SLOVENSKI STANDARD
SIST EN 1811:2011

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Nadomešča:
SIST EN 1811:1999
SIST EN 1811:1999+A1:2008

Primerjalna preskusna metoda za sproščanje niklja iz izdelkov, vstavljenih v prebodene dele človeškega telesa, in izdelkov, ki so v neposrednem in daljšem stiku s kožo

Reference test method for release of nickel from post assemblies which are inserted into pierced parts of the human body and products intended to come into direct and prolonged contact with the skin

Referenzprüfverfahren zur Bestimmung der Nickellässigkeit von sämtlichen Stäben, die in durchstochene Körperteile eingeführt werden und Produkten, die unmittelbar und länger mit der Haut in Berührung kommen

Méthode d'essai de référence relative à la libération du nickel par les assemblages de tiges qui sont introduites dans les parties percées du corps humain et les produits destinés à entrer en contact direct et prolongé avec la peau

Ta slovenski standard je istoveten z: **EN 1811:2011**

ICS:

39.060	Nakit	Jewellery

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EUROPEAN STANDARD

NORME EUROPÉENNE

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EN 1811

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ICS 39.060

Supersedes EN 1811:1998+A1:2008

English Version

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This European Standard was approved by CEN on 5 February 2011.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 1811:2011) has been prepared by Technical Committee CEN/TC 347 “Methods for analysis of allergens”, the secretariat of which is held by DS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by March 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document, together with EN 16128:2011, supersedes EN 1811:1998 +A1:2008.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document supports essential requirements of Commission Regulation (EC) No 1907/2006 (REACH) of the European Parliament and the Council.

List of the significant technical changes that have been made in this new version of EN 1811 in comparison with the former edition EN 1811:1998+A1:2008:

The scope of the former European Standard was divided: EN 1811:2011 is applicable for all products but spectacle frames and sunglasses; EN 16128:2011 is applicable for spectacle frames and sunglasses;

List of the significant technical changes of EN 1811:2011 as compared to the former European Standard:

- The scope was expanded to include all post assemblies which are inserted into pierced parts of the human body;
- The preparation of the test solution was tested and changed;
- The correction factor was eliminated and the concept of measurement uncertainty introduced;
- The Standard contains a new normative Annex C on the preparation of articles prior to nickel testing;

EN 16128:2011 is technically unchanged as compared to the former European Standard EN 1811:1998+A1:2008.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.,

Introduction

Adverse skin reaction to nickel has been known for many decades. Nickel is the most frequent cause of contact allergy in Europe, and 10 % to 20 % of the patch tested female population and 1 % to 3 % of the patch tested male population are allergic to nickel. Skin absorption of nickel ions, which are released from some nickel-containing materials which are inserted into pierced ears or other pierced parts of the human body or which are in direct and prolonged contact with the skin, causes sensitisation. Further exposure to soluble nickel salts results in allergic contact dermatitis. It is known that sensitisation to nickel requires higher exposure levels than does the elicitation in already sensitised individuals. There is a large variation in the degree of sensitivity to nickel between individuals. This widespread health problem has forced the introduction of a number of measures designed to reduce its prevalence. These measures include the requirements of this standard which provides an *in-vitro* chemical test that correlates as far as possible with the variable human biological reactions that occur when metallic articles containing nickel are in direct and prolonged contact with the skin and pierced parts of the body. The standard provides a measure of the amount of nickel release from an article immersed for one week in artificial sweat. The standard also describes the preparation of a quality control material intended to assist a laboratory in achieving an acceptable precision.

Clinical patch-testing of a small selection of nickel-containing alloys and coatings on nickel-sensitized persons indicates that high and low results achieved with the present analytical method correspond closely with patch-test reactivity. Moreover, a nickel migration limit of 0,5 µg/cm^2/week for articles intended to come into direct and prolonged contact with the skin and a nickel migration limit of less than 0,2 µg/cm^2/week for all post piercing assemblies inserted into pierced ears and other pierced parts of the human body has been set in Commission Regulation (EC) No 1907/2006 of the European Parliament and the Council (in the current version).

1 Scope

This European Standard specifies a method for simulating the release of nickel from all post assemblies which are inserted into pierced ears and other pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin in order to determine whether such articles are in compliance with No. 27 Annex XVII of Regulation (EC) No 1907/2006 of the European Parliament and of the Council (REACH).

Spectacle frames and sunglasses are excluded from the scope of this European Standard.

