



TOWARDS THE NEXT GENERATION OF STANDARDS FOR SERVICE
LIFE OF CEMENT-BASED MATERIALS AND STRUCTURES

RRT⁺

**Main phase of the Extended Round
Robin Testing programme for
TU1404**

TESTING PROTOCOLS

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1 GENERAL INSTRUCTIONS

The 'Main experimental phase' is performed as a central part of the extended Round Robin Testing (RRT⁺), during which a comprehensive and extended testing of properties of concrete at different scales and different ages, using standardised and non-standardised methods, is performed by various European laboratories.

The aims of the Main phase:

- round robin on non-standardized test methods performed in different laboratories,
- new test methods developed by the laboratory benchmarked vs standardized or "classical" methods used in other laboratories
- testing of more sustainable / ecological version of the mix

Several points need to be considered when starting tests within the Main phase:

1. Tests are performed on concrete, mortar and paste, depending on the property tested and equipment used. Regardless which scale of material is used, it is necessary **to follow mix design given in the Protocols**. Certain test methods need several mixes to perform benchmarking. Therefore, there is ordinary and modified concrete mix proposed, as well as ordinary and modified mortar mix and modified cement paste mix (ordinary cement paste is not applicable, since it would be too fluid with such a high water-to-cement ratio).
2. Concrete is prepared using **the same procedure applied in the Initial phase**, except if there are clear instructions from the GP leader to follow a different procedure. If certain method requires different procedure of conditioning, mixing and curing and sampling, this needs to be explained once the results are submitted.
3. If there are no protocols given in this document, the test will either be performed in the later stage of the Main phase or participants are free to define and apply their own protocol. Please check within each chapter of GP which tests should be performed now and which will be performed later in the RRT⁺.
4. Results and description of tests need to be sent directly to GP leaders and should be returned in the form provided by the GP leader. Please try to give as much details as possible on your experiments, to make the group analysis of data manageable.

Group priority	Name, Surname	Contact
GP1a	Ivan Gabrijel (CRO)	gabrijel@grad.hr
GP1b	Ozlem Cizer (BE)	ozlem.cizer@bwk.kuleuven.be
GP1c	Sreejith Nanukuttan (GB)	s.nanukuttan@qub.ac.uk
GP1d	Violeta B. Bosiljkov (SLO)	violeta.bokan-bosiljkov@fgg.uni-lj.si
	Bernhard Pichler (AUT)	bernhard.pichler@tuwien.ac.at
GP1e	Emmanuel Rozière (FR)	emmanuel.Roziere@ec-nantes.fr
GP1f	Aljoša Šajna (SLO)	aljosa.sajna@zag.si
	Theodore Matikas (GR)	matikas@otenet.gr

2 MATERIALS

The ordinary concrete mixture OC which is used in the Main phase is optimised ordinary concrete mix based on the one utilized in the Initial phase. In the mix the amount of superplasticizer is changed, since during the Initial phase critical stability of the mix was obtained.

Following materials need to be used:

- Cement **CEM I 52.5 N-SR3 CE PM-CP2 NF HRC Gaurain**
- Sand 0-4 mm, REC GSM LGP1 (13 % of CaO and 72 % of SiO₂)
- Gravel 4-11mm, R GSM LGP1 (rounded, containing silicate and limestone)
- Gravel 8-16 mm, R Balloy (rounded, containing silicate and limestone)
- Plasticizer SIKAPLAST Techno 80 (water content 80%)

The cement that was shipped in August 2015 should be used, *i.e.* the same cement that was used in the Initial phase. The cement shipped in 2016 was partially hydrated in all labs and must not be used in the RRT⁺.

Identical as in the Initial phase, all the mix designs presented hereafter are based on the mass of fully water saturated gravel (both 4-11 mm and 8-16 mm), dry sand and water necessary to add to the mixer.

3 PRECONDITIONING OF MATERIALS

All the constituting materials (sand, aggregate, cement, plasticizer and water) have to be kept under controlled environment (laboratory conditions - ambient temperature $T_A = 20 \pm 2^\circ\text{C}$, relative humidity $RH \geq 60\%$) at least 7 days prior to mixing. If prescribed temperature and humidity conditions cannot be achieved, the real temperature and humidity need to be noted and reported.

3.1 Preconditioning of gravel (4-11 mm and 8-16 mm)

The absorption coefficient WA_{24} is the ratio of water absorbed after 24-hour immersion in water $WA_{abs. 24h}$, to the mass of dry aggregates M_{dry} .

$$WA_{24} = \frac{W_{abs.24h}}{M_{dry}} \cdot 100$$

Table 3.1 Water absorption of aggregates of Vercors ordinary concrete mixture.

Aggregates	Absorption coefficient WA_{24}
Coarse aggregate 8/16 R Balloy	2.25
Coarse aggregate 4/11 R GSM LGP1	2.61
Sand 0/4 REC GSM LGP1	0.77

The initial water saturation S_W of aggregates added in the mix is the ratio of actual water content W_{act} (in % of mass of dry aggregates) to the absorption coefficient.

$$S_W = \frac{W_{act}}{WA_{24}}$$

In the present RRT⁺, fully water saturated gravel aggregates will be used in all the mixes ($S_W = 100\%$). It is obligatory to perform the hereafter described procedure of preconditioning of the coarse aggregates (4-11 mm and 8-16 mm).

Gravel preconditioning at least 7 days before mixing

1. The gravel content of each gravel fraction (4-11 mm and 8-16 mm) to be added in the mix is calculated (for example 22 kg). A slightly higher content M_{tank} is weighed (for example 25 kg) and put in a sealed tank. The initial water content W_0 (in % of mass of dry aggregates) is assessed, by taking at least 3 samples from the same aggregate that originated M_{tank} weighting them (M_{sample}) and then drying at 80°C until constant mass (M_{dry}). Mass can be defined as constant mass when the difference in the weight of two subsequent measurements performed during 1 hour is less than 0.1% (according to EN 1097-5 Tests for mechanical and physical properties of aggregates. Determination of the water content by drying in a ventilated oven).

$$W_0 = \frac{M_{sample} - M_{dry}}{M_{dry}} \cdot 100$$

- Water M_W (in kg) is added in the tank to reach the fully saturated state of aggregate (absorption coefficient WA_{24} of each aggregate fraction given in Table 3.1), and then the tank is sealed.

$$M_W = M_{\text{tank}} \cdot \frac{WA_{24} - W_0}{100 + W_0}$$

- Aggregates and water in tank are carefully mixed to homogenize the water content and left sealed for at least 7 days before mixing.

The described procedure allows ensuring that aggregate has sufficient time to fully absorb water. Aggregate and water, that will be absorbed, need to be kept inside the sealed tank to minimize evaporation of water during 7 days. Regardless of these sealed conditions, an additional check of absorbed water is performed prior to mixing and adjusted if necessary, using following procedure.

Weighting gravel and adjusting water content prior to mixing

- From the tank, exact amount of needed fully saturated gravel, M_{gravel} , is weighted and placed in the mixer.
- The actual water content W_{act} (in % of mass of dry aggregates) is assessed, by taking at least 3 samples from leftover gravel in the sealed tank prior to concrete mixing (M_{act}), weighting them and drying at 80°C until constant mass (M_{dry}). From an average of the 3 samples an actual water content is calculated, W_{act} . This actual water content, W_{act} , should theoretically be equal to absorption coefficient of each gravel type, WA_{24} . If this is not the case, additional water should be added to the mixer, M_{Wadded} , to ensure that a fully saturated state of the gravel is met.

$$M_{\text{Wadded}} = M_{\text{gravel}} \cdot \frac{WA_{24} - W_{\text{act}}}{100 + W_{\text{act}}}$$

3.2 Preconditioning of sand (0-4 mm)

Taking into account that preparation of fully saturated sand is not straightforward and could create differences between laboratories, and that wet sand could bring a significant amount of additional water to the mix, it was decided to prepare mix with fully dry sand.

Sand preconditioning more than 24 hours before mixing

- The sand content to be added in the mix is calculated (for example 10 kg). A slightly higher content M_{cont} is weighed (for example 12 kg) and put in a container that can be used for drying sand in an oven. Sand in container is dried in the oven at 60°C for 24 hours. On 3 samples from the container it should be tested that a fully dry state of the sand is achieved, by ensuring that the sand is dried until constant mass. Mass can be defined as constant mass when the difference in the weight of two subsequent measurements performed during 1 hour is less than 0.1% (according to EN 1097-5 Tests for mechanical and physical properties of

aggregates. Determination of the water content by drying in a ventilated oven).

2. The container with the sand is taken out from the oven and kept in laboratory conditions (ambient temperature $T_A = 20 \pm 2^\circ\text{C}$, relative humidity $\text{RH} \geq 60\%$) at least several hours before mixing until the temperature of the sand is $20 \pm 2^\circ\text{C}$.
3. From the container exact amount of sand, M_{sand} , necessary for mix is weighted and added to the mixer after the gravel.

4 MIXING

4.1 Concrete

The mixing procedure for concrete needs to be performed at the following laboratory conditions: ambient temperature $T_A = 20 \pm 2^\circ\text{C}$, relative humidity $\text{RH} \geq 60\%$. If mixing takes place at differing conditions, the real temperature and humidity have to be noted and reported. The following mixing procedure based on EN 480-1 standard [3] should be adopted to ensure repeatability of results:

1. Wipe the inside of the mixer with a damp cloth (if dry).
2. Introduce gravel and then sand into the mixer.
3. Mix the inert materials alone during 30 seconds.
4. Introduce the cement in the vessel and mix during 30 seconds. The start of the mix of this stage, i.e. the first contact of cement with mixing water is noted and defined as time zero (t_0) of the reaction.
5. Introduce the water with the superplasticizer over the next 30 seconds.
6. Mix for 1 minute. Stop mixing and remove the material from the walls of the vessel. Mix for another 1 min then stop mixing. Generally, this is the end of the mixture procedure.
7. If the mix does not seem homogeneous enough at such time, the mixing procedure should be continued until the desirable homogeneity is achieved. The additional time should be noted.

4.2 Mortar

The procedure described here should be followed when preparing mortar mixtures.

4.2.1 Laboratory conditions during test

The laboratory in which specimens are prepared and tested shall be maintained at a temperature of $(20 \pm 2)^\circ\text{C}$ and a relative humidity of not less than 50 %. The temperature and relative humidity of the air in the laboratory shall be recorded at least once per day during testing and reported. If prescribed temperature and humidity conditions cannot be achieved, the real temperature and humidity need to be noted and reported.

4.2.2 Materials preconditioning

All materials (including water) shall be kept under controlled environment (laboratory conditions - ambient temperature $T_A = 20 \pm 2^\circ\text{C}$, relative humidity RH not less than 50%) at least 24 hours before mixing. Target temperature of all components at the moment of mixing is 20°C . The initial temperature of all constituting materials prior to mixing has to be measured and reported to an accuracy of 0.1°C .

Sand has to be fully dried, following the procedure in the chapter 3.2, to avoid additional water that might be brought by wet sand.

Tap water shall be used for preparing the mixture.

4.2.3 Mixing procedure

Mixing procedure needs to be performed at the following laboratory conditions: ambient temperature $T_A = 20 \pm 2^\circ\text{C}$, relative humidity $\text{RH} \geq 50\%$. If mixing takes place at differing conditions, the real temperature and humidity have to be noted and reported. The following mixing procedure based on EN 480-1 standard should be adopted to ensure repeatability of results:

1. Wipe the inside of the mixer with damp cloth (if dry).
2. Put the sand and cement into the mixer and mix for 30 seconds.
3. Add the water with the superplasticizer over the next 30 seconds. This moment shall be recorded as "zero time".
4. Continue mixing for 60 seconds.
5. Stop mixing and remove the material from the walls of the vessel during 30 seconds.
6. Restart mixing for another 60 seconds and then stop mixing. Mixing is finished at this point. The entire mixing process lasts for 3 min 30 seconds. However if the mix does not seem homogeneous enough at such time, the mixing procedure should be continued until the desirable homogeneity is achieved. The additional time should be noted and reported.

4.3 Cement paste

The procedure described here should be followed when preparing cement paste mixtures.

4.3.1 Laboratory conditions during test

The laboratory in which specimens are prepared and tested shall be maintained at a temperature of $(20 \pm 2)^\circ\text{C}$ and a relative humidity of not less than 50 %. The temperature and relative humidity of the air in the laboratory and the temperature of the water in the storage containers shall be recorded at least once per day during testing.

4.3.2 Cement, water and superplasticizer conditioning

Cement paste, water and superplasticizer prior to mixing shall be stored at a temperature of $20 \pm 2^\circ\text{C}$. Temperature of all components shall be recorded prior to mixing to an accuracy of 0.1°C . **Tap water shall be used for preparing cement paste, except for hydration analysis where deionised water is to be used.**

4.3.3 Apparatus

- Analytical high precision balance (precision min 0.01g)
- Lab spoon, scoop and spatula
- Mixer in accordance to EN 196-3 and EN 196-1
- Timer

4.3.4 Mixing procedure

Mixing procedure described here closely follows procedure from standard EN 196-3. Mixing procedure is as follows:

1. Wipe the interior of the mixing bowl and mixing blades with a damp cloth prior to mixing.
2. Place the water with superplasticizer into the bowl and then add the cement taking care to avoid loss of water or cement. Complete the addition of cement within 10 s.
3. Immediately start the mixer at low speed ($140 \pm 5 \text{ min}^{-1}$) and start timing of the mixing stages. Record the time to the nearest minute as 'zero time'. (NOTE 'Zero time' is the point from which the initial and final setting times are calculated).
4. Stop the mixer after 90 s for 30 s during which remove by means of a suitable rubber or plastics scraper all the paste adhering to the wall and bottom part of the bowl and place it in the middle of the bowl;
5. Restart the mixer and run at low speed for a further 90 s. The total mixer running time shall be 3 min.



Mixer for preparing cement paste in accordance to EN 196-1 and EN 196-3



Blade to be used for preparing cement paste in accordance to EN 196-1 and EN 196-3

Figure 4.1 Equipment for mixing cement paste

5 MIX DESIGN

All mix designs presented below give **the amount of constituting materials that should be actually placed inside the mixer**. There is no need of additional calculation, only preconditioning of material is necessary.

Added water was calculated as an effective water (obtained from w_{eff}/c ratio) – $0.8 \cdot$ amount of the Sp (80% of Sp mass is water) + water theoretically absorbed by the sand (0.77% as coefficient of absorption). Therefore, the water that is given in the following tables is the amount of water that needs to be added in the mixer, with the condition that gravel is fully saturated and sand fully dry.

5.1 Ordinary concrete mixture (OC)

The OC mixture is the same reference mixture as used in the Initial experimental phase except the change in the amount of superplasticizer.

