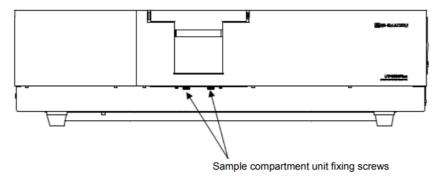
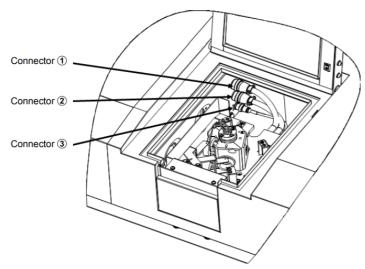
OPT4 Operating Procedure for Integrating Sphere Shimadzu UV-3600 UV-Vis-NIR Spectrophotometer

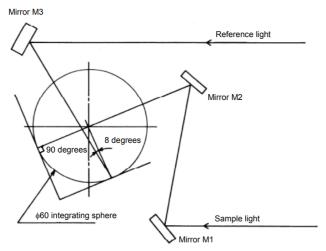
- 1. Create or Start a Reservation for OPT4 in CoreResearch
- 2. Load the integrating sphere into the instrument.
 - a. Remove the standard sample accessory inside the instrument:
 - i. Unscrew the front two screws until the screws drop.



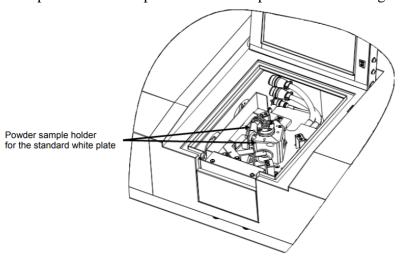
- b. Remove standard sample accessory.
- 3. Install the ISR. Note: The ISR should never be installed or removed when the instrument is on.
 - a. Make sure the slot at the back of the integrating sphere goes under the screw at the back of the holder.
 - b. Connect the cables.
 - i. The bottom outlet is for the small cable, "3".
 - 1. Note the direction of this cable.
 - ii. The middle outlet is for the cable labeled "2".
 - iii. The top outlet is for the cable labeled "1".



- c. Tighten the two screws at the front of the instrument.
- d. Note the optical diagram of the ISR:



- 4. If you feel any kind of tension, this means that the integrating sphere is not aligned.
- 5. Insert the white plates at the sample and reference position in the integrating sphere.



- 6. Turn on the green power switch located on the side of the UV instrument. Wait 20 minutes for instrument to warm up.
- 7. Open the "Lab Solutions UV-vis" on the computer.
- 8. Click "spectrum".
 - a. Environment settings is where the number of sig figs can be changed. Click number of digits and set values to change this.
- 9. Click "instrument control".
 - a. Load your previously saved method file. If you do not have a saved method file, load the "integrating sphere (ISR) default method".
- 10. To create or edit a method file, click "edit".
 - a. Enter starting and ending wavelength.
 - b. Set desired data interval (typical value is 1 nm).
 - c. Set desired scan speed (typical is medium).
 - d. Set the value type and measurement type to reflectance.
- 11. Click on "Advanced".
 - a. Set the desired slit width to 5 nm.
 - b. Slit program should be "standard".

- c. Light source should be "automatic".
- d. Detector unit, sample side should be "external (3 detectors)".
- e. S/R switch should be "standard".
- f. Ensure that "perform stair correction" is checked.
- g. Click "OK".
- 12. Click on "close after creating new parameter file".
 - a. Enter filename of new parameter file and save in parameter folder.
 - b. Click "close".
- 13. Set file and sample name.
- 14. Click on the "Connect" button. The system will go through several initialization checks that will take 10 minutes. When completed, click the OK button.
- 15. Perform a baseline correction
 - a. A baseline correction should be performed at start-up, and anytime you change the parameter settings.
 - b. Make sure the white plates are installed at the sample and reference position.
 - c. Click "baseline". Confirm wavelength range. Click "OK".
- 16. Load the sample into the sample position and keep the standard white plate in the reference position.
- 17. Click the "Start" button to initiate the measurement.
- 18. Adjust the overlay graph Y axis values as desired (the X axis will scale automatically)
- 19. When the scan is complete the data is only stored in temporary memory, but not to the computer disk.
- 20. To save the data to the computer disk:
 - a. On "data print table", there is an excel button. Open a blank excel book and then click the "excel" button. Your date should populate the open excel book. Save this file to your folder in the user data folder.
 - b. Click "text out" to save data as a .txt file.
- 21. To workup your data, click the "active tab" in the top left corner of graph.
 - a. "Peak" will peak pick for you. Click "execute" on the right side.
 - b. "Peak area" will give a peak area for you when you select a desired wavelength range.
 - c. "Peak pick" will let you select a peak and it will read its absorbance.
- 22. Shut down the system
 - a. Unload your sample and the plate at the reference position.
 - b. Click "disconnect" in the top right of the software.
 - c. Close the "Lab Solutions UV vis" software.
 - d. Turn off the green power switch on the side of the UV-3600 instrument
 - e. Unscrew the two screws at the front of the instrument.
 - f. Remove samples and standard white plates from the ISR.
 - g. Unplug the cables of the ISR. Be sure to rotate cable 3 to remove. Remove ISR and place back into plastic bag and then into box.
 - h. Install the standard sample accessory.
- 23. Stop or Update Actual Usage for your OPT4 reservation in CoreResearch