



**SLOVENSKI STANDARD**  
**oSIST prEN 10179:2023**  
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**Jekla - Določevanje dušika (v sledih) v jeklih - Spektrofotometrična metoda**

Steels - Determination of nitrogen (trace amounts) - Spectrophotometric method

Stähle - Bestimmung von Stickstoff (Spurengehalte) - Photometrisches Verfahren

Aciers - Détermination de l'azote (à l'état de traces) - Méthode spectrophotométrique

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English Version

## Steels - Determination of nitrogen (trace amounts) - Spectrophotometric method

Aciers - Détermination de l'azote (à l'état de traces) -  
Méthode spectrophotométrique

Stählen - Bestimmung von Stickstoff (Spuren Gehalte) -  
Photometrisches Verfahren

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 459/SC 2.

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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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## European foreword

This document (prEN 10179:2023) has been prepared by Technical Committee CEN/TC 459/SC 2 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 10179:1989.

In comparison with the previous edition, the following technical modifications have been made:

- Normative references: updated;
- Clause 3; Terms and definitions: added;
- References 5.10, 5.11, 5.14, 5.17 and 5.18: added;
- Clause 10: updated;

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**prEN 10179:2023 (E)****1 Scope**

This document specifies a spectrophotometric method for the determination of nitrogen in steels.

The method is primarily intended for the determination of total nitrogen in very low contents in non-alloy steels.

It can be used, however, for any low nitrogen ferrous alloy that is soluble in hydrochloric acid provided that the acid-resistant form of silicon nitride is not present. This highly resistant nitride has been found only in samples of silicon steels manufactured without aluminium addition and then only in sheet material.

The method is applicable to nitrogen contents from 0.000 5 % (by mass) to 0.005 % (by mass).

**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 648, *Laboratory glassware — Single-volume pipettes (ISO 648)*

EN ISO 1042, *Laboratory glassware — One-mark volumetric flasks (ISO 1042)*

EN ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition (ISO 14284)*

**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:

— IEC Electropedia: available at <https://www.electropedia.org/>

— ISO Online browsing platform: available at <https://www.iso.org/obp>

**4 Principle**

Dissolution of the test portion with hydrochloric acid and separation of the acid-insoluble residue by centrifugation.

Decomposition of the acid-insoluble residue by intense fuming with sulphuric acid and addition of the extract to the solution of the test portion containing the acid-soluble nitrogen.

Recovery of the total nitrogen as ammonia by steam distillation over sodium hydroxide.

Spectrophotometric measurement of the coloured complex produced by a reaction with indophenol blue.

## 5 Reagents

During the analysis use only reagents of recognized analytical grade and which are known to give a very low nitrogen blank. The same batch of each reagent shall be used for every test sample and blank determination in a given series of analysis.

All references to "water" relate to ammonia-free water. Ammonia-free water shall be prepared by passing distilled water through a cation exchange column (5.1). It is essential that the resin column shall be acid washed before use to ensure its conversion to the hydrogen form, H<sup>+</sup>. This is most conveniently done by passing 2 l of a solution of hydrochloric acid (5.2), diluted 1 + 9, through the column and then washing with water until freed from acid.

### 5.1 High purity iron or steel with a very low nitrogen content

High purity iron powder or very low nitrogen content mild steel [ $<0,001$  % (by mass) nitrogen] may be used for this purpose. It should be cleaned in the same way as the sample.

### 5.2 Hydrochloric acid, $\rho_{20}$ 1,19 g/ml approximately

### 5.3 Sulphuric acid, $\rho_{20}$ 1,84 g/ml approximately

Sulphuric acid supplied shall be tested individually and selected for a low content of combined nitrogen in any form (less than 0,5  $\mu\text{g/g}$ ). Nitrogen as ammonia will usually be the major source of contamination and this may be tested by normal methods, but nitrates may also be present and should be detected by using the method described below.

Add 6 ml of sulphuric acid (5.3) to 2 ml of water and cool to 60 °C. Add one drop of hydrochloric acid (5.2) and one drop of diphenylamine (5.8). No blue colour should appear.

