

Gold Analysis — Fire Assaying and Alternative Methods

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Abstract — This paper describes fire assaying and ‘high tech’ alternatives for gold analysis, including advantages and disadvantages of each method. These ‘high tech’ methods include INAA (instrumental neutron activation), *aqua regia* digestion/AA (atomic absorption), GFAA (graphite furnace-atomic absorption) or ICP/MS (inductively coupled plasma emission mass spectrometry) and a combination of cyanidation with AA, GFAA or ICP/MS methods.

Fire assay remains the stalwart among analytical methods for gold but, even with its multiple analytical finishes, is not fool-proof for recovering 100% of the gold present. Consumers of assays should not blindly trust the assay results without consideration of quality control data. Laboratories can and do make mistakes. Some of these can be easily discovered if *you do your own due diligence*.

All these measures should ensure that gold analysis values from successive campaigns and from different laboratories meet acceptable levels of accuracy and precision, and can be correlated together, particularly when they occur in neighboring holes. This is essential just as much for prospect evaluation as for project feasibility. © 1999 Canadian Institute of Mining, Metallurgy and Petroleum. All rights reserved.

Introduction

The nature of many gold deposits (low grade — high tonnage) and the increasing sophistication of deposit assessment procedures and exploration geochemistry demand better quality analytical data, particularly for the lower range of gold values near and below cut-off grade. Modern geological models attribute formation of many of these gold deposits to hydrothermal solutions that have added elements other than gold, or leached some elements from the host rock, so that there is usually a multi-element metasomatic effect in and around a deposit. Analyzing gold by itself rarely will provide useful details regarding geochemical halos, environmental baseline levels for potential problem elements and data on potential metallurgical complications. Although fire assay remains the analytical method of choice for gold deposit evaluation, there are other viable modern alternatives, many with a multi-element capability; time and technology have not stood still.

This paper describes fire assaying and ‘high tech’ alternatives for gold analysis, including advantages and disadvantages of each method. These ‘high tech’ methods include INAA (instrumental neutron activation), *aqua regia* digestion/AA (atomic absorption), GFAA (graphite furnace-atomic absorption) or ICP/MS (inductively coupled plasma emission mass spectrometry) and a combination of cyanidation with AA, GFAA or ICP/MS methods.

Sample Preparation Considerations

Sample preparation, although not the main theme of this contribution, is critical to producing “correct” results and a few comments are warranted. These will not address the actual sampling representivity itself but are concerned with the subsequent sub-sampling to produce a smaller mass for analysis; this small mass must be representative of the material submitted.

Today, the most widely accepted procedure for rock preparation is to submit the entire sample to a modern jaw crusher and crush to about 10 to 20 mesh on the primary crushing. The crushed sample is then split mechanically, either in a riffle or rotary splitter, and a sub-sample is pulverized in a ring and puck (swing) mill. The pulverized material can range from several hundred grams to 5 kg in a single milling. Sample size and pulverized dimension should be based on Gy’s sampling formula; selection can be eased by using a sample reduction diagram (François-Bongarçon, 1998; Sketchley, 1998).

A commonly encountered problem with sample preparation involves the dust collection system. In an attempt to reduce airborne contamination, a laboratory may use too efficient a dust collector which sucks out the fines preferentially. In one investigation of a mine laboratory, the authors could actually hear the sample rattling down the dust collector. This was exacerbated by the use of a disc pulverizer connected to the same dust collection system; this situation caused an apparent increase in gold grade of 30% by prefer-

Table 1. Comparison of effective range of analytical methods for Au and comparison of multi-element capabilities, turnaround and cost

Au ppb									Multi-element	Possible* Turnaround Days	Cost US\$ (30 g sample)
0.01	0.1	1	10	100	1000	10 000	100 000	1 000 000			
----- FA-GRAVIMETRIC -----									no	1-3	9-12
----- FA-AA, DCP, ICP -----									yes (Au, Pd+Pt)	1-3	8-10 (Pt, Pd + \$5)
----- FA-INAA -----									no	2-4	10-11
----- INAA -----									yes "Au+34"	8	10-15 (multi incl.)
----- BLEG-GFAA -----									no	2-4	14-40 (1 kg sample)
----- BLEG-ICP/MS -----									yes "Au+50"	2-4	35 (multi incl.) (1 kg sample)
----- CYANIDATION-AA -----									no	2-4	6-8
----- AQUA REGIA-AA -----									yes "Au+30"	1-3	5-7 (multi + \$5)
----- AQUA REGIA-GFAA -----									yes "Au + 30"	1-3	7-10 (multi + \$5)

