

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***CORAL**

You must be a “Qualified Self-User” to operate this instrument

You must be on the labs “Instrument Reservation Schedule” before touching the instrument for any reason.

When you arrive, Engage the instrument using CORAL.

When you leave, Disengage the instrument using CORAL.

Any problems, STOP, post a note on the instrument and send an email to mtim@mit.edu immediately.

Room Hazards (13-4139 & 13-4148)

Cryogenic Liquids, Chemicals & Lasers.

Safety

Required Apparel – Gloves, Lab Jacket, Safety Glasses.

No Hydro Fluoric Acid usage.

No Viable Biological Samples.

EMERGENCIES DIAL100 (From an MIT phone)

MIT Police (617) 253-1212

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Startup**

Open Omnic.

Wait for the self-test to finish.

Modify the “Experiment Setup” to suite your needs.

Run the measurement

Load the background sample holder if used.

Collect a background.

Save the data.

Load the sample.

Collect a sample.

Save the data.

Process the data.

Resave the data.

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Measurement End:

Walkaway with a copy of your data.

You will find a shortcut to the data folder on the desktop.

Do not count on your data being stored on this computer.

Close Omnic. And any other windows you might have opened.

Shutdown

Cleanup – Neatness Counts.

Do not leave your samples here.

Finishing Up

Disengage CORAL at the front of the lab.

Report any problems, comments or suggestions to: mtim@mit.edu

Problems?

It is ok to restart the bench and computer.

Email mtim@mit.edu immediately if you have to do this.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Emergency Shutdown**

Ok to leave as is.

Secure your chemicals.

Walk away.

Startup after a power loss

Restart Computer.

Restart the Bench.

Restart the Microscope.

Procedure for Extended Shutdown

Secure chemicals in exhaust hood.

Close the Bench.

Turn Off the Bench.

Turn Off the Microscope.

Turn Off Computer.

Important: Maintain Purge.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***ATR Procedures****Diamond ATR in the IS50 Bench**

Push the button next to the ATR.

Collect a background.

Save it.

Place your sample on the Crystal.

Collect a sample.

Save it.

When finished with your ATR measurements

collect a sample with the crystal in air.

You should not see any residue from your samples.

If you do, clean the crystal.

To clean the diamond ATR in the bench:

Put a little methanol on a clean room wipe and wipe the crystal clean.

To clean the Germanium Tip ATR Crystal:

Rinse the crystal with warm water.

Rub crystal with hand soap for 30 seconds.

Rinse & dry.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Microscope Reflection**

Chill the MCT-A (or B) detector on the microscope with Liquid Nitrogen.

MCT-A is more sensitive but only goes down to 600cm⁻¹.

MCT-A is less sensitive but goes down to 450cm⁻¹.

Open Experiment Setup: on the second tab choose the Microscope on Right side in %R.

Open the Altus Window.

Set Aperture to default (100um x 100um).

Set the Objective Compensation Ring (Calibrated in mm of salt).

Place the gold mirror on the stage.

Turn on the Reflected Light Illumination.

Adjust the Eyepiece Reticle to your eye.

While looking in the eyepieces adjust the stage height until the mirror surface is in focus.

Illuminate the Reflex Aperture.

Verify the Reflex Aperture is in focus and centered in the eyepiece reticle.

Collect a Background.

Save the data.

Place your sample on the stage.

While looking in the eyepieces adjust the stage height until the sample surface is in focus.

Use the joystick to move the microscope stage to your area of interest.

While looking in the eyepieces re-adjust the stage height until the sample surface is in focus.

Collect a Sample.

Save the Data.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Microscope transmission**

Chill the MCT-A (or B) detector on the microscope with Liquid Nitrogen.

MCT-A is more sensitive but only goes down to 600cm⁻¹.

MCT-A is less sensitive but goes down to 450cm⁻¹.

Open Experiment Setup: on the second tab choose Microscope on Right in %T.

Open the Altus Window.

Set Aperture to default (100um x 100um).

Set the Objective Compensation Ring (Calibrated in mm of salt).

Set the Condenser Compensation Ring (Calibrated in mm of salt).

Place the Microscope Alignment Slide on the stage.

Turn on the Reflected Light Illumination.

Using the button on the front of the microscope, choose the Reflected Light configuration.

Adjust the Eyepiece Reticle to your eye.

