BRITISH STANDARD

BS EN 1810:1998

Body-piercing post assemblies — Reference test method for determination of nickel content by flame atomic absorption spectrometry

The European Standard EN 1810:1998 has the status of a British Standard

ICS 39.060

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National foreword

This British Standard is the English language version of EN 1810:1998.

The UK participation in its preparation was entrusted to Technical Committee STI/53, Jewellery, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 7 and a back cover.

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 November 1998

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English version

Body-piercing post assemblies — Reference test method for determination of nickel content by flame atomic absorption spectrometry

Ensembles de perçage de parties du corps — Méthode d'essai de référence pour la détermination de la teneur en nickel par spectrométrie d'absorption atomique de flamme

Stecker, die durch Teile des Körpers gestochen werden — Referenzprüfverfahren zur Bestimmung des Nickelgehalts durch Atomabsorptionsspektrometrie

This European Standard was approved by CEN on 10 April 1998.

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Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This European Standard 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.

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of an identical text or by endorsement, at the latest by November 1998, and conflicting national standards shall be withdrawn at the latest by November 1998.

Annex A is informative.

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Introduction

This method is used to determine whether the nickel content in post assemblies which are inserted into pierced ears and other pierced parts of the human body during epithelization of the wound caused by piercing, whether subsequently removed or not, is equal to or less than 0,05 % (*m/m*).

1 Scope

This European Standard specifies a method for the determination of nickel in aluminium, titanium, copper, silver, gold and their alloys and in steels by flame atomic absorption spectrophotometry. The method is primarily suitable when the nickel content of a sample lies between 0,03 % and 0,07 % (*m/m*).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to, or revisions of, any of these publications apply to this standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

prEN 1811, *Precious metals — Reference test method for release of nickel from products intended to come into direct and prolonged contact with the skin.*

3 Principle

A test portion is dissolved in an acid medium. The resulting solution is atomized into an air-acetylene flame of the atomic absorption spectrophotometer, and the absorption of the resonance energy of nickel (232,0 nm) is compared with that of calibration solutions.

4 Reagents

All reagents shall be of recognized pro-analysis, p.a., grade or better, and shall be free of nickel.

NOTE Only those reagents appropriate to the matrices under examination are required.

WARNING. Concentrated acids are hazardous. Wear safety glasses or goggles and carry out dissolution of metals in a well-ventilated fume cupboard.

4.1 *Deionized water*, specific conductivity maximum 1 µS/cm.

4.2 *Hydrochloric acid*, 38 % (*m/m*), ρ = 1,19 g/ml.

4.3 *Dilute hydrochloric acid*, 20 % (*m/m*), ρ = 1,10 g/ml.

Carefully add 125 ml of hydrochloric acid (see **4.2**) to approximately 110 ml of deionized water in a 500 ml beaker. Stir and cool to room temperature. Transfer the solution into a 250 ml volumetric flask and make up to volume with deionized water.

4.4 *Hydrogen peroxide solution*, 30 % (*m/m*).

4.5 *Hydrofluoric acid*, 40 % (*m/m*), ρ = 1,13 g/ml.

4.6 *Nitric acid*, 65 % (*m/m*), ρ = 1,40 g/ml.

4.7 *Dilute nitric acid*, 33 % (*m/m*), ρ = 1,20 g/ml.

Carefully add 44 ml of nitric acid (see **4.6**) to approximately 40 ml of deionized water in a 250 ml beaker. Stir and cool to room temperature. Transfer the solution into a 100 ml volumetric flask and make up to volume with deionized water.

4.8 *Perchloric acid*, 60 % (*m/m*), ρ = 1,54 g/ml.

4.9 *Aqua regia*

Cautiously mix 120 ml of hydrochloric acid (see **4.2**) and 40 ml of nitric acid (see **4.6**). This mixture shall be prepared immediately before use.