Table 5.1 Composition of the ordinary concrete OC mix used in the RRT⁺ program

Basic Material	Type of the material	Amount [kg/m ³]
Cement	CEM I 52,5 N CE CP2 NF Gaurain	320
Dry sand	0-4 mm, REC GSM LGP1 (13 % of CaO and 72 % of SiO ₂)	830
Fully saturated gravel	4-11mm, R GSM LGP1 (rounded, containing silicate and limestone)	449
	8-16 mm, R Balloy (rounded, containing silicate and limestone)	564
Admixtures	Plasticizer SIKAPLAST Techno 80	1.44
Added water	Water that needs to be added to the mixer	172.4
w_{eff}/c		0.52

5.2 Modified ordinary concrete mixture (MOC)

The MOC mixture is based on the OC mixture. The modifications are namely a decreased w/c ratio as well as the corresponding necessary changes in regard to the plasticizer and paste amount to achieve different properties of fresh and hardened material when compared to OC. The composition of MOC mixture is presented in Table 5.2. The mix is prepared with water (effective)-to-cement ratio 0.40, high-strength Portland cement and higher addition of chemical admixture in a form of plasticizer, compared to OC mix. The composition presented hereafter is based on the mass of fully water saturated gravel (4-11 mm and 8-16 mm), dry sand (0-4 mm) and added water. Concrete mix design, preconditioning of all materials (especially aggregate) and mixing should be done following the same procedure as described in sub-chapters 3 and 4.

Table 5.2 Composition of modified ordinary concrete MOC mix used in the RRT⁺ program

Basic Material	Type of the material	Amount [kg/m ³]
Cement	CEM I 52,5 N CE CP2 NF Gaurain	439
Dry sand	0-4 mm, REC GSM LGP1 (13 % of CaO and 72 % of SiO ₂)	772
Fully saturated gravel	4-11mm, R GSM LGP1 (rounded, containing silicate and limestone)	525
	8-16 mm, R Balloy (rounded, containing silicate and limestone)	424
Admixtures	Plasticizer SIKAPLAST Techno 80	3.73
Added water	Water that needs to be added to the mixer	178.4
w_{eff}/c		0.40

5.3 Ordinary (OM) and modified mortar (MOM) mixture

Composition of ordinary OM and modified mortar mixture are presented in Table 5.3 and Table 5.4.

Table 5.3 Composition of ordinary mortar OM mix used in the RRT⁺ program

Basic Material	Type of the material	Amount [kg/m ³]
Cement	CEM I 52,5 N CE CP2 NF Gaurain	525
Dry sand	0-4 mm, REC GSM LGP1 (13 % of CaO and 72 % of SiO ₂)	1361
Admixtures	Plasticizer SIKAPLAST Techno 80	2.36
Added water	Water that needs to be added to the mixer	282.7
w_{eff}/c		0.52

* Added water = Effective water (obtained from w_{eff}/c ratio) – 0.8*amount of the Sp (80% of Sp mass is water) + water theoretically absorbed by the sand (0.77% as coefficient of absorption)

Table 5.4 Composition of modified ordinary mortar MOM mix used in the RRT⁺ program

Basic Material	Type of the material	Amount [kg/m ³]
Cement	CEM I 52,5 N CE CP2 NF Gaurain	689
Dry sand	0-4 mm, REC GSM LGP1 (13 % of CaO and 72 % of SiO ₂)	1212
Admixtures	Plasticizer SIKAPLAST Techno 80	5.85
Added water		280.1
w_{eff}/c		0.40

5.4 Modified cement paste mixture (MCP)

Composition of modified cement paste MCP mixture is presented in Table 5.5. Only modified cement paste is given in the Protocols, since cement paste equivalent to the ordinary concrete would be too fluid (water-to-cement ratio of ordinary concrete is 0.5).

Table 5.5 Composition of modified ordinary cement paste OCP mix used in the RRT⁺ program

Basic Material	Type of the material	Amount [kg/m ³]
Cement	CEM I 52,5 N CE CP2 NF Gaurain	1299
Admixtures	Plasticizer SIKAPLAST Techno 80	5.85
Water		514.8
w_{eff}/c		0.40

6 GP1.a FRESH PROPERTIES AND SETTING

Hereafter protocols for heat of hydration, setting time and dynamic/ultrasonic test are given. Procedures from preparing cement paste, mortar and concrete for named tests are also given. Rheology tests can be performed in this phase, however no protocols are given, since this is highly depended in the available equipment of participants. Therefore, those participants who will perform rheology tests should submit a short description of the procedure that they used, together with their results.

6.1 Heat of hydration test on cement paste

Cement paste should be prepared as described in the chapter 4.3. Modified cement paste (MCP) should be used, using mix design given in Table 5.5.

Heat output shall be measured at least during the first 72 hours of hydration and maximum up to 7 days. If possible information about heat output should be recorded at 1-5 minutes interval. Heat output shall be reported in J/g or in W/g_(of cement).

Information about temperature of the specimen during test shall be reported, especially if the sample under test was not under isothermal conditions.

If test is performed on 2 or more specimens, from the same batch simultaneously, then result for each specimen shall be reported.

Measurement shall be repeated for 2 batches for each cement paste.

6.2 Heat of hydration test on mortar

Mortar should be prepared as described in the chapter 4.2. Ordinary mortar (OM) and modified mortar mix (MOM) should be used, using mix design given in Table 5.3. and 5.4.

Heat output shall be measured at least during the first 72 hours of hydration and maximum up to 7 days. If possible information about heat output should be recorded at 1-5 minutes interval. Heat output shall be reported in J/g or in W/g.

Information about temperature of the specimen during test shall be reported, especially if the sample under test was not under isothermal conditions.

If test is performed on 2 or more specimens, from the same batch simultaneously, then result for each specimen shall be reported.

Measurement shall be repeated for 2 batches for each mortar mixture.

6.3 Heat of hydration test on concrete

Concrete should be prepared as described in the chapter 4.1. Ordinary concrete (OC) and modified concrete mix (MOC) should be used, using mix design given in Table 5.1. and 5.2.

Samples for testing of setting time and dynamic/ultrasonic test shall be taken from the same batch.

Heat output shall be measured at least during the first 72 hours of hydration and maximum up to 7 days. If possible information about heat output should be recorded at 1-5 minutes interval. Heat output shall be reported in J/g or in W/g.

Information about temperature of the specimen during test shall be reported, especially if the sample under test was not under isothermal conditions.

If test is performed on 2 or more specimens, from the same batch simultaneously, then result for each specimen shall be reported.

Measurement shall be repeated for 2 batches for each concrete mixture.

6.4 Setting time test and dynamic/ultrasonic test on cement paste

Cement paste should be prepared as described in the chapter 4.3. Modified cement paste (MCP) should be used, using mix design given in Table 5.5.

6.4.1 Equipment

1. Balance, capable of weighing to an accuracy of ± 1 g
2. Mixer, conforming to EN 196-1
3. Thermometer capable of measuring temperature to an accuracy of $0,1^{\circ}\text{C}$
4. System for measuring temperature during the entire test at an interval of 5 minutes or less

Cement paste should be prepared as described in the chapter 4.3. Modified cement paste (MCP) should be used, using mix design given in Table 5.5.

6.4.2 Setting time test

For determination of setting time procedure described in standard EN 196-3 shall be applied for all cement paste mixtures. Vicat apparatus described in EN 196-3 shall be used for testing (manual or automatic) with the total mass of moving parts equal to (300 ± 1) g. Vicat apparatus shall be equipped with steel needle and in the form of a right cylinder of effective length of at least 45 mm and diameter $(1,13 \pm 0,05)$ mm.

The Vicat mould to contain the paste under test shall be of hard rubber. It shall be of truncated conical form $(40,0 \pm 0,2)$ mm deep and shall have an internal diameter of (75 ± 10) mm. It shall be adequately rigid and shall be provided with a baseplate larger than the mould

and at least 2,5 mm thick, constructed of impermeable material resistant to attack by cement paste, e.g. plane glass.

Filling the mould

Transfer the paste immediately to the lightly oiled mould, which has previously been placed on the lightly oiled base-plate, and fill it to excess without undue compaction or vibration. Remove any voids in the paste by gently tapping the slightly overfilled mould. Remove the excess by a gentle sawing motion with a straight-edged implement in such a way as to leave the paste filling the mould and having a smooth upper surface.

6.4.3 Determination of initial and final setting time

Place the filled mould and base-plate in the container, add water (tap water) so that the surface of the paste is submerged to a depth of at least 5 mm, and store in the temperature controlled enclosure at $(20,0 \pm 1,0)$ °C. At the time of measurement, position the mould and base-plate under the needle of the Vicat apparatus. Lower the needle gently until it is in contact with the paste. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the needle to penetrate vertically into the paste. Read the scale when penetration has ceased, or 30 s after the release of the needle, whichever is the earlier. Record the scale reading, which indicates the distance between the end of the needle and the base-plate (rounded to 1 mm), together with the time from "zero time". Repeat the penetration on the same specimen at conveniently spaced positions, not less than 8 mm from the rim of the mould or 5 mm from each other and at least 10 mm from the last penetration position, at conveniently spaced intervals of time, e.g. at 10 min intervals. Between penetrations keep the specimen in the container in the temperature controlled enclosure. Clean the Vicat needle immediately after each penetration.

The time elapsed between "zero time" and the time at which the distance between the needle and the base-plate is (6 ± 3) mm, measured to the nearest minute, is the initial setting time of the cement. After reaching the initial setting time continue making penetrations at a 10 minutes interval **without inverting the mould**. Penetrations will be made until the needle first penetrates not more than 0,5 mm into the specimen. The elapsed time in minutes, measured from "zero time" to that at which the needle first penetrates not more than 0,5 mm into the specimen, is the final setting time of the cement paste.

Report all readings from the time when needle has not reached the base plate for the first time.

For determination of setting time 3 specimens will be made from the same batch. Two specimens will be used for testing setting time and one specimen will be used for measuring temperature inside the specimen. Penetration results shall be reported for each specimen. Temperature sensor shall be placed approximately in the centre of the specimen and temperature shall be measured at an interval of 1 - 5 minutes. All specimens shall be kept in the container submerged to a depth of at least 5 mm.

6.4.4 Ultrasonic or other advanced techniques for monitoring setting

Transfer the paste immediately after the end of mixing to the mould for testing. Only light tapping of the mould is allowed during filling (no vibration shall be applied).

Measurement should start as soon as possible after mixing.

Temperature of the environment shall be maintained at $20 \pm 2^\circ\text{C}$.

If possible a layer of water will be maintained on the top of the tested specimen.

Temperature shall be measured in the specimen under test (or in a "twin" specimen) at an interval of 1-5 minutes.

Readings of ultrasonic parameters shall be made at regular intervals (preferably 1-5 minutes) during the entire setting process and at least for first 24 hours.

Measurement for each cement paste shall be repeated on specimens sampled from 2 batches. If test is performed on 2 or three specimens simultaneously then result for each specimen shall be reported.

6.4.5 Additional test using modified mixing procedure

Due to large impact of the mixing speed on bleeding of cement paste additional experiments can be made in which a high speed mixer is used for mixing of cement paste instead of the standard mixer conforming to EN 196-1.

High-speed overhead mixer with a mixer propeller diameter of 2-4 cm shall be used for mixing. Cement paste shall be mixed for 2 minutes at a speed of 1500 rpm. Immediately after mixing cement paste will be transferred to mould for setting time test and/or ultrasonic test.

6.4.6 Measuring density of hardened cement paste

After the setting time test and test using ultrasonic testing technique are finished samples will be stored under water until the age of 28 days is reached. Bulk density of the samples will be determined as an additional check of the homogeneity of samples.

Dry the specimen in the oven at a temperature of $105 \pm 5^\circ\text{C}$ until the constant mass is reached. The specimen is considered to have reached constant mass if two consecutive weighing's 2 h apart during the drying do not differ by more than 0,2% of the mass of the dry specimen. Record the mass of the specimen, $m_{s,dry}$ in kg to the nearest 0,1 %.

Immerse the specimen in water at $20^\circ\text{C} \pm 2^\circ\text{C}$ until no further increase in apparent mass is observed. This is considered reached when two consecutive weighing's, 15 min apart during the immersion do not differ by more than 0,2% by mass. Weigh the wet specimen after the removal of any surplus water, using a moist cloth, and record its mass, $m_{s,sat}$ in kg to the nearest 0,1%.

The volume of the specimen may now be determined by weighing in water. During weighing avoid trapping air bubbles on the sides of the specimen and record the mass of the specimen $m_{s,i}$ in kg in immersed position.

The volume of the specimen in m^3 is given by the formula:

$$V_s = \frac{m_{s,sat} - m_{s,i}}{\rho_w}$$

where ρ_w is density of water (998 kg/m^3).

Bulk density of each specimen is the ratio of $m_{s,dry}$ to the volume V_s .

6.5 Dynamic/ultrasonic measurement and setting time test on mortar

Mortar should be prepared as described in the chapter 4.2. Ordinary mortar (OM) and modified mortar mix (MOM) should be used, using mix design given in Table 5.3. and 5.4.

6.5.1 Fresh mortar properties

Just after the mixing, the following properties of fresh mortar have to be tested:

- Consistence of fresh mortar by flow table (EN 1015-3)
- Air content – using pressure method described in standard EN 1015-7
- Density – using methods described in standard EN 1015-6

Each property shall be tested at least once.

Density shall be determined in the same measuring vessel by which the air content of the fresh mortar is determined. Vessel shall be filled in one layer and vibrated on vibrating table. Time (in seconds) needed for compaction shall be reported.

6.5.2 Setting time test

Determination of setting time shall be conducted by following procedure described in standard EN 480-2 and/or ASTM C403 / C403M.

Procedure according to EN 480-2

If procedure described in standard EN 480-2 is used than Vicat apparatus described in EN 196-3 shall be used for testing (manual or automatic) with the total mass of moving parts equal to (1000 ± 2) g. Steel needle of effective length of at least 50 mm and diameter $(1,13 \pm 0,05)$ mm is used.

The Vicat mould to contain the paste under test shall be of hard rubber. It shall be of truncated conical form $(40,0 \pm 0,2)$ mm deep and shall have an internal diameter of (75 ± 10) mm. It shall be adequately rigid and shall be provided with a baseplate larger than the mould and at least 2,5 mm thick, constructed of impermeable material resistant to attack by cement paste, e.g. plane glass.

Moulds of other shape or of larger diameter can be used but the depth shall be $(40,0 \pm 0,2)$ mm. If this is the case than dimensions of the mould shall be reported.

Mould shall be sealed to the baseplate so that the mortar can be vibrated.

Filling the mould

Transfer the mortar immediately after mixing to the lightly oiled mould, which has previously been placed on the lightly oiled base-plate and compact it using the same method as will be applied for dynamic/ultrasonic measurement.

Remove the excess by a gentle sawing motion with a straight-edged implement in such a way as to leave the mortar filling the mould and having a smooth upper surface.

Determination of initial and final setting time

Keep the filled mould at a temperature of $(20 \pm 2,0)$ °C and at a relative humidity of not less than 50 %. Mould shall be covered by a plastic sheet to reduce evaporation from the sample.

At the time of measurement, position the mould and base-plate under the needle of the Vicat apparatus. Lower the needle gently until it is in contact with the mortar. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the needle to penetrate vertically into the mortar. Read the scale when penetration has ceased, or 30 s after the release of the needle, whichever is the earlier. Record the scale reading, which indicates the distance between the end of the needle and the base-plate (rounded to 1 mm), together with the time from "zero time". Repeat the penetration on the same specimen at conveniently spaced positions, 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 immediately after each penetration.

The time elapsed between "zero time" and the time at which the distance between the needle and the base-plate is 4 mm, measured to the nearest minute, is the initial setting time of the mortar.

