International Materials Institute for New Functionality in Glass

Sponsored by US National Science Foundation

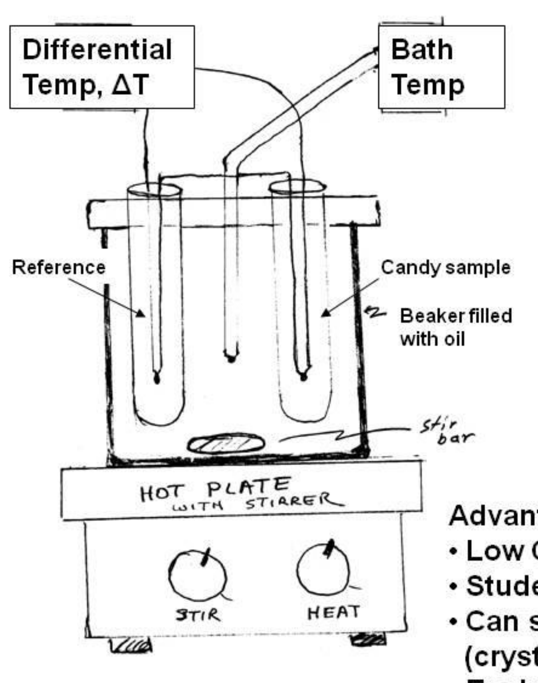
# A Low-Cost Student Built DTA for Exploring the Glass Transition

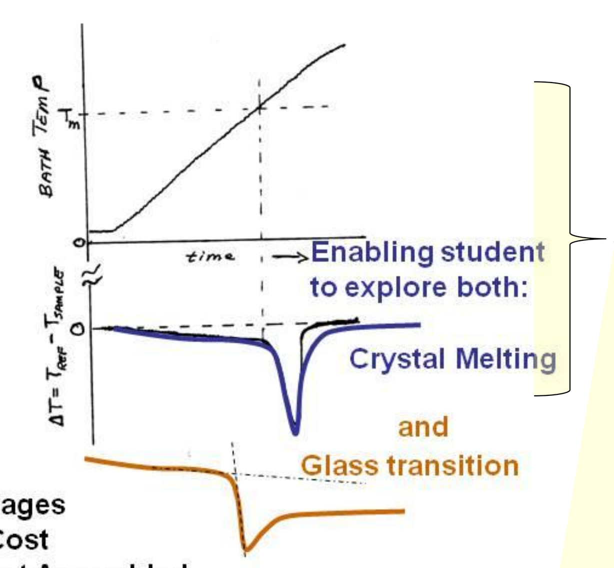
William Heffner, IMI-NFG, Lehigh University, Bethlehem, PA

**Objective: As part of a series of low-cost experiments for students to explore glass science, we have developed a simple student-assembled apparatus for measuring the glass transition ($T_g$). While DSC is the common technique for measuring $T_g$, such apparatus is expensive and generally unavailable outside of the research laboratory. Differential thermal analysis (DTA) is somewhat simpler to implement and provides essentially the same information.**

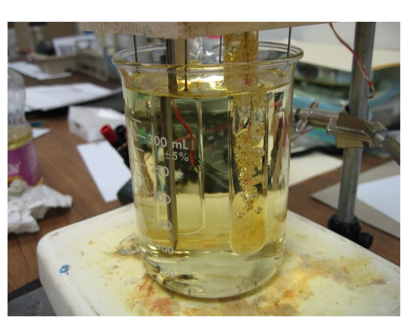
**Our DTA consists of measuring the temperature difference between test tubes with the sample and a reference material, while both test tubes are heated in an oil bath. It provides excellent resolution of the $T_g$ of two low temperature glasses - sugar glass and PET. and would be suitable for an undergraduate material science laboratory.**

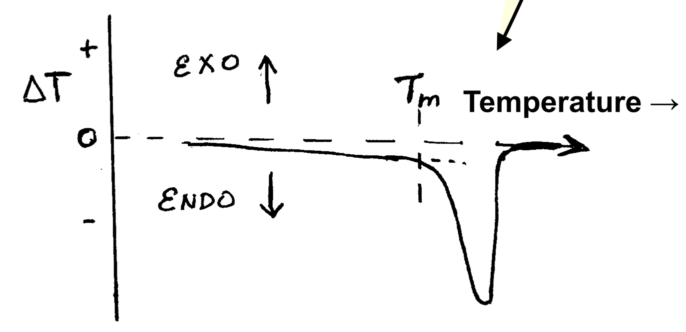
## Thermal Analysis Apparatus for the Student




