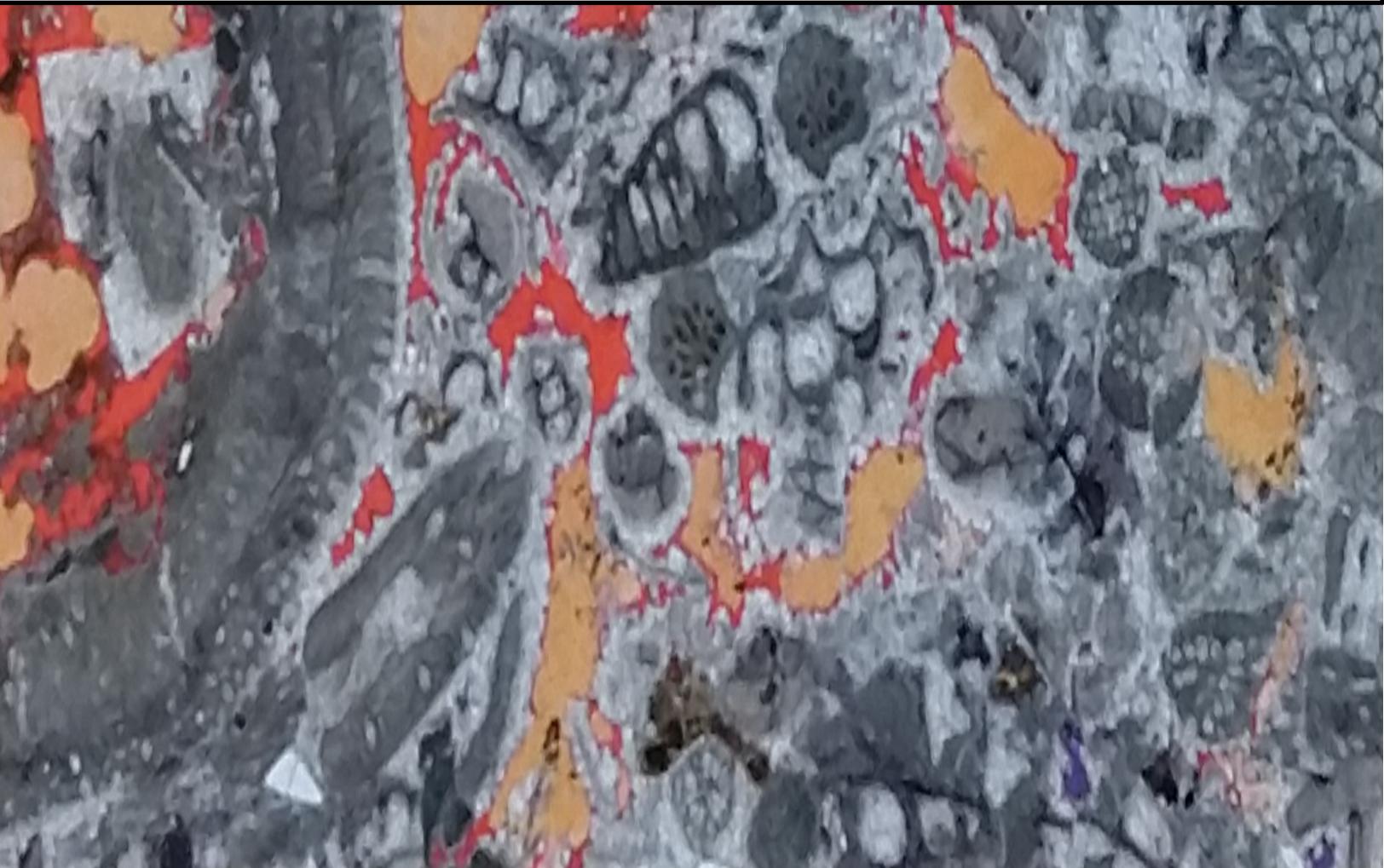


# **MATE 474 Lab Manual**

**Materials & Metallurgical Engineering Dept.  
New Mexico Tech, Socorro, NM 87124**





# **MATE 474 Lab Manual**

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## Table of Contents

828/DEA Batching Manual.....	1
IsoMet Buehler Saw Manual.....	14
RMS 800 Annealing Manual.....	21
Bluehill Instron Compression Manual.....	29
Differential Scanning Calorimetry (DSC) Manual.....	49
ARES Rheometer Testing Manual.....	66
KaleidaGraph Plotting and Data Analysis Manual.....	78
Instron Compression Test Plotting.....	79
DSC Test Plotting.....	94



# 828/DEA Batching Manual

## Introduction

Getting accurate, reliable data on epoxy starts with creating well-made epoxy samples. Epoxy has two primary components: a resin, and a hardener. Other additives may be included in order to optimize certain material characteristics. This manual details the batching instructions to make an 828 resin, DEA hardener epoxy mixture.

## Safety Information

- Never work with chemicals without at least one partner. Working unassisted and unmonitored can result in disaster in the event of injury or incapacitation. Make sure that one or more qualified lab workers are available to help you batch before you begin.
- Personal protection equipment is required. Please consult the “Materials” section for specifics.
- Long pants and closed-toed shoes should be worn. Long hair should be worn up. Gloves should be changed in the event of an epoxy spill.
- Uncured epoxy resins will cause skin sensitivities to develop if contact is experienced on a regular basis. If possible, it should be handled in a hood. After the epoxy mixture has cured, the material is non-reactive and safe to be handled without gloves or safety glasses.
- Particular care must be taken with additives such as DBA, which is both corrosive and flammable. Carefully read all additives’ MSDS sheets before batching.

## Procedure

This manual contains the following sections:

- A. Before Starting (2 steps)
- B. Preparation (13 steps)

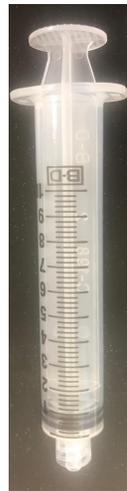
- C. Mixing Chemicals (6 steps)
- D. Degassing (6 steps)
- E. Pouring and Cure (7 steps)
- F. Cleanup (4 steps)
- G. Recovering Cylinders (8 steps)

### **Materials**

- Personal Protection Equipment: Safety glasses (stored in 130B), a lab coat (also stored in 130B), and nitrile/latex gloves (stored in upper cabinet on window side of 130-Main).



*Personal protective equipment*



*10mL syringe*

- Chemicals: Diethanolamine (DEA), 828 Epon Epoxy-Resin, additives if necessary
- Bunsen Burner and lighter
- 8 10mL syringes
- A rack (a plastic container with holes cut in it works)

- Top loading balance
- Waste disposal container
- 3 Popsicle sticks
- Labeling Tape
- 3 Pipettes
- Batching Sheet
- Acetone

**Batching:**

Before Starting:

1. Allow for an hour beforehand to heat the 828 Epon Epoxy-Resin (EPON 828) and Diethanolamine (DEA) in an oven set to 70°C. This reduces viscosity and makes mixing easier. As the chemicals and prepared mixture come to room temperature the consistency will become more viscous.
2. Fill out a batching sheet as shown below.

828 DBA + Octanol

828/DEA Batching Sheet		Date
Batch No.		9/22
		Made by: EH KH
Materials Used		Mass (g)
Use mix ratio of 100:12 (828:DEA) by weight		
Resin: Epon 828	50 g 828	13.35 g Octanol
Hardener: Diethanolamine		7.37 g DBA
Procedure		
<input checked="" type="checkbox"/>	Pre-heat resin and hardener at 70C for at least one hour prior to use. **Prepare the DSC pans and cylinder molds that will be used for this batch.	
<input checked="" type="checkbox"/>	Weigh out the appropriate masses of resin and hardener.	
<input checked="" type="checkbox"/>	Mix hardener and resin thoroughly until the mixture becomes white.	
<input checked="" type="checkbox"/>	Place the mixture in the oven at 70°C for 5 minutes.	
<input checked="" type="checkbox"/>	After 5 minutes check to see if the epoxy mixture has cleared	
<input checked="" type="checkbox"/>	— If not, place back into the oven and check in 30 second intervals - removing and stirring until the mixture becomes clear.	
<input checked="" type="checkbox"/>	Make samples	
DSC Pans		Compression Cylinders
**prior to batching, pans must be weighed out and sample envelopes labeled		Degas batch at 1-3 torr for a maximum of 3 minutes.
Place 1 drop of mixed epoxy in the prepared pan with a pipette, ensuring it doesn't exceed the pan top.		Carefully pour epoxy into the cylinders, angling them such that the epoxy can slide smoothly from the cup to the cylinders.
Crimp the lid onto the pan.		
Test the samples immediately or store them in the freezer for testing at a later date.		Place samples into the oven to cure at 70°C for 24 hours.
Samples Made:		
		8 cylinders
Clean up work space and ensure all materials used (gloves, stir sticks, etc.) are also cured at 70°C for 24 hours.		
Notes: <u>Very clear</u> after a few minutes in oven <u>Very easy</u> to pour		

*Batching sheet for 828-DBA with Octanol*

Preparation:

I. Preparing syringes to hold epoxy samples.

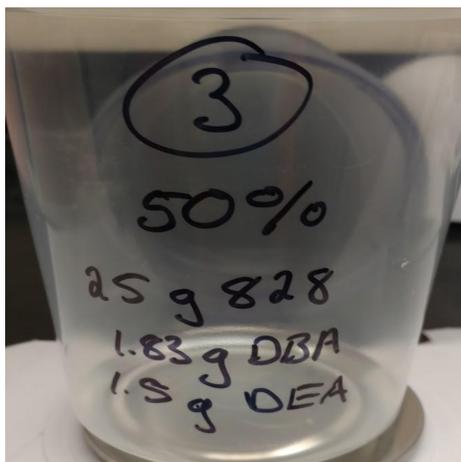
1. Unwrap 10mL syringes.
2. Unplug all syringes. Save only one plunger- the rest may be disposed of.

3. Set up the syringe rack next to the workspace.
4. Turn on and light Bunsen burner.
5. Carefully melt the needle adapter of the syringe (the tip where liquid would be pushed out) by holding it over the flame. Let gravity help you seal the opening by rotating the syringe on its side. Take caution during this step: you should not ignite the plastic, just heat it enough to liquify it. **Note: The open flame must be used away from the chemicals used for batching – Particularly DBA which is flammable.**
6. Place the freshly sealed syringe tip-down in the rack to cool.
7. Repeat steps 5 and 6 until all syringes have been sealed.
8. Turn off the Bunsen Burner and put it away.
9. Allow each syringe to cool for several minutes.
10. Using the plunger you saved earlier, attempt to push air through the cooled syringes. If properly sealed, the plunger should resist the motion, and should slide backwards once released. If the syringe does not pass this test, repeat steps 5 and 6.

## II. Set up mixing workspace.

11. Prepare workspace inside the fume hood by laying out paper towels around the balance and plugging the balance into the wall outlet. Check the bubble level to make sure the balance is level, and adjust as necessary. The rack holding the sealed syringes should be nearby and readily available.
12. Take out popsicle sticks and pipettes. Label each for what chemical they will be handling with colored electrical tape, using a consistent labeling system (DBA:blue, DEA:green, 828: White).

13. Label a plastic container with the proportions of your batch. **Note: All materials that come in contact with the epoxy/hardener mixture must be cured in the oven for 24 hours before going into the trash (after this period, the resin is fully cured and is no longer hazardous)**



*Plastic epoxy mixing container, labeled with batch proportions.*

Mixing Chemicals:

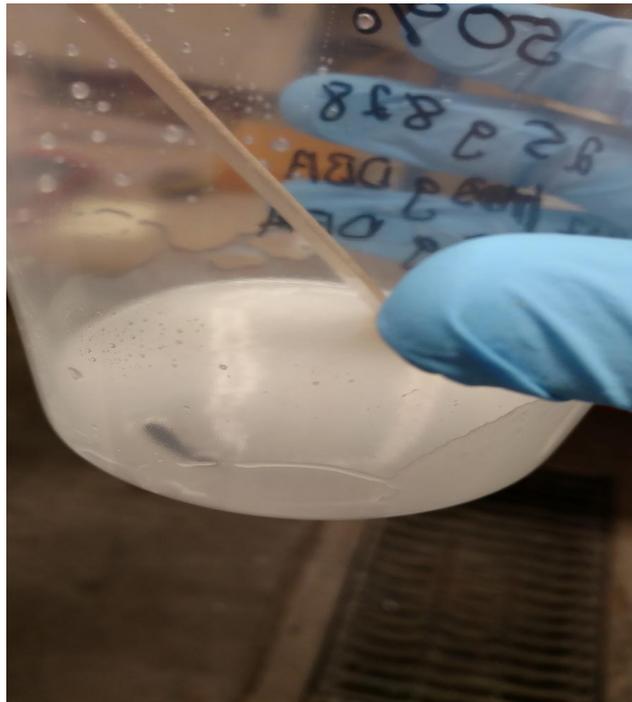
1. Begin by positioning your plastic container on the balance in the fume hood. Tare by pressing the “zero” button.



*Tared Scale*

2. Take the 828 out of the oven and carefully pour it into the plastic container. As you get closer to your desired figure, use the designated 828 pipette to add the resin at a more controlled rate. Try your best to get within 0.01g of the target mass. Put the 828 back in the oven when you are done with it.
3. Tare the scale again, and add the appropriate amount of DEA using its designated pipette. Again, try to get within 0.01g of the target mass. Put the DEA back in the oven when you are done using it. **Note: From here you will be handling DBA. DBA should not be inhaled as it can produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. (DBA has a fishy odor).**
4. Tare the scale and add the appropriate amount of DBA using the designated DBA pipette. Try to get within 0.01g of the target mass. Carefully seal and store the DBA bottle when you are finished using it.

5. Set a timer for five minutes. Carefully stir the epoxy mixture with a popsicle stick for the duration of this period. Keep the container under the fume hood at all times while stirring, and point the opening away from you to minimize exposure to fumes. The mixture should turn from clear to opaque after five minutes of stirring.
6. Place the mixture in the 70°C oven for five minutes. Take out and stir, checking to see if the mixture has reverted from opaque to clear; if not, place in the oven for another five minutes. Repeat this process until mixture is clear.



*This mixture, still slightly opaque, could use more time in the oven.*

#### Degassing:

1. Place the mixture in the pressure chamber, and close the chamber door securely. Turn on the pump using the switch located on the lower right side of the pressure chamber.



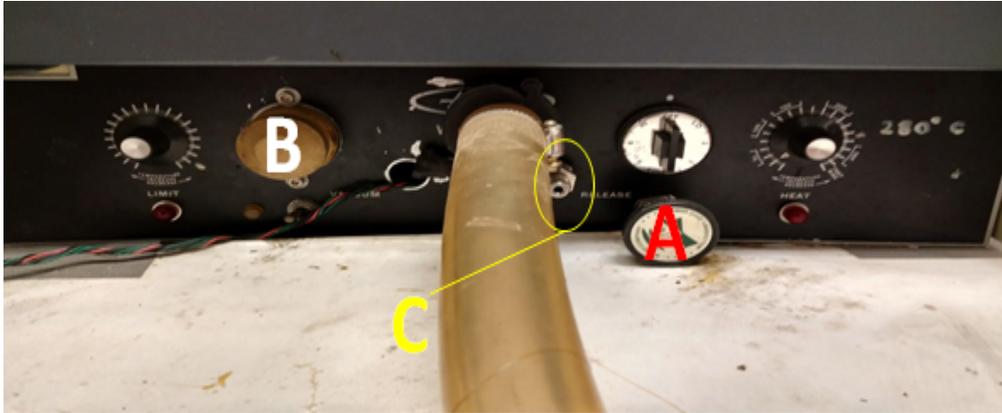
*Pump switch on side of pressure chamber*

2. Next, turn on the power strip to the left of the pressure chamber. It should be beneath the neighboring oven.



*The power strip in the off position*

3. Turn the release valve (A) (see picture below) to the right as tight as possible. If the valve is not sealed, air will enter the chamber and you will hear a whistling sound.



*The vacuum chamber's different mechanisms*

4. Switch on the vacuum toggle (C). Begin to turn the pressure valve (B) to the left. This will start dropping the pressure. Unlike the release valve, you generally do not want to turn this knob to the tightest possible level.
5. Once a satisfactory pressure has been attained in the chamber, set a timer for five minutes. Keep an eye out for overboiling in your mixture: miniscule, quickly rising bubbles are quite normal, but larger bubbles are a bad sign. Feel free to adjust the pressure in the chamber as needed using the two valve knobs.
6. If after five minutes the mixture seems adequately degassed (that is, if the bubbling has significantly slowed), you may equalize the pressure in the chamber. Simultaneously open the release valve and close the pressure valve, and wait until the chamber reaches equilibrium to open the vacuum door.

#### Pouring and Cure:

1. Place your degassed mixture along with your sealed syringes+syringe rack under the fume hood.
2. Bend the lip of the mixture container into a spout shape.

3. Hold the opening of the syringe to the makeshift spout so that the sides of the two containers are near parallel. The quality of this configuration is what will determine how much air is incorporated back into the mixture during the pouring process, so line this up carefully. **Note: The goal of degassing is to remove air from the samples and decrease inconsistencies. Thus it is of utmost importance that while pouring the degassed epoxy into our molds, no air is re-incorporated into the mixture.**
4. Slowly pour the mixture down the side of the syringe. Fill the syringe up to at most a centimeter from the top. Overfilling the syringe can make the epoxy removal process difficult, while underfilling can lead to wasted epoxy when it comes time to cut samples for testing.
5. Carefully place the filled syringe back onto the rack.
6. Repeat steps 3-5 until you run out of epoxy. Seal and cool extra syringes if needed; no epoxy should go to waste.
7. Place the rack of filled syringes inside the oven. Cure for the desired time. **Note: The cure time is usually in the magnitude of several hours, but should nonetheless be handled with precision; as such, make sure that either you or another qualified lab assistant will be there to take out the epoxy cylinders exactly when specified.**

### Cleanup

1. Any trash that has come in contact with any of the chemicals should be stored in the mixing container and cured in the 70°C oven for twenty-four hours. It is only after this curing period that the trash can be disposed of.



*Trash in the curing oven*

2. Wipe your work surfaces with acetone and paper towels.



*Acetone and paper towels*

3. Put your lab coat and safety glasses back in 130B.
4. Wash your hands for at least a minute to ensure no chemicals linger on your skin.

### Recovering Cylinders:

1. Suit up with proper PPE. Safety glasses, thick heat gloves (located in the drawers of 130C), and closed-toe shoes are a must for this step. Wearing a lab coat is recommended.
2. Lay out all necessary tools for this process- hammer, pliers, ziploc baggies, and sharpie. The first two can be found in the 130C toolbox, and the last two on the counter in 130B.
3. Using the heat gloves, take the rack of cured epoxy cylinders out of the oven.
4. While still wearing heat gloves (which will protect you from both the heat of the epoxy and the potential impact of the hammer) begin hammering at the sealed tip of a syringe with firm but controlled taps. The objective is not to crush the syringe tip, but rather to knock the epoxy cylinder down the length of the syringe. Aim to get the cylinder down at least a centimeter before proceeding with the pliers. **Note: Hammering the syringes can send sharp plastic debris into the air. Be sure to wear your safety glasses to prevent injury.**
5. Using the pliers, pinch the newly empty tip of the syringe in order to push the epoxy cylinder further out. Continue with this pinching action down the length of the syringe until a good portion of the epoxy protrudes from the syringe, at which point you can simply use your hands to completely pull the cylinder out.
6. Repeat steps 4 and 5 until all epoxy cylinders have been extracted.
7. Store the cylinders in baggies labeled with the material, the appropriate batching number, and (if necessary) the cure schedule.
8. Throw away the syringe husks and sweep up the plastic debris from the hammering step. Put your tools and PPE away.

# IsoMet Buehler Saw Manual

## Introduction

In order to perform tests on the Instron or DSC, samples must be cut from the epoxy cylinders on the IsoMet Buehler saw in 130B. This manual details how to safely and precisely operate this piece of equipment for this purpose.



*The IsoMet Buehler saw*

## **Safety Information**

- Personal protection equipment is required. Consult the ‘Materials’ section for specifics.
- Keep your hands and fingers away from the saw while it is running. Avoid flailing movements.
- The glass of the fume hood can serve as a valuable barrier while the saw runs, and may be lowered down as far as is practical during cutting or sharpening (keep in mind that relatively easy access to the buttons of the saw must be maintained).
- Long hair should be worn up, and loose, hanging sleeves rolled back.

## **Procedure**

This manual contains the following sections:

- A. Preparation (2 steps)
- B. Loading, Measuring, and Cutting (6 steps)
- C. Cleanup (2 steps)

## **Materials**

- Personal Protection Equipment: Safety glasses (stored in 130B)
- Cured epoxy cylinders
- Allen wrench
- Sharpie or other marker
- Digital Calipers
- Aluminum oxide block
- Sandwich bags
- Paper Towels

## **Cutting Epoxy Samples:**

### **Preparation:**

1. Fill the bath beneath the saw blade about two-thirds full with water- enough so that a decent piece of the blade is covered by the water when the bath is in the ready position.

Once this is completed, the bath can be jiggled back up into the ready position and propped by the aluminum block.



*The water bath beneath the saw*

2. The blade must be sharpened next. During cutting, the blade is slowly run through the white, pressed aluminum oxide blocks on the front end of the saw. The sharpening blocks can be moved by the attached knobs. Run the blade about halfway through the block and as close to the other cut marks as possible to preserve the life of the sharpening block. The blade must be sharpened every three cuts, and the water in the bath must be changed every other sharpening to keep particle debris to a minimum.

Loading, Measuring, and Cutting:

1. To mount the epoxy cylinders, use the blue nut driver or an appropriately sized allen wrench to loosen the screws on the arm above the saw blade. Loosen them just enough to slide the cylinder into the arm, then tighten them enough so that the cylinder is stable. Make sure that the end with the spout is facing out, as removing that end will be the first cut.



*Saw arm with sample loading mechanism*



*Loaded cylinder*

2. Lower the cylinder onto the (still off!) saw blade. Make any necessary final adjustments to the position of the cylinder using the knob behind the saw. The initial spout cut should be as small as possible in order to maximize the amount of remaining usable epoxy.
3. Ensure that the speed is set to the seven position before starting. Start the saw with the black start button.



*The control panel of the saw*

4. Stop the saw using the red “stop” button as soon as any cut is completed. The samples have a tendency of falling into the bath once severed, and can jam the saw if it continues to run. Recover any of these fallen samples from the bath after stopping the saw.
5. Once its spout has been removed, the cylinder can be cut into samples. There are two types of samples one could create:

*Instron Sample:* This sample should be exactly 1 inch long. Use a digital caliper and a sharpie to mark the cylinder at this length. Draw your mark across the full circumference of the cylinder. Do not mark several sample lengths on a single cylinder at one time; measurements should be made only from the site of a fresh cut to ensure maximum accuracy.

*DSC Sample:* This sample should be only a few millimeters thick- about the thickness of a nickel. This measurement can be visually approximated; the use of a caliper and/or a sharpie is unnecessary. Keep in mind that, in this case, it is best to err on the thicker side of a cut.

6. Repeat the process of loading the cylinder, positioning the cut, and running the saw. Don't forget to periodically sharpen the blade and change the bath water as specified.

Cleanup:

1. When you are done using the saw, clean up after yourself: dump the bath water, wipe down the counter, and lower the glass of the fume hood, leaving a bit of a gap in order to facilitate air circulation. Put your safety glasses back on the shelf by the door.
2. Samples should be stored in appropriately labeled bags separated by epoxy type, batch, and sample type. The scraps of the epoxy cylinders can be stored either with the usable samples, or in a separate labeled bag in a designated scrap container.

