國家同步輻射研究中心
National Synchrotron Radiation Research Center

紅外光譜實驗站
使用者手冊

User Manual of BL 14A1,

Infrared Microspectrocopy Operation Guide

顯微鏡
(μ Scope)

主機
(main)

Synchrotron-based FT-IR system

Table of Contents

I. **Safety Instructions and Precautions**

1. All users must complete NSRRC safety training and comply with all experimental regulations.

2. Samples brought into the facility must comply with NSRRC safety standards and be correctly documented on the safety form.

3. For special or hazardous samples, users must consult the beamline spokesperson or manager at least two weeks before the experiment. Do not bring hazardous samples without approval.

4. Use liquid nitrogen carefully to avoid overfilling, which could damage instruments. (Refill every 8 hours; allow 15–20 minutes for thermal equilibrium.)

5. Maintain a clean and organized work environment. Return all items to their proper place after use.

6. Be aware of potential oxygen deficiency due to liquid nitrogen. If the oxygen detector alarm sounds, open the door and exit the lab. Resume work only when the alarm stops. Contact the beamline manager if unsure.

7. Turn off lights when leaving to conserve energy. After the experiment, turn off the IR source and the computer (main unit and monitor), but **do not switch off** the microscope or spectrometer.

8. Do not use your USB devices to access or store data.

Contact Information:
Infrared Beamline (BL14A1), National Synchrotron Radiation Research Center
Tel: (03) 578-0281 ext. 1141, 1142
Beamline Spokesperson: Dr. Yao-Chang Lee ext. 7333, Mobile: 0921250566
Beamline Manager: Ms. Pei-Yu Huang ext. 7329, Mobile: 0911326696

II. Principle of the Synchrotron-based Infrared Microspectroscopy

This experiment is conducted at the BL14A1 Infrared Microspectroscopy beamline of the National Synchrotron Radiation Research Center (NSRRC). The beamline integrates a synchrotron radiation source with a Fourier-transform infrared (FT-IR) spectrometer (Nicolet 6700, Thermo-Nicolet Instruments, Madison, WI, USA) and a confocal infrared microscope (Continuμm, Spectra-Tech, Oak Ridge, TN, USA).

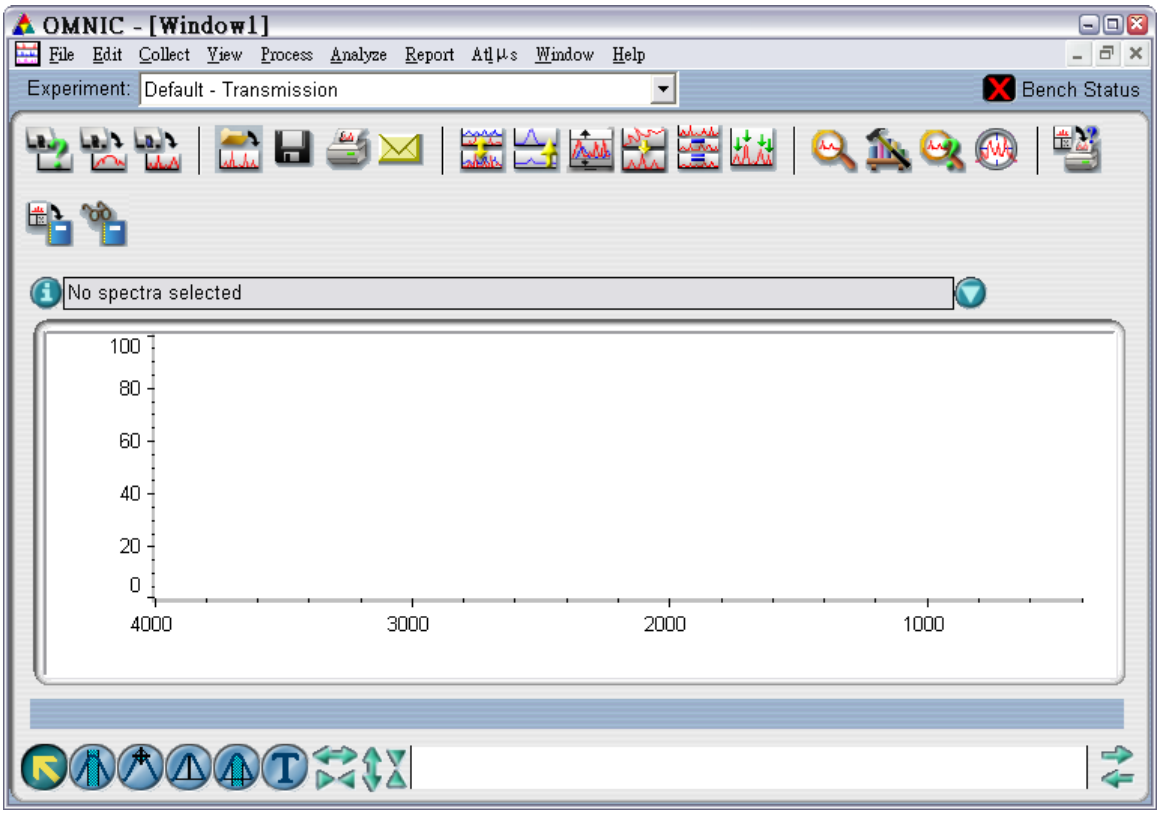
The FT-IR spectrometer is equipped with a potassium bromide (KBr) beam splitter, and the infrared signals are detected using a liquid nitrogen-cooled mercury cadmium telluride (MCT) detector operating at 77 K. This detector has a sensing area of 50 × 50 μm², a detectivity (D*) of 5.0×10^{10} cm·Hz$^{1/2}$·W^{-1}, and covers a spectral range from 4000 to 650 cm^{-1}.

