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NATIONAL STANDARD
OF THE PEOPLE'S REPUBLIC OF CHINA
GB/T 19719-2005

Jewellery - Determination of the Release of Nickel - Method of Spectrometry

首饰 镍释放量的测定 光谱法

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Issued on: March 23, 2005

Implemented on: September 1, 2005

**Jointly Issued by: General Administration of Quality Supervision, Inspection
and Quarantine of the People's Republic of China;
Standardization Administration Committee of the People's
Republic of China.**

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Foreword

This Standard is modified in relation to European Standard EN 1811:1998 "Reference Test Method for Release of Nickel from Products Intended to Come into Direct and Prolonged Contact with the Skin" (English edition).

In order to be convenient for application, this Standard makes the following editorial changes:

- a) "This European Standard" was changed into "this Standard";
- b) The comma "," served as decimal point is replaced by the decimal point ".";
- c) "cm³" is replaced by "mL";
- d) The Foreword of the international standard was deleted;
- e) Add Appendix E (Informative) for guidance.

Appendixes A, B, C, D, E and F of this Standard are informative.

This Standard was proposed by China National Light Industry Council.

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

Drafting organizations of this Standard: National Jewellery Quality Supervision and Inspection Center, National Center of Quality Supervision & Inspection on Gold-Silver Products (Nanjing), and National Center of Quality Supervision & Inspection on Gold-Silver Products (Shanghai).

Chief drafting staffs of this Standard: Li Wujun, Wang Donghui, Fang Mingxu, Fan Jifang, Li Yukun, and Li Suqing.

Jewellery - Determination of the Release of Nickel - Method of Spectrometry

1 Scope

This Standard specifies a method for simulating the release of nickel from jewellery, in order to determine whether the item releases nickel at a rate greater than $0.5 \mu\text{g}/(\text{cm}^2 \cdot \text{week})$.

This Standard is applicable to the determination of the release of nickel from jewellery, as well as the products intended to come into direct and prolonged contact with the skin (see 4.3.2 of GB 11887-2002).

2 Normative References

The following normative documents contain provisions which, through reference in this text, constitute provisions of this Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

GB 11887-2002 Jewellery — Fineness of Precious Metal Alloys and Designation (ISO 9202:1991, NEQ)

3 Principle

The item to be tested for nickel release is placed in an artificial sweat test solution for one week. The concentration of dissolved nickel in the solution is determined by atomic absorption spectrometry, inductively-coupled plasma spectrometry or other appropriate analytical method. The nickel release is expressed in micrograms per square centimetre per week [$\mu\text{g}/(\text{cm}^2 \cdot \text{week})$].

4 Reagents

Except where indicated, all reagents, distilled water or deionized water or equivalent shall be of recognized analytical pure grade.

4.1 Sodium chloride.

water (7.1.1). Before use, ensure that the pH of the test solution is in the range 6.40 to 6.60. Use the test solution within 3 h of preparation.

7.2 Release procedure

7.2.1 Place the sample, suspended by its holder, in the test vessel (5.4). Add an amount of test solution corresponding to approximately 1 mL per cm² sample area. The suspended sample shall be totally immersed. However, it is not essential to immerse areas which are completely protected by wax or lacquer. The minimum volume of test solution added shall be 0.5 mL irrespective of the surface area. Note the sample area and the amount of the test solution used. Close the vessel with a tight lid in order to prevent evaporation of the test solution. Leave the vessel undisturbed in a thermostatically-controlled water-bath or oven (5.3) at (30 ± 2) °C for 168 h without agitation. After 1 week, remove the sample from the test solution and rinse it with a small quantity of deionized water, adding the rinsings to the test solution. Quantitatively transfer the test solution to an appropriately-sized volumetric flask washed with acid. The choice of volumetric flask size should take into account the sensitivity of the instrumentation used for the nickel determination (see 5.2.2). In order to prevent redeposition of dissolved nickel, add dilute nitric acid (4.8) and deionized water to the test solution to achieve a concentration of about 1 % nitric acid when the flask is made up to volume (V ml). The minimum final volume to which the test solution may be diluted is 2 mL.

7.3 Determination of nickel

Determine the nickel content of the test solution using an analytical spectrometer (see 5.2).

7.4 Number of replicates

Whenever possible, the determination shall be carried out on at least two identical samples.

