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Jewellery and precious metals — Non destructive precious metal fineness confirmation by ED-XRF

Joaillerie, bijouterie et métaux précieux — Confirmation du titre de métal précieux par analyse non destructive ED-XRF

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Foreword

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

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

This document describes a non-destructive method to verify (confirm) the fineness of finished and semifinished jewellery items considered homogeneous by ED-XRF (energy-dispersive X-ray fluorescence).

Multiple methods are available to determine the fineness of precious metal alloys. They however are all requiring the destruction of the sample and long analysis time; for example gold cupellation by ISO 11426. Under some circumstances, destruction of the sample is not an option. This method proposes a non-destructive alternative, which allows validating a declared fineness.

The standard is not suitable for the regulatory hallmarking application. Because of the inherent higher uncertainty associated with ED-XRF measurements, some results might be inconclusive.

The document gives guidelines on the

— instrumentation,

— number and composition of calibration standards needed for calibration,

— composition of reference material needed to verify the calibration,

— number of measurement and replicates on the sample whose fineness is to be verified

— uncertainty calculation, and

— interpretation of the results.

Jewellery and precious metals — Non destructive precious metal fineness confirmation by ED-XRF

1 Scope

This document describes a non-destructive method to verify (confirm) the precious metal fineness of finished and semifinished jewellery item(s) considered homogeneous by ED-XRF (energy dispersive X-ray fluorescence), including alloys according to ISO 9202.

This document is not suitable for any coated items. WD-XRF (wavelength dispersive X-ray fluorescence) equipment cannot be used.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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 http://www.electropedia.org/

3.1
ED-XRF
energy-dispersive X-ray fluorescence
X-ray fluorescence elemental analysis method where all elements in the sample are simultaneously excited and where the intensities of the characteristic fluorescence radiation emitted by each element are quantified by separating the differential energies specific to each element

3.2
spot
target area on the sample where the X-ray beam strikes the surface

3.3
calibration standard
material with appropriate homogeneity whose exact composition is known and which can be used to calibrate the *ED-XRF* (3.1) instrument

3.4
reference material
RM
material with appropriate homogeneity whose exact composition is known and which has been established to be fit for *ED-XRF* (3.1) measurement

3.5
major element
main precious metal element of interest in the alloy whose concentration is to be verified, as well as any other element whose concentration is higher

EXAMPLE In the case of gold 375 ‰ alloy with 500 ‰ of copper, both gold and copper are the major elements.

3.6
minor element
any non-*major element* (3.5) in the alloy whose concentration is at or above 1 ‰

3.7
trace element
element present in the alloy whose concentration is below 1 ‰

Note 1 to entry: Trace elements are not taken into account for calibration. The sum of all trace elements shall be below 1 ‰.

3.8
coating
thin layer of covering on the surface of the item

4 Principle

A specific calibration is required for each alloy composition. This calibration is obtained from at least 3 standards whose composition is matching approximately the sample to be analysed. A reference material is analysed to verify the calibration. Samples can then be analysed and the precious metal fineness verified.

This document is not suitable for any coated items; the coating shall be removed by a suitable means if the fineness determination is necessary on a coated item.

5 Apparatus

5.1 ED-XRF, with the following specifications:

— X-ray tube: adapted for precious metals analysis, minimum rating 50 kV and 40 W;

— primary filter: adapted for precious metals analysis;

— collimator: minimum 2 collimators, at least one <1 mm and one ≥1 mm;

— camera: with magnification of the image of the measuring area;

— detector: Si-PIN, SDD;

— energy resolution: ≤160 eV (for Kα of Mn line), the lower eV value the better;

— detectable elements: Z = 22 (Ti) to Z = 92 (U).

6 Procedure

6.1 Calibration

6.1.1 Calibration standards

To calibrate the instrument for a specific alloy, calibration standards with the following features shall be used:

— flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

— sufficiently thick (at least 0,5 mm);

— homogeneous;

— known exact composition;

— composition of major and minor elements matching approximatively the sample to be analysed; trace elements may be ignored; a minimum of 3 standards shall be used, with the major element(s) composition covering a range up to 50 ‰.

— minor elements below 5 ‰ do not need to be present in the standards but shall be declared in the calibration; they may be calibrated using the instrument maker's calibration.

EXAMPLE For the measurement of an alloy with Au = 750 ‰, the fineness of Au in the calibration standards may be 730 ‰, 750 ‰, and 770 ‰.

6.1.2 Method calibration

The ED-XRF instrument (5.1) shall be specifically calibrated for each sample to be analysed. Each standard shall be analysed at least on 5 different positions. The standard deviation obtained for the measurements of each calibration standard shall not exceed 1,2 ‰ for the major element.

Inter-element interference corrections should be applied.

Analysis of the calibration standards, the reference material and the samples shall be performed using the same collimator size and using the same parameters (tube voltage, current, etc.). The largest possible collimator compatible with the sample target area should be used.

6.2 Verification

6.2.1 Reference material

To verify the calibration, a reference material with the following features shall be used:

— flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

— sufficiently thick (at least 0,5 mm);

— homogeneous;

— known exact composition;

— not being used as calibration standard;

— certified reference material (CRM) and materials prepared under the ISO 17034 accreditation are preferred;

— the composition of the reference material should be matching as far as practicable that of the sample; for major elements there shall be no more than 10 ‰ absolute difference between the concentration in the sample and in the reference material; for minor elements, that difference shall be no more than 20 ‰ absolute difference; trace elements may be ignored.

