Methods of testing portland and blended cements

Method 7: Determination of temperature rise during hydration of portland and blended cements

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PREFACE

This Standard was prepared by the Standards Australia Committee BD/10, Cement, to supersede AS 2350.7—1980, *Methods of testing portland and blended cements*— Method 7: *Heat of hydration of portland cement*, which was only applicable to portland cement. The new method is applicable to both portland and blended cements.

The term 'normative' has been used in this Standard to define the application of the appendix to which it applies. A 'normative' appendix forms an integral part of a Standard.

METHOD

1 SCOPE This Standard sets out the method for the determination of temperature rise during the hydration of portland and blended cements, under precisely defined conditions.

NOTE: A procedure for calculating the heat of hydration of portland and blended cements is presented in AFNOR NF 15-436.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS	
2349	Method of sampling portland and blended cements
2350	Methods of testing portland and blended cements
2350.12	Method 12: Preparation of a standard mortar and moulding of specimens
IEC 751	Industrial platinum resistance thermometer sensors
AFNOR	
NF 15-436	Measuring the hydration heat of cements by means of semi-adiabatic calorimetry (Langavant method)

3 PRINCIPLE A fixed quantity of plastic cement mortar prepared in accordance with AS 2350.12 is introduced into a calorimeter, and the subsequent rise in temperature over a period of time is recorded.

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4 LABORATORY AND EQUIPMENT

4.1 Laboratory The temperature of air within the laboratory where the apparatus is located shall be maintained at a temperature of $23 \pm 2^{\circ}$ C. The apparatus shall be located away from sources of heat and ventilation.

4.2 Enclosure The velocity of ventilation air around the calorimeters shall be minimized by the construction of an enclosure around the calorimeter. The enclosure shall have no lid and be large enough to accommodate all calorimeters (if more than one calorimeter is used) allowing at least 120 mm clearance between the calorimeters and the inside walls of the enclosure. The walls of the enclosure shall be at least 25 mm higher than the calorimeter. The base of the enclosure shall be insulated and sealed, the airflow within the enclosure shall be less than 0.5 m per second and the enclosure shall be located well away from sources of heat and ventilation.

The temperature within the enclosure for the duration of the test shall be maintained at $\pm 0.5^{\circ}$ C of the temperature at the commencement of testing.

The temperature of the materials and equipment in Clauses 4.3 and 4.4 shall be brought to within $\pm 1.0^{\circ}$ C of the temperature of the enclosure and shall be 23 $\pm 2^{\circ}$ C.

4.3 Calorimetric apparatus The calorimeter (see Figure 1) shall be a cylindrical vacuum flask with an insulated stopper and placed in a rigid casing which acts as support for the flask. The calorimeter shall be calibrated in accordance with Appendix A. The apparatus shall be as follows:

NOTE: Calorimeters supplied by Laboratoire National d'Essais and Maurice Perrier & C^{ie} comply with the requirements of this Standard.

- (a) Vacuum flask The vacuum flask is made of silvered borosilicate glass with an internal diameter of 95 mm, an internal depth of 280 mm and an external diameter of 120 mm. At the bottom of the vacuum flask is a rubber disc approximately 85 mm diameter and 20 mm thick. This disc acts as a support for the mortar container and distributes the load evenly on the glass wall.
- (b) *Casing* The casing of the vacuum flask shall have a sufficiently wide base to ensure stability of the whole unit and shall be of rigid construction (see Figure 1). The casing shall provide approximately 5 mm air space from the internal wall of the flask and shall allow the flask to rest on a support 4 to 5 mm thick, made of material of low thermal conductivity, e.g. expanded polystyrene. There shall be an abutment in the form of a 5 to 10 mm thick rim fixed to the casing where the upper edge of the flask shall be in contact. This abutment is to hold the flask in position and to provide a bearing surface for the stopper so as to ensure the tightness of the flask stopper unit. The abutment shall be made of material of low thermal conductivity. A rubber gasket shall be placed between the edge of the flask and its rim.

NOTE: Material such as an aluminium alloy 3 mm thick may be used for constructing the casing.

- (c) Stopper unit The stopper unit shall be made of the following parts (see Figure 1):
 - (i) *The lower part*—part of the stopper unit which is inserted into the flask to provide maximum prevention of heat loss into the external environment. This part of the stopper shall be cylindrical in shape with a diameter equal to the internal diameter of the flask and shall have a thickness of approximately 55 mm. It shall be made of material of low thermal conductivity, such as expanded polystyrene, with its base protected by a 2 mm thick plate of rigid plastic.

- (ii) *The central part*—part of the stopper unit which provides rigidity for the calorimeter whilst contributing to the reduction of heat loss. This part of the stopper unit shall be made of a foam rubber disc of approximately 120 mm in diameter.
- (iii) *The upper part*—part of the stopper unit which allows the stopper to be correctly positioned on the top of the flask. It shall be a 8 mm thick disc made from a rigid material. This upper part of the stopper shall fit tightly against the casing by means of a snap locking device which compresses the rubber central part.

NOTE: The 8 mm thick disc may be made from material such as aluminium alloy.

4.4 Mortar container The mortar container shall be a watertight cylindrical container with a lid, fabricated from tin plate (see Figure 1). The container shall have a maximum diameter of 82 mm and a maximum depth of 170 mm. The lid of the container shall be provided at its centre with a brass tube designed to receive the temperature-measuring device. The internal diameter of the brass tube shall be slightly larger than the sleeve of the temperature-measuring device wires. The internal length of the brass tube shall be $110 \pm 10 \text{ mm}$.

4.5 Temperature-measuring device The temperature-measuring device shall be a 4-wire resistance temperature device (RTD) conforming to IEC 751, Class A.

The length of the measuring element of the RTD shall be 120 mm ± 10 mm.

The RTD shall have an integral connected extension lead which shall have an overall maximum outer sheath diameter of 3 mm and shall be made of a non-metallic low thermal conductivity material.

4.6 Temperature recorder A data logger with a precision of $\pm 0.01^{\circ}$ C shall be used.

5 MATERIALS

- 5.1 General All materials specified in AS 2350.12 are required.
- 5.2 Oil A light oil and appropriate dispenser shall be used.
- 5.3 Sealer A pliable sealer shall be used.

NOTE: Plasticine is a suitable sealer.

6 PROCEDURE

6.1 Preparation of test mortar The procedure for preparing the test mortar shall be as follows:

- (a) Weigh an empty mortar container.
- (b) Prepare the mortar using the composition, weight of ingredients and mixing procedure as specified in AS 2350.12.
- (c) Immediately after completion of the mixing sequence, weigh 1575 ± 1 g of the mortar into the mortar container, using a suitable scoop. Put the lid on and ensure that it is tight.
- (d) Place 2 to 3 mL of oil in the centre tube of the lid.

NOTE: The oil improves the thermal contact between the tube and the RTD. The volume of oil added must be small enough to ensure that the tube does not overflow when the RTD is inserted.

(e) Reweigh the container, and record the weight.

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