

# IMSERC User Manual for FTIR

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## INTRODUCTION

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Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at <http://imserc.northwestern.edu/pcm-instruments.html>. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at <http://imserc.northwestern.edu/pcm-instruments.html> by pressing on the 'User manual' button under "Bruker Tensor 37 FTIR (MiD IR / ATR)". If while using the system, something happens that you do not understand, please **stop**, and **get help**. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

Please remember to:

1. The amplitude of the incident IR radiation **MUST** be recorded before starting any data acquisition and after all the measurements are done by a user
2. Leave the acquisition software open when you are done with the measurement
3. End your reservation from NUcore when you are done with the experiment.
4. Leave lab tables clean and tools/accessories organized
5. Report problems with the instrument (see troubleshooting section for more details, if any)

## SAFETY

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All users of IMSERC must review the general safety policies at <http://imserc.northwestern.edu/about-policies.html> and the Crystallography specific policies at <http://imserc.northwestern.edu/crystallography-policies.html>. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

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- Laboratory Safety
- Personal Protective Equipment

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at <https://learn.northwestern.edu>. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in room B176. Protective eyewear is required in this room, and gloves should be removed when using the computer.

## DATA MANAGEMENT

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Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied in your supervisor's group folder on 'imsercdata.northwestern.edu' under 'Others/FTIR' once every hour. Please follow instructions at <http://imserc.northwestern.edu/about-general-faq.html#data> for details about data access.

## SOFTWARE

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Data reduction and analysis can be performed with the software 'OPUS'. Software is installed on the instrument computer. For offline analysis after your instrument reservation is complete, please use the following resources:

- For registered IMSERC users, software can be used remotely through IMSERC's Terminal Server. Please read more information at the 'What is IMSERC's Terminal Server and how do I get access to it?' section under the 'Software Access' category at <http://imserc.northwestern.edu/about-general-faq.html>
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore

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## DEFAULT INSTRUMENT STATUS

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In describing the steps involved in collecting a data set, several assumptions have been made regarding the status and conditions of the instrument. The default working condition of the spectrometer is listed below:

1. Computer screen is by default deactivated. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore
2. The default 'FTIR' user account should be logged in. In case the computer was restarted, the password for the 'FTIR' account is
3. The instrument power is always turned on. Never turn the power off
4. The sample chamber is always flushed with nitrogen gas from facilities
5. The **default mode for the FTIR instrument is ATR mode**, hence the **users must reinstall the ATR module** in case they use the transmission module for their experiment. Watch the '[ftir-atr-stage-mount-unmount](#)' video for a visual demonstration
6. The ATR attachment is mounted on the spectrometer
7. Clean the crystal only with the provided cotton swabs dipped in isopropanol to avoid damage to the crystal
8. The ATR tip can be cleaned with an ultrafine sandpaper (can be found on the shelf above the FTIR instrument)

When you are done with your measurement, please remember to:

1. Leave the acquisition software open when you are done with the measurement
2. End your reservation from NUcore when you are done with the experiment
3. Leave lab tables clean and tools/accessories organized
4. Report problems with the instrument (see troubleshooting section for more details)

If there is an error or problem with the instrument which is not addressed under the '[Troubleshooting](#)' section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
3. Contact a staff member for instructions


