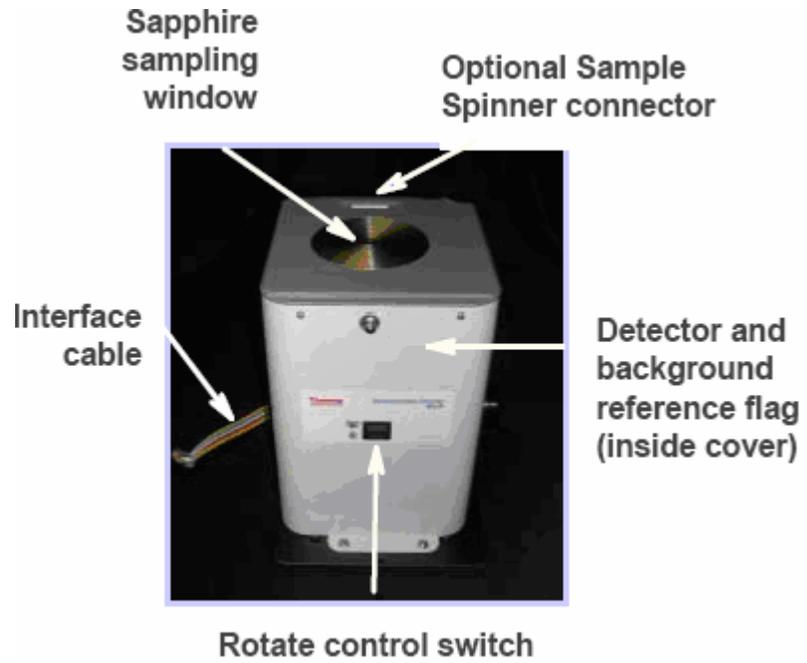


# NIR Integrating Sphere Operation

The Thermo Scientific near infrared (NIR) integrating sphere is an extremely high efficiency collector of back-scattered, NIR radiation. The embedded indium-gallium-arsenide (InGaAs) detector provides excellent stability and sensitivity over the full NIR spectral range. The spectrometer needs to be equipped with a NIR beamsplitter—either CaF<sub>2</sub> or XT-KBr are recommended—and a NIR tungsten-halogen source. OMNIC™ software should be installed and running during the installation of the integrating sphere. Because of the high sensitivity of the detector, a set of neutral density screens is provided.

The integrating sphere has an internal gold flag, used to shut off the system output for collection of the background. The flag will flip open for data collection. The flag open/close function is controlled by the sample shuttle driver in OMNIC. The shuttle driver delays data collection during a sample move, which is required when this function is used to ensure proper positioning of the sample. For the integrating sphere, this feature appears as a delay in data collection when switching between background and sample collection (or vice versa).

The unit may also include a sample spinner accessory. This is turned on or off through a rotate control switch on the front panel. Power for the spinner is drawn from the interface cable.



## Installation

Follow these steps to install the integrating sphere:

- 1. Remove the integrating sphere from the shipping box, and place the integrating sphere on a table.**

- 2. Examine the shipping box and integrating sphere for visible signs of damage.**

Report any damage to the shipping agent before attempting installation.

- 3. Ensure that the spectrometer is correctly configured for NIR data collection.**

Make sure the correct source and beamsplitter are installed and selected in OMNIC.

Purge is not critical for NIR data collection, although there may be some small peaks due to water. Typically these are tiny compared with the signal and can be ignored. Due to the sensitivity of the InGaAs detector, using a set of neutral density screens or an installed screen wheel is recommended.

- 4. If the sample compartment cover is installed on the spectrometer, remove it using the knob at the back center.**

- 5. Lift out the sample compartment baseplate, if it is installed.**

This may require removal of the four corner screws. Also, remove the purge tubes from the U-shaped saddles on both sides of the compartment, if they are installed.

The integrating sphere is mounted on a black baseplate, and there is a cable protruding from an opening in the rear. The cable connector mates to the connector in the rear of the sample compartment; do not connect the cable yet.

- 6. Turn off the spectrometer power.**

7. **Lift the integrating sphere onto the front lip of the spectrometer sample compartment, with the cable positioned to the rear of the compartment.**

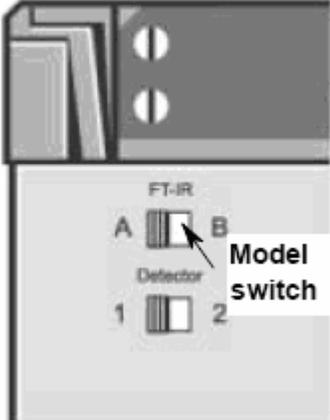
Do not place the integrating sphere in the saddles yet.

