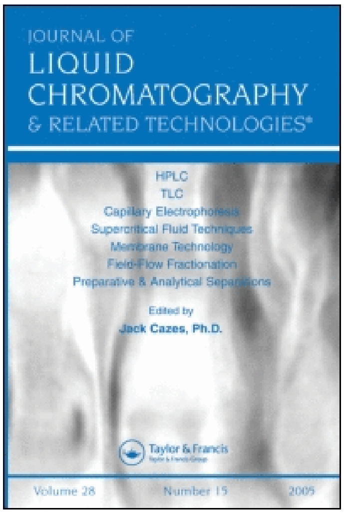

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Thin-Layer Chromatographic Separation of Copper and Mercury Chelates

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Thin-Layer Chromatographic Separation of Copper and Mercury Chelates

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Thin-layer chromatographic separation of metal ions has become immensely popular in analytical chemistry in view of its speed, selectivity and applicability at tracer level. Quinoline-2-aldehyde thiosemicarbazone[1,2] was found to form yellow coloured chelates with metal ions such as copper, nickel, zinc, mercury, iron and lead at pH 7.5 and extract into chloroform. This, however reduces the sensitivity of the reagent as a chromogenic agent but the chromatographic run of the chelates on a thin layer of silica gel coated on glass plate increases the specificity and selectivity of the reagent. In this article we propose a method for separation of copper and mercury chelates from binary mixtures containing chelates of zinc, lead, iron and nickel. The method is applicable for analysis of synthetic mixtures and standard samples.

EXPERIMENTAL

A slurry of silica gel G (BDH, AR) and binder was used for coating glass plates (20 x 5 cms) and ascending chromatography was performed in 21.5 x 5.2 cms glass jars saturated with vapours of the solvent. pH was measured on Philips pH meter.

Metal solutions containing 100 µg/ml of metal ions were prepared by dissolving guaranteed grade copper sulphate, zinc sulphate, lead chloride, ferrous ammonium sulphate, mercuric chloride and nickel sulphate salts.

Quinoline-2-aldehyde thiosemicarbazone (QAT) was synthesized as earlier reported[1] and 0.1 % w/v solution in 1:1 DMF : H_2O was employed for forming chelates.

Solvents used for development were purified and distilled.

Preparation of Metal-QAT Chelates and TLC Separation Procedure :

Take an aliquot (20 ml) of solution containing 200 to 300 µg of metal ions, add 4 ml of 0.1 % QAT solution and adjust the pH to 7.5 with diluted $NaOH/H_2SO_4$ solution, in a total volume of 25 ml. Transfer the solution into a 100 ml separating funnel and extract twice each for 20 sec with 5 ml of chloroform. Collect the chloroform extract, dry it with anhydrous sodium sulphate and spot it on silica coated glass plates with micro capillaries. After drying the spots, the chromatograms were developed in jars saturated with vapors of solvent mixtures. The solvent's were allowed to ascend 10 cm from the starting line on the plate and the required time was noted. Developed plates were dried and cations were detected with appropriate spraying agents to find out R_f values.

RESULTS AND DISCUSSION

Several solvent mixtures were tried for the development of individual metal chelates and R_f values are noted. The most suitable solvent mixtures, the corresponding R_f values and the average time required for the development are reported in Table 1. Conditions were developed for separation of copper and mercury chelates from other metal chelates in a binary mixture. Copper is separated from zinc, lead and iron using ethanol:acetone (1:1) solvent mixture whereas methanol:ethanol (1:3) solvent mixture was employed for separation of copper from mercury and nickel. The results of separation are given in Table 2. Mercury was similarly separated from zinc, lead, nickel and iron using either ethanol:acetone, ethanol:benzene or methanol:ethanol solvent mixtures. The mercury separation are reported in Table 3.

Detection of Copper and Zinc in brass :

About 1.03 g of brass is dissolved in nitric acid, evaporated to dryness, extracted with 0.5 M H_2SO_4 and diluted to 250 ml. An aliquot portion of the brass solution was taken and further procedure for chelate formation and TLC separation was followed as described above. Copper and zinc spots were located on the glass plates with appropriate spraying agents.

