12. TESTING OF CEMENT – PART 1.

12.1 Density

The density is the fundamental physical characteristic of the material. Density is defined by mass of a unit volume of a material substance, expressed as kilograms per cubic metre.

$$\rho = \frac{m}{V_h}$$

where ρ is density in $kg.m^{-3}$ ($g.cm^{-3}$)

m mass of the material sample in kg (g)

V_h volume of the specimen in m^3 (cm^3)

The mass can be determinate by scale (usually analytic balance), to determine volume of loose material is more difficult. For determination of cement density the pycnometric method is used.

12.1.1 Determination of Density by Pycnometric Method.

Pycnometric method is indirect method; the measuring of volume is substituted by several weighing of material in pycnometer.

Pycnometer is glass (or metal) bottle with a tight fitting stopper having a small hole in its centre. The hole in the stopper allows excess liquid to escape and the volume of the liquid in the pycnometer is always the same.

Density by pycnometry is calculate from basic formula:

$$\rho = \frac{m \times \rho_k}{m + m_4 - m_3}$$

where m is mass of the sample of the tested material ($m = m_2 - m_1$)

m_1 mass of dry empty pycnometer including stopper

m_2 mass of dry pycnometer with sample and stopper

m_3 mass of closed pycnometer with sample and measuring liquid

m_4 mass of the closed pycnometer with measuring liquid

ρ_k density of measuring liquid at tested temperature

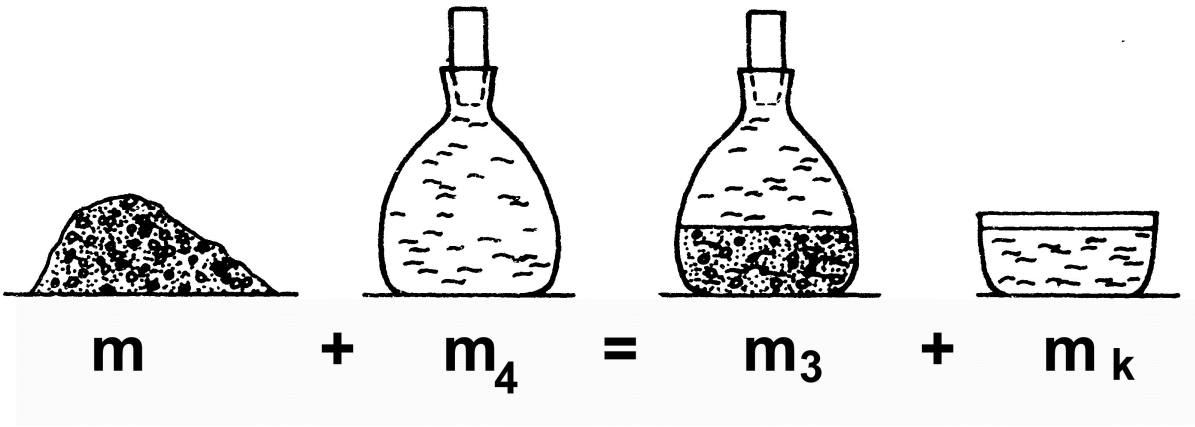
Note: Measuring liquid could not be water in case of measuring cement density! Usually is used alcohol.

Testing procedure

- weight mass of the empty pycnometer with stopper - m_1
- fill the pycnometer with measuring liquid. Replace stopper carefully, allowing excess liquid to escape through the hole in the stopper. Make sure there are no bubbles. Dry outside and weight – m_4
- fill the dry, empty pycnometer about 1/3 full of the sample. Closed it and weight again – m_2

- add measuring liquid to the sample. Fill pycnometer about 2/3 full. Mix up with caution and boil the pyknometer for 2 hours. After cooling refill with the liquid, closed the pycnometer and weight - $\mathbf{m_3}$
- calculate the mass of the sample from formula: $\mathbf{m} = m_2 - m_1$
- count the density from the basic formula.

Fig.:29 Derivation of the formula for density determined pycnometric method



where m_k is mass of liquid, volume of which was pushed up by volume of the solid material

12.2 Fineness

Standard EN 196-6 describes two methods of determining the fineness of cement:

- sieving method
- air permeability method (Blaine)

12.2.1 Sieving Method

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

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.

12.2.2 Air Permeability Method (Blaine Method)

The fineness of cement is measured as specific surface. Specific surface is expressed as the total surface area in square metres of all the cement particles in one kilogram of cement. The higher the specific surface is, the finer cement will be.

