March 25, 2024

Troy Charpentier
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Kean Miller LLP
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Baton Rouge, Louisiana 70802

Chemical Fingerprint of Surface Oil near Bubble Site 24 – February 26, 2024
Westlake Sulphur Dome Study

Dear Mr. Charpentier,

NewFields is pleased to provide you with this report of chemical fingerprinting results for a sheen sample collected on February 26, 2024 as part of the on-going investigation of the Westlake US 2 LLC (Westlake) salt dome caverns in the Sulphur Mines oil field, Calcasieu Parish, Louisiana (the Site).

This study follows 10 earlier chemical fingerprinting studies at the Site (**Table 1**). These earlier studies included five oils from the 7B cavern well (collected between January and October 2023), 13 crude oil samples from nine Yellow Rock wells and the Yellow Rock tank battery. Among other conclusions, these earlier studies showed:

- The 7B cavern oils are chemically distinct from the locally produced (Yellow Rock) crude oils studied, which varied only slightly among themselves.
- There was no change in composition of the 7B cavern oil between January 2023 and October 2023, indicating no local crude oil(s) had or was presently entering Cavern 7.

Several earlier studies also included nine floating surface oils/sheens/materials from six surface locations (Bubble Sites No. 14, 19, 20, 22, and 24 and Central Lake) between January 2023 and October 2023, which showed:

- Any oil present in the surface samples was derived from locally produced crude oil(s), not 7B cavern oil.

The present study provides results on a surface oil sample collected near Bubble Site 24 on February 26, 2024. I understand that there were no actual (gas) bubbles observed but that floating liquid oil was present at the marsh surface. I further understand the sample was collected at the request of LDNR.

Samples

Table 2 provides an inventory that includes the surface oil sample from this study – along with those previously studied for ease of reference. As noted above, the bubble site oil studied was collected on February 26, 2024 by personnel from ERM. A sufficient volume of floating oil was present so that the oil could be collected directly into a glass sample jar. The sample was sent to NewFields' alliance laboratory, Alpha Analytical (Alpha; Mansfield, Massachusetts, USA), on

February 28, 2024 where it arrived safely the next day. A copy of the chain-of-custody document received with the shipment is found in **Attachment 1**.

Objective

The objective of the current study was to determine the character of the surface oil near Bubble Site 24 collected February 26, 2024 and compare it to oils recovered from Cavern 7 (7B cavern oil), locally produced oils from nearby Yellow Rock wells, and previously studied surface oils/sheens. Of specific concern was whether the oil present in the surface oil sample may be derived from recent or active seepage of subsurface oil from Cavern 7 and/or local reservoirs/wells.

Methods

This objective was pursued using specific chemical fingerprinting analyses and interpretation protocols employed in oil spill identification studies,[1] as were described in the previous reports and their attachments.[2]

As first described in the study of May 2023 oils (Report 4, Table 1), this study also included the (re-)analysis of the cavern 7B oil collected in January 2023, that was adopted as a *site-specific reference oil*. This oil is being re-analyzed for quality control with each "batch" of samples analyzed from the Sulphur Dome site to assess the long-term precision of diagnostic ratios (DRs) used in the quantitative (statistical) comparison of samples from the site. The results obtained herein for the site-specific reference oil yielded only very minor revisions to the short- and long-term relative standard deviation values (RSD_r and RSD_R) for the 30 DRs assessed throughout the on-going study. As in earlier studies, the updated RSD values identified 17 DRs with the most precisely measured DRs are presented in **Attachment 2** herein. (See Attachment 3 within Report 6 for additional discussion of short- and long-term precision.)

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 L2410930). The tabulated results for the targeted compounds in each analysis performed are contained in **Attachment 3**. The full-size GC/FID chromatogram obtained in Tier 1 (modified EPA Method 8015D) analysis is provided in **Attachment 4** and selected extraction ion profiles (EIPs) obtained in Tier 2 (modified EPA Method 8270D) are provided in **Attachment 5**.

Specific results most relevant to the study's objectives are presented in **Table 3 and Figures 1 to 5**. Discussion of these results is provided in the following sections.

<u>Tier 1 – General Character of the Surface Oil near Bubble Site 24</u>

Figure 1 shows the GC/FID (C8+) chromatogram for the surface oil collected near Bubble Site 24 studied herein (Fig. 1A) and the 7B cavern (reference) oil re-analyzed herein (Fig. 1B), which are described in the following paragraphs.

The chromatogram for the surface oil collected near Bubble Site 24 studied is comprised of a broad "hump" referred to as an unresolved complex mixture (UCM). The UCM spans from ~C12 to C45 with no resolved peaks characteristic of n-alkanes, acyclic isoprenoids (Pr or Ph), or

[1] 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.

[2] See Attachments 2 and 3, Reports 1, 3, 4, 5, and 6 (per Table 1).

prominent biomarkers (e.g., hopane or norhopane; Fig. 1A). The only resolved peaks present appear to be some (apparently recalcitrant) biomarkers (e.g., diasteranes and aromatic steroids). These Tier 1 features indicate that:

- The surface oil collected near Bubble Site 24 on February 26, 2024 is comprised of a severely biodegraded crude oil.

Oppositely, and unsurprisingly based upon all previous analyses of this same 7B cavern (reference) oil, the 7B cavern oil studied herein has a chromatogram showing that it is comprised of unweathered crude oil (Fig. 1B), i.e., very different from the surface oil collected near Bubble Site 24 studied herein.

Tier 2 – Detailed Character/Comparison of the Surface Oil near Bubble Site 24

As in the earlier Sulphur Dome chemical fingerprinting studies conducted by NewFields, diagnostic (source-specific and weathering resistant) features/ratios among PAHs, sulfur-containing aromatics, and petroleum biomarkers based on Tier 2's GC/MS results were used herein to qualitatively and quantitatively reveal detailed characteristics of the crude oil comprising the surface oil collected near Bubble Site 24 sample studied herein.

Figure 2 shows the *m/z* 191 extraction ion profiles (EIPs) for surface oil near Bubble Site 24 collected February 26, 2024 (Fig. 2A), the 7B cavern (reference) oil re-analyzed herein (Fig. 2B), and a typical, locally produced crude oil as represented by oil from the Yellow Rock well 185997 (Fig. 2C; previously reported in Report 5, Table 1).

The most obvious observation in Figure 2 is that the surface oil near Bubble Site 24 studied herein (Fig. 2A) exhibits a very different distribution of terpenoid biomarkers compared to the other oil samples (Fig. 2B-C). The surface oil contains a relative abundance of tricyclic terpanes (T4-T10), tetracyclic terpane (T6a), oleanane (T18), norneohopane (T16), diahopane (T17) and an overall lack of hopanes (T11, T12, T15, T19, T21-T35; Fig. 2A). These features are evidence that the surface oil near Bubble Site 24 studied herein is so severely biodegraded that the hopanes have been biodegraded thereby allowing the more resistant terpenoids to predominate (Fig. 2A).[3] Also predominating is the 22R-bishomohopane epimer (T27; Fig. 2A), which was previously recognized as an interfering (apparent) marker compound common to all locally produced crude oils studied to date (e.g., see Fig. 2C). Although presently unidentified, this marker compound appears resistant to the severe biodegradation experienced by the surface oil near Bubble Site 24 studied herein (Fig. 2A).

As has been previously established in multiple earlier reports, the locally produced crude oils exhibit a lower relative abundance of tricyclic terpanes (T4-T10), bisnorhopane (T14a), norhopane (T15), and homohopanes (T21 to T35) and higher relative abundance of oleanane (T19) and moretanes (T17 and T20; Fig. 2C) compared to the 7B cavern oil (Fig. 2B).

Among other features, the prominence of oleanane (T18) in the severely biodegraded surface oil near Bubble Site 24 studied herein (Fig. 2A) is evidence that it must be derived from a locally produced oil (which universally contain oleanane; e.g., Fig. 2C) that has been more severely

[3] For example: Oleanane and tricyclic terpanes are considered more resistant to biodegradation than hopanes [thus, T18/T19 and (T7 to T10)/T19 DRs (Fig. 4D and 4F) may increase; e.g., Peters et al. (2004), *The Biomarker Guide*]. Notably, 25-norhopanes are not present in the surface oil near Bubble Site 24 studied herein. These somewhat unusual compounds were only found in one of the locally produced crude oils studied to date; Yellow Rock 253998; see Report 6 (per Table 1).

biodegraded than the locally produced (Yellow Rock) oils obtained from subsurface reservoirs – and not from 7B cavern oil (which contains no oleanane; Fig. 2B). Notably, however, numerous other sheens and oils collected from the surface environment at Sulphur Dome are as severely biodegraded as the oil near Bubble Site 24 studied herein (see below, *Review of Surface Oils/Sheens Studied to Date*).

Given to the obvious effect of severe biodegradation on terpenoid biomarkers in the surface oil near Bubble Site 24 studied herein (Fig. 2A), **Figure 3** shows the *m/z* 231 EIPs of the far more biodegradation-resistant biomarkers, *viz*., triaromatic steroids (TAS), in the same three oils shown in Figure 2. Inspection of these shows that the TAS distribution of the surface oil near Bubble Site 24 studied herein (Fig. 3A) is distinct from that of the 7B cavern oil (Fig. 3B) and highly comparable to that of the locally produced oil (except for the absence of TAS05 to TAS08; Fig. 3C).[4] The absence of these four early-eluting TAS, i.e., C20-C22 pregnanes, in the surface oil sample studied (Fig 3A) is reasonably attributable to these compounds' loss due to their dissolution in surface water, which has not affected these TAS in either of the subsurface oils (Fig. 3B-C).

Thus, qualitative comparisons of biomarkers show that:

- Crude oil comprising the surface oil collected near Bubble Site 24 is derived from locally produced crude oil – and not from 7B cavern oil.

Although being clearly distinct based on their qualitative comparisons described above, in keeping with previous fingerprinting studies, a quantitative (statistical) comparison was conducted between the surface oil near Bubble Site 24 sample studied and the 7B cavern (reference) oil studied herein using the 30 diagnostic ratios (DRs) employed to date in previous Sulphur Dome fingerprinting studies. These DRs are contained in **Table 3** wherein the 7B cavern (reference) oil collected in January 2023, which was re-analyzed as part of this study, is statistically compared to the surface oil near Bubble Site 24studied herein.