# RMS 800 Annealing Manual

## **Introduction**

Annealing samples allows for the aging history of the sample to be reset. By heating the samples and returning them to room temperature, residual stresses and strains can be reduced in the polymer, allowing for more accurate tests to be run. This manual will detail the process of annealing the cut Instron samples in the RMS 800, located in 130B.

## **Safety Information**

- This process involves bringing samples up to a high temperature. Avoid touching the machine while the test is running to prevent injury.
- This machine has moving parts. Avoid touching the motor while turning it on.
- When the test is started, stay to ensure that the RMS 800 chamber temperature does not climb more than a few dozen degrees above the set temperature. If temperature badly overshoots (50+ degrees F), turn oven switch off. Keep in mind that it is typical for the machine to *slightly* overheat and then cool down.

## **Procedure**

This manual contains the following sections:

- A. Turning on the Air (3 steps)
- B. Running the RMS 800 (23 steps)
- C. Shutting Down the RMS 800: (8 steps)

## **Materials**

- RMS 800
- 1-inch epoxy samples

## Annealing

### Turning on the Air:

1. Begin by turning on the Main Air, located on the west wall of room 130B. To do this, turn the red handle one-quarter turn to the left. Air pressure should read around 90 psi.



Figure 1: Main Air in the “Off” position. Notice that the air pressure is 0 psi.



Figure 2: Main Air in the “On” position. Notice that the air pressure is around 90 psi.

2. Connect the hose from the Hankison (located on the north wall of room 130B) to the blue RMS 800 hose.



Figure 3: The Hankison



Figure 4: The Hankison’s power switch and air pressure gauges.

3. Turn the Hankinson on. Air pressure should read around 80 psi.

Running the RMS 800:

1. Go to the RMS 800, located on the south wall in room 130B, and check the air pressures on the gauges located on the bottom half of the machine. The oven air and transducer air should read 40 psi and the motor air should read 60 psi.
2. Place the samples you wish to anneal in the hatch, making sure that the samples do not block the motor that is on the top of the hatch, or the thermocouples that are on each side of the hatch. You may stack the samples vertically.

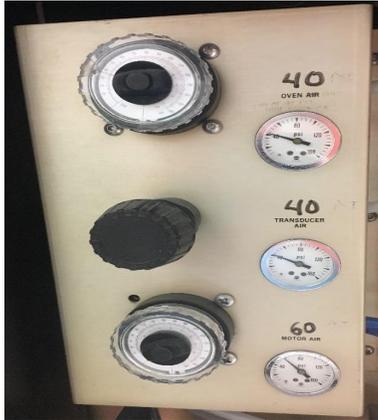


Figure 5: The pressure gauges on the RMS 800.

3. Close and latch the hatch.



Figure 6: Hatch of the RMS 800

4. Turn on the main power by pushing the Main Power button.



Figure 7: Power buttons on the RMS 800

5. Turn on the motor by pushing the Motor button.

6. Go to the computer and open the RSI Orchestrator.
7. Open the Utilities menu using the tab at the top of the program.
8. Click Service.
9. Click Check Instrument Address. The address should be “A00B”. If it is not, change it to be so and click Ok.

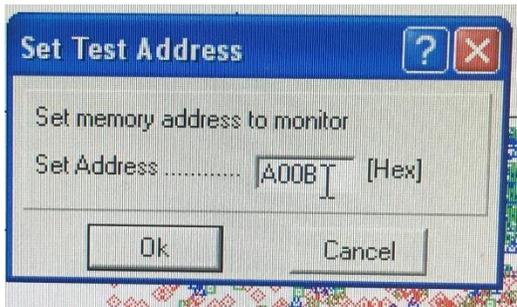


Figure 8: The Instrument Address Code

10. Open the Control menu.
11. Click Instrument Control Panel.
12. Set the Temperature to “25.0” and the Temperature Control to “Oven (Air, Chiller or LN2 Dewar.)” and click Ok.

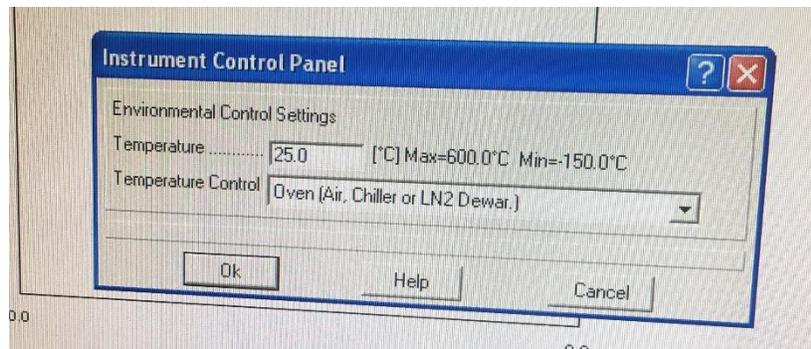


Figure 9: Instrument Control Panel set up

LN2 Dewar.)” and click Ok.

13. Click on the green start button. Clicking this button does not actually start the test, it just opens the editing menu.
14. Select the Torsion Rectangular Geometry from the Geometry drop down menu.
15. Select the Dynamic Temperature Ramp Test from the Test Setup drop down menu.
16. Click on the Edit Test button. If you are testing 828DEA, set every variable to be equal to the ones in Figure 8. If you are testing any other material, set “Initial Temp” to 30 degrees F above the material’s Tg, “Final Temp” to approximately 40 degrees F below the Tg, and all other variables to the ones in Figure 8.

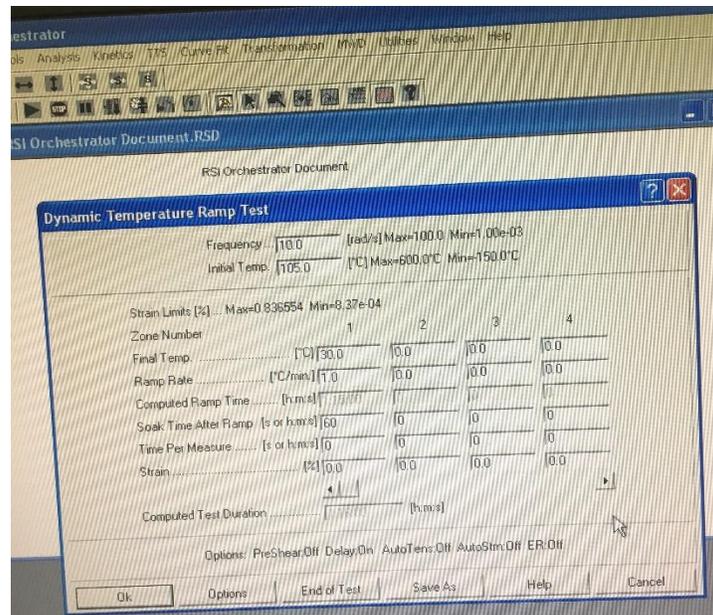


Figure 10: The Dynamic Temperature Ramp Test setup

17. Click on the Options button and select Delay Before Test.

18. Check the Delay Before Test box and type 1800 into the text box.

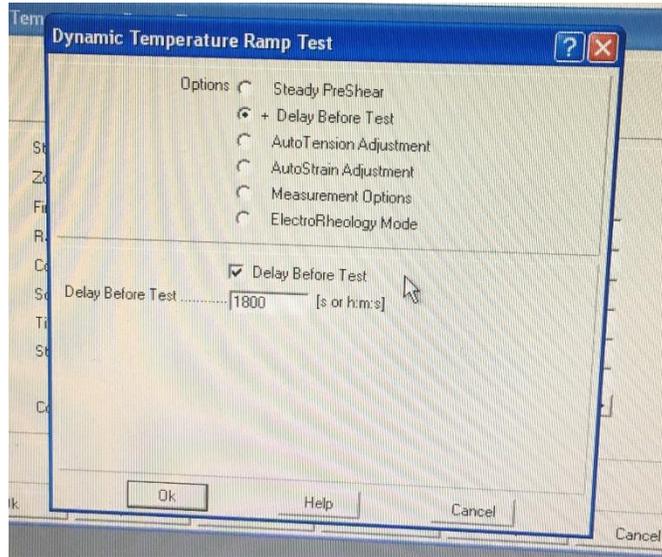
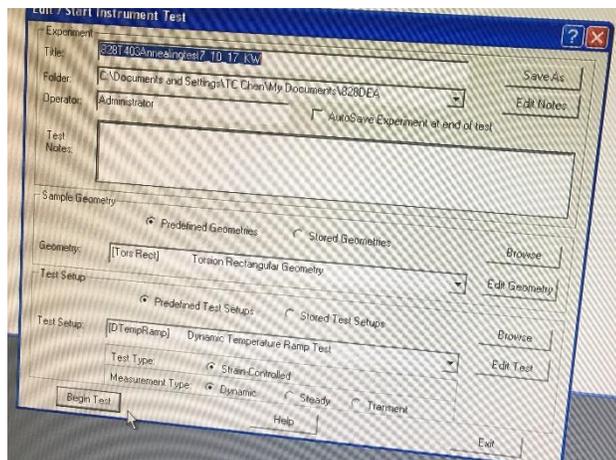


Figure 11: The Dynamic Temperature Ramp Test options menu setup.

19. Click Ok to exit the Options menu, and click Ok again to exit the Dynamic Temperature Ramp Test menu.

20. Return to the RMS 800 and turn the transducer on by pushing the Transducer On button.

21. Push the Oven On button.



22. Return to the computer and press the Begin Test button.

Figure 12: Overall setup of the test.

23. Wait until you have seen the temperature reach the set value and begin to decrease (See “Safety Information” section) before leaving the test to run its course.

Shutting Down the RMS 800:

1. Take the samples out of the oven as soon as the test is complete. If you leave the samples at room temperature for several hours, they will regain an aging history and will have to be annealed again.
2. Once the test is complete, go to the RMS 800 and push the Oven Off button.
3. Turn off the transducer by pushing the Transducer Off button.
4. Turn off the motor by pushing the Motor Off button.
5. Turn off the main power by pushing the Main Power Off button.
6. Open the hatch and remove your samples.
7. Go to the Hankison and switch the power off.
8. Go to the main air valve and close the value.

# Bluehill Instron Compression Manual

## **Introduction**

Subjecting a material to large amounts of stress can teach us a lot about that material's properties and merits. After annealing, 1-inch epoxy cylinder samples undergo compression tests with the Bluehill Instron, located in lab 177. This manual will detail how to perform a compression test using the Bluehill Instron Machine

## **Safety Information**

- Depending on the test being run, the oven and its contents may be hot.
- Large machines with moving parts are present. Keep hands, clothes, and hair away from these parts. The top compression speed of this machine is very slow, but caution should nonetheless be employed. The machine is equipped with an emergency stop button, as well as two pins placed on the frame that act as circuit breakers if they are contacted.



*Emergency off button and circuit breaking pins*

## **Procedure**

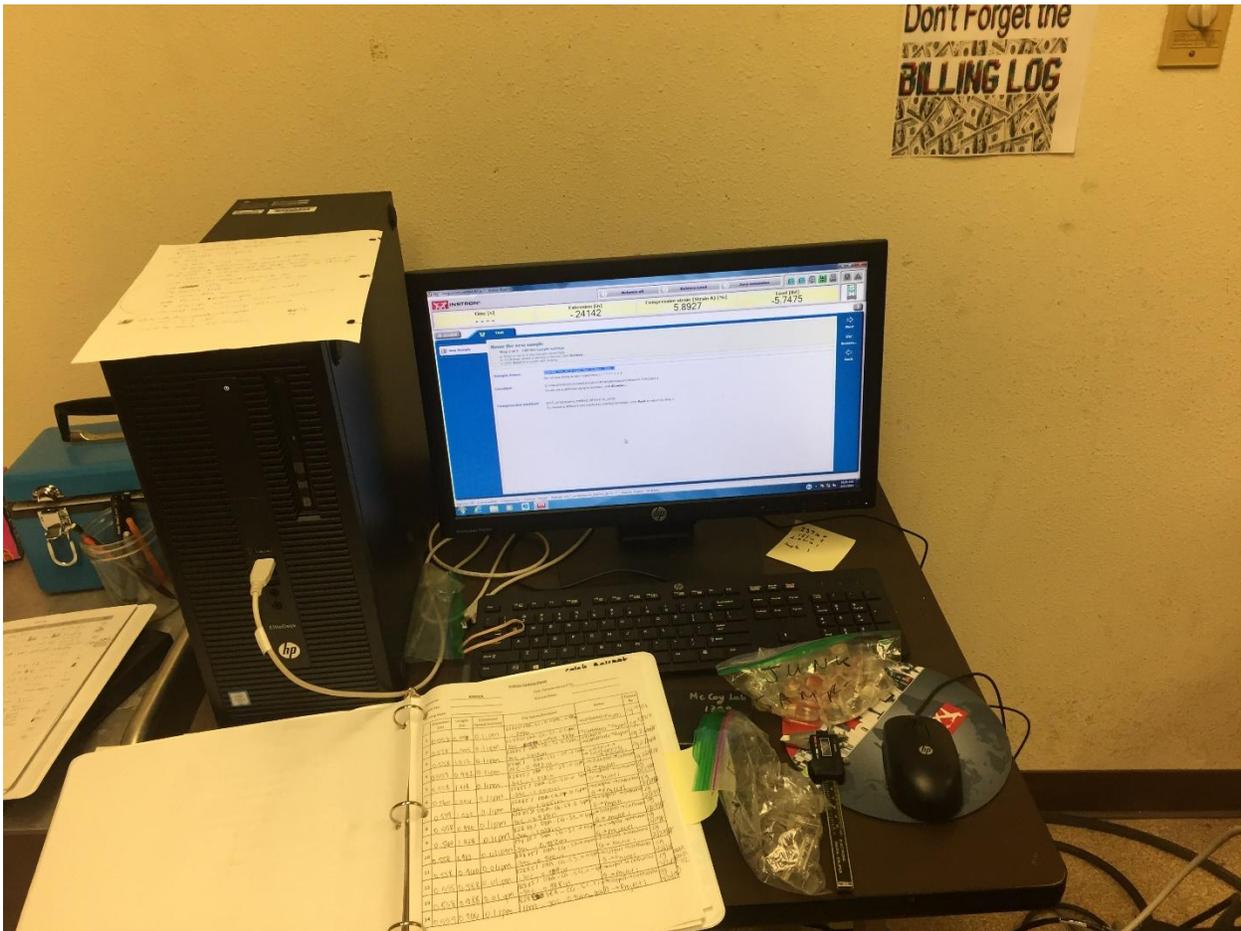
This manual contains the following sections:

- A. Sample Preparation (steps 1-2)
- B. Creating the File (steps 3-8)
- C. Programming the Test (steps 9-12)
- D. Running the Test (steps 13-18)

## E. Finishing the Test (steps 19-21)

### Materials

- 1-inch annealed epoxy cylinder samples
- Digital caliper
- Instron logbook



*Instron computer and logbook*

### Compression Test

#### Sample Preparation

1. Measure the height and diameter of the sample using the digital caliper. Record the values in the Instron logbook.

Serial: 828DEA Test Temperature (°C): 30  
 Sample No: 2590 Anneal Date: \_\_\_\_\_  
 Testing Date: \_\_\_\_\_

Diameter (in)	Length (in)	Crosshead Speed (in/min)	File Name/Location	Notes	Tested By
.562	0.985	0.1			CB
2.558	0.928	0.01			CB
<del>561</del>	<del>0.915</del>	<del>0.1</del>		24 hr	CB
558	1.008	.1 in		96 hr	CB
558	1.041	.1		336 hr	CB
62	.950	.1		72 hr	CB
562	.956	.1		199 hr	CB
60	.944	.1		48 hr	CB
1	1.016	.1		159 hr	CB
0	.947	.01		48 hr	CB
	.955	.01		336 hr	CB

*Instron logbook page*

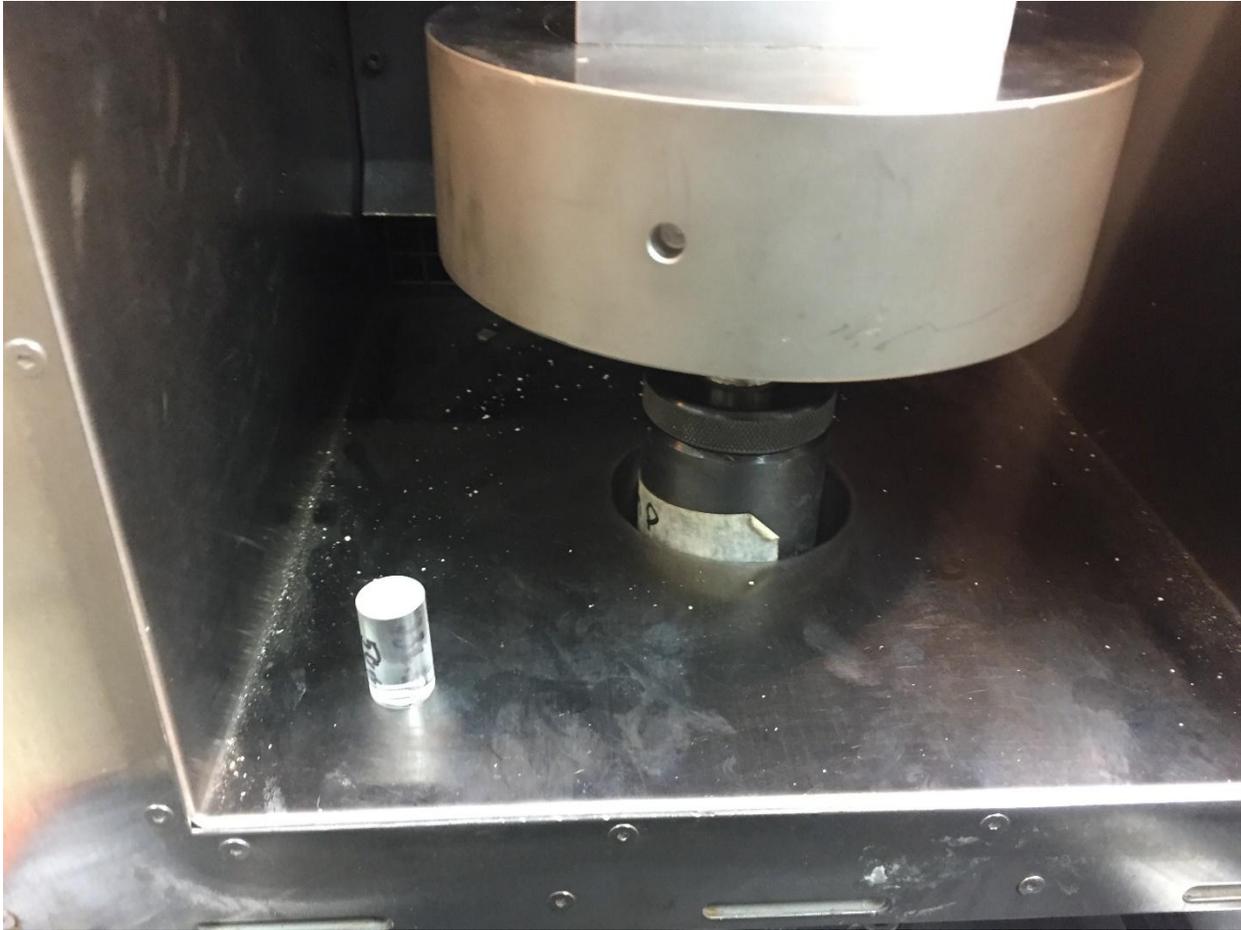
2. Allow the temperature of the sample to come to equilibrium with the Instron oven. To do this, place the sample inside the oven for at least thirty minutes before testing. Ensure that the door to the Instron is closed and sealed while the sample is equilibrating. **Turn off the fan using the fan switch anytime that the doors open.**



*Make sure to turn off the fan using this switch anytime that the doors open.*



*Press the “Light” button in order to illuminate the Instron chamber.*



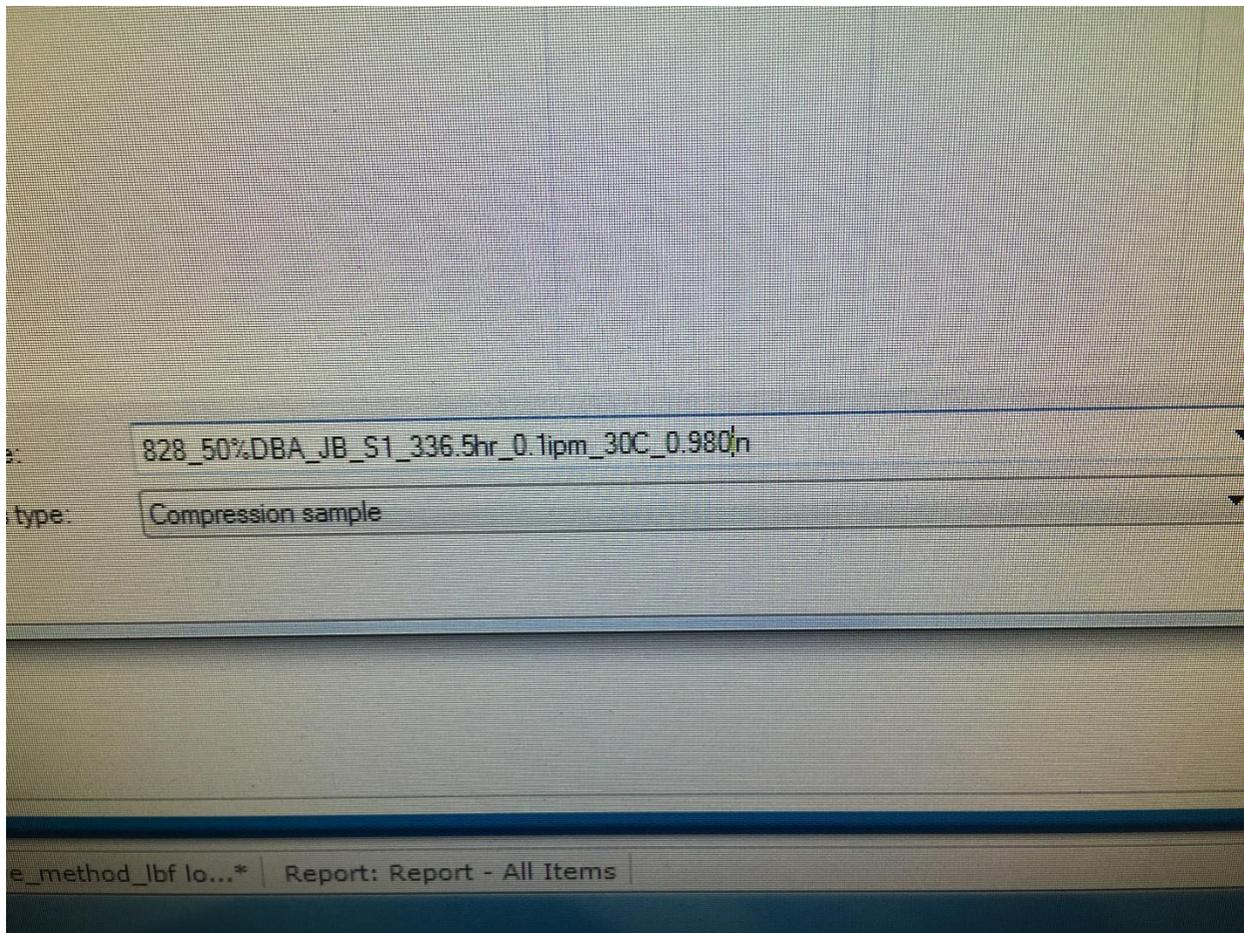
*Set the sample in the oven like so to be equilibrated.*

#### Creating the File

3. On the computer, open the Bluehill application by double clicking it.
4. Select the 2017 Compression program from the menu.
5. Name your file. In general, follow this format:

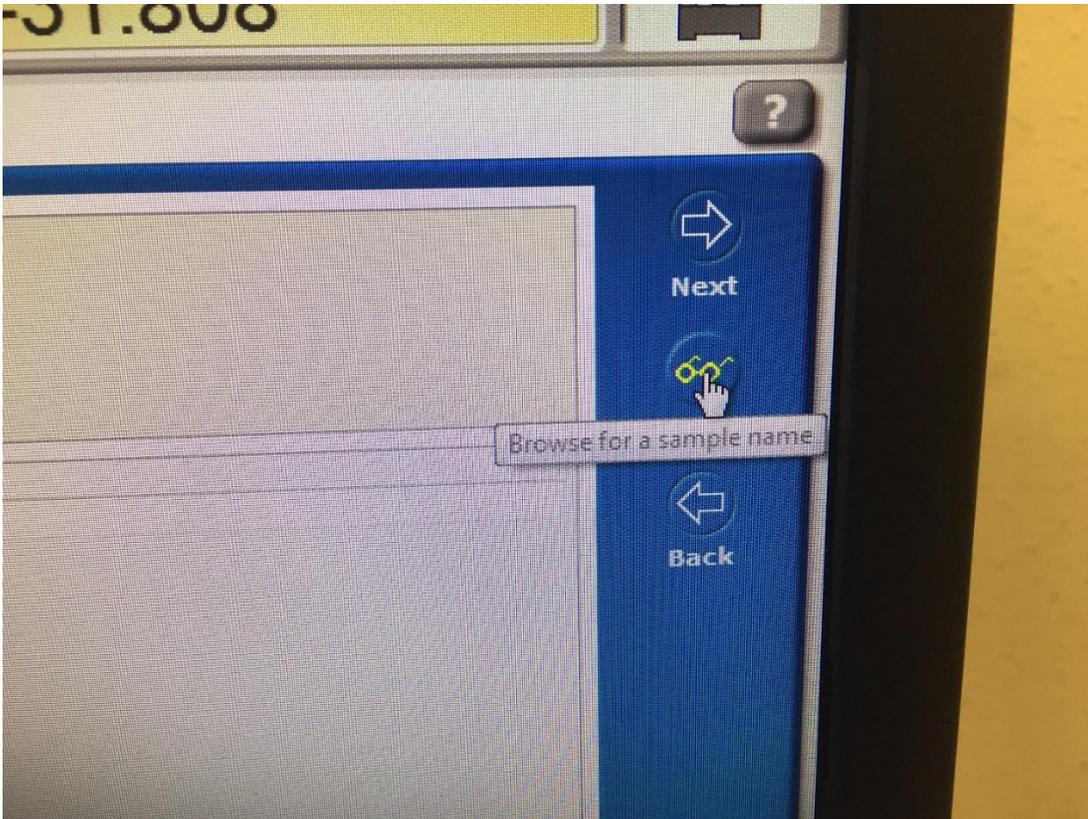
*[Material] [Batch Number] [Sample Number] [Crosshead Speed] [Length of Sample]*  
i.e.: *828DEA25%DBA\_B091716\_S2\_0.1ipm\_0.987in*

Other factors, such as oven temperature and sample aging history, may be included in the file if needed.



