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# INTERNATIONAL STANDARD



# 1018

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

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## Hard coal — Determination of moisture-holding capacity

*Houille — Détermination de la capacité de rétention d'humidité*

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**Descriptors** : coal, chemical analysis, determination of content, humidity, holding capacity.

## 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 1018 and found it technically suitable for transformation. International Standard ISO 1018 therefore replaces ISO Recommendation R 1018-1969 to which it is technically identical.

ISO Recommendation R 1018 was approved by the Member Bodies of the following countries :

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

The Member Bodies of the following countries expressed disapproval of the Recommendation on technical grounds :

Canada  
Japan  
U.S.A.

The Member Bodies of the following countries disapproved the transformation of ISO/R 1018 into an International Standard :

Czechoslovakia  
Japan

# Hard coal — Determination of moisture-holding capacity

## 0 INTRODUCTION

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.

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the moisture-holding capacity of hard coals.

## 2 REFERENCE

ISO 1988, *Hard coal — Sampling*.

## 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 figure 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 a two-blade propeller at approximately 1 500 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**, made of glass or corrosion-resistant metal, approximately 50 mm diameter by 10 mm deep, with well-fitting lids.

### 5.2 Reduced pressure method

**5.2.1 Conditioning vessel** (see figure 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 figure 2), thermostatically controlled at a temperature of  $30 \pm 0,1$  °C.

**5.2.3 Dishes** made of glass or corrosion-resistant metal, 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 Apparatus for determining moisture** by a method which precludes oxidation of the coal.<sup>1)</sup>

**5.3.5 Filter paper**, diameter about 200 mm.

1) The apparatus and procedure of Method B described in ISO 589, *Hard coal — Determination of total moisture*, are suitable for this purpose and the description is reproduced in annex A.

## 6 PREPARATION OF SAMPLE

It is essential that the coal be in a fresh, unchanged state. If the sample cannot be examined immediately, it shall 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 1988. The production of an excessive amount of fines shall 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  $30 \pm 0,1^\circ\text{C}$  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.

### 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 min, 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 7.2). Place the dish in the conditioning vessel, controlled to a temperature of  $30 \pm 0,1^\circ\text{C}$ , 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 h (see 7.3.2, note 1). 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 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 2 to 2,5 kPa. Place the vessel into the water bath, maintained at a temperature of  $30 \pm 0,1^\circ\text{C}$ , and re-evacuate. The pressure should rise quickly to about 4 kPa, which is the vapour pressure of the saturated solution of potassium sulphate at  $30^\circ\text{C}$ . If the pressure rises above 4 kPa, re-evacuate the vessel without otherwise disturbing it.

After a period of  $24 \pm 2$  h, with the vessel still in the water bath, restore the pressure to atmospheric by slowly admitting dry air at  $30^\circ\text{C}$  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 figure 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 tube G (see figure 2), so that the time taken to restore atmospheric pressure is approximately 15 min. 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.

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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, is given by the formula

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

where

$m_1$  is the mass, in grams, of empty dish and lid;

$m_2$  is the mass, in grams, of coal, dish and lid after conditioning;

$m_3$  is the mass, in grams, of dried coal, dish and lid.

The result, preferably the mean of duplicate determinations (see clause 9), shall 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	
	Repeatability	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, shall 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, shall not differ by more than the above value.

## 10 TEST REPORT

The test report shall include the following particulars :

- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard, or regarded as optional.

