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Program validacije standardizirane preskusne metode za preskušanje penetracije kloridov in karbonatizacije

Validation testing program on chloride penetration and carbonation standardized test methods

Validierungsprogramm für genormte Prüfverfahren zur Bestimmung der Chlorideindringung und der Karbonatisierung

Programme d'essai de validation des méthodes d'essai normalisées relatives à la pénétration des chlorures et à la carbonatation

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Validation testing program on chloride penetration and carbonation standardized test methods

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Validierungsprogramm für genormte Prüfverfahren zur Bestimmung der Chlorideindringung und der Karbonatisierung

This Technical Report was approved by CEN on 24 May 2022. It has been drawn up by the Technical Committee CEN/TC 104.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

European foreword

This document (CEN/TR 17172:2022) has been prepared by Technical Committee CEN/TC 104 "Concrete and related products", the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document reports the data obtained in the Validation Testing Program (VTP) on chloride penetration and carbonation organized by CEN/TC 51/WG 12 starting from 2009 as from document CEN/TC 51/WG 12 – Doc. N 229/2009, where the preparation of specimens, the collection of results and the statistical analysis were performed by the Institute of Construction Sciences "Eduardo Torroja" of the CSIC of Spain, IETcc–CSIC, under the managing activities of Prof. Carmen Andrade.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

Introduction

The procedure for the determination of chloride penetration is described in EN 12390-11:2015, *"Testing hardened concrete — Determination of the chloride resistance of concrete — Unidirectional diffusion*" and it has been published by CEN. The method is based on natural diffusion; a concentration profile after 90 days of contact with the chloride solution is used to fit Fick's law in order to calculate the chloride surface concentration, C_S and the non-steady-state chloride diffusion coefficient, D_{nss}. The method specifies three different modes of contact of the salt solution with one face of the specimen, immersion (DCL1), ponding (DCL2) and inversion (DCL3).

CEN/TC 51/WG 12 has also produced two methods addressed to the determination of the carbonation resistance of the concrete, the first one refers to natural condition and has been published as CEN/TS 12390-10, "*Testing hardened concrete — Part 10: Determination of the relative carbonation resistance of the concrete*", the second one, referring to accelerated condition, has been prepared by CEN/TC 51/WG 12/TG 5, but it has been disapproved by National Members at Formal Vote CEN TCA[1)].

The upgrading to EN standard of the aforesaid documents should require as first step the evaluation of robustness and precision data.

Having in mind these needs, CEN/TC 51/WG 12 organized a "Validation Testing Program (VTP) on chloride penetration and carbonation" for the preliminary evaluation of the robustness and the precision data of the test methods.

For the scope of the present work as robustness is intended the sensitivity of the test method to a composition change of concrete that are expected to produce an appreciable change in related performance.

1) FprCEN/TS 12390-12:2010, *Testing hardened concrete — Part 12: Determination of the potential carbonation resistance of concrete: Accelerated carbonation method.*

1 Scope

The objective of the report consists in testing concrete mixes for particular aggressive environments with the test methods being standardized by TC51/WG12 on chloride penetration and carbonation in order to verify their robustness and coherence.

NOTE See EN 206 for additional information.

2 Normative References

There are no normative references in this document.

3 Terms and Definitions

No terms and definitions are listed in this document.

4 Concretes and specimens

For the VTP four concrete mixes were designed considering the limiting values indicated in Table F.1 of EN 206 and the scope of assessing the robustness of the methods.

Three composition parameters (cement type, w/c ratio and cement content) were suitably chosen.

The following cement type and class were chosen: CEM II/A-LL 42.5 R and CEM II/B-V 32.5 R.

The w/c ratio was intentionally changed to substantially affect the concrete performances.

Two cement contents were used, the first one (300 kg/m^3) for carbonation, the second one (350 kg/m^3) for chloride penetration.

Aggregate "round shaped" of siliceous nature and with a maximum diameter of 14 mm was used. In Table 1 the composition of concrete mixes is shown.

The use of superplasticizer admixture was modulated, where necessary, to obtain a slump class S3 (100 mm – 150 mm). Table 1 gives the nominal proportions of the mixes used.

