

Discovery DSC X3 SOPs

Users must read the Instrument Hazards and Best Practices before using this instrument.



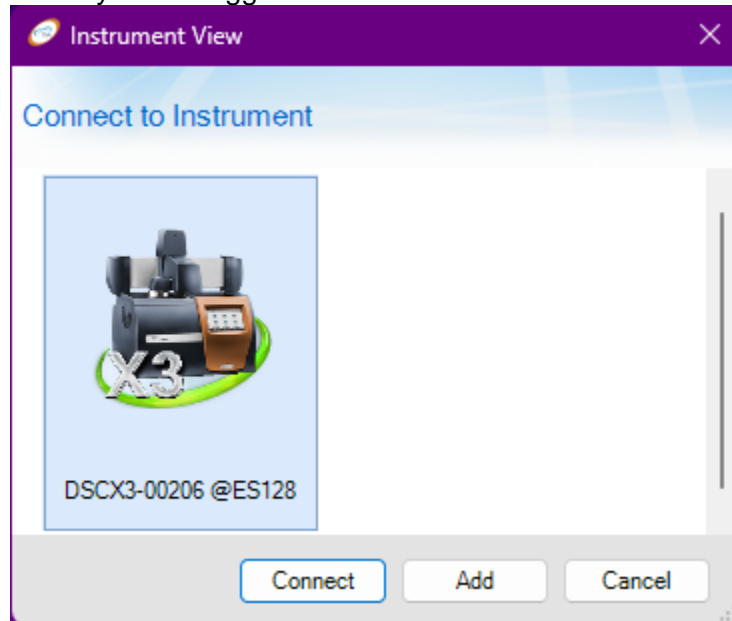
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1. Instrument connection

You must first login to FOM and login to the instrument in order to connect to the DSC. Ensure that you have reserved enough time for the experiments to complete. If not, you can reserve the time directly after your time slot if it is available. If you don't have enough time, and there is a user scheduled right after you, you will need to cut one or more runs to stay within your reserved time. The following user has the right to cancel your tests at the start of their timeslot.

Open **TRIOS** from the desktop once logged in to FOM. If the halo around the instrument is grey, you will need to restart **TRIOS** and confirm that you are logged in on FOM.



Double click the instrument or click Connect. The software will connect to the instrument and open.

2. Sample prep

Sample preparation is the most important part about DSC setup and will lead to the best data possible. Often, bad data quality can be traced back to an improper sample prep procedure.

Recommended sample size is between 1-5 mg with up to 10 mg being acceptable if you don't have a choice. The most important part is to maximize surface area contact with the bottom of the pan. If your sample is a powder or a liquid, this isn't an issue and the sample will have high surface area contact.

For samples that are not flat, attempting to cut, break, or otherwise cause a surface to be flat is recommended. Then place that flat side into the bottom of the pan. For films, there are punches that can be used to help achieve a uniform circle that will fit nicely in the pan.

It is recommended to go through the entire sample prep procedure one at a time to prevent mixing up samples. Once they are sealed in the pans, they are impossible to tell apart if they get mixed up.

