Report: PWSRCAC Alaska North Slope [2015]

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Subject: Physical Properties, Behaviour and Composition of Alaskan North Slope [2015] Crude Oil

One sample was received from Prince William Sound Regional Citizens' Advisory Council for analysis of physical properties and simulated behaviour testing. Upon arrival, the sample was stored in a dark cold room at 2ºC. Upon reception, the sample was weathered by rotary evaporation (see method of preparation below) to form simulated evaporatively weathered oils without chemical oxidation. The percentage of mass lost by evaporation was monitored. Table 1 summarizes the original sample and the three evaporatively weathered fractions which were prepared.

Table 1 Description of the samples

ESTD Code	Evaporative Mass Loss (%)	Sample Description
2015/03/22-2713.1	0.00	Alaska North Slope Crude Oil [2015]
2015/03/22-2713.2.3	12.42	Lightly-weathered oil
2015/03/22-2713.2.2	24.56	Moderately-weathered oil
2015/03/22-2713.2.1	36.76	Heavily- weathered oil

Samples have been analyzed for physical properties, environmental behaviours, dispersant effectiveness and chemical composition. The results of these analyses can be found the following tables and figures:

Table 1	Sample information
Table 2	Physical Properties
Table 3	Emulsion formation
Table 4	GC-Detectible Hydrocarbons (Total Petroleum Hydrocarbons)
Table 5	Hydrocarbon Groups Analysis
Table 6	Simulated Distillation
Table 7	*n*-Alkanes composition and ratios
Table 8a	PAH and aPAHs
Table 8b	Individual PAH and aPAH
Table 8c	PAH and alkyl PAH diagnostic ratios
Table 9	Decalin and alkyl decalins
Table 10a	Hopanes and saturated steranes
Table 10b	Triaromatic Steranes
Table 10c	Biomarker diagnositic ratios

Figure 1 Pan evaporation for Alaska North slope [2015]

Figure 2 Simulated distillation of Alaska North slope [2015]

Figure 3 Distribution profiles of alkanes

Figure 4 Distribution profiles of aPAHs and PAHs

Figure 3 Distribution profiles of decalines

Figure 4 Distribution profiles of selected petroleum biomarkers (terpanes and saturated steranes)

Figure 5 Distribution profiles of triaromatic steranes

Brief descriptions of the techniques used to determine these results follow in an appendix. In addition, the selected chromatographs used to determine the chemical compositional data are includes as a PDF file as follows:

Figure S1 Gas chromatography with flame ionization detection **(**GC/FID) chromatograms of saturated fractions

Figure S2 GC/FID chromatograms of aromatic fractions

Figure S3 GC/FID chromatograms of total petroleum hydrocarbons (TPH)

Figure S4 Gas chromatography with mass spectrometry (GC/MS) single ion chromatograms of n-alkanes (m/z=57)

Figure S5 GC/MS single ion chromatograms of terpanes (m/z=191)

Figure S6 GC/MS single ion chromatograms of steranes (m/z=217)

Figure S7 GC/MS single ion chromatograms of steranes (m/z=218)

Figure S8 GC/MS total ion chromatograms of parent PAHs

Figure S9 GC/MS total ion chromatograms of alkylated PAHs

Figure S10 GC/MS single ion chromatograms of triaromatic steranes (m/z=231)

1 Attachment:

[PWSRCAC ANS [2015] Final Report 05-Feb-16 Supporting information.pdf]

Table 2: Physical properties, Alaska North Slope 2015 (ESTS #2713)

		% Evaporative Mass Loss			
		0.00%	12.42	24.56	36.76
Density (g/mL)	0ºC	0.8751	0.9091	0.9330	0.9563
	15ºC	0.8639	0.8983	0.9218	0.9444
API Gravity		31.32	25.30	21.46	17.93
Dynamic Viscosity (mPa·s)	0ºC	17.92	94.10	745.9	8427
	15ºC	9.852	36.64	170.8	1413
Surface Tension (mN/m)	0ºC	27.69	28.31	29.33	31.73
	15ºC	27.14	28.73	30.17	31.16
Interfacial Tension – Oil/Water (mN/m)	0ºC	24.09	27.57	29.89	NM
	15ºC	21.32	21.74	24.93	23.84
Interfacial Tension – Oil/Brine, 33% NaCl (mN/m)	0ºC	22.81	25.58	27.16	NM
	15ºC	19.75	22.15	28.05	23.71
Sulfur Content (%w/w)		0.9	1.1	1.3	1.4
Water Content (%w/w)		0.27%	0.01%	0.01%	0.01%
Flash Point (ºC)		NM	23.3	82.7	138
Pour Point (ºC)		-51	-48	-36	-3
Reid Vapor Pressure (K Pa) at 37.8°C		55.3	3.7	1.1	NM
Adhesion (g/m^2)		18.0	29.3	34.3	56.4
Swirling-Flask Dispersibility		45.9	51.4	51.6	4.9
Baffled-Flask Dispersibility		97.2	96.2	81.6	74.8
Evaporation mass loss (see Figure 1)		%Ev = (2.71 – 0.045T)ln t			

Note: NM: Not Measurable due to viscosity

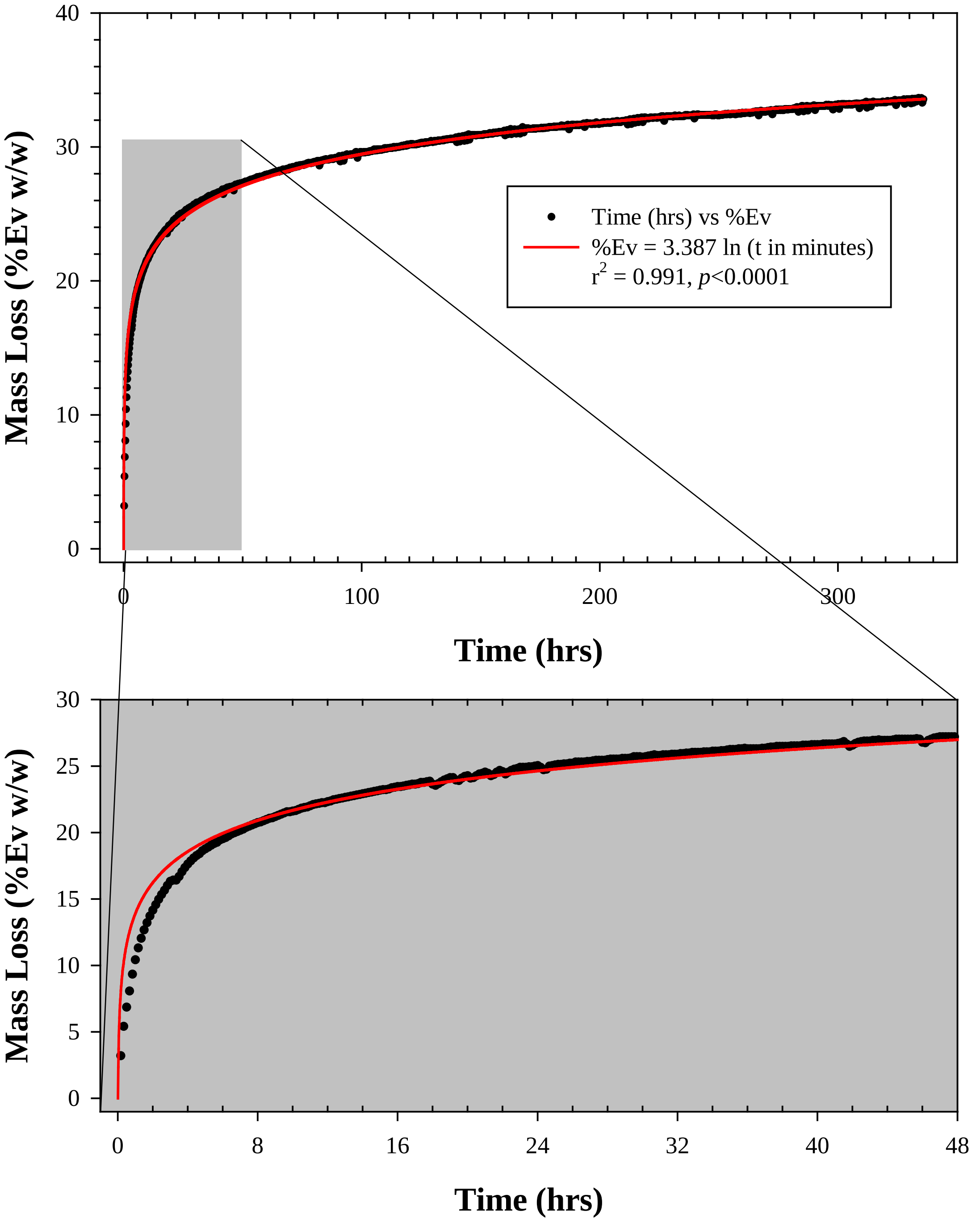

Figure 1. Mass loss (%Ev) of ANS [2015] at 15ºC for 14 days (top), with detail for first 2 days (bottom). Black dots are every 10th measured data point, red line is fit, as indicated.

