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Chemical Fingerprinting of Additional Yellow Rock Well Oils – mid- and late-August 2023 Westlake Sulphur Dome Study

Dear Mr. Charpentier,

NewFields is pleased to provide you with this report of chemical fingerprinting results for four crude oil samples collected in August 2023 as part of the on-going investigation of the Westlake US 2 LLC (Westlake) salt dome caverns in the Sulphur Mines oil field, Calcasieu Parish, Louisiana (the Site). The four oil samples studied were collected from three Yellow Rock, LLC wells in the area, including two samples from different reservoir zones in one of the wells.

This study follows five earlier chemical fingerprinting studies at the Site.[1,2,3,4,5] These earlier studies included multiple oils from the 7B cavern well (collected in Jan., March, May and June 2023) and eight crude oil samples from six nearby Yellow Rock wells [110159, 185997 (twice), 210185, 252112, 109963, 209459 (twice)] and the Yellow Rock tank battery. Among other conclusions, these earlier studies showed:

- The 7B cavern oils are chemically distinct from the locally produced (Yellow Rock) crude oils studied, which varied only slightly among themselves.
- There was no change in composition of the 7B cavern oil between January and June 2023, indicating no local crude oil(s) had or was presently entering Cavern 7.
- Surface sheens at Bubble Site 22 (Jan. 2023), Bubble Site No. 20 (March 2023), Bubble Site No. 24 (May 2023), and from Central Lake (June 2023) were derived from locally produced crude oil(s), not 7B cavern oil.

The present study expands upon these earlier studies through the chemical fingerprinting of four oils from three additional Yellow Rock wells that were not previously sampled/analyzed.

[1] Stout, S.A. (2023) Chemical fingerprinting of oils, Westlake Sulphur Dome Study. NewFields Report dated March 10, 2023.
[2] Stout, S.A. (2023) Chemical fingerprint of oily net – No. 20, Westlake Sulfur Dome Study. NewFields Report dated April 27, 2023.
[3] Stout, S.A. (2023) Chemical fingerprint of 7B cavern oil – March 30, 2023, Westlake Sulfur Dome Study. NewFields Report dated May 3, 2023.
[4] Stout, S.A. (2023) 7B Cavern Oil, Cavern 4 Oil, Select Yellow Rock Well Oils, and a Bubble Site 24 Sheen – May 2023, Westlake Sulphur Dome Study. NewFields Report dated July 11, 2023 – Amended July 14, 2023.
[5] Stout, S.A. (2023) Chemical fingerprint of 7B cavern oil, selected Yellow Rock well oils and a Central Lake sheen – June 2023, Westlake Sulfur Dome Study. NewFields Report dated July 25, 2023.

Samples

Table 1 provides an inventory of samples included in this study – along with those previously studied for ease of reference. The four locally produced crude oil samples studied herein were collected on August 17 and August 29, 2023. The two oils collected on August 17 represent splits of samples collected by Yellow Rock that were provided to ERM. The two oils collected August 29 represent the top and bottom of the oil column present in Yellow Rock 189416. All of the samples were sent to NewFields' alliance laboratory, Alpha Analytical (Mansfield, Massachusetts, USA), on two separate dates where they all arrived safely. A copy of the chain-of-custody documents received with each shipment is found in **Attachment 1**.

Objectives

The objective of the current study was to expand the population of locally produced crude oils studied in order to further assess the chemical variability that exists among local crudes, which may aid in determining the specific source (e.g., well/reservoir zone) of any local oil(s) if such ever becomes evident within Cavern 7. This study expands the number of locally produced crude oils studied to thirteen (Table 1).

Methods

This objective was pursued using specific chemical fingerprinting and interpretation methods based on the CEN oil spill identification protocol,[6] as was described in the original study's report, its attachments, and the flowchart depicted in **Figure 1**.[7] The chemical fingerprinting analyses performed herein remain unchanged from the previous reports and are described in **Attachment 2**. However, as first described in the study of May 2023 oils (Footnote 4), this study also included the (re-)analysis of the cavern 7B oil collected in January 2023, that was adopted as a *site-specific reference oil*. This oil is being re-analyzed for quality control with each "batch" of samples analyzed from the Sulphur Dome site to assess the long-term precision of diagnostic ratios used in the quantitative (statistical) comparison of samples from the site. An expanded discussion of this topic, which has been updated based on the new results of the site-specific reference oil reported herein, is included in **Attachment 3**.

Results & Discussion

The complete Alpha Environmental Testing Report (ETR) including all sample preparation data, instrument calibrations, QC data and chromatograms is maintained on file by NewFields (ETR L2348036). The tabulated results for the targeted compounds in each analysis performed are contained in **Attachment 4**. The full-size GC/FID chromatogram obtained in Tier 1 (modified EPA Method 8015D) analysis is provided in **Attachment 5** and selected extraction ion profiles (EIPs) obtained in Tier 2 (modified EPA Method 8270D) are provided in **Attachment 6**. The crude oil assay data provided to me on the four oil samples included in this study are provided in **Attachment 7**.

Specific results most relevant to the study's objectives are presented in **Tables 2 to 4** and **Figures 2 to 6**. Discussion of these results is provided in the following sections.

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[6] Kienhaus, P.G.M. et al. 2016. CEN methodology for oil spill identification. In: *Standard Handbook of Oil Spill Environmental Forensics: Fingerprinting and Source Identification*, 2nd Ed., S.A. Stout and Z. Wang, Eds., Elsevier Publishing Co., Boston, MA, p. 685-728.

[7] Stout (2023) dated March 10, 2023; see above.

Character of the Yellow Rock Oils Studied and Their Comparison to 7B Cavern Oil

The four locally produced crude oils from the three Yellow Rock wells studied herein (Table 1) exhibit sufficiently similar characteristics that allow them to be described together in the following sections.

Tier 1 Character of the Yellow Rock Well Oils

Figure 2 shows the Tier 1 GC/FID (C8+) chromatograms for the four Yellow Rock oils studied herein (Fig. 2A-D). The chromatogram for the 7B cavern (reference) oil (re-)analyzed with these Yellow Rock oil samples is shown in Figure 2E for comparison.

Three of the four Yellow Rock oils' GC/FID chromatograms are dominated by a prominent unresolved complex mixture (UCM) that appears as a large, broad bimodal hump on the chromatograms (Figs. 2A-2C). One of these, Yellow Rock 23998, contains minor n-alkanes (Fig. 2A), which are absent in the both the shallow and deep oils from Yellow Rock 189416 (Fig. 2B-C). Acyclic isoprenoids (Pr and Ph) also are absent or virtually absent. The other prominent resolved peaks present in these three Yellow Rock oils are alkylated benzenes, decalins, and naphthalenes below ~C15 and triterpane biomarkers (norhopane and hopane) around ~C30 (Fig. 2A-C). These three Yellow Rock oils' Tier 1 features match those exhibited by the nine Yellow Rock well oils studied (and described) previously; see Footnotes 1, 4, and 5.

The Yellow Rock 41842 oil's GC/FID chromatogram, however, differs from the others studied herein and previously in that it contains prominent n-alkanes atop only a minor UCM (Fig. 2D). In this single aspect, it arguably resembles the 7B cavern oil although some differences are evident. For example, the Yellow Rock 41482 oil's prominent acyclic isoprenoids (Pr and Ph) occur in a (Pr/Ph) ratio of ~2.3 (Fig. 2D), which is clearly higher than the 7B cavern oil (~1.0; Fig. 2E; Table 2). Despite their presence, the n-alkanes in the Yellow Rock 41842 oil are likely reduced somewhat, as evidenced by the predominance of pristane and phytane. In addition, the Yellow Rock 41842 oil's chromatogram also exhibits the prominent norhopane and hopane peaks, which are not present in the 7B cavern oil. [Additional differences are discussed below in the Tier 2 data.]

Collectively, these Tier 1 features indicate that:

- The three oils collected in August 2023 from the Yellow Rock 253998 well and from both shallow and deep intervals of the Yellow Rock 189416 well are comprised of slightly varying, moderately biodegraded crude oils.
- The one oil collected in August 2023 from the Yellow Rock 41842 well is comprised of a slightly biodegraded crude oil.

Notably, biodegradation often tends to occur in shallower oil reservoir zones (where conditions for microbial activity are often favorable). This is notable because the Yellow Rock 41842 well is reportedly perforated and producing oil from 6718 to 6728', whereas the other Yellow Rock wells studied (Table 1), all of which contained more highly biodegraded oils, are perforated at shallower depths (roughly 2900' to 4300').[8] Its slightly biodegraded character distinguishes the Yellow Rock 41842 oil from other Yellow Rock well oils studied to date, which may be of significance in the event locally produced crude oil(s) ever become evident within cavern 7.

Tier 2 Character of the Yellow Rock Well Oils and Comparison to 7B Cavern Oil

[8] State of Louisiana on-line database: https://sonlite.dnr.state.la.us/sundown/cart_prod/cart_con_wellinfo1

Qualitative comparisons of the various PAH- and biomarker-based extracted ion profiles (EIPs; Attachment 6) yield no anomalous observations. Specifically, despite the less biodegraded nature of the 41842 oil, all the four Yellow Rock oils studied appear quite similar to one another and are distinct from the 7B cavern (reference) oil. This is unsurprising since the PAH- and biomarker-based compounds are utilized in oil identification studies because they are considered resistant to the effects of all but the most severe level(s) of biodegradation.

In regard to the first of these qualitative comparisons, **Figure 3** shows the partial (*m/z* 191) EIPs showing the distributions of terpenoid biomarkers in the four Yellow Rock oils (Fig. 3A-D) and in the 7B cavern (reference) oil studied herein (Fig. 3E). A general similarity among these oils' terpenoid biomarkers is evident.[9] However, close inspection reveals the proportions (as reflected by the relative size of the peaks) of different terpenoid biomarkers vary.

As was evident in earlier fingerprinting studies including Yellow Rock oils (Footnotes 1, 4, and 5), qualitative comparison of the terpenoid biomarker distributions in the four Yellow Rock oils studied herein shows they are highly comparable to one another (Fig. 3A-D) and are universally and unequivocally distinct from the 7B cavern (reference) oil (Fig. 3E). Specifically, the Yellow Rock oils exhibit relatively low abundances of tricyclic terpanes (T4 to T10), bisnorhopane (T14a), norhopane (T15), and homohopanes (T21 to T35) and relatively high abundances of oleanane (T19) and moretanes (T17 and T20; Fig. 3A-D). As was also observed in the earlier studies, the four Yellow Rock oils studied herein exhibit the unusual abundance of the 22R-bishomohopane epimer (T27; Fig. 3A-D). As described previously, this feature is atypical for most crude oils (worldwide; in my experience) and indicates the Sulphur Dome's locally produced crude oils likely contain a co-eluting and anomalous compound, i.e., a possible "marker" for locally produced crude oils. This warranted monitoring the diagnostic ratio (DR-32abS/32abR or T27/T26) that capitalizes on this unusual feature of the locally produced oils (Table 2; described below).

There is one new observation regarding the Yellow Rock oils studied herein, which is the presence of several 25-norhopanes (e.g., see T14b; Fig. 3A) in the Yellow Rock 253998 oil that are not present in the other Yellow Rock oils studied herein or those studied previously (Footnotes 1, 4 and 5). The presence of 25-norhopanes in the Yellow Rock 253998 oil was confirmed upon inspection of the *m/z* 177 EIP shown in **Figure 4** (Fig. 4A). These compounds are sometimes produced in biodegraded oils from the removal of a methyl group (C-10) from regular hopanes.[10] The seemingly unique (to date) presence of 25-norhopanes in the Yellow Rock 253998 oil (Fig. 3A and 4A) distinguishes this well's oil from other Yellow Rock oils studied (to date), which may be of significance in the event locally produced crude oil(s) ever become evident within cavern 7.[11]

Quantitative (statistical) comparisons among the samples are achieved using the 30 diagnostic ratios (DRs) calculated for the oils studied. These DRs are contained in **Table 2** wherein the 7B cavern (reference) oil collected in January 2023, which was re-analyzed as part of this study, is statistically compared to four Yellow Rock oils studied herein. As is described in Attachment 3, those DRs that are presently determined to be less precisely measured over both the short term and long term of the Sulphur Dome studies (per Table A3) are "greyed out" as they tend to exhibit higher standard error (RSD_r and RSD_R) under repeatability and/or reproducibility conditions using

[9] As explained in my March 10, 2023 report, this general similarity is unsurprising as many oils will contain terpenoid biomarkers derived from comparable suites of ancient organic matter that gave rise to the oil over geologic time. Oil fingerprinting relies upon "the details", not general similarities.

[10] Peters, K., et al. (2005) The Biomarker Guide, Vol. 2 (2nd Ed.), Cambridge Univ. Press, pp. 675-680.

the CEN protocol's 95% confidence level criteria.[12] The green and red color-coding in Table 2 reveals those DRs that statistically match (green) and statistically differ (red) from the 7B cavern (reference) oil analyzed herein.

As expected, based upon the disparate qualitative (Tier 1 and Tier 2) results versus those of the 7B cavern oil described above (Figs. 2-3), the four Yellow Rock crude oils studied herein show that most DRs are statistically distinct from those of the 7B cavern oil (Table 2). These multiple differences warrant a "non-match" conclusion per CEN protocol (Fig. 1) for the four Yellow Rock oils studied *versus* the 7B cavern oil. Therefore,

- The four locally produced Yellow Rock crude oils studied [from wells 253998, 41842, and 189416 (170' and 1250')] are unequivocally distinct from the 7B cavern oil.

Although the "non-match" conclusion between the four Yellow Rock oils and the 7B cavern oil studied is clearly expressed in Table 2, it is worthwhile to visually convey these – and all previous oils' – results in a graph format that may be easier to visualize. Toward this end, **Figure 5** was prepared to allow one to see the disparity that exists between the populations of 7B cavern oil and the Yellow Rock well oils analyzed to date using a variety of diagnostic ratio (double ratio) cross-plots. Also plotted in Figure 5 are the available results for the four surface sheen samples collected from Bubble Sites (20, 22, and 24) and the Central Lake that were analyzed previously (Footnotes 1,2,4, and 5). (All 7B cavern oil, Yellow Rock oil, and surface sheen samples plotted in Figure 5 are inventoried in Table 1.)

Inspection of Figure 5 shows:

- The population of 7B cavern oils are highly consistent with one another as demonstrated by their clustering on all of the diagnostic ratio cross-plots (Fig. 5A-H).[13]
- The population of locally produced Yellow Rock crude oils are only generally consistent with one another as demonstrated by their scatter in both their levels of biodegradation (Fig. 5A-B) and various source-specific features expressed by sulfur- (Fig. 5C) and biomarker-based diagnostic ratios (Fig. 5D-H).
- Of particular note is that the oil from Yellow Rock 253998 (and its duplicate) exhibits several diagnostic features that tend to distinguish it from the other Yellow Rock oils studied, including slightly higher relative abundances of moretane (Fig. 5E), bisnorhopane (Fig. 5F), C35-homohopanes (Fig. 5G), and the T27 unknown and C28-C29 tricyclics (Fig. 5H). In addition, this oil uniquely contains 25-norhopanes (see discussion above; Fig. 4). This well's oil thereby presently appears distinguishable from the other Yellow Rock oils studied,
- The population of locally produced Yellow Rock crude oils are, in all instances, clearly distinct from the 7B cavern oils studied to date (which were collected between January

[12] The quantitative (statistical) comparisons relied upon the 95% confidence level under conditions of repeatability ($r_{95\%}$) for each diagnostic ratio wherein:

$$r_{95\%} = 2.8 * RSD_r \text{ where } RSD_r = 5\% \text{ standard error, thus}$$

$$r_{95\%} = 14\%$$

If the $r_{95\%}$ between the measured diagnostic between two samples <14% the ratios were considered to statistically **match**, and *vice versa*. The comparable criterion ($R_{95\%}$) is used to compared precisely measured DRs under conditions of reproducibility (see Attachment 3).

[13] The minor scatter among the 7B cavern (reference) oil analyzed over time, which are indicated by the larger, open blue circles in Figure 5, helps visualize the long-term precision of the various DRs plotted.

and June 2023, with the January 2023 sample having been repeatedly analyzed over time).

It is anticipated that if locally produced crude oil(s) were to enter Cavern 7 the resulting "mixed" oils would exhibit diagnostic ratios that are intermediate between the two populations. Notably, with this in mind, the seemingly distinguishing features of the Yellow Rock 253998 oil (noted in the 3rd bullet above) cannot be explained by mixing of a more typical Yellow Rock oil with 7B cavern oil. In addition, Figure 5 also shows:

- The population of surface sheens are consistent with locally produced crude oils and inconsistent with 7B cavern oil.

This last bullet point is notable as it demonstrates the surface sheens studied to date are consistent with, i.e., derived from, locally produced crude oils and not from 7B cavern oil.

Crude Oil Assay Results – Summary to Date

The crude oil samples studied to date via chemical fingerprinting (Table 1) have been analyzed via standard crude oil assay.[14] (The assay data for the four crude oils studied herein are contained in Attachment 7. These data were collected by ERM and provided to me.) In addition, prior to any chemical fingerprinting, the assays for 7B cavern oil and Yellow Rock oil from well 189416 collected in November 2022 were both performed (and subsequently provided to me).

In addition to the assay data collected during the Westlake studies, in response to a FOIA request you made to the US DOE, I was provided with crude oil assay data for the Strategic Petroleum Reserve (SPR) oils from Caverns 2, 6, and 7 for samples collected in August, May, and June 1989, respectively. The SPR data included three MS Word Files and one MS Excel file, which were provided previously in Attachment 8 to an earlier report (Footnote 5).[15] Thus, these historic assay data would seemingly represent oils present in these caverns only shortly before the SPR facility at Sulphur Dome was poised to be decommissioned in 1990.[16] A separate report (available on-line) provides limited average API gravity and sulfur content data for the oils stored in these same caverns (but also explains Cavern 2 was actually connected to Caverns 4 and 5).[17]

Crude oil assays are normally obtained to provide "bulk" features of an oil that are most useful in assessing potential issues in an oil's handling/refining (i.e., its value). Although not as detailed as the (molecular-level) chemical fingerprinting results discussed above, some assay features, however, can provide a basis to determine the similarity/difference among oil samples.

Table 3 contains selected crude oil assay results for the 7B cavern oils, locally produced Yellow Rock oils, the few "other oils" from the site studied to date (Table 1), and for the historic (1989) SPR oils. The selected assay results include the API gravity, sulfur content, and nickel (Ni) and

[14] Assays of the Central Lake oil/sheen, and previously studied surface oils/sheens, were not obtained since their small volumes were insufficient for assay analysis.
[15] Note that there is a disparity between the data present in the Word file versus Excel file provided in the FOIA response for SM007 and SM006, but not for SM002. The reason for the disparity is unexplained.
[16] U.S. DOE (1990) Environmental assessment: Strategic Petroleum Reserve Sulphur Mines Decommissioning and Big Hill Expansion. US DOE report DOE/EA-0401, dated January 1990. Accessed online at: https://www.osti.gov/servlets/purl/7081078
[17] U.S. DOE (1989) Sulphur Mines Underground Storage Facility Description. US DOE report DOE/FE-0136, dated July 1989. Accessed online at: https://www.osti.gov/servlets/purl/7081078#:~:text=Sulphur%20Mines%20is%20one%20of,in%20three%20salt%20dome%20caverns.

vanadium (V) concentrations, the latter of which are used to calculate the V/(V+Ni) ratios listed. The basis and utility for comparing API gravity, sulfur content, and the ratio V/(V+Ni) also were discussed previously (Footnote 5). Most of the data in Table 3 were previously provided but the four new results presented herein are indicated by the red box in Table 3.

Although evident upon inspection of Table 3, the obvious disparity in these bulk features between the 7B cavern oils and the locally produced Yellow Rock oils studied to date is visually evident in **Figure 6**, which also includes the historic (~1989) results available for the SPR oils formerly stored in the Sulphur Dome caverns. Inspection shows the sulfur content, API gravity, and V/(V+Ni) ratio in the Yellow Rock oils are markedly lower than in the 7B cavern oils (Fig. 6).

There are no detailed chemical fingerprinting data available for the historic SPR oils from Sulphur Dome (to my knowledge). However, as was previously observed (Footnote 5), a comparison of the SPR oils' sulfur contents, API gravities, and V/(V+Ni) ratios shows them to be relatively comparable to those of the 7B cavern oils, with the former exhibiting slightly higher sulfur contents than the latter (Fig. 6).

While any attempt to interpret the details/scatter in Figure 6, especially in regard to the 30+ year-old 1989 SPR oil data, is imprudent,[18] it is perhaps notable that the six 7B cavern oils studied recently (between Nov. 2022 and June 2023; Table 4) appear highly consistent (tightly clustered) whereas the locally produced Yellow Rock oils are clearly scattered (Fig. 6). There is an inverse correlation (r^2=0.74) between API gravity and sulfur content of the Yellow Rock oils (Fig. 6A), which is reasonably attributable to the varying levels of biodegradation among the locally produced crude oils. In this regard it is perhaps notable that the Yellow Rock 253998 oil studied herein appears more highly biodegraded than the other oils (Fig. 6A) – and this oil also uniquely contained 25-norhopanes (see above; Fig. 4).[19]

In summary, the available "bulk" crude oil assay data show:

- The 7B cavern oil has remained consistent (between November 2022, when it was first analyzed, and June 2023) and is clearly distinguishable from the locally produced, Yellow Rock oils.
- The Yellow Rock oils exhibit some heterogeneity mostly attributable to the varying levels of biodegradation among the locally produced oils.
- The 7B cavern oils exhibit "bulk" features generally consistent with SPR oil(s) historically stored in the Sulphur Dome caverns.

The latter of these conclusions tends to support the apparent predominance of residual SPR oil presently with Cavern 7, although some contribution of a non-local crude oil blanket cannot be ruled out.[20]

[18] This is because the precision of these bulk feature data is unmeasured.
[19] The presence of some n-alkanes in the Yellow Rock 253998 oil (Fig. 2A) along with 25-norhopanes (Fig. 4A) may indicate this well's reservoir zone contains a multiple charges of oil, e.g., a fresher oil charge arriving more advanced biodegradation of an earlier oil charge.
[20] See Footnote 14 in the July 25, 2023 report (per Footnote 5 herein). This footnote concludes the cavern 7B oil is most likely predominantly (perhaps even exclusively) comprised of residual SPR oil formerly stored in the cavern. However, some component of a non-local crude oil used as an oil blanket within the cavern may also be present and mixed with residual SPR oil.

Summary of Findings – Presently and To Date

Chemical fingerprinting of four additional locally produced crude oils collected from three Yellow Rock wells [SN 253998, 41842, 189416 (1250'), and 189416 (170')] on August 17 or 29, 2023 expanded the population of locally produced crude oil studied to 13 oils from nine different Yellow Rock wells and the Yellow Rock tank battery.

The four new Yellow Rock oils' results are comparable to those collected in January (110159), May (185997, 209459, 210185, and tank battery), and June 2023 (109963, 185997, 209459, and 252112). Collectively,

- The 13 locally produced (Yellow Rock) crude oils fingerprinted to date consist of slightly-to-moderately biodegraded crude oils. There is some variability in the level of (in-reservoir) biodegradation among these oils, which is not unexpected for oils produced from different reservoir zones.
- The current study found that crude oil the deepest reservoir zone studied to date (Yellow Rock 41482 at ~6720') is only slightly biodegraded whereas other oils apparently obtained from shallower reservoir zones (roughly 2900' to 4300') are varying, but moderately biodegraded.
- The current study also found that crude oil from Yellow Rock 253998 uniquely contains distinct biomarkers only formed under specific in-reservoir biodegradation process/conditions (i.e., 25-norhopanes). Their presence renders this well's oil distinct from the other Yellow Rock oils studied to date, which may be of significance in the event locally produced crude oil(s) ever become evident within cavern 7.

The source-specific (weathering-independent) fingerprinting features among the four new and previously-studied, locally produced Yellow Rock crude oils are only slightly varying from one another. This variability demonstrates that:

- There is only subtle heterogeneity in the source-specific features among the locally produced crude oils present in the area's different wells/reservoir zones. However, the crude oil from Yellow Rock 253998 exhibits multiple features that (along with its 25-norhopanes; described above) subtly distinguish it from the other Yellow Rock oils.

