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Calibration of a QAB calorimeter

TEST PRINCIPLE

Executing the quasi-adiabatic test on concrete (called the QAB test) requires accurate knowledge of both the total heat loss coefficient α and heat capacity μ of the calorimeters introduced, with these two parameters capable of being determined by calibration.

This method, adapted from Standard NF EN 196-9 (entitled "Cement test methods – Part 9: Hydration heat – The semi-adiabatic method"), consists of replacing the test sample inside the calorimeter to be calibrated with a same-sized calibration cylinder equipped with an electrical resistance. By applying a known potential difference to this resistance so as to trigger a temperature rise, the amount of applied electrical energy corresponds to both the temperature rise taking place in the calorimeter and the heat dissipated towards the atmosphere. Heat loss can then be calculated based on the electrical energy required to maintain constant temperature.

As a next step, the heat capacity is determined by measuring the rate at which temperature is decreasing inside the calorimeter once the electrical source has been disconnected.

INSTRUMENTATION

In order to apply this method, the following instruments are required:

- a digital multimeter, used to measure both the calibration cylinder resistance and test voltages;
- a temperature measurement chain composed of Pt100 4-wire probes;
- a stabilized power source;
- a calibration cylinder (see [Fig. 2b](#)), containing an aluminum specimen with an identical shape to the test specimen (i.e. $\varnothing 16$, $H = 32$ cm) and a heating resistance of approx. 2,500 ohms with a very low coefficient of electrical resistivity (a constantan or manganin wire). The resistance connection wires are designed with a small cross-section (max. 0.05 mm^2) to avoid heat leaks;
- a calorimeter used as a control device, equipped with an inert cylinder of an identical design to the calibration cylinder, yet without a heating resistance;
- a climate-controlled room set at a temperature of between 19 and 21°C and programmed to remain stable to within $\pm 0.5^\circ\text{C}$.

OPERATING PROCEDURE

■ Preliminary conditions

The calorimeter to be calibrated is placed immediately adjacent to the control calorimeter in the climate-controlled room (Fig. 1). Both devices are positioned on a table to withstand the influence of heat exchange conditions at floor level, which is where conditions can differ considerably from one testing room to another.

Figure 1
View of the two calorimeters

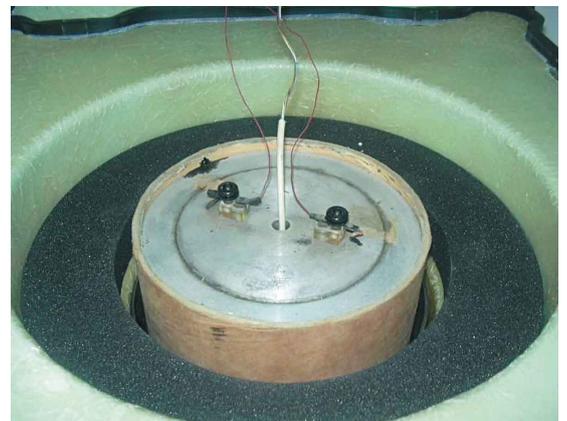


A calibration cylinder, connected to the power supply, is placed inside the calorimeter to be verified, while an inert cylinder is placed in the control calorimeter. A platinum resistance probe is also positioned at the center of each cylinder in the test set-up.

Figure 2
a) View of the calorimeter to be calibrated with its heating resistance;
b) Close-up view



a | b



The opening on the calorimeter cap (compression gland) (see Figs. 1 and 2a), which serves to route the connection wires of both the calibration cylinder and the resistance probe, is hermetically sealed so as to prevent convection currents between the calorimeter interior and the ambient environment.

■ Determination of the total heat loss coefficient α

The application of a voltage at the calibration cylinder resistance terminals causes this cylinder to warm by means of the Joule effect.

The total heat loss coefficient α of the calorimeter is obtained by measurement, during an established steady-state mode, using:

- the amount of heat released by the Joule effect inside the calibration cylinder
- warming of this cylinder in comparison with the temperature of the inert cylinder placed in the control calorimeter.

Once the steady state has been reached (the time required to achieve thermal equilibrium is on the order of 14 days), the heat generated is completely dissipated towards the external environment.

The loss equation can therefore be written as follows:

$$P = V^2 \cdot R^{-1} = \alpha \cdot \theta$$

yielding: $\alpha = V^2 \cdot R^{-1} \cdot \theta^{-1}$ (in $\text{W} \cdot ^\circ\text{C}^{-1}$)

or alternatively: $= 3600 V^2 R^{-1} \theta^{-1}$ (in $\text{J} \cdot \text{h}^{-1} \cdot ^\circ\text{C}^{-1}$)

