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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 671

CHEMICAL ANALYSIS OF STEEL AND CAST IRON

DETERMINATION OF SULPHUR

(METHOD AFTER COMBUSTION IN A CURRENT OF OXYGEN, AND TITRATION WITH SODIUM BORATE)

1st EDITION
February 1968

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 671, *Chemical analysis of steel and cast iron – Determination of sulphur (Method after combustion in a current of oxygen, and titration with sodium borate)*, was drawn up by Technical Committee ISO/TC 17, *Steel*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1958 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In February 1966, this Draft ISO Recommendation (No. 915) was circulated to all ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

Three Member Bodies opposed the approval of the Draft :

France
Portugal
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 671 February 1968

CHEMICAL ANALYSIS OF STEEL AND CAST IRON

DETERMINATION OF SULPHUR

(METHOD AFTER COMBUSTION IN A CURRENT OF OXYGEN, AND TITRATION WITH SODIUM BORATE)

1. SCOPE

This ISO Recommendation describes methods of combustion and titration for the determination of sulphur in steel and cast iron.

2. PRINCIPLE OF THE METHOD

2.1 Combustion

Metal chips are burnt in a small refractory boat introduced into a tube heated by an electric furnace and through which is passed a current of oxygen of known rate of flow. The temperature of the metal can be attained either by the temperature of the furnace itself (general case) or by the addition of a metal flux (particular case of refractory steels).

2.2 Titration

The gases resulting from combustion, freed from oxide dust, are absorbed by a solution of potassium sulphate and hydrogen peroxide and then titrated with standard sodium borate solution (see Note 8.6).

3. REAGENTS

3.1 *Oxygen.* Minimum purity 99 %, free from sulphur-bearing compounds.

3.2 *Soda asbestos* (particle size : approximately 2 mm) and *magnesium perchlorate* (particle size : approximately 2 mm).

3.3 *Absorbent solution.* Dissolve 5 g of potassium sulphate in 200 ml of boiled and cooled distilled water. Add 100 ml of hydrogen peroxide 100 volumes (30 %) and dilute to 2500 ml with boiled and cooled distilled water.

3.4 *Standard sodium borate solution.* Weigh 2.3839 g of sodium borate, $Na_2B_4O_7.10H_2O$, analytical reagent quality, dissolve in boiled and cooled distilled water and make up the volume to 2000 ml with the same water.

1 ml = 0.010 % of sulphur for 1 g of test sample.

3.5 *Mixed indicator* (see Note 8.1). Dissolve 0.1 g of methylene blue and 0.3 g of methyl red (salt form) in water and dilute to 500 ml with water.

4. APPARATUS

Dimensions in millimetres

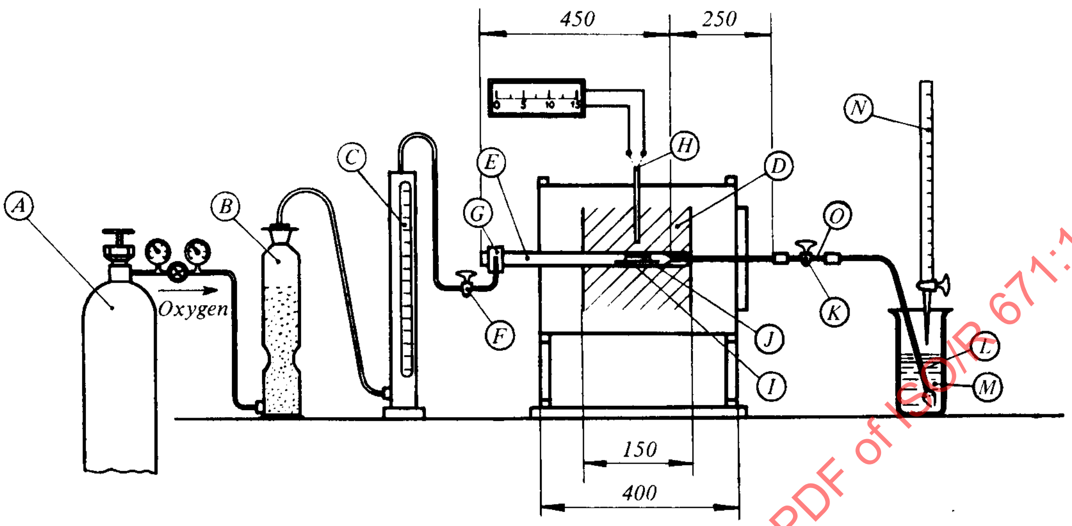


FIG. 1 – **Example of apparatus for the determination of sulphur. (Method after combustion in a current of oxygen)**

