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Reversed phase extraction chromatographic separation of gold(III) with high molecular mass liquid anion exchanger on silica gel

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Abstract

A novel method has been developed for the extraction chromatographic separation of gold(III) with N-n hexylaniline (N-n-HA) liquid anion exchanger coated on silanised silica gel. Its quantitative extraction has been achieved by 1.5 mol L^{-1} hydrochloric acid and eluted with ammonia buffer solution. The various parameter studied viz. Effect of acid concentration, stripping agent for extraction and elution have been evaluated, flow rate and effect of varying concentration of gold(III). Method extends its application from analysis of ayurvedic samples containing gold and analysis copper-silver-gold alloy permits quantitative recovery of gold.

Keywords: N-n hexylaniline, Extraction, Gold(III), Ayurvedic samples, Alloy

Introduction

Gold is one of the precious metals occurs in the nature in the elementary state in the earth's crust about 0.004 ppm. Gold mainly known for its colour, electrical conductivity, ductility and corrosion resistance. It has an application as a catalyst in hydrogenation of some organic compounds. The beauty and rarity of gold has led to its use in jewellery, in coinage, as a monetary standard and dentistry. The infrared reflectivity of gold leads to its use in the aeronautics and space industries. Gold alloys such as binary alloys of gold and palladium used in electroplating and the analysis of plating baths. Ternary alloys of gold, silver and copper used in jewelry. Gold compounds used in photography, medicine, electroplating industry and with sulfurized Venice turpentine as an ink or paint. The economy of nation depends to a significant extent on the size of its gold reserves. Since many decades every human is having great desire of suvarna i.e. Gold. By nourishing the brain cells it improves memory, increases the strength and immunity of the body and improves the pronunciation. It increases blood corpuscles, hemoglobin and improves cardiac functions as well. Gold has low abundance, high prices and wide range of applications hence it is essential to develop an innovative method for its separation is an analytical merits.

Gold(I) is extracted by solvent extraction gold(I) from cyanide solution by dibutylcarbitol (DBC) with n-octanol but extraction of gold(I) is only 96.2% and for striping of gold with 4% sodium thiosulfate is required[1]. Au(I) is extracted with TBP from polysulfide solution using 0.5 mol L^{-1} N1923-0.5 mol L^{-1} TBP/n-octane as the org. phase at pH < 8 or pH > 11[2]. Solvent Extraction of Au(III), Pt(IV), and Pd(II) Ions with bis(2-ethylhexyl) N-butyl-N-octylaminomethylphosphonate in chloroform and xylene was studied. The quantitative recovery found in hydrochloric acid solution and separation of associated Fe(III), Cu(II), Ni(II), and Co(II) ions carried out by this

method[3]. Liquid liquid extraction of gold by N-n-octylaniline, using xylene as a diluents from aq. sodium malonate medium at pH 1.0 extraction is carried out at organic acid media which is economical and less hazardous[4]. Gold determination by radioactive isotope-dilution method[5]. ICP-AES method was used for analysis of Au-Zr alloy[6]. spectrometric methods for determination of gold with feroine; (b)[7]. High molecular weight amines N-n-octylaniline have been used for solvent extraction of gold(III) in halide media[8] and 4-heptylaminopyridine used for solvent extraction of gold(III)[9]. N-n-Hexylaniline is used for solvent extraction of Bi(III)[10] and Zn(II)[11] but solvent extraction is techniques having some drawbacks viz. costly solvent, third phase formation to overcome these drawbacks the Reversed phase extraction chromatography method has been proposed for extraction and separation of gold(III). Separation of gold (III) from ayurvedic medicines and alloys by extraction chromatography using N-n-octylailine coated on silica gel in hydrochloric acid solution reported[12]. Reversed Phase Extraction Chromatographic method has become a very powerful separation method because it is very simple, rapid, selective and sensitive. This method does not require any sophisticated instruments. It is quite effective and separation can be carried out at macro and microgram concentrations. The efficiency of ion exchange separation could be easily improved by increasing column height or in other words increasing the number of theoretical plates (HETP). The advantages of this method are regenerating the resin and use it again and again therefore the method becomes economical. On account of the advantages of regeneration of exchangers, the process becomes most economical[13]. Extraction chromatography provides a facile, adequate, efficient and yielding method for the analytical and preparative-scale separation of a variety of metal ions However there is no work proclaimed on separation of gold(III) with N-*n*-hexylaniline. In view of all above sequential extraction and separation of gold (III) have been receiving considerable engrossment by the researchers

