Thermo-Nicolet 6700 Series FTIR User's Guide

Version 3.0

Edited by Dr. Hartmut G. Hedderich

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The following guide describes the use of the Thermo-Nicolet 6700 FTIR spectrometer. The guide is intended to assist instrument users after the initial training session.

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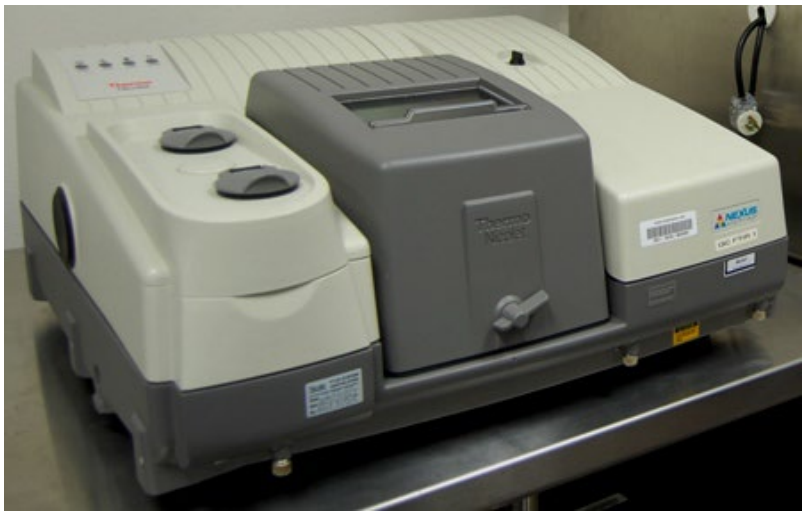
1. Hardware:

The 6700 FTIR is a compact bench unit. It contains separate compartments for light source, sample, beam splitter, and detector.



The 6700 FTIR is ***ALWAYS***(!) turned on. Never try to turn the system off!

The spectrometer has two detectors – an MCT/A detector and a DGTS detector. The MCT/A detector is liquid nitrogen cooled. It needs to be filled prior to the experiment – cool-down time is in the order of 10-15 minutes. The DGTS detector will be installed on request. It has a lower cutoff (350 cm^{-1} instead of 650 cm^{-1} for the MCT/A)

The system needs to be purged with nitrogen to suppress water and carbon dioxide bands. There is a valve marked FTIR on the manifold which will supply nitrogen gas from the liquid nitrogen tank boil-off. This valve is always ON! There is a blue valve in front of the instrument. Turn it to the ON position to purge the FTIR. You want to run the purge for at least 45 minutes before starting your experiments. Do not forget to turn the valve OFF when you are done with your experiments!

Hardware configuration and data collection is fully computer-controlled via OMNIC software (Version 8.3).

In the standard configuration the 6700 FTIR is running with the Smart-iTR module. For gas samples and liquid samples, it can be equipped with a transmission box (see picture above).

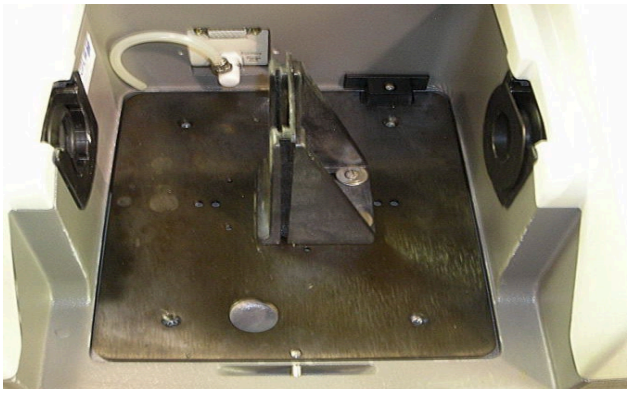
2. **Samples**

Thermo Nicolet FTIR spectrometers can measure gaseous, liquid and solid samples.

