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Third edition

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

PROOF/ÉPREUVE

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Foreword

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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 first 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 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 standard sodium chloride or potassium chloride 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 Sodium chloride solution, c(NaCl) = 0,1 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(KCl) = 0,1 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 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 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/Hg_2SO_4 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(HCl) = 0,1 mol/l. After suitable surface preparation, apply an anodic current density of 1 mA/cm^2 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.