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Jewellery and precious metals — Determination of high purity gold, silver, platinum and palladium — Difference method using SPARK-OES

Joaillerie, bijouterie et métaux précieux — Dosage de l'or, de l'argent, du platine et du palladium à haute pureté — Méthode par différence utilisant la SPARK-OES

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Foreword

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

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Jewellery and precious metals — Determination of high purity gold, silver, platinum and palladium — Difference method using SPARK-OES

1 Scope

This document specifies an analytical procedure for the determination of gold, silver, platinum and palladium with a nominal content of and above 999 ‰ (parts per thousand, by mass), using an optical emission spectrometer with excitation by spark discharge (SPARK-OES).

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 and precious metals — Sampling of precious metals and precious metal alloys*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

3.1
SPARK-OES
optical emission spectrometer with excitation by spark discharge, suitable for the quantification of trace elements in a solid metallic sample

3.2
spot
target area on the sample where the spark strikes the surface and creates a burn mark

3.3
certified reference material
CRM
reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a reference material certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO 17034:2016, 3.2, modified — Notes to entry have been deleted.]

3.4
calibration standard
material with appropriate homogeneity whose exact composition is known and which can be used to calibrate the *SPARK-OES* (3.1)

3.5
quality control sample
sample with appropriate homogeneity containing most or all impurities to be measured, used to detect drift of the *SPARK-OES* (3.1)

3.6
standardization standard
setting up sample
SUS
material with appropriate homogeneity used to recalibrate the *SPARK-OES* (3.1)

Note 1 to entry: It is not necessary to have a reference value for the concentration of each impurity.

4 Principle

The sample is prepared to obtain a flat surface. The impurities are determined by SPARK-OES, and the precious metal content is obtained by subtraction of the total content of impurities in the sample from 1 000 ‰.

Application of an electrical discharge between the sample and an inert counter-electrode generates a radiation whose wavelengths are characteristic of each element. The intensity of each wavelength is compared to calibration curves obtained from calibration standards, which allows to determine the concentration of each impurity in the sample.

Calibration standards, quality control samples and standardization standards are specific for each of the four matrixes (high purity gold, silver, platinum and palladium).

5 Reagents and equipment

5.1 Reagents

5.1.1 Argon gas, with a minimum purity of 99,998 % or as recommended by the SPARK-OES manufacturer.

5.2 Equipment

5.2.1 SPARK-OES, spectrometer with spark excitation suitable for the measurement of the intensity of optical radiation emitted at specific wavelengths; The spectrometer should have a detection limit around 1 mg/kg for each quantified impurity.

5.2.2 Hydraulic press, of appropriate strength.

5.2.3 Milling or turning machine for the sample surface preparation.

6 Sampling

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

The samples to be analysed may be in the form of disk or some other shape sufficiently massive to prevent, once flattened, undue heating during analysis and to cover entirely the hole of the analysis table.

7 Procedure

WARNING — Suitable health and safety procedures should be followed.

7.1 Surface preparation

To avoid variation of results due to the influence of surface finish, the same surface preparation shall be applied for all calibration standards, quality control samples, standardization standards and test samples.

The surface of the standard or sample shall be prepared by pressing the material with a hydraulic press (5.2.2) if needed, followed by milling or turning (5.2.3) to a finish that is sufficiently flat and smooth in order to tighten the sample chamber. Once the surface has been prepared, any contamination shall be avoided (for example fingerprints). The standard or sample shall be reasonably free from contaminants, pores, cracks, inclusions and shrinkage cavities which might otherwise affect analytical results.

7.2 Standard and sample measurement

To avoid cross-contamination between different matrixes (for example between high purity gold and high purity silver), all relevant components of the machine shall be thoroughly cleaned before use or separate tools shall be used, following the manufacturer's recommendation.

Set up the instrument in accordance with the manufacturer's instructions.

Standard or sample to be measured shall be placed on analysis table so that the hole is completely covered and there is no air leak into the discharge area. Follow the manufacturer's recommendation for cleaning the electrode and the analysis table between each sample.

The spot of spark marks shall be examined carefully; if any measurement is obviously defective (for example an unusually important burn mark is observed), further sparks shall be carried out to obtain the minimum acceptable measurements.

7.3 Calibration procedure

7.3.1 General

Calibration of the spectrometer is normally performed when the apparatus is installed. The calibration shall be in accordance with the spectrometer manufacturer's instruction manual.

Calibration of the spectrometer is performed using a series of calibration standards with the same matrix and prepared the same way as the samples to be analysed.

NOTE The preparation includes material casting, mechanical deformation and powder metallurgy.

When possible, certified reference materials should be used as calibration standards.

The number of sparks carried out on each calibration standard shall be not less than three. The average of the acceptable measurements is used for calibration.

7.3.2 Calibration curve

The calibration curve for any particular element should be composed of a minimum of three calibration standards when the calibration function is first-degree. The concentration of these calibration standards should be fairly evenly spaced over the calibration curve.

