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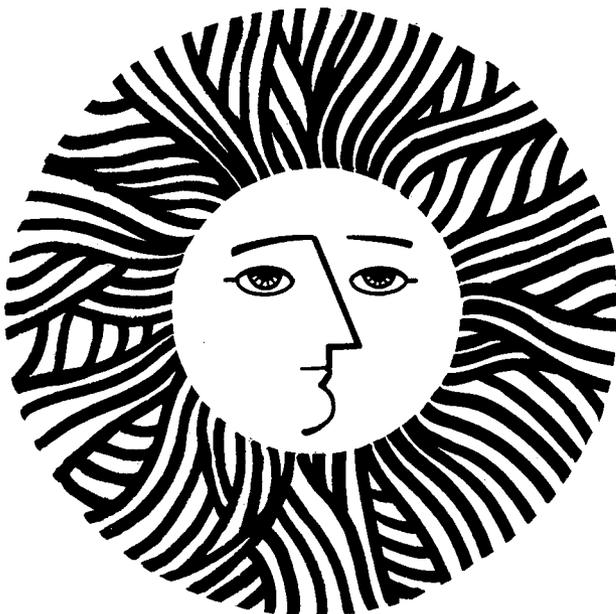
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October 5, 1981

TO: Charles Grua

FROM: Richard H. Sakaji, Bonnie M. Jones, and Jerome F. Thomas;
Christian G. Daughton (SEEHRL)

Lawrence Berkeley Laboratory, University of California, Berkeley, CA 94720

RE: Monthly Progress Report for September
Spent Shale as a Control Technology for Oil Shale Retort Waters
LBID-444

Visitors from DOE and the Oil Shale Task Force (A. Hartstein, R. Poulson, D. Sheesley, W. Chappell, and K. Petersen) toured our facility at the Richmond Field Station on September 14, 1981. They were introduced to Professor Thomas, and we presented a brief overview of our approach to oil shale wastewater research and a review of our results. Copies of drafts of the slides that we prepared for this review are enclosed.

TASK 1. ANALYTICAL METHODS DEVELOPMENT

Oil and Grease Determination

An additional refinement in our procedure for oil-and-grease determination minimized the background contribution from the C-18 Sep Paks. By applying a larger volume of sample (10 mL) to each prewashed, activated cartridge and diluting the organic eluate, the background absorbance was reduced to 0.002 compared with sample absorbances of 0.086 to 0.102 for Oxy-6 retort water. This technique eliminates the background problem for oil-and-grease determinations, but cannot be applied to our oil determination because the samples are not diluted prior to quantitation. We will investigate the background absorbances of new lots of Sep Paks and reverse-phase cartridges from other manufacturers.

If the organic material retained by a reverse-phase cartridge can be quantitated by means other than IR spectroscopy, our proposed method for oil-and-grease determination would be simpler and more versatile than existing methods which must rely on either gravimetric or spectrophotometric analyses. To investigate this possibility, we determined the COD and the DOC of the polar fraction, calculated the respective values of the retained fraction by difference, and compared these results to the oil-and-grease concentrations

determined by IR absorbance of the Freon eluate. The results are summarized in Table I. The reproducibilities of the alternative methods of analysis are better than that of IR quantitation. These methods, however, measure slightly different fractions of the raw wastewater. The IR spectrophotometric method measures only the components of the retained fraction that can be eluted by Freon, whereas the DOC and COD results reflect the contribution from the entire retained fraction (i.e., the eluted and the irreversibly retained compounds). This difference makes interpretation of values obtained by the alternative methods of analysis more difficult.

TASK 4. SPENT AND RAW SHALE STUDIES

Batch Isotherm Studies

All previous batch experiments for determining the time required for a sorbent-solute system to reach equilibrium were conducted at ambient temperature. Two batch experiments were completed under isothermal conditions, using TOSCO II spent shale and Hydrodarco activated carbon (HDC) as sorbents. These demonstrated that the time required for the sorbent-solute system to reach equilibrium may be shorter than previously determined but that the equilibrium loading (i.e., mass of solute sorbed per mass of sorbent) for spent shale was identical to previous experiments. Since the brand of activated carbon used this month was different from that used in our previous experiments, we were not able to demonstrate that the equilibrium loading for the activated carbon was unaffected by temperature.

