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Part 6

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English version

Methods of testing cement; Determination of fineness.

Méthodes d'essai des ciments; Détermination de la finesse.

Prüfverfahren für Zement; Bestimmung der Mahlfeinheit.

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CEN

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BRIEF HISTORY

This European Standard was drawn up by the Technical Committee CEN/TC 51 'Cement' the Secretariat of which is held by IBN.

In accordance with the Common CEN/CENELEC Rules, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom

Foreword

The standard EN 196 on methods of testing cement consists of the following Parts:

Part 1: Determination of strength

Part 2: Chemical analysis of cement

Part 3: Determination of setting time and soundness

Part 4: Quantitative determination of constituents

Part 5: Pozzolanicity test for pozzolanic cements

Part 6: Determination of fineness

Part 7: Methods of taking and preparing samples of cement

Part 21: Determination of the chloride, carbon dioxide and alkali content of cement

Contents

1 Object and field of application

This European Standard describes two methods of determining the fineness of cement.

The sieving method serves only to demonstrate the presence of coarse cement particles. This method is primarily suited to checking and controlling the production process.

With the air permeability method (Blaine) the specific surface (mass related surface) is measured by comparison with a reference cement sample. The determination of the specific surface serves primarily to check the consistency of the grinding process of one and the same plant. This method only allows a limited assessment to be made of the properties of the cement in use. [1)]

The methods are applicable to all the cements defined in ENV197 [2)].

2 References

ENV 197	Cement : Composition, specifications and conformity criteria 2)
ISO 383-1976	Laboratory glassware - Interchangeable conical ground joints
ISO 565-1983	Test sieves - Woven metal wire cloth, perforated plate and electroformed sheet - Nominal sizes of openings
ISO 3310/1- 1982	Test sieves - Technical requirements and testing Part 1: Test sieves of metal wire cloth
ISO 4803-1978	Laboratory glassware - Borosilicate glass tubing

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1) The air permeability method may not give significant results for cements containing ultrafine materials.

2) At present at the draft stage.

3 Sieving method

3.1 Principle

The fineness of cement is measured by sieving it on standard sieves. The proportion of cement of which the grain sizes are larger than the specified mesh size is thus determined.

A reference sample having a known proportion of material coarser than the specified mesh size is used for checking the specified sieve.

3.2 Apparatus

3.2.1 ***Test sieve***, comprising a firm, durable, non-corrodible, cylindrical frame of 150 mm to 200 mm nominal diameter and 40 mm to 100 mm depth, fitted with 90 µm mesh sieve cloth of woven stainless steel, or other abrasion-resisting and non-corrodible metal wire.

The sieve cloth shall comply with the requirements of table 1 of ISO 565-1983 and ISO 3310/1 and shall be free of visible irregularities in mesh size when inspected optically by the methods of ISO 3310/1. A tray fitting beneath the sieve frame and a lid fitting above it shall be provided to avoid loss of material during sieving.

3.2.2 ***Balance***, capable of weighing up to 10 g to the nearest 10 mg.

3.3 Material for checking the sieve

A reference material of known sieve residue shall be provided for checking the sieve.

The material shall be stored in sealed, airtight containers to avoid changes in its characteristics due to absorption or deposition from the atmosphere. The containers shall be marked with the sieve residue of the reference material.

3.4 Procedure

3.4.1 *Determination of the cement residue*

Agitate the sample of cement to be tested by shaking for 2 min in a stoppered jar to disperse agglomerates. Wait 2 min. Stir the resulting powder gently using a clean dry rod in order to distribute the fines throughout the cement.

Fit the tray under the sieve. Weigh approximately 10 g of cement to the nearest 0,01 g and place it in the sieve, being careful to avoid loss. Disperse any agglomerates. Fit the lid over the sieve. Agitate the sieve by swirling, planetary and linear movements until no more fine material passes through it. Remove and weigh the residue. Express its mass as a percentage, R_1, of the quantity first placed in the sieve to the nearest 0,1 %. Gently brush all the fine material off the base of the sieve into the tray.

