INTERNATIONAL STANDARD

ISO 609

Second edition
1996-02-15

Solid mineral fuels — Determination of carbon and hydrogen — High temperature combustion method

Combustibles minéraux solides — Dosage du carbone et de l'hydrogène — Méthode par combustion à haute température

Reference number
ISO 609:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 609 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 609:1975), which has been technically revised.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

An alternative method to that specified in this International Standard is given in ISO 625:1996, *Solid mineral fuels — Determination of carbon and hydrogen — Liebig method.*

Solid mineral fuels — Determination of carbon and hydrogen — High temperature combustion method

1 Scope

This International Standard specifies a method of determining the total carbon and the total hydrogen in hard coal, brown coal and lignite, and coke, by a high temperature combustion method.

The results include the carbon in the carbonates and the hydrogen combined in the moisture and in the water of constitution 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 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method.*

ISO 687:1974, *Coke — Determination of moisture in the analysis sample.*

ISO 925:1980, *Solid mineral fuels — Determination of carbon dioxide content — Gravimetric method.*

ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases.*

ISO 1988:1975, *Hard coal — Sampling.*

ISO 2309:1980, *Coke — Sampling.*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method.*

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis.*

3 Principle

A known mass of coal or coke is burnt in a stream of oxygen, at a temperature of 1 350 °C, in a tube impervious to gases. All the hydrogen is converted to water and all the carbon to carbon dioxide. These products are absorbed by suitable reagents and determined gravimetrically. The chlorine and oxides of sulfur which are formed are retained by a silver gauze roll at the outlet end of the tube.

4 Reagents and materials

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

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

4.1 Magnesium perchlorate, anhydrous, less than 1,2 mm in size and preferably within the size range 1,2 mm to 0,7 mm.

WARNING — Due regard must be taken of local regulations when disposing of exhausted magnesium perchlorate. Regeneration of magnesium perchlorate must not be attempted, owing to the risk of explosion.

4.2 Sodium hydroxide on an inert base, preferably of a coarse grading, for example 3,0 mm to 1,5 mm, but not finer than the grading 1,2 mm to 0,7 mm, and preferably of the self-indicating type.

4.3 Aluminium oxide (alumina), finely divided, approximately 0,1 mm in size.

4.4 Sodium tetraborate, standard volumetric solution, $c(Na_2B_4O_7) = 0{,}025$ mol/l.

Dissolve 9,534 2 g of sodium tetraborate decahydrate in water and dilute to 1 litre. Mix thoroughly.

4.5 Hydrogen peroxide, approximately 30 % (*m/m*).

4.6 Pure silver gauze, of mesh approximately 1 mm, made of wire approximately 0,3 mm in diameter.

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

4.8 Mixed indicator solution

4.8.1 Solution A

Dissolve 0,125 g of 2-(4-dimethylaminophenylazo)benzoic acid, sodium salt (methyl red) in 100 ml of water.

4.8.2 Solution B

Dissolve 0,083 g of 3,7-bis(dimethylamino)phenothiazine-5-ylium chloride (methylene blue) in 100 ml of water. Store in a dark bottle.

4.8.3 Mixed solution

Mix equal volumes of solution A and solution B. Store in a dark bottle. Discard the mixed solution after 1 week.

4.9 Air, compressed.

4.10 Glass wool.

5 Apparatus

5.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.2 Graduated glassware, conforming to the requirements for class A in the International Standards prepared by ISO/TC 48, *Laboratory glassware and related apparatus*.

5.3 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 a series of U-tubes or Midvale tubes containing the following reagents in the order stated, in the direction of flow:

a) magnesium perchlorate (4.1) for absorbing water;

b) sodium hydroxide on an inert base (4.2) for absorbing dioxide;

c) magnesium perchlorate for absorbing the water evolved in the reaction between carbon dioxide and sodium hydroxide.

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

NOTE 1 Midvale tubes that have been freshly packed with absorbent and used in the purification train are thereby conditioned for subsequent use in the absorption train.

5.4 Combustion assembly

5.4.1 Heating unit, an electrically heated furnace or furnaces, designed to carry a combustion tube (5.4.2) and heat it to 1 350 °C over a distance of 125 mm in the hot zone, and yield a temperature-distribution profile similar to that shown in figure 1. The heating unit normally requires an auxiliary furnace to ensure that the silver gauze roll (5.9) is maintained at the correct temperature (approximately 600 °C to 800 °C). Suitable furnaces are, for example:

a) molybdenum or tungsten wire wound;

b) platinum or platinum-rhodium wire wound;

c) heated by silicon carbide rods.

