

iD1H Transmission Accessory User Guide

Introduction

The Thermo Scientific iD1H Transmission accessory is designed for taking near-infrared (NIR) transmission measurements of liquid samples in vials, culture tubes and cuvettes. It is an integrated accessory for the Thermo Scientific™ Nicolet™ iS™ 5N Fourier Transform Near-Infrared (FT-NIR) spectrometers. This accessory features:

- Self-adjusting sample holder for use with multiple vials and cuvettes
- Fixed 40 °C heater for measuring samples at a consistent temperature
- Polystyrene reference cuvette for running system suitability tests
- Easy installation and removal
- Pinned-in-place prealigned optics
- Automatic recognition and experiment setup
- Fixed internal aperture for optimal resolution and line shape

This document explains how to install and use the features of the iD1H transmission accessory. It also describes how to operate the accessory to analyze liquid samples in vials or cuvettes. You will also learn how to properly maintain and store the accessory when it is not being used.



Product Features

The iD1H accessory can be used to measure liquid samples in vials or cuvettes. The spring-loaded sample holder accommodates a variety of vial and cuvette sizes and shapes. The fixed 40 °C heating element with temperature sensor and LED indicator allows consistent sampling at elevated temperatures.



- **Sample holder.** Accommodates vials, culture tubes and cuvettes from 3 mm to 12 mm in diameter and at least 40 mm tall. Clear glass or quartz sampling containers are recommended. (Plastic absorbs near-infrared radiation and will affect the data. If you use plastic sample containers, be sure you know what the effects are so you can account for them in your results.)
- **Screen slot.** Used to insert an attenuation screen to control the light that reaches the detector (prevents detector saturation which affects instrument performance).

The accessory kit included with the iS5N spectrometer contains four screens (labeled A through D) for this purpose. The instrument is configured to expect the C screen for system suitability tests. Depending on your sample type, screens that block more or less of the light may be useful. For details, see “Installing Attenuation Screens” in the *Nicolet iS5N User Guide* or the Spectrometer Help Topics in the OMNIC Help menu.

- **Heater on/off.** Turns the 40 °C sample holder heater on and off (idle). An LED indicator shows the heater status. The LED meanings are explained below.

LED status	Meaning
Yellow	Idle
Green - flashing	Heating
Green - steady	At temperature

- **Cover.** Can be open or closed for sample measurements. Keep the cover closed when the accessory is not in use.
- **Purge inlet.** For attaching an accessory purge line if your spectrometer is purged with nitrogen or dry air.
- **Heater power supply port.** For attaching the power supply for the sample heater.
- **Polystyrene reference cuvette.** For running system suitability tests with the iD1H accessory. Insert the cuvette with the polystyrene material facing the right side of the accessory.

Installing and Removing the iD1H Accessory

To Install the iD1H Accessory

The iD1H accessory is easy to install into and remove from the Nicolet iS5N spectrometer. A preconfigured experiment can be selected and opened automatically. After you install the accessory, the software runs a quick performance test to verify the accessory is operating properly.

❖ To install the iD1H accessory

1. Make sure the spectrometer is turned on and is operating correctly. Refer to the *Nicolet iS5N User Guide* or the Spectrometer Help Topics in the OMNIC Help menu for assistance.
2. Grasp the iD1H accessory by the top and bottom (not the sides), gently lower it into the spectrometer sample compartment over the locator pins and press gently downward until the accessory is seated.



3. Plug the power supply into the electrical port on the back of the iD1H accessory.

The power supply supports the accessory's internal heater.



Power supply port Purge inlet

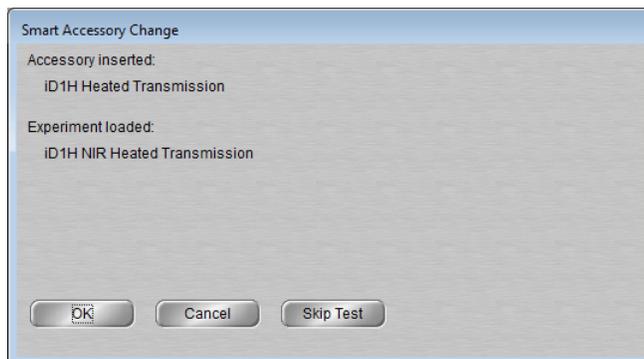
4. If your spectrometer is purged with dry air or nitrogen, attach the purge source line labeled “Accessory” to the purge inlet on the back of the accessory. See “Installing a Purge Kit” in the *Nicolet iS5 User Guide* or the Spectrometer Help Topics in the OMNIC Help menu for more information.

