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Methods for the chemical analysis of high-purity gold - Part 2: Determination of impurity elements by inductively coupled plasma mass spectrometry with standard addition correction and internal standardisation

高纯金化学分析方法 第2部分：ICP-MS-标准加入校正-内标法 测定杂质元素的含量

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Table of Contents

1 Scope

This part of GB/T 25934 specifies the determination of impurity elements in high purity gold by inductively coupled plasma mass spectrometry, using standard addition correction together with an internal standard.

It applies to the determination of impurity elements in gold of 99.999 per cent purity, over the elements and the concentration ranges given in Table 1.

3 Principle

The test portion is dissolved in aqua regia, the solution is made up, an internal standard is added, and the impurities are determined by ICP-MS. The calibration is by standard addition: known quantities of the analytes are added to portions of the sample solution itself, and the result is read from the intercept.

The gold is not separated out. It stays in the solution at several grams per litre throughout, and the whole design of the method is an accommodation to that fact.

4 Two corrections doing two different jobs

The standard addition corrects for the matrix effect on the analytical signal: because the additions are made into the sample solution, the calibration and the sample experience the same suppression, and the slope obtained is the one that applies.

The internal standard corrects for what changes during the run: nebuliser drift, small variations in the sample uptake, plasma fluctuation. An element not present in the sample is

added at a constant level and every analyte signal is expressed as a ratio to it.

The two are not alternatives and neither covers the other. Standard addition alone is defeated by drift between the aliquots; an internal standard alone does not fix a calibration slope measured in the wrong matrix. This method uses both because at five nines the residual error from either would be a large fraction of what is being measured.

Measuring what is not there

Gold at 99.999 per cent purity contains ten parts per million of everything else combined. Certifying it means determining a dozen elements at concentrations of parts per million and below, in a solution that is almost entirely gold.

Removing the matrix would be the classical answer, and it is the wrong one here. Separating the gold - by solvent extraction, by precipitation, by ion exchange - risks carrying analytes away with it or contributing them from the reagents, and at these levels a separation introduces more uncertainty than it removes.

So the gold stays in, and the price is severe signal suppression that also drifts as the run proceeds. Standard addition and internal standardisation together are what make that workable, and it is why this method is a distinct part of the series rather than an application of a general ICP-MS procedure.

The matrix stays in

Part 1 removes the gold before measuring. This part keeps it and corrects for it.

Harder analytically, but every separation risks losing an analyte - and mass spectrometry is sensitive enough to work in the matrix directly.

Which is what makes 99.999 measurable

The detection limits sit one to two orders of magnitude below what the emission method reaches.

But the suppression is severe and moving

Percent-level gold suppresses the ion signal of everything else, and not by a constant factor: it depends on the element and the dissolved solids.

And it drifts during a run as gold deposits on the sampler and skimmer cones - so a calibration curve made in clean acid is simply wrong.

Two corrections, two errors

Standard addition puts the calibration in the same matrix as the sample, so the suppression

applies equally to both.

The internal standard, added at a known concentration and measured alongside, takes out the drift and the uptake variation within and between runs.

Why five nines is a commercial number

Gold at 99.99 per cent is jewellery and bullion. Gold at 99.999 is a different product with a different market: bonding wire for semiconductors, sputtering targets, electrical contacts in instruments, and the metal used where a trace of another element changes an electrical property.

The price difference between the two is real and it rests entirely on an analytical certificate. Nobody can see the difference, no physical test distinguishes them, and the only evidence that a bar is five nines rather than four is a determination of the impurities it does not contain.

That is what a method like this one is for. It is not a process control measurement; it is the document that supports a price, which is why it specifies two independent corrections and why the series it belongs to is built around certifying purity rather than around monitoring a refinery.

Table 1 is the standard

The scope says the method applies to the elements and the ranges given in Table 1, and that table is where the real content of this part sits. It names each impurity determinable by this route and the concentration band over which the determination has been validated.

Outside those bands the method is not simply less accurate; it is unsupported. Below the lower bound the analyte is at or under the blank the reagents contribute, and above the upper one the standard addition is no longer working on a linear part of the response.

For a certificate of analysis that matters more than it would elsewhere. A laboratory reporting an element outside the validated range has reported a number this standard does not stand behind, and a buyer relying on the certificate has no way of telling from the figure alone.