As in the past studies, those DRs that are presently determined to be less precisely measured over both the short term and long term of the Sulphur Dome studies (Attachment 2, Table A2-1) are "greyed out" as they tend to exhibit higher standard errors under repeatability and/or reproducibility conditions (RSD_r and RSD_R) using the CEN protocol's 95% confidence level criteria.[5,6] The green and red color-coding in Table 3 reveals those DRs that statistically match (green) and statistically differ (red) from the 7B cavern (reference) oil re-analyzed herein.

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[4] It is worthwhile to remind the reader that a general similarity among biomarkers is unsurprising as many oils will contain biomarkers derived from comparable suites of ancient organic matter that gave rise to the oil over geologic time. Oil fingerprinting relies upon "the details", not general similarities, which in this case (Fig. 3) is reflected by the relative peak heights/areas of the different TAS.

[5] 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.

[6] 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).

The severity of biodegradation of terpenoid biomarkers (hopanes) in the surface oil near Bubble Site 24 studied herein (Fig. 2A) renders many hopane-based DRs equivocal in this statistical comparison. Those DRs based on the biodegradation resistant TAS (Fig. 3), however, show multiple non-matching DRs between the sheen and 7B cavern oil (e.g., TAS09/TAS01 and TAS03/TAS01; Table 3). Thus, despite the severity of biodegradation of the surface oil near Bubble Site 24 studied herein, it is, nonetheless, a statistical "non-match" to the 7B cavern oil (Table 3).

As was provided in earlier fingerprinting reports, **Figure 4** shows a series of DR-based cross-plots that allow one to visualize the character of all samples from Sulphur Dome studied to date, i.e., 7B cavern oil, Yellow Rock well oils, and surface sheens/oils.[7] The surface oil near Bubble Site 24 collected February 26, 2024 is among the population of "Surface Sheens/Oils" plotted in Figure 4. [Note that not all 12 DRs cross-plotted in Figure 4 were measurable in the surface oil near Bubble Site 24 (Table 3) and therefore do not appear in Figure 4.]

The effect of severe biodegradation of hopanes in the Bubble Site No. 14 sheen sample collected September 20, 2023 has clearly effected those hopane-based DRs plotted in Fig. 4C, D, and E, and thereby causing anomalous data points to be plotted. However, those DRs based upon the relative abundance of sulfur-containing aromatics and the TAS are minimally affected so that Figs. 4A-B, respectively, show:

- The surface oil collected near Bubble Site 24 on February 26, 2024 is consistent with the locally produced crude oils and inconsistent with the 7B cavern oil.

Also evident in Figure 4, and as was previously concluded:

- The oils comprising the previously studied surface sheens/oils (from Bubble Sites No. 14, 20, 22, and 24 and Central Lake; Table 1) also are consistent with locally produced crude oils and inconsistent with 7B cavern oil.[8]

<u>Review of Surface Oils/Sheens Studied to Date</u>

In an earlier report,[9] I reviewed the character of all nine of the surface oil/sheen (net) samples from the Site that were studied to date. That review revealed that two surface samples contained no petroleum of any kind, but rather were comprised entirely of naturally occurring biogenic material.[10] Two other sheens comprised predominantly of naturally occurring biogenic material also contained a trace and minor amount of petroleum.[11] The remaining five sheens were all overwhelmingly comprised of petroleum, i.e., variably evaporated and biodegraded crude oil with no biogenic material obviously present.[12] The surface oil collected near Bubble Site 24 studied herein is now an additional sample to this last group.

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[7] These same six plots were included in an earlier report (e.g., see Fig. 5 in Report 6 or Fig. 4 in Report 7), so the only additions to these plots are for the two samples analyzed herein. Because the current report is focused on a surface oil, which was devoid of n-alkanes and acyclic isoprenoids (Fig. 1A), previously-presented plots involving these compounds (see Fig. 5A and 5B in Report 6) are not repeated in Fig. 4 herein.

[8] Two surface "sheen" samples studied did not contain any oil, only biogenic material, and these are not plotted in Figure 4; i.e., Central Pond "sheen" collected January 25, 2023 and Bubble Site 19 "sheen" collected Oct. 15, 2023; see Reports 1 and 9 (per Table 1) and the next section.

[9] See Report 9 (per Table 1).

[10] Central Lake (Jan. 25, 2023) and Bubble Site No. 19 (Oct. 15, 2023).

[11] Central Lake (Sept. 11, 2023) and Bubble Site No. 14 (Sept. 11, 2023).

[12] Bubble Site No. 22 (Jan. 25, 2023), Bubble Site No. 20 (Mar. 9, 2023), Bubble Site No. 24 (May 22, 2023), Central Lake (June 12, 2023), and Bubble Site No. 14 (Sept. 20, 2023).

The GC/FID chromatograms for these six surface sheen/oil samples comprised of evaporated and biodegraded are shown in **Figure 5**. The surface oil collected near Bubble Site 24 (Fig. 5F) is quite typical within this group but is among the least evaporated and most biodegraded, the latter of which is evidenced by the absence of hopanes (T15 and T19; as seen in Fig. 2A). Interestingly, the surface sheen collected from Bubble Site No. 24 on May 22, 2023 is more highly evaporated that the surface oil studied herein, but the former's biomarkers were also severely biodegraded, i.e., hopanes depleted and oleanane enriched, as observed in the surface oil near Bubble Site 24 studied herein. The comparability in the severe degree of biodegradation in the May 2023 sheen and February 2024 surface oil at/near Bubble Site 24 indicates:

- There is a degree of homogeneity in the crude oil observed at/near Bubble Site 24 in May 2023 and February 2024 – and both are clearly severely biodegraded local crude oil.

Chemical fingerprinting of these two crude oils found at/near Bubble Site 24, as well as the other surface locations containing crude oil (Bubble Sites 14, 20, 22 and Central Lake), were all demonstrated to all contain variably biodegraded, locally produced crude oil as represented by the 13 Yellow Rock oils studied to date (Figure 4). As previously discussed,[13] the origin of all of these local crude oils in the area's near surface environment likely derives from natural oil seepage, which first promulgated oil exploration/production at Sulphur Dome more than 150/100 years ago, or from spillage over decades of local oil production.[14]

None of the 11 surface sheens/oil studied to date contain 7B cavern oil leaked from Cavern 7.

Summary of New Findings

Chemical fingerprinting of a surface oil collected near Bubble Site No. 24 on February 26, 2024 shows:

- The oil is a severely (biodegraded) weathered crude oil that is consistent with locally produced crude oils – and inconsistent with 7B cavern oil.

This result adds to the previous conclusion, viz.:

- All nine of the surface sheens/oils collected since January 2023 that contained oil are unequivocally distinct from the 7B cavern oil but are all consistent with locally produced crude oils.

The severe weathering (biodegradation) of the local crude oils in these surface oils/sheens – *versus* the slight-to-moderate weathering (biodegradation) of the local crude oils within different wells/reservoir zones – argues:

- The area's surface oils/sheens studied to date are most likely derived from long-extant, local crude oils present (i.e., natural seepage and/or spillage) in Sulphur Dome's surface environment and not any recent/active seepage of deep subsurface local oil – and certainly not from 7B cavern oil leaked from Cavern 7.

[13] See Report 7 (per Table 1).

[14] Law Engineering Testing Company (1980) Geologic characterization of Sulphur Mines SPR site, Sulphur, Louisiana. Report to Sandia Laboratories dated Oct. 10, 1980 that appears as Section II in SPR Geotechnical Division Report SAND80-7141, dated March 1981. [The Dome's first oil wells were drilled in 1867 based on surface seeps with commercial production commencing in 1915 (p. 4-12).]

Please let me know if you have any questions.

Sincerely,

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

Attachments:
1: Chain-of-custody
2: Updated RSD table
3: Tabulated concentrations of TPH/SHC, PAH, and biomarkers
4: Full size GC/FID chromatograms
5: Selected GC/MS extraction ion profiles

Table 1: NewFields reports on the Westlake Sulfur Dome Study prepared to date.

All reports authored by S.A. Stout

Report No.	Title	Report Date
1	Chemical fingerprinting of oils, Westlake Sulphur Dome Study.	Mar. 10, 2023
2	Chemical fingerprint of oily net – No. 20, Westlake Sulfur Dome Study.	Apr. 27, 2023
3	Chemical fingerprint of 7B cavern oil – March 30, 2023, Westlake Sulfur Dome Study.	May 3, 2023
4	7B Cavern Oil, Cavern 4 Oil, Select Yellow Rock Well Oils, and a Bubble Site 24 Sheen – May 2023, Westlake Sulphur Dome Study.	July 11, 2023 – Amended July 14, 2023
5	Chemical fingerprint of 7B cavern oil, selected Yellow Rock well oils and a Central Lake sheen – June 2023, Westlake Sulfur Dome Study.	July 25, 2023
6	Chemical fingerprinting of additional Yellow Rock well oils – mid- to late-August 2023, Westlake Sulphur Dome Study.	Oct. 4, 2023
7	Chemical fingerprint of floating materials, Central Lake and Bubble Site 14 – September 11, 2023, Westlake Sulphur Dome Study.	Oct. 5, 2023
8	Chemical fingerprint of Bubble Site 14 sheen – September 20, 2023, Westlake Sulphur Dome Study.	Oct. 17, 2023
9	Chemical fingerprint of Bubble Site 19 sheen – October 15, 2023, Westlake Sulphur Dome Study.	Nov. 7, 2023
10	Chemical fingerprint of 7B cavern oil – October 2023, Westlake Sulfur Dome Study.	Nov. 21, 2023

Table 2: Inventory of samples from the current study and studied previously.

