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QUARTERLY REPORT

CHARACTERIZATION OF SUBSTANCES IN PRODUCTS, EFFLUENTS
AND WASTES FROM SYNTHETIC FUEL PRODUCTION TESTS

M. R. Petersen, J. S. Fruchter
and J. C. Lau1

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Battelle
Pacific Northwest Laboratories
Richland, Washington 99352

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PREFACE

Battelle-Northwest is working under contract with the Energy Research and Development Administration to characterize materials from promising synthetic fossil fuels. The objectives of this work are to determine what materials may possibly enter the environment as a result of the full-scale commercial operation of such processes which may be detrimental from an environmental or health standpoint. The early characterization of such materials would permit ample time for appropriate control techniques to be implemented prior to full-scale commercialization.

These quarterly reports are intended to provide up-to-date information on the status of these characterization efforts, both to those concerned with techniques and abatement process development and to the biological and environmental scientists working in this area.

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Numerous coal conversion processes are being tested on a laboratory scale and in pilot plants. PNL is conducting a program in which we are characterizing products, effluents, and wastes from promising processes. The characterization program involves a comprehensive analysis of inorganic constituents, including chemical species determination, together with the analysis of organic materials. The organic materials are separated into several fractions, which are then characterized chemically. Sample fractions and individual compounds identified in the analysis may then be made available to biologists for toxicity testing, especially for mutagenesis and carcinogenesis tests. The initial work is being conducted at the solvent refined coal (SRC) plant operated by Pittsburg and Midway Coal Mining Company at Ft. Lewis, Washington. It is our intent to carry out the characterization program for several pilot plants as necessary arrangements are concluded.

Organic and Inorganic Constituents in the Solvent Refined Coal Process

In work conducted to date, we have identified about 125 different organic and inorganic compounds in products, byproducts, effluents, and wastes from the solvent refined coal process. Two sets of samples have been analyzed in the work to date. Separation techniques have been used to partition the organic compounds into classes to aid in this characterization work.

Samples obtained from the SRC plant were collected on site during May, 1976 and include the feed coal, SRC product, mineral residue, retention pond sediment, byproduct sulfur, process water at various treatment stages, process solvent, wash solvent, byproduct light oil or "naphtha", off-gas samples, and aerosol material collected over the cooling product. Since the SRC plant is a pilot operation, operating conditions and materials are changed frequently for experimental purposes. Therefore, analytical results based on individual samples may vary substantially between plant runs. We thank the staff of the SRC plant for making the samples available to us for these studies.

The water samples were preserved with hydrochloric acid or methylene chloride for inorganic and organic analysis, respectively. The sediment samples were frozen immediately with dry ice; the organic liquids were kept under refrigeration.

The separation scheme which permitted quantitative analysis of the samples for some components consisted of dissolving the sample in isooctane and partitioning it into basic, acidic, neutral, and polynuclear aromatic fractions. This is accomplished by extracting with 1N HCl, 1N NaOH and finally treating the neutral fraction with dimethylsulfoxide to remove the polynuclear aromatic hydrocarbons (PNA).

The resulting four fractions: basic, acidic, neutral, and PNA are analyzed by gas chromatography and by gas chromatography-mass spectrometry (GC/MS).

The samples of light oil, wash solvent, and process solvent are a set of progressively higher boiling cuts, which are distilled just prior to recovery of the dissolved solvent refined coal. The distribution of organic compounds in these samples reflect this boiling range. Substituted benzenes and indans predominate in the light oil; naphthalene and methylnaphthalenes are the most abundant (at least 3% each) in the wash solvent; phenanthrene/anthracene are the major compounds in the process solvent. A total of about 65 aromatic, 25 aliphatic, 15 phenolic, and 15 heterocyclic compounds have been identified in these samples. Polycyclic aromatic compounds up through alkyl substituted benzopyrenes have been identified.