For each impurity in each calibration standard, the mean intensity is correlated to the corresponding concentration and a regression is calculated. The calibration functions are first- or second-degree mathematical equations expressed as given by Formula (1) or Formula (2):

$$W_i = a + b \times I_i \tag{1}$$

or

$$W_i = a + b \times I_i + c \times I_i^2 \tag{2}$$

where

W_i is the mass portion of element i, in mg/kg;

I_i is the measured intensity of element i;

a, b, c are the coefficients of the calibration function.

Possible optical interferences and/or inter-elements effects shall be carefully investigated, and suitable corrections shall be made.

The range of the method is determined by the range of each impurity in the calibration standards used to establish the calibration curves.

EXAMPLE For an impurity calibrated with standards containing between 0 mg/kg and 400 mg/kg of that impurity, the range of the method for that impurity is 0 mg/kg to 400 mg/kg.

When the calibration range of any particular element is large, two calibration curves covering different parts of that range may be used; in this case, each calibration curve shall be created and controlled independently as described in 7.4.

The detection limit is defined as three standard deviations of the concentration of each individual element measured in a low calibration standard or setting up sample (SUS) low standardization standard.

The correctness of the calibration shall be verified by measuring certified reference materials that were not used for the calibration.

7.4 Control procedure

Due to the natural tendency of the SPARK-OES spectrometer to drift overtime, one or several quality control samples shall be used to check the stability of the instrument at regular intervals. It is common practice to document the status of the spectrometer using a statistical process control (SPC). If the values fall outside the control limits, a standardization shall be made.

7.5 Standardization procedure

7.5.1 Standardization standards

For each impurity to be quantified, a minimum of two concentrations are recommended: low and high. The SUS low should be the precious metal as pure as possible. The SUS high should contain impurity concentrations around the maximum of the range of calibration and leading to intensities at least three times the intensities measured in the SUS low.

NOTE Several SUS high can be used, each containing different impurities; this prevent putting all impurities at high concentration in the same standard, which can change the matrix significantly and generate interferences between elements.

The standardization standards shall be initially measured during the calibration procedure, and then on regular basis to correct the drift of the spectrometer. If one of the SUS has become unusable (for example, after getting too thin to be prepared properly), it shall be replaced according to the manufacturer's instructions.

The number of sparks carried out on each standardization standard shall be not less than three. The average of the acceptable measurements is used for standardization.

7.5.2 Standardization

Long-term instrumental drift affects the intensity readings of the individual spectral lines, thus altering the coefficients a, b, c, of the calibration functions given in Formula (1) and Formula (2).

Instead of correcting the coefficients by performing a full recalibration, a standardization of the measured intensities, $I_{i,\text{meas}}$, is normally used to enable the intrument's software to use the original calibration functions.

Standardizations can be done either for all analytical channels (global standardization), or only for individual analytical channels (selective standardization), following the procedure described by the SPARK-OES instrument manufacturer.

The standardization Formula (3) allows to convert the measured intensities into their corresponding values at the time of initial calibration:

$$I_{i,\text{std}} = \alpha \times I_{i,\text{meas}} + \beta \tag{3}$$

where

$I_{i,\text{std}}$ is the standardized (drift corrected) intensity of element *i*, in the sample, to be used in Formula (1) or Formula (2);

$I_{i,\text{meas}}$ is the measured (drift affected) intensity of element *i*, in the sample.

The correction coefficients α and β are expressed as follows:

$$\alpha = \frac{I_{i,\text{nom}(\text{SUS}_\text{H})} - I_{i,\text{nom}(\text{SUS}_\text{L})}}{I_{i,\text{meas}(\text{SUS}_\text{H})} - I_{i,\text{meas}(\text{SUS}_\text{L})}} \tag{4}$$

where

$I_{i,\text{nom}(\text{SUS}_\text{H})}$ is the nominal intensity of element *i*, in the standardization standard SUS high;

$I_{i,\text{nom}(\text{SUS}_\text{L})}$ is the nominal intensity of element *i*, in the standardization standard SUS low;

$I_{i,\text{meas}(\text{SUS}_\text{H})}$ is the measured intensity of element *i*, in the standardization standard SUS high;

$I_{i,\text{meas}(\text{SUS}_\text{L})}$ is the measured intensity of element *i*, in the standardization standard SUS low.

and

$$\beta = I_{i,\text{nom}(\text{SUS}_\text{L})} - \alpha \times I_{i,\text{meas}(\text{SUS}_\text{L})} \tag{5}$$

NOTE The nominal intensity is the intensity measured at the time of initial calibration.

The correctness of the standardization shall be verified by either measuring quality control samples or certified reference materials.

7.6 Analysis procedure

The number of sparks carried out on each sample shall be not less than two, with a recommended number of three. The average of the acceptable measurements is used for the calculations.

8 Calculation and expression of the results

8.1 Calculation

By means of the calibration functions (see 7.3.2), convert the standardized intensity values, $I_{i,\text{std}}$, into mass portion values, W_i.

NOTE The conversion of the measured intensities, $I_{i,\text{meas}}$, into drift corrected intensities $I_{i,\text{std}}$ is normally operated automatically by the instrument's software.