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Analytical Instruments

Continued improvements in analytical instrumentation are perhaps the most crucial factor in the vitality of scientific research. This issue presents a sampling of further advances in analytical capabilities. In recent years the kinds of substances that can be studied have increased. Sensitivity of detection has been enhanced. Abilities to isolate minor constituents of complex mixtures have been improved. The time required for analytical procedures has diminished.

Gas chromatography coupled with mass spectrometry has been for some time the analytical method of choice for volatile substances. Even so, it continues to be improved, for example, through the introduction of durable, coated capillary columns. With such a device it has been possible to separate a mixture of 100 plant hydrocarbons in only 6 minutes.

Liquid chromatography is especially useful in dealing with hydrophilic substances, including many large molecules of biological importance. Resolution and speed of liquid chromatography columns have been improved. However, the usefulness of liquid chromatography has been less than ideal. The bottleneck has been detector systems. In special cases spectrophotometry, electrophoresis, or fluorescence may be employed, but these methods lack the power and versatility of mass spectrometry. Earlier when mass spectrometry was employed, the usual practice was to collect a peak, evaporate off the eluting liquid, and coat part of the residue on an electrode that was subsequently inserted into the mass spectrometer. The solid was then subjected to a high electric field, fast atom ion bombardment, or other excitation that created ions. New techniques now permit better interfacing of liquid chromatography with mass spectrometry. A thermospray method described in this issue facilitates removal of the eluting liquid and ionization of the eluted substances. Thus the fluid from the liquid chromatography can be injected directly into the mass spectrometer. The molecular weights of undegraded molecules can be determined as well as the weights of fragments.

The mass spectrometer can be used to provide information about solids that ordinarily cannot be analyzed by liquid chromatography. A small portion of the material is subjected to practically instantaneous pyrolysis within the mass spectrometer. The fragments produced give a good representation of the nature of the parent substances.

Mass spectrometers have been undergoing development with, for example, new ion sources and the tandem configuration. An interesting version is the Fourier transform mass spectrometer. The crucial volume in the device is a cube 2.54 centimeters on a side, bounded by electrodes, and maintained in a magnetic field. The device is operated as an ion trap with decay times of as much as half a day. Within the trap a series of operations is conducted, including ion formation, storage, manipulation, and detection. Resolution at a mass to charge ratio of 18 is 100,000,000.

The value of applications of nuclear magnetic resonance continues to increase. Organic chemists find it almost indispensable in the determination of structure of compounds present in a liquid phase. It is being applied increasingly to study of solids, and the earlier emphasis on observations of protons has been supplemented by measurements of other nuclides. One dynamic application is use in noninvasive imaging in medicine. For many tissues nuclear magnetic resonance is superior to the CAT scan.

Lasers are being increasingly used as key components in analytical processes. One use involves the application of tunable dye lasers and takes advantage of resonant multiple photon ionization to attain excellent selectivity. Illustrative of the power of the method is a demonstration that 10^3 atoms of krypton-81 can be sorted out and counted in a sample along with 10⁷ atoms of krypton-80 and of krypton-82 plus 10¹² atoms of other atoms or molecules. With the same techniques it was possible to detect 10^{-8} to 10^{-10} of a monolayer of anthracene and naphthalene adsorbed on graphite.

-PHILIP H. ABELSON