Table 1 — Proportions and cement types of the mixes prepared

	CARBONATION				CHLORIDE			
	MIX 1	**MIX 2**	**MIX 3**	**MIX 4**	**MIX 5**	**MIX 6**	**MIX 7**	**MIX 8**
Cement type	CEM II/A-LL 42.5R		CEM II/B-V 32.5R		CEM II/A-LL 42.5R		CEM II/B-V 32.5R	
Cement amount (kg/m^3)	295	296	296	300	345	351	349	357
Water (l/m^3)	144	173	144	175	137	173	138	176
w/c ratio	0,49	0,58	0,49	0,58	0,4	0,49	0,4	0,49
Gravel (kg/m^3)	1 049	1 011	1 054	1 025	1 005	977	1 019	993
Sand (kg/m^3)	857	827	861	838	816	793	827	806
Superplasticizer (% cement weight)	0,60	0,20	0,50	0,15	0,79	0,23	0,57	0,10

	CARBONATION				CHLORIDE			
	MIX 1	MIX 2	MIX 3	MIX 4	MIX 5	MIX 6	MIX 7	MIX 8
Cement type	CEM II/A-LL 42.5R		CEM II/B-V 32.5R		CEM II/A-LL 42.5R		CEM II/B-V 32.5R	
Density (kg/dm^3)	2 260	2 265	2 294	2 285	2 273	2 313	2 330	2 330
Air content (%)	5,75	4,9	5,15	3,75	5,7	3,8	4,4	2,3
Slump (cm)	11	10	12	10	10	10	13,5	10

For each laboratory and test method 2 cubes (150 mm) were produced.

In Annex A a summary of the experimental details of the preparation of the specimens and their submission is described. It is worth noting that the specimen preparation was centralized in one laboratory (IETcc) in order to have a better homogeneity.

5 Participating laboratories

Fourteen laboratories from different European countries participating in the VTP are indicated in Table 2 with the tests methods they performed.

Table 2 — List of laboratories participating in the exercise with the test types they perform

COUNTRY	LABORATORY	NATURAL CARBONATION		ACCELERATED CARBONATION	DIFFUSION CHLORIDE PENETRATION		
		NCA1	NCA2	ACA	DCL1	DCL2	DCL3
France	Lafarge	X			X		
France	LRPC						X
Spain	University of Alicante				X	X	
Spain	IETcc	X	X	X	X	X	X
Belgium	CRIC			X	X		
The Netherlands	KEMA				X	X	X
The Netherlands	TNO	X	X	X			
The Netherlands	SGS INTRON	X	X	X	X		
United Kingdom	Dundee University			X	X	X	X
Sweden	CBI			X	X		
Germany	FIZ/VDZ			X	X	X	X
Italy	Polytechnic Milan	X		X	X	X	X
Poland	ISCMOIB/OMMB	X	X				

6 Testing program

The tests performed are summarized in Table 3.

Table 3 — Test methods and standards used in the program

Test methods	Reference	Methodology	Label
Accelerated carbonation	[a]	At 4 % of CO_2	ACA
Natural carbonation	CEN/TS 12390-10[b]	Natural exposure	NCA1
		Climatic chamber [c]	NCA2
Chloride diffusion	EN 12390-11	Immersion	DCL1
		Ponding	DCL2
		Inversion	DCL3

[a] The draft test method used for this research received a negative vote and the Work Item was withdrawn; however it was published by BSI as BS 1881-210. A new accelerated test is being prepared for standardization with the main change being a carbon dioxide concentration of 3 %. The robustness and precision determined by the research is expected to be the same as for the test method under development.

[b] The procedure described in CEN/TS 12390-10:2007 differs from the method followed in the present report. CEN/TS 12390-10 prescribes that test specimens shall be tested when they have reached the 50 % of the compressive strength of the reference concrete. In the present report there was no reference concrete as the purpose was not to determine the relative carbonation but the robustness of the testing procedures.

[c] At 350 ppm of carbon dioxide. An EN to replace CEN/TS 12390-10 is under development and the most significant change is to increase the carbon dioxide concentration in the chamber test to 400 ppm. This change is unlikely to impact the robustness and precision determined by this research.

7 Statistical analysis

The statistical treatment has been made following ISO 5725-2, "*Determination of the accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*".

According to this standard, the parameters to be calculated are the mean value (m), the repeatability standard deviation (s_r), the reproducibility standard deviation (s_R). In addition, the limiting values of the repeatability and reproducibility, r and R, have been also calculated.

The basic model is simple of type I applied to a single experiment. It has been applied in three steps:

1) A critical exam of all data in order to identify and to treat any anomalous value or irregularities which could prevent the correct application of ISO 5725-2.

2) Once the outliers' results are identified and discarded the repeatability, r and r (%) and reproducibility, R and R (%) of the remaining results (excluding the outliers) is calculated.

