TECHNICAL NOTE

Differential Scanning Calorimetry

New Design Improvements of the DSC 8000/8500 Double-Furnace Differential Scanning Calorimeter

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Calorimeter background

A calorimeter is a device that measures the heat exchange of a sample with its environment. Since heat is usually generated or consumed during a physical transition or chemical reaction, calorimetry is a universal tool to study such processes. Modern Differential Scanning Calorimetry (DSC) has been used widely for material characterization purposes in various industries, especially polymer and pharmaceutical. The applications of DSC include quality control of raw material or final products, phase transition studies, kinetics analysis, polymorphism investigation and amorphous and crystalline content determination.

Generally speaking, there are two basic types of DSC: heat-flux DSC and power controlled DSC. Although both are based on the differential method of measurement, which measures the differential signal between sample side and reference side, they are quite different in their design and measuring principle.

Single-Furnace or Heat-Flux DSC

The modern heat-flux DSC has evolved from Differential Thermal Analysis (DTA), which measures the temperature difference that develops between the sample and the reference material as both are heated together in a single furnace. There are several different designs of heat-flux DSC including disk-type design, turret-type design and cylinder-type design. Each has its own advantages and limitations. Because of its simplicity the disk-type is probably the most used design. In this design, the heat exchange between furnace and sample takes

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place through a disk, sometimes with raised pedestals, which also serve to support the sample and reference crucible. The temperature sensors, normally thermocouples, are attached under or integrated in the disk. To minimize temperature gradients it usually utilizes a single, heavy furnace which acts also as a heat sink to buffer heat from the sample. As in DTA, the temperature difference between sample and reference materials is measured as a voltage. This voltage is processed by the manufacturer's software to obtain heat flow rate based on a calibration constant that takes into account the thermocouple, cell and sample pan characteristics.

Hardware-wise, the disk-type heat-flux DSC is a simple and robust design, but its heating and cooling rate are limited by the mass of the furnace. Furthermore, achieving accurate energy measurement is difficult because of the limitations of the DTA measuring principle.

Double-Furnace or Power Controlled DSC

Power controlled DSC was invented by PerkinElmer in the early 1960s. Since then, PerkinElmer has pioneered the power controlled DSC technology for more than 40 years. It utilizes a different design and operating principle than heat-flux DSC. The most prominent difference is that power controlled DSC has two independent identical small furnaces, one for sample material and one for reference material. Each is made of platinum/iridium alloy for best chemical resistance. Each has its own heater and temperature sensor. A platinum resistance thermometer is used as a temperature sensor for more linear, stable and accurate temperature measurement.

The furnaces are surrounded by an aluminum block, an isothermal heat sink, which is maintained at a cold temperature by a cooling accessory. Due to the low thermal mass of the DSC furnaces, power controlled DSC has a smaller time constant with respect to temperature control and more rapid response toward sample reaction. It makes heating and cooling rates up to 500 ˚C/min possible.

Besides this hardware difference, the control scheme is also very different from that of heat-flux DSC. The furnaces are controlled by two control loops: the average temperature control loop and the differential temperature control loop. During temperature scanning, the average temperature control loop delivers equal power to sample and reference sides to ensure that the average temperature tracks the program temperature. The differential control loop is designed to minimize the temperature difference between the sample and reference positions. If there is a transition, the differential control loop will supply differential power to control the energy of the sample reaction such that the immediate enveloping environment of the sample is maintained at the program temperature. This underlies the name "power controlled". So the result is that both sample and reference temperature will follow the program temperature closely and the energy to the sample material is recorded directly. It gives the benefit of direct and accurate energy measurement. Because of these unique features of power controlled DSC, it has become an indispensable analytical tool of thermal analysis. Especially now with the increasing use of fast scan DSC technology, many new applications have been developed which were previously not possible.

DSC 8000/8500 Design

The new DSC 8000 and DSC 8500 have many improvements in hardware and software. As mentioned above, one of the advantages of power controlled DSC is its small furnace size. In the DSC 8000/8500, new manufacturing technology has been employed to further reduce the mass of the furnace by 25%. This allows quicker response and faster scanning rates. The maximum controlled heating and cooling rate in the DSC 8500 is 750 ˚C/min.