Table 3: Emulsion Formation, Alaska North Slope 2015 (ESTS #2713)

% Evaporative Mass Loss	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)	
Emulsion Age	24-hrs	24-hrs	24-hrs	24-hrs	7-day
Visual Stability Emulsion	Unstable	Unstable	Unstable	Meso	Meso
Stress Tau (Pa)	0.4	0.4	0.6	6.1	7.0
Complex Modulus (Pa)	0.35	0.5	1.6	15.4	12.2
Storage Modulus (Pa)	0.2	0.1	0.1	0.9	1.1
Loss Modulus (Pa)	0.3	0.4	1.2	15.4	12.1
tan delta (V/E)	1.9	3.2	12.0	21.3	12.9
Complex Viscosity (Pa.s)	0.066	0.1	0.2	2.4	1.9
Water content (%w/w)	0.04	0.2	1.03	22.8	14.8

Notes: the W3 emulsion contains an amount of water that is very low for a meso-stable emulsion, too high to be unstable, and the oil was observed to be too fluid to class it as entrained. Previously, this behaviour might have been deemed to be unstable, but experience suggests that chemically stabilized water in the mix becomes significant when the bulk oil is weathered and stripped. We suggest that this be classified as a meso-stable emulsion, and broaden that category to include this type of situation.

Table 4: GC-Detectible Hydrocarbons, Alaska North Slope 2015 (ESTS #2713)

% Evaporative Mass Loss	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
TPH (mg/g oil)	547	572	614	558
TSH (mg/g oil)	353	382	396	353
TAH (mg/g oil)	194	190	218	206
Resolved Peaks (mg/g oil)	135	152	146	100
TSH/TPH (%)	64.5	66.8	64.5	63.2
TAH/TPH (%)	35.5	33.2	35.5	36.8
Resolved Peaks/TPH (%)	24.9	26.6	22.2	16.1
CCME (mg/g oil)				
F1 (Σ *n*-C6 to *n*-C10)	52.4	48.0	15.8	0.00
F2 (Σ *n*-C10 to *n*-C16)	153	161	170	75.5
F3 (Σ *n*-C16 to *n*-C34)	281	297	353	375
F4 (Σ *n*-C35+)	60.9	66.1	75.9	108

Table 5: Hydrocarbon Groups Analysis, Alaska North Slope 2015 (ESTS #2713)

% Evaporative Mass Loss	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
%Saturates (%w/w oil)	57.8	58.9	55.6	51.5
%Saturate: Waxes (%w/w oil)	(4.0)	(4.4)	(4.8)	(6.0)
%Aromatics (%w/w oil)	31.9	29.3	30.7	30.5
%Resins (%w/w oil)	6.5	7.2	8.3	10.7
%Asphaltenes (%w/w oil)	3.8	4.6	5.4	7.7

Table 6: Simulated Distillation, Alaska North Slope 2015 (ESTS #2713)

% Evaporative Mass Loss	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
(%w/w)	(ºC)	(ºC)	(ºC)	(ºC)
IBP (0.5%)	-11.7	72.0	149.8	228.6
5	60.3	118.3	195.3	264.3
10	91.9	150.4	223.6	287.5
15	119.4	179.5	248.3	307.7
20	147.9	210.7	271.3	327.7
25	180.6	237.0	295.8	347.9
30	215.7	264.0	317.6	368.8
35	247.7	285.4	342.6	389.8
40	277.7	315.7	367.1	411.3
45	308.0	342.5	391.5	431.6
50	339.1	369.5	417.1	453.7
55	371.2	397.8	441.7	478.7
60	404.3	425.8	470.5	505.9
65	436.3	454.9	502.3	536.5
70	473.3	488.9	538.6	570.2
75	516.4	527.9	579.1	604.5
80	567.6	572.5	619.9	637.4
85	622.6	619.0	657.4	672.7
90	680.2	663.3	708.6	719.4
%Recovery	92.44	94.66	90.79	90.02
FBP (100%)	>720	>720	>720	>720

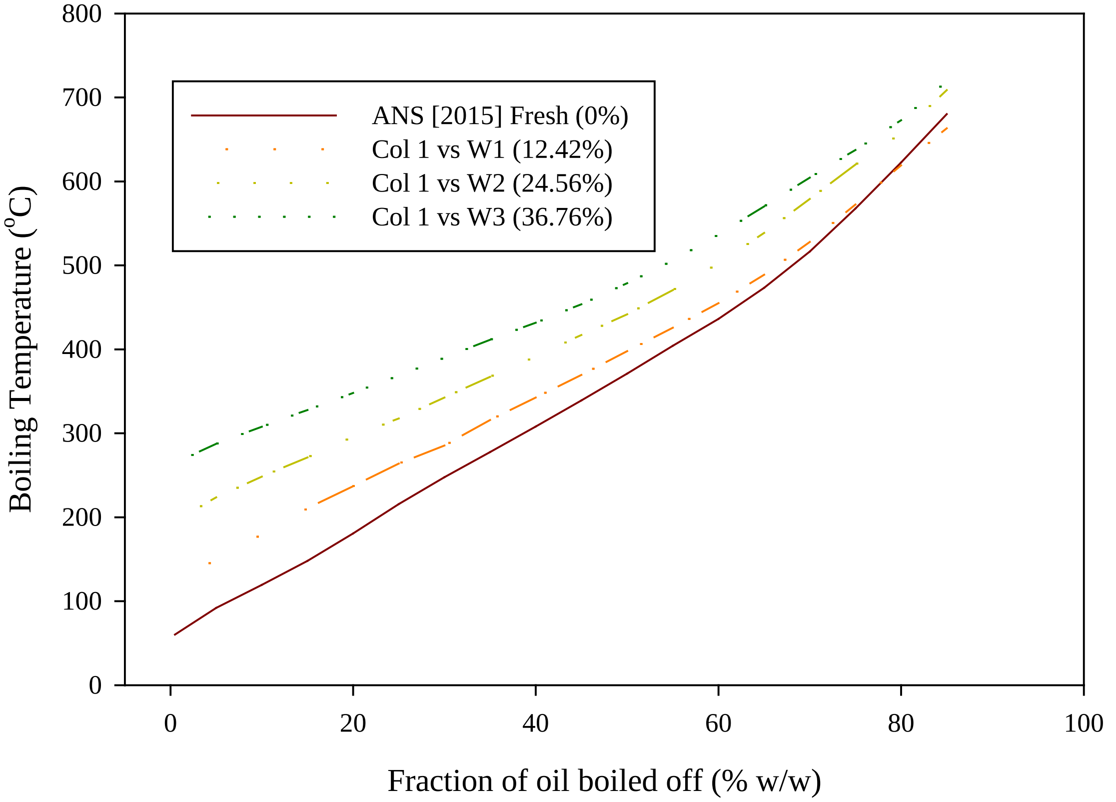

Figure 2 Simulated Distillation of Alaska North Slope 2015 (ESTS #2713)

Table 7: *n*-Alkanes, Alaska North Slope 2015 (ESTS #2713)

% Evaporative Mass Loss	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
	ug/g	ug/g	ug/g	ug/g
Alkanes				
n-C9	4102	3888	1222	1.20
n-C10	3656	3694	2771	0.48
n-C11	3208	3322	3235	3.01
n-C12	3057	3228	3402	308
n-C13	2850	3031	3234	1648
TMD	840	927	1009	723
n-C14	2599	2855	3069	2570
n-C15	2199	2392	2576	2574
n-C16	2024	2212	2382	2574
TMP	1037	1145	1265	1404
n-C17	2093	2265	2436	2363
Pristane	882	971	889	1106
n-C18	1686	1791	1964	2235
Phytane	974	1169	1285	1442
n-C19	1579	1675	1880	2103
n-C20	1467	1598	1730	1941
n-C21	1406	1498	1667	1901
n-C22	1343	1466	1594	1778
n-C23	1279	1399	1524	1714
n-C24	1213	1315	1386	1558
n-C25	1152	1261	1430	1583
n-C26	1082	1188	1301	1494
n-C27	876	918	1042	1197
n-C28	691	760	874	993
n-C29	576	662	722	827
n-C30	480	474	646	686
n-C31	403	454	524	623
n-C32	307	348	403	479
n-C33	262	299	348	417
n-C34	204	238	275	334
n-C35	194	227	266	327
n-C36	151	169	209	246
n-C37	116	166	190	224
n-C38	88.4	106	137	152
n-C39	60.3	71.9	88.6	104
n-C40	43.1	56.7	64.6	79.6
Total *n*-alkanes (μg/g)	46178	49237	49039	39709
Diagnostic indexes				
n-C17/Pristane	2.37	2.33	2.74	2.14
n-C18/Phytane	1.73	1.53	1.53	1.55
Pr/Ph	0.91	0.83	0.69	0.77
Odd alkanes	22354	23527	22384	17607
Even alkanes	18333	19570	20148	15582
CPI	1.22	1.20	1.11	1.13

