Appendix C: Porosity - Guidelines used for testing
GUIDELINES FOR TESTING (1989)
SIA 162/1 Test No. 7: Porosity

1. INTRODUCTION

The test described here serves to determine the amount of pores that can be filled by capillary absorption of water (hydration pores), the total porosity and the dry bulk density.

These values provide the basis for the determination of the frost resistance FS.

From the amount of fillable pores and the cement content, the water content in the fresh concrete or the water cement value may also be deduced.
2. APPARATUS AND TOLERANCES

2.1 Balance
The maximum loading should be \( \geq 1'000 \ldots 1'500 \) \( g \), and the weight should be able to be read to \( \pm 0.01 \) \( g \) or less. The balance must be equipped for weighing under water.

2.2 Vacuum pump
The pumping capacity must be suited to the size of the recipient and the amount of samples to be treated at the same time. A vacuum of approx. \( 10^{-3} \) mbar should be able to be attained.

2.3 Recipient
The samples of the same series should be located at the same height and must be exposed to the water on all sides.

A device must be provided for flooding under vacuum. The recipient must be equipped with a manometer for measuring the internal pressure.

2.4 Drying oven
The drying oven must fulfil the following requirements:
- temperature range \( 40 \ldots 110 \) °C
- precision \( \pm 1 \) °C

2.5 Exsiccator
After drying at \( 105\) °C, the samples are cooled off in the exsiccator in air of relative humidity \( 0\% \) (exsiccant: freshly regenerated silica gel), in order to avoid weight changes due to water absorption.
3. SAMPLING METHOD, SAMPLES

Samples are prepared from cores or blocks that are extracted either from separately produced samples or from the building construction itself.

Chiselled samples or fragments are also permissible if there is no danger of stones or pieces of mortar breaking out of the surface upon handling.

Sections of cores with $d = 50$ mm and $H = 50$ mm are normally employed. With maximum grain diameters $\leq 32$ mm, at least 5 core sections are required for a test series.
4. EXECUTION

The test is conducted according to the following scheme. Parameters in brackets may be determined optionally.

<table>
<thead>
<tr>
<th>Time [days]</th>
<th>Treatment, storage</th>
<th>Measurement</th>
<th>Parameters measured</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Drying at 50°C</td>
<td>Weigh</td>
<td>M₁</td>
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<td>2</td>
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<td>3</td>
<td>Cooling to room temp</td>
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<tr>
<td>4</td>
<td>Samples had immersed in water at 20°C; approx. 85% RH</td>
<td>Weigh</td>
<td>M₂₀</td>
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<td>5</td>
<td>Storage in water (approx. 20°C)</td>
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<tr>
<td>10</td>
<td>Drying for at least 23h at 50°C. Then evacuate for 2h. Flood under vacuum</td>
<td>Weigh above and under water</td>
<td>M₂₀, V₂₀</td>
</tr>
<tr>
<td>11</td>
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<tr>
<td>12</td>
<td>Storage in water (in evaporation recipient) → do not expose to air to change position.</td>
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<td>13</td>
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<tr>
<td>14</td>
<td>Drying at 105°C</td>
<td>Weigh above and under water</td>
<td>M₅₀, V₅₀</td>
</tr>
<tr>
<td>15</td>
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<tr>
<td>16</td>
<td></td>
<td>Weigh</td>
<td>M₁₀5</td>
</tr>
<tr>
<td>17</td>
<td>Cooling in associator</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Definition of the parameters to be measured according to the test scheme:

- \( M \) = mass of sample
- \( V \) = volume of sample
  - \( V = \frac{M_{\text{above water}} - M_{\text{under water}}}{\rho_w} \)

where \( \rho_w = \text{density of water} = 1'000 \text{ kg/m}^3 \)

Indices:

- A = as delivered
- 50 = after two days drying at 50°C = beginning of absorption test
- E = after 7 days storage in water = end of absorption test
- TOT = after vacuum saturation
- 105 = after three days drying at 105°C
5. EVALUATION

