

# Ice Nucleation Controller – Manual

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## Abstract

Ice Nucleation Controller is a system used to perform high precision controlled ice nucleation experiments. System experiment temperature range is from +10°C to -30°C. A specified temperature gradient is held throughout experiments using a cooling circuit consisting of a closed water loop and a PID controlled pair of peltier elements. Temperatures are measured using a FLIR A655sc thermal camera and run-time calibrated using a thermistor in a fixed calibration cavity. Freezing temperatures for each sample are estimated using extracted temperature gradients and can be used for further post processing.

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# 1 Safety Information

## 1.1 General safety instructions



### DANGER

Danger to life or serious injury can occur when live parts are touched. Do not touch or modify electrical installations without proper training or guidance.



### WARNING

Danger to life, serious injury, or equipment damage can occur due to incorrect handling of equipment. Only trained personnel may service and modify system.



### CAUTION

Risk of skin irritation or burn when handling gallium. Always handle with care in both solid and liquid form.



### CAUTION

Risk of adverse health effects from long-term use and high humidity build-up if not ventilated. Always operate system in properly ventilated room.

### NOTICE

Risk of damage to sensitive equipment and/or loss of calibration due to incorrect handling. Always handle equipment marked SENSITIVE with care.

### NOTICE

Risk of damage to camera lens or calibration due to incorrect handling. Under no circumstance touch or affect camera lens as this can damage readings. Always transport camera with lens-cover attached.

### NOTICE

Risk of damage to cooling base fittings and seals due to tube-connection strain. Exert caution when manipulating tubes and cooling base.

## 2 Overview

### 2.1 Components

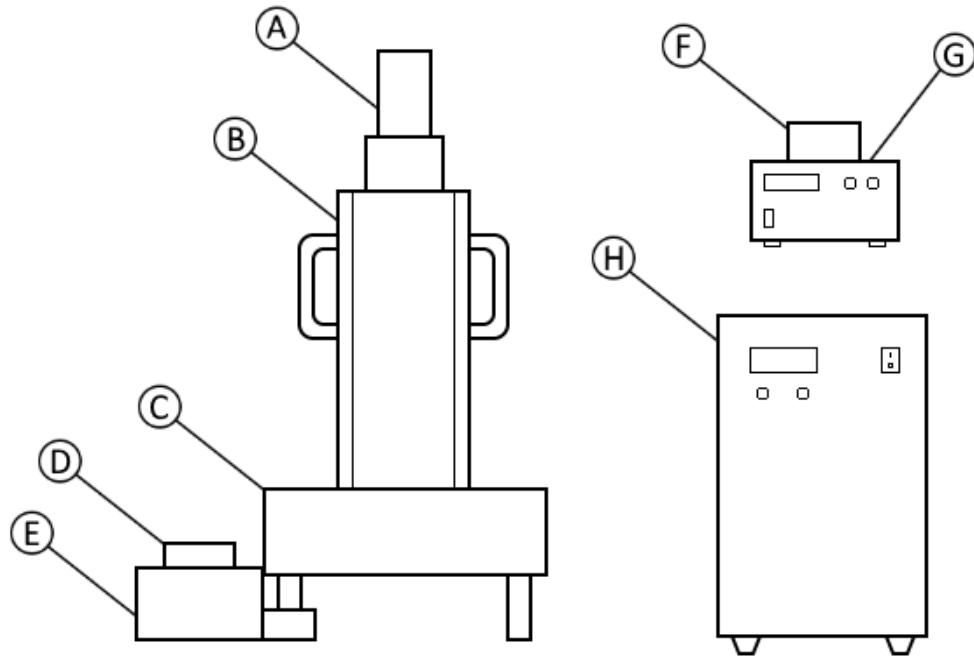


Figure 1: System Components.

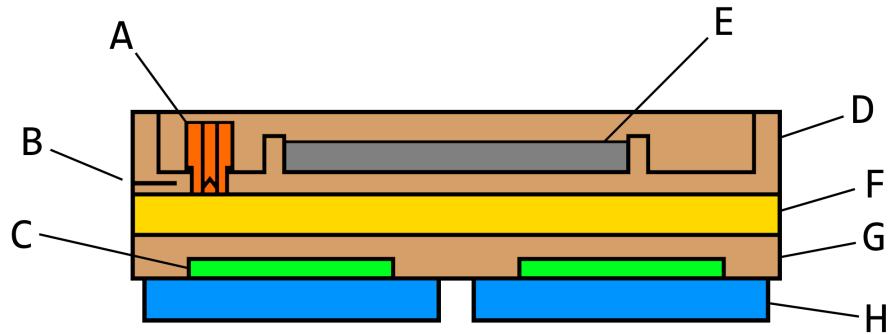
### 2.2 Description

| Component | Description          | Notes                              |
|-----------|----------------------|------------------------------------|
| A         | Thermal Camera       | FLIR A655sc (SENTIVE) [1]          |
| B         | Camera Tower         | -                                  |
| C         | Cooling Base         | Protruding Sensor Cables (SENTIVE) |
| D         | cDAQ Module          | NI 9219 and cDAQ-9171 [4] [5]      |
| E         | Sensor PCB Housing   | Protruding Sensor Cables (SENTIVE) |
| F         | Peltier Power Switch | MCU and 4x SS Relay [8]            |
| G         | Power Supply         | PeakTech (P1580) [7]               |
| H         | Water Cooler         | Alphacool Eiszeit 2000 Chiller [6] |

### 3 System Description

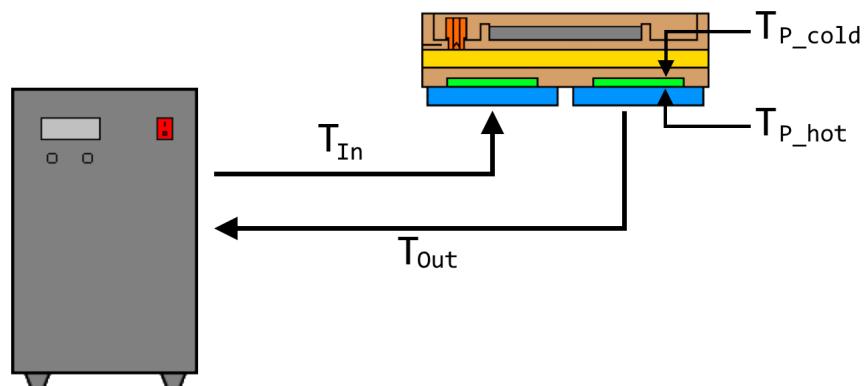
#### 3.1 Cooling Base

Temperature control is achieved



| Component | Description        | Notes  |
|-----------|--------------------|--|
| A         | Fix-point Cavity   | -  |
| B         | Thermistor Probe   | TE Connectivity 2.252 kOhm [2]                       |
| C         | Peltier Elements   | QuickCool HighTech 270W [3]                          |
| D         | Top Copper Base    | -  |
| E         | Gallium            | Above 99.99% purity                                  |
| F         | Vapor Chamber      | -  |
| G         | Bottom Copper Base | -  |
| H         | Water Cooler       | Aqua Computer cuplex kryos NEXT sTRX4 Full Cover [6] |

