RILEM Technical Committee TDC

Compilation of Test Methods to Determine Durability of Concrete

A Critical Review

Prof. Dr.-Ing. Christoph Gehlen
Dipl.-Ing. Amir Rahimi
Technische Universität München

December 2011

1. Test methods to determine durability of concrete

1.1 Frost damage

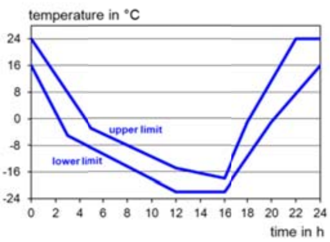
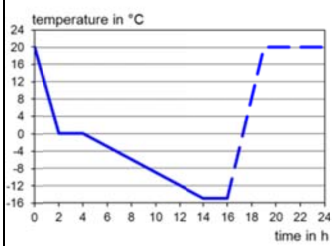
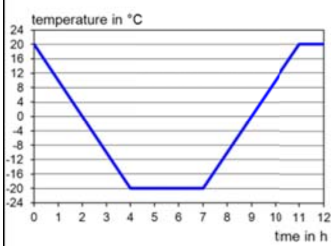
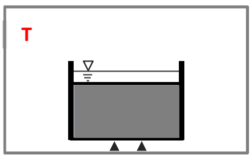
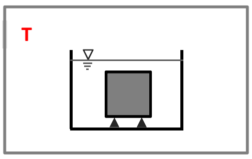
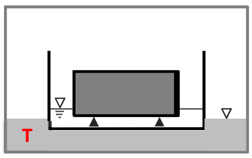
<u>CEN/TS 12390-9:</u> Testing hardened concrete - Part 9: Freeze-thaw resistance – Scaling (pre-standard), 2006.

This pre-standard demonstrates the following three test methods to determine the freeze-thaw resistance of concrete:

1. Slab-Test according to: SS 137244 – "Concrete testing – Hardened Concrete – Frost Resistance" 2005 (Swedish Standard).
2. Cube-Test according to: Bunke, N.: Prüfung von Beton – Empfehlungen und Hinweise als Ergänzung zu DIN 1048. Berlin: Beuth. – In: Schriftenreihe des Deutschen Ausschusses für Stahlbeton (1991), No. 422, pp.12-15.
3. CF- / CDF-Test according to: RILEM TC 117-FDC Recommendation: CDF Test, Test Method for the Freeze-Thaw-Resistance of concrete with sodium chloride solution, 1996.

The test methods contain three steps, (1) curing and preparing the specimens, (2) pre-saturation and (3) freeze-thaw cycles. The test liquid simulates a deicing agent and contains 3 % by weight of NaCl and 97 % by weight of (demineralized) water in case of the test of the freeze-thaw and deicing salt resistance and demineralized water to test the freeze-thaw resistance of concrete respectively. Scaling of the specimens is measured after a well defined number of freeze-thaw cycles and leads to an estimate of the resistance of the tested concrete against frost damage.
The test methods differ however in their procedures and conditions. Table 1.1-1 summarizes these three methods.

Table 1.1-1: lab test methods for freeze-thaw resistance of concrete according to CEN/TS 12390-9

–	Slab-Test	Cube-Test	CF- / CDF-Test[1)]
	1	2	3
curing	7 d water storage, 21 d 20 °C / 65 % RH	7 d water storage, 20 d 20 °C / 65 % RH	7 d water storage, 21 d 20 °C / 65 % RH
specimen preparation	cutting, gluing a rubber surround	–	lateral sealing by epoxy resin or aluminium foil with butyl rubber
specimen and test surface	cut surfaces of four 150 x 150 x 50 mm³ samples	formed surfaces of four cubes with edge length of 100 mm	with teflon formed surfaces of at least five150 x 140 x 50 mm³ samples, total surface > 0.08 m²
application of test liquid	one-sided, 3 mm on the top side	all sides, submerged	One-sided, capillary suction of 5 mm
Pre-saturation	3 d with 3 mm demineralized water on the top side	1 d submerged in test liquid	7 d capillary suction of 5 mm test liquid
duration of each freeze-thaw cycle	24 h	24 h	12 h
temperature curve			
temperature regulation	air-cooling, temperature recording in the middle of the test liquid above one specimen	air-cooling, temperature recording in the middle of a reference specimen	liquid-cooling (under the specimen container), continuous temperature recording of the liquid
scaling measurement	brushing the loose particles from the specimen surface, filtering the test liquid, drying	brushing the loose particles from the specimen surfaces, filtering the test liquid, drying	gathering the loose particles from the specimen surface by ultrasonic bath, filtering the test liquid, drying
freeze-thaw duration	56 d	56 d (100 d /Bunke/[3)])	14 d (CDF) 28 d (CF)
recommended acceptance criteria[2)]	scaling < 1,000 g/m² after 56 freeze-thaw cycles	scaling < 3 % by weight after 56 freeze-thaw cycles	scaling < 1,500 g/m² after 28 freeze-thaw cycles
schematic			

