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APPLICATION OF ATOMIC ABSORPTION SPECTROPHOTOMETRY TO ANALYSIS OF MILL PRODUCTS FROM METAL MINING OPERATIONS

5. EAGLE GOLD MINES LTD., JOUTEL, QUE.

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R. J. GUEST

EXTRACTION METALLURGY DIVISION

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R.J. Guest*

SUMMARY

Procedures are described for the determination of gold in sample material from Eagle Gold Mines Ltd. and on products from cyanidation treatment of this type of sample material. An unusual feature of some of the Eagle Gold Mines sample material was its apparently large content of graphitic carbon, hence a preliminary roasting treatment was found to be necessary on this type of sample. A study of the effect of variations in analysis results caused by roasting time and temperature was carried out and a recommendation was made for the most satisfactory conditions.

A comparison of results obtained by atomic absorption and fire assay procedures was made, which showed that the two procedures compared very well. Where sufficient data was available, the precision of the atomic absorption procedure was calculated. An estimation of the time required for analyses was provided in order to aid the mine in its evaluation of the applicability of atomic absorption procedures to its process control.

*Research Scientist, Chemical Analysis Section, Extraction Metallurgy Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

INTRODUCTION

A request was received from H.V. Pyke, Manager, Eagle Gold Mines Ltd., Joutel, Que., for an evaluation of the applicability of atomic absorption spectrophotometry for gold analysis on sample material from Eagle Gold Mines Ltd. One sample was submitted but this was relatively high in gold content (1.0 oz/ton) compared to some other feed materials from Eagle Gold Mines being used for leaching tests at the Mines Branch. Accordingly, several of the samples from the metallurgical test program were analysed for gold as it was felt that this would give a more representative indication of the value of atomic absorption procedures for routine analytical work.

The gold procedure used for this investigation was based on ones described in earlier reports in this series, and modified to compensate for the unusual characteristics of some of the sample material from Eagle Gold Mines. Primary consideration was given to the development of a procedure which would best combine speed with reasonable accuracy and precision. A comparison was made, therefore, between results obtained by atomic absorption procedures and fire assay, and an estimation made of the time requirement using atomic absorption procedures.

The type of sample material analysed varied widely and consisted of (a) high-grade ore, (b) a mixture of low-grade and high-grade ore, and (c) residues resulting from cyanidation tests.

Mineralogical studies of the head samples used in these tests were carried out here and have been described by Kaiman[1]. These samples, which contained large proportions of pyrite, varied widely in carbonaceous material, and apparent graphitic carbon. Some of the samples analysed were products from tests on a low-grade gold ore containing a large amount of black carbonaceous material (graphite).** It was understood that this type of sample material represented a typical mixture of low-grade and high-grade ore from Eagle Gold Mines.

EXPERIMENTAL - ANALYTICAL PROCEDURE

Determination of Gold

Gold is determined following its extraction with methyl isobutyl ketone (MIBK) from a chloride medium (2). The gold content of the ore is leached by either hot or cold aqua regia. Although complete dissolution of the sample is not obtained by this treatment, the gold content is effectively

** hereafter referred to as graphite or graphitic.

leached providing the sample is adequately ground and, for certain types of sample material, has undergone a preliminary roasting step.

Roasting

If roasting of the sample is necessary (graphite present), weigh the sample into a porcelain crucible and roast at 635°C for two hours with occasional mixing. (See footnote (c), page 5). Transfer to an appropriate beaker or flask for leaching.

Sample dissolution

Hot aqua regia leach

Weigh out an appropriate sample (0.3 - 15g) of the well-pulverized ore (minus 200-mesh) into a 100 ml or 250 ml beaker (see Roasting) and add 40 ml of aqua regia. Digest on a low-heat hot plate for 1-2 hours, cool, and transfer the solids and solution into a separatory funnel capable of holding approximately 150 ml, dilute to 100 ml with water, and add enough hydrochloric acid to make its strength 25%.