Detection of Copper and Lead in Gun Metal :

About 1 g of gun metal was dissolved in nitric acid. Tin was removed as metastannic acid and filtrate was diluted

Table 1 : R_f values (x 100) of Metal-QAT chelates in different solvent mixtures

S.No.	Solvent system	Copper	Nickel	Mercury	Iron	Average time for development (min)
1	Ethanol:acetone (1:1)	81	80	90	20	25
2	Ethanol:chloroform (1:1)	41	57	81	30	31
3	Ethanol:chloroform (1:3)	30	70	79	67	36
4	Methanol:ethylacetate (1:1)	23	87	85	16	25
5	Methanol:ethanol (1:3)	10	74	90	20	35
6	Ethanol:benzene (1:1)	51	79	90	67	31
7	Ethanol:Ethylmethylketone (1:)	77	79	90	62	30

Zinc and lead chelates remain at the origin.

Table 2 : Separation of copper chelates from binary mixture

	Mixture	Solvent mixture used for development	Time (min) required for development	Spraying agents used for detection	Colour of spots	R_f x 100
1	Cu, Zn	Ethanol:acetone (1:1)	24	PCAF + RA for both Cu and Zn	Cu - G Zn - R	Cu - 75.0 Zn - 0.0
2	Cu, Pb	Ethanol:acetone (1:1)	24	dithizone for Pb	Cu - G Pb - R	Cu - 75.0 Pb - 0.0
3	Cu, Hg	Methanol:ethanol (1:3)	42	dithizone for Hg	Cu - G Hg - OR	Cu - 10.0 Hg - 90.0
4	Cu, Fe	Ethanol:acetone (1:1)	24	colour of the chelate itself	Cu - G Fe - b	Cu - 73.0 Fe - 12.0
5	Cu, Ni	Methanol:ethanol (1:3)	34	Ni-DMG	Cu - G Ni - P	Cu - 11.0 Ni - 76.0

PCAF - Trisodium Pentacyanoammino-ferrate

RA - Rubeanic Acid

Table 3 : Separation of Mercury chelates from binary mixture

	Mixture	Solvent mixture used for development	Time (min) required for development	Spraying agents used for detection	Colour of spots	R_f x 100
1	Hg, Zn	Ethanol:acetone (1:1)	24	Dithizone PCAF + RA	Hg - OR Zn - R	Hg - 90.0 Zn - 0.0
2	Hg, Zn	Ethanol:benzene (1:1)	31	Dithizone PCAF + RA	Hg - OR Zn - R	Hg - 93.0 Zn - 0.0
3	Hg, Pb	Ethanol:acetone (1:1)	24	Dithizone for both	Hg - OR Pb - R	Hg - 90.0 Pb - 0.0
4	Hg, Pb	Ethanol:benzene (1:1)	31	Dithizone for both	Hg - OR Pb - R	Hg - 93.0 Pb - 0.0
5	Hg, Ni	Ethanol:acetone (1:1)	24	Ni-DMG	Hg - OR Ni - P	Hg - 90.0 Ni - 73.0
6	Hg, Ni	Ethanol:benzene (1:1)	31	"		Hg - 93.0 Ni - 65.0
7	Hg, Fe	Ethanol:acetone (1:1)	24	Colour of chelate itself	Hg - OR Fe - brown	Hg - 90.0 Fe - 12.0
8	Hg, Fe	Methanol:ethanol (1:3)	35	"	Hg - OR Fe - brown	Hg - 90.0 Fe - 13.0

to 250 ml. An aliquot portion of solution was taken for separation of copper and lead as described in the general procedure.

SUMMARY

A method is proposed for TLC separation of metal chelates formed by copper and mercury with quinoline-2-aldehyde thiosemicarbazone. The chelates formed at pH 7.5 are extracted into chloroform and chloroform extract is developed on a thin-layer of silica gel using different solvent mixtures. Optimum conditions are developed for separation of copper and mercury from iron, nickel, lead and zinc in a binary mixture. The results of separation and analysis of some standard samples are reported.

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