

Solid phase extraction and preconcentration of uranium(VI) and thorium(IV) on Duolite XAD761 prior to their inductively coupled plasma mass spectrometric determination[☆]

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Abstract

A simple and effective method is presented for the separation and preconcentration of thorium(IV) and uranium(VI) by solid phase extraction on Duolite XAD761 adsorption resin. Thorium(IV) and uranium(VI) 9-phenyl-3-fluorone chelates are formed and adsorbed onto the Duolite XAD761. Thorium(IV) and uranium(VI) are quantitatively eluted with 2 mol L⁻¹ HCl and determined by inductively coupled plasma-mass spectrometry (ICP-MS). The influences of analytical parameters including pH, amount of reagents, amount of Duolite XAD761 and sample volume, etc. were investigated on the recovery of analyte ions. The interference of a large number of anions and cations has been studied and the optimized conditions developed have been utilized for the trace determination of uranium and thorium. A preconcentration factor of 30 for uranium and thorium was achieved. The relative standard deviation ($N=10$) was 2.3% for uranium and 4.5% for thorium ions for 10 replicate determinations in the solution containing 0.5 µg of uranium and thorium. The three sigma detection limits ($N=15$) for thorium(IV) and uranium(VI) ions were found to be 4.5 and 6.3 ng L⁻¹, respectively. The developed solid phase extraction method was successively utilized for the determination of traces thorium(IV) and uranium(VI) in environmental samples by ICP-MS.

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1. Introduction

Uranium and thorium find extensive application as nuclear fuel in power plants and their main sources are soil, rocks, plants, sand and water [1]. Uranium and thorium are known to cause acute toxicological effects for human and their compounds are potential occupational carcinogens [1]. These elements and compounds are highly toxic which cause progressive or irreversible renal injury. Nuclear spent fuels generally contain actinides like uranium, thorium and various fission products [2]. Due to these importances of uranium and thorium, the determination of uranium and thorium in environmental and biological samples has considerable potential as a tool for assessment of human exposure [3]. The direct determination of

uranium and thorium by the instrumental techniques including inductively coupled plasma atomic emission spectrometry and inductively coupled plasma-mass spectrometry is still difficult because of insufficient sensitivity, lack of selectivity, presence of complex matrix, poor precision and accuracy [4–6]. To solve these problems, enrichment and separation techniques including solvent extraction, coprecipitation, ion-exchange, electrodeposition, etc. [7–11] have been used in the analytical chemistry laboratories for uranium and thorium. Solid phase extraction is one of the important preconcentration-separation procedures for trace heavy metals ions, due to its simplicity and limited usage of the organic solvents [12–17]. Solid phase extraction of uranium and thorium is also a popular subject in the analytical chemistry [18–20]. Shamsipur et al. [21] have proposed a solid phase extraction procedure for ultratrace uranium(VI) in natural waters using octadecyl silica membrane disks modified by tri-*n*-octylphosphine oxide. A chelating resin has been synthesised by Gladis and Rao [22] by the reaction with Amberlite XAD-4 and 5-aminoquinoline-8-ol for the uranyl ion uptake. The determina-

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tion of uranium and thorium in natural waters with a high matrix concentration using solid phase extraction inductively coupled plasma-mass spectrometry has been performed by Unsworth et al. [23]. Metilda et al. [24] have prepared catechol functionalized aminopropyl silica gel for the separation and preconcentration of uranium(VI) from thorium(IV) in soil and sediment samples. Ghasvand and Mottaabed [25] have proposed a solid phase extraction procedure of ultratrace uranium by mixtures of dicyclohexyl-18-crown-6 and tri-*n*-octylphosphine oxide. The selective enrichment and separation of U(VI) and Th(IV) in trace and macroscopic levels using malonamide grafted polymer from acidic matrices has been performed by Raju and Subramanian [26].

In the presented work, a solid phase extraction procedure for the separation-preconcentration of ultratrace U(VI) and Th(IV) as 9-phenyl-3-fluorone chelates on Duolite XAD761 resin has been presented prior to their ICP-MS determination. The effects of analytical parameters including pH of the solution, amounts of 9-phenyl-3-fluorone and Duolite XAD761, sample volume, etc. were investigated.

