



EXTRACTIVE SPECTROPHOTOMETRIC STUDIES OF ACETOPHENONE 2', 4'-DIHYDROXY THIOSEMICARBAZONE WITH IRON

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ABSTRACT

Acetophenone 2', 4'-Dihydroxy thiosemicarbazone (APDHTS) as a reagent for the extractive spectrophotometric determination of Iron. The reagent APDHTS gave instantaneous and stable violet colour with Iron at pH 5.0. A linear calibration graph over the concentration range 1 ppm to 10 ppm with a 3 σ limit of detection of 0.66 ppm was obtained by applying the spectrophotometric method at wavelength 370 nm. The stoichiometry of the complex is established as 1:1 (M: L) by Job's method of continuous variation and confirmed by mole ratio method. The standard deviation and the coefficient of variations are presented. The molar absorptivity and Sandal's sensitivity of the complex is 0.4469 x 10³ L mol⁻¹cm⁻¹ and 0.0125 μ g cm⁻² respectively. The results of the prescribed procedure applied for the determination of the micro amounts of Fe (III) in various synthetic and standard steel samples are presented.

KEYWORDS: Extractive Spectrophotometric Determination, Acetophenone 2',4'-dihydroxy thiosemicarbazone (APDHTS), Iron, Beer's Law, Molar Absorptivity, Sandell's Sensitivity.

INTRODUCTION

Iron is the most common element (by mass) forming the planet Earth as a whole, forming much of Earth's outer and inner core. It is the fourth most common element in the Earth's crust. Iron's very common presence in rocky planets like Earth is due to its abundant production as a result of fusion in high-mass stars. Pure Iron is soft but is unobtainable by smelting. A certain proportion of carbon (between 0.2% and 2.1%) produces steel, which may be up to 1000 times harder than pure Iron. Colorimetric and Atomic Emission or Atomic Absorption methods¹ are most commonly used for the determination² of Fe (III). However, colorimetric methods are generally preferred; as they involve less expensive instrumentation and afford better sensitivity when appropriate chromogenic reagents³ and solvent extraction pre-concentration steps are employed. Most of the extractive spectrophotometric methods⁴ developed for Fe (III)⁵ are based on reactions with suitable colour producing reagents⁶. In this present paper, extractive spectrophotometric determination of Iron with Acetophenone 2',4'-Dihydroxy thiosemicarbazone (APDHTS)⁷ is presented.

EXPERIMENTAL

Electrochemically and spectrally pure water obtained from triple distillation of deionized feeder water was used for conducting analytical operations⁸. All the chemicals were of Analytical grade quality. The inorganic salt solutions of various metal ions in higher concentrations were prepared by dissolving appropriate salts in requisite quantities in triple distilled water to give a solution containing 200 ppm and few drops of a suitable acid were added before dilution wherever necessary to prevent hydrolysis. A number of buffer solutions

ABSORPTION SPECTRUM

The absorption spectra of Fe (III): Acetophenone 2', 4'-dihydroxy thiosemicarbazone (APDHTS) complex was recorded from 200-800 nm against reagent blank. From the spectra, the absorption maximum (λ max) of the complex is observed at 370 nm at which, the absorbance of the metal ion and the ligand are negligible. Hence for the

of constant ionic strength of 0.2 M covering a wide pH range were prepared. The ligand APDHTS was synthesized, recrystallised, dried and used for preparing a 0.1% solution in methanol. Solutions of ions for interference studies were prepared by dissolving the amount of each compound needed to give 200 ppm of the ion concerned. All the solutions were stable for several weeks.

PROCEDURE FOR THE EXTRACTION

1.0 cm³ of aqueous solution containing 2 μ g of Iron metal and 1 cm³ of reagent were mixed in a 50 cm beaker. The pH of the solution adjusted to 5.0 It must be noted that the total volume should not exceed 10 cm³. The solution was transferred to 100 cm³ reparatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was passed through anhydrous Sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 cm³ measuring flask and made up to the mark with organic solvent if required. The amount of Iron present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 370 nm.

RESULTS AND DISCUSSION

The results of various parameters involved during the extraction are discussed below with all different parameters.

EXTRACTION AS A FUNCTION OF pH

The optimum pH range in which the metal complex shows maximum and constant absorbance was carried out at different pH range. The maximum extraction was obtained in pH range 4.0 - 6.0 and for all further extractions the pH was maintained at 5.0 (Fig no.1).

spectrophotometric study, all the measurements were carried out at 370 nm.

EFFECT OF SOLVENTS AND EQUILIBRATION TIME

The absorbance value and the percentage of extraction of Fe (III) - APDHTS decreased in the order n-butanol (99.3%) > Cyclohexanone (93.2%) > benzene (91.2%) > chloroform (76.2%) > carbon tetrachloride (74.5%) > amyl alcohol

(63.6%). It was observed that the extraction was quantitative in 1 minutes of equilibration. Hence to get reproducible results 1 minutes of equilibration was carried out for all extractions.

EFFECT OF REAGENT CONCENTRATION ON COMPLEX AND STABILITY OF THE COMPLEX

The minimum amount of reagent to acquire maximum colour intensity with a given amount of Fe (III) ion was found that 1 cm³ of the reagent is sufficient for colour development. The absorbance of the complex is stable even up to 48 hrs.

