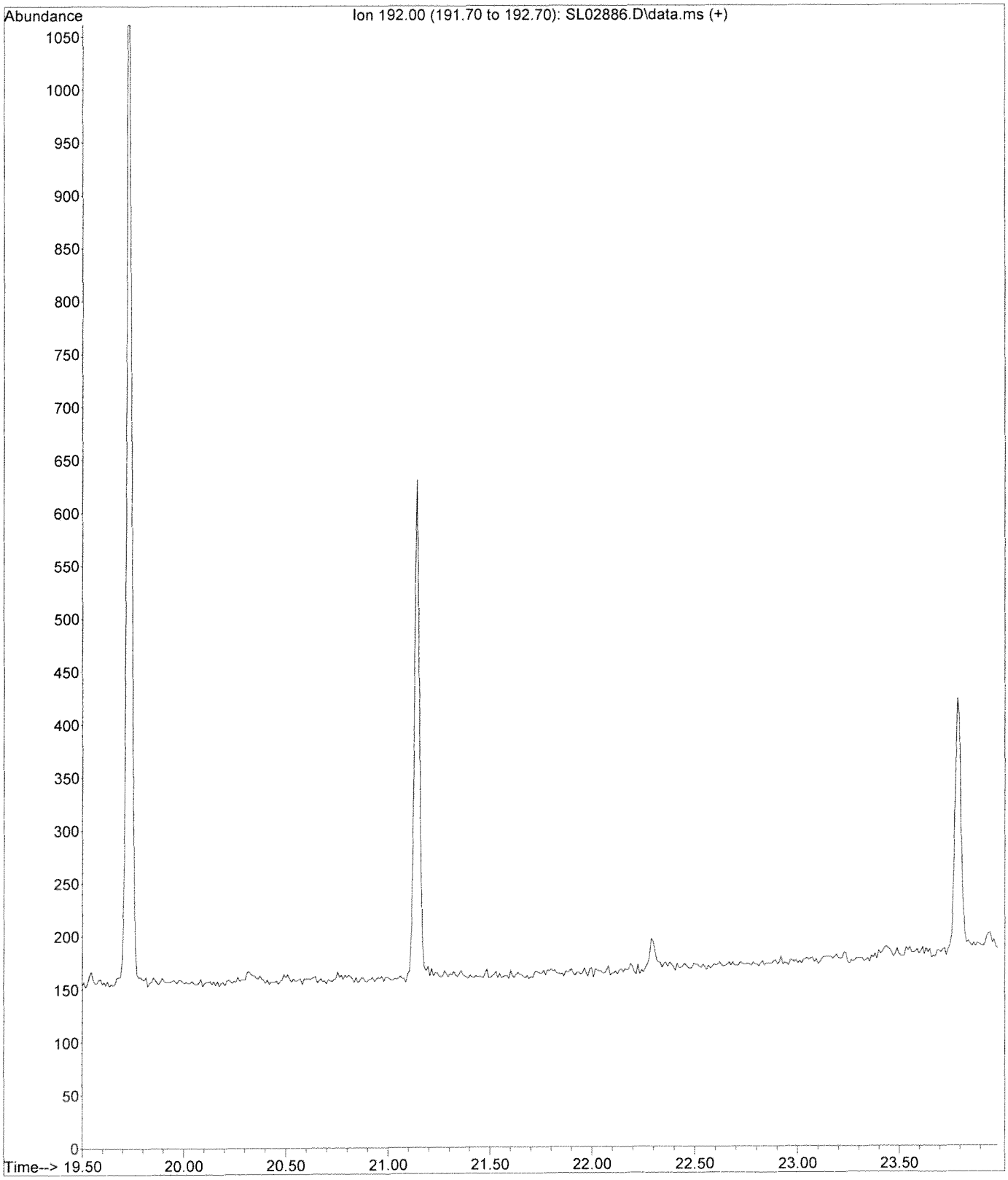
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File        :C:\msdchem\1\DATA\030112\SL02886.D
Operator    : Syslo
Acquired    :  1 Mar 2012  15:41      using AcqMethod DROTPHX51611.M
Instrument  :   Slick2
Sample Name: BLK030112
Misc Info   : Waste Dilution Blank 3/1/2012
Vial Number: 2
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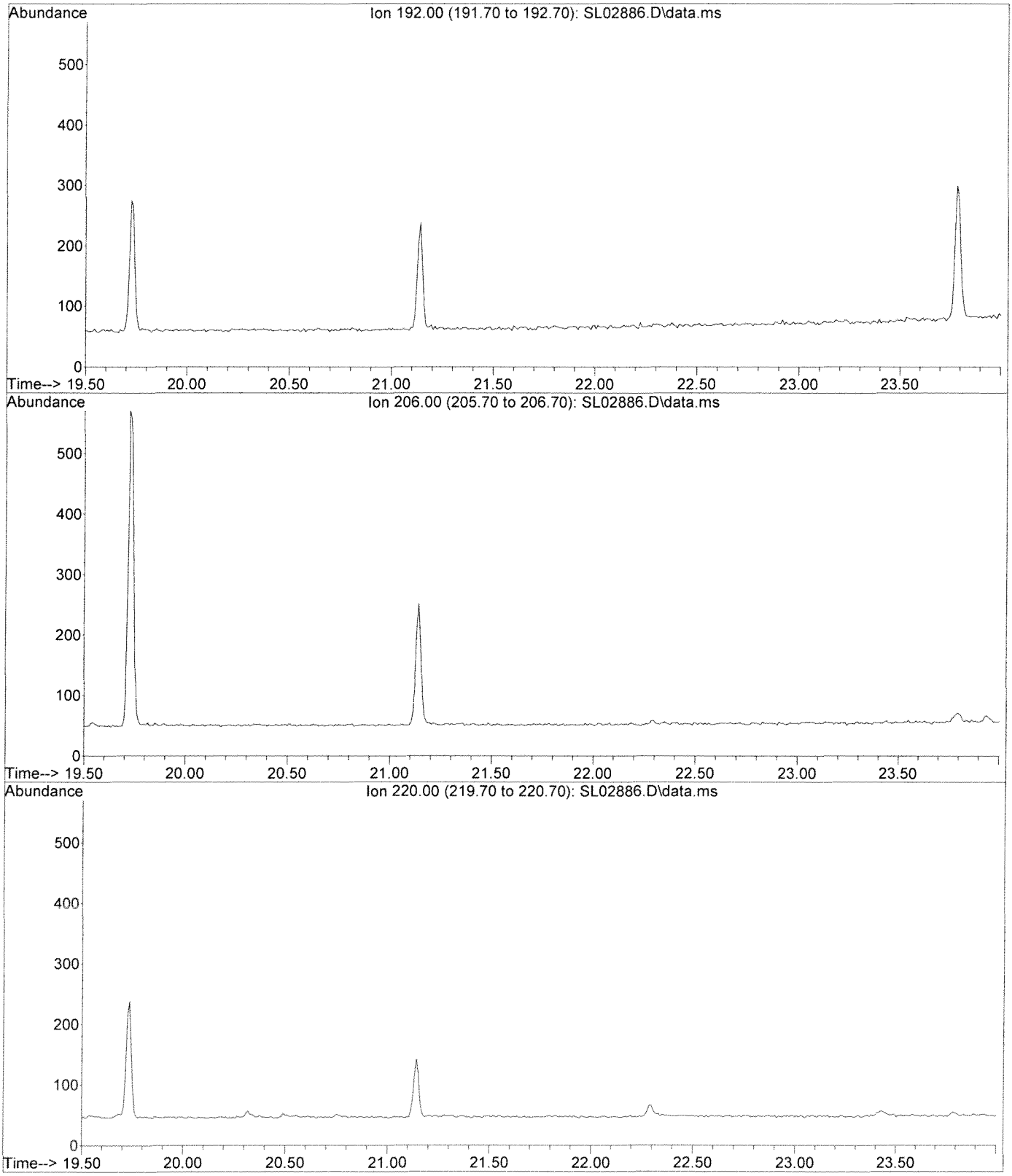
US EPA ARCHIVE DOCUMENT

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Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
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Misc Info : Waste Dilution Blank 3/1/2012
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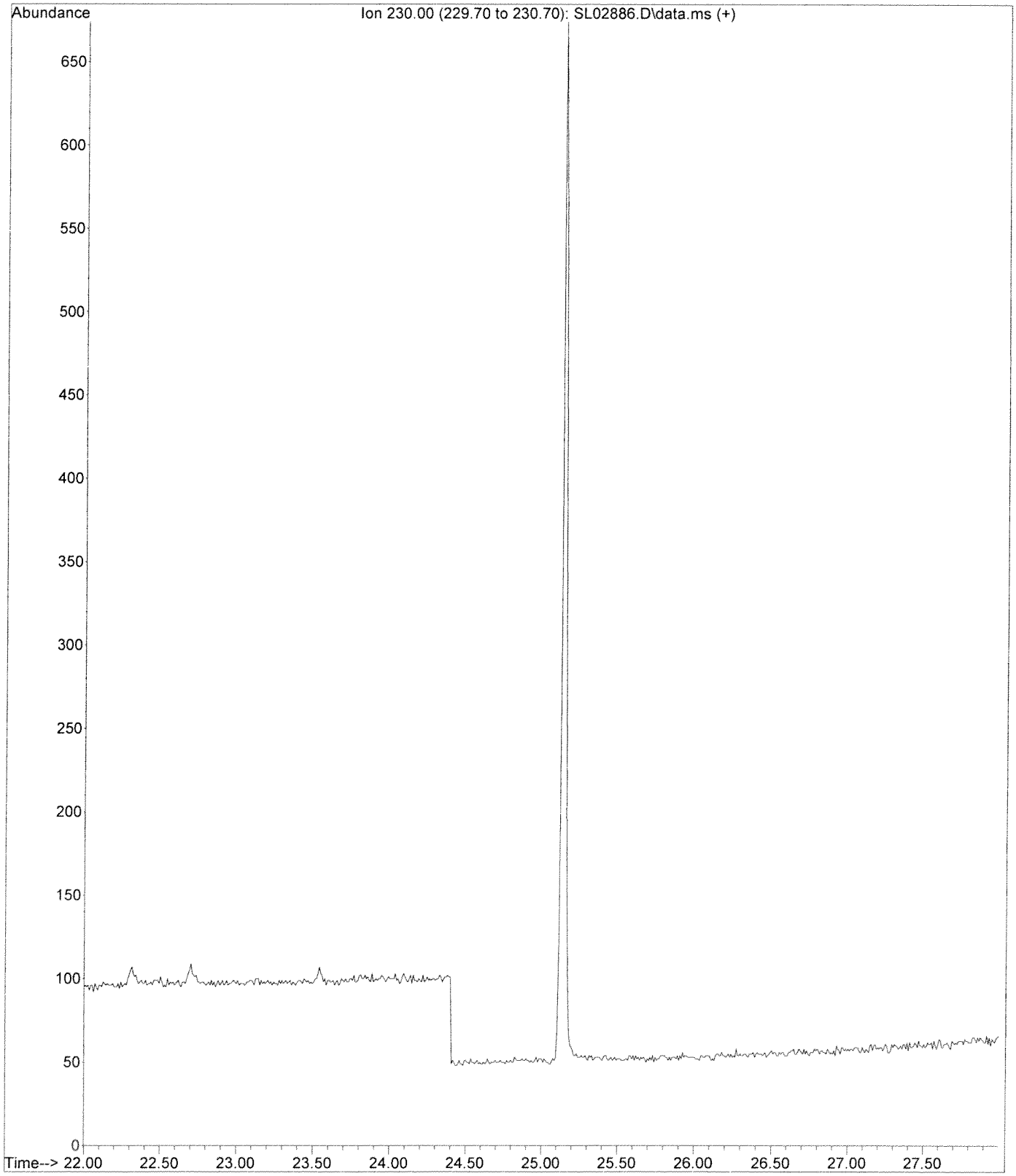
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



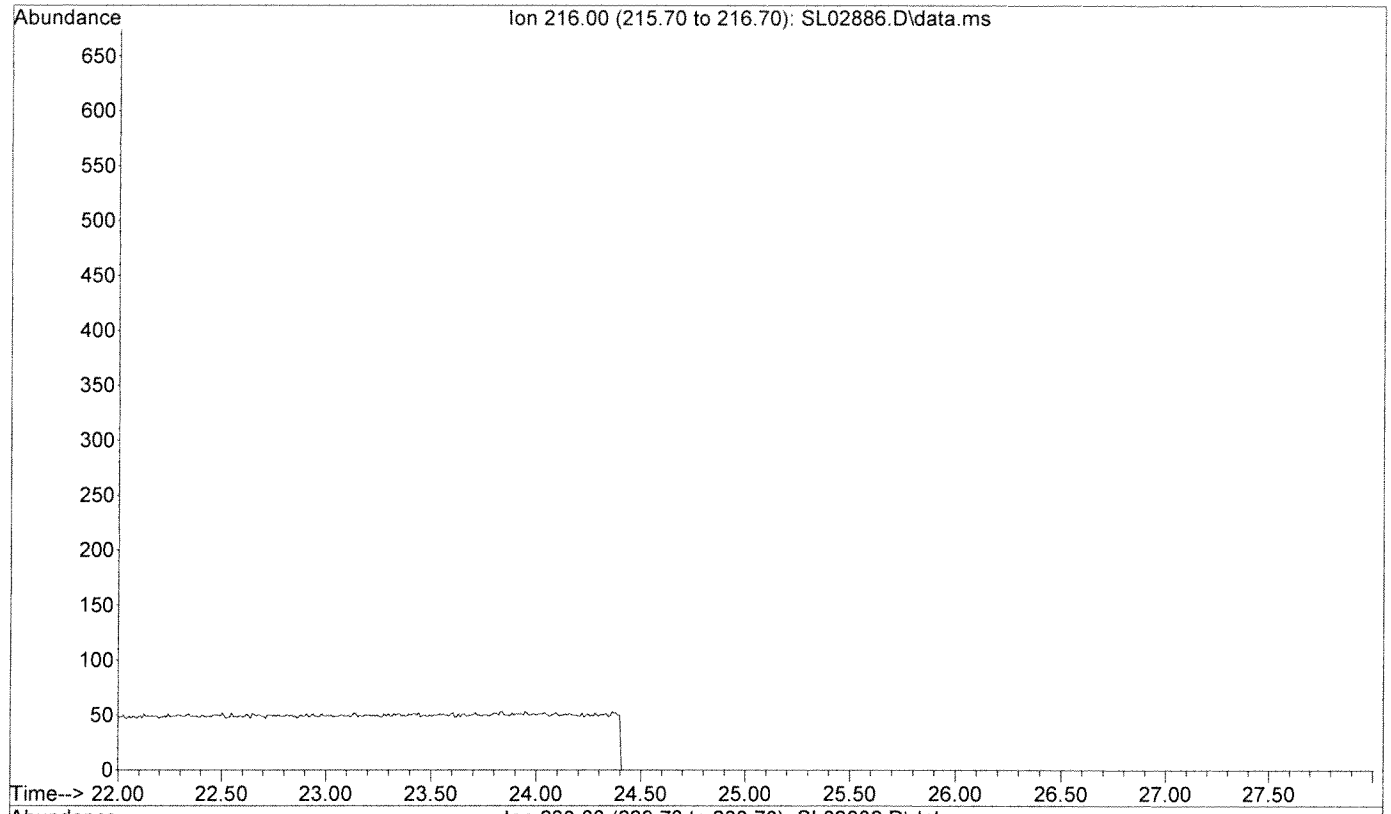
US EPA ARCHIVE DOCUMENT

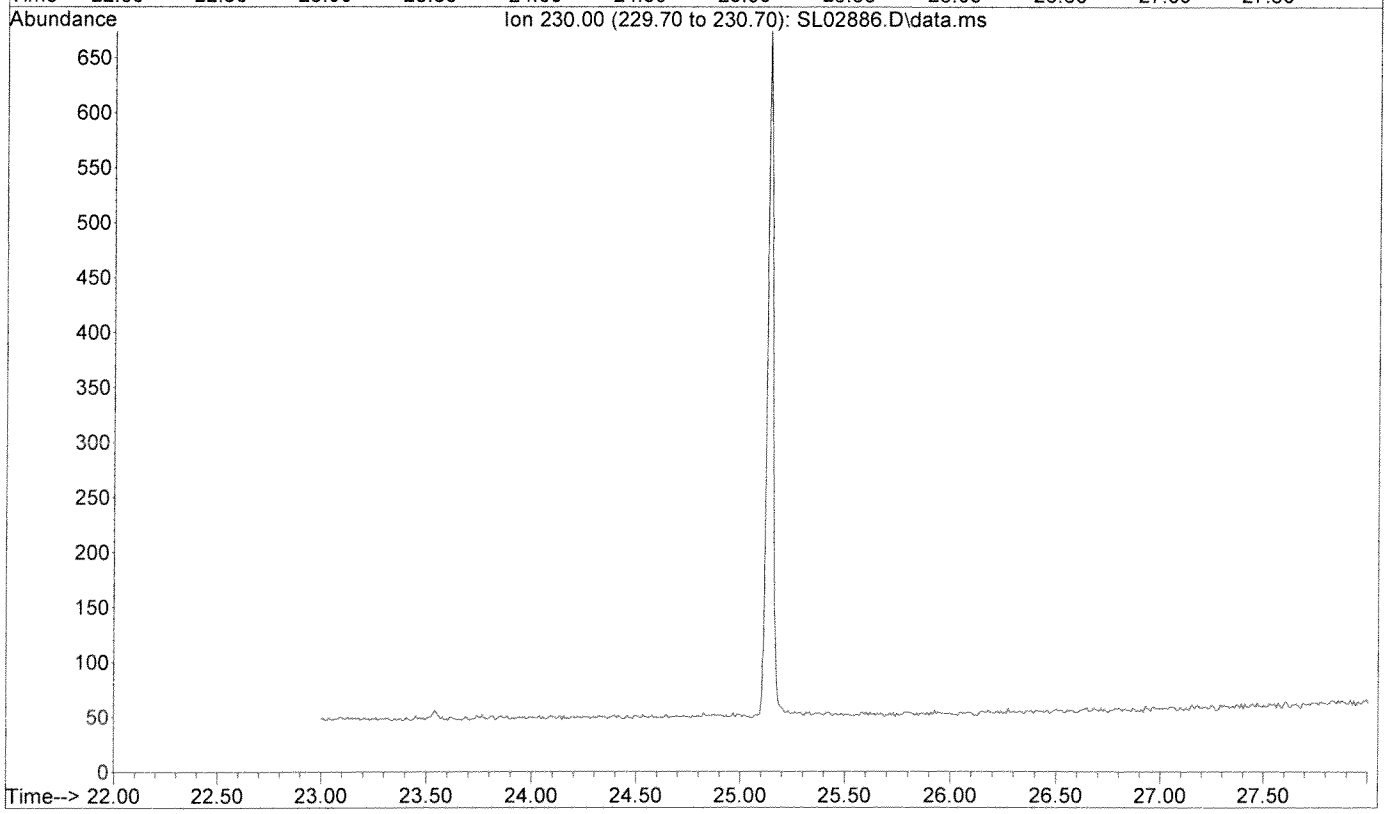
File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



US EPA ARCHIVE DOCUMENT

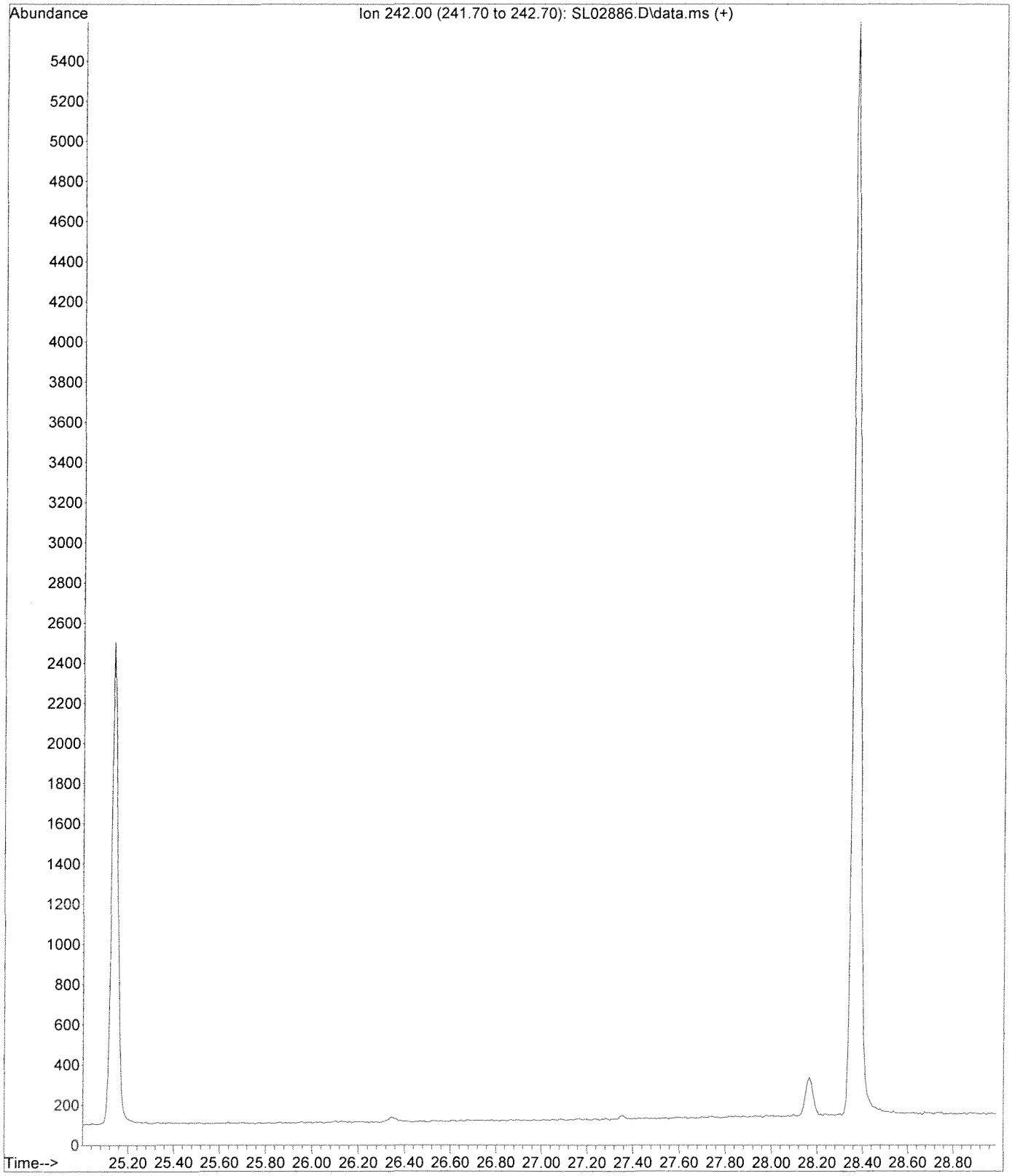
```
File        :C:\msdchem\1\DATA\030112\SL02886.D
Operator    : Syslo
Acquired    :  1 Mar 2012  15:41      using AcqMethod DROTPHX51611.M
Instrument :    Slick2
Sample Name: BLK030112
Misc Info   : Waste Dilution Blank 3/1/2012
Vial Number: 2
```





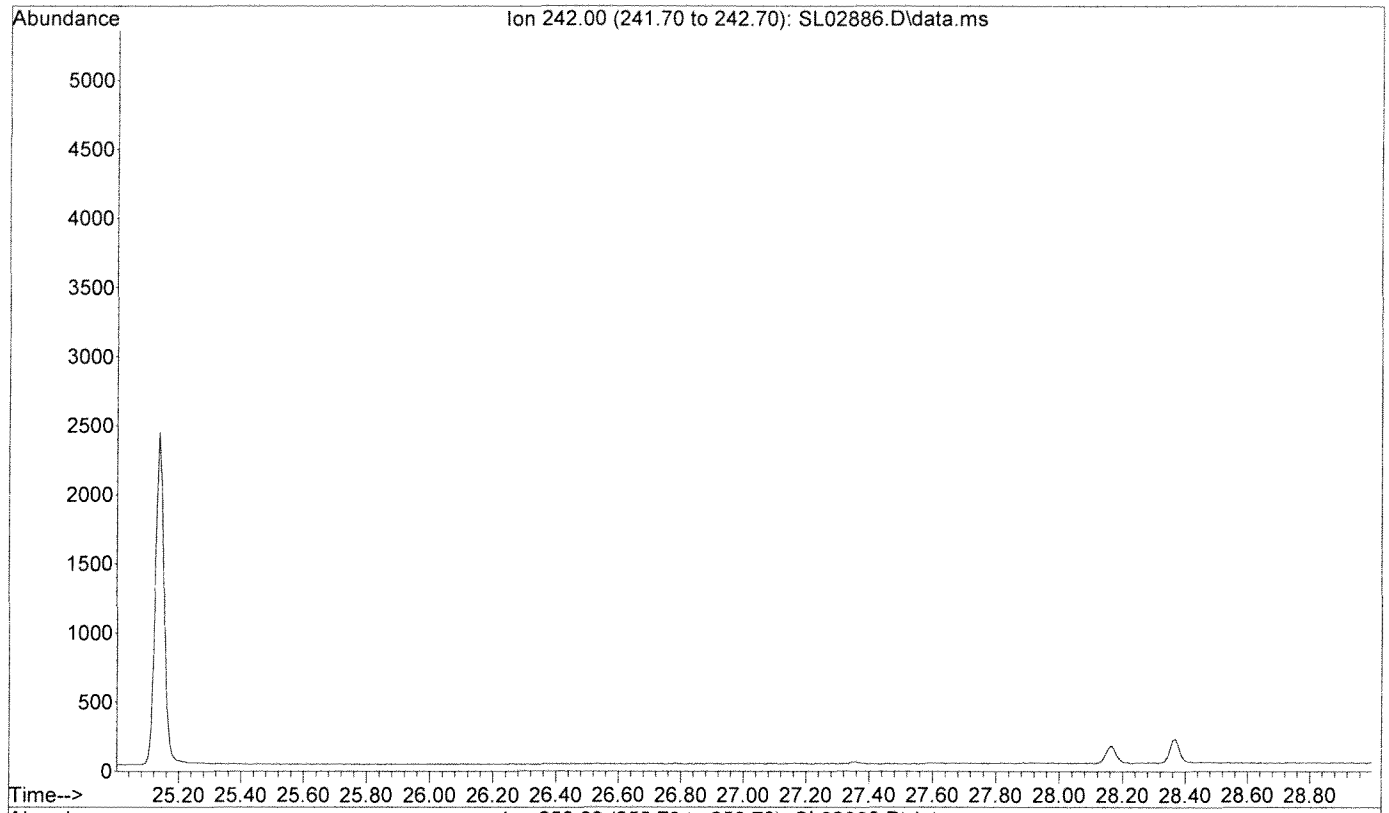
US EPA ARCHIVE DOCUMENT

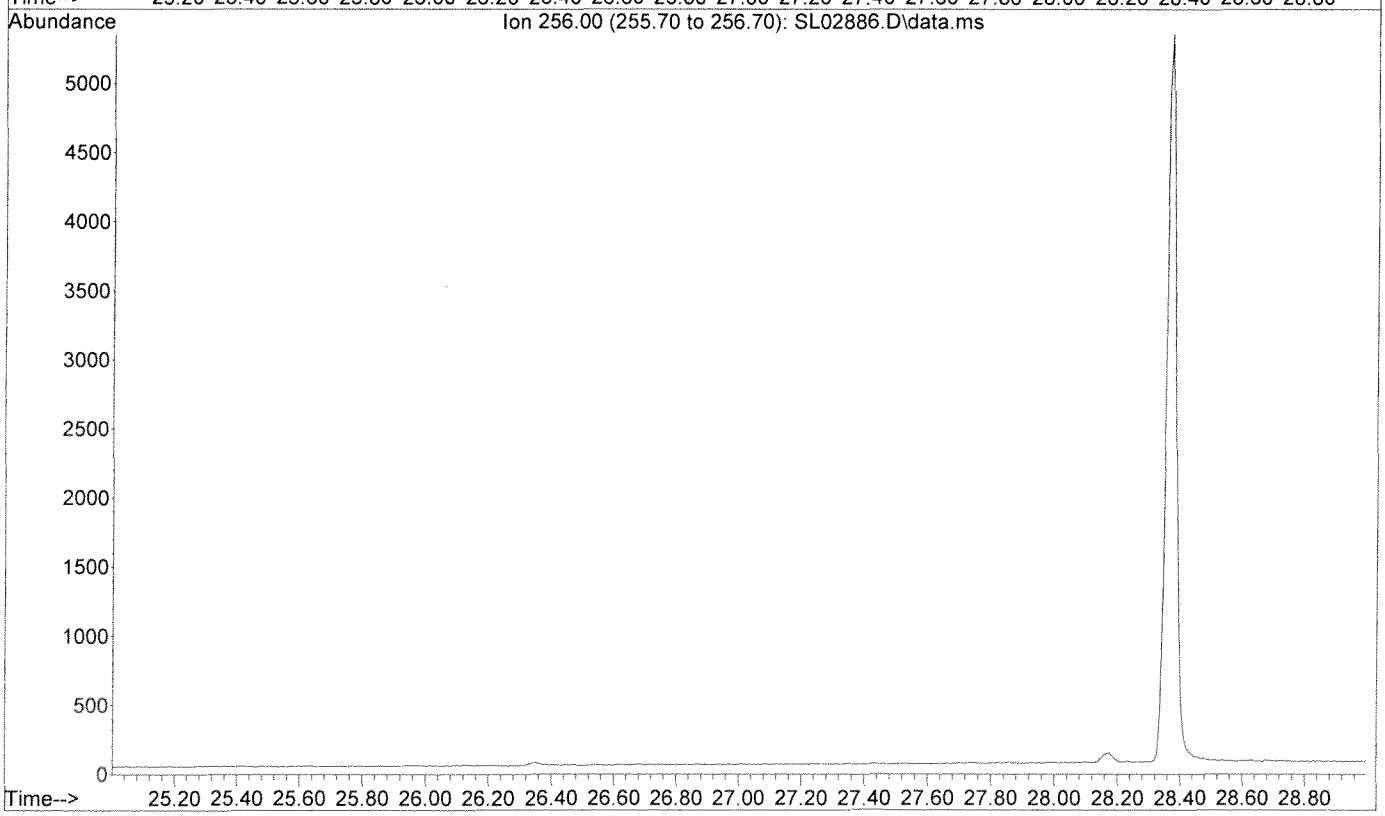
File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



US EPA ARCHIVE DOCUMENT

```
File         :C:\msdchem\1\DATA\030112\SL02886.D
Operator     : Syslo
Acquired     :  1 Mar 2012  15:41      using AcqMethod DROTPHX51611.M
Instrument   :   Slick2
Sample Name: BLK030112
Misc Info    : Waste Dilution Blank 3/1/2012
Vial Number: 2
```





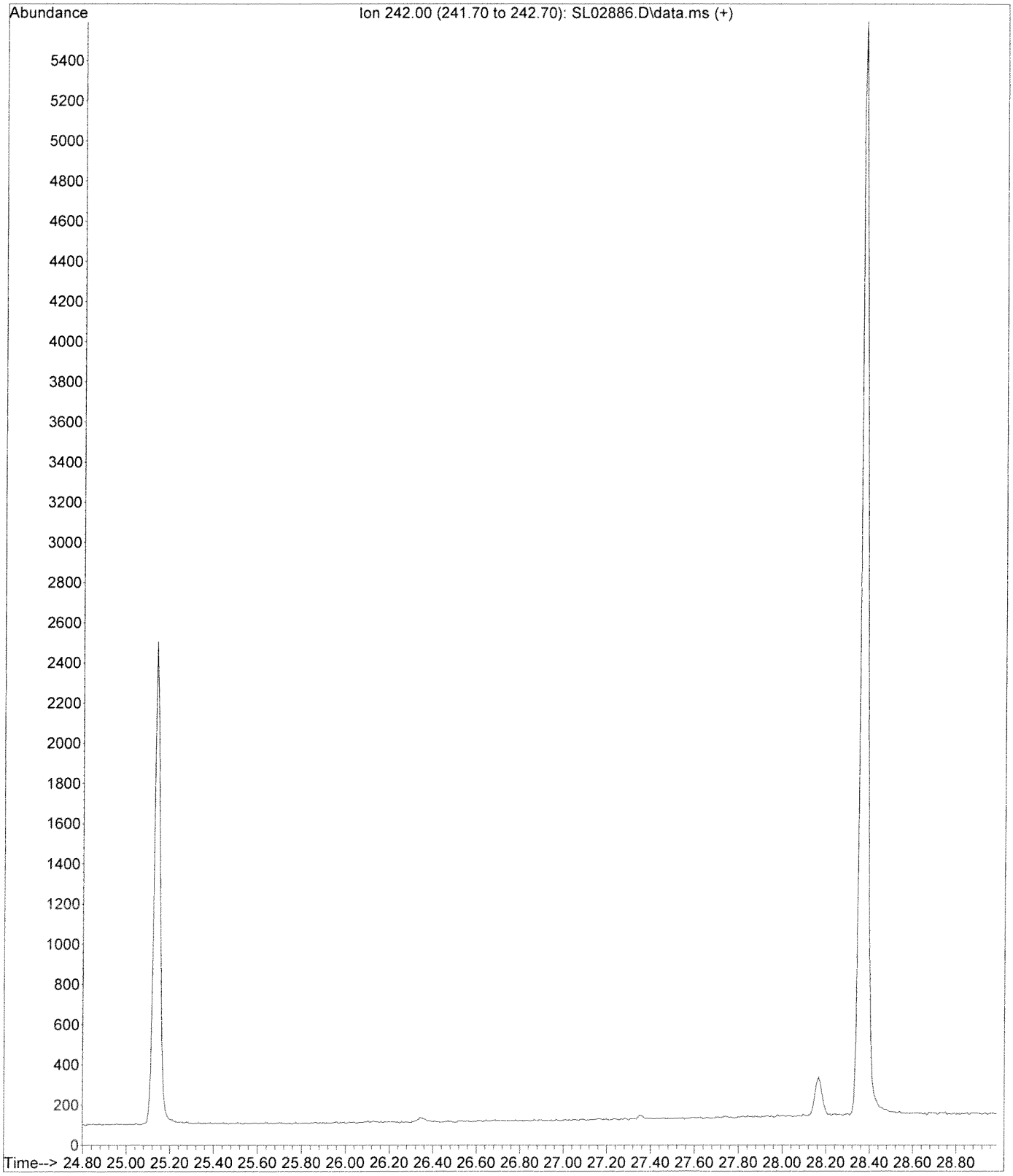
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02886.D
Operator : Syslo
Acquired : 1 Mar 2012 15:41 using AcqMethod DROTPHX51611.M
Instrument : Slick2
Sample Name: BLK030112
Misc Info : Waste Dilution Blank 3/1/2012
Vial Number: 2



US EPA ARCHIVE DOCUMENT

SERAS-017-0005

SL02890.D

US EPA ARCHIVE DOCUMENT

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02890.D
Acq On : 1 Mar 2012 20:37
Operator : Syslo
Sample : SERAS-017-0005
Misc : WCS-6B-072223-092910-JPS-KA-002-20
ALS Vial : 6 Sample Multiplier: 1

Quant Time: Mar 01 21:27:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	324307	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	97344	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	131670	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	115625	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.821	188	570872	16.55	ug/mL	0.00
Spiked Amount 20.000			Recovery = 82.75%			
3) d14-o-Terphenyl {S}	19.733	244	266587	17.59	ug/mL	0.00
Spiked Amount 20.000			Recovery = 87.95%			
4) 5a-Androstane {S}	21.140	260	37544	20.36	ug/mL	0.00
Spiked Amount 20.000			Recovery = 101.80%			
7) d62-Triacontane {S}	28.156	66	239975	20.60	ug/mL	0.00
Spiked Amount 20.000			Recovery = 103.00%			
Target Compounds						Qvalue

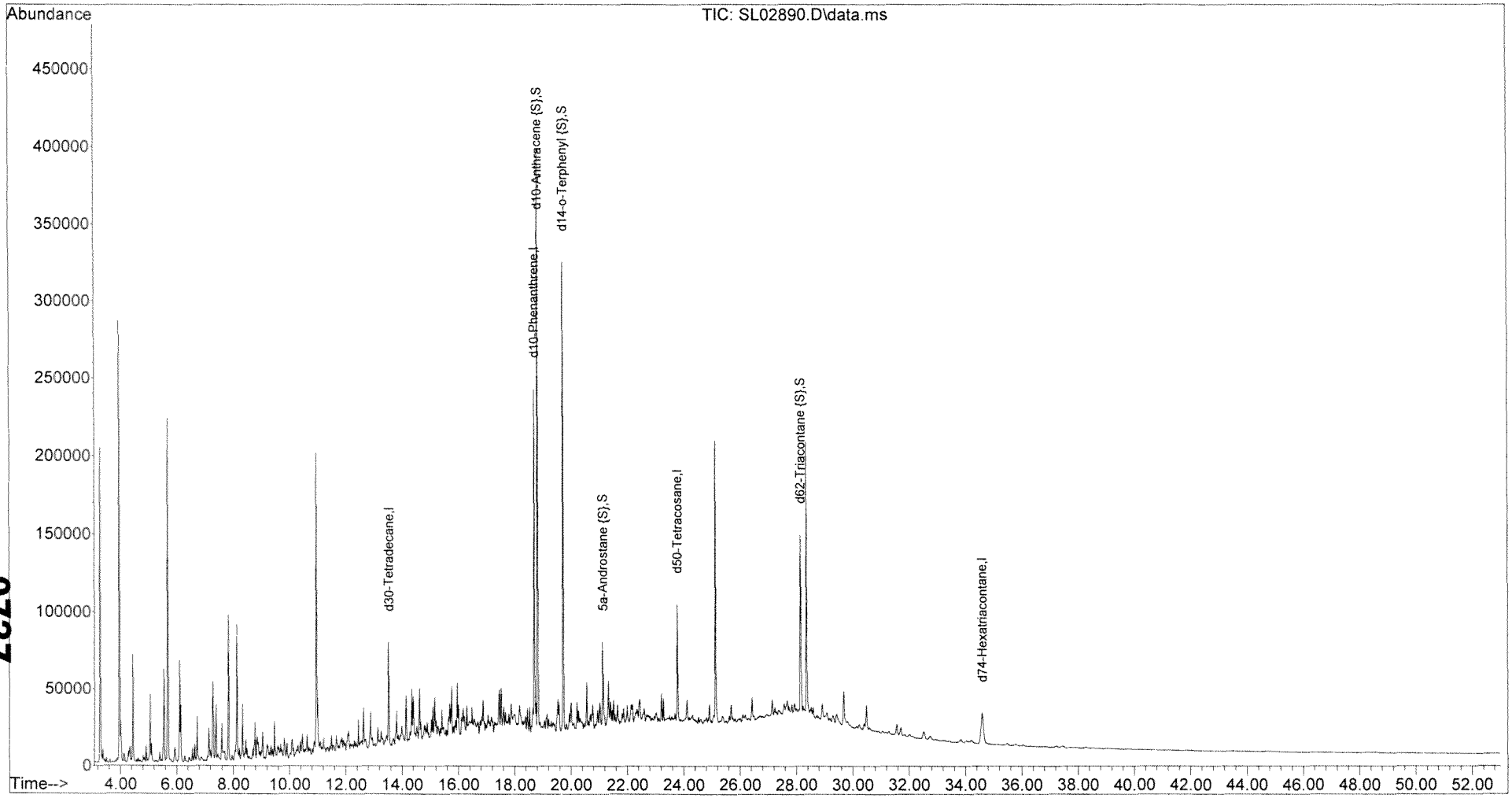
(#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02890.D
Acq On : 1 Mar 2012 20:37
Operator : Syslo
Sample : SERAS-017-0005
Misc : WCS-6B-072223-092910-JPS-KA-002-20
ALS Vial : 6 Sample Multiplier: 1

Quant Time: Mar 01 21:27:02 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration



0727

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

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US EPA ARCHIVE DOCUMENT

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

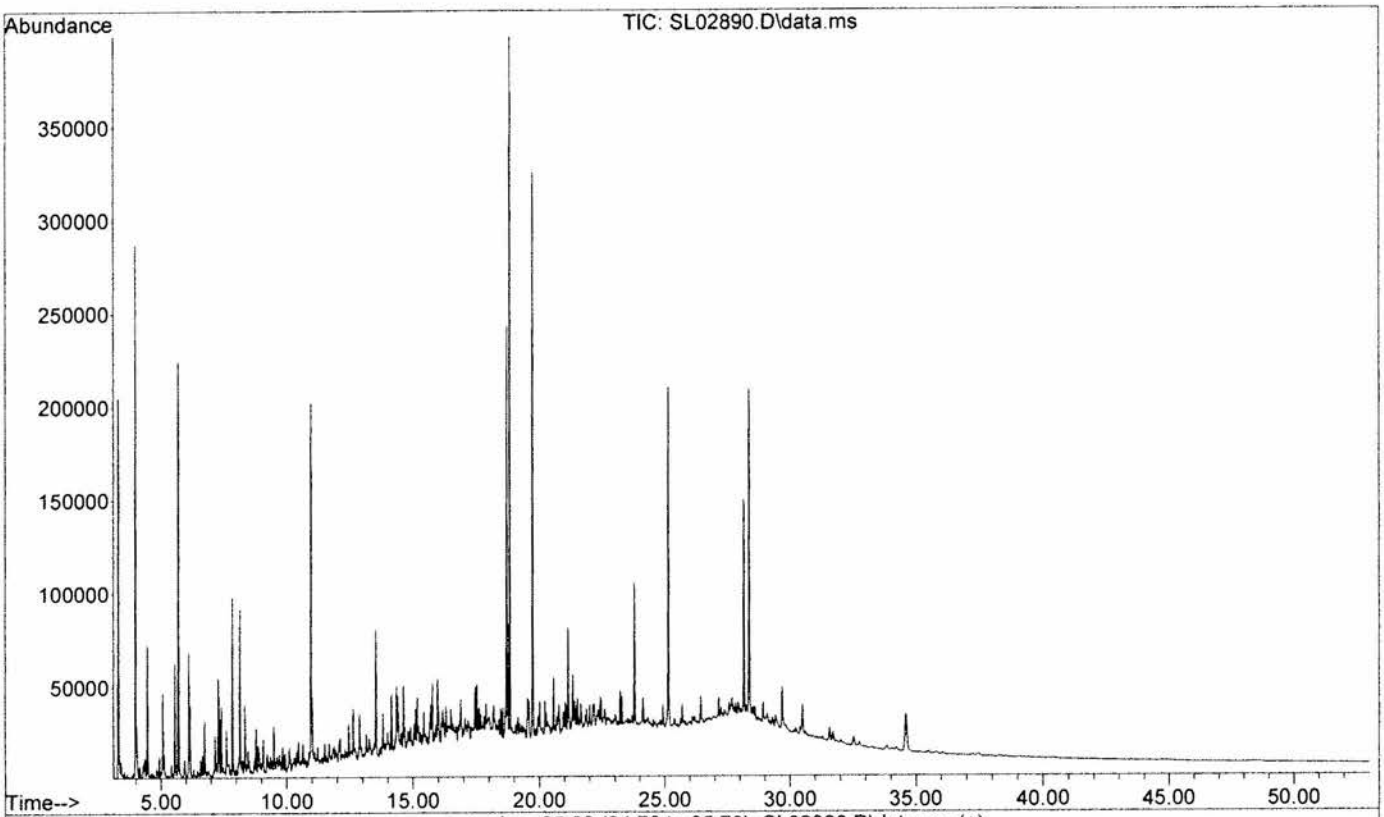
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

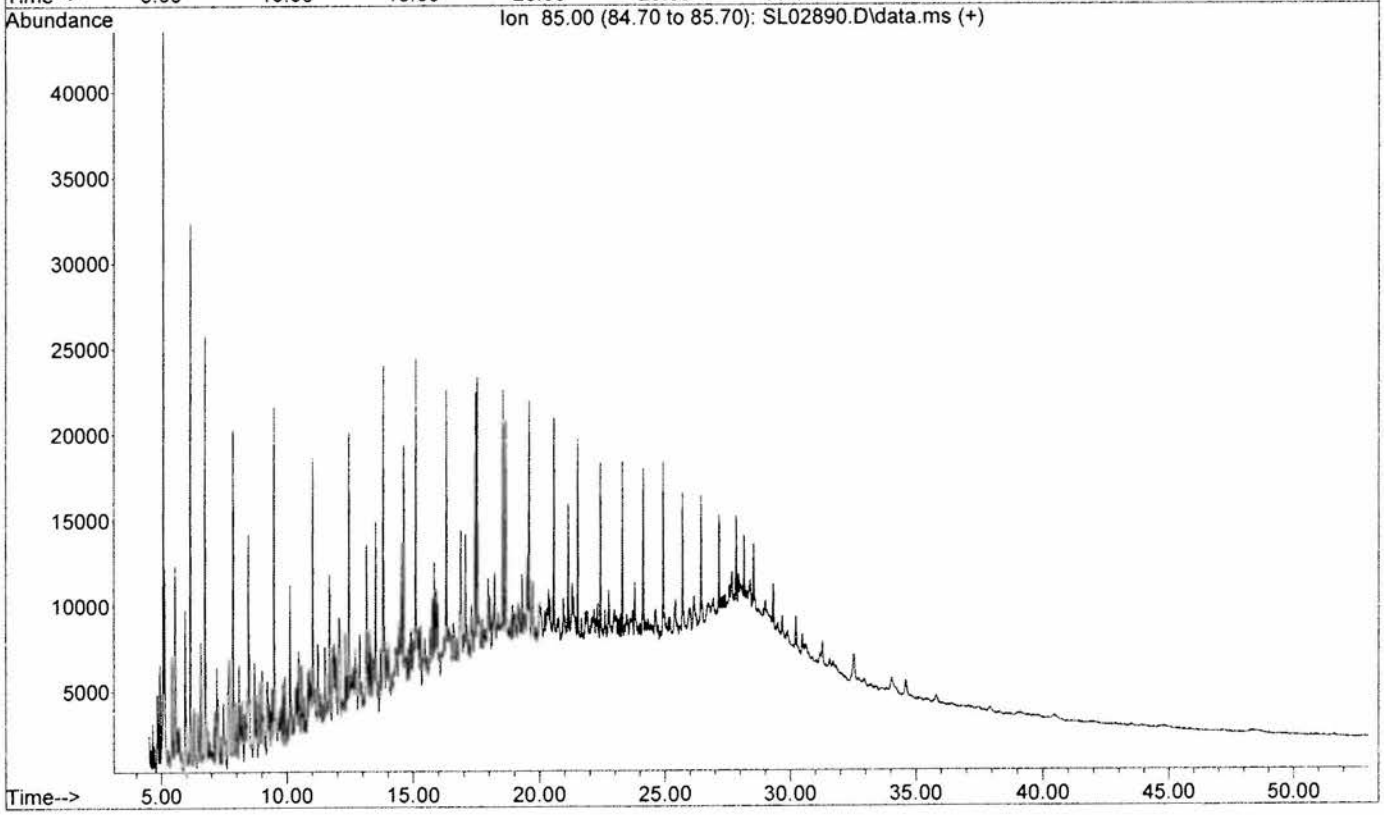
US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```



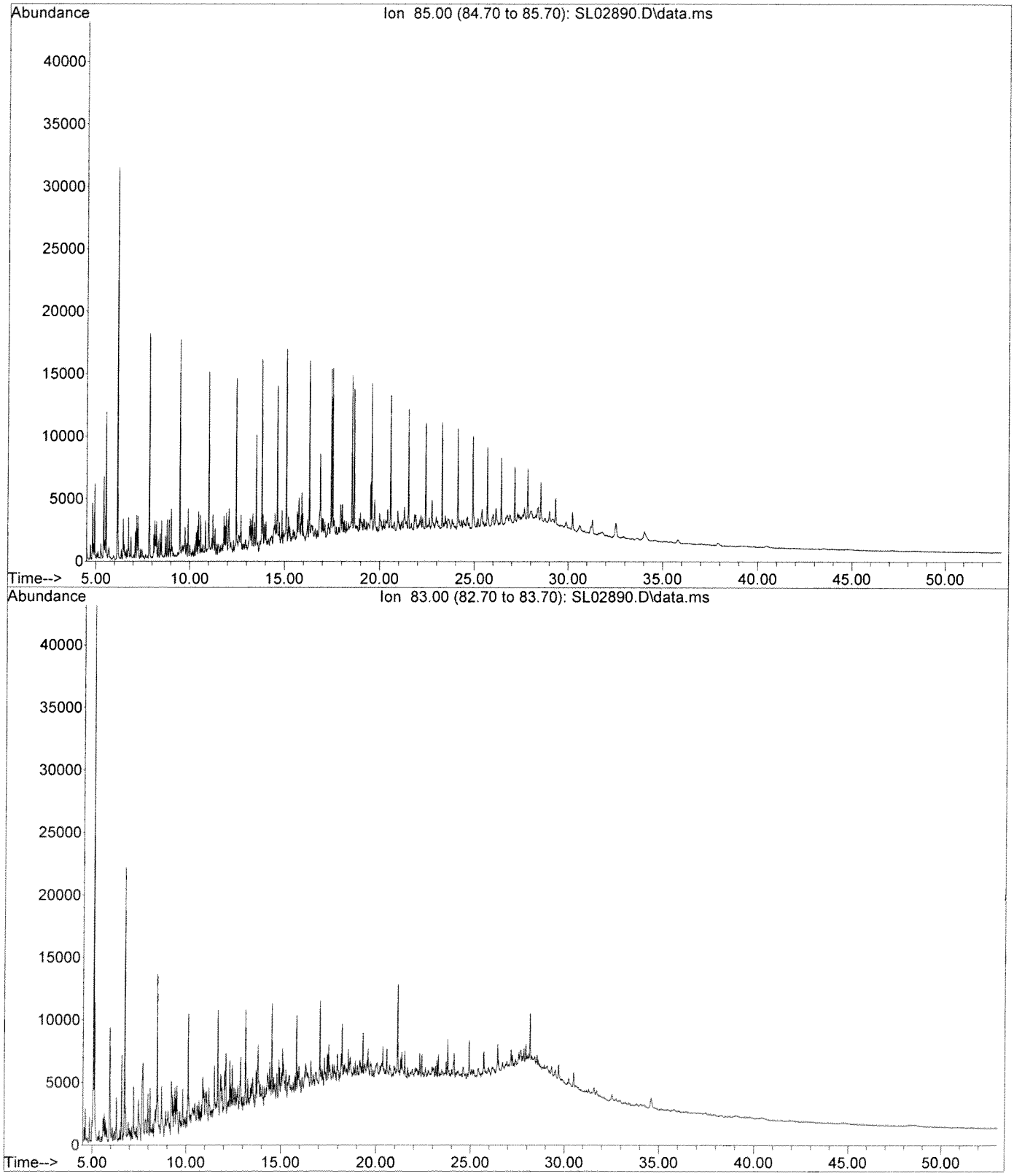


US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```



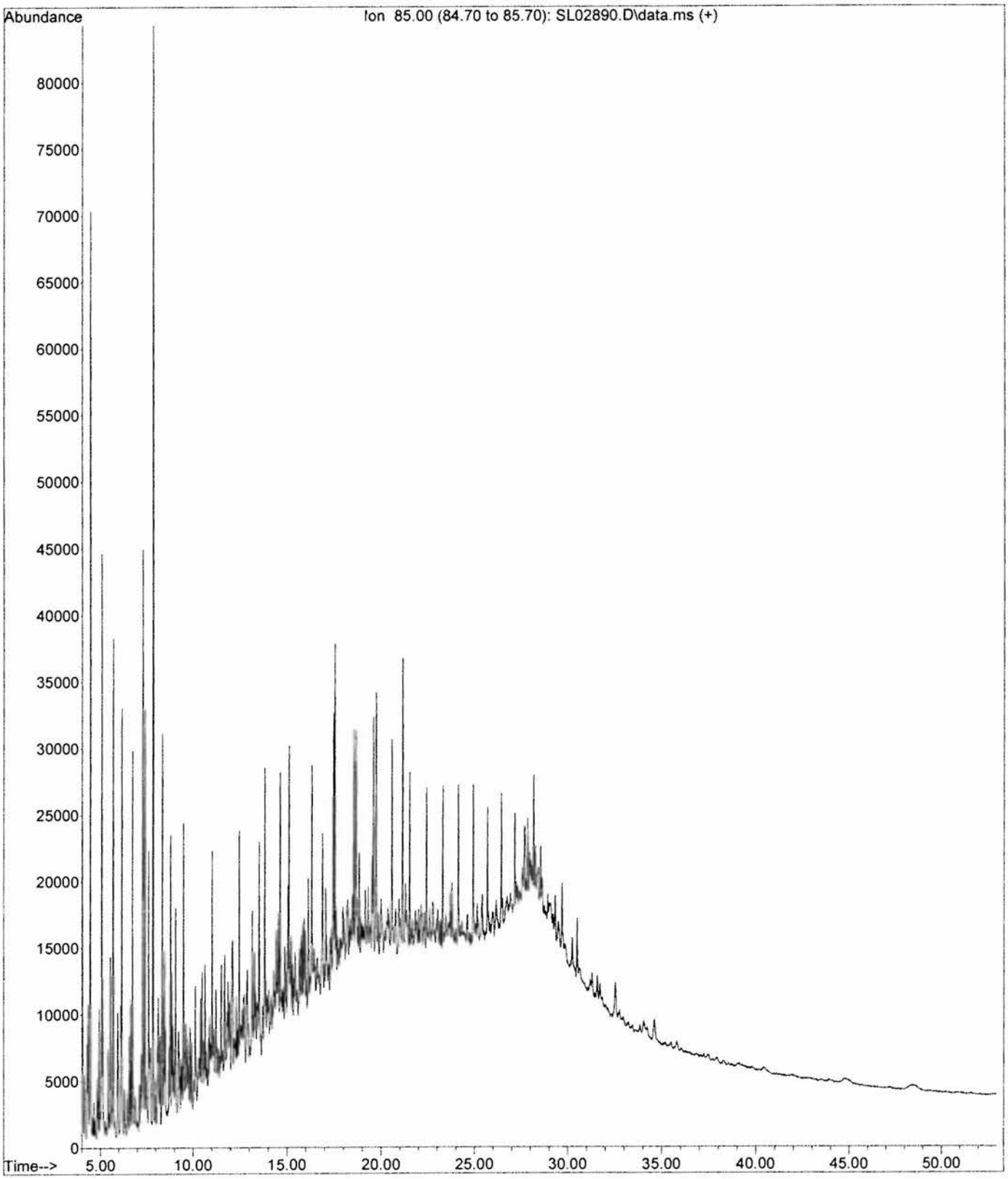
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



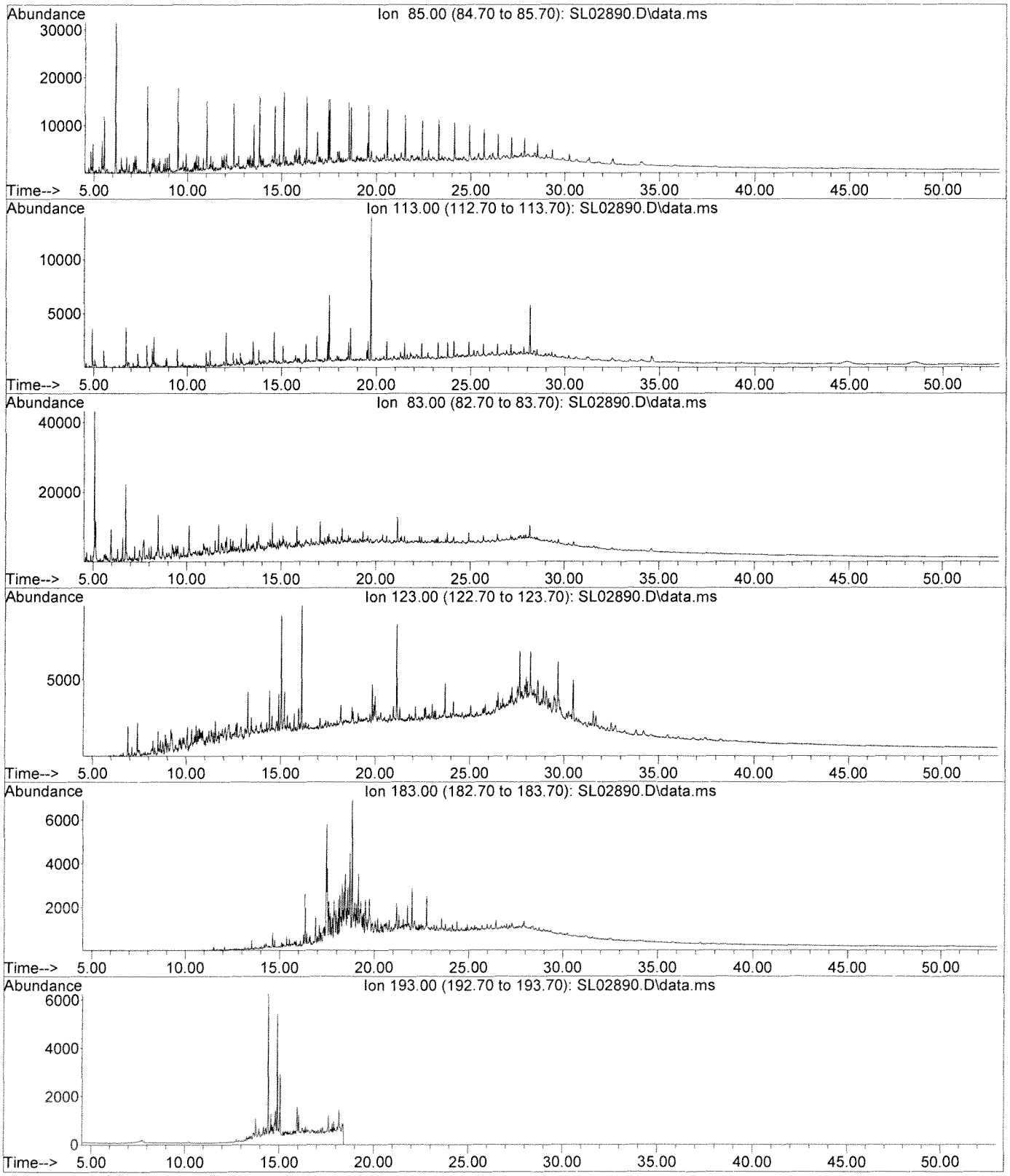
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```



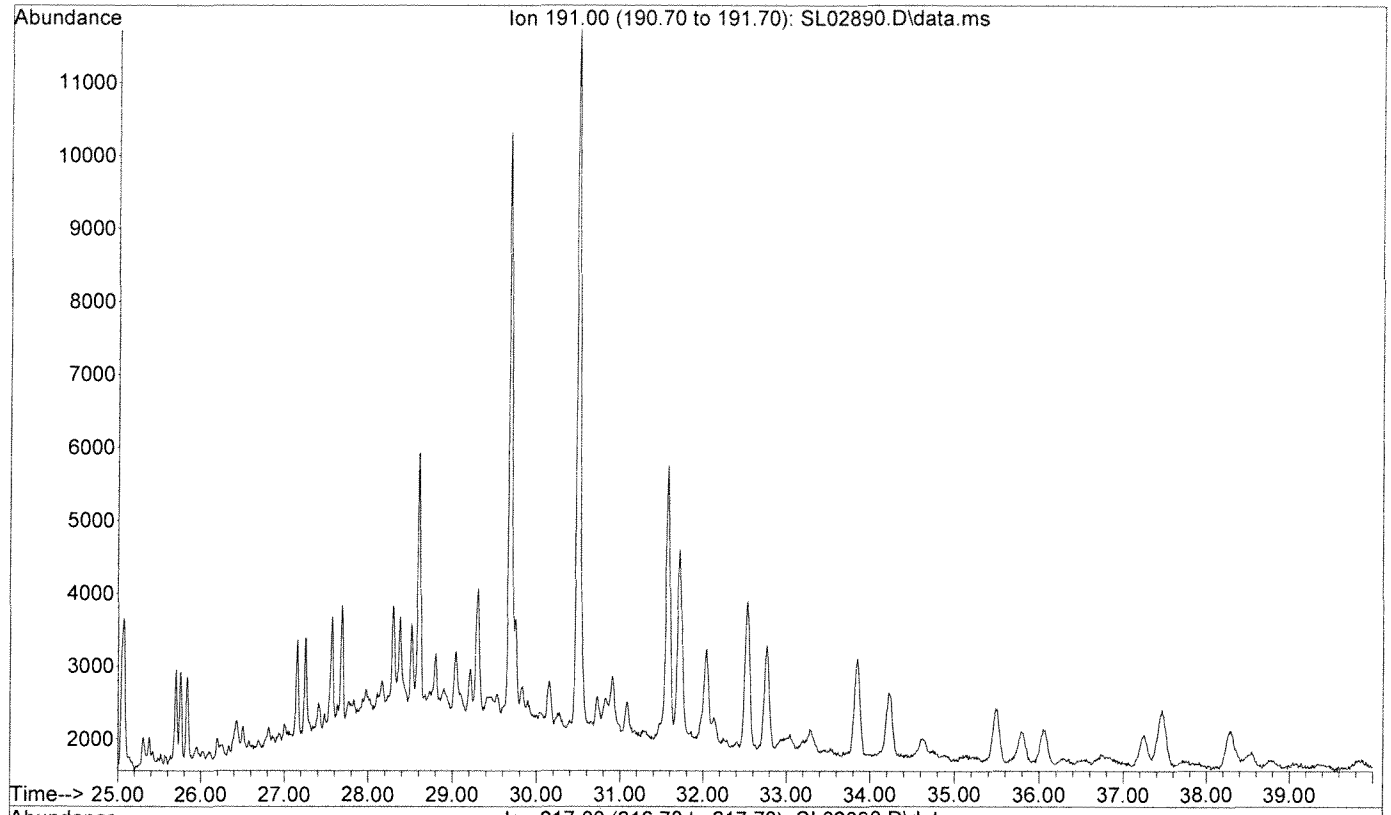
US EPA ARCHIVE DOCUMENT

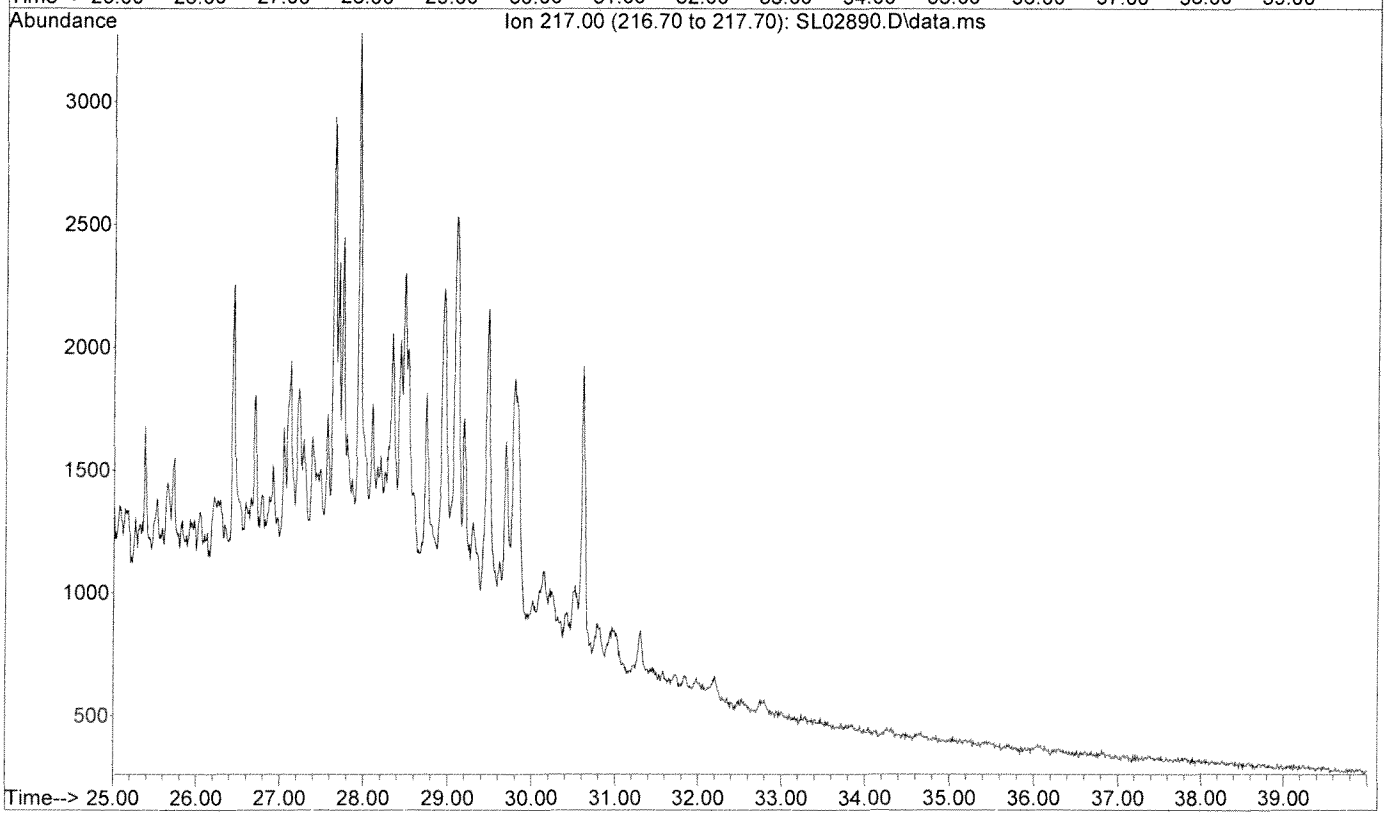
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37     using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```





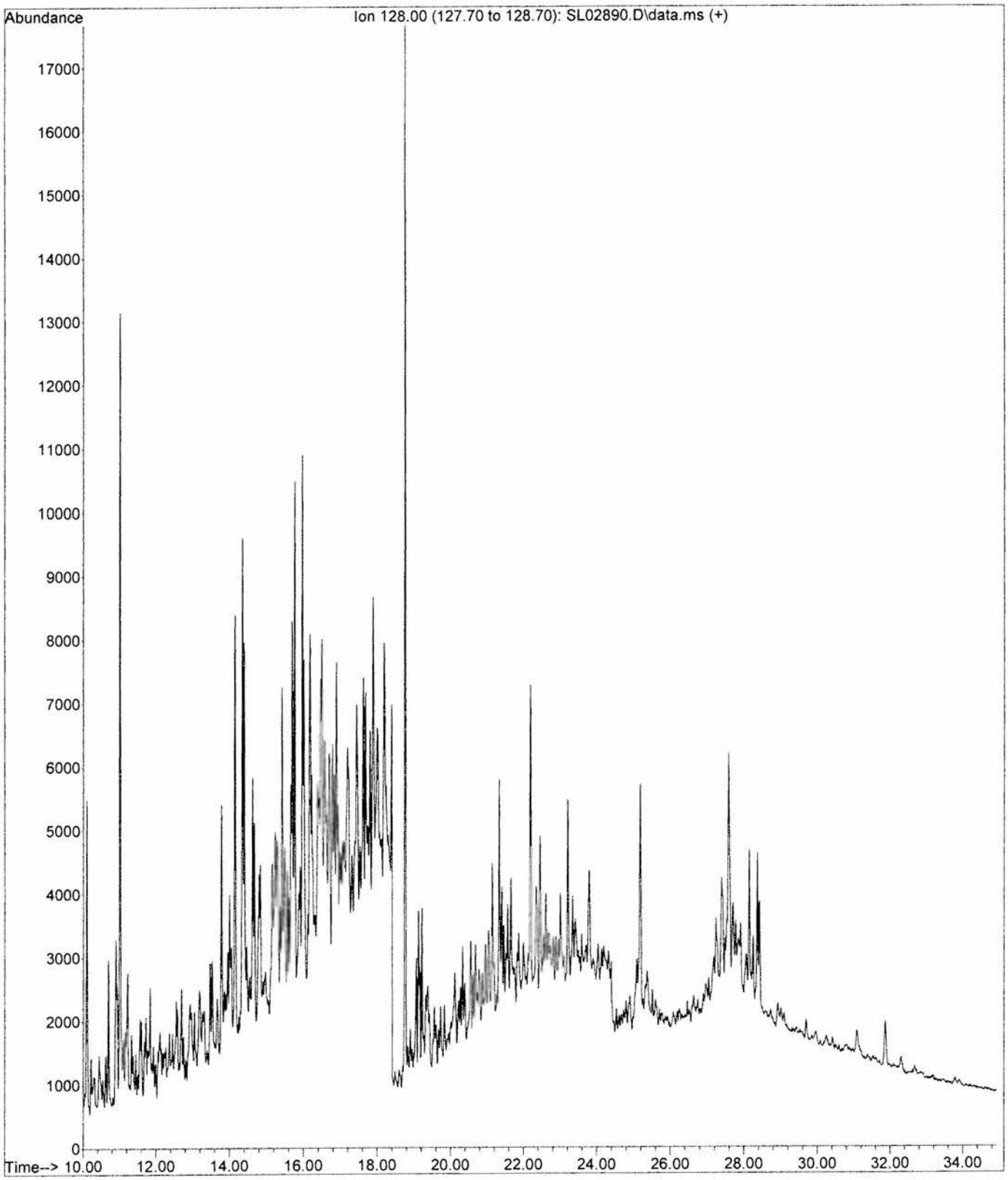
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



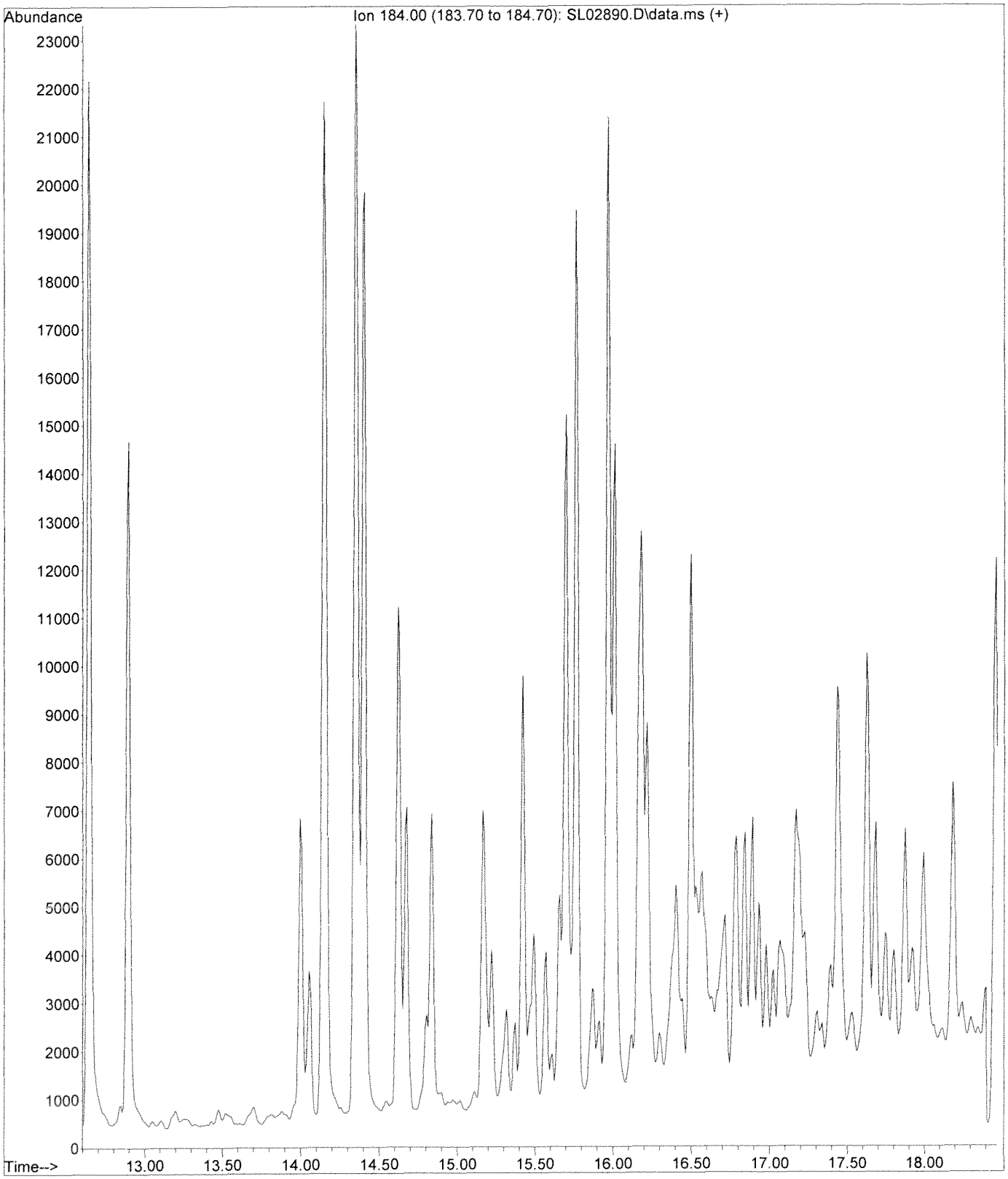
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

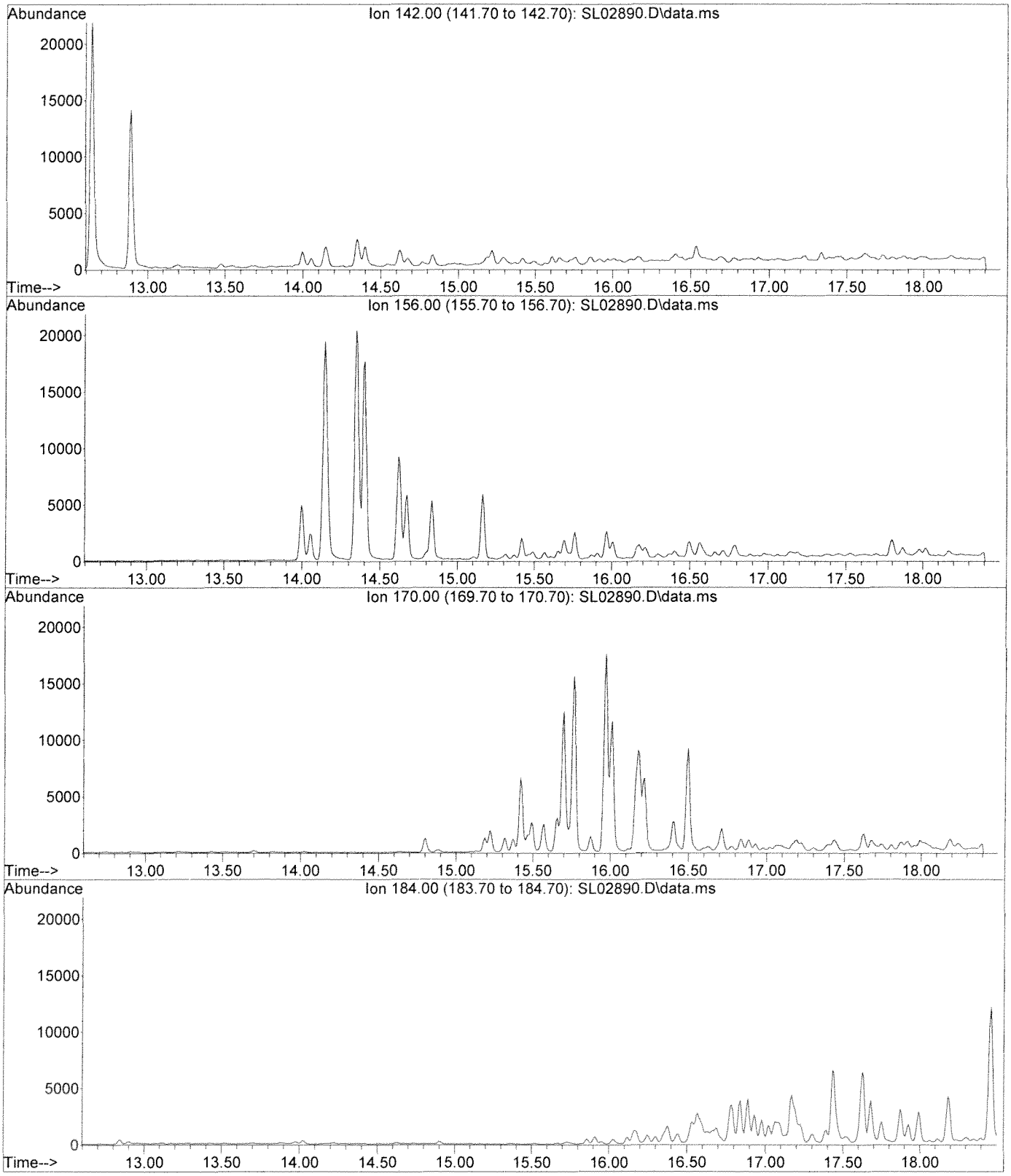
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



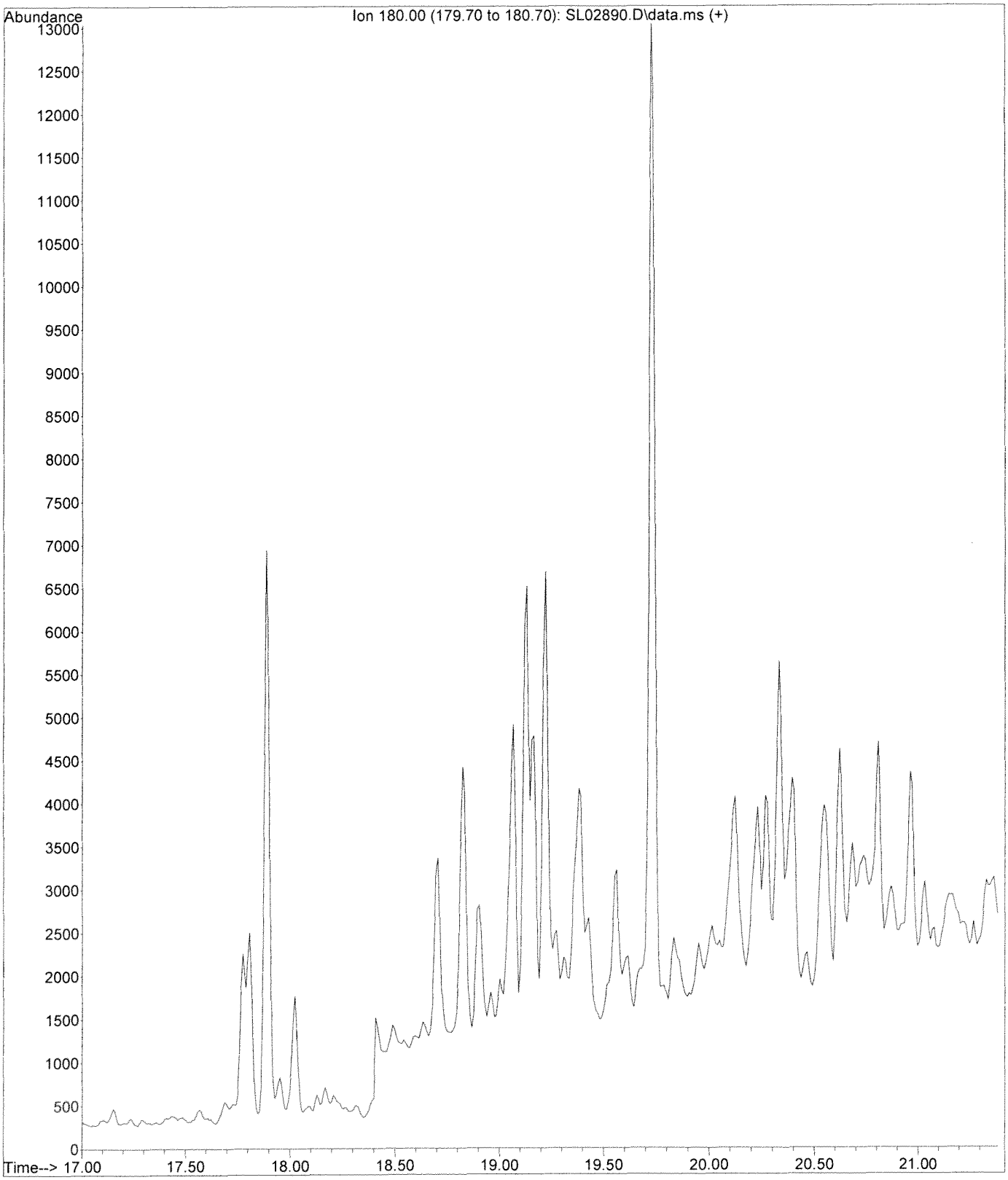
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37     using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```