Repeat the whole procedure using a fresh 10 g sample to obtain R_2. Then calculate the residue of the cement R as the mean of R_1 and R_2 as a percentage, expressed to the nearest 0,1 %.

When the results differ by more than 1 % absolute, carry out a third sieving and calculate the mean of the three values.

The sieving process is carried out manually by a skilled and experienced operator.

NOTE - Alternatively a sieving machine may be used provided that it can be shown to give the same results as the manual operation.

3.4.2 *Checking the sieve*

Agitate the sample of cement to be tested by shaking for 2 min in a stoppered jar to disperse agglomerates. Wait 2 min. Stir the resulting powder gently using a clean dry rod in order to distribute the fines throughout the cement.

Fit the tray under the sieve. Weigh approximately 10 g of the reference material (3.3) to the nearest 0,01 g and place it in the sieve, being careful to avoid loss. Carry out the sieving procedure as in 3.4.1 including the repeat determination of residue to yield two values P_1 and P_2 expressed to the nearest 0,1 %.

The two values of P_1 and P_2 for a satisfactory sieve should differ by not more than 0,3 %. Their mean P characterizes the state of the sieve.

Given the known residue on the 90 µm mesh of the reference material, R_0, calculate R_0/P as the sieve factor, F, expressed to the nearest 0,01. The residue, R, determined as in 3.4.1 shall be corrected by multiplying by F, which may have a value of 1,00 ± 0,20.

Check the sieve after every 100 sievings.

NOTE - Any other checking procedure, such as the optical methods described in ISO 3310/1 may be used. All sieves will wear slowly and consequently their sieve factor, F, will slowly change.

3.5 Expression of results

Report the value of R, to the nearest 0,1 %, as the residue on the 90 µm (ISO 565) sieve for the cement tested.

The standard deviation of the repeatability is about 0,2 % and of the reproducibility is about 0,3 %.

NOTE - Where there is local difficulty in obtaining ISO sieves, the same procedure can be followed with the nearest available Standard sieve but the report is to state on which Standard sieve mesh the cement residue has been determined.

4 Air permeability method (Blaine method)

4.1 Principle

The fineness of cement is measured as specific surface by observing the time taken for a fixed quantity of air to flow through a compacted cement bed of specified dimensions and porosity. Under standardized conditions the specific surface of cement is proportional to $\sqrt{t}$ where t is the time for a given quantity of air to flow through the compacted cement bed. The number and size range of individual pores in the specified bed are determined by the cement particle size distribution which also determines the time for the specified air flow.

The method is comparative rather than absolute and therefore a reference sample of known specific surface is required for calibration of the apparatus.

4.2 Apparatus

4.2.1 ***Permeability cell***. The cell shall be a rigid right cylinder of the dimensions and tolerances shown in figure 1 a). It shall be of austenitic stainless steel or other abrasion-resisting, non-corrodible material. The top and bottom faces shall be flat and normal to the axis of the cylinder, as shall the upper surface of the ledge at the bottom of the cell. The outer surface of the cylinder shall be tapered to form an airtight fit with the conical socket of the manometer (ISO 383, Joint 19/34).

4.2.2 ***Perforated disc***. The disc shall be of non-corrodible metal, perforated with 30 to 40 holes of 1 mm diameter, and shall have the dimensions and tolerances shown in figure 1 b). When in position on the ledge in the cell, its plane surfaces shall be normal to the axis of the cell.

4.2.3 ***Plunger***. The plunger is a piston, capable of sliding freely in the measuring cell by means of a clearance to be applied in such a way that, when the cap of the plunger comes to rest on the upper face of the cell cylinder, a distance of 15 ± 1 mm will be maintained between the upper face of the perforated disc and the lower face of the piston.

This piston shall be provided with a flat connected to an annulus around the head to enable air to escape.

The plunger shall be of austenitic stainless steel or other abrasion-resisting and non-corrodible material; it shall have the dimensions and tolerances shown in figure 1 c). A plunger can only be used with the corresponding cell the dimensions of which match within the permitted tolerances.

4.2.4 ***Manometer***. The manometer shall be a rigidly and vertically mounted U-tube of borosilicate glass tubing (ISO 4803) arranged as in figure 1 d) and having the dimensions and tolerances shown in this figure.

