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# INTERNATIONAL STANDARD



# 501

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Coal — Determination of the crucible swelling number

*Charbon — Détermination de l'indice de gonflement au creuset*

First edition — 1974-12-01

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## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 501 and found it technically suitable for transformation. International Standard ISO 501 therefore replaces ISO Recommendation R 501-1966 to which it is technically identical.

ISO Recommendation R 501 was approved by the Member Bodies of the following countries :

|                |                       |                |
|----------------|-----------------------|----------------|
| Australia      | Italy                 | Spain          |
| Austria        | Japan                 | Sweden         |
| Belgium        | Netherlands           | Switzerland    |
| Brazil         | New Zealand           | Turkey         |
| Chile          | Philippines           | United Kingdom |
| Czechoslovakia | Poland                | U.S.A.         |
| Denmark        | Portugal              | U.S.S.R.       |
| Germany        | Romania               | Yugoslavia     |
| India          | South Africa, Rep. of |                |

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France\*

No Member Body disapproved the transformation of ISO/R 501 into an International Standard.

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\* Subsequently, this Member Body approved the Recommendation.

# Coal – Determination of the crucible swelling number

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the swelling properties of a coal when heated in a covered crucible.

## 2 PRINCIPLE

The coal is heated in a covered crucible under standard conditions to a final temperature of  $820 \pm 5$  °C. The coke button obtained is classified by comparison with the outlines of a set of standard profiles. The number of the profile most closely corresponding to the coke button obtained is the crucible swelling number.

Gas or electrical heating may be used. When using an electrically heated apparatus the results obtained should be the same (within 1/2 unit) as those obtained by the gas heating method.

## 3 PREPARATION OF SAMPLE

The coal used for the determination of swelling number is the analysis sample ground to pass a 0,2 mm sieve. Avoid both very fine grinding and undue exposure of the ground coal to the atmosphere since they are liable to lead to erroneous results (see note). Prepare the coal on the same day that the test is carried out. With coals especially liable to oxidation carry out the test as soon as possible and not more than 2 h after grinding, unless the sample can be stored in an inert atmosphere.

Before commencing the determination, mix the air-dried sample of coal thoroughly for at least 1 min, preferably by mechanical means.

NOTE – The sample may, with advantage, be air-dried in an inert atmosphere and at a temperature not exceeding 45 °C.

## 4 GAS HEATING METHOD

### 4.1 Apparatus

#### 4.1.1 Crucible and lid (figure 1)

Translucent silica crucible, squat form, and silica lid with ring handle. (If the lower surface of the crucible lid is not flat, difficulty may be experienced in assessing the swelling number of the coal. A small mica plate shall then be

inserted between the lid and crucible before carrying out the test.) Crucibles and lids of other ceramic materials may be used provided that the results agree with those obtained with silica crucibles. The crucible shall conform to the following specifications :

External height  $26 \pm 0,5$  mm

External diameter at top  $41 \pm 0,75$  mm

Minimum internal diameter at base 11 mm

Mass 11 to 12,75 g

Capacity 16 to 17,5 ml

Dimensions in millimetres

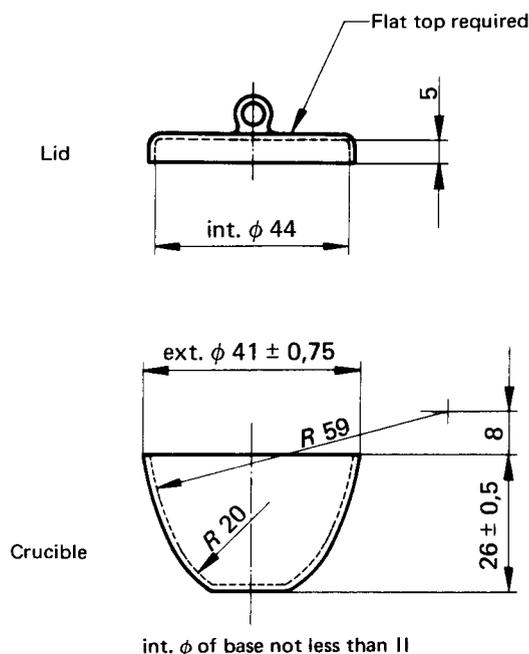


FIGURE 1 – Crucible with lid for swelling test

4.1.2 Pierced silica lid (figure 2)

Similar to that above but with a 6 mm hole to accommodate the thermocouple.

