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NOTE

Application of *o*-Carboxyphenylazo-*bis*-acetoxime in Spectrophotometric Determination of Iron(III)

SABA KHAN* and REHANA KHANAM†
Department of Chemistry, Vidhya Bhawan Rural Institute, Udaipur-313 001, India
E-mail: masoom_75@rediffmail.com

o-Carboxyphenylazo-*bis*-acetoxime has been used for spectrophotometric determination of iron(III) at 376 nm, keeping the pH at 2.8-4.0. Beer's law is obeyed in the range (4 to 24) × 10^{-5} M. The molar absorptivity and Sandell's sensitivity values are 2.446 dm^3 mol^{-1} cm^{-1} and 22.83 ng cm^{-2}, respectively.

Key Words: Arylazo-*bis*-acetoxime, Spectrophotometry, Iron(III).

The hydroxytriazenes are used for the determination of transition metals[1,2]. Herein, an application of a similar compound, *o*-carboxyphenylazo-*bis*-acetoxime (*o*-CPABA) for spectrophotometric determination of iron(III) is reported.

o-Carboxyphenylazo-*bis*-acetoxime was prepared by reported method[3].

Stock solution: A 1.0 × 10^{-2} M ferric nitrate nonahydrate (BDH) was prepared in distilled water and to prevent hydrolysis, few drops of (1 M) conc. HNO_3 were added to it. The solution was standardized with EDTA. A 1 × 10^{-2} M solution of the reagent *o*-CPABA was prepared in acetone. *Tris*-buffer (2 %, v/v) was prepared. A UV/Vis Systronic 108 spectrophotometer and a Systronic 324 pH meter were used.

Method: Spectrum of *o*-carboxyphenylazo-*bis*-acetoxime was measured in the wavelength region 360-460 nm against solvent blank. Iron and *o*-CPABA solutions were taken in 1:5 ratio and the spectrum of iron complex was recorded against reagent blank in the range 360-460 nm. The working wavelength was found to be 376 nm. A set of solutions containing Fe(III) and *o*-CPABA reagent in ratio 1:5 was prepared and pH was varied between 2 to 6. The pH range of constant maximum absorbance was found to be between 2.8 to 4.0. Composition of the complex was determined by Job's method and moles ratio method of Yoe and Jones. The study revealed that composition of iron(III) complex is 1:2 (M:R). Absorbance of set of six solutions containing Fe(III) to *o*-CPABA in ratio 1:5 was measured at corresponding working wavelength against reagent blank. Beer's law was obeyed in concentration range 4 × 10^{-5} to 24 × 10^{-5} M. Interference of 23 cations and anions in the determination of iron were studied. To the set of solutions containing iron to reagent 1:5 ratio, 10 ppm of different foreign ions were added at optimum conditions. Absorbance was measured against reagent blank. Those ions, which did not interfere

†Department of Chemistry, M.L. Sukhadia University, Udaipur-313 001, India.

at 10 ppm level their interference was again studied at 50 ppm level. In case either no or little change in absorbance was observed as compared to the absorbance without any foreign ion, then for those ions interference was studied again at 100 ppm level. Iron(III) was found to form 1:2 complex with *o*-carboxyphenylazo-*bis*-acetoxime.

Stability constants: Harvey and Manning's method[4] and Purohit's method[5] have been used to determine the stability constants. Validity of the methods can be confirmed from the value of log β obtained from both the methods. The log β values agree quite well. Further the precision studies were carried out by measuring the absorbance of 10 sets of solution containing 11.17 ppm of iron(III) and *o*-CPABA in 1:5 ratio, under optimum conditions. The absorbance was measured against reagent blank at working wavelength (376 nm). Iron was successfully determined at 11.17 ppm level with good precision.

TABLE-1
VALUE OF log β AND ΔG RESPECTIVELY BY TWO DIFFERENT METHODS

Method	Reagent	Composition of complex	Conc. of complex (M)	E_m	E_S	α	$K_{inst.}$	β	log β	DG at 27 ºC (kcal/mol)
A	*o*-CPABA	1:2	1×10^{-4}	0.235	0.194	0.174	2.50×10^{-10}	4.00×10^{9}	9.60	-13.18
B	*o*-CPABA	1:2	4×10^{-4}	0.870	0.778	0.105	8.21×10^{-10}	1.21×10^{9}	9.08	-12.46
		1:2	2×10^{-4}	0.435	0.385	0.115	2.70×10^{-10}	3.70×10^{9}	9.56	-13.12

A = Harvey& Manning's method; B = Purohit's method.
o-CPABA = *o*-Carboxyphenylazo-*bis*-acetoxime.

Interference of several cations and anions in the determination of iron was studied at 10, 50 and 100 ppm level. Interference was studied using following 23 cations and anions *viz.*, Na^+, K^+, NH_4^+, Ba^{2+}, Cr^{3+}, Co^{2+}, Pb^{2+}, Ca^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, Hg^{2+}, F^-, Cl^-, Br^-, I^-, NO_2^-, SO_4^{2-}, CO_3^{2-}, $C_2O_4^{2-}$, SO_3^{2-}, PO_4^{3-}. It was seen that at 10 ppm level Na^+, K^+, NH_4^+, Ba^{2+}, Pb^{2+}, Ca^{2+}, Cd^{2+}, Hg^{2+}, Cl^-, Br^-, I^-, NO_2^-, SO_4^{2-}, CO_3^{2-}, $C_2O_4^{2-}$ did not interfere hence interference of these ions were then studied at 50 ppm level. Here Na^+, K^+, NH_4^+, Ba^{2+}, Pb^{2+}, Cd^{2+}, Hg^{2+}, Cl^-, Br^-, I^-, NO_2^-, SO_4^{2-} did not interfere. Further, it was seen that at 100 ppm level the following ions still did not interfere *viz.*, Na^+, K^+, NH_4^+, Ba^{2+}, Pb^{2+}, Hg^{2+}, Cl^-, Br^-, I^-. Thus, it can be seen that iron(III) can be determined even in presence of number of interfering species present at 100 ppm level. Thus, from the above studies it can be concluded that *o*-carboxyphenylazo-*bis*-acetoxime can be used successfully for spectrophotometric determination of iron(III).

REFERENCES

1. D.K. Gorji, R.S. Chauhan, A.K. Goswami and D.N. Purohit, *Rev. Anal. Chem.*, **17**, 223 (1998).
2. S. Kumar, A.K. Goswami and D.N. Purohit, *Rev. Anal. Chem.*, **22**, 73 (2003).
3. J. Mai, *Chem. Ber.*, **25**, 1685 (1892).
4. A.E. Harvey and D.L. Manning, *J. Am. Chem. Soc.*, **72**, 4488 (1950); **74**, 4744 (1952).
5. S. Ressalan, R.S. Chauhan, A.K. Goswami and D.N. Purohit, *Asian J. Chem.*, **11**, 123 (1999).

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