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NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 18043-2013
Replacing GB/T 18043-2008

Jewellery — Determination of precious metal content — Method using X-Ray fluorescence spectrometry

首饰 贵金属含量的测定 X 射线荧光光谱法

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Foreword

This Standard was drafted according to the rules specified in GB/T 1.1-2009.

This Standard replaces the GB/T18043-2008 *Jewellery - Determination of Precious Metal Content - Method Using X-Ray Fluorescence Spectrometry.* There are mainly the following changes, compared with the GB/T 18043-2008:

— Revise the application scope. Clearly define this test method as the screening test;

— Add the terms;

— In detail, describe the basic principles of X-ray fluorescence spectrometry. Clearly state the application scope of this method in terms of working principles;

— Improve the resolution requirements of this method TO X-ray fluorescence spectrometer;

— Refine the test process. Clarify the operation steps of this method;

— Add the content description and illustration for screening and determination in accordance with measurement result's range.

— Add the description of limitations of the method;

— Add Annex A, B and C.

This Standard was proposed by China National Light Industry Council.

This Standard shall be under the jurisdiction of National Technical Committee (SAC/TC 256) on Jewellery of Standardization Administration of China.

Drafting organizations of this Standard: National Gold and Silver Products Quality Supervision and Testing Center (Nanjing), National Jewellery Quality Supervision and Inspection Center, China Institute of Metrology, National Gemstone Quality Supervision and Testing Center, National Gold and Silver Products Quality Supervision and Testing Center (Shanghai), National Gold Diamond Products Quality Supervision and Testing Center, Tianjin Product Quality Supervision and Testing Technology Research Institute, and Chengdu Product Quality Supervision and Testing Institute.

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The previous editions replaced by this Standard are as follows:

— GB/T 18043-2000, GB/T 18043-2008.

Introduction

The test laboratory that uses this Standard is required to develop implementation details such as - personnel, facilities and environmental conditions, equipment (including working reference materials), and uncertainty evaluation.

During use of this Standard, it is necessary to refer to Annex A, Annex B, and Annex C to perform sample testing, data processing, and result determination.

Jewellery — Determination of precious metal content — Method using X-Ray fluorescence spectrometry

1 Scope

This Standard specifies the methods and requirements for applying X-ray fluorescence spectrometry to determine the precious metal content in jewellery.

This Standard applies to qualitative analysis for jewellery and other art-wares. And it also applies to the screening test of precious metal content (gold, silver, platinum, and palladium) in jewellery and other art-wares.

2 Normative references

The articles contained in the following documents have become part of this document when they are quoted herein. For the dated documents so quoted, all the modifications (Including all corrections) or revisions made thereafter shall be applicable to this document.

GB 11887 Jewellery - Fineness of precious metal alloys and designation

3 Terms and definitions

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

3.1

Screening

A kind of analysis method that preliminarily quantifies the element's content in the substance to be tested.

3.2

Working reference materials

A kind of material or substance that has the adequate uniformity and the well-determined properties. It is used to calibrate the measurement device, evaluate the measurement method, or assign the value to materials. Under the condition of without the national certified reference materials and national standard samples, it is the reference material that is prepared by the laboratory independently, and of which the property value is traceable.

— Content of precious metal;

— Uniformity degree of the tested sample (including segregation, welding agent, etc.).

8 Processing of measurement results

8.1 Considering the various factors in Chapter 7, the testing laboratory shall evaluate the uncertainty on the measurement results (the evaluation method shall reference to Annex B). Determine the measurement results' range in accordance with the evaluation results (the measurement results ± measurement uncertainty). Along with the decreasing of the main-content of the precious metal, the range of the measurement results will increase.

8.2 According to the range of measurement results, conduct the screening and determining:

a) Conformity: The lower limit of the range of measurement results is greater than or equal to the low limit of the nominal value.

b) Inconformity: The upper limit of the range of measurement results is less than the low limit of the nominal value. But this Standard cannot be used alone as the basis of determination of inconformity.

c) Unable to determine: The range of measurement results includes the low limit of the nominal value. The method in the Annex C can be chosen to conduct sample treatment and result calculations. And further analysis for the sample can also be conducted in accordance with the arbitration method as stipulated in GB 11887.

