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ANALYSIS OF ANCIENT SEDIMENTS AND  
EXTRA-TERRESTRIAL MATERIALS

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Introduction

The studies initiated in June 1966 were designed to isolate and identify organic components of meteorites, recent and ancient sediments. The basic intention, in addition to the gathering of important scientific information, was to gain additional experience that could be applicable to the lunar sample recovery program.

It was decided to concentrate on low molecular-weight organic compounds that are normally volatile. In this manner it may be possible, first, to determine what organic gases were sampled from the solar nebula during formation of planetary objects and, second, to differentiate between biogenic and nonbiogenic components.

In the first half of the budgetary year experiments were planned, an extraction line was built and calibrated, and a gas chromatography unit was installed. Enclosed is a description of the procedure that has been employed.

A second approach is the study of the distribution of naturally occurring stable isotopes of light elements. In collaboration with Dr. Melvin Silverman, an investigation is being undertaken on the fractionation of  $C^{13}/C^{12}$  in methane and carbon dioxide in the products of metabolism of methane oxidizing and methane forming bacteria. A dual-collecting high-precision mass spectrometer has been

calibrated against known standards and is now performing satisfactorily. It is capable of measuring micromole quantities of carbon dioxide.

In connection with isotope measurements, a vacuum line has been established for the quantitative reduction of sulfate with graphite to carbon dioxide. The  $^{18}\text{O}/^{16}\text{O}$  variation will be measured in terrestrial and meteorite sulfate to determine if differences exist. A description of the technique is enclosed.

1. Low-boiling compounds.

Our efforts during the period from June through November 1966 have been directed toward the setting up of preparative and analytical instruments for the isolation and identification of the low-boiling organic compounds from soils, sediments, and meteorites. A gas collection-transfer manifold has been constructed from glass and metal components to eliminate the contaminative effects of greased stopcocks and polymer tubing. Sample crushing is carried out in an evacuated high-purity, impermeable alumina capsule and is connected to the manifold via a flexible metal tubing. Gas transfer is accomplished by a mercury-powered Toepler pump which compresses the volatile material into a small volume collection tube that can be sealed in glass for further analysis. We are currently testing the use of cryogenic trapping of the organic gases by the use of silica gel at liquid nitrogen temperatures. This can be done quantitatively for all hydrocarbon gases including methane, but the problem of recovery of the aromatic gases from the silica gel may preclude the use of this technique.

Coincidentally, we have purchased and set up a versatile gas-chromatographic instrument for the separation and identification of the volatile compounds. Since gas-chromatography may be the sole means of identification due to the low

amount of material present in the samples, we have decided on a high-resolution capillary system so that there will be no ambiguities in establishing identity of the compounds. As a further aid in identifying, we are simultaneously using a second means of detection - an electron-capture detector which is sensitive to the electrophilic character of volatile compounds. The effluent gases from the capillary column is split in two so that half goes to each detector. The relative response of each compound can then be used as a means of identification.

We are currently involved in working out a reproducible method of temperature-programming from sub-ambient temperatures and in calibrating the apparatus.

2.  $^{18}\text{O}/^{16}\text{O}$  ratios in sulfates.

A method has been developed to quantitatively reduce barium sulfate to barium sulfide and carbon dioxide by reaction with graphite. Since one of the products of the reaction is carbon monoxide, it is converted to carbon dioxide by disproportionation in a corona discharge. The following technique has been adopted. Approximately 20 mg of barium sulfate and 20 mg spectrographic grade graphite are weighed on a piece of glazed paper. The mixture is then ground in a small steel mortar and pestle and transferred completely to a platinum combustion crucible. The crucible fits inside a molybdenum cylinder to prevent tipping over and is then placed inside the furnace. The system is degassed at  $600^{\circ}\text{C}$  for 10 minutes by vacuum pumping. The pressure should be less than one micron after 10 minutes.

Before reacting the sample, the U trap is immersed in liquid nitrogen to collect carbon dioxide from the reaction and a spot of liquid nitrogen is placed on the tip of the corona vessel to collect  $\text{CO}_2$  from the disproportion reaction.

Valve 1 is shut and valve 2 and valve 3 are open. The pumps are shut off and the furnace is heated from 950°-1000°C. When the pressure begins to rise, valve 2 is adjusted so that the pressure of CO in the corona is kept under 100 microns and the corona is turned on. The pressure in the corona chamber remains high as long as the reaction is continuing, but as the CO is converted to CO<sub>2</sub> and frozen in the bottom of the trap, the pressure begins to fall after about 10 minutes when the reaction is over. The furnace and high voltage are then shut off. The liquid nitrogen on the U trap is exchanged for dry ice - acetone to catch any water and to allow the CO<sub>2</sub> collected from the reaction to distill over into the corona vessel. If there is any CO present the corona is activated to bring the pressure down. Valve 2 is shut, nitrogen is placed on the cold finger and the CO<sub>2</sub> is allowed to freeze over and be measured and collected.

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