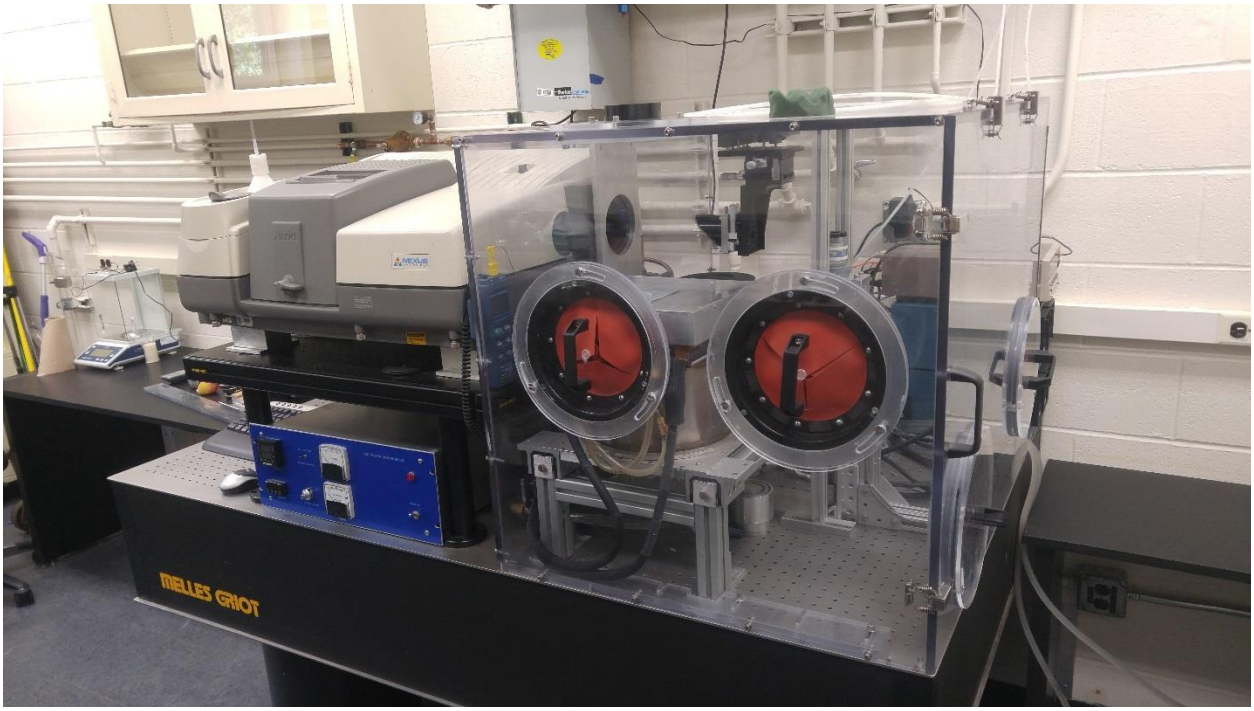
The IVIS Spectroscopy Laboratory (v1.5)



A how-to guide for taking spectral measurements and maintaining the laboratory

Written by

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Introduction

This is intended as a guide to allow people to take Thermal Infrared (TIR) measurements using the Nicolet Nexus 670 FTIR spectrometer. The lab currently has two experiments that are operational; a low temperature set up modelled on work done at Arizona State University, and a high temperature furnace for taking spectral measurements of melt samples, developed by Dr. Rachel Lee. This work will give an overview of the operations of the laboratory, and provide the necessary methods and protocols for maintaining and operating the equipment.

Acquiring TIR Emissivity Spectra

Sample preparation

Samples should be left in the oven to reach ~80 ˚C. This should be done the day before you intend to run your samples in the spectrometer. All samples, bulk or powder should be placed onto a piece of weighing paper in the drying oven with the sample name, your initials and date the sample was placed in there written on it. Bulk samples can be placed straight onto the paper. Powder samples should be placed into the copper sample cups. Larger mineral samples should be washed with acetone prior to this, in order to remove any clinging fine particles that can affect spectral measurements. Once placed in a cup don't "level" the sample out. Stir the sample in the cup to randomly orientate the grains, therefore preventing any axial effects.

