Nicolet Summit X FTRN Quick Start Guide

This guide demonstrates how to set up the Thermo Scientific™ Affirma™The Flow-Through Transmission Lubricant Analysis System for the Nicolet™ Summit™ X Fourier transform infrared (FT-IR) spectrometer and perform a basic analysis.

The system consists of a Flow-Through Transmission (FTRN) accessory and baseplate that fit in the spectrometer sample compartment.

Our OMNIC Paradigm software and customized workflows step you through each sample analysis.



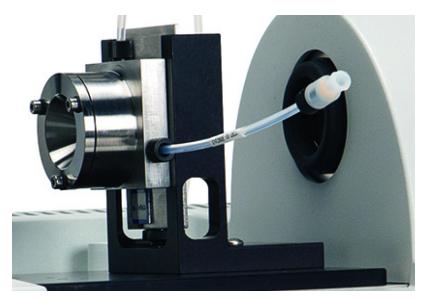


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Unpack the FTRN Accessory

To prevent condensation, which can damage internal optics, leave the FTRN accessory and components in their plastic shipping bags for 4 hours after delivery to allow them to slowly adjust to room temperature. Save the packing materials for storing or transporting the accessory.

- To unpack the FTRN accessory
- 1. Remove all components from their shipping boxes.
- 2. Wait 4 hours and then remove any components from their plastic shipping bags.
- 3. Verify the order is complete (compare against packing list).

Prepare the Nicolet Summit X Spectrometer

Before you install the FTRN accessory, follow these steps to prepare the Nicolet Summit X spectrometer.

To prepare the spectrometer

- Set up and install the Nicolet Summit X spectrometer and its associated computer according to the provided instructions. Skip this step if you are using a Nicolet Summit X spectrometer that is already installed.
- 2. Remove any existing accessory from the spectrometer.
 - Grasp the front and back of the accessory and lift it straight up and off the spectrometer.
- 3. If the spectrometer is not powered on, turn it on by pressing the power button on the back panel.

Note If you powered on the spectrometer in step 3 above, wait 4 to 6 hours for the spectrometer to warm up before acquiring data with the Affirma Lubricant Analysis System.

Install the FTRN Accessory

The Lubricant Analysis System includes a FTRN accessory and its custom baseplate designed to fit in the sample compartment of a Nicolet Summit X spectrometer. The baseplate fits over two alignment pins in the spectrometer sample compartment and is held in place by strong magnets.

To install the FTRN accessory

- Install the iD Base Adapter baseplate with the attached slide-mount sample holder in the spectrometer sample compartment.
 - Once the two alignment holes in the baseplate line up with the alignment pins in the spectrometer sample compartment, the baseplate seats itself.
- 2. Slide the flow-through cell into the slide-mount sample holder.

The cell does not ship with tubing attached as shown here. We explain how to attach tubing later in this document.

Figure 1-1: Flow-through cell with tubing



Note To remove the baseplate (with or without the FTRN accessory installed), grasp the slide-mount sample holder and lift the sample holder and baseplate straight up and off the spectrometer. Store the accessory and baseplate in a clean, dust-free environment such as its shipping box or a cabinet.

Install the software and Affirma workflow package

Follow the steps below to install the Affirma workflow packages.

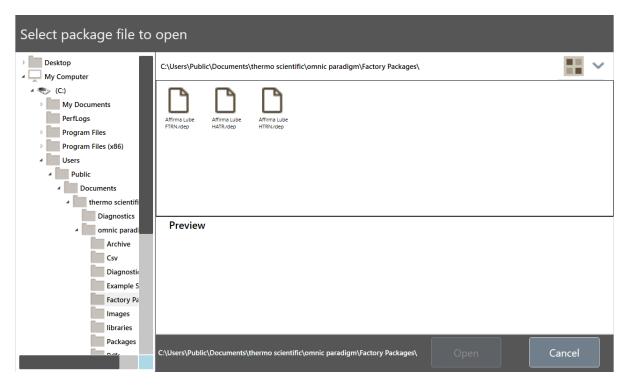
- To install the Affirma workflow package
- 1. Run the **Setup.exe** file to install the corresponding Affirma package for your accessory, then reboot your computer. Each accessory has its own file.
- 2. Start OMINC Paradigm software.
- 3. Connect to your instrument. To do so, go to Configure > Connectivity > Nicolet Summit.
- 4. Go to **Configure > View > Operator**. This will put the software in Operator mode.
- 5. In the upper left-hand corner, select the triple bar button, then choose **Administrator** and enter the password.

