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cement — Solution method**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Cement Association (JCA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS R 5203:1995** is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

Determination of the heat of hydration of cement — Solution method

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **ISO 29582-1** published in 2009 without modification in the basic measuring principle, but with addition of unique contents of **JIS** that are: tolerances for the measurement of heat of solution of hydrated cement and solution conditions for respective types of cement.

The portions with continuous sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with the explanations is given in Annex JA. The comparison table between previous and current editions of this Standard on technically significant revisions is given in Annex JB.

1 Scope

This Standard specifies the testing method for heat of hydration of portland cement, portland blast-furnace slag cement, and portland fly-ash cement.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 29582-1 : 2009 *Methods of testing cement — Determination of the heat of hydration — Part 1 : Solution method* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

- JIS K 0050 *General rules for chemical analysis*
- JIS K 8001 *General rule for test methods of reagents*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8405 *Zinc oxide (Reagent)*
- JIS K 8541 *Nitric acid (Reagent)*
- JIS Z 8801-1 *Test sieves — Part 1 : Test sieves of metal wire cloth*

3 General requirements for test

3.1 Expression of measurement results

Round the measurement results to the digit specified in the relevant subclause.

3.2 Tolerances

In the measurement of thermal capacity, measurement of heat of solution of anhydrous cement and measurement of heat of solution of hydrated cement, when the difference between the results of two measurements is greater than the specified tolerance, carry out a third measurement, and report the mean of the two measurements the difference of which satisfies the tolerance.

If, among measurements including the third measurement, there are two pairs of measurements satisfying the tolerance, report the mean of all the three measurements. If, in the three measurements, there are no pair of measurements satisfying the tolerance, carry out the measurement from the start.

Tolerance for each measurement item shall be as specified in the relevant subclause.

4 Reagents and sample

4.1 Nitric acid (2 mol/L), of guaranteed grade specified in JIS K 8541 or equivalent or better reagent, measured in the amount specified in Table 1, to which distilled water or ion-exchanged water is added to make 20 L.

Table 1 Amount of nitric acid weighed out

Density of nitric acid (g/ml)	Amount weighed out (ml)
1.42	2 550
1.40	2 760
1.38	2 980

After mixing the prepared nitric acid (2 mol/L) thoroughly and cooling it to room temperature, check if its concentration is $2.000 \text{ mol/L} \pm 0.004 \text{ mol/L}$ according to either of the following methods a) or b). When the concentration of nitric acid (2 mol/L) is outside the range, make adjustment by dripping concentrated nitric acid or adding distilled water or ion-exchanged water, and check the concentration again. Carry out this operation until the concentration of nitric acid (2 mol/L) becomes within the predetermined range.

- a) Heat sodium carbonate of the reference material for volumetric analysis specified in JIS K 8005 at $600 \text{ }^{\circ}\text{C} \pm 10 \text{ }^{\circ}\text{C}$ for 1 h and cool it. Immediately weigh out about 2 g of this material correctly to the nearest 0.000 1 g and transfer to a beaker. Add about 200 ml of distilled water or ion-exchanged water to dissolve the material. Add a few drops of methyl orange (1 g/L) as an indicator, and standardize it by dropping nitric acid solution through a burette.

Calculate the concentration of nitric acid (2 mol/L) from Equation (1).

$$N = \frac{18.868 \times m \times (a/100)}{v} \dots\dots\dots (1)$$

where, N : concentration of nitric acid (2 mol/L) (mol/L)
 m : mass of sodium carbonate weighed out (g)
 a : purity of sodium carbonate weighed out (%)
 v : titre of nitric acid (2 mol/L) (ml)

- b) Using a transfer pipette, take correctly 15 ml or 20 ml of nitric acid (2 mol/L) into a beaker, add a few drops of phenolphthalein indicator (1 g/L-ethanol solution), titrate it by either a 1 mol/L sodium hydroxide solution prepared and standardized according to **JA.6.4 r) 1)** of Annex JA of **JIS K 8001** or a commercially available 1 mol/L sodium hydroxide solution standardized in the same way and obtain the concentration of nitric acid (2 mol/L) from Equation (2).

$$N = \frac{v_2 \times f}{v_1} \dots\dots\dots (2)$$

where, N : concentration of nitric acid (2 mol/L) (mol/L)
 v_1 : aliquot of nitric acid used (2 mol/L) (ml)
 v_2 : titre of 1 mol/L sodium hydroxide solution (ml)
 f : factor of 1 mol/L sodium hydroxide solution

4.2 Ammonium fluoride, 95 % or higher in purity.

Since it has deliquescence, it shall be stored in a silica gel desiccator.