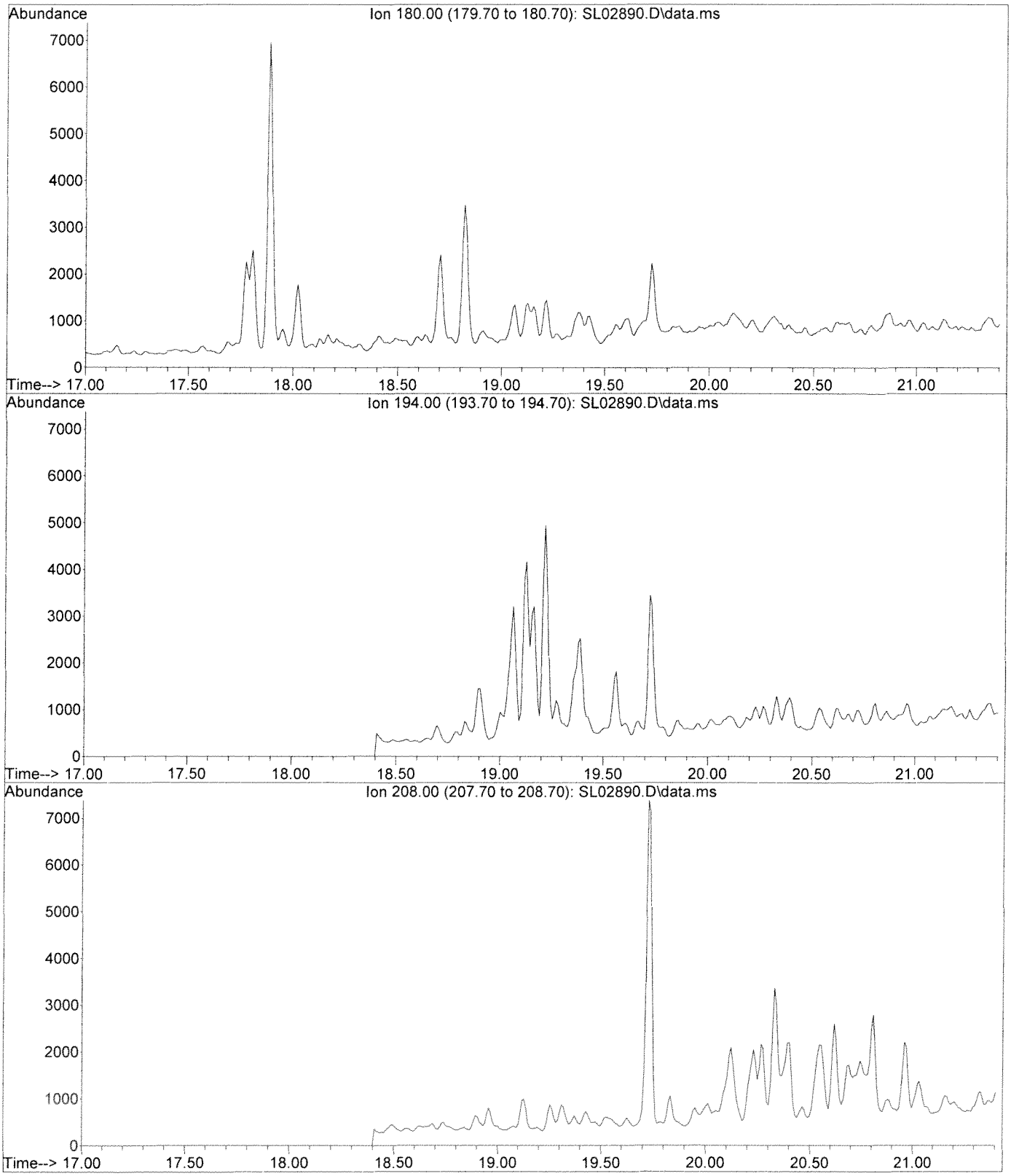
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



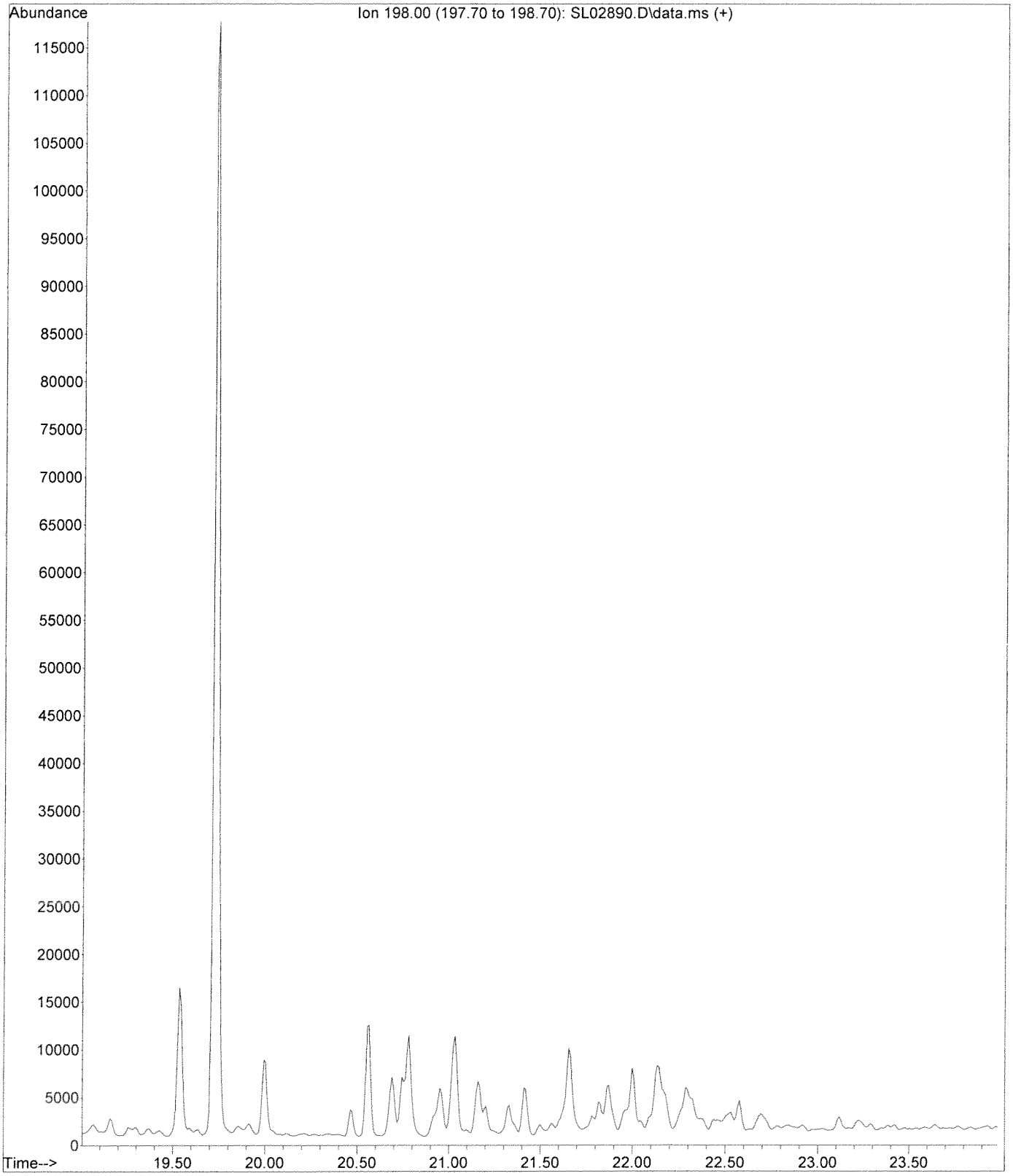
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



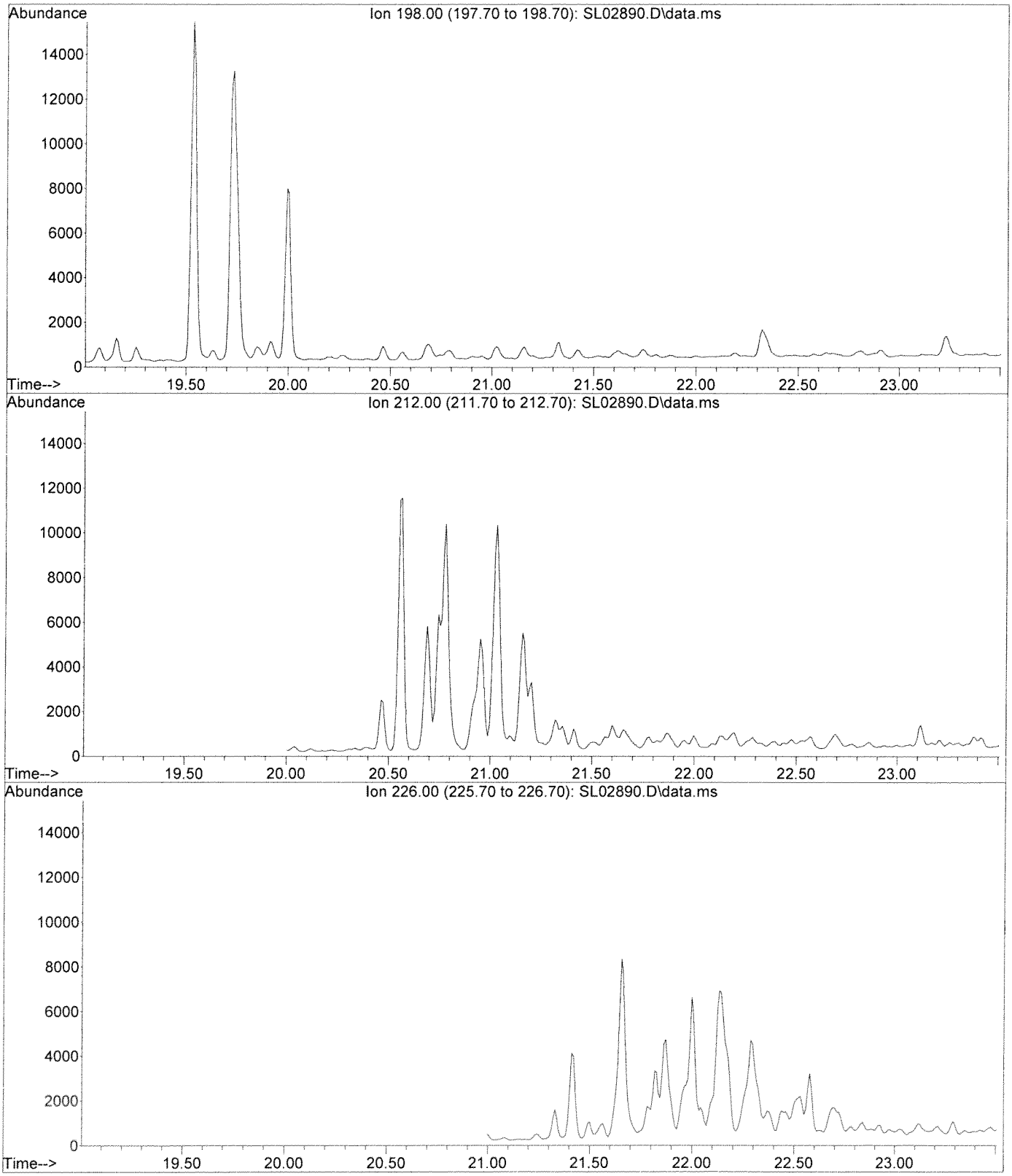
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



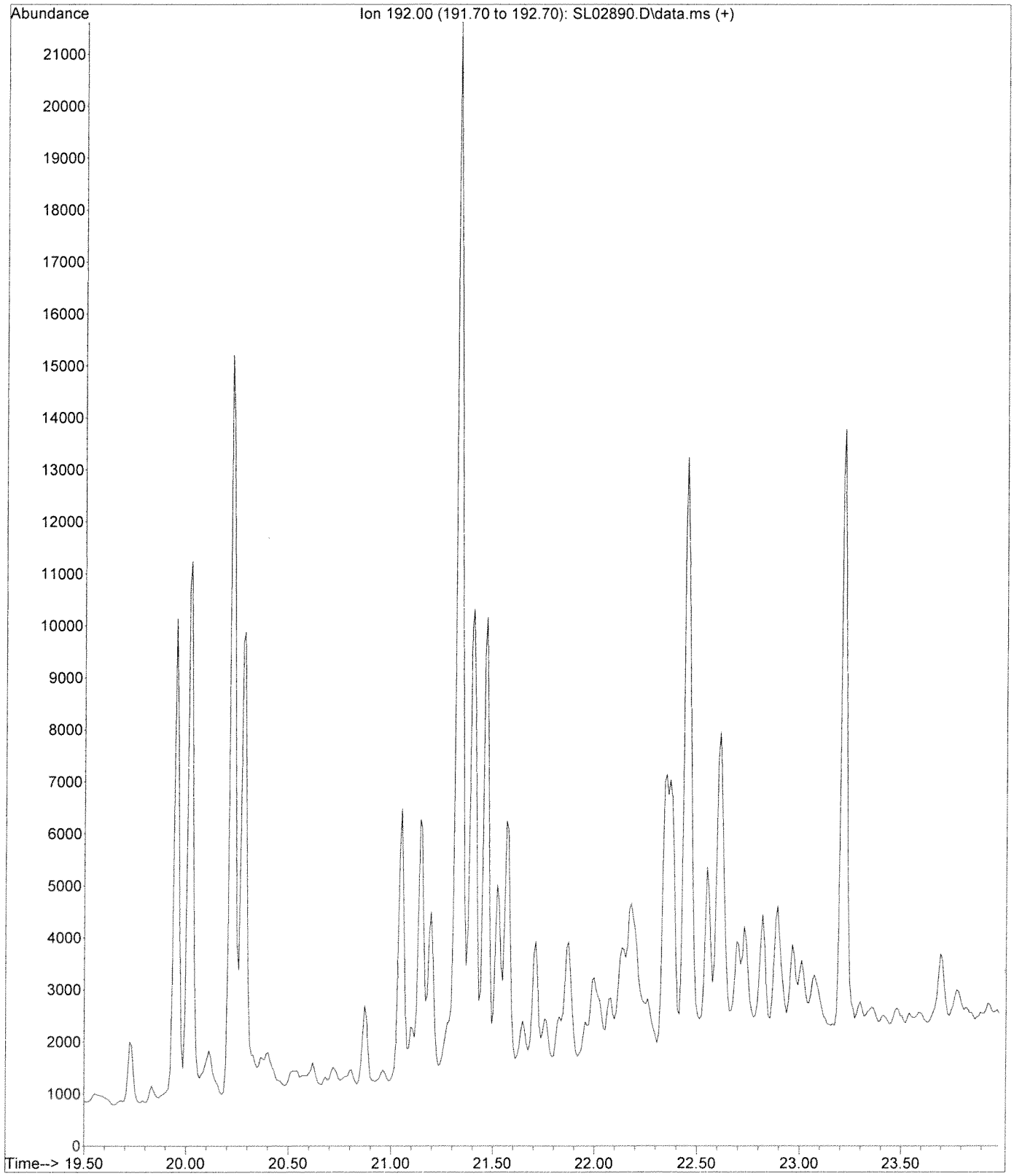
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



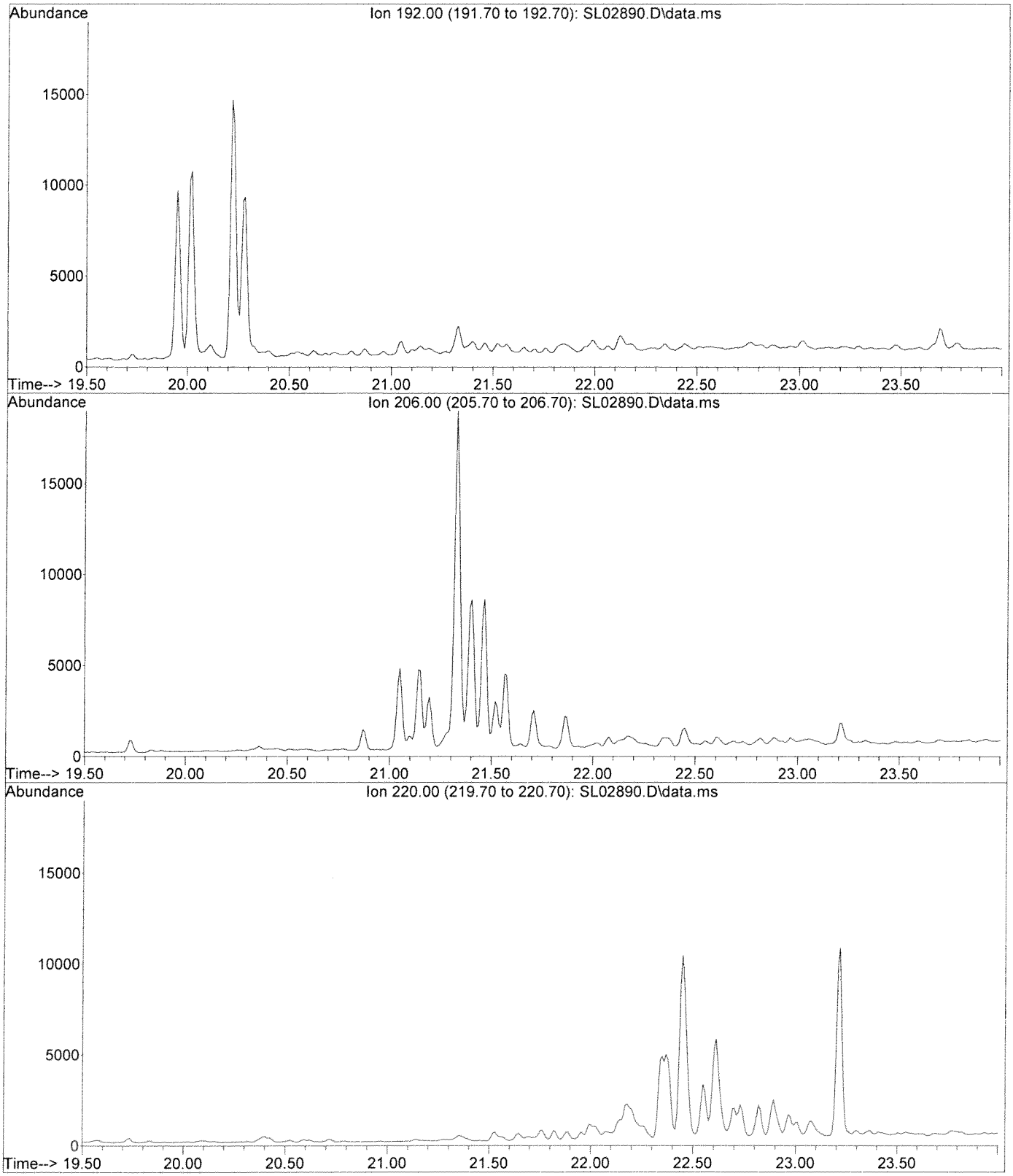
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37     using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```



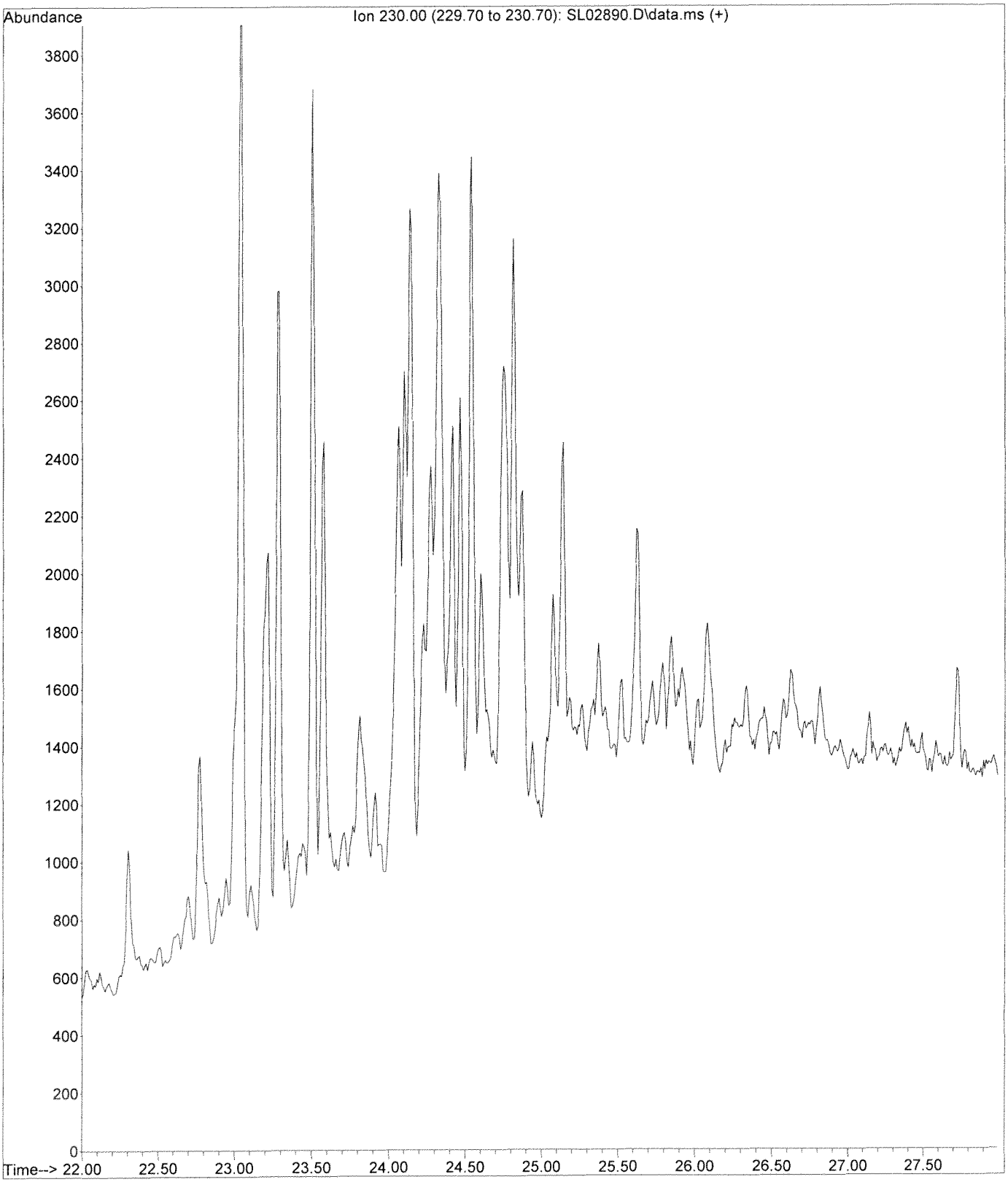
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



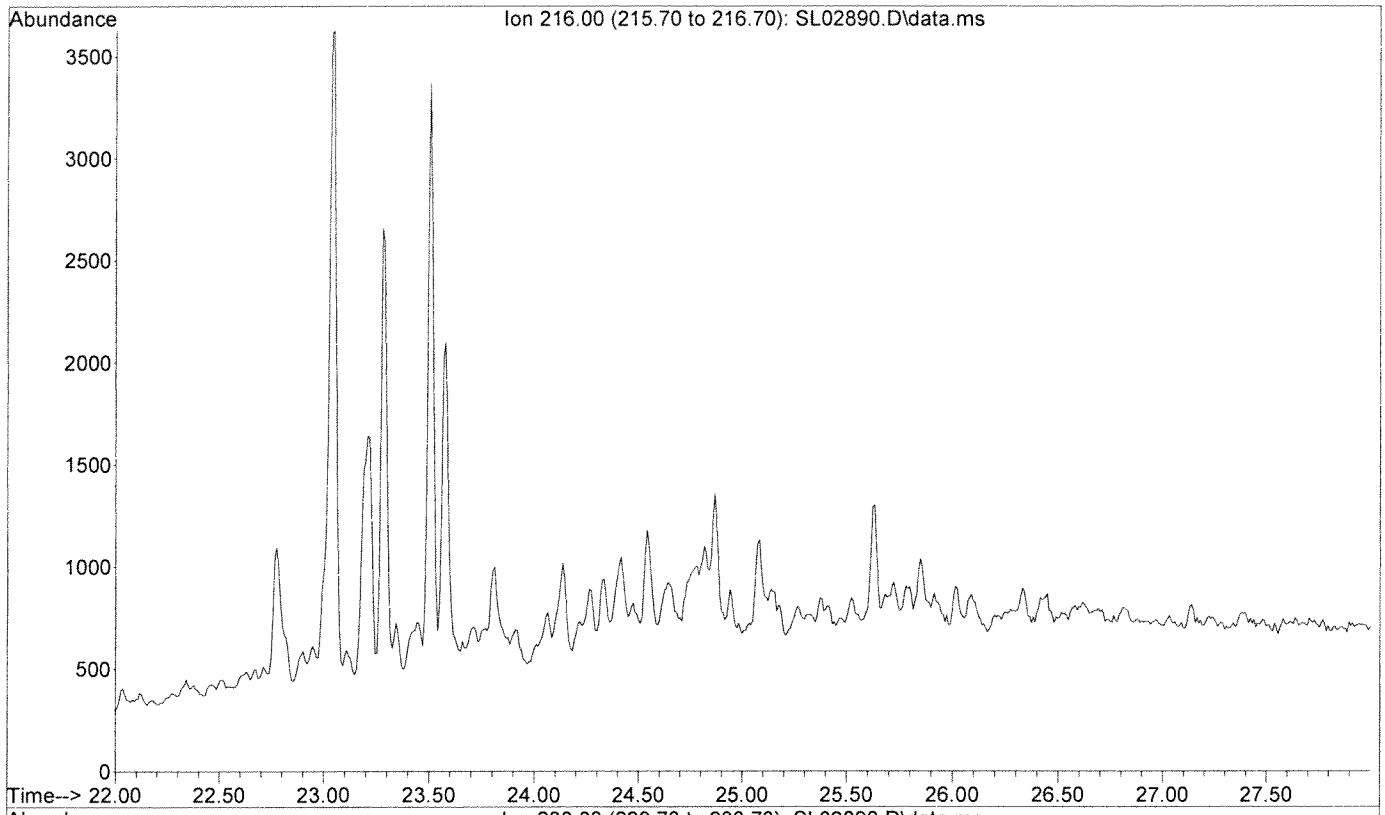
US EPA ARCHIVE DOCUMENT

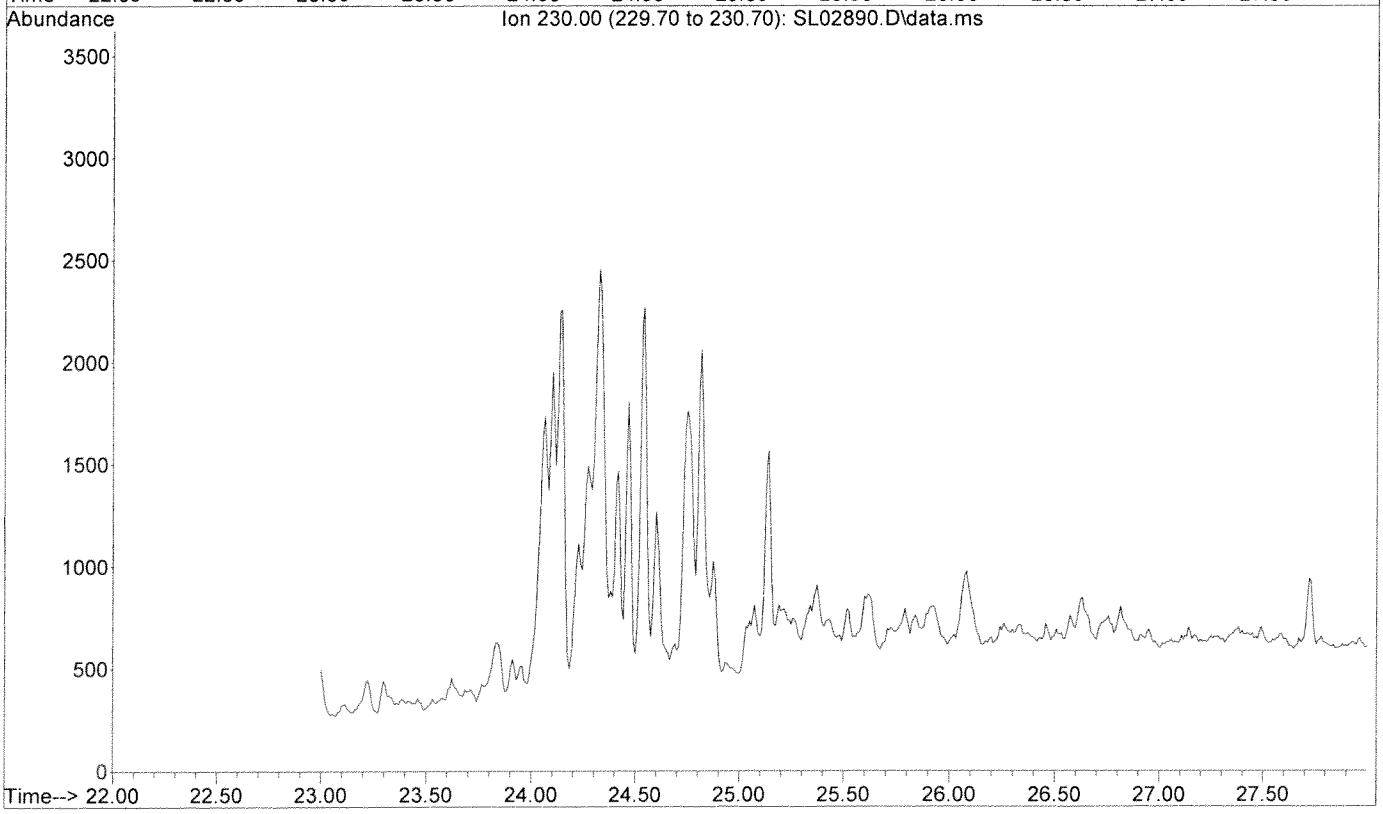
File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