## 3.2 Cooling Circuit



Gallium, Water loop, peltier elements, PID controlled

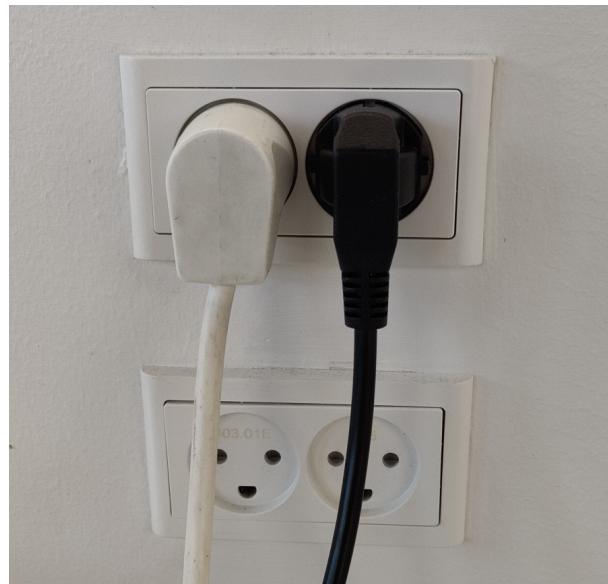
## 3.3 Temperature Measurements

Thermal Camera, Fixed Cavity Calibration, thermistor + circuit + calibration ...s

## 4 Procedure

### 4.1 Starting the system

1. **Plug in Main Power Cable** 230VAC mains voltage.



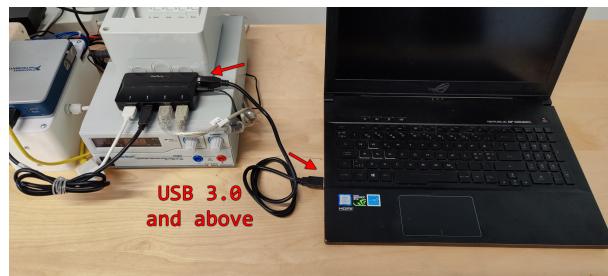
2. **Start Water Pump** It should beep once, if continuously beeping see 6.4.



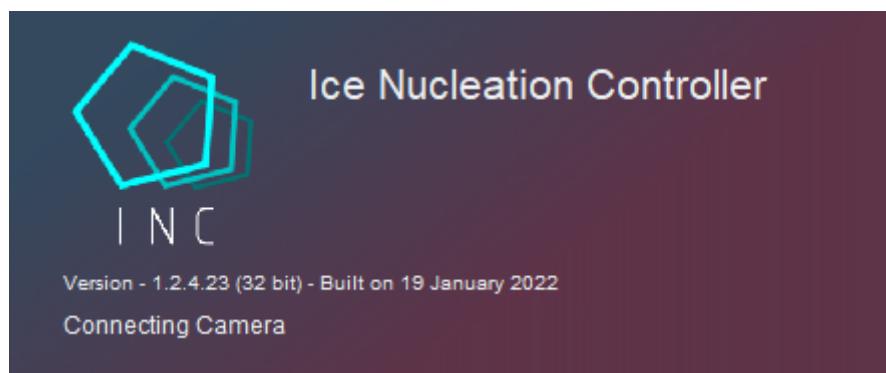
**3. Start Power Supply** It will write a sequence of start messages, once completed it should say either [O P OFF] or a low voltage and current (0 to 3 V).