8. **Connect the cable to connector on the rear panel of the sample compartment.**

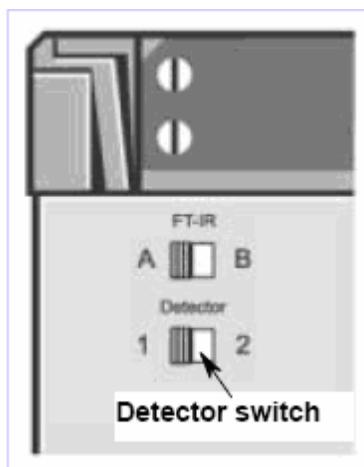
The connector is orientation- and location-specific. Make sure it is pressed in firmly.

9. **Set the FT-IR switch on the back of the integrating sphere according to the table below.**

Switch Setting	Spectrometer Model
A	Nicolet™ 8700 USB, Nicolet x700, Nicolet 380, Avatar™
B	Nicolet iS™10, Nicolet 8700 (non-USB), Nexus



10. Set the Detector switch on the back of the integrating sphere to Position 2 for all spectrometers except for the Nicolet Nexus 870 and the non-USB versions of the Nicolet 8700.



**Note** If the software does not recognize the InGaAs detector of the integrating sphere, try toggling this switch setting to the other position. ▲

11. Carefully lift the integrating sphere up, shift it back over the sample compartment, and lower it onto the saddles.

Be sure not to pull on the cable when doing this. You may need to gently rock the integrating sphere so that the baseplate sits flat, engaging the alignment pins in the sample compartment. The system is optically aligned through the cylindrical black tube, not through the base plate position, so proper seating in the saddles will ensure correct performance.

12. Wait at least 90 seconds and then turn on the spectrometer power.

## Software setup

The integrating sphere is provided with a Smart™ chip, which should read in the correct software settings for the accessory. Check the settings as explained below to ensure this has occurred.

- 1. Choose Experiment Setup from the Collect menu of OMNIC, and set Detector on the Bench tab to InGaAs.**

If InGaAs is not available in the list, try closing the Experiment Setup dialog box and then choose the command again. If that does not work, try restarting OMNIC and/or the spectrometer and set the Detector parameter again.

- 2. Make sure the correct beamsplitter and source are selected for NIR operation.**

Typically a CaF<sub>2</sub> beamsplitter and white light source are used.

- 3. Activate the sample shuttle by selecting Sample Shuttle on the Bench tab.**

The sample shuttle drivers are used to trigger the in-out position of the internal reference flag.

### **Warning**

Do not look directly into the top window. The laser emission, though very weak, emerges from this window. ▲

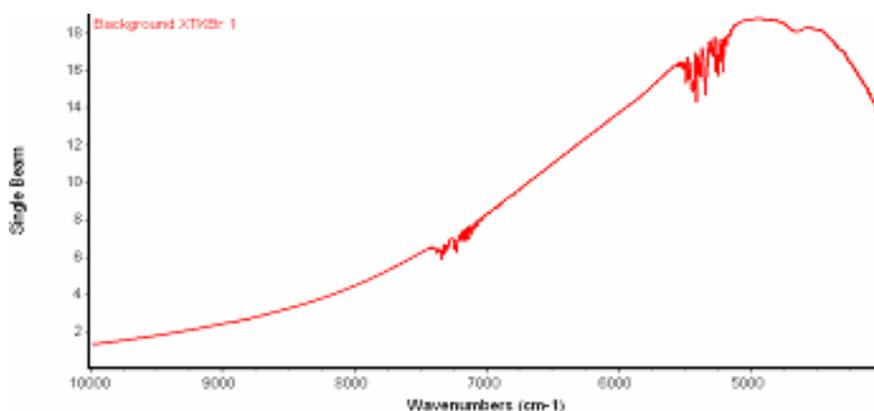
- 4. Place a piece of paper on top of the integrating sphere.**

If the beam is blocked (no light is shining on or through the paper), you are ready to select the proper gain and filters as explained in the next steps.

