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Liquid—Liquid Extraction of Goid(III) with Diluted Tributyl Phosphate

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NOTE

Liquid–Liquid Extraction of Gold(III) with Diluted Tributyl Phosphate

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Summary

A new method is developed for the solvent extraction of gold with diluted tributyl phosphate. In this method, 50% tributyl phosphate in toluene extracts gold quantitatively from 3 *M* hydrochloric acid containing 2 *M* lithium chloride as the salting-out agent. Gold from the organic phase is stripped with 1 *M* ammonium hydroxide and determined photometrically as its complex with stannous chloride. It is possible to extract gold in the presence of a large number of ions.

Most methods for the solvent extraction separation of gold are by ion-association extraction systems (*1*). Tributyl phosphate (TBP) (*2*) has been used to study the extraction behavior of many elements, including gold in nitric acid solution. The extraction of gold with TBP from hydrochloric acid (*3*) is efficient with xylene as the diluent. TBP–carbon tetrachloride has been used for the extractive separation of gold from lanthanides (*4*). The composition of the extractable chlorocomplexes of gold and a few other metals with TBP as an extractant has been established (*5*). Triphenylpropyl and isopropyl phosphonium compounds have been used for the extraction of gold (*6*). However, systematic studies on the solvent extraction of gold with diluted tributyl phosphate are lacking. We present such a study in this paper. The proposed method is rapid, simple, and applicable at tracer concentrations.

EXPERIMENTAL

Apparatus and Reagents

Type ϕ 3KH-57 photoelectric photometer, wrist action flask shaker, Cambridge pH indicator, tributyl phosphate (B.D.H.).

A stock solution of gold(III) was prepared by dissolving about 2 g brown gold chloride (Johnson Matthey & Co., London) in a liter of distilled water. The solution, on gravimetric standardization (7) with hydroquinone, was found to contain 95.6 μg of gold/ml.

TABLE 1

Distribution Ratio as a Function of Acidity[a]

TBP concn (*M*)	HCl (*M*, initial)	Extraction (% *E*)	Distribution ratio (*D*)
20% (0.73)	1	30.0	1.07
	2	40.0	1.67
	3	50.0	2.50
	4	60.0	3.75
30% (1.09)	1	43.3	1.91
	2	57.7	3.41
	3	63.3	4.31
	4	70.0	5.83
50% (1.83)	1	70.0	5.83
	2	80.0	10.00
	3–4	100.0	∞
50% (1.83) in the absence of salting-out agent	0.5	40.0	1.67
	1.0	50.0	2.50
	2.0	60.0	3.75
	3.0	66.7	5.18
	4.0	76.7	8.05
	5.0	53.3	2.85
75% (2.74)	1	73.3	6.87
	2	83.3	12.47
	3–4	100.0	∞
100% (3.66)	1	80.0	10.00
	2	90.0	22.50
	3–4	100.0	∞

[a] Au(III) = 191.2 μg, 2 *M* LiCl.

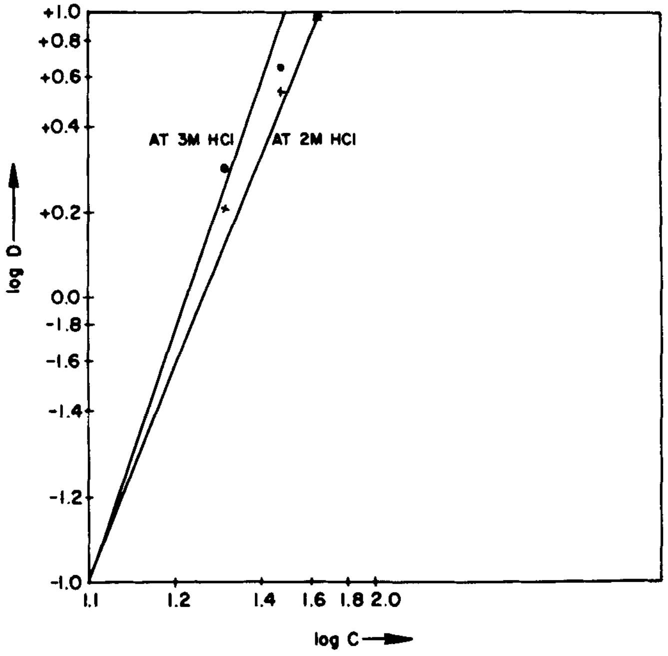

FIG. 1. Extraction as a function of TBP concentration.

