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Cements — Test methods — Part 6: Determination of fineness

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National foreword

Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Trade, Industry and Cooperatives established under Cap 327, of the Laws of Uganda, as amended. UNBS is mandated to co-ordinate the elaboration of standards and is

(a) a member of International Organisation for Standardisation (ISO) and

(b) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and

(c) the National Enquiry Point on TBT Agreement of the World Trade Organisation (WTO).

The work of preparing Uganda Standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of representatives of consumers, traders, academicians, manufacturers, government and other stakeholders.

Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council.

This Uganda Standard, US EAS 148-6: 2017, Cement — Test methods — Part 6: Determination of fineness, is identical with and has been reproduced from an East African Standard, EAS 148-6: 2017, Cement — Test methods — Part 6: Determination of fineness, and adopted as a Uganda Standard.

The committee responsible for this document is Technical Committee UNBS/TC 3, *Building and construction*.

This standard cancels and replaces US 100-6:2016, Cement — Test methods — Part 6: Determination of fineness (2nd Edition), which has been technically revised.

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EAS 148-6: 2017

ICS 91.100.10

EAST AFRICAN STANDARD

Cements — Test methods — Part 6: Determination of fineness

EAST AFRICAN COMMUNITY

Second Edition 2017

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Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

In order to achieve this objective, the Community established an East African Standards Committee mandated to develop and issue East African Standards.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

EAS 148-6: 2015 was prepared by Technical Committee EASC/TC/026, *Cement, lime, clay and related products.*

In the preparation of this East African Standard, reference was made to the following standard:

EN 196-6: 2005; *Methods testing cement — Part 6: Determination of fineness.*

Assistance derived from the above source is hereby acknowledged with thanks.

This second cancels and replaces the first edition (EAS 148-6:2000) which has been technically revised.

EAS 148 consists of the following parts, under the general title *Cement — Test methods*:

—*Part 1: Determination of strength*

— *Part 2: Chemical analysis of cement*

— *Part 3: Determination of setting times and soundness*

— *Part 4: Quantitative Determination of constituents*

— *Part 5: Pozzolanicity test for pozzolanic cement*

— *Part 6: Determination of fineness*

—*Part 7: Method of taking and preparing samples of cement*

—*Part 8: Heat of hydration — Solution method*

Cement — Test methods — Part 6: Determination of fineness

1 Scope

This East African Standard describes three methods for determining the fineness of cement and applies to all the cements defined in EAS 18-1.

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.

The air –jet sieving method measures the retention on sieving and is suitable for particles which substantially pass a 2.0- mm test sieve. It may be used to determine the particle size distribution of agglomerates of very fine particles. This method may be used with test sieves in a range of aperture sizes, for example 63 µm and 90 µm.

The air permeability method (Blaine) measures the specific surface (mass related surface) 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 enables a limited assessment to be made of the properties of the cement in use.

NOTE The air permeability method may not give significant results for cement containing ultrafine materials.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EAS 18 –1, *Cement — Part 1: Composition, specifications and conformity criteria for common cement*

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 3310 -1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 4803, *Laboratory glassware — Borosilicate glass tubing*

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 – 200 mm nominal diameter and 40 mm – 100 mm depth, fitted with, for example 90 μm, mesh sieve cloth of woven stainless steel, or other abrasion-resisting and non-corrodible metal wire.

The sieve cloth shall conform to the requirements of ISO 565 and 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.

NOTE Sieving may be carried out manually or on a sieving machine.

3.2.2 Balance, capable of weighing up to 25 g to the nearest 0.01 g

3.3 Material for checking the sieve

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

3.3.2 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

3.4.1.1 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.

3.4.1.2 Fit the tray under the sieve. Weigh (25 ± 0.5) g of cement to the nearest 0.01g 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.

3.4.1.3 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.

3.4.1.4 Repeat the whole procedure using a fresh 25 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 %.

3.4.1.5 When the results differ by more than 1 % absolute, repeat the whole procedure a third time and calculate the mean of the three values.

