

Methods of testing cement

Part 8: Heat of hydration — Solution method

ICS 91.100.10

National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Foreword

This document (EN 196-8:2010) has been prepared by Technical Committee CEN/TC 51, "Cement and building limes", the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

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This document supersedes EN 196-8:2003.

EN 196, *Methods of testing cement*, consists of the following parts:

- *Part 1: Determination of strength*
- *Part 2: Chemical analysis of cement*
- *Part 3: Determination of setting times and soundness*
- *Part 5: Pozzolanicity test for pozzolanic cement*
- *Part 6: Determination of fineness*
- *Part 7: Methods of taking and preparing samples of cement*
- *Part 8: Heat of hydration — Solution method*
- *Part 9: Heat of hydration — Semi-adiabatic method*
- *Part 10: Determination of the water-soluble chromium (VI) content of cement*
- CEN/TR 196-4, *Methods of testing cement — Part 4: Quantitative determination of constituents*

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1 Scope

This European Standard describes a method of determining the heat of hydration of cements by means of solution calorimetry, also known as the solution method. The heat of hydration is expressed in joules per gram of cement.

This standard is applicable to cements and hydraulic binders whatever their chemical composition.

NOTE 1 Another procedure, called the semi-adiabatic method, is described in EN 196-9. Either procedure can be used independently.

NOTE 2 It has been demonstrated that the best correlation between the two methods is obtained at seven days for the solution method (EN 196-8) compared with 41 h for the semi-adiabatic method (EN 196-9).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 197-1:2000, *Cement — Part 1: Composition, specifications and conformity criteria for common cements*

3 Principle

The method consists in measuring the heats of solution, in an acid mixture, of anhydrous cement and cement hydrated under standardized conditions for a predetermined period of time, e.g. seven days.

These standardized hydration conditions are as follows:

- water/cement ratio 0,40;
- use of neat cement paste;
- storage at constant temperature of $(20,0 \pm 0,2)$ °C during the whole hydration process.

The heat of hydration for each period, H_i , is obtained from the difference between the heat of solution of anhydrous cement, Q_a , and that of hydrated cement, Q_i .

4 Materials

4.1 Acid mixture

Analytical reagent quality acid mixture, obtained by adding 2,760 g of 40 % hydrofluoric acid (HF) for every 100,0 g of $(2,00 \pm 0,01)$ mol/l nitric acid (HNO_3), or 2,600 ml of hydrofluoric acid for every 100,0 ml of nitric acid.

WARNING — Hydrofluoric acid can cause painful skin burns which heal only with difficulty and precautions in handling this very corrosive substance should be strictly observed.

The quantity (mass or volume) of acid to be used, which is common to all tests, shall be measured to $\pm 0,2$ %.

4.2 Zinc oxide (ZnO)

Use zinc oxide of analytical quality to determine the thermal capacity of the calorimeter. Weigh 40 g to 50 g. Ignite at (950 ± 25) °C for 1 h. Cool in a desiccator. Grind to pass a 125 μm sieve. Store in a desiccator.

4.3 Anhydrous cement

Store anhydrous cement, from which metallic iron has been removed with a magnet, in a sealed container to avoid absorption of water or carbon dioxide. Bring the test sample to ambient temperature and carefully homogenize it before use.

4.4 Hydrated cement

Prepare the hydrated cement test sample by vigorously mixing, either manually or mechanically, $(100,0 \pm 0,1)$ g of anhydrous cement with $(40,0 \pm 0,1)$ g of distilled or deionised water for 3 min at ambient temperature. Place the resulting paste in plastics or glass cylindrical vials (three for each hydration period to be tested) so that each vial contains 15 g to 20 g of material. Seal the vials by means of a stopper and, if necessary, with paraffin wax or similar material and store them horizontally in a thermostatic bath maintained at a temperature of $(20,0 \pm 0,2)$ °C.

