

MicroNIR

Integration Time, Reference Spectra & Signal Strength

Summary

- Typical MicroNIR reference signals have a maximum intensity of ~50,000 counts near 1600 nm.
- Use the default integration time programmed into the MicroNIR to enable temperature normalization.
- Use a measurement collar to ensure reproducible spectra.

Integration Time

VI.AVI MicroNIR spectrometers are programmed with a default integration time at the time of manufacture. This integration time, which is different for each instrument, is intended to deliver a signal strength that will provide the optimum signal-to-noise ratio in routine analysis. In nearly all situations, the MicroNIR user should use the default integration time.

The default integration time is recorded in the MicroNIR firmware and cannot be changed by users. In MicroNIR Pro versions 3.0 and above, it is displayed in the default spectrometer configuration in Developer’s Space, as shown below. For this instrument, the default integration time is 15.3 ms. This value is grayed out and cannot be changed in the default configuration. To choose a different integration time, create and save a new spectrometer configuration.

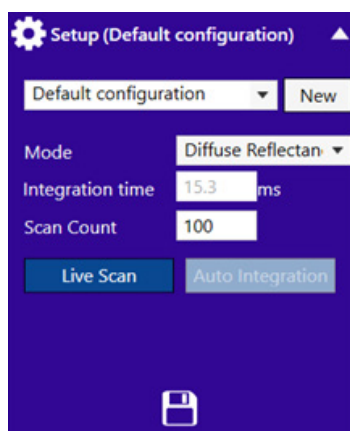


Figure 1. Default spectrometer configuration

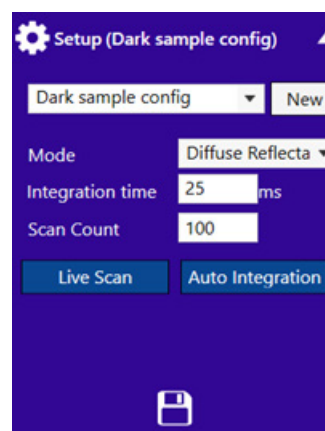


Figure 2. New spectrometer configuration

Signal strength, saturation and nonlinearity

Under normal operating conditions the MicroNIR detector behaves in a linear manner; that is, the signal recorded is linearly proportional to the photon flux collected. However, the MicroNIR uses a 16-bit analog-to-digital converter in the signal train. The maximum signal that can be recorded is hence $2^{16}-1$ or 65,535 digital counts. If the reference signal is 50,000 counts and the integration time is doubled, the signal recorded will be, not the expected 100,000 counts, but a saturated signal of 65,535 counts. Signal strengths greater than ~55,000 counts (this varies from one instrument to the next) may show some degree of nonlinearity or 'soft saturation'. The default integration time programmed into the MicroNIR has been set so that, under the acquisition conditions used in manufacturing, the maximum reference signal will be approximately 50,000 counts (the exact value used has changed slightly over the years). Using the default integration time ensures that the MicroNIR will behave in a linear manner.

Each MicroNIR has its own integration time. If the reference signal is too large (as when using an accessory like the Mini Probe), or too small (as when using the Side View Vial Holder), create and save a new spectrometer configuration with a different integration time. The Auto Integration feature in MicroNIR Pro can be used for this purpose. In an enterprise application where multiple MicroNIRs are in use VIAVI recommends using the default value for each instrument rather than a single integration time for different MicroNIRs.

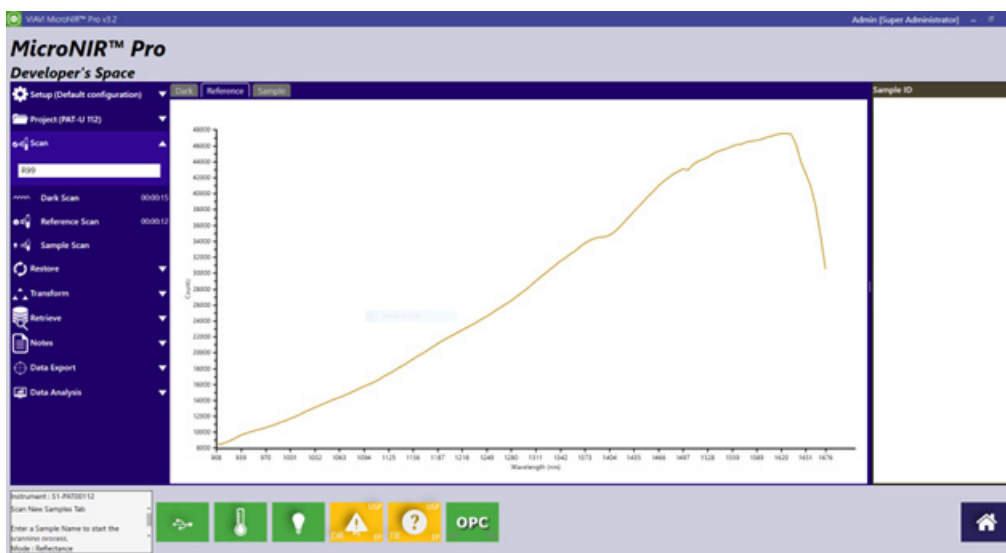


Figure 3. Typical MicroNIR reference spectrum. The maximum signal is ~47,500 counts, within the normal range.