After reaching the initial setting time continue making penetrations at a 10 minutes interval **without inverting the mould**. Penetrations will be made until the needle first penetrates not more than 2,5 mm into the specimen. The elapsed time in minutes, measured from "zero time" to that at which the needle first penetrates not more than 0,5 mm into the specimen, is the final setting time of the mortar.

Report all readings from the time when needle has not reached the base plate for the first time.

For determination of setting time 3 specimens will be made from the same batch. Two specimens will be used for testing setting time and one specimen will be used for measuring temperature inside the specimen. Penetration results shall be reported for each specimen. Temperature sensor shall be placed approximately in the centre of the specimen and temperature shall be measured at an interval of 1 - 5 minutes.

Procedure according to ASTM C403 / C403M

In this procedure penetration needles with 6 different diameters are used.

Filling the mould

Transfer the mortar immediately after mixing to the lightly oiled mould and compact it using the same method as will be applied for dynamic/ultrasonic measurement. Cylindrical moulds shall be used for testing having the diameter of 150 mm and height 150 mm.

Determination of initial and final setting time

Keep the filled mould at a temperature of $(20 \pm 2,0)$ °C and at a relative humidity of not less than 50 %. Mould shall be covered by a plastic sheet to reduce evaporation from the sample.

After each penetration, penetration resistance in MPa shall be calculated. All readings from the start of the test shall be reported. Initial and final setting times shall be calculated by curve fitting into the penetration resistance-time data.

For determination of setting time 3 specimens will be made from the same batch. In one specimen thermocouple shall be installed for measuring temperature inside the specimen.

Penetration results shall be reported for each specimen. Temperature sensor shall be placed approximately in the centre of the specimen and temperature shall be measured at an interval of 1 - 5 minutes.

6.5.3 Ultrasonic or other advanced techniques for monitoring setting

Transfer the mortar immediately after the end of mixing to the mould for testing and compact it using the same method as will be applied for setting time measurement.

Measurement should start as soon as possible after mixing.

Temperature of the environment shall be maintained at $20\pm 2^{\circ}\text{C}$.

Upper surface of the specimen shall be covered by plastic sheet to reduce evaporation.

Temperature shall be measured in the specimen under test (or in a "twin" specimen) at an interval of 1-5 minutes.

Readings of ultrasonic parameters shall be made at regular intervals (preferably 1-5 minutes) during the entire setting process and at least for first 24 hours.

Measurement for each mortar shall be made at least twice. If test is performed on 2 or three specimens simultaneously that average value shall be reported.

6.5.4 Measuring density of hardened mortar

After the setting time test and test using ultrasonic testing technique are finished samples will be stored under water until the age of 28 days is reached. Bulk density of the samples will be determined as an additional check of the homogeneity of samples.

Dry the specimen in the oven at a temperature of $105\pm 5^{\circ}\text{C}$ until the constant mass is reached. The specimen is considered to have reached constant mass if two consecutive weightings 2 h apart during the drying do not differ by more than 0,2% of the mass of the dry specimen. Record the mass of the specimen, $m_{s,dry}$ in kg to the nearest 0,1 %.

Immerse the specimen in water at $20^{\circ}\text{C}\pm 2^{\circ}\text{C}$ until no further increase in apparent mass is observed. This is considered reached when two consecutive weightings, 15 min apart during the immersion do not differ by more than 0,2% by mass. Weigh the wet specimen after the removal of any surplus water, using a moist cloth, and record its mass, $m_{s,sat}$ in kg to the nearest 0,1%.

The volume of the specimen may now be determined by weighing in water. During weighing avoid trapping air bubbles on the sides of the specimen and record the mass of the specimen $m_{s,i}$ in kg in immersed position.

The volume of the specimen in m^3 is given by the formula:

$$V_s = \frac{m_{s,sat} - m_{s,i}}{\rho_w}$$

Where ρ_w is density of water (998 kg/m^3).

Bulk density of each specimen is the ratio of $m_{s,dry}$ to the volume V_s .

6.6 Dynamic/ultrasonic measurement and setting time test on concrete

6.6.1 Fresh concrete properties

Just after the mixing, the following properties of fresh mortar have to be tested:

- Consistence of fresh concrete by slump test (EN 12350-2)
- Air content – using pressure method described in standard EN 12350-7
- Density – using methods described in standard EN 12350-6

Density shall be determined in the same measuring vessel by which the air content of the fresh mortar is determined. Vessel shall be filled in 2 layers and vibrated on vibrating table. Time (in seconds) needed for compaction shall be reported.

Each mandatory test has to be performed at least once. Time of each test needs to be noted in the provided forms.

6.6.2 Setting time test (OPTIONAL)

Determination of setting time shall be conducted by following procedure described in standard ASTM C403 / C403M.

Procedure according to ASTM C403 / C403M

In this procedure penetration needles with 6 different diameters are used. Mortar sample shall be obtained by sieving the concrete through 4 mm sieve.

Filling the mould

After sieving, mortar will be thoroughly remixed by hand before filling the mould. Transfer the mortar immediately after re-mixing to the lightly oiled mould and compact it. Method of compaction should be selected based on the consistency of sieved mortar (sieved mortar is generally of fluid consistency).

Cylindrical moulds shall be used for testing having the diameter of 150 mm and height 150 mm.

Determination of initial and final setting time

Keep the filled mould at a temperature of $(20 \pm 2,0)$ °C and at a relative humidity of not less than 50 %. Mould shall be covered by a plastic sheet to reduce evaporation from the sample.

After each penetration, penetration resistance in MPa shall be calculated. All readings from the start of the test shall be reported. Initial and final setting times shall be calculated by curve fitting into the penetration resistance-time data.

For determination of setting time 3 specimens will be made from the same batch. In one specimen thermocouple shall be installed for measuring temperature inside the specimen. Penetration results shall be reported for each specimen. Temperature sensor shall be placed approximately in the centre of the specimen and temperature shall be measured at an interval of 1 - 5 minutes.

6.6.3 Ultrasonic or other advanced techniques for monitoring setting

Transfer the concrete immediately after the end of mixing to the mould for testing and compact it on the vibrating table or using a vibrating needle.

Measurement should start as soon as possible after mixing.

Temperature of the environment shall be maintained at $20 \pm 2^\circ\text{C}$.

Upper surface of the specimen shall be covered by plastic sheet to reduce evaporation.

Temperature shall be measured in the specimen under test (or in a "twin" specimen) at an interval of 1-5 minutes.

Readings of ultrasonic parameters shall be made at regular intervals (preferably 1-5 minutes) during the entire setting process and at least for first 24 hours.

Measurement for each concrete shall be made at least twice. If test is performed on 2 or three specimens simultaneously that average value shall be reported.

6.6.4 Measuring density of hardened concrete (MANDATORY)

After the tests using dynamic/ultrasonic testing technique are finished samples will be stored under water until the age of 28 days is reached. Bulk density of the samples will be determined as an additional check of the homogeneity of samples.

Dry the specimen in the oven at a temperature of $105 \pm 5^\circ\text{C}$ until the constant mass is reached. The specimen is considered to have reached constant mass if two consecutive weighings 24 h apart during the drying do not differ by more than 0,2% of the mass of the dry specimen. Record the mass of the specimen, $m_{s,dry}$ in kg to the nearest 0,1 %.

Immerse the specimen in water at $20^\circ\text{C} \pm 2^\circ\text{C}$ until no further increase in apparent mass is observed. This is considered reached when two consecutive weighings, 24 hours apart during the immersion do not differ by more than 0,2% by mass. Weigh the wet specimen after the removal of any surplus water, using a moist cloth, and record its mass, $m_{s,sat}$ in kg to the nearest 0,1%.

The volume of the specimen may now be determined by weighing in water. During weighing avoid trapping air bubbles on the sides of the specimen and record the mass of the specimen $m_{s,i}$ in kg in immersed position.

The volume of the specimen in m^3 is given by the formula:

$$V_s = \frac{m_{s,sat} - m_{s,i}}{\rho_w}$$

Where ρ_w is density of water (998 kg/m^3).

Bulk density of each specimen is the ratio of $m_{s,dry}$ to the volume V_s .

7 GP1b: CHEMICAL AND MICROSTRUCTURAL CHARACTERIZATION

As a first part of the Main phase only characterization of cement (particle size distribution, particle density, specific surface area, chemical composition and mineral phase composition) and hydration analysis (TG and QXRD) are performed. Microstructure and pore structure should follow later, once results of the first part are analysed.

7.1 Cement characterization protocols

This document describes the protocols for round robin characterization of cement using five techniques, i) Particle size distribution, ii) Particle density, iii) Specific surface area, iv) Chemical composition, v) Mineral phase composition.

7.1.1 Materials

- CEM I 52,5 N CE CP2 NF GaurainN (supplied by the coordinator)

7.1.2 Apparatus

- *Particle size distribution*
 - Laser diffraction particle size analyzer
 - Ultrasonic stirrer
 - Dropper
- *Specific surface area*
 - Blaine apparatus
 - Filter paper
- *Particle density*
 - Pycnometer
 - Water bath at 20 °C
 - Vacuum chamber
- *Chemical composition*
 - X-ray fluorescence spectrometer
- *Mineral phase composition*
 - X-ray Diffractometer with Rietveld software
- **Sampling**
 - Sampling of cement should be done using the scoop method.
 - First mix the cement in the bucket by using a spatula, and then take the sample from the middle.
 - Avoid surface region for mixing and sampling because this region may be affected by segregation or alterations such as carbonation and hydration. First remove the surface region.
 - Take several small samples from the middle at different positions rather than taking one large sample from one position.
 - Mix the different samples afterwards to homogenise and to obtain a single sample for characterisation.

- **Particle size distribution by wet laser diffraction**

- Mix approximately 500 mg of cement in 100 ml dispersant of ethanol or isopropanol to apply wet dispersion of cement particles.
- Mix thoroughly with a spatula and transfer the dispersion to ultrasonic bath with stirrer (at around 1,700 rpm rate) and mix for 5 minutes.
- Transfer the dispersion to the laser diffraction particle size analyzer using a dropper till 15-20% obscuration level
- Prepare three dispersions and carry out three repetitions with each dispersion (ie. Repeat the measurement three times for each).
- Report average results of cumulative and differential particle size distributions along with D_{10} , D_{50} and D_{90}
- If possible, use optical parameters of $n = 1.7$ and $k = 0.1$ (where n and k are the real and the imaginary parts of the complex index of refraction). Otherwise, participants should report the optical parameters of the equipment and keep them constant for the measurements.
- Report the conditions, parameters and results in the following table.

	Cement sample I	Cement sample II	Cement sample III
Instrument name			
Powder weight (mg)			
Dispersing agent (isopropanol or ethanol)			
Dispersing liquid volume (mL)			
Ultrasonic treatment (W, kHz, min)			
Stirring rate (rpm)			
Data acquisition time (s)			
Obscuration			
Optical parameters: n , k , liquid			
D_{10} (μm)			
D_{50} (μm)			
D_{90} (μm)			
$SSA_{\text{PSD-LD}}$ (m^2/g)			

- **Particle density**
 - Follow the procedure described in the European Standard EN 196-6 which describes the pycnometer method (on the basis of volumetric displacement of a fluid) for the determination of the density of cement.
 - Perform three measurements at 20 °C
 - Use white spirit as a non-reactive liquid
 - Keep pycnometer in vacuum for 24 hours after filled with cement and white spirit to extract all air out of specimen
 - Report the mean of the three results to the nearest 0,01 g/cm³ as the density

- **Specific surface area by air permeability test (Blaine method)**
 - Perform the test according to the method described in the European Standard EN 196-6.
 - Use the particle density of the cement
 - Prepare a bed of binder of porosity 0.5 in the cell on top of perforated disc and put filter paper on top and bottom of the bed
 - Compact it using a plunger
 - Find the time required for the mercury level to drop using automatic or manual Blaine apparatus
 - Find the surface area according to time and calibration parameters of the equipment
 - Perform three measurements
 - Report the mean of the three results in cm²/g

- **Chemical composition by XRF**
 - Grind the particles to pass a 63 μm sieve
 - Perform analysis using an X-ray fluorescence spectrometer to evaluate quantitatively oxide and elemental composition of cement
 - Perform three times using a new sample of cement
 - Normalize the results to 100%
 - Report the mean of the three results of the oxide composition and elemental composition in %

- **Mineral phase composition by QXRD**
 - The XRD analysis will be used to calculate the quantitative phase composition of main anhydrous phases of cement: alite (C₃S), belite (β-C₂S), aluminat (C₃A), ferrite (C₄AF) and gypsum.
 - Measurement settings:
 - Grind the particles to pass a 63 μm sieve
 - Back-load or side-load the sample into the holder to minimize preferred orientation
 - Scan parameters:
 - Angular range: 5-70 °2θ (CuKα)
 - Step size: 0.04 °2θ
 - Time per step (or equivalent time per step): 2 s
 - Rietveld analysis:

- The XRD data shall be analyzed by Rietveld analysis to determine the crystal structure parameters (lattice parameters, peak width determining parameters) of the anhydrous phases.
- To avoid deviations and to maintain consistency in the results, a list of pattern and crystal structure references is given in Table 7.6 to be introduced in the refinement software.
- Data analysis and settings:
 - Qualitative analysis software: Diffrac.Suite Eva, HighScore, etc
 - Quantitative analysis software: Topas (Academic), HighScore (Plus), etc.
 - Refined global parameters, and sequence of refinement: Specimen displacement, background type and number of coefficients
 - Refined phase specific parameters, and sequence of refinement: Scale factors, lattice parameters, peak shape type and parameters, preferred orientation correction
 - Parameter constraints: Limits on lattice parameter shift, peak shape parameters
 - Fixed parameters: Atomic positions, atomic displacement parameters,...
- Use internal standard approach for phase quantification. Attention must be paid to achieve a thorough blending and intermixing of the sample and standard (eg. ZnO). A 10 wt.% addition is advised.
- Do not expose the sample to atmospheric conditions for prolonged time as the samples may carbonate.
- Perform three measurements and analyses.
- Report the mean result of phase compositions.

7.1.3 Guidelines to data reporting

The participants shall:

- Enter the final results in the RRT excel file and send it to the GP1B group leader (ozlem.cizer@kuleuven.be). Please take care to fill out the correct values taking into account the appropriate normalization basis.
- Report briefly on the analysis procedure, conditions and storage of samples.
- Send the original files in a common format (.txt., .csv,...) to the group leader.

