PROFICIENCY TESTING SCHEME FOR DETERMINATION OF CONCRETE PROPERTIES BY A LIMITED NUMBER OF PARTICIPANTS

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Abstract

The proficiency testing (PT) scheme is developed for assessment of results of concrete slump and compressive strength determination based on a metrological approach. The experiment designed in the scheme supposes preparation of a test portion/sample of a concrete in-house reference material (IHRM) at a reference laboratory (RL) in the same conditions for every PT participant. Therefore, IHRM instability is not relevant here as a source of measurement/test uncertainty, while intra- and between-samples inhomogeneity parameters are evaluated using the results of RL testing of the samples taken at the beginning, the middle and the end of the PT experiment. The IHRM assigned slump and compressive strength values are calculated as averaged RL results. Their uncertainties include the measurement/test uncertainty components and the components arising from the material inhomogeneity.

The test results of the PT participants (25 laboratories accredited according to ISO 17025) were compared with the IHRM assigned values taking into account both the uncertainties of the assigned values and the measurement/test uncertainties of the participants. Zeta-score was used for the individual laboratory performance assessment, and bias of the results mean from the IHRM assigned value was applied for assessment of comparability/compatibility of all the results as a group (collective assessment). Comparability/compatibility of both the slump and the compressive strength determination results was found satisfactory.

Key words

Proficiency testing, limited number of participants, concrete, slump, compressive strength, in-house reference materials
The main problem is inhomogeneity and instability of fresh concrete. To overcome this problem, every participant of the Concrete Proficiency Sample Program of CCRL at NIST, overseen by ASTM, is provided with a sample consisting of the concrete RM components (N > 100).

A participant mixes these components at its laboratory, i.e. prepares the concrete sample independently. It allows to start the test immediately after preparing and to avoid instability. However, inhomogeneity of the concrete prepared in different laboratories influences the test results.

### Methods of testing and SOP

- Israeli Standard 26, part 2 “Methods of testing concrete: Qualities of fresh concrete” → SOP of PT participants for slump determination
- Israeli Standard 26, part 4 “Methods of testing concrete: Strength of hardened concrete” → SOP of PT participants for strength determination on the 7th day and on the 28th day
- INPL SOP PT 5.9.3-1 “PT Scheme for Determination of Slump and Compressive Strength Values of Concrete”

### Design of experiment

Order of the batches:

Homogeneity study should be done for every property under testing. More details see in: L.Kimhi, C. Zlotnikov, I.Kuselman. ACQUAL (2006) 11: 577-583.

### Reference material

Fresh concrete of type B30 was produced by RL using Pan Mixer of 55L, company “Controls”, Italy. The volume prepared for every testing group (material batch) was of 35L

<table>
<thead>
<tr>
<th>No.</th>
<th>Component</th>
<th>Mass, kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Crushed aggregates till 22 mm</td>
<td>19.60</td>
</tr>
<tr>
<td>2</td>
<td>Crushed aggregates till 14 mm</td>
<td>10.85</td>
</tr>
<tr>
<td>3</td>
<td>Crushed aggregates till 7 mm</td>
<td>11.90</td>
</tr>
<tr>
<td>4</td>
<td>Sea sand</td>
<td>23.80</td>
</tr>
<tr>
<td>5</td>
<td>Cement 42.5 CEM II/ALL</td>
<td>10.85</td>
</tr>
<tr>
<td>6</td>
<td>Water</td>
<td>6.82</td>
</tr>
</tbody>
</table>

### Homogeneity study in slump units

<table>
<thead>
<tr>
<th>Lab number</th>
<th>Prepar. method</th>
<th>(X_1) mm</th>
<th>(X_2) mm</th>
<th>(u_{\text{RL}}) mm</th>
<th>Range mm</th>
<th>Limit mm</th>
<th>(X_{\text{avg}}) mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>RL hand</td>
<td>105</td>
<td>110</td>
<td>3</td>
<td>5</td>
<td>8</td>
<td>107.5</td>
<td></td>
</tr>
<tr>
<td>RL hand</td>
<td>110</td>
<td>115</td>
<td>3</td>
<td>5</td>
<td>8</td>
<td>112.5</td>
<td></td>
</tr>
<tr>
<td>RL hand</td>
<td>115</td>
<td>115</td>
<td>3</td>
<td>0</td>
<td>8</td>
<td>115</td>
<td></td>
</tr>
<tr>
<td>RL hand</td>
<td>115</td>
<td>110</td>
<td>3</td>
<td>5</td>
<td>8</td>
<td>112.5</td>
<td></td>
</tr>
</tbody>
</table>

\[ X_{\text{avg}} = \frac{(X_1 + X_2)}{2} \]