US EPA ARCHIVE DOCUMENT

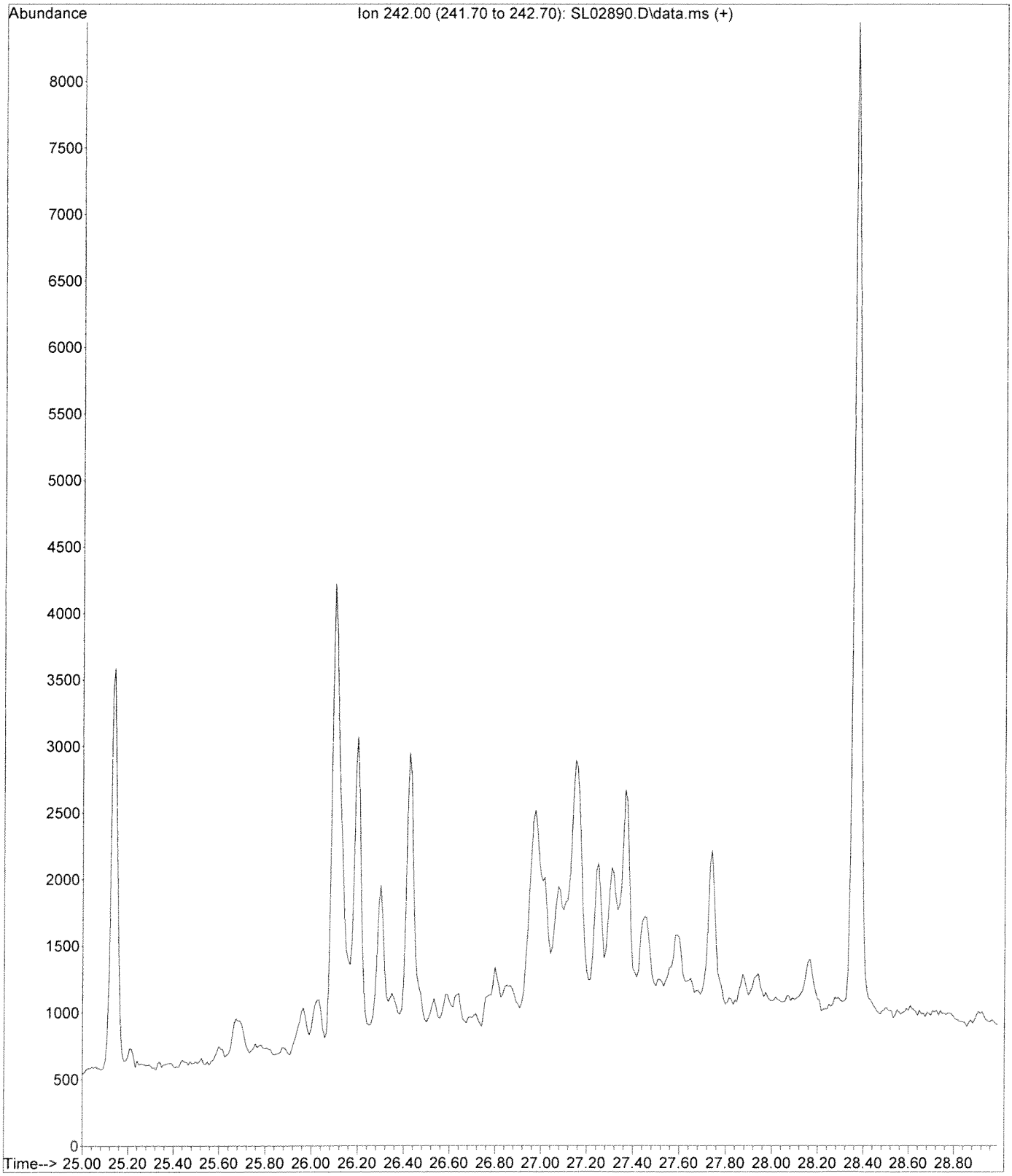
```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37     using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```





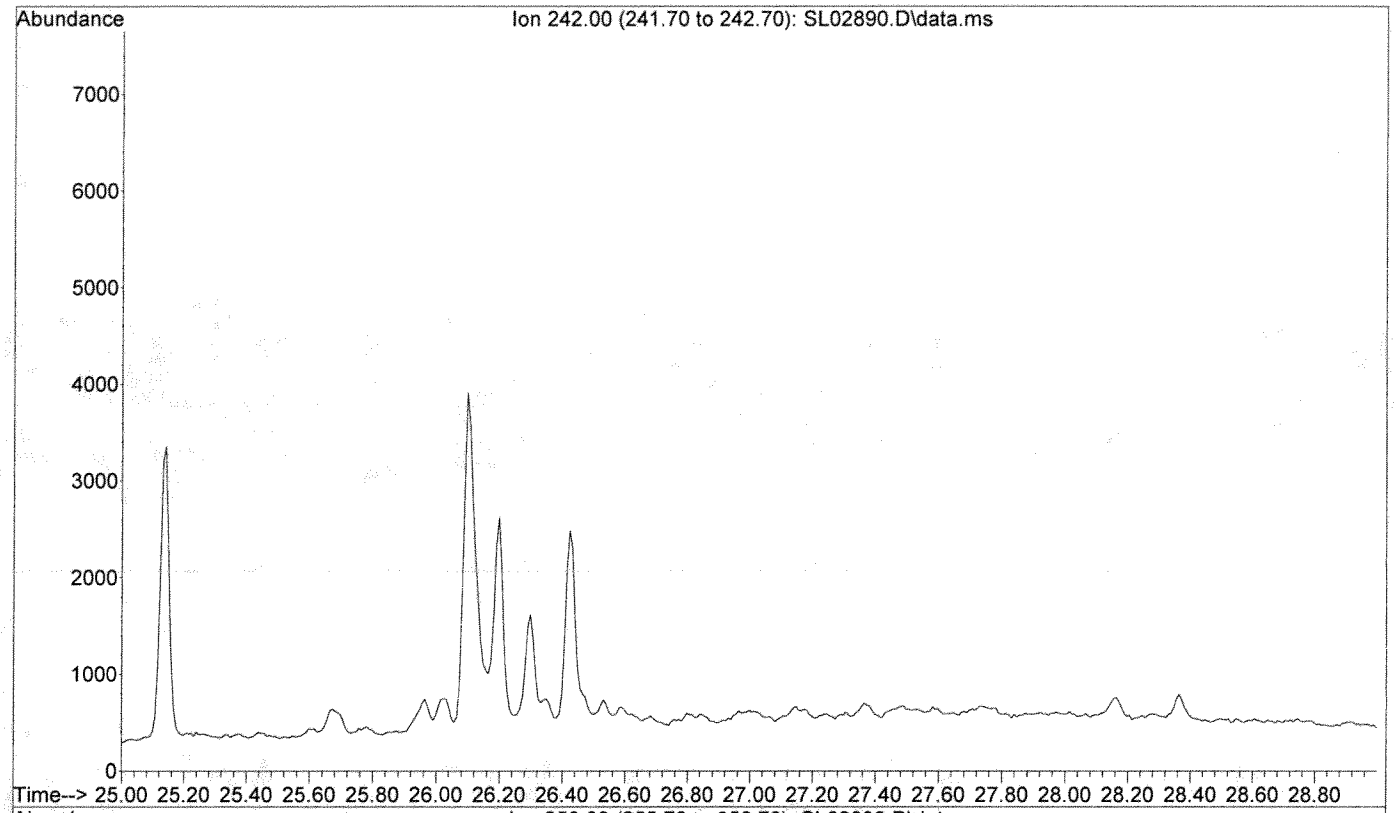
US EPA ARCHIVE DOCUMENT

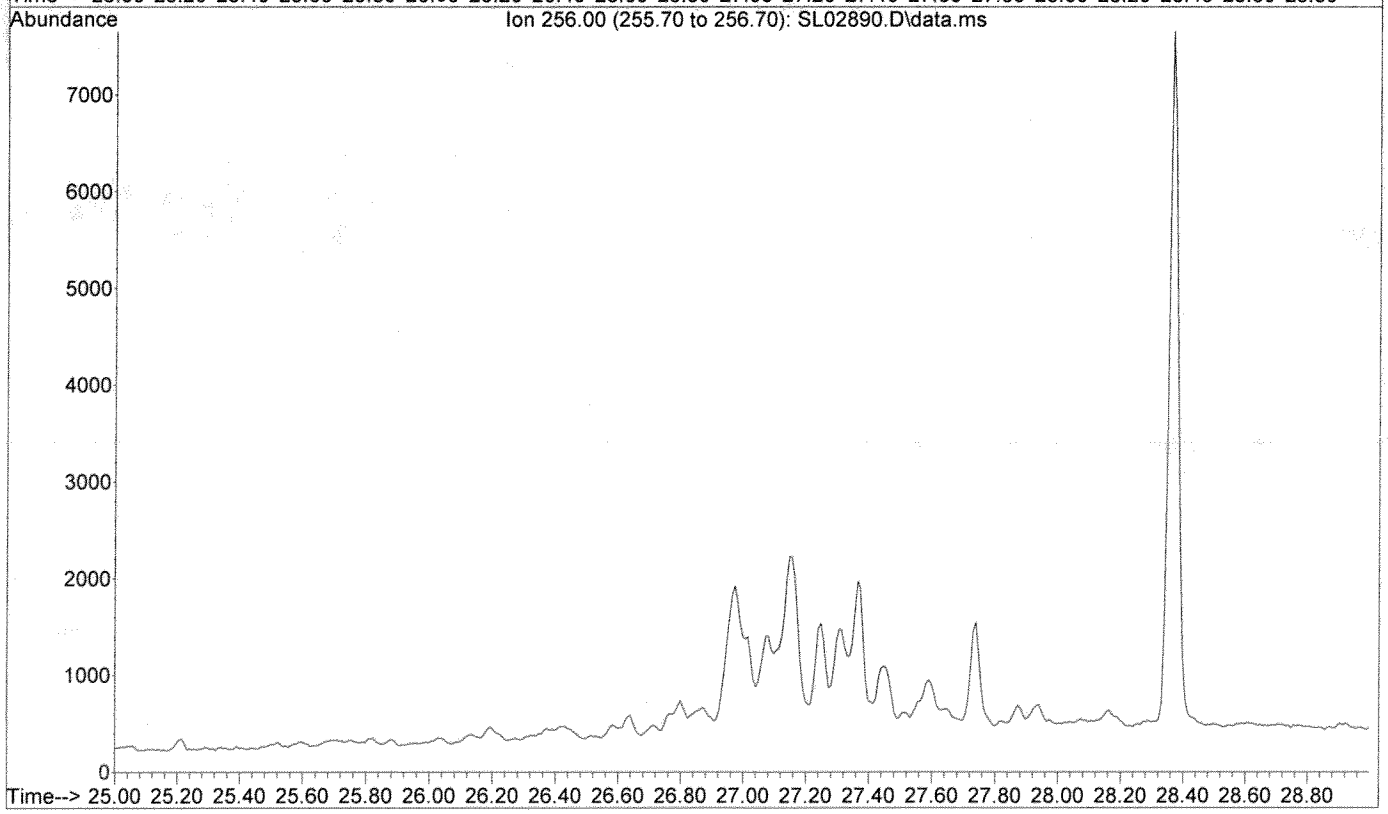
```
File        :C:\msdchem\1\DATA\030112\SL02890.D
Operator    : Syslo
Acquired    :  1 Mar 2012  20:37     using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0005
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6
```



US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6





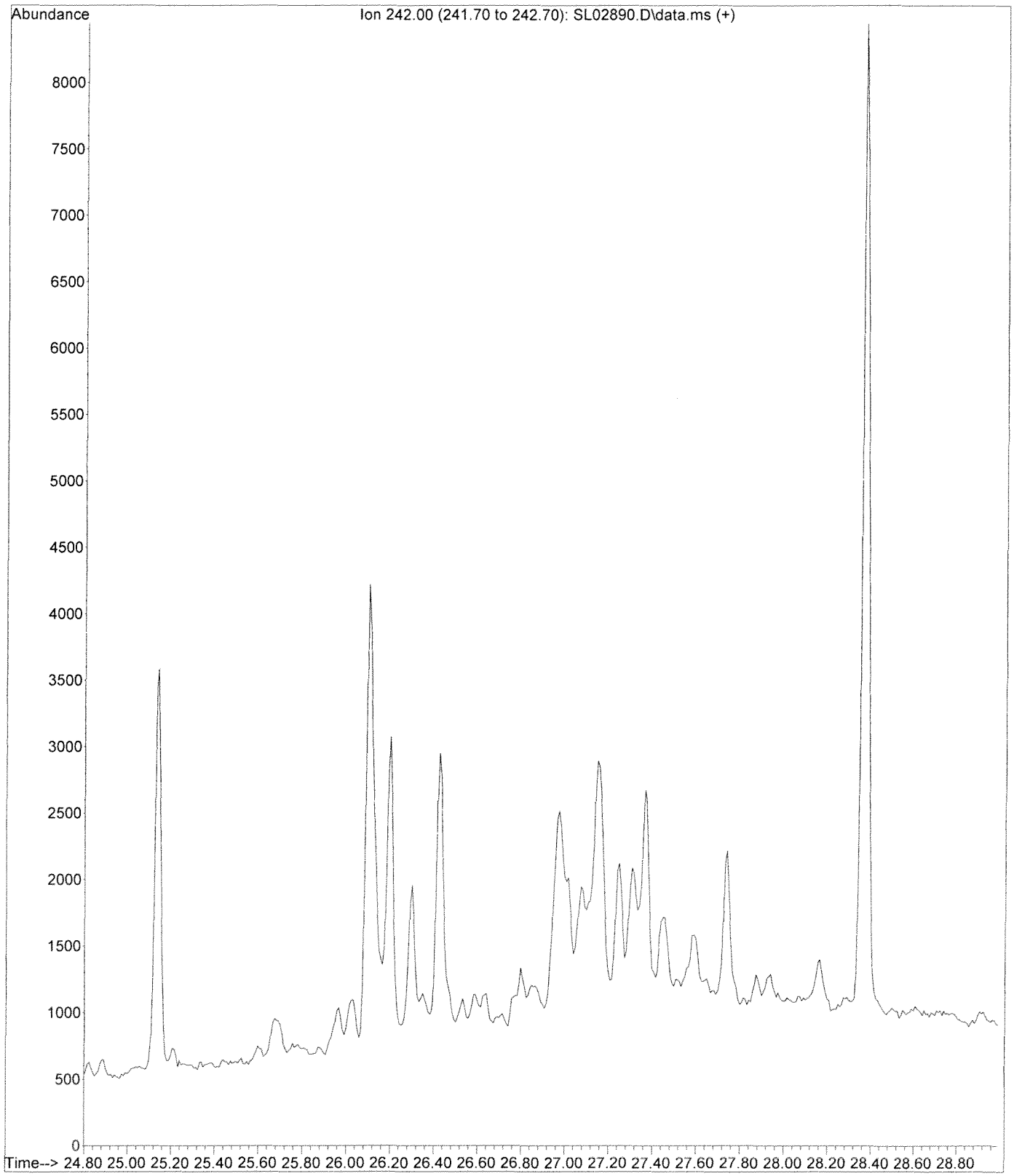
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02890.D
Operator : Syslo
Acquired : 1 Mar 2012 20:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20
Vial Number: 6



US EPA ARCHIVE DOCUMENT

SERAS-017 1085 Dup
SL02891.D

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02891.D
Acq On : 1 Mar 2012 21:37
Operator : Syslo
Sample : SERAS-017-0005 DUP
Misc : WCS-6B-072223-092910-JPS-KA-002-20 dup
ALS Vial : 7 Sample Multiplier: 1

Quant Time: Mar 01 22:27:47 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	322263	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	94462	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	129000	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	116518	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.822	188	510366	14.89	ug/mL	0.00
Spiked Amount 20.000			Recovery =	74.45%		
3) d14-o-Terphenyl {S}	19.733	244	240252	15.95	ug/mL	0.00
Spiked Amount 20.000			Recovery =	79.75%		
4) 5a-Androstane {S}	21.140	260	31965	17.44	ug/mL	0.00
Spiked Amount 20.000			Recovery =	87.20%		
7) d62-Triacontane {S}	28.156	66	219419	19.28	ug/mL	0.00
Spiked Amount 20.000			Recovery =	96.40%		
Target Compounds						Qvalue

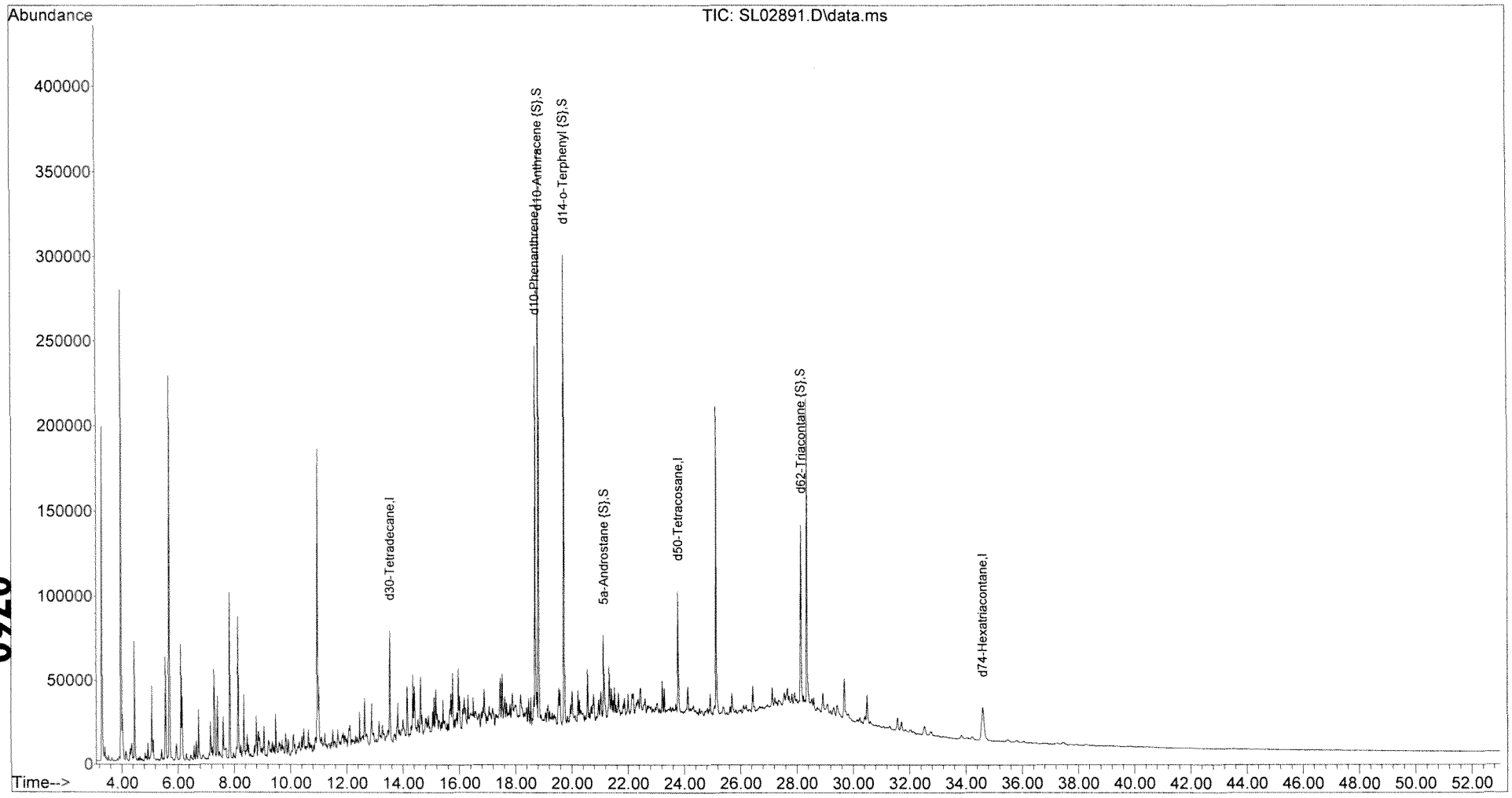
(#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02891.D
Acq On : 1 Mar 2012 21:37
Operator : Syslo
Sample : SERAS-017-0005 DUP
Misc : WCS-6B-072223-092910-JPS-KA-002-20 dup
ALS Vial : 7 Sample Multiplier: 1

Quant Time: Mar 01 22:27:47 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration



0760

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

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Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

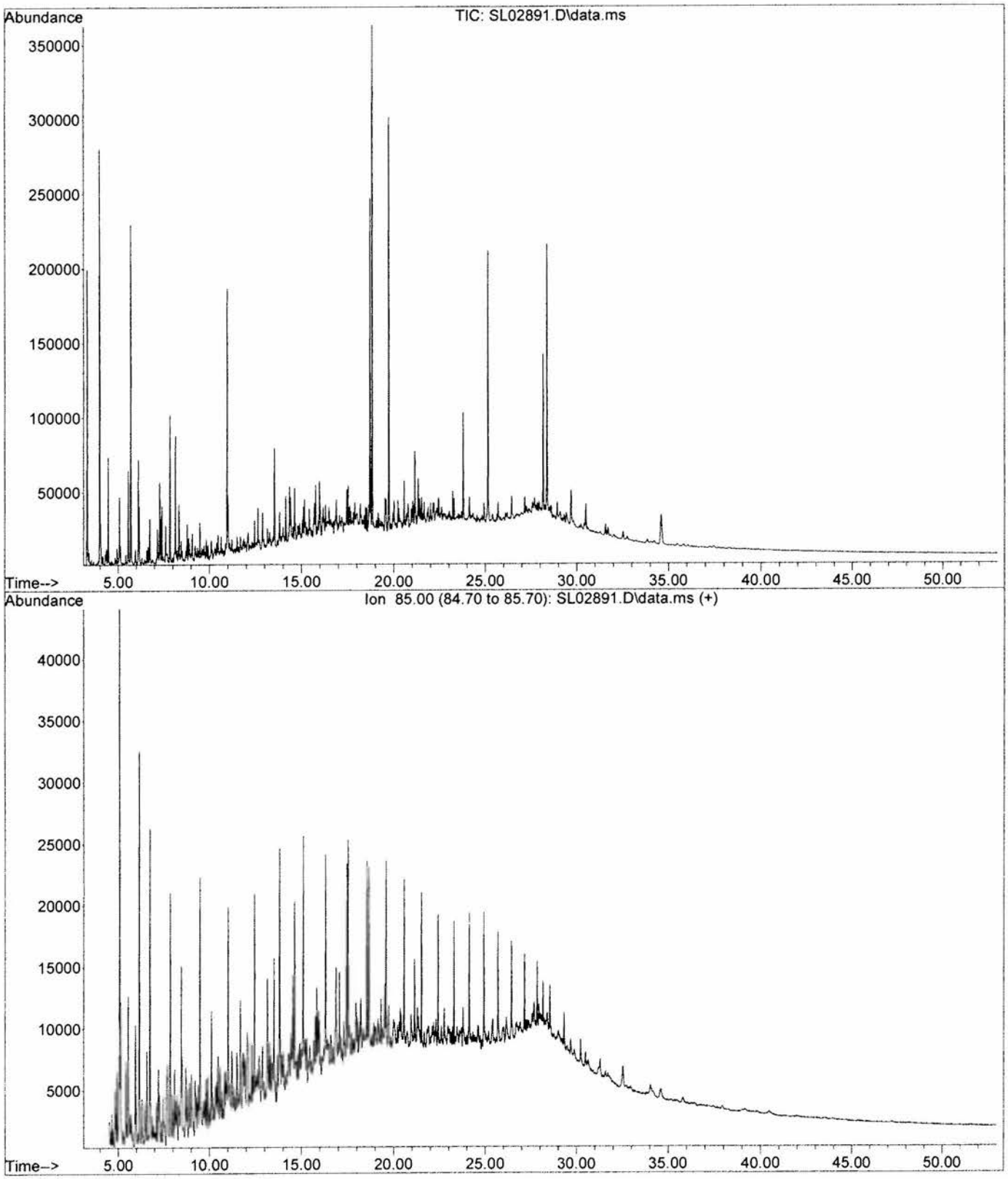
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```

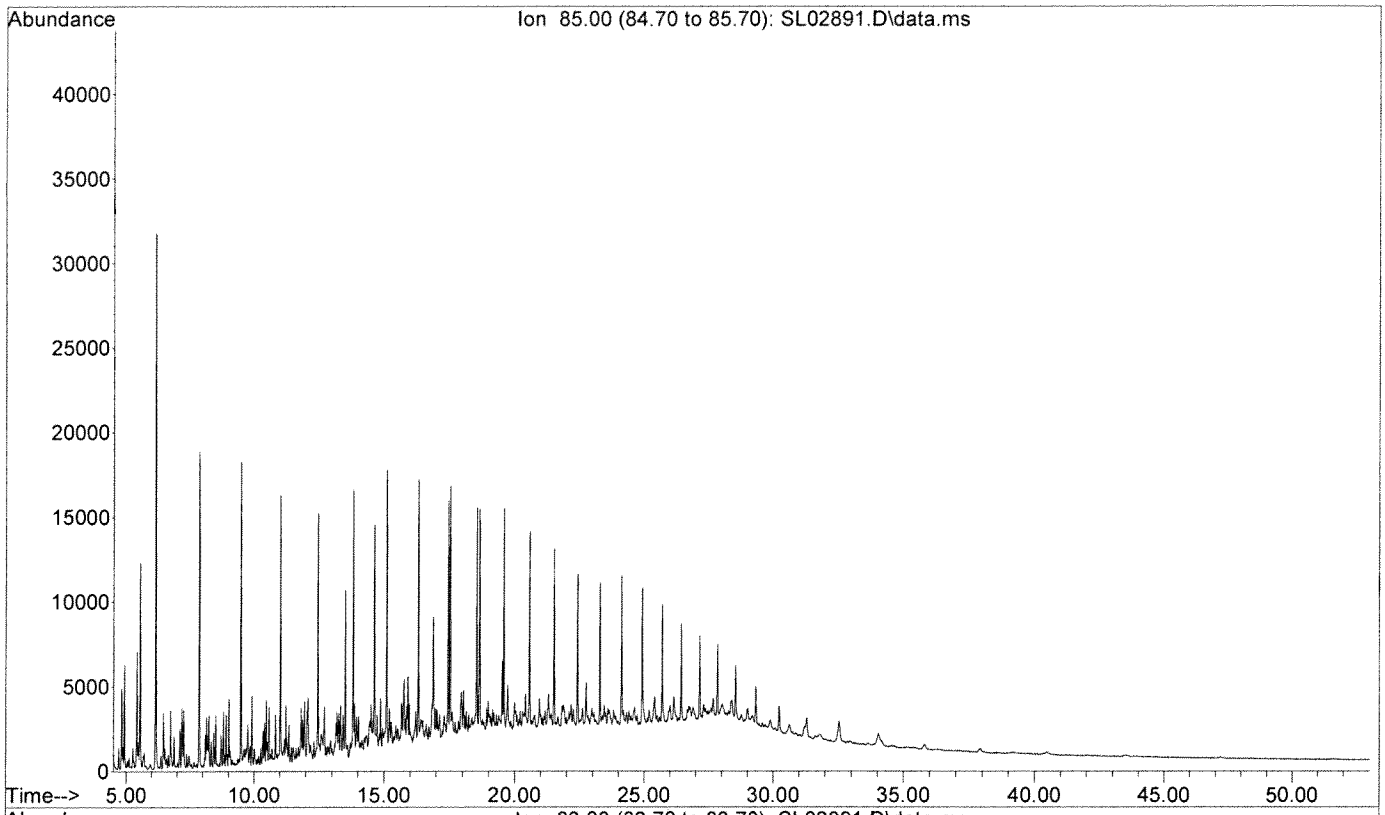


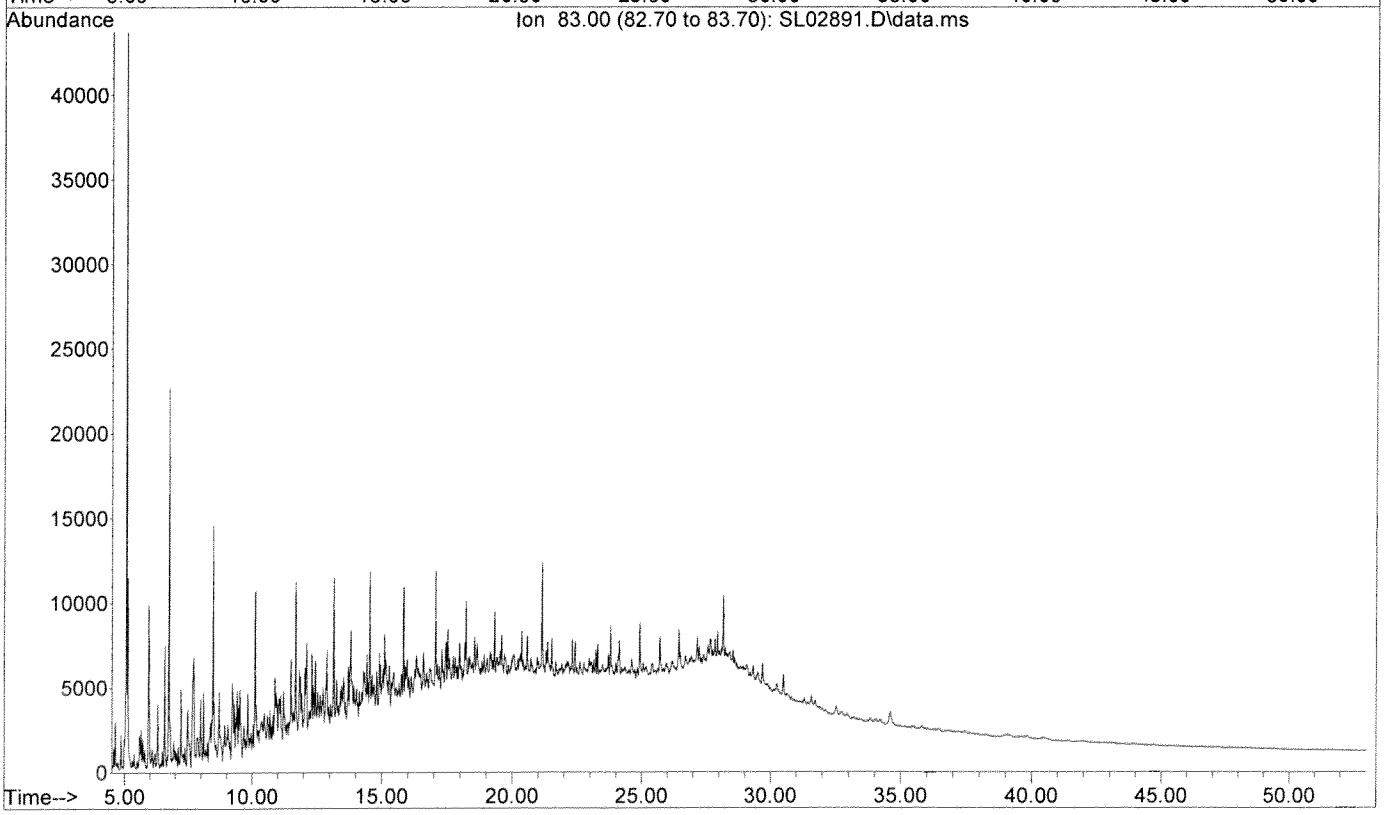
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```





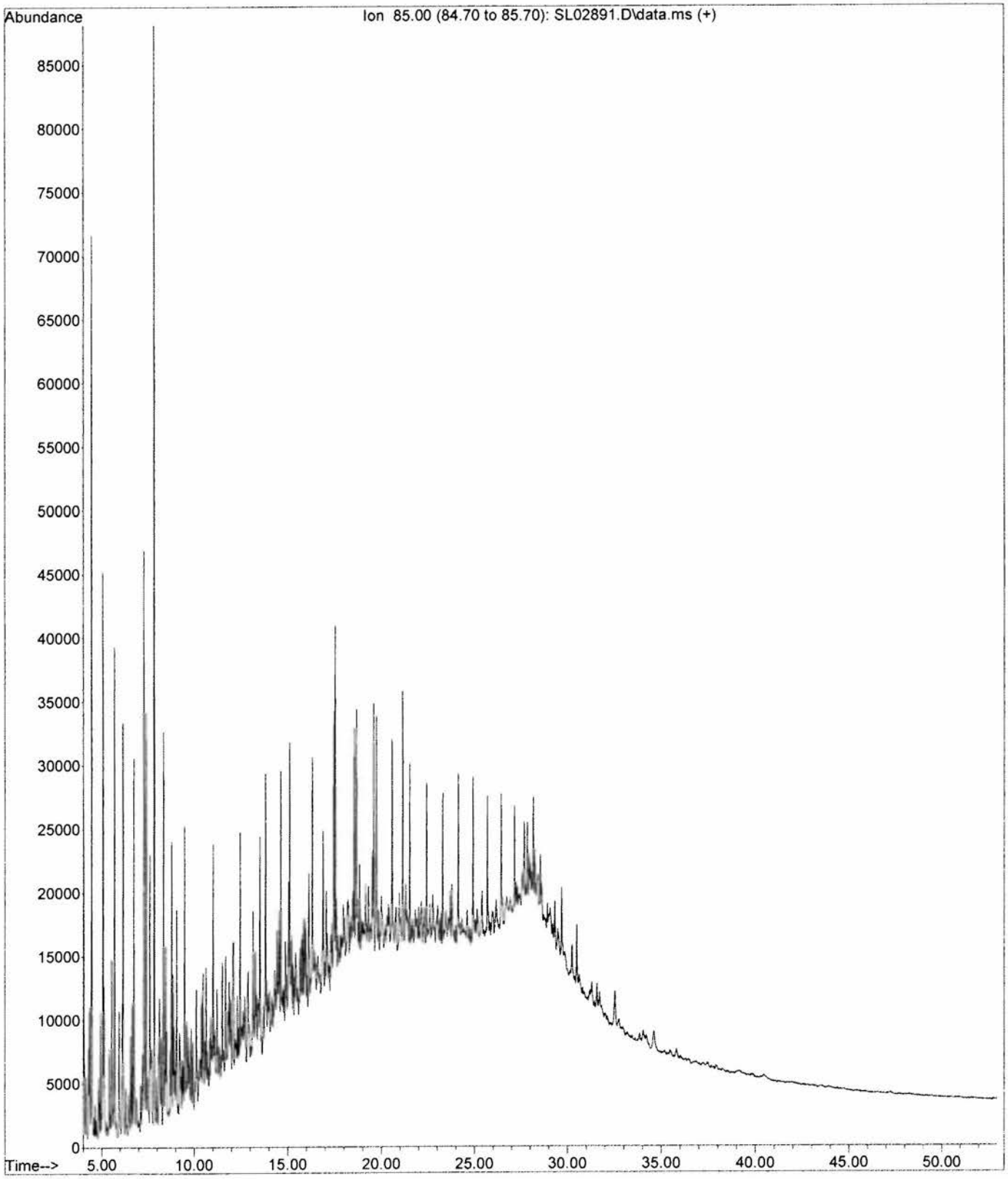
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02891.D
Operator : Syslo
Acquired : 1 Mar 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7



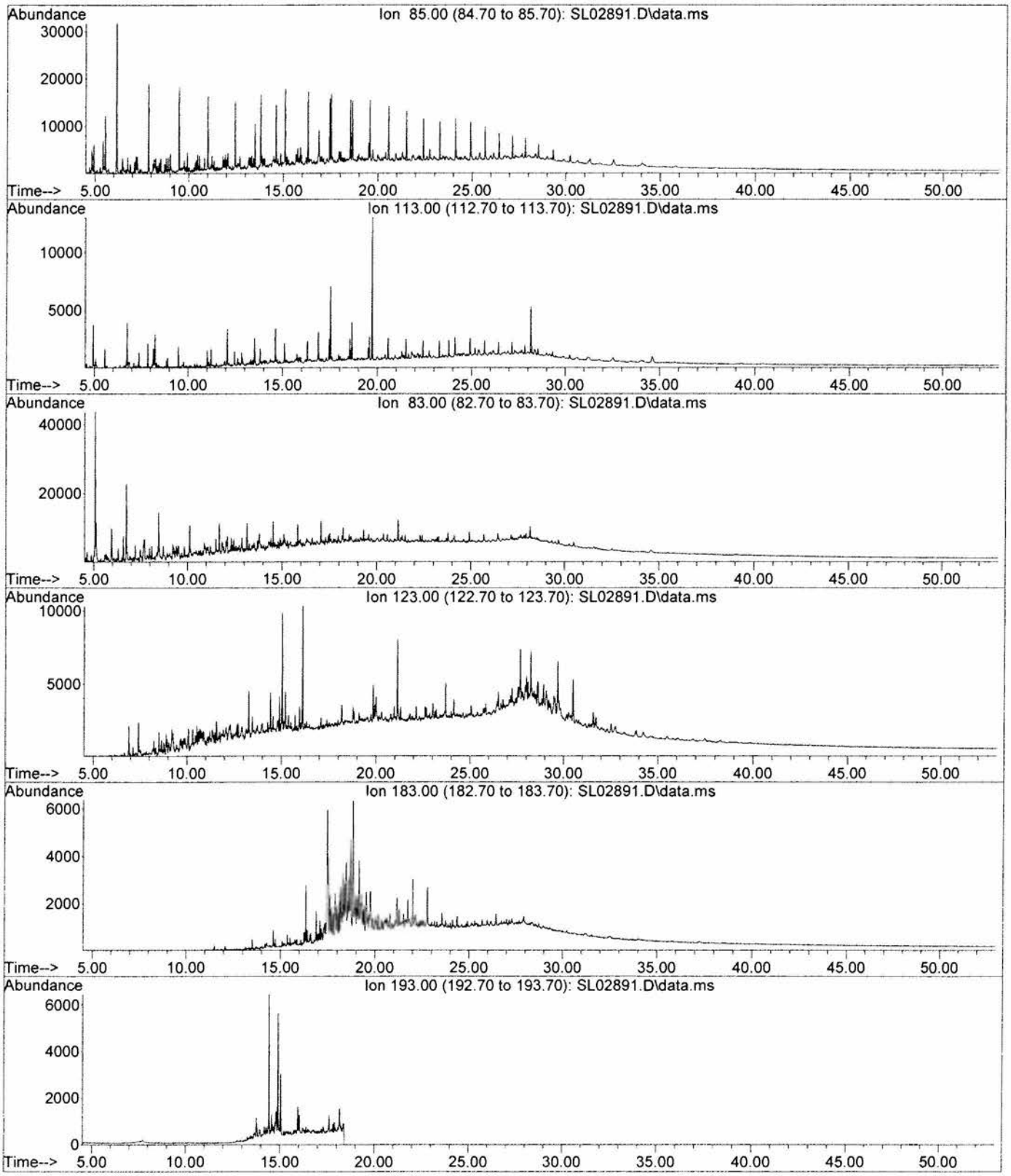
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



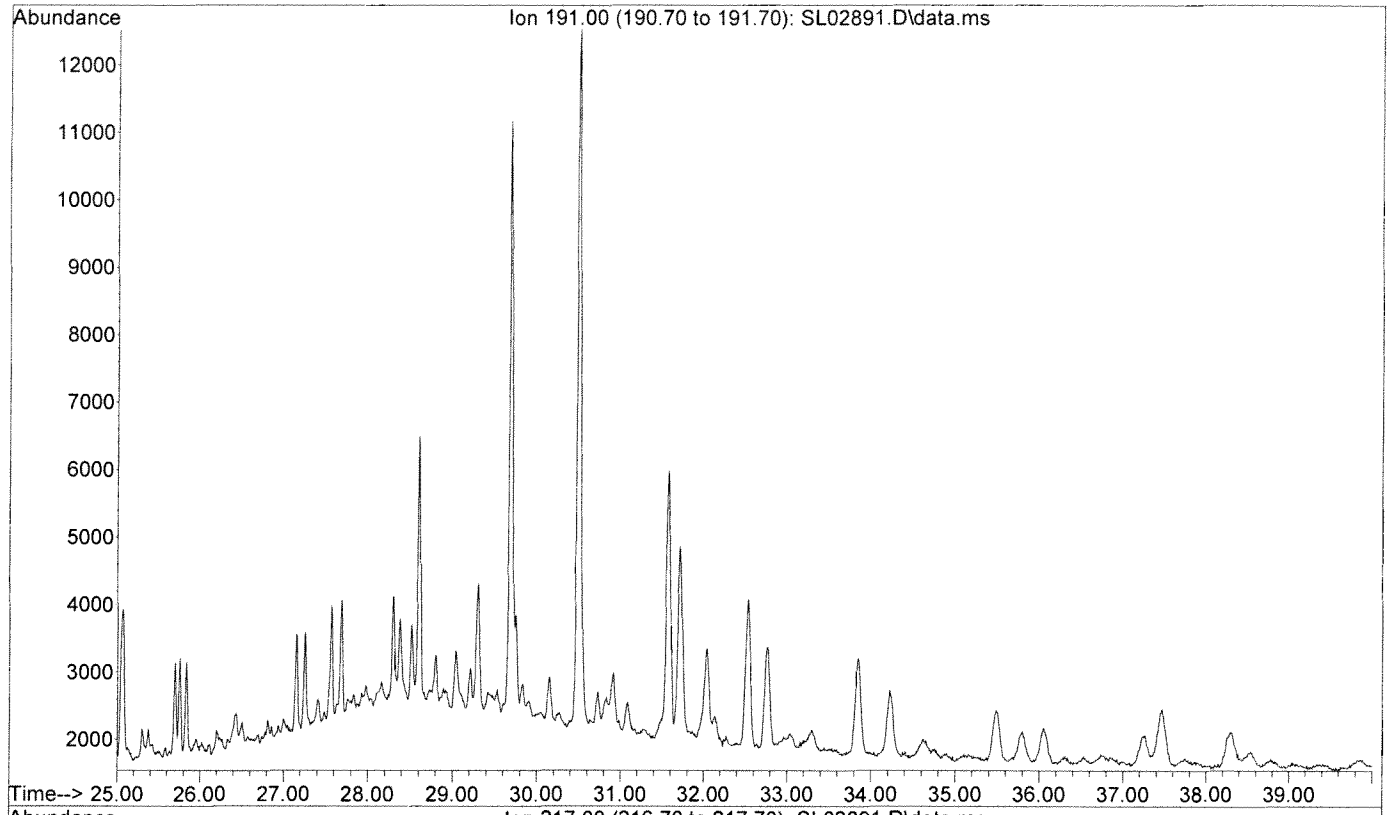
US EPA ARCHIVE DOCUMENT

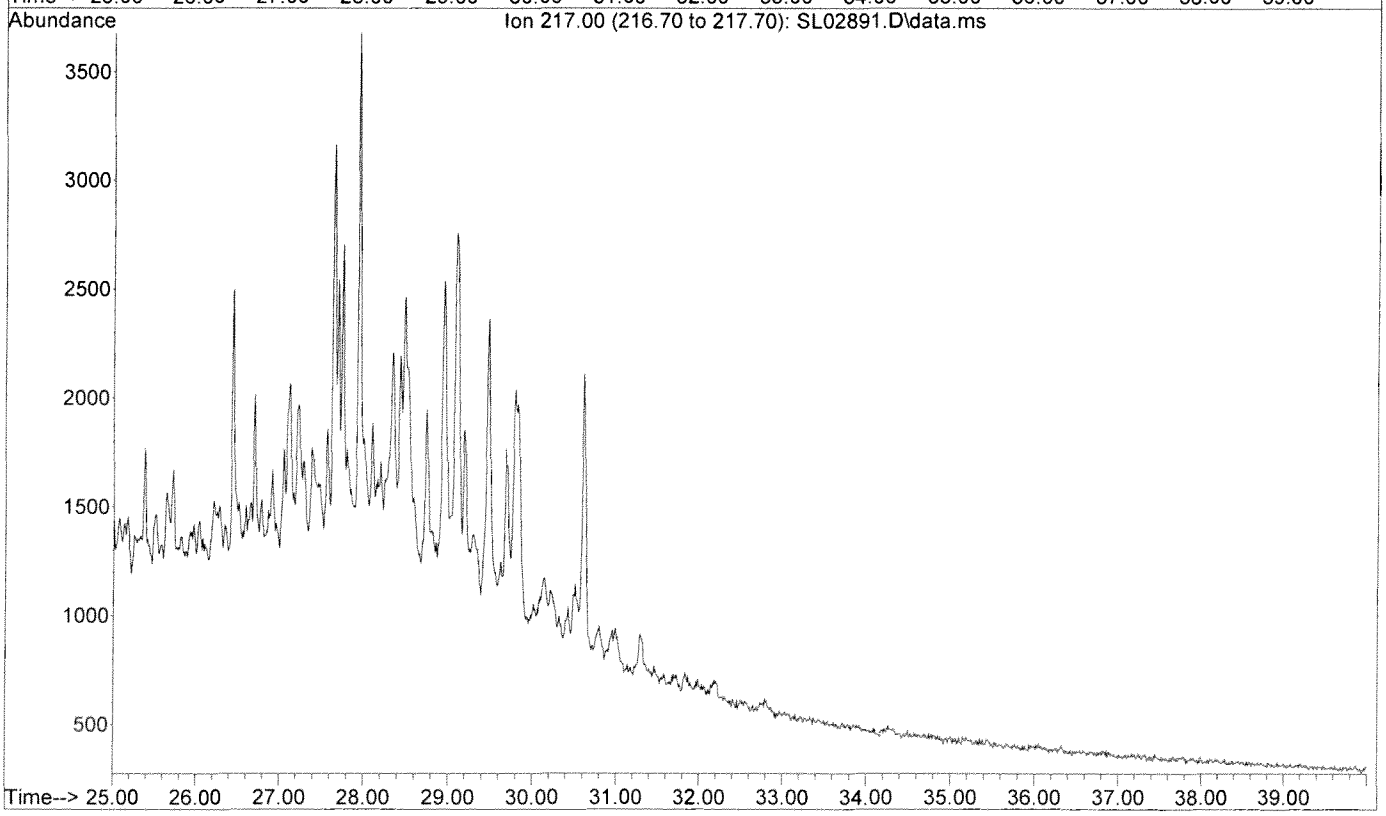
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```





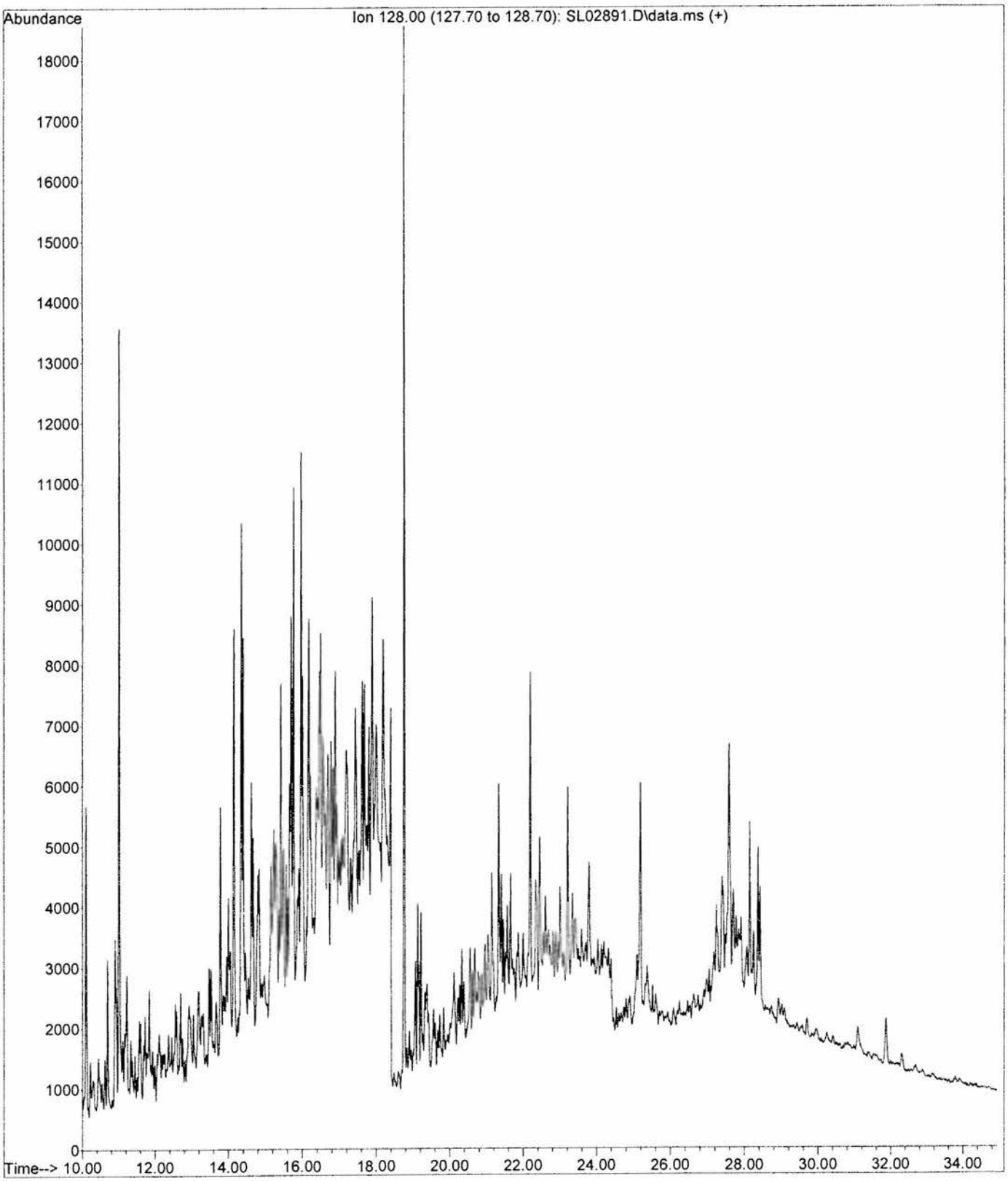
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



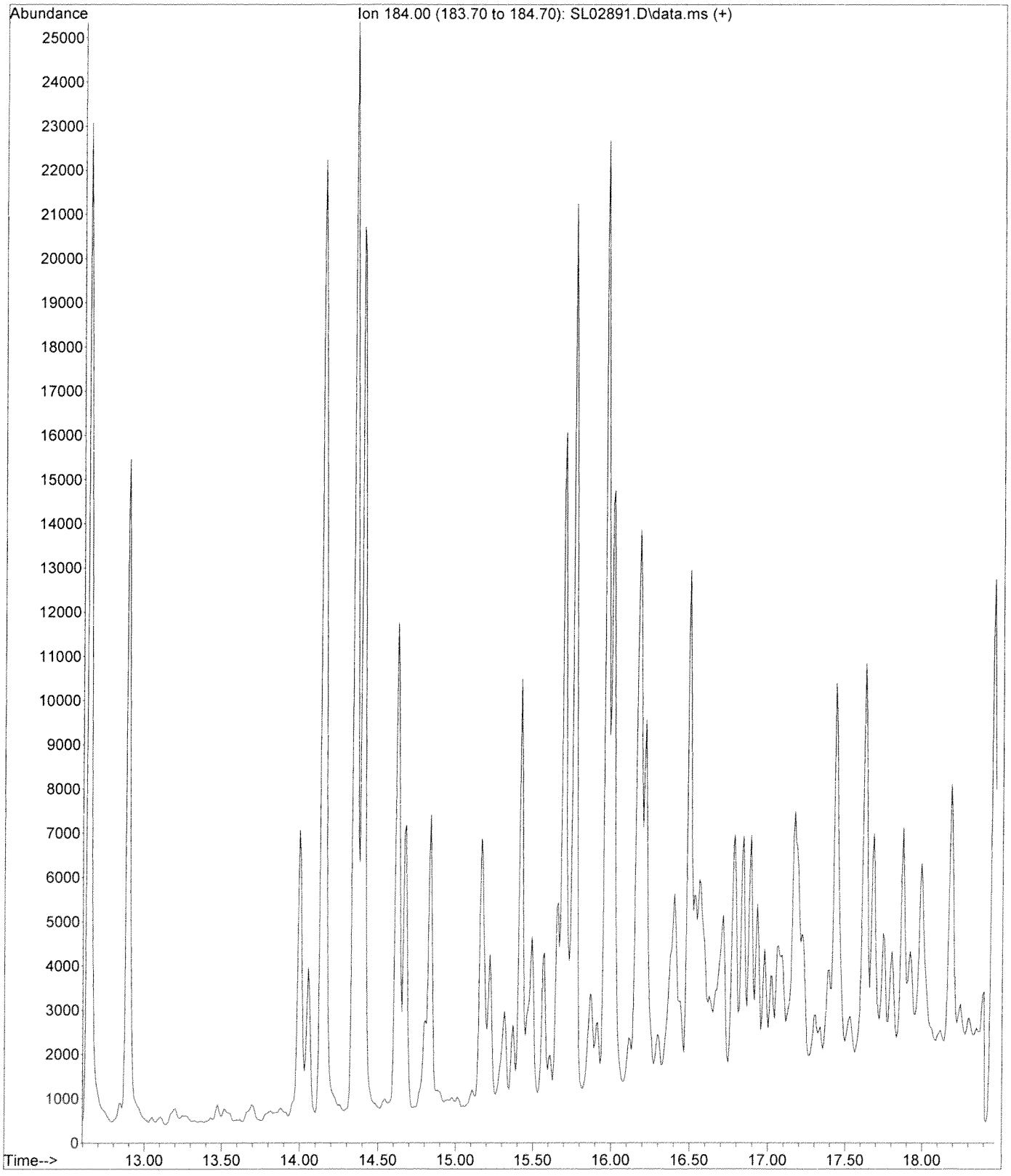
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
                macro "MCco.mac",go
                macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

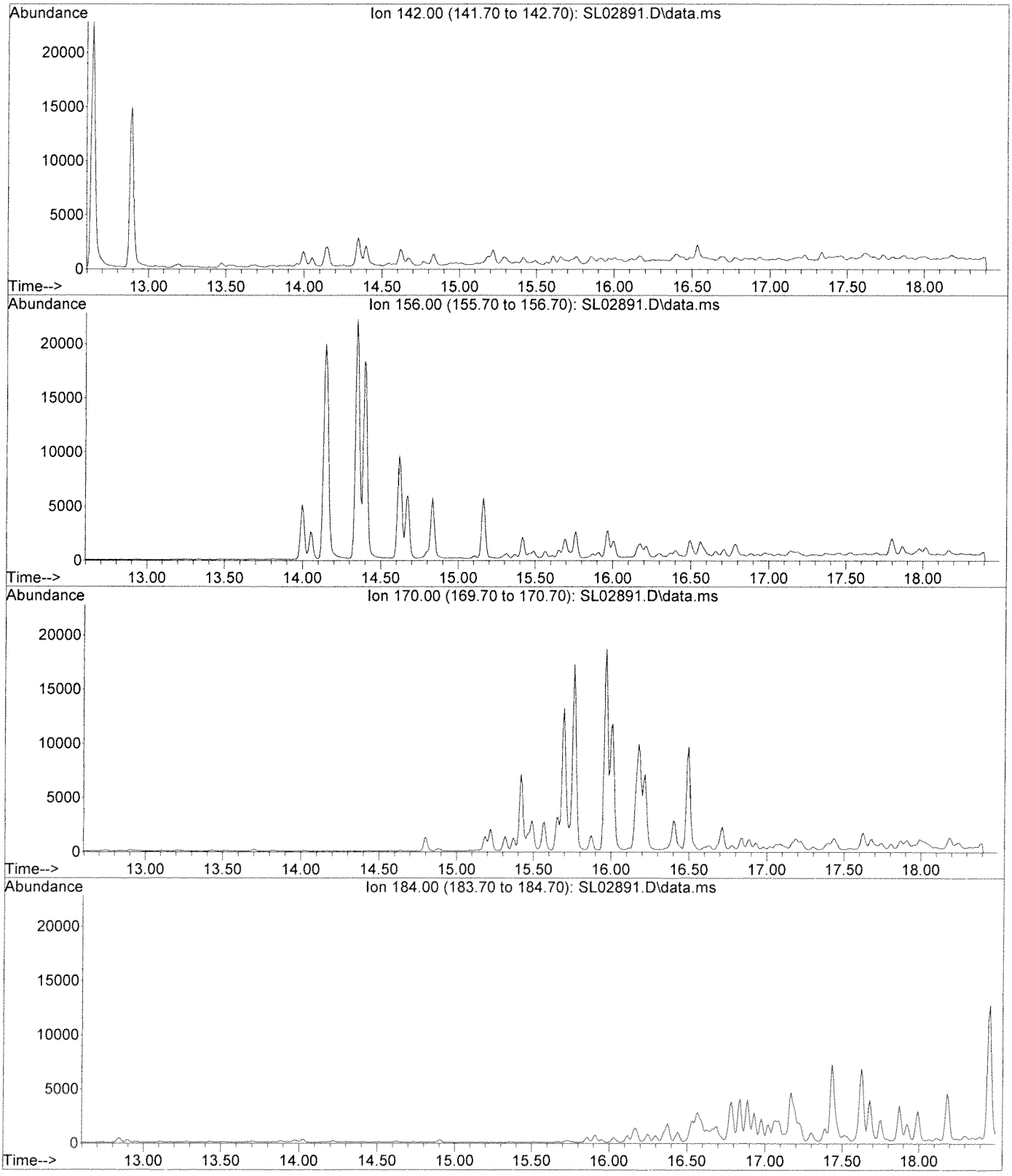
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



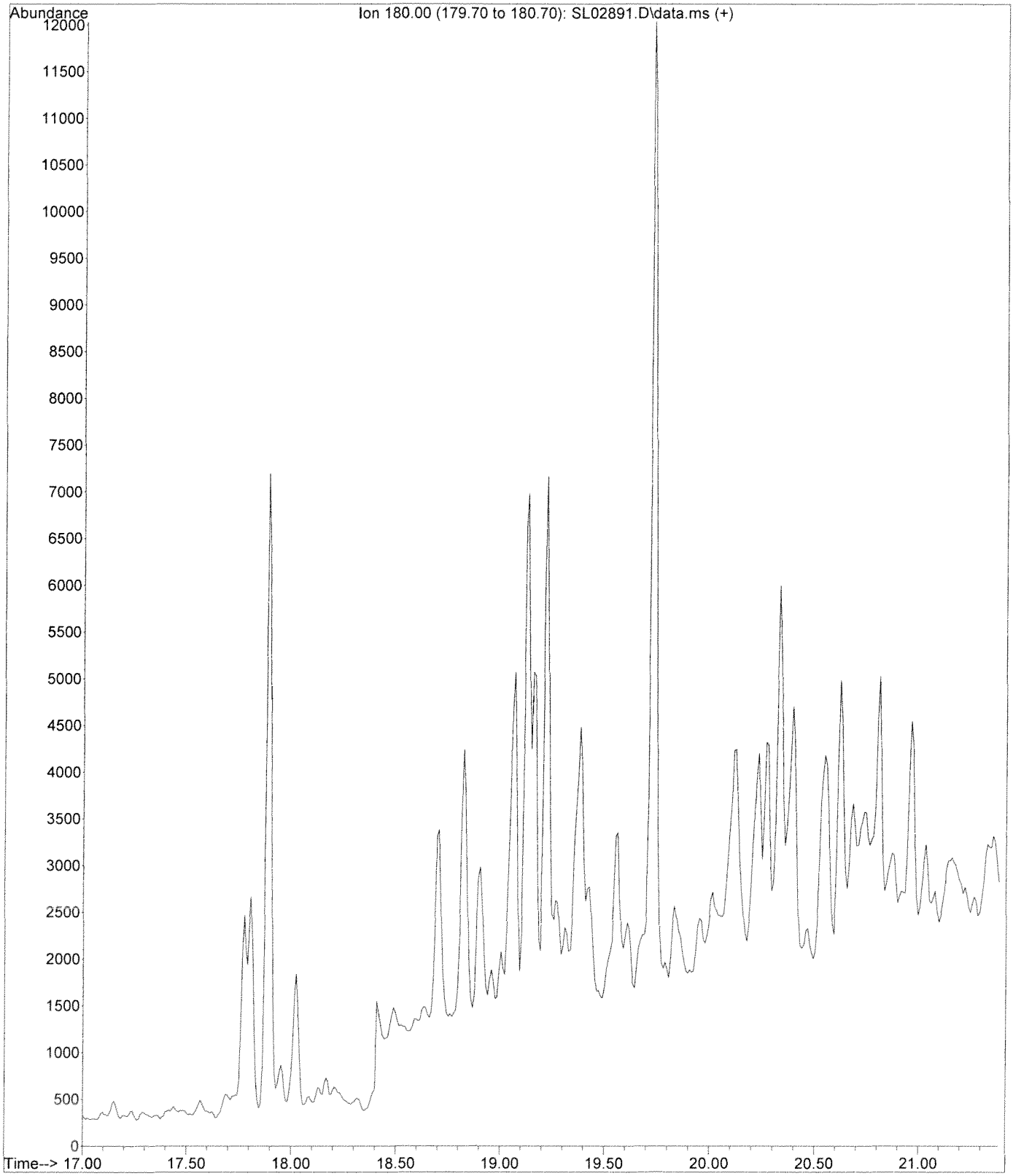
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



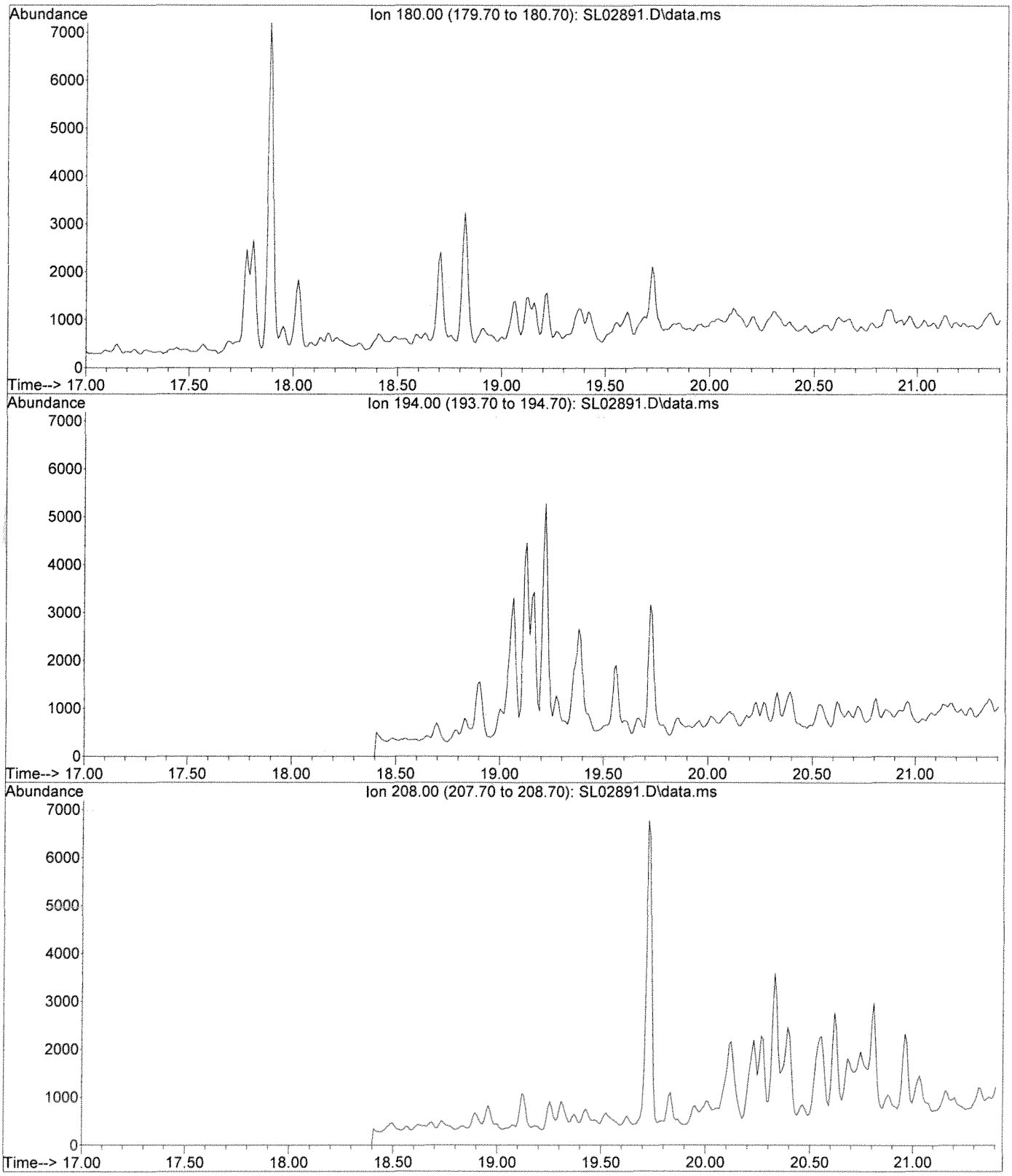
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02891.D
Operator : Syslo
Acquired : 1 Mar 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7



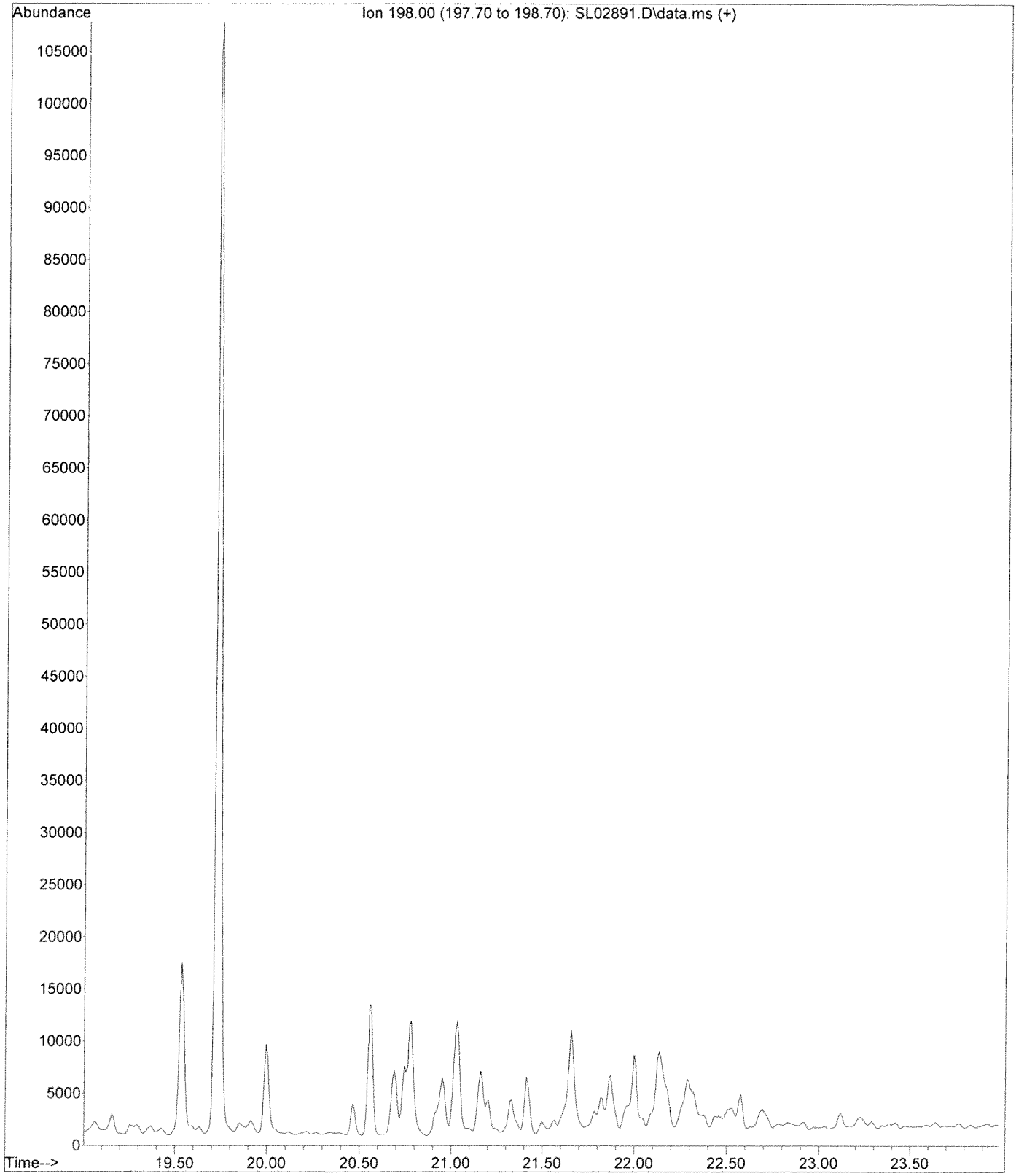
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



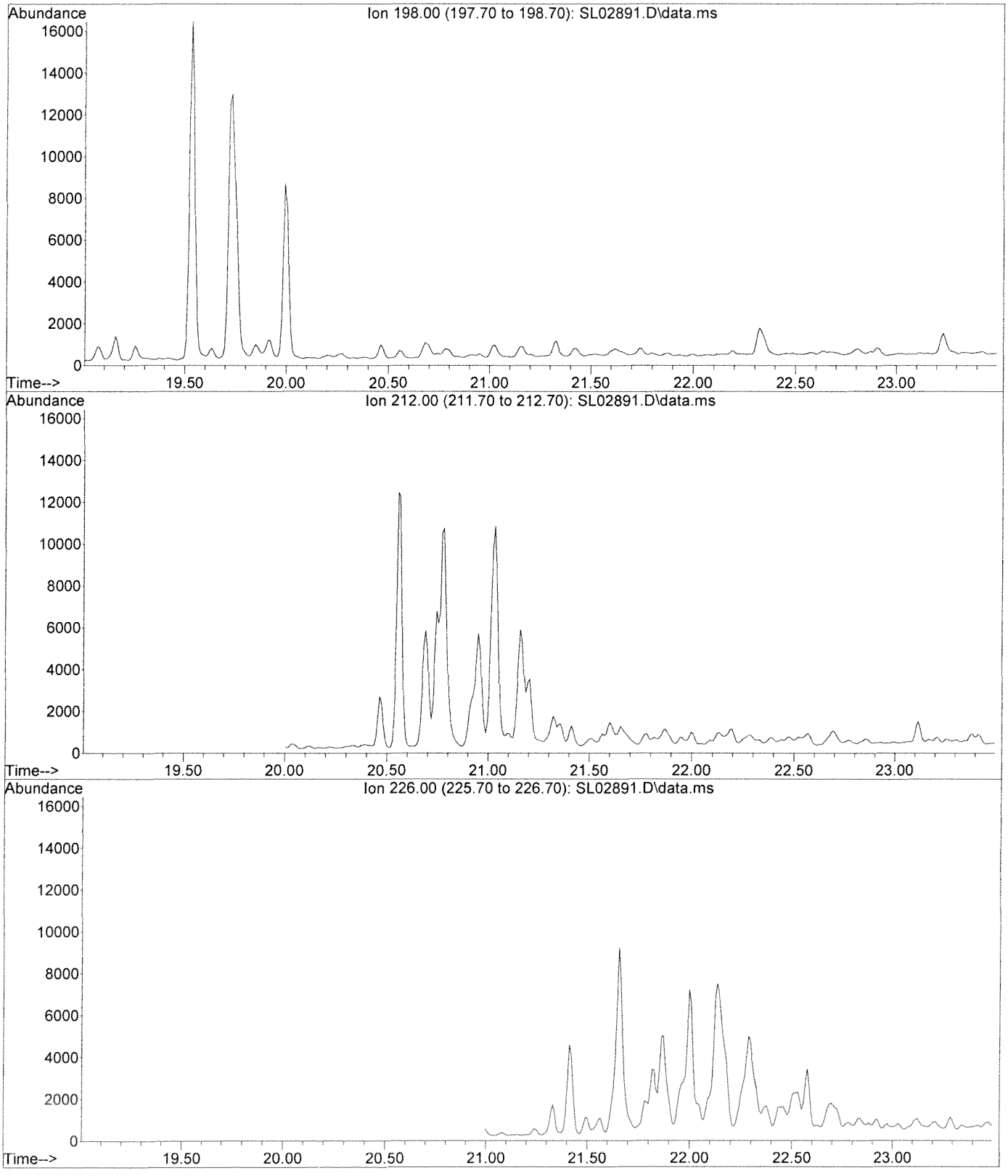
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02891.D
Operator : Syslo
Acquired : 1 Mar 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7



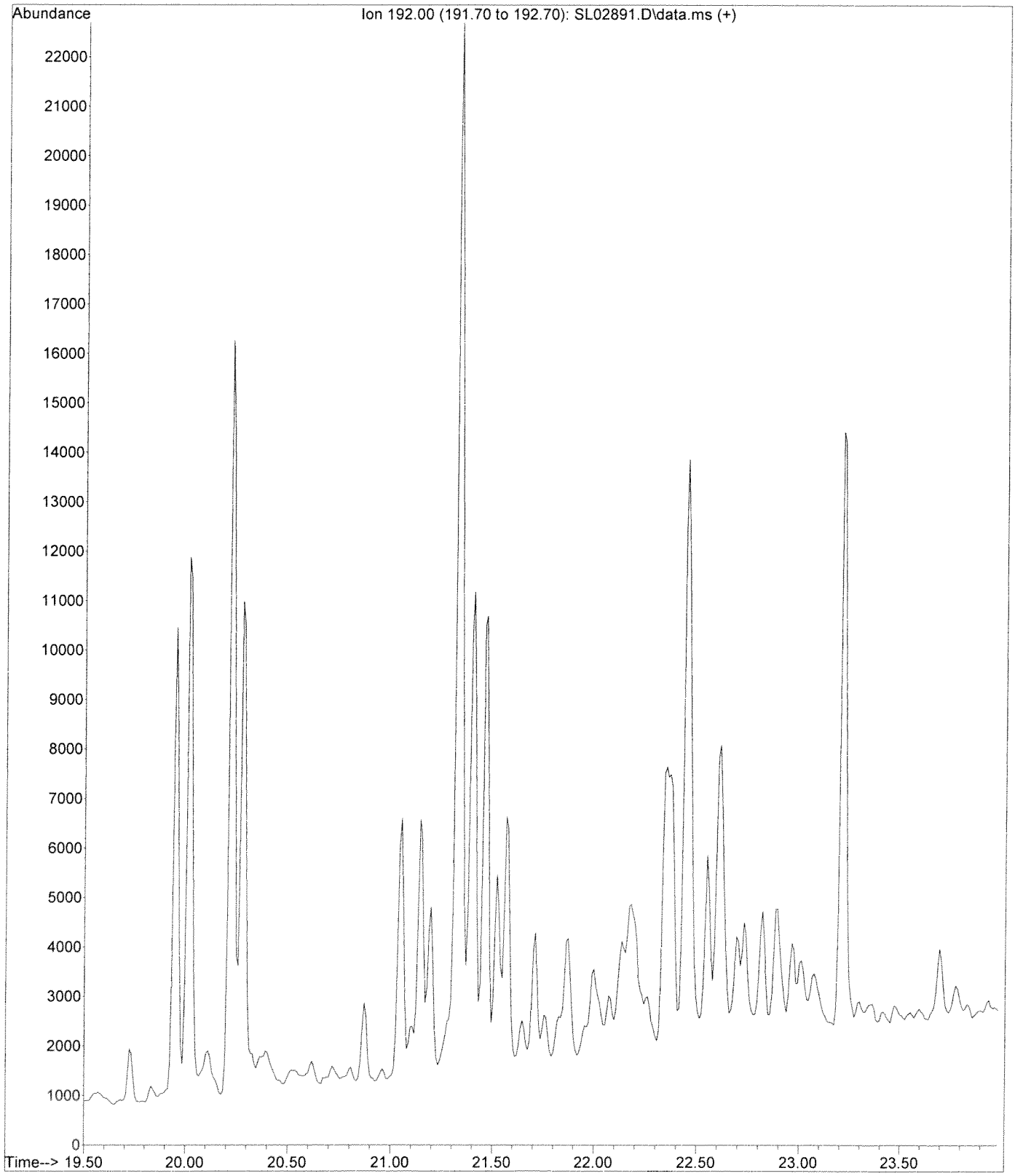
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37     using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



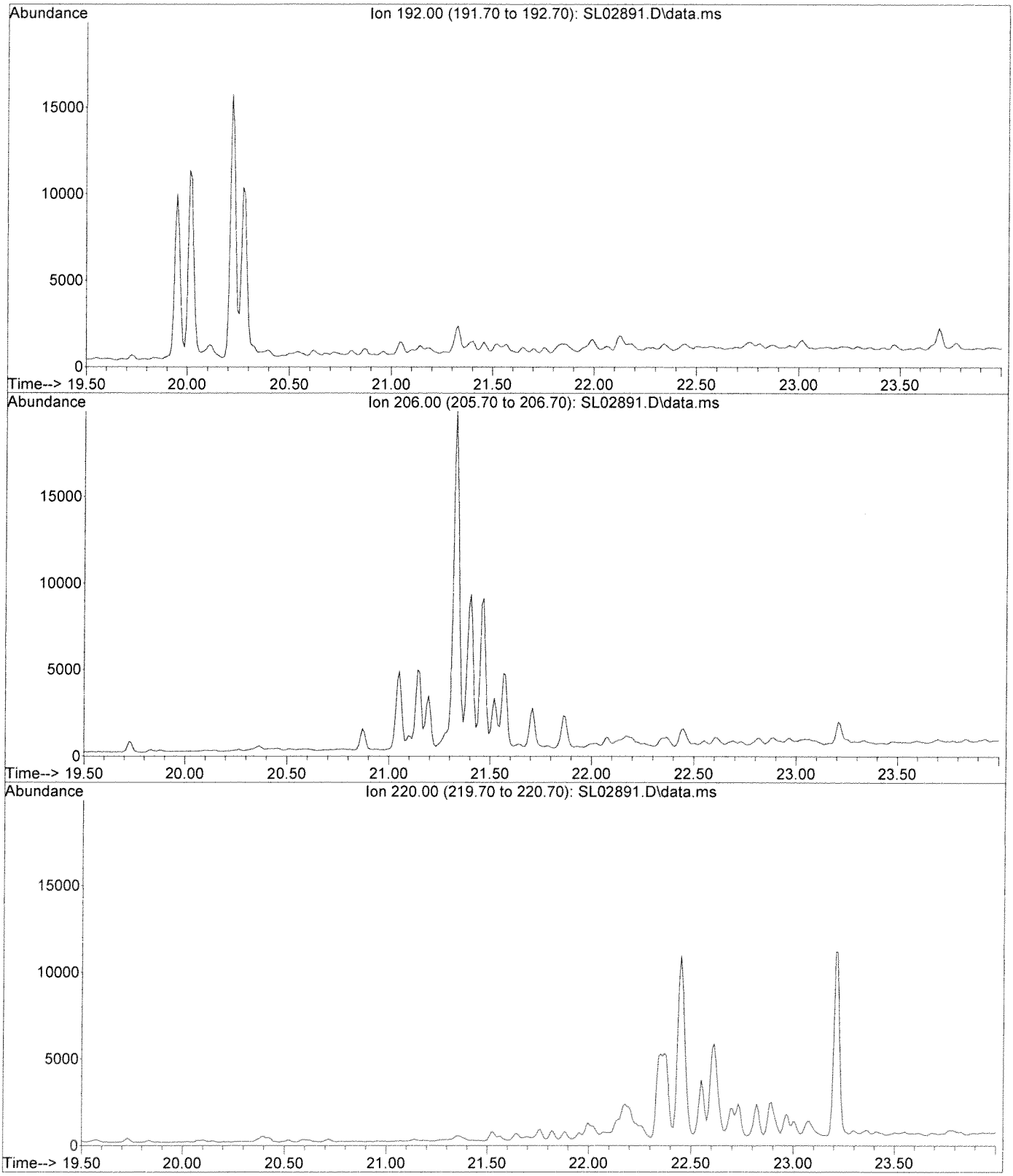
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



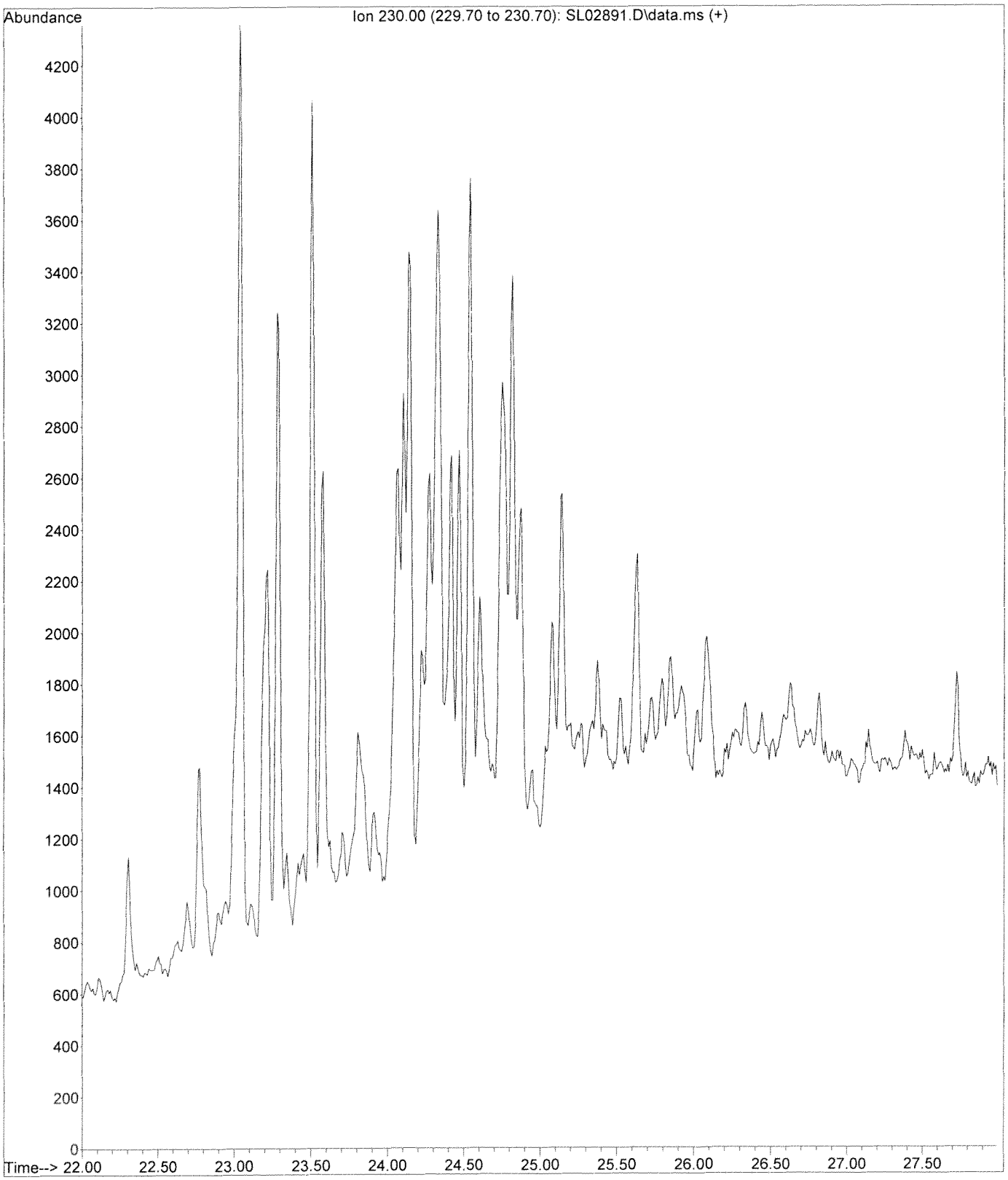
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



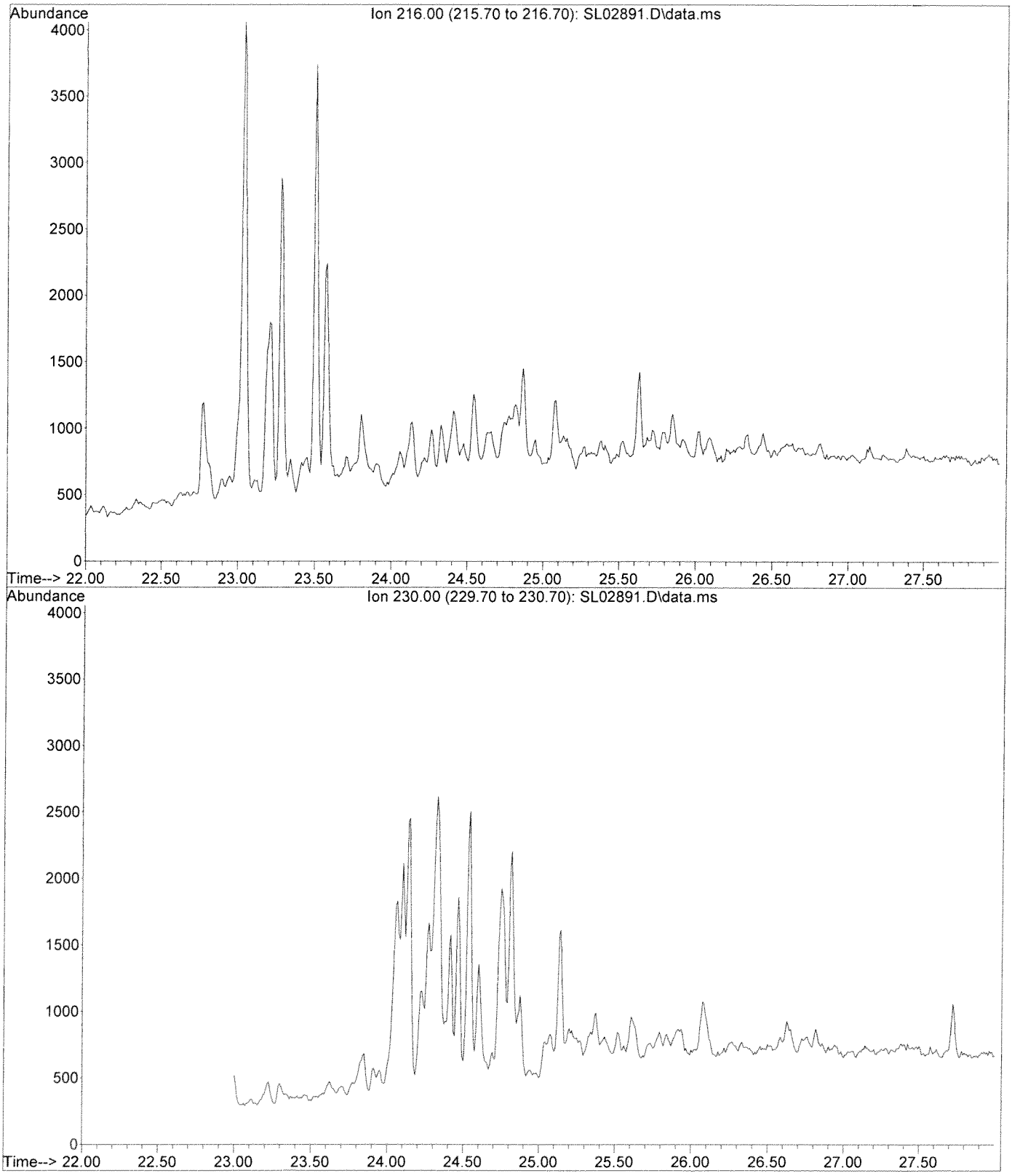
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



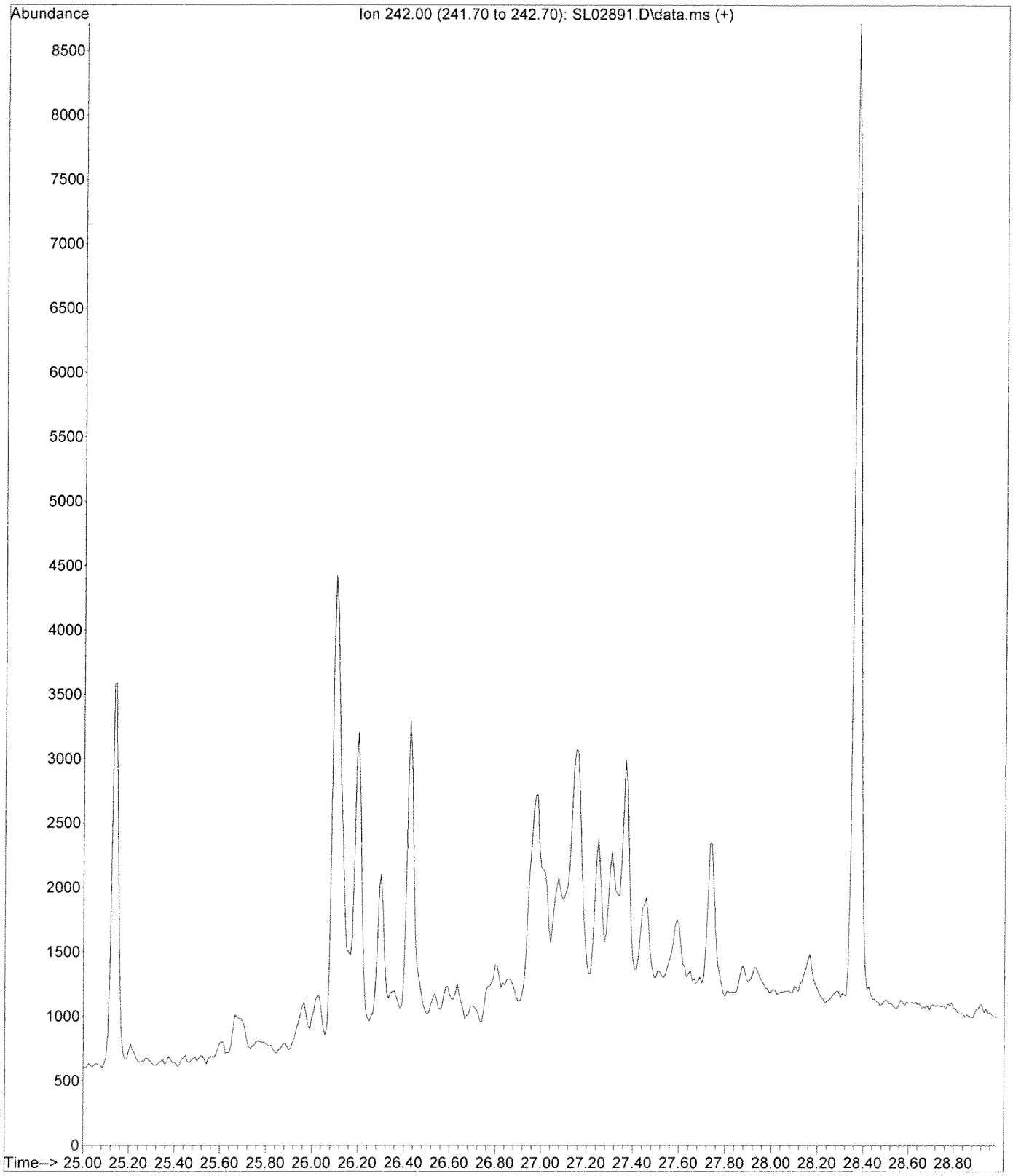
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02891.D
Operator : Syslo
Acquired : 1 Mar 2012 21:37 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7



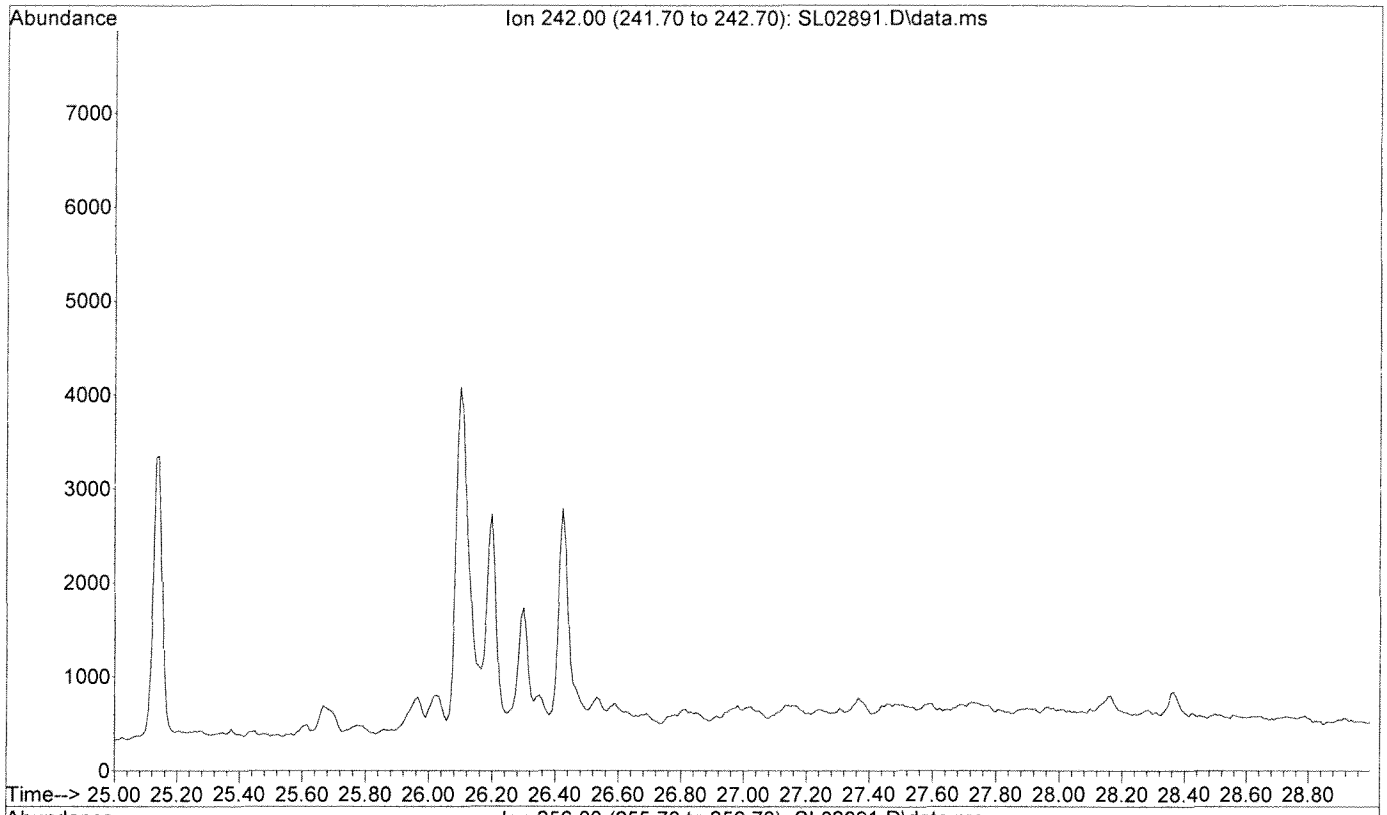
US EPA ARCHIVE DOCUMENT

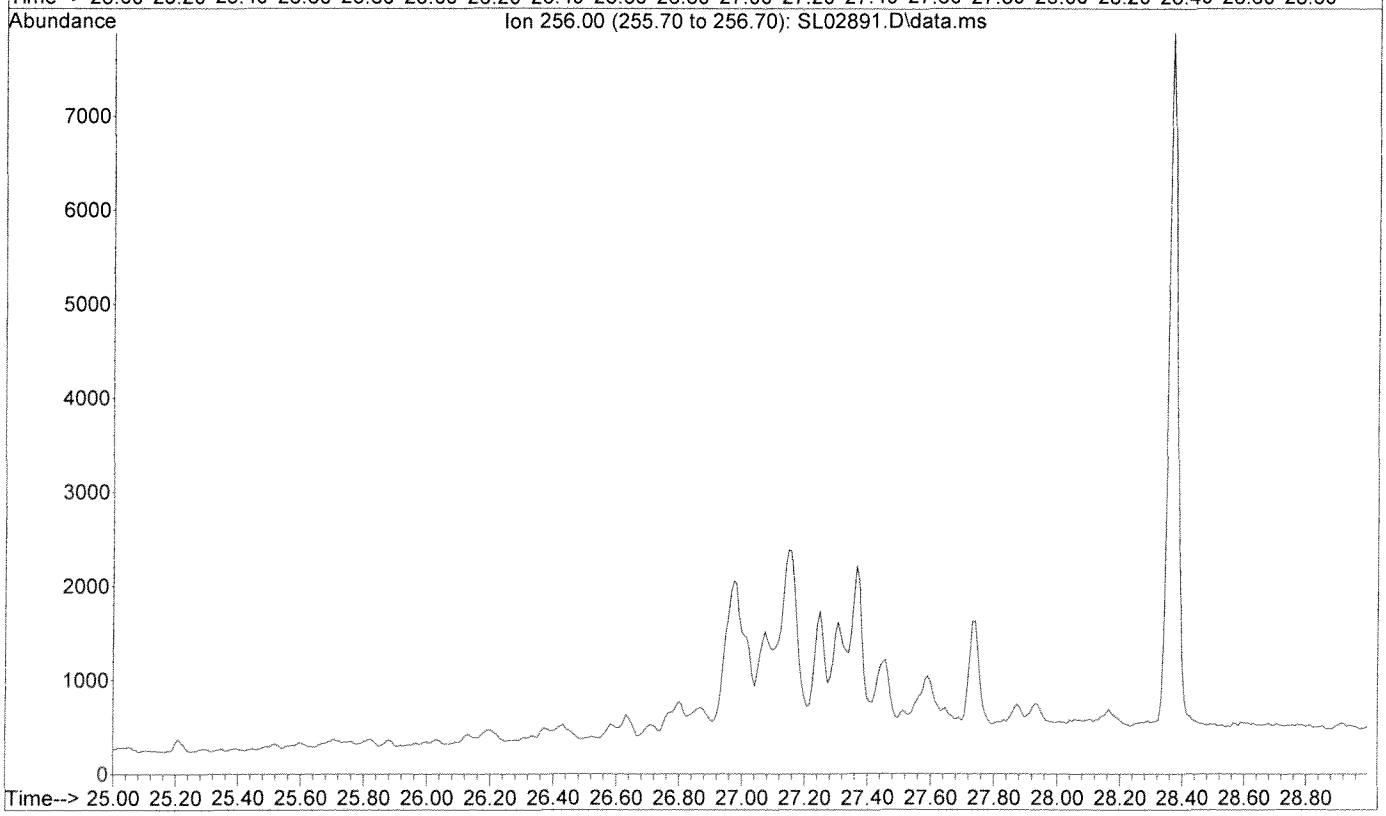
```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```





