

Micro Determination of Lanthanum (III) Using p-nitrobenzene Azochromotropic Acid (Chromotrope 2B) as a Chromogenic Reagent

By SATENDRA P. SANGAL

Abstract

p-nitrobenzene azochromotropic acid (Chromotrope 2 B) forms a 1:1 reddish violet chelate (λ_{max} 540 m μ) with lanthanum(III) at pH 6.0. The value of stability constant expressed as $\log K = 4.5$ and 4.9 as determined by two different methods. The colour of the chelate is stable over a wide range of temperature (0–100°) between pH 4.0 and 7.0. The system obeys BEER's law over a range of 0.34 p. p. m. to 16.7 p. p. m. of lanthanum.

p-nitrobenzene azochromotropic acid (Chromotrope 2 B) has been used as a chromogenic reagent for thorium¹), copper²), lanthanum³) and the rare earths. The composition of lanthanum(III)-Chromotrope 2 B chelate has been found to be La(Chromotrope 2 B) at pH 6.0. The value of $\log K$ (stability constant) as determined by two different methods are 4.5 and 4.9.

This communication records our observations on the spectrophotometric determination of lanthanum(III) with Chromotrope 2 B in micro amounts.

Experimental

Materials

Stock solutions of p-nitrobenzene azochromotropic acid (disodium salt, Chromotrope 2B) BDH and lanthanum chloride (JOHNSONS and MATTHEY sample of lanthanum oxide dissolved in concentrated hydrochloric acid) were prepared and standardised by their usual methods.

Instruments

A Unicam SP 500 spectrophotometer was used for the spectrophotometric measurements. 1 cm. thickness of the solutions were employed using glass cells supplied with the instrument.

¹) S. K. BANERJI and A. K. DEY, J. Ind. Chem. Soc. **38**, 139 (1961).

²) S. P. SANGAL and A. K. DEY, Indian J. Chem. **1**, 870 (1963).

³) S. P. SANGAL, S. C. SRIVASTAVA and A. K. DEY, J. Ind. Chem. Soc. **40**, 275 (1963).

For the colorimetric measurements a KLETT SUMMERSON photoelectric colorimeter (test tube model) with a KLETT filter No. 56 was used.

pH measurements were done with a LEEDS and NORTHRUP direct reading pH indicator.

All the experiments were carried out in a thermostatically controlled air conditioned room maintained at $25 \pm 2^\circ$.

Results and Discussions

Rate of colour formation

The colour formation was found to be instantaneous but the solutions were kept for 15 minutes before measurements for attaining equilibrium.

Influence of temperature on the colour

A mixture containing $2.0 \cdot 10^{-4}$ M Chromotrope 2 B and $1.33 \cdot 10^{-4}$ M lanthanum chloride when kept for 48 hours at room temperature retained its absorbance value, i. e., 0.800 at 600 m μ . The same mixture was when heated to different temperatures it was found that there was no change in the colour intensity from 5° to 100° .

Influence of pH on the colour intensity

The effect of pH on the colour intensity of the chelate was studied and it was found that the colour is stable between pH 4.0 and 7.0 (Table 1).

Table 1

Influence of pH on the colour intensity of the chelate
Concentration of Chromotrope 2 B = $2.0 \cdot 10^{-4}$ M; Concentration of lanthanum chloride = $1.33 \cdot 10^{-4}$ M; wave length of observations 600 m μ .

pH	2.0	3.0	4.0	5.0	6.0	7.0	8.0
Absorbance	0.350	0.350	0.800	0.800	0.800	0.800	0.750

Adherence to BEER's Law

Several mixtures containing (1, 2, 3, . . . 9 ml) of lanthanum chloride and 10 ml of Chromotrope 2 B were prepared and their pH was adjusted to 6.0. The total volume was kept 25 ml in each case and their absorbance readings were noted with a Unicam SP 500 spectrophotometer at 600 m μ . It was found that the system adheres to BEER's law from 0.34 p. p. m. to 16.7 p. p. m. of lanthanum.

Effect of added foreign ions

The influence of various cations and anions was studied with a KLETT SUMMERSON photoelectric colorimeter with KLETT filter No. 56 and their tolerance limits were calculated. It was found that the following cations and

anions interfere at all concentrations: copper, scandium, yttrium, lanthanum and rare earths, titanium, zirconium, hafnium and thorium, phosphate, acetate, oxalate, tartrate, citrate, borate and carbonate. The following ions were found to be tolerated in large excess: sulphite, sulphate, thiosulphate, chloride, bromide, chlorate, bromate, lithium, sodium, potassium, beryllium, calcium, strontium, barium, magnesium, aluminium, indium, tin(IV), lead, arsenic(III), antimony(III), bismuth, silver, gold, zinc, cadmium, mercury, iron(III), cobalt, nickel, ruthenium, rhodium, palladium, platinum, and other platinum metals.

Sensitivity

The sensitivity as defined by SANDELL⁴⁾ is 0.139 r/cm² and practical 1.39 r/cm².

The author is thankful to his teacher Dr. A. K. DEY for his guidance in this work and to the Council of Scientific and Industrial Research for an award of a fellowship.

⁴⁾ E. B. SANDELL, Colorimetric determination of traces of metals, 2nd Edit. Inter. Sci., New York (1928).

Allahabad (India), Chemical Laboratories, University of Allahabad.

Bei der Redaktion eingegangen am 17. September 1963.

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 7851. Z. Z. gilt Anzeigenpreisliste 4; Verlag Johann Ambrosius Barth, Leipzig C 1, Salomonstraße 18B; Fernruf 27681 und 27682. Veröffentlicht unter der Lizenz-Nr. 1395 des Presseamtes beim Vorsitzenden des Ministerrates der DDR

Printed in Germany



Druck: Paul Dünnhaupt, Köthen (IV/5/1) L 186/64