The 6700 FTIR standard configuration uses the Smart-iTR setup. The Smart iTR is an ultra-high-performance, versatile **A**ttenuated **T**otal **R**eflectance (ATR) sampling accessory.

Solid and liquid (see restrictions below) samples usually can be measured without further preparation by putting them directly on the ATR crystal. The 6700 system can be configured to hold liquid and gas cells. Most liquids (corrosive, poisonous, BSL-II samples, most solvents) must be put in a liquid cell. Ethanol, methanol, iso-propanol and hexane can be put directly on the ATR crystal. Please contact RIC staff if you don't know if your liquid sample should be put in a liquid cell.

The transmission sample chamber contains a standard holder (see picture). It fits standard IR pellet holders, thin film holders, salt plate holders and gas/liquid cells (user is responsible for supplying those).

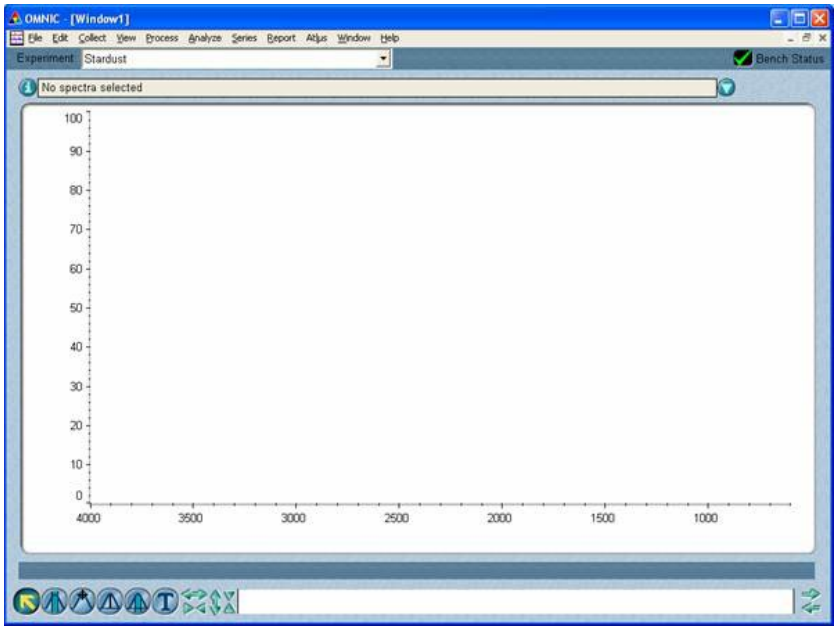


As a rule of thumb, I suggest purging for 1 minute every time you open the sample chamber to change samples. This will give you pretty equal conditions for every background and sample data collection. At the start of your experiment and when the chamber is open for

longer times I suggest purging for several minutes. Check the progress of the purge by taking a background spectrum.

3. **OMNIC Software**

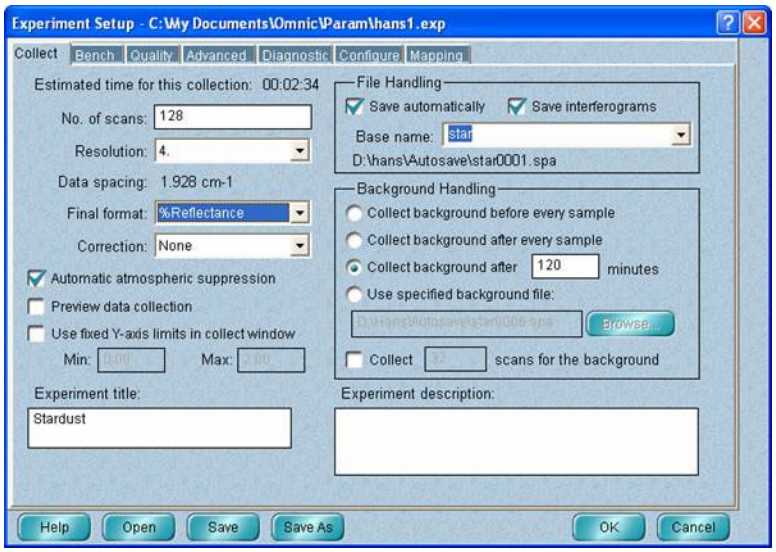
Start the OMNIC Software by double clicking on its icon from the desktop. The program will auto-detect the installed hardware (Smart-iTR or Transmission) and load the correct experiment file. Press OK button. You will see this screen (the screens look a little different with the latest version of OMNIC, but have the same functions):



