Differential Scanning Calorimetry at Dalhousie University

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Instrument available for use as part of the
IRM Facilities for Materials Characterization

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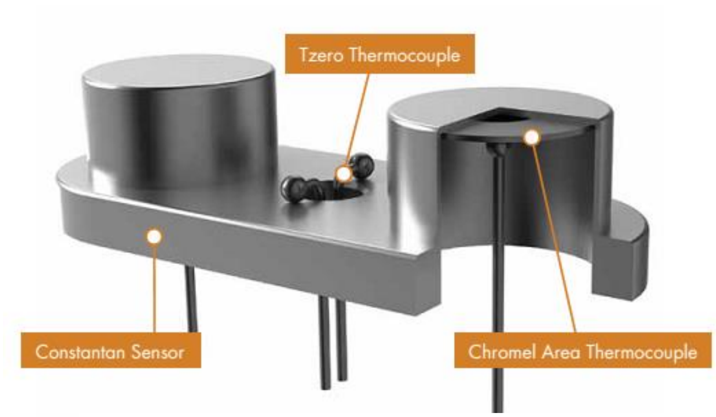
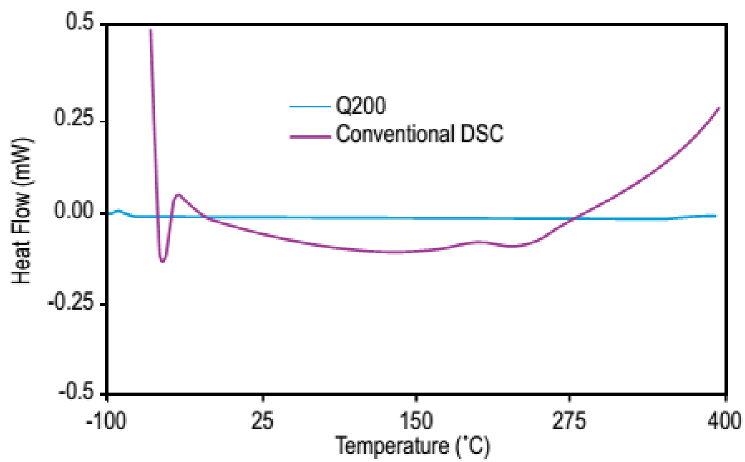
Differential Scanning Calorimeter set-up

- TA Instruments Q200 differential scanning calorimeter (DSC)
- Key features:
  - Temperature range of -170 °C to 500 °C
  - Two cooling accessories:
    - Air cool (ambient to 500 °C)
    - Liquid nitrogen cooling (-170 °C to 400 °C)
  - Tzero® technology

Advantage of Tzero®





From TA instrument literature.

- Tzero® functionality comes from additional thermocouples to detect capacitance and resistance imbalances in the cell and compensate for it in the heat-flow algorithm.
- **Result:**
  - **Flatter, reproducible baseline**
  - **Increased sensitivity**
  - **Better resolution**

