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Steel and cast iron — Determination of total carbon content — Combustion gravimetric method

Aciers et fontes — Dosage du carbone total — Méthode gravimétrique après combustion

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 437 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 of which has been technically revised.

Introduction

The first edition of ISO 437 published in 1985 specified the use of asbestos, which is now known as a carcinogenic substance. The main revised items of the second edition from the first edition are the replacements of substances containing asbestos with substances free of asbestos. Some points have been also revised editorially and/or technically in accordance with the updated ISO/IEC Directives, Part 2 and recent specifications in International Standards prepared by ISO/TC 17/ SC1.

Steel and cast iron—Determination of carbon—Combustion gravimetric method

1 Scope

This International Standard specifies a gravimetric method for the determination of carbon in steel and cast iron after combustion of the test portion in a current of oxygen.

The method is applicable to mass fractions of total carbon not less than 0,1 %.

2 Normative references

The following referenced documents are indispensable for the application 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 Principle

Combustion of a test portion at a high temperature (1 200 °C to 1 350 °C) in a current of pure oxygen, if necessary in the presence of a flux and Conversion of carbon into carbon dioxide.

Absorption of the carbon dioxide carried by the current of oxygen in a carbon dioxide absorbent contained in a weighed absorption bottle, and determination of the increase in mass.

4 Reagent

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

4.1 Oxygen, minimum purity 99 % (volume fraction)

4.2 Magnesium perchlorate, $Mg(ClO_4)_2$, anhydrous

WARNING — Contact between magnesium perchlorate and organic substances should be avoided because of possible risk of explosion.

4.3 Fluxes, copper oxide, tin, pure iron, etc.

4.4 Iron, high purity quality containing less than 0,001 0 % mass fraction of carbon

4.5 Manganese dioxide or silver orthovanadate

Prepare as follows:

4.5.1 Manganese dioxide

When a chemically active grade is not available, it may be prepared as follows:

it may be prepared as follows (about 80 g of manganese dioxide is obtained):Dissolve 200 g of manganese(II) sulphate tetrahydrate (MnSO4·4H2O) in 2,5 l of water in a 4 l beaker and add ammonium hydroxide for making this solution clearly ammoniacal. Add 1 l of ammonium persulphate solution (225 g/l) freshly prepared and bring the whole to boiling point. Continue boiling for 10 min and add ammonium hydroxide solution as frequently as is necessary to maintain the solution ammoniacal. Allow the precipitate to settle.
If the supernatant liquid is not clear, or the precipitate does not settle quickly, add 50 ml to 100 ml of ammonium persulphate solution and boil again for 10 min, keeping the solution constantly ammoniacal.

When precipitation appears to be complete, allow the manganese dioxide to settle completely, carefully siphon off the supernatant liquid and wash the precipitate by decantation with 3 l to 4 l of hot water in portions of 500 ml to 600 ml. Stir the manganese dioxide well in the water and allow to settle after each washing and before decantation. Finally, wash twice in the same way with very dilute sulphuric acid.

In the meantime, prepare a 15 cm diameter funnel fitted with a 5 cm filter disc covered with a thin layer of purified fibreglass pulp (it is also permitted to use a porcelain funnel of the Buchner type).

After the last washing, transfer the manganese dioxide onto the filter and wash with hot water until it is free from sulphate ions. Then place this on a porcelain dish and dry it in an oven at 105 °C.

Grind the manganese dioxide in a mortar so that it passes through a sieve with apertures of 0,8 mm and dry it again completely at 105 °C.

4.5.2 Silver orthovanadate

Dissolve 60 g of sodium orthovanadate (Na_3VO_4) in 400 ml of water. Boil for 15 min. Also dissolve 170 g of silver nitrate ($AgNO_3$) in 200 ml of water.

Add the silver nitrate solution, drop by drop, into the warm solution of sodium orthovanadate. An abundant yellow orange precipitate appears.

Filter this precipitate on a Buchner funnel and wash it with water until free from silver ions (Ag^+) and verify this by means of a solution of chloride ions (Cl^-).

Dry the precipitate overnight at about 80 °C. It may darken slightly.

Grind and keep it shaded from light.

NOTE Ammonium orthovanadate is not sufficiently soluble to be specified.

4.6 Carbon dioxide absorbent, inert ceramic impregnated with sodium hydroxide, in granules of about 2 mm diameter.

Avoid contact with air.

4.7 Oxidation catalyst, copper (II) oxide or platinum

4.8 Chromic/sulphuric acid mixture

Saturate a solution of sulphuric acid (*ρ* approximately 1,84 g/ml) with chromic acid. The solution shall have a permanent red colour.

5 Apparatus

The apparatus consists of a source of oxygen and the unit its purification, the furnace with the combustion tube, the purification train and the carbon dioxide absorption system.

These different parts, which are joined together with connecting tubes forming an air-tight seal, are shown in **Figure 1.**

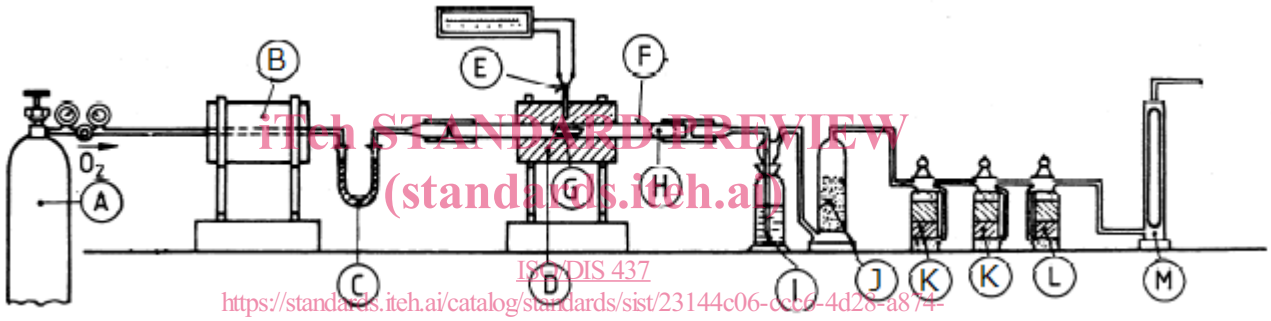

Figure 1 — Apparatus of combustion and carbon dioxide absorption system

5.1 Source of oxygen (4.1) (A), with pressure-regulating valve

5.2 Wire-wound furnace (B), with non-porous porcelain combustion tube containing the oxidation catalyst (4.7) heated to 625 °C.

5.3 Unit for drying and purifying the oxygen (C), containing magnesium perchlorate (4.2) and carbon dioxide absorbent (4.6) separated by glass wool (diameter of tubes 25 mm, height 100 mm approximately) connected by tubing.

5.4 Wire-wound or resistor rod furnace (D), made of metal or carborundum and capable of raising the temperature of the combustion tube up to 1 350 °C.

NOTE Induction furnaces may also be used.