The synchrotron infrared beam is guided to the endstation and modulated by the FT-IR spectrometer before being focused onto the sample using a 32× reflective Cassegrain objective lens within the confocal microscope. The focused beam has a full width at half maximum (FWHM) of approximately 13 × 10 μm².

All interferograms obtained are corrected using the Happ-Genzel apodization function to minimize artifacts introduced by the finite range of the Fourier transform.

Source	Acceptance Angle H × V (mrad²)	Energy (cm^{-1})	Beam Size FWHM H × V (micron²)	Interferometer Resolution (cm^{-1})	Flux (photons/ sec)
BM	70 × 30	4000-650	13 × 10	0.125	2×10^{12}

III. **Main Interface of OMNIC 9.0 Software**



The commonly used main functions in OMNIC software are listed below:
Main Functional Buttons

 Experiment Setup: Set up parameters for the experiment

 Collect Background: Acquire background spectrum

 Collect Sample: Acquire sample spectrum

Shortcut Keys:

Ctrl+D→ Set the upper and lower bounds of the spectral range
Ctrl+F→ Display the Fit Window
Ctrl+H→ Hide the spectrum (Hint)

一、 Experiment Parameter Setup

After launching the software, click  to enter the Experiment Setup screen.

Start by selecting the Bench tab and configure the following parameters based on experimental needs:

- **Sample Compartment**→Choose the measurement system

Main: main body of the FTIR system

μScope%R: microscope in reflectance mode

μScope%T: microscope in transmittance mode

- **Source**→Select the light source

IR (Globar source) → Beam size: 50 × 30 μm^2

External (SR source) → Beam size: 13 × 10 μm^2

- **Recommended Range**→Set the measurement wavenumber range

4000–650 cm^{-1} (for MCT/A Detector)
4000–400 cm^{-1} (for DTGS Detector)

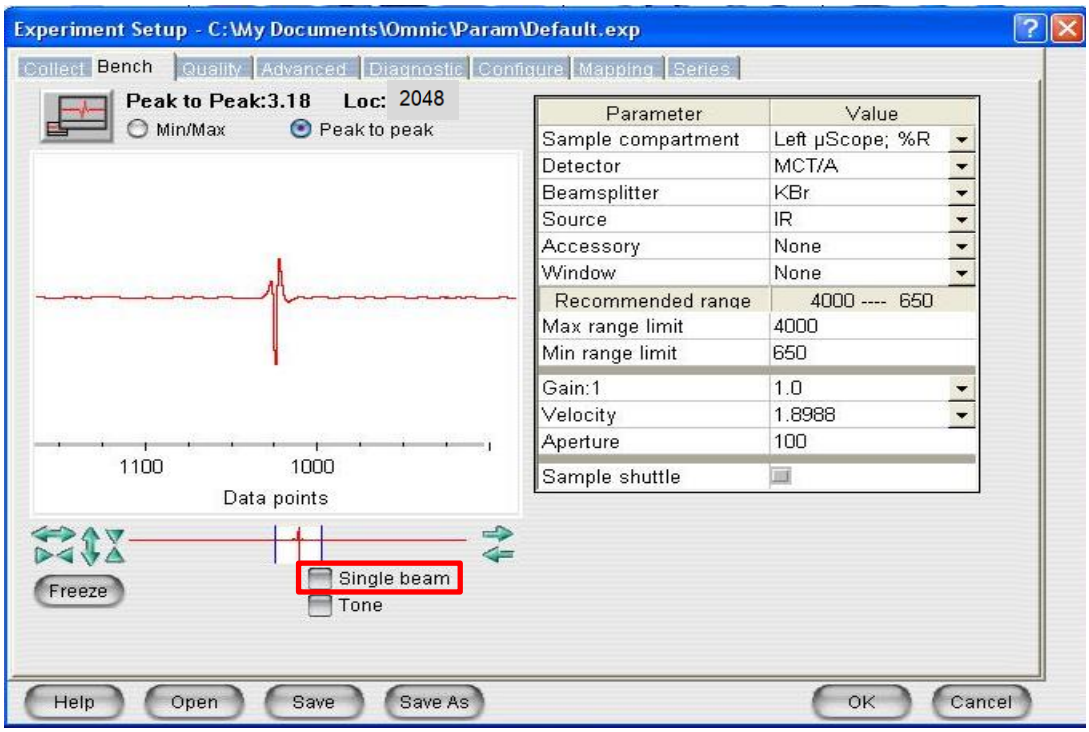
- **Velocity**→Set the moving mirror velocity

