SLOVENSKI STANDARD
SIST-TP CEN/TR 12471:2022

01-september-2022

Nadomešča:
SIST CR 12471:2004

Presejalna metoda za prisotnost niklja v izdelkih, vstavljenih v prebodene dele človeškega telesa, in izdelkih, namenjenih neposrednemu in daljšemu stiku s kožo

Screening test for the presence of nickel in articles which are inserted into pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin

Screeningverfahren für die Nickelabgabe aus Erzeugnissen, die in durchstochene Körperteile eingeführt werden, und Erzeugnissen, die unmittelbar und länger mit der Haut in Berührung kommen

Méthode de tri pour la libération du nickel sur les articles qui sont introduites dans les parties percées du corps humain et les produits destinée à entrer en contact direct et prolongé avec la peau

Ta slovenski standard je istoveten z: **CEN/TR 12471:2022**

ICS:

39.060	Nakit	Jewellery

SIST-TP CEN/TR 12471:2022 **en**

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN/TR 12471

February 2022

ICS 39.060

Supersedes CR 12471:2002

English Version

Screening test for the presence of nickel in articles which are inserted into pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin

Méthode de tri pour la présence de nickel dans les articles introduits 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 Technical Report was approved by CEN on 24 January 2022. It has been drawn up by the Technical Committee CEN/TC 347.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. CEN/TR 12471:2022 E

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European foreword

This document (CEN/TR 12471:2022) has been prepared by Technical Committee CEN/TC 347 “Methods for analysis of allergens”, the secretariat of which is held by SNV.

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 CR 12471:2002.

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.

Introduction

This document provides a completely revised and restructured edition of CR 12471:2002, *Screening tests for nickel release from alloys and coatings in items that come into direct and prolonged contact with the skin*.

This document has been prepared for the detection of nickel release. The described method is cost-effective. It has particular relevance in relation to nickel contact dermatitis. In order to decrease the incidence of nickel contact dermatitis, the European Commission introduced in 1994 the Nickel Directive (94/27/EC), which specified a limit value for nickel content in articles inserted into pierced parts of the human body and a limit value for nickel release from articles intended to come into direct and prolonged contact with the skin.

The Nickel Directive has been replaced by REACH regulation (EC) No 1907/2006 in 2006. According to REACH regulation Annex XVII, entry no 27, the nickel release for articles inserted into pierced parts of the human body, has to be less than 0,2 µg/cm^2/week, whereas for articles intended to come into direct and prolonged contact with the skin, it is 0,5 µg/cm^2/week or less.

European Standard EN 1811, *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*, is used to determine whether such articles are in compliance with the Regulation (EC) No 1907/2006 of the European Parliament and of the Council (REACH), Annex XVII, entry no 27, as amended.

1 Scope

This document provides a screening test based upon the use of dimethylglyoxime for detecting the presence of nickel in articles that are inserted into pierced parts of the human body and those that are intended to come into direct and prolonged contact with the skin.

This screening test is suitable for manufacturers and importers as a qualitative method for detecting the presence of nickel in articles.

NOTE The reference method for the measurement of nickel release is EN 1811, or for spectacle frames and sunglasses, 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 ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological 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/

4 Principle

The method is based on the formation of a red coloured metal complex when nickel ions react with a solution of dimethylglyoxime in the presence of ammonia.

In order to increase the sensitivity of the screening test, pre-treatment with artificial sweat and heat is used to induce corrosion of the surface, simulating the influence of sweat when the article is in contact with the skin. This screening method provides a result in a short time.

5 Reagents

All reagents should be of *pro analysis* grade or better.

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

5.2 Ammonia solution, 10 % ammonia.

This solution may be prepared from a more concentrated ammonia solution, for example, one with a mass fraction of 24 % or 30 % NH_3.

5.3 Sodium chloride, NaCl.

5.4 DL-Lactic acid, $CH_3CHOHCOOH$, > 88 % (m/m).

5.5 Urea, $CO(NH_2)_2$.

5.6 Dimethylglyoxime, $C_4H_8N_2O_2$, 99 % or test strips (6.8) containing dimethylglyoxime.

5.7 Ethanol, C_2H_5OH, > 95 % (V/V).

5.8 Sodium hydroxide, pellets, purity ≥ 98 %, anhydrous.

5.8.1 Preparation of 1 M sodium hydroxide solution:

Weigh 4 g ± 0,01 g sodium hydroxide (5.8), transfer to a 100 ml beaker and add 50 ml deionized water (5.1). Stir and cool to room temperature. Transfer the solution to a 100 ml volumetric flask and make up to volume with deionized water (5.1).

Alternatively, it is possible to use 1 M ready-to-use solution.

5.8.2 Preparation of 0,1 M sodium hydroxide solution:

Transfer 25 ml of the 1 M sodium hydroxide solution (5.8.1) to a 250 ml volumetric flask and make up to volume with deionized water (5.1).

Alternatively, it is possible to use 0,1 M ready-to-use solution.

5.9 Hydrochloric acid, 32 % (m/m).

5.9.1 Preparation of 0,1 M hydrochloric acid:

Transfer 1 ml hydrochloric acid (5.9) to a 100 ml volumetric flask and make up to volume with deionized water (5.1).

Alternatively, it is possible to use 0,1 M ready-to-use solution.