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## DATA COLLECTION

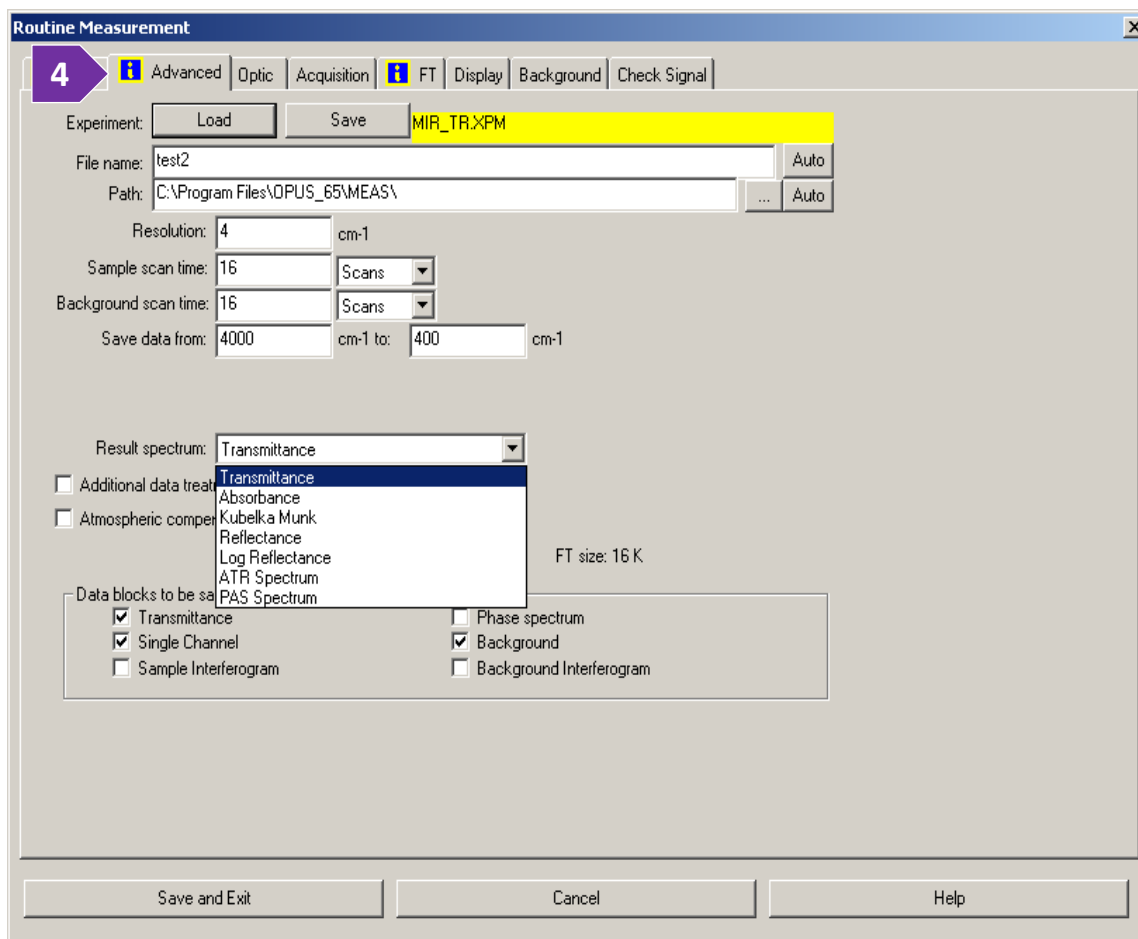
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This standard operating procedure is meant for training students/postdocs with the FTIR instrument available at IMSERC. Do not run this instrument without this training or approval from IMSERC staff. Failure to do so may cause damage to the instrument and result in additional fees and/or removal of all IMSERC privileges. This short set of instructions is meant to serve as a guide for 'routine' usage on the instrument.