**4. Connect PC to USB-hub** Only use USB 3.0 and above.

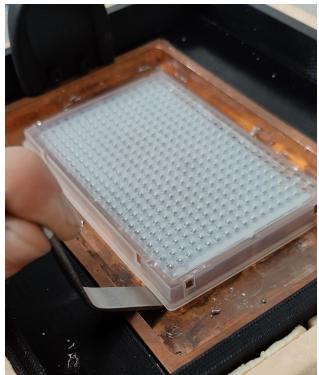


**5. Start INC Software** If everything is correctly setup no errors should appear.

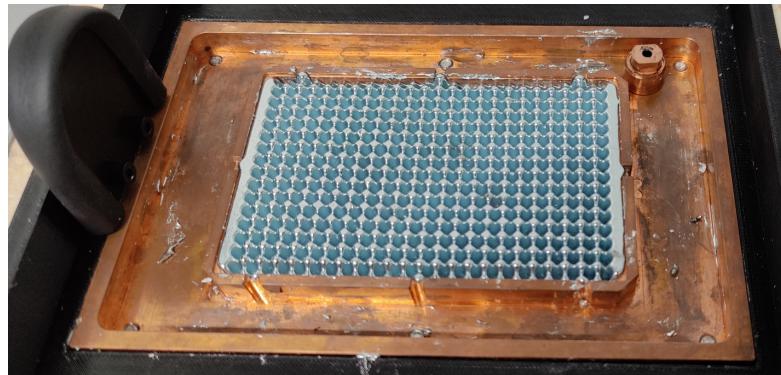


## 4.2 Preparing Sample

1. Remove Old PCR Plate use crowbar tool to remove previous PCR plate if present.

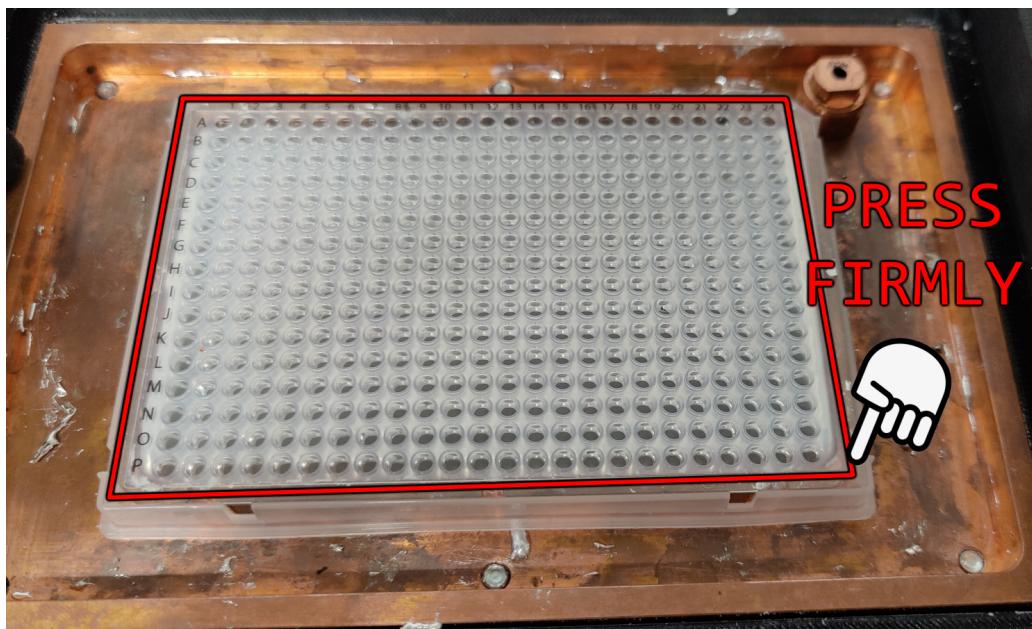


(a) Crowbar Tool

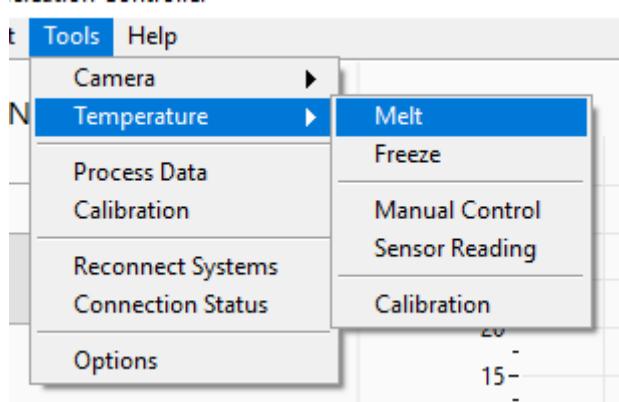


(b) PCR plate removed

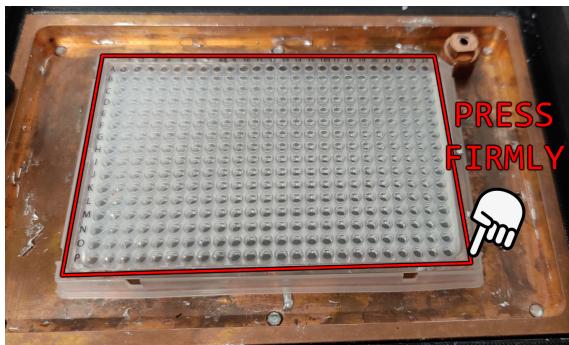
2. Insert New PCR Plate STERILE, make sure to press firmly on every corner and sides.



**3. Melt Gallium** with new PCR plate inserted [Tools] > [Temperature] > [Melt]. Wait until melting process is complete to continue.



**4. When Gallium is melted, firmly press on every corner of PCR plate** make sure PCR plate is firmly secured, pressing on each corner and side. Make sure to not press on the center of PCR plate as liquid gallium can spill out. See 6.2 in case of gallium spill.



(a) Press along edge

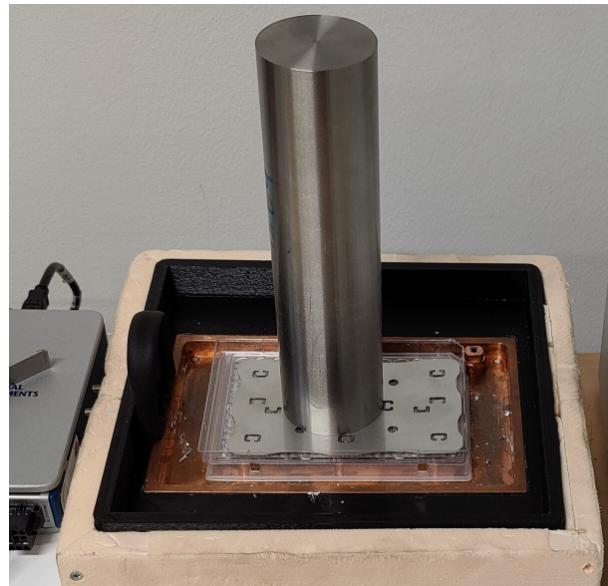


(b) DO NOT PRESS CENTER

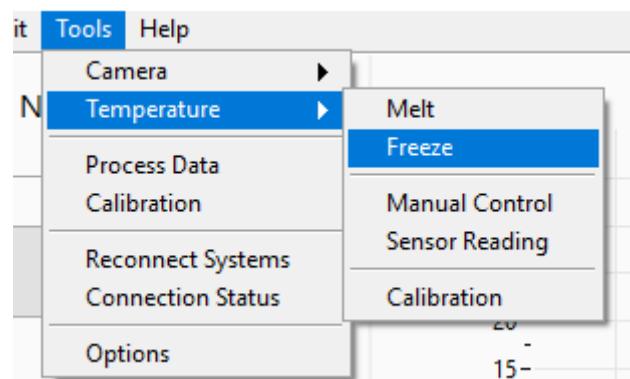


**DO NOT PRESS CENTER**

**5. Place Lid, Plate and Weight** STERILE on top of PCR plate to weigh down PCR plate during cooling process.



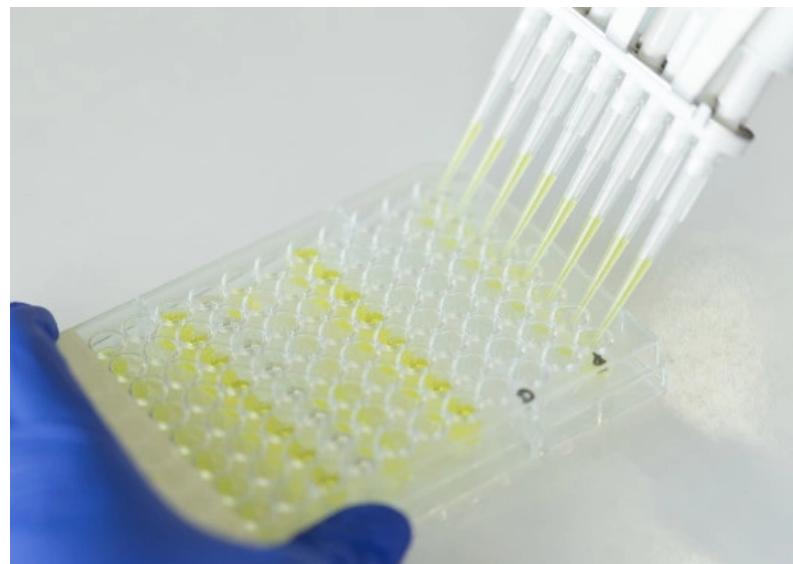
**6. Freeze Gallium** with `Tools > Temperature > Freeze` to ensure ideal thermal contact.