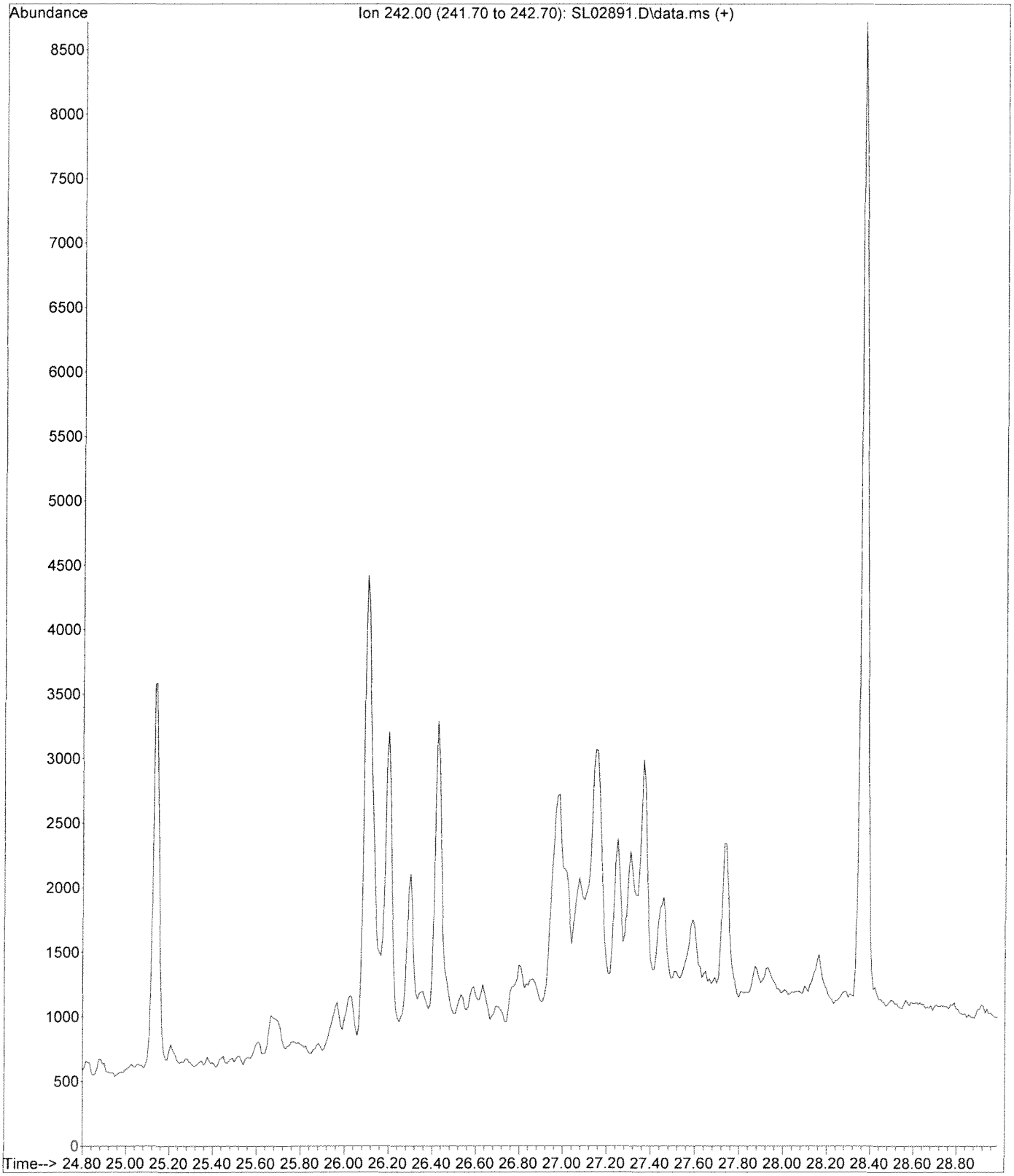
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02891.D
Operator    : Syslo
Acquired    :  1 Mar 2012  21:37     using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0005 DUP
Misc Info   : WCS-6B-072223-092910-JPS-KA-002-20 dup
Vial Number: 7
```



US EPA ARCHIVE DOCUMENT

SERAS-017-DTM

SL02892-D

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02892.D
Acq On : 1 Mar 2012 22:38
Operator : Syslo
Sample : SERAS-017-0006
Misc : CL-6B-072223-092710-JPS-KA-001-33
ALS Vial : 8 Sample Multiplier: 1

Quant Time: Mar 01 23:28:30 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.704	188	321005	10.00	ug/mL	0.00
5) d30-Tetradecane	13.532	66	94522	10.00	ug/mL	0.00
6) d50-Tetracosane	23.784	66	131524	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.595	66	117275	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	18.821	188	532149	15.59	ug/mL	0.00
Spiked Amount 20.000			Recovery = 77.95%			
3) d14-o-Terphenyl {S}	19.733	244	250024	16.67	ug/mL	0.00
Spiked Amount 20.000			Recovery = 83.35%			
4) 5a-Androstane {S}	21.140	260	33364	18.28	ug/mL	0.00
Spiked Amount 20.000			Recovery = 91.40%			
7) d62-Triacontane {S}	28.156	66	229799	19.78	ug/mL	0.00
Spiked Amount 20.000			Recovery = 98.90%			

Target Compounds Qvalue

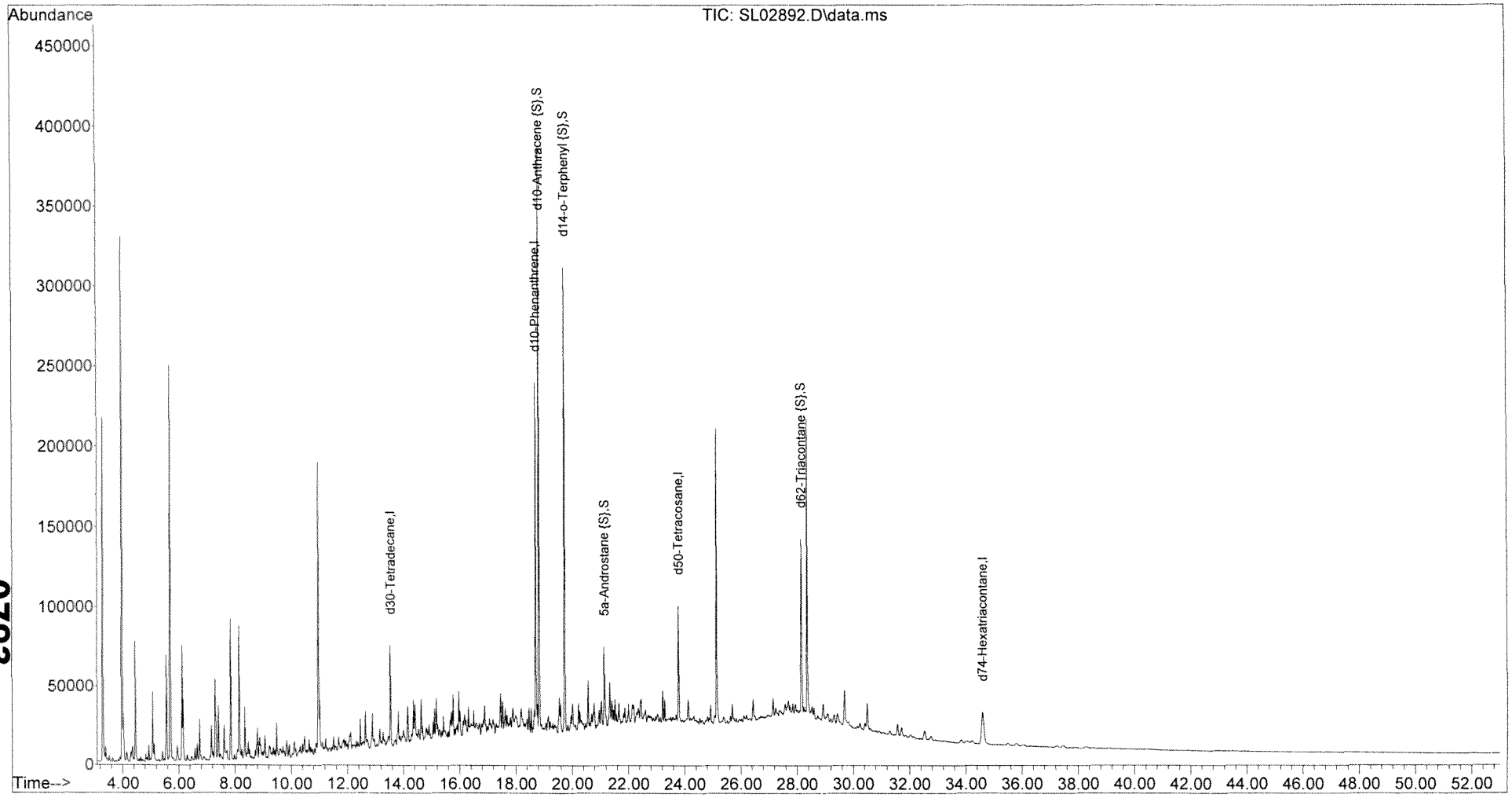
#) = qualifier out of range (m) = manual integration (+) = signals summed

SERAS-017-DTM-011413_3

Quantitation Report (Not Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02892.D
Acq On : 1 Mar 2012 22:38
Operator : Syslo
Sample : SERAS-017-0006
Misc : CL-6B-072223-092710-JPS-KA-001-33
ALS Vial : 8 Sample Multiplier: 1

Quant Time: Mar 01 23:28:30 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration



0793

