TA Instruments

Thermal Analysis & Rheology

# THERMAL APPLICATIONS NOTE

## Hints for Good Purity Determinations

1. **SAMPLE PANS**

   - Use aluminum hermetic pans. Do not use coated aluminum hermetic pans.
   - Clean sample pans and lids in a solvent (such as acetone) and heat them in an oven at 100°C for approximately 5 minutes. Once the pans are cool, store in a sealed jar for future use.

2. **SAMPLE ENCAPSULATION**

   - When sealing the hermetic pans, place the cover in an inverted position on the top of the pan to minimize the internal volume of the sealed pan.
   - To affect better sealing of the cover, use a 10 to 15 cm stainless steel rod of 5.9 mm diameter. Place the covered pan in the hole of the inverted DSC sample press preforming tool to hold it in position for use of the stainless steel rod. Take the stainless steel rod and place its end onto the cover. Gently tap the rod with a hammer. This action forms the cover lip to the pan lip and permits a better seal. Then, place the entire pan assembly in the sample press and crimp the pan as usual using the preforming tool.

3. **IMPURITY LEVEL:** <2 mole %

4. **SAMPLE CONDITIONS:**

   - Impurities should not form a solid solution with the main component
   - Sample should not decompose upon melting
   - Impurities are soluble in the liquid phase of the main component
   - No other thermal events in the vicinity of the melting region, i.e. no volatile losses or polymorphic transitions

5. **OPTIMUM SAMPLE SIZE:** 1.7 mg (± 0.3 mg)
   Smaller sample sizes are recommended for large ΔH values.

6. **RECOMMENDED DATA SAMPLING INTERVAL:** 1 second / data point (120 data points/°C)

7. **OPTIMUM HEATING RATE:** 0.5°C/minute
   Slower heating rates are recommended for purity levels greater than 99.5%.

8. **ANALYSIS OF DATA**

   - Integrate the melting endotherm with the DSC Purity program. The area correction value should not exceed 20%.
   - Weigh the cooled sample pan after the experiment is complete. If there is a weight loss exceeding 1%, repeat the run. It is possible that a satisfactory seal was not obtained causing a loss of sample.

**REFERENCE:** J. E. Hunter, III and R. L. Blaine, *Optimization of Accuracy and Precision in the Differential Scanning Calorimetry Dynamic Purity Method*, Purity Determinations by Thermal Methods, ASTM STP 838, 1983, 29-38.

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