Pain-Free Melting Point Determination

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Stanford Research Systems

Who is Stanford Research Systems?

- In business since 1980
- Full catalog is over 200 pages
- Famous for first digital lock-in amplifier
- Successful quadrupole mass spectrometer (RGA)
- Now makes 3 kinds of melting point apparati

Introduction

Student grade melting point apparatus

Integral RTD thermometer

Microprocessor controlled temperature ramps

PID gives fast preheats without overshoot

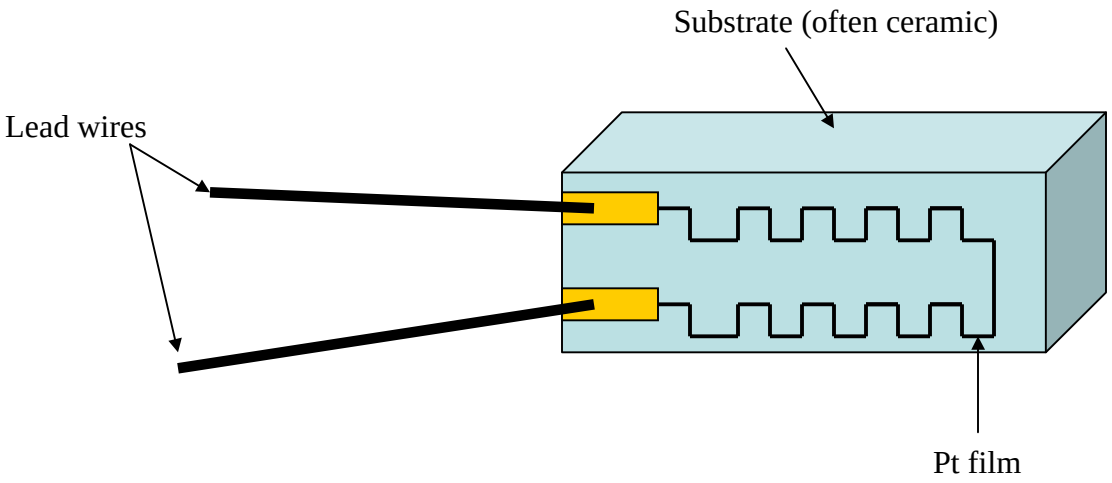
Tube Tapper (integral capillary packing device)

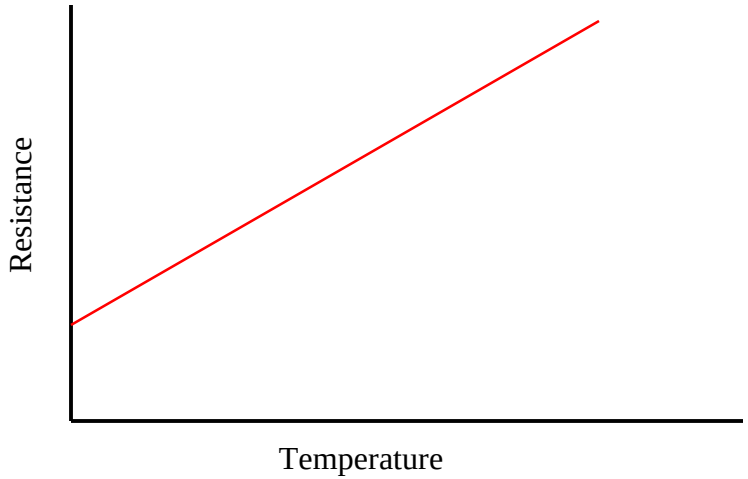
Easy to clean, maintain

Price (single unit) : $790

What is an RTD?

Resistance Temperature Detectors (RTDs) are simply materials whose resistivity changes as a function of temperature.





What is PID?

P.I.D. control allows tight temperature control of the oven using 3 types of error correction:

Proprotional – Make a correction term proportional to the error

Integral – Make a correction term that integrates the error with respect to time

Derivative – Make a correction term that differentiates the error with respect to time

Correction = $Pe(t) + I\int e(t)dt + D\,de/dt$
(where e(t) is the error signal with respect to time,
P, I, and D are constants)

Temperature Measurement

MEL-TEMP

Liquid in-glass (often Mercury)

Accuracy typically +/- 2 degree

Resolution typically 0.2 degree

Note that the accuracy of the thermometer isn't really the accuracy of the MelTemp. The device must be calibrated against melting point standards to give a slope and offset correction to the thermometer reading.