4.10 *Solution of hydrochloric acid and nitric acid*

Cautiously add 50 ml of hydrochloric acid (see **4.2**) to 40 ml of deionized water, with stirring. Add 20 ml of nitric acid (see **4.6**) and mix well. Prepare this solution immediately before use.

4.11 *Solution of nitric acid and perchloric acid*

Cautiously mix 5 ml of nitric acid (see **4.6**) and 40 ml of perchloric acid (see **4.8**). Transfer the solution into a 50 ml volumetric flask and make up to volume with deionized water.

4.12 *Nickel stock solution*, (1,000 0 ± 0,000 2) g/l.

Prepare nickel stock solution, for example, by using a certified standard solution ampoule.

4.13 *Aluminium matrix*, 10 g/l stock solution.

Weigh, to the nearest 0,001 g, 2,000 g of aluminium (containing less than 0,000 5 % nickel) and transfer to a 125 ml conical flask. Add, in small portions, 60 ml of dilute hydrochloric acid (see **4.3**) and warm gently until complete dissolution. Add a few drops of hydrogen peroxide (see **4.4**) and heat for about 2 min to remove excess hydrogen peroxide. Cool to room temperature, transfer the solution into a 200 ml volumetric flask and make up to volume with deionized water.

4.14 *Titanium matrix*, 10 g/l stock solution.

Weigh, to the nearest 0,001 g, 2,000 g of titanium (containing less than 0,000 5 % nickel) and transfer to a 125 ml conical flask. Add, in small portions, 60 ml of hydrofluoric acid (see **4.5**) and warm gently until complete dissolution. Cool to room temperature, transfer the solution into a 200 ml volumetric flask and make up to volume with deionized water. Use laboratory apparatus appropriate for hydrofluoric acid, e.g. polytetrafluoroethylene (PTFE).

4.15 *Copper matrix*, 10 g/l stock solution.

Weigh to the nearest 0,001 g, 2,000 g of copper (containing less than 0,000 5 % nickel) and transfer to a 125 ml conical flask. Add, in small portions, 40 ml of dilute hydrochloric acid (see **4.3**) and 10 ml of hydrogen peroxide (see **4.4**). Cool until the violent reaction has ceased. When completely dissolved, heat the solution to boiling and continue boiling for approximately 1 min to remove excess hydrogen peroxide, then cool to room temperature. Transfer the solution into a 200 ml volumetric flask and make up to volume with deionized water.

4.16 *Silver matrix*, 10 g/l stock solution.

Weigh, to the nearest 0,001 g, 2,000 g of silver (containing less than 0,000 5 % nickel) and transfer to a 125 ml conical flask. Add 60 ml of dilute nitric acid (see **4.7**) and warm gently until complete dissolution. Heat, without boiling, until evolution of nitrogen oxide fumes ceases. Cool to room temperature, transfer the solution into a 200 ml volumetric flask and make up to volume with deionized water.

4.17 *Gold matrix*, 10 g/l stock solution.

Weigh, to the nearest 0,001 g, 2,000 g of gold (containing less than 0,000 5 % nickel) and transfer to a 125 ml conical flask. Add 80 ml aqua regia (see **4.9**) and warm. Add aqua regia, as required, until complete dissolution. Note the amount of aqua regia used. Cool to room temperature, transfer the solution into a 200 ml volumetric flask and make up to volume with deionized water.

4.18 *Iron matrix*, 10 g/l stock solution.

Weigh, to the nearest 0,001 g, 2,000 g of iron (containing less than 0,000 5 % nickel) and transfer to a 125 ml conical flask. Add 50 ml of the hydrochloric acid-nitric acid solution (see **4.10**) and warm until complete dissolution. Cool to room temperature and filter if necessary. Transfer the solution into a 200 ml volumetric flask and make up to volume with deionized water.

5 Apparatus

Normal laboratory equipment, and:

5.1 *Balance*, capable of weighing in grams to four decimal places.

