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Coal — Determination of caking power — Gray-King coke test

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National foreword

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Charbon — Détermination du pouvoir agglutinant — Essai Gray-King

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Foreword

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The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 05, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 502:1982), which has been technically revised.

Introduction

The purpose of the Gray-King coke test, which is one of the parameters adopted for the International Classification of Hard Coal by Type by the United Nations Economic Commission for Europe, is to assess the caking properties of a type of coal or a blend of coals by carbonizing under standard conditions.

Although the Gray-King test and the Roga test both assess the caking properties of a coal, they do not measure precisely the same parameters and are not recommended to be regarded as alternative methods.

Coal — Determination of caking power — Gray-King coke test

1 Scope

This International Standard specifies a method of assessing the caking power of coal under standard conditions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1014, *Coke — Determination of true relative density, apparent relative density and porosity*

3 Principle

The sample is heated under standard conditions to a final temperature of 600 °C. The coke residue obtained is classified by reference to a series of standard residues. If the coke residue produced is so swollen that it fills the cross-section of the retort tube, the determination is repeated with the coal admixed with a suitable quantity of electrode carbon or equivalent material. For these highly swelling coals, the Gray-King coke type is defined by the minimum amount of electrode carbon required to produce a strong hard coke residue of the same volume as the original coal and electrode carbon mixture.

4 Reagent

4.1 Standard electrode carbon (see 10.1)

High temperature electrode carbon:

Moisture	less than 1 %
Volatile matter	less than 1,5 %
Ash	less than 5 %
Bulk density at 25 °C (see Annex A)	1,00 g/cm^3 to 1,05 g/cm^3
Relative density at 25 °C (see 10.2)	2,05 to 2,09

Size analysis:

Retained on 212 µm test sieve	nil
Through 212 µm test sieve, retained on 125 µm test sieve	less than 26 %
Through 125 µm test sieve, retained on 63 µm test sieve	10 % to 40 %
Through 63 µm test sieve	50 % to 85 %

It is recommended that a dust mask is used while using the inert carbonaceous material which can contain undesirable trace elements from the original processing of this material.

5 Apparatus

5.1 Furnace, horizontal electric, 50 mm internal diameter and 300 mm long, with one end closed and the other carrying a plug of insulating material which is bored centrally with a hole 25 mm in diameter. The winding of the furnace shall be such that the middle 200 mm is at a uniform temperature within ±5 °C at both 300 °C and 600 °C. Alternatively, the furnace may be constructed from an electrically-heated aluminium-bronze block, with one or several, bores of 25 mm diameter. The furnace shall be insulated and located in a cover of metal or other suitable material, and shall be equipped with a suitable thermocouple, lying above the retort tube when the latter is in position and with the junction at the centre of the furnace. An indicator shall be provided for showing the furnace temperature with an accuracy of ±5 °C. A suitable means of controlling the energy input shall also be provided to permit an increase in temperature at a rate of 5 °C/min. A multiple tube furnace to allow simultaneous determinations is convenient. The furnace may be of the fixed type or mounted on rails. Suitable furnaces are shown in Figure 2 and Figure 3.

5.2 Retort tube (see Figure 4), a heat-resistant glass or transparent silica tube, 20 mm internal diameter and 300 mm long, closed at one end, with a side arm, 8 mm internal diameter and 50 mm long, sealed in at a distance of about 20 mm from the open end. The tube shall be smooth and either of uniform bore or with a slight taper (19 mm to 21 mm) such that the open end is the larger.

5.3 Distance rod, with a flat disk at one end to assist in the packing of the coal and to indicate the free end of the coal sample in the retort tube.

5.4 Receiver and outlet tube

A glass vessel of adequate size, suitably supported and attached to the side arm of the retort tube, fitted with an outlet tube leading to atmosphere or to a piece of small bore silica tubing at the end of which the gas leaving the receiver can be burned through Bunsen burner (in a fume cabinet) to ensure toxic fumes are burnt before venting to the atmosphere through the fume cabinet.

The receiver may conveniently be a U-tube which can be immersed in water.

