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TECHNICAL NOTE

Extraction of Gold(III) with Triphenylphosphine Sulfide and Subsequent Spectrophotometric Determination

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ABSTRACT

A method is described for the solvent extraction of gold from bromide media with triphenylphosphine sulfide dissolved in toluene as an extractant. The method permits determination of gold in Ayurvedic medicines. The average recovery of gold is ≥ 99.0%. Log-log plots are used to ascertain the probable extractable species. The method also permits separation of gold(III) from copper(II), lead(II), palladium(II), and platinum(IV).

INTRODUCTION

Gold is a precious metal found in iron pyrites and copper and lead concentrates. It is also a component of many Ayurvedic preparations. In view of this there, is a need for the selective separation and determination of gold from associated elements. Various solvent extraction methods for gold have been summarized by different authors (1, 2), but the existing methods suffer from such drawbacks as preequilibration, multiple extractions, scrubbing, and coextraction of a large number of ions. Of the neutral thiophosphorus extractants, Tachibana et al. (3) reported the extraction of gold with triisobutylphosphine sulfide from thiourea leach solution; however, the method requires use of high molecular weight amines. Neutral thiophosphorus compounds (4) such as tri(alkyl) monothiophosphate, tri-*n*-butyl and tri-*n*-octyl phosphine sulfide were used for the extraction

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of gold from nitrate media, but the extraction requires use of salting out agents and an extraction period of 10 minutes at 25°C. Extractants such as benzothizolethiol (5), α-aminophosphonate (6), dibutyl sulfide (7), and trioctyl phosphine oxide (8) were also used for the extraction of gold. However, these methods suffer from such limitations as the need for high acidic media and the lack of reproducibility. The present method of extraction of gold from bromide media with triphenylphosphine sulfide dissolved in toluene and subsequent spectrophotometric determination at 411 nm is free of these limitations.

EXPERIMENTAL

Apparatus and Reagents

A Shimadzu UV VIS 160 A spectrophotometer and a Plasma 300 Thermo Jarell Ash, USA ICP-AES were used for absorbance measurements.

A stock solution of gold(III) was prepared by dissolving 1 g of gold chloride (Johnson Matthey and Co., London) in 100 mL of distilled water containing 1% (v/v) hydrochloric acid. The solution was standardized gravimetrically (9).

1% (w/v) Triphenylphosphine sulfide (TPPS) (Fluka) dissolved in toluene was used for our extraction studies.

All other chemicals used were of analytical reagent grade.

Extraction Procedure and Subsequent Spectrophotometric Determination of Au(III)

To an aliquot of an aqueous solution containing 200 μg of gold(III), add hydrobromic acid so that its molarity is 2.0–2.4 M in a total volume of 10 mL. Transfer the solution into a separatory funnel and equilibrate for 60 seconds with 5 mL of 1% (w/v) triphenylphosphine sulfide (TPPS) dissolved in toluene. Separate the layers and discard the aqueous solution. The yellow bromoaurate complex of gold in the TPPS phase is determined spectrophotometrically at 411 nm against the blank prepared analogously. The extraction of gold in TPPS is confirmed by estimating the unextracted gold in the discarded aqueous layer by ICP-AES at 242.795 nm.

RESULTS AND DISCUSSION

The variation in the concentration of hydrobromic acid (0.22–3.11 M, v/v) and TPPS concentration (0.01–2.00%, w/v) dissolved in toluene showed that the quantitative extraction of gold(III) occurs with 0.96–1.06% (w/v) TPPS dissolved in toluene from an aqueous solution containing 2.0–2.4 M (v/v) of hydrobromic acid. Of the various diluents tried, including toluene, xylene, benzene, chloroform, and carbon tetrachloride, toluene gave satisfactory results and also facilitates quick phase separation. Extraction of gold in the other diluents is between 27.04 and 89.05%.

Nature of the Extracted Species

A log-log graph of the distribution ratio versus HBr concentration (at 1% w/v TPPS concentration) (Fig. 1) or of the distribution ratio versus