One arm of the manometer shall be provided at the top with a conical socket (ISO 383, Joint 19/34) to form an airtight fit with the conical surface of the cell. The same arm shall also have four etched lines and a T-joint whose positions shall have the dimensions and tolerances shown in figure 1 d). The side branch of the T-joint shall lead to an airtight stopcock beyond which shall be attached a suitable aspiration device such as the rubber tube and bulb shown in figure 1 d).

Fill the manometer tube with the liquid (4.2.5) to wet the inner surface. Empty the tube and refill it so that the manometer liquid is level with the lowest etched line (l1 in figure 1 d)). This manometer liquid shall be changed (or cleaned) after servicing or before a new calibration.

NOTE - Other forms of cell and plunger and other arrangements of the joint between cell and manometer may be used provided that they can be shown to give the same results as the specified apparatus.

4.2.5 ***Manometer liquid***. The manometer shall be filled to the level of the lowest etched line (l1 in figure 1 d)) with a non-volatile, non-hygroscopic liquid of low viscosity and density, such as dibutyl phthalate or light mineral oil.

4.2.6 ***Timer***, having a positive starting and stopping mechanism, readable to 0,2 s or better, and accurate to 1 % or better over time intervals up to 300 s.

4.2.7 ***Balance(s)***, capable of weighing about 3 g to the nearest 1 mg (for the cement) and about 50 g to 110 g to the nearest 10 mg for the mercury).

4.2.8 ***Pyknometer***, or other convenient means of determining the density of cement.

4.3 Materials

4.3.1 Mercury, of reagent grade or better.

4.3.2 **Reference cement**[3], of known specific surface.

4.3.3 **Light oil**, to prevent formation of mercury amalgam on the inner surface of the cell.

4.3.4 **Circular discs of filter paper**, having a smooth circumference adapted to the dimensions of the cell. The filter paper is of medium porosity (mean pore diameter 7 µm).

4.3.5 **Light grease**, for ensuring an airtight joint between cell and manometer, and in the stopcock.

4.4 Test conditions

The laboratory in which the air permeability test is carried out shall be maintained at a temperature of 20 ± 2 °C and a relative humidity not exceeding 65 %. All materials for test and calibration shall be at the laboratory temperature when used and shall be protected from absorption of atmospheric moisture during storage.

4.5 Compacted cement bed

4.5.1 ***Basis***

The compacted cement bed comprises a reproducible arrangement of cement particles with a specified volume of air included between the particles. This air volume is defined as a fraction of the total volume of the bed and is termed the porosity, e.

It follows that the volume fraction occupied by the cement particles is (1 - e). If V is the total volume of the bed, the absolute volume of cement is $V(1 - e)$ (cm^3), and the mass of cement, m is $\rho V(1 - e)$ (g) where ρ is the solid density of the cement particles (g/cm^3)

Thus, knowing ρ, a mass of cement can be weighed to produce a desired porosity, e, in the compacted bed of total volume V. The determination of ρ is described in 4.5.3 and that of V in 4.7.1.

4.5.2 ***Preparation of the sample***

Agitate the sample of cement to be tested by shaking for 2 min in a stoppered jar to disperse agglomerates. Wait 2 min. Stir the resulting powder gently using a clean dry rod in order to distribute the fines throughout the cement.

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3) At present reference cements are available from:

National Bureau of Standards, Office of Standard Reference Materials, Chemistry Building, Washington DC, 20234, USA.

4.5.3 *Determination of density*

Determine the density of the cement using a device such as a pyknometer (4.2.8). Use a non-reactive liquid in the determination. The quantity of cement used will depend on the nature of the apparatus but shall be such that the value of ρ determined is accurate to 0,01 g/cm^3. Verify this accuracy by a repeat determination and record the mean of the two determinations to the nearest 0,01 g/cm^3 as the density.

4.5.4 *Formation of the bed*

To give a cement bed of porosity e = 0,500 weigh a quantity of cement, m_1, calculated from

$$m_1 = 0{,}500\ \rho V \ (g) \qquad (1)$$

where

ρ is the density of the cement (g/cm^3) (4.5.3)

V is the volume of the cement bed (cm^3) (4.7.1).

