



SLOVENSKI STANDARD

SIST CR 12471:2004

01-januar-2004

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Screening tests for nickel release from alloys and coatings in items that come into direct and prolonged contact with the skin

Schnelltest für die Nickelabgabe aus Legierungen und Auflagen auf Gegenständen, die mit der Haut in direkte und länger andauernde Berührung kommen

Méthode de tri pour la libération du nickel des alliages et revêtements présents sur les articles de consommation entrant en contact direct et prolongé avec la peau

Ta slovenski standard je istoveten z: CR 12471:2002

ICS:

39.060

Nakit

Jewellery

SIST CR 12471:2004

en

ICS 39.060

English version

Screening tests for nickel release from alloys and coatings in items that come into direct and prolonged contact with the skin

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This CEN Report was approved by CEN on 13 April 2002. It has been drawn up by the Technical Committee CEN/TC 283.

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Foreword

This document (CR 12471:2002) has been prepared by Technical Committee CEN/TC 283 "Precious metals - Applications in jewellery and associated products", the secretariat of which is held by UNI.

Introduction

This document has been developed as a simple, quick and inexpensive means for qualitatively testing for nickel release from (mainly) consumer items. It has particular relevance in relation to allergic contact dermatitis due to nickel allergy. The tests described are quick and easy to conduct and two of them are suitable for application outside of the laboratory.

European Parliament and Council Directive 94/27/EC (OJ No. L 188 of 22.7.94) has set a nickel release rate threshold of $0,5 \mu\text{g}/\text{cm}^2/\text{week}$. Although the present document will give information concerning nickel release, results obtained from its application do not constitute confirmation or otherwise of compliance with the directive. In order to show compliance with the directive, it is necessary that items are tested in accordance with European Standards EN 1810, EN 1811 or EN 12472, as appropriate, depending upon the nature of the item to be tested.

1 Scope

This document presents a screening procedure, based primarily on the use of dimethylglyoxime, for the detection of nickel release from items that come into direct and prolonged contact with the skin.

The tests provide qualitative, not quantitative, results.

NOTE - EN 1811 specifies a quantitative reference test for the release of nickel.

2 Short description of the methods

The test methods are based on the formation of a coloured complex when nickel ions come into contact with dimethylglyoxime or dithiooxamide. In order to increase the sensitivity of the method, pre-treatment with artificial sweat and heat is used to induce corrosion of the surface, simulating the influence of sweat when the item is in contact with the skin. This screening method gives a result in a short time. The result is indicative and provides guidance when evaluating items for nickel release.

3 Reagents

All reagents shall be of *pro analysi* grade or better.

3.1 Deionized water

3.2 Ammonia solution, about 10 % (m/m) NH_3

NOTE - This solution may be prepared from a more concentrated ammonia solution; for example, one containing 24 % or 30 % (m/m) NH_3 .

3.3 Sodium chloride, NaCl

3.4 Lactic acid, $\text{CH}_3\text{CHOHCOOH}$, $\rho = 1,21 \text{ g/ml}$, > 88 %

3.5 Urea, $\text{CO}(\text{NH}_2)_2$

3.6 Dimethylglyoxime, $\text{C}_4\text{H}_8\text{N}_2\text{O}_2$, 99 %, or test strips for the detection of nickel, containing dimethylglyoxime or other colorimetric reagent(s) with approximately equivalent selectivity and sensitivity to nickel.

