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July 11, 2023 – Amended (July 14, 2023)

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### *Chemical Fingerprint of 7B Cavern Oil, Cavern 4 Oil, Select Yellow Rock Well Oils, and a Bubble Site 24 Sheen – May 2023*
### *Westlake Sulphur Dome Study*

Dear Mr. Charpentier,

NewFields is pleased to provide you with this amended report, which replaces my original report issued July 11, 2023. At your request I have issued this amendment to include the crude oil assay data provided to me on the six oil samples included in this study. These crude oil assay data, which are contained in the newly added **Attachment 7**, are discussed in a new section that begins on page 8, which references a new table added herein (Table 4). There are no other changes to my original July 11 report.

This report contains the chemical fingerprinting results for six oil samples and one oil/sheen sample collected in the course 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). All seven samples were collected on various dates in May 2023.

This study follows three earlier chemical fingerprinting studies at the Site.[1,2,3] These earlier studies included multiple oils from the 7B cavern well (collected in Jan. 2023 and March 2023) and one oil sample from a nearby Yellow Rock well (110159). Among other conclusions, these earlier studies showed:

- There was no change in composition of the 7B cavern oil between January and March 2023.
- Both 7B cavern oils were chemically distinct from the lone local crude oil studied (Yellow Rock 110159).
- Surface sheens at No. 22 excavation (Jan. 2023) and Bubble Site No. 20 (March 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 oils from three additional nearby Yellow Rock wells and the Yellow Rock tank battery, as well as oils collected (again) from the 7B cavern well and from Cavern 4. Additionally, a sheen sample collected from Bubble Site No. 24 also was assessed.

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[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.

***Samples***

**Table 1** provides an inventory of samples included in this study – along with those previously studied for ease of reference. The newly studied samples were collected on three dates in May 2023 by personnel from ERM. The samples were sent to NewFields' alliance laboratory, Alpha Analytical (Mansfield, Massachusetts, USA), on four 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 (again) determine if the character of the oil within the 7B cavern on May 25, 2023 has remained as it was in January and March 2023, or possibly has changed in a manner that suggests locally produced crude oil has or may have entered the cavern. In addition, the study of four additional locally produced oils allowed for a preliminary assessment of 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 7B. Finally, the character of oils within Cavern 4 and comprising a sheen at Bubble Site No. 24 were studied to determine their possible relationship to 7B cavern oil or locally produced crude oil(s).

***Methods***

These objectives were pursued using specific chemical fingerprinting and interpretation methods based on the CEN oil spill identification protocol[4], as were described in the original study's report, its attachments, and the flowchart depicted in **Figure 1**.[5] The chemical fingerprinting analyses performed herein remain unchanged from the previous reports and are described in **Attachment 2**. However, this study includes the (re-)analysis of the cavern 7B oil collected in January 2023, hereafter adopted as a *site-specific reference oil*. This oil will be repeatedly re-analyzed 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 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 L2325505). 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**.

Specific results most relevant to the study's objectives are presented in **Tables 2 and 3** and **Figures 2 to 5**. Discussion of these results is provided in the following sections.

Character of May 2023 7B Cavern Oil

**Figure 2** shows the Tier 1 GC/FID (C8+) chromatogram for 7B cavern oil collected on May 25, 2023 (Fig. 2A), along with those for the 7B cavern oils collected previously in January and March

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[4] 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.

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

2023 (Fig. 2B and 2C). Inspection reveals an obvious similarity between all three 7B cavern oil samples collected to date.

The 7B cavern oil samples contain compounds that extend up to ~C40. Resolved compounds (peaks) over this range are dominated by n-alkanes that decline in abundance with increasing carbon number. These prominent n-alkanes occur atop a broad, low unresolved complex mixture (UCM) spanning both oils' chromatograms. Also resolved are numerous acyclic isoprenoids, including pristane (Pr) and phytane (Ph) that occur in similar but not identical proportions to each other in all three cavern oils (Pr/Pr ~1.0) and to nearby n-alkanes (C17/Pr and C18/Ph; see Fig. 2 insets and Table 2).

The more detailed characteristics of the 7B cavern oil collected in May 2023 are revealed in its Tier 2 GC/MS extracted ion profiles (EIPs) contained in Attachment 6, which reveal the distributions of various polycyclic aromatic hydrocarbons (PAHs; methyl-phenanthrenes and methyl-fluoranthenes) and petroleum biomarkers (triterpanes, steranes, and triaromatic steroids) used in oil spill fingerprinting. Inspection of these EIPs further reveals the comparability of the 7B cavern oil collected in May 2023 to the two 7B cavern oils collected and analyzed previously.[6]

**Table 2** provides an inventory of the 30 diagnostic ratios (DRs) calculated from the concentrations of selected PAHs and biomarkers in the 7B cavern oils collected to date, including the May 2023 sample analyzed herein. (Because each of the 7B cavern oil samples were analyzed in duplicate the average DRs of each duplicate pair are given.) 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 in Attachment 3) are "greyed out" as they are less likely to exhibit true differences among these oils using the CEN protocol's 95% confidence level criteria.[7]

The green and red color-coding in Table 2 reveals those diagnostic ratios that statistically match (green) and statistically differ (red) from the January 2023 cavern 7B oil (avg.). All 17 of the most precisely measured DRs in the May 2023 7B cavern oil are statistically matched to the January 2023 cavern oil and 16 of these 17 DRs are statistically matched to the March 2023 cavern oil. Thus, both the previously studied March 7B cavern oil and the currently studied May 7B cavern oils are "positive matches" to the January 7B cavern oil.

Collectively, these results indicate that:

- The specific character of the 7B cavern oil has not changed since monitoring of the cavern oil began in January 2023.
- All three cavern oils analyzed to date are comprised of the same unweathered[8] crude oil.

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[6] The EIPs for these previously analyzed 7B cavern oil samples from January and March were contained in attachments to earlier reports.

[7] 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).

[8] *Unweathered* is used here since this oil exhibits no obvious evidence of *weathering*, a term that refers to changes an oil can experience due to various processes (e.g., evaporation, water-washing, photo-oxidation, biodegradation). The changes due to weathering are well recognized and accounted for in oil

- Notable distinguishing features of the 7B cavern oil are reflected in several DRs (Table 2) including:
  - Relatively high amounts of tetracyclic terpane (high T6a/T6bc ratio), Ts (T11/T12 and T11/T19), bisnorhopane (T14a/T19), and homohopanes (T21/T19 and T34 to T35/T19);
  - Near equal amounts of pristane and phytane (Pr/Ph) and of norhopane and hopane (T15/T19); and
  - Relatively low amounts of oleanane (T18/T19) and moretanes (T20/T19)

In addition, and as was previously observed, the 7B cavern oils exhibit conventional proportions of C32 homohopane epimers (T27/T26), a feature that is distinct from the Yellow Rock locally produced crude oils (see below).

## Character of the Other Oils Studied and Their Comparison to 7B Cavern Oil

Three types of "Other Oils" were studied herein. These included oil from Cavern 4, four locally produced crude oils from three individual Yellow Rock wells and the Yellow Rock tank battery, and oil/sheen from Bubble Site No. 24 (Table 1). In this section, these three types of oils are described and compared to the 7B cavern oil. Facilitating in this discussion is **Figure 3** that shows the Tier 1 GC/FID (C8+) chromatograms for these six other oils.

### *Tier 1 Character of the Other Oils Studied*

**Cavern 4 Oil**: The Cavern 4 oil's chromatogram (Fig. 3A) is dominated by n-alkanes that decline in abundance with increasing carbon number, a broad, low unresolved complex mixture (UCM) spanning the oil's chromatogram, and numerous acyclic isoprenoids, including pristane (Pr) and phytane (Ph) that occur in similar proportions to each other (Pr/Pr ~0.9) and to nearby n-alkanes (C17/Pr and C18/Ph; see Fig. 2A inset). These Tier 1 features indicate that:

- The oil collected from Cavern 4 on May 25, 2023 is comprised of an unweathered crude oil that appears to be, based on Tier 1, consistent with oils collected from the 7B cavern (compare Fig. 3A with Fig. 2).

**Yellow Rock Wells and Tank Battery Oils**: The four oils collected from the three Yellow Rock wells and from the Yellow Rock tank battery exhibit comparable Tier 1 chromatographic features that allow them to be described together.

All four Yellow Rock oils' chromatograms are dominated by a prominent unresolved complex mixture (UCM) that appears as a large, broad bimodal hump on the chromatograms (Figs. 2B-2E). There are only minor n-alkanes present and, except for the Yellow Rock tank battery oil (Fig. 3E), the acyclic isoprenoids (Pr and Ph) appear significantly reduced. The most prominent resolved peaks present in the Yellow Rock oils studied are alkylated benzenes, decalins, and naphthalenes below ~C15 and triterpane biomarkers (norhopane and hopane) around ~C30 (Fig. 3B-E). Notably, the Pr/Ph ratio in the Yellow Rock tank battery oil is ~3.1, i.e., much higher than the Cavern 4 oil (Fig. 3A) or the 7B cavern oils (Fig. 2). However, these four Yellow Rock oils' Tier 1 features match those exhibited by the only Yellow Rock well oil studied (and described) previously; Yellow Rock 110159.[9] These Tier 1 features indicate that:

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spill identification protocol, which instead focuses upon those chemical fingerprinting features resistant to weathering.

[9] Stout, S.A. (2023) Chemical fingerprinting of oils, Westlake Sulphur Dome Study. NewFields Report dated March 10, 2023.

- The four Yellow Rock oils collected in May 2023 from three nearby wells (209459, 185997, 210185) and from the Yellow Rock tank battery are comprised of slightly varying moderately biodegraded crude oils.

**Bubble Site No. 24 Oil/Sheen**: The Tier 1 chromatogram for the oil collected from Bubble Site No. 24 is essentially devoid of compounds boiling below ~C15 (Fig. 3F). This feature indicates the oil/sheen has experienced severe evaporation, which is not surprising given its collection from the bubble site. The remaining oil is dominated by a UCM spanning from ~C15 to C45 with no resolved n-alkanes. Resolved peaks recognized as norhopane or hopane are absent but there are some resolved compounds and a “pointy” unresolved mass. This “pointy” mass is a characteristic feature of elemental sulfur, which is present in the oil/sheen. This sulfur in not part of the oil but apparently derived from the area’s sulfur-rich surface environment. These Tier 1 features indicate that:

- The oil/sheen present at Bubble Site No. 24 on May 22, 2023 is comprised of a severely evaporated and severely biodegraded crude oil containing excess elemental sulfur.

Based only on the Tier 1 results described in the preceding paragraphs, the Cavern 4 oil does not exhibit any significant differences from the 7B cavern oil whereas the locally produced Yellow Rock oils and Bubble Site No. 24 oil/sheen clearly do (Fig. 3). The latter could justify a “non-match” conclusion between the 7B cavern oil and any Yellow Rock oils (per Fig. 1) but because the dominant differences are due to weathering (biodegradation and/or evaporation) of the Yellow Rock oils it is still prudent to conduct more detailed Tier 2 comparisons reliant on weathering resistant features afforded by PAHs and biomarkers.

*Tier 2 Character of the Other Oils Studied and Comparison to 7B Cavern Oil*

Qualitative comparisons of the various PAH- and biomarker-based EIPs (Attachment 6) yield no anomalous observations. Specifically, (1) most details of the Cavern 4 oil appear quite similar to those of the 7B cavern oil, (2) details of the four Yellow Rock oils appear quite similar to one another but are distinct from the 7B cavern oils (and Cavern 4 oil), and (3) the Bubble Site No. 24 oil/sheen is so severely weathered that many (but not all) PAHs and biomarkers are altered by weathering.

As an example, demonstrating the first two of these three observations, **Figure 4** shows the partial (*m/z* 191) EIPs showing the distributions of terpenoid biomarkers in the 7B cavern oil, Cavern 4 oil, and one of the Yellow Rock oils (209459) studied herein. The similarity between the 7B cavern oil (Fig. 4A) and the Cavern 4 oil (Fig. 4B) is evident in their relative high abundances of tricyclic terpanes (T4 to T10), tetracyclic terpane (T6a), norhopane (T15), and homohopanes (T21 to T35) and relative low abundances of oleanane (T19) and moretanes (T17 and T20). Despite these similarities a notable difference is also evident in the low relative abundance of bisnorhopane (T14a) in the Cavern 4 oil (Fig. 4B) compared to the 7B cavern oil (Fig. 4A). This difference, reflected in the diagnostic ratio T14a/T19, is notable because it cannot be explained by weathering; i.e., it is a source-specific difference; see more below.

Comparison of the terpenoids in the Yellow Rock well (209459) oil to the 7B cavern oil reveals multiple differences including the former’s relatively low abundances of tricyclic terpanes (T4 to T10), norhopane (T15), and homohopanes (T21 to T35) and relatively high abundances of oleanane (T19) and moretanes (T17 and T20; Fig. 4C). The Yellow Rock well oil also exhibits the unusual abundance of 22R-bishomohopane (T27; Fig. 4C), which was also recognized in the lone, previously studied Yellow Rock well (110159) oil.[10] As described previously, this feature is

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[10] Footnote 1

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.

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 3** wherein the 7B cavern oil collected in May 2023 is statistically compared to those for the Cavern 4 oil, the four Yellow Rock oils, and the Bubble Site No. 24 oil/sheen. As was described above in reference to Table 2, 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 in Attachment 3) are "greyed out" as they are less likely to exhibit true differences among these oils using the CEN protocol's 95% confidence level criteria.

The green and red color-coding in Table 3 reveals those DRs that statistically match (green) and statistically differ (red) from the January 2023 cavern 7B oil (avg.). Inspection shows that 13 of the 17 most precisely measured DRs in the Cavern 4 oil are statistically matched to the 7B cavern oil. Among the four non-matching DRs is the T14a/T19 ratio, which reflects that relative absence of bisnorhopane in the Cavern 4 oils (that was evident in Fig. 4B, described above). This unexplained difference, along with the others, precludes concluding that the Cavern 4 oil is a "positive match" to the 7B cavern oils. However, its overall comparability evident in both the Tier 1 and Tier 2 data does justify that a "probable match" exists (Table 3). Therefore,

- Although the Cavern 4 oil is very similar to the 7B cavern oil, it is not identical.
- The Cavern 4 oil is, however, unequivocally distinct from the locally produced crude oil as represented by the four Yellow Rock oils studied herein.

Taken together, and assuming the oils in both Cavern 4 and Cavern 7 consist of residual oil from their historic use as part of the Strategic Petroleum Reserve (SPR), it seems evident that Cavern 4 stored a slightly different (blend of) SPR crude oil(s) than was stored in Cavern 7.

As expected, based upon the disparate qualitative (Tier 1 and Tier 2) results described above, the four Yellow Rock crude oils studied show that most DRs statistically differ from those of the 7B cavern oil (Table 3). These multiple differences warrant a "non-match" conclusion for the four Yellow Rock oils studied *versus* the 7B cavern oil. By the same basis, the Bubble Site No. 24 oil/sheen is also concluded to be a "non-match" to the 7B cavern oil (Table 3).[11] Therefore,

- The four locally produced Yellow Rock crude oils (three wells and tank battery) studied herein are unequivocally distinct from the 7B cavern oil; and
- The Bubble Site No. 24 oil/sheen is unequivocally distinct from the 7B cavern oil.

Owing to the consistent character of the 7B cavern oil between January and May 2023 (see above; Table 2) and the 7B cavern oil's "non-match" character *versus* the locally produced Yellow Rock oils (Table 3):

- There is no chemical evidence that a locally produced crude oil had or is presently entering Cavern 7.

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[11] Despite the severe biodegradation of this oil/sheen those DRs based on highly recalcitrant biomarkers (e.g., triaromatic steroids and diasteranes) are still statistically distinct from the 7B cavern oil, which allows for the "non-match" conclusion (rather than "inconclusive".

Preliminary Review of the Heterogeneity of Locally Produced Oils

The disparity between the 7B cavern oils and the locally produced oils studied to date is obvious based on their contrasting chemical fingerprints and diagnostic ratios (e.g., Fig. 2 and Table 2). If, in the future, locally produced oil(s) were to enter and mix with the extant 7B cavern oil it should be quite easy to recognize as any mixture of extant cavern oil and locally produced crude oil would exhibit an intermediate chemical fingerprint and diagnostic ratios proportional to the mixture. More difficult may be the ability to determine the specific source (well/reservoir zone) of locally produced oil that may enter and mix with the extant oil in Cavern 7. Therefore, it is worthwhile to preliminarily review any heterogeneity that exists among the five locally produced (Yellow Rock) crude oils' chemical fingerprints and diagnostic ratios studied to date (Table 1).

My basic understanding of the Sulphur Dome area's geology and petroleum system suggests that the potential exists for some variability in the specific character of the locally produced crude oils. Specifically, literature demonstrates this potential often exists in the Gulf Coast region's Tertiary clastic geology wherein the complex deltaic strata, which contains discontinuous and interbedded reservoir and non-reservoir strata that are often cross-cut by faulting (or pinched by salt diapers), can promote reservoir compartmentalization. These compartments can accumulate oil generated from slightly varying source rock strata of the same age (downdip), contain slightly varying amounts of deep-sourced oils migrating vertically along faults, or contain oils that have been variably altered by migration/phase fractionation. Some Gulf Coast oil fingerprinting studies have shown the source-specific character of crude oils from different reservoir strata,[12] and even different reservoir zones within a single oil field,[13,14] can vary.

Inspection of the DRs for the four Yellow Rock oils studied herein (Table 3) shows they share an overall comparability. This overall comparability is not unexpected since any source-specific differences between the wells are expected to be "subtle". Two of the 17 most precisely measured DRs (per Attachment 3), however, exhibit a somewhat greater variability among the four oils, *viz*., C18/Ph and T27/T26. The former ranges from values of 0.22 to 2.38 and the latter ranges from 1.30 to 1.59 (Table 3).

The C18/Ph ratio is not normally considered to be source-specific (in oil spill studies) since it reflects with the level of biodegradation of an oil (and spilled oils will biodegrade over time). However, in this instance the level of biodegradation could be considered source-specific since it clearly varies among the different Yellow Rock oils studied, which reflects different levels of (in-reservoir) biodegradation exist in the different reservoir zones produced by the different Yellow Rock wells studied (to date).

The T27/T26 ratio's variability also may be potentially useful in distinguishing the locally produced crude oils from different wells/reservoir zones. This ratio reflects the relative and unusual abundance of the 22R-bishomohopane (T27) in the locally produced oils. The atypical and varying abundances of T27 likely reflects varying amounts of a co-eluting and anomalous

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[12] Wegner, L.M. et al. (1990) Molecular characteristics of Smackover, Tuscaloosa, and Wilcox-reservoired oils in the eastern Gulf Coast. Proc. Gulf Coast Section – Society of Economic Paleontologists and Mineralogists, 9th Ann. Res. Conf, p. 37-57.

[13] Thompson, K.F.M. (1988) Gas-condensate migration and oil fractionation in deltaic systems. Mar. Petrol. Geol. 5: 237-246.

[14] Kaufman, R.L. et al. (1990) Gas chromatography as a development and production tool for fingerprinting oils from individual reservoirs: Applications in the Gulf of Mexico. Proc. Gulf Coast Section – Society of Economic Paleontologists and Mineralogists, 9th Ann. Res. Conf, p. 263-282.

compound present in the different Yellow Rock oils, perhaps the result of migration-contamination.[15]

**Figure 5** shows a double-ratio plot of these two DRs for all of the 7B cavern oils and Yellow Rock oils studied to date, which includes oil from well 110159 from January 2023 (Table 1). The consistency in the 7B cavern oil composition is reflected in the tight cluster of points representing these samples whereas the scatter among the Yellow Rock oils reflects their variability (Fig. 5). Although preliminary, it can be envisioned that mixtures of the different Yellow Rock oils with the 7B cavern oil may follow different mixing lines/curves – as depicted by the dashed lines in Figure 5. This approach, or a comparably one based on other DRs or metrics (e.g., metals or sulfur content) may provide a reasonable basis to determine which particular Yellow Rock oil might explain a mixture of local crude oil with the extant 7B cavern oil currently present in Cavern 7.

If/when there is evidence that the 7B cavern oil has become mixed with a locally produced crude oil this preliminary approach, or a comparable one, may be revisited and require further development.

## Crude Oil Assay Results – Summary to Date

As part of this amended report, this new section summarizes the standard crude oil assay data that (to my knowledge) were collected over the past several months as part of the Cavern 7 investigation/monitoring. The crude oil assay data collected on the six oil samples studied herein are presented in Attachment 7.

Crude oil assay data are often of limited utility in the chemical fingerprinting of oils. The reason for this is that assay data measure the "bulk" features of crude oils that can be affected by weathering (e.g., biodegradation) and very often do not provide the precision and specificity to distinguish genetically similar oils. However, in this instance, the obvious disparity between the 7B cavern oil and the locally produced (Yellow Rock) oils (see detailed chemical fingerprinting results above) is also reflected in the bulk assay data.

**Table 4** contains selected crude oil assay results for the 7B cavern oil and the locally produced oils studied to date. Inspection reveals differences in API gravity between the two groups, which is markedly lower for the locally produced crude oils owing to their variably biodegraded character. The 7B cavern oil exhibits a consistent and higher sulfur content than the locally produced oils, which themselves most likely vary with biodegradation.[16]

The trace metals most abundant in crude oil – nickel and vanadium – are long-recognized as varying in concentration and proportion among different crude oils.[17] Because these metals resided in a very high boiling (recalcitrant) fraction of crude oil their absolute concentrations can increase with weathering. However, their relative proportion to one another are unaffected by weathering and is most often expressed in the V/(V+Ni) ratio. Inspection of Table 4 shows that the 7B cavern oils contain more vanadium than nickel whereas the locally produced oils contain less vanadium than nickel, causing these two groups of oil to exhibit very different V/(V+Ni) ratios.

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[15] The basis for this hypothesis lies in the ability of migrating oil to "extract" non-indigenous compounds from rocks through which it may migrate or in which it is reservoired; see Curiale, J.A. (2002) A review of the occurrences and causes of migration-contamination in crude oil. Org. Geochem. 33(12): 1389-1400.

[16] Biodegradation tends to increase the sulfur content of oils. Therefore, the higher sulfur content in the (non-biodegraded) 7B cavern oil (Table 4) indicates it is truly a higher sulfur crude oil than the locally produced crude oils.

[17] These differences derived from depositional conditions millions of years ago when an oil's source rock was accumulating; Lewan, M.D. (1984) Factors controlling the proportionality of vanadium and nickel in crude oils. Geochim. Cosmochim. Acta 48, 2231–2238.

In summary;

- The available "bulk" crude oil assay data show that the 7B cavern oil has remained consistent (between November 2022, when it was first analyzed, and May 2023) and is clearly distinguishable from the locally produced oils.

***Summary of Findings***

Chemical fingerprinting of cavern 7B oil collected on May 25, 2023 was conducted and the results were compared to cavern 7B oils collected on January 25, 2023 and on March 30, 2023. Results showed that all three 7B cavern oils are comprised of the same specific type of unweathered crude oil and are "positive matches" to one another. This result shows:

- There has been no change in the chemical composition of the crude oil recovered from the 7B cavern well between (at least) January 25 and May 25, 2023.

Comparison of the 7B cavern oil to four locally produced Yellow Rock crude oils collected between May 2 and 25, 2023 from three wells (209459, 185997, and 210185) and the tank battery are all "non-matches" to the 7B cavern oil. (This conclusion parallels that of the lone Yellow Rock well (110159) oil studied previously; collected in Jan. 2023.) In addition to being variably biodegraded, all of the locally produced crude oils studied to date exhibit many source-specific features that readily distinguish them from the 7B cavern oil studied to date. As such,

- There is no chemical evidence that a locally produced crude oil had or is presently entering Cavern 7. Rather, the distinct 7B cavern oil is assumed to consist of residual (non-local) crude oil from the cavern's tenure as part of the Strategic Petroleum Reserve (SPR).

Oil collected on May 25, 2023 from Cavern 4 is an unweathered crude oil that is similar to, but not identical to, the 7B cavern oil; these oils are "probable matches". The Cavern 4 oil is, however, unequivocally distinct from the locally produced crude oil (as represented by the Yellow Rock oils studied to date). This result indicates:

- There is no chemical evidence that a locally produced crude oil is present in Cavern 4. Rather, Cavern 4 seemingly contains residual (non-local) crude oil from its tenure as part of the SPR, but the SPR oil formerly stored in Cavern 4 oil was slightly different from the SPR oil formerly stored in Cavern 7.

The oil/sheen collected from the Bubble Site No. 24 on May 22, 2023 consists of a severely weathered (evaporated and biodegraded) crude oil. It is contaminated with elemental sulfur likely derived from the local, sulfur-rich environment. The oil/sheen is a "non-match" to 7B cavern oil and, despite its severely weathered character, is consistent with the locally produce crude oil (as represented by the Yellow Rock oils studied to date). Thus,

- The oil/sheen present at Bubble Site No. 24 is derived from (seepage or spillage of) a local crude oil. It is not derived from 7B cavern oil.

A preliminary comparison among the five locally produced Yellow Rock crude oils studied to date (Table 1) shows them to exhibit slightly varying degrees of (in-reservoir) biodegradation. Minor/subtle differences in source-specific features are (perhaps best) expressed in varying abundances of one particular biomarker analyte (T27). The minor/subtle differences among the locally produced crude oils studied to date suggest it may be possible to distinguish the source (well/reservoir zone) of any locally produced oil if any were to become evident within Cavern 7, i.e., become mixed with the extant 7B cavern oil.

Please let me know if you have any questions.

Sincerely,

Scott A Stout

PROFESSIONAL GEOLOGIST
SCOTT A. STOUT
No. 6225
STATE OF CALIFORNIA

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: Crude oil assay data

## 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 |
|---|---|---|---|---|
| 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 |

***Previously-Studies Samples***

| Client/ Field ID | Lab ID | Matrix | Date Collected | Description of Sample |
|---|---|---|---|---|
| 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 within cavern blanket |
| 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 oils collected in May 2023 and March 2023 *versus* the 7B cavern oil collected in January 2023 and analyzed over a span of approximately 4.5 months.

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 | Cavern Oil 7B (Jan. 21, 2023; Avg. n=2) | Cavern Oil 7B (March 30, 2023; Avg. n=2) | Cavern Oil 7B (May 25, 2023; Avg. n=2) |
|---|---|---|---|---|
| | Analysis Date | 2/4/2023 | 4/26/2023 | 6/18/2023 |
| NR-C17/pris | C17/Pr | 2.38 | 2.41 | 2.66 |
| NR-C18/phy | C18/Ph | 2.17 | 2.14 | 2.13 |
| NR- pris/phy | Pr/Ph | 1.01 | 1.00 | 0.90 |
| NR-4-MD/1-MD | 4-MDBT/1-MDBT | 2.14 | 2.71 | 2.15 |
| NR-2-MP/1-MP | 2-MP/1-MP | 1.01 | 0.97 | 1.06 |
| NR-27Ts/30ab | T11/T19 | 0.23 | 0.23 | 0.24 |
| NR-27Tm/30ab | T12/T19 | 0.29 | 0.26 | 0.26 |
| NR-28ab/30ab | T14a/T19 | 0.20 | 0.18 | 0.19 |
| NR-29ab/30ab | T15/T19 | 0.84 | 0.83 | 0.83 |
| NR-30O/30ab | T18/T19 | 0.04 | 0.03 | 0.00 |
| NR-31abS/30ab | T21/T19 | 0.59 | 0.59 | 0.58 |
| NR-27dbR/27dbS | S4/S5 | 0.50 | 0.53 | 0.46 |
| NR-27bb/29bb | (S14+S15)/(S26+S27) | 0.85 | 0.88 | 0.86 |
| NR-SC26/ RC26+SC27 | TAS09/TAS01 | 0.13 | 0.14 | 0.14 |
| NR-SC28/RC26 + SC27 | TAS02/TAS01 | 0.69 | 0.71 | 0.69 |
| NR-RC27/RC26+ SC27 | TAS03/TAS01 | 0.75 | 0.78 | 0.78 |
| NR-RC28/RC26+SC27 | TAS04/TAS01 | 0.58 | 0.59 | 0.58 |
| DR-Ts/Tm | T11/T12 | 0.82 | 0.87 | 0.92 |
| DR-29Ts30ab | T16/T19 | 0.21 | 0.20 | 0.20 |
| DR-29bb/29aa | (S26+S27)/(S25+S28) | 1.15 | 1.82 | 1.33 |
| DR-C2-dbt/C2-phe | DBT2/PA2 | 2.28 | 2.07 | 2.06 |
| DR-C3-dbt/C3-phe | DBT3/PA3 | 2.62 | 2.23 | 2.32 |
| DR-C28C29/30ab | T7 to T10/T19 | 0.19 | 0.23 | 0.24 |
| DR-29aaS/29aaR= | S25/S28 | 1.36 | 1.22 | 1.28 |
| DR-C20TA/C21TA | TAS05/TAS06 | 0.95 | 1.30 | 1.19 |
| DR-TA21/ RC26+SC27 | TS06/TAS01 | 0.49 | 0.40 | 0.40 |
| DR-C24Tet/C26Tri | T6a/T6bc | 1.60 | 1.67 | 1.55 |
| DR-30ba/30ab | T20/T19 | 0.07 | 0.08 | 0.07 |
| DR-35ab/30ab | (T34 to T35)/T19 | 0.33 | 0.33 | 0.31 |
| DR-32abR/32abS | T27/T26 | 0.74 | 0.72 | 0.74 |
| | | **Conclusion:** | **Positive Match** | **Positive Match** |

red: statistical non-match to 7B Cavern Oil (May 25, 2023; Avg.)

green:s statistical match to 7B Cavern Oil (May 25, 2023; Avg.)

grey: indicates less precision ratio (per Attachment 3)

