

# Solvent extraction of Nickel (II) from hydrochloric acid media using DMABIMTT

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## Abstract

The extraction of Nickel(II) from hydrochloric acid media by 4-(4-dimethylaminobenzylideneimino)-5-methyl-4H-1, 2, 4-triazole-3-thiol (DMABIMTT) in chloroform has been studied. Effect of acid concentration, reagent concentration, effect of diverse ions, effect of diluents, aqueous to organic phase ratio, stripping agents and loading capacity were investigated for quantitative extraction of Nickel (II). Nickel (II) was selectively extracted and separated from various metals ions and synthetic mixtures. The method has been applied to separate and estimate Nickel (II) from various commercial samples. The proposed method is simple rapid and selective.

**Keywords:** Nickel (II), Acid media, Solvent extraction, DMABIMTT, Synthetic mixtures.

## Introduction

Solvent extraction technique is a part of analytical chemistry and has been recognized as an excellent separation method because of its ease, simplicity, speed, and wide scope [1-3]. Utilizing apparatus no more complicated than a separatory funnel, requiring just several minutes, at the most to perform, applicable both to trace and macrolevels of metals, extraction procedures offers much to the analytical chemist. A further advantage of the extraction method over the widely used precipitation method lies in the cleaner separations that can be achieved by the former.

Solvent extraction or liquid-liquid extraction by high molecular weight organic amines and Schiff bases has become increasingly popular in recent years in studying metal complexes. The extent of extraction by the organic bases depends on their nature, structure, size, concentration and the nature of the organic solvent used as diluent. The extraction of many metal ions from various aqueous solutions by high molecular weight amines and Schiff bases have been reviewed by Khopkar [4] and Green [5,6]. Solvent or liquid-liquid extraction is based on the principle that solute can distribute itself in a certain ratio between two immiscible solvents [7]. Various extractants used for liquid-liquid extraction of many metals from different aqueous solutions are listed as: Aliquat 336 (tricapryl methyl ammonium chloride)[8], Amberlite LA-1 (N-dodecyl trialkyl methyl amine)[9], LA-2 (N-lauryl trialkyl methyl amine), n-octyl aniline [10], TOA (tri-n-octyl amine)[11], Triphenyl phosphene oxide [12]. Different diluents used in nickel extraction are n-hexane, chloroform [13], cyclohexane [14], xylene [15], toluene [16,17], kerosene[18], n-heptane [19]. Nickel is a hard, silvery-white metal which can be combined with other metals, such as iron, copper, chromium, and zinc, to form alloys. These alloys are used to make coins, jewelry, and items such as valves and heat exchangers. Most nickel is used to make stainless steel [20]. Other uses of nickel are in electroplating and batteries and as a catalyst [21].

### **Instrumentation and chemicals**

A Elico Visible spectrophotometer (SL 171) with 1 cm cells was used for measurement, pH measurements were carried out with an Elico digital pH meter model LI120( $\pm 0.01$ ). All the chemicals used were of analytical grade. Double distilled water was invariably used throughout the measurements. A stock solution of Nickel(II) was prepared by dissolving 1g of Nickel chloride hydrate in dilute analytical reagent grade hydrochloric acid (1M) diluted to 100 ml with distilled water and standardized gravimetrically. A working solution of 200  $\mu\text{g ml}^{-1}$  of Nickel(II) was prepared by diluting the stock solution with distilled water. Other standard solutions of different metal ions used to study the effect of foreign ions were prepared by dissolving weighed quantities of respective salts in distilled water or dilute hydrochloric acid. Recommended method An aqueous solution containing 200 $\mu\text{g}$  Nickel(II) mixed with a sufficient quantity of 1M HCl to make its concentration. Then the pH of the solution was adjusted to 1.0 using dilute hydrochloric acid and sodium hydroxide. The solution was transferred into a 125 ml separating funnel and shaken with 10 ml of 0.1M DMABIMTT in chloroform for just 30 seconds. After equilibration, the mixture

was allowed to separate and the metal was stripped from the organic phase. The extracts were evaporated to moist dryness. The residue was dissolved in minimum amount of 1M hydrochloric acid and Nickel (II) was determined spectrophotometrically.

### **Results and discussion:**

#### **Effect of acidity:**

The extraction of Ni(II) was carried out from different organic acids such as Hydrochloric acid, salicylic acid, tartaric acid, acetic acid. Quantitative results were obtained in the hydrochloric acid media at pH 1.0. While in Sulphuric acid, salicylic acid and acetic acid very less extraction was found. Hence the hydrochloric acid is used for further studies. (Table.1)

#### **Effect of diluents:**

The extractions were performed from hydrochloric acid medium using 0.1M DMABIMTT in various solvents as diluents. Different diluents used in nickel extraction are n-hexane, chloroform [13], cyclohexane [14], xylene [15], toluene [16,17], kerosene[18], n-heptane [19]. Chloroform provide a higher partition (i.e., a better extraction) than did the other. Separation factor strongly dependent on the diluent, While a poor separation was achieved when using xylene and CCl<sub>4</sub> as diluent, resulted in a good separation of Ni(II). For a better result in the liquid-liquid extraction of nickel using such Schiff bases as DMABIMTT, diluent properties have to be consulted.

