Title

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

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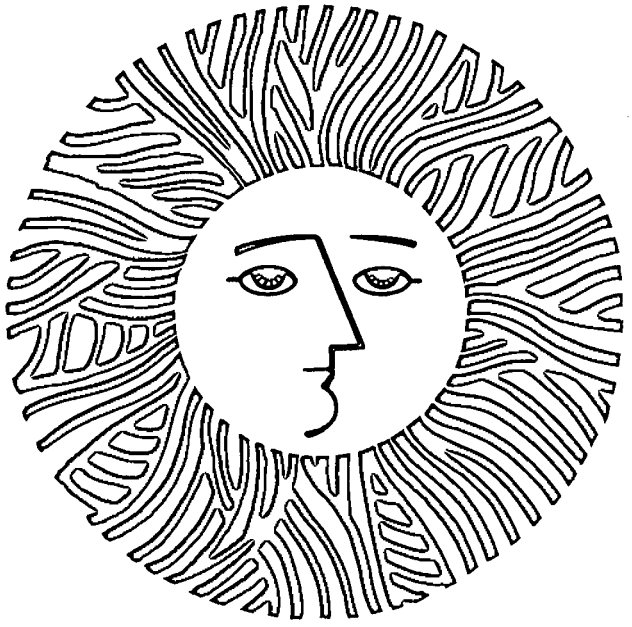

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October 14, 1980

TO: Bob Thurnau and Pat Fair

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

RE: September Monthly Progress Report
Distribution of As, Cd, Hg, Pb, Sb, and Se
During Simulated In-Situ Oil Shale Retorting
LBID-303

TASK 1. ANALYTICAL METHODS FOR OIL AND WATER

In July, five samples of shale oil were submitted for neutron activation analysis (NAA). The results of this analysis were received in September and were compared to the results previously obtained with other analytical methods. In general, Hg concentrations obtained with the latest version of the WO_3 combustion - amalgamation - ZAA method described in the July monthly report agree, within measurement errors, with concentrations determined by NAA. However the analytical errors for the WO_3 method are higher than those for NAA. Decomposition of shale oil in acid digestion bombs and analysis using an amalgamation technique produced Hg concentrations that were one-half of concentrations by NAA.

Since Hg concentrations by NAA are more precise and inherently more accurate than concentrations obtained by the other techniques, NAA will be used for Hg analysis of all oil samples. The disadvantage of NAA is that total sample analysis time is approximately two months. Consequently, the WO_3 combustion - amalgamation - ZAA method will be employed for the purpose of initially characterizing oil samples.

Ambiguities in Hg concentrations in the oils produced during retort runs LBL-02 and LBL-03 have been eliminated by the NAA results. Therefore, it is now possible to calculate elemental balances for these runs. These calculations confirm our earlier suspicion that Hg either was lost to the ceramic retort vessel or escaped detection in the gas phase.

TASK 2. ANALYTICAL METHODS FOR GAS SAMPLES

During September, we examined the response of the ZAA Hg signal to superheating the gas stream with HGA-2000 furnace before the gas stream enters the stainless steel (SS) ZAA furnace. Nitrogen was used as the carrier gas for this experiment since N_2 is the major constituent (80-90%) of offgas from inert gas retorting at temperatures below 750°C. The addition of 2% (v/v) H_2S to the carrier gas did not affect ZAA response to a constant concentration of Hg with the SS ZAA furnace operating at 900°C. This is consistent with results described in the November 1979 monthly report for ZAA furnace temperatures of 750°C. However, when the N_2-H_2S-Hg mixture was passed through the HGA furnace at approximately 2800°C before entering the ZAA furnace, a 15% reduction in ZAA response to the same concentration of Hg was observed. We suspect that the high temperature graphite surfaces in the HGA produced sulfur radicals which combined with a fraction of the elemental Hg upon exiting the HGA and thus rendered this Hg undetectable with the cooler ZAA furnace. The reduction in ZAA response, therefore, appears to be an artifact of the series combination of the HGA and ZAA furnaces. Consequently, this series combination cannot be used to eliminate the H_2S suppression as initially suspected, and all work on this arrangement has been suspended. The experiment did, however, demonstrate that gas stream reactions which mask Hg can occur and that a different technique is required to determine if all of the offgas Hg is detected using the ZAA furnace.

A more desirable approach to the problem of detection is to pass the ZAA optical beam through the hot region of the HGA furnace at right angles to the gas flow and thus, measure Hg at the point of maximum temperature before recombination can occur. This strategy, which we plan to use for offgas Cd measurements, will be attempted for Hg during retort run LBL-06 or LBL-07. An HGA-2000 will be modified to serve as a prototype of a Cd furnace, which is now in the design stage. The Cd ZAA gas monitor, assembled last year, will be fitted with a Hg light source and the Cd prototype furnace and will be run in conjunction with the existing ZAA Hg gas monitor equipped with a SS furnace. Simultaneous measurements of Hg can then be made with these instruments. This test will provide both a shakedown for the prototype Cd furnace and an evaluation of the ability of the SS furnace to atomize all the Hg in the offgas stream.

The Cd ZAA light source and light source high voltage driver were not tested this month due to the emphasis placed on the design of the new Cd furnace.

TASK 3. DESIGN AND CONSTRUCTION OF EXPERIMENTAL APPARATUS

The stainless steel retort vessel was constructed, loaded with shale for the next run, and installed in the furnace.

TASK 5. FIELD STUDIES

The analysis of Hg in spent shale samples from Lawrence Livermore Laboratory's interrupted steam-air retort run, L-3, is well underway. The batch sample ZAA is being used for this analysis. Preliminary results for the vertical profile samples taken at 6 inch intervals over the entire 20 foot length of the shale bed show a significant vertical concentration gradient with the highest Hg concentrations occurring in the lower intervals of unretorted shale.

PROJECTED WORK

The projected work for October is as follows:

Task 1. Analytical Methods for Oil and Water

No methods development work is scheduled for October.

Task 2. Analytical Methods for Gas Samples

We will initiate construction of a prototype Cd ZAA furnace using an HGA-2000 furnace. Work also will begin on the configuration of the Cd ZAA gas monitor with a Hg light source in order to evaluate the prototype Cd furnace and to make a comparison between the SS furnace and the prototype Cd furnace for Hg analysis.

Task 4. Laboratory Partitioning Studies

Retort run LBL-05 will be conducted using the new SS retort vessel. The major objectives for LBL-05 are: (1) to determine if losses of Hg from the vapor phase are reduced using the SS retort vessel; and (2) to obtain additional data for the Au-amalgam and ZAA method comparison.

Task 5. Field Studies

Mercury analysis of shale samples from LLL's L-3 steam-air retort run will continue.

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