Interlaboratory Test Program to determine the precision of the R3 test method (ASTM C1897-20) for measuring reactivity of supplementary cementitious materials

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Abstract:

The substitution of Portland clinker by supplementary cementitious materials (SCMs) is one of the main avenues towards further decarbonization of cement production. To secure future SCM supplies, generic and performance-related test methods for SCMs are required. RILEM Technical Committee 267-TRM recommended the so-called R3 test methods for SCM reactivity testing, ASTM adopted the test as C1897 in 2020. This paper presents the results of an interlaboratory study (ILS) that was carried out to develop precision statements for the ASTM C1897-20 test methods. The ILS was conducted and interpreted according to ASTM C802-14, and precision statements were prepared following ASTM C670-15. The ILS involved testing of 3 centrally supplied conventional SCMs by both test methods (R3 hydration heat and bound water) by at least 11 labs. The processing and analysis of the ILS data to come to the statements for single operator and multilaboratory precision are presented.

1. Introduction

As economies are moving away from carbon-intensive production processes, several well-known sources of SCMs such as coal combustion fly ashes or ground granulated blast furnaces slags are facing gradual depletion in the coming decades [1]. To maintain or even extend the use of SCMs in low carbon blended cements and concrete, a wide range of potential new sources of SCMs are being investigated

[2]. To facilitate the introduction of these new SCMs into practice, more generic, performance-based test methods and specifications are of interest [3, 4]. To address this perceived need,

RILEM technical committee TC 267-TRM “Tests for Reactivity of Supplementary Cementitious Materials” convened a group of academic and industrial experts to collaborate on the evaluation and development of reactivity tests for SCMs. The objective of the committee was to recommend test methods that were robust, applicable to a wide range of SCMs, and related closely to their performance in terms of contribution to strength development. In a first phase, existing and novel test methods were evaluated against robustness criteria and correlation to strength for conventional SCMs by round robin testing [5]. In a second stage, the test methods that performed best were investigated in more depth to further improve their performance and the description of the test procedures [6, 7]. As a result, the most promising test methods, i.e. the R3 test methods for heat release and bound water measurement, were recommended and presented to standardization committees. The R3 test was adopted by ASTM as C1897 in 2020 [8].

This interlaboratory study (ILS) was conducted by RILEM TC 267-TRM in line with ASTM C802 – 14 [9] that provides guidelines to design, execution and analysis of the data with the objective of developing precision statements of the R3 test method (ASTM C1897-20) [8]. The precision statements provide acceptance values for the single-operator and multilaboratory standard deviations and difference limits expressed according to ASTM C670-15 [10].

The ILS was preceded by a preliminary test program within RILEM TC 267 TRM to develop and refine the R3 test method [5, 6, 11] . The conduction of the preliminary test program involved all laboratories participating in the ILS, enabling them to get acquainted and receive training on the execution of the test method. Based on the preliminary test program results, critical conditions were identified and specified in a draft R3 test method to be subjected to the ILS within RILEM TC 267-TRM. After a series of editorial changes this test method was accepted by ballot as ASTM C 1897-20 [8]. The R3 test method describes two equivalent techniques to measure the reactivity of an SCM. For both the test mixture comprises the SCM, $Ca(OH)_2$, $CaCO_3$ and a potassium solution containing KOH and K_2SO_4 , simulating the reaction environment in a hydrating Portland cement (mix proportions are given in [8]). To avoid interferences in the measurements no actual Portland cement is used. The test mixture is cured at 40 °C to expediate the SCM reaction. To determine the reactivity of the SCM oOne technique measures the hydration heat by an isothermal calorimeter, the other technique measures the bound water by heat treatment in a laboratory oven. In the ILS the two techniques were tested and evaluated separately .

2. Materials and Methods

In the ILS, 3 materials, i.e. one ground granulated blast furnace slag (S1), one siliceous coal combustion fly ash (SFA-R), one calcined clay (CC3) were tested. As such the selection covered both pozzolanic (calcined clay and siliceous fly ash) and latent hydraulic (slag) SCMs and included materials of intermediate (fly ash) to high (slag, calcined clay) reactivity as SCM. The physico-chemical properties of the test materials are reported in Table 1 and Table 2.

Table 1. Physical properties, and chemical composition of the test materials [12].

Parameter	Calcined clay CC3	Slag S1	Fly ash SFA_R
Origin	India	France	Germany
SiO_2 (wt.%)	49.28	36.61	54.31
Al_2O_3 (wt.%)	42.28	12.21	22.72
TiO_2 (wt.%)	3.63	0.35	1.08
MnO (wt.%)	0.01	0.14	0.05
Fe_2O_3 (wt.%)	2.22	0.85	10.28
CaO (wt.%)	0.63	41.59	4.29
MgO (wt.%)	0.03	7.18	1.47
K_2O (wt.%)	0.06	0.28	2.08
Na_2O (wt.%)	0.16	0.18	0.87
SO_3 (wt.%)	0.06	0.63	0.32
P_2O_5 (wt.%)	0.09	0.40	0.55
L.O.I. 950 °C (wt.%)	-	-0.03	-
L.O.I. 1050 °C (wt.%)	2.26	-	1.32
Density (g/cm^3)	2.67	2.48	2.17
Dv_{10} (µm)	0.2	3.6	2.3
Dv_{50} (µm)	4.30	10.0	18.6
Dv_{90} (µm)	32.7	36.3	81.2

Table 2. Mineralogical composition of the test materials [12].

Content (wt.%)	Calcined clay CC3	Slag S1	Fly ash SFA_R
Anatase	1.5	-	-
Anhydrite	-	-	1.3
Calcite	-	0.5	-
Hematite	0.9	-	1.5
Kaolinite	5.3	-	-
Magnetite	-	-	1.4
Mellilite	-	0.3	-
Mullite	12.0	-	11.6
Quartz	0.8	-	8.8
Rutile	1.3	-	-
Amorphous	78.0	99.2	75.0

The test determinations were carried out on paste samples incorporating SCMs following the mix design of the model paste shown in Table 3. [10, 12]. All paste constituents were weighed and brought to 40 °C before mixing.

Table 3. Mass proportions of the R^3 test model paste. [10, 12]

Ingredient	SCM	Calcium hydroxide	Calcium carbonate	Potassium solution*
Mass (g)	10.0	30.0	5.0	54.0

* The potassium solution consists of 4.00 g/L KOH and 20.0 g/L of K_2SO_4 dissolved in de-ionised water.

After mixing, the pastes were tested for their bound water after 7 days of curing and for their heat release at 3 and 7 days by isothermal calorimetry. For the bound water test, the paste samples were cured in sealed plastic containers at 40 °C for 7 days. Afterwards, the samples were crushed and sieved on a 2 mm sieve and dried in an oven at 40 °C for 24 h. The dried samples were heated at 350 °C for 2 h and cooled in a desiccator for 1 h. The bound water (for hydrates, excluding portlandite) was calculated according to Equation 1, where w_0 is the total mass of the dried paste and crucible, w_h is the total mass of the 350 °C - dehydrated paste and crucible, and w_c is the mass of the empty crucible.

$$H_2O_{bound,dried}\left(\frac{g}{100g\ of\ dried\ paste}\right)=\frac{w_0-w_h}{w_0-w_c}\times 100 \qquad \text{Equation 1}$$

The heat of hydration of the test pastes was measured by isothermal calorimetry at 40 °C for 7 days. The cumulative heat release per gram of SCM (H_{SCM}) was calculated using Equation 2, where H is the cumulative heat from 75 minutes after mixing until 3 or 7 days, m_p is the mass of paste in the calorimeter vial, and 0.101 is the mass fraction of the SCM in the paste specimen.

$$H_{SCM}\left(\frac{J}{g\ of\ SCM}\right) = \frac{H}{(m_p \times 0.101)} \qquad \text{Equation 2}$$

Full details of the testing protocol can be found in ASTM C1897 [10].

