Space Sciences Laboratory
University of California
Berkeley, California 94720

Semi-Annual Report on

ANALYTICAL TECHNIQUES FOR IDENTIFICATION AND STUDY OF ORGANIC MATTER IN RETURNED LUNAR SAMPLES

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Principal Investigator: Professor A. L. Burlingame

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The initial tenure (ten months) of NGR 05-003-134 has been both hectically active and fruitful and will be discussed in this report as Phase I of the proposed program. The report will be divided into six sections: I. introductory statement on general subjects and objectives of the project; II. facilities upgrading; III. SDS 930 on-line system; IV. publications; V. personnel; and VI. outline of Phase II.

I. INTRODUCTION

Recently, high resolution mass spectrometry has provided a very few laboratories with the capability for the routine determination of molecular structures by computer-aided interpretation of high resolution mass spectral fragmentation patterns. Such a mass spectral approach to the determination of the specific structural architecture of matter on a molecular level has several physico-chemical advantages: (1) minute sample size — microgram and submicrogram amounts; (2) impurities from chemical isolation and separation procedures do not generally interfere; (3) sample does not have to be crystalline; (4) technique not limited to specific classes or types of molecules or to any particular atomic composition; and (5) precise atomic constitution of the molecule and each fragment thereof is obtained on such minute amounts of sample ($\sim$ microgram).

In this Laboratory major effort is being devoted to the development of a closed-loop computer-controlled high resolution mass spectrometry facility. The dual role of a real-time on-line high speed digital computer system for the simultaneous functions — instrument regulation and high quality data acquisition — offers the potential analytical capability for an unprecedented large-scale systematic survey and characterization of organic matter of terrestrial and lunar origin.

Recently, we have completed the first stage in "computerized" high resolution mass spectral data acquisition, processing, and interpretation, which will lead to the closed-loop computer control of a high resolution mass spectrometry and capillary gas-liquid chromatography facility to be used for the complete (structural and isotopic) characterization of organic matter in lunar samples returned by NASA's Apollo mission.

The sophisticated facility made possible by such development of automated instrumentation will bring the most powerful analytical technique of this decade to bear on problems encountered in the study of the organic constitution of terrestrial, meteoritic, and lunar materials.

It is expected that the knowledge gained through our studies and equipment automation will continue to provide the fundamental body of fact and experience that will prove indispensable both in assessing the fate of ancient terrestrial life and in laying the foundation upon which the later evaluation of the analyses of lunar samples will rest.

A further consequence of this research and development will be to lay the groundwork of experience that the Berkeley Group needs in order to develop a realistic conceptual and detailed design of an automated lander with either a Martian or Venusian mission in the 1970's or 1980's. The research experience gained in this earth-based computer-controlled laboratory will be invaluable for extension to remote characterization of the molecular composition on planetary surfaces, which should allow assessment of the existence of extraterrestrial life.

Completion of Stage I in the "computerization" of high resolution mass spectral data acquisition and processing has provided the initial necessary design criteria for the proposal and negotiation (jointly with Massachusetts Institute of Technology) of a contract with the Manned Spacecraft Center, Houston, for design, fabrication, and testing of a "computerized" low resolution mass spectrometer to be used at the Lunar Receiving Laboratory in Houston for preliminary characterization of organic matter in returned lunar samples. The success of the Lunar Receiving Laboratory program (Professor Burlingame, UCB, and Professor Biemann, MIT) is heavily dependent upon this current development program and its personnel and facilities in Berkeley.

II. UPGRADING OF EXISTING EQUIPMENT AND FACILITIES; NEW EQUIPMENT

Courage to undertake implementation of this program welled up with the occupancy of the new Space Sciences Laboratory in March and April 1966.*

Existing equipment, previously housed in Leuschner Observatory and the College of Chemistry, that was moved and installed on the first floor of the new Space Sciences Laboratory included:

1. CEC 21-110 high resolution mass spectrometer
2. Jarrall-Asch Model 23-500 microphotometer and associated electronic logic circuitry
3. Ampex TM-7 ID incremental magnetic tape memory unit
4. Chemical laboratory equipment

The disassembly, modification, and rebuilding of the CEC 21-110B high resolution mass spectrometer was accomplished partially by funds made available from NASA Grant NsG 101 and was aided considerably by funds from the present grant. This instrument's performance was increased approximately a factor of three and the operating reliability was enhanced significantly in the new electrostatically shielded (Faraday cage) room.

