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Separation and enrichment of gold(III) from environmental samples prior to its flame atomic absorption spectrometric determination

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Abstract

A simple and accurate method was developed for separation and enrichment of trace levels of gold in environmental samples. The method is based on the adsorption of Au(III)–diethyldithiocarbamate complex on Amberlite XAD-2000 resin prior to the analysis of gold by flame atomic absorption spectrometry after elution with $1\,\mathrm{mol\,L^{-1}}$ HNO_3 in acetone. Some parameters including nitric acid concentration, eluent type, matrix ions, sample volume, sample flow rate and adsorption capacity were investigated on the recovery of gold(III). The recovery values for gold(III) and detection limit of gold were greater than 95% and $16.6\,\mu\mathrm{g\,L^{-1}}$, respectively. The preconcentration factor was 200. The relative standard deviation of the method was <6%. The adsorption capacity of the resin was $12.3\,\mathrm{mg\,g^{-1}}$. The validation of the presented procedure was checked by the analysis of CRM–SA–C Sandy Soil certified reference material. The presented procedure was applied to the determination of gold in some environmental samples.

Keywords: Solid-phase extraction; Separation and enrichment; Gold; Amberlite XAD-2000; Diethyldithiocarbamate; Flame atomic absorption spectrometry

1. Introduction

Gold, as a global currency, an investment and simply a thing of beauty, is one of the precious metals and held an allure for thousands of years. Gold possesses a unique combination of properties that have resulted in its use in a wide range of industrial applications [1]. Cyanide, as a leaching agent, is commonly used in industrial gold extraction [2,3]. The recovery of gold by the adsorption of auro-cyanide complexes onto activated carbon is a well-established process [4]. However, great interest in the use of non-cyanide methods for the dissolution of gold arises from concerns regarding the toxicity of cyanide and the inability of cyanide solution to effectively leach carbonaceous or complex ores. Also, these conventional industrial processes for recovering precious metals generally consist of multiple steps of dissolution, conditioning and precipitation. These processes are not only labor-intensive but also time-consuming, and hence much work has been conducted on the development of alternative methods [5].

The concentration of gold in environmental, geological and metallurgical materials is usually too low to be determined directly by AAS owing to insufficient sensitivity and matrix interferences. Hence, separation and enrichment of analyte are important aspects in determination of gold in addition to the effect of other trace metal ions in saline matrices [6–9]. For this purpose, several separation and enrichment techniques for gold (and for trace metal ions) including solvent extraction [10–12], co-precipitation [13–15], sorption, [16–20] etc., have been developed. One of this technique, solid-phase extraction (SPE) by Amberlite resins has been widely used for separation and enrichment of almost all the other trace metals, besides gold [21–29].

Sodium diethyldithiocarbamate (Na-DDTC) is a well-characterised reagent that reacts with over many metal ions such as Au, Cu, Fe, Pb, Mn, Zn, Cd, Ni, Bi and Cr to form very stable

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complexes [6,7]. These chelates have been used for the solid-phase extraction of some metal ions from aqueous solutions [6,7,28,29].

In this study, a solid-phase extraction method for gold(III)-diethyldithiocarbamate on Amberlite XAD-2000 resin has been presented. The conditions including nitric acid concentration, amounts of reagents, eluent type, sample volume for the quantitative recoveries for gold(III) were investigated.

2. Experimental

2.1. Instrumentation

A Unicam AA-929 model atomic absorption spectrometer (AAS) equipped with single hollow cathode lamp (6.0 mA current for gold) and 5 cm of burner head was operated the determinations. Gold ions in solution were atomized by air/acetylene mixture. Wavelength was set at 242.8 nm and the spectral bandwidth at 0.5 nm for gold.

Milestone Ethos D microwave digestion unit with closed vessels and 1450 psi max pressure was used for digestion of ore and soil materials.

2.2. Reagents and solutions

All reagents were of analytical reagent grade and purchased from Merck and Fluka. Stock solution of Au(III) with a concentration of 1000 mg L^{-1} in 2.0 mol L^{-1} HCl was used. Model and standard solutions were prepared by diluting this stock solution using suitable ratios. Distilled-deionized water was used in all experiments.