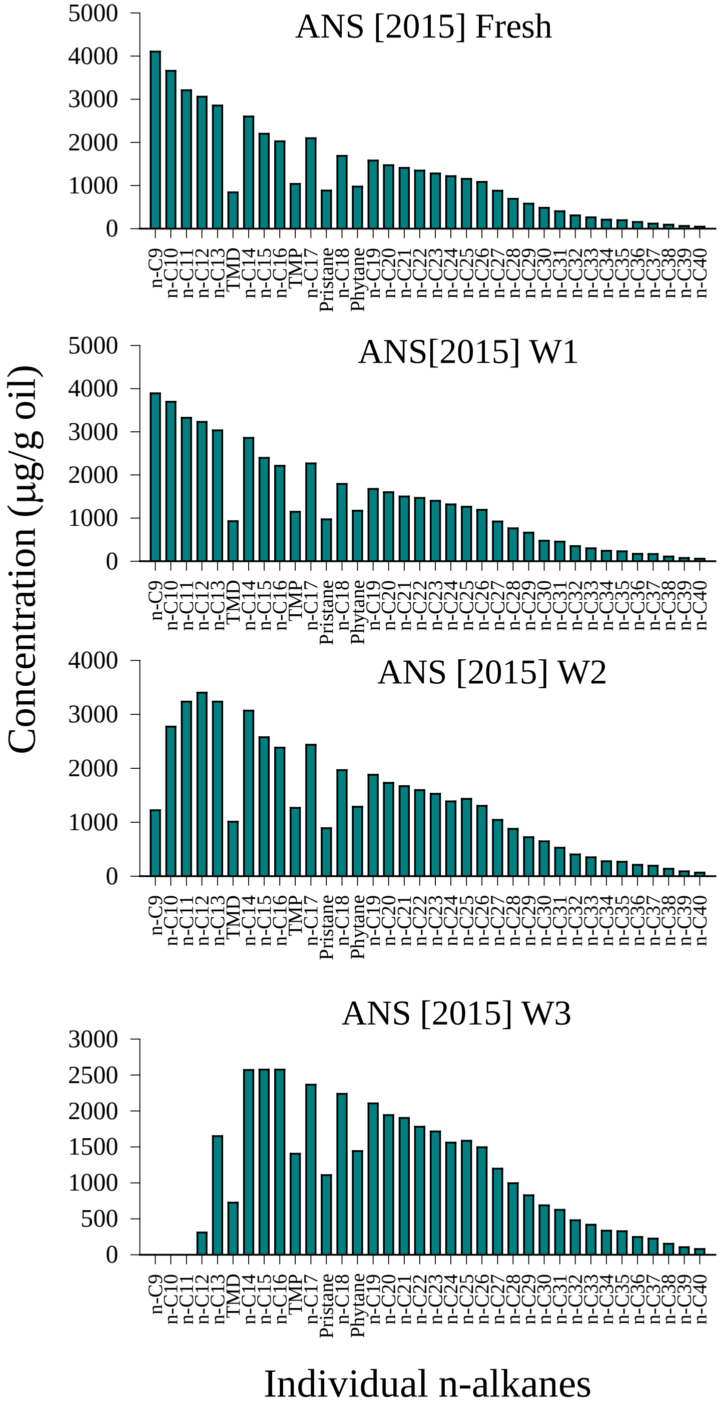

Figure 3 **Distribution profiles of alkanes**

Table 8a: PAH and alkyl PAH, Alaska North Slope 2015 (ESTS #2713)

Alkylated PAHs		GMM g/mol	QI m/z	Fresh (0%) ug/g oil	W1 (12.42%) ug/g oil	W2 (24.56%) ug/g oil	W3 (36.76%) ug/g oil
Naphthalene	C0-N	128	128	501	497	562	18.6
	C1-N	142	142	1377	1430	1645	651
	C2-N	156	156	2071	2163	2550	2074
	C3-N	170	170	1809	1877	2253	2358
	C4-N	184	184	961	1034	1232	1361
	Sum			6719	7001	8242	6464
Phenanthrene	C0-P	178	178	177	189	223	259
	C1-P	192	192	486	513	610	698
	C2-P	206	206	510	555	651	773
	C3-P	220	220	378	419	481	591
	C4-P	234	234	176	201	235	274
	Sum			1727	1877	2200	2595
Dibenzothiophene	C0-D	184	184	107	114	136	155
	C1-D	198	198	223	242	285	325
	C2-D	212	212	314	329	400	466
	C3-D	226	226	259	297	354	416
	C4-D	240	240	178	194	220	263
	Sum			1081	1176	1395	1624
Fluorene	C0-F	166	166	66.2	69.8	83.7	88.0
	C1-F	180	180	152	166	189	223
	C2-F	194	194	220	242	301	334
	C3-F	208	208	234	241	289	341
	Sum			672	719	862	986
Fluoranthene/pyrene	C0-Fl	202	202	4.26	4.80	5.56	6.67
	C1-Fl	216	216	50.8	56.9	60.4	77.6
	C2-Fl	230	230	87.8	95.9	107	136
	C3-Fl	244	244	104	111	132	151
	C4-Fl	258	258	78.5	86.2	99.4	116
	Sum			325	355	405	488
Benzonaphthothiophene	C0-B	234	234	41.1	43.0	52.2	62.1
	C1-B	248	248	137	148	178	211
	C2-B	262	262	108	116	112	128
	C3-B	276	276	174	191	226	262
	C4-B	290	290	115	128	146	171
	Sum			575	626	714	834
Chrysene	C0-C	228	228	29.7	32.7	39.9	44.9
	C1-C	242	242	53.5	58.9	71.4	84.3
	C2-C	256	256	79.0	81.3	96.9	119
	C3-C	270	270	82.6	108	113	123
	C4-C	284	284	110	134	144	165
	Sum			355	415	465	536

GMM = Gram Molecular Mass

QI = Quantitation Ion

Table 8b: Individual PAH and aPAH, Alaska North Slope 2015 (ESTS #2713) (continued)

Alkylated PAHs	Abbrev.	GMM g/mol	QI m/z	Fresh (0%) ug/g oil	W1 (12.42%) ug/g oil	W2 (24.56%) ug/g oil	W3 (36.76%) ug/g oil
Retene	R	234	234	22.2	22.6	26.8	32.5
4-Methyldibenzothiphene		198	198	109	117	137	159
2-/3-Methyldibenzothiphene		198	198	72.4	79.3	94.9	109.1
1-Methyldibenzothiphene		198	198	37.8	40.5	47.9	56.1
3-Methylphenanthrene		192	192	99	106	125	147
2-Methylphenanthrene		192	192	111	119	141	166
1-Methylanthracene		192	192	3.12	3.39	3.66	3.64
9/4-Methylphenanthrene		192	192	152	162	191	224
1-Methylphenanthrene		192	192	116	124	146	173
2-Methylnaphthalene		192	192	792	823	940	353
1-Methylnaphthalene		192	192	586	607	704	299
2,6-Dimethylnaphthalene		156	156	253	267	314	240
1,6,7-trimethylnaphthalene		170	170	136	143	171	181
Total alkylated PAHs				11454	12169	14282	13527
Other parent PAHs							
Biphenyl (Bph)	Bph	154	154	125.2	127.4	150.9	124.3
Acenaphthylene (Acl)	Acl	152	152	13.6	13.9	16.6	13.5
Acenaphthene (Ace)	Ace	154	153	14.8	13.8	18.5	13.5
Dibenzofuran	DB	168	168	47.1	48.4	57.3	47.3
Anthracene (An)	An	178	178	3.32	4.26	4.27	4.16
Fluoranthene (Fl)	Fl	202	202	5.18	5.65	6.36	5.52
Pyrene (Py)	Py	202	202	15.8	16.7	18.7	16.3
Benzo[b]fluorene(BbF)	BbF	216	216	21.2	24.1	28.0	34.8
Benz(a)anthracene (BaA)	BaA	228	228	2.81	2.98	4.02	2.91
Benzo(b)fluoranthene (BbF)	BbFl	252	252	4.74	5.31	6.20	5.18
Benzo(k)fluoranthene (BkF)	BkFl	252	252	0.00	0.00	0.00	0.00
Benzo(e)pyrene (BeP)	BeP	252	252	7.57	8.32	9.94	8.12
Benzo(a)pyrene (BaP)	BaP	252	252	2.08	1.70	2.01	1.66
Perylene (Pe)	Pe	252	252	3.29	4.04	4.82	3.94
Indeno(1,2,3-cd)pyrene (IP)	IP	276	276	0.56	0.60	0.86	0.59
Dibenzo(ah)anthracene (DA)	DA	278	278	1.19	1.36	1.54	1.33
Benzo(ghi)perylene (BgP)	BgP	276	276	2.94	3.09	3.74	3.02
Total EPA priority PAHs				271	282	334	286
Total aromatic compounds (all alkyl and EPA PAH)				11725	12451	14616	13813

