FTIR 6700

Last Updated: 4/21/2020

You must be a "Qualified Self-User" to operate this instrument independently.
You must be on the labs "Instrument Reservation Schedule" before touching the instrument for any reason.
Any problems, STOP, Post a note on the instrument and send an email to mtim@mit.edu immediately.
Do not perform any maintenance.
Do not install any software
Do not adjust any optics.

FTIR6700 Specific Hazards
Electrical: 110-120V, 60Hz
Cryogenic Liquids in use (LN2, LHe)

System Found State
Purge Gas: ~50sccm
Bench: On
Computer: On
Software: Closed
Microscope: On
Microscope ATR Stage (wired) in place.

Verify the instrument performance

Open the "Omnic" Software.
Collect a Background.
Verify Moisture & CO2 levels are acceptable (see example printout).
Check data: at least 50 single beam units (Gain of 2) and acceptable CO2 (2400cm-1) and Moisture levels.
Low SB units? Try realigning the interferometer using the "Align" button in the experiment setup window on the "Diagnostic" tab.

Default Bench Configuration
Mid IR 4000 – 400 cm-1
Module: Main Bench
Source: IR
Aperture: 100
Beamsplitter; KBr

Detector: DTGS (Back Position)
Note: The FTIR will be reset to the Default Bench Configuration when Omnic is opened.

Operation

Transmission in the Bench Mid IR (4000 – 400 cm-1) Range.
Open Omnic
Open the Experiment Setup window and set it up for your experiment.
Collect Background.
Save the data.
Insert Sample
Collect Sample
Save the Data.

Reflection in the Bench Mid IR (4000 – 400 cm-1) Range.
Open Omnic
Open the Experiment File: OTC_(Reflection acc name)
Install the reflection accessory.
Collect Background.
Save the data.
Insert Sample
Collect Sample
Save the Data.
Disengage CORAL.

Utilities:
Electricity (wall).

Emergency Shutdown:
Shutdown the computer.
Turn off the spectrometer power.
Power switch is located on side of the spectrometer facing the wall.

Restart after an emergency:
Restart the computer.
Turn on the spectrometer power.
Power switch is located on side of the spectrometer facing the wall.

Changing the Beamsplitter

Hold the beamsplitter by the metal tab on top.

Inspect Beamsplitters for damage/fingerprints, etc.. when removing or installing.

Beamsplitters should always be positioned verticlyu, no sag.

Realignment of the beamsplitter should be performed in the bench configuration with the source you intend to use.

1. Open the Beamsplitter compartment door.
2. Open Beamsplitter Lock (180 degrees only).
3. Lift beamsplitter out of the interferometer.
4. Lift the new beamsplitter out of the storage compartment.
5. Store the old beamsplitter in the storage compartment.
6. Place the new beamsplitter into the interferometer.
7. Close the Beamsplitter Lock (180 degrees only).
8. Close the beamsplitter compartment door.
9. Change the beamsplitter label.
10. Choose the Source" you want to use.
11. In the Experiment Setup, Diagnostic Tab, Align the bench.
12. You are ready to collect data.
13. When finished, replace the original (KBr) beamsplitter and change the label.
14. In the Experiment Setup, Diagnostic Tab, Align the bench.
15. Verify the background is ok (50 Single beam units with a gain of 2).

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 Atlus 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 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.

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 Atlus 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 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.

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

Using the “move stage” men 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.

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 Atlus Window.
Click on - ATR Contact icon at the bottom of the atlus window.
Collect a sample.
Save the data.
Lower the microscope stage (Down - thumbs towards you).
Clean the Crystal as needed.

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.

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.

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, clan 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.

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 Morter & 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.

Place the metal parts in the ultrasonic cleaner when finished.

Data Analysis

Omnic Software
Spectral Resources:
MIT Library page where they have gathered the spectra resources available through MIT Site License:
http://libguides.mit.edu/content.php?pid=94486&sid=706303

Here is a site called ramansearch.com or FTIRsearch.com:
http://ramansearch.com/
This is a commercial site. You can search it using your spectra but will have to have an account to view the results. Tis is good for FTIR or Raman spectra.