**7. Remove Lid and Weight** once freezing process is complete.



**8. Fill PCR wells with desired content ...**

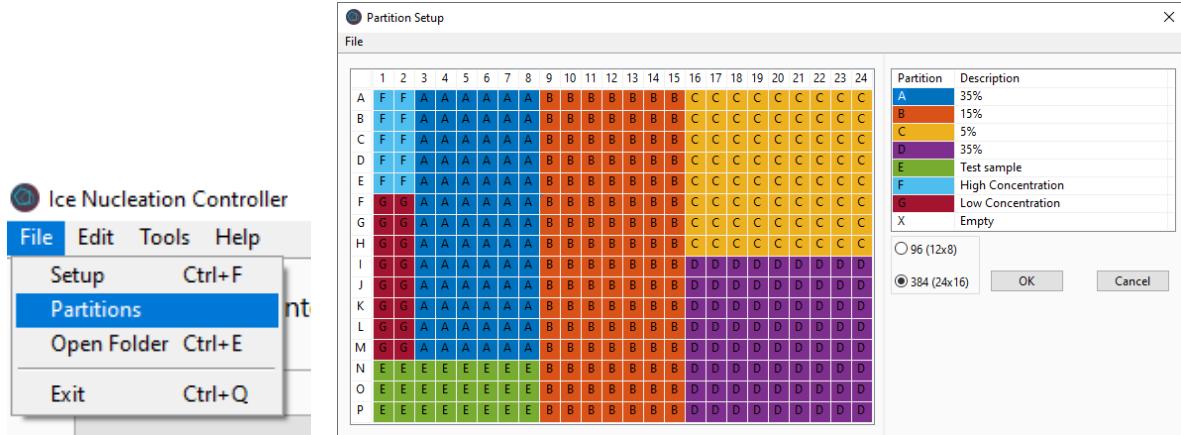


**9. Place Camera Tower on Cooling Base** make sure tower is firmly secured and pressed to the bottom.



## 4.3 Configure Partitions

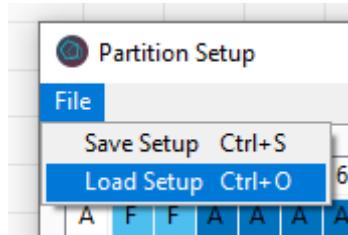
Open partition settings **Files > Partitions**.



(a) Open Partitions

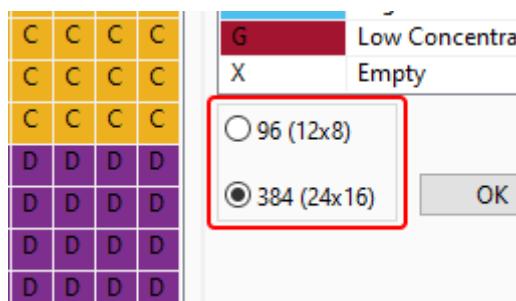
(b) Partition Menu

**Load Partition (Optional)** Load and edit previously created partition setup. If none are available, create a new Partition Setup.



### 4.3.1 Creating a new Partition Setup

**1. Select PCR size** Select between 96 and 384.

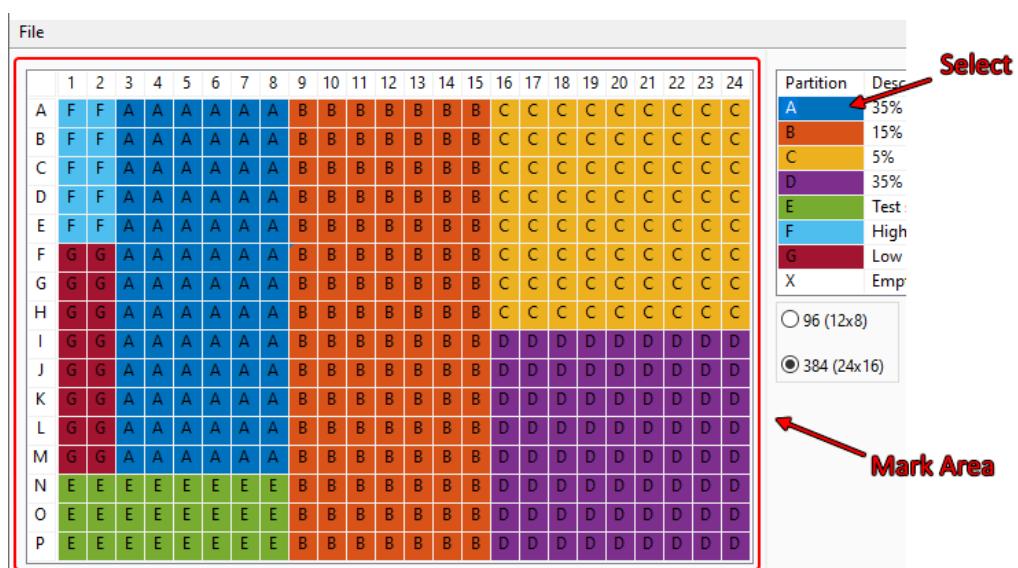


**2. Configure Partitions** Select partition and left click description to edit contents. Fill out as required.

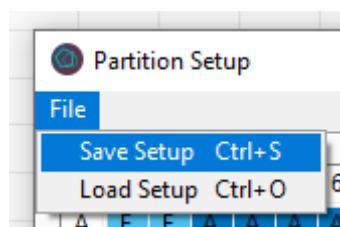
| 24 | Partition | Description        |
|----|-----------|--------------------|
| C  | A         | 35%                |
| C  | B         | 15%                |
| C  | C         | 5%                 |
| C  | D         | 35%                |
| C  | E         | Test sample        |
| C  | F         | High Concentration |
| C  | G         | Low Concentration  |
| X  |           | Empty              |

○ 96 (12x8)

**3. Mark Partitions** Select a partition (A, B, C, etc.). Mark an area of the PCR plate containing contents by left click and dragging. Fill out as required.

