



TA Instruments
TAM AIR

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Lindon, UT USA

Elstree, United Kingdom

Shanghai, China

Beijing, China

Taipei, Taiwan

Tokyo, Japan

Seoul, Korea

Bangalore, India

Paris, France

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Milano, Italy

Barcelona, Spain

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Mexico City, Mexico



TAM AIR

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TAM AIR ISOTHERMAL MICROCALORIMETRY



The ideal tool for large scale calorimetric experiments,
capable of measuring 8 samples simultaneously

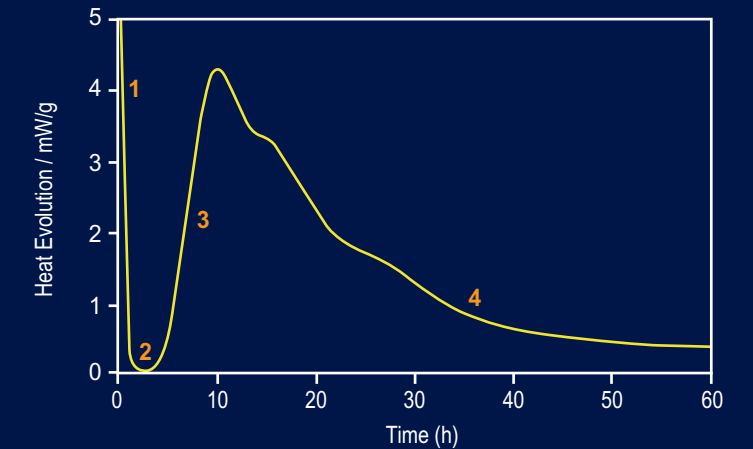
A POWERFUL TOOL FOR THE STUDY OF CEMENT HYDRATION PROCESSES

Determining the heat of hydration of cement is important and traditionally, the heat of hydration has been determined by measuring the heat of solution (ASTM C186). More recently, isothermal calorimetry tests using TAM Air are increasing because it accurately and reliably measures the heat of hydration (ASTM C1702). The samples tested in the TAM Air are usually paste samples, where the cement hydration process can continuously be followed over time. The shape of the heat flow curve will reflect the cement hydration process and the different phases of the complex process can be determined. The addition of admixtures will change the shape of the heat flow curve, and the admixture effect can be quantified. The integrated heat flow over time will give the extent of hydration. Using isothermal calorimetry, the heat of hydration is measured with TAM Air by monitoring the heat flow from the specimen while both the specimen and the surrounding environment are maintained at the same temperature. The TAM Air is widely used for studying the reaction kinetics of pure cement pastes as well as the temperature dependence of the reaction. TAM Air is an excellent tool for quality control in cement plants, for optimization of additives to give a cement a certain property as well as a general research tool for the cement laboratory.



Instrument of choice for standardized testing on cement

- Phase 1:** Rapid initial process - Dissolution of ions and initial hydration
- Phase 2:** Dormant period - Associated with a low heat evolution and slow dissolution of silicates
- Phase 3:** Acceleration period - Silicate hydration
- Phase 4:** Retardation period - Sulphate depletion and slowing down of the silicate hydration process



TAM AIR SPECIFICATIONS



Thermostat Specifications

Calorimeter Positions	8
Operating Temperature Range	5 - 90 °C
Thermostat Type	Air
Thermostat Stability	± 0.02 °C
Limit of Detection	4 µW
Precision	±20 µW

Calorimeter Specifications

Short Term Noise	< ± 2.5 µW
Precision	± 20 µW

Baseline over 24 hours

Drift	<40 µW
Deviation	<±10 µW
Error	<±23 µW

TAM AIR TECHNOLOGY

Monitoring the thermal activity or heat flow of chemical, physical and biological processes provides information which cannot be generated with other techniques. Isothermal microcalorimetry is a powerful technique for studying heat production or consumption and is non-destructive and non-invasive to the sample. The TAM Air offers unmatched sensitivity and long term temperature stability with flexible sample requirements.

When using microcalorimetry there is little or no sample pretreatment required; solids, liquids and gasses can all be analyzed. Unlike other techniques that may only give time interval snapshots of data, microcalorimetry presents continuous real-time data that reflects the process or processes taking place in the sample.