References:

- 1) Eleni C. Arvaniti, Maria C. G. Juenger, Susan A. Bernal, José´e Duchesne, Luc Courard, Sophie Leroy, John L. Provis, Agnieszka Klemm, Nele De Belie, Physical characterization methods for supplementary cementitious materials, *Materials and Structures* (2015) 48:3675–3686.
- 2) Eleni C. Arvaniti, Maria C. G. Juenger, Susan A. Bernal, José´e Duchesne, Luc Courard, Sophie Leroy, John L. Provis, Agnieszka Klemm, Nele De Belie, Determination of particle size, surface area, and shape of supplementary cementitious materials by different techniques, *Materials and Structures* (2015) 48:3687–3701

- 3) Ferraris, C. F., V. A. Hackley, A. I. Aviles and C. E. Buchanan (2002a). 'Analysis of the ASTM Round Robin Test on Particle Size Distribution of Portland Cement: Phase I'. NIST Interagency or Internal Report 6883."
- 4) "Ferraris, C. F., V. A. Hackley, A. I. Aviles and C. E. Buchanan (2002b). 'Analysis of the ASTM Round Robin Test on Particle Size Distribution of Portland Cement: Phase II'. NIST Interagency or Internal Report 6931."
- 5) EN 196-6 (2010) Methods of testing cement—Part 6: Determination of fineness
- 6) Karen L. Scrivener, Barbara Lothenbach, Nele De Belie, Elke Gruyaert, Jørgen Skibsted, Ruben Snellings, Anya Vollpracht, TC 238-SCM: hydration and microstructure of concrete with SCMs: State of the art on methods to determine degree of reaction of SCMs, Materials and Structures (2015) 48:835–862.
- 7) Karen Scrivener, Ruben Snellings, Barbara Lothenbach, A Practical Guide to Microstructural Analysis of Cementitious Materials. CRC Press, 2015.

7.2 Sample preparation protocol for hydration analysis of cement

Cement paste should be prepared as described in the chapter 4.3. Modified cement paste (MCP) should be used, using mix design given in Table 5.5. **Deionized water or distilled water (20°C) should be used to prepare cement paste.** Four replicates of cement paste should be casted in total for the designated hydration ages (1, 3, 7 and 28 days) following the procedure below.

Procedure:

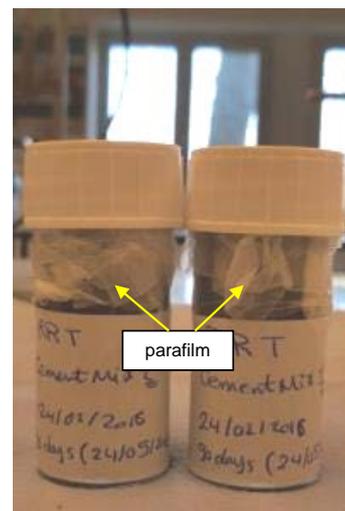
- Cast the paste in the 40-50 mL plastic vessels in three layers, tap gently to remove air bubbles in the paste, fill up the vessel until no space is left. **Cast 5 vessels in total** (4 vessels for analysis at designated ages and 1 vessel reserved in case needed)
- First cover the vessel opening with two layers of parafilm, then close it with the screw cap and make sure that it is well-closed to prevent evaporation and leaking out of mixing water.
- Store the samples at 20 ± 0.5 °C for 28 days.



Use 50 mL plastic vessel for casting cement pastes



Cast cement paste in plastic vessels and fill it up



Seal the vessel opening with at least 2 layers of parafilm before screwing the cap

Figure 7.1 Procedure for preparation of samples for hydration analysis

7.3 Hydration stoppage protocol

This document describes hydration stoppage protocol for hydrated cement pastes by means of solvent exchange method using isopropanol. This method is based on diluting and removing the water present in the pores of the hydrated cement paste by a solvent.

7.3.1 Materials

- Cement paste hydrated for 1, 3, 7, 28 days (sealed)

7.3.2 Apparatus

- Sampling:
 - Mortar and pestle
 - Disk saw to cut slices
- Solvent exchange:
 - Isopropanol
 - Diethyl ether
 - Büchner filter, flask and beaker
 - Vacuum pump
 - Filter paper: diameter less than 2 μm
 - Watch glass

7.3.3 Procedure

- Sampling:
 - **On 1, 3, 7, 28 days** of hydration of pastes casted in vessels, sample approximately 3 g of hydrated paste using a saw to cut a slice **from the middle part** of the hardened cylinder paste as illustrated in Figure 7.1.
 - Avoid sampling slices or pieces close to the upper surface of the hardened paste. Remove the outer edges (3 mm). They may be carbonated or dried out.
 - Crush and grind the sliced piece using a mortar and pestle to < 1 mm. Pay attention not to wait too long or spend too much time while grinding. Samples may carbonate.
- Solvent exchange:
 - Install the Büchner filter and flask under a fume hood
 - Carefully fix the filter paper on the Büchner filter and pour some isopropanol on the filter to fix it properly without any leaks so that the vacuum is properly performed.
 - Immerse the ground sample in a beaker with 100 mL of isopropanol and leave it for 15 min
 - Stir the suspension, pour it gently on the filter, and let the isopropanol percolate under vacuum (Figure 7.2 and 7.3).
 - Rinse the residue using isopropanol one time (1x)
 - Rinse the residue using diethyl ether two times (2x). Diethyl ether is extremely flammable and should not be inhaled.
 - Put the residue and filter paper on a watch glass and dry at 40 °C for 8 min in a ventilated oven

- Store dried samples in a low vacuum dessicator over silica gel until the time of analysis. Preferably, TG and XRD analysis should be done as soon as possible (1-3 days) after drying. Report the storage time and conditions.
- Repeat this procedure for each hydrated cement paste at designated hydration age (1, 3, 7, 28 days)

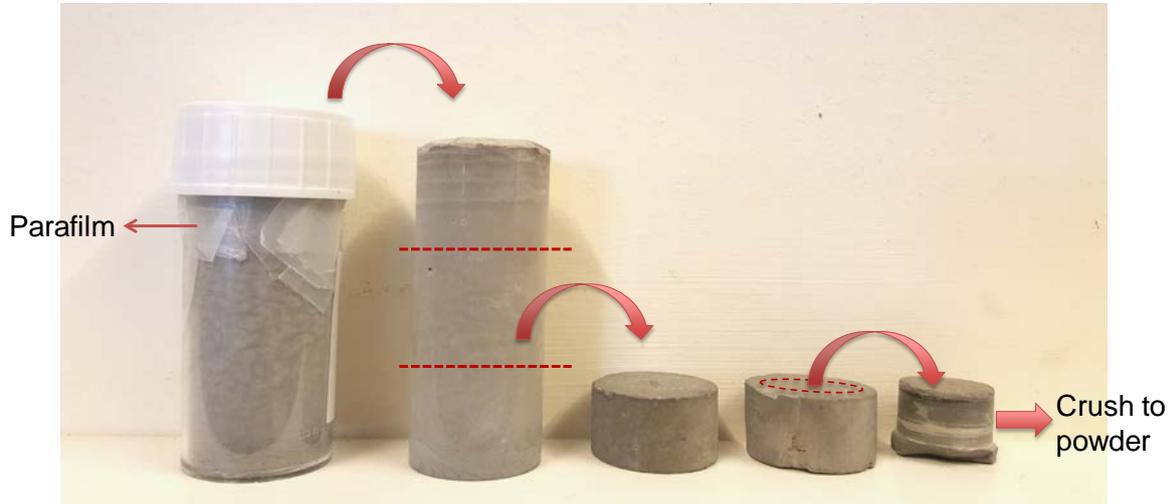


Figure 7.2 Sampling of hydrated/hardened cement paste for hydration stoppage.



Figure 7.3 Set-up components for solvent exchange method

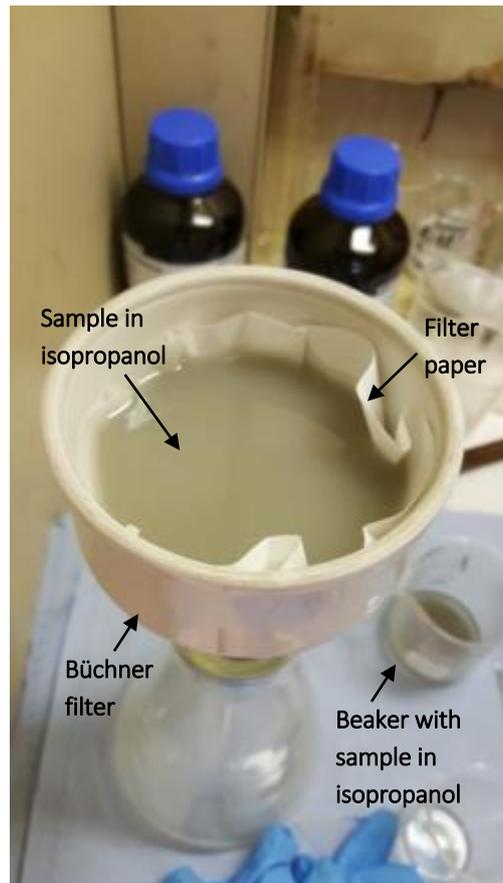


Figure 7.4 Sample powder in isopropanol percolating through the filter paper fixed on a Büchner filter under vacuum.

7.4 Hydration analysis protocols (TG and QXRD)

This document describes measurement settings and procedure for thermogravimetric (TG) and quantitative X-ray diffraction (QXRD) analysis of hydrated cement pastes, as well as guidelines to data analysis and data reporting.

7.4.1 Materials

- Portland cement hydrated for 1, 3, 7, 28 days after hydration is stopped using solvent exchange method.

7.4.2 Apparatus

- Thermogravimetric (TG) analyzer with a crucible (eg. Alumina crucible with a volume of 150 μL)
- X-ray Diffractometer with Rietveld software

7.4.3 Measurement settings and procedure

- TG
 - Grind samples in a mortar and pestle to pass through 63 μm sieve
 - Weigh the empty crucible
 - Weigh in 50 mg powder sample

- Apply heating regime from 20 to 1000 °C at 10 °C/min
- Use inert nitrogen atmosphere as purging gas at a flow rate of 30 to 50 mL/min
- Determine a blank curve for the selected crucible, heating procedure and purging gas before each series of measurement. Subtract the blank curve from the sample measurement.
- XRD
 - Grind the particles to pass a 63 µm sieve
 - Back-load or side-load the sample into the holder to minimize preferred orientation
 - Scan parameters:
 - Angular range: 5-70 °2θ (CuKα)
 - Step size: 0.04 °2θ
 - Time per step (or equivalent time per step): 2 s

7.4.4 Guidelines to data analysis

- TG data:

The TG data shall be analysed to calculate portlandite content and bound water content.

 - Portlandite (Ca(OH)₂) decomposes generally between 400 and 500°C to CaO and H₂O. Calculate the portlandite content (WL_{Ca(OH)₂}) using the tangential approach as illustrated in Figure 7.4 and formulas below using molecular mass of portlandite (M_{Ca(OH)₂} = 74 g/mol) and water (M_{H₂O} = 18 g/mol) (Scrivener et al. 2015a, Scrivener et al. 2015b, pp. 177-211):
$$\text{Ca(OH)}_{2,\text{measured}} = \text{WL}_{\text{Ca(OH)}_2} \times M_{\text{Ca(OH)}_2} / M_{\text{H}_2\text{O}} = \text{WL}_{\text{Ca(OH)}_2} \times 74/18$$
 - The bound water content should be calculated using the stepwise approach, an integral from 40°C - 650°C (Scrivener et al. 2015b, pp. 196-200). The bound water content should be reported based on 1) the initial mass basis (mass at 40°C), 2) the ignited mass basis (mass at 650°C).

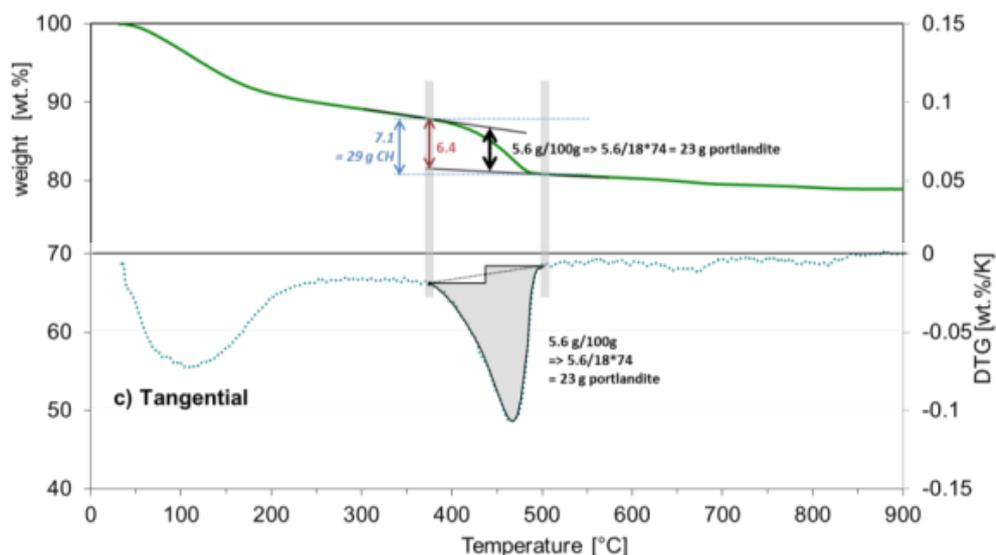


Figure 7.5 Tangential method to quantify portlandite content in a mix of C-S-H (75%) and portlandite (25%, i.e. 23% CH and 2% calcite) (taken from Scrivener et al. 2015b, p. 199)

- XRD data:

The XRD analysis will be used to calculate the quantitative phase composition of the hydrated samples: portlandite, ettringite and AFm (monosulfoaluminate, hemi/mono-carboaluminate) content.

- XRD data should be collected for all four hydrated samples (hydration stopped samples at 1, 3, 7, 28 days) using the same measurement settings
- Rietveld analysis should be used for data analysis and quantification:
 - The XRD data shall be analyzed by Rietveld analysis to determine the crystal structure parameters (lattice parameters, peak width determining parameters) of the anhydrous and hydrated phases.
 - To avoid deviations and to maintain consistency in the results, a list of pattern and crystal structure references is given in Table 7.1 in appendix to be introduced in the refinement software.
 - Data analysis and settings:
 - Qualitative analysis software: Diffrac.Suite Eva, HighScore, etc
 - Quantitative analysis software: Topas (Academic), HighScore (Plus), etc.
 - Refined global parameters, and sequence of refinement: Specimen displacement, background type and number of coefficients
 - Refined phase specific parameters, and sequence of refinement: Scale factors, lattice parameters, peak shape type and parameters, preferred orientation correction
 - Parameter constraints: Limits on lattice parameter shift, peak shape parameters
 - Fixed parameters: Atomic positions, atomic displacement parameters,...
- Use internal standard approach for phase quantification. Attention must be paid to achieve a thorough blending and intermixing of the sample and standard (eg. ZnO). A 10 wt.% addition is advised.
- Do not expose the sample to atmospheric conditions for prolonged time as the samples may carbonate.
- Perform three measurements and analyses.
- Report the mean result of phase compositions.