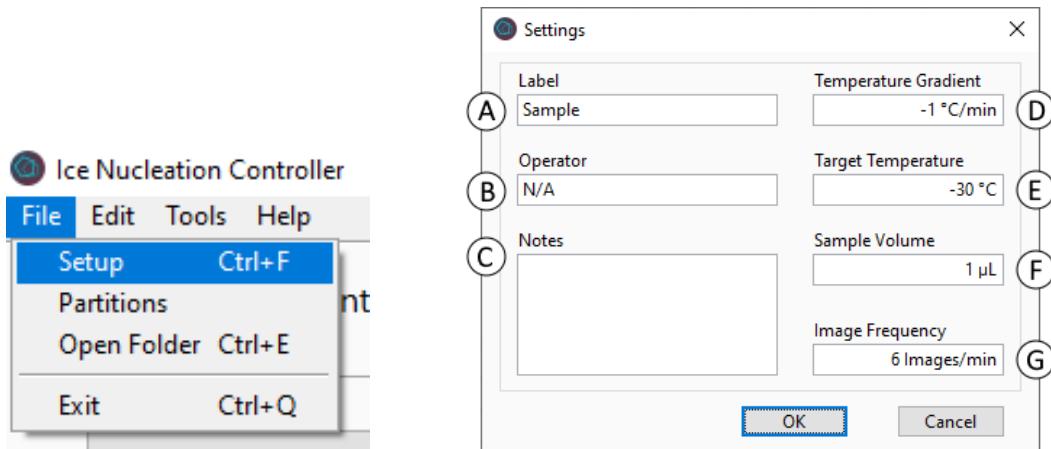


**4. Save Partition (optional)** Save created partition setup for future uses.



## 4.4 Configure Setup

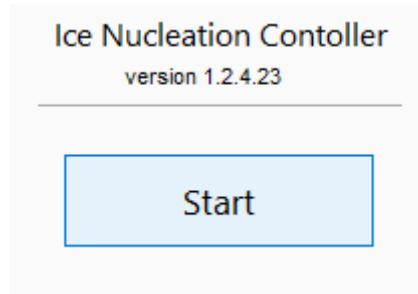
Open Setup settings **Files > Setup** and fill out.



| Description |                                  |
|-------------|----------------------------------|
| A           | Title of Experiment              |
| B           | Initials of operator             |
| C           | Any additional information       |
| D           | Target gradient of experiment    |
| E           | Ending temperature of experiment |
| F           | Volume present in each PCR well  |
| G           | Camera and data output rate      |

## 4.5 Start Experiment

Once previous steps are completed press the START-button to start the experiment. Avoid shaking or moving any parts of the setup while experiment is running.



## 5 Process Data

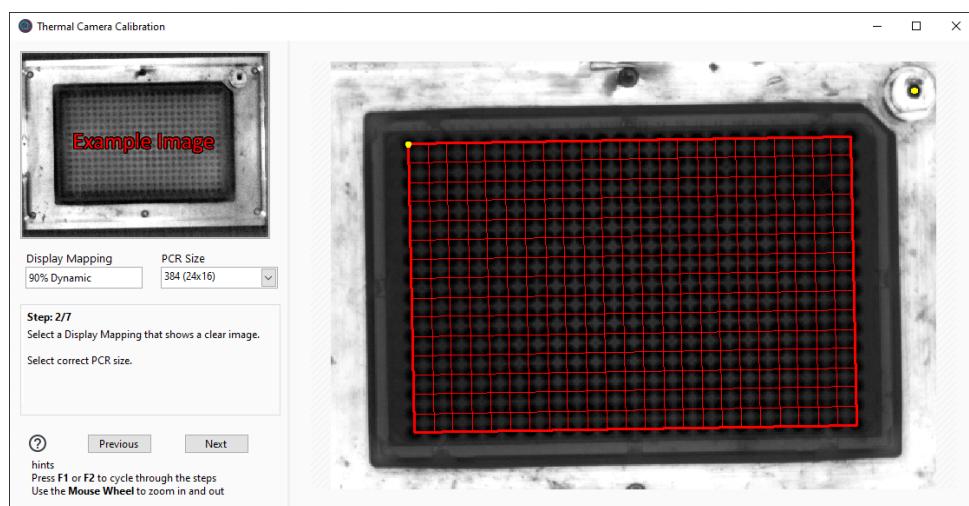
### 5.1 Thermal Camera Calibration (TCC)

System setup is susceptible to minor movement, which can affect camera feed positions. For every experiment a camera calibration must be performed. To perform a calibration open TCC **Tools > Calibration**.

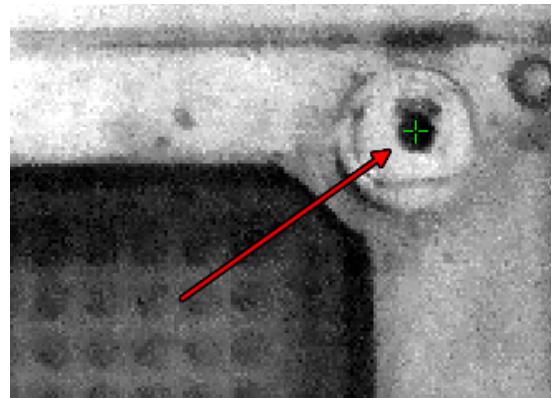


**1. Load Calibration Image** navigate to experiment folder and select any image (.../Data/Experiment\_Label/Images).

**2. Image Settings** select PCR size and modify Display Mapping until a clear image shows.

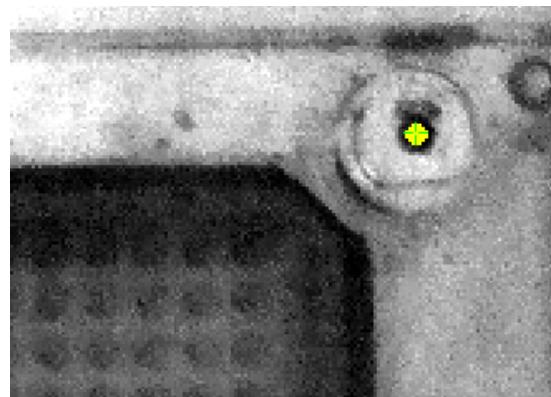


**3. Select Fix-point** Zoom in with mouse-wheel and left-click to mark the fix-point cavity.

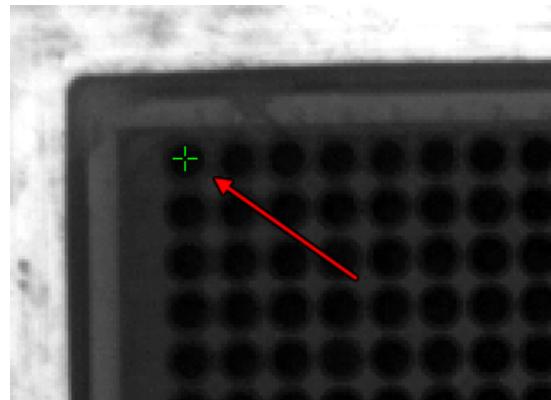


hint: position can be moved by left click and dragging pointer.

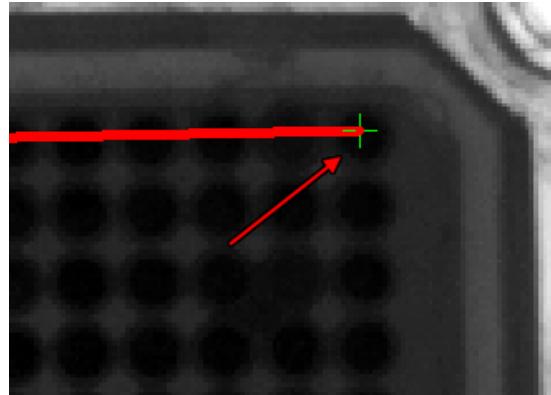
**4. Fix-point Radius** increase size until cavity is sufficiently covered by yellow pixels while still within cavity.