DigiMelt

RTD accuracy is typically
± 0.6 degree C <200 C
± 1.0 degree C >200 C

Resolution is 0.1 degree C

SRS takes care of calibration for you. We calibrate every unit we sell to our stated accuracy.

Temperature Measurement

MEL-TEMP

500 C is reachable, thermometer often only goes to 400 C

Thermometer can easily be broken or taken!

DigiMelt

Temperatures > 260 C disallowed

RTD is never missing

RTD is difficult to destroy

Temperature Control

MEL-TEMP

Open Loop Variac: No feedback
Student is the controller
Typical student is a poor controller
Overshoot leads to fanning the block
which leads to breakage

DigiMelt

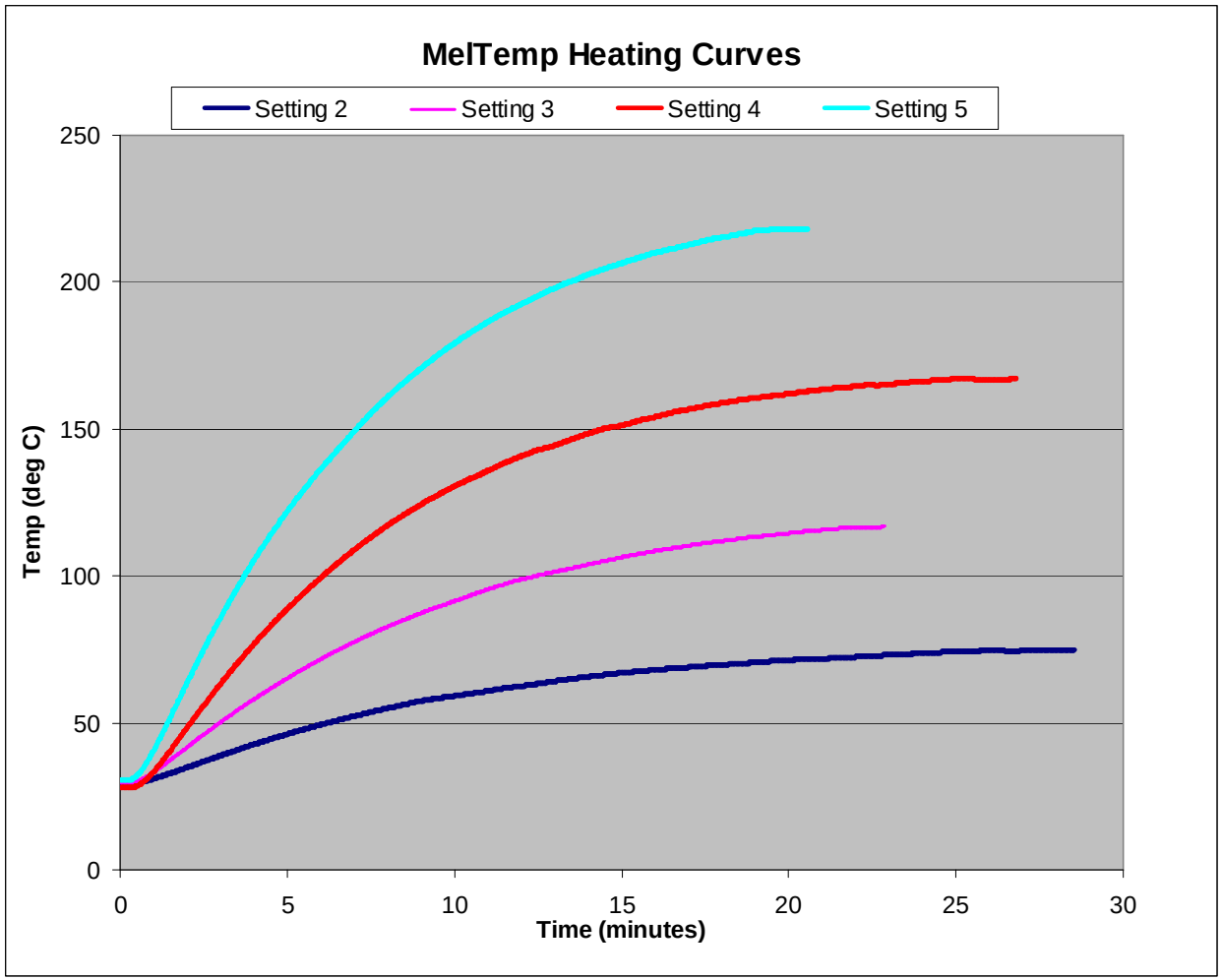
Full PID control with RTD feedback

Microprocessor lets students focus on their samples

Ramp rates of 0.5, 1, 2, 5 C

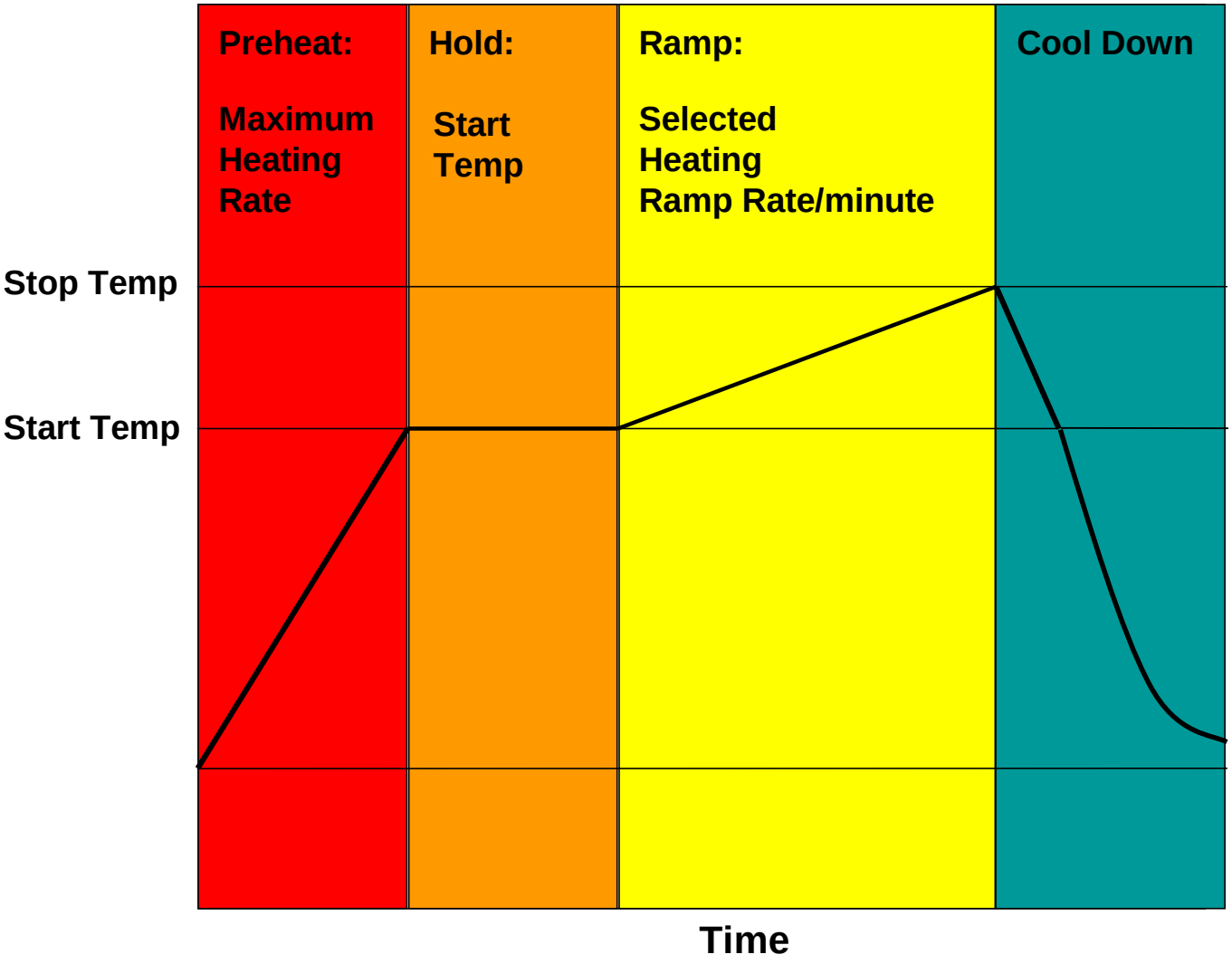
Oven turns off after 30 minutes of idle

MelTemp Delivers a Set Power, not a Set Temperature



When students turn up the knob on the variac, they often don't realize that the delivered ramp is very non-linear with the power setting. This often leads to students turning up the variac too high and overshooting their target temperature. This wastes time and frustrates many students.