The untreated process water contains several percent of organic matter, and the preliminary analyses indicate that phenols are the predominate species present. Other hydrocarbons, such as PNA's, are present at the ppm level. The mineral residue and product contain many PNA's in 100 ppm ranges. The aerosol material arising during cooling of the product is composed of many high molecular-weight PNA's with a distribution of compounds similar to that of SRC product. The concentrations are

high in the particulate samples that were collected directly over the molten product. However, the emission control equipment was shut down during the period of collection of samples. Quantitative data are given in Table I for some of the samples which have been analyzed thus far. These data have not been corrected for losses of the low molecular weight compounds, of less than 150 amu. The organic liquids contain considerable tar material, e.g., the process solvent is at least 60% asphaltene. This material precipitates during the partitioning procedure and a satisfactory analysis of the tar has not been obtained. Table I-A presents a list of compounds which have been detected and are now being quantitatively determined.

Figure 1 is a gas chromatogram of the PNA extract of a process "naphtha". The page following the figure gives the identity of the numbered peaks.

Analytical procedures are being improved to determine the organics remaining in the treated water. The compounds are present in ppm concentrations and tend to be water soluble. Some additional work is necessary to permit quantitative analysis of the trace organic components in the treated water.

An important part of the research program at the SRC plant is the defining of the distribution and chemical species of the trace elements. Samples of products, effluents, and wastes have been collected and analyses of approximately 30 trace elements are being carried out. Measurements of these elemental concentrations in the coal, mineral residue, and the solvent refined coal product are presented in Table II. The variability in the composition of coal is illustrated in this table. On comparing the composition of feed coal and the NBS standard coal mixture (columns 2 and 6), one finds up to six-fold differences in concentration. This variability of coal leads us to expect similar variability in the absolute trace element content of products and effluents from a given coal liquefaction process. However, in the SRC process, the most significant observation is that most of the bromine is carried from the coal into the product. Also, a substantial fraction of the zinc, tantalum, and chromium are carried into the product. In general, it appears that the refractory and alkaline metals remain almost entirely with the mineral residue.

TABLE I

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ANALYSES OF SAMPLES COLLECTED AT SOLVENT-REFINED COAL PLANT
Concentration (ppm)

PNA Fraction	Light Oil	Wash Solvent	Process Solvent	Raw Process Water	Mineral Residue	Solvent-Refined Coal	Particulate Filter (concentration $\mu\text{g}/\text{m}^3$)
xylene		1300					
o-ethylbenzene	9800	1700					
m/p-ethylbenzene		700					
C ₃ -benzene	3900	1500					
C ₄ -benzene		500					
indane	4300	13000			85		
methylindane	510	2500		15	25		
methylindane	180	1400					
methylindane	240	2300			55		
dimethylindane	< 5	40			25		
tetralin	330	4100		< 0.1	110		
dimethyltetralin	< 5	1500		0.5	35		
6-methyltetralin	110	3200			50		
naphthalene	1630	32000	100	5	1500	1	3
2-methylnaphthalene	690	32000	3800	2	740	8	16
1-methylnaphthalene	110	12000	930		180	5	4
dimethylnaphthalene	80	13000	11200	0.3	260	6	170
dimethylnaphthalene	70	700	1700		60	3	20
dimethylnaphthalene		4000	4200		150		
dimethylnaphthalene	10	160	650	2	< 2		10
2-isopropylnaphthalene		40	50	0.7	< 1		< 0.5
1-isopropylnaphthalene		210	1400	2	15		20
C ₄ -naphthalene		5	50		< 1		4
cyclohexylbenzene		410			5		
biphenyl	80	10000	5900	0.2	270	2	75
acenaphthylene	2	500	3400	< 0.1	45	8	60
dimethylbiphenyl	15	35	2100	0.5	30	9	130
dimethylbiphenyl	21	30	560	0.2	20	7	40
dibenzofuran	8	400	5800	0.6	60	9	160
xanthene	10	30	840	0.1	20	5	40
dibenzothiophene	3	50	4200	1.5	70	30	180
methyl dibenzothiophene		15	320	< 0.1	8	4	60
dimethyl dibenzothiophene	5	15	1200	< 0.05	20	13	130
thioxanthene		2	3300	0.1	5	3	120
fluorene	15	250	6600	0.3	80	27	200
9-methylfluorene	15	110	3100	0.3	40	11	150
1-methylfluorene	10	10	3000	0.2	50	18	100
anthracene/phenanthrene	25	130	23000	1.1	500	300	1500
methylphenanthrene	6	15	6200	0.3	100	50	400
1-methylphenanthrene	6		3900	0.2	50	30	300
C ₂ -anthracene	6	25	500	< 0.05	10	1	30
fluoranthene	15	35	10500	0.4	200	180	700
dihdropyrene	6	25	1200	< 0.05	10	1	30
pyrene	20	40	11200	0.6	200	280	900
NEUTRAL FRACTION (n-alkanes)							
n-octane	16000	900		2.3			
n-nonane	8700	2700					
n-decane	9800	5000					
n-undecane	3900	8300	50	0.3	90	4	
n-dodecane	1400	21000	80	0.3	550	10	
n-tridecane	470	14000	30	0.4	9100	8	4
n-tetradecane	170	11000	340	0.3	210	7	12
n-pentadecane	60	4000	1000	0.2	80	12	18
n-hexadecane	10	400	2000	0.2	50	8	50
n-heptadecane	10	120	3100	0.02	20	3	35
n-octadecane		40	920		10	3	18
n-nonadecane		500	800		16	22	30
n-eicosane			930		14		20
n-heneicosane			600		14		35
n-docosane			670		16		55
n-tricosane			980		14		35
n-tetracosane			900		14		45
n-pentacosane			740		10		43
n-hexacosane			450		8	5	40
n-heptacosane			300		6	2	25
n-octacosane			150		5	2	28
n-nonacosane			90		4	1	18
n-triacontane			60		2	1	22
n-hentriacontane			40		1	1	15
n-dotriacontane			10		< 1		11
n-tritriacontane			5				7

