Technical Information Sheet No 70

Calibration of the Accelerating Rate Calorimeter

Calibration of the Accelerating Rate Calorimeter is necessary such that a stable isothermal temperature will exist during the Seek mode. It is not a calibration in the usual sense of the word as it is more of an instrument set-up, however the term calibration has historically been used.

The calibration test uses a specific method to measure the isothermal stability and minimise the temperature drift. It is usual to calibrate over the temperature range 50 - 400°C to obtain a stability of greater than 0.01°C/min. This is appropriate when standard tests are required.

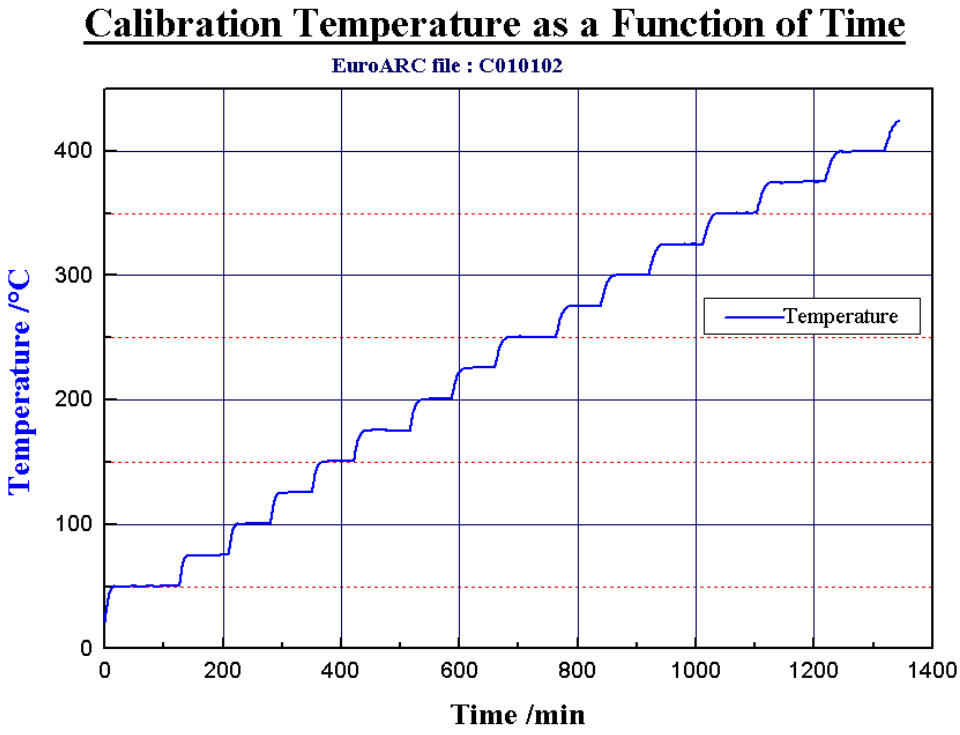
The calibration is required because in use the Accelerating Rate Calorimeter compares the temperature of the “bomb thermocouple” with that of the “side zone” thermocouple. Though at an isothermal temperature these will read the same temperature, the output in mV will be unlikely to be the same.

What is a calibration?

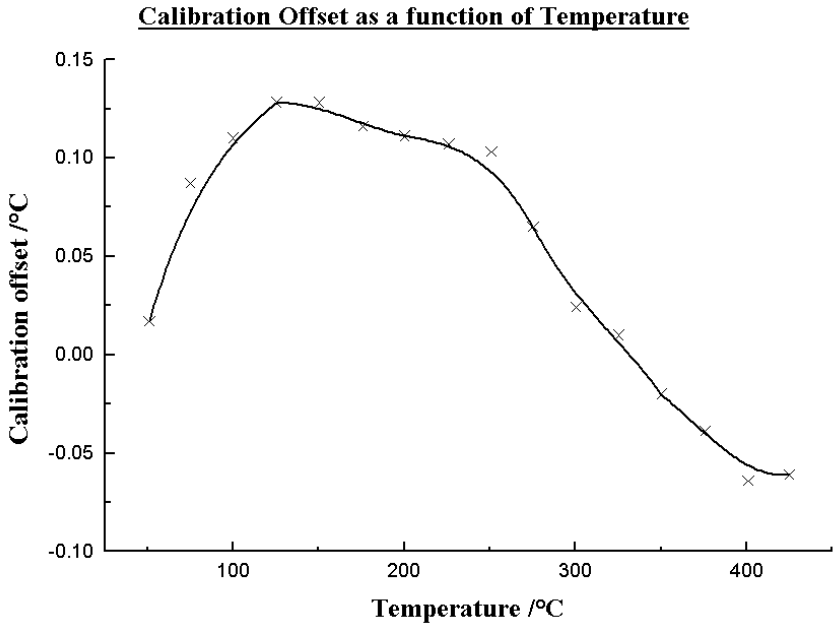
The calibration determines the difference in mV between the two thermocouples at various temperatures. It determines an "offset" that is then applied such that in use when the temperatures are identical so will the voltage output that is assessed by the instrument. The calibration is performed by an iterative process and requires offsets that give a drift reproducible to below the chosen sensitivity (usually ±0.01°C/min) before proceeding to measure at the following temperature.

The more closely matched the thermocouples the smaller the measured offsets will be. With time, or misuse the thermocouple mV output changes and re-calibration becomes necessary.

Table showing calibration offsets as a function of temperature and Graph showing typical calibration temperature and time.



Temp /°C	Offset /°C
51.1	0.017
75	0.087
100.1	0.11
125.4	0.128
150.3	0.128
175.8	0.116
200.4	0.111
225.9	0.107
250.9	0.103
275.1	0.065
300.4	0.024
325.1	0.01
350.2	-0.02
375.7	-0.039
400.8	-0.064
425	-0.061



What is a good calibration?

1. Small offsets (+ve or -ve)
2. Smooth curve through the origin produced when offset is plotted against temperature. N.B. This curve may flatten or invert at higher temperatures. (as shown above)
3. Consistent length of calibration step - the time at each temperature should be roughly equal (not more than 2 hours)
4. Repeat values from previous test should be better than 10% except where replacement thermocouples have been introduced.

When should you recalibrate?

Calibration should be carried out:

1. After bomb rupture.
2. If it is determined that temperature stability is outside acceptable boundaries. (see below)
3. After replacement of the bomb thermocouple.

During all sample tests the temperature drift during the “Seek” phase can be used as a measure of thermal stability and the validity of the current calibration. Alternatively a drift check can be used to “check” the validity of the current calibration. Drift checks are detailed in Ti sheet 72.

Calibrations should be carried out with a low mass titanium bomb. These results should be applicable to all usual bomb types and it is unnecessary to recalibrate when changing from one type to another. It may be necessary to recalibrate if a specific sample holder of unusual dimension or shape is used especially if this disrupts the radial position of the bomb thermocouple.

The data given here have been collected with a THT design square calorimeter, which is approximately 4 years old.

At a sensitivity of 0.01°C/min providing the thermocouples are functioning normally and there is no detrimental chemical contamination there should be no problems achieving satisfactory calibration to 450°C or above. A new calorimeter will provide better performance and calibration can be achieved at higher sensitivities. However these data show the quality of data that can be measured with old equipment.