



# Synergistic enhancement effect of room temperature ionic liquids for cloud point extraction combined with UV–vis spectrophotometric determination nickel in environmental samples

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## ABSTRACT

A new method based on enhancement effect of room temperature ionic liquids for cloud point extraction trace amounts of nickel combined with UV–vis spectrophotometric determination was developed. Room temperature ionic liquids (RTILs) and diethyldithiocarbamate (DDTC) were used enhancement reagent and chelating reagent, respectively. The addition of room temperature ionic liquids leads to 3.0 times improvement in the determination of nickel. The nonionic surfactant Triton X-100 was used as the extractant. When the temperature of the system was higher than the cloud point of Triton X-100, Ni-DDTC complex was extracted into Triton X-100 and separation of the analyte from the matrix was achieved. Some parameters that influenced cloud point extraction and subsequent determination were evaluated in detail, such as the concentrations of RTILs, DDTC and Triton X-100; pH of sample solution, as well as interferences. Under optimized conditions, an enrichment factor of 72 could be obtained, and the detection limit (LOD) for Ni was  $0.5 \text{ ng mL}^{-1}$ . Relative standard deviations for five replicate determinations of the standard solution containing  $50 \text{ ng mL}^{-1}$  Ni was 3.9%. The proposed method was successfully applied to the determination of nickel in certified reference materials with satisfactory results.

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## 1. Introduction

Nickel has been the subject of many investigations due to it is essential for living organism but toxic when it is excessive. The concentration of nickel in environmental and biological samples is very low. In order to obtain reliable results, an efficient separation and preconcentration step is usually necessary prior to analysis. Several preconcentration methods including liquid–liquid extraction (LLE) [1,2], solid phase extraction (SPE) [3–5], cloud point extraction (CPE) [6,7], liquid phase microextraction (LPME) [8], Hollow fiber based-liquid phase microextraction [9], ionic liquid extraction [10] and chemical vapor generation (CVG) [11,12] have been reported for nickel analysis.

Room temperature ionic liquids (RTILs) are ionic media resulting from combination of organic cations and various anions that are liquids at room temperature. RTILs have some unique physicochemical properties such as negligible vapor pressure, environment-friendly, non-flammability as well as good extractability for various organic compounds and metal ions [13]. These characteristics make ionic liquids suitable to be used as the

extraction solvent for separation and preconcentration of various organic compounds and metal ions [14–19]. RTILs were also used as enhancement agent on the chemical vapor generation of Au, Ag and Cu [20]. Recently, synergetic enhancement effect of ionic liquid and chelating agent on the chemical vapor generation of Fe, Co and Ni were reported [11,21]. To the best of our knowledge, synergetic enhancement effect of RTILs for cloud point extraction trace amounts of nickel combined with UV–vis spectrophotometric determination has not been reported yet.

The present work was aimed to develop a new nickel analysis method. Synergetic enhancement effect of RTILs for cloud point extraction combined with UV–vis spectrophotometer were used for determination nickel. Traditional cloud point extraction combined with UV–vis spectrophotometric determination nickel only have an enrichment factor of 24 could be obtained. The addition of 1-butyl-3-methylimidazolium hexafluorophosphate ( $[\text{C}_4\text{MIM}][\text{PF}_6]$ ) (RTILs) leads to 3.0 times improvement in the determination of nickel. Some parameters that influenced cloud point extraction and subsequent determination, and interference of coexisting ions were investigated systematically. The proposed method possesses the advantages of high enrichment factor, simplicity, sensitivity, low cost, selectivity and environmental friendship. This method was successfully applied to determination nickel in certified reference environmental samples with satisfactory results.

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## 2. Experimental

### 2.1. Apparatus

A model UV-2550 spectrophotometer (Shimadzu Instrumental Co., Japan) was used for the determination of nickel. A centrifuge Model 80-2 (Changzhou Guohua Scientific Instrument Corporation, Jiangsu, China) was used to assist phase separation. A HH-S thermostated water bath (Jintan, Medical Instrument Corporation, Jiangsu, China) was used to heat up the sample solution. The pH values of samples solution were determined by PHS-3C pH meter (Leici Instruments, Shanghai, China). A 25- $\mu$ L microsyringe (Anting Corporation, Shanghai, China) was used for transferring sample solution.

### 2.2. Standard solutions and reagents

The stock standard solutions of Ni(II) ( $1 \text{ mg mL}^{-1}$ ) were purchased from the National Center for Reference Materials (Beijing, China). Working standard solutions were obtained by serial dilution of the stock standard solutions. All chemicals used were at least of analytical-reagent grade and all solutions were prepared with double distilled water (DDW). Diethyldithiocarbamate (DDTC) was used as the chelating reagent for nickel. RTIL of 1-butyl-3-methylimidazolium hexafluorophosphate ( $[\text{C}_4\text{MIM}]\text{PF}_6$ ) (Chengjie Chemistry Corporation, Shanghai, China) and non-ionic surfactant Triton X-100 (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) were used as cloud point synergic enhancement reagent and extractant, respectively. DDTC, sodium acetate, acetic acid, ethanol, methanol, tetrachloromethane ( $\text{CCl}_4$ ), benzene, acetone, nitric acid and sodium hydroxide (NaOH) used in this experiment were all obtained from Jingmi Chemical Reagent Corporation (Nanning, China)

