SLOVENSKI STANDARD
oSIST prEN ISO 11494:2019

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Nakit in plemenite kovine - Določevanje platine v zlitinah platine - Metoda ICP-OES z uporabo notranjega standardnega elementa (ISO/DIS 11494:2018)

Jewellery and precious metals - Determination of platinum in platinum alloys - ICP-OES method using an internal standard element (ISO/DIS 11494:2018)

Schmuck und Edelmetalle - Bestimmung von Platin in Platinschmucklegierungen - ICP-OES-Verfahren unter Verwendung eines internen Standardelements (ISO/DIS 11494:2018)

Joaillerie et métals précieux - Dosage du platine dans les alliages de platine - Méthode par l'ICP-OES, utilisant un étalon interne (ISO/DIS 11494:2018)

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Jewellery and precious metals — Determination of platinum in platinum alloys — ICP-OES method using an internal standard element

Joaillerie et métals précieux — Dosage du platine dans les alliages de platine — Méthode par l'ICP-OES, utilisant un étalon interne

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Contents

Page

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 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: 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 11494:2014), which has been technically revised.

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

a) removed the definition of bracketing in clause 3;

b) removed the recommended lines in clause 4;

c) changed and removed reagents in clause 5 and changed the requirements about yttrium in 5.4;

d) changed the preparation of the preparation of the internal standard solution in 8.1;

e) changed the list of standards to be prepared and precisions about qualification of them by linearity as well as way to choose the low and high standards in 8.2;

f) removed the way of preparation by aliquots for both standard and sample solutions in 8.2 and 8.3;

g) changed the preparation of both standard and sample solutions in 8.2 and 8.3;

h) added precisions about quantity of acids to be used in case of dissolution under pressure in 8.4;

i) added of the definition of bracketing and recommended lines in 8.5;

j) adapted of the formulas in 8.6 after having removed the way of preparation by aliquots;

k) removed the emission line as an information to be mentioned in the test report in clause 10;

l) document editorially revised.

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.

Introduction

The following definitions apply in understanding how to implement an ISO International Standard and other normative ISO deliverables (TS, PAS, IWA):

— “shall” indicates a requirement;

— “should” indicates a recommendation;

— “may” is used to indicate that something is permitted;

— “can” is used to indicate that something is possible, for example, that an organization or individual is able to do something.

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.1 defines a requirement as an “expression in the content of a document conveying criteria to be fulfilled if compliance with the document is to be claimed and from which no deviation is permitted.”

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.2 defines a recommendation as an “expression in the content of a document conveying that among several possibilities one is recommended as particularly suitable, without mentioning or excluding others, or that a certain course of action is preferred but not necessarily required, or that (in the negative form) a certain possibility or course of action is deprecated but not prohibited.”

Jewellery and precious metals — Determination of platinum in platinum alloys — ICP-OES method using an internal standard element

1 Scope

This document describes an analytical procedure for the determination of platinum in platinum alloys with a nominal content up to 990 ‰ (parts per thousand), including alloys according to ISO 9202.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11596, *Jewellery — Sampling of precious metal alloys for and in jewellery and associated products*

ISO/DIS 9202, *Jewellery and precious metals — Fineness of precious metal alloys*

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:

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

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

4 Principle

At least two accurately weighed samples are dissolved in aqua regia and made up to an exactly weighed mass. These sample solutions are mixed with the internal standard and made up to the standard measuring volume.

Using ICP-OES, the platinum content of the sample solution is measured by comparison of the ratio intensities of the spectral emission of platinum and appropriate internal standard (e. g. yttrium) line(s) with the ratios for solutions containing known masses of platinum and internal standard (e. g. yttrium) using the bracketing method.

Minor modifications are required when the alloy contains ruthenium, rhodium, iridium, or tungsten.

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 Hydrochloric acid (HCl), approximately 30 % to 37 % HCl (mass fraction).

5.2 Nitric acid (HNO_3), approximately 65 % to 70 % HNO_3 (mass fraction).

5.3 Platinum (Pt) of 999,9 ‰ minimum purity; if a lower platinum content (*e.g.* 999,5 ‰) is used, appropriate corrections shall be applied.

5.4 Yttrium compound like yttrium chloride ($YCl_3 \cdot 6H_2O$ or Y_2O_6), in analytical grade.

5.5 Copper of 999,9 ‰ minimum purity, and platinum free.

5.6 Orthophosphoric acid (H_3PO_4), 85 % (mass fraction).

6 Equipment

6.1 Customary laboratory apparatus.

6.2 ICP-OES, capable of simultaneously measuring the platinum emission lines and the emission line of the internal standard (e. g. yttrium), with a minimum optical resolution of 0,02 nm.

6.3 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 Internal standard solution

Dissolve approximately 20 mg YCl_3 $6H_2O$ (5.4) in 200 ml water and make up to 1000 ml with water. Alternatively, prepare a solution in order to obtain a concentration of approximately 6 mg/l of Yttrium. Due to the sensitivity of the instrument, the concentration may be changed to achieve optimum performance.

8.2 Calibration solutions

The sequence by bracketing needs the use of only two standards that shall correspond to the nearest points (low standard and high standard as used in 8.5) to the expected platinum sample content. It is recommended to prepare a set of at least three standards and to check their linearity.

Weigh approximately 45 mg, 55 mg, 65 mg, 75 mg, 82,5 mg, 87,5 mg, 92,5 mg, 97,5 mg and 100 mg platinum accurately to 0,01 mg each into a glass beaker. Heat gently the sample in the glass beaker covered with a watch glass in a mixture of 100 ml HCl (5.1) and 30 ml HNO_3 (5.2) until complete dissolution, and continue to heat to expel the nitrogen oxides. Transfer the solution in a 1 000 ml flask. Add 100 g accurately to 0,01 g of the internal standard solution (8.1). Add 100 ml HCl (5.2) and make up to 1000 ml with water. Mix thoroughly.

In the presence of certain other elements (e.g. silver), it can be necessary to increase the HCl concentration to a maximum of 500 ml. The acid concentration of calibration solutions and sample solutions shall be consistent.

8.3 Sample solutions

Weigh 100 mg sample accurately to 0,01 mg into a glass beaker and dissolve and treat the sample as described in 8.2.

