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Ferronickels — Determination of carbon content — Infrared absorption method after induction furnace combustion

Ferronickels — Détermination de la teneur en carbone — Méthode par absorption dans l'infrarouge après combustion dans un four à induction

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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 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 155, *Nickel and nickel alloys.*

This second edition cancels and replaces the first edition (ISO 7524:1985), which has been technically revised. The main changes compared with the previous edition are as follows:

— the scope has been limited to ferronickels only;

— the former Clauses 5 and 7 have been technically revised;

— the former Annexes A and C have been deleted;

— the precision data have been updated.

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.

Ferronickels — Determination of carbon content — Infrared absorption method after induction furnace combustion

1 Scope

This document specifies an infrared absorption method after combustion in an induction furnace for the determination of the carbon content in ferronickels in the range of 0,004 % to 2,5 %.

The method is applicable to normal production operations. It uses commercially available equipment, which is calibrated using steel and/or ferronickel certified reference materials (CRMs).

2 Normative references

There are no normative references in this document.

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

Combustion of a test portion in a high-frequency induction furnace at high temperature in a current of pure oxygen, and in the presence of accelerators and fluxes.

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, use only reagents of recognized analytical grade.

5.1 Oxygen, high purity (mass fraction minimum 99,5 %).

An oxidation catalyst [copper(II) oxide or platinum] tube heated at 600 °C, followed by appropriate carbon dioxide and water absorbents, shall be used when the presence of organic contaminants is suspected in the oxygen.

5.2 Inert ceramic (attapulgus clay), impregnated with sodium hydroxide and having particle sizes from 0,7 mm to 1,2 mm for the absorption of carbon dioxide.

5.3 Magnesium perchlorate [$Mg(ClO_4)_2$], having particle size from 0,7 mm to 1,2 mm for the absorption of moisture.

5.4 Glass-wool.

5.5 Fluxes and accelerators.

5.5.1 General

A flux addition has the effect of bonding together small particles for more effective furnace coupling and it helps to produce a more fluid melt.

An accelerator addition allows:

a) a good coupling medium for the induction heating of otherwise unsatisfactory samples (finely divided samples, materials of complex composition);

b) a higher combustion temperature;

c) an increase of the mass of material in the crucible when the test portions are small.

Any flux or accelerator shall have low carbon content and shall be used in the calibration procedure. The total blank from all sources (oxygen, refractories, flux and accelerator) shall not exceed 0,001 % (mass fraction) carbon.

Some materials act as both a flux and an accelerator.

5.5.2 Fluxes

Common fluxes are tin, copper plus tin, copper or tungsten.

5.5.3 Accelerators

Common accelerators are copper, iron, tungsten or nickel.

5.6 Steel and/or ferronickel certified reference materials (CRMs), containing from 0,001 % to 2,6 % (mass fraction) carbon.

All reference materials used for calibration shall be certified by internationally recognized bodies. Preference shall be given to materials that are certified using referee methods, e.g. ISO 9556, and traceable to SI units as opposed to those based on other CRMs.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Carbon analyser.

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

Follow the manufacturer's instructions for the operation of the equipment. A pressure regulator is required to control the oxygen pressure to the furnace according to the manufacturer's specification.

6.1.2 Purify the oxygen supply using tubes packed with inert ceramic (5.2) and magnesium perchlorate (5.3) and maintain a flow rate of about 0,5 l/min while on stand-by.

6.1.3 Maintain a glass-wool filter (5.4) between the furnace chamber and the analyser and change as necessary. The furnace chamber, pedestal post and filter trap should be cleaned frequently to remove oxide residues.

6.1.4 The manufacturer may recommend setting the programming unit to give a pre-burn period before oxygen enters the furnace chamber. The test portion should be at a red heat during the pre-burn period. When oxygen is introduced for the combustion stage, the temperature increases substantially.

6.1.5 The temperature reached during combustion depends on the furnace, and the type and quantity of metal in the crucible. A high temperature (>1 700 °C) is maintained after the test portion is fused so that the carbon dioxide can be completely removed from the furnace to the infrared analyser.

6.1.6 The flow rate of oxygen can vary from one instrument to another but is usually about 2,0 l/min during the combustion period.

6.1.7 After the equipment has been idle for a few hours or after cleaning the furnace chamber or filters, the instrument should be stabilized as described in 8.1.

NOTE Features of commercial equipment are given in Annex A.

6.2 Ceramic crucibles and lids.

Ceramic crucibles, containing the sample and any additions that can be necessary, are required for the combustion. They shall be of precise dimensions for the system and fit the supporting pedestal post so that the test portion in the crucible is positioned correctly within the induction coil for heating.

Typical dimensions of combustion crucibles are: a height of 25 mm, an external diameter of 25 mm, an internal diameter of 20 mm, a wall thickness of 2,5 mm and a thickness of base of 8 mm. The dimension of the hole of the lid should be larger than 10 mm.

Crucibles and lids shall be as specified by the manufacturer of the instrumentation used and shall be capable of withstanding combustion in an induction furnace without evolving carbon-containing chemicals so that achieving and maintaining blank values within specification is possible.

In order to remove any carbon contamination, for the determination of carbon levels of less than 0,05 % (mass fraction), pre-ignite the crucibles in air or oxygen in a furnace for not less than 1 h at 1 100 °C and store in a desiccator or a closed container. A resistance furnace with a combustion tube through which a flow of oxygen passes may also be used.

