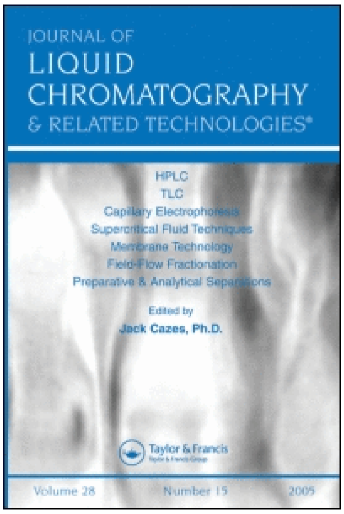


Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Determination of Microgram Amounts of Copper by Peak Paper Chromatography

Bhajan Singh[a]; Pritam Singh Thind[a]
[a] Department of Chemistry, Guru Nanak Dev University, Amritsar, Punjab, India

To cite this Article Singh, Bhajan and Thind, Pritam Singh(1981) 'Determination of Microgram Amounts of Copper by Peak Paper Chromatography', Journal of Liquid Chromatography & Related Technologies, 4: 2, 251 — 257
To link to this Article: DOI: 10.1080/01483918108064813
URL: http://dx.doi.org/10.1080/01483918108064813

JOURNAL OF LIQUID CHROMATOGRAPHY, 4(2), 251-257 (1981)

DETERMINATION OF MICROGRAM AMOUNTS OF COPPER BY PEAK PAPER CHROMATOGRAPHY

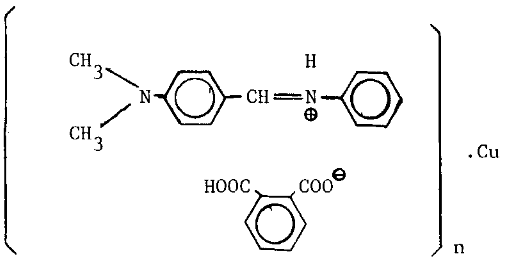
Bhajan Singh And Pritam Singh Thind
Department of Chemistry,
Guru Nanak Dev University,
Amritsar, Punjab (India).

ABSTRACT

A chromatographic method has been developed for determining 1.00 to 6.00 µg l^{-1} of copper. The test solution (1 µl) was applied to paper impregnated with 0.1% phthalate salt of aniline and p-dimethylaminobenzaldehyde (p-DAB) based schiff's base solution and the chromatogram was developed with methanol, then immersed in aqueous 5% solution of potassium ferrocyanide for 35. The heights of the reddish brown peak shaped zones produced were related to concentration of copper. Equal amounts of silver, nickel and cobalt did not interfere. The error was $\leqslant \pm 0.20$ with standard deviation 0.105.

INTRODUCTION

Peak paper chromatography is a useful tool for the quantitative determination of microgram amounts of ions especially when one has to carry out a large number of estimations on small volume of samples (1-4). Ions are determined by peak chromatography on paper which has been impregnated with a sparingly soluble compound containing a precipitant ion for the test ion, the compound formed between the two being even less soluble than the impregnant itself. Microgram amounts of sulfate (5) and chloride (6) has been determined using barium chloride and mercury nitrate as precipitant respectively. Bismuthol-2 impregnated papers have been used for the determination of tellurium in presence of cadmium (7). Copper forms with phthalate salt of aniline and p-dimethylaminobenzaldehyde based schiff's base a bright green complex which is sparingly soluble in methanol.



In the present report we used the formation of this complex as the basis of a method for determining microgram amounts of copper in presence of silver, nickel and cobalt by peak paper chromatography.

EXPERIMENTAL

Reagents and Apparatus

Standard Stock solutions: The standard solution of copper was prepared by dissolving 1.0 g of copper metal of grade BDH (AR) in conc. HNO_3 on heating. Nitric acid was removed by evaporation until the moist-salt was reached, finally the residue was dissolved in water and transferred to a 100ml volumetric flask. The concentration of this solution was 10 mg cu/ml. Weaker solutions were prepared by diluting this stock solution.