GMM = Gram Molecular Mass

QI = Quantitation Ion

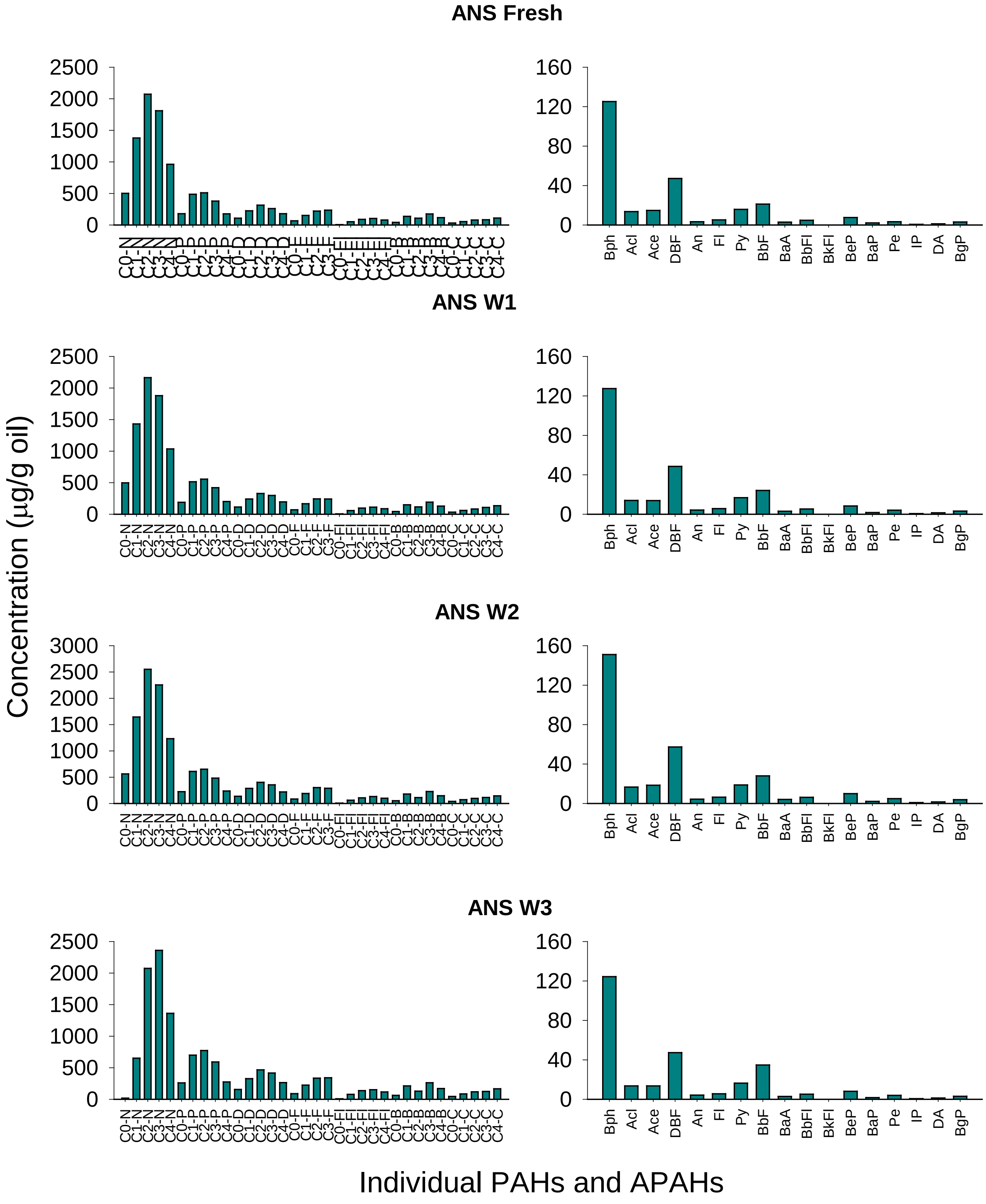

Figure 4 Distribution profiles of APAHs and PAHs

Table 8c: PAH and alkyl PAH Diagnostic Ratios, Alaska North Slope 2015 (ESTS #2713)

Diagnostic Ratios	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
2-m-N:1-m-N	1.35	1.36	1.33	1.18
(3-+2-)/(4-/9-+1-m-phen)	0.79	0.79	0.79	0.79
4-:2-/3-:1-m-DBT	1.00:0.66:0.35	1.00:0.68:0.35	1.00:0.69:0.35	1.00:0.68:0.32
(C2D/C2P):(C3D/C3P)	0.62:0.69	0.59:0.71	0.61:0.74	0.60:0.70
C0N:C1N:C2N:C3N:C4N	0.52:1.43:2.16:1.88:1.00	0.48:1.38:2.09:1.81:1.00	0.46:1.33:2.07:1.83:1.00	0.01:0.48:1.52:1.73:1.00
Naphs:Phens:DBTs:Fluors:Chrys	3.89:1.00:0.63:0.39:0.21	3.73:1.00:0.63:0.38:0.22	3.75:1.00:0.63:0.39:0.21	2.49:1.00:0.63:0.38:0.21

Table 9: Decalin and alkyl homologues, Alaska North Slope 2015 (ESTS #2713)

Compound	GMM g/mol	QI m/z	Fresh (0%) ug/g oil	W1 (12.42%) ug/g oil	W2 (24.56%) ug/g oil	W3 (36.76%) ug/g oil
Decalin	138	138	403	441	368	0.05
1-Methyl decalin	142	142	2208	2452	2362	6.57
2-Methyl decalin	142	142	2160	2400	2350	11.1
C2-Decalins	156	156	3123	3902	3745	322
C3-Decalins	180	180	720	916	910	328
C4-Decalins	194	194	812	1076	1139	787
Sum			**9426**	**11187**	**10874**	**1455**

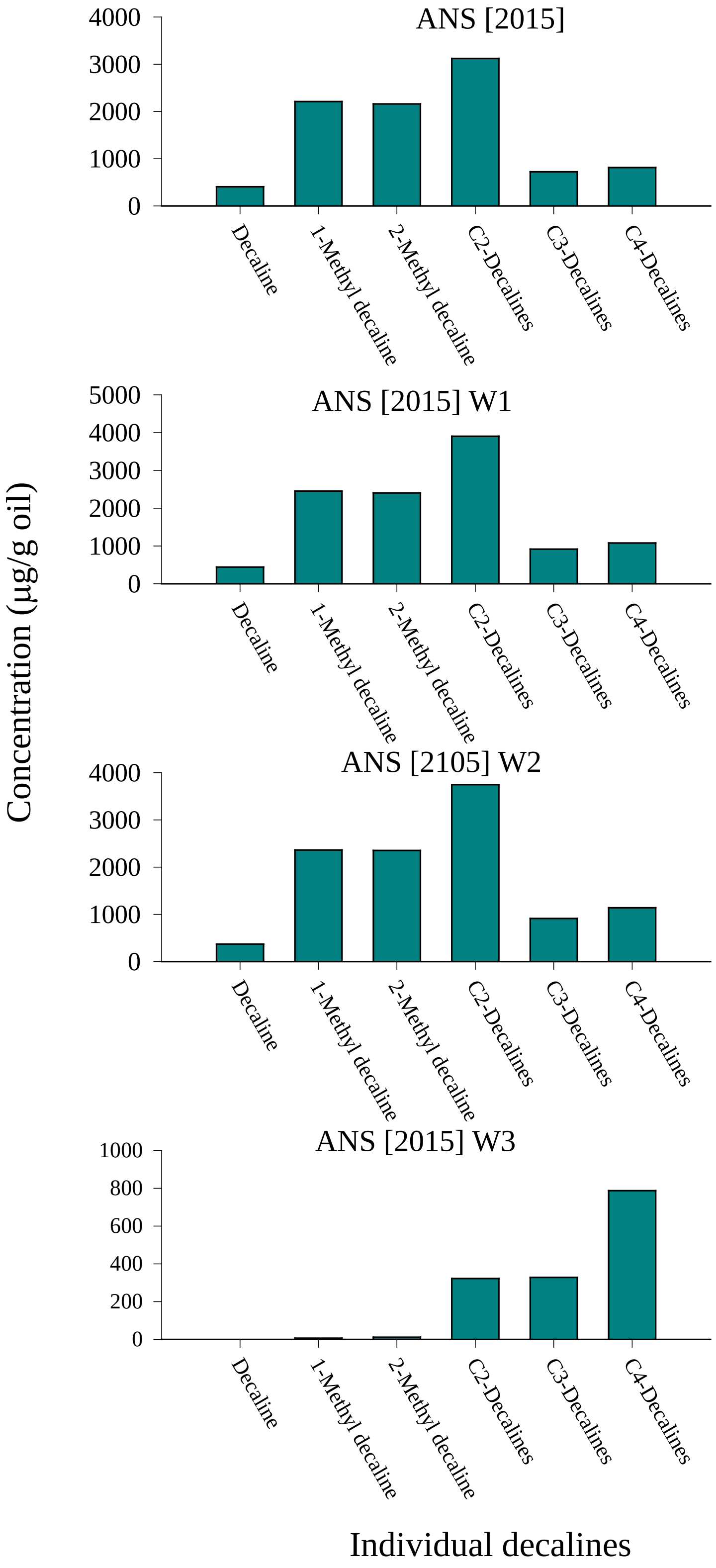

Figure 5 Distribution profiles of decalines

Table 10a: Hopane and Sterane Biomarkers, Alaska North Slope 2015 (ESTS #2713)

