



SLOVENSKI STANDARD

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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

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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

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

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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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Contents

	page
Foreword.....	3
Introduction	4
1 Scope	5
2 Short description of the methods	5
3 Reagents.....	5
4 Equipment.....	5
5 Procedure	6
5.1 Preparation of solutions.....	6
5.2 Sample preparation	6
5.3 Test methods.....	7
6 Interpretation of results.....	8
6.1 Positive result.....	8
6.2 Negative result	9
6.3 Uncertain result.....	9
7 Test report	9
Bibliography	10

[SIST CR 12471:2004](https://standards.iteh.ai/catalog/standards/sist/029afd09-ba1a-4f8f-a32c-bd391d2507e2/sist-cr-12471-2004)

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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.

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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.

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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) NH₃

NOTE - This solution may be prepared from a more concentrated ammonia solution; for example, one containing 24 % or 30 % (m/m) NH₃.

3.3 Sodium chloride, NaCl

3.4 Lactic acid, CH₃CHOHCOOH, ρ = 1,21 g/ml, > 88 %

3.5 Urea, CO(NH₂)₂

3.6 Dimethylglyoxime, C₄H₈N₂O₂, 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, C₂H₅OH, > 95 %

3.8 Hydrogen peroxide, H₂O₂, 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), C₂H₄N₂S₂. (Not required for the pre-test or the field test)

3.10 Sodium acetate trihydrate, C₂H₃NaO₂·3H₂O. (Not required for the pre-test or the field test)

3.11 Acetic acid, glacial, C₂H₄O₂. (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°C - 100°C) ± 1°C

CR 12471:2002 (E)

- 4.3** Pasteur pipette or, for the field test, a drop-dispensing bottle capable of dispensing drops with an approximate volume of 50 μ l.
- 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 $\mu\text{l} \pm 20 \mu\text{l}$) 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.

5.2.2 The presence of coatings, such as paint, lacquer and electroplate, can prevent the detection of a nickel-releasing substrate. Therefore, abrasion of the surface prior to testing for nickel-release should be considered, especially if a negative result (see 6.2) has been obtained on the unabraded item. This may be achieved using emery paper, emery board or other nickel-free abrasive material.

CAUTION This procedure is likely to damage the item.

NOTE - If a positive result is obtained in these circumstances, testing in accordance with EN 12472 is required to check compliance with EU Directive 94/27/EC.

5.3 Test methods

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

5.3.2 Check the reactivity of the test solutions, as necessary, by applying the test to a known nickel-releasing surface, e.g. a cupro-nickel coin.

5.3.3 Either conduct the pre-test (5.3.4) and/or the laboratory test (5.3.5) or the field test (5.3.6).

- 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 (see 5.3.8);
- b) If the pre-test is conducted and a negative result (absence of red colouration) is observed, conduct the laboratory test (5.3.5) or the field test (5.3.6).

NOTE - The pre-test only gives a positive result in the presence of nickel salts or where nickel is available for release from an item without prior corrosion (see clause 2).

5.3.4 Pre-test for nickel release: moisten a cotton-wool-tipped stick (4.4) with one or two drops of dimethylglyoxime solution (5.1.1) and one drop of ammonia solution (3.2). Check that there is no discolouration. Rub firmly the cotton wool tip for 15 s against the surface to be tested. View the stick against a white background. The appearance of a red colour, from light pink to strong cerise, indicates nickel release.

5.3.5 Laboratory test: place the test item on a dish (4.1) and pre-heat it to approximately 50°C. Using a Pasteur pipette, transfer one drop of artificial sweat (5.1.2) onto the surface to be tested. Dry the item in the laboratory oven (4.6) until its surface is completely dry. The temperature shall be 50°C ± 3°C. Drying time will be about 15 min. Perform the test for nickel release (5.3.7, 5.3.8 or 5.3.9, as appropriate).

5.3.6 Field test: place the test item on a dish (4.1) and using a Pasteur pipette or a drop-dispensing bottle (4.3) put one drop of artificial sweat (5.1.2) on the surface to be tested. Dry the surface completely with the hair dryer (4.7.1). If necessary, keep the item in place using any suitable means. Place a thermometer (4.2) close to the test item. The temperature shall not exceed 50°C.

Alternatively, the test item can be dried by the heat from an electric light bulb using the equipment described in 4.7.2. The light bulb is switched on and allowed to warm up. Raise or lower the lamp holder to obtain a constant temperature of approximately 50°C just above the surface on the flat base directly beneath the lamp. Place the test item on a dish directly beneath the centre of the lamp. Pre-heat the item for about 10 min then put one drop of artificial sweat on the surface to be tested. Allow the surface to dry completely.

Perform the test for nickel release (5.3.7).