

# IMSERC User Manual for UV-Vis-NIR Spectrometer

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# IMSERC User Manual for UV-Vis-NIR Spectrometer (v1.02)

## INTRODUCTION

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Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at <http://imserc.northwestern.edu/pcm-instruments.html>. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and available under the corresponding instrument section at <http://imserc.northwestern.edu/pcm-instruments.html> by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please **stop**, and **get help**. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

## SAFETY

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All users of IMSERC must review the general safety policies at <http://imserc.northwestern.edu/about-policies.html>. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at <https://learn.northwestern.edu>. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room B172 at the north side. Protective eyewear is required in this room, and gloves should be removed when using the computer.

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## DATA MANAGEMENT

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Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied in your supervisor's group folder on 'imsercdata.northwestern.edu' under 'others/UV-Vis' every 15 minutes. Please follow instructions at <http://imserc.northwestern.edu/about-general-faq.html> for details about data access.

## SOFTWARE

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No additional software is needed for data analysis. Please export your data as CSV for offline analysis.

## DEFAULT INSTRUMENT STATUS

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The default measurement mode of the UV-Vis-NIR spectrophotometer is **diffuse reflectance for powders**. Please notify the right [staff member](#) well in advance if you would like to run an experiment in a different mode than the one listed above, and you are not trained to perform the require mode-switch. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at <http://imserc.northwestern.edu/pcm-instruments.html>.

The default working condition of the Cary 5000 UV-Vis-NIR spectrometer is as follows:

1. Computer screen is by default deactivated. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore
2. The default 'UV' user account should be logged in. In case the computer was restarted, the password for the 'UV' account is \_\_\_\_\_ (see hardcopy by the instrument)
3. The main power switch located at the bottom left front side of the spectrometer must be at the 'Off' position
4. Front cover of the sample chamber and top cover must be closed (figure 4)
5. The diffuse reflectance accessory must be installed, and the reference powder cell with pure BaSO<sub>4</sub> must be installed on the accessory

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When you are done with your measurement, please remember to:

1. Close the acquisition software 'Scan'
2. Turn off the main power switch located at the bottom left front side of the spectrometer
3. End your reservation in NUcore and (if applicable) add the appropriate accessories based on the measurements you ran
4. Leave lab tables and drawers clean and tools/accessories organized

If there is an error or problem with the instrument which is not addressed under the '[Troubleshooting](#)' section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
3. Contact a staff member for instructions



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## GENERAL STEPS FOR DATA ACQUISITION

### A. MEASURE POWDER SAMPLES

Please follow the general steps for basic data acquisition on powder samples using the diffuse reflectance accessory which is the default mode of operation. See a staff member when you'd like to perform measurements in a different mode or configuration:

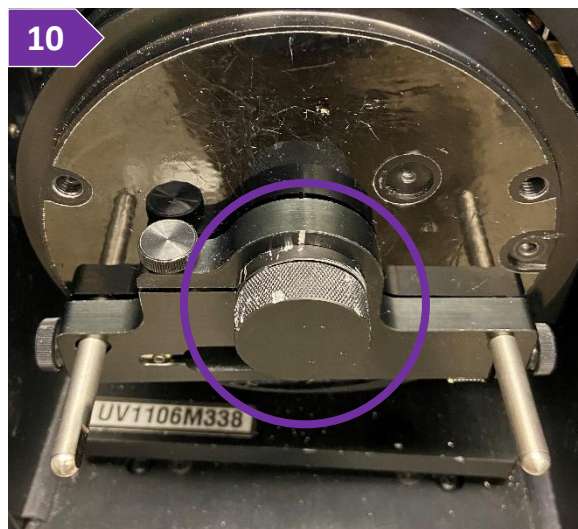
1. Verify that instrument is off by checking that the power switch at the bottom left front side of the spectrometer
2. Ensure that the diffuse reflectance attachment is installed (figure 2). Please, do not install/uninstall an attachment unless you have been trained and authorized by IMSERC staff to do so
3. Turn the spectrometer on by flipping the power switch to the 'on' position. Green light next to the switch will turn on
4. Instrument will go through a series of internal check steps and initialize for about one minute
5. While waiting for the initialization process, start the acquisition software by double-pressing on the desktop icon called 'Scan'. Initialization status is shown at the bottom left corner of the acquisition window
6. Once the initialization is complete and all components have been checked by the system, the large 'Start' button with the traffic light icon at the top of the window will be enabled
7. Press on the 'Setup' button located on the left side of the window and wait for a few seconds until the 'Setup' window appears on the screen
8. On the 'Setup' window:
  - a. Select the option 'Cary Options' from the left tree menu and select the appropriate options on the right panel:
    - i. Under the 'X Mode' panel, select the wavelength units and the appropriate spectral window for the measurement. Note that every time the spectral window is modified, a new baseline must be collected
    - ii. Under the 'Y Mode' panel, select the units for the intensity axis. Units will be printed in the exported CSV file
    - iii. [Optional] Under the 'Cycle' panel, provide the number of cycles/repetitions needed for the measurement