	GMM	QI		Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
			Abbrev.	ug/g oil	ug/g oil	ug/g oil	ug/g oil
C21 tricyclic terpane (C21T)	290	191	C21T	29.3	31.0	32.3	41.5
C22 tricyclic terpane (C22T)	304	191	C22T	13.0	14.3	14.7	18.3
C23 tricyclic terpane (C23T)	318	191	C23T	77.8	82.7	86.4	106.5
C24 tricyclic terpane (C24T)	332	191	C24T	43.5	45.9	47.7	60.3
C25 tricyclic terpane (C25T)	346	191	C25 T	45.7	46.5	48.4	68.0
C24 tetracyclic +C26-22S-tricycliic terpane C24T+C26TS)	330,360	191	C24TE+C26 TS	16.6	17.7	18.9	24.2
C26-22R tricyclic terpane (C26TR)	360	191	C26TR	31.3	33.3	35.2	44.5
C28, 22S tricyclic terpane (C28TS)	388	191	C28TS	16.8	17.6	19.8	24.4
C28, 22R tricyclic terpane (C28TR)	388	191	C28TR	20.3	22.7	24.1	31.1
C29,22S tricyclic terpane (C29TS)	402	191	C29TS	21.0	25.2	23.2	34.2
C29,22R tricyclic terpane (C29TR)	402	191	C29TR	23.1	25.0	26.3	33.6
18α,22,29,30-trisnorneohopane (C27Ts)	370	191	C27Ts	29.8	30.6	32.9	41.7
17α(H)-22,29,30-Trisnorhopane (C27Tm)	370	191	C27Tm	39.2	43.0	43.3	56.8
C30,22S tricyclic terpane (C30TS)	412	191	C30TS	13.4	15.2	15.8	24.3
C30,22R tricyclic terpane (C30TR)	412	191	C30TR	18.2	19.6	20.4	26.5
17α/ß,21ß/α 28,30-bisnorhopane (H28)	384	191	H28	10.9	11.7	12.6	15.7
17α(H),21ß(H)-25-Norhopane(NOR25H)	398	191	NOR25H	6.13	6.60	7.05	8.97
30-Norhopane(H29)	398	191	H29	99.0	107	117	133
18α(H)-20-Normeohopane (C29Ts)	398	191	C29Ts	23.0	29.6	25.3	31.5
17α(H)-Diahopane (DH30)	412	191	DH30	12.7	13.7	14.7	18.0
30-Normoretane (M29)	398	191	M29	10.5	11.7	12.1	14.8
18α(H)&18ß(H)-Oleananes (OL)	412	191	OL	0.96	0.78	1.50	1.76
Hopane (H30)	412	191	H30	131.8	142.2	148.4	187.7
Moretane (M30)	412	191	M30	20.8	21.7	22.4	28.2
30-Homohopane-22S(H31S)	426	191	H31S	58.1	63.2	65.5	83.1
30-Homohopane-22R(H31R)	426	191	H31R	38.7	46.4	48.6	62.1
T22a-Gammacerane/C32-diahopane (G)	412	191	G	10.3	11.7	11.0	17.2
30,31-Bishomohopane-22S(H32S)	440	191	H32S	41.1	44.4	47.1	59.0
30,31-Bishomohopane-22R(H32R)	440	191	H32R	29.9	32.6	35.4	42.5
30,31-Trishomohopane-22S(H33S)	454	191	H33S	31.8	34.9	35.5	44.2
30,31-Trishomohopane-22R(H33R)	454	191	H33R	21.1	22.8	24.0	30.8
Tetrakishomohopane-22S(H34S)	468	191	H34S	22.7	25.0	26.3	33.4
Tetrakishomohopane-22R(H34R)	468	191	H34R	14.8	16.2	17.1	21.8
Pentakishomohopane-22S(H35S)	482	191	H35S	24.6	26.9	28.1	36.2
Pentakishomohopane-22R(H35R)	482	191	H35R	16.9	18.9	19.2	24.9
13ß(H),17α(H)-20S-Diacholestane(DIA27S)	372	217	DIA27S	38.2	39.7	43.2	55.2
13ß(H),17α(H)-20R-Diacholestane(DIA27R)	372	217	DIA27R	27.8	27.6	31.2	40.1
13ß(H),17α(H)-20S-Methyldiacholestane(DIA28ßαS)	386	217	DIA28ßαS	24.0	25.9	27.1	34.8
14α(H),17α(H)-20S-Cholestane(C27αααS)	372	217	C27αααS	28.6	31.1	32.7	41.5
13ß(H),17α(H)-20S-Ethyldiacholestane(DIA29ßαS)	400	217	DIA29ßαS	60.5	65.5	65.5	87.3

	GMM	QI	Abbrev.	Fresh (0%) ug/g oil	W1 (12.42%) ug/g oil	W2 (24.56%) ug/g oil	W3 (36.76%) ug/g oil
14α(H),17α(H)-20R-Cholestane(C27αααR)	372	217	C27αααR	28.5	30.9	33.0	42.1
13ß(H),17α(H)-20R-Ethyldiacholestane(DIA29ßαR)	400	217	DIA29ßαR	41.1	48.0	42.9	57.2
Unknown steranes (UN)		217	UN	28.3	30.4	32.4	41.9
13α,17ß-20S-Ethyldiacholestane(DIA29αßS)	400	217	DIA29αßS	9.4	12.6	11.1	13.0
14α,17α-20S-Methylcholestane(C28αααS)	386	217	C28αααS	31.3	33.5	35.4	44.9
14α,17α-20R-Methylcholestane(C28αααR)	386	217	C28αααR	29.1	31.4	33.7	42.2
5α,14α,17α-20S-Ethylcholestane(C29αααS)	400	217	C29αααS	33.8	37.2	35.5	45.7
5α,14α,17α-20R-Ethylcholestane(C29αααR)	400	217	C29αααR	32.6	34.9	37.7	47.9
14ß(H),17ß(H)-20R-Cholestane(C27αßßR)	372	218	C27αßßR	45.5	49.0	47.6	65.3
14ß(H),17ß(H)-20S-Cholestane(C27αßßS)	372	218	C27αßßS	35.2	41.7	30.5	54.2
14ß(H),17ß(H)-20R_Methylcholestane(C28αßßR)	386	218	C28αßßR	40.1	43.3	45.5	58.3
14ß(H),17ß(H)-20S_Methylcholestane(C28αßßS)	386	218	C28αßßS	36.4	39.7	41.4	52.5
14ß(H),17ß(H)-20R-Ethylcholestane(C29αßßR)	400	218	C29αßßR	47.3	50.1	53.8	68.0
14ß(H),17ß(H)-20S-Ethylcholestane(C29αßßS)	400	218	C29αßßS	42.6	43.4	50.0	58.6

Table 10b: Triaromatic steranes Biomarkers, Alaska North Slope 2015 (ESTS #2713)

Biomarker compounds	GMM	QI		Fresh (0%) **ug/g**	W1 (12.42%) **ug/g**	W2 (24.56%) **ug/g**	W3 (36.76%) **ug/g**
C20 triaromatic sterane	260	231	C20TA	15.5	17.7	19.7	23.7
C21 triaromatic sterane	274	231	C21 TA	16.9	18.4	21.5	25.9
C22 triaromatic sterane	288	231	C22 TA	3.40	3.83	4.41	4.97
C26 triaromatic cholestane (20S)	344	231	SC26 TA	14.7	15.4	19.2	22.3
C26 triaromatic cholestane (20R)+C27 triaromatic ergostaene (20S)	344, 358	231	RC26TA +SC27 TA	51.6	56.0	65.8	78.6
C28 triaromaticstigmastane (20S)	372	231	SC28 TA	7.79	8.4	9.8	11.9
C27 triaromatic ergostaen (20R)	358	231	RC27 TA	30.2	33.1	38.7	46.0
C28 triaromatic stigmastane(20R)	372	231	RC28 TA	6.30	7.07	8.18	9.67
			Total	146	160	187	223

Table 10c: Biomarkers Diagnostic Ratios, Alaska North Slope 2015 (ESTS #2713)

Diagnostic Ratios	Fresh (0%)	W1 (12.42%)	W2 (24.56%)	W3 (36.76%)
C23/C24	1.79	1.80	1.81	1.76
C23/C30	2.46	2.38	2.39	2.09
C24/C30	1.37	1.32	1.32	1.19
C29/C30	1.39	1.44	1.37	1.33
H31(S)/H31(R)	1.50	1.36	1.35	1.34
H32(S)/H32(R)	1.38	1.36	1.33	1.39
Ts/Tm	0.76	0.71	0.76	0.73
C27αββ/ C29αββ	0.90	0.97	0.75	0.94
H30/(H31+H32+H33+H34+H35)	0.44	0.43	0.43	0.43