(For 4 cm^{-1} resolution, the moving mirror must move 0.125 cm per wave)
MCT/A velocity : 1.8988 cm/s
DTGS velocity : 0.4747 cm/s
Detector Area:
MCT/A = 50 × 50 μm^2
MCT/B = 250 × 250 μm^2

- **Aperture**→Aperture diameter inside the FTIR main body

The aperture is positioned at the focus of the IR source. Changing the aperture size alters the IR beam diameter. At higher spectral resolutions, the mirror travel distance increases. Since the internal IR source is incoherent, beam divergence increases with path length. If the beam size exceeds the mirror surface, significant phase differences between the center and edge beams may occur, degrading spectral resolution.

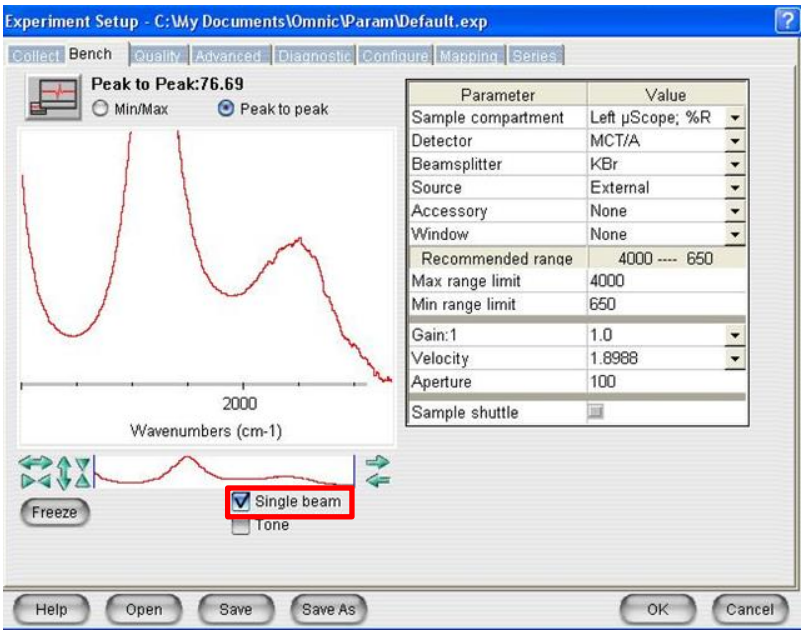
Therefore, when the resolution is increased, the system automatically calculates and recommends a suitable aperture size.



Peak to Peak

Click ☑Single Beam → This converts the time-domain

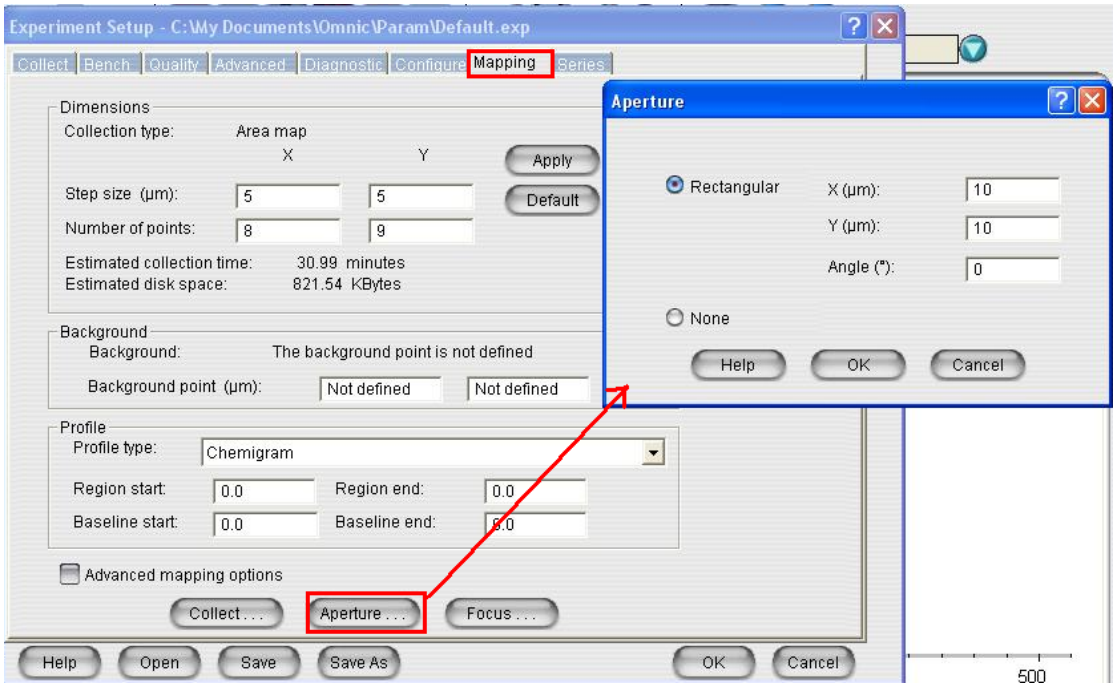
interferogram into a frequency-domain spectrum using Fast Fourier Transform (FFT).