Dup: sample prepared and analyzed in duplicate

Avg: average of duplicate ratios

ndp: no determination possible/division by zero

## Table 3: Diagnostic ratios for the 7B cavern oils collected in May 2023 *versus* Other Oils studied herein.

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 | Cavern Oil 7B (May 25, 2023; Avg. n=2) | Cavern 4 (May 2023) | Yellow Rock 209459 (May 2023) | Yellow Rock 185997 (May 2023) | Yellow Rock 210185 (May 2023) | Yellow Rock Tank Battery (May 2023) | No. 24 Net Sample (May 2023) |
|---|---|---|---|---|---|---|---|---|
| | Analysis Date | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/15/2023 |
| NR-C17/pris | C17/Pr | 2.66 | 3.05 | 0.18 | 0.37 | 0.29 | 0.05 | ndp |
| NR-C18/phy | C18/Ph | 2.13 | 2.24 | 0.85 | 2.38 | 1.27 | 0.22 | 0.67 |
| NR- pris/phy | Pr/Ph | 0.90 | 0.88 | 3.77 | 2.84 | 3.18 | 3.10 | 0.00 |
| NR-4-MD/1-MD | 4-MDBT/1-MDBT | 2.15 | 2.21 | 3.57 | 3.92 | 3.38 | 4.52 | ndp |
| NR-2-MP/1-MP | 2-MP/1-MP | 1.06 | 1.19 | 1.17 | 1.09 | 1.17 | 1.29 | 1.76 |
| NR-27Ts/30ab | T11/T19 | 0.24 | 0.23 | 0.15 | 0.15 | 0.15 | 0.16 | 0.49 |
| NR-27Tm/30ab | T12/T19 | 0.26 | 0.29 | 0.20 | 0.19 | 0.21 | 0.20 | 0.71 |
| NR-28ab/30ab | T14a/T19 | 0.19 | 0.05 | 0.05 | 0.05 | 0.05 | 0.04 | 0.00 |
| NR-29ab/30ab | T15/T19 | 0.83 | 0.93 | 0.61 | 0.60 | 0.61 | 0.58 | 1.29 |
| NR-30O/30ab | T18/T19 | 0.00 | 0.05 | 0.10 | 0.10 | 0.10 | 0.10 | 3.05 |
| NR-31abS/30ab | T21/T19 | 0.58 | 0.56 | 0.26 | 0.25 | 0.26 | 0.25 | 0.00 |
| NR-27dbR/27dbS | S4/S5 | 0.46 | 0.42 | 0.66 | 0.66 | 0.62 | 0.63 | 0.70 |
| NR-27bb/29bb | (S14+S15)/(S26+S27) | 0.86 | 0.80 | 0.76 | 0.67 | 0.72 | 0.69 | 0.76 |
| NR-SC26/ RC26+SC27 | TAS09/TAS01 | 0.14 | 0.11 | 0.30 | 0.34 | 0.32 | 0.35 | 0.32 |
| NR-SC28/RC26 + SC27 | TAS02/TAS01 | 0.69 | 0.80 | 0.80 | 0.81 | 0.80 | 0.81 | 0.80 |
| NR-RC27/RC26+ SC27 | TAS03/TAS01 | 0.78 | 0.80 | 0.63 | 0.64 | 0.60 | 0.64 | 0.59 |
| NR-RC28/RC26+SC27 | TAS04/TAS01 | 0.58 | 0.66 | 0.67 | 0.65 | 0.65 | 0.64 | 0.67 |
| DR-Ts/Tm | T11/T12 | 0.92 | 0.79 | 0.77 | 0.78 | 0.73 | 0.78 | 0.70 |
| DR-29Ts30ab | T16/T19 | 0.20 | 0.20 | 0.24 | 0.24 | 0.25 | 0.22 | 0.54 |
| DR-29bb/29aa | (S26+S27)/(S25+S28) | 1.33 | 1.57 | 1.10 | 1.05 | 0.97 | 0.93 | ndp |
| DR-C2-dbt/C2-phe | DBT2/PA2 | 2.06 | 2.26 | 0.28 | 0.26 | 0.28 | 0.23 | ndp |
| DR-C3-dbt/C3-phe | DBT3/PA3 | 2.32 | 2.53 | 0.38 | 0.35 | 0.37 | 0.29 | ndp |
| DR-C28C29/30ab | T7 to T10/T19 | 0.24 | 0.19 | 0.06 | 0.06 | 0.07 | 0.06 | 1.31 |
| DR-29aaS/29aaR | S25/S28 | 1.28 | 1.17 | 1.23 | 1.10 | 1.21 | 1.06 | ndp |
| DR-C20TA/C21TA | TAS05/TAS06 | 1.19 | 1.24 | 1.27 | 1.33 | 1.22 | 1.29 | 0.00 |
| DR-TA21/ RC26+SC27 | TS06/TAS01 | 0.40 | 0.45 | 0.24 | 0.23 | 0.23 | 0.24 | 0.02 |
| DR-C24Tet/C26Tri | T6a/T19 | 0.14 | 0.16 | 0.04 | 0.04 | 0.04 | 0.04 | 1.00 |
| DR-30ba/30ab | T20/T19 | 0.07 | 0.09 | 0.19 | 0.18 | 0.21 | 0.19 | 0.00 |
| DR-35ab/30ab | (T34 to T35)/T19 | 0.31 | 0.27 | 0.06 | 0.05 | 0.05 | 0.04 | 0.00 |
| DR-32abR/32abS | T27/T26 | 0.74 | 0.72 | 1.50 | 1.59 | 1.48 | 1.30 | ndp |
| | | **Conclusion:** | **Probable Match** | **Non-Match** | **Non-Match** | **Non-Match** | **Non-Match** | **Non-Match** |

red: statistical non-match to 7B Cavern Oil (May 25, 2023; Avg.)

green:s statistical match to 7B Cavern Oil (May 25, 2023; Avg.)

grey: indicates less precision ratio (per Attachment 3)

Dup: sample prepared and analyzed in duplicate

Avg: average of duplicate ratios

ndp: no determination possible/division by zero

## Table 4: Selected crude oil assay results for the 7B cavern oils and locally produced (Yellow Rock) crude oils studied to date.

na – not analyzed

**7B Cavern Oils**

| Client ID | Date Collected | API gravity | Sulfur (wt%) | V (ppm) | Ni (ppm) | V/ (V+Ni) |
|---|---|---|---|---|---|---|
| 7B Cavern Oil | 11/2/2022 | 32.8 | 1.38 | 23 | 6.1 | 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 |
|  | Average | 33.3 | 1.38 | 40 | 10 | 0.78 |
|  | St. Dev. | 0.4 | 0.02 | 41 | 10 | 0.01 |

**Locally Produced (Yellow Rock) Oils**

| Client ID | Date Collected | API gravity | Sulfur (wt%) | V (ppm) | Ni (ppm) | V/ (V+Ni) |
|---|---|---|---|---|---|---|
| 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 |
|  | Average | 24.0 | 0.39 | 1.4 | 7.3 | 0.16 |
|  | St. Dev. | 2.3 | 0.07 | 0.7 | 2.3 | 0.04 |

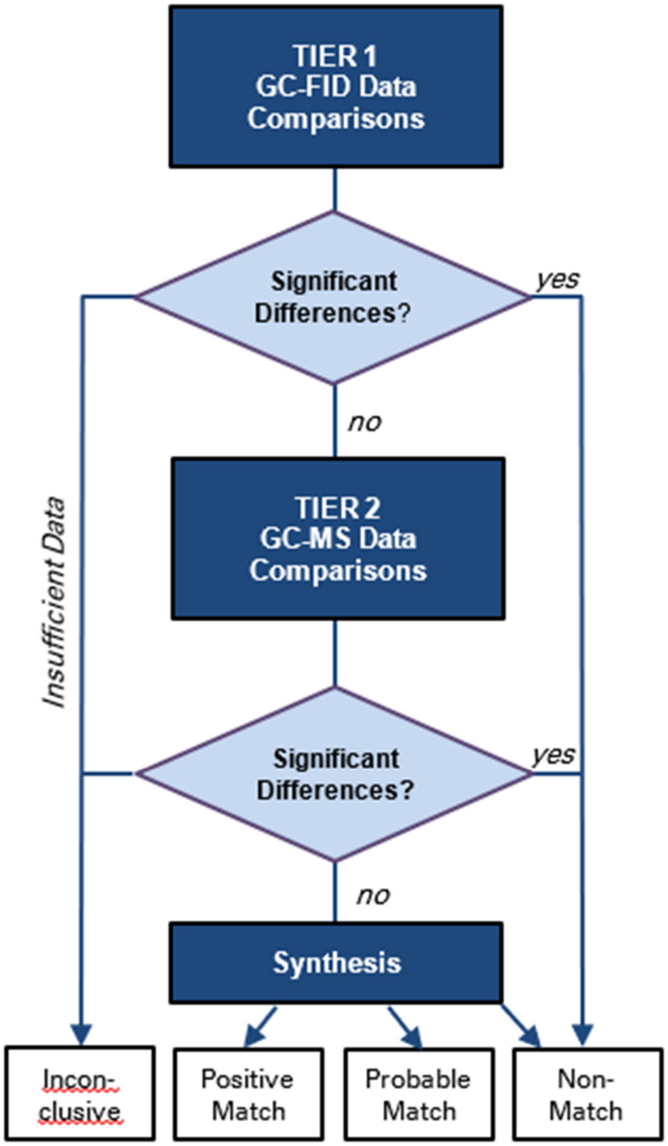


**Figure 1: Simplified flowchart depicting the CEN (2012) oil spill identification protocol.**

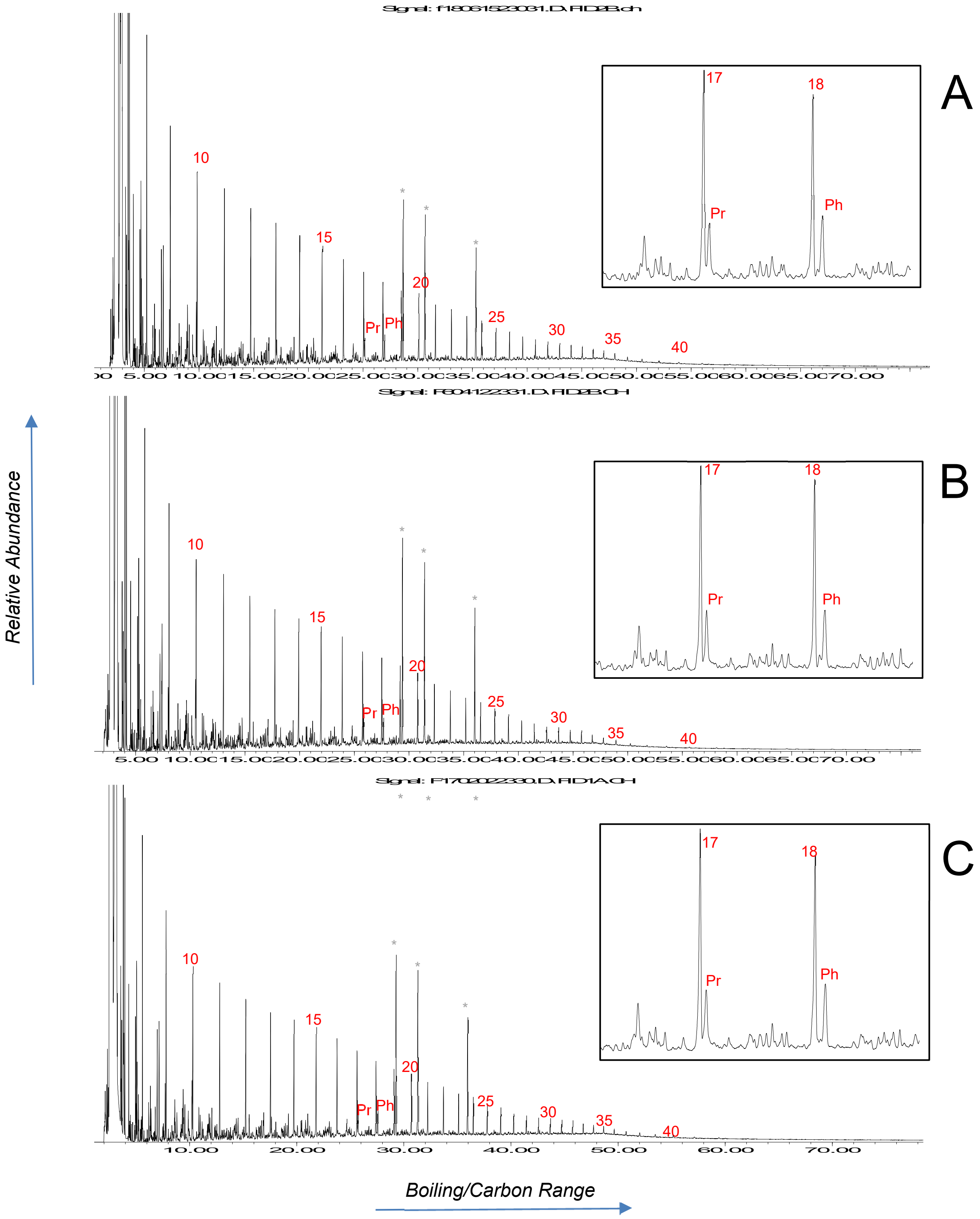


**Figure 2:** GC/FID (C8+) chromatograms for (A) 7B Cavern oil (May 25, 2023), (B) 7B Cavern oil (March 30, 2023), and (C) 7B Cavern oil (Jan. 21, 2023). Insets show further expanded view of C17-C18 range. #: n-alkane carbon number; Pr: pristane; Ph: phytane; UCM: unresolved complex mixture; *: internal standard.

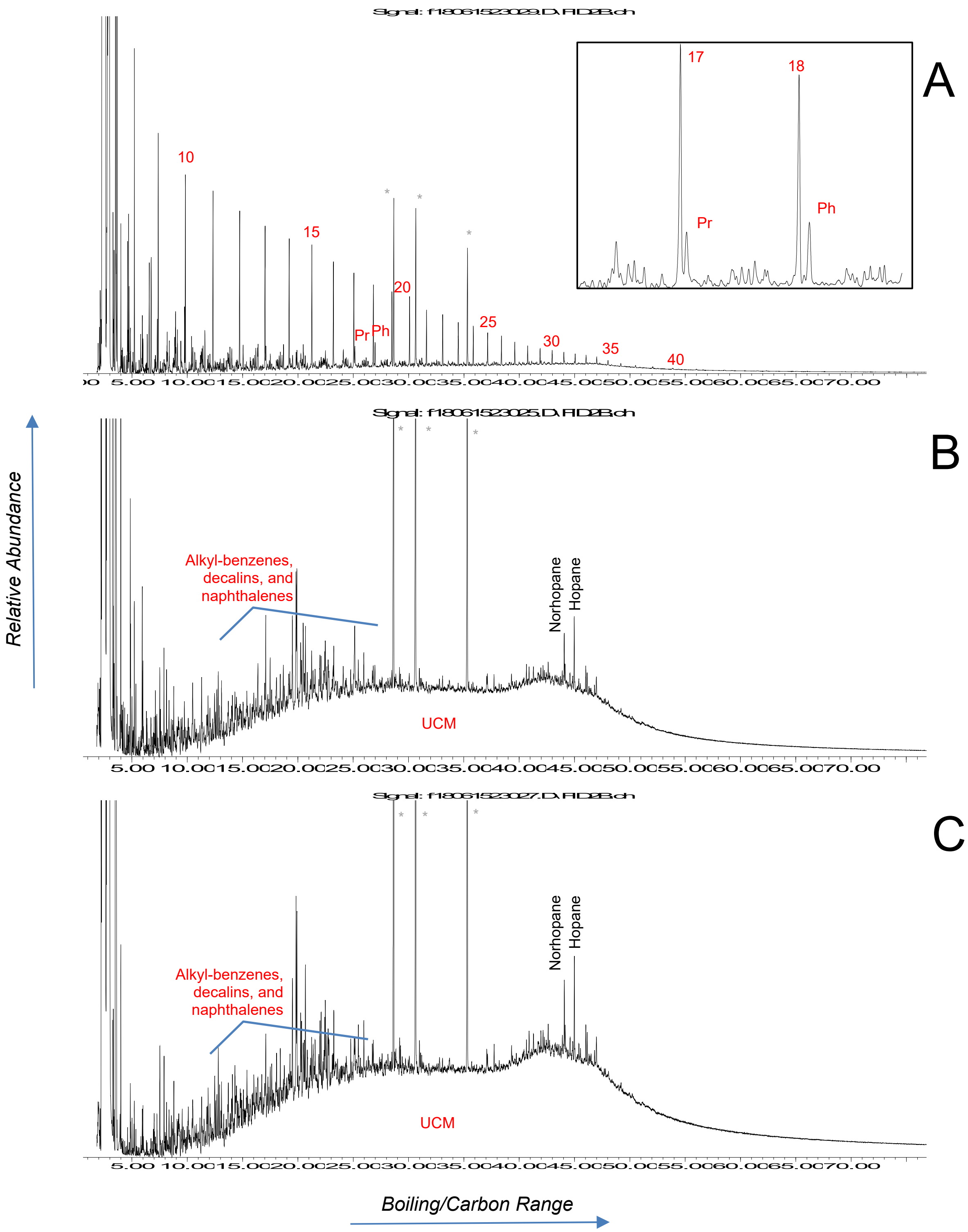


**Figure 3:** GC/FID (C8+) chromatograms for (A) Cavern 4 oil (May 25, 2023), (B) Yellow Rock 209459 oil (May 2, 2023), (C) Yellow Rock 185997 oil (May 2, 2023), (D) Yellow Rock 210185 oil (May 25, 2023), (E) Yellow Rock tank battery oil (May 25, 2023), and (F) No. 24 bubble site oil/sheen (May 22, 2023). Insets show further expanded view of C17-C18 range. #: n-alkane carbon number; Pr: pristane; Ph: phytane; UCM: unresolved complex mixture; *: internal standard.

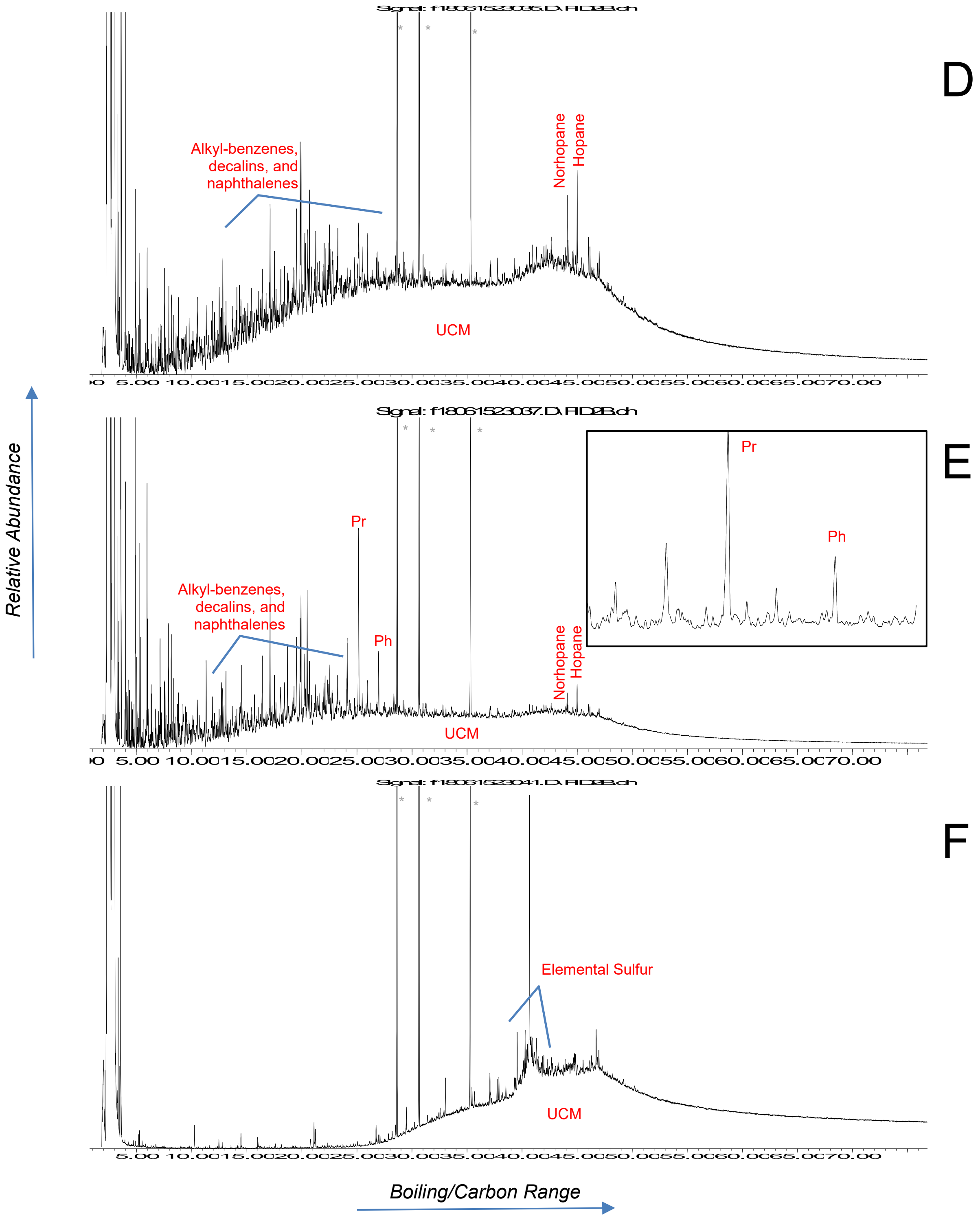


**Figure 3:** continued

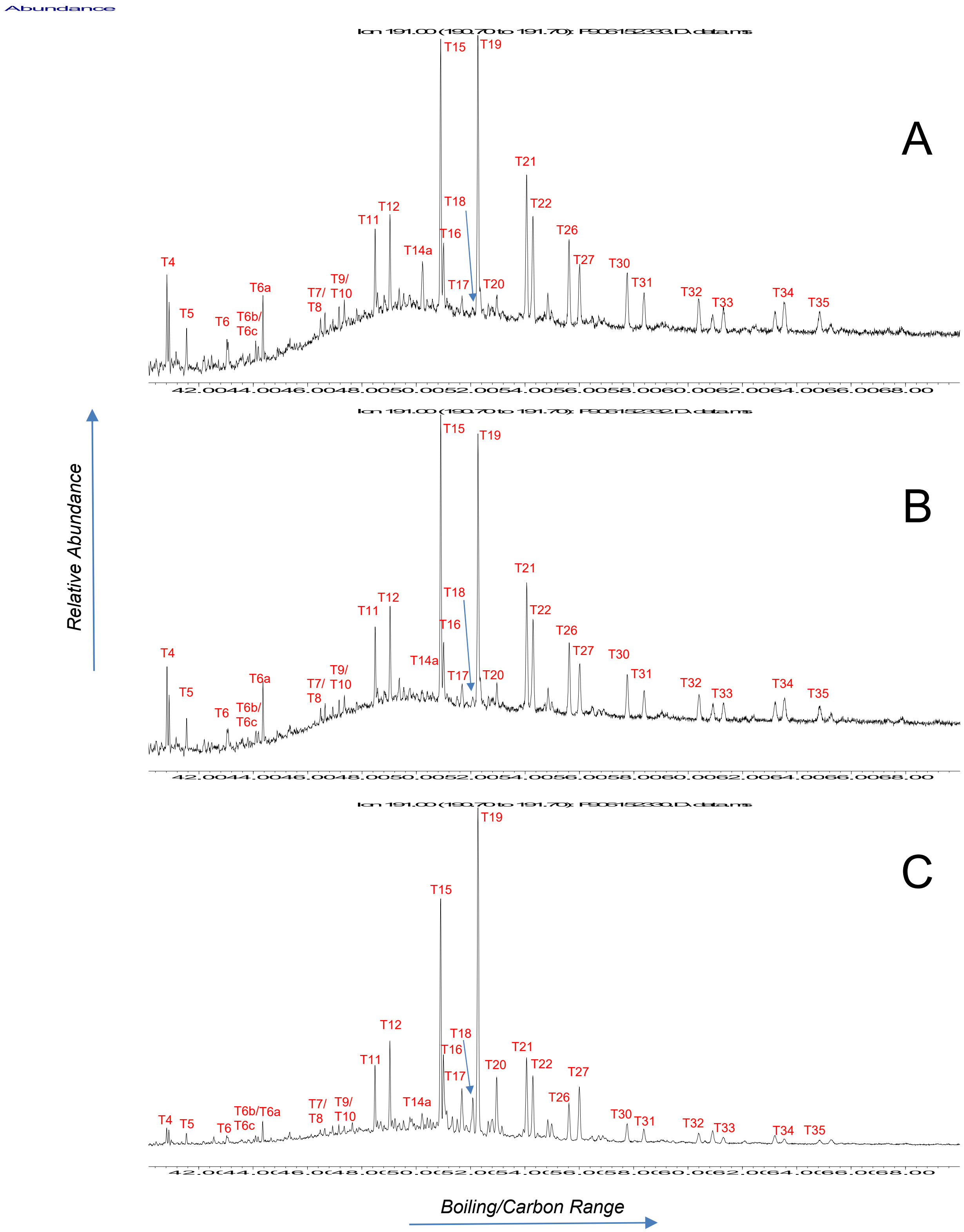


**Figure 4:** Partial extracted ion chromatograms (*m/z* 191) for selected samples studied. (A) 7B Cavern Oil (May 25, 2023), (B) Cavern 4 Oil (May 25, 2023), and (C) Yellow Rock Well 209459 (May 2, 2023). red labels: various triterpane biomarkers, see Attachment 4, Table 4-2 for compound names.

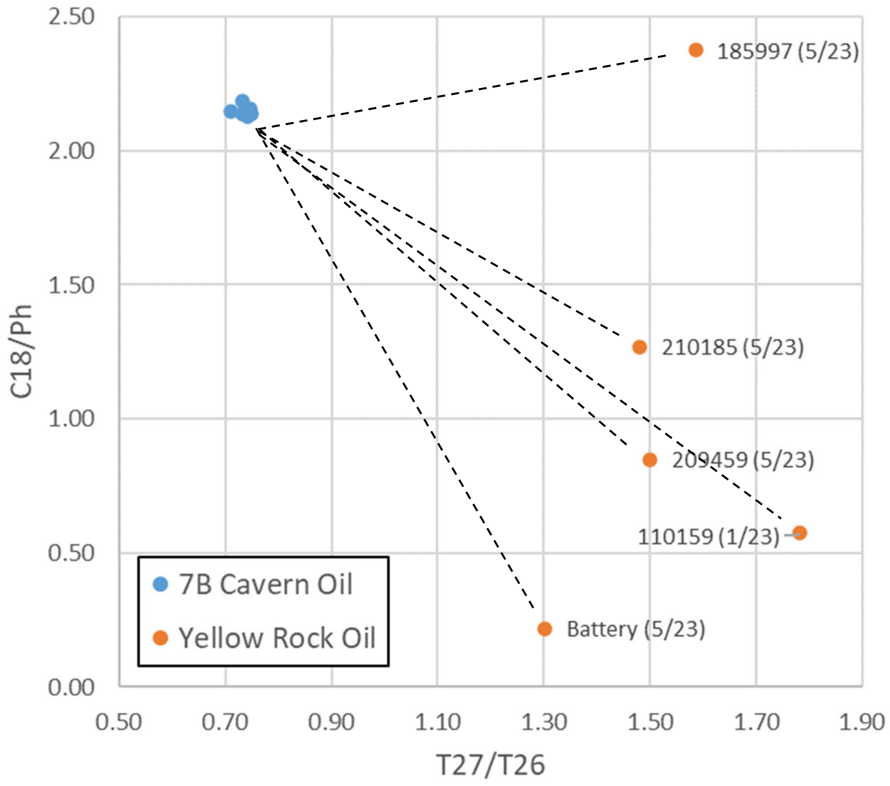


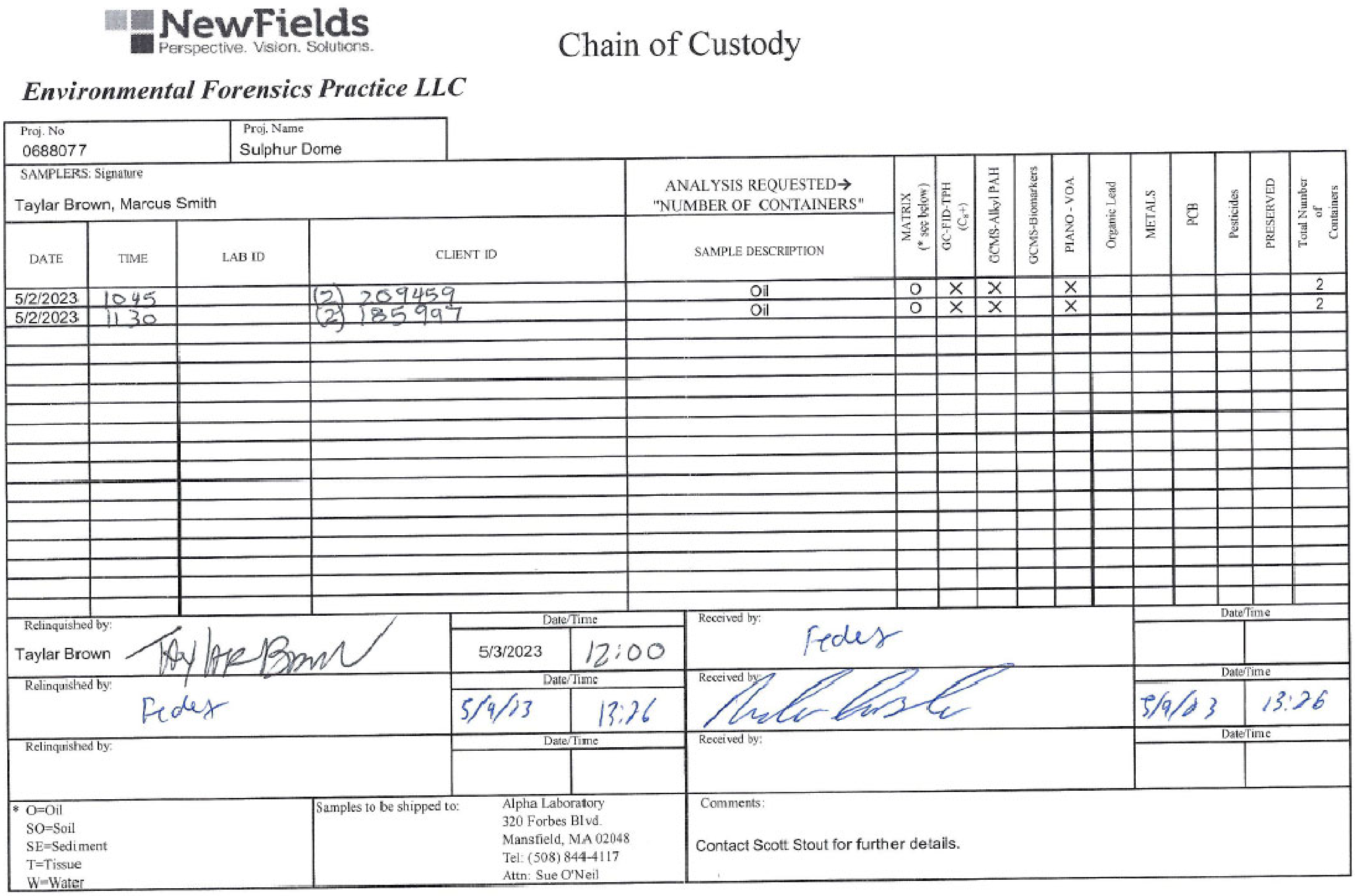
**Figure 5:** Double-ratio plot of two diagnostic ratios that preliminarily demonstrate the potential to distinguish the Yellow Rock oils collected from different wells/reservoir zones if they become mixed with 7B cavern oil.

Theoretically, mixing of these five Yellow Rock oils with the 7B cavern oil would plot along different mixing (dashed) lines indicated. (These mixing lines would need to re-calculated using absolute concentrations of the ratios' four analytes and may not necessarily appear linear thereafter, but rather as curves. Nonetheless, this demonstrates the concept and the potential utility of these, and perhaps other, DRs or metrics.)

# ATTACHMENTS

# Attachment 1

## Chain-of-Custody



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### Chain of Custody

Proj. No: 0688077
Proj. Name: Sulphur Dome
SAMPLERS: Signature: Taylar Brown, Marcus Smith

|  |  |  |  | ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS" |  |  |  |  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| DATE | TIME | LAB ID | CLIENT ID | SAMPLE DESCRIPTION | MATRIX (* see below) | GC-FID-TPH ($C_8$+) | GCMS-Alkyl PAH | GCMS-Biomarkers | PIANO - VOA | Organic Lead | METALS | PCB | Pesticides | PRESERVED | Total Number of Containers |
| 5/2/2023 | 1045 |  | (2) 209459 | Oil | O | X | X |  | X |  |  |  |  |  | 2 |
| 5/2/2023 | 1130 |  | (2) 185997 | Oil | O | X | X |  | X |  |  |  |  |  | 2 |

| Relinquished by: | Date/Time |  | Received by: | Date/Time |  |
|---|---|---|---|---|---|
| Taylar Brown | 5/3/2023 | 12:00 | Fedex |  |  |
| Fedex | 5/4/23 | 13:26 |  | 5/4/23 | 13:26 |
|  |  |  |  |  |  |

* 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.

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# Chain of Custody

**Environmental Forensics Practice LLC**

Proj. No: 0668077

Proj. Name: Sulphur Dome

SAMPLERS Signature: Scott Himes

| DATE | TIME | LAB ID | CLIENT ID | ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS" SAMPLE DESCRIPTION | MATRIX (* see below) | GC-FID-TPH ($C_9$+) | GCMS-Alkyl PAH | GCMS-Biomarkers | PIANO - VOA | Organic Lead | METALS | PCB | Pesticides | PRESERVED | Total Number of Containers |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5/25/2023 | 1015 | | Cavern 4 | Oil | O | X | X | | X | | | | | | 2 |
| 5/25/2023 | 1045 | | Cavern 7B | Oil | O | X | X | | X | | | | | | 2 |
| 5/25/2023 | 1110 | | 210185 | Oil | O | X | X | | X | | | | | | 2 |
| 5/25/2023 | 1245 | | Tank Battery | Oil | O | X | X | | X | | | | | | 2 |
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| | 6/1/2023 | 1600 | Fedex | | |
| Fedex | 6/2/23 | 10:09 | | 6/2/23 | 10:09 |
| | | | | | |

* 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:

Please contact Scott Stout for further details.

