

# Lawrence Berkeley National Laboratory

## Recent Work

### Title

DISTRIBUTION OF As, Cd, Hg, Pb, Sb, AND Se DURING SIMULATED IN-SITU OIL SHALE RETORTING

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# Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

## ENERGY & ENVIRONMENT DIVISION

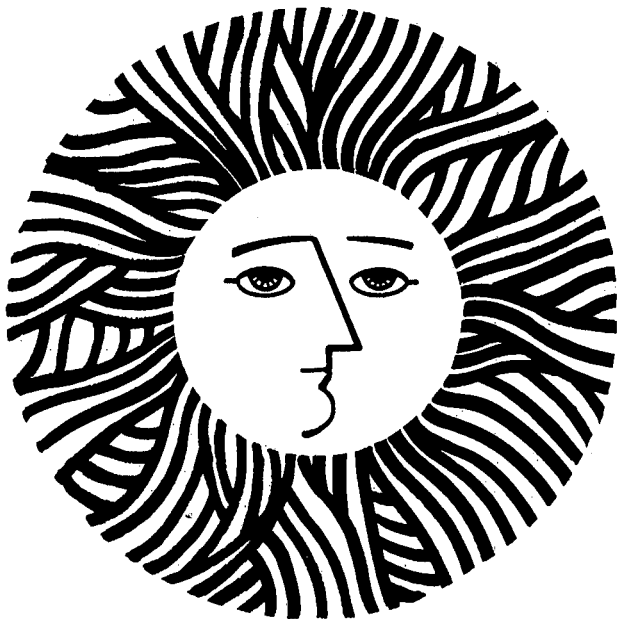
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LBID-363 c.1

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February 9, 1981

TO: Pat Fair

FROM: D. C. Girvin and A. T. Hodgson

RE: Monthly Progress Report for January  
Distribution of As, Cd, Hg, Pb, Sb, and Se During Simulated In-Situ  
Oil Shale Retorting  
LBID-363

TASK 1. ANALYTICAL METHODS FOR OIL AND WATER SAMPLES

No development work on an analytical method for cadmium in shale oil samples was conducted during January. Neutron activation analysis and x-ray fluorescence analysis of oils from retort runs LBL-04 and LBL-05 were completed. These instrumental methods produced widely different results for both arsenic and selenium. The cause of these discrepancies must be resolved before additional samples are submitted for analysis.

TASK 2. ANALYTICAL METHODS FOR GAS SAMPLES

A design for a modified HGA-2000 graphite furnace for offgas Hg and Cd analyses was presented in the October monthly progress report (LBID-311). The construction of this furnace is now complete. During initial tests, severe arcing occurred between the graphite tube and the supporting graphite cones which greatly reduced the life of the graphite tube. The cause and means to control this arcing were discovered during subsequent tests. Additional performance evaluations are required before the furnace will be ready for use with the Cd ZAA gas monitor.

TASK 4. LABORATORY PARTITIONING STUDIES

An inert gas laboratory retort run, LBL-07 was conducted on January 14. All retorting equipment and operating parameters were identical to those used for retort run LBL-06 in December. The primary objective of run LBL-07 was to validate the results of the previous run. The secondary objective was to collect and analyze gas phase amalgamation samples for comparison with ZAA Hg monitor data.

The results of run LBL-06 were essentially duplicated during run LBL-07. Eighty-five percent of the Hg originally present in the raw shale was released to the offgas phase by the time the retorting temperature had reached 320°C. The Hg monitor was kept operational by the addition of O<sub>2</sub> and N<sub>2</sub> until 340°C when oil production resulted in the extinction of the analytical line. An additional 16% of the original Hg was detected in this 20°C interval. The additional Hg may have evolved from the retort during this period. Alternately, the Hg may have previously trapped out on a cool (85°C) section of tubing at the inlet of the ZAA furnace and evolved upon addition of O<sub>2</sub> when combustion of organic residue elevated the temperature of the tubing. Analyses of the other retorting products have been completed. Approximately 7% of the initial Hg partitioned to the oil, less than 1% of the Hg partitioned to the water, and less than 2% remained in the spent shale. Total Hg recovery for LBL-07 was, therefore, about 110%. A preliminary estimate of the total propagated error for the Hg balance has been made based upon the analytical errors for each of the retorting components. This estimate indicates that a recovery error of ±10% is to be expected.

Ten offgas samples were obtained during the LBL-07 Hg peak with Au-amalgamation tubes. Offgas was routed from the heated stainless steel sample line to the amalgamation tubes with 50 cm of polyethylene tubing. Mercury concentrations by the Au-amalgamation technique were, on the average, 40% of the concentrations determined by the ZAA Hg monitor. This difference may perhaps be attributed to loss of Hg to the room temperature polyethylene tubing. Loss to this potential sink could be eliminated by the collection of Au-amalgamation samples directly from the heated sample line. This arrangement would require rapid cooling of the offgas in the amalgamation tubes.

Gas composition was determined throughout run LBL-07 with the automated gas chromatographic system.

#### PROJECTED WORK

The projected work for February is as follows:

##### Task 1. Analytical Methods for Oil and Water Samples

We will resume the evaluation of analytical methods for cadmium in oil and shale.

Task 2. Analytical Methods for Gas Samples

We will continue to evaluate the performance of the modified HGA-2000 furnace. The Cd ZAA spectrometer will be tested using a Perkin Elmer, single slot, atomic absorption burner head and aqueous Cd standards.

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