Range = \(X_{\text{max}} - X_{\text{min}}\); Limit = 2.77\(u_{\text{RL}}\) is the limit of the Range at the level of confidence 95%; \(X_{\text{avg}}\) = (\(X_1 + X_2\))/2.
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Two-factor ANOVA without results of the slump

<table>
<thead>
<tr>
<th>Summary</th>
<th>Count</th>
<th>Sum</th>
<th>Average</th>
<th>Variance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Row 1</td>
<td>2</td>
<td>215</td>
<td>107.5</td>
<td>12.5</td>
</tr>
<tr>
<td>Row 2</td>
<td>2</td>
<td>225</td>
<td>112.5</td>
<td>12.5</td>
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<tr>
<td>Row 3</td>
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<td>230</td>
<td>115</td>
<td>0</td>
</tr>
<tr>
<td>Row 4</td>
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<td>12.5</td>
</tr>
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<td>Column 1</td>
<td>4</td>
<td>445</td>
<td>111.25</td>
<td>22.91667</td>
</tr>
<tr>
<td>Column 2</td>
<td>4</td>
<td>450</td>
<td>112.5</td>
<td>8.333333</td>
</tr>
</tbody>
</table>

Source | SS    | df | MS | F |
<table>
<thead>
<tr>
<th></th>
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</thead>
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<tr>
<td>Rows</td>
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<td>3.125</td>
<td>0.272727</td>
</tr>
<tr>
<td>Error</td>
<td>34.375</td>
<td>3</td>
<td>11.4583333</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>96.875</td>
<td>7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\[
X_{avg} = \bar{X}_{avg}/4 = 119 \text{mm}
\]

Comparability of the slump

\[
|X_{ass} - X_{PT/avg}|/S_{PT} = 0.28 < 0.34 \text{ at } \gamma = u/S_{PT} = 0.27
\]
- comparability of the results is satisfactory at the level of confidence 95%.

Cramer von Mises test for goodness

For observations \(x_1, x_2, \ldots, x_n, n \geq 50\), transformed into \(z_1 \leq z_2 \leq \ldots \leq z_i \leq \ldots \leq z_n\), where \(z_j = (x_i - X_{avg})/S\)

\[
\omega_n^2 = -n -2 \sum_{j=1}^{n} \left[ (j-1)/2n \right] \ln \Phi(z_j) + \left[ 1 - (j-1)/2n \right] \ln [1 - \Phi(z_j)]
\]

\(H_0\) is rejected, when \(\omega_n^2\) is in critical range, i.e. \(\omega_n^2 > \Omega_n^2\) at the required level of confidence (\(P = 1 - \alpha\)).

Two-sigma plot in strength-7th day

Comparability of strength-7th day

\[
|X_{ass} - X_{PT/avg}|/S_{PT} = 0.78 < 1.02 \text{ at } \gamma = u/S_{PT} = 1.44
\]
- comparability of the results is satisfactory at the level of confidence 95%.
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**Two-sigma plot in strength-28th day**

![Two-sigma plot for the lab strength measurement results](image)

**Comparability of strength-28th day**

![Comparability of results](image)

**Conclusions**

1. Material produced for the PT is **homogeneous** on the level of measurement uncertainties declared by RL.

2. Slump determinations were performed by some laboratories not completely by their SOPs, or the measurement uncertainty $u_{\text{mlp}}$ declared in the SOPs are evaluated not adequately. However, all results are comparable and distributed normally.

3. Results of compressive strength determinations are **satisfactory** according to all criteria for all laboratories.

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**IUPAC**

**Current Project**

**Analytical Chemistry Division**

**Number:** 2005-019-2-500

**Title:** Selection and use of proficiency testing schemes for a limited number of participants

**Task Group**

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