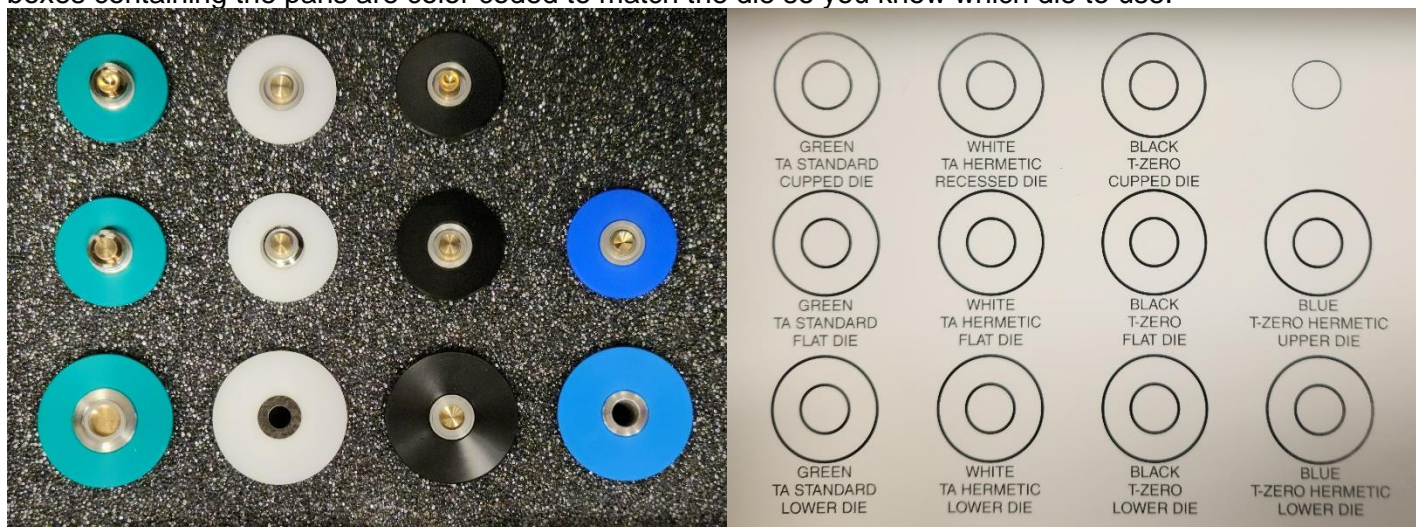
3. Prepare pans

All samples will be pressed into aluminum pans before being placed into the instrument. The mass of the pan and lid for each sample must be recorded into the software. It is recommended to write these down as well to make things easier to set up. **Never touch pans with hands, only tweezers.**

We have two pan types available. The first is TZero Pan and Lid. This is the standard pan style that should be used for most situations where air sensitivity or solvent loss isn't a concern. For liquid samples, the press should not be used and the lid should be placed on the pan and pressed with the side of the tweezers or a spatula until the lip of the lid does not stick above the pan rim. If you press liquid samples, the liquid will squish out above the lid.

The second type is the TZero Pan with TZero Hermetic Lid. This uses the same pan base as the standard pan style, but a different lid and it seals the pan so it is air tight. This is useful for samples that may lose solvent as they are waiting to be ran or samples that are air sensitive. The press can be used in a glove box if samples are really sensitive.

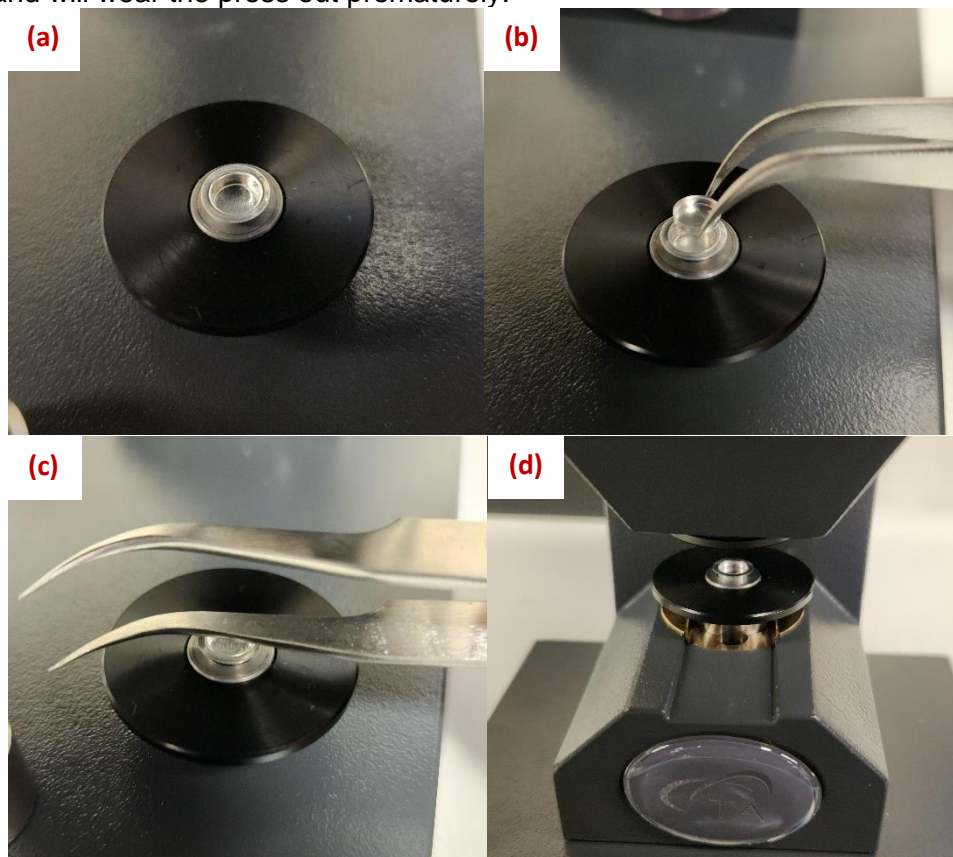
For the different pan types, you will need to select the appropriate die set from the box near the press. The boxes containing the pans are color coded to match the die so you know which die to use.



