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Prevention of Thermal Cracking in Concrete at Early Ages

Edited by
R. Springenschmid

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RILEM REPORT 15

Prevention of Thermal Cracking in Concrete at Early Ages

State-of-the-Art Report prepared by
RILEM Technical Committee 119
Avoidance of Thermal Cracking in Concrete at Early Ages

Edited by

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Preface

This book describes the work of RILEM Technical Committee -119 TCE on the "Avoidance of Thermal Cracking in Concrete at Early Ages" which was performed between 1989 and 1997. It also includes important results presented at the International Symposium on Thermal Cracking in Concrete at Early Ages between October 10th and 12th, 1994 in Munich.

The avoidance of cracking of young concrete is one of the main problems of concrete technology. The aim of the work of the members of the RILEM TC 119 and many civil engineers and scientists all over the world was to replace earlier methods, based purely on field experience, by modern concepts for the prediction of stresses in concrete at early ages and the factors influencing them.

The book should be a basis for further research. Furthermore – and this is even more important – it should serve as a basis for the avoidance of thermal cracking by the engineers who have to design and construct mass or medium sized concrete structures and by those who have to select the concrete materials and design the concrete mix.

As Chairman of RILEM TC 119, I would like to thank the authors of the sections of this book and all others who have contributed to this work. They passed on their experience generously. Mr. J.-L. Bostvironnois assisted me in most of this editorial work. I would like to take this opportunity to thank him.

Rupert Springenschmid
Munich, March 1998

Introduction

Cracks in mass concrete structures caused by the hydration heat of the cement are a well-known phenomenon since the beginning of this century. Methods of avoiding such cracks have been developed mainly for large concrete dams and other massive hydraulic engineering structures. In order to reduce the heat development, pozzolanas, and since 1932, low-heat cements have been used. Further progress aimed at reducing the high maximum temperature caused by the hydration heat has been made by the use of very low cement contents, coarse aggregates, cooling the concrete materials, limitation of lift-heights and by pipe cooling.

Although the processes of heat generation and dissipation were familiar to civil engineers, the specification of the maximum permissible temperature in the concrete bulk and the temperature difference between the base and the inside of the concrete block was based solely on experience. The decisive properties of a particular concrete such as its tensile strength or its coefficient of thermal expansion were not considered.

In the past decades, cracks in the structural concrete of foundations, bridges, tunnel linings and other medium-sized concrete elements have become an increasing problem. It has been found that drying shrinkage is often of minor importance. The heat of hydration as well as other temperature changes have been found to be the primary cause of restraint stresses and cracks in unreinforced as well as reinforced concrete.

In the late sixties, the first attempts were made to estimate the size of stresses due to restrained thermal deformations and compare them with the increasing tensile strength of the concrete at early ages. Two points turned out to be extremely problematic:

- The results of thermal stress calculations depend strongly on the evaluation of the stiffness of the concrete as it increases during its transformation from a semi-liquid to a solid state. However, the stiffness is difficult to measure and predict.
- Restraint stresses could not be determined with conventional methods and therefore no data were available to verify the results of stress calculation.

In 1969 the first laboratory equipment, the cracking frame, was developed in order to perform model tests. By measuring the stress response of concrete at early ages to changing temperature we gained a deeper understanding of the changes which occur when the expansion or contraction of a concrete element is prevented and stresses are produced. The Munich Temperature Stress Testing Machine (1984) and similar machines in several other research institutes now allow stress measurement for any degree of restraint.

In recent years much research work has been devoted to the calculation of the early age restraint stresses and to the determination of the risk of cracking. Computer programs have taken account of the materials properties, the hydration heat development, the increase of stiffness and the decrease of relaxation capacity, the increasing tensile strength, the coefficient of thermal expansion and the influence of chemical reactions on the deformation. All these factors depend largely on age, temperature, cement type, cement brand and concrete mix composition. Realistically speaking, it is only possible to assess roughly the effects of these factors.

Promising new methods have been developed in Japan, France and Germany to measure restraint stresses in situ. The comparison of test results both in the laboratory and in the field with the results of calculations is a source of further progress in this area.

In recent years high-strength concrete has proved to be an extremely sensitive material regarding cracking at an early age. This is not only a consequence of the hydration heat. Autogenous shrinkage due to self-desiccation and chemical reactions of the sulphate phase may also be important.

Unexpected cracking of structural or mass concrete cannot always be attributed to the inexperience of the practical engineers. The limited knowledge of many problems in this area has strongly encouraged research all over the world. In 1989 RILEM, the International Union of Testing and Research Laboratories for Materials and Structures, established a Technical Committee, TC 119, on „the Avoidance of Thermal Cracking in Concrete at Early Ages". The tasks of this committee are as well as the exchange of opinions and experience, the preparation of State of the Art Reports and to make RILEM Recommendations for the following test methods:

- Determination of the semi-adiabatic hydration heat
- Determination of the adiabatic hydration heat
- Restraint stress measurements in the laboratory with the cracking frame
- Restraint stress measurements in situ with the stress-meter.

In the meantime the recommendations have been published in *Materials and Structures/Matériaux et Constructions*, **30**, October 1997, pp 451-464, and are reprinted here in chapter 10.

