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International Standard



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Steel and cast iron — Determination of sulphur content — Combustion titrimetric method

Aciers et fontes — Dosage du soufre — Méthode titrimétrique 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 671 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 671-1968, which had been approved by the member bodies of the following countries :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Italy	Sweden
Canada	Japan	Switzerland
Chile	Korea, Rep. of	Turkey
Czechoslovakia	Netherlands	United Kingdom
Denmark	New Zealand	Yugoslavia
Egypt, Arab Rep. of	Norway	
Germany, F. R.	Poland	

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

France
Portugal
USA

Steel and cast iron — Determination of sulphur content — Combustion titrimetric method

1 Scope and field of application

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

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, perforated plate and electroformed sheet — Nominal sizes of openings.*²⁾

3 Principle

Combustion of a test portion at a high temperature (1 450 °C) in a current of pure oxygen, if necessary in the presence of a metal flux. Transformation of sulphur into sulphur dioxide. Absorption of the gases resulting from combustion, freed from oxide dust, by a solution of potassium sulphate and hydrogen peroxide.

Titration of the absorbent solution with a standard sodium borate solution.

4 Reagents

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

NOTE — Verify by blank tests that reagents are free from sulphur. The results should be corrected accordingly.

4.1 Oxygen, minimum purity 99 % (V/V), free from sulphur-bearing compounds.

4.2 Soda asbestos, particle size : approximately 2 mm.

4.3 Magnesium perchlorate, $Mg(ClO_4)_2$, particle size : approximately 2 mm.

4.4 Pure iron, of known low sulphur content.

4.5 Fluxes, iron, copper or tin, of known low sulphur content.

4.6 Absorbent solution

Dissolve 5 g of potassium sulphate in 200 ml of boiled and cooled water. Add 100 ml of hydrogen peroxide and dilute to 2 500 ml with boiled and cooled water.

4.7 Sodium borate, standard solution, corresponding to 0,010 % of sulphur for 1 g/ml of test portion.

Weigh, to the nearest 0,000 1 g, 2,383 9 g of sodium borate, $Na_2B_4O_7 \cdot 10H_2O$, dissolve in boiled and cooled water and dilute to exactly 2 000 ml with the same water.

1 ml of this standard solution contains 0,010 % of sulphur for 1,000 g of test portion.

4.8 Mixed indicator

Dissolve 0,1 g of methylene blue and 0,3 g of methyl red in water and dilute to 500 ml with water.

NOTE — The proportions of the indicators may be adjusted to the sensitivity of the eye of the operator. The coloration of the indicator may be evaluated by means of an optical device.

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

2) At present at the stage of draft. (Revision of ISO 565-1972.)

5 Apparatus

Ordinary laboratory apparatus and the apparatus consisting of a source of oxygen and the unit for purifying it, the furnace with combustion tube and the sulphur 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 (A), fitted with a sensitive regulating valve and a pressure gauge.

5.2 Purifying and drying bottle, containing soda asbestos (B) and magnesium perchlorate (4.3).

5.3 Flow meter (C), capable of measuring a current of oxygen of 0,2 to 3 l/min.

5.4 Electric furnace (D), capable of maintaining the combustion tube (E) at 1 420 °C in continuous operation.

5.5 Combustion tube (E), in refractory material capable of ensuring continuous operation at 1 450 °C, having a wide part of inside diameter 27 mm and length 450 mm, and a narrow part of inside diameter 3 mm and length 250 mm.

NOTES

- 1 The restricted part of the combustion tube is placed in the hottest central region of the furnace.
- 2 The junction between the combustion tube (E) and the gas outlet tap (K) in contact with the very hot gaseous mixture leaving the fur-

nace should be cooled, especially if it is made from tubing of natural or synthetic rubber. The cooling should be such that the temperature of the inner surface in contact with the gaseous mixture does not exceed 40 °C.

5.6 Oxygen inlet tap (F).

5.7 Oxygen inlet device (G), with a window permitting visual observation of the progress of combustion (see figure 2 as an example).

5.8 Platinum/platinum-rhodium thermocouple (H), in which the hot junction is placed near the external surface of the tube close to the boat and cartridge assembly. The relationship between the inner temperature of the tube (E) and the indications of the pyrometer should be established and checked periodically.

5.9 Boat of refractory material (I), with a wide, flat base capable of withstanding two heatings at 1 420 °C (length 80 to 100 mm, height 8 to 9 mm, width 15 to 16 mm) (see figure 3).