Experimental

Instrumentation

Equiptronics Model EQ-820 digital spectrophotometer with 10 mm path length quartz cuvettes was used for the absorbance measurements. Calibration of the spectrophotometer for UV region was done by measuring the absorbance of 0.1 mol/L potassium nitrate at 305 nm. For visible region by measuring the absorbance of 0.001 mol/L potassium permanganate and potassium dichromate in 1.0 mol/L sulfuric acid at 425 nm and 545 nm respectively [14].

Reagents

A stock solution of gold(III) was prepared by dissolving 1.0 gm AR. Grade chloroauric acid ($HAuCl_4$. xH_2O) (Loba Chemie Pvt. Ltd., Mumbai. India) in 250 mL of distilled water and standardized gravimetrically[15]. A working solution 200 μg/mL was made by appropriate dilution.

N-n hexylaniline (N-n-HA) has been prepared using method reported by (Gardlunds 1973)[16]. The working reagent solution (5.0×10^{-2} mol L^{-1}) of N-n-HA was prepared in chloroform.

Preparation of anion exchange material

Silica gel (60 -120 mesh) obtained from British Drug House India Ltd., dried at 120^0C for 2-3 h, stored in desiccators. It was packed in a U tube through which a stream of nitrogen was bubbled though a small Durand bottle containing 20.0 mL dimethyldichlorosilane (DMCS) (Acros Organics. New Jersey. USA). DMCS vapor was continued for 4 h. The silica gel was washed with anhydrous methanol, then dried. A portion of 5.0 gm silaned silica gel was soaked with 0.05 mol L^{-1} *N-n*-hexylaniline which was previously equilibrated with hydrochloric acid (1.5 mol L^{-1}), then the solvent was evaporated to get nearly dried gel. The slurry of *N-n*-hexylaniline coated silica gel in distilled water was prepared by centrifuging at 2000 r/min. Then the coated silica gel was packed into a chromatographic column to give a 6.0 cm bed. The column was made from borosilicate glass tube, having bore 8 mm, length 30 cm, fitted with glass-wool plug at the bottom. The bed was then covered with a glass wool plug.

General procedure

200 μg of gold(III) was mixed with hydrochloric acid in the concentration range of 0.25 to 8.0 mol L^{-1} in a total volume of 10 mL. the solution was then passed through a column preconditioned with the same concentration of hydrochloric acid as that of the sample solution, at a flow rate of 0.5 ml/minute. The column was then washed with same concentration of hydrochloric acid. The extracted gold(II) was then eluted with different eluting agents at a flow rate 0.5 mL/minute fractions were collected and gold content was determine by spectrophotometrically with $SnCl_2$ at 400 nm[17].

Results and Discussion

Extraction of Gold(III) on N-n hexylaniline (N-n-HA) as a Function of Hydrochloric Acid Concentration

Extraction studies of gold(III) were carried out from 0.25 to 8.0 mol L^{-1}. After extraction gold(III) was eluted with ammonia buffer solution (NH_4NO_3+ NH_4OH, pH 10.1). It was found that there was quantitative (100 %) recovery of gold (III) from 0.75-8.0 mol L^{-1} hydrochloric acid. The results are shown in (Figure 1). The subsequent extraction and separation studies on gold (III) were carried out with 1.5 M Hydrochloric acid.