3.1. TZero Pans

For the standard TZero pans, you will choose the TZero pan and TZero lid boxes and remove the appropriate number of pans and lids. Choose the **Black Die Set**. For most samples, choose the flat die as opposed to the cupped die. The cupped die is for non-uniform samples and the cup will help shape the lid so the samples is held tighter. Place the upper die, with the narrower outer disc, in the upper portion of the press. It is held magnetically. The press lever should be towards the back for this.

Weigh and record the pan and lid mass. Weigh and record your sample. Place the sample in your pan and then place the pan into the lower die **(a)**. Place the lid onto the pan in a cup-like orientation **(b)** and use the flat of the tweezers or a spatula to ensure the lid is properly seated **(c)**. Then place the assembly into the lower slot in the press **(d)** and press the sample. Do not apply an extra force to the press lever. It will not press the sample anymore and will wear the press out prematurely.

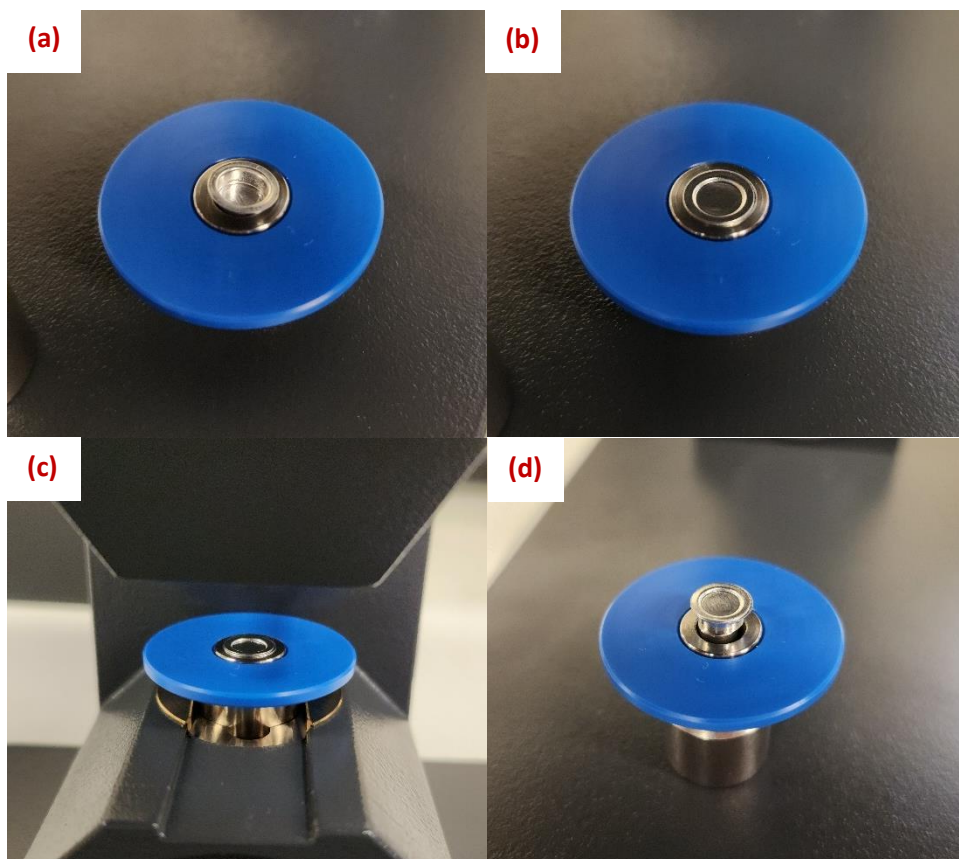


Immediately place the pan into the autosampler and enter the details in to the software as seen in [Setting up a run](#). Ensure you match the reference and sample pan types. See [Reference pans](#) for more info.