Advantages
- Low Cost
- Student Assembled
- Can see what's happening (crystallization, melting, gas evolution, etc)
- Enables exploration of subtleties of Tg

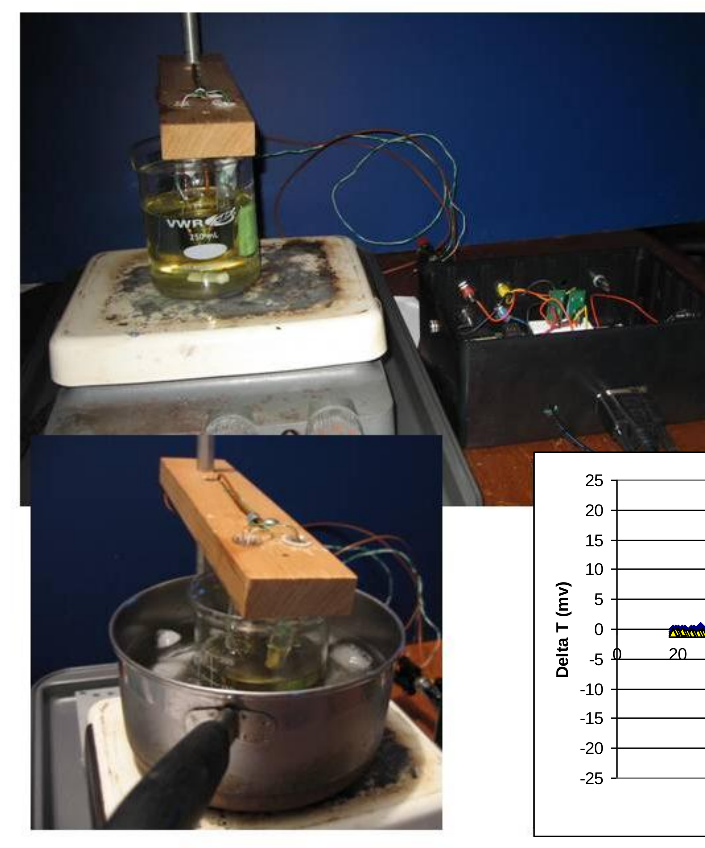


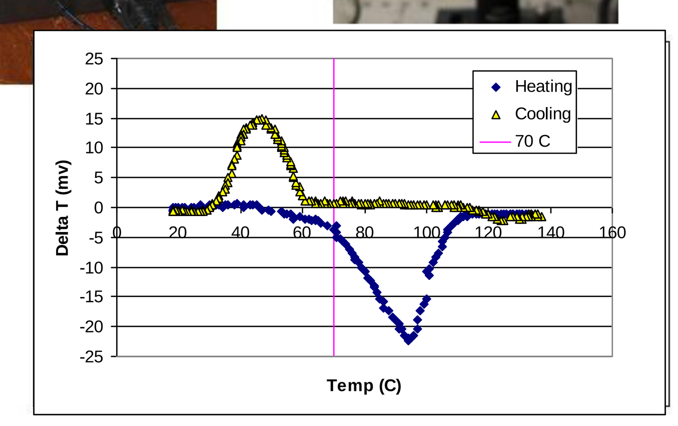



**Apparatus**

A standard hot plate with magnetic stirring is used to heat a beaker full of vegetable oil and the two test tubes . The same cooking oil is used as the reference material. The differential thermocouple is made using a 6" piece of constantan soldered to two copper lead wires. The pair are connected to the thermocouple meter so that the exotherm is positive.

**DTA with Home Built Data Logging and Cooling Option**






Stearic Acid – calibration

### Samples

Ideal sample materials for student investigation should be:
easily accessible and low in cost with moderate transition temperatures (ideally 50 -130 C), to avoid working with very hot oil and yet be sufficiently above RT for baseline stabilization.