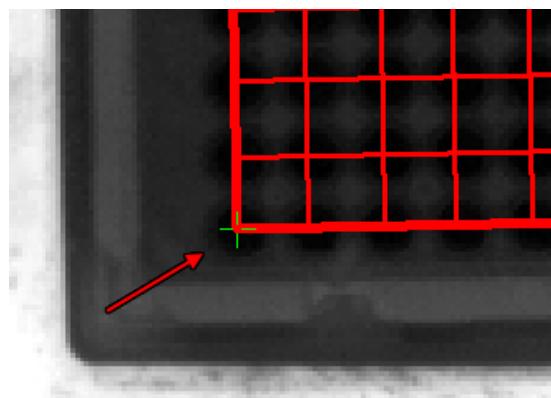
**5. Mark Top Left Corner** of PCR, zoom in with mouse wheel and left click on the top left well.



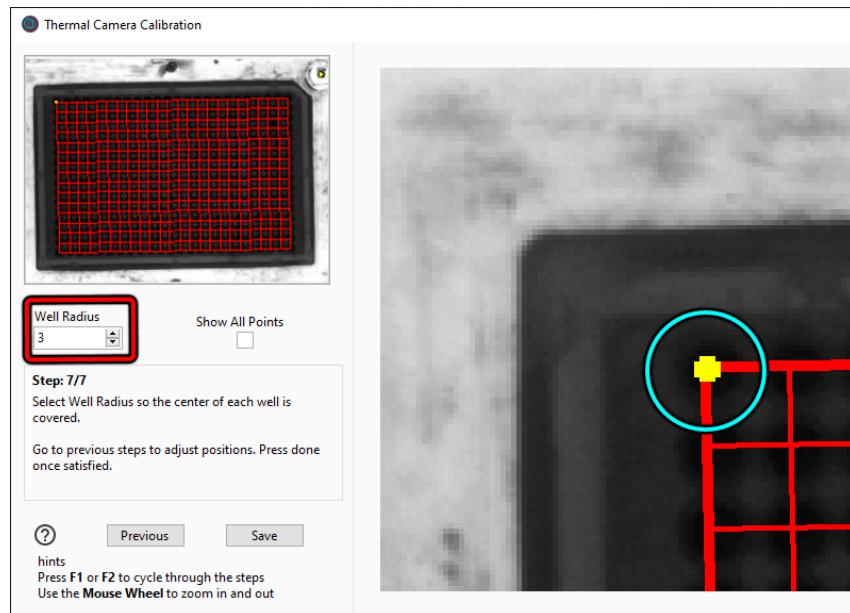
**6. Mark Top Right Corner** of PCR, zoom in with mouse wheel and left click on the top right well.



**7. Mark Bottom Left Corner** of PCR, zoom in with mouse wheel and left click on the bottom left well.

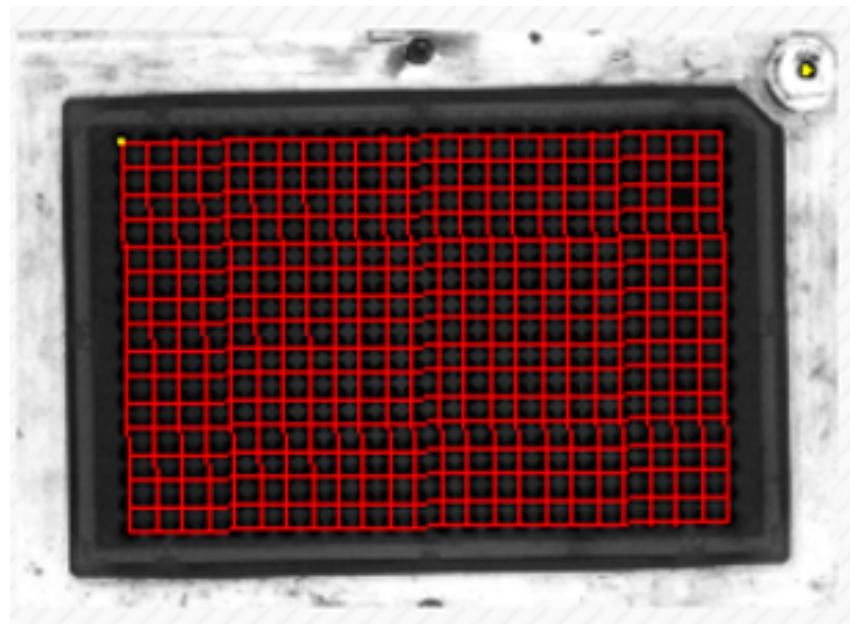


**8. Well Radius** increase size until well is sufficiently covered and within well boundaries. Check all grid points are within correct well position.



hint: enable  Show All Points to check measurement area of all wells.

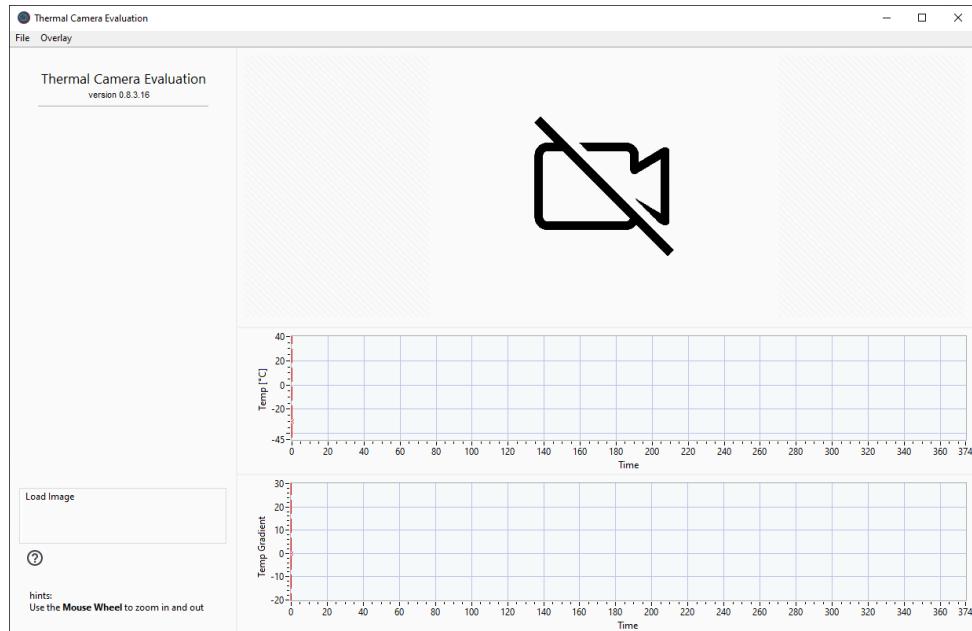
**9. Verify All Positions** are correct, navigate back to previous steps to alter positions until satisfied.



