SLOVENSKI STANDARD
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Primerjalna preskusna metoda za sproǥļ anje niklja iz izdelkov, vstavljenih v prebodena uǥesa ali druge prebodene dele ļ loveǥkega telesa, ki so v neposrednem in daljǥem stiku s koǵo

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

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 347.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (prEN 1811:2009) has been prepared by Technical Committee CEN/TC 347 “Methods for analysis of allergens”, the secretariat of which is held by DS.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 1811:1998.

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 % - 20 % of the patch tested female population and 1 % - 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. They include this standard which attempts to provide 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 release rate threshold of 0,5 µg/cm^2/week has been set in European Parliament and Council Directive 94/27/EC (OJ No. L188 of 22.7.94) for products intended to come into direct and prolonged contact with the skin. For all post piercing assemblies inserted into pierced ears and other pierced parts of the human body a nickel release rate threshold of less than 0,2 µg/cm^2/week has been set in Commission Directive 2004/96/EC (OJ No. L 301/51 of 27.9.2004).

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 items are in compliance with the European Directive 76/769/EEC as amended by 94/27/EC and 2004/96/EC.

DRAFTING REMARK Subject to the final positive decision of the European Commission, CEN/TC 347/TG 1 supports the following wording to be included in the scope of this standard:
Spectacle frames and sunglasses are excluded from the scope of this European Standard.

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*

ISO 3696, *Water for analytical laboratory use - Specification and test methods*

3 Reagents

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

3.1 Deionised water according to ISO 3696

3.2 Sodium chloride

3.3 DL-Lactic acid, ρ = 1,21 g/ml, > 88 % (m/m)

3.4 Urea

3.5 Sodium Hydroxide in solid tablets, 99,8 % pure dehydrate

3.6 Preparation of 1 M sodium hydroxide solution

Weigh 4 g of sodium hydroxide (3.5) ± 0,01 g and transfer into a 100 ml beaker containing 50 ml of deionised water (3.1). Stir and cool to room temperature. Transfer the solution to a 100 ml volumetric flask and make up to volume with deionised water (3.1).

3.7 Preparation of 0,1M sodium hydroxide solution

Add 25,0 ml of 1 M sodium hydroxide (3.6) in a 250 ml volumetric flask and make up to volume with deionised water (3.1).

3.8 Hydrochloric acid, ρ = 1,18 g/ml, 37 % v/v

3.9 Preparation of 0,1 M hydrochloric acid solution

Transfer 10 ml of hydrochloric acid (3.8) into a 100 ml volumetric flask and make up to volume with deionised water (3.1).

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

3.11 Dilute nitric acid, approximately 5 % (m/m)

Transfer 30 ml of nitric acid (3.10) into a 500-ml beaker containing about 350 ml of deionised water. Stir and cool to room temperature. Transfer the solution to a 500-ml volumetric flask and make up to volume with deionised water.

3.12 Degreasing solution

Dissolve 5 g of an anionic surface-active agent such as sodium dodecylbenzene sulfonate or sodium alkylaryl sulfonate in 1000 ml water. An appropriately-diluted, neutral, commercially available detergent may be used.

3.13 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 tested for nickel release according to clause 6 (see Annex C).

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

4 Apparatus

4.1 A pH-meter, accurate to ± 0,05 pH

4.2 An analytical spectrometer capable of detecting a concentration of 0,01 mg nickel per litre in the final test solution. It is recommended that either an inductively-coupled plasma spectrometer (optical emission or mass spectrometer) or an electrothermal excitation atomic absorption spectrometer is used.

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

4.4 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 minimize contact of the sample area (5.1.1) 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 object 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 (3.11) for at least 4 h. After acid treatment, rinse the vessel and holder with deionised water and dry.

4.5 Digital caliper with a minimum resolution of 50 µm or a micrometer with a minimum resolution of 5 µm.

5 Samples

5.1 Sample area

5.1.1 Definition of sample area

The surfaces that are immersed in the test solution and that are not covered with a masking agent are defined as "sample area" and shall be used for calculation purposes (7.1).

As far as possible, 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 should be tested.

However, where articles appear to be made from 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 are introduced by the masking process (see also C.5.1).

The test laboratory shall consult Annex C.5 in order to determine which surfaces are to be tested.

5.1.2 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 item is worn or used as intended (see Annex C) and measuring it by an appropriate measuring device (4.5). 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 items may be treated together to obtain this minimum area.

NOTE If an item is being tested to ascertain its conformity with Directives 94/27/EC and 2004/96/EC, the accuracy with which the sample area of the item has to be determined is dependent on the nickel release of this item.

The closer the nickel release is to the limit laid down in the directive, the more accurately the surface area has to be determined.

5.1.3 Areas other than sample areas

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 may be achieved after degreasing (see 5.2) by the application of one or more coatings of a wax or lacquer (3.13) which has been shown to protect from nickel release.

The test laboratory shall consult Annex C.5 in order to determine which surfaces are to be tested.

5.2 Sample degreasing before testing

Gently swirl the sample for 2 min in degreasing solution (3.12) at room temperature. Rinse thoroughly with deionised water and dry using an absorbing cloth. After degreasing, items should 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. It will also substantially remove any nickel contamination that might be present on the surface of the item. If there is a requirement to determine this nickel contamination, the cleaning stage should be omitted. Omission of this cleaning stage might affect the nickel release from the item.

5.3 Quality control samples

As a quality control check, the nickel release from a quality control sample shall be determined (see Annex A and Annex B).

The quality control sample shall be degreased in the same way as the sample according to 5.2 and shall be used only once.

6 Procedure

6.1 Principle of the procedure

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 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).

6.2 Preparation of test solution

The test solution consists of deionised water (3.1) containing:

— 0,5 % (m/m) sodium chloride (3.2);

— 0,1 % (m/m) lactic acid (3.3);

— 0,1 % (m/m) urea (3.4); and

— NaOH, in solid tablets, 99,8 % pure dehydrate (3.5)

The test solution shall be prepared as follows:

Add 900 ml of freshly prepared deionised water (3.1) to a 1000 ml beaker. Add 1,00 ± 0,01 g of urea (3.4), 5,00 ± 0,05 g of sodium chloride (3.2) and 1,00 ± 0,01 g of lactic acid (3.3), 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 (3.6) until a pH of 5,5 ± 0,05 is reached and with continuous stirring, add slowly and gently drop by drop a volume of 0,1 M sodium hydroxide (3.7) until a pH 6,5 ± 0,05 is reached and remains stable.

Measure the pH after 10 minutes 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 1000 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 (3.9).

The test solution shall be used within 3 hours of preparation.

6.3 Release procedure

In the following text the term "test solution" represents the solution as prepared according to 6.2, the "release solution" is the solution resulting from the release procedure.
Place the sample, suspended by its holder, in the test vessel (4.4). 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 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 (4.3) at (30 ± 2°C) for 168 ± 2 h without agitation.
If a quality control sample (5.3) is to be determined, it should be suspended in an appropriate volume of test solution and treated in the same manner as a sample.
After 168 ± 2 h, slowly remove the sample from the release solution. To remove solution contained in cavities of the sample, the sample shall be turned appropriately. The sample shall not be rinsed.

Quantitatively transfer the release solution to an appropriately-sized (see note) volumetric flask washed with dilute nitric acid (3.11). In order to prevent redeposition of dissolved nickel, add dilute nitric acid (3.11), to