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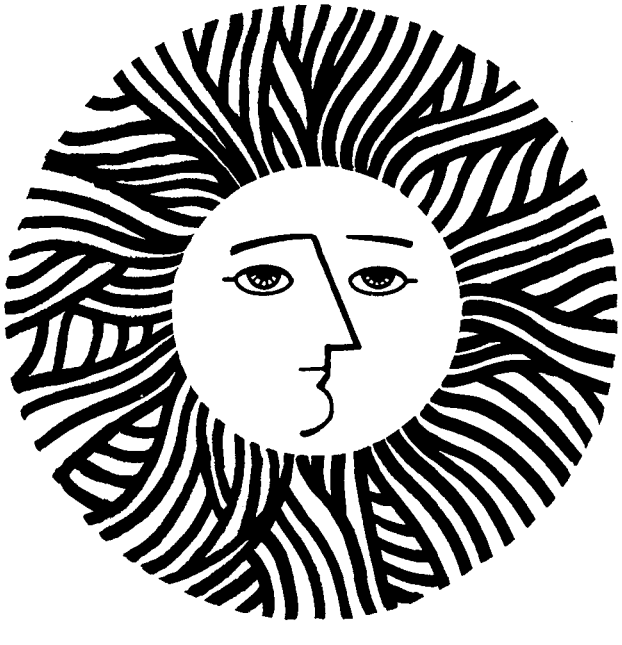

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LAWRENCE BERKELEY LABORATORY
Room: 128 Bldg.: 70 Ext.: 6698

March 13, 1980

TO: Bob Thurnau

FROM: D. C. Girvin, A. T. Hodgson and P. Fox

RE: February Monthly Progress Report
Distribution of As, Cd, Hg, Pb, Sb and Se During
Simulated In-Situ Oil Shale Retorting
LBID-179

TASK 1. ANALYTICAL METHODS FOR OIL AND WATER

During February we investigated the use of ozone as an oxidant for retort water prior to Hg analysis by cold vapor atomic absorption spectroscopy (AAS). The advantage of ozone over potassium permanganate/persulfate oxidation is that ozone is noncontaminating. A variety of ozone oxidation procedures were evaluated, and a tentatively useful procedure has been selected for further investigation. A literature review of procedures for preservation and storage of water samples for subsequent Hg analysis has been initiated.

The batch sample Hg ZAA spectrometer has been assembled and is operational. It will be used for the analysis of Hg in oil and in raw and spent shale.

TASK 2. ANALYTICAL METHODS FOR GAS SAMPLES

An attempt was made to use the EPA reference method, designed for determination of particulate and gaseous Hg emissions from stationary sources, as a check on the accuracy of the dynamic calibration device used with the ZAA Hg monitor. This method employs an acidic iodine monochloride (ICl) solution for collection of Hg. Although the ICl solution readily removes inorganic Hg from inert gas streams, we experienced difficulty in reducing and purging the Hg from the solution for analysis by cold vapor AAS. In addition, the ICl method is not compatible with

the Perkin Elmer MHS-10/sodium borohydride cold vapor technique which we have been using. Because of these difficulties and the importance of obtaining a check on the calibration device, a literature review of alternate methods for collection of gaseous Hg has been initiated. Amalgamation of Hg on gold- or silver-plated glass beads coupled with thermal desorption and cold vapor AAS detection appears promising.

The circuitry for the Hg light source temperature controller has been designed. This controller will sense the light source temperature and drive a resistance heating element which will buck the thermoelectric heat pump in the cooling mode.

TASK 4. LABORATORY PARTITIONING STUDIES

No laboratory retort run was conducted during this reporting period. Analysis and evaluation of data from LBL-2 were continued during February. The volume of offgas was determined, the other products were weighed, and a material balance was calculated. Only 97.7% of the original shale mass could be accounted for. A portion of the loss is due to accumulation of oil inside the retort vessel and other components. We have weighed all small components to reduce the error. Other factors which may also have contributed to the nonclosure of the material balance equation are being investigated. No elemental balance has been calculated as the masses of Hg in the raw and spent shale and in the oil have not yet been determined.

PROJECTED WORK

The projected work for March is as follows:

Task 1. Analytical Methods for Oil and Water Samples.

- We will continue working on the development of a method for the determination of total Hg in retort water.

Task 2. Analytical Methods for Gas Samples.

- We will attempt to implement an alternate method for the determination of Hg in gas streams as a check on the accuracy of the ZAA dynamic calibration device.
- The new light source temperature controller for the Hg ZAA field monitor will be constructed.

Task 4. Laboratory Partitioning Studies.

- Raw shale for approximately 15 retort runs will be ground, split into equal 5.5 kg portions, and sampled for elemental analyses.
- A retort run for the purpose of obtaining additional Hg partitioning data will be conducted in late March or early April.

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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