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# Chain of Custody

L2325505

Proj. No 0688077

Proj. Name Sulphur Dome

SAMPLERS: Signature Scott Hines

| DATE | TIME | LAB ID | CLIENT ID | ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS"<br>SAMPLE DESCRIPTION | MATRIX (* see below) | GC-FID-TPH ($C_9$+) | GCMS-Alkyl PAH | GCMS-Biomarkers | PIANO - VOA | Organic Lead | METALS | PCB | Pesticides | PRESERVED | Total Number of Containers |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5/25/23 | 12:45 | 25505-06 | Tank Battery | Oil | O | X | X |  | X |  |  |  |  |  | 2 |
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| Relinquished by: | Date/Time |  | Received by: | Date/Time |  |
|---|---|---|---|---|---|
| Taylor Brown Taylor Brown | 6/5/23 | 3:26pm | Fedex |  |  |
| Fedex |  |  |  | 6/6/23 | 10:15 |
|  |  |  |  |  |  |

* 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:

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**Environmental Forensics Practice LLC**

# Chain of Custody

Proj. No 0688077

Proj. Name Sulphur Dome

SAMPLERS: Signature Scott Himes

| | | | | ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS" | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| DATE | TIME | LAB ID | CLIENT ID | SAMPLE DESCRIPTION | MATRIX (* see below) | GC-FID-TPH ($C_8$+) | GCMS-Alkyl PAH | GCMS-Biomarkers | PIANO - VOA | Organic Lead | METALS | PCB | Pesticides | PRESERVED | Total Number of Containers |
| 5/22/23 | 1115 | | BS-24 | Sheen (bubble site 24) | O | X | X | X | X | | | | | | 1 |
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| Relinquished by: | Date/Time | | Received by: | Date/Time | |
|---|---|---|---|---|---|
| | 5/23/2023 | 1600 | Fedex | | |
| Fedex | 5/25/23 | 10:04 | | 5/25/23 | 10:04 |
| | | | | | |

* 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:

# 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; 5$\alpha$-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 5$\beta$(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 $\mu$L) 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 oil and net 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

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[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.

$\mu$m film) fused silica capillary column. Extracts were injected (1 $\mu$L, pulsed splitless) into the GC programmed from 40°C (1 min) and ramped at 6°C/min to 315°C (30 min) using $H_2$ (~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/$\mu$L to 200 ng/$\mu$L. 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 $\mu$L, pulsed splitless) into the GC containing a 60m x 0.25 mm ID, 0.25 $\mu$m film, Phenomenex ZB-5 capillary column and the oven programmed from 35$^o$C (1 min) and ramped at 6$^o$C/min to 315$^o$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/$\mu$L for PAH and 0.01 to 20.0 ng/$\mu$L 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 17$\alpha$(H),21$\beta$(H)-hopane, and steranes and triaromatic steroids were quantified using the RF of 5$\beta$(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.

### On-Going Assessment of Diagnostic Ratio Precision

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

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[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 four sets of duplicate oils in four different batches of oil samples and one site-specific reference oil (7B cavern oil collected on January 25, 2023) on two occasions spanning approximately 4.5 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 three 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% criterion to $RSD_R$ is appropriate since only those DRs that are precisely measured over the long-

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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.

term ($\text{RSD}_R$<5%) are equally reliable.[11] Inspection of Table A3 shows that just over half (18 of 30) of the DRs employed in this program have $\text{RSD}_R$ below 5%.

Considering both $\text{RSD}_r$ and $\text{RSD}_R$ 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: $\text{RSD}_r$ and $\text{RSD}_R$ calculated for the 29 diagnostic ratios used in the Sulphur Dome monitoring studies.**

| | | Sulphur Dome Site Precision | | |
|---|---|---|---|---|
| | | Repeatability | Reproducability | Most Precise Ratios* |
| CEN - Diagnostic Ratios | CEN Diagnostic Ratios per Alpha Abbreviations | $\text{RSD}_r$ | $\text{RSD}_R$ | |
| NR-C17/pris | C17/Pr | 1.6 | 7.1 | |
| NR-C18/phy | C18/Ph | 0.5 | 1.0 | x |
| NR- pris/phy | Pr/Ph | 2.0 | 7.1 | |
| NR-4-MD/1-MD | 4-MDBT/1-MDBT | 3.7 | 0.2 | x |
| NR-2-MP/1-MP | 2-MP/1-MP | 3.4 | 2.2 | x |
| NR-27Ts/30ab | T11/T19 | 1.7 | 0.6 | x |
| NR-27Tm/30ab | T12/T19 | 1.5 | 1.5 | x |
| NR-28ab/30ab | T14a/T19 | 2.9 | 4.8 | x |
| NR-29ab/30ab | T15/T19 | 2.3 | 0.2 | x |
| NR-30O/30ab | T18/T19 | 7.9 | 17.0 | |
| NR-31abS/30ab | T21/T19 | 1.1 | 1.3 | x |
| NR-27dbR/27dbS | S4/S5 | 4.6 | 13.4 | |
| NR-27bb/29bb | (S14+S15)/(S26+S27) | 1.7 | 1.9 | x |
| NR-SC26/ RC26+SC27 | TAS09/TAS01 | 3.1 | 4.5 | x |
| NR-SC28/RC26 + SC27 | TAS02/TAS01 | 1.9 | 0.4 | x |
| NR-RC27/RC26+ SC27 | TAS03/TAS01 | 1.8 | 0.6 | x |
| NR-RC28/RC26+SC27 | TAS04/TAS01 | 3.3 | 0.7 | x |
| DR-Ts/Tm | T11/T12 | 2.2 | 2.1 | x |
| DR-29Ts30ab | T16/T19 | 3.4 | 0.7 | x |
| DR-29bb/29aa | (S26+S27)/(S25+S28) | 4.5 | 15.5 | |
| DR-C2-dbt/C2-phe | DBT2/PA2 | 1.0 | 6.5 | |
| DR-C3-dbt/C3-phe | DBT3/PA3 | 0.6 | 8.0 | |
| DR-C28C29/30ab | T7 to T10/T19 | 4.7 | 11.5 | |
| DR-29aaS/29aaR | S25/S28 | 8.1 | 15.8 | |
| DR-C20TA/C21TA | TAS05/TAS06 | 4.9 | 12.7 | |
| DR-TA21/ RC26+SC27 | TS06/TAS01 | 0.7 | 11.4 | |
| DR-C24Tet/C26Tri | T6a/T19 | 4.6 | 4.9 | x |
| DR-30ba/30ab | T20/T19 | 4.3 | 7.0 | |
| DR-35ab/30ab | (T34 to T35)/T19 | 5.6 | 1.3 | |
| DR-32abR/32abS | T27/T26 | 1.6 | 1.2 | x |

*both $\text{RSD}_r$ and $\text{RSD}_R$ < 5% based on current QC datasets

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[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 | **209459** | **185997** | **CAVERN 4** | **CAVERN 7B** | **CAVERN 7B (Dup)** | **210185** | **TANK BATTERY** | **7B** | **BS-24** |
|---|---|---|---|---|---|---|---|---|---|
| Lab ID | L2325505-01 | L2325505-02 | L2325505-03 | L2325505-04 | WG1791156-4 | L2325505-05 | L2325505-06 | L2325505-07 | L2325505-08 |
| Date Collected | 5/2/2023 | 5/2/2023 | 5/25/2023 | 5/25/2023 | NA | 5/25/2023 | 5/25/2023 | 1/25/2023 | 5/22/2023 |
| Date Analyzed | 6/16/2023 | 6/16/2023 | 6/16/2023 | 6/16/2023 | 6/16/2023 | 6/16/2023 | 6/16/2023 | 6/16/2023 | 6/16/2023 |
| Analytes | Result | Result | Result | Result | Result | Result | Result | Result | Result |
| n-Nonane (C9) | 159 | nd | 9,760 | 9,790 | 10,100 | 119 | 163 | 10,100 | 21 |
| n-Decane (C10) | 251 | 127 | 8,860 | 8,810 | 9,100 | 217 | 299 | 9,180 | 34 |
| n-Undecane (C11) | 392 | 144 | 8,510 | 8,410 | 8,680 | 324 | 570 | 8,720 | 20 |
| n-Dodecane (C12) | 252 | 118 | 7,920 | 7,800 | 8,100 | 215 | 313 | 8,070 | 26 |
| n-Tridecane (C13) | 538 | 375 | 7,470 | 7,340 | 7,530 | 463 | 413 | 7,390 | 32 |
| 2,6,10 Trimethyldodecane (1380) | 596 | 196 | 1,160 | 1,420 | 1,470 | 344 | 1,580 | 1,390 | nd |
| n-Tetradecane (C14) | 373 | 214 | 6,700 | 6,600 | 6,880 | 374 | 477 | 6,880 | 36 |
| 2,6,10 Trimethyltridecane (1470) | 1,290 | 648 | 1,900 | 1,970 | 2,030 | 954 | 3,340 | 2,040 | nd |
| n-Pentadecane (C15) | 765 | 576 | 6,180 | 6,120 | 6,330 | 660 | 896 | 6,660 | 324 |
| n-Hexadecane (C16) | 456 | 366 | 5,550 | 5,330 | 5,490 | 389 | 512 | 5,440 | 53 |
| Norpristane (1650) | 583 | 178 | 1,220 | 1,290 | 1,320 | 377 | 2,260 | 1,290 | nd |
| n-Heptadecane (C17) | 236 | 149 | 4,760 | 4,760 | 4,880 | 234 | 251 | 4,720 | 81 |
| Pristane | 1,320 | 400 | 1,560 | 1,780 | 1,840 | 814 | 5,030 | 1,820 | nd |
| n-Octadecane (C18) | 297 | 335 | 3,970 | 4,280 | 4,300 | 325 | 353 | 4,240 | 108 |
| Phytane | 350 | 141 | 1,770 | 2,010 | 2,010 | 256 | 1,620 | 1,970 | 162 |
| n-Nonadecane (C19) | 243 | 219 | 3,650 | 3,610 | 3,690 | 260 | 290 | 3,630 | nd |
| n-Eicosane (C20) | 272 | 229 | 3,580 | 3,510 | 3,610 | 289 | 150 | 3,510 | 71 |
| n-Heneicosane (C21) | 80 | nd | 2,740 | 2,700 | 2,760 | 133 | 89 | 2,730 | nd |
| n-Docosane (C22) | 163 | 138 | 2,380 | 2,370 | 2,430 | 177 | 176 | 2,360 | 559 |
| n-Tricosane (C23) | 76 | 37 | 2,080 | 2,090 | 2,140 | 88 | 72 | 2,080 | 78 |
| n-Tetracosane (C24) | 87 | 55 | 1,900 | 1,950 | 1,990 | 96 | 128 | 1,930 | 56 |
| n-Pentacosane (C25) | 196 | 170 | 1,660 | 1,690 | 1,720 | 227 | 334 | 1,680 | 192 |
| n-Hexacosane (C26) | 76 | nd | 1,470 | 1,460 | 1,500 | 109 | 152 | 1,450 | 84 |
| n-Heptacosane (C27) | 71 | nd | 1,140 | 1,230 | 1,220 | 135 | 93 | 1,170 | nd |
| n-Octacosane (C28) | 67 | nd | 877 | 971 | 1,020 | 73 | 64 | 966 | nd |
| n-Nonacosane (C29) | 122 | nd | 809 | 892 | 914 | 135 | 87 | 928 | 345 |
| n-Triacontane (C30) | nd | nd | 737 | 866 | 864 | nd | nd | 850 | nd |
| n-Hentriacontane (C31) | 108 | 117 | 621 | 724 | 728 | 132 | 103 | 742 | 208 |
| n-Dotriacontane (C32) | 61 | 60 | 535 | 615 | 636 | 75 | 58 | 661 | 119 |
| n-Tritriacontane (C33) | 299 | 327 | 518 | 558 | 579 | 323 | 256 | 620 | nd |
| n-Tetratriacontane (C34) | 279 | 334 | 448 | 574 | 540 | 270 | 280 | 601 | 396 |
| n-Pentatriacontane (C35) | nd | nd | 374 | 514 | 556 | nd | nd | 609 | nd |
| n-Hexatriacontane (C36) | 73 | 76 | 230 | 294 | 311 | 88 | 66 | 326 | 145 |
| n-Heptatriacontane (C37) | nd | nd | 218 | 270 | 270 | nd | nd | 318 | nd |
| n-Octatriacontane (C38) | nd | nd | 167 | 238 | 257 | nd | nd | 320 | nd |
| n-Nonatriacontane (C39) | nd | nd | 129 | 192 | 198 | nd | nd | 266 | nd |
| n-Tetracontane (C40) | nd | nd | 104 | 160 | 168 | nd | nd | 228 | nd |
| Total Saturated Hydrocarbons | 10,100 | 5,730 | 104,000 | 105,000 | 108,000 | 8,670 | 20,500 | 108,000 | 3,150 |
| Total Petroleum Hydrocarbons (C9-C44) | 634,000 | 714,000 | 636,000 | 612,000 | 632,000 | 682,000 | 711,000 | 637,000 | 636,000 |

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

| | Client ID | 209459 | 185997 | CAVERN 4 | CAVERN 7B | CAVERN 7B (Dup) | 210185 | TANK BATTERY | 7B | BS-24 |
|---|---|---|---|---|---|---|---|---|---|---|
| | Lab ID | L2325505-01 | L2325505-02 | L2325505-03 | L2325505-04 | WG1791156-4 | L2325505-05 | L2325505-06 | L2325505-07 | L2325505-08 |
| | Date Collected | 5/2/2023 | 5/2/2023 | 5/25/2023 | 5/25/2023 | NA | 5/25/2023 | 5/25/2023 | 1/25/2023 | 5/22/2023 |
| | Date Analyzed | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/15/2023 |
| | Analytes | Result | Result | Result | Result | Result | Result | Result | Result | Result |
| D0 | cis/trans-Decalin | 474 | 276 | 209 | 228 | 235 | 283 | 1,060 | 232 | nd |
| D1 | C1-Decalins | 743 | 540 | 392 | 405 | 421 | 490 | 1,450 | 421 | nd |
| D2 | C2-Decalins | 994 | 1,100 | 363 | 360 | 377 | 968 | 1,240 | 372 | nd |
| D3 | C3-Decalins | 753 | 837 | 242 | 236 | 244 | 780 | 769 | 243 | nd |
| D4 | C4-Decalins | 948 | 1,090 | 235 | 241 | 232 | 954 | 1,100 | 235 | nd |
| BT0 | Benzothiophene | 4.05 | 4.42 | 8.86 | 7.96 | 8.15 | 3.62 | 6.46 | 8.36 | nd |
| BT1 | C1-Benzo(b)thiophenes | 23.2 | 28.1 | 44.1 | 40.4 | 42.1 | 22.8 | 30.9 | 43.3 | nd |
| BT2 | C2-Benzo(b)thiophenes | 23.8 | 27.1 | 179 | 153 | 160 | 21.8 | 31.0 | 164 | nd |
| BT3 | C3-Benzo(b)thiophenes | 45.7 | 50.5 | 317 | 268 | 279 | 45.9 | 51.2 | 283 | nd |
| BT4 | C4-Benzo(b)thiophenes | 34.6 | 38.8 | 259 | 222 | 229 | 34.6 | 32.2 | 232 | nd |
| N0 | Naphthalene | 67.8 | 18.6 | 186 | 202 | 209 | 74.1 | 696 | 214 | 3.24 |
| N1 | C1-Naphthalenes | 543 | 222 | 680 | 709 | 734 | 424 | 1,290 | 757 | 5.25 |
| N2 | C2-Naphthalenes | 1,470 | 1,540 | 1,110 | 1,100 | 1,140 | 1,160 | 2,390 | 1,170 | 13 |
| N3 | C3-Naphthalenes | 1,200 | 1,390 | 960 | 942 | 979 | 1,060 | 1,840 | 992 | 8.28 |
| N4 | C4-Naphthalenes | 739 | 852 | 555 | 545 | 565 | 728 | 981 | 571 | nd |
| B | Biphenyl | 13 | 3.37 | 33.9 | 35.6 | 36.9 | 16.1 | 6.0 | 38.7 | 7.57 |
| DF | Dibenzofuran | 30 | 7.09 | 21.6 | 27.8 | 28.6 | 15.8 | 61.5 | 29.6 | 39 |
| AY | Acenaphthylene | 6.4 | 7.46 | 3.96 | 4.10 | 4.33 | 5.58 | 11.3 | 4.08 | nd |
| AE | Acenaphthene | 11.8 | 11.7 | 8.87 | 8.57 | 9.57 | 12.7 | 11.4 | 9.58 | 4.20 |
| F0 | Fluorene | 63.4 | 75.1 | 50.1 | 56.0 | 58.6 | 51.2 | 83.2 | 59.0 | 9.10 |
| F1 | C1-Fluorenes | 212.0 | 260.0 | 153.0 | 153.0 | 161.0 | 193.0 | 274.0 | 162.0 | 4.12 |
| F2 | C2-Fluorenes | 348 | 401 | 270 | 253 | 265 | 339 | 432 | 266 | nd |
| F3 | C3-Fluorenes | 301 | 336 | 296 | 267 | 280 | 306 | 343 | 279 | nd |
| A0 | Anthracene | 6.59 | 8.98 | 7.48 | 9.06 | 8.10 | 7.28 | 8.16 | 8.52 | 2.94 |
| P0 | Phenanthrene | 148 | 183 | 112 | 117 | 121 | 139 | 181 | 125 | 52 |
| PA1 | C1-Phenanthrenes/Anthracenes | 393 | 472 | 344 | 338 | 352 | 382 | 463 | 358 | 11 |
| PA2 | C2-Phenanthrenes/Anthracenes | 451 | 531 | 420 | 404 | 417 | 447 | 514 | 421 | nd |
| PA3 | C3-Phenanthrenes/Anthracenes | 301 | 351 | 318 | 297 | 307 | 311 | 320 | 307 | nd |
| PA4 | C4-Phenanthrenes/Anthracenes | 142 | 163 | 167 | 148 | 156 | 148 | 143 | 155 | nd |
| RET | Retene | 65 | 76 | nd | nd | nd | 71.8 | 64.0 | nd | nd |
| DBT0 | Dibenzothiophene | 30 | 33 | 236 | 233 | 238 | 25.7 | 31.0 | 244 | 3.85 |
| DBT1 | C1-Dibenzothiophenes | 111 | 127 | 598 | 551 | 571 | 106 | 116 | 585 | 2.76 |
| DBT2 | C2-Dibenzothiophenes | 126 | 140 | 951 | 835 | 858 | 123 | 116 | 878 | nd |
| DBT3 | C3-Dibenzothiophenes | 113 | 123 | 804 | 688 | 714 | 114 | 94.2 | 720 | nd |
| DBT4 | C4-Dibenzothiophenes | 51.6 | 56.8 | 425 | 365 | 375 | 54.0 | 44.3 | 382 | nd |
| BF | Benzo(b)fluorene | 5.35 | 6.27 | 4.20 | nd | nd | 4.97 | 6.28 | nd | nd |
| FL0 | Fluoranthene | 4.27 | 5.06 | 1.19 | 1.73 | 1.71 | 4.56 | 4.45 | 1.57 | 6.87 |
| PY0 | Pyrene | 8.32 | 9.65 | 13.5 | 12.9 | 13.3 | 11.70 | 8.8 | 12.6 | 4.71 |
| FP1 | C1-Fluoranthenes/Pyrenes | 54.6 | 64.0 | 49.5 | 49.4 | 51.2 | 56.3 | 58.6 | 51.9 | nd |
| FP2 | C2-Fluoranthenes/Pyrenes | 82.4 | 89.3 | 94.8 | 92.8 | 94.1 | 86.1 | 82.0 | 96.9 | nd |
| FP3 | C3-Fluoranthenes/Pyrenes | 103 | 114 | 137 | 126 | 132 | 110 | 104 | 132 | 36.2 |
| FP4 | C4-Fluoranthenes/Pyrenes | 96.3 | 102 | 136 | 122 | 129 | 97.1 | 94.0 | 125 | 53.6 |
| NBT0 | Naphthobenzothiophenes | 8.67 | 10.2 | 55.8 | 55.1 | 57.7 | 10.2 | 9.35 | 58.7 | nd |
| NBT1 | C1-Naphthobenzothiophenes | 30.3 | 33.6 | 205 | 185 | 192 | 31.5 | 27.5 | 195 | nd |
| NBT2 | C2-Naphthobenzothiophenes | 40.4 | 44.9 | 325 | 278 | 290 | 44.1 | 36.6 | 294 | nd |
| NBT3 | C3-Naphthobenzothiophenes | 30.7 | 32.9 | 284 | 238 | 247 | 32.5 | 27.2 | 249 | 57.6 |
| NBT4 | C4-Naphthobenzothiophenes | 31.4 | 33.3 | 201 | 167 | 171 | 33.8 | 28.7 | 175 | 37.6 |
| BA0 | Benz[a]anthracene | 2.07 | 2.67 | 1.23 | 1.22 | 1.56 | 2.40 | 2.21 | 1.43 | 0.93 |
| C0 | Chrysene/Triphenylene | 13.2 | 15.2 | 17.3 | 16.8 | 17.5 | 14.5 | 13.1 | 18.3 | 1.62 |
| BC1 | C1-Chrysenes | 33.6 | 39.4 | 43.2 | 39.2 | 41.3 | 36.2 | 34.9 | 40.8 | nd |
| BC2 | C2-Chrysenes | 51.8 | 59.7 | 73.1 | 65.8 | 70.2 | 53.6 | 53.2 | 68.5 | 22.0 |
| BC3 | C3-Chrysenes | 60.4 | 72.4 | 106.0 | 95.2 | 98.2 | 65.1 | 59.3 | 89.4 | 72.6 |
| BC4 | C4-Chrysenes | 43.7 | 50.6 | 81.0 | 78.4 | 84.9 | 47.2 | 43.5 | 75.2 | nd |

# Table 4-2: continued

| | Client ID | 209459 | 185997 | CAVERN 4 | CAVERN 7B | CAVERN 7B (Dup) | 210185 | TANK BATTERY | 7B | BS-24 |
|---|---|---|---|---|---|---|---|---|---|---|
| | Lab ID | L2325505-01 | L2325505-02 | L2325505-03 | L2325505-04 | WG1791156-4 | L2325505-05 | L2325505-06 | L2325505-07 | L2325505-08 |
| | Date Collected | 5/2/2023 | 5/2/2023 | 5/25/2023 | 5/25/2023 | NA | 5/25/2023 | 5/25/2023 | 1/25/2023 | 5/22/2023 |
| | Date Analyzed | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/15/2023 |
| | Analytes | Result | Result | Result | Result | Result | Result | Result | Result | Result |
| BBF | Benzo[b]fluoranthene | 1.72 | 2.12 | 2.11 | 3.14 | 2.92 | 2.08 | 1.97 | 2.64 | 1.44 |
| BJKF | Benzo[j]fluoranthene/Benzo[k]fluoranthene | nd | nd | nd | nd | nd | nd | nd | nd | 0.79 |
| BAF | Benzo[a]fluoranthene | nd | nd | nd | nd | nd | nd | nd | nd | nd |
| BEP | Benzo[e]pyrene | 2.78 | 3.04 | 7.86 | 7.29 | 8.23 | 3.06 | 2.18 | 7.71 | 1.62 |
| BAP | Benzo[a]pyrene | 0.97 | 1.06 | 1.36 | 1.44 | 1.31 | 0.86 | 0.69 | 1.46 | nd |
| PER | Perylene | 7.37 | 9.23 | 1.34 | 1.23 | 1.38 | 8.32 | 10.20 | 1.43 | 3.74 |
| IND | Indeno[1,2,3-cd]pyrene | nd | nd | nd | nd | nd | nd | nd | nd | nd |
| DA | Dibenz[ah]anthracene/Dibenz[ac]anthrace | nd | nd | nd | nd | nd | nd | nd | nd | nd |
| GHI | Benzo[g,h,i]perylene | 1.03 | 0.88 | 1.82 | 2.13 | 2.51 | 1.07 | 0.97 | 2.25 | 2.12 |
| CAR | Carbazole | 3.15 | 4.80 | 8.80 | 8.59 | 8.42 | 4.07 | 3.67 | 9.06 | 0.83 |
| 4MDT | 4-Methyldibenzothiophene | 41.0 | 48.6 | 256 | 234 | 243 | 37.9 | 42.6 | 248 | 0.90 |
| 2MDT | 2/3-Methyldibenzothiophene | 55.2 | 60.9 | 223 | 205 | 212 | 52.7 | 58.8 | 218 | nd |
| 1MDT | 1-Methyldibenzothiophene | 11.5 | 12.4 | 116 | 109 | 113 | 11.2 | 9.42 | 116 | nd |
| 3MP | 3-Methylphenanthrene | 90.0 | 103 | 64.4 | 63.1 | 64.6 | 82.2 | 117 | 67.1 | 1.59 |
| 2MP | 2-Methylphenanthrene | 90.0 | 106 | 83.2 | 77.8 | 81.0 | 85.7 | 113 | 82.4 | 2.40 |
| 2MA | 2-Methylanthracene | 4.22 | 7.60 | 3.72 | 3.20 | 3.48 | 4.11 | 4.77 | 3.54 | nd |
| 9MP | 9/4-Methylphenanthrene | 125 | 149 | 120 | 118 | 123 | 125 | 135 | 124 | 1.56 |
| 1MP | 1-Methylphenanthrene | 77.1 | 97.6 | 69.8 | 72.9 | 76.8 | 73.1 | 87.7 | 80.3 | 1.36 |
| 2MN | 2-Methylnaphthalene | 535 | 212 | 580 | 595 | 617 | 411 | 1,470 | 637 | 6 |
| 1MN | 1-Methylnaphthalene | 345 | 143 | 529 | 562 | 582 | 273 | 602 | 598 | 3 |
| 26DMN | 2,6-Dimethylnaphthalene | 902 | 928 | 577 | 545 | 565 | 694 | 1,620 | 581 | 7 |
| 235TMN | 2,3,5-Trimethylnaphthalene | 178 | 208 | 146 | 170 | 148 | 187 | 273 | 131 | 1 |
| PY2 | 2-METHYLPYRENE | 4.31 | 4.59 | 3.30 | 3.16 | 3.23 | 4.51 | 3.84 | 3.41 | nd |
| PY4 | 4-METHYLPYRENE | 7.54 | 8.41 | 13.8 | 12.5 | 13.0 | 8.07 | 7.12 | 13.3 | nd |
| PY1 | 1-METHYLPYRENE | 4.80 | 5.04 | 7.95 | 7.28 | 7.50 | 5.07 | 4.24 | 7.83 | nd |
| T4 | C23 Tricyclic Terpane | 22.2 | 26.6 | 30.2 | 28.1 | 28.8 | 22.6 | 17.2 | 26.4 | 46.2 |
| T5 | C24 Tricyclic Terpane | 18.7 | 19.8 | 11.8 | 13.7 | 14.9 | 20.7 | 15.7 | 14.8 | 32.6 |
| T6 | C25 Tricyclic Terpane | 23.1 | 23.1 | 15.7 | 15.6 | 17.9 | 25.9 | 18.6 | 15.5 | 44.5 |
| T6a | C24 Tetracyclic Terpane | 31.8 | 33.2 | 22.8 | 17.8 | 18.5 | 32.6 | 27.9 | 19.2 | 69.2 |
| T6b | C26 Tricyclic Terpane-22S | 11.1 | 11.7 | 6.34 | 6.10 | 7.13 | 12.1 | 10.0 | 7.35 | 17.1 |
| T6c | C26 Tricyclic Terpane-22R | 8.25 | 7.79 | 6.04 | 4.75 | 5.39 | 8.58 | 7.78 | 4.30 | 20.4 |
| T7 | C28 Tricyclic Terpane-22S | 7.68 | 8.67 | 6.74 | 5.99 | 6.16 | 7.11 | 7.56 | 7.18 | 16.2 |
| T8 | C28 Tricyclic Terpane-22R | 14.2 | 15.7 | 7.52 | 7.36 | 8.30 | 15.5 | 10.9 | 6.73 | 19.2 |
| T9 | C29 Tricyclic Terpane-22S | 15.5 | 16.7 | 7.03 | 8.54 | 8.72 | 16.8 | 10.5 | 8.11 | 28.9 |
| T10 | C29 Tricyclic Terpane-22R | 12.2 | 14.6 | 6.58 | 9.44 | 8.11 | 13.7 | 9.5 | 6.54 | 25.7 |
| T11 | 18a-22,29,30-Trisnorneohopane-TS | 118 | 131 | 32.8 | 30.6 | 32.8 | 118 | 101 | 30.9 | 34.1 |
| T11a | C30 Tricyclic Terpane-22S | 20.7 | 21.0 | 8.41 | 9.24 | 9.64 | 20.3 | 18.1 | 9.51 | 43.1 |
| T11b | C30 Tricyclic Terpane-22R | 10.0 | 9.94 | 6.68 | 6.89 | 6.65 | 10.2 | 8.14 | 7.36 | 21.6 |
| T12 | 17a(H)-22,29,30-Trisnorhopane-TM | 154 | 167 | 41.4 | 34.0 | 35.0 | 162 | 130 | 35.7 | 48.8 |
| T14a | 17a/b,21b/a 28,30-Bisnorhopane | 39.3 | 41.0 | 7.08 | 23.8 | 25.1 | 42.1 | 26.4 | 24.2 | nd |
| T14b | 17a(H),21b(H)-25-Norhopane | 10.8 | 13.9 | 3.99 | nd | 2.50 | 13.3 | 7.62 | 3.83 | 25.8 |
| T15 | 30-Norhopane | 472 | 519 | 133 | 107 | 109 | 473 | 376 | 107 | 88.9 |
| T16 | 18a(H)-30-Norneohopane-C29Ts | 189 | 208 | 28.2 | 26.5 | 25.7 | 191 | 143 | 27.1 | 37.4 |
| X | 17a(H)-Diahopane | 35.1 | 39.5 | 4.46 | 3.94 | 4.87 | 34.4 | 34.5 | 3.70 | 88.6 |
| T17 | 30-Normoretane | 122 | 133 | 15.7 | 8.5 | 11.5 | 129 | 97.1 | 10.8 | 83.7 |
| T18 | 18a(H)&18b(H)-Oleananes | 79.0 | 89.4 | 7.30 | nd | nd | 80.9 | 64.6 | 6.85 | 210 |
| T19 | Hopane | 778 | 860 | 143 | 128 | 133 | 776 | 645 | 132 | 68.9 |
| T20 | Moretane | 150 | 157 | 12.8 | 9.72 | 8.86 | 165 | 122 | 8.41 | nd |
| T21 | 30-Homohopane-22S | 199 | 216 | 79.8 | 73.6 | 78.2 | 203 | 164 | 78.7 | nd |
| T22 | 30-Homohopane-22R | 165 | 186 | 62.5 | 60.2 | 60.9 | 165 | 141 | 60.6 | nd |