High Performance Temperature Control and Stability

The TAM Air is an air based thermostat, utilizing a heat sink to conduct the heat away from the sample and effectively minimize outside temperature disturbances. The calorimeter channels are held together in a single removable block. This block is contained in a thermostat that uses circulating air and an advanced temperature regulating system to keep the temperature very stable within $\pm 0.02^\circ\text{K}$. The high accuracy and stability of the thermostat makes the calorimeter well suited for heat flow measurements over extended periods of time, e.g. weeks. The baseline drift is less than $40 \mu\text{w}/24$ hours with very low short term noise. TAM Air Assistant™, a powerful, flexible and easy-to-use software package is used for instrument control, experimental setup, data analysis and reporting of results.

Isothermal Microcalorimetry

When heat is produced in a sample, isothermal microcalorimetry measures the heat flow. The sample is placed in an ampoule that is in contact with a heat flow sensor that is in also in contact with a heat sink. When heat is produced or consumed by any process, a temperature gradient across the sensor is developed. This will generate a voltage, which is measured. The voltage is proportional to the heat flow across the sensor and to the rate of the process taking place in the sample ampoule. This signal is recorded continuously and in real-time.

For each sample there is a reference that is on a parallel heat flow sensor. During the time that the heat flow is monitored, any temperature fluctuations entering the instrument will influence both the sample and the reference sensors equally. This architecture allows a very accurate determination of heat that is produced or consumed by the sample alone while other non-sample heat disturbances are efficiently factored out.



TAM Air 8-Channel Calorimeter

The TAM AIR 8 Channel calorimeter consists of an eight (8) channel calorimeter block and data logging system required for use with the TAM AIR thermostat. The calorimeters are twin-type (sample and reference), and designed for use with 20 ml glass or plastic ampoules or the 20 ml Admix ampoules.

TAM AIR AMPOULES

The ampoules used in the TAM Air are designed to handle up to 20 ml volumes. Either glass, stainless steel or plastic (HDPE) closed ampoules are available, which enables maximum flexibility for sample management and maximum sensitivity.



Admix Ampoule

The Admix Ampoule is a 20 ml accessory available for initiating reactions inside the calorimeter, and can be used for monitoring a reaction from the initial injection. The Admix ampoule can be configured with or without a motor for stirring. For suspensions such as mixtures of cement and water, manual stirring is recommended. For liquid systems, a motor may be used for stirring. The admix ampoule can only be used with 20 ml disposable glass ampoules.

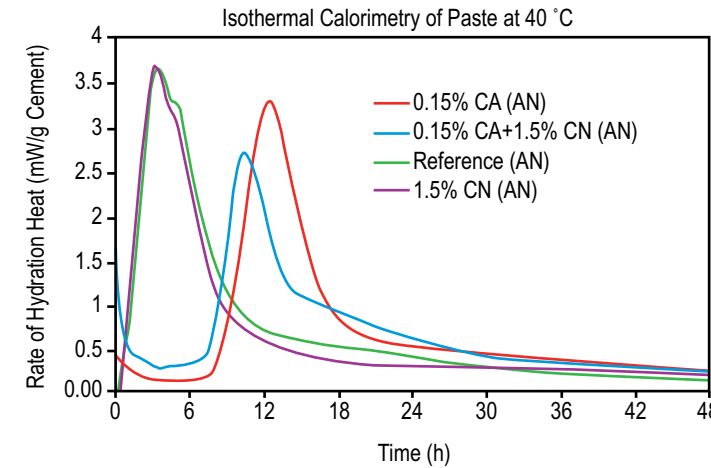
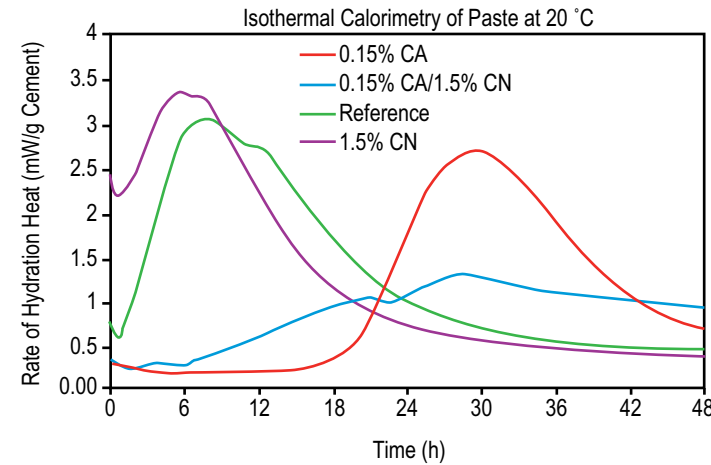