Despite the heterogeneity among the locally produced crude oils they are all unequivocally distinct from those of the 7B cavern oil, i.e.,

- The 7B cavern oil is unequivocally not a locally produced oil. As such, the Yellow Rock oils studied are easily distinguished from and are clearly statistical "non-matches" to the 7B cavern oils studied to date, the last of which was sampled on June 16, 2023.
- The available data indicate that the non-local crude oil comprising the 7B cavern oil is most likely predominantly (perhaps even exclusively) comprised of residual SPR oil formerly stored in the cavern.

The "bulk" crude oil assay data on the four new Yellow Rock oils studied – as well as for previously-studied Yellow Rock and 7B cavern oils and historic data for the Sulphur Dome SPR oils from ~1989 – support the conclusions based on the detailed chemical fingerprint summarized above. An inventory of some of these "bulk" and "detailed fingerprinting" features are summarized in **Table 4**. If, at some point in time, local crude oil was to enter and mix with the extant 7B cavern oil the resulting mixture(s) would be expected to It is anticipated that if locally produced crude oil(s) were to enter Cavern 7 the resulting "mixed" oils would be anticipated to exhibit both "detailed fingerprinting" and "bulk" features that are intermediate to those given in

Table 4 and visually evident in Figures 5 and 6. Any such mixing cannot explain the multiple, subtly different features of the Yellow Rock 253998 oil noted above.

Although no new data for surface oils/sheens were collected in this study, the available (and new Yellow Rock oil) chemical fingerprinting results continue to show:

- Oil/sheens collected from the multiple surface locations studied to date indicate they are derived from (seepage or spillage of) local crude oil(s), and not from 7B cavern oil leaked from Cavern 7.

Please let me know if you have any questions.

Sincerely,

Scott A. Stout, Ph.D., P.G.
Sr. Geochemist

Attachments:
1: Chain-of-custody
2: Analytical Methods
3: Interpretation Methods
4: Tabulated concentrations of TPH/SHC, PAH, and biomarkers
5: Full size GC/FID chromatograms
6: Selected GC/MS extraction ion profiles
7: Bulk assay data for the samples studied

Table 1: Inventory of samples from the current study and studied previously.

Current Study Samples

Client/ Field ID	Lab ID	Matrix	Date Collected	Description of Sample
253998*	L2348036-01	Oil	6/16/2023	Yellow Rock 253998
41842	L2348036-02	Oil	6/16/2023	Yellow Rock 41842
189416 (1250')	L2348036-04	Oil	6/16/2023	Yellow Rock 189416 from 1250' (bottom of oil column)
189416 (170')	L2348036-05	Oil	6/16/2023	Yellow Rock 189416 from 170' (top of oil column)
7B**	L2348036-03	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)

Previously-Studies Samples

Client/ Field ID	Lab ID	Matrix	Date Collected	Description of Sample
Pad Oil	L2335058-01	Oil	6/16/2023	Stock tank oil used as cavern blanket/pad
7B*	L2335058-02	Oil	6/16/2023	Cavern oil from brine well 7B
252112	L2335058-03	Oil	6/16/2023	Yellow Rock 252112
109963	L2335058-04	Oil	6/16/2023	Yellow Rock 109963
185997	L2335058-05	Oil	6/16/2023	Yellow Rock 185997
209459	L2335058-06	Oil	6/16/2023	Yellow Rock 209459
Sheen	L2335058-07	Net	6/12/2023	Surface sheen from central lake
7B**	L2335058-08	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
209459	L2325505-01	Oil	5/2/2023	Yellow Rock 209459
185997	L2325505-02	Oil	5/2/2023	Yellow Rock 185997
CAVERN 4	L2325505-03	Oil	5/25/2023	Cavern oil from brine well PPG No. 4
CAVERN 7B*	L2325505-04	Oil	5/25/2023	Cavern oil from brine well 7B
210185	L2325505-05	Oil	5/25/2023	Yellow Rock 210185
TANK BATTERY	L2325505-06	Oil	5/25/2023	Yellow Rock Tank Battery
7B**	L2325505-07	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
BS-24	L2325505-08	Net	5/22/2023	Surface sheen from bubble site No. 24
CAVERN 7B*	L2317387-01	Oil	3/30/2023	Cavern oil from brine well 7B
No. 20	L2313362-01	Net	3/9/2023	Surface oil sheen on water body west of the salt dome
7B*	L2305221-04	Oil	1/25/2023	Cavern oil from brine well 7B
110159	L2305221-02	Oil	1/25/2023	Yellow Rock 110159
Stock Tank	L2305221-03	Oil	1/25/2023	Stock tank oil used as cavern blanket/pad
Brine Well 22 BS*	L2305221-01	Net	1/25/2023	Surface oil brine well 22 excavation
Central Pond	L2305221-05	Net	1/25/2023	Surface sheen from central pond

* sample prepared and analyzed in duplicate

**re-analysis of Jan. 25, 2023 oil (L2305221-04) for quality control only

Table 2: Diagnostic ratios for the 7B cavern oil collected in January 2023 (re-analyzed herein) *versus* the four Yellow Rock oils studied herein (Table 1).

Top three ratios are derived from Tier 1 GC/FID data; all others from Tier 2 GC/MS data.

CEN - Diagnostic Ratios	CEN Diagnostic Ratios per Alpha Abbreviations	7B Cavern Oil (Jan 2023)	Yellow Rock 253998	Yellow Rock 253998 (Dup)	Yellow Rock 41842	Yellow Rock 189416 (1250')	Yellow Rock 189416 (170')
	Analysis Date	9/20/2023	9/19/2023	9/19/2023	9/20/2023	9/21/2023	9/20/2023
NR-C17/pris	C17/Pr	2.58	2.42	2.26	0.62	0.44	0.37
NR-C18/phy	C18/Ph	2.16	5.43	5.18	2.22	1.62	1.25
NR- pris/phy	Pr/Ph	0.94	2.31	2.25	3.76	2.12	1.91
NR-4-MD/1-MD	4-MDBT/1-MDBT	2.41	3.39	3.30	3.45	3.66	3.65
NR-2-MP/1-MP	2-MP/1-MP	1.05	1.46	1.45	0.84	1.15	1.16
NR-27Ts/30ab	T11/T19	0.23	0.23	0.23	0.14	0.15	0.15
NR-27Tm/30ab	T12/T19	0.26	0.30	0.30	0.20	0.21	0.20
NR-28ab/30ab	T14a/T19	0.17	0.09	0.09	0.04	0.05	0.05
NR-29ab/30ab	T15/T19	0.83	0.66	0.64	0.67	0.61	0.60
NR-30O/30ab	T18/T19	0.06	0.13	0.14	0.13	0.10	0.10
NR-31abS/30ab	T21/T19	0.57	0.26	0.26	0.24	0.26	0.25
NR-27dbR/27dbS	S4/S5	0.50	0.69	0.59	0.65	0.56	0.67
NR-27bb/29bb	(S14+S15)/(S26+S27)	0.84	0.69	0.74	0.71	0.72	0.72
NR-SC26/ RC26+SC27	TAS09/TAS01	0.14	0.32	0.32	0.33	0.35	0.34
NR-SC28/RC26 + SC27	TAS02/TAS01	0.71	0.92	0.87	0.74	0.81	0.83
NR-RC27/RC26+ SC27	TAS03/TAS01	0.77	0.64	0.63	0.61	0.64	0.61
NR-RC28/RC26+SC27	TAS04/TAS01	0.60	0.67	0.66	0.62	0.68	0.67
DR-Ts/Tm	T11/T12	0.89	0.75	0.76	0.68	0.72	0.73
DR-29Ts30ab	T16/T19	0.21	0.33	0.33	0.20	0.20	0.19
DR-29bb/29aa	(S26+S27)/(S25+S28)	1.26	1.69	1.73	0.64	0.97	1.04
DR-C2-dbt/C2-phe	DBT2/PA2	2.23	0.28	0.28	0.33	0.30	0.30
DR-C3-dbt/C3-phe	DBT3/PA3	2.37	0.34	0.34	0.39	0.39	0.38
DR-C28C29/30ab	T7 to T10/T19	0.26	0.13	0.14	0.05	0.07	0.06
DR-29aaS/29aaR	S25/S28	1.81	1.22	1.05	1.18	1.13	1.41
DR-C20TA/C21TA	TAS05/TAS06	1.35	1.14	1.22	1.23	1.30	1.32
DR-TA21/ RC26+SC27	TS06/TAS01	0.41	0.24	0.21	0.13	0.25	0.23
DR-C24Tet/C26Tri	T6a/T19	1.61	1.19	1.39	1.93	1.59	1.61
DR-30ba/30ab	T20/T19	0.08	0.25	0.25	0.18	0.21	0.20
DR-35ab/30ab	(T34 to T35)/T19	0.36	0.10	0.09	0.05	0.07	0.06
DR-32abR/32abS	T27/T26	0.72	2.00	1.98	1.99	1.51	1.49
		Conclusion:	**Non-Match**	**Non-Match**	**Non-Match**	**Non-Match**	**Non-Match**

red: statistical non-match to 7B Cavern Ref. Oil (analyzed concurrently)

green:s statistical match to 7B Cavern Ref. Oil (analyzed concurrently)

grey: indicates less precision ratio (per Attachment 3)

Dup: sample prepared and analyzed in duplicate

Table 3: Selected crude oil assay results for the 7B cavern oils, Yellow Rock (locally produced) crude oils, and Other oils from Sulphur Dome along with historic data for Strategic Petroleum Reserve oils from Sulphur Dome from 1989. The new data obtained on samples studied herein are contained within the red box.

na – not analyzed

	Client ID	Date Collected	API gravity	Sulfur (wt%)	V (ppm)	Ni (ppm)	V/ (V+Ni)
	7B Cavern Oils						
Cavern Oils	7B Cavern Oil	11/2/2022	32.8	1.38	23	6.1	0.79
	7B Cavern Oil	1/18/2023	34.0	1.40	23	5.9	0.79
	7B Cavern Oil	1/25/2023	na	na	12	3.8	0.76
	7B Cavern Oil	3/30/2023	33.6	1.37	100	26	0.79
	7B Cavern Oil	5/25/2023	33.5	1.40	23	6	0.79
	7B Cavern Oil	6/16/2023	34.0	1.35	25	6	0.81
		Average	33.6	1.38	34	9.0	0.79
		St. Dev.	0.5	0.02	32	8.4	0.01
	Yellow Rock (Locally Produced) Oils						
Yelllow Rock Oils	189416	11/2/2022	26.0	0.302	1.2	7.0	0.15
	110159	1/25/2023	na	na	0.4	3.7	0.10
	209459	5/2/2023	22.8	0.435	2.0	8.0	0.20
	185997	5/2/2023	21.5	0.407	2.0	9.0	0.18
	210185	5/25/2023	22.8	0.476	2.0	10.0	0.17
	Tank Battery	5/25/2023	27.0	0.327	1.0	6.0	0.14
	252112	6/16/2023	27.7	0.295	1.0	5.0	0.17
	109963	6/16/2023	24.1	0.431	2.0	8.0	0.20
	185997	6/16/2023	23.0	0.411	2.0	10.0	0.17
	209459	6/16/2023	21.6	0.433	2.0	9.0	0.18
	253998	8/17/2023	16.9	0.747	2.3	9.6	0.19
	41842	8/17/2023	26.6	0.403	2.0	22.2	0.08
	189416 (1250')	8/29/2023	20.7	0.450	<0.1	10.9	<0.01
	189416 (170')	8/29/2023	20.8	0.447	<0.1	8.3	<0.01
		Average	23.2	0.428	1.7	9.1	0.16
		St. Dev.	3.1	0.112	0.6	4.3	0.04
	Other Oils						
Other	Stock Tank	1/25/2023	na	na	19.3	4.7	0.80
	Cavern 4	5/25/2023	31.2	1.55	42	9	0.82
	Pad Oil	6/16/2023	29.3	1.27	18	5	0.78
	Sulphur Mines Strategic Petroleum Reserve Oils						
SPR Oils	SM007	6/14/1989	31.9	1.69	23.1	10.1	0.70
	SM007*	6/14/1989	32.4	1.75	32.7	9.0	0.78
	SM006	5/31/1989	31.0	1.42	34.0	20.6	0.62
	SM006*	5/31/1989	32.9	1.56	41.6	11.6	0.78
	SM002	8/14/1989	32.7	1.63	52.8	10.6	0.83
	Cavern 7	1989 rpt.	32.5	1.80	na	na	na
	Cavern 6	1989 rpt.	32.7	1.60	na	na	na
	Cavern 2-4-5	1989 rpt.	32.9	1.60	na	na	na

* sample values from Word files provided by DOE; other SM### sample values from Excel file provided

Table 4: Comparison of bulk and detailed fingerprinting features presently evident between 7B cavern oils and locally produced (Yellow Rock) oils studied to date.

	7B Cavern Oil		Yellow Rock Oils	
Bulk Features [1]	Avg.	σ	Avg.	σ
API gravity	33.6	0.5	23.2	3.1
Sulfur content (wt%)	1.38	0.02	0.43	0.11
V/(V+Ni) ratio	0.79	0.01	0.16	0.04
Fingerprinting Features [2]				
Biodegradation	none		slight-to-moderate[3]	
Pristane/Phytane	~1.0		>2.0	
Relative sulfur content (DBTs)	higher		lower	
Notable Biomarker Abundances				
Tricyclics (T4 to T10)	moderate		trace	
Bisnorphopane (T14a)	moderate		trace	
Norhopane (T15)	higher		lower	
Moretanes (T17 and T20)	low		moderate	
Oleanane (T18)	trace/absent		low	
Homohopanes (T21-T35)	high		low	
Unknown (T27)	absent		moderate	

[1]Data from Table 3.

[2]Fingerprinting data for all samples collected to date

[3]Slight biodegradation only evident in deeper reservoired oil (Yellow Rock 41842)

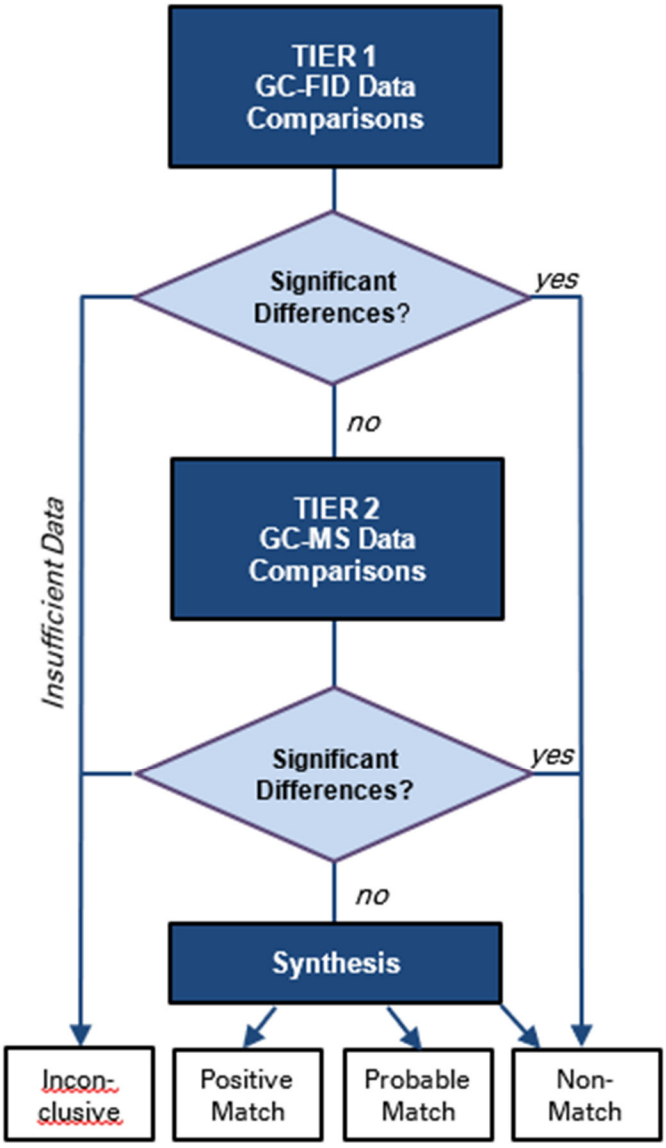

Figure 1: Simplified flowchart depicting the CEN (2012) oil spill identification protocol.

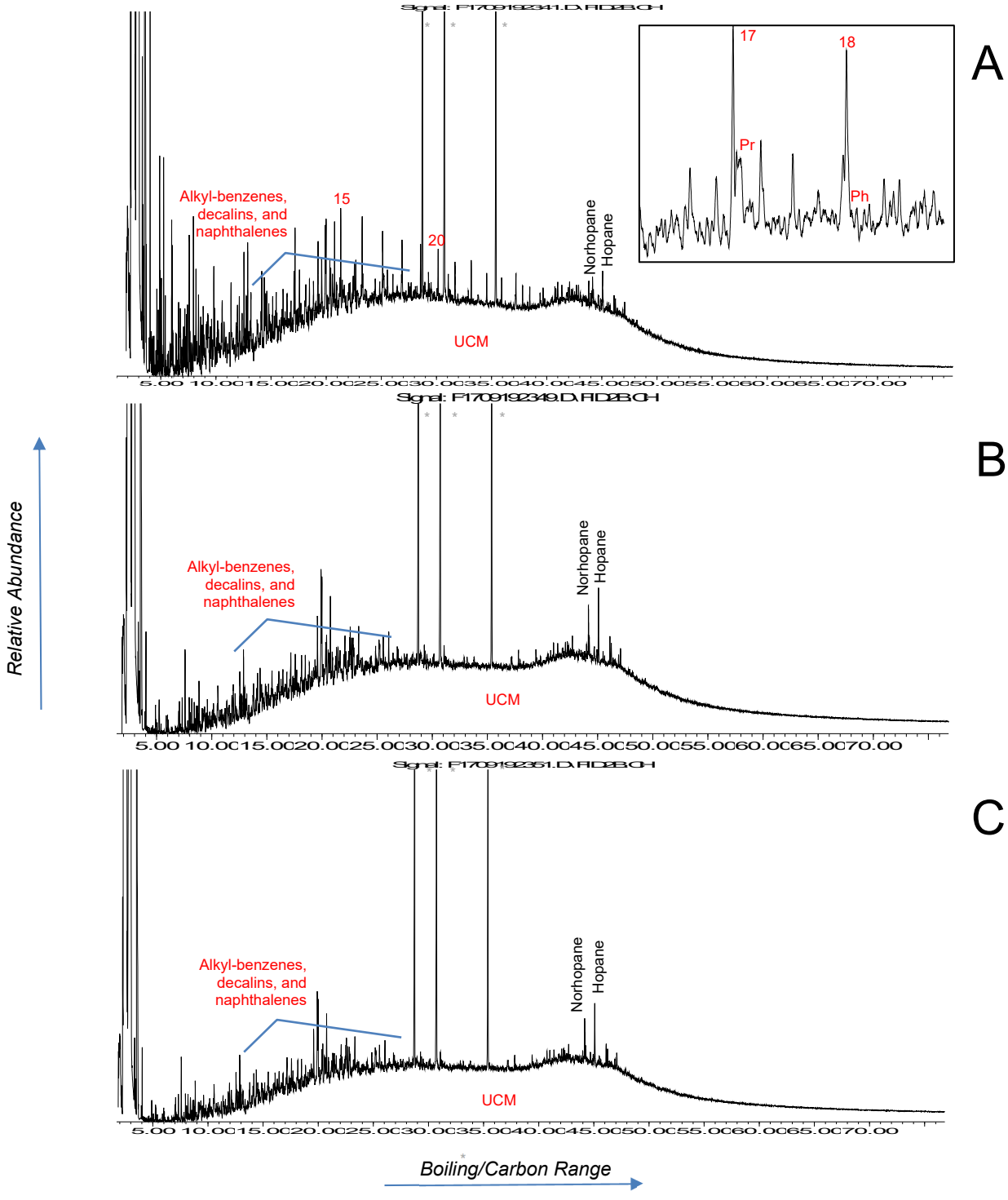

Figure 2: GC/FID (C8+) chromatograms for (A) Yellow Rock 253998 oil, (B) Yellow Rock 189416 (1250') oil, (C) Yellow Rock 189416 (170') oil, (D) Yellow Rock 41842 oil, and (E) Cavern 7B oil (January 2022, re-analyzed in Sept. 2022) studied herein. Insets show further expanded view of C17-C18 range. #: n-alkane carbon number; Pr: pristane; Ph: phytane; UCM: unresolved complex mixture; *: internal standard.