1) CF: **C**apillary suction and **F**reeze-thaw, CDF: **C**apillary suction of **D**eicing solution and **F**reeze-thaw
2) only for test with 3 % NaCl
3) German guideline DAfStb no. 422, Bunke N.: Prüfung von Beton-Empfehlungen und **H**inweise als Ergänzung zu DIN 1048 (1991).

<u>CIF-Test:</u> RILEM TC 176-IDC Recommendation: CIF Test, Test method of frost resistance of concrete (Capillary suction, internal damage and freeze-thaw test) reference method and alternative methods A and B, 2004.

Besides the above test methods which estimate the freeze-thaw resistance of concrete by means of the scaling rate, in CIF-Test the internal damage of specimen is analyzed. By testing the freeze-thaw and deicing salt resistance usually the scaling is the dominant factor for an evaluation. But in freeze-thaw loading without deicing salt the internal damage of specimen dominates. The CIF-test is based upon the CDF-Test. It differs from the CDF-Test only in test liquid (demineralized water instead of 3 % NaCl). In CIF-Test also the scaling of the specimens is measured but it treated as a secondary criterion. The reference method to determine the internal damage is measuring of the “relative dynamic modulus of elasticity (ultrasonic transit time)” in specimens and its changes during the freeze-thaw loading. The concrete is defined as damaged when the relative dynamic modulus of elasticity transgresses below the 80 % level. Alternative methods to determine the internal damage are “measurement of fundamental transverse frequency” and “measurement of length changes” which could possess a higher precision in comparison with the reference method but are usually more complex.
Table 1.1-2 provides an overview of the acceptance criteria of CIF-, CDF-, and CF-Test according to the corresponding standards and guidelines. It must be pointed out that these criteria are for application of concrete in sever environmental conditions, namely exposure classes XF4 (freeze-thaw and deicing attack) and XF3 (freeze-thaw attack) according to EN 206-1.

<u>Table 1.1-2:</u> acceptance criteria

standard / guideline	CIF-Test		CDF-Test	
	RDM 1)	Scaling 2)	Scaling 1)	RDM 2)
	1	2	3	4
RILEM TC 176-IDC	80 % after n FTC 3)	–	–	–
RILEM TC 117-FDC	–	–	≤ 1,500 g/m² after 28 FTC	–
CEN TS 12390-9	–	–	≤ 1,500 g/m² after 28 FTC 4)	–
BAW-Merkblatt „Frostprüfung“ 5)	≥ 75 % after 28 FTC	≤ 1,000 g/m² after 28 FTC	≤ 1,500 g/m² after 28 FTC	≥ 75 % after 28 FTC

RDM: relative dynamic modulus of elasticity
FTC: freeze-thaw cycle
1) decisive acceptance criterion
2) secondary acceptance criterion
3) the number of FTC is to be declared
4) for CF-Test the scaling rate after 56 FTC is decisive, but no limit value is defined
5) Federal Waterways Engineering and Research Institute (Germany), Code of practice „Frost Resistance Testing of Concrete”, 2004

1.2 Carbonation

RILEM CPC-18: Measurement of hardened concrete carbonation depth, 1996

This method consists of determining the depth of the carbonated layer on the surface of hardened concrete by means a solution of 1 % phenolphthalein in 70 % ethyl alcohol as indicator. Suitable specimens are concrete prisms with a cross-section of 100 x 100 mm² that can be split into lengths of 50 mm for each date of testing. For mortar are prisms of 40 x 40 x 160 mm³ recommended from which a slice of 20 mm is split off at each date of testing. A temperature of 20 °C, a relative humidity of 65 % and a roughly 0.03 % CO_2 concentration define the climatic conditions of storage. The following dates of testing are recommended: 28, 90, 180 days; 1, 2, 4, (8, 16, …) years after the first exposure to CO_2.