The International Symposium in Munich between October 10th and 12th, 1994 thus took place in a dynamic period of development. A number of questions regarding the avoidance of early age cracking have already been solved and the results are ready for practical application. To answer other questions we have many suggestions based on test results or from theoretical considerations.

The avoidance of early age cracking is a task which requires theoretical knowledge, sound engineering judgement and extensive experience. Furthermore, dedicated engineers must ensure that all the necessary considerations are carried out in practice.

Rupert Springenschmid
Munich, March 1998

1

Methods to Determine the Heat of Hydration of Concrete

P. Morabito

1.1 INTRODUCTION

In order to minimise the risk of thermal cracking in concrete structures a knowledge of the expected temperature rise during the hydration of cement is desirable. The factors controlling the rate of temperature rise, the maximum temperature and the thus temperature distribution are complex and many and include:

- Rate of heat evolution
- Total amount of heat evolved
- Proportion and composition of cement
- Dimensions of pour
- Environmental conditions - temperature, wind, humidity
- Temperature of concrete at placing
- Mix proportions
- Aggregate type.

Numerous laboratory techniques have been developed to measure the temperature rise in concrete, ranging from sophisticated calorimeters used to monitor the temperature in very small samples of cement to temperature measurements at the centre of a large insulated block of concrete. The testing methods being used can be divided into the following categories: isothermal, adiabatic and semi-adiabatic. Isothermal methods are usually applied to pure cement pastes and make them possible to measure the heat generated by samples kept at constant temperature. The majority

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of standard tests [1-4] currently in use rely on this method. Adiabatic methods use calorimeters for measuring the temperature rise in a sample being hydrated with no heat exchanges with the external medium. Semi-adiabatic methods use calorimeters or equipment where the heat exchange with the external environment is limited.

The ideal testing method should be able to reproduce the temperature = f(time) curve of each type of concrete during concreting. Since the isothermal tests do not take account of the change in reactivity of the cement with changing temperature, prediction of temperature rise of concrete from these results can be very difficult; the isothermal conditions of the test do not reflect the conditions in the real structure where the temperature is continually changing.

Adiabatic and semi-adiabatic methods are to be preferred. True adiabatic conditions are impossible to achieve but they do at least more closely resemble conditions at the centre of a large pour than do the isothermal conditions. A more realistic test would perhaps be to use semi-adiabatic methods where some heat is lost from the calorimeter. It should be realised however that even these tests may not truly reflect the conditions in the structure where the rate of heat loss is continually changing and would be difficult if not impossible to accurately reproduce them in the laboratory.

Adiabatic and semi-adiabatic calorimeters have been developed to measure the temperature rise in cement pastes, cement mortars and concrete samples. Although those developed for use with pastes and mortars [5, 6] are often much smaller than those used for concretes, the results obtained do not give a direct measure of the temperature rise in the concrete. The results can only be used to estimate the behaviour of the concrete from a knowledge of the mix proportions and thermal characteristics of the aggregates. Calculations of this kind can be extremely difficult because the kinetics involved are significantly different due largely to the buffering effects of the aggregate [30].

This report is concerned with the characteristics of adiabatic and semi-adiabatic calorimeters designed to measure the temperature rise and/or heat evaluation in concrete samples.

1.2 DEFINITIONS

For the purpose of this report the following definitions shall apply.

Adiabatic calorimeter. - Being impossible to have materials with an infinite thermal resistance, heat losses are prevented by controlling the temperature of the environment surrounding the sample in order to be as close as possible to the temperature of the sample at any time of the hydration. In order to achieve this conditions some form of heat will need to be supplied to the environment in which the test specimen is kept. However no calorimeter can be claimed to be truly adiabatic, even the most sophisticated are prone to some heat loss.

Semi-adiabatic calorimeter. - In the majority of semi-adiabatic calorimeters no attempt is made to heat the environment in which the specimen is placed. Most calorimeters of this type rely only on some form of insulation to slow down the rate of heat loss from the sample.

1.3 REVIEW OF AVAILABLE METHODS

1.3.1 Adiabatic calorimeter

In this report adiabatic calorimeters will be broadly classified by the method used to control the temperature of the environment in which the specimen is kept. For this purpose researchers have adopted one of three basic approaches. In the majority of cases the sample is surrounded by a heated water jacket, in some the air surrounding the specimen is heated and in others the container in which the specimen is kept has been heated. In addition, in the majority of calorimeters some form of insulation is provided between the sample and the control environment.

(a) Adiabatic calorimeters using water as the insulating medium
Work carried out by the Sumitomo Cement Company of Japan [7-10] has led to the development and production of a commercially available adiabatic calorimeter. The original development work involved making comparisons between two different basic types of calorimeters, one using air and the other using water as the insulating medium. By making comparisons with temperature measurements taken at the centre of a large insulated block, they concluded that the temperature rise measured by the apparatus using water (Type A) agreed very well with that from the large block. The results from the apparatus using air (Type B) were lower than that of the core temperature of the block.

The Type A apparatus was also used to examine the effect of specimen size on adiabatic temperature rise. Two cylindrical specimens of different sizes were used, namely: 60 cm diameter x 60 cm and 30 cm diameter x 30 cm high; there was very little difference between the two.

