

FT-IR



1. Syllabus

10:00-11:30, 6 March

Practice 6,7,13,14,20,21 March

Test 27 or 28 March

The logo for Ulsan National Institute of Science and Technology (UNIST). It features the acronym 'UNIST' in a bold, blue, sans-serif font. The letters are composed of horizontal segments, giving it a digital or technological appearance. The background of the slide is a dark blue with abstract, glowing patterns of dots and lines, resembling a network or data visualization. In the bottom right corner, there is a circular emblem with a geometric, crystalline design.

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Qualification for FT-IR operation

1. FT-IR self-user training

- 1) Theory class (FT-IR manager Seonhye Son, 4175)
- 2) Operation class (FT-IR manager Seonhye Son, 4175)
 - 10:00-12:00 A.M : Manager explains about FT-IR
 - 13:30-16:30 P.M : Each person practices with manager

2. Practice FT-IR yourself

- Each person practice with manager 3 times.
- Please contact manager and make an appointment.

3. Attend the FT-IR test

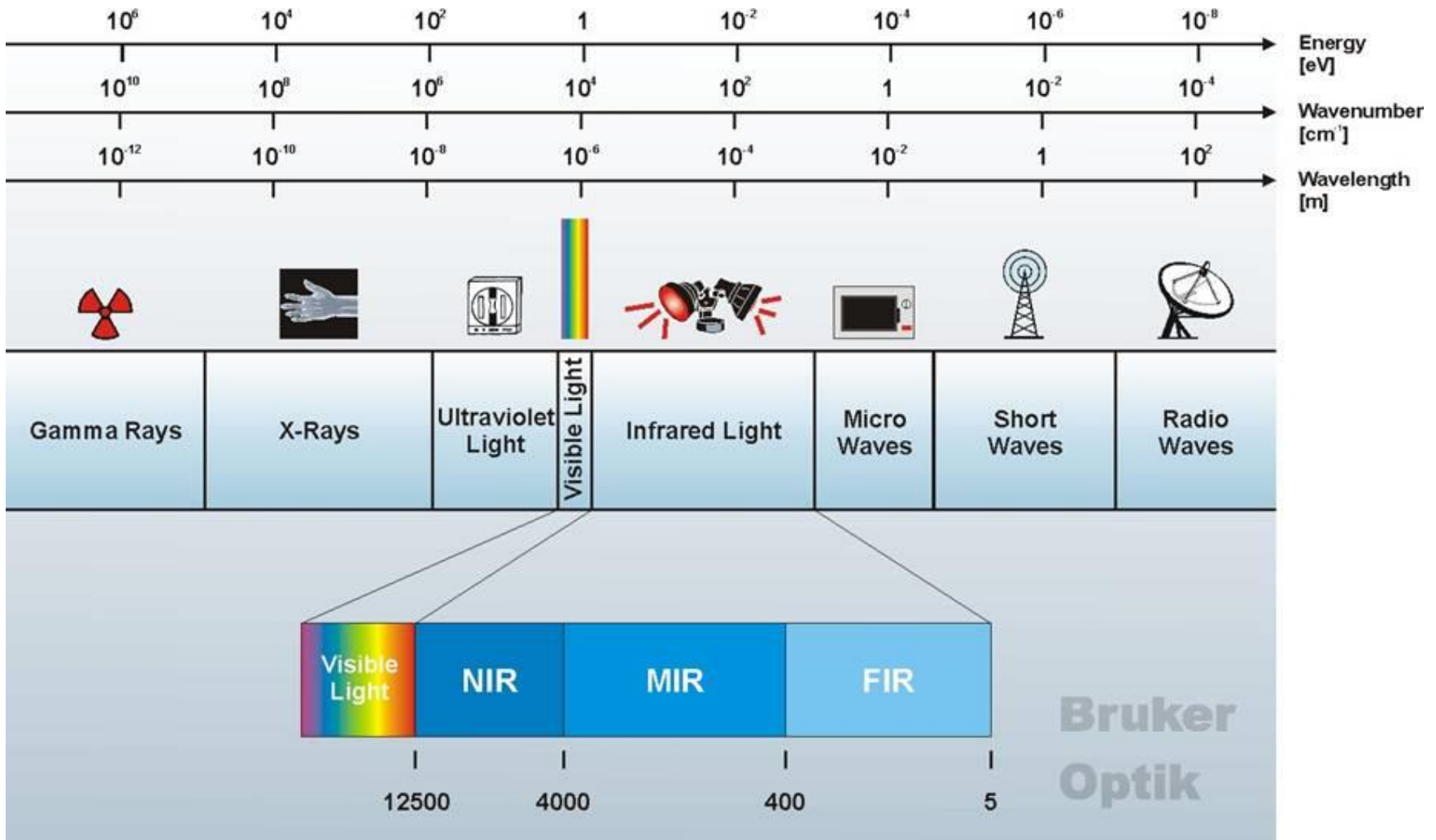
- 20 min.test
- Explain about IR and measurement methods.
- Sample measurement with ATR or other accessories.

2. Basic Principles

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Electromagnetic radiation



NIR : 12500 – 4000 cm^{-1} (0.8 – 2.5 μm , 1.55 – 0.5 eV)

- Overtones and combination vibrations
- low absorption coefficient \Leftrightarrow high sample concentrations
- Advantage : Quartz is transparent \rightarrow fiber optics, in glass vials
- Source : tungsten lamp
- Optical material : Quartz
- Detector : Ge, InGaAs

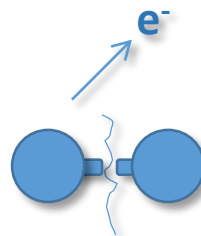
MIR : 4000 – 400 cm^{-1} (2.5 – 25 μm , 0.5 – 0.05 eV)

- Fundamental molecular vibrations : stretch and deformation vibrations
- high absorption coefficient \Leftrightarrow low sample concentrations
- Source : Globar
- Optical material : KBr, ZnSe
- Detector : DTGS, MCT

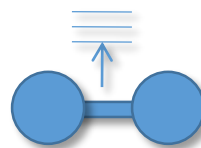
FIR : 400 – 5 cm^{-1} (25 – 1000 μm , 0.05 – 0.0012 eV)

- Backbone vibration of large molecules, molecules with heavy atoms
- low absorption coefficient, strong water vapor absorption \rightarrow vacuum spectrometer
- Source : Globar, Hg lamp
- Optical material : PE, CsI
- Detector : DTGS, Bolometer

Bond breaking and ionization



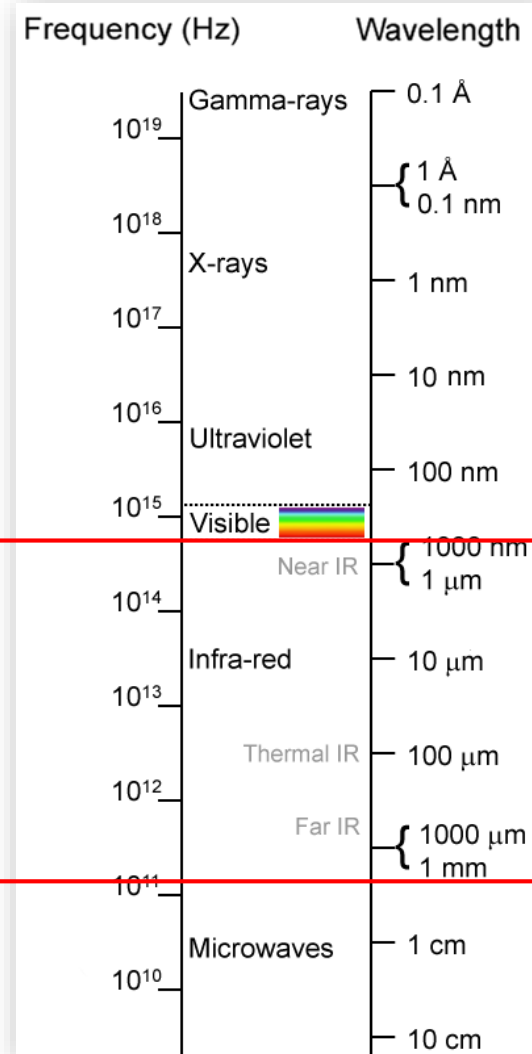
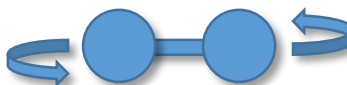
Electronic excitation



Vibration

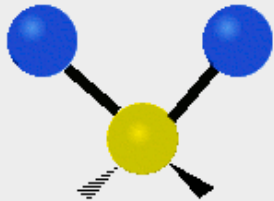


Rotation

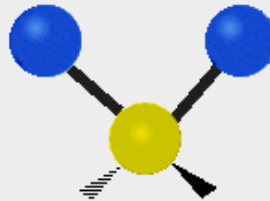


<http://upload.wikimedia.org/wikipedia/en/8/8a/Electromagnetic-Spectrum.png>

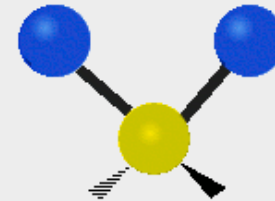
Symmetric stretching



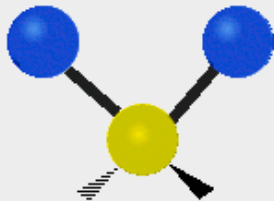
Asymmetric stretching



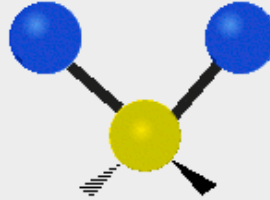
Scissoring



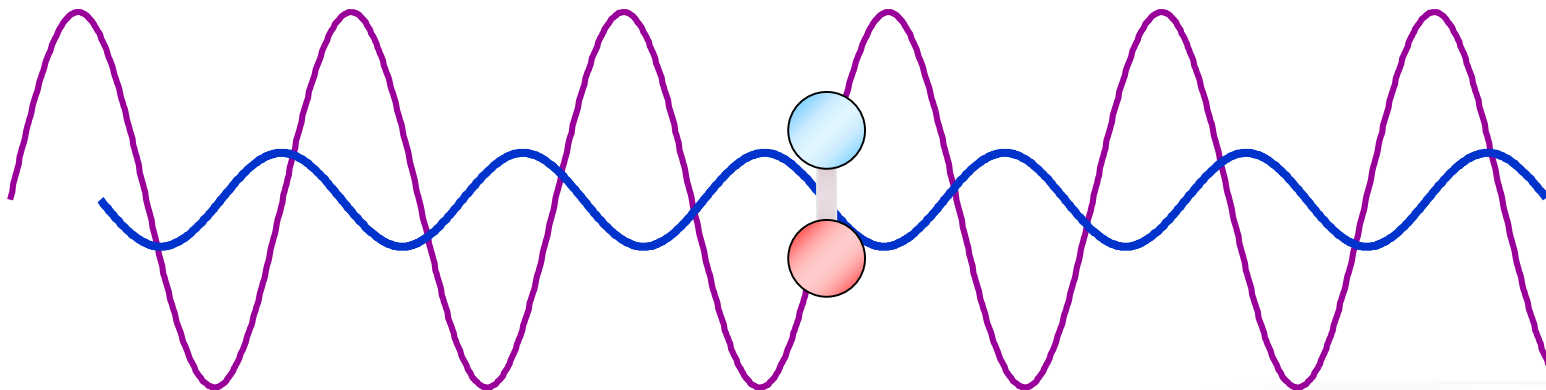
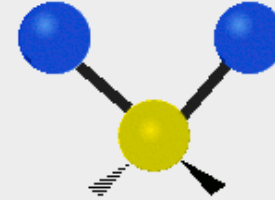
Rocking



Wagging



Twisting



❖ Selection Rules

A molecule will absorb infrared radiation if the change in vibrational states is associated with a change in the **dipole moment (μ)** of the molecule.

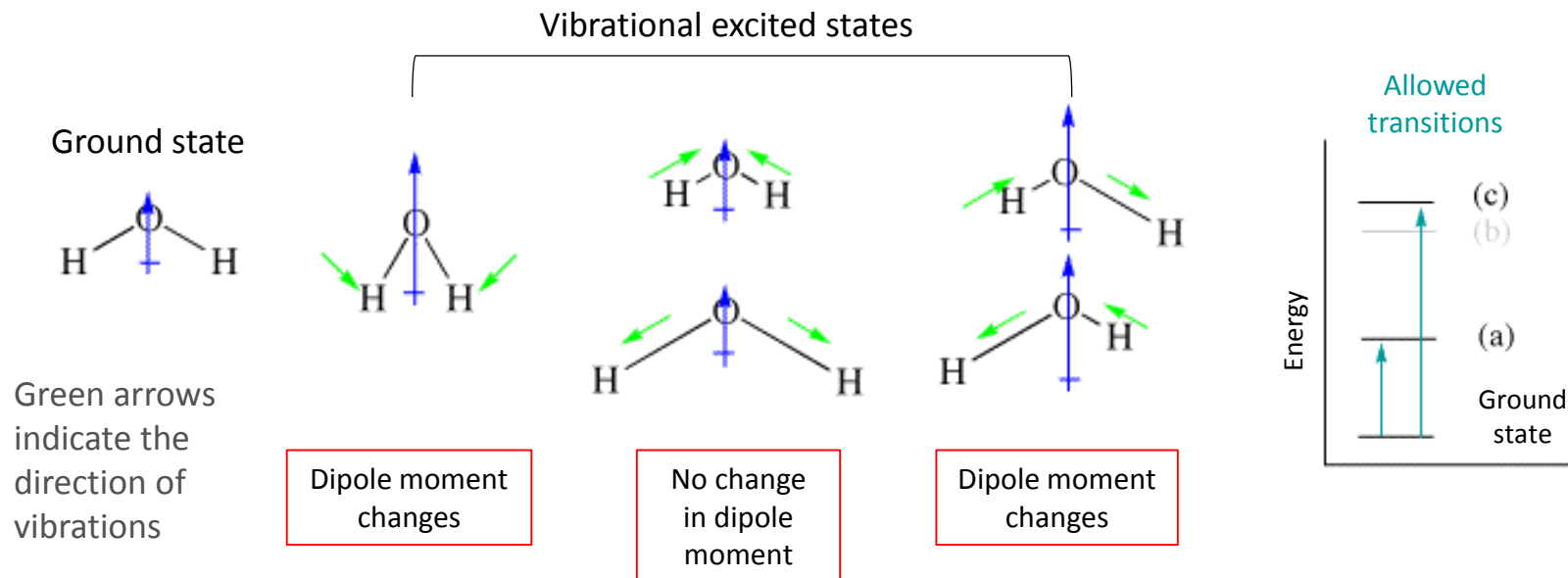
Dipole moment (μ)

A dipole moment is a quantity that describes two opposite charges separated by a distance.

$$\mu = q \times r$$

q: separated charge(positive and negative charge)

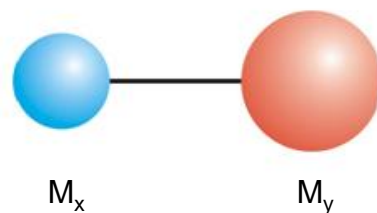
r: distance between center of charges



For a harmonic oscillator it is possible to calculate the vibrational frequency of a diatomic molecule as follows.

$$\nu = \frac{h}{2\pi c} \sqrt{\frac{k}{\mu}}$$

$$\mu = \frac{M_x \cdot M_y}{M_x + M_y}$$



ν = vibration frequency(cm^{-1})

c = vibration frequency(cm^{-1})

K = Force constant of bond(dynes/cm)

M_x and M_y = Mass of each

Single bond $K \cong 5 \times 10^5 \text{ dyne/cm}$

$c = 19.8 \times 10^{-24} g$, $H = 1.64 \times 10^{-24} g$

$\nu_{CH} = 3040 \text{ (obs)} 2960 - 2850$

Bonding	Force constant f (dyne/cm)	Absorbance range (cm^{-1})	
		Calculation	Measurement
C — O	5.0×10^5	1113	1300 — 800
C — C	4.5×10^5	1128	1300 — 800
C — N	4.9×10^5	1135	1250 — 1000
C = C	9.7×10^5	1657	1900 — 1500
C = O	12.1×10^5	1731	1850 — 1600
C \equiv C	15.6×10^5	2101	2150 — 2100
C — D	5.0×10^5	2225	2250 — 2080
C — H	5.0×10^5	3032	3000 — 2850
O — H	7.0×10^5	3553	3800 — 2700

C = C bond

$$\bar{\nu} = 4.12 \sqrt{\frac{K}{M_x M_y / M_x + M_y}}$$

$$K = 10 \times 10^5 \text{ dynes/cm}$$

$$\frac{M_x M_y}{M_x + M_y} = \frac{(12)(12)}{12 + 12} = 6$$

$$\bar{\nu} = 4.12 \sqrt{\frac{10 \times 10^5}{6}} = 1682 \text{ cm}^{-1} (\text{calculated})$$

$$\bar{\nu} = 1650 \text{ cm}^{-1} (\text{experimental})$$

C – H bond

$$\bar{\nu} = 4.12 \sqrt{\frac{K}{M_x M_y / M_x + M_y}}$$

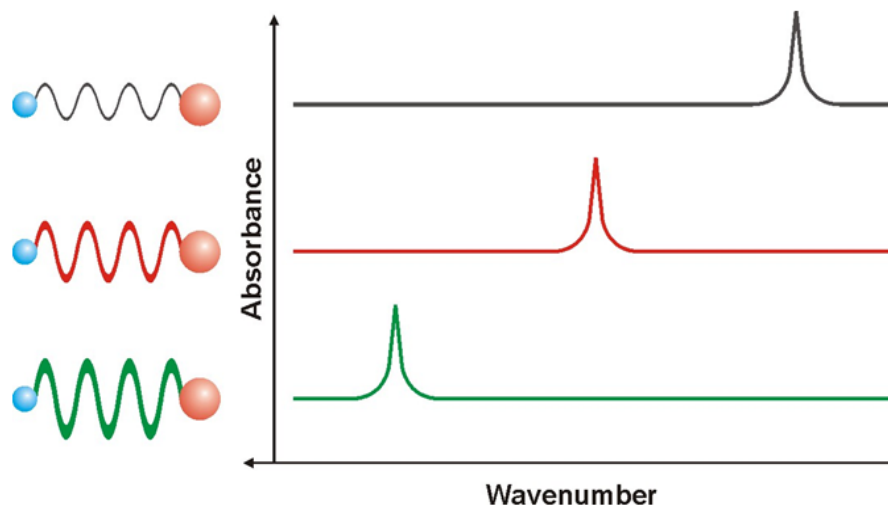
$$K = 5.0 \times 10^5 \text{ dynes/cm}$$

$$\frac{M_x M_y}{M_x + M_y} = \frac{(12)(1)}{12 + 1} = 0.923$$

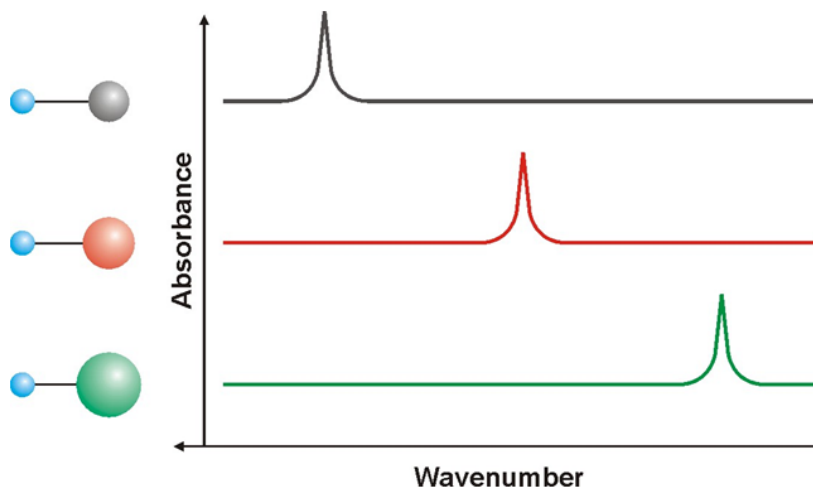
$$\bar{\nu} = 4.12 \sqrt{\frac{5.0 \times 10^5}{0.923}} = 3032 \text{ cm}^{-1} (\text{calculated})$$

$$\bar{\nu} = 3000 \text{ cm}^{-1} (\text{experimental})$$

1) The higher the force constant (k , the bond strength), the higher the vibrational frequency (wavenumber).



2) The larger the vibrating atomic mass, the lower the vibrational frequency.



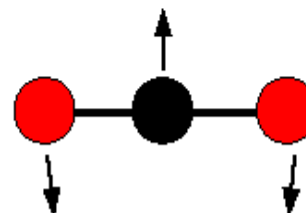
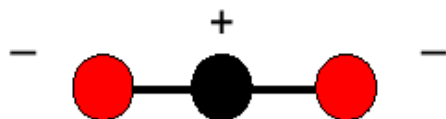
Example: CO₂

symmetrical stretching
1340 cm⁻¹

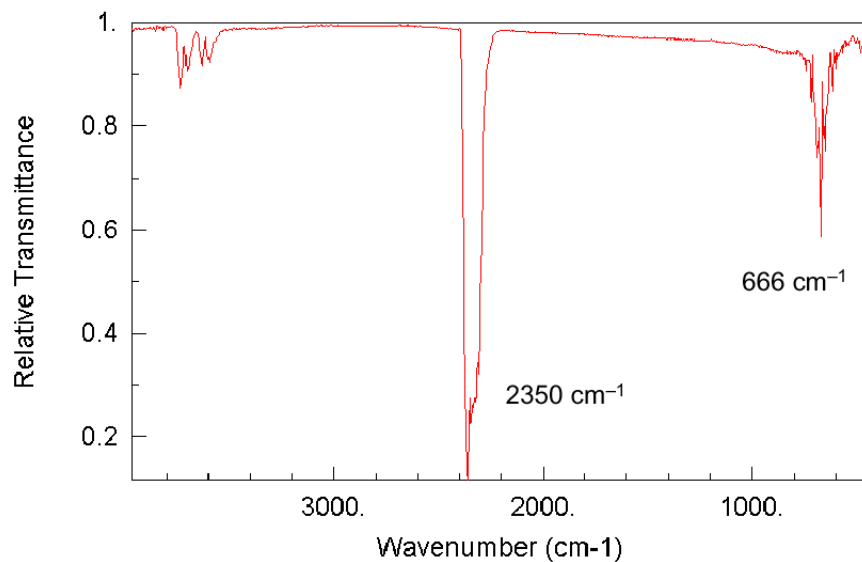


asymmetrical stretching
2350 cm⁻¹

scissoring bending
666 cm⁻¹



scissoring bending
666 cm⁻¹



The theoretical number of fundamental vibrations (absorption frequencies) will seldom be observed.

The overtones (multiples of a given frequency), combination (sum of two other vibrations) or difference (the difference of two other vibrations) tones increase the number of bands.

3. Hardware

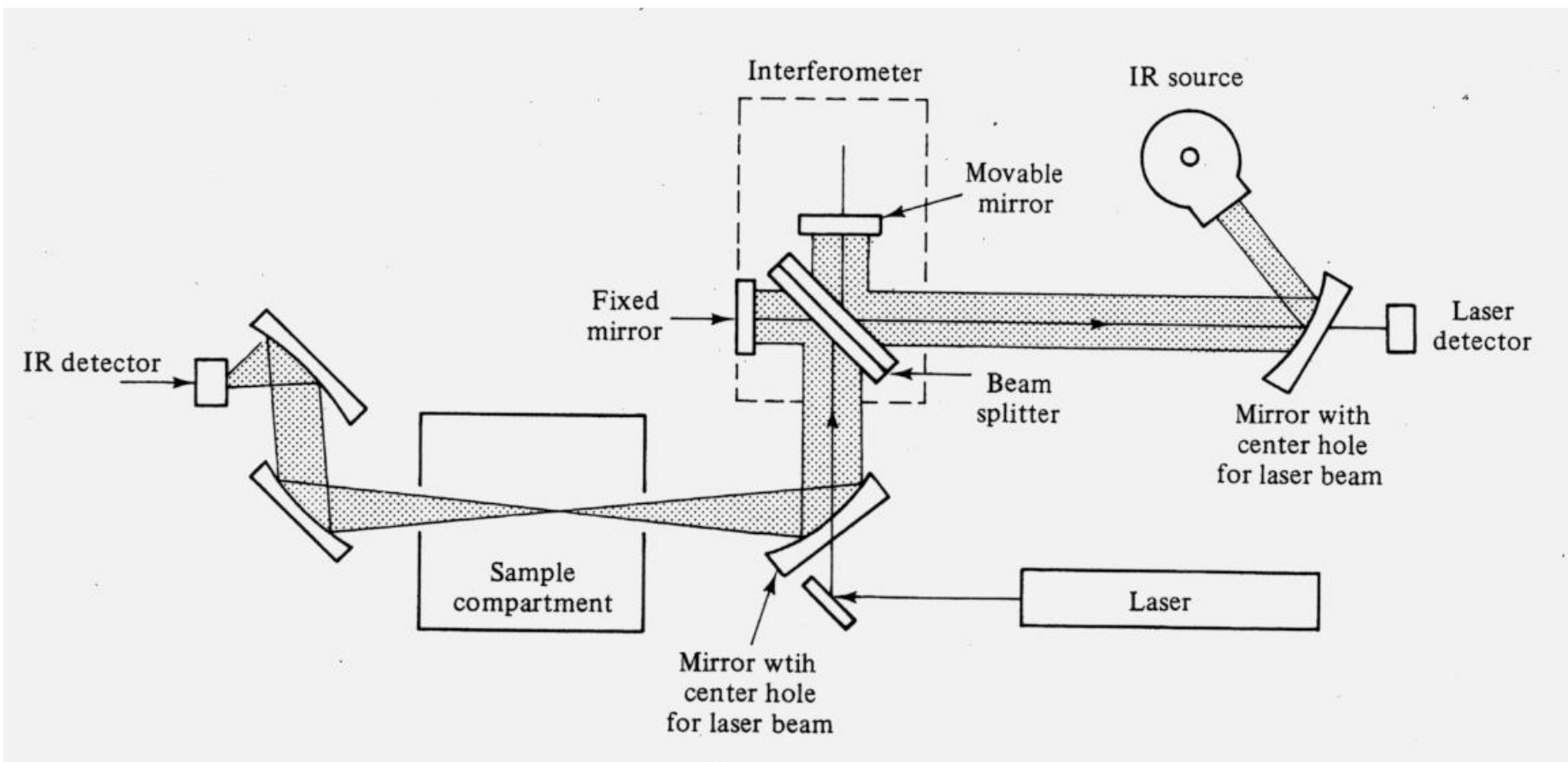
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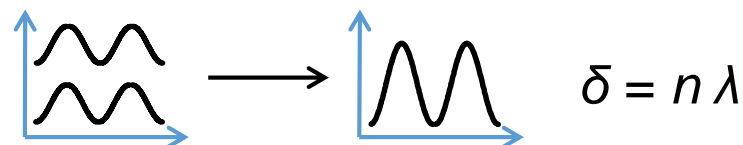
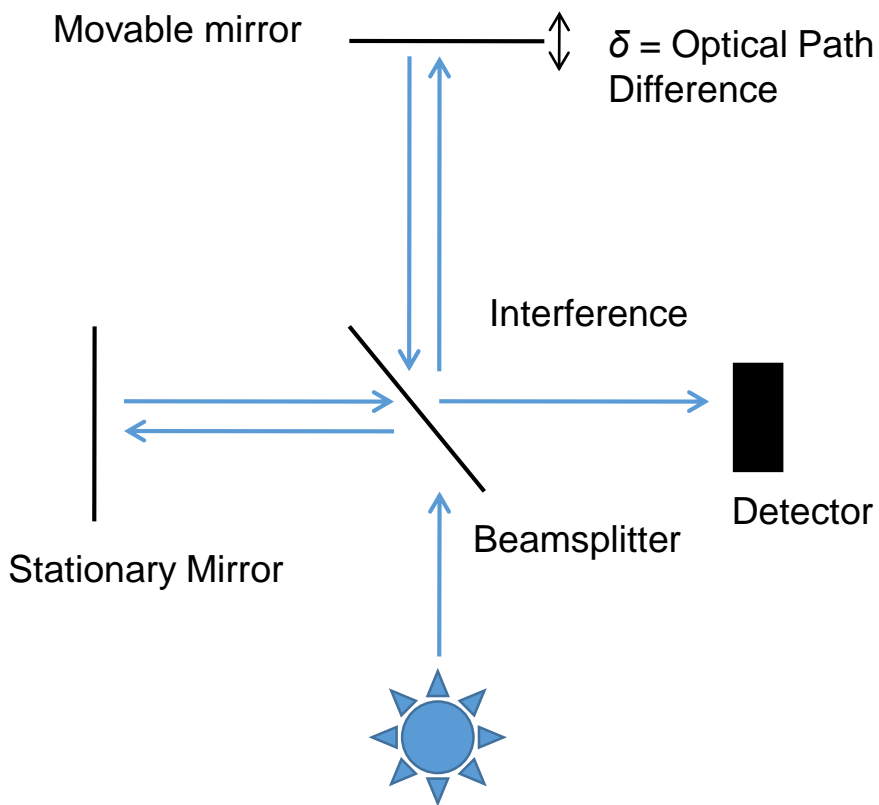
Fourier Transform Infrared Spectrometer (FT-IR)

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Single-beam FTIR Spectrometer



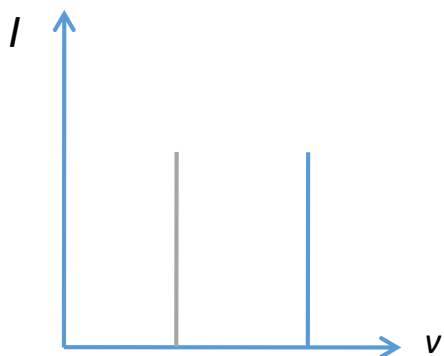
Michelson interferometer



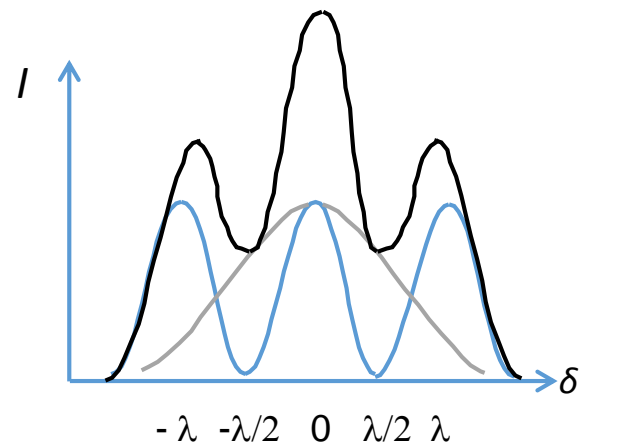
Fourier Transform Infrared Spectrometer (FT-IR)

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Dichromatic source

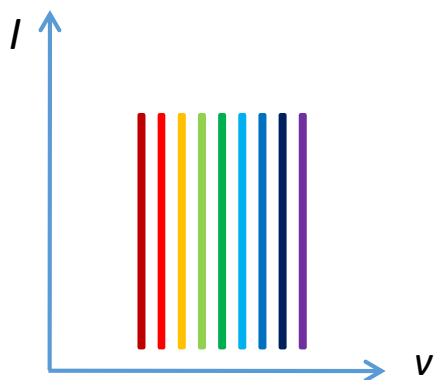


Interferogram

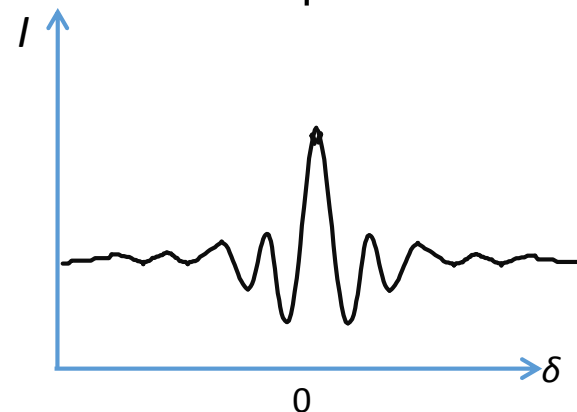


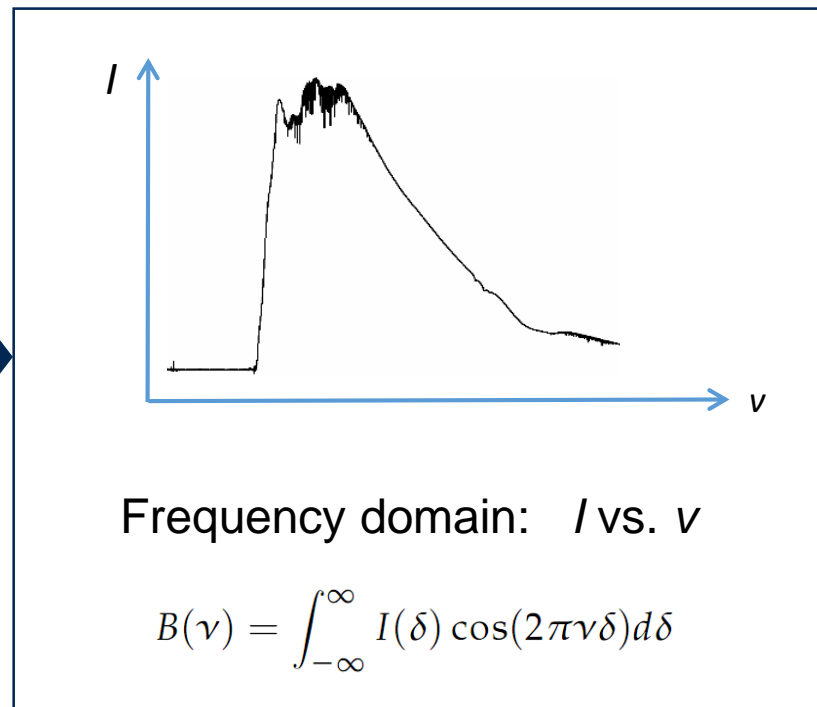
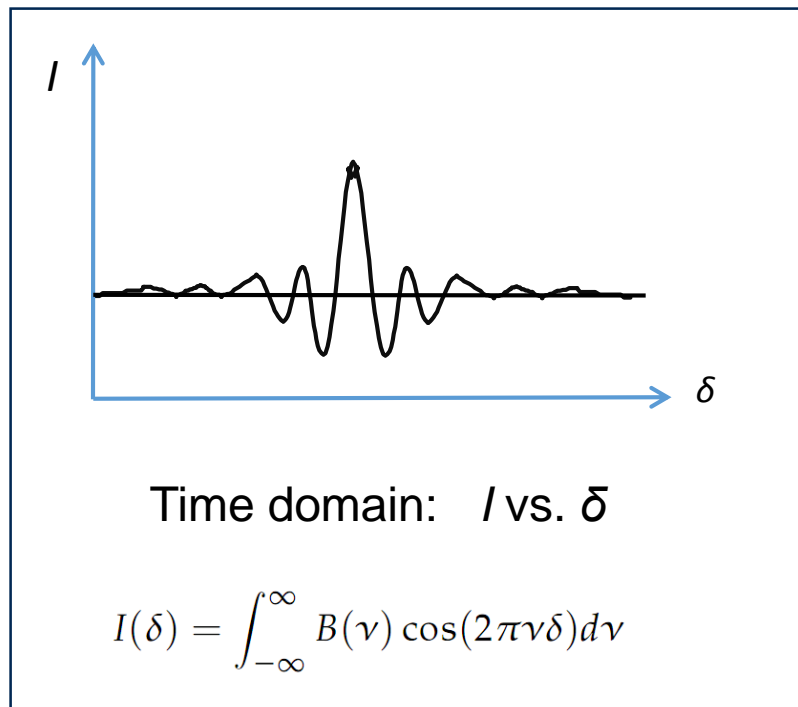
Moveable mirror