**10. Save Calibration** once satisfied with the calibration, save and exit software.

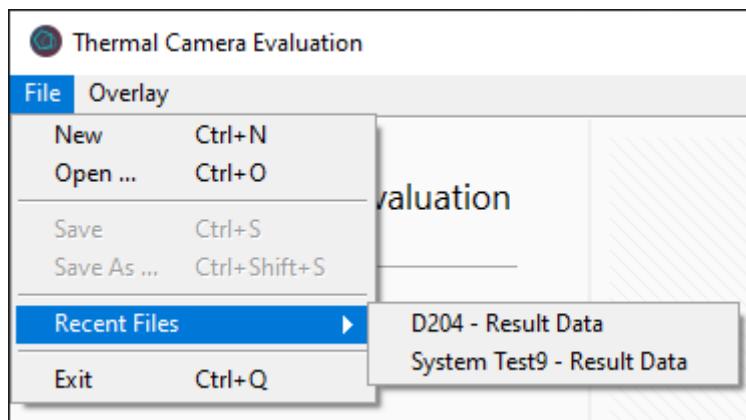
## 5.2 Thermal Camera Evaluation (TCE)

To process experiment data open TCE (**Tools**  $\gg$  **Process Data**).



### 5.2.1 View Processed Data

To open already processed data **File**  $\gg$  **Open** and navigate to an experiment folder containing processed data. Alternatively use **File**  $\gg$  **Recent Files**  $\gg \dots$  and select a previously opened experiment.

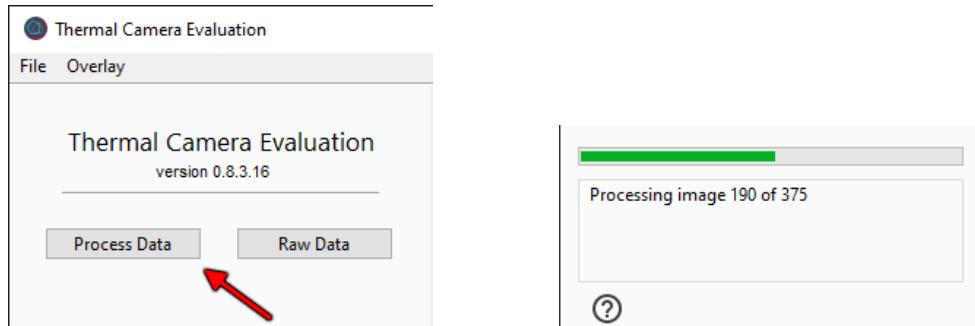


### 5.2.2 Process New Data

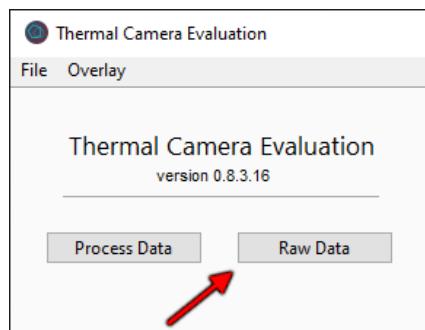
To process new data **File**  $\gg$  **New** and navigate to an experiment folder.

**1. Select Calibration File** system will auto-load calibration file if correctly saved. Alternatively navigate to a calibration file.

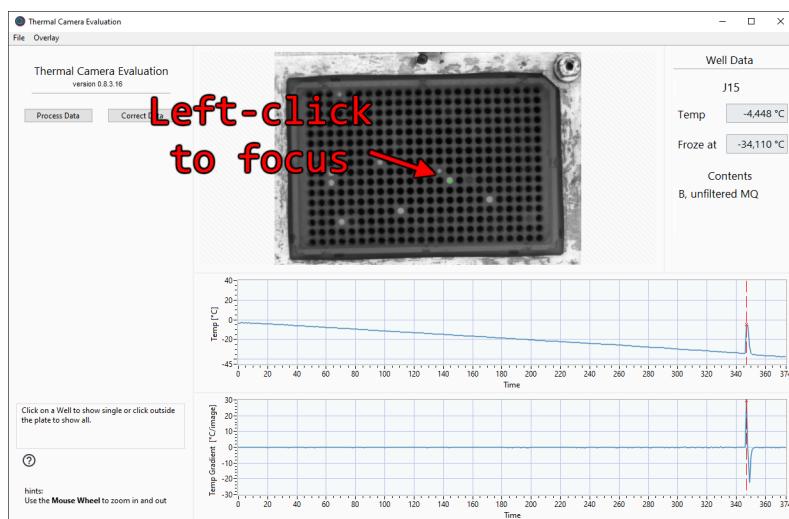
**2. Process Data** press **Process Data** and wait until progress bar finishes.



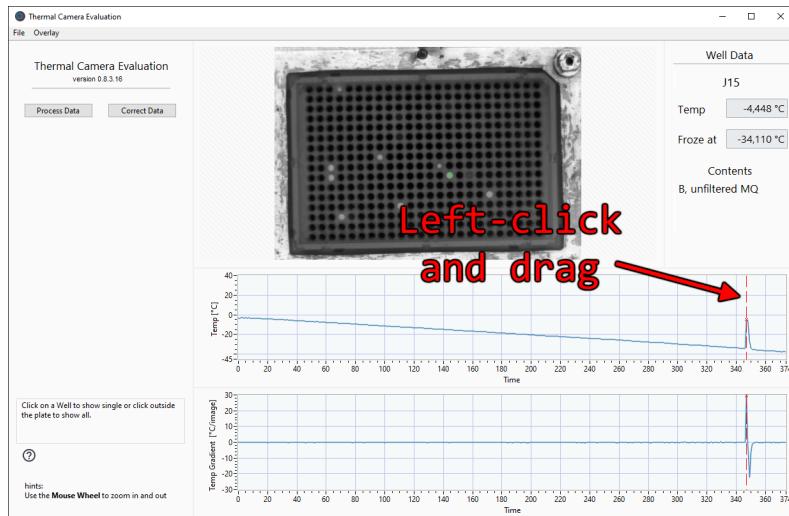
**3. Evaluate Results** Press **Correct Data**/**Raw Results** to switch between thermistor compensated results or raw thermal camera results.



