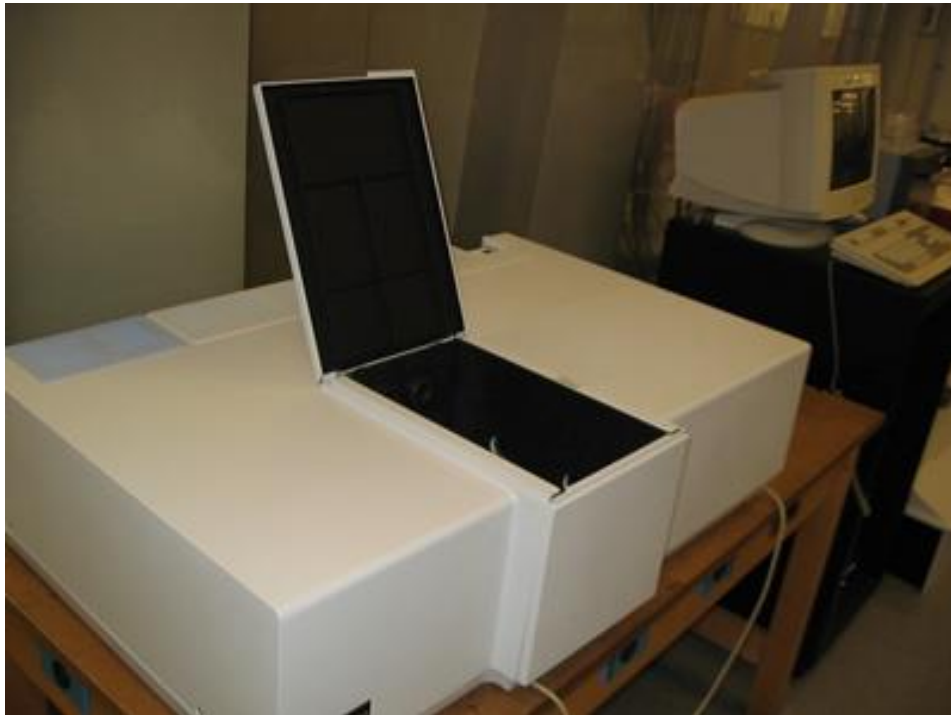# Spectrophotometer

January 6, 2014

Location:　　10K Area
Primary Trainer:　　Scott Munro　　(587-879-1517, smunro@ualberta.ca)

OVERVIEW

The Perkin Elmer Lambda 900 Spectrophotometer is a dual beam spectrophotometer with a wavelength range of 185-3300nm. Transmission, absorbtion and reflectance measurements can be performed. Available modules include the standard reflection/transmission unit, as well as an optical bench with an integration sphere for user defined pathlengths and data collection..

There is a fairly comprehensive user manual in addition to this SOP. Refer to the Perkin Elmer UV Winlab software guide for more detailed information.

SAFETY PRECAUTIONS

Normal laboratory practices apply; gloves should be worn when handling any samples or optics (mirrors, windows). It is recommended that the four silica windows be placed over the four openings in the sample compartment to protect the internal optical components from damage, contamination, and changes in humidity.

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Take care not to spill liquids onto the spectrophotometer, and immediately clean up any spills with cleanroom wipes. Damage to the optics and electronics can result if liquids are spilled and run inside the instrument.

This equipment contains a power supply that delivers hazardous voltages, and operates in the UV region. Do not operate if any panels are missing.

If you are bringing any new materials into the NanoFab for use in your process, it is necessary to fill out a chemical import form (available on our website, http://www.nanofab.ualberta.ca) and supply an MSDS data sheet to Stephanie Bozic.