Continuous source



Continuous IR spectrum





❖ Advantages of FT-IR

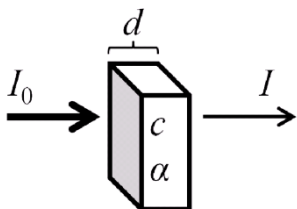
Throughput Advantage_Circular aperture, high signal intensity → high signal to noise ratio

Multiplex Advantage_All frequencies are measured at the same time

Precision Advantage_Internal laser control the scanner – built in calibration

y axis is %T or A

x axis is wavenumber (or wavelength)



$$T = I/I_0$$

$$\%T = 100 I/I_0$$

T transmission /
transmittance

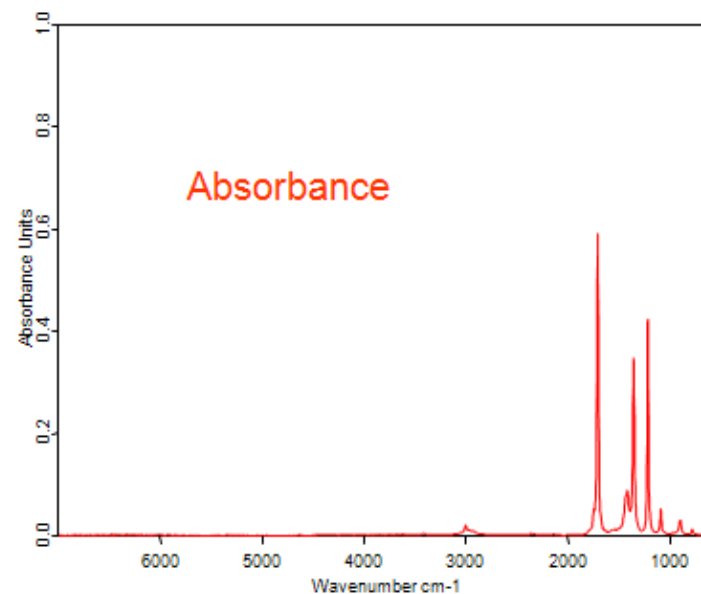
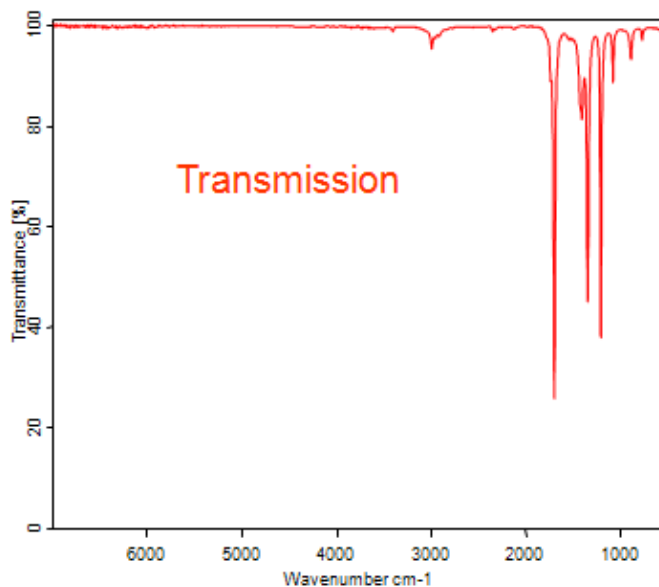
$$A = -\log T$$

$$A \text{ absorbance (no units)} = c d \alpha$$

(Note A (but not T) \propto concentration) d = sample thickness

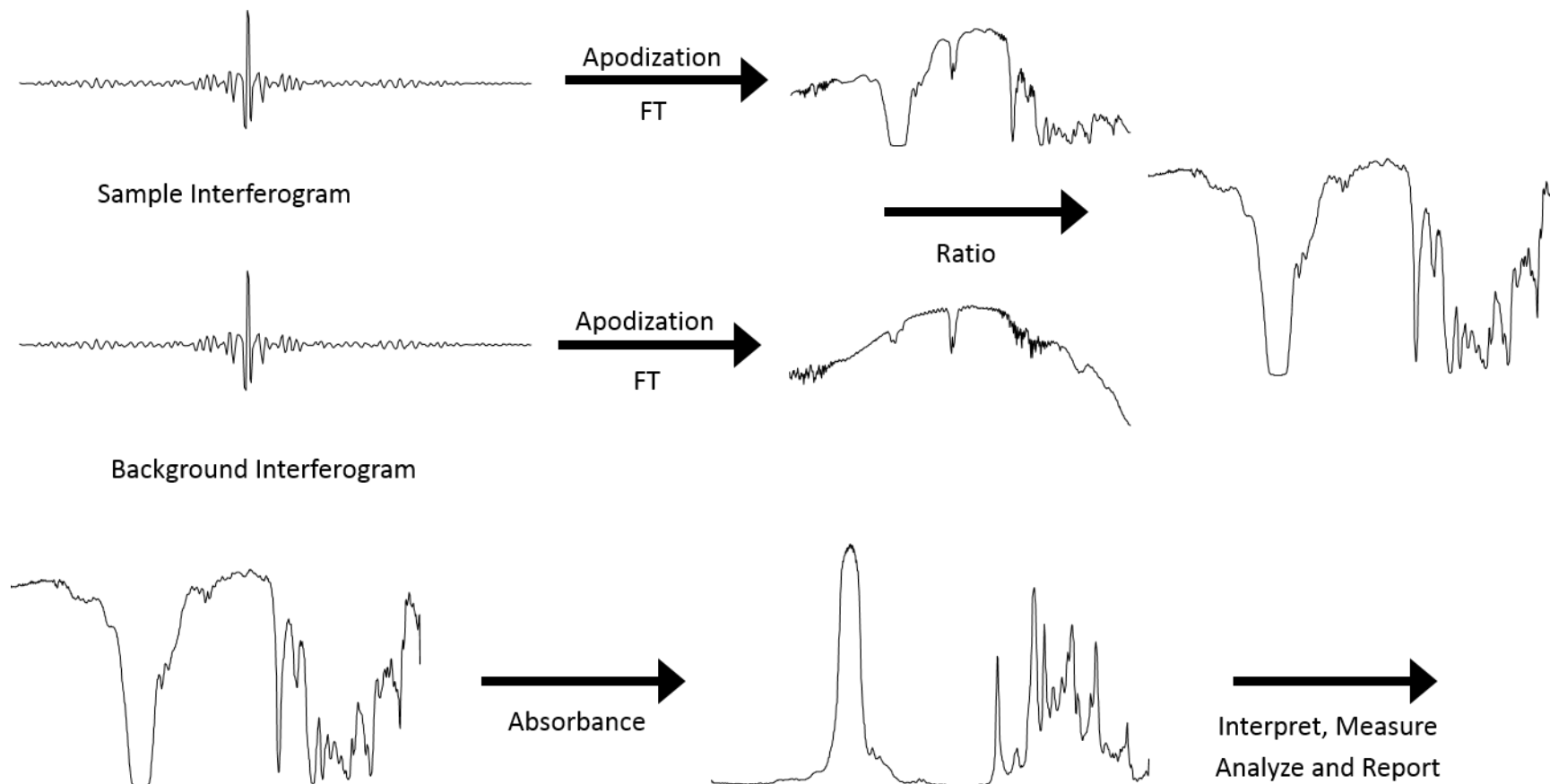
c = absorbant concentration

α = absorption coefficient

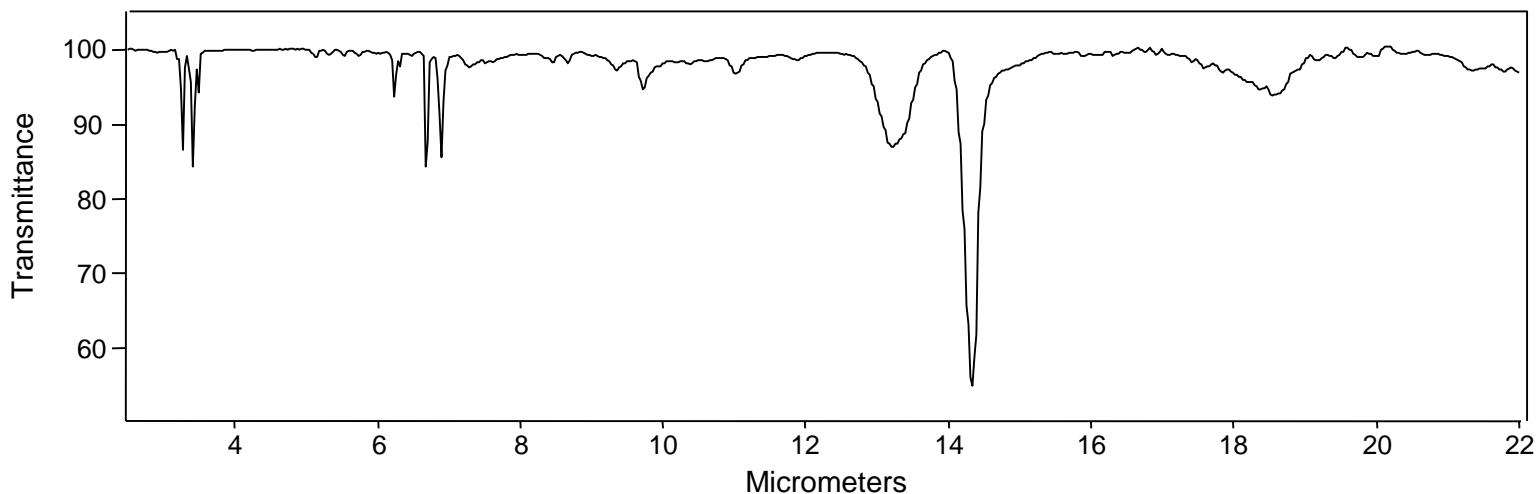


❖ Fourier Transformation)

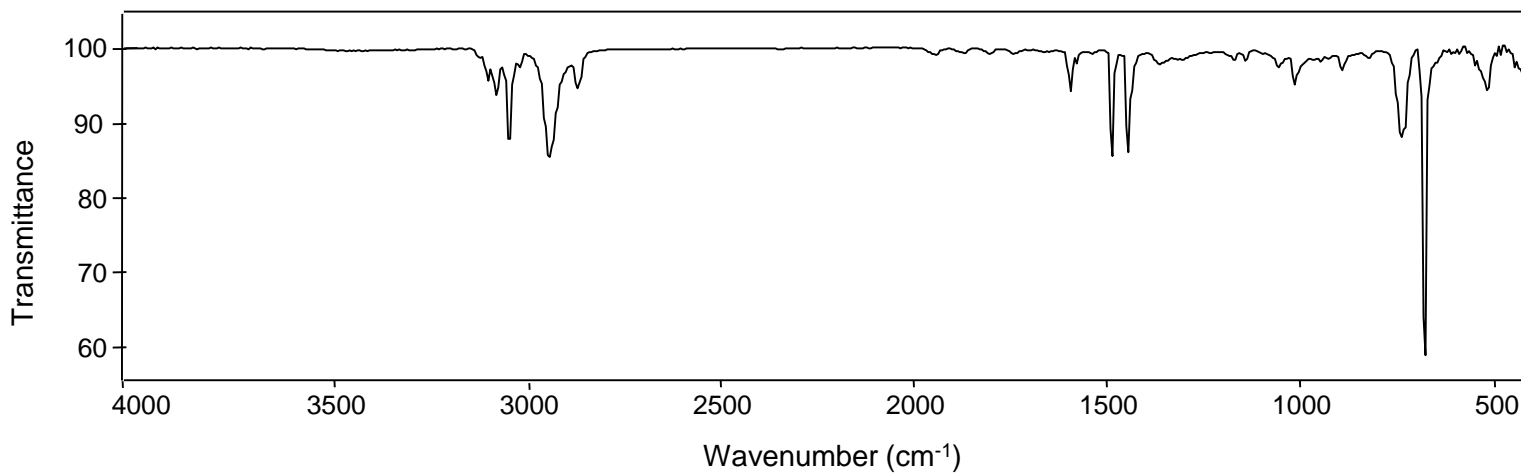
$$E_{\text{sample}} / E_{\text{background}} \times 100 = \% \text{Transmittance}$$



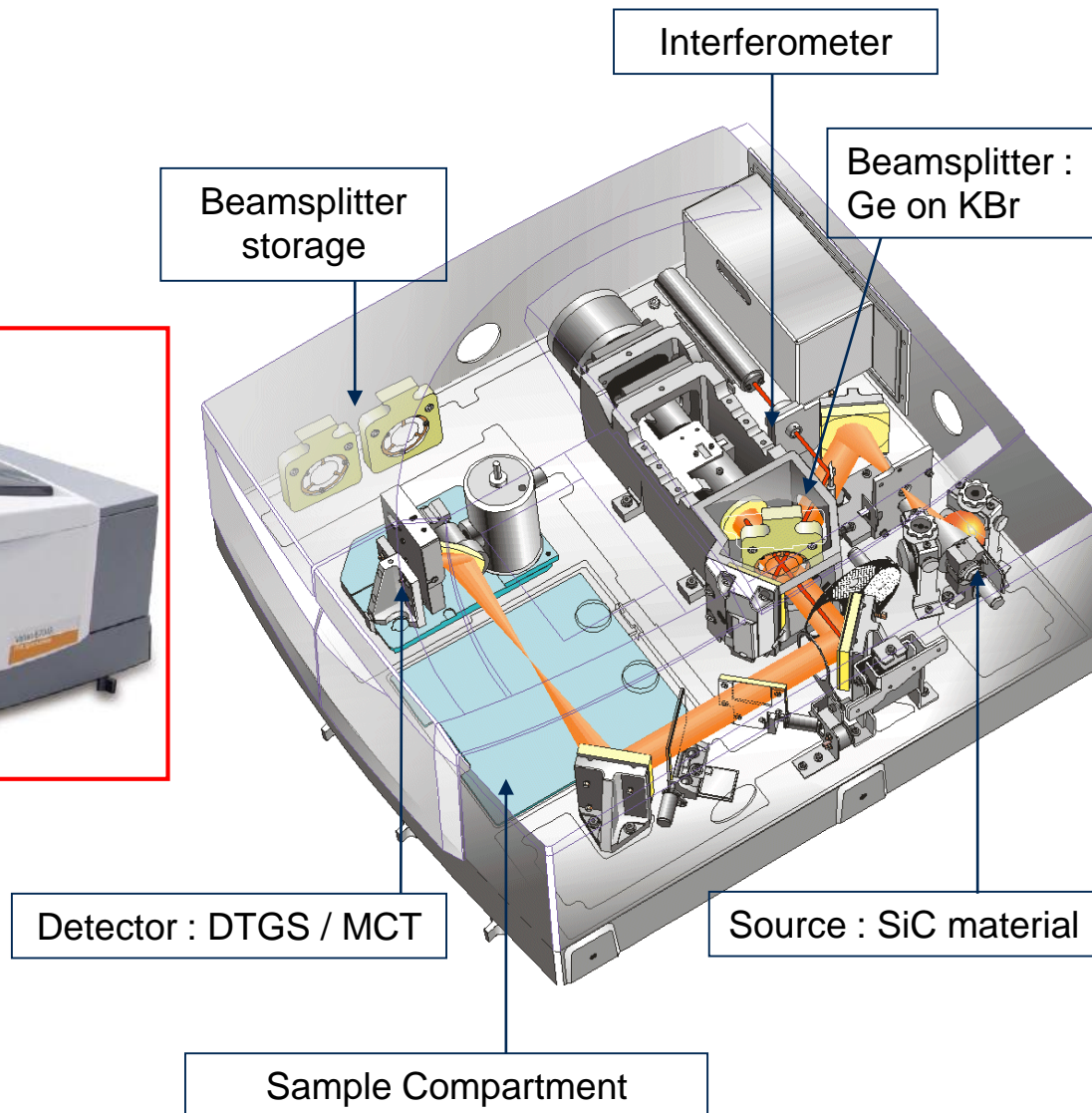
IR Spectrum in Wavelength (μm)



IR Spectrum in Wavenumber (cm^{-1})



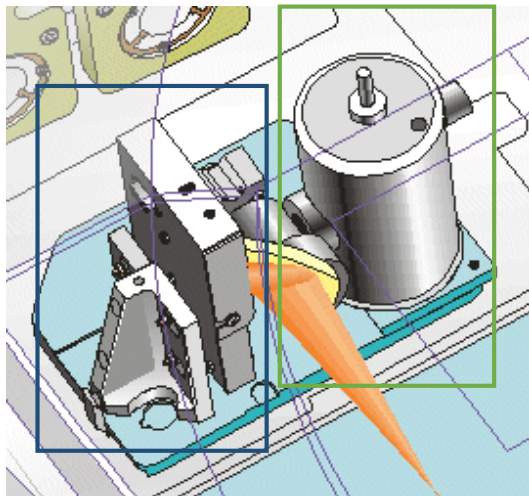
670 FT-IR Systems



❖ Source

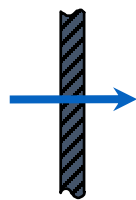
Nernst Glower	heated rare earth oxide rod (~1500 K)	1-50 μm (mid- to far-IR)
Globar	heated SiC rod (~1500 K)	1-50 μm (mid- to far-IR)
W filament lamp	1100 K	0.78-2.5 μm (Near-IR)
Hg arc lamp	plasma	50 - 300 μm (far-IR)
CO2 laser	stimulated emission lines	9-11 μm

❖ Detector

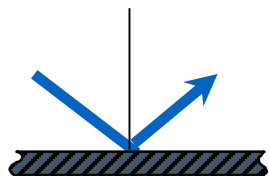


- **DLaTGS (Deuterated, L-alanine doped TriGlycine Sulfate)**
Pyroelectric detector (mid IR)
DLaTGS detector provides linear response over a very wide range of FT-IR throughput, which is beneficial in qualitative and quantitative FT-IR sampling.

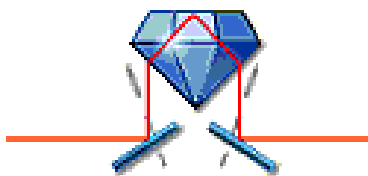
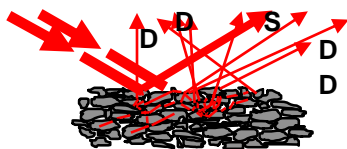
- **MCT (Mercuric Cadmium Telluride)**
Quantum detector
High MCT sensitivity will produce a large signal in a low-flux measurement. It demonstrates a relatively constant signal versus data-collection speed.



$I^0=0^\circ$



$I^0=R^0$



Transmission

(Powder, Drugs-KBr pellet/Films, Coatings, Paints-film holder/Liquid-window cell)

Absolute reference measurement

Sample preparation can be difficult and time consuming

Specular Reflectance

Sample must have a back reflective layer or must be on a mirror
(layer thickness = single molecule)

Diffuse Reflectance

Solids and powders, diluted in a matrix of KBr or KCl

Analysis of non-reflective materials

ATR (Attenuated Total Reflectance)

(Powder, Drugs, Films, Coatings, Paints, Liquid, Rubber)

The infrared beam is focused into a crystal

Creating an evanescent wave about 1 – 2 microns deep

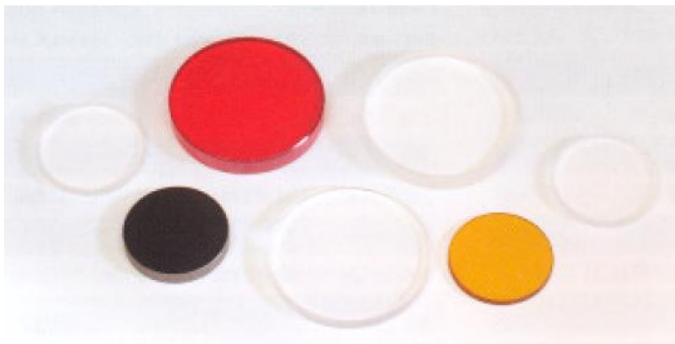
No sample preparation

Transmission_Powder, Drugs, Film

Pellet sample (13 mm die) and film sample



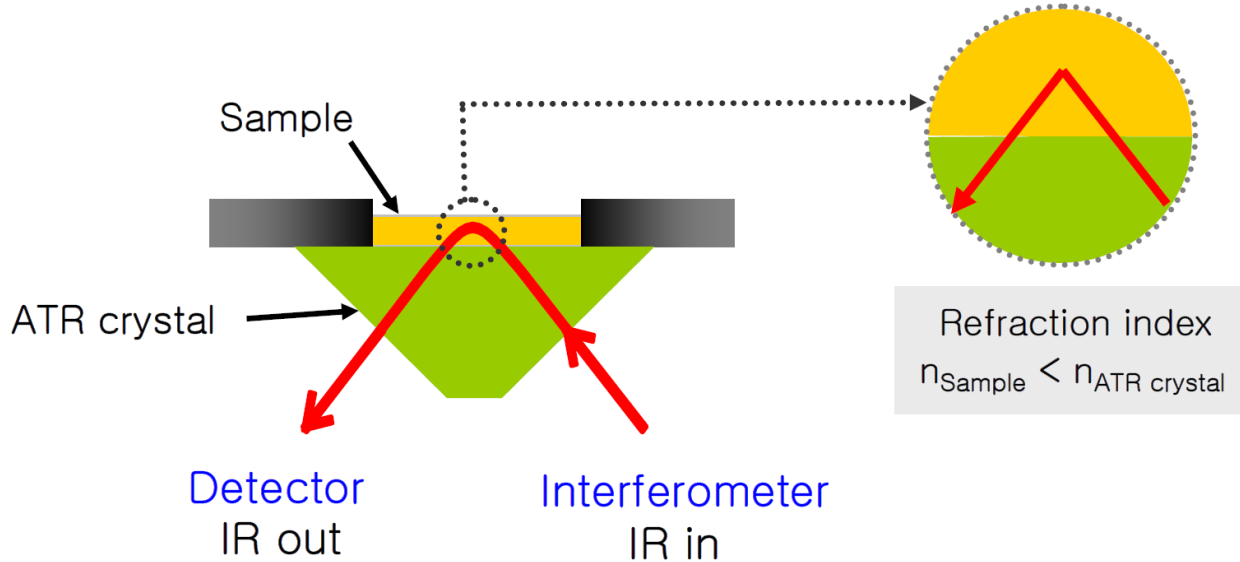
Transmission_Liquid sample (window material)



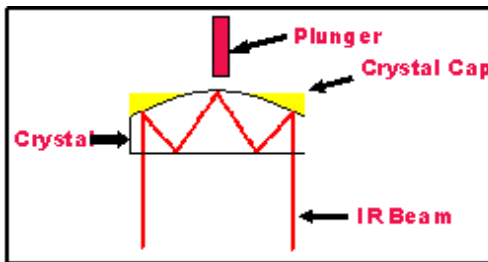
Type of window materials for liquid sample

Material	Comments	Max. Temp. in air (°C)	Transmission Range (cm ⁻¹)	pH Range	Solvents which attack material
AgCl	Silver Chloride	200	25000-360	N/A	Complexing agents
Al ₂ O ₃	Sapphire	1700	20000-1780	1-14	acids, alkalis
AMTIR	SeAsGe glass, brittle	300	1100-593	1-9	alkalis
BaF ₂	Barium Fluoride	500	65000-700	5-8	NH ₄ ⁺ , salts, acids
CaF ₂	Calcium Fluoride	900	70000-1100	1-9	NH ₄ ⁺ , salts, acids
CsI	Cesium Iodide	200	40000-200	N/A	Lower alcohols "wet" solvents
Diamond	Diamond	750	40000-2500 & 1667-33	1-14	K ₂ Cr ₂ O ₇ , H ₂ SO ₄
Ge	Germanium	270	5500-625	1-14	H ₂ SO ₄ aqua regia
KBr	Potassium Bromide	300	40000-400	N/A	Lower alcohols "wet" solvents
KRS-5	Thallium Bromide/Thallium Iodide, extremely toxic!	200	17900-204	5-8	Complexing agents
NaCl	Sodium Chloride	400	40000-625	N/A	Lower alcohols "wet" solvents
Si	Silicon, strong IR absorbance between 624-590 cm ⁻¹	300	8900-624	1-12	HF, HNO ₃
SiO ₂	Silicon Dioxide (Quartz)	1200	40000-2500	1-14	HF, some hot acids and bases
ZnS	Zinc Sulfide	300	17000-690	5-12	acids
ZnSe	Zinc Selenide	300	20000-454	5-9	Acid, strong alkalis

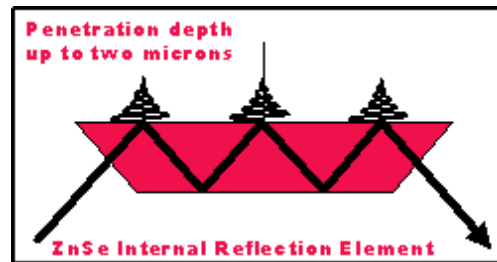
ATR (Attenuated Total Reflectance)



< ATR system >



Single-bounce ATR



Multi-bounce ATR



ATR (Attenuated Total Reflectance)

➤ 감쇠 전반사 과정

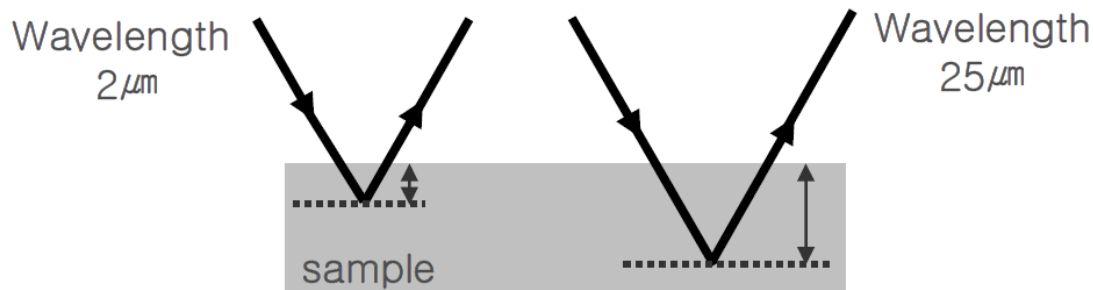
- ① 적외선 복사선이 굴절률이 큰 crystal에서 굴절률이 작은 시료로 통과할 때 내부 반사에 의해 소멸파(evanescent wave) 발생
- ① 소멸파는 시료의 수 μm 내로 침투
- ② 시료에 따라 특정 파장의 소멸파를 흡수하면 선택적으로 에너지 감소
- ③ 감소한 에너지 파장과 원래의 파장 비교
- ④ 입사 에너지의 반사된 분율이나 반사도를 파장이나 파수에 대해 도식하여 스펙트럼 작성

ATR (Attenuated Total Reflectance)

➤ ATR 스펙트럼의 특징

- ① 흡수 정도는 입사되는 파장, 두 물질의 굴절률, 입사각에 의존하며 시료 두께와 무관
Depth of penetration (dp) of the IR beam into the sample

$$d_p = \frac{\lambda}{2\pi(n_1^2 \sin^2 \theta - n_2^2)^{1/2}}$$

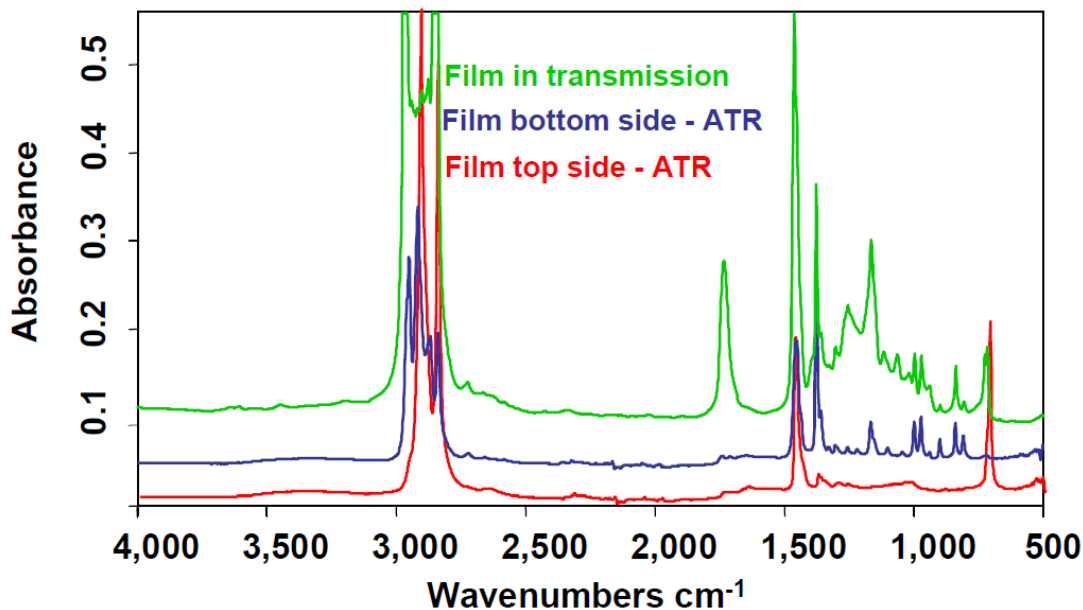


Crystal	Refraction index at 1,000cm ⁻¹	Depth of penetration at 45°	Depth of penetration at 60°
Diamond	2.4	1.66	1.04
Si	3.4	0.81	0.61
Ge	4.0	0.65	0.5

ATR (Attenuated Total Reflectance)

➤ ATR 스펙트럼의 특징

- ② IR 흡수 스펙트럼과 같은 위치에서 피크가 관찰되지만 상대적 세기가 다름
- ③ 소멸파의 침투깊이는 $0.5 \sim 2.0 \mu\text{m}$ 이므로 표면에 대한 정보 제공



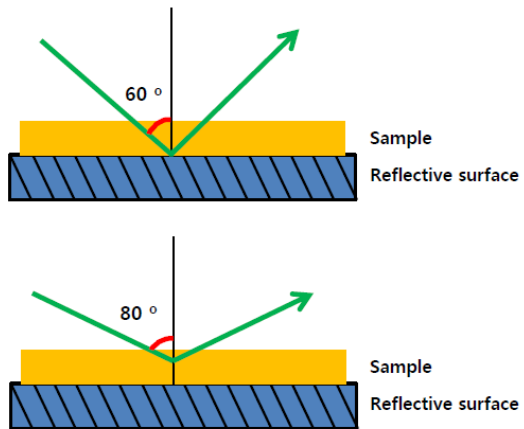
<세 층으로 구성된 고분자 film의 분석 결과>

ATR crystal

Material	ATR Spectral Range (cm ⁻¹)	Refractive Index	Depth of Penetration (μ) (at 45° & 1000 cm ⁻¹)	Uses
Germanium	5,500 - 675	4	0.66	Good for most samples, especially strong absorbing samples, such as dark polymers
Silicon	8,900 - 1,500 & 360-120	3.4	0.85	Resistant to basic solutions
AMTIR	11,000 - 725	2.5	1.77	Very resistant to acidic solutions
ZnSe	15,000 - 650	2.4	2.01	General use
Diamond	25,000 - 100	2.4	2.01	Good for most samples. Extremely caustic or hard samples

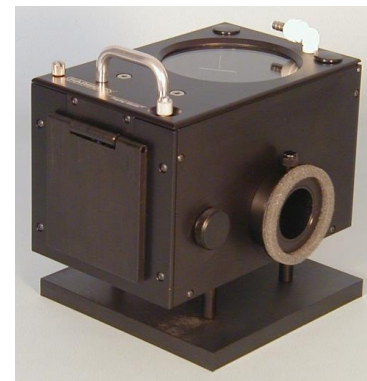
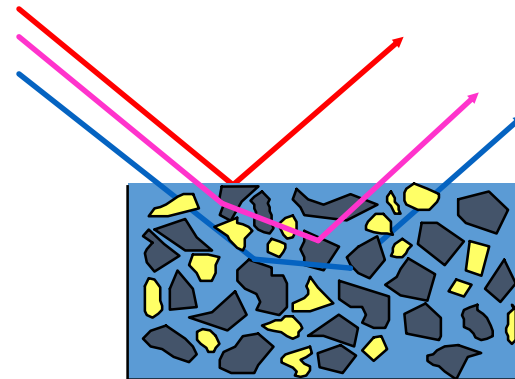
Reflectance method

Specular reflectance



Seagull

Diffuse reflectance (DRIFTS)



Praying Mantis

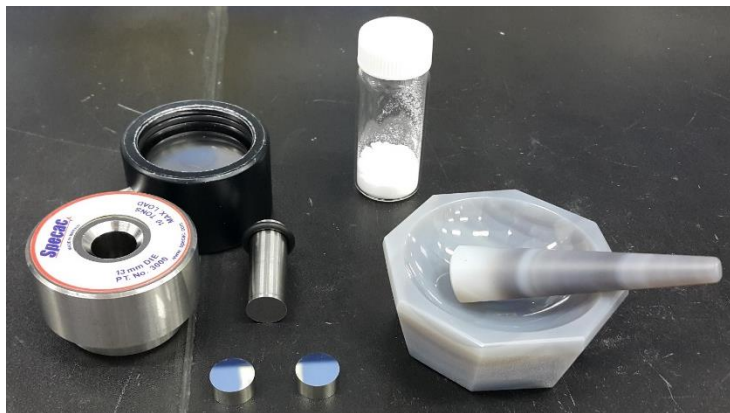
4. Pre-treatment

- 시료 준비
- 전처리

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Preparing KBr Pellets



Step 1. The powder sample and KBr must be ground to reduce the particle size to less than 5 μm in diameter. Otherwise, large particles scatter the infrared beam and cause a slope baseline of spectrum.

