

Extraction-spectrophotometric Determination of Iron using Benzyltriethylammonium Chloride

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A number of quaternary ammonium salts have been used with considerable advantage in the spectrophotometric determination of many metal ions¹. Here, we have observed that iron and PAR forms a complex which could be extracted into n-butanol in the presence of benzyltriethylammonium (BTA) chloride. Extraction into n-butanol of the ion-pair formed between iron and PAR complex with BTA cation derived from BTA chloride and measurement of the organic extract forms the basis of the present investigation.

Results and Discussion

When PAR was added to iron(II) solution in the presence of a buffer solution (phosphate buffer of pH 3.40–7.80), a reddish orange complex was obtained, which was not extractable into n-butanol. On addition of a solution of BTA chloride, it formed a ternary complex which was found to be extractable into n-butanol. The organic extract exhibits two absorption maxima at 492 and 712 nm. The reagent blank and the pure solvent do not show any significant absorbance at these wavelengths. Maximum absorbance was obtained when the extraction was carried out in the pH range 3.40–7.80. The pattern of the absorption spectrum of the complex, extracted throughout this pH range remains unchanged. This indicates the presence of a single variety of the complex species in the system. At pH higher than 7.80, the reagent blank develops an intense colour.

The iron complex showed a maximum absorbance when the concentration of PAR was maintained from 1×10^{-2} to 1×10^{-4} M. The iron complex gave quantitative extraction and maximum absorbance when BTA chloride concentration was maintained within 0.5–2.5%.

The composition of iron : PAR in the complex corresponds to 1 : 2 according to the Job's method which was further confirmed by the molar-ratio method.

Various solvents like benzene, chloroform, carbon tetrachloride, dichloromethane, methyl isobutyl ketone, ethyl acetate, isoamyl alcohol and n-butanol were tested for the extraction of the iron complex. The complex was extracted quantitatively into n-butanol only. It required only about 1 min shaking for the complete extraction.

Beer's law was obeyed upto a concentration of 1.6 ppm of iron. The molar absorptivities and Sandell's sensitivities

were 5×10^4 dm³ and 2.93×10^4 dm³ mol⁻¹ cm⁻¹, and 0.001 and 0.0019 μ g cm⁻² respectively, at 492 and 712 nm. Iron could be determined using either of the two wavelengths i.e. 492 nm and 712 nm. However, the complex showed maximum absorbance at 492 nm. The maximum absorption at 492 nm is found to be more sensitive compare to that at 712 nm.

Effect of diverse ions : The effect of different foreign ions in the determination of iron (8 μ g) was studied. The tolerance limit was set at $\pm 3\%$ error in the recovery of iron. The following foreign ions (in μ g), Cu^{II}(250), Ag^I(100), Zn^{II}(500), Cd^{II}(1500), Hg^{II}(250), Bi^{III}(150), Cr^{III}(500), Mn^{II}(1500), Al^{III}(1000), Mo^{VI}(50), W^{VI}(60), U^{VI}(500), Rh^{III} (80), Pt^{IV} (40), Pd^{II} (50), F⁻ (1500)⁻, Br⁻(1500), I⁻ (1500), SCN⁻ (500), NO₃⁻ (1000), NO₂⁻ (1000), PO₄³⁻(1200), CH₃COO⁻ (1200) and thiourea (1500) caused no interference. Equal amounts of Au^{III}, Sb^{III}, Co^{II}, V^V and Ni^{II} interfered. Interference due to Au^{III}, Sb^{III} and Co^{III} could be removed by using KBr, citrate and KF as masking agents respectively. Interference due to V^V and Ni^{II} could be removed by addition of H₂O₂ prior to the addition of other reagents and extraction in chloroform as nickel-dimethylglyoximate respectively. However, EDTA and CN⁻ interfered even when present in very small amounts.

The method was applied to determine iron in high tension brass (No.10 G) and lead concentrate (No. 42 G), both procured from BAS, Ltd., England. Dissolution of the samples were done following literature procedure. The recovery of iron in both the cases were found to be below the tolerance limit ($\pm 3\%$). Results of 10 determinations of iron (8 μ g) showed that standard deviations and coefficient of variations were 0.000 45, 0.000 27, and 0.062, 0.064% respectively, at 492 and 712 nm.

The present method is simple, rapid, precise and sensitive. The total operation time in each run is 10–15 min.

Experimental

A Hitachi U-3210 spectrophotometer and an Elico LI-10 pH meter were used.

All chemicals and solvents were of A.R. grade.

A stock solution of iron(II) was prepared from ferrous ammonium sulphate hexahydrate (B.D.H.) and standardised. A solution of PAR (monosodium salt, Sigma) was prepared in distilled water. BTA chloride was prepared the

NOTE

reported method². Buffer solutions of different pH values were prepared using standard procedures. Standard solutions of diverse ions were prepared from their chlorides, nitrates or sulphates (in case of cations) and sodium, potassium or ammonium salts (in case of anions).

Procedure : To an aliquot containing upto 16 µg of iron(II), 1×10^{-3} M PAR (1 ml) and buffer solution (4 ml; phosphate buffer of pH 4.80) were added and the volume was made upto 10 ml with distilled water. To the above mixture, 1.5% BTA chloride solution (1 ml) and n-butanol (10 ml) were added. The resulting mixture was shaken for

about 1 min and then the phases were allowed to separate. The separated organic layer was dried over anhydrous sodium sulphate. The absorbance of the organic extract was measured at 492 and 712 nm against the reagent blank. The amount of iron present was then computed.

References

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