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Jewellery and precious metals — Determination of high purity silver — Difference method using ICP-OES

Joaillerie et métaux précieux — Dosage de l'argent à haute pureté — Méthode par différence utilisant l'ICP-OES

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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 documents 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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 15096:2014), which has been technically revised. The main changes compared to the previous edition are as follows:

a) change of title of standard;

b) change of scope for measuring also silver with a nominal content above 999 ‰ (parts per thousands);

c) revision of this document in order to comply in structure with ISO 15093;

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 high purity silver — Difference method using ICP-OES

1 Scope

This document specifies the analytical procedure for the determination of silver with a nominal content of and above 999 ‰ (parts per thousand).

This document specifies a method intended to be used as the recommended method for the determination of silver of fineness of and above 999 ‰. For the determination of fineness of and above 999,9 ‰, modifications described in Annex B apply.

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 — Sampling of precious metal alloys for and in jewellery and associated products*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The sample is weighed and dissolved in nitric acid to prepare a 10 g/l solution (higher concentration is used for fineness of and above 999,9 ‰). The suspension, which can be present in that solution, is isolated by centrifugation or microfiltration and dissolved in aqua regia. Both nitric and aqua regia solutions are analysed separately by ICP-OES and the total content of each impurity (see Table A.1 for wavelengths) in the sample is obtained by adding together the results of the two analyses. The silver content is obtained by subtraction of the total content of impurities in the sample from 1 000 ‰. For the determination of fineness of and above 999,9 ‰, modifications described in Annex B shall be applied.

5 Reagents

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

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 Aqua regia (should be prepared just before use). Mix three volumes of hydrochloric acid (5.1) and one volume of nitric acid (5.2).

5.4 Stock solution A (shall not contain any chloride), Al, Cd, Cr, Cu, Fe, Mg, Mn (100 mg/l each) in 3 % to 7 % HNO_3 (5.2) (mass fraction).

5.5 Stock solution B (shall not contain any chloride), Ni, Sb, Se, Te, Ti (100 mg/l each) in 3 % to 7 % HNO_3 (5.2) (mass fraction).

5.6 Stock solution C (shall not contain any chloride), As, Bi, Co, Pb, Pt, Si, Sn, Zn (100 mg/l each) in 3 % to 7 % HNO_3 (5.2) (mass fraction).

5.7 Stock solution D (shall not contain any chloride), Ga, Ge, Hg, In, Pd, Tl (100 mg/l each) in 3 % to 7 % HNO_3 (5.2) (mass fraction).

NOTE 1 Elements which do not need to be analysed can be omitted. Other elements can be added, provided they are stable and do not generate significant interferences.

NOTE 2 Stock solutions “A” to "D” are typically prepared by mixing 10 % (of the total volume) of each 1 000 mg/l monoelemental solution with 5 % of nitric acid (5.2) (volume fraction) and making up with water. They can be kept for up to 12 months under proper storage conditions.

5.8 Stock solution E (may contain both chlorides and nitrates), Al, Au, Cr, Fe, Mg, Ni, Pt, Sn (100 mg/l each) in 3 % to 7 % aqua regia (5.3) (volume fraction).

NOTE Stock solution “E” is typically prepared by mixing 10 % (of the total volume) of each 1 000 mg/l monoelemental solution with 5 % of aqua regia (5.3) (volume fraction) and making up with water. It can be kept for up to 12 months under proper storage conditions.

5.9 Reference materials: silver of 999,9 ‰ minimum purity. The content of each impurity shall be specified and taken into account in the calibration.

6 Apparatus

6.1 Customary laboratory apparatus.

6.2 ICP-OES, with a minimum optical resolution of 0,02 nm, a detection limit of 0,02 mg/l or better, and capability of background correction.

NOTE For preferably used wavelengths, see Annex A.

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

6.4 Centrifuge, suitable for 10 ml to 50 ml tubes and rotating at least at 3 000 r/min (revolution per minute), or

6.5 Microfiltration system, with cellulose membrane filter (approximately 0,45 µm pore size) and a vacuum system.

7 Sampling

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