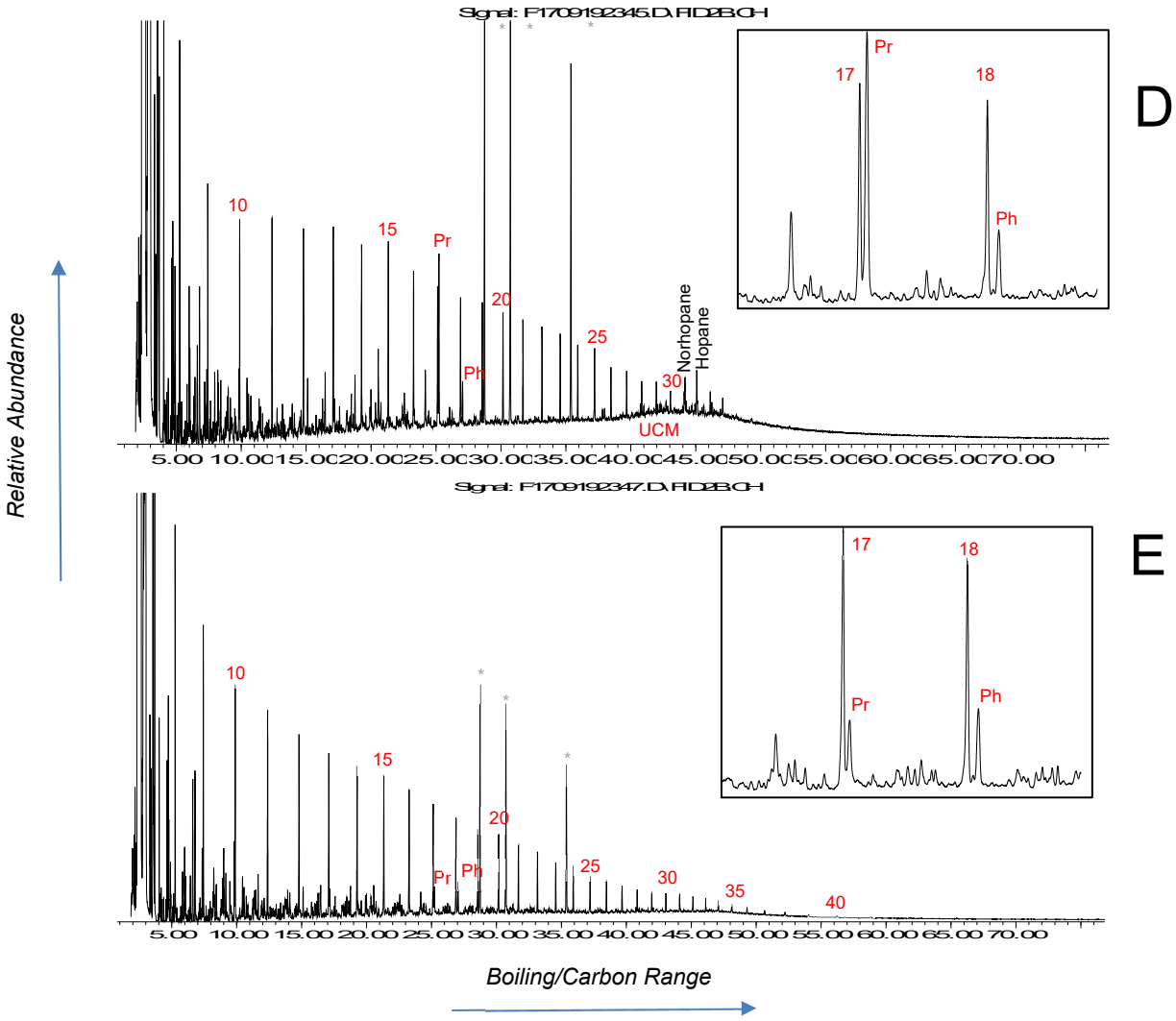

Figure 2: continued

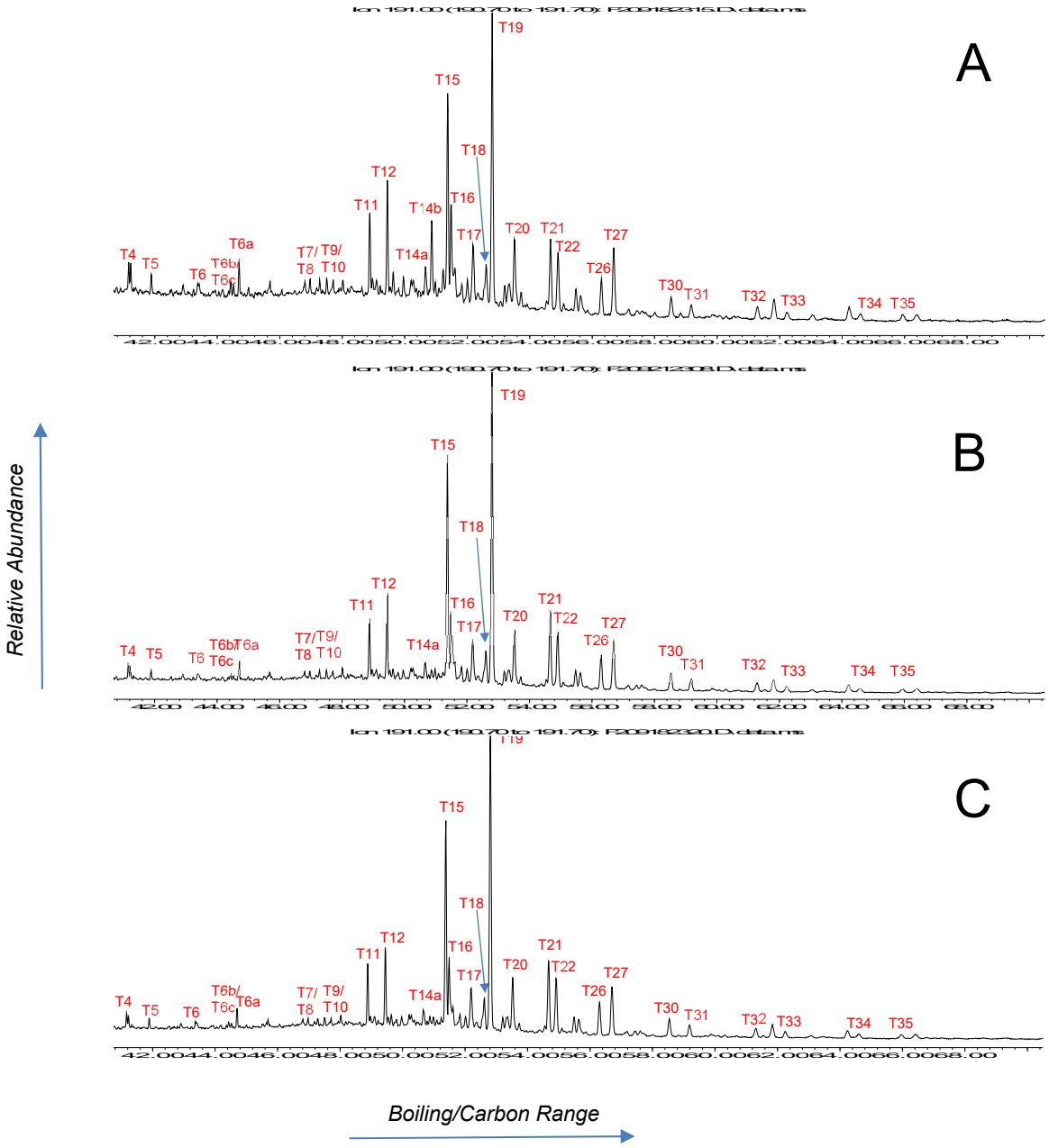

Figure 3: Partial extracted ion chromatograms (*m/z* 191) for (A) Yellow Rock 253998 oil, (B) Yellow Rock 189416 (1250') oil, (C) Yellow Rock 189416 (170') oil, (D) Yellow Rock 41842 oil, and (E) Cavern 7B oil (January 2022, re-analyzed in Sept. 2022) studied herein. red labels: various triterpane biomarkers, see Attachment 4, Table 4-2 for compound names.

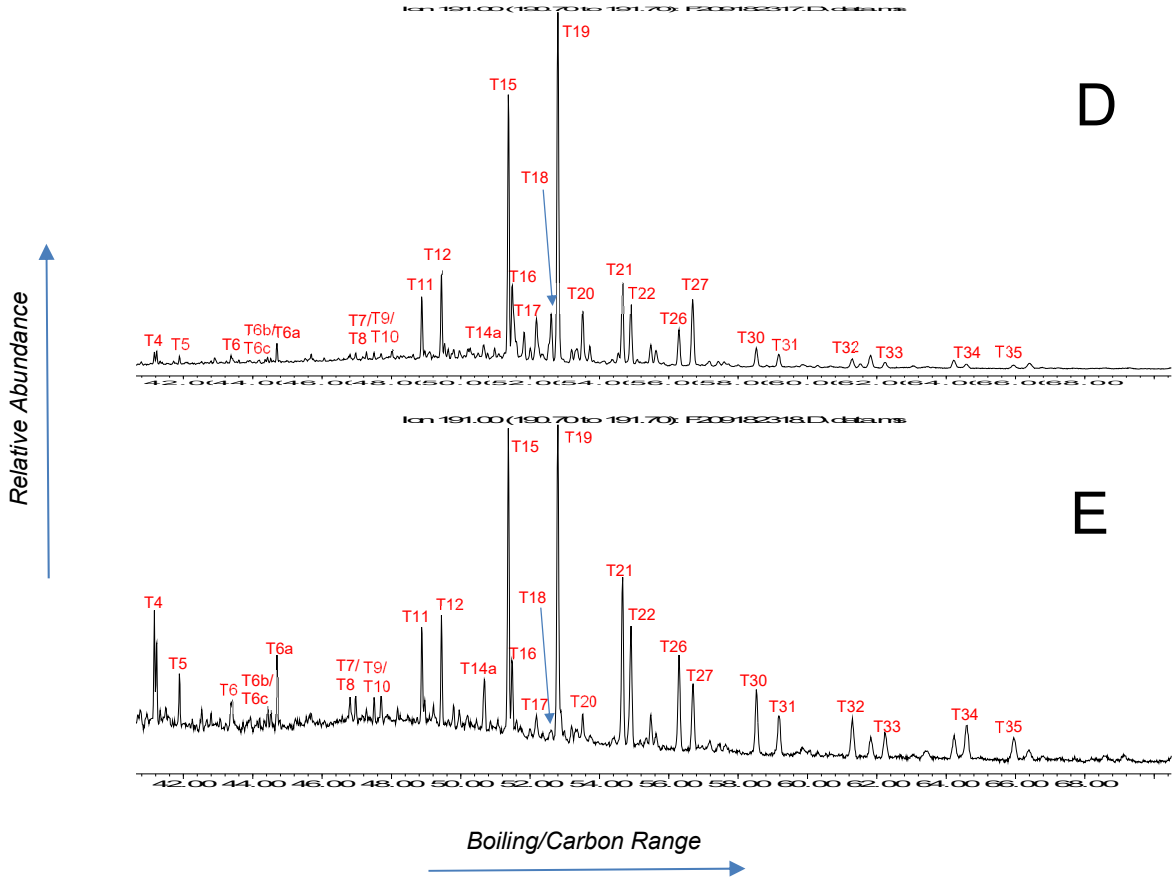

Figure 3: continued

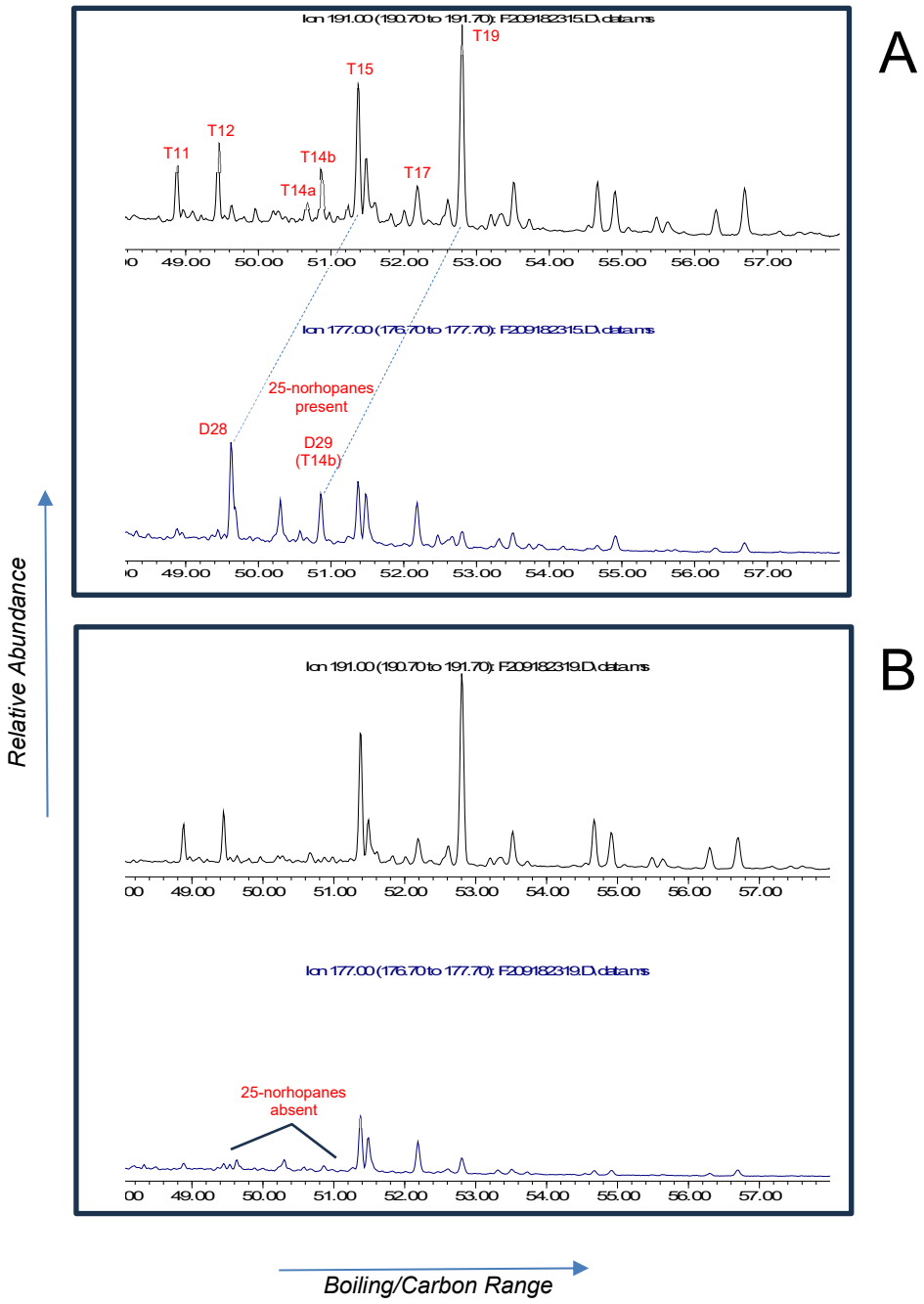

Figure 4: Stacked partial extracted ion chromatograms (*m/z* 191 and *m/z* 177) for (A) Yellow Rock 253998 oil and (B) Yellow Rock 189416 (170') oil showing the unique presence of 25-norhopanes (D27, D28, and D29, the latter measured as T14b) in (A). red labels: various triterpane biomarkers, see Attachment 4, Table 4-2 for compound names.

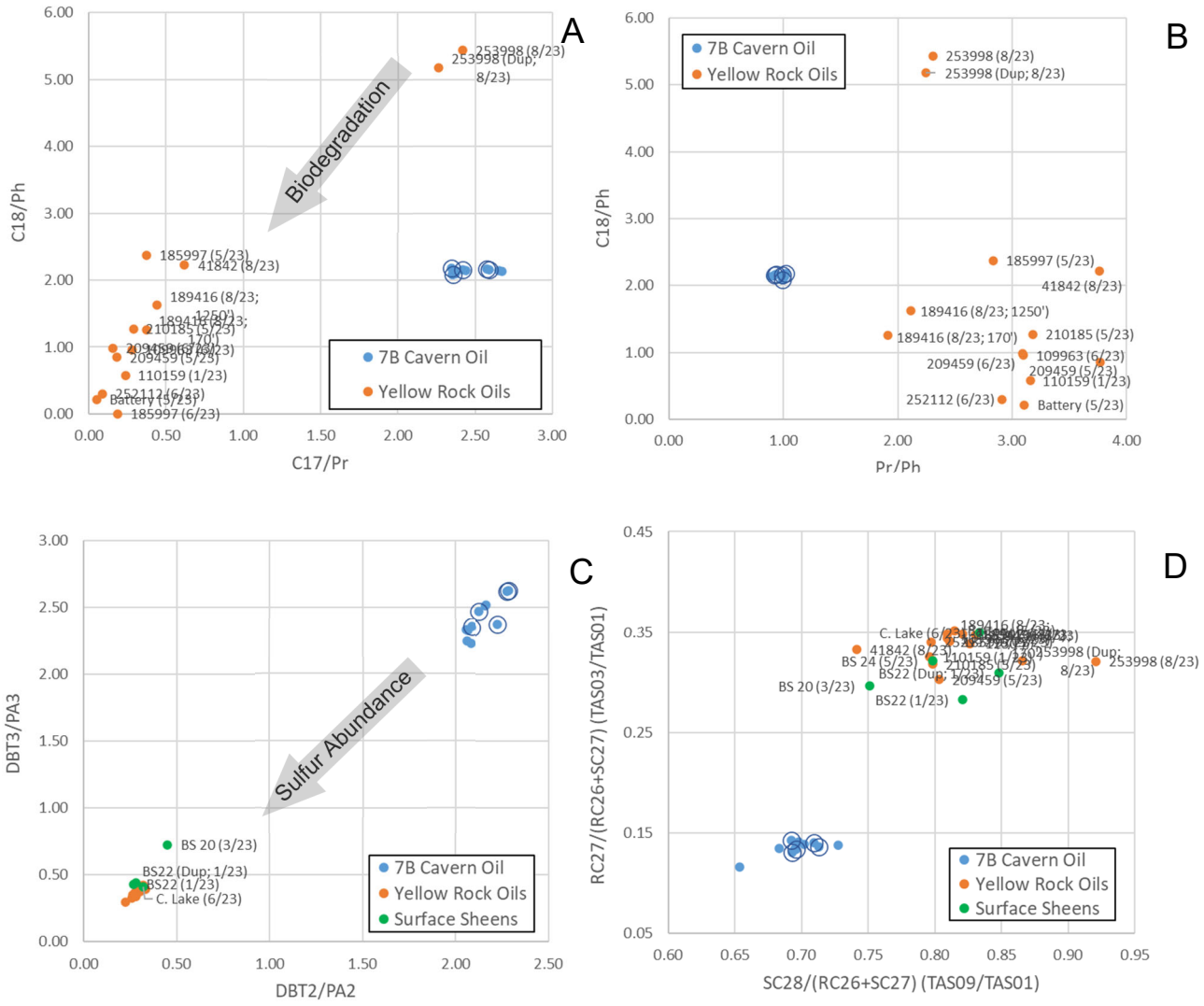

Figure 5: Cross-plots of select diagnostic ratios for all oil and sheen samples studied to date that visually conveys the disparity between 7B cavern oil and Yellow Rock well oils. The few (Bubble site or Central Lake) surface sheens studied previously are included and demonstrate their general comparability to the Yellow Rock oil population. 7B cavern oil points within open circles are the January 2023 reference oil re-analyzed over time, which visually demonstrates the long-term reproducibility of these data.

(A) isoprenoid/ n-alkane biodegradation indices, (B) acyclic isoprenoid indices, (C) relative sulfur abundance indices, (D) triaromatic steroid indices, (E) hopanoid indices, (F) oleanane and bisnorhopane indices, (G) homohopanes abundance indices, and (H) tricyclic terpane and unknown "marker" (at T27) indices. All ratio data are contained in this or previous reports. Note: Ratios in (A) and (B) are not available for surface sheens owing to their severe levels of evaporation. See text for descriptions.

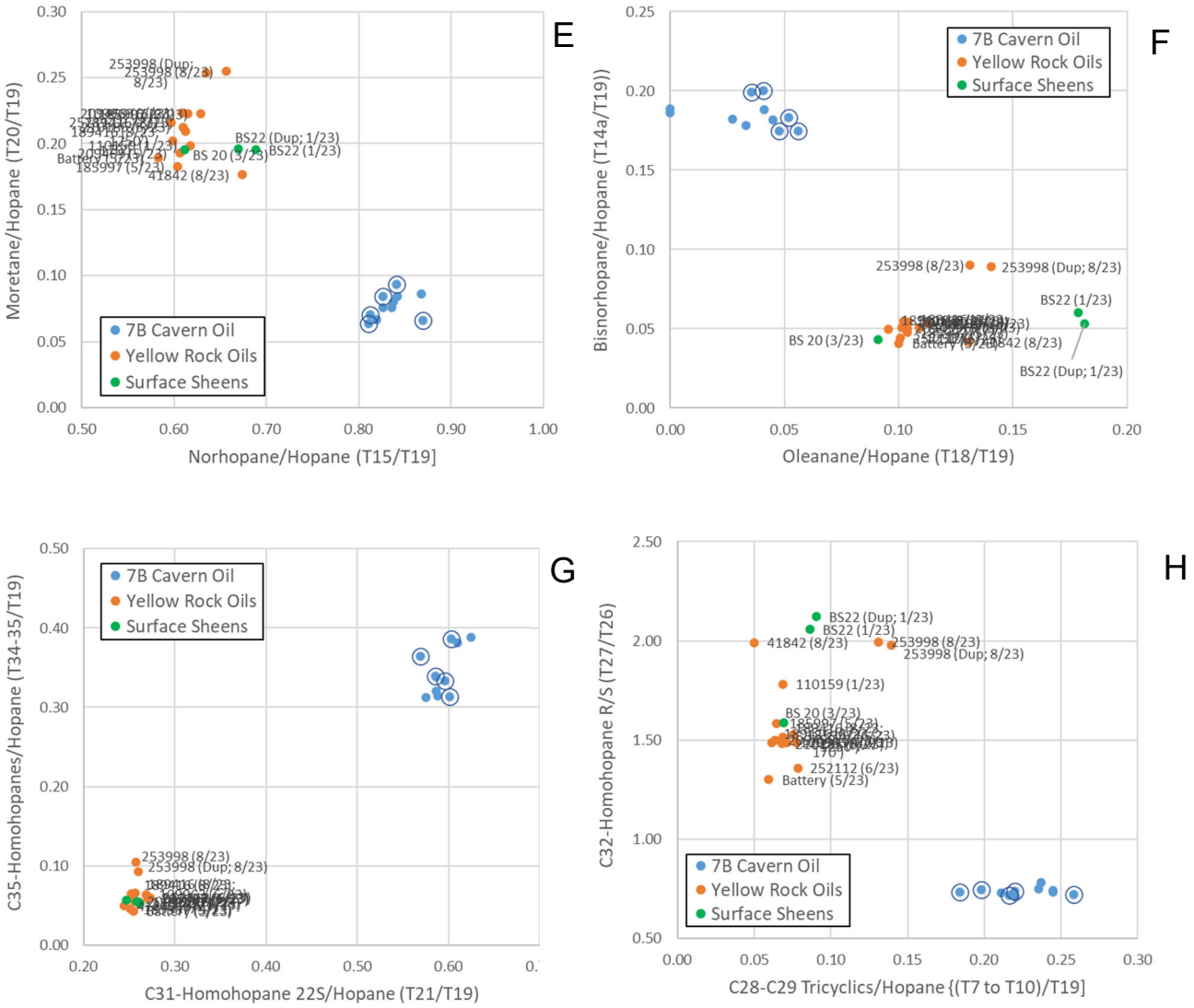

Figure 5: continued

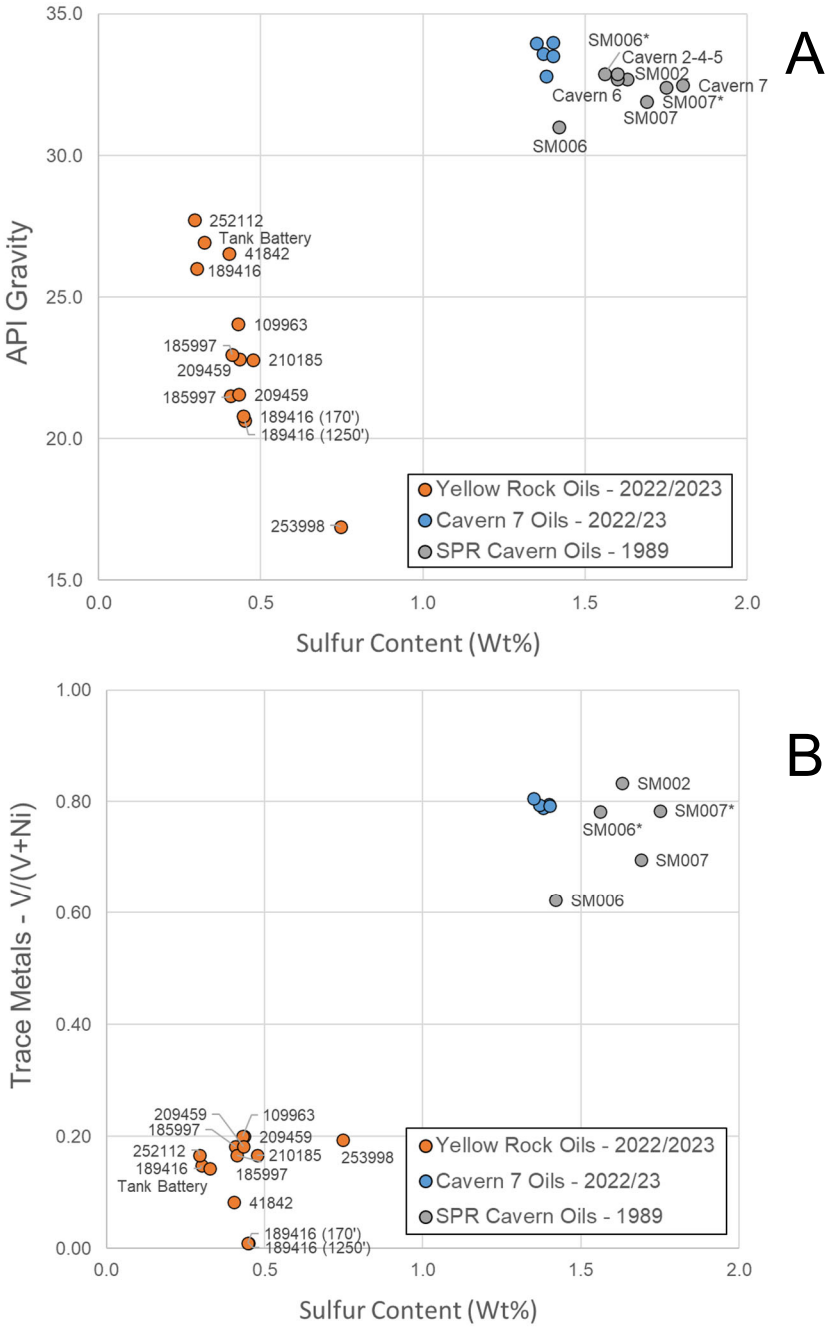

Figure 6: Cross-plots of crude oil assay results for the Cavern 7 oils and Yellow Rock (locally produced) oils recently studied and historic SPR cavern oils from 1989. All data from Table 3.