5 Apparatus

5.1 Calorimeter.

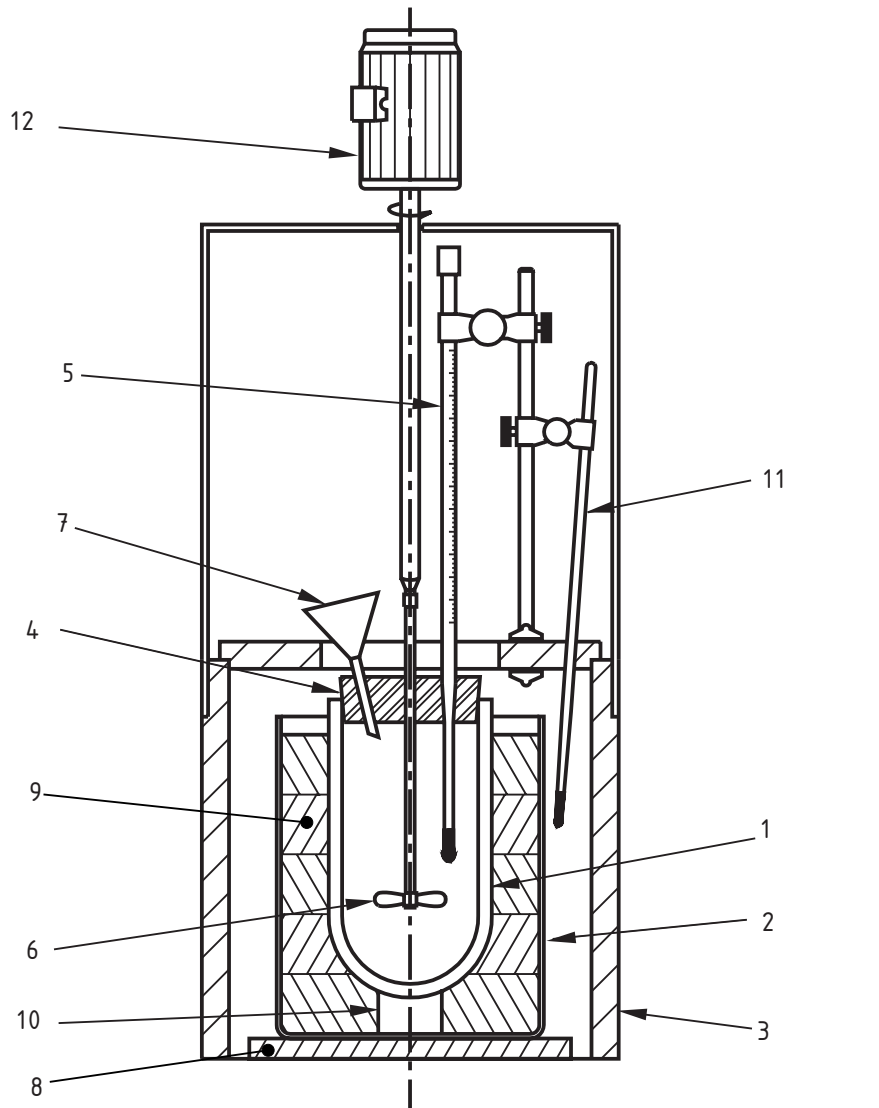
NOTE The method does not deal with the standardization of the calorimetric apparatus, or the measuring instruments. Insulated flasks with a volume of about 650 ml have proved to be suitable.

A suitable calorimeter (see Figure 1) comprises the following:

- a) **Dissolution vessel**, consisting of: an insulated flask (e.g. Dewar flask), placed either in a heat insulated container set inside a box constructed of insulating material (e.g. wood, plastics), or immersed in a thermostatic water bath regulated to $\pm 0,2$ °C; and an insulated stopper (made of cork or plastics) through which holes are provided for the thermometer, the stirrer and the funnel used for introducing the sample. The insulation of the calorimeter shall ensure that the thermal leakage coefficient, K (determined in accordance with 6.3), is less than 0,06 K per 15 min for each Kelvin above ambient temperature. The internal surface of the flask, that part of the thermometer immersed in the acid mixture and the lower part of the stopper, shall be acid mixture resistant.
- b) **Thermometer**, either a Beckmann thermometer with a 5 °C to 6 °C scale and subdivisions every 0,01 °C or other measurement apparatus of an equal or higher accuracy such as a thermistor or platinum resistance thermometer, positioned such that the end of the thermometer is at least 4 cm below the level of the liquid surface.

Express temperature readings with a resolution of $\pm 0,002$ °C. Adjust the zero of the Beckmann thermometer so that the upper limit of the scale is approximately the ambient, or water bath, temperature. Calibrate the thermometer in a thermostatic bath against a 0,01 °C graduated and calibrated thermometer.

