TAM III Microcalorimetry: Standard operating procedure

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Description

TAM III micro-calorimeter is an ultra-sensitive heat flow measurement device for real-time monitoring and dynamic analysis of chemical/physical processes, over a period of hours or days. It can be used to determine the onset, rate, extent and energetics of various processes for specimens in small ampoules (3–20 ml) at a constant set temperature (ca. 15°C–150°C) or temperature ramp (0-2K/hr).

Before starting:

1) Ensure that the work area is clear of dust, debris, material samples, and any lab components not directly used for the experiment.
2) Put on appropriate personal protective gear (safety glasses, lab coat and gloves)

Loading TAM III ampoules using glovebox in Lab 221 for sensitive materials:

1) Place approximately 30-60 mg of wet sample (under hexane) in 50 ml glass vial.
2) Place glass vial, 20ml disposable crimp seal TAM ampoules, and ampoule cover inside glove box under argon.
3) Using balance located inside glove box, accurately record stabilized weight of sample as hexane evaporates.
4) Add heavy paraffin oil, CAS: 8042-47-5 to completely immerse the sample.
5) Seal TAM ampoules using the crimpling tools and attach hanger at the top of ampoule.
6) Remove the ampoule from the glove box and carry it to Lab 107 where TAM III is located.

Loading TAM III:

1) Select appropriate experimental mode on TAM III interface: isothermal/scanning
   Note: Only two samples can be loaded at a given time.
2) Input sample weight and other sample details into device software.
3) For isothermal mode: select experimental furnace temperature: 30-140^{o}C.
   a. Allow device to acquire initial baseline heat flow signals for at least 30 min.
   b. Remove ampoule lifter from device and connect with hanger attached to the sealed ampoule.
   c. Lower sample into equilibration position using the indicated mark on the ampoule lifter and held it there for 15 min.
   d. Lower sample into the measuring position and start recording data.
   e. Stop after desired experimental time (hours/days).
   f. Remove samples and allow device to acquire final baseline.
4) For scanning mode: select desired heating rate: 0-2 K/hr
   a. Input desired temperature program.
   b. Remove ampoule lifter from device and connect with hanger attached to sealed ampoule.
   c. Lower sample directly into measuring position without automatic baseline.
   d. Allow heat flow signals to stabilize for 3-6 hours.
   e. Start temperature program.
   f. Remove samples upon completion of temperature program.

Unloading TAM III ampoules:

1) Safely dispose remaining material from ampoule into waste container for reactive powder ensuring appropriate (1:3) dilution with sand.