

Lawrence Berkeley National Laboratory

Recent Work

Title

THE PARTITIONING OF MAJOR, MINOR, AND TRACE ELEMENTS DURING SIMULATED IN-SITU OIL SHALE RETORTING - MAY MONTHLY PROGRESS REPORT

Permalink

<https://escholarship.org/uc/item/8254p89q>

Authors

Fish, Richard
Fox, Phyllis.

Publication Date

1980-06-01

no files

UC-91 & 4
LBID-239 c.1



Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

**ENERGY & ENVIRONMENT
DIVISION**

For Reference

Not to be taken from this room



RECEIVED
LAWRENCE
BERKELEY LABORATORY

MAY 1981

LIBRARY AND
DOCUMENTS SECTION

LBID-239
c.1

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.

June 13, 1980

TO: Brian Harney and Art Hartstein
FROM: Richard Fish and Phyllis Fox
RE: May Monthly Progress Report
The Partitioning of Major, Minor, and Trace Elements
During Simulated In-Situ Oil Shale Retorting,
LBID-239

SPECIATION OF INORGANIC AND ORGANOMETALLIC COMPOUNDS IN OIL
SHALE PROCESS WATERS

Dr. Ken Jewett, a visiting scientist from the National Bureau of Standards, assisted in setting up the high performance liquid chromatograph-graphite furnace atomic absorption detector (HPLC-GFAA) combination.

We reanalyzed the seven process waters previously studied at NBS for inorganic and organoarsenic compounds. This allowed us to ascertain that several of our previous tentative identifications were misleading. By diluting the process water samples, we were able to eliminate the organic matrix problem and dramatically improve our separations. This work indicates that arsenate (AsO_4^{3-}) was the predominant compound in all of the process waters studied. Smaller but significant amounts of phenyl- and methylarsonic acid were also present in most of the process waters studied.

The samples were spiked with known arsenic standards to enable us to more clearly define our identifications. We are presently attempting to define the detection limits for arsenite, a known carcinogen, that we have not been able to detect in any of the process waters to date.

We have also made a simple collection device for preping unknown inorganic or organometallic compounds by placing a peristaltic pump in line with the effluent being removed from the laminar flow cell. The effluent from the outlet of the

pump is collected by an automatic fraction collector. As far as we know, there have been no attempts by people using HPLC-GFAA to preparatively isolate unknown compounds emerging from the HPLC column during GFAA detection.

We are presently investigating the possible origin of the inorganic and organoarsenic compounds identified in process waters by repeating our speciation studies on artificial retort water samples. These samples were produced by contacting samples of oil shale and shale oil with an ammonium bicarbonate solution to simulate the contact between water, dissolved gases, and raw shale within an in-situ retort.

ARSENIC COORDINATION CHEMISTRY

Arsenic occurs at high concentrations, 5 to 50 ppm, in shale oils and is considerably enriched compared to conventional crudes which typically have As concentrations of about 0.01 ppm. These elevated levels are significant from both an environmental and processing standpoint. If shale oils are combusted directly, as in an oil-fired boiler, As emissions may be a factor of 1000 or more higher than from boilers fired with conventional crude. Additionally, As is a known cracking catalyst poison and must be removed before shale oil can be refined. Previous attempts to remove As on an adsorption bed have limited commercial application due to the high costs associated with bed replacement or regeneration. Therefore, we have initiated work to develop an alternate method to remove As from shale oils using the techniques and knowledge we have developed in oil shale speciation work.

Our approach is to coordinate various arsenic species with ligands that can be readily placed in a polymeric backbone. We have begun a study to ascertain how these species, e.g., arsenate, methyl- and phenylarsonic acids, and trialkylarsine oxides, coordinate with a variety of new organic ligands being synthesized by colleagues in LBL's Materials and Molecular Research Division (MMRD). Several new arsenic coordination complexes were synthesized and their structures elucidated by 90 and 250 MHz

nuclear magnetic resonance spectroscopy. We hope to be able to define their rates of formation and continue to evaluate more functionally substituted ligands capable of coordinating any arsenic compound.

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.

TECHNICAL INFORMATION DEPARTMENT
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720