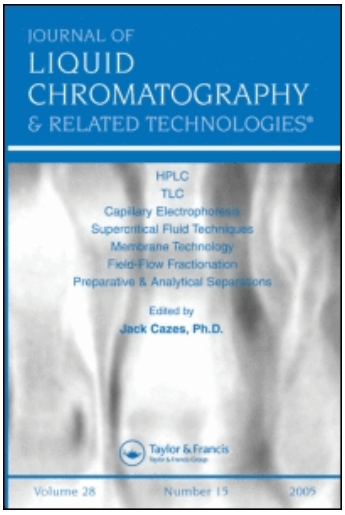

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Quantitative TLC of Toxic Elements on Inorganic Ion-Exchangers. IV. Cadmium and Lead

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QUANTITATIVE TLC OF TOXIC ELEMENTS ON INORGANIC ION-EXCHANGERS. IV. CADMIUM AND LEAD

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ABSTRACT

Thin layers of Stannic Silicate, an inorganic ion-exchanger, have been used to develop fast and selective methods for the separation of micro-quantities of Cadmium and Lead from other elements. Direct determinations of Cadmium and Lead have been made under chosen sutiable conditions by the help of Camag TLC Scanner.

INTRODUCTION

Due to high selectivity of the synthetic inorganic ion-exchangers, their use in Thin Layer Chromatography (1 - 6) has transformed this technique into a very effective method at micro-level. In continuation to our studies on Mercury (7), Selenium(8) and Thallium (9) we report here fast and selective methods for the separation and determination of Cadmium and Lead from numerous elements using the Stannic Silicate, an inorganic ion-exchanger(10).

EXPERIMENTAL

Apparatus

TLC Scanner-I, Nanomat-I, Automatic TLC Coater and a twin trough chamber were all from Camag.

Reagents

All chemicals and reagents were of Analytical grade from E. Merck or B.D.H.

Preparation of Ion-Exchange Plates

By the help of Automatic TLC coater 0.3mm thick uniform layers containing the ion-exchanger were prepared as reported earlier(6).

Cation Solutions

A standard 0.2M solution of Cadmium was prepared by dissolving 5.5423g of cadmium nitrate in 25ml demineralized water. A standard 0.05M solution of Lead was prepared by dissolving 1.6560g of lead nitrate in 100ml demineralized water. Solutions of other metal ions were prepared as described earlier(6).

Detection Reagents

For cadmium it was prepared by dissolving 0.1460g of ferric sulphate and 0.25g of α-α'-dipyridyl in 50ml demineralized water. For detection of lead a saturated solution of Sodium Rhodizonate was prepared. Detection reagents for other cations were prepared as reported earlier(6).

Procedure

Varying amounts of cadmium or lead mixed with different cations were applied on the ion-exchanger plates by the help of Camag Nanomat-I. In case of separation of cadmium the plates were developed in 1M NH_4Br solution upto a distance of 12 cm. After the plate was dried in air, it was sprayed first with α-α'dipyridyl

solution and then with 10% KI solution. Cadmium appears as pink spots. In case of separation of lead the plates were developed in 0.5M Lactic Acid solution upto a distance of 12 cm. After the plate was dried in air it was first sprayed with solution of sodium Rhodizonate and then exposed to HCl vapours. Lead appears as violet colored spots on a white background.

Quantitative Determinations

The Camag TLC Scanner-I was used for direct determinations of separated amounts of Cadmium on the plates under chosen conditions; wave length 530 nm ; photomultiplier sensitivity 6 ; recorder voltage range 200 mV ; slit width 6 nm ; scan speed 2mm/sec ; recorder paper speed 100mm/min. While for the determination of the separated amounts of Lead on the plates the chosen conditions were; wave length 550 nm ; photomultiplier sensitivity 4 ; recorder voltage range 100 mV ; slit width 6 nm ; scan speed 2mm/sec ; recorder paper speed 100mm/min.

RESULTS AND DISCUSSION

The quantitative results recorded in Table-I show that fast separation of cadmium from other metal ions has been achieved on thin layers of stannic silicate in 1M NH_4Br solution. Here cadmium forms the anionic complex $CdBr_4^{-2}$(11) which is not adsorbed by the cation-exchanger and results in high Rf values for cadmium leaving other elements behind resulting in clear separations. Direct determination of separated cadmium on the plate by the help of TLC Scanner provides a speedy method for the selective determination of Cadmium(4). Results of Separation of Lead from other elements are recorded in Table-II. Here lead is selectively adsorbed on the ion-exchanger while other elements move faster due to the formation of lactate complexes of varying strength(12). A fast method for the separation of lead is developed here and the direct determination of micro-quantities has been made satisfactorily, which is an improvement over the earlier reported method(4).

TABLE-I. Quantitative Separation of Cadmium in Binary Mixtures.
Solvent System : 1M NH_4Br ; Time of Development: 12 min.

S.No.	Mixture Separated	Amount of other element added (/ug)	Amount of Cadmium Taken (/ug)	Found* (/ug)	Difference (/ug)
1.	As(III)-Cd(II)	As(1.87)	11.24	11.25	+0.01
2.	Tl(I)-Cd(II)	Tl(5.19)	11.24	11.35	+0.11
3.	Cu(II)-Cd(II)	Cu(1.59)	11.24	11.75	+0.51
4.	UO_2(II)-Cd(II)	UO_2(6.75)	11.24	11.65	+0.41
5.	Co(II)-Cd(II)	Co(1.47)	11.24	11.90	+0.66
6.	Sb(III)-Cd(II)	Sb(3.04)	11.24	13.25	+2.01
7.	Fe(III)-Cd(II)	Fe(1.39)	22.48	20.25	-2.23
8.	Zn(II)-Cd(II)	Zn(1.63)	22.48	22.43	-0.05

* Average of four determinations.

TABLE-II. Quantitative Separation of Lead in Binary Mixtures.
Solvent System : 0.5M Lactic Acid ;Time of Development: 21 min.

S.No.	Mixture Separated	Amount of other element added (μg)	Amount of Lead Taken (μg)	Amount of Lead Found* (μg)	Difference (μg)
1.	Pb(II)-Al(III)	Al(0.67)	5.20	5.20	0.00
2.	Pb(II)-Cd(II)	Cd(2.81)	5.20	5.23	+0.03
3.	Pb(II)-Hg(II)	Hg(5.01)	5.20	4.42	-0.78
4.	Pb(II)-Ba(II)	Ba(3.43)	5.20	4.45	-0.75
5.	Pb(II)-Be(II)	Be(4.44)	10.40	10.67	+0.27
6.	Pb(II)-Ni(II)	Ni(1.47)	10.40	11.19	+0.79
7.	Pb(II)-Mg(II)	Mg(0.61)	10.40	10.42	+0.02
8.	Pb(II)-Mn(II)	Mn(1.37)	10.40	10.50	+0.10

* Average of four determinations.

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