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Note

Detection of radioactive compounds during development on a thin layer

F. JIMENO DE OSSÓ
Junta de Energía Nuclear, Dirección de Física y Química Nuclear, Madrid-3 (Spain)

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Continuous flow development in thin-layer chromatography (TLC) with evaporation of the solvent at the upper end of the layer[1] is a method of increasing the number of theoretical plates[2] and carrying out the separation over an unlimited period. Compounds that have similar polarities can be separated by this procedure with solvents of low polarity, which enhance the differences in the polarities of the compounds; however, when they are mixed with substances with different polarities, the latter migrate together with the solvent front or remain at the origin after the plate has been developed and dried.

By placing a detector for radioactive compounds on the wall of a chromatographic chamber, above the point of application on the plate, the radioactive substances in the initial mixture will be detected as they are developed along the plate and their activities can also be recorded, although they may be found together at the top end of the plate. This procedure can obviously be used when the radioactive compounds do not require development with a continuous solvent flow; the subsequent scanning of the plate can therefore be avoided. A similar procedure has been used[3,4] for the detection of compounds by measuring their electrical conductivity.

EXPERIMENTAL AND RESULTS

A chamber of rectangular section (60 × 20 cm) made of stainless steel is used. In the top of the chamber is a slit for the plate so that the solvent will evaporate once the front has emerged from the slit. The radioactivity on the plate is detected with a Geiger–Müller counter (Philips, Model 18505) through another slit (24 × 3 mm) on the side of the chamber (Fig. 1). The counter is covered with a plastic film (Terphane; 1.8 mg/cm^2) which protects it from solvent vapour. The distance from the counter window to the surface of the TLC layer is 4 mm and the distance from the side slit to the bottom of the chamber is 10 cm. The associated equipment involves a scaler and a rate meter with a power supply, and a recorder.

The procedure has been applied to checking the radiochemical purity of labelled compounds. The advantages can be illustrated by the results obtained with a 2.5-year-old sample of sodium [1-^{14}C]acetate which was initially pure, with a specific activity of 13 mCi/mmole and stored at $-25°$*.

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* Prepared by Dr. Suarez Contreras.

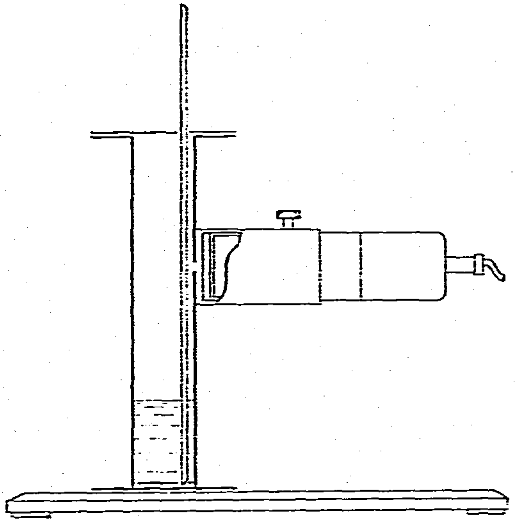

Fig. 1. Chromatographic chamber with Geiger–Müller counter.

The chromatography was performed in the chamber shown in Fig. 1 with saturating paper and pre-coated silica gel plates of dimensions 200 × 30 mm (Merck 5715). At 8 cm from the lower edge of the plate, 5 μl of an aqueous solution containing 3 mg/ml of the compound were applied and development was carried out with ethanol–water–ammonia solution (146:14:32)[5]. The results are given in Fig. 2; after develop-

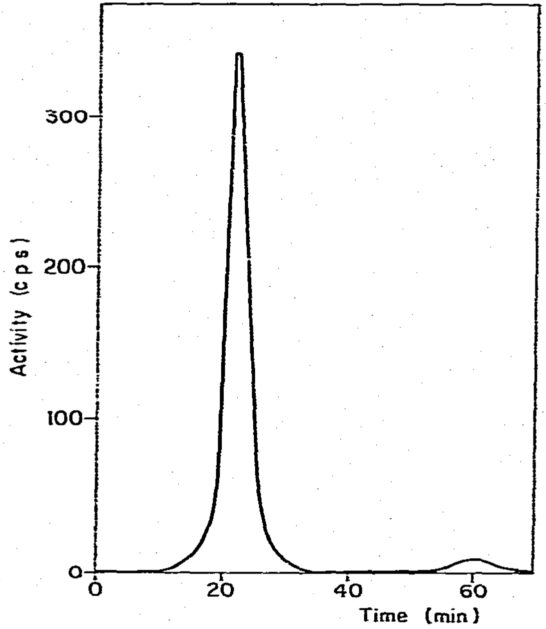

Fig. 2. Radiochemical purity of sodium [1-^{14}C]acetate.

ment for 60 min 5% of an impurity occurred, the development time for sodium acetate being 22 min. The minimum activity that can be detected is 0.05 μCi. In the conventional procedure with the same plate and solvent system, the R_F values of the sodium acetate and the impurity are 0.69 and 0.29, respectively; 125 min are required for development for 110 mm from the point of application, and a further 40 min for detection.

The developed compounds are identified by comparison of their development times with those obtained for reference compounds under the same conditions. If the reference compounds are not radioactive, they are developed beside the test compound and rendered visible at the moment that the activity is recorded; for positive identification, the spot of the reference material must be found in front of the detector slit when the activity of the unknown compound is recorded. By modifying the position of the point of application, different separations can be obtained according to the nature of the applied product.

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