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

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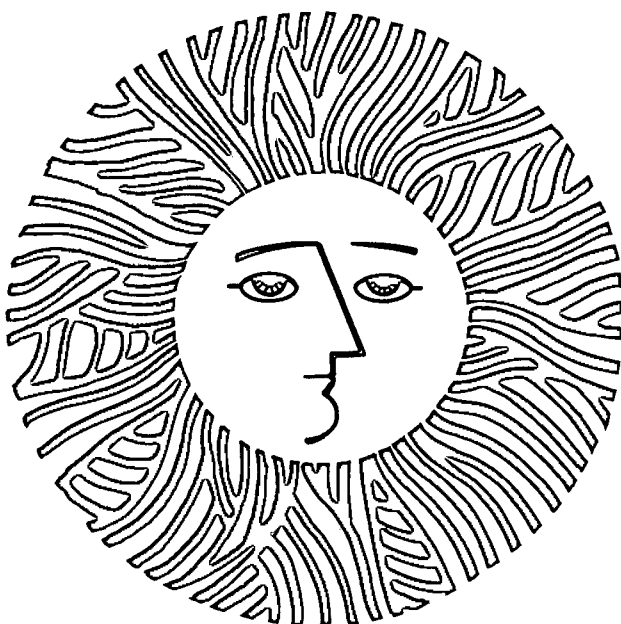
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November 6, 1980

TO: Charles Grua

FROM: Amos Newton and Phyllis Fox

RE: Monthly Progress Report for October
Environmental Effect and Controls for
Coal-Water Systems
LBID-310

PHENOLS IN SLURRY WATER

Previous studies have been made with Wyodak coal which is a sub-bituminous coal. Studies have now been started on Illinois No. 6 coal, a bituminous coal. The slurry from this coal is much more easily clarified than the slurry from Wyodak coal. Based on visual observation of water clarity, the system appears to be cleaner.

Investigations have been made to determine the phenol content of slurry water from Illinois No. 6 coal. A slurry of 20% coal in water was prepared. This was centrifuged and perdeuterophenol was added to the slurry water as an internal standard. Perdeuterophenol was added in various experiments at concentrations of 5, 10, and 100 ppb. The spiked solution was made basic with NaOH and methylated at 0°C with a large excess of dimethyl sulfate. Hexane extracts of these solutions were analyzed by GC/MS for anisole and 2,3,4,5,6-deuteroanisole. Deuteroanisole was observed only with the 100-ppb spike, and no anisole was observed in any of the solutions. The yield of deuteroanisole is less than 20% of that observed in Wyodak coal slurry water methylated by the same procedure. It is apparent that there is a component of the slurry water from Illinois No. 6 coal which is acting as a catalyst for the hydrolysis of dimethyl sulfate and thereby preventing its methylation of the phenol and deuterophenol present at the parts-per-billion levels.

On the assumption that the catalyst destroying the dimethyl sulfate reagent was a metal, EDTA was added to the slurry water to chelate it. The principal metallic ions in the coal slurry

water were iron, calcium, and strontium. The addition of various amounts of EDTA to the slurry water resulted in no change in the yield of deuterioanisole produced. Steam distillations of phenols from deuterophenol-spiked Illinois No. 6 coal slurry water and ordinary water spiked with perdeuterophenol and acidified with H_3PO_4 were made and the resulting distillates were each methylated. The yield of deuterioanisole from the distillate of Illinois #6 coal slurry water was the same as from an undistilled sample. Methylation of the distillate of spiked tap water gave the normal yield of deuterioanisole. This suggests that the interference is not a metallic ion.

Further studies on coal slurry water from Illinois No. 6 coal are in progress with attention centered on the development of a method for determining phenols in such water.

POLYCYCLIC AROMATIC HYDROCARBONS IN SLURRY WATER

A standard solution of some representative perdeuteropoly-cyclic aromatic hydrocarbons has been prepared in ethanol. Experiments are in progress in which these hydrocarbons are added to water before slurry preparation to determine possible contamination of coal slurry water from Illinois No. 6 coal with these compounds. The compounds and the GC/MS sensitivity for the molecular ion of each are shown in Table 1.

Table 1. GC/MS sensitivities for perdeutero polycyclic aromatic hydrocarbons.

Compound	M/Z Molecular Ion	Emergence time in seconds ^a	Ion Intensity ^b for 100 ng injected
Acenaphthene-d ₁₀	164	2191	16000
Phenanthrene-d ₁₀	188	2730	17500
Pyrene-d ₁₀	212	2750	16000
p-terphenyl-d ₁₄	244	3325	12300
1,2-benzanthracene-d ₁₂	240	3610	12500
Chrysene-d ₁₂	240	3625	13000
Perylene-d ₁₂	264	4325	3400

^aOV-101 WCOT capillary column.

^bIntensity in arbitrary units.

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