UDC 662.66 : 543.81

Ref. No. : ISO/R 1018-1969 (E)

ISO

transformée

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 1018

DETERMINATION OF THE MOISTURE-HOLDING CAPACITY OF HARD COALS

1st EDITION
March 1969

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 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
Austria
Belgium
Czechoslovakia
Denmark
France
Germany
India
Iran
Italy
Korea, Rep. of
Netherlands
New Zealand
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Sweden
Switzerland
Turkey
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

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.

ISO Recommendation R 1018 March 1969

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 ± 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 ± 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 ± 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 ± 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 ± 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 ± 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

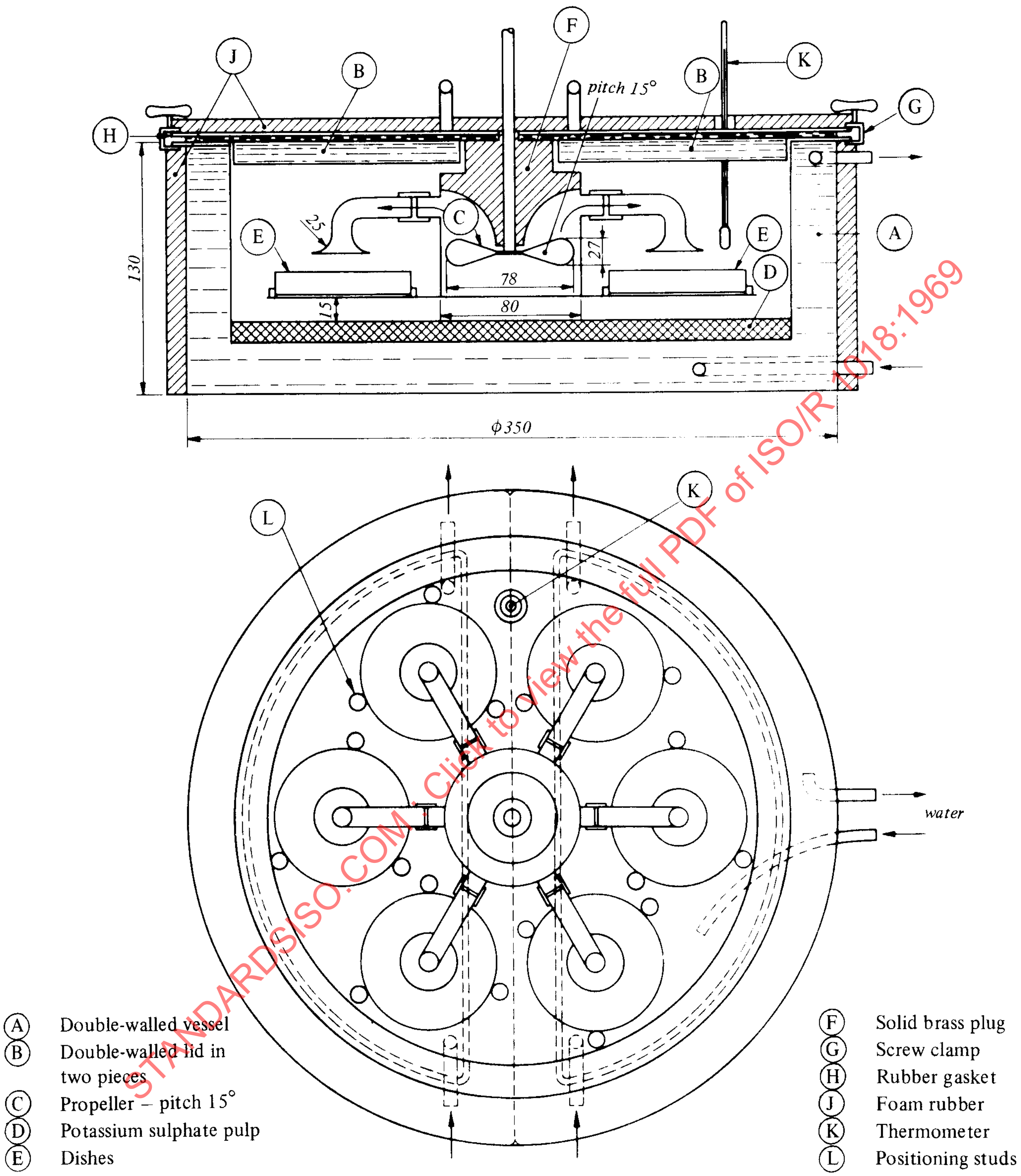
9.1 Repeatability

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

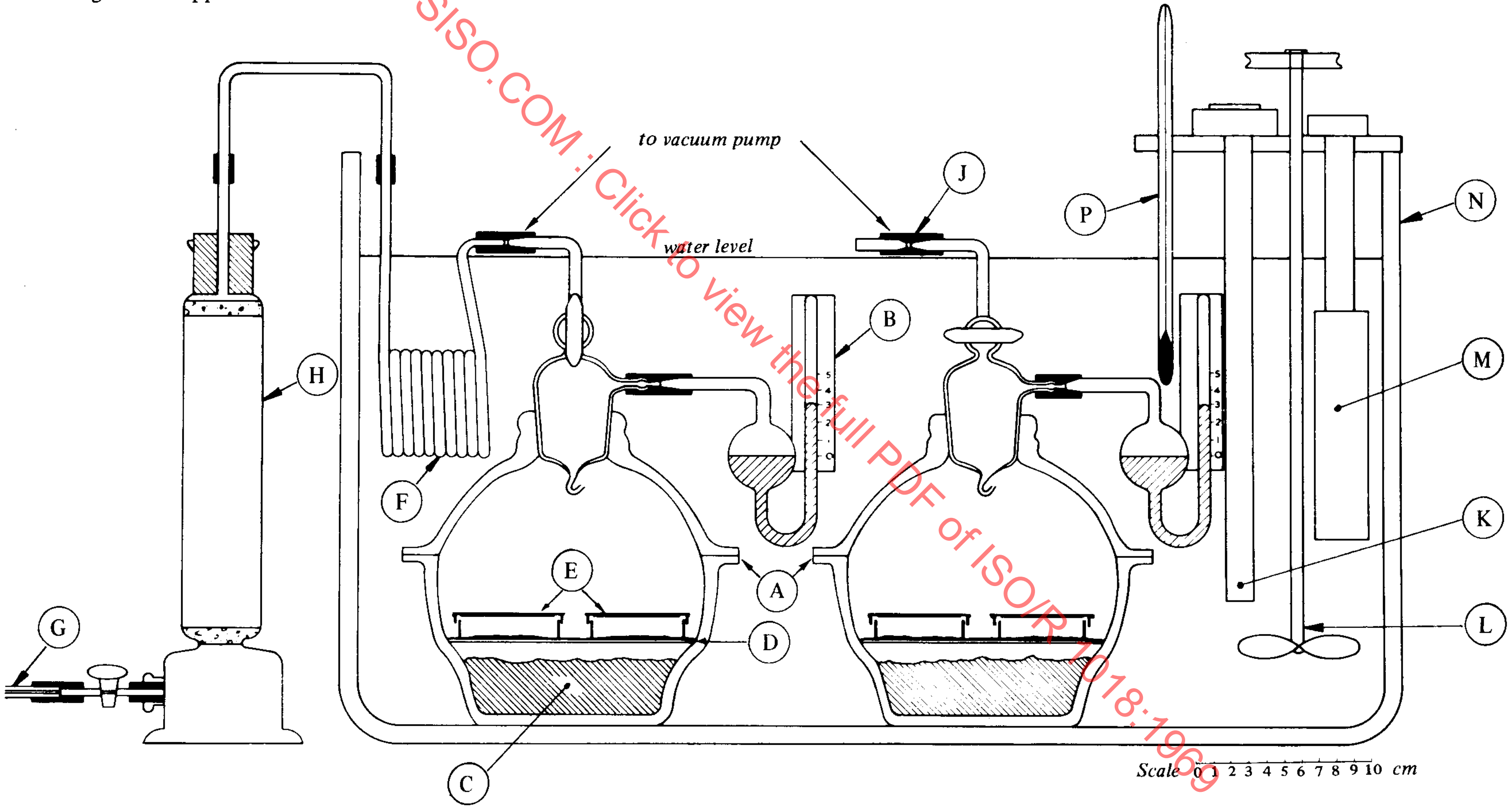