3) Definitive calculation of the mean and precision values, including the relations between the mean and the precision for each level (ISO 5725-2).

8 Results

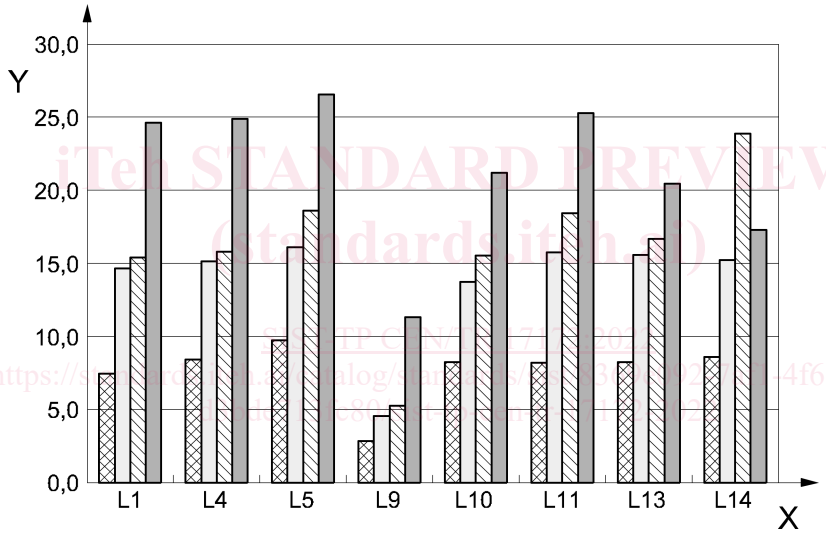
8.1 Accelerated carbonation test prEN 12390-12

The individual results of carbonation depth are presented in Figure 1. The testing procedure ranks concretes with CEM II/A-LL 42.5 R as more resistant than concrete with CEM II/B-V 32.5 R for the same w/c ratios.

Also it can be noticed that the method is sensitive to changes in the w/c ratio for the concrete with the same cement as it was evidenced by an increase of the carbonation depth with the increase of w/c ratio.

It is not possible to draft any conclusion on the effect of cement type because the strength class of cement is different. Consequently, different cement lead to different concrete strength and porosity and they are subjected to the exposure with different degree of porosity evolution in concrete.

Lab 9 has been considered outlier because its results were much lower compared to the other laboratories. It was identified later that its chamber did not control the relative humidity properly and this parameter was higher than prescribed in the standard. Then the results of the Lab 9 in this test have not been used in the calculations. Table 4 shows the results of the statistical analysis.



Key

X laboratory number

Y dk average (mm)

Figure 1 — Individual results averaged for each mix of accelerated carbonation depth

Table 4 — Summary of mean values of carbonation depth obtained for ACA method

Accelerated carbonation			**Mix 1**	**Mix 2**	**Mix 3**	**Mix 4**
	Average values (mm)		**8,38**	**15,17**	**16,79**	**23,85**
Standard deviation	Repeatability	s_r (mm)	0,76	0,56	0,60	0,90
	Reproducibility	s_R (mm)	0,86	0,87	1,41	2,30
Variation coef.	**Repeatability**	$\boldsymbol{CV_r}$ **(%)**	**9,12**	**3,69**	**3,56**	**3,76**

Accelerated carbonation			Mix 1	Mix 2	Mix 3	Mix 4
	Reproducibility	**CV_R (%)**	**10,27**	**5,75**	**8,37**	**9,66**
Limit values	Repeatability	*r* (mm)	2,14	1,57	1,67	2,51
	Reproducibility	*R* (mm)	2,41	2,44	3,93	6,45
Limit values/average	Repeatability	*r* (%)	25,53	10,33	9,96	10,53
	Reproducibility	*R* (%)	28,77	16,10	23,42	27,04

8.2 Remarks from laboratories participating to the testing procedure CEN/TS 12390-12 (ACA)

The main aspect that should be mentioned is that 2 of 8 laboratories did not consider the indication of protecting 2 parallel faces of specimen (point 6.2) and then it can be recommended that the description of the testing procedure regarding measurements points and the number of faces should be improved.

9 Natural carbonation CEN/TS 12390-10 (NCA)

9.1 Natural carbonation

The main aim of this test was to check its comparative ability and the robustness of the two alternative testing procedures. The "normalization" described in the chapter 6 of CEN/TS 12390-10 was not in the scope of the VTP.