While looking in the eyepieces adjust the stage height until the mirror surface is in focus.

Illuminate the Reflex Aperture.

Verify the Reflex Aperture is in focus and centered in the eyepiece reticle.

Move the stage to the open area of the microscope alignment slide.

Using the button on the front of the microscope, choose the Transmitted Light configuration.

Turn on the Transmitted Light Illumination (knob on side of microscope).

Adjust the Condenser height until the reflex aperture image is in focus.

Adjust the Condenser position until it is centered in the eyepiece reticle.

Collect a Background.

Save the data.

Place your sample on the stage.

While looking in the eyepieces adjust the stage height until the sample surface is in focus.

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Use the joystick to move the microscope stage to your area of interest.
Using the “move stage” menu item, choose to set the current position to home.
While looking in the eyepieces re-adjust the stage height until the sample surface is in focus.
Move the stage to the open area of the microscope alignment slide.
Adjust the Condenser height until the reflex aperture image is in focus.
Adjust the Condenser position until it is centered in the eyepiece reticle.
Move the stage to the home position.
Collect a Sample.
Save the Data.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Microscope ATR**

Chill the MCT-A detector on the microscope with Liquid Nitrogen.

Verify the Contact alert stage is installed and working properly (wired microscope stage), the green light comes on.

Clean the ATR Crystal.

Install the Slide on ATR onto the bottom of the 15X IR Objective.

Move the ATR Crystal slider to the contact position.

Open the Experiment Setup

Bench tab – Choose Microscope on the right side in %R.

1st tab – choose ATR correction.

Set Aperture to default (100um x 100um).

Note: ATR sampling size will be 25x25um.

Adjust the Compensation objective to optimize the peak to peak voltage of the interferogram (~12.5 Peak to Peak Voltage with a Gain of 2).

Collect a Background (in air).

Save the data.

Place your sample on center of the contact alert stage area and verify the green light comes on when contact is made.

Move the slider to the view position.

Find your area of interest on the sample.

Move the slider to the contact position.

Open the Altus Window.

Click on - ATR Contact icon at the bottom of the atlas window.

Collect a sample.

Save the data.

Lower the microscope stage (Down - thumbs towards you).

Clean the Crystal as needed.

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GE ATR Crystal Cleaning

Review the manual for the for proper handling of the delicate crystal.

Wear Gloves.

Use N2 Gun to remove debris.

Clean with Hand Soap and warm water.

Dry using N2 Gun.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Grazing Angle ATR (GATR)**

Collect a background with the default bench configuration.

Install the GATR

Inspect crystal.

Plug the purge line into the bench sample area connection.

Mount the GATR in bench using screw to hold it down.

Attach purge line to GATR.

Verify Throughput

Collect a sample.

Expected Throughput in Transmission

Unpolarized:

Perpendicular Polarized: 5.5sb

Parallel Polarized: 5.5sb

Cool the MCT-A Detector

Wear the appropriate safety apparel; Safety Glasses or Goggles, Cryo Gloves, Lab Jacket.

Fill completely.

Wait five minutes.

Open the GATR Experiment File

Review the experimental settings and update as needed.

Collect a Background (Air, no sample).

Save the data.

Place your sample on the GATR

Do not touch the crystal or let your sample slide (scratch) the crystal.

While holding the sample to prevent rotating, slowly bring the plunger into contact with your sample.

Twist the torque wrench until the plunger will no longer move.

Collect a sample

Save the data.

Repeat for all your samples.

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When Finish Measuring Samples

Verify the crystal is not contaminated.

- Close the software.

- Open the software.

- Collect a sample.

- Review the spectra for contamination peaks.

If contamination is found, clean the crystal. (You will have to be trained by lab staff to clean the crystal).

Remove the GATR and Purge tubing from the bench.

- Replace the Transmission sample holder.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***VMAX III Variable Angle Measurement Accessory**

Specular Reflection, ATR, Polarizers, Electrochemical Cell.

SpecificsMeasurement Range: 12,500cm⁻¹ to 50cm⁻¹.

Specular Reflection 30-80 degrees.

ATR Germanium 65 degrees.

Zinc Selenide Polarizer.

Far IR Polarizers.

Jackfish Electrochemical Cell.

Do not adjust optics.

Wear Gloves.

Keep Polarizer covered when not in use.

Specular Reflection

Connect Purge Line.