### A. SETTING THE ACQUISITION PARAMETERS

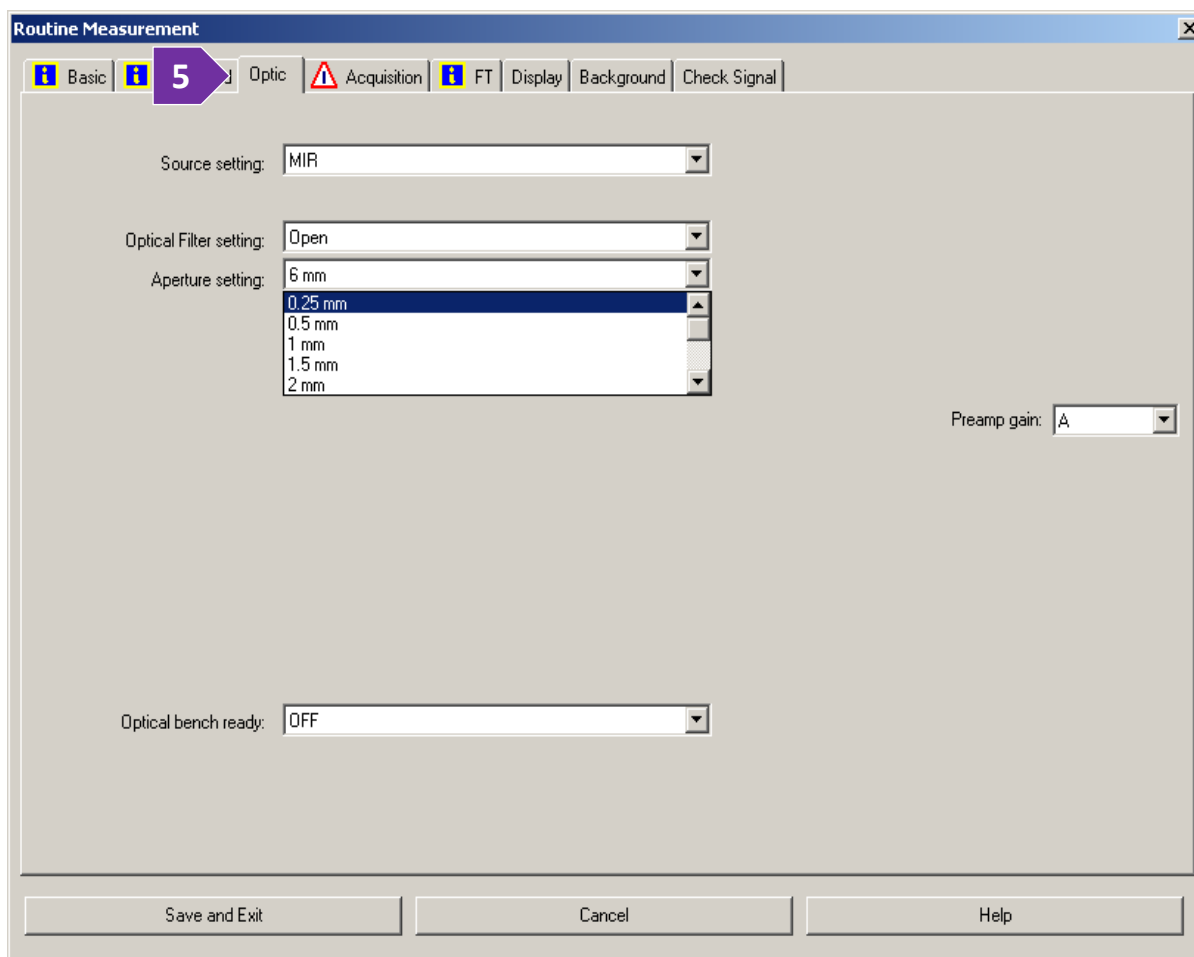
1. Open the OPUS software. The password is 'OPUS' (all caps)
2. Press on the  icon on the top right of the 'OPUS' software
3. Under the 'Basic' tab, load the 'Experiment' file, i.e., 'MIR\_TR.XPM (%T)' or 'MIR\_ATR.XPM (for ATR)', depending on the mode of operation. Please **do not overwrite** these two files. Ensure that the appropriate attachment, i.e., ATR or transmission stage, is attached. Watch the ['ftir-atr-stage-mount-unmount'](#) or ['ftir-transmission-stage-mount-unmount'](#) video for a visual demonstration for the proper installation of these attachments. Next, set the 'File name' and file 'Path'
4. Press on the 'Advanced' tab (figure 4). You do not need to change any setting here other than the first two items except for any specialized experiment(s)
  - a. Enter the 'File name'
  - b. Choose the path of the file to be saved. You must create a folder during the training session in the 'User data' folder which can be found in desktop. Usually, the directory path is 'UserData'/'PIName'/'LastName-FirstName'
  - c. Set the resolution between 2 – 8 (for solid and liquid) and down to 0.3 for gases
  - d. Number of background and sample scans should be the same. They can be set to 16, 32, 64 and so on. Increasing the number of scans will increase the signal/noise ratio but the data collection time will also increase. Typically, it takes around 1 minute to complete 16 scans
  - e. Range should be set in the range of 4000 to 400  $\text{cm}^{-1}$  for transmittance mode and 4000 to 600  $\text{cm}^{-1}$  for ATR mode
  - f. Result spectrum should be set as 'Transmittance'
  - g. Data blocks to be saved are usually- Background (reference), Single channel (sample), and %Transmittance (Sample/Ref) or Abs (log (1/%T))

