
International Standard



437

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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 institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 437 was developed by Technical Committee ISO/TC 17, *Steel*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 437-1965, which had been approved by the member bodies of the following countries :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Canada	Iran	Spain
Chile	Italy	Sweden
Czechoslovakia	Japan	Switzerland
Egypt, Arab Rep. of	Netherlands	Turkey
France	Norway	United Kingdom
Denmark	Poland	USSR
Finland	Portugal	Yugoslavia

The member bodies of the following countries had expressed disapproval of the document on technical grounds :

Belgium
Germany, F. R.
USA

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

1 Scope and field of application

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

The method is applicable to products having carbon contents not less than 0,1 % (*m/m*).

2 References

ISO 377/2, *Selection and preparation of samples and test pieces of wrought steels — Part 2 : Samples and test pieces intended for the determination of the chemical composition.*¹⁾

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

3 Principle

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

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

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Oxygen, minimum purity 99 % (*V/V*).

4.2 Magnesium perchlorate, $\text{Mg}(\text{ClO}_4)_2$, anhydrous.

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

4.4 Electrolytic iron.

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 :

Weigh about 50 g of manganese dioxide, dissolve in a 4 l beaker 200 g of manganous sulphate tetrahydrate ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$) in 2,5 l of water. After 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 if the precipitate does not settle quickly, add 50 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 to 4 l of warm water in portions of 500 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 asbestos 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 warm 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.

1) At present at the stage of draft. (Partial revision of ISO/R 377-1964.)

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 should form.

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 for it to be specified.

4.6 Soda asbestos, asbestos impregnated with sodium hydroxide, in granules of about 2 mm diameter.

Avoid contact with air.

4.7 Platinized asbestos.

4.8 Chromic/sulphuric acid mixture.

Saturate a solution of sulphuric acid (ρ approximately 1,83 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 for purifying it, 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.

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

5.2 Mercury valve (B).

5.3 Wire-wound furnace (C), with non-porous porcelain combustion tube containing the platinized asbestos (4.7) heated to 625 °C.

5.4 Unit for drying and purifying the oxygen (D), containing magnesium perchlorate (4.2) and soda asbestos (4.6) separated by glass wool (diameter of tubes 25 mm, height 100 mm approximately) connected by tubing.

5.5 Wire-wound or resistor rod furnace (E), 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.

5.6 Thermocouple (F), for measuring the temperature. The tip of the thermocouple, protected by a sheath, is placed near the external surface of the combustion tube. The relation between the internal temperature of the tube and the pyrometer readings shall first be established.

5.7 Combustion tube (G), made of refractory material which is not porous at the test temperature, 20 to 30 mm internal diameter and at least 650 mm long so that the ends of the tube remain cold during combustion.

Pressure-tight connections at both ends of the combustion tube should be fitted by external sleeves.

5.8 Boat (H), with eyelet made of refractory material (length 80 to 100 mm, width 12 to 14 mm, depth 8 to 9 mm), first calcined in a current of oxygen at the temperature of use.

5.9 Plug (I), of calcined asbestos wool or quartz wool to filter the gas.

5.10 Washing bottle (L), containing the manganese dioxide or silver orthovanadate (4.5). When analysing test portions of high sulphur content, it is preferable to use two washing bottles of manganese dioxide or one washing bottle of manganese dioxide and one of the chromic/sulphuric acid mixture (4.8).

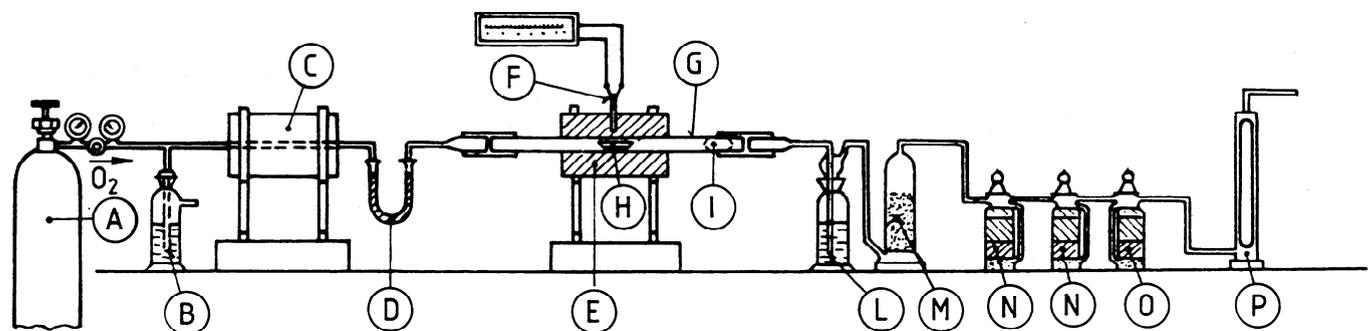


Figure 1

5.11 Drying bottle (M), containing the magnesium perchlorate (4.2).

5.12 Weighed absorption bottles (N), for absorbing the carbon dioxide, containing the soda asbestos (4.6) and a certain amount of the magnesium perchlorate (4.2) to trap the water which forms during absorption of the carbon dioxide. The total mass of each absorption bottle ready for use (see figure 2) should not exceed 100 g.