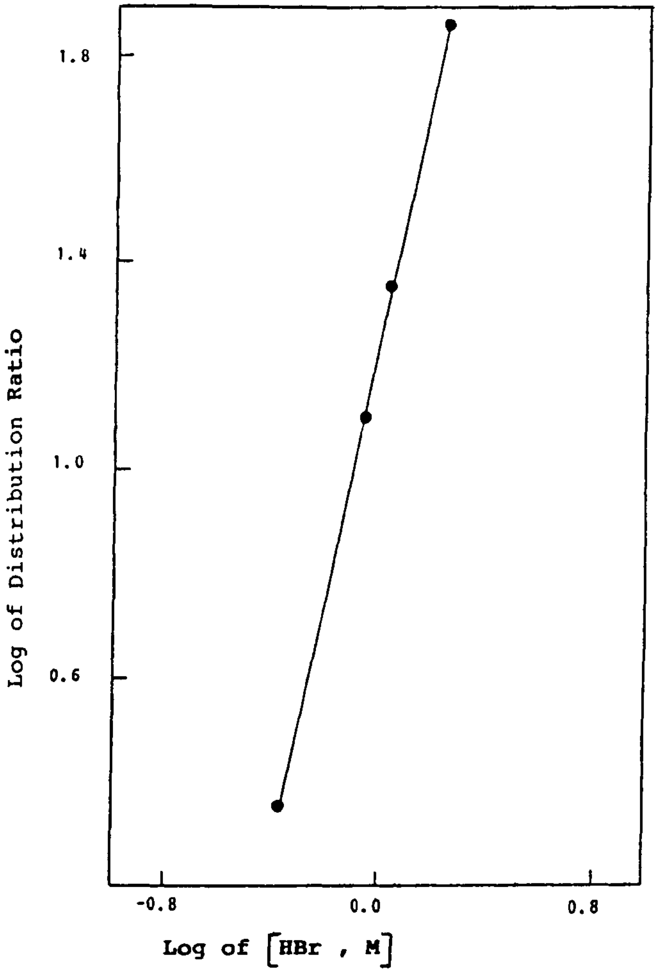

FIG. 1 A plot of log of distribution ratio for gold(III) versus log of hydrobromic acid concentration (at 1% w/v TPPS concentration).

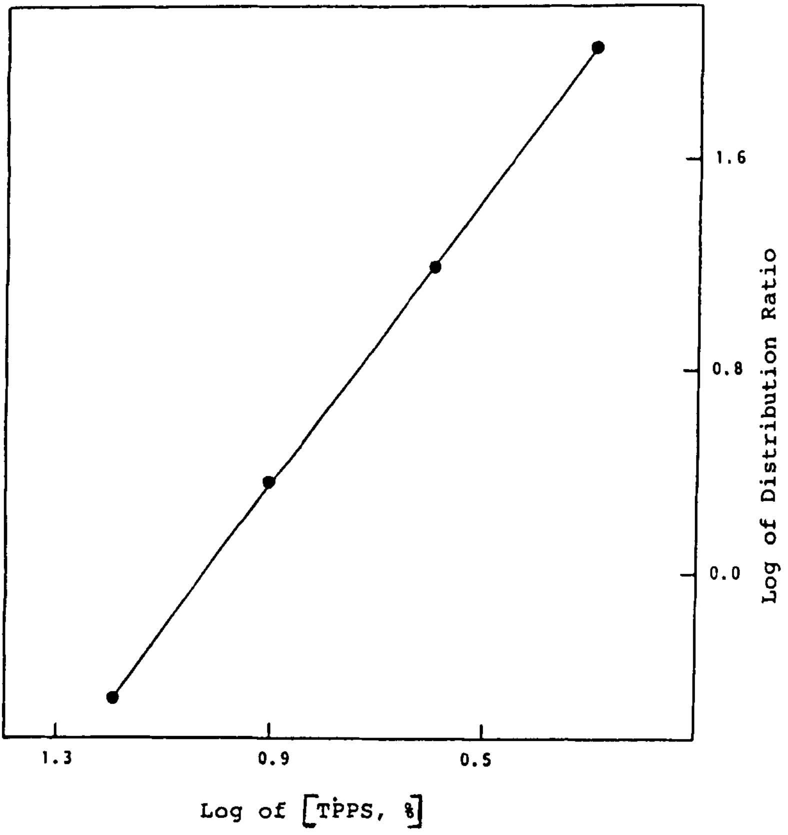

FIG. 2 A plot of log of distribution ratio for gold(III) versus log TPPS concentration (at 2.2 M, v/v hydrobromic acid concentration).

TPPS concentration (at 2.2 M, v/v HBr concentration) (Fig. 2) indicates a molar ratio of 1:4 with respect to bromide and 1:3 with respect to extractant. Hence the extracted species is an ion pair of the type $H^+AuBr_4^-$ which is solvated by TPPS to give the species $H(TPPS)^+$, $AuBr_4^- \cdot 2TPPS$. Both the proton and the ion pair are solvated. The presence of TPPS renders the ion pair hydrophobic and causes its extraction.

Spectral Characteristics

The yellow bromoaurate complex extracted in TPPS dissolved in toluene is shown in Fig. 3. The λ_{max}, Sandell sensitivity, and molar absorptivity are 411 nm, 6.7×10^{-2} $\mu g \cdot cm^{-2}$ and 2942 $L \cdot mol^{-1} \cdot cm^{-1}$, respec-