Two results were considered:

— *dk* (mm) the penetration depth;

— k_c (mm/year$^{0.5}$) the carbonation rate.

9.2 Penetration depth *dk*

The test was performed by 6 laboratories, although not all performed both tests (in climatic chamber and outdoors), number of specimens and testing times. The durations on each test taken by each laboratory were not exactly those of the standard, but the data collected at different ages by each lab (see Table 5 and 6) were close enough to have relevance: all the tests were performed by taking some data before or at 1 year and other, at around two years. Due to these different testing ages, the direct comparison of the penetration depths using *dk* in mm is made with not all laboratories, however all data were used in the case of the rate of carbonation in mm/year$^{0.5}$ because each *dk* was divided by the exact testing period.

For the sake of appraising the environmental conditions, Annex C gives the average temperature and relative humidity of the atmospheres where the participating laboratories are located. Their relative differences are presented also in the psychrometric diagram.

Table 5 — Days of exposure by each laboratory in a climatic chamber or outdoor sheltered from rain

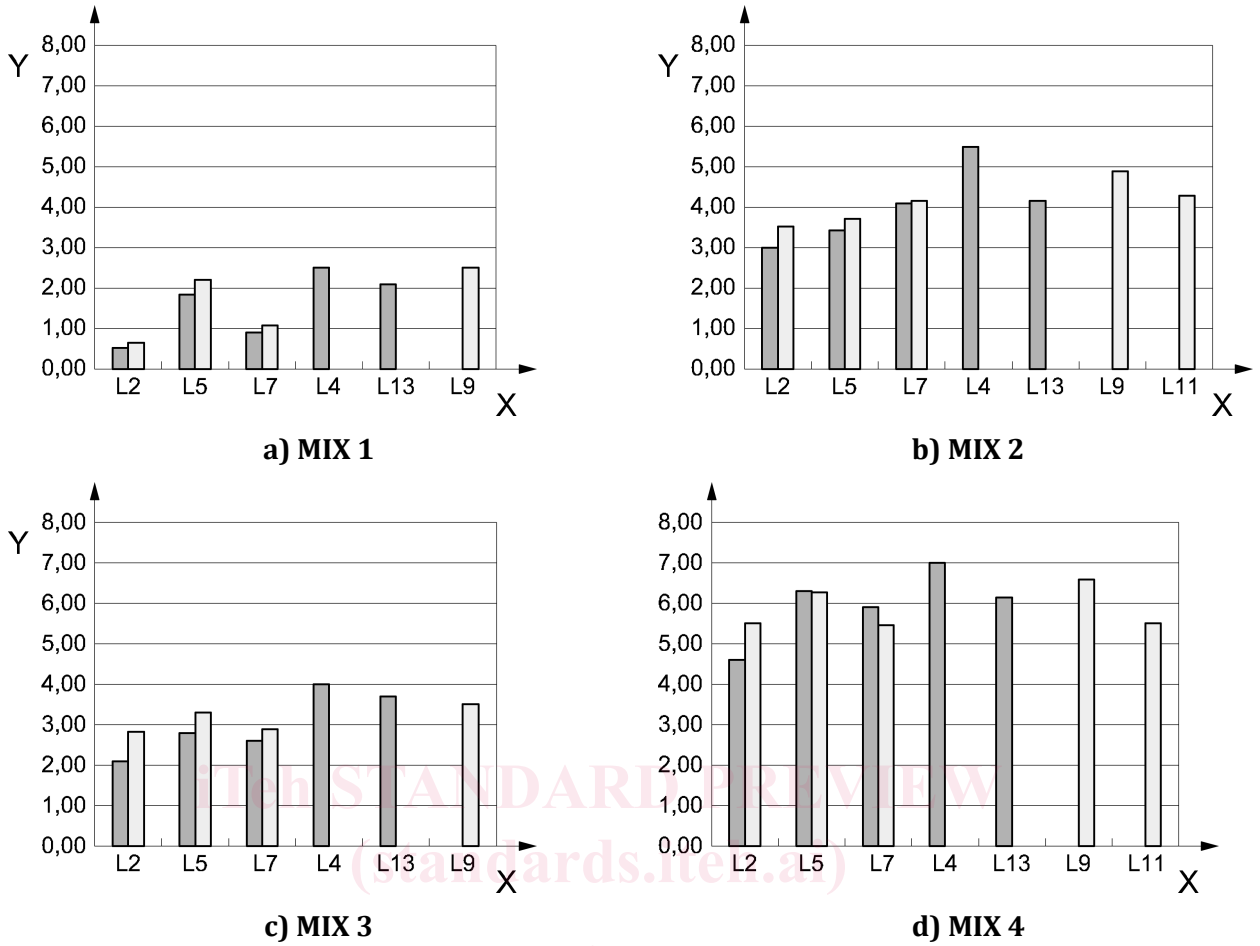
LABORATORIES	TESTING TIMES (days)
L2	365, 730
L5	375, 801
L7	365

