Thermal Analysis

# DSC 8000 Performance Check


When one can't afford to miss something in your DSC analyses, you need the PerkinElmer 8000 DSC (Differential Scanning Calorimeter). Missing important information can make a big difference to your organization's success through:

- Product failures
- Additional manufacturing expenses
- Lower productivity
- Inferior Product

With the PerkinElmer-exclusive double-furnace DSC, one can be confident in achieving fast, accurate and reproducible results. PerkinElmer, the leader in high sensitivity thermal analysis instrumentation, pioneered the double-furnace DSC in 1963 over 54 years ago. Double-furnace DSC provides better resolution and higher sensitivity in comparison to conventional single-furnace DSC systems currently available in the market.

## The Double-Furnace DSC Principle

With the double-furnace DSC, the sample and the reference materials are placed in two independent but nearly identical furnaces. When an exothermic (energy released) or endothermic (energy absorbed) event occurs in the sample, power is either applied or removed from the sample calorimeter to compensate for the change in energy and maintain a "thermal null" state. The amount of power required to maintain system equilibrium is directly proportional to the energy changes occurring in the sample. No complex heat-flux equations (used in single-furnace DSC) are necessary with a double-furnace DSC because the system directly measures energy flow to and from the sample.


## Abstract

The present technical studies carried out to demonstrate PerkinElmer DSC 8000 performance in terms of baselines. It was observed that few scientists want to know finer aspects of baseline performance at various temperatures and condition to evaluate low transitions as well as low enthalpy measurements. In the present note, we have measured few important specifications such as baseline deviation, baseline reproducibility, and noise test.


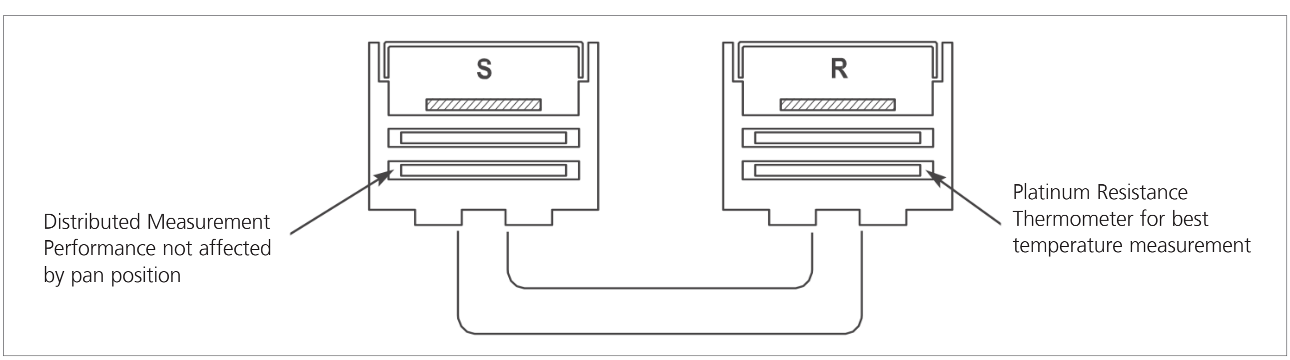


*Figure 1.* Double Furnace DSC design

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## Introduction

The Differential Scanning Calorimetry (DSC) measures the amount of energy (heat) absorbed or released by a sample as it is heated, cooled or held at a constant temperature. DSC is used to determine important material properties including melting points, crystallization temperatures, glass transition temperatures and phase transition temperatures. This technique is extensively used in the pharmaceuticals, food, polymer and specialty chemicals industries.

Use of double-furnace DSC offers many advantages over single-furnace DSC such as, direct measurement of heat flow (without any mathematical treatment), higher resolution and greater sensitivity, much faster heating and cooling due to low mass furnace design, and higher calorimetric precision and accuracy.

The present work demonstrates performance of PerkinElmer's DSC 8000 by measuring baseline deviation, baseline repeatability, and noise test.