Alternatively, following sample digestion, filter the sample using a Buchner funnel and Whatman No. 3 paper, washing the residue well with water or weak hydrochloric acid solution, and transferring the filtrate and washings into a separatory funnel capable of holding approximately 150 ml. Add enough hydrochloric acid to make its strength 25%.

Cold aqua regia leach

Weigh out an appropriate sample (0.3g - 15g) of the well-pulverized ore (minus 200-mesh) into a 300 ml Erlenmeyer flask and add 40 ml of aqua regia. Place on a suitable mechanical shaker and allow the sample to leach in the cold for 1-2 hours with moderate mixing. Transfer the solids and solution into a separatory funnel capable of holding approximately 150 ml, dilute to 100 ml with water, and add enough hydrochloric acid to make its strength 25%.

Alternatively, following sample digestion, filter the sample using a Buchner funnel and Whatman No. 3 paper, washing the residue well with water or weak hydrochloric acid solution, and transferring the filtrate and washings into a separatory funnel capable of holding approximately 150 ml. Add enough hydrochloric acid to make its strength 25%.

Carry an aliquot of a standard gold solution through whichever of the above procedures is to be used, during the procedure on the sample.

Extraction

The volume of the sample and standard solutions should be approximately similar at this point, and the number and volumes of extractant portions used should be the same for any particular group of samples which are to be compared. For this reason, group low-grade samples separately from high-grade samples and analyze each group, with its appropriate standards, using a fixed routine of extractions and washings(a).

Add 10 ml or more of methyl isobutyl ketone (Fisher), depending on the amount of gold present (Table 1), shake vigorously for 2 minutes and allow the layers to separate. Drain off and discard the aqueous layer. Wash the organic layer three times by shaking with 30 ml of 15% hydrochloric acid to remove contaminating ions. If the third wash is coloured, continue washing with acid until no further change is noted. Filter a portion of the organic fraction through a Whatman No. 40 filter paper and collect the filtrate in a small test tube or other suitable container.

Atomic Absorption Measurement

Carry out two or more atomic absorption readings and compare the readings with those obtained on prepared standards read at the same time as the samples (b). Use a recorder for the measurement if greater precision is required.

(a) The relationship between the gold present in the original aqueous solution and the absorbance in the organic medium tends to depart from linearity as the gold content of the MIBK extract increases. In addition, the final volume of the organic extract obtained will depend both on the volume and nature of the aqueous layer, due to the solubility of the organic solvent in the aqueous portion. Although the more accurate procedure would be to transfer the organic layer to a suitable volumetric flask and dilute to the mark with MIBK, for most work, we prefer to use the organic extracts directly for the atomic absorption measurement, to shorten the time required for the determination and to aid in providing the higher sensitivity required for the determination of traces of gold.

(b) Some loss of accuracy would be caused by using standard curves because of the difficulty of obtaining identical operating parameters each time an analysis is carried out.

For details of the operating procedure refer to APPENDIX.

TABLE 1

Suggested Sample Size for Gold Determination
on Various Types of Sample Material

Sample Type	Gold Concentration oz/ton Au	Sample Size Grams	Methyl Isobutyl Ketone Added ml MIBK
Feed	1	2	40
Feed	0.4	5	40
Feed	0.2	5	25
Residue	0.08	10	25

RESULTS AND DISCUSSION

It was found that atomic absorption procedures for gold
gave results on Eagle Gold Mines Ltd. sample material which were
in good agreement with those found by Fire Assay at the Mines
Branch (Table 2), providing certain modifications were made in
the procedure when analysing some types of sample material. A
procedure using aqua regia attack, either in the cold with
continual mixing or using low-temperature heating, appeared to work
well on high-grade feed material. This procedure did not prove to
be satisfactory, however, on comparatively low-grade, high-graphite,
sample materials, because results were very low. It was found necessary,
therefore, on this type of sample, especially, to carry out a
preliminary roast in a muffle furnace to convert the ore into a
form amenable to acid attack. Roasting was also found to be
necessary in one of the tests, (E-7 Residue, Table 2), on a residue
from a cyanidation test on what is believed to have been ore of
low graphite content.