Step 2. Take two stainless steel disks. Place a piece of the precut cardboard on top of one disk and fill the cutout hole with the finely ground mixture.

Put the second stainless steel disk on top.
(Fill the ground sample on more smooth side of disk.)



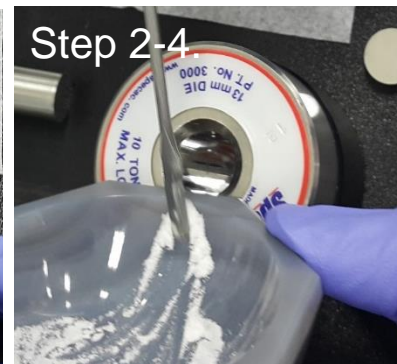
Step 1.



Step 2-1.



Step 2-2.



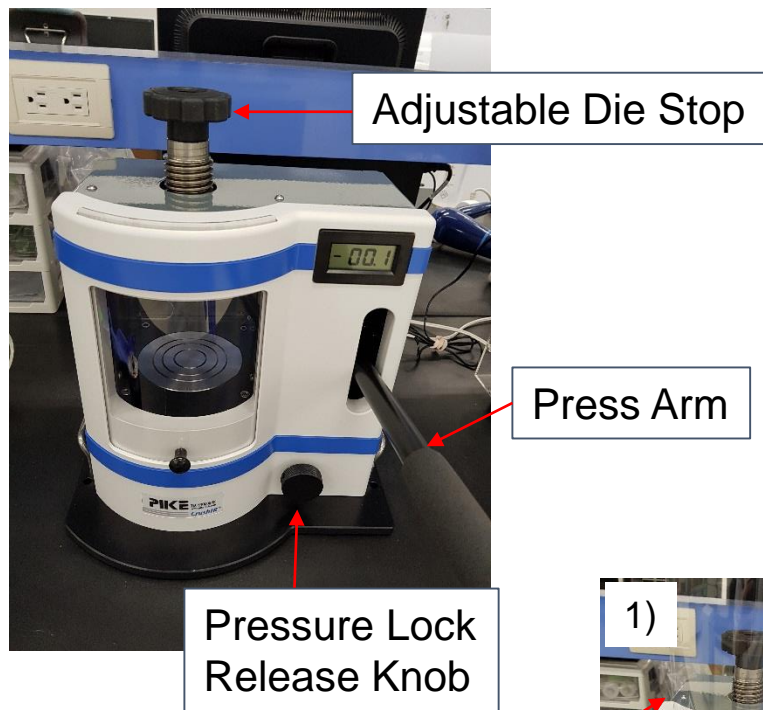
Step 2-4.



Step 2-4.

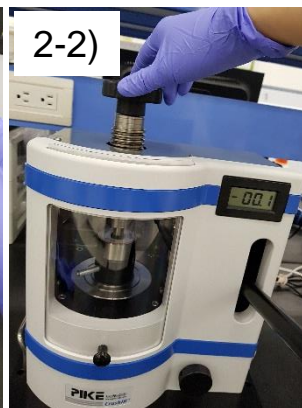
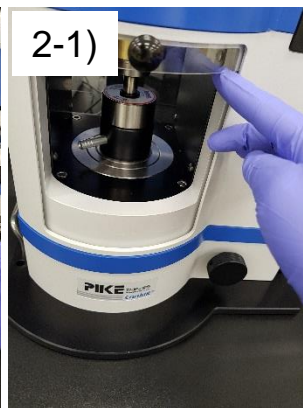
Preparing KBr Pellets_Pressing pellet

Step 3. Pressing pellet



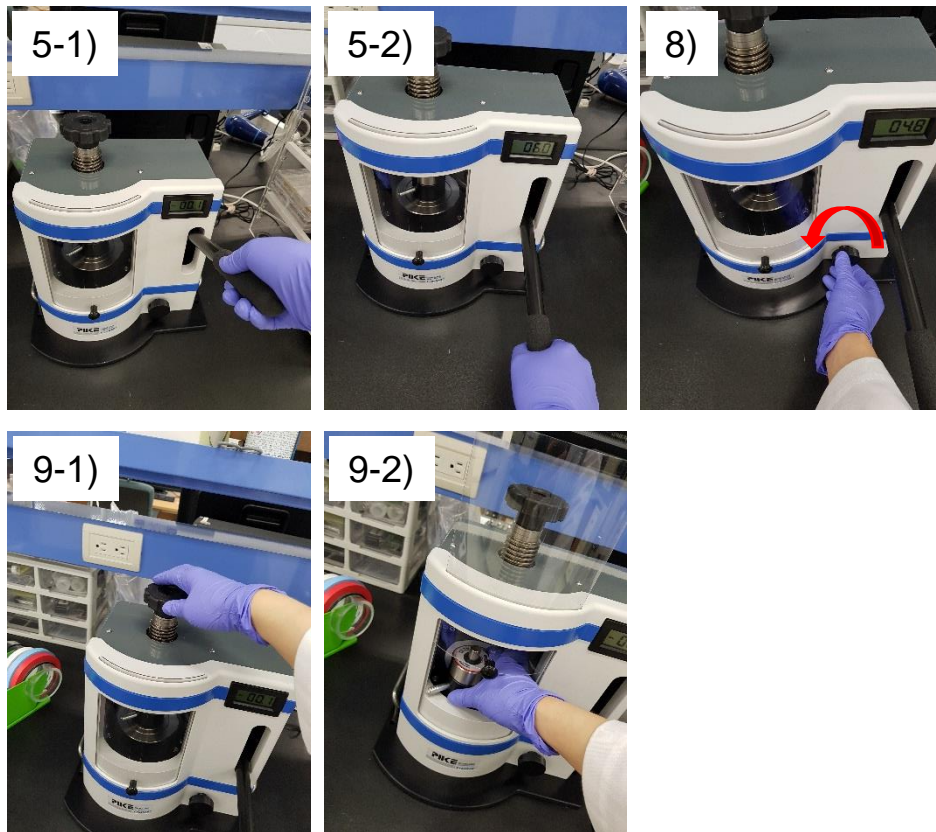
- 1) Place your pellet press into the CrushIR adjusting the die stop as needed to provide a comfortable amount of clearance.
- 2) Position the pellet press so that it's top and bottom are centered relative to the die stop and the hydraulic ram.
- 3) Close the protective shield.
- 4) Turn the pressure lock/release knob fully clockwise to close.

Protective shield



Preparing KBr Pellets_Pressing pellet

Step 3. Pressing pellet



- 5) Apply force to the pellet press by pumping the press arm up and down.
- 6) Continue applying force to the pellet press until you reach its maximum rating.

In the case of the PIKE Evacuatable Pellet Press, the maximum load is 10 US tons. **WARNING - Never exceed the maximum force rating of the pellet press.**

- 7) Allow the force to stay at the maximum value for about 1 minute.
- 8) Then release the force by turning the pressure lock/release knob counterclockwise.
- 9) Open the protective shield and remove the pellet press to extract the pellet.

NOTE: KBR POWDER IS SOFT AND COMPRESSIBLE. WHEN MAXIMUM FORCE IS APPLIED TO THE POWDER IT IS NORMAL FOR THE DIGITAL READING TO DROP INITIALLY BEFORE IT SETTLES AND REACHES A STEADY-STATE READING.

Transmission_powder sample

Preparing KBr Pellets_Pressing pellet

Step 4. Disassemble the die set and put the pellet onto the sample holder.

Step 5. Please clean the die after you finish your experiment.

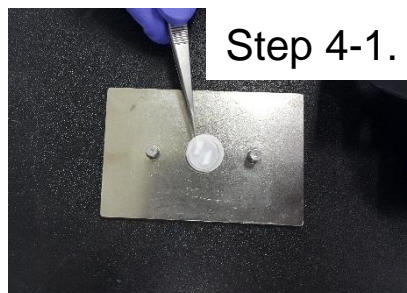
Step 6. Measurement background spectrum.

Step 7. Take a small amount of powder sample (about of 0.1-1% of the KBr amount) mix with the KBr powder. Subsequently grind the mixture for 3-5 minutes.

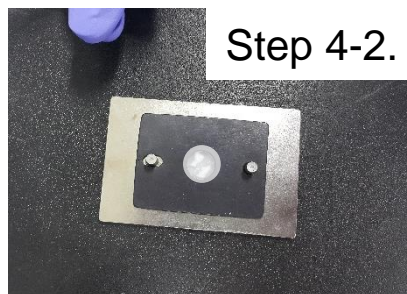
Step 2~6.

Step 8. Measurement sample spectrum.

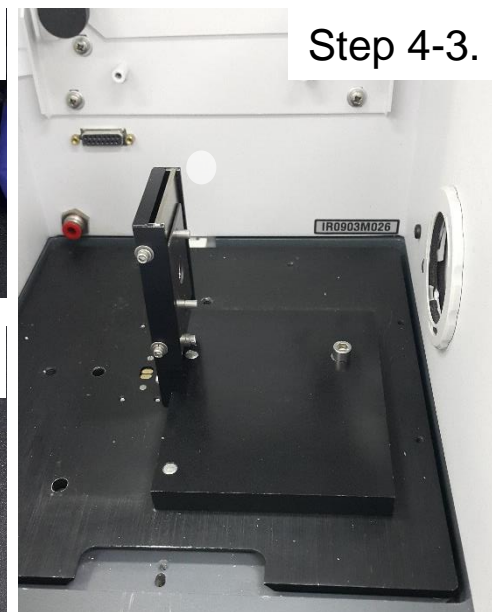
- ✓ A good KBr pellet is thin and transparent. Opaque pellets give poor spectra and white spots in a pellet indicate that the powder is not ground well enough, or is not dispersed properly in the pellets.



Step 4-1.



Step 4-2.



Step 4-3.

Transmission_liquid sample

Step 1. Choice the window material.(ZnSe or CaF_2)

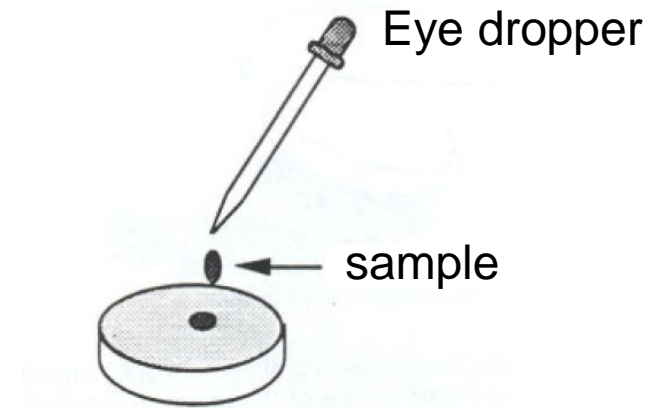
Step 2. Measurement background spectrum with window material.

Step 3. Place a small amount of sample onto the window using the eye dropper or spatula.

Step 4. Once enough sample is deposited on the window, place the other infrared window on top of the sample.

Step 4. Twist the windows together in opposite directions to get rid of air bubbles and to decrease the pathlength.

Step 5. Measurement sample spectrum with sample and window material.



Apply pressure and twist top window

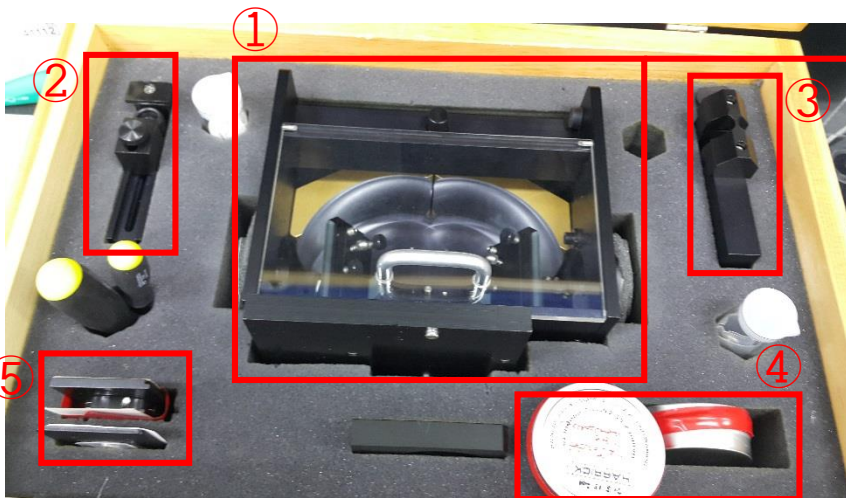


Apply pressure and twist bottom window

PREPARATION OF SEAGULL

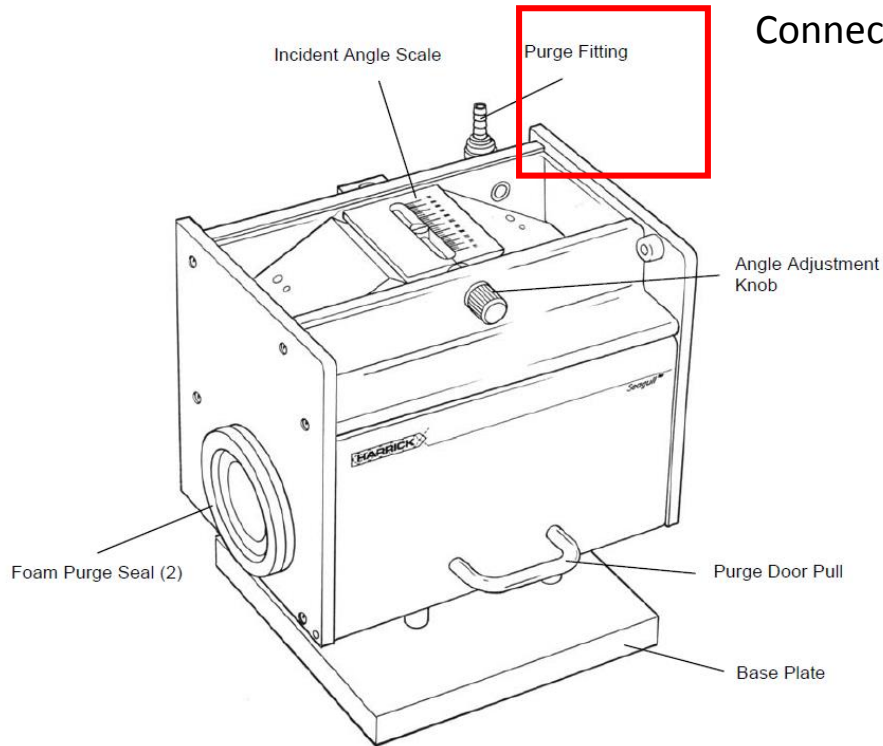


Place Seagull part to FT-IR main body.

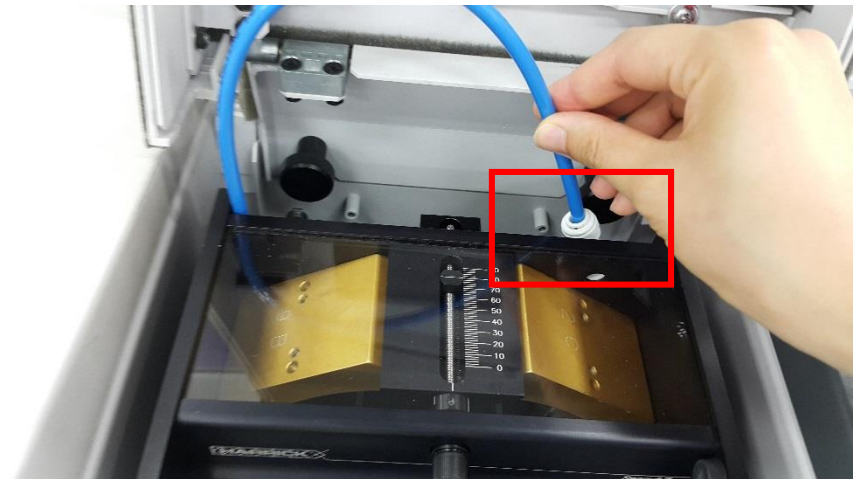
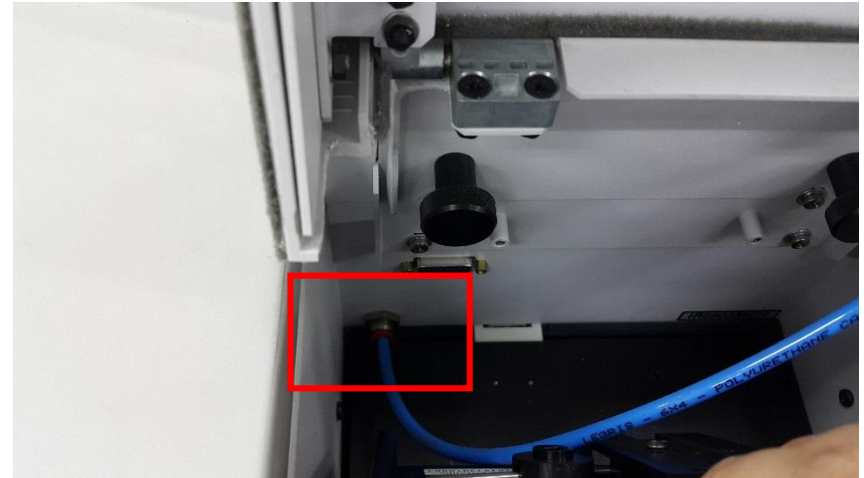


- ① Seagull part
- ② Sample holder
- ③ ATR sample holder
- ④ ATR crystal(Ge and ZnSe)
- ⑤ Polarizer

PREPARATION OF SEAGULL

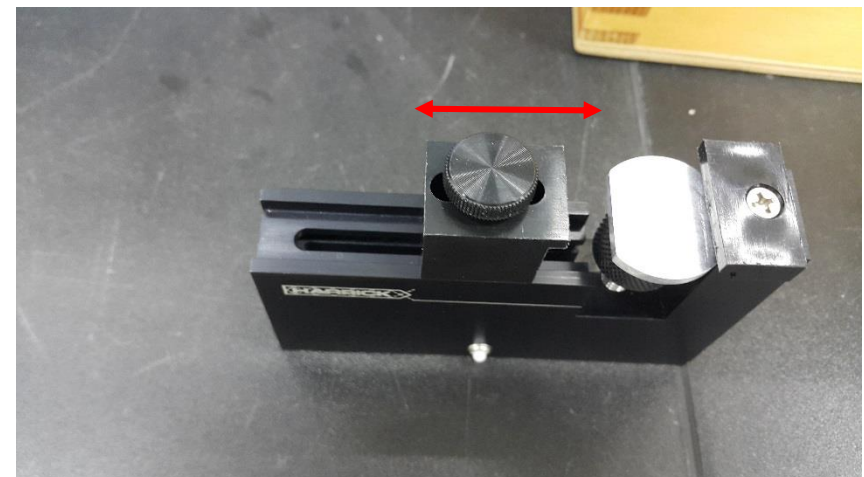
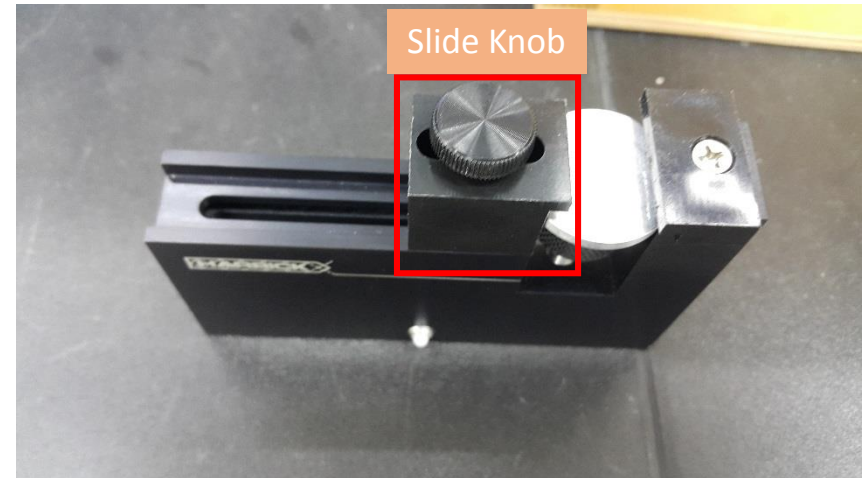
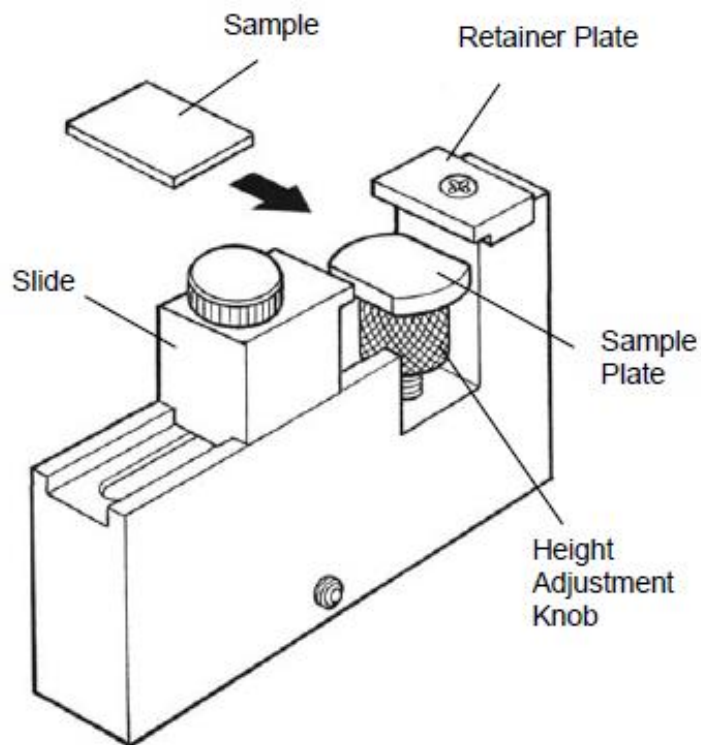


Connect an additional purge line to the fitting on the Seagull.

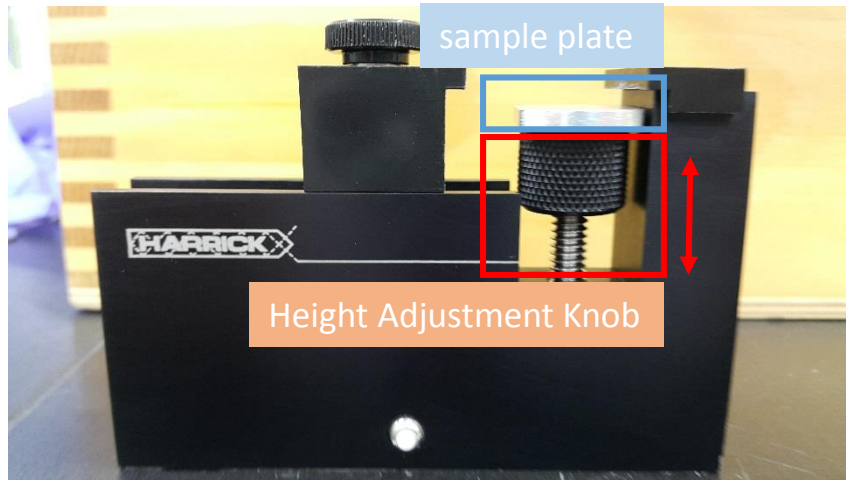


PREPARATION OF THE SAMPLE HOLDER

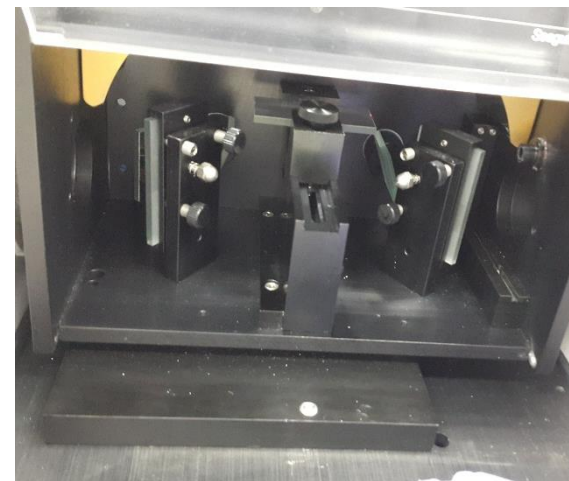
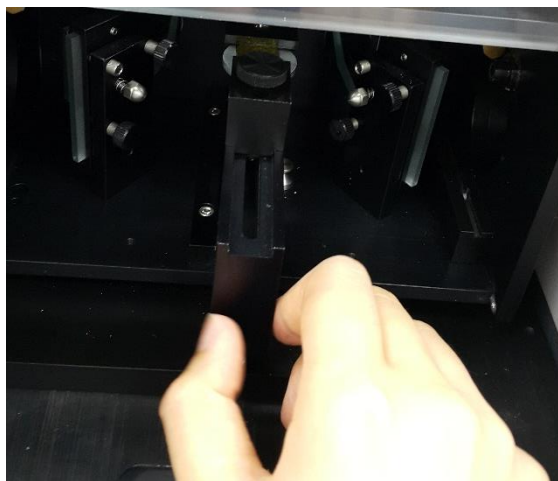
1) Turn the slide knob on the external reflection sample holder clockwise (Figure) until the slide is free to move.



PREPARATION OF THE SAMPLE HOLDER

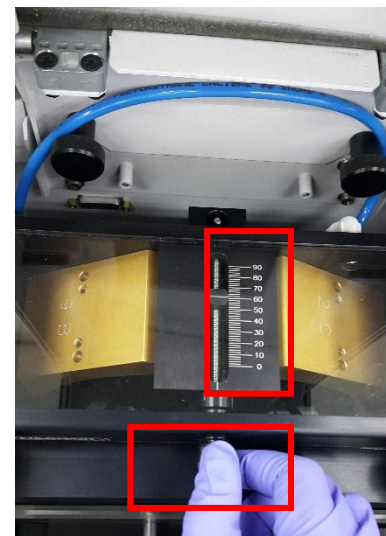
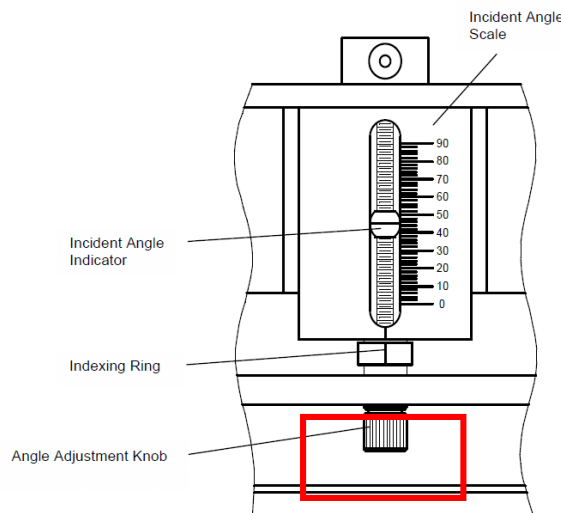
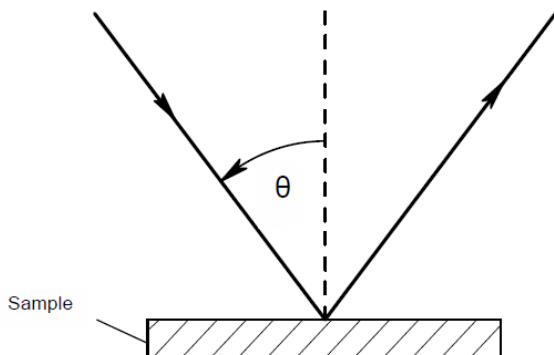


- 2) Lower the height adjustment knob by rotating it clockwise.
- 3) Locate the sample plate on the height adjustment knob.
- 4) Place the reference face up on the sample plate and move the slide so it lightly touches the reference.
- 5) Secure the slide by turning the slide knob clockwise.
- 6) Elevate the reference by rotating the height adjustment knob counterclockwise until the mirror is held in place against the retainer plate and slide.
- 7) Open the purge door and slide the sample holder into the Seagull™.



INCIDENCE ANGLE AND REFLECTANCE OPERATION

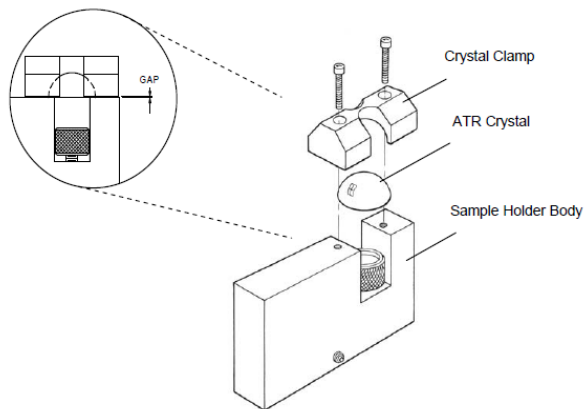
The incident angle θ (Figure 5) is read from the scale on top of the Seagull.



- 8) Set the incident angle.
- 9) Collect the background spectrum.
- 10) Replace the reference with the sample.
- 11) Collect the sample spectrum.

ATR SAMPLE HOLDER

The ATR sample holder is used for ATR operation.

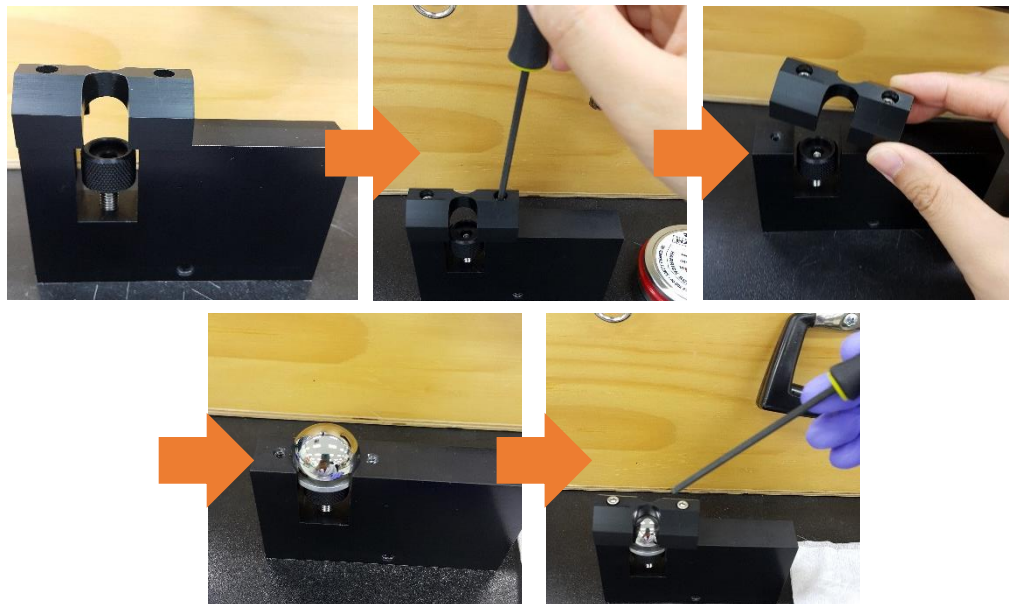


MOUNTING THE ATR CRYSTAL

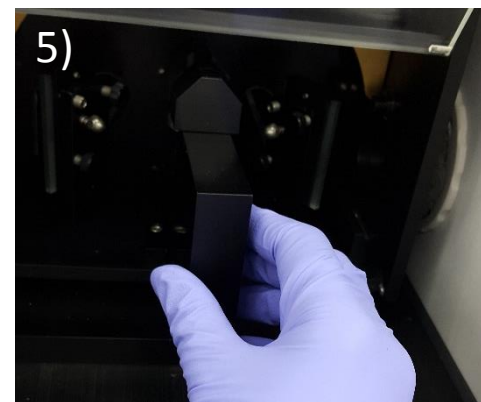
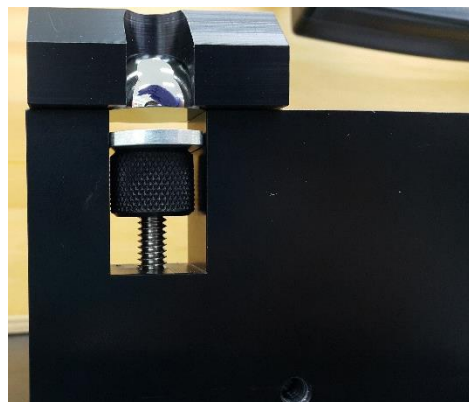
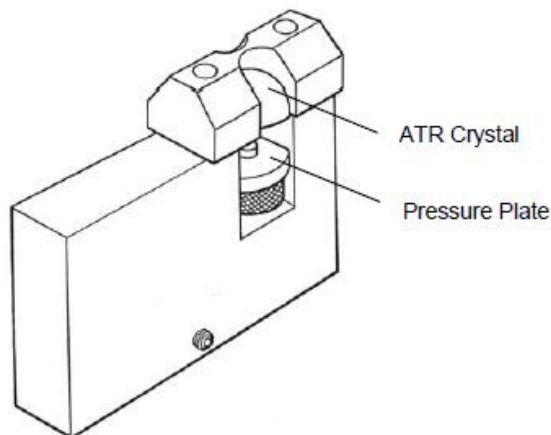
- 1) Mount the ATR crystal into the ATR sample holder using a 7/64" ball driver undo the two screws on the crystal clamp of the ATR sample holder.
- 2) Remove the crystal clamp.
- 3) Carefully place the crystal on the ATR sample holder body as shown in Figure.
- 4) Carefully replace the crystal clamp and tighten the screws evenly so the crystal clamp is seated with equal gaps on both ends.