One new feature called ballistic cooling is also available to better mimic polymer processes. In this mode, the power to the furnace is cut completely and the furnace temperature drops at the maximum allowed rate. Test results show that the instantaneous cooling rate can reach over 2100 ˚C/min. The data rate has also increased from the previous 20 points/second to a maximum 100 points/second to accurately record data at fast scanning rates.

The aluminum block around the furnace and the shroud outside of the block are also redesigned. The distance between the sample furnace and reference furnace is increased for more even temperature distribution within the block. The new super shroud assembly better isolates the measurement system from the surrounding and offers superior ambient rejection. This leads to better reproducibility. Together with the new purge gas design, frost formation is minimized.

Figure 1. DSC 8000/8500 with vacuum wand.

Mass flow control is used to control the purge gas rate. It enables easy control of purge gas flow rate and gas switching from the Pyris™ software. Even in the case of sudden power loss where the mass flow controller stops working, the specially designed purge gas bypass system will continue to purge the furnace and block area to prevent frost build-up when the furnace and the block are still cold.

Sample handling becomes easy with new motorized rotating and environmental covers. LED lights on the environmental cover turn on when the cover slides open and help illuminate the furnace areas. A vacuum wand facilitates sample loading and unloading.

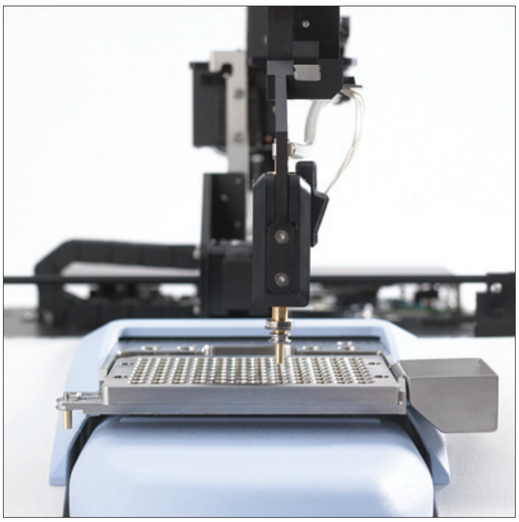

Figure 2. DSC 8000/8500 with autosampler.

The autosampler has been completely redesigned. It features a 96 position sample or reference pan holder and handles multiple pan types. It is field installable so there is no need to return your instrument to the factory in order to add an autosampler at a later time. It delivers reliable performance and increased productivity and is optimal for fast-scan DSC experiments.

From the electronic point of view, the new calorimetry board is designed to be less sensitive to ambient temperature and provides lower noise baselines. It also has more processing power to run advanced algorithms available on DSC 8000/8500.

Four cooling accessories are available to cover the full temperature range from -180 ˚C to 750 ˚C. They include a chilled water circulator, Intracooler II, Intracooler III and CLN2. The Intracooler III and CLN2 are new to the DSC 8000/8500. The Intracooler III extends the low temperature limit to -100 ˚C with the same 750 ˚C high temperature limit to support more applications. N2 can be used as the purge gas. The new CLN2 features "dial-a-temperature" operation which allows the operator to set the block temperature from -190 ˚C to +35 ˚C. Depending on the block temp, helium purge may not be necessary, which helps to reduce operating cost. Unlike previous double-furnace models, the new DSC 8000/8500 has one configuration that accommodates all four cooling accessories making it easier to change a cooling accessory in the laboratory if necessary based on your applications.

On the software side, the new SmartScan™ algorithm takes into account the imbalance in the measuring system and corrects it to give a flat baseline. The denoising algorithm facilitates reduction of random noise without degrading peak resolution.

Summary

The new DSC 8000/8500 inherits the superior power controlled design. It has incorporated numerous new features. It pushes performance to a new level and at the same time makes the operation more convenient.

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