Crucible lids, used to help retain the solid oxidation products in the hot zone of the induction coil, shall be pre-ignited in a similar manner.

6.3 Crucible tongs, capable of handling recommended crucibles (6.2).

7 Sampling and sample preparation

7.1 Sampling and sample preparation shall be carried out by normal agreed procedures or, in cases of dispute, by appropriate national standards.

7.2 The laboratory sample is normally in the form of a powder, granules, millings or drillings and no further preparation of the sample is necessary.

7.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned by washing with high purity acetone and drying in air.

7.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test portion should be obtained by riffling.

8 Procedure

WARNING — 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 appropriate 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.

8.1 Preparing and stabilizing the instrument

8.1.1 Assemble the apparatus and prepare it for operation according to the manufacturer's instructions. Test the furnace and analyser to ensure the absence of leaks.

8.1.2 Condition and stabilize the equipment by combusting several samples, similar to those to be analysed, using appropriate fluxes and accelerators, before attempting to calibrate the system or determine the blank.

NOTE It is not necessary to use pre-ignited crucibles.

8.1.3 Allow the instrument to cycle several times with oxygen flowing and adjust the zero of the instrument.

8.2 Blank test and zero adjustment

8.2.1 Carbon contents ≤ 0,1 %

For each instrument range, transfer the selected mass of flux (5.5.2), to the nearest 0,005 g, into a pre-ignited crucible (6.2), add the selected mass of a CRM (5.6) having a very low carbon content, and then cover it with the selected mass of accelerator (5.5.3).

The type and mass of the flux and accelerator used in the blank test should accord with those used for determinations (details as in 8.4.1). Record the mass of the CRM. Place the crucible and contents on the furnace pedestal and operate the furnace in accordance with the manufacturer's instructions. Repeat the determination three more times. Average the results.

Subtract the carbon content of the CRM from the average to determine the blank value. If the blank is greater than 0,001 % (mass fraction) and the related standard deviation is greater than 0,000 2 % (mass fraction), find the cause of the problem, fix it and repeat the experiment.

Record the average blank value into the analyser in accordance with the manufacturer's instructions.

NOTE 1 The reading obtained corresponds to the blank due to the crucible, flux and accelerator.

If the analyser does not have automatic blank correction, the blank value should be subtracted from the total result prior to any calculation.

NOTE 2 An alternative procedure is to record the reading of the blank test and make the correction using a calibration graph.

8.2.2 Carbon contents > 0,1 %

It is preferable not to carry out a blank correction. However, a check of the blank level shall be done. The value shall remain below 0,001 % (mass fraction).

8.3 Calibration

8.3.1 Select a set of steel and/or ferronickel CRMs (5.6) for calibration and verification which, at a minimum, fall at the bottom, top and quartile points of each operational operating range.

8.3.2 If the instrument has more than one carbon detector (measurement system), carry out the adjustment described in this section on each one.

Establish all experimental parameters for each range of carbon. Parameters to be specified include:

— crucible: to be pre-burned or not;

— flux and accelerator: type and mass;

— test portion: mass.

8.3.3 For each detector (see 8.3.2) weigh an appropriate amount (usually 0,50 g) of a CRM having a carbon content corresponding to the top of each operating range into a pre-ignited crucible (6.2). Add the pre-selected amounts of flux (5.5.2) and accelerator (5.5.3) and combust as described in 8.4.1. Repeat this process twice.

If the results are situated in the interval "Certified Value ± 2σ", adjust the net instrument reading to correspond to the certified content of carbon in the reference material, according to the manufacturer's instructions.

8.3.4 Check the linearity of the calibration by analysing at least three times a CRM having a carbon content in the middle of the range of the detector. The result shall be situated in the interval "Certified Value ± 2σ".

Correct any non-compliant conditions before continuing to the next step.

8.4 Determination

8.4.1 Weigh, to the nearest 0,001 g, 0,5 g to 0,6 g of the test sample, and transfer into a pre-ignited crucible (6.2) containing an appropriate amount of the selected flux (5.5.2), if required. Then cover the test sample with the appropriate quantity of accelerator (5.5.3) and, if necessary, place the lid on the crucible.

NOTE The flux and accelerator used will depend on the individual characteristics of the equipment and the type of material being analysed. For ferronickels, 1,0 g to 1,5 g of tungsten plus tin (7 to 9 parts of tungsten + 1 part of tin) can be used.

8.4.2 Place the crucible and contents on the pedestal post of the furnace, raise to the combustion position and lock the system. Operate the furnace in accordance with the manufacturer's instructions.

8.4.3 Record the analyser reading and repeat the determination at least once.

NOTE 1 It is important that a high temperature be maintained after the sample is fused to ensure the complete transfer of the carbon dioxide from the furnace to the infrared analyser.

NOTE 2 Quiescent combustion is necessary to avoid splashing on to the crucible lid.

8.4.4 Analyse, at least twice, CRMs having carbon contents slightly above and slightly below the content found for each unknown sample. The corresponding result shall be situated in the interval "Certified Value ± 2σ". The CRMs used for checking the accuracy of the determination shall be different from those used for the calibration (see 8.3).