7.4.5 Guidelines to data reporting

The following information should be reported by the participants:

- Sample properties and storage conditions
 - Storage history of cement
 - Method to stop hydration
 - Storage history between hydration stoppage and analysis
 - Amount and fineness of the sample
- Boundary conditions for TG
 - TG instrument
 - Crucible type, size and volume
 - Temperature interval and heating rate

- Type and rate of the purging gas
- Results for TG
 - Complete TG curves, weight loss including temperature interval and quantification method
 - Report if carbonation is observed
- Boundary conditions for XRD
 - XRD instrument
 - Scan parameters
 - Scan time
- Normalisation to “g/100 g anhydrous”

The total mass of solid fraction is changing during cement hydration. Free water is bound into hydration products (Figure 7.5) and the initial porosity is filled. Therefore, both XRD and TG results need to be normalised to “g/100 g anhydrous” to compare samples of different ages on an equal basis using the following formula where (Scrivener et al. 2015b, p. 137-138):

$$\text{per 100 g anhydrous: } W_{j,anhydrous} = W_{j,measured} / (1 - H_2O_{bound,paste basis})$$

where W_j corresponds to the weight fraction of phase j , and H_2O bound refers to the bound water content versus the initial mass basis

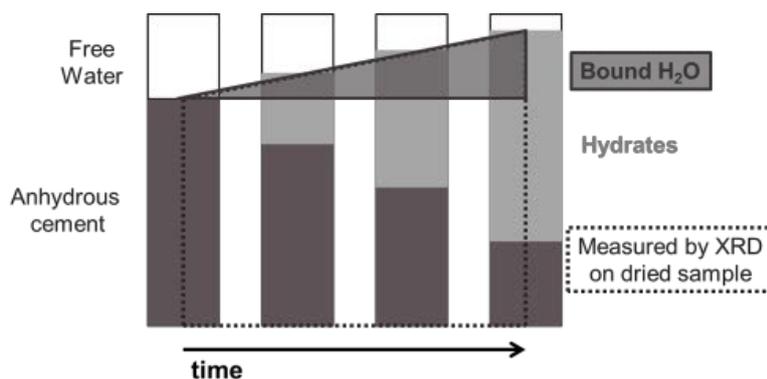


Figure 7.6 Diagram illustrating the need for back-calculation of the XRD/TG quantification results to a common basis (for instance 100 g of anhydrous cement) to enable comparison of time series data (Scrivener et al. 2015b, p. 137).

The participants shall:

- Enter the final results in the RRT excel file and send it to the GP1B group leader (ozlem.cizer@kuleuven.be). Please take care to fill out the correct values taking into account the appropriate normalization basis.
- Report briefly on the analysis procedure, conditions and storage of samples.
- Send the original files in a common format (.txt., .csv,...) to the group leader.

References:

- 1) Karen L. Scrivener, Barbara Lothenbach, Nele De Belie, Elke Grunyaert, Jørgen Skibsted, Ruben Snellings, Anya Vollpracht, TC 238-SCM: hydration and microstructure of concrete with SCMs: State of the art on methods to determine degree of reaction of SCMs, *Materials and Structures* (2015a) 48:835–862.

2) Karen Scrivener, Ruben Snellings, Barbara Lothenbach, A Practical Guide to Microstructural Analysis of Cementitious Materials. CRC Press, 2015b.

Table 7.1 Crystal structure models for Rietveld analysis (Courtesy of Ruben Snellings, Scrivener et al. 2015b)

Phase name	Chemical formula	Space group	Lattice parameters						ICSD number	PDF number	Reference
			a	b	c	α	β	γ			
Alite M3	Ca ₃ SiO ₅	Cm	33.	7.0	18.	-	94.	-	9474	-	(De la Torre et al. 2002)
Alite M1	Ca ₃ SiO ₅	Pc	108	36	521	-	137	-	2	85-1378	(de Noirfontaine et al. 2012)
Belite β	Ca ₂ SiO ₄	P2 ₁ /n	27.	7.0	12.	-	.03	-	-	-	(Mumme et al. 1995)
C ₃ A cubic	Ca ₃ Al ₂ O ₆	Pa-3	5.5	6.7	9.3	-	94.	-	8109	33-302	(Mondal and Jeffery 1975)
C ₃ A orthorhombic	Ca ₃ Al ₂ O ₆	Pa-3	12	58	14	-	58	-	6	38-1429	(Mondal and Jeffery 1975)
Ferrite	Ca ₂ Al _{0.99} Fe _{1.01} O ₅	Pbca	26	-	-	-	-	-	1841	01-070-	(Nishi and Takeuchi 1975)
Mullite	Al _{2.34} Si _{0.66} O _{4.83}	Pbca	10.	10.	15.	-	-	-	1880	0859	(Redhammer et al. 2004)
Periclase	MgO	Pbca	875	859	105	-	-	-	9883	01-074-	(Redhammer et al. 2004)
α -Quartz	SiO ₂	lbm2	5.5	14.	5.3	-	-	-	6	3672	(Voll et al. 2001)
Rutile	TiO ₂	Pbam	7.5	7.6	2.8	-	-	-	1580	-	(Sasaki et al. 1979)
Corundum	Al ₂ O ₃	Fm-3m	65	88	85	-	-	-	98	-	(Voll et al. 2001)
Arcanite	K ₂ SO ₄	P322	17	-	-	-	-	-	9863	43-1022	(Le Page and Donnay 1976)
Ca-langbeinite	Ca ₂ K ₂ (SO ₄) ₃	P42/mnm	4.9	4.9	5.4	-	-	-	174	46-1045	(Ballirano and Caminiti 2001)
Anhydrite	CaSO ₄	R-3c	4.5	-	2.9	-	-	-	9309	-	(Ballirano and Caminiti 2001)
Hemihydrate	CaSO ₄ ·0.5(H ₂ O)	R-3c	93	-	61	-	-	-	7	21-1276	(Caminiti 2001)
Gypsum	CaSO ₄ ·2(H ₂ O)	P2 ₁ /n	4.7	-	12.	-	-	-	7781	-	(Riello et al. 1997)
Calcite	CaCO ₃	Pmcn	60	-	992	-	-	-	0	10-173	(Riello et al. 1997)
Portlandite	Ca(OH) ₂	P2 ₁ 2 ₁	5.7	10.	7.4	-	-	-	7	5-613	(Ojima et al. 1995)
Ettringite	Ca ₆ Al ₂ (SO ₄) ₃ (OH) ₁₂ (H ₂ O) ₂₆	2 ₁	70	071	78	-	-	-	7	4098	(Speer and Salje 1986)
Monosulfaluminate	Ca ₄ Al ₂ (OH) ₁₂ (SO ₄) ₃ (H ₂ O) ₆	Amm	10.	10.	10.	-	-	-	9	20-867	(Cheng and Zussman 1963)
Hemicarboaluminate	Ca ₄ Al ₂ (OH) ₁₂ (OH)(CO ₃) _{0.5} (H ₂ O) ₅	a	6.9	6.9	6.2	-	-	-	1587	-	(Cheng and Zussman 1963)
Monocarboaluminate	Ca ₄ Al ₂ (OH) ₁₂ (CO ₃) _{0.5} (H ₂ O) ₅	l2	91	96	38	-	-	-	6	37-1496	(Zussman 1963)
			12.	6.9	12.	-	90.	-	7952	-	(Zussman 1963)
			032	27	671	-	27	-	9	41-224	(Bezou et al. 1995)
			6.2	15.	6.5	-	127	-	4095	-	(Bezou et al. 1995)
			84	2	23	-	.41	-	81	33-311	(Boeyens and Ichharam 2002)
			4.9	-	17.	-	-	-	7344	-	(Maslen et al. 1993)
			91	-	062	-	-	-	6	5-586	(Maslen et al. 1993)
			3.5	-	4.9	-	-	-	2022	-	(Chaix-Pluchery et al. 1987)
			92	-	06	-	-	-	20	4-733	(Chaix-Pluchery et al. 1987)
			11.	-	21.	-	-	-	1553	-	(Goetz-Neunhoeffer and Neubauer 2006)
			229	-	478	-	-	-	95	41-1451	(Goetz-Neunhoeffer and Neubauer 2006)
			5.7	-	26.	-	-	-	1001	01-083-	(Goetz-Neunhoeffer and Neubauer 2006)
			59	-	795	-	-	-	38	1289	(Allmann 1977)
			5.7	-	48.	-	-	-	-	-	(Runčevski et al. 2012)
			76	-	812	-	-	-	-	41-0221	(Runčevski et al. 2012)
			5.7	8.4	9.9	64.	82.	81.	5932	01-087-	(François et al. 1998)
			75	69	23	77	75	43	7	0493	(François et al. 1998)

8 GP1c: TRANSPORT PROPERTIES AND BOUNDARY EFFECTS

Tests in this work group are divided into two segments: (1) resistivity and chloride diffusivity and (2) air permeability and water absorption. Participants may refer to the following standardised procedures to carry out the tests. Protocols for all tests are given in the excel files that are provided to participants. Should participants wish to take deviations from the stated procedure, they should elaborate the steps taken and reasons whilst submitting test results.

8.1 Resistivity and chloride diffusivity

- Surface electrical resistivity – Florida Method of test for concrete resistivity.
- Non-steady state chloride diffusivity – EN12390.
- Non-steady state chloride diffusivity – NT Build 443.
- Non-steady state chloride migration – NT Build 492.
- Bulk electrical resistivity – Rilem procedure for assessing resistivity of concrete cylinders. This test should be performed on samples prepared for NT Build 492 or 443.

8.2 Air permeability and water absorption

- Capillary absorption – ASTM C1585.
- Capillary absorption – EN13057.

8.3 Non-standardised tests for transport properties

In addition, the following non-standardised tests will also be performed within GP1.c. Should any participant wish to perform these tests, discuss this directly with the GP leader.

- Colorimetric method for assessing chloride penetration.
- French method for air permeability.
- Rilem 116-PCD test for gas permeability.
- Modified Rilem 116-PCD test for capillary absorption.
- Autoclam air permeability test – in situ testing
- Autoclam water absorption test – in situ testing.
- Permit chloride migration test – in situ testing.

8.4 Concrete mixing, curing and preconditioning

Follow sections 3-5 of this report for preparing constituent materials, storage and concrete mixing. Mixing and reporting of fresh properties should be in accordance with the procedure outlined for initial phase. Concrete should be water cured until 28 days of age before initiating the preconditioning for different tests.

Should the participants wish to avail further information about any of the tests or data analysis support, discuss this directly with the GP leader.

9 GP1d: MECHANICAL PROPERTIES

Tests under GP1d can be broadly divided into parts. Part 1 will include determination of basic mechanical properties of cement paste, mortar and concrete that is compressive strength, splitting tensile strength and Young's modulus (with Poisson's ratio) and dynamic characterization of isotropic elasticity by ultrasound test. Part 2 will include tests for creep behaviour and advanced methods (such as nano-indentation). The advanced test methods will be carried out by limited number of laboratories and therefore the test protocols will be agreed among themselves.

9.1 Preconditioning of constituent materials

Precondition materials according to the instructions provided in section 3.

9.2 Mixing procedure

Concrete should be prepared according to the procedure outlined in the section 4.1, mortar in section 4.2 and cement paste in section 4.3.

9.3 Fresh properties (mandatory)

9.3.1 Consistency

Consistency of fresh cement paste should be evaluated by modified flow table test (EN 1015-3, [1]). Spreading of the test specimen after lifting of the mould and completing of the paste spreading should be measured (no dropping of the flow table should be performed). One test should be carried out for each batch of the paste.

Consistency of fresh mortar should be evaluated by flow table test (EN 1015-3, [1]). One test should be carried out for each batch of the mortar.

Consistency of fresh concrete should be evaluated according to EN 12350-2 [2]. One test should be carried out for each batch of the concrete.

9.3.2 Density

For cement paste and mortar procedure described in EN 1015-6 [3] should be followed, and for concrete procedure given in EN 12350-6 [4].

9.3.3 Stability

Expansion and bleeding test should be used to determine the amount of water that separates from freshly mixed cement paste and accumulates on its surface. The EN 445:2008 [5] or ASTM C 940 [6] standard procedure should be followed. However volume of grout tested should be reduced to 400 ± 10 ml.

Cement paste is placed in a graduated glass cylinder, and the change in total volume and the accumulation rate of bleed water on the surface of the paste is observed over a period of time. The volume of bleed water as a percentage of the total volume of grout is an indication of the extent of separation of the liquid and solid phases.

Equipment and Materials

- 500 mL graduated glass cylinder reading to 10 mL
- 25 mL graduated glass cylinder reading to 1 mL
- 10 mL syringe with cannula (#20 or smaller)
- thermometer accurate to within 1°C
- Parafilm or any other type of plastic wrap that will prevent evaporation of bleed water during testing



Procedure

1. Place the 500 mL graduated cylinder on a level surface that is free from vibration.
2. Mix the paste and immediately measure and record its temperature.
3. Introduce the paste into the graduated cylinder until the volume of the sample is 400 ± 10 mL. Volume measurements should begin within 3 minutes after mixing.
4. Cover the top of the graduated cylinder using Parafilm to prevent the evaporation of bleed water.
5. Record the total initial volume of the sample (V_0) and the time at which the reading was taken.
6. Record the volume at the upper surface of the bleed water layer (V_t) and at the upper surface of the cement paste (V_g), to the nearest 5 mL at 15 minute intervals for the first 60 minutes, and thereafter at hourly intervals until two successive readings show no further change in the volume of the paste.
7. At the end of the test, transfer the bleed water into a 25 mL graduated cylinder by tilting the cylinder and drawing the water off with the syringe. Record the final volume of the bleed water (V_w) to the nearest 0.5 mL.

Results

Calculate the bleeding of the paste (B) to the nearest 0.2% for each prescribed interval:

$$B = (V_t - V_g)/V_0 \times 100$$

where V_0 is the volume of the specimen at the beginning of the test; V_t is the volume of the specimen at prescribed intervals, measured at the upper surface of the water layer; and V_g is the volume of the grout portion of the specimen at prescribed intervals, measured at the upper surface of the grout, all in millilitres.

Calculate the final bleed water (FB) as a percentage of the initial volume of the paste, to the nearest 0.2%:

$$FB = V_w/V_0 \times 100$$

where V_0 is the volume of the specimen at the beginning of the test, and V_w is the volume of decanted bleed water, both in millilitres.

9.4 Casting of specimen and curing

Three specimens should be cast for each test for a given age of testing.

From cement paste and mortar, prisms of dimensions 40x40x80 mm should be cast. This can be done by inserting metal plate in the middle of the length of the standard mould 40x40x160 mm. Other equivalent measures to obtain the 40x40x80 mm hydrated cement paste and mortar prisms can be applied. The reason for this deviation in dimensions compared to the standard is the fact that mechanical properties will be used as an input for creep testing, where samples have ratio between width and length 1:2. As an alternative, laboratory can use also 40x40x160 mm prisms. Moulds used to prepare the paste and mortar specimens have to conform to the standard EN 196-1 [7]. Special care should be taken in order to assure demanded tolerances of dimensions, flatness and perpendicularity of prisms.

For concrete, prism of size 100x100x200 mm should be cast in such a way that in standard 100x100x400 mm moulds metal prism 100x100x200 mm should be inserted. Special care should be taken in order to assure demanded tolerances of dimensions, flatness and perpendicularity of prisms. Also other equivalent measures to obtain the 100x100x200 mm concrete prisms can be applied. As an alternative, laboratory can use also 100x100x400 mm prisms or cylinders with a diameter of 150 mm and length of 300 mm. Moulds used to prepare the concrete specimens should conform to the standard EN 12390-1 [8].

If specimens of other dimensions are used, this should be clearly indicated in the Excel template provided.

Specimens should be wet cured in water at $20 \pm 2^\circ\text{C}$ or in chamber with controlled humidity at more than 95% and temperature of $20 \pm 2^\circ\text{C}$ as per the procedure described in EN 12390-2 [9].

