



CENTER FOR MICROANALYSIS OF MATERIALS
LASER AND SPECTROSCOPY FACILITY

Varian CARY 5G

Operation instructions

MAKE SURE YOU SIGNED UP FOR TIME ON THE ON-LINE SCHEDULE FOR THIS INSTRUMENT **BEFORE USING IT.**

LOG YOUR USE ON THE LOG BOOK BY THE INSTRUMENT.

Specifications

Spectral Range:

175-3300nm (0.376-7.08eV) (3030-57140 wavenumbers)

Available accessories:

- Diffuse reflectance
- Specular reflectance
- Total fluorescence
- Liquid sample holder
- Peltier 1x1 Temperature controller (for liquid samples in adequate cuvette).

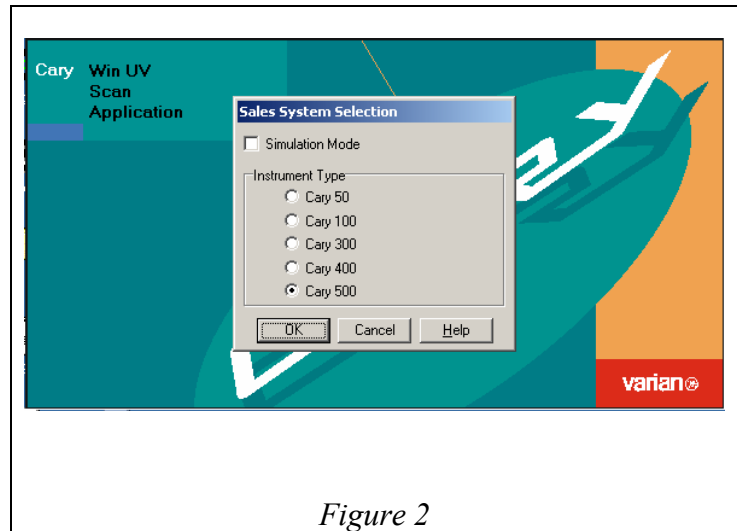
Temperature Range:

-20 to 100°C (for liquid samples in adequate cuvette).

Initialization

Initialization

- 1) Verify that there isn't any aperture plate or other accessory blocking the reference beam in the sample compartment. Close the sample compartment.
- 2) Turn the switch button on the bottom of the spectrophotometer to ON (Fig. 1).
- 3) Start the program "Scan". Select the Cary 500 system. (Fig. 2)
- 4) Wait until the instrument is initialized. When this is complete, the wavelength and transmission values should turn red. (Fig. 3)



Preparing for Data collection

Preparing for Data collection

I. Solid sample transmission

- 1) Install the aperture plate holders (Fig. 4) in the sample chamber, observing the labels on the base plate: Install the holder labeled “back” on the reference beam path, and the one labeled “front” on the sample beam path.
- 2) Select the appropriate sized aperture plate to fit the region of interest of your sample covering the central aperture.
- 3) Place one of the three identical plates in the reference beam holder (you may mount a reference sample on it, if desired).
- 4) Mount a reference sample on another of the plates, and mount your sample on the third one.

II. Liquid sample transmission

- 1) Install the cuvette holders (Fig. 5) in the sample chamber, observing the labels on the base plate: Install the holder labeled “back” on the reference beam path, and the one labeled “front” on the sample beam path.
- 2) Make sure the cuvettes you use are transparent (at least partially) over the entire range you want to measure at, and is not attacked or dissolved by the sample or buffer solutions.
- 3) Prepare a reference cuvette (e.g., empty cuvette or with buffer solution). You may want to prepare two of these and place one in the reference beam sample holder.
- 4) Prepare an identical cuvette with your sample.