The outlet shall be open to the atmosphere to prevent pressure build-up.

Fumes are toxic and it is recommended that the process is carried out in a fume cupboard.

The tar receiver should be cleared on a regular basis, by placing tar in a heat resistant crucible and burning off in a furnace.

WARNING — THE FUMES ARE TOXIC AND DUE CARE SHOULD BE EXERCISED IN THEIR DISPOSAL.

6 Preparation of sample

The coal used for the determination of the Gray-King coke type is the analysis sample ground to pass a sieve of 212 µm aperture. If necessary, expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere.

Before commencing the determination, mix the air-dried sample thoroughly for at least 1 min, preferably by mechanical means. The sample shall be prepared on the same day as the determination is carried out.

7 Procedure

7.1 Coals with a Gray-King coke type within the range A to G_2 (see **10.3**)

Raise the temperature of the furnace until it is steady at 325 °C.

Weigh on a scoop 20,0 g ± 0,1g of the sample and transfer it to the retort tube (5.2), held in such a manner that the coal cannot enter the side arm. Complete the transfer with a soft brush and allow the coal to fall to the far end of the retort tube. Hold the tube horizontally, insert the distance rod (5.3) so that the face of the disk is 150 mm from the closed end of the retort tube and spread the coal into a layer of uniform depth by shaking and turning. Withdraw the distance rod and insert a flattened pad of asbestos wool or a notched asbestos disk to retain the coal in position. Without disturbing the position of the coal, close the open end of the retort tube with a heat-resisting stopper. Connect the receiver (5.4) to the side arm and insert the retort tube in position in the furnace (5.1) so that the centre of the coal layer coincides with the centre of the furnace. If the furnace is mounted on rails, clamp the retort tube in a horizontal position and run the furnace into position.

Raise the energy input to the furnace in such a manner that the temperature of 325 °C is regained in 3 min to 7 min and maintain a uniform rate of rise of 5 °C/min thereafter until a temperature of 590 °C is reached. At this point, regulate the energy input to the furnace so that a temperature of 600 °C is reached, and maintain this temperature for 15 min.

Withdraw the retort tube (or retract the furnace) and allow it to cool. Detach the receiver, remove the stopper and slide the coke residue out for examination.

NOTE If the coal has an ash greater than 10 %, the Gray-King result might be affected.

7.2 Coals with a Gray-King coke type greater than G_2 (see **10.3**)

Weigh X g of the electrode carbon (4.1), where X is always an integer, into a weighing bottle and add $(20 - X)$ g of the coal sample. Insert the stopper and mix the contents thoroughly.

Transfer the mixture to the retort tube and proceed exactly as specified in 7.1.

Repeat the determination if necessary, varying the amount of electrode carbon in 20 g of the mixture, until a coke residue of type G is obtained using the minimum mass of electrode carbon.

8 Expression of results

Report the Gray-King coke type of a coal by reference to Figure 1 and Table 3, where the appearance of typical coke residues is described and illustrated. For coals giving a coke type with an index greater than G_2, the subscript defines the minimum number of grams of electrode carbon added to produce a standard G type coke residue.

9 Precision of the method

9.1 General

Table 1

Type of coke	**Maximum acceptable difference between results**	
	Same laboratory (Repeatability)	**Different laboratory** (Reproducibility)
A to G_1 Greater than G_1	One letter One unit in the subscript	One letter One unit in the subscript

9.2 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator using the same apparatus on the same analysis sample, shall not differ by more than the above value.

9.3 Reproducibility

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 after the last stage of sample preparation, shall not differ by more than the above value.

10 Notes on procedure

10.1 It has been shown that anthracite may be used as an alternative to electrode carbon. Any material which has been found by experiment to produce results equivalent to those obtained when using standard electrode carbon may be used.