5.2 *An atomic absorption spectrophotometer*, a nickel hollow-cathode lamp and supplies of air and acetylene (free from water, oil and nickel) sufficiently pure to give a steady clear flame. The apparatus shall, after optimization according to **6.5**, meet the performance criteria given in **5.2.1** to **5.2.3**. Additionally, it is recommended that the apparatus conforms to the performance criteria given in **5.2.4**.

5.2.1 *Minimum precision*

The standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution (see **6.4**) shall not exceed 1,0 % of the mean absorbance of the most concentrated calibration solution.

The standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero calibration solution) (see **6.4**) shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

5.2.2 *Characteristic concentration*

The characteristic concentration for nickel in a matrix similar to the final test solution shall be no greater than 0,10 mg/l of nickel at 232,0 nm.

5.2.3 *Limit of detection*

The limit of detection shall be considered as twice the standard deviation of 10 measurements of the absorbance of a solution containing nickel at a concentration level selected to give an absorbance just above that of the zero calibration solution.

The limit of detection of nickel in a matrix similar to the final test solution shall be better than 0,15 mg/l at 232,0 nm.

5.2.4 *Graph linearity*

The slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) should be not less than 0,7 times the value of the slope for the bottom 20 % of the concentration range.

In this standard, the calibration graph is drawn at the lower part of the optimum working range, normally defined as 20 to 125 times the characteristic concentration.

NOTE The performance criteria given in **5.2.1** to **5.2.4** are based on provisions given in ISO/CD 13204.

6 Procedure

6.1 Detection of surface layers

Samples shall be subjected to the nickel release test according to prEN 1811 to detect nickel contamination on the surface, and abraded with an abrasive material to check visually for the presence of coatings. When checking for the presence of nickel on the surface, samples shall not be degreased prior to analysis.

NOTE If coatings are suspected, X-ray analysis may assist recognition.

6.2 Composition of sample

Identify the approximate composition of the sample by any appropriate means, for example, by X-ray fluorescence spectrometry (XRF) or other semi-quantitative method. This is necessary in order to classify the sample as an aluminium, titanium, copper, silver, gold or steel alloy.

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6.3 Sample preparation

6.3.1 *Aluminium*

Weigh, to the nearest 0,001 g, 0,125 g of the test sample and transfer to a 25 ml beaker. Add 5 ml of dilute hydrochloric acid (see **4.3**) and warm gently until complete dissolution. Add two drops of hydrogen peroxide solution (see **4.4**) and heat for about 2 min to remove excess hydrogen peroxide. Filter if necessary. Cool, transfer the solution to a 25 ml volumetric flask and make up to volume with deionized water.

6.3.2 *Titanium*

Weigh, to the nearest 0,001 g, 0,125 g of the test sample and transfer to a 25 ml beaker. Add, in small portions, 5 ml of hydrofluoric acid (see **4.5**) and warm gently until complete dissolution. Cool to room temperature, transfer the solution to a 25 ml volumetric flask and make up to volume with deionized water. Use laboratory apparatus appropriate for hydrofluoric acid, e.g. polytetrafluoroethylene (PTFE).

6.3.3 *Copper*

Weigh, to the nearest 0,001 g, 0,125 g of the test sample and transfer to a 25 ml beaker. Add 3 ml of dilute hydrochloric acid (see **4.3**) and 1 ml of hydrogen peroxide solution (see **4.4**). Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat the solution to boiling and continue boiling for approximately 1 min to remove excess hydrogen peroxide. Cool to room temperature, transfer the solution to a 25 ml volumetric flask and make up to volume with deionized water.

6.3.4 *Silver*

Weigh, to the nearest 0,001 g, 0,125 g of the test sample and transfer to a 25 ml beaker. Add 5 ml of dilute nitric acid (see **4.7**) and warm gently until complete dissolution. Heat, without boiling, until evolution of nitrogen oxide fumes ceases. Cool to room temperature, transfer the solution to a 25 ml volumetric flask and make up to volume with deionized water.