3.2. TZero Hermetic Pans

For the Hermetic TZero pans, you will choose the TZero pan and TZero Hermetic lid boxes and remove the appropriate number of pans and lids. Choose the **Blue Die Set**. Place the upper die, with the narrower outer disc, in the upper portion of the press. It is held magnetically. The press lever should be towards the back for this.

Weigh and record the pan and lid mass. Weigh and record your sample. Place the sample in your pan and then place the pan into the lower die **(a)**. Ensure the pan is seated all the way into the die. Place the lid onto the pan in a cup-like orientation **(b)**. Then place the assembly into the lower slot in the press **(c)** and press the sample. Do not apply an extra force to the press lever. It will not press the sample anymore and will wear the press out prematurely. You can use the pin attached the fron of the press to help remove the pan from the lower die **(d)**.



Immediately place the pan into the autosampler and enter the details in to the software as seen in [Setting up a run](#). Ensure you match the reference and sample pan types. See [Reference pans](#) for more info.

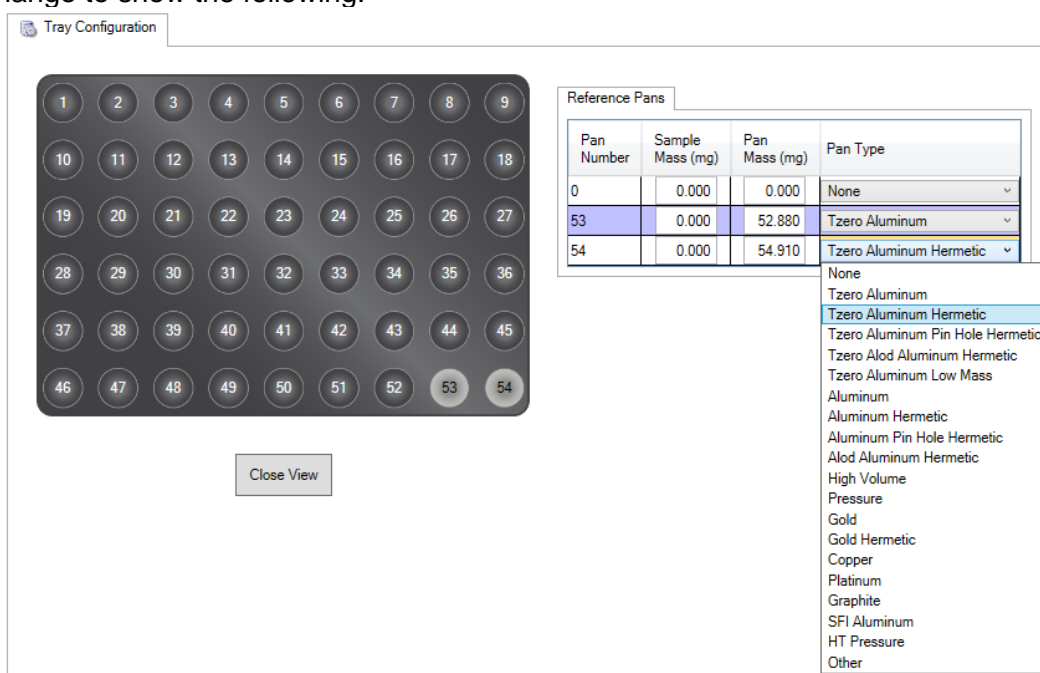
4. Reference pans

The reference pan is an empty pan of the same type as your sample pan(s). You cannot have pans of a different type in the cell at the same time for a run. For example, two of my samples are in hermetic pans and one is in a standard pan. This would mean that I need two separate runs even if the parameters of the run are the same. Therefore, it may be beneficial to match all the pan types even if it isn't necessary for all your samples.

Reference pans can be used many times over and should not be remade except by someone who knows what they are doing. The reference pans will be kept in **autosampler slots 50-54** and will be placed back there after the run has completed.

If there is already a reference pan of the type you need, then you can select that during your [run setup](#). Otherwise, you will have to add one yourself. To add a reference pan, prepare the pan as if you were preparing a sample, but leave it clean and empty. Then mass the pan and record the mass.

Navigate to the **Instrument** tab on the main software ribbon at the top. Choose the **Tray** button and the windows will change to show the following.



Select the position that you'd like to place your reference pan. Do not delete any of the reference pans already in place unless you are replacing it due to contamination or if the pan was damaged or lost. The selected position will highlight grey and a new line will appear in the table. Ensure you enter the mass of the pan that was recorded and choose the correct pan type from the dropdown menu.

