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Organic Constituents in Process Water from the In-Situ Retorting of Oil from Oil-Shale Kerogen

Occidental Oil Shale, Inc., Logan Wash No. 6
Retort Experiment

L. A. Raphaelian and W. Harrison

ARGONNE NATIONAL LABORATORY
Energy and Environmental Systems Division

prepared for
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ABSTRACT

Capillary-column gas-chromatography/mass-spectrometry (GC/MS) was performed on the acid, base, and neutral fractions of liquid- and particulate-phase methylene chloride extracts of a composite sample of raw process water collected from separator Tank 6 by the Laramie Energy Technology Center. Of the 160 extractable and chromatographable organic compounds tentatively identified, the following compound classes were found (listed in decreasing order of abundance): quinolines and lower fatty acids, aminoindoles, neutral oxygenated heterocyclics, pyridines, pyrroles, pyrazoles, phenols, and alkanes. Noticeably absent or in low concentration were alkyl benzenes and alkenes. Assuming 100% extraction efficiency, these organics constitute approximately 0.035% of the retort water; approximately 50% of this amount is represented by the quinolines, fatty acids, aminoindoles, and oxygenated heterocyclics. The following differences were noted in the composition of the particulate and liquid extracts of the neutral and base fractions, respectively: (1) alkanes are a major portion of the particulates, whereas oxygenated heterocyclics are most prominent in the liquid; and (2) aminoindoles are only a minor portion of the particulates, but are prominent in the liquid phase. The concentration of a compound occurring in both the liquid and particulate extracts is approximately 40-100 times higher in the liquid than in the particulate extract.

1 INTRODUCTION

One of the leading oil-shale extractive technologies under consideration for commercialization involves the underground retorting of oil shales found in the Green River formation of the western United States. Occidental Oil Shale, Inc., a subsidiary of Occidental Petroleum (Oxy), has demonstrated a modified in-situ (MIS) retorting process. The process involves digging a tunnel to the bottom of an oil-shale bed and then excavating shale to create a room of the same cross section as the future retort. Holes are

then drilled through the roof of the room to the desired height of the retort. Explosives are inserted and detonated, creating a column-shaped underground retort filled with shale rubble.

After sealing the access tunnel, air and burning fuel gas are injected into the top of the rubble pile through a specially constructed injection hole. Heat from the combustion of the top layers moves downward in the gas stream and oil vapors are carried down the retort to a sump at the bottom, from which they are removed to the surface by pumping. The combustion zone moves slowly down the retort, fueled by residual carbon in the retorted layers. Combustion ceases when the burn zone reaches the bottom of the retort and the flow of air is stopped. Formation and combustion-process water is released during a burn and this "process water," contaminated with organic combustion products, is the type characterized in the present study.

Previous work on chemical characterization of oil-shale waters has been directed primarily toward inorganic species and water quality parameters. Fox, Farrier, and Poulson (1978) have summarized the earlier studies and presented new information on Omega-9 water from the in-situ combustion experiment at the Laramie Energy Technology Center's (LETC's) site at Rock Springs, Wyoming. Pellizzari et al. (1979) did a GC/MS study of organics in the same Omega-9 in-situ retort water. They identified 97 compounds, compared to the 160 compounds identified in the present study.

2 FIELD EXPERIMENT

Oxy's work began in 1972 along Logan Wash, a private tract on the southern fringe of the Piceance basin, about 50 mi northeast of Grand Junction, Colorado. Oxy uses a vertical burn in its MIS process; that is, the combustion zone progresses vertically through the shale bed (Office of Technology Assessment, 1980, p. 132). The burning of Oxy's No. 6 retort began in 1978. About 40% of the oil in place was recovered.

3 SAMPLE HISTORY AND SAMPLE WORKUP

Water studied by Argonne represents oily water from retort No. 6 that was sampled at point 2 (Fig. 3.1)* on February 11, March 10, April 22, and May 17, 1979. Sampling point 2 is known as "Tank 6." The following historical statement for Sample point 2, Tank 6 water, was provided by the Division of Environmental Sciences, Laramie Energy Technology Center (LETC).

Sample point 2....is located outside the mine on the lower mine bench. Tank 6 was not in operation from January-April 1979. The January-April samples were taken from a pipe downstream of the small separator tanks (1 and 1B) underground and were pumped by a Moyno pump into the drum with an existing hose at the sample location. The May sample was pumped from the TK 6 water compartment. A two-inch hose was run from an opening in the top of the tank to the drums in the refrigerated trailer. When the oily water was pumped to TK 6, the small separator tanks were by-passed. Therefore the early samples, as well as the May sample, represent the first oil-water separation after the Retort 6 bulkhead. There is no difference between the use of TK 1 and 1B or TK 6 for oil-water separation. Sample point 2 was preceded by oil-water separation in the tank. In both cases the water was in the tanks 8-24 hours prior to pumping. This sample was loaded into a refrigerated trailer within 24 hours, usually during the same shift. In the case of the TK 6 (May sample), it was immediately loaded into the refrigerated trailer.

