

**ISO**

*transformation*

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

# ISO RECOMMENDATION R 1018

DETERMINATION OF THE MOISTURE-HOLDING CAPACITY  
OF HARD COALS

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## BRIEF HISTORY

The ISO Recommendation R 1018, *Determination of the moisture-holding capacity of hard coals*, 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 led to the adoption of a Draft ISO Recommendation.

In August 1967, this Draft ISO Recommendation (No. 1285) 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	Sweden
Austria	Korea, Rep. of	Switzerland
Belgium	Netherlands	Turkey
Czechoslovakia	New Zealand	U.A.R.
Denmark	Poland	United Kingdom
France	Portugal	U.S.S.R.
Germany	Romania	Yugoslavia
India	South Africa, Rep. of	
Iran	Spain	

Three Member Bodies opposed the approval of the Draft :

Canada  
Japan  
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1969, to accept it as an ISO RECOMMENDATION.

## DETERMINATION OF THE MOISTURE-HOLDING CAPACITY OF HARD COALS

### 1. SCOPE

This ISO Recommendation describes a method of determining the moisture-holding capacity of hard coals.

### 2. FIELD OF APPLICATION

The moisture-holding capacity indicates the rank of hard coals and is used in coal classification for correcting the calorific value of the sample to the moist mineral matter free basis. The full moisture-holding capacity is that of the coal in equilibrium with an atmosphere saturated with water vapour. Since there are insuperable experimental difficulties in working with such an atmosphere, the determination is carried out at 96 % relative humidity.

### 3. PRINCIPLE

The coal is brought to equilibrium with an atmosphere of 96 % relative humidity at 30 °C and then dried to constant mass at 105 to 110 °C. The conditioning of the coal may be carried out either at atmospheric pressure or under reduced pressure. The moisture-holding capacity is reported as a percentage, by mass, of the conditioned moist coal.

### 4. REAGENT

*Potassium sulphate pulp.* Add sufficient potassium sulphate to water to form a pulp.

### 5. APPARATUS

#### 5.1 Atmospheric pressure method

- 5.1.1 *Conditioning vessel* (see Fig. 1). A double-walled vessel, manufactured of copper sheet, with a double-walled lid made in two pieces. The lid and vessel are covered by a foam-rubber insulation jacket. Each half of the lid is secured by three equally spaced clamps, or by the addition of a 2 kg weight.
- 5.1.2 *Electric motor*, capable of driving the two-blade propeller at approximately 1500 rev/min.
- 5.1.3 *Pump*, for circulating water through the conditioning vessel to maintain it at a temperature of  $30 \pm 0.1$  °C.
- 5.1.4 *Dishes*. Glass or corrosion-resistant metal dishes, approximately 50 mm diameter by 10 mm deep, with well-fitting lids.

## 5.2 Reduced pressure method

5.2.1 *Conditioning vessel* (see Fig. 2). A vacuum desiccator weighted to overcome its buoyancy when immersed in water. The desiccator is fitted with a mercury vacuum manometer, and a glass or corrosion-resistant metal stand is provided to carry dishes above the level of the pulp, so that the dishes are protected from spray due to frothing. The volume of free space in the desiccator is kept to a minimum by the choice of a suitable design, by increasing the volume of the pulp material or by adding inert material such as glass beads or washed sand to the pulp.

5.2.2 *Water bath* (see Fig. 2), thermostatically controlled at a temperature of  $30 \pm 0.1$  °C.

5.2.3 *Dishes*. Glass or corrosion-resistant metal dishes, approximately 22 mm diameter by 15 mm deep. The lids for these dishes should form a sliding fit.

## 5.3 For both methods

5.3.1 *Vacuum pump*.

5.3.2 *Filter crucible or funnel*.

5.3.3 *Filter flask*.

5.3.4 *An apparatus for determining moisture* by a method which precludes oxidation of the coal.\*

5.3.5 *Filter paper*, diameter about 200 mm.

## 6. SAMPLE

It is essential that the coal should be in a fresh unchanged state. If the sample cannot be examined immediately it should be protected from oxidation by storing under water.

Crush the sample to pass a sieve of 0.2 mm aperture, using the procedures described in ISO Recommendation R ...,\*\* *Sampling of hard coal*. The production of an excessive amount of fines should be avoided.

## 7. PROCEDURE

### 7.1 Preparation of apparatus

7.1.1 *Both methods*. Fill the conditioning vessel with the potassium sulphate pulp to the level indicated in Figure 1 or Figure 2.