* Assumes no other samples in the lab.

entially sucking out and discarding the fines. Native gold will have a tendency to smear out on disc pulverizer plates which could lead to contamination of subsequent samples. Widespread use of hammermills in Australia stopped when it was discovered that these mills acted as dry placers and were concentrating gold within the mill. This could lead to lower values in some samples or contamination in others.

We have now reached the analytical stage. Are low level (geochemical) or ore grade (assay) values required? Table 1 indicates an approximate range of usefulness for each of the techniques. Make sure to specify the analytical method and sample size required to the laboratory. Finally, providing feedback to the laboratory can only help to improve the quality of the analytical data.

Conventional Fire Assaying

Procedure

The method of choice for gold analysis through time has been fire assay. The first recorded use of the fire assay technique was that of Erker — assayer to the Holy Roman Empire. In fact, fire assaying probably even predated that time. The method used has not changed appreciably with time except perhaps with better quality reagents and furnaces and now there are a number of potential modern analytical finishes to "read" the fire assay bead, in addition to the classical weighing of the bead. In resource/reserve estimation it is common practice to reduce a sample in size (mass) to one assay ton (ca. 30 g) for analysis, although larger and small amounts are used in certain cases.

The basic procedure for fire assay involves mixing an aliquot of powdered sample (10 g, 15 g, 30 g, or 50 g are the common sizes used) with soda ash (sodium carbonate), borax (sodium borate), litharge (PbO), flour (baking flour used to add carbon as a reductant), silica and possible nitre (potassium nitrate). To this mixture, Ag or Pd as a collector can be added in solution or as a foil. The well mixed mater-

ial is fired at temperatures ranging from 1000°C to 1200°C (depending on the laboratory). As the Pb and Ag (or Pd) in the melt settle to the bottom of the crucible, it scavenges the Au (\pm Pd, Pt) from the melt.

The hot molten mixture is poured into a mold and when cool, the slag should, but does not always separate cleanly from the Pb button, which should be a certain weight if the proper sample flux has been used. This lead button is placed into pretreated cupels (made from magnesia or bone ash) and the Pb will be absorbed into the cupel leaving an Ag or Pd bead (depending on collector used) which has collected the gold and some of the platinum group elements.

The gold is separated from the Ag bead by parting (dissolving in nitric acid), leaving a bead which can be weighed (gravimetric finish). Alternatively, the entire bead can be dissolved in acid and Au (\pm Pt, Pd) can be determined by atomic absorption (AA) most commonly. Less common analytical methods include ICP (inductively coupled plasma emission spectrometry), ICP/MS (inductively coupled plasma emission mass spectrometry, for best sensitivity for Au, Pt, and Pd), or DCP (direct current plasma emission spectrometry). The bead can also be read directly by irradiation followed by instrumental neutron activation (INAA) for lowest detection limits. A variation of fire assay called metallic screen analysis is used for samples where coarse gold may be a problem for obtaining representative assays. This involves crushing, pulverizing and screening the entire sample using a ring and puck pulverizer. The gold is assayed on the +80 mesh (or +150 mesh) material and the +80 mesh (or +150 mesh) material. Based on a weighted average of the two-size fraction components, a final assay is determined. Details of various fire assay procedures are given by Beamish and Van Loon (1977).