5.10 Refractory filter-cartridge (J), (inside diameter 14 mm, outside diameter 16 mm, length 50 mm) (silica content 12 to 15 % (m/m), the remainder being alumina). The porosity of the cartridge is defined by the flow of gas through the whole surface at a given pressure : 4 to 5 l/min of gas at a pressure of 250 mm of water (see figure 3).

Boats and cartridges should be calcined at 1 420 °C for 10 min in a current of oxygen, and should be kept in a desiccator after calcination.

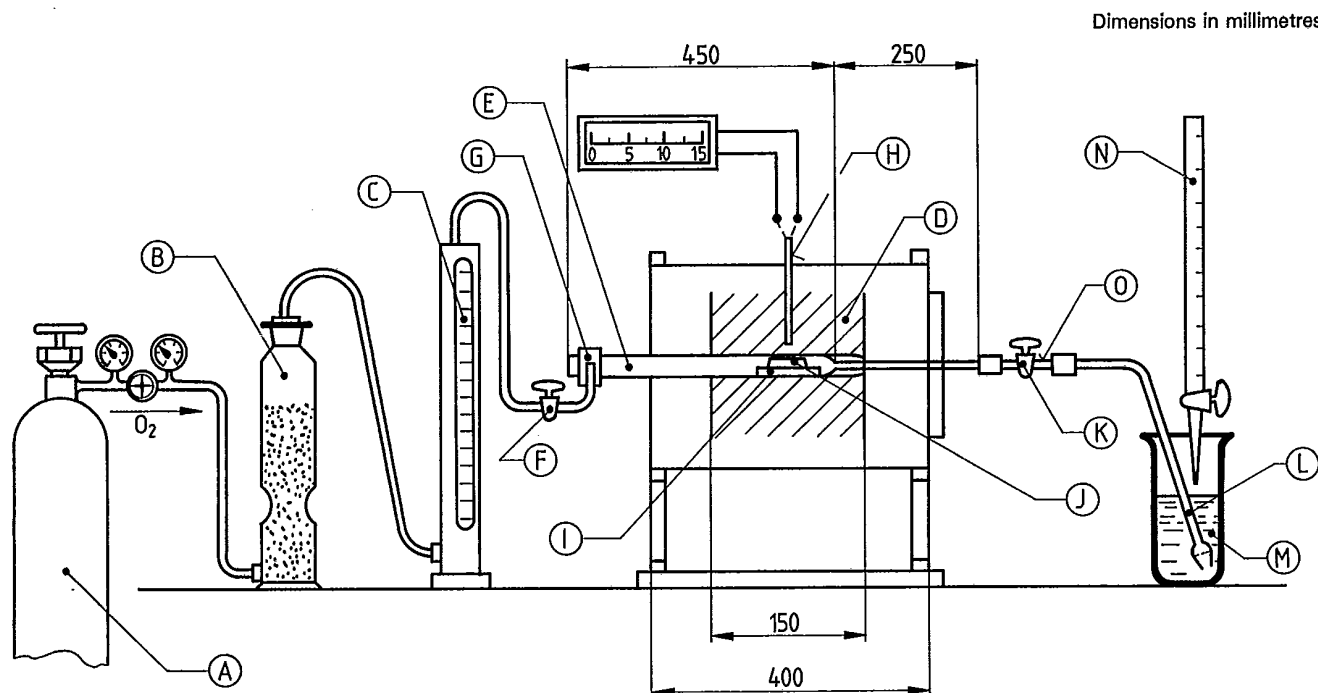
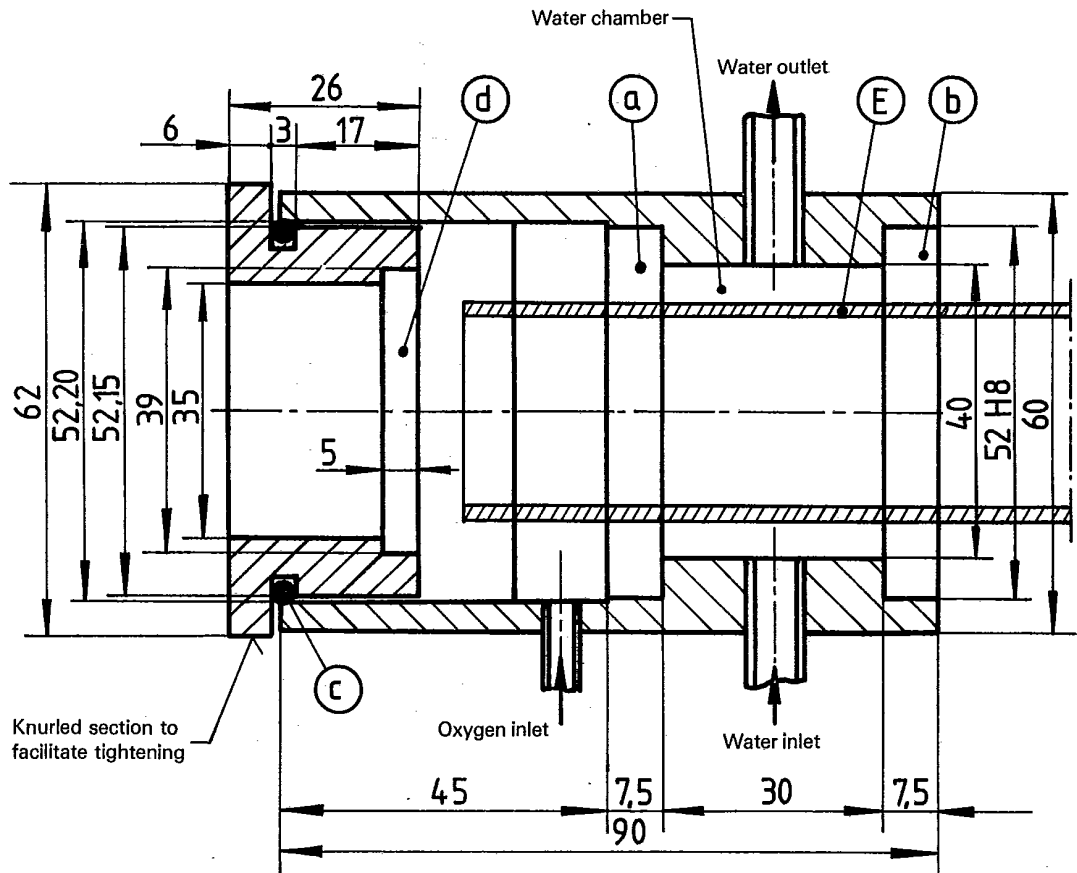