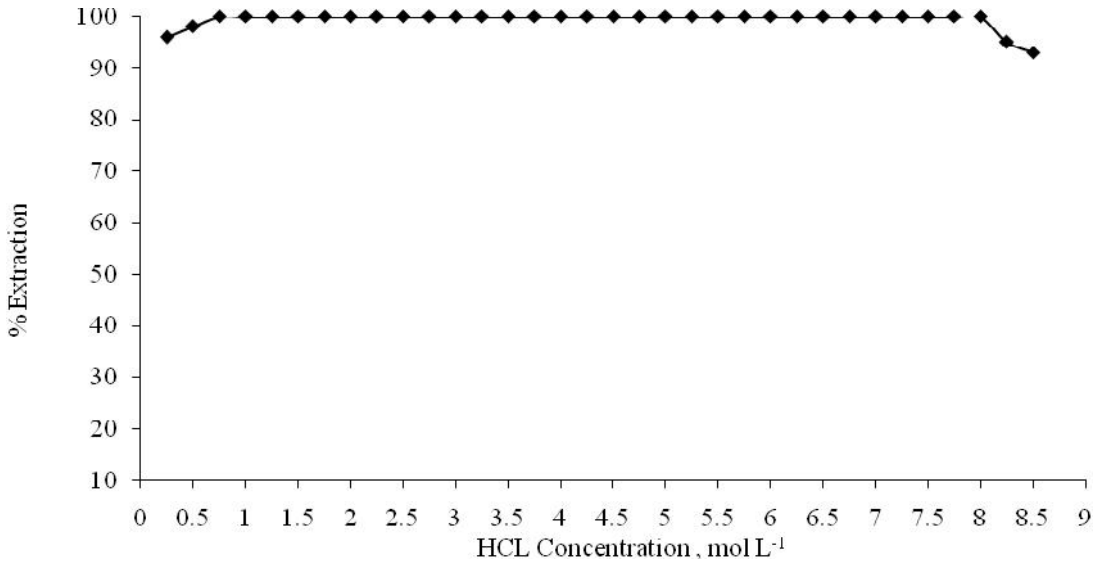

Figure 1. Extraction of gold(III) on N-n-hexylaniline as a function of hydrochloric acid concentration.

Elution Studies of Gold(III) with Various Eluting Agents

100 μg of gold(III) extracted on N-n hexylaniline column at 1.5 mol L^{-1} hydrochloric acid concentration. After extraction gold(III) was eluted from the column with various eluents such as water, ammonia, NaOH, KOH, EDTA and Acetate buffer solution The concentrations of eluting agents were, varied from 0.1-8.0 mol L^{-1} except water. Various elution studies revealed that there was quantitative elution of gold(III) with 20 mL of ammonia buffer solution (NH_4NO_3+ NH_4OH, pH 10.1) where as water, ammonia, NaOH, KOH, EDTA and Acetate buffer solution were found to be inefficient eluents for quantitative elution of gold(III). Further elution studies of gold(III) in this work were carried out with ammonia buffer solution (NH_4NO_3+ NH_4OH, pH 10.1).

Effect of Varying Concentration of Gold(III)

The capacity of N-n hexylaniline for gold(III) was evaluated using 0.05 mol L^{-1} N-n hexylaniline and extraction studies were carried out from 1.5 mol L^{-1} hydrochloric acid using ammonia buffer as eluent. The volume of gold(III) sample solution used was 20.0 mL. The concentration of gold(III) was varied from 200 to 3000 μg of Gold(III) / 20 mL of solution. The results in (Figure 2) showed that the extraction of gold(III) was quantitative up to 2000 μg / 20 mL. The extent of extraction of gold(III) decreased with increasing concentration of gold(III).

