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Qualitative and Quantitative Compositional Analysis of Shale Derived by Gas Chromatography and Combined Gas Chromatography/Mass Spectrometry

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INTRODUCTION

As part of its program to insure uninterrupted supplies of diesel marine fuel, The Navy Alternate Fuels Program evaluates the performance characteristics as well as the chemical and physical characteristics of synfuels. Incumbent in this series of evaluations is the need for qualitative and quantitative compositional data for both liquid conventional and synthetic DFM's and their more volatile or headspace components. These data can permit a theoretical assessment of the potential toxicological, environmental, and hygiene impact that various synfuels may have on Navy personnel and their surroundings. For example, analysis of the fuel headspace at various practical temperatures can be used to predict workplace concentrations for individual fuel components, both benign and potentially toxic. Simple modifications of the methods employed in this work can permit direct comparisons of several conventional and synfuels for their headspace volatiles.

This report summarizes results for a shale derived DFM (Sohio). More detailed experimental descriptions and data compilations are presented in the companion reports.

OBJECTIVES

The objectives of this work were several. (1) Determine the qualitative and quantitative composition of the headspace volatiles at various temperatures for this shale derived DFM. (2) Relate these data to the bulk liquid fuel composition. (3) Evaluate the bulk liquid and headspace components for potential toxicological problems as related to exposure of Navy personnel. (4) Extract from methodology used and developed in this program, a reliable, inexpensive, and representative acceptance protocol with which to evaluate individual lots of conventional DFM and synfuel DFM for volatiles.

METHODS

Because of the very complex nature of the samples, compound separation was effected by fused silica capillary column gas chromatography. Detection of individual components was accomplished with a flame ionization detector or a mass spectrometer. The performance characteristics of this instrumentation permit detection limits of 100 ppb and lower for individual components in the headspace and bulk fuel samples. A complete discussion of the experimental methods is found in the companion reports.

RESULTS AND DISCUSSION

The accompanying tables summarize the results for the identification and quantification of headspace volatiles for the shale derived DFM (Sohio W375). Summation of the values for the individual components yields total hydrocarbon values of approximately 987 ppm (3447 mg/M^3 as hexane) for 90° F experiments and 2262 ppm (7894 mg/M^3 as hexane) for 120° F experiments. Quantitative measurements are presented for each identified compound at the two experimental temperatures. These values can in turn be related to concentrations for the individual species in the liquid fuel.

The experimental set-up employed in this program differs slightly from previous ones. A 3-liter round bottom flask was charged with 8.248 grams (10 ml.) of fuel and sealed with a septum. The entire flask was immersed in a stirred water bath thermo-statted at the appropriate temperature. Liquid fuel and headspace vapors in the flask were also stirred during the entire sampling process. This protocol was adopted for several reasons. (1) The simplicity of the set-up will allow a routine QC test for volatiles from different fuels (or from different lots of the same fuel) to be easily implemented. Results to date strongly suggest side by side comparative tests under standardized conditions will produce the best data. This experimental set-up, coupled with capillary column gas chromatography (flame ionization detection) provides an effective and inexpensive means of quickly evaluating DFM's. (2) We were quite concerned about pressure build up in the closed system producing false results. This set-up was monitored with a differential manometer and found to exhibit no rise in pressure at 120° F. (3) Expanding the volatiles from a known quantity of fuel into a known volume should make an extrapolation to the shipboard environment more straightforward. It must be re-emphasized that the measured values from this study are likely lower than those obtained for this fuel using previous set-ups. We feel these measurements are equal to or even superior to previous ones as regards their utility in predicting workplace levels. Direct comparisons on the same experimental apparatus will provide the best information.

Inspection of the individual compounds and their concentrations revel a few noteworthy facts. Concentrations of benzene and hexane are comparable to those determined in previous studies. Furthermore, the relative levels of cyclic hydrocarbons compared to open chain olefins is higher than expected.

This is perhaps the result of extensive hydrogenation. The classes of compounds not present in the headspace of this fuel are equally interesting. Extensive searches were conducted for phenols, nitrosoamines,halogenated compounds, aldehydes, ketones, thiols, and nitro compounds among the volatiles. No potentially toxic members of these classes were detected. The situation in the bulk liquid fuel is similar. Normal alkanes, cyclic alkanes, olefins, and alkyl aromatics are the dominant species. Their concentrations as ppm hexane were measured and are tabulated in the companion report. Direct analysis failed to detect polyaromatic hydrocarbons larger than three rings (anthracene/phenanthrene). Class searches for those previously noted were negative. When the fuel was subjected to simple open column chromatographic separation, the polyaromatic hydrocarbon fraction was concentrated and separately analyzed. Several PAH's were detected at concentrations in the low parts per billion on a fuel basis. None of the more mutagenic PAH derivatives (nitro, hydroxy, dinitro) were found within the analysis detection limits (approximately 100 parts per trillion). A closer examination of the tabular data reveals additional salient facts. Among the headspace volatiles, greater than 50% of the total are normal or branched alkanes. Cyclic alkanes constitute a surprisingly high percentage (approximately 28%) of the total. (It should be pointed out that the high percentage of cyclics was verified by two independent means). Aromatics and olefins comprise the remaining 16% and 5% respectively. These relative concentrations change very little on going from 90° F to 120° F. It also appears bulk liquid component concentrations and the measured headspace concentrations are self consistant. This suggests bulk fuel measurements could be used to approximate headspace concentrations under various pressure volume conditions. Some examples will support the point. (1) Normal heptane was found in the bulk fuel at a concentration of 265.9 ng/ul (2659 ug in the 10 ml flask charge). If the average molecular weight of the fuel is estimated to be 198, the mole fraction for heptane is 6.384×10^{-4} in the fuel. The vapor pressure for heptane at 120° F is approximately 120 MM Hg leading to a calculated partial pressure of 7.66×10^{-2} MM Hg in the 3 l. flask. This translates to a calculated vapor phase concentration of 1140 ug/heptane in the 3 l. flask. The measured value for heptane at 120° F was 285.2 ug/M^3 or 855.9 ug heptane in the 3 l. flask vapor. This compares favorably with the estimate of 1140 ug. (2) Similar calculations for toluene lead to an estimate of 598

ug toluene in the 3 l. flask, based on a measured concentration of 186.3 ng/ul of the bulk fuel. This also compares favorably with the measured headspace concentration of 185.8 ug/M^3 or 557.4 ug in the 3 l. flask vapor.

Attached to the end of this summary are representative chromatograms for the headspace and bulk fuel analyses. Simple inspection reveal the chromatograms for the volatiles are quite different from those of the bulk liquid. More than 95% of the 90° F volatiles and 85% of the 120° F volatiles are composed of compounds which themselves comprise less than 16% of the bulk fuel. Within this framework it is difficult to relate the concentration of the headspace volatiles as a concentration of diesel fuel, since they are so dissimilar in character. The more volatile materials for example toluene, are 100 times more concentrated in the headspace compared with bulk liquid.