- c) **Funnel**, of acid mixture resistant plastics, through which the sample is introduced into the flask and which extends below the lower part of the stopper by 5 mm to 6 mm and is sealed during the test.
- d) **Stirrer**, of acid mixture resistant plastics, positioned such that the blades are as close as possible to the bottom of the flask and rotated by a motor at a speed of (450 ± 50) min^{-1} . The motor shall be low power rated (e.g. a motor of a few watts) so as to prevent any excessive heat emission from affecting measurements.



Key

- | | | |
|-------------|---------------|------------------------|
| 1 flask | 5 thermometer | 9 insulating material |
| 2 container | 6 stirrer | 10 flask support |
| 3 box | 7 funnel | 11 ambient thermometer |
| 4 stopper | 8 support | 12 stirrer motor |

Figure 1 — Typical heat of solution calorimeter apparatus

5.2 Thermostatic bath, e.g. water bath, for storing the hydrated samples at a temperature of $(20,0 \pm 0,2)$ °C.

5.3 Mortar or electric grinder, for crushing the hydrated samples.

5.4 Plastics or glass vials, of capacity approximately 20 ml, for storing the hydrated paste.

5.5 Sieve, of mesh size 125 µm.

5.6 Sieve, of mesh size 600 µm.

5.7 Chronometer, graduated in seconds, for timing the temperature readings.

5.8 Two platinum crucibles, of capacity approximately 20 ml, for ignition of samples.

5.9 Electric furnace, naturally ventilated, capable of operating at (950 ± 25) °C, for ignition of samples.

5.10 Analytical balance, capable of weighing to an accuracy of $\pm 0,000$ 1 g.

5.11 Balance, of capacity 2 kg, capable of weighing to an accuracy of $\pm 0,2$ g.

6 Calorimeter calibration

6.1 Principle

Calibration of the calorimeter is carried out in order to determine its thermal capacity and thermal leakage coefficient. These characteristics are determined by dissolving the ignited zinc oxide (4.2) in the acid mixture (4.1) and measuring the temperature of the calorimeter at fixed intervals of time. The temperature of acid mixture shall be so set that after the dissolution reaction the calorimeter temperature is at least 0,5 °C below the ambient temperature. Where a water bath is used the temperature of the bath is considered to be the ambient temperature for the calorimeter.

6.2 Procedure

Measure a quantity of acid mixture (4.1) by mass or volume to $\pm 0,2$ % such that the liquid level will be approximately 2 cm below the calorimeter stopper. Place the acid mixture in the flask. Immediately before the determination of the thermal capacity, ignite the zinc oxide at (950 ± 25) °C for a maximum of 5 min and cool in a desiccator to room temperature. The quantity of zinc oxide to be used, weighed to $\pm 0,000$ 1 g, is that required to satisfy Equation (1):

$$\frac{\text{Mass of acid mixture}}{\text{Mass of zinc oxide}} = 60 \pm 1 \quad (1)$$

Carry out the procedure as follows:

- a) Preliminary period
Stir the acid mixture for 40 min to 50 min.
- b) Pre-period
When the rate of temperature increase is constant, start the timing using the chronometer (5.7) and record the initial temperature, \bar{T}_{-15}
- c) Sample introduction
After 15 min record the temperature, T_0 , and immediately add the zinc oxide sample to the acid mixture, taking not more than 1 min.

- d) Dissolution period
 Stir the mixture for 30 min, after which the dissolution is considered as being complete, and then record the temperature, \bar{T}_{30} . Record the ambient temperature, T_a . If the difference between T_a and \bar{T}_{30} is less than 0,5 °C then repeat the test.
- e) Post-period
 Record the final temperature \bar{T}_{45} after a further 15 min.

In order to reduce reading errors, determine temperatures \bar{T}_{-15} , \bar{T}_{30} and \bar{T}_{45} as the average of five different readings recorded at intervals of 1 min over the period 2 min before to 2 min after the prescribed time, T_i (i.e. T_{i-2} , T_{i-1} , T_i , T_{i+1} , T_{i+2}). Determine the value of T_0 by extrapolation of the function of temperature against time in the period T_{-4} to T_{-1} . If the extrapolated value differs from the T_0 reading by more than $\pm 0,002$ °C then replace T_0 by the extrapolated value. If more than a trace of zinc oxide is found adhering to the tip of the funnel or to the stopper when the calorimeter is opened, repeat the calibration.