EXAMPLE If an alloy with a 750 ‰ Au – 240 ‰ Ag – 10 ‰ Cu is to be analysed, the concentrations in the reference material are between 740 ‰ and 760 ‰ for Au, between 260 ‰ and 220 ‰ for Ag, and between 30 ‰ and 0 ‰ for Cu.

6.2.2 Method verification

Before each batch analysis, the calibration shall be controlled by analysing the reference material.

The reference material shall be analysed at least on 5 different positions. The analysis time for each replicate shall not exceed the time used for the calibration. The standard deviation obtained for the measurements in the reference material shall not exceed 1,2 ‰ for the major element. The calibration is verified by comparing the fineness measured for the reference material (obtained by the mean value

of the analyses) and its declared value. The difference, Δ_{RM}, between those two values shall not be greater than 1,2 ‰ and will be taken into the uncertainty evaluation of the method.

If the standard deviation for the measurements or the difference between the fineness measured for the reference material and its declared value are outside the tolerances, the calibration shall be repeated.

The reference material shall be analysed at regular intervals under the same conditions to monitor the stability of the instrument.

6.3 Analysis

6.3.1 Sample preparation

Samples to be analysed may be cleaned (typically with alcohol, but also possibly by mechanical polishing of a very thin layer) and prepared for measurement for better accuracy and to exclude potential systematic errors.

NOTE Repeatability and accuracy is improved if the surface to be analysed is prepared or scraped.

6.3.2 Sample analysis

The sample shall be analysed at least on 3 different positions. Each position is analysed with a minimum of 3 replicates. The analysis time for each replicate shall be equal to the time used for the reference material.

For the major element, the standard deviation obtained for each set of replicates shall not exceed 1,2 ‰, and the standard deviation obtained for each measurement, σ_{sample}, shall not exceed 1,2 ‰.

If those standard deviations are higher than the tolerances, the result is not valid and the analysis has to be performed again.

7 Calculation and expression of the results

7.1 Calculation

The fineness of the sample, in part per thousand (‰), is directly obtained from the ED-XRF software and shall be the mean of all measurements performed.

7.2 Uncertainty

Uncertainty is evaluated by taking into account the standard deviations obtained on the reference material and the sample measurements, as well as the difference between the fineness measured for the reference material and its declared value (taking hence into account the uncertainty linked to the calibration).

$$U_{+} = \max\left(2\times\sqrt{\sigma_{RM}{}^{2}+\sigma_{sample}{}^{2}} - \Delta_{RM}; 0\right)$$

$$U_{-} = \max\left(2\times\sqrt{\sigma_{RM}{}^{2}+\sigma_{sample}{}^{2}} + \Delta_{RM}; 0\right)$$

where

σ_{RM} standard deviation on the reference material measurements;

σ_{sample} standard deviation on the sample measurements;

Δ_{RM} difference between the fineness measured for the reference material and its declared value.

7.3 Result interpretation

Fineness validation of the sample is based on ISO 10576–1. If the result falls in the inconclusive range, the measurement shall be performed again. Should the final result still be inconclusive and below the declared fineness, a destructive analysis allowing for higher precision may be performed.

The ranges are shown in Figure 1 and defined as:

— Assurance of non-conformity: $F_{measured} < F_{declared} - U_{+}$

— Inconclusive range: $F_{declared} - U_{+} \leq F_{measured} \leq F_{declared} + U_{-}$

— Assurance of conformity: $F_{measured} > F_{declared} + U_{-}$

where

$F_{measured}$ measured fineness of the sample

$F_{declared}$ declared fineness by the manufacturer

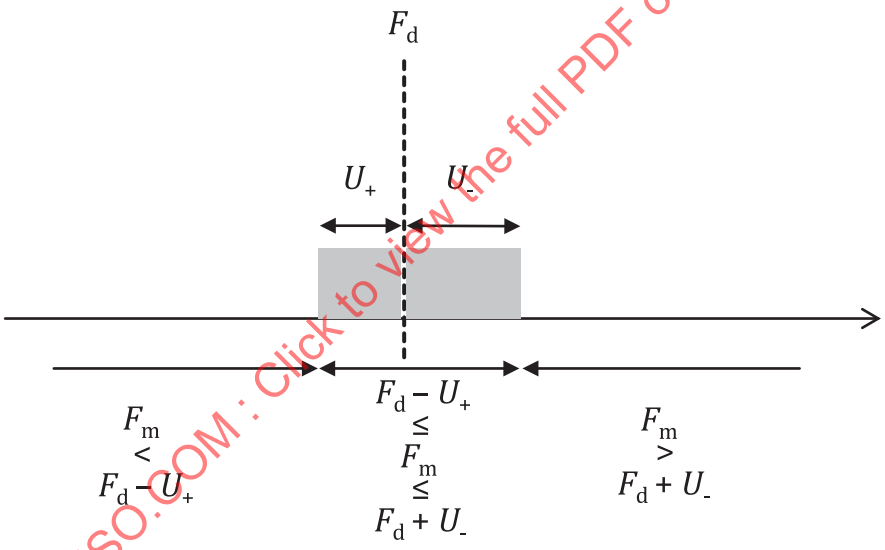

Figure 1 — Ranges for result interpretation

8 Test report

The test report shall include at least the following information:

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

b) method used by reference to this document, i.e. ISO 23345:2021;

c) result of the test: conforming, non-conforming or inconclusive result;

d) if relevant, any deviations from the method specified in this document;

e) any unusual features observed during the determination;

f) date of test;

g) identification of the laboratory carrying out the test;