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Cast irons — Determination of non-combined carbon content — Infrared absorption method after combustion in an induction furnace

Fontes — Détermination du carbone non combiné — Méthode par absorption dans l'infrarouge après combustion dans un four à induction

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

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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 on 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 the following URL: www.iso.org/iso/foreword.html.

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

This first edition Technical Specification replaces the first edition Technical Report (ISO/TR 10719:1994), which has been technically revised. The following changes have been made:

— steels have been deleted from the scope;

— the possibility to establish the calibration curve by means of certified reference materials has been added;

— assessment of the precision data has been revised.

Cast irons — Determination of non-combined carbon content — Infrared absorption method after combustion in an induction furnace

1 Scope

This document specifies an infrared absorption method after combustion in an induction furnace for the determination of non-combined carbon content in cast irons.

The method is applicable to non-combined carbon contents between 1,0 % (mass fraction) and 3,0 % (mass fraction).

Elements ordinarily present do not interfere. However, some alloyed cast irons, when extensively heat treated, contain carbides that are not soluble when using this method and may give high values for non-combined carbon.

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 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

For the purposes of this document, the following terms and definitions apply.

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

3.1
non-combined carbon
graphitic carbon
carbon that is not dissolved by methanol and nitric and hydrofluoric acids

4 Principle

Decomposition of a test portion with nitric acid in the presence of methanol and treatment with hydrofluoric acid. Removal of the non-combined carbon by filtering through a glass-fibre filter.

Combustion of the glass-fibre filter containing the non-combined carbon in a flow of oxygen at a high temperature, using a high-frequency induction furnace, in the presence of pure iron and an accelerator. Transformation of carbon into carbon dioxide and/or carbon monoxide.

Measurement by infrared absorption of the carbon dioxide and/or carbon monoxide, carried by the current of oxygen.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only water with a low content of organic matter, i.e. grade 2 or grade 1 water as specified in ISO 3696.

5.1 Water, free from carbon dioxide.

Boil water for 30 min, cool to room temperature and allow oxygen (5.2) to bubble through it for 15 min. Prepare just before use.

5.2 Oxygen, minimum purity 99,5 % (volume fraction).

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

5.3 Iron, containing less than 0,001 0 % of carbon (mass fraction).

5.4 Solvent, appropriate for cleaning greasy or dirty test samples, for example, acetone.

5.5 Methanol, minimum purity 99,5 % (volume fraction).

5.6 Barium carbonate.

Dry barium carbonate [minimum purity > 99,5 % (mass fraction)] at 105 °C to 110 °C for 3 h and cool in a desiccator before use.

5.7 Cast iron certified reference materials (CRMs), containing 1 % to 3 % (mass fraction) carbon.

The cast irons to be used for this purpose shall have white structure.

5.8 Accelerator.

Copper, tungsten-tin mixture or tungsten containing less than 0,001 0 % carbon (mass fraction).

5.9 Nitric acid, ρ about 1,40 g/ml.

5.10 Hydrofluoric acid, ρ about 1,15 g/ml.

5.11 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 1.

5.12 Sodium hydroxide, 120 g/l solution.

Cautiously dissolve 60 g of sodium hydroxide in about 200 ml of water (5.1). When dissolution is complete, cool, dilute to 500 ml with water, mix and store in a plastic bottle.

5.13 Magnesium perchlorate [$Mg(ClO_4)_2$], particle size from 0,7 mm to 1,2 mm.

5.14 Inert ceramic (attapulgus clay), impregnated with sodium hydroxide, particle size from 0,7 mm to 1,2 mm.

6 Apparatus

The apparatus required for combustion in a high frequency induction furnace and the subsequent infrared absorption measurement of the evolved carbon dioxide and/or carbon monoxide may be obtained commercially from a number of manufacturers. Follow the manufacturer's instructions for

the operation of the instrument. A pressure regulator is required to control the oxygen pressure to the furnace according to the manufacturer's specification.

Ordinary laboratory apparatus and the following shall be used.

6.1 Filter, made of glass fibre, 47 mm in diameter, pore size 0,3 µm.

6.2 Vacuum filtering apparatus, for use with 47 mm glass-fibre filters (6.1) and suitable for use with acids.

6.3 Ceramic crucible, capable of withstanding combustion in an induction furnace.

Ignite crucibles in an electric furnace in air or in a current of oxygen, for not less than 2 h at 1 100 °C, and store in a desiccator before use.

7 Sampling

Sampling shall be carried out in accordance with ISO 14284 or with an appropriate national standard for cast irons (see Annex C).

NOTE For cast irons, use drillings with lengths of more than 2 mm. For spheroidal graphite cast irons, use solid portions, approximately 10 mm x 10 mm x 0,3 mm.

8 Procedure

SAFETY INSTRUCTIONS

The risks related to combustion analysis are mainly burns in pre-igniting the ceramic crucibles and in the fusions. Use crucible tongs at all times and suitable containers for the used crucibles. Normal precautions for handling oxygen cylinders shall be taken. Oxygen from the combustion process shall be removed effectively from the apparatus, since a high concentration of oxygen in a confined space can present a fire hazard.

Take care not to mix methanol with concentrated nitric acid because of the risk of explosion.

8.1 General operating instructions

Purify the oxygen supply (5.2) using tubes packed with the inert ceramic (5.14) impregnated with sodium hydroxide (5.12) and magnesium perchlorate (5.13), and maintain a quiescent flow rate while on standby. Maintain a glass-fibre filter or a stainless steel filter screen as a dust collector. Clean and change as necessary. The furnace chamber, pedestal post and filter trap shall be cleaned frequently to remove oxide build-up.

When the main supply is switched on after being out of action for any length of time, allow the equipment to stabilize for the time recommended by the equipment manufacturers.

After cleaning the furnace chamber and/or changing filters, or after the equipment has been inoperative for a period, stabilize the apparatus by burning several samples, of similar type to the samples to be analysed, prior to setting up for analysis.

Flush oxygen through the apparatus and adjust the instrument controls to give a zero reading.

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

Select a cast iron certified reference material with a carbon content close to the maximum carbon content in the calibration series, measure the corresponding carbon content in accordance with the manufacturer's instructions and adjust the reading of the instrument to the certified value.