The green check mark on the bench status shows that the spectrometer is ready to go. To check the signal strength and set the spectrometer parameters, go to the Collect Menu, and choose Experiment Setup.

I. Experiment Setup – Collect Tab

The program will load the **Smart-iTR** setup as default (when the Smart-iTR is installed). For most experiments the default values of the experiment setup are exactly what you need – you do not have to make any changes to the configuration. If you have a sample that needs a different configuration setup (due to very weak or strong signals), please make changes according to the explanation for each tab below. You can save your own setup file under a new name. Please note that the actual screens may look a little bit different due to the version of the software, the functionality is the same!



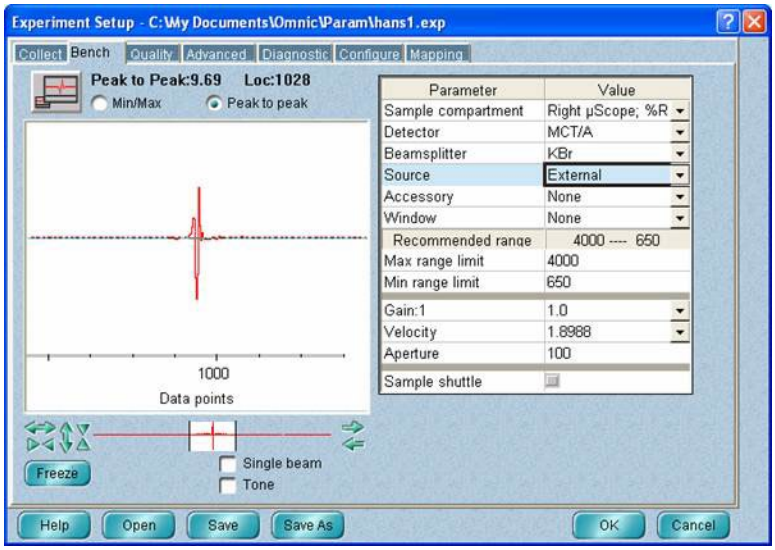
- *No. of scans*: set the number of scans to collect and co-add to obtain your spectrum (more scans will reduce noise but will add to the collection time). **Default = 36**.
- *Resolution*: set appropriately for your sample. Most solid and liquid samples are fine with a resolution of 4 cm^{-1} but check your data to see if any sharp peaks are limited by this resolution setting in which case you may want to increase the resolution to 2 or 1 cm^{-1}. The lower the resolution the faster the data collection time, so only use higher resolutions if you need to and are willing to spend more time collecting each scan. **Default = 4**.
- *Final format*: select the way the data will be processed and presented to you. Most users display their spectra in Absorbance (lines up) or %Transmittance (lines down). %Reflectance will automatically divide your sample spectra by a reference spectrum and present the results in percentage reflectivity. **Default = Absorbance**. Always use absorbance for ATR measurements!
- *Correction*: most users leave this on 'None'. It will select a method to automatically correct for using an ATR (attenuated total reflectance) sampling technique and a few other corrections not typically used with the spectrometer.
- *Automatic atmospheric suppression*: check to have software remove any lines from residual H_2O and CO_2.
- *File Handling*: leave 'Save automatically' deselected and 'Save interferograms' selected. This way you will only save the files you choose to save. By saving the interferogram you will have the ability to go back and reprocess your data using a different background or lower resolution later. If you choose to save all files automatically, you may type a four-character filename that will be incremented and saved in your autosave directory.
- *Background Handling*: 'Collect background before every sample' and 'Collect background after every sample' are usually impractical since they force you to take a

new background before or after each sample. Most users have 'Collect background after *xxx* minutes' selected. This will remind you that after *xxx* minutes have passed that your background spectrum is getting old, and you should really acquire a new one. Pretty much any value between 10 and 120 minutes will work fine. You will need a new background when inverted water lines show up in your spectrum.

