SLOVENSKI STANDARD
SIST EN 1811:2023

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Nadomešča:
SIST EN 1811:2011+A1:2015

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 all post assemblies which are inserted into pierced parts of the human body and articles 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 Erzeugnissen, die unmittelbar und länger mit der Haut in Berührung kommen

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

Ta slovenski standard je istoveten z: **EN 1811:2023**

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39.060	Nakit	Jewellery

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EUROPEAN STANDARD

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English Version

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This European Standard was approved by CEN on 2 January 2023.

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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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 1811:2023) has been prepared by Technical Committee CEN/TC 347 “Methods for analysis of allergens”, the secretariat of which is held by SNV.

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of an identical text or by endorsement, at the latest by August 2023, and conflicting national standards shall be withdrawn at the latest by August 2023.

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

This document supersedes EN 1811:2011+A1:2015.

EN 1811:2023 includes the following significant technical changes with respect to EN 1811:2011+A1:2015:

— unit for nickel release changed to $\mu g \cdot cm^{-2} \cdot week^{-1}$ (expressed as $\mu g/cm^2$/week in the Regulation);

— Note 1 in Clause 1 shortened;

— Clause 3 Terms and definitions updated, terms surface finish and disassemble were added;

— Clause 5, permission of ready to use solutions in reagents added;

— notes for application of wax or lacquer added;

— information of number of test samples added,

— definition of sample area clarified;

— handling of small samples and filtering of release solution in the release procedure clarified;

— filtering of release solutions and blank solutions clarified;

— test report updated;

— Annex B, Requirements for Quality control material deleted;

— Annex B for preparation of samples revised;

— Table B.1, General procedure for post assemblies and inhomogeneous articles added;

— Figure B.9, Flowchart for sample preparation and testing procedure for complete watches added;

— Bibliography updated.

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

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

Introduction

Nickel is the most frequent cause of contact allergy in Europe and adverse skin reaction to nickel has been known for many decades.

Skin absorption of a sufficient amount 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, is required to cause sensitization [9].

Once sensitized, further exposure to soluble nickel ions results in allergic contact dermatitis. It is known that sensitization to nickel requires higher exposure levels than does the elicitation in already sensitized 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 document which provides an *in vitro* chemical test that correlates as far as possible with the variable human biological reactions that occur when metallic or coated articles containing nickel are in direct and prolonged contact with the skin or inserted into pierced parts of the body. This document provides a test method to determine the release of nickel from an article immersed for one week in artificial sweat. Clinical patch-testing of a 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, Regulation (EC) No 1907/2006 of the European Parliament and of the Council (in the current version) [9] sets a nickel migration limit of 0,5 $\mu g \cdot cm^{-2} \cdot week^{-1}$ (expressed as $\mu g/cm^2$/week in the Regulation) for articles intended to come into direct and prolonged contact with the skin and a limit of less than 0,2 $\mu g \cdot cm^{-2} \cdot week^{-1}$ (expressed as $\mu g/cm^2$/week in the Regulation) for all post piercing assemblies inserted into pierced ears and other pierced parts of the human body.

1 Scope

This document 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 in 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 document.

NOTE Spectacle frames and sunglasses are subject to the requirements of EN 16128.

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.

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

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

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
homogeneous
of uniform composition throughout that cannot be separated into different materials

Note 1 to entry: A coated material is not homogeneous.

3.2
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 and foreseeable conditions of use

3.3
surface finish
appearance and texture of a surface characterized by its smoothness or roughness

Note 1 to entry: In order of increasing smoothness, the surface finish can be typically described as cut finish, machined finish, brushed finish, matt finish, satin finish, mirror or polished finish.

3.4
sample area
a
surface(s) that is/are immersed in the test solution and not covered with a masking agent

3.5
disassemble
separate into its constituent parts without damaging these parts or their surface condition

3.6
post assembly
ear stud or body piercing article

3.7
test solution
solution as prepared according to 8.1

3.8
release solution
solution resulting from the release procedure according to 8.2

4 Principle of the procedure

The article to be tested for nickel release is placed in a solution of artificial sweat for one week undisturbed. 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 ($\mu g \cdot cm^{-2} \cdot week^{-1}$).

Articles with non-nickel-containing outer coatings that come into direct and prolonged contact with the skin shall be tested according to EN 12472, followed by the procedure in this document.

NOTE Information on the presence of soluble nickel can be obtained by performing one of the tests specified in CEN/TR 12471.

5 Reagents

Except where indicated, all reagents, materials and apparatus that can come into contact with test parts or reagents shall be demonstrably free of nickel, and all reagents shall be of recognized analytical grade or better.

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

5.2 Sodium chloride, NaCl.

5.3 DL-lactic acid, $CH_3CHOHCOOH$, mass fraction of > 88 %.

5.4 Urea, $CO(NH_2)_2$.

5.5 Sodium hydroxide pellets, purity mass fraction of ≥ 98 %, anhydrous.

5.5.1 Preparation of 1 mol/l sodium hydroxide solution.

Weigh 4 g ± 0,01 g sodium hydroxide (5.5), transfer to a 100 ml beaker and add 50 ml 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).

Alternatively, it is possible to use 1 mol/l ready-to-use solution.

5.5.2 Preparation of 0,1 mol/l sodium hydroxide solution.

Transfer 25 ml of the 1 mol/l sodium hydroxide solution (5.5.1) to a 250 ml volumetric flask and make up to volume with deionised water (5.1).