Current Study Samples

Client/ Field ID	Lab ID	Matrix	Date Collected	Description of Sample
Bubble Site Oil	L2410930-01	Oil	2/26/2024	Surface oil collected near bubble site No. 24
7B**	L2361423-02	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)

Previously-Studies Samples

Client/ Field ID	Lab ID	Matrix	Date Collected	Description of Sample
7B Oil*	L2363877-01	Oil	10/25/2023	Cavern oil from brine well 7B
7B**	L2361423-02	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
Westlake #19	L2361423-01	Net	10/15/2023	Surface sheen from bubble site No. 19
7B**	L2361423-02	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
No. 14 Sheen Sample	L2355855-01	Net	9/20/2023	Surface sheen from bubble site No. 14
7B**	L2355855-02	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
Algae Sample Central Lake	L2353106-02	Net	9/11/2023	Sheen with pond "scum/algae"; suspected biologic
No. 14 Sheen Sample	L2353106-03	Net	9/11/2023	Surface sheen from bubble site No. 14
7B**	L2353106-04	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
253998*	L2348036-01	Oil	6/16/2023	Yellow Rock 253998
41842	L2348036-02	Oil	6/16/2023	Yellow Rock 41842
189416 (1250')	L2348036-04	Oil	6/16/2023	Yellow Rock 189416 from 1250' (bottom of oil column)
189416 (170')	L2348036-05	Oil	6/16/2023	Yellow Rock 189416 from 170' (top of oil column)
7B**	L2348036-03	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
Pad Oil	L2335058-01	Oil	6/16/2023	Stock tank oil used as cavern blanket/pad
7B*	L2335058-02	Oil	6/16/2023	Cavern oil from brine well 7B
252112	L2335058-03	Oil	6/16/2023	Yellow Rock 252112
109963	L2335058-04	Oil	6/16/2023	Yellow Rock 109963
185997	L2335058-05	Oil	6/16/2023	Yellow Rock 185997
209459	L2335058-06	Oil	6/16/2023	Yellow Rock 209459
Sheen	L2335058-07	Net	6/12/2023	Surface sheen from central lake
7B**	L2335058-08	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
209459	L2325505-01	Oil	5/2/2023	Yellow Rock 209459
185997	L2325505-02	Oil	5/2/2023	Yellow Rock 185997
Cavern 4	L2325505-03	Oil	5/25/2023	Cavern oil from brine well PPG No. 4
Cavern 7B*	L2325505-04	Oil	5/25/2023	Cavern oil from brine well 7B
210185	L2325505-05	Oil	5/25/2023	Yellow Rock 210185
Tank Battery	L2325505-06	Oil	5/25/2023	Yellow Rock Tank Battery
7B**	L2325505-07	Oil	1/25/2023	Site-specific reference oil; 7B Cavern Oil (Jan 2023)
BS-24	L2325505-08	Net	5/22/2023	Surface sheen from bubble site No. 24
Cavern 7B*	L2317387-01	Oil	3/30/2023	Cavern oil from brine well 7B
No. 20	L2313362-01	Net	3/9/2023	Surface oil sheen on water body west of the salt dome
7B*	L2305221-04	Oil	1/25/2023	Cavern oil from brine well 7B
110159	L2305221-02	Oil	1/25/2023	Yellow Rock 110159
Stock Tank	L2305221-03	Oil	1/25/2023	Stock tank oil used as cavern blanket/pad
Brine Well 22 BS*	L2305221-01	Net	1/25/2023	Surface oil brine well 22 excavation
Central Pond	L2305221-05	Net	1/25/2023	Surface sheen from central pond

* sample prepared and analyzed in duplicate

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

Table 3: Diagnostic ratios for the 7B cavern reference oil *versus* the surface oil near Bubble Site 24 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	7b Cavern Oil (Jan 2023)	Surface Oil near Bubble Site 24
	Analysis Date	3/16/2024	3/16/2024
NR-C17/pris	C17/Pr	2.59	ndp
NR-C18/phy	C18/Ph	2.07	ndp
NR- pris/phy	Pr/Ph	0.96	ndp
NR-4-MD/1-MD	4-MDBT/1-MDBT	2.36	ndp
NR-2-MP/1-MP	2-MP/1-MP	1.06	ndp
NR-27Ts/30ab	T11/T19	0.25	0.32
NR-27Tm/30ab	T12/T19	0.27	0.32
NR-28ab/30ab	T14a/T19	0.20	0.12
NR-29ab/30ab	T15/T19	0.88	0.81
NR-30O/30ab	T18/T19	0.05	2.29
NR-31abS/30ab	T21/T19	0.61	0.07
NR-27dbR/27dbS	S4/S5	0.65	0.60
NR-27bb/29bb	(S14+S15)/(S26+S27)	0.84	0.81
NR-SC26/ RC26+SC27	TAS09/TAS01	0.13	0.35
NR-SC28/RC26 + SC27	TAS02/TAS01	0.71	0.82
NR-RC27/RC26+ SC27	TAS03/TAS01	0.77	0.57
NR-RC28/RC26+SC27	TAS04/TAS01	0.60	0.67
DR-Ts/Tm	T11/T12	0.92	1.00
DR-29Ts30ab	T16/T19	0.19	1.52
DR-29bb/29aa	(S26+S27)/(S25+S28)	1.31	1.00
DR-C2-dbt/C2-phe	DBT2/PA2	2.27	ndp
DR-C3-dbt/C3-phe	DBT3/PA3	2.49	0.00
DR-C28C29/30ab	T7 to T10/T19	0.23	0.99
DR-29aaS/29aaR=	S25/S28	1.78	ndp
DR-C20TA/C21TA	TAS05/TAS06	1.32	0.37
DR-TA21/ RC26+SC27	TS06/TAS01	0.41	0.05
DR-C24Tet/C26Tri	T6a/T6bc	1.61	2.08
DR-30ba/30ab	T20/T19	0.08	0.00
DR-35ab/30ab	(T34 to T35)/T19	0.36	0.11
DR-32abR/32abS	T27/T26	0.72	ndp

Conclusion: Non-Match

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

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

grey: indicates less precision ratio (per Attachment 3)

ndp: no determination possible/division by zero

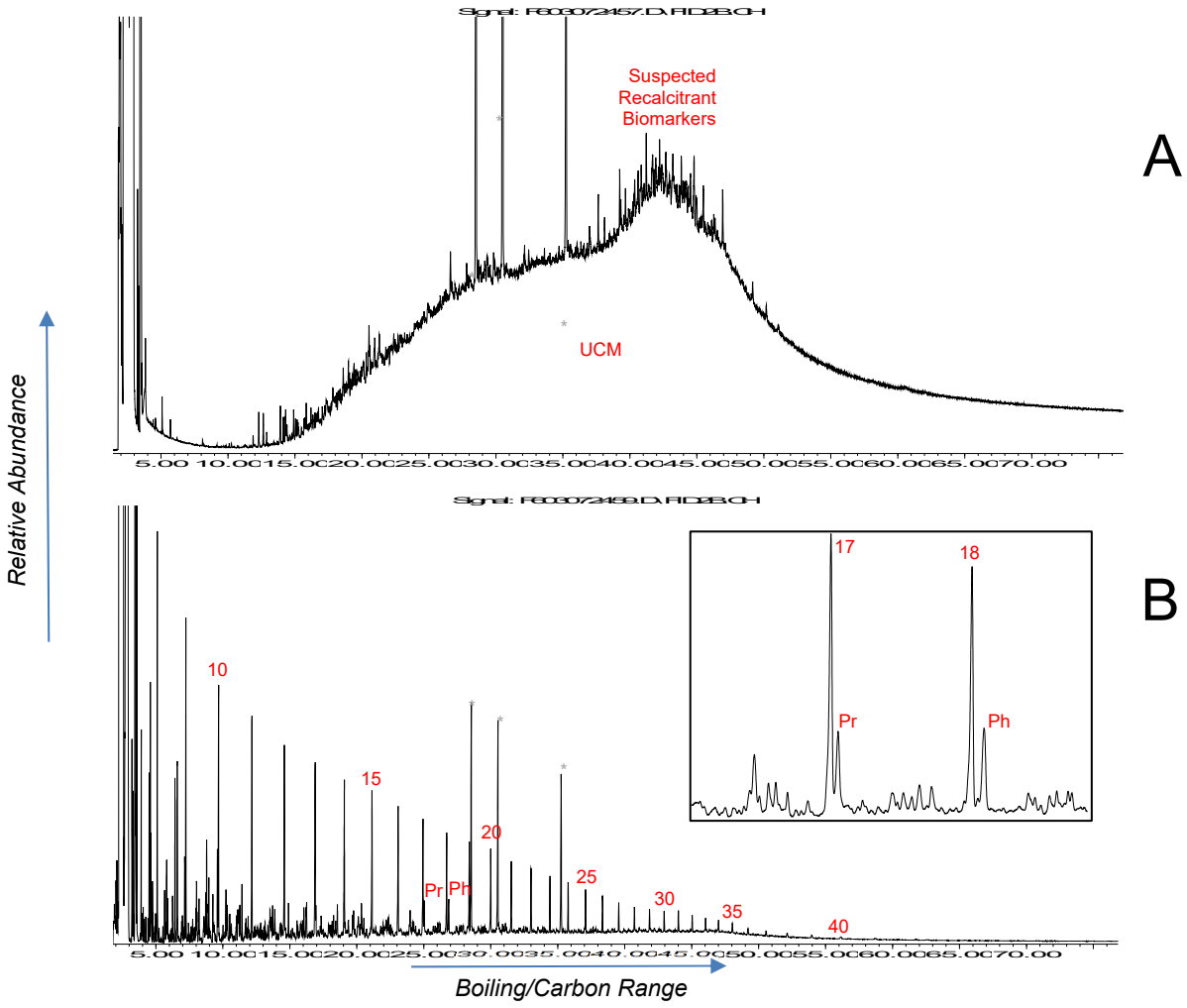

Figure 1: GC/FID (C8+) chromatograms for (A) Surface oil near Bubble Site 24 (Feb. 26, 2024) and (B) 7B cavern (reference) oil (Jan. 25, 2013) re-analyzed herein. #: n-alkane carbon number; Pr: pristane; Ph: phytane; UCM: unresolved complex mixture; *: internal standard.

