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

Joaillerie, bijouterie et métaux précieux — Dosage de l'argent — Méthode potentiométrique utilisant le bromure de potassium

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Contents

Foreword

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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/TC 410, *Consumer confidence and nomenclature in the diamond industry*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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 5.4.

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/

4 Principle

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**, $c(\text{KBr})$ = 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.

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

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 two 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 (6.2) under continuous stirring, sufficient potassium bromide solution (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).

8.1.4 Calculation of the potassium bromide standard solution factor

The potassium bromide standard solution factor, *F*, is calculated using Formula (1):

$$F = \frac{m_{\mathrm{AgF}}}{V_{\mathrm{AgF}}} \qquad (1)$$

where

F	is the value of the potassium bromide standard solution factor, expressed in milligrams of silver for each millilitre of solution;
m_{AgF}	is the mass of silver, in milligrams;
V_{AgF}	is the volume of potassium bromide solution at equivalence point, in millilitres.

The successive values of factor determinations shall not differ from each other by more than 0,05 % relative value. The mean value, $\overline{F}$, shall be used in subsequent calculations for maximum accuracy. The potassium bromide solution factor shall be determined immediately before analysis of the sample portions.

8.2 Determination

8.2.1 Preparation of the sample solution

Weigh, to the nearest 0,01 mg, two sample portions, each containing between 300 mg and 500 mg of silver, and transfer them to glass beakers. Add 5 ml nitric acid (5.1) and warm gently to dissolve the alloy. Tops of the beakers can be covered with watch glasses. Heat until evolution of nitrogen oxides ceases. Allow to cool. Rinse the watch glasses into beakers. Transfer to the titration apparatus (6.3). Add the minimum volume of water required to satisfy the requirements of the titration apparatus (6.3) in respect of measurement and stirring.

8.2.2 Elimination of palladium

Palladium, if present, shall be precipitated by adding an aqueous solution of disodium dimethylglyoxime octahydrate (5.3). For each 100 mg of palladium, add 50 ml of this solution before commencing titration. A filtration of the precipitate is not needed.

8.2.3 Titration of the sample solution

Proceed exactly as for the standard solution. It can be necessary to carry out a pilot determination to obtain an approximate value of the silver content.

9 Calculation and expression of results

9.1 Calculation

The mass of silver, m_{Ags}, in milligrams in the sample portion is calculated using Formula (2):

$$m_{\mathrm{Ags}} = \overline{F} \cdot V_{\mathrm{Ags}} \qquad (2)$$

where

$\overline{F}$	is the mean value of the potassium bromide standard solution factor, expressed in milligrams of silver for each millilitre of solution;
V_{Ags}	is the volume of potassium bromide at equivalence point, in millilitres.

The silver content of the sample, $W_{\mathrm{Ag},}$ in parts per thousand (‰) by mass is calculated using Formula (3):

$$W_{\mathrm{Ag}} = \frac{m_{\mathrm{Ags}}}{m_{\mathrm{s}}} \cdot 10^3 \qquad (3)$$

where m_{s} is the mass of the sample (8.2.1), in milligrams.

9.2 Repeatability

The results of duplicate determinations shall agree to better than 1 part per thousand (‰) by mass of silver. If the variation is greater than this, the assay shall be repeated.

10 Test report

The test report shall include the following information:

a) identification of the sample including source, date of receipt, form of sample;

b) sampling procedure;

c) the method used by reference to this document (ISO 11427:2024);

d) silver content of the sample, in parts per thousand (‰) by mass, as single values and mean value;

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 laboratory carrying out this analysis;

i) signature of laboratory manager and operator.