The sample provided to Argonne by LETC consisted of 5 gal of a 3330-gal composite sample from the following four sampling efforts:

<u>Sampling Date</u>	<u>Amount (gal)</u>
2/11/79	810
3/10/79	840
4/11/79	840
5/17/79	840

LETC designated Argonne's sample as 79-OXY-6-02W-00C.

Upon arrival at Argonne, the sample was filtered and the water and particulates were each separated by a liquid-liquid extraction, with methylene chloride as the solvent, into acid, base, and neutral fractions for GC/MS analysis.

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*Figures and tables appear consecutively at the end of the report.

4 ANALYTICAL PROCEDURES

4.1 TYPE OF EQUIPMENT AND OPERATING CONDITIONS EMPLOYED

This study used capillary-column GC/MS to identify the organic constituents extracted from the aqueous and particulate phases and capillary-column GC with flame ionization detection (FID) to quantify the amount of each organic constituent. Quantification was based on a set of standard compounds that were representative of those found in the sample extracts.

The GC used in this study was the Hewlett-Packard (HP) 5840 fitted with a variety of accessories, including cryogenic cooling with liquid carbon dioxide, extra peak storage for integrated areas of up to 500 peaks, and the second generation HP capillary-column injection port (18835B), a modification of the Grob-type, splitless injection system. (The port could also be used to perform split injections.) An automatic sampler, the HP 7671, was used and the amount injected was set at either 1 or 3 μ L. The capillary column used was the HP 50-meter fused-silica methyl silicone (SP-2100) column with Carbowax 20M deactivation and a quoted maximum operating temperature of 280°C. The oven was programmed during runs from 20 to 280°C at 2°C/min with a 2-min hold at 20°C and a 20-min hold at 280°C. Injection port temperature was set at 225°C and FID temperature was set at 250.

The GC/MS used in this study was the HP 5993A equipped with an HP 21MXE Computer with 32K, 16-bit-word, core memory, 7900A Dual Disc Drive with two 2.5M bytes discs (one fixed and one removable), and a Tektronix 4012 Graphic Display Terminal. Peripheral equipment included a Tektronix 4631 Hard Copy Unit and a Zeta 130-10 Incremental Plotter. The capillary-column injection port delivered with the system was replaced with parts from an HP 18835B Capillary Inlet System. A digital electronic timer was used for turning on or off the valves used to purge solvent in Grob-type splitless injection and to by-pass the solvent peak to avoid overload of the mass spectrometer's pumping system.

Operating conditions for the GC portion of the GC/MS were the same as those used in the GC-only runs (see above). That is, the oven was programmed from 20-280°C at 2°C/min with a 2-min hold at 20°C and a 18-min hold at 280°C. The injection port was set at 225°C and the column was an HP 50-m, fused-silica methyl silicone (SP-2100) with Carbowax 20M deactivation.

Collection and analysis of data was done with the SIMAQ and SPEED programs, giving foreground collection of data and background analysis of data. The mass range scanned was 50-400 AMUs and, depending upon the A/D measurements made per datum point (each 0.1 AMU), the cycle time was either approximately 2 sec (for 1 A/D) or 4 sec (for 2 A/Ds). Other parameters were threshold at 20 and electron multiplier at 2200 or 2400 V, depending upon the concentration of the sample.

Standards used in determining response factors and reproducibility of the HP 5840 GC were obtained from Aldrich Chemical Company, Milwaukee, Wisconsin; Eastman Kodak Company, Rochester, New York; or Fischer Scientific Company, Fair Lawn, New Jersey.

4.2 METHODS USED FOR IDENTIFICATION AND QUANTIFICATION

Each of the fractions was run, as is, on the GC using a split ratio of approximately 20 and an injection volume of 1 μ L. An inspection of each GC chromatogram was then made to guide the subsequent analysis as follows:

1. If it was satisfactory, the fraction was used without dilution or concentration for quantitation of peaks,
2. If it showed "overload," the fraction required dilution (an estimate of the dilution required was made and the fraction was rerun at that dilution),
3. If it was slightly "weak," indicating that an additional small amount of the fraction was required to produce a satisfactory chromatogram, the fraction was then rerun with 3- μ L injection and a split-ratio of approximately 20,
4. If it was very weak, indicating that considerably more of the fraction would be required to produce an acceptable run, the GC injector was modified to a Grob-type splitless injector and a 1- μ L sample was run,
5. If the chromatogram from the 1- μ L splitless injection was found to be slightly weak, the fraction was run at 3- μ L splitless; if found to be very weak, the fraction was concentrated and run at 3- μ L splitless.

Once the concentration and method of injection had been established, capillary-column GC/MS was run to identify the peaks in the GC chromatograms. That is, the capillary-column GC/MS was used for identification and the GC for quantification of the identified peaks (and for establishing the quantity, dilution or concentration of sample, and

injection method, split or splitless, required for both quantification by GC and identification of peaks by GC/MS).