4.3 Zinc oxide, guaranteed reagent specified in **JIS K 8405**, or equivalent or better reagent.

Heat zinc oxide to be used for the measurement of thermal capacity of a calorimeter at $950\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ for 1 h. After allowing it to cool in a silica gel desiccator, pass the whole volume through a test sieve of aperture size $150\text{ }\mu\text{m}$ or $125\text{ }\mu\text{m}$ specified in **JIS Z 8801-1** and store in the desiccator.

4.4 Anhydrous cement Extract cement so that it represents the average quality of the inspection unit and then reduce it to obtain a representative sample. Use the sampling and reduction methods determined by the agreement between the parties concerned with delivery. Further reduce the representative sample by a sample divider or method of quartering and take out about 500 g of this reduced sample.

Pass the sample taken out through a test sieve of aperture size $850\text{ }\mu\text{m}$ specified in **JIS Z 8801-1** to remove impurities and then as necessary, use magnet to remove iron. Store in a dampproof airtight container. Place this container in a laboratory prior to testing to bring it to the laboratory temperature.

4.5 Hydrated cement Weigh 250.0 g of anhydrous cement to the nearest 0.1 g , then weigh 100.0 g of distilled water or ion-exchanged water to the nearest 0.1 g and add it to the cement. Mix for 3 min either manually or mechanically, put the resulting paste in the required number of curing vials [three for each hydration period tested (includ-

ing the reserves), e.g. when the measurement is carried out at hydration age 7 d and at 28 d, six vials] so that each vial contains approximately the equal amount. Seal the vials by means of a stopper and store them in a thermostatic bath maintained at a temperature of $20.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$.

The amount of anhydrous cement and distilled or ion-exchanged water weighed out may be other than the above, provided that the same mass ratio (anhydrous cement 2.5, water 1) is observed. In this case, however, ensure that the amount of anhydrous cement weighed out is not less than 100 g.

5 Apparatus and instruments

5.1 Calorimeter, composed of components given in the following 5.1.1 to 5.1.5. See Figure 1 and Figure 2 for examples of its structure.

