UDC 662.6/.7:620.1

Ref. No.: ISO/R 609 - 1967 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 609

DETERMINATION OF CARBON AND HYDROGEN IN COAL AND COKE BY THE HIGH TEMPERATURE COMBUSTION METHOD

1st EDITION
June 1967

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

COPYRIGHT RESERVED

The copyright of ISO Recommendations and ISO Standards belongs to ISO Member Bodies. Reproduction of these documents, in any country, may be authorized therefore only by the national standards organization of that country, being a member of ISO.

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.

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

BRIEF HISTORY

The ISO Recommendation R 609, *Determination of Carbon and Hydrogen in Coal and Coke by the High Temperature Combustion Method,* was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1950 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In October 1963, this Draft ISO Recommendation (No. 605) 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:

Argentina
Australia
Austria
Belgium
Bulgaria
Canada
Chile
Czechoslovakia
Denmark
Germany
India
Italy
Japan
Netherlands
New Zealand
Poland
Portugal
Republic of South Africa
Romania
Spain
Switzerland
U.A.R.
United Kingdom
U.S.A.

Three Member Bodies opposed the approval of the Draft:

France
Turkey
U.S.S.R.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in June 1967, to accept it as an ISO RECOMMENDATION.

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

ISO Recommendation R 609 June 1967

DETERMINATION OF CARBON AND HYDROGEN IN COAL AND COKE BY THE HIGH TEMPERATURE COMBUSTION METHOD

1. SCOPE

This ISO Recommendation describes the high temperature combustion method of determining the total carbon and the total hydrogen in hard coal, brown coal and lignite, and coke; the results include the carbon in the carbonates and the hydrogen combined in the moisture and in the water of hydration of silicates. A determination of moisture is carried out at the same time, and an appropriate correction is applied to the hydrogen value obtained by combustion. A determination of carbon dioxide may also be made and the total carbon value corrected for the presence of mineral carbonates.

2. PRINCIPLE

The sample is burned in a rapid current of oxygen at a high temperature in an impervious tube; all the hydrogen is converted to water and all the carbon to carbon dioxide. These products are absorbed by suitable reagents and determined gravimetrically. Oxides of sulphur and chlorine are retained by a silver gauze roll at the outlet end of the tube.

3. REAGENTS

All reagents should be of analytical reagent quality. Distilled water should be used throughout.

3.1 **Magnesium perchlorate,** anhydrous (anhydrone), less than 1.2 mm in size and preferably within the size range 1.2 to 0.7 mm.

NOTE. — Regeneration of the magnesium perchlorate should not be attempted, owing to the risk of explosion. When exhausted, the magnesium perchlorate should be washed down the sink with a current of water.

3.2 **Soda asbestos,** preferably of coarse grading, e.g. 3.0 to 1.5 mm, and not finer than the grading 1.2 to 0.7 mm, and preferably of self-indicating type.

3.3 **Alumina,** finely divided, approximately 0.1 mm in size.

3.4 **Sodium borate solution,** 0.05 N.

3.5 **Hydrogen peroxide,** 1% neutral solution.

3.6 **Pure silver gauze,** approximately 10 meshes per centimetre, made of wire approximately 0.3 mm in diameter.

3.7 **Oxygen,** hydrogen-free. The oxygen should preferably be prepared from liquid air and not by electrolysis. Electrolytically prepared oxygen should be passed over red-hot copper oxide before use, to remove any trace of hydrogen.

4. APPARATUS

4.1 **Two purification trains,** one for absorbing water vapour and carbon dioxide from the oxygen used for the combustion and the other for similarly treating the air used for sweeping out the absorption train before and after a determination. Assemble each train using the following reagents in the order stated:

(1) Magnesium perchlorate (3.1) for absorbing water,
(2) Soda asbestos (3.2) for absorbing carbon dioxide,
(3) Magnesium perchlorate (3.1) for absorbing the water evolved in the reaction between carbon dioxide and soda asbestos.

The purification trains should be large enough to render frequent recharging unnecessary even with continuous use.

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

4.2 Combustion device

4.2.1 *A furnace,* capable of carrying a tube approximately 25 mm outside diameter and heating it over a length of approximately 12.5 cm to over 1250 °C and up to a maximum of 1350 °C. A temperature-distribution curve for a typical furnace is shown in Figure 1. Suitable furnaces are, for example,

(*a*) molybdenum or tungsten wire wound,

(*b*) platinum or platinum-rhodium wire wound,

(*c*) heated by silicon carbide rods.

Type (*c*) has the lowest initial cost and has proved satisfactory in use.

NOTE. — Furnaces of the type normally used for the determination of carbon or sulphur in steel are not suitable because of the absence of the auxiliary section to maintain the silver gauze roll at the correct temperature.

4.2.2 *A combustion tube,* approximately 20 mm internal diameter and approximately 70 cm long, made of refractory aluminous porcelain which is not permeable to gases at 1400 °C.