Organic compounds were identified by inspection of the fragmentation patterns of the mass spectra and the associated retention time and/or by comparison with spectra or data in the ASTM *Index of Mass Spectral Data* (1969), or Stenhagen, Abrahamson, and McLafferty's *Registry of Mass Spectral Data* (1974), or Heller and Milne's *EPA/NIH Mass Spectral Data Base* (1978).

The organic compounds identified were quantified using the integrated areas (area counts) of the HP 5840 GC and the response factors of representative compounds developed in standard runs with split and splitless injection, and with 1- and 3- μ L injection (Tables 4.1-4.4).^{*} As can be seen from Tables 4.3 and 4.4, the split ratio lies between 18 and 22 for most compounds; the ratio of area counts for a 3- μ L to a 1- μ L injection appears to be approximately 3. Thus, for calculating the concentration of compounds in these samples, a split ratio of 20 was used, and it was assumed that a 3- μ L sample was three times as large as a 1- μ L sample. For determining concentration, average and estimated values for area counts per nanogram for a 1- μ L splitless injection were used, as shown in Table 4.5. From these data, corresponding data can be developed for a 3- μ L splitless injection (three times the number of area counts), a 1- μ L (20:1) injection (one-twentieth the number of area counts), and a 3- μ L (20:1) injection (three-twentieths of the number of area counts).

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^{*}Tables and figures appear consecutively at the end of the report.

5 RESULTS

GC chromatograms for the liquid and particulate phases of the neutral fraction are shown in Fig. 5.1, for the base fraction in Fig. 5.2, and for the acid fraction in Fig. 5.3. Tentative compound identifications and concentration in $\mu\text{g/L}$ of compounds in the neutral, base, and acid fractions (for both liquid and particulates) are given in Tables 5.1-5.6. These compound concentration data represent the methylene-chloride extractable and GC-chromatographable compounds. (Some polar compounds, such as the lower fatty acids, would be expected to be poorly extractable.)

Although the GC chromatogram traces (Figs. 5.1-5.3) have been normalized for comparison, it can be readily seen from the tables that the concentration of any compound occurring in both the liquid and particulate fractions is approximately 10-400 times higher in the liquid than in the particulate extract. This increased concentration is not unexpected because the surface area of the particulates is small and continual solvation or partitioning is taking place between the particulates and the liquid.

In the neutral fraction, with the exception of the n-alkanes and other alkanes, which are nicely detailed (Fig. 5.1) in the particulates extract, a number of unresolved peaks in the liquid phase generally could not be identified by GC/MS due to high background (or poor resolution due to the complexity of the mixture). Where identification was possible, there were some alkylated furans, thiazoles, and cyclohexanes. Also, it is apparent that the particulates extract contains more alkanes (relative to other neutral compounds) than does the liquid. Surprisingly, alkenes and alkyl benzenes were found in only small amounts.

In the base fraction, as shown in Fig. 5.2, the GC chromatograms of both the particulate and liquid extracts are remarkably similar except that the liquid extract contains some compounds that have been tentatively identified as aminoindoles. The predominant compounds are quinolines (and the aminoindoles) with smaller amounts of pyridines, pyrroles, and pyrazoles. Saturated compounds such as piperidines or pyrrolidines were noticeably lacking.

In the acid fraction (Fig. 5.3), considerable similarity exists between the particulates and liquid fractions. The particulate extract contains fatty

acids from acetic (C_2) through pelargonic (C_9), and the liquid, from C_3 (propionic) through C_{10} (capric). Small amounts of phenols are also present in the extracts.

Generally, the predominant extractable and chromatographable organic constituents in the Oxy 6 process wastewater are the non-neutral compounds, quinolines and lower fatty acids and, to a lesser extent, pyridines, pyrroles, pyrazoles, and phenols. A class of basic compounds that appears to be aminoindoles is also present in relatively high concentration. In the neutral fraction, some neutral oxygenated heterocyclics appear to be present in moderate amounts and some alkanes, in small amounts. Overall, the samples are abundant in heteroatoms, particularly nitrogen and oxygen, and tend toward aromaticity and unsaturation with the exception that alkyl benzenes and alkenes are either not present or present in small quantities.

6 DISCUSSION AND CONCLUSIONS

An examination of the differences in organic constituents in the liquid and particulate phases is in order. In the neutral fraction of the particulates extract, alkanes (particularly n-alkanes) along with phytane and pristane predominate; in the neutral fraction of the liquid extract, however, the n-alkanes are only a minor component. Although the particulates contain many of the compounds present in the liquid, there is a clear difference in compounds adsorbed onto or inherently part of the particulates. Moreover, because of the difficulty in resolving and identifying the major components of the liquid and the discrimination of the alkanes, it would seem that these unidentified neutral compounds are the more polar neutral substances, such as furans, cyclohexanones, dioxanes, pyrans, etc. To identify these complex mixtures, further fractionation (with, for example, HPLC) would be required to simplify them. Thus, both the particulates and the liquid phases of the neutral fraction contain the same compounds, but a difference exists in that the particulates phase contains mostly alkanes.

