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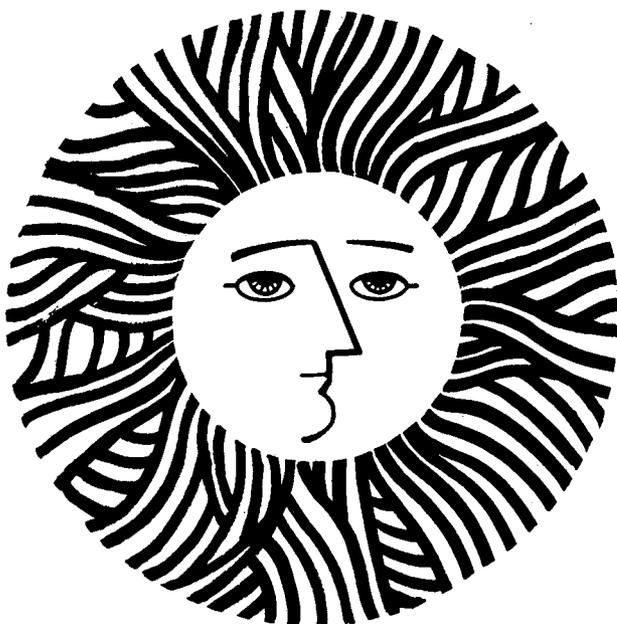
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5 January 1982

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RE: Monthly Progress Report for December

LBID-460

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MILESTONES

Enclosed with this month's report are the validated colorimetric and distillation/titration ammonia protocols, entitled "Quantitation of Ammonia in Oil Shale Process Wastewaters" (LBID-465), by J. Cantor, B.M. Jones, R.H. Sakaji, and C.G. Daughton.

TASK 1. ANALYTICAL METHODOLOGY

Organic Carbon Determination

Experiments to determine the feasibility of quantitating DOC by using low-temperature UV-promoted persulfate oxidation coupled with coulometric titration were initiated this month. Persulfate oxidation promoted by UV irradiation has several possible advantages over oxidation by high temperature combustion. These include: (i) elimination of quartz combustion tubes, which must be frequently replaced because they are attacked by alkali metals such as Na, (ii) elimination of combustion-tube packing materials, which are easily fouled by retort water organic materials; this problem necessitates the dilution of samples prior to analysis, causing errors and increasing sample preparation time, (iii) instrument down-time and operating expenses would be

greatly reduced by eliminating the need for conditioning new combustion tubes and the need for silver halide-scrubbers, (iv) sample injection loops can be used, thereby increasing precision and accuracy, (v) the ability to inject concentrated samples takes advantage of the large titration capacity of the coulometer, and (vi) long warm-up times for furnaces are eliminated.

The unit we have fabricated uses a coulometer to capture and titrate the carbon dioxide that is evolved during the oxidation process. The advantages of coulometric titration versus non-dispersive infrared detection include: (i) standards are required only to check the operation of the coulometer; standard curves are not necessary, (ii) problems with fouling of IR cell reflective surfaces are eliminated, (iii) interferences are fewer, and (iv) the linear dynamic range of the coulometer is orders of magnitude greater; dilutions are unnecessary. The combination of the low-temperature combustion unit with the coulometer is possibly a great improvement over the commercial units, all of which employ either high-temperature combustion with coulometric detection or low-temperature oxidation coupled with infrared detection. Initial start-up has involved assembly of the apparatus and checking for background interferences. An initially high background was traced to the silicone peristaltic pump tubing. We are currently attempting to eliminate or decrease this background to an acceptable level.

Ammonia Determination

Additional work was done to verify the suitability of Weatherburn's version of the phenate method. Fortification studies yielded recoveries of 93 to 110% of the theoretical values of ammonia added to raw and "spent" Oxy-6 retort water. The response curve in the 10- to 100-ppm range was determined to be linear ($r = 0.999$). Interfering substances caused by ambient light as reported by another laboratory were shown not to be a significant factor in our analyses.

Work on the colorimetric ammonia method has been completed, as discussed under MILESTONES.

TASK 2. BIOLOGICAL TREATMENT OF PROCESS WATERS

In last month's report, we presented preliminary data in support of our hypothesis that a large portion of the biorefractory organic solutes in Oxy-6 retort water comprises nitrogenous heterocycles. The catabolism of these compounds in retort water is probably limited by one or several factors, which include the following considerations. (i) Although the overall concentration of organic nitrogen is high, this nitrogen may be derived from

many different nitrogenous heterocycles; (ii) each heterocycle may be present at a low concentration; these concentrations may be below the affinity constants of the requisite enzymes; (iii) the cells may not be capable of synthesizing the requisite enzymes; (iv) requisite enzymes may not be constitutive and their induction may require the absence of inorganic nitrogen sources such as ammonia; and (v) the enzyme complements for heterocycle degradation are often highly specific.

The biodegradation of this chemical class in retort waters will probably succeed only by the use of multiple stages (to segregate the organic-nitrogen degrading bacteria from those that degrade the easily metabolized fraction), and by the use of complex mixtures of species, each of which is capable of degrading a different heterocycle or homologous series. We have begun extensive enrichment cultures, using various sources of inocula, with spent Oxy-6 and other surface and in-situ retort waters as sole sources of carbon, nitrogen, or carbon and nitrogen.

TASK 3. PHYSICOCHEMICAL TREATMENT OF PROCESS WATERS

Steam Stripping Studies

The problem with the digital temperature readout probes (RTD's) has been rectified. Calibration instructions from the manufacturer were received at the beginning of the month. The probes were all found to be within specifications. The wiring from the control panel to the RTD probes was checked and no problems were found. The probe readouts were checked against each other and no problem could be detected. When the wiring was reassembled all the probe readouts varied within two degrees of each other. In addition, when the steam generator was started, the probe in the region of the flash evaporator gave a proper temperature response.

Miscellaneous

Lawrence Berkeley Laboratory was closed from 17 December 1981 to 4 January 1982. Only those functions necessary to the maintenance of experimental work were performed.

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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