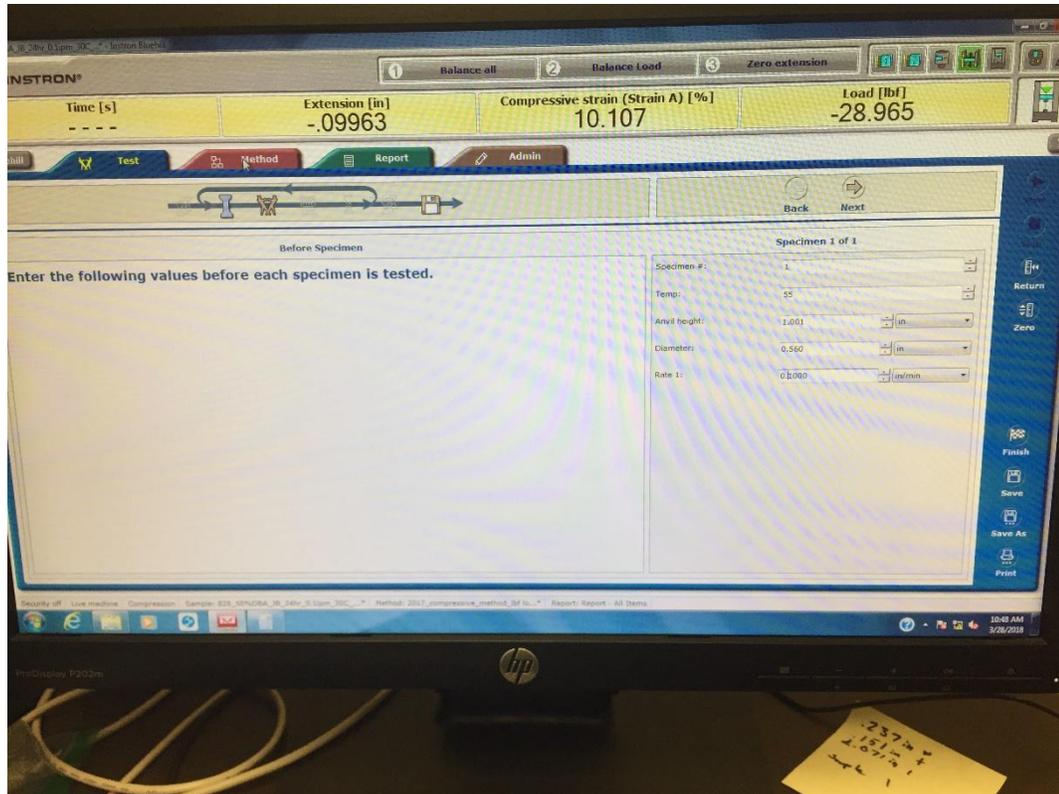
*Naming your file in Bluehill*

6. On the right hand side of the screen, click “Browse”.



*Toolbar on right side of screen, with functions “Next”, “Browse”, and “Back”*

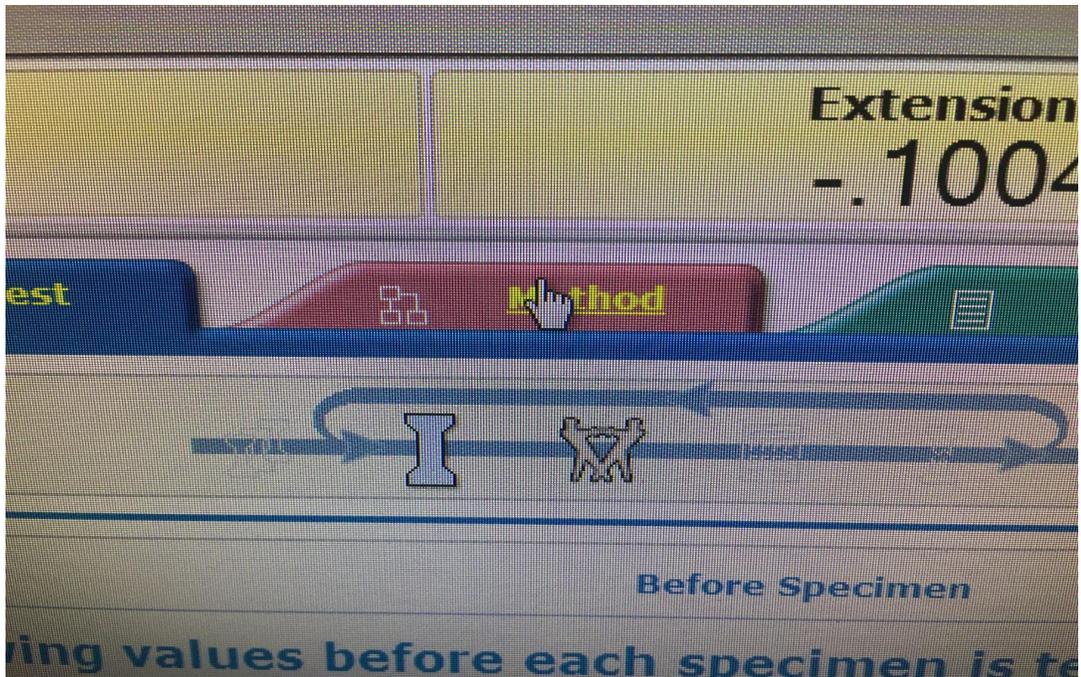
7. Choose your file destination. If necessary, create a folder or net of folders to properly archive your tests. Click “Next”.
  
8. Input the sample parameters on the right side of the screen: sample number, oven temperature, sample length (“anvil height”), sample diameter, and crosshead speed. Click “Next”.



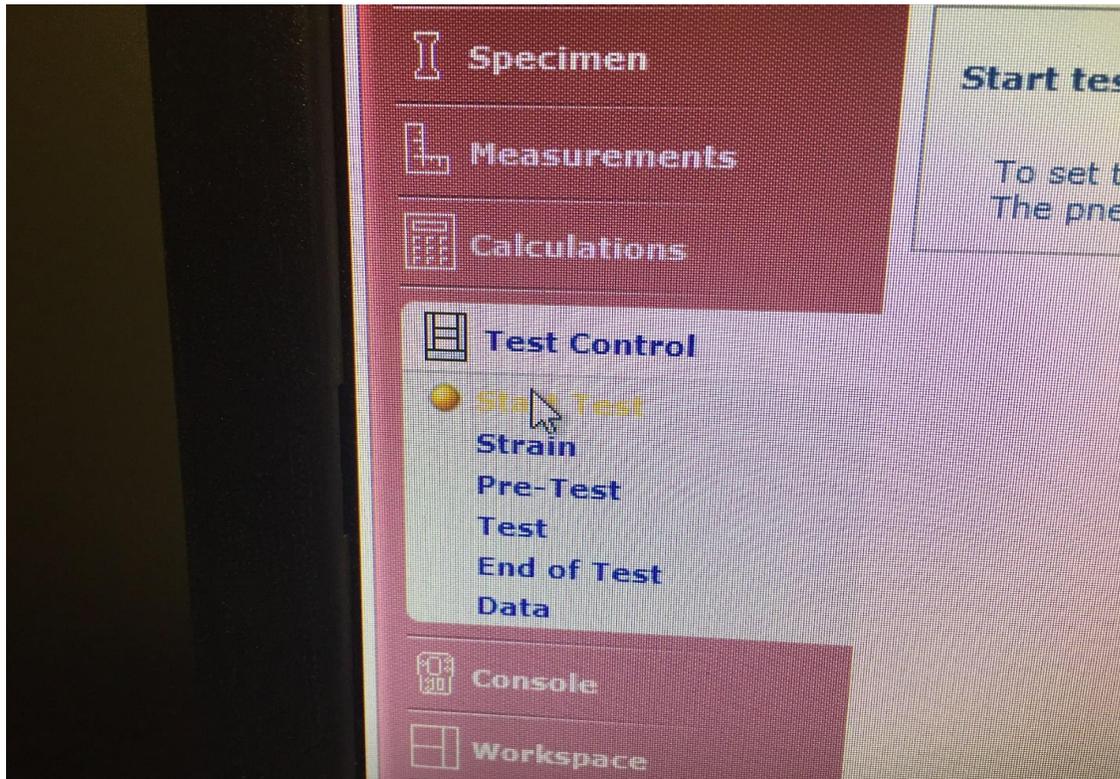
*Input sample and test parameters to the window on the right*

### Programming the Test

9. Click on the red tab near the top of the screen that reads “Method”. From there, click on “Test Control” and then “Pre-Test”. Make sure that none of the boxes are checked on this page.

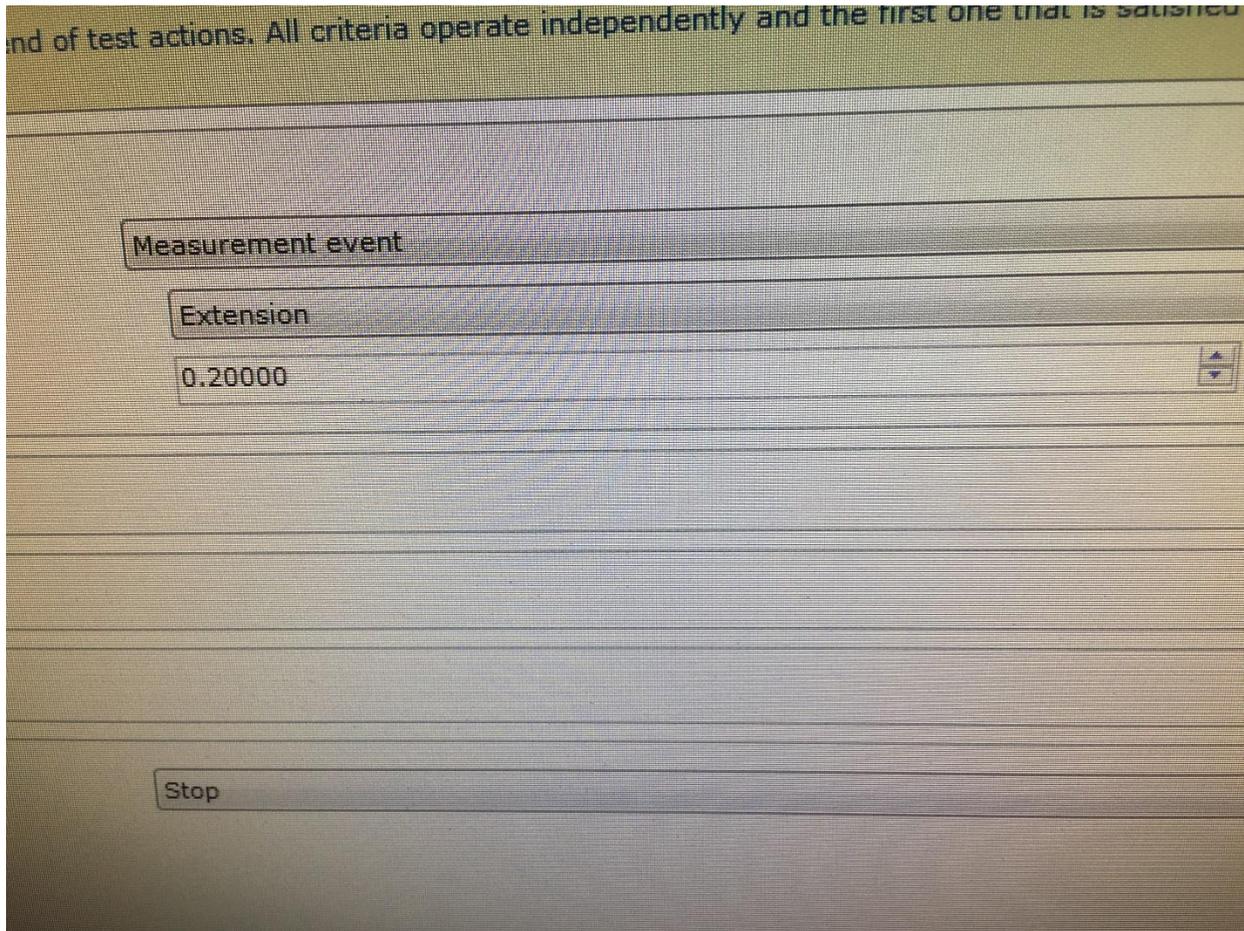


*The tab ribbon at the top of the test window, with tabs “Test”, “Method”, “Report” and “Admin”*



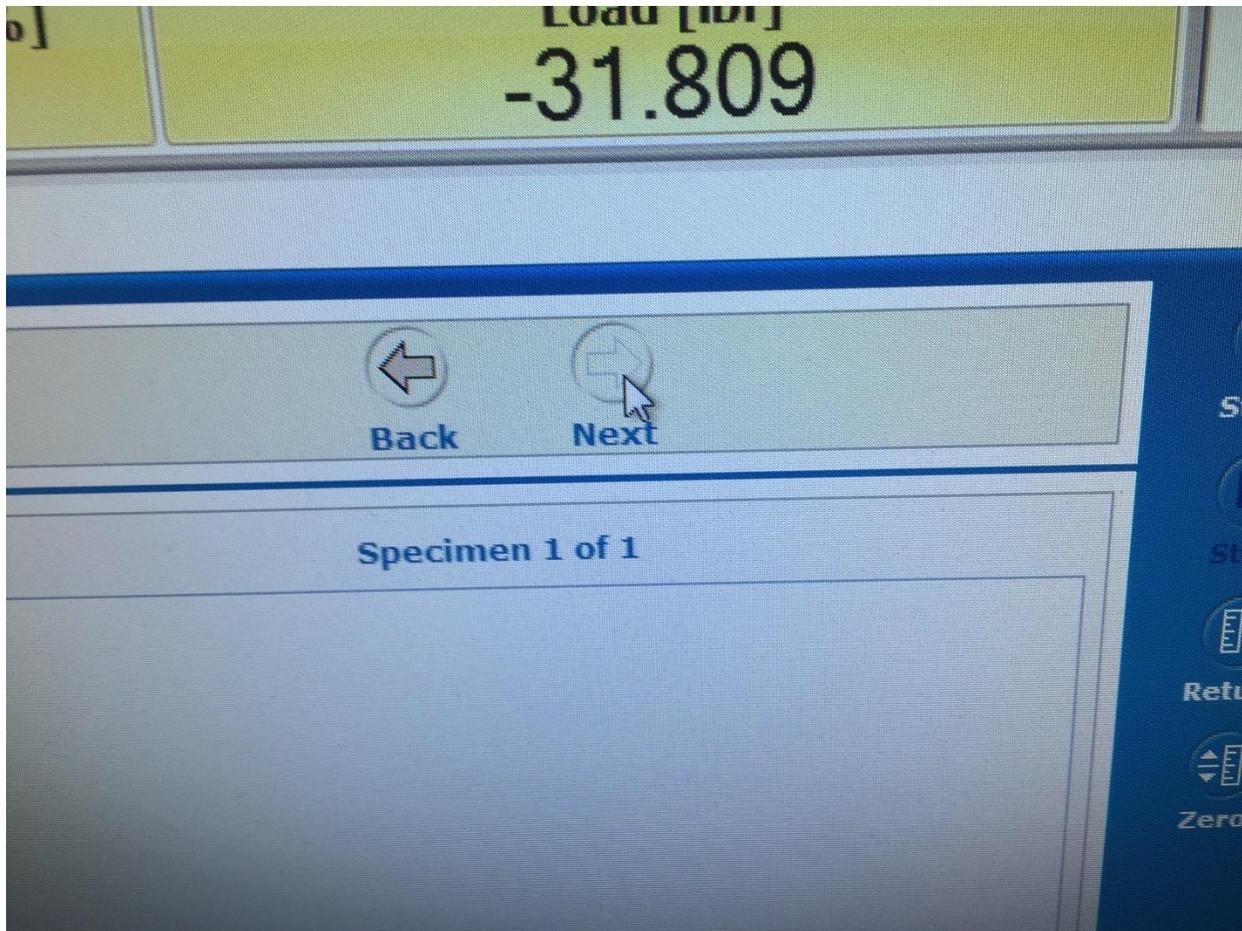
*The “Test Control” section under the “Test” tab*

10. Under “Test Control”, click into “Test” and input your desired crosshead speed.
11. Under “Test Control”, click into “End of Test” and input your desired extension. (Most tests have an extension of 0.3 inches.)



*“End of Test” window*

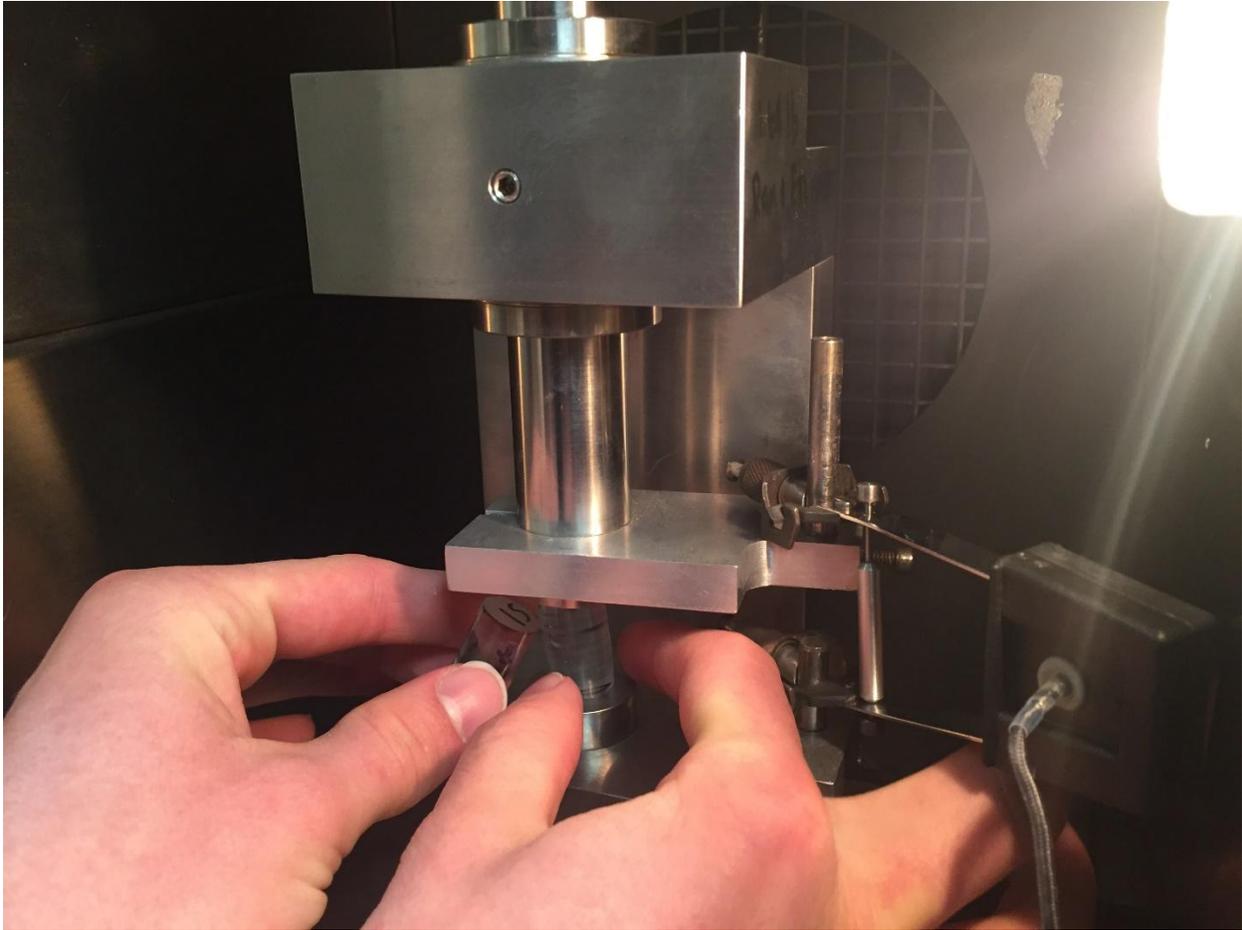
12. Back at the top of the screen, click on the blue “Test” tab, and then the “Next” button.



*The “Next” and “Back” buttons to the upper right of the “Test” tab*

### Running the Test

13. Open the Instron doors, and replace the placeholder sample in the compression mechanism with the sample you wish to test. Leaving the machine without a sample beneath it is hard on its workings, so make the exchange quickly and smoothly. **Be sure to turn the fan off when the doors are open and to turn it back on once the door is closed.**



*Carefully switch the epoxy samples, jostling the module as little as possible.*

14. Once the sample is loaded and the oven door latched closed, begin lowering the load frame using the “jog down” button on the control panel. Stop once the large compression plate is a centimeter or two away from the cap of the sample module.

