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Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 9 DETERMINATION OF HEAT OF HYDRATION

(First Revision)

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Indian Standard

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0. FOREWORD

0.1 This Indian Standard (Part 9) (First Revision) was adopted by the Bureau of Indian Standards on 22 April 1988, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate the physical properties of different types of hydraulic cements. The procedure for conducting chemical tests of hydraulic cement is covered in IS : 4032-1985*.

0.3 Originally all the tests to evaluate the physical properties of hydraulic cements were covered in one standard; but for facilitating the use of this standard and future revisions, it has been decided to print the different tests as different parts of the standard and accordingly, this revised standard has been brought out in thirteen parts. This will also facilitate updating of individual tests. Further, since publication of the original standard in 1968 a number of standards covering the requirement, of different equipment used for testing of cement, a brief description of which was also covered in the standard, had been published. In this revision, therefore, reference is given to different instrument specifications deleting the description of the instruments, as it has been recognized that reproducible and repeatable tests results can be obtained only with standard testing equipment capable of giving desired level of accuracy. This part (Part 9) covers determination of heat of hydration of cement.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Method of chemical analysis of hydraulic cement (*first revision*).

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part 9) covers the procedure for determining the heat of hydration of cement as expressed in kilojoules per kilogram.

2. SAMPLING AND SELECTION OF TEST SPECIMENS

2.1 The samples of the cement shall be taken in accordance with the requirements of IS : 3535-1986* and the relevant standard specification for the type of cement being tested. The representative sample of the cement selected as above shall be thoroughly mixed before testing.

*Methods of sampling hydraulic cements (*first revision*).

3. TEMPERATURE

3.1 The temperature of moulding room, dry materials, appliances and water shall be maintained at 27± 2°C.

4. APPARATUS

4.1 Calorimeter — Calorimeter conforming to IS : 11262-1985*.

4.2 Mortar and Pestle — Approximately 200 mm in diameter mortar and pestle for grinding partially hydrated samples.

4.3 Glass/Plastic Vials — Glass/plastic vials having the dimension approximately 80 × 20 mm with tight fitting stoppers or caps.

*Specification for calorimeter for determination of heat of hydration of hydraulic cement.

4.4 Stop Watch or Timer — The timer shall have a positive starting and stopping mechanism and shall be capable of being read to the nearest 0·5 s or less. The timer shall be accurate to 0·5 s or less for time interval up to 60 s and to 1 percent or less for time intervals of 60 to 300 s.

4.5 Sieve — 150 μm and 850 μm IS sieve conforming to IS : 460 (Part 1) - 1985*.

4.6 Muffle Furnace — Muffle furnace capable of maintaining a temperature of 900 to 950°C.

4.7 Analytical Balance — Analytical balance capable of reproducing results within 0·000 2 g with an accuracy of ± 0·000 2 g.

NOTE — Self-indicating balance with equivalent accuracy may also be used.

4.8 Standard Weights

4.9 Weighing Bottles

4.10 Camel Hair Brush

5. MATERIAL

5.1 Nitric Acid — of 2·00 ± 0·05 N strength, made in bulk from analytical reagent quality materials. Whenever a new batch is prepared, the heat capacity of the calorimeter shall be redetermined.

5.2 Hydrofluoric Acid — 40 percent (*w*/*w*), analytical reagent quality.

5.3 Zinc Oxide — Analytical reagent quality.

5.4 Wax — paraffin wax.

5.5 Distilled Water — conforming to IS : 1070-1977†.

6. PROCEDURE

6.1 Determination of the Heat Capacity

6.1.1 Inspect the wax lining for faults. Measure into the calorimeter 9·6 ± 0·1 ml of hydrofluoric acid and 388·0 ± 0·1 ml of 2·0 N nitric acid at a temperature of 27 ± 2°C. For convenience in measuring the nitric acid, a special standard flask of 388 ml capacity calibrated at 27°C shall be constructed. For measuring the hydrofluoric acid, a small measuring cylinder shall be made up by sealing a 15-cm length of 1-cm diameter 'polythene' resin tube to a flat plate of the same material with a small gas jet.

