International Materials Institute for New Functionality in Glass

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A low-cost electrometer for measuring conductivity and glass transition in sugar glass

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Objective: As part of a series of low-cost experiments for students to explore glass science, we have developed a simple student-built electrometer for measuring electrical conductivity in glass and determine the glass transition (T_g). We also developed two different conductivity cells appropriate for expedient measurement of T_g in simple sugar glass system from both heating and cooling profiles. The approach complements our earlier described DTA[1] and would be suitable for an undergraduate material science laboratory. This will be added to our other sugar glass experiments on our website at http://www.lehigh.edu/imi/.

Candy Glass –Synthesized in the Kitchen Lab

Sucrose, Corn Syrup (2:1 wt) and Water (5%) are combined and cooked-
- first to dissolve into a single liquid phase &
- then to remove most of the water.

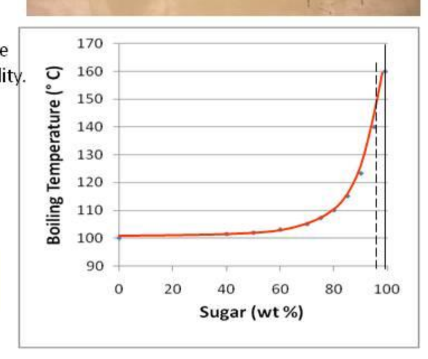
Boiling temp provides measure of the water content.
Boil to ~ 155° C. (< 2% water remaining)

Note:
2:1 sucrose to corn syrup (by wt.) to avoid crystallization problems

T_g of this candy glass is nominally about 40° C, but will depend on moisture and can change with humidity.

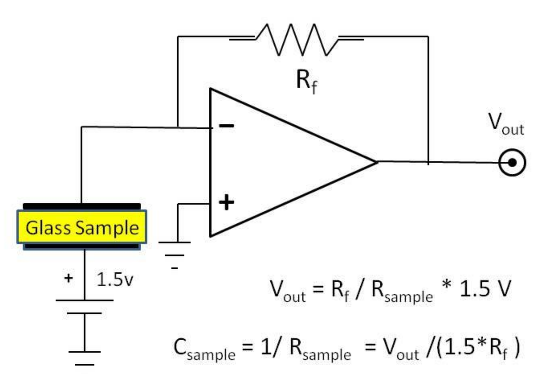
Corn syrup is a mixture of glucose, maltose and longer chain glucose molecules.

Cost ~ $5 in materials for many batches

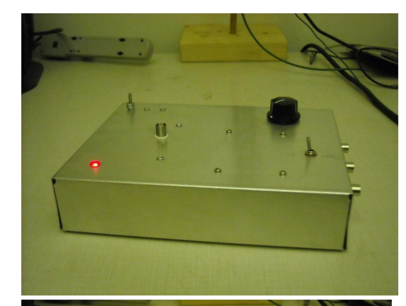


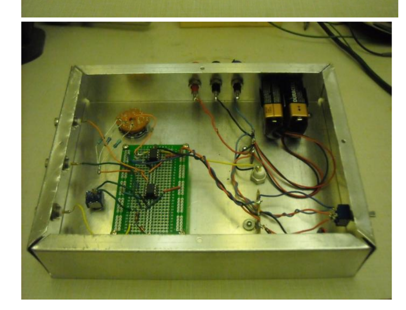
Data from Food Industries Manual, 24th ed, (1997).

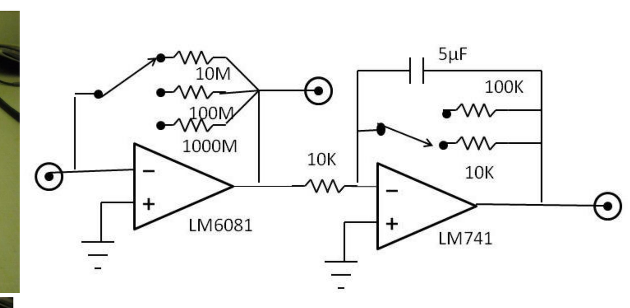
The DC Conductivity Measurement Approach



Low-Cost Student-Built Electrometer for DC Conductivity





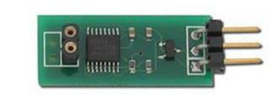


Input stage:
LMC6081 - Precision CMOS Operational Amplifi
- High Impedance input > 10 Tera Ω
- Ultra low input bias current: 10 fA
- Low offset voltage: 150 µV
- Cost < $5 in 8pin DIP package (DigiKey)

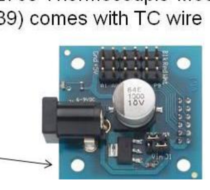
2nd Stage
- Isolation, filtering and additional gain