Obtaining Liquid Nitrogen

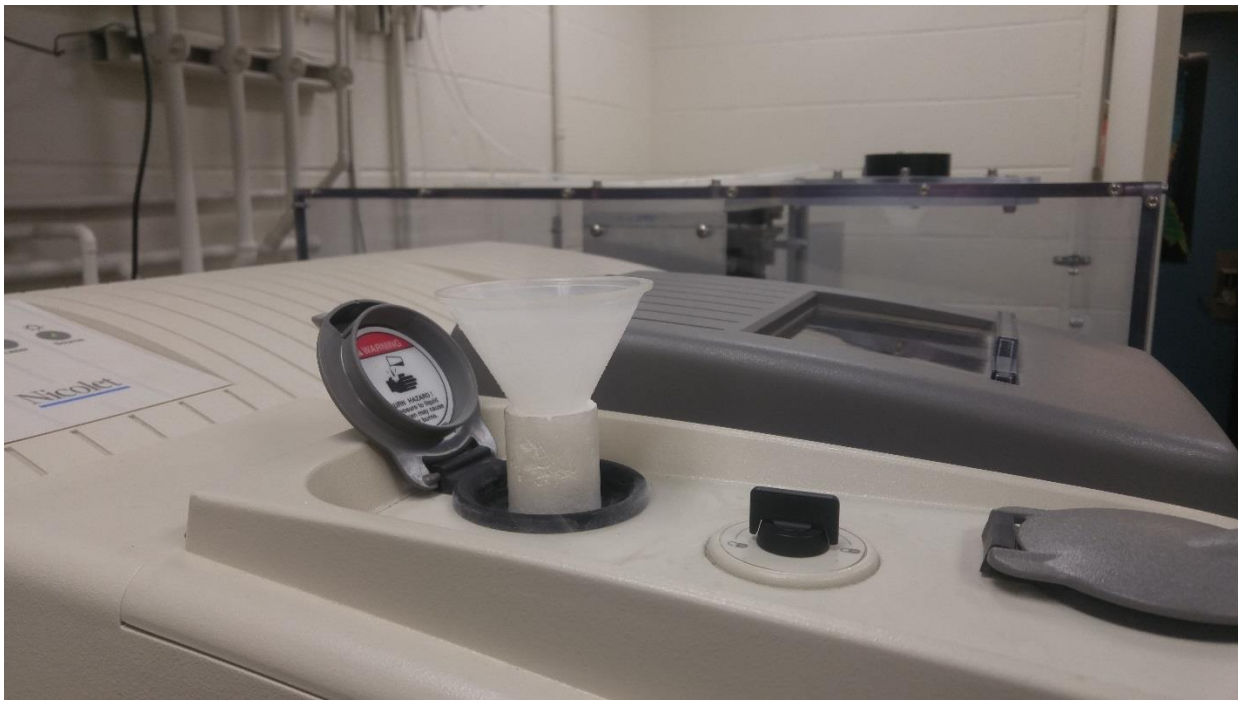
The spectrometer currently uses a MCT-B liquid nitrogen chilled detector. Liquid nitrogen can be purchased from the department purchasing officer, Laura Provolt. Contact her at geopurch@pitt.edu with a request for 1 liter of liquid nitrogen (catalog number NC0084820) from the Dietrich Scientific Stockroom. Once the purchase is confirmed, the liquid N_2 can be obtained from the stockroom, on the third floor of the Chevron Building. Fill up the container from the gas canister in the hallway.

Preparing the lab

First, please sign in using the laboratory sign in excel sheet. Use the sliding platform and move it to the far left, so that the low temperature can is aligned with the spectrometer port. Check to make sure that the mirror marked “new” is also placed in the mirror mount. If the “old” mirror is there instead, open the glovebox, slide the mirror out using the two tabs at the top, and then place the new mirror in. Close the glovebox and using the hygrometer, check that the humidity in the box reaches 0. Plug in the water bath and water pump, these are found under the desk next to the glove box, and turn the water bath on. The water is pumped into the low temperature can to maintain a constant temperature, and increase the signal to noise ratio. The water bath temperature should be set to 25 ˚C, and should not be adjusted. Turn on the three control boxes; these are for the heated sample cup stage and the two blackbodies.