After this, the new reference pan will be available from the run setup. If a reference pan already exists that matches your pan type, use it and do not create a new one.

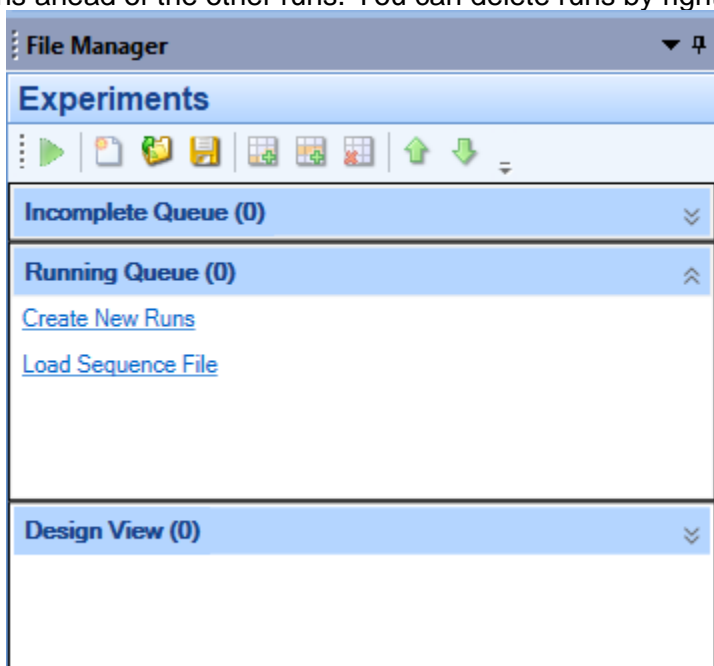
NOTE: Never modify pan zero. It will fundamentally mess with functions of the instrument.

5. Setting up a run

In order to setup a run, you will need to navigate to the **Experiments** tab in the software. This is located in the bottom left of the software.



In the **Experiments** section, there shouldn't be any runs left. Runs that have completed successfully will disappear from the **Running Queue**. If there are runs when you arrive to your reserved time, you may remove them, or simply put your runs ahead of the other runs. You can delete runs by right-clicking on them.



Click on **Create New Runs** to add a run. You should see the following in the middle of the **Experiments** window.

NOTE: This system can run 3 samples simultaneously. This means that all three samples will undergo the same thermal cycling. You will get individual data files for each sample. If you do not want all the samples ran with the same conditions, each sample will need its own run in the queue and you will need to deselect the other samples in each run so it doesn't try to run them.

Enter the **Sample Names** for each sample in *this* run. If you do not want to have all three samples ran in this run, deselect the check box next to the sample name. You should enter an **Operator** and **Project** name and all the filenames generated will have the operator and project name in them automatically as seen in the example.

Choose a **Reference** from the dropdown. Ensure that the reference that you choose is the same pan type as your sample pans as seen next to **Pan Type**. It will update depending on which reference you chose. See [reference pans](#) for more details.

Queued Run
Design View (0)
Running Queue (1)
Schedule

Run 1 in Running Queue

Sample

☒ Sample A Name SOP_A
☒ Sample B Name SOP_B
☒ Sample C Name SOP_C

	Pan Number	Sample Mass		Pan Mass	
Sample A	1	2.680 mg		52.940 mg	
Sample B	2	3.320 mg		50.680 mg	
Sample C	3	2.250 mg		51.550 mg	
Reference	53	0.000 mg		52.880 mg	

[Edit Tray Configuration](#)

Pan Type Tzero Aluminum
Operator Kyle
Project SOP Writing
Notes

File Name C:\Data\SOP_A-SOP_B-SOP_C (A,B,C)_Kyle_SOP Writing.tri

Expand the **File Name** carrot to select the save location. In order to have your data backed up online automatically, you will need to choose a location within the C:\Data\ directory. Please keep this directory organized and cleaned up. Students must always save within their research advisor's folder. Click the button with the **ellipses** in order to choose a file save location.

File Name C:\Data\Test_AMSEC_SOP Writing.tri

File Path C:\Data
Default
...