Looking at the influence of insulating the specimen when using the Type B apparatus they concluded that the temperature rise in the insulated specimen was lower than that in the non-insulated. They attributed this to the fact that some of the heat was consumed to heat the insulating material itself. The calorimeter developed by the Sumitomo Cement Company as a result of this work is shown in Fig. 1.1. The cylindrical specimen container is made of steel, the internal dimensions of which are 600 mm diameter x 600 mm in height. The container is placed inside a thermal jacket through which water is circulated at high speed and this thermal jacket is then itself placed inside an insulated container. The specimen container is separated from the thermal jacket by a 5 mm air gap. The water is heated or cooled to keep the surface of the sample of the same temperature as the core; it is claimed that the jacket temperature can be kept to within ± 0.1°C of the core temperature. Temperature measurements are made with platinum resistance thermometers and the sensitivity of the electronic control is quoted at ± 0.01°C. The apparatus can also be employed to take a smaller specimen container 300 mm in height and diameter; in addition, replacing the water with silicon oil as the thermal medium enables temperatures in excess of 100°C to be measured.

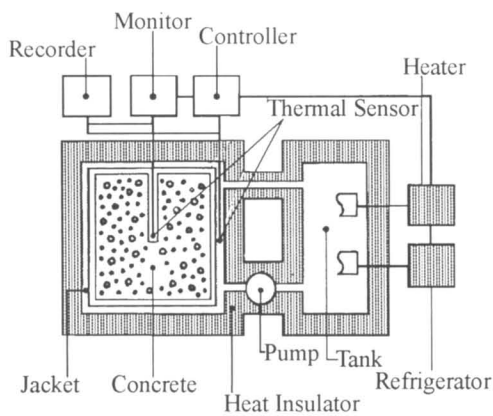

Fig. 1.1 Adiabatic calorimeter of the Sumitomo Cement Company.

A second commercially available calorimeter is marketed by ENEL spa [11] of Milan, Italy, and is similar in many respects to that of the Japanese described above. It uses a cylindrical specimen measuring 300 mm diameter x 360 mm high and the thermal jacket in this case is made of aluminium instead of steel. The only other significant differences are that in this apparatus the specimen container is separated from the thermal jacket by a layer of lightweight insulation, the jacket is made of aluminium and the temperature measurements are made with thermistors. The thermal jacket is heated by a mixing of water and glycol and the difference between the temperature of the concrete and the wall of the jacket is quoted at 0.1°C. The corresponding thermal loss is quoted at 0.002°C/h. To compensate for the high value of the apparent heat capacity of the calorimeter a thermal compensation technique has been applied during the hydration of the specimen; this consists of supplying to the sample by an electrical heater placed at the bottom of the sample container an external heat equal to that accumulated from the heat capacity of the calorimeter. From several tests performed on the same mix, the reproducibility in the measurement of the adiabatic temperature rise at seven days is quoted to be within 2.5%.

Similar but less sophisticated calorimeters have been developed elsewhere by a number of workers [5, 12-15] but none has been made available for production on a commercial basis.

A smaller but more versatile calorimeter has been developed in the UK by Coole [16] (see Fig. 1.2). A concrete sample weighing 1 kg is contained in a polypropylene cell within a larger insulated polypropylene container submerged in a well-stirred water bath.

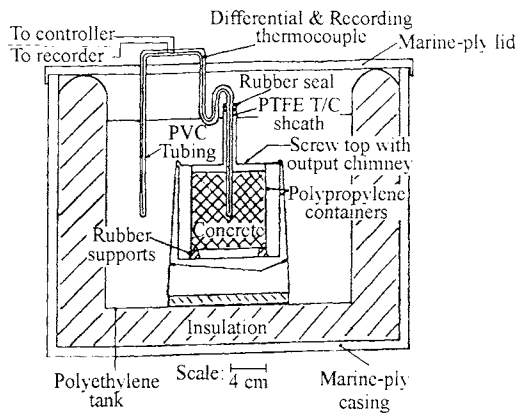

Fig. 1.2 Adiabatic calorimeter developed by Coole.

The significant difference between this and other calorimeters of a similar type is that there is no insulation between the specimen and the water bath. The temperature difference between the sample and the water is controlled by thermocouples connected to a thyristor control unit, this temperature difference can be varied to simulate heat losses from different size pours. When used as an adiabatic calorimeter the temperature difference is set at 0.01°C and the heat loss from the system has been measured at 0.01°C/h.

The calorimeters described previously have all been designed specifically to measure only the temperature rise in a concrete specimen. It is however often necessary to measure various properties of the concrete which has been subjected to such a temperature rise. The equipment developed by Wainwright [17] whose control system is shown in Fig. 1.3 was designed specifically to do this. As a calorimeter it is similar in many respects to those mentioned above with the 65 kg concrete specimen contained in an insulated mould which in turn is submerged in a temperature controlled water bath. The temperature of the water is controlled to follow that of the concrete but the volume of the bath is large enough to enable concrete specimens to be stored alongside the control cube.

