
**Jewellery and precious metals —
Determination of very high purity gold
— Difference method using ICP-MS**

*Joannerie, bijouterie et métaux précieux — Dosage de l'or de très
haute pureté — Méthode par différence utilisant la spectrométrie de
masse à plasma à couplage inductif (ICP-MS)*

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 174, *Jewellery and precious metals*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Jewellery and precious metals — Determination of very high purity gold — Difference method using ICP-MS

1 Scope

This document specifies an analytical procedure for the determination of very high purity gold with a nominal content of and above 999,99 ‰ (parts per thousand, by mass), using an inductively coupled plasma mass spectrometer (ICP-MS).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11596, *Jewellery and precious metals — Sampling of precious metals and precious metal alloys*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <https://www.electropedia.org/>

3.1

very high purity gold

gold with a content of and above 999,99 ‰ (parts per thousand, by mass)

4 Principle

The sample is weighed and dissolved in diluted aqua regia to prepare a 2 g/l solution. A blank calibration curve is used to determine the impurities coming from the preparation procedures. The impurities in the sample are determined by ICP-MS using internal standards (see [Table B.1](#) for mass number and internal standards), and the gold content is obtained by subtraction of the total content of impurities in the sample from 1 000 ‰.

5 Reagents

During the analysis and for the preparation of all solutions, unless otherwise stated, use only reagents of recognized analytical grade suitable for ICP-MS and ultrapure water with a resistivity around 18,2 MΩ·cm.

5.1 Hydrochloric acid (HCl), with a mass fraction of approximately 30 % to 37 % of HCl.

5.2 Nitric acid (HNO₃), with a mass fraction of approximately 65 % to 70 % of HNO₃.

5.3 Sulfuric acid (H₂SO₄), with a mass fraction of approximately 95 % to 98 % of H₂SO₄.

5.4 Hydrofluoric acid (HF), with a mass fraction of approximately 40 % of HF.

5.5 Diluted hydrochloric acid, consisting of a mix of one volume of hydrochloric acid (5.1) and one volume of water.

5.6 Diluted nitric acid, consisting of a mix of one volume of nitric acid (5.2) and one volume of water.

5.7 Diluted hydrochloric acid 1:9, consisting of a mix of one volume of hydrochloric acid (5.1) and nine volumes of water.

5.8 Aqua regia, consisting of a mix of three volumes of hydrochloric acid (5.1) and one volume of nitric acid (5.2), prepared freshly just before use.

5.9 Diluted aqua regia, consisting of a mix of one volume of aqua regia (5.8) and one volume of water.

5.10 Monoelemental standard stock solutions (1 mg/ml).

Monoelemental standard stock solutions can be either sourced externally or prepared in the laboratory.

NOTE Details on the preparation of monoelemental standard stock solutions can be found in [Annex A](#).

5.11 Gold standard stock solution (20 g/l).

Weigh $(10,0 \pm 0,1)$ g of gold (of minimum purity 999,99 ‰) to the nearest 0,01 g. Transfer into a 250 ml polytetrafluoroethylene beaker and add 50 ml of diluted aqua regia (5.9). Heat at an appropriate temperature until complete dissolution of the gold. Add 100 ml of aqua regia (5.8), transfer the solution into a 500 ml volumetric flask, make-up with water to 500 ml and mix thoroughly. Transfer the solution into a clean plastic bottle.

5.12 Stock solutions and standard solutions.

5.12.1 Concentrated stock solution (10 µg/ml).

Ag, Al, As, Bi, Cd, Cr, Cu, Fe, Ir, Mg, Mn, Na, Ni, Pb, Pd, Pt, Rh, Sb, Se, Sn, Te, Ti, Zn (10 µg/ml each): Pipette 1 ml of each monoelemental standard stock solution (5.10) into a 100 ml volumetric flask, add 20 ml of diluted aqua regia (5.9), make up with water to 100 ml and mix thoroughly.

5.12.2 Stock solution (0,1 µg/ml).

Ag, Al, As, Bi, Cd, Cr, Cu, Fe, Ir, Mg, Mn, Na, Ni, Pb, Pd, Pt, Rh, Sb, Se, Sn, Te, Ti, Zn (0,1 µg/ml each): Pipette 1 ml of Concentrated stock solution (5.12.1) into a 100 ml volumetric flask, add 20 ml of diluted aqua regia (5.9), make up with water to 100 ml and mix thoroughly.

If other elements are present in the sample, they shall be added. Elements which shall not be analysed can be omitted.

5.12.3 Concentrated internal standard solution (10 µg/ml).

Sc, Cs, Re (10 µg/ml each): Pipette 1 ml of each monoelemental standard stock solution of the standard stock solutions (5.10) into a 100 ml volumetric flask, add 20 ml of diluted aqua regia (5.9), make up with water to 100 ml and mix thoroughly.