Template <SAMPLENAME>_<OPERATOR>_<PROJECT>
Add Token

☐ Reset run number when saved

Choose your test template or create a custom run from the dropdown. You can save and load procedures from the blue **Procedure** bar. Save these procedures in the same folder that you would save your data, or create a procedures folder within your research advisor's folder. Enter the appropriate run parameters into the fields. This will not be covered in the SOPs as there are many procedures used for many different reasons. You can ask the lab manager for assistance if you are not sure.

Queued Run | Design View (0) | Running Queue (1) | Schedule

Procedure

Test: Custom

Name:

Segn:

No. Des.

Verify Cell Constant/Temperature

Verify Temperature

Verify Heat Flow

Modulated Conventional

Modulated Heat Only

Modulated Quasi-Isothermal

Edit

Advanced

Load Window

☒ Use Standby Temperature

End of Test

☐ Discard pan in waste bin at end of test

☒ Use Standby Temperature

Selected Calibrations

☒ Use default calibrations

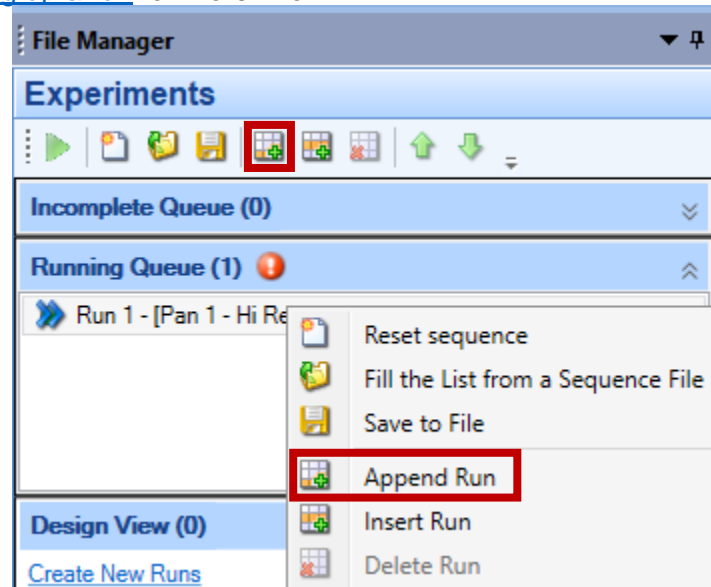
Under **Advanced** you may want to choose to keep or discard your pan once the run is finished. If the box is checked by “Discard pan...” then the pan will be dropped in the little bin on the instrument. If it is deselected, then the pan will be placed in the autosampler once finished. **DO NOT** change the other settings under **Advanced** besides this one.

NOTE: If you want to keep your pans after the run and you deselect the box. Ensure you leave a note in front of the instrument of what pans you want to keep and when you will return for them, otherwise they will be thrown away by the lab manager.

Once the run is finished being configured, [add another run](#) if necessary.

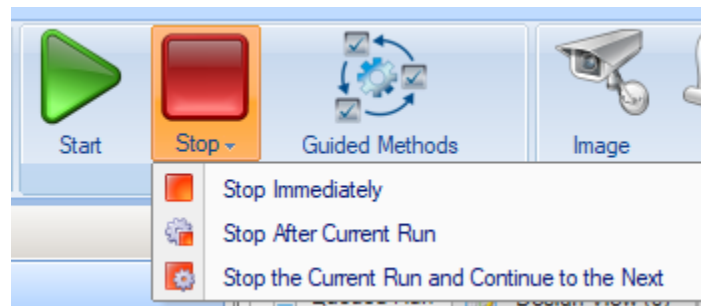
6. Adding runs

To add runs, choose the append option from the **Running Queue** in the **Experiments** section. It is important to choose **Append Run** as opposed to insert run. Append will copy all the settings from the run that you selected when you chose it. This is helpful since users typically have multiple runs with the same parameters. Once the run is appended, you will need to fill in the sample info and double check all the run parameters. Check the section on [setting up a run](#) for more info.



7. Starting and stopping runs

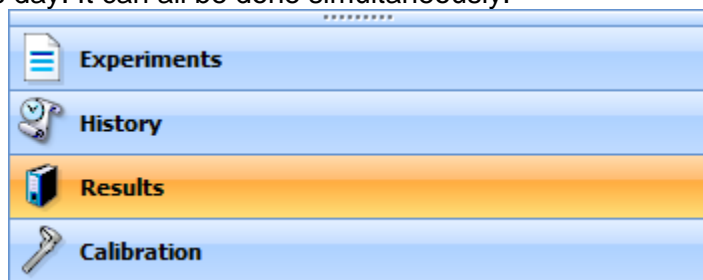
To start a run, choose the big green **Start** arrow from the **Experiment** tab on the main software ribbon at the top. This will start whatever run(s) that have been created in the **Running Queue** in the **Experiments** section of the software. The software will automatically take you to the **Results** section of the software once data collection has started.