CAUTION:

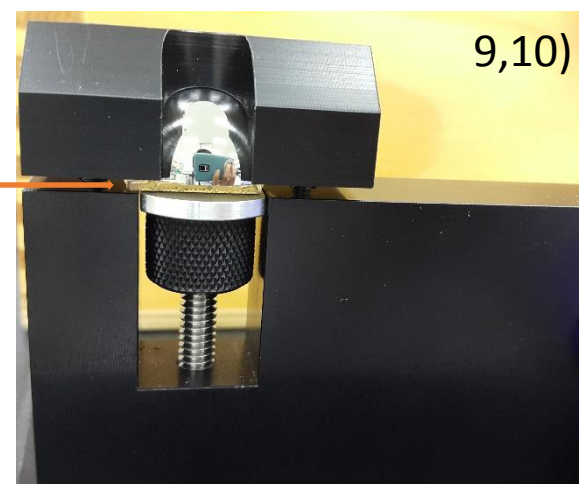
The ATR crystal scratches easily so it should be handled with care. Wipe only with damp lens tissue or cotton swabs.



ATR SAMPLE HOLDER



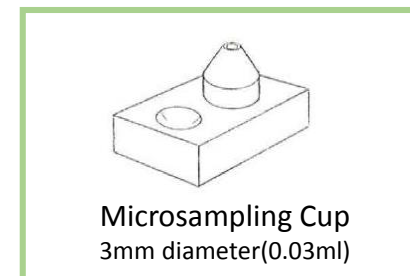
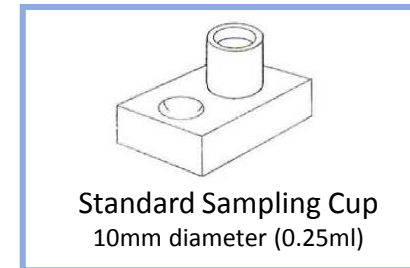
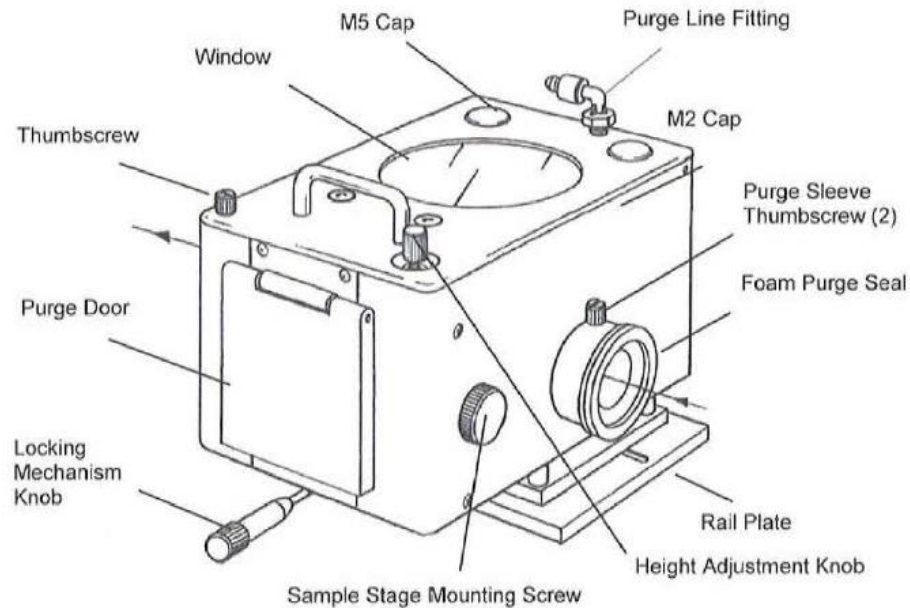
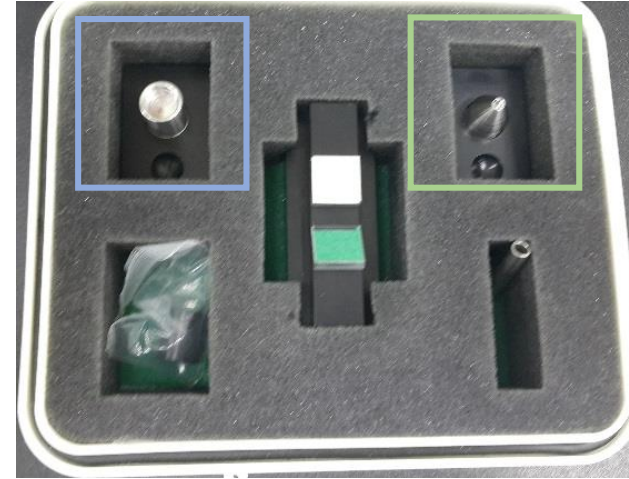
Sample



To use the ATR sample holder:

- 5) Install the ATR sample holder into the Seagull™.
- 6) Set the desired incident angle.
- 7) Collect the background spectrum.
- 8) Locate the pressure plate on the height adjustment knob of the ATR sample holder.
- 9) Place the sample on the pressure plate.
- 10) Raise the pressure plate to make contact between the crystal and the sample.
- 11) Collect the sample spectrum.
- 12) Clean the crystal with lens paper and IPA.

DRIFT(Diffuse Reflectance Infrared Fourier Transform)_Praying Mantis



DRIFT(Diffuse Reflectance Infrared Fourier Transform)

■ Factors for high quality DRIFT

Particle Size

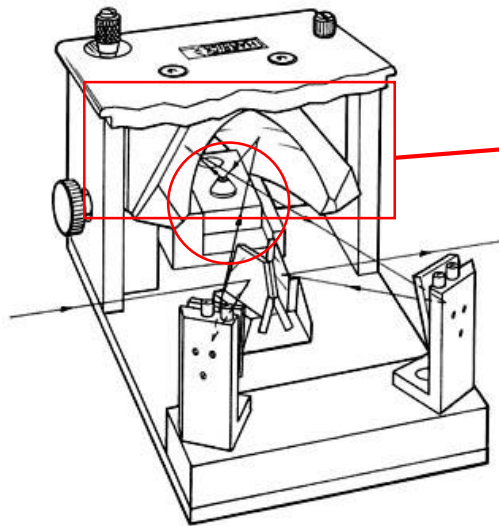
Smaller particles ($\leq 10 \mu\text{m}$) improve the quality of spectra.

Refractive Index

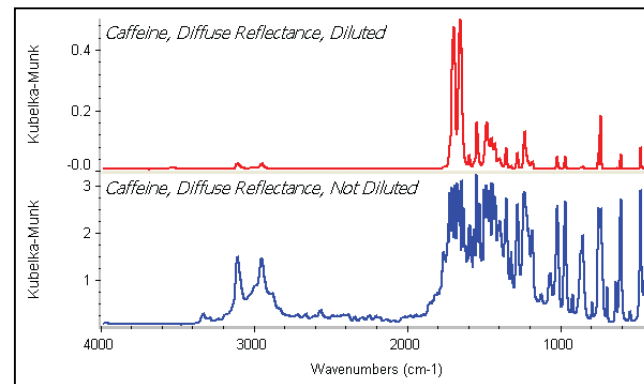
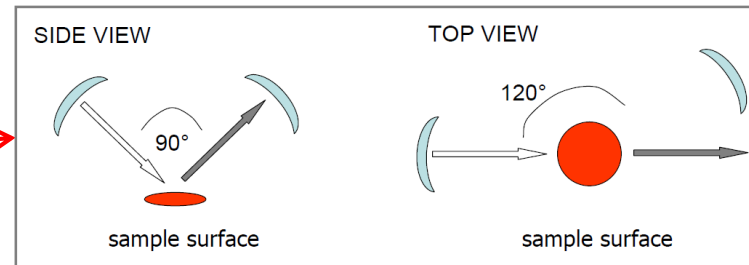
Sample dilution reduce the distorted spectra by Specular reflectance component.

Packing

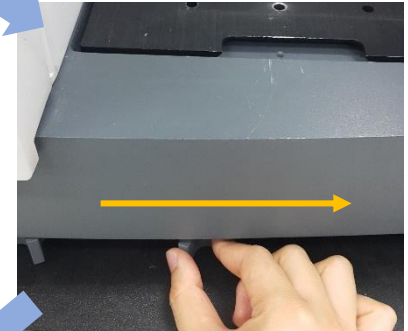
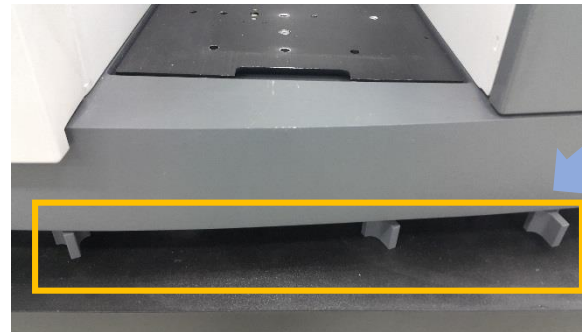
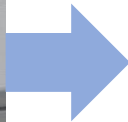
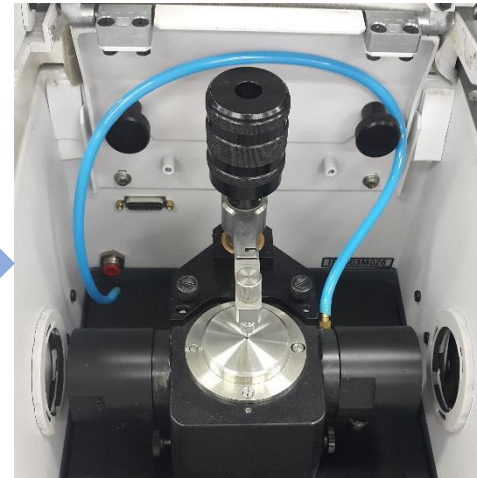
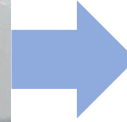
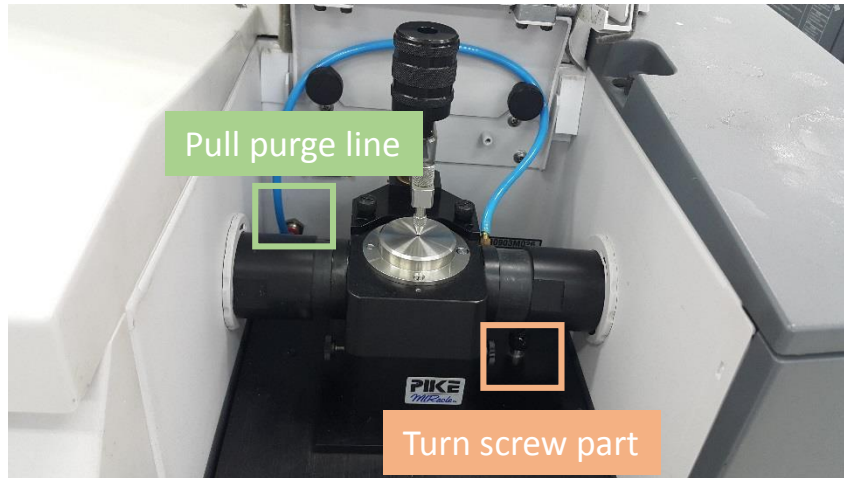
Loosely packing maximize IR beam penetration and minimize spectral distortions.



Interior View of the Praying Mantis

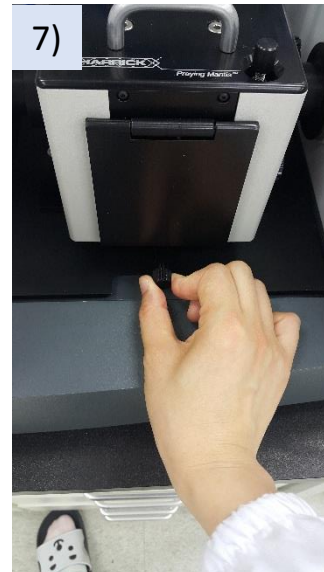


PREPARATION OF PRAYING MANTIS



The plate can be removed from FT-IR main body when this lever move from the left to the right.

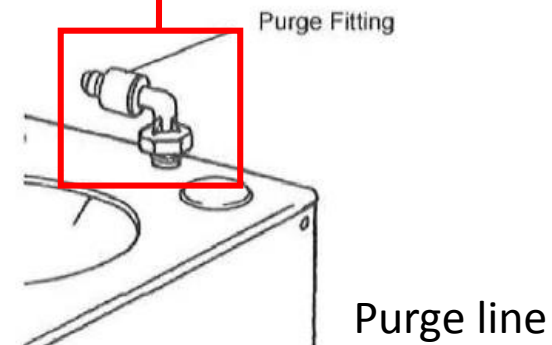
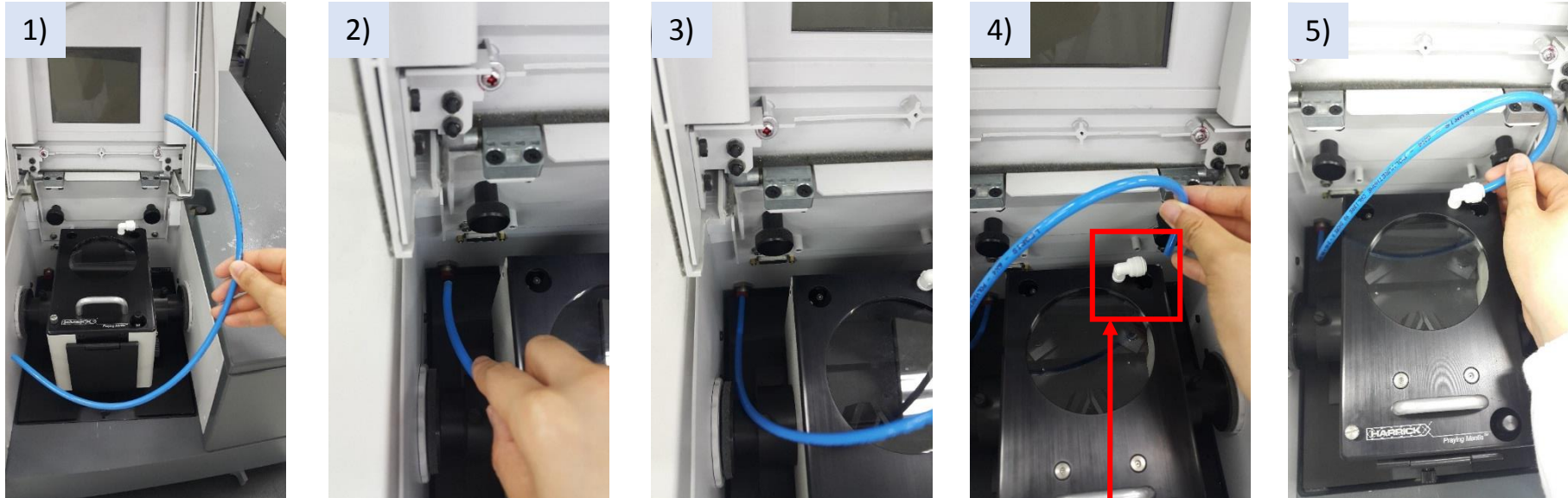
PREPARATION OF PRAYING MANTIS



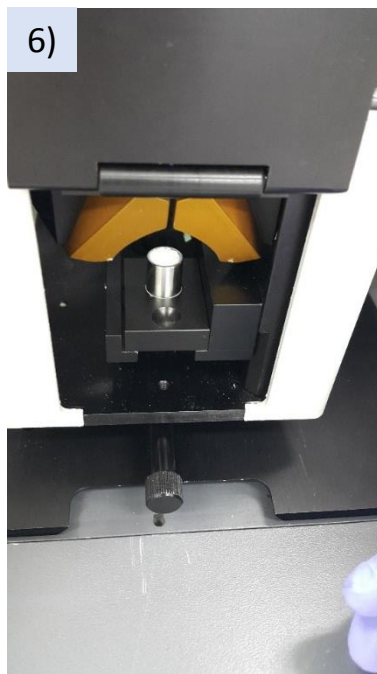
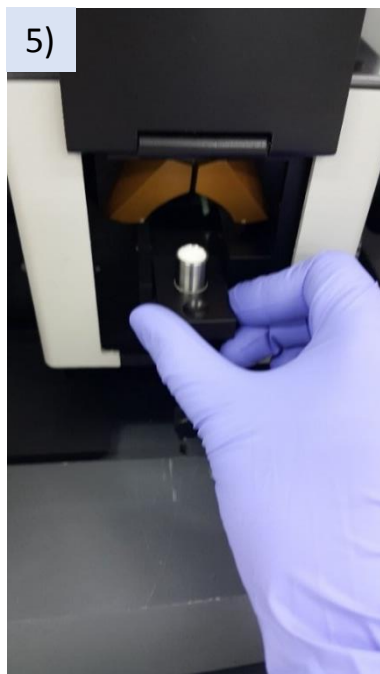
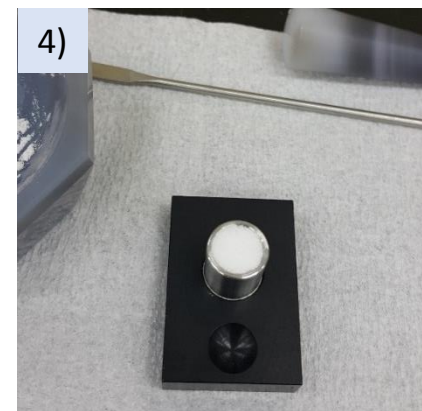
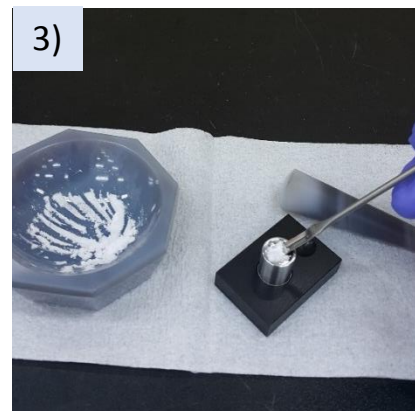
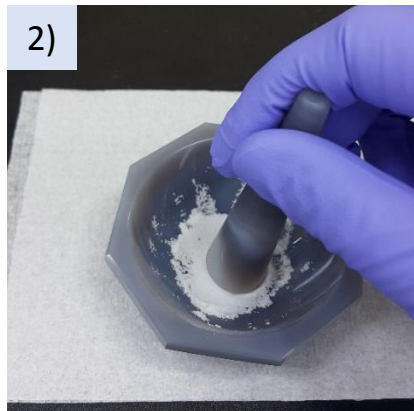
- 1) Remove the plate.
- 2) Change to the rail plate.
- 3,4) Change the lever position to fix the rail plate.
- 5) Put the Praying Mantis on the rail plate.
- 6) Move the Praying Mantis along the rails until the spectrometer focal point is in the center of the attachment.
- 7) Lock the Praying Mantis in place by tightening the locking mechanism knob.
- 8~10) Extend the purge sleeves until they firmly contact the sides of the sample compartment. Lock the purge sleeves in place with the thumbscrews.

PREPARATION OF PRAYING MANTIS

For quicker purging, connect an additional purge line to the fitting on the Praying Mantis.

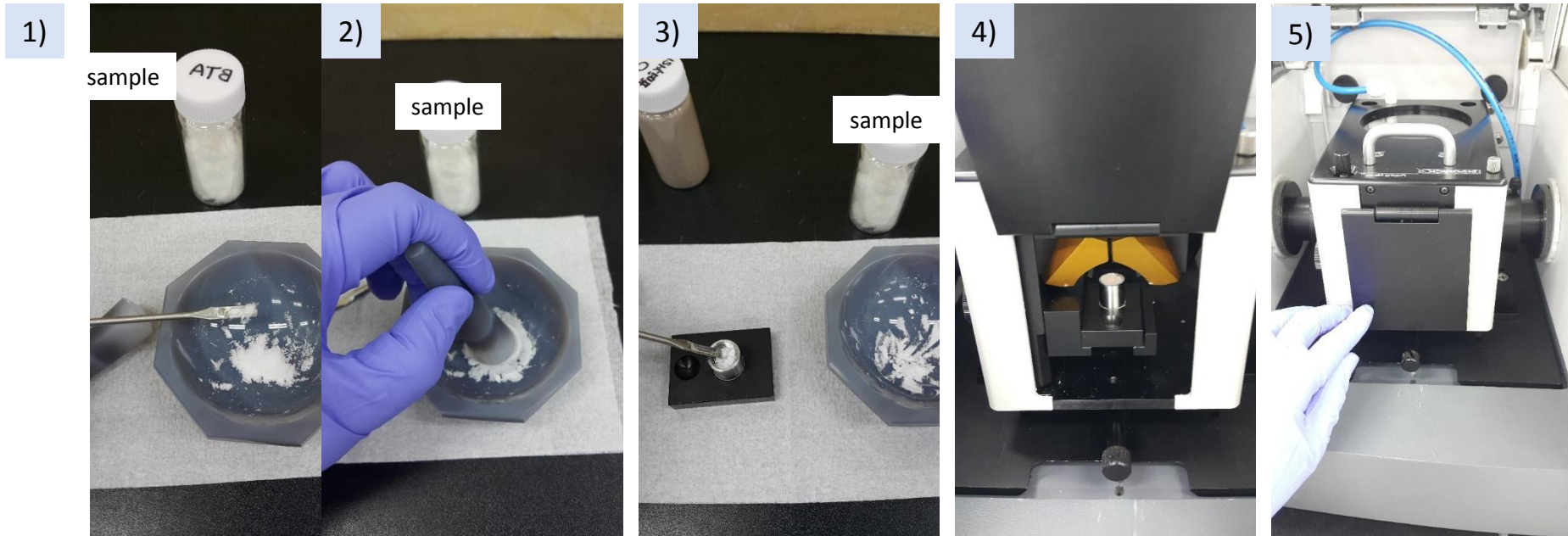


BACKGROUND SPECTRUM



- 1) Prepare reference material.
- 2) When the reference material is KBr, grind the powder with mortar and pestle.
The particle size should be smaller than 10 μm (i.e. not exceeding the wavelength of the incident radiation).
- 3) Overfill one of the sampling cups with the reference material (i.e. KBr).
- 4) Level off the surface using a flat blade.
- 5~6) Open the purge door and slide the sampling cup into the Praying Mantis, pushing it in against the stop.
- 7) Find the center of laser position on sample surface turning the Height adjustment knob.
- 8) Measure background spectrum.

SAMPLE SPECTRUM



1) Add sample to reference material(i.e. KBr).

2) Grind the sample with reference material.

If the sample is a strong absorber it may need to be diluted (approximately 1~5%) in a nonabsorbent reference matrix.

3) Overfill one of the sampling cups with the sample and level off the surface using a flat blade.

4) Open the purge door and slide the sampling cup into the Praying Mantis, pushing it in against the stop.

5) Close the purge door.

6) Measure sample spectrum.

5. FT-IR Operation

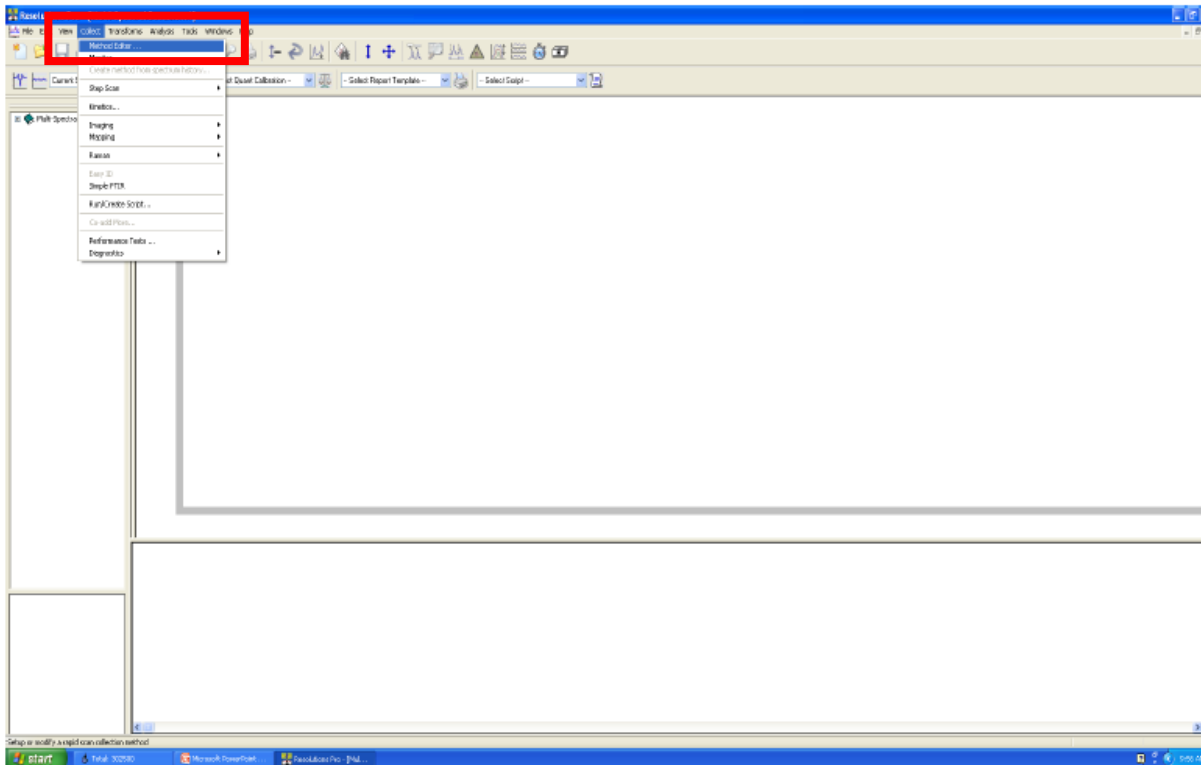
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SCIENCE AND TECHNOLOGY

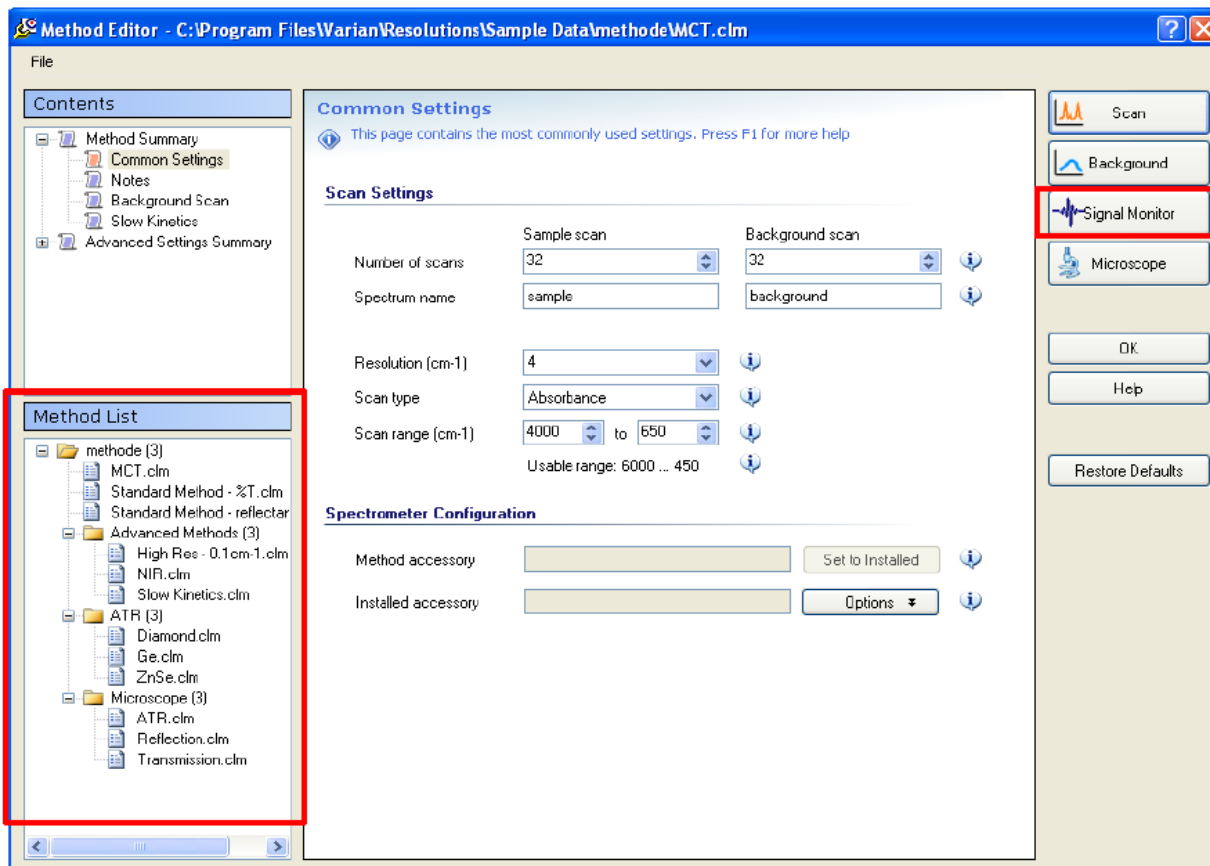
1. Agilent Resolution Pro. STATR



2. Collect ⇒ Method Editor

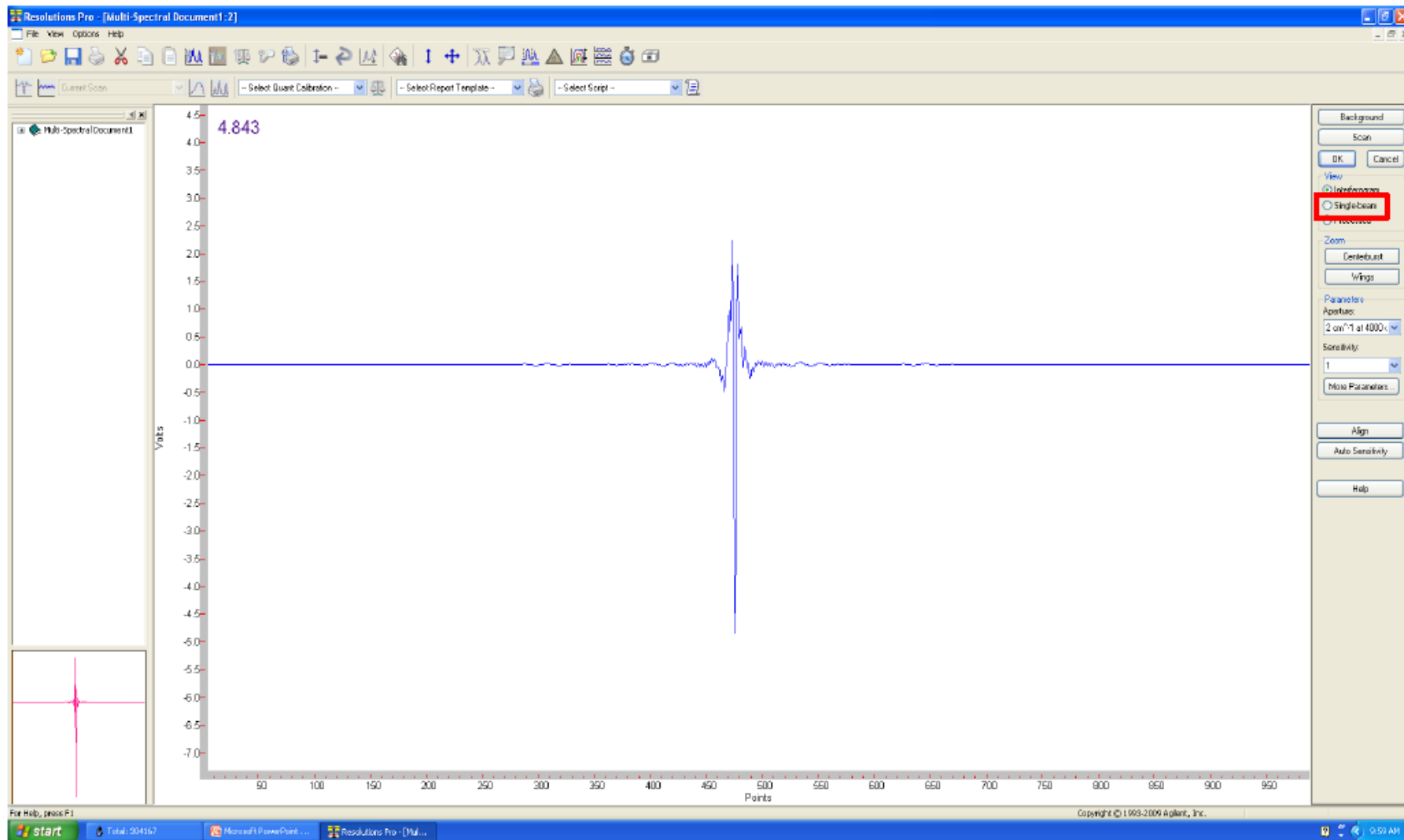


3. Method List ⇒ select method

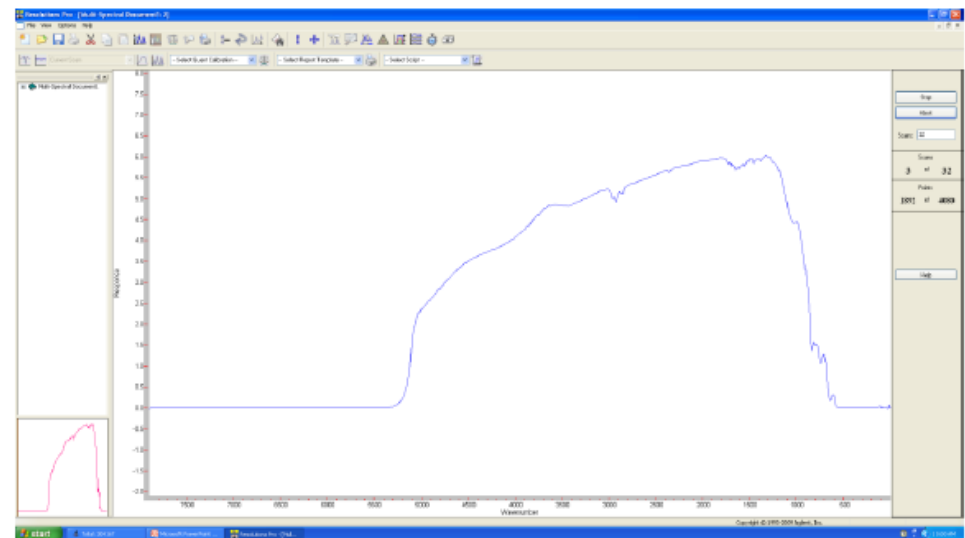
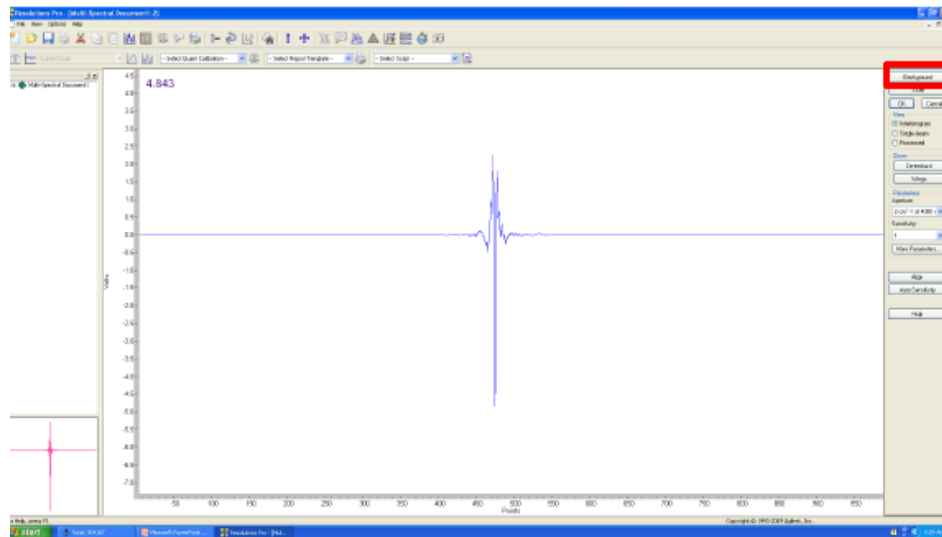


