Thin-layer chromatography using partially acetylated cellulose as adsorbent

The separation of polycyclic aromatic hydrocarbons by chromatography on acetylated paper (and also on columns of partially acetylated cellulose) has been described by SPOTSWOOD[1,2], and the technique has since found extensive application. The method suffers from the disadvantage that different batches of acetylated paper, and of acetylated cellulose, have somewhat different characteristics. It has now been found that polycyclic aromatic hydrocarbons can also be satisfactorily separated by thin-layer chromatography using acetylated cellulose. No adhesive is required to prepare a satisfactory plate. The separated compounds, readily visible under ultraviolet light, can be scraped from the plates and identified by spectroscopy.

This method has the advantage that a large batch of acetylated cellulose can be prepared and used for the preparation of a great many plates over a prolonged period. Reproducible results can be obtained with a given batch of acetylated cellulose, and it may also be noted that the acetylated cellulose can be recovered and used again.

It is to be hoped that good quality acetylated cellulose will become commercially available; if distinguished by a batch number, this would facilitate the comparison of R_F values determined in different laboratories.

TABLE I

Compound	R_F
3:4-Benzopyrene	0.105
3:4-Benzofluoranthene	0.150
3:4; 9:10-Dibenzopyrene	0.217
11:12-Benzofluoranthene	0.230
Perylene	0.342
1:2; 4:5-Dibenzopyrene	0.497
1:2; 3:4-Dibenzopyrene	0.559

Table I lists the R_F values obtained for several polycyclic aromatic hydrocarbons, the solvent system being methanol–ether–water (4:4:1).

Experimental

Whatman's cellulose chromatography powder (200 g) was acetylated by SPOTSWOOD's method[2] using a mixture of thiophen-free benzene (1700 ml) and acetic anhydride (800 ml) containing 92 % sulphuric acid (4 g) and 72 % perchloric acid (4 g).

A suspension of acetylated cellulose (35 g) in methanol or ethanol (60 ml) was applied to glass plates (20 cm × 20 cm) using a "Desaga" thin-layer spreading device, adjusted to give a 250 μ layer. The resulting plates were dried in air; compounds under test were applied in the usual way in 0.5 % solution; and the plates were developed by the ascending technique in a saturated chamber, the best results being obtained when the chromatography was carried out in an air-conditioned room (68°F). The solvent systems methanol–ether–water (4:4:1) and toluene–ethanol–water

(4:17:1) both proved satisfactory, but the former was found to be preferable as it gave more compact spots.

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[1] T. M. SPOTSWOOD, *J. Chromatog.*, 2 (1959) 9.
[2] T. M. SPOTSWOOD, *J. Chromatog.*, 3 (1960) 101.

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Note

Similar results were described by T. WIELAND, G. LÜBEN AND H. DETERMAN, *Experientia*, 18 (1962) 430, on September 15th, 1962, and it is unlikely that these had reached Australia when this paper was sent off (October 24th, 1962).

THE EDITOR

Notes

A gas chromatographic trap designed to collect compounds which tend to form aerosols

A number of gas–liquid chromatographic trap designs have been reported in the literature[1–6], but no simple device has been described for the recovery of aerosol-forming compounds. Conventional cold traps are very inefficient for the collection of compounds such as the polynuclear aromatic hydrocarbons, sterols and high molecular weight paraffins. Yields are often as low as 60 %[5]. In this laboratory, for example, a 2 ft. × 5 mm O.D. glass coil cooled in liquid air gave recoveries of 50–60 % for many aromatic hydrocarbons.

We wish to describe a Pyrex glass trap which has been used successfully to collect samples eluted from 1/4 in. to 5/8 in. GLC columns at flow rates of from 40 to 200 ml/min with recoveries greater than 92 %.

This trap (Fig. 1) is designed to achieve a differential temperature between the outer wall and the inner wall of the condensing surface. The inner wall is kept at room temperature by directing a flow of air into the inner well while the outside wall is kept at the temperature of liquid air. The temperature differential between the two walls through which the exit gas must flow creates a turbulence, thus increasing the number of contacts of the aerosol particle with the cold wall and improving the trapping efficiency.

The exit line leading to the cold trap is 1/8 in. O.D. stainless steel tubing. This