NOTE Spectacle frames and sunglasses are subject to the requirements of EN 16128:2011 which provides an unchanged re-publication of the technical requirements that had previously been specified in EN 1811:1998, but restricted in scope to apply only to spectacle frames and sunglasses.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 12472, *Method for the simulation of wear and corrosion for the detection of nickel release from coated items*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

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

3.1
barrette
component used to secure the watchstrap to the case

3.2
homogeneous
consisting of a single material having a common surface finish

3.3
post assembly
ear stud or body piercing article

3.4
release solution
solution resulting from the release procedure according to 8.2

3.5
representative
best estimate for the effective release rate of all surfaces which are in direct and prolonged contact with the skin or pierced parts of the body under normal conditions of use

NOTE This property is defined with respect to the release rate.

3.6
sample area
a
surface(s) that is(are) immersed in the test solution and not covered with a masking agent

3.7
test solution
solution as prepared according to 8.1

3.8
watch crown
winder used to alter the time/date

4 Principle of the procedure

The article 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 an appropriate analytical method, for example inductively-coupled plasma spectrometry. The nickel release is expressed in micrograms per square centimetre per week (µg/cm^2/week).

NOTE Indicative information on the extent of nickel release can be obtained by performing one of the tests specified in CR 12471.

5 Reagents

Except where indicated, all reagents shall be of recognized pro analysis, p.a., grade or better and shall be free of nickel.

5.1 Deionised water according to EN ISO 3696:1995, grade 2.

5.2 Sodium chloride.

5.3 DL-lactic acid, ρ = 1,21 g/ml, > 88 % (m/m).

5.4 Urea.

5.5 Sodium hydroxide in solid tablets, min 98 % pure dehydrate.

5.6 Preparation of 1 M sodium hydroxide solution.

Weigh 4 g ± 0,01 g of sodium hydroxide (5.5) and transfer into a 100 ml beaker and add 50 ml of deionised water (5.1). Stir and cool to room temperature. Transfer the solution to a 100 ml volumetric flask and make up to volume with deionised water (5.1).

5.7 Preparation of 0,1M sodium hydroxide solution.

Add 25 ml of 1 M sodium hydroxide (5.6) in a 250 ml volumetric flask and make up to volume with deionised water (5.1).

5.8 Hydrochloric acid, ρ = 1,16 g/ml, 32 % v/v.

5.9 Preparation of 0,1 M hydrochloric acid solution.

Transfer 10 ml of hydrochloric acid (5.8) into a 100 ml volumetric flask and make up to volume with deionised water (5.1).

5.10 Nitric acid, ρ = 1,40 g/ml, 65 % (m/m).

5.11 Dilute nitric acid, approximately 5 % (m/m).

Transfer 30 ml of nitric acid (5.10) into a 500-ml beaker containing about 350 ml of deionised water (5.1). Stir and cool to room temperature. Transfer the solution to a 500-ml volumetric flask and make up to volume with deionised water.

5.12 Degreasing solution.

Dissolve 5 g of an anionic surface-active agent such as sodium dodecylbenzene sulfate or sodium alkylaryl sulfate in 1 000 ml deionised water (5.1). An appropriately diluted, neutral, commercially available detergent may be used.

5.13 Wax or lacquer (suitable for electroplating purposes) capable of protecting a surface from nickel release.

The wax or lacquer shall be shown to prevent nickel release from a nickel-releasing surface when one or more coats of the wax or lacquer are applied in the same manner as on a test sample and shall not affect the nickel content of the release solution. The suitability of the wax / lacquer shall be tested.

NOTE Information on sourcing of a suitable wax or lacquer is available from the CEN/CENELEC Management Centre.

6 Apparatus

6.1 A pH-meter, accurate to ± 0,05 pH.

6.2 An analytical instrument capable of detecting a concentration of 0,01 mg nickel per litre in the final release solution.

It is recommended that either an inductively-coupled plasma spectrometer (ICP-OES, optical emission, or ICP-MS, mass spectrometer) or an electro thermal excitation atomic absorption spectrometer (GFAAS) is used.

6.3 Thermostatically controlled water-bath or oven with or without cooling option, capable of maintaining a temperature of (30 ± 2) °C.

6.4 A vessel with lid, both composed of a non-metallic, nickel-free and nitric-acid-resistant material, such as glass and/or polypropylene and/or polytetrafluoroethylene and/or polystyrene.

The sample shall be suspended in the liquid by a holder made from the same materials as listed above, so as to avoid contact of the sample area (7.1.1) with the walls and base of the vessel. The size and shape of vessel and holder shall be chosen so as to minimize the volume of test solution required to completely cover the article to be tested.

In order to remove any trace of nickel, the vessel and holder shall be pre-treated by being stored in a solution of dilute nitric acid (5.11) for at least 4 h. After acid cleaning, rinse the vessel and holder with deionised water and dry.