APPLICATIONS

Cement Paste Setting Time

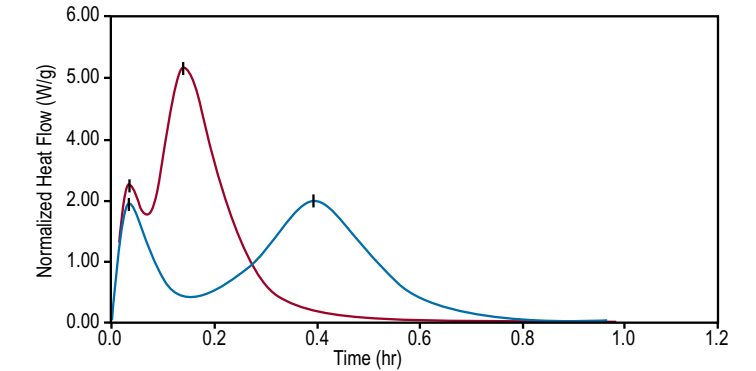
The synergy of citric acid (CA) and calcium nitrate (CN) is clearly seen from the rate of hydration heat. CA is essentially a setting retarder relative to the reference, although the heat of hydration is slightly reduced and CN is clearly a setting accelerator. Together they behave as a hardening retarder lowering the rate of hydration heat and distributing it over a longer time.

The rate of hydration heat for the same mixtures at 40 °C shows that the function as hardening retarder is reduced at higher temperature. This data along with the cumulative heat data indicate that the admixture combination may not function in the practical semi-adiabatic case of massive concrete.¹



Hydration of Calcium Sulfate Hemihydrate

Identical samples of 2g of Calcium Sulfate Hemihydrate powder were mixed with a hydrating agent at a liquid to solid ratio of 0.50 using an admix ampoule in the TAM Air. The blue curve shows a sample hydrated with deionized water. The red curve is a sample hydrated with a 5% Sodium Chloride solution. It is demonstrated that sodium chloride accelerates the calcium sulfate hydration reaction.