Funds granted in NsG 243 (Suppl. 5) provided minimal hardware to initiate the closed-loop computer control of a high resolution mass spectrometry facility. Specifically, these funds have been utilized to purchase a special high sensitivity double focusing mass spectrometer (AEI MS-9; delivery expected April 1967) and the purchase of the central processor and magnetic disc of a real-time data-acquisition and on-line control computer (SDS Sigma 7; delivery expected April 1967).

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* See the Report of the Space Sciences Laboratory University of California, Berkeley to the National Aeronautics and Space Administration on Its Facilities Grant NsG(F) 5, January 1967.

Funds are desperately needed to augment the real-time computer system so that work may proceed on the next phases of the program.

III. SDS 930 ON-LINE SYSTEM

The first stage of hardware interfacing and programming for high resolution data acquisition and processing has been successfully completed. This stage consisted of direct digitization of the ion multiplier and on-line data acquisition via the interim SDS 930 computer. Raw data acquisition software has been written to accept data up to approximately 16 Kc clock rates from the 38 Kc analogue-to-digital converter. Processing of this raw data includes: (1) peak smoothing and envelope definition; (2) establishment of mathematical relations between magnetic field strength, mass, and time; (3) calculation of accurate masses from digital data; and (4) assignment of elemental compositions corresponding to these calculated masses. These reduced data are then sorted according to each ion's heteroatom content, and the abundance is plotted vs the carbon-hydrogen ratio, e.g. C_nH_{2n+1}, for ease of mental assimilation and interpretation.

IV. TALKS AND PUBLICATIONS

Burlingame, A. L. On-Line Computer Techniques in High Resolution Mass Spectrometry. Presented at the NIH Symposium on Recent Developments in Research Methods and Instrumentation, Oct. 3—6, 1966, NIH, Bethesda, Maryland.

Smith, D. H. High Resolution Mass Spectrometry: Techniques and Applications to Molecular Structure Problems. Doctoral Dissertation, Department of Chemistry, UCB, March, 1967.

V. PERSONNEL

Under the direction of Professor A. L. Burlingame, the following personnel are engaged in various phases of the Apollo Program for lunar sample analysis. Mr. Richard W. Olsen is responsible for the instrumentation and the computer.

Dr. Pierre Longevialle	—	NATO Fellow, Research Associate
Dr. Peter Schulze	—	Assistant Research Chemist
Dr. Dennis H. Smith	—	Assistant Research Chemist
Joan Tesarek	—	Research Assistant
Richard W. Olsen	—	Senior Development Engineer
Roderick L. Jones	—	Associate Development Engineer
Robert E. Furey	—	Assistant Development Engineer
James T. Wilder	—	Spectroscopist
Gillian Ringland	—	Programmer

Supporting technical staff includes: Jan Hauser — Electronics Technician; Bernd R. T. Simoneit — Spectroscopist; Patricia C. Wszolek — Spectroscopist; Ellen Sloan — Laboratory Technician; Victoria Harlamoff — Laboratory Assistant.

VI. OUTLINE OF PHASE II

Delivery of two major pieces of equipment is expected in April 1967, e.g. AEI high resolution mass spectrometer and SDS Sigma 7 computer. Much initial work will consist of shakedown and interfacing of these new pieces of equipment, in addition to re-interfacing and re-programming Phase I for Sigma 7.

Testing operating parameters of on-line operation is the next major task, e.g., digitization rates vs scan rates, precision of measurement, deconvolution of partially resolved multiplets, etc.

We then will be in a position to apply the information and experience gained through our development of computer-controlled mass spectrometry facilities and automatic data acquisition and processing to the Lunar Receiving Laboratory program (initiated jointly with Professor Biemann's group at Massachusetts Institute of Technology).

In the ensuing period the hiring and training of additional personnel will receive careful attention to insure the rapid advance of both the technical and scientific aspects of our program.