6.5 Device for length measuring, for example a digital calliper with a minimum resolution of 50 µm or a micrometer with a minimum resolution of 5 µm.

7 Samples

7.1 Sample area

7.1.1 Definition of sample area

Only the surface(s) that come(s) into direct and prolonged contact with the skin and/or that have contact with the pierced parts of the body shall be tested (sample area).

In case of articles which are made of uniform material(s), consideration should be given to testing the whole surface (whether or not it is all in direct and prolonged contact with the skin or with pierced parts of the body) since errors can be introduced by the masking process (see 7.1.3).

The test laboratory shall refer to C.4 in order to determine which surfaces are to be tested.

7.1.2 Determination of sample area

Determination of the sample area (*a*) in square centimetres is achieved by marking the contour of the sample area assuming that the article is worn or used as intended (refer to Annex C) and measuring it by an appropriate measuring device (6.5). In order to achieve the required degree of analytical sensitivity, a minimum sample area of 0,2 cm^2 shall be tested. If necessary, identical articles may be tested together to obtain this minimum area.

The closer the nickel release is to the limits laid down in the regulation, or the smaller the sample area is, the more precise the surface measurement needs to be.

7.1.3 Masking of areas other than sample area

In order to prevent release of nickel from areas other than the sample area, such areas shall be removed or protected from the test solution. This can be achieved after degreasing (refer to 7.2) by the application of one or more coatings of a wax or lacquer (5.13) which has been shown to protect from nickel release.

The test laboratory shall refer to C.4 in order to determine which surfaces are to be tested.

7.2 Sample degreasing before testing

Gently swirl the sample for 2 min in degreasing solution (5.12) at room temperature. Rinse thoroughly with deionised water and dry using an absorbing cloth. After degreasing, articles shall be handled with plastic forceps or clean protective gloves.

NOTE This cleaning stage is intended to remove extraneous grease and skin secretions due to handling, but not any protective coatings.

7.3 Quality control samples

As a quality control check, the nickel release from a quality control sample shall be determined (refer to Annex B) on a frequent basis.

The quality control sample shall be degreased in the same way as the sample according to 7.2 and shall be used only once.

8 Procedure

8.1 Preparation of test solution

The test solution consists of deionised water (5.1) containing:

— 0,5 % (m/m) sodium chloride (5.2);

— 0,1 % (m/m) lactic acid (5.3);

— 0,1 % (m/m) urea (5.4); and

— 1 M (5.6) and 0,1 M (5.7) sodium hydroxide solution.

The test solution shall be prepared as follows:

Pour 900 ml of freshly prepared deionised water (5.1) to a 1 000 ml beaker. Add 1,00 ± 0,01 g of urea (5.4), 5,00 ± 0,05 g of sodium chloride (5.2) and 1,00 ± 0,01 g of lactic acid (5.3), and stir until dissolved.

Calibrate a pH meter in accordance with the manufacturer's instructions using freshly prepared buffer solutions.

Immerse the pH electrode into the test solution and measure the pH. Slowly and gently, add drop by drop a volume of 1 M sodium hydroxide (5.6) until a pH of 5,5 ± 0,05 is reached and subsequently with continuous stirring, add slowly and gently drop by drop a volume of 0,1 M sodium hydroxide (5.7) until a pH 6,5 ± 0,05 is reached and remains stable.

Measure the pH after 10 min from the last addition of 0,1 M sodium hydroxide to ensure that the pH is in the range 6,5 ± 0,05.

Transfer the solution to a 1 000 ml volumetric flask and make up to volume with deionised water. Before use, ensure that the pH of the test solution is in the range of pH 6,5 ± 0,05.

If it is necessary to reduce the pH of the solution to 6,5 ± 0,05 before testing, this shall be done by adding slowly and gently with continuous stirring drop by drop a volume of 0,1 M hydrochloric acid (5.9).

The test solution shall be prepared daily.

8.2 Release procedure

NOTE In the following text the term "test solution" represents the solution as prepared according to 8.1, the "release solution" is the solution resulting from the release procedure. See also definitions.

Place the sample, suspended by its holder, in the test vessel (6.4). Add an amount of test solution corresponding to approximately 1 ml per cm^2 sample area. The suspended sample area shall be totally immersed. It is not necessary to immerse areas which are completely protected by wax or lacquer (see 7.1.3). The minimum volume of test solution 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 (6.3) at (30 ± 2)°C for (168 ± 2) h without agitation.

The quality control sample (7.3) shall be determined and suspended in an appropriate volume of test solution. It shall be treated in the same manner as a sample.

After (168 ± 2) h, slowly remove the sample from the release solution. To collect solution contained in cavities of the sample, the sample shall be turned appropriately. The sample shall not be rinsed.