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NOTE

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Abstract

A simple and rapid method is described for the analytical separation of gold(III) from iron, cobalt, nickel, ruthenium, rhodium, iridium, palladium, platinum, copper, lead, bismuth, and manganese by solvent extraction with 4-methyl-2-pentanol from hydrochloric acid media. Very few cations and anions interfere.

The considerable amount of gold found in iron pyrites, and copper and lead concentrates has magnified the need for a simple and rapid method for the separation of gold. In this communication, 4-methyl-2-pentanol (methyl isobutyl carbinol), which has been used for the liquid-liquid extraction of molybdenum (1), tungsten (2), rhenium (3), and iron (4), is reported as a new extractant for gold. Gold(III) is quantitatively extracted with 75% carbinol in benzene from 1 to 3 *M* hydrochloric acid solution. The metal ion from the organic phase is back-stripped with 0.5 *M* ammonium hydroxide and subsequently determined photometrically by the stannous chloride method (5).

Many solvent extraction methods for gold have been reported in the literature (6, 7). Some of them include the extraction of tetrachloro- or tetrabromoaurate complexes into a variety of oxygen-containing solvents such as ethers (8-10), esters (11-14), ethyl methyl ketone (15), MIBK (16),

butyl alcohol (17), dibutyl carbitol (18), alkyl oxides (19–21), and TBP (22). In ether extractions (8–10), cations such as Pd, Pt, Rh, and Ir co-extract, whereas cations such as Cr, Fe, and Ni interfere. Esters (11–14) are good extractants but the method requires too many steps and thus is time consuming. The ethyl methyl ketone (15) method suffers from many coextractions and the final estimation of the metal ion requires about 5 hr. The method also involves many steps. The TOPO (19) method is time-consuming; mesityl oxide (21) method suffers interferences due to Sb(III), Cr(III), Mn(III), etc., and the TBP (22) method involves strong interference due to Bi(III), Ti(IV), Mn(II), Sb(III), and Cr(VI).

Thus, except for the dibutyl carbitol (17) and ethyl acetate methods, no other satisfactory method has been reported for the separation of gold from platinum group metals. Our proposed method, offers a selective and reasonably specific means for the extraction and separation of gold from metal ions such as iron, cobalt, nickel, ruthenium, rhodium, palladium, iridium, platinum, copper, lead, bismuth, and manganese. In addition, the method is simple, rapid, reproducible, and free from large coextractions.

EXPERIMENTAL

Apparatus and Reagents

A Zeiss spectrophotometer (Jena) with matched 1 cm quartz cells was used.

The stock solution of gold(III) was prepared by dissolving 0.5 g of gold chloride (Johnson Matthey & Co., London) in 100 ml of water containing 1% hydrochloric acid. The solution was standardized gravimetrically and then diluted to give a concentration of 50 $\mu\text{g Au/ml}$.

4-Methyl-2-pentanol (E. Merck, Germany, bp 131–134°C).

General Procedure

An aliquot of solution containing 200 μg of gold(III) was mixed with enough hydrochloric acid to make its concentration 3 *M* in a total volume of 10 ml. The solution was then introduced into a separating funnel and shaken for 1 min with 10 ml of 75% carbinol in benzene. The two layers were allowed to settle and separate. Gold was removed from the organic layer by back-extraction with 2 \times 10 ml of 0.5 *M* ammonium hydroxide, and the gold in the extract was determined colorimetrically by the stannous chloride method.

RESULTS AND DISCUSSION

Effect of Variable Conditions

The extraction of trivalent gold ($200\ \mu\text{g}$) into carbinol as a function of hydrochloric and hydrobromic acid concentration was investigated by varying the acid concentrations from 0.125 to 3 M and the carbinol concentration from 10 to 100% using benzene as the diluent. The extraction curves show (Fig. 1) that 75% carbinol extracts gold quantitatively from 1 to 3 M HCl and 0.5 to 3 M HBr solutions. For subsequent extraction studies, 3 M HCl solution was employed because it gives a clear-cut separation of the two layers. The $\log D$ vs $\log C$ (carbinol concentration) plot (Fig. 2) at 1 M HCl gives a slope of 1.88, which indicates that the probable composition of the extracted species is $\text{HAuCl}_4 \cdot 2\text{MIC}$.

Variation of shaking time from 15 to 120 sec showed that a minimum of 1 min shaking is required for complete extraction of the metal.