TABLE I-A

The following compounds have been identified but have not yet been quantitatively determined:

<u>PNA's</u>	<u>Phenols</u>	<u>Bases</u>
methylpyrenes	phenol	tetrahydroquinolines
benzofluorenes	o-cresol	methyltetrahydroquinoline
C ₂ -pyrene C ₂ -fluoranthane	m/p-cresol	quinoline
C ₂ -fluoranthene	2,6-xyleneol	isoquinoline
tetrahydrochrysene	2,5-xyleneol	methylquinolines
chrysene	2,4-xyleneol	dimethylquinolines
methylbenzofluorene	3,5-xyleneol	ethylquinolines
C ₃ -pyrene	o-ethylphenol	indole
C ₃ -fluoranthene	m/p-ethylphenol	methylindoles
methylchrysenes	trimethylphenol	dimethylindoles
methylbenzanthracenes	methylethylphenol	acridine
cholanthrene		methylacridines
tetrahydrobenzofluoranthene		carbazole
tetrahydrobenzopyrenes		methylcarbazoles
benzopyrenes		benzoquinolines
methylbenzopyrenes		methylbenzoquinolines
methylbenzofluoranthenes		benzoindoles
		methylbenzoindole

FIGURE 1. Gas Chromatogram of the Polynuclear Aromatic Hydrocarbon Fraction of Process Naphtha

POLYNUCLEAR AROMATIC HYDROCARBONS
IDENTIFIED IN CHROMATOGRAM

1. xylene	53. tetrahydrobenzofluoranthene/cholanthrene
2. ethylbenzenes	54. tetrahydrobenzopyrene
3. C ₃ -benzene	55. benzofluoranthene
4. C ₃ -benzene	56. benzopyrenes
5. n-decane	57. methylbenzopyrene or methylbenzofluoranthene
6. indane	58. methylbenzopyrene or methylbenzofluoranthene
7. C ₄ -benzene	59. unknown
8. methylindane	
9. n-undecane	
10. methylindane	
11. methylindane	
12. tetralin	
13. naphthalene	
14. n-dodecane	
15. cyclohexylbenzene	
16. dimethylindane	
17. 6-methyltetralin	
18. 2-methylnaphthalene	
19. 1-methylnaphthalene	
20. biphenyl	
21. dimethylnaphthalene/diphenylether	
22. dimethylnaphthalene	
23. dimethylnaphthalene	
24. dimethylnaphthalene	
25. 2-isopropylnaphthalene	
26. acenaphthene	
27. dibenzofuran	
28. fluorene	
29. 9-methylfluorene	
30. xanthene	
31. dimethylbiphenyl	
32. dimethylbiphenyl	
33. 1-methylfluorene	
34. dibenzothiophene	
35. anthracene/phenanthrene	
36. unknown	
37. thioxanthene	
38. methyl dibenzothiophene	
39. methylphenanthrene	
40. methylphenanthrene	
41. dimethyldibenzothiophene	
42. C ₂ -phenanthrene	
43. fluoranthene	
44. pyrene	
45. methylpyrene	
46. methylpyrene or 2,3-benzofluorene	
47. methylpyrene or benzofluorene	
48. C ₂ -pyrene or C ₂ -fluoranthene	
49. tetrahydrochrysene	
50. chrysene	
51. methylbenzofluorene, C ₃ -pyrene, or C ₃ -fluoranthene	
52. methylchrysene or methylbenzanthracene	

