Title

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

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November 7, 1980

TO: Bob Thurnau and Pat Fair

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

RE: October Monthly Progress Report
Distribution of As, Cd, Hg, Pb, Sb, and Se
During Simulated In-Situ Oil Shale Retorting
LBID-311

TASK 2. ANALYTICAL METHODS FOR GAS SAMPLES

Modification of an HGA-2000 furnace for offgas Hg and Cd analyses was initiated in October. A standard HGA-2000 furnace, designed for discrete liquid samples, is shown in Figure 1a. The optical path in this furnace runs along the radial axis of the graphite tube. We have redesigned the furnace so that the optical path passes through the hot region of the graphite tube perpendicular to the gas flow along the radial axis as shown in Figure 1b. With this configuration, Hg and Cd measurements can be made directly in the hottest region of the furnace. A baffle in the graphite tube increases heating efficiency.

TASK 3. DESIGN AND CONSTRUCTION OF EXPERIMENTAL APPARATUS

A Hewlett-Packard model 5880 gas chromatograph equipped with an automatic gas sampler and configured with columns for refinery gas analysis was plumbed into the retort offgas line and given a shakedown test during the October retort run. This fully automated gas chromatographic system determines N_2, H_2, O_2, CO, CO_2, methane, ethane, ethene, propane, and propene concentrations in the retort offgas at prescribed intervals. The resulting gas composition data along with information available from the LLL oil shale modeling group can be used to compare vapor phase conditions in our retort with conditions in other experimental or protype retorts.

TASK 4. LABORATORY PARTITIONING STUDIES

An inert gas laboratory retort run, LBL-05, was conducted on October 27,

using an all stainless steel retort vessel. The shale grade, particle size distribution (-1/4 inch to +30 mesh), nitrogen input-gas flow rate (2.0l/min), and heating rate (1.0°C/min) were the same as those used for previous runs. The objectives of the retort run were: (1) to determine if the use of a stainless steel retort vessel would result in an increased mass of Hg in the retort offgas and (2) to obtain additional data for the comparison of Au-amalgamation and ZAA analytical methods for measuring Hg in offgas.

During previous retort runs, the maximum temperature differential between the top and the bottom of the vertical shale column has not exceeded 20°C. Retort run, LBL-05, differed significantly in that a large vertical temperature gradient developed early in the run and increased throughout the run. At termination, the shale temperature at the top of the column was 117°C higher than the temperature at the bottom. This unbalanced condition resulted in Hg vaporization conditions and retorting conditions quite dissimilar to those produced during previous runs. Consequently, the results from LBL-05 can not be compared directly to earlier results. Even though the first objective of determining if the use of a stainless steel retort vessel would result in an increase in the mass of offgas Hg could not, therefore, be met, useful information was obtained, and data and sample analyses are in progress.

The large temperature gradient resulted from: (1) improper adjustment of the power distribution between the upper and lower heating elements of the vertical Lindberg furnace for the stainless steel retort vessel; and (2) inadequate insulation at the ends of the vertical furnace. Correction of these problems will be confirmed by heating tests before the next retort run is conducted.

The second objective for LBL-05 of obtaining additional data for the Au-amalgamation and ZAA method comparison was meet. The Hg peak observed for LBL-05 by the ZAA method was highly skewed and lower in magnitude than those observed for previous runs but still useful. Nine Au-amalgamation samples were collected during the LBL-05 Hg peak. The Hg concentrations in offgas determined by the Au-amalgamation technique were compared to the corresponding concentrations determined by ZAA. Seven of the Au-amalgamation concentrations fall within 30% of the ZAA concentrations, and no significant difference was found between the two sets of nine concentrations using a paired Student's t statistical test. The general agreement of the two methods is considered to be good. Additional comparisons are planned for future laboratory retort

runs and field studies.

TASK 5. FIELD STUDIES

The Hg analyses of shale samples from Lawrence Livermore Laboratory's steam air retort run, L-3, were not completed during October. Our batch type ZAA used for these analyses was displaced due to major building modifications. It has now been relocated and a new ventilation system installed so that analyses can resume in November.

PROJECTED WORK

The projected work for November is as follows:

Task 2. Analytical Methods for Gas Samples

The first version of the modified HGA-2000 furnace for Hg and Cd analyses will be constructed and tested.