#### **Effect of concentration of extractant (DMABIMTT):**

In order to optimize the conditions for extraction of Ni(II), chloroform solutions of DMABIMTT with varying concentration (0.01- 0.20 M) were employed. It was found that 10 ml of 0.06 M DMABIMTT was sufficient for quantitative extraction of 200µg Ni(II) from hydrochloric acid media, but in recommended procedure 0.1 M DMABIMTT in chloroform was used to ensure the complete extraction of metal ion. There was no adverse effect if one can use excess of extractant. However, a decrease in concentration of extractant resulted in lower distribution ratio, D values for Ni(II) that is less extraction.

**Effect of Time on Extraction:**

The effect of time was observed on the system for a period of 5s to 30min (hand shaking) the extraction was found quantitative over the periods longer than 30 seconds. But to ensure the complete extraction of Ni(II) 1 min equilibration time was recommended. However, a prolonged shaking period doesn't have any adverse effect on the extraction.

**Effect of divers ions:**

Nickel(II) was extracted in the presence of a large number of foreign cations and anions (Table1). The tolerance was set at the amount of the foreign ion that could be present to give an error less than  $\pm 2\%$  in the recovery of Nickel(II)). The results showed that in the extraction and determination 200 $\mu\text{g}$  of the Nickel (II), these ions has no significant effect. The reproducibility of Nickel (II) extraction investigated from six replicate measurements. (Table 2)

**Application:**

Binary separation of Nickel (II) from iron (III), cobalt (II) and copper (II) The method allowed separation and determination of Nickel (II) from a binary mixture containing either iron (III), cobalt (II) and copper (II). In a typical experiment, solution containing 200 $\mu\text{g}$  of Nickel (II) was taken and known amount of other metal were added. Nickel (II) was estimated spectrophotometrically. The recovery of Nickel (II) and that added ions was 99.5% and results are reported in (Table 3).

**Analysis of synthetic mixtures**

The separation of Nickel (II) from other metals that is Cobalt, Iron, Copper was carried out by taking advantage of differences in their optimum extraction and stripping conditions. The proposed method was successfully used in the determination of Nickel (II) from different synthetic mixtures (Table: 3). A solution containing 200 $\mu\text{g}$  of Nickel (II) was taken and known amount of other metals were added. Under the optimum extraction conditions of Nickel (II), there is a quantitative extraction of Ni (II), Cu(II), Fe(II) and Co(II) but the co-extracted metal ions cannot be back-stripped by 1M hydrochloric acid solution. Thus, the reagent (DMABIMTT) is made selective towards Nickel (II) by taking an advantage of the stripping agent used. (Table 4)

### Analysis of alloys

To ascertain the selectivity of the reagent, the proposed method was successfully used in the determination of Nickel (II) in alloys. The synthetic mixtures were prepared corresponding to the composition of alloy. The results of the analysis are reported in (Table 4). The average recovery of Nickel (II) was 99.5%.

### Conclusion

Nickel recovery from secondary sources is necessary from view point of both environmental protection and to meet increasing demand of it. Nickel is generally available at lower pH values for extraction. The present work points out that the synthesized extractant (DMABIMTT) shows a good potential for the extraction of Nickel (II) from hydrochloric acid media. The extraction time is short just 30 seconds and the extractant presents a good loading capacity and reusable. The proposed method is used for rapid and selective separation of Nickel (II) from associated ions in their binary mixtures, synthetic mixtures and alloys.

**Table:1 Extraction in different acids media**

<b>Acid media</b>	<b>% Extraction</b>
Hydrochloric acid	99
Acetic acid	80
Nitric acid	90
Sulphuric acid	70

**Table:2 Effect of various diverse ions on percentage extraction of Nickel(II)**

Ions added	Added as	Tolerance limit (mg)	Ions added	Added as	Tolerance limit (mg)
Ca(II)	CaCl <sub>2</sub>	20	Fe(III)	NH <sub>4</sub> Fe(SO <sub>4</sub> ) <sub>2</sub>	15
Co(II)	CoCl <sub>2</sub> · 6H <sub>2</sub> O	10	Hg(II)	HgCl <sub>2</sub>	15
Cd(II)	CdCl <sub>2</sub> · 2H <sub>2</sub> O	15	Mn(II)	MnCl <sub>2</sub> · 6H <sub>2</sub> O	10
Cr(III)	CrCl <sub>3</sub>	15	Zn(II)	ZnSO <sub>4</sub> · 7H <sub>2</sub> O	15
Mo(VI)	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·2H <sub>2</sub> O	20	Hg(II)	HgCl <sub>2</sub>	15
Cu(II)	CuSO <sub>4</sub> · 5H <sub>2</sub> O	5	Mn(II)	MnCl <sub>2</sub> · 6H <sub>2</sub> O	10

**Table 3 Binary separation of Nickel (III) from iron(III), cobalt(II) and copper(II)**

Composition of metal ions, (µg)	Recovery Ni(II) (%)	R.S.D (%)	Added metal ions* (%)	R.S.D (%)
Ni(II)100,Co(II)10000	99.5	0.19	99.6	0.13
Ni(II)100,Cu(II)5000	99.6	0.25	99.5	0.16
Ni(II)100,Fe(II)5000	99.5	0.17	99.6	0.19

**Table 4 Analysis of synthetic mixture**

Composition (µg)	Nickel found (µg)	Recovery (%)	R.S.D*
Ni(II) 200+Cu(II)500+Zn(III)200	99.5	99.9	0.06
Ni(II) 200+Fe(II)500+Co(III)200	99.4	99.7	0.07
Ni(II) 200+Cr(IV)500+Fe(III)200	99.4	99.7	0.07
Ni(II) 200+Cu(IV)500+Cr(II)200+Co(III)200	99.5	99.6	0.06

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