# SOFT MATERIALS CHARACTERIZATION CORE FACILITY OPERATING PROCEDURES

1) **What are the Instruments**: The Soft Materials Characterization Core Facility is comprised of a Thermogravimetric Analyzer (TGA), Dynamic Scanning Calorimeter (DSC), Zeta-sizer, (note: SAXS is part of XRay core)

2) **How do I use the instruments**: Follow this procedure:
   a. **First**: Contact the superuser or POC by email for the particular instrument you wish to use. Specifically, what the POC/superuser wants to know:
      i. what you want to do
      ii. what information you are seeking
      iii. at what temperature, force, etc is the phenomena you are looking for
      iv. what is the material, and what does it react with (specifically Al, steel and Pt)
      v. will the material off-gas at temperature and what is the gas (is it reactive or condensable?) **(I will need to see a TGA first BEFORE DSC)**
      vi. how many samples.
   b. **Second**: Once the superuser/POC says that you can use the instrument and what limits are necessary, you will be directed to schedule training and/or sample submission. YOU WILL NEED A VALID iLABS ACCOUNT TO DO THIS! We will log you in to verify.
   c. **Third**: Once training is complete, schedule time and use the instrument!

3) **Can I submit specimens for analysis rather than get trained**: Yes. Contact the POC to decide whether this is allowable (based on how many specimens/ how much time/how difficult).

4) **Scheduling**: This is done through iLabs. However, ½ day a week is reserved for maintenance/calibration by the superusers. Training is usually done at this time.

5) **How much does it cost**: Costs are below and are based on the approximate time of training during assisted use. For submitted samples the instrument is considered assisted use which adds $65 to the internal rate, with a minimum 1 hr of use.

| | | | Internal | External Submitted Sample | |
|---|---|---|---|---|---|
| | **Training** | **Hourly** | **Submitted Sample** | **Non-Profit** | **For-Profit** |
| TGA | $200 | $24.75 /hr | $69.6 /hr | $113.25 /hr | $113.25 /hr |
| DSC | $200 | $20.56 /hr | $65.41 /hr | $102.70 /hr | $102.70 /hr |
| Zeta-sizer | $200 | $31.7 /hr | $76.55/hr | $120.18/hr | $120.18/hr |

**No longer operational:** Rheometer, Contact angle goniometer

| **Not part of SMC** | | | | | |
|---|---|---|---|---|---|
| SAXS | hourly | $13.4/hr | $58.3/hr | $713.43/hr | $713.43/hr |

6) **Point of Contacts (POC)s**:
SMC Director/Point of Contact (POC): Prof. Jeffrey Youngblood (jpyoungb@purdue.edu)

**7) Superusers:**
TGA: Wei-Ting Chung (chung380@purdue.edu), Sam Schmidt (schmidsd@purdue.edu), Akshat Verma (verma163@purdue.edu)
DSC: Tuan Le (le316@purdue.edu)
Zetasizer: Siddhant Kalel (skalel@purdue.edu)
iLabs (confirming) Sam Schmidt (schmidsd@purdue.edu)
iLabs (billing/charging): Amy Stanley (stanley7@purdue.edu)

**No longer operational:** Rheometer, Contact Angle Goniometer, DMA

**Not part of SMC:**
SAXS: Matt Kaboolian (mkabooli@purdue.edu), Siddhant Kalel (skalel@purdue.edu)
BET: BET is within MSE dept room recharge. Sam Schmidt (schmidsd@purdue.edu)
FT-IR: FTIR is within MSE dept room recharge. Sam Schmidt (schmidsd@purdue.edu)

# SOFT MATERIALS CHARACTERIZATION CORE FACILITY OPERATING PROCEDURES

## TGA

1) **Operational limits**: This instrument has a range of Room Temp to 1000 C. It only measures mass change and cannot measure heat flow or differential temperature (ie IT IS NOT A DTA/SDSC). Only use a reasonable mass of materials – **12-25mg**.
2) **Temperature**: Know about what temperature your material loses mass - Only go about ~100 C above the lowest plateau (If there is no reason to go to 1000C then don't). If you don't know, then do a reasonable guess and run the material a bit high in temperature to see then adjust subsequent runs. Organic polymers typically are all done below 500 C or so. **Allow adequate time for the furnace to cool in between subsequent runs.**
3) **Materials**: Know your material – never go close to the boiling temperature. If your material evaporates, then stay WAY below the temperature that it does so. Know what your material reacts with, specifically platinum (There are a variety of platinum eutectics). If you ruin a pan or thermocouple, you will be charged for a new one.
4) **Evolved gases**: Know what your material off-gases – if it degrades to small molecule organics or CO2 (run in air) then it is fine. However, if OFF-GASSES ARE CONDENSIBLE AT ROOM TEMPERATURE OR REACTIVE THEN IT IS NOT ALLOWED. This means that materials such as zinc, mercury, etc cannot be run.
5) **Provide your own pan:** You will be expected to provide your own pan for samples. See below for proper pan size/material, but we can provide the vendor. They cost ~$600, but are reusable. They must be the proper type for this instrument and un-deformed or you will damage the hook and you will pay for that as well. ONLY PLATINUM (Pt) OR ALUMINA ($AL_2O_3$) ARE ALLOWED.
6) **Proper Pan**: Your material should not be anywhere near the top of the pan – do not overflow. If it is close, use a different pan that holds more. Know if your material shows a eutectic with Pt (P,B, Si, As), in which case use alumina pans and do it in air and **ONLY PANS FROM TA INSTRUMENTS**.
7) **Proper heating rate**: Generally, 20C/minute is ok (for loose powder with space between grains). However, some materials need extra time to heat or evolve gasses – foams, packed powders, and solid materials with residual solvent/liquid may need slower ramp rates. DO NOT EXPAND, FOAM, OR EXPLODE SAMPLES IN THE INSTRUMENT.
8) **Cleaning your pan**: Only use a blow torch, low pressure air and possibly acid wash to clean the Pt Pan. Never scrape, wire brush, abrade or mechanically clean it. Also, treat the pan gingerly as it is mechanically weak. DEFORMING THE PAN CAN RUIN IT AND DAMAGE THE INSTRUMENT! We have an air furnace to burn out any char and a neat trick is to switch your gas to air and heat to 600C for a bit to burn out any char.