3.4.1.6 Sieving by the manual process requires a skilled and experienced operator.

3.4.2 Checking the sieve

3.4.2.1 Sieves should be cleaned and checked for damage after each sieving for example that the mesh is taut and is not dented or perforated. In addition, check the sieve after every 100 sievings as follows:

3.4.2.2 Agitate the sample of reference material, to be used for checking the sieve, by shaking or 2 min in a stoppered jar to disperse agglomerates. Wait 2 min. Stir the resulting powder gently using a clean dry rod to distribute the fines throughout the reference material.

3.4.2.3 Fit the tray under the sieve. Weigh approximately (25 ± 0.5) g of the reference material (3.3) to the nearest 0.01 g and place it in the sieve, being careful to avoid loss. Sieve the material in accordance with 3.4.1 including the repeat determination of residue to yield two values P_1 and P_2 expressed to the nearest 0.1 %.

3.4.2.4 The two values of P_1 and P_2 for a satisfactory sieve should differ by not more than 0.6 %. Their mean $\overline{P}$ characterizes the state of the sieve.

3.4.2.5 Given the known residue on the sieve 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.

3.4.2.6 When factor F exceeds the permitted value, 1.00 ± 0.20, the sieve shall be discarded.

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

3.5.1 Report the value of *R*, to the nearest 0.1 %, as the residue, the sieve mesh size and details of the cement tested.

3.5.2 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 should state on which standard sieve mesh the cement residue has been determined.

4 Air Permeability Method (Blaine Method)

4.1 Principle

4.1.1 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 airflow.

4.1.2 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.

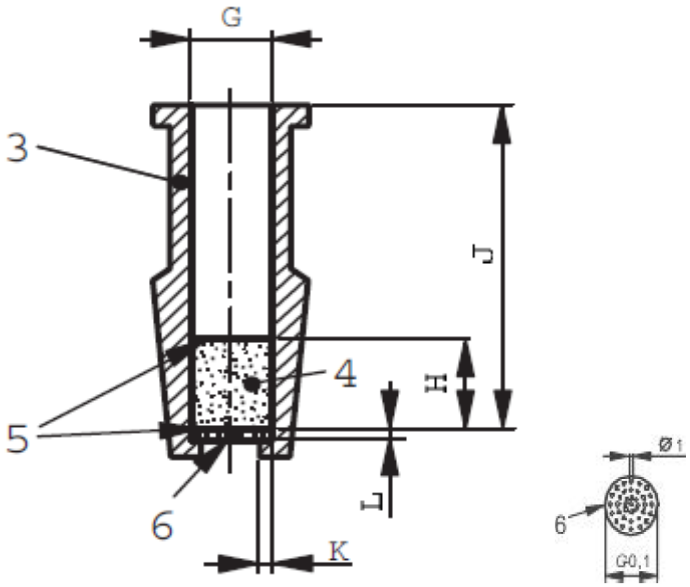
The disc shall be of non-corrodible metal, perforated with 30- 40 holes of 1- mm diameter, and shall have the dimensions and tolerances shown in Figure 1(b). When in position on the ledge in the cell, its plane surfaces shall be normal to the axis of the cell.