Both the liquid- and particulate-phase extracts of the base fractions contain essentially the same compounds. However, relative to the neutral fraction, the ratio of the concentration of any one compound in the liquid to that in the particulate phase is considerably greater and is generally of the order of 100 to 400. Differences in this ratio are apparent, however, within this group of compounds; for example, the pyrroles, whose pair of electrons make a sextet, thereby stabilizing the molecule and rendering it relatively nonpolar, have lower ratios than the pyridines. The predominant classes of compounds in this fraction seem to be the quinolines, their parents, the pyridines, and a third class, presently unidentified, that is possibly aminoindoles. It also appears that benzoquinolines may be present, although they were not found in this study.

Although the acid fractions of most fossil-fuel products typically contain a variety of alkylated phenols, those of both the liquid- and particulate-phase extracts were remarkably similar and contain relatively small amounts of phenols and large quantities of carboxylic acids (up to C_{10}). What was surprising was to find an abrupt drop-off after C_8 (Fig. 5.3; Tables 5.5 and 5.6); only the lower acids were found.

Overall, the Oxy 6 process water sample contains largely acid and base organic compounds and relatively small amounts of alkanes. The neutral fraction does contain some heteroatom compounds, such as furans, thiazoles, and related classes. Thiophenes, benzothiophenes, and other sulfur species normally present in petroleum oils appear to be lacking.

Future work on characterization of the organic constituents in this type of water should concentrate upon:

1. Fractionation of the neutral fraction by HPLC and further identification of the apparent oxygenated compounds by GC/MS,
2. Fractionation by HPLC and derivatization of the base fraction to aid in further identification of the apparent heterocyclic compounds present, and
3. Derivatization (to methyl esters) of the acid fraction for a more precise look at the fatty acids present, or direct injection of the retort water onto the GC column.

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Fig. 3.1 Schematic of Oxy Retort 6 Experiment Showing Location of Sampling Point 2 and Tank 6 (TK6)

Fig. 5.1 Capillary Column GC Chromatograms of Neutral-Fraction Particulates (upper trace) and Liquid (lower trace) Extracts

Fig. 5.2 Capillary Column GC Chromatograms of Base-Fraction Particulates (upper trace) and Liquid (lower trace) Extracts.

Fig. 5.3 Capillary Column GC Chromatograms of Acid-Fraction Particulates (upper trace) and Liquid (lower trace) Extracts

Table 4.1 Determination of FID/GC Sensitivity of
Some Representative Neutral Organic
Compounds (1 μ L injection, splitless)

Compound	Amount Injected (ng)	Area Counts		Average Counts Per Nanogram
		1	2	
n-decane	29.2	22,820	23,800	798
n-tetradecane	30.5	29,430	30,600	984
cis-1,3-dimethyl cyclohexane	31.4	24,070	24,410	772
ethyl cyclohexane	31.5	24,040	24,760	775
i-propyl cyclohexane	32.1	27,450	28,500	871
t-butyl cyclohexane	33.2	29,300	30,480	900
toluene	34.7	30,550	30,830	884
o-xylene	35.9	31,840	32,930	902
p-diethyl benzene	34.5	29,320	28,840	843
tetralin	38.9	35,270	36,500	922
decyclohexyl	34.6	40,370	42,000	1,190
debenzofuran	43.5	45,020	43,770	1,021
benzaldehyde	41.8	25,590	26,320	621
benzonitrile	40.4	30,350	31,030	760
benzyl alcohol	41.8	32,200	32,210	770
5-nonanone	33.0	23,600	24,450	728

Table 4.2 Determination of FID/GC Sensitivity
of Some Representative Basic Organic
Compounds (1 μ L injection, splitless)

Compound	Amount Injected (ng)	Area Counts		Average (Counts/ng)
		1	2	
pyridine	39.1	17,710	17,670	452
2-picoline	37.7	17,320	16,930	454
2,4-lutidine	37.1	18,690	18,010	495
2,4,6-collidine	36.7	19,480	19,160	526
aniline-d ₅	40.9	8,936	7,966	207 ^a
o-toluidine	40.2	33,190	32,060	812
2,6-xylylidine	39.4	26,150	25,320	653
mesidine	38.5	24,110	23,340	616
quinoline	43.8	29,490	28,730	664
indole	NM ^b	293,100	285,900	--
benzyl amine	39.2	19,630	19,160	495
N-methyl aniline	39.6	24,590	23,760	610

^aLow due to deuteration.

^bNot measured; used for reproducibility and retention time studies.