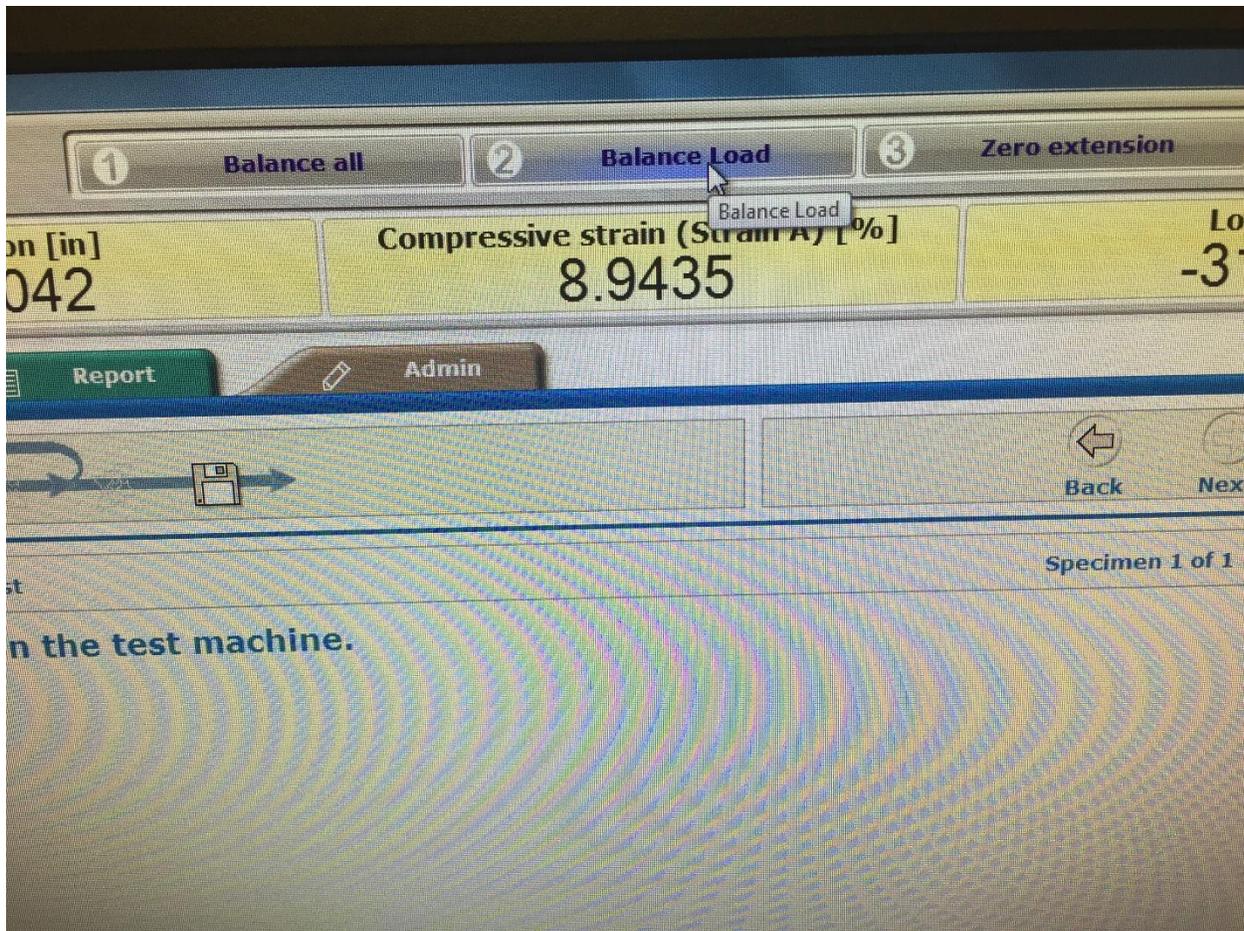


*Instron Control Panel with “Jog Up” /“Jog Down” buttons and a “Fine Position” knob.*



*Hold the “Jog Down” button until the large plate at the top of the Instron is nearly touching the cylinder beneath it, as pictured here.*

15. Click the “Balance Load” button in the Bluehill program.

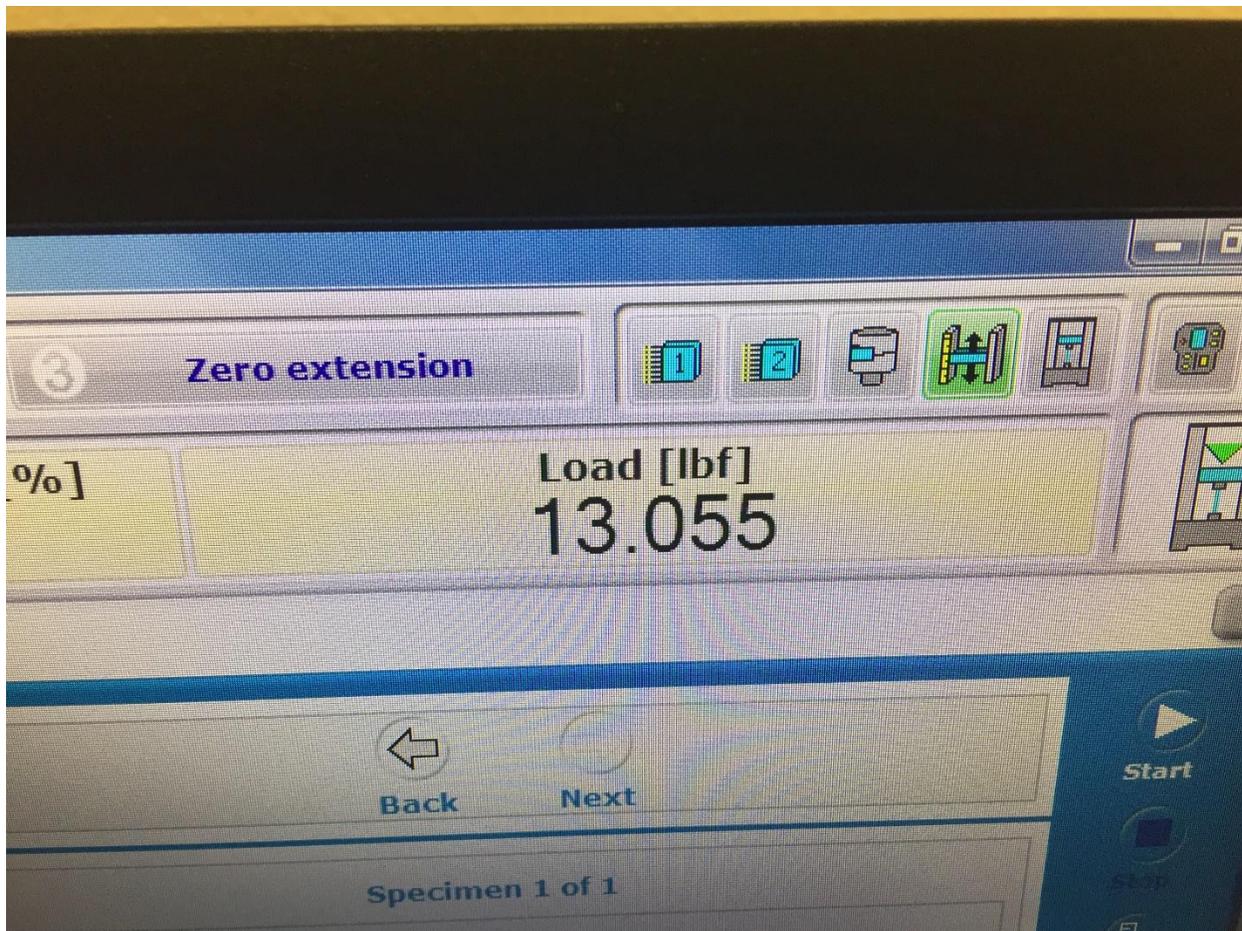


*Ribbon at top of test window functions “Balance All”, “Balance Load”, and “Zero Extension”.*

16. Use the “Fine Position” knob on the panel to slowly and carefully lower the compression plate until “Load”, on the upper right corner of the Bluehill program, reads between 10 and 15 pounds of force. Do not to go far over this point, as it will preemptively compress the sample and skew the results of the test.



*Use the “Fine Position” knob on the panel for a slower, more controlled lowering of the load frame.*



*“Load” should read between 10 and 15 lbf*

17. Once the sample is done being manually pre-loaded, click “Zero Extension”.
18. Click the “Start” button on the far right of the screen. Wait until the sample is finished compressing. Long tests may be ran overnight with Dr. McCoy’s permission.

#### Finishing the Test

19. Once the test is complete, click “Return” on the far right of the screen. This will return the compression plates to their initial positions before the test was begun.
20. Open the Instron door, **again toggling the fan when the doors open and close**. Remove the now compressed sample and replace it with the placeholder. If more space is needed between compression plates, you may use the “Jog Up” button on the control panel.

21. Click the “Finish Sample” button on the computer- **not the “Continue Testing” button, which will not save the experimental data.** The raw data for your test is now saved to the computer. If another sample needs testing, begin this process from the “Sample Preparation” stage.

# Differential Scanning Calorimetry (DSC) Manual

## **Introduction**

The DSC-2000 machine is used to heat and cool samples at a uniform rate under an inert gas to prevent oxidation. By changing the temperature at a set, uniform rate, the change in the heat capacity of the sample can be plotted either as a function of temperature or time. Samples with different compositions and aging times will respond differently to the applied heat flow. This manual will detail how to run a test on the DSC.

## **Safety Information**

- The DSC machine uses nitrogen as a purge gas while it is running. The nitrogen is kept in pressurized cylinders. Use caution when interacting with the nitrogen tanks in case of a blown seal. Stay away from the metal connection hoses when opening the tank valve.
- The DSC machine is kept in a relatively well-sealed room. If one of the nitrogen tanks leaks, the room may fill with nitrogen. This is an inhalation hazard that can quickly lead to hypoxia and unconsciousness. Never work in the lab alone.

## **Procedure**

This document contains the following sections:

- A. Pre-Run Preparations (4 steps)
- B. File Creation and Storage (6 steps)
- C. Running the Test(12 steps)
- D. Post-Run Operations (5 steps)
- E. Exporting Test Data (16 steps)

## Materials

- Pre-Cut, Pre-Weighed DSC Sample
- DSC-2000 Machine
- TA Instruments Program
- Nitrogen Gas
- Analytical Balance
- Tzero Aluminum DSC Pans
- Tweezers
- Flash Drive (for data exportation)

## DSC Test

### Pre-Run Preparations

1. Open TA Instruments program on lab computer and select the Q2000 machine.
2. Under **Control** in the top left corner of the window, select **Event>>On**.

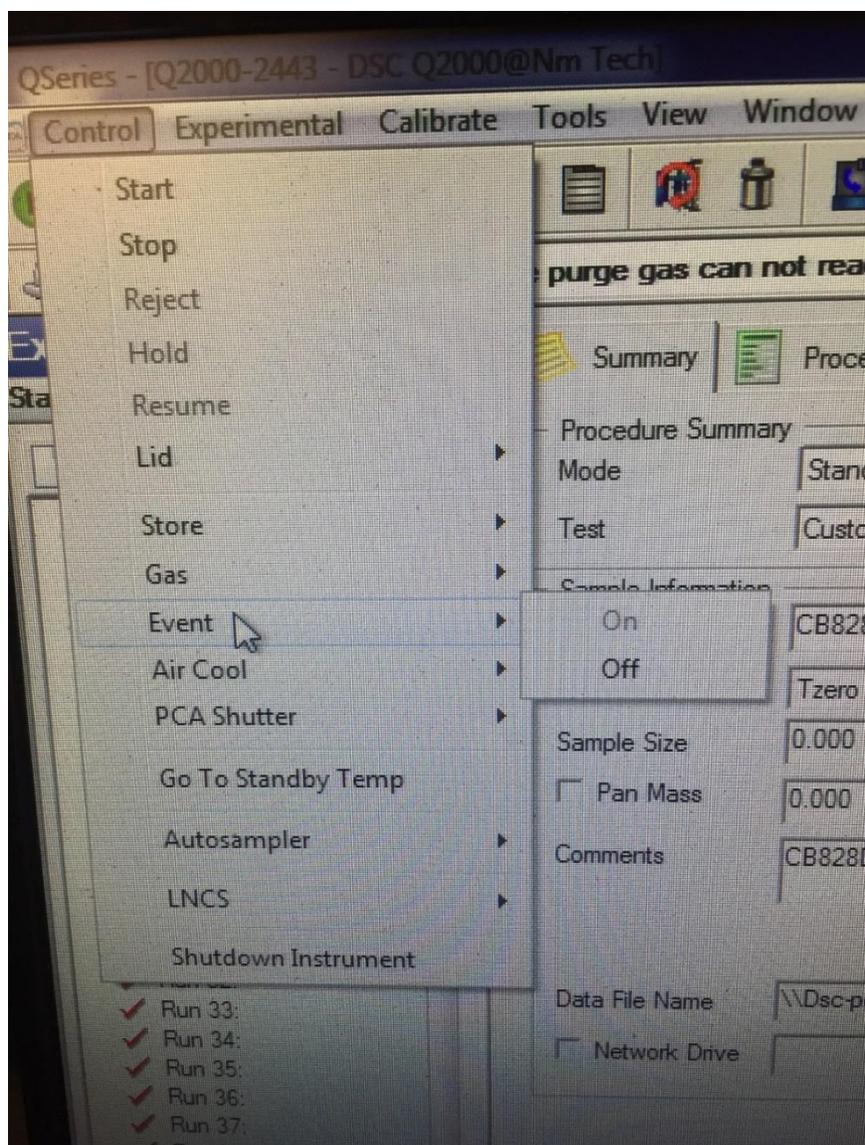


Figure 1. Computer control menu.

3. Go to nitrogen tanks and open the valve. The tanks will hiss when they have been opened, and the pressure gauges will show the change in pressure. **Caution: Keep face and body away from the metal connecting hoses in case the seal between them and the tanks breaks.**



*Figure 2. Nitrogen Tanks.*



*Figure 3. Nitrogen tank pressure gauges.*

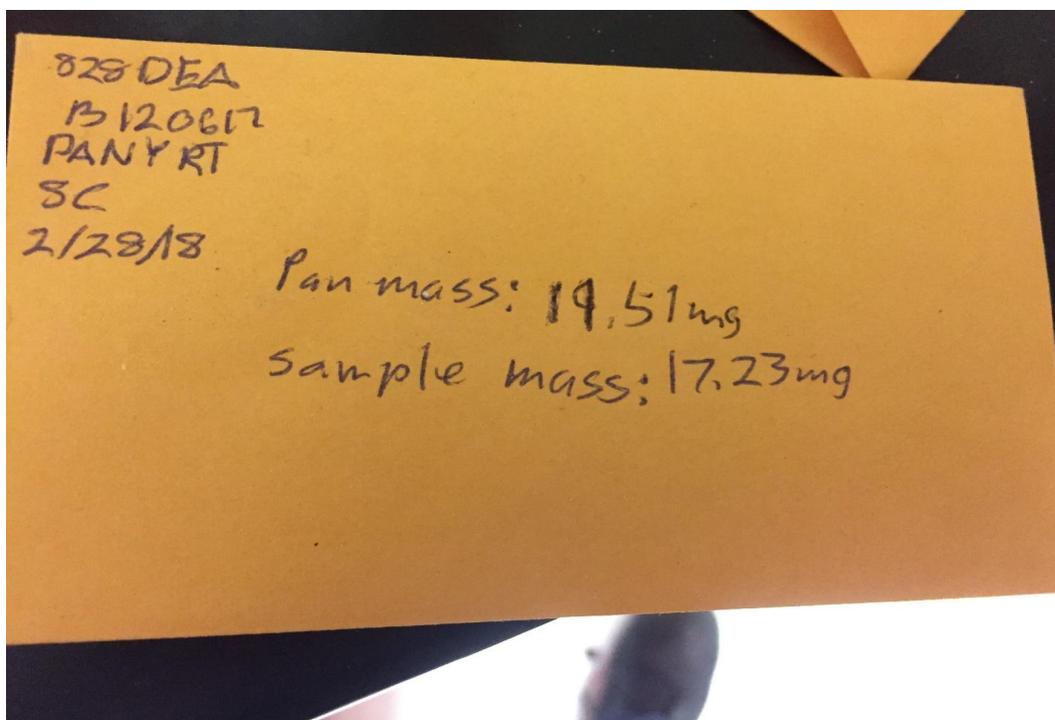
4. Remove the wooden lid over the DSC-2000 machine.



*Figure 4. DSC Machine.*

#### File Creation and Storage

1. Obtain a pre-cut, pre-weighed DSC sample. **Note: DSC samples are extremely small and should be stored in the envelope they arrive in. The envelope should have information pertaining to the composition, creator, aging time, and mass of the sample written on it.**



*Figure 5. DSC sample envelope*

2. In the TA Instruments window, on the leftmost panel, select the button with the folder and the green plus sign. This creates a new file.

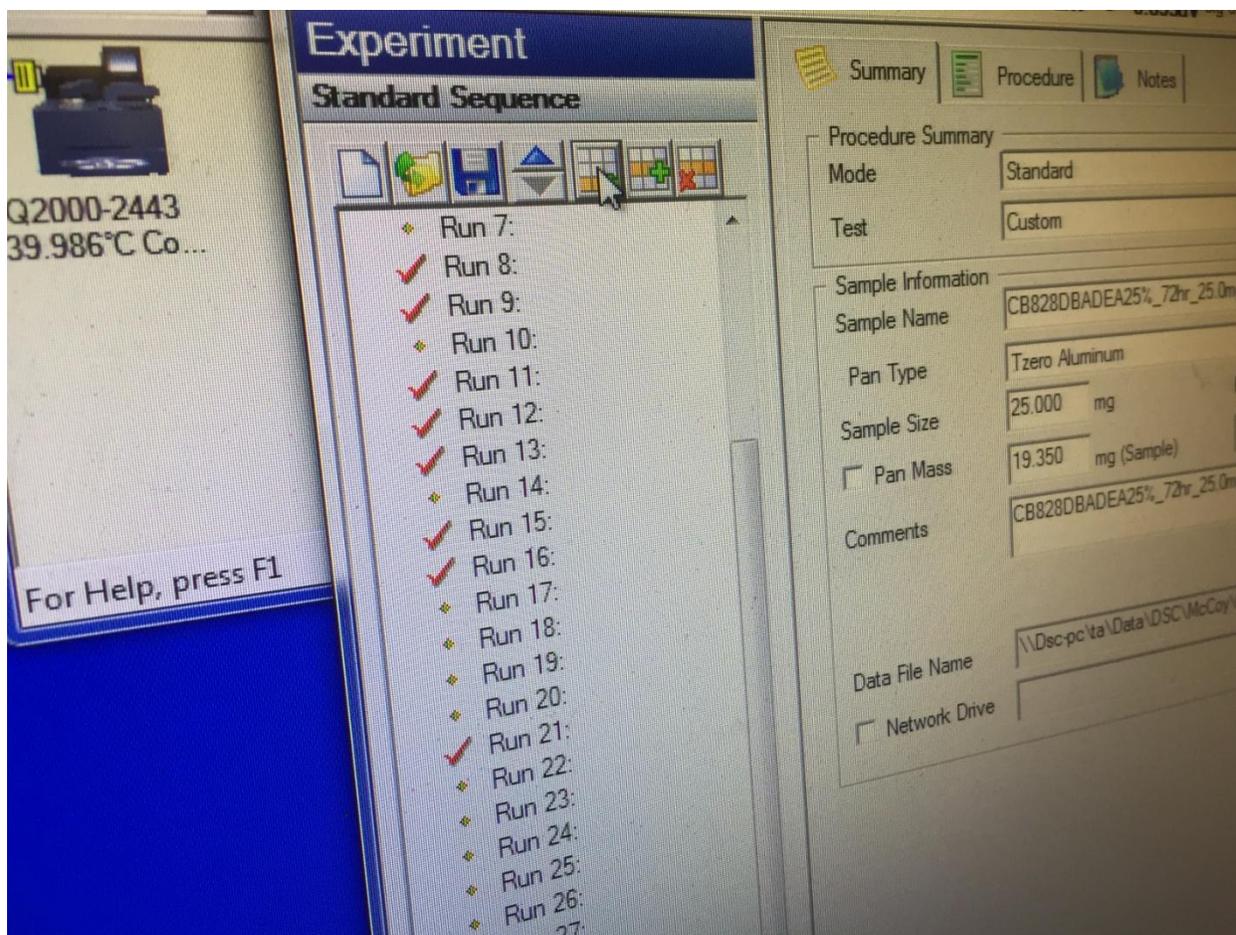


Figure 6. DSC Run Panel

3. In the central panel, under the **Summary** tab, in the box labeled **Sample Name**, type in the sample information. Include the initials of the creator, the composition of the sample, the time the sample was aged, and its mass. Each piece of information should be separated with underscores.

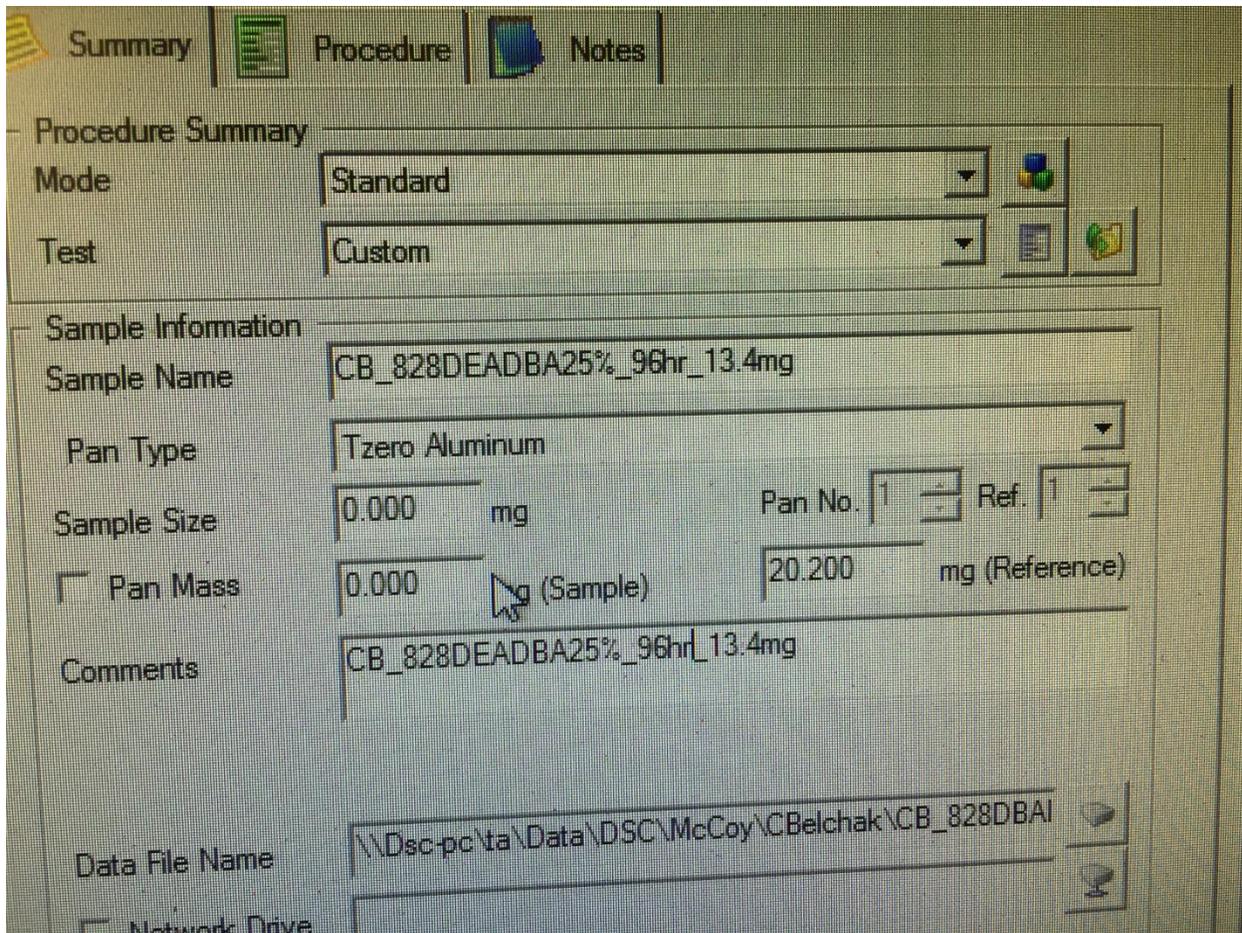


Figure 7. Sample Information Panel

4. Copy the sample information and paste it into the **Comments** box of the **Summary** tab, the **Notes** box under the **Procedure** tab, and in the **Extended Test** box under the **Notes** tab.
5. In the **Data File Name** box under the **Summary** tab, click the button to the right of the box.
6. Navigate the save directory to match the initials of the sample creator under the **McCoy** file and open the file.