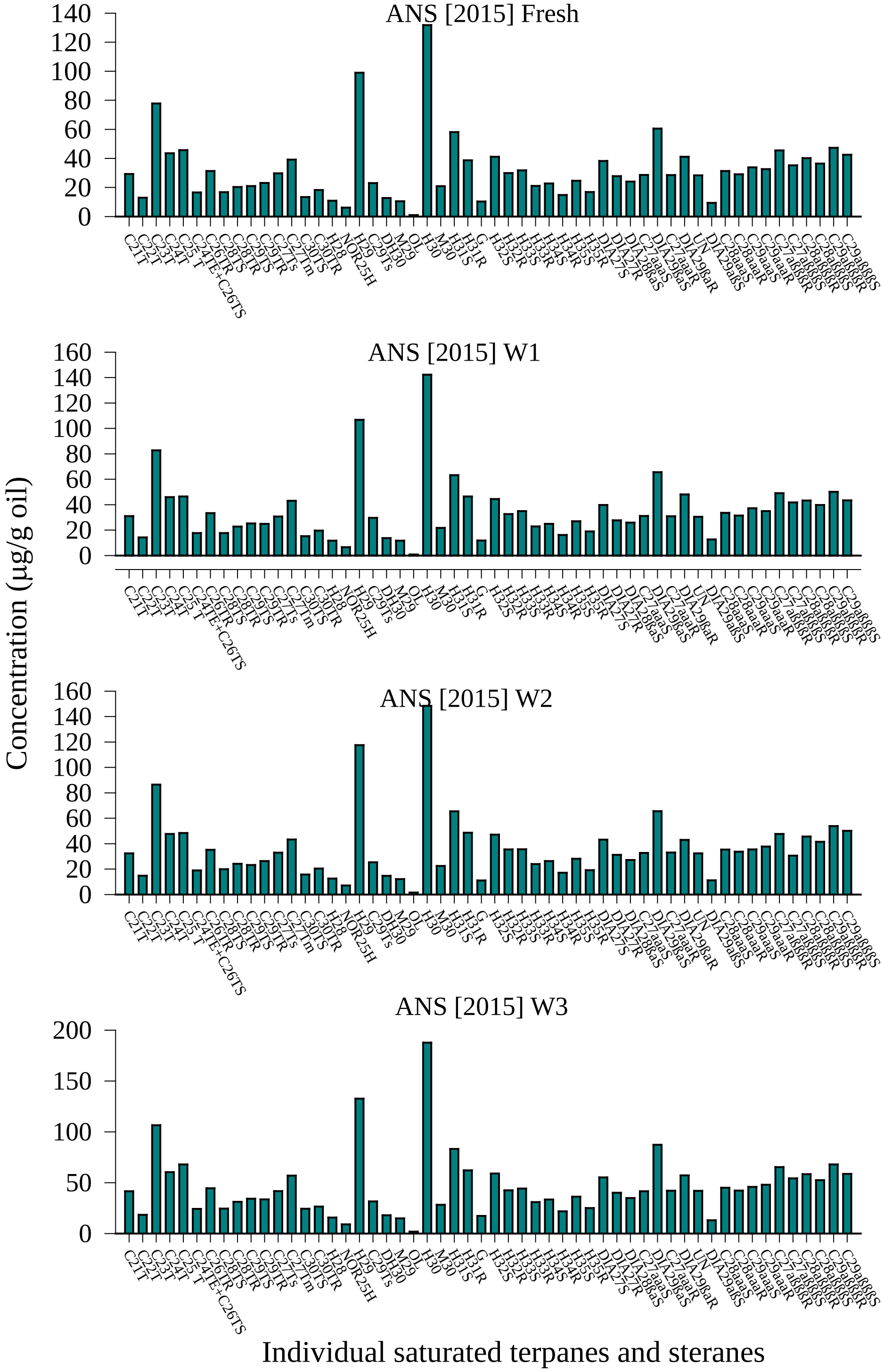

Figure 6 **Distribution profiles of selected petroleum biomarkers (terpanes and saturated steranes)**

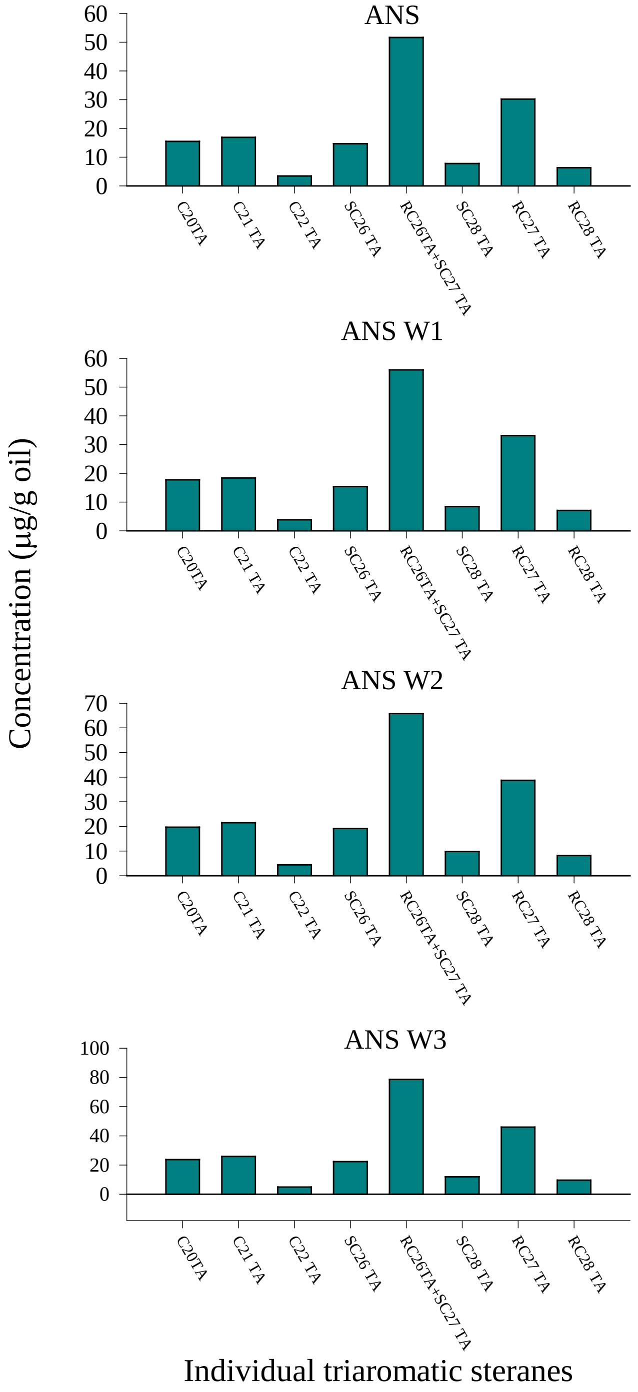

Figure 7 Distribution profiles of triaromatic steranes

Appendix: Analytical Methods

Preparation of Weathered Fraction

A laboratory oil-weathering technique by rotary evaporation is used by Emergencies Science and Technology Division to artificially weather oils with varying degrees of weight loss. The oil weathering system consists of a Buchi R220 rotary evaporator with a 10 L flask, an integral water bath, a circulating bath and a vacuum pump (Buchi Labortechnik AG, Flawil, Switzerland). The water bath in contact with the rotary flask is at ± 80 °C. The rotation speed is set to 135 rpm. The following evaporation procedure is used to evaporate oils:

- The water bath is brought to a temperature of 80 °C.
- The empty rotary flask is weighed, approximately 2 L of oil added and the flask reweighed.
- The flask is mounted on the apparatus and the flask partially immersed in the water bath and spun at 135 rpm. A constant flow of air of 13 L/min through the flask is maintained by the vacuum pump;
- At set intervals the sample flask is removed and weighed. Periodically, a sample of about 1 g is reserved for chemical analysis.

Typically, three weathered fractions are prepared for each oil sample. The initial weathering period is 48 hours, a duration chosen to simulate a heavily-weathered state of an oil in the environment. In addition, intermediate fractions of approximately one- and two-thirds of the 48-hour loss by weight are prepared. The exact time taken to prepare these intermediate fractions is determined by estimation from the measured fractional mass-loss as a function of time for the 48-hour sample. The fraction mass-loss is calculated as:

$$\%weathering = (m_i - m_f) / (m_i - m_e) \text{ x } 100\%,$$

where,

$\%weathering$ is the percentage evaporative mass-loss over the 48 hour period,
m_i is the initial mass of the flask and oil,
m_f is the final mass of the flask and oil, and
m_e is the mass of the empty flask.

A graph of *%weathering* as a function of time is plotted using the interval weighing data. The times for one-third ($t_{1/3}$) and two-thirds ($t_{2/3}$) of the final mass loss are interpolated from the time-weathering graph.

Density

The density of the oil samples, in g/mL, is measured using an Anton Paar Stabinger Viscometer SVM 3000 (Anton Paar, Montreal PQ, Canada). Measurements were performed at 0.0°C and 15.0°C. Method and operator performance is monitored by periodic measurement of a viscosity & density reference standard from from *Paragon Scientific ltd.* Means of triplicate measurements are reported as the sample density. API gravity at 60 °F (15.56 °C) is calculated by extrapolation from these two points.

Dynamic Viscosity

The dynamic viscosities of the samples, in mPa s (cP) are measured using an Anton Paar Stabinger Viscometer SVM 3000 (Anton Paar, Montreal PQ, Canada). Measurements were made at 0.0°C and 15.0°C. The instrument calibration is checked with a viscosity & density reference standard from *Paragon Scientific Ltd.* Means of triplicate measurements are reported as the sample viscosity.

Interfacial Tension and Surface Tension

The interfacial tensions in both fresh (distilled, deionized) water and brine (3.3% w/w NaCl in distilled, deionized water) are determined by a pendent drop technique using a CAM 200 contact angle and surface tension meter (KSV Instruments, Helsinki, Finland). The interfacial tension between oil and water is determined by forming an oil drop in water phase. A J-hook needle is used to form a rising drop of oil sample in the denser water. The surface tension between oil and air is determined by forming a hanging oil drop in air.

The tensiometer records droplet images and automatically analyzes the droplet shape to determine the interfacial/surface tension. The droplet shape is a function of the interfacial tension of the liquid, gravity and the density difference between sample liquid and surrounding medium. In the case of a liquid-liquid interfacial tension, the surrounding fluid must be clear, so that a good image may be generated.

For oil in water this requires that the oil be suspended in water. However, as most oils are less dense than water, a rising oil drop, rather than the pendant drop is measured. In this case, the image is inverted in software and, instead of the force of gravity, the buoyant force, determined as the fraction of gravity based on the specific gravity of the oil is used:

$$b = g\,(\rho_{water} - \rho_{oil})/\,\rho_{water}$$

where b is the buoyant force, g is the acceleration due to gravity, ρ_{water} is the density of water at the measurement temperature and ρ_{oil} is the oil density.