- Type in an *experiment description* and an *experiment title* that will be shown on the main OMNIC window.

Once all the parameters are set, you can click on the 'Save As…' button at the bottom of the window and save the experiment file. WARNING: you need to give the experiment a new title and file name. If you don't make these changes, your file will be set as the new default setup file! When you want to go back to the saved experiment setup, just open the Experiment Setup window, click on the 'Open…' button and select the experiment file you want to open.

II. Experiment Setup – Bench Tab



This screen shows the real-time interferogram signal and allows the user to do some spectrometer configuration setup. Assuming you have a sample (which could also be just the air in the compartment) in the sample chamber and that the MCT detector is cooled with liquid nitrogen, you should see a signal like the one shown above.

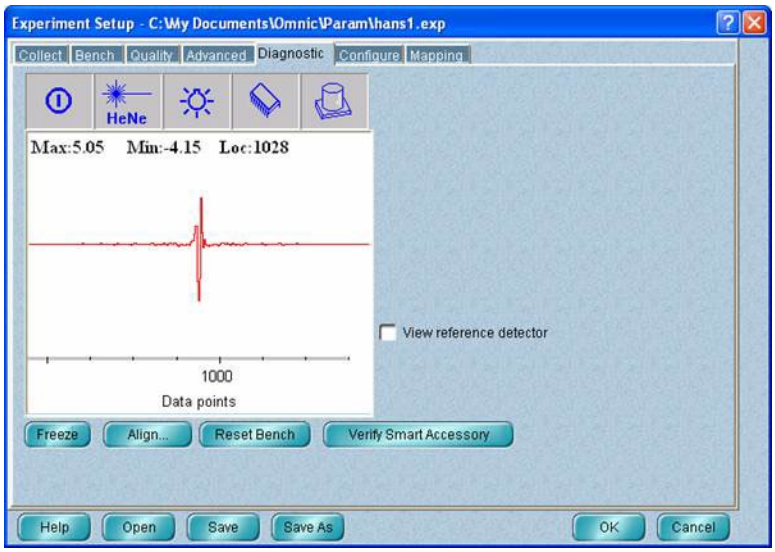
The parameters in this window are:

- *Sample compartment:* Main (only choice).

- *Detector:* The 6700 detector choices are MCT/A and DTGS.
- *Beamsplitter*: The bench is equipped with a KBr beamsplitter.
- *Source:* Only the internal IR source (IR) is available.
- *Accessory*: Depending on setup Smart-iTR or transmission ESP.
- *Window*: With the ATR set to diamond, otherwise choose the window material of your cell if you use one, or KBr for pellets.
- *Spectral Range:* Using the IR source with the KBr beamsplitter, the maximum range is 350-7,400cm^{-1}, the suggested range (best throughput) depends on the detector. The cut-off for the MCT/A detector is 650cm^{-1} and 350 cm^{-1} for the DTGS detector, respectively. You can select a smaller range to match your sample interests.
- *Gain*: Set preamplifier gain to enhance small sample signals. The interferogram signal should be between 4 and 8 for best performance, depending on setup (the **max** value will indicate this on the interferogram display). The system will begin to saturate if the maximum peak signal is > 10, so make sure that you do not turn the gain up beyond these levels. Usually, it is best to leave the gain at the auto setting.
- *Velocity*: Set the mirror speed in the interferometer (measured in cm/sec). A speed of 1.8988 cm/s is typically a good compromise between speed and signal to noise.
- *Aperture*: Best around 10 when using the ATR unit. Using a transmission setup, the aperture range can be anywhere between 20 and 200 depending on the experimental setup used. When optimizing the signal, you can change the value to see which size gives you the best signal (shape and intensity). The smaller the aperture the higher the resolution but the signal strength will get weaker (less light reaching the detector).
- *Screen Wheel*: The screen wheel is at the open position for ATR experiments. Using the transmission setup, the screen wheel can be set to 2%, 10% and 20% attenuation. This will be necessary to avoid detector saturation.