Temperature Baseline Normalization (TBN)

The MicroNIR uses an uncooled InGaAs detector which is sensitive to the ambient temperature. MicroNIR instruments are programmed with a set of temperature correction factors that mitigates this effect. TBN correction is automatically applied to the measured absorbance (not the individual dark, reference or sample scans) such that the absorbance is unaffected by instrumental temperature changes. In MicroNIR Pro v3.0 and above an icon is displayed when an absorbance scan is recorded using TBN correction, as shown below. All MicroNIR instruments manufactured since approximately 2014 include the TBN correction in firmware except for the networked instruments the PAT-W, -Wx and -Ux. These instruments do not store TBN correction data in firmware, but in a file on the PC that is used to operate the MicroNIR. To download the TBN data file from the cloud for these instruments, click the TBN button in System Setup in MicroNIR Pro v3.2 and above (earlier versions do not support TBN correction for these instruments). Note that, as the TBN data are stored on a PC, the file download process **must be repeated** when a new PC is used.

TBN correction data are acquired during manufacturing over a range that is greater than the MicroNIR rated operating range of 0-40 °C. The MicroNIR will acquire meaningful data when used outside the rated range, but VIAVI recommends staying within it for best results.

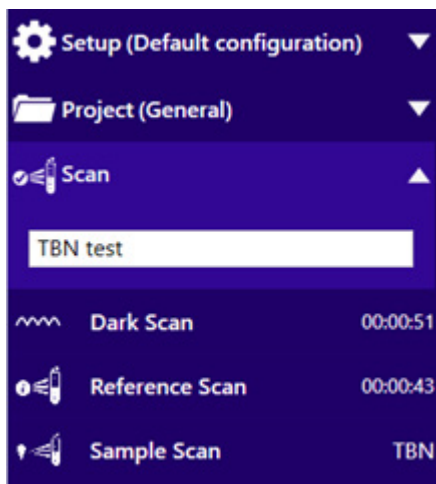


Figure 4. Temperature Baseline Normalization

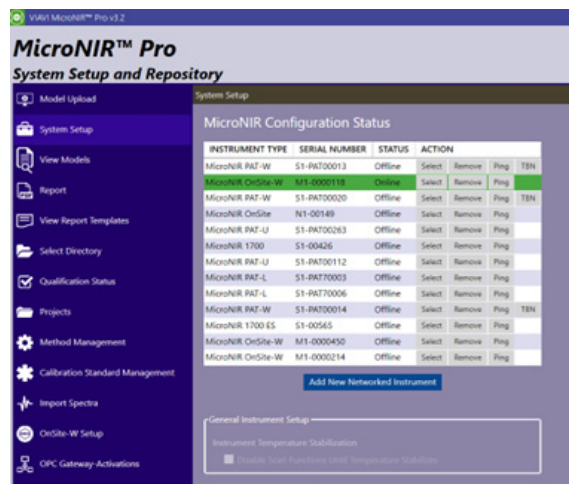


Figure 5. TBN setup for the MicroNIR PAT-W/Ux/Wx

It is important to note that the TBN correction is applied only when the default integration time for the instrument in question is used. If the default integration time is 15.3 ms and an integration time of 15.2 ms, 15.4 ms or any other value is used, TBN will not be applied. This is why VIAVI recommends using the default integration time. If a different integration time is used, VIAVI recommends that dark and reference spectra should be reacquired whenever the instrumental temperature changes by more than a few degrees Celsius. **When the default integration time is used and TBN is applied, VIAVI recommends that dark and reference spectra should be acquired daily** or whenever there is a change in acquisition conditions. MicroNIR Pro includes functionality to restore dark and reference spectra from disk in order to permit acquisition of sample spectra when acquiring reference spectra is inconvenient, as when the MicroNIR is mounted in a process.