9.5 Age of specimen for testing for hardened properties

Tests should be carried out at 24 h, 30 h, 36 h, 2 d, 3 d and 7 d. If possible, laboratories should perform the tests also at 12 h, 18 h, 28 d, 56 d and 91 d.

9.6 Compressive strength test

Compressive strength should be assessed by following procedure described in standard EN 196-1 [7] for hydrated cement paste and mortar, and procedure described in standard EN 12390-3 [10] for concrete. Test specimens should be the same as that used for determination of unloading modulus from compression.

9.7 Unloading modulus from compression – modified standard method (EN 12390-13 method B)

Elasticity is intrinsically related to recoverable mechanical energy. Therefore, it is conceptually important to determine elastic properties from unloading, during which energy is recovered out of the tested specimen.

According to the EN 12390-13 [11], the test specimens dimension d (diameter or width) shall be at least 3.5 times maximal aggregate size, D_{max} . The ratio between the specimen length, L and the dimension, d shall be in the range $2 \leq L/d \leq 4$.

Preferred specimen dimensions are 40x40x80 mm for hydrated cement paste and mortar, and 100x100x200 mm for concrete.

For hydrated cement paste and mortar, also prisms of size 40x40x160 mm can be used. For concrete, prisms of size 100x100x400 mm or cylinders of 150 mm diameter and 300 mm in height may be used.

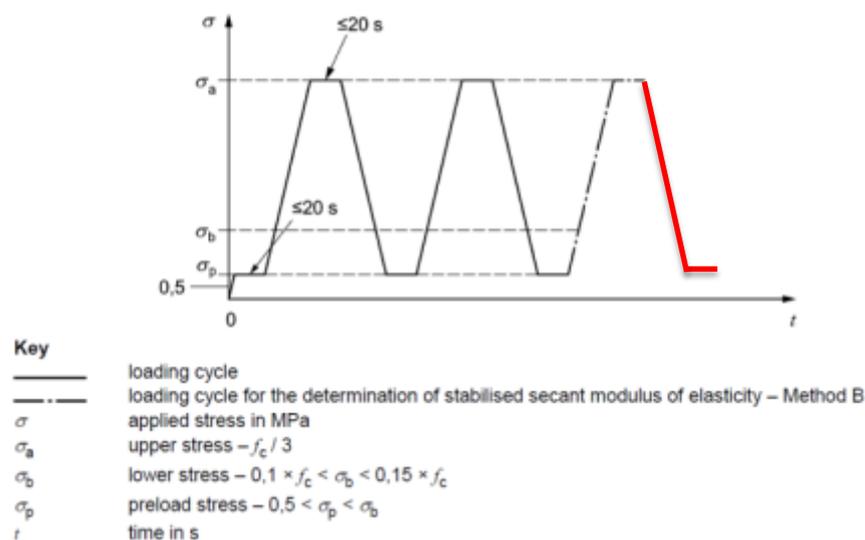


Figure 9.1 Cycle for the determination of stabilised secant modulus of elasticity (Method B) [11].

Other details needed:

Number of loading and unloading cycles - 3

Lower load level – preload stress: 10% of compressive strength

Upper load level – upper stress: 25% of compressive strength

Loading and unloading speed: 1.5 ± 0.125 MPa/s for paste and mortar and 0.6 ± 0.2 MPa/s for concrete

9.8 Splitting tensile strength test (EN 12390-6)

Splitting tensile strength test should be performed according to the procedure described in standard EN 12390-6 [12] for hydrated cement paste, mortar and concrete. Test specimens should be the same as that used for determination of unloading modulus from compression.

For the hydrated cement paste and mortar, packing strips should have different dimensions as those for the concrete (EN 12390-6 [12]). All other properties of the packing strips should be the same as for concrete.

Dimensions of the packing strips for the paste and mortar specimens should be: width (a) = (5 ± 0.5) mm; thickness (t) = (1 ± 0.2) mm and a length greater than the length of the line of contact of the test specimen. A packing strip shall be used only once.

9.9 Determination of ultrasonic pulse velocity (EN 12504-4)

Determination of ultrasonic pulse velocity should be performed by following procedure described in standard EN 12504-4 [13] for hydrated cement paste, mortar and concrete. The tests should be carried out on all specimens, prior to testing compressive strength, splitting tensile strength and unloading modulus of compression.

The least dimensions of the test specimen must exceed the wavelength of the ultrasonic vibrations. The wavelength of the vibrations equals the pulse velocity divided by the frequency of vibrations. For example, for a frequency of 54 kHz and a pulse velocity of 3500 m/s, the wavelength is $3500/54000 = 0.065$ m = 65 mm. Therefore, specimens of size >65mm shall be used in such instance.

References

- [1] EN 1015-3:2001 Methods of test for mortar for masonry - Part 3: Determination of consistence of fresh mortar (by flow table).
- [2] EN 12350-2:2009 Testing fresh concrete – Part 2: Slump-test
- [3] EN 1015-6:1999 Methods of test for mortar for masonry - Part 6: Determination of bulk density of fresh mortar
- [4] EN 12350-6:2009 Testing fresh concrete – Part 6: Density
- [5] EN 445:2007 Grout for prestressing tendons – Test methods
- [6] ASTM C 940 Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory.
- [7] EN 196-1:2016 Methods of testing cement – Part 1: Determination of strength
- [8] EN 12390-1:2012 Testing hardened concrete – Part 1: Shape, dimensions and other requirements for specimens and moulds
- [9] EN 12390-2:2009 Testing hardened concrete – Part 2: Making and curing specimens for strength tests
- [10] EN 12390-3:2009 Testing hardened concrete – Part 3: Compressive strength of test specimens

[11] EN 12390-13:2013 Testing hardened concrete – Part 13: Determination of secant modulus of elasticity in compression

[12] EN 12390-6:2009 Testing hardened concrete – Part 6: Tensile splitting strength of test specimens

[13] EN 12504-4:2004 Testing concrete - Part 4: Determination of ultrasonic pulse velocity

10 GP1.e: VOLUME STABILITY

Protocols for all tests that will be performed within GP1e are given in this chapter; excel files for result reporting were provided enclosed to this document. If participants intend to use methods which are not described here, they are kindly asked to coordinate this beforehand with the GP leader.

10.1 Chemical shrinkage

Le Chatelier (1900) has first shown that the hydration of cement causes shrinkage, because the volume of hydration products is lower than the volume of reactants. This is known as chemical shrinkage. As long as cement paste remains fluid, autogenous deformation is equal to chemical shrinkage. Then, as the stiffness of the paste increases, the hydration of cement causes cavitation and the creation of voids in the cement matrix. Autogenous deformation does not take into account this volume. From setting autogenous deformation is actually much lower than chemical shrinkage. The internal voids created in the cement paste cause self-desiccation shrinkage.

Measurements of the volume change of cement paste or mortar can be conducted through hydrostatic weighing. Immediately after mixing, a paste/mortar sample is poured in a container. The container is slowly filled with water in order to avoid diluting the sample in added water. Then the container is hung under a balance using a thin stainless steel wire and carefully immersed in the water bath at the chosen temperature (usually 20°C). The sample mass is approximately 100 g. The bath temperature is controlled at 20°C and recorded. The volume change results from a change in the buoyancy force and causes variations of the weight reading on the balance. Both the bath temperature and measured mass are continuously logged on the computer at 1-min intervals for a period exceeding 48 h.

Alternatively, the chemical shrinkage can be assessed through the capillary tube method, according to ASTM standard.

Table 9.1 Overview of proposed tests of chemical shrinkage

Test number	Mixture	Method	Duration
1	OCP	Hydrostatic weighing	48 hours or more
2	OCP	Capillary tube	48 hours or more
3	OM	Hydrostatic weighing	48 hours or more

References

Le Chatelier H., Sur le changement de volume qui accompagne le durcissement des ciments, Bulletin de la Société de l'encouragement pour l'industrie nationale, 5 (5) :54-57, 1900.

Powers T.C., Brownyard T.L., Studies of the physical properties of hardened Portland cement paste (nine parts), J. Am. Concr. Inst. 43 (Oct. 1946– April 1947), Bulletin 22, Research Laboratories of the Portland Cement Association, Chicago, 1948.

ASTM C 1608 – 06, Standard Test Method for Chemical Shrinkage of Hydraulic Cement Paste

10.2 Autogenous Shrinkage

The autogenous shrinkage is caused by self-desiccation as the hydration of cement goes on and the cement paste starts hardening.

Autogenous deformation can be defined as the bulk deformation of a closed, isothermal, cementitious material system not subjected to external forces, and self-desiccation as autogenous relative humidity change of a cementitious material system after setting, caused by chemical shrinkage (Lura, 2003; Jensen, 2001). In a homogeneous and isotropic cementitious system, autogenous shrinkage is theoretically an isotropic deformation, so volumetric measurements as well as linear methods may be used to assess autogenous deformation of early age concrete. Numerous test setups have been designed and experimental studies have been published. Jensen & Hansen (2001) have pointed out the need for round robin tests to conclude on the relevance of different measurement techniques.

Even if the main phenomena involved in autogenous shrinkage are known, the experimental assessment of autogenous shrinkage has not come to such a consensus between research teams because of many practical difficulties. Several artefacts actually have to be avoided to measure autogenous shrinkage as defined in the previous section.

The specimen must be perfectly sealed to avoid any external drying or water uptake. The temperature must be kept constant, which requires external control because the hydration of cement releases heat. The test rig should be designed to limit the friction with the specimen.

The temperature of paste, mortar, or concrete is controlled. For each time of measure, two temperatures are given: the external temperature and the temperature at the center of the specimen.

Displacements and temperatures are monitored from the placement of paste, mortar, or concrete in the mould, or as soon as possible, until 48 hours or more.

The initial concrete mixture was modified to design the Modified Ordinary Concrete (MOC). The paste volume was increased and the water-to-cement ratio was decreased to 0.40, so that MOC show significant autogenous shrinkage.

Four series of tests will be done on OC:

- a. under isothermal conditions, at 10, 20, and 40°C,
- b. under realistic temperature conditions, given by Vercors project (atmospheric conditions from Vercors will be provided later on).

Table 9.2 Overview of testing parameters for autogenous shrinkage

Test number	Mixture	Temperature
1	OC	10°C
2	OC	20°C
3	OC	40°C
4	OC	Realistic temperature conditions, given by Vercors project (atmospheric conditions from Vercors will be provided later on)
5	OM	20°C
6	MOC	20°C

References

ASTM C1698-09. Standard Test method for autogenous strain of cement paste and mortar. In: ASTM international, West Conshohocken (PA); 2010.

Darquennes A., Staquet S., Delplancke-Ogletree M.-P., Espion B., Effect of autogenous deformation on the cracking risk of slag cement concretes, *Cement & Concrete Composites* 33 (2011) 368–379.

Lura, P., Jensen, O.M., van Breugel, K. Autogenous shrinkage in high-performance cement paste: An evaluation of basic mechanisms *Cement and Concrete Research* 33 (2003) 223 – 232

Jensen, O.M., Hansen, P.F. Autogenous deformation and RH-change in perspective, *Cement and Concrete Research* 31 (2001) 1859 – 1865

10.3 Plastic shrinkage

Plastic shrinkage results from self-desiccation and external drying of paste, mortar, or concrete from fresh state to early hardening. It develops when the evaporation of water at the surface of concrete exceeds the water provided by bleeding (Wittmann, 1976). As thermal dilation occurs during the same time period, the temperature of the test rig is controlled. The temperature and relative humidity (RH) in air surrounding the test rig is controlled and monitored. If possible, the speed of air is measured near the concrete specimen.

The tests on concrete (OC) will be done under isothermal conditions at 20°C and 50% RH.

Plastic shrinkage measurements should be associated to the monitoring of weight loss. This can be done on the same specimen or another specimen of equal height.

As drying rate depends on the sample Volume to Surface exposed to drying ratio, a common depth of plastic shrinkage specimens and weight loss will be adopted by all the laboratories: 60 mm (Turcry, 2006, Schleibinger).

Table 9.3 Overview of testing parameters for plastic shrinkage

Test number	Mixture	Temperature	RH
1	OC	20°C	50%

References

Wittmann F.H., "On the Action of Capillary Pressure in Fresh Concrete," Cement and Concrete Research, vol 6, pp. 49-56, 1976.

Turcry Ph. and Loukili A., Evaluation of plastic shrinkage cracking of Self-Consolidating Concrete, ACI Materials Journal, July-August 2006, pp. 272-279.

Schleibinger Geräte Teubert u. Greim GmbH, The Schleibinger Shrinkage Drain, 2 p.

10.4 Restrained shrinkage

Restrained shrinkage testing procedures have been developed to assess shrinkage-induced stresses in controlled laboratory conditions. They also allow the assessment of creep and relaxation.

10.4.1 Dog-bone specimens

Temperature-Stress Testing Machine (TSTM) is used to generate restrained shrinkage at early-age. TSTM allows full restraint through the application of a compressive/tensile load on a dog-bone shaped concrete specimen to keep its length constant with a manual or an automatic system.

Restrained shrinkage starts generating stresses when the stiffness of the concrete specimen starts increasing, i.e. at setting time. Different methods can be used to assess initial and final setting times, thus different values can be obtained for a given mixture. In order to avoid discrepancies between the participating laboratories due to the assessment of setting time, the control of TSTM will start when concrete reaches maximal temperature, thus when it actually begins to shrink.

The TSTM system includes two temperature-controlled molds. In the first mold the specimen shrinks freely. This twin mold is used to assess the shrinkage ϵ_{sh} and thermal ϵ_{th} deformations. In the second mold shrinkage is restrained. According to the principle of independence of the deformation components, the total deformation is the sum of the elastic ϵ_e , creep ϵ_{cr} , shrinkage ϵ_{sh} , and thermal ϵ_{th} deformations. The combination of the two simultaneous measurements allows determining the creep and elastic deformations at early age, which are of major interest to understand the evolution of cracking sensitivity of concrete.

In the restrained shrinkage mold, a load is applied when the deformation reaches a threshold value in order to come back to total deformation equal to zero. Then the load is kept constant and shrinkage develops: another compensation cycle begins. At each cycle the tensile stress increases. The test is stopped when cracking of the specimen can be observed.

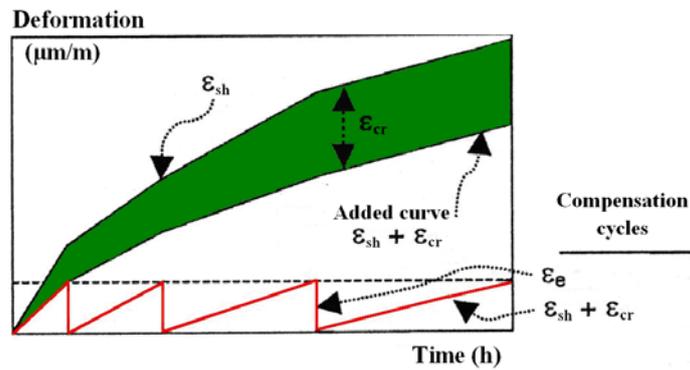


Figure 10.1 Assessment of free and restrained deformation during TSTM test (Kovler, 1994).

As the conditions of the tests are likely to vary from one laboratory to another (Staquet, 2012), the Table gives the parameters for RRT+ tests.