Figure 1 — Example of apparatus for the determination of sulphur (Method after combustion in a current of oxygen)

Dimensions in millimetres



- (a) and (b) = Pressure-tight rubber joints
- (c) = Toroidal joint
- (d) = Coloured glass mounted in epoxy resin
- (E) = Combustion tube

Figure 2 — Water-cooled tube closure

Dimensions in millimetres

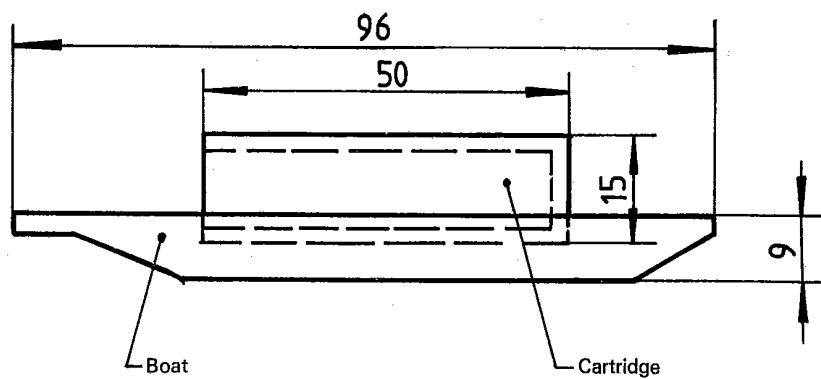


Figure 3 — Boat and cartridge

5.11 Gas outlet tap (K), with 2,5 mm inside diameter, to prevent the absorbent solution from rising into the bubbler tube

- when the furnace is opened in order to introduce the boat and filter cartridge assembly,
- during pre-heating, which causes a partial vacuum in the furnace as a result of the oxidation of the metal.

5.12 Bubbler tube with holes (L), (see figure 4).

5.13 Absorption vessel (M), with a minimum diameter of 35 mm and a height of 140 mm.

5.14 Burette (N), of 10 ml, graduated in 1/20 ml, containing the standard sodium borate solution (4.7).

5.15 Gas outlet pipe (O), which should be as short as possible.

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 apparatus

In order to verify the pressure tightness of the apparatus, the regularity of the combustion and any liberation of sulphur by the refractory materials, carry out preliminary determinations on a mass of steel or cast iron with a known sulphur content. The check of pressure tightness of the apparatus can, however, be made without the boat and cartridge.

