

S-13930

# Development of Analytical Techniques for Hydrocarbons in Mineral Aggregates for National Aeronautics and Space Administration

Contract No. NASw-438

Quarterly Status Report No. 4

July - September, 1964

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DEVELOPMENT OF ANALYTICAL TECHNIQUES FOR  
HYDROCARBONS IN MINERAL AGGREGATES

for

National Aeronautics and Space Administration

under

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Written by: D. P. Stevenson  
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SHELL DEVELOPMENT COMPANY  
Emeryville, California

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In the previous phase of this investigation, a method was developed, and tested, for the recovery and characterization of small quantities of C<sub>14</sub> plus, nominally saturated hydrocarbons that may be associated with mineral matter, particularly sedimentary rocks. The method, consisting of Soxhlet extraction of the sample followed by elution chromatography over silica gel and characterization of the isolated fractions by ultraviolet-, infrared- and mass spectrometry, was designed for the purpose of examining the organic chemicals content of meteoritic or other extra-terrestrial matter for possible biological origin.

In the present phase of our work, we plan to apply this method to the examination of a variety of meteoritic specimens, and terrestrial rocks spanning a wide geological period. Analytical data on samples in the latter group are to serve as background for the interpretation of results of analyses of extra-terrestrial specimens.

There are five meteoritic specimens, from 150 mg to 1 g each, on hand. Of these, three (Orgueil, Murray and Felix) represent Classes I, II and III of the carbonaceous chondrites. The other two meteorites are also stony but non-carbonaceous. The terrestrial sedimentary rock samples range from a series of young marine sediments to the nearly three billion-year old pyritic slates from Western Australia (see Table 1). Quantities on hand promise to yield extracts of adequate size for detailed examination.

The analytical approach to be followed is basically that described in our Report covering the work period of July, 1962 to June, 1963. It is expected, however, that some new techniques will be added and some of the established ones will undergo refinements necessary to assure maximum cleanliness in sample handling.

Thus, samples are prepared for extraction by crushing them with carbide-free tools followed by pulverization with a ceramic ball in a high-alumina vial of an impact shaker. In addition to Soxhlet extraction, the transfer of the organic constituents into a proper solvent by dissolving the inorganic framework of the sedimentary samples is being studied. As another alternative, the use of ultrasonics is being contemplated. Beside rapid and more complete recovery, attention is focused on avoiding any change in the molecular nature of the extracts. Preliminary experiments with both techniques are encouraging.

Gas-liquid partition chromatography has been proven to be one of the most powerful analytical tools. Capillary column equipment suitable for the fractionation of samples in the molecular weight range of our extracts is available and is being tested.

Table 1. LIST OF SEDIMENTARY ROCK SAMPLES

| Rock Type                              | Geographical Location                                 | Geological Period | Approx. Age, 10 <sup>6</sup> yrs. |
|--|---|-------------------|-----------------------------------|
| Pacific Ocean Sediments<br>(9 samples) | ca. 300 mi. north of Darwin, No. Territory, Australia | Pliocene          | 1 to 8                            |
| Coarse-grained "white" limestone       | San Bernardino County, California                     | Pennsylvanian     | 310                               |
| Fine-grained "blue" limestone          | San Bernardino County, California                     | Pennsylvanian     | 310                               |
| "Gray" limestone                       | Mono County, California                               | Mississippian     | 350                               |
| Chattanooga Shale                      | Young's Bend, Tennessee                               | Upper Devonian    | 365                               |
| Black Shale                            | Haggum, Sweden  | Upper Cambrian    | 510                               |
| Black Shale                            | Billingen, Sweden                                     | Upper Cambrian    | 510                               |
| Pyritic Slate<br>(2 samples)           | Kalgoorlie, Western Australia                         | Pre-Cambrian      | 2700                              |