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Short Communication

Spectrophotometric determination of rhodium(III) with {2-[di-(2-pyridyl) methylidenehydrazino] quinoline}

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Abstract

A simple, rapid and selective procedure for spectrophotometric determination of Rh(III) has been developed. Rh(III) forms a pink coloured complex with {2-[di(2-pyridyl)methylidenehydrazino]-quinoline} in the pH range 1·0–3·0 (λ_{max} 530 nm). Beer's law is obeyed up to 10·30 ppm. The molar absorptivity and Sandell sensitivity are $1{\cdot}42 \times 10^4$ l mole^{-1} cm^{-1} and 0·0072 μg/cm^2, respectively.

Key words: Spectrophotometry, rhodium(III), {2-[di-(2-pyridyl)methylidenehydrazino] quinoline}

1. Introduction

In recent years many nitrogen containing heterocyclic hydrazones derived from 2-hydrazinoquinoline[1–4] have been prepared and tested as possible analytical reagents. Their analytical applications have been reviewed by Katyal *et al*[5]. In our studies on hydrazones[6–10] the synthesis of {2-[di-(2-pyridyl)-methylidenehydrazino] quinoline} (DPMHQ) and its application in the micro-determination of cobalt[11], zinc[12] and vanadium[13] have been reported earlier. In this work DPMHQ has been examined for use as a reagent for the spectrophotometric determination of rhodium(III).

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2. Experimental

All the absorbance measurements were made on a Unicam SP600 spectrophotometer. A Beckman Expandomatic-SS2, pH-meter was used for pH measurements.

DPMHQ solutions were prepared in 95% ethanol and stored in amber glass bottles. Such solutions are stable for several weeks. A stock solution of Rh(III) was prepared by dissolving rhodium trichloride (Johnson Matthey, London) in 1N hydrochloric acid. The solution was standardised by standard method. Dilute solutions of hydrochloric acid and sodium hydroxide were used for pH adjustments.

All other solutions of cations and anions were prepared by dissolving analytical reagent grade chemicals in doubly distilled water.

3. Results and discussion

Rh(III) forms a pink coloured complex on heating with ethanolic solution of DPMHQ in the pH range 1·0–3·0 for 30 min. However, further heating has no effect on the absorbance reading. The complex is soluble in 50% ethanol and shows maximum absorbance at 530 nm. At least 3-fold molar excess of DPMHQ is necessary to obtain constant and reproducible absorbance. Beer's law is obeyed up to 10·30 ppm of Rh (III). The optimum concentration range evaluated by Ringbom method is 1·03–7·72 ppm. The Sandell sensitivity is 0·0072 μg Rh cm^{-2} and the molar absorptivity is 1·42 × 10^4 l mole^{-1} cm^{-1} at 530 nm. Composition of the complex as determined by Job's method of continuous variation shows that metal to ligand ratio is 1 : 1.

Procedure

To an aliquot containing 10·3–77·2 μg of rhodium, add 1·0 ml of ethanolic 1 × 10^{-2} M solution of DPMHQ. Adjust the pH between 1·0 and 3·0 with dilute solutions of sodium hydroxide and hydrochloric acid and heat it for ~ 45 min on water bath. After cooling to room temperature, raise the volume to 10 ml (maintaining 50% ethanol–water ratio) and measure the absorbance at 530 nm against reagent blank prepared under identical condition.

Effect of various ions

Synthetic solutions containing known amounts of Rh(III) and varying amounts of diverse ions were prepared and the recommended procedure was followed for determination of Rh (III). An error of ± 2% in the absorbance reading was considered tolerable. In the determination of 2·57 ppm of Rh(III), ions tolerated (in ppm given in parentheses) are as follows.

Bromide, iodide, nitrate (2000 each); fluoride, borate, phosphate, tartrate (1000 each); citrate (400); nitrite, thiourea, oxalate (200 each); thiocyanate (100); Ca(II), Sr(II),

Ba(II), Mg(II) (600 each); Pb(II), Mn(II), W(VI), Mo(VI) (400 each); Zn(II), Cd(II), Hg(II), Sb(III), Bi(III) (100 each); Ru(III), Os(VIII), Ir(III), Au(III), Pt(IV) (50 each); Ni(II), Cu(II) (5 each): Pd(II), Fe(II), Co(II), CN^- and EDTA interfere seriously. However, Pd(II) (5) can be tolerated by extracting its complex into $CHCl_3$ before heating the solutions. Attempts to mask Co(II) and Fe(II) were unsuccessful.

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References

1. Heit, M. L. and Ryan, D. E. — *Anal. Chim. Acta*, 1966, 34, 407.
2. Singhal, S. P. and Ryan, D. E. — *Anal. Chim. Acta*, 1967, 37, 91.
3. Afghan, B. K. and Ryan, D. E. — *Anal. Chim. Acta*, 1968, 41, 167.
4. Frei, R. W., Jamro, G. H. and Navratil, O. — *Anal. Chim. Acta*, 1971, 55, 125.
5. Katyal, M. and Dutt, Y. — *Talanta*, 1975, 22, 151.
6. Katyal, M., Kundra, S. K., Goel, D. P. and Singh, R P. — *J. Indian Chem. Soc.*, 1974, 51, 576.
7. Ray, H. L., Garg, B. S. and Singh, R. P. — *Curr. Sci.*, 1973, 42, 852.
8. Singh, R. B., Jain, P., Garg, B. S. and Singh, R. P. — *Anal. Chim. Acta*, 1979, 104, 191.
9. Singh, R. B., Garg, B. S. and Singh, R. P. — *Indian J. Chem.*, 1979, 17A, 318.
10. Kulshreshtha, H., Singh, R. B. and Singh, R. P. — *Analyst*, 1979, 104, 572.
11. Singh, R. B., Ray, H. L., Garg, B. S. and Singh, R. P. — *Talanta*, 1979, 26, 898.
12. Singh, R. B., Jain, P., Garg, B. S. and Singh, R. P. — *Analyst*, 1979, 104, 1188.
13. Singh, R. B., Kulshreshtha, H., Garg, B. S. and Singh, R. P. — *Curr. Sci.*, 1979, 48, 109.