4. Signal Monitor click

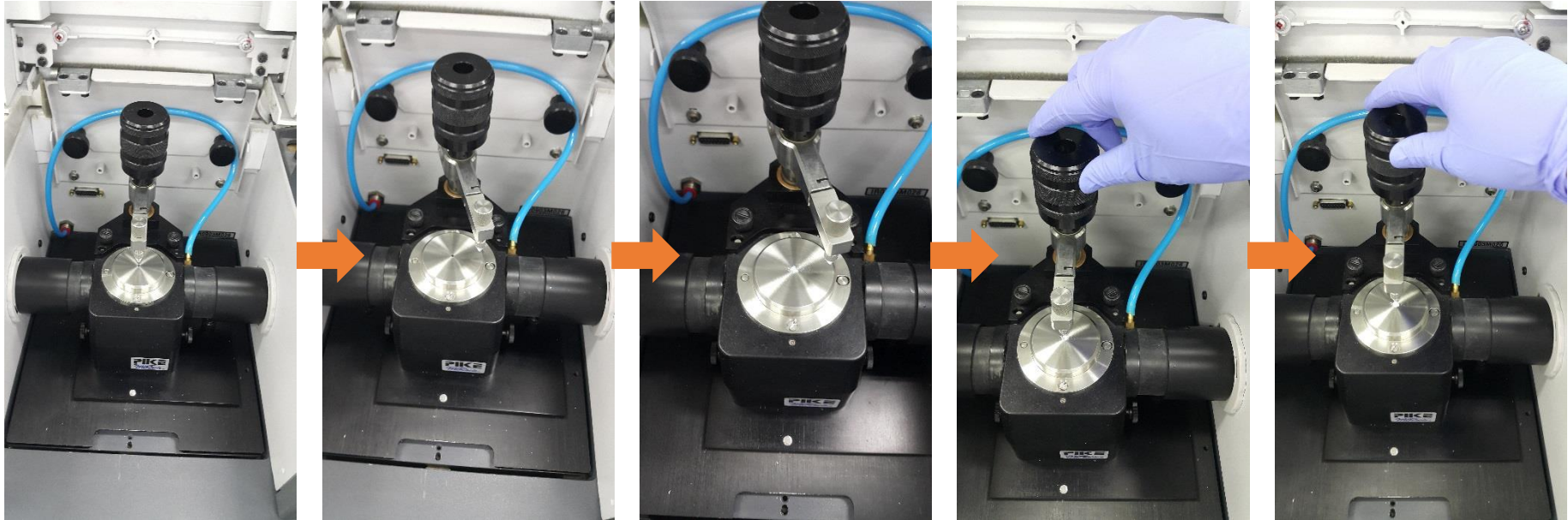
5. Single Beam click



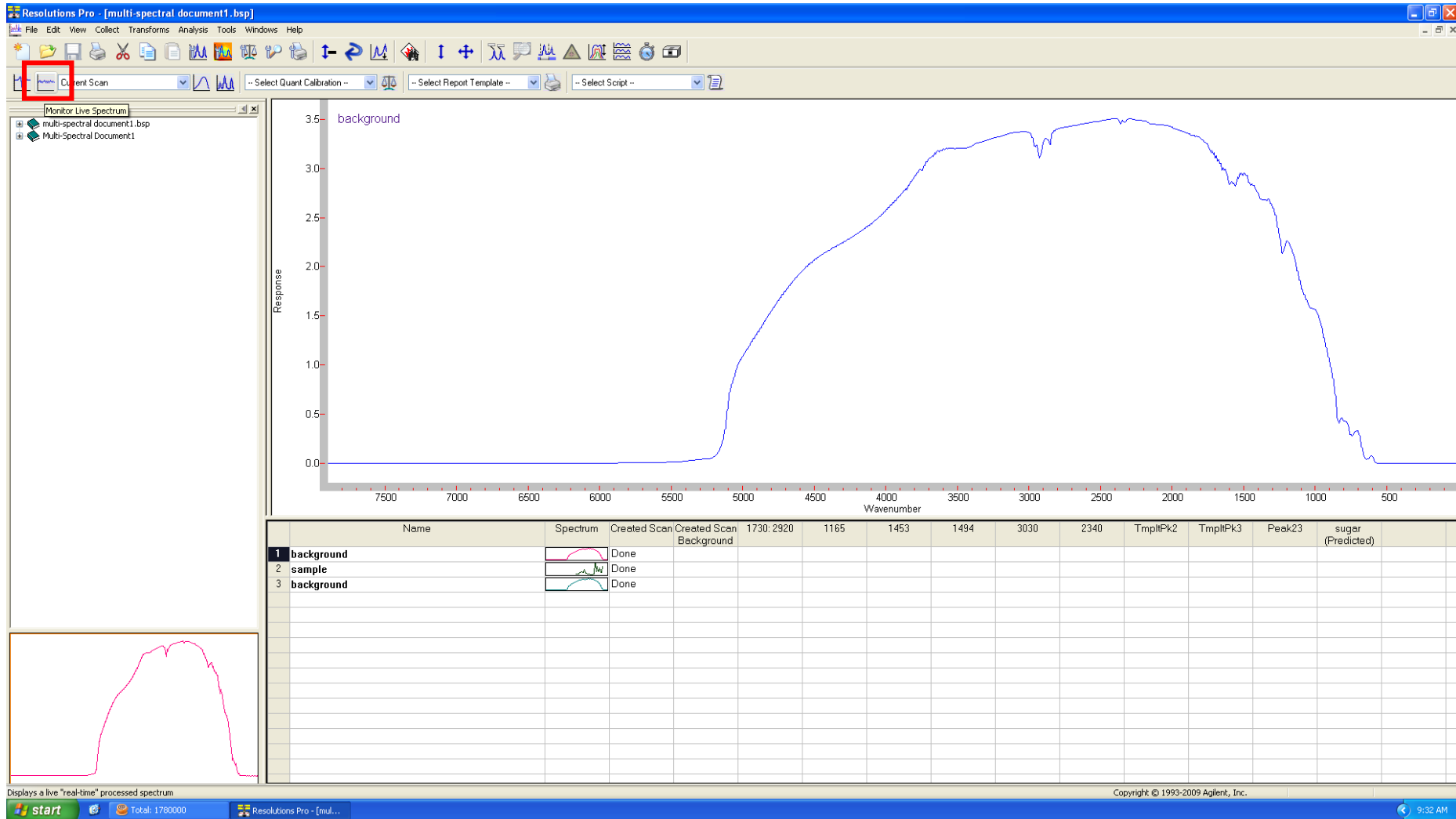
6. Background click



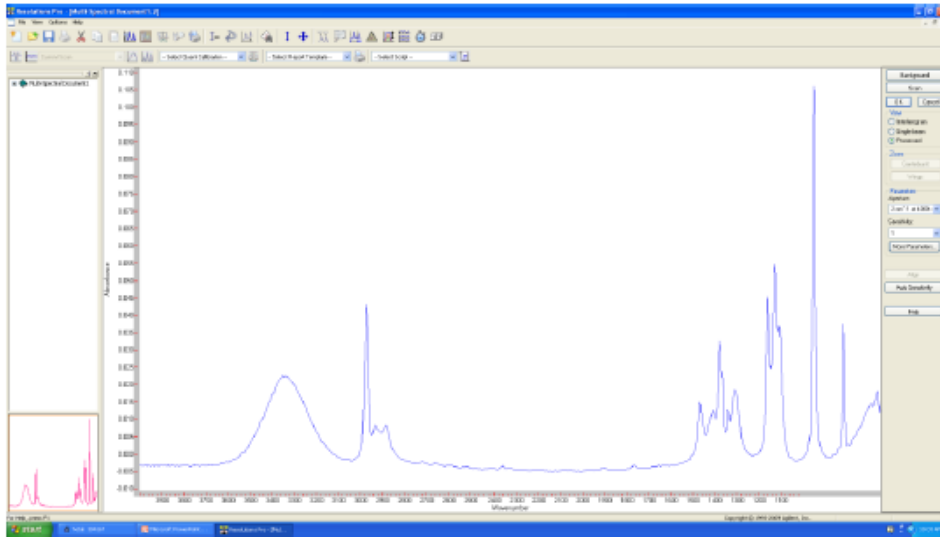
7. Sample loading and press



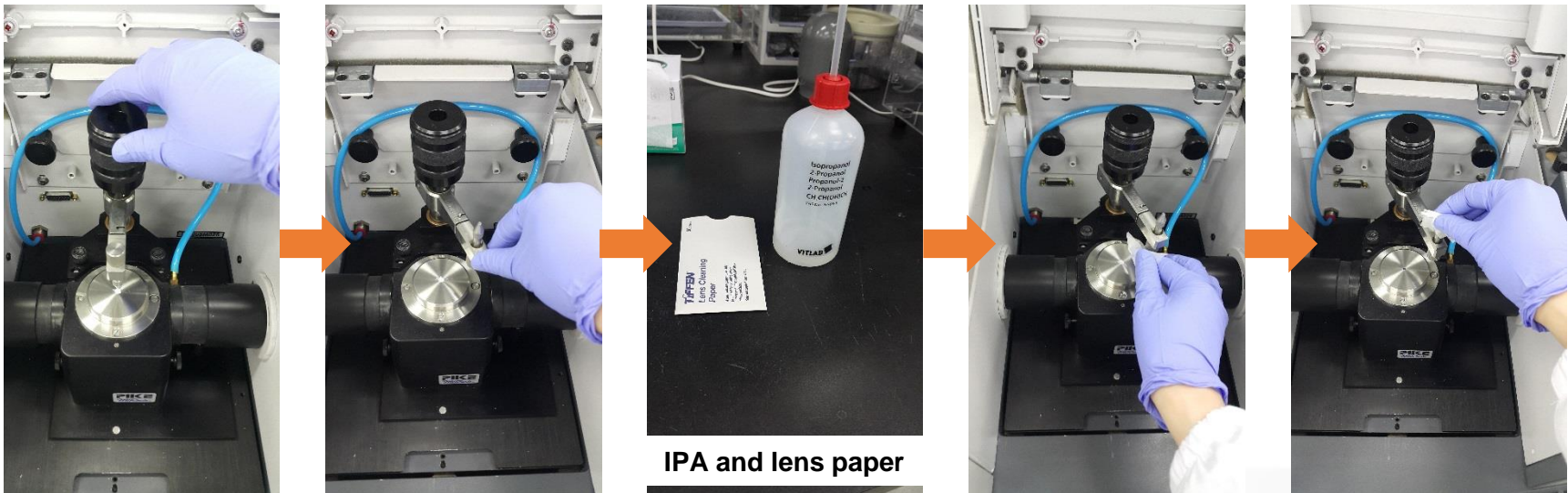
8. Monitor live spectrum click



9. Scan click



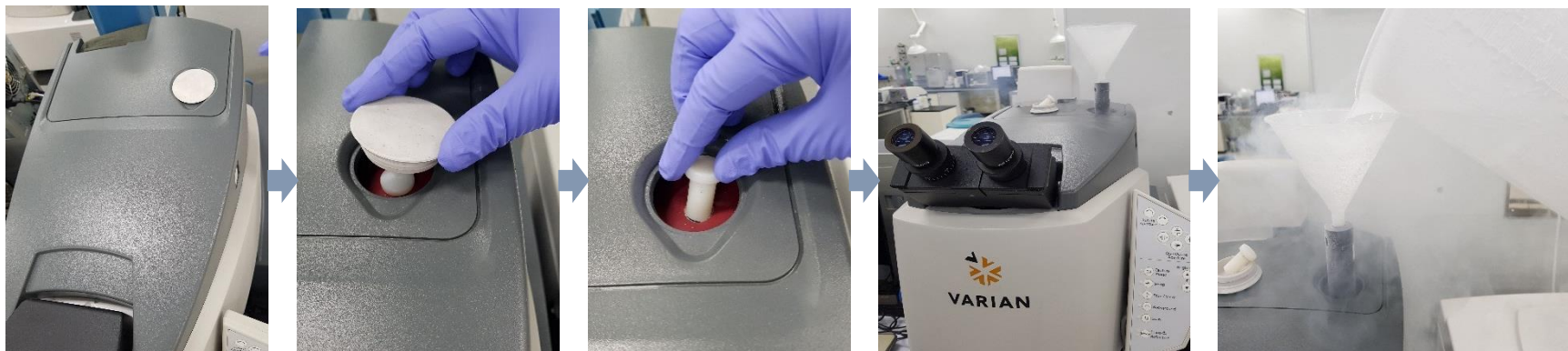
10. ATR cleaning



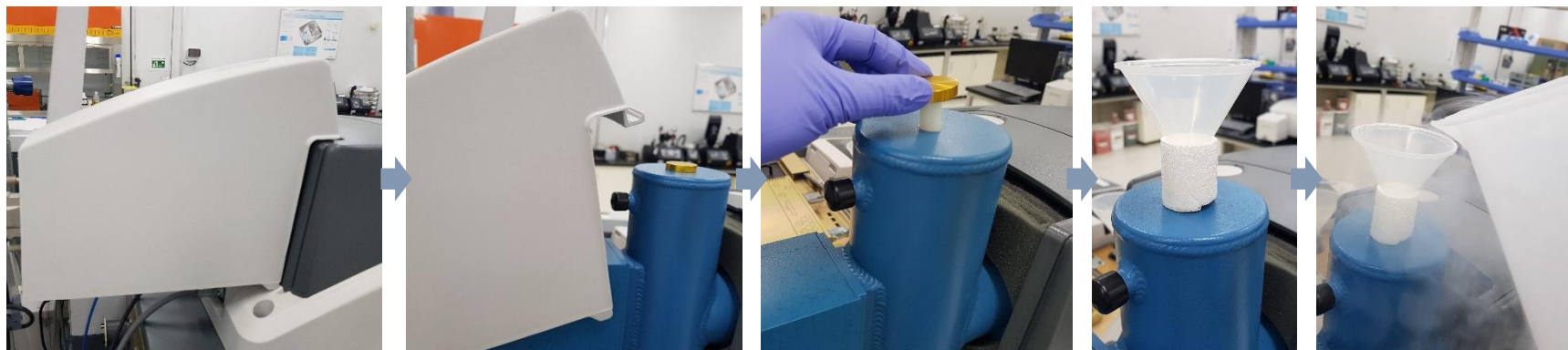
The screenshot shows the Resonance Pro software interface. The main window displays a spectral analysis plot with a blue waveform. A red rectangular box highlights a specific frequency range on the x-axis, which is labeled 'Frequency [Hz]'. The y-axis is labeled 'dB SPL'. The right sidebar contains various settings and controls, including a 'Control' button highlighted with a red box. The bottom status bar shows the software version and copyright information.

- Fill the detector with Liquid Nitrogen.

1) Microscope MCT detector(for single spectrum) need about 500mL liquid nitrogen.



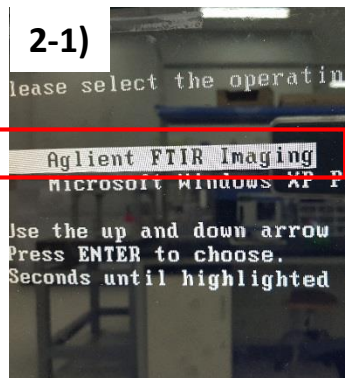
2) FPA detector(for image scan) need about 1L liquid nitrogen.



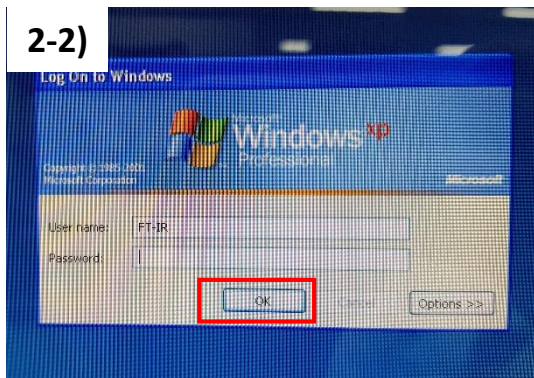
Restart the computer and Switch on the microscope part.

- 1) Select Agilent FTIR Imaging.
- 2) Click OK on Long On to Windows.
- 3) Switch on the microscope part(backside of IR microscope).
- 4) Switch on the FPA detector(only for the IR image scan).
- 5) Switch on the Joy stick for the stage control.
- 6) Open the beam line between mainbody and microscope part.

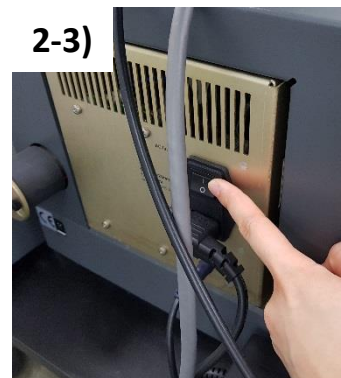
2-1)



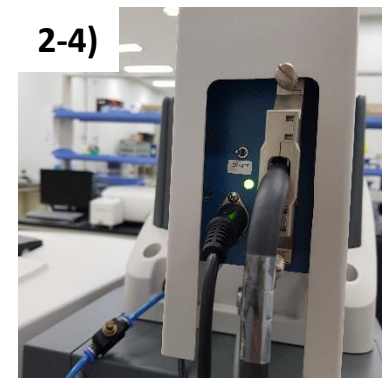
2-2)



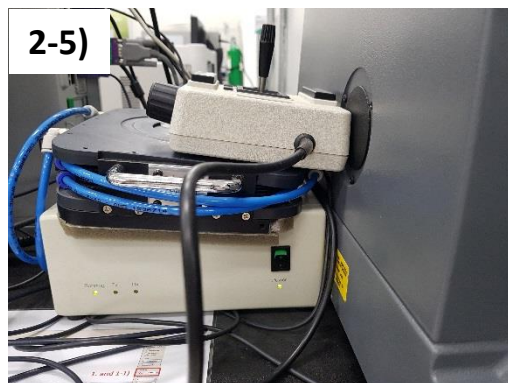
2-3)



2-4)



2-5)

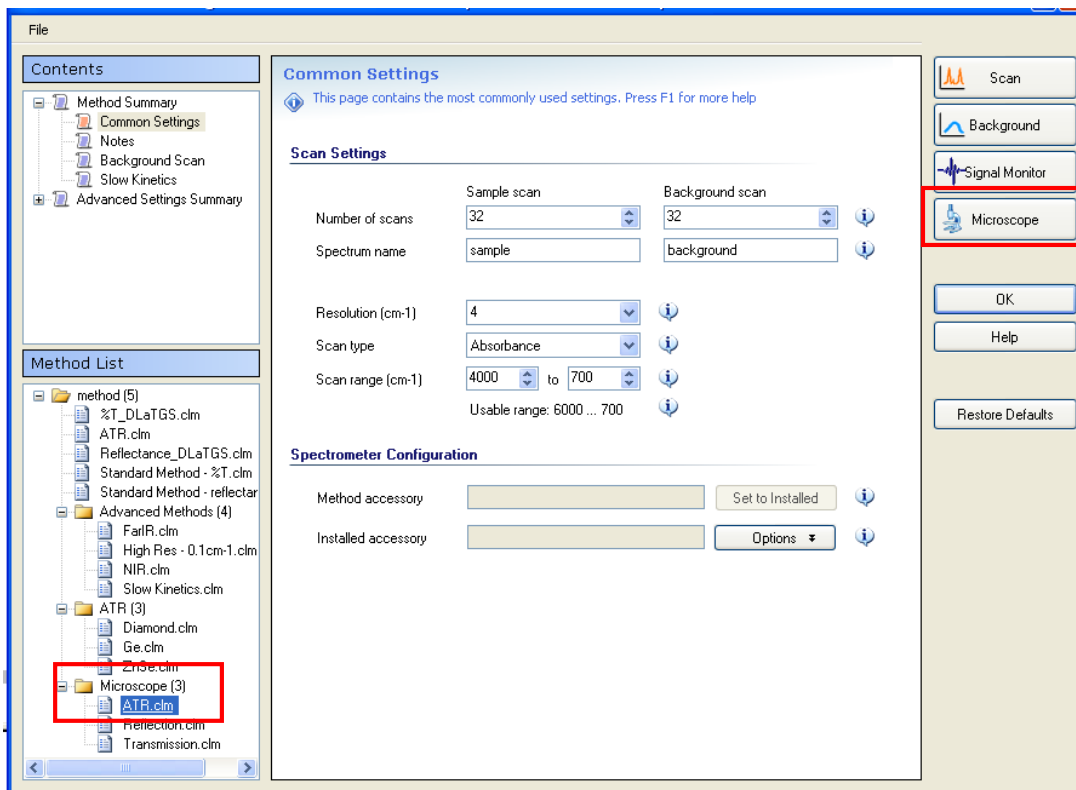


2-6)

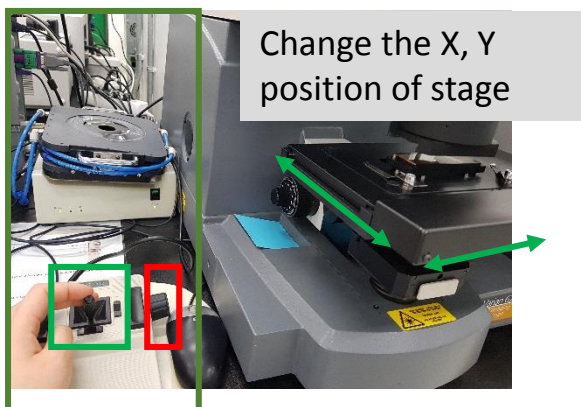
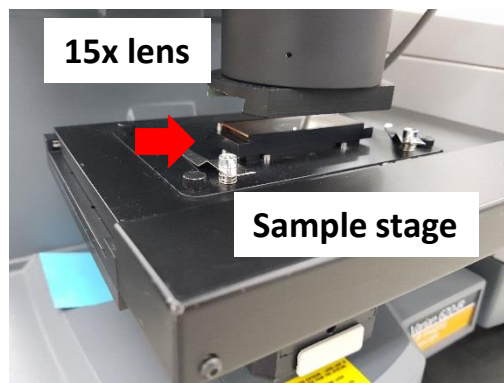


■ FT-IR Microscope_ATR

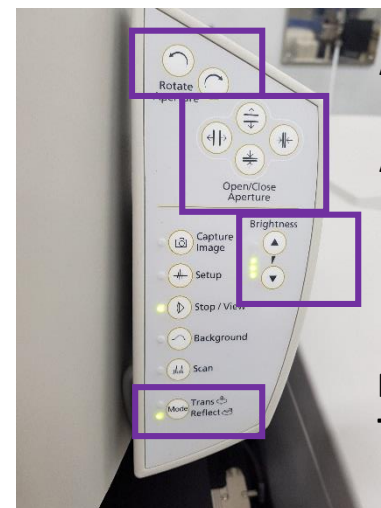
1. Open the Agilent Resolution Pro. Program.
2. Place the sample on the sample stage.
3. Method editor – Method List – Microscope.
4. Find focus and select the measurement point of sample changing the stage position.



■ FT-IR Microscope_ATR



Turn **Right** – Stage **DOWN**
Turn **Left** – Stage **UP**



Aperture rotation

Aperture size

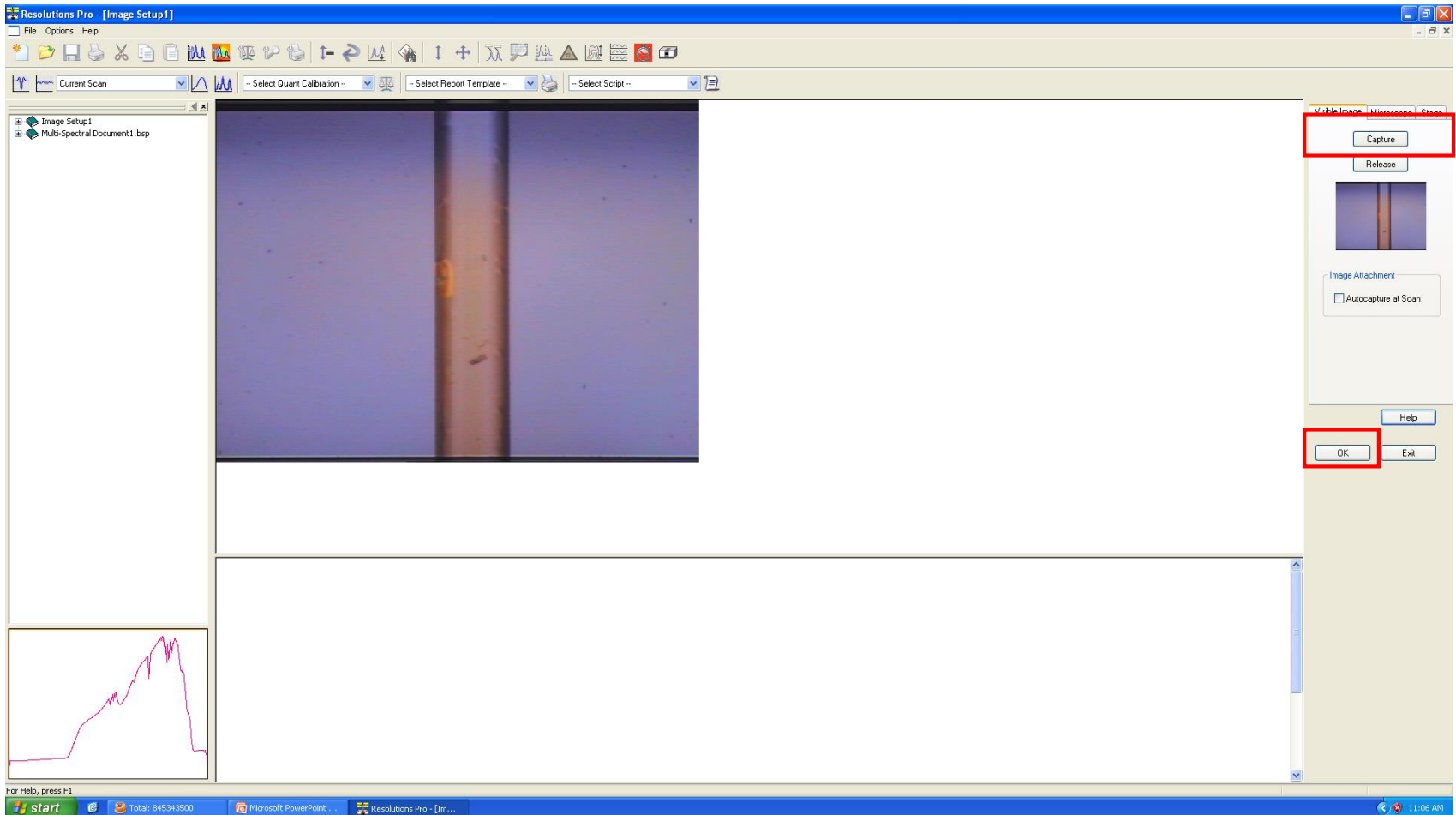
Brightness

Reflectance/
Transmittance mode

■ FT-IR Microscope_ATR

5. Click Capture.

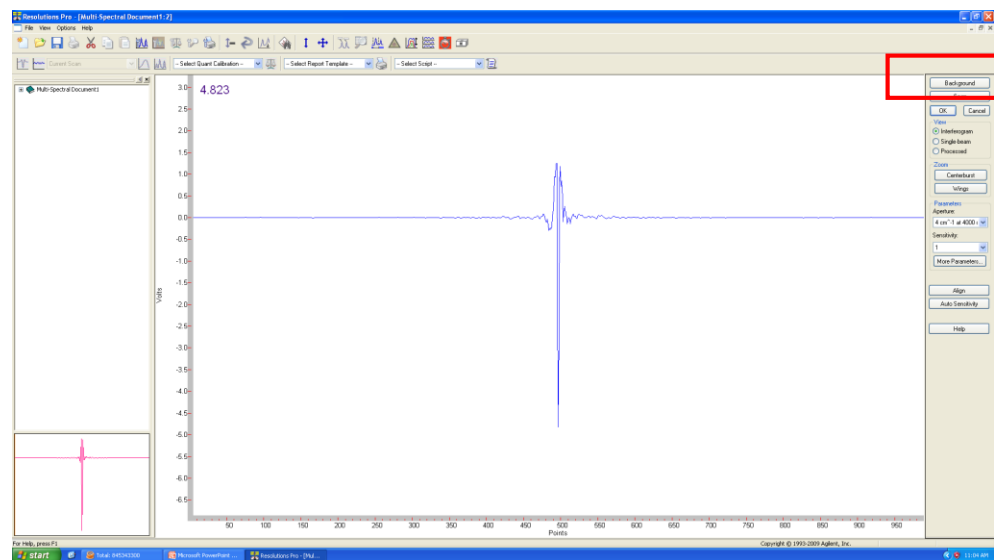
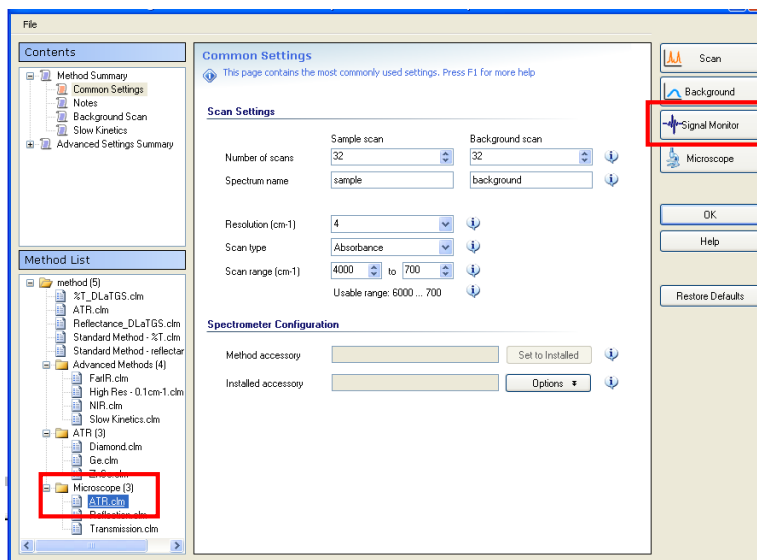
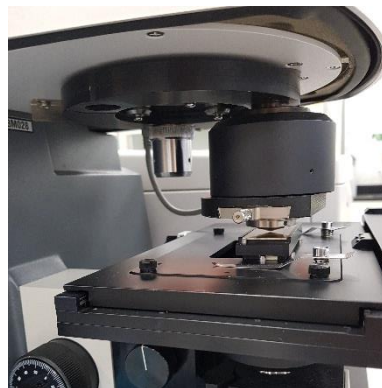
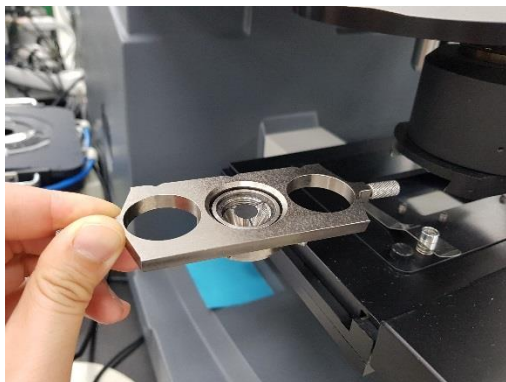
6. Click OK.