CONCLUSION

Direct comparisions of individual synfuels on a simple apparatus such as described herein is the most expedient means of evaluating the abundance and character of their headspace volatiles. Few toxicologically significant compounds were found in these studies and their concentrations, coupled with knowledge of air exchanges, can be used to estimate shipboard levels. Examination of the bulk liquid also revealed few compounds of concern at very low concentrations.

Headspace Volatiles - Identification and Quantitation

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
1	0.3	1.1		0.2	0.8	carbon dioxide
2	2.5	8.7		1.5	5.2	propane
	0.1	0.3		0.1	0.3	carbonyl sulfide
	0.1	0.3		0.1	0.3	sulfur dioxide
3	4.1	14.3		3.1	10.9	2-methyl propane ,methanol (10%)
4	0.3	0.9		0.3	0.9	1-butene
5	6.9	24.1		8.1	28.2	n-butane
6	0.1	0.3		0.1	0.3	cis & trans 2-butene
7	0.1	0.3		0.1	0.3	cyclobutane
8	0.1	0.3		0.1	0.3	3-methyl 1-butene
9	0.6	2.0		0.1	0.3	acetone
10	9.2	31.3		13.2	46.3	2-methyl butane
11	0.3	1.0		0.4	1.4	1-pentene
12	0.1	0.3		0.1	0.3	3-methyl-1-pentene
13	7.5	26.2		11.8	41.1	n-pentane
14	0.1	0.3		0.1	0.3	cis-2-pentene
15	0.1	0.3		0.1	0.3	trans-2-pentene
16	0.2	0.8		0.4	1.3	2-methyl-2-butene , carbon disulfide (5%)

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification
17	1.0	3.3	1.3	4.6	2,2 dimethyl butane
18	not detected		0.1	0.3	cyclopentene
19	0.3	1.1	0.4	1.3	4 methyl 1-pentene
20	3.8	13.3	4.6	16.2	2,3 dimethyl butane
	0.1	0.3	0.2	0.7	cyclopentane
21	13.5	47.1	16.6	58.1	2-methyl pentane
22	not detected		0.1	0.3	C_7H_{16} isomer
23	10.5	36.5	13.2	46.3	3-methyl pentane
24	0.4	1.4	0.5	1.8	1-hexene
25	26.0	90.9	35.1	122.8	n-hexane
26	0.1	0.3	0.1	0.3	C_6H_{12} isomer
27	0.2	0.6	0.3	0.9	2-butene, 2,3 dimethyl
28	0.1	0.3	0.1	0.3	2-methyl, 2-pentene , C_6H_{10} isomer (10%)
29	not detected		0.1	0.3	C_6H_{12} isomer & methoxyethanol
30	0.1	0.3	0.2	0.7	3-methyl, 2-pentene
31	0.7	2.4	1.0	3.3	2,2,3 trimethyl butane

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification
32	9.8	34.3	13.6	47.7	methyl cyclopentane
33	2.2	7.8	3.2	11.1	2,4 dimethyl pentane
34	0.1	0.3	0.2	0.5	C_7H_{16} isomer
35	10.0	34.9	15.7	54.7	benzene
36	0.6	2.3	1.0	3.5	3,3 dimethyl pentane
37	16.1	56.2	24.5	85.8	cyclohexane
38	not detected		0.2	0.6	C_7H_{14} isomer
39	14.7	51.5	23.3	81.4	2-methyl hexane, C_7H_{14} isomer (5%)
40	11.8	41.3	18.6	65.0	2,3 dimethylpentane
41	0.6	2.1	1.1	3.6	1,1 dimethyl cyclopentane
42	32.8	114.4	54.6	190.9	3-methyl hexane
43	3.7	12.9	5.9	20.5	1,3 dimethyl cyclopentane (cis/trans)
44	6.1	21.4	9.9	34.5	1,3 dimethyl cyclopentane (cis/trans)
45	6.9	24.1	11.2	39.2	1,2 dimethyl cyclopentane-trans
46	not detected		0.1	0.3	1,2 dimethylcyclopentane-cis
47	64.6	225.9	81.6	285.2	n-heptane

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
48	0.1	0.3		0.2	0.5	C_7H_{14} isomer
49	0.2	0.5		0.3	0.9	1-pentene,2,3 dimethyl
50	0.2	0.5		0.3	0.9	C_7H_{14} isomer
51	47.0	164.3		81.6	285.2	methylcyclohexane
52	0.8	2.9		1.5	5.2	1,1,3 trimethylcyclopentane
53	8.4	29.5		15.2	53.0	ethylcyclopentane
54	7.4	26.1		13.7	48.0	2,4 dimethylhexane
55	3.3	11.7		6.1	21.4	1,2,4 trimethylcyclopentane, C_8H_{18} (30%)
56	2.6	4.8		4.8	16.9	1,2,3 trimethylcyclopentane
57	1.0	3.7		1.9	6.8	1-hexene,4,5 dimethyl
58	28.4	99.3		53.2	185.8	toluene
59	4.7	16.6		9.6	33.5	2,3 dimethylhexane
60	4.8	16.7		9.9	34.5	3-ethyl-2-methyl pentane
61	23.5	82.2		49.4	172.8	2-methyl heptane
62	9.2	32.0		19.2	67.3	4-methyl heptane
63	3.9	13.6		7.7	27.0	3,4 dimethylhexane

Headspace Volatiles- continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
64	20.4	71.4		39.7	138.8	3-methylheptane
65	5.1	17.8		11.5	40.3	C_7H_{14} isomer
66	13.1	45.8		26.2	91.6	trans-1,2 dimethylcyclohexane
67	3.5	12.1		7.3	25.5	trans-1,4 dimethylcyclohexane
68	1.4	4.8		2.7	9.6	1,1 dimethylcyclohexane
69	3.0	10.3		5.9	20.5	trans-1-ethyl-3-methylcyclopentane
70	2.1	7.1		4.0	13.8	cis-1-ethyl-3-methylcyclopentane
71	5.7	19.8		11.5	40.1	cis-1-ethyl-2-methylcyclopentane
72	not detected			0.3	1.2	C_7H_{14} isomer
73	8.6	29.9		17.2	60.1	dimethylcyclohexane isomer
74	46.6	163.0		98.9	345.9	n-octane

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification
75	8.9	31.2	17.0	59.6	dimethylcyclohexane isomer
76	0.2	0.8	0.5	1.6	1,4 pentadiene,2,2,3 trimethyl
77	1.4	5.0	2.9	10.3	cyclopentane-(1-methylethyl)
78	not detected		0.3	0.9	1,1,3,4-tetramethylcyclopentane
79	not detected		0.4	1.5	tetramethylcyclopentane isomer
80	0.4	1.4	0.5	1.9	4,4,dimethylheptane
81	2.2	7.7	4.6	16.2	cis-1-ethyl-2-methylcyclopentane
82	1.7	6.1	3.7	13.0	C_8H_{14} isomer
83	5.6	19.4	13.0	45.6	2,4 dimethylheptane
84	0.2	0.6	0.4	1.5	Silicone
85	21.9	76.4	47.8	167.1	Propylcyclopentane

