ANALYSIS OF THERMAL AND ACID RELEASED ORGANOGENIC COMPOUNDS IN APOLLO 16 LUNAR SAMPLES, D.A.Flory, J. Oró, S.A. Wikstrom, D.A. Beaman, and A. Lovett, Department of Biophysical Sciences, University of Houston, Houston, Texas 77004.

The identity and concentrations of the various volatile organogenic compounds released by stepwise heating and deuterium labeled acidolysis of Apollo 16 lunar samples have been determined by gas chromatography-mass spectrometry.

We have received six samples of fines and four rock samples as follows: 1.5 g of 63321 fines collected at Station 13, 3.0 g of 64421 fines taken from a trench bottom at Station 4, 3.0 g of 64501 fines from the rake soil collected at Station 4, 5.0 g of 65901 fines taken 5-15 cm below the surface at Station 5, 2.0 g of 68121 fines and 3.0 g 68501 fines (a rake soil) collected at Station 8, 8.4 g of Type IV rock 60017 (a medium to dark gray vesiculated breccia) including interior and exterior chips collected at the LM-ALSEP sampling site, 4.0 g of Type II rock 60025 (a white cataclastic anorthosite) collected at the LM-ALSEP sampling site, 8.0 g of Type III rock 60315 (a medium light gray hornfelsed diabase or basalt porphyry) consisting of an interior and exterior chip and 9.7 g of Type I rock 67016 (a light gray breccia) consisting of an interior and exterior chip. All soil samples are less than 1 mm sieve fractions.

The experimental equipment and techniques have been modified extensively since our work reported at the Third Lunar Science Conference (1). Most of these changes were described in our Apollo 15 paper (2) but additional modifications have been made since the Apollo 15 samples were analyzed to allow better separation of N_2 and CO and improved quantitation of all volatiles released. In our previous analyses the volatiles from the pyrolysis oven and acidolysis reactor were swept out by helium for 3-4 minutes and trapped on the front end of a cold(-30°C) Carbosieve B column. This method did not allow good quantitative collection of the evolved gases and resulted in poor chromatographic separation because of the long injection time. The evolved gases are now collected in a liquid nitrogen-cooled adsorbent trap (Tenax-GC:Applied Sciences Laboratories, State College, PA) and then injected by rapid thermal (300°C) desorption onto the cold column.

Blanks were run on the system prior to the analyses to insure against interference from artifacts for both the pyrolyses

ORGANOGENIC COMPOUNDS

Flory, D.A. et al.

and acidolyses. Calibration of the GC-MS for quantitation was accomplished by injecting a calibration mixture containing CO, CH_4, C_2H_4, C_2H_6, C_3H_8 and N_2. The CO calibration was used for H_2O, CO_2 and Ar. Some fines samples were sieved by passing them through organically cleaned brass sieves with copper screens. Multiple ion plots were constructed from the spectra taken during analysis of the acidolysis volatiles to aid in identification of partially deuterated species.

The volatiles released by heating were trapped separately and analyzed at temperatures of 200°C, 500°C, 800°C and 1100+°C. Ar, N_2, CO , CH_4, CO_2, H_2O, C_2H_4, C_2H_6, C_3H_6 and C_3H_8 were released by the thermal treatment in quantities totalling several hundred ppm for N_2, CO, CO_2 and H_2O and several ppm, or less, for the hydrocarbons in the fines samples. Generally the quantities of CO_2 released are greater than CO at temperatures below 500°C. Above 500°C a reversal is observed, the CO being higher than CO_2. There are samples, however, where the CO_2 concentration remains highest to temperatures greater than 1100°C. The temperature release behavior of the individual gases was similar to that observed in lunar samples from previous missions. (1,2). The hydrocarbons are seen predominantly in the 500-800°C temperature step. Substantial proportions of N_2 and water are released at temperatures above 500°C in most samples, indicating the presence of chemically bound or physically trapped (in vesicles) N_2 and water.

Acidolysis with DCl also released Ar, N_2, CO, CH_4, CO_2 and all the possible completely deuterated, partially deuterated and completely hydrogenated species of methane, ethane, ethylene, propane and propylene. Water was probably released also but cannot be distinguished because of the traces of water in the DCl solution. Some samples exhibited deuterated to non-deuterated methane ratios of 1.7 which is lower than observed previously (1,2).

The quantity of the trench sample 65901 received was sufficient to allow separation into size fractions by sieving. Size fractions obtained were 420μ-1000μ, 250μ-420μ, 125μ- 250μ and less than 125μ. Pyrolysis of these separate fractions indicate correlation of volatiles concentration with particle size but there does not appear to be a continuous increase in concentration of the gases evolved with decreasing particle size. The 420μ-1000μ size fraction indicated an unusually large content of C_3 hydrocarbons (>100 times the usual). This result was obtained on a repeat run with a much smaller amount of the same size fraction, but more analyses of this fraction will have to be carried out before we can appraise the full significance

of this result.

Complete data will be presented for pyrolysis and acidolysis of all samples described above including the five samples received just prior to compilation of this abstract.

REFERENCES

(1) Flory, D.A., Wikstrom, S., Gupta, S., Gibert, J.M., and Oro´ J., Analysis of Organogenic Compounds in Apollo 11, 12 and 14 Lunar Samples, Proceedings of the Third Lunar Science Conference, Geochim. Cosmochim. Acta. Suppl. 3, Vol. 2, pp. 2091-2108, MIT Press.

(2) Flory, D.A., Oro´,J., Wikstrom, S., and Beaman, D., Analysis of Organic Compounds in Apollo 15 Samples, J. W. Chamberlain and C. Watkins, eds. pp. 275-278, LSI.