Table 4.3 Determination of Reproducibility and of FID/GC Sensitivity of Some Representative Neutral Organic Compounds (3 μ L injection, split; split ratio approximately 22)

Compound	Amount Injected (ng/ μ L)	Area Counts				Average Counts Per Nanogram	Split Ratio ^a
		1	2	3	4		
n-decane	29.2	3,007	3,449	2,910	3,227	35.9	22.2
n-tetradecane	30.5	3,782	3,801	3,315	3,842	40.3	24.4
cis-1,3-dimethyl cyclohexane	31.4	3,499	4,027	3,398	3,779	39.0	19.8
ethyl cyclohexane	31.5	3,379	3,941	3,291	3,664	37.8	20.5
i-propyl cyclohexane	32.1	3,718	4,308	3,642	4,056	40.8	21.3
t-butyl cyclohexane	33.2	3,951	4,565	3,851	4,297	41.8	21.5
toluene	34.7	4,333	4,695	3,868	4,286	41.3	21.4
o-xylene	35.9	4,276	4,909	4,158	4,567	41.6	21.7
p-diethyl benzene	34.5	4,717	4,995	4,067	4,486	44.1	19.1
tetralin	38.9	4,727	5,152	4,381	4,868	41.0	22.5
decyclohexyl	34.6	5,718	5,890	5,005	--	53.3	22.3
benzaldehyde	41.8	3,462	3,814	3,193	3,521	27.9	22.2
benzonitrile	40.4	4,072	4,439	3,789	4,135	33.9	22.4
5-nonanone	33.0	3,066	3,368	2,865	3,206	31.6	23.1

^aDetermined by dividing the average counts per nanogram for splitless injection by that for split injection.

Table 4.4 Determination of Reproducibility, FID/GC Sensitivity, and Split Ratio of Some Representative Basic Organic Compounds (1- and 3- μ L injection, split)

Compound	Amount Injected (ng/ μ L)	Area Counts for 1 μ L Injection			Average Counts Per Nanogram	Split Ratio ^a	Area Counts for 3 μ L Injection			Average Counts Per Nanogram	Split Ratio ^a	Ratio Counts 3 μ L to 1 μ L Injection
		1	2	3			1	2	3			
pyridine	39.1	939	804	967	23.1	19.6	2,893	2,809	2,903	24.5	18.5	2.83
2-picoline	37.7	877	789	955	23.2	19.6	2,725	2,736	2,825	24.4	18.6	2.85
2,4-lentidine	37.1	1,028	861	1,112	27.0	18.4	3,039	3,002	3,169	27.6	17.9	2.92
2,4,6-collidine	36.7	1,541	1,345	1,614	40.9	12.9	--	--	4,685	42.6	12.4	2.88
aniline-d ₅	40.9	410	323	536	10.3 ^b	20.0	--	--	1,355	11.0 ^b	18.8	2.82
o-toluidine	40.2	1,789	1,469	1,931	43.0	18.9	5,182	5,113	5,480	43.6	18.6	2.95
2,6-xylidine	39.4	1,473	1,245	1,631	36.8	17.7	4,201	4,050	4,419	35.7	18.3	3.10
mesidine	38.5	1,337	1,130	1,499	34.3	17.9	3,960	3,784	4,123	34.2	18.0	3.02
quinoline	43.8	1,607	1,331	1,751	35.7	18.6	4,916	4,659	5,138	37.3	17.8	2.87
quinaldine	42.3	1,589	1,327	1,716	36.5	NMS ^d	4,834	4,510	4,967	37.6	NMS ^d	--
2,6-dimethyl quinoline	NM ^c	--	--	--	--	--	1,447	1,320	1,484	--	NMS ^d	--
1,2,3,4-tetrahydroquinoline	42.4	1,721	1,465	1,817	39.3	NMS ^d	5,308	4,978	5,517	41.4	NMS ^d	--
benzyl amine	39.2	1,400	1,177	1,480	34.5	14.3	3,930	3,801	4,051	33.4	14.8	3.10
indole	NM ^c	16,900	14,210	18,230	--	17.6	48,730	44,880	49,770	--	18.2	3.10

^a Determined by dividing the average counts per nanogram for splitless injection by that for split injection.

^b Low due to deuteration.

^c Not measured.

^d Not measured splitless due to interferences.

Table 4.5 Average Area Counts Determined from Tables 4.1 and 4.2
(1 μ L injection, splitless)

Type of Compound	Area Counts Per Nanogram
Neutral Compounds	
alkanes	830
cyclohexanes	830
aromatic hydrocarbons	880
cyclic compounds	1040
polar compounds	720
Basic Compounds	
pyridines	480
anilines	690
miscellaneous	590
Miscellaneous Polar Compounds	500

Table 5.1 Compounds in the Neutral Fraction of the Liquid Extract (1 μ L splitless, 1:50 dilution)

Retention Time, Min	Tentative Identification	Area Counts ^a	Concentration μ g/L
15.32	C ₂ -furan	498	103
19.02	C ₂ -furan	3,032	627
20.26	methyl thiazole	4,583	1,705
20.85	methyl thiazole	908	338
21.44	unidentified	841	-
23.21	unidentified	743	-
24.79	n-decane	4,896	1,023
25.24	unidentified	910	-
26.14	C ₂ -thiazole	1,698	632
28.11	C ₂ -thiazole	4,840	1,800
28.46	C ₂ -thiazole	2,298	855
28.78	C ₂ -thiazole	2,200	818
29.91	C ₃ -thiazole	4,016	1,494
30.22	C ₃ -thiazole	5,741	2,136
31.03	C ₃ -thiazole	4,093	1,523

^aArea counts for 1:50 diluted sample.