3.7 Ethanol, $\text{C}_2\text{H}_5\text{OH}$, > 95 %

3.8 Hydrogen peroxide, H_2O_2 , approximately 30 % (m/V) solution (100 volume). (Not required for the pre-test, 5.3.4 or the field test, 5.3.6)

3.9 Dithiooxamide (rubeanic acid), $\text{C}_2\text{H}_4\text{N}_2\text{S}_2$. (Not required for the pre-test or the field test)

3.10 Sodium acetate trihydrate, $\text{C}_2\text{H}_3\text{NaO}_2 \cdot 3\text{H}_2\text{O}$. (Not required for the pre-test or the field test)

3.11 Acetic acid, glacial, $\text{C}_2\text{H}_4\text{O}_2$. (Not required for the pre-test or the field test)

4 Equipment

4.1 Flat-bottomed dish, made of glass or other non-metallic material. (Not required for the pre-test)

4.2 Thermometer, $(0^\circ\text{C} - 100^\circ\text{C}) \pm 1^\circ\text{C}$

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4.3 Pasteur pipette or, for the field test, a drop-dispensing bottle capable of dispensing drops with an approximate volume of 50 µl.

4.4 Cotton-wool-tipped sticks (white)

4.5 pH meter or narrow-range pH paper

4.6 Laboratory oven, capable of maintaining a temperature of $50^{\circ}\text{C} \pm 3^{\circ}\text{C}$ (required for the laboratory test, 5.3.5)

4.7 Heating/drying apparatus (required only for the field test)

4.7.1 Hand-held hair dryer, or

4.7.2 Apparatus consisting of a light bulb, reflector type, (IEC 887: R80; 60 W to 100 W) mounted vertically downwards in a lamp holder positioned above a flat base, such that the distance of the face of the bulb above the flat base may be varied between approximately 40 mm and 120 mm.

5 Procedure

5.1 Preparation of solutions

Unless otherwise specified, the following solutions are stable for 6 months at temperatures below 25°C .

5.1.1 Dimethylglyoxime, 0,8% (m/V) alcoholic solution. Weigh $0,8 \text{ g} \pm 0,05 \text{ g}$ of dimethylglyoxime (3.6), dissolve and make up to 100 ml in ethanol (3.7).

5.1.2 Artificial sweat. Transfer $1 \text{ g} \pm 0,02 \text{ g}$ urea (3.5), $5 \text{ g} \pm 0,1 \text{ g}$ sodium chloride (3.3) and $1,13 \text{ g} \pm 0,02 \text{ g}$ ($940 \text{ µl} \pm 20 \text{ µl}$) lactic acid (3.4) into a 2-litre beaker. Add 1000 ml deionized water (3.1) and stir. Using a pH meter or pH paper (4.5), adjust the pH to $6,5 \pm 0,2$ by the dropwise addition of ammonia solution (3.2) with stirring. Artificial sweat is stable for 6 months when kept in the dark below 8°C in a closed container.

NOTE - The use of a pH meter is preferable to the use of narrow-range pH paper.

5.1.3 Dithiooxamide, 0,5% (m/V) alcoholic solution. Weigh $0,5 \text{ g} \pm 0,05 \text{ g}$ of dithiooxamide (3.9), dissolve and make up to 100 ml in ethanol (3.7).

5.1.4 Sodium acetate buffer solution, pH 4.5. Weigh 5,6 g sodium acetate trihydrate (3.10) and add 2,4 ml glacial acetic acid (3.11). Dissolve and make up to 10 ml with water.

5.2 Sample preparation

5.2.1 The surfaces to be tested are those that come into direct and prolonged contact with the skin. The surface(s) of the item to be tested shall be cleaned with ethanol (3.7) using a cotton-wool-tipped stick (4.4).

NOTE 1 - This cleaning stage is intended to remove extraneous grease and skin secretions due to handling, but not any protective coatings. However, it will also substantially remove any nickel salts present on the surface of the test item. If there is a requirement to detect the presence of surface contamination by nickel, this cleaning stage should be omitted.

NOTE 2 - An item may be composed of parts made of different materials, each of which may require testing if they come into direct and prolonged contact with the skin.

NOTE 3 - Contamination by objects containing nickel (paper-clips, rivets, coins, etc.) may give false positive results if such objects come into contact with test items, surfaces, reagents or the hands. This possibility should be eliminated by avoidance of nickel-containing objects, washing the hands before performing the tests and performing blank tests.