NOTE The temperature readings will therefore effectively start 17 min before the zinc oxide sample is introduced and the last reading will take place 47 min after its introduction. The total duration of the calibration is 64 min.

6.3 Calculation of calibration characteristics

6.3.1 Corrected temperature increase, ΔT_c

Calculate the corrected temperature increase, ΔT_c , in Kelvins, from Equation (2):

$$\Delta T_c = (\bar{T}_{30} - T_0) - 2(\bar{T}_{45} - \bar{T}_{30}) \quad (2)$$

where

\bar{T}_{30} and \bar{T}_{45} are the average values of five measurements made at intervals of 1 min.

6.3.2 Thermal leakage coefficient, K

Calculate the thermal leakage coefficient, K , in Kelvins per 15 min per Kelvin temperature difference, (K/15 min·K⁻¹), from Equation (3):

$$K = \frac{(T_0 - \bar{T}_{-15}) - (\bar{T}_{45} - \bar{T}_{30})}{(\bar{T}_{30} - T_0)} \quad (3)$$

6.3.3 Thermal capacity, C

Calculate the thermal capacity, C , in joules per Kelvin, from Equation (4):

$$C = \frac{P}{\Delta T_c} [1\,077,43 + 0,364(30 - T_f) + 0,50(T_a - T_f)] \quad (4)$$

where

- P is the mass of zinc oxide, in grams (g);
- T_f is the temperature at the end of the dissolution period, in degrees Celsius (°C) (i.e. \bar{T}_{30} + temperature in degrees Celsius corresponding to the zero on the Beckmann thermometer);
- T_a is the temperature (i.e. ambient temperature) of zinc oxide when introduced into the calorimeter, in degrees Celsius (°C);

1 077,43 is the heat of solution of zinc oxide at 30 °C, in joules per gram ($\text{J}\cdot\text{g}^{-1}$);

- 0,364 is the temperature coefficient of the heat of solution of zinc oxide, in joules per gram per Kelvin ($\text{J}\cdot\text{g}^{-1}\cdot\text{K}^{-1}$);

0,50 is the specific heat of zinc oxide, in joules per gram per Kelvin ($\text{J}\cdot\text{g}^{-1}\cdot\text{K}^{-1}$).

Calculate the thermal capacity, C , expressed to two decimal places, and the thermal leakage coefficient, K , expressed to four decimal places, as the mean values of five calibrations of the calorimeter. If K is not less than 0,06 K per 15 min per Kelvin temperature difference then the calorimeter does not meet the requirements (see 5.1, a)).

NOTE Calibration characteristics should be redetermined whenever:

- the thermometer has been recalibrated;
- either the thermometer, the stirrer or the flask have been renewed or modified;
- the operator considers that it is necessary.

7 Determination of heat of solution

7.1 Heat of solution of anhydrous cement

7.1.1 Procedure

7.1.1.1 General

Use acid mixture of the same composition, quantity and initial temperature as used for calibration of the calorimeter (see 6.2). The quantity of sample, weighed to $\pm 0,000$ 1 g, is the amount required to satisfy Equation (5):

$$\frac{\text{Mass of acid mixture}}{\text{Mass of anhydrous cement}} = 140 \pm 2 \quad (5)$$

After the preliminary stirring period of the acid mixture (see 6.2, a)), follow the procedures given in 7.1.1.2 applicable to all cements and hydraulic binders or 7.1.1.3 applicable only to Portland cements.

7.1.1.2 Procedure applicable to all cements and hydraulic binders

Record the temperature, \bar{T}_{-15} , and start the chronometer. Record the temperature, T_0 , after 15 min and immediately introduce the sample, taking not more than 1 min. Record the temperature, \bar{T}_{30} , after a dissolution period of 30 min.

Follow the procedure for reading temperature given in 6.2 for the anhydrous sample, to avoid reading errors.

7.1.1.3 Portland cement (CEM I according to EN 197-1:2000)

Record the temperature, T_0 , start the chronometer and immediately introduce the sample, taking not more than 1 min. After a dissolution period of 30 min record the temperature, \bar{T}_{30} . After a further post-period of 15 min record the temperature, \bar{T}_{45} .

Follow the procedure for reading temperature given in 6.2 for the anhydrous sample, to avoid reading errors.