- 6. Select **Open** in the upper right-hand side of the screen and select the following path:
 - C:\Users\Public\ Documents\Thermo Scientific\OMNIC Paradigm\Factory Packages

Figure 1-1: Open folder icon in OMNIC Paradigm software Operator mode



Figure 1-2: OMNIC Paradigm software Operator mode Factory Packages location



7. Select the .rdep file for your accessory and select **Open**.

Note The .rdep file will only appear if you have followed these steps in order and installed the appropriate **Setup.exe** file.

8. In the upper left-hand corner, select the triple bar button, then select **Home** to exit Operator mode.

The Affirma workflows for your accessory are now available in Operator mode.

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Figure 1-3: OMNIC Paradigm software home screen with Affirma workflows

Perform a background measurement

The system requires a background spectrum for making measurements with the Affirma The Flow-Through Transmission Lubricant Analysis System. The background should be updated after 2 hours of continuous use.

To perform a background measurement

- 1. Remove the flow-through cell from the spectrometer sample compartment. (You can leave the baseplate and sample holder installed.)
- 2. In OMNIC Paradigm software Operator mode, select the **FTRN Setup** category and then select the **Background FTRN** workflow.
- 3. Follow the instructions in the workflow to complete the background measurement.

When the background measurement is completed, follow the instructions in the next section to run the calibration check.

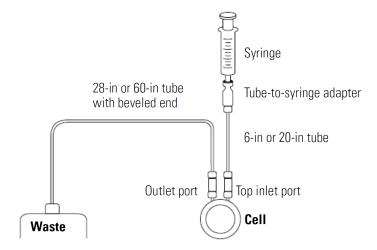
Prepare the flow-through cell for sampling

This section explains how to connect the flow-through transmission cell for manual operation using a syringe to fill the cell.

Materials required:

- · Small volume of heptane or isooctane
- Clean 10 ml Luer-Lok syringe (latex and silicone-oil free)
- · Powder-free nitrile gloves
- · Safety glasses

Figure 1-1: Plumbing for flow-through manual operation



The stainless steel cell has an internal volume of about 0.2 ml and is pressure rated to 100 psi.

The following table gives the labels on the items shown in the preceding illustration. Tubes labeled "Long" can be used in place of the regular tube if greater length is desired.

Description ¹	Label
6-inch tube	Syringe to cell
20-inch tube	Syringe to cell (long)

¹All tubing is 1/8 in OD; 1/6 in ID

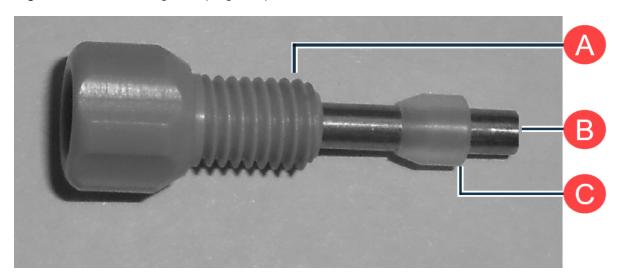
Description ¹	Label	
28-inch tube, beveled end	Cell to waste	
60-inch tube, beveled end	Cell to waste (long)	
Luer-Lok adapter	Fittings	
10 ml Luer-Lok™ Syringes (latex and silicone-oil free)	10 ml BD™ Luer-Lok™ syringe ²	

♦ To connect the cell tubing for manual operation

- 1. Connect one end of the 6-in (or 20-in) tubing to the desired cell inlet port (top or side). Tighten the connector finger tight.
- 2. Insert the sealed plug (provided) into the unused inlet port and tighten it finger tight.