A. *Source of oxygen* (3.1) fitted with a sensitive regulating valve and a pressure gauge.

B. *Purifying and drying column* containing soda asbestos and magnesium perchlorate (3.2).

C. *Flow meter* capable of measuring a current of oxygen of 0.2 to 3 litres per minute.

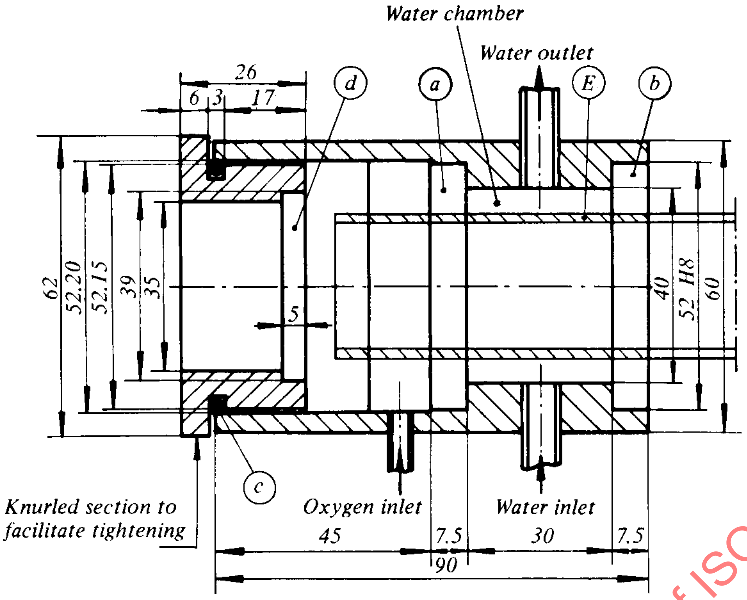
D. *Electric furnace* capable of maintaining the combustion tube (E) at 1420 °C in continuous operation.

E. *Combustion tube* in refractory material capable of ensuring continuous operation at 1450 °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 (see Notes 8.2 and 8.3).

F. *Oxygen inlet tap.*

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

Dimensions in millimetres



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

FIG. 2 – **Water-cooled tube closure**

H. *Platinum/platinum-rhodium thermocouple* 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.

I. *Boat of refractory material* with a wide flat base capable of withstanding two heatings at 1420 °C (length 80 to 100 mm, height 8 to 9 mm, width 15 to 16 mm) (see Fig. 3).

Dimensions in millimetres

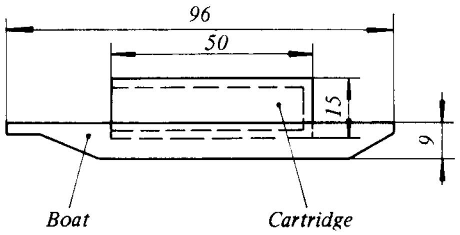


FIG. 3 – **Boat and cartridge**

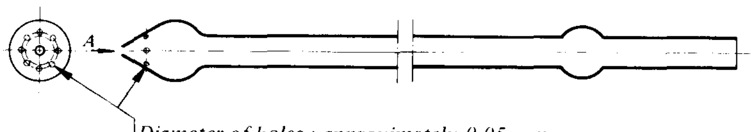
J. *Refractory filter-cartridge* (inside diameter 14 mm, outside diameter 16 mm, length 50 mm) (silica content 12 to 15 %, the remainder being alumina). The porosity of the cartridge is defined by the flow of gas through the whole surface at a given pressure : 4 to 5 litres of gas per minute at a pressure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

Boats and cartridges should be calcined at 1420 °C for 10 minutes in a current of oxygen, and should be kept in a desiccator after calcination.

K. *Gas outlet tap* 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.

L. *Bubbler tube with holes* (see Fig. 4)



Diameter of holes : approximately 0.05 mm

FIG. 4 – **Bubbler tube with holes**

M. *Absorption vessel* with a minimum diameter of 35 mm and a height of 140 mm.

N. *Burette* of 10 ml, graduated in 1/20 ml, containing the standard sodium borate solution (3.4).

O. *Gas outlet pipe* which should be as short as possible.

5. SAMPLING

In accordance with ISO Recommendation R 377, *Selection and preparation of samples and test pieces for wrought steel*, or the appropriate national standards for cast iron.

6. PROCEDURE

6.1 Preparation of the test sample

Insert the test sample in the form of short chips several tenths of a millimetre thick into the cartridge J. The mass (m) of the test sample should be

1 ± 0.001 g for assumed sulphur contents less than 0.10 %
0.5 ± 0.001 g for assumed sulphur contents between 0.10 and 0.20 %.

For sulphur contents greater than 0.20 %, calculate the mass of the test sample so that it contains at the most 1000 µg of sulphur. To create the same conditions of combustion, add to the test sample a complementary quantity up to 1 g of iron with a known low sulphur content.

6.2 Control test

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.

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 sample used should not be significant.