Aperture Selection Guidelines:

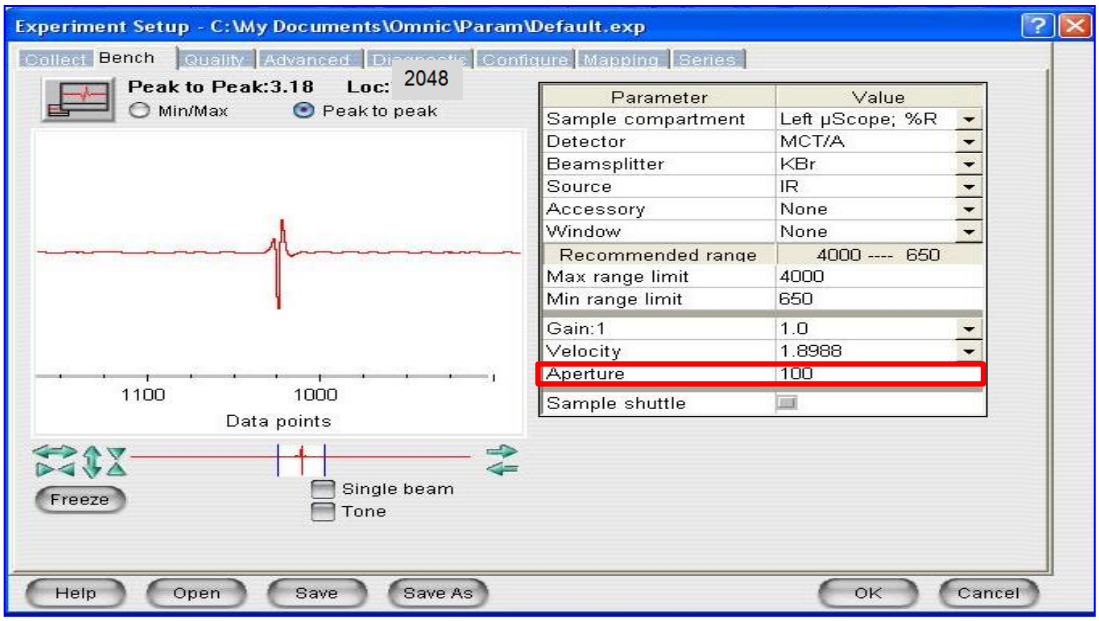
(a) If using the microscope (µScope%R or µScope%T):

Set the aperture value in the Mapping tab.

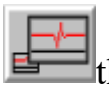


(b) If using the FTIR main unit (main):

Set the aperture value in the Bench tab.

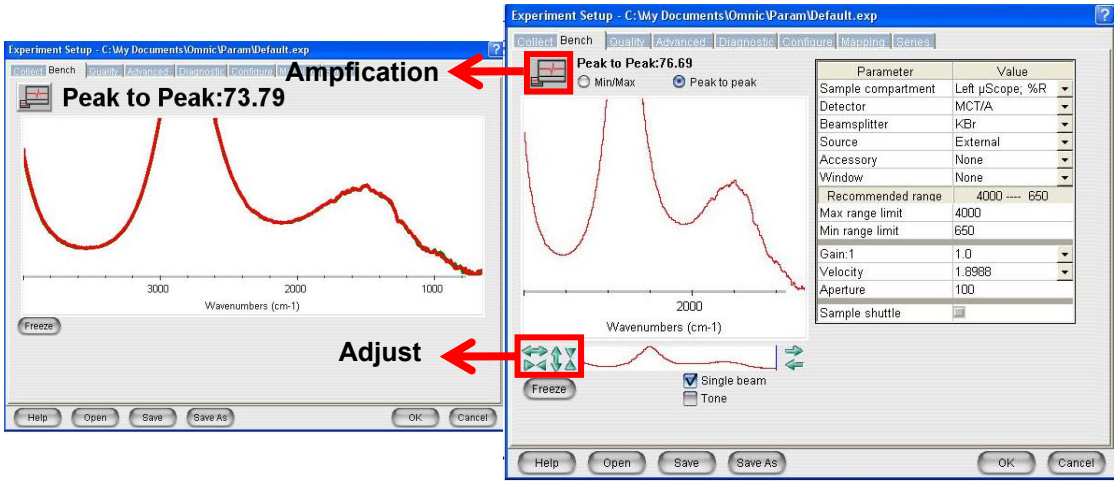


IV. Signal Adjustment, Focusing, and Interferometer Alignment

In the Bench tab, check  the following options to aid in signal adjustment:

Then click 

the button to open the spectrum window in a separate view, making signal optimization easier, as shown below.



When clicking the measurement button, the spectrum displayed in green will remain fixed, representing the signal under the current focus. This serves as a reference for focusing adjustment. Normally, a clear signal will appear when the sample is in focus. Adjust the focal position to the point where the signal is strongest.

If no signal is detected even when the sample (or the slide) surface is in clear focus under the microscope, check the following before proceeding to interferometer alignment:

a) Ensure liquid nitrogen has been refilled (every 8–10 hours)
b) If using the SR source, check whether the storage ring is operating normally (1.5 GeV, 360 mA)

c) When shutting down, switch the light source from External (SR) → IR → OFF in order.

V.Spectral Scanning and Parameter Settings

After selecting the Collect tab, configure the following parameters:

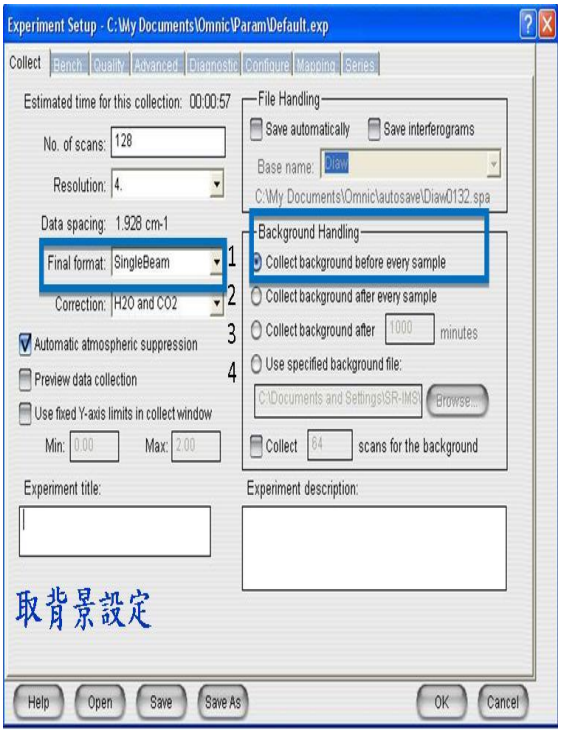
- Scan: 16 (Number of scans; recommended to use powers of 2)

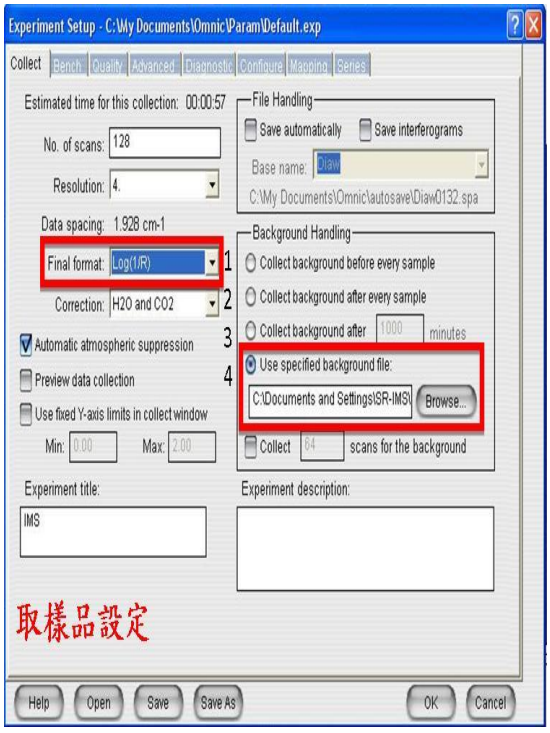
- Resolution: 4 (Spectral resolution; configurable from 0.125 to 32 cm^{-1})

- Final Format:
  – For background: Single Beam
  – For sample: Log(1/R)

Spectral Definitions
T% = I/I_0 * 100
Abs.=log(1/T)=-log(I/I_0)=-logT

- Background Handling:
  – When collecting the background: select 1 – collect background before every sample
  – When collecting the sample: select 4, then load a pre-acquired background spectrum file

