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Complexometric Determination of Zinc (II) in Pharmaceutical Samples Using Hydroxytriazenes

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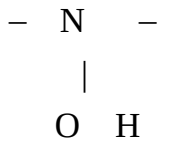
ABSTRACT
The present paper describes complexometric determination of Zn (II) using three hydroxytriazenes as metallochromic indicators in three medicines- Zivinal-CD, Becozine, and Vi - Syneral-Z. The hydroxytriazenes used were 3-hydroxy-3-phenyl-1-p-carboxyphenyltriazene (HP*p*CPT), 3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene (HP*o*CPT), 3-hydroxy-3-*p*-tolyl-1-p-sulphonamidophenyltriazene (H*p*T*p*SPT), respectively. Determination of zinc (II) was done by back titration method.

Keywords: Hydroxytriazenes, Complexometric, Zinc, Pharmaceutical sample.

INTRODUCTION

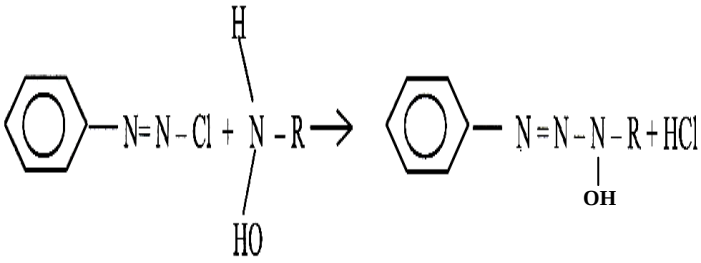
Hydroxytriazenes are compounds having the functional group.



They are also called as triazene oxide and diazohydroxyamines. Their utility as spectrophotometric reagents as well as metallochromic indicators for complexometric determination of transition metals is well established by various reviews. [1-4] However not much work has been done on metal analysis in pharmaceutical samples using hydroxytriazenes. [5-8] In view of this, some methods have been developed to determine Zn (II) in Zinc containing pharmaceutical samples complexometrically using hydroxytriazenes as metallochromic indicators.

MATERIALS AND METHODS

Synthesis of hydroxytriazenes: All the three hydroxytriazenes were synthesized by using Mai's method [9] which involve coupling of alkyl or aryl hydroxylamine with the diazotized aromatic amine in sodium acetate medium of *p*H- 5.0 and temperature range of 0-5°C. Reaction of the method can be represented as:



Where R is alkyl or aryl

The following general procedure was adopted to determine Zn (II) in Zivinal -CD, Becozine, and Vi-Syneral-Z:

a) Digestion of pharmaceutical sample.
b) Complexometric determination of Zn (II) in the digested sample using 3- hydroxy-3-phenyl-1-p-carboxyphenyltriazene, 3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene and 3-hydroxy 3-p-tolyl-1-sulphonamido-phenyltriazene.

For the decomposition of organic part, required amount of pharmaceutical sample was taken in a China dish and treated with concentrated Nitric acid and heated up to dryness. This process was repeated at least 8-10 times, dry residue was then boiled with double distilled water and the mixture was filtered in a volumetric flask. The solution was made up to the mark with double distilled water, thus getting 1×10^{-2}M Zn (II) solution.

Determination of Zinc (II) was done by back titration method. In this method, 10 ml aliquot of pharmaceutical sample and 20 mL EDTA solution were taken in 250 ml conical flask. The *p*H of this solution was adjusted between 6.0 to 7.5 by using 1 % perchloric acid or 5 % sodium acetate

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Table 1: Results of complexometric determination of zinc (II) in pharmaceutical samples using hydroxytriazenes as indicators

Pharmaceutical sample	Indicator	Volume of aliquot used for titration (mL)	*p*H	Volume of EDTA consume (mL)				Colour change at the end point
				Conc. 1.0×10^{-2}M	Conc. 5.0×10^{-3}M	Conc. 2.0×10^{-3}M	Conc. 1.0×10^{-3}M	
Zivinal -CD	HP*p*CPT	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
	HP*o*CPT	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
	H*p*T*p*CST	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
Becozine	HP*p*CPT	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
	HP*o*CPT	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
	H*p*T*p*CST	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
Vi-Syneral-Z	HP*p*CPT	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
	HP*o*CPT	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow
	H*p*T*p*CST	10.0	6.5-7.5	10.0	10.0	10.0	10.0	Colourless to bright yellow

solution as per the need. Finally 10-15 ml of sodium acetate - acetic acid buffer was added to keep the *p*H in the range 6.5 - 7.5. Throughout the titration four to five drops of 0.2 % - 0.5 % solution of indicator (hydroxytriazene) were added. The solution was titrated with zinc solution (solution of pharmaceutical sample) at room temperature. A colourless solution was obtained on addition of indicator solution. The solution so obtained was titrated very slowly with Zinc solution of same concentration. Close to the end point 2-3 drops of indicator solution were again added so as to make the end point clearer. At the end point there was sharp colour change from colourless to bright yellow in all the hydroxytriazenes as indicator

RESULTS AND DISCUSSION

The Zinc (II) content of each pharmaceutical sample was also checked with standard titrating indicator xylenol orange which was found almost same with Zn (II) content found using all the three hydroxytriazenes. Pharmaceutical samples of different concentrations were prepared and titrated with equimolar E.D.T.A. solution and three concordant readings were taken (Table - 1).

Thus the present study establishes three new metallochromic indicators for Zinc (II) determination complexometrically in the pharmaceutical samples. Its easy synthesis, higher yield and economic method for the preparation further enhance its application as metallochromic indicator for Zinc (II).

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