Accelerated Carbonation Test: DARTS: Durable and Reliable Tunnel Structures: Data European Commission, Growths 2000, Contract G1RD-CT-2000-00476, Project GrD1-25633, 2004

Within the DARTS project an accelerated carbonation test (ACC) is developed. The specimens are concrete prisms of 100 x 100 x 500 mm³ which are demoulded 1 day after casting und stored for 6 days under water. The next 21 day of dry storage carried out at 20 °C and 65 % RH. At the age of 28 days the prisms are stored in a carbonation chamber with a 2.0 % CO_2 concentration, 20 °C and 65 % RH for 28 days. At the age of 56 days the prisms are crushed and the carbonation depth is measured with phenolphthalein solution. The inverse carbonation resistance of tested concrete as a characteristic material parameter is evaluated as fallows:

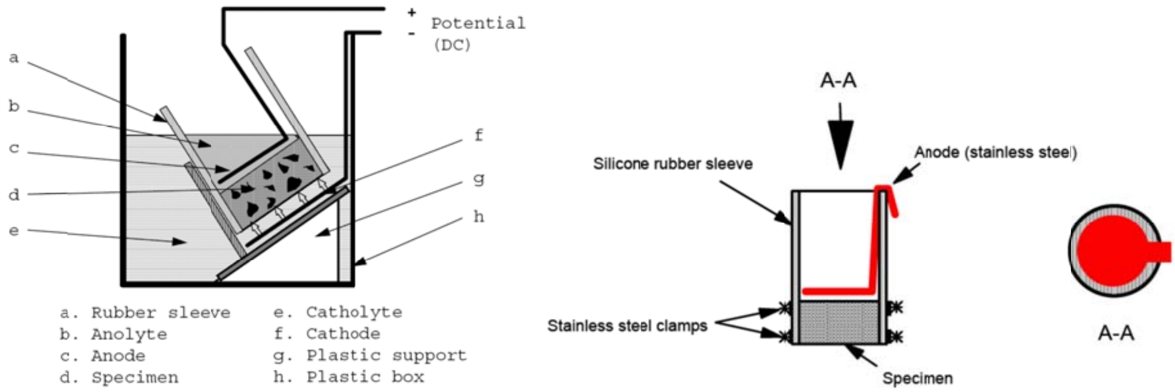
$$R_{ACC}^{-1} = \left(\frac{X_c}{\tau}\right)^2$$

with:
R_{ACC}^{-1}: inverse carbonation resistance in [m²/s/kgCO$_2$/m³]
X_c: carbonation depth in [m]
τ: time constant in [(s/kgCO$_2$/m³)$^{0.5}$]

1.3 Chloride penetration

<u>NT BUILD 492:</u> Chloride migration coefficient from non-steady-state migration experiments

This guideline presents a rapid chloride migration (RCM) test. An external electrical potential is applied axially across the specimen and forces the chloride ions outside to migrate into the specimen. After a certain test duration, the specimen is axially split and a silver nitrate solution is sprayed on to one of the freshly split sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation, after which the chloride migration coefficient can be calculated from this penetration depth.
At least three concrete cylinders of 100 mm diameter and 50 mm thickness are used. The specimens are stored in water up to the test. The specimens are fit in a rubber sleeve to prevent water absorption of lateral sides and placed on the cathode holder in the catholyte reservoir. The sleeves above the specimens are filled with anolyte solution (0.3 M NaOH) and the anodes are immersed in the solution. Cathodes and anodes are connected to a power supply. The test duration is up to the density of specimen and the applied voltage, and varies between 4 hours and ca. 1 week.
Figure 1.3-1 shows the experiment setup.



