Get the most reliable heat measurement from your sample with 3D sensors

2D SENSOR

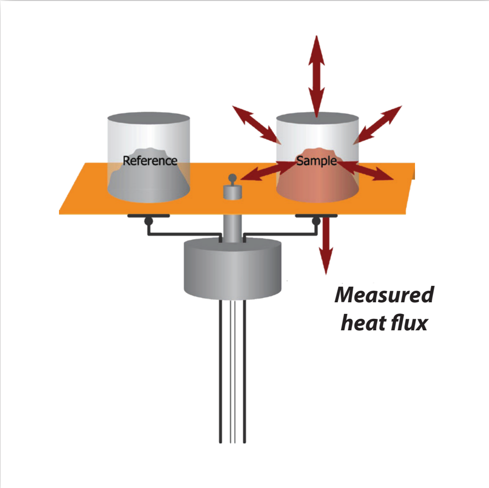

Figure 1a - Schematics of a 2D sensor

3D SENSOR

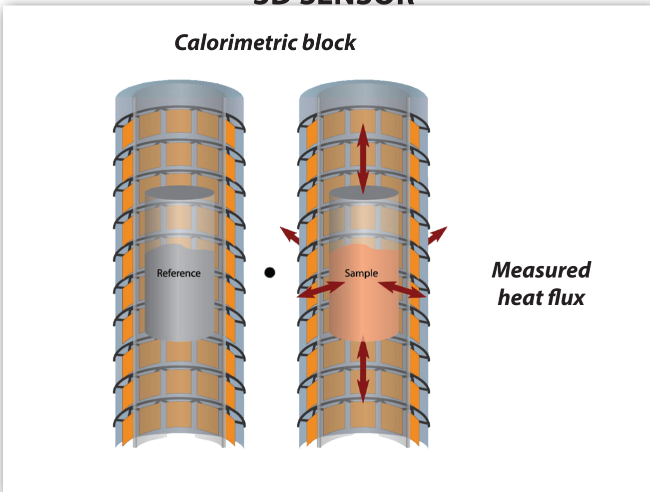

Figure 1b - 3D calorimetric sensors

SURFACE MEASUREMENT

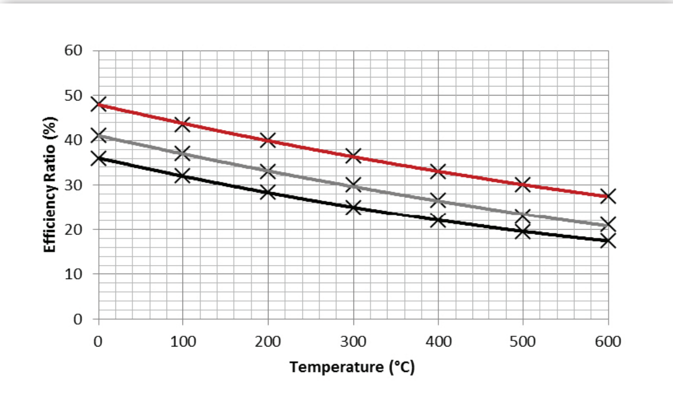

Figure 2a

Measured heat = **30-50%** of the total heat evolved by the sample + **70-50%** from heat calibration.
High calibration uncertainty, with an impact on a large part of the signal.
↓
High heat determination uncertainty

VOLUMIC MEASUREMENT

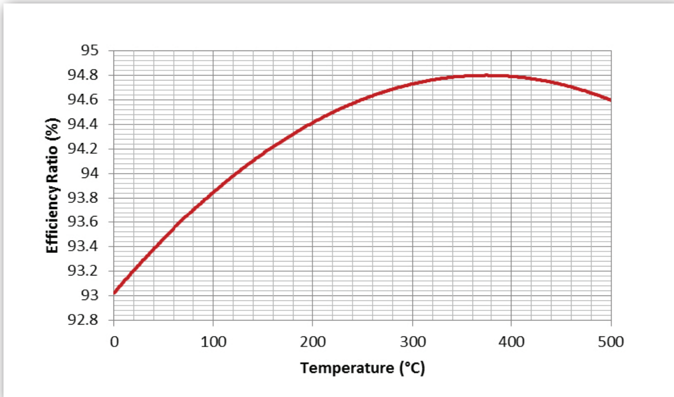

Figure 2b

Measured heat > **90%** of the total heat evolved by the sample + <**10%** from heat calibration.
Low calibration uncertainty, with an impact on a small part of the signal.
↓
Low heat determination uncertainty

Figure 3a

Figure 3 – Schematics of a Joule Effect cell with the red line representing the metallic wire of known electrical resistance (a) and calibration points and polynomial regression of a µSC 3D sensor calorimeter over its main usable temperature range (b).

