Shimadzu UV/Vis/NIR Spectrophotometer (190 to 3300 nm)

Reserve time on the CCMR Coral scheduling system if desired. You may show up and use the system without a reservation, however users with reservations have priority.

There is a main compartment with sliding door for Transmission/Absorption measurements. We have one flat substrate holder and one cuvette holder (standard cell size is a Starna 1-Q-10 which holds about 3mL of fluid; other custom cells are available from Starna). There is also one internal specular reflection module (reference mirror required) and a module which directs light into the large sample compartment for absolute specular reflectance or diffuse reflectance (integrating sphere) measurements.

1) **To start up the Shimadzu:**
    a. Enable the tool in Coral. The power switch should be left on, so enabling turns the system on.
    b. Remove any samples from the beam path and close the sample compartment door.
    c. Double-click the **UVProbe 2.35** icon on the desktop. Typically the Spectrum module is used, but Kinetic or Photometric may be chosen for other techniques.
    d. Configure the spectrometer hardware and/or method as needed (see below).
    e. Click **Connect** to initialize the spectrometer. Full initialization takes ~8 minutes.
    f. Click **OK** after all self-tests finish (all should say "Passed" in blue).
2) **To set up Scan Method:**
    a. If you have a method saved, click **File, Open,** and select Files of Type "Method File (*.smd)" in the Shimadzu/UVProbe/Methods folder.
    b. If you don't have a method saved, you may start with one of the following:
        - 1_Transmittance283nmto3300nmDeuteriumLampOFF
        - 2_Transmittance190nmto3300nmAutoLamps
        - 3_Absorbance283nmto3300nmDeuteriumLampOFF
        - 4_Absorbance190nmto3300nmAutoLamps
        - 5_Reflectance283nmto3300nmDeuteriumLampOFF
        - 6_Reflectance190nmto3300nmAutoLamps

        Note that the higher wavelength range methods leave the deuterium lamp off, but it can be manually turned on any time.
    c. To modify test parameters, open the Methods box by clicking the green 'M' icon in the menu bar or pressing Ctrl-M. Enter the desired parameters in the **Measurement** tab. More changes can be made in the **Instrument Parameters** tab, including the **Detail** button for more advanced settings like single-beam measurement, fixed slit programs, and detector/light source change wavelengths.
3) **Baseline measurement -** When lamps have had sufficient time to warm up (at least 10-15 minutes, though ~30 minutes is desirable for precise UV measurements) take a baseline measurement. Baseline measurements may need to be repeated hourly for accuracy. Click **Baseline** to start. The software will ask for a wavelength range, and will default to the range specified in the method. Baselines should be performed with air in both Sample/Reference paths (or the equivalent 100% reflectance standards for reflectance measurements). Subsequent measurements should have a control sample in the reference path which is subtracted from the sample of interest in the sample path. (Note - There is an application note at the system comparing single-beam spectrometers to 2-beam spectrophotometers such as this one.)
4) **Sample Measurement -** Insert your sample and reference in the respective paths and click **Start**. The spectrum will be graphed in real time; axes can be adjusted by clicking and changing the end values or right-clicking and auto-scaling.

5) When the spectrum is complete, you will be prompted to save a spectrum (.spc) file, which is a proprietary format used by Shimadzu. (Note – You may install UVProbe to perform analysis on your own computer.) See "Measurement/Data Tips" for info on saving/exporting data in other forms.
6) In the default Overlay view, subsequent spectra will fill in on the same graph. Spectra to be displayed can be checked or un-checked in the list at the top-right of the software.
7) **When finished:**
    a. Remove samples and clean up. Please leave the area cleaner than you found it.
    b. Clean any cuvettes that you have used.
    c. Click **Disconnect** and close the software.
    d. **Disable the tool in Coral.** This will turn the spectrometer off.

Measurement/Data Tips

- **Auto Sampling Interval** will generally produce the best results, but manually setting the interval may be useful if higher resolution is needed.
- For reflectance measurements using the external chamber, change the **Detector Unit** to **External (2 Detectors)** and insert the mirror set into the main chamber.
- **Slit Width –** For typical absorption/transmission measurements in the UV/Vis range, use 5 nm. Use a larger slit width in order to get greater intensity in IR measurements and/or when using the external chamber (Shimadzu recommends the largest slit width which does not give a "Baseline Energy OVER!" message when the baseline is finished). The table attached to the spectrometer indicates the spectral band width used at various slit settings.
- **Source Lamp** can generally be left on **AUTO**, but setting it to **D2** (deuterium, UV) or **WI** (tungsten, Vis/IR) will leave the other lamp off (thus extending its life) if only one is needed.
- To save a method for future use, click **File – Save As…**, change the file type to **Method File (*.smd)**, and save.
- To see the light path, click **Go To WL,** set to 500 nm (green light), turn out the room lights, and place a piece of paper in the sample compartment.
- For sample alignment, follow the above step and visible green light will show where the light passes through the sample or sample holder. Use black electrical tape or another suitable mask if you need to make the analysis area smaller.
- It is possible to perform reflectance measurements, timed/kinetic measurements, as well as determination of sample concentrations. Consult the UV Probe software manual and/or facility staff for more information.
- To copy the full graph as an image, right-click in the graph and click **Copy**.
- The top left portion of the software window provides various functions for data viewing/analysis which are chosen using the **Operations** menu bar item.
    - **Data Print** shows numerical data for any spectra saved in the current session. Right-click and select **Properties** to change the displayed data interval and/or range (which do not need to equal the sampled data interval and range). Click **File – Save As…**, change the file type to **Data Print Table (*.txt)**, and save to save the displayed table.
    - **Peak Pick** shows a table of 'peaks' automatically detected by the software.
    - **Point Pick** allows the user to pick data points manually.
    - Any data in these tables can be selected and copied, then pasted as text or as a table in Excel.

Remote Access

The computer can be controlled remotely using TeamViewer software, which can be downloaded for free. The computer ID is 127 635 310 and the permanent password is ccmrshimadzu. Note that by using Coral and TeamViewer, you can turn on and initialize the spectrometer and take a baseline from your own computer.