

Note

New detection technique in liquid chromatography

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At present, liquid column chromatography is passing through a period of rapid development as a result of the latest advances in the theory and engineering of high-efficiency columns and in instrumentation. Unfortunately, however, no detector has been developed whose performance can match that of the flame ionization detector, which has a high sensitivity, a broad linear dynamic range, a high degree of versatility and a short response time¹. The transport detector² is very promising, but the current designs of this detector are not optimal³.

This paper deals with a new detection technique⁴ which, in our opinion, is simpler and more sensitive than the present transport detectors. The essence of the proposed technique is that the first two steps, *viz.*, (1) application of the eluate solution containing the separated compounds on the transport element and (2) evaporation of the volatile solvent, are segregated, in space and in time, from the subsequent two steps, (3) detection of the separated zones and (4) regeneration (preparation) of the transport element for the next analysis. This permits a simplification of the design of the detector as a whole, an enhancement of its reliability, an improvement of the detection sensitivity and an increase in the detector efficiency.

First, the eluate containing the separated compounds from the column is continuously applied on a slowly moving conveyor (*e.g.*, a belt moving at a linear speed of V cm/min). Under the conditions of a supply of heated gas and moderate heating, the volatile mobile phase evaporates leaving the non-volatile components on the belt. At this stage the belt is not fed into the detector. After the separation, the belt carrying the separated compounds moves into an independent or semi-independent flow detector (*e.g.*, flame ionization or other destructive detectors), and at the second stage of detection it moves at a higher speed, W (cm/min), than at the first stage when the separated compounds were applied on the belt.

To a first approximation, a comparison of the sensitivity threshold of detecting the separated compounds by the two-stage method and by the straight-through continuous method suggests that the sensitivity threshold of the proposed method is higher. Indeed,

$$S_{11} = \frac{W}{V} \cdot S_1 \quad (1)$$

where S_I and S_{II} are the sensitivity thresholds of the straight-through (I) and two-stage (II) methods, respectively.

Fig. 1 shows two chromatograms representing squalane zones of the same length and sample size (10 l), obtained with the aid of a flame ionization detector. The first chromatogram was taken at a belt speed of 30 cm/min and the second, at 100 cm/min. As can be seen, the squalane peak height has increased by a factor of 3.5, which is close to the calculated value. Different types of transport elements (belt, disk, wire, mesh, etc.) can be used, as well as different detectors, and the analyzed compounds may undergo different chemical transformations. It should be noted that the detector unit in which the second stage takes place may be associated with several column chromatographs at one time since the detection state (registering the separation results) is, in this case, several times faster than the chromatographic separation itself. This renders the required instrumentation less expensive and simpler.

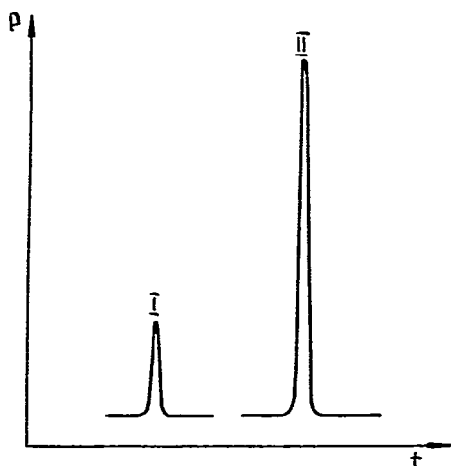


Fig. 1. Chromatograms obtained by detection of the squalane zone using the conventional two-stage method (I) and the proposed two-stage method (II). P = Reading of the flame ionization detector; t = time.

In some cases, the proposed technique should also be used in a rigidly coupled column-detector system, while programming the speed of the transport element. The new technique appears to be promising for liquid chromatography.

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