- Stearic acid as standard ($T_m$ ~ 70° C)
- PET plastic for $T_g$ and Crystallization ($T_g$ ~ 73°, $T_{xtal}$ ~ 125° C)
- Candy Glass for $T_g$ ($T_g$ ~ 30-50° C, depending on water content)

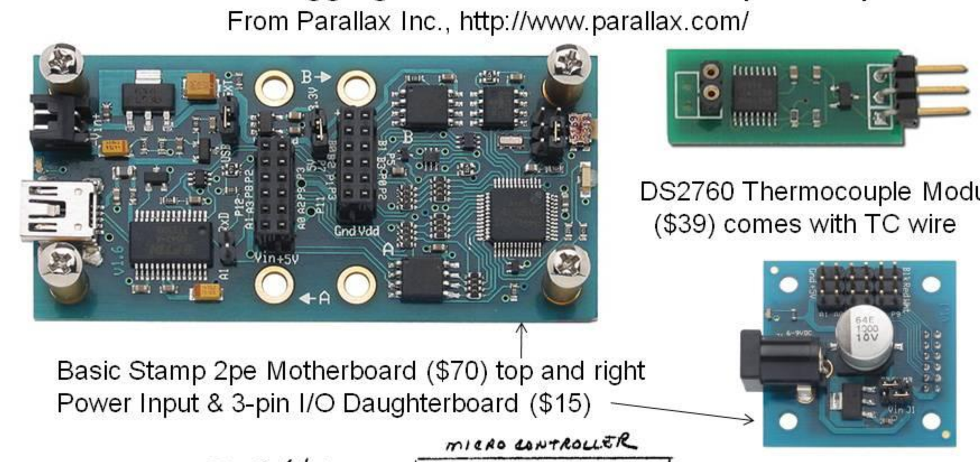
Also tested Polystyrene and Benzoic acid, but T too high for student safety!

## Student-built Data Logger

While the DTA can easily be implemented with manual data collection, this is some what tedious and a distraction from observing the sample during its heating. Automated data collection is especially helpful when measuring the multiple runs required for the development of a project or a new method. One could use lab acquisition products such as Vernier or Pascal, standard in most high school physics labs . However, keeping with the low cost, hands-on approach, we chose to build our own data logging apparatus from a simple but powerful microprocessor, the Basic stamp, from Parallax Inc. (http://www.parallax.com/), combined with their add-on thermocouple module. The data is collected and sent to the PC over the serial (USB) line and pulled directly into an active Excel spreadsheet, where data can be plotted real-time, as it is collected. Details of design and software can be found on our website.

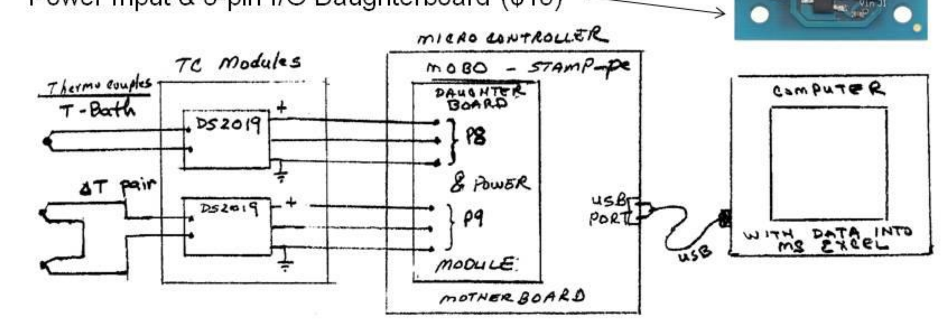
Flexible Data Logging based on Basic Stamp Microprocessor

From Parallax Inc., http://www.parallax.com/



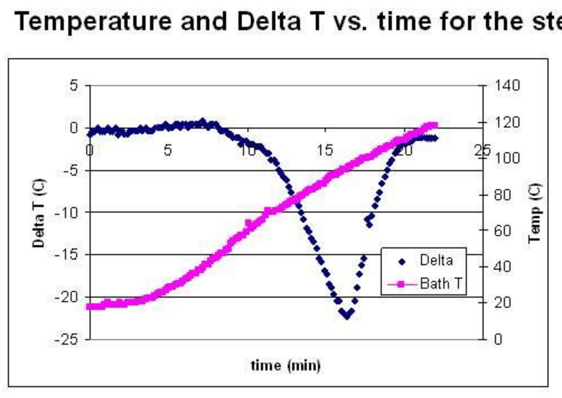
DS2760 Thermocouple Module ($39) comes with TC wire

