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NOTE

Extractive Spectrophtometric Determination of Iron(II) with *p*-Methylisonitrosoacetophenone

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p-Methylisonitrosoacetophenone (MINAP) extracts iron(II) quantitatively (99.5%) into chloroform from an aqueous solution of pH 7.5–8.5. The chloroform extract shows an intense peak at 650 nm (λ_{max}). Beer's law is obeyed over the Fe(II) concentration range 0.1–10 μg/mL. The molar absorptivity is 6515 L mole^{-1}cm^{-1} at 650 nm. The composition of extracted species is found to be 1 : 2 (Fe : MINAP) by Job's continuous variation and mole ratio methods. The interference by various ions has heen studied. The proposed method has been applied to determination of Fe(II) in pharmaceutical sample (Cap. Dexorange)

Key Words: Extractive, Spectrophotometric determination, Iron(II), *p*-Methylisonitrosoacetophenone.

Various reagents[1] are available for the spectrophotometric determination of Fe(II) of which isonitrosoketones constitute an important class.[2] *p*-Methylisonitrosoacetophenone (MINAP) has been used for extraction and spectrophotometric determination of many metal ions[5–7]. In the present communication, we descrihe the extraction and spectrophotometric determination of Fe(II) with MINAP.

Carl-Zeiss VSU and Spectronics-20 spectrophotometers with optically matched quartz and glass cells of 1 cm path length respectively were used for absorbance measurement. An ELICO LI 120 pH-meter was employed for pH measurements. The reagent (MINAP) was synthesized by the procedure recommended by Muller and Pechmann[4] and its solution was prepared in ethanol (1 : 1). A stock solution of Fe(II) was prepared by dissolving ferrous ammonium sulphate in distilled water containing, dilute sulphuric acid; it was standardized by Ferric oxide method.[3] Working solutions of Fe(II) were made by suitable dilution. All other reagents useds were of AR grade and all the solution were prepared in doubly distilled water.

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pH of the solution was adjusted to 8.0 with dilute solution of NaOH and/or HCl, keeping the total volume of solution to 10 mL. The solution was then equilibrated for 2 min with 10 mL of chloroform and the phases were allowed to separate. The chloroform extract was collected in a 10 mL measuring flask and made up to the mark with chloroform. The absorbance of chloroform extract was measured at 650 nm against a reagent blank prepared under identical conditions. The Fe(II) content of the sample solution was determined from calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH.

Determination of Fe(II) in pharmaceutical sample

0.1–0.2 g sample of pharmaceutical sample (cap. Dexorange) was dissolved in boiling with 10 mL of aqua regia containing 2–3 mL of perchloric acid. The solution was evaporated to dryness and the residue was dissolved in 5 mL of dilute HCl. The resulting solution was diluted to 100 mL with distilled water. To an aliquot of this solution (1 mL) analysed for Fe(II) by the procedure as decribed earlier.

Fe(II) could be extracted by MINAP into chloroform from an aqueous solution of pH 7.5 to 8.5 containing 2 mL of 0.5 M sodium thiosulphate.

The organic solvents used for extraction of Fe(II) can be arranged on the basis of their extraction coefficient values as chloroform > ethyl acetate > toluene > benzyl alcohol > carbon tetrachloride > isobutanol > nitromethane > benzene > nitrobenzene. Chloroform was found to be the best extracting solvent, hence, it was selected for extraction throughout the work. The chloroform extract of Fe-MINAP complex showed an intense peak at 650 nm. The absorbance due to the reagent is negligible at this wavelength; so the absorption measurements were taken at this wavelength. The result shows that the system conformed to Beer's law at this wavelength over a Fe(II) concentration range 0.1 to 10 μg/mL.

The molar absorptivity of the extracted complex on the basis of Fe(II) content was calculated to be 6515 L $mole^{-1}cm^{-1}$. It was found that 1 mL of 1% ethanolic solution of MINAP was sufficient to extract 100 μg of Fe(II). The colour of the chloroform extract was found to be stable for at least 48 h at room temperature.

Effect of other ions: Fe(II) (50 μg) was determined in the presence of various ions. The following ions, in the amount indicated, did not interfere in the spectrophotometric determination of Fe(II) (50 μg): 10 mg of each of Ca(II), Mg(II), Al(III), Li(I), Mo(VI), Ba(II) W(VI), Tl(I), Se(IV), Ir(IV), V(V), Bi(III), As(III), Sb(III), Pt(IV); 0.5 mg of Rh(III); 0.01 mg of Ni(II). 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphite, thiosulphate, nitrate, nitrite, phosphates; acetate and 10 mg of pyrophosphate. Interference due to citrate, tartrate and oxalate could be removed by adding sodium molybdate. In the presence of sodium fluoride, metal ions like Mn(II), Zn(II) U(VI), Th(IV), Zr(IV), Ce(IV) and Be(II) did not interfere. Pd(II) and Ru(III) did not interfere in the presence of thiourea. Similarly Cd(II), Ag(I) and Hg(II) did not interfere in the presence of potassium iodide. Cr(III) and Cu(II) could be masked by ammonium acetate and sodium thiosulphate respectively.

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Composition of the Extracted Complex

The composition of the extracted complex was found to be 1 : 2 (Fe : MINAP) by Job's continuous variation and mole ratio methods.

Precision, accuracy, sensitivity and application of method

The precision and accuracy of the spectrophotometric method were tested by analyzing the solution containing a known amount of Fe(II) following the recommended procedure. The average of 10 determinations of 10 μg of Fe(II) in 10 mL solution was 9.86 μg which is varied between 9.78 μg and 10.14 μg at 95% confidence limit and the standard deviation was ±0.39. The proposed method has been applied to the determination of Fe(II) in pharmaceutical sample. The results of analysis of the samples were comparable with those obtained by the thiocyanate method[3] (Table-1).

TABLE-1
DETERMINATION OF IRON IN PHARMACEUTICAL SAMPLE

Sample	Fe(II) found*%	
	Present method	Thiocyanate method[3]
Dexorange-capsule	43.979	43.980

*Average of three determinations.

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