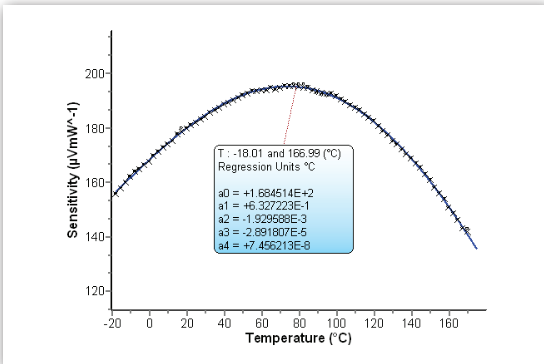

Figure 3b

1. Plate-shaped, 2D Sensors

There are two main concepts for heat flow sensors: plate-shaped (2D) or cylindrical (3D). **2D sensors** can operate under different principles (heat flux, power compensation) but in the majority of cases, the sample, contained in a crucible, is placed and centered on the sensor plate (the orange surface on Figure 1a - 2D Sensor) through which the heat flows. Only a part of the heat released or absorbed by the sample is transferred by thermal conduction to the sensors, and a significant part is dissipated through the walls and the cover of the crucible (red arrows on figure 1a).
Heat transfer modeling of such sample + crucible + sensor systems allows the efficiency ratio to be determined, i.e. the heat flux transferred to the sensor versus the total heat flux generated by the sample at any given temperature. Figure 2a shows that **the efficiency rapidly decreases with the temperature** and the thickness of the plate. The heat transfer and therefore the efficiency ratio are also affected by: the sample amount, the thermal conductivity of the crucible and of the gas used to flow through the calorimeter furnace. In this way, the sensor calibration allows the determination of heat flow signals despite these heat losses.

The calibration of a plate-type DSC is very critical and has to be run with experimental conditions that are similar to the final sample test conditions. Moreover, the **calibration procedure involves the measurement of the enthalpy of melting of certified standard materials**. Only a few substances are available because to be considered as a standard, a material needs to fulfill different conditions: to be easily available at very high purity, to be thermally and chemically stable, non-hygroscopic, not to be volatile, etc. It means that the calibration of 2D sensor based calorimeters can be accurately achieved only at a few temperatures (typically 3-5) corresponding to the melting temperatures of the standard materials used. Between two temperatures, the calibration is determined by polynomial regression, which introduces measurement uncertainty. **And the propagation of this uncertainty is favored by the fact that a large part of the signal is corrected by a biased calibration.**

2. Sample surrounding 3D sensors

The **3D type senso**r offers another technical option. The detection concept is based on a three dimensional heat flow meter sensor. The heat flow meter element consists of a ring of several thermocouples in series (Figure 1b). The corresponding thermopile of high thermal conductivity surrounds the experimental volume within the calorimetric block. The radial arrangement of the thermopiles guarantees an almost complete integration of the heat. This is verified by the calculation of the efficiency ratio that indicates that an average value of 94 ± 1% of heat is transferred through the sensor on the full range of temperature of the 3D sensor type DSC (Figure 2b). Another very important point is that except in a few extreme cases, the sensitivity of the DSC is no longer significantly affected by the type of crucible, the type of purge gas and flow rate.
Moreover, 3D sensors can be calibrated by a method which is an alternative to the certified reference materials method. It is called the **Joule Effect method** as it involves a special crucible or cell including a metallic wire of known electrical resistance R (See figure 3). A power module can inject an accurately controlled current intensity (I) in this metallic wire, leading to a dissipated thermal power equal to $P = R.I^2$. This operation allows for a calibration at almost any temperature. For practical reasons, it is applied under slow heating conditions and calibration points are equally distributed over the temperature range of the calibrated calorimeter. Figure 3b shows an example with a calibration point every 3°C. **The polynomial regression which is still used to determine the calibration of the sensor between two points is thus much more accurate because it is based on many more points.**
Finally, **this more accurate calibration helps to correct an intrinsically more accurate measurement, which leads to accuracy propagation!**