with: V (V) = supply line voltage at the resistance terminals

R (Ω) = resistance of the heating winding

P (W) = dissipated power

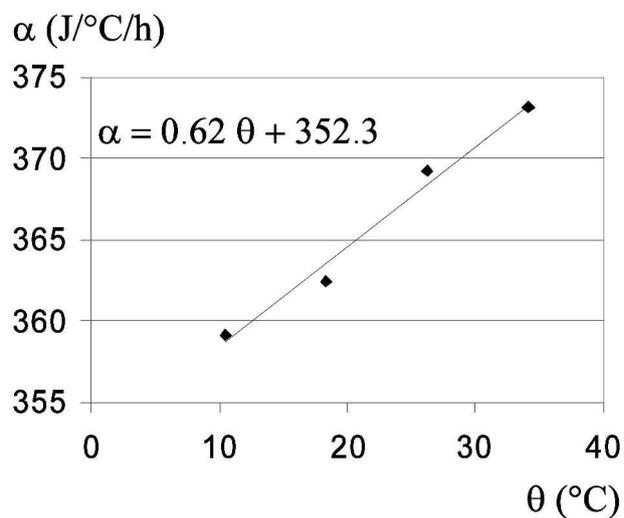
θ ($^\circ\text{C}$) = temperature difference between calibration cylinder and inert cylinder

α ($\text{J h}^{-1} \cdot ^\circ\text{C}^{-1}$) = total heat loss coefficient of the calorimeter at temperature θ .

The coefficient α is determined for four values of temperature increases θ covering the temperature range observed during the test. A linear variation of α with respect to θ (i.e. a straight calibration line) is thus obtained: $\alpha = a + b \cdot \theta$.

The constants a and b are calculated using the least squares method, with the linear correlation coefficient needing to exceed 0.97. **Figure 3** shows an example of this result.

Figure 3
Sample calibration line



■ Determination of heat capacity μ

The calorimeter heat capacity μ is measured by means of the spontaneous cooling method when the expression of total heat loss coefficient α is known.

Once the heat capacity C_T of the calorimeter containing a cylinder with known heat capacity C_E (i.e. a calibration cylinder) has been determined, it then becomes possible to deduce the heat capacity of the empty calorimeter μ .

During cooling (after shutting off the power supply), the heat exchange equation can be written as follows:

$$-C_T d\theta = \alpha \theta \cdot dt = (a + b\theta) \theta \cdot dt$$

$$\text{which leads to: } C_T = \frac{a \cdot t}{\log_e \frac{\theta_0 \alpha_t}{\theta_t \alpha_0}}$$

with: t (in hours) = cooling time from the initial instant (time 0)

θ_0, α_0 = temperature rise and total heat loss coefficient at time 0

θ_t, α_t = temperature rise and total heat loss coefficient at time t

Four measurements of θ_t are recorded for cooling periods of 24, 26, 28 and 30 hours.

After determining the corresponding C_T values, the heat capacity of the empty calorimeter displays the following value:

$$\mu = 1/4 \sum_{i=1}^4 \mu_i$$

with: $\mu_i = C_{Ti} - C_E$

where μ is expressed in Joules per degree Celsius.

PERIODICITY

In order to account for the deterioration in calorimeter insulation characteristics, device inspection must be repeated every 4 years and moreover becomes mandatory any time the calorimeter has been modified or refurbished.

EXAMPLE

Figure 4 presents a sample calibration report of a QAB calorimeter.

CONCRETE CALORIMETERS

Measurement sheet

E.C. No. 2009-256

Instrument identification:

QAB calorimeter No. 100116

Calibration methods and conditions:

- References tied to national benchmarks (designation and two-standard deviation uncertainty)

Digital multimeter FLUKE 8840A No. 5765018	Direct voltage	$u = \pm 10 \text{ mV}$
	Resistances	$u = \pm 2 \Omega$
Pt100 - 4-wire probes	Specimen temperature	$u = \pm 0.15^\circ\text{C}$
	Reference temperature	$u = \pm 0.10^\circ\text{C}$
- Conditions (values and tolerances of the selected influence factors):
 - Temperature: $20^\circ\text{C} \pm 2^\circ\text{C}$

Determination of the total heat loss coefficient α

Specimen temperature	(°C)	29.99	39.58	58.43	77.02
Reference temperature	(°C)	19.34	19.81	19.17	19.00
Temperature rise θ	(°C)	10.65	19.77	39.25	58.02
Voltage	(V)	51.007	70.051	99.982	123.927
Resistance	(Ω)	2500.3	2500.3	2500.3	2500.3
Coefficient α	(J/h/°C)	351.70	357.45	366.67	381.10

Straight line equation: $\alpha \text{ (J/h/}^\circ\text{C)} = (344.9 + 0.604 \theta) \pm 6.3$ Linear correlation coefficient: 0.99

Heat capacity measurement of the empty calorimeter μ

Time	(h)	0	24	26	28	30
Specimen temperature	(°C)	77.02	53.48	52.04	50.66	49.35
Reference temperature	(°C)	19.00	19.18	19.19	19.19	19.20
Temperature rise θ	(°C)	58.02	34.30	32.85	31.47	30.15
Total capacity	(J/°C)		16988	16984	16981	16982
Cylinder capacity	(J/°C)		13544	13544	13544	13544
μ	(J/°C)		3444	3440	3437	3438

Average thermal capacity: $\mu \text{ (in J/}^\circ\text{C)} = 3,440$

Inspector: DURAND F.	Calibrated on: October 22, 2009 Next calibration: October 2013	Page 3 / 3
Inspection is valid for 4 years, provided no calorimeter modification and normal operating conditions.		

Figure 4:
Sample calibration report