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
86	0.9	3.2		2.2	7.6	cyclic C_9H_{18} isomer
87	20.3	71.0		45.3	158.4	1,1,3 trimethyl cyclohexane
88	0.6	2.1		3.0	10.4	C_9H_{18} isomer
89	0.7	2.4		1.6	5.5	C_9H_{18} isomer
90	0.8	2.7		1.7	5.8	cyclic C_9H_{18} isomer
91	not detected			1.8	6.3	unknown
92	10.4	36.8		22.9	80.0	dimethylbenzene (xylene) isomer
93	4.3	15.1		9.7	33.8	1,3,5 trimethylcyclohexane
94	1.0	3.5		2.2	7.6	dimethylcyclohexane isomer
95	16.3	56.8		37.1	129.6	dimethylbenzene (xylene)isomer or ethylbenzene
96	8.9	31.1		20.3	71.1	dimethylbenzene (xylene)isomer or ethylbenzene

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
97	1.6	5.6		3.2	11.3	3-ethyl-4-methyl hexane
98	2.0	6.9		5.0	17.4	C_8H_{14} isomer
99	1.3	4.7		2.9	10.3	C_9H_{18} isomer cyclic
100	4.9	17.1		12.7	44.3	2-methyl octane
101	4.2	14.7		8.7	30.6	C_9H_{18} isomer olefinic
102	2.7	9.4		4.7	16.4	1-ethyl-3-methyl cyclopentane (cis/trans)
103	10.7	37.4		25.9	90.5	olefinic C_9H_{18} isomer
104	2.6	9.1		6.1	21.3	trimethylcyclohexane isomer
105	10.0	35.0		23.7	82.9	dimethylbenzene (xylene) isomer or ethyl benzene, & trimethylcyclohexane isomer (15%)
106	2.2	7.7		5.3	18.5	trimethylcyclohexane isomer
107	2.4	8.4		5.9	20.6	olefinic $C_{10}H_{20}$ isomer

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F ppm	120°F mg/m^3	Identification
108	4.1	14.3	10.1	35.3	ethyl-methyl cyclohexane isomer
109	1.8	6.4	4.6	16.0	ethyl-methyl cyclohexane isomer
110	1.1	3.8	2.6	8.9	cyclic C_9H_{18} isomer
111	0.8	2.8	2.0	7.1	cyclic C_9H_{18} isomers
112	31.1	108.6	80.3	280.6	n-nonane & C_9H_{18} isomer (5%)
113	4.2	14.6	9.9	34.6	ethyl-methyl cyclohexane isomer olefinic $C_{10}H_{20}$ isomer (20%)
114	1.0	3.6	2.4	8.4	ethyl-methyl cyclohexane isomer
115	0.5	1.6	1.1	3.9	1-methylethyl benzene
116	1.3	4.7	3.4	11.9	C_9H_{16} isomer & $C_{10}H_{18}$ isomer (10%)
117	0.5	1.8	1.3	4.6	propyl cyclohexane & $C_{10}H_{20}$ olefinic isomer (10%)
118	2.4	8.4	5.2	18.2	olefinic $C_{10}H_{20}$ isomer

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
119	1.2	4.0		3.2	11.3	olefinic $C_{10}H_{20}$ isomer
120	not detected			0.4	1.4	$C_{10}H_{20}$
121	0.2	0.6		0.6	2.0	unknown
122	1.8	6.3		4.8	16.8	C_9H_{18} isomer & $C_{10}H_{20}$ isomer (40%)
123	5.4	19.0		15.3	53.5	C_3 alkyl cyclohexane (propyl or isopropyl)
124	1.7	6.0		6.3	22.0	butylcyclopentane isomer & olefinic $C_{10}H_{20}$ (40%)
125	6.9	24.1		18.0	62.9	$C_{10}H_{22}$ isomer
126	3.3	11.6		6.9	24.1	C_3 alkyl benzene & $C_{10}H_{20}$ isomer (15%)
127	6.3	22.1		16.2	56.7	$C_{10}H_{22}$ isomer & $C_{10}H_{20}$ isomer (18%)
128	1.0	3.4		1.4	4.8	olefinic $C_{10}H_{20}$ isomer
129	5.3	18.6		16.4	57.6	C_3 alkyl benzene

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
130	3.6	12.6		9.2	32.1	C_3 alkyl benzene
131	9.5	33.2		24.5	85.6	$C_{10}H_{20}$ isomer cyclic
132	0.9	3.3		2.5	8.6	C_3 alkyl benzene
133	1.4	4.9		4.1	14.3	$C_{10}H_{20}$ isomer cyclic $C_{10}H_{20}$ isomer olefinic(40%)
134	4.7	16.5		12.2	42.7	$C_{10}H_{22}$ isomer
135	5.2	18.3		13.9	48.7	C_3 alkyl benzene
136	1.4	5.1		3.7	13.0	$C_{10}H_{22}$ isomer
137	3.8	13.1		9.5	33.3	$C_{10}H_{20}$ isomer
138	4.8	16.8		14.0	49.0	propyl methyl cyclohexane isomer (35%) diethyl cyclohexane isomer, $C_{10}H_{22}$ isomer(20%)
139	2.5	8.6		5.6	19.6	olefinic $C_{10}H_{20}$ isomer
140	0.5	1.9		1.4	4.9	cyclic $C_{10}H_{20}$ isomer

Headspace Volatiles - continued

Peak #	Concentration 90°F ppm	90°F mg/m^3	(hexane)	120°F ppm	120°F mg/m^3	Identification
141	8.0	27.8		20.0	70.0	C_3 alkyl benzene & $C_{10}H_{20}$ isomer (10%)
142	2.4	8.4		7.2	25.2	propyl methyl cyclohexane isomer
143	2.5	8.6		3.4	11.9	olefinic $C_{10}H_{20}$ isomer
144	3.7	13.0		11.5	40.1	butyl cyclohexane isomer
145	1.1	3.8		2.7	9.5	olefinic $C_{10}H_{20}$ isomer
146	1.9	6.7		5.3	18.4	cyclic $C_{10}H_{20}$ isomer
147	0.5	1.8		1.3	4.6	diethylcyclohexane isomer
148	1.8	6.3		4.7	16.6	C_4 alkyl benzene isomer & methyl propyl cyclohexane isomer (40%)
149	31.1	108.6		91.4	319.4	n-decane
150	0.8	2.9		2.6	9.1	C_4 alkyl benzene isomer & methyl propyl cyclohexane isomer (35%)
151	5.4	18.8		15.0	52.5	C_3 alkyl benzene isomer & $C_{10}H_{20}$ isomer (10%)