This mass, correctly compacted, will produce a bed of porosity 0,500. Place the perforated disc (4.2.2) on the ledge at the bottom of the cell (4.2.1) and place on it a new filter paper disc (4.3.4). Ensure that the filter paper disc fully covers the perforated disc and is flat by pressing with a clean dry rod. Place the weighed quantity of cement, m_1, in the cell taking care to avoid loss. Tap the cell to level the cement. Place a second new filter paper disc on the levelled cement. Insert the plunger (4.2.3) to make contact with the filter paper disc. Press the plunger gently but firmly until the lower face of the cap is in contact with the cell. Slowly withdraw the plunger about 5 mm, rotate it through 90 ° and gently but firmly press the bed once again until the plunger cap is in contact with the cell. The bed is now compacted and ready for the permeability test. Slowly withdraw the plunger.

NOTE - Too rapid and vigorous pressing may change the particle size distribution and therefore change the specific surface of the bed. The maximum pressure should be that comfortably exerted by a thumb on the plunger.

4.6 **Air permeability test**

4.6.1 *Basis*

The specific surface, S, is given in 4.9.1 but is conveniently expressed as

$$S = \frac{K}{\rho} \times \frac{\sqrt{e^3}}{(1 - e)} \times \frac{\sqrt{t}}{\sqrt{0{,}1\ \eta}} \ (cm^2/g) \qquad (2)$$

where

K is the apparatus constant (4.7.2)

e is the porosity of the bed

t is the measured time (s)

ρ is the density of cement (g/cm³) (4.5.3)

η is the viscosity of air at the test temperature taken from table 1 (Pa.s)

With the specified porosity of e = 0,500 and temperature of 20 ± 2 °C

$$S = \frac{524,2\ K \times \sqrt{t}}{\rho}\ (cm^2/g) \qquad (3)$$

4.6.2 *Procedure*

Insert the conical surface of the cell into the socket at the top of the manometer, using if necessary a little light grease (4.3.5) to ensure an airtight joint. Take care not to disturb the cement bed.

Close the top of the cylinder with a suitable plug. Open the stopcock and with gentle aspiration raise the level of the manometer liquid to that of the highest etched line (8 in figure 1 d)). Close the stopcock and observe that the level of the manometer liquid remains constant. If it falls, remake the cell/manometer joint and check the stopcock. Repeat the leakage test until the improved sealing produces a steady level of the liquid. Open the stopcock and by gentle aspiration adjust the level of the liquid, to that of the highest etched line. Close the stopcock. Remove the plug from the top of the cylinder. The manometer liquid will begin to flow. Start the timer as the liquid reaches the second etched line (9 in figure 1 d)) and stop it when the liquid reaches the third etched line (10 in figure 1 d)). Record the time, t, to the nearest 0,2 s and the temperature to the nearest 1 °C.

Repeat the procedure on the same bed and record the additional values of time and temperature. Prepare a fresh bed of the same cement with a second sample following the procedure of 4.5.4 or, where there is little cement available, by breaking up the first bed and reforming it as in 4.5.4. Carry out the permeability test twice on the second bed, recording the values of time and temperature as before.

4.7 Calibration of apparatus

4.7.1 *Determination of the bed volume*

Owing to the need for clearance between the cell and the plunger, the volume of the compacted cement bed varies for each cell-plunger combination. The volume of the compacted cement bed shall be established for a given cell-plunger clearance. This volume is to be determined as follows.

Apply a very thin film of light mineral oil (4.3.3) to the cell interior. Place the perforated disc on the ledge in the cell. Place two new filter paper discs on the perforated disc and ensure that each covers the base of the cell whilst lying flat by pressing with a rod.