APPLICABILITY OF BEER'S LAW

A linear plot was obtained when the measured absorbance values are plotted against the amount of Fe (III) in the concentration range of 1-10 ppm at 370 nm (Fig.no.2). The molar absorptivity and Sandal's sensitivity of the complex is 0.4469 x 10³ L mol⁻¹cm⁻¹ and 0.0125 µg cm⁻² respectively.

THE NATURE OF THE EXTRACTED SPECIES

The composition of the complex responsible for the observed Violet colour in the reaction between the metal ion and the APDHTS is established from the Job's method, mole ratio method and slope ratio method was found to be 1:1 (M:L) (Fig no.3)

EFFECT OF DIVALENT IONS AND FOREIGN IONS

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10

ppm of Iron. The ions which show interference in the spectrophotometric determination of Iron were overcome by using appropriate Masking agents. (Table no 1).

PRECISION AND ACCURACY

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 8 µg of Iron in 10 cm³ the aqueous phase. The average of ten determinations was 7.994 and variation from mean at 95% confidence limit was ± 0.0165.

LIMIT OF DETECTION

1 cm³ of 0.1 % methanolic APDHTS is diluted with buffer of pH 5.0 which is then extracted with n-butanol as per the procedure and then its absorbance is measured between 370 nm by taking solvent as a blank. The process is repeated for five times separately and five observations are obtained. The limit of detection for Iron for the method was found to be 0.66 ppm.

APPLICATION

The proposed method was successfully applied for the determination of Iron from various alloys, ores and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method (Table 2).

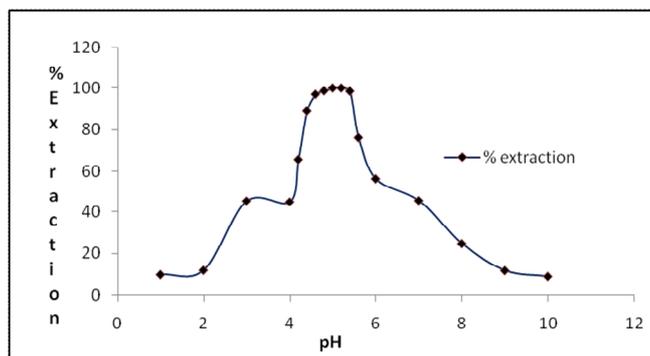


Fig No.1. Function of pH

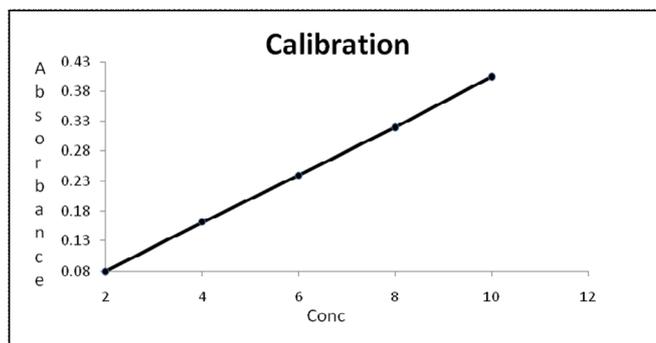


Fig.No.2 Beer's Lambert's Range

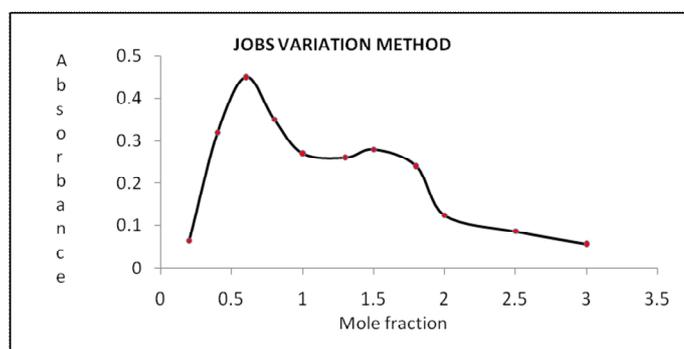


Fig. No. 3 Job's Continuous Variation Method

Table No. 1. Masking agents used

Sr. No.	Interfering Ion	Masking agent
1	Ni (II)	Sodium cyanide
2	U(VI)	8-hydroxy quinoline
3	Cu (II)	Sodium thiosulphate
4	Mo (III)	Citrate
5	Ag (I)	Potassium iodide
6	V (V)	Thiourea
7	EDTA	Boiled with concentrated HNO ₃
8	CN ⁻	Boiled with concentrated HNO ₃ and formaldehyde

TABLE.2. OBSERVATION TABLE FOR DETERMINATION OF Fe (III) USING APDHTS

Sr. No.	Samples	Amount of Fe (III) by standard method in $\mu\text{g}/\text{cm}^3$	Amount of Fe (III) by present method in $\mu\text{g}/\text{cm}^3$
Alloy/ Ore			
a)	Haematite	35.0	34.84
b)	Steel	67.2	67.17
Capsules / Tablet			
a)	Injection Viol	50	49.75
b)	HB-UP Tablet	1.5	1.49
Synthetic mixture			
a)	Fe (III) (5) +Zn (II) (5)	4.98 ppm	4.956 ppm
b)	Fe (III) (5) +Mg (II) (5)	4.98 ppm	4.96 ppm

CONCLUSION

The proposed method permits the determination of trace amounts of Iron without any prior separation. The major advantage of the proposed method is that the colour development is instantaneous at room temperature and stable. The proposed method has higher sensitivity and selectivity than that of the methods reported earlier.

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