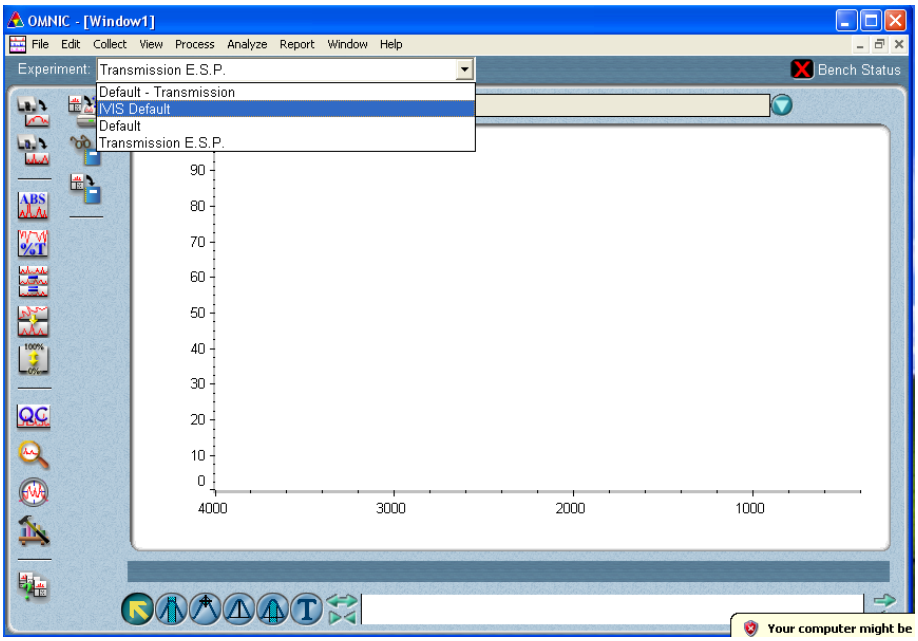
These controller boxes have been preset to three set point temperatures. Blackbody one is set to 70˚C, blackbody two has been set to 100˚C and the heated sample stage has been set to 80˚C. These should not need to be changed, however if there is an issue with the temperature of any of these, the set point temperature can be changed by pressing the metal switch to the right, and whilst holding that there, the dial marked “Set I” can be rotated to change the temperature. The two blackbodies, heating stage and water bath can all be turned on before adding liquid N_2 to the detector to allow them to equilibrate.

Nitrogen then needs to be poured into the detector. Open the flap on the back and place the funnel in. Pour nitrogen from the big canister into the smaller flask. Fill this roughly halfway. In small quantities pour the nitrogen in. Replace the screw in the hole and seal the cap. The spectrometer should then be left for 20-30 minutes.

