Characterizing Solid Compounds by DSC and TGA

1. Differential scanning calorimetry (DSC) represents a much more modern and precise method of obtaining melting and decomposition point data. There is also the possibility of observing an extra phase transition (besides the melting point), which might be due to liquid crystal intermediates, some type of solid-to-solid phase transition, etc. Just about everything you need to know is contained in this outstanding review article:

   Cammenga, H. K.; Epple, M. *Angew. Chem.* **1995**, *107*, 1284; *Angew. Chem., Int. Ed.* **1995**, *34*, 1171.

   We commonly give a representative trace as a Figure, and report the following values: T_i, initial peak temperature; T_e, extrapolated peak-onset temperature; T_p, maximum peak temperature; T_c; and T_f.

   For cases where two or more phase transitions are observed, it is important to record a DSC trace with both heating and cooling (phase transitions observed in both directions, but shifted). The behavior must be reproduced with additional samples.

   http://plc.cwru.edu/tutorial/enhanced/files/lc/intro.htm

   http://plc.cwru.edu/tutorial/enhanced/files/lc/phase/phase.htm (see figures a and b for DSC traces recorded in both directions)

   Ways in which DSC data can be reported include:

   DSC: endotherm with T_i, 181.6 °C; T_e, 244.0 °C; T_p, 263.7 °C; T_c, 270.3 °C; T_f, 270.2 °C; exotherm with T_i, 271.0 °C. (from paper #357)

   DSC: desolvation endotherm with T_i, 45.3 °C; T_e, 71.4 °C; T_p, 99.5 °C; T_c, 112.9 °C; T_f, 131.2 °C; exotherm with T_i, 172.9 °C; T_e, 190.9 °C; T_p, 203.1 °C; T_c, 211.8 °C; T_f, 215.2 °C; endotherm with T_i, 215.3 °C; T_e, 227.9 °C; T_p, 241.1 °C; T_c, 246.1 °C; T_f, 246.1 °C. TGA: mass loss 1.9% between 60 °C and 102 °C (calculated for $(CH_2Cl_2)_{0.67}$, 1.7%); onset of further mass loss (T_e), 236.3 °C. (from paper #357)

2. Thermogravimetric analysis (TGA) is a way to characterize the weight loss of a sample. Since a melting point doesn't involve a weight loss, and a decomposition point may or may not involve a weight loss, this technique is less useful for us. In the right cases, it can give some idea of how tightly a solvate molecule is bound to a compound or in the crystal lattice.

   Ways in which TGA data can be reported include:

   TGA: onset of mass loss (T_e), 222 °C

   TGA: onset of first mass loss regime (T_e), 90 °C (theory for $(benzene)_{2.0}$, 8.2%; observed, 7.7%); onset of second mass loss regime (T_e), 234 °C.

   See also the above DSC examples