

Reference

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Separation of Al, Ti, In, Fe and Ga by Thin-Layer Chromatography on Cellulose

Trennung von Al, Ti, In, Fe und Ga
durch Dünnschicht-Chromatographie auf Cellulose

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A method has been worked out for the separation of Al, Ti, In, Fe and Ga on thin layers of cellulose. Some of these elements have already been separated in earlier investigations of other authors.

Experimental

Thin-Layer Plates. A homogenised suspension of microcrystalline cellulose in water (1:3.5) is spread over glass plates (20×20 or 10×20 cm) by means of a Desaga apparatus (thickness of layers 0.25 mm).

Procedure. About 1 µl of a solution of each cation or 2–3 µl of their mixture were spotted on the plates. Development was carried out over 10 cm in a Desaga chamber according to the ascending technique at room temperature. The time of development was dependent on the solvent system used and is about 4 h for propanol and butanol systems. Various systems have been investigated. The best results were obtained with those listed in Table 1. Detection was performed by spraying with a saturated solution of

Table 1. *R_f*-values of the cations investigated

No.	Solvent system	<i>R_f</i> -values				
		Al	Ti	In	Fe	Ga
1.	Acetone/HCl/H ₂ O (20:10:1)	0.20		0.85	1.0	1.0
2.	HCl conc., sat. with ether	0.04		0.58	0.98	0.98
3.	n-Propanol/HCl/H ₂ O (20:10:10)	0.36	0.41	0.56	0.85	0.99
4.	n-Butanol/HCl/H ₂ O (40:10:10)	0.12	0.20	0.57	0.85	0.99
5.	n-Butanol/HCl/H ₂ O (80:10:10)	0.05	0.12	0.32	0.66	0.92
6.	n-Butanol, sat. with 3 N HCl	0.20	0.31	0.45	0.88	0.99

alizarin in ethanol [or aqueous solution (1 g/1 l) with addition of NaOH until red-violet], exposing to NH₃ vapours and drying with a fan. The following colours were obtained: Al red, Ti red, In red-violet, Fe blue-violet, Ga red. *R_f*-values are given in the Table 1.

Discussion

R_f-values of the elements examined are in general dependent on the HCl and H₂O content of the solvent system. Most satisfactory results have been obtained with those given in Table 1, which have been selected from a great variety of organic solvents and acids tested. Greatest differences of *R_f*-values are obtained with n-butanol/HCl/H₂O (80:10:10). The spots are completely separated, compact, small and without tails. The gallium spot is narrow and elongated in the direction of the solvent front. Yet it is located still under the front so that the impurities present do not affect the Ga detection.

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N-Chloroacetamide as a Redox Reagent for the Determination of Thiourea and Its Organic Derivatives

N-Chloroacetamid als Redoxreagens zur Bestimmung
von Thioharnstoff und seinen organischen Derivaten

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Due to the increasing use of N-haloamides for organic oxidations, it is of interest to study such compounds as oxidimetric reagents and N-chloroacetamide has been found to be quite promising. The compound was prepared by the method of I. G. Farben-ind. [1], and it melted at 111°C after crystallization. It was found to be extremely soluble in water. Its solution in the presence of a small amount of chloroform (1 ml per litre of solution) does not

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