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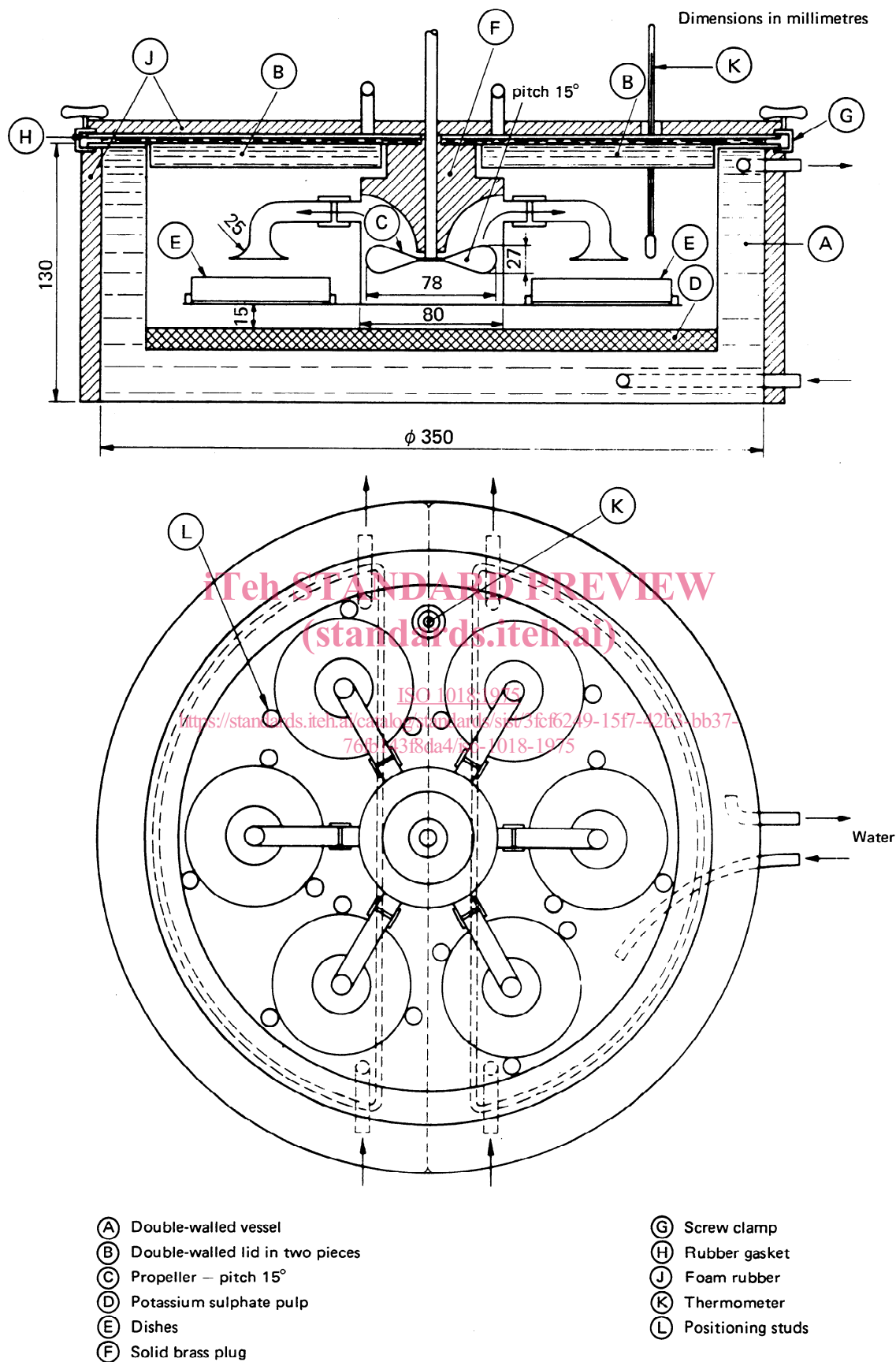


FIGURE 1 — Apparatus for determination of moisture-holding capacity of coal (atmospheric pressure method)