Calculations:

Dry bulk density

\[ \rho_{R\text{105}} = \frac{M_{\text{105}}}{V_E} \]  
[\text{kg/m}^3]

Total porosity

\[ n = \frac{M_{\text{TOT}} - M_{\text{105}}}{\rho_w \cdot V_E} \cdot 100 \]  
[\text{vol-\%}]

Density

\[ \rho_{105} = \rho_{R\text{105}} \cdot \frac{100}{(100 - n)} \]  
[\text{kg/m}^3]

Water content after drying for 2 days at 50°C

\[ U_{50} = \frac{M_{50} - M_{\text{105}}}{\rho_w \cdot V_E} \cdot 100 \]  
[\text{vol-\%}]

Content of fillable pores

- hydration pores

\[ U_E = \frac{M_E - M_{\text{105}}}{\rho_w \cdot V_E} \cdot 100 \]  
[\text{vol-\%}]

Content of non-fillable pores = air and compaction pores

\[ n - U_E \]  
[\text{vol-\%}]

The test record contains the following details:

- designation of series of samples
- type and number of test samples
- time and age at beginning of testing
- single values, means of series (arithmetic mean) and standard deviation for

- Water content after 2 days drying at 50°C
- Content of fillable pores
- Total porosity
- Content of non-fillable pores
- Dry bulk density
- Density
Remarks

The determination of the total porosity by vacuum saturation is a delicate matter. Therefore, before the calculated values for $n$ or the measured values for $\mathcal{M}_{\text{TOT}}$ are further used, it must be checked by means of the following three criteria whether all the pores have been filled in the test:

1. The straight line regression $\varrho_R = a - b \cdot n$ for the test sample series does not differ significantly from the theoretical relation

$$\varrho_{R105} = \varrho_{105} - \frac{\varrho_{105}}{100} \cdot n$$

(e.g.: $t$-test; null hypothesis: $b = \frac{\varrho_{105}}{100}$)

2. $\varrho_{105}$ agrees roughly with the value that can be calculated from the components of the concrete.

(As a guide: $\varrho_{105} = 2'700 \text{ kg/m}^3$)

3. The standard deviation of the individual value of $\varrho_{105}$ is smaller than that of $\varrho_{R105}$.

If not all the criteria are fulfilled, then the individual values must be examined more closely and if necessary the density $\varrho_m$ determined in a pyknometer on a ground, average sample. The correct individual values $n^*$ for the total porosity are then obtained from the relation:

$$n^* = \left( 1 - \frac{\varrho_{R105}}{\varrho_m} \right) \cdot 100 \text{ [vol-%]}$$
Frost resistance $FS$

$$FS = \frac{n - U_E}{\bar{n} - U_{*kr}}$$

where $n - U_E$ = mean content of non-fillable pores

$\bar{n}$ = mean total porosity

$U_{*kr} = 0.94 \cdot \bar{n} - 0.37 \cdot (n - U_E) + 0.3 \text{ vol.-%}$

= critical water content calculated from empirical relation

As a guide

high frost resistance $FS \geq 1.5$

low frost resistance $FS \leq 1.0$

Water content in fresh concrete

The water content of fresh concrete is calculated by

$$W_0 = \frac{U_E}{100} \cdot \varrho_W + (0.17 \ldots 0.25) \cdot \alpha \cdot Z$$

where $U_E$ = mean content of fillable pores

$\varrho_W$ = density of water = 1'000 kg/m$^3$

$\alpha$ = degree of hydration ($\approx 0.6$ at test age $= 28 \text{ d}$)

($\approx 0.8$ at test age $= 1 \text{ a}$)

$0.17 \ldots 0.25$ = factor taking into account

- water bound by hydration = 25 M-% of $Z$
- max. water absorption dependent on $U_{50}$

$Z$ = cement content (or cement dosage)
1. INTRODUCTION

The test described here serves to determine the critical degree of saturation.