7.1.2 *Atmospheric pressure method*. Connect the conditioning vessel to the pump and circulate the water at a rate sufficient to maintain a temperature of  $30 \pm 0.1$  °C in the vessel.

7.1.3 *Reduced pressure method*. Evacuate the desiccator several times until frothing ceases. To minimize subsequent frothing, the desiccator should be kept evacuated when not in use.

\* The apparatus and procedure of Method B described in ISO Recommendation R 589, *Determination of total moisture in hard coal*, are suitable for this purpose and the description is reproduced in Annex A.

\*\* At present Draft ISO Recommendation No. 1988.

## 7.2 Preliminary treatment of the sample

Place about 20 g of the sample, crushed to pass a sieve of 0.2 mm aperture, into a conical flask, add about 100 ml of distilled water, shake several times and filter on a filter crucible or funnel. Wash the filtered coal with two or three separate portions of 25 ml of distilled water, taking care that the surface of the coal is not dried by excessive suction. Remove the coal from the filter and spread out between two layers of the filter paper, each layer consisting of eight sheets of the filter paper. The thickness of the coal layer should not be more than 4 mm. Place a weight of about 10 kg on the top layer of filter paper; a considerable part of the adherent surface water is removed by this pressure. After about 10 minutes, remove the weight and thoroughly mix the coal with a spatula.

## 7.3 Conditioning

**7.3.1 Atmospheric pressure method.** Weigh the empty dish and lid and spread uniformly into it approximately 2 g of the treated coal (see clause 7.2). Place the dish in the conditioning vessel, controlled to a temperature of  $30 \pm 0.1$  °C, so that it is below an air circulation exit. Replace the two halves of the lid and secure by means of clamps or weights. Start the propeller motor and pass air for a period of 3 to 6 hours (see Note 1, clause 7.3.2). Switch off the motor, remove the dish and securely replace its lid. Transfer the dish on a metal tray to a balance and weigh quickly to within 0.2 mg.

**7.3.2 Reduced pressure method.** Weigh the empty dish and lid and spread uniformly into it approximately 1 g of the treated coal (see clause 7.2). Cover the loaded dish loosely with its lid and place in the conditioning vessel. Replace the lid of the conditioning vessel and evacuate it to a pressure of 20 to 25 mbar. Place the vessel into the water bath, maintained at a temperature of  $30 \pm 0.1$  °C, and re-evacuate. The pressure should rise quickly to about 40 mbar, which is the vapour pressure of the saturated solution of potassium sulphate at 30 °C. If the pressure rises above 45 mbar, re-evacuate the vessel without otherwise disturbing it.

After a period of  $24 \pm 2$  hours, with the vessel still in the water bath, restore the pressure to atmospheric by slowly admitting dry air at 30 °C through the train, consisting of a capillary tube, a tower charged with dry magnesium perchlorate (see Note 2) and a coiled copper tube immersed in the water bath and connected to the inlet of the vessel (see Fig. 2). In order to avoid changes in the moisture content it is essential to ensure that there is no disturbance of the local atmosphere immediately adjacent to the conditioned coal. This may be achieved by adjusting the length and diameter of the capillary G (see Fig. 2), so that the time taken to restore atmospheric pressure is approximately 15 minutes. Remove the vessel from the bath, remove the lid, transfer the covered dish immediately to a balance and weigh quickly to within 0.2 mg.

### NOTES

1. The period required for equilibrium to be reached will depend on the rank of the coal. In the case of an "unknown" coal, several samples of the coal should be placed in the vessel and the moisture-holding capacity determined after various conditioning times.
2. Regeneration of 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 stream of water.

## 7.4 Determination of moisture content

Determine the moisture content of the conditioned sample by the method described in Annex A, using the dish in which the coal was conditioned.

### 8. EXPRESSION OF RESULTS

The moisture-holding capacity (*MHC*) of the sample as analysed, expressed as a percentage, by mass, is calculated from the following formula :

$$MHC = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

- $m_1$  is the mass of empty dish and lid, in grammes;
- $m_2$  is the mass of coal, dish and lid after conditioning, in grammes;
- $m_3$  is the mass of dried coal, dish and lid, in grammes.

The result, preferably the mean of duplicate determinations (see section 9), should be reported as follows :

Moisture-holding capacity	Report result to the nearest
Less than 5 %	0.1 %
5 % and over	0.5 %

### 9. PRECISION OF THE METHOD

Moisture-holding capacity	Maximum acceptable differences between results obtained	
	in the same laboratory (Repeatability)	in different laboratories (Reproducibility)
	0.5 % absolute	1.2 % absolute

#### 9.1 Repeatability

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.