The plug has three components that fit together to provide a leak-proof seal. Make sure the flat end of the pin and the flat end of the ferrule face out as shown below.

Figure 1-2: Flow-through cell plug components



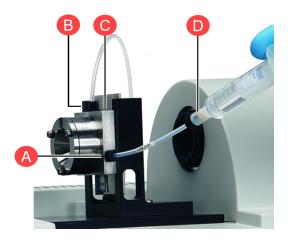
¹All tubing is 1/8 in OD; 1/6 in ID

²BD is either a trademark or a registered trademark of Becton, Dickonson and Company in the United States and/or other countries.

Α	Body
В	Pin (flat end faces out)
С	Ferrule (flat end faces out)

3. Screw the wide end of a Luer-Lok tube-to-syringe (white) adapter to the other end of the inlet tubing and tighten the adapter finger-tight. The narrow end of the adapter fits the 10 ml Luer-Lok syringes.

Figure 1-3: FTRN accessory ports



Α	Slide inlet port
В	Cell outlet port
С	Top inlet port
D	Tube-to-syringe adapter

- 4. Screw the adapter end of the 28-in (or 60-in) tubing to the cell outlet port and tighten it finger tight.
- 5. Route the free (beveled) end of the outlet tubing to your waste receptacle.

6. Fill a clean 10 ml Luer-Lok syringe with about 2 ml of heptane or isooctane and use it to leak test the cell plumbing.

CAUTION



Splash hazard. Avoid applying excessive pressure to the flow-through cell (via the syringe). Sudden leaks can cause a splash hazard for skin and eyes.

Screw the end of the syringe into the Luer-Lok adapter on the flow cell inlet tubing and slowly inject enough liquid to fill the cell. Continue injecting until the liquid starts to flow into the outlet tubing.

Tighten any connections that are leaking fluid. All connections should be finger tight.

Note If the plug leaks, refer to the previous section for a diagram of how the plug components fit together.

7. If desired, inject a syringe full of air through the cell to remove the heptane.

Run the Affirma calibration check

After you complete the background measurement and prepare the flow-through cell for sampling, run the calibration check to verify sample pathlength.

Materials required:

- FT-IR Squalane Liquid Calibration Kit (includes high purity squalane and dropper bottle)
- Clean, non-abrasive laboratory wipes (unscented and lotion-free; paper napkins work well)
- Clean 10 ml Luer-Lok syringe (latex and silicone-oil free)
- · Powder-free nitrile gloves
- · Safety glasses

CAUTION



Avoid chemical exposure. Squalane is a chemical irritant. When handling squalane:

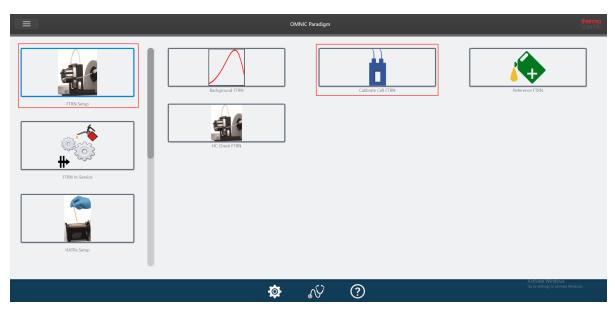
- · avoid contact with skin and eyes
- · avoid inhaling vapor or mist
- · wear gloves that provide adequate protection

Note Use only high grade (>95%) squalane. Do not sample squalane directly from its original container. Pour it into another clean, labeled container for routine use.

To run the calibration check

- 1. Make sure the FTRN accessory is installed in the spectrometer sample compartment and ready for sampling, and make sure the flow-through cell is clean.
- 2. Make sure the appropriate Background workflow has been performed. See <u>Perform a background measurement</u>. If a message indicates that a new background measurement is needed, follow the instructions in that section and restart the calibration workflow.
- 3. In OMNIC Paradigm software Operator mode, select the **FTRN Setup** category and then select the **Calibrate Cell FTRN** workflow.