4.2.3 *A combustion boat* of iron-free unglazed porcelain, approximately 6 cm long and 12.5 mm wide and 10 mm deep, capable of withstanding a temperature of 1350 °C.

NOTE. — Boats should not blister, discolour or change in mass on heating in oxygen at 1350 °C for 3 hours. A suitable boat lasts for about 10 to 20 determinations and should then be discarded because of the accumulation of fused ash. For high ash coals, it may be convenient to line the boat with alumina before adding the sample, in order to prevent fusion of the ash to the boat.

4.3 **An absorption train,** for absorbing the water and carbon dioxide evolved by the combustion of the sample. Midvale tubes (Fig. 2), which provide a large area of reaction, are used in order to reduce the back-pressure in the apparatus and so obviate the danger of leakage through the rubber sleeve carrying the pusher. Assemble the train using the following reagents in the order stated:

(1) Magnesium perchlorate (3.1) for absorbing the water evolved during the combustion,

(2) Soda asbestos (3.2) for absorbing carbon dioxide,

(3) Magnesium perchlorate (3.1) for absorbing the water evolved in the reaction between carbon dioxide and soda asbestos.

Cotton wool is placed above and below the absorbents to prevent carryover of dust by the rapid flow of oxygen and the cracking of the soda asbestos tube by the heat of reaction. A typical absorption train with details of the packing is shown in Figure 3, page 12. *A* is the absorber for water; *B* and *C* are the absorbers for carbon dioxide, *C* serving as a control to indicate when the packing in *B* is in need of replacement. Any water released in *B* by the reaction between soda asbestos and carbon dioxide is absorbed in *C*.

4.4 **Two flowmeters,** one capable of measuring rates of flow up to 300 ml per minute and the other to 250 ml per minute.

4.5 **A U-gauge** to measure the resistance of the system

NOTE. — The normal back-pressure on the system is 50 to 70 mmH$_2$O.

4.6 **A heat-resisting stopper** (acrylonitrile or chloroprene) for connecting the absorption train to the combustion tube.

4.7 **A silica pusher** with a disk end 12 mm in diameter for pushing the boat into the furnace. It is about 45 cm long, made from 6 mm diameter silica rod flattened at one end, or from a 6 mm diameter silica tube sealed and flattened similarly.

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

The pusher passes loosely through a glass T-piece, one end of which fits into the rubber stopper which closes the inlet end of the combustion tube, the other being sealed with a rubber sleeve*, through which the pusher slides. Oxygen is admitted through the limb of the T-piece. The pusher is marked from the disk end for convenience in ascertaining the position of the boat in the combustion tube during pushing.

4.8 **Balance** sensitive to 0.1 mg.

5. PREPARATION OF APPARATUS

5.1 **Preparation of the combustion tube.** Insert the combustion tube into the furnace so that it projects 7.5 cm at the exit end. Wrap this projecting portion with asbestos string to prevent condensation of water in the tube. Insert the rubber stopper carrying the silica pusher at the inlet end of the combustion tube and connect the purified oxygen supply to the limb of the glass T-piece.

5.2 **Preparation and location of the silver gauze roll.** The roll of silver gauze (3.6), through which passes a stout silver wire provided with a loop for convenience of withdrawal, should be 7.5 cm long and of sufficient diameter to ensure a close sliding fit in the combustion tube. Insert the silver gauze roll into the exit end of the combustion tube to a distance of about 7.5 cm. With the furnace at its working temperature of 1350 °C, connect a bubbler containing a 1% solution of the hydrogen peroxide (3.5) to the exit end of the combustion tube by means of a heat-resisting stopper. Burn 0.5 g of a coal containing 1 to 2% of sulphur by the procedure described below. Determine the amount of oxides of sulphur passing the silver gauze roll by titration of the hydrogen peroxide with the sodium borate (3.4). Move the silver gauze roll in 5 mm stages and repeat the test until a position is found where the volume of the sodium borate (3.4) corresponding to the end point is not more than 0.2 ml, which will represent at least 99% retention of the oxides of sulphur by the silver gauze roll. Note its exact location.

NOTE. — The silver gauze roll can absorb sulphur equivalent to 7% of the sample; for higher sulphur contents, the mass of the coal sample should be proportionally reduced. The roll should be periodically cleaned by boiling in water (to remove silver sulphate), in concentrated ammonia solution (to remove silver chloride) and finally in water, after which it should be dried. The total sulphur content of samples examined between two cleanings of the roll should not exceed 0.035 g.

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

6. PROCEDURE

Raise the furnace to its working temperature of 1350 °C and confirm that the silver gauze roll is in its correct position. Admit the oxygen (3.7) to the apparatus about 30 minutes before the temperature reaches 1350 °C and adjust the rate of flow to 300 ml/min. Before commencing the determination, mix the air-dried sample of coal or coke, ground to pass a sieve of 0.2 mm aperture, thoroughly for at least 1 minute, preferably by mechanical means. A small portion of the sample should be re-exposed to the air for a few hours and again mixed before weighing out the quantity required for the determination.