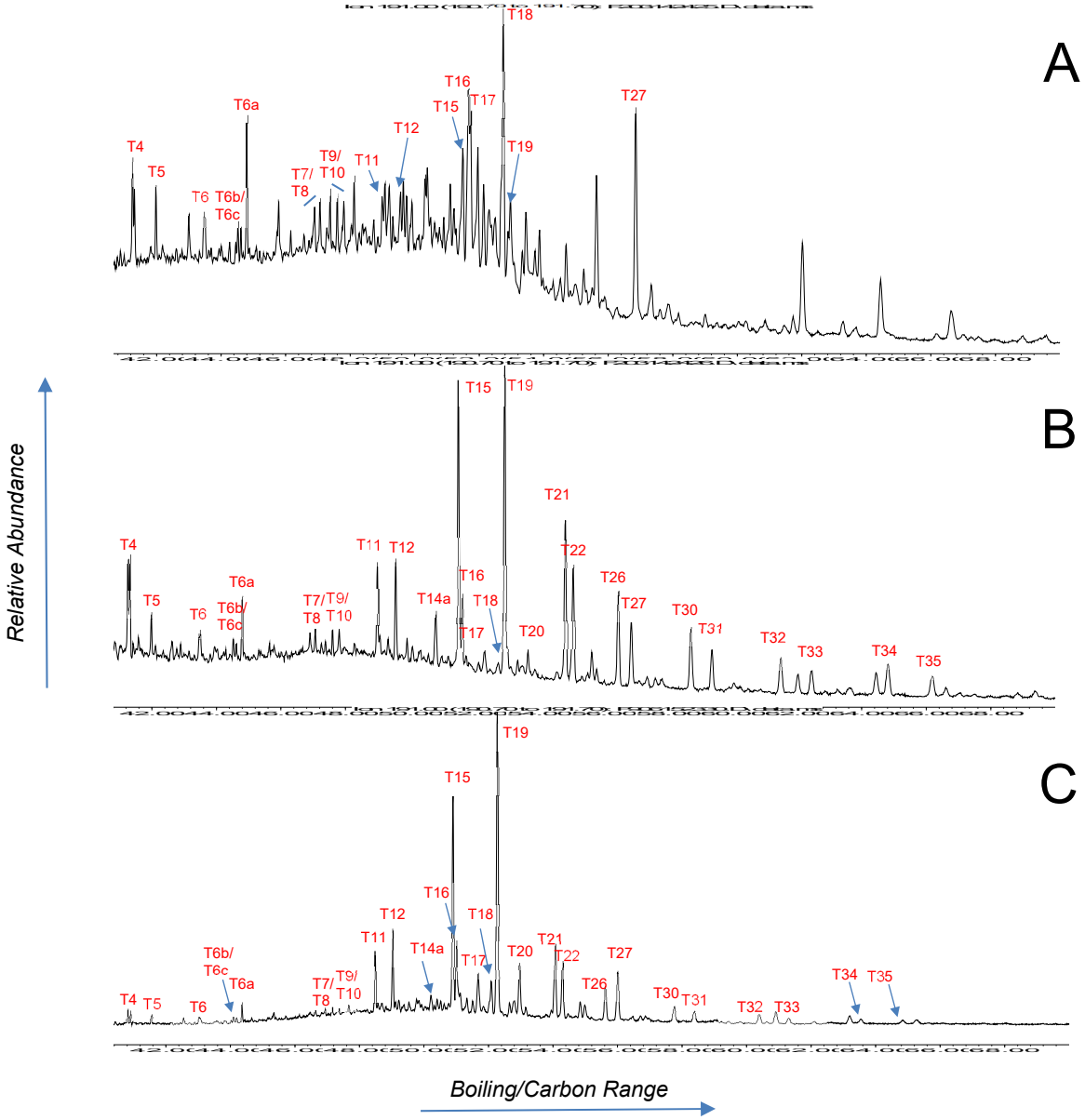

Figure 2: Partial extracted ion chromatograms (*m/z* 191) for (A) surface oil near Bubble Site 24 (Feb. 26, 2024) (B) 7B cavern (reference) oil (Jan. 25, 2023) analyzed herein and (C) a typical locally produced crude oil (Yellow Rock Well 209459). red labels: various triterpenoid biomarkers, see Attachment 3, Table A3-2 for compound names. See text for description.

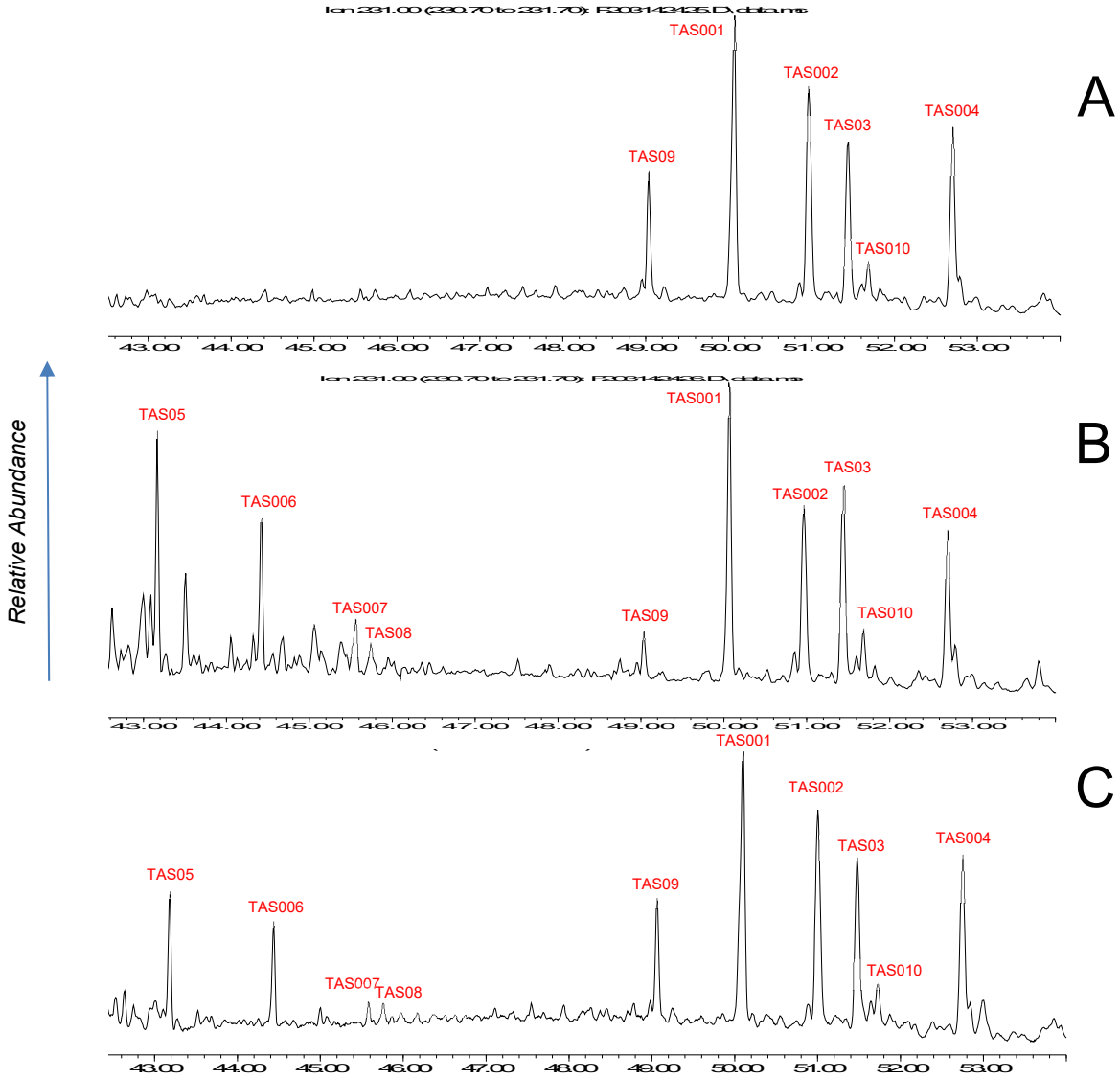

Figure 3: Partial extracted ion chromatograms (*m/z* 231) for (A) surface oil near Bubble Site 24 (Feb. 26, 2024) (B) 7B cavern (reference) oil (Jan. 25, 2023) analyzed herein and (C) a typical locally produced crude oil (Yellow Rock Well 209459). red labels: various triterpenoid biomarkers, see Attachment 3, Table A3-2 for compound names. See text for description.