#### 9.2 Reproducibility

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

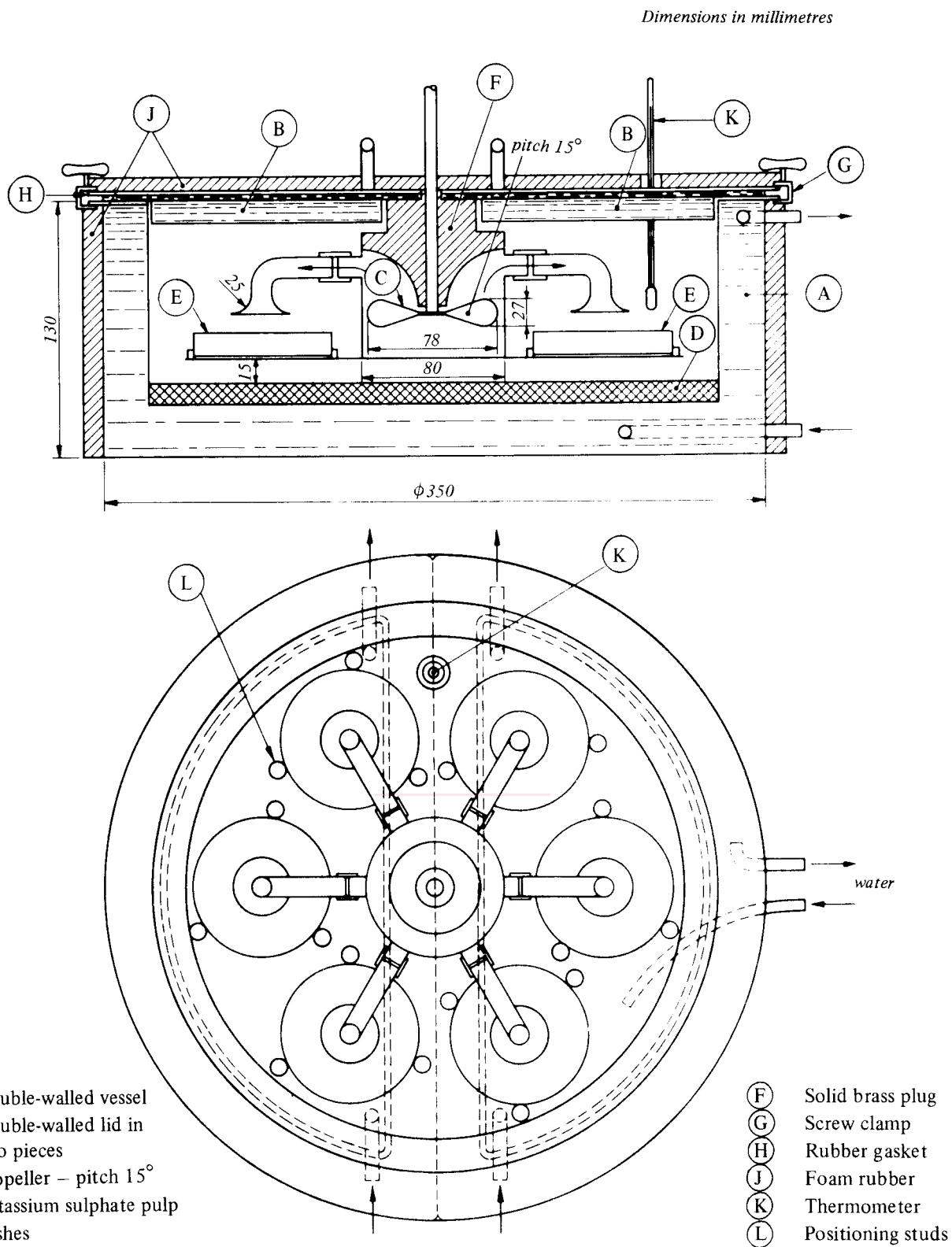


FIG. 1 – Apparatus for determination of moisture-holding capacity of coal (Atmospheric pressure method)

