

## INTRODUCTION

The ASTM Standard E1269 using traditional DSC has been used for heat capacity measurements for decades, but it requires three experiments under the same experimental conditions [1] and can take hours to finish. Additionally, the accuracy is less than desired. Modulated DSC® is a good tool to measure heat capacity and only requires one experiment. It can be used on a simultaneous DSC-TGA SDT650 to obtain heat capacities and provides great accuracy and repeatability up to 1500 °C. Calibration and sample testing can be easily set up in Trios.

## CALIBRATION AND EXPERIMENTAL

The measurement of heat capacity using MDSC is based on the amplitude of the modulated heat flow. The calculation of the MDSC heat flow and MDSC reversing heat capacity are discussed in detail in the TA Apps Note TP007 [2]. With MDSC, only one experiment is needed to obtain heat capacity. A calibration using sapphire or other standard materials is required. On an SDT650, experiments can go up to 1500 °C for heat capacity determination. The recommended MDSC experimental procedure including the MDSC parameters is shown below: starting modulation at  $T_1$  and then ramping at 5 °C/min to  $T_2$ .

1. Data Off
2. Equilibrate at  $T_1$  °C
3. Modulate Temperature 3.00 °C for 200.0 s
4. Isothermal 5.0 min
5. Data On
6. Ramp 5 °C/min to  $T_2$  °C

To calibrate heat capacity with a standard material such as sapphire, access the Calibration Setup window in Trios and choose the MDSC Reversing Heat Capacity Calibration method. Create a method similar to that above using either alumina or platinum cups. It is important to match the reference pan mass with the sample pan mass for heat capacity measurements. The mass difference between the reference pan and the sample pan should be less than 1 mg. The method will calibrate the heat capacity of sapphire as a function of temperature and generate a calibration constant at each data point covering the entire temperature range. After the calibration is finished, the calibration data will be saved to Trios automatically.

## RESULTS

Two sapphire standard disks were tested in  $N_2$  in this work. They were 60.762mg and 60.892mg, separately. One sapphire disk was used to calibrate the heat capacity on the SDT650 from 300 °C to 1250 °C. Then these two sapphires were used as samples to measure heat capacity values of sapphire after the calibration. Calculation of heat capacity is done in Trios. When using the MDSC method for heat capacity measurements, the reversing heat capacity (Normalized) signal is plotted vs. temperature. The heat capacities of the two sapphire disks from the tests are illustrated in the graph below as compared with the literature values (Figure 1).

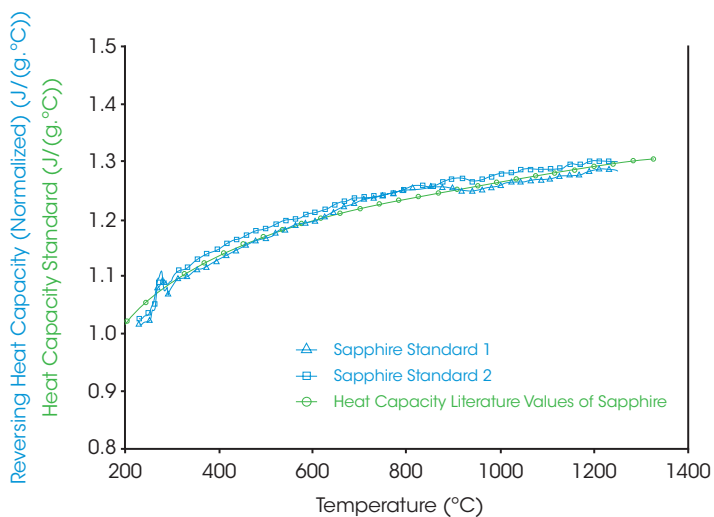


Figure 1. Measured heat capacities of the two sapphire standard disks and literature values of sapphire up to 1250 °C.

The percent error of the two tests was calculated from 400 °C to 1250 °C at 50 °C increments as shown in Table 1. Over the entire temperature range, the percent error from the two tests is within 1.6%. This shows good accuracy and reproducibility of heat capacity measurements on the SDT650 at high temperatures.

Temperature	Heat capacity literature values	Heat capacity Sapphire 1	Heat capacity Sapphire 2	% error Sapphire 1	% error Sapphire 2
°C	J/(g.°C)	J/(g.°C)	J/(g.°C)	%	%
400	1.137	1.149	1.13	1.0554	0.6157
450	1.155	1.17	1.152	1.2987	0.2597
500	1.171	1.184	1.168	1.1102	0.2562
550	1.185	1.199	1.185	1.1814	0.0000
600	1.197	1.211	1.196	1.1696	0.0835
650	1.208	1.227	1.216	1.5728	0.6623
700	1.218	1.238	1.233	1.6420	1.2315
750	1.227	1.244	1.242	1.3855	1.2225
800	1.236	1.254	1.251	1.4563	1.2136
850	1.244	1.258	1.256	1.1254	0.9646
900	1.251	1.268	1.254	1.3589	0.2398
950	1.258	1.267	1.251	0.7154	0.5564
1000	1.265	1.279	1.26	1.1067	0.3953
1050	1.272	1.287	1.265	1.1792	0.5503
1100	1.278	1.288	1.269	0.7825	0.7042
1150	1.284	1.297	1.276	1.0125	0.6231
1200	1.29	1.302	1.287	0.9302	0.2326
1250	1.296	1.3	1.284	0.3086	0.9259

Table 1. Percent (%) difference from the measured heat capacities of the two sapphire disks vs. the literature values from 400 °C to 1250 °C at 50 °C increments.

## CONCLUSION

Modulated DSC® (MDSC®) is a well-established technique for obtaining heat capacity. It is simple and straight forward compared to the ASTM method E1269. A calibration with sapphire or other standard materials is required prior to running samples. With MDSC, heat capacity measurements can be performed at higher temperatures up to 1500 °C on an SDT650 with excellent accuracy and repeatability.

## ACKNOWLEDGEMENTS

This paper was written by Yue Schuman, Ph.D., Applications Support Engineer at TA Instruments.

## REFERENCES

1. ASTM E1269-11, Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry, ASTM International, West Conshohocken, PA, 2011, [www.astm.org](http://www.astm.org).
2. Modulated Paper #2; Modulated DSC Basics; Calculation and Calibration of MDSC Signals; TA Instruments Technical Paper (TP 007).

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