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Infrared Microspectrometer Using Helium Recoil to Monitor Capillary Gas Chromatography Effluents

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NOTE

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Summary

Various types of optical spectra could be obtained with the sensitivity of mass spectrometry by combining components from the latter and from molecular-beam technology as proposed.

The infrared (IR) spectra of organic vapors eluted from open tubular columns can be obtained by low-energy electron scattering (*1*), inelastic electron tunneling (*2*, *3*), and other methods (*4*). However, the construction of the required instruments is rather difficult in a chemical laboratory. In this paper, a simpler spectrometer is proposed for monitoring the IR spectra of capillary effluents as shown in Fig. 1.

The essential part of this model is a molecular-beam source chamber (I) that is irradiated by spectrally resolved radiation from a fast-scan IR source (III).

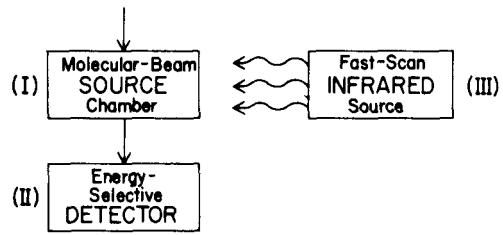

FIG. 1. Block diagram of the helium-recoil microspectrometer.

From the capillary column, effluent organic vapor and helium carrier gas enter I, which is maintained at a pressure of ca. 1 torr. The intense radiation from III raises the vibrational-rotational energy of some of these molecules.

As this IR radiation is monochromatic, it is absorbed by just one vibrational energy level of a molecule, e.g., a stretching vibration. The absorbed energy is next distributed over the molecule by filling lower energy levels, e.g., bending, rocking, wagging, twisting, and deforming vibrations. Most of the resulting vibrational modes will have an energy which is at least as large as the mean translational energy of the molecules. Under the conditions of the experiment, a molecule thus excited will undergo, within its lifetime, some 10^3 collisions with the helium atoms that are present in a large majority. The multitude of excited vibrational modes makes it highly probable that some of these collisions will be successful, removing the excess energy of the molecule by one vibrational level step at a time. In a successful collision, this vibrational energy is converted into translational energy of both collision partners. The latter rebound and, according to the principle of conservation of momentum, almost all of the resulting acceleration is imparted to the helium atom, due to its much smaller mass. Thus, this atom momentarily attains a velocity which can exceed the range of its thermal velocity distribution.

Some of the recoiling helium atoms leave I through a source-canal (*5*) slit, entering a molecular beam which continuously effuses from there.

Part II of the microspectrometer is a molecular-beam detector, of the electron-bombardment type, which is made selective for the accelerated helium atoms.

The helium atoms which emerge from the source slit are converted into singly-charged ions by an electron-impact source of low voltage. The resulting ion beam is introduced into an electrostatic energy-selector (*6*)—consisting of two coaxial, sector-shaped, cylindrical electrodes—which is aligned with the source slit. The radial electric field of the selector is adjusted so that it permits the passage of only those ions whose kinetic energy exceeds the maximum available from thermal distribution in I. An ion detector such as a magnetic electron multiplier is placed at the image plane produced by the electric field.

The IR spectrum of the effluent will be recorded by the microspectrometer as the plot of the wavelength of the radiation from III vs. the ion current detected in II when the spectrum is scanned much more

rapidly than the vapor band is eluted from the column. If the scan time is not negligible compared to the elution time, then the IR absorption will vary with the changing concentration of the vapor flowing through I. To compensate for this dependence, the response of II must be electrically counterbalanced by that of whatever gas chromatographic detector is used in conjunction with the microspectrometer.

Ultraviolet spectra, and, e.g., the circular dichroism of enantiomers, could be plotted similarly.

Many other types of energy selectors or velocity filters could also be used in II. The most versatile ion analyzer for this is the double-focusing mass spectrometer. It can simultaneously function as a gas chromatographic detector by intermittently monitoring the total ion current at an electron energy momentarily held below the ionization potential of helium.

For optical spectrometry, this mass spectrometer could be used as II by setting it to monitor mass 4. The helium ions should then be electrostatically accelerated only in the region between the electrostatic energy-selector and the magnetic analyzer by accelerator slits inserted there.

For mass spectrometry, the simplest procedure would be to leave the capillary column directly coupled to the inlet of I, thus dispensing with previous enrichment by a molecular separator, rather than to use a separate inlet leak into II. The mass spectrometer should then be operated with conventional ion acceleration but at a lower electron accelerating voltage, e.g., 20 V, so that the carrier gas is not ionized (*7*).

When such a mass spectrometer is used in II, three types of spectra—IR, UV, MS—become available for the structure elucidation of an eluted compound by rapid consecutive scannings of a single peak with the combined instrument.

Microspectrometers that are similar but more sophisticated (*8*, *9*) have the compensating advantage of utilizing radiative cooling of the molecular beam. They are more sensitive by thus suppressing the background due to molecules which are thermally excited to a higher vibrational state. They also can be used for optical spectrometry of unstable free radicals even though these are in an excited state at the moment of formation.

REFERENCES

1. F. M. Probst and T. C. Piper, *J. Vac. Sci. Technol.*, **4**, 53 (1967).
2. J. J. Lambe and R. C. Jaklevic, U. S. Patent 3,469,184 (September 23, 1969).

3. T. D. Clark, Brit. Patent 1,171,426 (November 19, 1969).
4. S. Behrendt, *J. Gas Chromatogr.*, **6**, 226 (1968).
5. M. Smoluchowski, *Ann. Phys.*, 33, 1559 (1910).
6. R. Herzog, *Z. Phys.* **89**, 447 (1934).
7. R. E. Ryhage, U. S. Patent 3,430 040 (February 25, 1969).
8. S. Behrendt, *Nature,* **201**, 70 (1964).
9. S. Behrendt and H. Richtering, *J. Chromatogr.*, **24**, 1 (1966).

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