BUILDING MATERIALS:
RETENTION CURVE AND PORE SIZE DISTRIBUTION

Key words: Building materials, moisture, retention curve, pore size distribution, pressure plate apparatus, pressure membrane apparatus, sand box apparatus, test method

1 SCOPE
This test method covers the determination of the retention curve and the pore size distribution of porous building materials and soils using a combination of pressure plate, pressure membrane and/or sand box apparatuses.

2 FIELD OF APPLICATION
The test method is applicable to porous building materials and soils.

3 REFERENCES

4 DEFINITIONS
Contact angle
The contact angle is the angle between the surface of a solid and the surface of a droplet on the solid. The angle is measured inside the droplet between its tangent at the liquid-solid-air interface and the surface of the solid. Generally, the contact angle is determined by the surface tension between the solid and the air, the surface tension between the liquid and the air and the surface tension between the solid and the liquid.

Degree of vacuum saturation
The degree of vacuum saturation $S_{\text{vac}}$ is the ratio between the actual mass of evaporable water and the mass of evaporable water at vacuum saturation.

Density
The density $\rho_0$ (kg/m$^3$) is the ratio between the mass of ovendry material and the volume of the ovendry material.

Moisture content
The moisture content mass by mass $u$ (kg/kg) is the ratio between the mass of evaporable water and the ovendry mass of material.

Moisture content volume by volume $\psi$ (m$^3$/m$^3$) is the ratio between the volume of evaporable water and the volume of ovendry material.

The moisture content mass by volume $w$ (kg/m$^3$) is the ratio between the mass of evaporable water and the volume of ovendry material.

Open porosity
The open porosity $n$ is the ratio between the volume of pores that can be filled with water and the volume of the material.

Relative humidity
Relative humidity $\varphi$ or (RH) of air is the ratio between the actual partial water vapour pressure and the saturation water vapour pressure at the temperature of the air (equal to the ratio between the actual vapour concentration and the saturation vapour concentration of the air), i.e.

$$\varphi = \frac{p}{p_{\text{sat}}} = \frac{v}{v_{\text{sat}}}$$

Solid density
The solid density $\rho_s$ (kg/m$^3$) is the ratio between the mass of ovendry material and the volume of the solid part of the material.

Suction
The suction $s$ (Pa) is the difference between the pore water pressure and the ambient total pressure.
5  SAMPLING
The size of the sample is such that is possible to prepare from it at least 10 test specimens with a minimum contact area of 15 cm² and a height of 15 mm.
The way in which the samples have been taken shall be stated in the test report.

6  METHOD OF TEST
6.1 Principle
Vacuum saturated test specimens are brought in moisture equilibrium at a number of different air pressures which control the suction. The mass of the specimens is determined when in equilibrium at each applied pressure. Finally, the test specimens are dried, and the moisture content is calculated.
The observed moisture content at equilibrium versus the respective suction pressures constitutes the retention curve of the material.

6.2 Apparatus
- Ventilated oven capable of maintaining a desired temperature in the range 30-105 °C with an accuracy of ± 2°C.
- Balance with an accuracy of at least 0.01 g.
- Desiccator.
- Vacuum pump capable of evacuating a desiccator to 100 Pa pressure.
- Container.
- Sponge.
- Thermometer with 0.5 K precision.
- Porous plate apparatus and/or pressure membrane apparatus and/or sand box apparatus.
- Outflow burette with 0.05 cm³ resolution.
- Fine meshed cloth.
- Kaolin powder.

6.3 Preparation of test specimens
A minimum of 10 test specimens, each with a minimum contact area of 15 cm² and a height in the range 5-15 mm, are prepared from the sample. The minimum height depends on the structure of the material. For homogeneous materials a small height should be chosen because the equilibration time is proportional to the square of the height.

6.4 Procedure
6.4.1 The test should be carried out at 23 °C ± 1 °C.
6.4.2 The specimens are dried until repeated weighings, at intervals of at least six hours, show a difference in mass of 0.1 per cent or less. The mass of the test specimens in the dry state is denoted \( m_0 \).

