

Issues in the analyze of low content gold mining samples by fire assay technique

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The classic technique analyze of samples with low gold content – below 0.1 g/t (=100 ppb = parts per billion), either ore or gold sediments, involves the preparation of sample by fire assay extraction, followed by the chemical attack with aqua regia (hydrochloric and nitric acid) and measuring the gold content by atomic absorption spectrometry or inductively coupled mass spectrometry.

The issues raised by this analysis are well known for the world laboratories, commercial or research ones. The author's knowledge regarding this method of determining the gold content, accumulated in such laboratory from Romania (with more than 40 years of experience, even if not longer available from 2014) confirms the obtaining of reliable results required a lot of attention, amount of work and the involving of an experienced fire assayer specialist.

The analytical conclusion for a research laboratory is that most reliable and statistically valid results are till reached for samples with more than 100 ppb gold content; the degree of confidence below this value is lower than 90%. Usually, for samples below 50 ppb, it does not exceed 50-70 %, unless without very strictly control of each stage, that involve additional percentage of hours allocated for successive extracting tests and knowing more precisely the other compounds that appear in the sample (Cu, Sb, As, sulfur / sulphides, Te, organic matter, etc.) or impurities.

The most important operation is the preparation, namely:

- grinding and splitting of sample (which can cause uneven distribution of gold flakes in the double samples for analyzed);
- pyro-metallurgical recovery of gold = fire assay stage, involving the more precise temperature control in furnace during all stages (fusion and cupellation) and adjusting of the fire assay flux components to produce a successful fusion depending of the sample matrix and content;
- reducing the sample weight to decrease the amount of impurities that can be concentrated in the lead button during oxidation stage.

The better metal recovery and the decreasing of the amount of errors for low gold content samples are controlled in this case by:

- the management of the quantity of one or more components of the flux, depending on the chemical composition of the sample (sometimes just by observing the behavior and the visual characteristics of lead Au + Ag button/bead and the resulted slag);
- addition of gold-free silver, which will be removed by chemical reduction with aqua regia after the fire assay stage.

Regarding the instrumental analyze stage of the samples with less than 100 ppb gold content, there were obtained similar values by both techniques: atomic absorption and inductively coupled mass spectrometry, taking into account each of them has different detection limit.

It is mandatory the quality control with a certified reference material with known content, both in the fire assay stage and the reading instrumental stage.

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