Sample morphologies

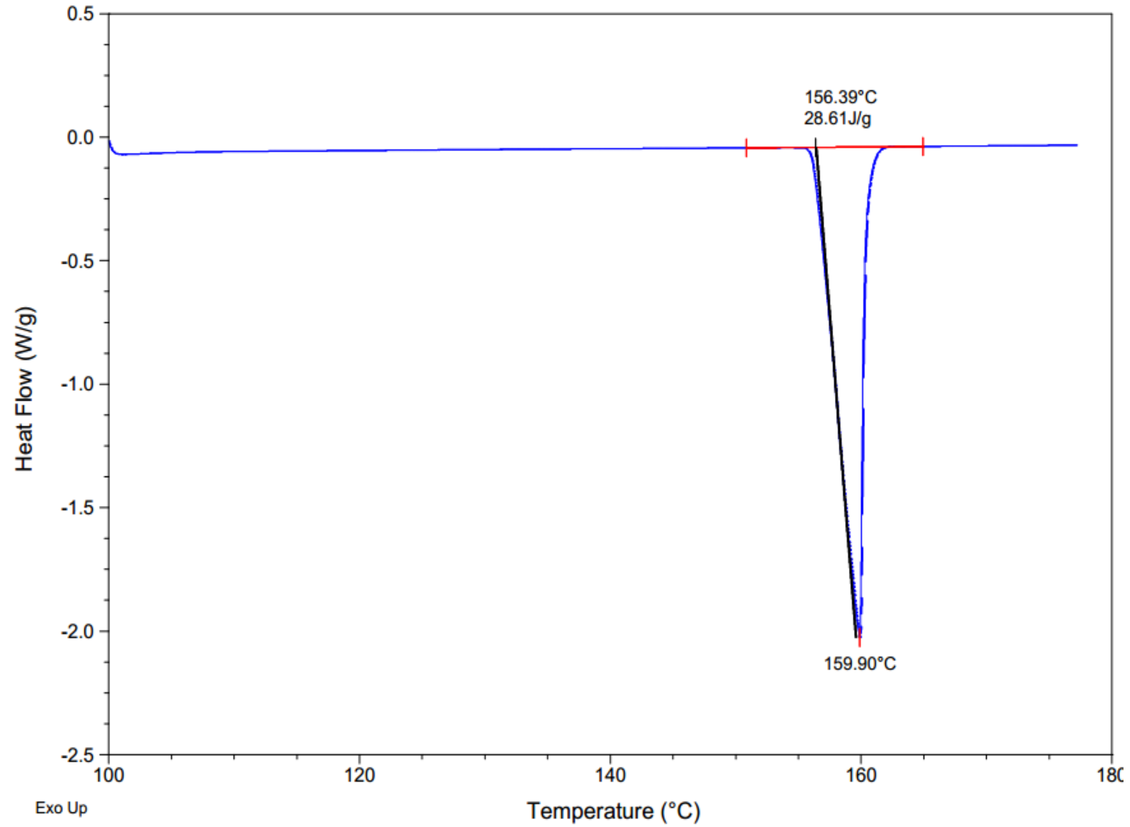
- Sample sizes for analysis ranges from 3 to 70 milligrams.
- Sample morphology for DSC are solid, liquid, and gel samples.
  - Solid samples include powders, glasses, metals, crystals, and films.
- Samples are generally encapsulated in hermetic aluminum pans.
  - Air sensitive samples can be encapsulated in an inert atmosphere.

Applications

- Chemistry, biology, engineering, and material science can find use with DSC:
  - Glass transition
  - Cold crystallization
  - Phase change(s)
  - Melting
  - Crystallization
  - Product stability
  - Cure/cure kinetics
  - Oxidative stability

