



**International
Standard**

ISO 13756

**Jewellery and precious metals —
Determination of silver —
Potentiometry using sodium
chloride or potassium chloride**

*Joaillerie, bijouterie et métaux précieux — Dosage de l'argent —
Méthode potentiométrique utilisant le chlorure de sodium ou le
chlorure de potassium*

**Third edition
2024-04**

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

This third edition cancels and replaces the second edition (ISO 13756:2015), 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 silver from 100 to 999 parts per thousand (‰) by mass;
- addition of oxygen content for reference pure silver in 5.5.

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 sodium chloride or potassium chloride

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 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/>

4 Principle

The sample is dissolved in dilute nitric acid. The silver content of the resulting solution is determined by titration with sodium chloride or potassium chloride standard 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 (HNO₃), 33 % (mass fraction), with sufficiently low content of halides (check with silver nitrate test).

5.2 Sodium chloride solution, $c(\text{NaCl}) = 0,1 \text{ mol/l}$.

Dissolve 5,84 g of sodium chloride (dried at 105 °C) in water and dilute to 1 000 ml.

5.3 Potassium chloride solution, $c(\text{KCl}) = 0,1 \text{ mol/l}$.

Dissolve 7,44 g of potassium chloride (dried at 105 °C) in water and dilute to 1 000 ml.

5.4 Disodium dimethylglyoxime octahydrate solution.

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

5.5 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 met.

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 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 or silver chloride coated silver electrode and Hg/Hg₂SO₄ electrode or other suitable reference electrode.

A silver chloride coating can be obtained by electrolysis, branching a silver electrode as anode in a dilute hydrochloric acid solution $c(\text{HCl}) = 0,1 \text{ mol/l}$. After suitable surface preparation, apply an anodic current density of 1 mA/cm² for approximately one hour, until the silver surface is completely covered with silver chloride.

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 sodium chloride or potassium chloride standard 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, each of 300 mg to 500 mg of silver, to the nearest 0,01 mg (5.5), and transfer them to 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. 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 standard silver solution

Add, via the plunger-burette (6.2) under continuous stirring, sufficient sodium chloride standard solution (5.2) or potassium chloride standard solution (5.3) 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 sodium chloride or potassium chloride standard 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 sodium chloride or potassium chloride standard solution factor

The sodium chloride or potassium chloride standard solution factor, F , is calculated using [Formula \(1\)](#):

$$F = \frac{m_{\text{AgF}}}{V_{\text{AgF}}} \quad (1)$$

where

F is the sodium chloride or potassium chloride standard solution factor, in milligrams of silver for each millilitre of solution;

m_{AgF} is the mass of silver, in milligrams;

V_{AgF} is the volume of sodium chloride or potassium chloride 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, \bar{F} , shall be used in subsequent calculations for maximum accuracy. The sodium chloride or potassium chloride standard 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 of nitric acid (5.1) to each and warm gently to dissolve the alloy. Keep the tops of the beakers 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 water as per 8.1.2.

8.2.2 Elimination of palladium

Palladium, if present, shall be precipitated by adding an aqueous solution of disodium dimethylglyoxime octahydrate (5.4). 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 per the standard solution. It may 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 \(2\)](#):

$$m_{\text{AgS}} = \bar{F} \cdot V_{\text{AgS}} \quad (2)$$

where

\bar{F} is the sodium chloride or potassium chloride standard solution factor, in milligrams of silver for each millilitre of solution;

V_{Ags} is the volume of sodium chloride or potassium chloride at equivalence point, in millilitres.

The silver content of the sample W_{Ags} in parts per thousand (‰) by mass is calculated using [Formula \(3\)](#):

$$W_{\text{Ags}} = \frac{m_{\text{Ags}}}{m_s} \cdot 10^3 \quad (3)$$

where m_s is the mass of the sample (see [8.2.1](#)), in milligrams.

9.2 Repeatability

The results of duplicate determinations shall correspond to better than one 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, i.e. ISO 13756:2024;
- 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.