4.2.3 Plunger.

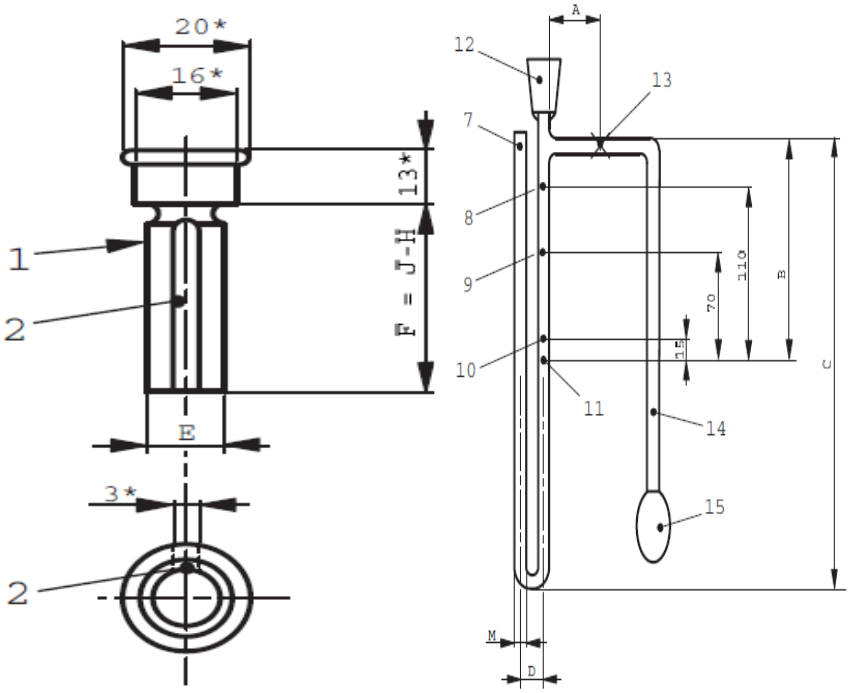
The plunger is a piston, capable of sliding freely in the measuring cell by means of a clearance to be applied in such a way that, when the cap of the plunger comes to rest on the upper face of the cell cylinder, a distance of (15 ± 1) mm will be maintained between the upper face of the perforated disc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

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

Dimensions in millimetres



a) Cell **b) Perforated disc**



* Recommended

c) Plunger

d) Manometer

Key

1	Piston
2	Flat for air vent
3	Cell
4	Compacted cement disc
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	spirator bulb

Key		**Recommended mm**	**Key**		**Obligatory mm**
A	Minimum dimension between conical joint and stopcock	≤ 50	G	Cell diameter at base of cell	12.7 ± 0.1
B	Dimension between upper arm of T-joint and lowest etched line on arm of manometer tube	135 ± 10	E	Diameter plunger/piston	G - 0.1
C	Dimension between upper arm of T-joint and base of U-tube	275 ± 25	H	Height of cement bed	15 ± 1
D	Dimension between centre lines of arms of U-tube	23 ± 1	F	Dimension/depth between tip of plunger and shoulder	J-H
J	Inner dimension/height of the cell	50 ± 15			
K	Width of cell ledge	0.8 ± 0.2			
L	Thickness perforated disc	0.9 ± 0.1			
M	Diameter of arms of manometer tube	9.0 ± 0.4			

Figure 1 — Blaine permeability apparatus

4.2.3.2 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 shall only be used with a

cell of the specified dimensions and tolerances such that, when used together, the specified distance between the upper face of the perforated disc and the lower face of the piston is satisfied.

4.2.4 Manometer

4.2.4.1 The manometer shall be a rigidly and vertically mounted U-tube of borosilicate glass tubing conforming to ISO 4803 arranged as in Figure 1(d) and having the dimensions and tolerances shown in that figure.

4.2.4.2 One arm of the manometer shall be provided at the top with a conical socket conforming to ISO 383: 1976, 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)**.**

4.2.4.3 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 (11 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 (11 in Figure1 (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

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)

Balance(s), capable of weighing about 3 g to the nearest 0.001 g (for the cement) and about 50 g - 110 g to the nearest 0.01 g (for the mercury).

4.2.8 Apparatus

Apparatus, to determine the density of cement, for example pycnometer or Le Chatelier flask

4.3 Materials

4.3.1 **Mercury**, of reagent grade or better

4.3.2 **Reference cement**, of known specific surface

NOTE Results may vary when using cements of different types or when using cements of the same type from different suppliers.

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**, of medium porosity having a smooth circumference adapted to the dimensions of the cell.

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) ºC and a relative humidity not exceeding 65 %. All materials for test and calibration shall be a laboratory temperature when used and shall be protected from absorption of atmospheric moisture during storage.

