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Thin-layer chromatography of aminobenzoates and salicylates*

A five-component solvent system for the separation of aminobenzoic and salicylic acid derivatives by TLC has been developed. This solvent system is capable of separating a greater variety of related compounds than other published solvent systems¹⁻⁴. The relative mobilities of over 30 different compounds in the new solvent system are reported.

Non-activated Merck pre-coated SGF thin-layer plates were used and the solvent composition was: petroleum ether (b.p. 30-70°)-chloroform-methanol-glacial acetic acid-benzene (70:10:5:5:10).

TABLE I

R_F VALUES FOR VARIOUS AMINOBENZOATES

<i>Compound</i>	<i>R_F value</i>
<i>o</i> -Aminobenzoic acid methyl ester	0.74
<i>o</i> -Aminobenzoic acid isobutyl ester	0.73*
	0.66
	0.43
<i>o</i> -Aminobenzoic acid ethyl ester	0.61
<i>m</i> -Aminobenzoic acid ethyl ester	0.51
Benzoic acid	0.50
<i>o</i> -Aminobenzoic acid	0.48
4- <i>N</i> -Dimethylaminobenzoic acid	0.46
<i>p</i> -Aminobenzoic acid isopropyl ester	0.44
<i>p</i> -Aminobenzoic acid isobutyl ester	0.41
<i>p</i> -Aminobenzoic acid ethyl ester	0.37
<i>p</i> - <i>N</i> -Methylaminobenzoic acid	0.34*
	0.16
<i>p</i> -Aminobenzoic acid methyl ester	0.33
2-Hydroxy-4-aminobenzoic acid	0.33
<i>p</i> -Aminobenzoic acid	0.19
<i>m</i> -Aminobenzoic acid	0.13
2-Butoxy-4-aminobenzoic acid diethylaminoethyl ester	0.00

* When more than one spot was observed the asterisk indicates the major component.

Solutions of the compounds were dissolved in 95% ethanol and 1-2 μ l, containing 5-10 μ g of chemical, was applied to the plate. For the most part chemicals were obtained from K & K Fine Chemicals Ltd., New York. The plates were developed at room temperature until the solvent front was approximately 2 cm from the top of the 20 cm plate. Less day-to-day variation in *R_F* values was observed if chromatography was carried out under conditions of constant temperature and humidity. The compounds were located by viewing in a black box under short-wave U.V. light.

Table I gives the *R_F* values for a series of compounds related to aminobenzoic acid and Table II the *R_F* values for salicylate derivatives.

* A preliminary report of this work was presented at the 156th National Meeting of the American Chemical Society in Atlantic City, N.J., September 9, 1968.

TABLE II

R_F VALUES FOR SALICYLATES AND RELATED COMPOUNDS

<i>Compound</i>	<i>R_F value</i>
Octyl salicylate	1.00
2-Ethylhexyl salicylate	1.00
Salicylaldehyde	0.88
<i>o</i> -Salicylsalicylic acid	0.48
5-Methyl-2-hydroxybenzoic acid	0.46
Salicylic acid (<i>o</i> -hydroxybenzoic acid)	0.44
Acetylsalicylic acid	0.42
<i>m</i> -Cresotinic acid	0.41
Salicylic acid methylamide	0.37
Salicylamide	0.33
<i>m</i> -Hydroxybenzoic acid	0.19
<i>p</i> -Hydroxybenzoic acid	0.19
Salicyluric acid	0.13

This solvent system has proved useful both in drug metabolism studies, in quality control and analytical research. The use of non-activated pre-coated plates makes it a convenient method for the detection and separation of a wide variety of related compounds when present in microgram amounts.

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