From the critical degree of saturation $S_{kr}$ and the maximum attainable degree of capillary saturation $S_{E(kap)}$ (cf. Test No. 5), the frost resistance $\Delta S = S_{kr} - S_{E(kap)}$ is obtained.

Above all when employing alternative aggregate materials or other types of cement and additives, it is recommended, to carry out preliminary trials to directly determine the critical degree of saturation and to compare it with the value obtained from experience (see Test No. 7, Porosity).
2. APPARATUS AND TOLERANCES

2.1 Balance

The maximum loading should be 1'000...1'500 g, and the weight should be able to be read to ± 0.01 g or less. The balance must be equipped for weighing under water.

2.2 Vacuum pump

The pumping capacity must be suited to the size of the recipient and the amount of samples to be treated at the same time. A vacuum of approx. $10^{-3}$ mbar should be able to be attained.

2.3 Recipient

The samples of the same series should be located at the same height and must be exposed to the water on all sides.

A device must be provided for flooding under vacuum. The recipient must be equipped with a manometer for measuring the internal pressure.

2.4 Drying oven

The drying oven must fulfil the following requirements:
- temperature range: 40...110 °C
- precision: ± 1 °C

2.5 Exsiccator

After drying at 105 °C, the samples are cooled off in the exsiccator in air of relative humidity 0% (exsiccant: freshly regenerated silica gel), in order to avoid weight changes due to water absorption.
2.6 Deep freezer

The deep freezer must guarantee the following automatic frost cycle:

\[+20^\circ C\]
\[\text{Thawing in water}\]
\[0^\circ C\]
\[\text{Freezing in air}\]
\[-25^\circ C\]

The temperature precision must be at least \(\pm 2^\circ C\).

3. SAMPLING METHOD, SAMPLES

Samples are prepared from cores or blocks that are extracted either from separately produced samples or from the building construction itself.

The volume of the samples must be approx. 100 cm\(^3\).

Sections of cores with \(d = 50\) mm and \(H = 50\) mm are normally employed. With maximum grain diameters \(\leq 32\) mm, at least 12 core sections are required for a determination of \(S_{kr}\).

Remark:

The samples for the determination of \(S_{E}(kap)\) and \(S_{kr}\) must be of the same size and be taken from the same random sample! Hence for a determination of the frost resistance \(\Delta S\), at least 17 sections of cores with \(d = 50\) mm and \(H = 50\) mm are necessary.
4. EXECUTION

The test is conducted according to the following scheme. Parameters in brackets may be determined optionally.

<table>
<thead>
<tr>
<th>Time [days]</th>
<th>Treatment, storage</th>
<th>Measurement</th>
<th>Parameters measured</th>
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<tr>
<td>1</td>
<td>Drying at 50°C</td>
<td>Weigh</td>
<td>( M_A )</td>
</tr>
<tr>
<td>2</td>
<td>Cool to room temp.</td>
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<tr>
<td>3</td>
<td>Sample half immersed in water at approx. 20°C and approx. 85% RH</td>
<td>Weigh</td>
<td>( M_50 )</td>
</tr>
<tr>
<td>4</td>
<td>Storage in water (approx. 20°C)</td>
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</tbody>
</table>
| 11          | 2 samples stored in water (approx. 20°C) | weigh above and under water | \( M_G \) \( \cdot \) \( \gamma \) \( \cdot \) \( \beta \)
|             | Remaining samples stored in air (room climate) |             | \( V_\text{volat} \) |
| 12          | Remaining samples evacuated and stored at 105°C for various lengths of time | Weigh | \( M_{91} \) |
| 13          | As samples packed upright |             |                    |
| 14          | 6 more cycles |             |                    |
| 15          | All samples unpacked | Weigh | \( M_{91} \) |
| 16          | At least 24 h storage in water (approx. 20°C) | weigh above and under water | \( V_\text{water} \) |
| 17          | Drying for at least 22 h at 50°C. Then evacuation for 24 h. Flood under vacuum. | Weigh | \( V_\text{dry} \) |
| 18          | Storage in water (in recipient) |             |                    |
| 19          |                     |             |                    |
| 20          | Drying at 105°C | weigh above and under water | \( M_{\text{TOT}} \) \( \cdot \) \( \gamma_{\text{TOT}} \) |
| 21          |                     |             |                    |
| 22          | Cooling in cooler | Weigh | \( M_{105} \) |