4.5 Compacted cement bed

4.5.1 Basis

4.5.1.1 The compacted cement bed comprises a reproducible arrangement of cement particles with a specified volume (see 4.5.4) 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.

4.5.1.2 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)* in cubic centimetres (cm^3), and the mass of cement, *m* is ρ *x V (1 - e)* in grams (g) where ρ is the solid density of the cement particles in grams per cubic centimetre (g/cm^3).

4.5.1.3 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 of 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.

4.5.3 Determination of density

Determine the density of the cement using a device such as a pycnometer (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

$$m_1 = 0.500\ \rho V, \text{ in grams (g)} \qquad (1)$$

where

ρ is the density of the cement in grams per cubic centimetre (g/cm^3) (4.5.3); and

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 90º 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, in square centimetres per gram (cm^2/g), as

$$S = \frac{k}{\rho} \times \frac{\sqrt{e^3}}{(1-e)} \times \frac{\sqrt{t}}{\sqrt{10 \times \eta}} \quad (2)$$

where

K is the apparatus constant (4.7.2);

e is the porosity of the bed;

t is the measured time in seconds (s);

ρ is the density of cement in grams per cubic centimetre (g/cm^3) (4.5.3);

η is the viscosity of air at the test temperature taken from Table 1, in Pascal second (Pa.s).

With the specified porosity of *e* = 0.500 and temperature of (20 ± 2) °C

$$S = \frac{52.43K \times \sqrt{t}}{\rho} (cm^2/g) \quad (3)$$

4.6.2 Procedure

4.6.2.1 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.

4.6.2.2 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 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.

4.6.2.3 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.

4.6.2.4 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

4.7.1.1 Determination by mercury volume

4.7.1.1.1 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.

4.7.1.1.2 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.

4.7.1.1.3 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. Using a trial quantity of 2.90 g of cement (see Note 1) form a compacted cement bed (see Note 2) using 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

$$V = \frac{m_2 - m_3}{\rho Hg} (cm^3) \quad (4)$$

where

ρ_{Hg} 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.

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

NOTE 1 It is not necessary either to use the reference cement (4.3.2) or to achieve particular bed porosity in the determination of the bed volume.

NOTE 2 The compacted bed of cement should be firm. If it is too loose or if the cement cannot be compacted to the point where the lower face of the cap is in contact with the cell (see 4.5.4), the trial quantity of cement should be adjusted.

4.7.1.2 Determination of measurement

4.7.1.2.1 Apparatus

4.7.1.2.1.1 **Depth gauge**, accurate to 0.01 mm

4.7.1.2.1.2 **Internal micrometer**, accurate to 0. 01 mm

4.7.1.2.2 Procedure

4.7.1.2.2.1 Calibrate the cement bed volume by dimensional measurements in a room maintained at a temperature of (20 ± 2) °C and with relative humidity not exceeding 65 %.

4.7.1.2.2.2 Carry out all dimension measurements to the nearest 0.01 mm.

4.7.1.2.2.3 Place two filter paper discs on the perforated disc placed at the bottom of the cell. Measure the inner dimension (height: J) or the cell with a depth gauge. Repeat five times and record the mean to the nearest 0.01 mm.

4.7.1.2.2.4 Stand the plunger on its cap and, using the depth gauge, measure the length of plunger inside the cell (*F*) as the "depth" from the tip to the shoulder. Repeat five times and record the mean to the nearest 0.01 mm.

4.7.1.2.2.5 Measure the cell diameter (*G*) at the bottom (where the cement bed is formed) with the internal micrometer to obtain its radius *(r =G/2).*Repeat five times and record to the nearest 0.01 mm.

4.7.1.2.2.6 Calculate the cement bed height *(H)* to the nearest 0.01 mm as *J – F*.

4.7.1.2.2.7 The bed volume (*V*), in cubic centimetres (cm^3), is given by:

$$V = H \times r^2 \times 3.14/1000 \quad (5)$$

Where

H is the cement bed height, in millimetres (mm);

r is the radius of the cell, in millimetres (mm);

Record the result in cubic centimetres (cm^3) to the nearest 0.001 cm^3.