PROCESS COMPONENTS OR FEATURES
Before starting, make sure the proper modules are installed for your use. Either the standard module or the optical bench will be installed, use care when switching units, and ensure the main power switch is off. Use the alignment pins as a guide for mounting the detector module or the optical bench. Gently press the electrical connection when you are sure it is aligned properly. You should feel the module press in smoothly, do not force it. If either unit is not seated properly, the tool will not initialize in the software. Refer to the **Accessories** section for further information

OPERATING INSTRUCTIONS

1. Samples should be clean and ready for measurement. Liquid samples should be contained in spectroscopic cells designed for the cell holder.   Users are required to provide their own cuvettes for analysis.

2. Turn on the system power by switching the green switch located at the rear right corner of the system. The lamp must be turned on and the system allowed to warm and initialize for at least 10 minutes before starting the software program.

3. Once 10 minutes has transpired, open the **UV Winlab** software on the desktop to start the program. You will be prompted for a username and password to continue; use **nanofab** for both fields.

4. Select a **method** for analysis. If you do not yet have a method, the **Scan** method is the default method. It is recommended that users save their own method for future use. A second **Scan** window will open, likely in the Sample Info screen. The software should begin initializing, and switch to **Idle** within ~1 minute. If the software fails to initialize, it's likely not enough time has elapsed since the hardware was turned on. Close the **Scan** window, wait a few more minutes, and re-open.

5. Once in the **Sample Info** screen, enter the number of samples in the labeled cell. Enter a **Sample ID** for each sample as required.

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6. Click on the **Data Collection** tab in the Task List to access the method settings. Edit the desired scan parameters as required. The wavelength ranges settings must be set to run from high to low. Typically it's the range, increment and mode that are changed, but more advanced changes can be made in the **Instrument** tab. Available modes include %T, A, %R E1, and E2. E1 and E2 are energy scans, and are not likely useful for standard processes.

7. To view the instrument setup and to access more advances features, select the **Instrument** tab. Here an overview of the beampath and optics can be viewed and edited. Most default settings should remain the same, and users should be careful adjusting the Monochromator or Detector settings.
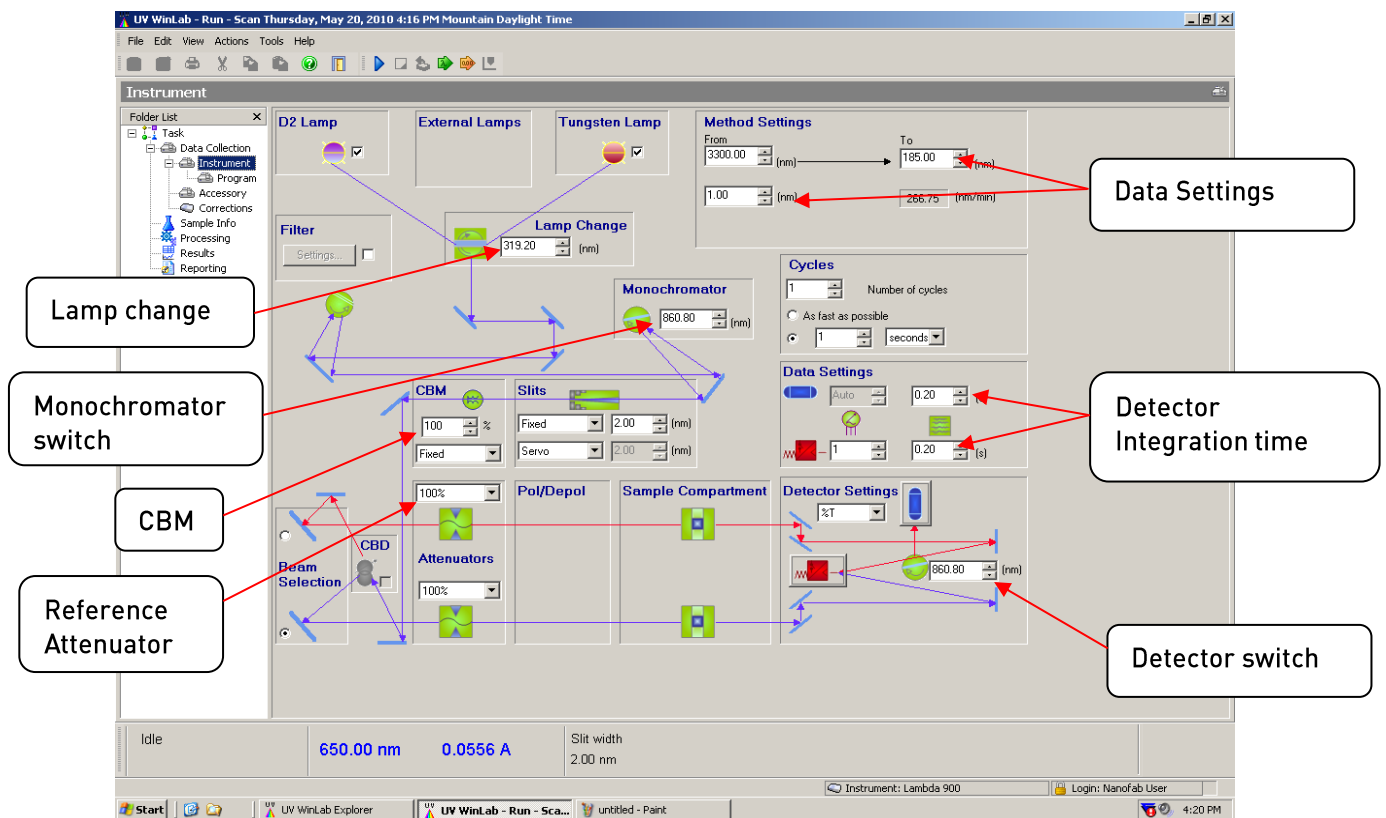