5.12.4 Internal standard solution (0,1 µg/ml).

Sc, Cs, Re (0,1 µg/ml each): Pipette 1 ml of concentrated internal standard solution (5.12.3) into a 100 ml volumetric flask, add 20 ml of diluted aqua regia (5.9), make up with water to 100 ml and mix thoroughly.

6 Apparatus

6.1 Customary laboratory apparatus

6.2 ICP-MS (inductively coupled plasma mass spectrometer), suitable for the quantification of trace elements in a solution.

6.3 Analytical balance, with a reading accuracy of 0,1 mg.

7 Sampling

The sampling procedure shall be performed in accordance with ISO 11596.

8 Procedure

WARNING — Suitable health and safety procedures should be followed.

8.1 General

Unless mentioned otherwise, all vessels and reagent bottles shall be made of fluoropolymer (e.g. PTFE, PFA, FEP). Special care should be taken to keep all vessels and reagent bottles as clean as possible, as any contamination will impact the results.

8.2 Sample preparation

The sample should be etched, washed and dried before use to remove any surface contamination, following this procedure.

Place between 0,5 g and 2 g of the sample, cut in small pieces, into a beaker, add 20 ml of an ethanol/water solution (1:1) and heat to boil for 5 min. Discard the solution and rinse the sample 3 times with water. Add 20 ml of diluted hydrochloric acid (5.5) and heat to boil for 5 min. Discard the solution and rinse the sample 3 times with water. Dry the sample at around 105 °C in an oven.

8.3 Sample solution

For each sample to be analysed, two sample solutions shall be prepared as follow and analysed.

Weigh (100 ± 1) mg of the sample portion to the nearest 0,1 mg, transfer into a 50 ml fluoropolymer beaker, and add 2,5 ml of diluted aqua regia (5.9). Heat at an appropriate temperature until complete dissolution of the sample. Allow to cool, transfer into a 50 ml volumetric flask, add 2,5 ml of the internal standard solution (5.12.4), make up with water to 50 ml, and mix thoroughly.

8.4 Blank solution

A blank solution is prepared in parallel by following procedure 8.3 in the absence of the sample.

8.5 Calibration solutions

8.5.1 Blank calibration solutions

Add 0 ml, 0,50 ml, 2,50 ml, 5,00 ml and 10,00 ml of stock solution (5.12.2) to five different 50 ml volumetric flasks, then add in each flask 2,50 ml of diluted aqua regia (5.9) and 2,50 ml of internal standard solution (5.12.4). Make up each flask to 50 ml with water and mix thoroughly. Those solutions are unstable over time and should be prepared just before use, or their stability validated.

8.5.2 Sample calibration solutions

Add 0 ml, 0,50 ml, 2,50 ml, 5,00 ml and 10,00 ml of stock solution (5.12.2) to five different 50 ml volumetric flasks, then add in each flask 5,00 ml of gold standard stock solution (5.11) and 2,50 ml of internal standard solution (5.12.4). Make up each flask to 50 ml with water and mix thoroughly. Those solutions are unstable over time and should be prepared just before use, or their stability validated.

The volumes of stock solutions (5.12.2) used in 8.5.1 and 8.5.2 shall be lowered to match the concentration of impurities in the sample.

8.6 Measurement

Set up the instrument in accordance with the manufacturer's instructions and choose appropriate background correction positions. A clean torch, spray chamber, and sample uptake tubes shall be used and the plasma shall be stabilized before use, following the recommendations of the instrument's manufacturer.

Spray the calibration solutions (see 8.5.1 and 8.5.2) in accordance with the defined instrument calibration procedure "standard addition" and then run the analytical procedure respectively for the blank solution (see 8.4) and sample solution (see 8.3).

Each solution shall have a stabilization time of at least 30 s, followed by at least three integrations each for the determination of the net intensities (background-corrected).

The rinsing time between each measurement shall be sufficient to allow the signal of each impurity to come back to the baseline.

9 Calculation and expression of the results

9.1 Calibration curves

Set the concentration in the calibration solutions (see 8.5.1 and 8.5.2). The blank calibration curve and the sample calibration curve are obtained by spraying separately the calibration solutions (see 8.5.1 and 8.5.2). The blank calibration curve is used to determine $c_{b,i}$, the concentration of each element in the blank solution (see 8.4). The sample calibration curve is used to determine $c_{s,i}$, the concentration of each element in the sample solution (see 8.3).

Calculate the calibration curve using the intensity ratio between each element and its corresponding internal standard (see Annex B).

Collision cell technology or correction of spectral interferences with an appropriate mathematical model shall be used.