ATTACHMENTS

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Chain of Custody

Environmental Forensics Practice LLC

Proj. No	Proj. Name
070.093	Westlake

SAMPLERS: Signature

DATE	TIME	LAB ID	CLIENT ID	SAMPLE DESCRIPTION	MATRIX (* see below)	GC-FID-TPH (C_6+)	GCMS-Alkyl PAH	GCMS-Biomarkers	PIANO - VOA	Organic Lead	METALS	PCB	Pesticides	PRESERVED	Total Number of Containers
8/7/23	1030		253998	Oil/liquid		X	X		X						2
8/7/23	1050		41842	Oil/liquid		X	X		X						2

ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS"

Relinquished by:	Date/Time	Received by:	Date/Time
	8/17/23 1440	Fedex	
Relinquished by: Fedex	Date/Time	Received by:	Date/Time 8/18/23 10:05
Relinquished by:	Date/Time	Received by:	Date/Time

* O=Oil
SO=Soil
SE=Sediment
T=Tissue
W=Water

Samples to be shipped to: Alpha Laboratory
320 Forbes Blvd.
Mansfield, MA 02048
Tel: (508) 844-4117
Attn: Sue O'Neil

Comments: ERM
SCOTT HIMES - 832 - 209 - 8811
840 W. SAM HOUSTON PKWY N, Suite 600
HOUSTON, TX 77024

Chain-of-Custody

Attachment 1

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Chain of Custody

Environmental Forensics Practice LLC

Proj. No	Proj. Name
0701093	Sulphur Dome

SAMPLERS: Signature

Scott Himes

DATE	TIME	LAB ID	CLIENT ID	SAMPLE DESCRIPTION	MATRIX (* see below)	GC-FID-TPH (C_6-C_{44})	GCMS-Alkyl PAH	GCMS-Biomarkers	PIANO - VOA	Organic Lead	METALS	PCB	Pesticides	PRESERVED	Total Number of Containers
				ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS"											
8/29/23	0915		SN 189416 (1,250')	Oil	O	X	X		X						2
8/29/23	0930		SN 189416 (170')	Oil	O	X	X		X						2

Relinquished by:	Date/Time	Received by:	Date/Time
	8/30/23 1400	FEDEX	
FEDEX	8/31/23 10:19		8/31/23 10:19

* O=Oil
SO=Soil
SE=Sediment
T=Tissue
W=Water

Samples to be shipped to:
Alpha Laboratory
320 Forbes Blvd.
Mansfield, MA 02048
Tel: (508) 844-4117
Attn: Sue O'Neil

Comments:

Contact Scott Stout for further details.

Attachment 2

Analytical Methods

Sample Preparation

An aliquot (~100 mg) of each oil sample was diluted in dichloromethane (DCM: 10 mg/mL). A 1.0 mL aliquot of the extract was then spiked with recovery internal surrogates (RIS; 5α-androstane, acenaphthene-d_{10}, chrysene-d_{12}) and surrogate internal standards (SIS; o-terphenyl, n-tetracosane-d_{50}, 2-methylnaphthalene-d_{10}, pyrene-d_{10}, benzo(b)fluoranthene-d_{12}, and 5β(H)-cholane) prior for instrument analysis. Net samples were spiked with RIS and serially-extracted (3x) using fresh DCM on a shaker table. The extracts were combined, passed through glass wool, dried with sodium sulfate, concentrated to 1.0 ml, and spiked with SIS prior to instrument analysis. No silica-gel cleanup of the sample extracts was performed.

Each analytical batch included a procedural blank (PB; 1 mL of DCM), a laboratory control sample (LCS) and LCS duplicate (LCSD), each consisting of 1 mL of DCM spiked with selected hydrocarbons in known concentrations to monitor method accuracy, a reference (North Slope) crude oil standard, and at least one sample duplicate (i.e., a single oil prepared twice) as a measure of short-term precision of the data.

In addition, owing to the increasing longevity of this program, the original Cavern 7B oil collected on January 25, 2023 (L2305221-04) was prepared for use as a project-specific reference oil that can be analyzed with each batch of samples hereafter. An aliquot (~50 mg) of the archived oil was diluted with ~ 2-3 mL of DCM, spiked with RIS and SIS and further diluted to 10 mL with DCM. The diluted oil was then immediately transferred to a 20 mL glass vial (Teflon cap). Aliquots (~100 μL) of this reference oil are transferred to a GC vial with a low volume insert and aluminum crimp cap for analysis. Each reference oil sub-sample is given a new lab ID within the current batch of samples being analyzed.

Sample Instrument Analysis

Two analytical methods were employed in the chemical analysis of the extracts. These methods are routinely employed in oil spill investigations and are modifications of US EPA methods. The modifications include; (1) expansion of the prescribed target analyte lists to include many additional (conventionally, non-target analyte) hydrocarbons that are useful in distinguishing differences between and changes in petroleum after its release into the environment and (2) increasing the sensitivity of the instrumentation used through adjustments that lower the method detection limit (MDL) for targeted analytes providing few “non-detections” among the results.

In brief, the samples were analyzed using a (1) modified EPA Method 8015B and (2) modified EPA Method 8270D as described in the following paragraphs. The latter analysis was performed twice, once on the whole extract targeting PAHs and related compounds and once on the F1 fraction targeting aliphatic biomarkers. Additional details of these methods are described elsewhere.[1]

Modified EPA Method 8015D was conducted via gas chromatography-flame ionization detection (GC-FID; Agilent 6890) equipped with a Restek Rtx-5 (60m x 0.25 mm ID, 0.25 μm film) fused silica capillary column. Extracts were injected (1 μL, pulsed splitless) into the GC programmed from 40°C (1 min) and ramped at 6°C/min to 315°C (30 min) using

[1] Douglas, G.D., Emsbo-Mattingly, S.D., Stout, S.A., Uhler, A.D., and McCarthy, K.J. (2015) Hydrocarbon Fingerprinting Methods. In: *Introduction to Environmental Forensics, 3rd Ed.*, B. Murphy and R. Morrison, Eds., Academic Press, New York, pp. 201-309.

H_2 (~1 mL/min) as the carrier gas. This analysis was used to determine the concentrations of GC-amenable total petroleum material (TPH; C_9-C_{44}) and individual *n*-alkanes (C_9-C_{40}) and (C_{15}-C_{20}) acyclic isoprenoids. Prior to sample analysis a minimum five-point calibration was performed to demonstrate the linear range of the analysis. The calibration solution was composed of selected aliphatic hydrocarbons within the *n*-C_9 to *n*-C_{40} range. Analyte concentrations in the standard solutions ranged from 1 ng/μL to 200 ng/μL. Target analytes that were not in the calibration solution had the average response factor (RF) of the nearest eluting compound(s) assigned as follows: RF of *n*-C_{14} assigned to C_{15} isoprenoids, *n*-C_{15} assigned to C_{16} isoprenoids; *n*-C_{17} assigned to nor-pristane, and *n*-C_{40} assigned to *n*-C_{39}. All calibration solution compounds that fall within the window were used to generate the average RF for TPH. TPH was quantified by integrating the total C_9-C_{44} area after blank subtraction. Calibration check standards representative of the mid-level of the initial calibration and the instrument blank were analyzed every 10 samples. The check standard's response was compared versus the average RF of the respective analytes contained in the initial calibration. All authentic samples and quality control samples were bracketed by passing mid-check standards.

Modified EPA Method 8270D was conducted via gas chromatography-mass spectrometry (GC-MS; Agilent 7890 GC with 5975c MS) with the MS operated in the selected ion monitoring (SIM) mode for improved sensitivity. The oil and net extracts were injected (1 μL, pulsed splitless) into the GC containing a 60m x 0.25 mm ID, 0.25 μm film, Phenomenex ZB-5 capillary column and the oven programmed from 35°C (1 min) and ramped at 6°C/min to 315°C (30 min) using He as the carrier gas.

The analysis was used to determine the concentrations of 79 parent and alkylated decalins, polycyclic aromatic hydrocarbons (PAH), and sulfur-containing aromatics, as well as 62 petroleum biomarkers, including tricyclic and pentacyclic triterpanes, regular steranes, rearranged steranes, and triaromatic steroids.

In each analysis, prior to sample analysis, the GC-MS was tuned with perfluorotributylamine (PFTBA) at the beginning of each analytical sequence. A minimum 5-point initial calibration consisting of selected target compounds was established to demonstrate the linear range of the analysis. Analyte concentrations in the standard solutions ranged from 0.01 to 10.0 ng/μL for PAH and 0.01 to 20.0 ng/μL for biomarkers. Quantification of target compounds was performed by the method of internal standards using average response factor (RF) determined in the 5-point initial calibration. Alkylated PAHs were quantified using the RF of the corresponding parent, triterpanes were quantified using the RF's for 17α(H),21β(H)-hopane, and steranes and triaromatic steroids were quantified using the RF of 5β(H)-cholane. Biomarker identifications were based upon comparison to selected authentic standards (*Chiron Laboratories*), elution patterns in the peer-reviewed literature, and mass spectral interpretation from full scan GC/MS analyses conducted at Alpha.

Aliquots of each sample extract were used to determine the gravimetric weight of the recoverable oil, thereby allowing the concentrations of target analytes in the oil and net samples to be reported on an oil weight basis (mg/kg_{oil}). All concentrations are not surrogate corrected.

Attachment 3

Interpretation Methods

Data Interpretation

The chemical fingerprinting data collected were evaluated using current geochemical practice utilized in oil spill investigations.[2] For those objectives requiring detailed comparison among samples, the chemical fingerprinting data collected were evaluated using a multi-tiered approach based upon the Centre for European Norms (CEN) oil spill identification protocol, which is used worldwide by many laboratories.[3] Tier 1 involved a qualitative review of each sample's overall (GC/FID) fingerprint that determined the character, boiling range, and weathering state of any oil present. Tier 2 was a 2-step comparison whereupon (a) the first step involved a qualitative review of each sample's PAH (GC/MS EIPs, *m/z* 198, 192, 216, and 242) and biomarker fingerprints (GC/MS EIPs, *m/z* 83, 85, 191, 177, 217, 218, and 231) and (b) the second step utilized the CEN protocol's statistical comparison of diagnostic ratios calculated from PAH and/or biomarker concentrations.[4] Finally, a synthesis of the Tier 1 and Tier 2 results serve to as a confirmation check, before reaching one of the following conclusions:

> **Positive Match**: the samples are considered to match to a high degree of scientific certainty; any differences are explained by weathering and/or are less than the precision of the method.
>
> **Probable Match**: the samples are considered to match to a reasonable degree of scientific certainty; any differences are possibly explained by weathering, mixing, and/or sample heterogeneity.
>
> **Inconclusive**: the samples results preclude any other conclusion, often owing to small sample size leading to low data quality.
>
> **Non-Match**: the samples are considered to not match to a high degree of scientific certainty; any differences are not explained by weathering and/or are greater than the precision of the method.

<u>On-Going Assessment of Diagnostic Ratio Precision</u>

As this monitoring program progressed it became increasingly necessary to evaluate the precision to which the diagnostic ratios (DRs) used in oil spill fingerprinting are measured by the laboratory, especially given the long-term (multi-month or year) nature of the monitoring program. Two

---

[2] Stout, S.A. and Wang, Z. (2016). Chemical fingerprinting methods and factors affecting petroleum fingerprints in the environment. In: *Standard Handbook of Oil Spill Environmental Forensics: Fingerprinting and Source Identification*, 2nd Ed., S.A. Stout and Z. Wang, Eds., Elsevier Publishing Co., Boston, MA, p. 61-130.

[3] Kienhaus, P.G.M. et al. 2016. CEN methodology for oil spill identification. In: *Standard Handbook of Oil Spill Environmental Forensics: Fingerprinting and Source Identification*, 2nd Ed., S.A. Stout and Z. Wang, Eds., Elsevier Publishing Co., Boston, MA, p. 685-728.

[4] The quantitative (statistical) comparisons relied upon the 95% confidence level ($r_{95\%}$) for each diagnostic ratio wherein:

$$r_{95\%} = 2.8 * RSD_r \text{ where } RSD_r = 5\% \text{ standard error, thus}$$

$$r_{95\%} = 14\%$$

If the $r_{95\%}$ between the measured diagnostic between two samples <14% the ratios were considered to statistically **match**, and *vice versa*.

aspects of precision must be considered, namely, *repeatability* and *reproducibility*, which refer to the precision of laboratory data collected over short and long intervals of time, respectively.[5]

The relevance of these concepts to the Sulphur Dome oil monitoring studies is that the detailed chemical fingerprinting has and will continue to be conducted under both sets of conditions. Specifically, oil (or surface sheen) samples prepared and analyzed within a given batch of samples submitted to the lab are analyzed under conditions of *repeatability* whereas samples prepared and analyzed months/years apart are analyzed under conditions of *reproducibility*.

How these results are compared must consider the precision in data, i.e., the DR precision achieved in the short term versus that achieved in the long term. This is due to the inherent variability of the intra-laboratory conditions[6] over time despite following documented standard operating procedures (SOPs) and stringent quality control (QC) measures. This intra-laboratory variability also was demonstrated during the *Deepwater Horizon* NRDA studies whereupon a single reference oil (MC-252) was analyzed hundreds of times over several years in a single lab.[7,8] the experience of which provides the basis for the multi-month/year monitoring of the Sulphur Dome oils (or surface sheens).

Site-specific QC samples included in the Sulphur Dome oil monitoring study since January 2023 have (to date) included five sets of duplicate oils in five different batches of oil samples and one site-specific reference oil (7B cavern oil collected on January 25, 2023) on three occasions spanning approximately 5.4 months. These QC samples were each prepared and analyzed separately and thereby provide the means to assess intra-laboratory precision over both short-term (repeatability of duplicate pairs) and long-term (reproducibility of reference oil).

Table A3 contains the current[9] relative standard deviations (RSD) calculated for the 30 DRs employed to date in the Sulphur Dome oil monitoring study based on the available site-specific QC sample set. The average RSD for the existing duplicate pairs represents the precision achievable in the short-term (RSD_r) and the RSD calculated from the existing reference oil analyses represents the precision achievable in the long-term (RSD_R).

Based upon the CEN (2012) oil spill identification protocol diagnostic ratios with an RSD_r below 5% are considered most reliable and sufficient to determine if two samples are or are not a "match".[10] As can be seen, only four of the 30 DRs employed in this program to date have RSD_r above 5% (Table A3). (One of these is oleanane/hopane (T18/T19) whose precision is reduced owing to the virtual absence of oleanane in the 7B cavern oil samples.) Extending this same 5%

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[5] ASTM (2004) Standard practice for use of the terms precision and bias in ASTM test methods. E-177. Am. Soc. Testing & Materials, Conshohocken, Pennsylvania, USA.

[6] These conditions include things like the different laboratory (GC/FID and GC/MS) instruments, different age GC columns, different data analysts performing the peak integrations.

[7] Litman, E. et al. (2016) Critical review of an interlaboratory forensic dataset: Effects on data interpretation in oil spill studies. In: *Standard Handbook of Oil Spill Environmental Forensics: Fingerprinting and Source Identification*, 2nd Ed., S.A. Stout and Z. Wang, Eds., Elsevier Publishing Co., Boston, MA, p. 1-23.

[8] Stout, S.A. (2016) Oil spill fingerprinting method for oily matrices used in the *Deepwater Horizon* NRDA. Environ. Forensics 17(3): 218-243.

[9] These values will change, hopefully only minimally, as additional duplicate pairs and reference oils are analyzed moving forward.

[10] Kienhaus, P.G.M. et al. 2016. CEN methodology for oil spill identification. In: *Standard Handbook of Oil Spill Environmental Forensics: Fingerprinting and Source Identification*, 2nd Ed., S.A. Stout and Z. Wang, Eds., Elsevier Publishing Co., Boston, MA, p. 685-728.

criterion to RSD_R is appropriate since only those DRs that are precisely measured over the long-term (RSD_R<5%) are equally reliable.[11]

Considering both RSD_r and RSD_R, Table A3 shows that 17 of the 30 DRs have both RSD values below 5% indicating these 17 DRs are measured precisely over both the short-term and long-term and are good candidate ratios to employ in the long-term Sulphur Dome study. Those DRs with one or both RSD values above 5% are less reliable and their use requires greater caution.

The result of this assessment of DR precision is that:

- The 17 DRs identified in Table A3 that (to date) are demonstrated to be precisely measured over both the short-term and long-term should be (and will be) heavily relied upon for recognizing changes in the specific nature of the cavern oil or those of the locally-produced crude oils.

The precision of these DRs will be adjusted as the monitoring program advances and the QC dataset grows.

Table A3: RSD_r and RSD_R calculated for the 30 diagnostic ratios used in the Sulphur Dome monitoring studies.

		Sulphur Dome Site Precision		
		Repeatability	Reproducibility	Most Precise Ratios*
CEN - Diagnostic Ratios	CEN Diagnostic Ratios per Alpha Abbreviations	RSD_r	RSD_R	
NR-C17/pris	C17/Pr	2.0	5.5	
NR-C18/phy	C18/Ph	1.0	2.0	x
NR- pris/phy	Pr/Ph	1.7	4.7	x
NR-4-MD/1-MD	4-MDBT/1-MDBT	3.0	6.7	
NR-2-MP/1-MP	2-MP/1-MP	2.6	2.2	x
NR-27Ts/30ab	T11/T19	3.4	1.4	x
NR-27Tm/30ab	T12/T19	2.3	2.5	x
NR-28ab/30ab	T14a/T19	2.5	9.6	
NR-29ab/30ab	T15/T19	2.3	1.7	x
NR-30O/30ab	T18/T19	6.8	13.3	
NR-31abS/30ab	T21/T19	1.1	2.5	x
NR-27dbR/27dbS	S4/S5	5.1	9.1	
NR-27bb/29bb	(S14+S15)/(S26+S27)	3.7	1.9	x
NR-SC26/ RC26+SC27	TAS09/TAS01	2.5	2.8	x
NR-SC28/RC26 + SC27	TAS02/TAS01	2.8	1.4	x
NR-RC27/RC26+ SC27	TAS03/TAS01	2.1	0.5	x
NR-RC28/RC26+SC27	TAS04/TAS01	3.2	1.2	x
DR-Ts/Tm	T11/T12	2.5	2.5	x
DR-29Ts30ab	T16/T19	3.3	1.1	x
DR-29bb/29aa	(S26+S27)/(S25+S28)	4.6	14.7	
DR-C2-dbt/C2-phe	DBT2/PA2	0.8	4.2	x
DR-C3-dbt/C3-phe	DBT3/PA3	0.5	5.2	
DR-C28C29/30ab	T7 to T10/T19	5.2	13.9	
DR-29aaS/29aaR	S25/S28	8.8	20.6	
DR-C20TA/C21TA	TAS05/TAS06	4.5	14.0	
DR-TA21/ RC26+SC27	TS06/TAS01	4.8	8.4	
DR-C24Tet/C26Tri	T6a/T19	4.5	3.5	x
DR-30ba/30ab	T20/T19	3.2	17.4	
DR-35ab/30ab	(T34 to T35)/T19	5.3	6.9	
DR-32abR/32abS	T27/T26	2.0	1.1	x

*both RSD_r and RSD_R < 5% based on current QC datasets

[11] Note that the RPD criterion used for oil spill fingerprinting (5%) are much more stringent than are common to SW-846 EPA Methods, i.e., 30% relative percent difference (for duplicates) and 25% RSD (for triplicates or more).

Attachment 4

Tabulated Concentrations

Table 4-1: Concentrations (mg/kg) of n-alkanes and isoprenoids in the samples studied.

Client ID	253998	253998 (Dup)	41842	7B (Ref. Oil)	189416 (1,250')	189416 (170')
Lab ID	L2348036-01	WG1829150-4	L2348036-02	L2348036-03	L2348036-04	L2348036-05
Date Collected	8/17/2023	8/17/2023	8/17/2023	1/25/2023	8/29/2023	8/29/2023
Date Analyzed	9/20/2023	9/20/2023	9/20/2023	9/20/2023	9/20/2023	9/20/2023
Analytes	Result	Result	Result	Result	Result	Result
n-Nonane (C9)	487	454	3,850	9,840	nd	nd
n-Decane (C10)	463	486	3,610	8,680	113	106
n-Undecane (C11)	487	446	3,810	8,340	87	80
n-Dodecane (C12)	344	307	3,630	7,740	nd	nd
n-Tridecane (C13)	596	590	3,810	7,000	nd	nd
2,6,10 Trimethyldodecane (1380)	93	75	1,060	1,370	143	152
n-Tetradecane (C14)	971	916	3,510	6,410	150	153
2,6,10 Trimethyltridecane (1470)	798	697	2,140	1,990	808	767
n-Pentadecane (C15)	1,040	974	3,530	6,160	524	490
n-Hexadecane (C16)	831	811	2,840	5,300	269	256
Norpristane (1650)	191	216	1,530	1,270	226	195
n-Heptadecane (C17)	632	595	2,630	4,740	129	115
Pristane	261	263	4,250	1,840	292	310
n-Octadecane (C18)	614	606	2,510	4,240	224	203
Phytane	113	117	1,130	1,960	138	162
n-Nonadecane (C19)	478	466	2,150	3,640	210	197
n-Eicosane (C20)	477	466	2,020	3,400	178	210
n-Heneicosane (C21)	407	375	1,880	2,720	nd	nd
n-Docosane (C22)	389	367	1,670	2,420	122	128
n-Tricosane (C23)	269	255	1,550	2,090	nd	37
n-Tetracosane (C24)	267	248	1,360	1,990	nd	nd
n-Pentacosane (C25)	351	320	1,580	2,010	152	204
n-Hexacosane (C26)	215	212	1,020	1,480	nd	nd
n-Heptacosane (C27)	198	188	968	1,220	nd	nd
n-Octacosane (C28)	164	152	682	999	nd	nd
n-Nonacosane (C29)	171	167	649	979	nd	nd
n-Triacontane (C30)	169	151	543	871	nd	nd
n-Hentriacontane (C31)	217	212	451	785	nd	nd
n-Dotriacontane (C32)	110	96	260	709	nd	nd
n-Tritriacontane (C33)	215	208	556	646	312	324
n-Tetratriacontane (C34)	233	200	544	604	302	299
n-Pentatriacontane (C35)	nd	nd	132	638	nd	nd
n-Hexatriacontane (C36)	68	84	157	365	100	106
n-Heptatriacontane (C37)	nd	nd	78	350	nd	nd
n-Octatriacontane (C38)	nd	nd	91	354	nd	nd
n-Nonatriacontane (C39)	nd	nd	nd	283	nd	nd
n-Tetracontane (C40)	nd	nd	nd	256	nd	nd
Total Saturated Hydrocarbons	12,300	11,700	62,200	106,000	4,480	4,490
Total Petroleum Hydrocarbons (C9-C44)	668,000	648,000	614,000	646,000	712,000	727,000

Table 4-2: Concentrations (mg/kg) of PAHs, related compounds and petroleum biomarkers in the samples studied.

