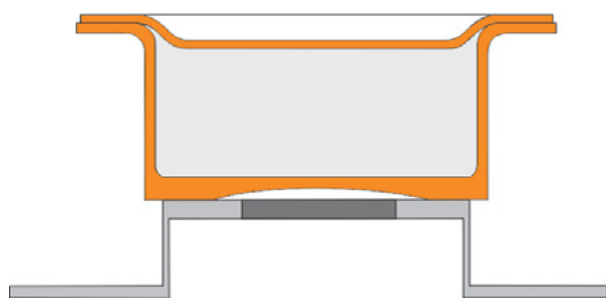




Concavus® Crucibles for DSC Measurements: Choosing the Best

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- 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).

Introduction

It is generally assumed that DSC measurements require a flat crucible bottom in order to ensure ideal sample-to-crucible-to-sensor contact. However, due to the manufacturing process, perfectly flat crucible bottoms do not exist: They are always slightly curved, either to the inside or to the outside. For this reason, standard aluminum crucibles are neither perfectly flat nor reproducible in shape, what may certainly influence the reproducibility of DSC measurements.

In contrast, the Concavus® crucibles are intentionally manufactured with a slightly concave bottom (figure 1).

This yields better reproducibility of the shape of the crucible bottom and thus, higher reproducibility of the DSC results.

In the following, measurements were carried out on samples prepared in Concavus® and in standard aluminum crucibles in order to compare the two types.

Test Conditions

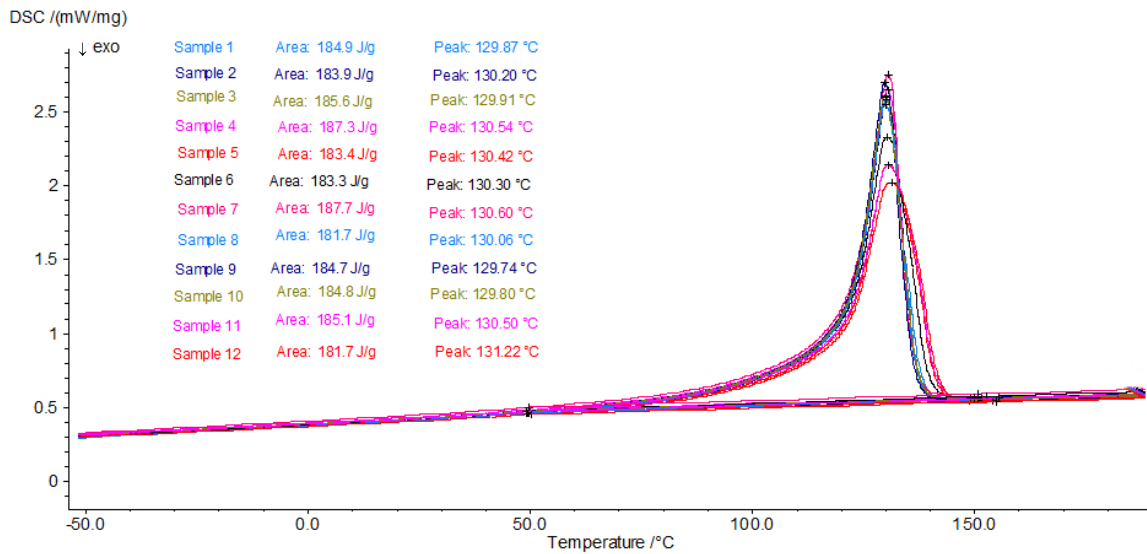
24 samples were prepared from the same HDPE tube. For this, round pieces with a diameter of 4 mm and a mass of 12.0 mg each were cut from the tube. Half of these prepared samples were placed in standard aluminum crucibles and the others were placed in Concavus® crucibles.

All 24 samples were measured with the DSC 214 Polyma. For the measurements, the samples were heated twice between -60°C and 190°C at a rate of 10 K/min. Between these two heating runs, they were cooled at 10 K/min. The 2nd heating of each measurement was analyzed by means of *AutoEvaluation* to guarantee the objectivity of the peak temperature and enthalpy results.

