International Standard

ISO 24476

Steel — Determination of oxygen — Infrared absorption method after fusion under inert gas (Routine method)

Aciers — Détermination de l'oxygène — Méthode par absorption dans l'infrarouge après fusion sous gaz inerte (Méthode de routine)

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Foreword

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

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 oxygen — Infrared absorption method after fusion under inert gas (Routine method)

1 Scope

This document specifies a routine method after fusion under inert gas for the determination of oxygen in steel.

The method is applicable to contents of oxygen between 0,001 % (mass fraction) and 0,02 % (mass fraction).

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 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 terminology databases for use in standardization at the following addresses:

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

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Fusing of a test portion in a single-use graphite crucible under an inert gas stream (He or Ar) at a minimum temperature of 2 000 °C. The sample is melted in a graphite crucible. The released oxygen combines with carbon to form carbon monoxide.

Depending on the instrument design, the carbon monoxide is oxidized to carbon dioxide or left as carbon monoxide and swept by the inert gas stream to an infrared detector.

The detector output is compared to that obtained from similar certified reference materials and is displayed as oxygen content of the sample.

The calibration curve is established using steel certified reference materials (CRMs).

5 Reagent and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

5.1 Helium or argon, of ultra-high purity, total impurity content 0,000 5 % (mass fraction) (99,999 % purity minimum by volume).

5.2 Copper(II) oxide, on granulated support.

This reagent is used in some instruments to oxidize carbon monoxide to carbon dioxide. Use the purity specified by the instrument's manufacturer.

5.3 Magnesium perchlorate, (commercial designation: anhydrone), particle size: from 1,2 mm to 2,0 mm.

This reagent is used in the instrument to absorb water. Use the purity specified by the instrument's manufacturer.

5.4 Sodium hydroxide on clay (commercial designation: ascarite), particle size from 0,6 mm to 1,2 mm, for example.

This reagent is used in some instruments to absorb carbon dioxide. Use the purity specified by the instrument's manufacturer.

5.5 Appropriate solvent, suitable for washing greasy or dirty test samples, e.g. high-purity acetone

5.6 Steel reference materials (RMs), material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.

5.7 Steel certified reference materials (CRMs), reference material (RM) characterized by a metrologically valid procedure for one or more specified properties, accompanied a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

6 Apparatus

6.1 Oxygen analyser, 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.

6.2 Graphite crucible, single use. Use high-purity graphite crucibles suitable for use with the instrument.

6.3 Crucible tongs, for handling the graphite crucibles used.

6.4 Glass-wool filters.

7 Sampling

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

8 Procedure

WARNING — The risks involved when using an apparatus for fusing a test portion are mainly burn risks. It is therefore essential to use crucible tongs (6.3) and appropriate containers for the crucibles used.

8.1 General

Assemble the apparatus as recommended by the manufacturer. Make the required power, gas and water connections. Switch on the instrument and allow sufficient warm up time to stabilize.

After changing the filter and/or reagents, 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 blank, calibration and preparation tests as indicated in 8.3 and 8.4 before analysing the sample.

Place a graphite crucible (6.2) on the furnace pedestal and then degas by heating above the degassing temperature of the sample.