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Simultaneous Spectrophotometric Estimation of Cobalt(II) and Nickel(II) with Phenylazobenzaldoxime

K.C. KALIA*, ANIL KUMAR, PARVEEN and RAJEEV SHARMA
Department of Chemistry
Punjabi University
Patiala-147 002, India

Phenylazobenzaldoxime, $C_6H_5—\underset{\underset{\|}{NOH}}{C}—N=NC_6H_5$(I) precipitates quantitatively cobalt(II) and nickel(II) from their aqueous solutions to form water insoluble colored complexes tris(phenylazobenzaldoximato)cobalt(II) and bis-(phenylazobenzaldoximato)nickel(II) respectively. A method for the simultaneous estimation of both cobalt and nickel by measuring the combined optical density at two wavelengths has been reported.

INTRODUCTION

A number of oximes are being used now-a-days for the estimation of cobalt[1–7] and nickel[8–12] spectrophotometrically. A new oxime, phenylazobenzaldoxime,[13,14] was successfully employed by Kalia *et al.*[15] for the estimation of cobalt. Phenylazobenzaldoxime(I) also precipitates nickel quantitatively as bis(phenylazobenzaldoximato)nickel(II), from its aqueous solution.[16]

EXPERIMENTAL

The Spectronic-20, single beam spectrophotometer, was used for the spectrophotometric measurements.

All the reagents used were of AR grade. Doubly distilled water was used during estimations. The standard solutions of cobalt(II) chloride hexahydrate and nickel(II) chloride hexahydrate containing 1 mg of metal per ml were prepared in doubly distilled water and diluted further as required.

Simultaneous Estimation of Cobalt and Nickel

Both the metals can be determined simultaneously by the procedure given below:

A slight excess of reagent (I) in aqueous ammonia was added to a mixture of known amounts of cobalt(II) and nickel(II) solutions at pH 8. The precipitates obtained were digested for 15 minutes and then filtered in G-4 sintered crucible and washed with 5% ammonia followed by doubly distilled water. These

precipitates were dried for 2 hrs at 120°C and then dissolved in 100 ml $CHCl_3$. The absorbance of this solution was then measured at two wavelengths (460 and 550 nm) and the amounts of nickel and cobalt calculated with the simultaneous solution of two spectrophotometric equations as given below in the results and discussion section.

RESULTS AND DISCUSSION

Estimation of Ni

Nickel(II) precipitated quantitatively from its aqueous solution as bis(phenylazobenzaldoximato)Ni(II), is freely soluble in $CHCl_3$ giving maroon-yellow solution which has absorption maxima at 460 nm (ε_{max} 9,350) and obeys Beer-Lambert law from 0.2–2 ppm with percentage error of ± 2%. The results for the estimation of nickel by this method have been summarised in Table 1. This method gives better results for nickel than the method of spectrophotometric determination of nickel by DMG. The latter method is not suited in the presence of Co which seriously interferes with the estimation of nickel.[17]

TABLE 1
ESTIMATION OF NICKEL[a] SPECTROPHOTOMETRICALLY AT 460 nm

Amount of Nickel (in mg) taken	Amount of nickel[b] (in mg) found
0.440	0.436
0.880	0.890
1.320	1.335

[a]Nickel precipitated as bis(phenylazobenzaldoximato)Ni(II) was dissolved in 100 ml at $CHCl_3$ and 1 ml of this solution was diluted to 10 ml.
[b]Each value represents the average of three aliquots, the three being in good agreement.

Simultaneous Estimation of Ni and Co

In principle, two colored species in solution may be determined spectrophotometerically even when their absorption maxima show some overlap, provided the absorptivities of the colored species are independently known at two wavelengths. In applying this principle to the determination of cobalt and nickel, the two metals can be estimated simultaneously from their solutions by measuring the combined absorbance of the coloured complexes of the two metal ions at 460 nm (ε for Ni = 9,350; ε for Co = 9,450) and at 550 nm (ε for Ni = 4,250, ε for Co = 9,350) in $CHCl_3$ solution, as the complexes of cobalt and nickel with (I) do not react with each other in $CHCl_3$ and have absorption maxima at different wavelengths.

The two wavelengths are chosen in such a way that at one of these wavelengths, i.e. at 460 nm, absorbance of both the coloured components, are comparable whereas, at 550 nm, absorbance of one of the components is sufficiently lower than that of the other. By applying this method, combined optical densities of cobalt and nickel complexes are measured at 460 and 550 nm against a reagent blank and the concentration of each metal is then determined by the simultaneous solution of equations (1) and (2):

$$O.D._{(460)} = 9{,}350 \times C_1(Ni) + 9{,}450 \times C_2(Co) \quad (1)$$

$$O.D._{(550)} = 4{,}250 \times C_1(Ni) + 9{,}350 \times C_2(Co) \quad (2)$$

where C_1 and C_2 refer to the concentrations in moles/litre.

The results are given in Table 2. This method is suitable even when cobalt and nickel are present in comparable amounts. This fact is significant because it has been earlier reported that the retio of nickel to cobalt had to be 7 or above for an accurate simultaneous determination of these two metals.[17,18]

TABLE 2
SIMULTANEOUS ESTIMATION OF NICKEL AND COBALT[a]

Amount of nickel[b] (in mg)		Amount of cobalt (in mg)[b]	
Taken	Found	Taken	Found
0.190	0.182	0.945	0.936
0.380	0.372	0.630	0.612
0.506	0.487	0.630	0.618
0.639	0.600	0.630	0.610

[a]Simultaneously precipitated bis(phenylazobenzaldoximato)Ni(II) and tris(phenylazobenzaldoximato)Co(III) were dissolved in 100 ml of $CHCl_3$ and 1 ml of this solution was diluted to 10 ml.
[b]Each value represents the average of three aliquots, the three being in good agreement.

Absorbance measurements are least affected by time period upto 24 hrs and the intensities are constant over the temperature 20 to 45°C. The sensitivity for cobalt is 0.0057/ μg of Co per sq. cm. for $\log I_0/I = 0.001$ at 510 nm and for nickel is 0.0063/μg of Ni per sq. cm. at 460 nm.

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