

High Precision Specific Heat Determination at High Temperature: Introduction of a New 3D DSC Detector

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There is an increasing demand for measuring specific heat at high temperatures with a good precision, especially in the fields of oxide and engineering ceramics. The standard DSC measurement of specific heat using the continuous heating mode is based on the following DSC signal:

$$A_{(\mu V)} = S_{(\mu V/mW)} \times m_{(g)} \times Cp_{(mJ/g/K)} \times V_{(K/sec)}$$

In order to improve the precision of the Cp measurement, it is recommended to enlarge the DSC amplitude. This can be achieved, as described in the relation, by improving the sensitivity of the DSC detector, by increasing the amount m of sample to be investigated, and by increasing the scanning rate V . With most of the DSC detectors working at high temperature, there are severe limitations in the detector sensitivity and the volume available for the sample mass. In order to solve these limitations, Setaram has developed a new patented 3D high precision Cp DSC rod to be used on the new Labsys_{evo} analyzer. A top down configuration is used to enlarge the volume available for an increased mass of sample. The top level receives the sample to be investigated and the bottom level is used for the reference volume. Using such a configuration, a 3D detector (that means a detector surrounding the full volume designed for the sample) is designed based on 10 different thermocouples mounted in series and opposition. With such a design, a maximum volume of 380 μ l is available for the determination of the Cp of the sample. Such a construction has also improved in a large scale the sensitivity S of the DSC detector. In order to get faster heating rates, a new advanced furnace is used with the Labsys_{evo} analyser that allows one to reach 100 °C/min up to 1600 °C. The new furnace is also characterized by a large homogeneous zone of temperature to guarantee the high precision Cp determination with the new 3D DSC detector.