If anthracite is to be used, it should meet the following parameters:

Retained on 212 µm test sieve	nil
Through 212 µm, retained on 125 µm	5 % to 10 %
Through 125 µm, retained on 63 µm	20 % to 25 %
Through 63 µm	65 % to 75 %

10.2 Determine the true relative density using a density bottle (see ISO 1014). To ensure complete wetting of the electrode carbon, use a 1 % solution of a wetting agent and evacuate the density bottle containing the electrode carbon and wetting agent solution to a pressure of 8 kPa[1)] in a vacuum desiccator. Maintain this vacuum for 10 min before transferring the density bottle to a water-bath thermostatically controlled at 25 °C.

10.3 Although there is no precise relationship between the crucible swelling number and the Gray-King coke type, the following table shows the broad relationship to be expected. This can be found useful for indicating the necessity for blending with electrode carbon and the probable amount required.

Table 2

Crucible swelling number	Gray-King coke type
0 to 1/2	A to B
1 to 4	C to G_2
4 1/2 to 6	F to G_4
6 1/2 to 8	G_3 to G_9
8 1/2 to 9	G_7 or above

The above relationship is known to be applicable to United Kingdom coals and is intended only as a general guide. Each country should determine the correlation applicable to its own coals.

1) 8 kPa = 80 mbar.

11 Test report

The test report shall include the following particulars:

a) an identification of the product tested;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or regarded as optional.

