

Paper Chromatographic Separation and Determination of Copper and Aluminium in Aluminium-Bronze Alloy

By KAILASH NATH MUNSHI and ARUN K. DEY

Summary

Paper chromatographic separation of copper and aluminium in aluminium-bronze alloy has been achieved by ascending filter paper strip method, the solvent employed being a mixture of 75% n-butanol, 15% concentrated hydrochloric acid and 10% of water. The separated metal ions were extracted and quantitatively determined colorimetrically using sulpho-dichlorohydroxy-dimethyl fuchson dicarboxylic acid (Chrome Azurol S) as a colorimetric reagent. The results of analysis have been found to be satisfactory.

A large amount of work has been reported from these laboratories¹⁻³) on the separation of inorganic cations by filter paper chromatography. Also some new micro-analytical reagents have been developed by DEY et al⁴) for the colorimetric determination of metals when present in small quantities in aqueous solution. In the present work it has been attempted to utilise these findings for the separation by chromatography and subsequent colorimetric determination of copper and aluminium in a sample of aluminium-bronze alloy, using sulpho-dichlorohydroxy-dimethyl-fuchson dicarboxylic acid (Chrome Azurol S) as a reagent.

Experimental

Materials

Metal solution. Solutions of cupric sulphate and aluminium sulphate were prepared using BDH Analar grade chemicals and standardised as usual.

Colorimetric reagent. A standard solution of sulpho-dichlorohydroxy-dimethyl fuchson dicarboxylic acid (Chrome Azurol S) B. D. H. indicator trisodium salt, (Colour Index 723) was prepared by dissolving in doubly distilled carbon-dioxide free water. Fresh solutions of the reagent were used in all the experiments.

¹) E. J. SINGH and A. K. DEY, *Anal. Chim. Acta.* **24**, 444 (1961).

²) E. J. SINGH and A. K. DEY, *Anal. Chim. Acta.* **25**, 57 (1961).

³) E. J. SINGH and A. K. DEY, *J. Chromatog.* **3**, 146 (1960).

⁴) S. C. SRIVASTAVA, R. L. SETH, S. N. SINHA, S. P. SANGAL and A. K. DEY, XVIIIth International Congress of Pure and applied Chemistry, Montreal, 1961, Paper No. C 2-16.

Alloy solution. A known weight of the alloy was dissolved in concentrated nitric acid and converted to metal sulphates by evaporating to fumes with concentrated sulphuric acid.

Solvent. The solvent solution was a mixture of 75% n-butanol, 15% concentrated hydrochloric acid and 10% water. These concentrations were arrived at, by trial experiments and were found to be satisfactory.

In earlier trials, dioxan, different percentages of aqueous ethanol, with or without mineral acid, and pyridine with ammonia were tried as solvents, but were found to be unsuitable.

Apparatus

Colorimetric measurements. A KLETT-SUMMERSON photoelectric colorimeter with suitable standard KLETT-filters was employed for the colorimetric measurements. The solutions were taken in KLETT test tubes of a uniform diameter of 1 cm.

pH measurements. Hydrogen ion concentration measurements were made with Leeds and Northrup line operated pH indicator, using glass and calomel electrodes, supplied with the instrument. The meter was calibrated from time to time with Leeds Northrup standard buffer of pH 6.8.

Chromatographic Separation

Ascending filter paper strip method of GAGE, DOUGLASS and WENDER⁵⁾ was used for the chromatographic separation. 2.5 cm. wide strips of Whatman filter paper No. 1 was used. A known volume of the solution (measured with a micro pipette) was spotted, 3 cm. above the base of the strips, the end of which dipped into a petri dish containing the solvent. Two counter strips, spotted with the same volume and same concentration of alloy solution, were run for three hours, enclosed within a jar 40 cm. long, 18 cm. diameter, covered with two halved ground glass to hold the paper, superimposed by ordinary glass cover.

