

The separation and identification of the isomeric α -naphtholmonosulphonic acids by thin-layer chromatography

Some time ago we had to analyse a mixture of isomeric α -naphtholmonosulphonic acids. To our knowledge only KOLSĚC AND PERPAR¹ have described the paper chromatographic separation of these isomers (paper: Ederol 202; technique: descending; eluent: *tert.*-butanol-*n*-butanol-water, 4:3:3). Their R_f values (relative running distance values) are reported in Table I, column 1 (the R_f value 1 has arbitrarily been given to the 1,2-naphtholsulphonic acid). As the thus obtained R_f values are situated in a rather narrow range, we now wish to report a new method, based on thin-layer chromatography, and giving a better separation.

The experimental conditions are as follows:

Stationary phase: SiO₂ (Kieselgel HF₂₅₄, Merck) on glass plates of 20 × 20 cm. Thickness of the layer 0.2 mm. Activation: 1 h at 120°. Stored in a desiccator.

Eluents: either (a) methylene chloride-conc. ammonia-methanol, 80:6:30. (running time 3 h) or (b) acetonitrile-conc. ammonia, 80:6 (running time 2 h).

Technique: ascending.

The spots are applied on the thin layer as a 0.1% or a 0.01% solution in methanol. The spots are visible under U.V. light or become visible after spraying with diazotized *p*-sulphanilic acid.

TABLE I
 R_f VALUES

Isomer SO ₃ H	1	2	3
2	1.00	1.00	1.00
3	0.78	0.81	0.88
4	0.75	0.29	0.40
5	0.71	0.52	0.80
6	0.71	0.57	0.67
7	0.78	0.41	0.28
8	0.95	1.22	1.6

The R_f values (these values were chosen instead of the R_F values because the latter could not be determined, as the solvent continuously evaporated at the top of the plate during the long running periods) are represented in Table I, by column 2 (for eluent a) and column 3 (for eluent b).

A great advantage of this method, besides the gain in time, is that spots remain very small (< 0.5 cm) so that as little as 0.1 μ g of each isomer is easily detectable.

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¹ J. KOLSĚC AND M. PERPAR, *Chem. Z.*, 83 (1959) 712.

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