## Table 4-2:  continued

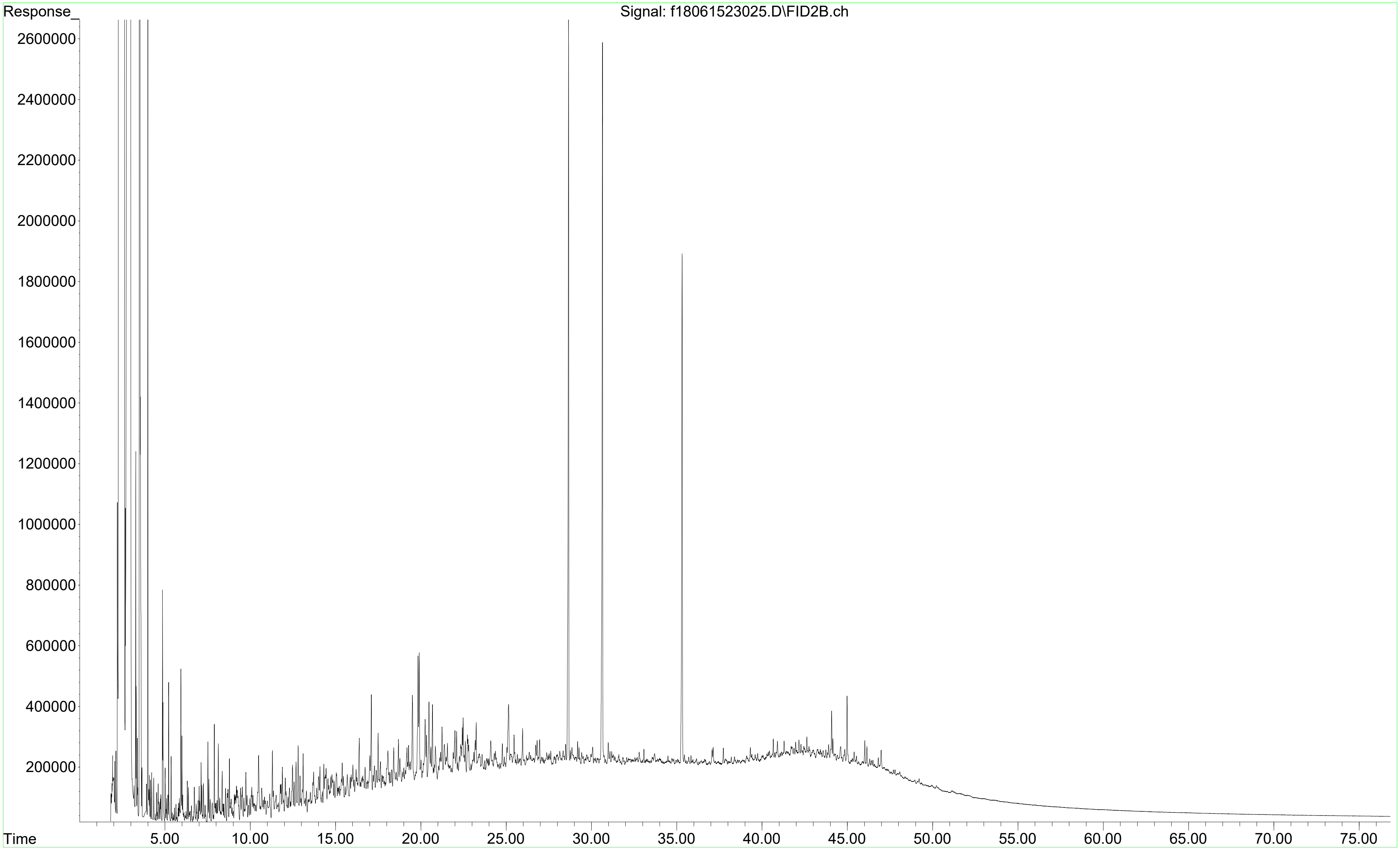
| | Client ID | 209459 | 185997 | CAVERN 4 | CAVERN 7B | CAVERN 7B (Dup) | 210185 | TANK BATTERY | 7B | BS-24 |
|---|---|---|---|---|---|---|---|---|---|---|
| | Lab ID | L2325505-01 | L2325505-02 | L2325505-03 | L2325505-04 | WG1791156-4 | L2325505-05 | L2325505-06 | L2325505-07 | L2325505-08 |
| | Date Collected | 5/2/2023 | 5/2/2023 | 5/25/2023 | 5/25/2023 | NA | 5/25/2023 | 5/25/2023 | 1/25/2023 | 5/22/2023 |
| | Date Analyzed | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/18/2023 | 6/15/2023 |
| | Analytes | Result | Result | Result | Result | Result | Result | Result | Result | Result |
| T22A | T22a-Gammacerane/C32-diahopane | 47.5 | 50.4 | 15.0 | 14.2 | 16.6 | 48.0 | 35.2 | 15.3 | 104 |
| T26 | 30,31-Bishomohopane-22S | 104 | 111 | 48.7 | 47.1 | 47.8 | 106.0 | 87.6 | 50.3 | nd |
| T27 | 30,31-Bishomohopane-22R | 156 | 176 | 35.3 | 34.9 | 35.8 | 157.0 | 114 | 36.2 | 176 |
| T30 | 30,31-Trishomohopane-22S | 61.8 | 70.0 | 33.4 | 33.1 | 35.7 | 61.9 | 51.3 | 33.7 | nd |
| T31 | 30,31-Trishomohopane-22R | 45.1 | 44.6 | 25.7 | 24.2 | 23.2 | 43.4 | 32.7 | 23.1 | nd |
| T32 | Tetrakishomohopane-22S | 41.7 | 42.0 | 23.2 | 26.4 | 24.6 | 40.6 | 31.3 | 26.9 | nd |
| T33 | Tetrakishomohopane-22R | 26.7 | 27.3 | 20.2 | 20.5 | 20.1 | 29.2 | 22.8 | 17.3 | nd |
| T34 | Pentakishomohopane-22S | 21.7 | 19.6 | 22.8 | 25.4 | 22.8 | 22.0 | 13.9 | 26.2 | nd |
| T35 | Pentakishomohopane-22R | 21.3 | 19.6 | 15.5 | 14.6 | 19.0 | 18.0 | 13.9 | 17.8 | nd |
| S4 | 13b(H),17a(H)-20S-Diacholestane | 81.6 | 88.8 | 26.2 | 32.2 | 34.5 | 90.3 | 63.7 | 35.0 | 127 |
| S5 | 13b(H),17a(H)-20R-Diacholestane | 54.2 | 58.5 | 11.0 | 15.1 | 15.7 | 56.1 | 40.2 | 15.1 | 88.5 |
| S23 | 14b,17b-20S-Methylcholestane | 43.9 | 45.1 | 33.3 | 36.0 | 35.0 | 50.0 | 36.2 | 33.8 | nd |
| S26 | 14b(H),17b(H)-20R-Ethylcholestane | 57.6 | 60.3 | 45.3 | 43.5 | 46.0 | 62.6 | 50.7 | 48.6 | 17.1 |
| S27 | 14b(H),17b(H)-20S-Ethylcholestane | 29.6 | 31.4 | 32.4 | 33.2 | 35.2 | 30.9 | 23.5 | 31.8 | 9.08 |
| TAS05 | C20 PREGNANE | 74.1 | 81.8 | 88.5 | 86.4 | 91.2 | 76.4 | 58.8 | 89.0 | nd |
| TAS06 | C21 20-METHYLPREGNANE | 58.2 | 61.5 | 71.2 | 72.8 | 76.0 | 62.5 | 45.6 | 76.2 | 7.95 |
| TAS07 | C22 20-ETHYLPREGNANE (A) | 13.7 | 13.6 | 38.1 | 34.2 | 34.7 | 13.6 | 9.9 | 39.6 | 8.97 |
| TAS08 | C22 20-ETHYLPREGNANE (B) | 17.5 | 16.5 | 18.5 | 19.7 | 19.1 | 15.6 | 13.2 | 18.3 | 8.94 |
| TAS09 | C26,20S TAS | 73.9 | 90.2 | 17.3 | 24.6 | 26.0 | 85.2 | 68.2 | 26.0 | 132 |
| TAS01 | C26,20R+C27,20S TAS | 244 | 265 | 157 | 183 | 188 | 268 | 194 | 182 | 411 |
| TAS02 | C28,20S TAS | 196 | 215 | 125 | 125 | 132 | 214 | 158 | 126 | 328 |
| TAS03 | C27,20R TAS | 154 | 170 | 125 | 140 | 148 | 162 | 124 | 140 | 242 |
| TAS04 | C28,20R TAS | 164 | 172 | 103 | 103 | 111 | 174 | 124 | 108 | 274 |
| TAS10 | C29,20S TAS | 59.1 | 56.8 | 29.2 | 44.7 | 43.2 | 66.3 | 43.4 | 47.3 | 82.2 |
| TAS11 | C29,20R TAS | 23.3 | 21.1 | 12.5 | 20.9 | 21.9 | 22.2 | 13.4 | 19.1 | 39.0 |

# Attachment 5

# GC/FID Chromatograms

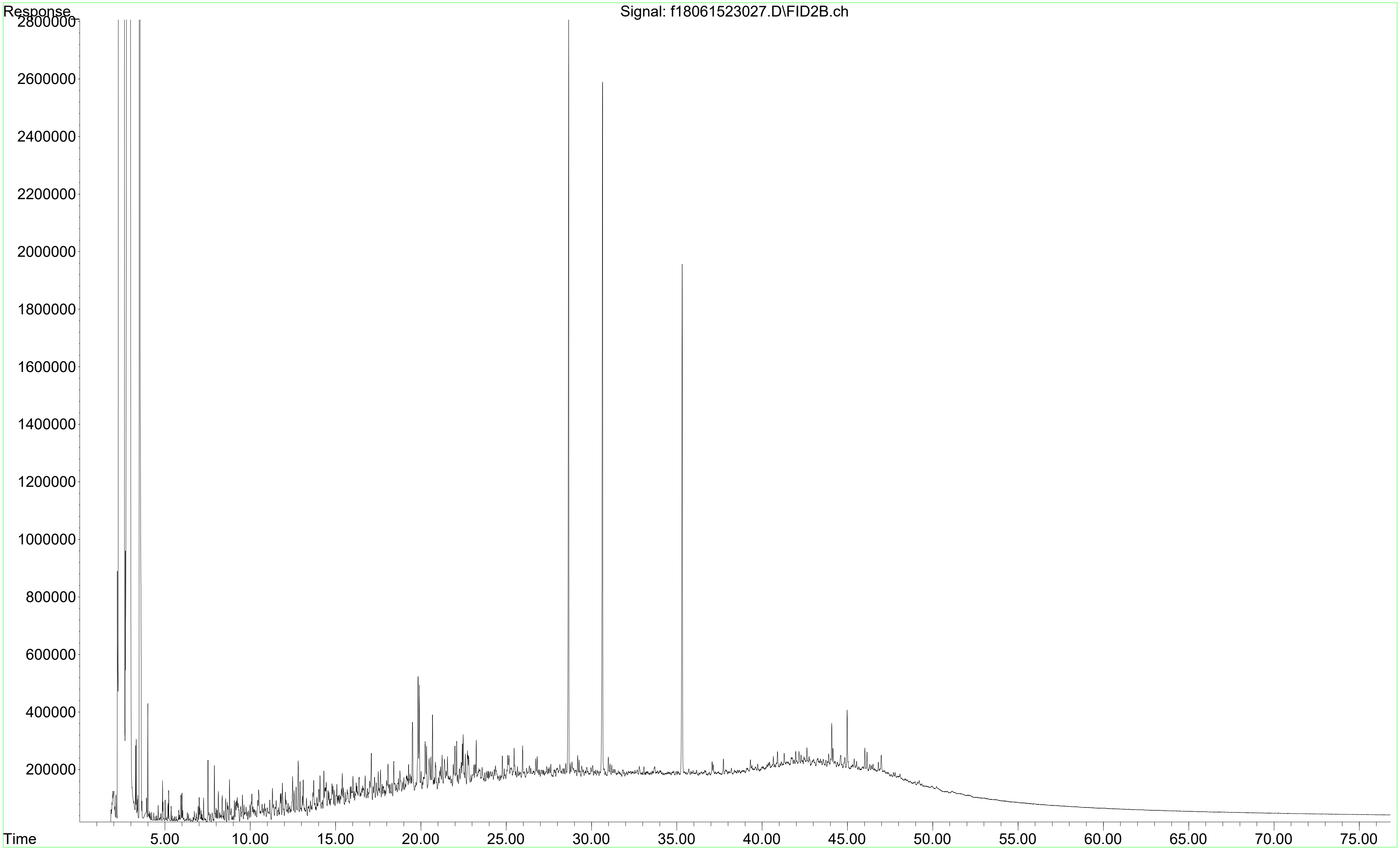
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...         C\f18061523025.D
Operator   : FID18:AMV
Instrument :   FID 18
Acquired   : 16 Jun 2023  02:31 am using AcqMethod FID18.M
Sample Name: L2325505-01
Misc Info  : WG1791862,WG1791156,ICAL19877
```

209459
L2325505-01



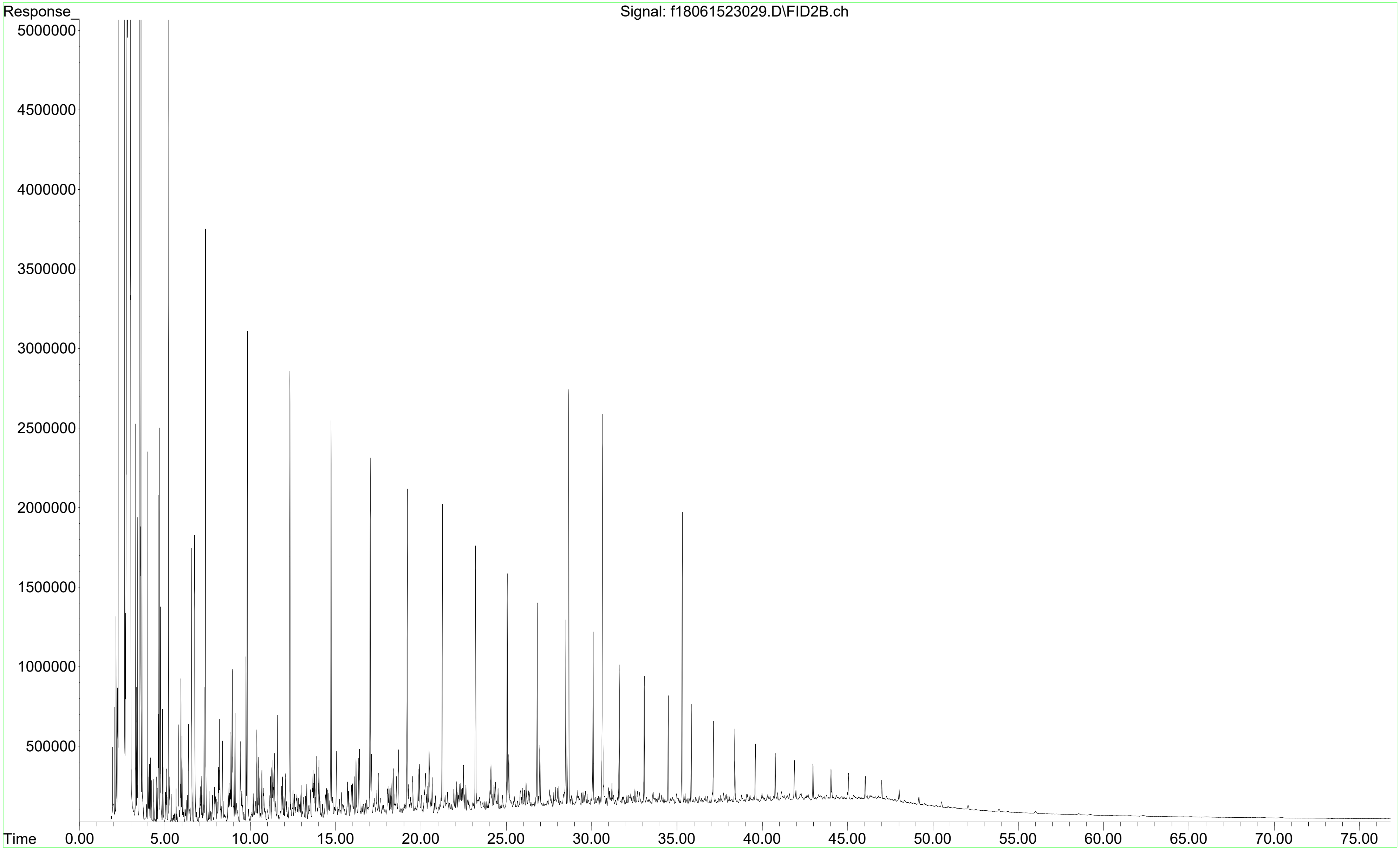
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...          C\f18061523027.D
Operator    : FID18:AMV
Instrument  :   FID 18
Acquired    : 16 Jun 2023  03:54 am using AcqMethod FID18.M
Sample Name: L2325505-02
Misc Info   : WG1791862,WG1791156,ICAL19877
```

185997
L2325505-02



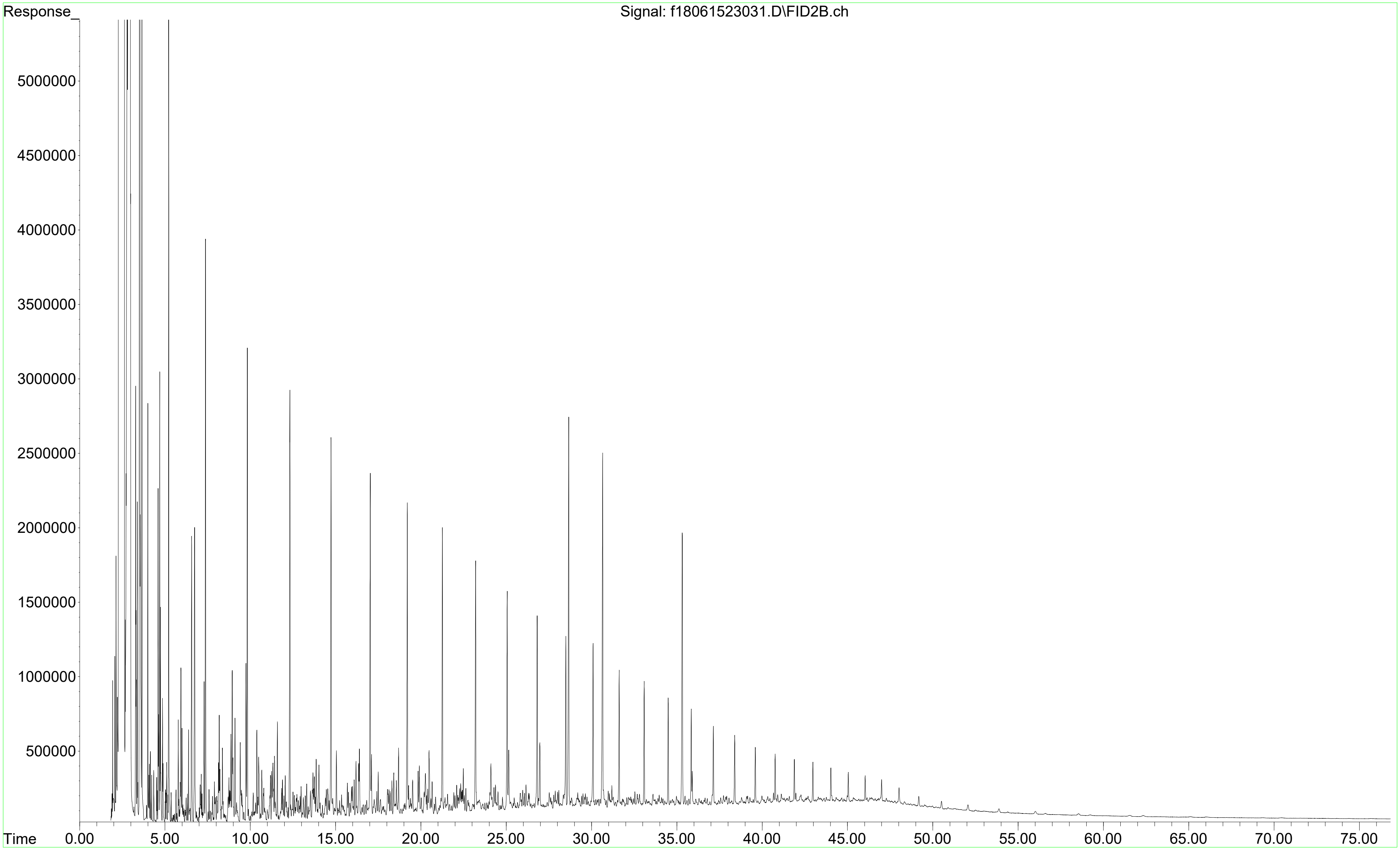
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...          C\f18061523029.D
Operator    : FID18:AMV
Instrument  :   FID 18
Acquired    : 16 Jun 2023  05:18 am using AcqMethod FID18.M
Sample Name: L2325505-03
Misc Info   : WG1791862,WG1791156,ICAL19877
```

CAVERN 4
L2325505-03



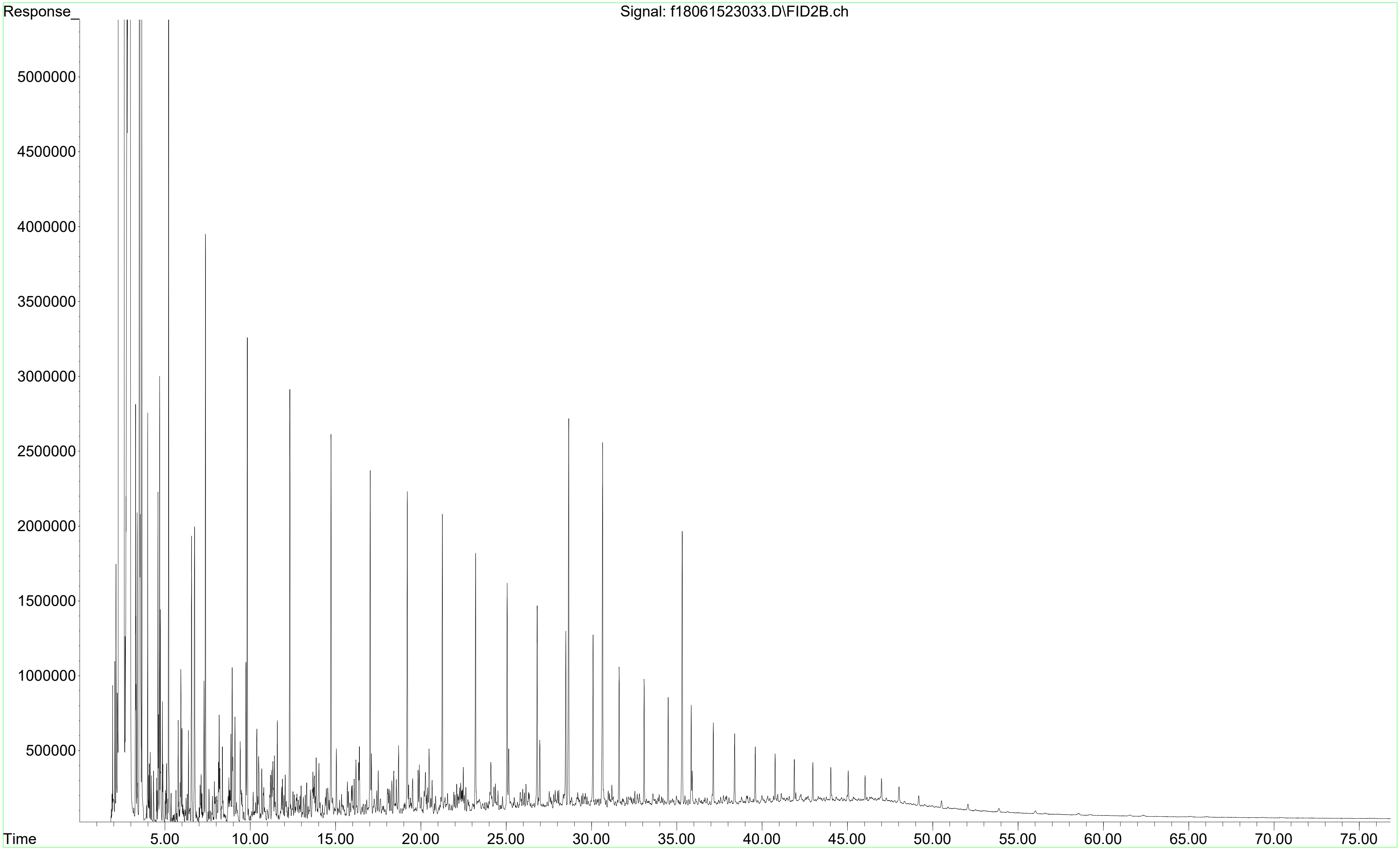
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...         C\f18061523031.D
Operator   : FID18:AMV
Instrument :   FID 18
Acquired   : 16 Jun 2023  06:42 am using AcqMethod FID18.M
Sample Name: L2325505-04
Misc Info  : WG1791862,WG1791156,ICAL19877
```

CAVERN 7B
L2325505-04



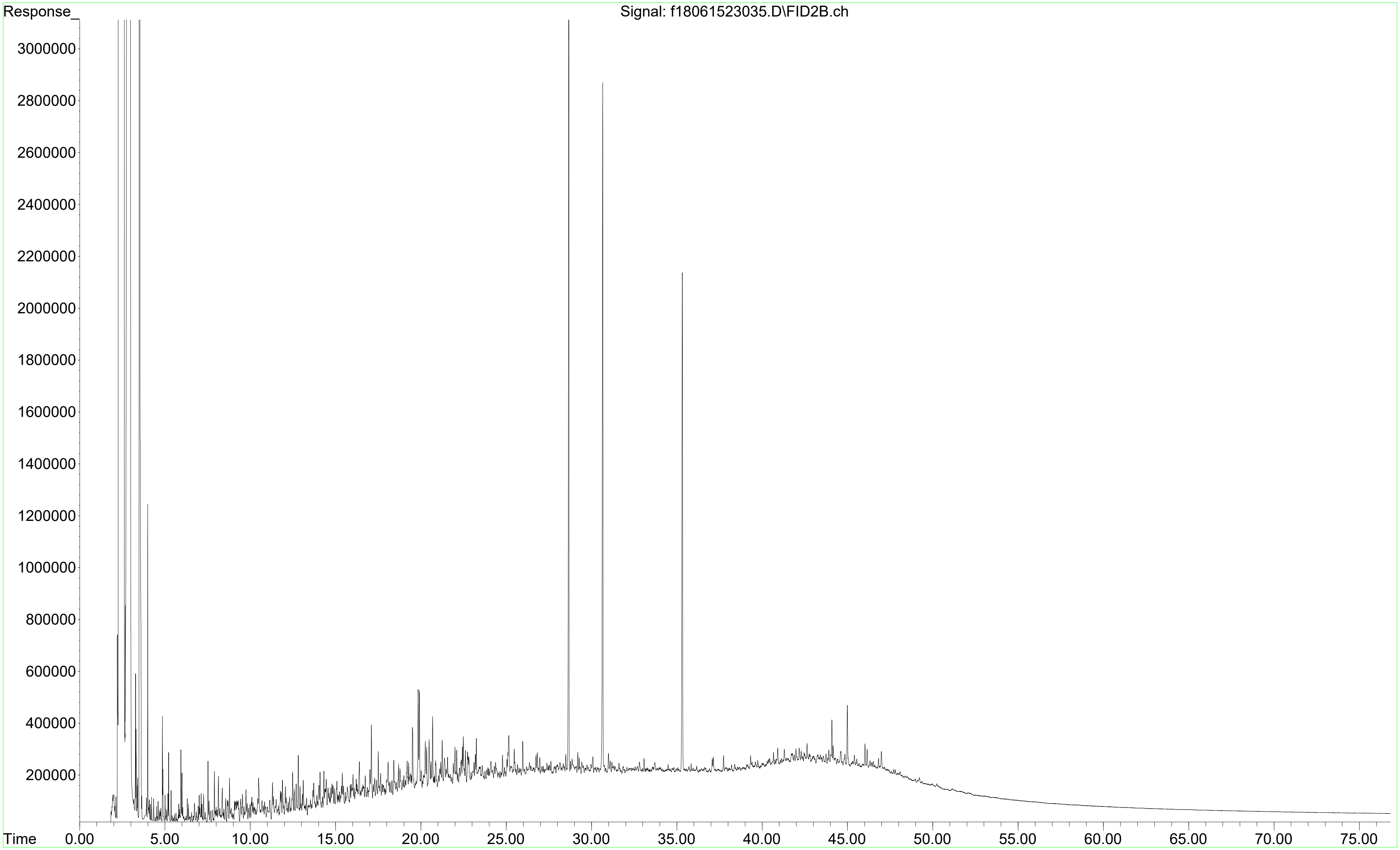
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...          C\f18061523033.D
Operator    : FID18:AMV
Instrument :   FID 18
Acquired    : 16 Jun 2023  08:06 am using AcqMethod FID18.M
Sample Name: WG1791156-4
Misc Info   : WG1791862,WG1791156,ICAL19877
```

CAVERN 7B Duplicate
WG1791156-4



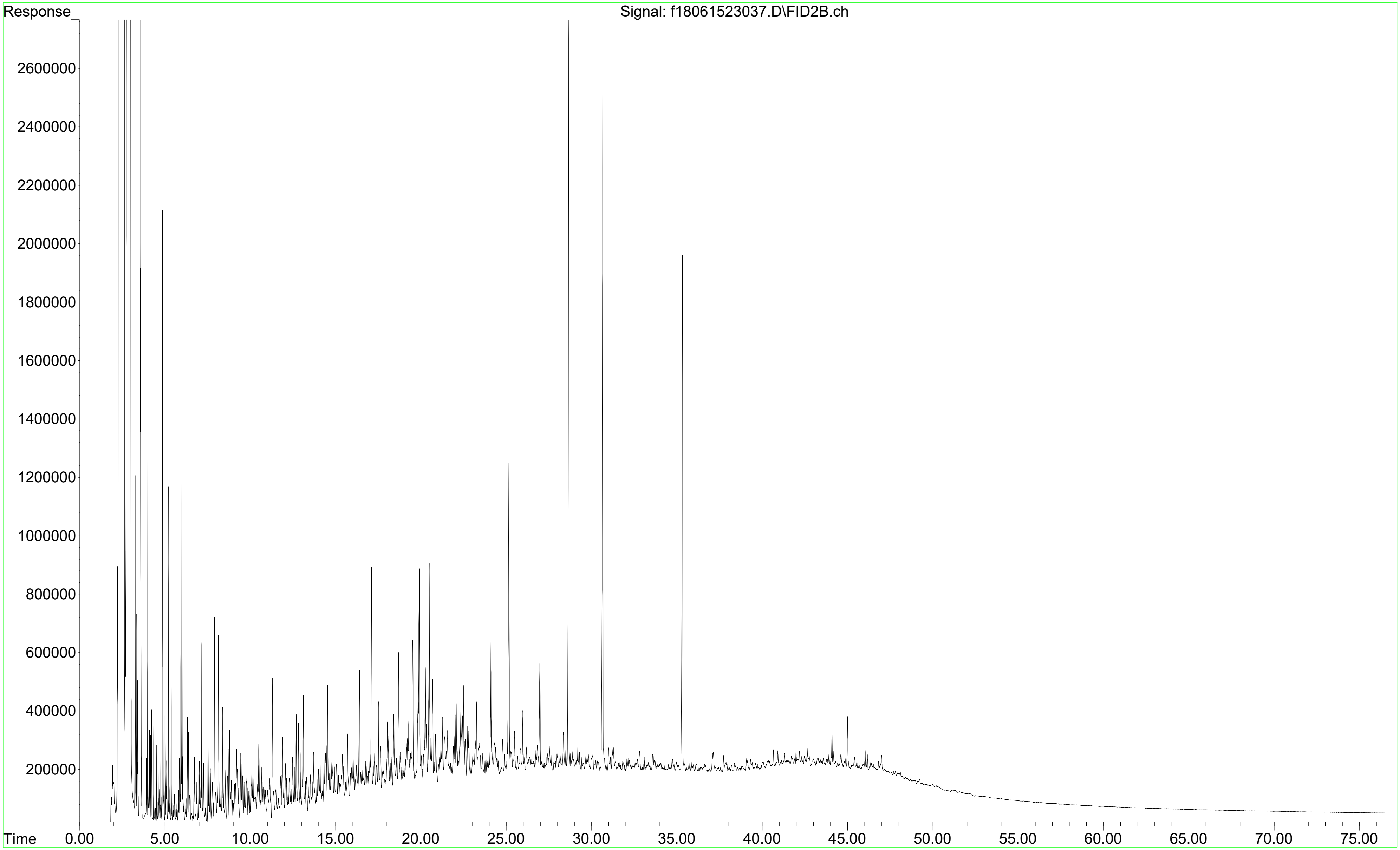
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...         C\f18061523035.D
Operator   : FID18:AMV
Instrument :   FID 18
Acquired   : 16 Jun 2023  09:30 am using AcqMethod FID18.M
Sample Name: L2325505-05
Misc Info  : WG1791862,WG1791156,ICAL19877
```