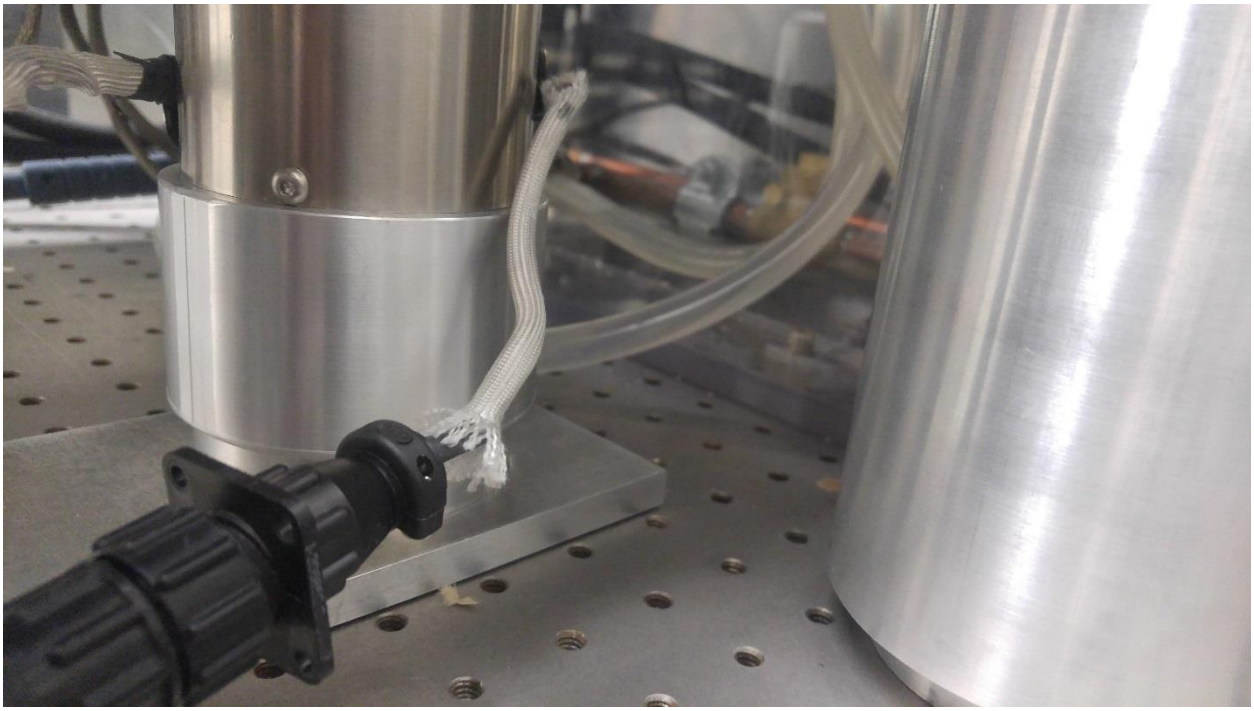


Obtaining Spectra in OMNIC

On the computer open the OMNIC software package. In the top right hand corner there is a box with the words bench status written next to it. When the detector is chilled, a green check mark will appear in this box. Open the experiment bar at the top middle and select “IVIS Default”.

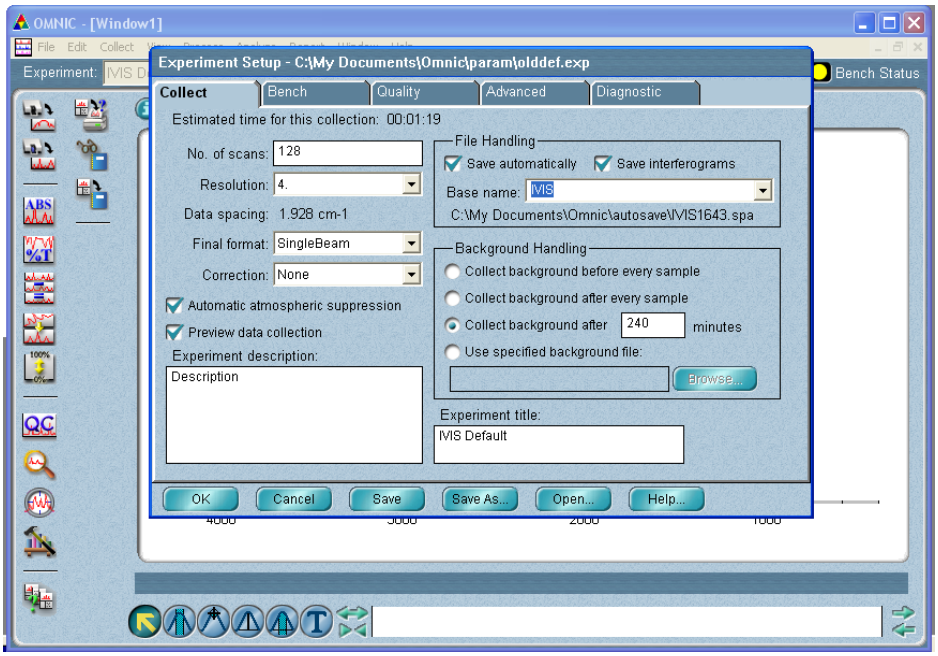


Check the temperature of the water bath and the blackbodies; once they reach a constant around 25°C, 70°C and 100°C respectively, the first blackbody can be placed on the elevator platform. Place it on top of the base which is slotted into the groove on the elevator platform. Use the blue control box to raise the blackbody up in the can.