Table 5.2 Compounds in the Neutral Fraction of the
Particulates Extract (1 μ L splitless)

Retention Time, Min	Tentative Identification	Area Counts	Concentration μ g/L
15.71	C ₂ -furan	168	0.7
19.59	C ₂ -furan	1,249	5.2
20.98	methyl thiazole	2,688	20.0
21.49	methyl thiazole	464	3.5
25.35	n-decane	1,518	6.3
26.01	C ₃ -furan	787	3.2
26.84	C ₂ -thiazole	677	5.0
28.87	C ₂ -thiazole	2,355	17.5
29.17	C ₂ -thiazole	746	5.6
29.34	C ₂ -thiazole	742	5.5
30.00	C ₃ -thiazole	1,128	8.4
30.90	C ₃ -thiazole	1,213	9.0
31.61	C ₃ -thiazole	3,598	26.8
32.41	C ₃ -thiazole	2,012	15.0
32.96	n-hendecane	4,222	17.4
40.39	n-dodecane	8,260	34.0
42.02	C ₂ -cyclohexane	4,557	20.4
43.07	C ₂ -cyclohexane	6,564	29.4
45.22	trimethyl cyclohexane	6,504	28.7
45.64	C ₂ -cyclohexane	8,600	38.5
46.68	C ₁₄ -alkane	10,790	47.2
47.45	n-tridecane	8,480	34.9
52.71	C ₁₅ -alkane	7,464	31.2
54.12	n-tetradecane	11,170	46.0
58.23	C ₁₆ -alkane	12,200	51.0
60.41	n-pentadecane	12,630	52.0
66.38	n-hexadecane	13,510	55.6
69.35	C ₁₈ -alkane	13,940	58.3
72.03	n-heptadecane	14,780	60.8
72.59	pristane	19,080	78.5
73.34	unidentified	3,182	-
73.66	unidentified	3,104	-
74.24	unidentified	2,849	-
77.37	n-octadecane	8,588	35.9
78.07	phytane	8,648	36.1
82.46	n-nonadecane	7,594	31.7
87.34	n-eicosane	4,235	17.7
91.97	n-heneicosane	2,642	11.0
96.43	n-docosane	1,802	7.5
100.68	n-tricosane	1,353	5.7
103.57	unidentified	1,967	-
104.79	n-tetracosane	890	3.7
108.73	n-pentacosane	1,091	4.6
112.50	n-hexacosane	646	2.7
116.15	n-heptacosane	685	2.9
119.68	n-octacosane	541	2.3
123.06	n-nonacosane	580	2.4

Table 5.2 (Contd.)

Retention Time, Min	Tentative Identification	Area Counts	Concentration ^a µg/L
126.36	n-triacontane	377	1.6
129.54	n-hentriacontane	307	1.3
132.64	n-dotriacontane	99	0.4
136.09	n-tritriacontane	114	0.5
140.12	n-tetratriacontane	50	0.2

Table 5.3 Compounds in Base Fractions of the Liquid Extract (1 μ L injection)

