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Methods of testing cement — Determination of the heat of hydration —

Part 1:
Solution method

Méthodes d'essai des ciments — Détermination de la chaleur d'hydratation —

Partie 1: Méthode par dissolution

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 29582-1 was prepared by Technical Committee ISO/TC 74, *Cement and lime*.

ISO 29582 consists of the following parts, under the general title *Methods of testing cement — Determination of the heat of hydration*:

— *Part 1: Solution method*

— *Part 2: Semi-adiabatic method*

Methods of testing cement — Determination of the heat of hydration —

Part 1:
Solution method

1 Scope

This part of ISO 29582 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 part of ISO 29582 is applicable to cements and hydraulic binders, whatever their chemical composition.

NOTE 1 Another procedure, called the semi-adiabatic method, is described in ISO 29582-2. Either procedure can be used independently.

NOTE 2 It has been demonstrated that the best correlation between the two methods is obtained at 7 d for the solution method in this part of ISO 29582 compared with 41 h for the semi-adiabatic method in ISO 29582-2.

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, *Cement — Part 1: Composition, specifications and conformity criteria for common cements*

3 Principle

The method consists of 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. 7 days.

These standardized hydration conditions are as follows:

- water/cement ratio of 0,40;
- use of neat cement paste;
- storage at a constant temperature of (20,0 ± 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 Reagents and materials

4.1 Acid mixture, analytical-reagent quality, obtained by adding 2,760 g of 40 % hydrofluoric acid (HF) per 100,0 g of (2,00 ± 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.

4.2 Acid mixture, proposed alternative, containing ammonium fluoride (NH_4F) instead of hydrofluoric acid, prepared as follows.

Place about 400 g of (2,00 ± 0,01) mol/l nitric acid and 7,2 g of ammonium fluoride in the flask. Then, add (2,00 ± 0,01) mol/l nitric acid to make a total mass of 425,0 g ± 0,1 g. Any other procedure in the preparation of acid mixture can be adopted, provided that the same mass ratio of nitric acid to ammonium fluoride is conserved.

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

In the case of a dispute , only the reference acid mixture containing hydrofluoric acid is used.

4.3 Zinc oxide (ZnO), analytical quality, used to determine the thermal capacity of the calorimeter and prepare as follows.

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.

4.4 Anhydrous cement, from which metallic iron has been removed with a magnet, stored 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.

4.5 Hydrated cement, test sample prepared as follows.

Vigorously mix, either manually or mechanically, (100,0 ± 0,1) g of anhydrous cement with (40,0 ± 0,1) g of distilled or deionized water for 3 min at ambient temperature. Place the resulting paste in plastic or glass cylindrical vials (three for each hydration period being 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 ± 0,2) °C.

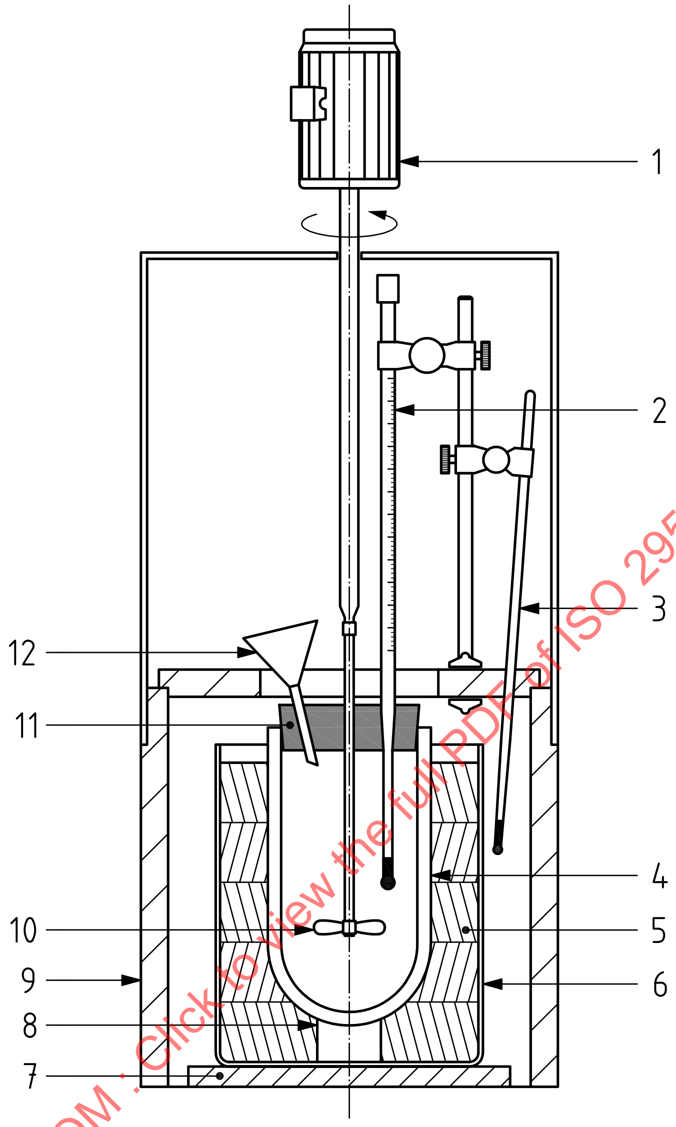
5 Apparatus

5.1 Calorimeter, comprised of the following (see Figure 1).

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.