Measurement Collars

MicroNIR spectrometers are shipped with measurement collars. A collar should always be used during spectral acquisition as it sets the optimum working distance from the MicroNIR to the sample (but remove the collar when using a MicroNIR PAT in conjunction with a separate process window). Windowed measurement collars use a sapphire window which is antireflection coated on the inner side (for the OnSite-W) or uncoated (for the PAT instruments). Window reflections contribute to the signal that the MicroNIR receives, as shown below. Figure 6 shows a lamps off dark scan with a mean signal strength of ~4950 counts (This is not dark noise but instrumental bias). Figure 7 shows a lamps on dark scan acquired with the instrument equipped with a windowed collar and held several inches from any object, i.e. an air spectrum. The signal arises from back reflections of the lamps from the MicroNIR collar and internal windows.

VIAVI recommends using a windowed collar for routine applications such as powder measurement. Use a windowless collar when working with specularly reflective samples such as smooth surfaced polymers or when performing the OQ routine in MicroNIR Pro. As the lamps on reference signal is weaker with a windowless collar, ensure that the reference and sample spectra are acquired using the same collar.

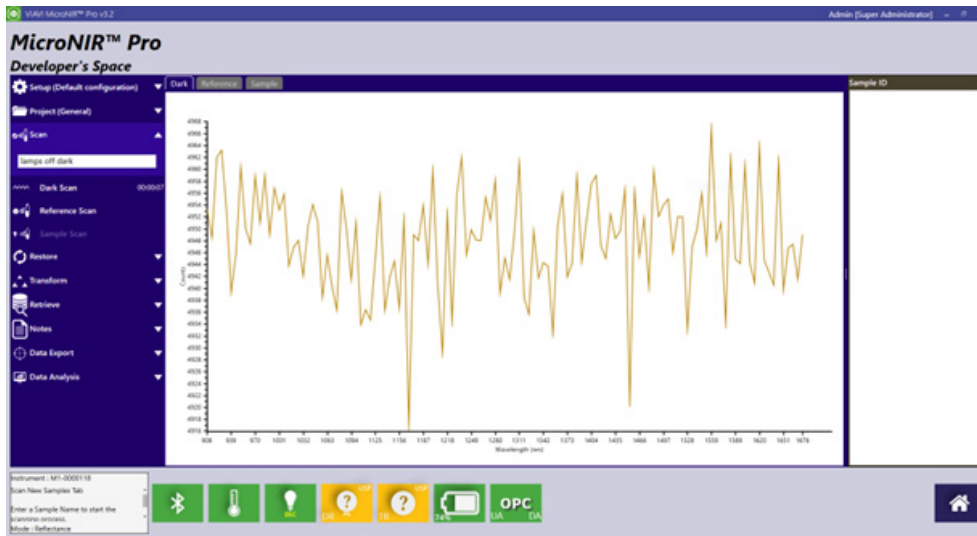


Figure 6. Typical lamps off dark scan.

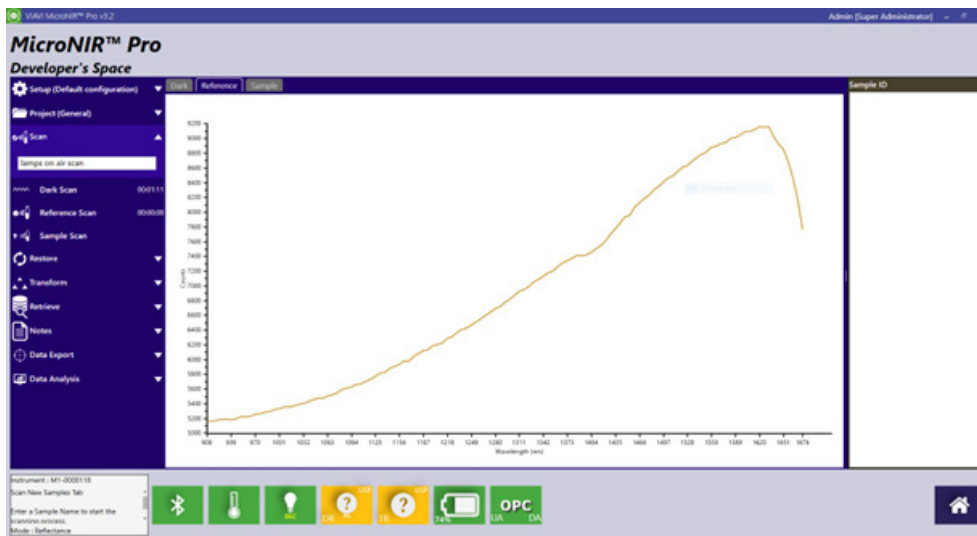


Figure 7. Typical lamps on dark scan with windowed collar.



Figure 8. Windowed OnSite-W collar (L), PAT collar (center), windowless collar (R).

