

probe eine entsprechende Ni²⁺-Menge zusetzt. — Das Verfahren wurde zur Analyse bororganischer Verbindungen nach ihrem Aufschluß mit Na₂O₂ benutzt [3].

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Separation and Detection of Cu, Pb, Bi, Cd, Hg, As, Sb and Sn by Thin-Layer Chromatography on Cellulose

Trennung und Nachweis von Cu, Pb, Bi, Cd, Hg, As, Sb und Sn durch Dünnschicht-Chromatographie auf Cellulose

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The separation and identification of the II. analytical group cations (Cu, Pb, Bi, Cd, Hg, As, Sb, Sn) has been investigated and it has been found that several solvent systems can be recommended for that purpose.

Experimental

10×15 cm glass plates are coated with a suspension of microcrystalline cellulose (for TLC, Merck) in water (1:3.5) and are dried at room temperature. Salts of the above metals are dissolved in 3 M HCl (HNO₃ in case of Pb) so that the concentrations are about 5 mg/ml.

Procedure. Apply 0.5 μ l of each cation solution and 1 μ l of their mixture on the plates and develop by the ascending technique over a distance of 10 cm in a 12×8×18 cm glass chamber at room temperature. Use one of the following solvent systems:

1. n-Butanol/3 M HCl (25:5).
2. n-Butanol/2 M HCl/acetylacetone (25:5:1).
3. n-Propanol/chloroform/6 M HCl/4 M HAc (30:5:5:5).

4. n-Butanol sat. with 4 M HAc/n-butanol/6 M HCl (30:20:10).
5. n-Butanol/chloroform/6 M HCl (40:5:5).
6. n-Butanol/chloroform/6 M HCl/acetylacetone (40:5:5:0.5).

For detection of the spots spray the plates with saturated solution of alizarin in ethanol, expose to NH₃ vapours and spray again with 0.1% dithizone solution. The following colours will be obtained (minimum amounts detectable are also given):

Cation	Cu	Pb	As	Bi
Colour	dark violet	light yellow	yellow	red-violet
Minimum amount (μ g)	$3 \cdot 10^{-5}$	$1 \cdot 10^{-1}$	$3 \cdot 10^{-1}$	$6 \cdot 10^{-3}$

Cation	Cd	Sb	Sn	Hg
Colour	reddish orange	violet intens.	orange	red-violet
Minimum amount (μ g)	$8 \cdot 10^{-5}$	$9 \cdot 10^{-3}$	$4 \cdot 10^{-3}$	$4 \cdot 10^{-2}$

R_F-values obtained with the different solvent systems are as follows:

Solvent system No.	R _F -values							
	Cu	Pb	As	Bi	Cd	Sb	Sn	Hg
1.	0.16	0.25	0.65	0.68	0.83	0.87	0.90	0.95
2.	0.12	0.18	0.58	0.67	0.75	0.78	0.82	0.89
3.	0.20	0.0	0.60	0.70	0.75	0.77	0.80	0.85
4.	0.15	0.20	0.47	0.53	0.60	0.64	0.70	0.84
5.	0.06	0.0	0.58	0.38	0.61	0.64	0.66	0.71
6.	0.06	0.0	0.60	0.42	0.67	0.69	0.73	0.75

Discussion

The cations of the IIa subgroup (Cu, Pb, Bi, Cd, Hg) and those of the IIb subgroup (As, Sb, Sn) can be separated in various solvents as already has been described in literature. Difficulties are encountered, however, if all 8 cations are present in a mixture. But, it is obvious from the above Table that satisfactory results are obtained with the proposed solvent systems provided that only small amounts of each cation are present. The spots of Bi and As are usually touching, the latter one being relatively diffuse and not intensive in colour. Large amounts of Pb usually remain at the start.

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