5.13 Unweighed absorption bottle (O), facing the opposite way to (N).

5.14 Flow meter (P).

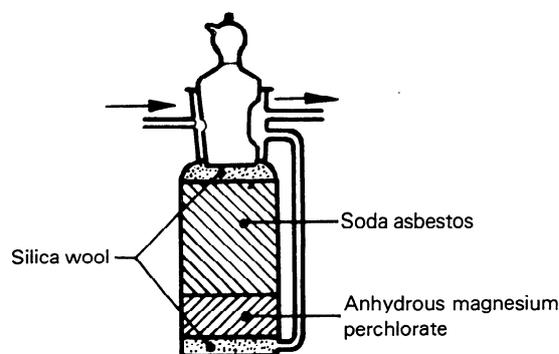


Figure 2

6 Sampling

Sampling shall be carried out in accordance with ISO 377/2 or appropriate national standards for cast iron.

7 Procedure

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

7.1 Preparation of the apparatus

After having verified the pressure tightness of the apparatus and the efficiency of the oxygen (4.1) purification and having reached the required temperature within the range of 1 200 to 1 350 °C in the combustion zone of the furnace (E) (use the higher temperature in the case of high alloy steels), allow oxygen to pass through the apparatus for 10 to 15 min at the rate of 300 to 500 ml/min of gas depending on the diameter of the tube used.

Then disconnect the absorption bottles (N) and weigh them at the ambient temperature and replace them in position.

7.2 Test portion

Prepare the test portion in the form of very short chips having a thickness of a few tenths of millimetre.

According to the presumed carbon content, weigh, to the nearest 0,001 g, the following mass (m_3) of the test portion :

- for C contents ranging from 0,1 to 1 % : m_3 about 2 g;
- for C contents ranging from 1 to 2 % : m_3 about 1 g;
- for C contents greater than 2 % : m_3 about 0,5 g.

7.3 Blank test

Carry out the blank test by the combustion of the boat (H) in the presence of the same quantity of flux (4.3) used in the analysis, and carefully determine the increase in mass (m_2) of the absorption bottles (N).

Where there is any doubt about the purity of the boat, this should be verified by the combustion of the same boat in the presence of the same quantity of flux (4.3) used in the analysis and carefully weighed, and a quantity of weighed electrolytic iron (4.4).

7.4 Combustion

Place the test portion (7.2) in the boat (H) and, if necessary, add 1 to 2 g of flux (4.3) to the test portion, especially in the case of high alloy steels.

Open the combustion tube at the end where the oxygen enters and, using a rigid nickel wire, place the boat (H) and the test portion in the middle of the heated zone of the tube. Quickly close the tube and, after about 1 min, pass through it a current of oxygen at the rate of 300 to 500 ml/min, depending on the diameter of the tube used. After about 1 min, when the combustion has ended, continue to pass oxygen through for another 15 to 20 min so that the carbon dioxide is completely removed from the combustion tube (G), the washing bottle (L) and the drying bottle (M).

NOTE — Shorter times may be used for low carbon contents and when using induction furnaces.

7.5 Determination

Cut off the oxygen current, close the taps of the weighed absorption bottles (N) and remove the porcelain boat from the combustion tube (G). It is necessary to verify that the combustion has been efficiently completed by examining the fused mass in the boat removed from the furnace.

Disconnect the absorption bottles (N) and weigh them at ambient temperature. It is recommended that any electrostatic charge on the absorption bottles should be eliminated by earthing the moving element of the balance. The increase in mass (m_1) represents the carbon dioxide absorbed.

8 Expression of results

The carbon content is given, as a percentage by mass, by the formula

$$0,273 \frac{m_1 - m_2}{m_3} \times 100$$

$$= 27,3 \frac{m_1 - m_2}{m_3}$$

where

m_1 is the mass, in grams, of the carbon dioxide obtained from the combustion of the test portion;

m_2 is the mass, in grams, obtained from the blank test;

m_3 is the mass, in grams, of the test portion;

0,273 is the carbon dioxide to carbon conversion factor.

9 Test report

The test report shall include the following particulars :

- a) the method used, by reference to this International Standard;
- b) the results, and the form in which they are expressed;
- c) any unusual features noted during the determination;
- d) any operation not specified in this International Standard, or any optional operations which may have influenced the result.

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