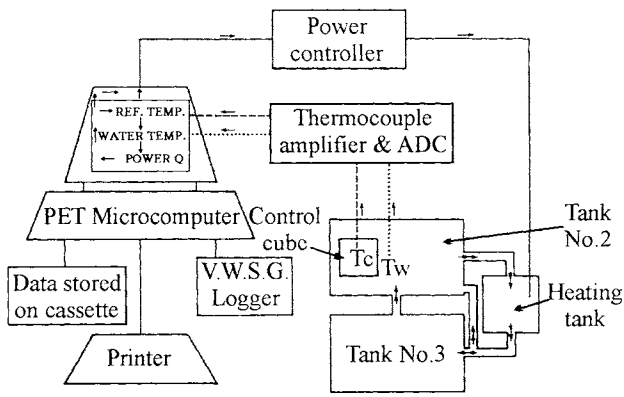

Fig. 1.3 Control and logging system of the adiabatic calorimeter developed by Wainwright.

A similar but somewhat smaller calorimeter has also been developed in Holland by Sarneel [18].

(b) Adiabatic calorimeters using air as the insulating medium

In terms of construction, calorimeters using air as the insulating medium tend to be somewhat simpler than those using water. In principle the

calorimeter consists of an insulated container in which the air temperature is controlled to follow that of the concrete specimen placed inside it. The existing calorimeters in this category differ only with respect to the type and quantity of insulation, the method of heating the air and the sophistication and sensitivity of the control system.

Bamforth [19] used a standard laboratory oven as the control medium in which he placed a 300 mm cube of concrete contained in a plywood mould lined with 25 mm of polystyrene. The temperature of the oven was controlled to within 0.2°C of the concrete temperature using a balancing unit linked to electrical resistance thermometers located in the concrete and the oven. The heat loss from the system measured on fully hydrated concrete specimens heated to 80°C was quoted at 0.08°C/h.

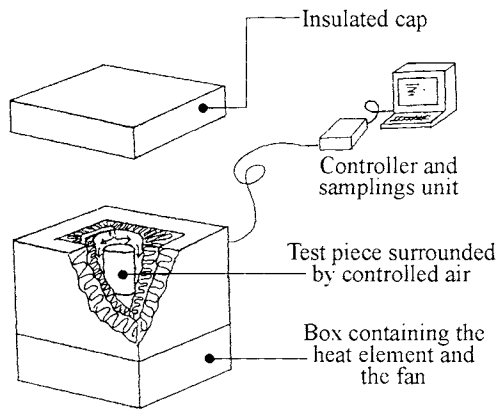

Fig. 1.4 Adiabatic calorimeter developed in Luleå University.

A similar but more sophisticated calorimeter has been developed in Sweden by Emborg [20] and is shown schematically in Fig. 1.4. The cylindrical test specimen weighing approximately 24 kg is placed inside a double insulated box with an air gap between the two. A box beneath the specimen contains the heating elements and circulating fan to control the temperature of the air surrounding the specimen. The temperature control for a known input of energy is calibrated using tests on aluminium specimens with known thermal properties. The reported maximum temperature loss at 70°C is 0.008°C/h.

According to Høyer [21] the Nordic draft test method for adiabatic calorimetry for the measurement of heat development in concrete proposes using air as the control medium. The draft gives no recommendations on specimen shape or size, design of calorimeter, control system,

sensitivity etc. It simply states that the apparatus shall consist of a box in which the specimen is surrounded by air the temperature of which must always be the same as the temperature of the concrete. The passive stability of the box measured using an inert specimen (e.g. water, mature concrete) heated to approximately 60°C shall not be greater than + 0.5°C/day (i.e. 0.02°C/h).

Jaegerman [22] has reported the existence of an adiabatic calorimeter supplied by Toni Technik of Berlin. The volume of the test specimen is 5 litres and the "control precision" is quoted as better than 0.02°C/h. It is not clear what is meant by the term control precision and no results from tests run with the apparatus are available.

Several other workers [23-26] have developed calorimeters for their own use which are similar in most respects to those described above.

(c) Adiabatic calorimeters using heated container as the insulating medium

A clear distinction between this type of calorimeter and the type described previously in b) is sometimes difficult to make, particularly where the specimen container is heated and this in turn heats any air gap that might exist between it and the specimen.

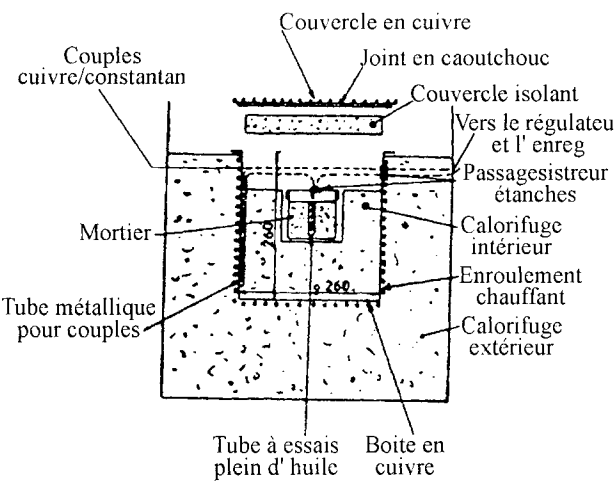

Fig. 1.5 Sketch of the CERILH calorimeter

The calorimeter used at the French Hydraulic Binders Research Institute (CERILH) and developed by Joisel [27] is shown in Fig. 1.5. The calorimeter consists of a cylindrical copper container which is enclosed in

an electric heating coil. The sample used is a 2 kg cylinder of mortar or concrete which sits inside the copper container surrounded by an insulating layer of expanded polystyrene placed inside a second outer container. The temperature of the copper container and the test sample are monitored by thermocouples. The calorimeter is also fitted with a refrigerator for testing concrete to be used for dams. The heat loss from the apparatus (measured using an inert sample heated to approximately 60°C) is quoted at between 0.2 to 0.3°C/day (i.e. 0.008 to 0.013°C/h). It is also suggested that if the weight loss due to evaporation of mixing water during the test exceeds 1 g (i.e. 0.05 % by weight of the original sample) then the test should be considered unreliable.

