
Steel — Determination of nitrogen — Spectrophotometric method

Acier — Détermination de l'azote — Méthode spectrophotométrique





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Published in Switzerland

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Foreword

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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

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

- the scope and applicable range has been expanded as a result of the interlaboratory test;
- addition of copper(II) sulfate pentahydrate for the treatment of the insoluble residue.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 the determination of nitrogen mass fraction between 0,000 6 % and 0,050 % in low alloy steels and between 0,010 % and 0,050 % in high alloy steels.

The method does not apply to samples containing silicon nitrides or having silicon contents higher than 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 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:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

Dissolution of a test portion in hydrochloric acid.

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

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 disodium pentacyanonitrosylferrate(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.

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

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

Add 30 ml of sulphuric acid (5.2) little by little into about 700 ml of water. After cooling, make up the volume to 1 l with water, and mix. Dilute 40 ml of this solution to 1 l with water and mix.

5.4 Sodium hydroxide, 500 g/l solution.

Add cautiously, while stirring and cooling, about 900 ml of water into a polyethylene bottle containing 500 g of sodium hydroxide pellets. Cool, dilute to 1 l with water and mix.

5.5 Sodium hydroxide, 7,5 g/l solution.

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

5.6 Potassium sulfate, K_2SO_4 .

5.7 Copper(II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$.

5.8 Disodium hydrogenphosphate, 0,1 mol/l solution.

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

5.9 Sodium hypochlorite, $NaClO$, approximately 0,1 mol/l solution, with approximately 0,3 % (mass fraction) of active chlorine.

Store this solution at a temperature less than 10 °C for at least 3 d or more before use.

5.10 Sodium phenoxide, solution.

Add, while stirring and cooling, 5 g of phenol (C_6H_6O) 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 Sodium pentacyanonitrosylferrate(III), 0,25 g/l solution.

Dissolve 10 g of sodium pentacyanonitrosylferrate(III) dihydrate (sodium nitroprusside dihydrate) [$Na_2Fe(CN)_5NO \cdot 2H_2O$] in water, make up the volume to 1 l with water, and mix.

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