Name	Date modified	Type	Size
CB828DBADEA25%_24hr_10.2mg	3/19/2018 10:48 AM	2MG File	
CB828DBADEA25%_72hr_25.0mg	3/9/2018 10:54 AM	0MG File	
CB828DBADEA25%_72hr_25.0mh	3/9/2018 10:55 AM	0MH File	
CB828DBADEA25%_72hr_25.0mi	3/9/2018 12:22 PM	0MI File	

File name: CB\_828DBADEA25%\_96hr\_13.4mg

Figure 8. File save directory

### Running the Test

1. Under the **Summary** tab, enter the mass of the sample in the **Sample Size** box.

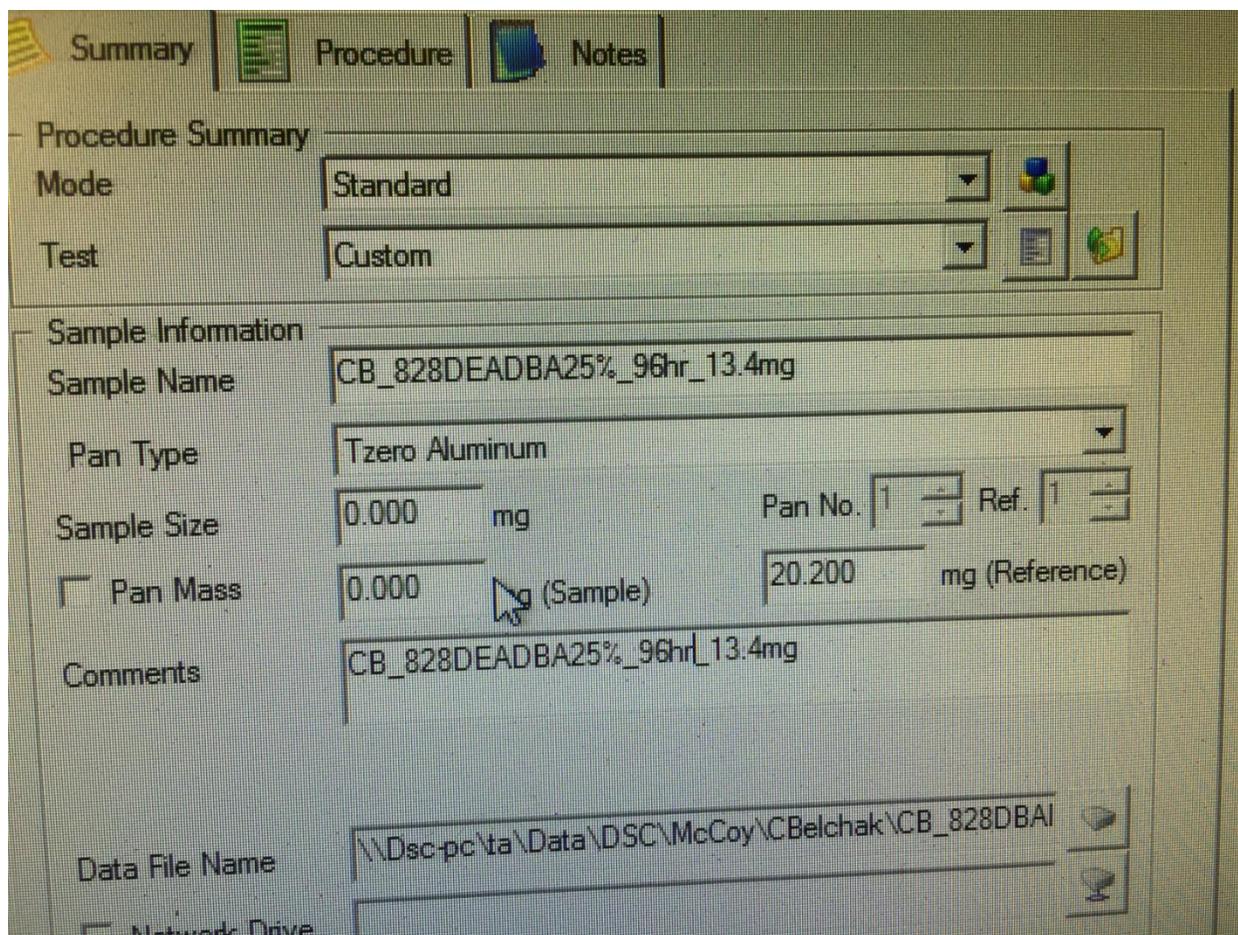
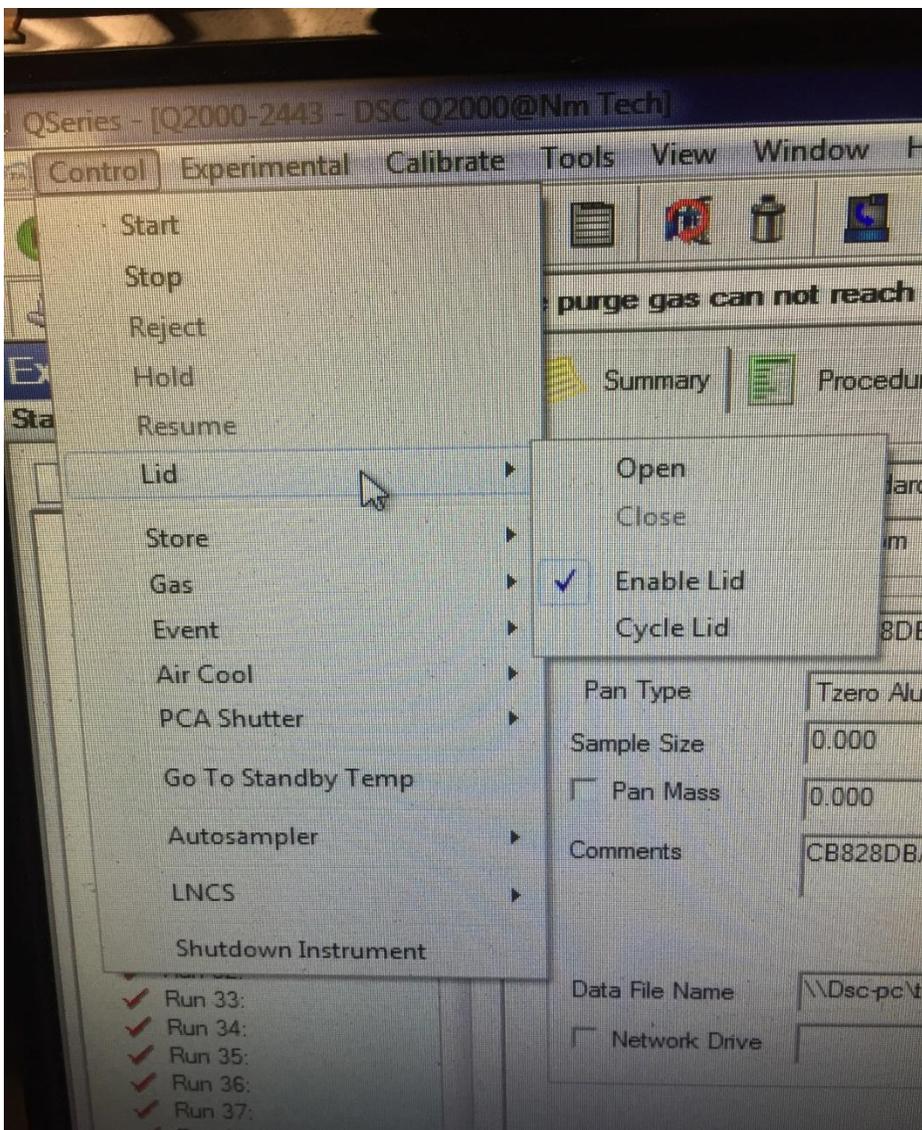


Figure 8. Sample Information Panel

2. Weigh a Tzero Aluminum DSC Pan on the analytical balance. Record the mass of the pan in the **Pan Mass** box. **Note: Do not touch the pan with your fingers. Doing so will affect the quality of the run. Use a tweezers to manipulate the pan instead**
3. Open the sample envelope and get the DSC sample from it. **Note: Do not touch the DSC sample with your fingers. Doing so will affect the quality of the data. Use the tweezers to manipulate the DSC sample.**
4. Place the DSC sample in the Tzero Pan.

5. On the computer, under **Control>>Lid**, select **Open**. **Note: The lid should be kept closed as much as possible to avoid contamination. Perform steps 6 and 7 as quickly as possible.**



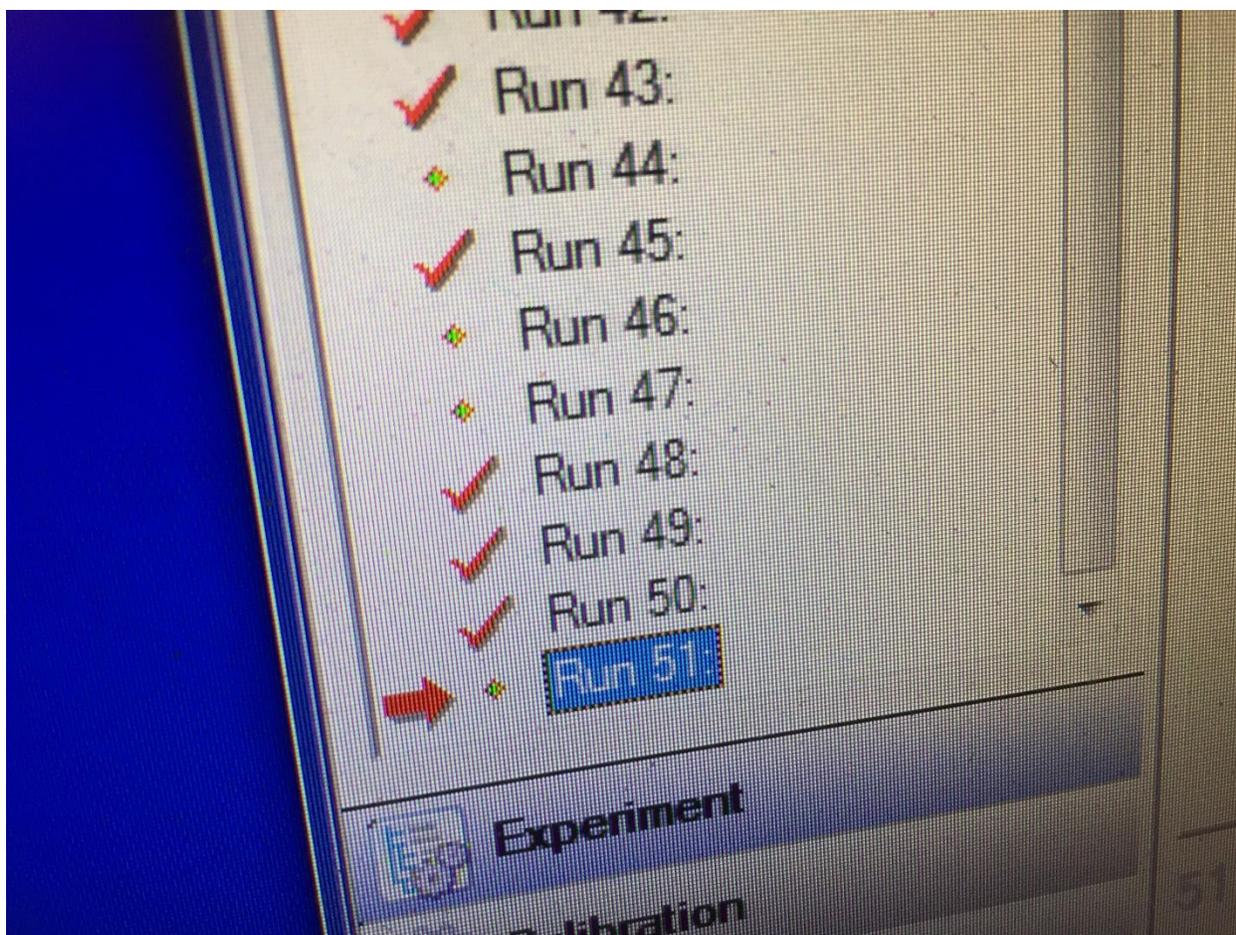
*Figure 9. Lid Control Menu*

6. Once the DSC lid is open, place the pan and DSC sample on the open pedestal inside the analysis chamber. **Note: There is a Reference pan in the analysis chamber. Do not remove it from its pedestal.**



*Figure 10. DSC sample chamber with sample pan and reference pan.*

7. On the computer, under **Control>>Lid**, select **Close**.
8. On the computer, on the leftmost panel, select the arrow. Select the space to the left of the latest run to move the arrow there.
9. Start the run. This can be done by double-clicking the arrow, clicking the green play button on the top left corner, or selecting **Control>>Run**.



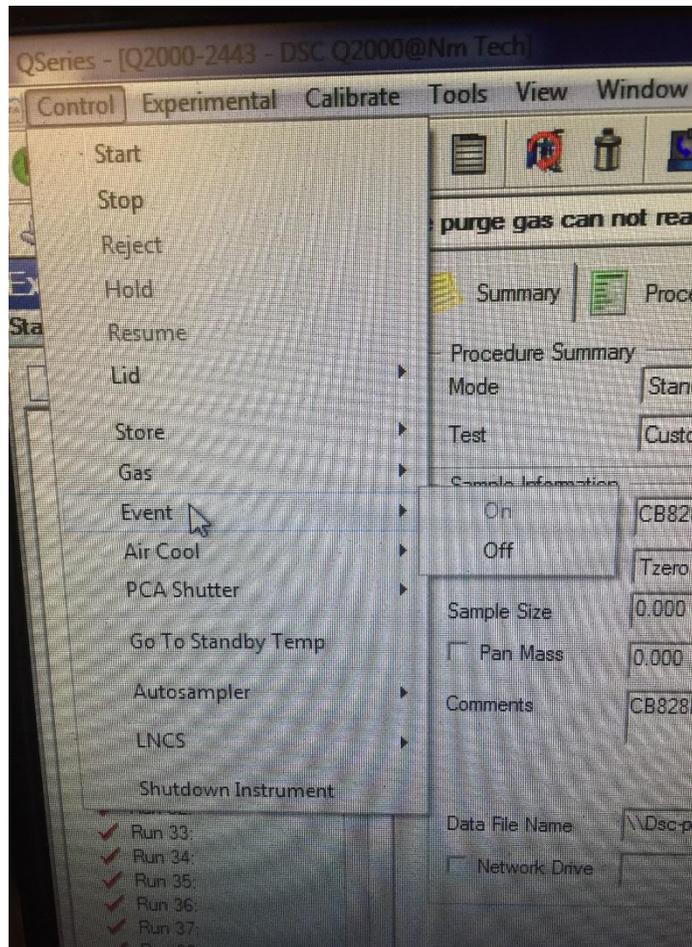
*Figure 11. Sample Information Panel*

10. Replace the wooden cover over the DSC machine.
11. Place the sample envelope near the computer.
12. As a final check, on the left panel on the TA Instruments window, there should be a green stick figure running in place to show that the run is underway.

#### Post-Run Operations

1. Remove the wooden DSC machine cover.
2. Select **Control>>Lid>>Open**.

3. Remove the pan with the DSC sample from the analysis chamber. Replace the DSC sample back into its original envelope.
4. Select **Control>>Lid>>Close**
5. Select **Control>>Event>>Off.**



*Figure 12. Event control panel*

### Exporting Test Data

1. Insert a flash drive into the DSC computer tower.
2. Open the “TA Universal Analysis” program.

3. Go to the “File” tab at the top left of the TA Universal Analysis window. Click “Open File”. This will open a file selection dialog box. Navigate to the desired .rar file. Once you select a file, dialog box will open in the program.
4. Within the dialog box, set “Exotherm” to “Down”.
5. Under “Signals”, and check the signal box which contains data. If more than one signal has data, set “Signals” to “Nothing”.
6. Click “Ok”. A graph should appear in the program window.
7. Change the X Axis of the graph to temperature by right clicking on the plot, going to “X Axis”, and selecting “Temperature”. Set the time ranges to the following:  
“First Heat”: 10-23  
“Second Heat”: 48-60
8. The graph should now resemble an "S"-like curve. If the data time limit is too large, then there will be steep drops on either side of the ramp. Reduce the time in order to get the desired data set. If the data time limit is too short, then the ramp will terminate on one or both ends before it levels off. Increase the time in order to get the desired data set.
9. Once the desired data set has been isolated, go to “File”, “Export Data File” and then “File Signals Only”.
10. An export dialog box will appear. Under the “Character Set” box, check “Windows (ANSI)”.
11. Under “Options”, check all three boxes: “No parameter block”, “No data breaks or flags” and “No invalid data points”.

12. Name and save the data file.
13. Open Windows Explorer, located on the taskbar.
14. Go to “Recent Places” and select your recently saved data.
15. Copy the data files onto a flash drive.(there should be one RAR file that the macs cannot open and a number of csv files that you created in this procedure)
16. Close all windows, and safely eject your flash drive.

# ARES Rheometer Testing Manual

## **Introduction**

The ARES is a rheometer, and measures the flow of a material under certain stresses and/or temperatures. The ARES in 130C has compression and torsion testing functions. For the former, flow is measured for a gel sample pushed between two parallel plates. For the latter, force is applied to cured epoxy in a twisting motion. This manual will detail how to perform these tests on the ARES rheometer.

## **Safety Information**

- This process involves bringing samples up to a high temperature. Avoid touching the machine while the test is running to prevent injury.
- This machine has moving parts. Avoid touching the motor while turning it on.
- Wear gloves while handling uncured epoxy. Skin sensitivities can develop upon consistent exposure.

## **Procedure**

This document contains the following sections:

- A. Powering On (6 steps)
- B. Parallel Plate Test (8 steps)
- C. After Testing (6 steps)
- D. Torsion Bar Test (6 steps)
- E. Exporting Test Data (2 steps)

## **Materials**

- Small spatula
- Allen wrench
- Uncured epoxy or cured epoxy torsion samples

## ARES Test

### Powering On:

1. Turn on the air, up to the left when you walk into the room.



*Off: the red handle is perpendicular to the pipes*



*On: the red handle is turned 90 degrees.*

2. Switch on the Hankison.



*The Hankison in 130B. There is now one specifically for the ARES in 130C.*

3. Switch on the ARES. The power switch is located on the right hand side of the console underneath the metal covering.



*The on/off switch on the side of the ARES*

4. Using the mirror located behind the ARES, confirm that the pressure is stable and let the machine warm up. Take this time to sign into the log book for the ARES. The machine should read the temperature of the room once ready to use.



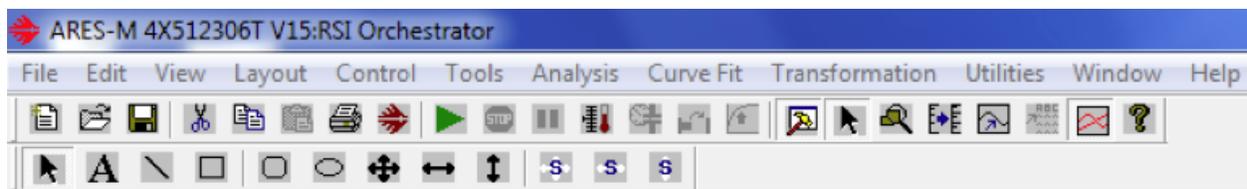
*The screen on the ARES reads 21.1 C here , about room temperature.*

5. The startup software is RSI Orchestrator. Open it up to prepare for testing.

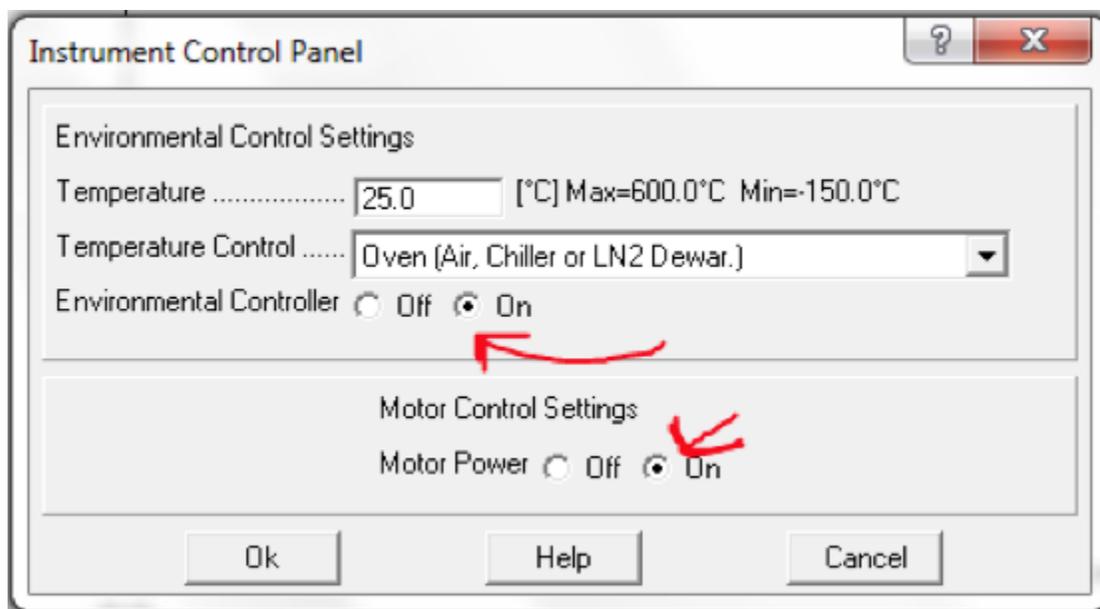


*The “RSI Orchestrator” program icon*

6. Under the **Control** menu, open **Instrument Control Panel** and set the environmental controller and the motor to **On**. Alternatively, click on the control panel button located in the shortcuts menu and it will open the window.



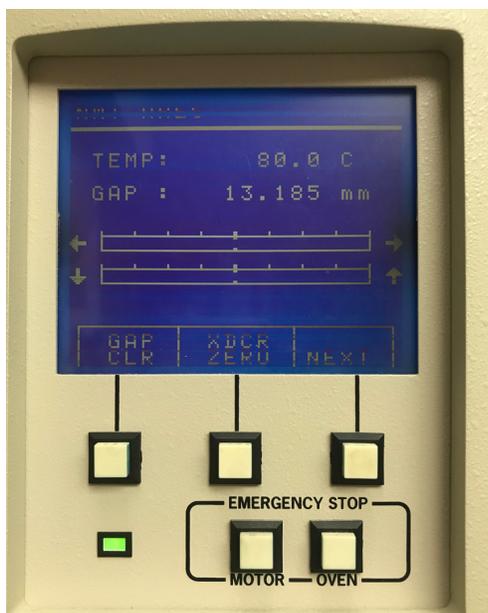
*The RSI Toolbar*



*The Instrument Control Panel, accessed from the “Control” panel.*

### Parallel Plate Test

1. Before beginning, install the proper fixtures for the parallel plate test. Use the allen wrench to loosen screws, and your hand to tighten them. Measure and record the diameter of the fixture where the sample will be added.
2. Start by zeroing the transducer, XDCR ZERO, so there is no normal force on the plate.