Eleven laboratories tested the materials for hydration heat, 13 laboratories measured bound water according to test method. The participating laboratories comprise laboratories of the cement industry, universities, and research institutes part of RILEM TC TRM-267 and disposing of testing equipment complying to the conditions described in the test method. All test determinations were requested to be carried out in triplicate. As such the ILS program comprises ILS data on two material characteristics, hydration heat and bound water reported by *p* = 11 and *p* = 13 laboratories, respectively; *q* = three materials; and *n* = three replicate test determinations per material. In consequence the ILS initially involved 33 and 39 degrees of freedom for single-operator standard deviation for measurement of hydration heat and bound water. For the hydration heat determination, 3 labs returned results in duplicate only, 1 additional lab provided only duplicate values for CC3. As such, for hydration heat the degree of freedom was 30 for S1 and SFA-R, and 29 for CC3. Hence, overall, the ILS is deemed to satisfy the C670-15 and C802-14 guidelines of involving at least 30 degrees of freedom for single-operator standard deviation and at least 10 participating laboratories for both characteristics.

The data analysis follows the procedure described in ASTM C802-14 to determine the single-operator and between-laboratory components of variance. Accordingly the statistical properties are calculated from the *n* replicate test determination within each of the *p* laboratories as follows:

x_{gij}	= single test determination *g* by laboratory *i* for material *j*
$\bar{X}_{ij} = \frac{\sum x_{gij}}{n}$	= average of *n* replicate test determinations for laboratory *i* on material *j*
$s_{rij}^2 = \frac{\sum\left(x_{gij} - \bar{X}_{ij}\right)^2}{n-1}$	= single-operator variance of replicate determinations for laboratory *i* on material *j*
$s_{rj}^2 = \frac{\sum s_{rij}^2}{p}$	= pooled single-operator variance for material *j*

$$\bar{X}_j = \frac{\sum \bar{X}_{ij}}{p}$$

= overall average for all laboratories for material *j*

$$s_{\bar{X}_j}^2 = \frac{\sum \left(\bar{X}_{ij} - \bar{X}_j\right)^2}{p-1}$$

= variance of laboratory averages for material *j*

$$s_{Lj}^2 = s_{\bar{X}_j}^2 - \frac{s_{rj}^2}{n}$$

= between laboratory component of variance for material *j*.

To verify the consistency of the ILS data, two statistic parameters are used. The *h*-value is used to check if the average value for a laboratory is consistent with the overall average for a given materials. The *k*-value for each laboratory is used to evaluate the consistency of the single-operator variability for a given material. The *h*-value and the *k*-value are calculated as follows:

$$h_{ij} = \frac{\bar{X}_{ij} - \bar{X}_j}{s_{\bar{X}_j}}$$

= *h*-value for verification of consistency of laboratory averages

$$k_{ij} = \frac{s_{rij}}{s_{rj}}$$

= *k*-value for verification of consistency of laboratory dispersion

The calculated *h*-values and *k*-values were compared to critical values that depend on the number of participating laboratories and the number of test replicates as given by ASTM C802-14 for a 0.5% significance level. Non-consistent results were further examined for reporting errors and for aberrations in the laboratory procedures or test equipment performance.

As a last step, statements for both test methods were developed for single-operator and multilaboratory precision following ASTM Practice C670.

3. Results

3.1. Reported data

The participating laboratories were given a data sheet for reporting their results. The reported data are summarized in Table 4 and Table 5 for hydration heat and bound water, respectively. Following ASTM C1897-20 the determination results for hydration heat are expressed as J/g per gram of SCM, and the determination results for bound water as g per 100 g of paste dried at 40 °C. Figure 1 provides a plot of the reported 3 day hydration heat results, Figure 2 is a plot of the 7 day hydration heat results, and Figure 3 is a plot of the bound water data. Visual examination of the plots does not reveal gross errors, however there are a few combinations of laboratories and materials that seem to deviate from general trends. These are subjected to a check for data consistency.

Table 4. Summary data sheet of the ILS for measurement of hydration heat according to the R3 test method (ASTM C1897-20)

Laboratory	Replicate	Material					
		CC3		S1		SFA-R	
		3 days	7 days	3 days	7 days	3 days	7 days
1	a	400.7	430.5	447.7	496.7	115.3	202.2
	b	412.4	443.2	447.2	496.1	113.7	202.0
	c	410.6	441.3	441.1	490.6	112.8	195.9
2	a	-	-	-	-	-	-
	b	513.5	559.4	441.2	493.7	106.2	192.9
	c	517.6	559.7	443.0	495.4	104.6	192.3
3	a	433.6	468.0	426.6	489.6	126.7	217.2
	b	423.2	448.6	425.1	494.9	122.9	209.5
	c	439.9	476.2	419.6	482.5	121.6	208.3
4	a	517.2	562.3	436.2	484.2	115.5	192.8
	b	519.5	567.0	441.6	492.5	120.0	203.2
	c	-	-	440.5	487.8	120.9	199.4
5	a	525.10	572.30	460.40	518.30	105.40	175.40
	b	531.00	572.2	461.90	518.4	103.00	171.6
	c	524.30	578.2	459.20	516.1	102.80	171.2
6	a	538.7	585.2	463.0	537.5	113.3	202.8
	b	540.0	583.5	455.5	514.0	116.0	201.5
	c	539.0	584.5	448.2	503.5	113.0	197.8
7	a	447.7	480.2	429.5	495.9	103.9	192.1
	b	444.6	479.1	449.5	547.7	103.5	201.1
	c	436.9	482.0	438.0	524.1	101.8	198.9
8	a	473.7	519.5	416.3	508.6	110.8	188.3
	b	494.4	544.0	430.7	536.1	110.3	187.6
	c	481.4	526.4	418.8	522.5	106.3	182.3
9	a	510.1	554.1	461.0	519.9	115.0	197.4
	b	516.0	558.1	463.5	516.2	119.6	201.0
	c	518.7	557.6	463.1	515.9	119.0	195.7
10	a	564.00	616.0	480.00	537.0	120.60	216.0
	b	568.0	619.0	495.0	554.0	119.5	215.0
	c	-	-	-	-	-	-

11	a	*497.7*	541.6	461.6	514.3	103.2	181.8
	b	494.6	535.9	452.3	508.0	103.2	181.4
	c	-	-	-	-	-	-

Table 5. Summary data sheet of the ILS for the measurement of bound water according to the R3 test method (ASTM C1897-20)

Laboratory	Replicate	Material		
		CC3	S1	SFA-R
1	a	10.24	7.83	4.36
	b	10.40	7.95	4.47
	c	10.26	8.14	4.63
2	a	11.94	8.93	5.99
	b	11.60	8.82	5.60
	c	11.94	8.93	6.25
3	a	10.23	8.16	4.27
	b	10.47	7.64	5.03
	c	10.21	7.65	5.04
4	a	10.21	8.42	4.28
	b	10.43	8.53	4.14
	c	10.59	8.61	5.06
5	a	10.54	8.06	4.94
	b	10.69	7.85	4.62
	c	10.39	8.12	4.67
6	a	8.95	6.91	4.30
	b	9.10	7.33	4.36
	c	9.13	7.32	4.44
7	a	10.30	7.40	5.20
	b	10.70	7.50	5.30
	c	10.20	6.80	4.90
8	a	10.85	8.13	6.10
	b	10.99	8.65	6.57
	c	10.95	8.65	6.72
9	a	8.48	7.92	4.97
	b	8.51	7.50	4.82
	c	7.86	7.87	4.87

10	a	10.16	7.58	5.01
	b	10.40	7.52	5.19
	c	10.62	7.57	5.06
11	a	10.52	7.69	4.76
	b	10.59	7.86	4.76
	c	10.89	7.39	4.76
12	a	10.93	7.74	5.89
	b	10.81	7.86	5.62
	c	10.52	7.96	5.94
13	a	9.90	7.40	5.10
	b	10.20	7.30	4.90
	c	10.10	7.60	5.20