Figure 1-1: Calibrate Cell FTRN workflow



4. Follow the on-screen prompts.

CAUTION

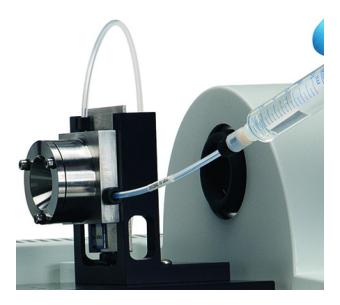


Splash hazard. Avoid applying excessive pressure to the flow-through cell (via the syringe). Sudden leaks can cause a skin and eyes splash hazard.

5. When the workflow asks you to load squalane, draw 1-2 ml of high grade squalane into a clean syringe, screw the end of the syringe into the adapter on the flow cell inlet tubing and slowly inject enough of the squalane to fill the cell (0.3-0.4 ml).

Continue injecting squalane until it starts to flow into the outlet tubing. If bubbles appear in the outlet tubing, continue injecting until no more bubbles are expelled.

Figure 1-2: FTRN accessory squalane injection



6. Follow the on-screen prompts to start the measurement.

Do not disconnect the syringe until all measurements are completed.

When the measurements are completed, the report is displayed.

7. Clean the flow-through cell and click to complete the workflow. See <u>Clean the flow-through</u> <u>cell</u> for instructions.

Note If one or more calibration test results are outside the specified limit(s), rerun the calibration test. If the system fails again, contact us.

The system is now ready for you to perform a sample analysis. Before you do that, review the remaining sections in this document which describe the software, explain the safety and operating precautions, and provide tips for running samples as well as cleaning and maintaining the system.

About OMNIC Paradigm software

Use OMNIC Paradigm software Operator mode to operate the Lubricant Analysis System. The main window provides features for installing and running customized workflows to operate the instrument and instrument diagnostics.

There are two categories of workflows in this package:

- FTRN Setup workflows
- FTRN In-Service Lubricant workflows

FTRN setup workflows

- Run Background. Performs a background measurement with the instrument (remove the cell from the sample holder). The background should be measured every 2 hours during continuous use.
- Calibrate Cell. Measures known material (squalane) to determine sample pathlength.
 Calculates pathlength and offset values used in sample measurements.

Note To ensure accurate analysis results, we recommend that you run the Calibrate Cell workflow at least once each week of instrument use.

- Collect Reference. Measures a reference oil sample. Uses the stored reference for sample measurements taken with a "differential" or "subtract" sample workflow.
- Clean Check. Performs a sample measurement through the flow cell when it is filled with cleaning solvent to determine if the flow cell has been thoroughly cleaned.

FTRN in-service lubricant workflows

- ASTM E2412 Direct Petroleum. Intended for petroleum-based sample oils to be measured using the ASTM E2412 standard practice.
- ASTM E2412 Direct Hydraulic. Intended for hydraulic sample oils to be measured using the ASTM E2412 standard practice.
- ASTM E2412 Direct Synthetic. Intended for synthetic sample oils to be measured using the ASTM E2412 standard practice.
- ASTM E2412 Subtract Petroleum. Intended for petroleum-based sample oils that have a
 matching pure reference oil, where the reference measurement is subtracted from the sample
 measurement. Use the Collect Reference workflow to measure and archive the reference oil.
- ASTM E2412 Weight Percent Soot. Calculates soot offset value.
- ASTM D7418 Direct. Intended for any sample oil to be measured using the ASTM D7418 standard practice.
- ASTM D7418 Differential. Intended for any sample oil that has a matching pure reference oil, where the sample measurement is divided by the reference measurement. Use the Collect Reference workflow to measure and archive the reference oil.

Symbol	Meaning
#	Categories for the FTRN (Flow-through Transmission) lubricant analysis system.
1	Workflows that measure sample oil content directly (without a reference oil). Direct sample workflows can be based on the ASTM E2412 or D7418 method.
Δ	Workflows that use a pure reference oil measurement to determine sample oil content.