TABLE II. ELEMENTAL CONTENTS OF FEED COAL,
SOLVENT REFINED COAL AND MINERAL RESIDUE
FROM FT. LEWIS, WASHINGTON

(ppm except as noted)

Element*	Feed Coal	Solvent Refined Coal (SRC)	Mineral Residue	Coal/SRC	NBS Coal [†]
Na	180	8.8	3150	20	420
(%) K	0.19	0.003	1.10	70	0.28
(%) Fe	2.29	0.027	13.3	85	0.81
Cr	18.4	7.5	150	2.5	19
Co	5.2	0.26	32	20	5.2
Ni	21	<6	120	4	20
Sc	2.6	0.45	14.7	5.8	3.4
Ba	65	--	260	--	390
Sr	76	<5	360	20	170
La	9.5	0.13	56	73	10.5
Ce	17	0.5	102	40	--
Nd	<10	--	<50	--	--
Sm	1.85	0.18	9.92	10	1.7
Eu	0.36	0.025	1.90	14	0.28
Tb	0.30	0.026	1.3	11	0.23
Yb	0.65	0.080	3.2	8.1	--
Lu	0.090	0.02	0.48	3.3	--
Hf	0.44	0.054	2.3	8.1	0.97
Ta	0.14	0.043	0.62	3.3	0.46
Th	1.9	0.19	10	10	3.4
Rb	13	--	77	--	19
Cs	0.95	0.012	5.8	80	1.4
As	19	2.1	77	9.0	5.7
Sb	1.4	0.066	7.4	21	3.7
Zn	26	8.1	124	3.2	37
Br	3.6	4.7	4.9	0.80	17
Se	3.6	0.17	20.4	21	3.3
Hg	0.16	0.02	< 0.005	8.0	0.10

* Determined by instrumental neutron activation analysis. Estimated errors based on counting statistics are ~1-3% for Na, La, Co, Fe, Sc, Br, Zn, Sb, Sm, Yb and As; ~5% for Cr, Ce, Th, Lu, Ta, K, Ba, Eu, Hf and Rb; and ~10% for Tb, Hg, Cs, Ni, Se and Sr.

† Abel and Rancitelli (1975).

A clue as to the mineralogy involved and its contribution to elemental distributions can be obtained by comparing the rare earth spectra in the coal feed, the product and the mineral residue as shown in Figure 2. Here the rare earth concentrations are normalized to that which is believed to be the composition of the primordial earth. The coal feed and mineral residue have similar rare earth distributions, and these are typical of those found in major terrestrial minerals. The rare earth distribution in the solvent refined coal product is substantially different and is actually similar to the accessory mineral hornblende, which crystallizes late in magmatic differentiation processes. Thus, we believe that the mineral phases in coal may provide a clue to the potential for release of toxic trace elements to the product or the gaseous effluent. Analyses are also being performed on process solvent naphtha and liquid effluents from the SRC plant. Chemical speciation measurements will be made of such trace elements as arsenic and mercury. We thus expect to have information on the movement of the various trace elements through the process and the resulting chemical forms.