General Procedure

An aliquot of solution containing 191.2 μg of gold was taken. In a total volume of 25 ml, lithium chloride and hydrochloric acid were added so that they had a concentration of 2 *M* and 3 *M*, respectively. The solution was transferred into a separatory funnel, and 10 ml of 50% tributyl phosphate in toluene was added. It was then shaken briskly for about 7 min. After allowing the phases to settle and separate, gold from the organic phase was stripped by shaking it with 10 and 5 ml of 1 *M* ammonium hydroxide. It was then determined photometrically in the aqueous phase by the tin(II) chloride method (*8*).

RESULTS AND DISCUSSION

Effect of Acidity and TBP Concentration

The concentration of hydrochloric acid was varied from 0.5 to 5.0 *M* in the presence of 2 *M* lithium chloride and that of TBP from 20–100%

TABLE 2

Effect of Salting-Out Agents[a]

Salting-out agents (M)	HCl (M, initial)	Extraction (% E)	Distribution ratio (D)
LiCl (1)	1	60.0	3.75
	2	70.0	5.87
	3	83.3	12.87
	4	93.3	13.97
(2)	1	70.0	5.83
	2	86.7	16.29
	3–4	100.0	∞
$MgCl_2$ (1)	1	53.3	2.85
	2	66.7	5.18
	3	73.3	6.87
	4	80.0	10.00
(2)	1	66.7	5.18
	2	86.7	16.29
	3–4	100.0	∞
NH_4Cl (1)	1	53.3	2.85
	2	60.0	3.75
	3	73.3	6.87
	4	80.0	10.00
(2)	1	60.0	3.75
	2	70.0	5.83
	3	80.0	10.00
	4	90.0	22.50

[a] Au(III) = 191.2 μg, 50% TBP-toluene.

(0.73–3.66 M) with toluene as the diluent. It was observed that for the quantitative extraction of gold it is essential to use 50% TBP–toluene (Table 1). The optimum hydrochloric acid concentration is 3 M in the presence of 2 M lithium chloride. An attempt was made to establish the probable composition of the extractable species by using a plot of log D vs log TBP concentration at 3 M hydrochloric acid. The slope at this acidity was 2.72 (Fig. 1), indicating that the extractable species is likely to be $H[AuCl_4(TBP)_3]$ or $AuCl_3 \cdot 3TBP$.

The study of the effect of various diluents, such as benzene, xylene, isobutyl methyl ketone, chloroform, carbon tetrachloride, and toluene, showed that toluene is the most suitable diluent because it gave the highest distribution ratio.

TABLE 3

Effect of Diverse Ions[a]