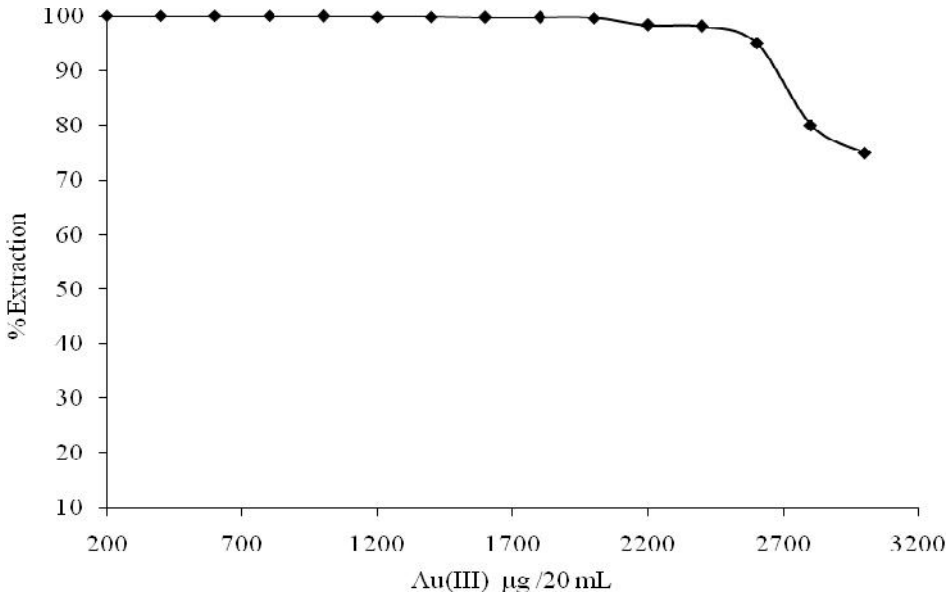

Figure 2. Effect of Varying Concentration of Gold(III).

Effect of flow rate on percentage extraction of gold(III)

The effect of flow rate on percentage extraction of gold(III) was studied from 0.5 to 5.0 mL/min. It was observed that the percentage extraction decreases with increase in the flow rate. Therefore the flow rate was kept at 1.0 mL/min for further extraction studies.

Applications

Analysis of gold(III) from ayurvedic samples

Gold containing Ayurvedic samples are useful for various chronic disorders, general debility, diseases which deplete immunity, anemia, mental disorders, respiratory tract disorders etc. The proposed method was successfully applied for the separation of gold(III) from bruhatwat chintamani, vasant kusumkar, suvarna malini vasant, suvarna sutshekhar, shwas kas chintamani, suvarna bhasma, brahmi vati and makardhwaja vati. The dissolution of ayurvedic sample (1 tablet) in 10 mL of aqua regia. The organic matter was destroyed by treatment with 2 mL of concentrated perchloric acid. The solution was evaporated to moist dryness. The residue was dissolved in hot dilute hydrochloric acid and filtered through Whatmann filter paper No. 1. The filtrate was diluted to required volume with water. Gold(III) was extracted by the proposed method and determined by the standard procedure. The results are in good agreement with certified value (Table 1).

Table 1. Extraction of gold(III) from ayurvedic samples

Sample	Au(III)	
	Certified/ %	Found/ %
Bruhatwat Chintamani	0.18	0.17
Vasant Kusumkar	0.15	0.14
Suvarna Malini Vasant	0.55	0.54
Suvarna Sutshekhar	2.13	2.10
Shwas Kas Chintamani	1.18	1.17
Suvarna Bhasma	7.60	7.55
Brahmi Vati	0.30	0.28
Makardhwaja Vati	0.10	0.09

Analysis of synthetic mixture corresponding to alloys

The proposed method was successfully applied for the separation of gold(III) from copper-silver-gold alloy. About 1.0 gm of sample was transferred into a 250 mL conical flask covered with a short-stem funnel and dissolved in 10 mL *aqua regia*. The solution was then treated with 10 mL of concentrated hydrochloric acid; the solution was evaporated almost to dryness on a steam bath after each addition. The residue was dissolved in dilute hydrochloric acid and the precipitated silver chloride filtered off and washed with dilute hydrochloric acid. The filtrate and washings were collected in a 250 mL standard flask and made up to volume with distilled water. An aliquot of the sample solution was taken and gold(III) was determined using the procedure described above. The results of analysis matched with certified values (Table 2).

Table 2. Extraction of gold(III) from copper-silver-gold alloy.

Composition of alloy	Gold(III) %	Gold(III) % Found
Cu, 49.35; Ag, 7.25;Au, 43.4	43.40	43.00
Cu, 35; Ag, 5;Au, 60	60.00	59.60

Conclusion

Separation of gold(III) by using reversed phase extraction chromatographic technique has become a very powerful separation method because it is very simple, rapid, selective and sensitive. This method does not require any sophisticated instruments except column. Reversed phase extraction chromatography method quite effective for extraction and separation of gold(III) can be carried out at macro and microgram concentrations. The advantages of this method are regenerating the resin and use it again and again. On account of the advantages of regeneration of exchangers, the process becomes most economical. Applicability of this method is analysis of gold(III) from ayurvedic samples viz. bruhatwat chintamani, vasant kusumkar, suvarna malini vasant, suvarna sutshekhar, shwas kas chintamani, suvarna bhasma, brahmi vati and makardhwaja vati and copper-silver-gold alloy.

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