Images are recorded for 10 seconds with a rate of 12 frames per second (10 s × 12 FPS = 120 frames). The images are analyzed with a drop profile fitting method, then averaged to determine the interfacial tension. Triplicate sets of measurements are averaged and the mean reported as the interfacial tension.

Water Content

The mass fraction of water in an emulsion, expressed as a percentage by mass, is determined by Karl Fischer titration using a Metrohm 701 automatic titrator. The method used closely follows ASTM method D 4377. The Karl Fischer reaction is an amine-catalyzed reduction of water in a methanolic solution:

$$CH_3OH + SO_2 + RN \rightarrow [RNH]^+ [SO_3CH_3]^-$$

$$2RN + H_2O + I_2 + [RNH]SO_3CH_3 \rightarrow [RNH]^+ [SO_4CH_3]^- + 2\,[RNH]^+ I^- \quad (7)$$

The amine, RN, or mixture of amines is proprietary to each manufacturer.

A sample of oil is accurately weighed and introduced to the reaction vessel of the auto-titrator. A solution of 1:1:2 (by volume) mixture of methanol: chloroform: toluene is used as a working fluid. The auto-titrator is loaded with 5-mg/mL, pyridine-free Karl Fischer reagent from a certified supplier. Samples are repeated in triplicate and the mean reported as the water percentage. The instrument calibration is checked by a series of five replicate titrations of 25 µL of distilled, deionized water.

Pour Point

Pour point analysis is performed following ASTM DD5949, "Standard Test Method for Pour Point of Petroleum Products Automatic Pressure Pulsing Method". A Phase Technology 70Xi Pour Point Analyzer was used for this analysis. The pour point is measured as the lowest temperature at which the liquid still flows before turn to semi-solid state or becoming adequately viscous and immovable under the test conditions. The detection of pour point in 70Xi Analyzer is performed by shooting a calculated pulse of compressed gas onto surface of the sample. The pulsed gas creates disturbances in the sample and the analyzer's optical sensors detect this movement. Absence of movement indicates that the liquid has reached its pour point. Means of triplicate measurements are reported as the sample pour point.

Flash Point

Flash point analysis is performed following ASTM D7094 using "Modified Continuously Closed Tester". A fully automated Grabner MiniFlash FLP or FLPL Analyzer with a 7 mL test cup with 2 mL sample and the stirring magnet are used for this analysis. The flash point is determined as the lowest temperature corrected to a pressure of 101.3 kPa at which application of an ignition source causes the vapors of a specimen of the sample to ignite momentarily under specified conditions of the test. For the flash point measurement, the system produces high voltage sparks. Then, the system monitors the resulting pressure differential after each spark and after the threshold pressure has been reached or exceeded, the temperature is recorded as the flash point, corrected to standard temperature and pressure conditions and rounded to the nearest whole degree. Means of triplicate measurements are reported as the sample flash point.

Sulfur Content

Sulphur content was determined using an Energy Dispersive X-ray Fluorescence Spectroscopy (ARL Quant'X EDXRF Spectrometer, Thermo Scientific) following ASTM D4294-10. In summary the sample is placed in the beam emitted from an X-ray tube. The resultant excited radiation is measured, and the accumulated count is compared with counts from previously prepared calibration samples to obtain the sulfur concentration in mass %. Means of triplicate measurements are reported as the sample sulfur content.

Vapor pressure

Reid Vapor pressure was measured by Grabner MINIVAP VPXpert instrument using ASTM D323 for non-pressurized crude oils at temperature of 37.8°C.The instrument performs fully automatically with less than 10 mL of samples. To avoid any contamination with the previous sample 7.5 mL sample is used for rinsing the measuring cell. Then, the vapor pressure is automatically determined with a 1 mL sample volume. Using the ASTM D323 method for analysis of non-pressurized crude, the instrument uses the following formula for correction of the obtained vapor pressure:

$$RVPE\ [D323]=0.915*Ptot$$

where Ptot is the total pressure**.**

Adhesion

Oil adhesion is defined as the mass of oil per unit area that will remain on a standard test surface, after ‘dipping and draining’ for 30 minutes.

This method requires the use of an analytical pan balance capable of weighing to 0.0001 g and with the provision for weighing from below the pan. In addition, a standard penetrometer needle adapted for hanging below the balance is required. In summary the adhesion test is performed via following procedure.

The balance is prepared for measurement by hanging a penetrometer needle, for which the surface area of the stainless steel has been calculated, from the balance hook and allowing the weight to stabilize. The weight of the clean needle is zeroed. Approximately 80 mL of samples is poured into a 100 mL beaker. The beaker is elevated, using a lab jack, until the top of the stainless steel needle meets the top of the oil. Care must be taken to avoid having the oil creep up onto the brass section of the needle, as the surface area calculation is based only on the stainless steel portion. The needle is left in the oil for 30 s, and then the beaker is lowered, allowing the needle to hang undisturbed. After 30 min, the weight of the oil on the needle is recorded. The oil adhesion is calculated as the average weight of oil remaining on the needle divided by needle’s surface area. Means of triplicate measurements are reported as the adhesion.

Oil/Brine Emulsion Formation Tendency and Stability

Water-in-oil emulsions are formed in 2.2-litre fluorinated vessels on an end-over-end rotary mixer (Associated Design, VA) at a nominal rotational speed of 50 RPM. Samples are prepared as follows:

- A 600-mL volume of salt water (3.3% w/v NaCl) is dispensed into a mixing vessel.
- A 30-mL aliquot of oil is added to each vessel for a 1:20 oil:water ratio.
- The vessels are sealed and placed in the rotary mixer such that the cap of each mixing vessel follows, rather than leads, the direction of rotation. The rotary mixer is kept in a temperature controlled cold room at 15 °C .
- The vessels and their contents are allowed to stand for approximately 4 hours before rotation begins, then mixed continuously for 12 hours.

- At the conclusion of the mixing time, the emulsions are collected from the vessels for measurement of water content, viscosity and the complex modulus. The emulsions are stored in the cold room at 15 °C for one week, then observed for changes in physical appearance.

Emulsions are classified into one of four stability classes: unstable, entrained water, meso-stable and stable.

Water content for the emulsions is measured using the method described above.

The complex modulus is measured on an RS300 RheoStress rheometer using a 35 mm plate-plate geometry. A stress sweep is performed in the range 0.1 to 10 mPa in the forced oscillation mode at a frequency of 1 Hz. The complex modulus value in the linear viscoelastic region is reported.

Chemical Dispersibility

Swirling Flask (ASTM F2059)

Chemical Dispersibility is assess as a relative ranking of effectiveness for the dispersibility of an oil sample by mixture with the surfactant Corexit 9500A. This method follows closely ASTM F2059-06.

A pre-mix of 1:25.0 dispersant:oil is made up by adding oil to 100mg of dispersant (approximately 2.50mL of oil in total).

Six side-spout Erlenmeyer flasks, as described in ASTM F2059, containing 120 mL of 33‰ brine are placed into an incubator-shaker. An aliquot of 100 µL of premix is added to the surface of the liquid in each flask, care being taken to not disturb the bulk brine. The flasks are mechanically shaken at 20.0 □C with a rotation speed of 150 rpm for exactly 20 minutes. The solutions are allowed to settle for 10 minutes.

Samples are extracted into 70:30 (v:v) dicholoromethane/pentane then analysed using a GC/FID to determine the oil concentration in the solvent. Total petroleum hydrocarbon content of the sample is quantified by the internal standard method.

The TPH values are calibrated using a series of six oil-in-solvent mixtures prepared from the premix for each oil. The volume of premix dispersant/oil solution for each standard is selected to represent a percentage efficiency of the dispersed oil. The volume of the premix is then carefully applied to the surface of the brine in a shaker flask and shaken exactly as one of the samples, as described previously. Upon removal from the shaker however, the entire contents of the flask is transferred to the separatory funnel. This is extracted with 70:30 (v:v) dichloromethane:pentane.

The TPH values are fitted to %effectiveness for the calibration standards are plotted. The sample TPH values are then used to determine the %effectiveness of the dispersant.

Baffled Flask

The Baffled Flask test was performed in a temperature-controlled room held at 15 ± 0.5 °C. A 120 mL volume of 3.3% aqueous sea salt (Sigma Aldrich) solution is added to twelve capped Baffled Flasks (150 mL trypsinizing flasks modified by a stop-cock port at the base). Six of the flasks are for samples and the other six serve as the calibration set. Three of each are run in a batch of six, two batches per experiment.

In the six sample flasks, a 100-µL volume of oil pre-mixed with dispersant at a 1:25 (w/w) dispersant-to-oil ratio was deposited on the surface of the water. The flasks were then mounted on an oscillatory table with 2.5 cm orbit and mixed for 10 minutes at 200 RPM, then the flasks were allowed to stand for 10 minutes to settle. A 30 mL sample of the oil/water mixture was then collected from the bottom of the flask and extracted in a 125mL separatory funnel with 3 x 5 mL volumes of dichloromethane, collected in a graduated cylinder, and made up to 15.0 mL.

The extract was analyzed by gas chromatography with flame ionization detector (GC-FID) to determine the total GC-detectable hydrocarbons (TPH). The set of six calibration flasks were treated in the same manner as the sample set, but the pre-mixed oil and dispersant were added at a percentage fraction of the volume of the sample set (e.g. 60-µL = 60%) in steps of 20%; 20, 60 and 100% are run in one batch, and 0, 40 and 80% in the other. Following mixing and settling, the full volume of each of the calibration set was extracted with 3 x 20 mL of dichloromethane and corrected to 60.0 mL, quantitatively transferring all oil into the extract. The extract was analyzed by GC-FID for TPH.

