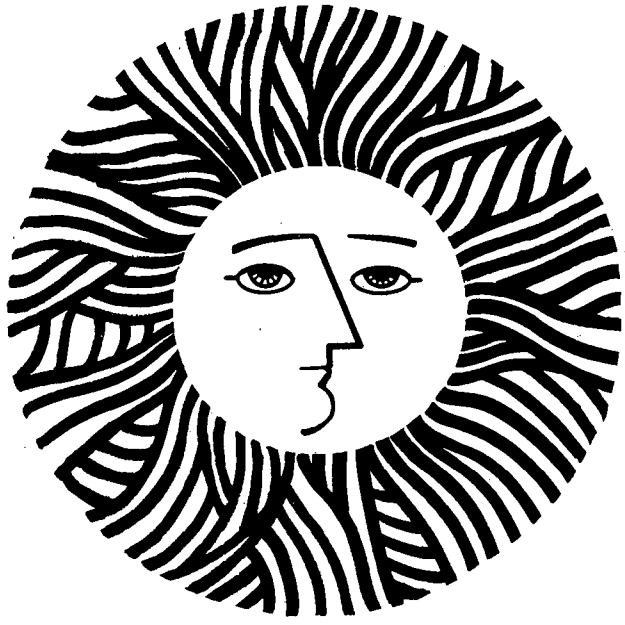
LBID-489
UC 91

Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

ENERGY & ENVIRONMENT DIVISION



DISCLAIMER

9 March 1982

TO: Art Hartstein

FROM: Bonnie M. Jones, Peter Persoff, Richard H. Sakaji, and Jerome F. Thomas
Lawrence Berkeley Laboratory
University of California, Berkeley
Berkeley, California 94720
and
Christian G. Daughton
Sanitary Engineering and Environmental Health Research Laboratory
University of California, Berkeley
Richmond, California 94804

RE: Monthly Progress Report for February 1982

LBID-489

This work was prepared for the Department of Energy
under Contract No. W-7405-ENG-48.

MILESTONES

Enclosed with this month's report is the chemical class fractionation protocol, entitled "Rapid Fractionation of Oil Shale Wastewaters by Reverse-Phase Separation" (LBID-485), by C.G. Daughton, B.M. Jones, and R.H. Sakaji.

TASK 1. ANALYTICAL METHODOLOGY

Nitrate Determination

The determination of nitrate and nitrite in retort waters by the Devarda's Alloy method gave nonreproducible results. Preliminary work on ion chromatography using a Wescan anion exchange column and conductivity detector, however, showed that nitrate additions of 50 ppm NO_3^--N could be easily detected in Oxy-6 retort water. Recovery of the nitrate spike was not affected by the sample matrix. Endogenous nitrate was not found in Oxy-6 water at a level above the detection limit.

Further evaluation of the method was curtailed when an electronic failure in the HPLC eluent pump produced excessive pressures, causing failure of an

injection valve.

Carbon Analysis

The quantitation of organic carbon by low-temperature UV-promoted persulfate oxidation coupled with coulometric titration has decided advantages over systems using high-temperature combustion or infrared detection. This was discussed in the December 1981 Monthly Report.

We have replaced the silicone peristaltic pump tubes with Viton fluoroelastomer pump tubes. This has greatly reduced the level of background interference. The syringe-septum injection system has been replaced with a 200-µL injection loop; this has facilitated ease of operation.

We have begun validation of the system. Accuracy and precision are being determined for various retort waters and standard solutions. Comparison of total dissolved carbon (TDC) values obtained using the two types of carbon analyzer for five retort waters showed agreement within ±4%. The precision for DOC determination on each retort water sample was also high (rsd = 2%). One of the major advantages of UV-persulfate oxidation versus high-temperature combustion is that less diluted samples can be analyzed directly. We have found, however, that a precipitate forms as a result of the acidification and sparging step required for the determination of DOC. This does necessitate a small amount of dilution prior to purging for direct DOC quantitation.

TASK 2. BIOLOGICAL TREATMENT OF PROCESS WATERS

Sample Acquisition

We have initiated sample requests for "sour" water and retort water from surface retorting operations. Several oil shale industry companies and LETC have been contacted.

Enrichment Studies

An extensive set of enrichment studies was begun this month. The wastewater for these studies was spent media from biologically treated Oxy-6 retort water, supplemented with phosphate buffer, 0.81 mM magnesium, and 0.01 mM iron. Inocula were derived from 45 sources, representing various industrial and domestic wastes and several soils. Each inoculum is being maintained in triplicate utilizing spent media as the sole carbon and nitrogen source. The three replicates are passed to fresh media at intervals of one, two, and three weeks. This ensures that species with slow growth rates or longer adaptation times will not be excluded from the selection process. Visual observations of these enrichment cultures are made with the phase

contrast microscope; after several passes the cultures exhibiting the greatest growth will be monitored quantitatively for removal of organic carbon.

TASK 3. PHYSICOCHEMICAL TREATMENT OF PROCESS WATERS

Steam Stripper

The steam drier was replaced with a 6 kW 240 V circulation heater. This unit has been mounted on the steam-stripper frame but has not yet been plumbed in. The thermostat that accompanied the circulation heater did not meet our specifications. Replacement of the thermostat and plumbing should be completed early next month. The bottoms collector and overheads condenser were insulated with 1-inch thick fiberglass in order to improve operation and control of the overall system.

The raw waste feed pump was calibrated by pumping water from a burette into the steam generator via the steam flush line. Two curves were generated, one with the vessel open to the atmosphere, and one with the vessel closed and nearly filled with water so that the pressure increased from 25 to 70 psig during the series of measurements. Linear regression yielded nearly identical calibration curves over the pump's range of 0 to 515 mL/min. The pump will apparently function at a constant rate under a range of pressure heads.

Redesign of the packed-bed glass stripping column has been proposed. This modified column will allow sampling of the countercurrent flows at different column heights. Each sampling port has five inlets that are located so that samples from equal cross-sectional areas of the column can be withdrawn. This modification should allow us to collect representative samples during operation.

TASK 5. RETORT ABANDONMENT FINAL REPORT

Numerical modeling for scenario studies

The model TRUST can now accurately simulate flow in anisotropic media; the "bug" that was reported last month has been located and corrected. Reinvasion calculations are being executed under various conditions. The requirement that very high permeability nodes (abandoned retorts) be located immediately adjacent to the very low permeability nodes (undisturbed Mahogany Zone) has caused some model instability; we are now attempting to resolve this by changing the way in which constant potential boundary conditions are modeled.

Report Preparation

Preparation of the final reports on grout development and on alternative strategies for retort abandonment is continuing. Sections detailing the various experiments that were done to enhance the setting properties of spent shale, and detailing the results of subsidence calculations were drafted.

ADMINISTRATION

First drafts of the Energy and Environment Division annual reports were completed and sent to the division office for editing and word processing. These reports will be reviewed prior to the production of galley proofs.

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.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

TECHNICAL INFORMATION DEPARTMENT
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720