If the flag is open (such that you see an image of the source on the paper), you need to close it. To do this, choose OK to close the Experiment Setup dialog box (do not choose Cancel or all your new settings will be lost), and then choose Collect Background from the Collect menu (you can also click the Collect Background toolbar button or type Control+B on the keyboard). You should hear a click as the flag closes, and the bright image on the paper should disappear. Exit without collecting a background, and return to the Experiment Setup dialog box.

**5. For the first installation, set Gain on the Bench tab to Autogain to let OMNIC select the correct gain.**

The InGaAs detector interferogram amplitude depends on many factors, most of them set at the factory. In some cases, you may need to insert an attenuation screen and then balance screens and the gain settings to achieve optimal interferogram amplitude. Typical interferogram amplitude will be at least 3 V at a gain of 1 with screens and apertures set to avoid saturation. You can detect saturation by examining the shape of the single-beam spectrum. A normal, unsaturated single-beam spectrum should appear as shown below.



You can also detect saturation by collecting a single-beam spectrum from 10,000  $\text{cm}^{-1}$  to 2,000  $\text{cm}^{-1}$ . Where there is no signal the line should be flat. If it dips down and then back up in a hump, the detector is saturated. If the spectrometer has an internal filter wheel, signal attenuation can be done within OMNIC. If there is no filter wheel, the filter screens need to be inserted between the right side of the black tube and the spectrometer saddle. There is a slight springiness in the saddle, allowing it to be pushed back for screen insertion. Start with a B screen and check the amplitude. It is generally better to use a higher throughput screen and a lower gain setting.

Leave the mirror velocity set to the default value.

- 6. Once the interferogram is optimized, fix the gain to the value selected by OMNIC.**

OMNIC could search for the optimal gain each time, but this causes a data collection time delay; setting to a fixed value is faster.

- 7. On the Collect tab, set Number Of Scans to 8, Resolution to 8, and Background Handling to Collect Background Before Every Sample. Select Preview Data Collection so that you can preview the data.**

- 8. Choose OK.**

- 9. Choose Collect Sample from the Collect menu.**

You can also click the Collect Sample toolbar button or type Control+S on the keyboard.

Preliminary background collection begins. The plot should appear “humped,” with the maximum around 5000  $\text{cm}^{-1}$  and a slow decrease towards 10,000  $\text{cm}^{-1}$ .

- 10. Click the Start Collection button to collect a background.**

This should be very fast under these conditions.

- 11. When you are prompted to collect the sample spectrum, place a white powder contained in a glass container or a tablet (like an aspirin) on the sample window, and then choose OK to start preliminary sample data collection.**

You should hear a click as the flag shifts to the open position. There will be a delay of several seconds, as noted above. Shortly, the data should appear.

Click the Start Collection button to collect a sample spectrum. Comparison of this spectrum with the expected result for the sample used should verify proper operation of the unit. For additional information see the Smart Integrating Sphere tutorial available through Sampling Techniques in the OMNIC Help menu.

- 12. When the collection is finished, close the Collect Sample window.**

To shut the flag, choose Collect Background from the Collect menu and then cancel the collection.

With performance verified, we will now save the experimental parameters so the next time you install the integrating sphere, operation will be easier.

- 13. Return to the Experiment Setup dialog box and set the number of scans and resolution as required by your application.**

Note that NIR bands tend to be broad, so high resolution (better than  $2\text{ cm}^{-1}$ ) tends to gain very little. Most studies can be done at  $8\text{ cm}^{-1}$  or even  $16\text{ cm}^{-1}$  resolution without loss of information, and this also makes the scanning very fast. If you prefer, turn off Preview Data Collection and select your preferred background handling method.

- 14. Once the parameters are set, click the Save As button and save the experiment file as NIRIntSph1.exp (or any other useful name).**

Later you can set all of the operational parameters simply by opening this file. You can also make multiple experiment files, if conditions may vary. Saving the experiment puts the present state of the sample shuttle (background) and the gain settings, as well as the other experimental parameter settings, into the file. If using manual screening, you will need to ensure that the correct screens are installed (automatic screens will be recalled).

When you run spectra, collect a background. The unit is highly stable, so you can use one background for some time (depending upon application). If you will be running many samples in succession, leave the flag open and place the samples on the window. Data collection begins immediately after you choose Collect Sample, with no sample shuttle delay.



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