2. Experimental

2.1. Apparatus

An Agilent model 7500a inductively coupled plasma mass spectrometer was used for the determination of uranium and thorium. The instrument was optimized daily before measurement and operated as recommended by the manufacturers. The conditions are given in Table 1.

A pH meter, WTW Inolab Level 3 Model glass-electrode was employed for measuring pH values in the aqueous phase. The water was purified in Millipore Synergy 185.

2.2. Reagents and solutions

High purity reagents were used for all preparations of the standard and sample solutions. Stock solutions of uranium(VI) and thorium(IV), 1000 mg L⁻¹ (Sigma, St. Louis) was diluted daily for obtaining working solutions. The standard solutions

used for the calibration procedures were prepared before use by dilution of the stock solution with 1 mol L⁻¹ HNO₃. Stock solutions of diverse elements were prepared from the high purity compounds (99.9%, E. Merck, Darmstadt).

A solution of 9-phenyl-3-fluorone (%0.05 m/v) was prepared by dissolving 0.05 g of 9-phenyl-3-fluorone (Sigma, St. Louis, MO, USA) in 1 mL of 0.1 mol L⁻¹ NaOH and diluting to 100 mL with water.

Duolite XAD761 is cross-linked phenol-formaldehyde polymer that was purchased from Sigma Chem. Co., St. Louis, USA. Its surface area is 300 m² g⁻¹. It was used for the removal of proteins, high MW colorants, organic impurities and is useful for the purification of pharmaceuticals [27,28]. A glass column containing 700 mg of Duolite XAD761 in water suspension was 10 cm long and 1.0 cm in diameter. The bed height in the column was approximately 2.5 cm. The resin on the column was preconditioned with pH 5.0 buffer solution prior to percolation of the sample.

2.3. Test studies for solid phase extraction

The presented procedure was checked with test solutions. An aliquot of test solution containing 0.5 μg U(VI) and Th(IV) ions was transferred into a beaker and 0.5 mL of 0.05% (m/v) 9-phenyl-3-fluorone solution was added. The pH of the solution was adjusted to working pH by the addition of diluted NH₃ or HCl. The solution was diluted to 20–30 mL with water and left at room temperature for 10 min for the formation of U(VI) and Th(IV) chelates. The sample solution was permitted to flow through the column under gravity. Then, the column was washed firstly with the blank solution that containing related buffer solution; the retained analytes were eluted with 5 mL of 2 mol L⁻¹ HCl at a flow rate of 4 mL min⁻¹. The metal concentration in the solution was determined by ICP-MS.

2.4. Analysis of real samples

0.1 g of NIST-SRM 2710 Montana I Soil standard reference material was digested with aqua regia (12 mL concentrated hydrochloric acid and 4 mL of concentrated nitric acid) at room temperature then it was heated to 95 °C. After the evolution of NO₂ fumes had ceased, the mixture was evaporated almost to dryness on a sand-bath and mixed with 8 mL of aqua regia. Then the mixture was again evaporated to dryness. After evaporation 8–9 mL of distilled water was added and the sample was mixed. The resulting mixture was filtered through a blue band filter paper. The filtrate was diluted to 25 mL with distilled water. pH of this solution was adjusted to 5 by the addition of 1 mol L⁻¹ NH₃. Then the preconcentration procedure given above was applied.

Water samples analyzed were filtered through a cellulose membrane filter (Millipore) of 0.45 μm pore size. One hundred and fifty millilitres of water sample was transferred to a beaker. Then 0.5 mL of 0.05% (m/v) 9-phenyl-3-fluorone solution was added. The pH of this solution was adjusted to 5.0. After 10 min waiting for complex formation, the sample was passed through the Duolite XAD761 column. The 9-phenyl-3-fluorone chelates

Table 1
Operating conditions of ICP-MS

Inductively coupled plasma	Agilent 7500 a
Nebulizer	Babington
Spray chamber	Quartz, double pass
RF power	1260 W
Frequency	27.12 MHz
Sampling depth	7.0 mm
Plasma gas flow rate	15 L min ⁻¹
Auxiliary gas flow rate	1.0 L min ⁻¹
Carrier gas flow rate	1.15 L min ⁻¹
Sample uptake rate	0.3 mL min ⁻¹
Detector mode	Auto
Integration time	0.10 s
Number of replicates	3
Analytical masses	²³⁸ U, ²³² Th
Internal standard	²⁰⁹ Bi

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