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Preparation of a firm polyamide adsorbent for thin-layer chromatography

A thin-layer chromatographic procedure which accomplishes the separation of isomeric flavanones has been reported[1]. The adsorbent employed is Polyamide Woelm* applied to glass plates as a slurry of 1 part polyamide in 7.5 parts benzene–methanol (2:3, v/v). However, due to the fragility of this adsorbent layer, the handling of the plate, removal of areas by scraping, marking and application of sample must be performed with extreme care. Furthermore, the flakiness of this adsorbent layer seriously reduces its use in autoradiography. This flakiness also increases the probability of instrument contamination when using a radiochromatogram scanner for locating radioactive areas.

The purpose of this investigation was to find a method which would give a firm polyamide layer and retain the polyamide's resolving power. Such a procedure has been found and studied with various flavonoids and under conditions necessary for the detection of radioactive areas.

* Use of a company and/or product name by the department does not imply approval or recommendation of the product to the exclusion of others which may also be suitable.

Experimental

Preparation of plates. A mixture of 0.8 g of rice starch[2–4], 0.4 g of silica gel (Fisher No. 1 impalpable powder*) and 9 ml of water in a covered 20 ml beaker was heated for 40 min on a steam bath with occasional stirring. Water (1–2 ml) was added as needed during the heating to prevent caking on the sides of the beaker. This mixture was rinsed with 3 ml of water into a 100 ml beaker containing 5.5 g of Woelm polyamide powder and 35–40 ml of methanol. This mixture was stirred and then blended in a Waring blendor* microcup for 3 min. The resulting mixture was spread as a 250 μ thick layer on 20 × 20 cm glass plates and allowed to dry 2 h at room temperature before use.

Results and discussion

The procedure described above resulted in an adsorbent layer which was as firm as starch-bound silica gel[2]. While the starch is sufficient to produce a firm polyamide adsorbent layer, the addition of silica gel gives a surface which can be easily written upon with a dull pencil. The use of a Waring blendor gave a slurry which formed a smoother surface than could be obtained by simply stirring the ingredients. After completion of the chromatogram, areas of interest may be readily scraped from the glass plate without loss of material. The firmness of this adsorbent layer allows the plates to be used with a radiochromatogram scanner without instrument contamination.

The resolution of different flavonoids including isomeric flavanones such as naringin, naringenin rutinoside, hesperidin and neohesperidin on polyamide–rice starch–silica gel was comparable to that obtained on polyamide alone[1].

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*U.S. Fruit and Vegetable Products Laboratory***, *600 Ave. S., N.W., Winter Haven, Fla. (U.S.A.)*

H. E. NORDBY
T. J. KEW
J. F. FISHER

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** One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U.S. Department of Agriculture.