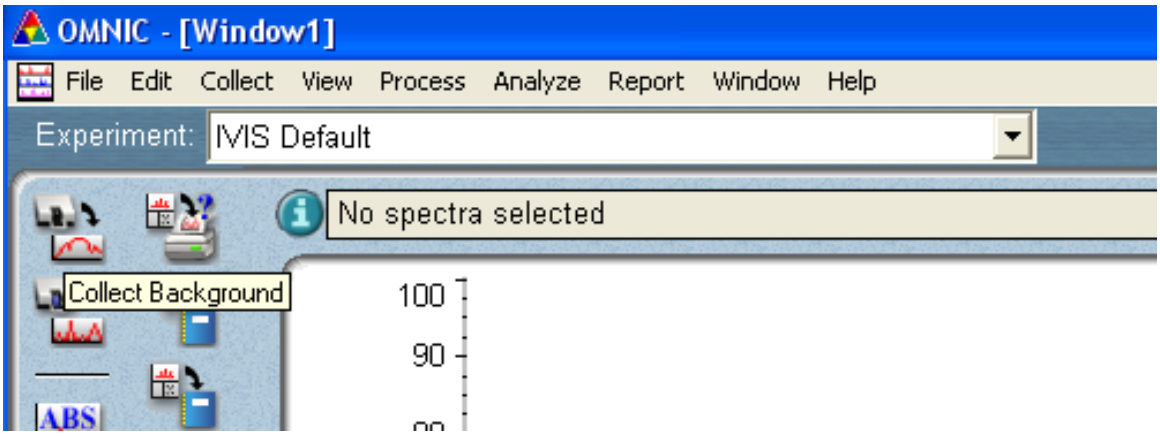


In OMNIC, click “Collect” and then “Experiment Setup”. Change the number of scans to the desired amount. Bulk rock samples will need no more than 128 (32 or 64 scans are also fine

for pure mineral crystals), for finer powders it is advised to set this to 256 or 512 to reduce noise in the spectrum.



At this point, open the “Shortcut to Spectrometer” folder and open the “template_new” Excel file. This will be used to take the RTD measurements on the blackbodies and the temperature of the can.



Once the blackbody is at the maximum height, in OMNIC click “Collect Background”. Then click start collection. Roughly halfway through the number of scans, go into the template file and select “ExceLINX”, scroll to “DMM Scan” and click start. Five numbers will then appear, in the columns 101, 102, 103, 107 and 108. For the blackbodies, 103 can be ignored, unless you wish to process the blackbody as a sample, as this is the can temperature. Take a note of the measurements for 101 and 102 for blackbody 1 and 107 and 108 for blackbody 2. These

are the resistance values for the blackbodies that are used to determine the average temperature. Once the scans are completed, remove the first blackbody from the can. Repeat the same process for blackbody 2. Following this, remove from the can and turn the blackbodies off, and place them in the front left corner of the glovebox to allow them to cool down. In your folder, save these as bbwarm.CSV and bbhot.CSV, as they will need to be in this format to be ready by the DaVinci emcal2 program.

To run a bulk rock sample, the sample must be removed quickly from the oven and placed into the rock holder. Slide this inside the can, and place the base underneath it, so that it locks in the outer ring. Take the sample up into the can to the desired height. Once there, click "Collect Sample" and subsequently press "Start Collection". Roughly halfway through use the DMM scan to take the temperature of the sample can, which will appear in the 103 column. Once the scans are completed, remove the sample and place back in the oven.

For fine grained samples, take the sample cup and place in the heated cup holder. Allow the sample to thermally equilibrate; this can take from 5 – 10 minutes depending on whether you are looking at a homogenous mineral sample, or a mixed sample of sediment.

Once the sample is ready, hold the cup holder inside the can, and place it on the base, locking it into the inner ring. Raise the cup holder into the can. Use the same process as for bulk samples to obtain the spectrum and take the can temperature measurement. Save each spectrum in the same folder as your black body data as ".CSV" files.

