# Baseline Correction of DSC Signals with *BeFlat+*

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## Introduction

If just an empty reference crucible and an empty sample crucible with the same mass were measured in a DSC device with perfectly symmetrical sensor and surrounding furnace chamber, zero DSC signal would be expected. The so-called baseline as a function of temperature would be a flat line at a value of zero. In reality, a DSC device is not perfectly symmetric and the crucible masses may be slightly different resulting in a temperature-dependent baseline different from zero. For many DSC applications, this is not disturbing because the DSC signal originating from the sample is by far higher than the baseline. For small sample signals, however, a baseline correction is recommended. For evaluation of the specific heat capacity of a sample from DSC signals, baseline correction is obligatory [1-3].

Subtraction of the DSC signal measured with empty reference and sample crucibles is in most cases the ideal baseline correction for DSC measurements when always carried out with the same temperature program and the same crucibles.

A more flexible alternative is a DSC baseline correction applying the NETZSCH *BeFlat+* software solution which is available as of *Proteus*® version 9.0. A baseline with two empty crucibles including two heating and two cooling rates needs to be measured just once leading to the *BeFlat+* baseline calibration according to:

$$DSC_{baseline}(T,\beta) = Offset(T) + \beta \cdot \left[K_{sensor}(T) + c_p^{crucible}(T) \cdot \left(m_{sample}^{crucible} - m_{reference}^{crucible}\right) \cdot Sens.(T)\right]$$

where $\beta$ is the heating rate, $c_p^{crucible}$ is the specific heat capacity of the crucible material, $m_{sample}^{crucible}$ and $m_{reference}^{crucible}$ are the masses of the sample and reference crucibles and *Sens.(T)* is the temperature-dependent calorimetric sensitivity of the DSC sensor. Since the calculated *BeFlat+* baseline takes the actual heating rate and crucible masses into account, it can be applied to DSC measurements with various temperature programs (with covered temperature range) and with different crucibles of the same type.

The *BeFlat+* baseline according to the equation above has a heating rate-independent part *Offset(T)*, which is due to any asymmetry of thermal resistances between the sample and reference side and due to finite asymmetry of the temperature profile in the DSC furnace chamber. The heating rate-dependent part is due to any asymmetric heat capacity of the DSC sensor reflected by $K_{sensor}(T)$ and due to different masses of the sample and reference crucibles.





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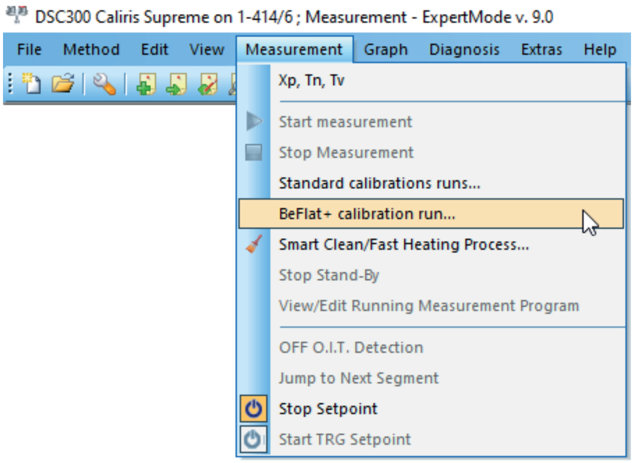
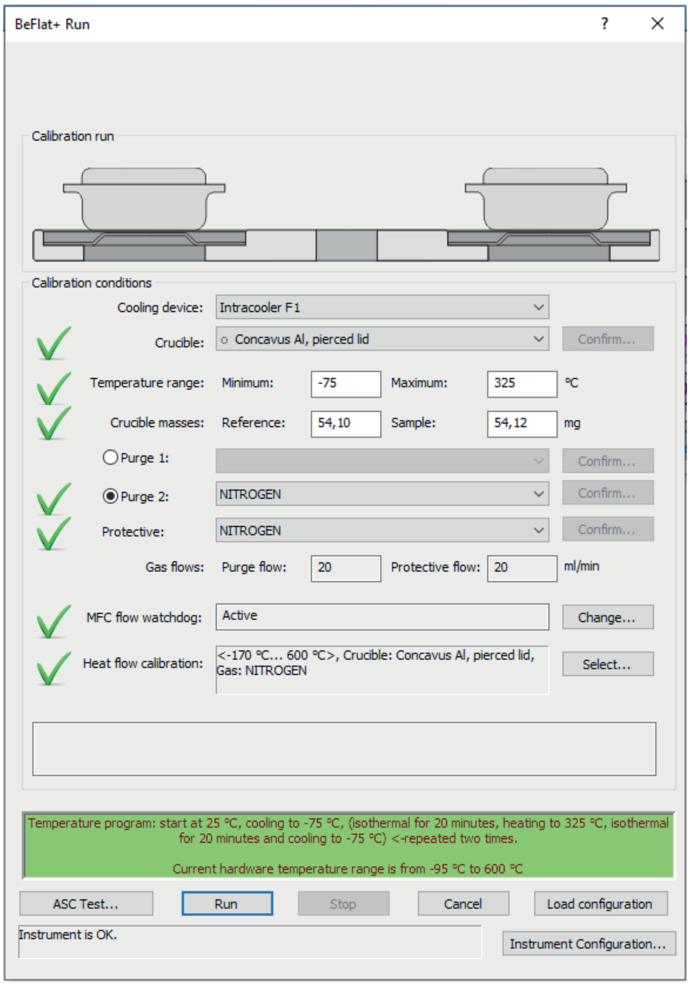
## *BeFlat+* Calibration Procedure