■ FT-IR Microscope_ATR

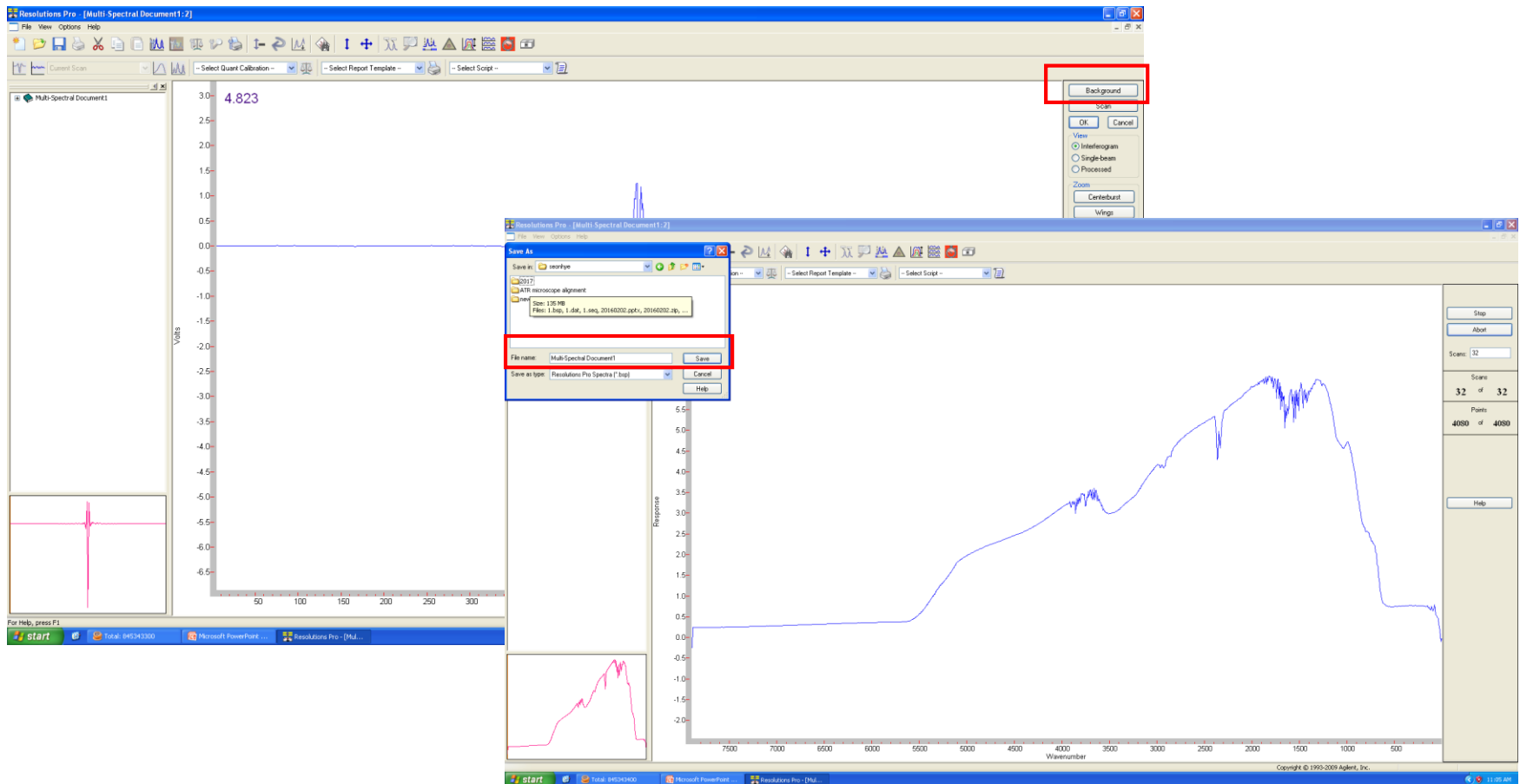
7. Put the ATR slide to the lens part.

8. Method editor – Method List – Microscope – Reflectance – Signal monitor



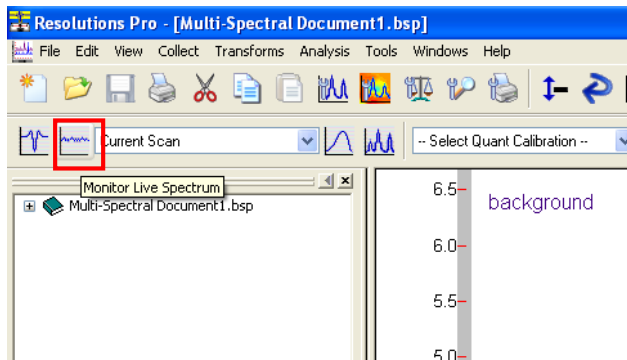
■ FT-IR Microscope_ATR

9. Background – Save

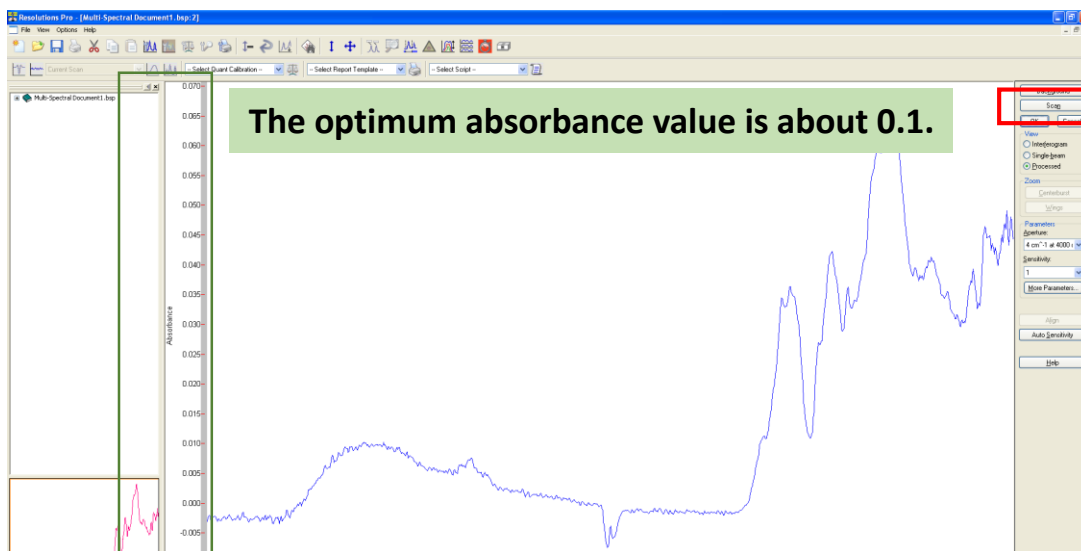


■ FT-IR Microscope_ATR

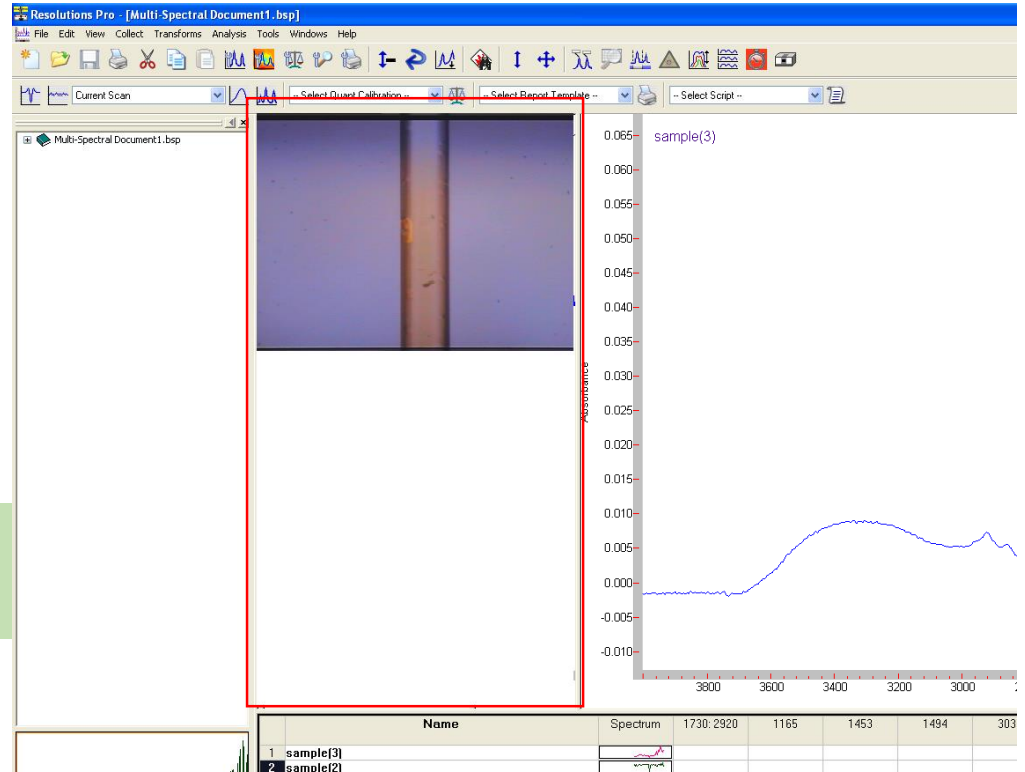
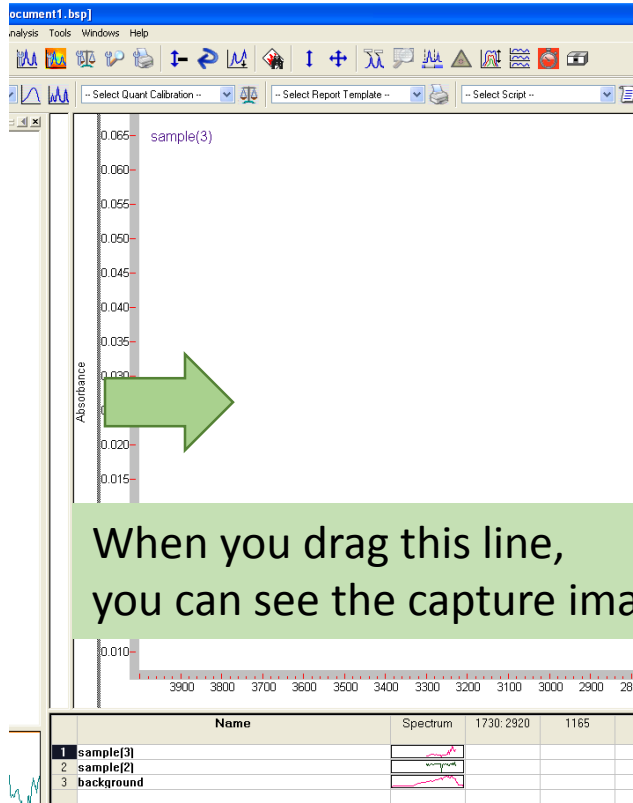
10. Monitor Live Spectrum



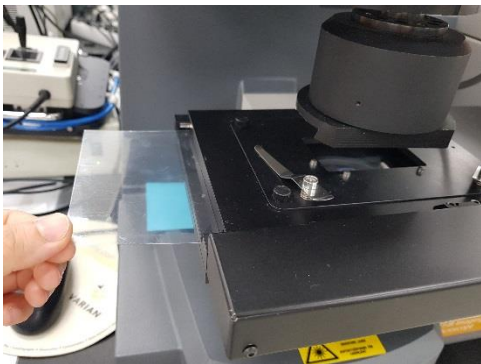
11. Move up the stage checking the IR signal through Monitor live spectrum.
12. Scan.



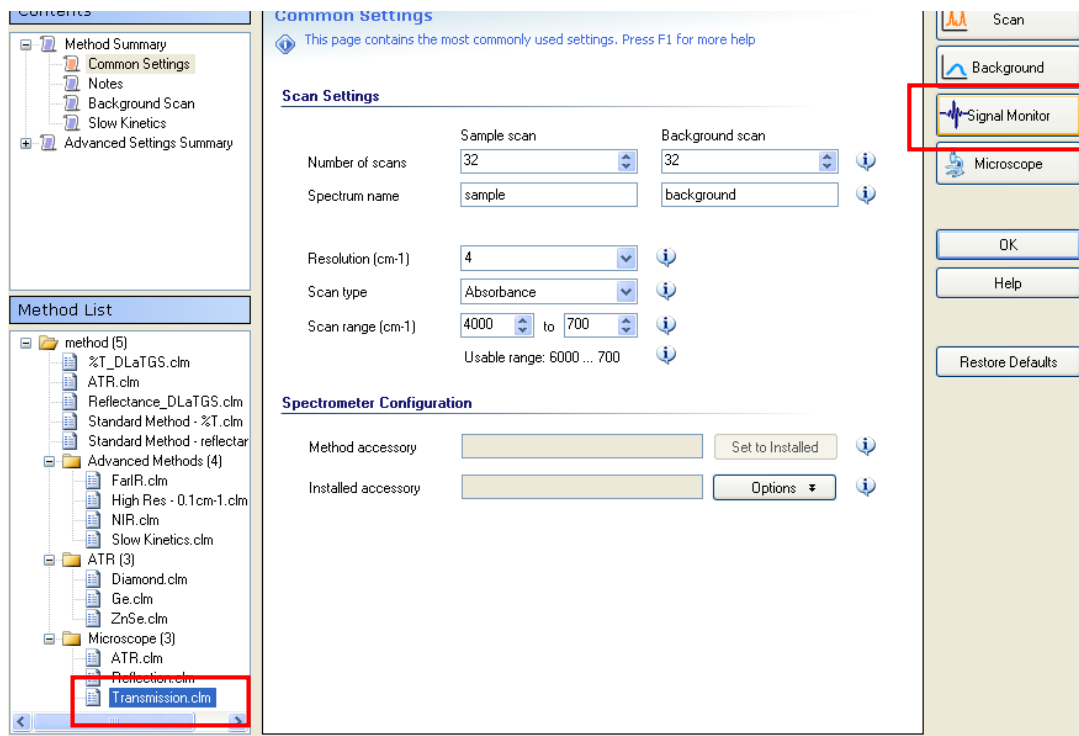
■ FT-IR Microscope_ATR



■ FT-IR Microscope_Transmittance

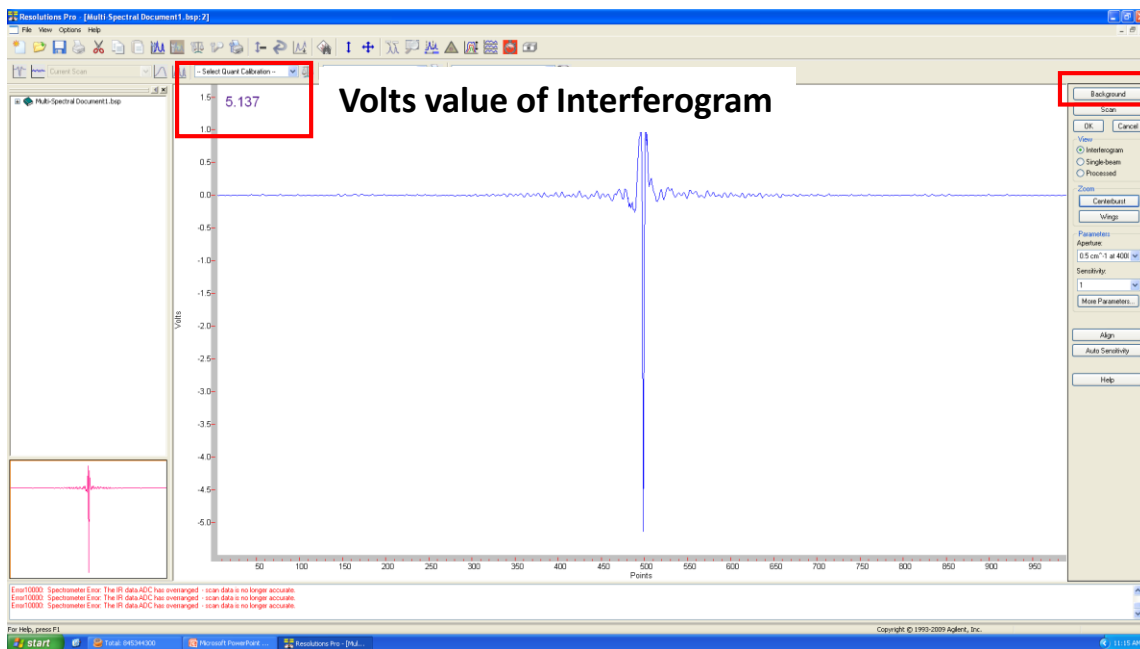
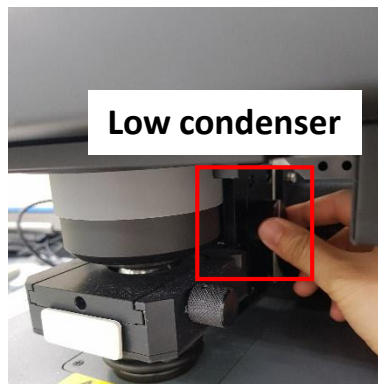


1. Remove the protective film in the stage.
2. Place the sample on the stage and find the focus.
3. Remove the sample from the stage.
4. Method editor – Method List – Microscope – Transmittance – Signal monitor



■ FT-IR Microscope_Transmittance

5. Adjust the maximum Interferogram volts value changing the low condenser.

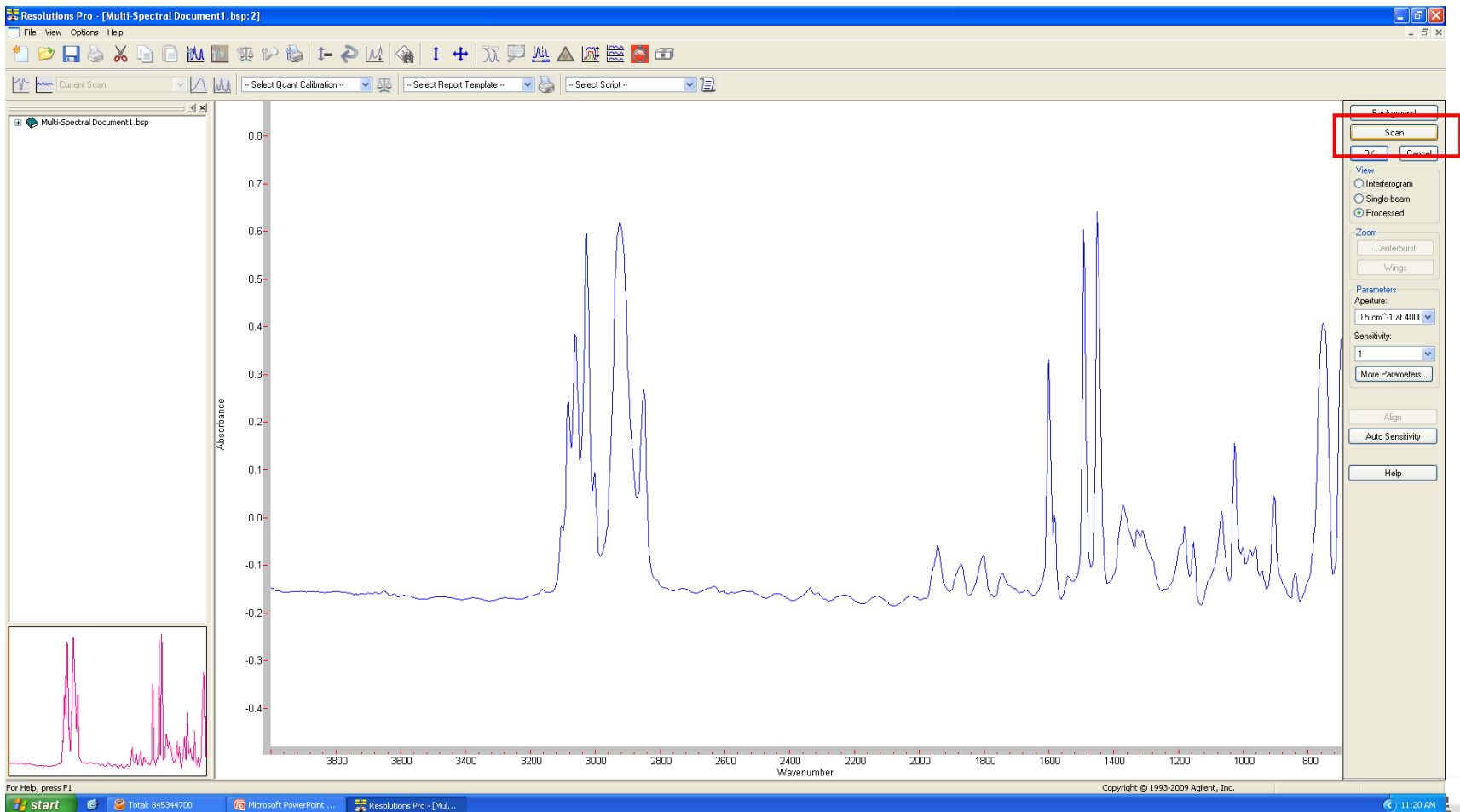


6. Collect Background.

7. Save.

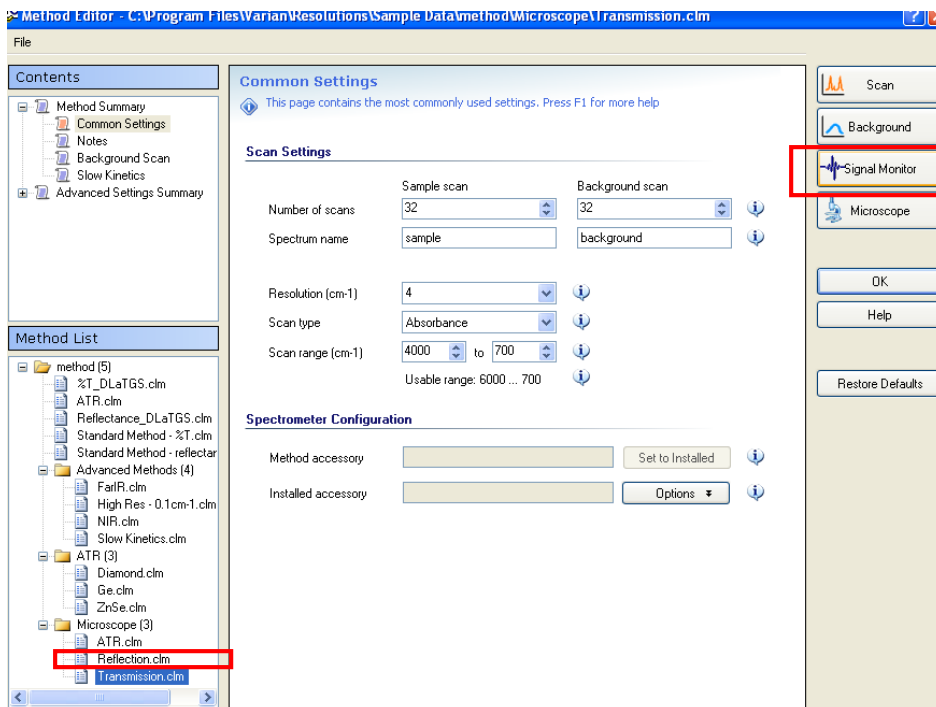
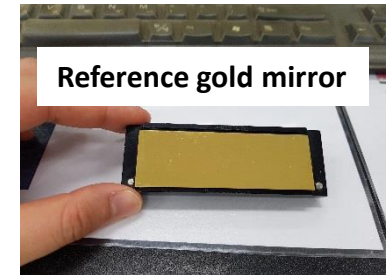
■ FT-IR Microscope_Transmittance

8. Place the sample on the stage and find the focus again.
9. Check the Monitor live spectrum.
10. Collect scan.



■ FT-IR Microscope_Reflectance

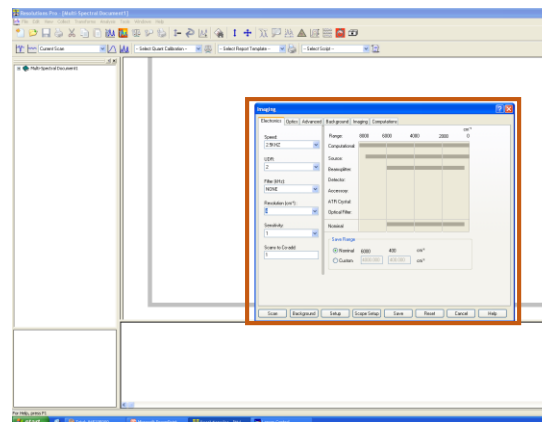
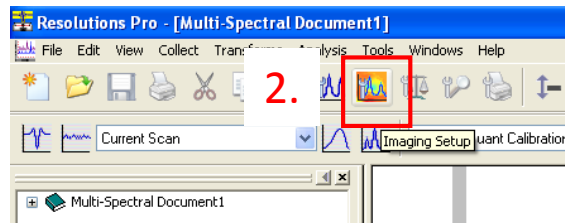
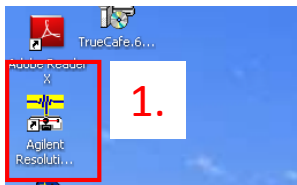
1. Place the reference gold mirror on the sample stage.
2. Find focus on surface of gold mirror.
Aperture size should be same with sample size.
3. Method editor – Method List – Microscope – Reflectance
– Signal monitor – Background – Save



4. Place the sample on the stage.
5. Find the focus and select the measurement point.
6. Check the spectrum with Monitor live spectrum.
7. Scan.

■ FT-IR Microscope_ATR image scan(mapping)

1. Open the Agilent Resolution Pro. Program.
 2. Click the Image Setup.
- You can change the Resolution, Sensitivity and Scan number.



Imaging

Electronics Optics Advanced Background Imaging Computations

Speed: 2.5KHZ

UDR: 2

Filter (kHz): NONE

Resolution (cm⁻¹): 4

Sensitivity: 1

Scans to Co-add: 16

Range: 8000 6000 4000 2000 0 cm⁻¹

Computational:

Source:

Beamsplitter:

Detector:

Accessory:

ATR Crystal:

Optical Filter:

Nominal

Save Range

☐ Nominal 6000 400 cm⁻¹

☒ Custom 4000.000 700 cm⁻¹

■ FT-IR Microscope_ATR image scan(mapping)

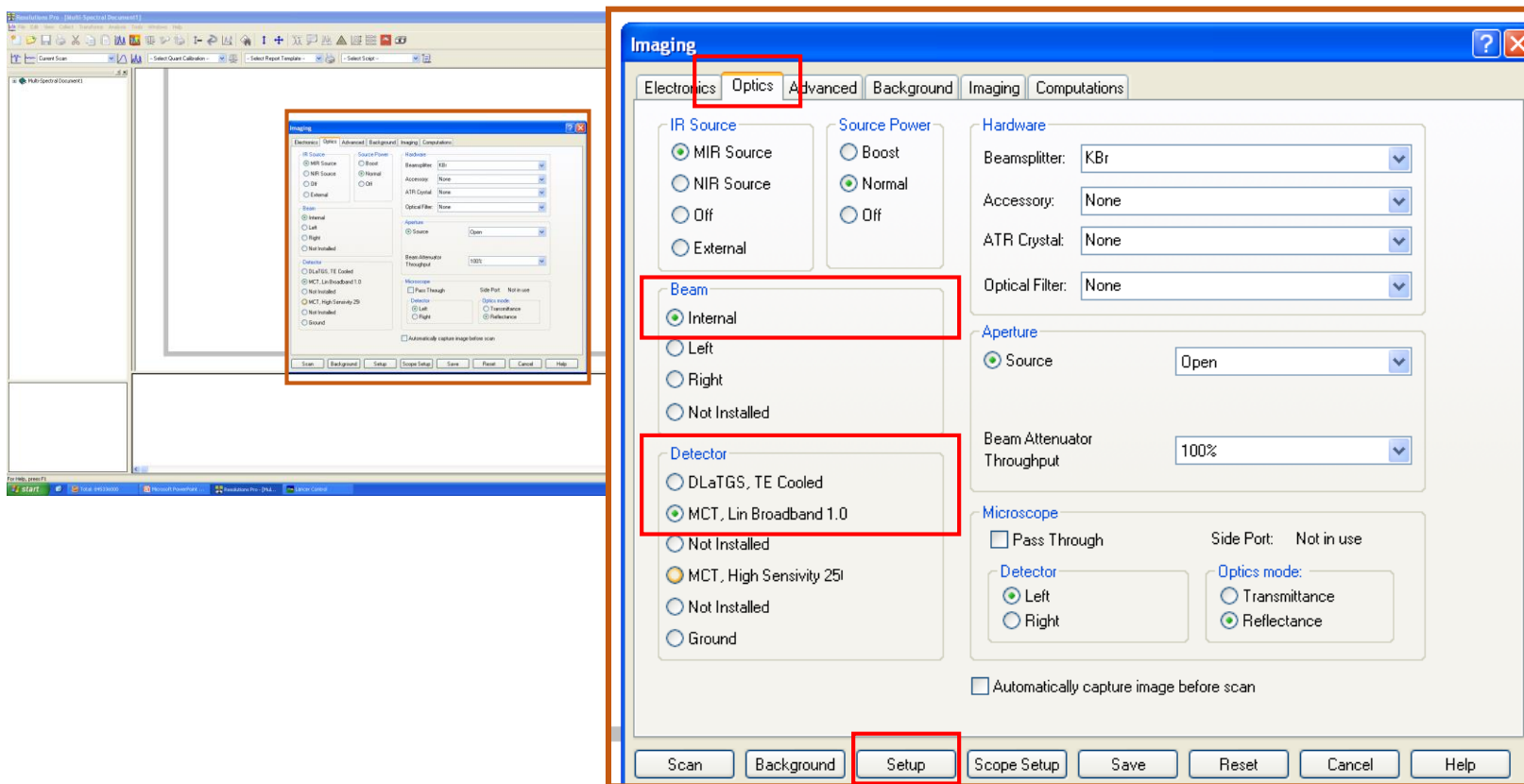
3. Find the beam line from the mainbody.

1) Select the Optics.

- Beam_Internal

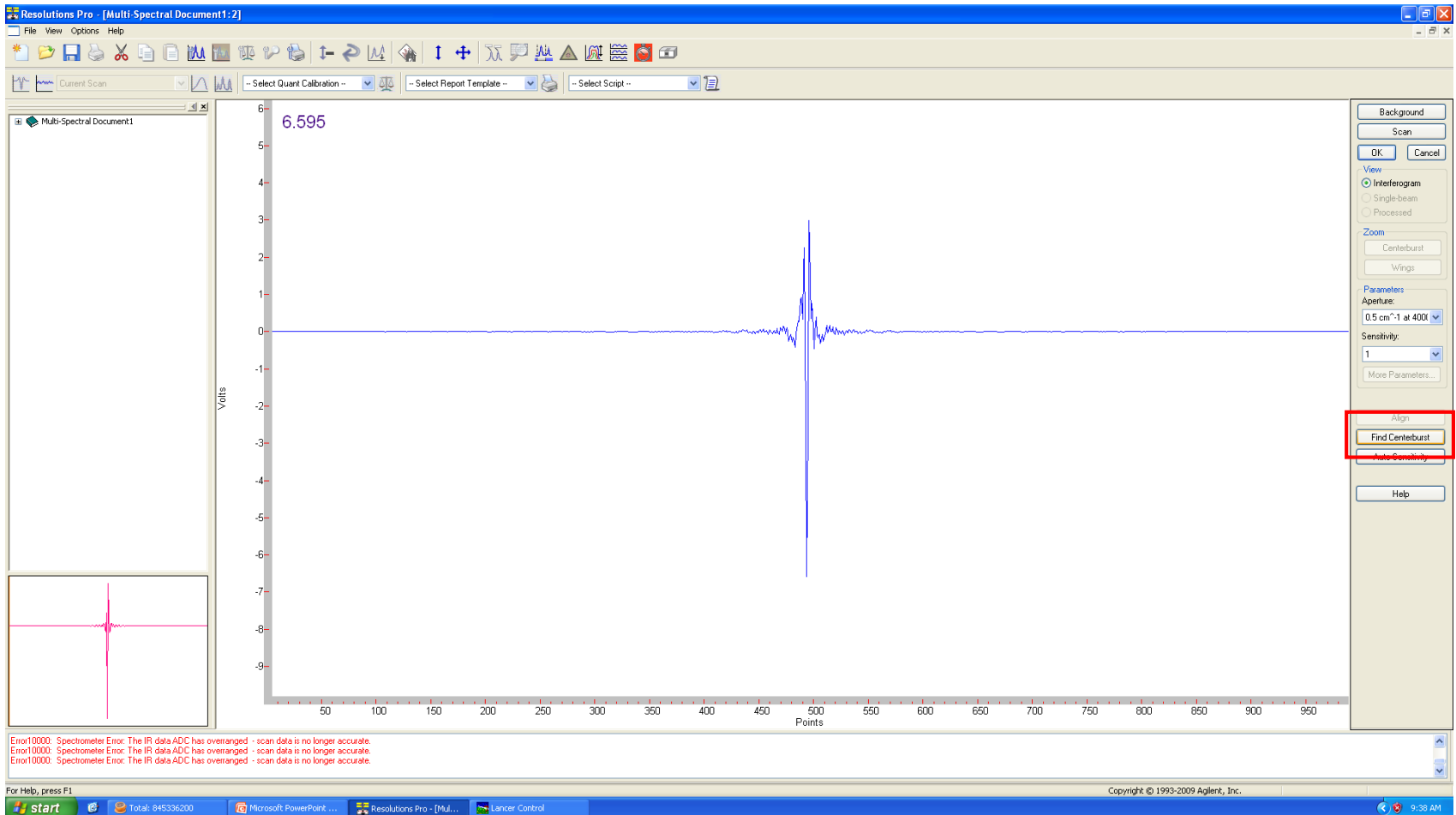
- Detector_Mainbody detector(DLaTGS, TE Cooled or MCT, Lin Broadband 1.0)

2) Click Set up.