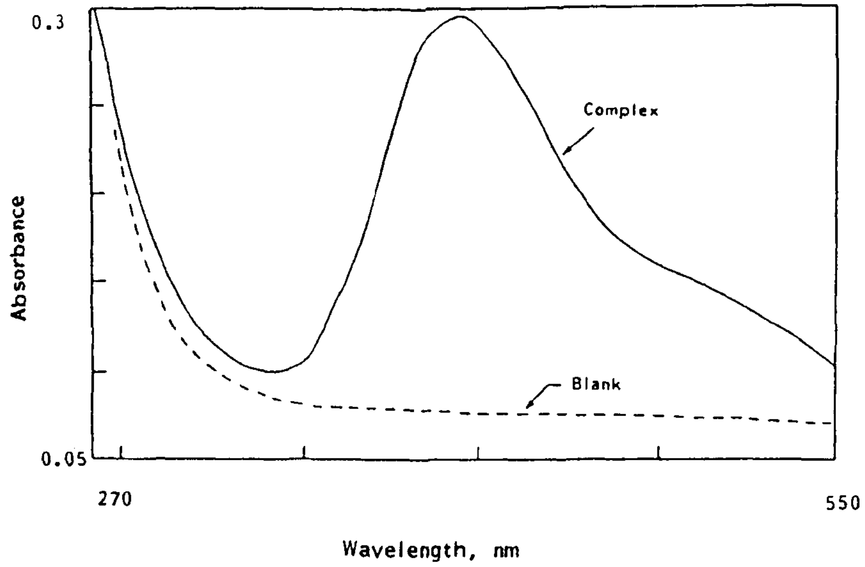

FIG. 3 Spectrum of blank and bromoaurate TPPS complex in toluene.

tively. The system conforms to Beer's law over the 4–60 μg·mL^{-1} concentration range of gold(III).

Effect of Diverse Ions

Varying amounts of foreign ions were added to a fixed amount of gold(III) (200 μg) to study their interference in the recommended procedure. The tolerance limit was set at the amount required to cause a ±2% error in the recovery of gold(III). The ions Co(II), Mn(II), Hg(II), Cr(VI), Pb(II), Th(IV), Tl(III), Ba(II), Cu(II), Zn(II), Cd(II), Ni(II), Re(VII), Hf(IV), U(VI), Os(IV), Te(IV), Al(III), tartarate, citrate, oxalate, and EDTA did not show interference in the 5000–3000 μg range. There was no interference from the ions Pd(II), Pt(IV), Fe(III), Ti(IV), Zr(IV), La(III), Ga(III), Ce(IV), Se(IV), Mo(VI), W(VI), V(V), Bi(III), and phosphate in the 1000–500 μg range. However, Ag(I), Sb(III), thiocyanate, and thiosulfate ions interfered and must be absent.

Separation of Gold(III) from Cu, Pb, Pd, and Pt

Extraction of gold with TPPS by the recommended procedure facilitates its separation from metal ions such as copper, lead, palladium and plati-

TABLE 1
Separation of Gold(III) from Mixtures

Composition of mixture (μg)	Recovery of gold[a] (%)	Relative standard deviation (%)	Recovery of added ion[a] (%)	Relative standard deviation (%)	Estimation procedure for added ion (%)
Au (200); Pb (200)	99.20	0.98	99.90	0.29	PAR (10)
Au (200); Pd (200)	99.30	0.89	99.20	0.95	PAR (11)
Au (200); Cu (200)	99.40	0.75	99.70	0.49	PAN (12)
Au (200); Pt (200)	99.20	0.98	99.30	0.89	$SnCl_2$ (13)

[a] Mean of triplicate analysis.

num as they do not extract into TPPS dissolved in toluene under the optimum extraction conditions of Au. Gold is determined spectrophotometrically as described in the recommended procedure whereas copper, lead, palladium, and platinum are determined spectrophotometrically with PAN (12), PAR (10, 11) and $SnCl_2$ (13). The recoveries of Au(III) as well as of added metal ions are ≥99.0% with a relative standard deviation between 0.29 and 0.98%. The results are reported in Table 1.

Determination of Gold in Ayurvedic Medicines

About 100 mg of Swarna Bhasma and Siddha Makkar Dhwaj special were each dissolved in 5 mL of aqua regia, boiled, cooled, filtered, and diluted to 25 mL. Aliquot volumes of the sample solutions were taken for the extraction and determination of gold by recommended procedure. Each determination took a total of 25 minutes. The amounts of gold found by the recommended procedure were compared to what was obtained by the ICP-AES technique. The results are reported in Table 2.

TABLE 2
Analysis of Ayurvedic Medicines

Name of the medicines	Amount found by ICP - AES[a] (ppm)	Amount found by proposed spectrophotometric method[a] (ppm)	Relative standard deviation (RSD, %)
Swarna Bhasma (SHREE Baidyanath, India)	73.00	72.50	0.32
Siddha Makkar Dhwaj special, (SHREE Baidyanath, India)	103.00	102.00	0.41

[a] Average of triplicate analysis.

CONCLUSION

The highlights of the proposed method are the following

1. The extraction is quantitative in a single step in 60 seconds.
2. The method permits separation of Au from Cu, Pb, Pd, and Pt.
3. Preequilibration, scrubbing of the organic phase, multiple extraction, and elevated temperature are not required.
4. The method is suitable for satisfactory determination of gold in Ayurvedic medicines.

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