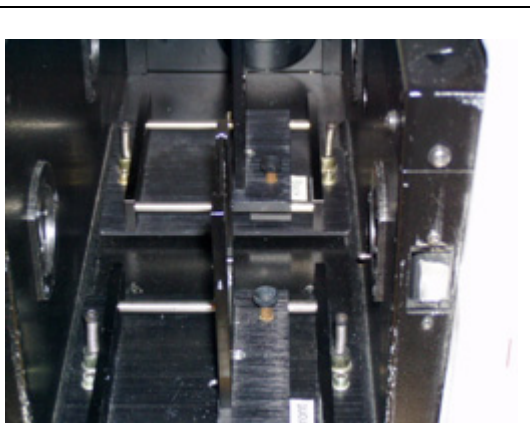


Figure 4

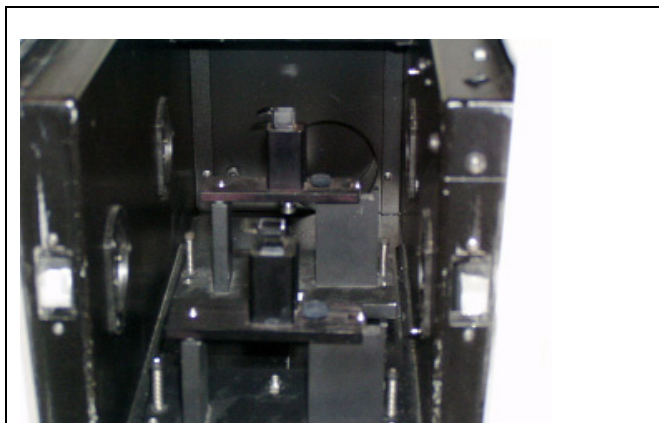


Figure 5

Preparing for Data collection

III. Specular reflectance

- 1) Install the extended sample chamber base. (Fig. 6)
- 2) Install the specular reflectance accessory (Fig. 7) as demonstrated during the training. Case you don't remember how to install it, or wasn't trained to use it, look for staff assistance. DO NOT GUESS.
- 3) Place the extended sample chamber cover. (Fig. 8)