### 2.3. Sample preparation

The following certified environmental reference materials were purchased from National Standard Material Research Center (Beijing, China), which were employed to validate the accuracy of the proposed method. GBW(E) 080195 (water sample), GBW 08607 (water sample) and GSBZ 50009-88 (water sample) were appropriately diluted with DDW, respectively. Certified reference materials of 0.2004 g soil (GBW 07403), 0.2012 g stream sediment (GBW 07312) and 0.2025 g stream sediment (GBW 07317) were accurately weighed and dissolved with 10 mL aqua regia in digestion vessel, respectively. Microwave digestion was chosen for decomposing the certified reference environmental samples and the digestion procedure was based on a reference method [22]. The residues were transferred into 50 mL centrifuge tubes, appropriate amounts of DDTC, RTILs and Triton X-100 were added and made up to the volume with DDW, then adjusting its pH to 6.0 with acetic acid and NaOH, respectively.

### 2.4. Cloud point extraction procedure

For CPE preconcentration, 50 mL analytical solution containing nickel ion and proper amount of chelating agent DDTC were mixed in 50 mL centrifuge tube. After adjusting the pH 6.0, the proper amounts of Triton X-100 and RTILs were added into the centrifuge tube. After shaking the centrifuge tube, the analytical solution was heated in a thermostated water bath at  $75^\circ\text{C}$  for 15 min. After heated, the resultant solution was subjected to centrifugation at 3500 rpm for 5 min for phase separation, and then cooled in an ice-water bath for 5 min to increase the viscosity of the surfactant-rich phase. The water phase was carefully removed with a syringe, and the surfactant-rich phase was diluted with 1.5 mL

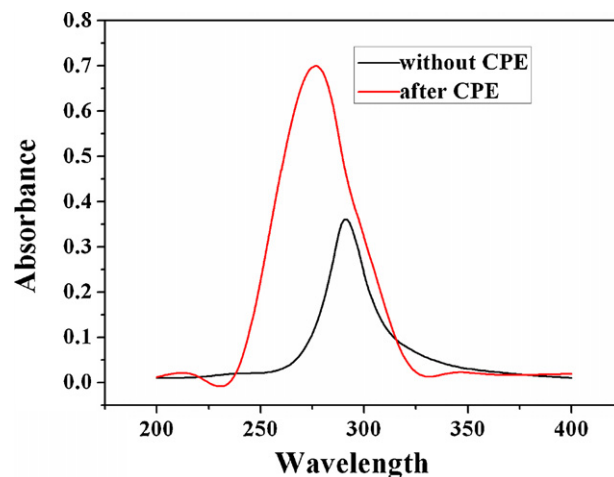


Fig. 1. Absorption spectra for the determination of Ni with CPE. Ni:  $50 \text{ ng mL}^{-1}$ ; DDTC: 0.02% (m/v); Triton X-100: 0.3% (v/v); RTILs: 0.005% (v/v); pH: 6.0; extraction time: 15 min.

ethanol because of the sampling demand of spectrophotometer. The resultant sample solution was transported to spectrophotometer for determination.

## 3. Results and discussion

### 3.1. Study on the absorption spectra of complex

UV-vis spectrophotometry was used to accomplish the determination of nickel. The maximum wavelength of absorption of the complex of Ni-DTC should be found out for the quantification analysis. After CPE, the surfactant-rich phase was diluted with 1.5 mL ethanol and determined in the range of wavelength from 200 nm to 400 nm. The maximum wavelength of absorption of the complex of Ni-DTC after CPE was found in 285 nm. The concentration of  $4000 \text{ ng mL}^{-1}$  Ni(II) was used for Ni-DTC wavelength scan without CPE. The maximum absorption without CPE was 289 nm. It was shown in Fig. 1. The blank absorbance of all reagents was used to correct in the process of the determination of nickel.

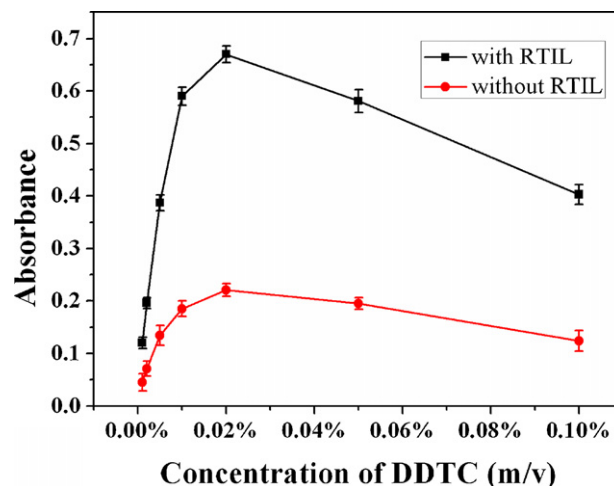


Fig. 2. Effect of concentration of DDTC on the absorbance of Ni in CPE. Ni:  $50 \text{ ng mL}^{-1}$ ; Triton X-100: 0.3% (v/v); RTILs: 0.005% (v/v); pH: 6.0; extraction time: 15 min.

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