A similar calorimeter has been developed in Italy by Costa [28] for Italcementi. The apparatus (Fig. 1.6) uses a cylindrical specimen 0.005m³ in volume cast inside a mould made from tin plate and fitted with a water tight lid.

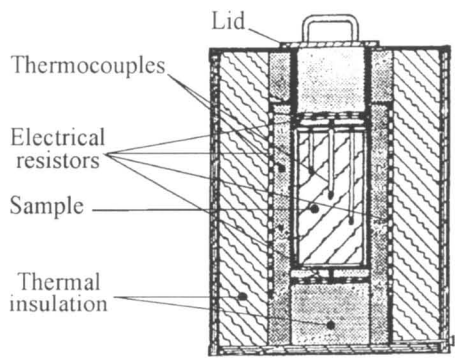

Fig. 1.6 Adiabatic calorimeter developed by Costa.

Five copper tubes are soldered to this lid and accommodate ten thermocouples measuring the temperature of the concrete at various locations. The sample container sits inside a second cylindrical container and is separated from it on all sides by a layer of insulation. This second cylinder together with two brass plates (situated at the top and bottom of the sample container) act as the isothermal surface which is attached to three electrical resistors connected in series. Ten more thermocouples are distributed inside the insulation to control its temperature and to match it to that of the concrete by means of a controller. The "error of adiabatism" was calculated using a concrete specimen heated to three different temperatures of 40, 50 and 60°C. Tests were carried out at each of these tem-

peratures in which the concrete specimen was placed in the calorimeter and measurements were taken to determine how well the temperature was maintained. At 40 and 50°C there was no change but at 60°C the temperature of the block rose 0.1°C per 24 hours.

Another calorimeter [29] also developed in Italy uses a 300 mm aluminium cubic box as the isothermal surface with electrical resistance wires connected to each face. A 30 mm thick layer of insulation covers the inner surfaces of the box which is insulated from the environment by a 70 mm thick layer of insulation. The concrete specimen is contained within commercial polystyrene cubic moulds whose internal and external dimensions are 150 mm and 230 mm respectively. Two thermoresistances are used to detect the temperature of the concrete sample and the isothermal box. This calorimeter is available commercially produced and marketed by Controls.

A second calorimeter of this type is also available for purchase, the Stema calorimeter [30] developed and manufactured in Denmark. Fig. 1.7 shows a section of the equipment.

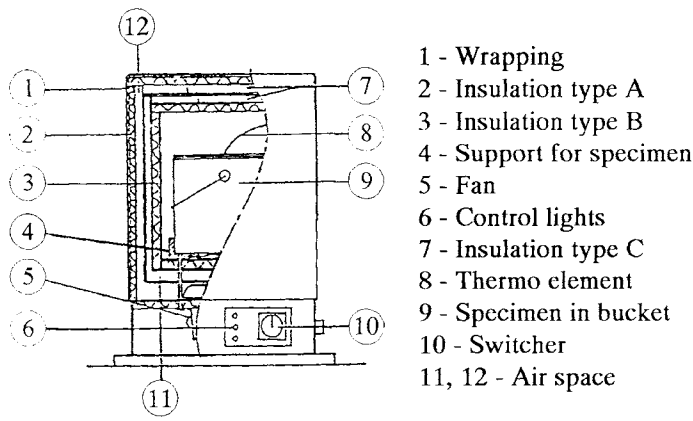

Fig. 1.7 Section of the Stema calorimeter.

1.3.2 Semi-adiabatic calorimeters

As mentioned in the introduction, this type of calorimeter is one in which the rate of heat loss from the concrete specimen is reduced only by some form of insulation, no external heat source is used to improve the efficiency of the apparatus.

These calorimeters can be broadly classified into two groups:

i) Those in which the insulation is provided by a thermos flask - the specimen size tends to be no more than about 2.5 kg.
ii) Those in which the specimen is surrounded by a layer of polystyrene or equivalent - the specimens tend to be cylinders or cubes and usually significantly larger than those used in (i).

According to the French standard, heat of hydration of cements can be measured using the Langavant test [6] which is essentially a semi-adiabatic calorimeter. The apparatus consists of a Dewar flask in which a 1.575 kg sample of 1:3 cement:sand mortar is placed within a water vapour tight cylindrical tin plate container. The temperature of the sample is measured using a thermometer connected to a recorder. No attempt is made to control the temperature of the environment in which the flask is placed to follow that of the sample. The temperature of the test specimen is recorded and compared with that of an inert specimen (i.e. one that is at least 3 months old) kept in a second reference calorimeter. Knowing the thermal capacity of the system and the recorded temperature differences between the reference and the active sample it is then possible to calculate the heat of hydration of the cement. There is no reason why such an apparatus should not be used for small concrete samples and why the adiabatic temperature curve should not be compacted from the semi-adiabatic curve.