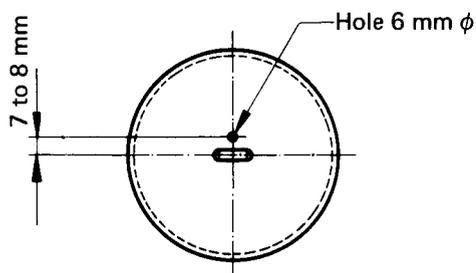


FIGURE 2 – Pierced crucible lid

4.1.3 Triangle

Consisting of silica tubing of 6 to 6,5 mm external diameter mounted on nickel/chromium wire, the length of side being approximately 65 mm.

Unprotected nickel/chromium wire may be used provided it is sufficiently rigid.

4.1.4 Burner

Capable of giving the required standard heating conditions; the following types have been found satisfactory :

- a) A Teclu type burner; with coal gas of calorific value of about 20 MJ/m<sup>3</sup>, the internal diameter of the burner tube should be approximately 12,5 mm.
- b) A Meker type burner; with natural gas of calorific value of about 40 MJ/m<sup>3</sup>, the external diameter of the burner grid should be approximately 30 mm.

4.1.5 Gauge

A gas pressure gauge.

4.1.6 Draught shield

Made from asbestos cement piping approximately 150 mm in length, 100 mm internal diameter and 110 mm external diameter. At one end the piping shall have three slots 25 mm deep in which the wire portions of the silica triangle rest (figure 3).

4.1.7 Thermocouple

Of fine wire of diameter not greater than 0,23 mm if made of platinum or 0,45 mm if made of base metal. The ends of the couple shall be in the form of a flattened loop.

4.1.8 Weight

A 500 g weight.

4.2 Preparation of apparatus

Assemble the apparatus as shown in figure 3. Place an empty crucible on the silica triangle and support it on the draught shield. Adjust the gas and air supplies to the burner so that the temperature of the inner surface of the base of the crucible reaches 800 ± 10 °C in 1,5 min and 820 ± 5 °C in 2,5 min from first igniting the gas.

If a Teclu type burner is used it will generally be found that a flame approximately 300 mm long, with the crucible positioned just above the tip of the blue cone, will give the standard temperature conditions.

If a Meker type burner is used, place the crucible approximately 10 mm above the burner grid and adjust the flame so that the standard temperature conditions are attained.

Measure the temperature by means of the thermocouple inserted through the pierced lid and having its unprotected junction and a portion of each wire in contact with the centre of the base of the empty crucible.

Check the apparatus at frequent intervals to ensure that the standard conditions apply.

Dimensions in millimetres

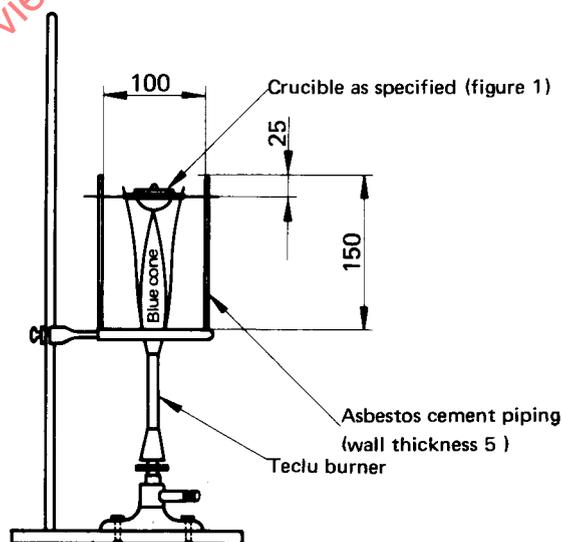


FIGURE 3 – Apparatus for the gas heating method

4.3 Procedure

Weigh 1,00 to 1,01 g of the freshly ground coal into a dry crucible and lightly tap the crucible about 12 times on the bench to level the surface of the coal. Cover the crucible with the unpierced lid and place it upright on the silica triangle supported in the draught shield. Light the gas and