Reference standards

MicroNIR spectrometers (apart from the PAT-L, which operates in transmission mode) are shipped with reference standards. The OnSite-W ships with a protected 99% reflectance (R99) standard and the other MicroNIRs ship with a bare, unprotected R99 standard made of Spectralon. The two standards are shown in Figure 9.



Figure 9. Protected (left) and unprotected (right) R99 reflectance standards

The protected standard is located behind an antireflection-coated window (the coating gives the window a pink color). As the distance between the MicroNIR and Spectralon surface is greater in the protected than in the bare standard, the signal strength is smaller in the former case. Back reflections from the window surfaces increase the signal strength vs. a windowless collar.

MicroNIR	Windowed collar	Windowless collar
OnSite-W w/ protected R99	48,000	44,000
PAT w/ unprotected R99	50,000	45,000

Table 1. Typical maximum reference signals at the default integration time.

Frequently Asked Questions

Q. Do my MicroNIR's lamps need replacement? I worry that the lamps might drift as the instrument ages.

A. The low powered quartz tungsten lamps used in the MicroNIR are expected to last well over 100,000 hours (over ten years), with negligible changes in output intensity or spectral content, as discussed in the *MicroNIR Lamp Lifetime Statement ED000217-01*. This is a key advantage of the MicroNIR over Fourier Transform or scanning benchtop NIR spectrometers that require frequent lamp replacement and recalibration. It is good practice to periodically record a reference signal obtained under standard acquisition conditions, and VIAVI recommends that all MicroNIR users do so. Typical causes of changes in reference signal include:

- The **reference standard** is soiled. If using the protected reference, clean the external window with a clean microfiber cloth and isopropyl alcohol, if necessary. Clean the unprotected reference as described below.
- A MicroNIR, measurement collar or protected reference **window** is soiled. Clean the window, if necessary, with a microfiber cloth and alcohol.
- The MicroNIR, with collar, is **not in direct contact** with the reference standard. Place the device on the standard (OnSite-W, PAT U/Ux, 1700 ES) or place the standard on the device (PAT-W/Wx).
- The measurement collar is **not fully seated**. Make sure to screw it in all the way so that it is hand tight. Ensure that the threads are clean and free of dust or debris, particularly if the MicroNIR has been used in a dusty environment such as a feed or flour mill.
- A different **integration time** was used. Ensure that you are using the default spectrometer configuration in MicroNIR Pro or that the integration time in the configuration you use has the default value. It is possible to set a different integration time in the OnSite-W application. The OnSite-W Service Tool software (versions 1.19 and below) applies a standard integration time of 10 ms, which might be more or less than the value used in your MicroNIR.

Q. My MicroNIR OnSite-W reference signal maximum is only 44,500 counts. The Quality Report that came with my instrument lists a maximum signal of 47,000 counts. Do I need to have it recalibrated?

A. No. The test setup that VIAVI uses in production is slightly different from a typical user setup. The MicroNIR will give good results with a reference signal ranging from under 40,000 to over 50,000 counts.

Q. I am analyzing dark asbestos-containing materials where the absorbance ranges from 1 to 1.5 OD units. Should I increase the integration time in order to improve the signal-to-noise ratio?

A. Possibly. If you double the integration time you will double the signal. However, you would also most likely saturate the MicroNIR detector when measuring the R99 reference. VIAVI sells 50% and 10% reflectance standards for this purpose. Use one of those to acquire the reference signal if you plan to double the integration time. Be sure to re-reference the MicroNIR periodically as the temperature changes, as the TBN correction will not be used. Increasing the number of replicates in your scans will improve the signal-to-noise ratio. Finally, consider using the Mini Probe accessory, which was designed to increase the illumination and collection efficiency of the MicroNIR with dark samples.

Q. I have a MicroNIR OnSite-W. Can I use the unprotected reference standard?

A. Yes, but be careful not to mix and match sample spectra acquired after different references.

Q. The R99 reflectance standard is visibly soiled. Should I keep using it?

A. You should clean it (instructions below) or purchase a new one from VIAVI.

Instructions for cleaning the unprotected R99 reference standard:

- The 99% diffuse reflectance panel included with the MicroNIR is an optical standard and should be handled in much the same way as other optical standards. Although the material is very durable, care should be taken to prevent contaminants such as finger oils from contacting the material's surface. It is recommended the user wear clean gloves when handling, or handle by the sides only while avoiding contamination on the surface coming into contact with the MicroNIR.
- If the material is lightly soiled, it may be air brushed with a jet of clean dry air or nitrogen. DO NOT use Freon. For heavier soil, the material can be cleaned by sanding under running water (Standards <10% should be sanded dry) with a 220–240 grit waterproof emery cloth until the surface is totally hydrophobic (water beads and runs off immediately). Blow dry with clean air or nitrogen or allow the material to air dry.



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