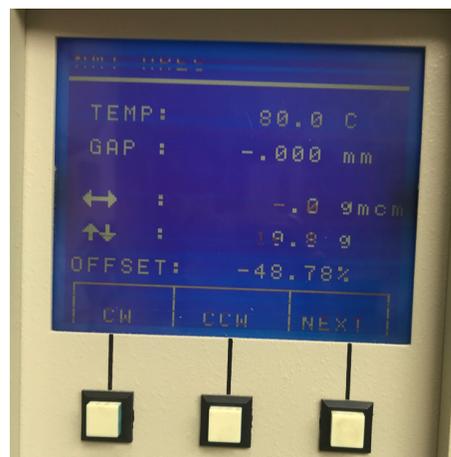


The XDZR ZERO button is the upper center one.

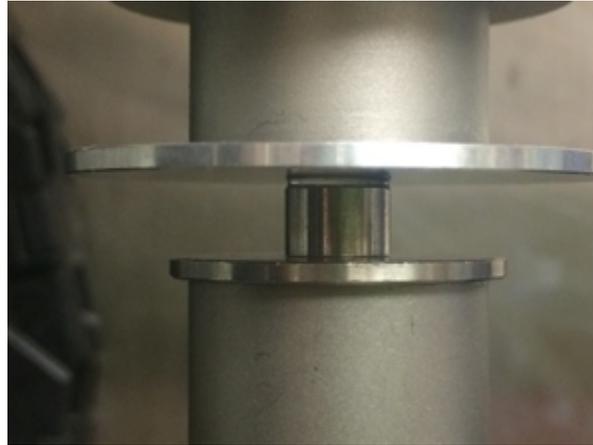


The jogging panel of the ARES

1. Now, simultaneously pressing the downward arrow and the middle button, jog the top plate down until it almost makes contact with the bottom fixture. There should be no normal force.
2. Once the plates are fairly close, use the bottom arrow only to very slowly bring the top plate down. Avoid overloading the machine by watching the value of the normal force on the screen . Bring it down until the normal force becomes slightly positive, around 10 grams.
3. Clear the gap using the GAP CLR button.
4. Jog the top plate back up using the upward arrow and the middle button.
5. Add a thin layer of the liquid/gel sample to evenly coat the bottom plate.

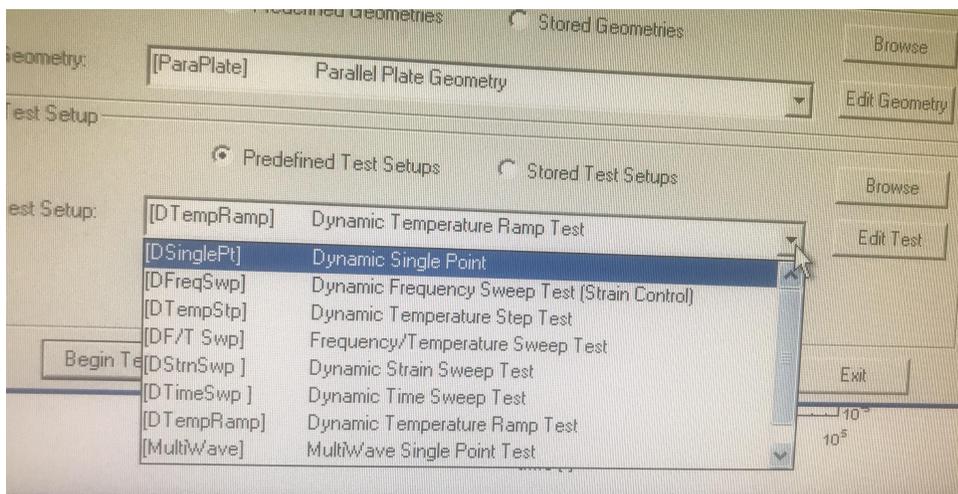


6. Jog down the top plate using both the bottom and middle arrow keys until the top fixture barely makes contact with the sample. Then, use only the bottom arrow until the sample is uniformly cylindrical, and does not dip inwards or outwards at its center. The sample sandwiched between the two plates should be about 1 to 2 mm thick.



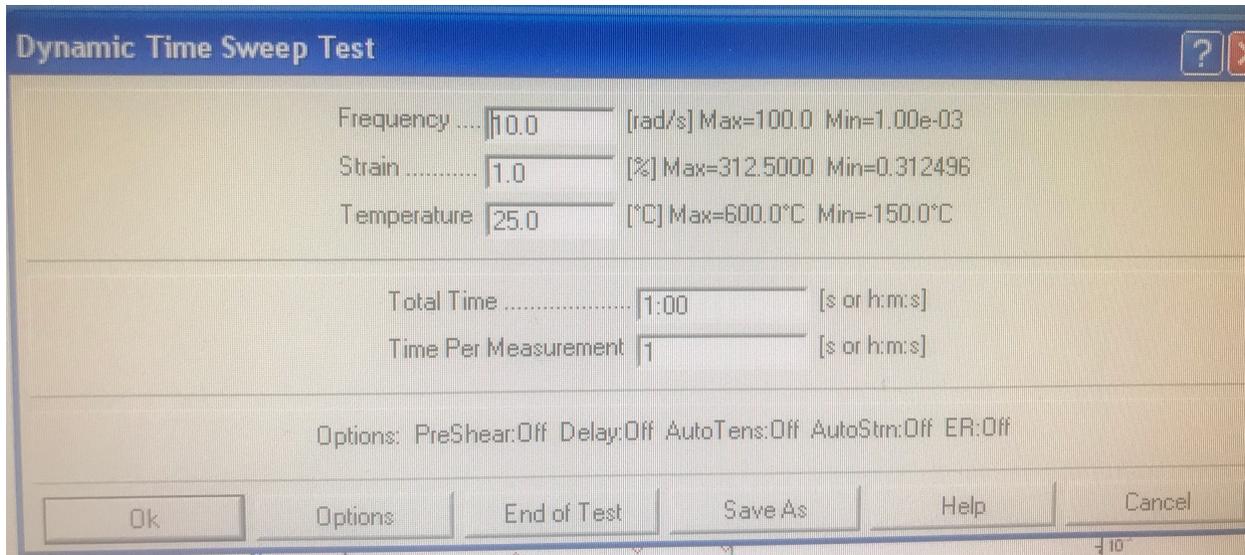
*The sample forms a proper cylinder between the plates. The test can now begin.*

7. Click the start button on the computer screen. Name your test file appropriately: make sure to include all relevant information, and use underscores to break phrases instead of spaces. Once this is done, the details of the test may be input to the computer.



*Test setup screen*

The test primarily being run at this time is the dynamic time sweep. Fill in the parameters and click ok.



*ARES test parameter window*

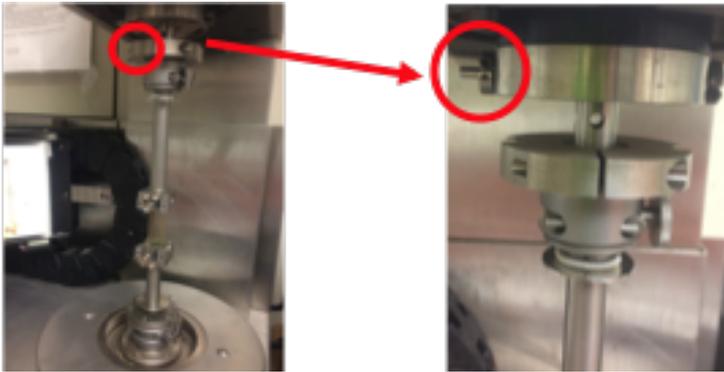
8. Once parameters have been defined, click the **Begin Test** button.

After Testing:

1. Before shutting down the machine, make sure you have saved you test data.
2. Turn the motor off by going to **Control** and then **Instrument Control Panel**.
3. Loosen the tested sample with an Allen wrench, and store appropriately.
4. Turn the power off using the switch on the main box.
5. Turn off the main air.
6. Export your test data if necessary (see “Exporting Data” segment).

### Torsion Bar Test

1. Install the proper fixtures to the ARES.  
Make sure the pin pictured is removed. Install the torsion bar fixture, loosening screws with an allen wrench and tightening them by hand.

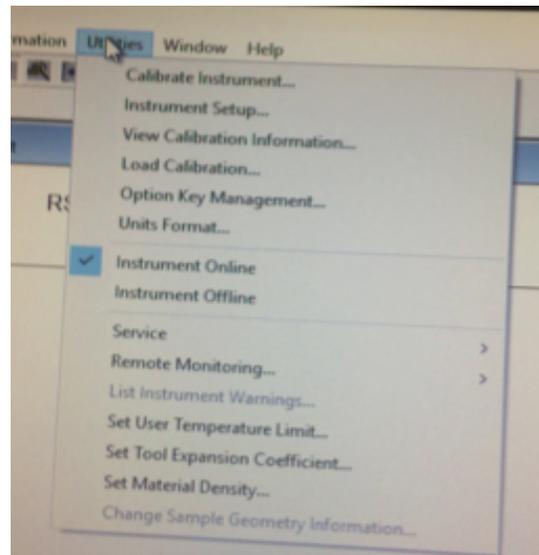


*This pin must be removed to install the torsion bars.*

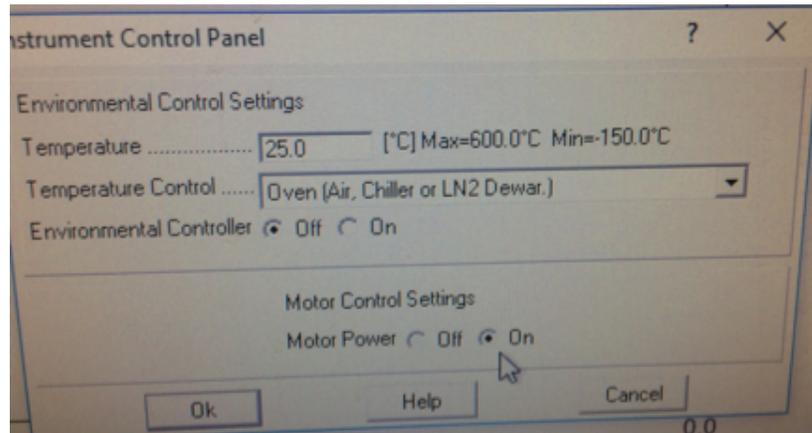
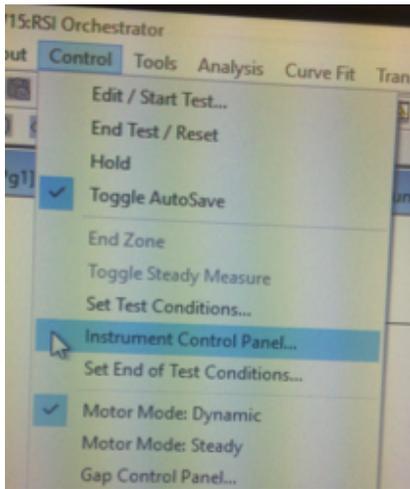


*The installed torsion bars.*

2. Make sure the instrument is online.
3. Turn the motor on. The motor must be on before installing a sample.



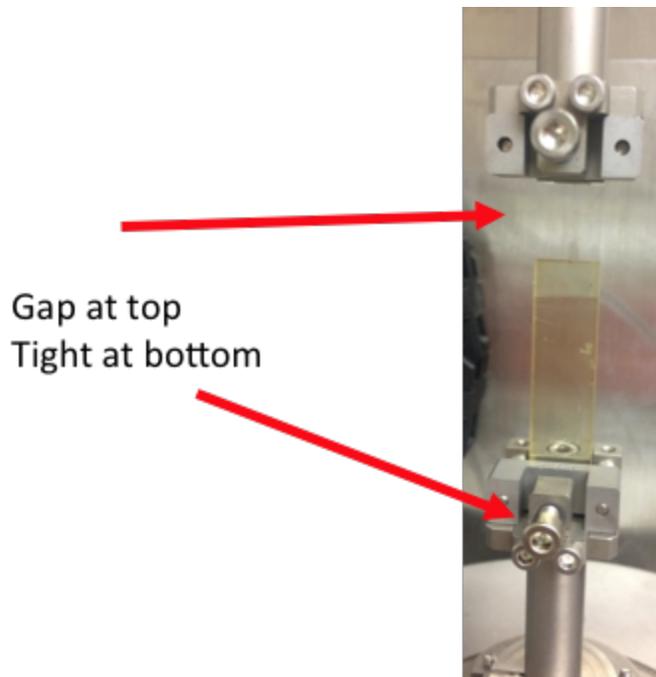
*Check under “Utilities” to confirm the instrument is online.*



*Access the Instrument Control Panel from the “Control” tab.*

*Turning the motor on can be done near the bottom of the Instrument Control Panel.*

4. When installing the torsion bar, slip the bar in place in the bottom fixture and tighten it with an Allen wrench. Make sure the upper grip is open, and then with the jog-up/jog-down buttons move the upper fixture down over the sample. Watch the screen where the torque and normal force are showing. Use the CW (clockwise) and CCW (counterclockwise) to rotate the lower grip to keep torque ~0 (between -10 and +10gcm). Also make sure the normal force does not get too high, keeping the value slightly negative.



*There should be a gap between the upper bar and the sample when first loaded. The upper bar will be jogged down once ready.*

5. Tighten the sample with an Allen wrench making sure the torque stays in range and adjust CW and CCW as the sample is tightened. Don't panic if it jumps to several hundred.

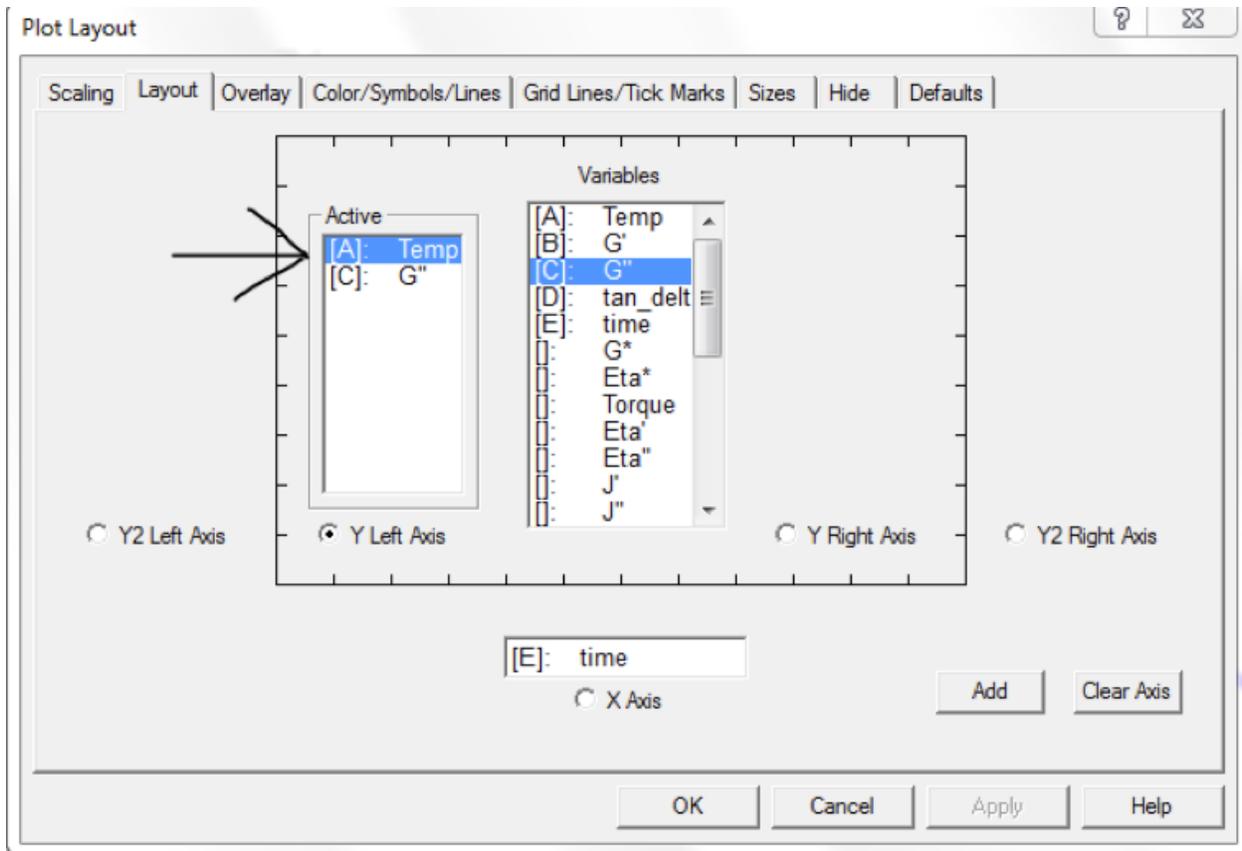
If torque overloads during installation of sample, two things must be done to correct:

- Reset by clicking "End Test/Reset" (the Stop sign)
- Turn motor back on

6. You are now ready to perform a test. Follow the same steps of running a test and shutting down the machine as defined in the "Parallel Plate Test" segment.

#### Exporting Test Data

1. In the RSI software, open the test file that you would like to export. Add all the desired data to the plot. Do this under **Layout** (double click on the plot).



*The “Layout” tab under “Plot Layout”*

2. Once the plot with all the data is open, click **File** and then **Export**. Set the data to save as ‘.txt’. Also set the desired destination of the exported file.

# KaleidaGraph Plotting and Data Analysis Manual

## **Introduction**

Once a sample has been tested, the test data must be made sense of. We use KaleidaGraph to plot and analyze our test data, since it is superior to Excel in its graphing capabilities. This manual will detail how to make proper graphs for Instron and DSC test data using KaleidaGraph.

## **Procedure**

This document contains the following sections:

- A. Instron Compression Test Plotting
  - a. Creating Data Table (7 steps)
  - b. Creating Plot (21 steps)
  - c. Data Analysis (12 steps)
  - d. Formatting Plot (12 steps)
- B. DSC Test Plotting
  - a. Creating Data Table (10 steps)
  - b. Creating Thermogram (7 steps)
  - c. Creating Heat Curves (16 steps)
  - d. Formatting Plot (1 step)

## **Materials**

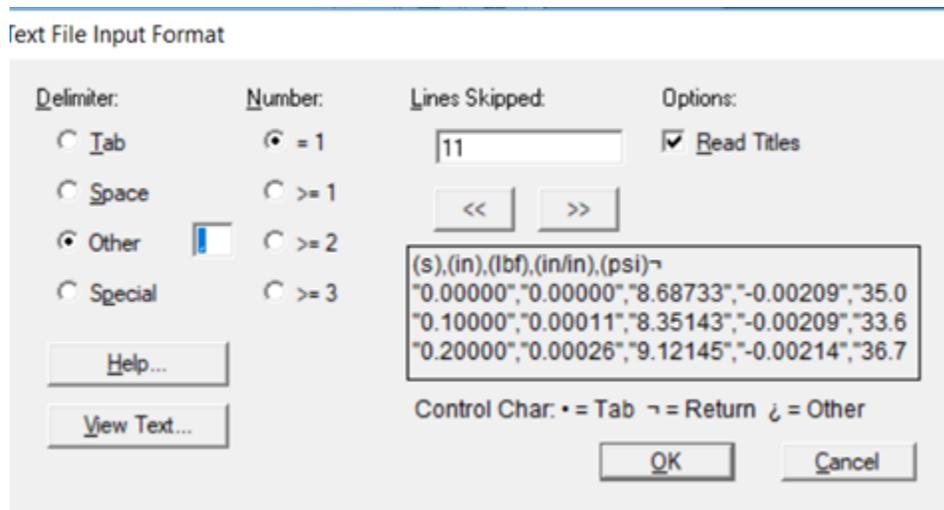
- KaleidaGraph (This can be downloaded on a student discount. The cost will be reimbursed by the Materials Department).
- All necessary data files, loaded onto the computer you are using to graph.

# Instron Compression Test Plotting

## Creating Data Table:

### *Opening Data File*

1. Open the KaleidaGraph program.
2. Go to the control ribbon at the top of the window. Follow the directory: **File » Open**, and find the location of your test data. There will be many files to choose from; open the folder with the suffix “\_RawData”, and choose the file named “Specimen\_RawData\_1”.
3. The “Test File Input Format” menu will open.
  - a. Set “Delimiter” to “Other”, typing a single comma into the specification box.
  - b. Set “Number” to “1”
  - c. Set “Lines Skipped” to 11
  - d. Check “Read Titles”
  - e. Click “OK”.



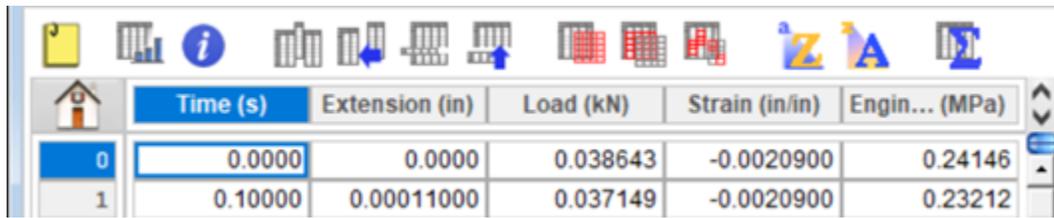
*“Text File Input Format” dialog box*

### *Formatting Data Table*

4. Clicking into the new data table, enter the keyboard commands **Ctrl+A** and **Ctrl+K** in order to highlight all your data and open a data formatting menu. In the menu, change “Type” to “Float” and click “Apply”.

5. Change the table’s column titles to read:

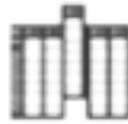
*Time (s) | Extension (in) | Load (kN) | Strain (mm/mm) | Engineering Stress (MPa)*



	Time (s)	Extension (in)	Load (kN)	Strain (in/in)	Engin... (MPa)
0	0.0000	0.0000	0.038643	-0.0020900	0.24146
1	0.10000	0.00011000	0.037149	-0.0020900	0.23212

*Correctly named column titles*

6. Add four columns using the “Insert Column” symbol (pictured below), which is located on the toolbar at the top of the data window.



*The “Insert Column” icon*

7. Name these new columns in the following way:

*Strain Adjust (in/in) | Engineering Strain (in/in) | True Strain (in/in) | True Stress (MPa)*

### Creating Plot:

*Strain Adjust*

1. Click the double-arrow symbol (pictured below) to reveal the variable labels for each column.

The screenshot shows a software window titled "906\_B072518\_S2\_0.1ipm\_1.001in 10:53:30 PM 10/26/2018". The interface includes a toolbar with various icons. Below the toolbar is a table with the following columns: "Extension (in)", "Load (kN)", "Strai...st (in/in)", "Engin...n (in/in)", and "Strain (in/in)". The table has two rows of data. A circled icon in the top right corner of the table area indicates a function to expand or collapse the column labels.

	Extension (in)	Load (kN)	Strai...st (in/in)	Engin...n (in/in)	Strain (in/in)
	c1	c2	c3	c4	c5
0	0.0000	0.038643	0.0020879	-0.0035904	-0.0020900
1	0.00011000	0.037149	0.0020879	-0.0035904	-0.0020900

Click the circled icon to expand or collapse the view of the column labels.

**Note:** By highlighting an individual column, the variable labels are shifted. To avoid confusion, make sure that no columns are highlighted during any of the data operations in this stage.

The screenshot shows the same software window as above, but now the "Strai...st (in/in)" column is highlighted in blue. This action has shifted the column labels: "c0" is now under "Strai...st (in/in)", "c1" is under "Engin...n (in/in)", and "c2" is under "Strain (in/in)". The table now contains 10 rows of data.