Safety and operating precautions

WARNING



Prevent fire and explosion. If using volatile solvents, provide an active venting system that is free of spark and other ignition sources and prevents flammable vapors from collecting in the atmosphere surrounding the instrument.

To prolong the life of your system, follow these precautions:

- Protect the flow-through cell from thermal and mechanical shock
- Avoid applying excessive pressure to the flow-through cell (via the syringe). Cell is pressure rated to 100 psi.
- Do not leave solvent in the sample cell for longer than 2 minutes
- Do not attempt to measure materials that are strong acids (pH less than 4), strong bases (pH greater than 11) or strong oxidizing agents
- Do not use chlorinated solvents or materials that may produce HCl or HF vapors with or near the system
- · Do not block the vents at the back of the spectrometer

NOTICE

Read the safety guide that came with the spectrometer before operating the spectrometer with the Affirma Lubricant Analysis System installed.

Sampling tips

For optimal sampling with the Affirma Lubricant Analysis System, follow these guidelines:

A background measurement is required at predefined intervals during continuous use. The
workflows display a message when a background is needed. Use the Run Background
workflow in the FTRN Setup category to acquire a spectrum. The background is measured
with nothing in the sample beam (slide the flow-through cell out of the sample holder). The
time between background collections should not exceed 2 hours.

- This Affirma Lubricant Analysis System can be used to measure aqueous and organic liquids and light oils such as transmission and hydraulic engine oils. (Not intended for viscous materials such as gels, greases and heavy gear oils.)
- Use one of the sample workflows in the Affirma FTRN In-Service Lubricant group to run a sample analysis.

Note Sample workflows based on a differential (or subtraction) require a reference spectrum. Use the Collect Reference workflow in the FTRN Setup category to acquire a spectrum from your reference material.

To inject a sample for measurement, follow these steps:

CAUTION



Splash hazard. Avoid applying excessive pressure to the flow-through cell (via the syringe). Sudden leaks can cause a skin and eyes splash hazard.

Draw 1 to 2 ml of sample into a clean 10 ml Luer-Lok syringe. Screw the end of the syringe into the adapter on the flow cell inlet tubing and slowly inject enough sample to fill the cell (0.3 to 0.4 ml). Continue injecting sample until it starts to flow into the outlet tubing. If bubbles appear in the outlet tubing, continue injecting sample until no more bubbles are expelled.

Start the measurement. Do not disconnect the syringe until the measurement has completed.

 Always clean the cell immediately after you finish the analysis. See <u>Clean the flow-through</u> cell for details.

Clean the flow-through cell

Make sure the flow-through transmission cell is clean before and after taking sample measurements with the Affirma Lubricant Analysis system.

Materials required:

- · Small volume of heptane or isooctane
- Clean 10 ml Luer-Lok syringe (latex and silicone-oil free)

- Clean, non-abrasive laboratory wipes (unscented and lotion-free; paper napkins work well)
- · Powder-free nitrile gloves
- Safety glasses

CAUTION

Avoid chemical exposure. Isooctane and heptane are chemical irritants. When handling isooctane or heptane:



- · do not ingest
- · avoid contact with skin and eyes
- · avoid inhaling vapor or mist
- · wear gloves that provide adequate protection
- · properly dispose of soiled containers, wipes, gloves, etc.
- To clean the flow-through transmission cell
- 1. If a sample syringe is connected to the cell inlet port, remove the syringe from the inlet port.
- 2. Use a clean laboratory wipe to remove any excess oil from the adapter connected to the cell inlet port.

CAUTION



Splash hazard. Avoid applying excessive pressure to the flow-through cell (via the syringe). Sudden leaks can cause a skin and eyes splash hazard.

- 3. Fill a clean syringe with about 5 ml of heptane or isooctane.
- 4. Screw the end of the syringe into the adapter on the cell inlet tubing and slowly inject all of the solvent through the inlet tubing, flow-cell, outlet tubing and into a waste receptacle.

Tip After you finish measuring the last sample for the day, it is a good idea to clean the cell with solvent and then inject a syringe full of air through the cell to remove the excess solvent.