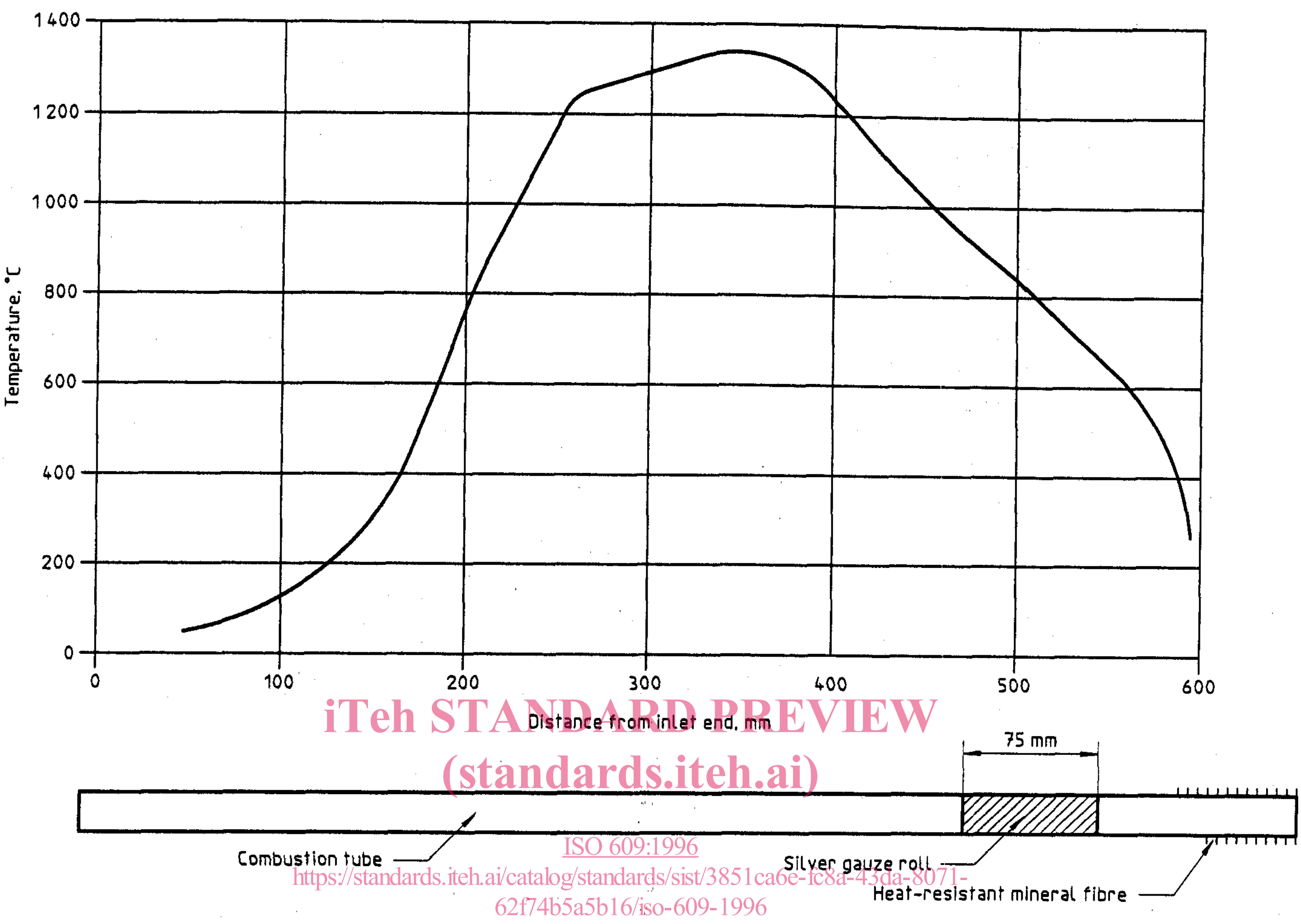


Figure 1 — Typical temperature-distribution curve for furnace

NOTES

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

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

5.4.2 Combustion tube, of approximately 28 mm external diameter, 3 mm wall thickness and 650 mm length, made of refractory aluminous porcelain which is impervious to gases up to a temperature of 1 400 °C. The end of the combustion tube shall be lagged with a suitable heat-resistant mineral fibre to prevent condensation in the tube.