<u>Figure 1.3-1:</u> Schematic experiment setup of RCM-test /NT BUILD 492/

1.4 Sulfate attack

Currently there is no standardized laboratory test method in Europe to estimate the sulfate resistance of concrete. Table 1.4-1 provides an overview of common test methods in Germany. Generally mortar specimens are used which undergo a rapid sulfate attack. The accelerated execution is achieved by increasing the sulfate concentration on the one hand, and debilitating the specimens by producing small samples with a high surface-volume ratio and a high water-cement ratio on the other hand. Changes in the physical properties of specimens are measured. The test methods estimate rather the sulfate resistance of used cement / mortar and not the sulfate durability of concrete, because the real concrete microstructure isn't simulated by the tests, and it differs from the structure of mortar substantially.

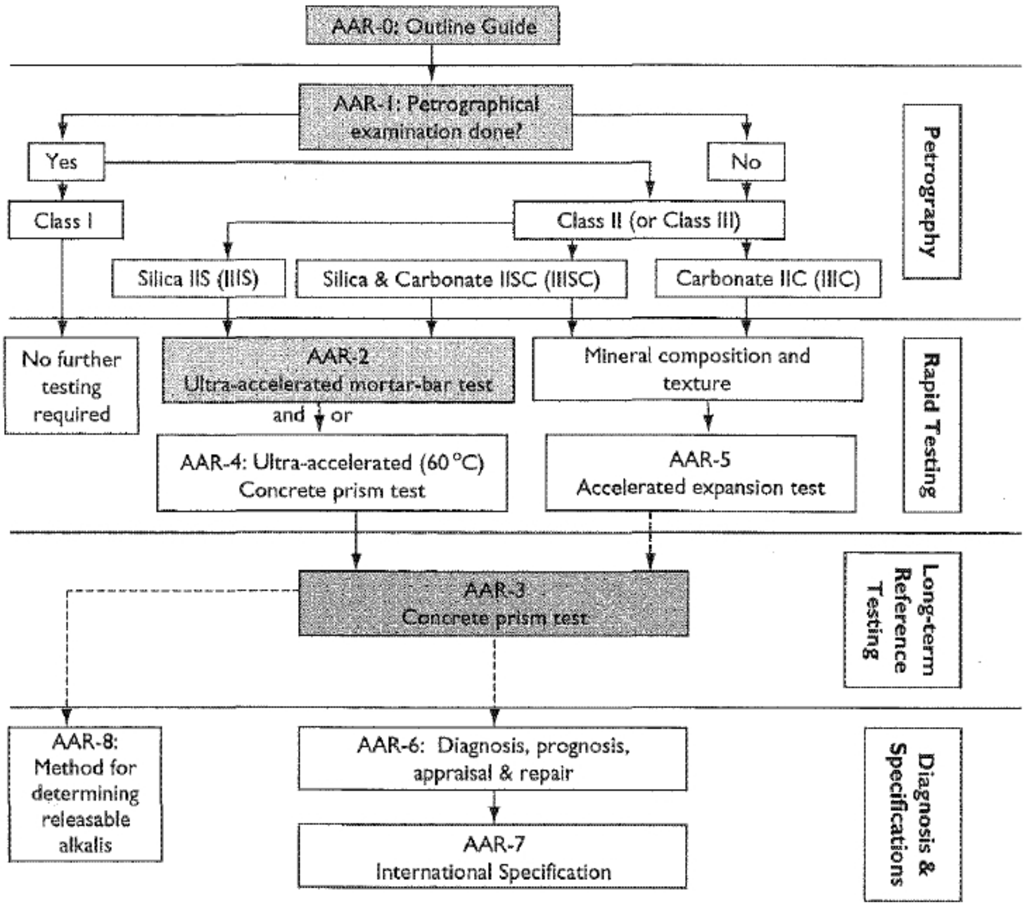
Table 1.4-1: common lab test methods for sulfate resistance of cement / mortar / concrete in Germany