Opening an iD1H Experiment

After you install the iD1H accessory, the system automatically shows the experiment files that are associated with the accessory. Each experiment file contains a complete set of parameters, which have already been optimized for collecting data with the accessory. You can select and save the sampling defaults using Experiment Setup in the OMNIC software.

❖ To select an experiment

After you install the accessory, the name of its associated experiments are displayed in a message box.



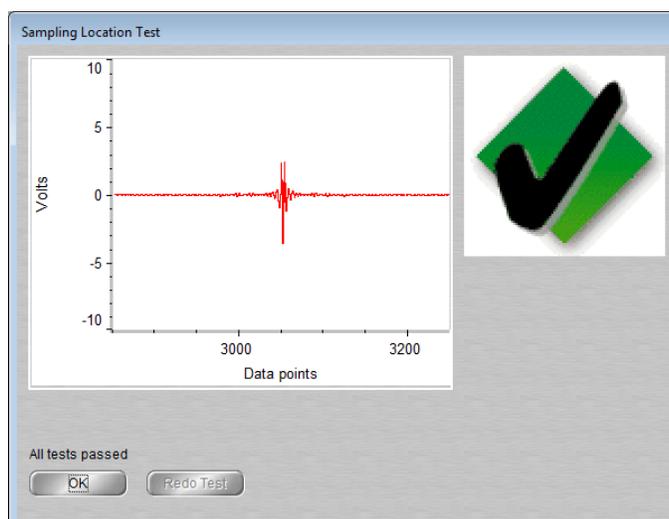
If multiple experiments are available, select an experiment and choose **OK**.

Verifying Accessory Performance

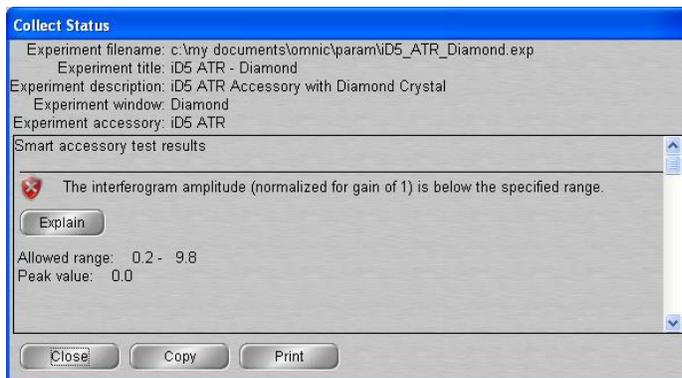
Each time you install the iD1H accessory, the system automatically runs a diagnostic test to evaluate its performance. The spectral-quality checking feature of your OMNIC software specifies the criteria used for the performance test. The performance diagnostics work in the background while the accessory is installed, ensuring high-quality spectra each time. The performance test starts automatically when you select an experiment. When the test is completed, a message box appears, displaying the results.

Note While the default accessory diagnostic test verifies the basic performance of the accessory, we recommend that you create a System Suitability test using the OMNIC System Performance Verification feature (you can use the polystyrene reference cuvette provided with the iD1H accessory if desired). The System Suitability test provides a more thorough evaluation of the accessory's performance and allows you to track its performance over time. For more information, view the System Performance Verification topic in the OMNIC Help Topics from the Help menu.