Principle of air permeability method is in observing the time taken for a fixed quantity of air to flow through compacted cement bed of specified dimension and porosity. Under standardised conditions the specific surface of cement is proportional to $\sqrt{\mathbf{t}}$ where $\mathbf{t}$ is the time for 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 determined 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.

A bed of cement is prepared in special permeability cell and have exact porosity e = 0,500 weight a quantity of cement. The weight of the cement is calculated from

$$m_1 = 0{,}500 \times \rho \times V \qquad [g]$$

where ρ is the density of the cement [$g.cm^{-3}$]
V volume of the cement bed [cm^3]

Specific surface S is expressed as

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

where K is the apparatus constant
e porosity of the bed
t the measured time [s]
ρ density of cement [$g.cm^{-3}$]
η the viscosity of air at the test temperature taken from Tab.:27

The apparatus constant is determinate by measuring permeability of reference cement of known specific surface. The procedure repeats three times for three samples of cement bed. For each sample is calculated constant K from formula:

$$K = S_0 \times \rho_0 \times \frac{(1-e)}{\sqrt{e^3}} \times \frac{\sqrt{0{,}1\eta_0}}{\sqrt{t_0}}$$

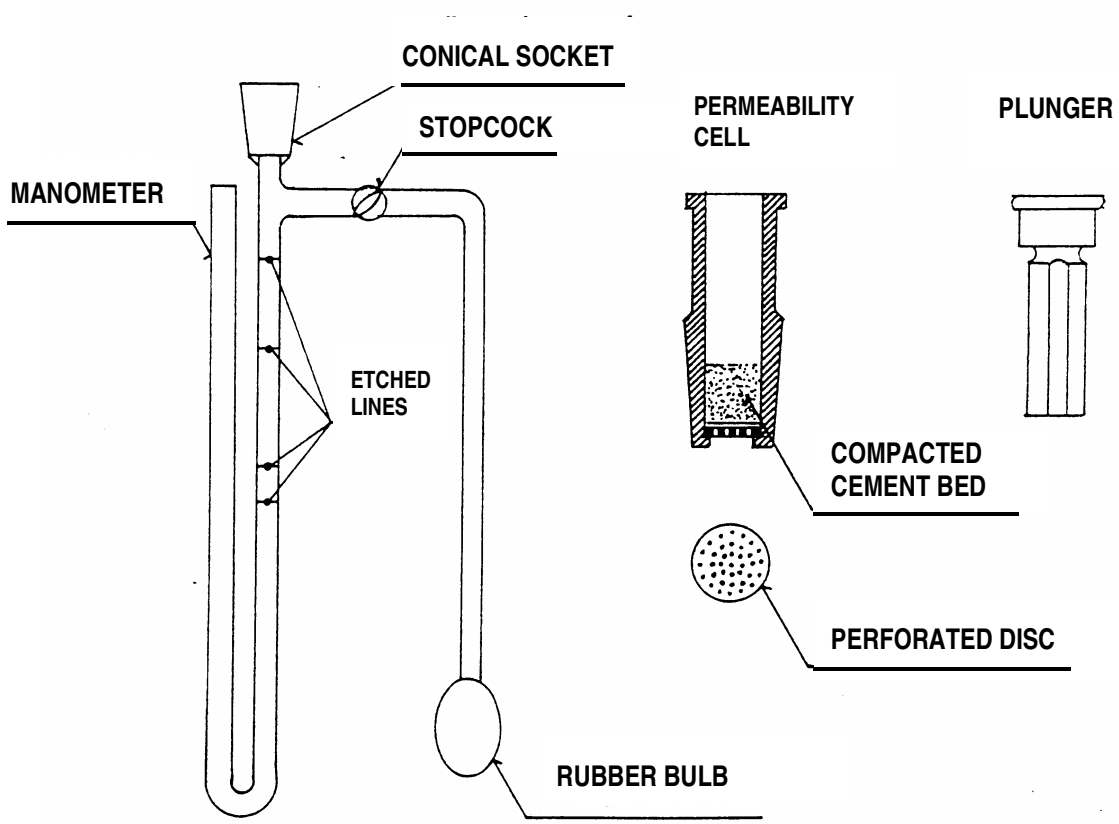
where S_0 is the specific surface of the reference cement [$cm^2.g^{-1}$]
ρ_0 density of the reference cement [$g.cm^{-3}$]
t_0 measured time [s]
η_0 air viscosity at the test temperature [Pa.s]

With the specified porosity of e = 0,500

$$K = 1{,}414 \times S_0 \times \rho_0 \times \frac{\sqrt{0{,}1\eta_0}}{\sqrt{t_0}}$$

The mean of three values of K is the constant K for the apparatus.