7.1.2 Calculation

7.1.2.1 Corrected temperature increase

On the basis of the temperature readings taken in accordance with 7.1.1.2 or 7.1.1.3 calculate ΔT_c from the Equations (6) or (7):

- a) All cements and hydraulic binders

$$\Delta T_c = (\bar{T}_{30} - T_0) - 2[(T_0 - \bar{T}_{15}) - K(\bar{T}_{30} - T_0)] \quad (6)$$

where

K is the thermal leakage coefficient, in Kelvins per 15 min per Kelvin temperature difference, ($K/15 \text{ min} \cdot K^{-1}$).

- b) Portland cements (CEM I):

$$\Delta T_c = (\bar{T}_{30} - T_0) - 2(\bar{T}_{45} - \bar{T}_{30}) \quad (7)$$

7.1.2.2 Heat of solution

Calculate the heat of solution of anhydrous cement, \bar{Q}_a , in joules per gram ($J \cdot g^{-1}$), from Equation (8):

$$\bar{Q}_a = \frac{C \times \Delta T_c}{P} + 0,8 (T_f - T_a) + 0,8 (T_f - 20) \quad (8)$$

where

ΔT_c is the corrected temperature increase, in Kelvins (K);

C is the thermal capacity of the calorimeter, in joules per Kelvin ($J \cdot K^{-1}$);

P is the mass of anhydrous cement, in grams (g);

T_f is the temperature at the end of the anhydrous cement dissolution period, in degrees Celsius ($^{\circ}C$);

T_a is the temperature of the anhydrous cement (i.e. ambient temperature) when introduced into the calorimeter, in degrees Celsius ($^{\circ}C$);

0,8 is the specific heat of the anhydrous cement, in joules per gram per Kelvin ($J \cdot g^{-1} \cdot K^{-1}$);

- 0,8 is the temperature coefficient of the heat of solution of the anhydrous cement, in joules per gram per Kelvin ($J \cdot g^{-1} \cdot K^{-1}$).

NOTE The last term in the equation has been introduced so as to correct the value of the heat of solution for any divergence from the reference temperature, 20 $^{\circ}C$.

7.1.3 Expression of results

Express the heat of solution of the anhydrous cement, \bar{Q}_a , as the mean of two measurements, expressed to one decimal place. If the difference between the two measurements is greater than $14 J \cdot g^{-1}$ carry out a third test. Reject any result which differs from the mean of these three values by more than $\pm 7 J \cdot g^{-1}$. If one result is rejected, calculate the mean from the remaining two values.

NOTE The standard deviation of repeatability, σ_r , for the measurement of heat of solution of anhydrous cement is $5 \text{ J}\cdot\text{g}^{-1}$. Therefore, results of two properly conducted tests by the same operator on samples of the same cement should not differ from each other by more than $14 \text{ J}\cdot\text{g}^{-1}$.

7.2 Heat of solution of hydrated cement

7.2.1 Procedure

Remove the sample of hydrated cement (4.4) from the vial, and crush quickly, so that the whole sample passes the $600 \mu\text{m}$ sieve (5.6). When crushing by means of a rapid crusher operate this for $(45 \pm 15) \text{ s}$. Complete the reduction in not more than 15 min so minimising contact with air and avoiding carbon dioxide absorption. Alternatively these procedures can be undertaken in a nitrogen atmosphere in a glove box.

Place the sample in a sealed container and homogenise by shaking, either manually or mechanically. Weigh the three samples from the same vial, required for the calorimetric determination and determination of bound water correction, in quick sequence to avoid loss of water or carbon dioxide absorption. Increase the quantity of the hydrated sample used to determine the heat of solution by 40 % over that used to test the anhydrous sample. Weigh the hydrated sample to $\pm 0,0001 \text{ g}$. Carry out the calorimetric determination as for the anhydrous cement (see 7.1.1).

Commence the heat of solution determination within the following times according to the specified hydration age:

- a) $\pm 30 \text{ min}$ for hydration ages less than three days;

NOTE In the case of time inconsistency when using a single calorimeter it is essential to use sample of hydrated cement taken from two separate mixes made at different time.

- b) $\pm 1 \text{ h}$ for hydration ages greater than, or equal to, three days and less than seven days;

- c) $\pm 2 \text{ h}$ for hydration ages greater than, or equal to, seven days.