Fill the cell with mercury (4.3.1). Remove any air bubbles with a clean dry rod. Ensure that the cell is full by pressing a glass plate on the mercury surface until it is flush with the cell top. Empty the cell, weigh the mercury to the nearest 0,01 g, m_2, and record the temperature. Remove one filter paper disc. Form a compacted cement bed by the method described in 4.5.4 and place on it a new filter paper disc. Refill the cell with mercury, removing air bubbles and levelling the top as before. Remove the mercury, weigh it to the nearest 0,01 g, m_3, and check the temperature. The bed volume V is given by

$$V = \frac{m_2 - m_3}{\rho_H} \ (cm^3) \qquad (4)$$

where ρ_H is the density of mercury at the test temperature taken from table 1.

Repeat the procedure with fresh cement beds until two values of V are obtained differing by less than 0,005 cm^3. Record the mean of these two values as V.

NOTE - Care should be taken to avoid spilling or splashing the mercury and any contact between it and the operator's skin and eyes.

4.7.2 *Determination of the apparatus constant*

From a supply of reference cement of known specific surface (4.3.2) prepare a compacted cement bed and measure its permeability by the procedures given in 4.5.2, 4.5.3, 4.5.4 and 4.6.2. Record the time, t, and the temperature of test. Using the same bed repeat twice the procedure of 4.6.2 and record the two further values of time and of temperature. Repeat the whole on two further samples of the same reference cement. For each of the three samples calculate the means of the three times and temperatures. For each sample calculate

$$K = \frac{S_o \rho_o (1 - e) \sqrt{0,1 \eta_o}}{\sqrt{e^3} \sqrt{t_o}} \qquad (5)$$

where

S_o is the specific surface of the reference cement (cm^2/g)

ρ_0 is the density of the reference cement (g/cm³)

t_0 is the mean of the three measured times (s)

η_0 is the air viscosity at the mean of the three temperatures (Pa.s) (table 1)

With the specified porosity of e = 0,500

$$K = 1{,}414 \cdot S_0 \rho_0 \frac{\sqrt{0{,}1\,\eta_0}}{\sqrt{t_0}} \qquad (6)$$

Take the mean of the three values of K as the constant K for the apparatus.

4.7.3 *Recalibration*

Repeated use of the apparatus may cause changes in the cement bed volume and in the apparatus constant (because of the wear of cell, plunger and perforated disc). These changes can be determined with the help of a so-called secondary reference cement whose specific surface has been measured.

The cement bed volume and the apparatus constant shall be recalibrated with the reference cement:

a) after 1000 tests

b) in the case of using

- another type of manometer fluid
- another type of filter paper
- a new manometer tube

c) at systematic deviations of the secondary reference cement.

4.8 Special cements

Certain cements having unusual particle size distributions and, in particular, fine cements of higher strength grades may prove difficult to form into a compacted bed of porosity e = 0,500 by the method of 4.5.4. Should thumb pressure on the plunger cap fail to bring it in contact with the top of the cell or if, after making contact and removing the pressure the plunger moves upwards, the porosity of e = 0,500 shall be considered unattainable.

For such cases the porosity required for a well-compacted bed shall be determined experimentally. The mass of cement, m_4, weighed to make the bed as in 4.5.4 then becomes

$$m_4 = (1 - e_1)\,\rho_1 V \text{ (g)} \qquad (7)$$

where e_1 is the porosity determined experimentally.

4.9 Simplification of the calculations

4.9.1 *Basic formula*

The specific surface, S, of the cement under test is calculated from the formula

$$S = \frac{\rho_0}{\rho} \times \frac{(1 - e_0)}{(1 - e)} \times \frac{\sqrt{e^3}}{\sqrt{e_0^3}} \times \frac{\sqrt{0,1\ \eta_0}}{\sqrt{0,1\ \eta}} \times \frac{\sqrt{t}}{\sqrt{t_0}} \times S_0 \ (cm^2/g) \qquad (8)$$

where

S_0 is the specific surface of the reference cement (cm^2/g) (4.3.2)

e is the porosity of the bed of cement under test

e_0 is the porosity of the bed of reference cement (4.7.2)

t is the measured time for the cement under test (s)

t_0 is the mean of the three times measured on the reference cement (s) (4.7.2)

ρ is the density of the cement under test (g/cm^3) (4.5.3)

ρ_0 is the density of the reference cement (g/cm^3) (4.7.2)

η is the air viscosity at the test temperature taken from table 1 (Pa.s).