TASK 5. SYSTEM STUDIES

Biological Oxidation Studies

A preliminary investigation was conducted to determine if UV radiation can alter the biodegradability of the organic solutes in retort water. One liter of Oxy-6 retort water was irradiated for 2 hours by a 450W medium pressure mercury-vapor quartz lamp. Samples of irradiated retort water were withdrawn at time intervals and inoculated with bacteria acclimated for growth on raw retort water. This intense irradiation did not alter the DOC concentration of the retort water. When compared with cultures grown on non-irradiated retort water, biological degradation of the dissolved organic solutes was neither enhanced nor hindered.

To further investigate the effects of photooxidation, we added 3 mg/L of riboflavin, a photosensitizer, to samples of Oxy-6 retort water. Photosensitizers absorb radiant energy and in the process of returning to ground state, transfer the excess energy to acceptor compounds that cannot absorb radiant energy of these excitant wavelengths. The acceptor molecule can dissipate this energy by several different pathways including dissociation. The addition of riboflavin, however, did not effect significant changes in either the DOC concentration or the biodegradability of the organic solutes.

Table I
Oxy-6 Retort Water Oil-and-Grease Values

<u>Method of Determination</u>	<u>Concentration (mg/L)</u>	<u>rsd % (n = 8)</u>	<u>Ratio (compared with IR)</u>
IR	262 (as Mineral Oil)	5.8	not applicable
COD	4210 (as O ₂)	3.0	16.1
DOC	1253 (as Carbon)	1.6	4.8

October 2, 1981

TO: Charles Grua

FROM: Richard H. Sakaji, Bonnie M. Jones, and Jerome F. Thomas;
Frank Pearson and Christian G. Daughton (SEEHRL)

RE: Monthly Progress Report for September
Steam Stripping Project
LBID-445

ANALYTICAL METHODS DEVELOPMENT

Ammonia Determination

Weatherburn's version of the phenate method for ammonia-nitrogen determination yielded a linear standard curve ($r^2 = 0.998$) for the 10- to 100-mg/L range. For accurate quantitation at 520 nm, this represents the lower range for this determination because of noise limitations, primarily imposed by the absorbance of the reagent blank.

An ammonia-sensing electrode (HNU Systems) with a bonded membrane was evaluated for use in oil shale process waters. A facile method for approximating the range of the ammonia concentrations in steam stripper samples would be helpful in gauging the dilution required for the phenate method. The probe showed adequate stability in and good logarithmic response to ammonium sulfate standards of 1, 10, 100, and 1000 ppm ammonia nitrogen; several problems, however, were noted. Upward drift, which did not stabilize in a reasonable period of time, was observed while the probe was immersed in a sample. Either upward or downward drift occurred between sample readings. The standard curve drifted and changed slope at a different rate than did the sample readings, causing very poor reproducibility despite recalibration of the instrument. The observed drift could not be accounted for by either temperature changes or ammonia volatilization. Measurement of ammonia nitrogen in Oxy-6 gas condensate and retort water yielded values of 7400 (n=2) and 1336 (n=2) respectively; these values were approximately 12 and 16 per cent higher than those determined by distillation/acidimetric titration and by the phenate method. We concluded that the performance of this ammonia electrode was not superior to the previously evaluated Beckman electrode, and that results from ammonia electrodes for oil shale wastewaters should be interpreted only as indicative of a possible concentration range.

STEAM STRIPPER DESIGN

Fabrication

The following units were installed: a safety climber; steam generator and condenser cooling water pumps; expansion joints and one glass column with Teflon o-rings; steam lines, including Venturi flowmeter with manometer, and discharge nozzle; steam generator water level regulator, bottoms receiver, overheads receiver, and overheads condenser sight glass; manometer for column pressure drop; wastewater feed pump; pressure switch. The temperature controllers and heaters were partly wired.

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