UDC 662.6 : 620.1

Ref. No.: ISO/R 502 - 1966 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 502

DETERMINATION OF THE GRAY-KING COKE TYPE OF COAL

1st EDITION
September 1966

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 502, *Determination of the Gray-King Coke Type of Coal*, 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 1951 and led, in 1960, to the adoption of a Draft ISO Recommendation.

In October 1962, this Draft ISO Recommendation (No. 552) 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	Italy	Spain
Austria	Japan	Switzerland
Belgium	Netherlands	Turkey
Brazil	New Zealand	United Kingdom
Chile	Poland	U.S.S.R.
Czechoslovakia	Portugal	Yugoslavia
Denmark	Republic of	
Germany	South Africa	
India	Romania	

One Member Body opposed the approval of the Draft:

France

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in September 1966, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 502 September 1966

DETERMINATION OF THE GRAY-KING COKE TYPE OF COAL

1. SCOPE

This ISO Recommendation describes one method of assessing the caking power of coal under standard conditions.

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 should not be regarded as alternative methods.

2. PRINCIPLE

The coal 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 (Fig. 1, page 6). 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 (see Note 1, page 11). 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.

3. APPARATUS

3.1 Furnace

Horizontal electric furnace, 5 cm internal diameter and 30 cm long, with one end closed and the other carrying a plug of insulating material which is bored centrally with a hole 2.5 cm in diameter. The winding of the furnace should be such that the middle 20 cm is at a uniform temperature within ± 5 degrees Celsius at both 300 and 600 °C. Alternatively the furnace may be constructed from an electrically-heated aluminium-bronze block, with one or several bores of 2.5 cm diameter. The furnace should be insulated and located in a cover of metal or other suitable material, and should 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 should be provided for showing the furnace temperature with an accuracy of ± 5 degrees Celsius. A suitable means of controlling the energy input should also be provided to permit an increase in temperature at a rate of 5 degrees Celsius per minute. 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 Figures 2 and 3, page 8.

FIG. 1. — **Gray-King coke types***

3.2 Retort tube (Fig. 4, page 9)

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 should 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.

3.3 Distance rod

A 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.

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

4. REAGENT

4.1 Standard electrode carbon (see Note 1, page 11)

High temperature electrode carbon:

Moisture	less than 1 per cent
Volatile matter	less than 1.5 per cent
Ash	less than 5 per cent
Bulk density at 25 °C (see Annex A, page 13)	1.00 to 1.05 g/ml
Relative density at 25 °C	2.05 to 2.09 (see Note 2, page 11)

Size analysis:

Retained on 0.2 mm mesh test sieve	nil
Through 0.2 mm mesh test sieve, retained on 0.124 mm mesh test sieve	less than 26 per cent
Through 0.124 mm mesh test sieve, retained on 0.066 mm mesh test sieve	10 to 40 per cent
Through 0.066 mm mesh test sieve	50 to 85 per cent

5. PROCEDURE

Before commencing the determination mix the air-dried sample of coal, ground to pass a sieve of 0.2 mm aperture, thoroughly for at least one minute, preferably by mechanical means. The sample should be ground on the same day as the determination is carried out.