6.1.2 Take a quantity of zinc oxide sufficient for about six determinations. Ignite it for one hour at 900 to 950°C, cool in a desiccator containing anhydrous calcium chloride and grind it to pass a 150 micron IS Sieve. For each determination, about 7·0 g of this ignited oxide shall again be heated to 900 to 950°C for 5 min and then cooled for not less than 2½ h and not more than 5 h in the desiccator containing anhydrous calcium chloride before weighing accurately.

6.1.3 Assemble the calorimeter and run the stirrer for at least 5 min to allow the temperature to become uniform. Take temperature reading correct to 0·001°C every minute for 5 min to determine the initial heating or cooling correction. Then introduce the zinc oxide from the funnel steadily over a period of 1 to 2 min. The funnel shall then be brushed clean with camel-hair brush. Take temperature readings at one minute intervals until the solution is complete, as indicated by a steady rate of heating or cooling of the calorimeter. The solution period shall not exceed 20 min. Continue the readings for a further period of 5 min, to determine the final heating or cooling correction.

6.1.4 Plot initial and final heating or cooling rates against the corresponding calorimeter temperature, namely the Beckmann readings at the beginning of the solution period and at the end, respectively. Join the two points by a straight line (*see* Fig. 1 and example in **7.1**). From this graph, the corrections are read off for each temperature reading during the solution period. These corrections shall be summed and the total added or subtracted as appropriate to the observed temperature-rise.

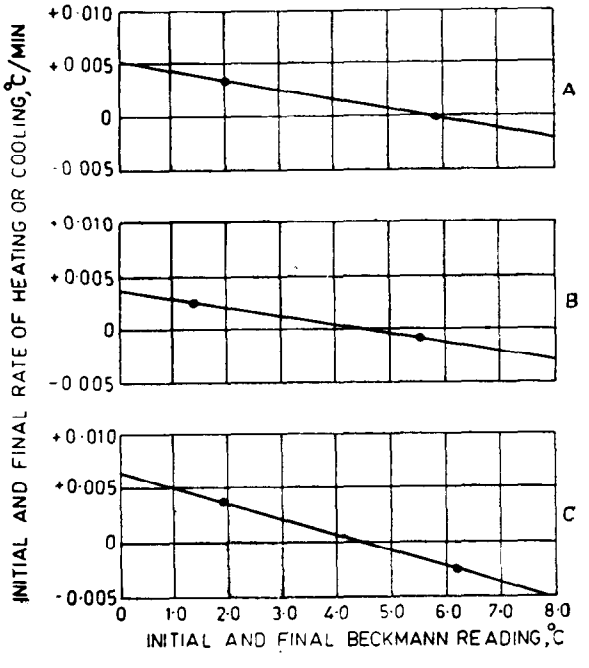

FIG. 1 HEATING OR COOLING CORRECTIONS

*Specification for test sieves : Part 1 Wire cloth test sieves (*third revision*).

†Specification for water for general laboratory use (*second revision*).

6.1.5 Calculate the heat capacity as follows:

Heat capacity

$$(J/°C) = \frac{\text{Mass of ZnO (g)}}{\text{Corrected temperature-rise}} \times [1\,072 + 0{\cdot}4\,(30 - \phi) + 0{\cdot}5\,(\phi_0 - \phi)]$$

where

1 072 = heat of solution of zinc oxide at 30°C,

0·4 = negative temperature coefficient of the heat of solution,

ϕ = final temperature of the calorimeter and contents in °C,

0·5 = specific heat of zinc oxide, and

ϕ_0 = room temperature in °C.

This expression simplifies to:

Heat capacity

$$= \frac{\text{Mass of ZnO } (1\,084 - 0{\cdot}9\,\phi + 0{\cdot}5\phi_0)}{\text{Corrected temperature-rise}}$$

6.2 Preparation of Cement Sample — Mix by hand for 4 min, 60 g of cement and 24 ml of distilled water which shall be between 15 and 25°C. Fill with this mixture 3 glass/plastic vials, cork and then seal with wax. Store the specimen vials with the mixture in a vertical position at 27 ±2°C.

