Title
THE PARTITIONING OF MAJOR, MINOR, AND TRACE ELEMENTS DURING SIMULATED IN-SITU OIL SHALE RETORTING

Permalink
https://escholarship.org/uc/item/60g0h75r

Authors
Fish, Richard
Giauque, Bob
Fox, Phyllis.

Publication Date
1980-04-01
DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.
April 10, 1980

TO: Art Hartstein

FROM: Richard Fish, Bob Giauque, and Phyllis Fox

RE: March Monthly Progress Report
The Partitioning of Major, Minor, and Trace Elements
During Simulated In-Situ Oil Shale Retorting
LBID-196

NAVY CORE PROJECT

All analyses, data reduction, and quality control checks were completed for both core holes and a draft of the final report was written.

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

We have continued our studies on the isolation and identification of organic ligands potentially associated with trace metals in oil shale process waters. The experimental approach, as before, includes extraction of the retort water (pH ~9) with methylene chloride, concentration of the methylene chloride extract, and adsorption of this residue on a dry Florisil column. The Florisil column is then eluted with hexane, methylene chloride, and isopropyl alcohol under controlled gradient conditions using both high performance liquid chromatographic pumps. Fractions of 4.0 ml are collected automatically with a fraction collector and those that have ultraviolet absorptions at 254 nm are grouped, concentrated, and analyzed using capillary column gas chromatography. The grouped fractions of interest are then analyzed by capillary column gas chromatography/electron impact mass spectrometry.

We have presently analyzed three retort water samples: Geo-kinetics', Laramie Energy Technology Center's 150-ton retort water, and Occidental's retort water. Using the techniques discussed, we have identified a series of ligands which include
methyl-substituted phenols, substituted pyridines, aliphatic nitrogen heterocycles, methyl-substituted quinoline compounds, and cyclic aliphatic ketones. It is interesting to note that all three retort water samples had a somewhat similar profile of bands of compounds absorbing at 254 nm in the ultraviolet (Figures 1, 2, and 3).

We are also measuring trace element concentrations in the various fractions eluted from the Florisil column. In this way, we hope to be able to correlate the organic ligands with the metal content and thereby infer the types of organometallic compounds and complexes that may be present. This month, we initiated these analyses for Zn using graphite furnace atomic absorption spectrometry. A methylene chloride extract (100 ml) of 150-ton retort water gave a Zn value of approximately 13 ppm. Analyses of several fractions containing the nitrogen aliphatic and aromatic heterocycles yielded Zn concentrations of about 1 ppm.

RHF:lmv
Figure 1. Plot of solvent composition versus fractions collected for a gradient elution experiment with Geokinetics' retort water. Bands represent ultraviolet absorption at 254 nm.
Figure 2. Plot of solvent composition versus fractions collected for a gradient elution experiment with 150-ton retort water. Bands represent ultraviolet absorption at 254 nm.
Figure 3. Plot of solvent composition versus fractions collected for a gradient elution experiment with Occidental retort water. Bands represent ultraviolet absorptions at 254 nm.
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.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.