■ FT-IR Microscope_ATR image scan(mapping)

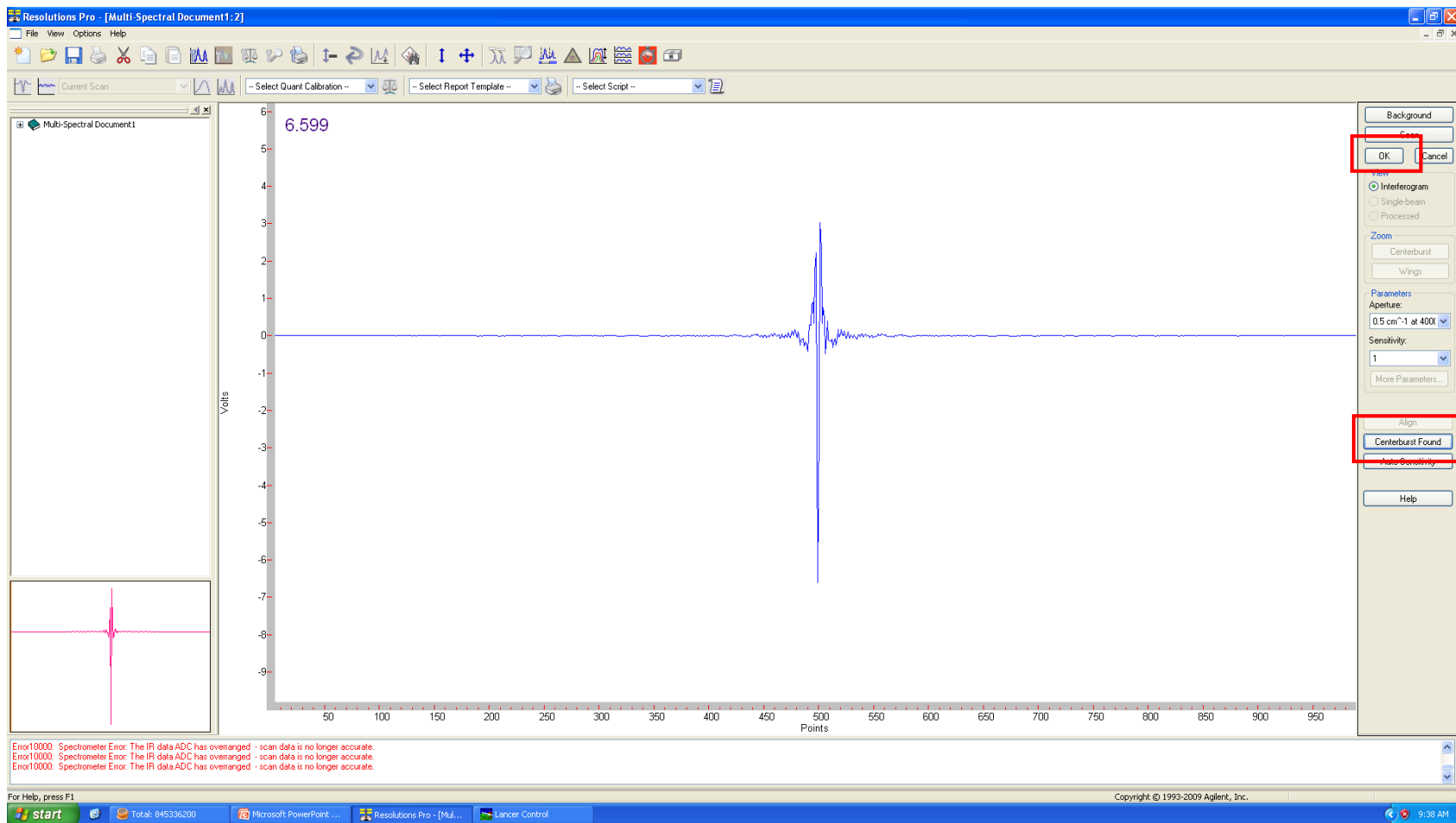
3) Click Find Centerburst.



■ FT-IR Microscope_ATR image scan(mapping)

4) Check Centerburst Found.

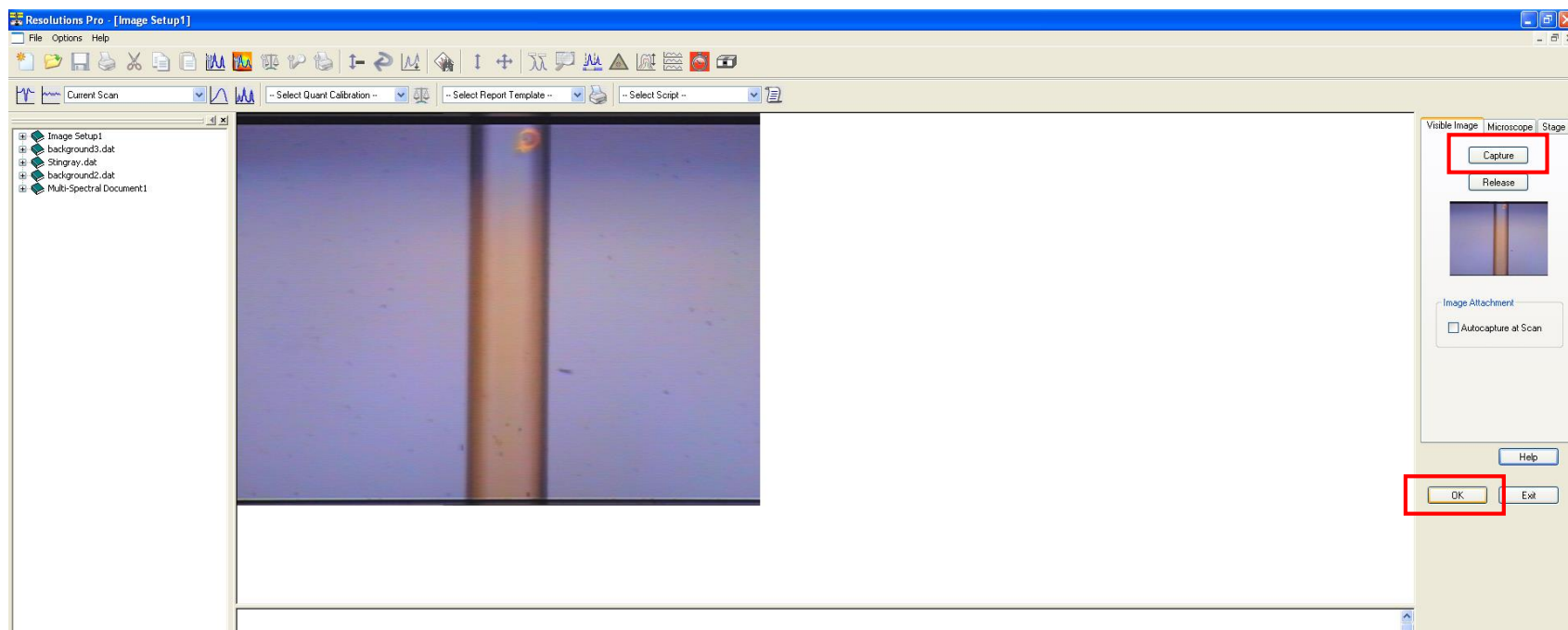
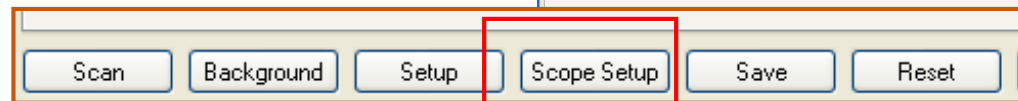
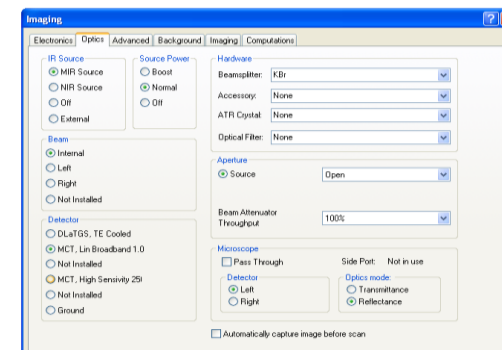
5) Click OK.



■ FT-IR Microscope_ATR image scan(mapping)

4. Imaging – Scope Setup

- 1) Find the focus of measurement point.
- 2) Change the aperture size and rotation of lens.
- 3) Click capture.
- 4) Click OK.

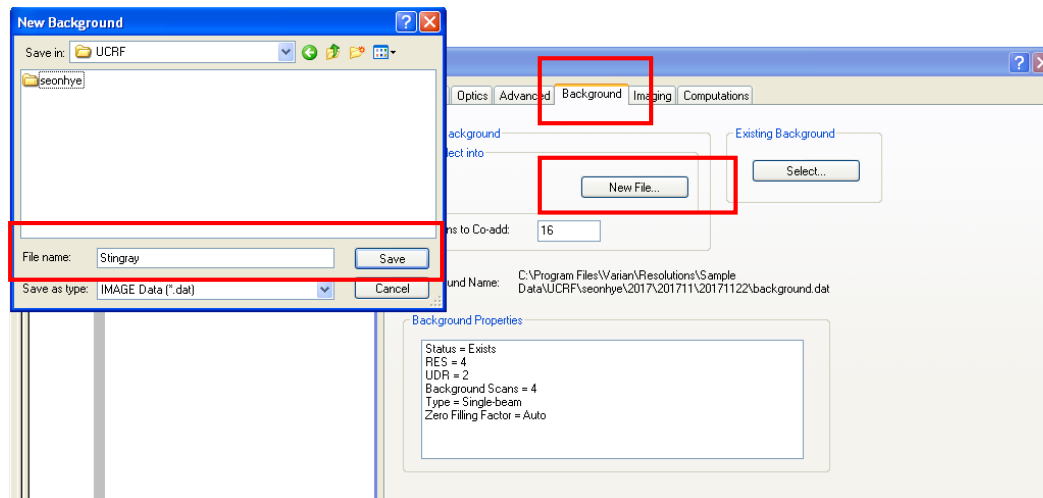


■ FT-IR Microscope_ATR image scan(mapping)

5. Put the ATR slide to the lens part.



6. Set the background file name and save condition.
Imaging – Background – New File - Save



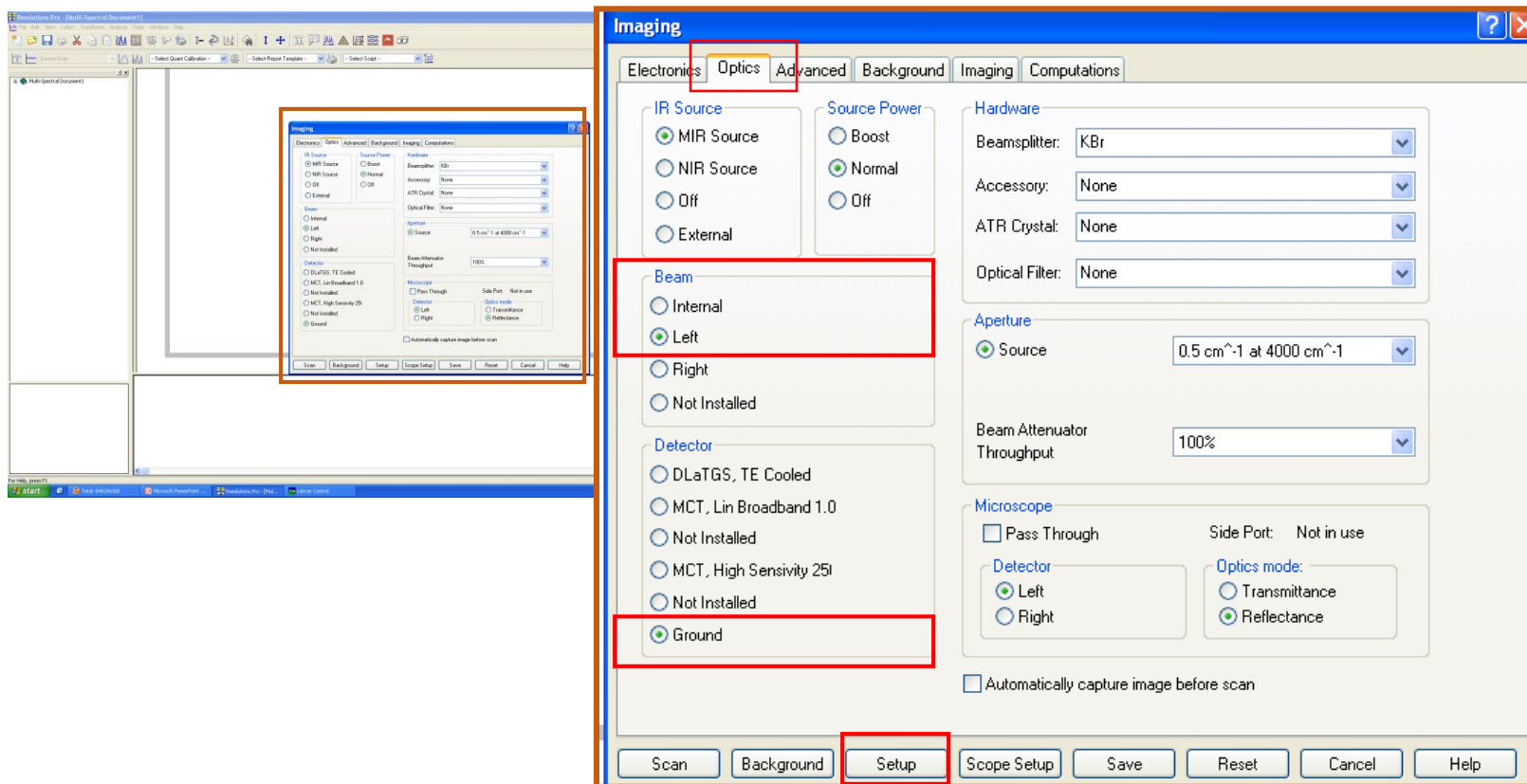
■ FT-IR Microscope_ATR image scan(mapping)

7. Imaging set up again.

1) Select the Optics.

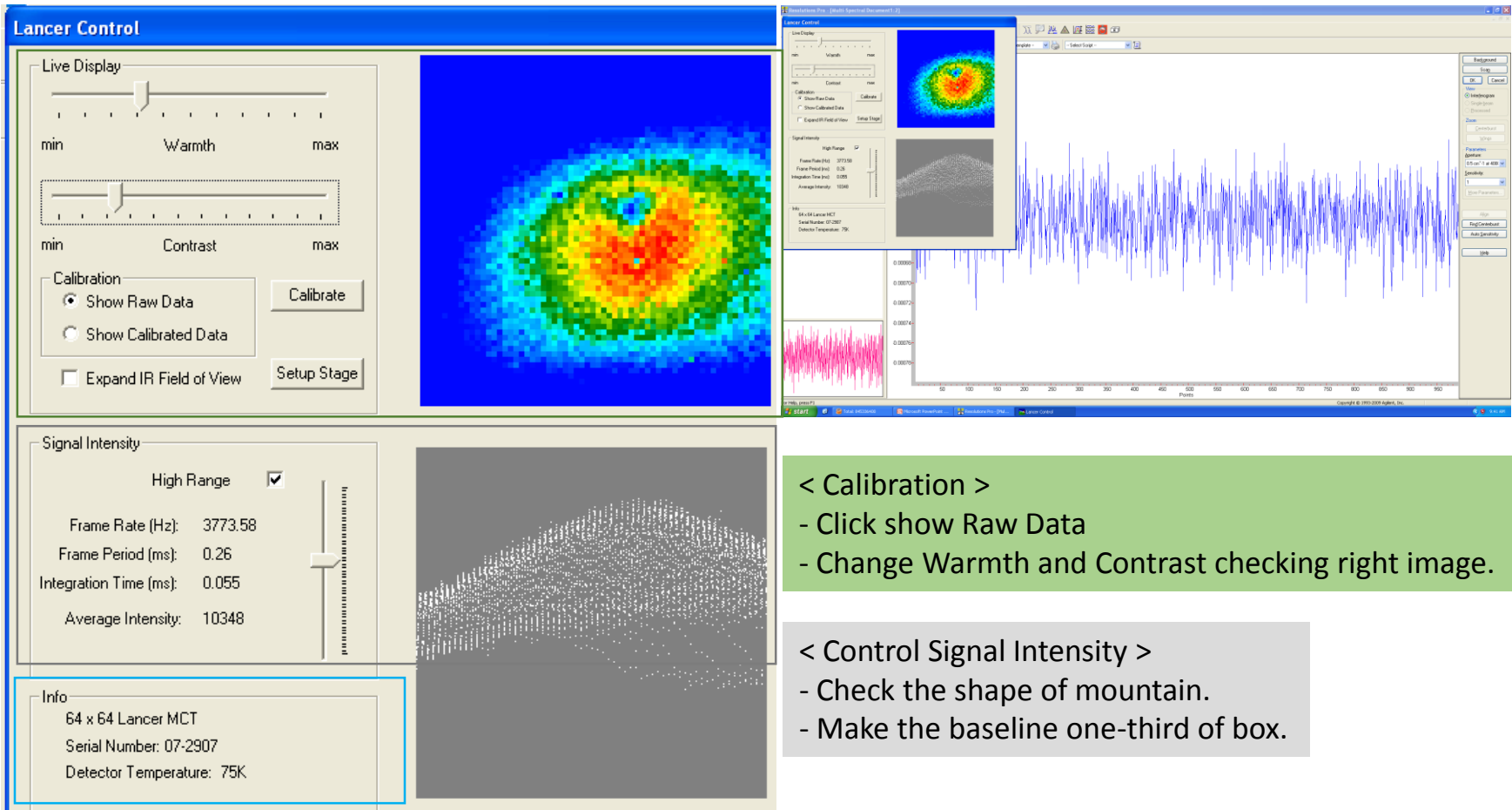
- Beam_Left
- Detector_Ground

2) Click Set up.



■ FT-IR Microscope_ATR image scan(mapping)

8. Lancer Control



< Calibration >

- Click show Raw Data
- Change Warmth and Contrast checking right image.

< Control Signal Intensity >

- Check the shape of mountain.
- Make the baseline one-third of box.

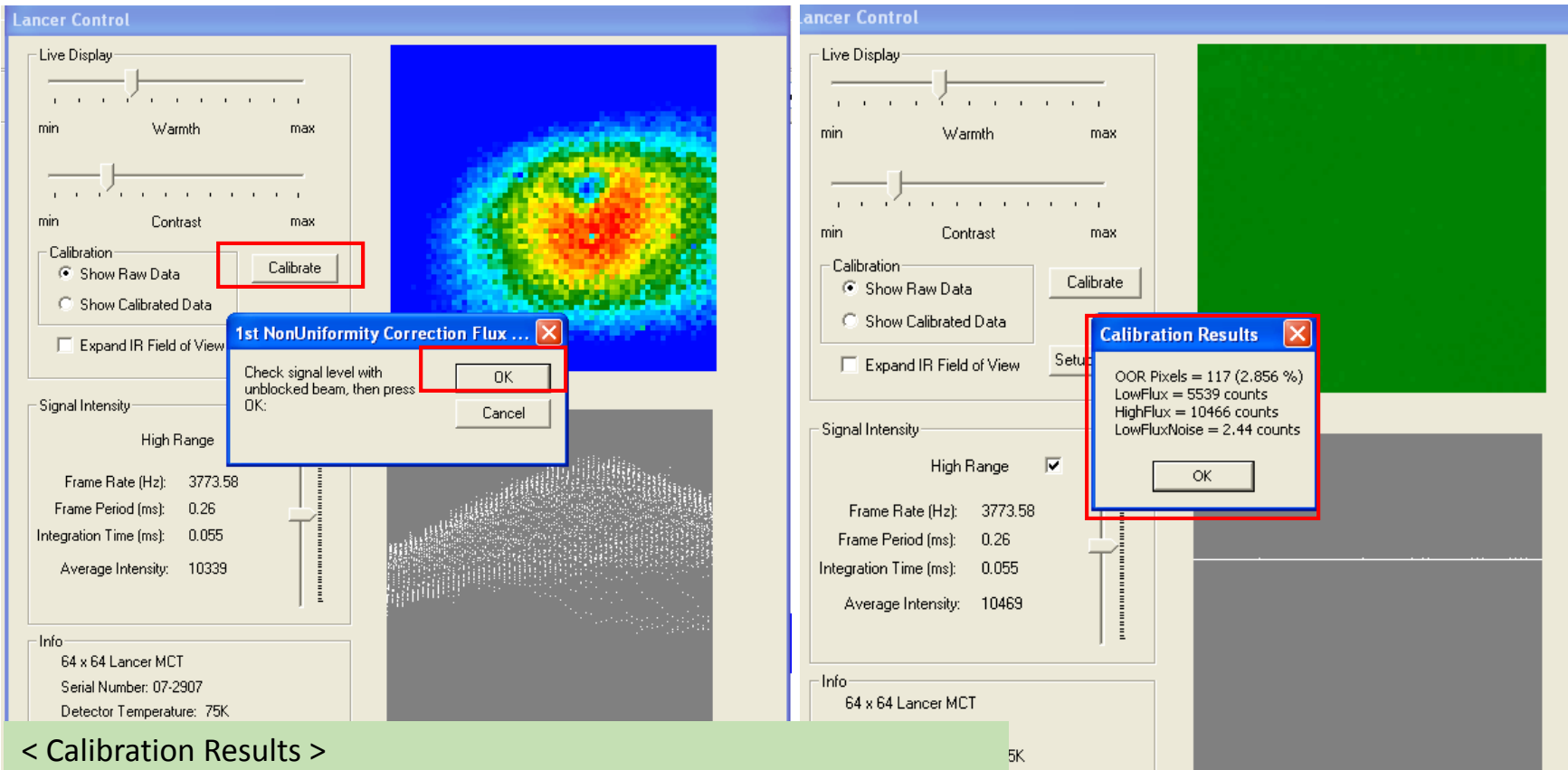
< FPA detector information >

You have to check the temperature.

When you see the red warning mark, please close the program and fill the liquid nitrogen again.

■ FT-IR Microscope_ATR image scan(mapping)

9. Click Calibrate – OK – Check the calibration Results – OK



< Calibration Results >

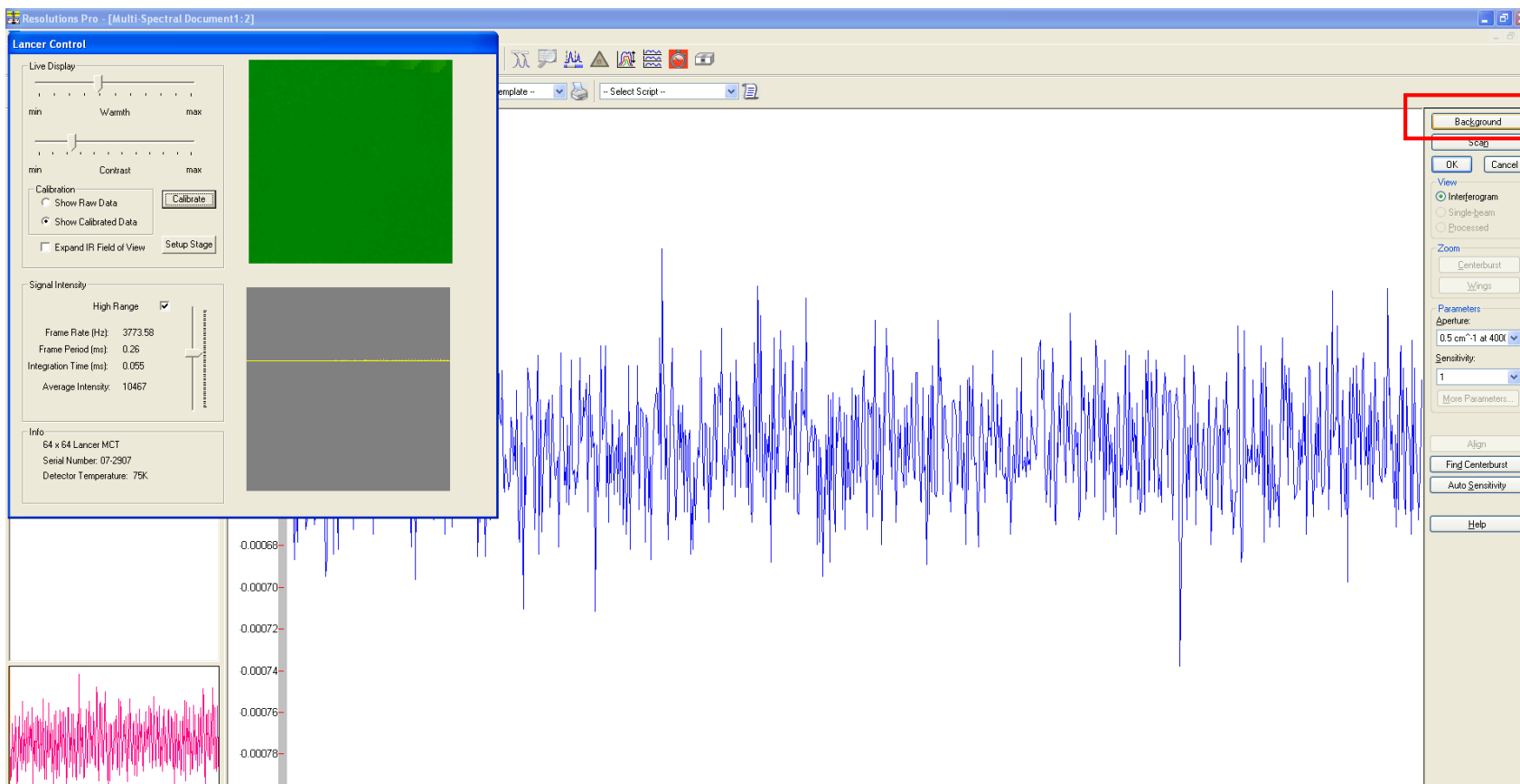
- **OOR Pixels** have to be below than **7%**.
- The difference value between **LowFlux** and **HighFlux** is about **3000**.
- ✓ Value > 3000, Calibration again after decrease Average Intensity .
- ✓ Value < 3000, Calibration again after increase Average Intensity.
- **LowFluxNoise** have to be below than **5**.

■ FT-IR Microscope_ATR image scan(mapping)

10. After calibration,

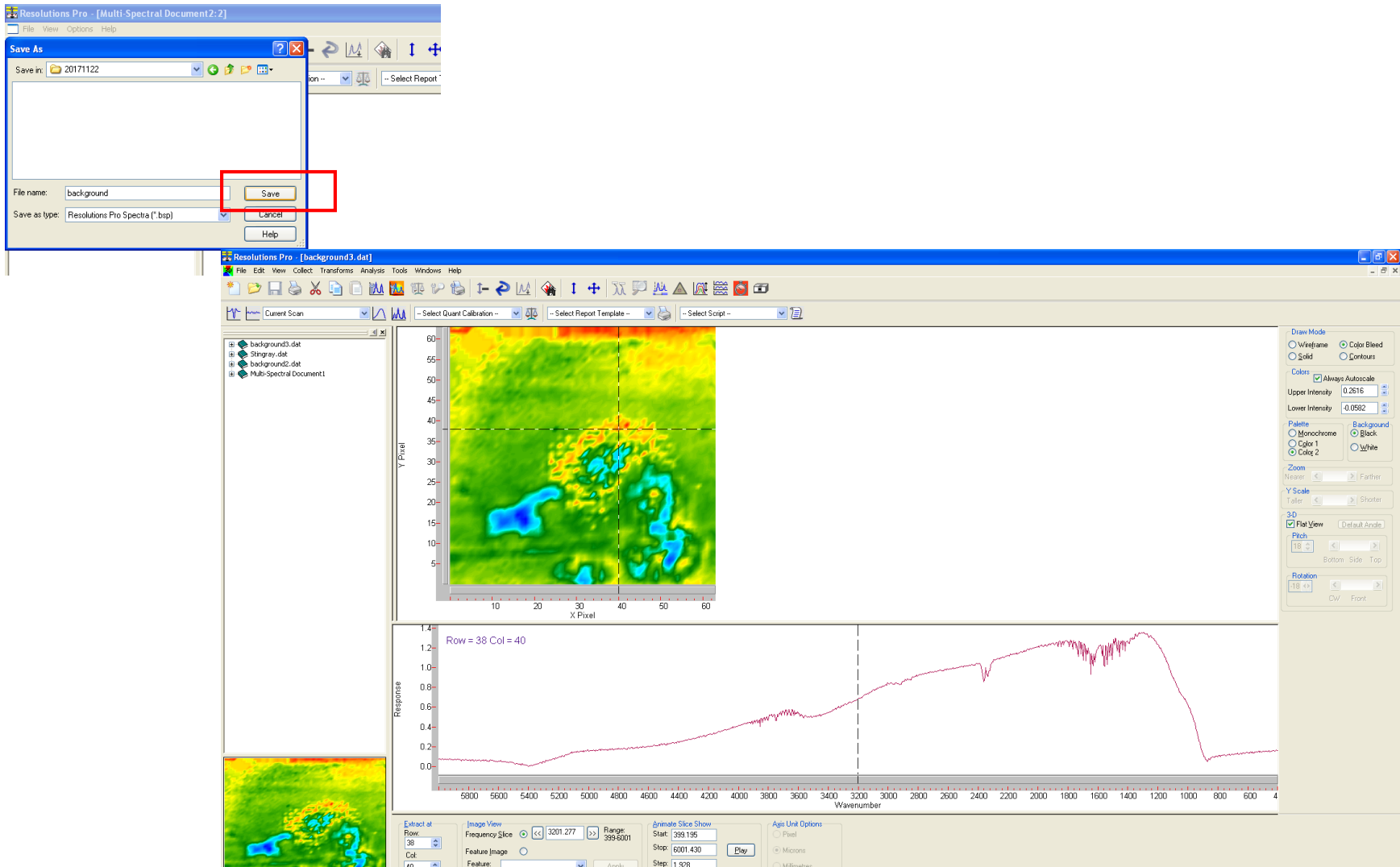
you can see the green color window at upper box and yellow line at lower box.
(uncalibrated data would be in white)

11. Click background.



■ FT-IR Microscope_ATR image scan(mapping)