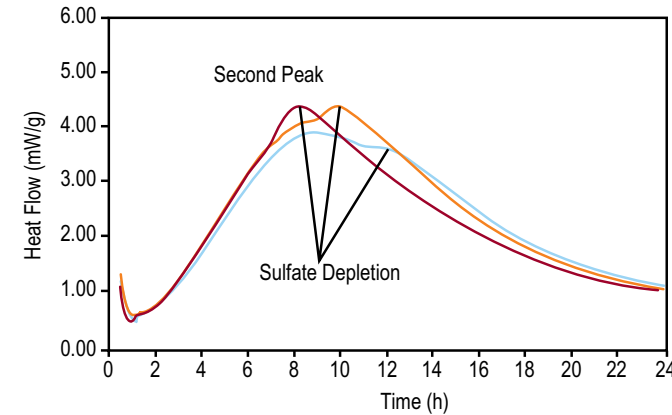


APPLICATIONS

Cement Sulfate Depletion

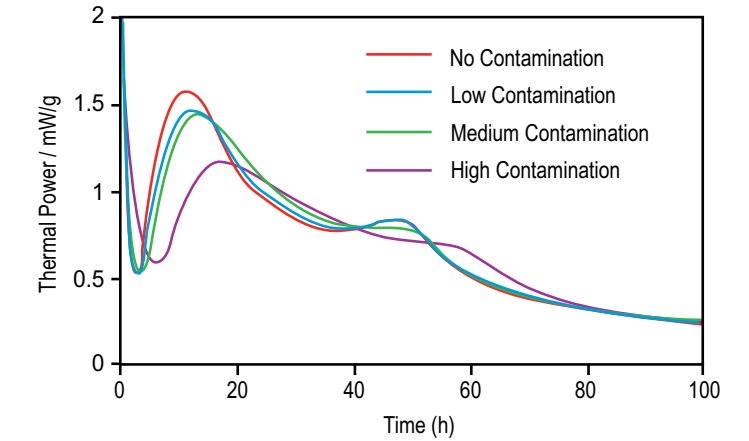
This figure shows how Lerch's criteria - sulfate depletion peak - can be used for a rapid indication of the optimum sulfate content of a laboratory ground clinker. The results of the laboratory screening shown indicates that 2.5% added SO_3 might be sufficient to bring the resulting Portland cement to optimum SO_3 level. Several factors may cause cement manufactured in the field to perform different as compared to the laboratory ground cement.

Calorimetry serves as an excellent indication as to the approximate values to aim for to avoid setting time and admixture incompatibility issues. Furthermore, the calorimetry can be used to assess the efficiency of any changes made to the cement production, such as changes in gypsum type or changes in raw material or fuel that may influence the reactivity of the aluminate phase and thereby the demand for soluble SO_3 .²



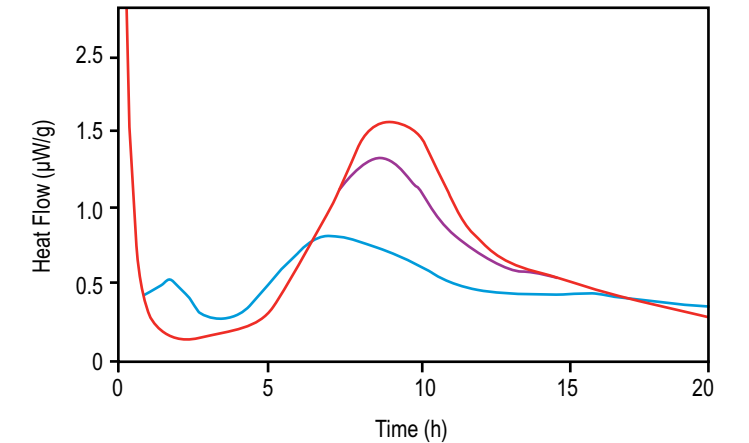
Cement Thermal Profiles with Contaminants

Cement setting thermal profiles can be influenced by contaminants. The graph shows the steady decrease in thermal power as the contamination of the cement mortar by a mixture of soil and sawdust increases (0; 0.9; 2.5 and 5.9% of $w/c=0.6$ cement mortar). Influence on hydration rate of a mixture of soil and sawdust.³



Setting Time of Cement

The TAM Air calorimeter has been shown to be excellent for diagnosis of problems related to setting time and premature stiffening of cement. The blue curve in the figure to the right represents an industrial cement produced with too little soluble calcium sulfate. This cement suffers from early stiffening because of the aluminate reactions at 1-1.5 hours hydration. It also suffers from low early strength, because the aluminate hydrates formed retard the strength-giving silicate hydration indicated by the unusually small silicate peak at 5-10 hours. When 0.5% (purple curve) and 1.0% (red curve) of calcium sulfate hemi-hydrate was added to the cement the undesired early peak disappeared, and the strength-giving silicate peak regained its normal shape. The results indicate that premature stiffening is caused by a lack of soluble calcium sulfate.