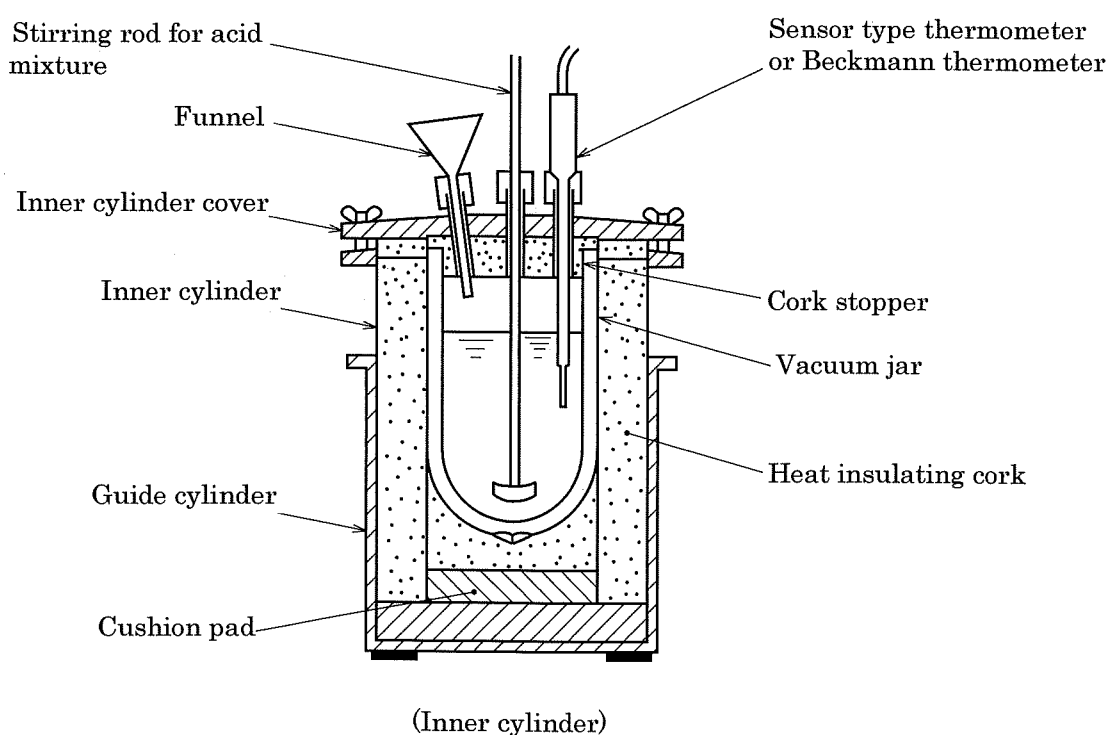
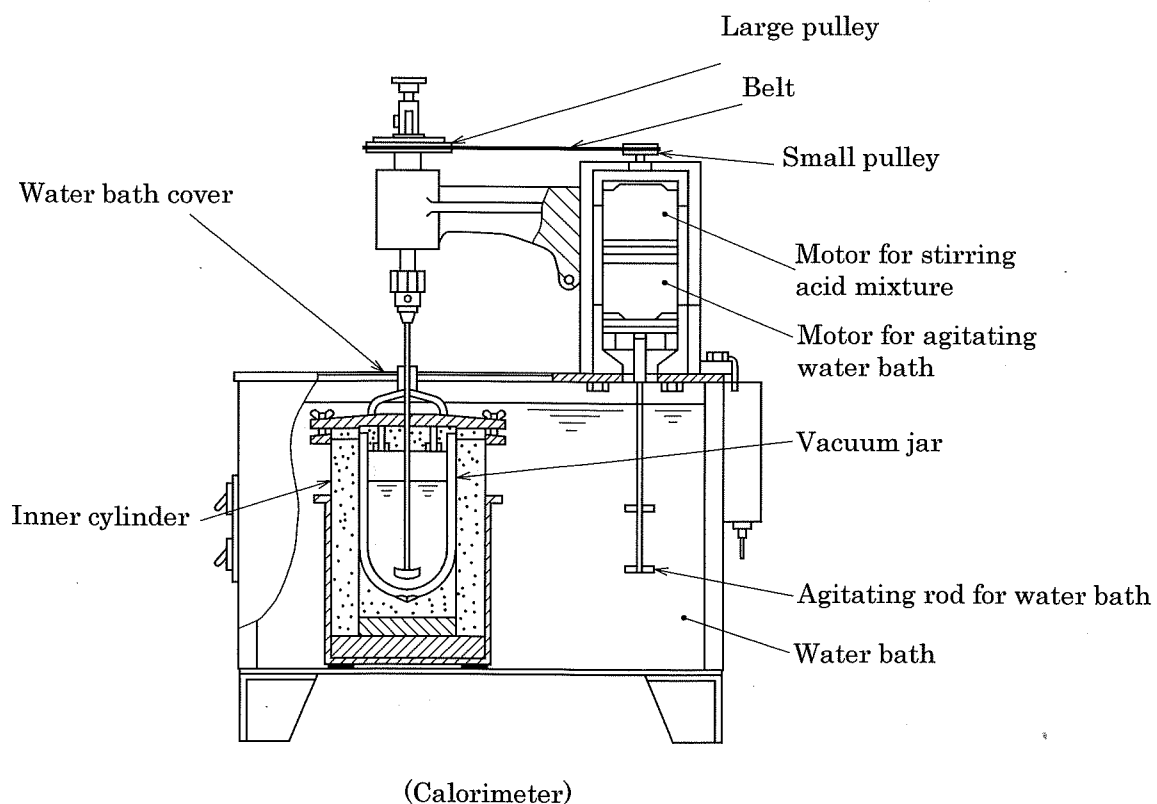
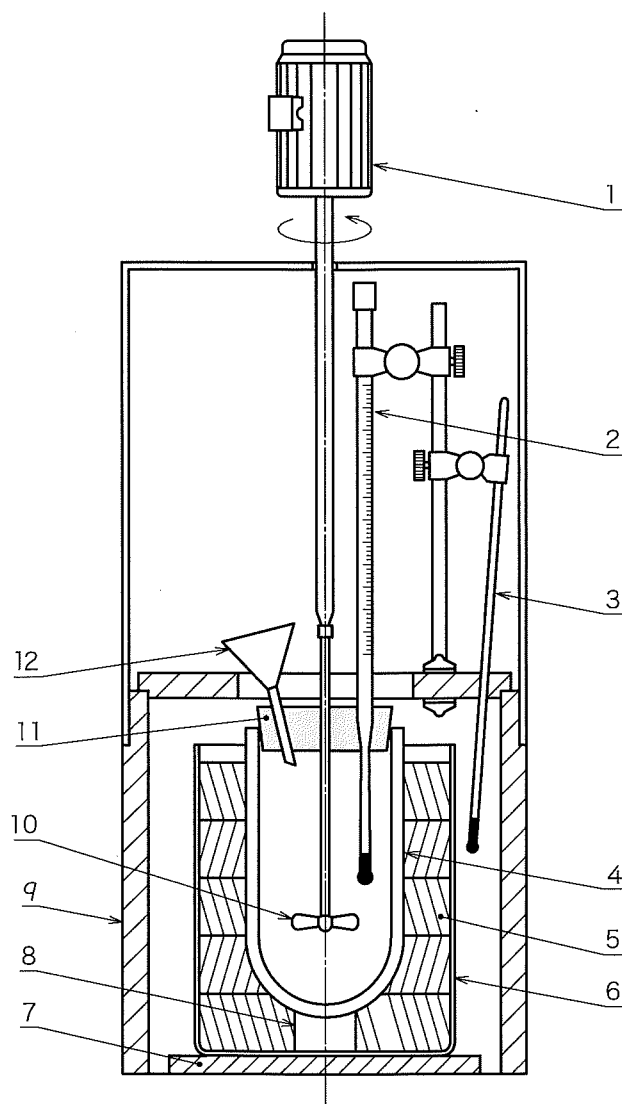


