Thermal Hazard Technology, 1 North House, Bond Avenue, Bletchley MK1 1SW, England.
Phone: +44 1908 646700 Fax: +44 1908 645209 E-mail: science@easynet.co.uk Web: www.science.org.uk

Technical Information Sheet No 11

Isothermal Age Testing with the Thermal Hazard Technology Accelerating Rate Calorimeter

Two methodologies have been developed to all stable isothermal operation of the THT Accelerating Rate Calorimeter. One of these is quasi-adiabatic and the other truly isothermal. In the latter it is the calorimeter jacket temperature that is the controlled temperature and in the former control is from the bomb thermocouple. The quasi-adiabatic isothermal mode was that originally devised. Though good for detection of exothermic reactions at high sensitivity, this method is problematic in that the temperature of control is not strict and drift will occur. The extent or rate of drift will depend on the quality of calibration of the instrument. Therefore in the original instrument an Isothermal Window of 2°C was advised and the system would drift within this band.

Quasi-adiabatic Isothermal Mode.

To improve on this a unique methodology was developed to all much better isothermal temperature control, this is used in the THT Accelerating Rate Calorimeter. The result of this is that Isothermal Age tests can be run with the following features.

- A very narrow temperature limit (or window) is used.
- This limit operates both above and below the isothermal temperature.
- Negligible overshoot occurs should some ‘heat’ be required.
- There is automatic modification of the calibration offset.

This latter feature is the key part. During the test to minimise drift, the rate of drift is determined and the calibration offset modified.

In an isothermal age test with the THT Accelerating Rate Calorimeter the temperature limit (window) may be set in the range 0.2 - 0.5°C. Therefore at the smallest recommended limit the system will be held to the isothermal temperature +/ - 0.2°C. The average temperature during the age period will then be very close to the chosen isothermal temperature itself (not 1°C below which usually was the case before now if a 2°C window is used). If 'heat' is required an overshoot of approximately 0.1°C may occur but this overshoot is rapidly 'lost' by natural cooling.

With the original method, isothermal temperature drift of the system may be 0.01°C/min (or possibly higher). High rate of drifting and a large temperature band (1°C or 2°C) are not acceptable for accurate isothermal age work because the system is not reproducibly held at a temperature sufficiently close to the isothermal temperature. This problem is eliminated with the THT system as it monitors the temperature variation with time and modifies the calibration offset value to minimise this drift. The drift will be minimised to +/- 0.001-0.002°C/min. The result is therefore a stable temperature very close to the isothermal temperature

Such performance of the THT Accelerating Rate Calorimeter is in part made possible because all calorimeter assemblies provided by THT are constructed with highest quality thermocouples, i.e the sets used by THT are matched to the highest precision. Thus calibration offsets are very often below 1.0µ at all temperatures.

The result is that the THT Accelerating Rate Calorimeter will allow excellent temperature stability within an accurately defined temperature band over any time period. Such performance allows, the Accelerating Rate Calorimeter for the first time, reliable and repeatable induction time studies to be carried out.

Typical results from tests at 75°C and 150°C are shown here.

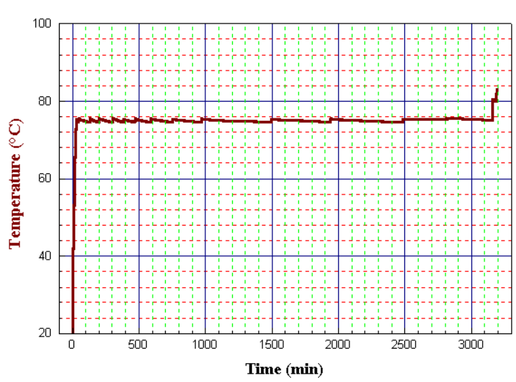
An isothermal age test over a time period of more than 2 days at 75°C is shown in Figure 1. This and all following figures are simply temperature against time. The temperature is that of the bomb as measured in the usual way and time is real time, zero being at the start of heating. In this test the temperature limit was set at +/- 0.5°C.

Figure 2 shows the result on a much enlarged scale. Here it is possible to see.

* The downward drifting
* The reset of temperature by 1°C (+ and - 0.5°C)
* The minimal temperature overshoot that is rapidly 'lost'
* The reduction in downward drift rate due to 'auto-correction'

Also it can be seen that when the temperature drifts upwards at a minimal rate a further correction is carried out (see data at 2900 minutes).

From Figure 2 it is possible to calculate the drift data at any temperature. For example between 2000 minutes and 2500 minutes the temperature decreases by 0.75°C - an average rate of - 0.0015°C/min.



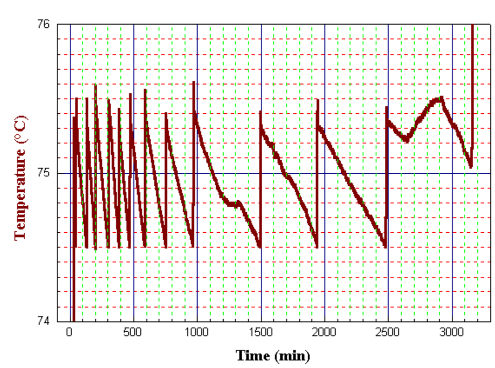


Fig 1 Fig 2

Figure 3 further 'zooms' the data. Figure 4 shows the result obtained at 150°C. Here the isothermal temperature window is +/- 0.2°C.