Table 9.4 Overview of testing parameters for dog-bone restrained shrinkage

Parameter of TSTM test	Value
Test start	Time when maximum temperature is reached
Deformation threshold	6 µm/m
Thermal control	yes

As OC mixture has relatively high effective water-to-cement ratio, its autogenous shrinkage deformation is low and relatively low stress levels are going to be generated in sealed isothermal conditions. In order to reach higher stress/strength ratios, additional tests will be done.

Table 9.5 Overview of testing parameters for autogenous shrinkage

Test number	Temperature control	Drying	Concrete mixture
1	Yes, 20°C	No	OC
2	Yes, 20°C	Yes, 50% RH	OC
3	Yes, 20°C	No	MOC
4	Yes, realistic temperature conditions, given by Vercors project (atmospheric conditions from Vercors will be provided later on)	No	OC

References

Kovler, K., 'Testing system for determining the mechanical behaviour of early age concrete under restrained and free uniaxial shrinkage', *Materials and Structures*, 27, (1994), pp. 324-330.

Staquet, S.; Delsaute, B.; Darquennes, A.; Espion, B. Testing Concrete since Setting Time under Free and Restraint Conditions with a Revisited TSTM. In *CONCRACK 3 – RILEM-JCI International Workshop on Crack Control of Mass Concrete and Related Issues Concerning Early-Age of Concrete Structures*, 15-16 March 2012, Paris, France; 2012; p 12.

10.4.2 Ring test

Ring tests can be used to define indicators of the cracking sensitivity of cement-based materials (Kovler&Bentur, 2009). They consist in casting a concrete ring around a steel ring. The shrinkage of concrete ring is restrained by the steel ring. Ring tests allow the determination of cracking time, tensile stresses and relaxation due to long-term restrained shrinkage. The monitoring of ring test includes:

- a. the deformation at inner radius of steel ring from the time of casting,
- b. the age of cracking.

In drying conditions, the concrete ring is exposed to drying 24 hours after casting. The temperature and relative humidity are monitored.

Different kinds of ring tests have been generated. Some have been standardized (AASHTO, ASTM C1581/C1581M-09a). The main differences are shape, dimensions, and sides exposed to drying. They influence the duration of the tests and produce different restraint levels as well as stress profiles in concrete specimens. If the outer side of concrete ring is exposed to drying (circumferential drying), the stress profile depends in the same time on the restraint due to the ring and on the restraint due to drying (Dean et al. 2006).

In RRT+ tests, only the top and bottom of the ring will be exposed to drying (Hossain&Weiss, 2006, Turcry et al., 2006). The outer side of the concrete ring will be sealed with a double layer of adhesive aluminum foil just after demolding to avoid circumferential drying. In order to allow the drying of bottom, the ring will be put on a grid.

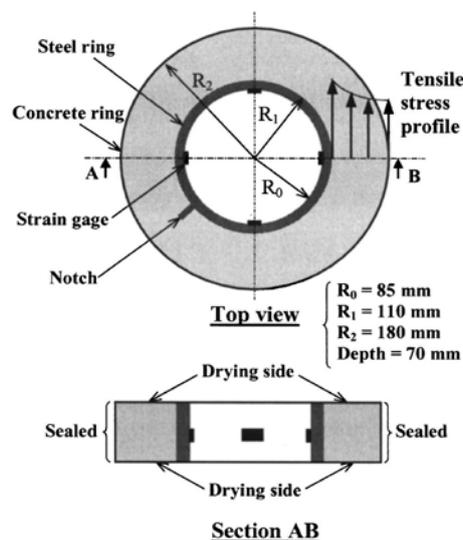


Figure 10.2 Schematic of ring specimen [Turcry et al., 2006]

Table 9.6 Overview of testing parameters for ring test restrained shrinkage

Test number	Age when exposed to drying	Drying	Concrete mixture
1	1 day	Yes, 50% RH	OC
2	28 days	Yes, 50% RH	OC
3	1 day	No	MOC

The ring test will be done on concrete from the same batch as concrete samples used to monitor free shrinkage of hardened concrete (see Long-term free shrinkage).

The monitoring of the test includes:

- Deformation at inner radius of steel ring from the time of casting ($\mu\text{m}/\text{m}$),
- Age of cracking (days)
- External temperature (T, °C)
- External relative humidity (RH, %)
- Time of formwork removal (h:min)
- Time of first measure (h:min)

References

Kovler K. and Bentur A., "Cracking Sensitivity of Normal- and High-Strength Concretes," *ACI Mater. J.*, no. 106, pp. 537–542, 2009.

ASTM, "ASTM C1581/C1581M-09a: Standard Test Method for Determining Age at Cracking and Induced Tensile Stress Characteristics of Mortar and Concrete under Restrained Shrinkage," 2004

Dean S., Moon J.-H., Rajabipour F., Pease B., and Weiss J., "Quantifying the Influence of Specimen Geometry on the Results of the Restrained Ring Test," *J. ASTM Int.*, vol. 3, no. 8, 2006.

Hossain A.B. and Weiss J., "The role of specimen geometry and boundary conditions on stress development and cracking in the restrained ring test," *Cem. Concr. Res.*, vol. 36, no. 1, pp. 189–199, Jan. 2006.

Turcry P., Loukili A., Haidar K., Pijaudier-Cabot G., and Belarbi A., "Cracking Tendency of Self-Compacting Concrete Subjected to Restrained Shrinkage: Experimental Study and Modeling," *J. Mater. Civ. Eng.*, 2006.

10.5 Long-term free shrinkage

Drying shrinkage generally show long characteristic times. Shrinkage time and potential magnitude depend on the ratio of volume to surface exposed to drying (called 'drying radius' or 'notional size of cross-section'), external relative humidity, and age when exposed to drying. Laboratory tests are generally done at relatively low values of notional size of cross-section, but the minimum dimension of the cross section must be higher than 5 times the maximum aggregate size.

Three sets of specimens (Figure 10.3) will be used for each test: one set of sealed specimens to assess autogenous shrinkage, one set of drying specimens for total shrinkage, and one set for weight loss. The sealing technique will be a double layer of adhesive-backed aluminum foil. Each specimen will be weighted at the beginning and at the end of shrinkage measurements. The duration of shrinkage and mass measurements will be 90 days.

Drying shrinkage will be assessed as the difference between autogenous shrinkage and total shrinkage.

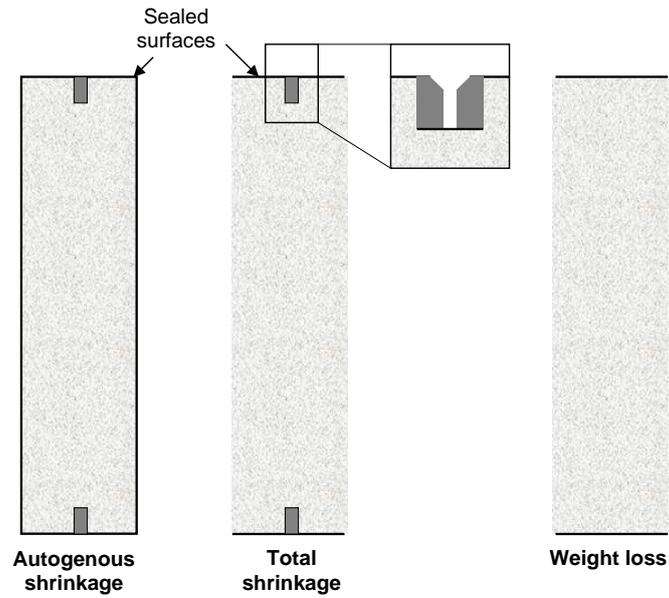


Figure 10.3 Specimens for the monitoring of autogenous, total shrinkage, and weight loss.

Table 9.7 Overview of testing parameters for long-term free shrinkage

Test number	Age when exposed to drying	Drying	Concrete mixture
1	1 day	50% RH	OC
2	2 days	50% RH	OC
3	7 days	50% RH	OC
4	28 days	50% RH	OC
5	1 day	No	MOC

11 GP1f: FRATURE PROPERTIES AND CRACKING

Protocols for all tests that will be performed within GP1f are given in this chapter. If participants are using method that is not described here, they should report about it directly to GP leader.

11.1 Fracture properties of mortar and concrete

The size of the beam specimens for mortar will be 160x40x40 mm.

The size of the beam specimens for concrete will be 400x100x100 mm.

The specimens should be beams with a central notch according to Fig. 1. The notch should always have a depth which is equal to half the beam depth ± 5 mm (For mortar specimens the notch should be 20 mm ± 5 mm. For concrete specimens the notch should be 50 mm ± 5 mm). It is recommended that it is sawn under wet conditions at least one day before the test. It may also be cast if a suitable saw is not available. If the notch is cast it is recommended to make the insert in the mould wedge-shaped with an angle of 10-15° in order to make it possible to loosen the mould without damaging the beam. If the notch is cast the beam must be handled very carefully during demoulding.

During the period of curing the room temperature shall be $20 \pm 2^\circ\text{C}$. The specimen should remain in the mould covered with PVC for at least 16 hours. After removal from the mould, the specimen should be stored acc. EN 12390-2 until less than 24 hours before testing. Tests will be performed on specimens at ages 7 and 28 days.

11.2 Determination of the fracture toughness by means of three-point bend tests on notched beams

For cementitious composite materials, such as mortar or concrete, the fracture toughness is determined using linear elastic fracture mechanics (LEFM) considerations.

The P-CMOD response is obtained from notched beams, subjected to the center-point loading configuration shown in Fig. 1.

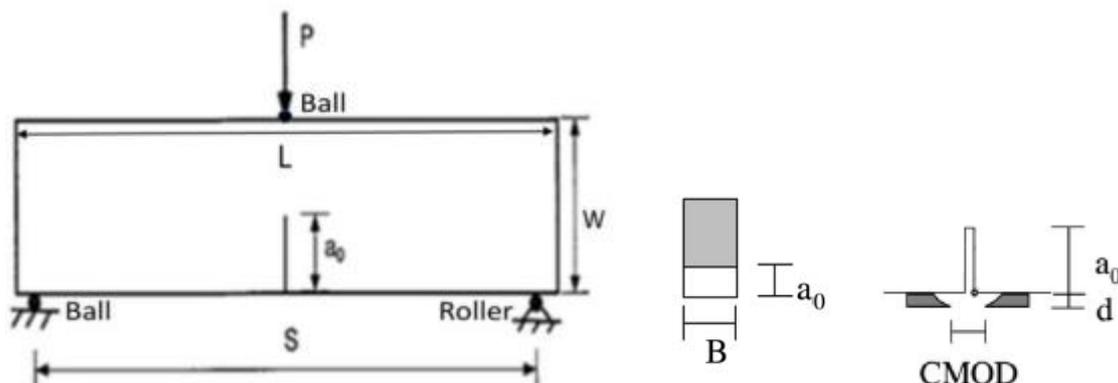


Figure 11.1 Three-point bend test configuration for notched beam specimen (S is the length, B is the thickness and W is the width of the specimen).

The tests are performed under deflection control, where this parameter is measured using a clip gage mounted on knife-edges. The thickness of the knife-edges, d , is taken into account in the calculations.

For this geometry, the stress intensity factor (K_I) is given by

$$K_I = \frac{1,5 P S \sqrt{\pi a}}{B W^2} f(\alpha)$$

where P is the applied load, a is the crack length and $\alpha = a / W$ is the relative crack length (with $S=3W$ and $\alpha_0=a_0/W=0.55$ for concrete and 0.625 for mortars). The geometry dependent function, $f(\alpha)$ is determined using two-dimensional plane stress analysis and is given by

$$f(\alpha) = f_1(\alpha) + f_2(\alpha)$$

Where

$$f_1(\alpha) = \frac{0,68 - 0,744\alpha}{1 - 2,155\alpha + 1,161\alpha^2}$$

$$f_2(\alpha) = 0,36 - 2,088\alpha + 4,611\alpha^2 - 6,499\alpha^3 + 4,232\alpha^4 \quad \text{when } 0,05 \leq \alpha \leq 0,80$$

$$f_2(\alpha) = 0 \quad \text{when } 0,80 \leq \alpha \leq 0,90$$

Also, in a similar way,

$$CMOD = \frac{6 P S a}{E B W^2} g(\alpha)$$

where the dimensionless geometry-dependent function, $g(\alpha)$, is given by

$$g(\alpha) = g_1(\alpha) + g_2(\alpha)$$

Where

$$g_1(\alpha) = \frac{0,995 - 0,574\alpha}{1 - 2,06\alpha + 1,063\alpha^2}$$

$$g_2(\alpha) = 0,342 - 1,761\alpha + 2,098\alpha^2 - 1,437\alpha^3 + 1,455\alpha^4 \quad \text{when } 0,05 \leq \alpha \leq 0,80$$

$$g_2(\alpha) = 0 \quad \text{when } 0,80 \leq \alpha \leq 0,90$$

As it is evident from Fig. 1, the CMOD is not measured exactly at the notch mouth but at a distance d , which must be taken into account for determining the real value of CMOD from that measured in the test (denoted as $CMOD^{Measured}$).

Therefore, $CMOD = k_d CMOD^{Measured}$

The conversion factor k_d can be determined, for $1 \text{ mm} \leq d \leq 6 \text{ mm}$, from

$$k_d(d, \alpha) = \frac{k_1 + k_2\alpha}{1 + k_3\alpha + k_4\alpha^2}$$

The coefficients k_1 , k_2 , k_3 and k_4 used to compute k_d for the CMOD correction are given in Table 10.1 for different knife-edge thicknesses, d .

Table 10.1 Coefficients used to compute k_d for the CMOD correction

d (mm)	k_1	k_2	k_3	k_4
1	0.0050	275.1	275.9	0.0399
2	0.1508	162.5	163.6	0.0118
3	0.1037	104.1	105.1	0.01631
4	0.0777	76.2	77.2	0.0137
5	0.0623	60.2	61.1	0.0117
6	0.0521	49.7	50.6	0.0106

For large α ($> 0,4$) the correction is insignificant, hence for specimens with relative notch lengths in the order of 0,4, no correction needs to be applied. Moreover, for thin knife-edges ($d < 2$ mm) the correction is always negligible (for calculation see test result sheet).

KIc is to be calculated at Pmax, assuming $a = a_0$.

11.2.1 Description of test procedure for fracture toughness measurement

The Crack Mouth Opening Displacement (CMOD) will be monitored during the test performed with controlled deflection in a closed-loop testing machine. The deflection of the specimen should be increased at a constant rate of 0.2 mm/min until the specified final deflection is reached. A plot of CMOD versus applied load will be produced.

11.3 Determination of the fracture energy by means of three-point bend tests on notched beams

The fracture energy is defined as the amount of energy necessary to create one unit area of a crack. The area of a crack is defined as the projected area on a plane parallel to the main crack direction. This test method is not recommended for fiber-reinforced concrete.