0.05 sec time constant for 60 Hz suppression

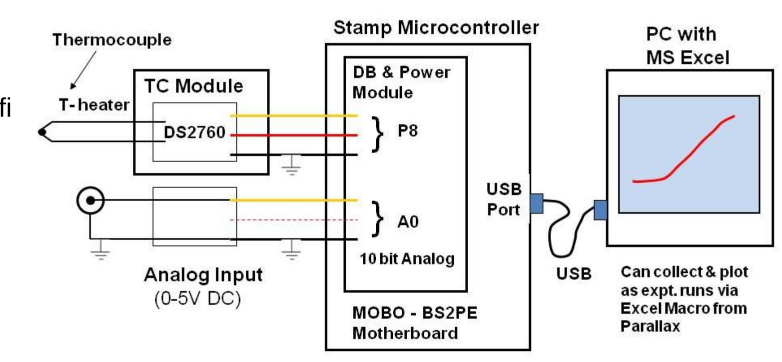
Low Cost Data Collection with Basic Stamp Microcontroller



DS2760 Thermocouple Module ($39) comes with TC wire



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

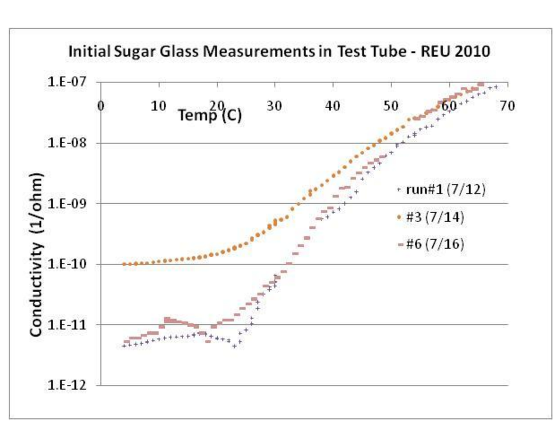


Our conductivity measurement involves monitoring the current flowing through the candy glass sample under an applied DC field (1.5 V in our case) over a range in temperatures. Because of the very high resistance in the glassy state an electrometer is required, which is essentially a very sensitive current amplifier, capable of measuring in the sub pico amp range. The student familiar with basic op amp circuitry should be able to understand and build the low cost electrometer circuit shown above. The total cost of the electrometer with chassis, connectors and all parts is well under $100.
Note: We are aware that DC conductivity measurements can have issues with polarization layer. However, this does not seem to be an issue in our case.

In keeping with the low-cost, student-built theme of our program, we also assemble our own data logger based on the Parallax Basic Stamp for under $150 in parts. The Stamp is programmed to send the data directly into an Excel spreadsheet for real-time plotting of results.

Simple Conductivity Probe & Initial Results from REU

Simple probe made from two Cu tabs soldered to leads running through two glass rods, epoxied to maintain spacing.
Probe is inserted into test tube containing molten sugar glass. Test tube assembly is then immersed in an oil bath heated on a hot plate.

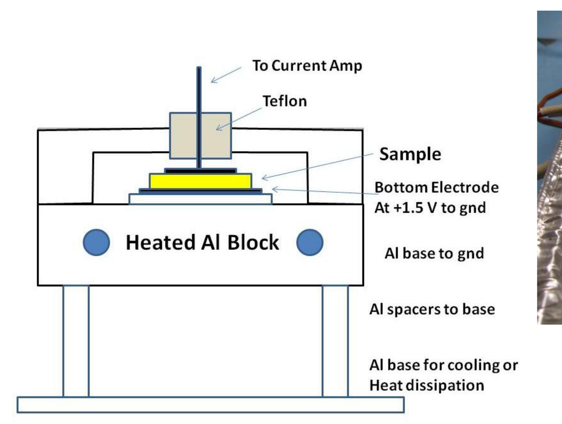


Cell Constant (Area/spacing) ≈ 0.715 cm

Electrical Conductivity in simple cell shows:
- distinct inflection indicating T_g (29° via DSC)
- data varies with run and wiggles unexpected

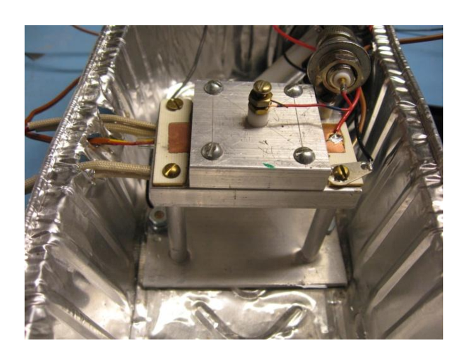
Some Issues with simple glass probe :
- epoxy softens after multiple uses, requiring repair
- limited control of heating rates
- cooling difficult , especially sub ambient
- desire smaller sample volume and
- greater ease of sample change