The equipment developed by Grube [31] is in fact a modification of the Langavant test and is shown in Fig. 1.8.

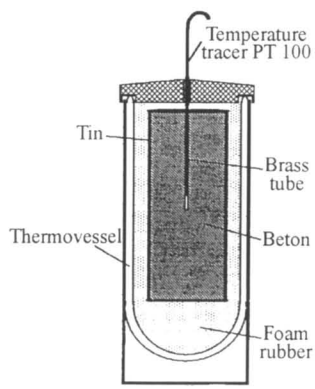

Fig. 1.8 Semi-adiabatic calorimeter developed by Grube.

The Dewar flask used is larger than in Langavant, the cylindrical concrete specimen measures 99 mm diameter x 230 mm high (i.e. approximately 4.25 kg) and is cast in a tin mould. The inside of the flask is protected by a lining of foamed rubber and the temperature of the concrete specimen is recorded using resistance thermometers. If required and as with Langavant the heat loss from the flask can be computed from a mature concrete specimen heated to a certain temperature and placed in the flask.

The calorimeters in which the specimen is surrounded by some form of insulating material differ only with respect to the size and shape of the test specimen and the thickness and type of insulation used. For example the test used at the Laboratoire Central des Ponts et Chaussées (LCPC) in Paris uses a relatively small sample (a 160 diameter x 320 mm cylinder) which is placed in an insulated box measuring approximately 400 x 400 x 550 mm high. The test is known as the "QAB" [32] semi-adiabatic test. At the other extreme the calorimeter developed by Breitenbücher [33] uses a cylindrical specimen measuring 360 mm diameter x 540 mm high which is placed in its mould inside a polystyrene mould which provides an insulation thickness of 56 mm top and bottom and 50 mm around the circumference. In addition an air gap of 2 mm is provided between the specimen mould and the polystyrene. The calorimeters developed by other workers [35-38] are all similar in principle to those described above.

The Nordic draft test method for an Insulated Block Calorimeter [34] states that a large scale well insulated concrete block shall be used for this test and it is assumed that the centre of the block will obtain approximately adiabatic conditions during the early stages of the hardening process. No technical specifications are given except that the cube should measure not less than 1 m and the coefficient of transmittance of the insulation shall be less than 5 kJ/(m^2.h.°C).

1.4 PREDICTION OF THE ADIABATIC TEMPERATURE RISE FROM ADIABATIC AND SEMI-ADIABATIC CALORIMETRY

The heat evolved during the hydration of a concrete sample in a calorimeter can be divided into the following three parts:

- heating of the specimen;
- heating of the calorimeter;
- heat loss.

The heating of the calorimeter is caused by the heat capacity of the components fitting the equipment. It should be kept as low as possible and should be accurately known. The heat losses play a different role for the two kinds of equipment. In adiabatic calorimeters they are usually small and almost constant along the duration of the test and give rise to a residual correction to determine the adiabatic temperature from the measured one. In semi-adiabatic calorimeters they are not constant throughout the runtime of the test as they increase with the increasing temperature of the sample and give the major contribution to the calculation of the adiabatic temperature rise. An example between measured and corrected curves is given in Fig. 1.9. The measured curve in an adiabatic calorimeter is usually only slightly lower than the corrected adiabatic curve whereas in a semi-adiabatic calorimeter the temperature of the sample is far below the adiabatic temperature; this in turn slows down the rate of hydration and, consequently, the corrections applied for heat losses will lead to a temperature rise which is still an underestimate of the adiabatic one.

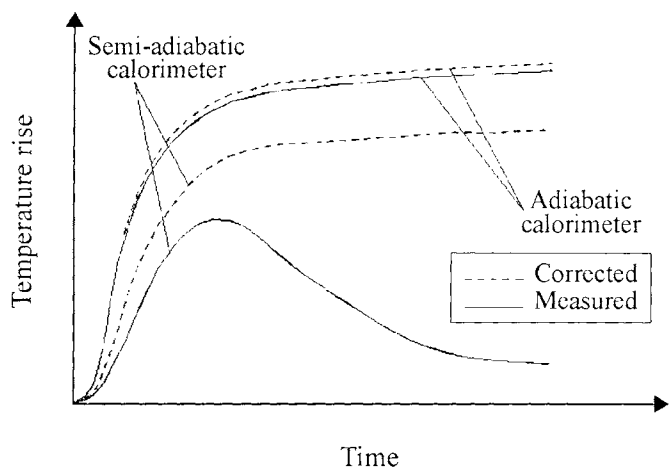

Fig. 1.9 Measured and corrected curves from adiabatic and semi-adiabatic tests.

To take account of the influence of the change in reactivity of the cement, some researches have adopted a further correction based on the assumption of a maturity function. This improves the estimate but however can give rise to some approximation in determining the true adiabatic temperature rise. Grube [31] considers the influence of temperature on

hydration by means of a maturity function based on the temperature factor of Arrhenius:

$$k_T = e^{\frac{E_A}{R}\left(\frac{1}{293}-\frac{1}{273+T}\right)} \qquad (1.1)$$

with: T temperature in °C;
E_A activation energy in J/mol;
R universal gas constant = 8.314 J/(mol·K);

$$\frac{E_A}{R} = \begin{cases} 4000 & for \quad T \geq 20°C \\ 4000 + 175 \cdot (20 - T) & for \quad T < 20°C \end{cases}$$ for Portland cements;

$\frac{E_A}{R} = 6000$ for slag cements.