Alternatively, it is possible to use 0,1 mol/l ready-to-use solution.

5.6 Hydrochloric acid, mass fraction of 32 % to 37 %.

5.6.1 Preparation of 0,1 mol/l hydrochloric acid solution.

Transfer 1 ml of hydrochloric acid (5.6) into a 100 ml volumetric flask and make up to volume with deionised water (5.1).

Alternatively, it is possible to use 0,1 mol/l ready-to-use solution.

5.7 Nitric acid, mass fraction of 65 % to 70 %.

5.7.1 Diluted nitric acid, approximately mass fraction of 5 %.

Transfer 30 ml of nitric acid (5.7) 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.

5.8 Degreasing solution.

Dissolve 5 g of an anionic surface-active agent, e.g. 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.

5.9 Wax or lacquer (e.g. 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 are applied in the same manner as on a test sample and shall not affect the nickel content of the release solution. The wax/lacquer shall be tested to demonstrate its suitability for the intended purpose.

NOTE 1 When using wax, the samples are dipped into the molten wax. To improve the adhesion of the wax, the article can be pre-warmed, provided that the article's properties are not changed.

NOTE 2 To cover cracks and fissures, it might be necessary to apply more than one coating of lacquer, 2 to 3 coats might be required to achieve the desired outcome. It can be helpful to inspect the quality of masking visually with a low-powered (e.g. 2x to 5x) magnifier.

6 Apparatus

6.1 Usual laboratory apparatus.

6.2 A pH-meter, accurate to ±0,05 pH.

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

It is recommended to use either an inductively-coupled plasma spectrometer (ICP-OES [optical emission], or ICP-MS [mass spectrometer]) or an electro thermal excitation atomic absorption spectrometer (GF-AAS).

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

6.5 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 limit contact of the sample area (7.2.1) 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.

Threads may be used to hang the samples in the liquid without touching the vessel.

In order to remove any trace of nickel, the vessel and holder should be pre-treated by being stored in a solution of diluted nitric acid (5.7.1) for at least 4 h. After acid cleaning, rinse the vessel and holder with deionised water and dry. Alternatively single-use vessels may be used.

6.6 Device for measuring length, for example a digital calliper accurate to at least 50 µm or a micrometer accurate to at least 5 µm.

7 Samples

7.1 Number of test samples

A minimum of three test samples of the same batch shall be submitted for testing wherever possible as items in a batch can vary.

7.2 Sample area

7.2.1 Definition of sample area

Only the surface(s) that come(s) into direct and prolonged contact with the skin and/or come(s) into contact with the pierced parts of the body shall be tested (i.e. the sample area). The test laboratory should refer to B.4 in order to determine which surfaces should be tested.

For the calculation of the sample area for the nickel release determination, the areas of non-nickel-containing parts and masked parts shall be excluded from the calculation.

7.2.2 Determination of sample area

Wherever possible the sample area (a) in square centimetres shall be determined by marking the contour of the sample area. It shall be assumed that the article is worn or used as intended (refer to Annex B) and measured by an appropriate measuring device (6.6). In cases where it is not appropriate to mark the contours, the sample area may be determined by using common geometrical shapes. (See B.5.3.)

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 specified in the REACH regulation, or the smaller the sample area is, the more precise the surface measurement needs to be.

7.2.3 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 (see 7.3) by the application of one or more coatings of a wax or lacquer (5.9) that has been shown to protect from nickel release.

The test laboratory should refer to B.4 in order to determine which surfaces should be tested.

NOTE If the surface in contact with the skin is representative of the whole article, consider testing it without masking. See B.4 for guidance.

7.3 Sample degreasing before testing

Gently swirl the sample for 2 min in degreasing solution (5.8) at room temperature. Rinse thoroughly with deionised water and dry using absorbing paper. 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.

8 Procedure

8.1 Preparation of test solution

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

— mass fraction of 0,5 % sodium chloride (5.2);

— mass fraction of 0,1 % lactic acid (5.3);

— mass fraction of 0,1 % urea (5.4); and

— 1 mol/l (5.5.1) and 0,1 mol/l (5.5.2) sodium hydroxide solution.

The test solution shall be prepared as follows:

Pour 900 ml of freshly prepared deionised water (5.1) into 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 buffer solutions of the appropriate range.

Immerse the pH electrode into the test solution and measure the pH. Slowly and carefully, add drop by drop a volume of 1 mol/l sodium hydroxide solution (5.5.1) until a pH of 5,5 is reached and subsequently with continuous stirring, add slowly and carefully drop by drop a volume of 0,1 mol/l sodium hydroxide solution (5.5.2) until a pH 6,50 ± 0,05 is reached and remains stable.

Measure the pH after 10 min from the last addition of 0,1 mol/l sodium hydroxide solution to ensure that the pH is in the range 6,50 ± 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 6,50 ± 0,05.

If it is necessary to reduce the pH of the solution to 6,50 ± 0,05 before testing, this shall be done by adding slowly and carefully with continuous stirring drop by drop a volume of 0,1 mol/l hydrochloric acid solution (5.6.1).

For each test, the test solution shall have been prepared within the last 24 h.

8.2 Release procedure

NOTE 1 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 3.7 and 3.8.

Place the degreased sample (7.3) in the test vessel (6.5). To limit contact of the sample with the container, it may be suspended by a holder, with threads or a support.