–	Wittekindt	CEN	SVA	Kochsteineger	MNS
	1	2	3	4	5
specimen	mortar flat prisms 1 x 4 16 cm³	mortar flat prisms 2 x 2 16 cm³	mortar flat prisms 1 x 4 16 cm³	mortar small prisms 1 x 1 x 16 cm³	fine concrete prisms 4 x 4 x 16 cm³ or cylinder Ø50 / L150 mm
water-cement ratio	0.6	0.5	0.5	0.6	0.5
curing	2 d wet storage + 5 d in water	1 d wet storage + 27 d in water	2 d wet storage + 12 d in saturated $Ca(OH)_2$ solution	1 d wet storage + 20 d in water	7 d water storage, 21 d 20 °C / 65 % RH + saturating with sulfate solution at 150 mbar vacuum
sulphate exposure	56 d in $Na_2So_4^-$ solution	up to 52 weeks in $Na_2So_4^-$ solution	91 d in $Na_2So_4^-$ solution	56 d in $Na_2So_4^-$ solution	56 d in $Na_2So_4^-$ solution
sulfate concentration [g/l]	14.4	16.0	29.8	29.8	33.8
temperature [°C]	20	20	20	20	8
renewal of the test liquid	monthly	monthly	monthly	daily titration with H_2SO_4	monthly
recommended acceptance criteria	relative elongation < 0.5 mm/m	not defined	relative elongation < 0.5 mm/m	relative bending tensile strength > 0.7	relative tensile strength ≥ 0.7

1.5 Alkali-aggregate reaction

RILEM TC 191-ARP / TC 106: Alkali-reactivity and prevention – Assessment, specification and diagnosis of alkali-reactivity

Figure 1.5-1 shows the approach of RILEM TC 191-ARP (and its predecessor TC 106) to assessment the alkali-aggregate-reactivity (AAR) of aggregates. It must be pointed out that this approach is only to detect the alkali-reactivity potential of aggregates and it's not a concrete durability test method. It is to prevent an alkali-aggregate reaction damage of concrete by using non alkali-reactive aggregates or binders with a low alkali content.



<u>Figure 1.5-1:</u> RILEM TC 106 / TC 191-ARP, integrated AAR assessment scheme

Only the principles AAR-1, -2, -3 and -5 have been published. These Principles are as fallows:

<u>AAR-1:</u> *Petrography*

Petrographic analysis is the first step in the assessment of the potential alkali-reactivity of concrete aggregates. As a result an aggregate should be classified as one of the following:
– very unlikely to be alkali-reactive – Class I
– alkali-reactivity uncertain – Class II
– very likely to be alkali-reactive – Class III

AAR-2: *Ultra-accelerated mortar-bar test*

In this method prisms are moulded from mortar prepared with the aggregate to be tested. The prisms are demoulded after 24 ± 2 hours and their initial length measured. The specimens are then placed in water, transferred to an oven at 80 ± 2 °C for 24 hours, removed from the water and the length measured immediately before the temperature has dropped substantially (zero reading). The specimens are immediately placed in containers with a 1 M NaOH solution already at 80 ± 2 °C, the containers sealed and placed in an oven at 80 ± 2 °C (subsequent 14 days). Length measurements are taken periodically.
Figure 1.5-2 shows a brief outline of the test procedure.