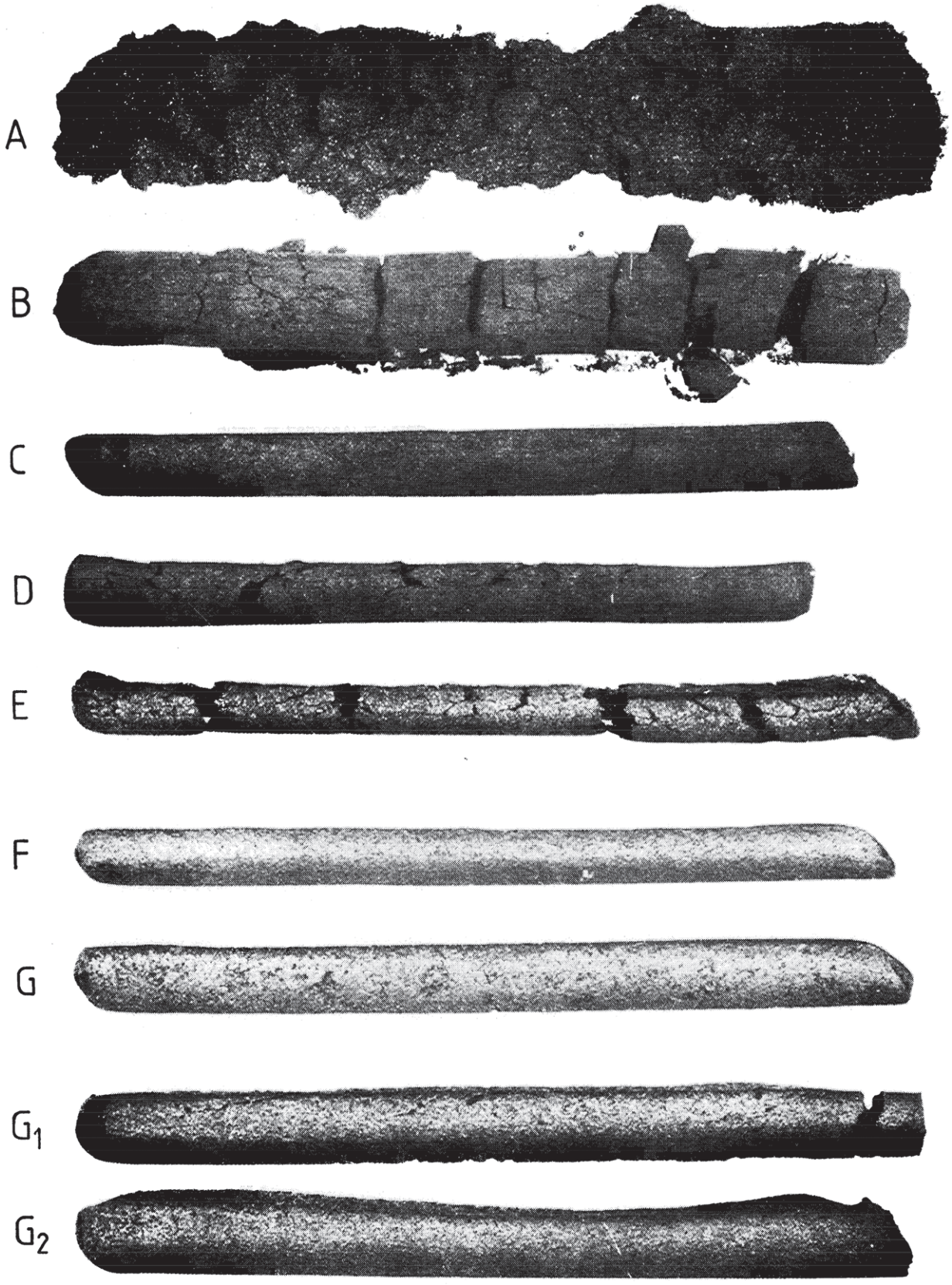
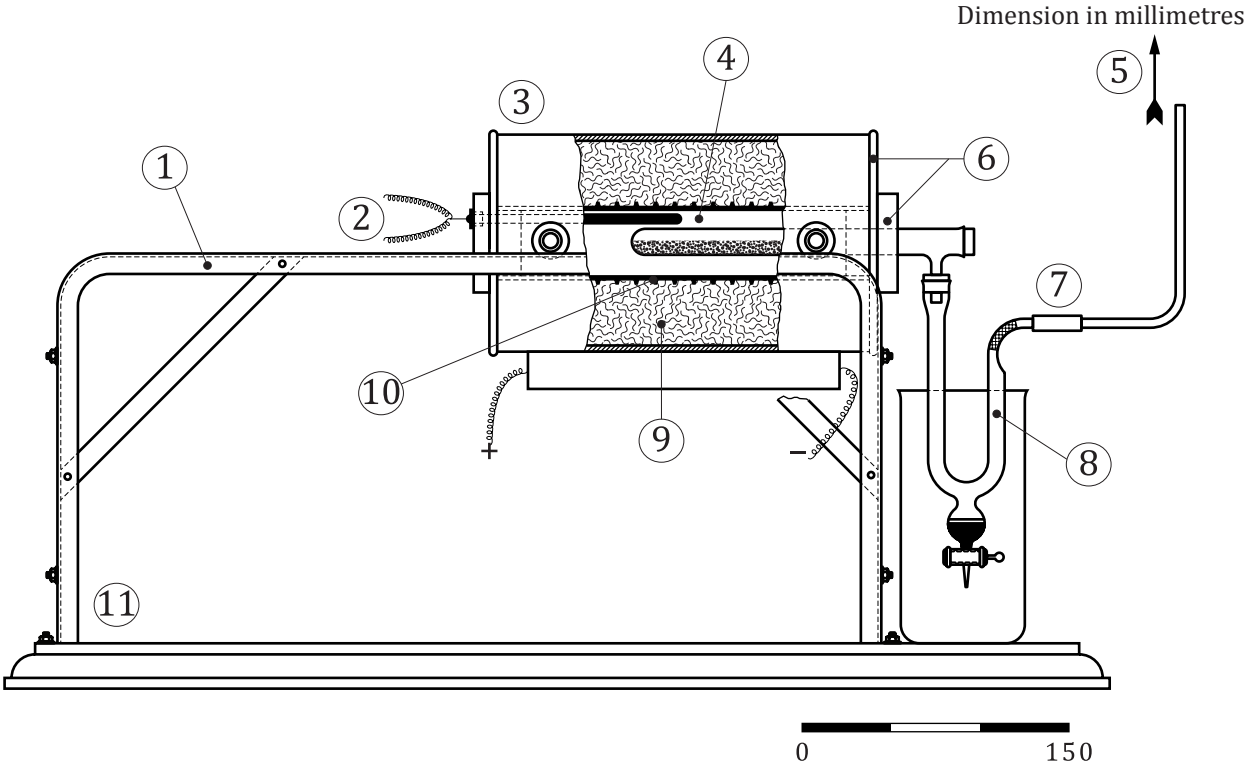


