Section 5. 17 Title: Using the UV-Vis Spectrometer **Revision Date:** 11/01/19
Prepared By: Christian Wallen and Michael Roy **P.I.:** Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
___ Carcinogen ___ Reproductive Toxin ___ High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:
Collection of electronic absorption data using visible and infrared light *via* dip probe in a sample solution.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*
Daniels Chemistry - 6315A

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
N/A	N/A

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*
No inherent hazards, but flammable solvents may be used in sample solutions.
Consult relevant SOP(s) and SDS as needed.

Exposure Controls: *(Check all that apply)*
PPE: _X_ Safety Glasses ___ Face Shield ___Chemical Splash Goggles
___Chemical Apron _X_ Gloves (Nitrile) _X_ Lab Coat
___Respirator (type) ___Other:

Engineering Controls:
___ Fume Hood ___Biosafety Cabinet ___ Glove box
___ Vented gas cabinet _X_ Other: Nitrogen line for air-free systems.

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

N/A

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Dispose of samples according to relevant SOPs and SDS.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

Training is required. Training is performed by the group member(s) responsible for this apparatus or another lab member they have approved. The procedure will be demonstrated at least once and new members will be supervised their first time.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry Signature: _____ Date: _____

Using the UV-Vis Spectrometer

Warmup

1. Locate the detector's power button on the USB hub and press to power on.
2. Turn on the light source using push-button switch on the back of the unit.
3. Allow the source to warm up for 30 minutes to achieve the most accurate results.
4. Write your initials, the date, and whether you are using N_2 in the logbook.

Collecting data

1. Remove the yellow plastic protector and clean the surface of the dip probe with a lab wipe.
2. Select the appropriate tip from UV-Vis drawer (2mm, 5mm, 10mm, 20mm) and carefully screw the tip onto the end of the dip probe.
3. Open SpectraWiz on the desktop. The software should automatically open in scope mode. If not, switch to scope mode by choosing View>Scope mode.
4. Insert the dip probe into sample of pure solvent and adjust the detector integration time using the scroll wheel, by clicking the wall clock button on the toolbar, or by navigating to Setup>Detector Integration Time. Detector integration time is constantly displayed on the bottom toolbar display (i.e. Time: 3ms). Choose the longest time that does not clip the top of the scope mode graph. Record the integration time in case it is accidentally changed later. This value can be adjusted later, but new light and dark references will need to be collected (see step 7).
5. Adjust the number of scans to average by navigating to Setup > Number_of_Scans_to_Average. This number is constantly displayed on the bottom toolbar display (i.e. Avg: 10). Choose a value that is long enough that the live spectra feed does not fluctuate significantly. This value can be adjusted later, but new light and dark references will need to be collected (see step 7).
6. If you are using the solvent sample from step 4 as your background, then skip to step 7. If not, remove the dip probe from the solvent sample and dry it with lab wipe, making sure to clean out all solvent from the inside of the tip. Insert the dip probe into your blank sample.
7. Switch to absorbance mode (View>Absorbance), power off the light source using the power button on the back of the unit, and left click dark lightbulb button to collect "dark" reference spectrum. Power on the light source and left click yellow lightbulb button to collect "light" reference spectrum. Note: ignore noise under 400nm.
8. Remove dip probe from your blank sample and dry the probe and tip thoroughly using lab wipe.
9. Insert dip probe into your first sample solution. Give the probe a gentle but quick shake to ensure that no bubbles are trapped inside the tip. For air-free work, use a septum with bored hole or glass thermometer adapter found in UV-Vis cabinet drawer and the nearby nitrogen Schlenk line (the valve is located behind IR). The vacuum pump has a switch on the power outlet under the manifold.
10. If the spectrum is satisfactory, then save it using the Save button on the toolbar or by choosing File>Save>Sample and navigating to Shortcut_to_data > UV-Vis > Berry > "Name" and then choosing your desired experiment folder. It is recommended to use

your initials and experiment number as the file name (example: CMW_067_01_solutionA). Save as .ABS. This file can be imported into Excel.

11. The first time you save a file after opening the program, an "export parameters" window will come up next. Type in the minimum wavelength, the wavelength interval, and the maximum wavelength into the corresponding boxes and select OK. These parameters will be used for all consecutive scans until you close the software or change then under File > Save > Export.
12. Remove the dip probe from your sample, rinse it with solvent (i.e. acetone) into a waste beaker, and clean the dip probe thoroughly using a lab wipe. The probe can then be inserted into the next sample solution.
13. When finished, thoroughly clean the probe and tip. Carefully remove tip and return in to the appropriate container in the UV-vis drawer. Wipe off the end of probe and replace the yellow plastic cover.
14. Close the software and power down the light source and the detector.
15. Check off your name in the logbook by writing a check mark under “Comments” column.

Notes on prepping samples

1. Make sure your solvent/solution is as clear as possible. When possible, filter solution through celite or syringe filter to remove particulate matter (i.e. dust from molecular sieves).
2. To collect NIR data, change the fiberoptic cable from the BLACK-Comet spectrometer (200-1100 nm) to the DWARF-Star (900-1700 nm). Follow the above instructions but the NIR probe needs to be turned on at the USB hub and an additional power switch to the right of the computer needs to be turned on to provide power to the NIR detector.
3. It is possible to use a Y-adaptor to perform simultaneous Vis-NIR. This has not always worked reliably. Consult manufacturer’s instructions for further information.

Episodic data collection

1. If you wish to collect data at specific time intervals, do not use the SpectraWiz software episodic data collection feature, since it is unreliable. However, there is an AutoHotKey program (GUIUV) located on the desktop that can be used to reliably gather scans with SpectraWiz at precise time intervals. Note: Do not initiate your reaction yet.
2. Under the "Comments" column in the logbook, write that you are performing an extended measurement so others know not to disrupt your sample or the computer.
3. Before starting the program, follow steps 1-11 from “Collecting Data” on your “time 0” sample. The episodic data collection will collect using the same parameters and save files to the same folder used for this “time 0” scan.
4. Before adding the final reagent (or other initiation step), minimize SpectraWiz and open the “GUIUV” program (“H” on green background). The program will warn you to complete initial steps above. Click “OK” to continue or “Cancel” to terminate the program.
5. In the “Parameters” window, type in your desired time interval in seconds, the maximum number of scans, and the filename text (example: CMW_068_01) in the corresponding boxes and then press “OK”. Note: the program can be stopped before collecting all the scans, so err on the side of too many scans.

6. The window will display the parameters you just typed in. If you wish to change them press Cancel and go back to step 3. Otherwise, you are ready to initiate your reaction. Add your final reagent, immediately press OK, and then DO NOT TOUCH THE MOUSE OR KEYBOARD.
7. The program will move the cursor at the designated time intervals to collect each spectrum. You can move the mouse and use the computer in between scans, but if you move the mouse during a collection it can mis-click, resulting in a skipped scan, the reference being reset, or the program closing. Therefore, it is best to avoid using the computer during collections.
8. The program will collect until the maximum number of scans (entered in step 4) is reached or until you terminate the program early. If you want to stop the collection before reaching the maximum scans you entered, wait until a break in the collection, move the mouse to the green H icon in the system tray (lower right), right-click the icon, and click "Terminate program".
9. All the scans will be saved in the folder from step using the filename text (entered in step 4) ending in "_1", "_2", etc. (Example: CMW_01_068_1, CMW_01_068_2, etc.)