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5. Next, press on the 'Optics' tab (figure 5)
  - a. Keep the "source setting" to MIR
  - b. Optical filter should be "open"
  - c. The default aperture value should not be changed under normal experimental conditions which is generally 6 mm for solid or liquid samples. Do not set the aperture setting higher than 6 mm. You can imagine the aperture length similar to slit length. It ensures that only the least divergent part of the IR radiation enters the interferometer. If you are working with gaseous samples, you may need to tune the aperture to a smaller number to increase the resolution of the instrument to resolve narrower peaks from gaseous phase.
  - d. Optical bench ready should be 'off'
6. Do not change any parameters under 'Acquisition', 'FT', and 'Display' tabs

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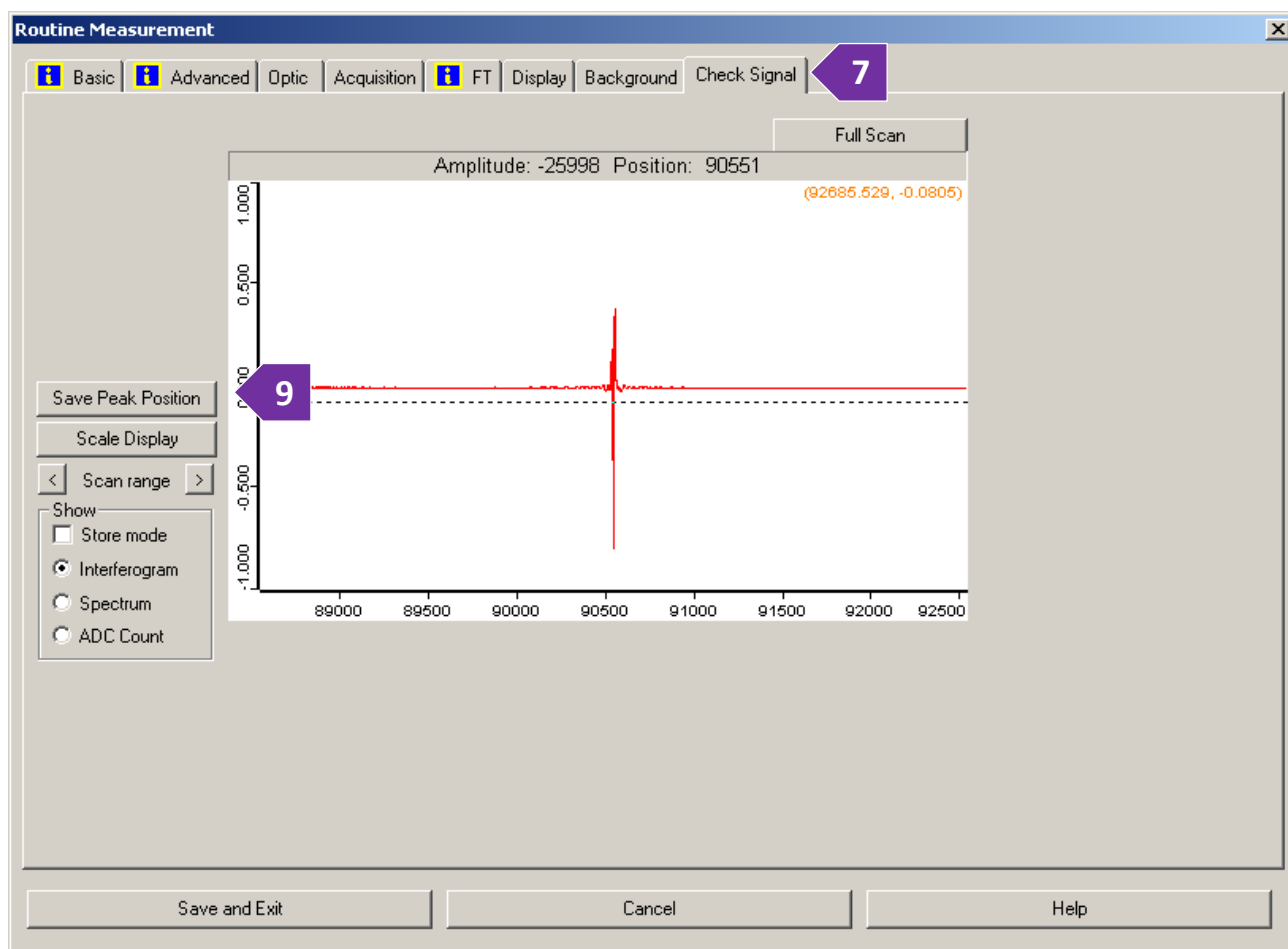
## B. CHECK SIGNAL