Figure 1 Example of calorimeter having a water bath



Key

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

Figure 2 Example of calorimeter having an air bath

5.1.1 Vacuum jar

5.1.1.1 Capacity of vacuum jar, amount of sample and amount of reagents The amount of sample and amount of reagents specified in this Standard are based on the assumption that a vacuum jar of about 75 mm in inside diameter, about 150 mm in depth and about 600 ml in internal volume is used for the test.

In addition, when a Beckmann thermometer is used as a thermometer for measuring the acid mixture temperature, the measurement shall be carried out with the amount

and volume specified in this Standard.

5.1.1.2 Specification of vacuum jar A vacuum jar of which the performance has been checked according to the following method shall be used.

Put about 425 ml of warm water maintained at a temperature higher by about 5 °C than room temperature in a vacuum jar, put a cork stopper, insert a thermometer for measuring the acid mixture temperature into the jar and read the temperature when the temperature drop becomes constant. After allowing it to stand for 30 min, read the temperature again and check that there is no temperature drop of 0.001 °C/min or more from the initial temperature per 1 °C difference between the warm water temperature and the ambient temperature of the vacuum jar. This ambient temperature, when the vacuum jar is put in an inner cylinder, shall be the temperature of the ambient of the inner cylinder.

The vacuum jar used shall have an inside surface coated with suitable hydrofluoric acid-resistant material (e.g. polyethylene, bees wax and fluororesin), and a heat insulated stopper (e.g. made of cork), through which three holes are provided for the thermometer, the stirrer and the funnel used for introducing the sample.

5.1.1.3 Inner cylinder The vacuum jar shall be put into an inner cylinder of which the inner surface is lined with heat insulating material like cork. When the inner cylinder is not used, a water bath of which the temperature can be controlled within ± 0.2 °C shall be used as the thermostatic bath of the calorimeter. The inner cylinder, when using a vacuum jar with about 600 ml inner volume specified in 5.1.1.1, shall be about 150 mm in inside diameter and about 230 mm in depth, and shall have an inner surface lined with heat insulating cork about 25 mm in thickness.

If the inner cylinder is put into the water bath, it shall be provided with a cover having three holes and the inner cylinder and the cover shall be fastened together using an O ring. The structure of the inner cylinder shall be such that water does not enter through the three holes of its cover.

5.1.2 Thermometer

5.1.2.1 Thermometer for measuring the acid mixture temperature Either a sensor type thermometer or a Beckmann thermometer shall be used.

The sensor-type thermometer shall have a resolution as accurate as 0.001 °C.

The Beckmann thermometer shall have a scale of about 6 °C and minimum graduation of 0.01 °C and shall be guaranteed.