5.1.1 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 ± 0,2 °C; and an insulated stopper (made of cork or plastic) 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.



Key

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

Figure 1 — Typical heat-of-solution calorimeter apparatus

5.1.2 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 ± 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.

5.1.3 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.

5.1.4 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 have a low power rating (e.g. a motor of a few watts) so as to prevent any excessive heat emission from affecting measurements.

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

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

5.4 Plastic 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 ± 0,000 1 g.

5.11 Balance, of capacity 2 kg, capable of weighing to an accuracy of ± 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 the 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 ± 0,2 % such that the liquid level is 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 mass of zinc oxide, m_{ZnO}, being used, weighed to ± 0,000 1 g, is that required to satisfy Equation (1):

$$\frac{m_{\text{acid}}}{m_{\text{ZnO}}} = 60 \pm 1 \qquad (1)$$

where m_{acid} is the mass of the acid mixture (4.1).

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.

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, $\overline{T}_{30}$. Record the ambient temperature, T_a. If the difference between T_a and $\overline{T}_{30}$ is less than 0,5 °C, then repeat the test.

e) Post-period: Record the final temperature, $\overline{T}_{45}$, after an additional 15 min.

In order to reduce reading errors, determine temperatures $\overline{T}_{-15}$, $\overline{T}_{30}$ and $\overline{T}_{45}$ 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 ± 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, therefore, effectively start 17 min before the zinc oxide sample is introduced and the last reading takes 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, expressed in kelvin, from Equation (2):

$$\Delta T_c = (\overline{T}_{30} - T_0) - 2\,(\overline{T}_{45} - \overline{T}_{30}) \qquad (2)$$

where $\overline{T}_{30}$ and $\overline{T}_{45}$ 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 kelvin per 15 min per kelvin temperature difference, (K/15 min·K^{-1}), from Equation (3):

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

6.3.3 Thermal capacity, c

Calculate the thermal capacity, c, expressed in joules per kelvin, from Equation (4):

$$c = \frac{m_{\text{ZnO}}}{\Delta T_{\text{c}}} [1\,077{,}43 + 0{,}364\,(30 - T_{\text{f}}) + 0{,}50\,(T_{\text{a}} - T_{\text{f}})] \qquad (4)$$

where

m_{ZnO}	is the mass of zinc oxide, expressed in grams;
T_{f}	is the temperature at the end of the dissolution period, i.e. $\overline{T}_{30} + T_{\text{Beck}}$, where T_{Beck} corresponds to the zero on the Beckmann thermometer, with all temperatures expressed in degrees Celsius;
T_{a}	is the temperature (i.e. ambient temperature) of zinc oxide when introduced into the calorimeter, expressed in degrees Celsius;
1 077,43	is the heat of solution of zinc oxide at 30 °C, expressed in joules per gram (J·g^{-1});
–0,364	is the temperature coefficient of the heat of solution of zinc oxide, expressed in joules per gram per kelvin (J·g^{-1}·K^{-1});
0,50	is the specific heat of zinc oxide, expressed in joules per gram per kelvin (J·g^{-1}·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 5.1.1.

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

Use acid mixture of the same composition, quantity and initial temperature as used for calibration of the calorimeter; see 6.2. The quantity of the anhydrous cement sample, m_{a}, weighed to ± 0,000 1 g, is the amount required to satisfy Equation (5):

$$\frac{m_{\text{acid}}}{m_{\text{a}}} = 140 \pm 2 \qquad (5)$$

where m_{acid} is the mass of the acid mixture (4.1). After the preliminary stirring period of the acid mixture [see 6.2 a)], follow the procedures given in 7.1.1.1 applicable to all cements and hydraulic binders or 7.1.1.2 applicable only to Portland cements.

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

7.1.1.2 Portland cement

This is designated CEM I in accordance with EN 197-1.

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 the 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 7.1.1.1 or 7.1.1.2, calculate ΔT_c from Equation (6), for all cements and hydraulic binders, or Equation (7), for portland cements (CEM I):

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

where K is the thermal leakage coefficient, expressed in kelvin per 15 min per kelvin temperature difference, (K/15 min·K^{-1}).