5.4.3 Combustion boat, of iron-free, unglazed porcelain, approximately 60 mm long, 12,5 mm wide and 10 mm deep, for hard coal and coke samples and approximately 75 mm long, 15 mm wide and 10 mm deep for brown coal and lignite, capable of withstanding a temperature of 1 350 °C.

NOTE 4 Combustion boats should not blister, discolour or change in mass on heating in oxygen at 1 350 °C for 3 h. A suitable boat lasts for about 10 to 20 determinations and should then be discarded because of the accumulation of fused ash. For coals with a high ash content, 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.

5.5 Absorption train, for absorbing the water and carbon dioxide evolved by the combustion of the sample. Midvale tubes (figure 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, in the direction of flow:

a) magnesium perchlorate (4.1) for absorbing the water evolved during the combustion;

Dimensions in millimetres

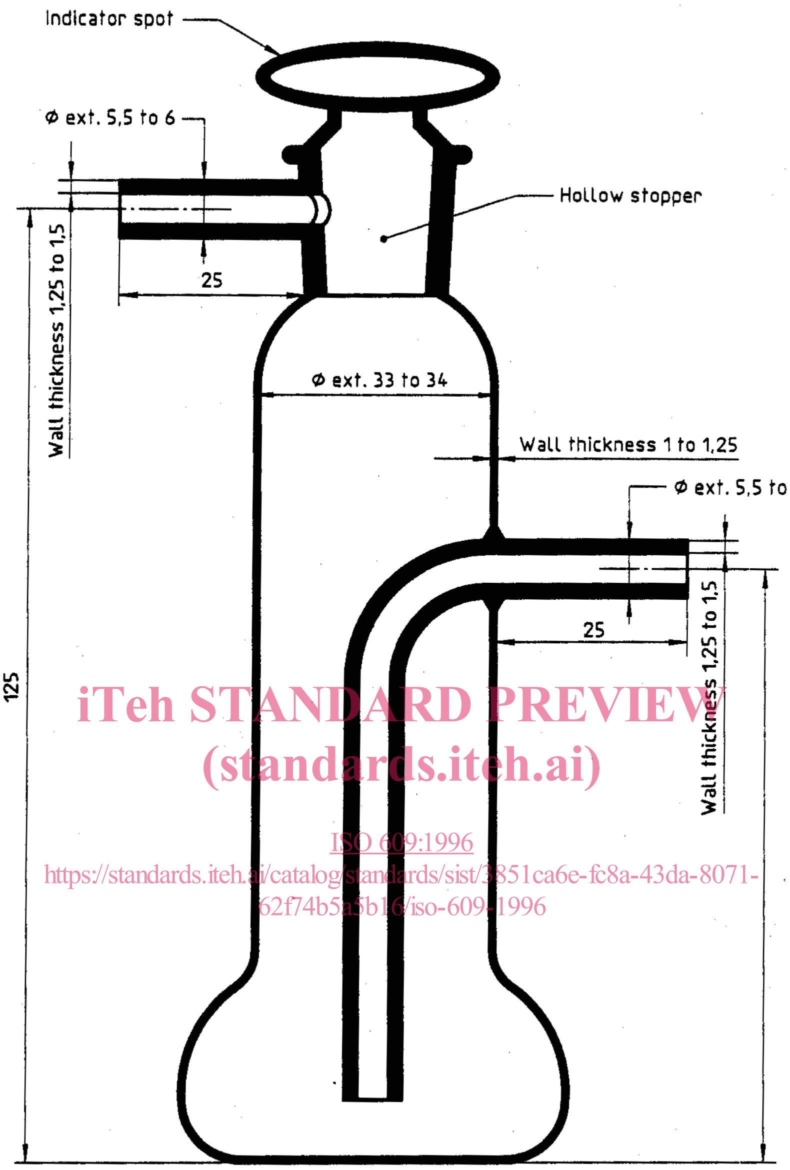


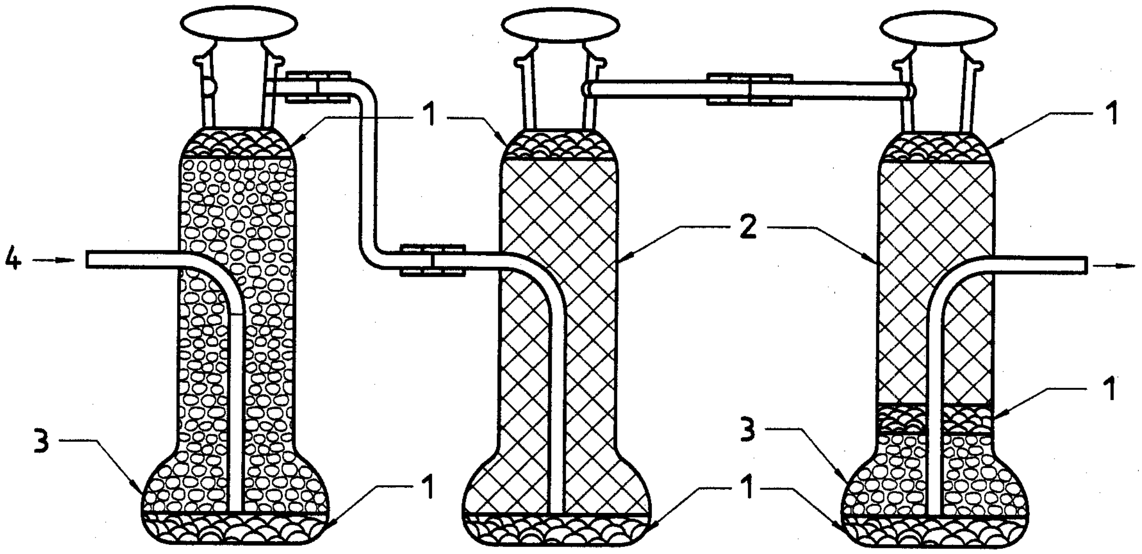
Figure 2 — Midvale tube

b) sodium hydroxide on an inert base (4.2) for absorbing carbon dioxide;

c) magnesium perchlorate for absorbing the water evolved in the reaction between carbon dioxide and sodium hydroxide.

Place glass wool (4.10), previously dried at 105 °C for 1 h, above and below the absorbents to prevent the carry-over of dust by the flow of oxygen, and to prevent the cracking of the Midvale tube by the heat of reaction. A typical absorption train with details of the packing is shown in figure 3. 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 sodium hydroxide and carbon dioxide is absorbed in C.

The inlet of the water-absorption tube shall be inserted through the heat-resistant stopper so that it is flush with the inner end of the stopper. All connections between the tubes shall be made of glass-to-glass, using the rubber connecting sleeves merely as seals.



Key

1 Glass wool

2 Sodium hydroxide (see 4.2)

3 Magnesium perchlorate, 1,2 mm to 0,7 mm size

4 Inlet

Figure 3 — Absorption train