NOTE — In these determinations, it will be necessary to obtain a consistent result taking into account the variation inherent in all methods. The difference between the average obtained and the known value of the content of the test portion used should not be significant.

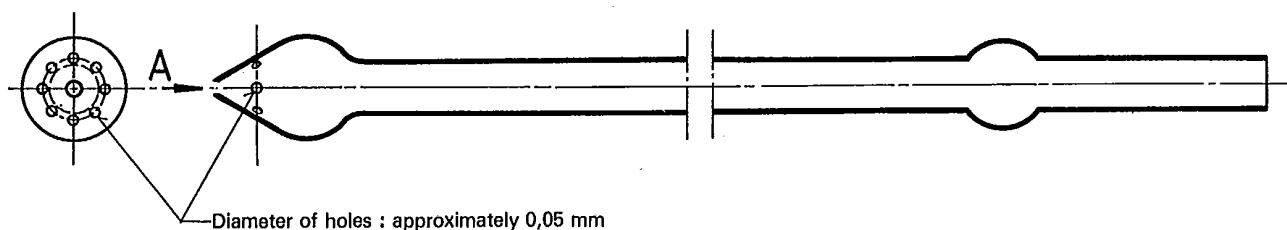


Figure 4 — Bubbler tube with holes

7.2 Test portion

The mass (m) of the test portion in the form of short chips several tenths of a millimetre thick should be

- a) $1 \pm 0,001$ g for presumed sulphur contents less than 0,10 % (m/m);
- b) $0,5 \pm 0,001$ g for presumed sulphur contents between 0,10 and 0,20 % (m/m);
- c) for sulphur contents greater than 0,20 % (m/m), calculate the mass of the test portion so that it contains at the most 1 000 μg of sulphur. To create the same conditions of combustion, add to the test portion a complementary quantity up to 1 g of pure iron (4.4).

7.3 Combustion and determination

7.3.1 Non-refractory steel and cast iron

Bring the electric furnace (D) to the temperature necessary to obtain a minimum temperature of 1 420 °C inside the combustion tube (E). Open taps (F) and (K) and regulate the flow of oxygen (1,2 l/min). In the absorption vessel (M), place sufficient absorbent solution (4.6) to raise the level of the liquid 80 mm above the holes in the bubbler tube (L) (see note 1) and add 4 or 5 drops of the mixed indicator (4.8). Neutralize by means of the standard sodium borate solution (4.7) until the indicator changes colour (greyish-green colouration). Note the burette reading V_1 in millilitres.

Shut off the current of oxygen by closing taps (F) and (K). Open the oxygen inlet device (G), and introduce the boat and filter-cartridge assembly containing the test portion well into the large section of the combustion tube (E). Close oxygen inlet device (G), and adjust the flow of oxygen to 1,2 l/min by opening inlet tap (F) first and, immediately afterwards, outlet tap (K).

Allow the test portion to burn for a period of 3 min, then for a further 4 min during which simultaneously titrate the absorbent solution by means of standard sodium borate solution (4.7) until a steady greyish-green colour of the indicator is obtained (see note 2). Note the burette reading V_2 in millilitres (see note 3).

NOTES

- 1 The liquid height of 80 mm is valid for a flow of oxygen of 1,2 l/min as specified in the procedure.

2 3 min is the minimum period necessary to flush carbon dioxide from the system before commencing titration.

3 The final titration may also be performed by means of instruments (e.g. potentiometric titration).

7.3.2 Refractory steel and cast iron

Since the combustion of these steels and cast irons is difficult to start, it is necessary to add a quantity of flux (4.5). Note that the dimensions of the boat and cartridge assembly are intended to burn a maximum mass of 1,2 g of metal.

8 Expression of results

8.1 Calculation formula

The sulphur (S) content is given, as a percentage by mass, by the formula

$$0,000\ 10 \times \frac{(V_2 - V_1)}{m} \times 100$$
$$= 0,010 \times \frac{(V_2 - V_1)}{m}$$

where

$(V_2 - V_1)$ is the volume, in millilitres, of the sodium borate solution (4.7) used for the titration;

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

0,000 10 is the equivalent, in grams of sulphur, of the sodium borate solution (4.7).

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.