Amberlite XAD-2000 resin (600 m^2 g^{-1} surface area and 20–50 mesh particle size) and Sodium diethyldithiocarbamate (Na-DDTC) were supplied by Sigma Chem. Gold in sandy soil standard (CRM–SA–C) from High-Purity Standards, Inc., was used as standard reference material.

2.3. Preparation of the mini column

Amberlite XAD-2000 resin was thoroughly washed with 1 mol L^{-1} NaOH, water, 1 mol L^{-1} HNO_3, water and acetone successively. It was finally washed with 10 mL of water and dried in desiccators for further use.

A glass column with stopcock and porous disk (1 cm × 10 cm i.d.) was used in all experiments. The glass column was loaded with 0.250 g of ground and sieved (150–200 μm) Amberlite XAD-2000 resin. After each use, the resin filled to column was washed with large volumes of distilled-deionized water and 1 mol L^{-1} HNO_3 in acetone, and was conditioned with HNO_3 solutions in different concentrations before being stored in water for the next experiment.

2.4. Preconcentration procedure

Fifty milliliters of distilled water containing 50 μg of Au(III) ions were prepared in 0.5–2.5 mol L^{-1} HNO_3. Then 5 mL of Na–DDTC (0.1%) was added to the solution. The solution was passed through the column at 10.0 mL min^{-1}. The adsorbed gold complex on the column was eluted with 1 mol L^{-1} HNO_3 in acetone and acetone in the eluent was evaporated to near dryness. The residue was diluted to 5.0 mL with 1 mol L^{-1} HNO_3 and the final solution was analyzed for gold by FAAS.

2.5. Application to real samples

The proposed method was applied to CRM–SA–C Sandy Soil, Mastra Gold Ore from Gumushane, Turkey and agricultural soil from Surmene, Trabzon, Turkey. The 1.250 g of fine powdered samples were digested in microwave digestion unit by addition of 8 mL of aqua regia and 1 mL of HF according to the microwave digestion program under 45 bar pressure (1 min for 250 W and 180 °C, 1 min for 0 W and 180 °C, 10 min for 650 W and 200 °C, 5 min for 250 W and 220 °C, respectively, vent: 3 min). The final mixture was evaporated to dryness in a hot plate. Twenty milliliters of 1 mol L^{-1} HNO_3 was added. The suspension formed was filtered through blue band filter paper. Then the enrichment procedure given in Section 2.4 was applied.

Another application of the presented procedure was performed to a mineral water from Araklı, Trabzon, Turkey and stream water from Of, Trabzon, Turkey. Fifty milliliters of water sample was taken then the procedure given above was applied.

3. Results and discussion

3.1. Effect of nitric acid concentration

The metal–diethyldithiocarbamate complexes were generally occurred at the acidic media [30]. The influence of nitric acid concentration on the retention of gold(III) and other some metal ions-diethyldithiocarbamate chelates on Amberlite XAD-2000 was studied in the HNO_3 concentration range of 0.5–2.5 mol L^{-1}. The results are depicted in Fig. 1. Gold(III)-diethyldithiocarbamate chelates was quantitatively recovered in all working range of nitric acid concentration. Gold(III) was effectively separated from other metal ions simultaneously in the 1.0–2.5 mol L^{-1} HNO_3 concentrations. Hence, 2.0 mol L^{-1} HNO_3 medium was selected for the next applications in order to

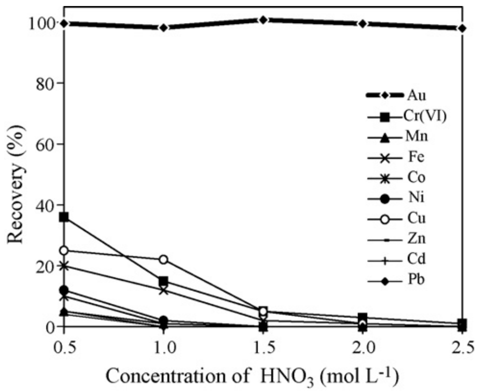

Fig. 1. The variation of HNO_3 concentration with recovery (eluent: 1 mol L^{-1} HNO_3 in acetone, resin quantity: 0.250 g, sample flow rate: 10 mL min^{-1}, $N = 4$).