The means of the results of duplicate determinations, carried out in two different laboratories, on representative portions taken from the same analysis sample, should not differ by more than the above value.

Dimensions in millimetres



Ⓐ	Double-walled vessel
Ⓑ	Double-walled lid in two pieces
Ⓒ	Propeller – pitch 15°
Ⓓ	Potassium sulphate pulp
Ⓔ	Dishes
Ⓕ	Solid brass plug
Ⓖ	Screw clamp
Ⓗ	Rubber gasket
Ⓙ	Foam rubber
Ⓚ	Thermometer
Ⓛ	Positioning studs

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



Ⓐ Reduced pressure vessels, suitably weighted inside or outside to overcome buoyancy
Ⓑ Mercury manometer
Ⓒ Pulp of potassium sulphate and water
Ⓓ Glass or corrosion-resistant metal stand to support the samples, also serving as a screen against froth from the potassium sulphate pulp. A second parallel plate over the one actually covering the pulp prevents fouling of the support for the dishes
Ⓔ Samples of coal contained in suitable glass or corrosion-resistant metal dishes
Ⓕ Spiral of copper tubing
Ⓖ Capillary tubing of suitable length and bore
Ⓗ Tower packed with fresh magnesium perchlorate
Ⓙ Rubber seal
Ⓚ Thermoregulator
Ⓛ Stirrer
Ⓜ Heater
Ⓝ Tank, preferably with glass sides
Ⓟ Thermometer

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