The part of the thermometer inserted into the vacuum jar shall be coated with suitable hydrofluoric-acid-resistant material (e.g. polyethylene, bees wax and fluororesin).

The temperature of the acid mixture shall be measured to the nearest 0.001 °C. When the temperature of acid mixture is measured using a Beckmann thermometer, the reading on Beckmann thermometer shall be converted to the reading of the ordinary thermometer.

5.1.2.2 Thermometers for measuring other than acid mixture temperature A ther-

nometer with minimum graduation of $0.1\text{ }^{\circ}\text{C}$ that has been calibrated.

The temperature other than that of acid mixture shall be measured to the nearest $0.1\text{ }^{\circ}\text{C}$.

5.1.3 Funnel, through which the sample is introduced into the acid mixture, and is sealed during the test.

5.1.4 Stirring device, having a glass or metallic stirring rod, of which the part inserted into the vacuum jar is coated with hydrofluoric-acid-resistant material (e.g. polyethylene, bees wax and fluororesin). The material of the stirring rod may be plastics or resin resistant to acid mixture.

The stirring rod may be rotated by the ball bearing provided in its bearing axle and driven through pulley and belt, or by being attached directly to the motor shaft. In the latter device, the motor shall have a low power rating so as to prevent any excessive heat emission from affecting measurements.

The stirring device shall have a mechanism capable of rotating the stirring rod at a speed of (450 ± 50) revolutions per minute and keeping this speed constant during the test.

5.1.5 Thermostatic bath for calorimeter Air bath or water bath, for storing the vacuum jar contained in the inner cylinder at a temperature of $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

In the case of an air bath storing an inner cylinder (see Figure 2), the whole calorimeter may be placed in a thermostatic bath or constant temperature room maintained at $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

In the case of a water bath, an agitator for making the water temperature homogeneous shall be provided.

5.2 Thermostatic water bath for curing A water bath capable of curing the cement paste at a temperature of $20.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$.

5.3 Grinder for hydrated cement An electric grinder, or an iron or stainless steel mortar, if required.

5.4 Curing vials Polypropylene bottles for storing the cement paste, with an inner diameter of about 25 mm and height of about 55 mm, which can be sealed with a lid.

5.5 Sieves The sieve used for preparing zinc oxide to be used for determination of thermal capacity of the calorimeter shall be a test sieve of $150\text{ }\mu\text{m}$ or $125\text{ }\mu\text{m}$ aperture size specified in JIS Z 8801-1.

The sieve used for removing impurities in anhydrous cement sample shall be a test sieve of $850\text{ }\mu\text{m}$ aperture size specified in JIS Z 8801-1.

The sieve used for preparation of the powdered hydrated cement sample shall be a test sieve of $850\text{ }\mu\text{m}$ or $600\text{ }\mu\text{m}$ aperture size specified in JIS Z 8801-1.

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

5.7 Other apparatus and instruments Apparatus and instruments specified in JIS K

0050.

6 Measurement of thermal capacity of calorimeter

6.1 Preparation

Immediately before the measurement, it shall be checked that the temperature of the thermostatic bath of the calorimeter in which the inner cylinder is to be placed is $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

Then, place about 400 g of nitric acid (2 mol/L) in a vacuum jar while maintaining it at a temperature about $5\text{ }^{\circ}\text{C}$ lower than the laboratory temperature so that the temperature of the acid mixture after dissolution will become slightly lower than the laboratory temperature. Add $7.2\text{ g} \pm 0.1\text{ g}$ of ammonium fluoride, followed by nitric acid (2 mol/L), so that the obtained acid mixture is $425.0\text{ g} \pm 0.1\text{ g}$ in total mass. For weighing ammonium fluoride, a glass vessel shall not be used. Put the vacuum jar in the inner cylinder, cover it with a lid, and put the inner cylinder inside the thermostatic bath.

In the case of a water bath, stir the water with an agitator for 10 min to control the water temperature to $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$, and then stop the agitator.

After stirring the acid mixture in the vacuum jar for 20 min, measure the temperature of the acid mixture. Again stir the acid mixture for a further 5 min, and confirm that the temperature rise per minute is approximately constant during this period. If it is not found to be constant, stir the mixture for another 5 min and check the temperature rise. If, after this, the temperature rise still remains unconstant, terminate the test and check the thermal leakage coefficient of the calorimeter according to 6.3.