Before collecting the background and prepare for your sample measurement, you need to check the quality and intensity of the signal:

7. Press on the 'Check Signal' tab (figure 7)
8. In transmission mode the amplitude of the intensity signal should be near 18,500 with no sample. In ATR mode the amplitude should be near 6,500 with no sample and when the tip is lowered. Watch the ['ftir-atr-lowering-raising-tip'](#) video for a visual demonstration for lowering and raising the ATR tip. Please note the following points carefully:
  - a. You **must** enter the amplitude values **before** and **after** the experiment in the Excel spreadsheet 'IntensityTracker.xlsx' which can be found on the desktop
  - b. You can look up the previous amplitudes recorded by previous users in the above-mentioned Excel spreadsheet. The value should be  $\pm 100$  compared to previous runs

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- c. If the amplitude is substantially off the above limit, clean the diamond surface and the ATR tip and check the signal again. Watch the [‘ftir-atr-cleaning’](#) video for a visual demonstration for cleaning the ATR attachment
  - d. Inform the staff if the problem persists even after performing the above step
9. Once the signal almost stabilizes (a few seconds), press on ‘Save Peak Position’ button (figure 9)



## C. BACKGROUND COLLECTION

It is highly recommended to collect the background spectrum before your measurement. Lower the ATR tip down before the background measurement. Watch the [‘ftir-atr-lowering-raising-tip’](#) video for a visual demonstration for lowering and raising the ATR tip

10. Go back to the ‘Basic’ tab and press on the ‘Background Single Channel’ button (figure 10). You can save this background for future use, but it highly recommended that a new background spectrum is collected before every sample data collection, because:
  - a. Taking a new background will help remove signals from impurities left by the previous samples



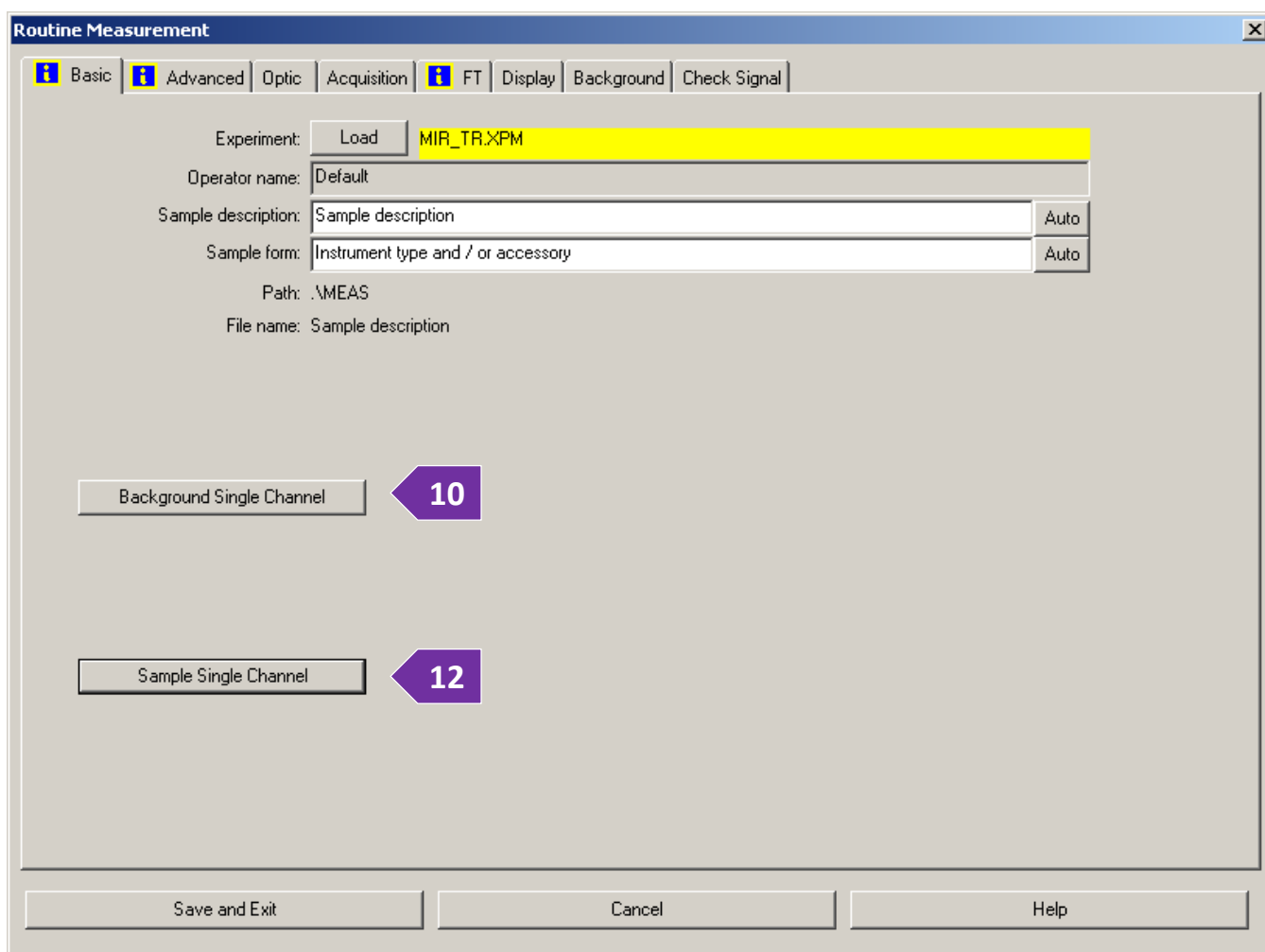
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- b. Counteract for any drift that may have occurred in the light source between experiments
- c. Corrects for any fluctuations of the environment. The nitrogen does not flow continuously, and sometimes if the flow is running while taking a background and not during the experiment (or vice versa), the CO<sub>2</sub> peak will differ in the spectrum