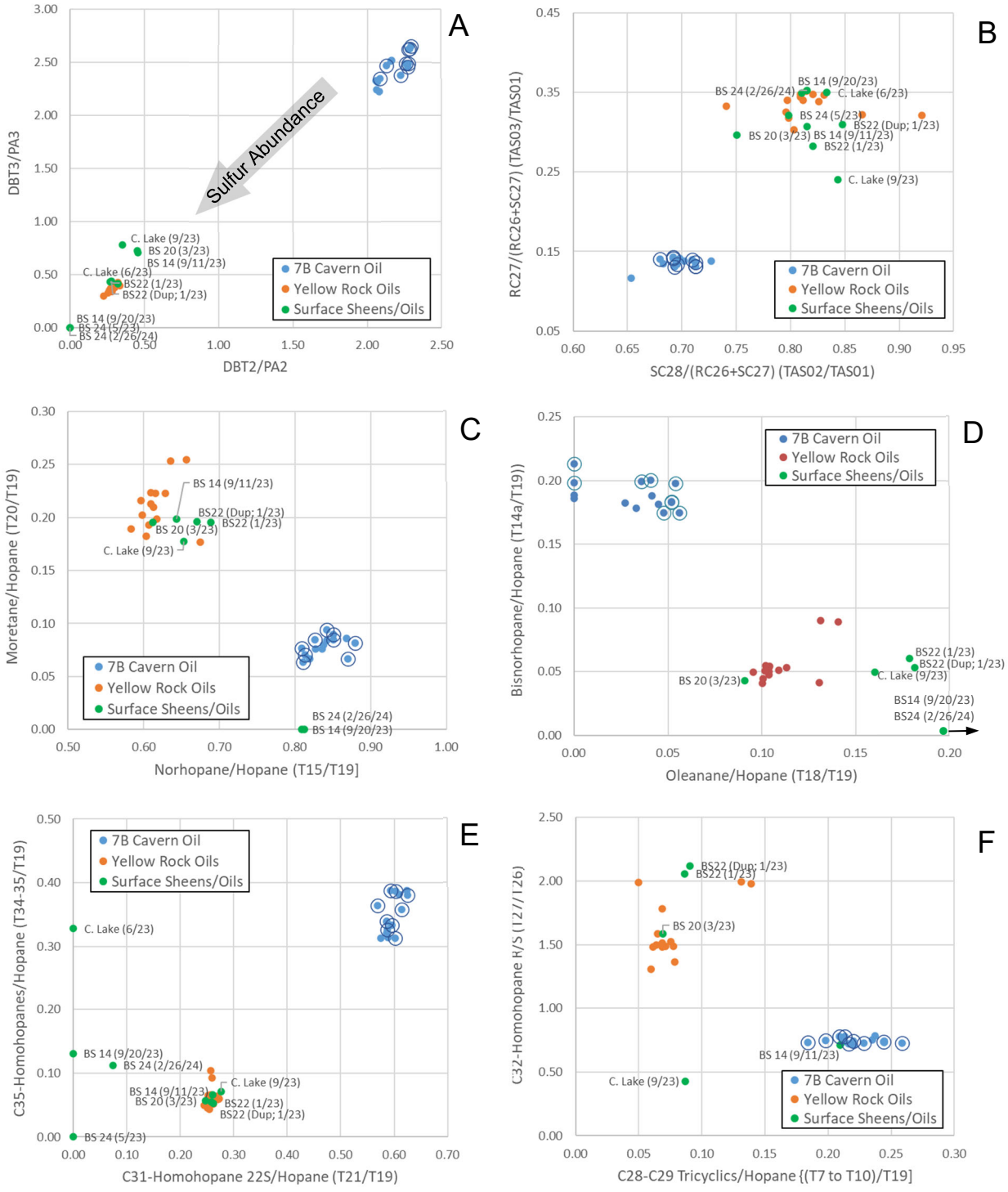

Figure 4: Cross-plots of select diagnostic ratios for all oil and surface oil/sheen samples studied to date that visually conveys the disparity between the populations of 7B cavern oil *versus* that of the Yellow Rock well oils and the area's surface oils/sheens. See text for description. Open black circles indicate 7B cavern (reference) oils re-analyzed over time.

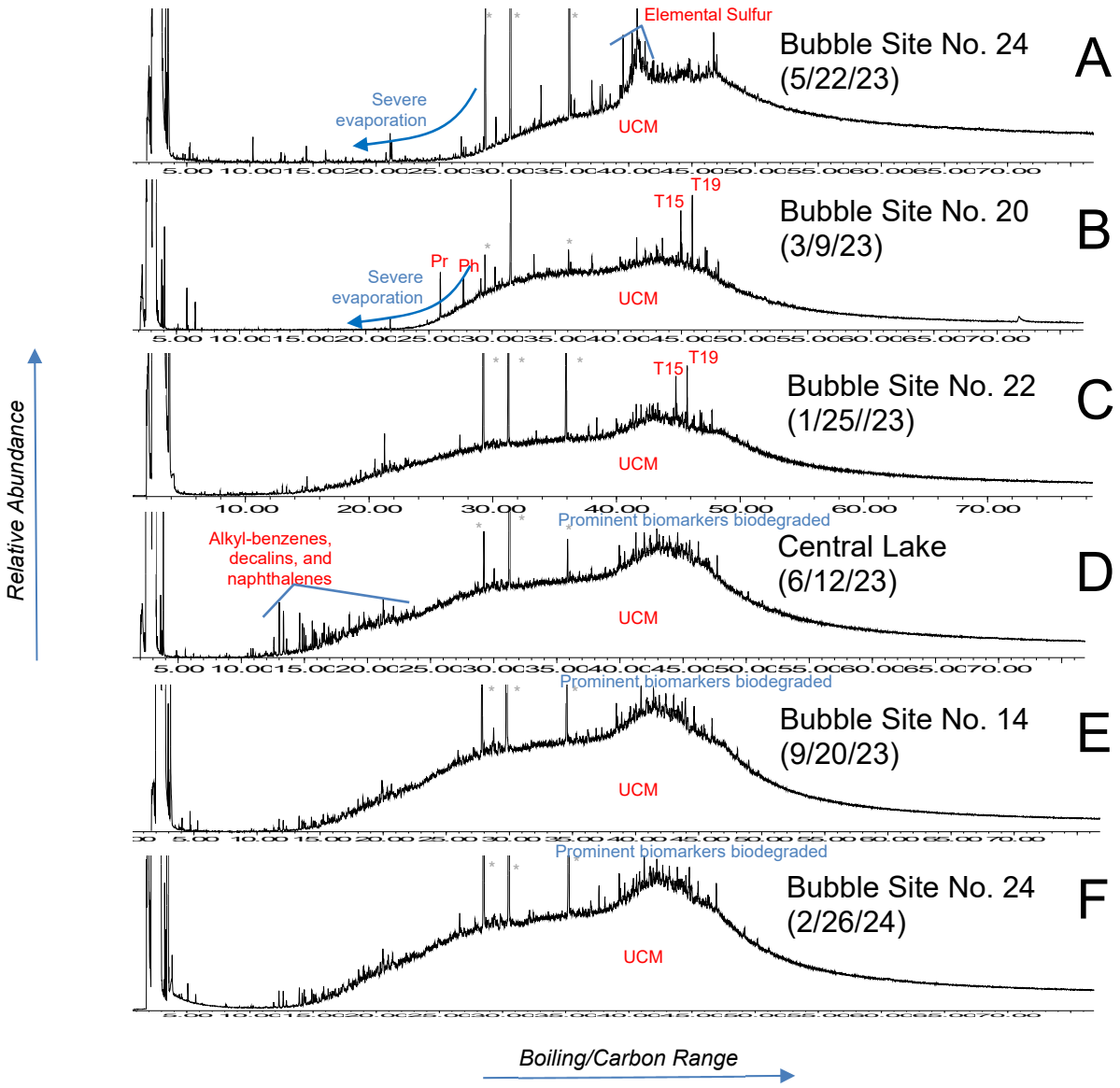

Figure 5: GC/FID (C8+) chromatograms for the six surface oils/sheens from the Sulphur Dome area studied to date overwhelmingly comprised of evaporated and biodegraded crude oil. UCM: unresolved complex mixture; *: internal standard. All chromatograms (except F) were previously presented and described in earlier fingerprinting reports.

ATTACHMENTS

Serial_No:03192410:12

NewFields
Perspective. Vision. Solutions.

Chain of Custody

2/29/24 L2410930

Environmental Forensics Practice LLC

Proj. No	Proj. Name
0722101	Sulphur Dome

SAMPLERS: Signature

DATE	TIME	LAB ID	CLIENT ID	SAMPLE DESCRIPTION	MATRIX (* see below)	GC-FID-TPH (C_6-)	GCMS-Alkyl PAH	GCMS-Biomarkers	PIANO - VOA	Organic Lead	METALS	PCB	Pesticides	PRESERVED	Total Number of Containers
				ANALYSIS REQUESTED→ "NUMBER OF CONTAINERS"											
2/26/2024	1445		Bubble Site - Oil	Oil	O	X	X	X	X						2

Relinquished by:	Date/Time	Received by:	Date/Time
	2/28/24 9:20	Fedex	
Fedex	2/29/24 10:07		2/29/24 10:07

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

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

Comments:
Contact Scott Stout for further details.

Attachment 1
Chain-of-Custody

Attachment 2

Table A2-1: Average short-term and long-term relative standard deviations (RSD_r and RSD_R) calculated for the 30 diagnostic ratios used in the Sulphur Dome monitoring studies to date.

CEN - Diagnostic Ratios	CEN Diagnostic Ratios per Alpha Abbreviations	Sulphur Dome Site Precision: Repeatability RSD_r	Sulphur Dome Site Precision: Reproducibility RSD_R	Most Precise Ratios*
NR-C17/pris	C17/Pr	2.0	5.0	
NR-C18/phy	C18/Ph	0.9	2.6	x
NR- pris/phy	Pr/Ph	1.6	4.2	x
NR-4-MD/1-MD	4-MDBT/1-MDBT	2.6	9.0	
NR-2-MP/1-MP	2-MP/1-MP	2.3	3.6	x
NR-27Ts/30ab	T11/T19	3.5	2.5	x
NR-27Tm/30ab	T12/T19	2.1	2.9	x
NR-28ab/30ab	T14a/T19	3.2	6.6	
NR-29ab/30ab	T15/T19	2.1	2.9	x
NR-30O/30ab	T18/T19	6.8	62.2	
NR-31abS/30ab	T21/T19	1.3	3.0	x
NR-27dbR/27dbS	S4/S5	5.3	16.2	
NR-27bb/29bb	(S14+S15)/(S26+S27)	3.5	2.4	x
NR-SC26/ RC26+SC27	TAS09/TAS01	2.6	4.7	x
NR-SC28/RC26 + SC27	TAS02/TAS01	2.9	1.9	x
NR-RC27/RC26+ SC27	TAS03/TAS01	1.9	1.9	x
NR-RC28/RC26+SC27	TAS04/TAS01	3.1	1.7	x
DR-Ts/Tm	T11/T12	2.7	3.1	x
DR-29Ts30ab	T16/T19	3.9	3.1	x
DR-29bb/29aa	(S26+S27)/(S25+S28)	4.4	12.7	
DR-C2-dbt/C2-phe	DBT2/PA2	0.7	3.3	x
DR-C3-dbt/C3-phe	DBT3/PA3	0.5	4.1	x
DR-C28C29/30ab	T7 to T10/T19	4.8	9.9	
DR-29aaS/29aaR=	S25/S28	7.7	22.4	
DR-C20TA/C21TA	TAS05/TAS06	4.1	9.3	
DR-TA21/ RC26+SC27	TS06/TAS01	4.2	8.8	
DR-C24Tet/C26Tri	T6a/T6bc	3.9	6.3	
DR-30ba/30ab	T20/T19	3.8	11.5	
DR-35ab/30ab	(T34 to T35)/T19	4.8	6.9	
DR-32abR/32abS	T27/T26	1.7	3.2	x

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

RSD_r = average RSD for sample duplicate pairs studied to date

RSR_R = average RSD for 7B cavern (reference) oil studied to date

Attachment 3

Tabulated Concentrations

Table A3-1: Concentrations (mg/kg) of n-alkanes and isoprenoids in the samples studied.