A determination of moisture should be made immediately on the remainder of this small sample.

Spread about 0.5 g of the sample evenly over the bottom of a dried boat and weigh accurately. Cover with about 0.5 g of the alumina (3.3) (with care, 0.5 g of alumina can be made to blanket completely the sample in the boat). Connect a previously weighed absorption train and insert the charged boat into the combustion tube to a position such that its centre is 22.5 cm from the centre of the hottest zone. With the silica pusher fully withdrawn, replace the rubber stopper and continue to pass the oxygen (3.7) at a rate of 300 ml/min. At the end of each of the next six one-minute periods push the boat forward about 3.75 cm, withdrawing the silica pusher each time to prevent distortion; after the last push the centre of the boat should be at the centre of the hottest zone (see Note 1, page 8).

---

* This sleeve should be changed periodically to avoid loss by leakage

Allow the boat to remain in the hottest zone for a further 4 minutes. Disconnect the absorption train, connect it to the purification train and purge it for 10 minutes with purified air at a rate of 200 to 250 ml/min. Disconnect the absorbers *A*, *B* and *C*, close the taps and stopper the central side-arms and allow to cool (see Note 2 below).

Wipe and remove the guard stoppers and weigh the absorbers (see Note 3 below).

A bent nickel-chromium wire can be used to withdraw the heated boat, which is conveniently pulled on to a thick sheet of asbestos.

NOTES

1. Certain coals which liberate volatile matter at a high rate in the early stages of heating may give a carryover of carbon particles. For such coals, the rate of pushing should be reduced according to the following procedure:

   Insert the charged boat so that its centre is 22.5 mm from the centre of the hottest zone. At the end of the first minute, move the boat forward 38 mm. At the end of each of the next 8 minutes move the boat forward by 19 mm, and at the end of the tenth minute move the boat forward by 38 mm. Allow the boat to remain in the hottest zone for a further 4 minutes.

   For unreactive cokes, e.g. those with less than 0.5% of volatile matter, the rate of pushing should also be reduced as described above.

2. Midvale tubes used for the absorption of carbon dioxide cool slowly and 60 minutes should be allowed before weighing.

3. When not connected to the apparatus, the Midvale tubes should be protected from atmospheric contamination by closing the taps and fitting guard stoppers of plugged rubber tubing to the open limbs. It is usual to weigh the absorption tubes without guard stoppers, after wiping.

7. CALCULATION AND REPORTING OF RESULTS

If

m_1 = mass of sample taken, expressed in grammes;
m_2 = increase in mass of absorbers *B* and *C*, expressed in grammes;
m_3 = increase in mass of absorber *A*, less the moisture in the alumina used (see Note page 9), expressed in grammes;
M = moisture in the sample as analysed, expressed as a percentage;
CO_2 = carbon dioxide in the sample as analysed*, expressed as a percentage;
C = total carbon in the sample as analysed, expressed as a percentage;
C_1 = total carbon less that present as carbon dioxide, expressed as a percentage;
H = total hydrogen in the sample as analysed, expressed as a percentage;
H_1 = total hydrogen less that present as moisture, expressed as a percentage;

then

$$\mathrm{C} = 27.29\, m_2/m_1$$
$$\mathrm{C}_1 = \mathrm{C} - 0.273\, \mathrm{CO}_2$$
$$\mathrm{H} = 11.19\, m_3/m_1$$
$$\mathrm{H}_1 = \mathrm{H} - 0.1119\, M$$

The results (preferably the mean of duplicate determinations, see section 8) should be reported to the nearest 0.1 per cent for the carbon determination and to the nearest 0.01% for the hydrogen determination. Calculation of the results to other bases is being studied** and will be the subject of a separate ISO Recommendation.

---

* See ISO Recommendation R ..., *Determination of Carbon Dioxide in Coal,* at present at the stage of draft proposal.

** By Technical Committee ISO/TC 27, *Solid Mineral Fuels.*

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967

NOTE. — The total moisture in the alumina is determined by heating 1 g to 1350 °C in the apparatus and absorbing the water in magnesium perchlorate (3.1).

Drying at 105 °C is unsuitable, since the water in the alumina is not liberated below 1000 °C. Only one or two determinations of moisture need be carried out on each bottle of alumina (3.3).

8. PRECISION OF DETERMINATION

	Maximum acceptable differences between results obtained	
	in the same laboratory	in different laboratories
carbon	0.25% absolute	0.5% absolute
hydrogen	0.12% absolute	0.25% absolute

8.1 In the same laboratory

The results of duplicate determinations carried out at different times in the same laboratory by the same operator with the same apparatus on representative portions taken from the same analysis sample should not differ by more than the above value.

8.2 In different laboratories

The means of the results of duplicate determinations carried out in each of two different laboratories on representative portions taken from the same analysis sample, should not differ by more than the above value.

STANDARDSISO.COM : Click to view the full PDF of ISO/R 609:1967