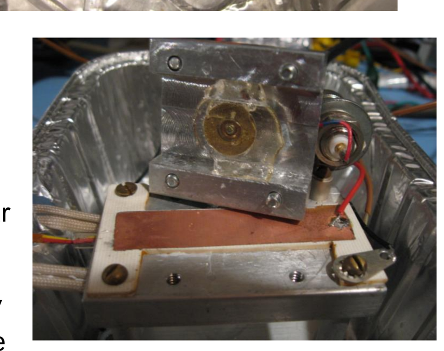
Improved Conductivity Cell – with Embedded Heaters and Cooling Option



Cell Constant (Area/spacing) ≈ 12.5 cm

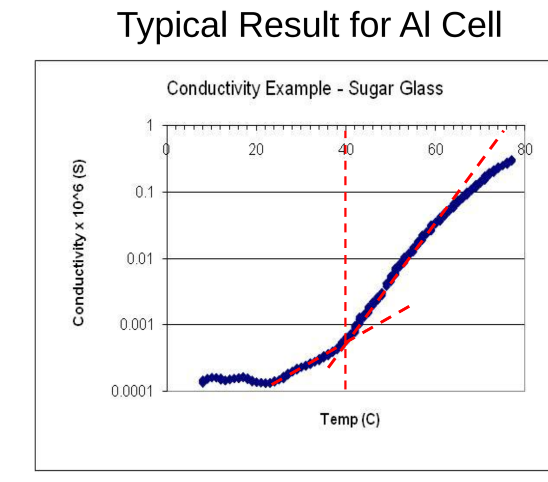
- Cell made from 3/8" Al bar stock with 25 W tubular heaters
- Sample surrounded by ground for shielding
- Cu clad PC board makes lower electrode at +1.5V
- Cooling accomplished by placing ice on lower plate





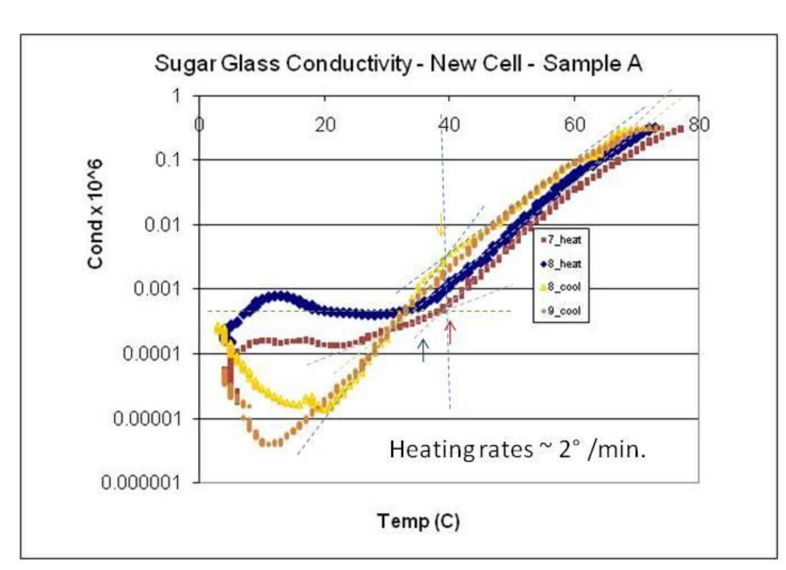
During measurement the conductivity cell is completely covered by an aluminum enclosure to provide shielding against 60 Hz and other electrical noise. Here a pair of Al bread pans are used for the bottom and top of the enclosure, both are grounded.

