CHROM. 4281

Separation of fructosans by thin-layer chromatography

During a study of the metabolism of fructosans in explants of Jerusalem artichoke tubers[1] a rapid chromatographic system for the separation of the lower members of the fructosan series was needed. Although systems have been worked out for the separation of fructosans either by paper chromatography[1,2] or by gel-filtration[3], these methods are too time-consuming for the simultaneous analysis of numerous samples. A thin-layer chromatographic one-dimensional separation technique on cellulose plates was therefore developed, which allows the separation within 8 h of fructose, sucrose and the first seven homologues of the fructosan series.

Thin-layer plates were prepared as follows: 22 g of cellulose (MN 300, Macherey, Nagel & Co., G.F.R.) were mixed in a Waring-blendor with 145 ml of 33 m*M* K_2HPO_4 solution, and the suspension spread on glass plates (5 × 20 cm) using a Desaga spreader (Desaga, Heidelberg) set at a thickness of 0.4 mm. After drying for 24 h at room temperature, 15 μl of a fructosan mixture (100 mg/ml), obtained from Dahlia tubers by water extraction, were applied to the start line by means of a motor-driven Hamilton syringe. After drying with warm air, the plates were chromatographed in one dimension using either of the following solvents:

Solvent A: *n*-propanol–ethyl acetate–water (75:10:15);

Solvent B: *n*-propanol–ethyl acetate–water (60:10:30).

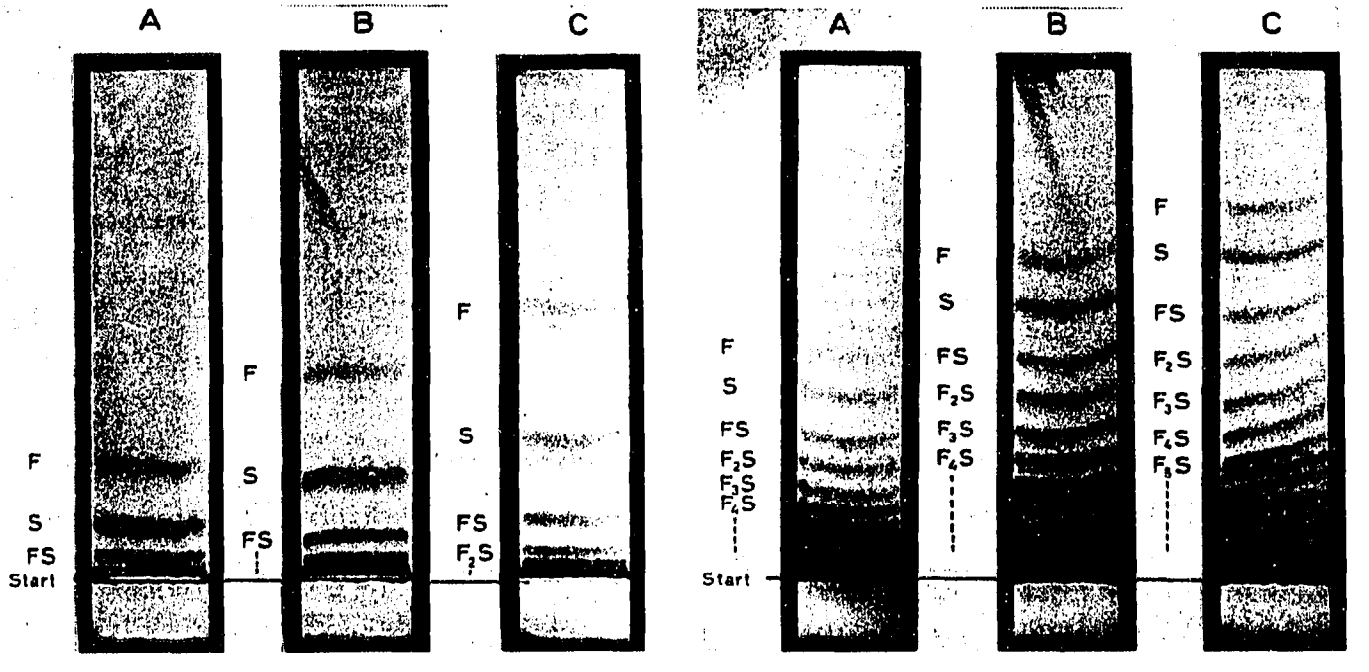

Fig. 1. Separations of fructosan mixtures by thin-layer chromatography in solvent A. The plates were stained with naphthoresorcinol[4]. (A) after one development; (B) after two developments; (C) after three developments.

Fig. 2. Separations of fructosan mixtures by thin-layer chromatography in solvent B. The plates were stained with naphthoresorcinol[4]. (A) after one development; (B) after two developments; (C) after three developments.

The chromatograms were routinely developed twice in the same solvent up to 16 cm above the start line. The plates were dried with warm air before the second development. Each run lasted about 3½ h. For documentation the plates were stained with naphthoresorcinol[4] and photographed on orthochromatic films.

Figs. 1 and 2 show the separations obtained using solvents A and B, respectively; in addition they illustrate the effect of multiple developments in one dimension using a single solvent. As can be seen solvent A is the most efficient for the separation of fructose, glucose, sucrose and FS*, while solvent B is more convenient for separation of the S, FS, F_2S . . . F_7S series.

As already mentioned the plates were coated with suspensions of cellulose in 33 m*M* K_2HPO_4. Cellulose plates prepared without phosphate gave a similar separation pattern, but severe tailing was usually observed. An evaluation of the effect of varying the phosphate concentration revealed that minimal tailing was obtained when the phosphate concentration was kept between 25 and 60 m*M*. Higher phosphate concentrations resulted in decreased R_F values.

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Institute of Biological Chemistry B,
University of Copenhagen (Denmark)

GERT KARLSSON

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* Abbreviations used: F = fructose; S = sucrose; FS = 1-fructosylsucrose; F_2S = (1-fructosyl)$_2$sucrose; F_nS = (1-fructosyl)$_n$sucrose.

CHROM. 4292

Dünnschichtchromatographisch-enzymatischer Nachweis phosphororganischer Insektizide

Zum dünnschichtchromatographischen Verhalten einiger weiterer Insektizide

In einer früheren Arbeit[1] ist über eine empfindliche dünnschichtchromatographisch-enzymatische Methode zum Nachweis insektizider Organophosphate sowie über die Möglichkeiten zur Steigerung der Nachweisempfindlichkeit schwacher bzw. indirekt hemmender Cholinesteraseinhibitoren durch "Aktivierung" berichtet worden. Das insbesondere für Rückstandsuntersuchungen gut geeignete Verfahren kann unter