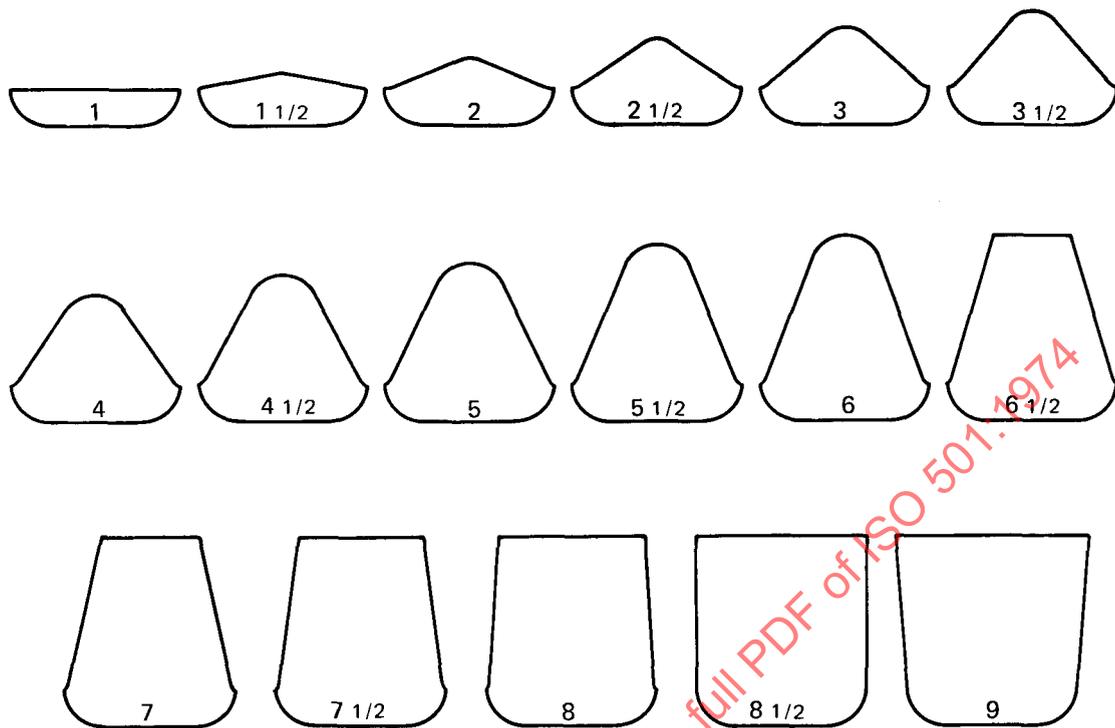


FIGURE 4 – Determination of crucible swelling number, standard profiles and corresponding swelling numbers

heat at the predetermined rate for such time as is required for the flame of the burning volatile matter to die out and in any case for at least 2,5 min. Turn off the gas and allow the crucible to cool.

Remove the residue carefully from the crucible.

If the residue is non-coherent, i.e. mostly loose powder, no further examination is needed and this result shall be designated swelling number 0.

If the residue is a coherent coke button but is not swollen, place it on a flat surface and rest the 500 g weight on it. If the button supports the 500 g weight without breaking into more than two or three hard pieces, designate it swelling number 1; if it crumbles, or disintegrates, then designate it swelling number 1/2.

If the coke button is swollen compare it with the standard numbered profiles shown in figure 4. Rotate the button about its axis so that its largest profile is presented for comparison; designate the result by the swelling number inscribed in the outline in figure 4 which it most nearly matches. A method of viewing to avoid the effect of parallax is shown in figure 5.

Carry out five tests; after each test burn off the carbon residue and wipe the crucible with a clean cloth.

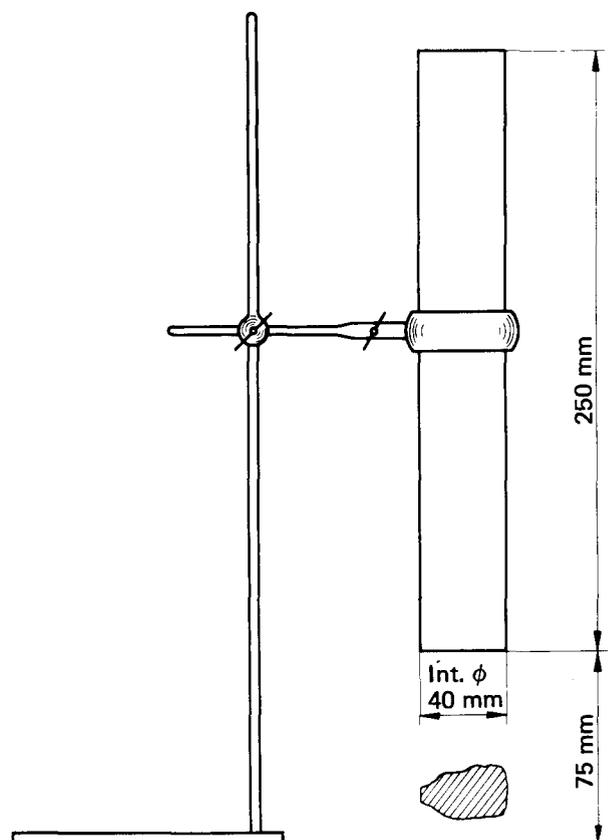


FIGURE 5 – Apparatus for viewing the button

## 5 ELECTRICAL HEATING METHOD

### 5.1 Apparatus

#### 5.1.1 Furnace

A suitable type of electrically heated furnace is shown in figure 6, although other types of furnaces may be used provided that the results obtained are the same (within 1/2 unit) as those obtained with the gas heating method.