Typical Result for Al Cell

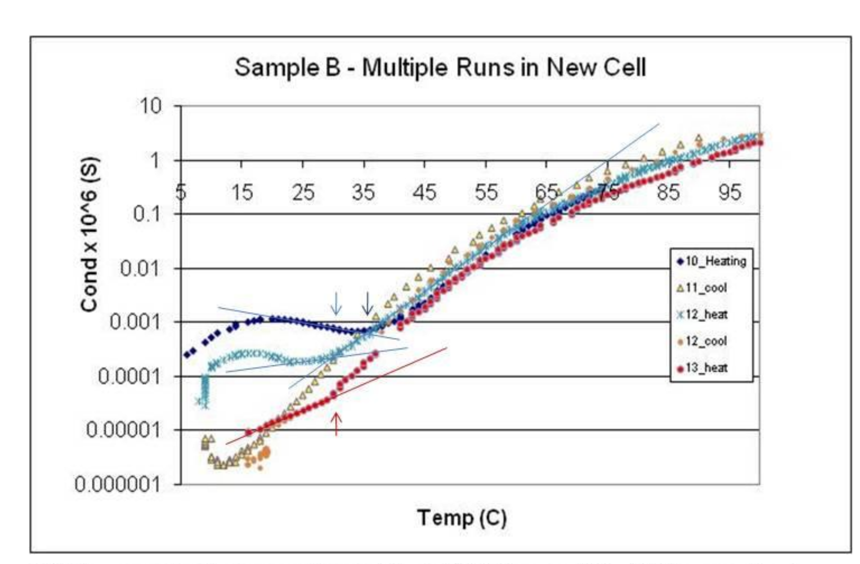


Distinct kink observed on heating near the Tg

Repeatability and effect of cooling history – Samples A & B



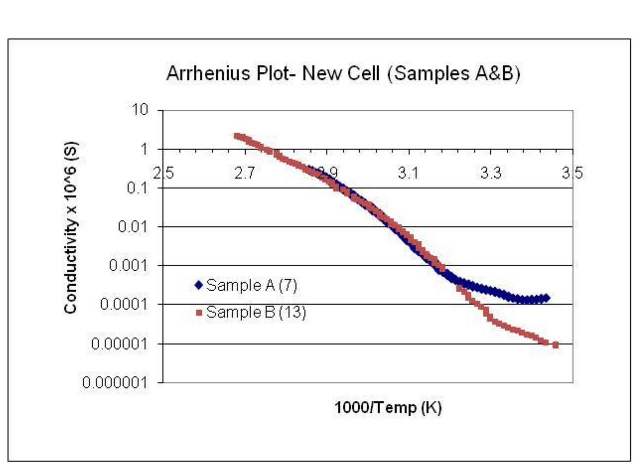
On heating: Distinct kink observed near 40 C on heating (35-40C)
flat region at low temp depends on cooling history
On cooling : Slight kink near 40C, from lower to higher slope
At low temp (< 15°C) conductivity reverses and increases
Behavior at low temp is unstable, always increasing with time



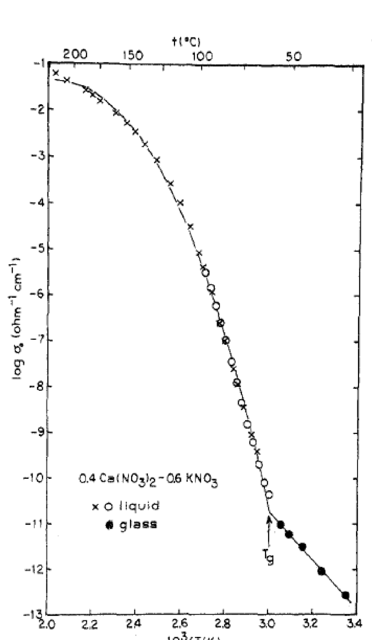
DSC for sample B gives midpoint T_g at 35°C (range 32°–40°) in excellent agreement with range observed in conductivity kinks.
T_g and conductivity of sugar glass are sensitive to moisture content and humidity.
By avoiding cooling below 17 C, unstable low temperature behavior is avoided (#13)

The two different samples above show similar behavior although the T_g of A is a few degrees higher that that of B, due to different moisture content. Both show distinct kink at T_g on heating, but the actual conductivity in the lower temperature regime is very sensitive to the preheat history. The cooling curves shed particular light on the apparent instability at lower temperatures, associated with a gradual increase in the conductivity with time starting below about 17º C. The best results are obtained when we avoid cooling below sub-ambient temperatures.

Comparison with Literature



"Dogleg" curve for both samples (A&B) of candy glass as plotted vs 1/T appear very similar to the classical result for CKN glass as shown on right. For CKN this kink was also attributed to the T_g.



F. S. Howell, R. A. Bose, P. B. Macedo, C. T. Moynihan J. Phys. Chem., 1974, 78 (6), pp 639–648

Accomplishments :
We have designed a low cost amplifier capable of exploring electrical conductivity in the 10^{-12} Siemens (1/ohm) range . The electrometer and associated conductivity cell are quite sufficient for exploring DC conductivity above and below the glass transition in the sugar glass model system. We find a distinct kink is observed in the conductivity vs. temperature curves, which is associated with the glass transition observed in DSC. The method would be appropriate for an undergraduate lab in material science for the student to explore both conductivity in glasses and the glass transition. It could also be extended to study conductivity in other glasses, even at higher temperatures with minor changes to cell design. Photoconductivity in chalcogenides would be another reasonable application.

As follow-up REU we plan to:
- Improve our understanding of the low temperature instability and
- Compare the data with theoretical and universality as a gateway for student learning

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

Reference:
1. W. Heffner, "A Low-Cost Student Built DTA for Exploring the Glass Transition", 2010 GOMD, Corning, NY
Also on IMI website at http://www.lehigh.edu/imi/.

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