	Client ID	253998	253998 (Dup)	41842	7B	189416 (1,250')	189416 (170')
	Lab ID	L2348036-01	/G1829150-4	L2348036-02	L2348036-03	L2348036-04	L2348036-05
	Date Collected	8/17/2023	NA	8/17/2023	1/25/2023	8/29/2023	8/29/2023
	Date Analyzed	9/19/2023	9/19/2023	9/20/2023	9/20/2023	9/21/2023	9/20/2023
	Analytes	Result	Result	Result	Result	Result	Result
D0	cis/trans-Decalin	567	507	523	206	188	178
D1	C1-Decalins	830	754	792	328	458	438
D2	C2-Decalins	1,270	1,180	677	284	1,200	1,160
D3	C3-Decalins	855	817	388	164	814	823
D4	C4-Decalins	913	917	498	158	896	950
BT0	Benzothiophene	4	4	4	10	7	7
BT1	C1-Benzo(b)thiophenes	27	27	27	47	37	38
BT2	C2-Benzo(b)thiophenes	28	27	23	158	28	28
BT3	C3-Benzo(b)thiophenes	56	54	36	264	58	59
BT4	C4-Benzo(b)thiophenes	35	36	27	204	39	40
N0	Naphthalene	353	318	317	251	43	41
N1	C1-Naphthalenes	659	606	521	715	320	319
N2	C2-Naphthalenes	1,010	943	650	1,070	1,360	1,400
N3	C3-Naphthalenes	737	691	674	907	1,310	1,360
N4	C4-Naphthalenes	576	540	443	470	820	860
B	Biphenyl	65	60	47	31	7	8
DF	Dibenzofuran	30	28	24	25	9	9
AY	Acenaphthylene	3.8	3.5	4.7	3.8	4.6	4.5
AE	Acenaphthene	18	17	8	8	13	12
F0	Fluorene	64	59	8	44	45	50
F1	C1-Fluorenes	196	185	33	111	183	191
F2	C2-Fluorenes	388	371	68	182	323	335
F3	C3-Fluorenes	353	323	74	179	274	289
A0	Anthracene	nd	nd	2	nd	nd	nd
P0	Phenanthrene	114	106	31	86	116	119
PA1	C1-Phenanthrenes/Anthracenes	338	316	75	233	331	342
PA2	C2-Phenanthrenes/Anthracenes	432	402	101	280	401	418
PA3	C3-Phenanthrenes/Anthracenes	309	289	94	203	270	286
PA4	C4-Phenanthrenes/Anthracenes	156	150	58	105	126	134
RET	Retene	nd	nd	46	nd	58	58
DBT0	Dibenzothiophene	23	22	8	185	27	28
DBT1	C1-Dibenzothiophenes	96	91	47	466	114	117
DBT2	C2-Dibenzothiophenes	120	113	34	623	122	126
DBT3	C3-Dibenzothiophenes	104	97	37	482	106	108
DBT4	C4-Dibenzothiophenes	54	51	23	250	54	55
BF	Benzo(b)fluorene	5.6	5.0	1.0	2.6	4.6	5.0
FL0	Fluoranthene	5.2	5.1	1.4	1.4	4.0	5.0
PY0	Pyrene	15	14	4	9	11	10
FP1	C1-Fluoranthenes/Pyrenes	68	61	14	33	50	55
FP2	C2-Fluoranthenes/Pyrenes	114	105	22	64	77	82
FP3	C3-Fluoranthenes/Pyrenes	140	132	29	87	91	99
FP4	C4-Fluoranthenes/Pyrenes	117	107	30	79	78	81
NBT0	Naphthobenzothiophenes	10	8	2	35	7	8
NBT1	C1-Naphthobenzothiophenes	31	28	8	110	22	25
NBT2	C2-Naphthobenzothiophenes	43	39	11	167	30	34
NBT3	C3-Naphthobenzothiophenes	30	31	9	140	22	25
NBT4	C4-Naphthobenzothiophenes	27	27	nd	104	26	28
BA0	Benz[a]anthracene	2.2	2.1	1.0	0.9	1.8	2.0
C0	Chrysene/Triphenylene	28	25	3	18	19	17
BC1	C1-Chrysenes	65	64	13	42	43	44
BC2	C2-Chrysenes	93	90	25	65	60	61
BC3	C3-Chrysenes	96	92	31	80	68	71
BC4	C4-Chrysenes	74	70	nd	65	53	57

Table 4-2: continued

	Client ID	253998	253998 (Dup)	41842	7B	189416 (1,250')	189416 (170')
	Lab ID	L2348036-01	WG1829150-4	L2348036-02	L2348036-03	L2348036-04	L2348036-05
	Date Collected	8/17/2023	NA	8/17/2023	1/25/2023	8/29/2023	8/29/2023
	Date Analyzed	9/19/2023	9/19/2023	9/20/2023	9/20/2023	9/21/2023	9/20/2023
	Analytes	Result	Result	Result	Result	Result	Result
BBF	Benzo[b]fluoranthene	3.7	3.5	0.9	2.4	2.3	2.1
BJKF	Benzo[j]fluoranthene/Benzo[k]fluoranthene	0.6	nd	nd	nd	nd	nd
BAF	Benzo[a]fluoranthene	nd	nd	nd	nd	nd	nd
BEP	Benzo[e]pyrene	5	4	1	7	3	3
BAP	Benzo[a]pyrene	1.2	0.9	0.4	1.3	1.2	0.9
PER	Perylene	18.8	18.4	4.0	1.4	9.8	10.5
IND	Indeno[1,2,3-cd]pyrene	0.5	nd	nd	nd	nd	nd
DA	Dibenz[ah]anthracene/Dibenz[ac]anthracene	0.6	nd	nd	nd	nd	nd
GHI	Benzo[g,h,i]perylene	1.6	1.3	1.5	2.2	1.1	1.5
CAR	Carbazole	2.9	2.8	2.0	7.3	2.6	3.0
4MDT	4-Methyldibenzothiophene	35	32	11	203	42	43
2MDT	2/3-Methyldibenzothiophene	46	44	30	171	57	56
1MDT	1-Methyldibenzothiophene	10	10	3	84	11	12
3MP	3-Methylphenanthrene	76	71	15	42	72	74
2MP	2-Methylphenanthrene	80	74	14	54	76	80
2MA	2-Methylanthracene	3.6	4.1	2.9	2.4	3.4	3.5
9MP	9/4-Methylphenanthrene	122	114	23	82	108	110
1MP	1-Methylphenanthrene	55	51	17	51	66	69
2MN	2-Methylnaphthalene	633	582	415	549	226	219
1MN	1-Methylnaphthalene	340	313	368	528	254	254
26DMN	2,6-Dimethylnaphthalene	536	498	245	449	676	696
235TMN	2,3,5-Trimethylnaphthalene	79	73	73	88	168	186
PY2	2-METHYLPYRENE	2.5	6.1	5.8	1.1	2.0	3.8
PY4	4-METHYLPYRENE	10.0	12.1	10.1	1.9	8.4	7.8
PY1	1-METHYLPYRENE	6.0	6.8	6.2	1.2	5.2	4.6
T4	C23 Tricyclic Terpane	22	23	18	20	25	26
T5	C24 Tricyclic Terpane	18	19	13	11	18	18
T6	C25 Tricyclic Terpane	18	19	19	11	23	23
T6a	C24 Tetracyclic Terpane	25	24	28	15	31	32
T6b	C26 Tricyclic Terpane-22S	12.5	11.2	8.3	4.7	11.7	12.0
T6c	C26 Tricyclic Terpane-22R	8.1	6.4	6.4	4.6	7.7	8.0
T7	C28 Tricyclic Terpane-22S	8.8	8.1	7.4	5.6	8.6	9.0
T8	C28 Tricyclic Terpane-22R	12.9	12.3	14.4	6.7	15.0	11.8
T9	C29 Tricyclic Terpane-22S	13.2	14.5	11.0	5.3	14.3	14.4
T10	C29 Tricyclic Terpane-22R	9	11	11	7	14	12
T11	18a-22,29,30-Trisnorneohopane-TS	75	76	118	22	112	114
T11a	C30 Tricyclic Terpane-22S	16.1	16.7	13.5	4.7	19.6	14.1
T11b	C30 Tricyclic Terpane-22R	6.0	7.4	8.4	5.0	8.1	9.6
T12	17a(H)-22,29,30-Trisnorhopane-TM	101	99	174	24	156	156
T14a	17a/b,21b/a 28,30-Bisnorhopane	30	29	36	16	37	39
T14b	17a(H),21b(H)-25-Norhopane	73.0	71.8	nd	nd	18.1	17.5
T15	30-Norhopane	220	209	588	78	459	465
T16	18a(H)-30-Norneohopane-C29Ts	109	108	173	19	151	145
X	17a(H)-Diahopane	38.2	29.1	30.6	3.2	36.1	34.2
T17	30-Normoretane	81	78	144	8	122	124
T18	18a(H)&18b(H)-Oleananes	44.0	46.3	114.0	5.3	71.6	80.7
T19	Hopane	335	329	872	94	749	777
T20	Moretane	85	83	154	8	157	157
T21	30-Homohopane-22S	86	86	213	53	192	196
T22	30-Homohopane-22R	83	80	170	42	158	165

Table 4-2: continued

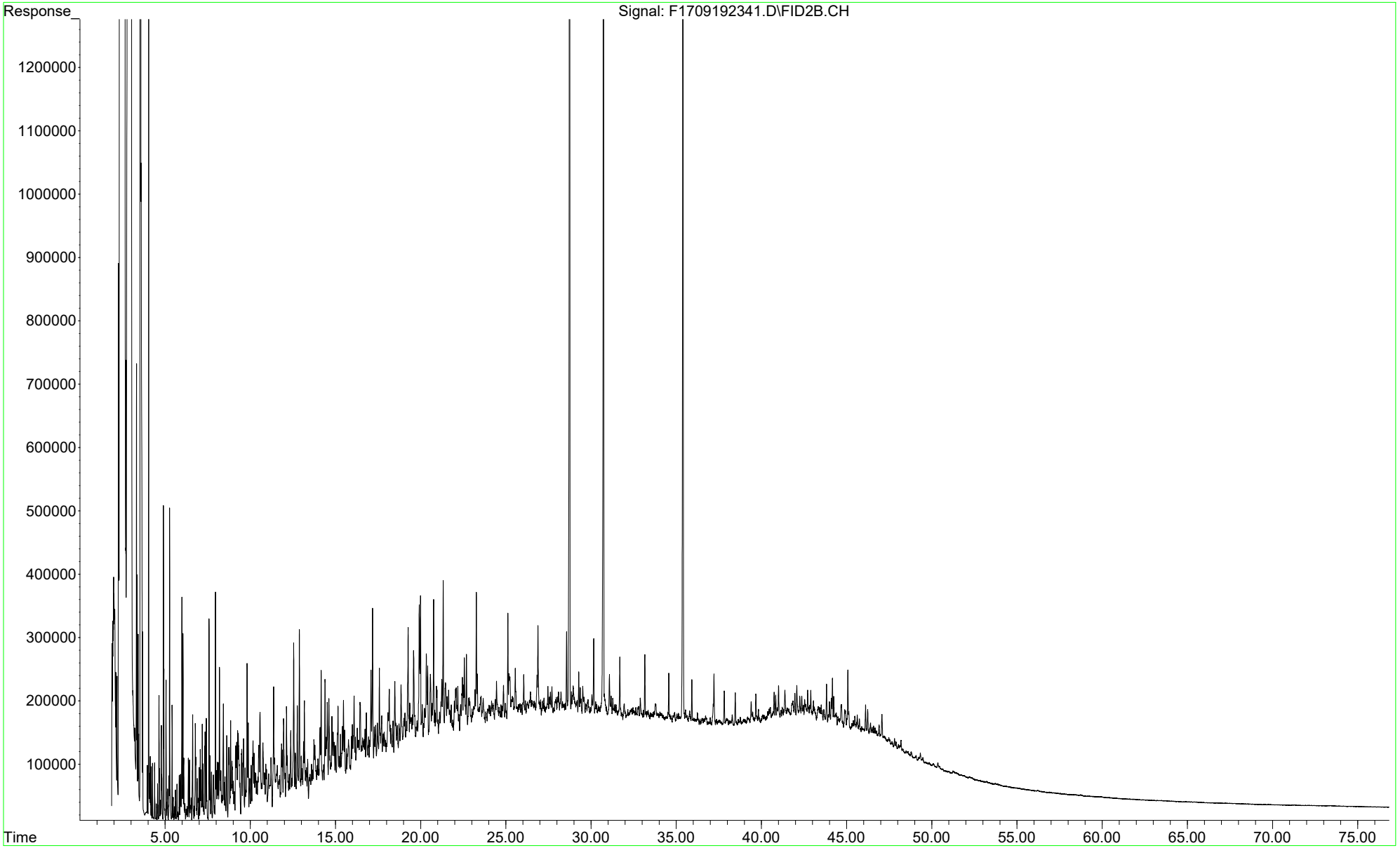
	Client ID	253998	253998 (Dup)	41842	7B	189416 (1,250')	189416 (170')
	Lab ID	L2348036-01	WG1829150-4	L2348036-02	L2348036-03	L2348036-04	L2348036-05
	Date Collected	8/17/2023	NA	8/17/2023	1/25/2023	8/29/2023	8/29/2023
	Date Analyzed	9/19/2023	9/19/2023	9/20/2023	9/20/2023	9/21/2023	9/20/2023
	Analytes	Result	Result	Result	Result	Result	Result
T22A	T22a-Gammacerane/C32-diahopane	30	29	53	10	48	49
T26	30,31-Bishomohopane-22S	51	49	107	34	103	105
T27	30,31-Bishomohopane-22R	102	97	213	25	156	156
T30	30,31-Trishomohopane-22S	36	36	73	28	64	62
T31	30,31-Trishomohopane-22R	24	24	49	20	44	43
T32	Tetrakishomohopane-22S	25	28	40	19	40	42
T33	Tetrakishomohopane-22R	19	19	28	13	29	29
T34	Pentakishomohopane-22S	18	16	21	19	25	24
T35	Pentakishomohopane-22R	17	14	22	16	25	26
S4	13b(H),17a(H)-20S-Diacholestane	78	78	66	30	98	102
S5	13b(H),17a(H)-20R-Diacholestane	54	46	43	15	55	68
S23	14b,17b-20S-Methylcholestane	53	46	35	32	52	56
S26	14b(H),17b(H)-20R-Ethylcholestane	56	55	39	43	64	60
S27	14b(H),17b(H)-20S-Ethylcholestane	39	38	22	29	38	48
TAS05	C20 PREGNANE	68.7	64.4	59.8	92.2	97.3	97.5
TAS06	C21 20-METHYLPREGNANE	60.2	52.9	48.5	68.5	74.7	74.1
TAS07	C22 20-ETHYLPREGNANE (A)	15.1	13.8	13.0	31.2	18.8	18.8
TAS08	C22 20-ETHYLPREGNANE (B)	19.6	18.7	14.9	23.7	20.4	17.3
TAS09	C26,20S TAS	80.9	81.4	122.0	22.8	104.0	107.0
TAS01	C26,20R+C27,20S TAS	252	253	367	167	299	316
TAS02	C28,20S TAS	232	219	272	119	242	261
TAS03	C27,20R TAS	162	159	224	129	191	192
TAS04	C28,20R TAS	169	166	226	101	204	211
TAS10	C29,20S TAS	52.2	46.9	79.0	40.6	62.6	65.4
TAS11	C29,20R TAS	21.3	28.0	24.4	14.9	22.3	30.0

Attachment 5

GC/FID Chromatograms

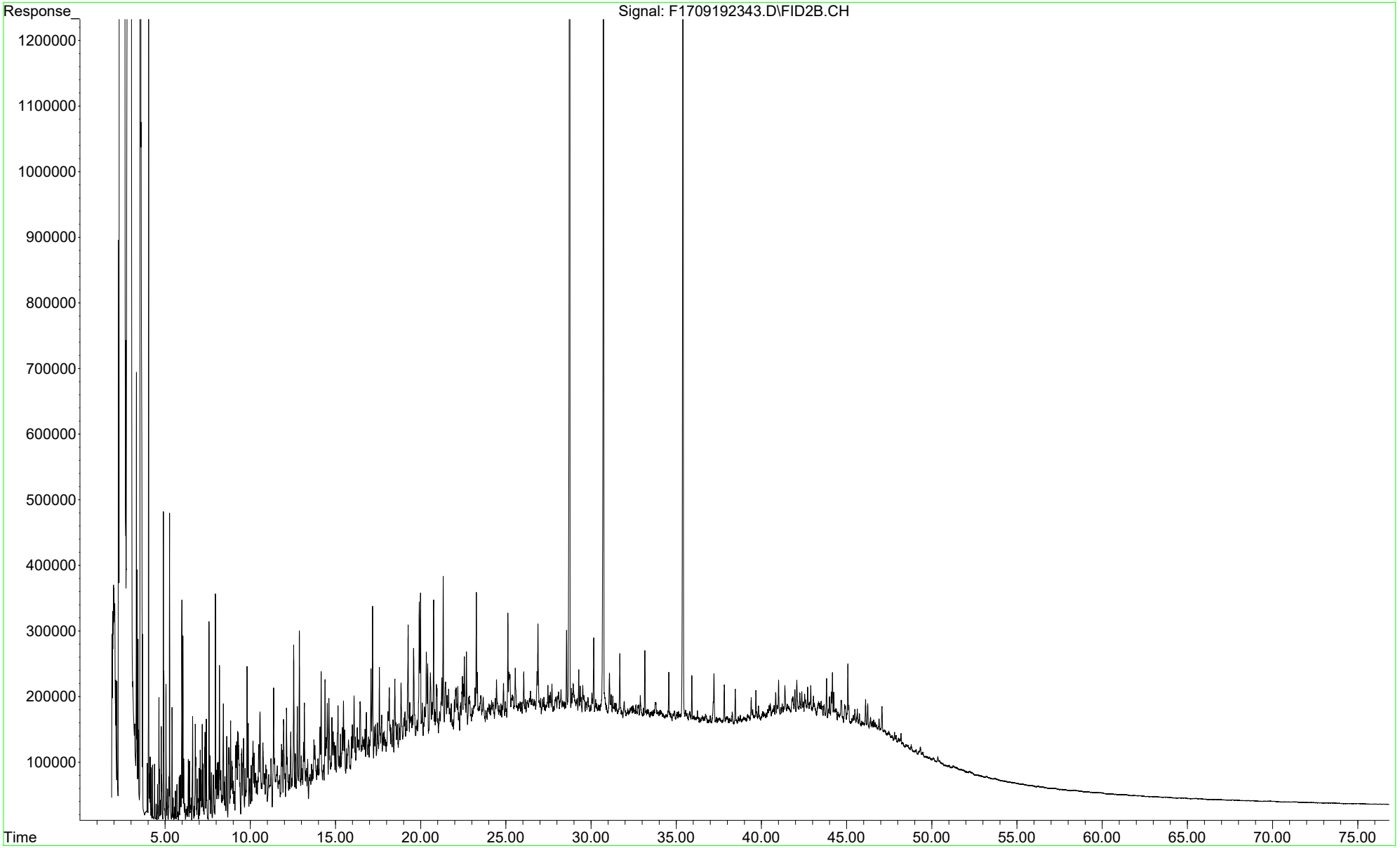
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\SH
... C\F1709192341.D
Operator : FID17:AMV
Instrument : FID17
Acquired : 20 Sep 2023 4:26 pm using AcqMethod FID17A.M
Sample Name: L2348036-01
Misc Info : WG1829341,WG1829150,ICAL20114

253998
L2348036-01



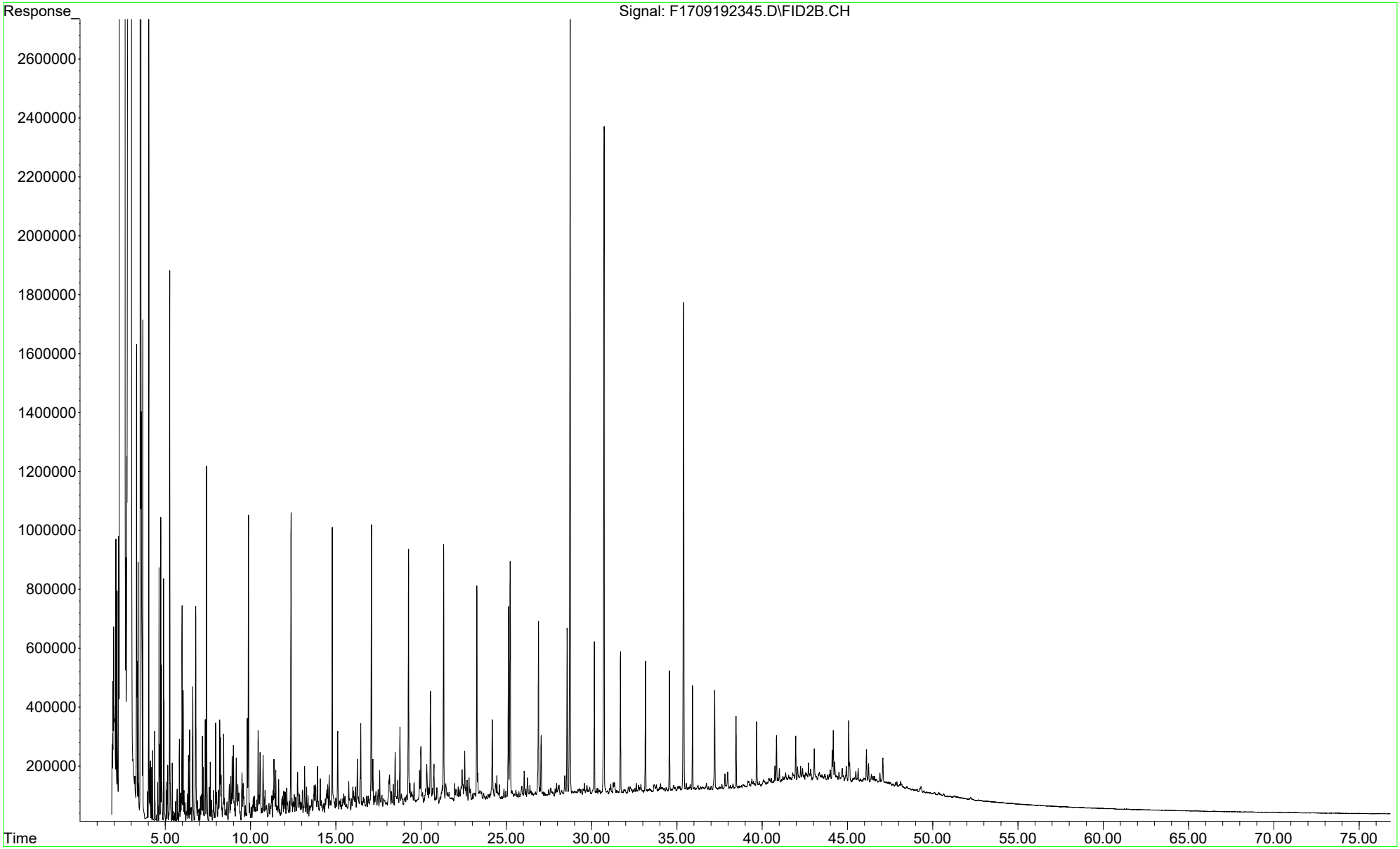
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\SH
... C\F1709192343.D
Operator : FID17:AMV
Instrument : FID17
Acquired : 20 Sep 2023 5:56 pm using AcqMethod FID17A.M
Sample Name: WG1829150-4
Misc Info : WG1829341,WG1829150,ICAL20114

L2348036-01 Duplicate
WG1829150-4



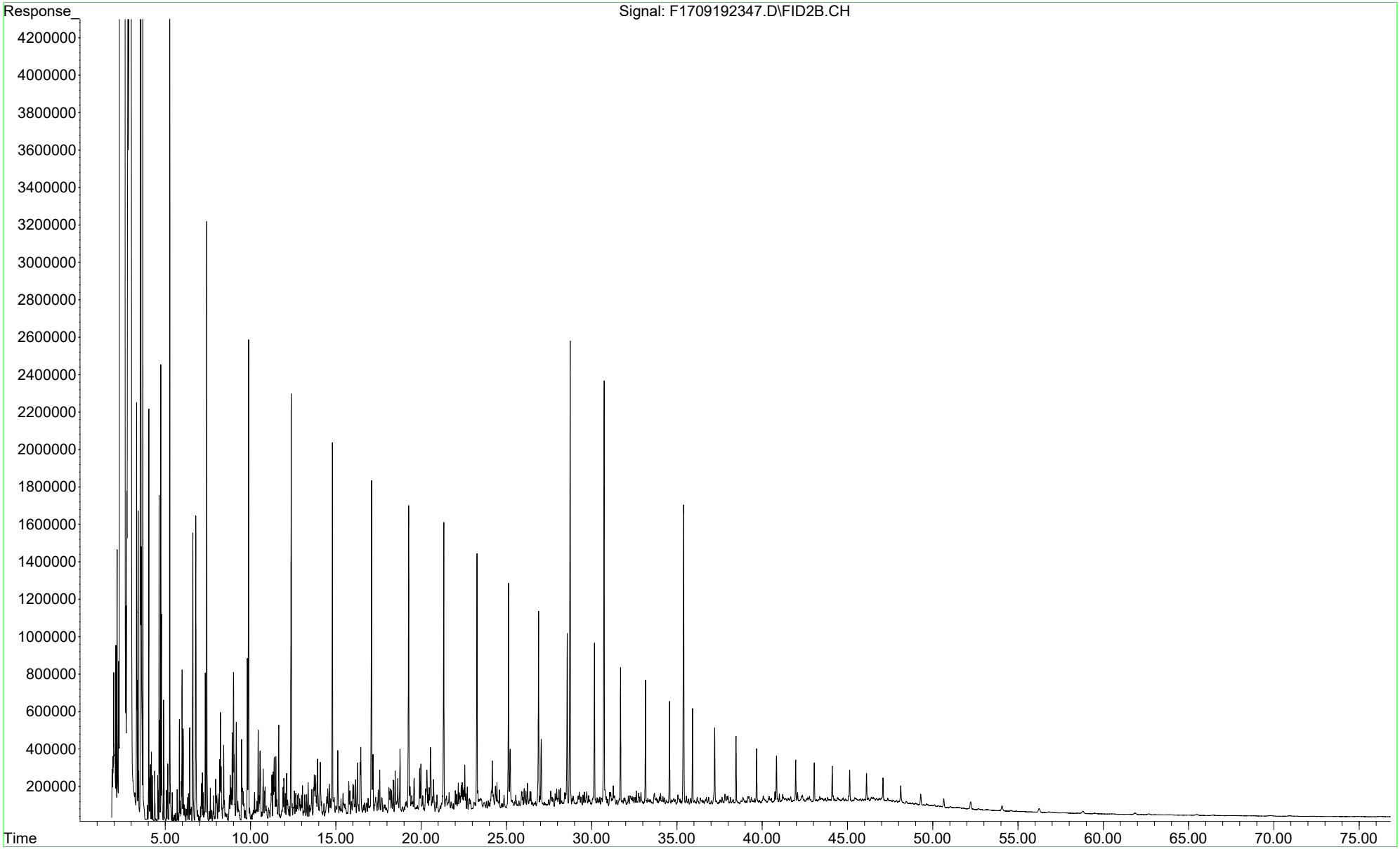
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\SH
... C\F1709192345.D
Operator : FID17:AMV
Instrument : FID17
Acquired : 20 Sep 2023 7:24 pm using AcqMethod FID17A.M
Sample Name: L2348036-02
Misc Info : WG1829341,WG1829150,ICAL20114