	Extension (in)	Load (kN)	Strai...st (in/in)	Engin...n (in/in)	Strain (in/in)
			c0	c1	c2
0	0.0000	0.038643	0.0020879	-0.0035904	-0.0020900
1	0.00011000	0.037149	0.0020879	-0.0035904	-0.0020900
2	0.00026000	0.040574	0.0021379	-0.0035404	-0.0021400
3	0.00044000	0.045643	0.0023177	-0.0033606	-0.0023200
4	0.00062000	0.037609	0.0026374	-0.0030409	-0.0026400
5	0.00080000	0.026337	0.0030170	-0.0026613	-0.0030200
6	0.00098000	0.032642	0.0031868	-0.0024915	-0.0031900
7	0.00116000	0.035476	0.0033467	-0.0023316	-0.0033500
8	0.00133000	0.038170	0.0034865	-0.0021918	-0.0034900
9					

Row: 0 Column: 3

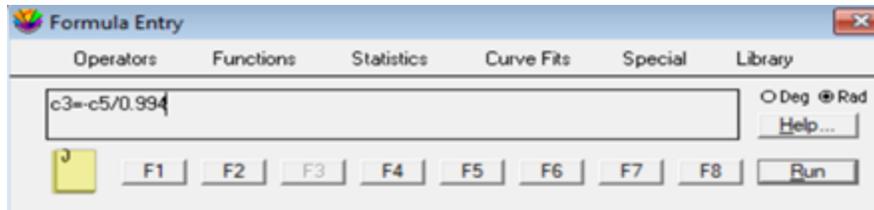
Highlighting a column shifts the formula labels.

2. Enter the keyboard command **Ctrl+F** to open the "Formula Entry" window.
3. Enter the following formula into the text box:

$$[Strain Adjust] = - [Strain] / \{length of test sample in inches\}$$

The length of the test sample should be available in the test's data file name.

**Note:** Column titles in brackets represent that column's corresponding variable (i.e. 'c8').

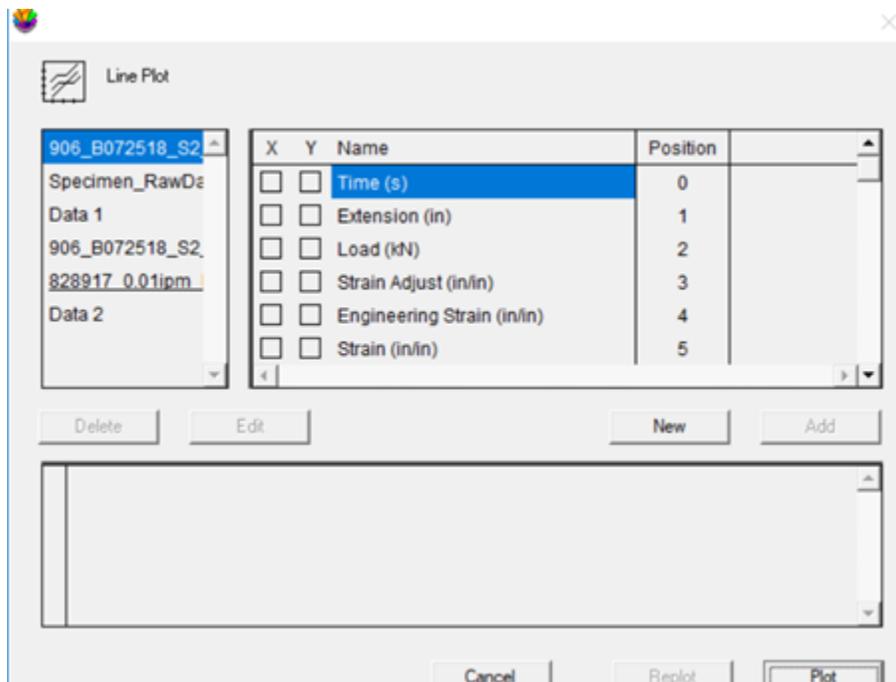


*“Formula Entry” window with correctly typed formula*

4. Click “Run” to apply the operation.

*Stress-Strain Toe Correction*

5. From the control ribbon at the top of the window, follow the directory: **Gallery » Linear » Line.**
6. The following window should open:

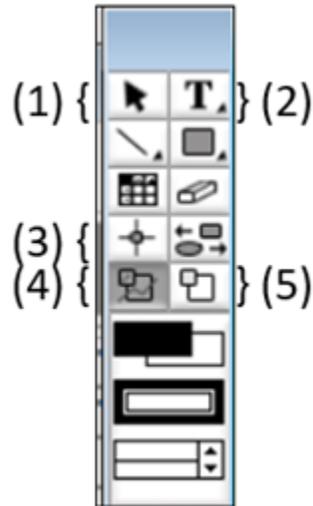


*The “Line Plot” dialog box*

7. Check “Strain Adjust (in/in)” under “X”, and “Engineering Stress (MPa)” under “Y”.
8. Click “Add”.

9. Click “Plot”.

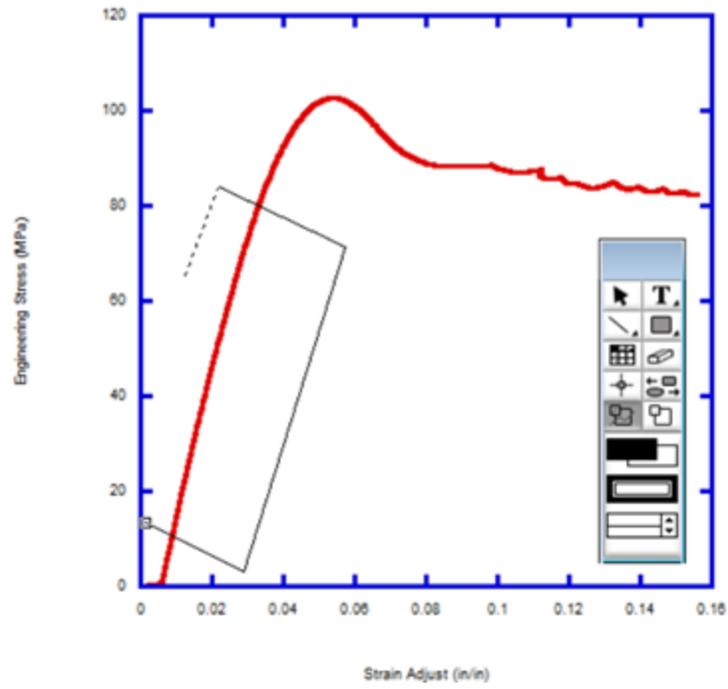
10. A stress/strain plot window, along with a separate plot toolbar (pictured below), should appear.



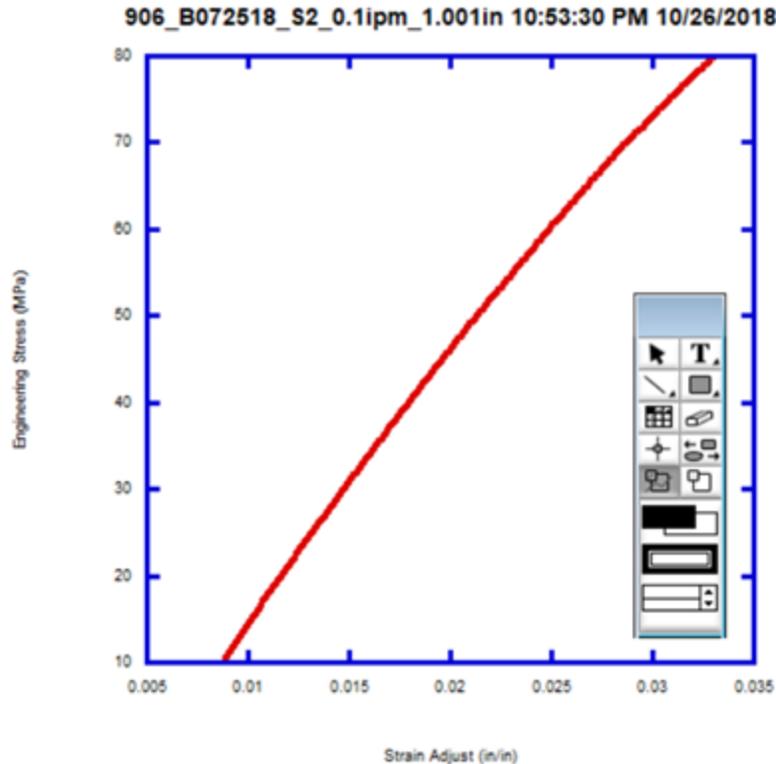
*Plot Toolbar: your default tool should be (1). Always click back to it after utilizing other tools.*

11. Using the masking function, (4), box in a linear portion of the curve. Click on the plot to ‘pin’ the corners of the box, and connect back to the starting point in order to ‘close’ the box and initiate the masking.

906\_B072518\_S2\_0.1ipm\_1.001in 10:53:30 PM 10/26/2018



*Boxing in linear portion of curve using masking tool.*



*The isolated portion of the curve.*

12. From the control ribbon, follow the directory: **Curve Fit » Linear**.
13. Check “Engineering Stress (MPa)” and click OK. A linear best-fit line should appear on the plot.
14. Right click on the plot, and check “Display Equation”. A linear ‘  $y = mx + b$  ’ equation will appear. Record the given ‘b’ value on paper, or copy it to the clipboard.
15. Before proceeding, it is essential that the data masking is undone by double-clicking (4) on the plot toolbar. **Note: The linear best-fit line will shift to accommodate the new data, and the ‘  $y = mx + b$  ’ equation will become unusable. This is why the initial ‘b’ value was recorded.**

16. Check that none of the cells in the data table are bordered in red (indicating masking). After this has been confirmed, the toe correction stress/strain plot may, under most circumstances, be discarded without saving. Consult with Dr. McCoy before doing so.

17. From the data table's formula window (opened using **Ctrl+F**), enter and run the following operations, in order:

$$[Engineering\ Strain] = [Strain\ Adjust] - \{ 'b' \}$$

$$[True\ Strain] = - \ln (1 - [Engineering\ Strain] )$$

$$[True\ Stress] = [Engineering\ Stress] * (1 - [Engineering\ Strain] )$$

18. From the control ribbon, follow the directory: **Gallery » Linear » Line**.

19. Check "Engineering Strain (in/in)" under "X" and "Engineering Stress (MPa)" under "Y". Click "Add".

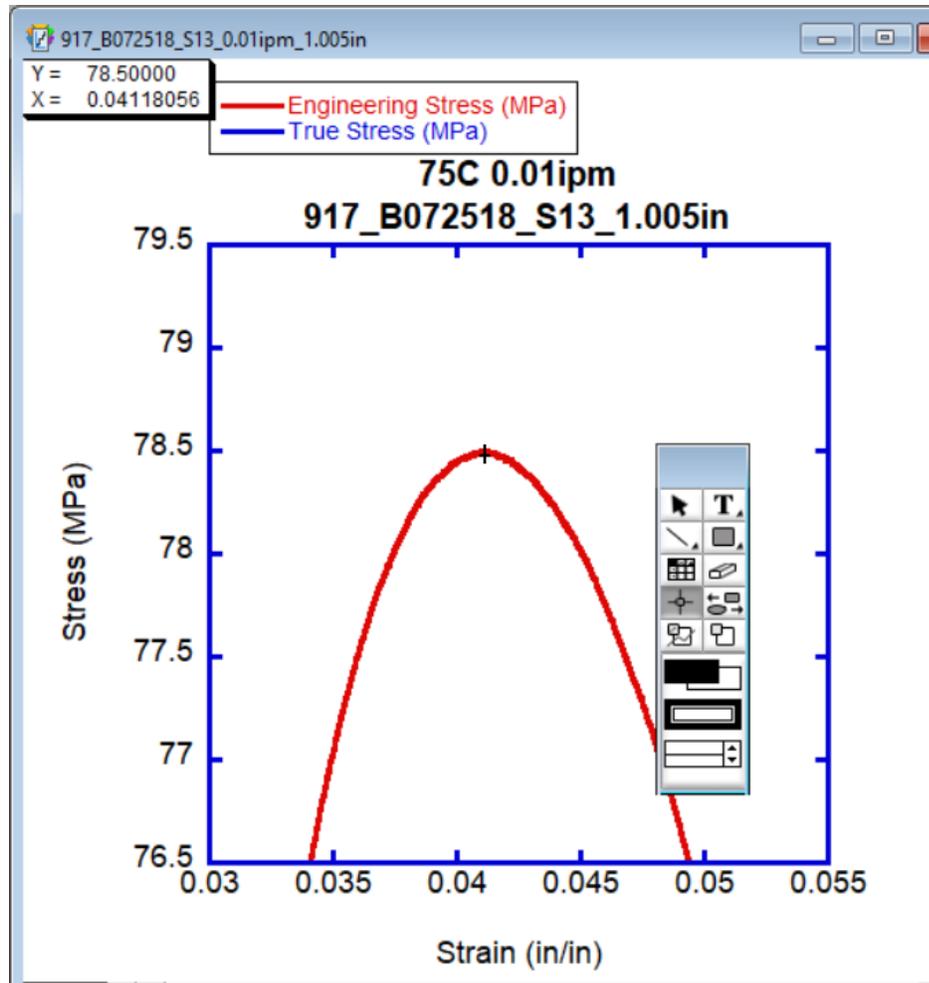
20. Check "True Strain (in/in)" under "X" and "True Stress (MPa)" under "Y". Click "Add".

21. Click "Plot". A plot with two stress/strain curves should appear.

Data Analysis:

*Yield Stress*

1. Use zoom, (5), on the plot toolbar to close in on the peak of the Engineering stress/strain curve.
2. Use the crosshair tool, (3), and click+hold on the peak. The strain/stress coordinates of where you have clicked will appear in the upper left corner of the plot window. Record the y-value to one decimal place, with units of MPa. Double-click on the zoom tool to zoom back out.

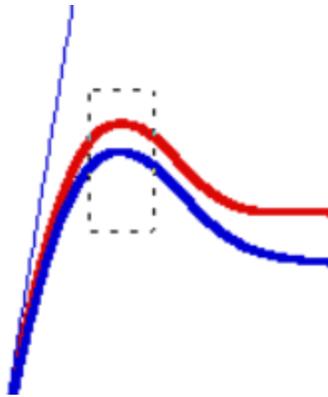


*Centering and clicking on the peak using the crosshair function*

3. Repeat steps 1-2 for the True stress/strain curve.

*Crosshead Speed at Yield*

4. Using the masking tool on the plot toolbar, (4), isolate the peaks of both curves at the same time.



*Masking both peaks at once.*

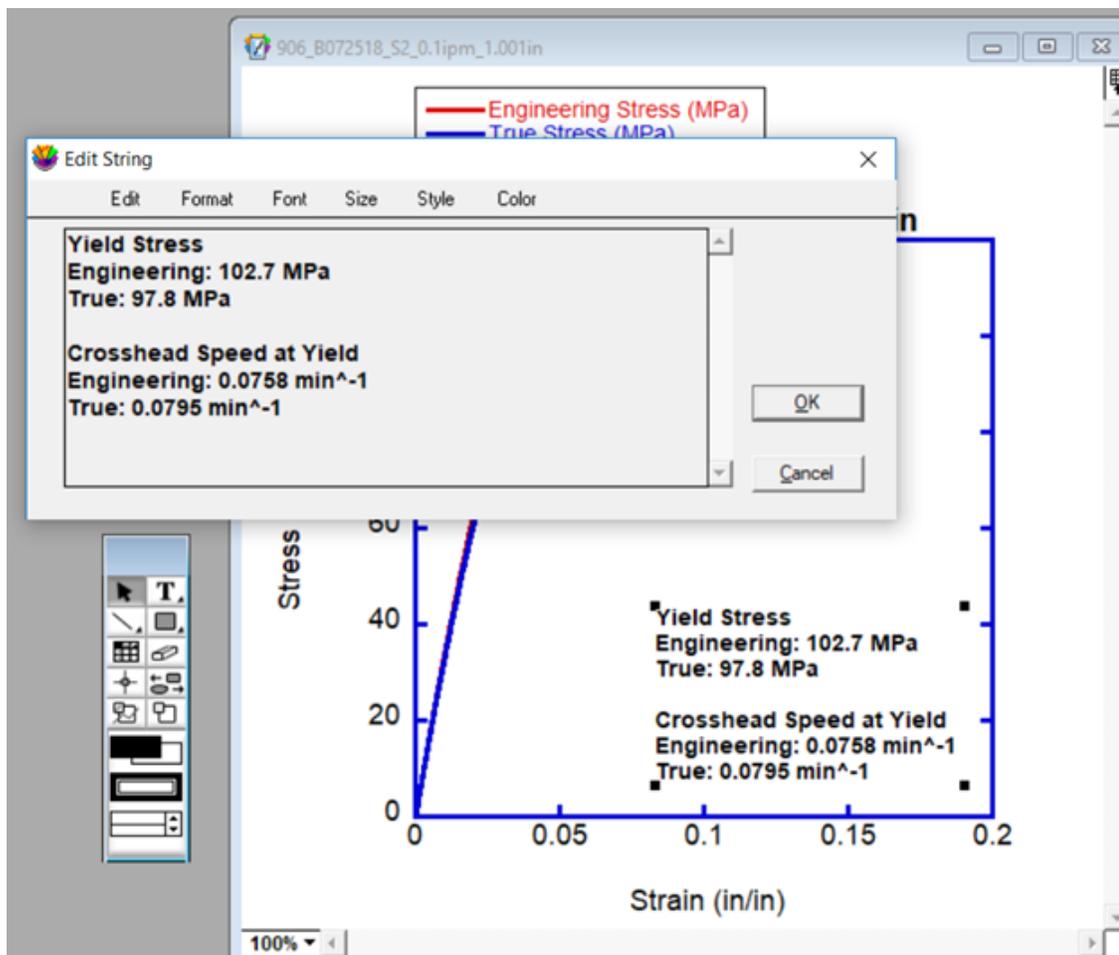
5. Go to the data table. If some data is bordered in red, the masking successfully carried over. If not, click on the grid icon on the upper right corner of the plot window to refresh the table-plot link.



*Plots in KaleidaGraph have a tendency to remove themselves from the jurisdiction of their parent data tables. Clicking this icon will make a new but identical data table appear- one with a link to the plot.*

6. From the control ribbon, follow the directory: **Gallery » Linear » Line**.
7. Check “Time (s)” under “X”. Under “Y”, check “Engineering Strain (in/in)”, as well as “True Strain (in/in)”. Click “Add” and then “Plot”.
8. Right click on the new strain/time plot. Check “Display Equation”. Two ‘  $y = mx + b$  ’ equations should appear, one for Engineering and one for True.

9. Multiply each 'm' value by 60. This is the Crosshead Speed at Yield for each curve. Record these values to three significant figures, with units of  $\text{min}^{-1}$ .
10. At this point the strain/time plot may be, under most circumstances, discarded without saving. Consult with Dr. McCoy before doing so.
11. Using the text box tool on the plot toolbar, (2), click on the stress/strain plot. Create an entry like the one pictured below, inputting the recorded values for yield stress and crosshead speed.

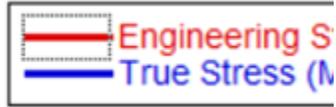


*A correctly written text box with data analysis values.*

12. Set the font size to 14 and the style to 'Bold'. Place the text in such a way that the stress/strain curves are obstructed as little as possible.

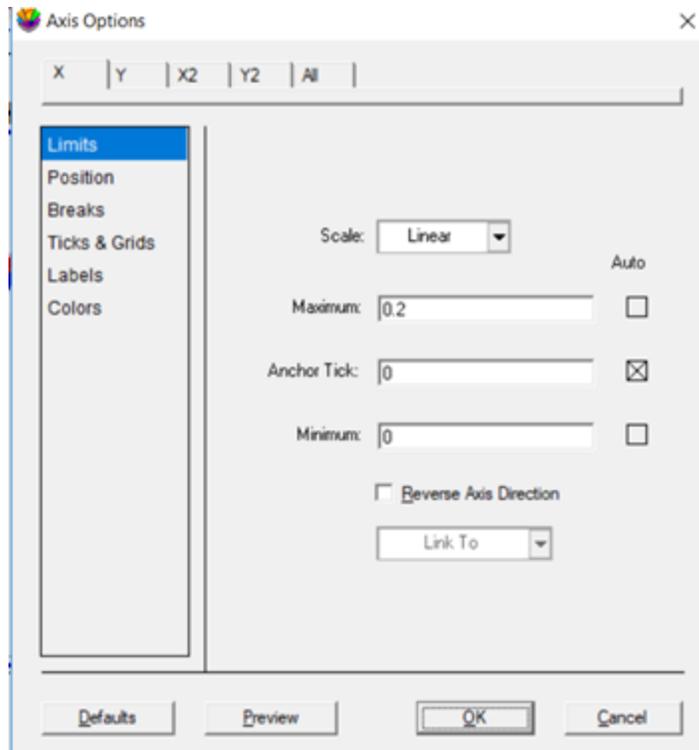
Formatting Plot:

1. Double-click on one of the lines in the plot legend. This will open the ‘Variable Settings’ window.



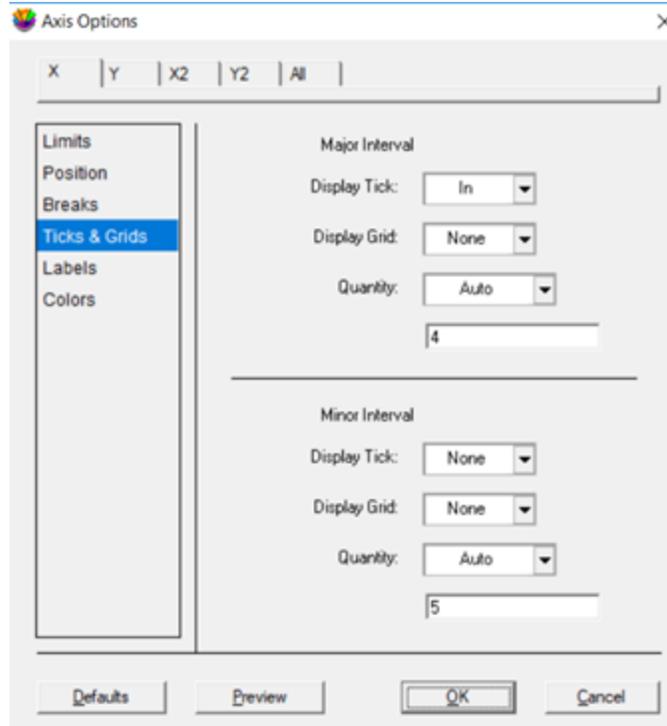
*If your graph does not have a legend, right-click on the plot and check “Display Legend”.*

2. Set “Show Markers” to “None”, “Line Style” to solid, and “Line Thickness” to 3.0. Click OK.
3. Repeat steps 1-2 for the other line.
4. Double-click on the x-axis line to open the “Axis Options” window. Under “Limits”, set “Minimum” to 0. If needed, do the same under the “Y” tab, located at the top of the window.