Instrument Tab

Items that can be adjusted include:
-Detector Integration Time – increase for more sensitive scans.
-CBM – common beam mask, adjusts the height of the beam. 100% = maximum height, ~1cm, 0% = ~0.5cm. Note the width remains the same, ~0.5cm
-Attenuators – 100% typical, adjust if the sample highly absorbing. The reference may be adjusted to bring it into the same order of magnitude as the sample.

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8. An **Autozero** must be performed prior to running any sampe scans. To view the settings, click the **Corrections** tab. Select the desired autozero setting. Typical settings should have the frequency set to **Always at task start**, and the **100% and 0% Baselines** checked.

9. To begin the autozero, press either the **Autozero** or **Start** icon. The Autozero will stop once the step is complete, pressing the Start will continue to the sample table once the Autozero is complete. When prompted, press **OK** to acknowledge the chamber is empty and the lid closed. If the autozero step fails within ~1min, press **Stop,** then try restarting the autozero step. If a windows error appears, the program will have to be force closed (Ctrl-Alt-Del), and restarting the software.

Hover the cursor above each icon to view function
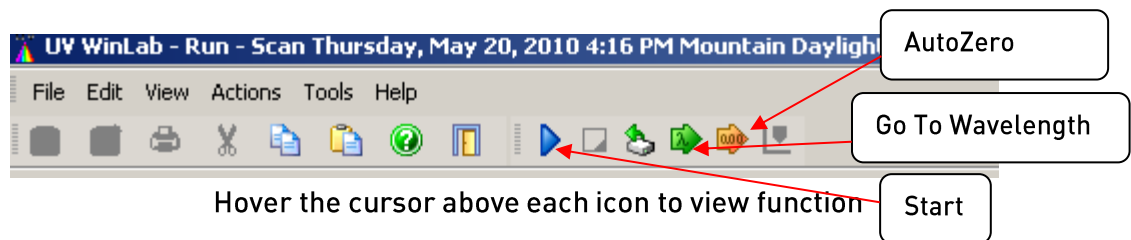
10. Wait until the autozero is complete before opening the sample compartment lid. Load the sample holder and sample as required. If using the variable angle sample holder, align the holder to the back flat portion of the sample compartment, ensure the angle is set to 0, and the sample is sitting vertical. If the liquid cell holder is installed, ensure the holder is properly aligned and screwed to the baseplate.

