

Fig. 4. Infrared spectra of the compound $2\text{NaC}_8\text{H}_8$ at different temperatures. 1. 25°C. 2. 80°C.

Information on research devices and a more comprehensive treatise of the results will be given later.

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“Solvent Peak Subtraction”. Gas Chromatographic Determination of Low-Boiling Compounds in the Presence of an Interfering Solvent

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In some cases one has to make a gas chromatogram of a mixture of low- and high-boiling compounds in a low-boiling solvent. The choice of solvent may be limited, or perhaps only one solvent is possible. To achieve good resolution of the high-boiling component peaks, perhaps only one column can be used. In such case it may be very difficult to separate the peak or peaks of the lowest boiling components from that of the solvent, even at low temperatures. It is, however, possible to improve the situation by what we call “solvent peak subtraction”.

In principle it would be possible to eliminate the solvent peak totally, leaving the low-boiling component peaks free. This could be achieved with a double-column system with parallel, identical

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columns, identical detectors and identical gas currents. An amount of sample could be injected onto one of the columns and, exactly at the same moment, exactly the same amount of solvent onto the other column. The second injection would then

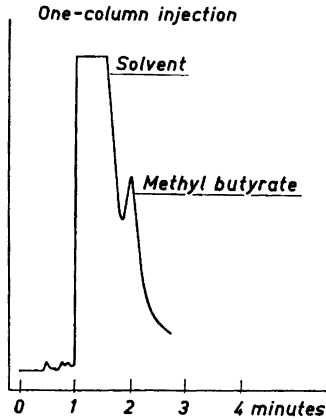


Fig. 1.

totally cancel the solvent peak. But the method requires an exact knowledge of the amount of solvent in the sample, very exact injection syringes, and either two persons injecting or some sort of "two-column injector", which has not yet been constructed. However, it is possible to subtract part of the solvent peak by injecting less solvent than in the sample onto the second column either a few seconds before or a few seconds after the sample injection.

An example is shown in Figs. 1 and 2. The sample is a mixture of fatty acid methyl esters from methyl butyrate upwards, dissolved in methylene chloride-

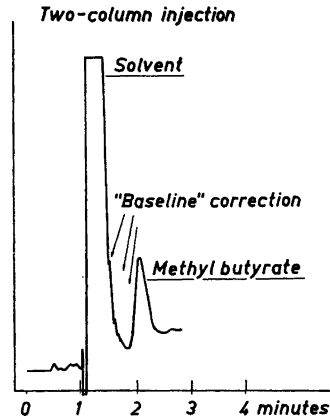


Fig. 2.

methanol 2:3 (the transesterification mixture from milk fat). The gas chromatograph is a Perkin-Elmer 880 with flame ionisation detectors, the columns (1/8" × 2 metres, steel) 10% DEGS on Chromosorb W, 80-100 mesh, acid-washed, DMCS-treated, the temperature 60°C (isothermal), and the carrier gas (helium) flow 30 ml/min. (The rest of the chromatograms, not shown in the figures, are performed with temperature programming and, at the end, isothermally at 200°C). In Fig. 1, 1 μl of sample is injected onto the first column. In Fig. 2 the same injection is done, but about 7 seconds later 0.5 μl of methanol is injected onto the second column. In this case some base line correction is necessary before the methyl butyrate peak arrives, but the peak is well separated from the solvent peak and well suited for integration.

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