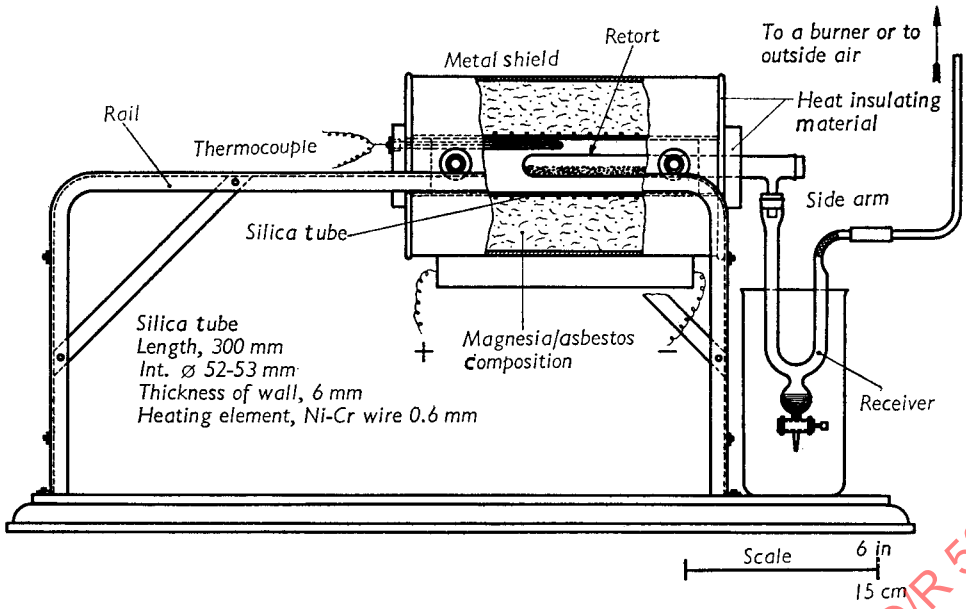

FIG. 2. — **Single tube furnace**

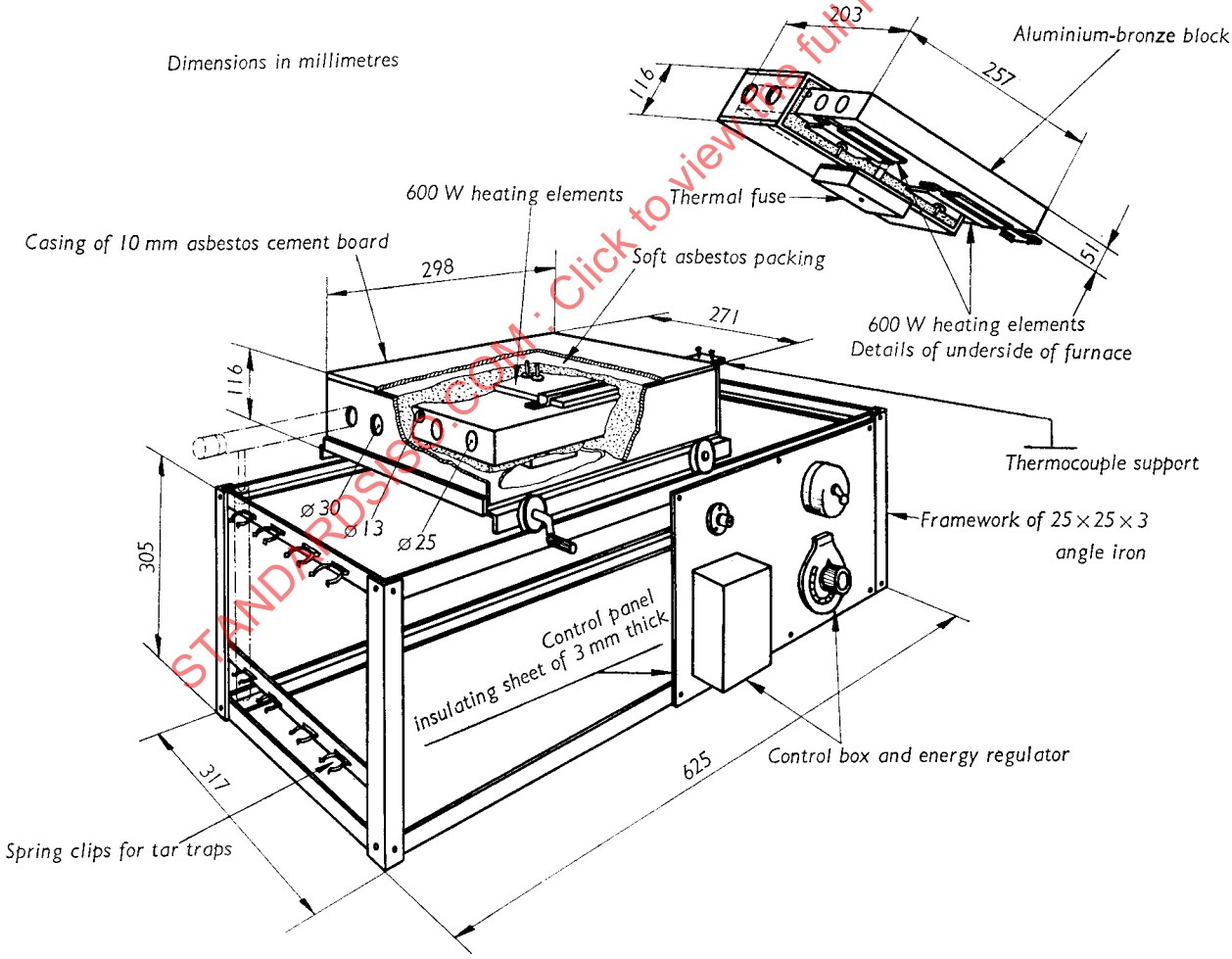

FIG. 3. — **Multiple tube furnace**

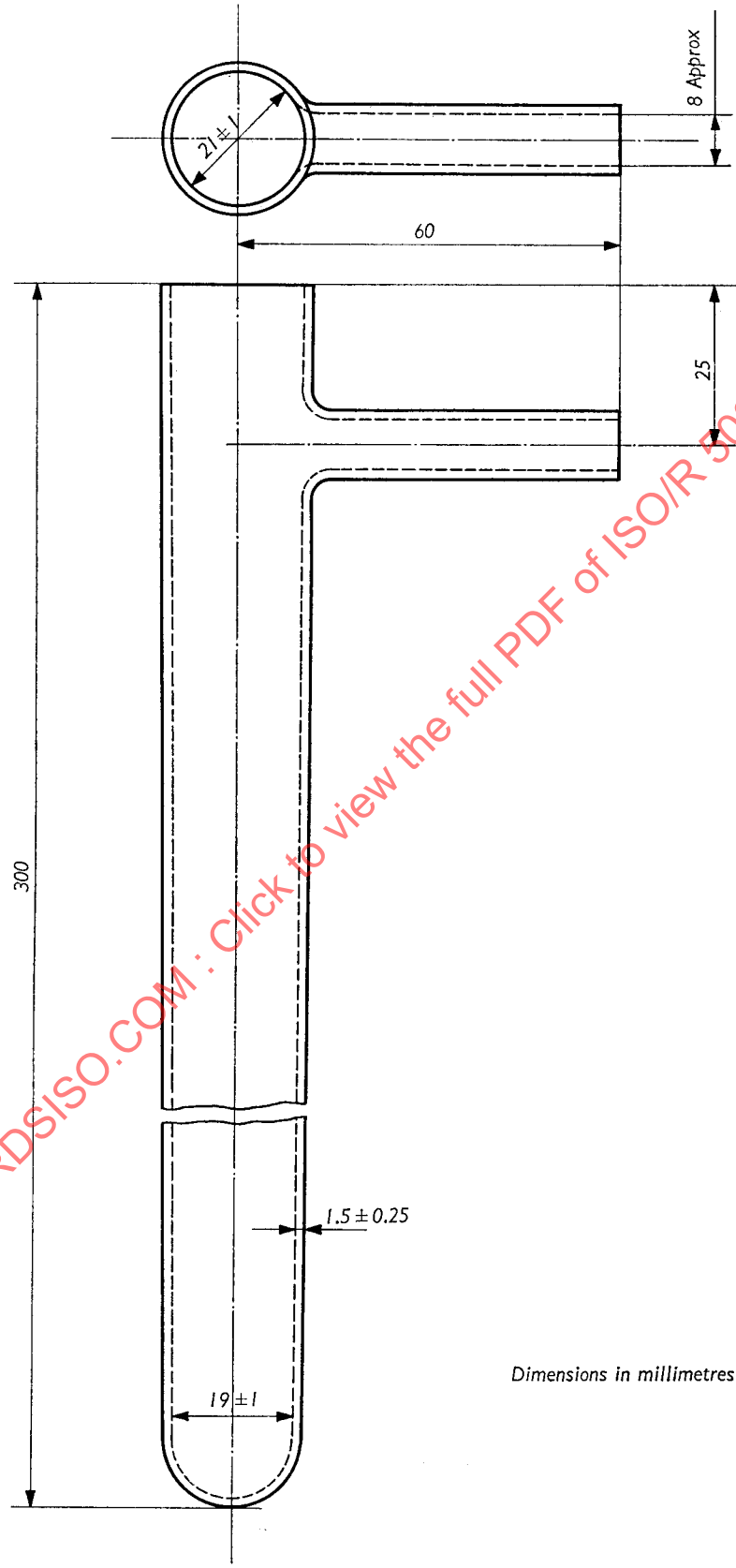

FIG. 4. — **Retort tube**

5.1 Coals with a Gray-King coke type within the range A – G_2 (see Note 3, page 11)

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

Weigh on a scoop 20 to 20.01 g of the coal sample and transfer it to the retort tube, held in such a manner that the coal cannot enter the side tube. 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 so that the face of the disk is 15 cm 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 to the sidearm and insert the retort tube in position in the furnace 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 to 7 minutes and maintain a uniform rate of rise of 5 degrees Celsius per minute 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 minutes.

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.

5.2 Coals with a Gray-King coke type greater than G_2 (see Note 3, page 11)

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 described in clause 5.1.

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.

6. REPORTING OF RESULTS

The Gray-King coke type of a coal is reported by reference to Table 1 and Figure (p. 12), 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 grammes of electrode carbon added to produce a standard G type coke residue.

7. ACCURACY OF DETERMINATION

7.1 In the same laboratory