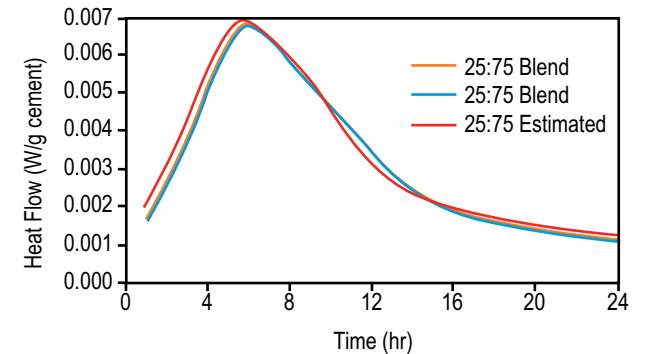
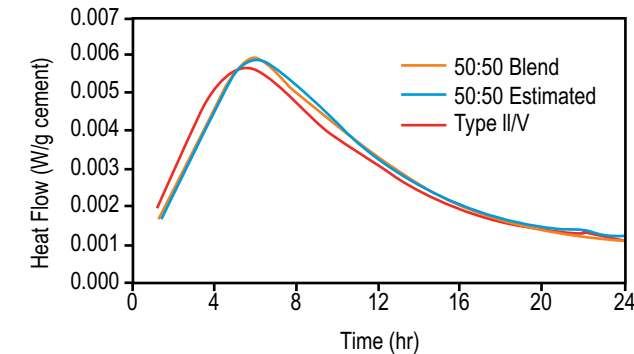
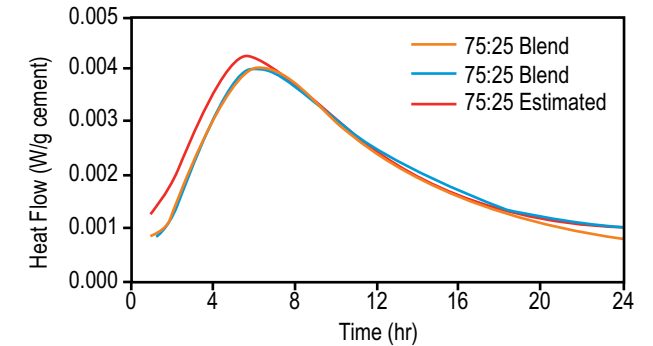
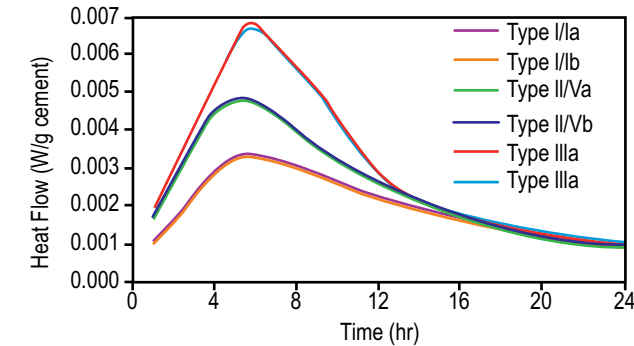


APPLICATIONS

Cement Blending

This figure provides plots of the heat release rate (heat flow) for the first 24 h of hydration for six cement pastes examined by Isothermal calorimetry (3 pure and 3 blends). In general, results for the two replicate specimens for each cement paste fall directly on top of one another. For the three initial cements, the heat release during the first 24 h increases with increasing cement fineness, as would be expected due to the increased (in contact with water) surface area. Interestingly, for these six cements based on a single clinker, the peak in heat release rate always occurs at about 6 h, while by 24 h, the heat release rate has diminished to a value close to 0.001 W/g cement.

The heat flows measured during the first 24 h for the three blended cements are predicted quite well by applying the simple law of mixtures. The results imply that for the $w/c = 0.4$ cement pastes examined in this study, the particles are likely hydrating independently of one another during the first 24 h, such that the degree of hydration of blends of the fine and coarse cements can be quite accurately computed simply as a weighted average of their (measured) individual hydration rates. ⁴



APPLICATIONS

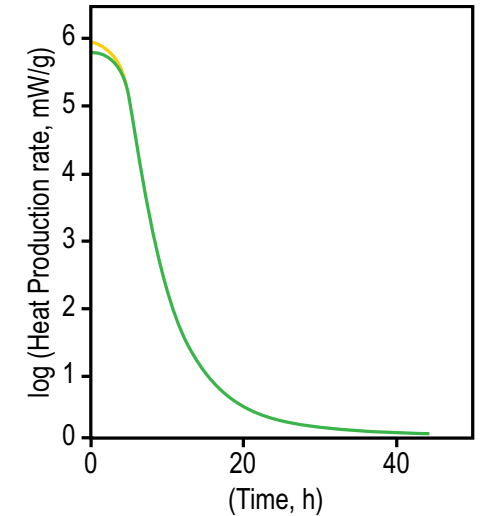
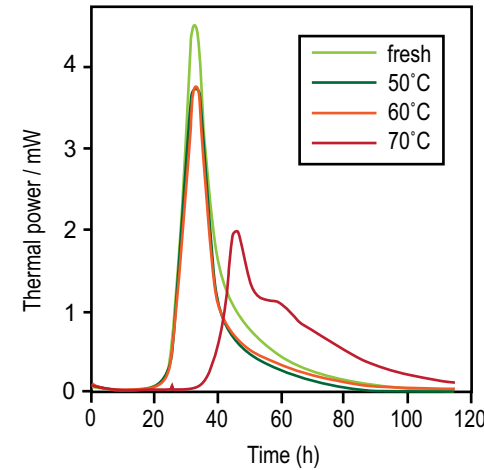
Food Testing