41842
L2348036-02



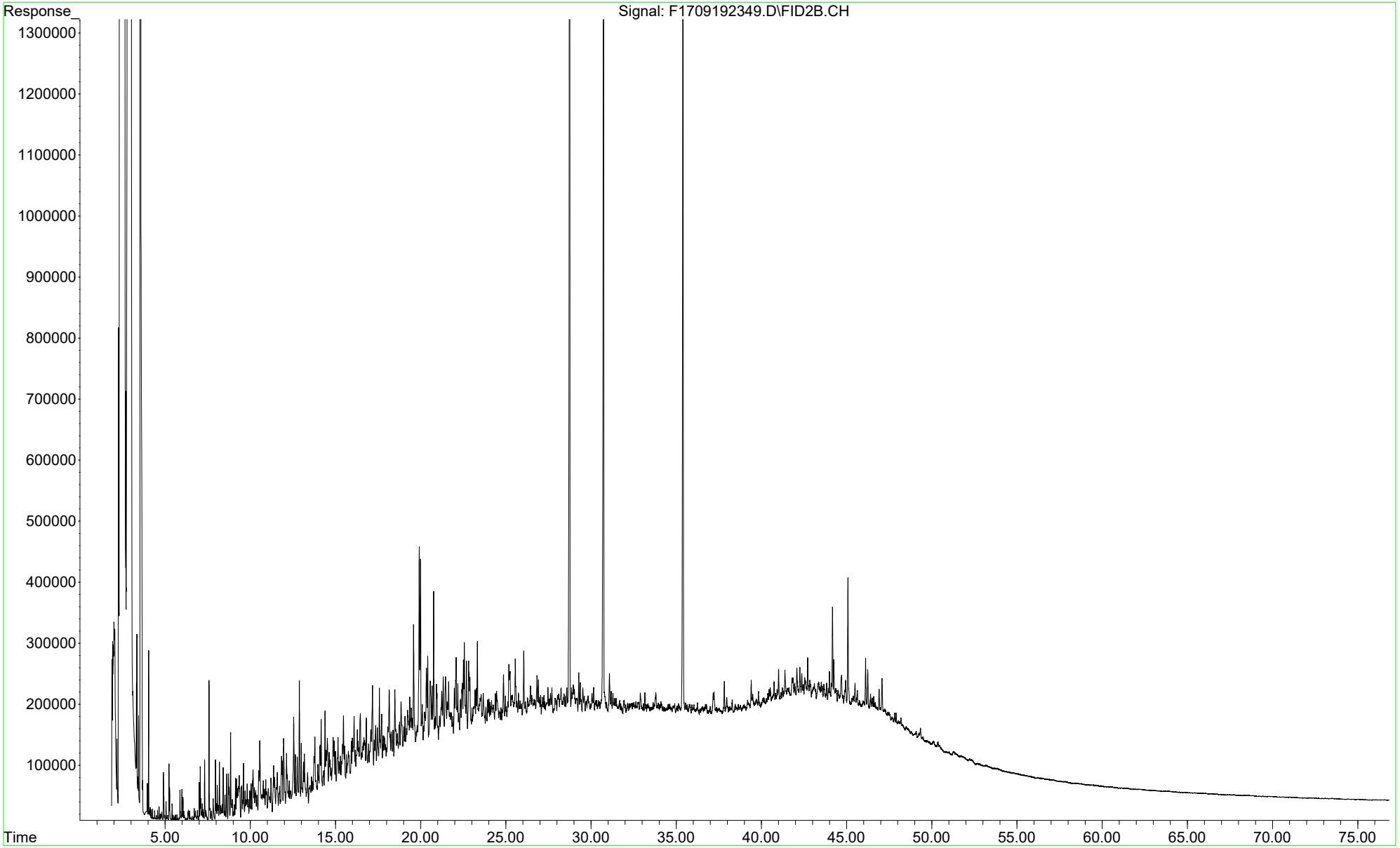
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\SH
... C\F1709192347.D
Operator : FID17:AMV
Instrument : FID17
Acquired : 20 Sep 2023 8:53 pm using AcqMethod FID17A.M
Sample Name: L2348036-03
Misc Info : WG1829341,WG1829150,ICAL20114

7B Reference Oil
L2348036-03



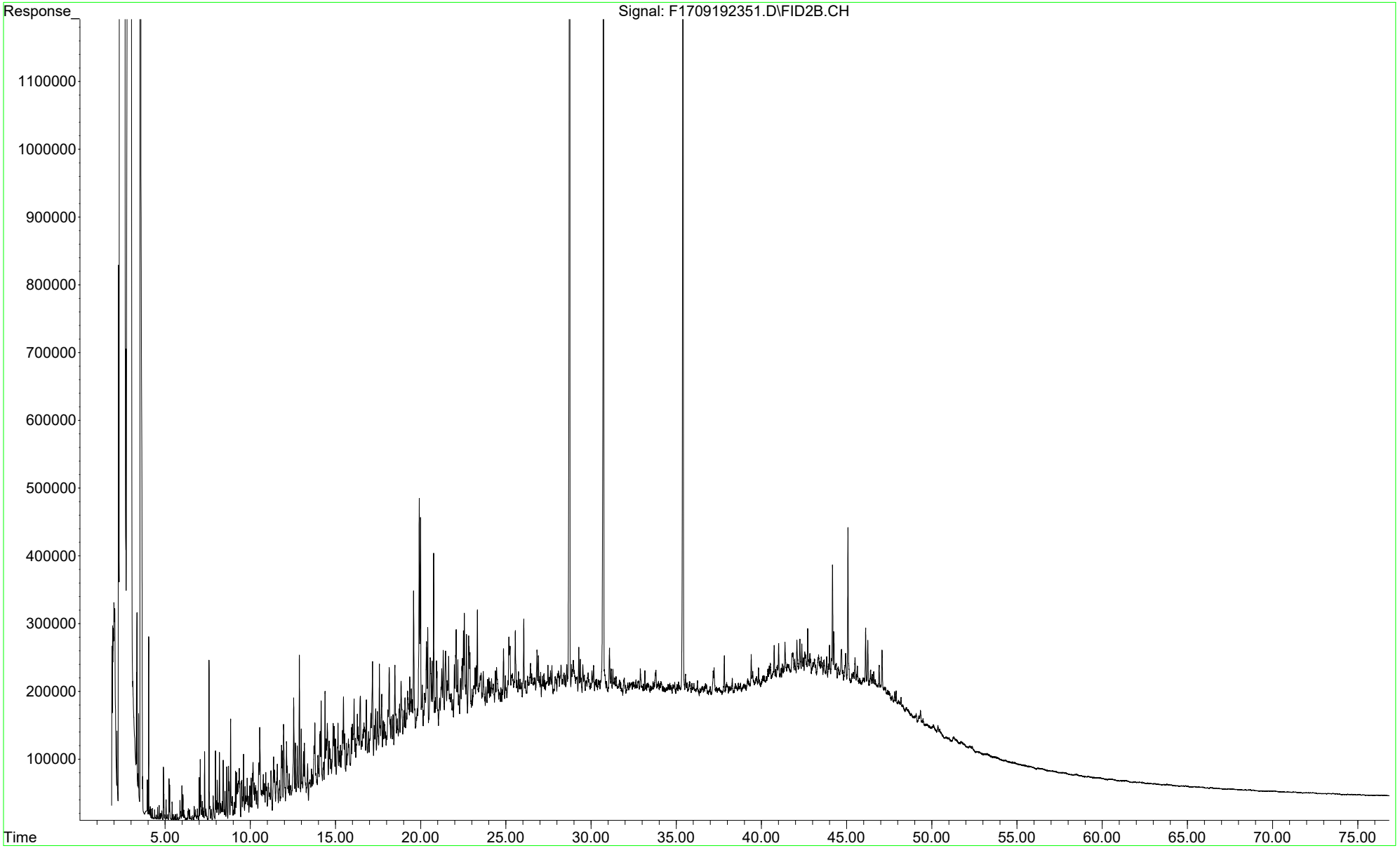
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\SH
... C\F1709192349.D
Operator : FID17:AMV
Instrument : FID17
Acquired : 20 Sep 2023 10:21 pm using AcqMethod FID17A.M
Sample Name: L2348036-04
Misc Info : WG1829341,WG1829150,ICAL20114

SN 189416 (1,250')
L2348036-04



File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\SH
... C\F1709192351.D
Operator : FID17:AMV
Instrument : FID17
Acquired : 20 Sep 2023 11:49 pm using AcqMethod FID17A.M
Sample Name: L2348036-05
Misc Info : WG1829341,WG1829150,ICAL20114

SN 189416 (170')
L2348036-05

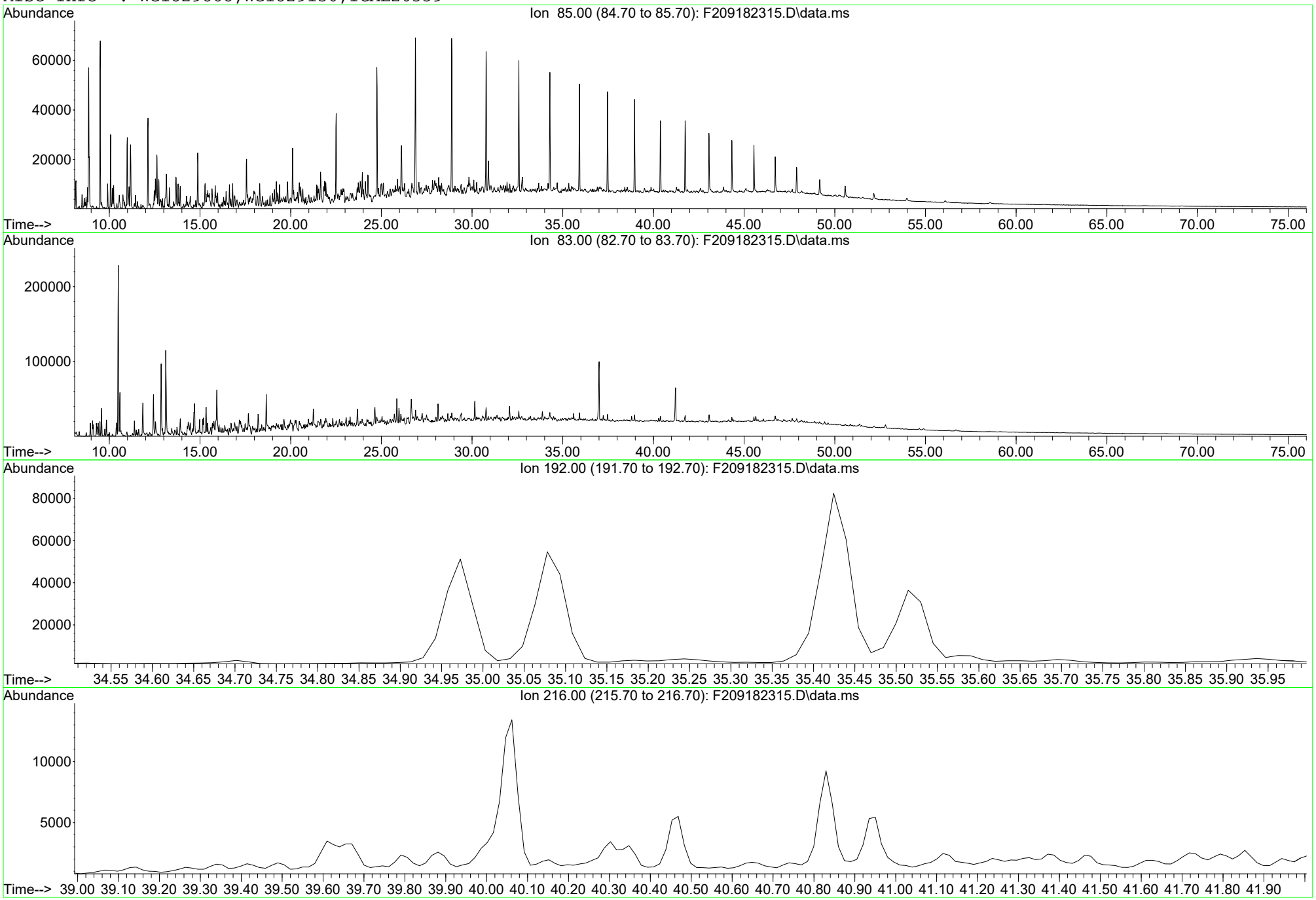


Attachment 6

GC/MS Extracted Ion Profiles

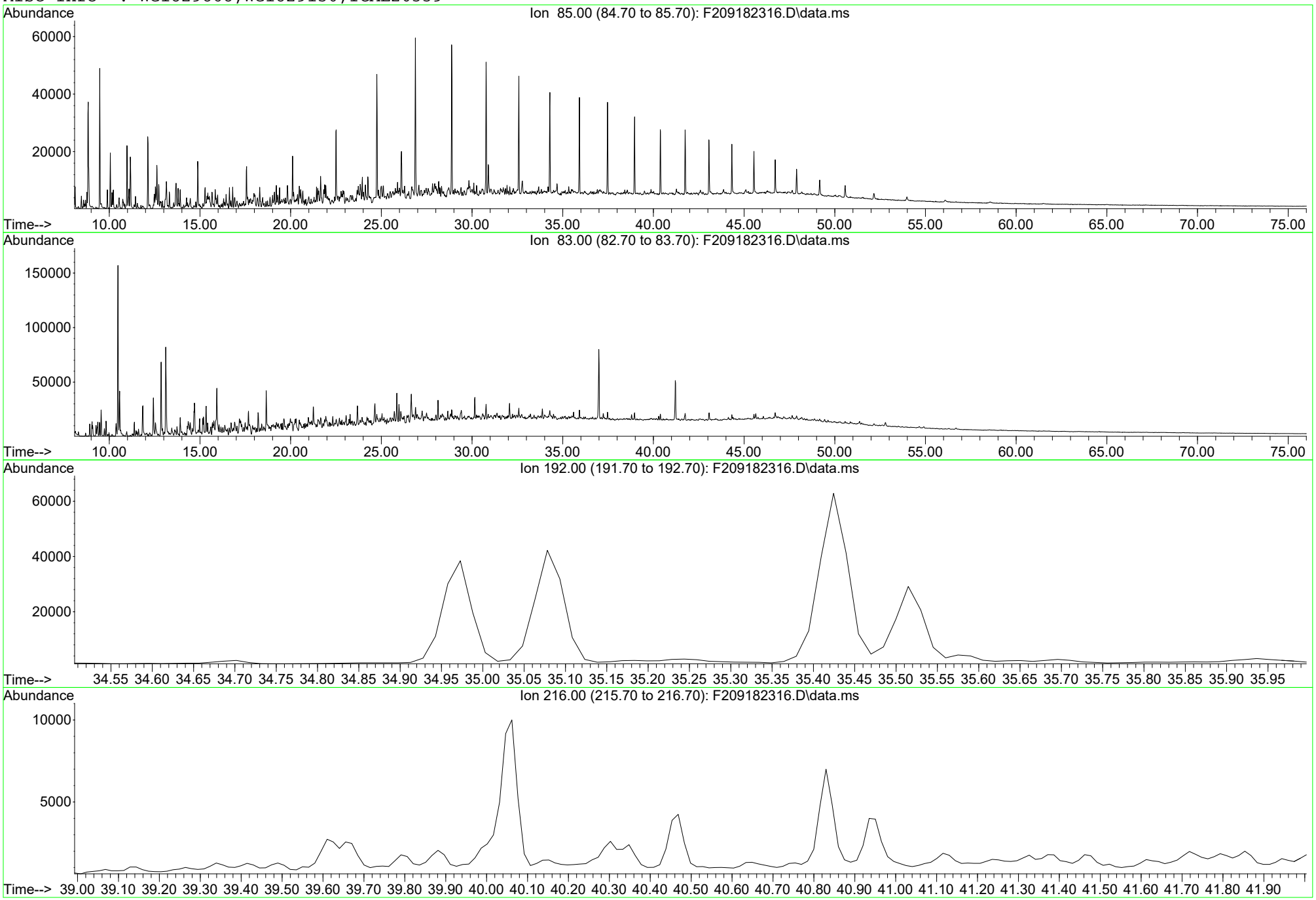
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182315.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 19 Sep 2023 10:33 pm using AcqMethod FRNC2A.M
Sample Name: l2348036-01,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

253998
L2348036-01



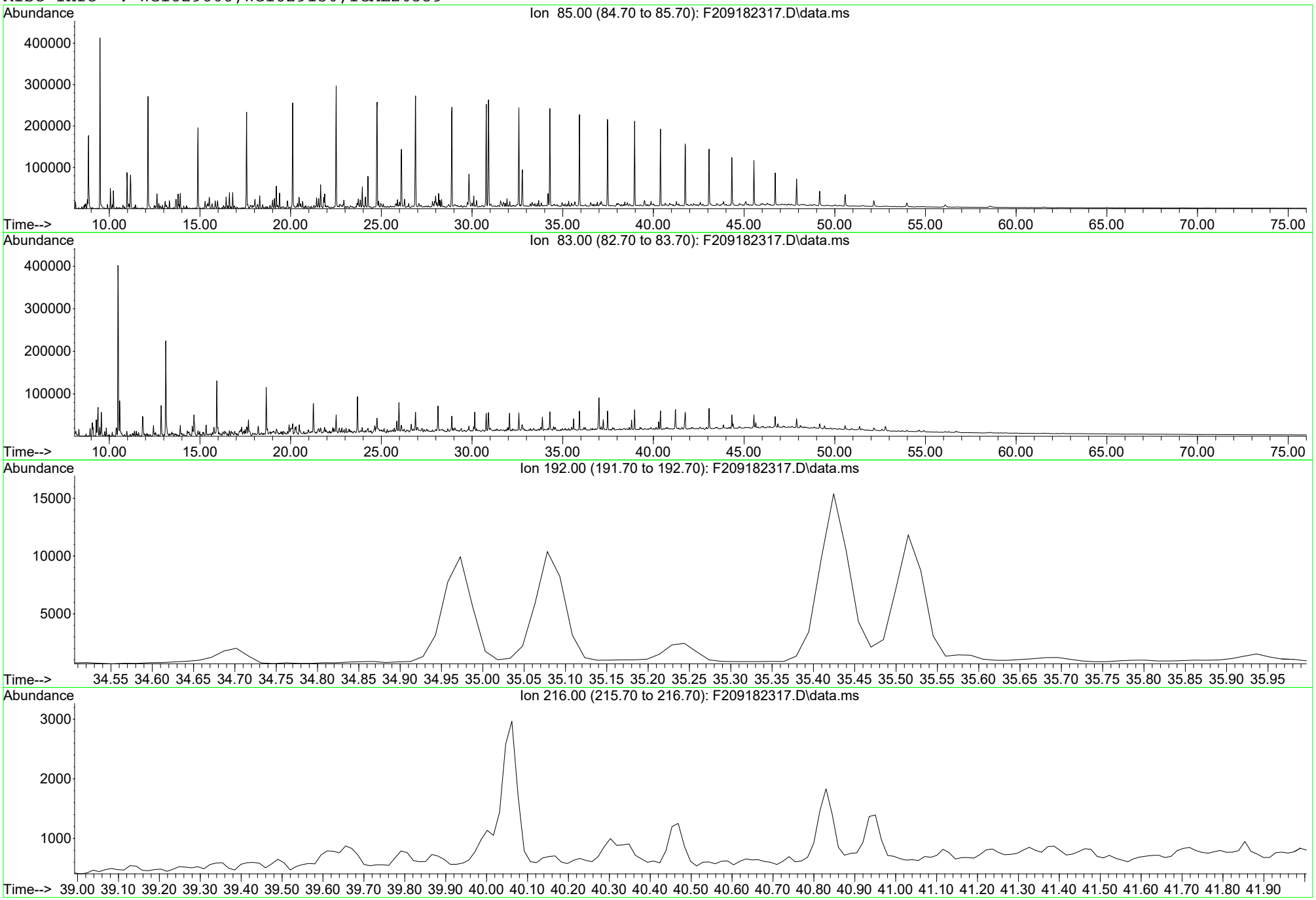
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182316.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 19 Sep 2023 11:59 pm using AcqMethod FRNC2A.M
Sample Name: wg1829150-4,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

L2348036-01 Duplicate
WG1829150-4



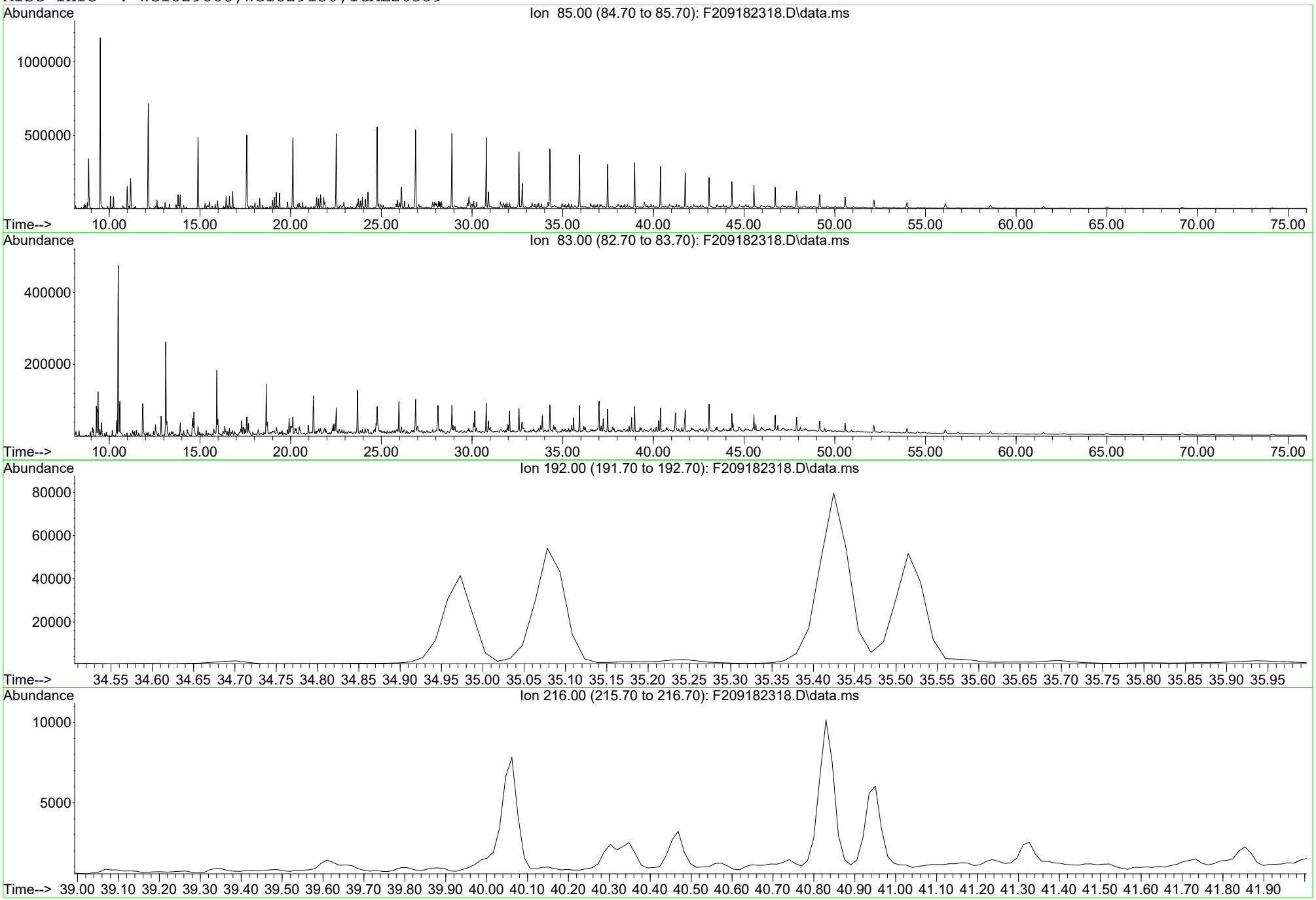
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182317.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 20 Sep 2023 1:24 am using AcqMethod FRNC2A.M
Sample Name: l2348036-02,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