Instrument validation - 1

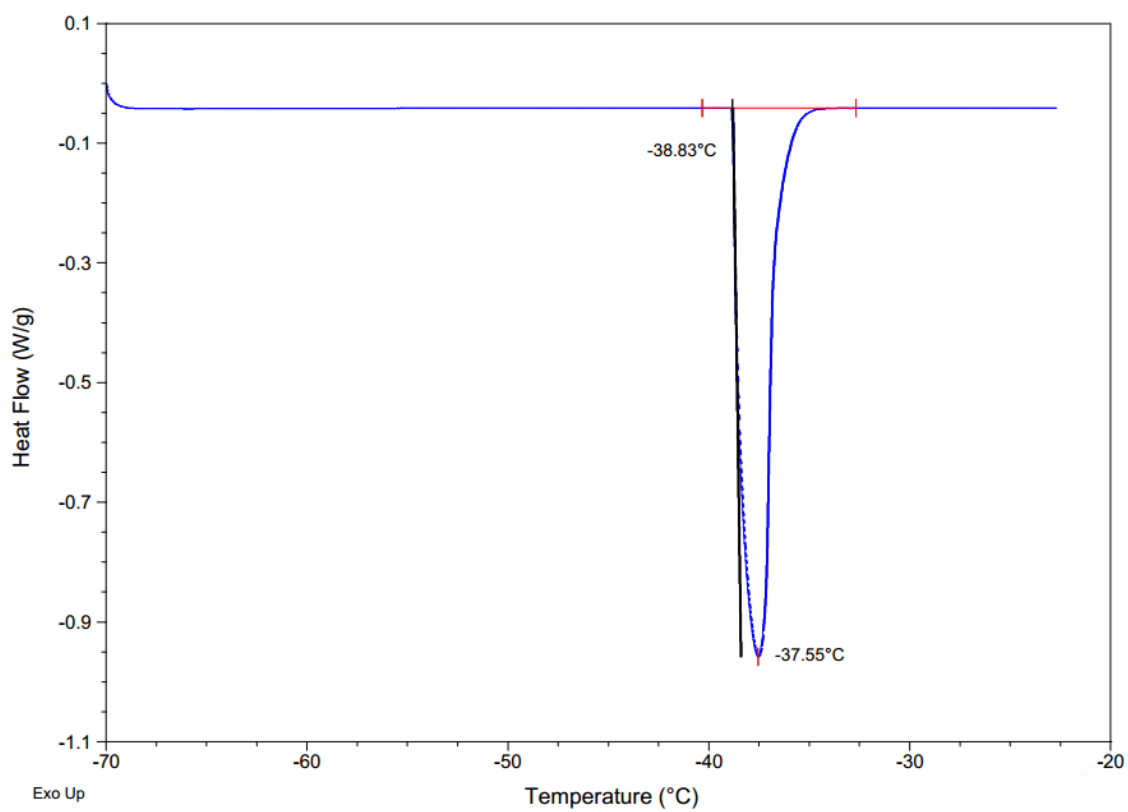
- Indium is used as a temperature calibration material for referencing
  - Good agreement with ASTM protocols[1]



[1]ASTM E967-08: Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

Instrument validation - 2

- Mercury is used as a low-temperature calibration material for *T*-referencing
  - Good agreement with ASTM protocols[1]
- Further checks are made with DPNCl (-29.34 °C)[2] and water (0 °C)



[1] ASTM E967-08: Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

[2] M.J.M. Van Oort and M.A. White. *Ber. Bunsenges*. *Phys. Chem*. **92**, 168 (1988)

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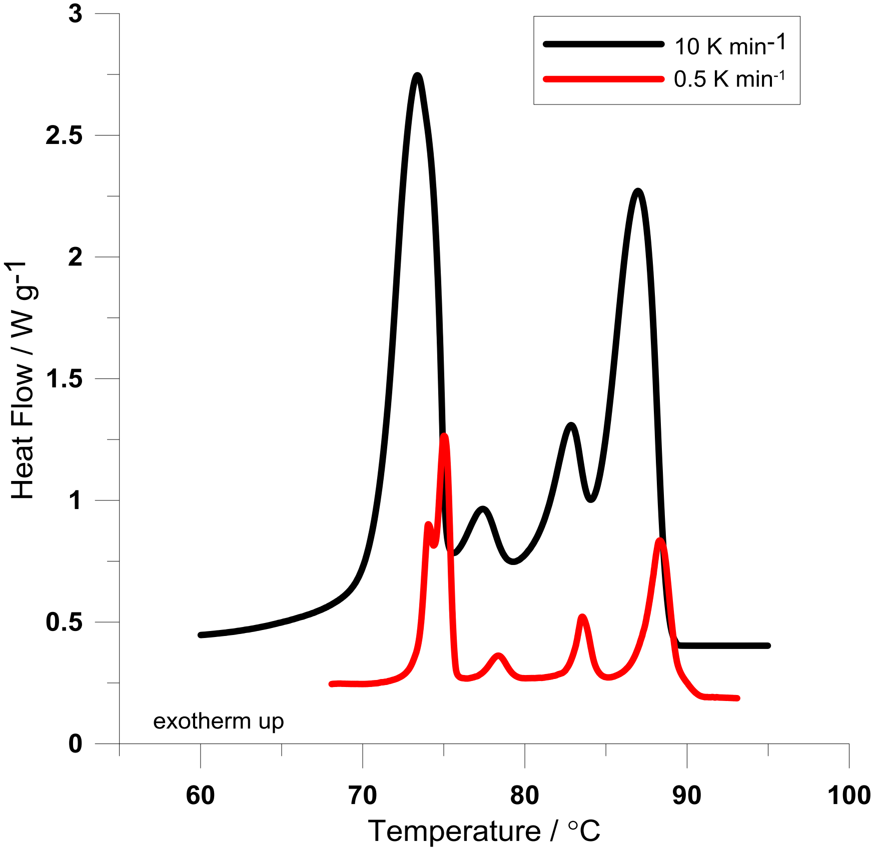
Experimental case: $CdCl_4$ complex