Foreign ion	Added as	Tolerance limit (μg)
Pb^{2+}	$Pb(NO_3)_2$	5,000
Pd^{2+}	$PdCl_2 \cdot 2H_2O$	1,000[b]
Ru^{3+}	$RuCl_3 \cdot 3H_2O$	500
Cu^{2+}	$CuSO_4 \cdot 5H_2O$	2,000
Sb^{3+}	$Sb_2(SO_4)_3$	None
Cd^{2+}	$2CdCl_2 \cdot 5H_2O$	1,000
Bi^{3+}	$Bi(NO_3)_3 \cdot 5H_2O$	None
Fe^{3+}	$FeCl_3 \cdot 6H_2O$	200[c]
Be^{2+}	$Be(NO_3)_2 \cdot 3H_2O$	2,500
Ti^{4+}	$TiCl_4 \cdot 4H_2O$	None
Zr^{4+}	$Zr(NO_3)_4$	3,000
Ce^{4+}	$Ce(SO_4)_2$	5,000
Th^{4+}	$Th(NO_3)_4 \cdot 4H_2O$	5,000
U^{6+}	$UO_2(NO_3)_2 \cdot 6H_2O$	2,500
Zn^{2+}	$ZnSO_4 \cdot 7H_2O$	5,000
Mn^{2+}	$MnCl_2 \cdot 4H_2O$	None
Co^{2+}	$CoCl_2 \cdot 6H_2O$	5,000
Ni^{2+}	$NiCl_2 \cdot 6H_2O$	2,500
Ca^{2+}	$CaCl_2 \cdot 2H_2O$	10,000
Sr^{2+}	$SrCl_2 \cdot 6H_2O$	10,000
Ba^{2+}	$BaCl_2 \cdot 2H_2O$	10,000
Rh^{3+}	$RhCl_3 \cdot 3H_2O$	1,000[d]
Os^{3+}	H_3OsO_3	500
Pt^{4+}	$H_2PtCl_6 \cdot xH_2O$	1,000
Ir^{3+}	$IrCl_3 \cdot 3H_2O$	200[d]
Rb^{+}	$RbCl$	5,000
Cs^{+}	$CsCl$	5,000
Ag^{+}	$AgNO_3$	500[e]
Tl^{+}	$TlNO_3$	5,000
CrO_4^{2-}	K_2CrO_4	None
$Mo_7O_{24}^{6-}$	$(NH_4)_6Mo_7O_{24}$	200
VO_3^{-}	NH_4VO_3	1,000
TeO_3^{2-}	Na_2TeO_3	None
SeO_3^{2-}	Na_2SeO_3	None
WO_4^{2-}	Na_2WO_4	200
SCN^{-}	NH_4SCN	None
$S_2O_3^{2-}$	$Na_2S_2O_3$	None
F^{-}	NaF	5,000
Br^{-}	$NaBr$	200
CH_3COO^{-}	CH_3COONH_4	1,000

(*continued*)

TABLE 3 (continued)

Foreign ion	Added as	Tolerance limit (μg)
PO_4^{3-}	Na_3PO_4	1,000
ClO_3^-	$KClO_3$	5,000
SO_4^{2-}	$Na_2SO_4 \cdot 10H_2O$	10,000
Cit^{3-}	Citric acid	3,000
$Tart^{3-}$	Tartaric acid	5,000
$Malon^{2-}$	Malonic acid	1,000
$C_2O_4^{2-}$	Oxalic acid	5,000
$Ascorb^{2-}$	Ascorbic acid	5,000
$EDTA^{4-}$	EDTA (disodium salt)	3,000
$P_2O_7^{2-}$	$(NH_4)_2P_2O_7$	1,000

[a] Au (III) = 191.2 μg, 3 *M* HCl + 2 *M* LiCl, 50% TBP-toluene.

[b] Extraction possible in the presence of pyrophosphate.

[c] Citric acid was used as the masking agent.

[d] Tartaric acid was used as the sequestering agent.

[e] EDTA used as masking agent.

Effect of Salting-Out Agent

The chlorides of lithium, magnesium, and ammonium were used as the salting-out agents in a hydrochloric acid concentration of 1–4 *M*. The results (Table 2) showed that gold can be quantitatively extracted from 3 *M* hydrochloric acid either in the presence of 2 *M* lithium or magnesium chloride as the salting-out agents. Other salting-out agents were not very effective. For routine work 2 *M* lithium and magnesium chloride have equal advantages.

Period of Shaking

The periods of equilibrium tested were of 1, 3, 5, 7, and 10 min. The corresponding percentage extraction was 47, 60, 80, 100, and 100, respectively, i.e., for complete extraction, the solution should be shaken for at least 7 min.

Effect of Diverse Ions

Various ions were tested for interference. The tolerance limit was set as the amount that would cause a ±2% error in the recovery of gold.

It was observed that gold can be extracted satisfactorily in the presence of a 1:50 ratio of alkaline earths, whereas ions such as lead(II), cerium(III), thorium, zinc, cobalt(III), rubidium, caesium, thallium, and organic complexing anions are tolerated at a ratio of 1:20. Other ions such as beryllium, uranium, nickel, palladium, copper, cadmium, platinum, and vanadate are tolerated in smaller ratios. The ions showing strong interference are bismuth, titanium, manganese, antimony, chromate, selenite, tellurite, thiocyanate, and thiosulfate. However, the interference of many common ions can be eliminated by using EDTA alkali pyrophosphate or citric or tartaric acids as the sequestering agents (Table 3).

Thus the method is simple, rapid, and selective. It is possible to accomplish clean-cut separation of gold at micro concentrations. The average recovery of gold was 99.80 ± 0.2%.

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