Figure 1 — Gray-King coke type

Table 3 —

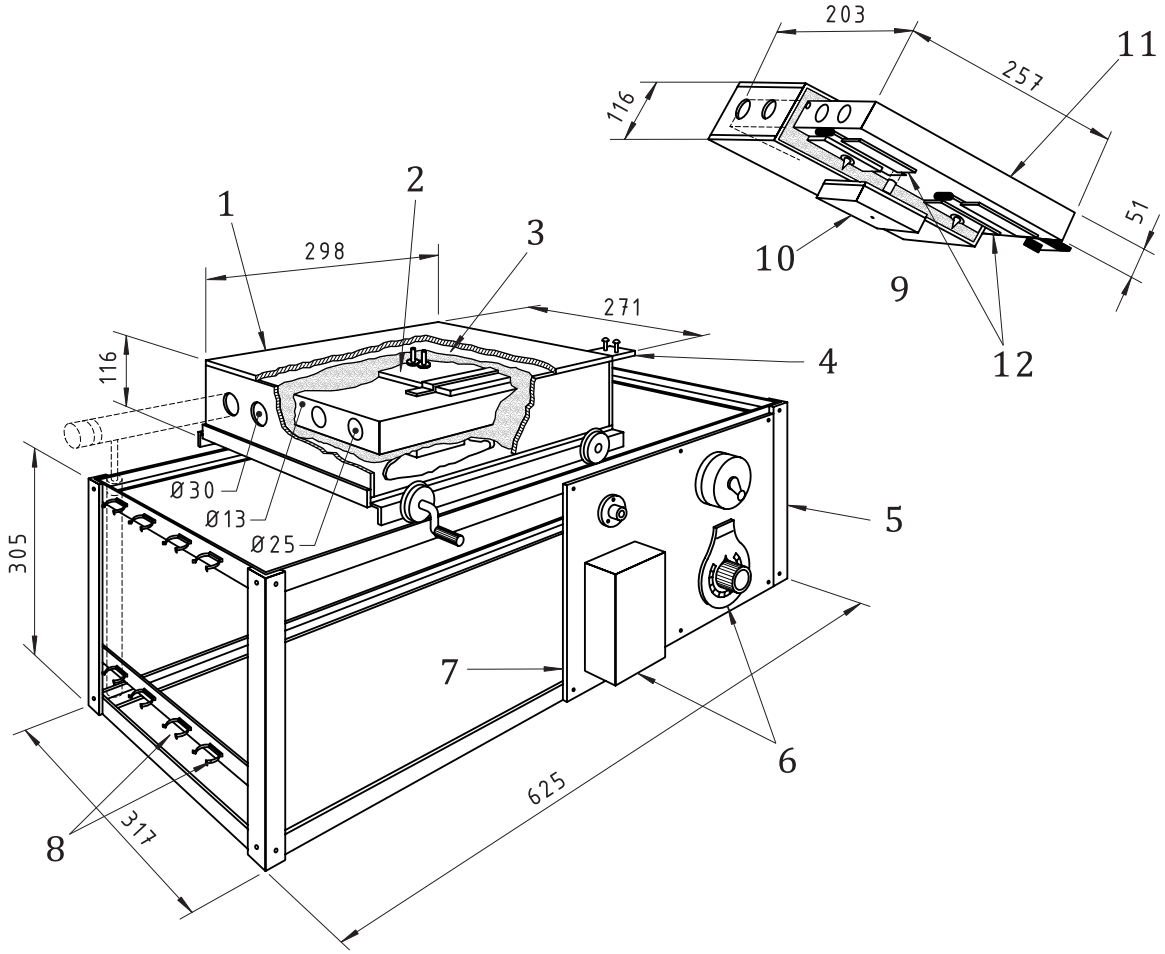


Key

1 rail
2 thermocouple
3 metal shield
4 retort
5 to a burner or to outside air
6 heat insulating material
7 side arm
8 receiver
9 magnesia/asbestos composition
10 silica tube
11 silica tube (length: 300 mm length, Ø int: 52 mm to 53 mm, wall thickness: 6 mm, heating element: Ni-Cr wire 0,6 mm)