```
! File  : ENOfingFinal.MAC
! J. Syslo MACRO for printing select fingerprints for Enbridge Oil, 01/09/2012

name ENOfingFinal
        macro "TICTPH.mac",go
        remove TICTPH
        macro "TPH8583Stacked.mac",go
        remove TPH8583Stacked
                        macro "TPHSERAS.mac",go
                                remove TPHSERAS
                        macro "ENOTPHSEPN.mac",go
                                remove ENOTHPSEPN
                        macro "HOPSTER.mac",go
                                remove HOPSTER
                        macro "ParentPAH.mac",go
                                remove PARENTPAH
                        macro "PAH1803N.mac",go
                        remove pah1803n
        macro "McDet.mac",go
        remove McDet
TIC
draw 2,x
remove ENOfingFinal
clear
return
```

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

Macros used to generate fingerprint chromatograms for the Enbridge Oil samples.

SERAS draft GC/MS SOP 1803 allows analytical characterizations and evaluation of petroleum oils for comparison purposes. Certain classes of source specific chemical compounds are targeted in this qualitative comparison; these target compounds are both unique descriptors of an oil and chemically resistant to environmental degradation. Spilled oil can be assessed in this way as being similar or different from potential source samples by direct visual comparison of specific extracted ion chromatograms (EIC).

The SERAS method implements several unique techniques to present and assess the "fingerprints" that are far superior to comparing just individual fingerprints and determining whether fingerprints are similar or not. The method utilizes several different *Visual Diagnostic Ratios* (VDR) where two or more different compound class fingerprints are presented together "stacked" on top of each other, where the fingerprints are usually not normalized. When presented in this manner, the total fingerprint match can be assessed by comparing the peak to peak ratios as well as the total intensity (based on concentration of those compounds) of the fingerprints relative to each other. These *Visual Diagnostic Ratios* are generated by the GC/MS data system using customized macros; the selection of ions/compounds are usually dictated or driven by project specific conditions, or by the nature or identity of the spilled oil.

Data Presentation & Direct Visual Comparison of Enbridge Oil Fingerprints (EIC): SERAS will generate only the relevant and appropriate selected EIC necessary to identify, differentiate, or supply the conclusive evidence for the oil samples and reference oils. The number of possible fingerprints that can be presented using either the single classes of compounds, or by various combinations of "stacked" fingerprints is almost unlimited and not practical. The EIC of the suspected source oils were compared to appropriate EIC of the spilled oil. The following macros were written to process, extract and present key fingerprints and "stacked" fingerprints to evaluate and compare the key VDR of the Enbridge Oil samples.

The following macros were used to produce the conclusive VDR that were used to evaluate the oil fingerprints:

- **ENOfingFinal.mac:** This macro ran the following 8 nested macros to produce VDR and fingerprints:
  - **TICTPH.mac:** Illustrates the VDR comparing the total ion chromatogram (TIC) relative to the Total Petroleum Hydrocarbon (TPH) fingerprint generated by macro **TPHSERAS.mac**.
  - **TPH8583stacked.mac:** A simple useful VDR that compares the ion 85 over ion 83 TPH non normalized fingerprints. Note that the 85 ion represents the saturated hydrocarbons while the 85 ion is representative of the cyclo- and naphthenic hydrocarbons.
  - **TPHSERAS.mac:** This macro extracts the signal for six ions (105, 83, 85, 113, 123, and 183) which cover the following: gasoline range organics (GRO) (105), naphthenes (83), saturated hydrocarbons (SHC) (113), isoprenoids (113 and 183) and bicyclic sesquiterpanes (123), and adds them together to form the TPH fingerprint of the sample. This combined 6 ion fingerprint is also used to quantitate TPH by GC/MS using draft SERAS SOP 1841, *Routine Analysis of Total Petroleum Hydrocarbons (TPH) in Soil and Water by GC/MS-SIM*. Most labs that attempt to quantitate TPH by GC/MS use just ion 85.
  - **ENOTPHSEPN.mac**: This is a similar macro to TPHSERAS.mac, but plots each of the 6 EIC's used for the combined TPH signal of TPHSERAS.mac. Currently, this presentation is being used to study the effects of biodegradation on each of the 6 separate signals (classes of compounds).
  - **Hopster.mac:** This is an example of a stacked set of fingerprints of ion 191 for terpanes and hopanes, over ion 217, steranes, biomarker compounds.
  - **ParentPAH.mac**: Shows the EIC's for just the individual non-alkylated PAH compounds for the entire sample. This extracts and prints a chromatogram for only the parent PAH's; it essentially separates and shows approximately 20 priority pollutant PAH's that are present in the oil.
  - **PAH1803N.mac:** Illustrates all the alkylated PAH homologues in a stacked non-normalized format, and as one combined "fingerprint" for each class (alkyl-naphthalenes, methyl-chrysenes, etc).
  - **MCDET.MAC:** Compiles and shows the total methyl- and dimethyl-chrysene fingerprint bracketed by an internal standard as a reference.

The following macro was used to provide a fingerprint to satisfy QC criteria listed in fingerprint method ASTM D-5739 : *Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry*

- **FireF117.mac**: This shows two stacked fingerprints composed of the ion 85 graphic trace for alkanes and the ion 191 hopane fingerprint. This presentation is primarily used to evaluate the CG-117 reference oil that is analyzed at the beginning and end of a 12 hour sequence, but has useful applications for unknown site oils.

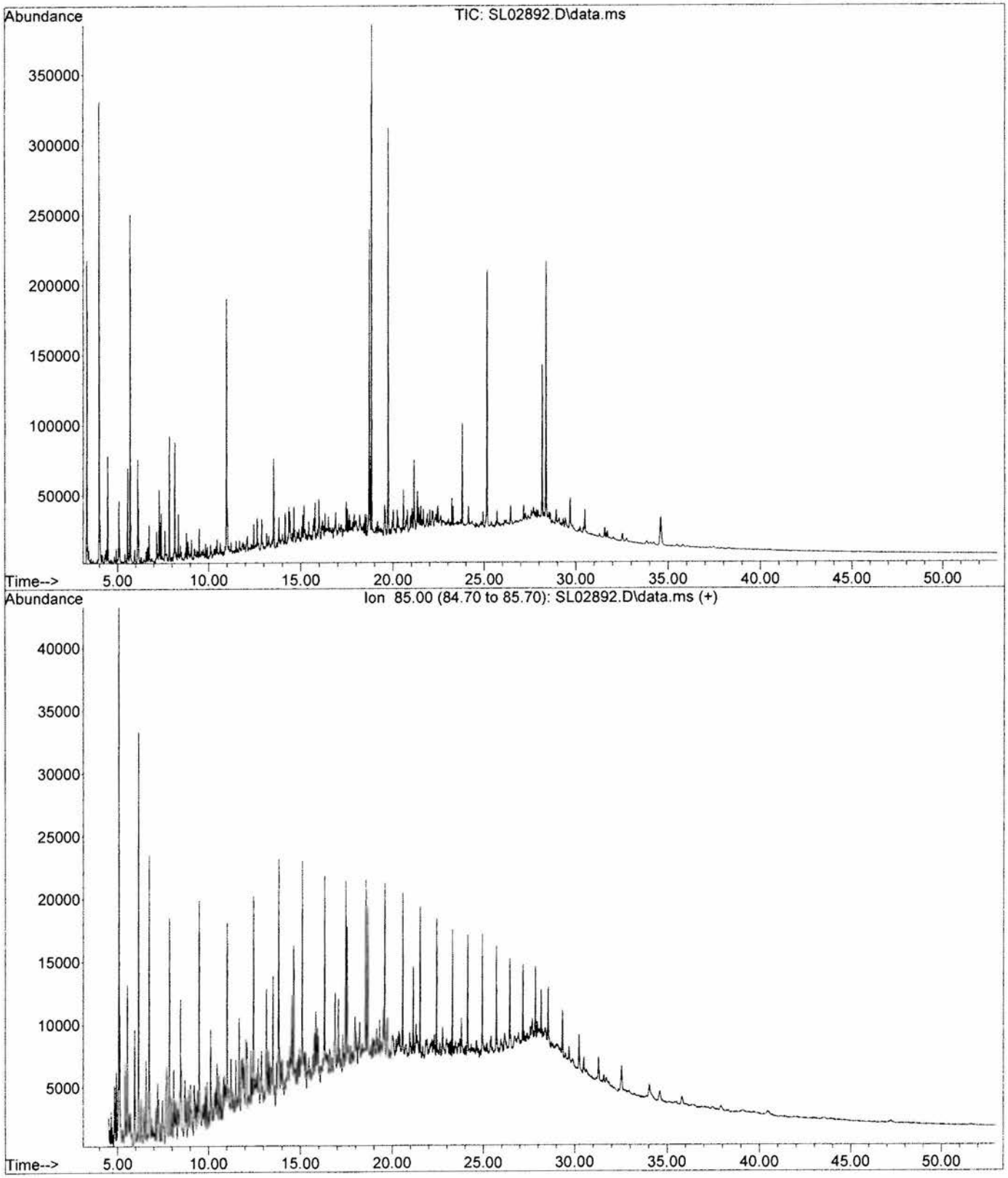
As stated previously, the number of combinations and construction of the stacked VDR are limitless, but the difficulty and challenge is to identify and determine the essential and unique fingerprints, or combination of stacked fingerprints that will aid in establishing the identity of petroleum products and comparing the key fingerprints of a suspected source with samples.

US EPA ARCHIVE DOCUMENT

```
! File  : TICTPH.MAC
! J. Syslo MACRO for presenting TIC vs. TPH as 85 &  83
!  Normalized
NAME TICTPH
format separated,,,1
EIC 4.5:55,85
EIC 4.5:55,83
r5=x+y
TIC 3:55
exchange r5,x
exchange r5,y
merge
draw 2,x
macro "printwin2",go
remove printwin2
tic 4.5:55
exchange x,r0
clear
draw 2,r0
remove ticTPH
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```

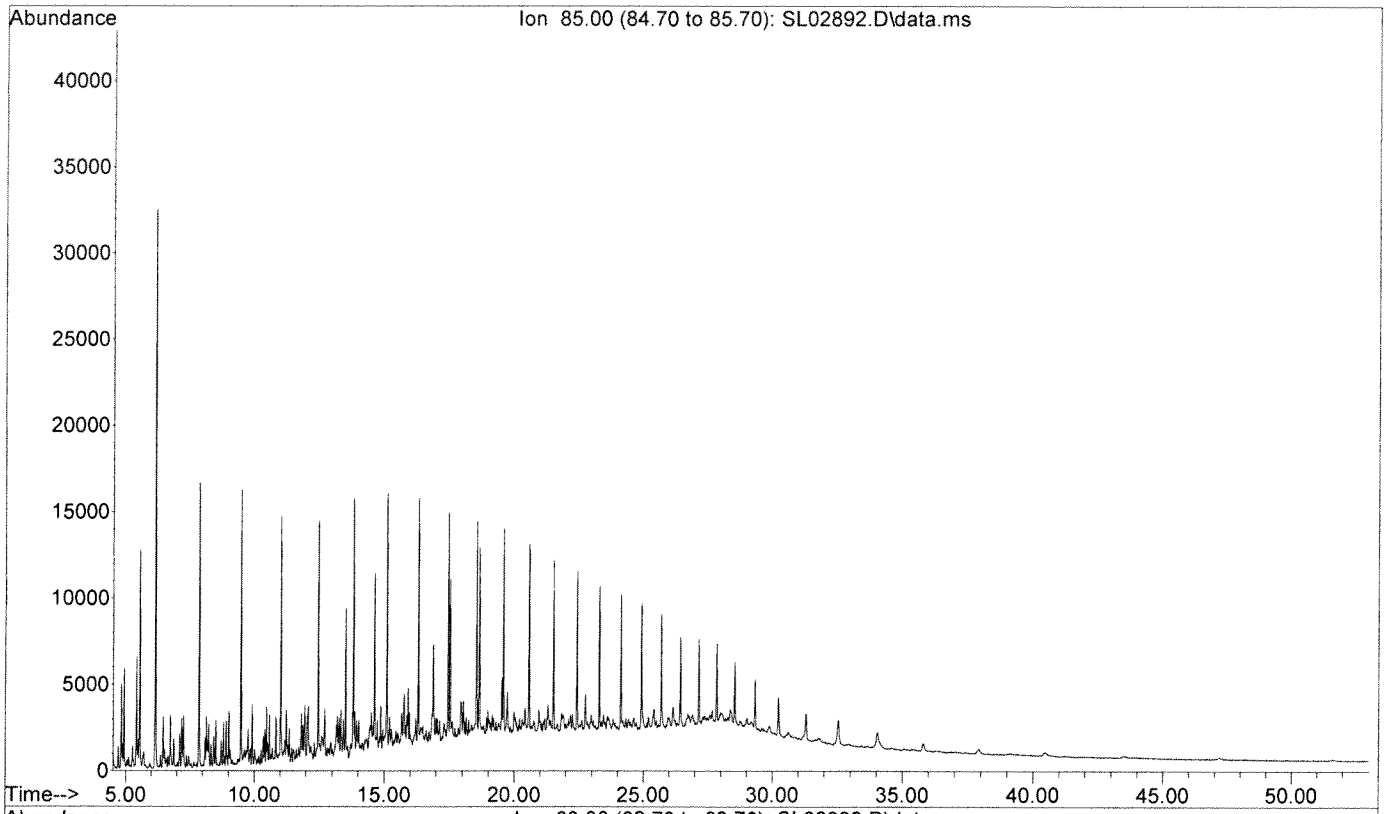


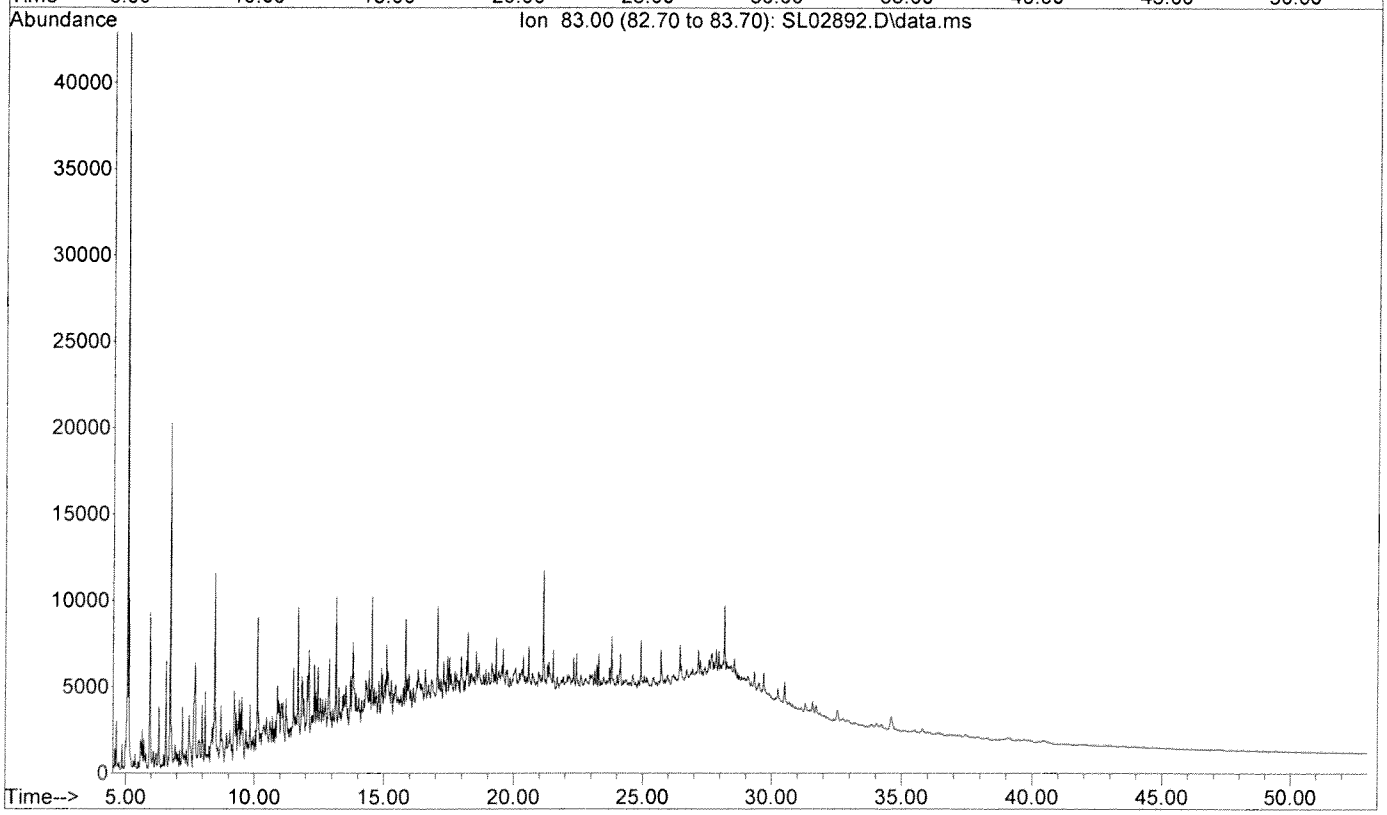
US EPA ARCHIVE DOCUMENT

```
! File  : TPH8583stacked.MAC
! J. Syslo MACRO for presenting  85 over the 83 fingerprint - Not Normalized.
Can determine
! early bildegradation by loss of ion 85 saturated hydrocarbons.  Simple
degradation will
! quickly earase the SHC's, yet leav the ion 83 fingerprint unchanged.  JS
        !    Not Normalized
NAME TPH8583stacked
format separated,,,0
EIC 4.5:55,85
EIC 4.5:55,83
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
exchange x,r0
clear
draw 2,r0
remove TPH8583stacked
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```





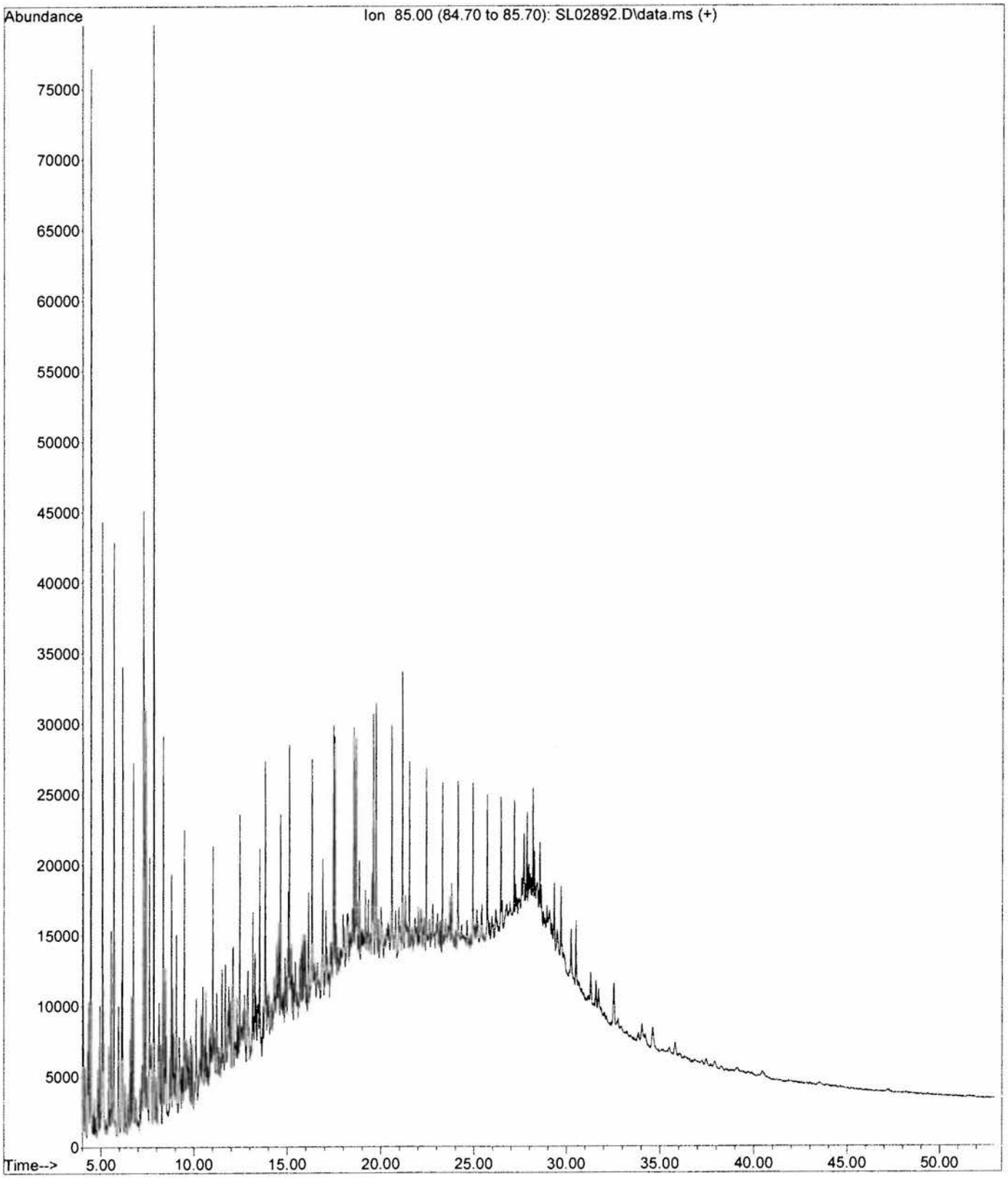
US EPA ARCHIVE DOCUMENT

```
! File  : TPHSERAS.mac
! J. Syslo MACRO for sum of  GRO (105) & DRO (85+83+113+123+183) TPH
! For total GRO & DRO +UCM  TPH quanted as total fingerprint
!   *** New way to do TPH . . .  with 6 ions not just 83 + 85****
!   *** Developed October 2009 for DRO in water PT sample by J. Syslo****
!
! Plots NOT normalized
! integratable in window 2
! This will automatically print the fingerprint, then after macro runs, show TPH
over TIC onscreen


NAME TPHSERAS
format separated,,,0
EIC 4:55,105
EIC 4:55,123
r6=x+y
eic 4:55,113
eic 4:55,183
r7=x+y
eic 4:55,85
eic 4:55,83
r8=x+y
r4=r6+r7+r8
exchange r4,r0
DRAW 2,r0
macro "printwin2.mac",go
!emove printwin2
TIC 4:55
draw 1,x
remove TPHSERAS
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



