SLOVENSKI STANDARD
oSIST prEN ISO 4945:2018

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Jeklo - Določevanje dušika - Spektrometrična metoda (ISO/DIS 4945:2017)

Steel - Determination of nitrogen - Spectrophotometric method (ISO/DIS 4945:2017)

Stahl - Bestimmung des Stickstoffgehalts - Spektralphotometrisches Verfahren (ISO/DIS 4945:2017)

Acier - Détermination de l'azote - Méthode spectrophotométrique (ISO/DIS 4945:2017)

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Steel — Determination of nitrogen — Spectrophotometric method

Acier — Détermination de l'azote — Méthode spectrophotométrique

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Foreword

I ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composion.*

This second edition cancels and replaces the first edition (ISO 4945:1977), which has been technically revised.

Steel — Determination of nitrogen — Spectrophotometric method

1 Scope

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

The method is applicable to determination of nitrogen mass fraction between 0,000 6 % and 0,050 % in low alloy steels and between 0,01 % and 0,050 % in high alloy steels.

However, the method is not applicable to samples containing silicon nitride.

The method doesn't apply to samples having silicon contents > 0,6 %

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.

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

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

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

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 http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

Dissolution of a test portion in hydrochloric acid.

Fuming of the acid-insoluble residue in sulphuric acid with potassium sulphate and copper(II) sulphate.

Distillation of the solution made alkaline with sodium hydroxide, and collection of ammonia in a receiver containing diluted sulphuric acid.

Formation of a blue-coloured complex between the ammonium ions and phenol in the presence of sodium hypochlorite and sodium pentacyanonitrosyIferrate(III) (sodium nitroprusside).

Spectrophotometric measurement of the complex at a wavelength of about 640 nm.

5 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and only freshly prepared grade 2 water as specified in ISO 3696.

5.1 Hydrochloric acid solution, 1 + 1

Add 500 ml of hydrochloric acid (ρ approximately 1,19 g/ml) to 500 ml of water and mix

5.2 Sulphuric acid, ρ approximately 1,84 g/ml, free from nitrogen compounds.

NOTE 1 It is preferable to prepare the acid by evaporating until the fume of sulphuric acid appears and continuing fuming for 20 - 30 min at the time of use.

5.3 Sulphuric acid solution, approximately 0,02 mol/l

Add 30 ml of sulphuric acid little by little into about 700 ml of water. After cooling, make up the volume to 1 litre with water, and mix. Dilute 40 ml of this solution to 1 litre with water and mix.

5.4 Sodium hydroxide, 500 g/l solution.

Dissolve 500 g of sodium hydroxide pellets in water. Cool and dilute to 1 litre with water and mix.

5.5 Sodium hydroxide 7.5 g/l solution.

Dilute 15 ml of sodium hydroxide (5.4) to 1 litre with water and mix.

5.6 Potassium sulphate

5.7 Copper(ll) sulphate pentahydrate, $CuSO_4 \cdot 5H_2O$.

5.8 Disodium hydrogenphosphate, 0,1 M solution.

Dissolve 36 g of disodium hydrogenphosphate dodecahydrate ($Na_2HPO_4 \cdot 12H_2O$) in water, make up the volume to 1 litre with water, and mix.

5.9 Sodium hypochlorite, NaClO, solution with approximately 0,3 % (mass fraction) of active chlorine.

Store this solution at a temperature less than 10 °C for three days or more.

5.10 Sodium phenoxide, solution.

Add, whilst stirring and cooling, 5 g of phenol to a mixture of 10 ml of a 250 g/l solution of sodium hydroxide and 80 ml of water. Make up the volume to 100 ml with water, and mix.

Prepare this solution at the time of use.

5.11 Disodium pentacyanonitrosylferrate(III), 0,25 g/l Solution.

Dissolve 10 g of disodium pentacyanonitrosylferrate(III) dihydrate (sodium nitroprusside dihydrate) $[Na_2\{Fe(CN)_5NO\}\cdot 2H_2O]$ in water, make up the volume to 1 litre with water, and mix.