Results of tests comparing results with and without a roasting step, and the effect of varying roasting conditions, are shown in Table 3, and Figure 2. It was clear from these results that the roasting temperature and roasting time were of vital importance in obtaining satisfactory results. Our choice for the most satisfactory temperature, from the standpoints of effective roasting and speed was 625 to 650°C for two hours (c). Higher temperatures tended to cause low results, possibly due either to the gold being volatilized, or being rendered more insoluble to acid attack. During these tests, however, no evidence of sample "dusting" was observed. Lower temperatures (600°C) gave good results but the roasting step required a longer period of time and practical considerations would put a limit on the amount of time allowable.

A brief evaluation of the precision to be expected from the procedure was made on two feed samples which did not require a roasting step. Calculation of the precision was made according to the procedures of Dean and Dixon (3) and Bauer (4), where four or more results were available. It was found that the precision obtained from procedures, using either hot or cold leaches, was similar (Table 4).

It was not fully established whether or not there is a consistent difference in results between procedures using a hot and cold acid leach. We used a hot leach for most of the work as it was more convenient due to some problems with our mechanical shaker.

Following roasting and acid leaching, a set of 20 samples can be analysed readily within a few hours by the procedure described here. Variations in laboratory organization, and requirements for precision and accuracy, will cause time requirements to vary widely.

The recommended procedure of the manufacturer (5) for using the hollow-cathode lamp and atomic absorption spectrophotometer was used with some modification. Details of the instrumental procedure used are given in the APPENDIX.

(c) For the roasting operation, we used Coors Porcelain Crucibles, type 1A, upper diameter of 5 cm, lower diameter 2.5 cm, height about 4 cm, to hold samples of 5 grams for the feed, and 10 grams for the residue. By using a much wider dish, and a smaller sample size, it is believed that considerably less time would be required for satisfactory roasting.

TABLE 2

Comparison of Gold Results by Atomic Absorption and Fire Assay

Sample Type	Fire Assay (a) oz/ton Au	Atomic Absorption Oz/ton Au(e)(f)			
		Sample Not Roasted		Sample Roasted (b)	
		Procedure 1(c)	Procedure 2 (d)	Procedure 3 (c)	Procedure 4 (d)
Feed sample A (sent by Eagle Gold Mines especially for A.A. analysis - low - graphitic)	1.01	0.98(7)	1.00(4)	–	–
Feed sample B (high-grade feed diluted to an unknown extent with low-grade graphitic feed)	0.38	0.36(5)	0.36(2)	–	–
Feed sample C (low-graphite)	0.45	0.43(2)	–	–	–
Feed sample D (high-graphite)	0.197	0.019(4)	–	–	0.19(3)
Residue(E-7) from cyanidation tests on feed sample of 0.71 oz/ton Au	0.10	0.01-0.04(3)	–	–	0.097(2)
Residue (E-9) from cyanidation tests on feed sample C (above)	0.04	0.027(2)	–	–	0.034(2)
Residue (E-10) from cyanidation tests on feed sample D (above) using cresylic acid	0.10	0.016(3)	–	–	0.11(2)
Residue (E-16) from cyanidation tests on feed sample D (above) using kerosene	0.075	0.006(3)	0.006(1)	0.063(1)	0.072 (5)

(a) Mineral Sciences Division Laboratory
(b) Roasted at 625 - 650°C
(c) Cold leach with aqua regia
(d) Hot leach with aqua regia
(e) The figure in parenthesis represents
     the number of determinations averaged
(f) All results obtained by procedure in which solids and
     solutions are extracted with MIBK