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
152	1.0	3.4		3.4	11.8	C_3 alkyl benzene & $C_{10}H_{20}$ isomer (50%)
153	not detected			1.7	6.1	C_3 alkyl benzene & olefinic $C_{10}H_{20}$ isomer (40%)
154	3.0	10.3		7.2	25.1	C_9H_{10} isomer (indane or propenyl benzene) & two $C_{10}H_{20}$ isomers (15% & 5%)
155	not detected			1.7	5.8	$C_{10}H_{20}$ isomer
156	7.8	27.3		23.4	81.8	$C_{11}H_{24}$ isomer & C_3 alkyl benzene (30%)
157	not detected			1.1	3.7	$C_{11}H_{24}$ isomer & $C_{10}H_{20}$ isomer (40%)
158	4.3	15.1		10.5	36.8	cyclic $C_{10}H_{20}$ isomer
159	1.7	5.9		4.3	15.0	olefinic $C_{10}H_{20}$ isomer & $C_{10}H_{18}$ isomer (20%)
160	2.0	6.9		5.4	18.8	C_3 alkyl benzene isomers (2)(10% &60%) $C_{11}H_{22}$ isomer
161	3.5	12.3		8.4	29.3	C_3 alkyl benzene isomer & $C_{11}H_{22}$ isomer (50%)
162	1.5	5.2		4.3	14.9	C_3 alkyl benzene isomer & $C_{11}H_{22}$ isomer(30%), & $C_{11}H_{24}$ isomer (5%)

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification
163	1.5	5.4	4.1	14.3	C_3 alkyl benzene isomers (2), $C_{11}H_{22}$ isomer(10%)
164	0.9	3.2	2.9	10.0	$C_{10}H_{18}$ isomer
165	3.2	11.0	8.7	30.5	C_3 alkyl benzene isomer
166	1.0	3.4	2.4	8.6	$C_{11}H_{22}$ isomer
167	0.9	3.1	2.2	7.8	$C_{11}H_{24}$ isomer & $C_{11}H_{22}$ isomer
168	3.7	12.8	12.0	42.0	$C_{11}H_{24}$ isomer & trace $C_{11}H_{22}$
169	2.7	9.3	7.9	27.8	C_3 alkyl benzene isomer & methyl indane (40%)
170	1.1	3.9	4.3	15.0	$C_{11}H_{24}$ isomer
171	2.5	8.6	14.7	51.3	methyl indane & $C_{11}H_{20}$ isomer (25%)
172	not detected		1.8	6.4	C_3 alkyl benzene isomer
173	4.0	13.9	13.1	45.9	$C_{11}H_{24}$ isomer

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
174	0.7	2.4		3.7	13.0	C_3 alkyl benzene & C_4 alkylbenzene isomer (5%) & $C_{11}H_{22}$ isomer (30%)
175	not detected			0.9	3.1	$C_{11}H_{22}$ isomer
176	0.6	2.0		1.8	6.2	cyclic $C_{11}H_{22}$ isomer & olefinic $C_{11}H_{22}$ isomer
177	1.0	3.4		2.8	9.8	$C_{11}H_{20}$ isomer & $C_{11}H_{22}$ isomer (50%)
178	0.4	1.3		2.0	7.1	C_4 alkyl benzene
179	1.3	4.4		4.6	16.1	C_3 alkyl benzene & C_4 alkyl benzene (20%)
180	1.1	4.0		4.7	16.5	C_4 alkyl benzene & $C_{10}H_{18}$ isomer (40%)
181	0.4	1.4		1.9	6.6	cyclic $C_{11}H_{22}$ isomer
182	0.8	2.8		3.5	12.3	C_4 alkyl benzene isomer & $C_{11}H_{22}$ isomer (50%)
183	17.0	59.4		60.9	212.8	n- undecane
184	1.2	4.1		4.1	14.4	C_3 alkyl benzene

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
185	0.7	2.3		2.9	10.2	C_4 alkyl benzene
186	0.2	0.7		2.1	7.4	$C_{12}H_{26}$ isomer
187	0.5	1.6		2.0	7.0	$C_{11}H_{22}$ olefin isomer
188	0.8	2.6		3.5	12.4	$C_{12}H_{24}$ olefin isomer
189	1.8	6.4		7.1	24.8	$C_{12}H_{24}$ olefin (10%); C_5 alkyl benzene isomer C_2 indane isomer (50%)
190	1.3	4.4		4.9	17.3	$C_{12}H_{26}$ isomer (50%); C_5 alkyl benzene isomer
191	2.9	10.3		10.6	37.0	C_5 alkyl benzene (15%); alkyl indane $C_{10}H_{12}$
192	0.5	1.8		5.5	19.1	C_5 alkyl benzene (20%); alkyl cyclohexane $C_{11}H_{22}$
193	2.8	9.9		10.7	37.3	C_4 alkyl benzene (40%) $C_{11}H_{20}$ isomer
194	0.4	1.5		2.1	7.2	benzaldehyde
195	not detected			1.6	5.8	C_5 alkyl benzene isomer

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m³	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m³	Identification
196	not detected		1.8	6.2	C_4 alkenyl benzene
197	1.7	5.8	6.2	21.6	C_5 alkyl benzene isomer
198	0.7	2.3	3.3	11.7	C_5 alkyl benzene
199	0.5	1.8	3.0	10.6	C_5 alkyl benzene (50%); $C_{11}H_{14}$ isomer
200	1.2	4.2	3.4	11.9	$C_{12}H_{24}$ cyclic isomer
201	not detected		1.9	6.6	C_5 alkyl benzene (10%); $C_{13}H_{28}$ isomer
202	not detected		1.9	6.6	$C_{12}H_{26}$ isomer
203	1.1	3.7	3.3	11.6	C_5 alkenyl benzene $C_{11}H_{14}$
204	1.3	4.4	5.4	19.0	C_5 alkenyl benzene isomer
205	0.9	3.0	4.0	14.0	C_5 alkenyl benzene (70%); alkyl indane
206	1.0	3.4	4.3	15.1	C_5 alkenyl benzene $C_{11}H_{14}$

Headspace Volatiles - continued

Peak #	90°F ppm	90°F mg/m^3	Concentration (hexane)	120°F ppm	120°F mg/m^3	Identification
207	0.4	1.5		3.3	11.6	C_6 alkyl benzene (25%), C_5 alkyl benzene $C_{12}H_{24}$ cyclic isomer
208	0.7	2.3		3.5	12.3	$C_{13}H_{28}$ isomer
209	0.1	0.4		1.2	4.1	C_5 alkenyl benzene
210	8.5	29.6		46.5	162.5	n-dodecane
211	0.4	1,3		2.4	8.4	C_5 alkenyl benzene (90%); $C_{12}H_{22}$ isomer
212	0.4	1.5		2.4	8.4	unknown
213	0.4	1.3		1.5	5.3	C_6 alkyl benzene
214	2.6	9.1		12.4	43.4	$C_{13}H_{28}$ isomer
215	0.4	1.2		1.4	5.0	$C_{12}H_{24}$ olefin
216	1.3	4.7		8.2	28.6	$C_{13}H_{26}$ olefin
217	0.5	1.9		2.0	7.0	unknown