For more accurate determinations of the moisture content the test specimens are dried in flasks. The determinations of the mass of the test specimens and the flasks are carried out with a precision of 0.005 g.
6.4.3 The dried test specimens are placed in a desiccator which is evacuated to 100 Pa for 3.0 hours.
6.4.4 Demineralised water is admitted into the desiccator until the test specimens are fully covered with water. The desiccator is reevacuated to 100 Pa and left for equilibration for 1 hour.
6.4.5 The masses of the saturated specimens under water \( m_{sw} \) are determined by weighing. Excess moisture is removed by gently wiping the specimens with a moist sponge. The masses of the specimens in saturated surface dry condition \( m_{ssd} \) are determined by weighing.

NOTE: Annex B gives an example of a form which can used for the registration of the measurements made in Clauses 6.4.2 and 6.4.5. The form is readily transformed into a computer worksheet.

6.4.6 The test specimens are equilibrated at the lowest suction pressures given in Table 1, which it is possible to use according to the resolution of the available equipment.

6.5 The values specified in Table 1 are used as set-points. It is important, if during a run the pressure rises above the set-point, never to lower the pressure again because this changes the measurement from desorption to adsorption.

Table 1. Suction pressure set-points and corresponding relative humidities.

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<tr>
<th>( S ) Pa</th>
<th>( S ) bar</th>
<th>( \phi^* )</th>
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<tbody>
<tr>
<td>10²</td>
<td>0.001 00</td>
<td>0.999 999 27</td>
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<tr>
<td>10².5</td>
<td>0.003 16</td>
<td>0.999 997 7</td>
</tr>
<tr>
<td>10³</td>
<td>0.010 0</td>
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<td>10³.5</td>
<td>0.031 6</td>
<td>0.999 977</td>
</tr>
<tr>
<td>10⁴</td>
<td>0.100 0</td>
<td>0.999 927</td>
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<td>10⁴.5</td>
<td>0.316 0</td>
<td>0.999 77</td>
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<tr>
<td>10⁵</td>
<td>1.00 0</td>
<td>0.999 27</td>
</tr>
<tr>
<td>10⁵.5</td>
<td>3.16 0</td>
<td>0.997 7</td>
</tr>
<tr>
<td>10⁶</td>
<td>10.0 0</td>
<td>0.992 7</td>
</tr>
<tr>
<td>10⁶.5</td>
<td>31.6 0</td>
<td>0.977</td>
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</table>

The relative humidity is calculated from the suction using the Kelvin equation with properties for water at 25 °C.
Equilibrium is said to have been achieved when the moisture outflow is less than 0.05 cm³ in 48 hours. The actual suction $s$ at equilibrium is registered. The specimens are removed and their masses $m_w$ are determined by weighing.

6.4.7 Step 6.4.6 is repeated until all suction pressures in Table 1 are covered. Note that a new layer of fresh kaolin paste must be applied after each weighing.

Where a combination of several different pressure plates/pressure membranes/sand boxes covering different suction pressure ranges is used there shall be a connection of measurements, i.e. the specimens shall be equilibrated in the higher range equipment at a pressure a little above the pressure at which the measurements ended in the lower range equipment.

EXAMPLE: A measurement with a 15 bar pressure plate apparatus is made at 10 bar. The next set-point in Table 1 is 31.6 bar which is out of range for the 15 bar pressure plate. Therefore, the measurement is continued in a 100 bar pressure membrane extractor with 10.1 bar as the first set-point.

NOTE: Annex C gives an example of a form which can be used for the registration of the measurements made in Clauses 6.4.6 and 6.4.7. One form is used at each suction. The form is readily transformed into a computer worksheet.

6.4.8 The specimens are dried using the procedure described in Section 6.4.2. The mass of the test specimens in the dry state is denoted $m_0$.