41842
L2348036-02



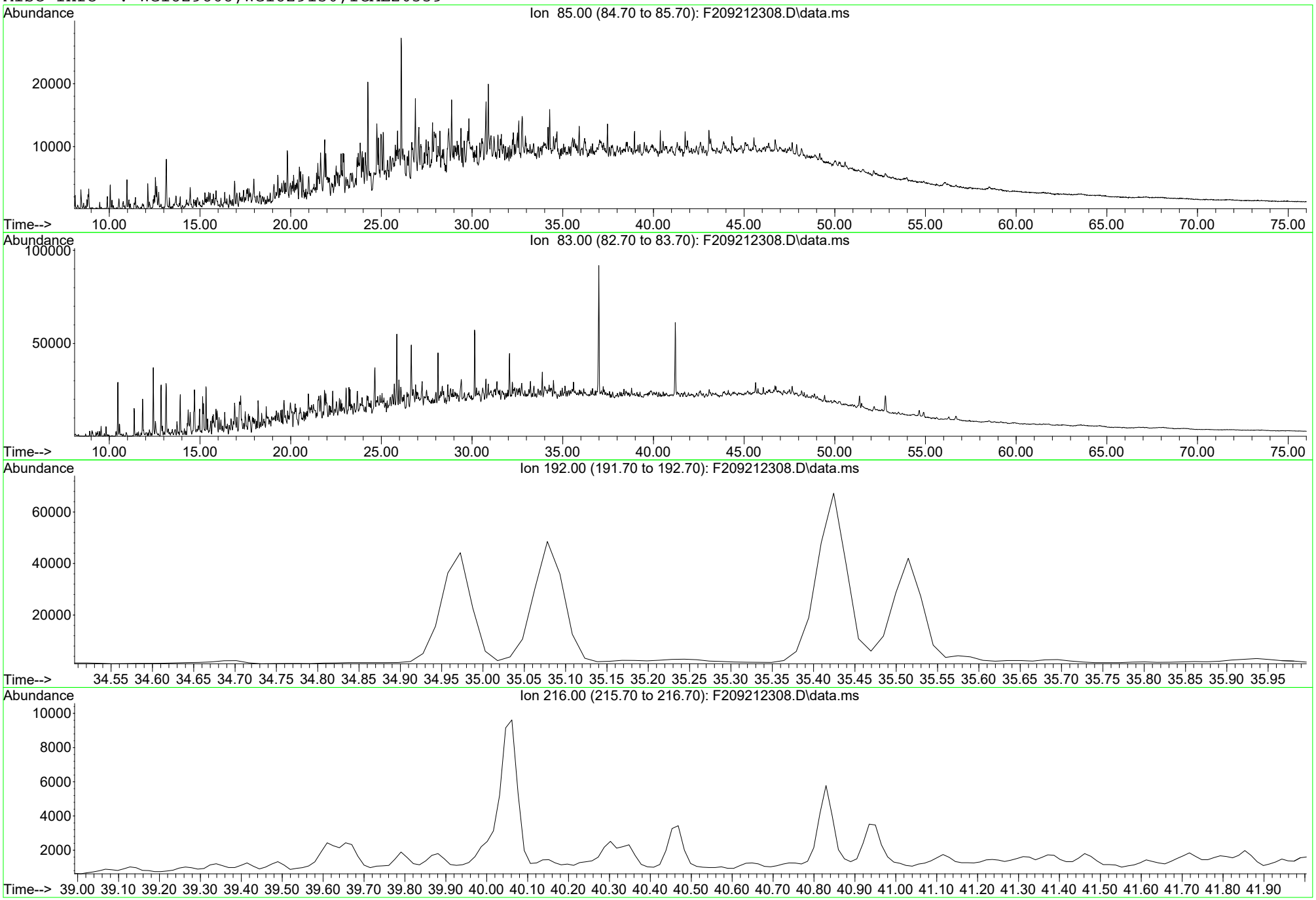
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182318.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 20 Sep 2023 2:50 am using AcqMethod FRNC2A.M
Sample Name: l2348036-03,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

7B Reference Oil
L2348036-03



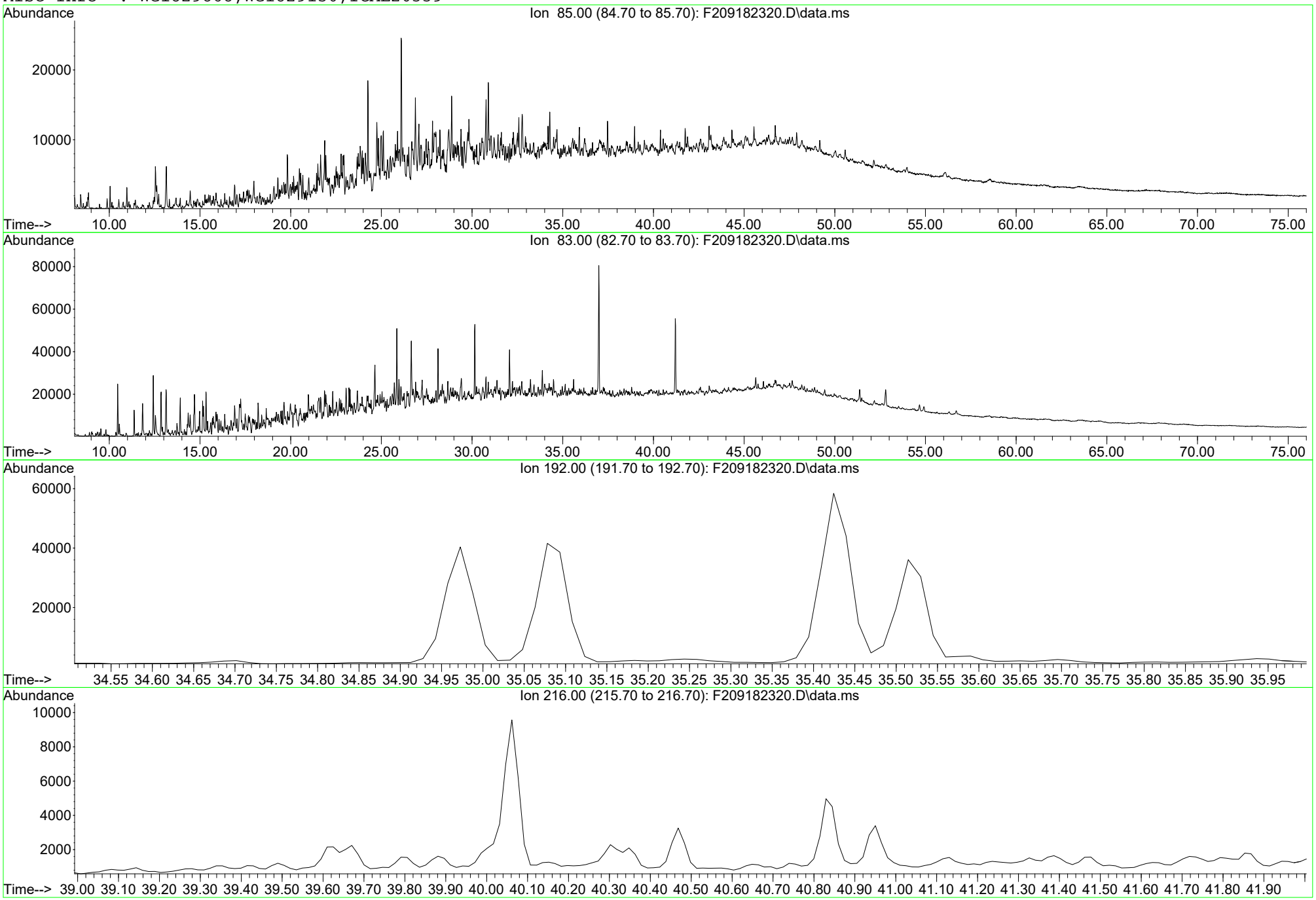
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209212308.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 21 Sep 2023 9:25 pm using AcqMethod FRNC2A.M
Sample Name: l2348036-04,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

SN 189416 (1,250')
L2348036-04



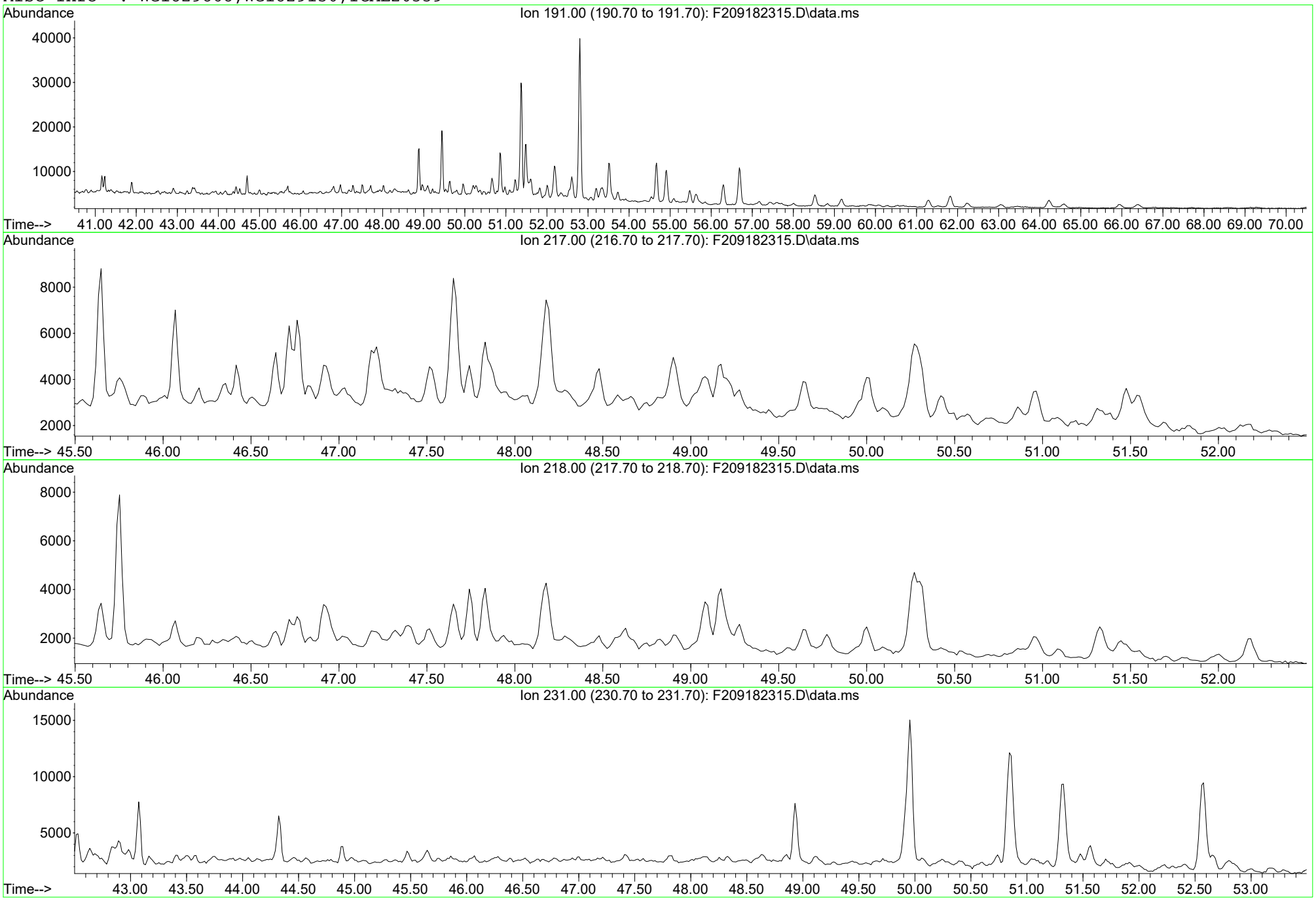
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182320.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 20 Sep 2023 5:40 am using AcqMethod FRNC2A.M
Sample Name: l2348036-05,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

SN 189416 (170')
L2348036-05



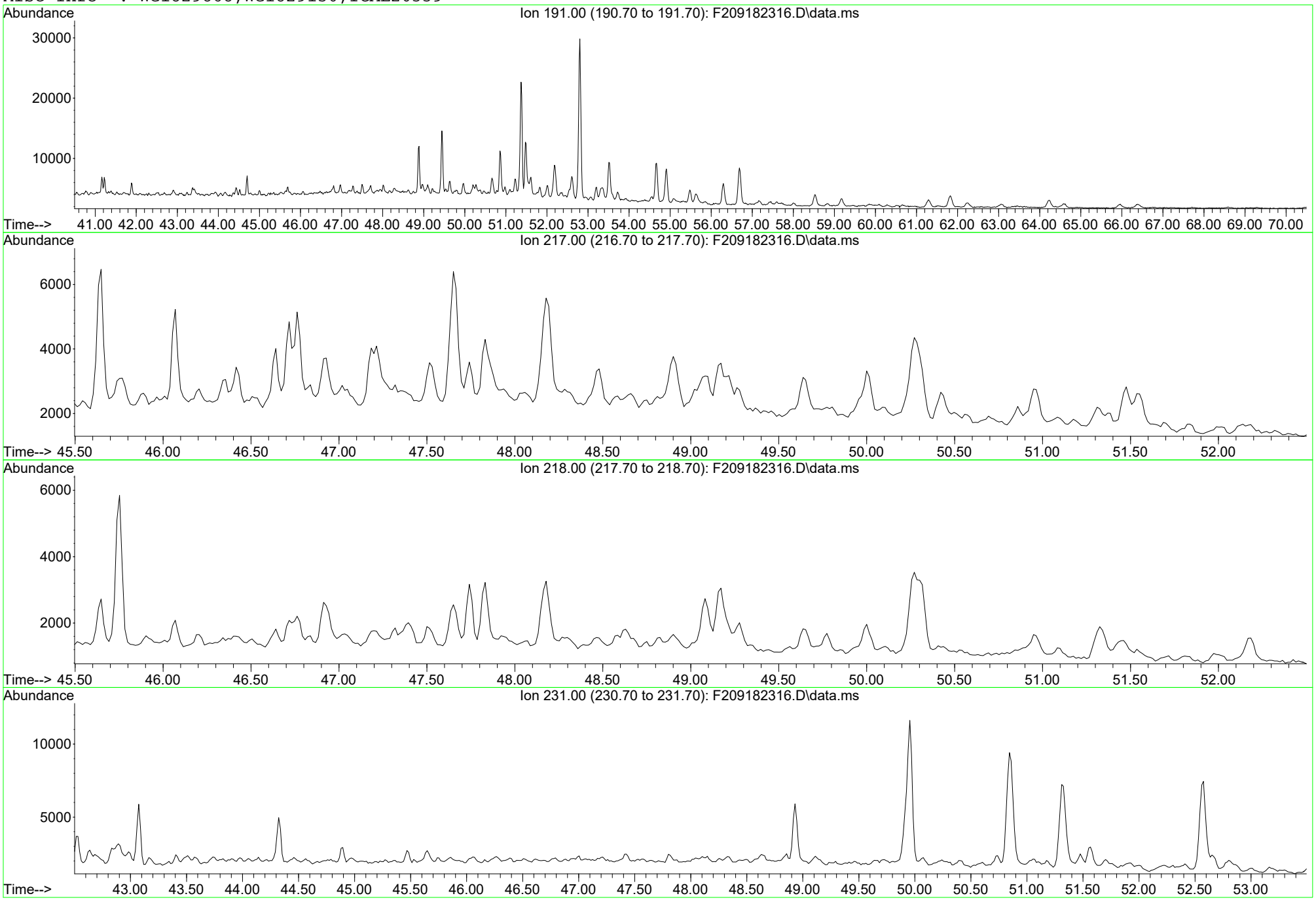
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182315.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 19 Sep 2023 10:33 pm using AcqMethod FRNC2A.M
Sample Name: l2348036-01,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

253998
L2348036-01



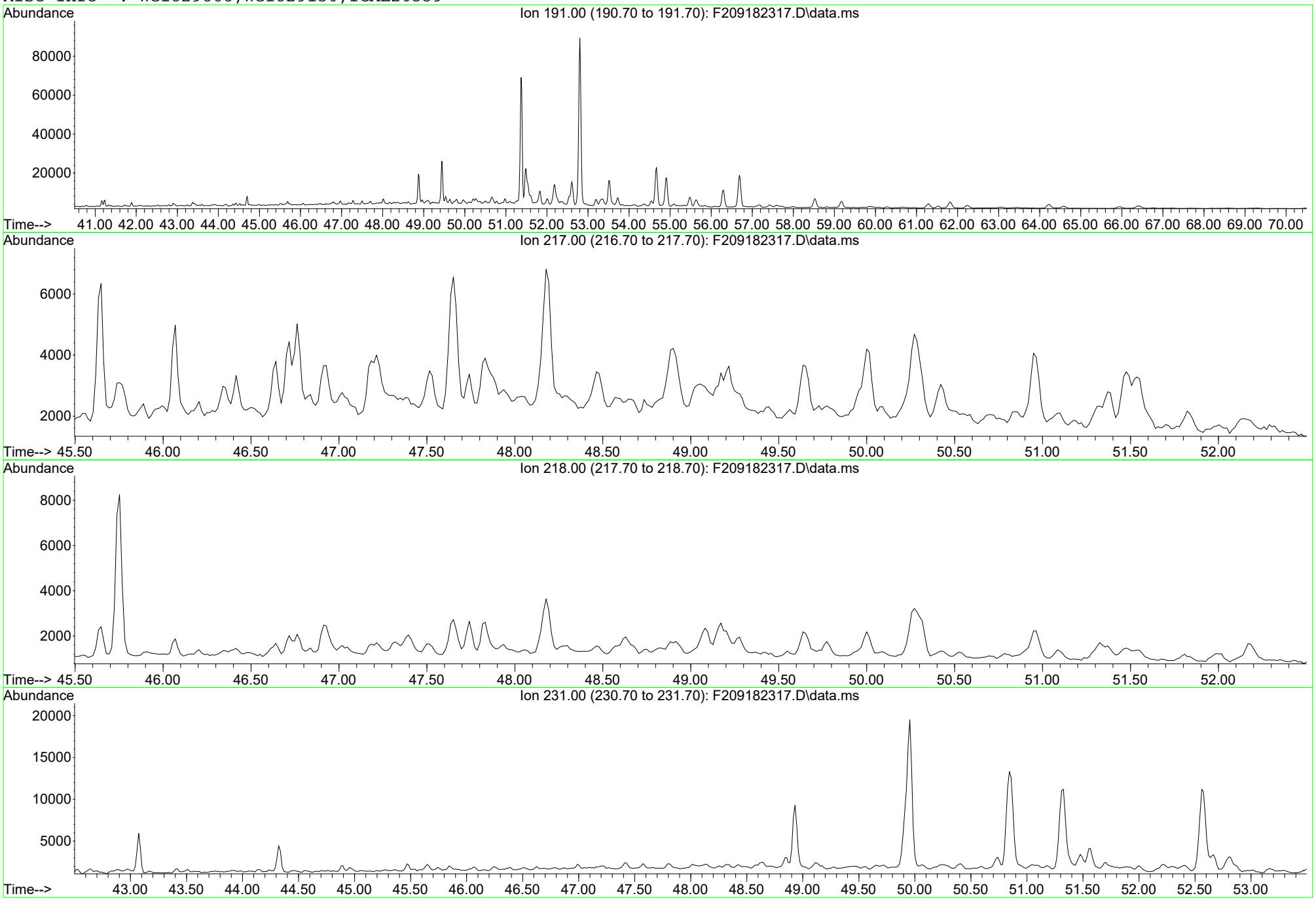
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182316.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 19 Sep 2023 11:59 pm using AcqMethod FRNC2A.M
Sample Name: wg1829150-4,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

L2348036-01 Duplicate
WG1829150-4



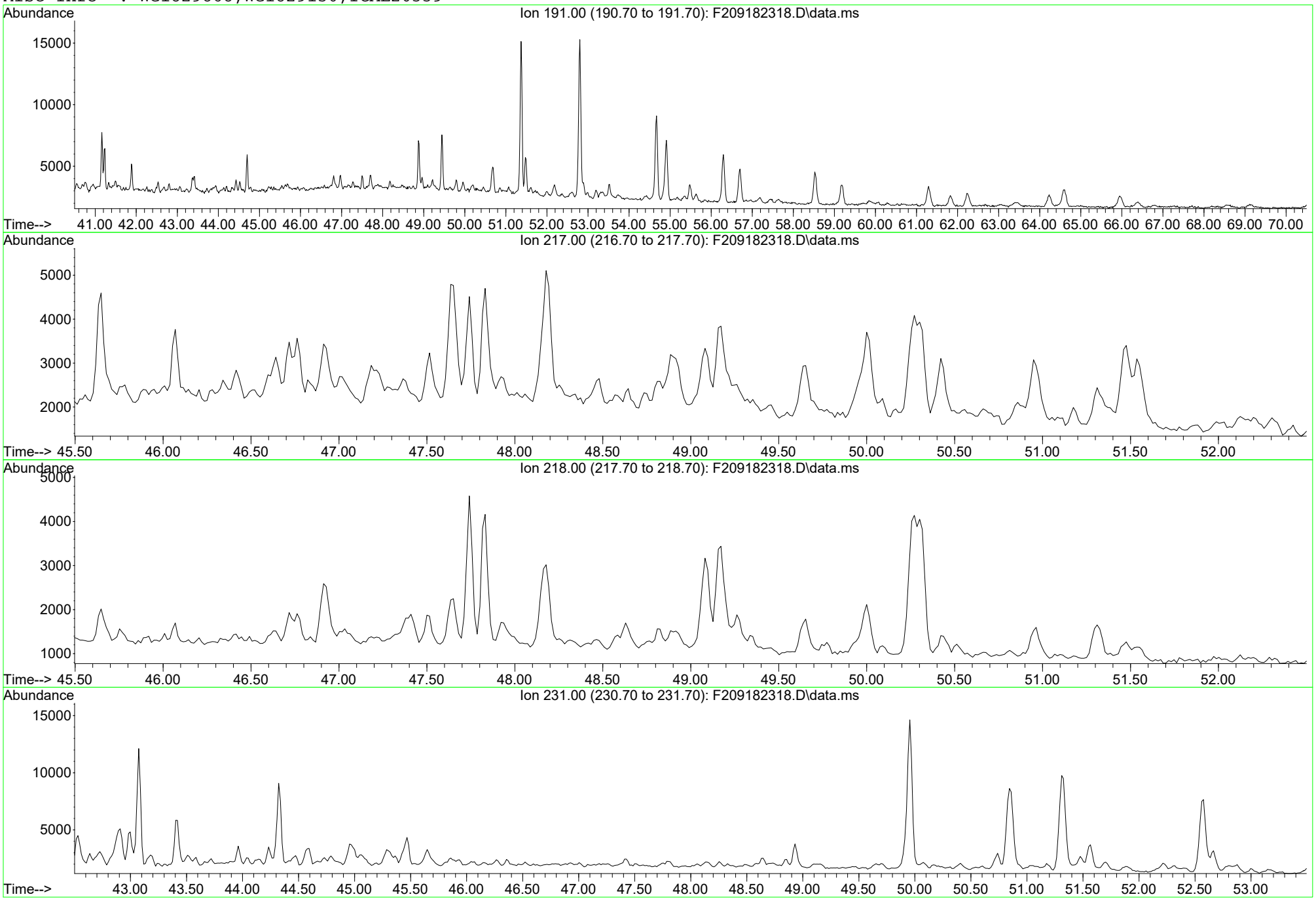
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182317.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 20 Sep 2023 1:24 am using AcqMethod FRNC2A.M
Sample Name: l2348036-02,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