210185
L2325505-05



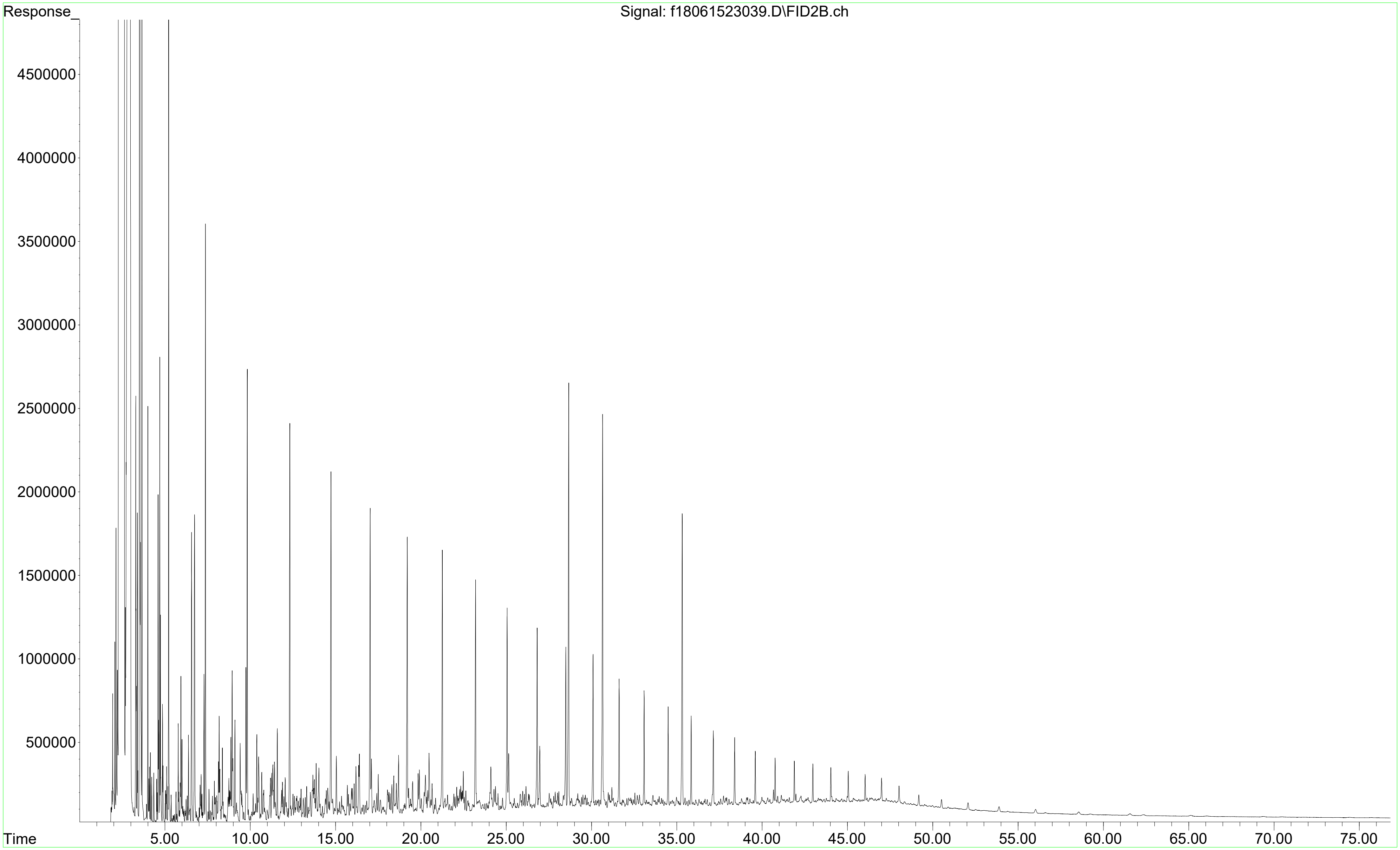
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...          C\f18061523037.D
Operator    : FID18:AMV
Instrument  :   FID 18
Acquired    : 16 Jun 2023  10:54 am using AcqMethod FID18.M
Sample Name: L2325505-06
Misc Info   : WG1791862,WG1791156,ICAL19877
```

TANK BATTERY
L2325505-06



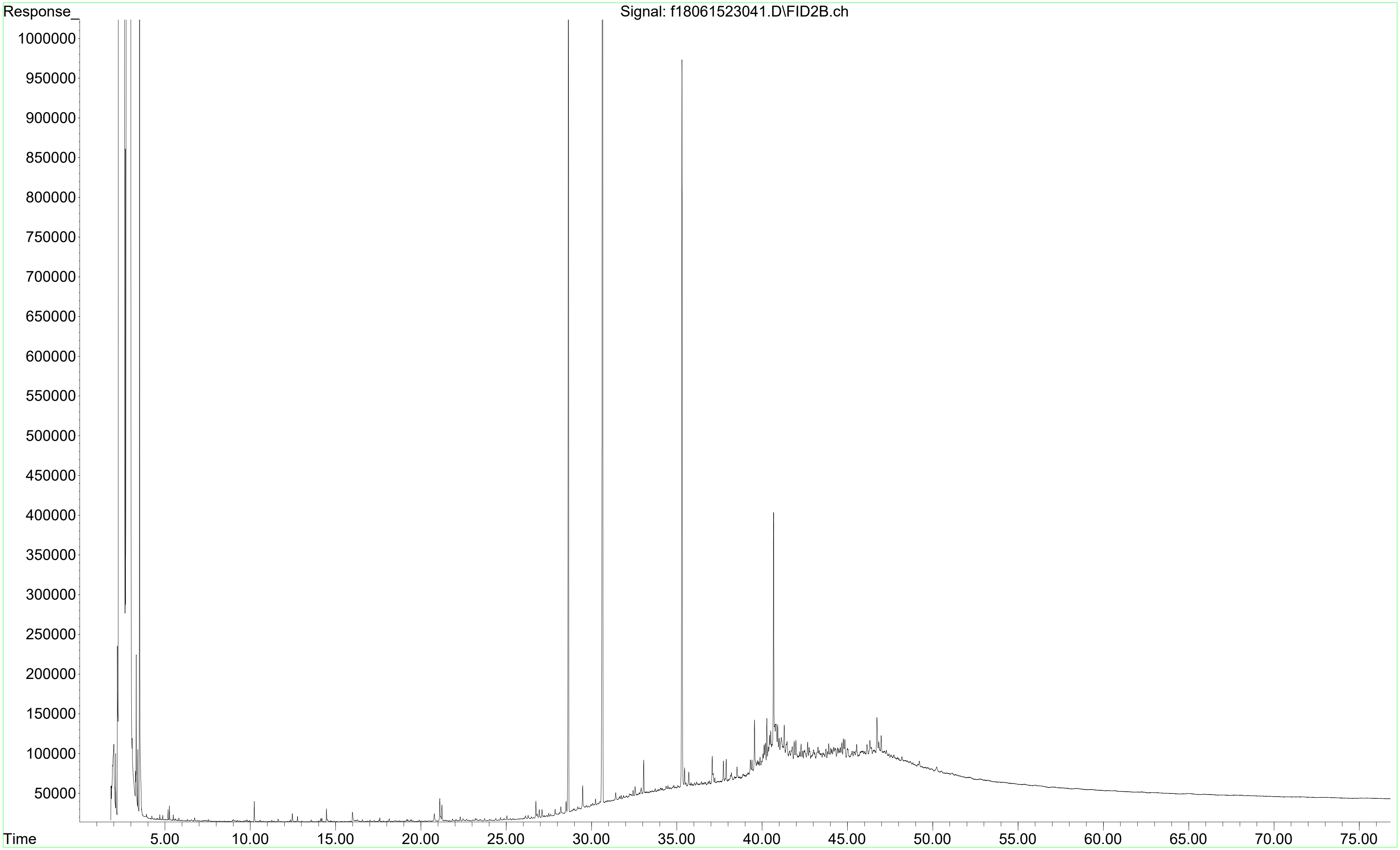
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...         C\f18061523039.D
Operator   : FID18:AMV
Instrument :   FID 18
Acquired   : 16 Jun 2023  12:18 pm using AcqMethod FID18.M
Sample Name: L2325505-07
Misc Info  : WG1791862,WG1791156,ICAL19877
```

7B
L2325505-07
Reference Oil



```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\SH
...          C\f18061523041.D
Operator    : FID18:AMV
Instrument  :   FID 18
Acquired    : 16 Jun 2023  01:42 pm using AcqMethod FID18.M
Sample Name: L2325505-08
Misc Info   : WG1791862,WG1790071,ICAL19877
```

BS-24
L2325505-08

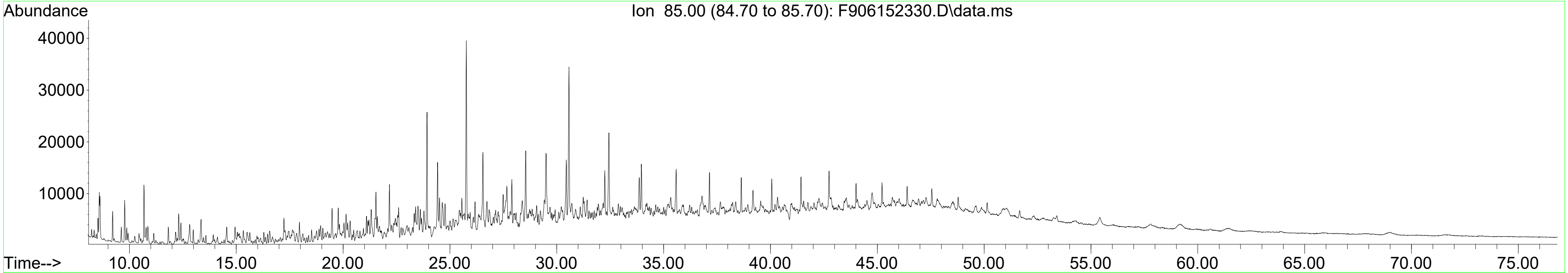


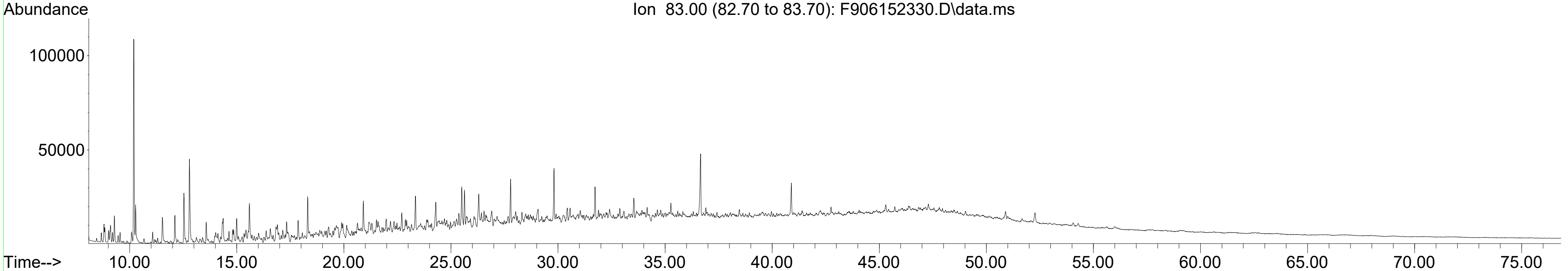
# Attachment 6

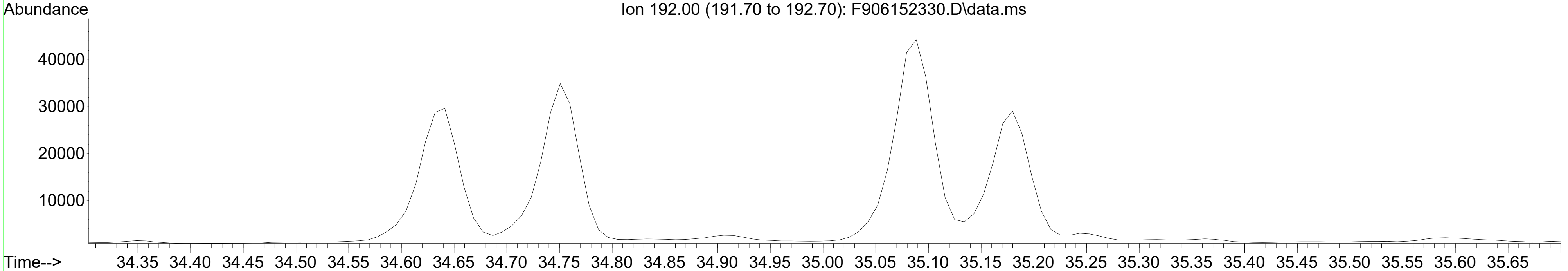
# GC/MS Extracted Ion Profiles

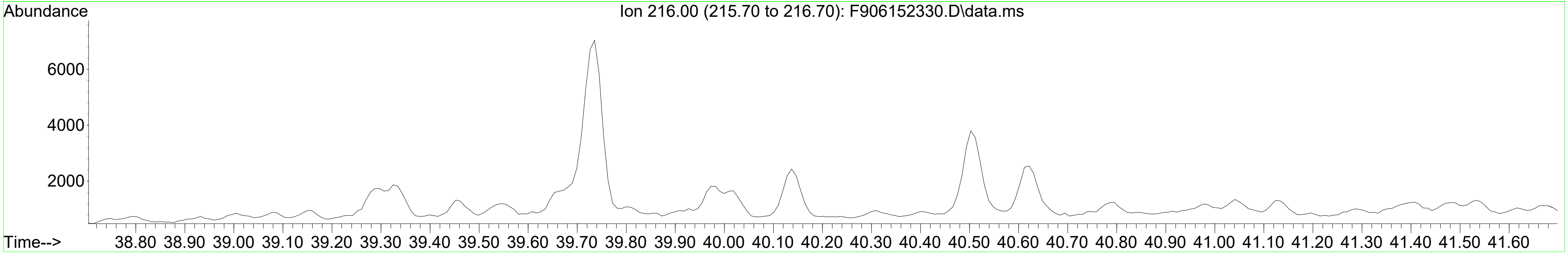
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152330.D
Operator    : PAH9:MJS
Instrument :    PAH9
Acquired    : 18 Jun 2023  12:16 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-01,32,,
Misc Info   : WG1793207,WG1791156,ICAL19995
```

209459
L2325505-01









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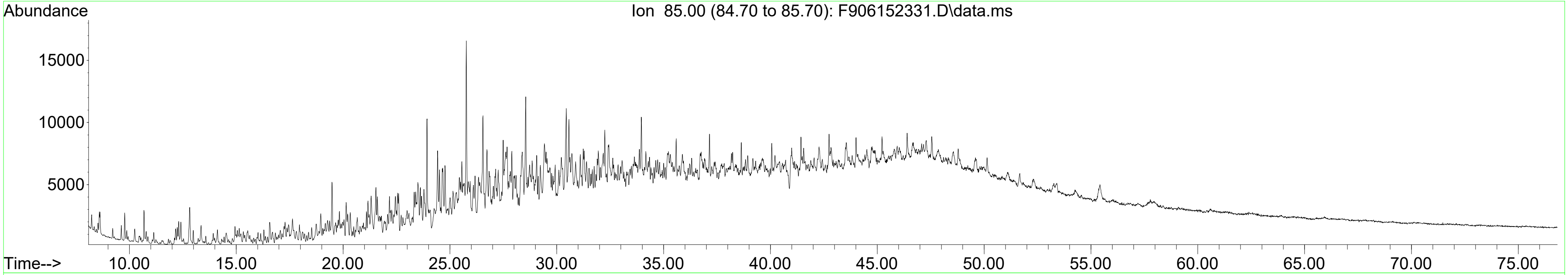
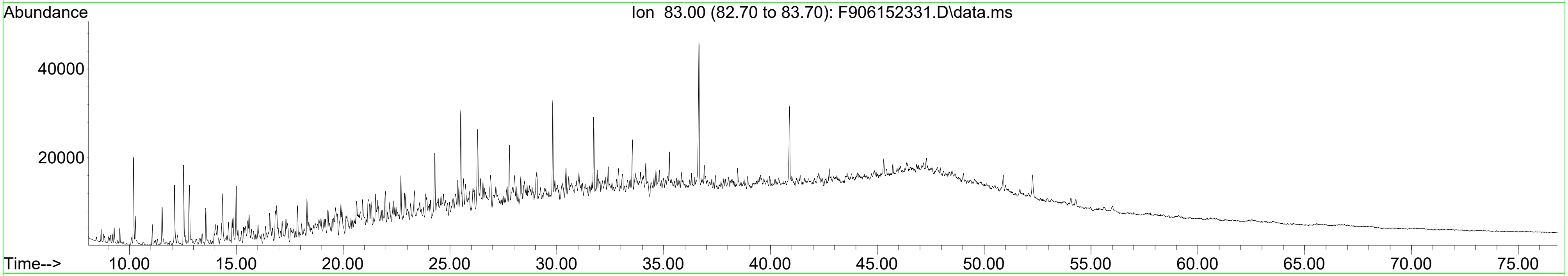
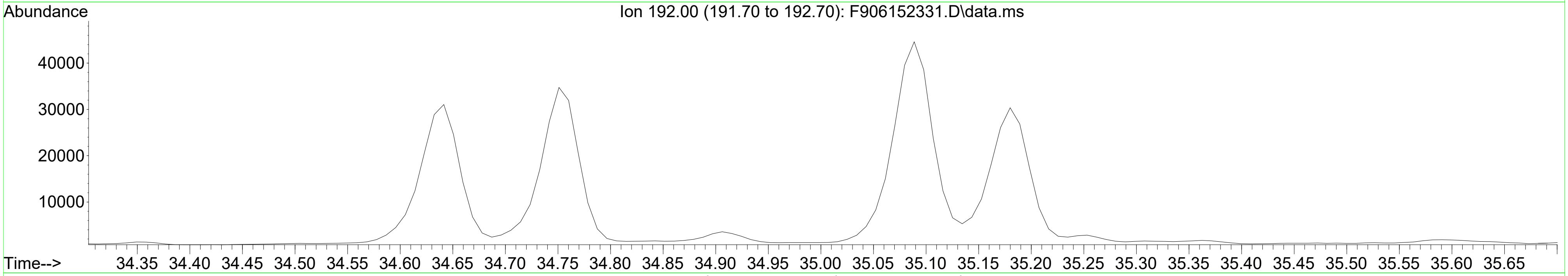
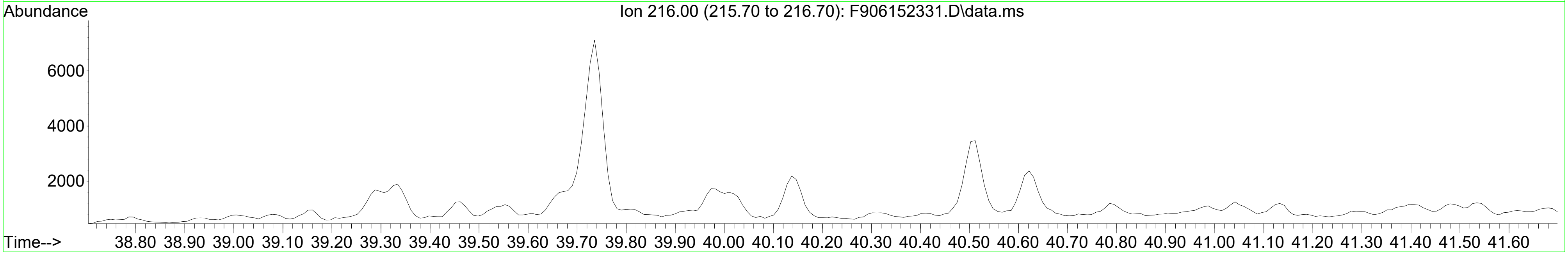
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152331.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023   1:39 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-02,32,,
Misc Info   : WG1793207,WG1791156,ICAL19995
```

185997
L2325505-02



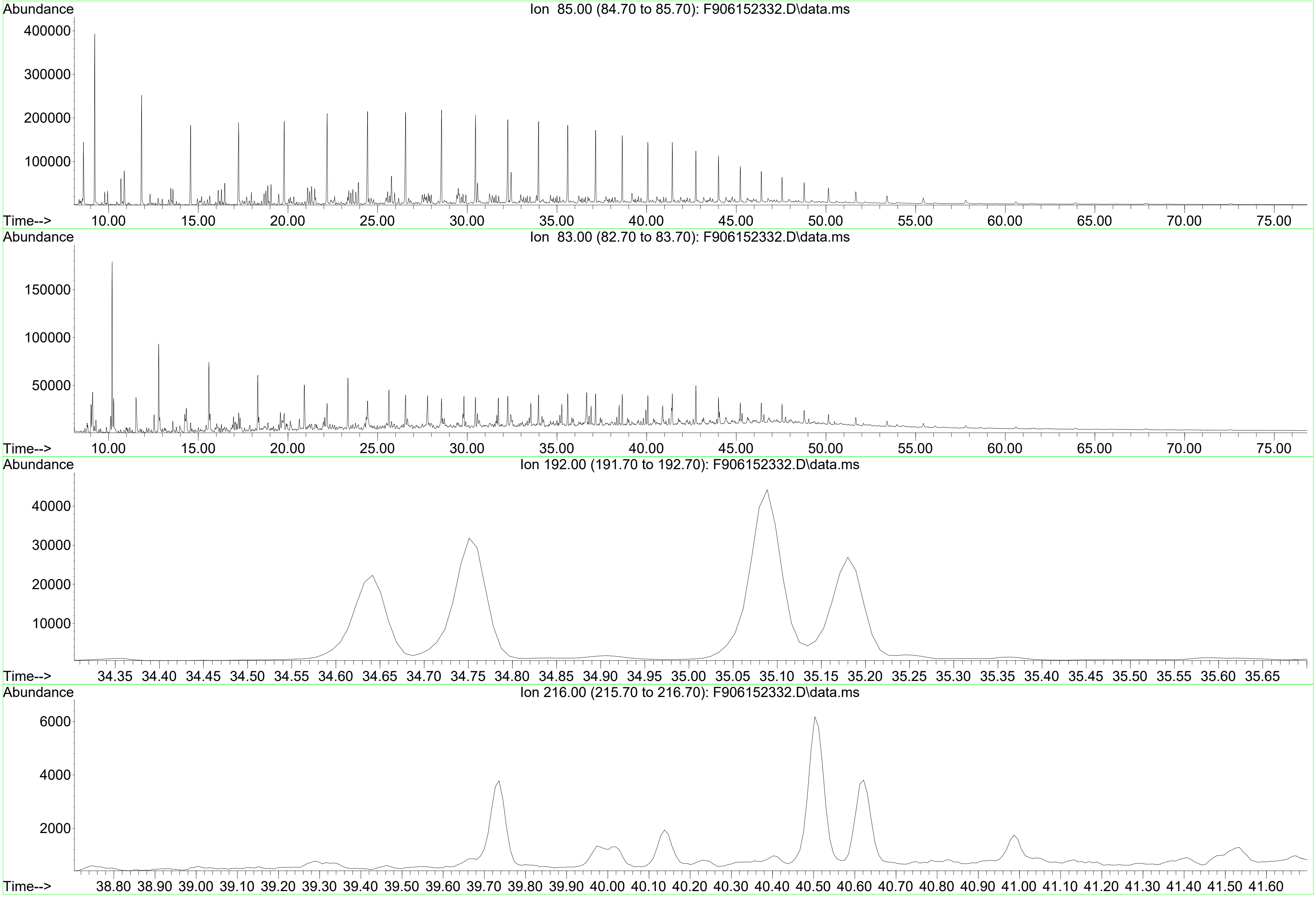






```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152332.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   3:03 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-03,32,,
Misc Info  : WG1793207,WG1791156,ICAL19995
```

CAVERN 4
L2325505-03


Ion 85.00 (84.70 to 85.70): F906152332.D\data.ms

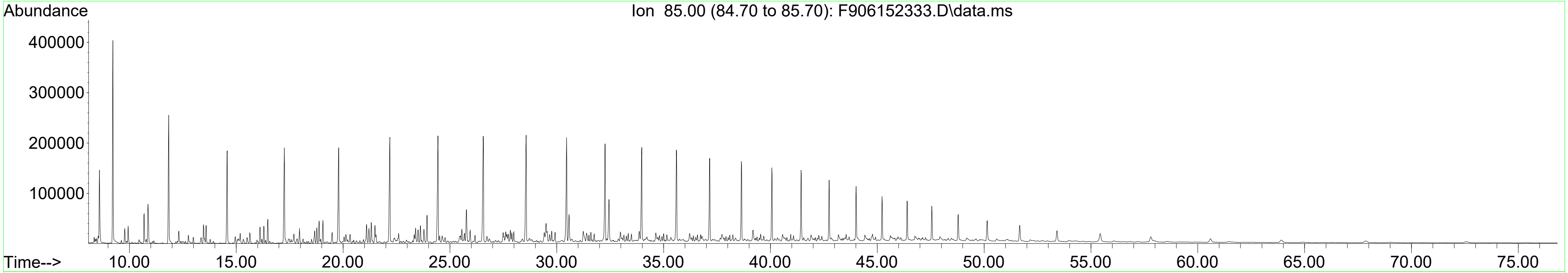
Ion 83.00 (82.70 to 83.70): F906152332.D\data.ms

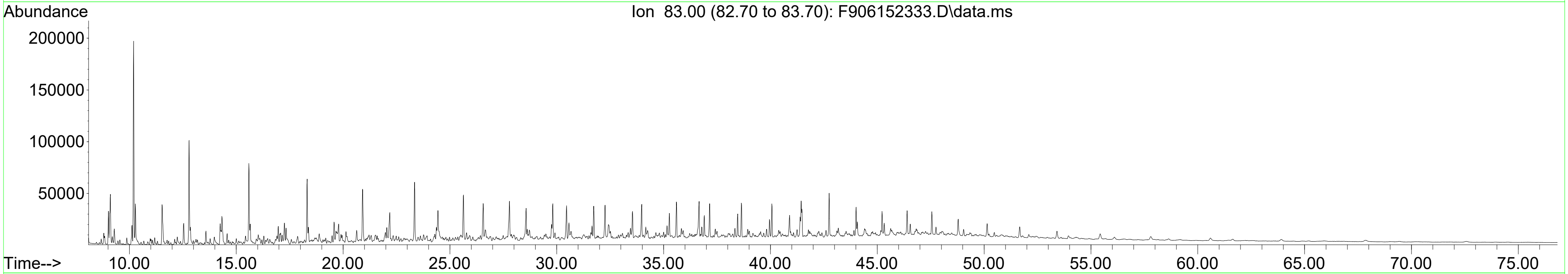
Ion 192.00 (191.70 to 192.70): F906152332.D\data.ms

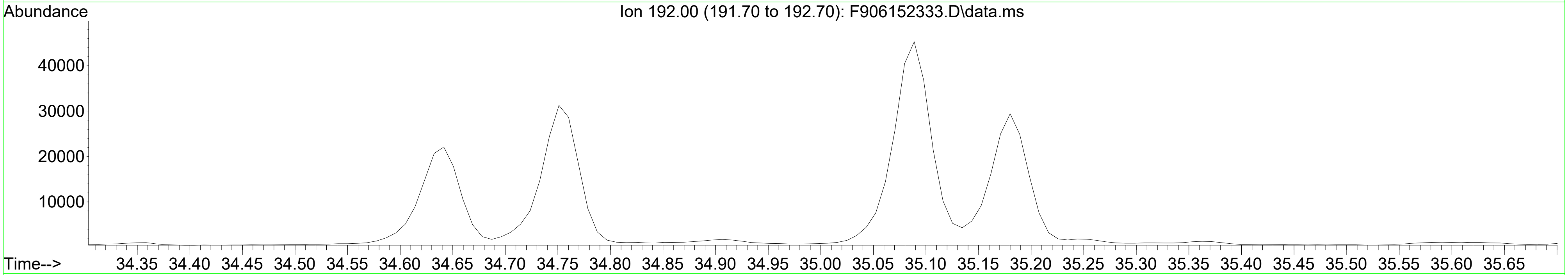
Ion 216.00 (215.70 to 216.70): F906152332.D\data.ms

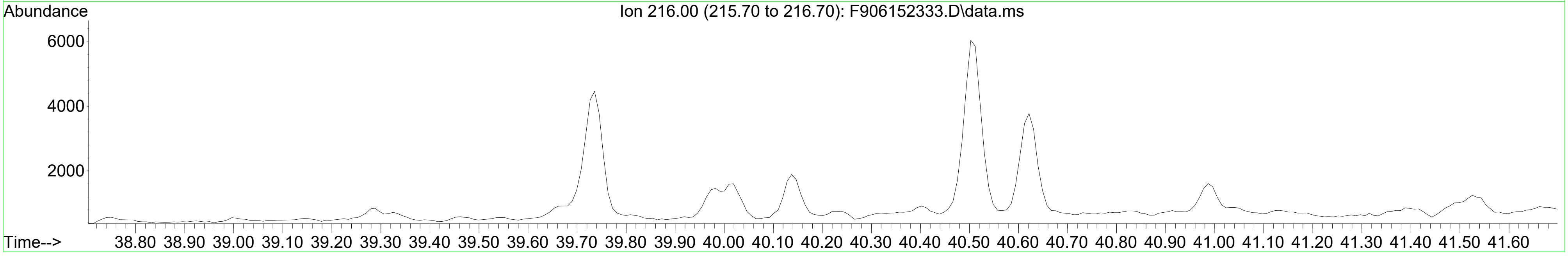
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152333.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   4:27 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-04,32,,
Misc Info  : WG1793207,WG1791156,ICAL19995
```

CAVERN 7B
L2325505-04









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File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152334.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023   5:50 pm using AcqMethod FRNC9ALT.M
Sample Name: WG1791156-4,32
Misc Info   : WG1793207,WG1791156,ICAL19995
```
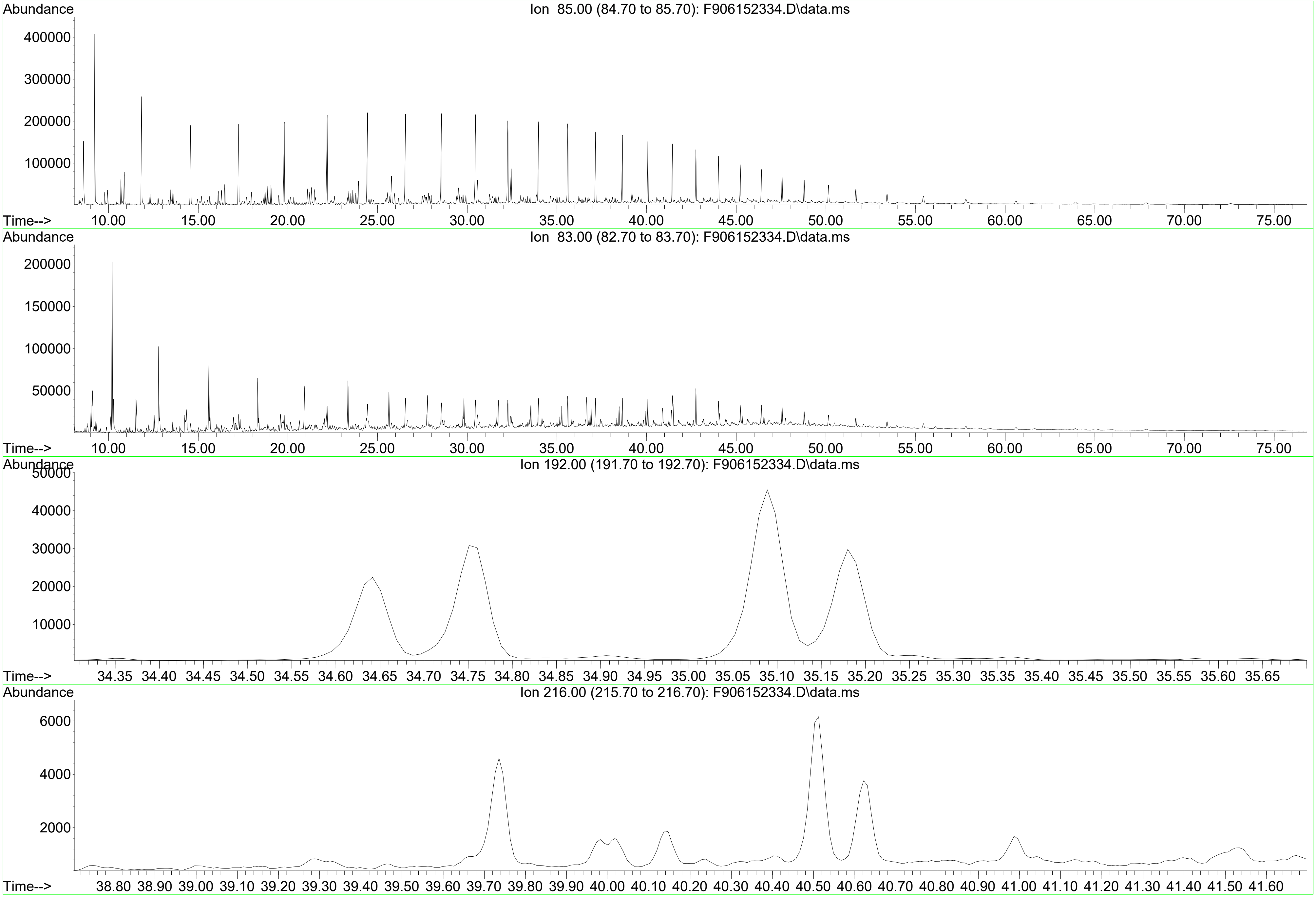
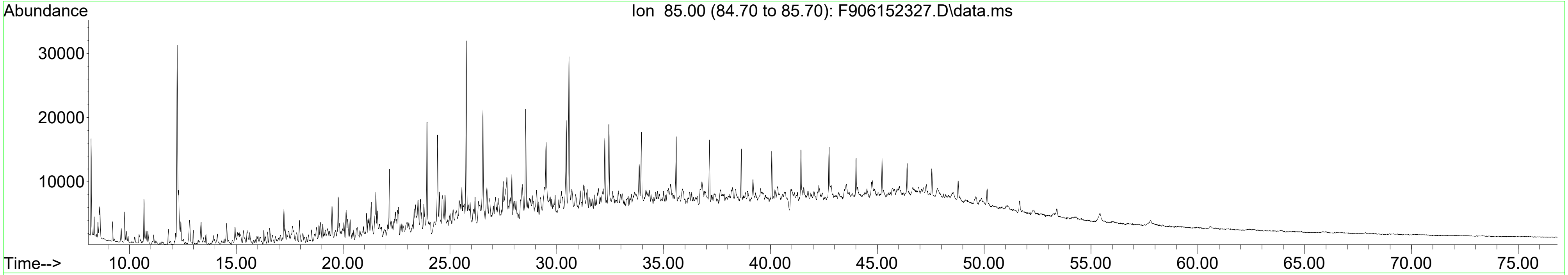
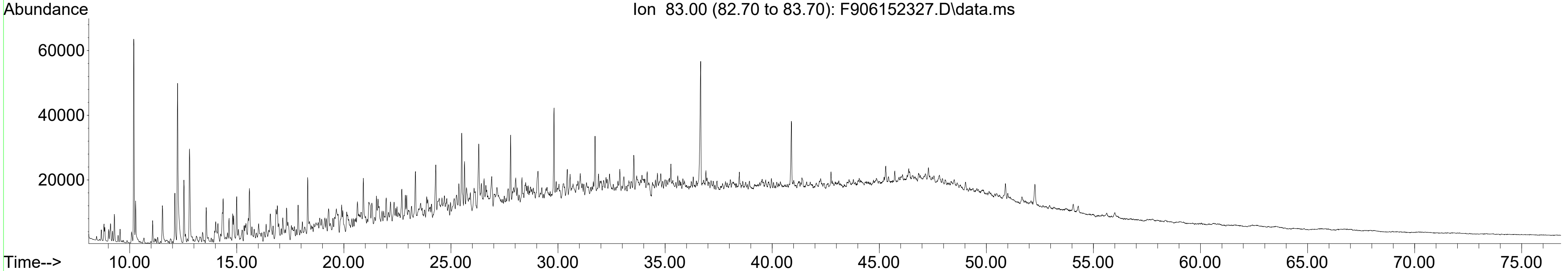