Oil Shale and Tar Sand Effluent Characterization

In-situ and above ground technologies are being investigated to determine their feasibility for extracting liquid petroleum products from oil shale and tar sands. The objective of the work reported here is to characterize products, effluents, and wastes that may be of concern from an environmental or health standpoint. To meet these objectives, the characterization has been conducted on samples obtained from the in-situ and above-ground retorting processing investigated by the Laramie Energy Research Center (LERC). We are grateful to LERC personnel for their cooperation in providing samples for analysis.

During the first months of this project, our efforts have been directed mainly towards characterization of samples obtained from the simulated in-situ LERC retorts at Laramie, Wyoming. We have determined inorganic elements and chemical forms in samples of raw shale oil, retort water, gaseous emissions, and raw and spent shale. In addition, we have

FIGURE 2. Comparison of the Distribution of Rare Earth Elements in Mineral Residue and Feed Coal

determined organic compounds in crude shale oil fractions and in the gaseous emissions. Two samples of oil shale, a sample of crude shale oil, a sample of process water, and several samples of the gaseous effluents were examined.

These initial inorganic element and speciation measurements indicated that concentrations of the heavy elements Hg, Ag, Zn and Se in one set of samples of crude shale oil, process water, and gas streams were higher than reported concentrations in crustal rock, river water, and urban air. In addition, the crude oil contains significant amounts of Co, and the process water contains significant Co, Br, and Sb. The data for the crude oil and process water are presented in Table III. When the abundances of trace metals in raw oil shale are normalized to average crustal abundances, one finds that raw oil shale is significantly enriched by the following factors: As(45), Sb(20), B(8), U(3.5), Cs(2.0), and Hg(2.0).

Raw shale oil samples have been separated into four organic fractions: neutral, polynuclear aromatics, acidic, and basic. The neutral fraction is composed largely of alkanes and alkenes. Although the polynuclear aromatic fraction has (see Figure 3) numerous aromatic hydrocarbons, it contains a surprising number of indoles. Identification of some of the compounds shown in this figure are based solely on mass spectrometry data and are subject to verification using gas chromatography retention time data. Numerous alkyl substituted indoles and carbazoles present in these fractions are indicative of the higher heteroatom content of shale oil. Higher numbers of the polycyclic compounds have been detected using selective ion monitoring including the benzopyrenes. The acidic fraction contains essentially all the isomeric forms of substituted phenols up to C₅-phenols. Some naphthols appear to be present. Further work using gas chromatography/mass spectrometry has shown that the basic fraction consists mainly of alkyl substituted pyridines, quinolines, and acridines.

Tables IV and V give the chemical and physical forms of arsenic and mercury in gaseous effluents from the 10-ton research retort at Laramie, both before and after the offgas burner, which serves as an air emission control device for organic materials at this test facility. Table IV

TABLE III. TRACE ELEMENTS IN SINGLE SAMPLES OF SHALE OIL AND PROCESS WATER
FROM THE ERDA LARAMIE - 150 TON RETORT, RUN NO. 13

	(Concentrations in ppm)	
	<u>SHALE OIL</u>	<u>RETORT WATER</u>
As	5.0	5.9
Br	0.08	0.28
Co	0.37	0.65
Hg	0.20	0.39
Sb	0.008	0.016
Se	0.86	0.98
Zn	2.7	0.43

FIGURE 3. Polycyclic Compounds in Crude Shale Oil

TABLE IV. ARSENIC IN GASEOUS EFFLUENTS FROM THE LARAMIE 10 TON RETORT

I.	Before Ignition of the Shale	
	GASEOUS	1100 ng/m ³
	PARTICULATE	500 ng/m ³
II.	After Ignition but Before Offgas Burner	
	GASEOUS	15,000 ng/m ³ *
	PARTICULATE	400 ng/m ³
III.	After Ignition and After Offgas Burner	
	GASEOUS	1400 ng/m ³
	PARTICULATE	2500 ng/m ³

* PROBABLY OCCURS MOSTLY IN THE FORM
OF ARSINE (AsH₃)

TABLE V. MERCURY CONCENTRATIONS AND SPECIES IN EFFLUENTS FROM THE LARAMIE 10 TON RETORT BEFORE AND AFTER THE OFFGAS BURNER