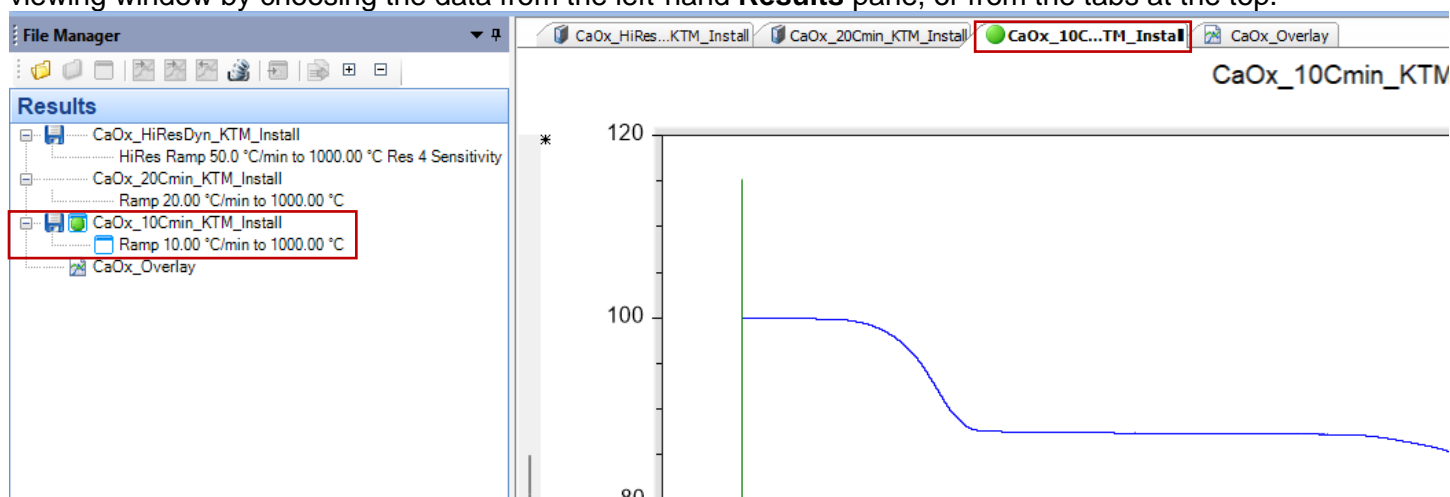
To stop a run, choose the big red **Stop** square from the **Experiment** tab on the main software ribbon at the top. You will then have to choose from the three options. Read them and choose accordingly. The system will then execute the selected option.

8. Viewing active runs and data

When a run is started, it will automatically change the view to the **Results** tab from the bottom left window selection. This is where you will view actively collecting data, successfully completed run data, and even open data collected on a previous day. It can all be done simultaneously.



The actively collecting data will be indicated by a green-filled circle in the **Results** pane as well as in the tabs above the plots. All data collected prior to that sample will show up higher up on the list and all data collected after the currently active sample will show up lower on the list. You can cycle which data is displayed on the viewing window by choosing the data from the left-hand **Results** pane, or from the tabs at the top.



In this SOP, we will not discuss all the possible ways to view and manipulate data in this software. This document is designed to help users operate the instrument. Data workup can be done with someone experienced on the system, or with the lab manager.

9. Instrument hazards and best practices: DSC

This document will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety training document that each user has attested to have reviewed at fom.wvu.edu/documents

Lab coats are to be provided by the user unless special hazards exist in which case they are located at the PPE station.

Hazards:

- Chemical exposure
- Extreme Temperatures (-90 – 550 °C)

1. Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-155-215).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

2. Extreme Temperatures

The DSC is designed to operate between -90 and 550 °C. This means that the furnace could be near or at those temperatures at any given time. The best way to ensure that no damage is caused by these temperatures is to simply avoid contact with the furnace. There is no situation in which it is necessary to come into contact with the furnace. Avoid allowing anything that may be degraded, damaged, or made more flammable to come into contact with the furnace at all times.