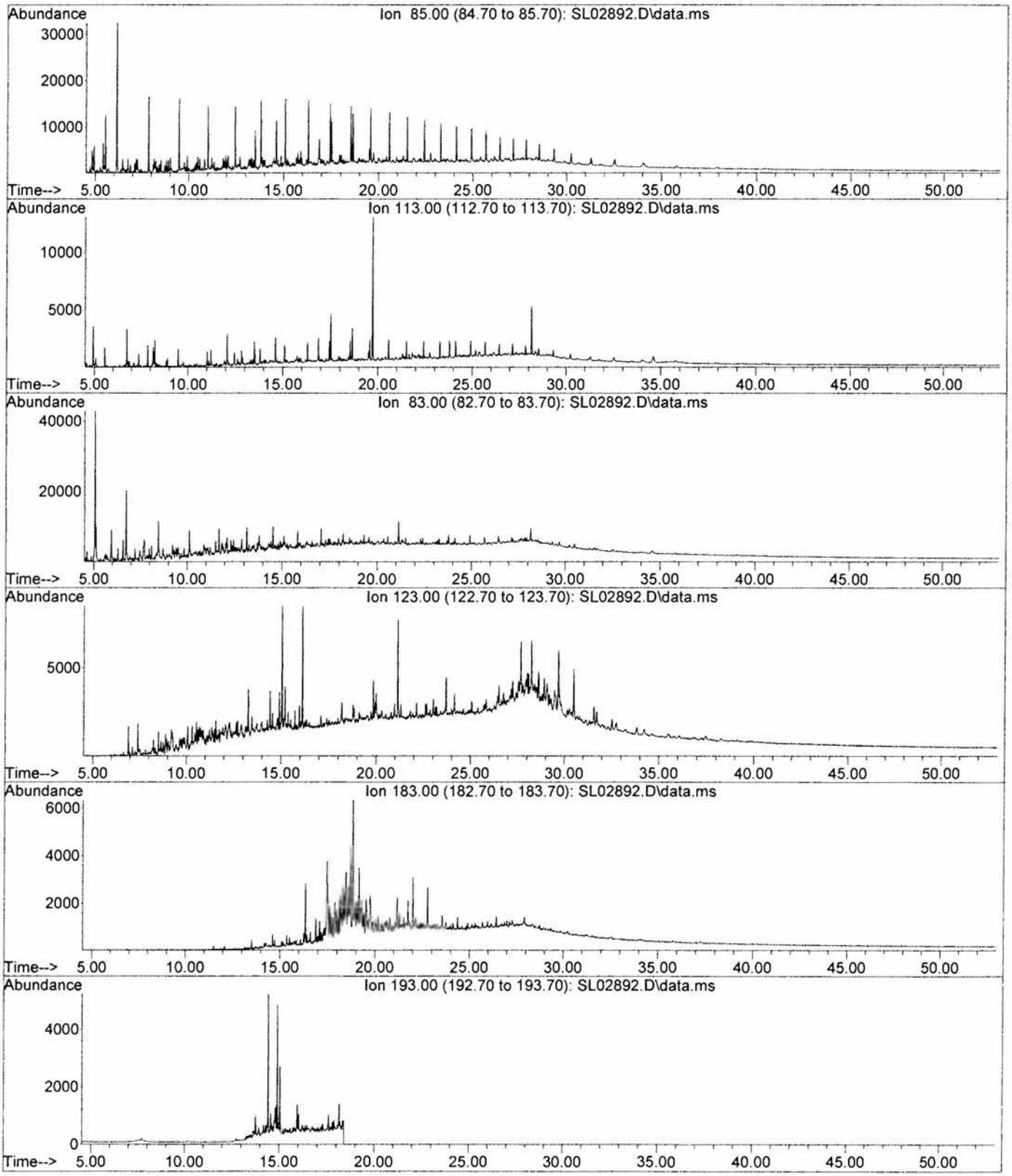
US EPA ARCHIVE DOCUMENT

```
! File  : ENOTPHsepn.mac
! J. Syslo MACRO for presenting TPH as separate 85 + 113 + 83 + 123 + 183 + 193
signals
 !    normalized and stacked for Enbridge Oil - left ion 105 out (formaldehyde
peaks)

NAME enoTPHsepn
format separated,,,1
EIC 4.5:55,85
eic 4.5:55,113
merge
EIC 4.5:55,83
merge
EIC 4.5:55,123
merge
EIC 4.5:55,183
merge
EIC 4.5:55,193
merge
!  EIC 4.5:55,105
!  merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
remove ENOTPHsepn
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012   22:38      using AcqMethod DROTPH051611.M
Instrument  :    Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



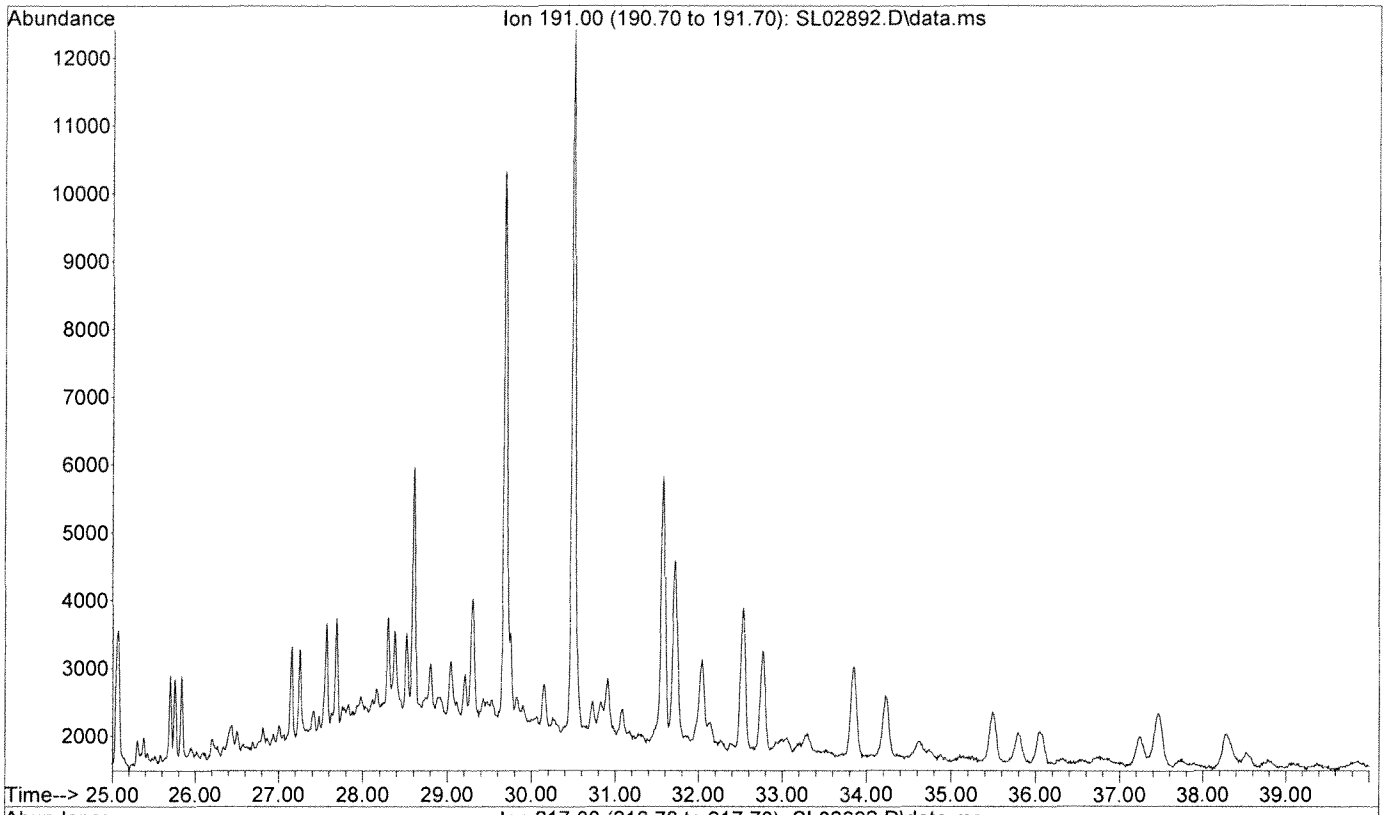
US EPA ARCHIVE DOCUMENT

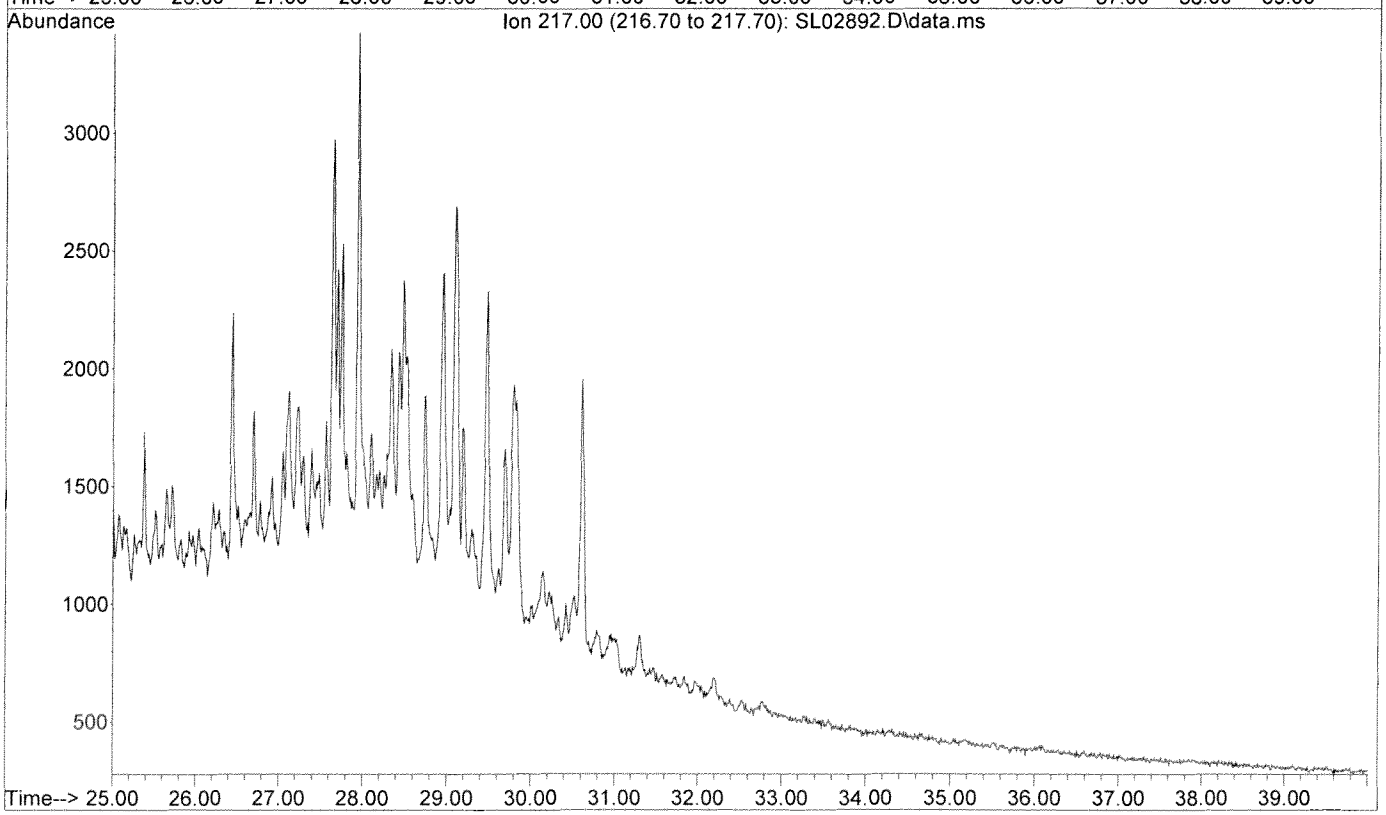
```
! File  : HopSter.MAC
! J. Syslo MACRO for hopane fingerprint from November 2007 program:
! for displaying both Hopane and Steranes on one page:  normalized chromatogram
 !  using it on Slick2 now 8/18/09  J. Syslo

NAME HOPSTER
format separated,,,1
EIC 25:40,191
EIC 25:40,217
merge
draw 2,x
macro "printwin2.mac",go
remove printwin2
tic 4:50
exchange x,r0
draw 2,r0
remove hopster
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```





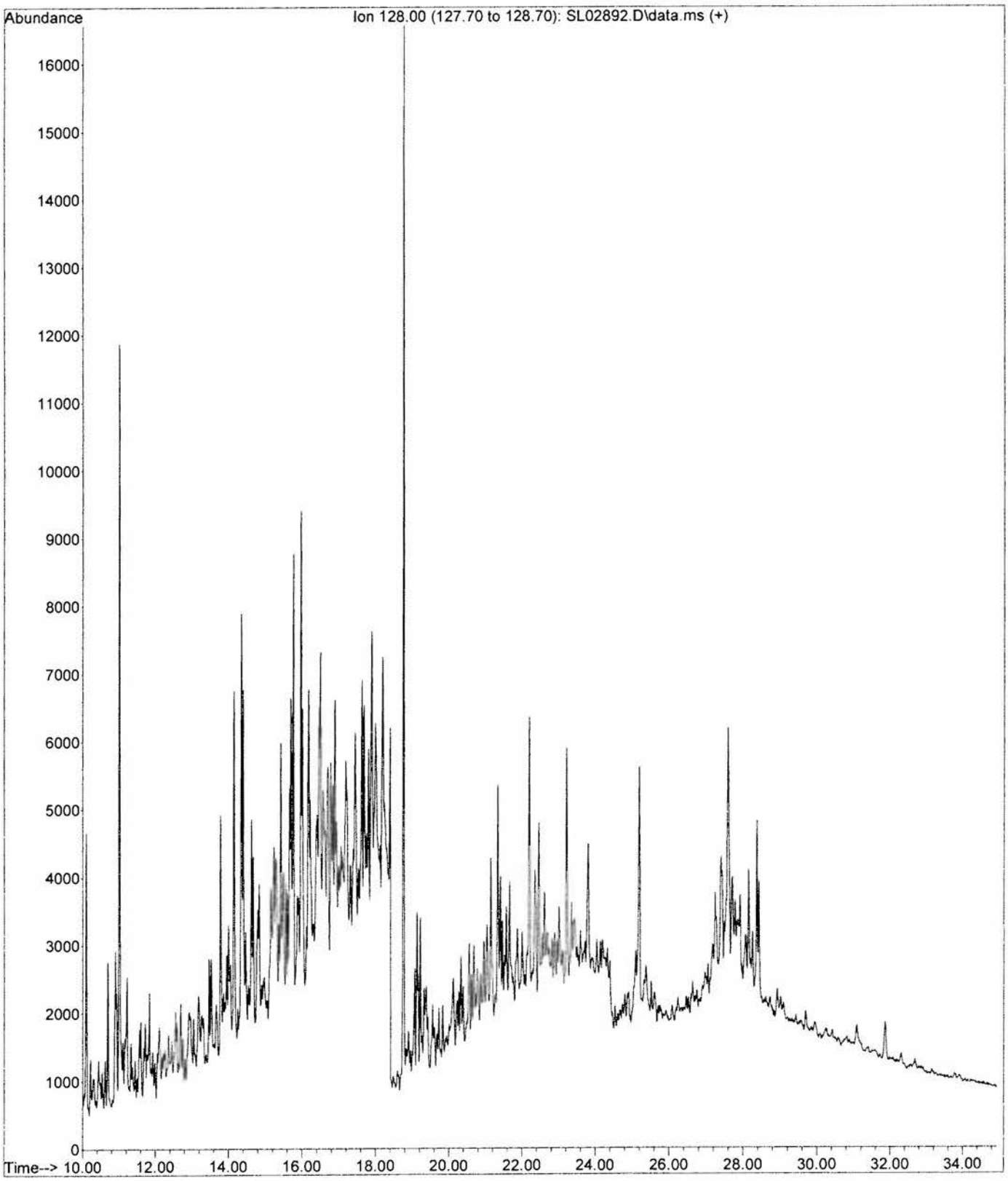
US EPA ARCHIVE DOCUMENT

```
! File  : ParentPAH.mac
! J. Syslo MACRO to show just the parent PAH's - 10 ions, 01/12/2012
! non-normalized data - all chromatograms added & automatically prints.

NAME ParentPAH
format separated,,,0
EIC 10:35,128
EXCHANGE X,R5
EIC 10:35,152
pap=x+r5
EIC 10:35,154
pap=x+pap
EIC 10:35,166
pap=x+pap
EIC 10:35,178
pap=x+pap
EIC 10:35,202
pap=x+pap
EIC 10:35,228
pap=x+pap
EIC 10:35,252
pap=x+pap
EIC 10:35,276
pap=x+pap
EIC 10:35,278
pap=x+pap
x=pap
exchange x,r0
DRAW 2,r0
        macro "printwin2",go
        remove printwin2
remove parentpah
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02892.D
Operator : Syslo
Acquired : 1 Mar 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0006
Misc Info : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8



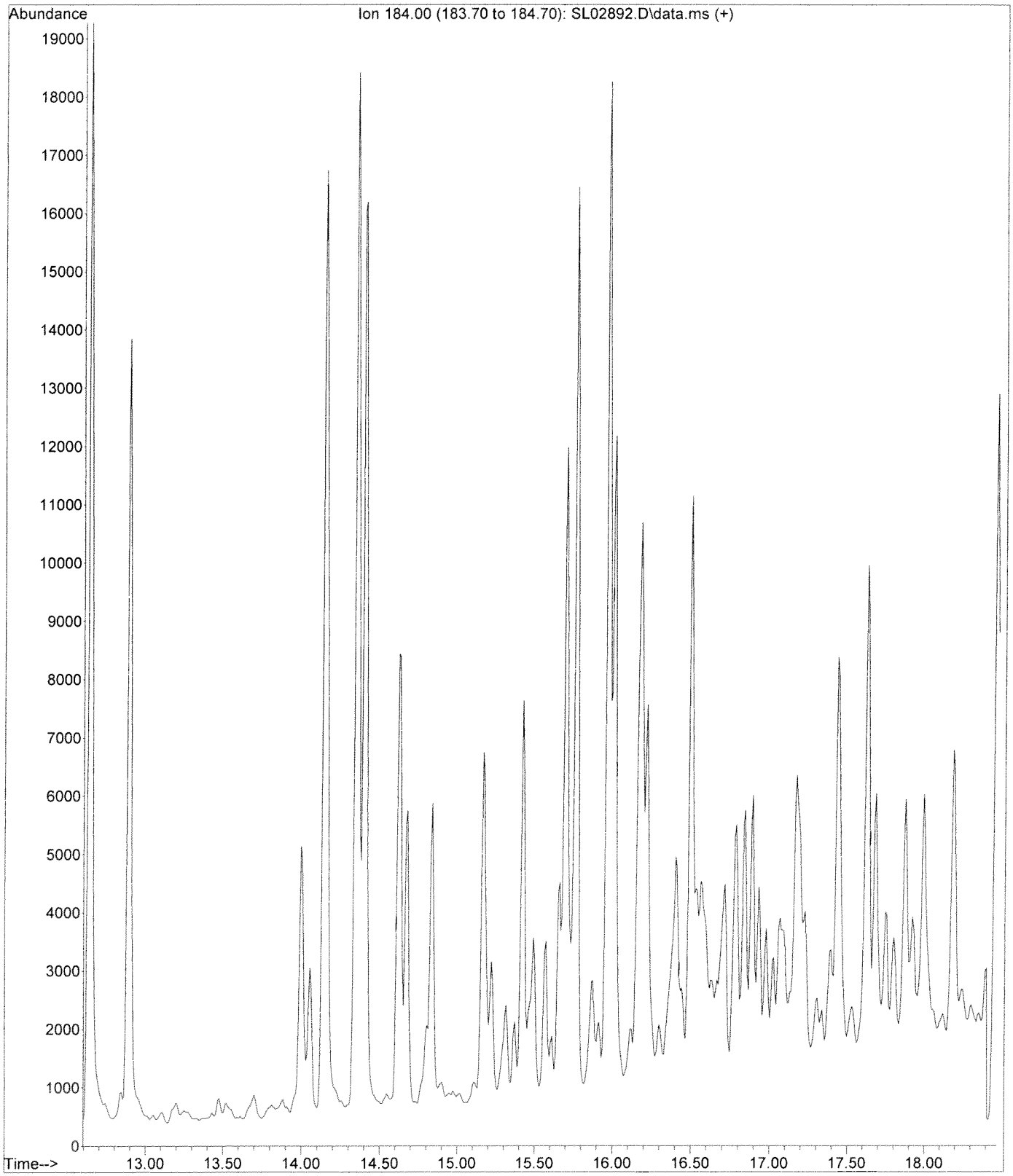
US EPA ARCHIVE DOCUMENT

```
! File  : PAH1803N.MAC
! J. Syslo MACRO for illustrating all PAH homologues:  4/10/08
! Separate fingerprints are not Normalized
! Combined fingerprints are not normalized.