Figure 2 — Single tube furnace

Dimension in millimetres



Key

1 casing of 10 mm heat resistant (asbestos free) packing
2 600 W heating elements
3 heat resistant (asbestos free) packing
4 thermocouple support
5 framework of 25 × 25 × 3 angle iron
6 control box and energy regulator
7 control panel insulating sheet, 3 mm thick
8 spring clips for tar traps
9 details of underside of furnace
10 thermal fuse
11 aluminium-bronze block
12 600 W heating elements

Figure 3 — Multiple tube furnace

Dimension in millimetres

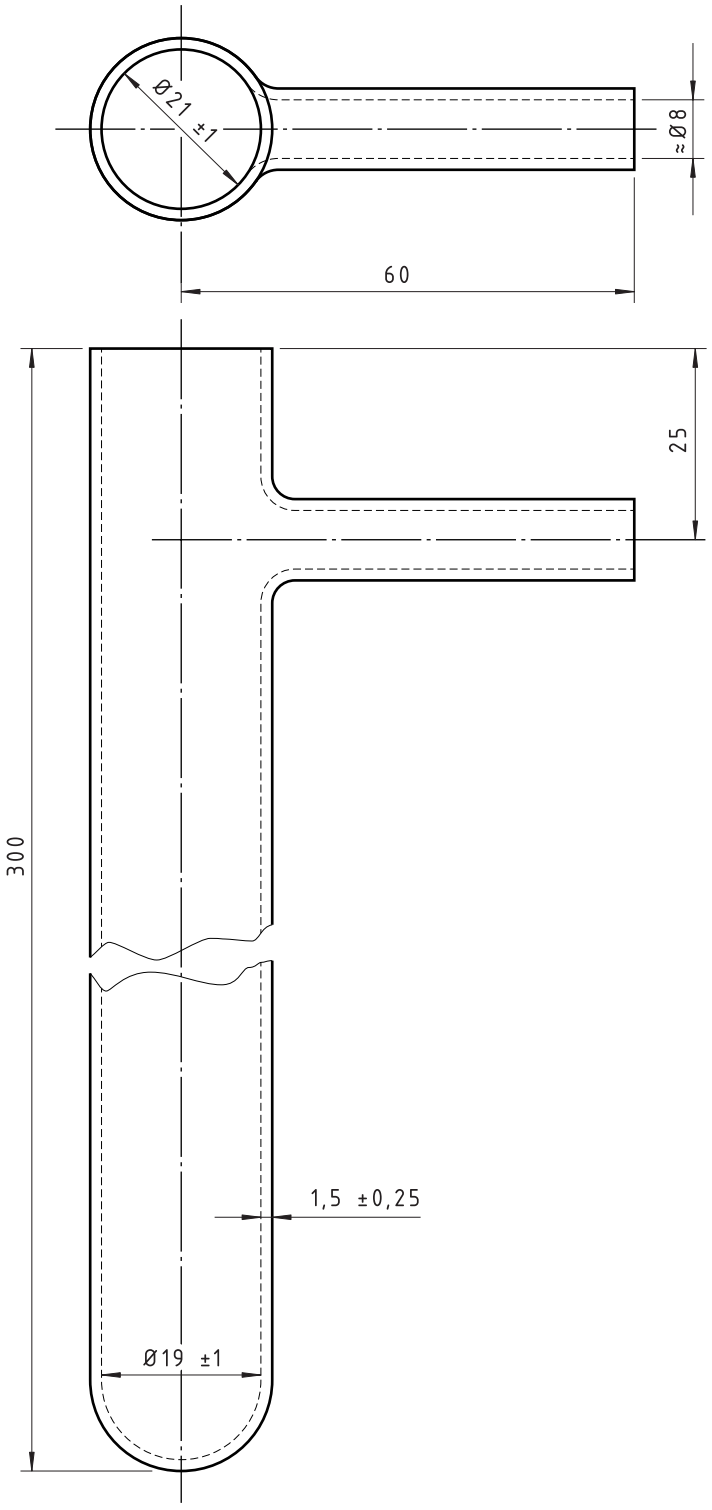


Figure 4 — Retort tube

Annex A
(normative)

Determination of the bulk density of electrode carbon

A.1 Apparatus

A.1.1 Dropping box (see Figure A.1), screwed to a rigid bench or table. The pad at the base of the box shall have a hardness of 71 IRHD to 80 IRHD.

