

Separation of chlorinated cresols and chlorinated xylenols by paper chromatography

This work arose out of studies on the chlorination of tar acids from low temperature tars. Paper chromatography has been applied to the separation and identification of isomeric cresols¹, xylenols², chlorophenols³ and chlorocresols⁴. Successful separation of chlorocresols and chloroxylenols in mixtures is now reported.

Materials

Whatman No. 1 (16 × 9 in.) filter was impregnated with methanol-formamide solution (30:100, v/v) and dried at room temperature for 15–30 min. Solutions (0.5–1 %) of each compound in acetone were spotted with a fine glass capillary (10–20 μg). Three solvent systems were used:

(A) Petroleum ether (80–100°) saturated with formic acid.

(B) Xylene saturated with formamide.

(C) The organic layer from a mixture of benzene-acetic acid-water (2:2:1 v/v).

The chlorocresols and chloroxylenols used for separation were chromatographically pure and obtained from Coalite and Chemical Products Ltd., Bolsover and Aldrich Chemical Company Inc., Wisconsin.

Detection of the spots. Phosphotungstomolybdic acid (Folin Denis reagent) was sprayed on the developed chromatogram. When exposed to ammonia, blue spots resulted.

TABLE I

R_F VALUES OF DIFFERENT CHLORINATED CRESOLS AND CHLORINATED XYLENOLS

Temperature of chromatography, $20 \pm 2^\circ$. Time of run for all solvent systems, ca. 2.5 h.

No.	Compounds	R_F values ($\times 100$) in various solvent systems			Mixtures of phenols (indicated as their No. in column 1)
		A	B	C	
1	<i>o</i> -Cresol	8	50	50	
2	6-Chloro- <i>o</i> -cresol	88	89	92	
3	4-Chloro- <i>o</i> -cresol	19	66	66	1–4
4	4,6-Dichloro- <i>o</i> -cresol	86	91	93	
5	<i>m</i> -Cresol	5	34	42	
6	6-Chloro- <i>m</i> -cresol	70	73	76	
7	4-Chloro- <i>m</i> -cresol	10	55	59	
8	4,6-Dichloro- <i>m</i> -cresol	62	84	84	5–9
9	2,4,6-Trichloro- <i>m</i> -cresol	73	90	94	
10	2,3-Dimethylphenol	20	62	67	
11	4-Chloro-2,3-dimethylphenol	26	73	79	10 and 11
12	2,5-Dimethylphenol	25	60	76	
13	4-Chloro-2,5-dimethylphenol	34	76	86	12 and 13
14	3,4-Dimethylphenol	8	44	56	
15	6-Chloro-3,4-dimethylphenol	64	79	85	14–16
16	2,6-Dichloro-3,4-dimethylphenol	79	92	94	
17	3,5-Dimethylphenol	11	59	63	17–19
18	4-Chloro-3,5-dimethylphenol	25	72	76	
19	2,4-Dichloro-3,5-dimethylphenol	78	91	92	
20	5-Ethyl-3-methylphenol	30	70	76	
21	4-Chloro-5-ethyl-3-methylphenol	34	81	89	20–22
22	2,4-Dichloro-5-ethyl-3-methylphenol	86	92	93	

Results and discussion

Table I lists the R_F values of the chlorocresols and chloroxylenols. The spots were free of tailing. 6-Chloro-*o*-cresol could not be separated from 4,6-dichloro-*o*-cresol with any of the solvent systems tried. Low R_F values were exhibited in solvent system A which has the lowest polarity. R_F values in solvents B and C were about the same. Generally the R_F values increased with increasing substitution in the nucleus; 6-chloro-*o*-cresol and 6-chloro-*m*-cresol showed comparatively high R_F values in solvent A. The R_F values for *o*-substituted compounds are higher than for the corresponding *m*-isomer, a result perhaps of the *ortho* effect⁵. Thus the $R_F \times 100$ values in solvent B were: *m*-cresol 34, *o*-cresol 50, 4-chloro-*m*-cresol 55, 6-chloro-*o*-cresol 73, 3,4-dimethylphenol 44, 6-chloro-3,4-dimethylphenol 79. The effect is more pronounced in the 2,6-disubstituted products as seen in the $R_F \times 100$ values for 4-chloro-*o*-cresol 66, 6-chloro-*o*-cresol 89, 6-chloro-3,4-dimethylphenol 79, and 2,6-dichloro-3,4-dimethylphenol 92. In these disubstituted products the relative order was generally maintained in all the three solvent systems.

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Papierchromatographische Analyse von Aerosolbestandteilen

Kennzeichnend für ein Aerosol sind seine Teilchengröße und elektrische Ladung, Teilchenkonzentration und seine chemische Zusammensetzung. Zahlreiche Untersuchungen über Aerosole sind unter vorwiegend physikalischen Aspekten durchgeführt worden, wie die der Teilchengröße, Teilchengrößenverteilung oder die der elektrischen Ladungsverhältnisse¹⁻³.

Um Kenntnis von der chemischen Struktur und dem quantitativen Verhältnis der einzelnen Komponenten eines komplexen Aerosols (Misch-aerosol) zu erhalten, ist eine chemische Analyse nach Abscheidung des Aerosols notwendig.

An Modellversuchen untersuchten wir die Möglichkeit einer unmittelbaren papierchromatographischen Analyse von homo- oder heterodispersen Misch-aerosolen, die mit den üblichen Methoden (Membranfilter, Cascade impector⁴⁻⁶) aufgefangen bzw. niedergeschlagen werden können. Es wurde von uns eine Farbstofflösung, die aus gleichen Volumina Methylenblau (12 g/l) und Fuchsin (1.7 g/l) bestand, durch einen kleinen Ultraschallvernebler (ca 20 W), der eine sehr dichte und feinverteilte Aerosolerzeugung gestattet^{7,8}, in den Aerosolzustand übergeführt. Die gebildeten

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