

NOTES**Spectrophotometric Determination of Cobalt with *m*-(Mercaptoacetamido) phenol.**

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The reagent *m*-(mercaptoacetamido) phenol forms a purple 1 : 2 complex with cobalt which is soluble in sodium hydroxide solution. The reagent has been used for the spectrophotometric estimation of cobalt. The complex has a maximum at 480 nm with molar absorptivity $1.04 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$. The effect of interfering ions has been studied and the method was applied to cobalt(II) determination in vitamin preparations.

Thioglycolic acid does not appear to be an efficient reagent for the spectrophotometric determination of cobalt. However, some of its derivatives have been used for this purpose.¹ The reagent *m*-(mercaptoacetamido) phenol (*m*-MAP)-a condensed product of thioglycolic acid and *m*-amino phenol has been used as a gravimetric reagent for the milligram level determination of platinum metals²⁻⁴. The reagent gives purple precipitate with cobalt between pH 6.5 to 7.0 which is soluble in sodium hydroxide solution due to the presence of the phenolic group in the reagent. This property has been used in the present study.

The proposed method for the determination of cobalt using *m*-MAP is simple and sensitive. The method has been successfully used for the determination of cobalt(II) in vitamin preparations.

Analytical grade chemicals were used in the study of interference. Deionized water was used throughout the study.

A stock solution of cobalt(II) was prepared by dissolving 2.2048 g of anhydrous cobalt(II) chloride in 1 l of water. The solution was standardized gravimetrically. Further dilutions were made as required.

Disodium hydrogen phosphate (0.2M) and citric acid (0.1M) were used to prepare the buffer. Sodium hydroxide (0.1M) was used to dissolve the complex.

The reagent was synthesized as described⁵. A solution (1.5 mg/ml) was prepared by dissolving 0.15 g in 20% ethanol. The reagent is slowly oxidized by air and light (disulphide formation) and hence the reagent solution should not be stored for more than three days. Use of fresh solution of the reagent is recommended.

Procedure

To an aliquot of solution containing 8 μg of Co(II) 0.8 ml of *m*-MAP solution was added followed by 0.5 ml of disodium hydrogen phosphate. The complex

was dissolved in 0.5 ml of sodium hydroxide and volume was made upto 5 ml with water. The reaction mixture was allowed to stand for about 45 min. The absorbance was measured at 480 nm against reagent blank using a cell of 10 mm path length. A calibration graph was prepared for eight points between 4–20 μg and was used to determine the amount of cobalt in vitamin preparations.

Determination of Cobalt(ii) in vitamin preparations

Two ampules (2 ml each) of the vitamin sample were decomposed in a silica crucible by heating with nitric acid. The solution was cooled and the procedure was repeated three times. Finally the residue was dissolved in 1 ml of concentrated hydrochloric acid and made to 25 ml. The cobalt contents were determined as described above. The results are given in Table 1.

TABLE-1
DETERMINATION OF COBALT(II) IN VITAMIN PREPARATIONS.
NUMBER OF DETERMINATIONS FIVE IN EACH CASE.

	Cobalt(II) $\mu\text{g}/\text{ampoule}$		error%
	Present	Found	
Macraberin Fort Injection (Glaxo India Ltd.)	42.8	42.1	1.6
Hydroxo 5000 (Anhar-Ronald, France)	218.9	223.3	2.0

Absorption Spectra

The absorption spectra of the complex was recorded in the range 400–600 nm against reagent blank. The complex showed an absorption maxima at 480 nm and all measurements were made at this wavelength. The system obeys Beer's law over a concentration range 1 to 20, $\mu\text{g}/5$ ml. (Fig. 1) The molar absorptivity and Sandell's sensitivity are 1.04×10^4 $\text{l mol}^{-1} \text{cm}^{-1}$ and 5.7 ng cm^{-2} respectively. Eight replicate analyses containing $8.0 \mu\text{g}/5$ ml gave $7.76 \mu\text{g}/5\text{ml}$ of cobalt with a standard deviation of 0.06.

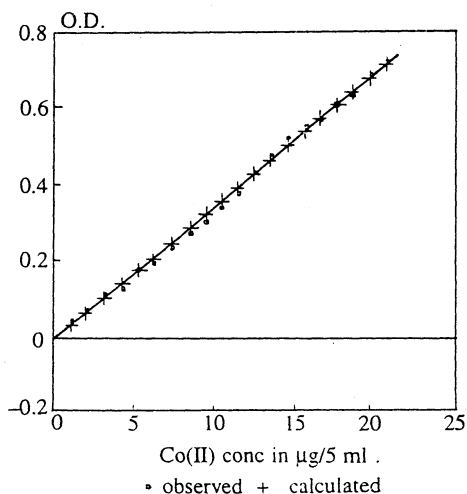


Fig. 1 The Beers law for Co-m-MAP complex

Effect of pH

Preliminary studies indicated that the complex starts precipitating around pH 6.5. A study was, therefore, carried

out over the pH range 6.0 to 10.0. The complex showed only one maxima at 480 nm indicating that only one type of specie is formed. The absorption remained constant and maximum between pH 8.0 and 10.0. A pH of 9.0 was selected for further study.

Effect of Reagent Concentration

The effect of reagent concentration was studied keeping the metal ion concentration constant and varying the mole ratio of reagent to cobalt (50 to 300 μg). It was found that 150 fold excess of the reagent was necessary for constant and maximum absorbance.

Effect of Foreign Ions

Solutions containing 8.0 $\mu\text{g}/5\text{ ml}$ of cobalt(II) and various amounts of other ions were prepared and the recommended procedure was followed for the estimation of cobalt(II). An error of more than 2% in the absorbance was taken as interference. The amount, in microgram, upto which various ions do not interfere are given in parentheses. Chloride (1000), bromide (1000), iodide (700), nitrate and sulfate (1000), fluoride (200), chromium, mercury, tin(II) antimony (100), zinc, strontium, barium, calcium (1000), lithium, sodium, potassium do not interfere while the interference due to copper, nickel, iron(II) and (III) and silver could not be avoided. This is because copper, nickel and silver precipitate at this pH and the reagent reacts with iron in the capacity of phenol.

Nature of the complex

The composition of the complex was determined by Job's method of continuous variation which gave a 1 : 2 (M : L) composition for the complex. Infrared spectrum indicated the absence of the band at 2560 cm^{-1} due to $-\text{SH}$ and a shift in the $-\text{C}=\text{O}$ (1632 cm^{-1}) frequency suggesting bonding through $-\text{SH}$ and $-\text{C}=\text{O}$ groups.

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