DigiMelt Follows a User-Defined Temperature Program



The student uses the keypad to enter a start, stop, and a rate. The microprocessor quickly heats the oven to the desired start temperature (typical time to 100 C is 2.5 minutes). After stabilizing at the start temperature, the microprocessor begins the melting ramp at the specified rate. Once the stop temperature is reached, the oven goes into cool down.

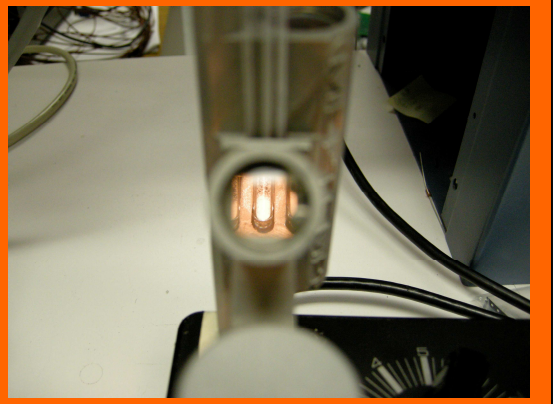
Sample Viewing

MEL-TEMP

Hard to see all three tubes

Light reflects from sample to eye via window, lens

Student must switch between viewing sample and viewing thermometer

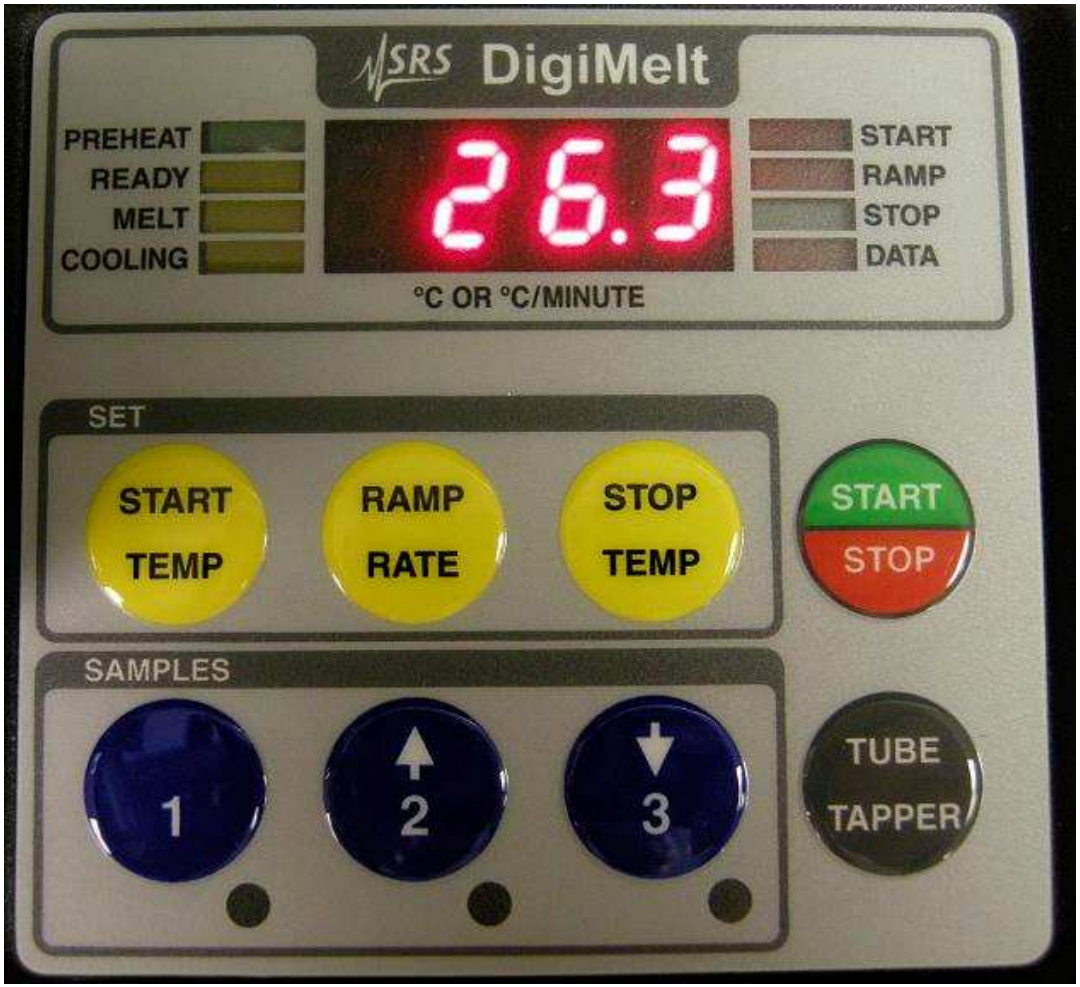