TABLE 3

Effect of Roasting on Gold Results

Sample	Roasting		Leaching	
	Temperature °C	Time Minutes	Cold Aqua Regia	Hot Aqua Regia
Feed (0.197 oz/ton) high graphite content 5 gm sample	unroasted		0.019	–
	650	30	0.11	0.11
	650	45	0.20	0.19
	650	90	0.19	0.18
	650	120	–	0.19
	650	180	–	0.195
	700	120	–	0.18
	700	180	–	0.185
Residue from feed (above) 10 gm sample	unroasted		0.006	0.006
	600	120	–	0.049
	600	180	–	0.077
	625	120	–	0.073
	625	180	–	0.073
	650	30	0.015	0.009
	650	45	0.017	0.017
	650	90	0.066	0.072
	650	120	–	0.070
	650	180	–	0.072
	675	120	–	0.058
	675	180	–	0.058
	700	120	–	0.051
	700	180	–	0.045

The cost of our atomic absorption equipment, including the spectrophotometer, chart recorder, digital readout, and hollow-cathode lamps, would be about eleven thousand dollars. Alternative instruments, with their accessories, are readily available at prices beginning at less than four thousand dollars. A large cylinder of acetylene (about 300 cu. ft.) would last from one to two weeks, assuming that the spectrophotometer was in almost constant use for one eight-hour shift each day.

TABLE 4

Precision of Analytical Results Obtained for Gold
on Samples from Eagle Gold Mines Ltd.

Sample Type	Individual Determinations by Atomic Absorption oz/ton Au		Average Result oz/ton Au		Precision			
					Standard Deviation		95% Confidence Limits for Average Result	
	Cold Leach Aqua Regia	Hot Leach- Aqua Regia	Cold Leach	Hot Leach	Cold Leach	Hot Leach	Cold Leach	Hot Leach
Feed	1.01 0.97 0.95 1.00 0.96 0.99 0.96	1.00 1.01 1.00 0.98	0.98	1.00	0.022	0.014	0.020	0.022
Feed	0.34 0.32 0.35 0.37 0.38	-	0.35	-	0.026	-	0.032	-

APPENDIX I

Atomic Absorption Operating Procedure - Gold

Carry out two or three readings for each determination, reading against MIBK as the blank. Use of a recorder will provide a considerable improvement in precision.

Instrumental conditions were as follows:

Hollow-cathode lamp	- Atomic Spectral Lamps, Australia, single element
Photomultiplier tube	- Hamamatsu R 106
Lamp current	- 4 mA, regulated
Phototube circuit	- 500 volts
Monochromator slit	- 300 microns (9.9Å bandpass)
Wave length	- 2428 Å
Burner height	- organic medium, 12mm below centre of light path for best sensitivity; 6mm below centre of light path for best stability. - aqueous medium, 6mm below centre of light path for best sensitivity; stability similar for 6- and 12-mm settings.
Flame type	- organic medium, an oxidizing flame gave about 20 per cent greater sensitivity than a reducing flame, but the flame setting is harder to maintain than when using the fringe shown by a slightly reducing flame. - aqueous medium, an oxidizing flame gave slightly better sensitivity than a reducing flame especially at smaller distances below the centre of the light path. The oxidizing flame setting is harder to maintain than when using the fringe shown by a slightly reducing flame.
Air supply	- 15 psi
Atomizer	- variable type
Sample uptake	- 3.5 ml per minute
Flame emission	- 2 per cent signal (with lamp off, the difference between flame off and flame on)
Flame absorption	- 10 per cent signal (with lamp on, the difference between flame off and flame on)

Sensitivity — 1-per-cent absorption - organic, 0.07ppm (approx)
— 1-per-cent absorption - aqueous, 0.25 " "
— 20-per-cent absorption - organic, 1.5 " "
— 20-per-cent absorption - aqueous, 5.4 " "

Calibration - Gold

A plot of absorbance versus concentration for gold in organic and aqueous medium, is shown in Figure 1.

With the described procedure, the relationship between the gold present in the original aqueous solution and the absorbance in the organic medium tends to depart from linearity as the gold concentration of the MIBK increases. It has been found more satisfactory when working in organic medium to use the lower end of the gold concentration range (ie, less than 0.125 absorbance). Thus, for higher concentrations it is advantageous to increase the amount of MIBK used, choose standards to cover this range by reference to Figure 1, and carry them through the procedure with the samples. The standards are prepared for extraction in aqueous chloride medium and, after extraction, the organic medium is atomized directly for both samples and standards. A stock gold solution is made up from gold metal or a suitable salt of gold, dissolved in aqua regia, diluted and made up to volume.