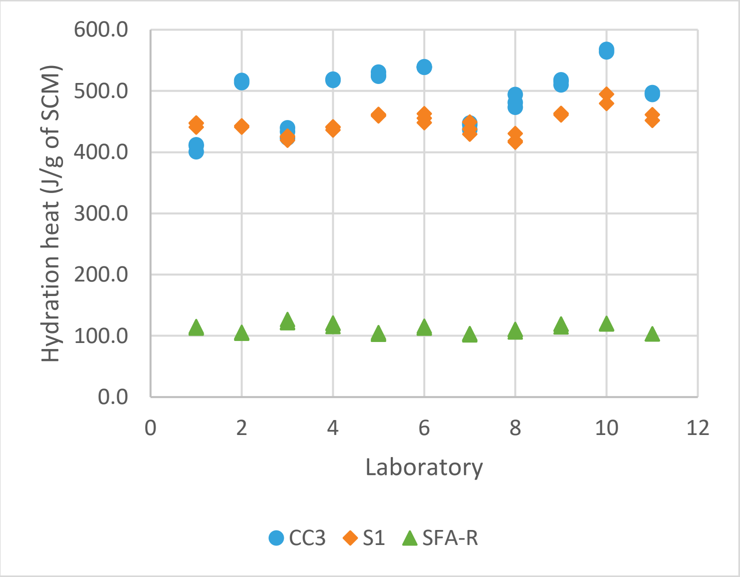

Figure 1. Plot of all 3 day hydration heat test determinations by laboratory

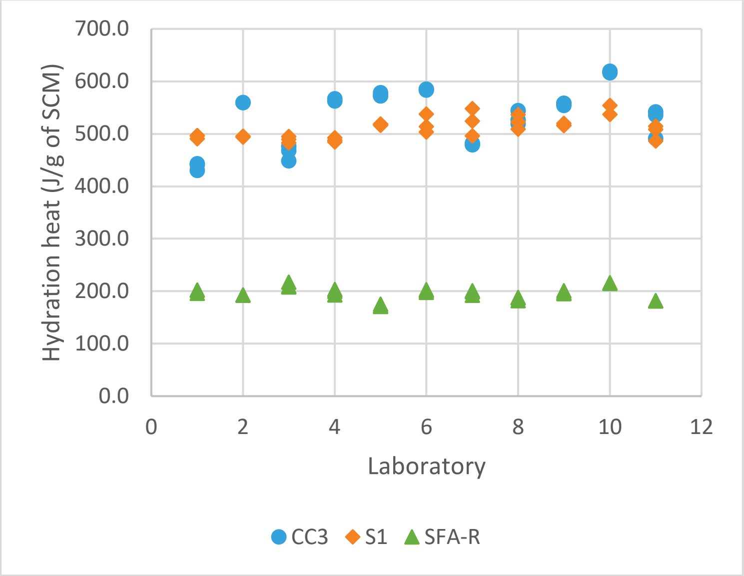

Figure 2. Plot of all 7 day hydration heat test determinations by laboratory

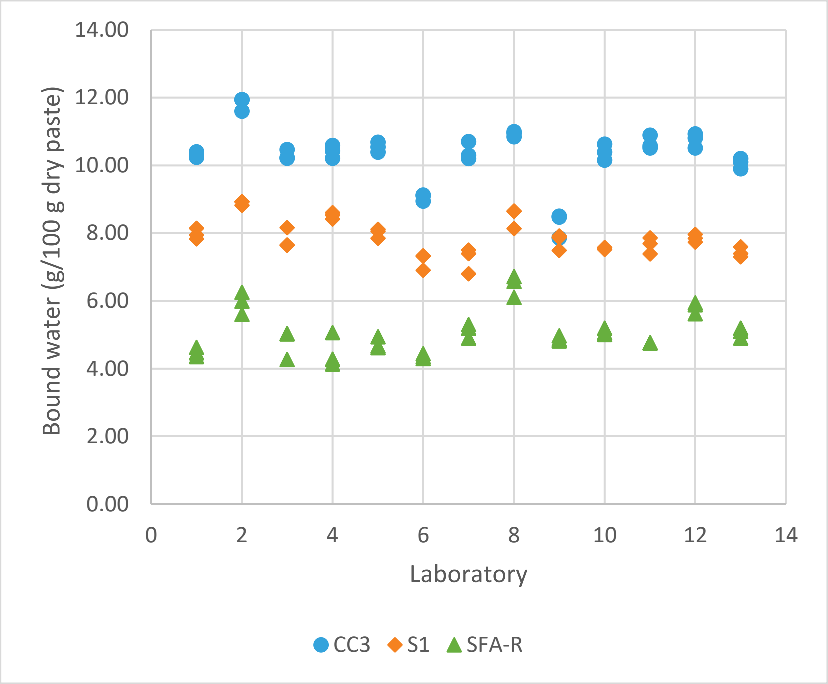

Figure 3. Plot of all bound water test determinations by laboratory

3.2. Data consistency

A preliminary analysis involves data from all laboratories and is needed to check for data consistency. The replicate data in each material column are transposed in tables for each material and characteristic as shown in Tables A.1-9 in the Annex. The average and the variance of the replicate test determinations of each characteristic by each laboratory are given in the last two columns of each of the tables in the Annex. Beneath each table the overall average, the pooled single-operator variance, the variance of the laboratory averages and the between-laboratory component of variance are given as calculated according to section 2.

The consistency of the data are evaluated by comparing *h*- and *k*-values calculated according to section 2 to the critical values given by ASTM C802-14. The *h*-values are used to check whether the average values for a laboratory are consistent with the overall averages, i.e. it is an indicator for between-laboratory consistency. Figure 4a and Figure 5a show the *h*-values for each tested material by laboratory for the hydration heat test determinations at 3 and 7 days, respectively. Figure 6a presents the h-values for the bound water test determinations. The critical value of *h* for the hydration heat measurements (p = 11 and n = 3) is ± 2.34; for the bound water test (p = 13 and n = 3) the critical *h*-value is ± 2.41. Examination of the *h*-value plots shows that there are no transgressions of the critical *h*-values for any of the test datasets. In case of hydration heat, the majority of laboratories show both positive and negative values, few laboratories have consistently positive or negative values. Laboratory 10 shows consistently high values. In case of bound water content the *h*-value patterns are similar, two laboratories (nr 2 and 8) appear to report consistently high values, laboratory 6 appears to always report low values. The observed patterns are considered regular. Since no excesses of critical values are observed the between-laboratory data consistency is deemed satisfactory, requiring no further investigation.

The *k*-values are used to evaluate the single-operator consistency. Figure 4b and Figure 5b report the *k*-values for each tested material by laboratory for the hydration heat test determinations at 3 and 7 days, respectively. Figure 6b shows the *k*-values for the bound water test determinations. The critical value of *k* for the hydration heat measurements (p = 11 and n = 3) is 2.13; for the bound water test (p = 13 and n = 3) the critical *k*-value is 2.15. There is no single laboratory showing excessive k-values for all or most materials. This indicates that the datasets are generally consistent. Two excesses of the k-values were noted. In case of 7 days hydration heat, laboratories 3 and 7 show high *k*-values for the CC3 and S1 test materials, respectively. Since for both laboratories the *k*-values for other tested materials are in line with the other laboratories, and there are no deviations for the 3 days hydration heat data either, there is no further need for examination or remediation. All data are therefore considered consistent and taken into account for the development for the precision statements. Plots of the *h*- and *k*-values grouped by material show similar trends and are given in the Annex as Figures A.1 to A.3.