The furnace illustrated consists of a grooved refractory plate (1), approximately 88 mm in diameter and 13 mm thick, carrying the heating element, which can be a spiralled metallic coil. The plate is supported by a second grooved plate (2). An inverted silica dish (3) of 1 mm wall thickness 10 mm high and approximately 85 mm external diameter, is placed over the windings and acts as a support for the crucible.

The plates are surrounded by a refractory cylinder (4) of approximately 140 mm diameter, bored to a depth of about 60 mm with a 90 mm diameter hole. The cylinder is fitted with a refractory lid (5), 20 mm thick; a hole 50 mm in diameter is bored in the centre of the refractory lid to allow for insertion of the crucible. The refractory cylinder rests on an asbestos sheet (6), 3 to 5 mm thick, and is surrounded by a 10 mm thick insulation of asbestos fibre and light magnesium oxide. The whole furnace is placed in an aluminium case (7).

A hole bored through the base of the furnace enables a thermocouple to be placed in contact with the underside of the silica dish.

The furnace shall be equipped with a suitable energy regulator and ammeter.

#### 5.1.2 Crucible and lid

As specified in 4.1.1.

#### 5.1.3 Pierced silica lid

As specified in 4.1.2.

#### 5.1.4 Thermocouples

- A fine wire thermocouple as specified in 4.1.7.
- A suitable base metal thermocouple to measure the temperature of the underside of the silica dish.

### 5.2 Preparation of apparatus

Switch on the furnace and adjust the energy input so that a steady temperature of about 850 °C is maintained at the base of a crucible resting on the silica plate. Remove the crucible and insert a cold crucible, covered with a pierced lid through which passes the fine wire thermocouple held so

that its unprotected junction and a portion of each wire rests on the base of the crucible. Ascertain that the standard heating conditions of 800 ± 10 °C in 1,5 min and 820 ± 5 °C in 2,5 min are attained from the time of inserting the crucible. If these conditions are not attained adjust the furnace temperature until the specified conditions are attained. Record the furnace temperature as indicated by the thermocouple at the underside of the silica dish, this temperature serving as a datum.

### 5.3 Procedure

Weigh 1,00 to 1,01 g of the freshly ground coal into a dry crucible and lightly tap the crucible about 12 times on the bench to level the surface of the coal. Cover the crucible with the unpierced lid and place it centrally in the furnace on the silica dish. Heat till the volatile matter ceases to be evolved and in any case for at least 2,5 min.

Remove the crucible from the furnace and allow it to cool. Examine the residue as described in 4.3.

Carry out five tests in succession, replacing one crucible with the next to avoid heat losses through the top of the furnace. Alternatively, a lid of refractory material (8) as shown in figure 6 may be used to maintain the furnace temperature between tests.

After each test burn off the carbon residue and wipe the crucible with a clean cloth.

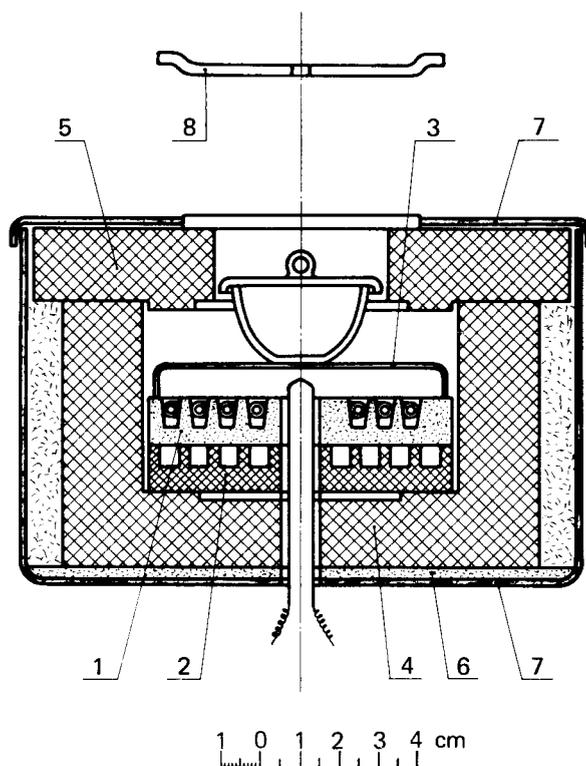


FIGURE 6 – Electrically heated furnace