The assumed maturity function is

$$M = \int_0^t k_T \, dt$$

In order to make comparisons between adiabatic and semi-adiabatic calorimeters a "Round Robin" test programme was conducted amongst some different organisations.

1.5 RILEM "ROUND-ROBIN" CO-OPERATIVE PROGRAMME

1.5.1 Adiabatic/semi-adiabatic calorimeter test programme

In order to obtain comparative data on the performance of a number of different types of calorimeters it was decided to carry out a "Round-Robin" test programme. The main aims of this programme were:

i) to compare the adiabatic temperature curves obtained from a number of different adiabatic calorimeters;
ii) to compare the predicted adiabatic temperature curves from semi-adiabatic calorimeters with those obtained from adiabatic calorimeters;
iii) to gain a greater understanding of the main factors affecting the results obtained from the different calorimeters.

1.5.2 Details of test programme

In all a total of 14 different organisations agreed to participate in the test programme. In order to minimise any errors all participants were sent appropriate quantities of the same materials and all were asked to use the same mix proportions.

Materials Used. - Samples of aggregate and cement were delivered in sealed air tight containers. The coarse aggregate was a 20 mm graded Thames Valley flint conforming to the requirement of BS 882 [39]. The fine aggregate was from the same source and was a mixture of five separate size fractions re-mixed before use to ensure conformity of grading. All aggregates were supplied in a saturated and surface dry condition.
The cement used was ordinary Portland cement (OPC) conforming to the requirements of BS 12 [40].

Mix Proportions. - Participants were requested to mix the materials in the following proportions by weight:

Cement	1.0
Fine	2.5
Coarse	3.5
Water	0.6

1.5.3 Results

The results from both the adiabatic and semi-adiabatic calorimeters are summarised in Tables 1.1 and 1.2 respectively.

Of the nine adiabatic results presented in Table 1.1, that of Luleå University is significantly lower than the rest; no reason can be given for this and for the purpose of this discussion this result will be discounted. The mean and the highest and lowest variability calculated at different ages are given in Tables 1.3.

Table 1.1 Summary of results from adiabatic tests

Laboratory	*Calorimeter*			*Duration of the test [h]*	*Measured temperature [°C]*			*Corrected temperature [°C]*				*Comments*
	Control Medium	*Sample size [l]*	*Thermal loss [K/h]*		*Start*	*Final*	*Total rise*	*Total Rise*	*Rise after 24 h*	*Rise after 48 h*	*Rise after 72 h*	
A	Water	7.5	-	240	22	70	48	48	35.8	43.4	45.2	Average of 2 runs
B	Water	50	0.029	168	22.8	65	42.2	44.0	36.3	40.8	41.7	
C	Heated container	1.5	0.013	168	20.0	68.6	48.6	48.6	37.3	44.8	46.6	
D	Water	25	0.002	168	22.0	70.0	48.6	49	40	46	47.5	Correction for heat loss and heat capacity of the calorimeter
E	Water	4	-	160	25.9	69.8	43.9	43.9	36.1	40.9	42.1	
F	Water	0.5	0.01	264	22	62.3	40.3	42.5	31.9	38.6	40.4	Average of 2 runs; correction for heat loss
G	Water	170	-	319	20	68.5	48.5	48.5	38.5	44.7	46.3	
H	Air	10	0.008	50	22	54.8	32.8	32.8?	29	32.1	-	
I	air/water	30	0	168	20	72.5	52.5	52.5	38	45.1	47.6	Negligible heat loss

Table 1.2 Summary of results from semi-adiabatic tests.

	Calorimeter		*Measured temperature [°C]*			*Corrected temperature [°C]*			
Lab.	*Type of insulation*	*Sample size [l]*	*Start*	*Time to peak [h]*	*Rise at time to peak*	*Rise after 24 h*	*Rise after 48 h*	*Rise after 72 h*	*Comments*
B	50 mm polystyrene	50	22.6	32	34.1	36.9	43	44.6	Correction by the method proposed by Grube
J	Dewar Flask	2	20	17	18	38	44	46.6	Average of 2 runs; adjustments made using method proposed by Grube
C		6.4	22.8	21	23	31.4	36.6	-	Adjustment made using similar method to that proposed by Grube
K		13	20	19	26	35.5	41	42.5	Adjustments made according to a maturity function
H	1 m^3 foamed plastic	15	20.4	21	23.7	-	-	-	No adjustments performed
L	?	5	22.3	19	26.5	37.6	43.3	44.8	Adjustments made using a similar method to that proposed by Grube

Table 1.3 Variability of adiabatic and semi-adiabatic test results.