η_0 is the air viscosity at the mean of the three temperatures (table 1) for the reference cement (Pa.s)

4.9.2 *Effect of specified porosity*

Use of the specified porosity, e = 0,500, for both the reference and test cements simplifies formula 8 to

$$S = \frac{\rho_0}{\rho} \times \frac{\sqrt{0,1\ \eta_0}}{\sqrt{0,1\ \eta}} \times \frac{\sqrt{t}}{\sqrt{t_0}} \times S_0 \ (cm^2/g) \qquad (9)$$

In the case of cements requiring a porosity other than e = 0,500, formula 9 cannot be used unless a reference cement has been tested at that porosity.

4.9.3 *Effect of controlled temperature*

As will be seen in table 1, the value of $\sqrt{0,1\ \eta}$ ranges from 0,0013-3 at 18 °C to 0,001353 at 22 °C. Under the specified laboratory conditions a value of 0,001349 can be taken to apply with an extreme error of 0,3 % and a more probable error of 0,3 % or less. This further simplification leads to the following formula

$$S = \frac{\rho_0}{\rho} \times \frac{\sqrt{t}}{\sqrt{t_0}} \times S_0 \ (cm^2/g)$$

4.9.4 *Effect of density of cement*

The only remaining possibility of simplification is the elimination of the density (ρ) terms. This has previously been done where the only cements in question were pure Portland cements for which a value of ρ of 3,15 was assumed to apply. That assumption is known to produce errors of up to 1 %. With the increasing use of Class CE II, III and IV cements (see ENV 197 [4]) much greater errors are certain. This standard requires the density of cement to be determined and used in the calculation of specific surface.

4.10 **Expression of results**

Where the porosity is e = 0,500, the four times and temperatures resulting from the procedure of 4.6.2 shall be examined to check that the temperatures all fall within the specified range of 20 ± 2 °C. If they do, the mean of the four times shall be inserted in equation 3 or equation 10 and the resulting value of S, to the nearest 10 cm^2/g, shall be reported as the specific surface of the cement.

A differance of 1 % between the means of the fineness measurements carried out on two different powder beds from one and the same sample is acceptable.

The standard deviation of the repeatability is about 50 cm^2/g and of the reproducibility is about 100 cm^2/g.

Where the porosity is not e = 0,500, equation 8 shall be used and the result to the nearest 10 cm^2/g reported as the specific surface of the cement.

If, owing to a breakdown in control or for other reasons, the four temperatures do not lie within the specified range of 20 ± 2 °C, a value of S shall be calculated for each combination of time and temperature using equation 2 or equation 8. The mean of the four values of S shall be reported, to the nearest 10 cm^2/g, as the specific surface of the cement.