7.5 Blank tests

Duplicate blank tests shall be carried out at the same time as the testing of the sample. Identical vessels and holders shall be used and the test procedure is identical except that no sample is placed in the vessels. Identical amounts of test solution, rinsing water and dilute nitric acid shall be used.

8 Calculations

8.1 Nickel release

The nickel release of a sample, d , expressed in micrograms per square centimetre per week [$\mu\text{g}/(\text{cm}^2 \cdot \text{week})$], is given by the equation:

$$d = \frac{V(c_1 - c_2)}{1000a} \quad (1)$$

Appendix C

(Informative)

Identification and Determination of Sample Area; and Coating of Non-significant Areas

C.1 Identification and determination of sample area

When identifying the sample area, account should be taken of the elasticity of the skin and of the manner in which items come into contact with the skin. Where articles appear to be made from uniform materials, consideration should be given to testing the whole surface (whether or not it is all in direct and prolonged contact with the skin) since errors may be introduced by the stopping-off process. For articles made from round wire (diameter < 3 mm) joined together, such as neck-chains or bracelets, the calculation of surface area should use the projected areas of all significant surfaces, unless the wire passes through the skin when the true surface is appropriate. Articles made from square, rectangular, oval, etc. sections or round wire (diameter ≥ 3 mm) can be assumed to depress the skin around the article to a depth of 2 mm. Equally, if the surface of the part of the article in contact with the skin contains indentations or depressions ≥ 2 mm, their projected areas should be included in the sample area. The surface area of articles made essentially from sheet material, such as watch-cases, some medallions and locket, can be assumed to be that area projected by all parts within 2 mm of the uncompressed skin-contact surface. The designer of an article may be able to advise on its surface area, especially when computer-aided design has been used. Autocatalytic coating techniques may be applied to measure the surface area.

C.2 Coating of non-significant areas

A number of stopping-off lacquers used in the electroplating industry have been suggested. The important points in their selection and use are:

- effective, preferably in a single coat, in preventing access of the artificial sweat to the non-significant surface;
- application using a brush is followed by air-drying;
- sufficient colour to enable their limits to be clearly visible and any deterioration during the test identified;
- do not contaminate the artificial sweat nor interfere with the release and analysis of nickel;
- are safe in use;
- degreasing prior to application.

Appendix D

(Informative)

Articles Made from Materials that can Release Small Amounts of Nickel

Where the sample area of an article is composed of one homogeneous material, it may be assumed that the nickel release from the sample area of the article is the same as that of the homogeneous material. Where the article is composed of several metals/alloys, all of which have an adjusted nickel release of less than $0.5 \mu\text{g}/(\text{cm}^2 \cdot \text{week})$, there is a strong probability that the area of the article in contact with the skin will also release less than $0.5 \mu\text{g}/(\text{cm}^2 \cdot \text{week})$ of nickel, although it is necessary for the manufacturer to check in the first instance and periodically thereafter that this is the case.

However there are a few instances in which the nickel release from such a composite may exceed the value of $0.5 \mu\text{g}/(\text{cm}^2 \cdot \text{week})$. It is therefore incumbent on the manufacturer to be aware of the situations in which this may occur. These include:

- the occurrence of bimetallic corrosion when a nickel-containing alloy is in electrical contact with a more noble metal/alloy in the sample area. Examples are
 - a) contact of a stainless steel of marginal passivity, due to low chromium content or a high sulphur content, with a more noble metal/alloy such as gold, platinum or a higher alloyed stainless steel;
 - b) brazing of a stainless steel with a silver-based alloy;
- change of surface condition as a result of welding, brazing, soldering or other heat treatment; or damage to the surface in the course of assembly;
- any degreasing, grinding or polishing operation that modifies the surface of the article.

In calculating the value of nickel release for nickel-containing materials, for which homogeneous samples of uniform shape can be obtained. In calculating the value of nickel release from articles it is necessary to multiply the observed value by 0.1 to take account of the above variation and also errors that arise in stopping-off non-significant areas; in measurement of the areas that come into direct and prolonged contact the skin; and in the testing of non-uniform shapes.

Where coated samples representative of the materials used in the production of finished articles are to be tested, they must be prepared at the same time as the articles that are to be placed on the market, using the same coating conditions, technique and solutions.

Bibliography

[1] EN 71-3:1994, Safety of Toys — Part 3: Migration of Certain Elements.

[2] ISO 5725, Accuracy (Trueness and Precision) of Measurement Methods and Results.

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