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Jewellery and precious metals — Determination of gold — Cupellation method (fire assay)

Joaillerie, bijouterie et métaux précieux — Dosage de l'or — Méthode de coupellation (essai au feu)

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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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/SS M21, *Precious metals - Applications in jewellery and associated products,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 11426:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

— extension of the scope to cover determination of gold in multiple types of alloys, not only in jewellery ones;

— purity of proof samples was re-defined in Clause 5;

— specific procedures are described in Clause 8 for samples with large amount of base metals, containing platinum or palladium, or with a silver/gold ratio higher than 3;

— calculation was adapted to take into account the addition of pure gold and the fineness of the gold used in the proof sample;

— repeatability requirements were changed;

— the use of scorification was removed.

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 gold — Cupellation method (fire assay)

1 Scope

This document specifies a cupellation method (fire assay) for the determination of gold on a material considered homogeneous. The gold content of the sample lies preferably between 100 and 999,5 parts per thousand (‰) by weight. Fineness above 999,5 ‰ can be determined using a spectroscopy method by difference (e.g. ISO 15093).

The procedure is applicable to most types of gold samples. Some modifications are indicated for specific cases (presence of large amount of base metals, platinum or palladium, silver). It is not compatible with the presence above trace levels of iridium, rhodium and ruthenium (more than 0,25 ‰ for the sum of all three elements).

This method is also intended to be used as the recommended method for the determination of fineness in jewellery alloys covered by ISO 9202.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

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

3.1
cornet
alloy of gold and precious metals shaped in a roll, prior the parting process

3.2
gold cornet
gold shaped in a roll, after the *parting* (3.3) process

3.3
parting
separation of silver and other metals from gold by digestion of those metals with nitric acid, in a chloride-free environment

3.4
proof sample
synthetic reference sample whose composition is as similar as possible to the sample; cupellation of the proof sample is performed together with the sample, and its result is used to correct the final assay

3.5
inquartation
addition of silver to gold alloys in a specific ratio in order to enable the *parting* (3.3) of gold from silver by means of nitric acid

4 Principle

The gold alloys are inquarted with silver, compounded with lead and cupelled in a cupellation furnace until a precious metal button is obtained. After flattening and rolling, the silver is extracted (parted) in nitric acid and the gold weighed. Possible systematic errors in the procedure are eliminated by assaying proof samples in parallel.

Annex A gives information on metals, that can influence the result of gold testing.

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 Nitric acid (HNO_3), approximately 33 % (mass fraction), with low content of halides (approximatively <2 mg/l; the presence of halides can be detected with silver nitrate test).

5.2 Nitric acid (HNO_3), approximately 49 % (mass fraction), with low content of halides (approximatively <2 mg/l; the presence of halides can be detected with silver nitrate test).

5.3 Lead, assay grade, free of gold and platinum group metals, containing less than 0,1 ‰ of bismuth, as foil, beads or tablets.

5.4 Pure silver, for inquartation, minimum purity 999,9 ‰, with low content of gold and platinum group metals (≤0,01 ‰ per element).

5.5 Pure gold, for proof samples, minimum purity 999,90 ‰, with a fineness determined to 5 significant digits; a purity of 999,99 ‰ is preferred.

5.6 Pure platinum, for proof samples, minimum purity 999,5 ‰, with low content of gold (≤0,01 ‰).

5.7 Pure palladium, for proof samples, minimum purity 999,5 ‰, with low content of gold (≤0,01 ‰).

5.8 Base metals, for proof samples, in a form of an appropriate pre-alloy (free of precious metals).

5.9 Copper (foil, wire, beads or tablets), minimum purity 999 ‰, free of gold and platinum group metals.

NOTE 1 The term “free of” corresponds to a concentration below ≤0,002 ‰ of each element.

NOTE 2 For determination of fineness of metals according to 5.4 to 5.9, the oxygen content is not taken into account.

6 Apparatus

6.1 Cupellation furnace, capable of reaching a relatively homogeneous temperature of 1 050 °C to 1 150 °C, and in which an oxidizing atmosphere can be maintained. A standard muffle furnace is not satisfactory for this purpose.

6.2 Magnesia cupels (or similar), in form of single or block cupels, capable of absorbing the lead and base metals during the cupellation.

6.3 Parting flasks or **nitric acid resistant basket with thimbles**.

6.4 Annealing crucibles, made of refractory or other non-contaminating materials.