UDC 669.14

Ref. No.: ISO/R 437 - 1965 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 437

CHEMICAL ANALYSIS OF STEELS
DETERMINATION OF TOTAL CARBON

(GRAVIMETRIC METHOD AFTER COMBUSTION
IN A STREAM OF OXYGEN)

1st EDITION
May 1965

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

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 437, *Chemical Analysis of Steels—Determination of Total Carbon (Gravimetric Method after Combustion in a Staeam of Oxygen)*, 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 1957, and led in 1962, to the adoption of a Draft ISO Recommendation.

In February 1963, this Draft ISO Recommendation (No. 528) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Australia	India	Romania
Austria	Iran	Spain
Canada	Italy	Sweden
Chile	Japan	Switzerland
Czechoslovakia	Netherlands	Turkey
France	Norway	U.A.R.
Denmark	Poland	United Kingdom
Finland	Portugal	U.S.S.R.
Hungary	Republic of South Africa	Yugoslavia

Three Member Bodies opposed the approval of the Draft:

Belgium
Germany
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in May 1965, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 437 May 1965

CHEMICAL ANALYSIS OF STEELS
DETERMINATION OF TOTAL CARBON

(GRAVIMETRIC METHOD AFTER COMBUSTION IN A STREAM OF OXYGEN)

1. SCOPE

1.1 The purpose of this ISO Recommendation is to describe the gravimetric method of determining total carbon in steel after combustion of the sample in a stream of oxygen.

1.2 The method is applicable to carbon contents of not less than 0.10 per cent.

2. PRINCIPLE OF THE METHOD

2.1 The sample is heated to combustion at high temperature (1200 to 1350 °C) in a stream of pure oxygen and the carbon is converted to carbon dioxide. If it is necessary a flux may be added.

2.2 The carbon dioxide, carried by oxygen, is passed through and absorbed by soda asbestos in a tared bulb, the increase in mass of which is proportional to the quantity of carbon dioxide formed.

$$C + O_2 = CO_2$$

$$2\ NaOH + CO_2 = Na_2CO_3 + H_2O$$

3. REAGENTS

3.1 *Ogygen*, at least 99 per cent purity.

3.2 *Anhydrous magnesium perchlorate*, $Mg(ClO_4)_2$.*

3.3 *Fluxes:* lead dioxide, analytical reagent grade; copper oxide, tin, pure iron, etc.

3.4 *Manganese dioxide or silver orthovanadate*, prepared as follows:

3.4.1 *Manganese dioxide.* When a suitable chemically active grade is not available, it may be prepared as follows:

To prepare about 50 g of manganese dioxide, dissolve in a 4-litre beaker 200 g of manganous sulphate $MnSO_4$, 4 H_2O in 2.5 litres of demineralized water. After making this solution clearly ammoniacal, add 1 litre of ammonium persulphate solution (225 g per litre) freshly prepared, bring the whole to boiling point. Continue boiling for 10 min and add ammonia 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 or 4 litres 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 filled with a thin layer of purified asbestos pulp (in place of the latter, it is also permitted to use a porcelain funnel of the Buchner type).

* Contact with organic substances should be avoided because of the potential hazard of an explosion.

After the last washing, transfer the manganese dioxide onto the filter and wash with warm water until it is freed from ion sulphates. 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 will pass through a sieve with apertures of 0.8 mm and dry again completely at 105 °C.

3.4.2 *Silver orthovanadate*

Reaction: $Na_3VO_4 + 3\ AgNO_3 \rightarrow Ag_3VO_4 + 3\ NaNO_3$

Dissolve 60 g of sodium orthovanadate in 400 ml of demineralized water. Boil for 15 min. Also dissolve 170 g of silver nitrate in 200 ml of demineralized water.

Add the silver nitrate solution dropwise to the warm solution of sodium orthovanadate. An abundant yellow orange precipitate should form.

Filter on a Buchner funnel and wash with water until free from positive silver ions (Ag^+), verify with negative chloride ions (Cl^-).

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

Grind and store protected from light.

NOTE. — Ammonium orthovanadate is not sufficiently soluble for it to be specified.

3.5 *Soda asbestos*, asbestos impregnated with sodium hydroxide, in granules of about 2 mm diameter. Avoid contact with air.

4. 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.

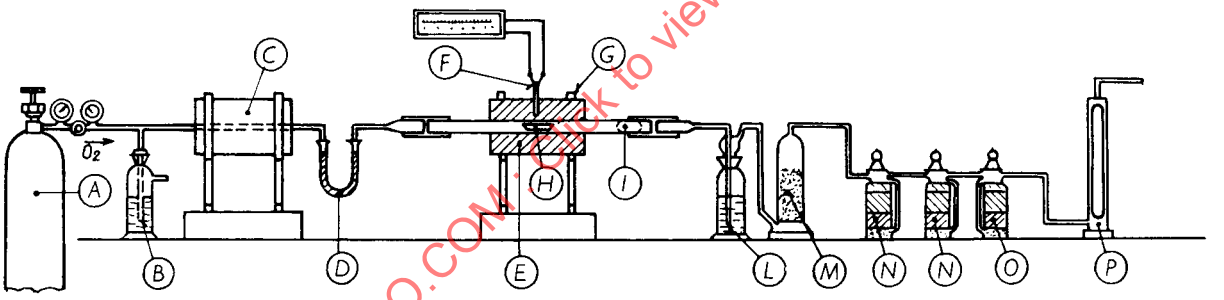

FIG. 1

A Source of oxygen (3.1) with pressure regulating valve.

B Mercury valve.

C Wire-wound furnace with non-porous porcelain combustion tube containing platinized asbestos heated to 625 °C.

D Unit for drying and purifying the oxygen, containing anhydrous magnesium perchlorate (3.2) and soda asbestos (3.5) separated by glass wool (diameter of tubes 25 mm, height 100 mm approximately) connected by tubing.

E Wire-wound or resistor rod furnace, made of carborundum or metal and capable of a temperature for combustion up to 1350 °C.*

F Thermocouple 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 tube temperature and the pyrometer readings will have been established.

* Induction furnaces may also be used.