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LAWRENCE BERKELEY LABORATORY
Room: 128 Bldg.: 70 Ext.: 6241

December 10, 1980

TO: Brian Harney and Art Hartstein

FROM: J. Cantor, L. Joseph, and P. Fox

RE: November Monthly Progress Report
The Partitioning of Major, Minor, and Trace Elements
During Simulated In-Situ Oil Shale Retorting
LBID-323

ARSENIC SPECIATION STUDIES

Matrix effects in ion-exchange HPLC/GFAA analysis of retort waters and shale leachates were investigated in November. We have tentatively concluded that ions other than those of interest are present in some waters in sufficient quantities to function as eluting counter-ions. This causes some compounds to be eluted wholly or in part by the sample itself and to emerge at the solvent front. Charge balance data for Omega-9 water (the only water for which this information is available) show an ion concentration comparable to that of the eluting buffer, which tends to support this conclusion.

Arsenite and dimethylarsinic acid are apparently being affected in this way in our present separation scheme. In addition, this implies that the "neutral" peak we've previously reported is at least, in part, an artifact caused by the eluting ability of the sample matrix. Attempts to separate arsenite and dimethylarsinic acid in an artificially anion-enriched matrix have not been successful.

Methyl arsonic acid, phenylarsonic acid, and arsenate in high ionic strength matrices all show greater retention times than those of standards prepared in distilled water. However, these three compounds are still sufficiently well resolved to identify by spiking with known compounds.

The samples for the interlaboratory studies are now being analyzed for those compounds that can be reliably identified. To date, arsenate has been identified in Paraho water. Meanwhile, work is beginning on alternate means of separating these compounds. We have enlisted the generous assistance of a local HPLC column manufacturer in our search for a more

effective method of separation.

IDENTIFICATION OF POTENTIAL ORGANIC COMPOUNDS AS LIGANDS OF METALS IN SHALE PROCESS WATERS

Work is now in progress to characterize the carboxylic acids present in Occidental's Retort No. 6 gas condensate and retort waters by capillary-column gas chromatographic analysis of their methyl ester derivatives. Because of the relatively dilute nature of the gas condensate water, our standard methylation and work-up procedure (used for the retort water) yielded extremely limited information. This month we were successful in modifying our technique, thus allowing for carboxylic acid analysis of gas condensate waters in general. The two methods are summarized in Table 1. Tentative identifications are now being made by comparison of retention times with those of known standards. Gas chromatography-mass spectrometry analysis is concurrently being done to confirm these identifications.

Table 1. Revised procedure for analyses of fatty acids in gas condensate.

Standard Procedure	Modified Procedure
-lyophilize 20 ml process water	-lyophilize 40 ml process water
-react residue with 20 ml, 14% BF_3/MeOH	-react residue with 30 ml, 14% BF_3/MeOH
-add 20 ml water to decompose excess BF_3	-add 40 ml water to decompose excess BF_3
-extract with 3x10 ml portions of benzene	-extract with 4x10 ml portions of benzene
-concentrate benzene extracts to 1 ml by fractional distillation	-concentrate benzene extracts to 1 ml by fractional distillation, further concentrate to 200 μm with nitrogen
-analyze concentrate by GC	-analyze concentrate by GC

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