

Technology Section

Pakistan J. Sci Ind. Res., Vol. 31, No. 7, July 1988

DETERMINATION OF GOLD BY RING-OVEN TECHNIQUE

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(Received February 9, 1986; revised August 11, 1988)

A method based on Weisz Ring-Oven Technique is reported for the determination of gold with ascorbic acid in alkaline media. It is, quick precise and sensitive method ranging from 0.2 ug to 2.0 ug/ul of gold with maximum error of 5.0%. The effect of diverse ions has also been studied. Some of the interferences were removed by using suitable masking agents. The method has also been applied to rock samples, which gives reasonably precise results. Thus the method is suitable for routine analysis of gold.

Key words: Telluride mineral, Determination of gold, Hard rock deposits.

INTRODUCTION

Gold occurs naturally in only two forms, as native gold and as a component of telluride mineral. The former exists mostly as an alloy with silver and occurs in hard rock deposits usually in quartz matrix and in sand, while the latter exists in association with telluride mineral such as petzite (Ag, Au) Te.

Numerous methods for its determination have been reported in literature [1-24] Beamish in particular has devised colorimetric procedures for the determination of gold [1-4] but these methods suffers from certain major drawbacks. Most of the colorimetric methods are carried out through the formation of a fine colloidal suspension of gold with different reagents and thus suffers from irreproducibility of the results. Tie, Gan and Rzesutko determined gold spectrophotometrically by employing different reagents i.e. Michler's thioketone [5], Polymethacrylate [6], and aniline [7] respectively. Gold was also determined by an atomic absorption method based on the extraction and separation of Au (111) as $(H^+ - AuCl_4)$ [8]. However, Grigoryan *et. al* [9] determined gold by extractive fluorometry in microgram quantities by using a basic dye acriflavine. Gold has also been determined in geological samples by many workers using molten bead evaporation technique [10], radiochemical neutron activation method [11,12] and extractive spectrographic method [13]. Besides this, gold has also been determined by solvent extraction method from solutions with different anions [14], by X-ray fluorescence spectrometry [15], Gravimetric techniques for the determination of gold have also been employed [16, 17, 18]. It has also been determined in the presence of halide ions by treating a sample with an organic complex forming substance [19], whereas Gadshevich [20] determined gold in electroplating baths using an ion selective electrode. Reversed phase extraction chromatography,

was employed by Jiang *et al.* for the separation of micro-amounts of gold from platinum, palladium and copper [21]. Shabenov *et al.* employed atomic absorption for the determination of gold and silver in geological samples [22], while sepctrophotometry was carried out by Sanchez-Pedreno *et al.* [23] and Simonova *et. al.* [24] for the determination of gold.

All these methods are either tedious or lengthy, involving the use of many chemicals and expensive equipment. Hence Weisz Ring-Oven Technique has been employed, which is quick, precise and economical. The apparatus required was prepared in our precision workshop, therefore, its maintenance and after sale service problems were eliminated. The apparatus is so simple that it can either be purchased at low cost or fabricated within common laboratories.

EXPERIMENTAL

Reagents. Gold solution.- 0.2 gms of pure gold was dissolved in a few ml of aqua regia and evaporated to dryness, 2-3 ml of HCl was added and again evaporated to dryness. It was then dissolved in 0.1N HCl and volume was made upto 100 ml with distilled water. Exact dilutions of the above solution were made for low concentrations.

Sodium hydroxide solution. 0.5% solution was prepared by dissolving 0.5 gms of sodium hydroxide in water and making the volume upto 100 ml with distilled water.

Ascorbic acid. 10% solution of ascorbic acid was prepared by dissolving 10 gms of ascorbic acid in distilled water and diluting it to 100 ml.

Rock solution. One gram of the finely ground rock (200 mesh) was digested in 10 ml of aqua regia and evaporated to dryness. Then 10 ml of concentrated hydrochloric acid were added to the resulting mass and heated again to almost dryness. It was then leached thrice with

distilled water through Whatman No. 40 and washings were made to 100 ml with distilled water.

