

UV-Vis Spectrometer Manual (Cary 5000)

Instrument Summary:

The Agilent Technologies Cary 5000 UV-Vis-NIR spectrometer offers high quality photometric performance with a 200 – 3300 nm spectral range. The Cary 5000 facilitates both absorbance and transmission measurements of solid and liquid based samples. Through utilization of a PbSmart detector, its detection range is extended into the NIR range. Additional configurations allow the incorporation of a universal measurement accessory to facilitate external diffuse reflectance measurements, permitting the measurement of opaque substances and the ability to perform kinetic studies.



Figure 1. The Agilent Technologies Cary 5000 UV-Vis-NIR spectrometer.

Safety and Precautions:

- **Warning:** Do not lift the UMA by yourself. Ask IAC staff for assistance when mounting the UMA.
- **Warning:** Check the compatibility of liquid samples with your cuvettes. Plastic cuvettes (Ex: Polystyrene) can absorb organic liquids (Ex: Toluene) and can leak in the instrument.
- **Warning:** Use the fume hood when preparing liquid and/or volatile samples.
- **Warning:** Never plug or unplug anything while the instrument is turned on.

I. Experiment Planning:

1. **Determine the Instrument Setup** – If you are using the UMA, schedule adequate time for instrument setup and reconfiguration after the conclusion of your experiment.
2. **Sample Considerations** – Consider the type of sample that you will be using, how the sample will be mounted, and any safety considerations. A fume hood is available for liquid samples.
***Cuvette Note:** Check the compatibility of liquid samples with your cuvettes. Plastic cuvettes (Ex: Polystyrene) can absorb organic liquids and could leak in the instrument.

II. Instrument Start Up and Sample Configuration:

1. **Turn the Instrument On** – Flip the “Toggle Switch” on the bottom left corner of the machine.
2. **Instrument Warm Up** – Allow the UV-Vis to warm-up for at least 20 minutes before continuing.
3. **Insert the Appropriate Sample Holder** – The Cary 5000 UV-Vis spectrometer can be configured to accept both solid and liquid samples.
 - **Changing the Holder:** Flip the lever on the front to the right to unlock the stage area. Remove the front panel by sliding it upwards. Remove the current sample holder by lifting up and out. Insert your sample holder. Match the three pegs with the holes. Flip the locking lever on the front to the left to lock the stage. Replace the front panel.
4. **Load Your Sample and a Reference Sample** – Load the sample that you would like to analyze in the front holder and a reference sample (if necessary) in the back holder.
 - **Films:** Select an aperture plate with a desired dimension, tape your film to the plate.
 - **Cuvettes:** Insert a non-frosted cuvette in each holder.
5. **Close the Sliding Door Located on top of the Instrument.**

III. Experimental Setup and Operation:

Note: The software has been pre-configured for two operating modes. These include a standard mode for operation without the UMA (see *noUMA_BaseMethod*) and a mode for use with the UMA (see *UMA_BaseMethod*).

1. **Open the UV-Vis Software** – For standard measurements which do not utilize the UMA, open the desktop icon titled: “*noUMA_BaseMethod*”.
2. **Select the Operating Conditions** – Click the “Setup” button and enter the desired operating conditions under: “Summary” → “Instrument Settings”.
 - **X-Mode:** Nanometers, Wavenumber, Angstroms, etc.
 - **Y Mode:** Absorbance, %Transmission, etc.
 - **Starting Range:** Variable (e.g. 800-250 nm)
 - **Beam Mode:** Double
 - **Slit Height:** Reduced
 - **Scan Rate:** ~600 nm/min
 - **Use UMA:** Off
 - **Spectral Band Width (SBW):** 4.0 nm
 - **Data Interval:** 1 nm
 - **Baseline Correction:** On
 - **UV-Vis Detector Changeover Wavelength:** 850 nm (*Important:* keep at 850 nm)
 - **Baseline Settings** – It is standard to select “Baseline Correction”.
3. **Auto Convert Data** – Under the “Reports Tab”, instruct the instrument to “Convert Data to .csv”.
4. **Save the Settings** – Press the “Ok” button in the bottom right corner of the setup window.