Task 4. Laboratory Partitioning Studies

Minor modifications are being made to the retort furnace in order to eliminate the vertical temperature gradient encountered during LBL-05. Correction of this problem will be confirmed by heating tests. Another inert gas retort run, LBL-06, will be conducted to determine if the use of a stainless steel retort vessel results in an increased mass of Hg in the offgas.

Task 5. Field Studies

Mercury analyses of shale samples from retort, L-3, will resume.

STANDARD AND MODIFIED VERSIONS OF AN HGA 2000 GRAPHITE FURNACE

Fig. Ia

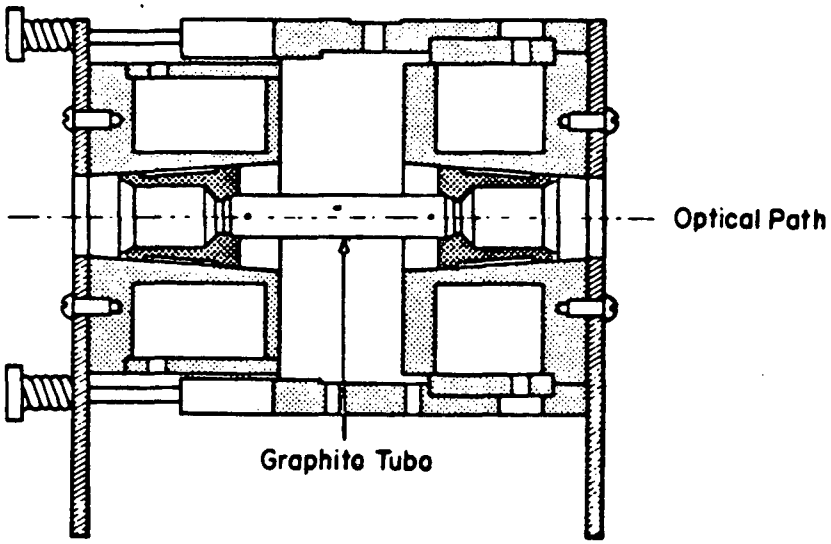
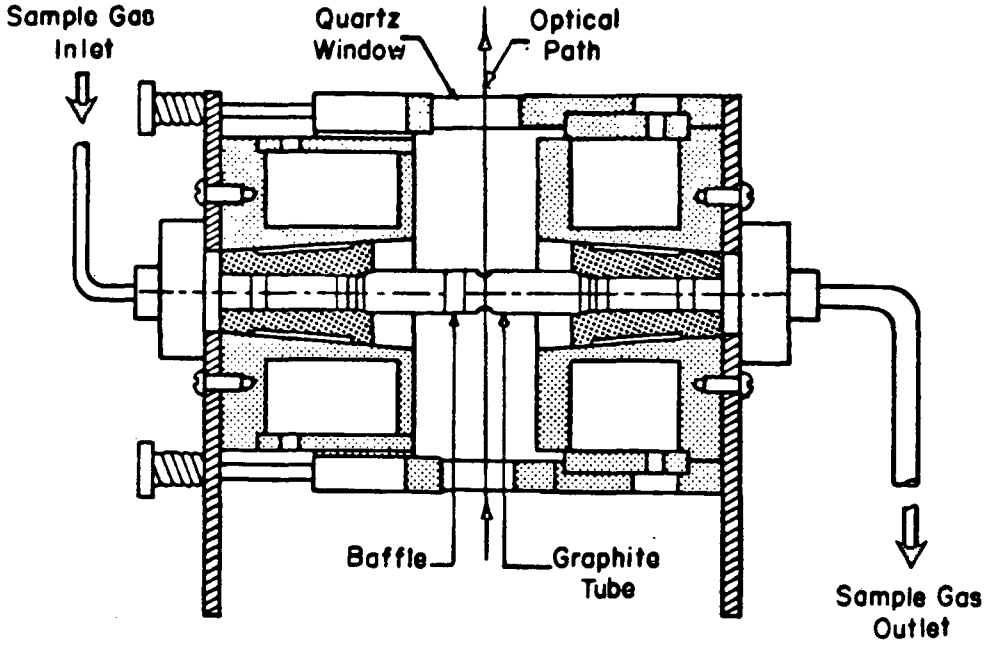


Fig. Ib



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