Once again, now that you have all these parameters set, you can click on Save As… (or Save) to save this experiment file so you won't have to reselect everything next time! The screen wheel will always go back to open, independent of how the experiment file was saved.

III. Experiment Setup – Diagnostics Tab



The Diagnostics tab window is where to go when something is not working. The five blue icons along the top of the window indicate the status of the power supplies, alignment laser, internal sources, spectrometer electronics, and IR detectors, respectively. If there is a problem with any of these, a diagonal red slash will appear over the appropriate icon. If you see any of these indications, please call Dr. Hartmut Hedderich at 494-6543 (hhedderi@purdue.edu). If nothing appears wrong, but you still do not see an interferogram signal, you may try pressing 'Reset Bench'. This will re-boot the bench electronics and will cause the spectrometer to re-initialize its zero positions. On occasion when the spectrometer has not had a signal in many hours, this button will make the spectrometer find the zero peak mirror displacement again and start functioning like normal. If you continue to have problems getting a signal, please contact me.

The 'Quality', 'Advanced', and 'Configure' tabs in the Experimental Setup window are generally not used for our experiments. The Quality tab allows you to set up hardware checks during data collection – i.e.: it checks if water and CO_2 levels are too high (no purge gas running), if there is ice buildup on the detector and so on. Should a problem arise, you will get a "warning" during data collection. The Advanced tab allows you to set some of the Fourier transform parameters. The default settings will work for just about everyone, but I would be happy to tell you more about them if you feel you want to get to this level of understanding with FTIR. The 'Configure' tab allows you to configure the FTIR bench. **Please do not change anything within the 'Configure' tab!**

Data Acquisition

Now that the Experiment Setup is complete, the data acquisition can begin!

- First you need to take a background spectrum. Once you are happy with the signal strength seen in the Bench Tab of the Experimental Setup window, close that window by pressing 'OK'. Hovering the mouse over the different icons will tell you which action the icon will perform. Push the Collect Background button. The system may prompt you again to make sure you are ready. Then it will acquire the number of scans you set in the Experiment Setup window. You can see the progress of the scans at the bottom left corner during data collection, and the screen will show you an occasionally updated average of the data you are collecting.

- Once the background is complete, you are ready to measure your sample.
    - Smart-iTR: Put solid sample over the window, use pressure tower to fasten down sample. After the tip touches the sample turn the knob until you hear a single "click". For liquid samples just put a droplet of the liquid onto the lens and collect a spectrum. Make sure to carefully clean the lens with alcohol after use.
    - Gas/liquid cell, pellet, thin film, etc.: Put sample in the sample chamber. Purge for about 1 minute with nitrogen. Then open the Experimental Setup window with the Bench tab selected and check the IR signal strength. When you are satisfied, close the Experimental Setup window by clicking 'OK'. Push the Collect Sample icon. Depending on the output format you have chosen you will get either a spectrum in absorbance or %transmittance.

Now that you have an IR spectrum, you can do a lot of different types of data processing and visualization using the Process and Analyze menus. These functions will not be covered here but can be found in the extensive on-line help files for OMNIC and the OMNIC manuals.

IV. Diamond ATR Crystal Features and Specifications

- Inert and robust – excellent for analysis of hard, abrasive, reactive, caustic, and corrosive materials
- Disadvantage – intrinsic absorption from about 1800 – 2300cm^{-1}
- Low wavenumber cutoff: 525cm^{-1}
- Depth of penetration: 2.03μm at 1000cm^{-1}
- Refractive index: 2.4
- Useful pH range: 1 to 14 (ZnSe lens 5-9)
- Incident angle: 42°

V. Notes