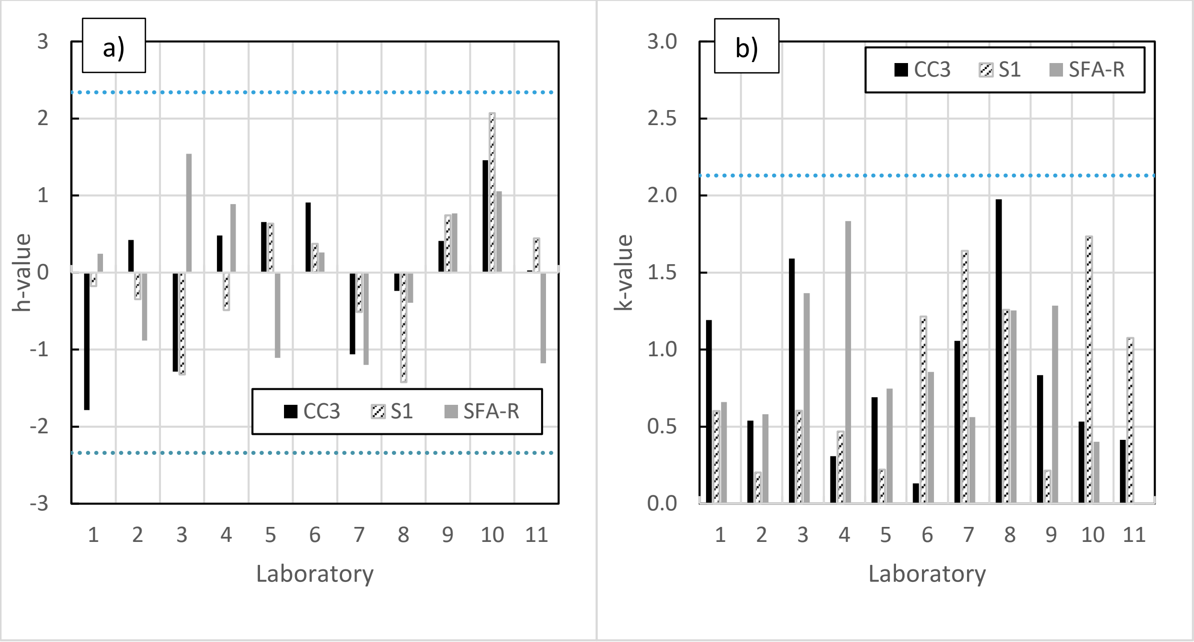

Figure 4. h-values (a, left graph) and k-values (b, right graph) grouped by laboratory for the 3 days hydration heat test determinations. The critical h- and k-values are indicated by the dotted lines.

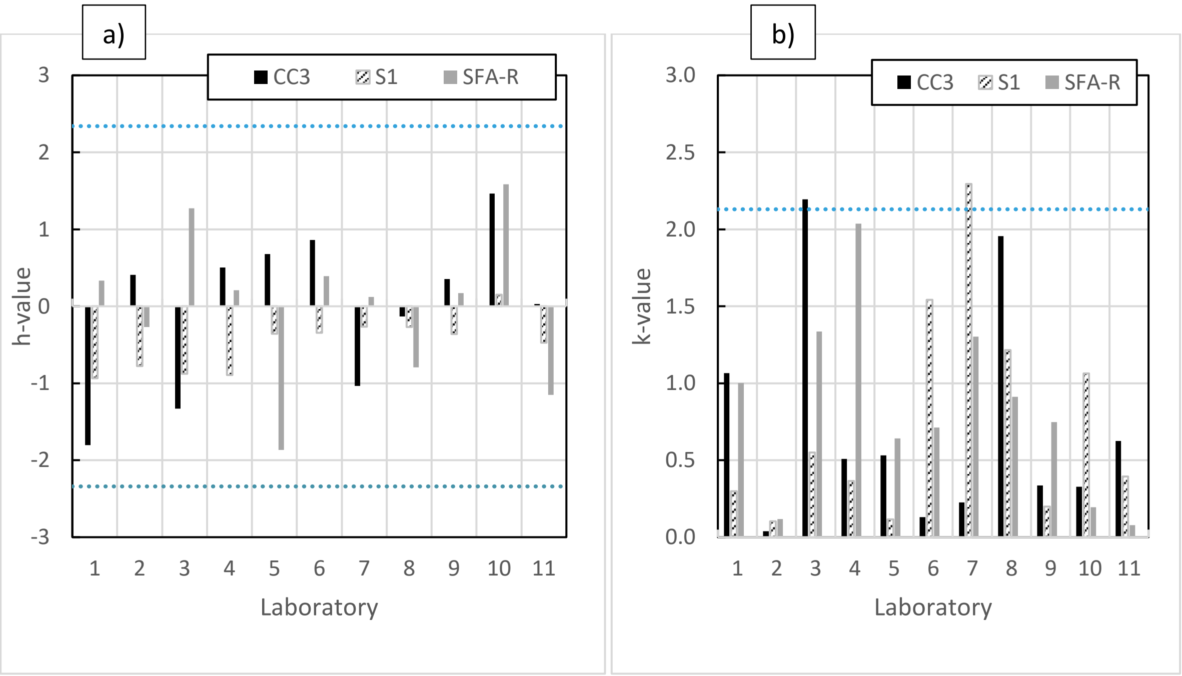

Figure 5. h-values (a, left graph) and k-values (b, right graph) grouped by laboratory for the 7 days hydration heat test determinations. The critical h- and k-values are indicated by the dotted lines.

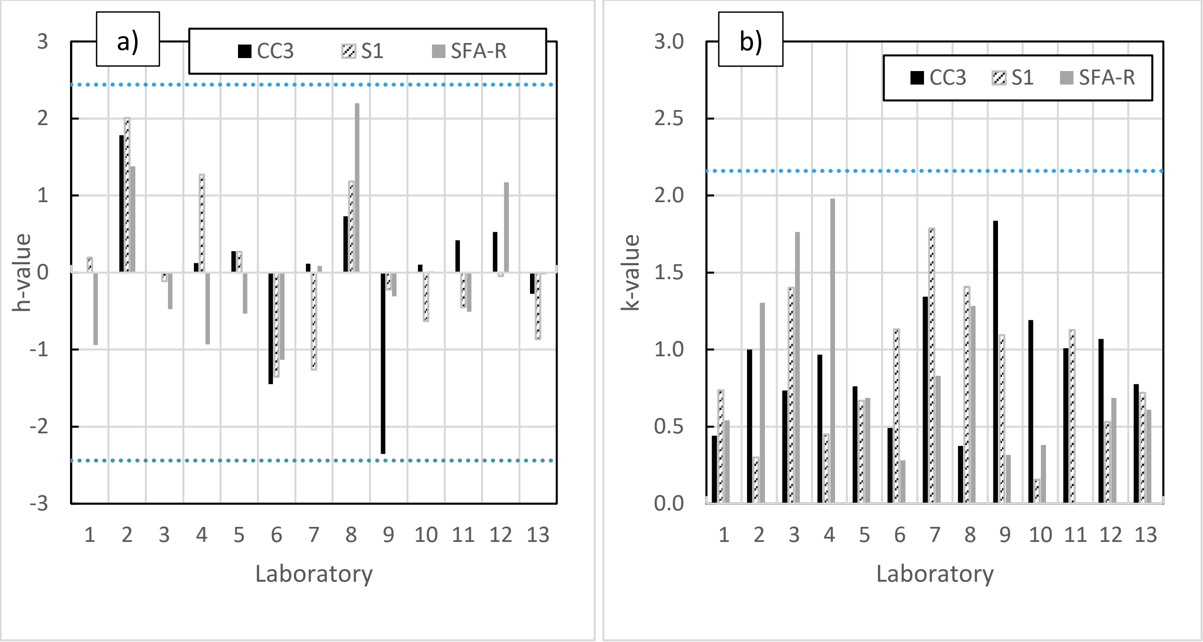

Figure 6. h-values (a, left graph) and k-values (b, right graph) grouped by laboratory for the bound water test determinations. The critical h- and k-values are indicated by the dotted lines.