It should be noted that in any DSC, sensitivity is directly proportional to heating rate, although resolution between adjacent events is inversely proportional to heating rate. This means that one way of increasing sensitivity is to increase heating rates, and avoids the issues of temperature gradients within the sample if smaller masses are used.

## Results and Discussion:

The following conditions were ensured before proceeding with the case studies:

- The DSC 8000 was calibrated with Indium and Zinc for temperature, heat flow, furnace and smart scan parameters.
- The furnace was cleaned thoroughly at 700 °C in open air.
- Highest purity 99.95% nitrogen purge gas was used.
- Standard platinum vented covers were inspected to ensure they were in satisfactory condition.
- Intracooler was connected to get the desired lower temperature before starting the run.

The following performance tests were carried out:

1. **Baseline deviation**

   For baseline deviation, following DSC conditions were used:

   I. Temperature range: -10 °C to 180 °C
   II. Heating rate: 10 °C/min
   III. Sample purge flow, $N_2$: 30 ml/min,
   IV. The baseline run with empty furnace and sample holder covers in place

Figure 2 shows the calculation of ΔY between minimum and maximum points being selected on the Y axis for complete temperature range and then an average taken.

Test Result Baseline Deviation as follows:

| Parameter | Test Result |
|---|---|
| Baseline Deviation | ≤ 15 μW |

The following performance tests were carried out:

2. **Reproducibility**

   For baseline reproducibility, following measurement conditions were used:

   I. Temperature range: -10 °C to 180 °C
   II. Heating rate: 10 °C/min
   III. Sample purge flow, $N_2$: 30 ml/min
   IV. The baseline run with empty furnace and sample holder covers in place

Three separate baseline runs were carried out using the parameters above.

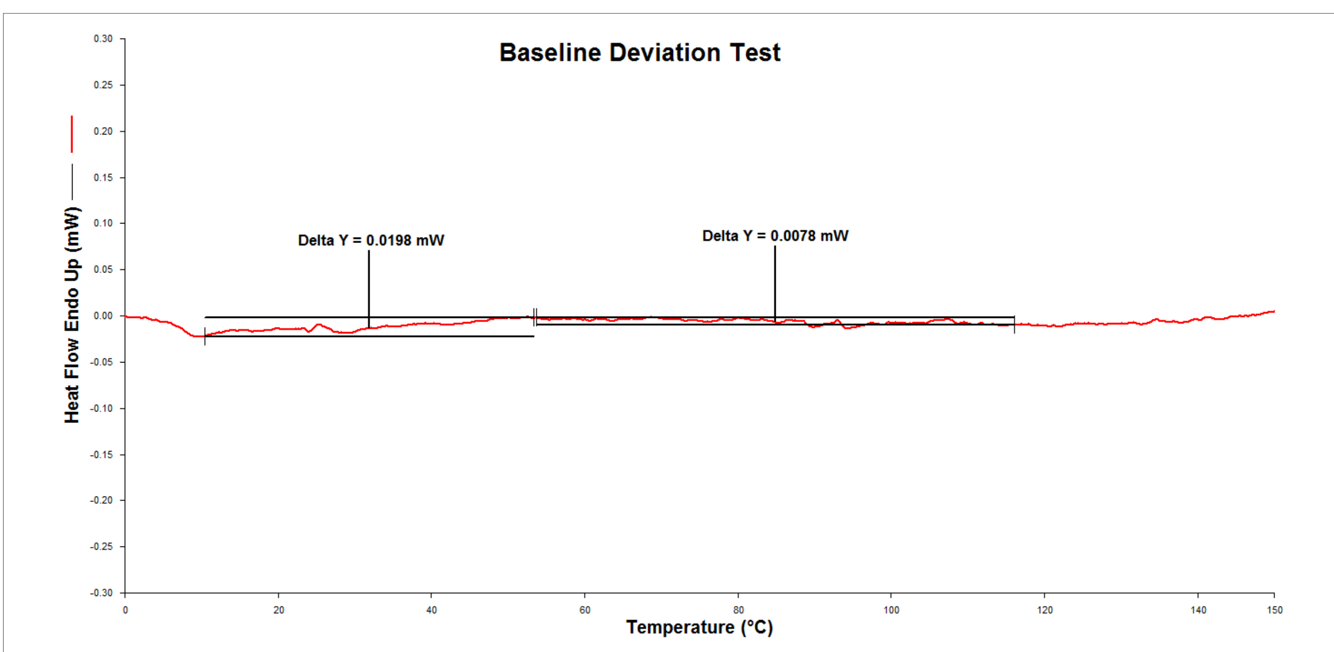


*Figure 2.* Baseline Deviation Graph calculation from 0 to 150 °C.