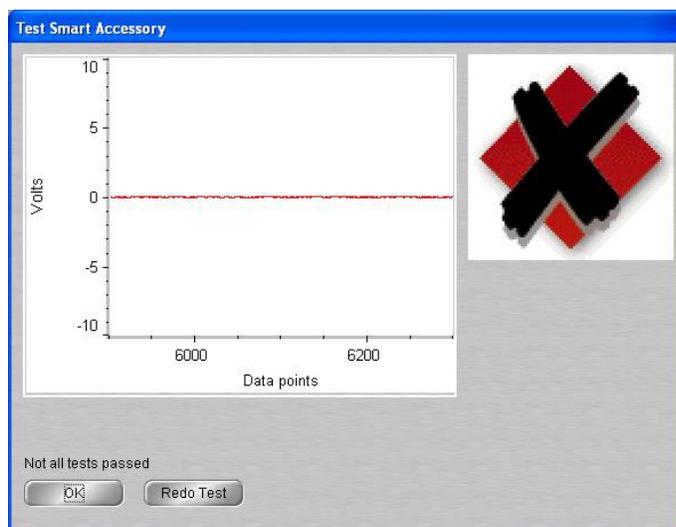
- **Performance test passed.** Look for the check mark in the green box (see image below), indicating that the system passed the performance test and is ready to collect data. Choose **OK** to continue.



- **Performance test failed.** If the accessory is set up incorrectly or the system does not meet the performance criteria, a message appears indicating there is a problem (see the example below).



Choose **Explain** for instructions on how to verify and fix the problem. When finished, choose **Close**. The Test Accessory screen indicates that a problem occurred with the performance test and allows you to restart the test. Make sure there is no sample cuvette in the sample holder, then choose **Redo Test** to rerun the performance test.



If the test still fails, see [Troubleshooting](#) for more information.

Removing and Storing the iD1H Accessory

When you are finished using the iD1H accessory, you can easily remove it from the spectrometer. When the accessory is not in use, store it in a dust-free environment such as a cabinet or box.

❖ To remove the accessory

1. Remove any sample container from the accessory's sample holder.
2. Grasp the accessory by the top and bottom (not the sides) and gently pull up to release it.



The “Accessory removed” message box is displayed.

3. Install another accessory if desired.

Background Spectrum

The background is an instrument reference spectrum which accounts for the unique optics of the iD1H accessory and the spectrometer. A background spectrum is needed to process the sample data into an infrared spectrum. Each sample spectrum is ratioed against an instrument background so the final spectrum is free of these features. The quality of the sample spectra you collect will be enhanced if the sample spectra are processed with an appropriate background. Here are some tips on collecting representative backgrounds:

- Other than Gain, Final Format, Corrections, and Number Of Scans, the settings used for the background and sample measurements should be exactly the same. The settings for Resolution and Optical Velocity must be the same.
- If you selected the iD1H accessory default experiment, all of the data collection parameters have been properly set for collecting background and sample data. If you are performing an alternate experiment, use Experiment Setup in the OMNIC Collect menu to display the Experiment Setup dialog box. Then check the settings on each of the tabs to make sure they are set to appropriate values for your experiments.

Measuring Samples by Transmission

Collecting a Transmission Background Spectrum

Follow these steps to measure transmission samples using the iD1H accessory.

The background data used to process each sample measurement to an infrared spectrum must be measured under the same conditions as the sample, but without the sample in place.

Use one of the following techniques to measure the background:

- Measure the background with the same vial or cuvette used for the sample measurements. (The software automatically removes any absorbance due to the empty container from the sample measurements.)
- Measure the background with an empty beam path and then measure the sample container (empty or with a reference material) as a sample reference or “blank.” If the blank measurement has unwanted absorbance, you can use the Subtract features in OMNIC software to remove those features from the sample spectra.

If you use the same type of sample containers and instrument settings to analyze all of your samples, you can use the same background to process multiple samples. However, we recommend collecting a new background at least every two hours.

If any condition described below is true, collect a new background immediately.

- You changed a component in your spectrometer or sampling accessory
- You changed one of the Collect, Bench, or Advanced settings in the selected experiment (except Gain, Final Format, Number Of Scans, or Correction)
- You see a change in the amount of water or carbon dioxide bands in the infrared spectra of your samples
- You see an unexpected change in the spectral baseline
- The quality of your spectral data is reduced (more noise or spurious peaks in the spectrum)