Retention Time, Min	Tentative Identification	Area Counts ^a	Concentration μ g/L
7.99	pyridine	1,986 ^b	153.9
11.49	2-methyl pyridine	1,995 ^b	154.6
11.92	methyl pyrazole	622 ^b	46.3
13.75	3-methyl pyridine	815 ^b	63.2
14.02	4-methyl pyridine	5,301 ^b	410.8
15.01	C ₂ -pyrrole	1,634 ^b	121.6
15.90	C ₂ -pyridine	1,192 ^b	92.4
16.74	C ₂ -pyrazole	6,388 ^b	475.3
18.86	2,4-dimethyl pyridine	11,160 ^b	864.9
19.33	C ₂ -pyrrole	2,597 ^b	193.2
19.76	C ₂ -pyridine	4,000 ^b	310.0
20.62	C ₂ -pyrazole	7,394 ^b	550.1
23.26	2,4,6-trimethyl pyridine	18,805 ^c	1457.4
23.80	C ₂ -pyridine	21,800 ^c	1689.5
24.86	C ₃ -pyridine	5,023 ^b	389.3
25.90	C ₃ -pyridine	6,335 ^c	491.0
28.40	C ₃ -pyridine	18,440 ^b	1429.1
29.08	C ₃ -pyridine	20,760 ^b	1608.9
29.94	aniline	18,620 ^c	1003.9
30.27	unidentified	8,705 ^c	-
30.44	C ₃ -pyridine	5,465 ^c	423.5
31.15	C ₄ -pyridine	9,110 ^c	706.0
32.72	methyl aniline	16,015 ^c	863.4
35.05	C ₄ -pyridine	25,485 ^c	1975.1
41.14	quinoline	33,190 ^c	1870.7
42.61	isoquinoline	18,090 ^c	1019.6
44.26	unidentified	14,310 ^c	-
44.57	unidentified	14,795 ^c	-
45.81	C ₁ -quinoline	31,815 ^c	1793.2
46.11	C ₅ -pyridine	11,640 ^c	902.1
46.34	C ₁ -quinoline	26,200 ^c	1476.7
47.64	C ₁ -quinoline	11,090 ^c	625.1
48.13	C ₁ -quinoline	11,580 ^c	652.7
48.32	unidentified	12,605 ^c	-
48.99	C ₁ -quinoline	10,010 ^c	564.2
49.15	C ₁ -quinoline	8,350 ^c	470.6
49.49	unidentified	10,975 ^c	-
50.29	C ₁ -quinoline	9,580 ^c	540.0
50.62	C ₁ -quinoline	11,040 ^c	622.3
52.81	C ₂ -quinoline	20,920 ^c	1179.1
53.93	C ₂ -quinoline	18,995 ^c	1070.6
55.13	C ₂ -quinoline	18,115 ^c	1021.0
55.64	C ₂ -quinoline	10,115 ^c	570.1
55.98	C ₂ -quinoline	8,200 ^c	462.2

Table 5.3 (Contd.)

Retention Time, Min	Tentative Identification	Area Counts ^a	Concentration $\mu\text{g/L}$
62.94	unidentified	10,188 ^d	-
63.65	unidentified	5,630 ^d	-
64.04	unidentified	8,869 ^d	-
64.69	unidentified	14,710 ^d	-
65.92	unidentified	8,964 ^d	-
66.79	unidentified	5,639 ^d	-
67.39	unidentified	10,803 ^d	-
68.78	unidentified	16,617 ^d	-
69.47	unidentified	14,847 ^d	-
70.09	unidentified	11,881 ^d	-
70.48	unidentified	7,640 ^d	-
70.82	unidentified	6,059 ^d	-
71.27	unidentified	4,678 ^d	-
71.85	unidentified	7,256 ^d	-
72.05	unidentified	6,302 ^d	-
72.36	unidentified	16,199 ^d	-
73.82	unidentified	4,311 ^d	-
74.27	unidentified	6,099 ^d	-
75.52	unidentified	8,119 ^d	-
75.83	unidentified	19,171 ^d	-
76.54	unidentified	2,668 ^d	-
78.43	unidentified	3,802 ^d	-
79.01	unidentified	6,397 ^d	-
79.55	unidentified	3,363 ^d	-

^aFor 1:10 diluted sample.

^bFrom 1:10 diluted sample, splitless injection.

^cFrom 1:50 diluted sample, splitless injection.

^dFrom 1:19 split, split injection.

Table 5.4 Compounds in the Base Fraction of the
Particulates Extract (1 μ L, splitless)

Retention Time, Min	Tentative Identification	Area Counts	Concentration μ g/L
8.89	pyridine	986	2.5
12.12	2-methyl pyridine	210	0.5
12.48	methyl pyrazole	752	1.9
14.22	3-methyl pyridine	63	0.2
14.51	4-methyl pyridine	875	2.3
15.45	C ₂ -pyrrole	407	1.0
16.31	C ₂ -pyridine	139	0.4
17.11	C ₂ -pyrazole	824	2.0
19.15	2,4-dimethyl pyridine	1,636	4.2
19.62	C ₂ -pyrrole	205	0.5
20.04	C ₂ -pyridine	450	1.2
20.86	C ₂ -pyrazole	618	1.5
23.36	2,4,6-trimethyl pyridine	7,704	19.9
23.97	C ₂ -pyridine	6,087	15.7
24.40	unidentified	583	-
25.03	C ₂ -pyridine	1,012	2.6
25.99	C ₃ -pyridine	2,538	6.6
27.98	unidentified	1,090	-
28.52	C ₃ -pyridine	4,500	11.6
29.15	C ₃ -pyridine	2,427	6.3
29.99	aniline	6,049	10.9
30.31	C ₃ -pyridine	4,437	11.5
30.76	unidentified	2,820	-
31.18	C ₄ -pyridine	3,213	8.3
32.11	unidentified	3,768	-
32.73	methyl aniline	8,556	15.4
34.69	unidentified	12,470	-
35.03	C ₄ -pyridine	10,450	27.0
35.53	unidentified	7,198	-
40.79	unidentified	8,606	-
41.10	quinoline	8,862	16.6
41.39	unidentified	4,047	-
42.24	unidentified	10,010	-
42.56	isoquinoline	7,310	13.7
44.52	unidentified	8,614	-
45.79	unidentified	8,710	-
45.99	unidentified	1,902	-
46.08	unidentified	5,044	-
46.31	C ₁ -quinoline	12,140	22.8
47.58	C ₁ -quinoline	4,903	9.2
48.27	C ₁ -quinoline	5,434	10.2
49.10	C ₁ -quinoline	6,379	12.0
49.46	C ₁ -quinoline	5,215	9.8
50.57	unidentified	8,500	-
51.28	unidentified	6,005	-

Table 5.4 (Contd.)