12. Save the data

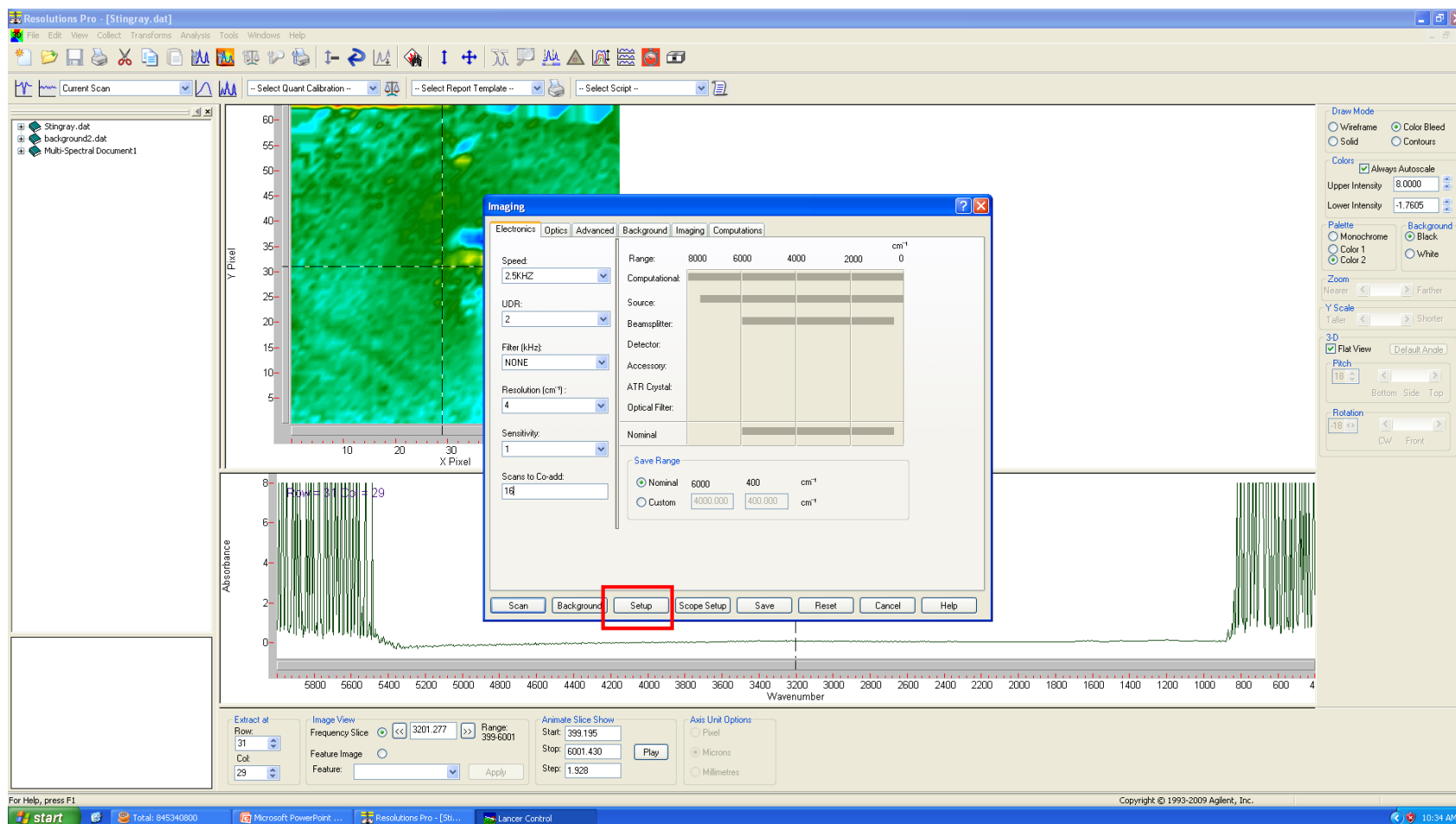


■ FT-IR Microscope_ATR image scan(mapping)

13. Place the sample on the stage and find the focus of measurement point.

14. Open Imaging Setup.

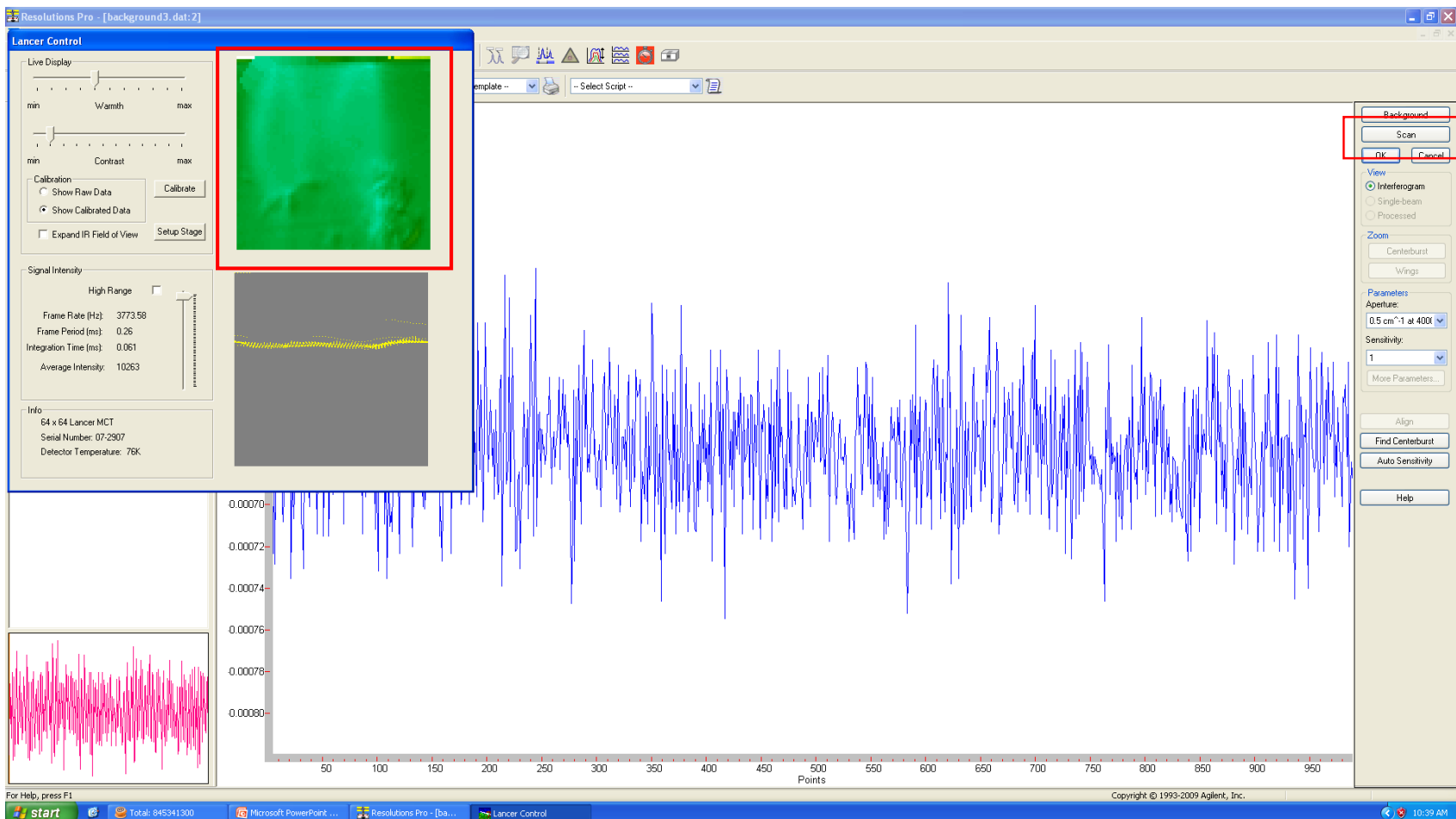
15. Click Setup.



■ FT-IR Microscope_ATR image scan(mapping)

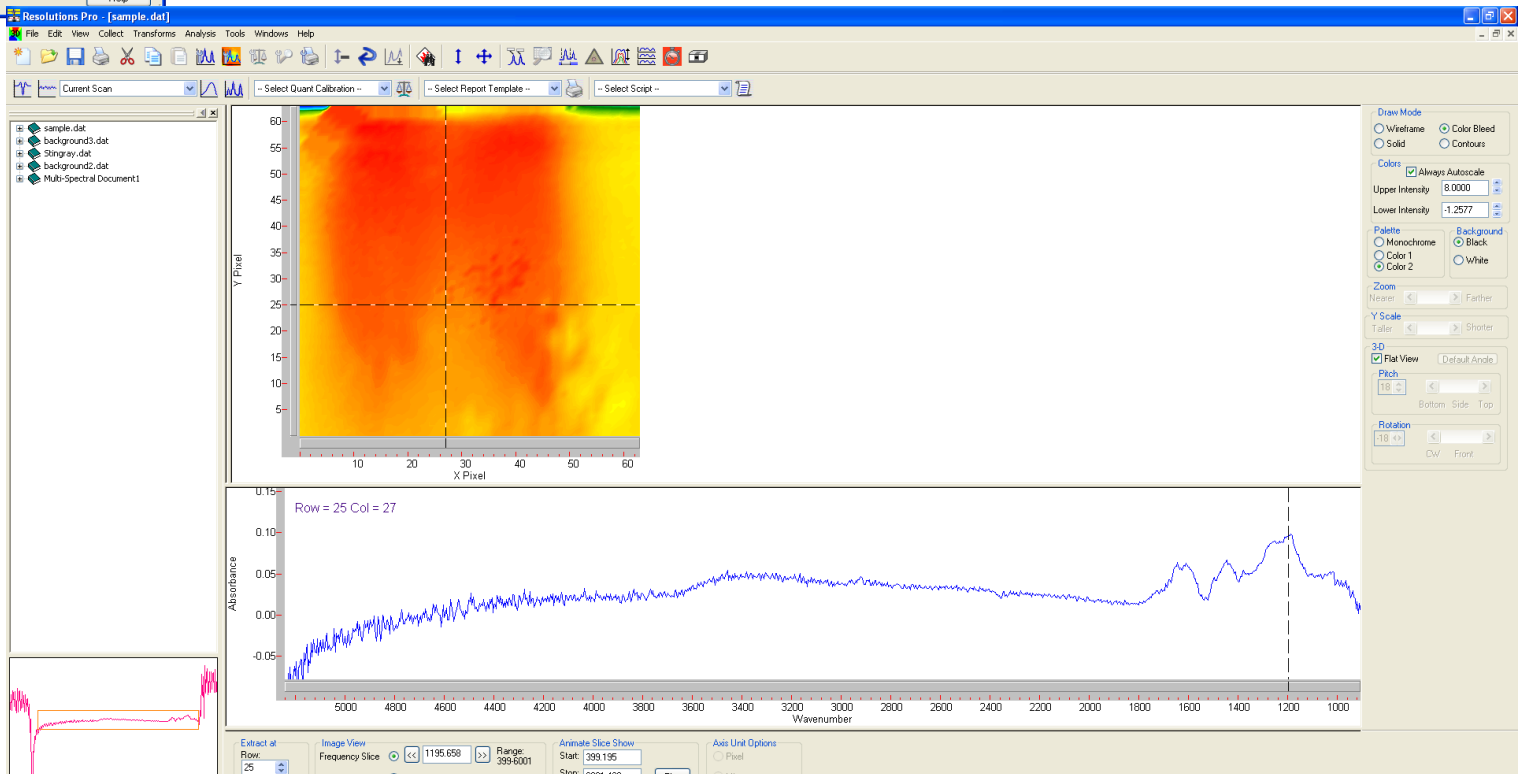
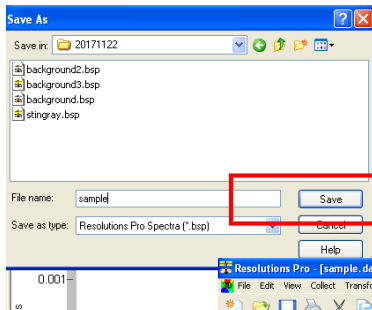
16. Move up the stage and check the Display image(green box) on Lancer Control.

17. Click **Scan** when you see the shape of sample surface.



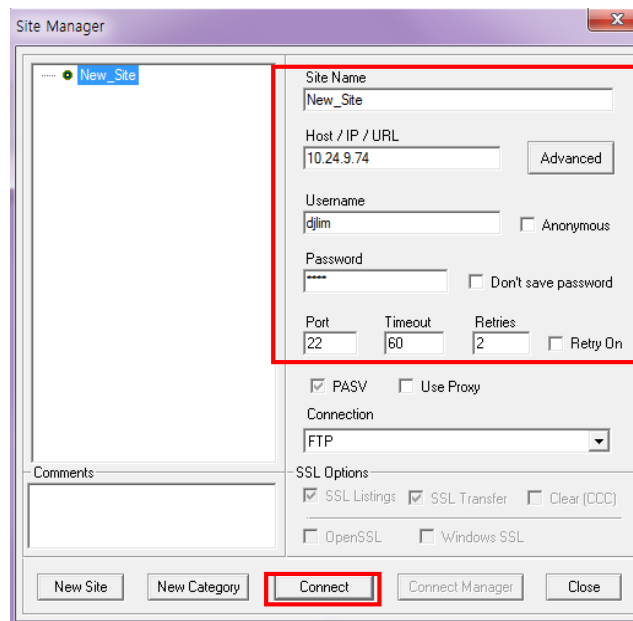
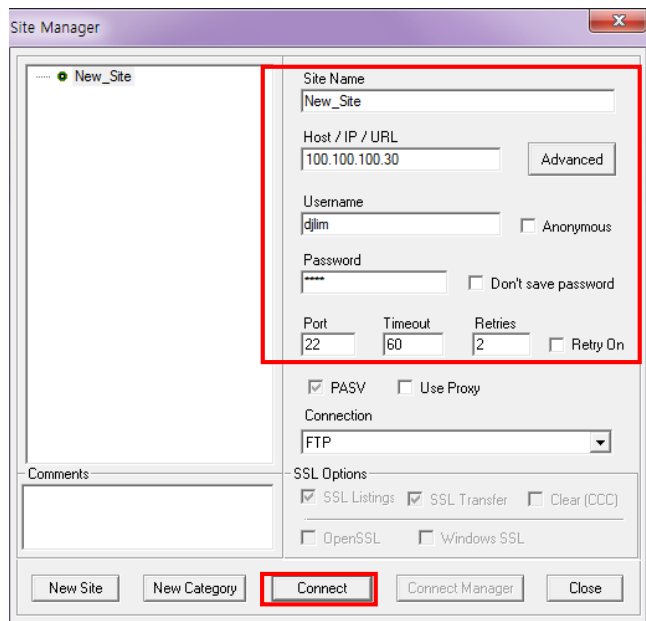
■ FT-IR Microscope_ATR image scan(mapping)

18. Save.



Data translation with Filezilla program

Open Fiezilla program_Open Site manager



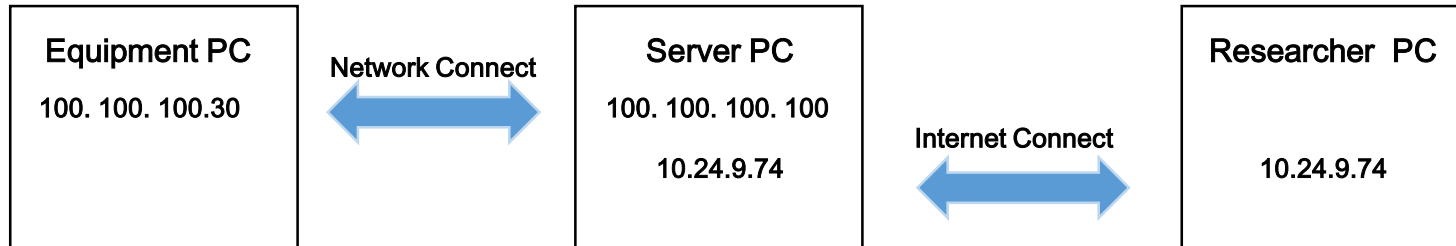
Host IP :
100.100.100.247 (in UCRF)
10.24.9.74 (from outside)

Username : djlim

PW : 0254

Port : 22

Data translation with Filezilla program



Core FTP LE - 10.24.9.42:22

File View Sites Manage Help

250 CWD command successful. "/" is current folder.
PwD
257 "/" is current directory.
PASV
227 Entering Passive Mode (10,24,9,42,146,187).
LIST
Connect socket #1020 to 10.24.9.42, port 37563...
150 Opening data connection
226 File sent ok
Transferred 4,523 bytes

UCRF PC or My PC

Server PC

Filename	Size	Date
..		09/13/13 09:07
Fax		08/13/13 11:28
Malvern Instruments		09/19/12 10:43
SAP		10/03/13 17:24
Scanned Documents		08/13/13 11:28
경남은행		08/07/13 15:49
Self 교육.xlsx	14 KB	09/26/12 17:59
XRF_data_보정.xls	112 KB	12/27/12 13:22
거래내역조회_2009_출력.pdf	45 KB	08/07/13 16:13
동위원소실가동률계산_박지혜.xlsx	32 KB	04/26/13 18:00

Filename	Size	Date
<..>		
..		
Company		10/04/13 11:...
Labs		10/06/13 20:...
Program		09/02/13 12:...
toengineer		01/30/13 10:...
touser		10/04/13 15:...
UCRF		10/02/13 17:...
UCRF 로그복관리		10/01/13 14:...
AFM 스캐너수리 요구내역.xlsx	0	04/27/13 15:...

6. FAQ

- Reservation control information
- Request for self user
- Reservation, cancel and input result
- Guideline for the Operation of the UCRF
- Penalty Points and Sanction Criteria

Reservation control information

Reservation time unit	Daily maximum reservation time	Cancelable timing	Fee (ATR)	
30 min.	3.0 hr	2.0 hr	Client	21,000/hr
			Self-user	15,000/hr

Create Account

www.ucrf.unist.ac.kr

UNIST | Central Research Facilities | About UCRF | Equipment Status | Data Room | Participation Space

1. Click [Sign up].

2. Click [UNIST Member].

3. Input [Portal id/pw]_Click [Confirm].

Please check your information.

4. Input professor name in [Principal investigation]

_Click [Professor search]_Click professor name.

5. Click [Create Account].

UNIST | UCRF

UNIST member | Industry member | External member

ID/E-mail: m*k*m@unist.ac.kr

Password: ***** Confirm

Name: 홍길동

Department: 연구지원본부

Student ID No. / Professor ID No. / Staff ID No.: 20*39

Contact: Extension 4064, Cell phone 010-**-****

Principal Investigator: 김교수 Professor Search

Create Account

Request for Self-user

www.ucrf.unist.ac.kr

Welcome 손선훈 | LOGOUT | **My Page** | Edit profile | KOR | ENG

Equipment Status | Data Room | Participation Space | 🔍

My Page
UNIST Central Research Facilities

- Request for Self-user** ▼
- Status of analysis request ▼
- Status of settlements ▼
- Status of education application ▼
- Status of tour application ▼
- Status of access permissions application ▼
- Status of penalty ▼

Home > MY PAGE > Status of analysis request

Status of analysis request

Equipment	Status	Application date	Result of analysis
<div><h4>Request for Self-user</h4><div><div>4-1 Materials Characterization Lab ▼</div><div>4-2 Surface Analysis ▼</div><div>4-3 Confocal Raman ▼</div><div>4-4 <button>Apply</button></div></div></div>			

After pass the test,

1. Login UCRF website.
2. Click [My Page].
3. Click [Request for Self user].
4. Select the equipment.
 - 1) Select [Materials Characterization Lab].
 - 2) Select [Surface Analysis].
 - 3) Select [Confocal Raman].
 - 4) Click [Apply].

portal.unist.ac.kr – Research Equipment– Equipment reservation/input result

Equipment Reservation

Detailed Navigation

- Equipment Reservation
- Equipment Reservation List
- Equipment Status

Favorite

Equipment reservation

Search condition

Reservation date: 2015.01.01 ~ 2015.08.26

☒ Reservation ☐ Input result ☐ Completed ☐ All

1st classification:

2nd classification:

Equipment name:

Equipment booking list

Select	Status	Sortation	Equipment name	Chief of research	Reservation date	Reservation time	Fee	1st classification	2nd classification name	Application date	Free_Test	Free_Longterm	Memo
<input type="checkbox"/>	Reservation	Admin	Confocal Raman	김영기	2015.08.17	13:00~16:30	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.04 18:44	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	AFM-Raman	김영기	2015.08.17	13:00~16:30	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.10 16:27	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	Confocal Raman	김영기	2015.08.17	09:00~11:30	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.04 18:44	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	AFM-Raman	김영기	2015.08.17	09:00~11:30	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.10 16:27	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.08.13	15:00~18:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.08.07 10:53	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.08.13	13:30~15:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.08.07 10:52	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.08.13	09:00~12:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.08.07 08:57	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	Confocal Raman	김영기	2015.08.12	15:30~17:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.07 17:15	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.08.12	10:30~11:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.08.07 14:57	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	Confocal Raman	김영기	2015.08.12	09:00~10:30	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.06 13:21	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.08.11	14:30~18:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.08.07 08:57	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	Confocal Raman	김영기	2015.08.11	13:30~14:30	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.05 11:42	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	Confocal Raman	김영기	2015.08.11	09:00~10:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.08.10 13:04	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.08.11	09:00~12:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.08.07 10:56	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.07.29	09:30~10:30	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.07.28 13:26	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Admin	FT-IR	김영기	2015.07.17	16:00~17:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.07.17 18:00	<input type="checkbox"/>	<input type="checkbox"/>	

Reservation

Equipment reservation help

Search condition Inquiry **Inquiry**

Reservation date: 2015.01.01 ~ 2015.08.04 ☒ Reservation ☐ Input result ☐ Completed ☐ All

1st classification: UMAL - 기기분석실 2nd classification: Surface Analysis Equipment name: Confocal Raman

Equipment booking list Application **Application**

Select	Status	Self	Equipment	Chief of research	Reservation date	Reservation time	Fee	1st classification	2nd classification name	Application date	Free_Test	Free_Longterm	Memo
<input type="checkbox"/>	Reservation	Self	AFM-Raman	김영기	2015.07.24	14:00~15:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.07.17 11:08	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	Confocal Raman	김영기	2015.07.24	14:00~15:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.07.17 11:07	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	FT-IR	김영기	2015.07.23	13:30~17:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.07.17 11:05	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	Confocal Raman	김영기	2015.07.22	13:00~14:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.07.20 11:20	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	Fluorometer	김영기	2015.07.20	14:00~14:30	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.07.17 11:03	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	Fluorometer	김영기	2015.07.20	13:30~14:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.07.16 16:55	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	FT-IR	김영기	2015.07.17	16:00~17:00	0.00	UMAL - 기기분석실	Spectroscopic Analysis	2015.07.17 18:00	<input type="checkbox"/>	<input type="checkbox"/>	

3

1

2

Application Close 참가<->펼치기

Select equipment

Client ID: shson35@unist.ac.kr 30678 / 손선혜 Subscriber: 30678 손선혜

1st classification: UMAL - 기기분석실 2nd classification: Surface Analysis 3rd classification: Confocal Raman

project information

Chief of research	Chief of research	Detail project number	detailed item	Executable amount
20032	김영기			0

Reservation control information

Reservation time unit	daily maximum reservation time	Reservation open timing	Cancellable timing	Fee
30 분	3.0 시간	5 일전	2 시간전	0.5 Hour 12,500 원

유의사항01 Laser power on/off
유의사항02 Keep clean lens to avoid contamination

Time/date	07/20(M)	07/21(T)	07/22(W)	07/23(T)	07/24(F)	07/25(S)	07/26(S)	07/27(M)	07/28(T)	07/29(W)	07/30(T)	07/31(F)	08/01(S)	08/02(S)
09:00~09:30	✓	✓												
09:30~10:00	✓	✓												
10:00~10:30	✓	✓												
10:30~11:00	✓	✓												
11:00~11:30	✓	✓												
11:30~12:00	✓	✓												
12:00~12:30	✓	✓												
12:30~13:00	✓	✓												
13:00~13:30	✓	✓												
13:30~14:00	✓	✓												
14:00~14:30	✓	✓												
14:30~15:00	✓	✓												
15:00~15:30	✓	✓												
15:30~16:00	✓	✓												
16:00~16:30	✓	✓												
16:30~17:00	✓	✓												

1. Select the classification and equipment


2. Select the time you want on white box.
Yellow box : my reservation
Red box : others reservation

3. Click [Application].

Reservation cancel

Equipment reservation

Search condition

 Inquiry

Reservation date: 2015.01.01 ~ 2015.08.04

1st classification: UMAL - 기기분석실

2nd classification: Surface Analysis

Equipment name: Confocal Raman

☒ Reservation ☐ Input result ☐ Completed ☐ All

Equipment Reservation cancel

 Application  Reservation cancel  Input result

Select	Status	Sortation	Equipment name	Chief of research	Reservation date	Reservation time	Fee	1st classification	2nd classification name
<input checked="" type="checkbox"/>	Reservation	Self	Confocal Raman	김영기	2015.07.24	14:00~15:00	0.00	UMAL - 기기분석실	Surface Analysis
<input type="checkbox"/>	Reservation	Self	Confocal Raman	김영기	2015.07.22	13:00~14:00	0.00	UMAL - 기기분석실	Surface Analysis

1. Select the reservation.
2. Click the [Reservation cancel].

Input result

After measurement, you have to input result instead of filling in log sheet

Equipment reservation

Search condition

Inquiry

Reservation date: 2015.01.01 ~ 2015.08.04

1st classification: UMAL - 기기분석실

2nd classification: Surface Analysis

Equipment name: Confocal Raman

Reservation ☒ Input result ☐ Completed ☐ All

Equipment booking list

Application Reservation cancel **Input result**

Select	Status	Sortation	Equipment name	Chief of research	Reservation date	Reservation time	Fee	1st classification	2nd classification name	Application date	Free_Test	Free_Longterm	Memo
<input checked="" type="checkbox"/>	Reservation	Self	Confocal Raman	김영기	2015.07.24	14:00~15:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.07.17 11:07	<input type="checkbox"/>	<input type="checkbox"/>	
<input type="checkbox"/>	Reservation	Self	Confocal Raman	김영기	2015.07.22	13:00~14:00	0.00	UMAL - 기기분석실	Surface Analysis	2015.07.20 11:20	<input type="checkbox"/>	<input type="checkbox"/>	

1. Select the reservation.
2. Click the [Input result].
3. Check the information and click [Save].

Save Close

Reservation information

Reservation number: 2015001217

Reservation date: 2015.07.24

Application date: 2015.07.17

Reservation time: 14:00~15:00

Client authorization: Self

Rate: 50

Equipment name: Confocal Raman

Project information

Chief of research	Chief of research	Detail project number	detailed item	Executable amount
20032	김영기			0

Fee

Cost	Unit quantity	Unit	unit amount	discount applying	Option applying	Amount	Fee	Rate	Amount
기본공정료	0.5	H	12,500	<input checked="" type="checkbox"/>		1.0	25,000	50	12,500
합계							25,000		12,500

Process condition

equipment status (problem and repair)

Mode **ATR**

Article 1 (Purpose)

This guideline is intended to provide detailed requirements for operating the Central Research Facilities at Ulsan National Institute of Science and Technology (UNIST) (hereinafter referred to as “UCRF”) in accordance with Article 10, Operational Regulations of Central Research Facilities at UNIST.

Article 2 (Scope)

This guideline shall apply to faculty, graduate students, undergraduate students and researchers at UNIST, as well as external clients, who request services from UCRF, and equipment managers.

Article 3 (Definitions)

Terms used in this guideline shall be defined as follows:

“Autonomous use” means that UNIST faculty members or students use UCRF's equipment without any help from the equipment manager.

“Autonomous user” refers to users who have qualifications for the “autonomous use” of the equipment in paragraph 1 above, according to procedures set by UCRF.

“Request for analysis and processing” is a request to the equipment manager to perform a series of analyses and processes, so autonomous users can use UCRF's common equipment to obtain the results of a test analysis or process.

Article 4 (Access Management)

- ① If any personnel want authorized access to laboratories with restricted access, they must fill out an application form and receive approval from the supervising professor and Center manager to register their ID.
- ② If any personnel needs to access laboratories for equipment maintenance and repair, they must be accompanied by a competent manager or have the manager's approval to gain access to the labs.
- ③ For laboratories that require safety training for personnel with access, approval for access will be withheld until they complete prior training, as specified for each laboratory.

Article 5 (Requests for Analysis and Processing)

- ① If a client requests for analysis and processing that can be supported by UCRF, the client should discuss with the equipment manager beforehand.
- ② A client who requests analysis and processing shall cooperate with the equipment manager in identifying the necessary information needed to maintain the normal operations and safety of equipment or facilities.
- ③ Analysis and processing services will be available to clients on a first-come-first-serve basis. In any special circumstances such as equipment inspection and repair is needed, requests for such services may be reserved or cancelled at the equipment manager's discretion.

- ④ If there are no special requests from the client, each manager may discard any specimens that are seven days or older after the results-notice date, and may also discard the outcome or results data produced by the analysis and processing service three months from the day of said notice or later.

Article 6 (Qualifications for Autonomous Use)

- ① Authorized persons who qualify for autonomous use shall be limited to graduate students, researchers, professors and authorized undergraduate students (with the supervising professor's approval) at UNIST.
- ② Qualifications for autonomous use shall be granted to any persons who satisfy the requirements specified by each laboratory (e.g. safety training, equipment user training, evaluation, etc.).
- ③ A list of autonomous users shall be updated every 6 months and shall be published on the UCRF homepage.
- ④ An autonomous user's qualifications may be cancelled if the equipment manager deems it necessary, or if the user does not frequently use the equipment (less than the minimum limit of 10 times in the last 6 months). In such cases, users may discuss with the manager and go through a re-orientation process to be qualified for autonomous use again.

Article 7 (Responsibility of Autonomous Users)

- ① Autonomous users should follow the instructions for using the equipment as they learned during the orientation. If there is something significant to report, they must discuss with a competent manager and help operate and maintain the safety of the research equipment facilities.
- ② Autonomous users will be liable for any accidents, equipment damage, failure and loss incurred as a result of their negligence when using the equipment.
- ③ Equipment reservations should be made a day (24 hours) prior to when they need to use the equipment, and may be cancelled no later than 12 hours before the booked start time. If a user wants to cancel their reservation, they must inform the equipment manager via phone or e-mail during regular work hours (weekdays: 09:00 - 18:00) or via e-mail during off-hours.
- ④ Any reservations that are made less than 24 hours in advance may be cancelled before the booked start time. If users want to cancel their reservation, they must inform the equipment manager via phone or e-mail during regular work hours (weekdays: 09:00 - 18:00) or via e-mail during off-hours.
- ⑤ After using the equipment at night or during the equipment manager's off-hours, authorized users should make sure the laboratory is put back in order, the lights are turned OFF, and the entrance door is properly locked before leaving.

Article 8 (Restrictions for Autonomous Use)

- ① For the convenience of other users, a comfortable research environment, and to promote proper use of the equipment, UCRF may sanction users.
- ② Sanction criteria from the above paragraph 1 shall follow "Table 1. Penalty Points and Sanction Criteria for Users of Common Equipment."

Article 9 (Billing for Test Analysis Fees)

- ① Clients or autonomous users will receive bills for test analysis fees in the following month after the analysis and processing has ended, and may only pay for these bills to UCRF's bank accounts.
- ② Clients or autonomous users shall follow the specified procedures to pay bills charged under the standards of test analysis fees in accordance with Article 8, "Operational Regulations of Central Research Facilities at UNIST."
- ③ The standards of test analysis fees, as stipulated in Article 7, Operational Regulations of Central Research Facilities at UNIST, may be provided to clients or users before request or use.
- ④ If this is their first request or first time using the equipment, clients and users should submit copies of their business license and their bank book to UCRF's administrative offices.
- ⑤ When there is any change to the business license, they shall inform the administrative manager of the change and send a copy of the new business license to the manager.
- ⑥ Bills for test analysis fees shall be issued by UCRF's administrative office, and clients or users shall pay the bill to UCRF no later than 1 month after the bill is sent to them. If the payment is overdue, UCRF may stop supporting services for users and laboratories in arrears.
- ⑦ If more time is required for analysis and process due to negligence on the part of clients, additional test analysis fees may be charged.

Penalty Points and Sanction Criteria

	Behaviors subject to penalty points	Penalty pts
[Eligibility to use equipment]		
1	Unauthorized use of equipment without permission	5
2	Use of equipment without a reservation	3
3	Someone other than the equipment lessee used the equipment	3
[Reservations for using equipment]		
4	Reserved and used equipment outside of permitted hours	1
5	Use of equipment beyond the time reserved without making another reservation beforehand for extra time	1
6	Failed to use the equipment during the reserved time and did not cancel reservation in advance	3
7	Cancelling reservations for equipment after the cancellation deadline, under Article 7, Guideline for the Operation of the UNIST Central Research Facilities (UCRF)	1
8	Use of any equipment without giving a prior notice to the equipment manager, after making a reservation	1
[Careless behaviors]		
9	Using functions on the equipment that are not permitted	3
10	Failure to promptly notify the manager of any errors or failures detected during use	3
11	Negligence that resulted in damages or failure to the equipment	5
12	Negligence that resulted in loss or damage to an equipment component or part	5
13	Failure to record in the equipment usage log after using any equipment, or misrepresentation or partial representation of the facts	1
14	Failure to provide specimen information required by the equipment manager to ensure normal operations and safety of equipment or facilities, thus resulting in damage or failure to the equipment	3
15	[Careless behaviors]	3
16	Using functions on the equipment that are not permitted	5
17	Failure to promptly notify the manager of any errors or failures detected during use	5

Penalty Points and Sanction Criteria

Classification	Penalty pts.	Follow-up actions
(Individual users of equipment)		
Sum up penalty points imposed to individuals	≥ 5 points	Equipment manager will notify user(s) and their supervising professor by email of their penalty points total, and shall post the details of their penalty points on the bulletin board of the equipment room. Users with penalty points 8 points or higher may not use the relevant equipment for 3 months.
	≥ 8 points	Equipment manager will notify user(s) and their supervising professor by email that the user(s) may not use the relevant equipment for 3 months until they complete the re-orientation course; will also forward an official notice to their supervising professor; and will post details of their penalty points on the bulletin board of the equipment room.
(User's laboratory)		
Sum up penalty points imposed on the students in the laboratory for the same equipment in the same laboratory	≥ 12 points	Equipment manager will notify the user(s) and their supervising professor by email that user(s) with penalty points 15 points or higher may not use the relevant equipment in the laboratory for 3 months.
	≥ 15 points	Equipment manager will email the supervising professor to inform that the user(s) may not use the relevant equipment in the laboratory for 3 months; will also forward an official notice to their supervising professor; and will post the details of their penalty points on the bulletin board of the equipment room.
Sum up penalty points imposed on the students in the laboratory for all UCRF equipment in the same laboratory	≥ 20 points	UCRF will notify students and their supervising professor by email that the user(s) with 25 penalty points or higher may not use any UCRF equipment in the laboratory for 1 month.
	≥ 25 points	UCRF will notify students and their supervising professor by email that user(s) may not use any UCRF equipment in the laboratory for 1 month; will also forward official notice to their supervising professor; and will post details of their penalty points on the bulletin board of UCRF.