A.1.2 Measuring cylinder, of glass, without a spout and closed by a rubber stopper. The combined mass of cylinder and stopper shall be 250 g ± 5 g. The cylinder shall have its base ground flat and be graduated in 2 ml divisions over the range 25 ml to 250 ml. The maximum permissible error of the graduation at any point shall be 1,5 ml. The length of the cylinder between the zero mark and the 250 ml graduation shall be between 220 mm and 240 mm.

The distance between the ground base of the cylinder and the rubber pad when the cylinder is raised to the permitted height shall be 25 mm ± 2 mm. This can be achieved by suitably packing the underside of the shelf in the dropping box. The assembled apparatus is shown in Figure A.2.

A.1.3 Timing device, to indicate seconds.

This may conveniently be either a metronome or a pendulum. A pendulum to indicate seconds may be improvised from a length of thread about 1 m long and a small pendulum bob. The upper end of the thread shall be clamped on a retort stand between two metal washers. The length of the pendulum shall be adjusted by checking against a stop-clock or watch; the time for 120 to-and-fro swings should be 240 s (complete to-and-fro motion = 2 s).

A.1.4 Balance or scales, of a type providing easy access to the pans, the pans being at least 10 cm in diameter. It is necessary that the pointer shall show a significant deflection for a change in load of 0,25 g

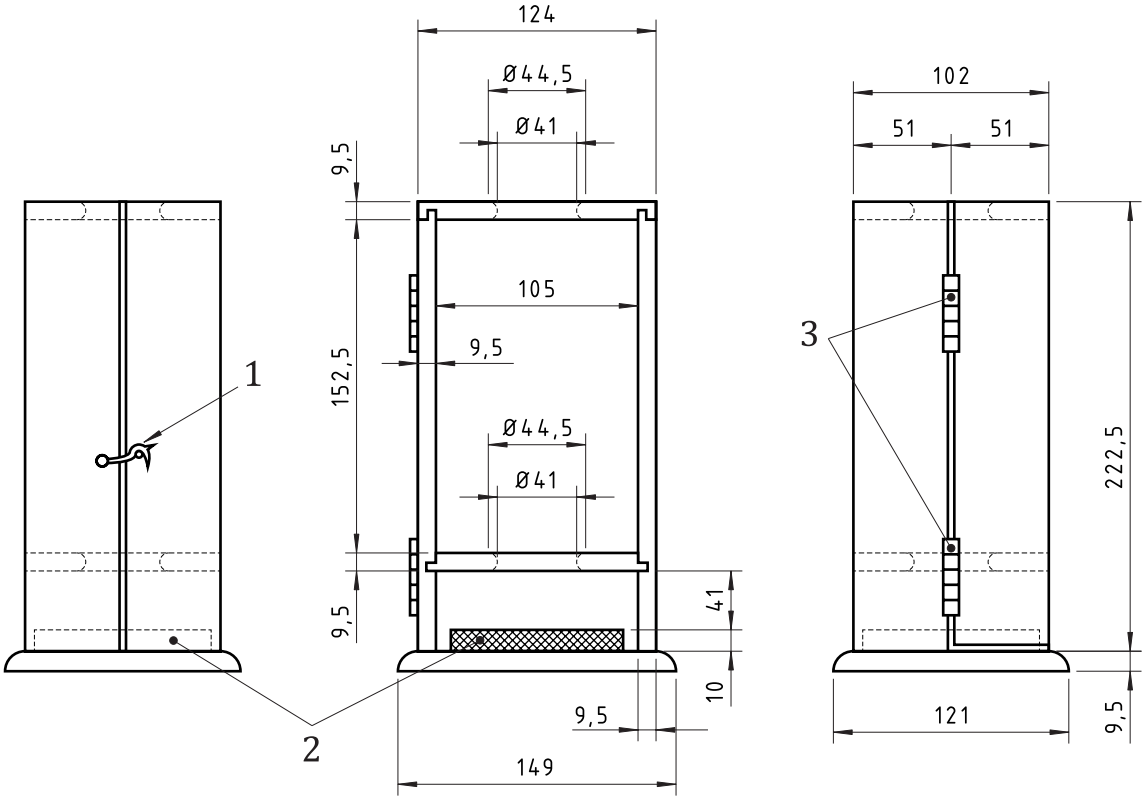
A.1.5 Sampling paper, (250 mm × 250 mm): black glazed.