A linear fit of TPH against %Effectiveness for the calibration set then provided a means to determine dispersant effectiveness of the test samples by substituting the calculated TPH value.

Evaporation Equation

The evaporation kinetics, reported as percentage mass loss (%Ev) as a function of time, are determined by measuring the weight loss over time from a shallow dish. Approximately 20 g of oil is weighed into a 139 mm petri dish. Measurements are conducted in a low-flow climate-controlled chamber at 15 °C. Temperatures are monitored by a digital thermometer. The oil weight is recorded by an electronic balance accurate to 0. 01 g at intervals and collected on a computer logging system. The evaporation period can last from a few days for light oils to weeks for heavier products. The time versus weight loss data series are fitted to either a square-root or a natural logarithm function is chosen as the simplest, most representative equation for evaporative mass loss. The best-fit equations have the form:

$$\%Ev = (\mathrm{A} + \mathrm{B}\,(T\text{-}15)) \ln t, \text{ and}$$
$$\%Ev = (\mathrm{A} + \mathrm{B}\,(T\text{-}15))\, t^{1/2}$$

where:

$\%Ev$ is the quantity of oil evaporated, as a percentage of the starting oil mass,
T is the oil surface temperature (°C),
t is time (in minutes), and
A and B are constants fitted to the measured data.

The relationship for the *A* and *B* constants are defined using the methods of Fingas, 2004. The apparatus is periodically checked by conducting a run with a 15-g mass in place of the oil.

Fingas, M.F., 2004, "Modeling evaporation using models that are not boundary-layer regulated", *Journal of Hazardous Materials*, 107 (1-2), pp. 27-36.

Chemistry Methods:

Simulated Distillation

A simulated distillation curve was measured according to ASTM D7169, by Intertek Commodities Division, Montreal, PQ.

Hydrocarbon Groups Fractional Analysis

Asphaltenes are precipitated from the original oil by using *n*-pentane in excess as the precipitating medium (25 ml *n*-pentane per gram of oil). The mixture is filtered through a pre-weighed 0.45 µm pore filter. The filter residue (asphaltenes) is rinsed with *n*-pentane, and then transferred with the filter to a weighing boat and dried in a desiccator. Mass is measured to calculate the asphaltene content.

The filtrate fluid (the “maltenes”) is rotary evaporated at low pressure to remove the solvent, and then quantitatively transferred into a 100 ml volumetric flask and diluted up to mark using n-pentane. 30 g of activated silica gel is packed into a column and topped off with 1 cm anhydrous Na2SO4. 5.00 ml aliquot of diluted maltene is pipetted into the column. 180 ml of 1:1 benzene/hexane mixture is used to elute the saturates and aromatics. Resins are then eluted with 100 ml methanol, followed by 100 ml dichloromethane and collected in a 500 ml boiling flask. The collected resin portion is rotary evaporated and then quantitatively transferred to a pre-weighed amber vial, and the content in the vial is blown down to dryness under nitrogen. Resin content is calculated by measuring the mass, then applying the dilution factor.

Waxes are separated by crystallization in polar solvent. The remaining diluted maltene after resin quantification (95.0 mL) is rotary-evaporated to remove the solvent and quantitatively transferred into a 250 mL Teflon Erlenmeyer flask, using 50 mL of 1:1 (v:v) dichloromethane (DCM)/Methyl Ethyl Ketone (MEK). The Teflon flask, Buchner funnel, 125 mL filtering flask, and a squeeze bottle of 1:1 DCM/MEK are placed in the -30 °C fridge to chill overnight, and for wax crystallization. The contents of the Teflon flask are filtered. The wax fraction retained on the filter is dried in the desiccator until a constant mass is achieved. Mass of the recovered waxes is measured to calculate the wax content (correction factor= (100/95).

Saturate and aromatic fractions are determined by estimation using the amounts of GC-saturate and GC-aromatics relative to the GC-TPH, respectively. The final percentages are computed by difference from the remainder of the oil with the asphalt and resin fractions removed.

Chemical compositional analysis.

Sample Preparation

Prior to use, all clean glassware was rinsed with acetone, DCM and hexane. The concentrated solvent blank was analyzed by GC/FID and GC/MS. The analysis results demonstrated the glassware was clean and target-analyte free.

The oil samples were transferred by dissolving about 800 mg of oil in 10 mL of hexane to a final stock solution with around 80 mg/mL concentration. A 200 µL aliquot of the stock solutions was spiked with surrogates (100 µL 200 ppm of *o*-terphenyl, 100 µL of 200 ppm of tetracosane-*d*50 ($C_{24}D_{50}$) and 100 µL of mixture of deuterated naphthalene, acenaphthene, phenanthrene, benz[a]anthracene, and perylene, 10 µg/mL each), and then quantitatively transferred into a 3-gram of silica gel chromatography column for sample cleanup and fractionation.

Hexane (12 mL) and 50% DCM in hexane (v/v, 15 mL) were used to elute the saturated and aromatic hydrocarbon fractions, respectively. These two fractions were concentrated under a gentle stream of nitrogen to appropriate volumes, spiked with appropriate internal standards (IS), (including 5-α-androstane, C30-ββ-hopane, d_{18}-decahydronaphthalene and d_{14}-terphenyl for TPH, alkanes, biomarkers and PAHs analysis, respectively), and then adjusted to an accurate pre-injection volume of 1.00 mL for GC/MS and GC/FID analyses.

For each sample, the hexane-eluted fraction (labeled as saturated fraction) was used for analysis of aliphatic hydrocarbons, including total GC-detectable total saturated hydrocarbons (TSH), alkanes, and biomarker of terpanes, saturated steranes, and decalines; the 50% DCM fraction (labeled F2) was used for the analysis of GC-detectable total aromatic hydrocarbons (TAH), alkylated homologous PAHs, other un-substituted PAHs and triaromatic steranes. The fraction eluted from the mixture of the hexane and the 50% DCM (1:1, v: v) were combined (labeled as the aromatic fraction) and used for determining the total GC-detectable total petroleum hydrocarbons (TPH) and the unresolved complex mixture of hydrocarbons (UCM).

For quality control and statistical analysis, ESTS reference oil Prudhoe Bay crude oil (13. 1% weathered) was used as the reference oil standard.

Instruments and Sample Analysis

The TPH (total petroleum hydrocarbons) concentration (n-C_8 through n-C_{50}) was determined on an Agilent 6890 GC-FID and an Agilent 7683 auto sampler. The system control and data acquisition was achieved with an Agilent ChemStation. A DB-5HT fused silica column (30 m × 0.25 mm id, 0.10 µm film thickness) was used. The carrier gas was hydrogen at 1.5 mL/min. The injector and detector temperature were set at 290 and 300°C, respectively. The temperature program used for TPH determination is the following: 40°C for 2 minutes, ramp at 20°C/min to 340°C, and held for 17 minutes.

Analyses of target *n*-alkanes, PAHs, and biomarker compounds were performed on an HP 6890 GC equipped with a HP 5973 mass selective detector (MSD). The GC separation was achieved using a DB-5MS capillary column (30 m × 0.25 mm ID × 0.25 µm film thickness) with a GC oven temperature program: 50°C for 2 min, heated to 300 °C at 6 ºC/min and held for 15 min at 300 °C. Samples were injected in splitless mode (injector temperature at 280°C) with helium as carrier gas. The MSD was operated in the selected ion monitoring (SIM) mode. System control and data acquisition were achieved with the Agilent Enhanced MSD ChemStation. The carrier gas was helium at 1.0 mL/min.

Aliphatic hydrocarbons including alkanes (n-C_9 to n-C_{40}), and biomarkers (terpanes, steranes and decalines) were identified and quantified in the saturate fraction. Parent PAHs including biphenyl (Bph), naphthalene (N), acenaphthylene (Acl), acenaphthene (Ace), dibenzofuran (DBF), fluorene (F), phenanthrene (P), anthracene (An), fluoranthene (Fl), pyrene (Py), benzo[b]fluorine (BbF), benzo(a)anthracene (BaA), chrysene (C), benzo(b)fluoranthene (BbFl), benzo(k)fluoranthene (BkFl), benzo(a)pyrene (BaP), indeno(1, 2, 3-cd)pyrene (IP), dibenzo(a, h)anthrathene (DA), and, benzo (e) pyrene (BeP) and perlyene (Pe), benzo(g, h, i)perylene (BgP), and seven alkylated PAH families (including some individual APAHs, see Table 6 for the detailed information) were quantified in the aromatic fraction. The seven quantified APAH families include naphthalenes (Ci-N), phenanthrenes (Ci-P), dibenzothiophenes (Ci-D), fluorenes (Ci-F), fluoranthenes (Ci-Fl), chrysenes (Ci-C), and benzonaphthothiophenes (Ci-B), where i=(0, 1, 2, 3, or 4) indicates the degree of alkylation. Additionally, representative triaromatic steroids were identified and quantified in the aromatic fraction (Table 8).

Quality Assurance

Measurements were performed for a minimum of three replicates. The minimum precision of the analysis was set for acceptable range of repeatability based on the corresponding ASTM method where applicable.