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Steel and iron — Determination of nitrogen content — Thermal conductimetric method after fusion in a current of inert gas

Aciers et fontes — Dosage de l'azote — Méthode par conductibilité thermique après fusion sous un courant de gaz inerte

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10720 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition.*

Annexes A to C of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Steel and iron — Determination of nitrogen content — Thermal conductimetric method after fusion in a current of inert gas

1 Scope

This International Standard specifies a thermal conductimetric method after fusion under inert gas for the determination of nitrogen in steel and iron.

The method is applicable to nitrogen contents between 0,000 8 % (*m*/*m*) and 0,5% (*m*/*m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:—[1], *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *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 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

ISO 14284 :1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition.*

3 Principle

Fusion of a test portion in a single-use graphite crucible under helium gas at a high temperature (e. g. 2 200 °C) Extraction of the nitrogen in the form of molecular nitrogen in the stream of helium.

Separation from the other gaseous extracts and measurement by thermal conductimetric method.

4 Reagents and materials

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

1) To be published. (Revision of ISO 1042:1983)

4.1 Water, prepare just before use.

4.2 Helium, high purity, total impurity content 0,000 5 % (*m*/*m*).

An oxidation reagent or catalyst [copper(II) oxide or platinum] tube heated to a temperature above 450 °C shall be used prior to a purifying unit, when the presence of organic contaminants is suspected in the helium.

4.3 Pure iron, of known low nitrogen contents less than 0,001 % (*m*/*m*).

4.4 Copper (II) oxide, on granulated support.

4.5 Anhydrous magnesium perchlorate, $Mg(ClO_4)_2$, particle size from 1,2 mm to 2,0 mm, or anhydrous calcium sulfate, particle size from 0,60 mm to 0,85 mm.

4.6 Sodium hydroxide, on granulated support.

Particle size from 0,7 mm to 1,2 mm.

4.7 Appropriate solvent, suitable for washing greasy or dirty test samples, e.g. acetone.

4.8 Potassium nitrate, standard solution.

After drying at between 100 °C and 105 °C for 2 h and allowing to cool in a desiccator, weigh, to the nearest 0,1 mg, the masses of potassium nitrate [purity > 99,9 % (*m*/*m*)] indicated in table 1.

Dissolve the potassium nitrate in about 50 ml of water (4.1), transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark with water (4.1) and mix.

1 ml of each standard solution contains the mass of nitrogen indicated in table 1.

Table 1 — Standard solutions

Name of the standard solution	Mass of potassium nitrate used g	Corresponding concentration of nitrogen mg/ml
4.8.1	9,022 8	12,5
4.8.2	7,218 2	10,0
4.8.3	5,413 8	7,5
4.8.4	3,609 1	5,0
4.8.5	1,804 6	2,50
4.8.6	0,902 3	1,25
4.8.7	0,360 9	0,50
4.8.8	[4.8.5 x 1/10] [1)	0,25
4.8.9	[4.8.6 x 1/10]	0,125
4.8.10	[4.8.7 x 1/10]	0,050

1) e.g. .transfer 10,0 ml of the standard solution (4.8.5) into a 100 ml one-mark volumetric flask, dilute to the mark with water (4.1) and mix.

5 Apparatus

During the analysis, unless otherwise stated, use only ordinary laboratory apparatus.

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

The apparatus required for fusion of the test portion, separation and measurement of the nitrogen extracted may be obtained commercially from a number of manufacturers. Follow the manufacturer's instructions for the operation of the instrument.

Features of commercial instruments are given in annex A.

5.1 Graphite crucible, single-use.

Use high purity crucibles suitable for use with the apparatus.

5.2 Micropipette, 100 µl and 200 µl, limit of error shall be less than 1 µl.

5.3 Nickel capsule.

For example, about 6 mm in diameter; 8 mm in height; 0,2 g in mass and 0,23 ml in volume, or about 6 mm in diameter; 12,5 mm in height; 0,5 g in mass and 0,35 ml in volume. In any case, the nitrogen content shall be less than 0,000 2 % (*m*/*m*).

5.4 Crucible tongs, for handling the crucibles used.

5.5 Glass-wool filters.

6 Sampling

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

7 Procedure

WARNING — The risks involved when using an apparatus for fusing the test portion are mainly risks of burns. It is therefore essential to use crucible tongs (5.4) and appropriate containers for the used crucibles.

7.1 General instructions

Keep the glass-wool filters (5.5) clean. Using a certified reference material, verify the effectiveness of the installed reagents (4.4, 4.5 and 4.6) and change them if necessary.

In certain instruments, it is necessary to clean the sample introduction pipe in the furnace after each analysis in order to eliminate carbon deposits. If the electricity supply has been switched off for a long time, allow time for the instrument to stabilize as recommended by the manufacturer.

After changing the filters (5.5) and/or reagents (4.4, 4.5 and 4.6), or when the apparatus has been inoperative for a period, stabilize the instrument by carrying out trial analyses, the results of which are to be disregarded, then proceed with calibration as indicated in 7.5 before analysing the sample.

If the instrument used provides a direct reading in percentage of nitrogen, adjust the instrument reading for each calibration range as follows.

Read the content of a certified reference material of high nitrogen content at various power settings. The required heating power for the determination of test samples is that at which the reading levels off.

In order to determine a high alloy test sample a high alloy certified reference material shall be used to know the required heating power.