

DSC signal optimization with BeFlat[®] and ISSP[®] in Pharmaceutical applications

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Differential Scanning Calorimeter measuring systems show time and temperature dependent signal changes because of the differences in the heat capacities of the sample and reference side and also because of the temperature and heating rate depending differences in the heat transfer conditions inside a DSC cell [1]. Tolerances in the production of DSC cells, sensors and crucibles and limitations in the electronic control are of influence especially when using cooling systems or not symmetrical measuring programs like the c_p determination according to the ratio method.

With the new calculation method BeFlat[®] ("baseline flattening" method) a recipe is introduced to eliminate the instrumental influences on a baseline and to achieve a linear, horizontal baseline with minimal deviation at the heat flow axis for any temperature and heating rate. This allows a much better and accurate evaluation of the mere sample effects from a DSC measurement. The raw data of the measurement are not changed when applying BeFlat[®].

Different approaches for modeling of the heat transfer process in a DSC allow a calculation and correction of the asymmetry at a given instrument [2]. Also design optimizations for heating and cooling systems additionally improve the baselines and reduce the signal noise. Further improvement of the signal to noise ratio, generally by a factor 3-4, is achieved applying ISSP[®] (integrated sensor signal processing). This newly developed mathematical treatment of the raw data allows a better evaluation of small, near to noise peaks, and improves the resolution in DSC measurements.

The new heat flux DSC concept including BeFlat[®] and ISSP[®] will be introduced with its high flexibility through replaceable sensors, effective cooling to -85 °C by an intracooler, or -180 °C by liquid nitrogen with an automatic supply system. For routine applications in research and quality control, the DSC can be equipped with an automatic sample changer for 64 crucibles, which fits in the same configuration also to the new TG 209 F1 Iris[®], because of a consequent platform concept for DSC and TG.

Calibrations with known standard samples and applications on drugs and excipients will be evaluated to demonstrate the improvements in the DSC signal.

References

- [1] G.W.H. Höhne, W. F. Hemminger, and H.-J. Flammersheim, Differential Scanning Calorimetry, 2nd ed., Springer, Berlin, 2003, p. 31 ff
- [2] U.S. Patent No. 6431747; 6488406; 6523998