Figure 6

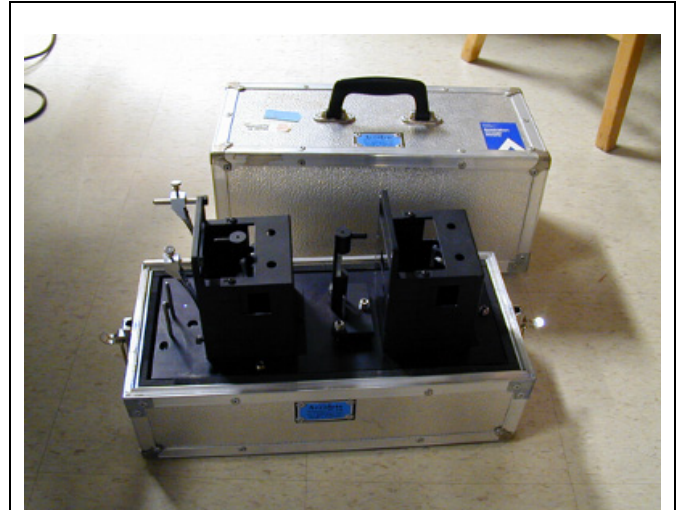


Figure 7

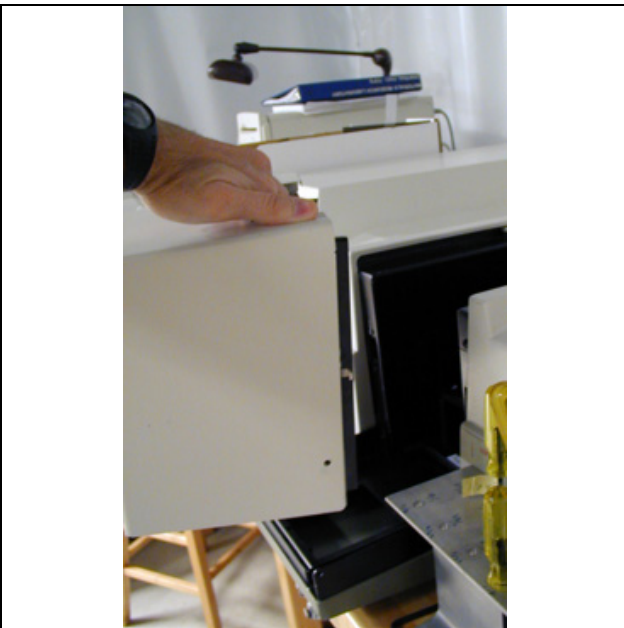


Figure 8



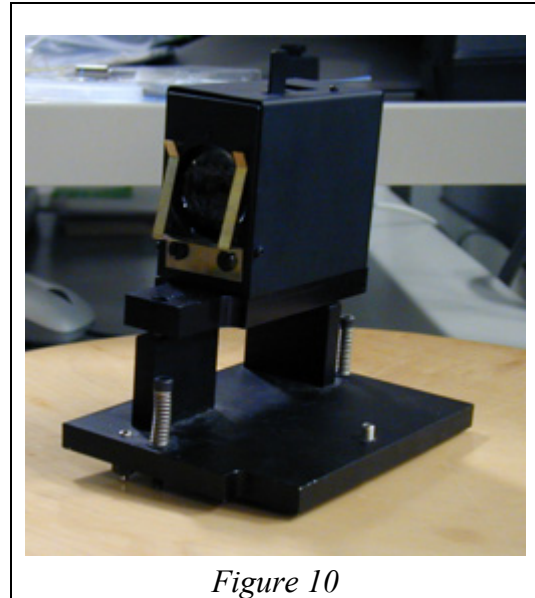
Figure 9

IV. Diffuse reflectance

- 1) Install the diffuse reflectance accessory (Fig. 9) as demonstrated during the training. Case you don't remember how to install it, or wasn't trained to use it, look for staff assistance. **DO NOT GUESS.**

V. Total fluorescence (10 x 10 mm or smaller cuvettes only).

- 1) Install the total fluorescence accessory (Fig. 10) as demonstrated during the training. Case you don't remember how to install it, or wasn't trained to use it, look for staff assistance. **DO NOT GUESS.**
- 2) If using cuvettes smaller than 10 x 10 mm, use the adapter for small cuvettes. Use only cuvettes with all four sides polished.



Data collection (transmission, absorption, reflection)

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- 1) Press the “Setup” button on the program window.
- 2) Choose the experimental parameters for your experiment. (Fig. 11)
- 3) Click on the “Baseline” tab and choose the baseline correction method you want to apply to your data. The most popular choice is the Zero/Baseline correction. In this case, a 100 % T (or R) with a blank sample (or mirror) is collected to which the data will be normalized, then a 0 % T (or R) is collected, which will be subtracted from the collected spectra.
- 4) Prepare the instrument to collect the baseline reference (100%) and click the “Baseline” button.
 - a. **For transmission**, insert the 100% transmission reference sample in the sample holder.
 - b. **For specular reflectance**, place the movable mirror of the sample beam to the “V” configuration position and leave the sample space empty.
 - c. **For diffuse reflectance**, put the 100% reflectance reference in the sample position.
 - d. Click OK and wait for the baseline to complete.

