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Jewellery and precious metals — Determination of silver — ICP-OES method using an internal standard element

Joaillerie, bijouterie et métaux précieux — Dosage de l'argent — Méthode par ICP-OES utilisant un étalon interne

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Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Principle ... **2**

5 Reagents ... **2**

6 Apparatus ... **2**

7 Sampling ... **3**

8 Procedure ... **3**
8.1 Internal standard solutions ... 3
8.2 Calibration solutions ... 3
8.3 Sample solutions ... 4
8.4 Measurement ... 4

9 Calculation and expression of results ... **4**
9.1 Calculation ... 4
9.2 Repeatability ... 6

10 Test report ... **6**

Annex A (informative) **Technical background for ICP -OES (inductively coupled argon plasma optical emission spectrometer)** ... **7**

Annex B (informative) **Interlaboratory testing** ... **8**

Bibliography ... **10**

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Foreword

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

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Jewellery and precious metals — Determination of silver — ICP-OES method using an internal standard element

1 Scope

This document specifies an analytical method for the determination of silver on a material considered homogeneous. The silver content of the samples lies preferably between 50 ‰ (parts per thousand) and 990 ‰ (parts per thousand). Fineness above 990,0 ‰ can be determined using a spectroscopy method by difference (see ISO 15096).

NOTE Lower contents of silver can be analysed according to this method.

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
calibration solution
solution used to calibrate the instrument, prepared from stock solution or solution prepared with certified reference material by adding acid, buffer, reference elements and salt as needed

3.2
bracketing
analytical method consisting of bracketing the measured emission line intensity of the sample between two measurements made on calibration solutions of neighbouring concentrations within the optimum working range

Note 1 to entry: Running of standards and samples in the following sequence: low standard – sample – high standard – sample – low standard – sample – high standard ...

[SOURCE: ISO 19376-1:2025, 3.3.7, modified — Note 1 to entry was added.]

3.3
laboratory sample
sample intended for laboratory inspection or testing

3.4
test sample
portion of material, resulting from the laboratory sample by means of an appropriate method of sample pretreatment, and having the size (volume/mass) necessary for the desired testing or analysis

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3.5
test sample solution
solution prepared after extraction or digestion of the test sample according to appropriate specification

3.6
internal standard
compound added to a sample in a fixed amount that has similar properties to the target analyte used to correct for instrument drift and matrix interference

[SOURCE: ISO 19376-1:2025, 3.3.8]

4 Principle

At least two accurately weighed samples are first dissolved in nitric acid and then carefully precipitated with concentrated hydrochloric acid and then dissolved again. These sample solutions are mixed with an exact amount of internal standard and made up to the measurement volume.

Using ICP-OES, the silver content of the sample solution is measured by comparison of the ratio intensities of the spectral emission of silver and appropriate internal standard line(s) with the ratio for solutions containing known masses of silver and internal standard using the bracketing method.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. All reagents shall be silver free.

5.1 Hydrochloric acid (HCl), 30 % to 37 % HCl (mass fraction).

5.2 Nitric acid (HNO_3), 65 % to 70 % HNO_3 (mass fraction).

5.3 Nitric acid (HNO_3), 33 % HNO_3 (mass fraction) - mixed one volume of distilled water and one volume nitric acid 5.2.

5.4 Silver (Ag) of 999,9 ‰ minimum purity and with an oxygen content <100 mg/kg.

If a lower silver content (e.g. 999,5 ‰) is used, appropriate correction factors shall be applied.

NOTE Silver sheets contain usually less than 100 mg/kg of oxygen. Confirmation of the analytical value or test report is recommended.

5.5 Internal standard element, e.g. yttrium, scandium, indium, germanium or lutetium, single element standard.

The adequate specification of single element standard stock solutions shall state the acid used. Single-element standard stock solutions can be made from high purity metals.

6 Apparatus

All equipment shall be carefully cleaned.

6.1 Customary laboratory apparatus.

6.2 Inductively coupled argon plasma optical emission spectrometer (ICP-OES), capable of simultaneously measuring the silver and the internal standard emission lines with a minimum optical resolution of 0,02 nm.

NOTE Annex A gives some technical background on ICP-OES.

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