6.3 Determination of the Heat of Solution

6.3.1 For determination of the heat of solution of unhydrated cement, weigh a sample of about 3·0 g. At the same time, weigh out another quantity approximately 7·0 g for the loss on ignition. Both the weighings shall be correct to the nearest 0·001 g. Carry out the determination of temperature-rise exactly as described for zinc oxide.

Calculate the heat of solution from the following formula:

Heat of solution (kJ/kg) of unhydrated cement

$$= \frac{\text{Heat capacity} \times \text{corrected temperature-rise}}{\text{Mass of sample corrected for ignition loss}} - 0{\cdot}8\,(\phi_0 - \phi)$$

where 0·8 is the specific heat of unhydrated cement.

6.3.2 The mean of three determinations which shall be carried out within 7 days of the mixing of the hydrated samples shall be taken.

6.3.3 For the determination of heat of solution of hydrated cement, break open one of the glass vials (*see* **6.2**). Remove the adherent wax and glass from the cement, then grind the cement (as rapidly as possible to avoid carbonation) to pass an 850-micron IS Sieve. Keep the ground sample in a stoppered weighing bottle from which weigh out samples of 4·2 and 7·0 g for heat of solution and loss on ignition, respectively. The loss on ignition shall be determined on each sample used for heat of solution. Carry out the determination of temperature-rise as before and calculate the heat of solution from the following formula:

Heat of solution of hydrated cement (kJ/kg ignited mass)

$$= \frac{\text{Heat capacity} \times \text{corrected temperature-rise}}{\text{Mass of sample corrected for ignition loss}} - 1{\cdot}7\,(\phi_0 - \phi)$$

where 1·7 is the specific heat of hydrated cement.

The mean of three determinations on separate vials shall be taken.

6.4 Ignition Loss — Place the sample in a cool furnace and raise the temperature of the furnace to 900°C over a period of one hour. Keep the sample at 900 ± 50°C for 3 to 4 h and then cool it in a desiccator containing anhydrous calcium chloride. Weigh after half an hour. All weighings shall be correct to the nearest milligram.

7. CALCULATION

7.1 Calculate the heat of hydration by subtracting the respective heats of solution of hydrated cement from the heat of solution of the unhydrated cement. The heats of hydration shall be determined at 7 and 28 days. Heats of solution shall be calculated to the nearest 0·5 kJ/kg and heats of hydration to the nearest 5 kJ/kg as given in the following example :

Example:

a) Determination of heat capacity

Time (min)	*°C Beckmann Calorimeter Temperature*	*Heating or Cooling Correction* (*see Graph C, Fig. 1*)
0	1·891	
1	1·894	
2	1·898	Initial correction
3	1·902	+0·003 4
4	1·905	
5	1·908	
6	2·550	−0·002 4
7	5·880	+0·002 2
8	6·175	+0·002 5
9	6·225	+0·002 5
10	6·241	+0·002 6
11	6·245	0·002 6
		= +0·010 0
12	6·243	
13	6·240	
14	6·237	Final correction
15	6·234	−0·002 6
16	6·232	
17	6·230	

Temperature-rise = 6·245 − 1·908 = 4·337

Correction = + 0·010

Corrected temperature-rise = 4·347

Mass of zinc sample = 7·00 g

Room temperature = 27·00°C

Final temperature of calorimeter and contents* = 27·75°C

$$\text{Heat capacity} = \frac{7{\cdot}00}{4{\cdot}347} \times (1\,084 - 0{\cdot}9 \times 27{\cdot}75 + 0{\cdot}5 \times 27)$$