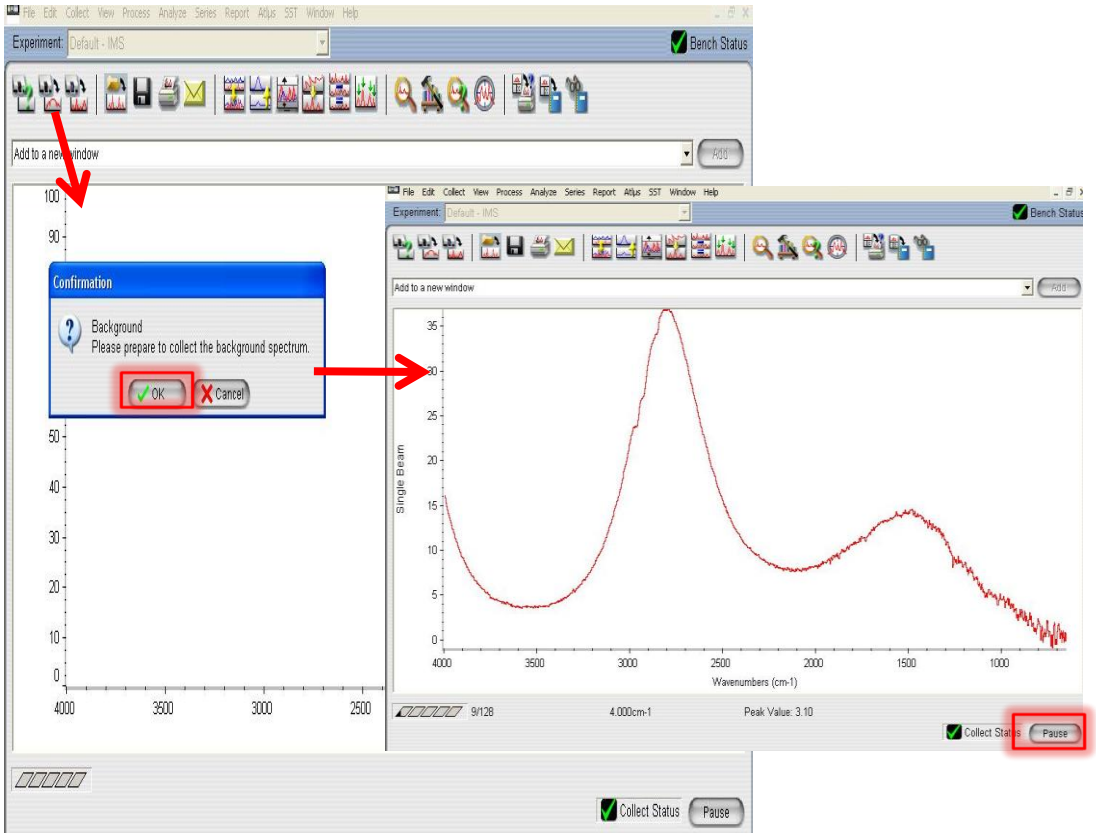




Collecting Background Spectrum

Once parameters are set as above, click  the background scan button on the main interface to begin background acquisition. Save the resulting background spectrum into your experiment folder.

Note: When using the synchrotron source (SR), place the mouse cursor over the Pause button and coordinate your acquisition with the injection timing of the storage ring (typically 55 seconds of usable beam per minute).



Collecting Sample Spectrum
Set all parameters as described above

- Load the background spectrum
- Set Final Format to Log(1/R)
- Click  the sample scan button to begin the measurement
- Save the resulting spectrum into your experiment folder