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	mg/m^3	120°F Concentration (hexane) ppm	mg/m^3	Identification Rationale
218	not detected		1.6	5.4	$C_{11}H_{14}$ isomer
219	0.4	1.4	4.5	15.7	$C_{13}H_{24}$ isomer
220	1.2	4.0	1.3	4.7	$C_{12}H_{22}$ isomer
221	0.4	1.5	5.5	19.3	$C_{11}H_{14}$ isomer
222	1.1	3.9	1.6	5.5	$C_{13}H_{26}$ olefin
223	0.4	1.5	1.2	4.2	$C_{13}H_{24}$ olefin
224	0.2	0.8	1.9	6.6	$C_{13}H_{24}$ olefin
225	not detected		1.5	5.1	$C_{13}H_{28}$ isomer
226	0.8	2.9	3.6	12.7	C_7 alkyl benzene
227	0.5	1.9	2.6	9.0	unknown alkane
228	2.6	9.1	11.1	38.7	$C_{14}H_{30}$ isomer

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification Rationale
229	0.2	0.7	1.2	4.0	$C_{12}H_{16}$ C_6 alkenyl benzene
230	0.8	2.9	3.9	13.6	$C_{14}H_{28}$ olefin
231	0.8	2.9	3.0	10.6	unknown
232	0.5	1.9	2.3	8.0	$C_{13}H_{26}$ olefin
233	not detected		0.9	3.0	$C_{14}H_{28}$ isomer
234	4.6	16.2	23.4	81.7	n-tridecane
235	0.7	2.4	3.1	10.7	C_6 alkenyl benzene; alkyl indane
236	0.4	1.5	2.5	8.8	C_6 alkenyl benzene; alkyl indane
237	not detected		0.6	2.0	unknown
238	0.4	1.5	1.5	5.3	$C_{14}H_{30}$ isomer
239	0.4	1.4	2.3	8.2	unknown

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification Rationale
240	not detected		0.6	1.9	$C_{13}H_{18}$ isomer; $C_{12}H_{16}$ isomer
241	not detected		0.7	2.5	unknown
242	0.8	2.8	5.7	19.9	$C_{13}H_{18}$ isomer
243	not detected		0.6	2.1	$C_{13}H_{26}$ olefin
244	0.2	0.8	1.4	4.9	unknown
245	not detected		0.9	3.2	$C_{14}H_{28}$ olefin; $C_{13}H_{18}$ isomer
246	not detected		0.4	1.5	$C_{13}H_{20}$ isomer C_7 alkyl benzene
247	0.3	0.9	1.1	4.0	$C_{14}H_{30}$ isomer
248	0.5	1.9	2.7	9.3	$C_{14}H_{30}$ isomer
249	0.2	0.8	1.2	4.2	$C_{14}H_{30}$ isomer
250	not detected		0.8	2.8	$C_{13}H_{18}$ isomer; $C_{13}H_{20}$ isomer (40%)

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification Rationale
251	not detected		0.7	2.5	unknown
252	1.6	5.4	6.8	23.7	$C_{15}H_{32}$ isomer
253	not detected		0.3	1.2	$C_{12}H_{16}$ isomer
254	not detected		1.1	3.9	$C_{14}H_{28}$ olefin
255	0.3	1.2	1.7	5.9	$C_{14}H_{28}$ olefin
256	not detected		0.1	0.3	C_2 naphthalene
257	2.0	6.9	12.5	43.7	n-tetradecane
258	0.3	1.2	2.0	6.9	$C_{15}H_{30}$ isomer; $C_{15}H_{32}$ isomer
259	not detected		0.5	1.9	C_3 naphthalene
260	not detected		0.9	3.3	C_3 napthalene
261	not detected		0.2	0.8	$C_{13}H_{18}$ isomer

Headspace Volatiles - continued

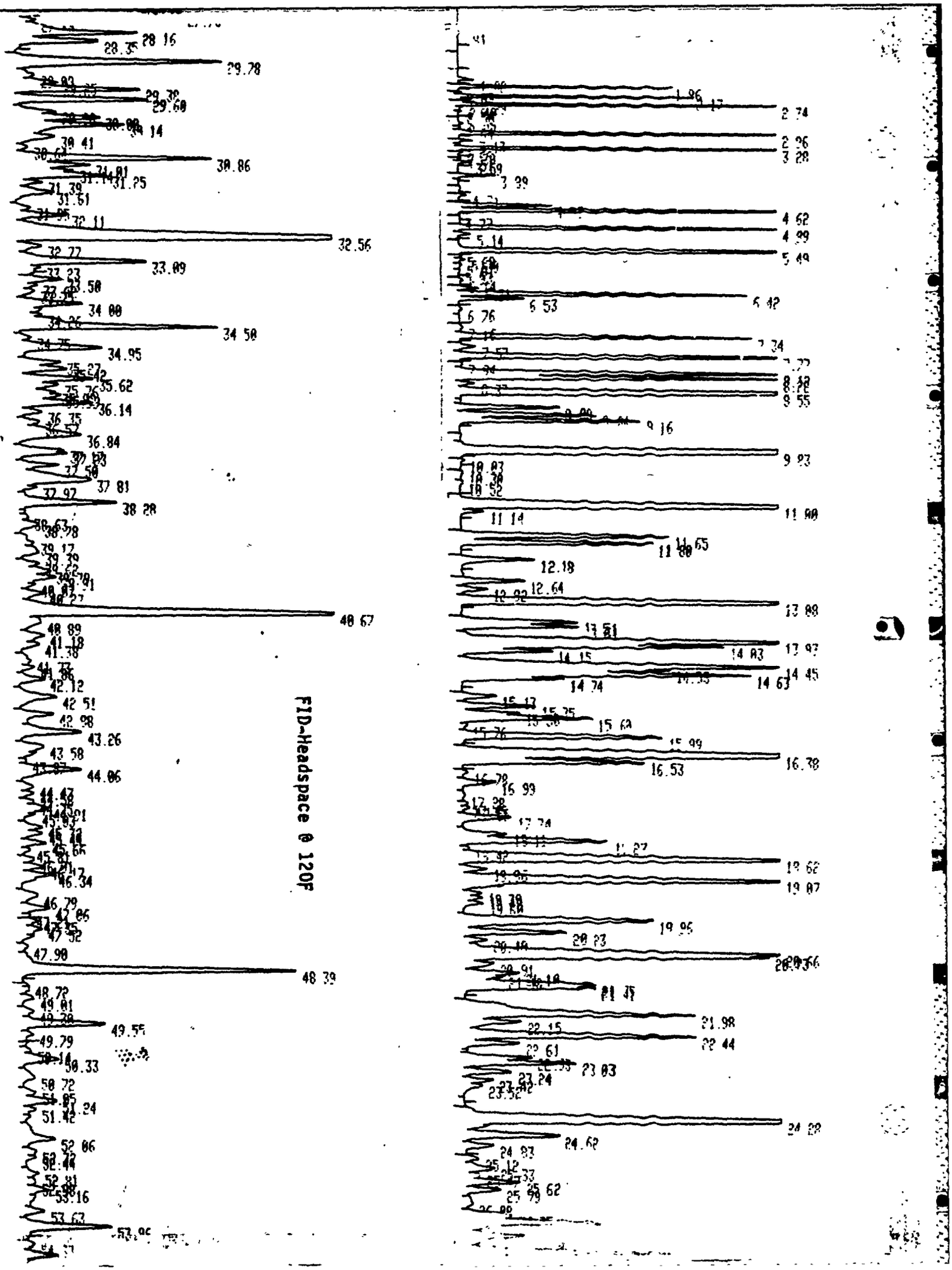
Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification Rationale
262	not detected		0.4	1.4	$C_{13}H_{18}$ isomer
263	not detected		1.0	3.4	$C_{13}H_{18}$ isomer
264	not detected		0.3	1.1	$C_{13}H_{18}$ isomer
265	not detected		0.3	1,1	$C_{15}H_{28}$ olefin
266	not detected		1.6	5.6	$C_{14}H_{28}$ olefin
267	not detected		0.3	1.1	unknown
268	not detected		0.4	1.3	$C_{14}H_{22}$ isomer; $C_{15}H_{30}$ olefin
269	1.1	3.8	5.1	18.0	$C_{16}H_{34}$ isomer
270	not detected		0.6	2.2	$C_{15}H_{32}$ isomer
271	not detected		0.7	2.4	$C_{15}H_{28}$ isomer - cyclic
272	not detected		0.7	2.3	$C_{15}H_{30}$ olefin