8.3 The determination results can provide the testing qualitative conclusion. Or it can provide the corresponding purity range according to GB 11887.

9 Limitations

9.1 Because there are differences on the aspects of component, shape etc. between reference materials and the tested sample, the range of test elements is uncertain. Multiple factors such as surface measurement may affect. There exists certain method-risk for those samples that are determined as conformity by this Standard.

9.2 If there is dispute of the testing result, it shall be tested in accordance with the arbitration method as stipulated in GB 11887.

9.3 For the samples containing solder and of which the nominal value of the precious metal purity is 999‰, the measured value shall consider the solder's

proportion in sample. Weighted calculation shall be conducted when calculating the specific measurement results.

b — Correction curve intercept;

m — Correction curve slope;

c_j — Standard value of concentration of reference materials;

n — Number of measurement of reference materials.

B.1.4 Combined uncertainty

Combined uncertainty is calculated according to Formula (B.4):

$$u(x)=\sqrt{u_a^2+u_b^2+u_c^2+u_d^2} \qquad \cdots\cdots(B.4)$$

Where:

$u(x)$ — Combined uncertainty:

u_a — Repeatability of measurement and uncertainty of sample uniformity;

u_b — Uncertainty of reference materials;

u_c — Uncertainty of linear correction curve;

u_d — Uncertainty of sample inconsistency.

B.1.5 Expanded uncertainty

The result of measurement is normally distributed. The expanded uncertainty is calculated according to Formula (B.5):

$$U=2\times u(x) \quad (k=2) \qquad \cdots\cdots(B.5)$$

Where:

U — Expanded uncertainty;

$u(x)$ — Combined uncertainty.

B.2 Examples of uncertainty evaluation

B.2.1 Pure gold necklace

B.2.1.1 Linear uncertainty of correction curve

For serial measurement of reference materials, the standard value and measured value are shown in Table B.1. Use standard value and measured value to draw the correction curve, it is shown in Figure B.1. Substitute the values in Table B.1 INTO Formula (B.2) and Formula (B.3) respectively. The calculated linear

C.4.3 Use electronic balance (C.3.1) to weigh. It is counted as the before-melted-mass m_1.

C.4.4 Select one of the following two melting methods to perform melting treatment:

a) Melted by welding torch

1) Roll up the samples and place on firebrick (C.2.3);

2) Melt the samples with jewellery welding torch (C.3.4);

3) After the sample is solidified, it shall be immersed into 1% dilute sulphuric acid (C.2.1) for 5-10 min;

4) Remove the samples. Use water to clean the acid on the surface. Dry it.

b) Melted in furnace

1) Preheat the cupellation furnace or high-frequency induction furnace (C.3.5);

2) Perform constant-weight to the crucible (C.3.6);

3) Place the cut sample in the constant-weight crucible (C.3.6);

4) Use long-handle stainless steel clamp (C.3.7) to place the crucible (C.3.6) into the furnace for appropriate 20 min or more (approximately 1g, extend the time in the furnace depending on the weight of the sample).

C.4.5 Weigh the sample that has been melted on electronic balance (C.3.1) with sensitivity of 0.01mg. It is counted as (after-melted mass) m_2.

C.4.6 Roll the melted samples on tablet machine into thin-slices with the thickness of 0.2mm.

C.4.7 Perform the test in accordance with Article 6.3.5~6.3.6 in this Standard.

C.5 Result calculation

C.5.1 Melting correction factor of the sample shall be calculated according to Formula (C.1):

$$k = \frac{m_2}{m_1} \qquad \text{.....(C.1)}$$

Where:

k — Melting correction coefficient;

m_1 — Sample mass before-melted; the unit is g;

m_2 — Sample mass after-melted; the unit is g.

The calculation result shall keep three decimal points.

C.5.2 The final measurement result of the sample is obtained by multiplying the measurement result of the sample (the average value of corrected values obtained from multiple measurements) WITH the melting correction factor *k*, expressed in per-thousand, accurate to single digit.

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