$$= \frac{7{\cdot}00}{4{\cdot}347} \times (1\,084 - 24{\cdot}975 + 13{\cdot}5)$$

$$= \frac{7{\cdot}00}{4{\cdot}347} \times 1\,072{\cdot}525$$

$$= 1\,727 \text{ J/°C}$$

b) Determination of heat of solution on anhydrous cement sample

Time (min)	°C *Beckmann Calorimeter Temperature*	*Heating or Cooling Correction* (*see Graph B, Fig. 1*)
0	1·225	
1	1·228	
2	1·230	Initial correction
3	1·232	+0·002 2
4	1·234	
5	1·236	
6	3·350	−0 000 6
7	4·460	+0·000 2
8	4·850	+0·000 5
9	5·090	+0·000 6
10	5·230	+0·000 7
11	5·330	+0·000 7
12	5·392	+0·000 7
13	5·432	+0·000 7
14	5·460	+0·000 7
15	5·475	+0·000 8
16	5·483	+0·000 8
17	5·489	+0·000 8
18	5·491	+0·000 8
19	5·492	+0·000 8
		+0·008
20	5·492	
21	5·491	
22	5·490	Final correction
23	5·490	−0·000 8
24	5·490	
25	5·488	
26	5·487	

Temperature-rise = 5·492 − 1·236 = 4·256

Correction = + 0·008

Corrected temperature-rise = 4·264

*Determined separately by a mercury-in-glass thermometer.

Mass of cement sample = 3·000 g

Ignition loss = 1·91 percent

Room temperature* = 27·00°C

Final temperature of calorimeter and contents* = 27·25°C

Heat capacity of calorimeter = 1·727 J/°C

$$\text{Heat of solution of anhydrous cement} = \frac{1\,727 \times 4{\cdot}264 \times 100}{3{\cdot}000 \times 98{\cdot}09} - 0{\cdot}8\,(27{\cdot}00 - 27{\cdot}25)$$

$$= \frac{1\,727 \times 4{\cdot}264 \times 100}{3{\cdot}000 \times 98{\cdot}09} + 0{\cdot}2$$

$$= 2\,502{\cdot}4 + 0{\cdot}2$$

$$= 2\,502{\cdot}6 \text{ kJ/kg}$$

$$= 2\,502{\cdot}5 \text{ kJ/kg (say)}$$

c) Determination of heat of solution on hydrated cement sample after 28 days' storage at 27°C

Time (min)	°C *Beckmann Calorimeter Temperature*	*Heating or Cooling Correction* (*see Graph A, Fig. 1*)
0	2·019	
1	2·022	
2	2·026	Initial correction
3	2·030	+0·003 2
4	2·032	
5	2·035	
6	5·000	−0·000 5
7	5·700	+0·000 2
8	5·815	+0·000 2
9	5·845	+0·000 2
10	5·858	+0·000 2
11	5·867	+0·000 5
12	5·872	+0·000 3
13	5·877	+0·000 3
14	5·880	+0·000 3
15	5·881	+0·000 3
16	5·882	+0·000 3
		+0·002
17	5·882	
18	5·882	
19	5·882	Final correction
20	5·882	−0·000 3
21	5·882	
22	5·881	
23	5·880	

*Determined separately by a mercury-in-glass thermometer.

Temperature-rise	= 5·882 − 2 035 = 3·847
Correction	= 0·002
Corrected temperature-rise	= 3·849
Mass of cement sample	= 4·200 0 g
Ignition loss	= 27·96 percent
Room temperature*	= 27·00°C
Final temperature of calorimeter and contents*	= 24·5°C
Heat capacity of calorimeter	= 1 727 J/°C

*Determined separately by a mercury-in-glass thermometer.

Heat of solution of hydrated cement $= \frac{1\,727 \times 3{\cdot}849 \times 100}{4{\cdot}200\,0 \times 72{\cdot}04} - 1{\cdot}7\,(27{\cdot}00 - 24{\cdot}5)$

$= \frac{1\,727 \times 3{\cdot}849 \times 100}{4{\cdot}200\,0 \times 72{\cdot}04} - 4\,25$

$= 2\,196{\cdot}93 - 4{\cdot}25$

$= 2\,192{\cdot}68$ kJ/kg

$= 2\,192\,5$ kJ/kg (say)

d) Determination of heat of hydration

Heat of hydration at 28 days $= 2\,502{\cdot}5 - 2\,192{\cdot}5$

$= 310{\cdot}0$ kJ/kg

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed, if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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