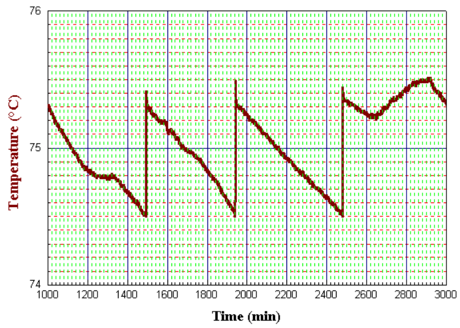


Fig 3

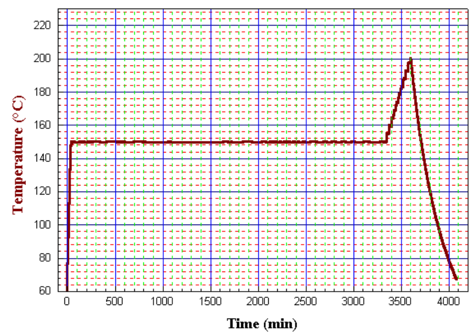


Fig 4

Figure 5-7 zoom the data and Figure 8 further zoom part of the data to a level where the individual data points can be seen. Data has been recorded every 30 seconds with a temperature precision of 0.01°C.

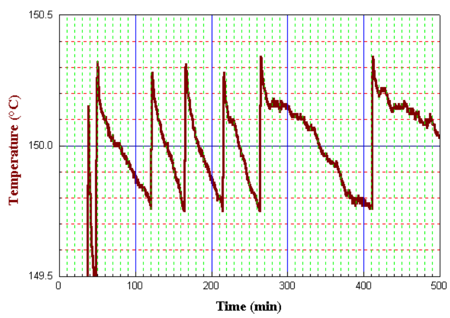


Fig 5

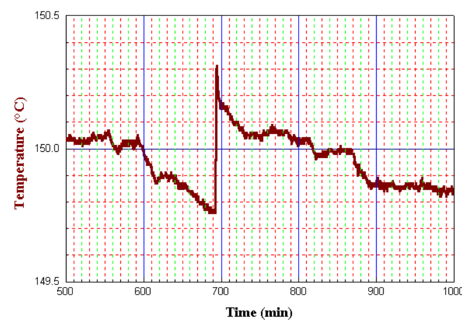


Fig 6

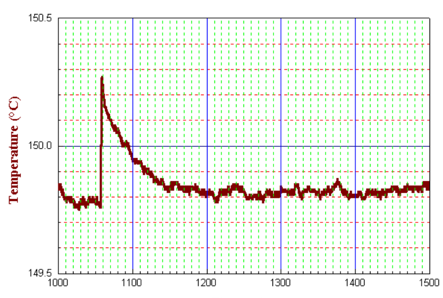


Fig 7

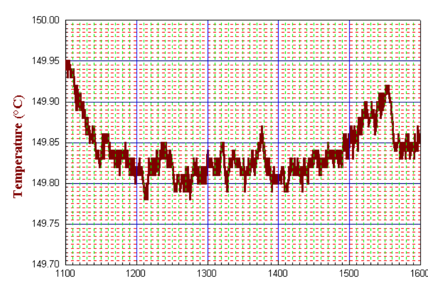


Fig 8

True Isothermal Mode.

The self-calibrating mode minimises drift but can never eliminate it. To give even greater isothermal precision, the THT instrument can be controlled from the side jacket thermocouple. The instrument is then working as a truly Isothermal Calorimeter.

It should be realised in this method that detection of exothermic reaction will not be at such a high sensitivity as in the quasi-adiabatic mode. It should also realised that in this mode the concept of ϕ does not hold. Phi has relevance and effect but its determination may not be precise. This method also can be used safely with the THT low-ϕ containers and it is possible to measure heat output rates.

Fig 9, Fig 10 and Fig 11 illustrate the result of a test carried out at 125°C. It can be seen that the stability is perfect.

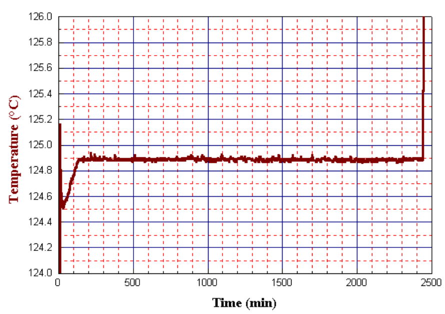


Fig 9

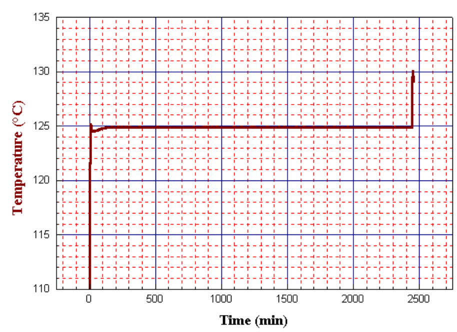


Fig 10

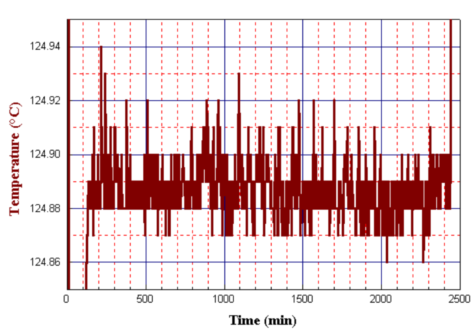


Fig 11