Advantages and Disadvantages of Fire Assaying

Fire assaying remains the industry standard procedure for most mineral deposit evaluations. Where conscien-

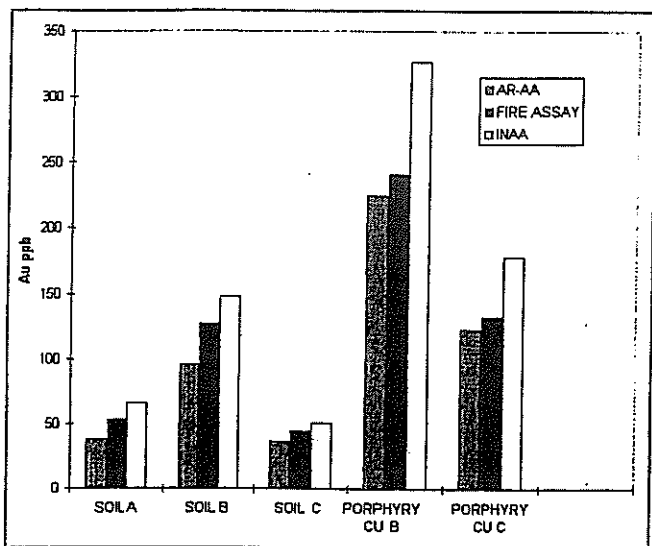


Fig. 1. Comparison of analytical techniques for gold (from Hall et al., 1989).

tiously performed, the procedure produces high-quality, reproducible results for most natural materials and mill products. The principal advantages include the long history of success of the method, easy adaptation to a mine setting, moderate cost and rapidity with which results can be obtained. Nevertheless, there are places in the procedure where errors can arise if appropriate care is not taken. Potential sources of error, avoidable with care, are discussed below.

During the fire assay procedure, most steps occur in unmarked crucibles and cupels and the procedure is very labor-intensive. This can easily result in sample mix-ups. Samples can, however, be monitored by insertion of blanks,

Table 2. Fire assay results from two Fire Assay-AA runs of the CANMET UMT-1 versus INAA gold values. Example of improper fluxing not in homogeneity of sample.

First Run Fire Assay-AA 30 g Aliquot		Second Run Fire Assay-AA 30 g Aliquot		INAA 30 g Aliquot
14	9	38	40	49
11	13	26	39	49
4	10	37	42	48
10	11	39	34	51
16	6	40	42	49
21	11	45	40	47
13	25	30	41	55
11	19	23	43	53
12	21	35	36	53
8	22	41	37	54
12	15		43	48
16	25			53
12	18			61
11	14			57
15	12			48
14	24			53
Average = 14 +/- 5		Average = 38 +/- 6		Average = 52 +/- 4

Average fire assay 14 ppb (32 aliquots) range: 4 to 25
 Average fire assay 38 ppb (21 aliquots) range: 26 to 45
 Average INAA 52 ppb (16 aliquots) range: 48 to 61

standards and identified samples so that certain types of mix-ups can be caught (i.e., turning around trays).

Certain matrices, for example, sulfide-bearing rocks or rocks with higher levels of base metals, must be pre-treated (roasted, leached or fluxed differently) and rocks of differing composition must be properly fluxed to achieve good recovery of precious metals. Hall et al. (1989) submitted five reference standards under the guise of samples to 15 reputable, widely used commercial laboratories and obtained some remarkable variation in gold assays. The worst results by fire assay were obtained from two porphyry copper standards with about 0.5% Cu. Details are shown in Figure 1. Other authors (Diamantatos, 1984, 1987) have also commented on the deleterious effect of Cu on Au collection by fire assay.

Table 2 shows replicate results on UMT-1, a CANMET mine tailings standard from an ultramafic host rock, which were submitted for homogeneity testing for Au by fire assay. Thirty-two, 30 g aliquots were fire assayed. First results ranged from 4 to 25 with a mean of 14. When the fire assay laboratory was informed these were standards and should be homogenous, 20 aliquots were re-fired and a higher recovery obtained (range of 26 ppb to 45 ppb — average 38). INAA analysis returned values of 52 ppb \pm 4 ppb. The certified value for gold is 48 ppb \pm 2 ppb. In this example, improper fluxing on both fire assay sets resulted in unacceptably low and erratic results.

Fire assaying utilizes a method blank which is usually 1 ppb to 3 ppb for a 30 g sample, but can routinely be much higher depending on the source of litharge (PbO) in the charge. Some laboratories' blanks are as bad as 50 ppb to 100 ppb and can result in a small but significant bias. Erratic gold values can also occur from contamination of crucibles by material which has accumulated on the roof of the furnace from "spitting" of samples as they are being fired. This 'roof' material can then drip down into crucibles fired later in the furnace. Occasional erratic blanks are as high as 1000 ppb to 2000 ppb. Heterogeneity of the sample is not always the answer when trying to explain why gold values do not reproduce; contamination is a possibility.