Basic Stamp 2pe Motherboard ($70) top and right Power Input & 3-pin I/O Daughterboard ($15)



## Stearic Acid – reference standard

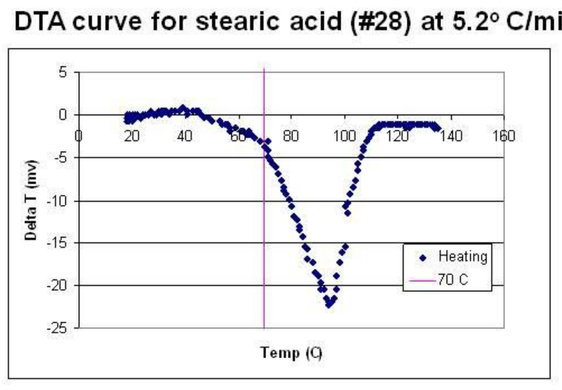
**Temperature and Delta T vs. time for the stearic acid**



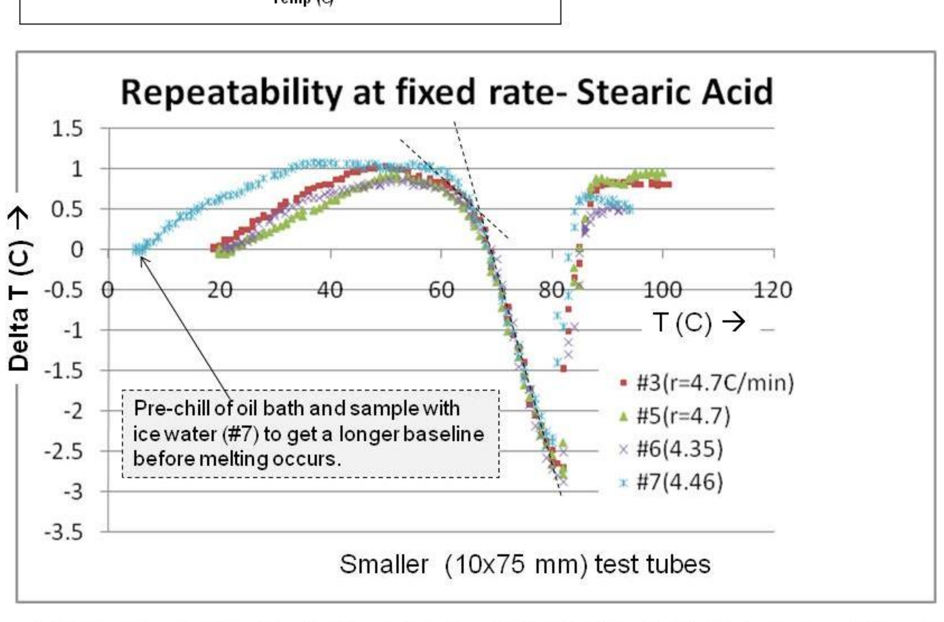

Note that the heating rate is fairly constant over most of the scan. Measured heating rate = 5.2° C/min.

Using 13x100 mm test tubes & technical grade stearic acid (MP reported as 69-71° C)