In the next step, the data are investigated for interactions. Figure 7 presents a dot plot of the average hydration heats at 3 and 7 days for each laboratory, grouped by material. Figure 8 shows the same for the average bound water content. In case of bound water content (Figure 8) all results follow the same pattern and the rating of the materials is the same for all laboratories. For the hydration heat results (Figure 7), a few laboratories show lower average values for CC3 than for S1. This pattern deviation may be indicative for a potential issue and therefore requires further examination to find the cause of the interaction. Further investigation indicated that the likely cause of this interaction is an unexpected heterogeneity of the CC3 test material. Unlike commercially available test materials S1 and SFA_R, the CC3 material was derived from a pilot calcination test. Heterogeneous processing conditions and inadequate homogenization of the CC3 material may therefore have affected the results. Eliminating all CC3 test results would reduce the number of test materials to a low number of 2, therefore the development of the precision statements is in first instance made including the CC3 data. As a consequence the precision statement values may be overestimated to some extent.

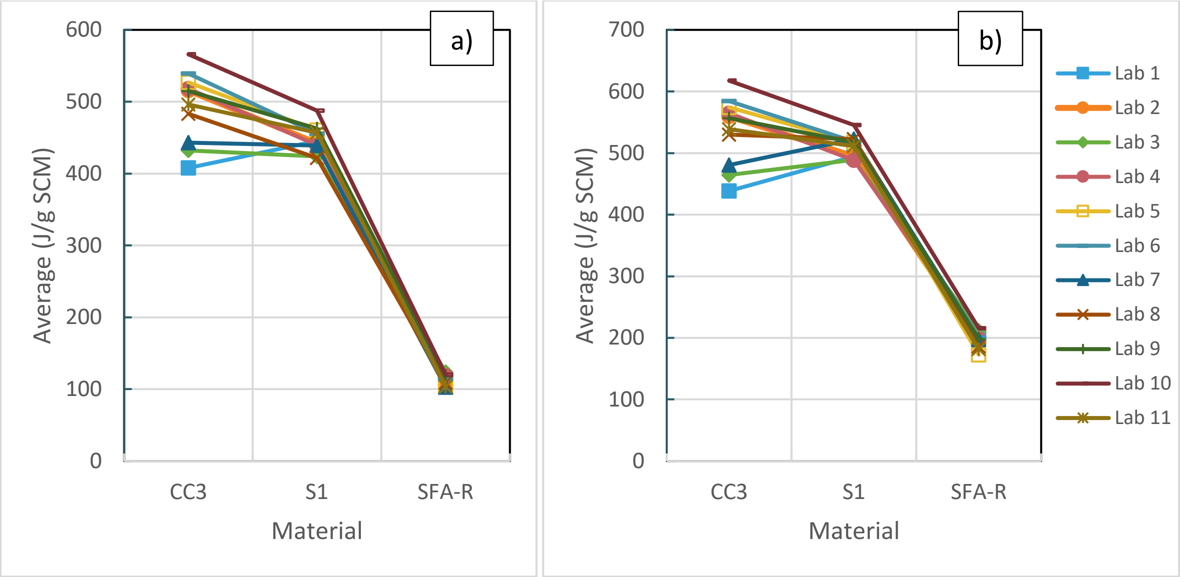

Figure 7. Average hydration heat for each laboratory by material at a) 3 days and b) 7 days of hydration.

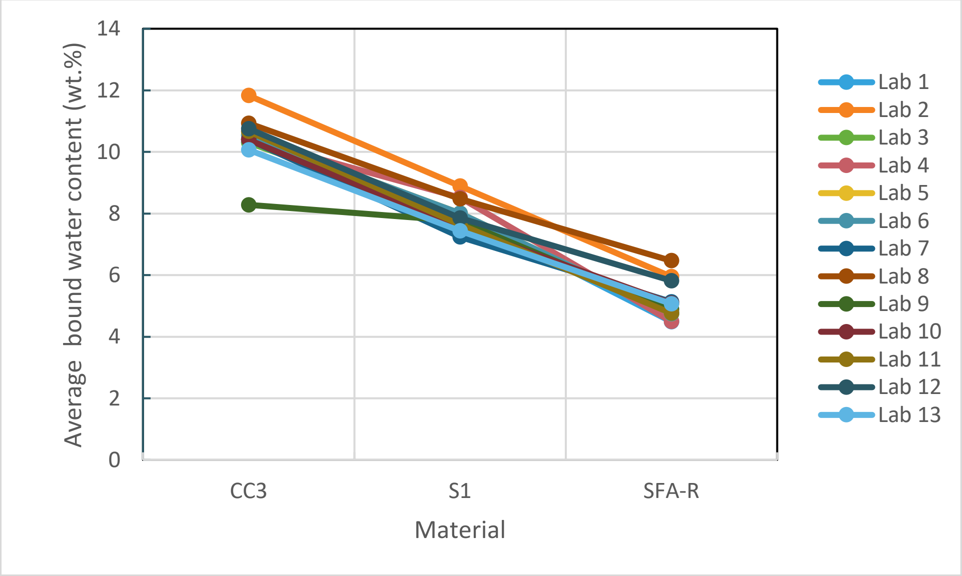

Figure 8. Average bound water content for each laboratory by material

3.3. Development of precision statements

The averages, standard deviations and coefficients of variation of the validated data set are collected and assembled in Table 6. To determine the appropriate expression of the precision statements, the single-operator and multilaboratory standard deviations and coefficients of variation are plotted versus the pooled averages for each material. Figure 9 is a scatter plot of the standard deviations versus average values for hydration heat at 3 and 7 days. As can be observed the standard deviation values increase more or less linearly with hydration heat, and are therefore not suitable to define precision statement values. Figure 10 gives a cross plot for the coefficients of variation and the averages for the hydration heat test. It is observed that the coefficient of variation is relatively constant. Therefore in case of hydration heat, the average single-operator coefficient of variation and the average multilaboratory coefficient of variation are to be used to write the precision statements in accordance with ASTM Practice C670.Figure 9. Standard deviations (SO: Single-operator, ML: multilaboratory) for hydration heat at 3 and 7 days versus average values.

Table 6. Averages, standard deviations, and coefficients of variation for all test methods and materials. Values in bold are used in the test method precision statements.

Summary table						
Measure	Material	Average	Standard Deviation		Coefficient of Variation	
			Single operator	Multilaboratory	Single-Operator	Multilaboratory
Hydration heat 3 d		J/g SCM	J/g SCM	J/g SCM	%	%
	CC3	494.8	5.3	48.9	1.1	9.9
	S1	448.6	6.1	19.4	1.4	4.3
	SFA-R	112.1	1.9	7.7	1.7	6.9
	Average	-	4.4	25.3	**1.4**	**7.0**
Hydration heat 7 d	CC3	537.2	6.5	55.1	1.2	10.3
	S1	511.0	11.3	20.0	2.2	3.9
	SFA-R	195.9	3.6	12.7	1.9	6.5
	Average	-	7.1	29.3	**1.8**	**6.9**
Bound water		Wt.%	Wt.%	Wt.%	%	%
	CC3	10.30	0.20	0.87	1.9	8.5
	S1	7.87	0.21	0.54	2.7	6.8
	SFA-R	5.08	0.25	0.66	4.9	13.0
	Average	-	**0.22**	**0.70**	3.2	9.4