In the case of using a vacuum jar with an internal volume other than that specified in 5.1.1.1, the preparation may be performed by measuring a quantity of acid mixture such that the liquid level is approximately 2 cm below the stopper and weighing a quantity of sample such that the mass of the acid mixture and the sample is in the relation as specified in 6.2, 7.1 and 7.2.2.

NOTE : In the case of using a water bath, put water in it up to about 15 mm below the inner cylinder cover and control the temperature.

6.2 Measurement of thermal capacity

Heat the zinc oxide, the whole volume of which has been passed through a test sieve of aperture size $150\text{ }\mu\text{m}$ or $125\text{ }\mu\text{m}$ specified in JIS Z 8801-1 and stored in a silica gel desiccator, at $950\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ for 1 to 2 min immediately before the measurement, allow it to cool in the silica gel desiccator to the laboratory temperature and weigh out 7.0 g of this zinc oxide correctly to the nearest 0.000 1 g.

Measure the temperature of the acid mixture in the vacuum jar and take it as t_{co} . Immediately after weighing, put gently the zinc oxide through the funnel of the calorimeter at a constant rate taking 1 min to 2 min. When not being used for introduction of zinc oxide, the funnel shall be stoppered with a lid so that the vacuum cylinder can be kept from ingress of outside air.

Determine the thermal capacity using the method specified in 6.2.1 or 6.2.2, depending on the type of cement under test.

In the case of using a vacuum jar with an internal volume other than that specified in 5.1.1.1, the amount of zinc oxide weighed out shall be such that the following is true : (mass of acid mixture/amount of zinc oxide weighed out) = 60 ± 1 .

The thermal capacity of the calorimeter should be checked at regular intervals. In the events as described in the following, checking of the thermal capacity of the calorimeter is mandatory.

- The thermometer for measuring the acid mixture temperature has been renewed or calibrated.
- Either the vacuum jar, inner cylinder or the acid mixture stirring device comprising the calorimeter has been renewed.

6.2.1 In the case of portland cement and portland blast-furnace slag cement

Measure the temperature of the acid mixture at exactly 20 min and 40 min after beginning introduction of zinc oxide into the calorimeter, and take the measured temperatures as t_{c20} , t_{c40} , respectively.

Calculate the thermal capacity, c_1 , of the calorimeter from Equation (3).

Obtain thermal capacity readings to one decimal place, and after ensuring that the difference between the two measurements is within the specified tolerance, round the mean value of the two measurements to an integer and use it to calculate heat of hydration.

Tolerance checking of the difference between the two measurements shall be performed after rounding each measurement result to an integer.

The tolerance shall be 5 J/K.

$$c_1 = \frac{w[1\,072.0 + 0.4(30 - t_{c20}) + 0.5(t_c - t_{c20})]}{(t_{c20} - t_{c0}) - (t_{c40} - t_{c20})} \dots\dots\dots(3)$$

- where,
- c_1 : thermal capacity of calorimeter (J/K)
 - w : mass of zinc oxide (g)
 - t_c : laboratory temperature during introduction of zinc oxide into the calorimeter (°C)
 - t_{c0} : temperature of acid mixture immediately before introduction of zinc oxide into the calorimeter (°C)
 - t_{c20} : temperature of acid mixture 20 min after beginning introduction of zinc oxide into the calorimeter (°C)
 - t_{c40} : temperature of acid mixture 40 min after beginning introduction of zinc oxide into the calorimeter (°C)

NOTE : When using a Beckmann thermometer, the temperature measurement may be made five times at intervals of 1 min over the period 2 min before to 2 min after the prescribed time and the mean of the five measurements may be calculated.

6.2.2 In the case of portland fly-ash cement

Measure the temperature of the acid mixture exactly 80 min and 120 min after beginning the introduction of zinc oxide into the calorimeter, and take the measured temperatures as t_{c80} , t_{c120} , respectively.

Making use of these results, calculate the thermal capacity, c_2 , of the calorimeter from Equation (4).

Ensuring that the two measurement results are within the specified tolerance, round the mean of the two results to an integer and use it to calculate heat of hydration.

Tolerance checking of the difference between the two measurements shall be performed after rounding each measurement result to an integer.