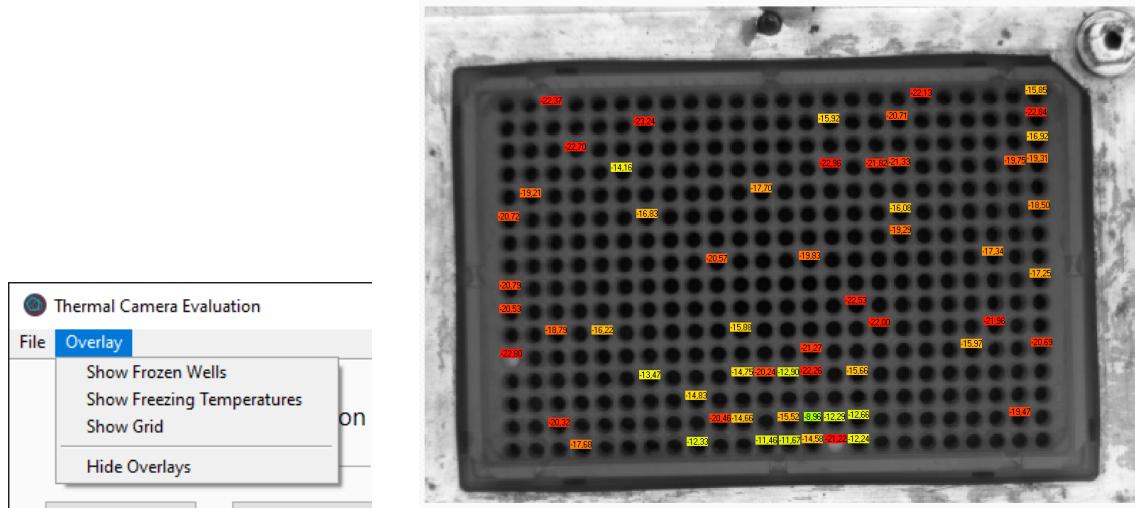
Left-click on any well to focus, displaying contents and temperature measurements. Click outside well area to un-focus.



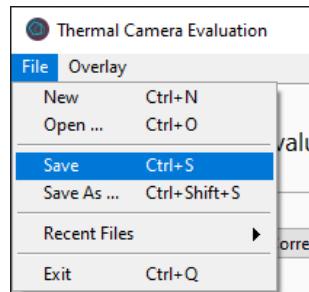
Left-click and drag red marker on graph to navigate timeline.



Use **Overlay** to display different overlays (Calibration Grid, Freezing Temperatures, etc.). Wait for overlay to complete then navigate timeline by left-click and dragging red marker to observe different overlays.



**4. Save Results** **File** **Save** results saved in .csv format, to use in further post-processing.



## 6 Common Errors

### 6.1 Thermal Camera

| Error                   | Cause                | Fix   |
|-------------------------|----------------------|---|
| Camera Connection Error | Camera Starting Up   | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                         | Cable Connection     | Check Ethernet and Power Cable Connection                       |
|                         | Connection Blocked   | Disconnect and reconnect Power and USB, then reconnect systems. |
| Camera Feed Dark        | Camera out of Focus  | Run Camera Focus/Autofocus                                      |
|                         | Camera Lens Blocking | Check and remove lens   |

### 6.2 Cooling Base

| Error                | Cause                     | Fix  |
|----------------------|---------------------------|--|
| Gallium Spill        | Center Pressure too high  | Remove spill with pipette (if liquid) avoid touching center while molten |
|                      | Too much Gallium          | Remove excess gallium and do re-leveling procedure                       |
| Water Leak           | Loose Connector           | Tighten loose connector(s)<br><b>Contact Service</b>                     |
|                      | Broken Seals              | Replace faulty connector(s)<br><b>Contact Service</b>                    |
| Uneven Gallium Level | Poor Gallium distribution | Perform re-leveling procedure  |
|                      | Low Gallium Level         | Refill gallium and perform re-leveling procedure                         |

## 6.3 NI cDAQ Module

| Error                      | Cause                     | Fix   |
|----------------------------|---------------------------|---|
| cDAQ Connection Error      | cDAQ Starting Up          | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                            | Cable Connection          | Check USB and Power Cable Connection                            |
|                            | Connection Blocked        | Disconnect and reconnect Power and USB, then reconnect systems. |
| No Temperature Measurement | cDAQ Initialization Error | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                            | Power Cable Missing       | Check Power Cable Connection                                    |
|                            | Sensor Cable Missing      | Check Sensor Cable (DSUB-9)                                     |

## 6.4 Water Pump

| Error                             | Cause                             | Fix   |
|-----------------------------------|-----------------------------------|---|
| Continuous Beeping                | Disconnected Water                | Check Water Line Connections  |
|                                   | Temperature Difference too high   | Room or Water Temperature too high, power off PSU and let water temperature drop. |
|                                   | Low Water Level                   | Check Water Level Indicator and refill to between MAX and MIN.                    |
| Water Temperature stays above 8°C | Room Temperature too high         | Lower Room Temperature or reduce temperature gradient                             |
|                                   | Temperature Gradient set too high | Lower Temperature gradient of experiment  |
| Water Leak                        | Loose Connectors                  | Tighten loose connector(s)<br><b>Contact Service</b>                              |
|                                   | Broken Seals                      | Replace faulty connector(s)<br><b>Contact Service</b>                             |

## 6.5 Power Supply Unit

| Error                | Cause              | Fix   |
|----------------------|--------------------|---|
| PSU Connection Error | PSU Starting Up    | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                      | Cable Connection   | Check USB and Power Cable Connection                            |
|                      | Connection Blocked | Disconnect and reconnect Power and USB, then reconnect systems. |
| No/Low/Static Output | Connection Error   | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                      | Mode Error         | Backside MODE is set to NORMAL.                                 |
|                      | Cable Disconnected | Output cables that connected.                                   |

## 6.6 Micro Controller Unit

| Error                | Cause              | Fix   |
|----------------------|--------------------|---|
| MCU Connection Error | MCU Starting Up    | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                      | Cable Connection   | Check USB and Power Cable Connection.                           |
|                      | Connection Blocked | Disconnect and reconnect Power and USB, then reconnect systems. |
| No Output            | Connection Error   | Reconnect Systems<br>Tools ➤ Reconnect System                   |
|                      | Cable Disconnected | Output cables that connected.                                   |

## References

- [1] <https://www.flir.eu/products/a655sc/>
- [2] <https://dk.rs-online.com/web/p/termistor-ic/8937234?sra=pstk>
- [3] <https://www.conradelektronik.dk/p/quickcool-qc-241-16-150m-hightech-peltier-elemen...>
- [4] <https://www.ni.com/da-dk/support/model.ni-9219.html>
- [5] <https://www.ni.com/da-dk/support/model.cdaq-9171.html>
- [6] <https://www.alphacool.com/shop/durchlaufkuehler/geraete/21410/alphacool-eiszeit-2000-chiller-black>
- [7] <https://www.elfadistrelec.dk/da/laboratoriestromforsyning\ -32v-30a-960w-justerbar-peaktech-p1580/p/11095172>
- [8] <https://www.matronics.dk/kategori/el-materiel/afbrydere-kontakter/230v/produkt/arbejdsrelae-din>