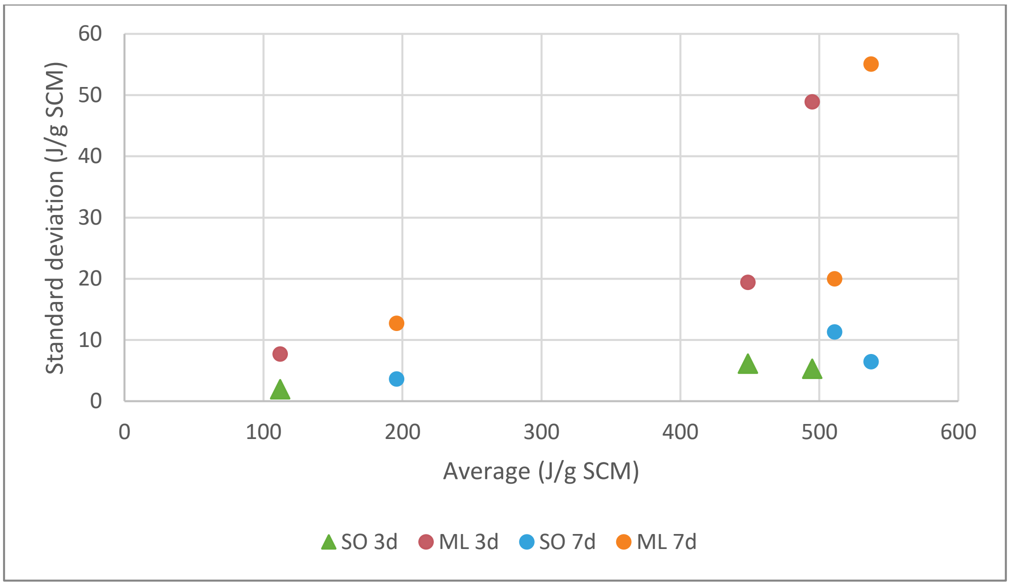

Figure 9. Standard deviations (SO: Single-operator, ML: multilaboratory) for hydration heat at 3 and 7 days versus average values.

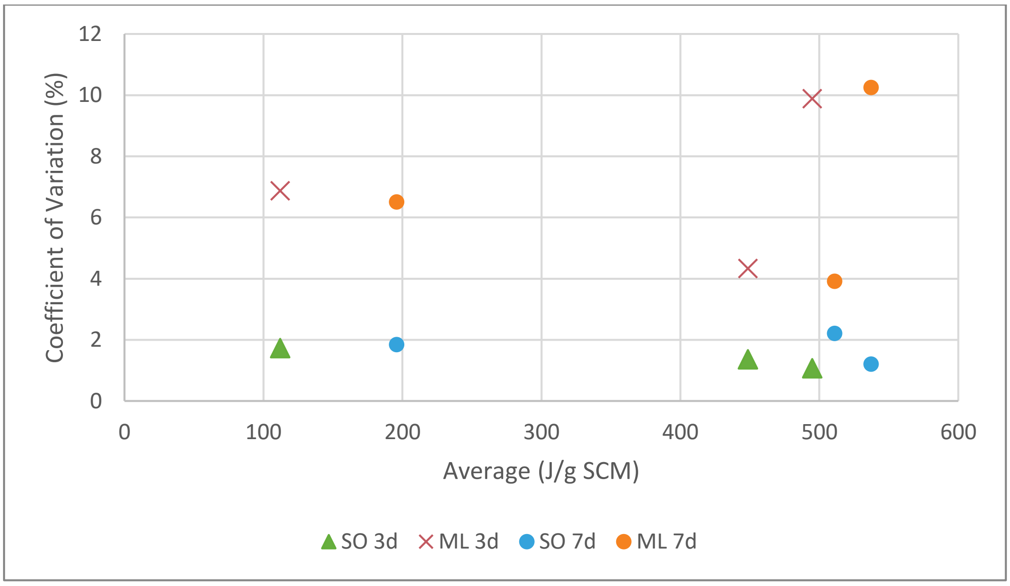

Figure 10. Coefficients of variation (SO: Single-operator, ML: multilaboratory) for hydration heat at 3 and 7 days versus average values.

The standard deviations and coefficients of variation for the bound water content determinations are plotted against the corresponding average values in Figure 11. In this case it can be observed that standard deviation values are more or less constant, while the coefficients of variation tend to decrease with increasing bound water content. Therefore, in case of bound water content, the average single-operator standard deviation and the average multilaboratory standard deviation are used in the precision statements.

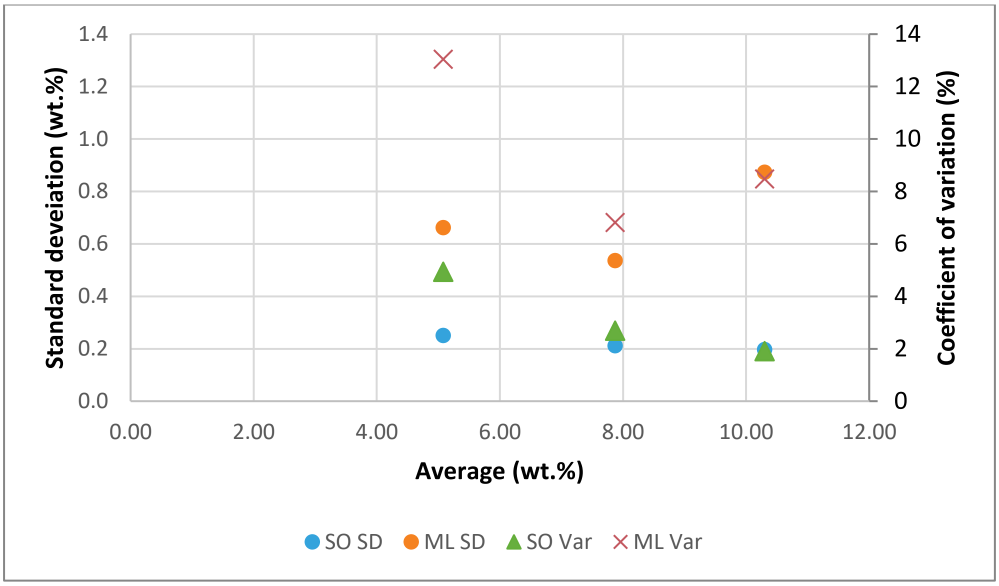

Figure 11. Standard deviations and coefficients of variation (SO: Single-operator, ML: multilaboratory) for bound water content versus average values.

In consequence, the precision statements for each test method are written according to ASTM C670 as follows.

3 day hydration heat

The single-operator precision coefficient of variation of a single test determination of 3 day hydration heat has been found to be 1.4 %. Therefore, results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 3.9 %.

The multilaboratory precision coefficient of variation of a single test determination of 3 day heat release has been found to be 7.0 %. Therefore, results of two properly conducted tests by two different laboratories on specimens of the same material are not expected to differ by more than 19.7 %.

7 day hydration heat

The single-operator precision coefficient of variation of a single test determination of 7 day hydration heat has been found to be 1.8 %. Therefore, results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 4.9 %.

The multilaboratory precision coefficient of variation of a single test determination of 7 day heat release has been found to be 6.9 %. Therefore, results of two properly conducted tests by two different laboratories on specimens of the same material are not expected to differ by more than 19.3 %.

Bound water content

The single-operator precision standard deviation of a single test determination of bound water content has been found to be 0.22 g per 100 g of dried paste. Therefore, results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 0.62 g per 100 g of dried paste.

The multilaboratory precision standard deviation of a single test determination of bound water content has been found to be 0.70 g per 100 g of dried paste. Therefore, results of two properly conducted tests by two different laboratories on specimens of the same material are not expected to differ by more than 1.97 g per 100 g of dried paste.

4. Conclusion

This paper reports the results of an interlaboratory test program that aimed at determining the precision of the R3 heat of hydration and bound water test methods (ASTM C1897-20). The design of the test program and the analysis and evaluation of the results was carried out following the ASTM Standard Practice C802-14. It is concluded that the test program produced a valid set of consistent data enabling the definition of precision statements for ASTM C1897-20. For hydration heat the precision statements may be somewhat overestimated due to heterogeneity in one of the test materials. An additional source of error may be the relative novelty of the test methods to several of the participating laboratories. A future ILS may enable to further reduce the allowed single-operator and multilaboratory precision values.

