Determination of the specific heat capacity of melts using a tabletop DSC

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The specific heat capacity of solids can be easily determined by DSC following a standardised procedure.[1] First, the instrument is calibrated for heat flow detection by recording the background-corrected heat flow signal resulting from applying a constant temperature ramp to a reference sample of well-known heat capacity. Second, the heat-flow calibration is used to determine the specific heat capacity of a sample from the background-corrected heat-flow curve derived by applying the same temperature ramp on a test specimen of known mass.

Differences in the thermophysical and geometrical properties of sample and reference sample, such as thermal conductivity, thermal contact resistivity and sample geometry, affect the response of the DSC instrument. To overcome these issues, isotherms are required before and after the heating ramp that allow for mathematical corrections of small interferences.

Melting of samples results in a displacement of the sample in the crucible including changes in sample geometry as well as thermal contact resistivities. These changes often result in apparent measurement artifacts which cannot be corrected by standardised methods.

A method is presented that allows for the correction of measurement artifacts due to sample melting which meets the following targets:

- The approach must result in reproducible results.
- The approach must not require further information about sample properties.
- The experimental effort should not exceed the effort for the standard procedure.
- Incorporation into commercial DSC software should be easily possible.

[1] DIN 51007:2019-04, Thermal analysis - Differential thermal analysis (DTA) and differential scanning calorimetry (DSC) - General Principles