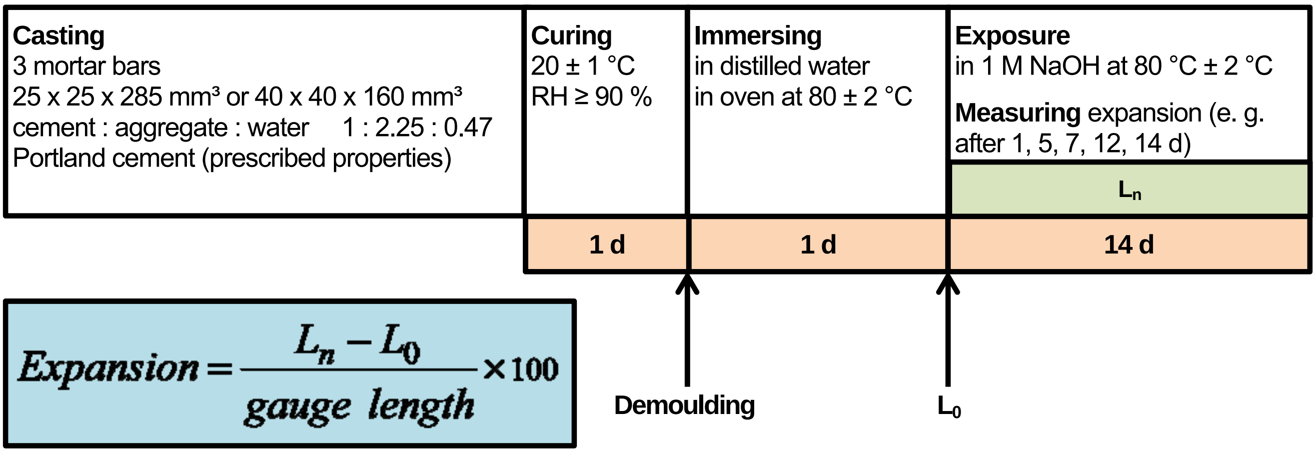


Figure 1.5-2: Procedure of AAR-2 of RILEM TC 106

A critical limit of 0.10 % expansion is suggested in order to different between nonreactive and reactive aggregates.

AAR-3: *concrete prism test*

Concrete test prisms are prepared from the aggregate combination under test and are stored in warm (40 °C), humid conditions for 12 months to promote any alkali-silica reaction. Measurements are made at periodic intervals to determine whether any expansion has occurred. To promote further any potential for reaction, the prisms are made with relatively high cement content and a high alkali cement such that the alkali level in the concrete is 5.5 kg Na_2O equivalent per m³ of concrete. Na_2O is added to the mix when necessary to enhance the alkali level. In order to identify the effect of specific aggregate combinations and any pessimistic effects, there are options to test the coarse and fine test aggregates together or either in combination with a non-reactive material. Figure 1.5-3 shows a brief outline of the test procedure.

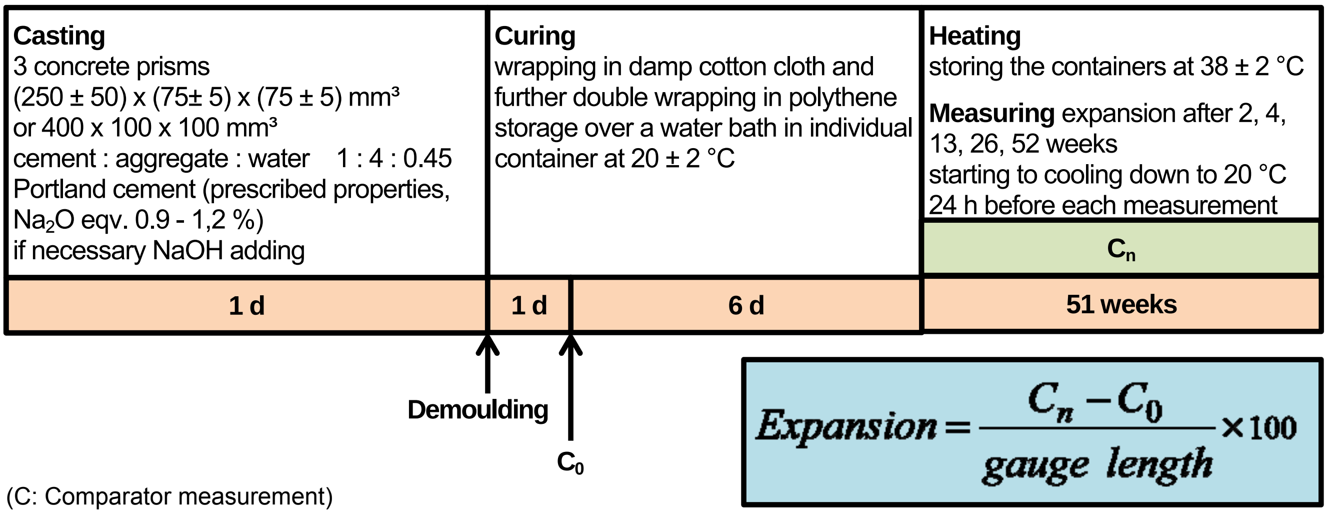


Figure 1.5-3: Procedure of AAR-3 of RILEM TC 106

A critical limit of 0.05 % expansion is suggested in order to different between nonreactive and reactive aggregates.

AAR-4: *Ultra-accelerated concrete prism test*

This method is similar to AAR-3: concrete test prisms are prepared from the aggregate combination under test and are stored in higher temperature (60 °C) than in AAR-3 and the duration of storage is only 20 weeks (instead of 51 weeks in AAR-3).
A critical limit of 0.03 % expansion is suggested in order to different between nonreactive and reactive aggregates.