NOTE Less than 0,1  $\mu\text{g/g}$  can be detected by this test.

### 5.4 Sulphuric acid, solution 1 + 4

Carefully add 40 ml of sulphuric acid (5.3) to 160 ml of water.

Allow to cool and mix.

### 5.5 Sodium hydroxide, 400 g/l

Dissolve 400 g of sodium hydroxide in water, dilute to 1 000 ml and mix.

This solution should be prepared in a polyethylene beaker (water-cooled if necessary) and stored in a polyethylene bottle.

### 5.6 Barium chloride, 100 g/l

Dissolve 100 g of barium chloride ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ) in water, dilute to 1 000 ml and mix.

### 5.7 Chromic-sulphuric acid

Dissolve 2 g of chromium trioxide ( $\text{CrO}_3$ ) in 50 ml of water and slowly add 100 ml of sulphuric acid (5.3) with constant stirring.

### 5.8 Diphenylamine, 1 g/l

To 25 ml of water, whilst stirring, cautiously add 75 ml of sulphuric acid (5.3) then add 0,1 g of diphenylamine, stir until dissolved and allow to cool.

**prEN 10179:2023 (E)****5.9 Phenol, 50 g/l**

Dissolve 50 g of phenol in water, transfer into a 1 000 ml volumetric flask, dilute to the mark with water and mix.

Store this solution out of direct sunlight in an amber coloured bottle.

**5.10 Potassium iodide (KI)**

**5.11 Glacial acetic acid**,  $\rho_{20}$  1,05 g/ml approximately

**5.12 Sodium hydroxide - sodium hypochlorite solution**

**5.12.1 Determination of chlorine in commercial sodium hypochlorite solution**

Transfer 10 ml of the sodium hypochlorite solution to be tested into a 250 ml volumetric flask, dilute to the mark with water and mix.

Transfer, 10 ml of this solution into a 100 ml conical beaker, add 2 g of potassium iodide (5.10) and 10 ml of glacial acetic acid (5.11). Titrate the liberated iodine using sodium thiosulphate (5.15) until the colour has almost disappeared. Add 2 ml of starch solution (5.16) and continue the titration until the blue colour has disappeared.

The chlorine content ( $w_{Cl}$ ) in per cent (m/v) is given by Formula (1)

$$w_{Cl} = 0,886 \times V_1 \quad (1)$$

where:

$V_1$  is the volume in millilitres of sodium thiosulphate (5.15) used in the titration.

**5.12.2 Calculation**

The volume,  $V_2$ , expressed in millilitres, of sodium hypochlorite solution necessary for the preparation of 1 l of sodium hydroxide - sodium hypochlorite solution (5.12) is given by Formula (2)

$$V_2 = \frac{2,1 \times 100 \times 70,91}{0,886 \times V_1 \times 74,44} = \frac{225,7}{V_1} \quad (2)$$

This volume is equivalent to 2,1 g of chlorine.

**5.12.3 Preparation of the solution 5.12**

Dissolve 25 g of sodium hydroxide in about 400 ml of water. Exactly add the calculated volume of sodium hypochlorite solution (5.12.2), dilute to 100 ml and mix.

Store this solution out of direct sunlight in an amber coloured bottle.

**5.13 Sodium pentacyanonitrosylferrate (sodium nitroprusside) ( $\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}] \cdot 2\text{H}_2\text{O}$ )**

Dissolve 0,1 g of sodium pentacyanonitrosylferrate in water, dilute to 100 ml and mix.

Prepare this solution freshly each day, store in an amber coloured bottle but do not use until at least 60 min. after the preparation.

**5.14 Sodium carbonate ( $\text{Na}_2\text{CO}_3$ )**

**5.15 Sodium thiosulphate**, 0,05 mol/l



Dissolve 24,821 g of sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) in water containing 0,1 g of sodium carbonate (5.14). Transfer into a 1 000 ml volumetric flask, dilute to the mark with water and mix.

#### 5.16 Starch, 5 g/l

Make a suspension of 0,5 g of starch in 10 ml of water. Add to 90 ml of boiling water. Allow to cool, dilute to 100 ml with water and mix.