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

$$K = \frac{S_O P_O (1-e)\sqrt{10 \times \eta_O}}{\sqrt{e^3 \sqrt{t_O}}} \quad (6)$$

where

S_o is the specific surface of the reference cement in square centimetres per gramme (cm^2/g);

ρ_o is the density of the reference cement in grams per cubic centimetre (g/cm^3);

t_o is the mean of the three measured times in seconds (s);

η_o is the air viscosity of the mean of the three temperatures, in Pascal seconds (Pa.s) (Table 1).

With the specified porosity of e = 0.500

$$K = 1.414\ S_o \rho_o \frac{\sqrt{10 \times \eta_o}}{\sqrt{t_o}} \quad (7)$$

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 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) when another type of filter paper or a new perforated disc is used;

c) when systematic deviations of the secondary reference cement results are noted; and

d) when another type of manometer fluid and/or a new manometer tube have been introduced their apparatus constant only shall be recalibrated with reference cement.

4.8 Special cements

4.8.1 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 it, after making contact and removing the pressure the plunger moves upwards, the porosity of e = 0.500 shall be considered unattainable.

4.8.2 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, in grams (g);

$$m_4 = (1 - e_1)\rho_1 V \qquad (8)$$

where

e_1 is the porosity determined experimentally.

4.9 Simplification of the calculations

4.9.1 Basic equation

The specific surface, S, of the cement under test is calculated, in square centimetres per gram (cm^2/g), from equation (9);

$$S = \frac{\rho o}{\rho} \times \frac{(1 - e_o)}{(1 - e)} \times \frac{\sqrt{e^3}}{\sqrt{e_o{}^3}} \times \frac{\sqrt{10 \times \eta_o}}{\sqrt{10 \times \eta}} \times \frac{\sqrt{t}}{\sqrt{t_o}} \times S_o \qquad (9)$$

where

S_o is the specific surface of the reference cement in square centimetres per gram (cm^2/g) (4.3.2);

e is the porosity of the bed of cement under test;

e_o is the porosity of the bed of reference cement (4.7.2);

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

t_o is the mean of three times measured on the reference cement in seconds (s) (4.7.2);

ρ is the density of the reference cement under test in grams per cubic centimetre (g/cm^3)(4.5.3);

ρ_o is the density of the reference cement in grams per cubic centimetre (g/cm^3) (4.7.2);

η is the air viscosity at the test temperature taken from Table 1, in Pascal seconds (Pa.s);

η_o is the air viscosity at the mean of the three temperatures (Table 1) for the reference cement in Pascal seconds (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 equation (9) to

$$S = \frac{\rho_o}{\rho} \times \frac{\sqrt{10 \times \eta_o}}{\sqrt{10 \times \eta}} \times \frac{\sqrt{t}}{\sqrt{t_o}} \times S_o \text{ in square centimetres per gram (cm}^2\text{/g)} \quad (10)$$

In the case of cements requiring a porosity other than e = 0.500, Equation (10) 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 $\sqrt{10 \times \eta}$ ranges from 0.013 454 at 18 °C - 0.013 524 at 22 °C. Under the specified laboratory conditions a value of 0.013 49 can be taken to apply with an extreme error of 0.5 % and a more probable error of 0.3 % or less. This further simplification leads to the following equation:

$$S = \frac{\rho_o}{\rho} \times \frac{\sqrt{t}}{\sqrt{t_o}} \times S_O \text{ in square centimetres per gram (cm}^2\text{/g)} \quad (11)$$

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 CEM II, III and IV cements (see FDEAS 18-1) much greater errors are certain.

4.10 Expression results

4.10.1 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 11 and the resulting value of *S,* to the nearest 10 cm^2/g, shall be reported as the specific surface of the cement.

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

4.10.3 The standard deviation of the repeatability is approximately 50 cm^2/g and of the reproducibility is approximately100 cm^2/g.