LABORATORIES	TESTING TIMES (days)
L9	190, 730
L11	365, 730, 886
L13	365

Table 6 — Number of laboratory results averaged given in Figures 4 and 7

NUMBER OF LABORATORIES FOR EACH AVERAGED VALUE Natural carbonation				
	NCA1		NCA2	
	Year 1	Year 2	Year 1	Year 2
MIX 1	5	4	4	2
MIX 2	5	5	4	3
MIX 3	5	4	4	2
MIX 4	5	5	4	3

For averaging the values, Table 6 shows the number of laboratories per mix considered giving valid values. Figures 2 and 3 show averaged "valid" results of the penetration depth per mix obtained by each laboratory.



a) MIX 1

b) MIX 2

c) MIX 3

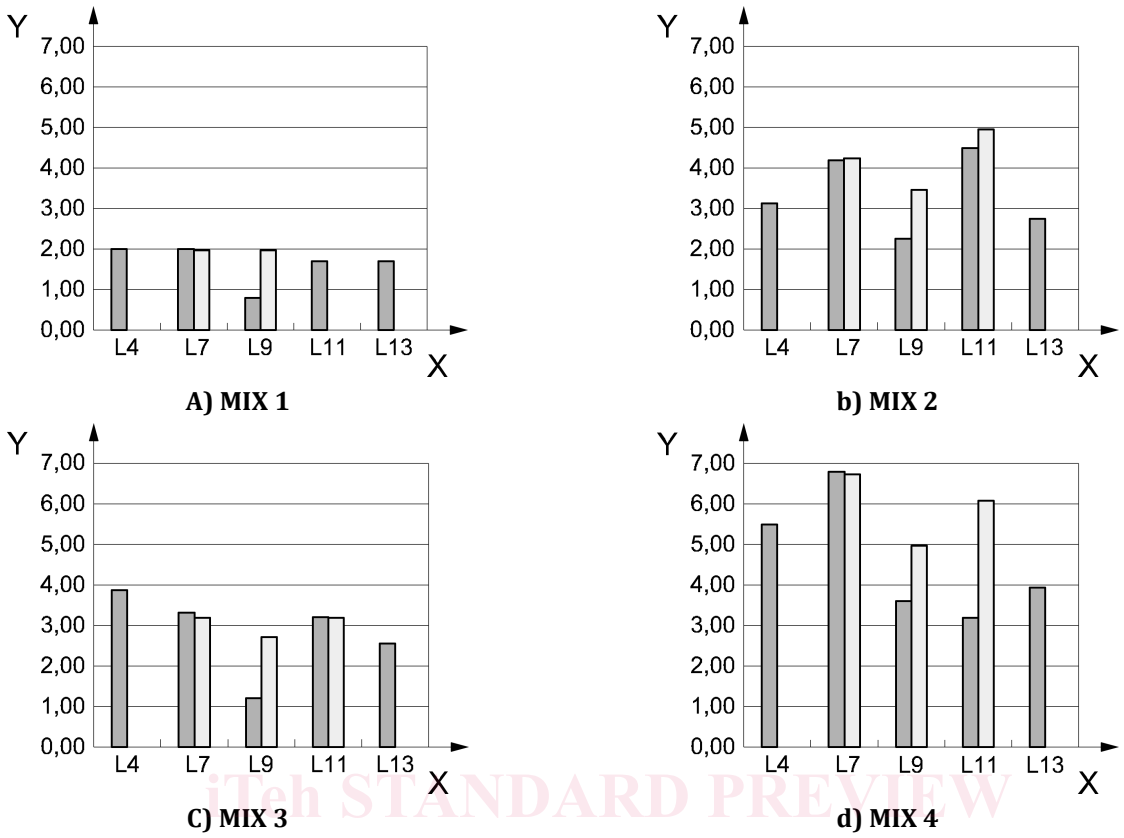
d) MIX 4

Key

x laboratories

y NCA1 dk (mm)