Diverse Ions

To study the effects of diverse ions on the overall procedure of extraction and determination of gold, varying amounts of representative cations

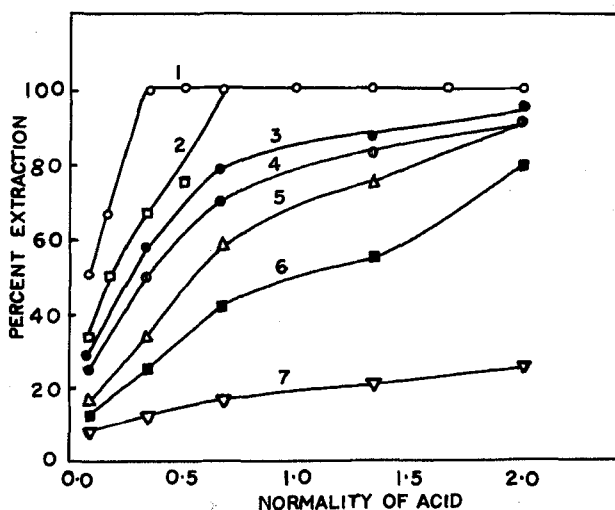


FIG. 1. Extraction curves of gold(III). (1) Extraction from HBr with 75% carbinol in benzene. (2)–(7) Extraction from HCl with 75, 60, 50, 40, 25, and 10% carbinol in benzene, respectively.

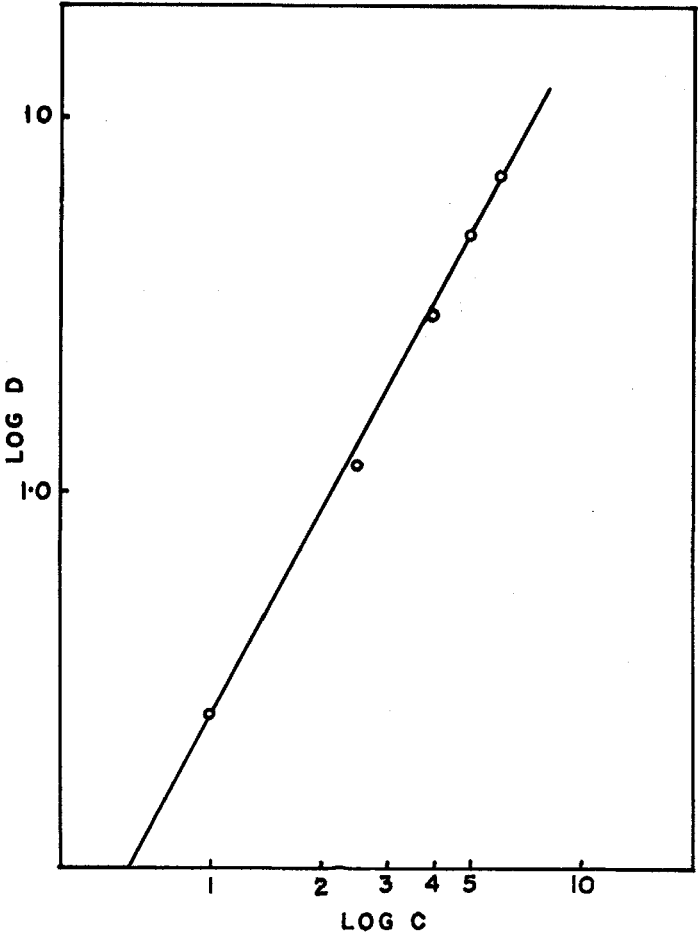


Fig. 2. Distribution ratio of gold(III) as a function of methyl isobutyl carbinol concentration.

and anions were added to known amounts of gold and extractions were carried out as outlined in the general procedure.

There was no interference from 5 mg of the following ions: Rh(III), Ga(III), Th(IV), Tl(I), Zr(IV), Ce(IV), and Se(IV). The system could also tolerate 2 mg of the following ions: Cu(II), Co(II), Fe(III), Ni(II), Pb(II), Cd(II), Bi(III), Cr(III), Mn(II), Sb(III), Mo(VI), Pt(IV), Ru(III), In(III), Tl(III), U(VI), W(VI), and V(V). Up to 4 mg of Ir(III) and 1 mg of Os(VIII) did not interfere. Of the anions tested, 2 mg each of tartrate, citrate, oxalate, EDTA, and fluoride were tolerated. However, mercury, tin, tellurium, and phosphate interfere severely and must be absent.