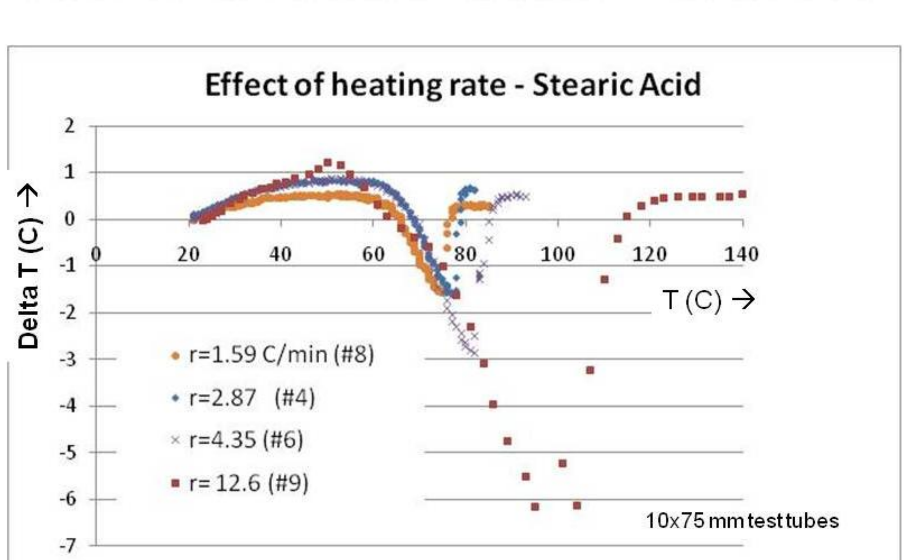
**DTA curve for stearic acid (#28) at 5.2° C/min.**




The MP is signaled as a kink in the DTA, which occurs here at 70° (purple line).

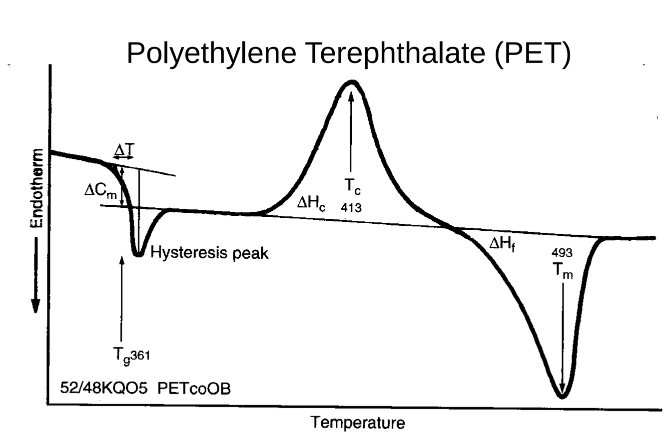



Excellent repeatability for the stearic acid melt standard. Note some difference from trace for same standard in the slightly larger test tube (#28 above).

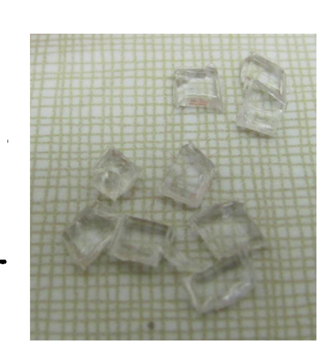


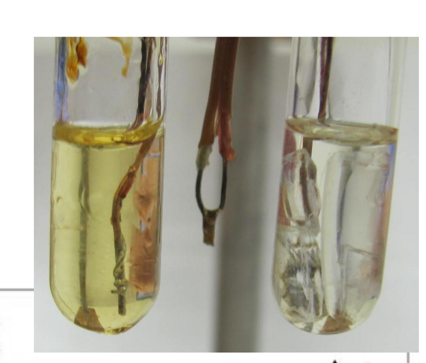

Width of melting endotherm increases with heating rate, as expected, while the onset of crystallization is much less dependent. The width for the 4.4° C/min. rate has approximately the same width as commercial DSC at 10° C/min. with a mass 100 times smaller. A rate of 2-3° C/min. appears optimal.