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- iv. [Optional] Under the 'Scan Controls' panel, adjust integration time, wavelength step, and scan rate. It is recommended not to change these values before evaluating a collection performed with the default scan controls, i.e., 0.1 s for average time, 1 nm for data interval, and 600 nm/min for scan rate
- b. Select the option 'Baseline' from the left tree menu and select the appropriate options on the right panel:
  - i. Most common option is the 'Zero/baseline' correction setting. Leave all other options unselected
  - ii. [Optional] In case you have a baseline measurement saved in a file, you have the option to reuse this baseline by pressing on the 'Retrieve Baseline file' button and loading the corresponding baseline file. It is highly recommended to measure the baseline once just before the measurement of all your samples in each session
9. After checking all options under the 'Cary Options' and 'Baseline' panels, press on the 'OK' button at the bottom of the 'Setup' window

10. Ensure that the reference cell loaded with pure BaSO<sub>4</sub> is mounted onto the diffuse reflectance attachment (figure 10)



11. Press on the 'Baseline' button at the left of the acquisition window.

- a. System will prompt to 'Insert a blank sample into the sample compartment to collect the 100% transmittance baseline scan'
- b. Press on the 'Ok' button
- c. Scan of the 100% transmittance collection will start
- d. Once scan is complete, system will prompt to 'Remove the sample to collect the 0% transmittance baseline scan' (do not press ok yet)
- e. Remove the reference cell from the diffuse reflectance attachment and close the covers (figure 11e)
- f. Press 'Ok' to collect the 0% transmittance baseline scan
- g. Once both baseline collections (100% and 0 %) are complete, you are ready to measure your sample(s)





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12. Mix your powder sample with BaSO<sub>4</sub> and pack it into the sample cell (not the reference cell) as instructed during training (figure 12)
13. Mount the sample cell onto the diffuse reflectance accessory
14. Press on the 'Start' button that has the traffic light icon to start the scan

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- a. System will prompt for a DSW file name. The DSW binary file might contain more than one sample collections, i.e., you have the option to save more than one spectrum/sample in a single binary DSW. Once you provide a filename and press on the 'Ok' button, system will prompt for the sample name (see step below)
- b. Provide the sample name (figure 14b), press on the 'Ok' button, and collection will start

15. Once the collection is complete, the system will prompt for the next sample name. If you want to save one sample name per DSW file or you are collecting on your last sample, press on the 'Finish' button. By pressing on the 'Ok' button, a new acquisition will start immediately, therefore ensure that you have loaded the next sample onto the attachment before pressing on the 'Ok' button



16. At any time, you have the option to select/unselect which spectra are plotted on the screen by pressing on the icon with 'S1, S2, S3' right next to the 'Setup' button. All visible spectra will be saved in a single CSV file upon export

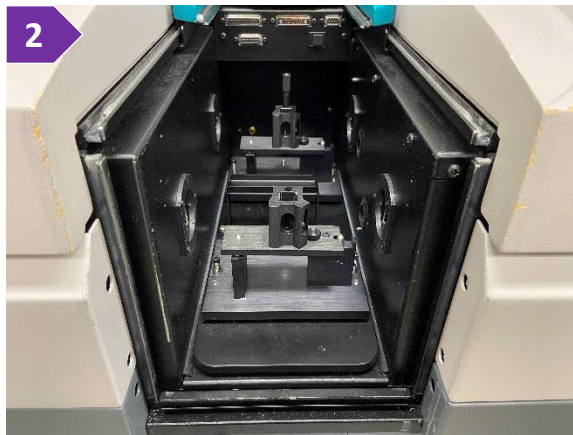
17. To export your data as CSV
  - a. Select 'Save as' under the 'File' menu
  - a. On the 'Save as' window, change the file type to CSV
  - b. Browse to your person folder
  - c. Press on the 'Save' button
  - d. All selected spectra and their corresponding intensity units will be saved in the CSV file
18. Close the 'Scan' software

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## B. MEASURE LIQUID SAMPLES

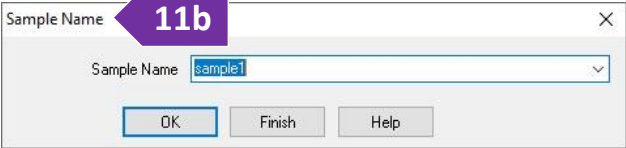
Please follow the general steps for basic data acquisition on liquid samples using the cell transmission accessory. See a staff member to have the cell attachment installed since by default the diffuse reflectance accessory is mounted on the spectrometer:

1. Verify that instrument is off by checking that the power switch at the bottom left front side of the spectrometer
2. Ensure that the cell transmission attachment is installed (figure 2). Please, do not install/uninstall an attachment unless you have been trained and authorized by IMSERC staff to do so
3. Turn the spectrometer on by flipping the power switch to the 'on' position. Green light next to the switch will turn on
4. Instrument will go through a series of internal check steps and initialize for about one minute
5. While waiting for the initialization process, start the acquisition software by double-pressing on the desktop icon called 'Scan'. Initialization status is shown at the bottom left corner of the acquisition window
6. Once the initialization is complete and all components have been checked by the system, the large 'Start' button with the traffic light icon at the top of the window will be enabled
7. Press on the 'Setup' button located on the left side of the window and wait for a few seconds until the 'Setup' window appears on the screen
8. On the 'Setup' window:
  - a. Select the option 'Cary Options' from the left tree menu and select the appropriate options on the right panel:
    - i. Under the 'X Mode' panel, select the wavelength units and the appropriate spectral window for the measurement. Note that every time the spectral window is modified, a new baseline must be collected
    - ii. Under the 'Y Mode' panel, select the units for the intensity axis. Units will be printed in the exported CSV file
    - iii. [Optional] Under the 'Cycle' panel, provide the number of cycles/repetitions needed for the measurement





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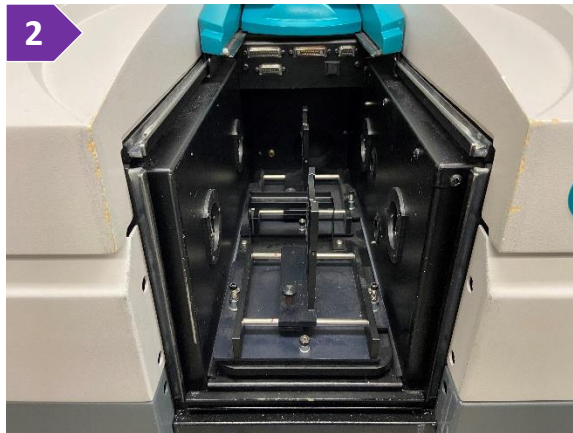
- iv. [Optional] Under the 'Scan Controls' panel, adjust integration time, wavelength step, and scan rate. It is recommended not to change these values before evaluating a collection performed with the default scan controls, i.e., 0.1 s for average time, 1 nm for data interval, and 600 nm/min for scan rate
9. Ensure that the reference cell (back position) is loaded with neat solvent or an empty cuvette
10. Mount the cuvette with the sample in the front position
11. Press on the 'Start' button that has the traffic light icon to start the scan
  - a. System will prompt for a DSW file name. The DSW binary file might contain more than one sample collections, i.e., you have the option to save more than one spectrum/sample in a single binary DSW. Once you provide a filename and press on the 'OK' button, system will prompt for the sample name (see step below)
  - b. Provide the sample name (figure 11b), press on the 'OK' button, and collection will start
12. Once the collection is complete, the system will prompt for the next sample name. If you want to save one sample name per DSW file or you are collecting on your last sample, press on the 'Finish' button. By pressing on the 'OK' button, a new acquisition will start immediately, therefore ensure that you have loaded the next sample onto the attachment before pressing on the 'OK' button
13. At any time, you have the option to select/unselect which spectra are plotted on the screen by pressing on the icon with 'S1, S2, S3' right next to the 'Setup' button. All visible spectra will be saved in a single CSV file upon export
14. To export your data as CSV
  - a. Select 'Save as' under the 'File' menu
  - b. On the 'Save as' window, change the file type to CSV
  - c. Browse to your person folder
  - d. Press on the 'Save' button
  - e. All selected spectra and their corresponding intensity units will be saved in the CSV file
15. Close the 'Scan' software

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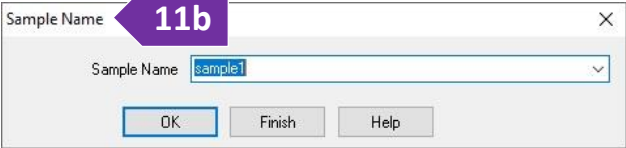
## C. MEASURE TRANSLUCENT SOLID SAMPLES OR THIN FILMS

Please follow the general steps for basic data acquisition on solid samples using the flat plate transmission accessory. See a staff member to have the cell attachment installed since by default the diffuse reflectance accessory is mounted on the spectrometer:

1. Verify that instrument is off by checking that the power switch at the bottom left front side of the spectrometer
2. Ensure that the cell transmission attachment is installed (figure 2). Please, do not install/uninstall an attachment unless you have been trained and authorized by IMSERC staff to do so
3. Turn the spectrometer on by flipping the power switch to the 'on' position. Green light next to the switch will turn on
4. Instrument will go through a series of internal check steps and initialize for about one minute
5. While waiting for the initialization process, start the acquisition software by double-pressing on the desktop icon called 'Scan'. Initialization status is shown at the bottom left corner of the acquisition window
6. Once the initialization is complete and all components have been checked by the system, the large 'Start' button with the traffic light icon at the top of the window will be enabled
7. Press on the 'Setup' button located on the left side of the window and wait for a few seconds until the 'Setup' window appears on the screen
8. On the 'Setup' window:
  - a. Select the option 'Cary Options' from the left tree menu and select the appropriate options on the right panel:
    - i. Under the 'X Mode' panel, select the wavelength units and the appropriate spectral window for the measurement. Note that every time the spectral window is modified, a new baseline must be collected
    - ii. Under the 'Y Mode' panel, select the units for the intensity axis. Units will be printed in the exported CSV file
    - iii. [Optional] Under the 'Cycle' panel, provide the number of cycles/repetitions needed for the measurement



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- iv. [Optional] Under the 'Scan Controls' panel, adjust integration time, wavelength step, and scan rate. It is recommended not to change these values before evaluating a collection performed with the default scan controls, i.e., 0.1 s for average time, 1 nm for data interval, and 600 nm/min for scan rate
9. Ensure that the reference plate (back position) is loaded with an empty substrate (case of a film) or nothing
10. Mount the solid sample or thin film in the front position
11. Press on the 'Start' button that has the traffic light icon to start the scan
  - a. System will prompt for a DSW file name. The DSW binary file might contain more than one sample collections, i.e., you have the option to save more than one spectrum/sample in a single binary DSW. Once you provide a filename and press on the 'OK' button, system will prompt for the sample name (see step below)
  - b. Provide the sample name (figure 11b), press on the 'OK' button, and collection will start
12. Once the collection is complete, the system will prompt for the next sample name. If you want to save one sample name per DSW file or you are collecting on your last sample, press on the 'Finish' button. By pressing on the 'OK' button, a new acquisition will start immediately, therefore ensure that you have loaded the next sample onto the attachment before pressing on the 'OK' button
13. At any time, you have the option to select/unselect which spectra are plotted on the screen by pressing on the icon with 'S1, S2, S3' right next to the 'Setup' button. All visible spectra will be saved in a single CSV file upon export
14. To export your data as CSV
  - a. Select 'Save as' under the 'File' menu
  - b. On the 'Save as' window, change the file type to CSV
  - c. Browse to your person folder
  - d. Press on the 'Save' button
  - e. All selected spectra and their corresponding intensity units will be saved in the CSV file
15. Close the 'Scan' software

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## PUBLICATION

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### A. EXPERIMENTAL SECTION

*Modify the text below according to the setup and conditions you used during the measurement:*

Diffuse reflectance: “The UV-Vis-NIR spectrum in diffuse reflectance mode was collected on lightly grounded powder using a Cary 5000 UV-Vis-NIR double beam spectrophotometer with a monochromator. BaSO<sub>4</sub> powder was used for the baseline collection, whereas a mixture of sample powder with BaSO<sub>4</sub> was used for the data collection at room temperature. Absorbance data was converted from reflectance data in-software using the Kubelka-Munk equation,  $\alpha/S = (1-R)^2/2R$ , where  $\alpha$  and  $S$  are the absorption and scattering coefficients, respectively, and  $R$  is the reflectance.”

Transmittance: “The UV-Vis-NIR spectrum in transmittance mode was collected at room temperature on a solid sample using a Cary 5000 UV-Vis-NIR double beam spectrophotometer with a monochromator. An empty substrate was used as a reference for the baseline collection”

### B. ACKNOWLEDGEMENT

“This work made use of the IMSERC facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University.”

# IMSERC User Manual for UV-Vis-NIR Spectrometer (v1.02)

## TROUBLESHOOTING

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### A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

### B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default 'UV' user account should be logged in. In case the computer was restarted, the password for the 'UV' account is \_\_\_\_\_ (see hardcopy by the instrument). See '[Default instrument status](#)' section for more details

### C. SOFTWARE WARNING AND ERROR MESSAGES

Sometimes various warning and error messages might appear. Please check the list below with known messages and solutions:

**1. When the 'Scan' acquisition software is (re-)started, a system exception regarding 'Floating point division by zero' occurs.**

Simply acknowledge the exception by pressing on the 'OK' button. This error message does not appear to create any problems with the acquisition or analysis.

### D. THERE IS AN ERROR OR PROBLEM WITH THE INSTRUMENT NOT LISTED IN THIS SECTION

If there is an error or problem with the instrument that is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
3. Contact a staff member for instructions

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## REVISIONS

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v1.02 2022/08/31	<ul style="list-style-type: none"><li>• Release of original version of the user manual for the spectrophotometer</li></ul>
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