7.2.2 Correction for bound water

Correct the mass of the hydrated sample to its anhydrous mass by determining the bound water by igniting portions of the same sample of the anhydrous cement and a sample of hydrated cement from a vial as used for the calorimetric determination. Weigh two 2 g samples to $\pm 0,0001 \text{ g}$. Ignite using platinum crucibles, at $(950 \pm 25) ^\circ\text{C}$ for 1 h, then cool in a desiccator to ambient temperature and weigh immediately.

The maximum deviation between the two determinations of percentage mass change on ignition for either the anhydrous, m_a , or hydrated, m_h , cement shall not be greater than 0,1 %.

NOTE 1 Alternative instrumental methods such as thermogravimetry or automatic water and carbon dioxide analysers may be used for this determination.

NOTE 2 The bound water correction may be determined from the calcium oxide (CaO) content by chemical analysis or X-ray fluorescence, instead of by ignition. Calcium oxide determination requires greater accuracy as an error in the calcium oxide content affects the heat of hydration by a factor of two compared with the percentage mass change on ignition method.

NOTE 3 If the cement contains oxidizable components then the determination by calcium oxide content should be used.

7.2.3 Calculation

7.2.3.1 Calculate the corrected temperature rise, ΔT_c , from Equations (6) and (7) for hydrated cement.

7.2.3.2 Calculate the heat of solution, \bar{Q}_i in joules per gram of hydrated cement, from Equation (9):

$$\bar{Q}_i = \frac{C \times \Delta T_c}{P \times F} + 1,7(T_f - T_a) + 1,3(T_f - 20) \quad (9)$$

where

P is the mass of hydrated cement, in grams (g);

T_f is the temperature at the end of the dissolution period of the hydrated sample, in degrees Celsius (°C);

T_a is the temperature of the hydrated cement (i.e. ambient temperature) when introduced into the calorimeter, in degrees Celsius (°C);

F is the correction factor for bound water;

1,7 is the specific heat of hydrated cement, in joules per gram per Kelvin ($\text{J} \cdot \text{g}^{-1} \cdot \text{K}^{-1}$);

- 1,3 is the temperature coefficient of the heat of solution of hydrated cement, in joules per gram per Kelvin ($\text{J} \cdot \text{g}^{-1} \cdot \text{K}^{-1}$).

Calculate the correction factor for bound water from Equation (10) or (11):

a) based on ignition:

$$F = \frac{100 - m_h}{100 - m_a} \quad (10)$$

where

m_h is the mass change on ignition of the hydrated sample, in percent by mass (%);

m_a is the mass change on ignition of the anhydrous sample, in percent by mass (%);

b) based on the lime content:

$$F = \frac{c_h}{c_a} \quad (11)$$

where

c_h is the calcium oxide content of the hydrated sample, in percent by mass (%);

c_a is the calcium oxide content of the anhydrous sample, in percent by mass (%).

7.2.4 Expression of results

Express the heat of solution of the hydrated sample, \bar{Q}_i , as the mean of two measurements, expressed to one decimal place. For each measurement use a new sample, taken from a different vial of the same set (see 4.4).

NOTE The precision and acceptance limits for results of determination of heat of solution of the hydrated sample are the same as those given for anhydrous cement (see 7.1.3).

8 Heat of hydration

8.1 Calculation of results

Calculate the heat of hydration of cement, H_i , expressed in joules per gram and referenced to 20 °C, as the difference between the heats of solution of anhydrous and hydrated cement obtained in accordance with 7.1.2 and 7.2.3 from Equation (12):

$$H_i = \overline{Q}_a - \overline{Q}_i \quad (12)$$

8.2 Reporting of results

Report the results for the heat of hydration, H_i , expressed in joules per gram of cement, to the nearest whole number.

8.3 Precision

8.3.1 Repeatability

The standard deviation of repeatability, σ_r , of the heat of hydration is 8 J·g⁻¹.

Therefore, if two results of properly conducted tests from the same laboratory on samples of the same cement are compared, they should not differ from each other by more than 22 J·g⁻¹.

8.3.2 Reproducibility

The standard deviation of reproducibility, σ_R , is 18 J·g⁻¹.

Therefore, if the results of two properly conducted tests from two different laboratories on samples of the same cement are compared, they should not differ from each other by more than 50 J·g⁻¹.

Bibliography

- [1] EN 196-9, *Methods of testing cement — Part 9: Heat of hydration — Semi-adiabatic method*

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