Definition of the parameters to be measured according to the test scheme:

\[ M = \text{mass of sample} \]
\[ V = \text{volume of sample} \]
\[ V = \frac{M_{\text{above water}} - M_{\text{under water}}}{\rho_W} \]

where \( \rho_W = \text{density of water} = 1'000 \text{ kg/m}^3 \)
Indices:

A = as delivered
50 = after two days drying at 50 °C
E = after 7 days storage under water
voll = after vacuum saturation before frost cycling
01 = before the first frost cycles
61 = after the first 6 frost cycles
TOT = after vacuum saturation
105 = after three days drying at 105 °C

5. EVALUATION

Calculations:

Dry bulk density $\rho_{R105} = \frac{M_{105}}{V_E}$ [kg/m$^3$]

Total porosity $n = \left(\frac{M_{TOT} - M_{105} - V_{TOT} - V_E}{\rho_w \cdot V_E \cdot V_E}\right) \cdot 100$ [vol-%]

Density (calculated) $\rho_{105} = \rho_{R105} \cdot \frac{100 - n}{100}$ [kg/m$^3$]

Degree of saturation at the end of the absorption test $S_E = \frac{(M_E - M_{105})}{\rho_w \cdot V_E \cdot n} \cdot 100$

Degree of saturation during the (first) 6 frost cycles $S_{06} = \frac{(M_{01} + M_{61})}{2} - M_{105} \cdot \frac{100}{\rho_w \cdot V_E \cdot n}$

or $\delta S_{06} = S_{06} - S_E$

Volume change resulting from the (first) 6 frost cycles $\delta V_{06} = \frac{V_{61} - V_E}{V_E} \cdot 1'000$ [%]

Critical degree of saturation $S_{kr} = \bar{S}_E + \delta S_{kr}$

where $\delta S_{kr} = \delta S_{06}$, for which the volume change $\delta V_{06}$ is on average $1^\circ/\cdot$.
Remarks:

$\delta S_{kr}$ may be determined with advantage graphically:
- Plot the points $x = \delta S_06; y = \delta V_06$
- Mark the point A with the largest $\delta S_06$ and $\delta V_06 < 1^\circ/\circ\circ \Rightarrow \delta S_06 (A)$
- Mark the point B with the smallest $\delta S_06$ and $\delta V_06 > 1^\circ/\circ\circ \Rightarrow \delta S_06 (B)$
- If $\delta S_06 (A) \leq \delta S_06 (B)$:

\[ \delta S_{kr} \text{ is obtained as the intersection of the connecting straight line AB with the straight line } \delta V_06 = 1^\circ/\circ\circ \]
- If $\delta S_06 (A) > \delta S_06 (B)$:

\[ \delta S_{kr} \text{ is obtained as the median of } \delta S_06 \text{ of all points with } \delta S_06 (B) \leq \delta S_06 \leq \delta S_06 (A) \]

$\delta S_{kr}$ cannot be determined exactly in the following cases:
a) None of the samples shows a $\delta V_06 > 1^\circ/\circ\circ$
   \[ \Rightarrow \delta S_{kr} \geq \delta S_06 (A) \]
b) All the samples show a $\delta V_06 > 1^\circ/\circ\circ$
   \[ \Rightarrow \delta S_{kr} \leq \delta S_06 (B) \]
c) Between $\delta S_06 (A)$ and $\delta S_06 (B)$ there is a large gap
   \[ \Rightarrow \delta S_06 (A) \leq \delta S_{kr} \leq \delta S_06 (B) \]