AAR-5: *Accelerated expansion test*

This method is similar to AAR-2: bars moulded from a mix containing the aggregate to be tested are demoulded after 24 hours, heated up in water to 80 °C during another 24 hours, then immersed in 1 M NaOH solution at 80 °C and the expansions are measured. However, since some carbonate aggregates produce deleterious expansions only if used in a larger particle size, with AAR-5 4 / 8 mm aggregate is used instead of 0 / 4 mm aggregate and 40 x 40 x 160 mm bars are used instead of 25 x 25 x 285 mm bars.

DAfStb-Guideline (Germany): Preventive methods against the damaging alkali-reaction in concrete, 2007

This German guideline divides aggregates into two groups. The first group consists of aggregates containing opal and flint. These aggregates have to undergo a physical analysis. The second group consists of other alkali-sensitive crushed aggregates. The test methods are as follows:

Rapid test (reference method): *the same as AAR-2 “ultra-accelerated mortar-bar test”*

As alternative method the mortar-bars can be casting with a higher Na_2O content by adding NaOH. The storage is carried out in containers over a water bath with a high temperature of 70 °C. Expansion is measured for 28 days after casting. A critical limit of 0.15 %

expansion is suggested in order to different between nonreactive and reactive aggregates.

Concrete prism test in fog chamber at 40 °C:

Concrete prisms of 100 x 100 x 500 mm³ to measure the expansion and a cube of 300 mm³ to supervision of cracking are stored in a fog chamber at 40 °C in age of 1 day for a duration of 9 months. The measurements are carried out immediately without cooling down. A critical limit of 0.06 % expansion is suggested in order to different between nonreactive and reactive aggregates.

1.6 Hydrolysis and leaching

Currently there is no standardized laboratory test method in Germany to estimate the leaching behavior of cementitious materials. Generally there are three kinds of laboratory test methods: batch method, column method and monolithic method. Figure 1.6-1 shows a sketch of these methods.

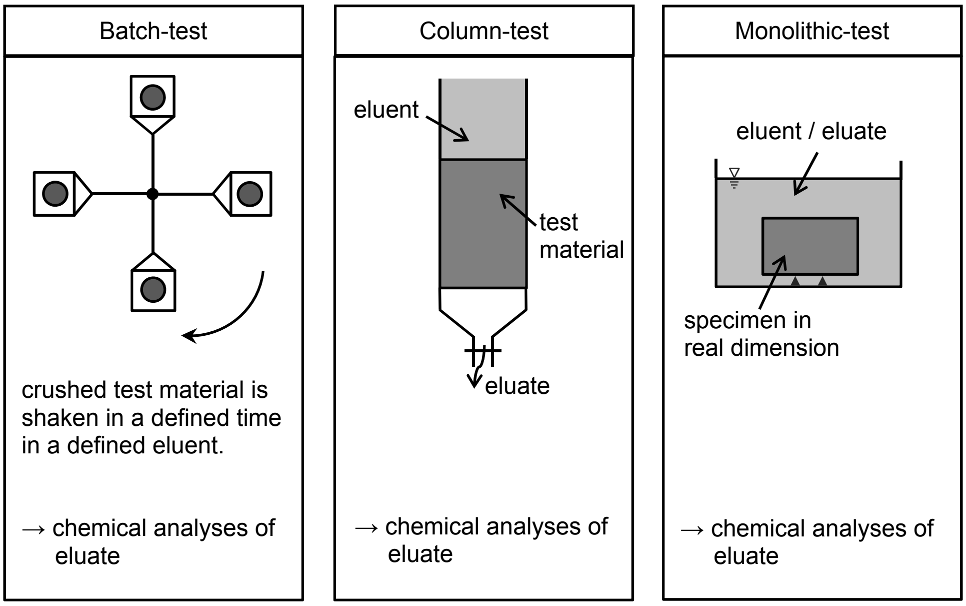


Figure 1.6-1: Schematic layout of basic test methods

1.7 Other actions

2. Critical appraisal of existing test methods

3. Proposal for test methods to study durability under combined actions