EXAMPLE The spectral intensity of As75 and Se82 can be corrected by the following formulae:

As75: $-3,128\ 819 \times \text{Se77} + 2,734\ 582 \times \text{Se82} - 2,756\ 001 \times \text{Kr83}$

Se82: $-1,007\ 833 \times \text{Kr83}$

9.2 Calculation

The mass concentration is obtained by the intensity ratio of test element and its corresponding internal standard. The mass fraction (W_i) of each element is calculated and expressed as mg/kg of gold using [Formula \(1\)](#):

$$W_i = \frac{c_{s,i} \times V_s - c_{b,i} \times V_b}{m} \times 10^3 \quad (1)$$

where

$c_{s,i}$ is the concentration of the element i in the sample solution, in $\mu\text{g/ml}$;

V_s is the volume of the sample solution, in ml;

$c_{b,i}$ is the concentration of the element i in the blank solution, in $\mu\text{g/ml}$;

V_b is the volume of the blank solution, in ml;

m is the mass of the metallic sample, in mg.

The precious metal fineness (W_{sp}), expressed in parts per thousand (‰), is thus calculated using [Formula \(2\)](#):

$$W_{\text{sp}} = 1\,000 - \left(\frac{\sum W_i}{1\,000} \right) \quad (2)$$

where $\sum W_i$ is the sum of the mass portion of each element found above its detection limit, in mg/kg of gold.

9.3 Repeatability

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Duplicate determinations shall give results differing by less than 25 % for the sum of impurities. If the difference is greater than this, the assay shall be repeated.

10 Test report

The test report shall include at least the following information:

- a) identification of the sample including source, date of receipt, and form of sample;
- b) sampling procedure;
- c) method used by reference to this document, i.e. ISO 5724:2023;
- d) gold content of the sample, in parts per thousand (‰) by mass, as single values and mean values, with the result reported with six significant figures;
- e) if relevant, any deviations from the method specified in this document;
- f) any unusual features observed during the determination;
- g) date of test;
- h) identification of the laboratory carrying out the test;
- i) signature of the laboratory manager and operator.

Annex A (informative)

Procedures for preparation of monoelemental standard stock solutions

A.1 Aluminium, standard stock solution (1 mg/ml): Weigh 100,0 mg of aluminium metal (of minimum purity 99,99 %) into a 100 ml beaker, add 20 ml of diluted hydrochloric acid (5.5) and heat gently to dissolve the aluminium. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with diluted hydrochloric acid 1:9 solution (5.7) and mix thoroughly.

A.2 Antimony, standard stock solution (1 mg/ml): Weigh 100,0 mg of antimony metal (of minimum purity 99,99 %) into a 100 ml beaker and add 20 ml of diluted aqua regia (5.9). Heat gently to dissolve the antimony and volatilize the nitrogen oxides. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.3 Arsenic, standard stock solution (1 mg/ml): Weigh 132,0 mg of arsenic trioxide (As_2O_3 , Primary reagent, previously dried at 100 °C~105 °C for 1 h), transfer into a 100 ml beaker and add 20 ml of aqua regia (5.8). Heat gently to dissolve the arsenic trioxide and volatilize the nitrogen oxides. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.4 Bismuth, standard stock solution (1 mg/ml): Weigh 100,0 mg of bismuth metal (of minimum purity 99,99 %) into a 100 ml beaker and add 20 ml of diluted nitric acid (5.6). Heat gently to dissolve the bismuth and volatilize the nitrogen oxides. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.5 Cadmium, standard stock solution (1 mg/ml): Weigh 100,0 mg of cadmium metal (of minimum purity 99,99 %) into a 100 ml beaker and add 20 ml of diluted nitric acid (5.6). Heat gently to dissolve the cadmium and volatilize the nitrogen oxides. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.6 Cesium, standard stock solution (1 mg/ml): Weigh 136,1 mg of cesium sulfate (CsSO_4 , guaranteed reagent, previous dried at 100 °C~105 °C for 1 h) into a 100 ml beaker, add 20 ml of water and heat gently to dissolve the cesium sulfate. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.7 Chromium, standard stock solution (1 mg/ml): Weigh 282,9 mg of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$, Primary reagent, previously dried at 100 °C~105 °C for 1 h), transfer into a 100 ml beaker and add 20 ml of diluted hydrochloric acid (5.5). Heat gently until complete dissolution of the potassium dichromate. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.8 Copper, standard stock solution (1 mg/ml): Weigh 100,0 mg of copper metal (of minimum purity 99,99 %) into a 100 ml beaker and add 20 ml of diluted nitric acid (5.6). Heat gently to dissolve the copper and volatilize the nitrogen oxides. Cool to room temperature, transfer the solution into a 100 ml volumetric flask, then dilute to the mark with water and mix thoroughly.

A.9 Iridium, standard stock solution (1 mg/ml): Weigh 229,4 mg of ammonium chloroiridate [$(\text{NH}_4)_2\text{IrCl}_6$, spectrum pure] into a 100 ml beaker, add 20 ml of diluted hydrochloric acid 1:9 (5.7) and heat gently to dissolve the ammonium chloroiridate. Cool to room temperature, transfer the solution