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NOTE

Complexometric Determination of Copper in Pharmaceutical Samples Using Hydroxytriazenes

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The present paper describes complexometric determination of Cu(II) using three hydroxytriazenes as metallochromic indicators in three drugs Tamra-bhasm-I, Tamra-bhasm-II and Tamra-bhasm-III. The hydroxytriazenes used were 3-hydroxy-3-methyl-1-*p*-sulphonamidophenyl triazene (HMSPT), 3-hydroxy-3-phenyl-1-*p*-chlorophenyl triazene (HP*p*CPT) and 3-hydroxy-3-phenyl-1-*m*-chlorophenyl triazene (HP*m*CPT), respectively.

Key Words: Complexometric, Copper, Pharmaceutical sample.

Hydroxytriazenes are compounds having the functional group $-\overset{OH}{\overset{|}{N}}-N=N-$, which is responsible for complex formation with metal ion. Hydroxytriazenes have been used as metallochromic indicators for a number of metal ions such as iron[1], copper[2, 3] and other transition metal ions[4–9]. In view of this, three hydroxytriazenes, *i.e.*, 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) have been used for the complexometric determination of copper(II) in three drugs Tamra-bhasm-I, Tamra-bhasm-II and Tamra-bhasm-III.

Synthesis of Hydroxytriazenes

All the three hydroxytriazenes were synthesized by using standard methods reported in literature[10], which involves coupling of alkyl or aryl hydroxylamine with the diazotised aromatic amine in sodium acetate medium of pH 5.0 and temperature range of 0–5°C. The compounds thus synthesized were crystallized and their compositions were verified by elemental analysis and melting point determination. The reaction can be represented as:

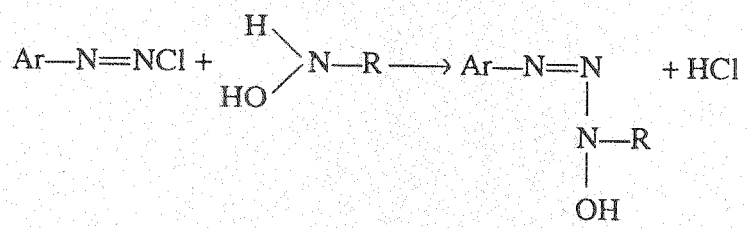

TABLE-1
RESULTS OF COMPLEXOMETRIC DETERMINATION OF COPPER(II) IN PHARMACEUTICAL SAMPLE USING HYDROXYTRIAZENES AS INDICATOR

Pharmaceutical sample	Indicator	Vol. of aliquot 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	
Tamra-bhasm-I	HMSPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
	HP*p*CPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
	HP*m*CPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
Tamra-bhasm-II	HMSPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
	HP*p*CPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
	HP*m*CPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
Tamra-bhasm-III	HMSPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
	HP*p*CPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow
	HP*m*CPT	10.0	5.0–6.0	10.0	10.0	10.0	10.0	Dark blue to light yellow

The following general procedure was adopted to determine Cu(II) in Tamra-bhasm-1, Tamra-bhasm-II and Tamra-bhasm-III.

(a) To prepare standard solution of Cu(II) by the digestion of pharmaceutical sample.

(b) Complexometric determination of Cu(II) 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 decomposition of organic part 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; the dry residue was then boiled with double distilled water and the mixture was filtered in a volumetric flask. The residue was washed with warm double distilled water for a number of times and washing was collected into the same volumetric flask. The solution was made up to the mark with double distilled water, thus getting 1×10^{-2} M Cu(II) solution.

For the determination of copper(II) 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 to 5.0–6.0 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 5.0–6.0. Now 0.2–0.5% indicator (hydroxytriazene) solution was added. The solution was titrated with EDTA at room temperature. A dark blue colour was developed on addition of indicator solution, but at the end point colour sharply changed from dark blue to light yellow. The same procedure was applied for all the three hydroxytriazenes. The copper(II) content of each pharmaceutical sample was also checked with standard titrating indicator eriochrome black-T, which was found very near or same as the Cu(II) content found using all the three hydroxytriazenes. Pharmaceutical samples of different concentrations were prepared and titrated with equimolar EDTA solution and three concordant readings were taken (Table-1). Thus all the three hydroxytriazenes act as very good metallochromic indicators for Cu(II) determination complexometrically in pharmaceutical samples.

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