Client ID	BUBBLE SITE OIL	7B
Lab ID	L2410930-01	L2410930-02
Date Collected	2/26/2024	1/25/2023
Date Analyzed	3/9/2024	3/9/2024
Analytes	Result	Result
n-Nonane (C9)	nd	9,650
n-Decane (C10)	8	8,850
n-Undecane (C11)	5	8,520
n-Dodecane (C12)	30	8,110
n-Tridecane (C13)	24	7,320
2,6,10 Trimethyldodecane (1380)	41	1,420
n-Tetradecane (C14)	80	6,730
2,6,10 Trimethyltridecane (1470)	131	1,940
n-Pentadecane (C15)	nd	6,320
n-Hexadecane (C16)	nd	5,660
Norpristane (1650)	nd	1,160
n-Heptadecane (C17)	nd	4,810
Pristane	nd	1,860
n-Octadecane (C18)	nd	4,000
Phytane	nd	1,930
n-Nonadecane (C19)	nd	3,540
n-Eicosane (C20)	nd	3,620
n-Heneicosane (C21)	nd	2,750
n-Docosane (C22)	nd	2,460
n-Tricosane (C23)	nd	2,070
n-Tetracosane (C24)	nd	2,030
n-Pentacosane (C25)	nd	1,730
n-Hexacosane (C26)	nd	1,520
n-Heptacosane (C27)	nd	1,240
n-Octacosane (C28)	nd	1,010
n-Nonacosane (C29)	nd	896
n-Triacontane (C30)	nd	862
n-Hentriacontane (C31)	nd	890
n-Dotriacontane (C32)	nd	679
n-Tritriacontane (C33)	nd	566
n-Tetratriacontane (C34)	nd	704
n-Pentatriacontane (C35)	nd	604
n-Hexatriacontane (C36)	nd	361
n-Heptatriacontane (C37)	nd	331
n-Octatriacontane (C38)	nd	341
n-Nonatriacontane (C39)	nd	268
n-Tetracontane (C40)	nd	234
Total Saturated Hydrocarbons	319	107,000
Total Petroleum Hydrocarbons (C9-C44)	492,000	633,000

Table A3-2: Concentrations (mg/kg) of PAHs, related compounds and petroleum biomarkers in the samples studied.

		BUBBLE SITE OIL	7B
	Client ID		
	Lab ID	L2410930-01	L2410930-02
	Date Collected	2/26/2024	1/25/2023
	Date Analyzed	3/16/2024	3/16/2024
	Analytes	Result	Result
D0	cis/trans-Decalin	0.4	212
D1	C1-Decalins	12	362
D2	C2-Decalins	61	316
D3	C3-Decalins	105	203
D4	C4-Decalins	313	208
BT0	Benzothiophene	nd	10
BT1	C1-Benzo(b)thiophenes	2.8	54
BT2	C2-Benzo(b)thiophenes	12	175
BT3	C3-Benzo(b)thiophenes	45	316
BT4	C4-Benzo(b)thiophenes	29	256
N0	Naphthalene	0.3	239
N1	C1-Naphthalenes	0.4	741
N2	C2-Naphthalenes	3.6	1,170
N3	C3-Naphthalenes	15	1,030
N4	C4-Naphthalenes	50	589
B	Biphenyl	nd	37
DF	Dibenzofuran	0.1	26
AY	Acenaphthylene	nd	4.2
AE	Acenaphthene	0.2	8.4
F0	Fluorene	nd	53
F1	C1-Fluorenes	3.6	131
F2	C2-Fluorenes	15	212
F3	C3-Fluorenes	nd	228
A0	Anthracene	1.2	nd
P0	Phenanthrene	nd	94
PA1	C1-Phenanthrenes/Anthracenes	5.8	252
PA2	C2-Phenanthrenes/Anthracenes	nd	328
PA3	C3-Phenanthrenes/Anthracenes	27	236
PA4	C4-Phenanthrenes/Anthracenes	59	114
RET	Retene	nd	nd
DBT0	Dibenzothiophene	0.9	203
DBT1	C1-Dibenzothiophenes	nd	507
DBT2	C2-Dibenzothiophenes	nd	746
DBT3	C3-Dibenzothiophenes	nd	587
DBT4	C4-Dibenzothiophenes	19	297
BF	Benzo(b)fluorene	nd	4.1
FL0	Fluoranthene	0.6	1.7
PY0	Pyrene	nd	10
FP1	C1-Fluoranthenes/Pyrenes	13	40
FP2	C2-Fluoranthenes/Pyrenes	21	77
FP3	C3-Fluoranthenes/Pyrenes	56	99
FP4	C4-Fluoranthenes/Pyrenes	82	89
NBT0	Naphthobenzothiophenes	nd	42
NBT1	C1-Naphthobenzothiophenes	7.0	132
NBT2	C2-Naphthobenzothiophenes	18	203
NBT3	C3-Naphthobenzothiophenes	24	175
NBT4	C4-Naphthobenzothiophenes	36	123
BA0	Benz[a]anthracene	nd	1.3
C0	Chrysene/Triphenylene	1.4	17
BC1	C1-Chrysenes	nd	36
BC2	C2-Chrysenes	29	58
BC3	C3-Chrysenes	74	81
BC4	C4-Chrysenes	65	56

Table A3-2 (cont.)

		BUBBLE SITE OIL	7B
	Client ID		
	Lab ID	L2410930-01	L2410930-02
	Date Collected	2/26/2024	1/25/2023
	Date Analyzed	3/16/2024	3/16/2024
	Analytes	Result	Result
BBF	Benzo[b]fluoranthene	1.4	2.4
BJKF	Benzo[j]fluoranthene/Benzo[k]fluoranthen	0.9	nd
BAF	Benzo[a]fluoranthene	nd	nd
BEP	Benzo[e]pyrene	2.2	7.5
BAP	Benzo[a]pyrene	0.9	1.5
PER	Perylene	13.3	1.3
IND	Indeno[1,2,3-cd]pyrene	0.6	0.7
DA	Dibenz[ah]anthracene/Dibenz[ac]anthrace	0.7	0.5
GHI	Benzo[g,h,i]perylene	2.6	2.4
CAR	Carbazole	nd	8.5
4MDT	4-Methyldibenzothiophene	nd	219
2MDT	2/3-Methyldibenzothiophene	nd	190
1MDT	1-Methyldibenzothiophene	nd	93
3MP	3-Methylphenanthrene	nd	46
2MP	2-Methylphenanthrene	nd	58
2MA	2-Methylanthracene	2.9	2.2
9MP	9/4-Methylphenanthrene	0.8	88
1MP	1-Methylphenanthrene	nd	55
2MN	2-Methylnaphthalene	0.3	582
1MN	1-Methylnaphthalene	0.3	540
26DMN	2,6-Dimethylnaphthalene	0.4	482
235TMN	2,3,5-Trimethylnaphthalene	0.7	139
PY2	2-METHYLPYRENE	nd	2.6
PY4	4-METHYLPYRENE	0.4	10.3
PY1	1-METHYLPYRENE	nd	6.2
T4	C23 Tricyclic Terpane	44	26
T5	C24 Tricyclic Terpane	27	10
T6	C25 Tricyclic Terpane	30	12
T6a	C24 Tetracyclic Terpane	54	14
T6b	C26 Tricyclic Terpane-22S	13.7	5.0
T6c	C26 Tricyclic Terpane-22R	12.0	3.7
T7	C28 Tricyclic Terpane-22S	14.8	4.9
T8	C28 Tricyclic Terpane-22R	22.8	4.9
T9	C29 Tricyclic Terpane-22S	21.9	5.6
T10	C29 Tricyclic Terpane-22R	20	6.8
T11	18a-22,29,30-Trisnorneohopane-TS	25	24
T11a	C30 Tricyclic Terpane-22S	28.0	4.8
T11b	C30 Tricyclic Terpane-22R	11.8	6.0
T12	17a(H)-22,29,30-Trisnorhopane-TM	25	26
T14a	17a/b,21b/a 28,30-Bisnorhopane	10	19
T14b	17a(H),21b(H)-25-Norhopane	nd	4.3
T15	30-Norhopane	65	85
T16	18a(H)-30-Norneohopane-C29Ts	121	19
X	17a(H)-Diahopane	64.4	3.2
T17	30-Normoretane	51	10
T18	18a(H)&18b(H)-Oleananes	183.0	5.2
T19	Hopane	80	97
T20	Moretane	nd	7.9
T21	30-Homohopane-22S	5.9	60
T22	30-Homohopane-22R	25	46

Table A3-2 (cont.)

		BUBBLE SITE OIL	7B
	Client ID		
	Lab ID	L2410930-01	L2410930-02
	Date Collected	2/26/2024	1/25/2023
	Date Analyzed	3/16/2024	3/16/2024
	Analytes	Result	Result
T22A	T22a-Gammacerane/C32-diahopane	84	12
T26	30,31-Bishomohopane-22S	nd	37
T27	30,31-Bishomohopane-22R	157	27
T30	30,31-Trishomohopane-22S	nd	29
T31	30,31-Trishomohopane-22R	3.5	20
T32	Tetrakishomohopane-22S	10	20
T33	Tetrakishomohopane-22R	nd	14
T34	Pentakishomohopane-22S	nd	21
T35	Pentakishomohopane-22R	9.0	14
S4	13b(H),17a(H)-20S-Diacholestane	133	26
S5	13b(H),17a(H)-20R-Diacholestane	80	17
S23	14b,17b-20S-Methylcholestane	nd	30
S26	14b(H),17b(H)-20R-Ethylcholestane	15	45
S27	14b(H),17b(H)-20S-Ethylcholestane	8.5	25
TAS05	C20 PREGNANE	8.7	89.7
TAS06	C21 20-METHYLPREGNANE	23.7	67.9
TAS07	C22 20-ETHYLPREGNANE (A)	14.2	33.8
TAS08	C22 20-ETHYLPREGNANE (B)	17.7	20.5
TAS09	C26,20S TAS	162.0	22.0
TAS01	C26,20R+C27,20S TAS	460	167
TAS02	C28,20S TAS	375	119
TAS03	C27,20R TAS	264	128
TAS04	C28,20R TAS	308	100
TAS10	C29,20S TAS	85.8	43.7
TAS11	C29,20R TAS	36.2	21.3