Aqueous silver, nickel and cobalt salt solutions were prepared from BDH (AR) silver nitrate, nickel sulphate and cobalt sulfate respectively. The stock solution contained 10 mg of metal/ml. Weaker solutions were prepared by diluting the stock solution. Synthetic mixture of Cu-Ag, Cu-Ni and Cu-Co were prepared from solution of appropriate concentrations.

Schiff's base solution: The schiff's base was obtained by mixing equimolar solution of aniline phtalate and p-DAB (1:1) and finally recrystallized from methanol. 0.1% solution of this schiff's base was prepared in methanol and used for impregnation.

Preparation of papers: Chromatography was performed on Whatman No.1 paper strips of size 20x3 cm using 25x5 cm glass jars. Paper strips were impregnated in 0.1% solution of schiff's base for 3 to 5 seconds and then dried at room temperature.

Detection reagent: A 5% solution of potassium ferrocyanide was used for locating the peak of copper on paper.

Solvent used for development: Double distilled methanol was used as developing solvent.

Procedure

Determination of Copper: A sample of test solution (1 µl) was applied by means of a microsyringe to a impregnated paper strip. The chromatograpms were developed by ascending technique as usual. The ascent of solvent in each case was 15 cms., then the strips were dried at room temperature for 15 minutes and finally impregnated with potassium ferrocyanide to locate the peaks. The height of the peaks was measured in millimeters.

Determination of copper in presence of silver nickel and cobalt: Synthetic mixtures containing equal amounts (1 to 5 µg) of Cu-Ag, Cu-Ni and Cu-Co were applied by means of microsyringe to a impregnated paper strip. Rest of the procedure was same as in case of copper.

RESULTS AND DISCUSSION

The method is based on the fact that a circular spot of the precipitate is formed when test solution is applied to the paper strip. On ascending via capillaries in the paper, methanol elutes from the spot amount of test ion in excess of the stoichiometric amount of the precipitant on the area occupied by the spot. The excess of test ion reacts with fresh portions of impregnant along the methanol ascent line leaving behind on the paper a trace in the form of a precipitate peak with a reasonably correct shape. The curves relating copper concentration to the height of the peak formed, calculated by the least square method, are linear for the copper concentraion. Table-1 gives the experimental and calculated data. The error and standard deviation was formed to be ±0.20 µg and 0.105 respectively.

Silver, nickel and cobalt also forms complexes with schiff's base whose solubility products are less than that of the copper complex with the same reagent. Experimental data showed that silver, nickel and cobalt taken in 1:1 ratio and in small excess with respect to copper (up to 5 µg) does not interfere with copper determination.

TABLE I. Experimental And Calculated Data For The Determination Of Copper In Chromatograms (n = 10).

Concentration of precipitant (%)	Mean Cu peak height (mm), y_i	Cu concentration in the drop applied (ug)	Amount of Cu determination (ug)	Error (ug)	Standard deviation (ug)
0.1	16.2	1.00	0.88	-0.12	
0.1	22.5	1.50	1.61	+0.11	
0.1	29.1	2.00	2.13	+0.13	
0.1	42.6	3.00	2.94	-0.06	0.105
0.1	51.6	4.00	3.88	-0.12	
0.1	57.2	5.00	5.00	0.00	
0.1	62.8	6.00	6.06	+0.06	

We analyzed mixtures (1:1) of Cu-Ag, Cu-Ni and Cu-Co at levels 1.0 to 5.0 µg in a drop of solution (1 µl). With this concentration range there is a linear relationship between copper concentration and the height of the peaks formed. The respective results are given in Table II. The error in determining copper in presence of silver, nickel and cobalt lies within the same limits as where these are absent.