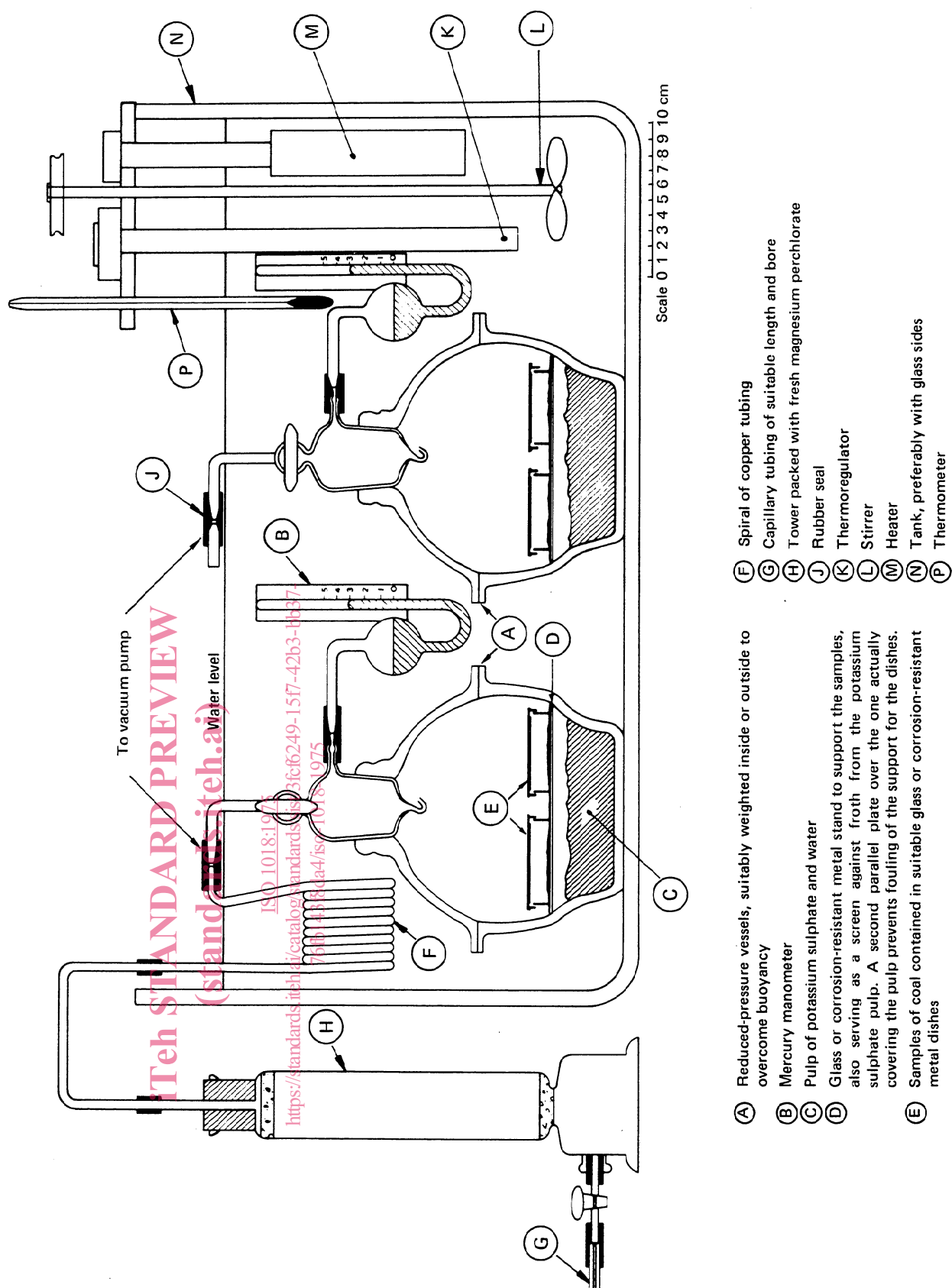


FIGURE 2 — Apparatus for determination of moisture-holding capacity of coal (reduced pressure method)



## ANNEX A

### DETERMINATION OF MOISTURE IN THE CONDITIONED COAL

#### A.1 REAGENTS

**A.1.1 Nitrogen**, dry, and containing less than 30 ppm of oxygen (see annex B).

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

#### A.2 APPARATUS

**A.2.1 Nitrogen oven**, capable of being maintained at a temperature within the range 105 to 110 °C and 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 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 min and weigh to within 0,2 mg.

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

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ANNEX B

### NITROGEN PURIFICATION TRAIN

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**B.1** It is essential that the nitrogen used for ventilating the oven should be comparatively pure, since even the small residual amount of oxygen found in commercial nitrogen causes oxidation of heated low-rank coal and hence produces a low result in the moisture figure determined by the loss in mass on drying. A good purification train for nitrogen is therefore required. A suitable one, capable of dealing with at least 600 ml per minute at a pressure of a few millibars, consists of a quartz tube 500 mm long and 37 mm internal diameter, containing 1,2 kg of reduced copper in wire form. A convenient source of the gas is a cylinder of compressed nitrogen. The purification tube is heated by a suitable furnace, which encloses the tube to a length of 380 mm and allows part of the copper wire filling to protrude approximately 80 mm at the exit end in order to produce a temperature drop in the gas stream. The reduced copper is heated to approximately 500 °C. The last traces of oxygen are removed by the very large surface area of the copper.

**B.2** If it is necessary to reduce any copper oxide formed in the nitrogen purification train, this can be done by passing hydrogen through the tube containing the copper, which is heated to 450 to 500 °C. During the reduction process the water formed is expelled to atmosphere. The tube should be purged with nitrogen before use, the reduced copper emptied from the tube and any agglomerated material broken down and the tube refilled. This avoids channelling.

**B.3** Finally pass the purified nitrogen through a column of magnesium perchlorate to remove traces of moisture which may be present.

**B.4** In the absence of a purification train, a commercial source of nitrogen may be employed, provided that the gas contains less than 30 ppm of oxygen.