

International Standard

ISO 11427

Jewellery and precious metals — Determination of silver — Potentiometry using potassium bromide Third edition

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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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 11427:2014) which has been technically revised.

The main changes are as follows:

- deletion of "in silver alloys" in the title;
- change of the scope by extending it to alloys containing from 100 to 999 parts per thousand;
- addition of oxygen content for reference pure silver in <u>5.4</u>.

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 silver — Potentiometry using potassium bromide

1 Scope

This document specifies a volumetric method for the determination of silver on a material considered homogeneous. The silver content of the sample lies preferably between (100 and 999,0) parts per thousand (‰) by mass. Fineness above 999,0 ‰ can be determined using a spectroscopy method by difference (e.g. ISO 15096).

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

2 Normative references

There are no normative references in this document.

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 https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

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4 Principle rds.iteh.ai/catalog/standards/iso/c4a1f25e-e257-44d4-bd8e-3e730901d203/iso-prf-11427

The sample is dissolved in dilute nitric acid. The silver content of the resulting solution is determined by titration with standard potassium bromide solution using a potentiometric indication of the equivalence point. Palladium can interfere with the measurement and is precipitated before commencing titration, other elements do not interfere with this method of determination.

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); 33 % (mass fraction), with sufficiently low content of halides (check with silver nitrate test).
- **5.2 Potassium bromide solution**, c(KBr) = 0.1 mol/l.

Dissolve 11,901 g of potassium bromide (dried at 105 °C) in water and dilute to 1 000 ml.

5.3 Disodium dimethylglyoxime octahydrate solution.

Dissolve 10 g of disodium dimethylglyoxime octahydrate in 1 000 ml of water.

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5.4 Pure silver, minimum purity 999,9 parts per thousand (‰) by mass and with an oxygen content <100 mg/kg. The laboratory shall guarantee by analysis or validation that this requirement is meet.

NOTE Silver sheets contain usually less than 100 mg/kg of oxygen.

6 Apparatus

- 6.1 Customary laboratory apparatus.
- **6.2 Motor-driven plunger or piston-type burette**, linked to a potentiometer or automatic titrator and capable of delivering increments of 0,05 ml at the equivalence point.
- **6.3 Titration apparatus**, with combination silver electrode coated with silver bromide and Hg/Hg_2SO_4 or other suitable reference electrode.
- **6.4 Analytical balance**, with a reading accuracy of 0,01 mg.

7 Sampling

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

8 Procedure

WARNING — Suitable health and safety procedures should be followed.

8.1 Determination of potassium bromide solution factor teh. al)

8.1.1 General

When the composition of the samples is unknown, a preliminary analysis by suitable means shall be used to determine the approximate composition of the material, e.g. XRF (X-ray fluorescence) analysis

8.1.2 Preparation of silver standards

Weigh two samples of silver (5.4); each of 300 mg to 500 mg, accurately to the nearest 0,01 mg, and transfer them into three glass beakers. Add 5 ml of nitric acid (5.1) to each beaker, and warm gently to dissolve the silver. Cover the beakers with watch glasses. Heat until evolution of nitrogen oxides ceases. Allow to cool. Rinse the watch glasses into beakers. Add the minimum volume of water required to satisfy the requirements of the titration apparatus (6.3) in respect of measurement and stirring. The approximate volume depends on the volume of the beaker. Put the beaker in the titration apparatus (6.3).

The mass of the standard silver samples should lie within 20 mg of the mass of silver in the sample portion.

8.1.3 Titration of the standard silver solution

Add, via the plunger-burette $(\underline{6.2})$ under continuous stirring, sufficient potassium bromide solution $(\underline{5.2})$ to precipitate about 95 % of the silver in the solution. Titrate the remaining silver in such a manner that the equivalence point can be interpolated from 0,05 ml additions of the potassium bromide solution.

NOTE This split titration approach can be effected automatically, using an automatic titrator with so-called dynamic volume dosing based on the measured potential difference across the electrodes in the titration apparatus (6.3).