6.3.5 *Gold*

Weigh, to the nearest 0,001 g, 0,125 g of the test sample and transfer to a 25 ml beaker. Add 5 ml aqua regia (see **4.9**) and warm until complete dissolution. If more acid was used in order to prepare the gold matrix solution, add an appropriate proportion of the excess here. Cool to room temperature, transfer the solution to a 25 ml volumetric flask and make up to volume with deionized water.

6.3.6 *Steels*

Weigh, to the nearest 0,001 g, 0,125 g of the test sample and transfer to a 25 ml beaker. Add 3 ml of the hydrochloric acid–nitric acid solution (see **4.10**) and warm until complete dissolution. If complete dissolution is not achieved, add 2 ml of the nitric acid–perchloric acid solution (see **4.11**) and evaporate until dense white fumes of perchloric acid appear. Continue fuming for approximately 1 min at such a temperature as to maintain a steady reflux of white perchloric acid fumes on the walls of the beaker. Allow to cool, add 5 ml of water and heat gently to dissolve salts. Cool and filter if necessary. Transfer the solution to a 25 ml volumetric flask and make up to volume with deionized water.

6.4 Preparation of the calibration solutions

To a series of six 50 ml volumetric flasks transfer 0 µl (the zero calibration solution), 75 µl, 100 µl, 125 µl, 150 µl and 175 µl of the nickel stock solution (see **4.12**), using a micro-pipette having an accuracy of ±1 %. Then add to each flask a volume, V_r, of one or more of the matrix solutions (see **4.13** to **4.18**), corresponding to the nature and proportion of the main element(s) of the test sample, given by the equation:

$$V_r = \frac{25 \times W}{100}$$

where

V_r is the volume of matrix solution, in millilitres;

W is the approximate content of an element in the sample (see **6.2**), expressed as a percentage by mass.

Make each flask up to volume with deionized water and mix.

The calibration solutions thus obtained contain 0, 1,5, 2,0, 2,5, 3,0 and 3,5 micrograms of nickel per millilitre (µg/ml).

NOTE By extending the range of the calibration solutions to include those with nickel concentrations of 0,5 µg/ml and 1,0 µg/ml (obtained by diluting 25 µl and 50 µl of the nickel stock solution to 50 ml), this method can be used to determine nickel contents down to 0,01 % (*m/m*).

6.5 Optimization of the atomic absorption spectrometer settings

Follow the manufacturer's instructions for preparing the instrument for use.

Set the lamp current, the wavelength and gas flows. Light the burner and spray deionized water until the absorbance has stabilized.

Adjust the flame characteristics and the burner height for nickel determination according to the manufacturer's instructions, taking into account the actual matrix. Spraying alternately the calibration solution of highest concentration and the zero calibration solution, adjust the gas flow and burner position (horizontally, vertically and rotationally) until the difference in absorbance between the calibration solutions is at a maximum.

Check that the spectrometer is set accurately to the required wavelength, 232,0 nm.

Select a damping setting or integration time to give a signal steady enough to fulfil the precision requirements of **5.2.1**. Evaluate the performance criteria (see **5.2.1** to **5.2.3**) to ensure that the instrument is suitable for the determination.

6.6 Atomic absorption measurements

Set the absorbance value at zero using the zero calibration solution.

Set the scale expansion so that the most concentrated calibration solution gives nearly full scale deflection. Aspirate the calibration solutions in ascending order repeatedly until each gives the specified precision, indicating that the instrument has achieved stability. Select two calibration solutions (bracketing solutions), one having an absorbance just lower than the test solution and one just higher. Aspirate these first in ascending order, then in descending order, with the test solution as the middle solution in each case.

If the processing routine in an automatic instrument only accepts two calibration solutions, the two bracketing solutions should not be used for the primary calibration but should be analysed alternately with the test solution.

NOTE 1 This procedure is intended for highest accuracy on a single sample, as in referee analysis. For routine batch operation a modified procedure can be adopted whereby a series of samples is run in sequence, "sandwiched" between the complete range of calibration solutions in ascending and descending order. Alternatively, if only an approximate nickel content is required, it can be determined by the method of standard additions.