Headspace Volatiles - continued

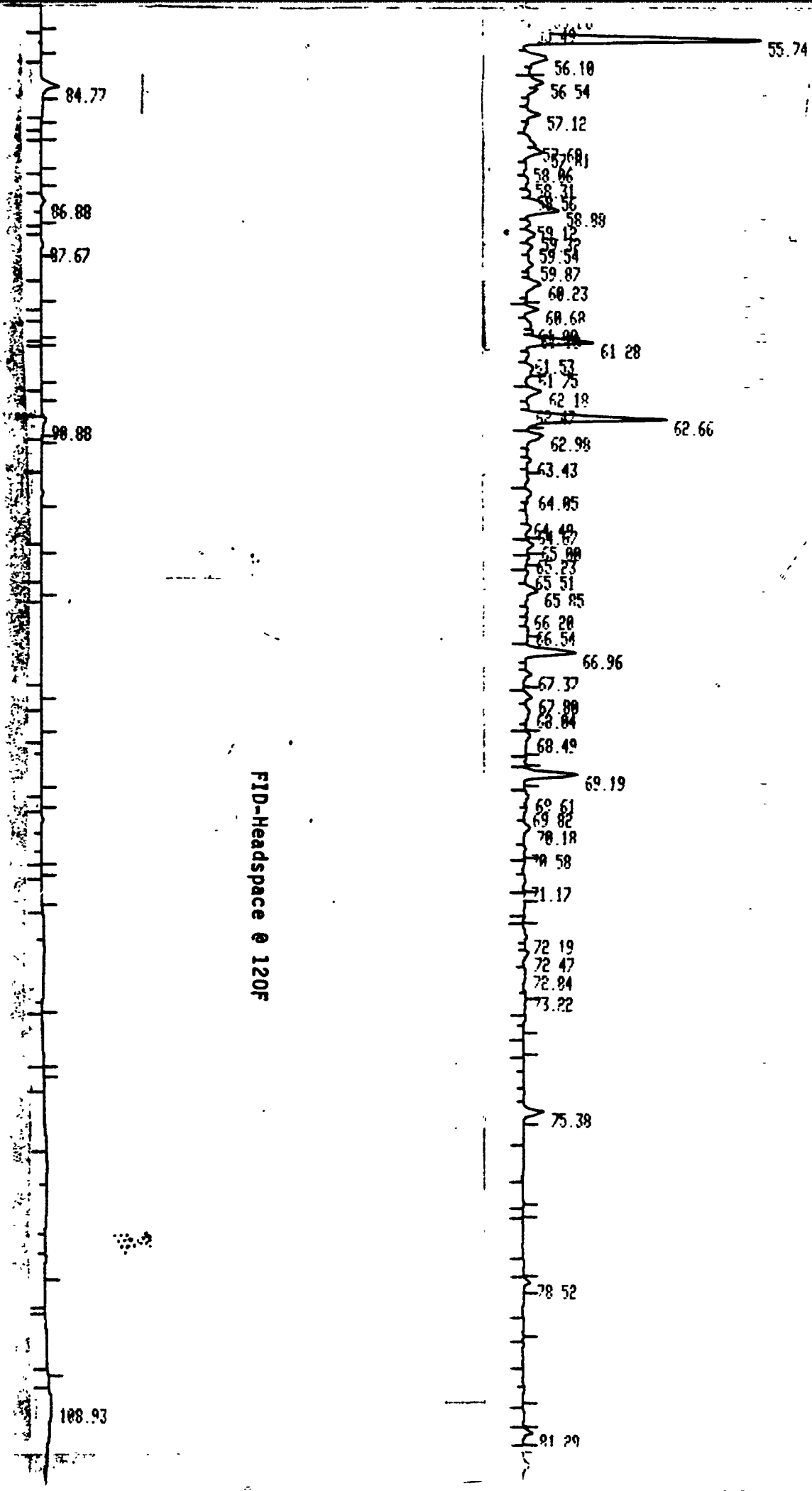
Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F Concentration (hexane) ppm	120°F Concentration (hexane) mg/m^3	Identification Rationale
273	not detected		0.7	2.3	$C_{16}H_{32}$ olefin
274	1.0	3.4	4.9	17.2	n-pentadecane
275	not detected		0.5	1.7	$C_{14}H_{20}$ isomer
276	not detected		0.3	1.2	unknown
277	not detected		0.8	2.7	$C_{13}H_{14}$ isomer
278	not detected		0.2	0.6	$C_{13}H_{14}$ isomer
279	not detected		0.3	0.9	$C_{16}H_{34}$ isomer
280	not detected		0.3	1.0	unknown
281	not detected		0.5	1.6	$C_{16}H_{32}$ isomer
282	not detected		0.3	1.1	unknown
283	not detected		0.3	0.9	unknown