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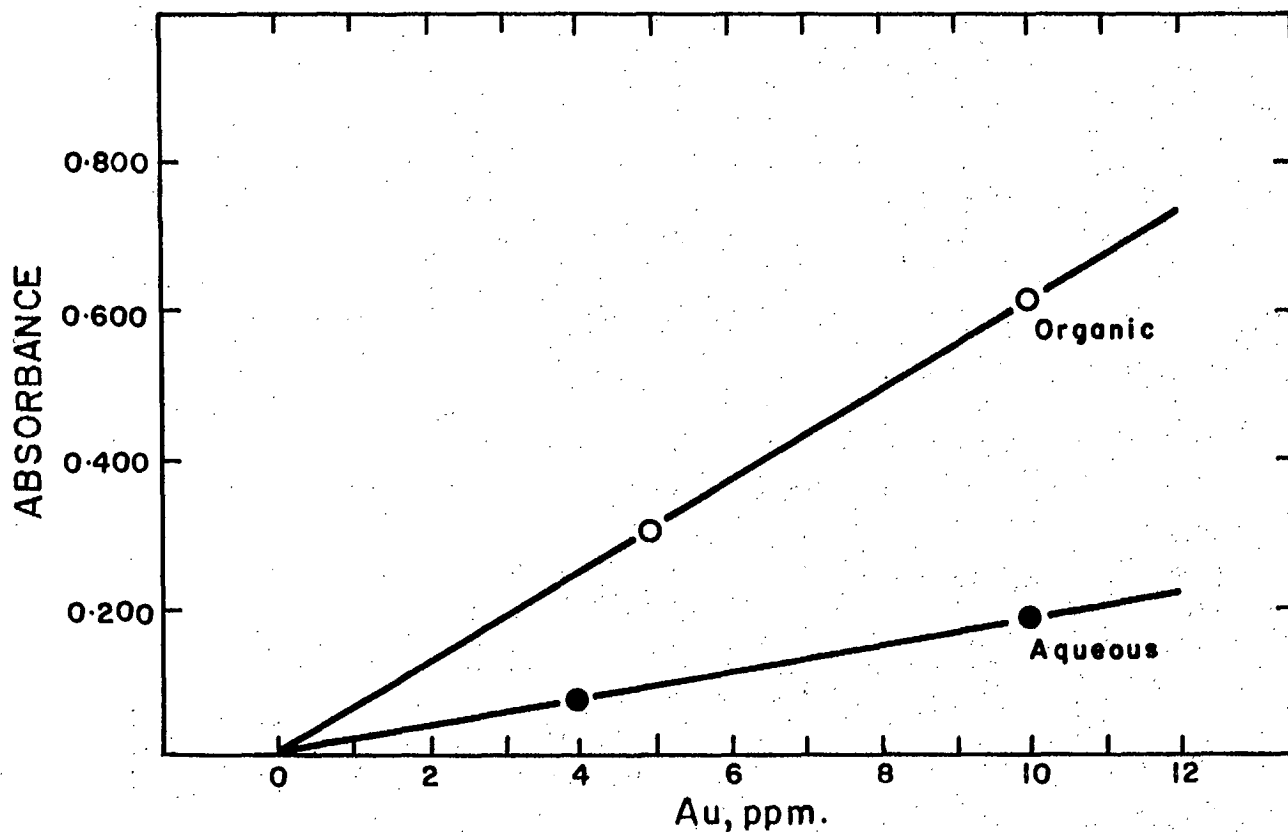
ACKNOWLEDGEMENTS

