

Suggested Procedures for Checking Performance of NIR
Spectrophotometer

- A. Photometric linearity of logarithmic photometer:
- 1a. At a fixed wavelength, adjust lamp to full intensity and set conditions for zero OD level.
 - 1b. Record zero level signal.
 - 2a. Insert neutral density screen of approximately 0.4 OD and record signal level.
 - 2b. Repeat 1b and 2a to obtain mean value of change in reading between screen in and out.
 - 3a. With screen out reduce lamp intensity to an OD level of 1.00 +/- 0.05 OD.
 - 3b. Record signal level.
 - 4a. Insert the neutral density screen and record signal.
 - 4b. Repeat 3b and 4a to obtain mean value of change with screen in and out.
 - 5a. With screen out reduce lamp intensity to an OD level of 2.00 +/- 0.05 OD.
 - 5b. Record signal level.
 - 6a. Insert the screen and record signal.
 - 6b. Repeat 5b and 6a to obtain mean value of change with screen in and out.
 - 7a. With no screen, reduce lamp intensity to an OD level of 2.50 +/- 0.05 OD.
 - 7b. Record signal level.
 - 8a. Insert screen and record signal.
 - 8b. Repeat 7b and 8a to obtain mean value of change with screen in and out.
 - 9a. With no screen, reduce lamp intensity to an OD level of 3.00 +/- 0.05 OD.

9b. Record signal level.

10a. Insert screen and record signal.

10b. Repeat 9b and 10a to obtain mean value of change.

Note: Two samples differing in Log (1/R) values of ~0.4 can be used in place of inserting the screen.

Typical results :

OD level	Δ OD
0.00	0.400 \pm 0.0005
1.00	0.399 \pm 0.001
2.00	0.398 \pm 0.001
2.50	0.397 \pm 0.002
3.00	0.395 \pm 0.003

B. Stray light: (Transmission only)

1a. Record the spectra for samples of corn oil of the following path lengths (d): 0.2, 0.4, 0.6, 0.8, 1.0, 1.2, 1.4, 1.6, 1.8, 2.0 mm. •

1b. Plot the measured Absorbance at 2.308 μm vs. sample path length. Draw a straight line through the linear data points. Draw a curved line through nonlinear region.

1c. At a sample path length giving well-defined difference between linear and nonlinear points, read the values $A(d)$ = linear data predicted, and $B(d)$ = nonlinear or measured value.

1d. Computed percent stray light:

$$\% \text{ SL} = 100 (10^{-b(d)} - 10^{-a(d)})$$

typical data:

$$\begin{aligned} A &= 3.12 \\ B &= 2.86 \\ \text{SL} &\pm 0.062\% \end{aligned}$$

2. Similar stray light test at 1.718 μm can be made using path lengths of: 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0, 5.5, 6.0, 6.5, 7.0, 7.5, 8.0 mm.

Note: The stray light test assumes the photometer is linear, and it measures any room stray light in addition to monochromator stray light.

C. Wavelength accuracy:

Record the signal obtained when tungsten source is replaced with a mercury arc lamp such as an H85A by General Electric. It is best to operate the arc lamp with direct current, but we have operated with the standard alternating current ballast.

The measurement should be made with normal instrument settings for scanning speed, response speed, and slit width to determine accuracy under standard operating procedures.

Compare recorded curve with established values for mercury lines (table and charts attached).

D. Wavelength precision or reproducibility:

1. Record the mercury lamp signal at least 10 times using standard instrument parameters.
2. Determine the measured wavelength for the 1.5295 μm mercury line within ± 0.005 nm by straight line extrapolations on each side of curve to intersection point, or use two-point ratio procedure as illustrated:



Choose wavelengths for A and B readings to give a ratio A/B equal to 1.000 to minimize intensity effects.

Determine K by shifting these wavelengths up or down one wavelength increment (I) and reading A_1 and B_1 .

$$K = \frac{I}{A_1/B_1 - A/B}$$

$$\lambda_0 = 1.5295 - K$$

E. Monochromator bandpass:

1. Record the spectrum of the mercury lamp at normal slit width setting.
2. Measure the wavelength spacing which gives a reading 0.301 OD down from the peak on either side. The 1.5295, 1.0140, or the 1.1299 μm lines are best for bandpass checks.

F. Overall noise:

Record sample vs. sample curve and determine peak-to-peak signal within a given wavelength interval.

Ceramic vs. ceramic should be compared to typical sample vs. sample. The RMS noise is a more useful term:

$$\text{RMS noise} = \sqrt{\frac{\sum_{\lambda_1}^{\lambda_2} (\log(1/R))^2}{N}}$$

where N is the number of wavelength intervals in scanning from λ_1 to λ_2