4.10.4 Where the porosity is not *e* = 0.500, equation 9 shall be used and the result to the nearest 10 cm^2/g reported as the specific surface of the cement.

4.10.5 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 3 (where *e*= 0.500) or Equation 9 (where *e* ≠ 0.500). 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.

NOTE To report the specific surface of the cement in SI units to the nearest square metre per kilogramme (m^2/kg), it is recommended to take the resulting value of *S* and divide it by 10.

Table 1— Density of mercury *ρHg*, viscosity of an air *η* and $\sqrt{10 \times \eta}$ as function of the temperatures[a]

Temperature ˚C	**Density of mercury *ρHg* g/cm³**	**Viscosity on air *η* *Pa s***	$\sqrt{10 \times \eta}$
16	13.560	0.000 018 00	0.013 416
17	13.560	0.000 018 05	0.013 435
18	13.550	0.000 018 10	0.013 454
19	13.550	0.000 018 15	0.013 472
20	13.550	0.000 018 19	0.013 487
21	13.540	0.000 018 24	0.013 506
22	13.540	0.000 018 29	0.013 524
23	13.540	0.000 018 34	0.013 543
24	13.540	0.000 018 39	0.013 561

a Intermediate values shall be obtained by linear interpolation.

5 Air-jet sieving method

5.1 Principle

The method determines the retention on sieving of particles which substantially pass a 2.0 -mm test sieve. The method can be used to determine the particle size distribution of the agglomerates of very fine particles. This method customarily uses test sieves with aperture sizes 63 µm or 90 µm.

NOTE Sieves with other aperture sizes may be used, preferably conforming to ISO 565.

5.2 Apparatus

5.2.1 Air-jet sieving apparatus of the general form shown in Figure 2. The apparatus shall be set to give a pressure difference of 2 kPa - 2.5 kPa across the sieve.

5.2.2 Test sieves, 200 mm diameter, aperture sizes for example 63 µm and 90 µm

NOTE The effective operation of some makes of air-jet apparatus can necessitate non-standard sieve frames and additional gaskets. In this case the sieving medium and general method of construction should comply with the requirements of this standard.

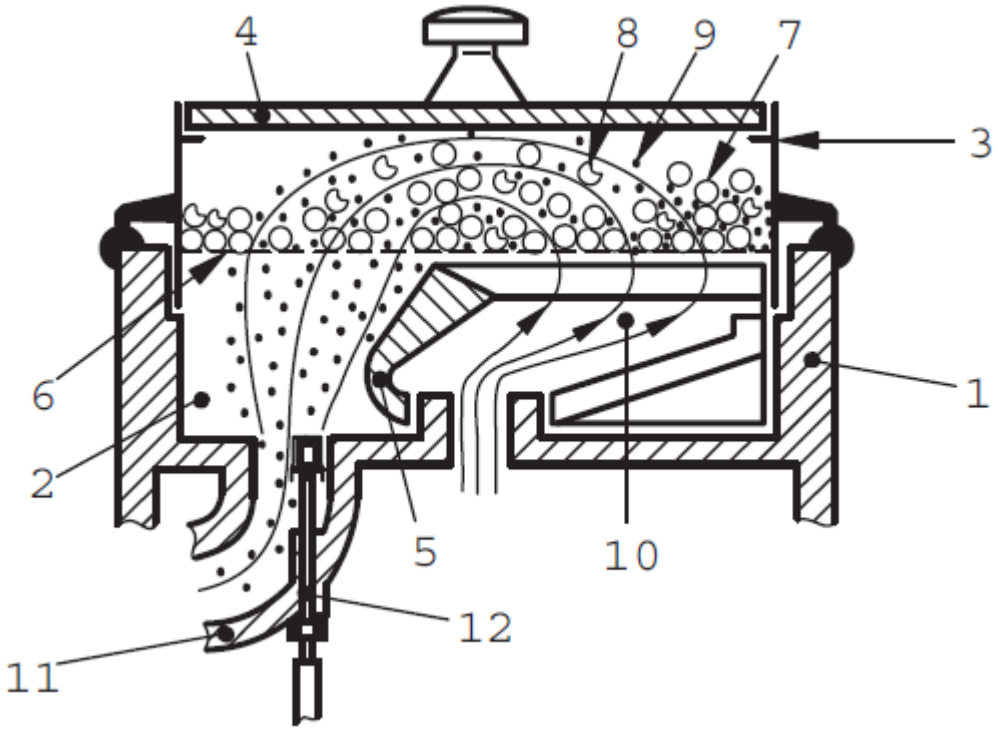
5.2.3 Trays or other suitable containers of sufficient size to contain the test portion