For analysis of gold, the gold must be put into solution (for atomic absorption analysis) or separated from the silver (Ag) gravimetric analysis, a process called parting. Occasionally when parting the Ag doré bead, the gold particles creep up the side of the test tube or parting dish as a result of surface tension and might not report to the assay (i.e., the assay says no gold even where gold is present). The authors have seen this problem with samples containing as much as 2000 ppb Au, although the smaller the Au abundance, the more likely this effect.

As with many analytical methods, the detection limits reported for much of the fire assay data from commercial laboratories are highly optimistic and should realistically be computed by the end-user with the formula: detection limits = blank + 3 times the standard deviation of the blank (Standards Council of Canada, 1997).

Fire assay methodologies vary from one laboratory to another. Some laboratories re-use crucibles (potential

contamination), some have lower flux to sample ratios or poorer-quality reagents (saves a few pennies in reagent costs), some decrease fusion time (higher throughout but perhaps not total gold recoveries) some do not custom flux based on sample composition (poor recovery). Some do not check high gold values (slows turnaround and adds costs). It is imperative to do your own due diligence and ask questions of your lab. There is a possibility that a "bogus" laboratory with a special or proprietary fire assay method will contaminate samples with reagents used, will not measure method blanks or will use some inappropriate analytical finishes to produce incorrect results.

Instrumental Neutron Activation Analysis (INAA)

Procedure

The INAA procedure is inherently simple. It involves encapsulating the sample powder in a polyethylene capsule. Samples can vary from a few milligrams (advantages for looking at selected mineral grains) to as large as a kilogram. Most commonly, a 30 g sample size is used for homogeneity and cost reasons. The samples are packaged together with control reference standards and an appropriate neutron flux monitor. The samples are then irradiated in a nuclear reactor. The neutrons from the reactor bombard the sample and interact with the nucleus of the elements present. Gold in nature occurs as the Au-197 isotope. When a neutron is added to the nucleus it becomes Au-198. The Au-198 decays back to Au-197 with a fixed half-life of 64.8 hours (half-life is the time it takes for 1/2 of the activated Au-198 to return to Au-197). In the process of decay, a gamma-ray specific to gold (energy 411.8 KeV) is emitted. A high-purity germanium detector measures these gamma-ray decays and through a series of electronics and multichannel analyzer the gamma-rays are sorted and quantified.

Generally, for geological materials, the samples are measured seven days after irradiation. This decay time allows Na-24 which has a half-life of 14.96 hours to decay away, creating a much lower background so that very low levels of gold in geological material can be measured along with 34 other elements simultaneously. The gamma-rays counted are corrected for decay time from the end of irradiation and for sample weight and compared (using the flux monitor) to a calibration previously developed from multiple certified reference materials. The control standard co-irradiated is an independent check on the analysis and is not used for calibration. The method has been described by Hoffman (1992) in some detail. The suite of elements obtainable is listed in Table 3.

Advantages and Disadvantages of INAA

The advantages of the INAA technique include: no chemical treatment required (simplicity = reliability); no

Table 3. Elements and detection limits for "Au + 34" by INAA

Au	2 ppb	Hf	1 ppm	Se	3 ppm
Ag	5 ppm	Hg	1 ppm	Sm	0.1 ppm
As	0.5 ppm	Ir	5 ppb	Sn	0.01%
Ba	50 ppm	La	0.5 ppm	Sr	0.05%
Br	0.5 ppm	Lu	0.05 ppm	Ta	0.5 ppm
Ca	1%	Mo	1 ppm	Th	0.2 ppm
Ce	3 ppm	Na	0.01%	Tb	0.5 ppm
Co	1 ppm	Nd	5 ppm	U	0.5 ppm
Cr	5 ppm	Ni	20 ppm	W	1 ppm
Cs	1 ppm	Rb	15 ppm	Yb	0.2 ppm
Eu	0.2 ppm	Sb	0.1 ppm	Zn	50 ppm
Fe	0.01%	Sc	0.1 ppm		

blank to subtract which results in very reliable low level data; method is non-destructive which is ideal if one wishes to re-examine samples which have provided surprises (not expecting gold but found it); and the true detection limit is 2 ppb on rocks, soils or sediments and 0.1 ppb on vegetation. The gold value provided will be total gold regardless of the form it may be in (organic complex or silica encapsulated). The method is matrix independent and suffers from no interferences. Samples analyzed by INAA can range from a fraction of a gram to a kilogram with no loss of sensitivity.