If $\delta S_06 (A) < 0.05$ or $\delta S_06 (B) > 0.00$, then a further 6 frost cycles must be carried out with samples in the corresponding saturation range. Only those samples from the first test may be reused for which $\delta V_06 < 1^\circ/\circ\circ$. 
The test record contains the following details:
- designation of series of samples
- type and number of test samples
- time and age at beginning of frost cycles
- mean of series (arithmetic mean) for
  - dry bulk density $\bar{\rho}_{R_{105}}$
  - total porosity $\bar{n}$
  - degree of saturation at $S_E$
end of absorption test
- graphic plot for the determination of $\delta S_{kr}$
- critical degree of saturation $S_{kr} = S_E + \delta S_{kr}$

Remarks:
The determination of the total porosity by vacuum saturation is a delicate matter. Therefore, before the calculated values for $n$ or the measured values for $H_{TOT}$ are further used, it must be checked by means of the following three criteria whether all the pores have been included in the test:

1. The straight line regression $\rho_{R_{105}} = a - b \cdot n$ for the test sample series does not differ significantly from the theoretical relation
   
   \[ \rho_{R_{105}} = \rho_{105} - \frac{\rho_{105}}{100} \cdot n \]
   
   (t - test; null hypothesis: $b = \frac{\rho_{105}}{100}$)

2. $\rho_{105}$ agrees roughly with the value that can be calculated from the components of the concrete.
   (As a guide: $\rho_{105} = 2'700 \text{ kg/m}^3$)

3. The standard deviation of the individual value of $\rho_{105}$ is smaller than that of $\rho_{R_{105}}$.

If not all the criteria are fulfilled, then the individual values must be examined more closely and if necessary the density $\rho_m$ determined in a pyknometer on a ground, average sample. The correct individual values $n^*$ for the total porosity are then obtained from the relation:

\[ n^* = \left(1 - \frac{\rho_{R_{105}}}{\rho_m}\right) \cdot 100 \text{ [vol-%]} \]
Frost resistance $\Delta S$

The frost resistance $\Delta S$ is defined as

$$\Delta S = S_{kr} - \overline{S}_{\text{E}} (\text{kap})$$

$\overline{S}_{\text{E}} (\text{kap})$ = maximum attainable degree of saturation by capillary suction (see Guideline Test No. 5)

As a guide: high frost resistance $\Delta S \geq 0.05$
low frost resistance $\Delta S \leq 0.00$
Appendix D: Practical on-site experience

Experiments sometimes lead to misinterpretation...
Kein terroristischer Akt
an Hundwilertobelbrücke

Wissenschaftlicher Versuch zum Frost

HERISAU ■ Vielen Lands-
gemeindegängerinnen und-
gängern sind sie aufgefallen: die Drähte und die
kleinen rot-weissen Plas-
tikdinger, die am Nord-
ende der Hundwilertobel-
brücke an der Brüstung
befestigt sind. Es handelt
sich dabei um Messson-
den, mit denen das Frost-
verhalten von Beton auf-
gezeichnet wird.

Die kleinen rot-weissen Plas-
tikkörper mit der Form eines
Schokoriegels liessen in so-
manchem Anrufer die Vermu-
tung einer bevorstehenden
Sprengung oder eines mögli-
chen terroristischen Aktes
hochkommen. Wie das kanto-
nale Tiefbauamt informiert,
handelt es sich jedoch um wis-
senschaftliche Versuche im
Rahmen einer Doktorarbeit an
der Eidgenössischen Material-
prüfungs- und Forschungsan-
stalt (Empa). Die Versuche
sind vom Tiefbauamt bewilligt
worden.

Der seit letztem Herbst laufen-
de Versuch soll aufzeigen, wie
sich Frost auf Betonbauwerke
auswirkt. Das Ziel der Messun-
gen ist, Erkenntnisse zu gewin-
nen, wie die Qualität und Dau-
nerhaftigkeit von Beton verbes-
sert werden kann.

bd.