Shutdown
Replace the wired microscope ATR stage if removed.
Walk away with a copy of your data. This computer's hard drive is periodically purged and will eventually crash.
Close and Re-Open the Omnic Software.
Collect a Background.
Check data: at least 50 single beam units.
Close the software.
Clean up.
Disengage CORAL.

Specifics
Thermo Fisher FTIR6700 Bench and Continuum FTIR Microscope

Computer Restarting
Name: administrator
Password: (blank)

Computing
Data Saving Capabilities
Export

Data Formats

Internet Access: Yes
USB: Yes
CD: Yes

DVD: Yes
Floppy: Yes

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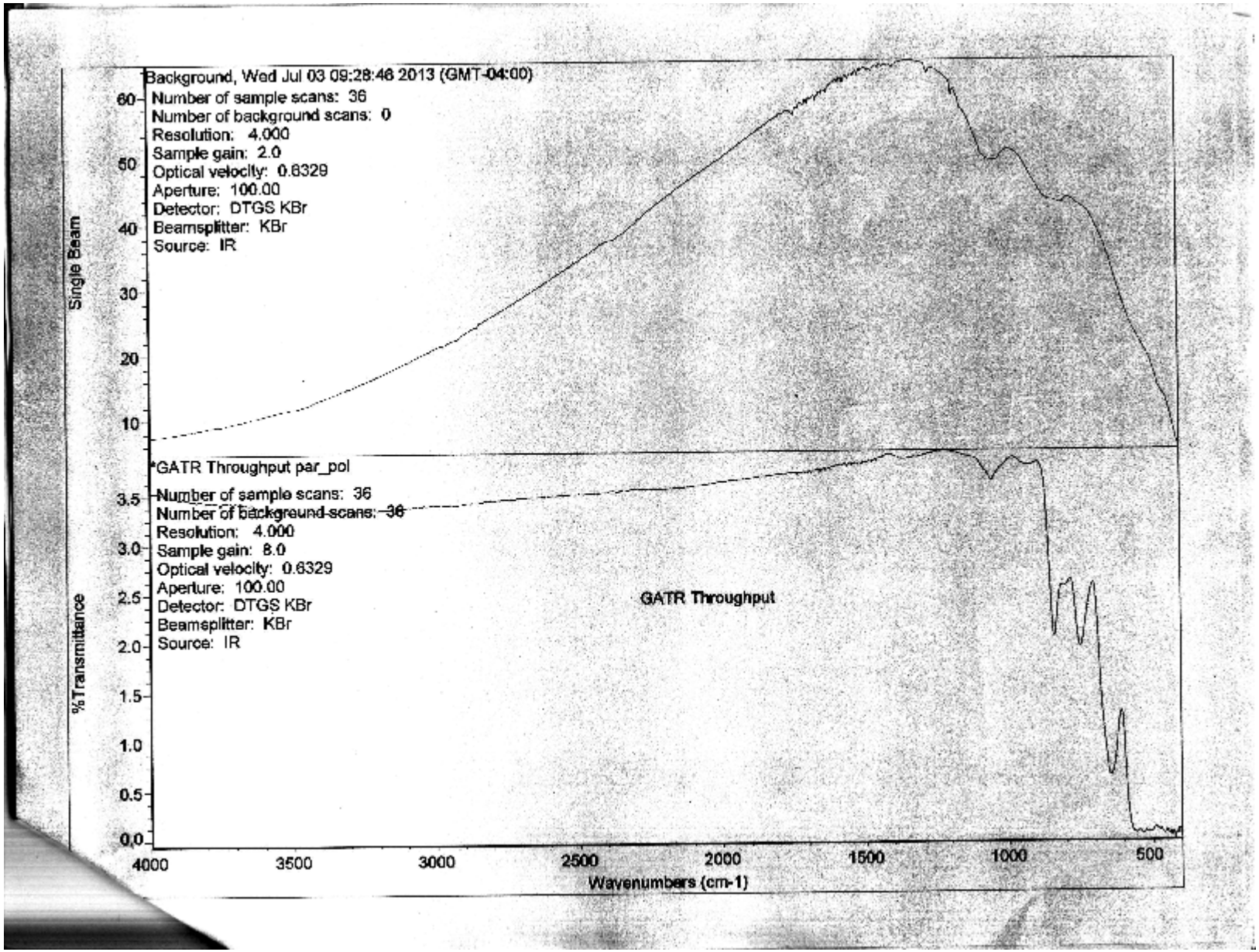
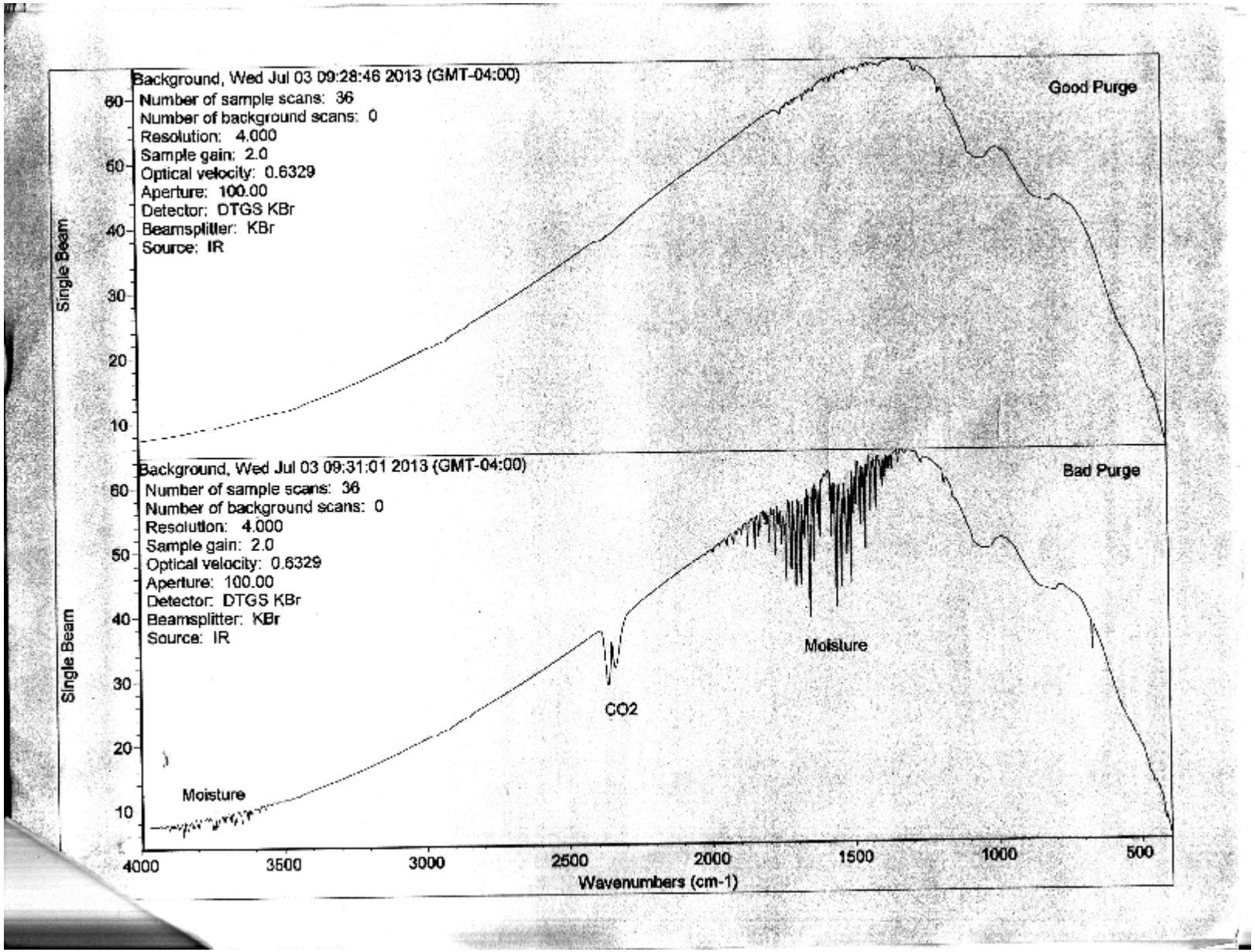