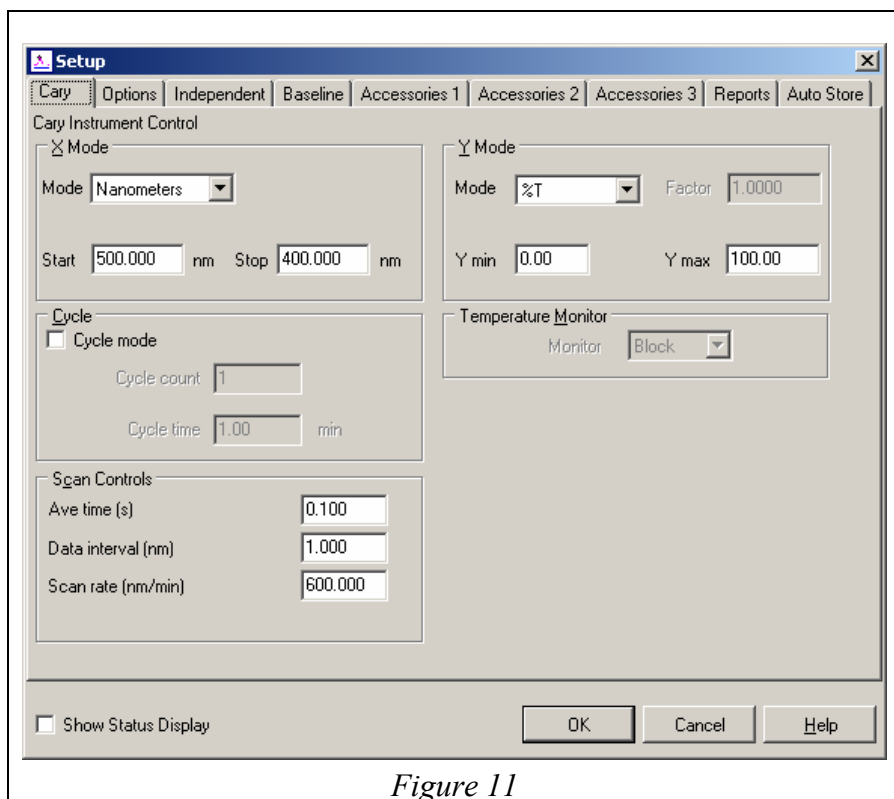


Figure 11

Data collection (transmission, absorption, reflection)

- 5) Remove the reference sample. Prepare the instrument for the zero reference baseline collection.
 - a. **For transmission**, insert the aperture-less plate or the blocked cuvette in the sample holder.
 - b. **For specular reflectance**, place the movable mirror of the sample beam to the “W” configuration position and leave the sample space empty.
 - c. **For diffuse reflectance** leave the sample space empty.
 - d. Click OK and wait until the zero reference baseline is complete.
- 6) Insert your sample in the sample chamber. Click the “Start” button to initiate collection of its spectrum.



- 7) Follow the on screen instructions. Remember to save the data to your own folder, not in a common area.
- 8) To save your data for analysis with a software other than the collection software, save your data as CSV, after you collect the last spectrum. The Scan program will save all the collected data and baselines in the memory, in a single file. If you prefer to save each spectrum in a different file, you can do so by checking the “Save only focused trace” check box. (Fig. 12)

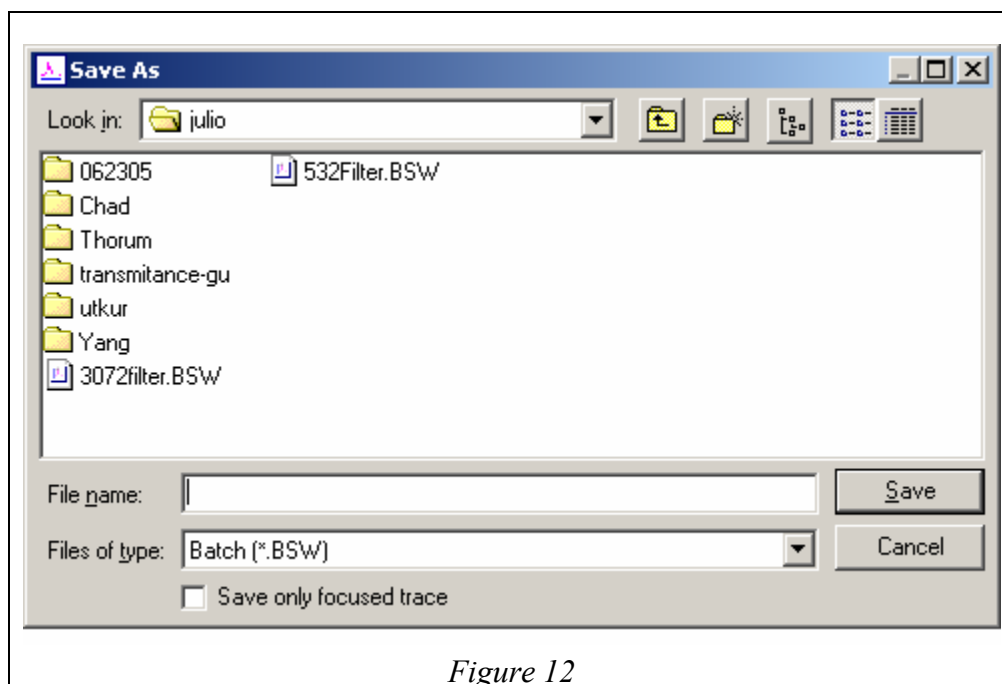


Figure 12

Shutting Down

Shutting Down

- 1) Turn Cary 5G off by turning the power switch to the OFF position.
- 2) Take any samples, aperture plates and accessories out of the sample compartment, and store in the designated place for those items. Make sure you clean all the tape or sample residues off the aperture plates before returning them to the proper storage place.
- 3) Make sure you saved all your data. Exit the Scan program.
- 4) Mark the time off on the log book.