Dilute 25 ml of this solution to 1 litre with water at the time of use.

5.12 Nitrogen, standard solution.

5.12.1 Stock solution, corresponding to 0,1 mg/ml of nitrogen.

Weigh, to the nearest 0,1 mg, 0,382 0 g of dry ammonium chloride, dissolve in water and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water, and mix.

1 ml of this stock solution contains 0,1 mg of nitrogen.

5.12.2 Standard solution, corresponding to 2 µg/ml of nitrogen.

Transfer 20,0 ml of the stock solution (5.12.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix.

Prepare this standard solution at the time of use.

1 ml of this standard solution contains 2 µg of nitrogen.

5.13 Methyl red, 0,1 g/l solution.

Dissolve 0.005 g of methyl red in ethanol, make up the volume to 50 ml with ethanol and mix.

6 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 648 and ISO 1042.

Ordinary laboratory apparatus and the following shall be used.

6.1 Spectrophotometer, suitable for measuring the absorbance of the solution at a wavelength of 640 nm with 10 mm optical cells.

6.2 Steam distillation apparatus, (see Annex A)

The apparatus consists of a steam generator, a distillation flask, a funnel, a condenser and a receiver. A trap can be used between the steam generator and the distillation flask.

Examples of recommended form of apparatus are illustrated in Figures A.1, A.2 and A.3.

The steam generator should be preheated to allow starting the distillation immediately after the introduction of the test solutions.

6.3 Hot water bath, suitable for boiling water.

7 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel .

8 Procedure

CAUTION — Carry out operations in a well ventilated room away from all sources of nitrogen compounds.

8.1 Test portion

Weigh, to the nearest 0,1 mg, a test portion of the sample in accordance with Table 1.

Table 1 — Mass of test portion

Expected nitrogen content % (mass fraction)	Test portion g
0,000 6 to 0,005 0	2,0
0,005 0 to 0,050	1,0

8.2 Blank test

Carry out a blank test simultaneously with the determination, following the same procedure and using the same quantities of all reagents as used for the determination.

8.3 Determination

8.3.1 Dissolution of the test portion

Place the test portion (8.1) in a 300 ml beaker or conical flask. Add 30 ml of hydrochloric acid (5.1), cover with a watch-glass or a funnel and heat until solvent action has apparently ceased.

Filter the solution through a filter paper (see NOTE 2) and collect the filtrate in a 300 ml beaker. Rinse the beaker or the conical flask with water, remove adherent particles with a rubber-tipped rod, and filter the rinsings through the same filter paper. Wash the filter paper with a minimum quantity of water. Keep the filtrate and the washings in the beaker. (This is sample solution S1.)

NOTE 2 A medium-texture filter paper is suitable for samples which do not contain fine nitrides. However, a close-texture filter paper is recommended if the sizes of nitrides are unknown. Vacuum filtration, using a nuclepore filter with pore size less than 0,2 µm, is necessary for samples which are known to contain fine nitrides such as boron nitride.

8.3.2 Treatment of the insoluble residue

Transfer the filter paper and the insoluble residue to the 500 ml conical flask and add 10 g of potassium sulphate (5.6), 1 g of copper(ll) sulphate pentahydrate (5.7) and 20 ml of sulphuric acid (5.2).

Heat gently until the water in the flask has evaporated, cover the flask with a watch-glass or a funnel and continue fuming (at 335 °C to 350 °C) for about 60 min to decompose the residue.

Cool to room temperature, add 50 ml of water and boil thoroughly for 5 min to remove sulphur dioxide from the solution. (This is sample solution S2.)

8.3.3 Steam distillation (see Figure A.1, A.2 or A.3)

To collect the distillate, transfer 5 ml of sulphuric acid (5.3) to a 100 ml volumetric flask with a ground neck and having a mark at 90 ml. Introduce the tapered tube extension of the condenser into the flask in such a manner that it is immersed in the 5 ml of sulphuric acid (5.3) solution.

Add 110 ml of sodium hydroxide solution (5.4) to the distillation flask (b) through the funnel (f) and rinse the funnel with a minimum quantity of water.