This figure shows the Thermal treatment of carrot juice resulting in increased shelf life only at the highest treatment temperature.

The measured thermal power is the heat from the micro-biological activity in the sample. It is seen that the lower treatment temperatures gave only slightly lower thermal powers, but that the 70 °C treatment gave a substantially delayed signal. At 20 °C the shelf life was thus increased by more than 50% by the 70 °C treatment.⁵

Epoxy Curing

Here we see the heat production and the heat production rate as a function of time. It can be seen that the spread of results is low and that after an initial reaction period of five hours the heat production rate decrease is similar to an exponential decay. After 45 h the thermal power is approx. 0.06 mW/g, i.e. 600 μ W for a 10g sample. As the detection limit for TAM Air is better than 3 μ W it would still be possible to follow the reaction for an even longer time than was done here.⁶



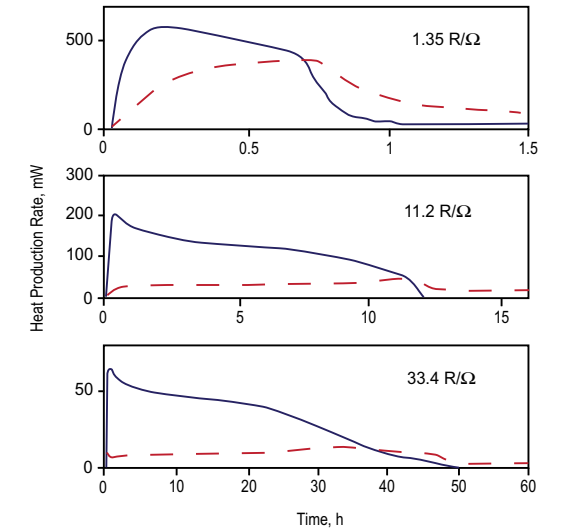
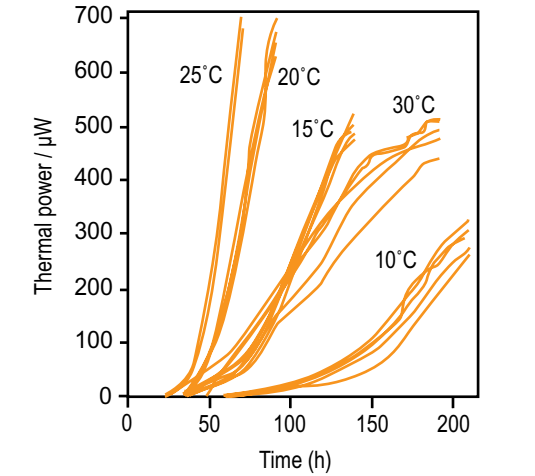
Fungal Growth

At each temperature multiple inoculated specimens were measured. This figure shows the results at the five temperatures. It is seen that the results for each temperature agrees rather well with each other. Calorimetric measurements can be a valuable addition to the measurement techniques for predictive microbiology.⁷

TAM Air Battery Testing

The properties of batteries during discharge with three different resistance loads are shown. Single channels in TAM Air were charged with 1.5 V alkaline batteries, size AAA. Three resistors of different values were placed in an adjacent channel for connection to the batteries. The solid line represents the useful energy in the battery which is the heat production measured in the resistor, while the dotted line is the heat production from the battery itself, i.e. the internal losses.

The batteries were fully discharged during the course of the evaluation in the TAM Air. The lowest resistances cause a rapid drain of the battery (e.g. as in a flashlight) whereas the highest resistances cause a very low rate of discharge (e.g. as in an alarm clock).⁸





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