

NOTE

Solvent Extraction and Spectrophotometric Determination of Co(II) with Isovanillin Thiosemicarbazone

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A new spectrophotometric method is proposed for the determination of Co(II) at microgram level. Isovanillin thiosemicarbazone reacts with Co(II) at pH 8.4 to form a complex which was extracted in ethyl acetate showing maximum absorption at 440 nm. The molar absorptivity and Sandell's sensitivity were calculated to be $0.135 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $9.89 \times 10^{-5} \text{ } \mu\text{g cm}^{-2}$, respectively. The stoichiometry of the complex was found to be 1 : 2. The developed method has been applied for the determination of Co(II) in synthetic and real samples.

Key Words: Spectrophotometry, Estimation, Cobalt(II), Isovanillin thiosemicarbazone.

Thiosemicarbazones have been widely used as analytical reagents^{1,2} in spectrophotometry, fluorimetry and also as visual indicators for a wide variety of metals. The reagent isovanillin thiosemicarbazone (IVTSC) is prepared by reacting isovanillin with thiosemicarbazone³. In this paper, a new spectrophotometric method for the determination of Co(II) using IVTSC is developed.

The stock solution of Co(II) was prepared by dissolving equivalent 100 mg in double distilled water and standardized by a known method⁴. The reagent solution (0.1%) was prepared in methanol. Absorbance and pH measurements were carried out on a Shimadzu UV-Vis 2100 spectrophotometer with 1 cm quartz cell and a digital pH-meter with combined glass electrode, respectively.

Procedure: To an aliquot of Co(II) solution, known amount of IVTSC (0.1%) was added. The pH of the solution was adjusted by adding 2 mL of buffer and volume was made up to 10 mL by distilled water. In a 60 mL separating funnel, the aqueous phase was equilibrated with 10 mL ($2 \times 5 \text{ mL}$) of ethyl acetate for 60 s. The organic extract was collected in 10 mL of standard volumetric flask and made up to mark with ethyl acetate. The absorbance of extracted Co-IVTSC complex measured at 440 nm against reagent blank. From the calibration curve, the amount of Co(II) in organic phase was determined while the amount of Co(II) in aqueous phase was determined by a known method.

The extraction of Co(II) (100 μg) was carried out at different pH and found to be quantitative at pH 8.4. The influence of shaking time for the extraction was

studied for 30–90 s. The quantitative extraction of Co(II) was achieved after 60 s with IVTSC and the complex was stable for 48 h.

Influence of diluents: Various solvents such as *n*-butanol, chloroform, ethyl acetate, ethyl methyl ketone, carbon tetrachloride, toluene and hexane were tried to achieve maximum extraction of Co(II). The extraction of cobalt was quantitative with IVTSC when both were dissolved in IVTSC aqueous solution taking ethyl acetate as diluent.

Effect of reagent concentration: The minimum amount of reagent required for the colour development of an 8 ppm Co(II) was found by varying the reagent concentration from 0.1 mL to 2 mL of methanolic solution of IVTSC (0.1%). It was found that 1.4 mL of IVTSC (0.1%) is sufficient for the extraction of 8 ppm of Co(II).

Nature of extracted species: The nature of the extracted species was ascertained from the plot of $\log D$ vs. $\log R$ from aqueous media (Fig. 1), where $\log D$ is the distribution coefficient of the complex and $\log R$ is the concentration of the reagent. The slope of the graph is 2.06. Thus stoichiometry ratio of metal to ligand was found to be 1 : 2 with the reagent.

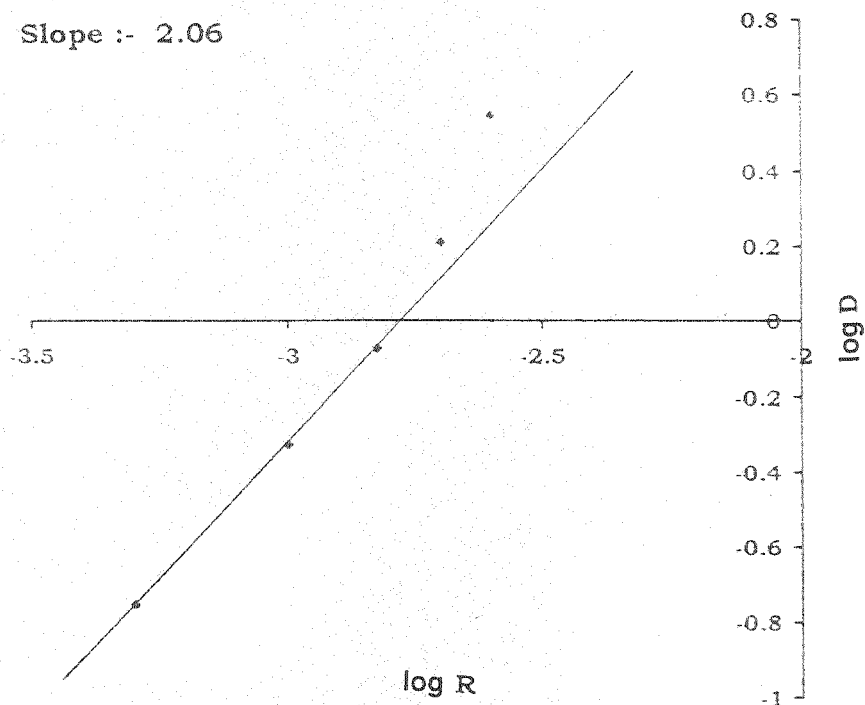


Fig. 1. Stoichiometric ratio of metal to reagent complex of Co(II) with IVTSC from aqueous media.

Beer's law and sensitivity: Calibration graph for Co(II) was constructed under the optimum conditions. The graph obeys Beer's law from the range of 2–80 μg for cobalt. The molar absorptivity and Sandell's sensitivity were found to be $0.135 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $9.89 \times 10^{-5} \mu\text{g cm}^{-2}$, respectively.

Influence of diverse ions on the percentage extraction of Co(II): An interference study showed that large number of cations and anions offer no

interference. The tolerance limit was set at the amount of foreign ions required to cause $\pm 1.0\%$ error in the recommended procedure. The presence of various salts of alkali and alkaline earth metals does not show any effect on the absorbance value of Co(II) IVTSC complex.

Applications: Various commercial samples and synthetic mixtures containing Co(II) were analyzed according to the recommended procedure and the results were compared to those obtained by standard method. The proposed method also facilitates to determine Co(II) from commercial samples. The results are shown in Table-1.

TABLE-1
SEPARATION OF Co(II) WITH IVTSC FROM VARIOUS
BINARY MIXTURES

Binary mixtures Metal taken (ppm)	Cobalt found (ppm)	
	Standard method	Present method
Co (5) + Mo (5)	4.98	5.01
Co (5) + V (5)	5.02	5.01
Co (4) + Sn (6)	4.01	3.99

DETERMINATION OF Co(II) FROM REAL SAMPLES

Sample	Certified value	R-nitroso method	Present method
Injectible			
Glaxo	4.35	4.289	4.347
Merck	8.70	8.698	8.701
Merind	8.76	8.752	8.760
Tablet/Capsules			
Surbex-T Tablet (Abbott)	0.33	0.319	0.321
High Speed Steel			
3082	9.64	9.630	9.632
3083	10.16	10.152	10.164
3084	9.56	9.500	9.522

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