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JUNE MONTHLY PROGRESS REPORT - ENVIRONMENTAL EFFECT AND CONTROLS FOR COAL-WATER SYSTEMS

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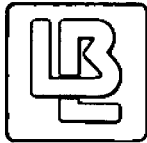
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Fox, Phyllis.

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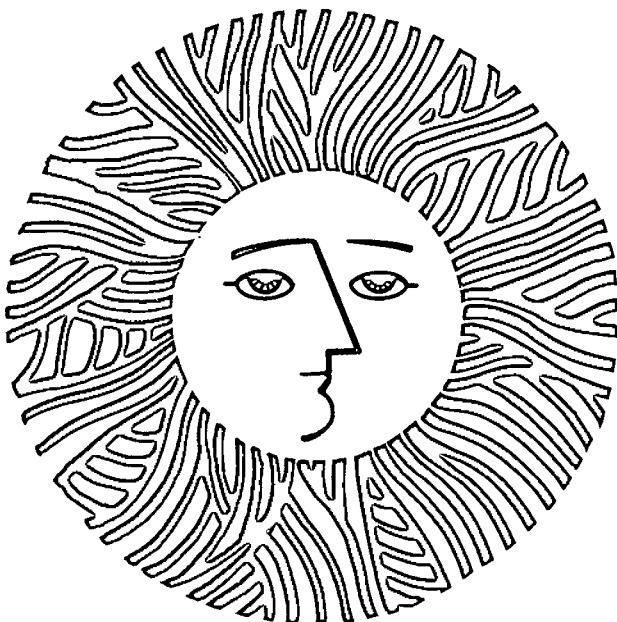
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July 18, 1980

TO: Charles Grua

FROM: Amos Newton and Phyllis Fox

RE: June Monthly Progress Report
Environmental Effect and Controls for Coal-
Water Systems
LBID-248

ADSORPTION OF PHENANTHRENE BY COAL

Because fully deuterated compounds act physically and chemically like ordinary hydrogen-containing compounds, the gas chromatographic (GC) retention times, solubility characteristics, etc. are the same for hydrogen and deuterated compounds. Using gas chromatography-mass spectrometry (GC-MS), the mass spectrometer data system easily separates the deuterated compound from its ordinary hydrogen-containing counterpart, allowing deuterated compounds to be used as unique internal standards.

A coal slurry was made with 4000 ml water to which was added 100 micrograms of perdeuterophenanthrene ($C_{14}D_{10}$) by the addition of 1 ml of a methanol solution with a concentration of 100 $\mu\text{g}/\text{ml}$ of perdeuterophenanthrene. The concentration of $C_{14}D_{10}$ in the water was 25 ppb. To this solution was added 1 kg of Wyodak Coal and the mixture slurried in the laboratory rod-mill for 1 hour. The slurry was centrifuged at 0°C and the clear centrifugate was extracted with hexane. The hexane was evaporated to about 1 ml in a Rotovap. A final concentration step to 10 microliters total volume was made in a small calibrated cone by evaporation with N_2 gas. The water blank (without coal) was extracted and concentrated by the identical procedure.

The recovery of deuterated phenanthrene from the water blank as measured by GC/MS was 85%. No ordinary phenanthrene or deuterophenanthrene was observed in the coal slurry water, and more than 99.98% of the perdeuterophenanthrene was extracted from the water by the coal. A concentration of 5 parts per trillion (5 parts $C_{14}D_{10}$ in 10^{12} parts water) would have been observed.

ORGANICS IN COAL SLURRY WATER

A collection of 10 liters of clear centrifugate from coal slurry (1 kg coal/4 l of water) was extracted with hexane and the extract concentrated to 100 microliters. No detectable chromatographic peaks were observed when injected into the Loenco GC in 5 μ l injections. At the sensitivity level used for the flame ionization detector (FID), the injection 10 ng of an organic impurity is readily detected as a distinct chromatographic peak.

No organic compounds were found in the coal slurry waters. An upper limit of 20 parts per trillion can be placed on the occurrence of any non-polar (i.e., extractable with hexane) organic compound in the water.

In another type of extraction procedure, 1 liter of coal slurry water was extracted with 0.5 ml of n-pentane. The pentane extract was concentrated to 10 microliters. No compounds were observed in the extract. An upper limit of 10 parts per trillion extractable organics would have been observed.

EXTRACTION OF ANISOLE FROM WATER

The distribution coefficients between water and various organic solvents for anisole (methyl-phenyl ether) have been determined at 25°C.

Solvent	$K = \frac{\text{Concentration anisole in solvent}}{\text{Concentration anisole in water}}$
Benzene	16.8
Diethyl Ether	15.2
Methylene Chloride	15.0
Hexane	17.5

DETERMINATION OF PHENOLS IN WATER BY METHYLATION

The distribution coefficients show that anisole has favorable extraction characteristics and the difficulty in determining phenols

in water must be due to unfavorable yields in the methylation process. The methylation of phenol by dimethyl sulfate is a second order process while the destruction of the methyl sulfate reagent by hydrolysis is a pseudo first order process. The rate of methylation of phenol is dependent on both the concentration of phenol and the concentration of dimethyl sulfate. The factors which can be altered are the concentration of NaOH used, the amount of dimethyl sulfate added, and the temperature.

These factors have been varied and the results are shown in Table 1, which contains some inconsistencies. Experiments 3, 4, and 7 appear to be inconsistent. In general, however, with adequate dimethyl sulfate and sodium hydroxide added, it appears that about 40% yields can be expected. By the addition of perdeuterophenol to the coal slurry water as an internal standard, the concentration of ordinary phenol in the water can be calculated from the yield of methyl ether of the perdeuterophenol and ordinary phenol. If no anisole is observed, from ordinary phenol, then an upper limit on the concentration of phenol in the coal slurry water can be established.

Table 1. Summary of experimental results in the methylation of phenol.

Expt. No.	Phenol (mg phenol/liter)	Dimethyl/Sulfate ml per liter	NaOH (Normality)	Temp (°C)	Yield Anisole (%)
1	1000	15	2	24	22
2	1000	15	2	24	17
3	1	15	2	24	4.3
4	500	15	2	24	4.3
5	100	50	1	26	43
6	10	150	0.5	26	39
7	110	150	1	2	82
8	2	50	1	10	36
9	10	150	0.1	2	0 ^a
10	10	150	0.5	2	20
11	10	150	1	2	39

^a No anisole detected, dimethyl sulfate present.

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

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