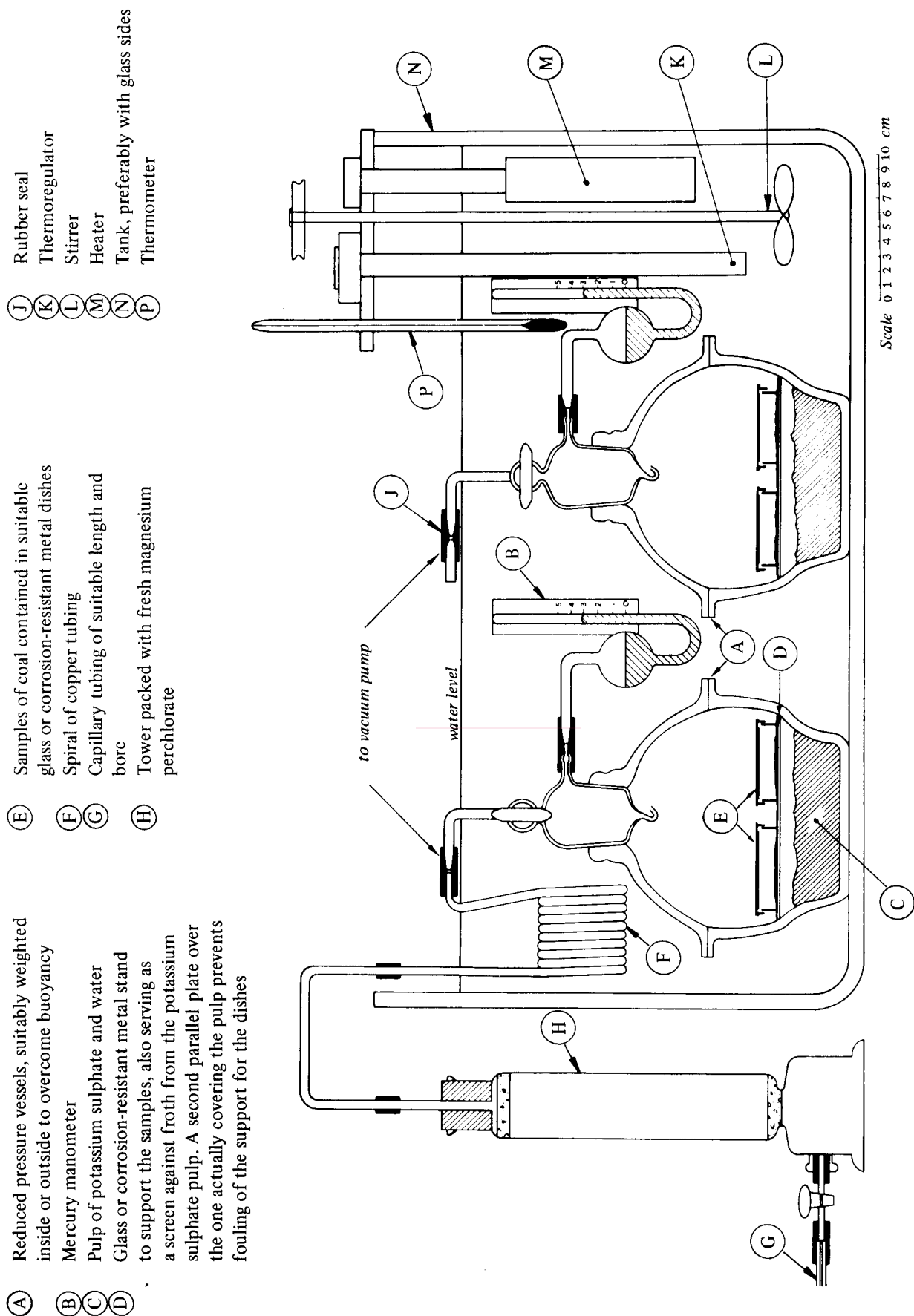


FIG. 2 - Apparatus for determination of moisture-holding capacity of coal (reduced pressure method)

## ANNEX A

### DETERMINATION OF MOISTURE IN THE CONDITIONED COAL

#### A.1 REAGENTS

A.1.1 *Nitrogen*. Dry, and containing less than thirty parts per million of oxygen (see Annex B).

A.1.2 *Desiccant*. Either fresh or freshly regenerated silica gel or other suitable desiccant, for use in the desiccator.

#### A.2 APPARATUS

A.2.1 *Nitrogen oven*. An oven capable of maintaining a temperature within the range 105 to 110 °C, with provision for passing a current of dry, oxygen-free nitrogen through it at a rate sufficient to change the atmosphere fifteen times per hour.

A.2.2 *Weighing vessels*. See clause 5.1.4 or 5.2.3.

#### A.3 PROCEDURE

Remove the lid from the dish and place it in the desiccator. Heat the uncovered dish in the oven at a temperature of 105 to 110 °C until constant in mass. Replace the lid, cool the dish rapidly on a cold plate, transfer to a desiccator for not more than 10 minutes and weigh to within 0.2 mg.

NOTE. — Heating for 1 hour is normally sufficient. Constancy in mass is defined as a change not exceeding 1 mg in a further period of heating of 30 minutes.