The fracture energy is determined from the equation,

$$G_F = \frac{W_o + m g \delta_o}{A_{lig}} \quad [\text{N/m (J/m}^2\text{)}]$$

Where, W_o (Nm), is the area under the load-deformation curve shown in Figure 10.2;

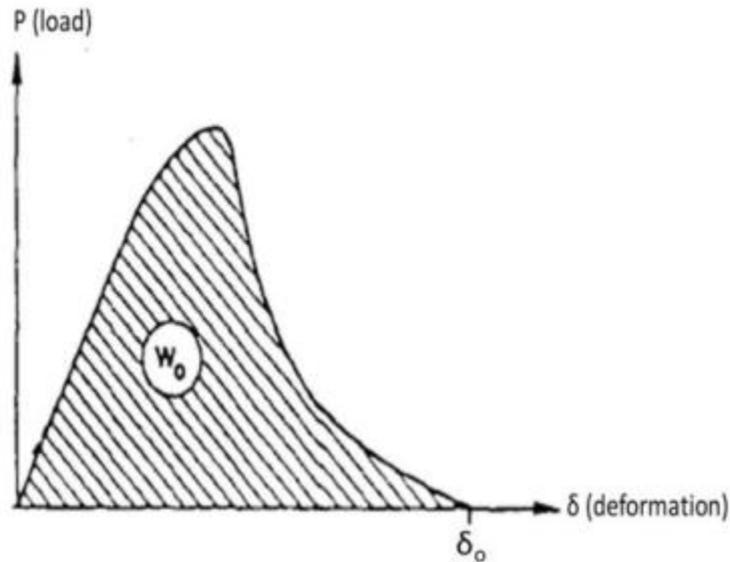


Figure 11.2 Load-deformation curve

$$m = m_1 + 2m_2 \quad (\text{kg}) ;$$

m_1 = weight of the beam between the supports, calculated as the beam weight multiplied by S/L (Fig. 1). The length L of the beam as well as the span S must be measured with an accuracy of at least 1 mm;

m_2 = weight of the part of the loading device touching the beam which is not attached to the testing machine, but follows the beam until fracture;

g = acceleration due to gravity ($9,81 \text{ m/s}^2$);

δ_0 = deformation of the beam at fracture (m), as shown in Figure 10.2;

A_{lig} = area of the ligament (m^2), defined as the projection of the fracture zone on a plane perpendicular to the beam axis.

11.3.1 Test procedure for fracture energy measurement

The test is performed with a constant rate of deflection of 0.2 mm/min . The deformation of the center of the beam and the corresponding load are registered until the beam is completely separated into two halves.

11.4 Determination of bending strength and fracture toughness of fiber-reinforced concrete

This part of the RRT+ will be defined in more details directly with the interested participants. In general, ordinary concrete mix (OC) will be used with the addition of fibers, provided only to participant who are interested in this part of the testing The final mix for fiber reinforced concrete will be defined depending on the interest of participants.

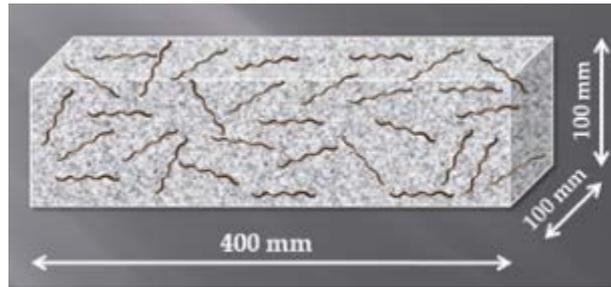


Figure 11.3 Dimensions of fiber-reinforced concrete specimen

Tests will be performed on specimens at ages 7 and 28 days.

11.4.1 Description of test procedure for bending strength and fracture toughness measurement

Four-point bending test will be performed under deflection control in a prismatic specimen of dimensions 400x100x100 mm, as shown in Fig. 3.

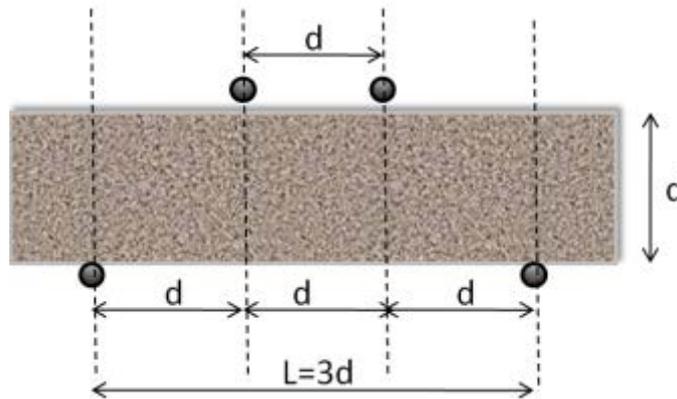


Figure 11.4 Four-point bend test configuration

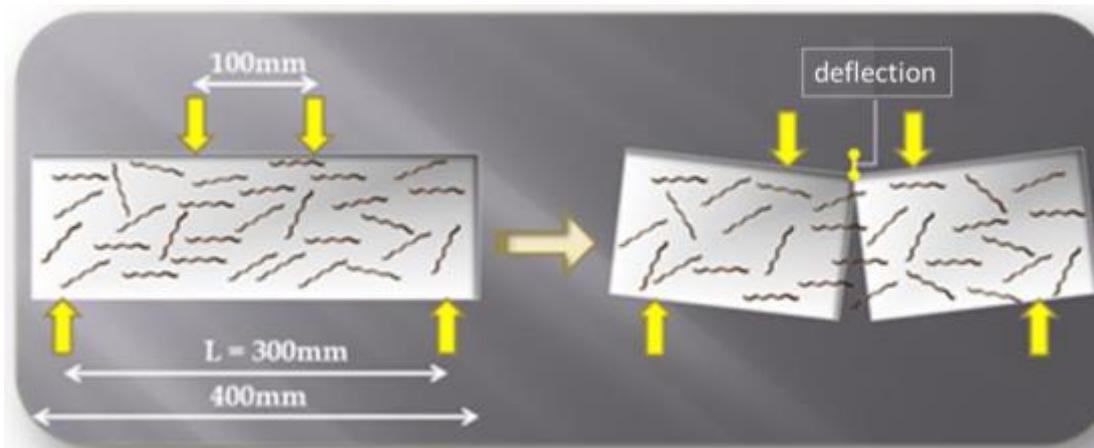


Figure 11.5 Specimen failure under bending test

An acceptable arrangement for measuring deflection during four-point bending testing employs a rectangular jig, which surrounds the specimen and is clamped to it at mid-depth directly over the supports (Fig. 6). An electronic displacement transducer or similar digital or

analog device mounted on the jig at mid-span measures deflection through contact with appropriate brackets attached to the specimen.

The test should be conducted at a displacement speed of 0.02 mm/min.



Figure 11.6 Measuring deflection during four-point bending test

11.4.2 Calculation of bending strength and fracture toughness

The bending strength (in MPa) is given by

$$f = \frac{PL}{bd^2}$$

Where, d=100mm, L=300mm, P (N) is the peak load, and b=100mm is the specimen width.

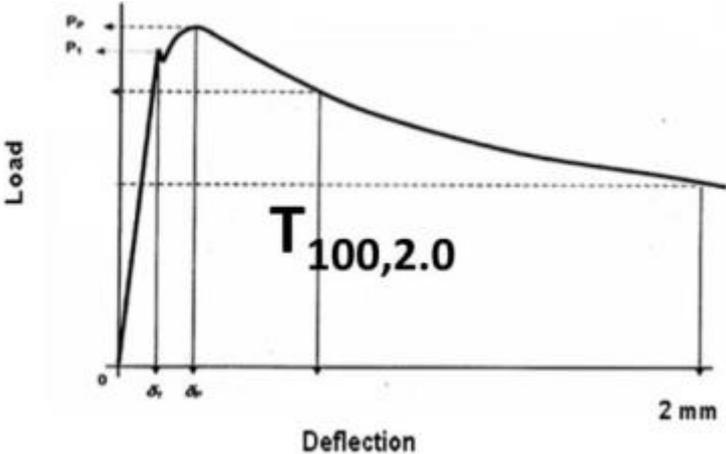


Figure 11.7 Load-Deflection curve during four-point bending test

The fracture toughness index $T_{100,2,0}$ to be determined is defined as the area under the load-deflection curve up to a deflection of 2mm, as shown in Figure 4.

11.5 Acoustic emission (AE) monitoring of fracture and cracking

Acoustic emission is described as transient elastic waves generated by the release of energy within a material. Microscopic fracture in concrete takes place with the release of stored strain energy during nucleation of microcracks and generates elastic waves. These waves are referred to as AE waves, which propagate inside a material and are detected by an AE sensor.

AE energy is a part of the fracture energy and has been correlated in cases of bending and compression. To monitor the AE energy during loading, the RILEM recommendations shall be followed [DOI 10.1617/s11527-010-9640-6, DOI 10.1617/s11527-010-9639-z, DOI 10.1617/s11527-010-9638-0]. Sensors can be either resonant (suggested 60 kHz or 150 kHz) or broadband. At least two sensors should be placed at the bottom either side of the notch, with suitable coupling (grease) as in Fig. 5. In case more acquisition channels are available, sensors can be placed to other sides. Magnetic clamps are preferred to hold the sensors during test, but simple tape works as well. It is proposed to enable location (even linear in case of two sensors) so that random data/noise can be better filtered out. The distance between sensors should not be more than 100-150 mm in order to adequately record the AE events occurring in the fracture zone in between.

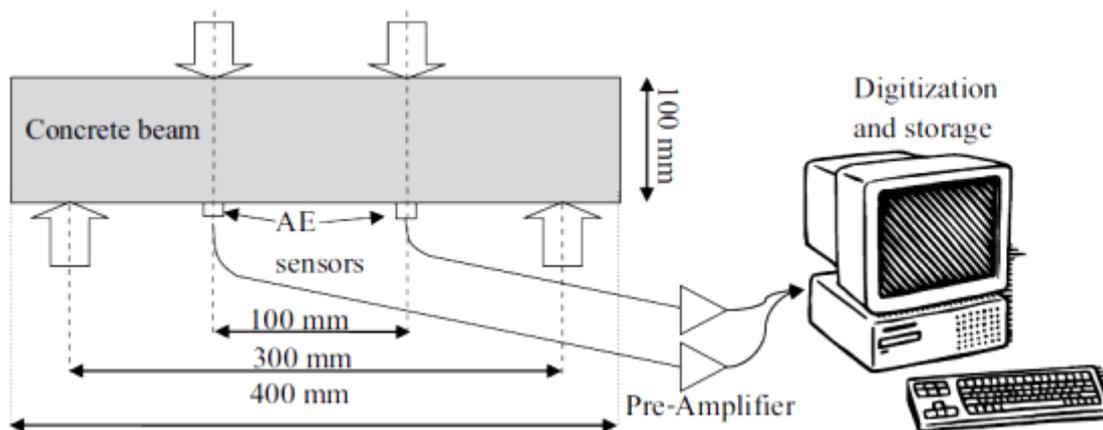


Figure 11.8 Schematic representation of AE setup during bending of concrete beams.

Sensitivity of AE measurement system shall be conducted routinely by employing the standard source of mechanical pencil lead break (0.5 mm diameter and 3 mm length). Variation of signals in dB within the different sensors shall be less than 3%. The threshold level for counting shall be set as low as possible, depending on the level of the noise before start of the test (usually 40 dB is adequate). Amplification gain should be 40 dB or more.

An indicative diagram of the setup is shown in Figure 10.8.

The following basic definitions are important:

Hit: Indication that a given AE channel has detected and processed one AE transient signal.

Event: A group of hits received from the same source at different sensors.

The basic parameters that should be recorded are (see Figure 10.9):

- 1) Threshold: A pre-set voltage that should be overpassed in order for acquisition to start (this is set and recorded at the start of the test).
- 2) Peak amplitude: Maximum voltage of the waveform.
- 3) AE energy: Measured Area under the Rectified Signal Envelope, MARSE.
- 4) Rise time: Delay between first thr. crossing and maximum peak.
- 5) Duration: Delay between first and last thr. crossing and last one.
- 6) External parameters: load, strain and so forth are preferably recorded in the system each moment a "hit" is received.

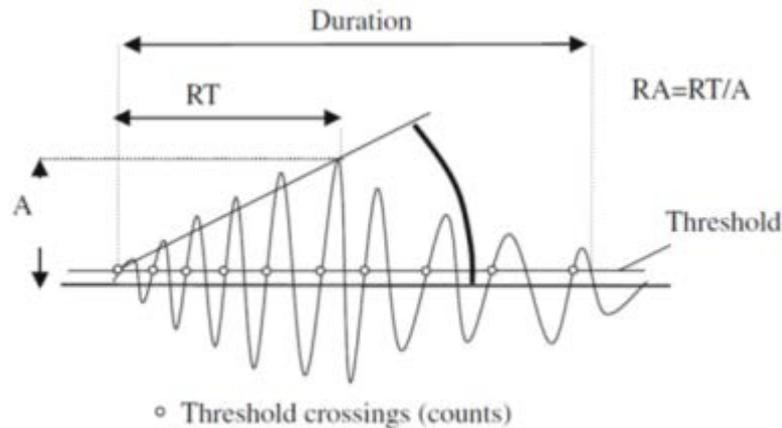


Figure 11.9 Basic parameters of an AE waveform.

The AE recording shall be started with load application and terminated when the loading stops in order to avoid possibility of recording irrelevant data.

In the analysis of data the cumulative number of recordings (hits/events) and the cumulative energy vs. time or load provide indications of the rate of fracture (see typical graph in Figure 10.10 a). Correlations shall be searched between the cumulative energy and the fracture toughness. The recording of the first event(s) reveals the load at which fracturing occurs.

In materials where a succession of fracture modes (e.g. fibre reinforced concrete, external reinforcing patches) is expected, of particular importance are the parameters average frequency (AF) measured as the number of threshold crossings over the duration and RA value which is the RT over the peak amplitude. A frequency decrease and RA increase indicates a shift from tensile mode to shear.

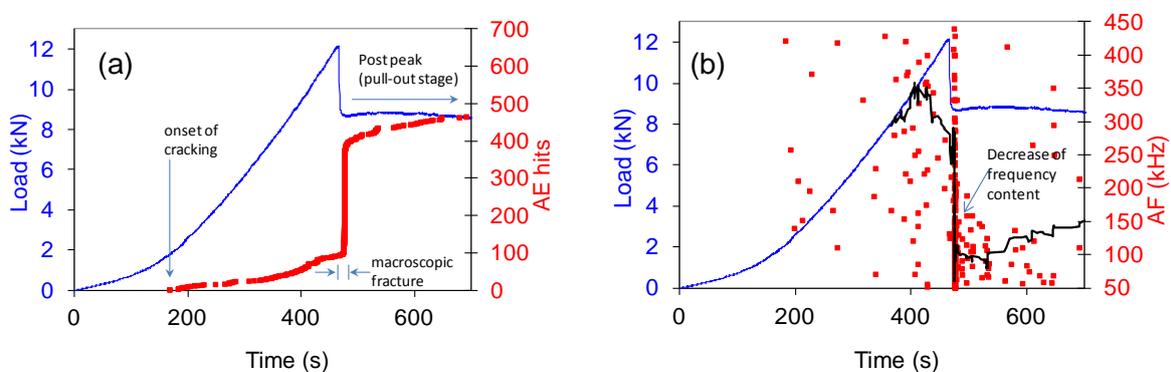


Figure 11.10 Load history and (a) cumulative AE activity, (b) AF of individual hits. The black line shows the moving average of recent 50 hits.