- $(n\text{-}C_{18}H_{37}NH_3)_2CdCl_4$ was analyzed by adiabatic calorimetry (AC) and DSC in 1984[3]
  - Sample showed 4 solid-solid transitions by AC
  - DSC was only able to resolve 2 peaks
- This was revisited with the TA Q200 DSC[4]
  - Able to easily resolve 4 peaks
  - At very slow heating rates, one peak splits suggesting further molecular movement



[3] M.A. White. *Thermochimica Acta*, 7**4**, 55 (1984)
[4] M.B. Johnson and M.A. White. *Thermal Methods*. Chapter 2 *in Inorganic Materials: Multi Length-Scale Characterisation*; D.W. Bruce, D. O'Hare and R. I. Walton, Editors. Wiley (2014)

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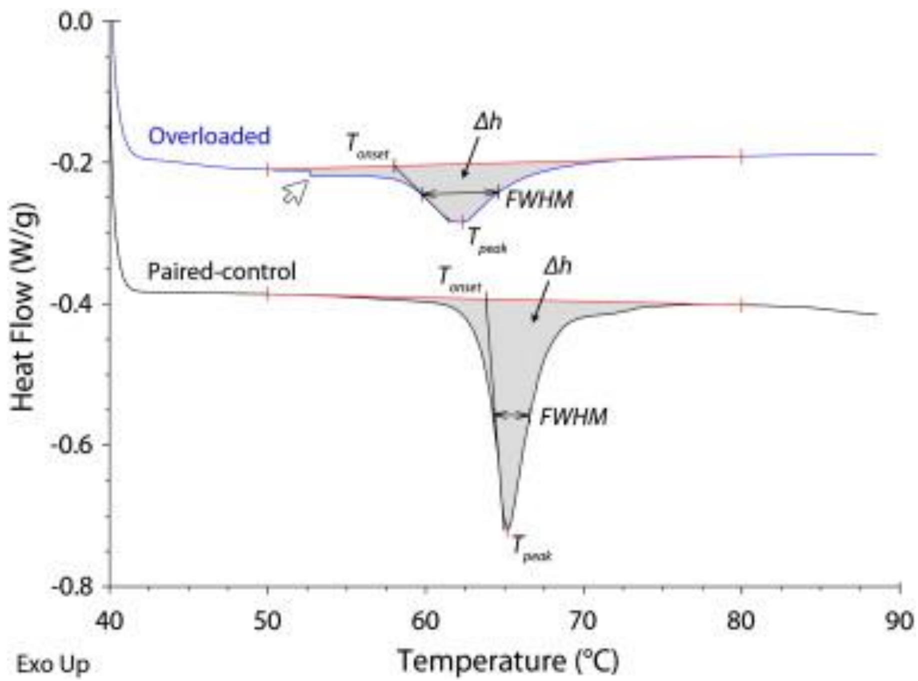
Experimental case: Denaturing

- Biological materials can also be analyzed with DSC. This specific case investigates denaturing temperatures and the tendon overload[5]
  - T_{onset} lowered by 4 °C in overloaded tendons
  - Enthalpy (Δh) decreased over 50% in overloaded tendons
    - 6.2 W g^{-1} in overload; 13.3 W g^{-1} in control
  - Consistent with expectation of damaged tendons requiring lower energy to denature



[5] S.P. Veres, J.M. Harrison, and J.M. Lee. *Matrix Biol.* **33**, 54 (2013)

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