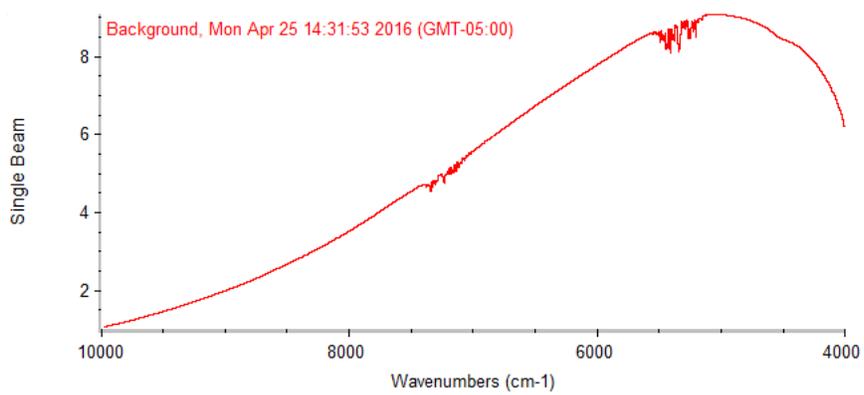
Collecting a Transmission Sample Spectrum

❖ To collect a transmission background spectrum

1. Prepare the accessory to collect the background spectrum:

2. Choose **Collect** (menu) > **Collect Background**, or click the **Collect Background** button  on the toolbar, if it is displayed.

The new background spectrum is displayed in the Collect Background window. The following images shows a typical transmission background spectrum.



The background remains in memory and is selected as the current background. It will be used to process all of the sample spectra you collect until you replace it by collecting another background.

Once the sample vial, cuvette or culture tube is positioned in the sample holder, you are ready to start collecting the sample data. The sample measurement shows how the near-infrared energy is absorbed by the sample.



CAUTION To avoid personal injury or damage to the iD1H accessory, always follow standard laboratory safety practices when preparing liquid samples. Do not heat any volatile liquid to its flash point, make sure the sample containers are capped, and always wear protective goggles and clothing.

❖ To collect a transmission sample spectrum

1. If the sample requires heating, press the Heater on/off button on the iD1H accessory to turn on the heater and wait until the green heater LED stops flashing.

NOTICE To avoid breaking your cuvettes, culture tubes, or vials and possibly injuring yourself or lodging them in the accessory, use only a moderate amount of force when inserting or seating samples into the sample holder.

2. When the green LED is on steady, insert the sample vial, cuvette or culture tube into the iD1H sample holder.

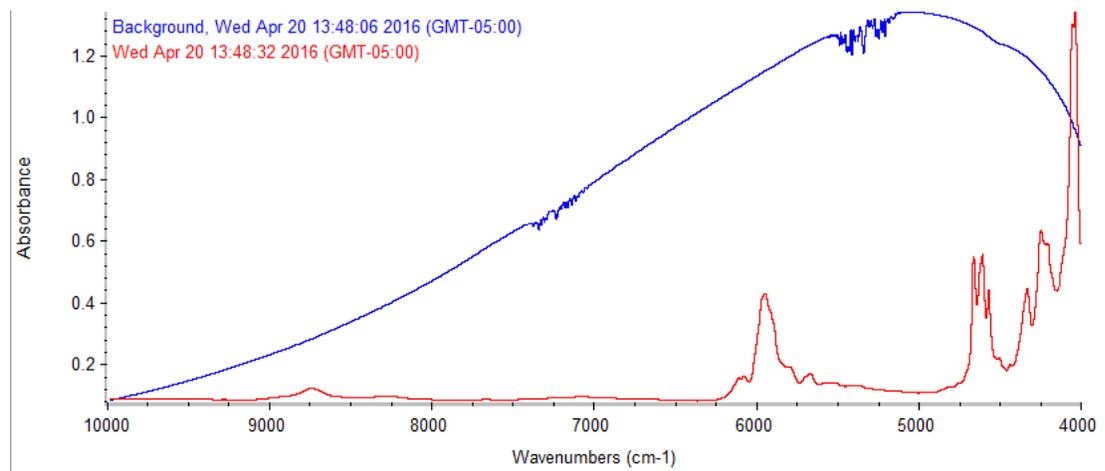
3. If the sample was preheated, depending on the size of the sample container and the initial temperature of the sample, wait 30 seconds or so for the sample to reach the target temperature. (Experiment to determine the exact wait time; for quicker heat times, store samples in a block heater preheated to the target sampling temperature.)

4. When the sample is at the desired temperature, choose **Collect** (menu) > **Collect Sample**, or click the **Collect Sample** button  on the toolbar, if it is displayed.