Duplicate determinations carried out at different times in the same laboratory by the same operator with the same apparatus, on two representative portions taken from the same sample after the last stage of the reduction process, should give the same coke type.

7.2 In different laboratories

Duplicate determinations carried out in each of two different laboratories, on representative portions taken from the same sample after the last stage of the reduction process, should give the same coke type.

NOTES

1. It has been shown that materials other than standard electrode carbon may be used, although it has not yet been possible to define parameters to include all such materials. Any material which has been found by experiment to produce results equivalent to those obtained when using standard electrode carbon may be used. To enable these experiments to be carried out, the United Kingdom is willing to supply a sample of standard electrode carbon or equivalent anthracite.

2. Relative density is determined by the normal method using the density bottle. To ensure complete wetting of the electrode carbon, a 1 per cent solution of a wetting agent is used and the density bottle containing the electrode carbon and wetting agent solution is evacuated to a pressure of 60 mmHg in a vacuum desiccator. This vacuum is maintained for 10 minutes before transferring the density bottle to a water-bath thermostatically controlled at 25 °C.

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 will be found useful for indicating the necessity for blending with electrode carbon and the probable amount required.

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.

TABLE

CLASSIFICATION OF GRAY-KING COKE TYPE

A, B and C Retains initial cross-section			D, E and F shrunken			G retains initial volume	$G_1 - G_x$ swollen		
Examine for strength			Examine for strength			Examine for strength	Examine for degree of swelling		
NON-COHERENT	**BARELY COHERENT**	**COHERENT**	**MODERATELY HARD AND SHRUNKEN**	**HARD AND VERY SHRUNKEN**	**HARD, STRONG AND SHRUNKEN**	**HARD AND STRONG**	**SLIGHTLY SWOLLEN**	**MODERATELY SWOLLEN**	**HIGHLY SWOLLEN**
Usually in powder form but may contain some pieces which, however, cannot be handled without breaking	In several pieces and some loose powder. Pieces can be picked up but break into powder on handling	Usually in one piece but easily broken; may be in two or three pieces with practically no loose powder; very friable and dull	May be fissured but can be scratched with fingernail and stains the fingers on rubbing the curved surface vigorously; usually dull and black and appearing fritted rather than fused	Usually very fissured; moderate metallic ring; does not stain the fingers on rubbing; grey or black with slight lustre	May be fissured; moderate metallic ring; does not stain the fingers on rubbing. Cross-section well fused and greyish	Well fused with a good metallic ring when tapped on a hard wooden surface			G_3 and higher. Guided by swelling number, blend with minimum number of parts of electrode carbon to give a standard G-type coke
A	B	C	D	E	F	G	G_1	G_2	G_x