

# Selective and sensitized spectrophotometric determination of trace amounts of Ni(II) ion using $\alpha$ -benzyl dioxime in surfactant media

Mehrorang Ghaedi\*

Chemistry Department, Yasouj University, Yasouj 75914-353, Iran

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## Abstract

Highly sensitive and interference-free sensitized spectrophotometric method for the determination of Ni(II) ions is described. The method is based on the reaction between Ni(II) ion and benzyl dioxime in micellar media in the presence of sodium dodecyl sulfate (SDS). The absorbance is linear from 0.1 up to 25.0  $\mu\text{g mL}^{-1}$  in aqueous solution with repeatability (RSD) of 1.0% at a concentration of 1  $\mu\text{g mL}^{-1}$  and a detection limit of 0.12  $\text{ng mL}^{-1}$  and molar absorption coefficient of 68,600  $\text{L mol}^{-1} \text{cm}^{-1}$ . The influence of reaction variables including type and amount of surfactant, pH, and amount of ligand and complexation time and the effect of interfering ions are investigated. The proposed procedure was applied to the determination of trace amounts of Ni(II) ion in tap water, river water, chocolate and vegetable without separation or organic solvent extraction. © 2006 Elsevier B.V. All rights reserved.

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## 1. Introduction

The determination of heavy metals in environmental samples such as water samples is a task for analytical chemists frequently asked by environmentalists, for the evaluation and phenomena interpretation of aquatic systems. There is a growing interest in nickel determination. Nickel is moderately toxic element as compared with other transition metals. However, it is known that inhalation of nickel and its compounds can lead to serious problems, including respiratory cancer [1]. Moreover, nickel can cause a skin disorder known as nickel–eczema [2]. The determination of nickel is important due to its toxic nature and its presence in industrial wastes and various other effluents. Nickel is present in low concentrations in raw meat, chocolates, hydrogenated oils, milk and milk products and canned food, etc. Cancer of nasal cavity and lungs, dermatitis, asthma, acute pneumonitis and disorders of central nervous system may be caused by nickel toxicity. The direct determination of traces of this ion from natural waters is limited and difficult when its concentration is too low to be determined directly and/or interference due to the matrix cannot be eliminated. In most cases pretreatment of samples, physical separation and non-universal instrumentation are required.

Due to low level of this element for their simple and ease spectrophotometric determination in various biological and industrial sample an aggregation and solubilization is required, that can be achieved using surfactants [3]. Micellar systems are convenient to use because they are optically transparent, readily available and stable [4]. In the field of metal ion complexation, at concentrations below or above the critical micelles concentration (CMC), micelles form a ternary complex with advantageous properties, such as hyperchromic and bathochromic displacements, that can modify sensitivity of the method by affecting the interferences and matrix effects [5]. The ability of micellar system to solubilize slightly insoluble or even very insoluble complexes and/or ligands has been used to enhance the analytical merit of given methods [6–8]. The ability of micelles to solubilize complexes in aqueous solution can eliminate the need for non-aqueous extraction step in a given analysis [6,9,10], which reduces the cost and toxicity of the method. Since, organic ions and molecule can bind the surfactant assemblies by electrostatic and hydrophobic interaction, therefore methods based on surfactants lead to modification and improving sensitivity, which emerged from the fact that non-polar part of solute molecules has a strong interaction with the exposed hydrocarbon chains of the surfactant and lead to improvement in method characteristics performance.

Consequently, many analytical procedures have been proposed for nickel determination, such as voltammetry [11,12],

\* Tel.: +98 741 2223048; fax: +98 741 2223048.  
E-mail address: [m.ghaedi@mail.yu.ac.ir](mailto:m.ghaedi@mail.yu.ac.ir).

on-line preconcentration [13], ion selective electrode [14,15], polarography [16]. Many of these methods are time consuming or require complicated and expensive instrument and some of them has low repeatability and need more care for sensor preparation.

However, the spectrophotometric method still has the advantages simplicity and requires no expensive or complicated test equipments. For this reason, a wide variety of spectrophotometric method for the determination of nickel have been reported [17–33]. Each chromogenic reagent has its advantages and disadvantages with respect to sensitivity, selectivity and rapidity. The most widely used reagents for nickel determination have been the oxime compounds, containing the group  $-C(\equiv NOH)-$ , which reacts selectively with nickel. A large number of oxime is used for spectrophotometric determination of trace amount of nickel ion [27,31].