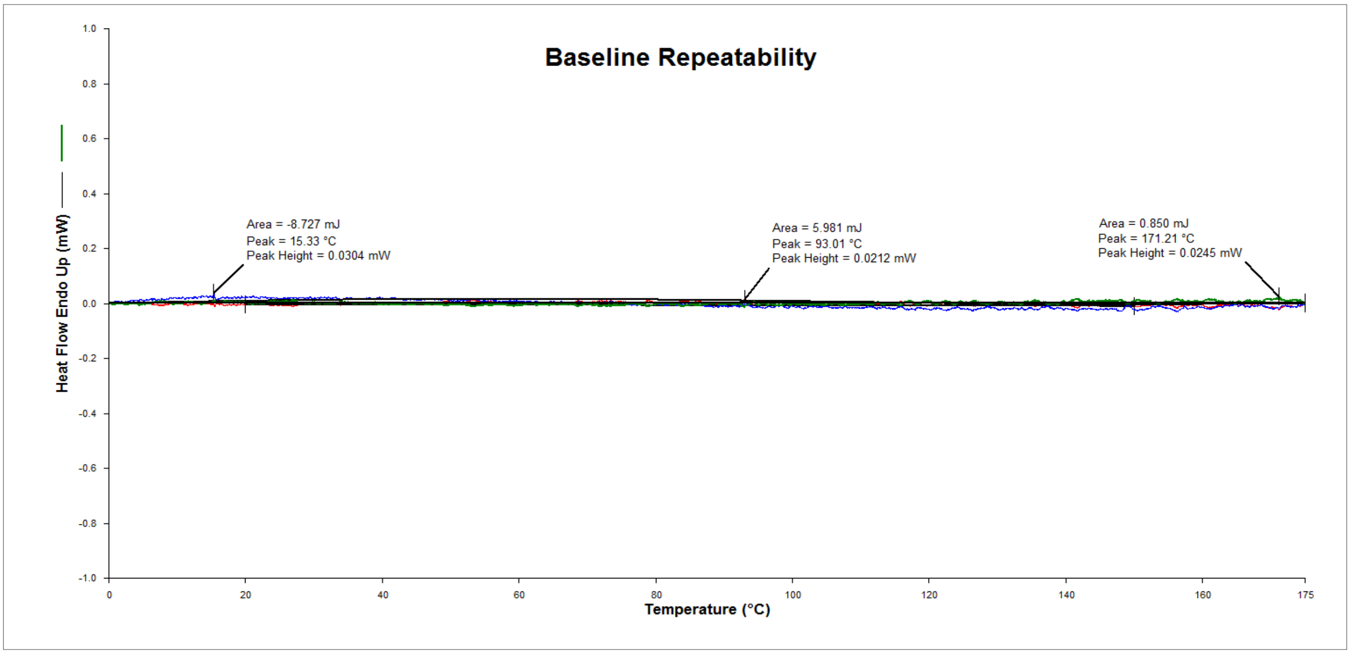


*Figure 3.* Baseline repeatability thermogram.

Calculation of baseline repeatability parameters:

- Recall the three individual baselines in temperature axis and align all at 0 °C by using the "shift curve" software feature.
- Calculate peak height between 0 °C and 175 °C points on the temperature scale.
- Calculate the difference between the maximum and minimum peak height values.