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Annex

Table A.1. Single-operator and between laboratory analysis for CC3 – 3 days hydration heat

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	400.7	412.4	410.6	407.9	39.9
2		513.5	517.6	515.5	8.2
3	433.6	423.2	439.9	432.2	71.1
4	517.2	519.5		518.4	2.7
5	525.1	531.0	524.3	526.8	13.4
6	538.7	540.0	539.0	539.2	0.5
7	447.7	444.6	436.9	443.1	31.4
8	473.7	494.4	481.4	483.2	109.6
9	510.1	516.0	518.7	514.9	19.5
10	564.00	568.0		566.0	8.0
11	497.7	494.6		496.2	4.8

Overall average = $\bar{X}$ = 494.8

Pooled single-operator variance = s_r^2 = 28.1

Variance of laboratory averages = $s_{\bar{X}}^2$ = 2374

Between-laboratory component of variance = s_L^2 = 2365

Table A.2. Single-operator and between laboratory analysis for S1 – 3 days hydration heat

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	447.7	447.2	441.1	445.3	13.5
2		441.2	443.0	442.1	1.5
3	426.6	425.1	419.6	423.8	13.6
4	436.2	441.6	440.5	439.4	8.2
5	460.4	461.9	459.2	460.5	1.8
6	463.0	455.5	448.2	455.6	55.1
7	429.5	449.5	438.0	439.0	100.7
8	416.3	430.7	418.8	421.9	59.3

9	461.0	463.5	463.1	462.5	1.7
10	480.00	495.0		487.5	112.5
11	461.6	452.3		457.0	43.2

Overall average = $\bar{X}$ = 448.6

Pooled single-operator variance = s_r^2 = 37.4

Variance of laboratory averages = $s_{\bar{X}}^2$ = 353

Between-laboratory component of variance = s_L^2 = 340

Table A.3. Single-operator and between laboratory analysis for SFA-R – 3 days hydration heat

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	115.3	113.7	112.8	113.9	1.6
2		106.2	104.6	105.4	1.3
3	126.7	122.9	121.6	123.7	7.0
4	115.5	120.0	120.9	118.8	12.6
5	105.4	103.0	102.8	103.7	2.1
6	113.3	116.0	113.0	114.1	2.8
7	103.9	103.5	101.8	103.0	1.2
8	110.8	110.3	106.3	109.1	5.9
9	115.0	119.6	119.0	117.9	6.2
10	120.6	119.5		120.1	0.6
11	103.2	103.2		103.2	0.0

Overall average = $\bar{X}$ = 112.1

Pooled single-operator variance = s_r^2 = 3.8

Variance of laboratory averages = $s_{\bar{X}}^2$ = 57

Between-laboratory component of variance = s_L^2 = 56

Table A.4. Single-operator and between laboratory analysis for CC3 – 7 days hydration heat

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	430.5	443.2	441.3	438.3	47.3
2		559.4	559.7	559.6	0.1
3	468.0	448.6	476.2	464.3	200.9

4	562.3	567.0		564.7	10.7
5	572.3	572.2	578.2	574.2	11.8
6	585.2	583.5	584.5	584.4	0.7
7	480.2	479.1	482.0	480.4	2.1
8	519.5	544.0	526.4	530.0	159.5
9	554.1	558.1	557.6	556.6	4.7
10	616.0	619.0		617.5	4.5
11	541.6	535.9		538.8	16.2

Overall average = $\bar{X}$ = 537.2

Pooled single-operator variance = s_r^2 = 41.7

Variance of laboratory averages = $s_{\bar{X}}^2$ = 3004

Between-laboratory component of variance = s_L^2 = 2991

Table A.5. Single-operator and between laboratory analysis for S1 – 7 days hydration heat

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	496.7	496.1	490.6	494.5	11.5
2		493.7	495.4	494.5	1.4
3	489.6	494.9	482.5	489.0	38.7
4	484.2	492.5	487.8	488.2	17.0
5	518.30	518.4	516.1	517.6	1.7
6	537.5	514.0	503.5	518.3	303.7
7	495.9	547.7	524.1	522.6	672.9
8	508.6	536.1	522.5	522.4	189.0
9	519.9	516.2	515.9	517.4	5.1
10	537.0	554.0		545.5	144.5
11	514.3	508.0		511.2	19.8

Overall average = $\bar{X}$ = 511.0

Pooled single-operator variance = s_r^2 = 127.7

Variance of laboratory averages = $s_{\bar{X}}^2$ = 314

Between-laboratory component of variance = s_L^2 = 272

Table A.6. Single-operator and between laboratory analysis for SFA-R – 7 days hydration heat

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	202.2	202.0	195.9	200.0	13.1
2		192.9	192.3	192.6	0.2
3	217.2	209.5	208.3	211.7	23.3
4	192.8	203.2	199.4	198.5	54.2
5	175.4	171.6	171.2	172.7	5.4
6	202.8	201.5	197.8	200.7	6.6
7	192.1	201.1	198.9	197.4	22.2
8	188.3	187.6	182.3	186.0	10.8
9	197.4	201.0	195.7	198.0	7.3
10	216.0	215.0		215.5	0.5
11	181.8	181.4		181.6	0.1

Overall average = $\bar{X}$ = 195.9

Pooled single-operator variance = s_r^2 = 13.1

Variance of laboratory averages = $s_{\bar{X}}^2$ = 154

Between-laboratory component of variance = s_L^2 = 149

Table A.7. Single-operator and between laboratory analysis for CC3 – bound water content

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	10.24	10.40	10.26	10.3	0.01
2	11.94	11.60	11.94	11.8	0.04
3	10.23	10.47	10.21	10.3	0.02
4	10.21	10.43	10.59	10.4	0.04
5	10.54	10.69	10.39	10.5	0.02
6	8.95	9.10	9.13	9.1	0.01
7	10.30	10.70	10.20	10.4	0.07
8	10.85	10.99	10.95	10.9	0.01
9	8.48	8.51	7.86	8.3	0.13
10	10.16	10.40	10.62	10.4	0.06

11	10.52	10.59	10.89	10.7	0.04
12	10.93	10.81	10.52	10.8	0.04
13	9.90	10.20	10.10	10.1	0.02

Overall average = $\bar{X}$ = 10.3

Pooled single-operator variance = s_r^2 = 0.04

Variance of laboratory averages = $s_{\bar{X}}^2$ = 0.74

Between-laboratory component of variance = s_L^2 = 0.72

Table A.8. Single-operator and between laboratory analysis for S1 – bound water content

Laboratory	Data			Average	Single Operator Variance
	a	b	c		
1	7.83	7.95	8.14	8.0	0.0
2	8.93	8.82	8.93	8.9	0.0
3	8.16	7.64	7.65	7.8	0.1
4	8.42	8.53	8.61	8.5	0.0
5	8.06	7.85	8.12	8.0	0.0
6	6.91	7.33	7.32	7.2	0.1
7	7.40	7.50	6.80	7.2	0.1
8	8.13	8.65	8.65	8.5	0.1
9	7.92	7.50	7.87	7.8	0.1
10	7.58	7.52	7.57	7.6	0.0
11	7.69	7.86	7.39	7.6	0.1
12	7.74	7.86	7.96	7.9	0.0
13	7.40	7.30	7.60	7.4	0.0

Overall average = $\bar{X}$ = 7.87

Pooled single-operator variance = s_r^2 = 0.04

Variance of laboratory averages = $s_{\bar{X}}^2$ = 0.26

Between-laboratory component of variance = s_L^2 = 0.24

Table A.9. Single-operator and between laboratory analysis for SFA-R – bound water content

Laboratory	Data

	a	b	c	Average	Single Operator Variance
1	4.36	4.47	4.63	4.49	0.02
2	5.99	5.60	6.25	5.95	0.11
3	4.27	5.03	5.04	4.78	0.20
4	4.28	4.14	5.06	4.49	0.25
5	4.94	4.62	4.67	4.74	0.03
6	4.30	4.36	4.44	4.37	0.00
7	5.20	5.30	4.90	5.13	0.04
8	6.10	6.57	6.72	6.46	0.10
9	4.97	4.82	4.87	4.89	0.01
10	5.01	5.19	5.06	5.09	0.01
11	4.76	4.76	4.76	4.76	0.00
12	5.89	5.62	5.94	5.82	0.03
13	5.10	4.90	5.20	5.07	0.02