Fig.:30 Blaine apparatus



Testing procedure

- Place the perforated disc on the ledge at the bottom of the cell and place on it a new filter paper disc. Place the weighed quantity of cement, m_1, in the cell. Place a second new filter paper disc on the levelled cement. Insert the plunger and press it gently but firmly until the lower face of the cap is in contact with the cell. Slowly withdraw the plunger, rotate it through 90 ° and press once again. The bed is now compacted and ready for the permeability test.
- Test is performed on the Blaine apparatus (see Fig.:30). It is practically manometer in the U-tube form. One arm of the manometer is provided at the top with conical socket to form an airtight fit with the conical surface of the cell. The same arm has four etched lines M_1 to M_4 and T-joint, which lead to an airtight stopcock beyond which is attached aspiration rubber bulb.
- Manometer is filled to the level of the lowest etched line with non-volatile, non-hygroscopic liquid of low viscosity and density (such as dibutylphthalate or light mineral oil).
- Insert the conical surface of the cell into the socket at the top of the manometer.
- Open the stopcock and with gentle aspiration raise the level of the manometer liquid to that of the highest etched line. Close the stopcock and the manometer liquid will begins to flow. Start the timer as the liquid reaches the second etched line and stop it when the liquid reaches the third etched line. Record the time, t, and the temperature, T.
- The procedure repeats three times
- Calculate three values of the specific surface and the mean of them.

Tab.:27 Viscosity of air and $\sqrt{0,1\eta}$

Temperature [°C]	**Air viscosity** η [Pa.s]	$\sqrt{0,1\eta}$
16	0.00001800	0.001342
17	0.00001805	0.001344
18	0.00001810	0.001345
19	0.00001815	0.001347
20	0.00001819	0.001349
21	0.00001824	0.001351
22	0.00001829	0.001353
23	0.00001834	0.001354
24	0.00001839	0.001356
linear interpolation can be used for the determination of the intermediate values		

12.3 Determination of Setting Time

The setting time is determined by observing the penetration of needle into cement paste of standard consistence until it reaches a specified value.

The laboratory shall be maintained at a temperature of 20 ± 2 ℃ and a relative humidity of not less than 65 %.

12.3.1 Standard Consistence Test

Cement paste of standard consistence has a specified resistance to penetration by a standard plunger. The water required for such a paste is determined by trial penetrations of pastes with different water contents. Content of water is expressed as percentage by mass of the cement.

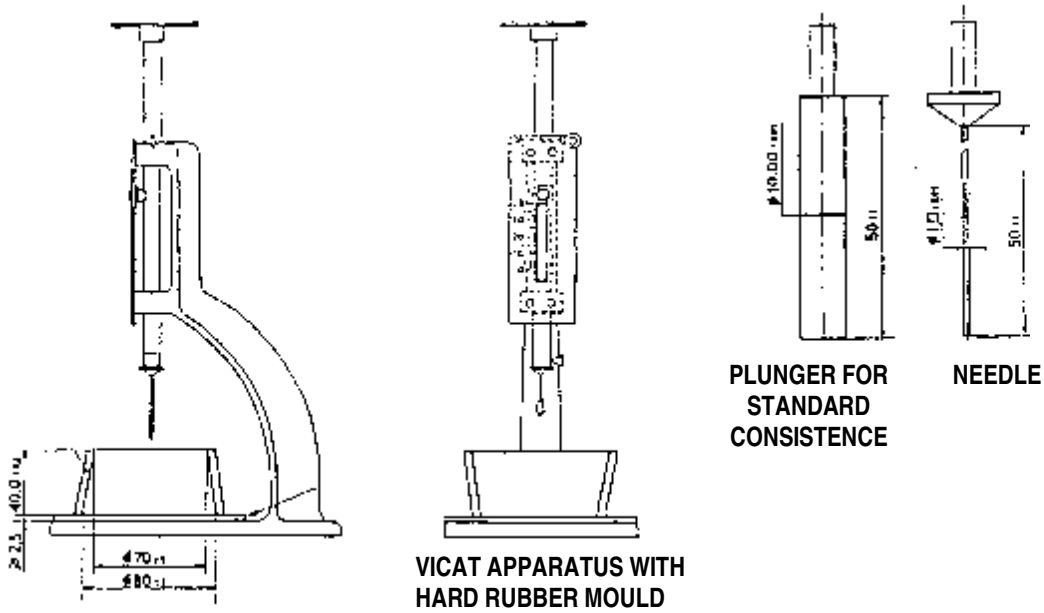
Vicat apparatus (see Fig.:31) with the plunger is used for the test. The plunger shall be of non-corrodible metal in the form of a right cylinder of 50 ± 1mm effective length and of 10,00 ± 0,05 mm diameter. The total mass of moving parts shall be 300 ± 1 g. Part of the Vicat apparatus is the mould from hard rubber (of truncated conical form) on the glass base-plate.