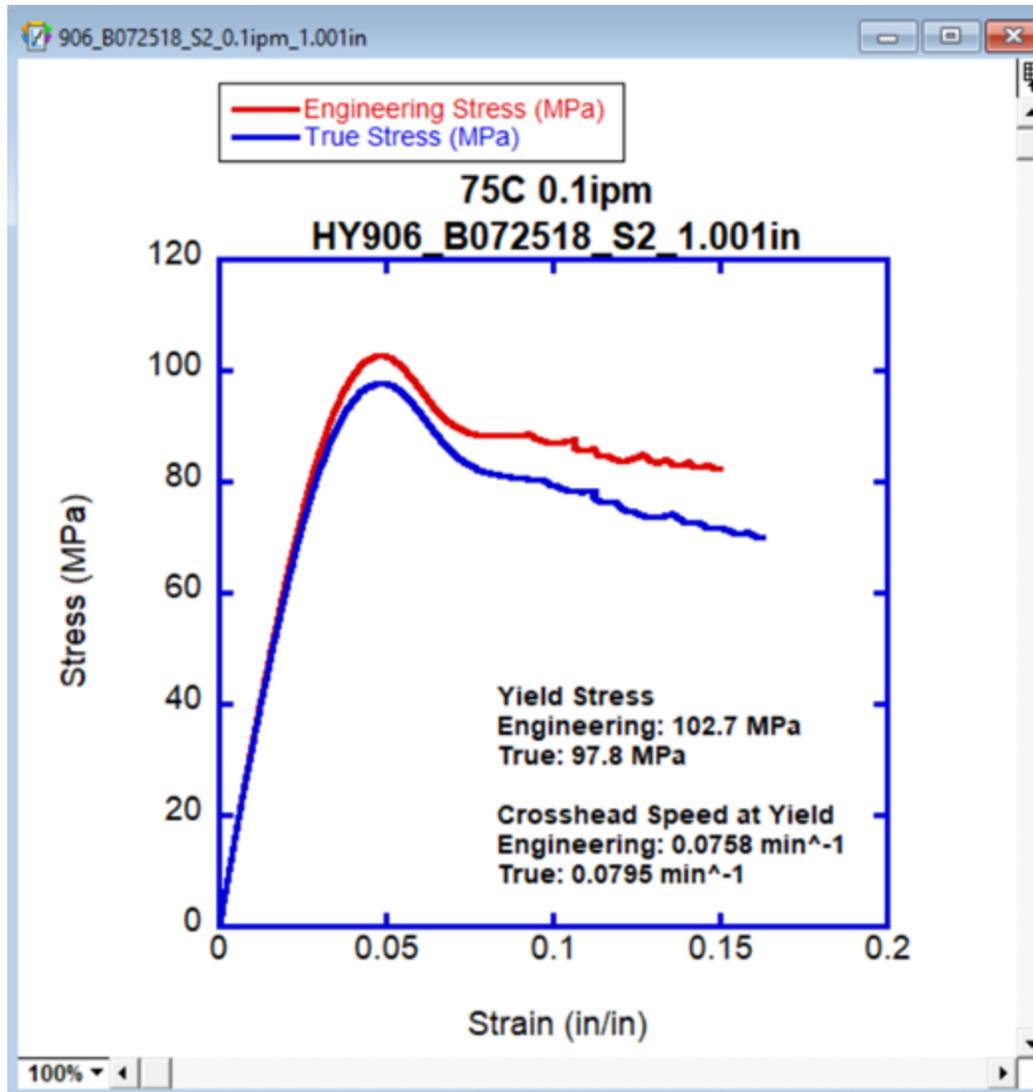
*“Limits” section in the “Axis Options” window.*

5. Go to the “Ticks & Grids” section on both “X” and “Y”, and make sure that the parameters are set to those pictured below.



*“Ticks and Grids” section in the “Axis Options” window.*

6. Under the “All” tab of the “Axis Options” window, go to “Thickness” and set both Tick and Axis Thickness to 3.0.
7. Double-click the x and y axis numbers and set “Size” to 18.
8. Double-click the axes labels and set “Size” to 18. Change the wording on these from “Engineering Stress (MPa)” and “Engineering Strain (in/in)” to just “Stress (MPa)” and “Strain (in/in)”
9. Double-click on the plot title, and change it to match the name of the test data file. Add more information if necessary. Set the font size to 20.
10. Double-click on the legend (but not on the legend lines) and set “Size” to 14.
11. Finally, name the plot file identically to the test data file using the keyboard command **Ctrl+R**.



*A complete, correctly formatted Instron compression plot*

12. Save the plot as a '.QPC' file, placing it with its corresponding test data files in a comprehensive archive. You do not have to save the data table; this can be extracted from the plot window at any time using the grid icon above the scroll bar.

## DSC Test Plotting

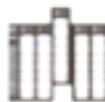
### Creating Data Table:

#### *Opening Data File*

1. Open the KaleidaGraph program.
2. Go to the control ribbon at the top of the window. Follow the directory: **File » Open**, and find the location of your test data. There will be many files to choose from: choose the one with the suffix “\_all”.
3. The “Test File Input Format” menu will open.
  - a. Set “Delimiter” to “Tab”, typing a single comma into the specification box.
  - b. Set “Number” to “1”
  - c. Set “Lines Skipped” to 0
  - d. Check “Read Titles”
  - e. Click OK

#### *Formatting Data Table*

4. Add two columns using the “Insert Column” symbol, located of the toolbar at the top of the data window.



*The “Insert Column” icon*

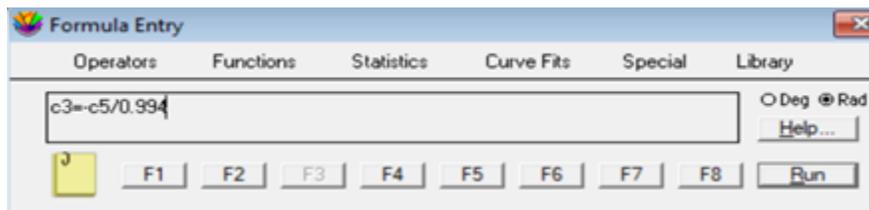
5. Name these columns “Heat Capacity (J.g\*C)” and “Heat flow (W/g)”.
6. Click the double-arrow symbol (pictured below), located above the scroll bar of the data table window, to reveal the variable labels for each column.



Click on this icon to expand or collapse the view of the column labels.

**Note: by highlighting an individual column, the variable labels are shifted. To avoid confusion, make sure that no columns are highlighted during any of the data operations.**

7. Enter the keyboard command **Ctrl+F** to open the “Formula Entry” window.



*“Formula Entry” window with correctly typed formula*

8. Enter the following formula into the text box:

$$[\text{Heat Flow (W/g)}] = [\text{Heat Flow (mW)}] / \{\text{mass of test sample}\}$$

The mass of the test sample should be available in the test’s data file name.

**Note : Column titles in brackets represent that column’s corresponding variable (i.e. ‘c8’).**

9. Click “Run” to apply the operation.

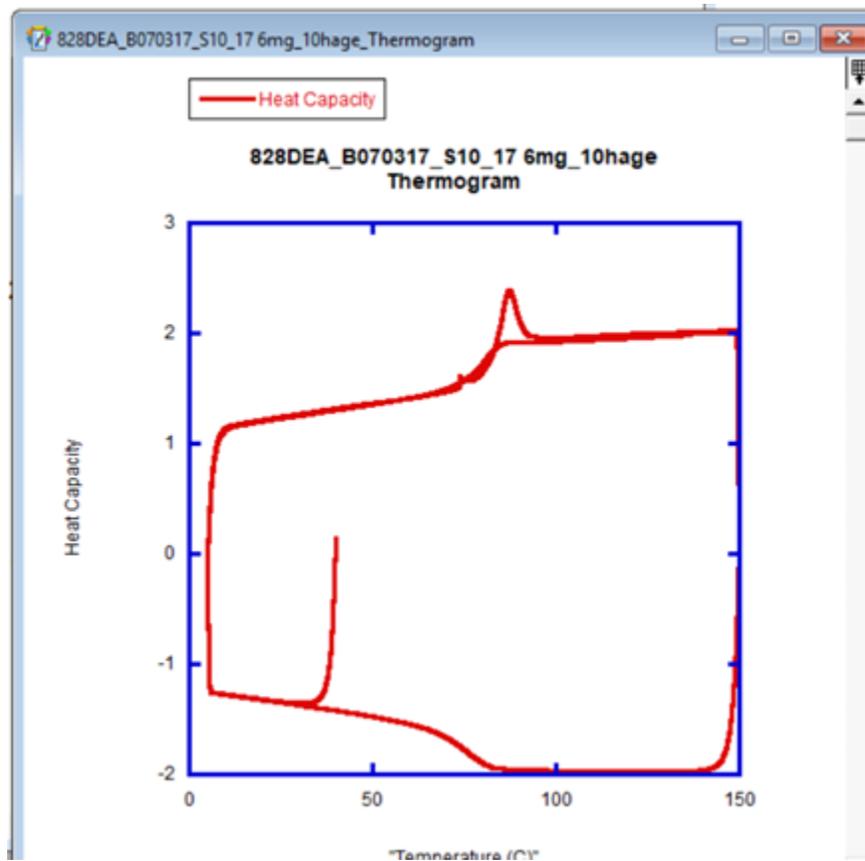
10. Enter the following formula into the Formula Entry box:

$$[\text{Heat Capacity (J.g*C)}] = [\text{Heat Flow (W/g)}] / 10 * 60$$

Click “Run”

### Creating Thermogram

1. From the control ribbon at the top of the window, follow the directory **Gallery » Linear » Line**.
2. Check “Temperature (C)” under “X”, and “Heat Capacity (J.g\*C)” under “Y”.
3. Click “Add”
4. Click “Plot”
5. A plot similar to the one pictured below should appear:



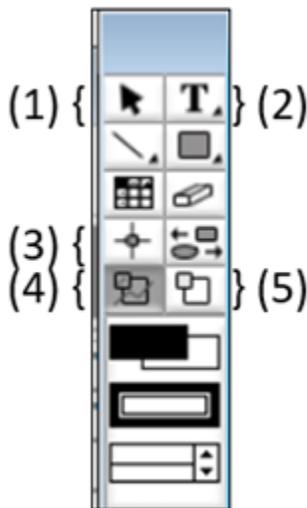
*A typical thermogram*

6. Name the plot file using the keyboard command **Ctrl+R**. Use the test data file name, adding a suffix of “\_thermogram”.

7. Save the plot as a '.QPC' file, placing it with its corresponding test data files in a comprehensive archive.

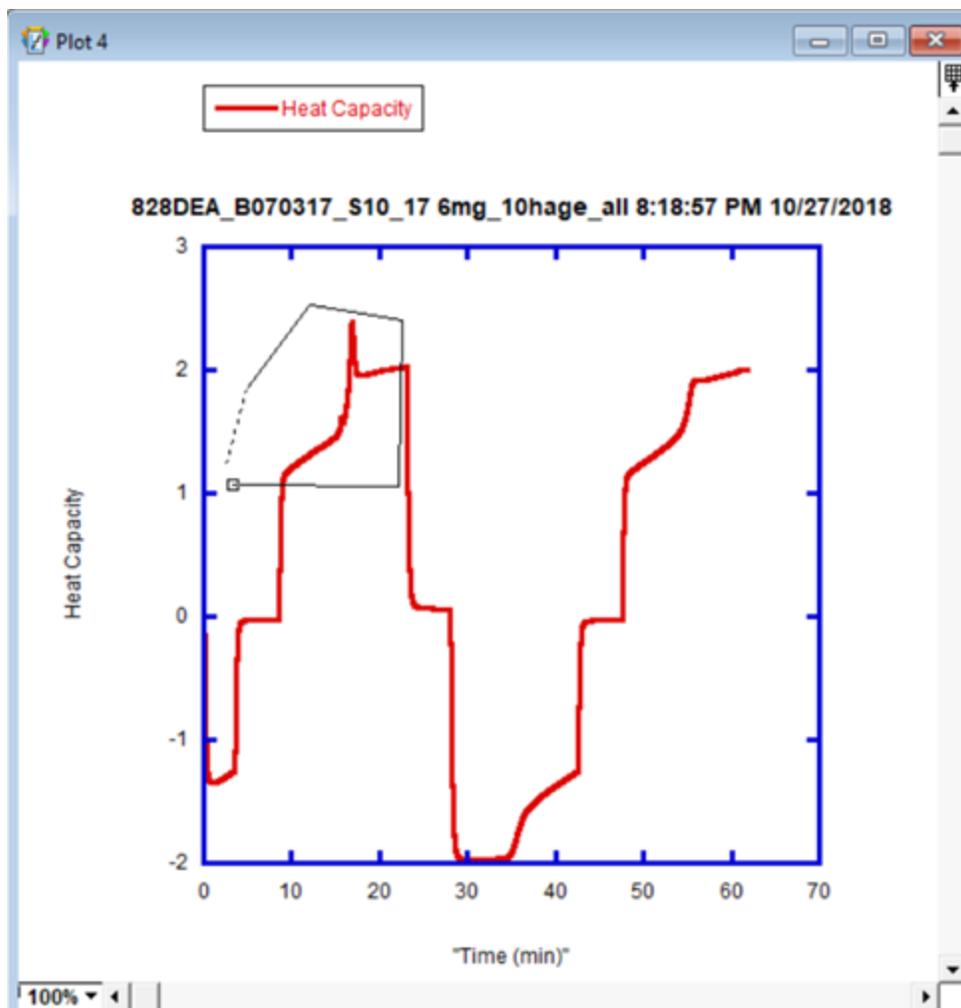
### Creating Heat Curves

1. Click back into the data table. From the control ribbon, follow the directory **Gallery » Linear » Line**.
2. Check "Time (min)" under "X", and "Heat Capacity (J.g\*C)" under "Y".
3. Click "Add".
4. Click "Plot".
5. A plot window and a plot toolbar (pictured below) should appear.



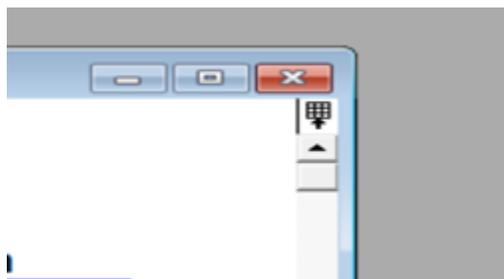
*Plot Toolbar: your default tool should be (1). Always click back to it after utilizing other tools.*

6. Using the masking function, (4), box in the nonlinear portion of the first peak (pictured below). Click on the plot to 'pin' the corners of the box, and connect back to the starting point in order to 'close' the box and initiate the masking.



*Isolating all non-linear data from the first peak using the masking function.*

7. Go to the data table. If some data is bordered in red, the masking successfully carried over. If not, click on the grid icon on the upper right corner of the plot window to refresh the table-plot link.



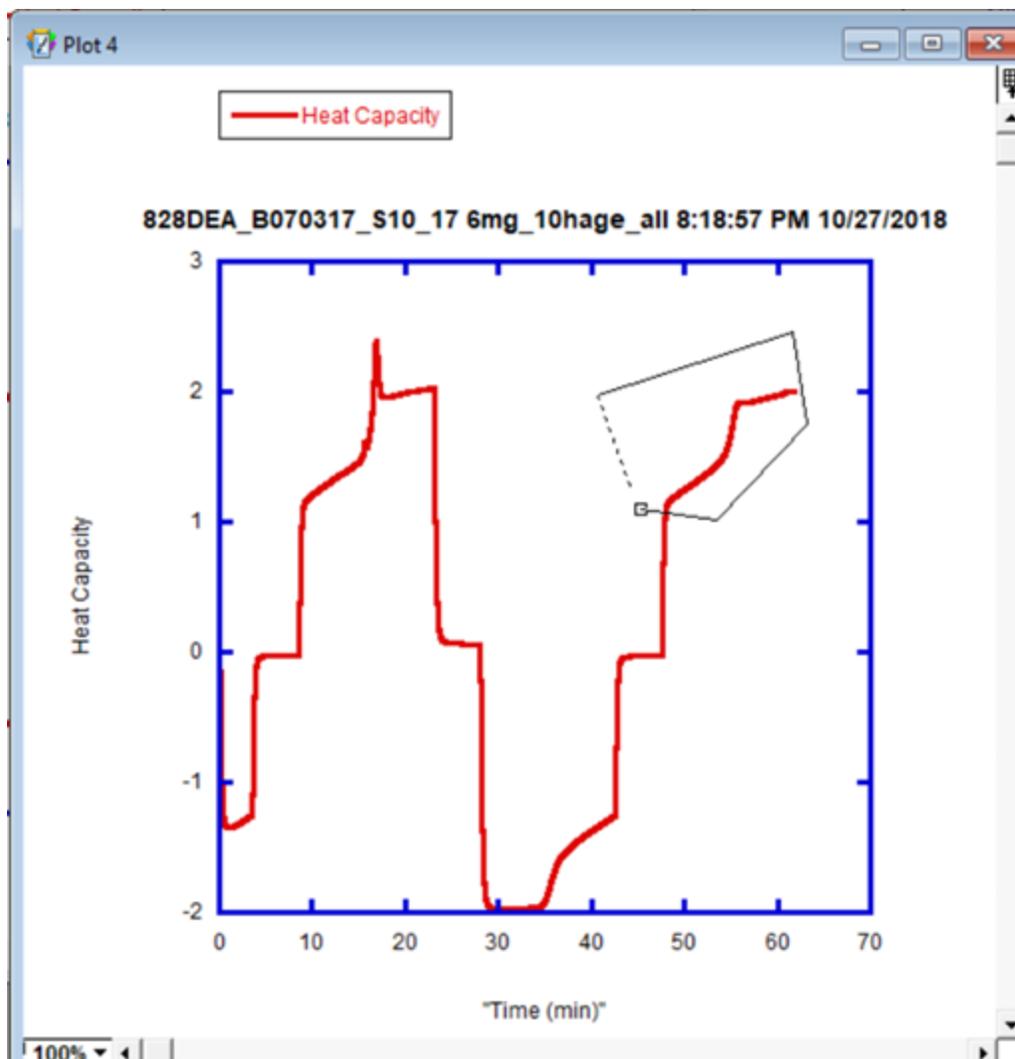
*Click the grid icon to refresh the plot-table link.*

8. Highlight all unmasked data from the data table, and copy to a new data table window.  
To open a new data table window, follow the directory **File » New**.

	Heat Capacity	"Heat Flow (mW)"	"Delta T (µV)"	"Delt...ro (µV)"
	C3	C4	C5	C6
2671	1.0174	-2.9844	-11.217	38.932
2672	1.0211	-2.9952	-11.261	38.982
2673	1.0248	-3.0062	-11.309	39.036
2674	1.0285	-3.0169	-11.356	39.090
2675	1.0313	-3.0252	-11.399	39.143
2676	1.0341	-3.0335	-11.441	39.195
2677	1.0372	-3.0423	-11.484	39.250
2678	1.0403	-3.0516	-11.525	39.308
2679	1.0418	-3.0561	-11.562	39.350
2680				

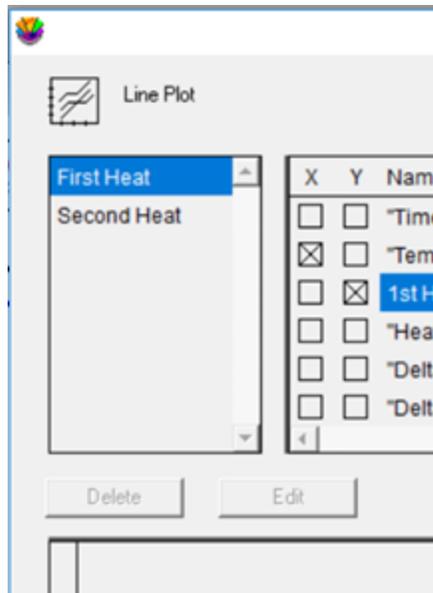
*Highlight and copy the data from all columns, starting from where the masking ends and finishing where it begins again.*

9. Name the new data table “First Heat” using the keyboard command **Ctrl+R**. Rename the “Heat Capacity” column “First Heat Capacity (J.g\*C)”
10. Repeat steps 3-6 for the second peak of the heat capacity/time (pictured below), but instead naming the new data table “Second Heat” and the “Heat Capacity” column title “Second Heat Capacity (J.g\*C)”



*Isolating non-linear data from the second peak.*

11. Click into the First Heat data table. From the control ribbon, follow the directory **Gallery** » **Linear** » **Line**.
12. From the "First Heat" tab, check "Temperature (C)" under "X", and "First Heat Capacity (J.g\*C)" under "Y".



*Information from both the “First Heat” and “Second Heat” data tables can be put into a single plot.*

13. Click “Add”.

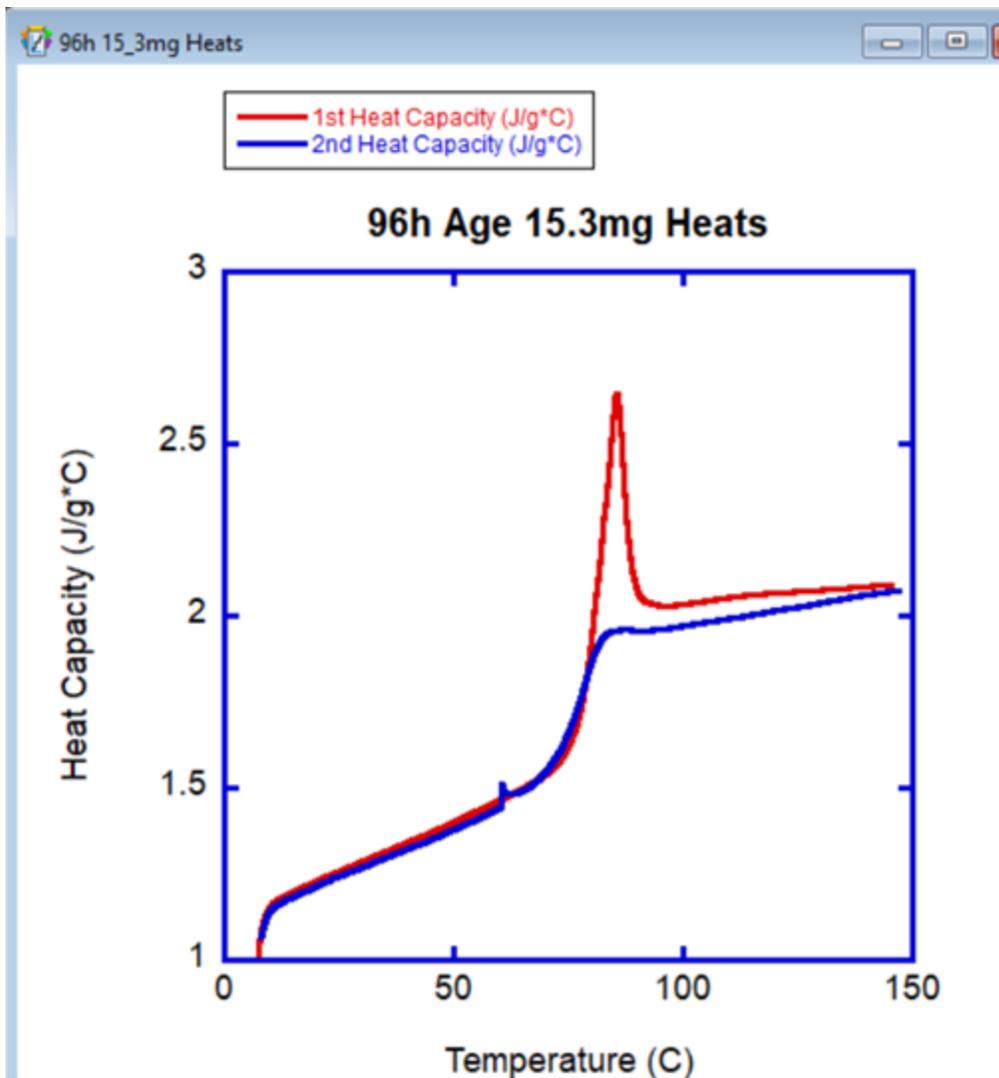
14. From the “Second Heat” tab, check “Temperature (C)” under “X”, and “Second Heat Capacity (J.g\*C)” under “Y”.

15. Click “Add”.

16. Click “Plot”

### Formatting Plot

1. See the “Formatting Plot” segment of the Instron Plotting Manual. Your complete plot should look like the one pictured below:



*A complete, properly formatted DSC test plot.*







