

Rapid Infrared Analysis of Gas-chromatography Peaks

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A MULTIPLE-SCAN interference spectrometer was recently described.¹ The instrument is highly sensitive, and exploratory experiments showed that it could be used for infrared transmission, reflection, and emission spectroscopy, especially in situations where the signals obtained are weak. The instrument has now been used for the infrared-spectral analysis of the effluent gas of a gas-chromatography column.

A Block model 200 interference spectrometer² was used. The instrument could scan the range 4–40 μ in one second with a resolution of approximately 20 cm^{-1} . The signals of consecutive scans, instead of being stored directly in the core memory of the computer that is an integral part of the instrument, were recorded on magnetic tape. It was then possible to process any one single signal of a sequence, or to sum the numerous signals, by transferring the taped signals into the computer memory. The latter could also function in a "subtract" as well as an "add" mode, so that it was possible to subtract "background" from a signal and thus consider only spectral information. The effluent of a gas-chromatography column was passed through a cylindrical sample cell (80 mm. long \times 3 mm.) fitted with KBr windows. That

cell, and others, could be positioned between a radiation source (a Globar) and the aperture of the Michelson interferometer, or between the interferometer and the detector.

An example of the type of data obtainable is given in Figures 1 and 2. As the effluent stream carrying a 1 μl . sample of acetone eluted from the column passed through the infrared cell, consecutive one-second scans were made. Figure 1 shows several of the spectra obtained at different times after the acetone sample was flowing through the infrared cell. These and other spectra show the intensities of the acetone absorption bands to vary with time. This is more clearly seen by the plot of the intensity of the most prominent absorption band near 5.8 μ as function of time, given in Figure 2 (not all spectra were plotted). Also shown is the shape of the eluted acetone peak as drawn by the recorder of the chromatograph. Comparison of the two plots indicates that the curve of infrared band intensity approximates the general shape of the chromatograph peak. The two curves differ slightly because the concentration gradients in the flow-through infrared cell did not exactly match those within the gas-chromatograph detector. Measurements of this type permit the

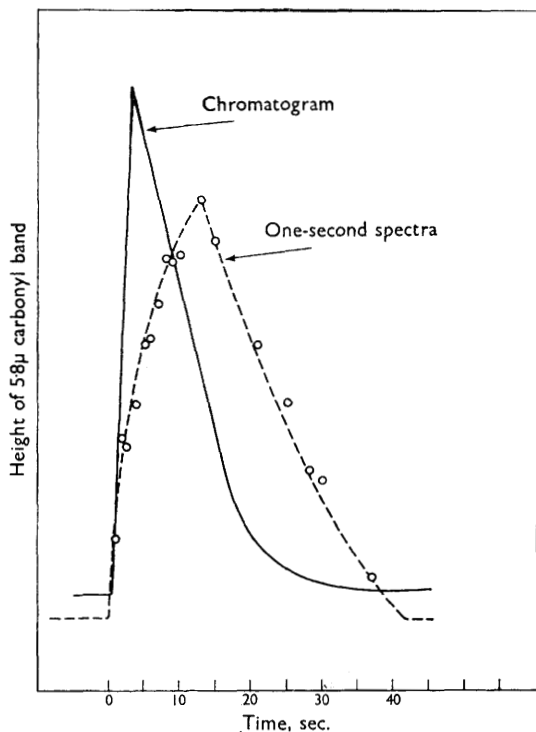


FIGURE 1. Single scan spectra. Consecutive spectra were taken at one-second intervals across the profile of a 1 μ l. acetone peak eluted from a column. The number next to each spectrum indicates the time in seconds that elapsed since the acetone sample entered the infrared cell. Each spectrum is corrected for background.

spectral examination of several segments of an eluted peak which is suspected to be incompletely resolved.

These and other experiments indicate that the following measurements are possible over the 4–40 μ range, using the techniques briefly outlined above. (a) Single-scan, one-second spectra over any segment of an eluted peak. (b) Consecutive one-second spectra across the profile of any

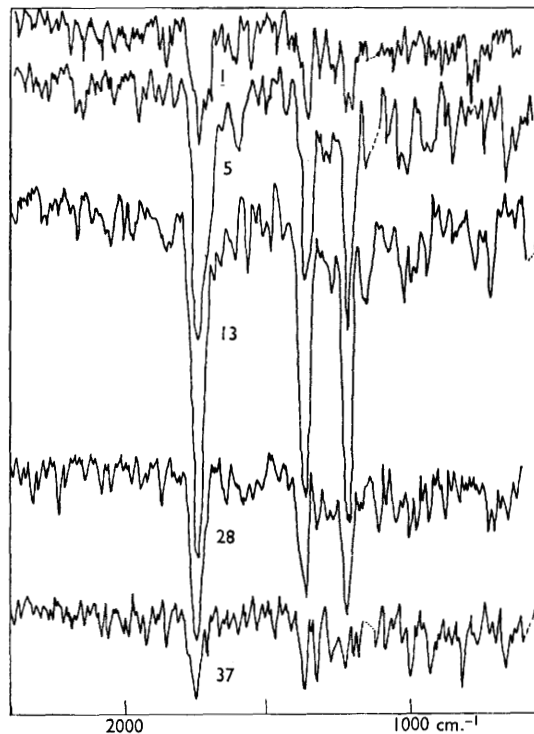


FIGURE 2. Profile of eluted 1 μ l. acetone sample.

peak or combination of peaks, the consecutive signals being recorded on magnetic tape. (c) If the gas stream is stopped or by-passed at a suitable time, an eluted sample, or fraction of an eluted sample, can be trapped within the infrared cell. Multiple scanning is then possible, the consecutive signals being recorded on tape or the cumulative signal being stored directly in the core memory of the computer. This permits the examination of small eluted samples.

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¹ M. J. D. Low and I. Coleman, *Spectrochim. Acta*, 1966, **22**, 369.

² Block Engineering Co., Cambridge, Mass.