The tolerance shall be 5 J/K.

$$c_2 = \frac{w[1\,072.0 + 0.4(30 - t_{c80}) + 0.5(t_c - t_{c80})]}{(t_{c80} - t_{c0}) - 2(t_{c120} - t_{c80})} \dots\dots\dots (4)$$

where, c_2 : thermal capacity of calorimeter (J/K)
 w : mass of zinc oxide (g)
 t_c : laboratory temperature during introduction of zinc oxide into the calorimeter (°C)
 t_{c0} : temperature of acid mixture immediately before introduction of zinc oxide into the calorimeter (°C)
 t_{c80} : temperature of acid mixture 80 min after beginning introduction of zinc oxide into the calorimeter (°C)
 t_{c120} : temperature of acid mixture 120 min after beginning introduction of zinc oxide into the calorimeter (°C)

6.3 Measurement of thermal leakage coefficient of calorimeter

In the cases where the vacuum jar or thermometer has been renewed, measure, as required, the temperature of the acid mixture additionally 10 min before introducing zinc oxide into the calorimeter at the time of measurement of the thermal capacity of the calorimeter, and determine the thermal leakage coefficient, K , from Equation (5) to verify that the calorimeter has an adequate performance.

K shall be less than 0.06 (K/20 min · K⁻¹).

$$K = \frac{2(t_{c0} - t_{c-10}) - (t_{c40} - t_{c20})}{(t_{c20} - t_{c0})} \dots\dots\dots (5)$$

where, K : thermal leakage coefficient (K/20 min · K⁻¹)
 t_{c-10} : temperature of acid mixture 10 min before introduction of zinc oxide into the calorimeter (°C)
 t_{c0} : temperature of acid mixture immediately before introduction of zinc oxide into the calorimeter (°C)
 t_{c20} : temperature of acid mixture 20 min after beginning introduction of zinc oxide into the calorimeter (°C)
 t_{c40} : temperature of acid mixture 40 min after beginning introduction of zinc oxide into the calorimeter (°C)

tion of zinc oxide into the calorimeter (°C)

7 Measurement of heat of solution

7.1 Measurement of heat of solution of anhydrous cement

Weigh out 3.0 g of anhydrous cement correctly to the nearest 0.000 1 g.

Prepare a calorimeter of which the thermal capacity has been determined in clause 6 according to 6.1, measure the temperature of the acid mixture in the vacuum jar, and take this temperature as t_{u0} . Carefully introduce the anhydrous cement, which has been weighed out, at a constant rate into the calorimeter using a funnel taking 1 min to 2 min.

Determine the heat of solution of anhydrous cement using the method specified in 7.1.1 or 7.1.2, depending on the type of cement under test.

In the case of using a vacuum jar with an internal volume other than that specified in 5.1.1.1, the amount of anhydrous cement weighed out shall be such that the following is true : (mass of acid mixture/amount of anhydrous cement weighed out) = 141 ± 1 .

7.1.1 In the case of portland cement and portland blast-furnace slag cement

Measure the temperature of the acid mixture exactly 20 min and 40 min after beginning introduction of anhydrous cement into the calorimeter, and take the measured temperatures as t_{u20} , t_{u40} , respectively.

Calculate the heat of solution of anhydrous cement, h_{u1} , from Equation (6).

Obtain heat of solution to one decimal place, and after ensuring that the difference between two measurements is within the specified tolerance, round the mean value of the two measurements to an integer and use it to calculate heat of hydration.

Tolerance checking of the difference between the two measurements shall be performed after rounding each measurement result to an integer.

The tolerance shall be 5 J/g.

$$h_{u1} = \frac{[(t_{u20} - t_{u0}) - (t_{u40} - t_{u20})] \cdot c_1}{W_{u1}} - 0.8(t_u - t_{u20}) \dots\dots\dots (6)$$

where, h_{u1} : heat of solution of anhydrous cement (J/g)
 t_u : laboratory temperature during introduction of anhydrous cement into the calorimeter (°C)
 t_{u0} : temperature of acid mixture immediately before introduction of anhydrous cement into the calorimeter (°C)
 t_{u20} : temperature of acid mixture 20 min after beginning introduction of anhydrous cement into the calorimeter (°C)
 t_{u40} : temperature of acid mixture 40 min after beginning introduction of anhydrous cement into the calorimeter (°C)
 c_1 : thermal capacity of calorimeter (J/K)
 W_{u1} : mass of anhydrous cement converted to the mass in the state after ignited at $950 \text{ °C} \pm 25 \text{ °C}$ for 90 min (g)