5.2.4 Balance, capable of weighing up to 25 g to the nearest 0.01g

5.2.5 Soft brush, for cleaning the mesh of the sieves, for example a camel hair brush

5.2.6 **Mallet**, if there is a tendency for material to adhere to the lid of the apparatus. A rubber or plastic tipped mallet is preferred

5.2.7 **Ventilated drying oven** (optional), thermostatically controlled to maintain a temperature of (105 ± 5) °C



Key

1 Housing

2 Dish

3 Sieve drum

4 Lid

5 Slit-nozzle

6 Sieve

7 Test sample

8 Oversize material

9 Undersize material

10 Air jet

11 Air discharge

12 Pressure gauge socket, with dust hood

Figure 2 — Air jet sieving apparatus

5.3 Procedure

5.3.1 If necessary, dry the laboratory sample to constant weight in the oven (5.2.7). Weigh to the nearest 0.01 g (25 ± 0.5) g of the cement (*m*). Fit the test sieve with the aperture size for example 90 µm into the apparatus and transfer all of the test portion onto the sieve mesh. Take care not to lose any of the test portion.

5.3.2 Fit the lid and switch on the apparatus. Check that the vacuum created is above the minimum value stated in the manufacturer's instructions, and that the slit nozzle is rotating properly.

5.3.3 If the material adheres to the lid of the apparatus, gently tap the centre of the lid with the mallet.

5.3.4 If the material agglomerates under the action of the air-jet, interrupt the sieving process, break up the agglomerates with the soft brush.

5.3.5 After (5.0 ± 0.2) min, switch off the apparatus and carefully remove the sieve. Transfer the material retained on the sieve into a tray or other suitable container. Carefully clean the mesh of the sieve over the tray using the soft brush.

5.3.6 Determine the mass of the residue, including the material brushed from the sieve mesh, and record the mass to the nearest 0.01 g.

5.3.7 Refit the sieve into the apparatus and transfer all of the residue back to the sieve. Repeat the weighing and sieving stages until the sieving end-point has been achieved, and record the end-point mass to the nearest 0.01 g. The sieving end-point is defined as being when not more than 0.2 % of the mass of the original test portion passes through the sieve in 3 min.

Express its mass as a percentage, R_1 of the quantity first placed in the sieve to the nearest 0.1 %.

5.3.8 Repeat the whole procedure using a fresh 25- 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 0.01 %.

5.4 Checking the sieve

Sieves should be cleaned and checked for damage after each sieving for example that the mesh is taut and it is not dented or perforated. In addition, check the sieve after every 100 sievings (see 3.4.2).

5.5 Expression of the results

The mass retained on each sieve expressed as per cent by mass (%), *m* ($P_{e.g\ 63\ or\ 90}$), is given by the following equation:

$$m(P\,e.g\ 63\,\text{or}\,90) = \frac{R \times 100}{m}$$

where

mm is the mass of the cement in grams (g);

RR is the mass of the residue retained on the test sieve in grams (g).

5.6 Repeatability and reproducibility

The precision of the method increases (standard deviation decreases) with the aperture size of the sieve. Typical values for determination of the residue on a 63- µm sieve are the following:

a) the standard deviation of repeatability is 0.1 %; and

b) the standard deviation of reproducibility 1.0 %.

Bibliography

[1] EN 196-6: 2005; *Methods testing cement — Part 6: Determination of fineness*