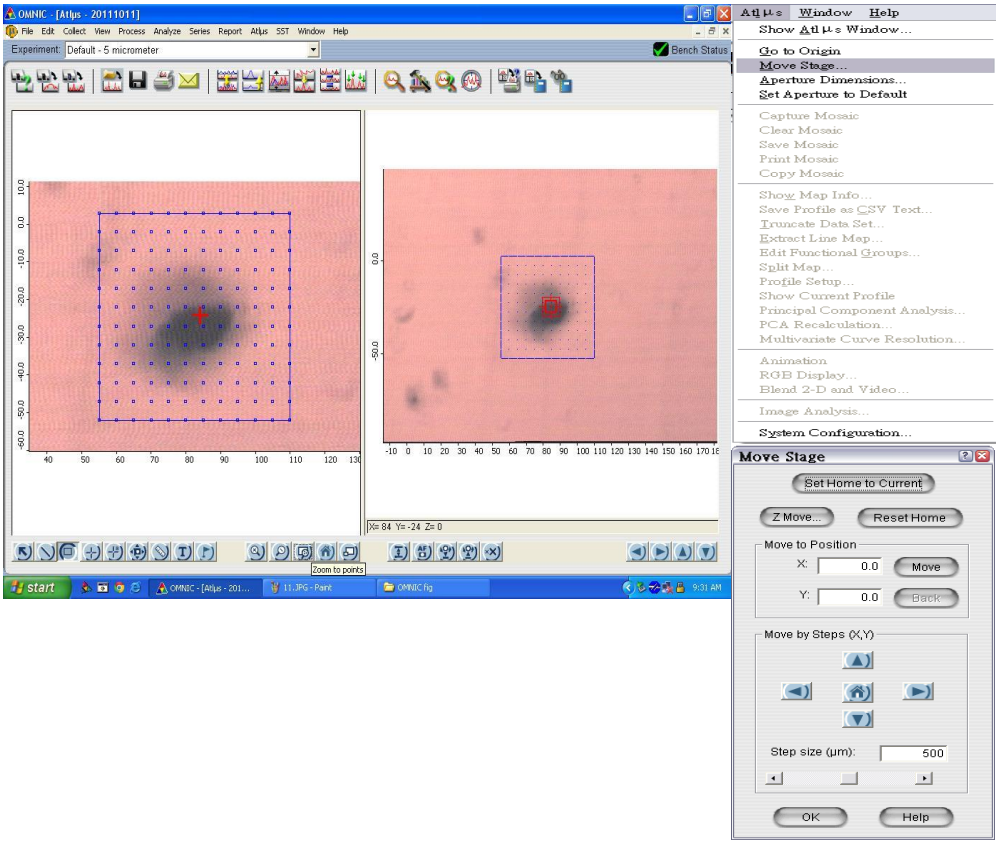
VI. FTIR Mapping Procedure and Parameter Settings

In the Altμs window, define the area to be scanned.

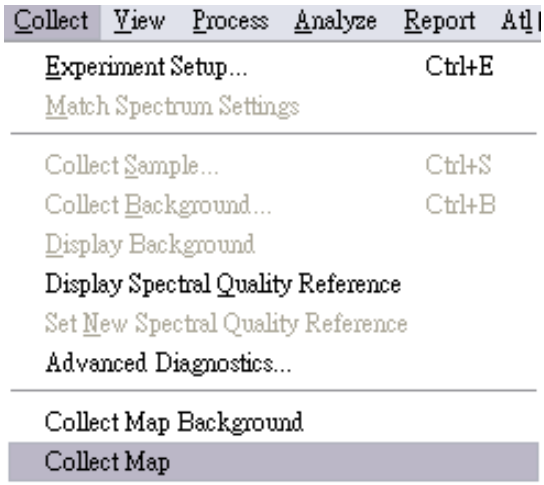
Set current to home: Move stage → Click to set the starting point (origin)

Stage Controls:

- Arrow Tool: Move the cursor
- Stage Movement Tool: Move the sample position
- Zoom to Point: Magnify the selected mapping region



Once the parameters (e.g., scan count, step size) and sample position are set, go to the Collect window, select Collect Map, assign a file name, and start scanning the sample.



Notes When Performing Mapping:

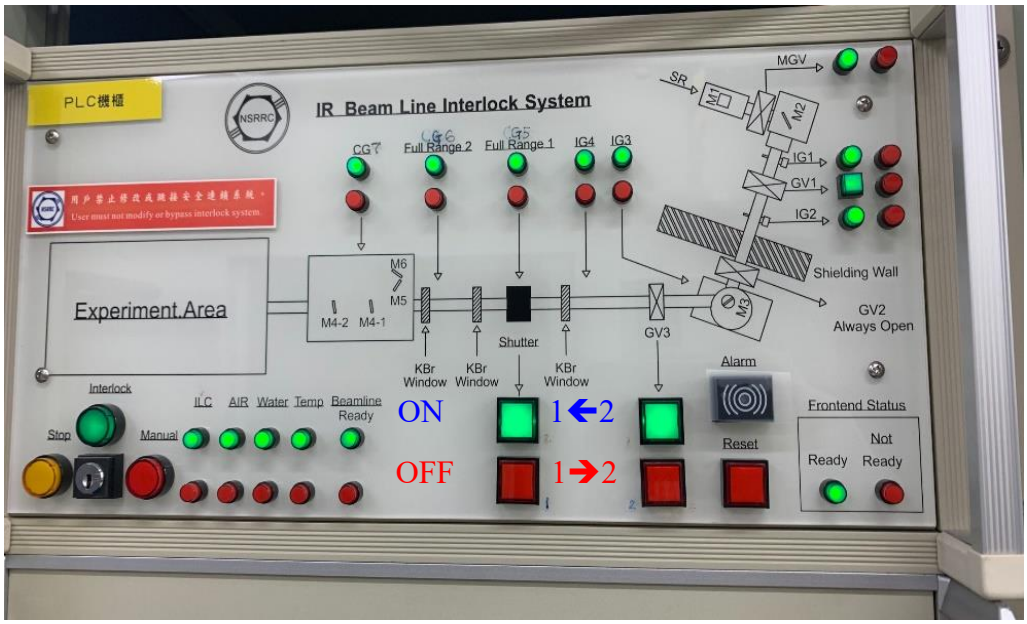
- After a large-area mapping scan, reinitialize the aperture to ensure that the beam is centered correctly.
- During beam outage (no synchrotron light), temporarily switch the source back to IR mode to prevent the interferometer from endlessly scanning while unable to find the zero-path-difference (ZPD = 0), which can lead to misalignment.