IV. Running an Experiment:

1. **Run a Baseline Scan** – In the main window, press the “Baseline” button. Configure the samples as desired in the instrument and press “Ok” to begin.
2. **Analyze the Sample** – Wait until the Baseline scan has finished. Press the “Start” button when it becomes available. Reposition the samples as necessary and select the location to save the file. Press the “Ok” button to begin the run. Press “Finish” to end the run.
3. **Data Collection** – The data has been pre-saved as a .csv file by setting the instrument to “Auto convert”. Additionally, the data can be set to save as a .dsw or .bsw by clicking *File* → *Save As* etc.

V. Running UMA (Universal Measurement Accessory) Experiments:

1. **Turn off the Instrument** – Make sure the instrument is completely powered down before attempting to attach the UMA. *Ask for help configuring the UMA prior to your 1st experiment.*
2. **Configure the UMA** – Remove the current stage and front cover. Insert the UMA stage into the UV-Vis. Plug in the cables. Insert desired apertures. Place the UMA box over the stage. Close the top cover.
3. **Turn on the Instrument** – Flip the toggle switch, allow the instrument to warm up for 20 min.
4. **Open the Software** – Open the “*UMA_BaseMethod*” file and allow it to initialize.
5. **Select the Operating Conditions** – Click the “*Setup*” button and enter the desired operating conditions under: “*Summary*” → “*Instrument Settings*”.
 - **Use UMA:** On
 - **Spectral Band Width (SBW):** 4.0 nm
 - **NIR Data Interval:** 1 nm
 - **Zero/Baseline Correction:** On
 - **Positive/Negative Angle Measurement:** Off
 - **UV-Vis Detector Changeover Wavelength:** *Important:* keep at pre-set value.
 - **Select Polarizer Settings** – Toggle the polarizer on or off as needed.
 - **Select Measurement Angles** – Multiple angles of interest can be selected.
6. **Save the Settings** – Press the “*Ok*” button in the bottom right corner of the setup window.
7. **Start the Experiment** – See [Section IV](#) above. To run a baseline you will need to run a control experiment before inserting your sample at the angles of interest and manually subtract this data from a second experiment with your sample inserted in the path of the beam.

VI. Running DRA (Diffuse Reflectance Accessory) Experiment:

1. **Turn off the Instrument** – Make sure the instrument is completely powered down before attempting to mount DRA. *Ask for help configuring the DRA prior to your 1st experiment.*
2. **Configure the DMR** – Remove the current stage and front cover. Insert the DRA unit into the UV-vis. Plug in the cable. Mount the sample. Place the DRA box over the stage. Close the top cover.
3. **Turn on the Instrument** – Flip the toggle switch, allow the instrument to warm up for 20 min.
4. **Open the Software** – Open the “*noUMA_BaseMethod*” file and allow it to initialize.
5. **Select the Operating Conditions** – Click the “*Setup*” button and enter the desired operating conditions under: “*Summary*” → “*Instrument Settings*”.
 - **X-Mode:** Nanometers, Wavenumber, Angstroms, etc.
 - **Y Mode:** %R
 - **Starting Range:** Variable (max. 900-200 nm)
 - **Beam Mode:** Double
 - **Slit Height:** Reduced
 - **Scan Rate:** < 600 nm/min
 - **Use UMA:** Off
 - **Spectral Band Width (SBW):** 2.0 nm
 - **Data Interval:** 0.5 - 1 nm
 - **Baseline Correction:** On
 - **UV-Vis Detector Changeover Wavelength:** 850 nm (*Important:* keep at 850 nm)
 - **Baseline Settings** – It is standard to select “*Baseline Correction*”.
6. **Save the Settings** – Press the “*Ok*” button in the bottom right corner of the setup window.

7. **Start the Experiment** – See **Section IV** above. To run a baseline, you will need to run a control experiment with an appropriate ~100% reflective standard.

VII. Instrument Shutdown:

1. **Turn off the Instrument** – Flip the main “**Toggle Switch**” and close the software.
2. **Sign the Log Book.**
3. **Log Off of the Instrument.**

Safety requirements

The laboratory in room ACEE 027B is a wet chemistry lab, in which some researchers may handle hazardous substances. The lab safety requires that users wear **eye protection** (safety goggles or glasses with side shields), **lab coats**, and **gloves** while using equipment in this room.

Emergency Information:

Medical Emergencies: **Contact 911 and Public Safety (609) 258-1000**

Room / facility emergencies: **Contact Public Safety (609) 258-1000**

Issues related to the instrument:

1. **Contact IAC Staff.**
2. **If unsure, leave system as is.**
3. **Try to power down the system.**

Audible/Siren Emergency Alerts:

Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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