

Perfectly Shaped – New Crucibles Guarantee High Reproducibility

Claire Strasser

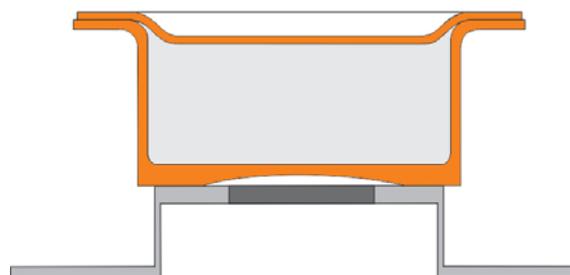


The qualities of a “good” DSC measurement with respect to sensitivity, resolution, baseline stability, etc., are usually attributed to DSC instrument performance and rarely to sample preparation and choice of crucible. Nevertheless, the latter two details also play a decisive role in the quality of the measurement. The choice of crucible, in particular, is key to achieving reproducible DSC measurement results.

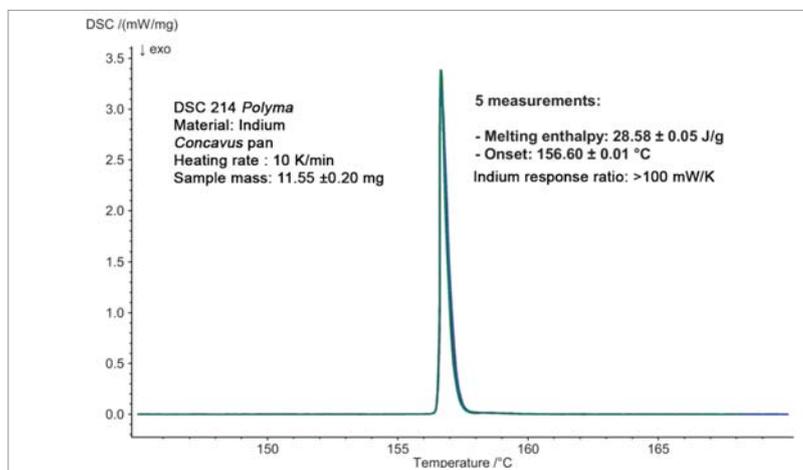
Shape Matters ... for Better Reproducibility

Until recently, it was assumed that DSC crucibles should feature an absolutely flat base in order to guarantee optimum contact between the sample and the sensor.

In reality, absolutely flat crucible bottoms are impossible to manufacture and are always subject to manufacturing tolerances; the crucible bottom can be slightly concave to the inside or convex to the outside so that no two crucibles from the same manufacturer are alike, let alone completely planar.



1 Schematic of the *Concavus* crucible on a flat sensor. For better illustration, the concavity of the crucible is exaggerated (in reality, it is only 10 to 20 μm).



2 Reproducibility of the measurement results on five different indium samples

A more important feature than a perfectly flat crucible bottom for reproducible measurements is reproducible contact between the crucible base and the sensor. NETZSCH has now achieved this goal with its new, patent-pending *Concavus* crucibles.

Concavus crucibles have slightly concave bottoms that make contact with the sensor along the outer ring of the crucible, whereas the inner side of the crucible is flat. Rather than maximizing contact area between the crucible and the sensor, this design maximizes the reproducibility of the contact area. Even if the crucible is not placed exactly in the middle of the sensor (see figure 1). As a result, DSC measurements with these crucibles exhibit excellent reproducibility.

Figure 2 shows DSC measurements on five different indium samples using *Concavus* crucibles. The evaluated onset temperatures and melting enthalpies differ by only 0.01°C (standard deviation of the onset temperatures) and 0.20% (relative standard deviation of the melting enthalpies), respectively.

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... for Better Pressure Stability

Another advantage of the *Concavus* crucible is its behavior under internal pressure. If gaseous products evolve from the sample during a DSC measurement, pressure is generated inside the crucible if it is not sealed with a pierced lid. If the crucible bottom is planar, the crucible floats as a result of the increased pressure considerably influencing the contact between the crucible and the sensor. If the crucible bottom, however – like that of the *Concavus* crucible – is concave, it is more pressure-resistant. Increasing the pressure inside the crucible initially leads to convexity of the lid and not of the bottom.

Safety due to Purity

Besides crucible choice, sample preparation also has an impact on the quality of the measurements. The sample should not be contaminated during its preparation in order to guarantee that all effects in the DSC curve result strictly from the sample itself. This means that crucibles must be clean and free of impurities. The *Concavus* crucibles are carefully cleaned and dried prior to their shipment.

Transport, Sampling and Archiving in One System: The 3in1-Box

Another advantage of the *Concavus* crucibles is their packaging. Normally, suppliers ship crucible in bulk in a single box. Crucibles packaged in this manner can suffer deformations and can stack on top each other, which makes them hard to separate. NETZSCH packages *Concavus* crucibles in 96 separate compartments in the 3in1 Box (figures 3 and 4), thereby minimizing deformations and allowing easy access to crucibles. Indexing of the compartments is also useful for sample archiving, and a sample identification booklet is included for this purpose.

Conclusion

Recognizing the importance of the crucible quality in DSC measurements, NETZSCH has developed a novel solution to guaranteeing measurement repeatability with the

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3 3in1 Box – Ideal for transportation of the *Concavus* crucibles and for archiving after the measurement



4 3in1 Box – individual compartments for 96 *Concavus* crucibles

Concavus crucible. The slightly concave geometry of crucible base guarantees consistent contact between the crucible and the sensor and reduces adverse effects of pressure build-up within the crucible during a measurement. The 3-in-1 Box is a new packaging concept that minimizes crucible damage during shipment, simplifies crucible selection and handling and facilitates the archiving of samples for future reference.