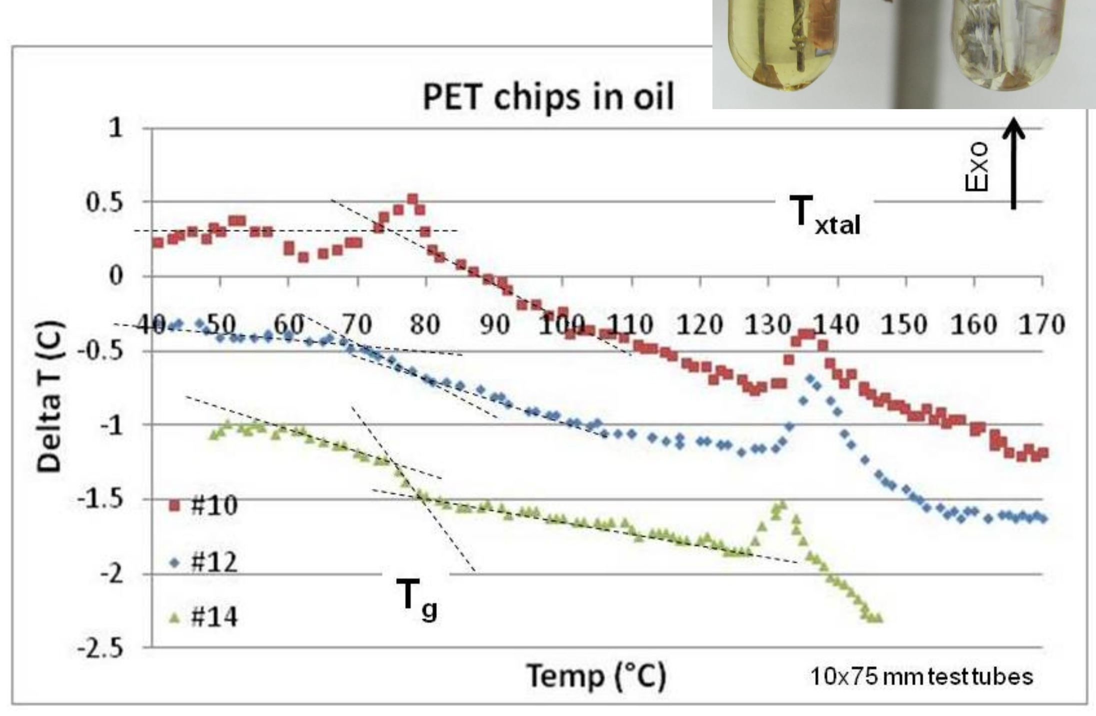
## PET – glass with crystallization




Sperling, Introduction to Physical Polymer Science p 369



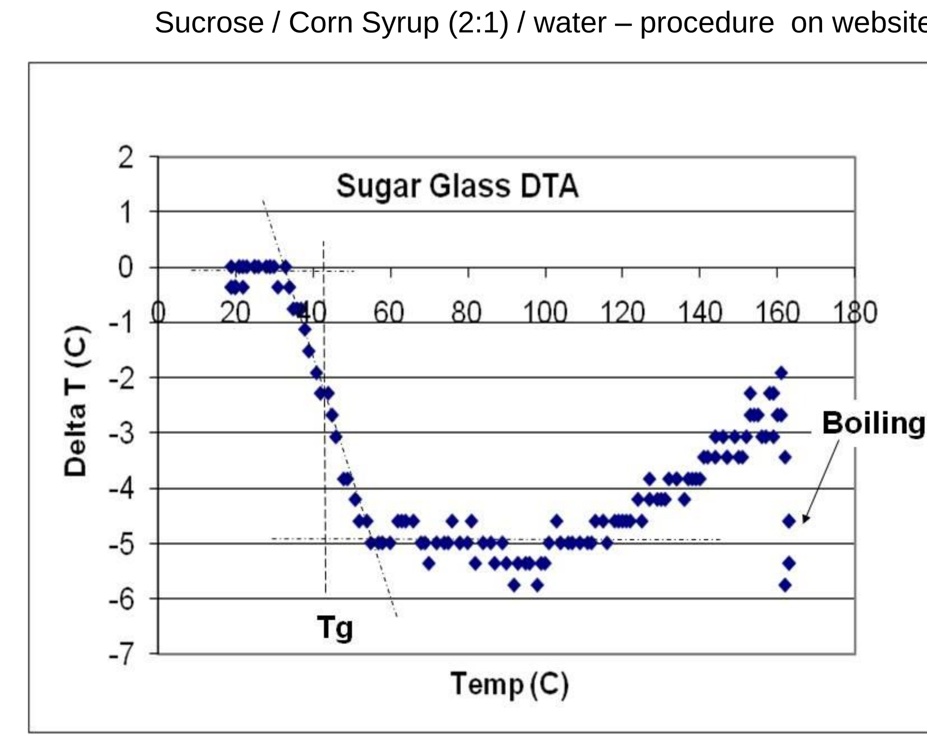


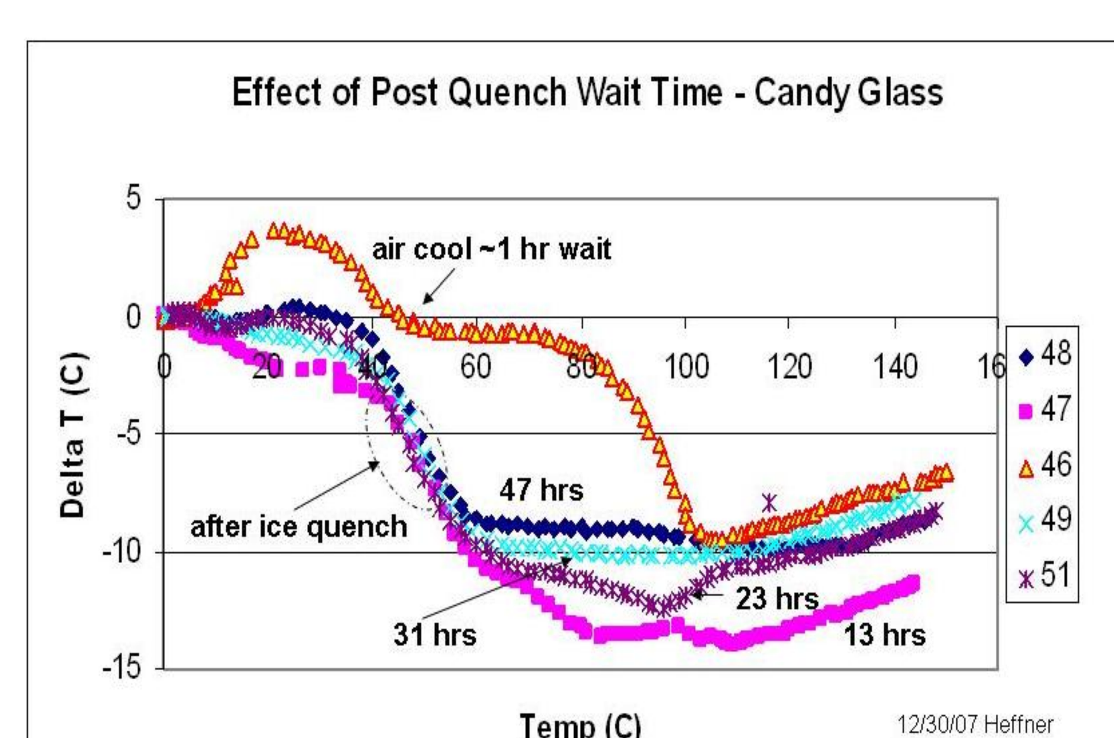



PET chips cut from the top of a Nestle water bottle with oil to provide thermal contact. Note clear signal of $T_g$ near the 73° value from DSC as well as the crystallization exotherm near 135° C. Scans at ~ 10° C/min.

## Sugar Glass – student made candy

Sucrose / Corn Syrup (2:1) / water – procedure on website




**Accomplishments :**

The DTA provides a relatively simple means for students to experimentally encounter thermal analysis, and in particular the glass transition and recrystallization phenomena. Stearic acid provides a low cost and low temperature melt standard and good repeatability is achieved. Candy glass and PET both provide accessible, low temperature $T_g$ materials appropriate for laboratory study with this apparatus.

**Issues and Future Plans:**

The materials with lower $T_g$ are also especially sensitive to thermal history and short term aging as they are essentially still annealing at room temperature. These effects, while quite interesting, also pose a challenge to reproducibility, unless careful attention is paid to all details of the sample conditioning. More work is needed to develop additional experiments to isolate and quantify some of the observed effects of thermal history and aging.

For additional details and future updates please see our education page at: www.lehigh.edu/GlassEducation.htm

LEHIGH UNIVERSITY

www.lehigh.edu/imi

**Acknowledgement:** This work has been supported by IMI-NFG, Lehigh University through National Science Foundations (NSF) Grants : DMR-0409588 and DMR-0844014