

Emission spectrographic estimation of gold

K C JOSEPH

Department of Geology and Mining, Government of Madhya Pradesh, Raipur 492 001, India

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Abstract. A procedure for estimating minute quantities (parts per billion) of gold is described, based on dissolution of the sample in aqua regia, adsorption on activated charcoal, arcing of the charcoal ash, and measurement of the intensity of the emitted spectra.

Keywords. Emission spectrography; estimation of gold; gold adsorption; arcing of activated charcoal ash.

1. Introduction

The method is based on the dissolution of the sample containing gold in aqua regia. The dissolved gold is adsorbed from the solution by activated charcoal. After desorption of other elements, gold is estimated by its emission spectral intensity at 2675.95 and 2427.98 Å on arcing of the activated coal ash.

2. Procedure

10 gms of the sample ground to – 200 mesh is roasted at 550–600°C for one and a half hours in a muffle furnace, in a flat bottomed porcelain dish making sure that the thickness of the sample material does not exceed 5 mm.

The roasted sample is transferred to a 250 ml beaker and 20 ml of aqua regia are added. The sample is attacked for about 4 to 5 hours at a room temperature of 70–75°C with constant stirring on a hot plate, after which the beaker is removed from the hot plate, cooled and the syrupy material diluted to about 200 ml with water and stirred well. This sample solution is filtered through double ashless filter paper (No. 40 Whatman) using a buchner funnel. The filtrate is collected in a 250 ml conical flask and the residue rejected. 0.5 gms of AR quality activated charcoal, ground to – 200 mesh is added to the filtrate in the conical flask (the ash content of the activated charcoal should not exceed 0.3–0.5 %). The conical flask with the solution is kept on a magnetic stirrer and stirred for 15 minutes at room temperature.

The solution is then filtered through a No. 40 Whatman filter paper using a buchner funnel and suction; the filtrate is rejected. The filter paper containing the sorbed charcoal is torn into small pieces (making sure that no charcoal is lost) and put into the same conical flask and 200 ml of 1 : 50 hydrochloric acid are added. The flask is then stirred for 25 minutes at room temperature. The contents of the conical flask are then filtered through a No. 40 filter paper, which is then transferred to a 30 ml flat-bottomed porcelain crucible and kept in a muffle furnace. The temperature of the furnace is slowly raised to 550°C and kept there for about one to one and a half hours, till the contents of

the crucible are completely converted to ash. During the ashing of the charcoal, access to air should be allowed in the muffle furnace.

2.1 Arcing procedure

After conversion of the charcoal into ash, the crucible is taken out from the furnace and cooled. Into the crucible 15 milligrams of flux* containing sp-2 graphite powder and AR lithium carbonate in the proportion of 1:1 are added. The ash and the flux is then transferred completely to a 50 mm dia small agate mortar with the help of a brush and ground well. The whole of it is then transferred to the crater of a graphite electrode with the help of a funnel and weighing paper. The electrode used was of Union Carbide (USA) make, 0.180" in diameter. The crater is of 8 mm depth and 3.2 mm dia. The crater containing the sample is then kept under an IR lamp for an hour and then arced using a counter electrode of 0.120" dia (Union Carbide, USA) and coned to 60°. The parameters of the arcing are given in table 1.

Gold is estimated by its spectral intensity at 2627.96 Å. When the concentration is higher, the second line is used. The result of analysis is taken as the mean value of two adjacent values of the standard.

3. Preparation of standards.

40 milligrams of pure gold (Fisher Scientific Company, USA) metal was dissolved in 50 ml aqua regia at a temperature of 70–75°C on a hot plate with occasional stirring. The dissolution is carried out in a partially covered 400 ml beaker in 1–2 hours. When the dissolution of gold is complete, the contents of the beaker are completely transferred to a 200 ml volumetric flask by washing the beaker 8–10 times with 6 N HCl

Table 1. Arcing parameters

1. Spectrograph used	1.5 Meter Wadsworth (Jarrel-Ash)
2. Film	Kodak No. 1
3. Current	10 amps DC
4. Preburn	Nil
5. Exposure time	45 seconds
6. Slit width	8 μ m
7. Slit height	1 mm
8. Arcing gap	3 mm adjusted throughout
9. Step filter	100/10
10. Developing	3 minutes at 72°F using Kodak 19 b developer with agitation.
11. Stop bath	30 seconds at 72°F with agitation.
12. Fixing	3 minutes using Kodak acid fixing salt with hardner and with agitation at 72°F.
13. Drying	5 minutes each of cold and hot blowing in automatic photoprocessor (Jarrel-Ash)

*Margaret B Bergen—UN Expert in Spectrography, unpublished report 1981.

and made upto the mark with the same acid and shaken well. The resultant concentration of gold in the solution is 200 ppm or $2 \times 10^{-2} \%$ or 200 gms/tonne. Further dilutions are made as per table 2.

The amount of gold in the above standard is estimated by pipetting out 10 ml each of the above standards into a 250 ml conical flask and then adding 10 ml aqua regia to it. This solution of the standard is then diluted to 150 ml with water. 0.5 grams of activated charcoal is then added and the procedure applied for estimating the sample is repeated for the standard to obtain the spectral intensity of gold at 2675.95 and 2427.96 Å. By a visual comparison of the spectrum of gold in the unknown sample with that of the known standard, the amount of gold in the unknown is easily obtained.

International standards have been analysed by this procedure and the results obtained were found very satisfactory. The results obtained are shown in table 3.

4. Conclusion

In the Department of Geology and Mining, Madhya Pradesh, 1225 samples which include soils, rocks and sediments have been analysed for gold by this procedure. The

Table 2. Dilutions made to prepare the standard solution

Au content in the in- itial gold solution (%)	Volume of initial gold solution taken (ml)	Volume of additional 6 N HCl added (ml)	Final volume of gold solution (ml)	Gold content in final solution	
				(%)	(gms/tonne)
2×10^{-2}	200	Nil	200	2×10^{-2}	200.0
2×10^{-2}	25	25	50	1×10^{-2}	100.0
1×10^{-2}	15	35	50	3×10^{-3}	30.0
1×10^{-2}	5	45	50	1×10^{-3}	10.0
3×10^{-3}	5	45	50	3×10^{-4}	3.0
1×10^{-3}	5	45	50	1×10^{-4}	1.0
3×10^{-4}	5	45	50	3×10^{-5}	0.30
1×10^{-4}	5	45	50	1×10^{-5}	0.10
3×10^{-5}	5	45	50	3×10^{-6}	0.03
1×10^{-5}	5	45	50	1×10^{-6}	0.01
3×10^{-6}	5	45	50	3×10^{-7}	0.003
1×10^{-6}	5	45	50	1×10^{-7}	0.001

Table 3. Results of estimation using international standards

Name of inter- national standard used	Number of estimations done	Average value of gold obtained by this procedure (ppm)	Recommended gold value (ppm)
Gold ore MA-2 [†]	6	Between 1 and 3	1.86*
Gold ore MA-1 [†]	4	Between 10 and 30	17.8*

[†]Standards obtained from Canadian certified, Reference Materials project.

*Canada centre for Mineral and Energy Technology Report 80-6 E, and 81-13 E.

procedure has been found to be accurate for estimation of gold in parts per billion levels and the cost of analysis is relatively very low.

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