Attachment 4

GC/FID Chromatograms

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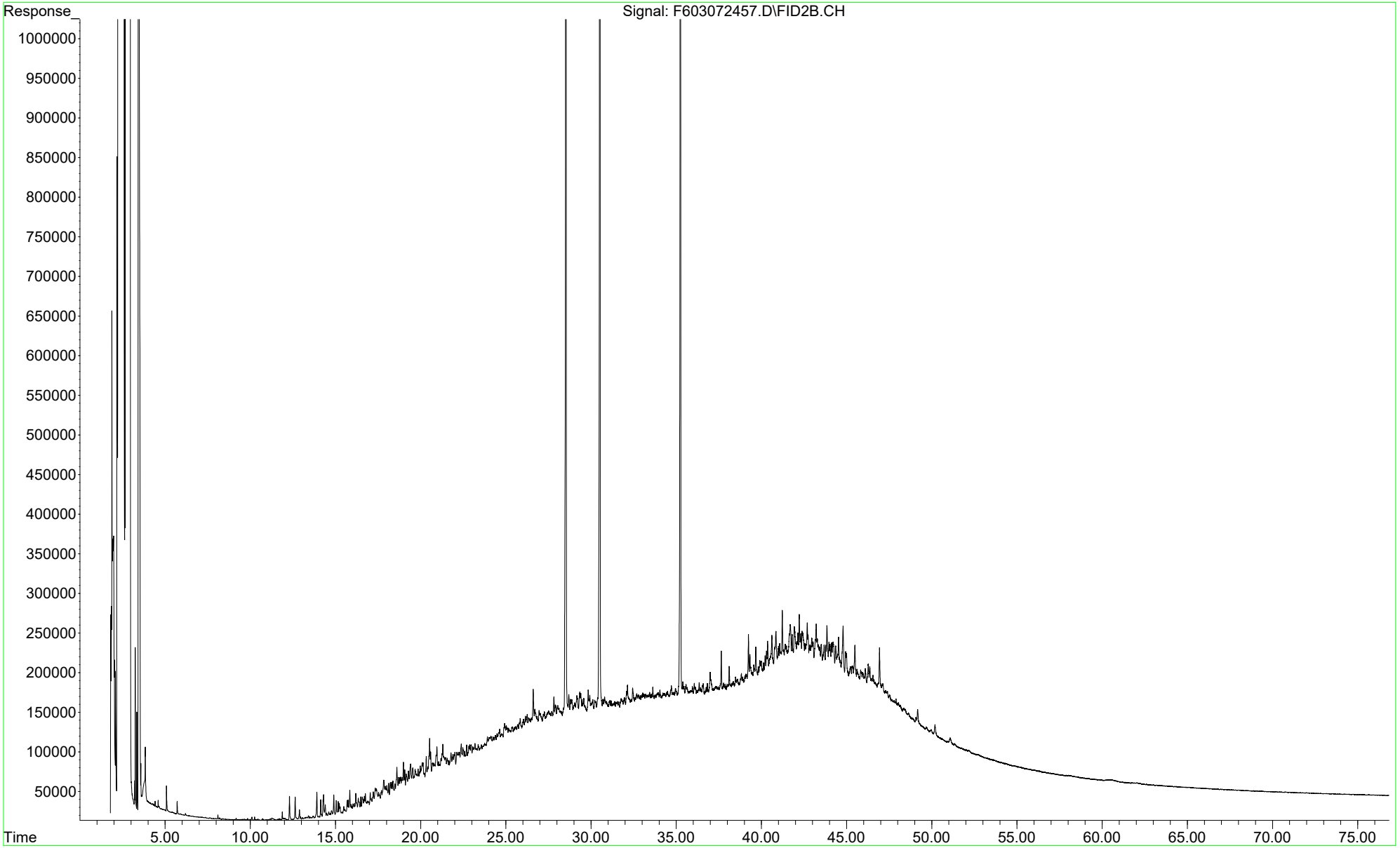
... C\F603072457.D
Operator : FID6:AMV
Instrument : FID6
Acquired : 09 Mar 2024 2:18 am using AcqMethod FID6A.M
Sample Name: L2410930-01,42,,,R4F
Misc Info : WG1893528,WG1892838,ICAL20449

BUBBLE SITE OIL
L2410930-01



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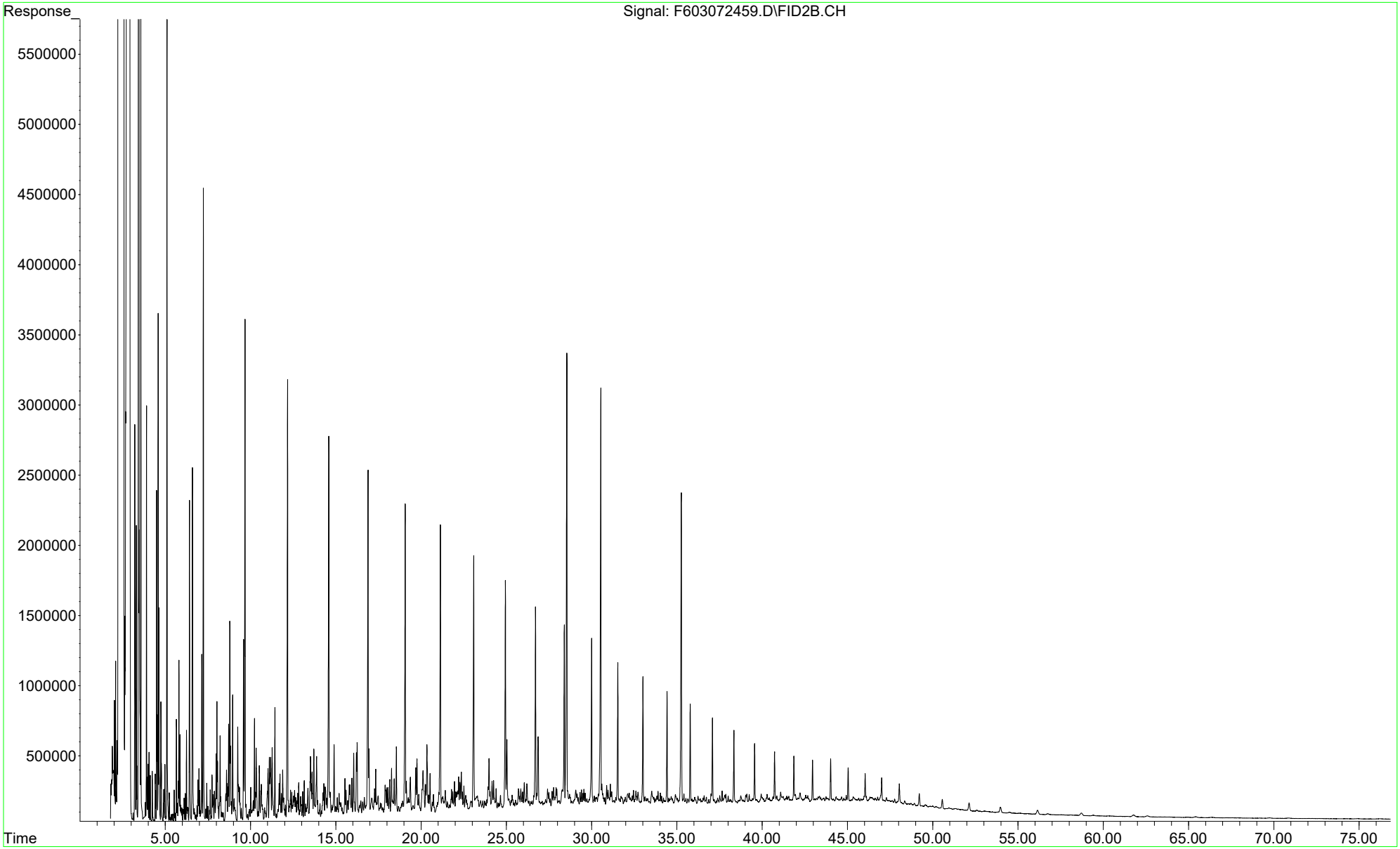
... C\F603072459.D
Operator : FID6:AMV
Instrument : FID6
Acquired : 09 Mar 2024 3:46 am using AcqMethod FID6A.M
Sample Name: L2410930-02,42,,,R4F
Misc Info : WG1893528,WG1892838,ICAL20449

7B
L2410930-02
Reference Oil



Attachment 5

GC/MS Extracted Ion Profiles

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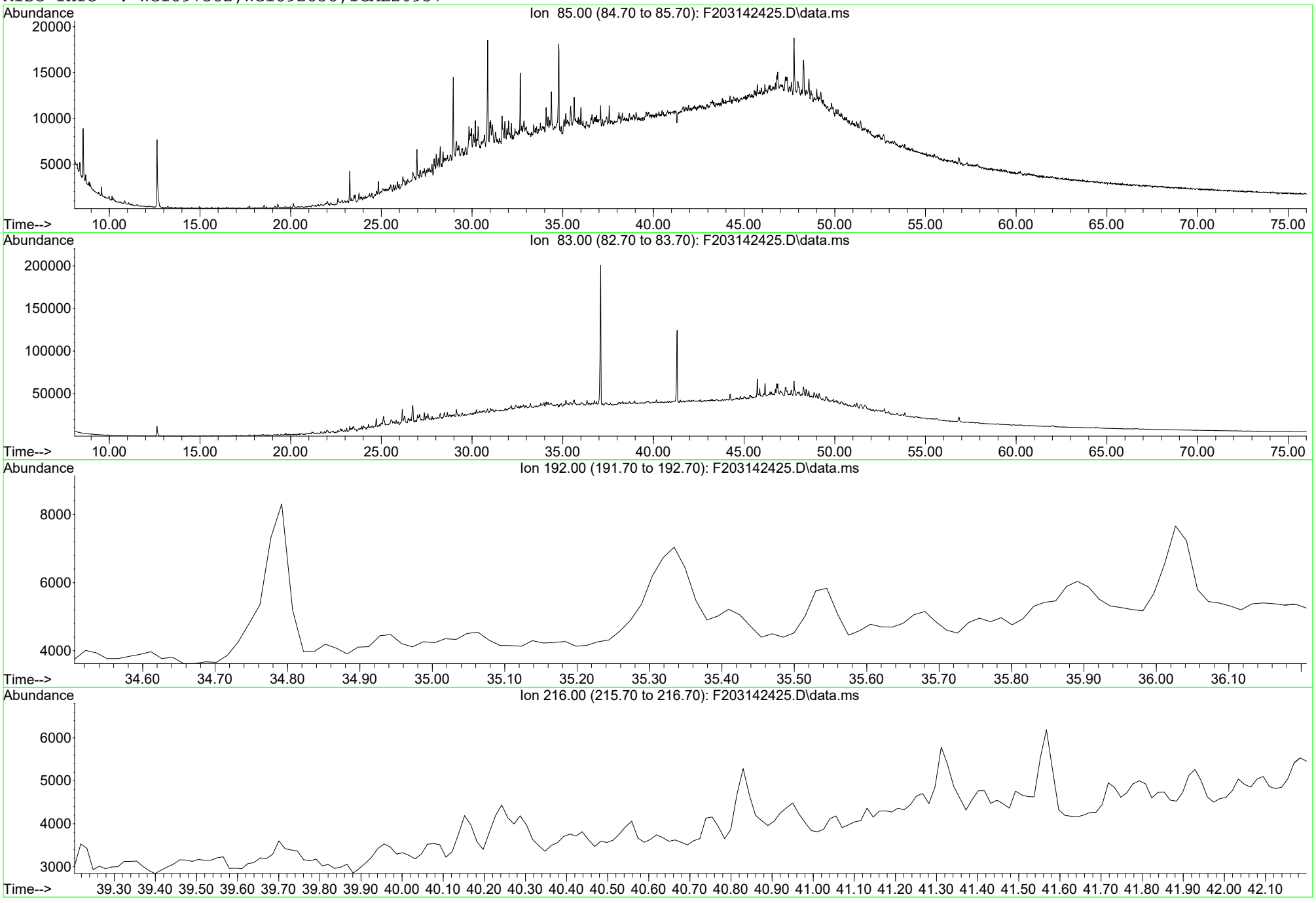
Instrument : PAH2
Acquired : 16 Mar 2024 3:43 am using AcqMethod FRNC2A.M
Sample Name: L2410930-01,32,,,R4F
Misc Info : WG1897582,WG1892838,ICAL20937