To assess the accuracy of determination of paper impregnated with 0.1% solution of schiff's base we performed seven replicate determinations of copper solution of known concentration (Table III). After the chroma-

TABLE II. Seven Replicate Determinations of Copper (4.0 μg Cu Taken)

Cu peak height (mm), $\bar{y}_i$	Cu detected (μg) Xi calc.	Error (μg)	Standard deviation (μg)
40.00	3.84	-0.16	
40.0	3.84	-0.16	
43.0	4.12	+0.12	
41.0	3.93	-0.07	0.245
44.5	4.27	+0.27	
45.5	4.36	∓0.36	
44.0	4.22	+0.22	
Mean 41.14	Mean 4.14		

togram had been developed the copper concentration was found from a calibration curve plotted within the coordinates:

Copper content (μg) verses peak height (mm).

The calibration curve calculated by the least square method is of the form

$$X = \frac{y_i + 564.55}{767.13}$$

Thus this method which is simple and rapid to carry out, can be used to analyze small amounts of material (a few μg). to work with small volumes of solution (a few ml) and to carry out a large number of determinations on such amounts.

TABLE III Experimental And Calculated Data For The Determination Of Copper In The Presence Of Equal Amounts Of Silver, Nickel And Cobalt.

Impurity added, µg	Amount of Cu applied µg, xi	Mean peak height, mm, yi	Yi	Amount of Cu determined µg	Error µg
1.0 Ag(I)	1.0	14.5	14.5	1.02	0.02
2.0 Ag(I)	2.0	29.7	31.1	1.91	0.09
3.0 Ag(I)	3.0	42.5	41.7	3.06	0.06
4.0 Ag(I)	4.0	49.0	50.0	3.92	0.08
5.0 Ag(I)	5.0	56.6	57.2	4.95	0.05
1.0 Ni(II)	1.0	16.4	14.2	1.15	0.15
2.0 Ni(II)	2.0	28.3	31.1	1.82	0.18
3.0 Ni(II)	3.0	42.3	41.7	3.04	0.04
4.0 Ni(II)	4.0	51.1	50.0	4.09	0.09
5.0 Ni(II)	5.0	57.1	57.2	4.99	0.01
1.0 Co(II)	1.0	17.2	14.2	1.21	0.21
2.0 Co(II)	2.0	29.5	31.1	1.90	0.10
3.0 Co(II)	3.0	42.2	41.7	3.04	0.04
4.0 Co(II)	4.0	52.1	50.0	4.17	0.17
5.0 Co(II)	5.0	57.1	57.2	4.99	0.01

Standard deviations for the determination of copper in presence of Ag(I), Ni(II) and Co(II) are 0.07, 0.13 and 0.145 respectively.

ACKNOWLEDGEMENT

The authors thank Prof. S.S. Sandhu for providing research facilities.

REFERENCES

1. V.B. Aleskovskii and V.N. Aleskovakaya, Quantitative Chromatography of Ions by the Peak Method. Scientific Proceedings of the Lensovet Leningrad Technological Institute (In Russian) No. 65, Leningrad (1972).

2. V.N. Aleskovaskaya and V.B. Aleskovakii, Zh. Analiticheskoi Khim., 24 2113 (1968).

3 E.S. Biochinova and V.B. Aleskovskii, Tr. Lenigr. Technol. In-ta Imbensovet., No. 48, 94 (1958).

4. Kh.Yu. Kokk and V.B. Abskovskii, Izv. Vyssh. Ucheb. Zavedemi. Ser. Khimiya, Khim. Tekhnol., 6, 564 (1964).

5. V.N. Aleskovskaya, V.V. Bardin, A.A. Mokhov, L.N. Plyushcheva, K.P. Sidorkina and V.A. Shichko, Zh. Anal. Khim, 32(11), 2182 (1977).

6. V.V. Bardin, A.A. Kokhov, V.A. Shickko, L.N. Plyuscheva and K.P. Sidorkina, Zh. Anal. Khim., 32(11), 2288 (1977).

7. R.A. Pasechnova, A.A. Mokhov and BF. Yudin, Zh. Anal. Khim, 29(11), 2292 (1974).