NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LUNAR SAMPLE ANALYSES PROGRAM

PROPOSAL TITLE:

Search for C_{15} to C_{30} Alkanes in Lunar Soils

by

W. G. Meinschein

22 February 1971

Principal Investigator: W. G. Meinschein

Co-Investigators: Eugene Cordes
V. J. Shiner, Jr.

Final Report

Prepared under Contract No. NAS 9-9974

by

Indiana University
Bloomington, Indiana 47401

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Manned Spacecraft Center
Lunar Receiving Laboratory
Houston, Texas

CR-114919
c.1

CONTENTS

	Page
Abstract	3
Introduction	4
Discussion & Summary	6

ABSTRACT

Benzene-methanol extracts of lunar fine samples #10086 (50g), #12001,31 (11g), #12001,32 (9g), and #12033,6 (5g) were separately obtained on the intact, pulverized, and hydrofluoric acid digested samples. These extracts did not contain detectable quantities of C_{15} to C_{30} alkanes. No C_{15} to C_{30} alkane was present in these extracts at concentrations exceeding 1 part per billion by weight.

Introduction

Alkanes are saturated organic compounds that are composed solely of carbon and hydrogen atoms. These hydrocarbons are ubiquitous but minor constituents of the waxes, fats, and oils of plants and animals (1). Because alkanes are less readily assimilated than other food substances and are chemically less reactive than most biological compounds, alkanes are preferentially preserved relative to other organic materials in sedimentary environments. Natural gas and petroleum deposits commonly appear to be concentrates of alkanes partially derived from preexistent organisms (2). Many alkanes from petroleum have molecular structures that are either identical or similar to alkanes and related compounds in plants, animals, soils, marine sediments, and sedimentary rocks (3). The marked structural and distributional resemblances between certain alkanes in rocks and compounds in organisms have led to the widespread use of alkanes as molecular or chemical fossils (4) and to the recommendation that C_{15} to C_{30} alkanes be employed in exobiological research (5). This report deals with a search for C_{15} to C_{30} alkanes in Apollo 11 and Apollo 12 lunar samples.

Experimental

Repeated tests established that all solvents, reagents, and apparatuses were free of detectable amounts of organic contaminants with vapor pressures equivalent to C_{15} to C_{30} alkanes. The gas-liquid chromatograph at the sensitivity settings used to monitor contamination gave 5 to 12 per cent of full scale deflection with 10^{-7} g of pristane (2, 6, 10, 14 - tetramethylpentadecane) for sample #10086 and 10 per cent of full scale deflection with 10^{-8} g of pristane for samples #12001,31, #12001,32, and #12033,6. Sensitivity

checks were run on standard solutions of pristane before and after each gas-liquid chromatogram of a lunar extract sample.

Each sample of lunar fines were initially extracted with an azeotropic mixture of benzene and methanol in a specially designed combination Soxhlet extractor and ball mill (6). Throughout these extractions, a positive pressure of nitrogen was maintained in the system. Nitrogen flowed through a drying tower filled with silica gel into the extractor, and the nitrogen stream was exhausted through a bubbler tube with a 1 cm head of mercury.

The analyses of the Apollo 11 and Apollo 12 fines were carried out in the same manner, but the extraction period was reduced for the Apollo 12 fines because the negative results on the Apollo 11 sample indicated that prolonged extractions were unnecessary.

Each extraction was accomplished similarly. After approximately 500 ml of solvent per hour had been refluxed through the lunar fines for a period of 24-170 hours, a 200 ml sample of solvent was collected as it flowed from the extractor. The volume of this sample was reduced to several microliters by removal of solvent in a stream of filtered nitrogen at 40°C, and all of the residual sample that could be taken into a 10-μl syringe was injected into a Apiezon L coated gas-liquid chromatographic column. No organic material except solvent was detected in this sample. The extractor was then disconnected from the reflux condenser and distillation flask, sealed with two glass stoppers, and rolled for 12-48 hours on a ball mill. All the extraction solvent was transferred from the distillation flask, and the volume of the extract was reduced by removal of solvent in a stream of filtered nitrogen at 40°C to approximately 100 μl. After a 2 μl portion of the remaining extract failed to yield detectable C_{15} to C_{30} alkane peaks in

the gas chromatograph, the volume of the extract was further reduced to a few microliters, which were injected into the gas chromatograph. Only solvent peaks were observed in the chromatograms of this extract of the different samples of lunar fines.

The extractor, containing each ballmilled lunar sample, was reconnected to the reflux condenser and distillation flask, and azeotropic benzene and menthanol were refluxed through the pulverized sample at a rate of approximately 500 ml per hour for 12-72 hours. Chromatograms of the individual extracts of each of the pulverized lunar sample, also, indicated an absence of organic compounds with vapor pressures equivalent to C_{15} to C_{30} alkanes.