11. To view the position of the beam relative to the sample, press the **Go To Wavelength** icon, and enter **0nm**, then **Apply** to turn on the white light. Use an opaque material (a business card works well) to help view the beam spot, and align the sample as required.

12. Once properly aligned, press the **Start** icon to begin the scan. Press **OK** when prompted, again ensuring the lid is closed. To view the scan in real time, click the **Sample Info** tab.

13. Once the scan is complete, a message stating so will appear. If multiple samples are being run, you will be prompted to load the next sample and press OK. If alignment is needed for each sample, simply press Cancel, and perform the white light alignment described above. To stop scanning at any point, press the **Stop** icon.

14. Once all samples have been scanned, the data must be manually exported. Begin by selecting the **Reporting** tab. Ensure the **Output to file** box is checked, and click the **Setup** button to select an output location. A second window will open. Press the **Data Export** box to select a location to save the data. Select a format for the saved data as well, typically XY Data (raw), which will save the data as a .csv file. Press **OK** when complete.

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15. To save the data to the location just selected, go to File → Save As → Task. Edit the folder name, and press OK. The report seen in the Reporting tab may also be saved. Select File → Print → Hardcopy. Select Adobe PDF, and press OK. If needed, the method may also be saved, ensure that a unique name is given if using a default method.

16. Once everything is complete, **close the program** and **turn off** the system power. The computer should remain running.

Accessories

A reflectance accessory is available for fixed angle absolute specular measurement accessory. The fixed angle is 8º (PELA-6008). Note that we do not have the standard baseplate, and the accessory requires the standard baseplate to be changed with the thick Al plate, or used in combination with the optical bench. Absolute reflectance measurements directly measure the reflectance of the sample without the need for a reference sample. However, to verify that the accessory is properly aligned and the system is providing accurate data, periodic checking with calibrated mirror is recommended. Absolute reflectance measurements require an accessory in which the optical path is identical except for the presence or absence of the sample. The V-N optical configuration is a convenient way of achieving this.

Note that the setup can be tedious, but is critical to obtain accurate results. A calibrated reference mirror would make setup trivial, but are quite expensive and the nanoFab does not carry these. Refer to the manual for more detailed information.

Also available is the General Purpose Optical Bench (PELA-1003), with an integrating sphere   and a variety of magnetic mounted mirrors, allowing for custom optical layouts and data acquisition. Use caution and wear gloves when using the optical bench, as the mirrors are expensive and can be difficult to clean. Refer the the manual for more detailed information.

To change out the standard module, first ensure the main power switch if **Off** (this is very important). Start by removing the various parts of the system and setting aside in a safe place. Items to be removed include the lid of the sample chamber, the front panel of the sample chamber, the lid of the detector module, the sample chamber itself, and the detector module. When removing the detector module, ensure the mounting screws are loose, and grasp the handles securely. Lift the module straight up to avoid bending the detector pins.

To install the optical bench, ensure the handles are securely installed. Verify the two alignment pins are installed in the baseplate . Use caution as the bench is quite heavy; two people may be required. Lift the bench onto the baseplate, using the alignment pins as a reference. Once roughly in place, gently press the rear right corner down to seat the pins. The only way to know they are seated properly is by turning on the system, allowing it to warm up, and initializing. Failure to initialize indicates the pins are not seated properly.

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Remove the optical bench and re-install the standard module in reverse order. Again, ensure the main power is off before removing or installing either module.

TROUBLESHOOTING

The lamps are typically due for replacement after ~1500hrs of use. The software tracks the lamp hours, and will notify the user when the lamps are due. Please notify nanoFab staff if you see this message.

If you encounter an unexpected error or require assistance please contact the primary or secondary trainer listed above. Should they not be available, please contact any staff member for assistance.

APPROVAL

Qualified Trainer: Scott Munro
Training Coordinator: Stephanie Boric

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