DigiMelt

Large lens allows view of three samples simultaneously

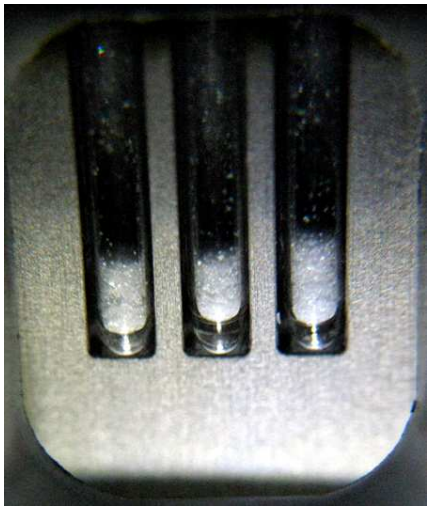
Student records critical temperatures (onset, meniscus, clear point) by touching keypad

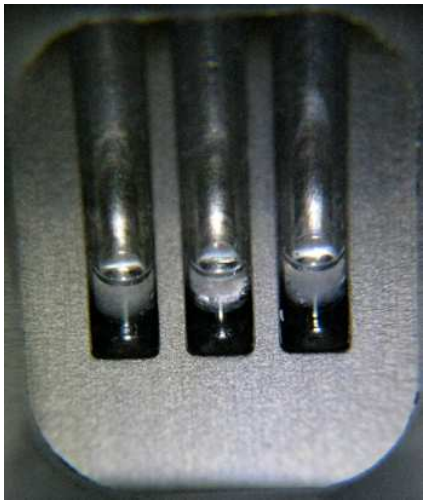
Using DigiMelt : Enter Settings



The keypad makes setting up a melt program very easy. The students push the yellow ‘START TEMP’ and then use the blue buttons with arrow keys to set the starting temperature. The students do the same for the ‘RAMP RATE’ and ‘STOP TEMP’ keys. Once the parameters are entered, they push the ‘START/STOP’ button and the microprocessor takes care of heating the block.