Headspace Volatiles - continued

Peak #	90°F Concentration (hexane) ppm	90°F Concentration (hexane) mg/m^3	120°F ppm	120°F mg/m^3	Identification Rationale
284	not detected		1.8	6.3	n-hexadecane
285	not detected		0.6	2.3	$C_{17}H_{36}$ isomer
286	not detected		0.7	2.5	n-heptadecane
287	not detected		0.7	2.5	$C_{18}H_{38}$ isomer
288	not detected		0.8	2.7	n-octadecane
289	not detected		0.3	1.0	unknown
290	not detected		0.2	0.6	unknown



FID-Headspace @ 120F



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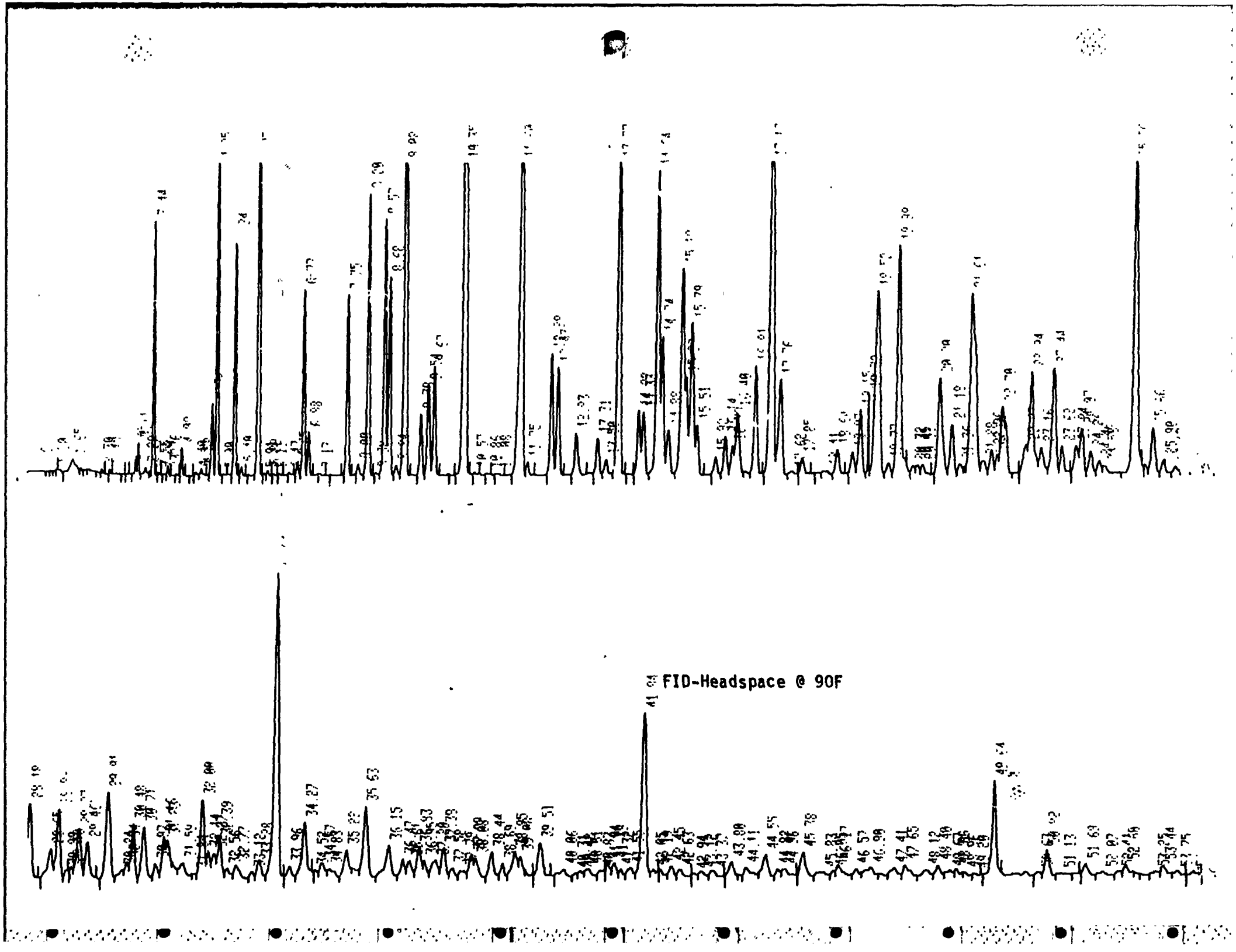
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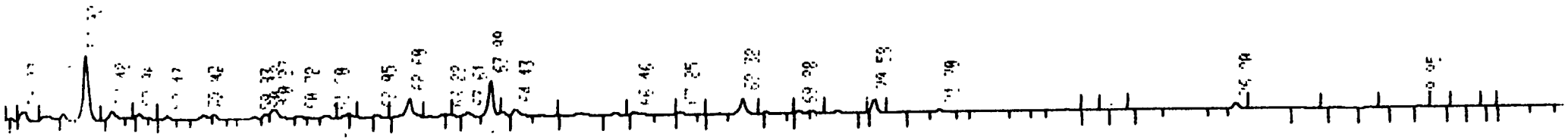

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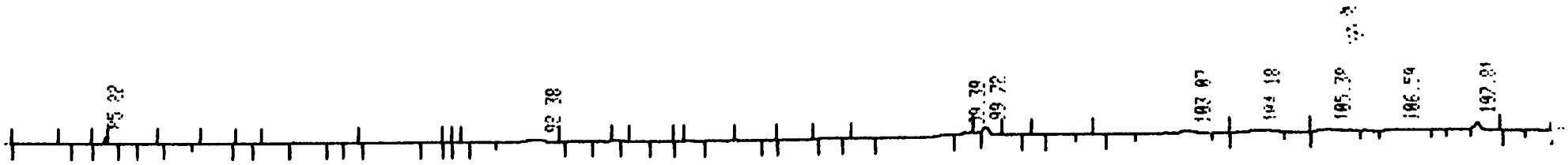