INAA is cost-effective, especially where other elements like As (0.5 ppm), Sb (0.1 ppm), Ba (50 ppm) or W (1 ppm) are required. The multi-element package also provides excellent low level Sb analysis (far superior to ICP Sb) and provides a total Ba value (barite is not soluble in any ICP acid extraction methodology other than fusion). Barite has increasingly been recognized as an indicator of mineralization formed by hydrothermal fluids.

The main disadvantage of the technique is the seven-day wait for decay of sodium prior to the analysis. Occasionally, very high levels of certain elements like Sb (500 ppm), rare earths (5000 ppm), and uranium (500 ppm) may cause an elevation in background and hence an increase in detection limits. In no case will this preclude the actual measurement of gold but may elevate the level which can be detected say from 2 ppb to 10 ppb. Certain INAA laboratories use "epithermal" neutrons for irradiation. This may cause a low bias depending on Au particle size due to an effect called "self-shielding." In this case, the gold particle is such a good absorber of epithermal neutrons that the neutrons will only activate the outer layers of coarse gold particles. This effect can, however, be avoided by using thermal neutrons. A final but significant disadvantage is that only a certain design of nuclear reactor can be used for commercial INAA and these reactors have very limited availability in the world.

Aqua Regia Dissolution — AA, GFAA or ICP/MS Finish

Procedure

The *aqua regia* dissolution procedure involves digestion from 5 g to 50 g (or possibly higher) of sample in *aqua regia* (a mixture of hydrochloric and nitric acid). This must

be followed by a solvent extraction (MIBK or DIBK) if AA or GFAA will be used as an analytical finish. The organic extraction is not necessary for an ICP/MS finish. The reason for this organic extraction is to leave behind Fe which will cause spectral interference on Au or platinum group metals and introduce analytical error which can be extreme. Minor Fe lines in the emission spectra look exactly like Au, Pt or Pd and can give very erroneous high values.

Advantages and Disadvantages

The advantage of the technique is that it offers the lowest cost of the gold analysis methods. This technique allows one to add a low-cost routine *aqua regia*-30-element ICP package to the gold analysis. This ICP "add on" trace element package is primarily useful for base metals and Ag exploration. Although As and Sb are included in the ICP package by most laboratories, the data for low levels (<30 ppm to 50 ppm) of these two elements is rarely of adequate quality and, in fact, Sb results are generally useless. In addition, Ba coming from barite will not report at all to an *aqua regia* digestion. Results can be obtained rapidly in as little as a few hours.

Depending on mineralogy, results for gold obtained by this method will usually be lower than reported by fire assay or INAA. This may be due to silica encapsulation or perhaps to the gold mineralogy itself. In a study of five reference materials, Hall et al. (1989) found on average that the *aqua regia* - AA values on soils were only 58% to 70% of the INAA values, and for porphyry Cu standards, only 69% of the INAA values (Table 2). They did note, however, that some ore samples could show recoveries of 100% depending on the nature of the samples. *Aqua Regia* - AA is widely used in Australia for grade control in the mine environment and in exploration as it offers the lowest cost choice for gold analysis. *Aqua regia* methodologies vary from one laboratory to another, as will recovery levels.

Various other acid digestions can be used in place of *aqua regia* depending on the problem to be solved. These various digestions are described by Chao and Sanzalone (1992).