Due to the well known effect of surfactants in extraction system which emerged from their ability in phase separation, solvent extraction, especially in surfactant media have been successfully applied in the extraction, preconcentration and purification of many species especially in the separation of metal ions [34–36]. Thus, analytical methods based on sensitized spectrophotometric reagents are often sufficiently sensitive to permit measurements in the nano to micro molar range and can exhibit good sensitivity emit an intense absorbance peak, allowing trace ion determination by conventional spectrophotometry.

In the present work a simple and highly selective and sensitive spectrophotometric method for determination of nickel(II) ions using BDO in surfactant media (SDS) was established. The effect of various parameters such as pH, type and amount of surfactant and amount of ligand were examined. Time dependency of complex and effect of interference of other metal ions were evaluated. The method has wide linear range, low detection limit, high sensitivity and selectivity and high repeatability that successfully have been used for determination of Ni(II) ion content in real samples.

## 2. Experimental

### 2.1. Instrumentation

A Shimadzu UV–vis 160 spectrophotometer was used to measure the absorbance of complex in SDS media. To adjust the pH and prepare the buffer solution a 691 pH/ion meter with a combined glass and calomel electrode has been used. The Ni(II) ion determinations was carried out on a Perkin-Elmer 603 atomic absorption spectrometer with a hollow cathode lamp and a deuterium background corrector, at an air–acetylene flame under the recommended conditions.

### 2.2. Reagent and solution

All chemicals such as nitrate of Ni(II) ion and other cation were of the analytical grade purchased from Merck Company. A 0.5% (w/v) all surfactant all from Merck company including sodium dodecyl sulfate (SDS), Triton X-100, Brij 58, cetyltrimethylammonium bromide (CTAB), *n*-

dodecyltrimethylammonium bromide (DTAB) was prepared by dissolving 0.5 g of surfactant in 100 mL volumetric flask with stirring. The ligand benzyl dioxime (BDO) was purchased from Merck company and used without further purification.

### 2.3. Procedure calibration curve

Standard Ni(II) solutions were prepared in the range of  $0.05\text{--}30.0\ \mu\text{g mL}^{-1}$ . Several aliquots of Ni(II) ion were added to 10 mL volumetric flask, and 0.8 mL of 0.1 M BDO and 0.6 mL of 0.07 M SDS were added to each flask, then 2 mL 0.01 M NaOH was added and filled to the mark and calibration curve of Ni(II) was constructed using a UV–vis 160 spectrometer.

### 2.4. Pretreatment of water samples

Analysis of water samples for determination of Ni(II) ion content was carried out as follows: 250 mL of river water or spring water was poured in a beaker and 30 mL concentrated  $\text{HNO}_3$  and 10 mL of  $\text{H}_2\text{O}_2$  of (30%) for elimination and decomposition of organic compound were added. While stirring, it heated to reach its volume to one tenth. After adjustment of samples pH to desired value the spectrophotometric experiment was performed according to general described procedure. A synthetic sample was prepared.

### 2.5. Chocolate pretreatment

A 20 g chocolate sample was ashed in silica crucible for 4 h on a hot plate and the charred material was transferred to furnace for overnight heating at  $450\ ^\circ\text{C}$ . The residue was cooled and treated with 10.0 mL concentrated nitric acid and 3 mL 30%  $\text{H}_2\text{O}_2$  again kept in furnace for 2 h at the same temperature so that no organic compound traces are left. The final residue was treated with 0.5 mL concentrated hydrochloric acid and 1–2 mL 70% perchloric acid and evaporated to fumes, so that all the nickel metal changes to nickel ions. The solid residue was dissolved in water and filtered. By keeping the pH at 12 by addition of KOH the spectrophotometric procedure has been carried out. The dissolved solution was suitably diluted and metals concentrations were determined with UV–vis spectrophotometer.

### 2.6. Soil sample pretreatment

Homogenized soil sample 20 g was weighed accurately and in a 200 mL beaker was digested in the presence of an oxidizing agent, following the method recommended by Jacson [36] then 10 mL Concentrated  $\text{HNO}_3$  and 2 mL  $\text{HClO}_4$  70% was added and heated for 1 h. The content of beaker was filtered through a Whatman No. 40 filter paper into a 250 mL calibrated flask and its pH was adjusted to desired value and diluted to mark with de-ionized water. In all of real and synthetic sample amount of Ni(II) ion was found by standard addition method.

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