Overall average = $\bar{X}$ = 5.08

Pooled single-operator variance = s_r^2 = 0.06

Variance of laboratory averages = $s_{\bar{X}}^2$ = 0.40

Between-laboratory component of variance = s_L^2 = 0.38

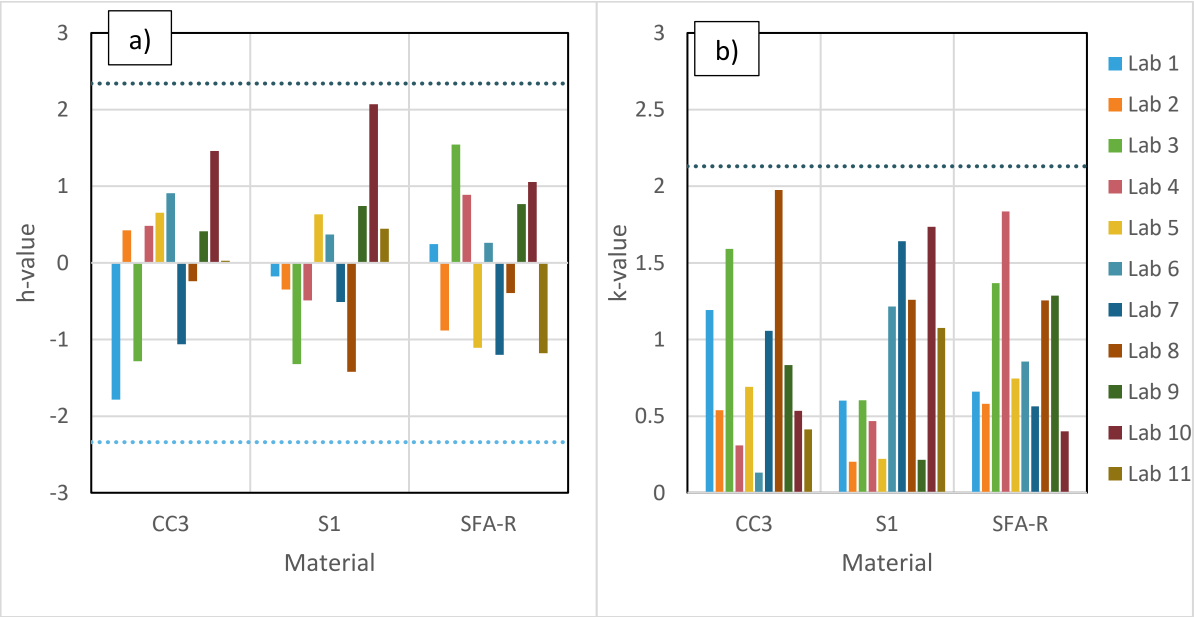

Figure A.1. h-values (a, left graph) and k-values (b, right graph) grouped by material for the 3 days hydration heat test determinations. The critical h- and k-values are indicated by the dotted lines.

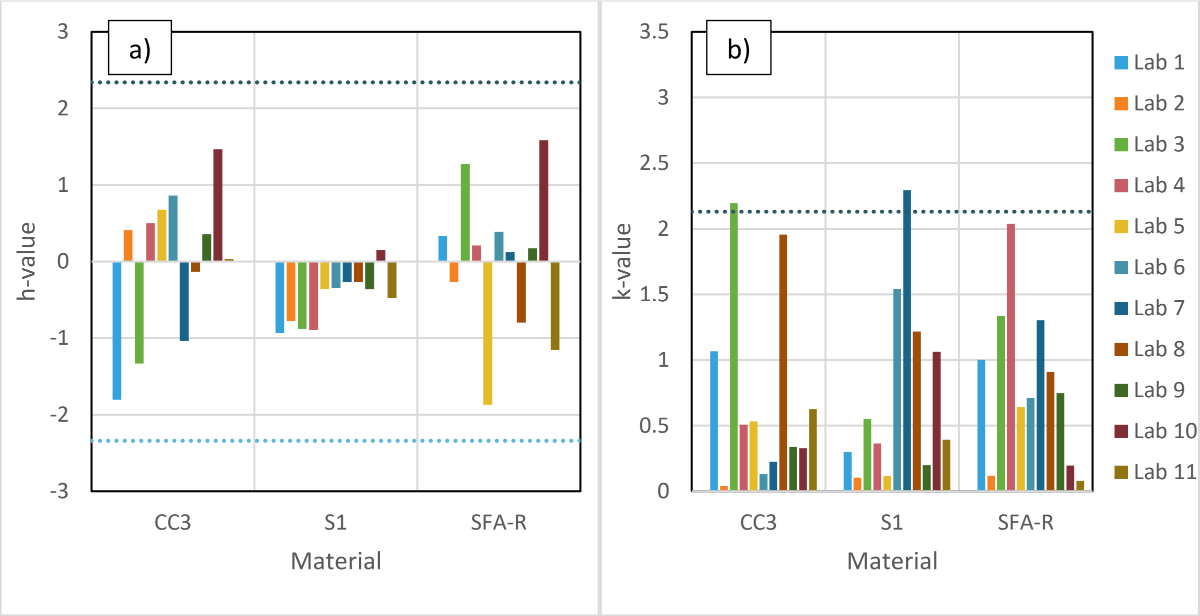

Figure A.2. h-values (a, left graph) and k-values (b, right graph) grouped by material for the 7 days hydration heat test determinations. The critical h- and k-values are indicated by the dotted lines.

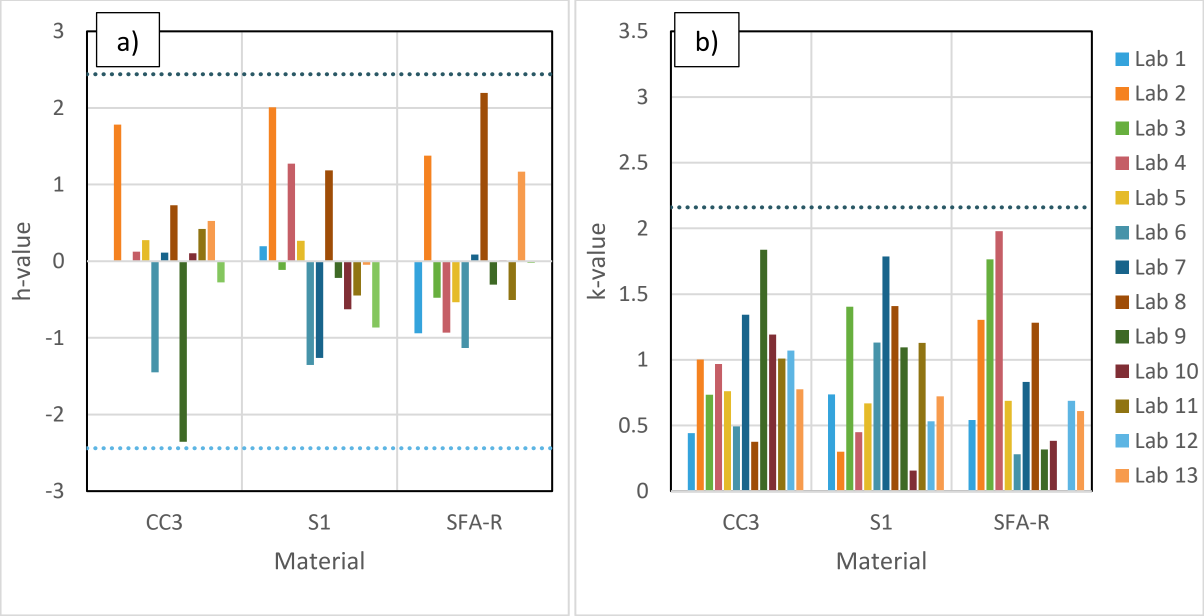

Figure A.4. h-values (a, left graph) and k-values (b, right graph) grouped by material for the bound water test determinations. The critical h- and k-values are indicated by the dotted lines.