Background, Wed Jul 03 09:28:46 2013 (GMT-04:00)
Number of sample scans: 36
Number of background scans: 0
Resolution: 4.000
Sample gain: 2.0
Optical velocity: 0.6329
Aperture: 100.00
Detector: DTGS KBr
Beamsplitter: KBr
Source: IR
Single Beam
60
50
40
30
20
10
GATR Throughput par_pol
Number of sample scans: 36
Number of background scans: 36
Resolution: 4.000
Sample gain: 8.0
Optical velocity: 0.6329
Aperture: 100.00
Detector: DTGS KBr
Beamsplitter: KBr
Source: IR
GATR Throughput
%Transmittance
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


Background, Wed Jul 03 09:28:46 2013 (GMT-04:00)
Number of sample scans: 36
Number of background scans: 0
Resolution: 4.000
Sample gain: 2.0
Optical velocity: 0.6329
Aperture: 100.00
Detector: DTGS KBr
Beamsplitter: KBr
Source: IR
Good Purge
Background, Wed Jul 03 09:31:01 2013 (GMT-04:00)
Number of sample scans: 36
Number of background scans: 0
Resolution: 4.000
Sample gain: 2.0
Optical velocity: 0.6329
Aperture: 100.00
Detector: DTGS KBr
Beamsplitter: KBr
Source: IR
Bad Purge
Moisture
CO2
Moisture
Single Beam
Single Beam
60
50
40
30
20
10
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

Specifics:

Iris setting	Iris dia.	Theoretical dia. in sample compartment	Image size at detector	Half angle Beam divergence	TGS Interferogram Volts p-p
APT=150	10.7mm	10.7mm	1.98mm	2.0 degrees	12.15
APT=115	9.3mm	9.3mm	1.72mm	1.75 degrees	11.70
APT=100	8.75mm	8.75mm	1.61mm	1.6 degrees	11.46
APT=75	7.52mm	7.52mm	1.38mm		10.62
APT=50	6.27mm	6.27mm	1.15mm		9.33
APT=32	5.08mm	5.08mm	0.94mm	0.95 degrees	7.57
APT=25	4.50mm	4.50mm	0.85mm		6.72
APT=9	3.00mm	3.00mm	0.55mm		3.73
APT=6	2.50mm	2.50mm	0.46mm	0.47 degrees	2.70
APT=4	2.00mm	2.00mm	0.37mm		1.96
APT=1	1.30mm	1.30mm	0.24mm	0.25 degrees	0.72
APT=0	0.75mm	0.75mm	0.14mm	0.14 degrees	0.26

Thermo Fisher 6700 FTIR Bench
Measurement Range: 12,500cm-1 to 50cm-1
Samples: Solids, Liquids and Gases
Measurement Modes: Transmission, Reflection ATR, Polarization, Emission & Time Dependent.

Bench Accessories:
Transmission
- Solids Typical Sample Size 1cm, 1mm minimum (with masking)
- Liquds
  - Liquid Cell - Zinc Selenide Windows, Adjustable pathlength
  - Demountable Liquid Cell
  - Horizon - Horizontal Attenuated Total Reflecion Accessory
  - Long Pathlength Glass Cell
  - Cuvette Holder

- Gases
  - Gas Cell

Specular Reflection
- ERA 12 deg
- ERA 80 deg
- ERA 85 deg
- VMax 30-80 deg

Diffuse Reflection
- Praying Mantis

Attenuated Total Reflection (ATR)
- Horizon - Horizontal Attenuated Total Reflecion Accessory
- Grazing Angle ATR
- MIcro - ATR, Germanium Sampling Area: 25x25um typical
- 4X Beam Condensor Transmission & ATR

Continuum FTIR Microscope
Measurement Range: 12,500 cm-1 to 400cm-1
Samples: Solids, Liquids and Gases.
Sampling Area: Adjustable 100um to 20um.
Measurement Modes: Transmission, Reflection ATR, Polarization, Mapping, Emission & Time Dependent.
Visible and Infrared Polarizers
Microscope Slide On ATR
Diamond Compression Cell
Contact Alert System
Temperature Controlled Microscope Stage
Refrachromat IR Objective: 15X/0.58 N.A.

Emission Port
- Bench
- Microscope

External Detector - Allows remote positioning of detector.

Detector Material Characterization Accessory - with and without amplifier

Sample Preperation Accesories:
Pellet Press
Ball Mill