MASS SPECTROMETRIC ANALYTICAL SERVICES AND RESEARCH ACTIVITIES
TO SUPPORT COAL-LIQUID CHARACTERIZATION RESEARCH

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ABSTRACT

A Perkin-Elmer 3920 gas chromatograph and associated software has been ordered. We are in the process of acquiring the materials necessary to interface it to the CEC 21-110B mass spectrometer and the other auxiliary components of the total GC/MS system.

Elemental composition data were obtained from thirty-five coal-liquid samples from the mass spectra of each sample recorded at high resolution on photographic plates.

OBJECTIVE AND SCOPE OF WORK

The objective of this program is to obtain qualitative and semi-quantitative information concerning the composition of synthetic fuels and related materials using the technique of mass spectrometry. Information relating to the compound types and/or compounds present in a sample will be obtained from high resolution electron-impact and field-ionization mass spectral, low-resolution field-ionization mass spectral, and combination gas-chromatographic/electron-impact and field-ionization mass spectral data. Field-ionization ion abundances and gas chromatograms will be used to arrive at semi-quantitative estimates of sample composition. Known mixtures of compounds structurally related to those either believed or shown to be present in these samples will be investigated with the view of 1) determining sensitivities towards field ionization and 2) ascertaining for sample quantification the accuracy obtained by various techniques, e.g., low-voltage electron-impact and field-ionization mass spectrometry and various methods of chromatography.

In this research program, attention will, as required, be devoted to the modification of existing and/or the development of new techniques associated with the mass spectral analysis of fossil fuels.

SUMMARY OF PROGRESS TO DATE

Commitments to provide high resolution mass spectral data and assignments of elemental compositions based upon these data are being met. Acquisition of components for proposed modifications to the mass spectrometer is on schedule, if not somewhat ahead.

The principal investigator has devoted ca. 25% of his time to this project. During the next quarter the principal investigator will devote 10% of his time to the project.

DETAILED DESCRIPTION OF TECHNICAL PROGRESS

1. Construction of GC-MS system. A Perkin-Elmer gas chromatograph has been ordered for interfacing to the existing CEC-21-110B mass spectrometer. This item will be used on the current project. Auxiliary equipment for the GC-MS system has been either obtained or ordered i.e., Keithley 417 picoammeter, Leads and Northrup dual pen recorder (at no charge to this grant), a single-stage, glass-jet molecular separator, and the electronic components for fabrication of a control unit to permit rapid magnetic scanning. Interfacing of the auxiliary equipment has largely been accomplished.
2. Field Ionization Mass Spectrometry. A field ionization source has been constructed and installed into the CEC-21-110B by Mr. G. J. Greenwood using funds obtained on a separate coal liquid research project. The field ionization system will be utilized in this project in the near future.
3. Processing of Coal Liquid Samples. Thirty-five coal liquid samples have been processed (17 Western Kentucky and 18 Synthoil samples) for the Energy Research Center at Bartlesville. High (70 eV) and low (8 eV) energy, low-resolution mass spectra and high-resolution spectra on photographic plates have been obtained for each samples. Empirical formulas for approximately 3000 ions have been obtained from the photographic plates by computer processing of the line positions for each ion measured with respect to lines corresponding to ions from perfluorokerosene.