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NOTE

p-Methylisonitrosoacetophenonehydrazone as an Extractive Agent for the Spectrophotometric Determination of Nickel(II)

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A simple and rapid extractive spectrophotometric method for the determination of Ni(II) is described. The complex formed between Ni(II) and *p*-methylisonitrosoacetophenonehydrazone is extracted into chloroform from aqueous solution at pH 7.5. The extracted species has an absorption maxima at 480 nm and obeys Beer's law over a range of 0.2–20 $\mu g\ mL^{-1}$ of Ni(II); the molar absorptivity being $4.87 \times 10^3\ L\ mole^{-1}\ cm^{-1}$. Interference from the foreign ions has also been studied.

Spectrophotometric methods coupled with a separation techniques like solvent extraction can be advantageously applied for the determination of metals at low concentration. Hydrazoxies[1,2] have been used for the extraction and spectrophotometric determination of metals at trace levels.

p-Methylisonitrosoacetophenonehydrazone (HIMAPH) forms brown coloured complex with Ni(II). The present work describes at method for the extractive spectrophotometric determination of Ni(II) in aqueous solution using HIMAPH as a reagent and chloroform as the extracting solvent.

A Backman DU-2 Spectrophotometer with optically matched quartz cells of 1 cm optical path length was used for absorbance measurements and pH values were determined with ELICO-LI 120 pH meter.

All chemical and solvents used were of analytical reagent grade. A stock solution of nickel having a strength of 1 mg/mL was prepared by dissolving Ni(II) sulphate (AR) in sulphuric acid and standardising as dimethylglyoximate[3]. Working solutions of nickel were prepared using double distilled water.

The reagent HIMAPH was synthesized as per the procedure reported by Day[4] for the preparation of phenylglyoxalhydrazoxime.

Solutions of diverse ions were prepared from their chlorides, nitrates or sulphates; or sodium, potassium or ammonium salts using double distilled water.

Procedure: An aliquot of nickel solution containing 0.1–100 $\mu g\ mL^{-1}$ of nickel was treated with 2 mL of 0.5 M sodium acetate and 1 mL of 1% ethanolic solution of HIMAPH and the pH of the solution was adjusted to desired value using dilute solutions of NH_4OH and/or HNO_3. The resulting mixture was equilibrated for 2 min with 10 mL of chloroform. The organic layer was separated and its absorbance was measured at 480 nm against a reagent blank prepared under idential conditions.

Amounts of nickel in unknown solutions were determined from the standard calibration curve. To study the interference, the respective foreign ions were added to the aqueous phase before the extraction and pH adjustment.

Nickel can be quantitatively extracted by HIMAPH into chloroform from an aqueous solution of pH 7.4–8.0. The organic solvents can be arranged in the following order on the basis of their extraction coefficient values:

chloform > diethylether > isobutanol = benzene > *n*-butanol > toluene > ethyl methyl ketone = carbon tetrachloride > ethyl acetate > isoamyl alcohol > nitrobenzene.

The absorption spectra of Ni-HIMAPH complex in chloroform show an absorption maximum around 480 nm and the Beer's law is obeyed over the concentration range 0.2–20 $\mu g\ mL^{-1}$ of nickel. Molar absorptivity of the extracted species is 4.87×10^3 L $mole^{-1}$ cm^{-1}. 1 mL of 1% ethanolic solution of HIMAPH was adequate for complete extraction of 50 μg of nickel and the colour of the complex was found to remain stable for 72 h at room temperature.

Effect of the foreign ions: Nickel (50 μg) was determined in the presence of various ions. The following ions, when present in amounts indicated do not interfere in the spectrophotometric determination of nickel:

10 mg each of Na(I), K(I), Cd(II), Sr(II), Mn(II); 5 mg each of Li(I), Be(II), Ba(II), Ca(II), Mg(II), Hg(II), Pb(II), Zn(II), Al(III), Bi(III), As(III), Se(IV), V(V), Mo(VI), W(VI); 10 mg each of oxalate, phosphate, acetate, sulphate, thiosulphate, fluoride; 20 mg each of persulphate, sulphite, chloride, iodide, bromide, nitrate, nitrite, chlorate, bromate, cyanate, iodate, pyrophosphate, perchlorate and thiourea.

The interference by the ions Ag(I), Fe(II), Cu(II), Pd(II), Fe(III), Zr(IV), Th(IV), Ce(IV), U(IV), cyanide, citrate, tartarate and EDTA can be removed using masking agents (Table-1).

TABLE-1
MASKING AGENTS REQUIRED TO SUPPRESS THE INTERFERENCE OF FOREIGN IONS

Interfering ions	Masking agents added
Ag(I)	Potassium iodide
Cu(II)	Sodium thiosulphate
Pd(II)	Thiourea
Fe(III)	Thiourea
Zr(IV), Th(IV), Ce(IV), U(VI)	Sodium fluoride
Cyanide	Boiling with HNO_3 and formaldehyde
Citrate	Sodium molybdate
Tartarate	Sodium molybdate
EDTA	Boiling with HNO_3

The composition of the extracted species was found to be 1 : 2 (Ni : HIMAPH) by Job's continuous variation method and mole ratio method.

Thus, present method proves to be very rapid and simple and provides an excellent the revovery of nickel in presence of most common ions.

The method was found to be quite suitable for the determination of nickel in alloys such as brass and steel. The results of the analysis were found to be in good agreement with those obtained by dimethylglyoxime method[3] (Table-2).

TABLE-2
ANALYSIS OF ALLOY SAMPLES

S. No.	Alloy	Nickel Found %	
		Present method average	DMG method average
1.	High speed steel	3.95	3.93
2.	Cupro-nickel	22.15	22.17

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