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Analytical application of phenylazo-bis-acetoxime in the spectrophotometric determination of Iron (III)

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ABSTRACT

Phenylazo-bis-acetoxime has been used for spectrophotometric determination of Iron (III) at 362nm, keeping the pH at 3.5-4.0. Beer's law is obeyed in the range (4 to 24)x10^{-5} M. The molar absorptivity and Sandell's sensitivity values are 1,852 dm^3 mol^{-1} cm^{-1} and 30.15 ng cm^{-2}, respectively.

Key words: Arylazo-bis-acetoxime, spectrophotometric determination, Iron (III).

The hydroxytriazenes[1-2] are used for the determination of transition metals. We report here application of a similar compound, phenylazo-bis-acetoxime (PABA) for spectrophotometric determination of iron(III).

Phenylazo-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) concentrated nitric acid were added to it. The solution was standardized with EDTA. A 1x 10^{-2} M solution of the reagent PABA 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 phenylazo-bis-acetoxime was measured in the wavelength region 360-460nm against solvent blank. Iron and PABA 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 362 nm. A set of solutions containing Fe(III) and PABA 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 3.5 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 PABA in ratio 1:5 was measured at corresponding working wavelength against reagent blank. Beer's law was obeyed in concentration range 4x10^{-5} to 24x10^{-5} M. Interference of 23 cations and anions in the determination of iron was 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 at 10 ppm level their interference was again studied at 50 ppm level. In case no or little change in absorbance was seen as compared to the absorbance without any foreign ion, then for those ions interference was studied again at 100 ppm level. However tolerance of still higher concentration was not studied.

Iron(III) was found to form 1:2 complex with phenylazo-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. Value of log β obtained by Harvey and Manning method and Purohit's method were 8.99 and 9.08 respectively.

The log β values agree quite well. The values of Δ G obtained for the Harvey and Manning method and Purohit's method were -13.18 and -13.12 (kcal/mole) at 27°C respectively. Further the precision studies were carried out by measuring the absorbance of 10 sets of solution containing 11.17 ppm of iron(III), and PABA in 1:5 ratio, under optimum conditions. The absorbance was measured against reagent blank at working wavelength (362 nm). Iron was successfully determined at 11.17 ppm level with good precision.

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^-. However tolerance of higher concentration was not studied. 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 phenylazo-bis-acetoxime can be used successfully for spectrophotometric determination of iron(III).

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