5.10 Sodium dodecyl sulfate or sodium alkyllauryl sulfate or another surface-active anionic detergent.

Additionally, the following reagents are required for the test with dithiooxamide (7.3.8):

5.11 Dithiooxamide, $C_2H_4N_2S_2$.

5.12 Sodium acetate trihydrate, $C_2H_3NaO_2 \cdot 3H_2O$.

5.13 Acetic acid, glacial acetic acid, $C_2H_4O_2$.

6 Apparatus

6.1 Appropriate laboratory apparatus.

6.2 Flat-bottomed dish, made of glass or another non-metallic material. Not required for the pre-test.

6.3 Graduated pipette, volume 50 µl.

6.4 Cotton-wool-tipped sticks (white cotton wool on a white stick).

6.5 pH-meter.

6.6 Laboratory oven, adjustable to a temperature of 50 °C ± 3 °C. Required for the laboratory test (7.3.5).

6.7 Abrasive paper, between grade 600 and 1200 inclusive.

6.8 Dimethylglyoxime test strips for detection of nickel ions (optional).

6.9 Balance, with an accuracy of 0,01 g.

7 Procedure

7.1 Preparation of solutions

7.1.1 Dimethylglyoxime test solution

The 0,8 % dimethylglyoxime alcoholic solution is prepared as follows:

Weigh 0,8 g ± 0,05 g of dimethylglyoxime (5.6) into a 100 ml volumetric flask, dissolve in ethanol (5.7) and make up to volume.

7.1.2 Artificial sweat solution

Fresh artificial sweat solution shall be prepared every day that testing is being performed.

The artificial sweat solution consists of deionized water (5.1) and the following:

— 0,5 % (m/m) sodium chloride (5.3);

— 0,1 % (m/m) lactic acid (5.4);

— 0,1 % (m/m) urea (5.5); and

— 1 M (5.8.1) and 0,1 M (5.8.2) sodium hydroxide solution.

The artificial sweat solution is prepared as follows:

Fill a 1 000 ml beaker with 900 ml deionized water (5.1). Add 1 g ± 0,01 g urea (5.5), 5 g ± 0,05 g sodium chloride (5.3) and 1 g ± 0,01 g lactic acid (5.4) and stir until all added reagents are dissolved.

Calibrate a pH meter using suitable calibration solutions according to the manufacturer's instructions.

Immerse the pH electrode into the artificial sweat solution and determine the pH. Slowly and carefully add 1 M sodium hydroxide (5.8.1) drop by drop until a pH of 5,5 is reached; subsequently, slowly and carefully add 0,1 M sodium hydroxide (5.8.2) with constant stirring until a stable pH of (6,5 ± 0,05) is reached.

Measure the pH 10 min after the last addition of 0,1 M sodium hydroxide (5.8.2) in order to ensure that the pH is still in the range of (6,5 ± 0,05).

Transfer the solution to a 1 000 ml volumetric flask and make up to volume with deionized water. Prior to use, make sure that the pH of the artificial sweat solution is in the range of (6,5 ± 0,05).

If it is necessary to reduce the pH of the solution to (6,5 ± 0,05) prior to the test, carefully add 0,1 M hydrochloric acid (5.9.1) drop by drop with constant stirring.

7.1.3 Dithiooxamide test solution

The 0,5 % dithiooxamide alcoholic solution is prepared as follows:

Weigh 0,5 g ± 0,05 g of dithiooxamide (5.11) into a 100 ml volumetric flask, dissolve in and make up to volume with ethanol (5.7).

7.1.4 Sodium acetate buffer solution

The pH 4,5 sodium acetate buffer solution is prepared as follows:

Weigh 5,6 g sodium acetate trihydrate (5.12) into a 10 ml volumetric flask, and add 2,4 ml glacial acetic acid (5.13), dissolve in and make up to volume with deionized water (5.1).

7.1.5 Degreasing solution

In a 1 000 ml volumetric flask, dissolve 5 g of an anionic surface-active agent, e.g. sodium dodecyl sulfate (5.10) or sodium alkyllauryl sulfate, in 1 000 ml deionized water (5.1). An appropriately diluted, neutral and commercially available detergent may be used.

7.2 Sample preparation — Cleaning of the surfaces to be tested

The surfaces to be tested are those inserted into pierced parts of the human body or intended to come into direct and prolonged contact with the skin. Gently swirl the article under test for 2 min in the degreasing solution (7.1.5) at room temperature. Subsequently, rinse thoroughly with deionized water (5.1) and dry using absorbent paper. After degreasing, the articles under test shall be handled with plastic or ceramic forceps or with clean protective gloves.

This cleaning stage is intended to remove extraneous grease and skin secretions, but not any protective coatings. However, it will also substantially remove any nickel salts present on the surface of the tested article. If it is required to detect the presence of surface contamination by nickel, this cleaning stage should be omitted.

An article may be composed of parts made of different materials, each of which may require testing if they are intended to come into direct and prolonged contact with the skin.

Secondary contamination by articles containing nickel (paper-clips, rivets, coins) may give false-positive results if they come into contact with test articles, surfaces, reagents or the hands. This possibility should be eliminated by avoiding nickel-containing articles and by washing the hands before performing the tests.

7.3 Test procedure

7.3.1 The person carrying out the tests shall have normal colour vision in regard to being able to detect red and pink colours.

7.3.2 Check the reactivity of the dimethylglyoxime test solution (7.1.1) and dithiooxamide test solution (7.1.3) by applying the test to a surface that is known to be nickel-releasing, e.g. a cupro-nickel coin.

7.3.3 Conduct the pre-test (7.3.4) and/or the laboratory test (7.3.5).

a) If the pre-test is conducted and a positive result (red colouration) is observed, further testing is unnecessary unless the presence of iron is suspected.

b) If the pre-test is conducted and a negative result (absence of red colouration) is observed, conduct the laboratory test. The test shall not be performed on the area that has already been tested with the pre-test (7.3.4).