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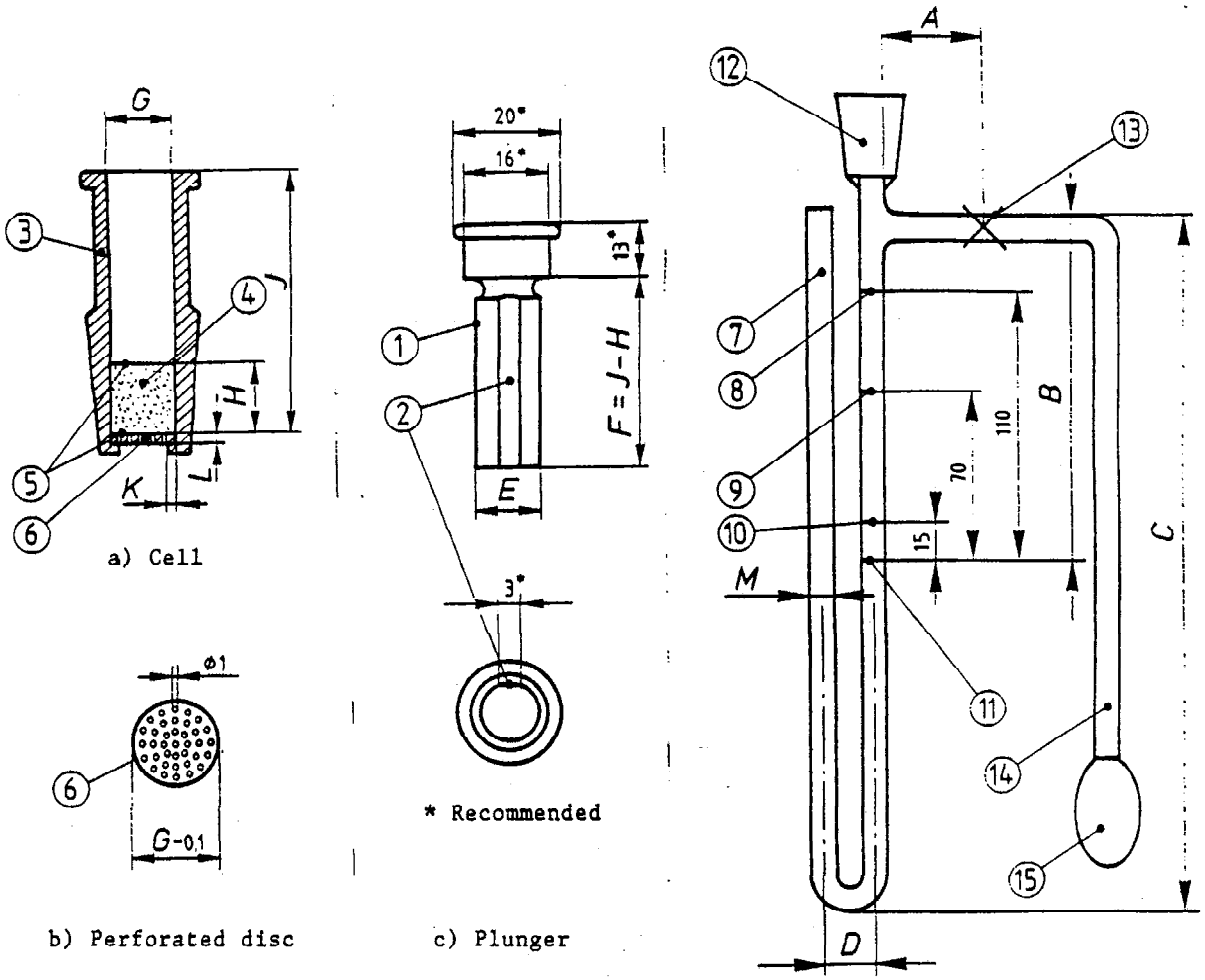
4) See page 4.



All dimensions in millimetres

Item	Description
1	Piston
2	Flat for air vent
3	Cell
4	Compacted cement bed
5	Filter paper disc
6	Perforated disc
7	Manometer
8, 9, 10, 11	Etched lines
12	Conical joint for cell
13	Stopcock
14	Rubber tube
15	Aspirator bulb

Recommended mm	Obligatory mm
A ≤ 50	G = 12,7 ± 0,1
B = 135 ± 10	
C = 275 ± 25	E = G - 0,1
D = 23 ± 1	
J = 50 ± 15	H = 15 ± 1
K = 0,8 ± 0,2	
L = 0,9 ± 0,1	
M = 9,0 ± 0,4	

Figure 1 - Blaine permeability apparatus

Table 1 – Density of mercury ρ_H, viscosity of air η and $\sqrt{0,1\ \eta}$ as function of the temperatures

Temperature °C	Density of mercury ρ_H g/cm³	Viscosity of air η Pa.s	$\sqrt{0,1\ \eta}$
16	13,560	0,000 018 00	0,0013 42
17	13,560	0,000 018 05	0,0013 44
18	13,550	0,000 018 10	0,0013 45
19	13,550	0,000 018 15	0,0013 47
20	13,550	0,000 018 19	0,0013 49
21	13,540	0,000 018 24	0,0013 51
22	13,540	0,000 018 29	0,0013 53
23	13,540	0,000 018 34	0,0013 54
24	13,540	0,000 018 39	0,0013 56

NOTE – Intermediate values shall be obtained by linear interpolation.