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Note

Thin-layer chromatographic separation of the lower alkanols as the xanthates

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The lower alkanols were first separated as their xanthates by paper chromatography by Lederer and Summerfield[1] using a short ascending development (*ca.* 6 h) on Whatman No. 1 paper with *n*-butanol–water–ammonia solution (50:45:5). Subsequently several workers tried to repeat these separations using overnight descending development and noted considerable decomposition of the xanthates (for a discussion see Gasparič and Borecký[2]); they proposed the use of more alkaline solvents to avoid decomposition of the xanthates.

It occurred to us that with the advent of high-performance thin-layer chromatography (HPTLC) the xanthates should be re-examined, as the decomposition seemed to be mainly a matter of the development time. We report our findings in this paper.

EXPERIMENTAL AND RESULTS

Preparation of xanthates

C_1–C_5 potassium xanthates were prepared by the following procedure. To 1 ml of alkanol were added 0.2 g of powdered potassium hydroxide. The alkaline solution was cooled to 0°C, then 0.3 ml of carbon disulphide were added dropwise with stirring. The precipitated xanthate was filtered off on a Büchner funnel and dissolved in acetone, then filtered again to separate it from solid potassium hydroxide. Finally, the solution was dried under vacuum. A xanthate mixture was obtained by the same procedure, starting with a mixture of alkanols in equal amounts.

A 100-ml volume of methanol–ethanol–water (1:40:59) mixture was examined to test the possible detection of small amounts of methanol in alcoholic beverages. The mixture was first distilled at 82°C to remove water and the first 4-ml fraction was subjected to the above procedure for preparation of the xanthates.

Chromatography of xanthates

About 10 mg of xanthates were dissolved in 2–3 ml of distilled water immediately before the analysis. Polygram CEL 400 (Macherey, Nagel & Co., Düren,

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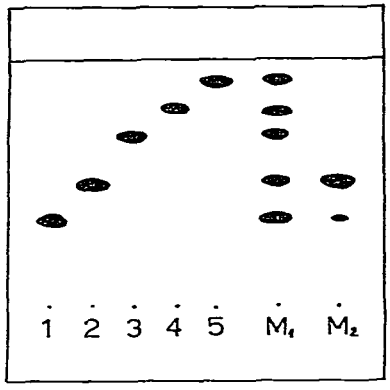

Fig. 1. Thin-layer chromatogram of xanthates of C_1–C_5 aliphatic alcohols on Polygram CEL 400. Solvent: *n*-butanol–water–ammonia solution (5:4:1). Xanthate derivatives from (1) methanol; (2) ethanol; (3) propanol; (4) butanol; (5) amyl alcohol; (M_1) mixture of equal amounts of C_1–C_5-alkanols; (M_2) first fraction from distillation of methanol–ethanol–water (1:40:59).

G.F.R.) thin layers (0.1 mm thick layers of microcrystalline cellulose) with a fluorescence indicator (254 nm) were equilibrated for 10 min before development in a chromatographic tank containing *n*-butanol–water–ammonia (5:4:1) as solvent. Samples of 10 μl were applied as streaks 1 cm apart from each other and 1 cm from the lower edge of the layer. After development the spots were detected by their dark brown fluorescence under ultraviolet light.

As shown in Fig. 1, a short run of 3 cm, developed in 25 min, yielded a complete separation of the C_1–C_5 alkanols; a longer development did not increase the resolution.

REFERENCES

1 M. Lederer and P. Summerfield, in E. Lederer and M. Lederer (Editors), *Chromatography —A Review of Principles and Applications*, Elsevier, Amsterdam, London, New York, Princeton, 2nd ed., 1957, p. 158.
2 J. Gasparič and J. Borecký, *J. Chromatogr.*, 4 (1960) 138.