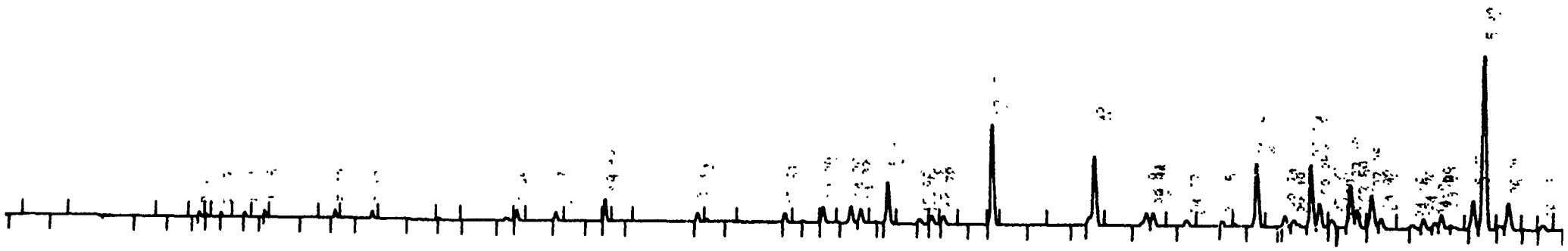

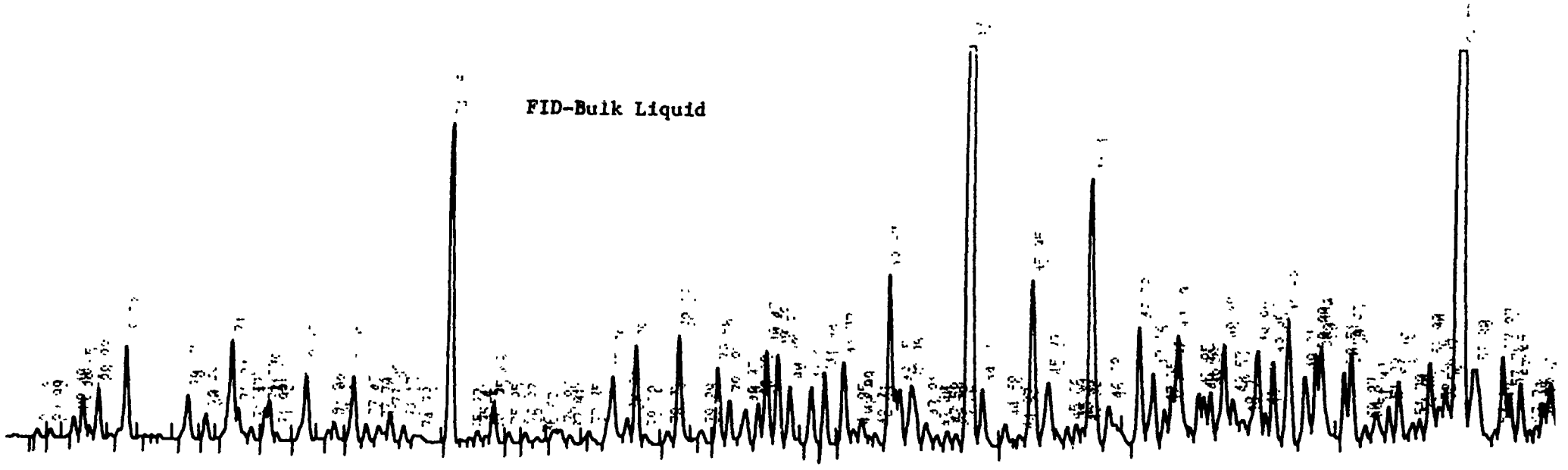
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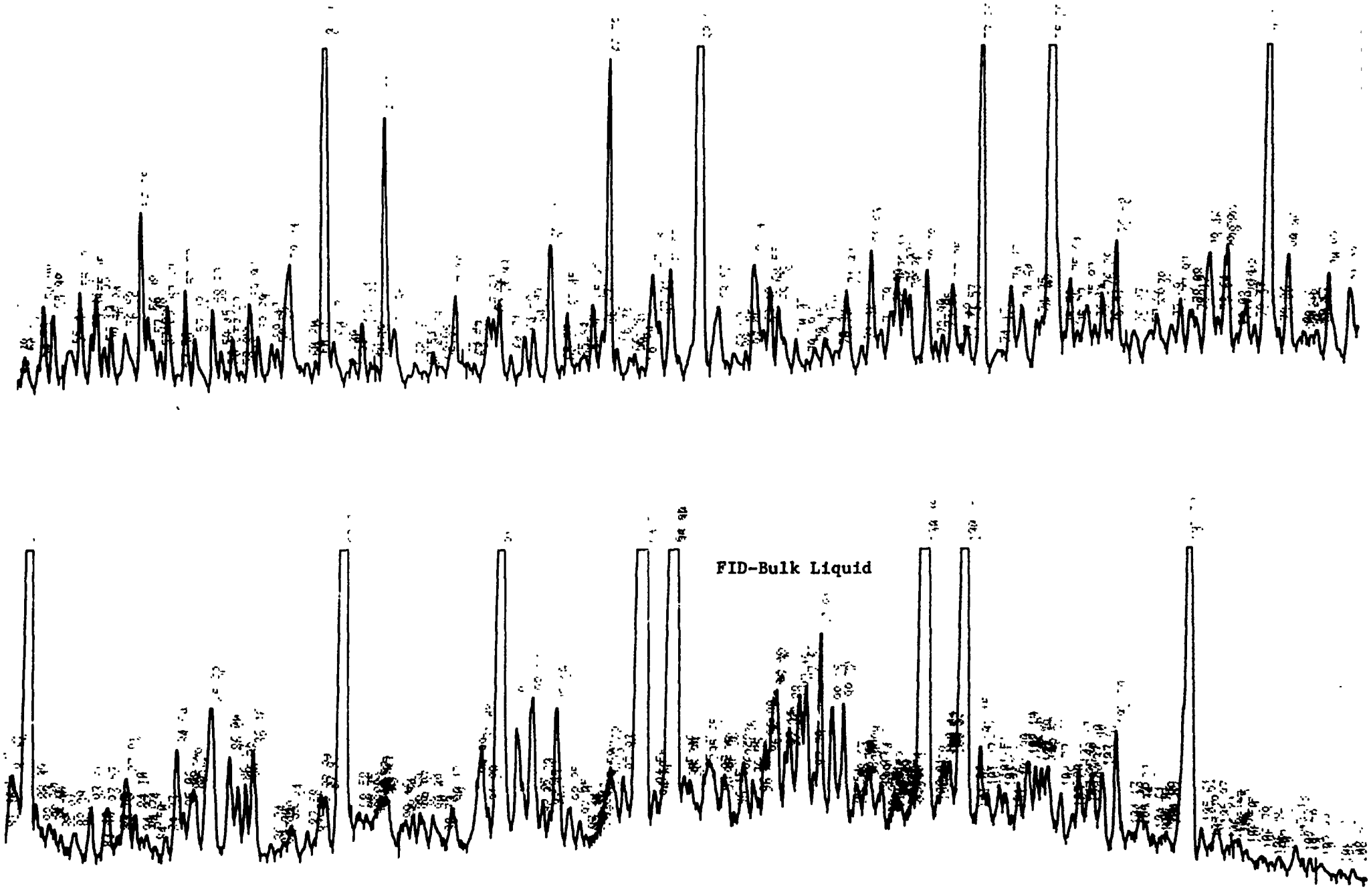
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