- After synchrotron light resumes, press the green square button to open the shutter (2 → 1 ON). Once the IR signal is confirmed to be stable, you may begin the experiment.

Shutter Operation:

1 ← 2: ON

1 → 2: OFF



VII. **IR Beam Line Interlock System**

- After submitting the Experiment Safety Approval Form and receiving authorization, synchrotron radiation (SR) light will be made available during your scheduled session. The control room staff will enable the Interlock Control (ILC) to provide access to the synchrotron source.
- If an abnormal condition occurs in the storage ring that causes the beam to shut off, the ILC indicator will change from green to red. In this case, users must shut down the system in the following sequence:

  → Close Shutter

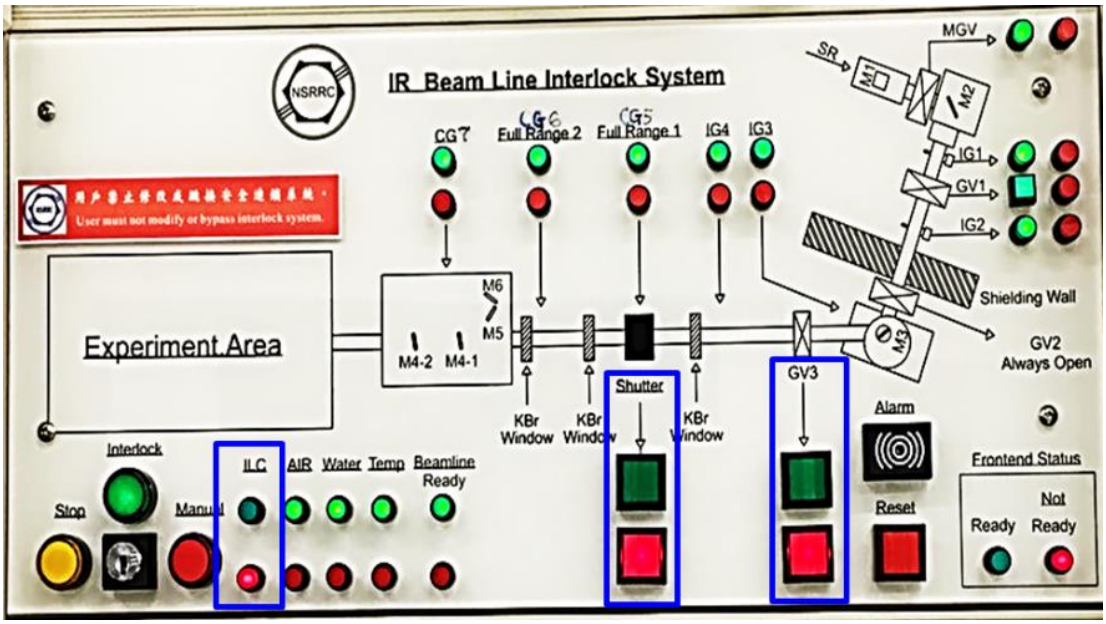
  → Close GV3 vacuum valve (by pressing the red button)

- Wait for the control room to broadcast that the synchrotron beam has resumed normal operation. Then, you may reopen the system in this sequence:

  → Open GV3

  → Open Shutter (by pressing the green button)

- After confirming the signal, experiments may resume.



Before Using the Synchrotron Source (External Mode):
Ensure that all indicator lights on the control panel are green, except for the ILC, GV3, and Shutter status indicators controlled by the control room. A green light indicates normal status.
If any other indicator lights are red, it means that specific component is malfunctioning. In such cases, the beamline cannot deliver light. Please contact the beamline manager: Pei-Yu Huang (Extension #7329)

Top-Up Mode Operation
During operation, the storage ring is maintained at a constant current of 360 mA using top-up mode, where electrons are injected into the ring every minute to compensate for losses.
When performing experiments at the infrared microspectroscopy beamline, users must connect the BNC (RG59/U) coaxial signal cable to the Injection Output (TTL) port. This triggers synchronization with mouse signal acquisition.

Shutter Synchronization (for Interlock Timing):
Set the electronic shutter to N.C. (normally closed) and perform a RESET. This synchronizes the shutter control timing with the PLC trigger signal, ensuring correct acquisition timing.