**NOTE**: We have an autoloader for a reason –

- **NEVER EVER MANUALLY LOAD OR UNLOAD THE PAN ON THE HOOK!**
- DO NOT PUT ANY MATERIAL CONTAINING THE FOLLOWING ELEMENTS IN A PLATINUM PAN: PHOSPHOROUS, BORON, SILICON, OR ARSENIC – EVEN IF OXIDES! USE ALUMINA PANS INSTEAD.
- ONLY RUN PHOSPHOROUS, BORON, SILICON, OR ARSENIC IN AIR (no other gases allowed).
- DO NOT RUN FLUORINATED MATERIALS (PTFE, PVDF, ETC) IN ALUMINA PANS AS THEY WILL CRACK. USE Pt PANS INSTEAD.
- TREAT THE PAN WITH CARE – DO NOT DEFORM IT.
- NEVER RUN A DEFORMED PAN IN THE TGA – it could damage the instrument and you will be charged.
- Never augment any aspect of the device except for changing the input gasses as needed and taring the tool. This includes changing the TGA counterweight pan (the same pan can be used regardless of the pan you use to run your samples), adjusting the auto loader dock height, changing a hook or thermocouple, or fishing out a pan that has fallen into the furnace. You will be charged for any damage you cause to the tool by adjustments you make to the tool.

# SOFT MATERIALS CHARACTERIZATION CORE FACILITY OPERATING PROCEDURES

## DSC

**NOTE: It is required that you perform a N2- TGA on your material prior to DSC using the same ramp program and set your temperature limits below 2% weight loss. Also, poke a small pinhole in the top to release pressure, just in case.**

1) **Experiment types:** thermal measurement of melting, freezing, and glass transitions, curing point.
2) **Operational limits**: Operational range is determined by the cooling accessory. With LN2 cooling -160°C to 400°C. (it may say up to 500C, but the lid doesn't go down properly and the instrument goes offline for repair)
   NOTE:
   - Isothermal experiments longer than and hour are not allowed on the LN2 unit, nor are ramp rates <1 deg/min.
3) **Changes to heaters**: Only superusers are allowed to switch heating units.
4) **Required materials:** Disposable aluminum pans are provided for users. The Air-Fin cooling system cannot use aluminum pans and users must provide their own sample pans (copper, platinum, graphite, etc. are available from TA Instruments).
5) **Sample requirements:**
   - User samples of mass between 1 mg to 4 mg are needed. Best measurement conditions have sample mass of 3 mg to 4 mg. Do not over fill the DSC pans which can ruin the pan press and the DSC sample chamber.
   - Do not allow material to decompose during the DSC run; no off-gassing should occur in DSC chamber. (IF YOUR MATERIAL EXPANDS TO FORM A BUBBLE ON THE TOP OF THE CRIMPED PAN, THEN IT IS OFF-GASSING TOO MUCH!) Test your material in the TGA first if these transitions are unknown and keep weight loss to 2% (if more than this dry it first as it is likely water).
   - If your material reacts with Aluminum, then use a more appropriate pan. If a material damages the instrument, you will be charged for repairs.
   - ***NO UNCURED RESINS ARE ALLOWED***.
6) **Machine Settings:** Always replace hard cap over sample chamber when experiment is complete. N2 and helium gas should be turned off at end of the experiment.

.

# SOFT MATERIALS CHARACTERIZATION CORE FACILITY OPERATING PROCEDURES

## Zetasizer

1) **Experiment types:** Particle Size Distribution and Zeta Potential
2) **Operational limits**: Particle size between 0.3 nm to 10 um.
3) **Required materials:** Disposable or glass cuvette for size analysis (Mavlvern DTS0012). Zeta potential cell for zeta potential measurements (Malvern DTS1070).
4) **Sample requirements:** Particles must be within the size range (0.3 nm to 10 um). Particles should be as close to spherical as possible as the software uses this assumption to calculate the size. If not, it will still report a particle size but it will not be accurate. Sample must be dispersed in a solvent. This dispersion should be stable and not rapidly separate (sediment or cream). Dispersion should also be transparent to translucent; opaque samples cannot be measured. To achieve this, it is recommended that dispersions be made between 0.01 and 1 wt% and tested to find the optimum concentration for measuring. The solvent used must be compatible with the measurement cell material (e.g. do not use acetone or toluene in a polystyrene cuvette) or the Zetasizer can be damaged.
5) **Notes:** Samples should always be capped when measurement is taking place. After use, Zetasizer should be turned off. Since this device is not on iLabs, the user must sign-in on notebook when they use the Zetasizer to keep track of users.