6.5 Expression of results

6.5.1 The apparent dry densities $\rho_0$ of the test specimens are calculated as

$$\rho_0 = \frac{m_0}{m_{ssd} - m_{sw}} \rho_w$$

The sample mean apparent density $\bar{\rho}_0$ and the sample standard deviation $s_{\rho_0}$ are calculated.

6.5.2 The solid densities $\rho_s$ of the test specimens are calculated as

$$\rho_s = \frac{m_0}{m_{ssd} - m_{sw}} \rho_w$$

The sample mean solid density $\bar{\rho}_s$ and the sample standard deviation $s_{\rho_s}$ are calculated.

6.5.3 The open porosities $n$ of the test specimens are calculated as

$$n = \frac{m_{ssd} - m_0}{m_{ssd} - m_{sw}}$$

The sample mean open porosity $\bar{n}$ and the sample standard deviation $s_n$ are calculated.

6.5.4 The vacuum saturation moisture contents mass by mass of the specimens are calculated as

$$u_{vac} = \frac{m_{ssd} - m_0}{m_0}$$

The sample mean vacuum saturation moisture content $\bar{u}_{vac}$ and the sample standard deviation $s_{u_{vac}}$ are calculated.

6.5.5 The vacuum saturation moisture contents mass by volume of the specimens are calculated as

$$w_{vac} = \frac{m_{ssd} - m_0}{m_{ssd} - m_{sw}} \rho_w$$

The sample mean vacuum saturation moisture content $\bar{w}_{vac}$ and the sample standard deviation $s_{w_{vac}}$ are calculated.

NOTE: The example form in Annex B, or a corresponding computer worksheet, can be used when performing the calculations in Clauses 6.5.1-6.1.5.

6.5.6 The capillary radii $r$ are calculated as

$$r = \frac{2 \sigma}{s} \cos \theta$$

where $\sigma$ is the surface tension of water, $\theta$ is the contact angle and $s$ is the actual measured suction. If no specific information is available a value of 0° for $\theta$ and a value of 0.072 N/m (pure water at 25 °C) for $\sigma$ can be assumed. The values chosen for $\theta$ and $\sigma$ must be stated in the report.

6.5.7 The degree of vacuum saturation of the test specimens is calculated as

$$S_{vac} = \frac{m_{ssd} - m_0}{m_{ssd} - m_{sw}}$$

The sample mean degree of vacuum saturation $\bar{S}$ and the sample standard deviation $s_s$ are calculated at each measured suction pressure.

6.5.8 The moisture contents volume by volume of the specimens are calculated as

$$\psi = \frac{m_0 - m_0}{m_{ssd} - m_{sw}}$$

The sample mean moisture content $\bar{\psi}$ and the sample standard deviation $s_{\psi}$ are calculated at each measured suction pressure.

6.5.9 If required, the moisture contents mass by mass of the specimens are calculated as

$$u = \frac{m_{sw} - m_0}{m_0}$$

where $m_w$ is the mass of the specimen in the moist state and $m_0$ is the mass of the specimen in the dry state. The sample mean moisture content $\bar{u}$ and the sample standard deviation $s_u$ are calculated at each measured suction pressure.
If required, the moisture contents mass by volume of the specimens are calculated as

\[
w = \frac{m_w - m_0}{m_{ssd} - m_{sw}} \rho_w
\]

The sample mean moisture content \( W \) and the sample standard deviation \( \sigma_w \) are calculated at each suction pressure.

NOTE: The example form in Annex C, or a corresponding computer worksheet, can be used when performing the calculations in Clauses 6.5.6-6.5.10.

The results from Clauses 6.5.1-6.5.10 as well the measured temperatures, measured suctions and applied thermodynamic properties of water are transferred to a form similar to the one presented in Annex D.

A plot of the sample mean degree of vacuum saturation versus actual suction pressure is made in a log scale (an example is shown in Fig. A1 in Annex A).

A plot of the sample mean porosity (i.e. moisture content volume by volume) versus capillary radius is made in a log scale (an example is shown in Fig. A2 in Annex A).