#### 5.17 Ammonium chloride standard solution corresponding to 100 $\mu\text{g}$ of nitrogen per millilitre

Dissolve 0,382 g of ammonium chloride ( $\text{NH}_4\text{Cl}$ ), previously dried to constant weight at 105 °C, in water, transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 100  $\mu\text{g}$  of nitrogen.

#### 5.18 Ammonium chloride standard solution corresponding to 1 $\mu\text{g}$ of nitrogen per millilitre

Transfer 10 ml of the nitrogen standard solution (5.17) into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1  $\mu\text{g}$  of nitrogen.

## 6 Apparatus

### 6.1 Ordinary laboratory equipment

All volumetric glassware shall be grade A, in accordance with ISO 648 or ISO 1042, as appropriate.

### 6.2 Cation exchange column

A column packed with a sulphonated polystyrene cation exchange resin (see Figure 1).

### 6.3 Aluminium alloy heating block

An aluminium alloy block containing a number of sockets for the purpose of supporting 50 ml centrifuge tubes (see Figure 2) and heated by contact with the surface of a hotplate.

It is essential that the hot plate is capable of heating the test solutions to a temperature of at least 320 °C. This should be checked by inserting a thermometer in sulphuric acid under test conditions.

### 6.4 Steam distillation apparatus

The distillation unit illustrated in Figure 3 has been found suitable.

### 6.5 Centrifuge

An electrically driven centrifuge capable of taking tubes of 50 ml capacity and attaining a steady speed of 3500 r/min.

### 6.6 Spectrometer

Equipped to measure absorbance at a wavelength of 635 nm, with cells of 1 cm optical path length.

## 7 Sampling

Sampling shall be carried out in accordance with EN ISO 14284 or appropriate national standards for steels.

## 8 Procedure

### 8.1 General

Nitrogen determinations shall be carried out in a laboratory room reserved for the purpose and it is particularly important that ammonium salts and ammonia fumes are removed.

In addition, the glassware should be reserved for this particular determination only and should be cleaned with the chromic-sulphuric acid solution (5.7), and thoroughly washed with ammonia-free water before use.

### 8.2 Test portion

Sample millings or drillings should be cleaned in diethyl ether in order to remove any contaminations by oil.

If the drillings or millings have been stored in the laboratory they should be cleaned in hot water and dried immediately before determining the nitrogen content.

Weigh to the nearest 1 mg, approximately 1,0 g of the test sample.

### 8.3 Blank test

In parallel with the determination and following the same procedure, carry out a blank test on a 0,25 g (weighed to the nearest 1 mg) test portion of pure iron (5.1), using the same quantities of all reagents as used for the determination.

### 8.4 Preparation of the steam distillation apparatus

Ammonia-free water shall be used exclusively in the steam generating flask A (see Figure 3). Any water left over from previous day should be discarded and the flask rinsed before refilling.

Prepare the apparatus (see Figure 3) for use as follows:

- a) Fill the steam generator A with water, replace the stopper G, open the screw clip K, and raise the funnel plug D.
- b) Heat the steam generator and boil the water strongly, allowing the steam to escape for 30 min. via funnel C and the drain tube from steam trap B.
- c) Close the screw clip K, transfer 5 ml of sodium hydroxide solution (5.5) to the distillation vessel E via funnel C and lower the funnel plug D.
- d) Collect and discard about 50 ml of distillate and rinse the condenser tip with water.
- e) Place a 100 ml one-mark volumetric flask under the condenser and distil until about 35 ml of distillate have been collected.
- f) Switch off the heating source of the steam generator A, allowing the contents of the distillation vessel E to syphon into trap B, then open the screw clip K and discard the solution.

To the 100 ml one-mark volumetric flask, add 8 ml of phenol (5.9), 8 ml of the sodium hydroxide-sodium hypochlorite solution (5.12) and 2 ml of sodium pentacyanonitrosylferrate (5.13), whilst stirring after each addition. Dilute to the mark with water and mix.

To a second one-mark 100 ml volumetric flask, add about 35 ml of water and carry out the same additions of reagents as to the distillate. Dilute to the mark with water and mix.