A.1.6 Finger stalls, of smooth rubber.

A.2 Procedure

Weigh 40 g of the electrode carbon onto a piece of the sampling paper. Wearing finger-stalls, pick up the paper containing the electrode carbon and form the paper into a chute. Allow the paper to lie between the thumb and fingers on the palm of the hand and introduce it for about 13 mm into the cylinder, which is inclined at an angle of 45° to the horizontal. Slide the electrode carbon into the cylinder smoothly and without jerking. Any tendency towards sticking can be overcome by gently tapping the bottom end of the chute with a finger. On no account, knock or jolt the cylinder or squeeze the electrode carbon in the paper during the filling of the cylinder.

Dimension in millimetres



Key

1 hook
2 rubber pad
3 hinges

Figure A.1 — Dropping box

Fit the rubber bung into the cylinder without jolting. Gently place the cylinder in the dropping box and start the timing device. With the thumb and fore-finger of one hand, gently grasp the upper part of the cylinder and during 1 s lift it to the full extent of its travel. Avoid any undue impact with the upper stop so that no jar is given to the electrode carbon. At the commencement of the next second, smartly release the cylinder by quickly and completely withdrawing the thumb and fore-finger.

Continue the process of lifting and dropping until 150 counted drops have been completed, the cylinder falling once every 2 s. Rotate the cylinder through an arc of about 10° during the lifting which precedes each drop, since this will help to impart a level surface to the electrode carbon for the final volume reading.

Immediately, when the 150 drops are completed, remove the cylinder from the dropping box, raise it to eye level and note the volume to the nearest 1 ml. Ignore any further drop in the level after standing.

A.3 Expression of results

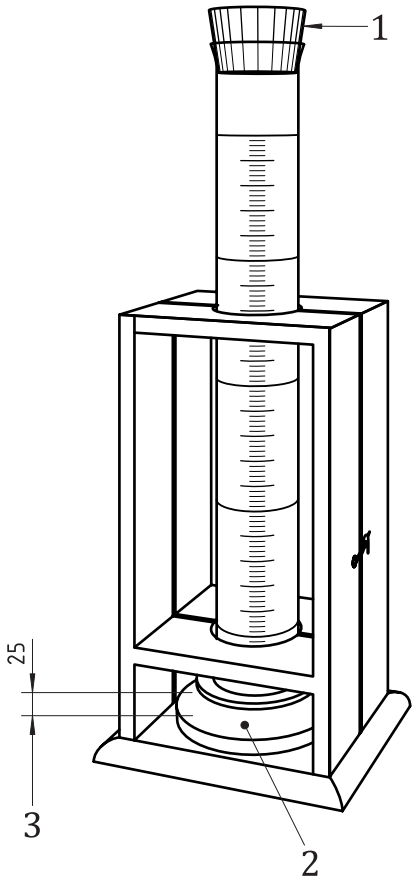
The bulk density, expressed in grams per millilitre, is given by the formula:

$$\frac{40}{V}$$

where

V is the volume occupied by the electrode carbon after 150 drops, expressed in millimetres.

Dimension in millimetres



Key

1 rubber stopper
2 rubber pad
3 distance between base of cylinder and rubber pad

Figure A.2 — Apparatus (assembled)

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Price based on 13 pages

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