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

7.1.2.2 Heat of solution

Calculate the heat of solution of anhydrous cement, $\bar{Q}_a$, expressed in joules per gram, from Equation (8):

$$\bar{Q}_a = \frac{c \cdot \Delta T_c}{m_a} + 0{,}8\left(T_f - T_a\right) + 0{,}8\left(T_f - 20\right) \qquad (8)$$

where

ΔT_c	is the corrected temperature increase, expressed in kelvins;
c	is the thermal capacity of the calorimeter, expressed in joules per kelvin (J·K^{-1});
m_a	is the mass of anhydrous cement, expressed in grams;
T_f	is the temperature at the end of the anhydrous cement dissolution period, expressed in degrees Celsius;
T_a	is the temperature of the anhydrous cement (i.e. ambient temperature) when introduced into the calorimeter, expressed in degrees Celsius;
0,8	is the specific heat of the anhydrous cement, expressed in joules per gram per kelvin (J·g^{-1}·K^{-1});
–0,8	is the temperature coefficient of the heat of solution of the anhydrous cement, expressed in joules per gram per kelvin (J·g^{-1}·K^{-1}).

NOTE The last term in the equation is introduced so as to correct the value of the heat of solution for any divergence from the reference temperature, 20 °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·g^{-1}, carry out a third test. Reject any result that differs from the mean of these three values by more than ± 7 J·g^{-1}. If one result is rejected, calculate the mean from the remaining two values.

The standard deviation of repeatability, σ_r, for the measurement of heat of solution of anhydrous cement is 5 J·g^{-1}. Therefore, the 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 J·g^{-1}.

7.2 Heat of solution of hydrated cement

7.2.1 Procedure

Remove the sample of hydrated cement (4.5) from the vial and crush quickly, so that the whole sample passes the 600 µm sieve (5.6). When crushing by means of a rapid crusher, operate this for (45 ± 15) s. Complete the reduction in not more than 15 min to minimize contact with air and avoid 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 homogenize 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 water loss 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 ± 0,000 1 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) ± 30 min for hydration ages less than 3 d[1];

b) ± 1 h for hydration ages greater than or equal to 3 d and less than 7 d;

c) ± 2 h for hydration ages greater than or equal to 7 d.

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 ± 0,000 1 g. Ignite using platinum crucibles, at (950 ± 25) °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 %.

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

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[1] In the case of time inconsistency when using a single calorimeter, it is necessary to use a sample of hydrated cement taken from two separate mixes made at different times.

The bound water correction may be determined from the calcium oxide (CaO) content by chemical analysis or X-ray fluorescence, instead of by ignition. The 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.

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 increase, ΔT_c, from Equations (6) and (7) for the hydrated cement.

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

$$\bar{Q}_\mathrm{i} = \frac{c \cdot \Delta T_\mathrm{c}}{m_\mathrm{h} \cdot F} + 1{,}7\left(T_\mathrm{f} - T_\mathrm{a}\right) + 1{,}3\left(T_\mathrm{f} - 20\right) \tag{9}$$

where

m_h is the mass of hydrated cement, expressed in grams;

T_f is the temperature at the end of the dissolution period of the hydrated sample, expressed in degrees Celsius;

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

F is the correction factor for bound water;

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

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

Calculate the correction factor, F, for bound water from Equation (10), based on ignition, or Equation (11), based on the lime content:

$$F = \frac{100 - \Delta m_\mathrm{h}}{100 - \Delta m_\mathrm{a}} \tag{10}$$

where

Δm_h is the mass change on ignition of the hydrated sample, expressed in percent by mass;

Δm_a is the mass change on ignition of the anhydrous sample, expressed in percent by mass.

$$F = \frac{w_\mathrm{h}}{w_\mathrm{a}} \tag{11}$$

where

w_h is the calcium oxide content of the hydrated sample, expressed in percentage mass fraction;

w_a is the calcium oxide content of the anhydrous sample, expressed in percentage mass fraction.

7.2.4 Expression of results

Express the heat of solution of the hydrated sample, $\bar{Q}_{\mathrm{i}}$, 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 4.5).

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 heat of solution of anhydrous and hydrated cement obtained in accordance with 7.1.2 and 7.2.3 from Equation (12):

$$H_{\mathrm{i}} = \bar{Q}_{\mathrm{a}} - \bar{Q}_{\mathrm{i}} \qquad (12)$$

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 $\mathrm{J \cdot g^{-1}}$.

Therefore, if the results of two 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 $\mathrm{J \cdot g^{-1}}$.

8.3.2 Reproducibility

The standard deviation of reproducibility, σ_{R}, is 18 $\mathrm{J \cdot g^{-1}}$.

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 $\mathrm{J \cdot g^{-1}}$.

9 Test report

9.1 The test report shall include the following:

a) identification of the test specimen;

b) date of test;

c) heat of solution of the hydrated sample, H_{i}, expressed in joules per gram of cement, to the nearest whole number;

d) any deviations from the standard method of testing;

e) declaration from the person technically responsible for the test that the testing was carried out in accordance with this part of ISO 29582, except as detailed in item d).