## D. SAMPLE COLLECTION

After the background data collection is over, you are ready to start collecting data on your sample:

11. Place very small quantity of sample just enough to cover the diamond surface (for solid) or a small drop (for liquids) on the diamond stage and lower the ATR tip down by rotating the cap until it clicks. Watch the [‘ftir-atr-lowering-raising-tip’](#) video for a visual demonstration for lowering and raising the ATR tip
12. Under the ‘Basic’ tab, press on the ‘Sample Single Channel’ button (figure 12)
13. Once the spectrum appears on the screen press ‘start’



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## PUBLICATION

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### A. EXPERIMENTAL SECTION

*Modify the text below according to the setup and conditions you used during the measurement:*

“FTIR data were collected at room temperature on a Bruker Tensor 37 FTIR Spectrometer equipped with a Mid IR detector and KBr beam splitter. The spectrum was collected in [***attenuated total reflectance (ATR) | transmittance***] mode in the range of ***XXXX*** to ***YYY***  $\text{cm}^{-1}$ . The data were averaged over ***XX*** scans. The OPUS software was used for the data acquisition.”

### B. ACKNOWLEDGEMENT

“This work made use of the IMSERC facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University.”

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## TROUBLESHOOTING

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### A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

### B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default 'FTIR' user account should be logged in. In case the computer was restarted, the password for the 'FTIR' account is . See '[Default instrument status](#)' section for more details.

### C. ACQUISITION SOFTWARE REQUIRES LOGIN AND A PASSWORD

The standard credentials for operating the acquisition software (OPUS) are user ID 'Default', and password 'OPUS'

### D. THERE IS AN ERROR OR PROBLEM WITH THE INSTRUMENT NOT ADDRESSED UNDER THIS TROUBLESHOOTING SECTION

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> add place the 'Stop' sign near the instrument computer. 'Stop' signs are located on the shelf above the computers in BG51 and online at the link above. Email or talk to a staff member
3. Email or talk to a staff member

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## REVISIONS

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v1.12 2021/9/22	<ul style="list-style-type: none"><li>• Troubleshooting section updated</li><li>• Training video files transferred from Box to OneDrive, and all relevant links were updated</li></ul>
v1.10 2020/8/28	<ul style="list-style-type: none"><li>• Links to online video modules added for demonstrating (un)mounting processes for the ATR and transmission stages and cleaning the ATR stage. Additionally, references to the videos for proper lowering and raising of the ATR tip were added in the corresponding sections</li><li>• Reformatted according to the latest template. Sections about 'Safety', 'Data management', 'Software', 'Publication', and 'Troubleshooting' were added</li></ul>
v1.00 2017/10/16	<ul style="list-style-type: none"><li>• First version of the user manual</li></ul>

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