NAME PAH1803N
format separated,,,0
! methyl-naphthalenes
        macro "MNSCO.mac",go
        macro "MNS.mac",go
! methyl-fluoranthenes
                macro "FLco.mac",go
                macro "FL.mac",go
! methyl-dibenzothiophenes (Sulful fingerprint)
        macro "SS216co.mac",go
        macro "ss216.mac",go
!methyl-phenanthrenes
                macro "PAco.mac",go
                macro "PA.mac",go
! methyl-fluoranthene/pyrenes
        macro "FPco.mac",go
        macro "FP.mac",go
! methyl-chrysenes
               macro "MCco.mac",go
               macro "mc.mac",go
! REMOVE PAH1803N
TIC 3:55
exchange x,r0
draw 2,r0
remove pah1803N
clear
return
```

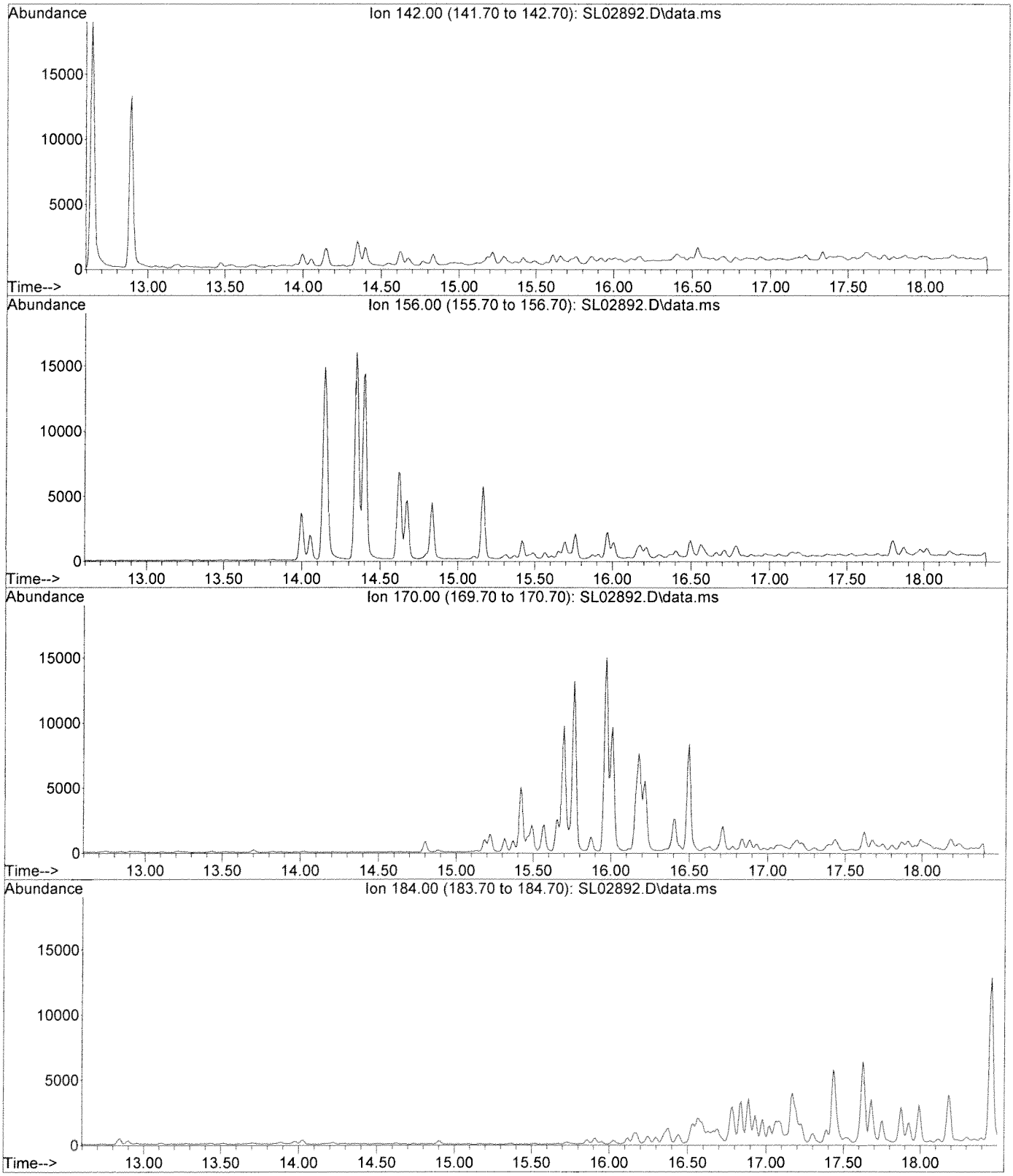
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



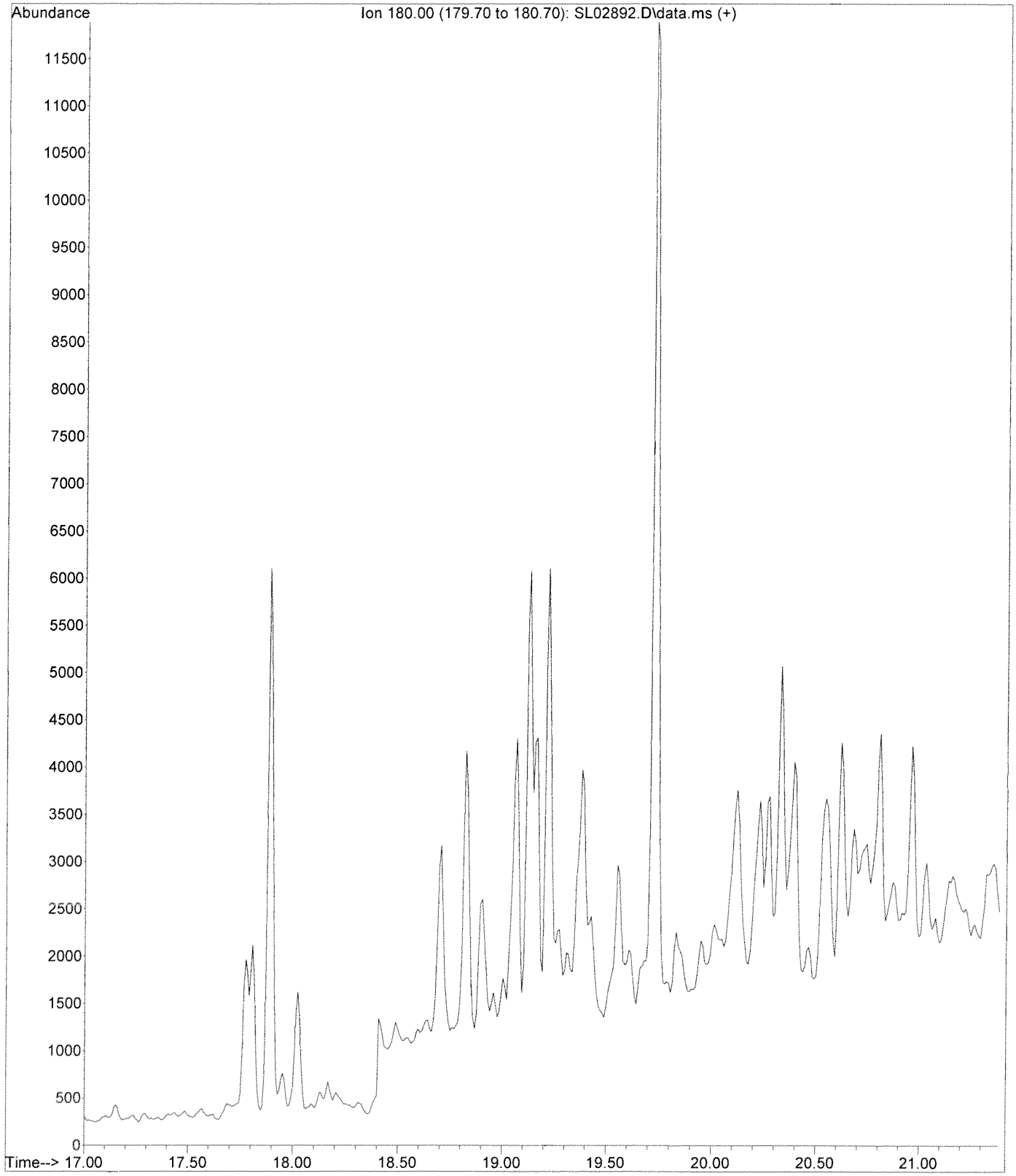
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument :    Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



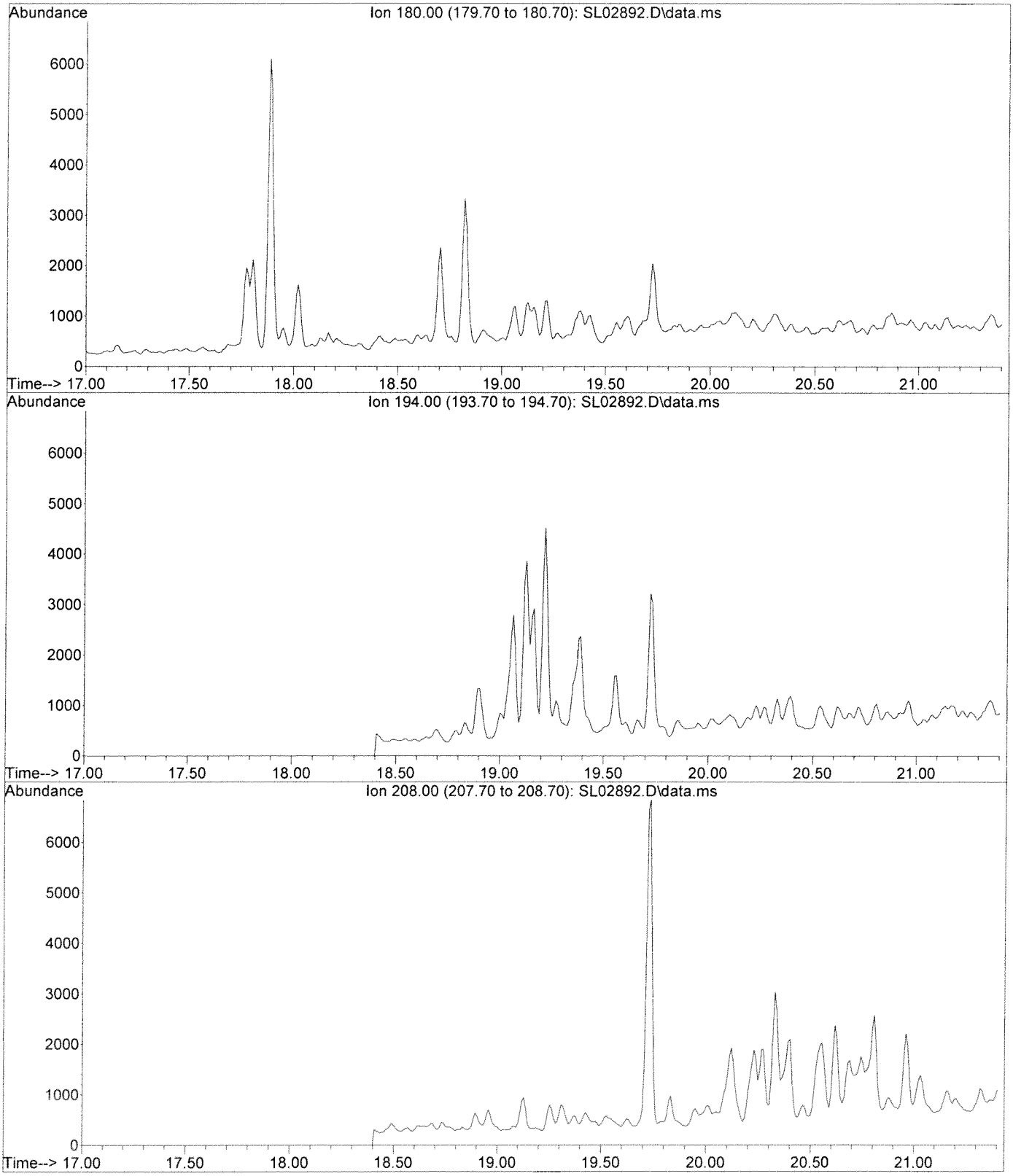
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



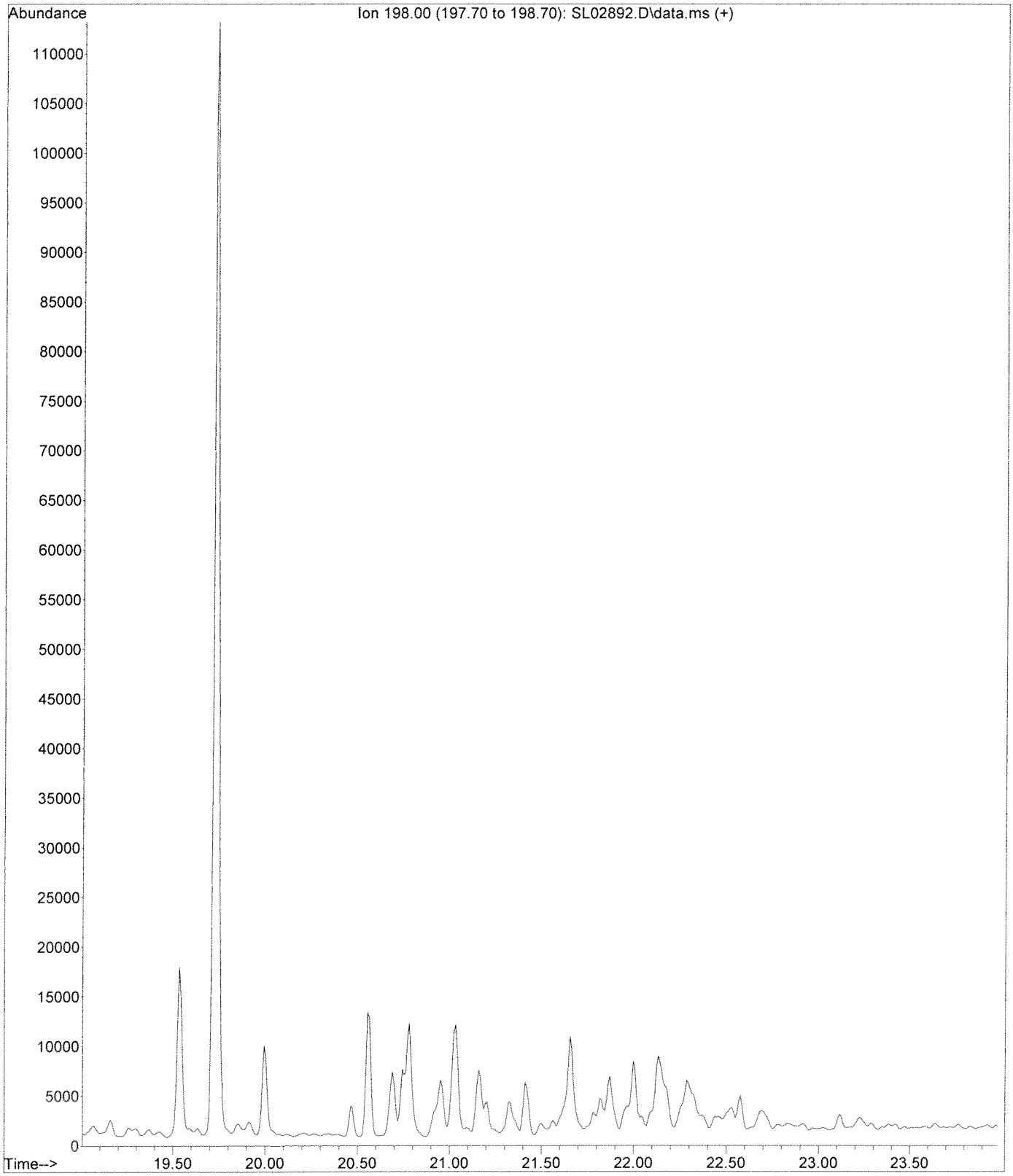
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



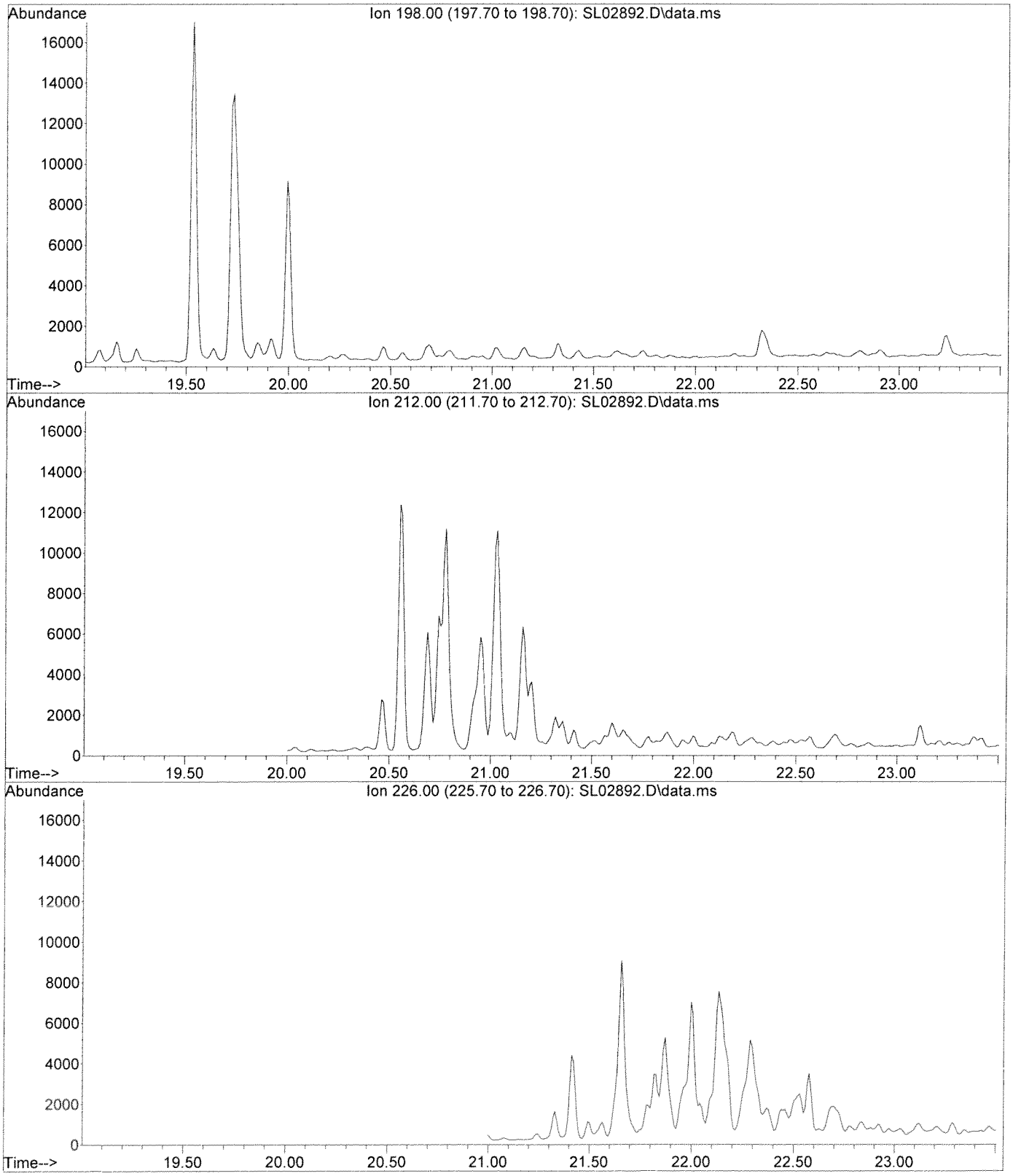
US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02892.D
Operator : Syslo
Acquired : 1 Mar 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0006
Misc Info : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8



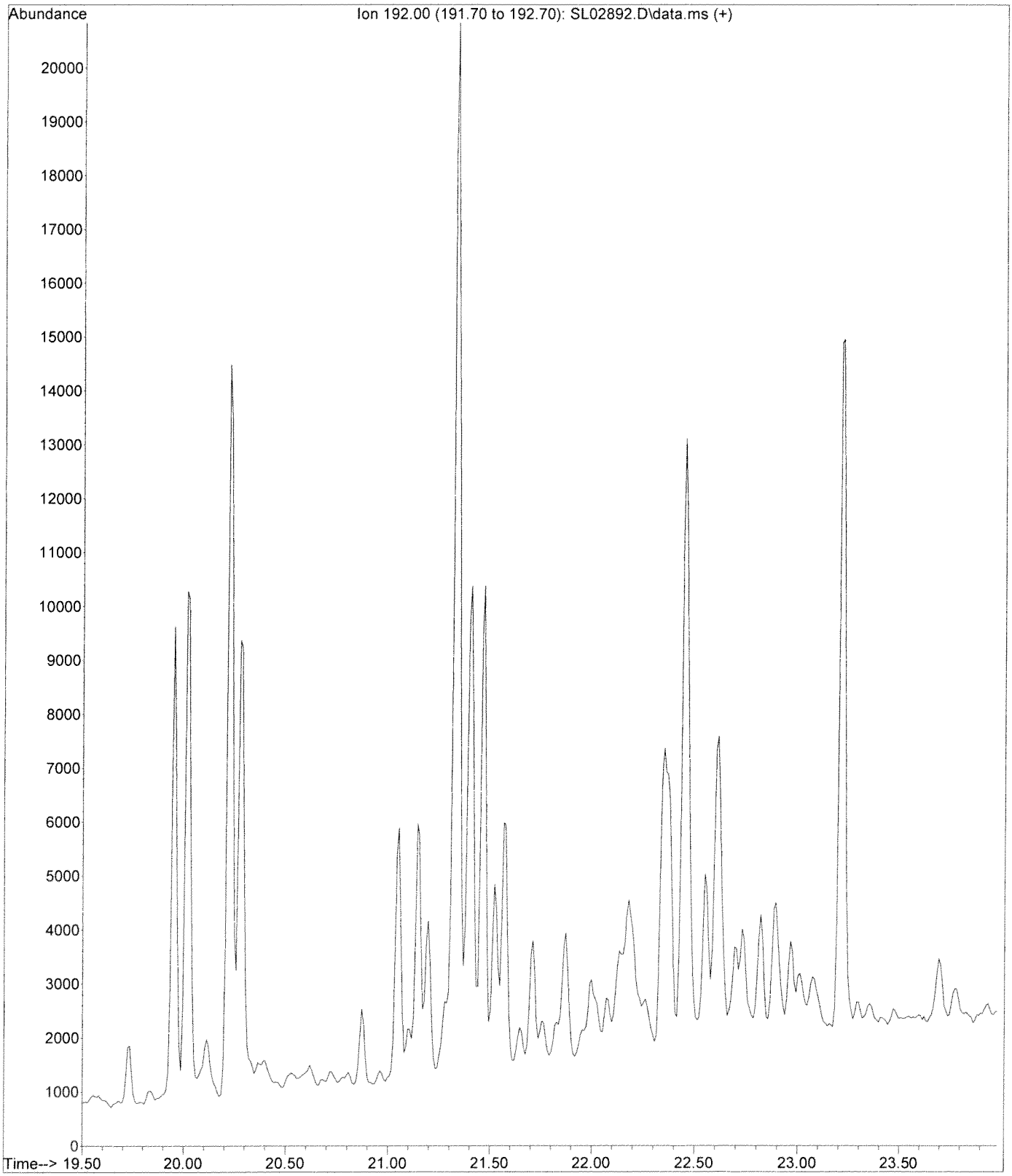
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



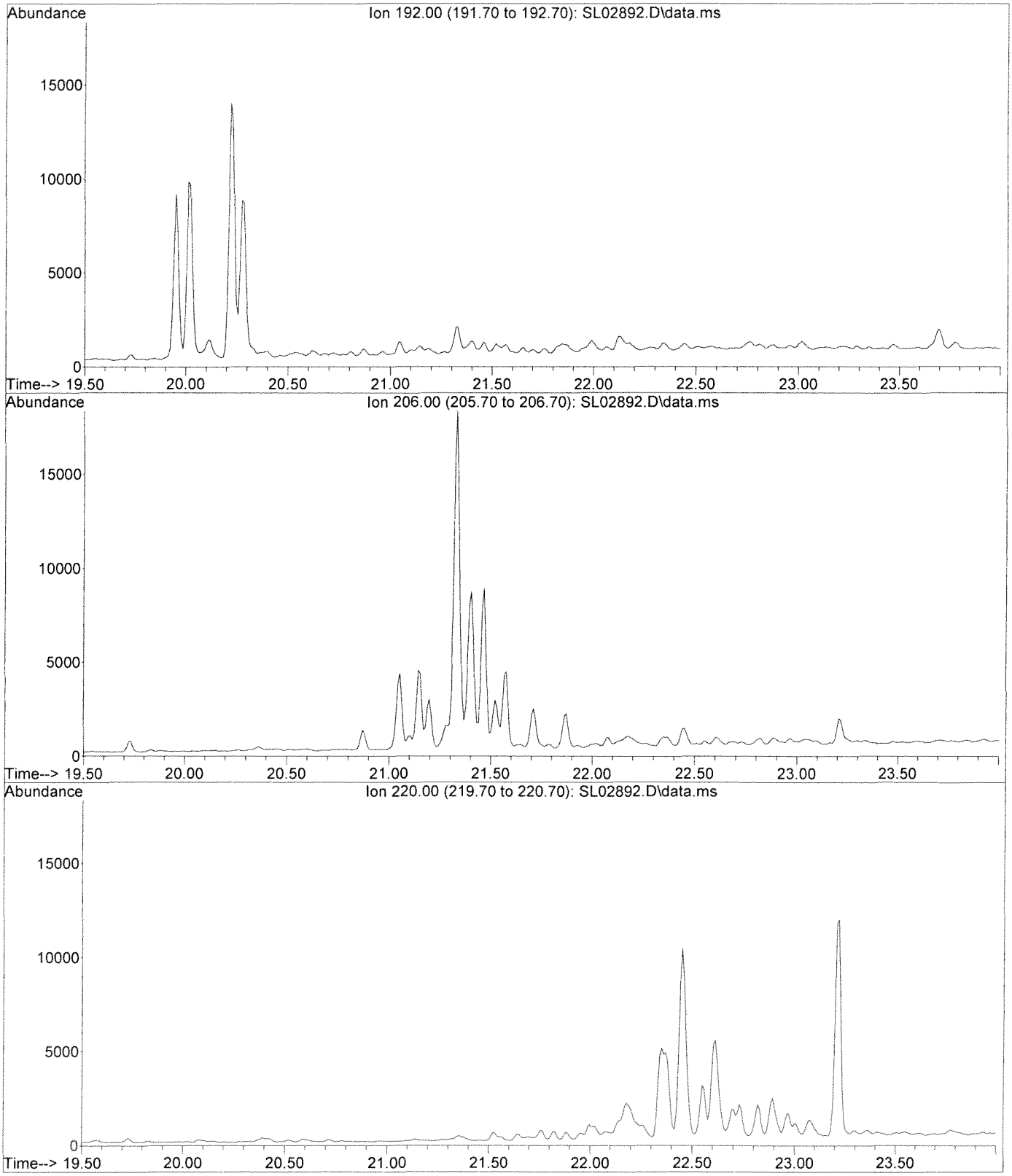
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



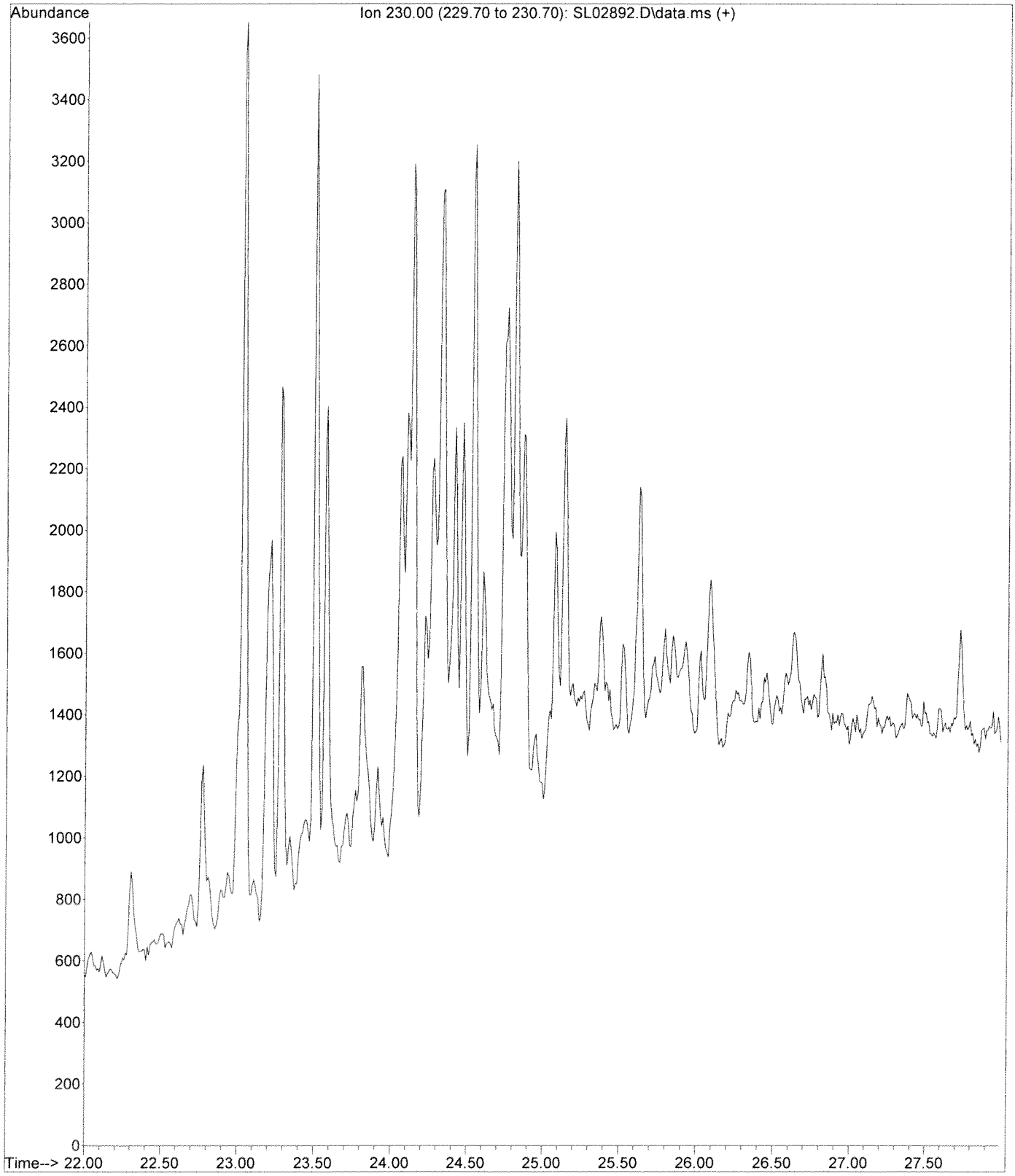
US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



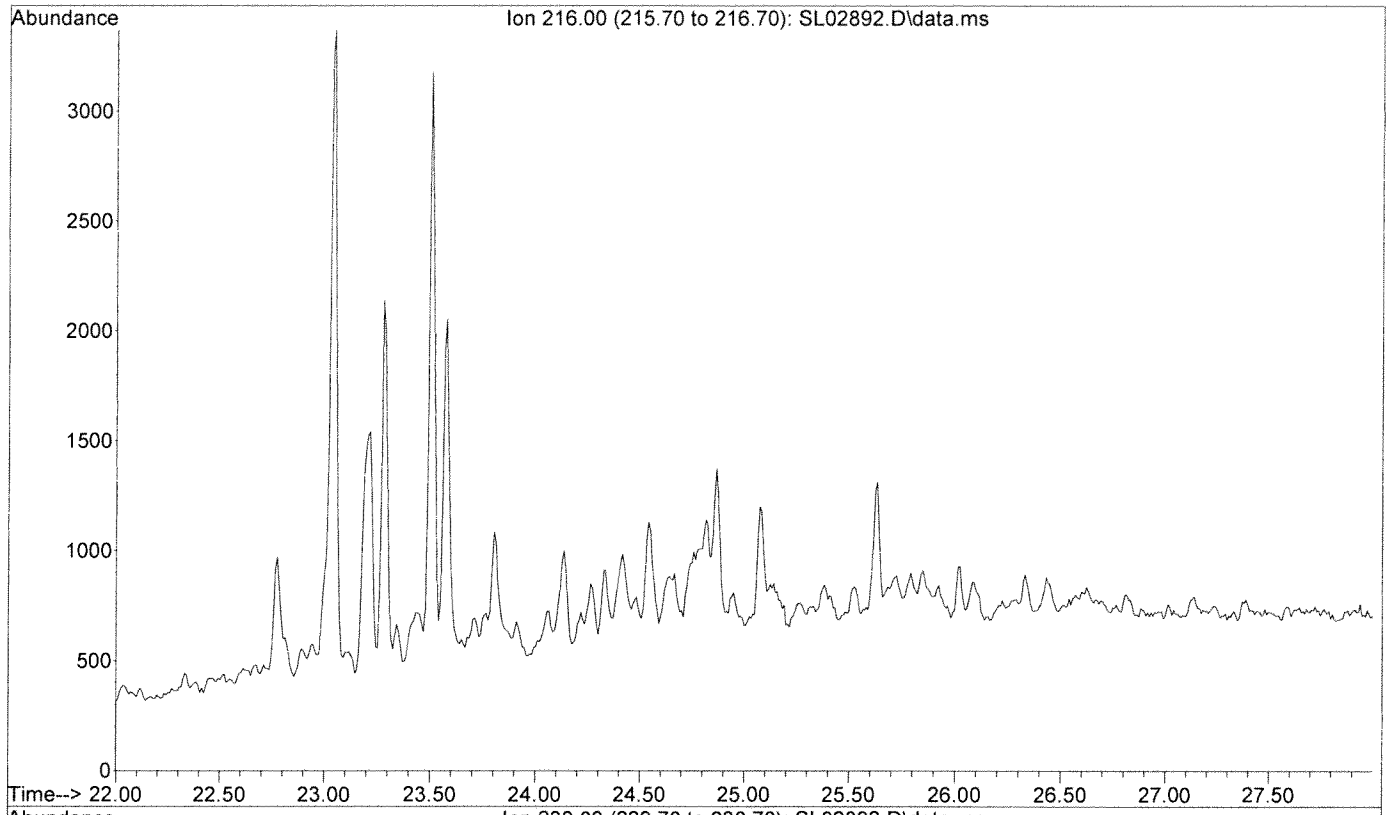
US EPA ARCHIVE DOCUMENT

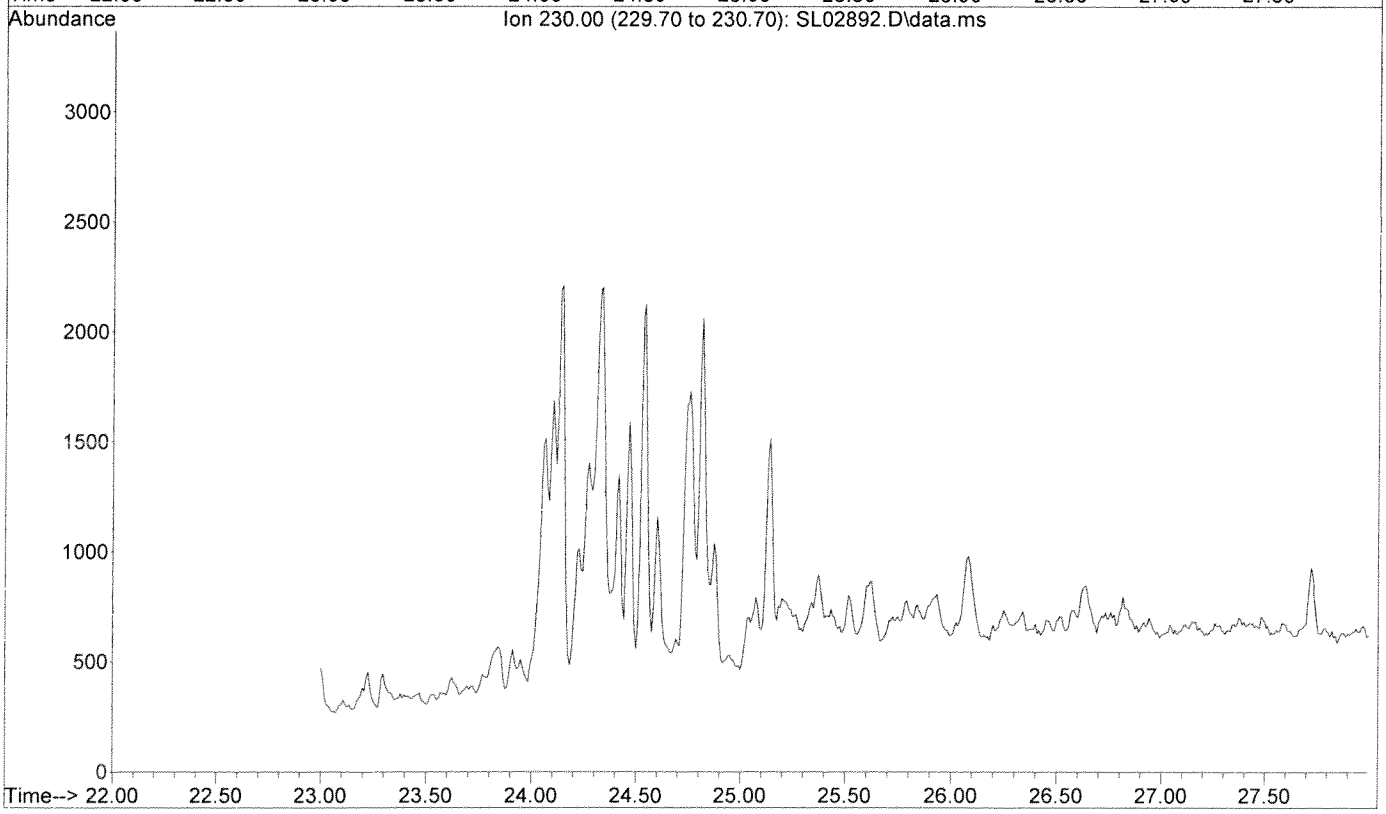
File :C:\msdchem\1\DATA\030112\SL02892.D
Operator : Syslo
Acquired : 1 Mar 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0006
Misc Info : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8



US EPA ARCHIVE DOCUMENT

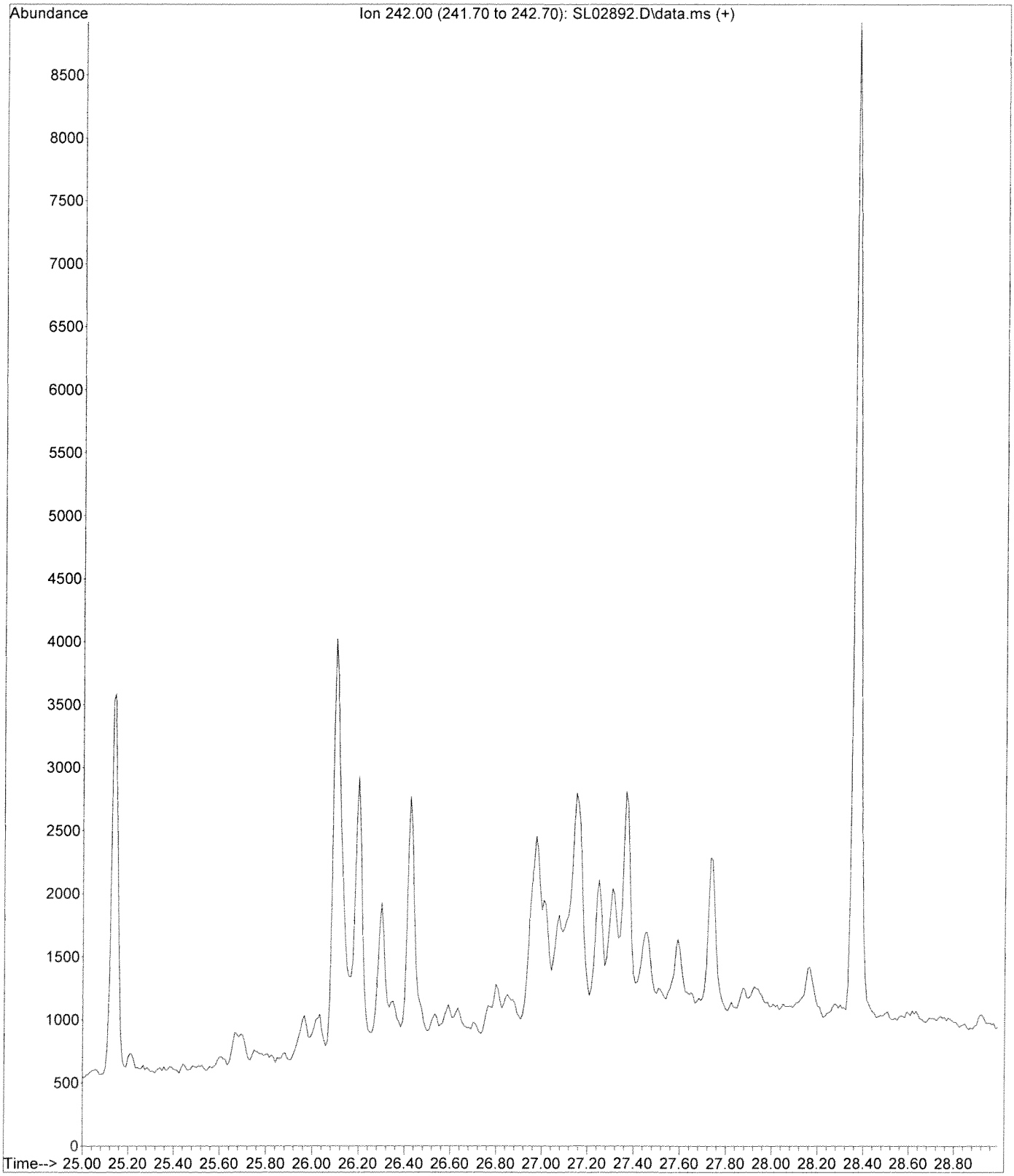
```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```





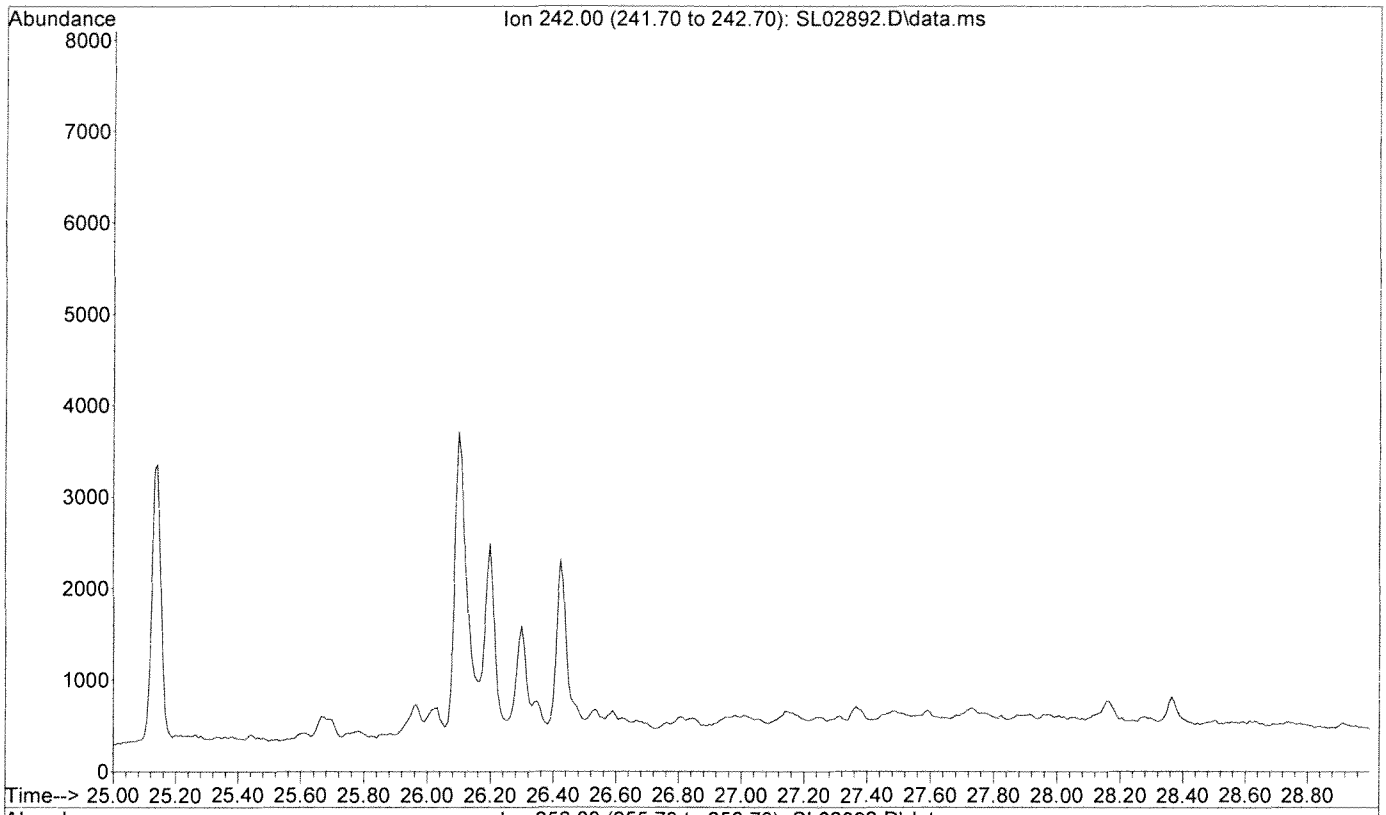
US EPA ARCHIVE DOCUMENT

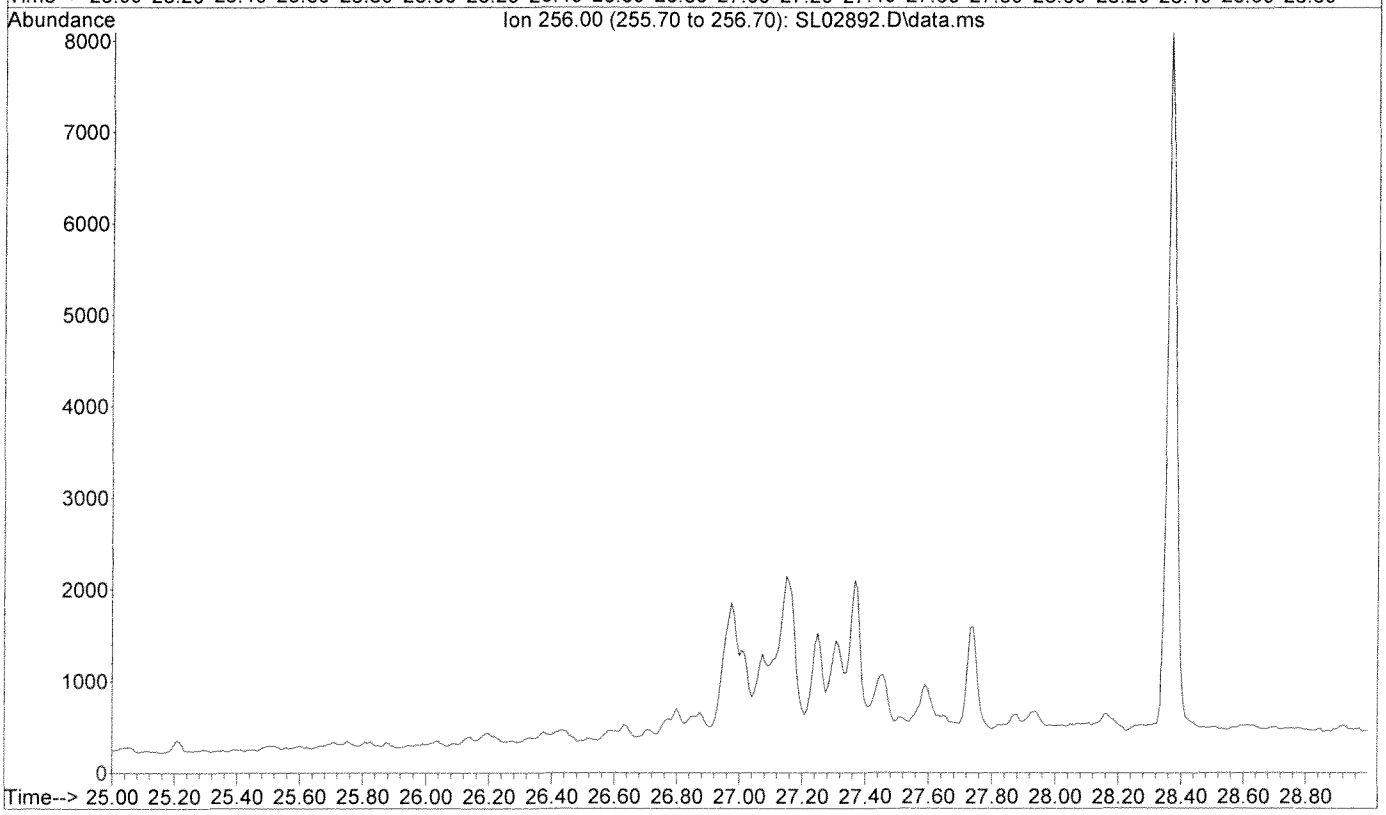
File :C:\msdchem\1\DATA\030112\SL02892.D
Operator : Syslo
Acquired : 1 Mar 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0006
Misc Info : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8



US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02892.D
Operator : Syslo
Acquired : 1 Mar 2012 22:38 using AcqMethod DROTPH051611.M
Instrument : Slick2
Sample Name: SERAS-017-0006
Misc Info : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8





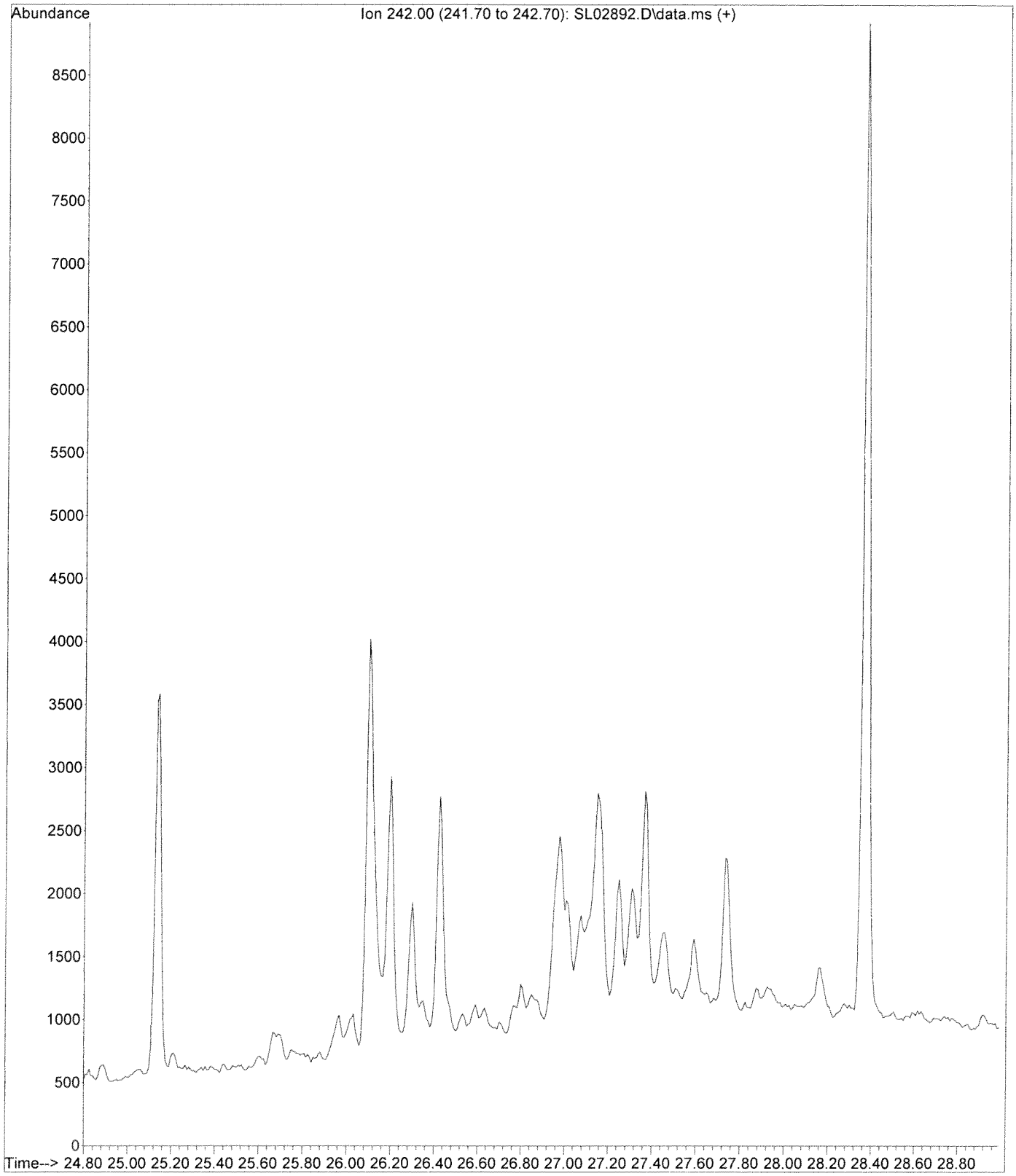
US EPA ARCHIVE DOCUMENT

```
! File  : MCDET.MAC
! J. Syslo MACRO for compiling methyl chrysene fingerprints in oil -   11/15/07
on Liberty

NAME MCDET
format separated,,,1
EIC 24.8:29,242
EIC 24.8:29,256
r5 = x+y
DRAW 2,r5
macro "printwin2.mac",go
remove printwin2
remove Mcdet
clear
return
```

US EPA ARCHIVE DOCUMENT

```
File        :C:\msdchem\1\DATA\030112\SL02892.D
Operator    : Syslo
Acquired    :  1 Mar 2012  22:38      using AcqMethod DROTPH051611.M
Instrument  :   Slick2
Sample Name: SERAS-017-0006
Misc Info   : CL-6B-072223-092710-JPS-KA-001-33
Vial Number: 8
```



US EPA ARCHIVE DOCUMENT

10K CG-117 Reference 0.1

US EPA ARCHIVE DOCUMENT

Quantitation Report (QT Reviewed)

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02894.D
Acq On : 2 Mar 2012 00:40
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003 EOS Reference
ALS Vial : 10 Sample Multiplier: 1

Quant Time: Mar 05 12:23:25 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc	Units	Dev(Min)
Internal Standards						
1) d10-Phenanthrene	18.708	188	727287	10.00	ug/mL	0.00
5) d30-Tetradecane	13.529	66	207961	10.00	ug/mL	0.00
6) d50-Tetracosane	23.792	66	281953	10.00	ug/mL	0.00
8) d74-Hexatriacontane	34.605	66	245390	10.00	ug/mL	0.00
System Monitoring Compounds						
2) d10-Anthracene {S}	0.000	188	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
3) d14-o-Terphenyl {S}	0.000	244	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
4) 5a-Androstane {S}	0.000	260	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
7) d62-Triacontane {S}	0.000	66	0d	0.00	ug/mL	
Spiked Amount 20.000			Recovery =	0.00%		
Target Compounds						Qvalue

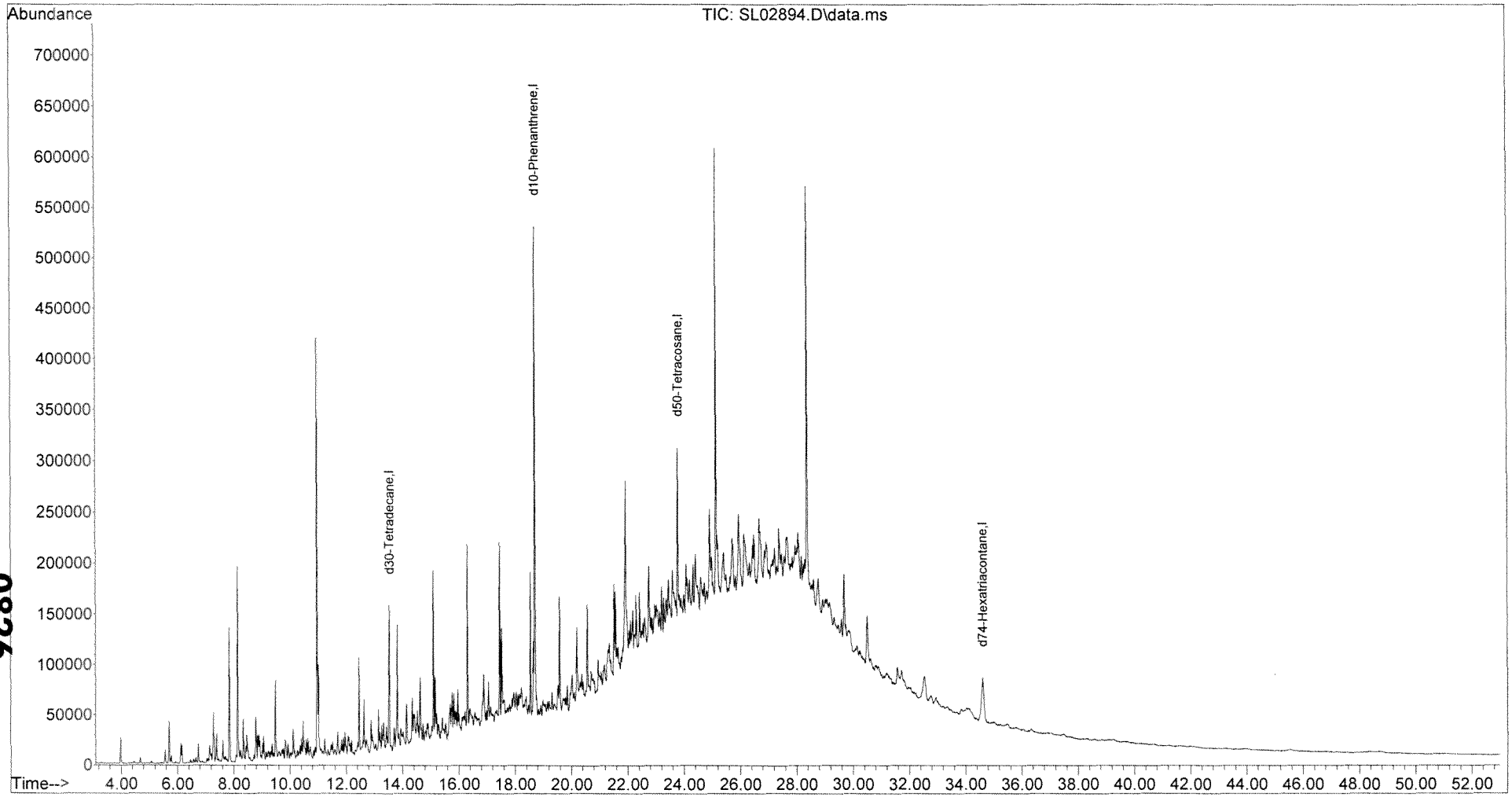
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Data Path : C:\msdchem\1\DATA\030112\
Data File : SL02894.D
Acq On : 2 Mar 2012 00:40
Operator : Syslo
Sample : 10K CG-117 Reference
Misc : RUL0003 EOS Reference
ALS Vial : 10 Sample Multiplier: 1

Quant Time: Mar 05 12:23:25 2012
Quant Method : C:\MSDCHEM\1\METHODS\DROTPH051611.M
Quant Title : DRO/TPH ICAL + Surr. 05/16/11
QLast Update : Thu Mar 01 17:23:08 2012
Response via : Initial Calibration

SERAS-017-DTM-011413_3

0826



```
! File  : FIREF117.MAC
! J. Syslo MACRO for reference fingerprint using CG117: 11/22/09
!  Hopanes are normalized for easy viewing!
! Draws chromatogram in window 2 and window 1 . .  you have to print "TIC &
Spectrum"
!  I have a lot of old CG-117's like that - better reference"  JS

NAME FIREF117
format separated,,,0
EIC 4:55,85
exchange r0,x
EIC 26:44,191
exchange r0,y
draw 2,y
draw 1,x
REMOVE FIREF117
clear
return
```

US EPA ARCHIVE DOCUMENT

File :C:\msdchem\1\DATA\030112\SL02894.D
Operator : Syslo
Acquired : 2 Mar 2012 00:40 using AcqMethod OILSIMS2.M
Instrument : Slick2
Sample Name: 10K CG-117 Reference
Misc Info : RUL0003 EOS Reference
Vial Number: 10