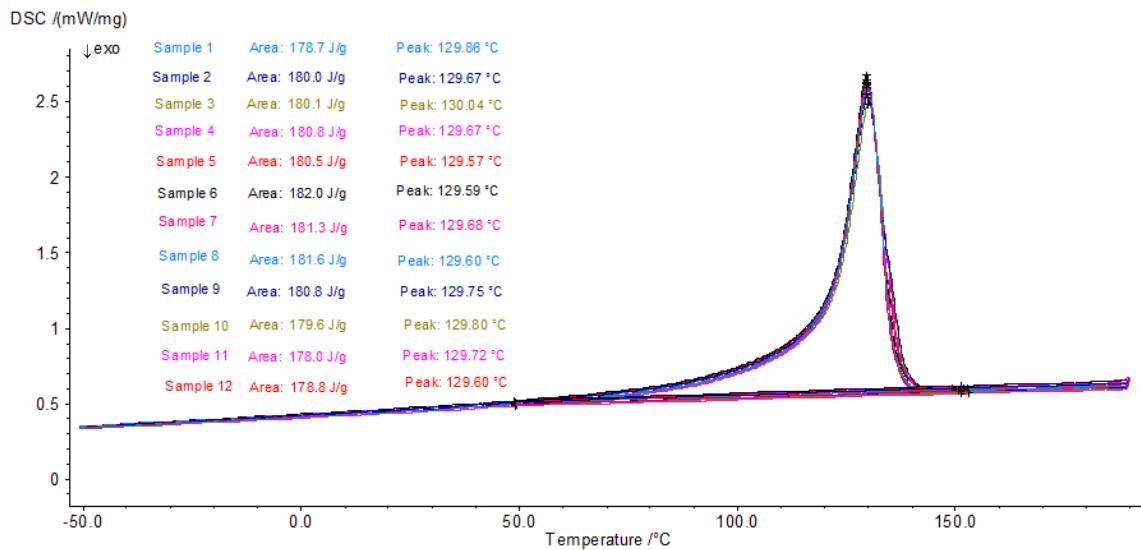
Test Results

The 2nd heatings of all measurements carried out in the standard aluminum crucibles are displayed in figure 2. The equivalent plot with the measurements in Concavus® pans is given in figure 3.

APPLICATIONNOTE *Concavus*[®] Crucibles for DSC Measurements: Choosing the Best



2 HDPE measurements in standard aluminum crucibles with pierced lid



3 HDPE Measurements in *Concavus*[®] crucibles with pierced lid

In all of the measurements, one peak was detected around 130°C, resulting from the melting of HDPE. The superiority of the *Concavus*[®] crucibles can be clearly seen here: All peaks of the measurements carried out with it are nearly identical in shape, whereas some outliers are

present for the measurements with standard aluminum crucibles (samples 6, 11, 12).

The peak temperatures and enthalpies of all these measurements are recapitulated in table 1.

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Tab. 1 Temperatures and enthalpies of the melting peak

Measurement	<i>Concavus</i> [®] Crucible		Standard Aluminum Crucible	
	Temperature [°C]	Enthalpy [J/g]	Temperature [°C]	Enthalpy [J/g]
1	129.86	178.74	129.87	184.95
2	129.67	179.97	130.20	183.88
3	130.04	180.06	129.91	185.62
4	129.67	180.81	130.54	187.35
5	129.57	180.54	130.42	183.39
6	129.59	182.00	130.30	183.32
7	129.68	181.27	130.60	187.72
8	129.60	181.62	130.06	181.67
9	129.75	180.75	129.74	184.72
10	129.80	179.61	129.80	184.81
11	129.72	177.96	130.50	185.11
12	129.60	178.84	131.22	181.74
Average	129.71 ±0.13 ¹	180.18 ±1.18 ¹	130.26 ±0.41 ¹	184.52 ±1.80 ¹
Relative standard deviation	0.10	0.65	0.31	0.98

¹Uncertainty calculated with the standard deviation

Conclusion

According to the relative standard deviation of peak enthalpy and temperature, the *Concavus*[®] pans are 34% better in enthalpy reproducibility and 68% better in

peak temperature reproducibility than standard aluminum crucibles.

This demonstrates the superiority of the *Concavus*[®] pans for achieving highly repeatable DSC measurements.