BUBBLE SITE OIL
L2410930-01



File :D:\West Lake Salt Dome_850.000079.023\Alpha Data\L2410930\AL
... KPAHBIO\F203142426.D
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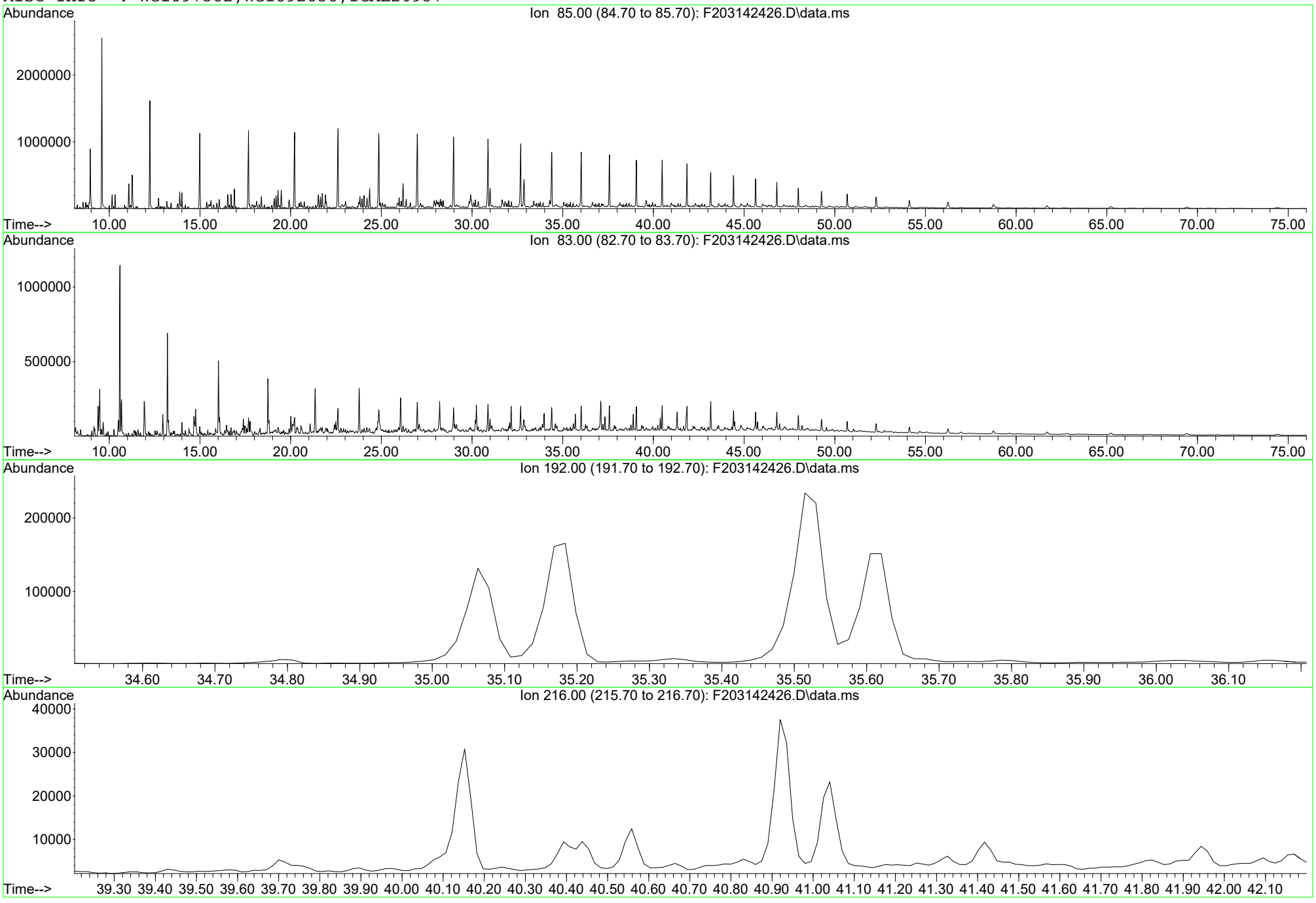
Instrument : PAH2
Acquired : 16 Mar 2024 5:12 am using AcqMethod FRNC2A.M
Sample Name: L2410930-02,32,,,R4F
Misc Info : WG1897582,WG1892838,ICAL20937

7B
L2410930-02
Reference Oil



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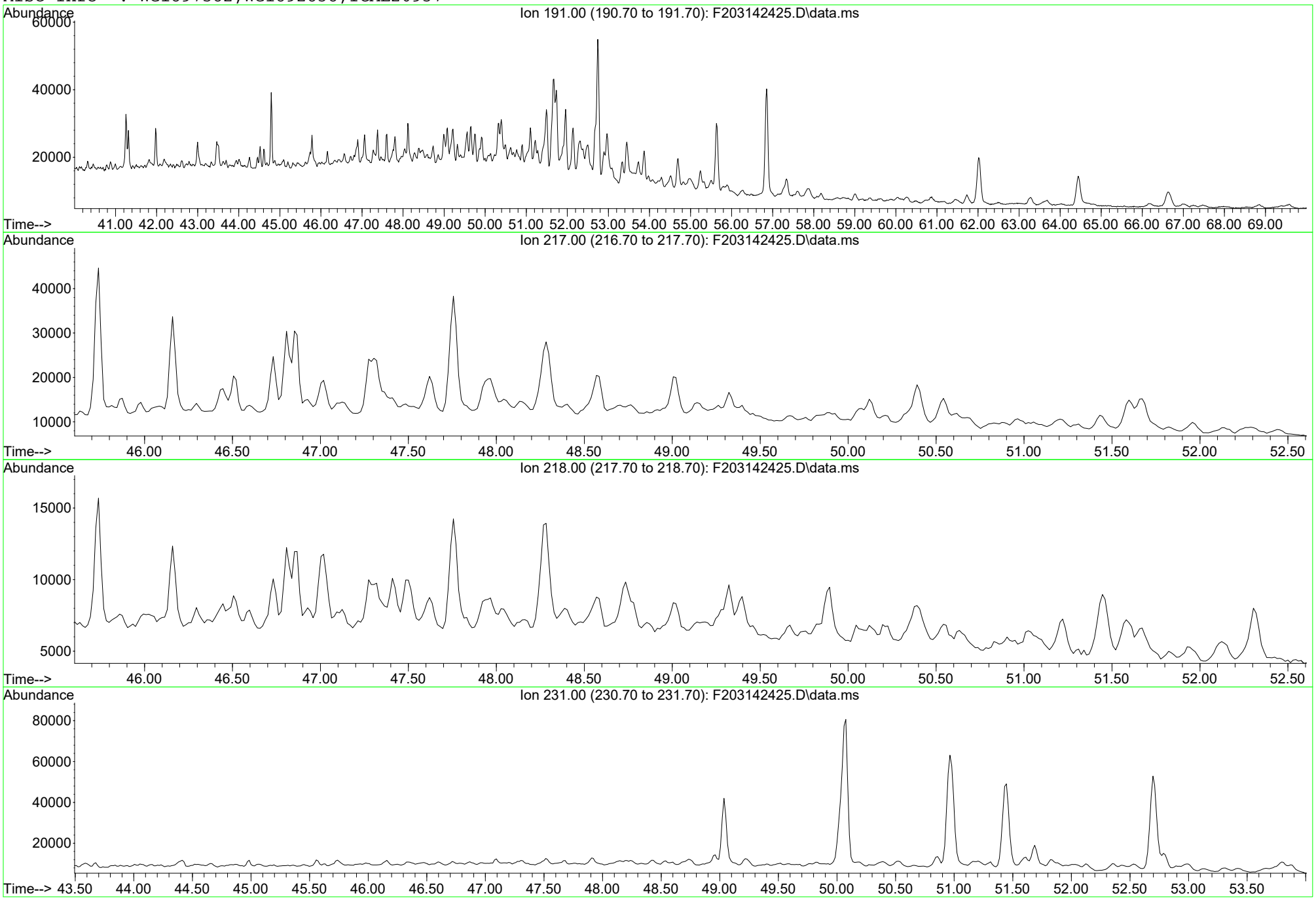
Instrument : PAH2
Acquired : 16 Mar 2024 3:43 am using AcqMethod FRNC2A.M
Sample Name: L2410930-01,32,,,R4F
Misc Info : WG1897582,WG1892838,ICAL20937

BUBBLE SITE OIL
L2410930-01



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Operator : PAH2:MJS
Instrument : PAH2
Acquired : 16 Mar 2024 5:12 am using AcqMethod FRNC2A.M
Sample Name: L2410930-02,32,,,R4F
Misc Info : WG1897582,WG1892838,ICAL20937

7B
L2410930-02
Reference Oil