Post Processing – Visualizing TIR Spectra in Davinci

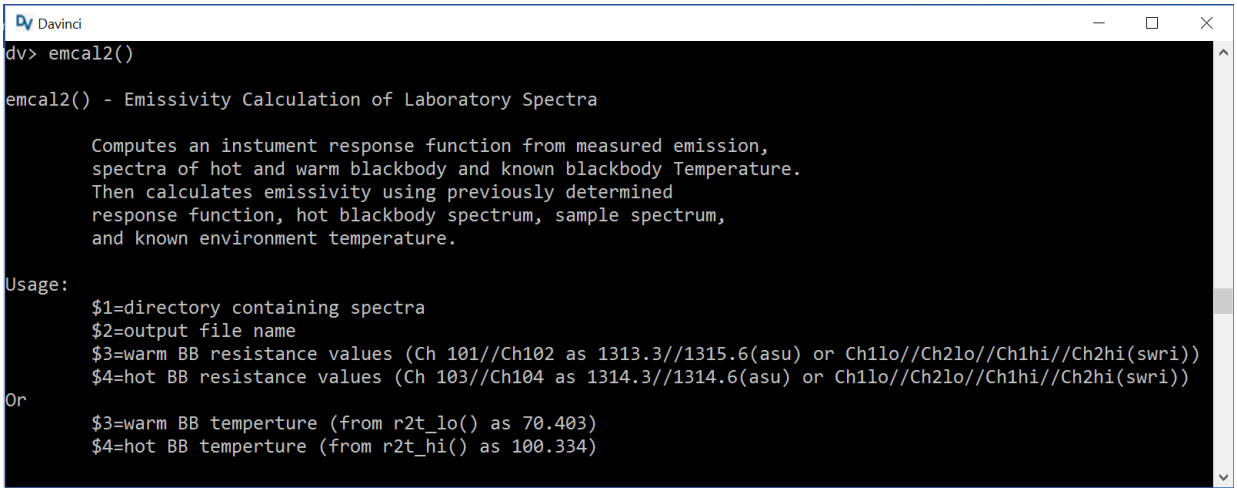
Once the spectra .CSV files have all be saved, these can finally be processed. These data can be processed on any computer by transferring the files via a thumb drive or by using the IVIS server. To use the IVIS server, first open Filezilla on the lab computer, and connect to the address: ivis.eps.pitt.edu. Type your IVIS user name and password in (which can be set up on request to Dr. Ramsey), and then type in 22 for the port number. This connects the computer to IVIS, the folder containing spectra can be then uploaded to the server by dragging it to your desired location in IVIS. Follow the same steps on any computer to retrieve the data.

The program Davinci is used to processes the data collected from the spectrometer into usable emissivity data. This program is compatible with multiple operating system and can be downloaded at the website: http://davinci.asu.edu/index.php?title=Download_Davinci. If this is the first time Davinci has been run on the computer being used, type in the command: "library_update(update=1)". This will update Davinci to it's most recent release, which includes the programs emcal, r2t_hi, and r2t_lo, which will be used in processing these data.

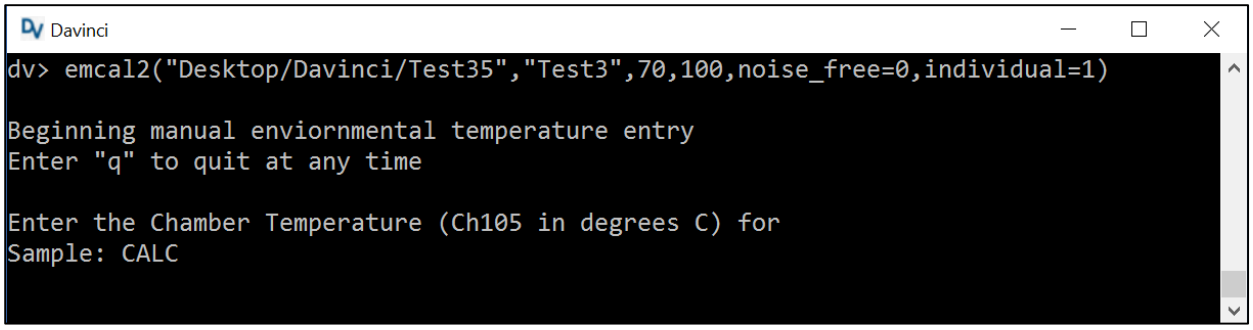
Before the data can be processed the measured temperature of both the hot and warm blackbody must be determined. The r2t_hi and r2t_lo program perform this function. In order to run this program, type the command: “r2t_hi (bbhot resistance value 1, bbhot resistance value2)”. If entered correctly, this program will output an average temperature value for the hot blackbody. An example of what this command looks like with theatrical resistance values as well as the expected output can be found below.

Repeat this same process using the program r2t_lo for the warm blackbody and record both of these temperatures for use in the emcal2 program.

To process data in Davinci first type in the command: “emcal2()”. If the program emcal is functioning properly, this command will give you the dialogue box seen below.



To run the emcal2 program with data from the cooled detector, enter the command: “emcal2(pathway to folder containing spectra data, file name for processed data, warm blackbody temperature, hot blackbody temperature, noise_free=0, individual=1)”. In this command, the argument noise_free=0 applies to corrections made to the instrument energy. For data collected from the uncooled detector, omit this argument. Also, the argument individual=1 commands emcal2 to produce individual text files for each sample spectra. An example of this command in Davinci can be seen below, take note of the quotes that are needed to surround the first two arguments.