CAVERN 7B Duplicate
WG1791156-4

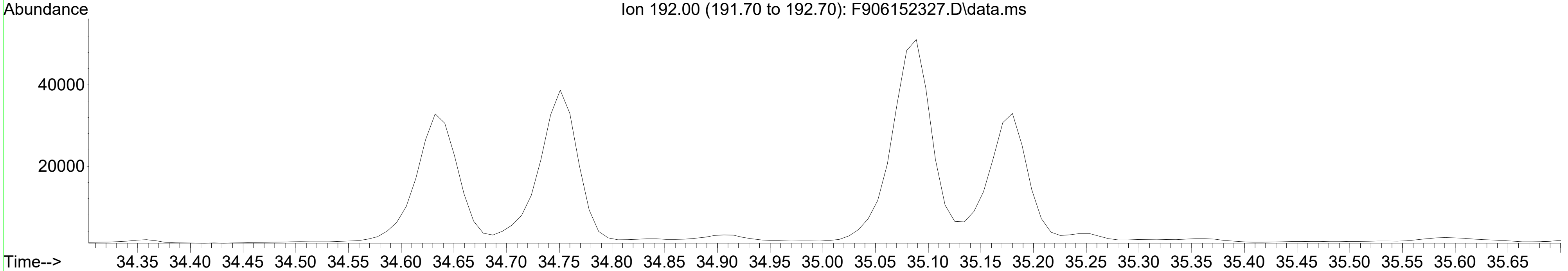


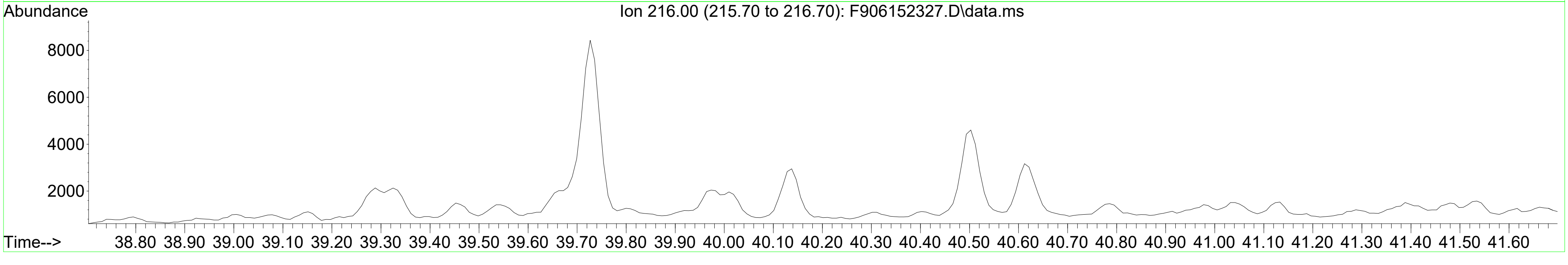
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152327.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023   8:05 am using AcqMethod FRNC9ALT.M
Sample Name: L2325505-05,32
Misc Info   : WG1793207,WG1791156,ICAL19995

210185
L2325505-05



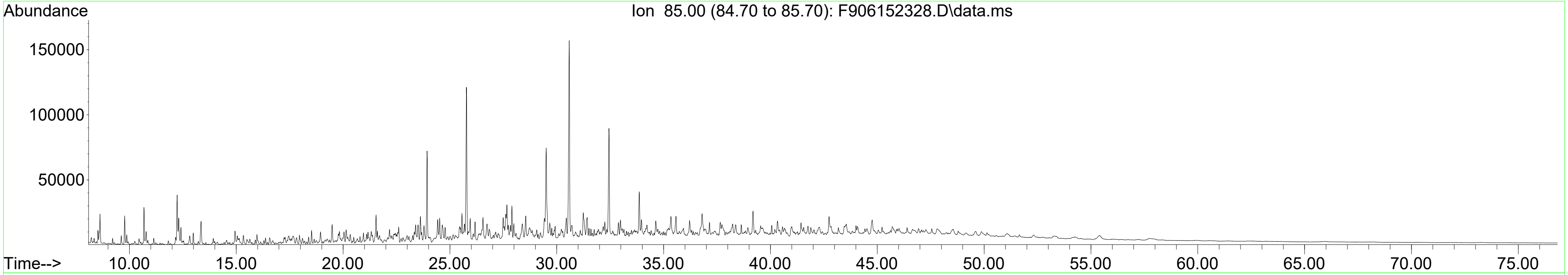


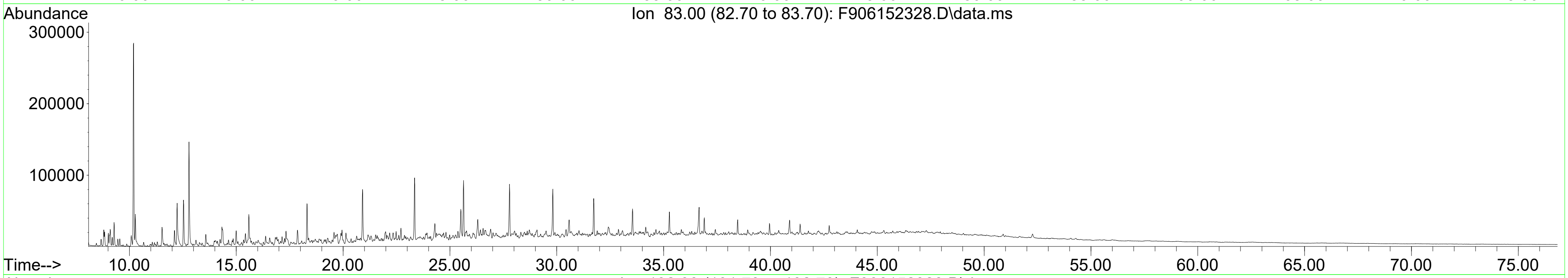


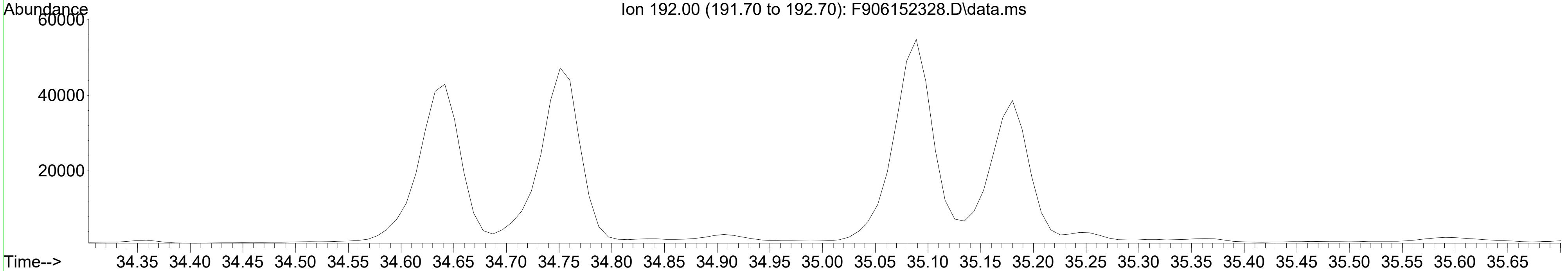


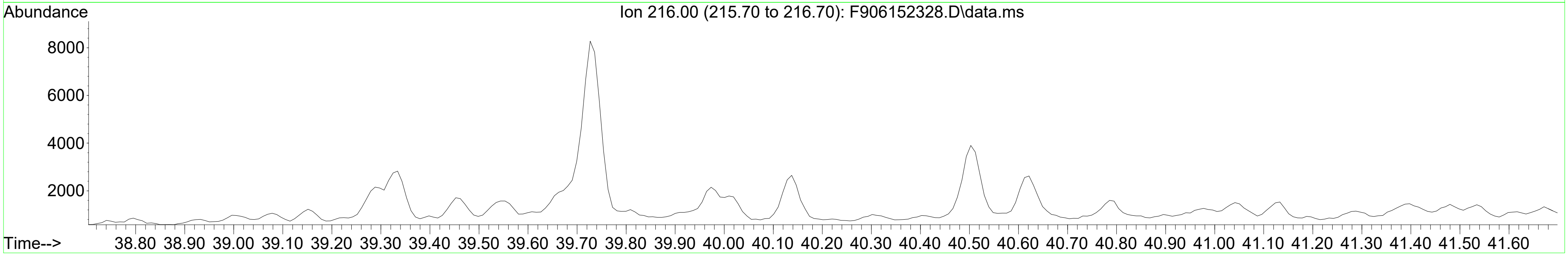
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152328.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   9:28 am using AcqMethod FRNC9ALT.M
Sample Name: L2325505-06,32
Misc Info  : WG1793207,WG1791156,ICAL19995
```

TANK BATTERY
L2325505-06



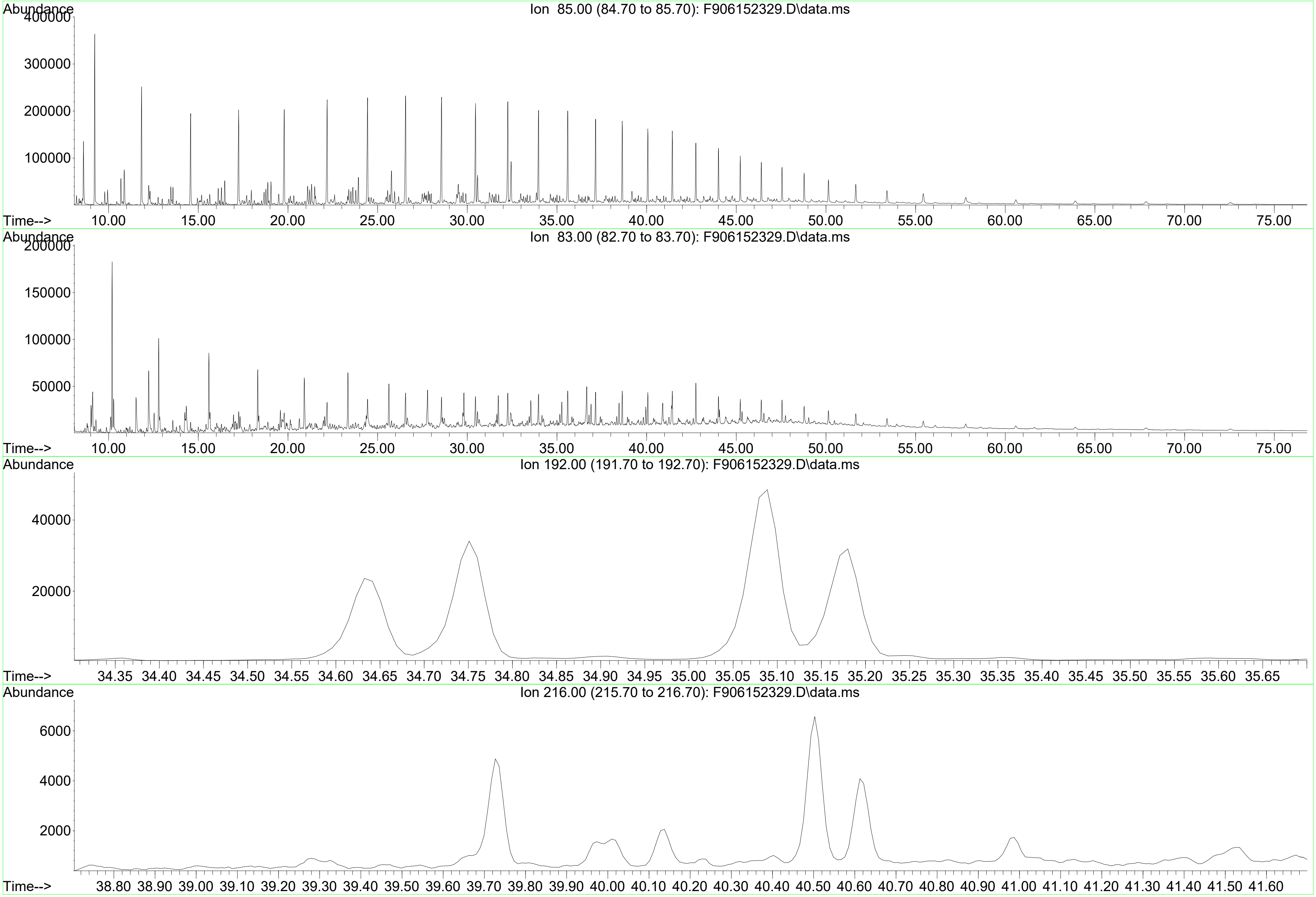






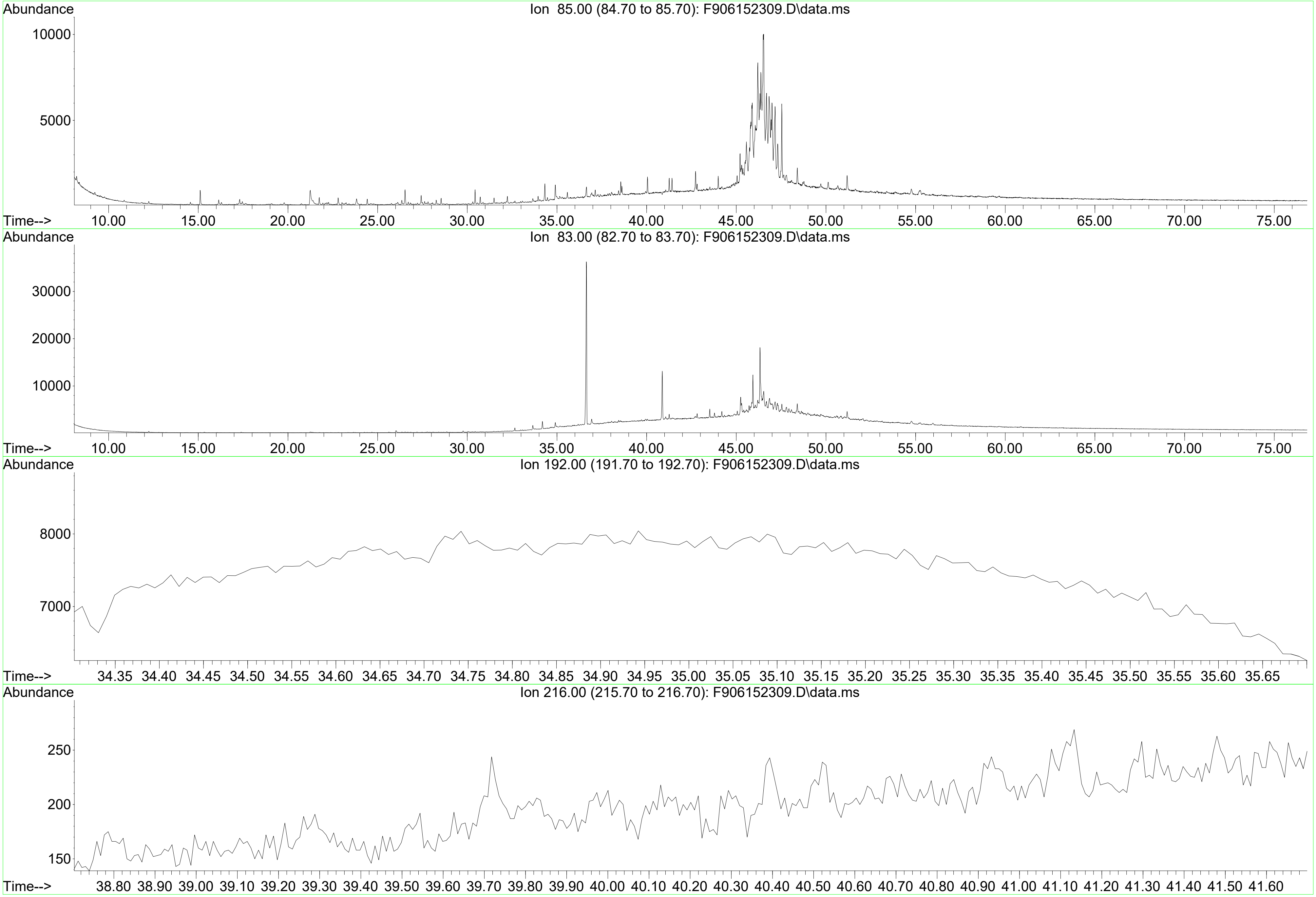
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152329.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023  10:52 am using AcqMethod FRNC9ALT.M
Sample Name: L2325505-07,32
Misc Info   : WG1793207,WG1791156,ICAL19995
```

7B
L2325505-07
Reference Oil



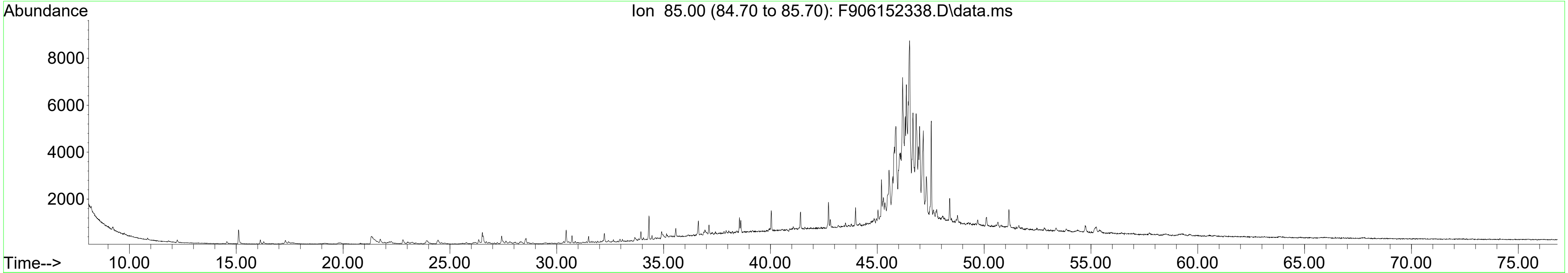
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152309.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 15 Jun 2023  10:33 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-08,32,,
Misc Info  : WG1793207,WG1790071,ICAL19995
```

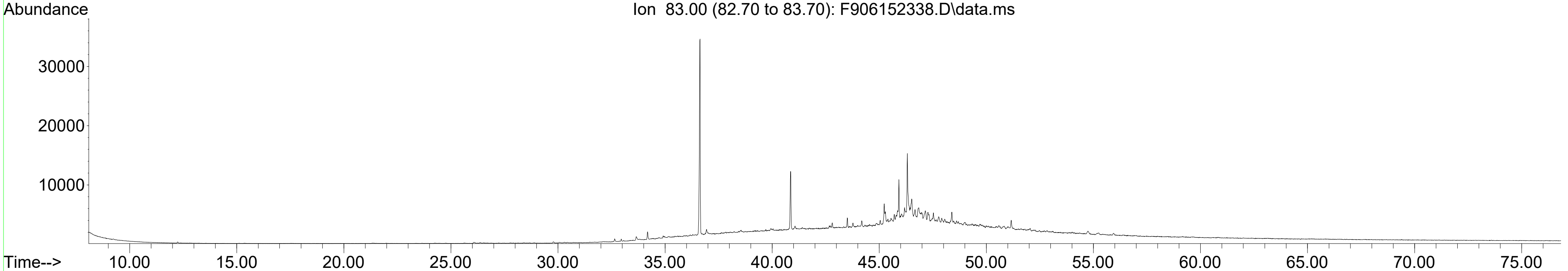
BS-24
L2325505-08

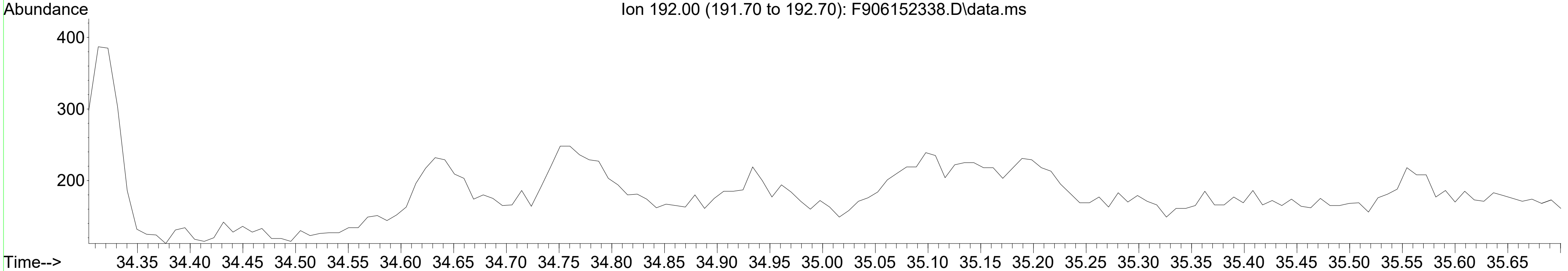


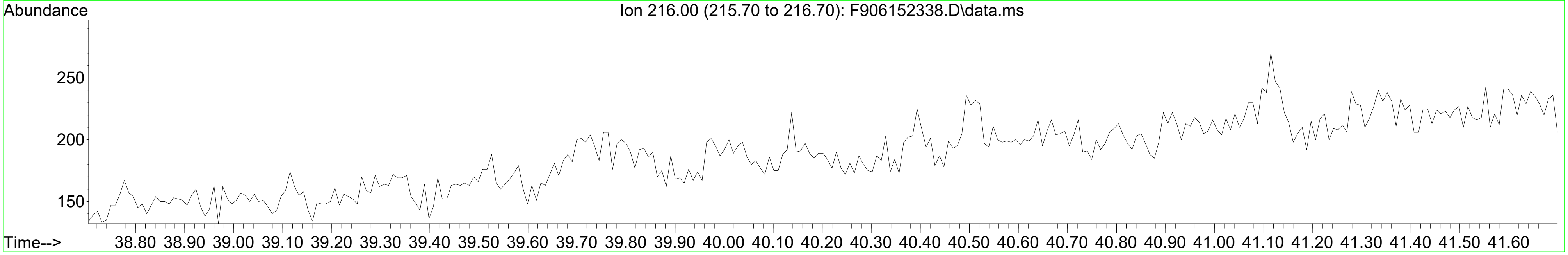
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152338.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 19 Jun 2023   1:13 pm using AcqMethod FRNC9ALT.M
Sample Name: l2325505-08,32,,
Misc Info   : WG1793207,WG1790071,ICAL19995
```

BS-24
L2325505-08
Post Copper Cleaned


Ion 85.00 (84.70 to 85.70): F906152338.D\data.ms


Ion 83.00 (82.70 to 83.70): F906152338.D\data.ms


Ion 192.00 (191.70 to 192.70): F906152338.D\data.ms


Ion 216.00 (215.70 to 216.70): F906152338.D\data.ms

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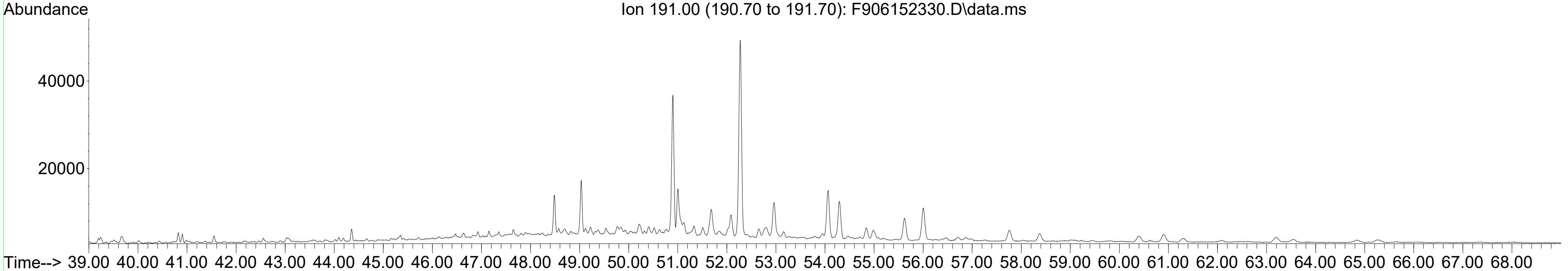
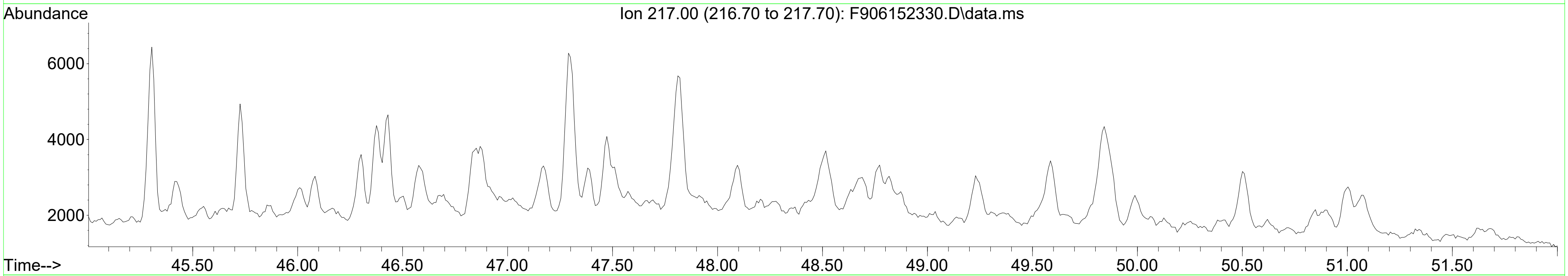
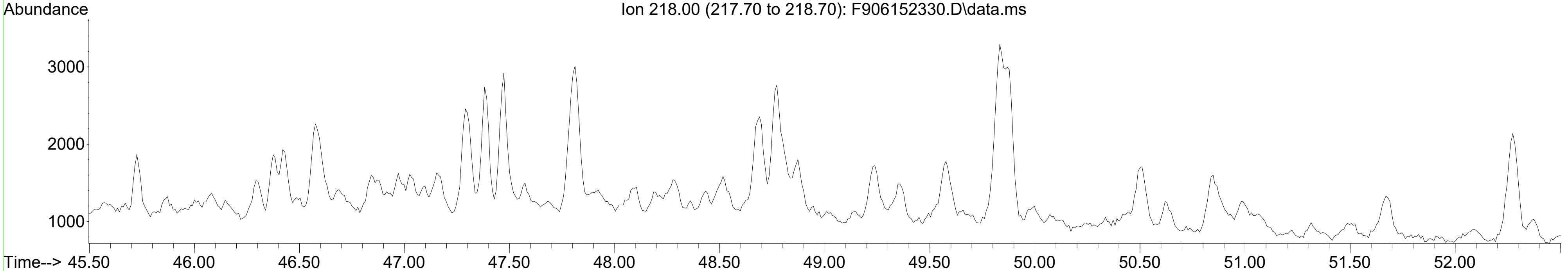
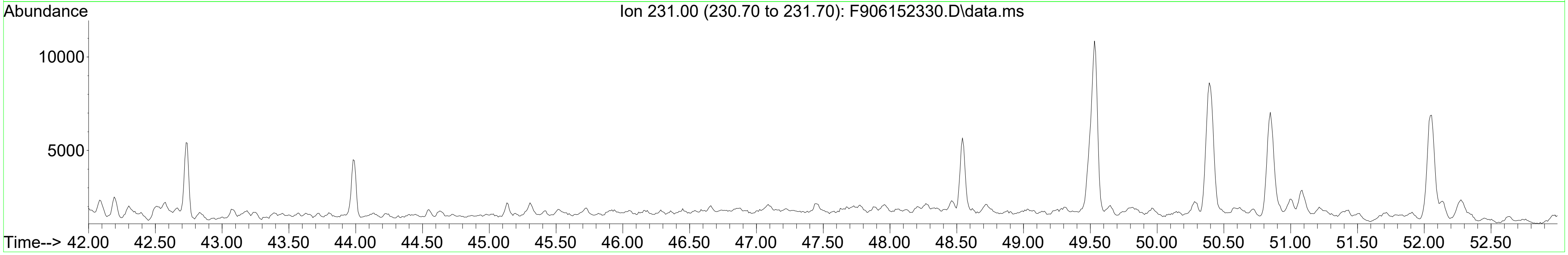
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152330.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023  12:16 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-01,32,,
Misc Info  : WG1793207,WG1791156,ICAL19995
```

209459
L2325505-01


Ion 191.00 (190.70 to 191.70): F906152330.D\data.ms


Ion 217.00 (216.70 to 217.70): F906152330.D\data.ms


Ion 218.00 (217.70 to 218.70): F906152330.D\data.ms


Ion 231.00 (230.70 to 231.70): F906152330.D\data.ms

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File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152331.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023   1:39 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-02,32,,
Misc Info   : WG1793207,WG1791156,ICAL19995
```
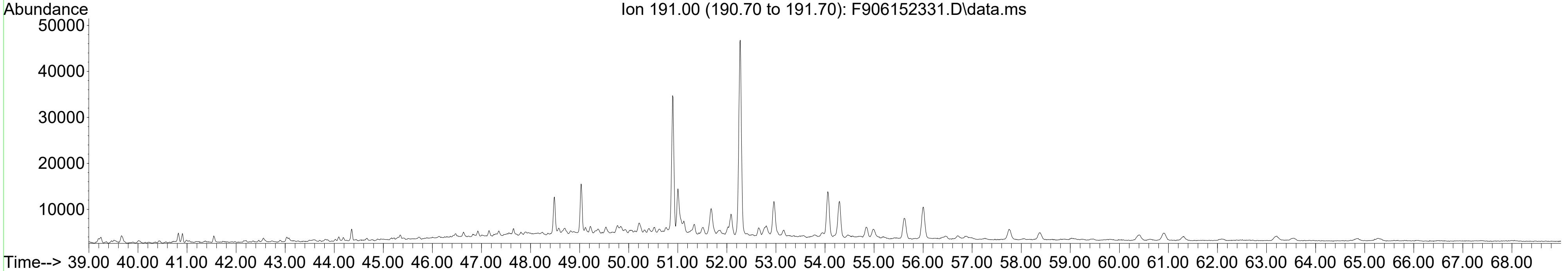
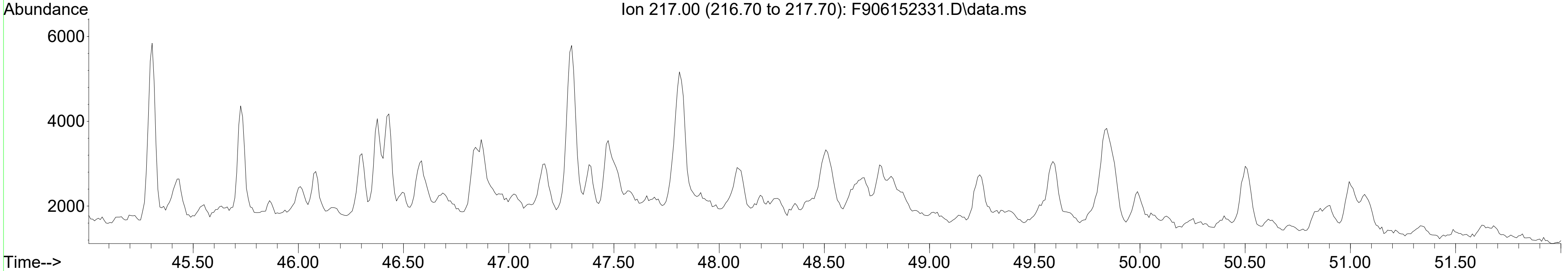
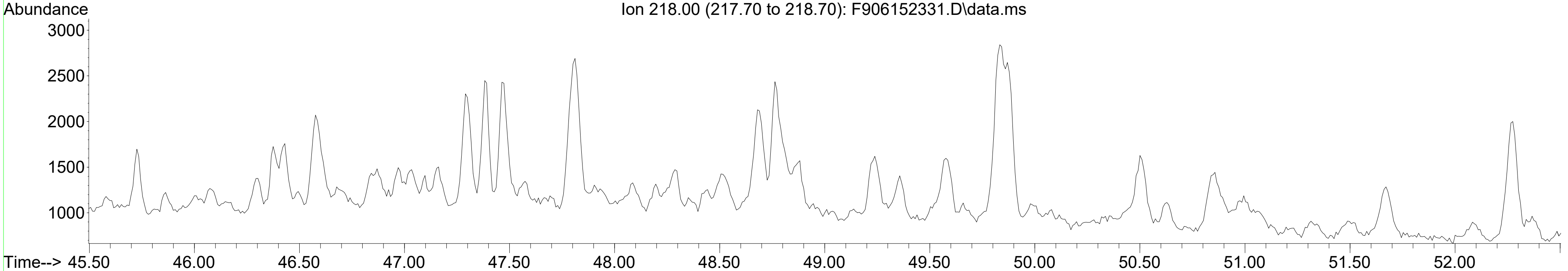
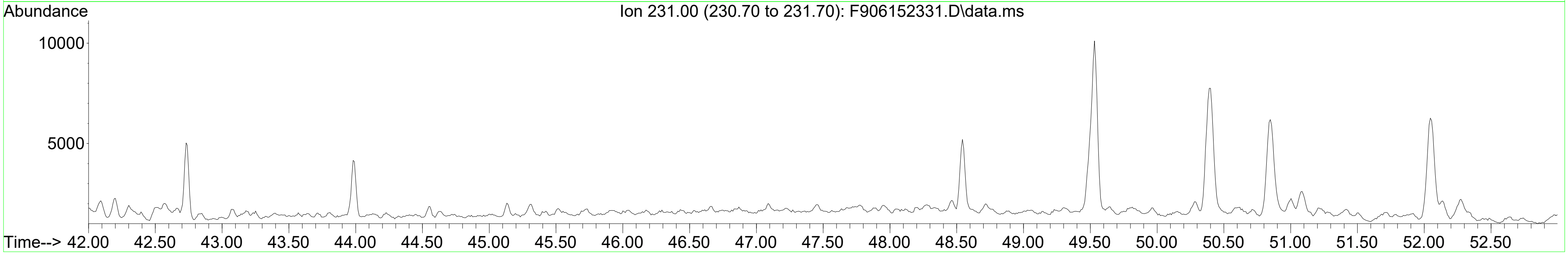