The *BeFlat+* calibration procedure can be accessed from the *Proteus*® measurement software (see figure 1). Before the calibration measurement can be started, the following items have to be selected or filled in as shown in figure 2: the cooling device and crucible type used, the temperature range, the crucible masses, the purge gases, the MFC flow watchdog and the DSC sensitivity calibration.




**1** *BeFlat+* calibration run, accessible in the *Proteus*® measurement software (*ExpertMode*).




**2** *BeFlat+* Calibration dialog for definition





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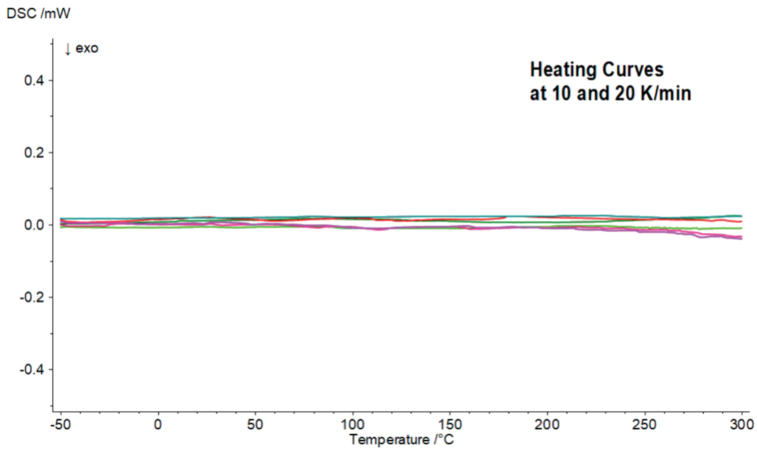
As mentioned before, the *BeFlat+* calibration contains two heating segments at heating rates of 10 K/min and 20 K/min and two cooling segments at -5 K/min and -10 K/min. In between these dynamic segments, there are isothermal segments for stabilization of the DSC signal. It should be noted that the so-called *AutoCooling* is used for *BeFlat+* calibration. This means that the cooling devices are automatically used in an appropriate manner so that the temperature program is accurately processed.

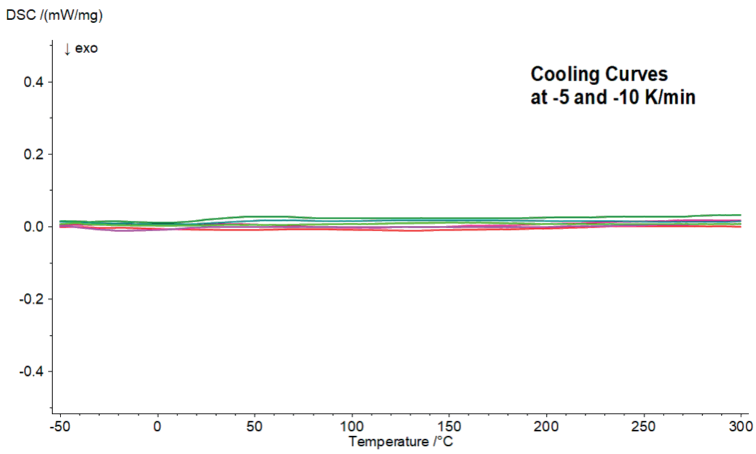
After the *BeFlat+* calibration has finished, the DSC curve has to be visually verified and the *BeFat+* calibration has to be stored (see figure 3).

