UC-11 & 91
LBID-335

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ENERGY & ENVIRONMENT DIVISION

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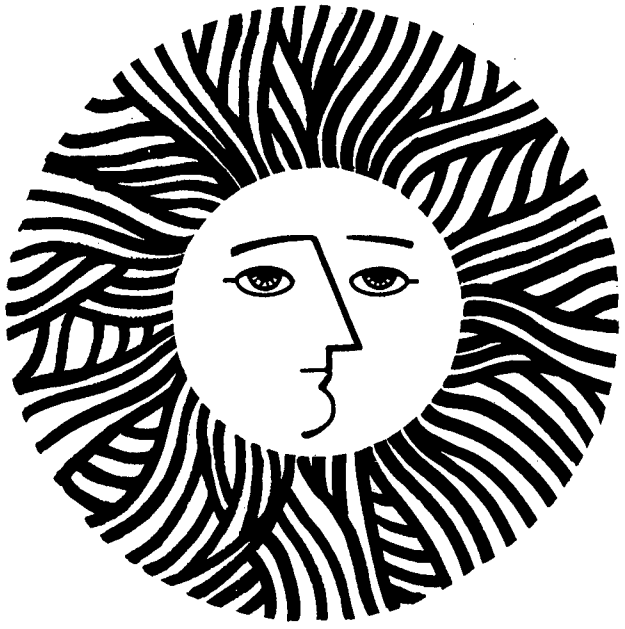

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LAWRENCE BERKELEY LABORATORY
Room: 128 Bldg.: 70 Ext.: 6698

January 5, 1981

TO: Bob Thurnau and Pat Fair

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

RE: December Monthly Progress Report
Distribution of As, Cd, Hg, Pb, Sb, and Se During Simulated In-Situ Oil Shale Retorting
LBID-335

TASK 1. ANALYTICAL METHODS FOR OIL AND WATER SAMPLES

We have initiated an evaluation of Parr acid digestion bombs for the decomposition of shale oil samples prior to Cd analysis by graphite furnace atomic absorption spectroscopy. The same technique will be used for shale samples. The accuracy of the technique is being determined by analysis of organo-Cd standards prepared in an oil base, by analysis of National Bureau of Standards SRM 1633 (coal fly ash), and by recovery of inorganic Cd spikes added to samples prior to decomposition. Precision will be determined from replicate analyses.

TASK 2. ANALYTICAL METHODS FOR GAS SAMPLES

The modified HGA-2000 furnace for offgas Hg and Cd analyses was not completed in time for testing during December.

TASK 4. LABORATORY PARTITIONING STUDIES

An inert gas laboratory retort run, LBL-06, was conducted on December 11 using the stainless steel retort vessel and a heated offgas line. The shale grade, particle size distribution (-1/4 inch to +30 mesh), nitrogen input-gas flow rate (2.0 L/min), and heating rate (1°C/min) were the same as those used for previous runs. The objectives of the run were: (1) to determine if the use of the stainless steel retort vessel and heated offgas line would improve the Hg balance; (2) to collect and analyze additional gas phase amalgamation samples for comparison with ZAA Hg monitor data; and (3) to collect particulate samples from the offgas and analyze these samples for Hg.

During retort run LBL-06, the entire shale column was heated uniformly with a maximum temperature differential between the top and bottom of the column of only 10°C. The 1/2 inch O.D. stainless steel sample line connecting the ZAA gas monitor to the retort was electrically heated to a minimum temperature of 120°C. The average temperature of the sample line was 175°C. Mercury in the offgas flowing through this line was continuously monitored. From the monitoring data, we determined that 80% of the Hg originally present in the raw shale was released to the offgas phase. This percentage is approximately two times greater than percentages determined for previous retort runs which utilized the ceramic retort vessel and cold offgas line. Preliminary analysis of a composite oil sample indicated that 5% of the Hg partitioned to the oil. Less than 1% of the Hg partitioned to the water, and less than 2% remained in the spent shale. Thus, the use of the stainless steel retort vessel and the heated offgas line greatly improved the Hg balance.

Thirteen offgas samples were obtained during LBL-06 with Au-amalgamation tubes. These samples were collected from the original unheated, stainless steel, offgas line. Mercury concentrations by the Au-amalgamation technique were approximately one-third of corresponding concentrations determined by the ZAA Hg monitor connected to the heated sample line. Since we had previously determined that there was good general agreement between the two analytical methods, the difference in concentrations between the two sets of data can be largely attributed to the difference in temperatures between the two sample lines. Consequently, no additional data were obtained for the Au-amalgamation and ZAA method comparison.

Two particulate samples were obtained from the offgas during the LBL-06 Hg peak. The filters and holders used to collect the samples were heated to 120°C to prevent condensation of water. Particulate phase Hg concentrations were determined to be less than 3% of total offgas Hg concentrations.

Gas composition was determined throughout run LBL-06 with the automated gas chromatographic system.

TASK 5. FIELD STUDIES

The analysis of Hg data obtained from Lawrence Livermore National Laboratory's retort run L-3 has been completed.

PROJECTED WORK

The projected work for January, 1981 is as follows:

Task 1. Analytical Methods for Oil and Water Samples

We will continue to evaluate analytical methods for cadmium in oil and shale.

Task 2. Analytical Methods for Gas Samples

Preliminary heating and pressure testing of the modified HGA-2000 furnace will be conducted. Any necessary modifications will be initiated.

Task 4. Laboratory Partitioning Studies

We are continuing to evaluate the results of retort run LBL-06. The next retort run will be conducted in January. The objectives, retort configuration, and operating conditions for this run will be the same as those for retort run LBL-06.

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.

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