NANOmetrics Nano 100UVIR Microspectrophotometer

I. Instrument Startup Procedure

A. Turn on the television monitor.

B. Turn on the reflectance (the knob on the beige box) or transmittance (the switch and slider on the bottom of the microscope) halogen lamp depending on your need. Turn the knob or slider to maximum intensity. The lamp should warm up for a least 30 minutes.

C. Turn on the CS-9 computer using the red POWER switch at the lower left side of the rear of the unit.

D. Turn on the computer, monitor and printer.

1. Click on Nanometrics
2. Click on Grams/386

II. Instrument Calibration

A. Prior to using the microspectrophotometer, 100% lines and holmium oxide (transmittance) or Munsell 6.25 Neutral Gray (reflectance) spectra should be run. All users should log their usage time as well.

B. Select microscope objective. Use the same magnification for both calibration/reference and sample scans.

C. Click on DATA ACQUISITION to initialize the insstrument.

D. Transmittance

1. Enter bright field (B.F.) Mode by pushing the knob above the objectives inward.
2. Set up the microscope for Koehler illumination:
   a. Place a sample on a slide onto the microscope stage and focus on it.
   b. Close the field diaphragm.
   c. Open the aperture diaphragm.
   d. Adjust the condenser until field diaphragm edges are in focus.
   e. Open the field diaphragm until it is just larger than the field of view.

3. Click on TRANSMISSION

4. Place an empty glass slide on the stage.

5. Click on PARAMETERS and enter the scan range, number of scans and scan speed. Suggested settings for calibration:

Starting wavelength:	400	
Ending wavelength	800	
Number of scan reference:	1	
Number of scan sample:		1
Scanning speed:		63 nm/sec

6. Click on SCAN BACKGROUND. During the scan, watch the intensity meter on the spectrometer head. The highest reading should be around 185 for best results. Adjust the GAIN knob on the monochrometer head and repeat scanning the reference to obtain this value.

   a. SHORTCUT!!! Since the maximum of the background spectrum occurs at 610 nm do the following: Click on PARAMETERS then OPTIMIZE. Enter 610 then o.k. The monochrometer will go to exactly 610 and hold. Adjust the GAIN until the intensity reads 185. Click CANCEL to exit the parameters screen. Click on SCAN BACKGROUND and the intensity should reach exactly 185.

   b. Once the GAIN is properly set, change the parameter settings to what will be used on your samples.

   c. SCAN BACKGROUND one last time using new settings.

7. Click on SCAN SAMPLE to obtain a 100% line.

   a. Save this spectrum in the qc-100 directory as the date (ex. 081899.spc).

   b. Save this spectrum to the backup diskette.

8. Holmium oxide is used as a reference.
   a. Place the holmium oxide on top of the bottom illuminator.

   b. Click on SCAN SAMPLE.

   c. Save this spectrum in the qc-holmi directory as hdate (ex. h081899.spc).

   d. Save this spectrum to the backup diskette.

e. The 460 nm band should be monitored for unusual deviations.

9. In the USAGE LOG note that a 100% line and transmittance reference were taken.

E. Reflectance

1. Enter dark field (D.F.) Mode by pulling the knob above the objectives outward.

2. Click on REFLECTANCE.

   a. Select COLOR, then OK.

3. Place the glass slide with the BaSO4 pellet on microscope stage and focus on the surface. You may need to decrease the intensity of the light to view the surface better. Be sure to turn it back to maximum before scanning!

4. Click on PARAMETERS and enter the scan range, number of scans and scan speed. Suggested setting to calibration:

Starting wavelength:	400	
Ending wavelength:	800	
Number of scan:		1
Number of scan sample:		1
Scanning speed:		63 nm/sec

5. Click on SCAN REFERENCE. During the scan, watch the intensity meter on the spectrometer head. The highest reading should be around 185 for best results. Adjust the GAIN knob on the monochrometer head and repeat scanning the reference to obtain this value.

   a. SHORTCUT!!! Since the maximum of the background spectrum occurs at 503 nm do the following: Click on PARAMETERS then OPTIMIZE. Enter 503 then ok. The monochrometer will go to exactly 503 and hold. Adjust the GAIN until the intensity reads 185. Click CANCEL to exit the parameters screen. Click on SCAN BACKGROUND and the intensity should reach exactly 185.

   b. Once the GAIN is properly set, change the parameter settings to what will be used on your samples.

   c. SCAN BACKGROUND one last time using new settings.

6. Click on SCAN SAMPLE to obtain a 100% line.

   a. Save this spectrum in the qc-100 directory as the date (ex. 081899.spc).

   b. Save this spectrum to the backup diskette.

7. Scan the Munsell Neutral Value 6.25 Matte Chip as a reference.

   a. Place this color chip on the microscope stage and focus on the surface.

   b. Click on SCAN SAMPLE.

   c. Save this spectrum in the qc-6.25g directory as gydate (ex. gy081899.spc).

   d. Save this spectrum to the back up diskette.

   e. In the USAGE LOG note that a 100% line and reference were taken.

III. Sample Analysis

A. For best results, it is recommended that samples be scanned in three different spots. These three spectra can be averaged to produce a final mean spectrum.

B. <u>Transmittance</u> (Bright Field Mode)

1. Place a glass slide on the microscope stage and focus on the surface.
2. Click PARAMETERS and enter settings. Suggested Settings:

Starting wavelength:	400
Ending wavelength:	800
Number of scan reference:	3

Number of scan sample: 3
Scanning speed: 63nm/sec

3. Click on SCAN BACKGROUND. During the scan, watch the intensity meter on the spectrometer head. The highest reading should be around 185 for best results. Adjust the GAIN knob on the monochrometer head and repeat scanning the reference to obtain this value.
4. Move the sample so that the portion to be analyzed is centered under the black dot.
5. Click SCAN SAMPLE
6. Click SAVE - SAVE AS to save the spectrum in your directory.

C. Reflectance (Dark Field Mode)

1. Place a glass slide with a $BaSO_4$ pellet on it on the microscope stage and focus on the surface of the $BaSO_4$ pellet.
2. Click PARAMETERS and enter settings. Suggested Settings:

Starting wavelength: 400
Ending wavelength: 800
Number of scan reference: 3
Number of scan sample: 3
Scanning speed: 63nm/sec

3. Click on SCAN BACKGROUND. During the scan, watch the intensity meter on the spectrometer head. The highest reading should be around 185 for best results. Adjust the GAIN knob on the monochrometer head and repeat scanning the reference to obtain this value.
4. Move the sample so that the portion to be analyzed is centered under the black dot.
5. Click SCAN SAMPLE
6. Click SAVE - SAVE AS to save the spectrum in your directory.

IV. Data Analysis

A. Refer to NANO metrics Operating and Maintaining Manual Chapter 3 for instructions on data analysis and processing.

V. Instrument Shutdown

A. Exit Grams/386 program.
B. Exit Windows.
C. Turn off power to all equipment.

VI. Safety Concerns

A. Halogen Lamp may be hot.
A. Mercury Lamp may be hot.

VII. Other Information

A. Refer to NANO metrics Operating and Maintaining Manual