41842
L2348036-02



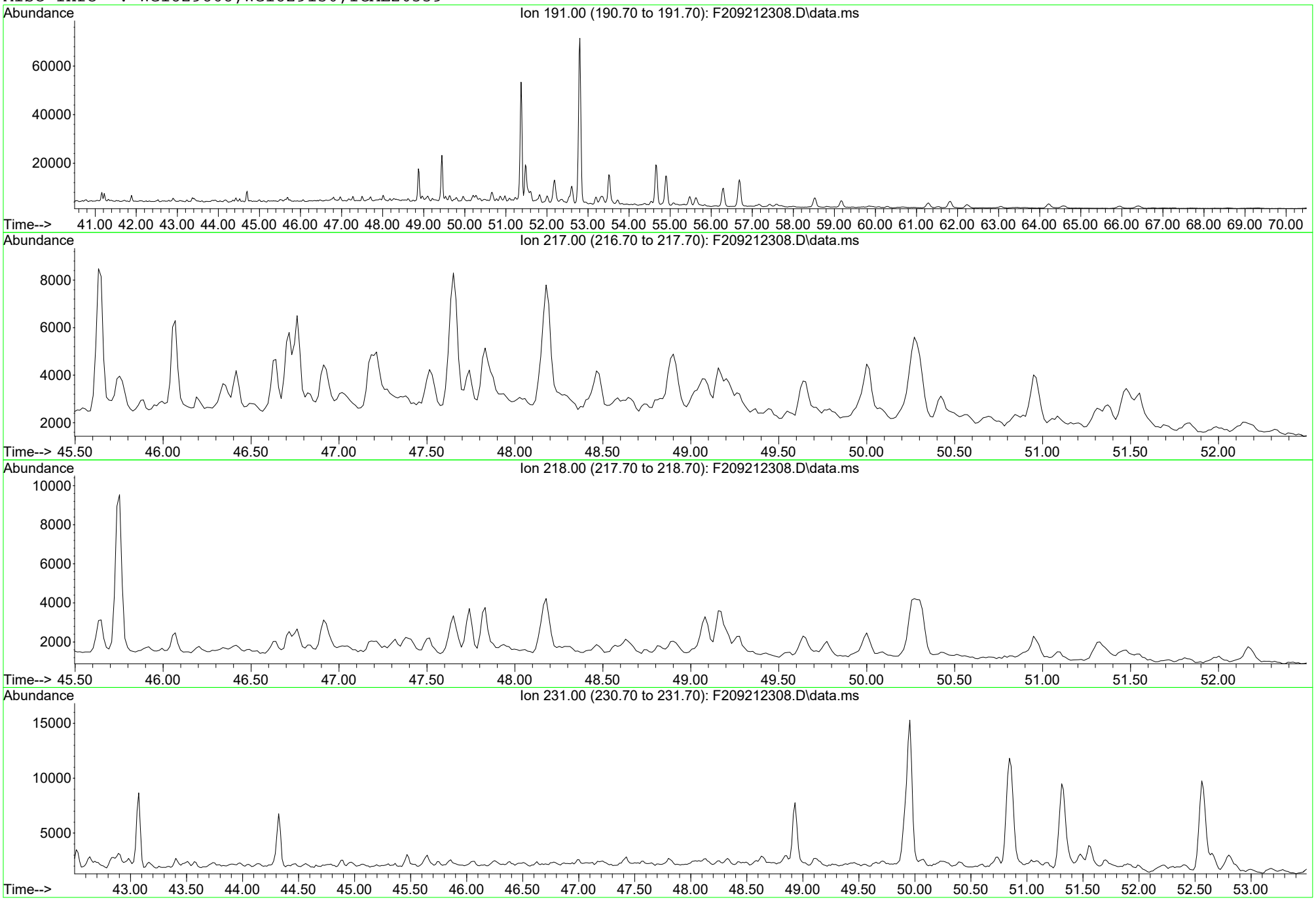
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182318.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 20 Sep 2023 2:50 am using AcqMethod FRNC2A.M
Sample Name: l2348036-03,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

7B Reference Oil
L2348036-03



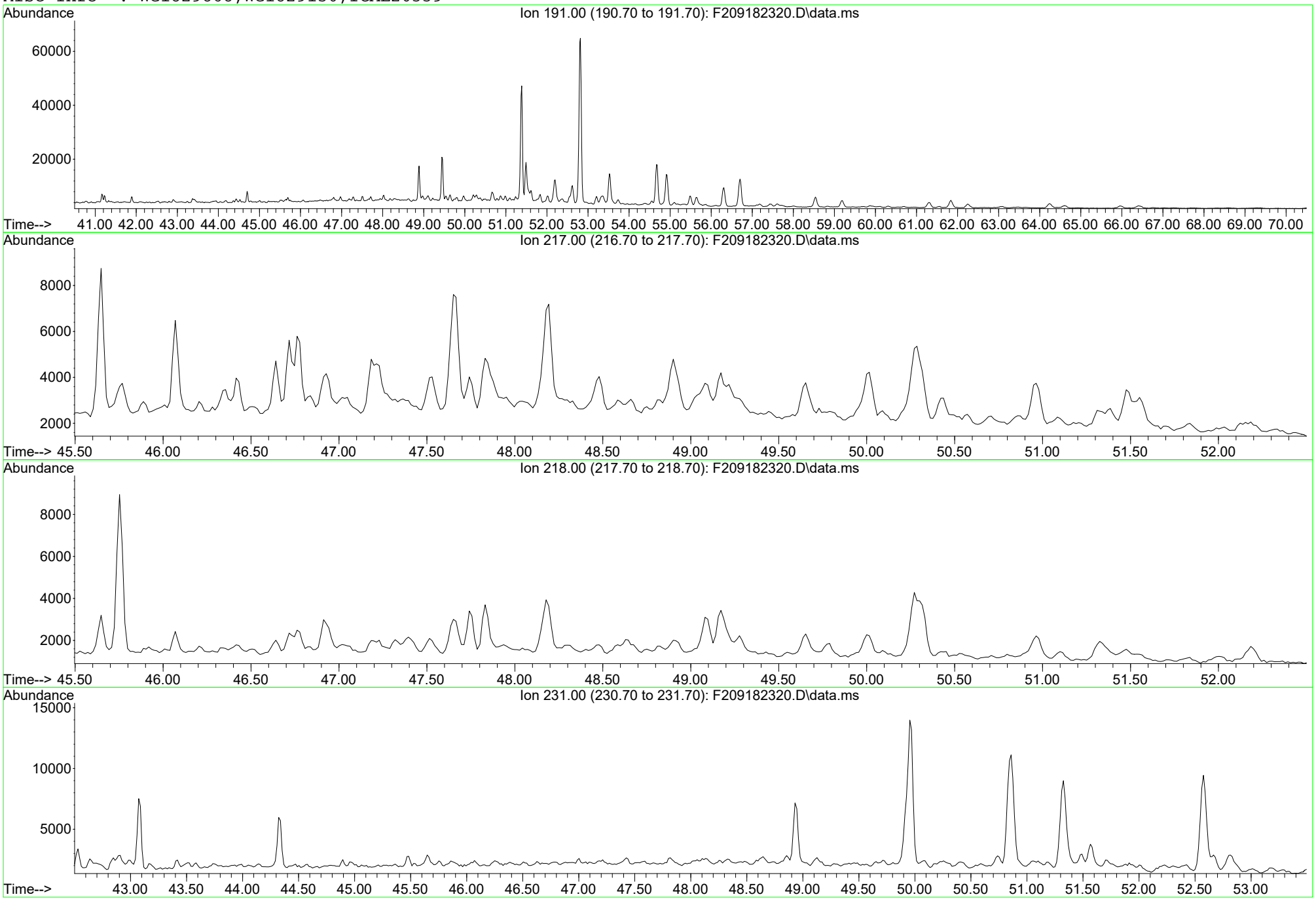
File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209212308.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 21 Sep 2023 9:25 pm using AcqMethod FRNC2A.M
Sample Name: l2348036-04,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

SN 189416 (1,250')
L2348036-04



File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2348036\AL
... KPAHBIO\F209182320.D
Operator : PAH2:CNC
Instrument : PAH2
Acquired : 20 Sep 2023 5:40 am using AcqMethod FRNC2A.M
Sample Name: l2348036-05,32,,
Misc Info : WG1829868,WG1829150,ICAL20339

SN 189416 (170')
L2348036-05



Attachment 7

Crude Oil Assay Results
Samples Studied Herein

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23080858-001A

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Sep. 18, 2023

Station Name: 253998
Method: ASTM D-86
Analyzed: 08/31/2023 00:00:00 by CMN

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 08/17/2023 10:30
Sample Conditions:

ASTM D-86 Distillation

% Recovery	°F @ 762 mm Hg
Initial Boiling Point	144
5	145
10	423
20	NR
30	NR
40	NR
50	NR
60	NR
70	NR
80	NR
85	NR
90	NR
95	NR
Final Boiling Point	437
Volume % Recovery	11.0
Volume % Residue	89.0
Volume % Loss	0.0

Comments: Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.1230

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23080858-001A

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Sep. 18, 2023

Station Name: 253998
Sample Conditions:

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 08/17/2023 10:30

Analytical Data

Test	Method	Result	Units	Detection Limit	Lab Tech.	Analysis Date
Salt in Crude Oil	ASTM D-3230	9850.0	lbs/1000 bbls		MG	08/24/2023
Sulfur Content by X-ray	ASTM D-4294	0.747	wt%		MG	08/24/2023
Organic Chloride	ASTM D-4929	<1.0	ppmw		FSN	09/01/2023
API Gravity @ 60.01 °F	ASTM D-5002	16.88	°		MG	08/24/2023
Specific Gravity @ 60.01/60.01 °F	ASTM D-5002	0.9536	_		MG	08/24/2023
Density @ 60.01 °F	ASTM D-5002	0.9527	g/ml		MG	08/24/2023

Comments:
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.1230

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

CERTIFICATE OF ANALYSIS

ERM
840 W. Sam Houston Parkway
North Suite 600
Houston TX, 77024
Attn: Scott Himes

Report Date: 9/15/2023
Laboratory ID: A230914021
Sample Type: Crude Oil
Sample Date 8/17/2023
Sample ID: 253998 23080858-001A

Tests Requested	Result	Units	Test Method
Iron	**11.7**	ppm	ASTM D5708A
Nickel	**9.6**	ppm	
Vanadium	**2.3**	ppm	

Report Prepared by,

Jaclyn Bazaldua, Lab Technician

Reviewed and Approved by,

Dillon Bagley, Lab Supervisor

1300 Corporate Drive E
Arlington, TX 76006
817-633-9119
817-633-9111 (fax)
info.finishedproducts@spl-inc.com
www.spl-inc.com

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23080858-002A

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Sep. 18, 2023

Station Name: 41842
Method: ASTM D-86
Analyzed: 08/24/2023 00:00:00 by MG

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 08/17/2023 10:50
Sample Conditions:

ASTM D-86 Distillation

% Recovery	°F @ 762 mm Hg
Initial Boiling Point	176
5	264
10	338
20	NR
30	NR
40	NR
50	NR
60	NR
70	NR
80	NR
85	NR
90	NR
95	NR
Final Boiling Point	400
Volume % Recovery	14.0
Volume % Residue	86.0
Volume % Loss	0.0

Comments: Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.1131

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23080858-002A

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Sep. 18, 2023

Station Name: 41842
Sample Conditions:

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 08/17/2023 10:50

Analytical Data

Test	Method	Result	Units	Detection Limit	Lab Tech.	Analysis Date
Salt in Crude Oil	ASTM D-3230	10.6	lbs/1000 bbls		MG	08/24/2023
Sulfur Content by X-ray	ASTM D-4294	0.403	wt%		MG	08/24/2023
Organic Chloride	ASTM D-4929	<1.0	ppmw		FSN	09/01/2023
API Gravity @ 60.01 °F	ASTM D-5002	26.55	°		DKK	09/06/2023
Specific Gravity @ 60.01/60.01 °F	ASTM D-5002	0.8953	_		DKK	09/06/2023
Density @ 60.01 °F	ASTM D-5002	0.8944	g/ml		DKK	09/06/2023

Comments:
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.1131

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

CERTIFICATE OF ANALYSIS

ERM
840 W. Sam Houston Parkway
North Suite 600
Houston TX, 77024
Attn: Scott Himes

Report Date: 9/15/2023
Laboratory ID: A230914022
Sample Type: Crude Oil
Sample Date 8/17/2023
Sample ID: 41842 23080858-002A

Tests Requested	Result	Units	Test Method
Iron	**16.3**	ppm	ASTM D5708A
Nickel	**22.2**	ppm	
Vanadium	**2.0**	ppm	

Report Prepared by,

Jaclyn Bazaldua, Lab Technician

Reviewed and Approved by,

Dillon Bagley, Lab Supervisor

1300 Corporate Drive E
Arlington, TX 76006
817-633-9119
817-633-9111 (fax)
info.finishedproducts@spl-inc.com
www.spl-inc.com

SPL, Inc.

Analysis Request Chain of Custody Record

SPL

SPL Work Order No.: | Acct. Mate Code: | Dept. Code | Page 1

Report To: (Company Name): ERM
Address: 840 W. Sam Houston Parkway North, Suite 600
City/State/Zip: Houston TX 77024-4613
Contact: Scott Himes Scott.Himes@erm.com
Phone: 832-209-8811 Fax:

Project/Station Name: | Project/Station Number: | Project/Station Location:

Special Instructions:

Requested TAT*: 10 business days

Indicate Billing Type: (Place "X", where appropriate) Net 30 day Acct. [ ] Check # Credit Card [ ] <<<Contact SPL, Inc for CC payment arrangements.

* Surcharges May Apply (See quote for details)

Invoice To: (Company Name): ERM
Address: 840 W. Sam Houston Parkway North, Suite 600
City/State/Zip: Houston TX 77024-4613
Contact: Scott Himes Scott.Himes@erm.com
Phone: 832-209-8811 Fax:

Client PO# or Ref. No.: 0701093
Contract/Proposal #: (i.e. SPLQ####) SPLQ10978

† Terms: Cylinders will be rented for $10/cyl. All cylinders checked out are to be returned within 21 days, whether they contain sample or not. Cylinders not returned after 30 days will be considered lost and will be billed at current replacement cost.

Requested Analysis (Place an "X" next to Sample ID below)

Sample ID (used to log/track sample)	Sample Date	Sample Time	Sample Type (Gas/Liq. /Solid)	Duplicate	Composite	Spot	Cylinder Tracking Info† Cylinder #	Date Out	Date In	AS-D-5002	AS-D-4294	ASTM D-5708	AS-D-3230	ASTM D-[illegible] (BP 400°F	AS-D-4929							Comments
253998	08/17/23	10:30	Liq			X				X	X	X	X	X	X							
41842	08/17/23	10:50	Liq			X				X	X	X	X	X	X							

Sampled By-Print Name: David Sanguinetti
Signature: [signature]

Received By-Company:

Relinquished By-Print Name: David Sanguinetti Date: 8/17/23 Time: 15:15
Signature: [signature]

Received By-Print Name: Date: Time:
Signature:

Relinquished By-Print Name: Date: Time:
Signature:

Received By-Print Name: Date: Time:
Signature:

Relinquished By-Print Name: Date: Time:

Received By-Print Name: [signature] Date: [illegible] Time:

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23090306-001A

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Sep. 21, 2023

Station Name: SN 189416 (1,250')
Method: ASTM D-86
Analyzed: 09/12/2023 00:00:00 by MG

Sampled By:
Sample Of: Liquid Spot
Sample Date: 08/29/2023 09:15
Sample Conditions:

ASTM D-86 Distillation

% Recovery	°F @ 762 mm Hg
Initial Boiling Point	392
5	481
10	NR
20	NR
30	NR
40	NR
50	NR
60	NR
70	NR
80	NR
85	NR
90	NR
95	NR
Final Boiling Point	500
Volume % Recovery	8.0
Volume % Residue	92.0
Volume % Loss	0.0

Comments: Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.0739

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23090306-001A

Sep. 21, 2023

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Station Name: SN 189416 (1,250')
Sample Conditions:

Sampled By:
Sample Of: Liquid Spot
Sample Date: 08/29/2023 09:15

Analytical Data

Test	Method	Result	Units	Detection Limit	Lab Tech.	Analysis Date
Salt in Crude Oil	ASTM D-3230	232.0	lbs/1000 bbls		ES	09/13/2023
Sulfur Content by X-ray	ASTM D-4294	0.450	wt%		EC	09/13/2023
Organic Chloride	ASTM D-4929	9.0	ppmw		FSN	09/21/2023
API Gravity @ 60.01 °F	ASTM D-5002	20.65	°		DKK	09/13/2023
Specific Gravity @ 60.01/60.01 °F	ASTM D-5002	0.9300	_		DKK	09/13/2023
Density @ 60.01 °F	ASTM D-5002	0.9291	g/ml		DKK	09/13/2023

Comments:
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.0739
Dilution Factor = 19.90

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

CERTIFICATE OF ANALYSIS

ERM
840 W. Sam Houston Parkway
North Suite 600
Houston TX, 77024
Attn: Scott Himes

Report Date: 9/15/2023
Laboratory ID: A230914037
Sample Type: Crude Oil
Sample Date 8/29/2023
Sample ID: SN 189416 (1,250')
23090306-001B

Tests Requested	Result	Units	Test Method
Iron	**204**	ppm	ASTM D5708A
Nickel	**10.9**	ppm	
Vanadium	**<0.1**	ppm	

Note: Vanadium result was below the detection limit

Report Prepared by,

Jaclyn Bazaldua, Lab Technician

Reviewed and Approved by,

Dillon Bagley, Lab Supervisor

1300 Corporate Drive E
Arlington, TX 76006
817-633-9119
817-633-9111 (fax)
info.finishedproducts@spl-inc.com
www.spl-inc.com

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23090306-002A

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Sep. 21, 2023

Station Name: SN 189416 (170')
Method: ASTM D-86
Analyzed: 09/12/2023 00:00:00 by MG

Sampled By:
Sample Of: Liquid Spot
Sample Date: 08/29/2023 09:30
Sample Conditions:

ASTM D-86 Distillation

% Recovery	°F @ 762 mm Hg
Initial Boiling Point	390
5	495
10	NR
20	NR
30	NR
40	NR
50	NR
60	NR
70	NR
80	NR
85	NR
90	NR
95	NR
Final Boiling Point	500
Volume % Recovery	6.0
Volume % Residue	94.0
Volume % Loss	0.0

Comments: Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.0521

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

Houston Laboratories
8820 Interchange Drive
Houston, TX 77054
Phone 713-660-0901

Certificate of Analysis

Number: 1030-23090306-002A

Sep. 21, 2023

Scott Himes
ERM
840 W. Sam Houston Parkway North
Houston, TX 77024-4613

Station Name: SN 189416 (170')
Sample Conditions:

Sampled By:
Sample Of: Liquid Spot
Sample Date: 08/29/2023 09:30

Analytical Data

Test	Method	Result	Units	Detection Limit	Lab Tech.	Analysis Date
Salt in Crude Oil	ASTM D-3230	88.0	lbs/1000 bbls		ES	09/13/2023
Sulfur Content by X-ray	ASTM D-4294	0.447	wt%		EC	09/13/2023
Organic Chloride	ASTM D-4929	6.2	ppmw		FSN	09/21/2023
API Gravity @ 60.01 °F	ASTM D-5002	20.80	°		DKK	09/13/2023
Specific Gravity @ 60.01/60.01 °F	ASTM D-5002	0.9291	_		DKK	09/13/2023
Density @ 60.01 °F	ASTM D-5002	0.9282	g/ml		DKK	09/13/2023

Comments:
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.0521
Dilution Factor = 19.95

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

Quality Assurance: The above analyses are performed in accordance with ASTM, UOP, GPA guidelines for quality assurance, unless otherwise stated.

CERTIFICATE OF ANALYSIS

ERM
840 W. Sam Houston Parkway
North Suite 600
Houston TX, 77024
Attn: Scott Himes

Report Date: 9/15/2023
Laboratory ID: A230914038
Sample Type: Crude Oil
Sample Date 8/29/2023
Sample ID: SN 189416 (170')
23090306-002B

Tests Requested	Result	Units	Test Method
Iron	**179**	ppm	ASTM D5708A
Nickel	**8.3**	ppm	
Vanadium	**<0.1**	ppm	

Note: Vanadium result was below the detection limit

Report Prepared by,

Jaclyn Bazaldua, Lab Technician

Reviewed and Approved by,

Dillon Bagley, Lab Supervisor

1300 Corporate Drive E
Arlington, TX 76006
817-633-9119
817-633-9111 (fax)
info.finishedproducts@spl-inc.com
www.spl-inc.com

In-person

SPL, Inc.

Analysis Request Chain of Custody Record

SPL Work Order No.:	Acct. Mate Code:	Dept. Code	Page	Pages
			1	1

Report To: (Company Name):	ERM			Project/Station Name:	Project/Station Number:	Project/Station Location:	Requested TAT*
Address:	840 W. Sam Houston Parkway North Suite 600			Special Instructions:			10 business days
City/State/Zip:	Houston	TX	77024-4613				
Contact:	Scott Himes	Scott.Himes@erm.com		Indicate Billing Type: (Place "X", where appropriate)	Net 30 day Acct.	Check #	* Surcharges May Apply (See quote for details)
Phone:	832-209-8811	Fax:			Credit Card	<<<Contact SPL, Inc for CC payment arrangements.	
Invoice To: (Company Name):	ERM						
Address:	840 W. Sam Houston Parkway North Suite 600			†Terms: Cylinders will be rented for $10/cyl. All cylinders checked out are to be returned within 21 days, whether they contain sample or not. Cylinders not returned after 30 days will be considered lost and will be billed at current replacement cost.			
City/State/Zip:	Houston	TX	77024-4613				
Contact:	Scott Himes	Scott.Himes@erm.com					
Phone:	832-209-8811	Fax:					
Client PO# or Ref. No.:	0701093						
Contract/Proposal #: (i.e. SPLQ####)	SPLQ10978						

Sample ID (used to log/track sample)	Sample Date	Sample Time	Sample Type (Gas/Liq. /Solid)	Duplicate	Composite	Spot	Cylinder Tracking Info† Cylinder #	Date Out	Date In	AS-D-5002	AS-D-4294	ASTM D-5708	AS-D-3230	ASTM D-86-IBP-400°F	AS-D-4929							Comments
										Requested Analysis (Place an "X" next to Sample ID below)												
SN 189416 (1,250')	08/29/23	09:15	Liq			X				X	X	X	X	X	X							
SN 189416 (170')	08/29/23	09:30	Liq			X				X	X	X	X	X	X							

Rec 08/30/23 14:37

Sampled By-Print Name:	Scott Himes			Received By-Company:			
Signature:							
Relinquished By-Print Name:	Scott Himes	Date:	Time:	Received By-Print Name:		Date:	Time:
Signature:		8/30/23	1436	Signature:			
Relinquished By-Print Name:		Date:	Time:	Received By-Print Name:		Date:	Time:
Signature:				Signature:			
Relinquished By-Print Name:		Date:	Time:	Received By-Print Name:		Date:	Time:
Signature:				Signature:	Trav Lay		

Choose SPL Facility>>>	Corporate HQ - Houston, TX	Ship to Address:	8820 Interchange Dr., Houston,TX 77054	Phone:	713.660.0901

Note - As a convenience to our clients, this form is available in an electronic format. Please contact one of our offices above for the form to be e-mailed to you.