Figure 1. Comparative Sensitivities of Gold Determination in Aqueous and in Organic Medium, by Atomic Absorption Spectrophotometry (Techtron AA3 Spectrophotometer without Scale Expansion).
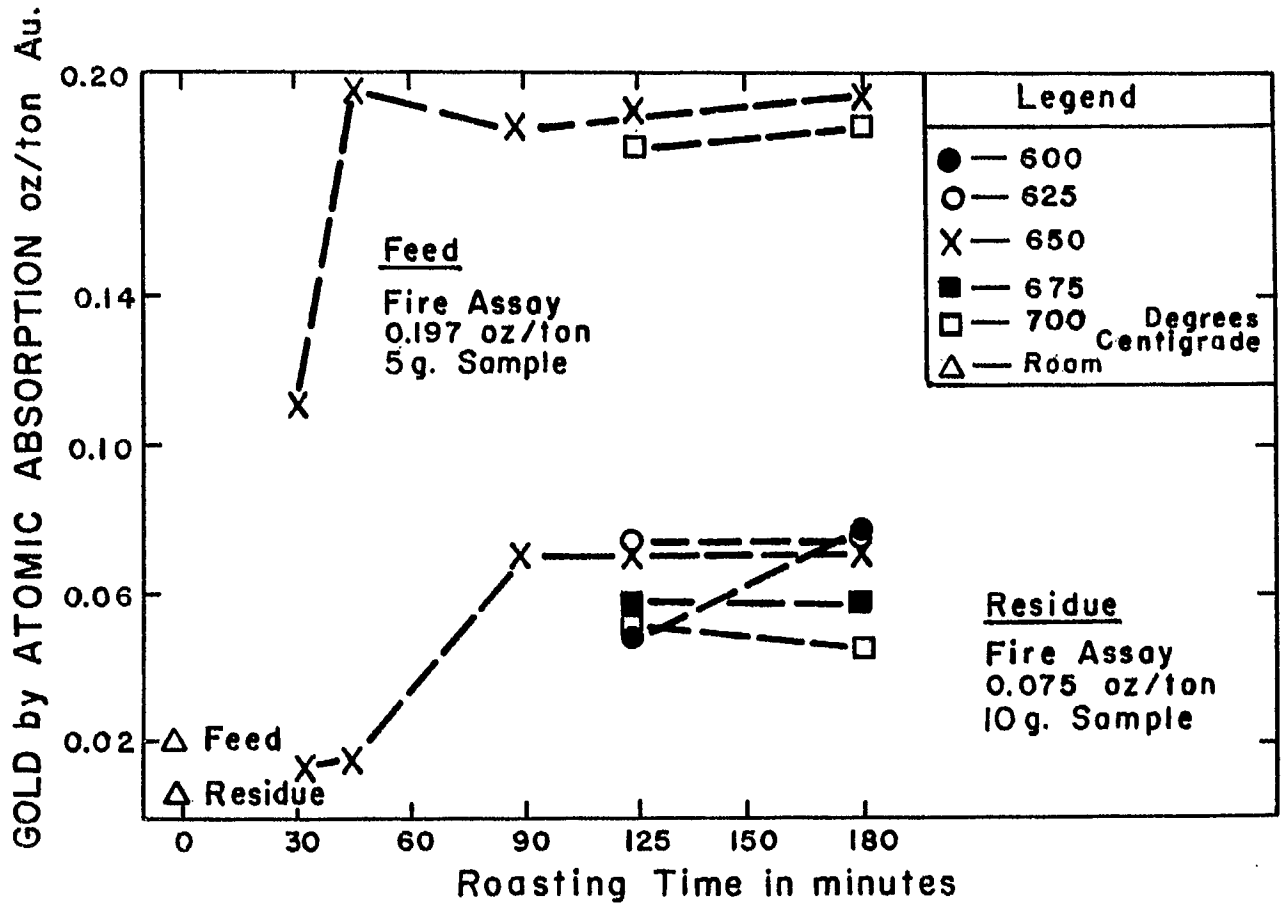
Figure 2. Effect of Temperature and Time of Roasting of Eagle Gold Sample Materials, on Atomic Absorption Assays.