Procedure. At the centre of the whatman filter paper no. 41, 1ul of gold test solution was applied. It was washed with water to the ring zone. After that 1ul of 0.5% sodium hydroxide and 2 ul of 10% ascorbic acid were applied at the same point and washed with distilled water to the ring zone. Ascorbic acid reacts with gold in alkaline media to produce a blue colouration. Thus a sharp coloured ring is formed at the outer zone. Three rings with various volumes of gold test solution were prepared by this method and all were evaluated visually against a standard scale (comprised of 6 standard rings prepared by taking 1,2,4,6,8 and 10 ul of standard gold solution containing 0.2-2 ug/ul of gold). The results were calculated by comparing the unknowns with standard rings. The same method was applied to the rock sample after masking the interfering metal ions.

RESULTS AND DISCUSSION

The reaction between gold and ascorbic acid is very quick a blue colouration is immediately produced on the application of the reactants on the filter paper in alkaline media. Different solvents were tried for washing the coloured reaction product to the ring zone, but all proved futile. To overcome this difficulty, only gold solution was applied at the centre of the filter paper and was then washed with distilled water to the ring zone. After this 1ul of the 0.5% sodium hydroxide followed by 2 ul of 10% ascorbic acid were applied and then washed with distilled water to the ring zone. The reaction between gold and ascorbic acid took place at the ring zone, resulting in the formation of a bright blue ring. 8 to 10 washings were enough for complete washing of the reactants of the ring zone.

It has been found that the colour of the complex changes from pink to violet to blue with change in pH. However, blue colouration is obtained in a fairly wide range of pH, i.e. 5.5-9.0.

The sensitivity of the method, as is reported in Table 1. is reasonably good and gold, in as low concentration as 0.2 ug/ul can be determined with quite good accuracy. The limits of determination are from 0.2 ug-2.0 ug/ul with a maximum error of 5% when 0.4 ug/ul sample was analysed (cf. Table-1). The method reported is very simple, accurate and sensitive. Moreover, the apparatus used is not expensive and convenient to handle.

The method was also applied for the determination of added gold in rock samples. The main interference in

Table 1. Determination of gold by ring-oven method.

S. No.	Amount taken ug/ul	Amount found ug/ul	Error %
1.	0.20	0.21	+ 5.00
2.	0.30	0.31	+ 3.33
3.	0.40	0.38	- 5.00
4.	0.50	0.49	- 2.00
5.	0.60	0.61	+ 1.66
6.	0.80	0.83	+ 3.75
7.	1.00	0.99	- 1.00
8.	1.20	1.25	+ 4.16
9.	1.50	1.54	+ 2.66
10.	2.00	1.93	- 3.50

Table 2. Determination of gold in rock sample.

S. No.	Amount added ug/ul	Amount found ug/ul	Error %
1.	0.22	0.23	+ 4.54
2.	0.35	0.34	- 2.85
3.	0.56	0.58	+ 3.57
4.	0.75	0.79	+ 5.33
5.	0.92	0.90	- 2.17

Table 3. Quantitative assessment of tolerable amount of different metal ions.

	Amount of gold taken (ug/ul)	Amount of metal ion added (ug/ul)	Amount of gold found (ug/ul)	Error %
Fe ³⁺	1.00	0.003	0.90	10.00
	1.00	0.002	0.95	5.00
	1.00	0.001	0.97	3.00
Cu ²⁺	1.00	0.800	0.95	5.00
	1.00	0.900	0.97	3.00
	1.00	1.00	0.98	2.00
Ag ⁺	1.00	0.003	0.90	10.00
	1.00	0.002	0.96	4.00
	1.00	0.001	0.98	2.00
Pb ²⁺	1.00	0.003	0.92	8.00
	1.00	0.002	0.96	4.00
	1.00	0.001	0.98	2.00
Pd ²⁺	1.00	0.030	0.92	8.00
	1.00	0.020	0.95	5.00
	1.00	0.010	0.97	3.00

this procedure was due to iron (111) which was masked by the addition of sodium fluoride. The results of which are reported in Table 2. The quantitative assessment of tolerable amount of different metal ions not interfering in assay by the above method is shown in Table 3. This clearly indicates the accuracy and sensitivity of the method for its use as a routine analysis of various samples.

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