5.6 Two flowmeters, both capable of measuring rates of flow up to 300 ml/min of air.

5.7 Heat-resistant stopper (acrylonitrile or chloroprene) for connecting the absorption train to the combustion tube.

5.8 Silica or steel pusher, made from rod or sealed tube, approximately 6 mm in diameter and 450 mm long, with a disc end, 12 mm in diameter, for pushing the boat into the hot zone of the combustion tube. The pusher passes loosely through a glass or metal T-piece, one end of which fits into the stopper which closes the inlet end of the combustion tube, the other being sealed by a rubber sleeve (see note 6), through which the pusher slides. The sleeve prevents the escape of oxygen which enters at the stem of the T-piece. The pusher is usually marked from the disc end, for convenience in ascertaining the position of the boat in the combustion tube during pushing.

NOTE 5 The rubber sleeve should be changed periodically to avoid leakage.

5.9 Silver gauze roll, for absorbing chlorine and oxides of sulfur. Roll the pure silver gauze (4.6) to form a plug, 75 mm long and of sufficient diameter to ensure a close sliding fit in the combustion tube (5.4.2). A stout, pure silver wire is passed through the centre of the roll to facilitate its removal from the tube.

5.10 Bubbler, for use when determining the correct location for the silver gauze roll (see 6.2). It may be a large boiling tube, wide-necked bottle, or Dreschel bottle, containing a sintered glass disc of 0,015 mm to 0,040 mm maximum pore size in the gas distribution tube. The diameter of the absorber shall be such that the disc is covered to a depth of at least 90 mm by the absorbing solution; a vessel 35 mm in diameter and 150 mm deep is suitable. A silica adapter, or the combustion tube fitted with a tubulure, is connected to the bubbler.

To avoid leakage at the rubber sleeve of the inlet end due to the resistance of the sintered glass bubbler, the bubbler is connected to a suction-pump through a pressure regulator adjusted to maintain slight suction at the inlet end of the combustion tube.