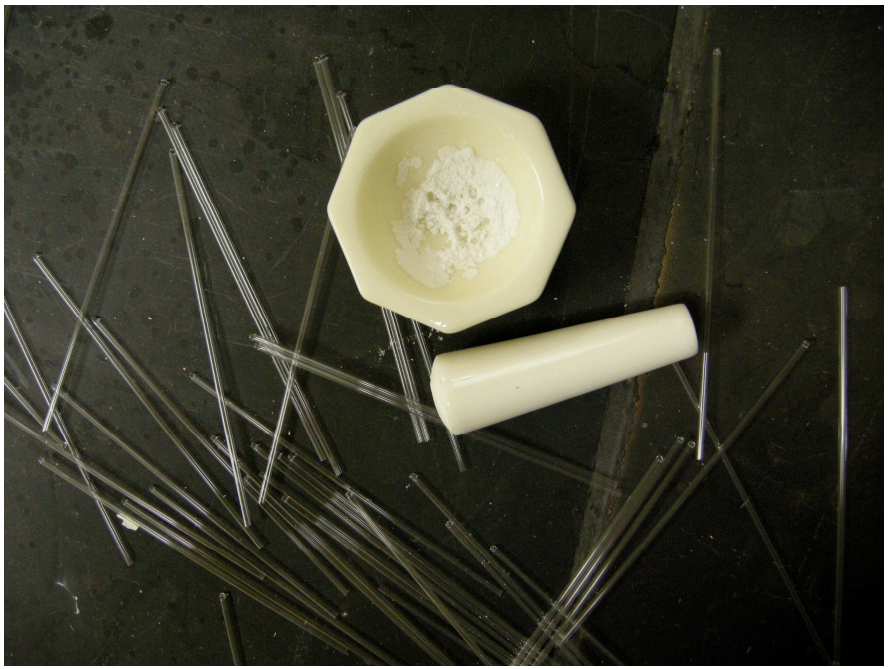
Using DigiMelt to Record Data





Students can ‘flag’ critical points in the melting process with the blue buttons on the keypad. Above are views through the lens showing a sample of vanillin during pre-heat, at the meniscus point, and at the clear point. After the melt is over (cooling LED is lit), the data are retrived by pushing the buttons again.

Packing Capillaries



When students tap sample into capillaries, lots of capillaries end up on the floor, lots of capillaries are broken, wasted

DigiMelt

Integral cell phone vibrator motor "Tube Tapper"
Capillaries can be packed 3 at a time
Students can get 3 samples with the same sample height

Maintenance

MEL-TEMP

Incandescent bulb will burn out

Mean time between failures
is about 1000 hrs

Broken thermometer = $200 (25%)

DigiMelt

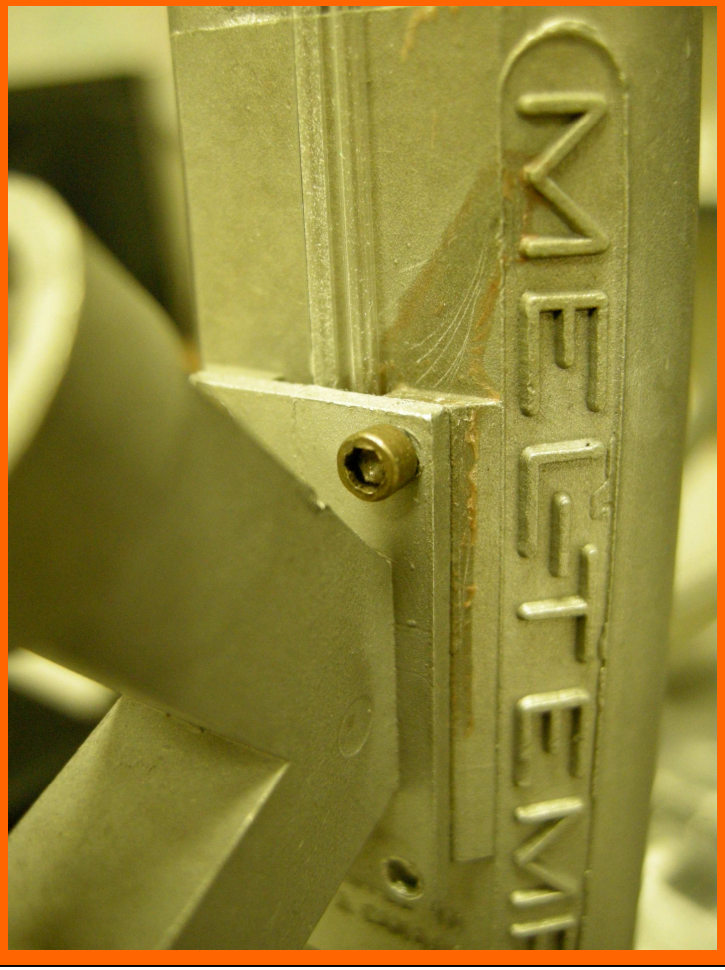
White LED has >100,000 hours
mean time between failures