	Rise after 24 hours		*Rise after 48 hours*		*Rise after 72 hours*	
	adiabatic	*semi-adiabatic*	*adiabatic*	*semi-adiabatic*	*adiabatic*	*semi-adiabatic*
Mean values [K]	36.7	35.9	43	41.6	44.7	44.6
Highest variability	+8.9%	+4.8%	+6.9%	+5.8%	+6.3%	+4.4% *
Lowest variability	-13.2%	-12.5%	-10.3%	-12%	-9.6%	-4.8% *

* Calculated on 4 tests

Looking at the results from adiabatic calorimeters, some laboratories did not apply any correction to the measured curves whereas others adopted corrective methods which can differ each other and that in some tests represent an increase of 4-5% to the measured curves. It is not possible to say what the effect on the overall test variability would be if everybody had corrected their results although it is unlikely that it would have a significant effect. However, 50% of the adiabatic tests fall into a narrow range of only 2K of variation of temperature rise. There is no obvious correlation between the temperature rise and either the specimen size or the start temperature.

As concern the results from the semi-adiabatic calorimeters, the mean values of the results are only of the order of 2-3% below those derived from adiabatic calorimeters; of the five tests, the data supplied from LCPC are significantly lower than the remainders four whose range of variation is within 3 K. This would suggest that the adiabatic temperature rise could be accurately derived from a semi-adiabatic calorimeter. However, it must be remembered that the results are derived from one set of tests with one cement content and one cement type. A far more substantial test programme is required using different mix proportions and more importantly different cement types to substantiate this fact. No other meaningful conclusions it is possible to draw about the influence of specimen size, measured temperature or method of adjustments on the corrected temperature rise; it is however reasonable to think that a standard procedure to how calibrate a calorimeter and how calculate the adiabatic temperature rise from the measured curves should lead to a reduction of the variability among different laboratories.

1.6 DRAFT RECOMMENDATIONS

From discussions by all the experts of the RILEM Technical Committee 119 and from results and experiences of the Round Robin test it was

agreed that the major influences over errors arising out of any form of adiabatic and semi-adiabatic calorimeter would be as follow:

a. The design of adiabatic calorimeter. Adiabatic calorimeters, by definition, must have a means by which the temperature of the enclosure surrounding the sample is carefully controlled. It was agreed that due to the physical and thermal properties of water that this should be the recommended medium with which to surround the sample for ease of circulation and close temperature control.

b. The thermal loss from adiabatic calorimeter. This should be of course be kept to a minimum in order to obtain as close as possible the adiabatic conditions required and to avoid large corrections to compensate for any non-adiabacity. Any large deviation from adiabatic conditions means that the cement will be subjected to a lower temperature than it should and, as cement hydration rate is accelerated by temperature to the degree of twice for every 10°C, the correct rate of reaction will not be obtained. It was agreed that the temperature loss of the sample should be less than 0.02 K/h.

c. The heat capacity of the calorimeter. This parameter produces the same effects of the thermal loss on the measured temperature rise. It must be kept much lower than the heat capacity of the sample. This can be achieved by testing large volumes of sample. The ratio between the apparent heat capacity of the calorimeter to the heat capacity of the sample should be limited to 0.1; if the condition is not satisfied a thermal compensation technique such that suggested by Morabito (1993) should be adopted.

d. The sensitivity of the temperature controller. This influences the amount by which the temperature of the control medium cycles around the set point and, of course, will even out to a certain extent. This variation must not exceed the 'adiabatic setting' otherwise there will be a net heat input to the system. The setting should be such to limit the thermal losses to 0.02 K/h. The layer of insulation between the sample and the control medium makes less critical the sensitivity of the temperature controller.

e. The concrete sample size. It was thought that any adiabatic or semi-adiabatic test method on concrete should take into account the thermal capacity effects of a maximum aggregate size of 32 mm and that the

smallest dimension of the sample should be at least three times the maximum size of the aggregate.

At last, both types of calorimeter must be calibrated for the thermal losses and the effects of the heat capacity of the equipment. These factors are incorporated into the following relationship:

$$\vartheta_{ad} = \left(1 + \frac{C_{cal}}{C_c}\right) \cdot \left[\vartheta_s(t) + \int_0^t a(t) \cdot dt\right] \tag{1.2}$$

where t is the time, C_{cal} and C_S [J/K] are the apparent heat capacity of the calorimeter and the heat capacity of the sample respectively, $\theta_S(t)$ the measured temperature rise, $a(t)$ the coefficient of temperature loss [K/h]. If $\theta_S(t)$ is measured by an adiabatic calorimeter then $T_{ad}(t)$ represents the adiabatic temperature rise of the concrete; if the test is performed by a semi-adiabatic calorimeter equation (1.1) leads to a lower estimate of the adiabatic temperature rise. The estimate is improved by considering the influence of the temperature on the hydration by means of a maturity function. In general, the temperature factor of Arrhenius gives a good approximation for the temperature dependence of the hydration of the cement.

The ways to how calibrate the two types of calorimeter and how calculations can be carried out are given in a RILEM Technical Recommendation.

1.7 APPLICATIONS OF ADIABATIC AND SEMI-ADIABATIC CALORIMETRY

The choice of type and design of a calorimeter will depend almost entirely on the reasons why the test is being carried out. If an estimate of the temperature rise in a particular pour is all that is required then some form of semi-adiabatic calorimeter will be sufficient as it can be argued that the conditions of this test come closer to those existing in a real structure than any form of adiabatic test. However the accuracy with which the measurement is required will also dictate the type of equipment used.

If the true adiabatic temperature rise and heat of hydration are required then it could be argued that an adiabatic calorimeter will give a more accurate result. However, even though the temperature loss in adiabatic calorimeter is much lower than in a semi-adiabatic calorimeter,