Analysis of Synthetic Mixtures

Several solutions containing gold and one or more other metals in known amounts were analyzed by the proposed method. The results in Table 1 reveal that there is no extraction of cobalt(II), nickel(II), ruthenium(III), rhodium(III), iridium(III), platinum(IV), copper(II), lead(II), bismuth(III), and manganese(II) under the conditions found most suitable for the extraction of gold, i.e., from 3 *M* HCl solution. Hence at this acidity gold was extracted into 75% carbinol in benzene, keeping other

TABLE 1
Percentage Extraction of Other Metals by 75% Carbinol in Benzene from Hydrochloric Acid

Metals	% Extraction from		Interference in the determination of gold
	1 <i>M</i> HCl	3 <i>M</i> HCl	
Fe(III)	1.5	22.0	—
Co(II)	0.0	0.0	—
Ni(II)	0.0	0.0	—
Ru(III)	0.0	0.0	—
Rh(III)	0.0	0.0	—
Pd(II)	1.0	8.0	—
Os(VIII)	10.0	22.0	—
Ir(III)	0.0	0.0	—
Pt(IV)	0.0	0.0	—
Cu(II)	0.0	0.0	—
Hg(II)	15.0	36.0	Interference
Pb(II)	0.0	0.0	—
Bi(III)	0.0	0.0	—
Te(IV)	14.0	36.0	Interference
Mn(II)	1.0	1.0	—

TABLE 2
Separation of Gold from Synthetic Mixtures^a

Ions added	% recovery of gold from duplicate analysis
Fe (2)	99.12
Co (2)	100.00
Ni (2)	100.00
Ru (2)	100.00
Rh (5)	100.00
Pd (1)	103.50
Ir (4)	100.00
Pt (2.7)	100.00
Cu (2)	99.62
Pb (2)	100.00
Bi (2)	100.00
Mn (2)	100.00
1 mg each of Co, Ni, Ru, Rh, Ir, Pt, Cu, Pb, Bi, and Mn	99.75
1 mg each of Fe and Pd	99.50

^a Numbers in parentheses indicate the milligram amounts of the element. Fe and Pd were separated from 1 M HCl solutions, whereas the other metal ions were separated from 3 M HCl solution. Au(III) = 200 μ g; 75% carbinol in benzene.

metal ions in the aqueous phase. The recovery of separated gold is quantitative. The recovery of other separated metal ions in the aqueous phase (estimated by standard procedure) is also almost quantitative (98.5 to 99.5%). The separation of gold from iron(III) and palladium(II) was performed from 1 M HCl solution as at this acidity iron and palladium show only slight extractions. The representative results are shown in Table 2. Osmium(VIII), tellurium(IV) and mercury(II) coextract with gold and hence their separation was not possible.

From 10 runs the average recovery of gold was $99.5 \pm 0.5\%$, with a standard deviation of $\pm 0.8\%$. Each determination took a total of 30 min.

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REFERENCES

1. S. P. Patil and V. M. Shinde, *Anal. Chim. Acta*, **67**, 473 (1973).
2. S. P. Patil and V. M. Shinde, *Anal. Lett.*, **6**(8), 709 (1973).

3. S. P. Patil and V. M. Shinde, *Separ. Sci.*, 9(3), 249 (1974).
4. S. B. Gawali and V. M. Shinde, *Talanta*, 21, 1212 (1975).
5. E. B. Sandell, *Colorimetric Determination of Traces of Metals*, 3rd ed., Interscience, New York, 1959.
6. J. Korkisch, *Modern Methods for the Separation of Rarer Metal Ions*, Pergamon, London, 1969.
7. A. K. De, S. M. Khopkar, and R. A. Chalmers, *Solvent Extraction of Metals*, Van Nostrand, London, 1970.
8. W. A. E. McBryde and J. H. Yoe, *Anal. Chem.*, 20, 1094 (1948).
9. Kh. Daiev and N. Iordanov, *Talanta*, 11, 501 (1964).
10. F. Panteni and G. Piccardi, *Anal. Chim. Acta*, 22, 231 (1960).
11. A. Chow and F. E. Beamish, *Talanta*, 10, 883 (1963).
12. S. Hireno, A. Mizuika, and K. Yamada, *Japan Analyst*, 9, 164 (1960).
13. V. M. Tarayan, Zh. M. Arstamyan, and D. A. Mikaelyan, *Zavod. Lab.*, 34, 1281 (1968).
14. V. Patrovsky, *Collect. Czech. Chem. Commun.*, 27, 1705 (1962).
15. N. Jordenov, St. Mareva, N. Krasnobaeva, and N. Nedylkova, *Talanta*, 15, 963 (1968).
16. F. W. E. Strelow, E. C. Feast, P. M. Mathews, C. J. C. Bothma, and C. R. Vanzyl, *Anal. Chem.*, 38, 115 (1966).
17. P. W. West and T. C. McCoy, *Ibid.*, 27, 1820 (1955).
18. D. F. C. Morris and M. Alikhan, *Talanta*, 15, 963 (1968).
19. H. B. Holbrook and J. E. Rein, *Anal. Chem.*, 36, 2451 (1964).
20. A. Alian, *Mikrochim. Acta*, 1968, 988.
21. V. M. Shinde and S. M. Khopkar, *Anal. Chim. Acta*, 43, 146 (1968).
22. A. A. Yadav and S. M. Khopkar, *Separ. Sci.*, 5, 637 (1970).

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