Figure 2 — Individual results of carbonation depth *dk* for each mix 1 to 4 in the order in natural exposure (NCA1) at equal or around 1 year (dark grey bar) and 2 years (light grey bar). (LAB 9 did not test at 365 days but at 190 days)



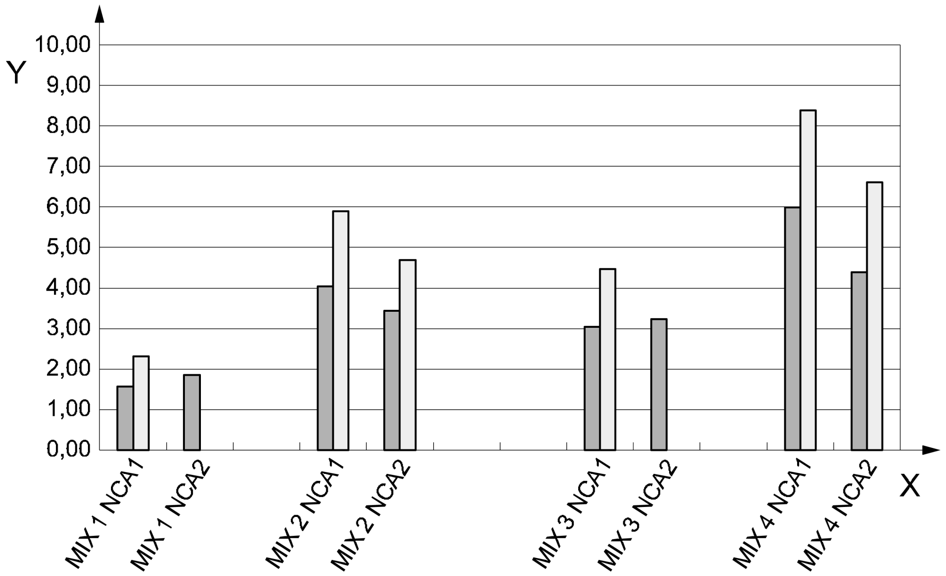
Key

x laboratories

y NCA2 dk (mm)

Figure 3 — Individual results of carbonation depth dk for each mix 1 to 4 in the order in climatic chamber (NCA2) at equal or around 1 year (dark grey bar) and at 2 years (light grey bar). (LAB 9 did not test at 365 days but at 190 days)

For comparative purposes between NCA1 and NCA 2 test procedures, the differences in the carbonation depth *dk* obtained at around 1 year are given in Figure 4. It is remarkable to deduce that, in spite of the very different climates in the countries of the different laboratories, the *dk* values in natural outdoors sheltered from rain exposure are reasonably similar to those values obtained through NCA2 (climatic chamber).



Key

y carbonation depth dk (mm) Average

x mix number and test type

Figure 4 — Comparison of carbonation depth of the testing procedures NCA1 (natural outdoors sheltered from rain) and NCA2 in the climatic chamber (LAB 9 did not test at 365 days but at 190 days). Individual values per mix at equal or around 1 year

The precision data of both test procedures (carbonation depth obtained in climatic chamber and outdoors sheltered from rain) are given in Table 7 and 8 for the results at 1 year only because it was considered that the number of laboratories making the tests at around two years was too few to allow the calculation of precision at that age. Due to the same reason of the limited number of laboratories, the repeatability was made with all data by variance analysis.

Table 7 — Summary of precision data of carbonation depth obtained for NCA1 method at one year testing

dk **Natural Carbonation NCA1 outdoors**			**Mix1**	**Mix2**	**Mix3**	**Mix4**
	Average	**(mm)**	**1,57**	**4,04**	**3,04**	**5,99**
Standard deviation	Repeatability	s_r (mm)	0,34			
	Reproducibility	s_R (mm)	0,86			
Variation coef.	**Repeatability**	CV_r **(%)**	**21,8**	**8,5**	**11,3**	**5,7**
	Reproducibility	CV_R **(%)**	**61,2**	**23,7**	**31,5**	**16,0**
Limit values	Repeatability	$r = 2{,}8\ s_r$ (mm)	0,96			
	Reproducibility	$R = 2{,}8\ s_R$ (mm)	2,42			
Limit values	Repeatability	$r = 2{,}8\ CV_r$ (%)	55,2	21,4	28,4	14,4
	Reproducibility	$R = 2{,}8\ CV_R$ (%)	154,5	59,9	79,6	40,4