Install the VMAX III.

Install the appropriate aperture for your sample.

Note, Smaller apertures require smoking to avoid sinusoidal wave from anodized coating.

Install Polarizer if needed and set angle.

Pike Polarizer Setting: 0deg = S (TE), 90deg = P (TM).

Set your measurement angle (30-80 degrees).

Experiment Settings – Final Format %Reflectance.

Collect Background (Save your data).

Replace the gold mirror with your sample.

Collect Sample (Save your data).

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Absolute Transmission Accessory**

Staff Only are authorized to change the optics.

Schedule time with mtim@mit.edu when you would like to use the Absolute Transmission Accessory.

Install ABS-T – Staff Only

Turn the bench power off.

Remove the Instrument front covers (Left & Right).

Remove the Focusing Mirror (Concave).

Install the ABS-T Focusing Mirror (Flat).

Remove the Detector (Concave).

Install the ABS-T Detector (Flat).

Install the ABS-T sample holder baseplate.

Replace covers.

Turn the bench power On.

Put note on instrument.

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Open Omnic.

Choose the “Absolute Transmission” Experiment File.

Place the ABS-T Baseplate into the bench.

Place the correct size aperture for your sample in the sample holder.

Collect a Background and save the data.

Place your sample in the sample holder.

Collect a sample and save the data.

Close Omnic.

Replace the original sample holder baseplate into the bench.

Post Sign – ABS-T now installed.

Email mtim@mit.edu your finished.

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Turn the bench power Off.

Remove the Instrument front covers (Left & Right).

Remove the ABS-T Focusing Mirror (Flat).

Install the original Focusing Mirror (Concave).

Remove the ABS-T Detector (Flat).

Install the Original Detector (Concave).

Install the original sample holder baseplate.

Replace covers.

Turn the bench power On.

Open Omnic.

Allow self-test to run.

If OK, Close Omnic.

Remove Note.

Carefully put all the pieces back in the box with the detector sealed and desiccated.

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The Absolute Transmission Accessory is
now installed

Collimated beam in the sample
compartment

Date: _____

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Making a KBr Pellet**

This procedure taken from the International Crystal Labs Literature
www.internationalcrytallabs.net

Hazards

(Insert KBr MSDS here)

Apparel

Gloves, Lab Jacket, Safety Glasses

Recipe for 13mm Pellet

300mg Potassium Bromide and 1-4mg sample
0.625-1 parts sample to 100 parts KBr

The KBr is stored in the oven at 150C (HOT).

Wear gloves when removing and place on a safe surface in the hood.

Procedure

Using the Agate Mortar & Pestle

Grind your sample.

Then Slowly grind the kBr into your sample ~4mg at a time.

Grind 3-5 minutes depending on sample composition.

Then mix your sample in the Wig-L-Bug for 2-5 minutes.

(Insert Wig-L-Bug SOP Here)

Press your pellet

(Insert Pellet Press SOP Here)

A KBr only pellet can be made and used as a Background to eliminate excessive moisture bands.

This is also a good way to check for contamination of the KBr.

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Place the metal parts in the ultrasonic cleaner when finished.

FTIR IS50**<https://mtim.mit.edu/ftir>***Last updated 20220125***Microscope Emission Measurement**

Note: The Dichroic in the microscope cuts off below 1000cm⁻¹.

Range: 10,000cm⁻¹ to 1000cm⁻¹

Focus the IR Microscope objective onto the emitter.

Set final format – single beam.

Set source to collimated emission.

Install MCT-A detector into bench and chill.

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Where to find software to process your optical spectroscopy data.

Omnic Anywhere Cloud based software – Works with PC, Mac or Linux. You need to create an account (free). Allows you to view and process data.

<https://www.thermofisher.com/us/en/home/industrial/spectroscopy-elemental-isotope-analysis/molecular-spectroscopy/fourier-transform-infrared-ftir-spectroscopy/cloud-enabled-ftir-spectroscopy.html>

Spectragryph – Free downloadable spectroscopy software made available by the Spectroscopy Ninja.

<https://www.effemm2.de/spectragryph/index.html>

An older version of Omnic is installed on the computer at the front of the lab (13-4139).

Raman Wire Software is available on the desktop of the Raman computer. Bring a Flash Drive.

ICP Expert Software is available on the desktop of the ICP computer (13-4148). Bring a Flash Drive.