

## Thin-layer chromatography of aromatic acids

Despite the fact that they should lend themselves readily to analysis by thin-layer chromatography, aromatic acids have largely been ignored in the many recent applications of this popular technique. A few scattered reports have appeared<sup>1-5</sup> mentioning aromatic acids in connection with other substances but no attempt has been made to develop a method specifically for the acids themselves.

This communication describes a simple procedure applicable to a variety of aromatic acids. Readily available materials are employed and no special apparatus beyond ordinary chromatographic equipment is required.

### Experimental

Commercially available, reagent grade solvents and aromatic acids were used without purification. Glass plates 10 × 20 cm were covered with a 0.3 mm thick coating of silicagel (Swiss type D-5 with calcium sulfate)\* using the "Camag" apparatus\*. The plates were air dried overnight and stored in a desiccator. Ether solutions containing 3-100 μg of each component of the mixture were spotted 2.5 cm from the lower end of the plate. The chromatograms were developed for 10 cm in an ascending manner using either benzene-pyridine (85:15) or *n*-heptane-glacial acetic acid (95:5) as the mobile phase. A development time of about 80 min was required in an

TABLE I  
*R<sub>B</sub>*\* VALUES OF VARIOUS AROMATIC ACIDS

Acid	<i>R<sub>B</sub></i> in solvent system**	
	A	B
<i>p</i> -Toluic	1.22	
<i>m</i> -Toluic	1.19	
<i>o</i> -Toluic	1.18	
Benzoic	1.00	1.00
Cinnamic	0.97	0.75
Phenylacetic	0.65	
<i>p</i> -Hydroxybenzoic	0.60	
<i>m</i> -Hydroxybenzoic	0.50	
<i>o</i> -Hydroxybenzoic (salicylic)	0.37	
Isophthalic	0.17	
Terephthalic	0.14	
Phthalic	0.06	

\*  $R_B = \frac{\text{migration distance of acid}}{\text{migration distance of benzoic acid}}$ ;  $R_B$  of benzoic acid in solvent system A: 0.60-0.68; in solvent system B: 0.25.

\*\* A = benzene-pyridine (85:15, v/v); B = *n*-heptane-glacial acetic acid (95:5, v/v).

unsaturated chamber. Excess solvent was removed from the plates by air drying for 3 h or by heating to 115° for 1 h. The acid spots were visualized by spraying with a 1% solution of bromocresol green in 90% alcohol (yellow spots on a green background).

\* A. H. Thomas Co., Philadelphia 5, Pa.

### Discussion

Chromatographic data for various aromatic acids are summarized in Table I. Migration rates are given as  $R_B$  values (where  $R_B$  is the migration distance of the acid divided by the migration distance of benzoic acid) rather than as  $R_F$  values. The  $R_F$  values for benzoic acid averaged about 0.65 in solvent system A and 0.25 in solvent system B. A somewhat better separation of benzoic and cinnamic acids was observed in *n*-heptane-acetic acid (solvent system B) than in benzene-pyridine. However, the latter system is more generally useful for separating a large number of acids.

Considerably sharper separations of substances with similar  $R_B$  values were obtained without solvent saturation of the chromatographic chamber. However, under conditions of saturation, development time was reduced by nearly 50 % and the slight "edge effect" was eliminated.  $R_F$  values were generally reduced by 1/3 by saturating the chamber. The values given are those obtained in an unsaturated atmosphere.

### Acknowledgements

The author is indebted to Mr. C. J. McCoy for assistance in preparing the chromatoplates and to the Esso Research and Engineering Co. for permission to report these findings.

Esso Research and Engineering Co., Linden, N.J. (U.S.A.)

J. W. FRANKENFELD

1 D. BRAUN AND H. GEENEN, *J. Chromatog.*, 7 (1962) 56.

2 M. GILLIO-TOS, S. A. PREVITERA AND A. VIMERCATI, *J. Chromatog.*, 13 (1964) 571.

3 J. W. COPIUS-PEEREBOOM AND H. W. BEEKES, *J. Chromatog.*, 14 (1964) 417.

4 G. PASTUSKA, *Z. Anal. Chem.*, 179 (1961) 355.

5 G. PASTUSKA AND H. J. PETROWITZ, *Chemiker Ztg.*, 86 (1962) 311.

Received August 21st, 1964

*J. Chromatog.*, 18 (1965) 179-180

## Auftrennung von Veratrum-Alkaloiden durch Dünnschichtchromatographie

(Herrn Prof. Dr. Ing. Dr. med. HELMUT NIEMER zum 65. Geburtstag gewidmet).

Die seit alten Zeiten schon in der Volksmedizin verwendete Veratrum-Droge ist seit 1945 als hypotensives Mittel wieder in die Therapie eingeführt worden. Da die Droge jedoch aus mehreren Alkaloiden besteht, treten demzufolge unsichere therapeutische Ergebnisse und Nebenwirkungen auf, sodass eine Methode zur schnellen Auftrennung und Bestimmung kleinerer und grösserer Mengen ihrer Inhaltsstoffe auch von pharmakologischem Interesse ist.

Besonders in Pflanzen der Liliaceen- (*Fritillaria*, *Schoenocaulon*, *Veratrum*, *Zygadenus*) und Solanaceen-Gattungen (*Lycopersicum*, *Solanum*) sind polycyclische Aminoalkohole enthalten, deren Kohlenstoffgerüst an das der Sterine erinnert, wobei die Seitenkette unter Stickstoffeinbau zu einem Heterocyclus umgewandelt ist.

*J. Chromatog.*, 18 (1965) 180-183