

## References

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### Rapid Extraction and Spectrophotometric Determination of Cobalt(II) with Isonitrosothiocamphor

G. S. KATIYAR\* and B. C. HALDAR

Inorganic and Nuclear Chemistry Laboratory,  
The Institute of Science, Bombay-400 032

Manuscript received 16 August 1985, revised 28 August 1986,  
accepted 5 September 1986

ISONITROSOTHIOCAMPHOR (HINTC) reacts with transition metal ions giving coloured complexes which are extractable into organic solvents<sup>1,2</sup>. The present paper reports a study of the extraction and spectrophotometric determination of Co<sup>II</sup> with HINTC.

#### Experimental

A Carl-Zeiss VSU-2P spectrophotometer was used for absorbance measurements. Radioactivity and pH were measured as described in an earlier communication<sup>3</sup>.

Reagents and chemicals used were of AnalaR grade. Standard solutions of cobalt were prepared by the standard method. Solutions for interference study were obtained by dissolving appropriate salts in water to give 10 mg of element per ml and standardised by the standard method. HINTC was synthesised by the literature method. Radioisotopes was supplied by Isotope Division, Bhabha Atomic Research Centre, Bombay.

**Procedure for extraction and spectrophotometric determination of cobalt:** For spectrophotometric determination, 2 M NH<sub>4</sub>Cl (2 ml) and 0.035% HINTC (1 ml) in alcohol was added to a solution ( $\approx$ 4 ml) containing 1–90  $\mu$ g of cobalt. The pH of the mixture was adjusted to 7 with dilute solutions of HCl and/or NH<sub>4</sub>OH, and after making the volume 10 ml, it was equilibrated for 1 min with chloroform (10 ml). The organic extract was collected in a 10 ml flask and made upto the mark with chloroform, if necessary. The absorbance of the solution was measured at 400 nm against a reagent blank. The amount of cobalt(II) was determined from a calibration curve.

Extraction coefficients of cobalt(II) were determined by using <sup>60</sup>Co tracer.

#### Results and Discussion

Extraction of cobalt is greater than 99.5% in the pH range 6.5–7.5 when HINTC and metal are in the molar ratio of 10 : 1 or more and equilibration time is 1 min.

The distribution coefficient (*E*) of cobalt in different solvents were: chloroform (520), benzene (511), methyl isobutyl ketone (504), carbon tetrachloride (160), methyl ethyl ketone (137), toluene (126), nitrobenzene (121) and chlorobenzene (118).

The absorption spectrum of Co–HINTC in chloroform exhibits a shoulder at 520 nm. However, measurements for the spectrophotometric determination of cobalt have been taken at 400 nm because absorbance at this wavelength is more than that at 520 nm and absorbance due to the reagent blank is 0.02. The absorbance of the extract remains constant for more than 2 days. Beer's law is obeyed in the range 0.1–9.0  $\mu$ g of Co<sup>II</sup> per ml. Molar extinction coefficient at this wavelength is 7 270 dm<sup>2</sup> mol<sup>-1</sup> cm<sup>-1</sup>.

Following ions and masking agents when present in amount indicated do not interfere in the spectrophotometric determination of 50  $\mu$ g of cobalt(II): 10 mg of each of Mn<sup>II</sup>, Mg<sup>II</sup>, As<sup>III</sup>, Ca<sup>II</sup>, Sr<sup>II</sup>, Ba<sup>II</sup>, W<sup>VI</sup> and Mo<sup>VI</sup>; 1 mg of each of Sb<sup>III</sup> and Rb<sup>I</sup>; 0.1 mg of each of Hg<sup>II</sup>, Cd<sup>II</sup>, V<sup>V</sup>, Se<sup>IV</sup>, Ru<sup>III</sup>, Pt<sup>IV</sup> and Bi<sup>III</sup>, Zn<sup>II</sup>, Al<sup>III</sup>, Fe<sup>III</sup> and Cr<sup>III</sup> were kept in solution by adding tartrate. In the presence of 100 mg of thiosulphate, 0.1 mg of each of Au<sup>III</sup> and Ag<sup>I</sup>, do not interfere. Interference by Ni<sup>II</sup> and Cu<sup>II</sup> can be masked by oxalate and thiourea, respectively. 100 mg of each of chloride, bromide, iodide, fluoride, nitrate, tartrate, sulphate, peroxysulphate, thiocyanate, thiourea, urea, thiosulphate, chlorate, bromate and iodate; 50 mg of sulphite; 10 mg of each of oxalate, acetate and phosphate; and 1 mg of citrate and pyrophosphate do not interfere. EDTA and cyanide interfere by hindering the extraction of cobalt.

**Nature of the extracted species:** Curves obtained by the mole-ratio method and by Job's continuous variation method show a break at the point corresponding to HINTC–Co ratio equal to 3 : 1 indicating that the extracted species is formed by the reaction of cobalt and HINTC in the ratio of 1 : 3.

**Precision, accuracy and sensitivity:** The average of 10 determinations with 50  $\mu$ g of cobalt in 10 ml solution is 50.2  $\mu$ g and it varies between 49.1 and 51.4  $\mu$ g at 95% confidence limit.

#### Acknowledgement

The authors are grateful to U.G.C., New Delhi, for the award of a Senior Research Fellowship to one of them (G.S.K.).

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