<u>Material</u>	<u>BEFORE BURNER</u>	<u>AFTER BURNER</u>
GASEOUS	2300 ng/m ³	* 1300 ng/m ³
PARTICULATE	150 ng/m ³	600 ng/m ³
<u>Chemical Species</u>		
* Hg ⁺⁺		100 ng/m ³
(CH ₃) Hg ⁺		300 ng/m ³
Hg ⁰ + (CH ₃) ₂ Hg		900 ng/m ³

presents arsenic concentrations in the process offgas. Preliminary specification measurements indicate that most of the arsenic at this stage of the process is in the form of rather toxic arsine gas. After the gas has been passed through the offgas burner, the arsenic concentration is reduced, due to dilution with air. After the offgas burner, a large fraction of the arsenic is trapped on a nucleopore filter and appears to be in the form of small arsenic trioxide particles.

The data in Table V show that mercury is largely in gaseous form both before and after the offgas burner. The mercury is seen to be largely in the form of Hg^0 plus dimethyl mercury. Similar data are available for iron, chromium, and zinc, all from the first transition series of the periodic table.

Analysis of the retort offgas before burning the organic constituents by GC/MS shows mostly hydrocarbons. A sample GC/MS trace is shown in Figure 4. Saturated hydrocarbons, alkenes, and alkynes, both as aliphatic and cyclic forms, were present. In addition, aromatic compounds, mostly substituted benzenes, were detected. No heterocyclics were identified in the offgas during this preliminary analysis.

Currently we are analyzing in depth the four shale oil fractions for organic constituents on GC/MS and will quantitatively determine the major organic components. Similar separation and GC/MS studies are underway for bitumen samples extracted from tar sand obtained from a quarry near Vernal, Utah. We are also analyzing the previously described samples of raw and spent shales, oil and process water for a number of additional trace elements using radiochemical neutron activation.

Fossil Fuel "Reference Research Materials": Preparation and Documentation

The objective of this program is to prepare reference research materials from fossil fuel production or conversion pilot plants to be used in toxicity studies. In evaluating the potential biological or environmental hazards associated with fossil fuel processes, it is important that representative materials from these processes, including products, effluents, and wastes, be available and that their composition be established as thoroughly as possible. In this program, representative materials

FIGURE 4. Gaseous Organic Components from 10-Ton Retort

from the more promising pilot operations are obtained, stored in a manner which minimizes degradation, and aliquots made available to ERDA laboratories and others designated by ERDA to conduct biological, health effects and environmental studies.

During the first quarter of this project, our efforts have largely been directed towards acquisition of materials from pilot operations to serve as "Reference Research Materials", together with the design and acquisition of storage and handling facilities. We have fractionated a sample of shale oil into four fractions as a preliminary research material and have supplied aliquots of the fractions to PNL biologists, who have conducted preliminary mutagenicity tests on them. Characterization of the four fractions, using GC/MS and neutron activation, has also been initiated by our group.

For the purpose of preparing reference research materials, two barrels of raw shale oil were acquired from the 150-ton research retort at the Laramie Energy Research Center. Contacts toward obtaining materials from additional sources have been made.

For storage and distribution of the reference research materials, we have considered a number of containers and storage procedures that should minimize degradation or contamination. Our plans are to collect samples in teflon-coated stainless steel barrels, transfer them to amber glass containers, and store them in a refrigerated facility maintained at -2° C under a nitrogen gas atmosphere.

In preparing the preliminary oil shale reference research material, a batch of shale oil from the 150-ton LERC retort was split into four fractions using the separations scheme depicted in Figure 5. The fractions obtained were acidic, neutral, basic, and polynuclear aromatic, plus a residual insoluble tar. The percentages of each of the fractions obtained from the shale oil are shown in Figure 6. We are proceeding with organic analysis, using principally the GC/MS, and with inorganic analysis, using neutron activation. Biological and environmental studies using reference materials will provide meaningful data that may be compared between laboratories and will contribute importantly to the estimation of potential impacts of components of oil shale and coal conversion products and emissions.

FIGURE 5. Extraction Scheme

FIGURE 6. Composition of Shale Oil

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