

## **Reproducibility of the Shimadzu DSC-50**

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### **Introduction**

The Shimadzu DSC-50 is a heat flux differential scanning calorimeter (DSC). In the DSC-50 the sample and reference are heated and/or cooled at the same gradient rate in the same furnace with a controlled atmosphere. As the sample is heated or cooled, it will go through phase transitions ( $T_g$ , melt, cure, crystallization, etc.). During these transitions, the sample will either absorb or radiate heat. This heat is characterized by a temperature change in the sample which is detected by a thermocouple and compared with the relative temperature of the reference cell ( $T_s - T_r = \Delta T$ ). According to standard heat-flux theory,  $\Delta T$  is proportional to changes in the calorimetric state of the sample and the resultant signal is reported in mW. Thus, the endothermic and/or exothermic reactions of the sample can then be measured and reported.

### **Hypothetical**

To ensure that the results being reported by the DSC are reliable, an instrument must meet certain criteria. Accuracy, precision, and the ability to repeat analyses are important to any researcher doing experiments using thermal instrumentation. The DSC-50, when properly calibrated, brings to the researcher precision (1%), accuracy ( $\pm 0.3^\circ\text{C}$ ) and repeatability that can be relied on to produce results of the highest caliber.

# Application Note

## Experimental

A typical DSC-50 was used in this experiment to check the precision, accuracy, and repeatability of the Shimadzu DSC. The DSC-50 furnace was reduced by us a 10% H<sub>2</sub>/90% N<sub>2</sub> gas mixture flowing at 50 ml/min at 700°C for two (2) hours. The DSC-50 was then calibrated according to published Shimadzu criteria<sup>1</sup>. Upon calibration an Indium (In) powder standard (99.999% pure) was run ten (10) times in the DSC. The heating program employed was a gradient heating rate of 10°C/min from ambient to 180°C. A N<sub>2</sub> atmosphere flowing at 50 ml/min was maintained during all ten (10) experimental runs. In has a recorded<sup>2</sup> melt peak temperature of 156.6°C, and onset temperature of 155.0°C, and an enthalpy of 28.5 J/g. Also measured during this experiment were Peak Height (mW/mg) and enthalpy in J/mol. The chart below details our findings:

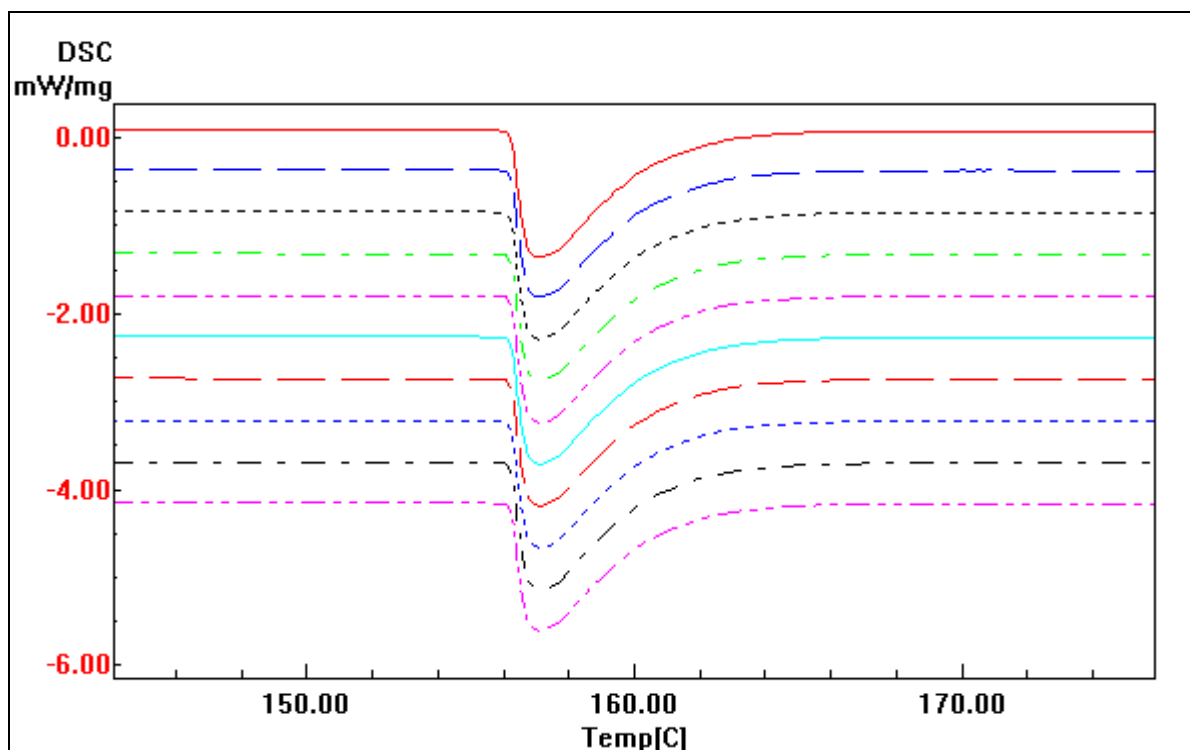
<b>Run</b>	<b>Peak</b>	<b>Onset</b>	<b>Peak Height</b>	<b>Enthalpy</b>	<b>Enthalpy</b>
<b>Units</b>	<b>°C</b>	<b>°C</b>	<b>mW/mg</b>	<b>J/g</b>	<b>J/mol</b>
Run 1	156.63	155.00	1.492	28.85	3313.05
Run 2	156.69	154.84	1.499	28.36	3255.99
Run 3	156.53	155.10	1.496	28.96	3325.03
Run 4	156.50	154.94	1.494	28.85	3312.84
Run 5	156.52	155.05	1.497	28.86	3313.43
Run 6	156.56	155.03	1.498	28.79	3305.37
Run 7	156.49	155.13	1.493	28.82	3309.62
Run 8	156.67	155.08	1.493	28.68	3292.47
Run 9	156.68	155.01	1.494	28.67	3292.26
Run 10	156.68	155.06	1.491	28.96	3325.04
<b>Average</b>	<b>156.59</b>	<b>155.02</b>	<b>1.495</b>	<b>28.78</b>	<b>3304.51</b>
<b>Std. Dev.</b>	<b>0.08</b>	<b>0.08</b>	<b>0.003</b>	<b>0.18</b>	<b>20.39</b>
<b>R.S.D.</b>	<b>0.05%</b>	<b>0.05%</b>	<b>0.18%</b>	<b>0.61%</b>	<b>0.62%</b>

Seen on the next page is an overlay plot of the ten (10) DSC analysis runs from which the above chart was taken. They have been laid on the same graph and the Y-Axis skewed for easy comparison.

<sup>1</sup> Shimadzu Technical Bulletin Ref. SSI-TA-JG-011

<sup>2</sup> N.I.S.T SRM 758, I.C.T.A. GM 758

# Application Note



## Conclusion

As is plainly seen from the data, the DSC-50 provides excellent accuracy, precision, and detailed repeatability. These statistics are well within Shimadzu published specifications<sup>3</sup> detailed above, and it would be hard to find similar instrumentation on the market today which could compare itself to these results. In conclusion, if the DSC-50 being properly calibrated, then the sample analysis results being reported can be relied upon to be correct.

<sup>3</sup> Shimadzu DSC-50 Instruction manual (222-00021) page 11