6.6 Accuracy

NOTE: In an interlaboratory comparison made on sandstone the following accuracy has been estimated:

**Repeatability standard deviations:**
- Apparent density: 14 kg/m³
- Solid density: 2 kg/m³
- Open porosity: 0.005 m³/m³
- Retention curve moisture content: 0.004 kg/kg

**Reproducibility standard deviations:**
- Apparent density: 24 kg/m³
- Solid density: 4 kg/m³
- Open porosity: 0.009 m³/m³
- Retention curve moisture content: 0.01 kg/kg

6.7 Test report

The test report shall include the following information, if relevant:

a) Name and address of the testing laboratory
b) Identification number of the test report
c) Name and address of the organisation or the person who ordered the test
d) Purpose of the test
e) Choice of points of measurements and other circumstances (date and person responsible for sampling)
f) Method of sampling and specimen preparation.
g) Address and a detailed description of the place where the samples have been taken out.
i) Date when the measurement was ordered
j) Date of the measurement
k) Test method
l) Conditioning of the sample type and probe, ambient conditions during the measurement (temperature, RH etc.)
m) Identification of the equipment used for measurement (product, model and laboratory equipment identification number)
n) Any deviations from the method of measurement
o) Test results according to Clauses 6.5.11, 6.5.12 and 6.5.13
p) Inaccuracy or uncertainty of the measurement
q) Date and signature.
Figure A1. Example of retention curve for sandstone.

Figure A2. Example of pore size distribution for sandstone.
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\[
\begin{align*}
m_0 & \text{ g} \\
m_{\text{wet}} & \text{ g} \\
m_{\text{damp}} & \text{ g} \\
\rho & \text{ kg/m}^3 \\
\gamma_{s} & \text{ kg/m}^3 \\
\eta & \text{ m}^2/\text{m}^3 \\
u_{\text{wet}} & \text{ kg/h} \\
u_{\text{dry}} & \text{ kg/h}
\end{align*}
\]

Balance laboratory ID No. Oven laboratory ID No.: Temperature measuring device laboratory ID No.
<table>
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<th>Date:</th>
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<tr>
<td>Nominal suction $s$ bar</td>
<td>Measured suction $s$ bar</td>
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<tr>
<td>Contact angle $\theta$ $^\circ$</td>
<td>Surface tension $\sigma$ N/m</td>
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<td>Capillary radius $r$ $10^{-6}$ m</td>
<td>Air temperature $^\circ$C</td>
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<td>$\bar{x}$</td>
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<td>$w$ m$^3$/m$^3$</td>
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<td>$w$ kg/m$^3$</td>
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<td>Suction apparatus laboratory ID No.:</td>
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</table>
# ANNEX D

**NT BUILD 481 BUILDING MATERIALS:**
RETENTION CURVE AND PORE SIZE DISTRIBUTION

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<tr>
<th>Measured suction $s$, bar $10^{-6}$m</th>
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<th>$s_{\text{vac}}$ m$^3$/m$^3$</th>
<th>$\overline{\psi}$ m$^3$/m$^3$</th>
<th>$s_{\text{wp}}$ m$^3$/m$^3$</th>
<th>$\bar{u}^*$ kg/kg</th>
<th>$s_{u^*}$ kg/kg</th>
<th>$\bar{w}^*$ kg/m$^3$</th>
<th>$s_{w^*}$ kg/m$^3$</th>
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<table>
<thead>
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<th>$\bar{\rho}_0$ kg/m$^3$</th>
<th>$s_{p,0}$ kg/m$^3$</th>
<th>$\bar{s}_{r,s}$ kg/m$^3$</th>
<th>$n=\psi_{\text{vac}}$ m$^3$/m$^3$</th>
<th>$s_{n}$ m$^3$/m$^3$</th>
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<th>$s_{w_{\text{vac}}}$ kg/m$^3$</th>
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<td>Water temperature °C</td>
<td>Density of water $\rho_w$ kg/m$^3$</td>
<td>Contact angle $\theta$ °</td>
<td>Surface tension $\sigma$ N/m</td>
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*: Optional