If the absorbance value of a sample solution lies under the lowest or over the highest absorbance values of the calibration solutions, it is not necessary to determine the exact nickel concentration.

NOTE 2 If the sample solution is too concentrated to be determined directly and an accurate result is required, the sample solution can be diluted, provided that the same matrix concentration as in the calibration solutions is maintained.

WARNING. Perchloric acid vapour can cause explosions in the presence of ammonia, nitrous fumes or organic matter in general. Keep the burner head clear of deposits caused by perchloric salts, etc. Ensure that the spray system and drainage system are washed free from perchloric acid after use.

7 Calculations

7.1 General

Calculations are only valid when the concentration of nickel in the sample solution (or diluted sample solution) lies between those of the lowest and the highest reference solutions.

7.2 Sample solution concentration

The nickel concentration of the sample solution, C_s, expressed in micrograms per millilitre (µg/ml), is given by the formula:

$$C_s = \frac{(C_2 - C_1) \times (A_s - A_1)}{(A_2 - A_1)} + C_1$$

where

C_1 is the concentration of nickel in the lower bracketing solution, in micrograms per millilitre (µg/ml);

C_2 is the concentration of nickel in the higher bracketing solution, in micrograms per millilitre (µg/ml);

A_s is the absorbance of the sample solution;

A_1 is the absorbance of the lower bracketing solution;

A_2 is the absorbance of the higher bracketing solution.

7.3 Nickel content of sample

The nickel content of the sample, X, expressed as percentage by mass, is given by the formula:

$$X = \frac{C_s \times V}{10^4 \times m}$$

where

C_s is the nickel concentration of the sample solution, in micrograms per millilitre (µg/ml);

V is the (final) dilution volume of the sample solution, in millilitres (ml);

m is the mass of the sample portion, in grams (g).

7.4 Final result

Report the arithmetical mean of acceptable determinations, rounded to the nearest 0,01 % for results below 1 % (*m/m*) nickel, and to the nearest 0,1 % for results above 1 % (*m/m*) nickel.

8 Precision

A planned trial of this method was carried out in 1993 and 1994 according to ISO 5725, involving seven laboratories and six different materials. Each laboratory made four determinations of each material. All the materials had a nickel content near the limit value of 0,05 % nickel.

8.1 Repeatability

Repeatability standard deviation, s_r = 0,002 1 % (*m/m*).

Repeatability, r = 0,005 9 % (*m/m*).

The difference between two single results found on identical test material by one analyst using the same apparatus within a short time interval should exceed the repeatability, r, on average not more than once in 20 cases, in normal and correct operation of the method.

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8.2 Reproducibility

Reproducibility standard deviation, $s_R = 0{,}004\,4$ % (m/m).

Reproducibility, $R = 0{,}012$ % (m/m).

The difference between two single and independent results found by two operators working in different laboratories on identical test material should exceed the reproducibility, R, on average not more than once in 20 cases, in normal and correct operation of the method.

9 Test report

The test report for each determination shall include at least the following information:

a) identification of the sample, including source, date of receipt, form;

b) sampling procedure;

c) a reference to this standard;

d) whether any surface layers have been detected;

e) the result of the nickel release test to detect the presence of surface nickel;

f) nickel content expressed as described in clause **7**;

g) if relevant, deviations from this standard method;

h) any unusual features observed during the determination;

i) date of test;

j) identification of the laboratory carrying out the analysis;

k) signatures of laboratory manager and operator.

Annex A (informative)

Bibliography

ISO 5725, *Accuracy (trueness and precision) of measurement methods and results.*

ISO/CD 13204-1, *Guidelines for the use of flame atomic absorption spectrometry (FAAS) — General guidelines.*

ISO/CD 13204-2, *Guidelines for the use of flame atomic absorption spectrometry (FAAS) — Guidelines for routine methods.*

ISO/CD 13204-3, *Guidelines for the use of flame atomic absorption spectrometry (FAAS) — Guidelines for referee methods.*

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