Procedure

- calibrate the Vicat apparatus by lowering the plunger to rest on the base-plate to be used and adjusting the pointer to read zero on the scale. Raise the plunger to the stand-by position.
- weight 500 g of cement (to the nearest 1g). Weight a quantity of water (distilled or deionized), e.g. 125 g, in the mixer bowl or measure the water from the graduated cylinder into the mixer bowl.
- ad the cement carefully to the water in order to avoid loss of water or cement. The time of addition shall be not less than 5 s nor more than 10 s. Note the time of completion of the addition as zero time, from which later measurements of time shall be made.
- start the mixer immediately and run at low speed. stop the machine after 90 s for 15 s during which remove with a suitable scraper any paste adhering to the bowl outside the mixing zone and return it to the mix. Restart the machine and run at low speed for further 90 s. The total mixer running time shall be 3 min.

- transfer the paste immediately to the mould and fill it to excess. Remove the excess gently and make a smooth upper surface
- immediately after levelling the paste, transfer the mould and base-plate to the Vicat apparatus and position it centrally under the plunger. Lower the plunger gently until it is in contact with the paste. Pause in that position for between 1s and 2s. Than release the moving part quickly and allow the plunger to penetrate vertically into the centre of the paste. The release of the plunger shall occur 4 min after zero time. Read the scale when penetration has ceased or 30 s after the release of the plunger, whichever is the earlier.
- record the scale reading, which indicates the distance between the bottom face of the plunger and the base plate. Clean the plunger immediately after each penetration.
- repeat the test with pastes containing different water contents until one is found to produce a distance between plunger and base-plate of 6 ± 1 mm. Record the water content of that paste to the nearest 0,5% as **the water for standard consistence**.

Fig.:31 Vicat apparatus with plunger and needle



12.3.2 Setting Time Test

Cement paste of standard consistence is used for this test.

Vicat apparatus is used, but the plunger is replaced by the steel needle in the form of a right cylinder of effective length 50 ± 1 mm and diameter 1,13 ± 0,05 mm. The total mass of moving part is 300±1 g.

Procedure

- calibration of Vicat apparatus with the needle is the same as for the apparatus with the plunger.
- mould, filled by paste of standard consistence and base plate transfer to the Vicat apparatus.
- procedure of penetration is the same as by using plunger.

- repeat the penetration test on the same specimen at conveniently spaced position, not less than 10 mm from the rim of the mould or from each other, at conveniently spaced intervals of time, e.g. at 10 min intervals.
- clean the Vicat needle after each penetration
- **initial setting time** is time measured from zero at which distance between the needle and the base-plate is 4 ± 1 mm . Record it to the nearest 5 min.
- **final setting time** is time measured from zero at which the needle first penetrates only 0,5 mm into the specimen

Automatic setting time machines are commercially available and may be used provided that they can be shown to give the same test results as the specified apparatus and procedure.

🕮 Vocabulary

cement paste	*cementová kaše*
coarse	*hrubý*
density	*hustota*
final setting time	*konec tuhnutí*
fine	*jemný*
fineness	*jemnost mletí*
initial setting time	*počátek tuhnutí*
mesh	*síto*
mould	*forma*
porosity	*porozita, mezerovitost*
setting time	*dobya tuhnutí*
sieving	*prosévání*
specific surface	*měrný povrch*
standard consistence	*normální hustota*
test specimen	*zkušební vzorek, zkušební těleso*
to harden – hardening time	*tvrdnout – doba tvrdnutí*
to set	*tuhnout – doba tuhnutí*