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Progress Report

During the period extending from July 1, 1965 through December 31, 1965, this laboratory completed its development of a fast scan high resolution mass spectrometer system for use in conjunction with gas chromatography instrumentation. From the results obtained to date, we are of the opinion that this system represents the most advanced state of the art and appears to have generated wide spread interest in the scientific community. Because of this, many laboratories are now in the process of adopting this technique. In essence we now have the capability of

a) Rapid High Resolution Scanning

A chromatographic peak can be scanned in 6 to 10 seconds, with a resolving power of at least 1 in 10,000, covering a decade in mass. This can be carried out on a peak containing approximately 0.6 microgram.

b) Rapid Retrieval of Data

The data is recorded on a magnetic tape system at 60 inches per second. One can then play it back into an oscillographic recorder at 1 7/8 ips. at any time to check the spectrum. The analog tape is then digitized. The digitized data is then analyzed by means

of computer programs and all the fragments of the spectrum are converted into accurate masses (accuracy better then 10 ppm) and the elemental composition of each mass is determined. The state of the technology is such that this information can be obtained in real time if a fast digitizer is available for on line operation. Approximately 60 mass spectra can be conveniently obtained from a chromatographic run if necessary.

c) Automatic switching from high to low resolution mode during the course of a chromatographic analysis. This enables one to procure low resolution spectra (1 in 1500) on trace components bands containing less than 0.3 microgram of material.

d) Rapid High Resolution Scans on relatively non-volatile samples introduced into the ion source by means of a direct insertion probe. By scanning over a 72 second to 100 second period, depending upon sample size and resolution desired (one in 10 to 20,000 plus) and recording on magnetic tape, 'element maps' can be procured very readily here.

Molecular Separators

One of the most important facets of the mass spectrometer-gas chromatograph tandem operation is the interfacing with a molecular separator. The efficiency of the separator in removing the bulk of the carrier gas prior to the entrance of the chromatographic effluents into the ion source contributes much to the overall

sensitivity of the system. Thus far three systems have been investigated.

a) The fritted tube system (Watson-Biemann). Under optimal conditions, this system was found to provide efficiencies of 8 to 20% depending upon flow rate, temperature, pumping speed, and the molecular weight of compounds being analyzed.

b) The molecular separator system (Ryhage). Under optimal conditions early versions of this device provided efficiencies in the 1-3% range. Changing the diameters of the jets and increasing the pumping speed on the second stage of the system provided a significant improvement. Efficiencies are now in the 40-50% range.

c) High temperature membranes. Early results here are intriguing and will be pursued. Using teflon or silicone films one notices absorption, solubilization and/or diffusion of organic molecules across these membranes, depending upon temperature, membrane thickness, pumping speed, time constants, etc. Indications are that one can procure organic molecules completely free of carrier gas under the proper circumstances. Experiments determining sample loss under optimal conditions will be undertaken shortly.

Support Coated Porous Layer Columns

This type of column developed several years ago by Horvath (who now is a member of our laboratory group) was carefully appraised for use in conjunction with the tandem combination of instruments. It appears ideally suited for this application for

the following reasons:

a) a 50 ft. 0.020 i.d. column yields 25,000 theoretical plates

b) it will accept loads varying from fractions of a microgram to hundreds of micrograms without appreciable loss of resolution

c) carrier gas flow rates are small - 4 to 10 ml. helium per minute

d) the bleed rate of liquid phase is exceedingly low - thus providing one with an excellent signal to noise ratio

e) sample splitting is not required

f) columns can be made reproducibly

Computer Programs

Programs were devised to find the masses and elemental composition of all of the fragments of a spectrum obtained during the fast scan mode.

Biochemical Analysis

Following the completion of the effort to optimize molecular separator design and functions, we will be in a position to start our investigations characterizing organic compounds found in different types of rocks, soils etc. In this regard it is also planned to perform pyrolysis studies on known biochemical compounds, bacteria, fungus, viruses etc. Pyrolysis conditions will be optimized and products identified in an attempt to reconstruct the nature of the original starting material.