185997
L2325505-02









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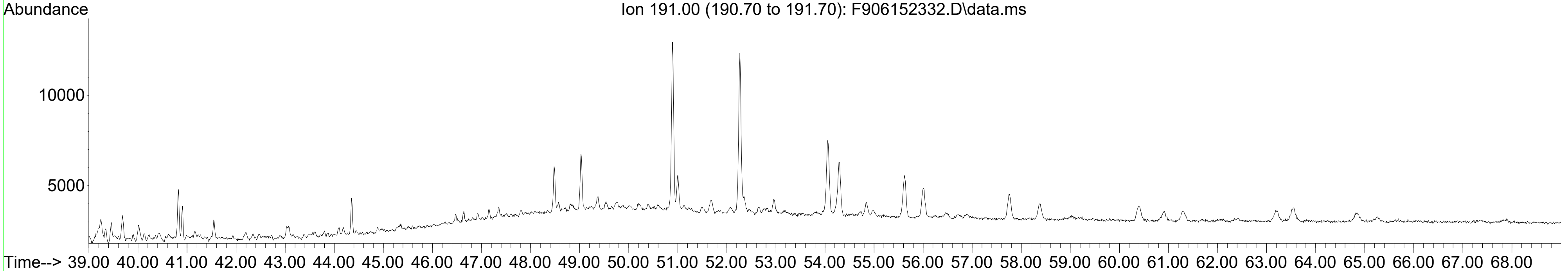
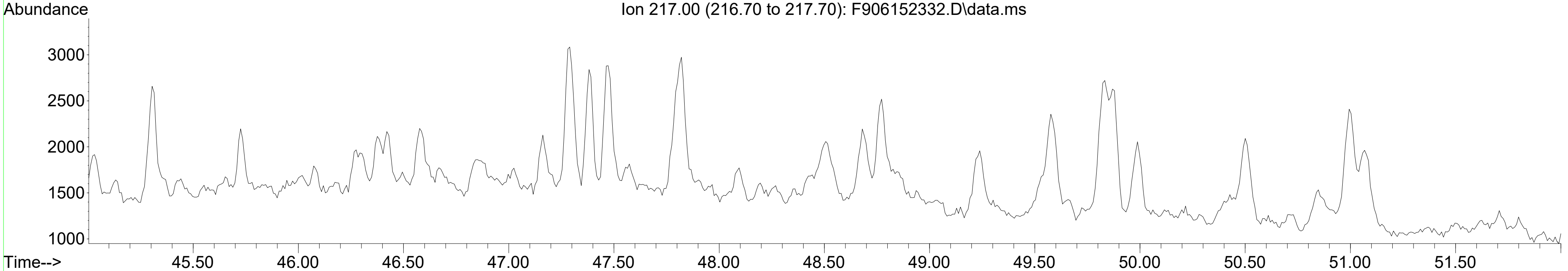
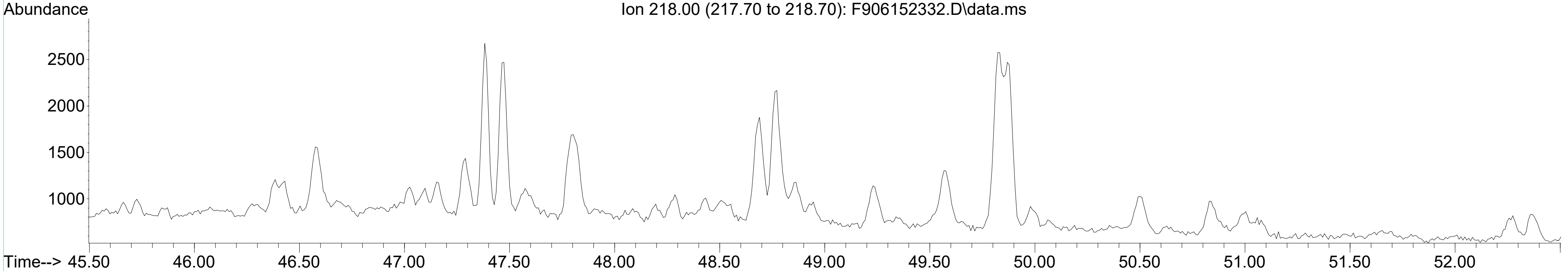
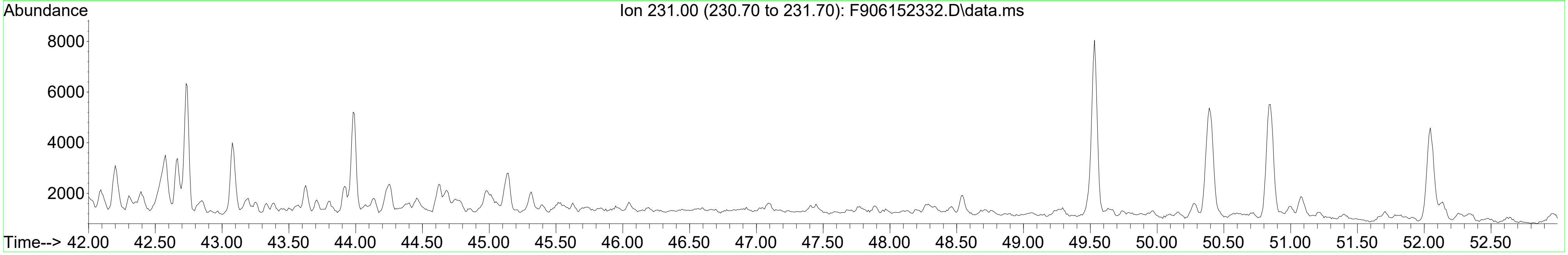
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152332.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   3:03 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-03,32,,
Misc Info  : WG1793207,WG1791156,ICAL19995
```

CAVERN 4
L2325505-03


Ion 191.00 (190.70 to 191.70): F906152332.D\data.ms

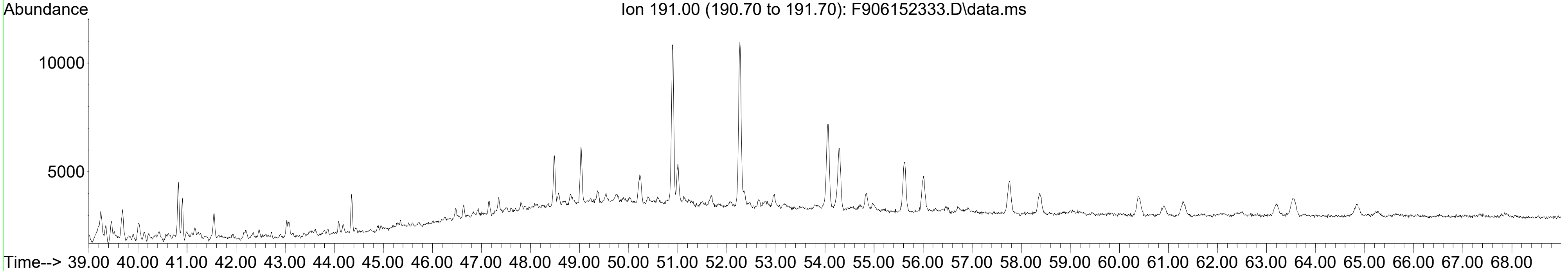

Ion 217.00 (216.70 to 217.70): F906152332.D\data.ms

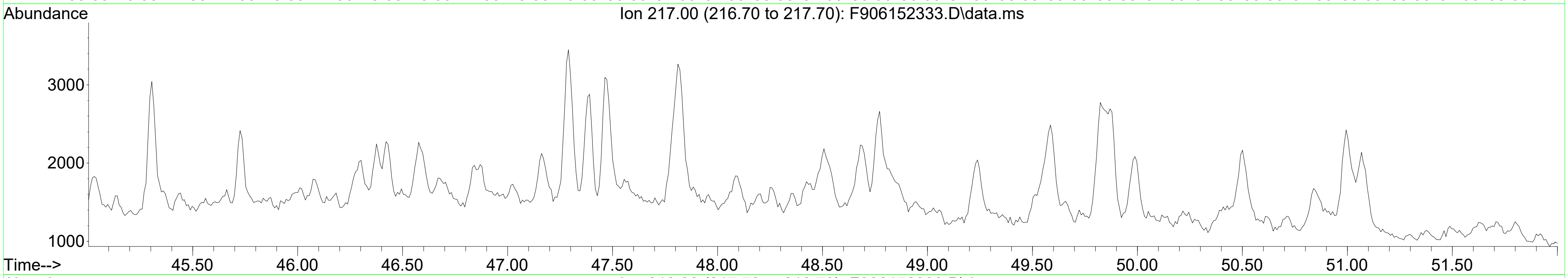

Ion 218.00 (217.70 to 218.70): F906152332.D\data.ms

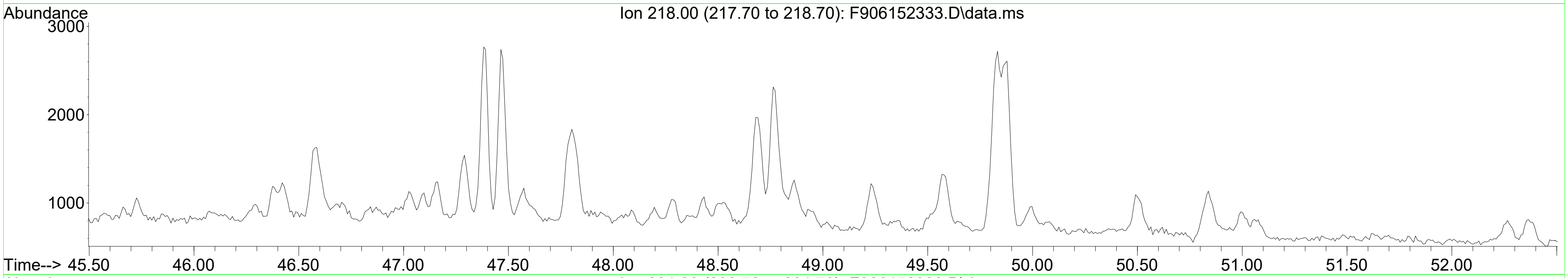

Ion 231.00 (230.70 to 231.70): F906152332.D\data.ms

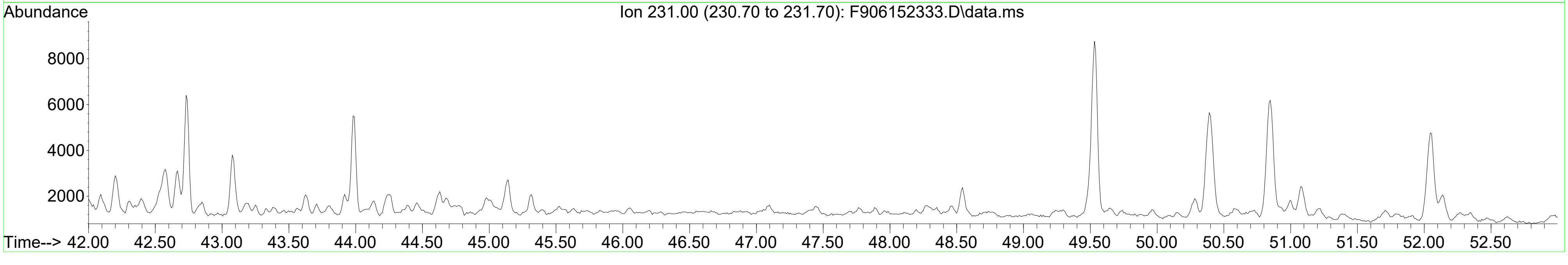
```
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152333.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023   4:27 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-04,32,,
Misc Info   : WG1793207,WG1791156,ICAL19995
```

CAVERN 7B
L2325505-04


Ion 191.00 (190.70 to 191.70): F906152333.D\data.ms

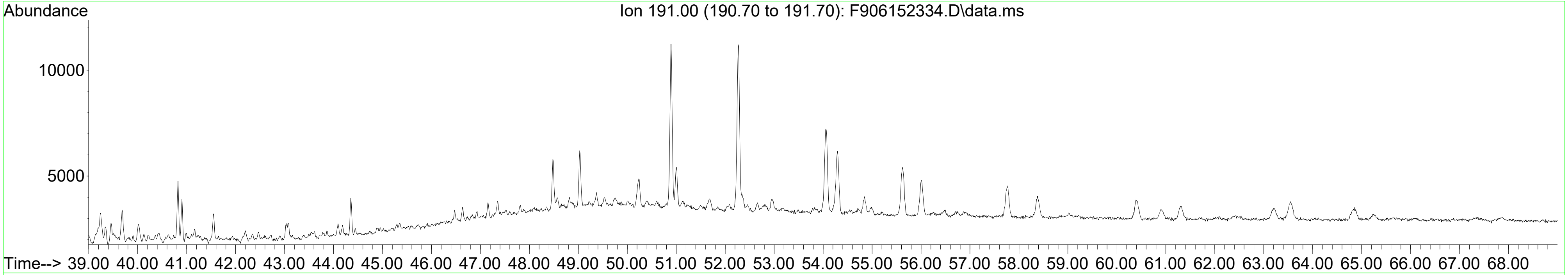

Ion 217.00 (216.70 to 217.70): F906152333.D\data.ms

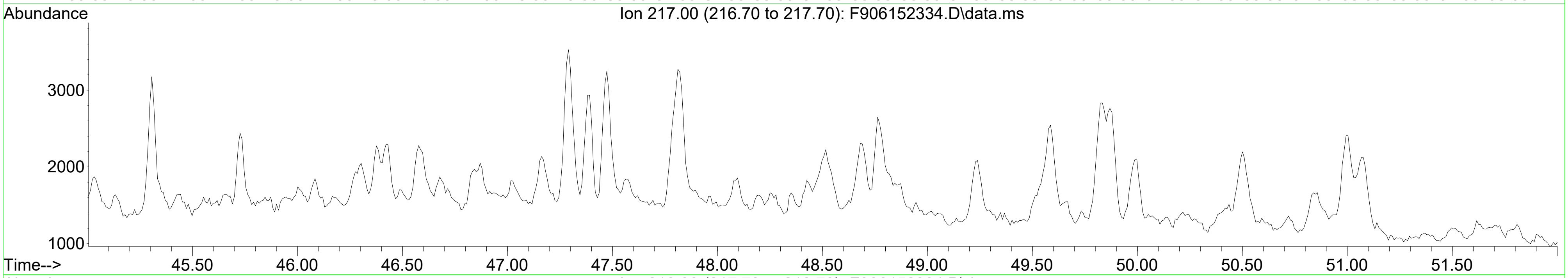

Ion 218.00 (217.70 to 218.70): F906152333.D\data.ms

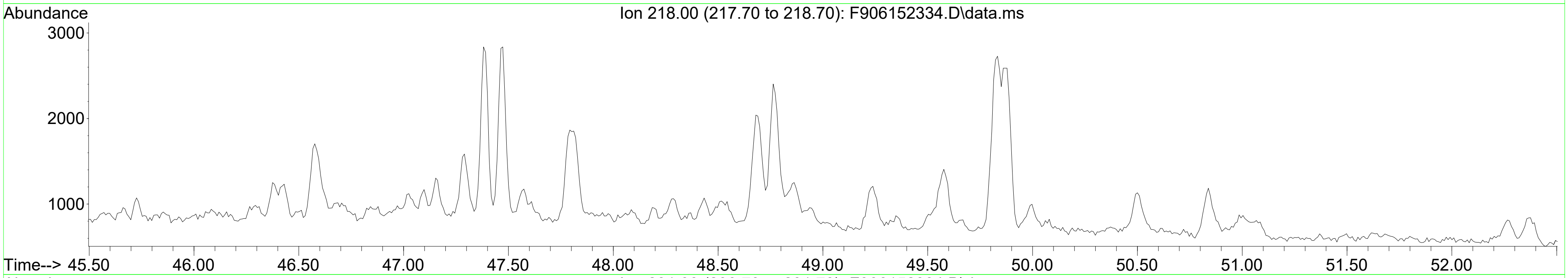

Ion 231.00 (230.70 to 231.70): F906152333.D\data.ms

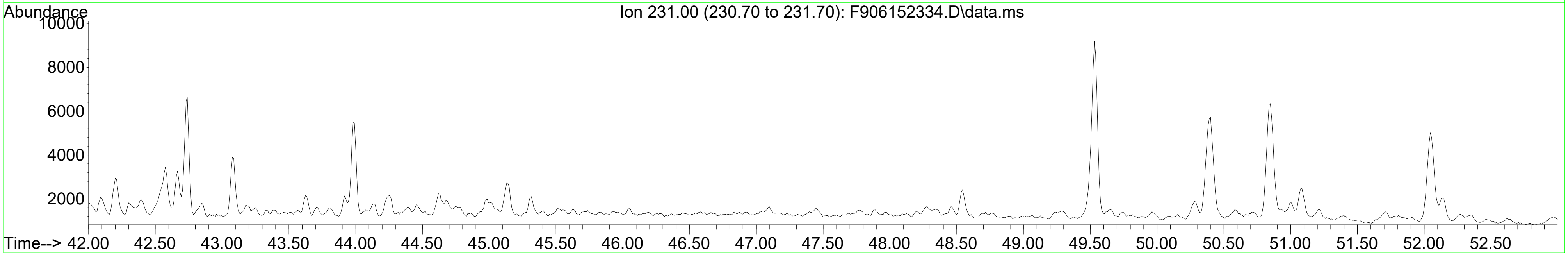
```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152334.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   5:50 pm using AcqMethod FRNC9ALT.M
Sample Name: WG1791156-4,32
Misc Info  : WG1793207,WG1791156,ICAL19995
```

CAVERN 7B Duplicate
WG1791156-4


Ion 191.00 (190.70 to 191.70): F906152334.D\data.ms

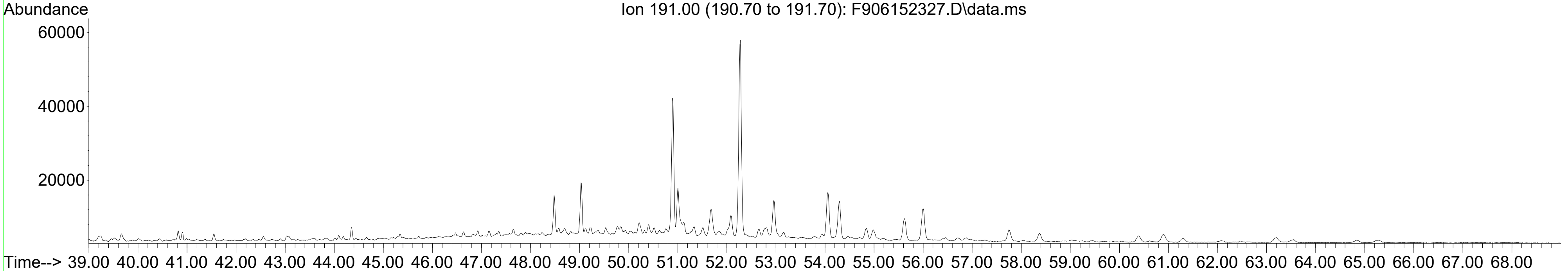

Ion 217.00 (216.70 to 217.70): F906152334.D\data.ms

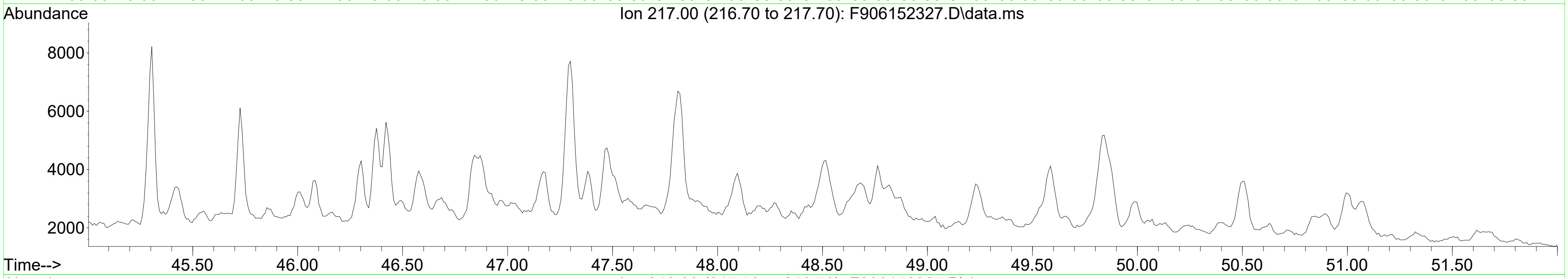

Ion 218.00 (217.70 to 218.70): F906152334.D\data.ms

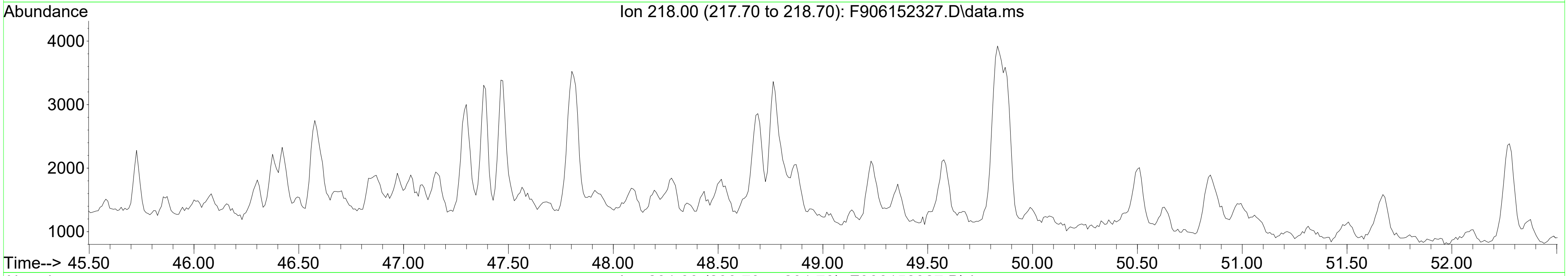

Ion 231.00 (230.70 to 231.70): F906152334.D\data.ms

```
File       :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...         KPAHBIO\F906152327.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   8:05 am using AcqMethod FRNC9ALT.M
Sample Name: L2325505-05,32
Misc Info  : WG1793207,WG1791156,ICAL19995
```

210185
L2325505-05


Ion 191.00 (190.70 to 191.70): F906152327.D\data.ms


Ion 217.00 (216.70 to 217.70): F906152327.D\data.ms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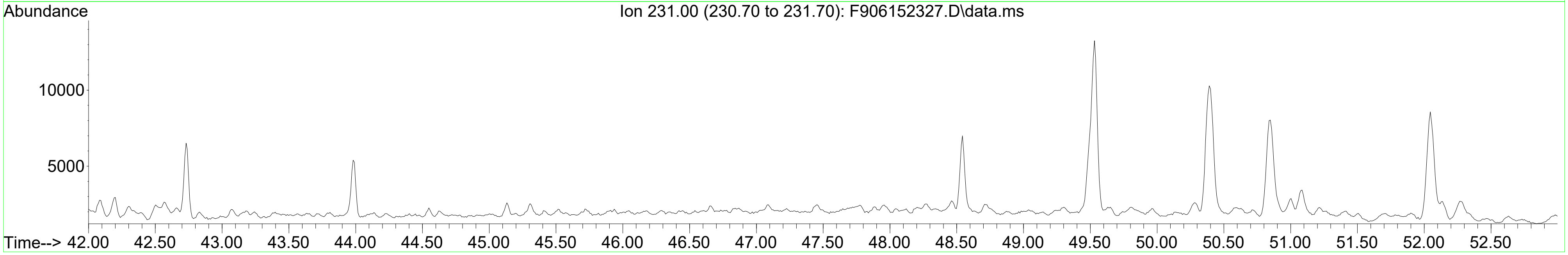
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...         KPAHBIO\F906152328.D
Operator   : PAH9:MJS
Instrument :    PAH9
Acquired   : 18 Jun 2023   9:28 am using AcqMethod FRNC9ALT.M
Sample Name: L2325505-06,32
Misc Info  : WG1793207,WG1791156,ICAL19995
```
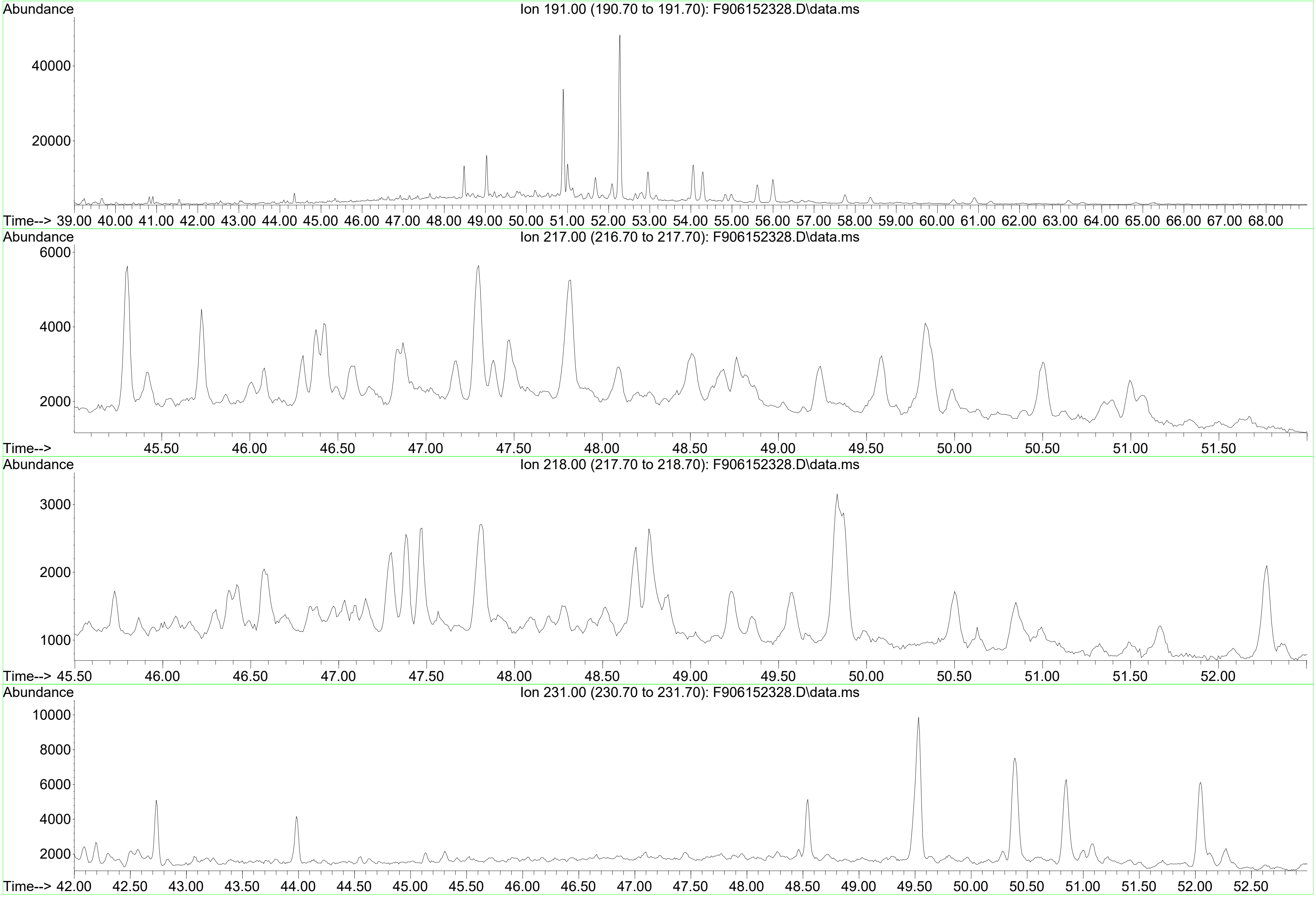