Follow the instructions provided by emcal and enter the can temperature (column 103 in OMNIC) where prompted. When this program has completed a file with the name you provided can be found in the same directory that the folder containing your spectral data in .hdf format as well as individual .txt files corresponding to each sample spectra. These ascii files can then be opened as tab separated txt files using Excel.

High-Temperature Emissivity Spectra

Coming soon.

Acquiring VSWIR Reflectance Spectra

Coming soon.

Laboratory Maintenance

Maintenance of the spectroscopy laboratory is paramount to allow measurements to be taken accurately and easily. Furthermore, maintaining the lab correctly and regularly allows others to successfully complete their work as well. If you are regularly using the lab equipment, please make sure that you perform the following:

Spectrometer Alignment

How often – Minimum once per week

How to – You should always make sure that the laser is sighted inside the sample can/furnace before running any spectra. Small adjustments to the mirror mount can be made. ONLY USE THE FORWARD/BACKWARD AND LEFT/RIGHT CONTROLS. The best practice here is raise a small sample cup into the can, and by standing over the glovebox, it is possible to see whether or not the laser beam falls inside the sample cup. However, it is also good to run a laser alignment through the Omnic software once per week. Go to "Diagnostic" and click the "Align" button. This will then align the laser through the beam splitters and will check that they are still placed correctly.

Post-alignment standard spectral measurements

How often – Once per month (after a spectrometer alignment)

How to – There are three laboratory standards that are used – A large piece of obsidian, a copper sample cup with 710 – 1000 µm quartz crystals and a copper sample cup with 710 – 1000 µm calcite crystals. These are to be run after performing an alignment to make sure that the equipment is working correctly. The piece of obsidian can be run for 32 scans, whilst the quartz and calcite should be run for 128 scans. The obsidian should be run straight away after removing from the oven, whilst the quartz and calcite should be allowed to equilibrate in the heated cup stage.

Cleaning the water bath

How often – Once per month

How to – The heating elements of the water bath can get quite dirty. It's therefore essential to clean it so as not to allow permanent damage to occur. Once a month, remove the water from the bath, and clean using a solution of water and white vinegar.

Gas purge filters and moisture indicators replacement

How often – Once per year/two years

How to – Parker recommend that either annually or bi-annually, the moisture indicator and cartridge filters in the 75-62 Parker Balston nitrogen gas purge be replaced. To do this, turn the air tap off and unplug the purge generator from the wall. Allow the system to depressurize, and when it has, the two cartridges are located at the base of the can be removed by twisting them anticlockwise. The filters can then be replaced by unscrewing the bottom piece and removing them. The moisture indicator is replaced by unscrewing the nut at the bottom of the indicator. The glass housing can then be pulled gently off and the moisture indicator replaced.

Sample removal from the oven

How often – Once a sample is finished with

How to – To allow everyone to be able to use the spectrometer in a timely manner, once you are done with your samples, please return them to storage. There is limited space in the oven, and we only have a certain number of sample cups. Once samples have been stored, please also use a Chem Wipe and acetone to clean the copper sample cups used.

Troubleshooting

Water leak in glovebox

If leak is where the pipe is connected to the can, use a flat head screw driver to make sure that all the hose clamps are tightened fully. Replace the pipe if necessary (spare piping is found in the lab drawers).

DMM Scan produces abnormal looking numbers

Exit and then restart Excel and run DMM Scan. If the problem persists check the set temperature of the blackbodies. If the set point is fine, check that the wires are properly connect to the blackbodies, the controller boxes, the multimeter, and the computer. If all of this is fine and everything is ok, contact Chuck Fleishaker (chuckchm@pitt.edu) in the Electronics Shop for assistance.

Spectra tail upwards when processing

Most likely problem is that the detector is warming to levels where it is unable to properly collect spectral data. Refill the detector with liquid nitrogen, wait to equilibrate and rerun the samples.

OMNIC Error – The source voltage is out of alignment

This is a problem with the power supply. Make sure it is fully connected to the spectrometer on the back. Check that the power supply is not overheating and that the fan is blowing – if it isn't, turn off the spectrometer, unplug the power supply, and take it to the electronics shop for cleaning/fan replacement.

Laser is not on

Turn the spectrometer off and then on again. Use the OMNIC troubleshooting software to see if it can detect the problem. If everything else in the spectrometer reads normal but the laser is still not coming on CONTACT DR. RAMSEY IMMEDIATELY.