Follow the on-screen instructions to collect the sample spectrum. The instructions depend on how Background Handling and other options are set in OMNIC software and in the selected experiment.

The software collects a few scans and then calculates and displays a spectrum. The spectrum is updated as new data is collected.

When the measurement is completed, the final spectrum is displayed. The following example shows a transmission spectrum of a polystyrene film.



Infrared spectra collected using a near-IR (white light) source, calcium fluoride (CaF_2) beamsplitter and indium gallium arsenide (InGaAs) detector are typically displayed in the range between 10,000 and 4,000 cm^{-1} as shown above. The locations and intensities of the spectral bands will be unique for a particular material.

Trouble Shooting

The iD1H accessory has been aligned and tested in the factory using a Nicolet iS5N spectrometer to ensure that it performs to specifications. When it is new, it should be ready to run without the need for adjustment. If you experience an accessory performance test failure, first replace the iD1H accessory with the iD1 transmission accessory and run a Performance Verification (PV) test to ensure the spectrometer is working correctly. See “Running Performance Verification” in the *Nicolet iS5N User Guide* or the Spectrometer Help Topics in the OMNIC Help menu for instructions.

If the spectrometer is performing as expected, the problem is likely to be with the iD1H accessory or an inappropriate experiment setting. If the accessory fails the automatic performance test (immediately after install), follow these steps to resolve the problem:

1. Ensure that the accessory is fully seated on the sample compartment pins.
2. Check that the C screen is installed in the slot furthest to the right inside the accessory (an attenuation screen is needed for all background, reference and sample measurements).
3. Make sure the light beam is not blocked by a sample holder or sample.
4. In Windows™ Explorer, open C:/My Documents/Omnic/param/factory/param, copy the file “iD1H Heated Transmission.exp” and paste it into this path:

C:/My Documents/Omnic/param

This overwrites the experiment used for the automatic performance verification test with the factory settings for that accessory.

5. Remove and reinstall the accessory. If the automatic performance verification test fails again, contact us.

Problems with Background Spectra

If a background spectrum is atypical from previously-collected backgrounds, the reason may be related to one or more of the following requirements:

- **Use attenuation screens consistently.** If you use attenuation screens, use the same screen to measure all of the backgrounds in an experiment.
- **Use references consistently.** Don't include a sample reference in some background measurements in an experiment and leave it out of others. If you do, you will see discrepancies in your background spectra and results.
- **Make sure the sample reference is valid.** If you use a sample reference and the reference material becomes contaminated or damaged, the backgrounds that use that reference may change. When this happens, replace the reference but test the replacement to make sure it is the same as the original.
- **System Suitability reference.** If you are running a System Suitability test with the iD1H, make sure the stored background reference is current. Choose **View > Configure System Status > System Suitability > Configure** and look at the Stored Background group. If there is no stored System Suitability background reference or if the stored background is two weeks or older, collect a new background reference using the iD1H accessory and the C screen (no sample or reference sample).

Problems with Sample Spectra

These factors may affect the quality of your spectral data:

- **Use sample holders consistently.** Variations in cuvettes, culture tubes and vials can affect your spectra. To quantify the magnitude of these effects, perform repeat collections using different cuvettes, culture tubes, or vials.
- **Use flat-sided cuvettes for high accuracy measurements.** The curvature of culture tubes and vials can cause a lensing effect that interferes with high accuracy measurements. Flat-sided cuvettes do not suffer from this effect, so when taking measurements that require high accuracy, use flat-sided cuvettes instead of vials or culture tubes.
- **Use attenuation screens consistently.** Using an attenuation screen for the background collection and not for the sample collection (or vice versa) will cause variations in your results. Always use screens consistently.
- **Use consistent temperature.** If you are analyzing a heated sample, make sure all measurements are done at the same temperature. Inconsistencies in temperature can cause significant variations in peak intensities and shifts in the X-axis.

Maintaining the Accessory

Information about installing and replacing hardware is available in the *Nicolet iS5N User Guide* or the Spectrometer Help Topics in the OMNIC Help menu. All other maintenance, troubleshooting or repair must be performed by one of our trained and certified service engineers. If you need to send the instrument or an accessory to us for repair, call or e-mail us first for any shipping requirements or other instructions.

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