The chromatograms were obtained at 30°C using different volumes of alloy solution in different experiments. The chromatograms were dried at 70°C in a closed oven to avoid the effect of the atmospheric humidity which was 75% during the experiments.

Extraction

For the extraction of the migrated metal ions into the solution, one of the two counter strips, was developed by the proper developing reagent to find out the exact position of the migrated metal ion and the second strip was cut into two portions, according to the position of two metal ions on the strip. The metals from the strip were extracted separately by boiling water acidified with hydrochloric acid. The process of extraction was repeated three times to ensure complete extraction. The extracts were collected in 50 ml. flasks. The amounts of copper and aluminium extracted from the chromatograms were determined quantitatively by colorimetric method.

Colorimetric determination of extracted copper

To the extracted solution of copper, 20 ml. of 5×10^{-4} M, Chrome Azurol S solution was added and the total volume was raised upto 50 ml. by the addition of water. The pH

⁵⁾ TH. B. GAGE, C. D. DOUGLASS and S. H. WENDER, J. chem. Educat. **27**, 159 (1950).

was adjusted at 6.4 in each case. The mixtures were allowed to stand for 15 minutes to attain equilibrium. The colour intensity was measured with the colorimeter with a green Klett filter No. 55 (transmission 520–600 $m\mu$). From these readings the quantity of the copper was read from the calibration curve for copper already plotted with the same reagent under identical conditions.

Colorimetric determination of extracted Aluminium

To the extracted solutions of aluminium from the chromatograms, 20 ml. of $2.5 \times 10^{-4} M$ solution of Chrome Azurol S was added and the total volume was raised up to 50 ml. The pH of the mixture was adjusted to 6.2. The colour intensity was measured using a green Klett filter No. 54 (transmission 520–580 $m\mu$). The quantity of aluminium present in the alloy solutions was known by comparing the colorimetric readings with the calibration curve already plotted.

The following table shows some of the results out of a large number of experiments using different volumes of the alloy solution, used for spotting.

Table 1
Quantitative analysis of Aluminium
Bronze Alloy

μ_g of metals in the alloy solution			
Copper		Aluminium	
Present	found	Present	found
30.93	32.50	3.47	3.32
61.86	64.40	6.94	6.76
92.79	97.20	10.41	9.91
123.72	120.96	13.88	13.25
154.65	152.20	17.35	16.90
185.58	187.40	20.84	20.52
216.51	207.90	24.29	23.48
247.44	236.46	27.76	26.94

The composition of alloy was found to be Cu 88.7%, Al 9.7% (actual Cu 89.9%, Al 10.1%) showing that the results obtained is satisfactory within permissible limits of experimental error.

R_L and R_T values of metal ions:

The R_L and R_T values of ions were measured relative to the solvent front in different concentrations of alloy solution, and were found to show negligible changes with concentration. The values are shown below:

Metal ion	R_L value	R_T value
Copper	0.25	0.10
Aluminium	1.00	0.80

The authors thank the Council of Scientific and Industrial Research, India, for supporting the work and for the award of a Research Fellowship to one of them (K. N. M.).

Allahabad/India, Chemical Laboratories, University of Allahabad.

Bei der Redaktion eingegangen am 23. März 1962.

Verantwortlich

für die Schriftleitung: Prof. Dr.-Ing. Dr. h. c. E. Leibnitz, Leipzig O 5, Permoserstraße 15;
für den Anzeigenteil: DEWAG-Werbung Leipzig, Leipzig C 1, Friedrich-Ebert-Str. 110, Ruf 78 51.
Z. Z. gilt Anzeigenpreisliste 4; Verlag: Johann Ambrosius Barth, Leipzig C 1, Salomonstraße 18B;
Fernruf 27 681 und 27 682, ZLN 5065

Printed in Germany

△ Druck: Paul Dünnhaupt, Köthen (IV/5/1) L 163/62