Retention Time, Min	Tentative Identification	Area Counts	Concentration µg/L
52.78	C ₂ -quinoline	8,778	16.5
53.62	unidentified	3,986	-
53.88	C ₂ -quinoline	8,932	16.8
54.41	unidentified	4,936	-
55.13	C ₂ -quinoline	17,010	32.0
55.62	C ₂ -quinoline	7,690	14.4
55.93	unidentified	6,065	-

Table 5.5 Compounds in the Acid Fraction of the Liquid Extract (1 μ L, splitless, 1:10 dilution)

Retention Time, Min	Tentative Identification	Area Counts ^a	Concentration μ g/L
10.56	propionic acid	44,150	3,280
14.54	isobutyric acid	65,380	4,860
16.55	n-butyric acid	60,610	4,510
16.65	unidentified	1,616	120
18.85	C ₅ -carboxylic acid	20,440	1,520
19.55	C ₅ -carboxylic acid	24,810	1,850
20.67	C ₅ -carboxylic acid	48,360	3,600
23.39	n-valeric acid	82,610	6,150
25.71	C ₆ -carboxylic acid	16,940	1,260
26.01	phenol	39,030	2,900
26.80	C ₆ -carboxylic acid	15,710	1,170
29.95	n-capric acid	98,880	7,360
30.32	cresol	10,490	780
32.26	cresol	7,410	550
32.34	cresol	15,170	1,130
33.16	unidentified	4,245	320
36.61	n-heptoic acid	105,400	7,840
38.61	C ₂ -phenol	15,510	1,150
42.78	n-caprylic acid	89,060	6,630
43.28	C ₂ -phenol	10,900	810
43.61	C ₂ -phenol	13,740	1,020
48.21	n-pelargonic acid	35,870	2,670
53.30	n-capric acid	4,916	370

^aArea counts based on 1:10 diluted sample.

Table 5.6 Compounds in the Acid Fraction of the
Particulates Extract (3 μ L, splitless)

Retention Time, Min	Tentative Identification	Area Counts	Concentration μ g/L
6.93	acetic acid	4,486	11.1
8.40	unidentified	687	-
9.09	unidentified	354	-
9.94	unidentified	526	-
11.15	propionic acid	9,910	24.6
15.25	isobutyric acid	46,360	115.0
15.82	hydroxy butyric acid lactone	717	1.8
16.92	n-butyric acid	20,690	51.3
18.54	unidentified	703	-
18.94	hydroxy valeric acid lactone	2,520	6.2
19.90	C ₅ -carboxylic acid	14,540	36.1
20.84	C ₅ -carboxylic acid	34,160	84.7
21.25	C ₅ -carboxylic acid	18,560	46.0
21.78	C ₅ -carboxylic acid	29,810	73.9
24.41	n-valeric acid	75,510	187.3
24.73	unidentified	2,982	7.4
25.16	unidentified	3,579	8.9
25.47	unidentified	3,962	9.8
26.33	C ₆ -carboxylic acid	9,939	24.6
27.52	C ₆ -carboxylic acid	34,590	85.8
28.09	C ₆ -carboxylic acid	15,350	38.1
28.93	phenol	40,760	101.1
30.54	n-caproic acid	50,550	125.4
30.60	unidentified	1,859	-
30.82	unidentified	3,053	-
31.03	unidentified	4,473	-
32.34	unidentified	10,700	-
32.53	cresol	14,090	34.9
33.37	C ₇ -carboxylic acid	17,370	43.1
33.87	unidentified	8,034	-
34.38	unidentified	6,934	-
34.78	cresol	26,300	65.2
36.65	n-heptoic acid	41,140	102.0
37.26	unidentified	5,189	-
38.01	C ₂ -phenol	6,896	17.1
38.42	C ₂ -phenol	9,512	7.9
38.84	C ₂ -phenol	7,348	18.2
39.62	C ₂ -phenol	10,040	24.9
40.17	C ₂ -phenol	6,000	14.9
40.52	C ₂ -phenol	29,200	72.4
41.43	C ₂ -phenol	3,197	7.9
42.70	n-caprylic acid	18,230	45.2
43.95	unidentified	6,686	-
45.42	unidentified	26,110	-
46.32	C ₃ -phenol	10,840	-
48.61	n-pelargonic acid	9,310	23.1

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