Cyanidation (BLEG)

Procedure

BLEG (Bulk Leachable Extractable Gold) involves weighing a large sample, usually 1 kg to 5 kg into a polyethylene bottle, adding an appropriate cyanide solution (0.25% to 1% NaCN, depending on the lab) and agitating (bottle rolling) for various periods of time, ranging from a few hours to several days. We have seen every variation of sample preparation performed prior to encapsulation in the polyethylene bottle. The samples should be treated consistently whether sieving to -80 mesh or -200 mesh. The result on a -80 mesh fraction may look completely different to the results on a -200 mesh fraction of the same sample. Don't

just send the sample to the lab and ask for BLEG analysis. Sample size, particle size, strength of NaCN leach solution, agitation time and method must be specified. The required parameters will vary depending on the property. It is important to know how the gold occurs on the property prior to running this method or to at least experiment with different parameters prior to the final choice for a survey. The gold can then be extracted into an organic solvent (to avoid Fe interference) and run by AA or GFAA for better detection limits. Some laboratories do not do an organic extraction. This may result in reporting gold values that are too high, particularly in the low gold range. Alternatively, the sample can be analyzed prior to the organic extraction by ICP/MS for a best detection limit of 0.05 ppb. With the ICP/MS method, Pt and Pd can be analyzed concurrently (to 0.05 ppb) as can '50 plus' other elements.

Advantages and Disadvantages

The advantage of this technique is the use of very large samples at relatively low cost. This method can be used quite successfully if there is a gold nugget effect. A large, 1 kg to 5 kg sample can be leached, and, as long as the residue is also analyzed (to see what is not extracted), this method will provide good quality data. The BLEG method has been used extensively in Australia where it was developed, and, to a much lesser extent, in Africa and Nevada, for general exploration. Evaluation of drill core or rock samples by the cyanidation method seems to be little used until the metallurgical testing stage has been reached.

Some disadvantages may be encountered. Depending on the sample material, there may be excess cyanide consumption (sulfides) or loss of gold by adsorption onto carbonaceous material present (preg robbing). Free cyanide should be monitored but this is rarely done for exploration samples. For low level gold results where anomalies of only a few ppb are significant, it is important to use an organic solvent to avoid Fe interference and the graphite furnace - AA finish or the ICP/MS finish (organic solvent not necessary). It is crucial to know what the method blanks are (as-run on your samples) to determine the true validity of the data. BLEG results generally will not be total gold (silica encapsulated or very large gold particles may not completely dissolve). Reuse of the bottle can easily lead to contamination. Cyanide methodologies are not standard for laboratories which run cyanide leach, so results may vary widely from laboratory to laboratory, with the potential to create correlation problems between different sample batches or between reruns of the same samples.

Conclusions

Fire assay remains the stalwart among analytical methods for gold, but, even with its multiple analytical finishes, is not fool-proof for recovering 100% of the gold present. As with all analytical techniques, the skills of the assayer or analyst are very important. As a consumer of assays, it is

advisable not to blindly trust the assay results without consideration of quality control data. Laboratories can and do make mistakes. Some of these can be easily discovered if you *Do Your Own Due Diligence*. Most importantly, there are a number of 'high tech' analytical methods available which can be very cost-effective in providing better quality data than fire assay at very low Au levels.

Due diligence should begin in the field by collecting field duplicates for analysis and should incorporate duplicate samples at various stages of the all important sample reduction scheme. Careful sample preparation to prepare a small sub-sample for analysis is critical. Portions of some rejects and pulps prepared by the laboratory should be checked by a sieve analysis at another laboratory to see if the fineness of the crushing and grinding are meeting the consumer's specifications or the specifications claimed by the laboratories; unfortunately, specifications commonly are not met. How large a pulp is being prepared? Is it representative? Submit a well mineralized sample (native gold-bearing) followed by a blank (barren rock). What is the carry-over of gold into the blanks? Insist on "silica blank cleaning" between samples being pulverized to minimize contamination. Do not let the laboratory compromise on this stage of preparation.

Random sample submittals and data monitoring will also assist in early localization of both sample preparation and analytical bias. Submit the occasional certified reference materials to the laboratory as blind samples. This will provide some information on accuracy. Ask for referrals from trustworthy people active in the areas. Ask whether the laboratory is certified and if it is enrolled in a proficiency testing program for gold analysis (e.g., CANMET in Canada) and if so, request results of all their proficiency tests.

All these measures should ensure that gold analysis values from successive campaigns and from different laboratories meet acceptable levels of accuracy and precision, and can be correlated together, particularly when they occur in neighboring holes. This is essential just as much for prospect evaluation as for project feasibility.

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