TANK BATTERY
L2325505-06



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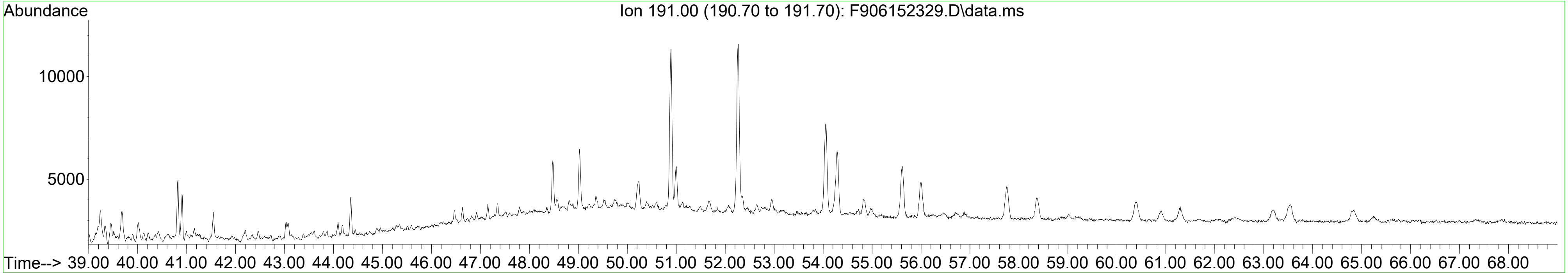
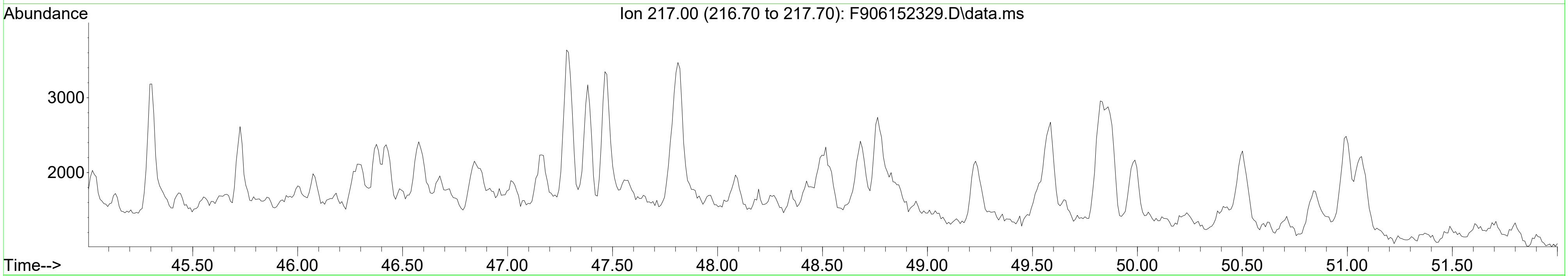
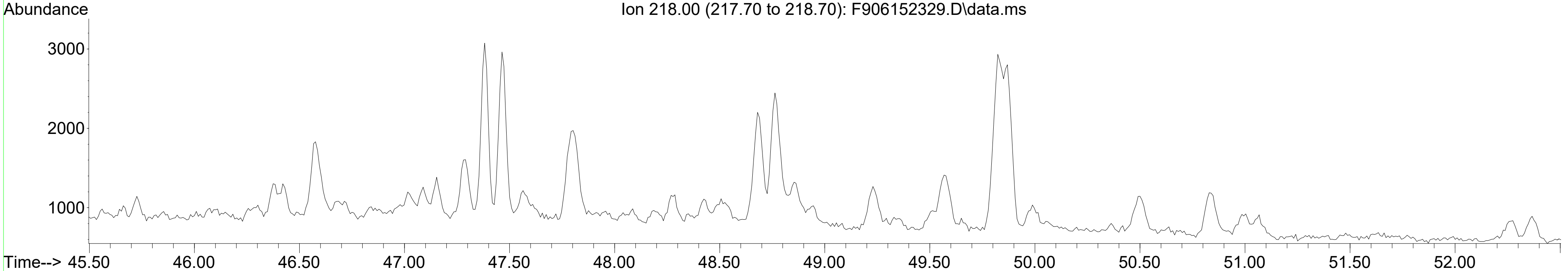
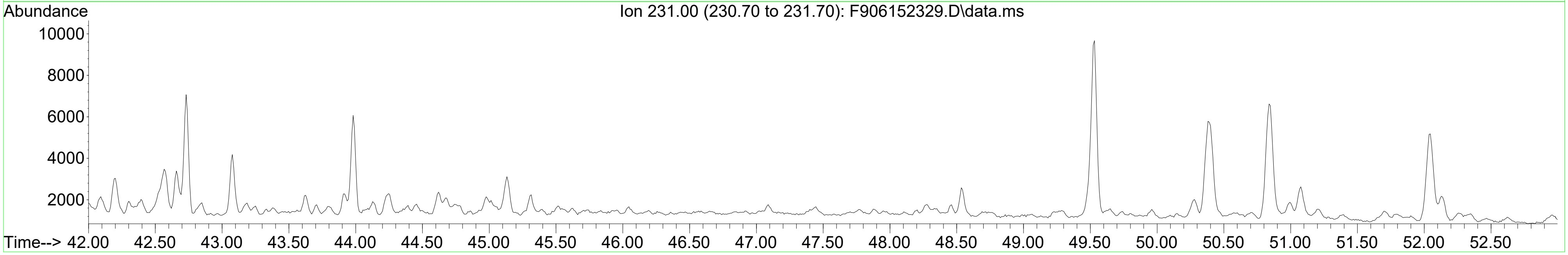
...          KPAHBIO\F906152329.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 18 Jun 2023  10:52 am using AcqMethod FRNC9ALT.M
Sample Name: L2325505-07,32
Misc Info   : WG1793207,WG1791156,ICAL19995
```

7B
L2325505-07
Reference Oil









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...          KPAHBIO\F906152309.D
Operator    : PAH9:MJS
Instrument  :    PAH9
Acquired    : 15 Jun 2023  10:33 pm using AcqMethod FRNC9ALT.M
Sample Name: L2325505-08,32,,
Misc Info   : WG1793207,WG1790071,ICAL19995
```
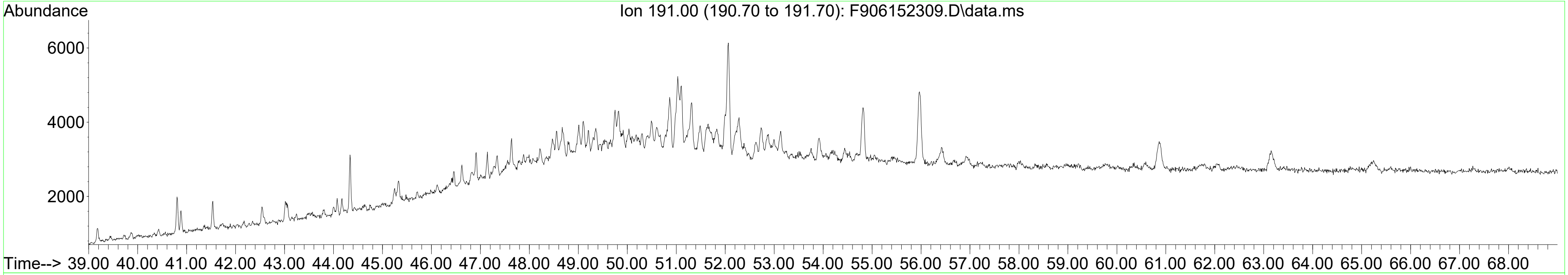

BS-24
L2325505-08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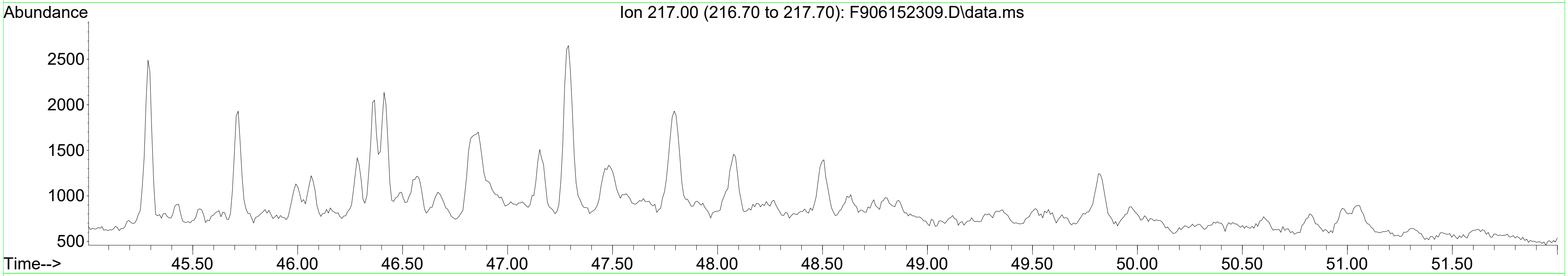
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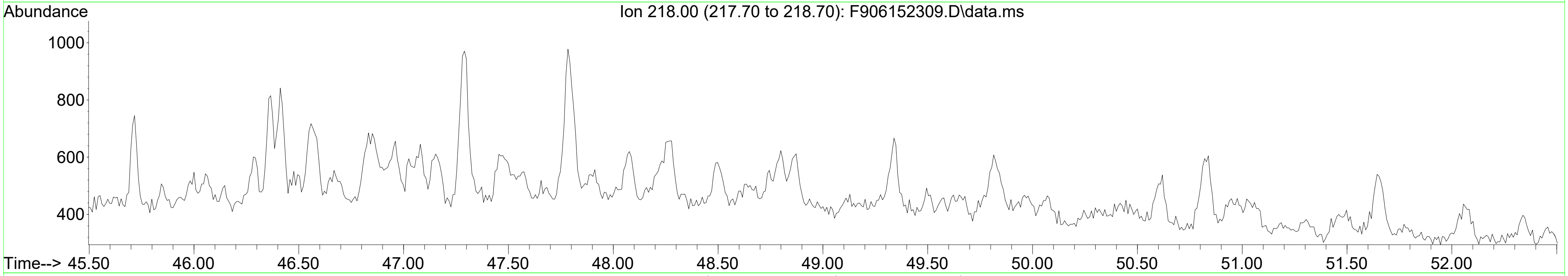
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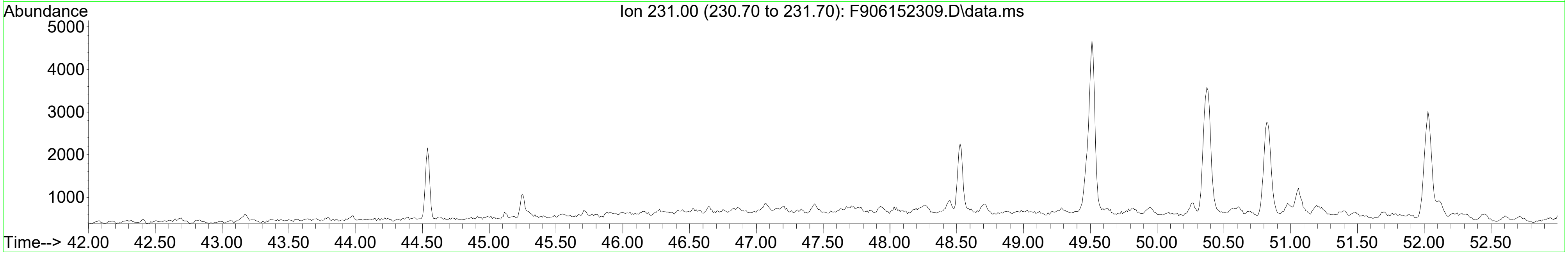
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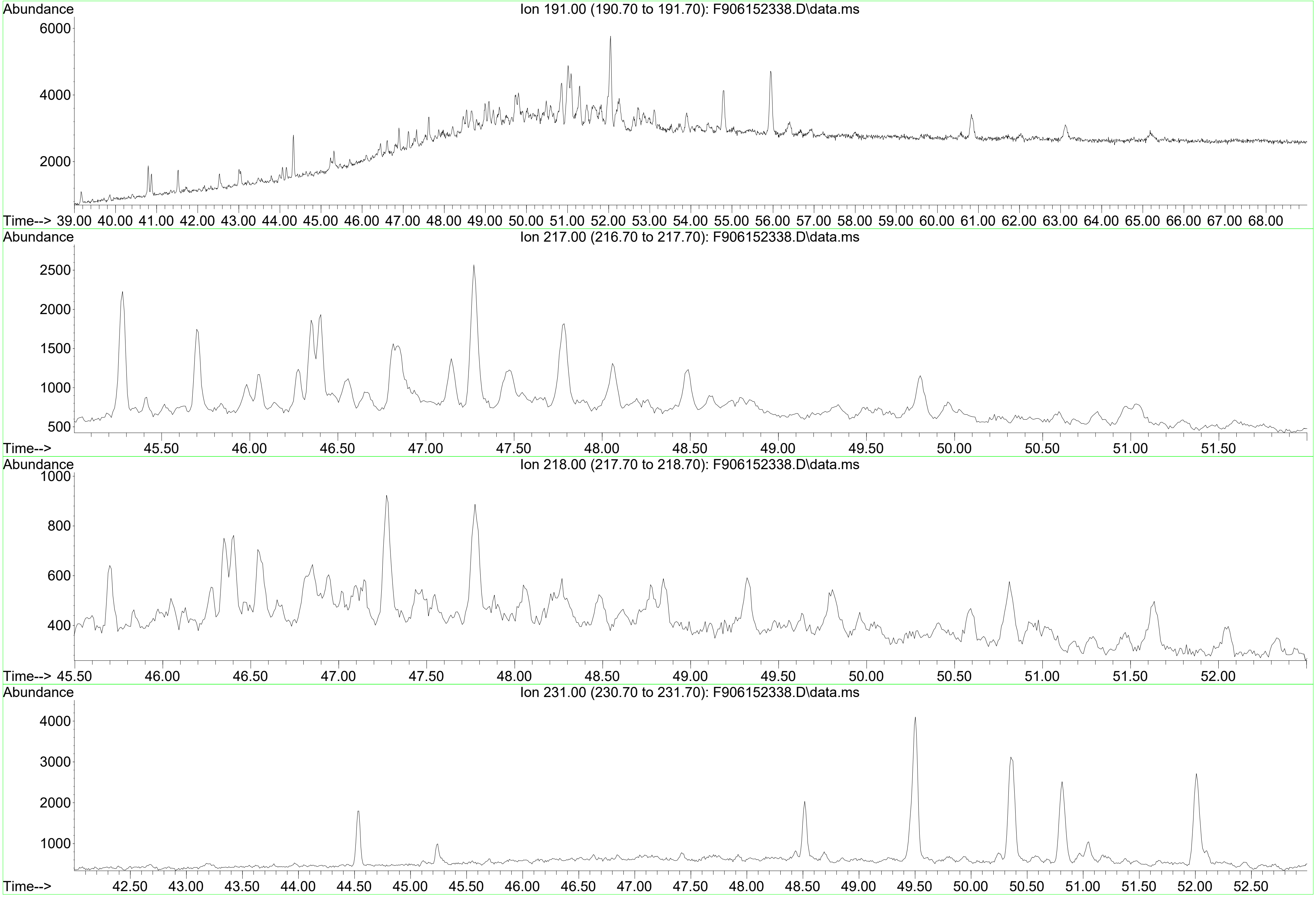
File        :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2325505\AL
...          KPAHBIO\F906152338.D
Operator    : PAH9:MJS
Instrument :    PAH9
Acquired    : 19 Jun 2023   1:13 pm using AcqMethod FRNC9ALT.M
Sample Name: l2325505-08,32,,
Misc Info   : WG1793207,WG1790071,ICAL19995

BS-24
L2325505-08
Post Copper Cleaned



# Attachment 7

# Crude Oil Assay Results

# Certificate of Analysis

Number: 1030-23060115-001A

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

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

June 19, 2023

Station Name: Cavern 4
Method: ASTM D-86
Analyzed: 06/06/2023 11:17:00 by FSN

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 10:15
Sample Conditions:

## ASTM D-86 Distillation

| % Recovery | °F @ 765 mm Hg |
|---|---|
| Initial Boiling Point | 176 |
| 5 | 234 |
| 10 | 286 |
| 20 | 376 |
| 30 | NR |
| 40 | NR |
| 50 | NR |
| 60 | NR |
| 70 | NR |
| 80 | NR |
| 85 | NR |
| 90 | NR |
| 95 | NR |
| Final Boiling Point | 400 |
| Volume % Recovery | 23.0 |
| Volume % Residue | 77.0 |
| Volume % Loss | 0.0 |

**Comments:** Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.1980

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

Number: 1030-23060115-001A

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

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

June 19, 2023

Station Name: Cavern 4
Sample Conditions:

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 10:15

## Analytical Data

| Test | Method | Result | Units | Detection Limit | Lab Tech. | Analysis Date |
|---|---|---|---|---|---|---|
| Salt in Crude Oil | ASTM D-3230 | 10.4 | lbs/1000 bbls | | MG | 06/06/2023 |
| Sulfur Content by X-ray | ASTM D-4294 | 1.548 | wt% | | MG | 06/07/2023 |
| Organic Chloride | ASTM D-4929 | <1.0 | ppmw | | FSN | 06/07/2023 |
| API Gravity @ 60.01 °F | ASTM D-5002 | 31.21 | ° | | MG | 06/07/2023 |
| Specific Gravity @ 60.01/60.01 °F | ASTM D-5002 | 0.8696 | _ | | MG | 06/07/2023 |
| Density @ 60.01 °F | ASTM D-5002 | 0.8688 | g/ml | | MG | 06/07/2023 |
| Nickel | ASTM D-5708A | 9 | ppmw | | CMN | 06/18/2023 |
| Vanadium | ASTM D-5708A | 42 | ppmw | | CMN | 06/18/2023 |
| Iron | ASTM D-5708A | 4 | ppmw | | CMN | 06/18/2023 |

**Comments:**
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.1980

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: 1030-23060115-002A

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

June 19, 2023

Station Name: Cavern 7B
Method: ASTM D-86
Analyzed: 06/06/2023 11:17:00 by FSN

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 10:45
Sample Conditions:

## ASTM D-86 Distillation

| % Recovery | °F @ 765 mm Hg |
|---|---|
| Initial Boiling Point | 138 |
| 5 | 214 |
| 10 | 264 |
| 20 | 350 |
| 30 | NR |
| 40 | NR |
| 50 | NR |
| 60 | NR |
| 70 | NR |
| 80 | NR |
| 85 | NR |
| 90 | NR |
| 95 | NR |
| Final Boiling Point | 400 |
| Volume % Recovery | 26.0 |
| Volume % Residue | 74.0 |
| Volume % Loss | 0.0 |

**Comments:** Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.2288

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: 1030-23060115-002A

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

June 19, 2023

Station Name: Cavern 7B
Sample Conditions:

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 10:45

## Analytical Data

| Test | Method | Result | Units | Detection Limit | Lab Tech. | Analysis Date |
|---|---|---|---|---|---|---|
| Salt in Crude Oil | ASTM D-3230 | 5.0 | lbs/1000 bbls | | MG | 06/06/2023 |
| Sulfur Content by X-ray | ASTM D-4294 | 1.401 | wt% | | MG | 06/07/2023 |
| Organic Chloride | ASTM D-4929 | <1.0 | ppmw | | FSN | 06/07/2023 |
| API Gravity @ 60.01 °F | ASTM D-5002 | 33.52 | ° | | FSN | 06/19/2023 |
| Specific Gravity @ 60.01/60.01 °F | ASTM D-5002 | 0.8575 | _ | | FSN | 06/19/2023 |
| Density @ 60.01 °F | ASTM D-5002 | 0.8566 | g/ml | | FSN | 06/19/2023 |
| Nickel | ASTM D-5708A | 6 | ppmw | | CMN | 06/18/2023 |
| Vanadium | ASTM D-5708A | 23 | ppmw | | CMN | 06/18/2023 |
| Iron | ASTM D-5708A | <1 | ppmw | | CMN | 06/18/2023 |

**Comments:**
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.2288
AS-D-5002: RERUN

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

Number: 1030-23060115-003A

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

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

June 19, 2023

| | | | |
|---|---|---|---|
| Station Name: | 210185 | Sampled By: | DS |
| Method: | ASTM D-86 | Sample Of: | Liquid Spot |
| Analyzed: | 06/06/2023 17:51:00 by FSN | Sample Date: | 05/25/2023 11:10 |
| | | Sample Conditions: | |

## ASTM D-86 Distillation

| % Recovery | °F @ 764 mm Hg |
|---|---|
| Initial Boiling Point | 206 |
| 5 | 438 |
| 10 | 500 |
| 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 | 10.0 |
| Volume % Residue | 90.0 |
| Volume % Loss | 0.0 |

**Comments:** Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.0899

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.

SPL

# Certificate of Analysis

Number: 1030-23060115-003A

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

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

June 19, 2023

Station Name: 210185
Sample Conditions:

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 11:10

## Analytical Data

| Test | Method | Result | Units | Detection Limit | Lab Tech. | Analysis Date |
|---|---|---|---|---|---|---|
| Salt in Crude Oil | ASTM D-3230 | 9.8 | lbs/1000 bbls |  | MG | 06/06/2023 |
| Sulfur Content by X-ray | ASTM D-4294 | 0.476 | wt% |  | MG | 06/07/2023 |
| Organic Chloride | ASTM D-4929 | <1.0 | ppmw |  | FSN | 06/07/2023 |
| API Gravity @ 60.01 °F | ASTM D-5002 | 22.79 | ° |  | MG | 06/07/2023 |
| Specific Gravity @ 60.01/60.01 °F | ASTM D-5002 | 0.9171 | _ |  | MG | 06/07/2023 |
| Density @ 60.01 °F | ASTM D-5002 | 0.9162 | g/ml |  | MG | 06/07/2023 |
| Nickel | ASTM D-5708A | 10 | ppmw |  | CMN | 06/18/2023 |
| Vanadium | ASTM D-5708A | 2 | ppmw |  | CMN | 06/18/2023 |
| Iron | ASTM D-5708A | 59 | ppmw |  | CMN | 06/18/2023 |

**Comments:**
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.0899

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

Number: 1030-23060115-004A

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

June 19, 2023

Station Name: Tank Battery
Method: ASTM D-86
Analyzed: 06/06/2023 17:51:00 by FSN

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 12:45
Sample Conditions:

## ASTM D-86 Distillation

| % Recovery | °F @ 764 mm Hg |
|---|---|
| Initial Boiling Point | 226 |
| 5 | 318 |
| 10 | 398 |
| 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 | 10.0 |
| Volume % Residue | 90.0 |
| Volume % Loss | 0.0 |

**Comments:** Temperatures are uncorrected for barometric pressure.
Visual color is Crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.0909

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: 1030-23060115-004A

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

June 19, 2023

Station Name: Tank Battery
Sample Conditions:

Sampled By: DS
Sample Of: Liquid Spot
Sample Date: 05/25/2023 12:45

## Analytical Data

| Test | Method | Result | Units | Detection Limit | Lab Tech. | Analysis Date |
|---|---|---|---|---|---|---|
| Salt in Crude Oil | ASTM D-3230 | 32.0 | lbs/1000 bbls |  | MG | 06/06/2023 |
| Sulfur Content by X-ray | ASTM D-4294 | 0.327 | wt% |  | MG | 06/07/2023 |
| Organic Chloride | ASTM D-4929 | <1.0 | ppmw |  | FSN | 06/07/2023 |
| API Gravity @ 60.01 °F | ASTM D-5002 | 26.95 | ° |  | MG | 06/07/2023 |
| Specific Gravity @ 60.01/60.01 °F | ASTM D-5002 | 0.8930 | _ |  | MG | 06/07/2023 |
| Density @ 60.01 °F | ASTM D-5002 | 0.8921 | g/ml |  | MG | 06/07/2023 |
| Nickel | ASTM D-5708A | 6 | ppmw |  | CMN | 06/18/2023 |
| Vanadium | ASTM D-5708A | 1 | ppmw |  | CMN | 06/18/2023 |
| Iron | ASTM D-5708A | 15 | ppmw |  | CMN | 06/18/2023 |

**Comments:**
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.0909

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.

## 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
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.: 0688077
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.

JUN 0 5 2023

**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-86-IBP-400°F | AS-D-4929 | | | | | | | Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cavern 4 | 05/25/23 | 10:15 | Liq | | | X | | | | X | X | X | X | X | X | | | | | | | |
| Cavern 7B | 05/25/23 | 10:45 | Liq | | | X | | | | X | X | X | X | X | X | | | | | | | |
| 210185 | 05/25/23 | 11:10 | Liq | | | X | | | | X | X | X | X | X | X | | | | | | | |
| Tank Battery | 05/25/23 | 12:45 | Liq | | | X | | | | X | X | X | X | X | X | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | |
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| | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | |

Sampled By-Print Name: David Sanguinetti
Signature:
Received By-Company:

Relinquished By-Print Name: David Sanguinetti
Signature:
Date: 5/6/23
Time:
Received By-Print Name: Hanah Baki
Signature:
Date:
Time:

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

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

**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.

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

# Certificate of Analysis

Number: 1030-23050143-001A

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

May 19, 2023

Station Name: 209459
Method: ASTM D-86
Analyzed: 05/15/2023 00:00:00 by FSN

Sampled By:
Sample Of: Liquid Spot
Sample Date: 05/02/2023 10:45
Sample Conditions:

## ASTM D-86 Distillation

| **% Recovery** | **°F @ 770 mm Hg** |
|---|---|
| Initial Boiling Point | 306 |
| 5 | 446 |
| 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 | 9.0 |
| Volume % Residue | 91.0 |
| Volume % Loss | 0.0 |

**Comments:** Visual color is crude.
IBP to 400°F Naphtha Cut Mass Fraction = 0.0902

Michael Staley

Data reviewed by: Michael Staley, ASTM Manager

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

# Certificate of Analysis

Number: 1030-23050143-001A

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

May 19, 2023

Station Name: 209459
Sample Conditions:

Sampled By:
Sample Of: Liquid Spot
Sample Date: 05/02/2023 10:45

## Analytical Data

| Test | Method | Result | Units | Detection Limit | Lab Tech. | Analysis Date |
|---|---|---|---|---|---|---|
| Salt in Crude Oil | ASTM D-3230 | 1290.0 | lbs/1000 bbls | | ES | 05/18/2023 |
| Sulfur Content by X-ray | ASTM D-4294 | 0.435 | wt% | | CMN | 05/16/2023 |
| Organic Chloride | ASTM D-4929 | <1.0 | ppmw | | FSN | 05/17/2023 |
| API Gravity @ 60.01 °F | ASTM D-5002 | 22.81 | ° | | DKK | 05/18/2023 |
| Specific Gravity @ 60.01/60.01 °F | ASTM D-5002 | 0.9170 | _ | | DKK | 05/18/2023 |
| Density @ 60.01 °F | ASTM D-5002 | 0.9161 | g/ml | | DKK | 05/18/2023 |
| Nickel | ASTM D-5708A | 8 | ppmw | | CMN | 05/09/2023 |
| Vanadium | ASTM D-5708A | 2 | ppmw | | CMN | 05/09/2023 |
| Iron | ASTM D-5708A | 13 | ppmw | | CMN | 05/09/2023 |

**Comments:**
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.0902

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: 1030-23050143-002A

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

May 19, 2023

Station Name: 185997
Method: ASTM D-86
Analyzed: 05/15/2023 00:00:00 by FSN

Sampled By:
Sample Of: Liquid Spot
Sample Date: 05/02/2023 11:30
Sample Conditions:

## ASTM D-86 Distillation

| % Recovery | °F @ 767 mm Hg |
|---|---|
| Initial Boiling Point | 202 |
| 5 | 474 |
| 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 | 7.0 |
| Volume % Residue | 93.0 |
| Volume % Loss | 0.0 |

**Comments:** IBP to 400°F Naphtha Cut Mass Fraction = 0.0663
Visual color is crude.

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

Number: 1030-23050143-002A

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

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

May 19, 2023

Station Name: 185997
Sample Conditions:

Sampled By:
Sample Of: Liquid Spot
Sample Date: 05/02/2023 11:30

## Analytical Data

| Test | Method | Result | Units | Detection Limit | Lab Tech. | Analysis Date |
|---|---|---|---|---|---|---|
| Salt in Crude Oil | ASTM D-3230 | 1015.0 | lbs/1000 bbls | | ES | 05/18/2023 |
| Sulfur Content by X-ray | ASTM D-4294 | 0.407 | wt% | | CMN | 05/16/2023 |
| Organic Chloride | ASTM D-4929 | <1.0 | ppmw | | FSN | 05/17/2023 |
| API Gravity @ 60.01 °F | ASTM D-5002 | 21.53 | ° | | DKK | 05/18/2023 |
| Specific Gravity @ 60.01/60.01 °F | ASTM D-5002 | 0.9246 | _ | | DKK | 05/18/2023 |
| Density @ 60.01 °F | ASTM D-5002 | 0.9237 | g/ml | | DKK | 05/18/2023 |
| Nickel | ASTM D-5708A | 9 | ppmw | | CMN | 05/09/2023 |
| Vanadium | ASTM D-5708A | 2 | ppmw | | CMN | 05/09/2023 |
| Iron | ASTM D-5708A | 6 | ppmw | | CMN | 05/09/2023 |

**Comments:**
AS-D-4929: Sample analyzed by ASTM D-4929 procedure B.
Mass Fraction = 0.0663

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.

In-person

# SPL, Inc.
## Analysis Request Chain of Custody Record

| | | | | |
|---|---|---|---|---|
| SPL Work Order No.: | Acct. M[illegible] Code: | Dept. Code | Page<br>1 | Pages<br>1 |

| | | | | |
|---|---|---|---|---|
| **Report To: (Company Name):** ERM | Project/Station **Name**: | Project/Station **Number**: | Project/Station **Location**: | **Requested TAT*** |
| Address: 840 W. Sam Houston Parkway North<br>Suite 600 | Special Instructions: | | | |
| City/State/Zip: Houston TX 77024-4613 | | | | **10 business days** |
| Contact: Scott Himes Scott.Himes@erm.com | Indicate Billing Type: (Place "**X**", where appropriate) | Net 30 day Acct. | Check # | |
| Phone: 832-209-8811 Fax: | | 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.:
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-86-IBP-400°F** | AS-D-4929 | | | | | | | Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 209459 | 5/2/23 | 1045 | Liq | | | X | | | | X | X | X | X | X | X | | | | | | | |
| 185997 | 5/2/23 | 1130 | Liq | | | X | | | | X | X | X | X | X | X | | | | | | | |
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|---|---|---|---|---|---|
| Sampled By-Print Name:<br>Signature: | | | Received By-Company: | | |
| Relinquished By-Print Name:<br>Signature: | Date: | Time: | Received By-Print Name:<br>Signature: | Date:<br>05/03/23 | Time: |
| Relinquished By-Print Name:<br>Signature: | Date: | Time: | Received By-Print Name:<br>Signature: | Date: | Time: |
| Relinquished By-Print Name:<br>Signature: | Date: | Time: | Received By-Print Name:<br>Signature: | Date: | Time: |

| | | | | | |
|---|---|---|---|---|---|
| **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.