Each pulverized lunar sample was separately transferred from the extractor to a 2-liter Teflon beaker with benzene, and 500g of 48 per cent hydrofluoric acid was slowly added with continuous stirring until reactions ceased. The beaker was warmed to 80°C with stirring until most of the benzene layer evaporated, then the reaction mixture was transferred to a 2-liter separatory funnel containing 400 ml of distilled water. This mixture was shaken thoroughly with three successive 200-ml portions of benzene. These benzene extracts were composed and concentrated. These extracts did not contain detectable quantities of volatile organic materials.

Discussion and Summary

The lunar fines analyzed contained no C_{15} to C_{30} alkane at a concentration exceeding 1 ppb by weight, whereas the concentrations of C_{15} to C_{30} alkanes in rocks from the surface of the earth commonly exceed 100 ppm by weight. These results provide no evidence that life has ever existed on the moon. Scientific publications have reported these results (7). All lunar samples analyzed by our group have been returned to the Curator of lunar samples at the Manned Spacecraft Center.

W. G. Meinschein
Department of Geology,
Indiana University, Bloomington

Eugene Cordes, V. J. Shiner, Jr.
Department of Chemistry,
Indiana University

References and Notes

1. H. W. Gerarde and D. F. Gerarde, Ass. Food Drug Officials U. S. Quart. Bull. 26, 65 (1962); J. D. Mold, R. E. Means, F. K. Stevens, H. M. Ruth, Biochemistry 3, 1293 (1964); G. Eglinton and R. J. Hamilton, Science 156, 1322 (1967).

2. E. G. Baker, Science 129, 871 (1959); W. G. Meinschein, Bull. Amer. Ass. Petrol. Geol. 43, 925 (1959); _____, Y. M. Sternberg, R. W. Klusman, Nature 220, 1185 (1968).

3. W. G. Meinschein, Bull. Amer. Ass. Petrol. Geol. 43, 925 (1959); Geochim. Cosmochim. Acta 22, 58 (1961); J. G. Bendoraitis, B. L. Brown, L. S. Hepner, Anal. Chem. 34, 29 (1962); R. A. Dean and E. V. Whitehead, Tetrahedron Lett. No. 21, 768 (1961); A. L. Burlingame, P. Haug, T. Belsky, M. Calvin, Proc. Nat. Acad. Sci. U. S. 54, 1406 (1965); I. R. Hills and E. V. Whiteheat, Nature 209, 977 (1967); _____, D. E. Anders, W. E. Robinson, Chem. Commun. 1966, 752 (1966); R. B. Johns, T. Belsky. E. D. McCarty, A. L. Burlingame, P. Haug, H. K. Schnoes, W. Richter, M. Calvin, Geochim. Cosmochim. Acta 30, 1191 (1966).

4. W. G. Meinschein, Bull. Amer. Ass. Petrol. Geol. 43, 925 (1959); B. Nagy, W. G. Meinschein, D. J. Hennessy, Ann. N. Y. Acad. Sci. 93, 25 (1961); W. G. Meinschein, B. Nagy, D. J. Hennessy, ibid, 108, 533 (1963); W. G. Meinschein, Nature 197, 833 (1963); _____, E. S. Barghoorn, J. W. Schopf, Science 145, 262 (1964); G. Eglinton, P. M. Scott,

T. Belsky, A. L. Burlingame, M. Calvin, ibid., p. 263; E. S. Barghoorn, W. G. Meinschein, J. W. Schopf, ibid, 148, 461 (1965); J. Oro, D. W. Nooner, A. Zlatkis, S. A. Wikstrom, E. S. Barghoorn, ibid., p. 77; P. E. Cloud, Jr., J. W. Gruner, H. Hagen, ibid., p. 1718; T. Belsky, R. B. Johns, E. D. McCarty, A. L. Burlingame, W. Richter, M. Calvin, Nature 206, 446 (1965); G. Eglinton and M. Calvin, Sci. Amer. 216, 32 (1967); J. Oro and D. W. Nooner, Nature 213, 1082 (1967).

5. W. G. Meinschein, "Preliminary proposal for government contract research on development of hydrocarbon analyses as a means of detecting life in space. Esso Research and Engineering Company," submitted to NASA, 31 January 1962.

6. W. G. Meinschein, C. Frondel, P. Laur, K. Mislow, Science 154, 377 (1966).

7. W. G. Meinschein, Eugene Cordes, and V. J. Shiner, Jr., Science 167, 753 (1970); W. G. Meinschein, T. J. Jackson, J. M. Mitchell, E. Cordes, and V. J. Shiner, Jr., Proc. Apollo 11 Lunar Sci. Conf., Geochim. Cosmochim. Acta Suppl. 1, Vol. 2, pp. 1875-77. Pergamon, London (1970); J. M. Mitchell, T. J. Jackson, R. P. Newlin, W. G. Meinschein, E. Cordes and V. J. Shiner, Jr. Proc. Apollo 12 Lunar Sci. Conf. (in press).

8. Sponsored by NASA contract NAS 9-9974. Development of laboratory for biogeochemical analyses supported by NSF grant GB-6583. We thank J. M. Mitchell, T. J. Jackson, R. P. Newlin, and S. M. Scherer for technical assistance.