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NOTE

Complexometric Determination of Iron in Pharmaceutical Sample Using Hydroxytriazenes

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The present paper describes complexometric determination of Fe (III) using three hydroxytriazenes as metallochromic indicators in three medicines "Dexorange" (Franco Indian Pharmaceuticals Ltd.) "Austrin" (Weyeth Lederlab Ltd.) and "Fesovit" (Smith-Kline Beechen Pharmaceuticals). The hydroxytriazenes used were 3-hydroxy-3-methyl-1-*p*-sulphonamidophenyltriazene (HMSPT), 3-hydroxy 3-phenyl-1-*p*-chlorophenyltriazene (HP*p*CPT) and 3-hydroxy-3-phenyl-1-*m*-chlorophenyltriazene (HP*m*CPT), respectively.

Key Words: Complexometric, Iron, Pharmaceutical sample.

Hydroxytriazenes are compounds having the functional group $-\overset{OH}{\overset{|}{N}}-N=N-$. 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[1–7]. However not much work has been done on metal analysis in pharmaceutical samples using hydroxytriazenes. In view of this, some methods have been developed to determine Fe(III) in Iron containing pharmaceutical samples complexometrically using hydroxytriazenes[8] as metallochromic indicators.

Synthesis of hydroxytriazenes

All the three hydroxytriazenes were synthesized by using standard methods[9] which involve coupling of alkyl or aryl hydroxylamine with the diazotized aromatic amine in sodium acetate medium of pH 5.0 and temperature range of 0–5°C. Reaction of the method can be represented as:

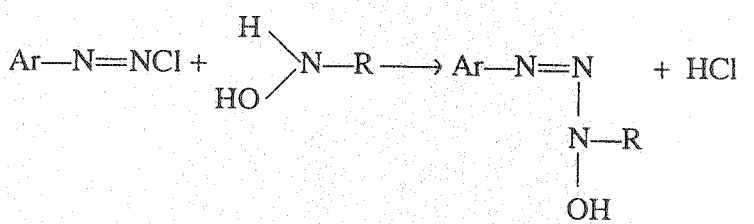

TABLE-1
RESULTS OF COMPLEXOMETRIC DETERMINATION OF IRON(III) IN PHARMACEUTICAL SAMPLE USING HYDROXYTRIAZENES AS INDICATOR

Pharmaceutical sample	Indicator	Vol. of aliqout used for titration (mL)	pH	Vol. of EDTA consumed (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	
Dexorange	HMSPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
	HP*p*CPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
	HP*m*CPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
Austrin	HMSPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
	HP*p*CPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
	HP*m*CPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
Fesovit	HMSPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
	HP*p*CPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow
	HP*m*CPT	10.0	2.0–4.0	10.0	10.0	10.0	10.0	Bluish-violet to yellow

The following general procedure was adopted to determine Fe(III) in dex-orange, austrin and fesovit:

(a) To convert Fe (II) into Fe (III) in the capsule to a titrable form by digestion of pharmaceutical sample.

(b) Complexometric determination of Fe(III) in the digested sample using 3-hydroxy-3-methyl-1-*p*-sulphonamidophenyl triazene, 3-hydroxy-3-phenyl-1-*p*-chlorophenyl triazene and 3-hydroxy-3-phenyl-1-*m*-chlorophenyl triazene.

For the conversion of Fe(II) into Fe(III) and the decomposition of organic part the required amount of pharmaceutical sample was taken in a china dish and treated with conc. HNO_3 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 Fe(III) solution.

For the determination of iron(III) by this method a 10 mL aliquot of pharmaceutical sample was taken in a 250 mL conical flask. The pH of this solution was adjusted between 3.0–5.0 by using 1% perchloric acid or 5% sodium acetate solution as per the need. Finally 10–15 mL of sodium acetate-acetic acid buffer was added to keep the pH in the range 3.0–5.0. Now 0.2–0.5% indicator (hydroxytriazene) solution was added. The solution was titrated with E.D.T.A. at room temperature. A dark bluish violet colour was developed on addition of indicator solution, but at the end point the colour sharply changed from bluish violet to light yellow. The same procedure was applied for all the three hydroxytriazenes. The iron(III) content of each pharmaceutical sample was also checked with standard titrating indicator sulphosalicyclic acid which was found almost same with Fe(III) 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 Fe(III) determination complexometrically in the pharmaceutical sample.

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