OPT4 Operating Procedure for Reflectance Attachment
Shimadzu UV-3600 UV-Vis-NIR Spectrophotometer

See "Instruction Manual – Specular Reflectance Attachment"

1. Create or Start a Reservation for OPT4 in CoreResearch
2. Turn on the green power switch located on the side of the UV-3600 instrument
3. Open the "Lab Solutions UV vis" software on the computer
4. 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.
5. Click "instrument control".
    a. Load your previously saved method file. If you do not have a saved method file, load the "spectrum default method".
6. To create or edit a method file, click "edit".
    a. Enter starting and ending wavelength.
    b. Set desired data interval (typical is 1 nm).
    c. Set desired scan speed (typical is medium).
    d. Set the value type and measurement type to reflectance.
7. Click on "Advanced".
    a. Set the desired slit width between 2-5 nm (typical is 5 nm).
    b. Slit program should be "standard".
    c. Light source should be "automatic".
    d. Detector unit, sample side should be "direct receiving of light".
    e. S/R switch should be "standard".
    f. Ensure that "perform stair correction" is checked.
    g. Click "OK".
8. Click on "close after creating new parameter file".
    a. Enter filename of new parameter file and save in parameter folder.
    b. Click close.
9. Set file and sample name.
10. Make sure that no samples are present in the sample chamber and that the sample chamber cover is closed
11. Click on the "Connect" button. The system will go through several initialization checks that will take 10 minutes. When completed, click the OK button.

Click HERE for OPT4 Reflectance Sample Holder Mounting Video

12. Load the reflectance sample holder in the sample compartment
    a. Remove the sample holder from the sample compartment
    b. Install the reflectance attachment with the "S" (sample) side to the front and tighten the screw in the center to fix it in place

13. Perform a baseline correction as follows:
    a. Screw the appropriate sample stages (5mm or 15mm aperture depending on the sample size) onto the reflectance attachment. Note that the small stage

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labeled “R” goes on the back reference beam path position, and the large stage labeled “S” goes on the front reference beam path position.

b. ***Do not touch the mirror surfaces with your fingers***. Mount the two mirror assemblies on the respective S and R sample stages with their mirror faces down. Note that the mirror for the reference position is labeled “R” and the mirror for the sample position is labeled “S”.
c. Close the sample compartment cover
d. Click “Baseline” in the software, verify the wavelength range and then click OK to start the baseline measurement

Click HERE for Reflectance Sample Loading and Spectra Acquisition Video

14. Load the sample:
    a. Remove the sample side reflecting mirror (keep the reference side mirror in place) ***Do not touch the mirror surfaces with your fingers***
    b. Place the sample face-down on the sample side stage
    c. Close the sample compartment cover
15. Click the “Start” button on the instrument bar to initiate the measurement
16. Adjust the overlay graph Y axis values as desired (the X axis will scale automatically)
17. When the scan is complete ***the data is only stored in temporary memory, but not to the computer disk.***
18. 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.
19. 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.
20. Shut down the system
    a. Unload your sample
    b. Remove the mirror assembly from the reference side. ***Do not touch the mirror surfaces with your fingers***
    c. Remove the sample stages
    d. Loosen the screw in the center of the reflectance attachment and remove the attachment from the sample chamber
    e. Put all mirrors, samples stages, and the reflectance attachment back in the black storage box
    f. Re-install the transmission sample stage and close the sample chamber cover
    g. Click “disconnect” in the top right of the software.
    h. Close the “Lab Solutions UV vis” software.
    i. Turn off the green power switch on the side of the UV-3600 instrument
21. Stop or Update Actual Usage for your OPT4 reservation in CoreResearch