RTD is well-protected

Maintenance

MEL-TEMP

Capillary holder uses socket head cap screws or other screws



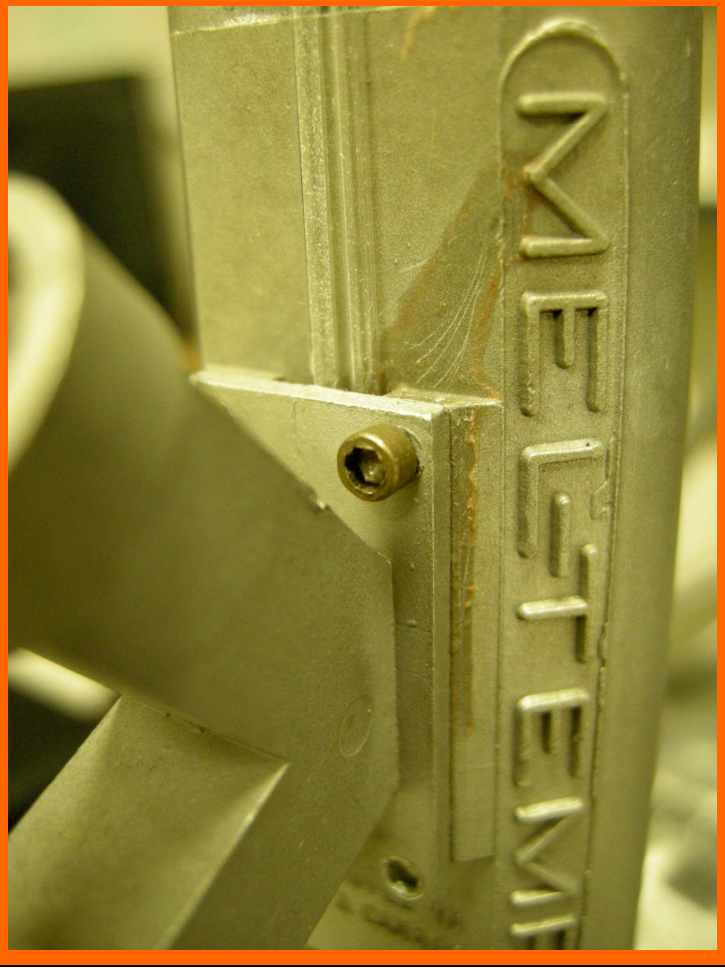
DigiMelt

No tools required

Maintenance

MEL-TEMP

Capillary holder uses socket head cap screws or other screws



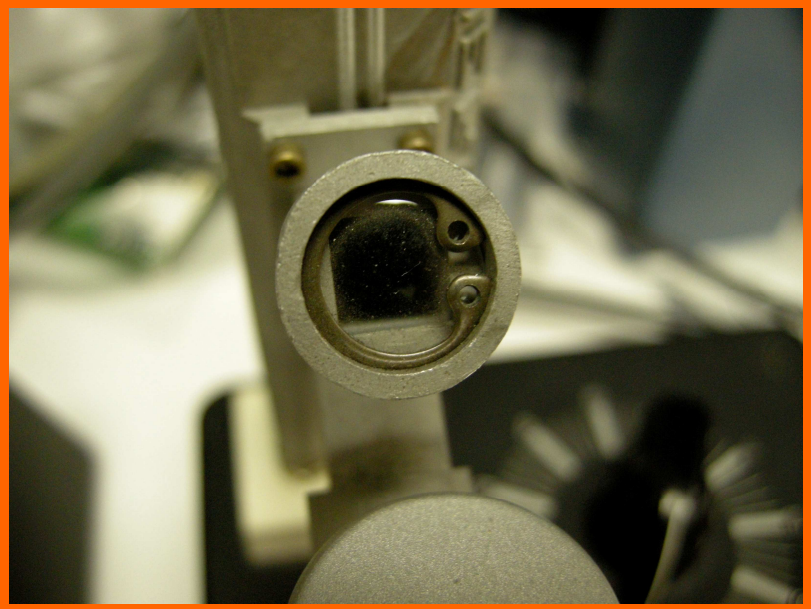
DigiMelt

No tools required

Maintenance

MEL-TEMP

Lens, window held with snap rings



DigiMelt

No tools required

Safety Certification

Student safety is paramount in our design. SRS had the entire DigiMelt design independently verified to comply with UL safety regulations by MET labs. Every unit undergoes safety testing to maintain our certification.

Summary

MEL-TEMP

Students know there's a better way

Wasted time
Hard to use equipment
Broken thermometers

FRUSTRATION

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DigiMelt

Students focus on the sample:
THE REAL EXPERIMENT
Faster labs
Safer labs
Less waste
Lower cost of ownership