Test Result:

| Parameter | Test Result |
|---|---|
| Baseline Repeatability | ≤ 10 μW |

The following performance tests were carried out:

**3. Noise test**

For noise test, following parameters conditions were used:

I. Hold for 10 min at 100 °C

II. Sample purge, $N_2$ = 20 ml/min

III. The baseline run with empty furnace and sample holder covers in place

Calculation of noise test parameters:

- Calculate the ΔY for short time segment approximately for one second at random points 1, 2 and 3.

Test Result:

| Parameter | Test Result |
|---|---|
| Noise | ≤ 0.1 μW (Average) |

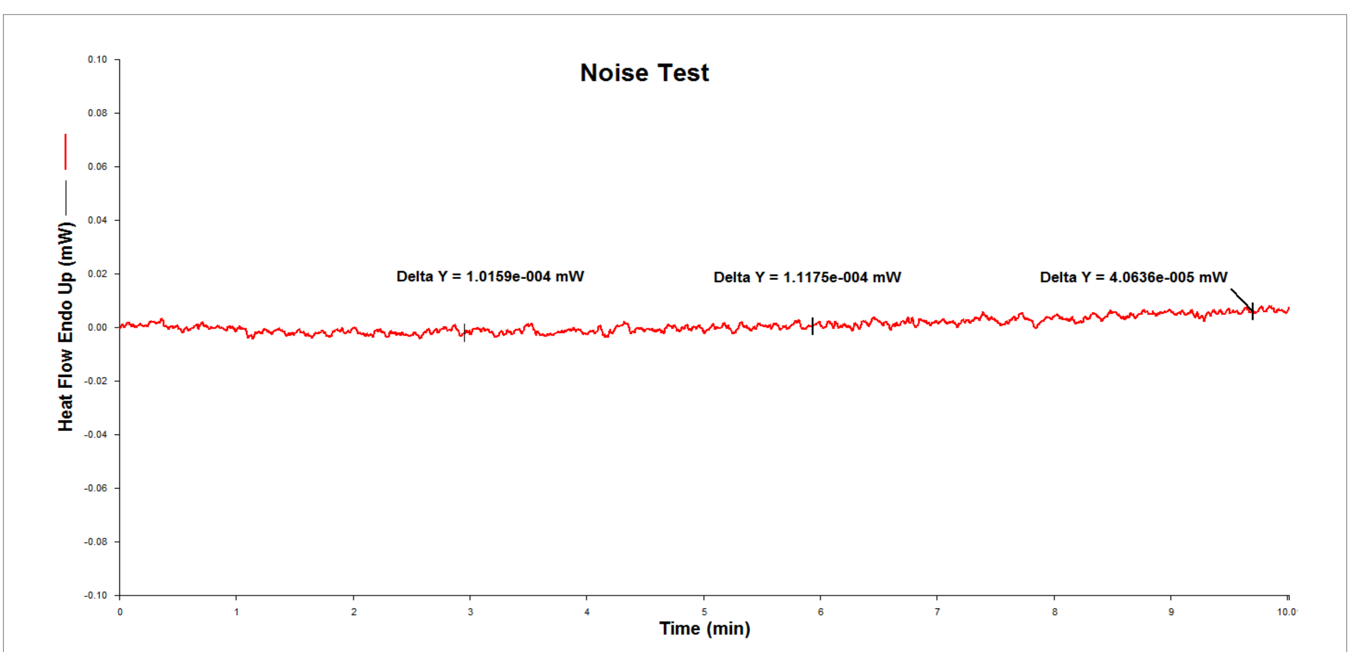


*Figure 4.* Noise test thermogram.

## Summary of the Performance Test Results

*Table 1.* Need info

| Sr No. | Performance Test | Test Result |
|---|---|---|
| 1 | Baseline deviation | ≤ 15 µW |
| 2 | Baseline repeatability | ≤ 10 µW |
| 3 | Noise | ≤ 0.1 µW (Average) |

## Conclusion

The test results carried out on DSC 8000 showed excellent performance with respect to various baseline parameters like reproducibility, deviation and noise. These important parameters will enable various applications to be addressed with better accuracy and precision.

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