Figures 4 and 5 display typical DSC baselines that are *BeFlat+* corrected. These measurements are repeatability tests with two empty *Concavus* crucibles with pierced lids, placed in a DSC 300 *Caliris*® equipped with H-Module and intracooler cooling. Heating rates of 10 und 20 K/min and cooling rates of -5 and -10 K/min used are the same as in the *BeFlat+* calibration which means that these measurements can be regarded as calibration checks. The *BeFlat+* baselines are flat and exhibit a typical repeatability of ± 20 µW.


**3** *BeFlat+* calibration dialog for confirmation




**4** *BeFlat+* corrected DSC baselines as a function of temperature. Shown are three heating curves, measured at 10 K/min (green) and three heating cuves, measured at 20 K/min (red, pink).




**5** *BeFlat+* corrected DSC baselines as a function of temperature. Shown are three cooling curves at -5 K/min (green) and three cooling curves at -10 K/min (red/pink).





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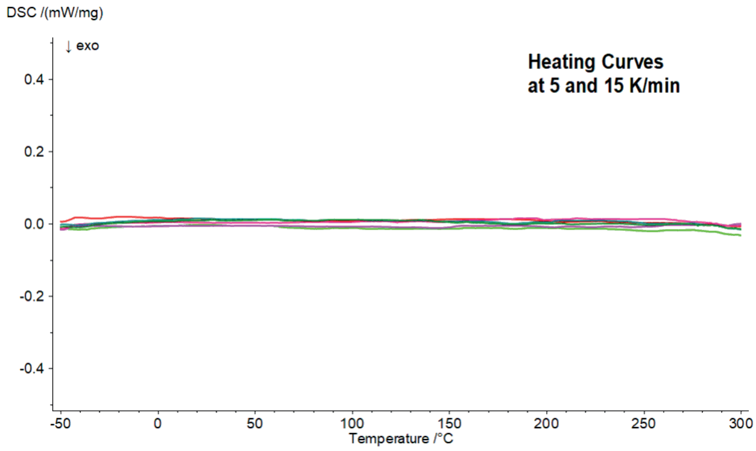
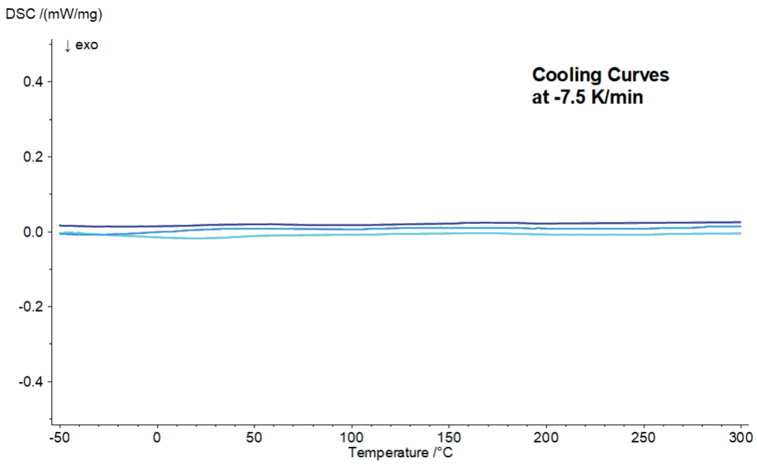
Further *BeFlat+* corrected DSC baselines were measured at heating rates of 5 und 15 K/min and a cooling rate of -7.5 K/min. These heating and cooling rates were not used during the calibration. Nevertheless, the *BeFlat+* baselines are also flat and they are within about ± 20 µW, as it can be seen in figures 6 and 7. This demonstrates on the one hand the correctness of the model equation on page 1 for the heating rate-dependent DSC baseline, and also very good repeatability on the other.




**6** *BeFlat+* corrected DSC baselines as a function of temperature. Shown are three heating curves, measured at 5 K/min (green) and three heating cuves, measured at 15 K/min (red, pink).




**7** *BeFlat+* corrected DSC baselines as a function of temperature. Shown are three cooling curves, measured at -7.5 K/min (blue, cyan).

## Summary

The NETZSCH *BeFlat+* baseline correction for DSC measurements is a flexible and efficient solution, because it can be used for various temperature programs covered by the temperature range of *BeFlat+* calibration and for different crucibles of the same type.

## Literature

[1] DIN 51007:2019-04: Thermische Analyse (TA) – Differenz-Thermoanalyse (DTA) und Dynamische Differenz-kalorimetrie (DSC) – Allgemeine Grundlagen.
[2] ASTM E1269-11: Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry.
[3] DIN EN ISO 11357-4:2021-05: Plastics – Differential scanning calorimetry (DSC) – Part 4: Determination of specific heat capacity.





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