

Thermo iN10MX FTIR Microscope Operating Procedures

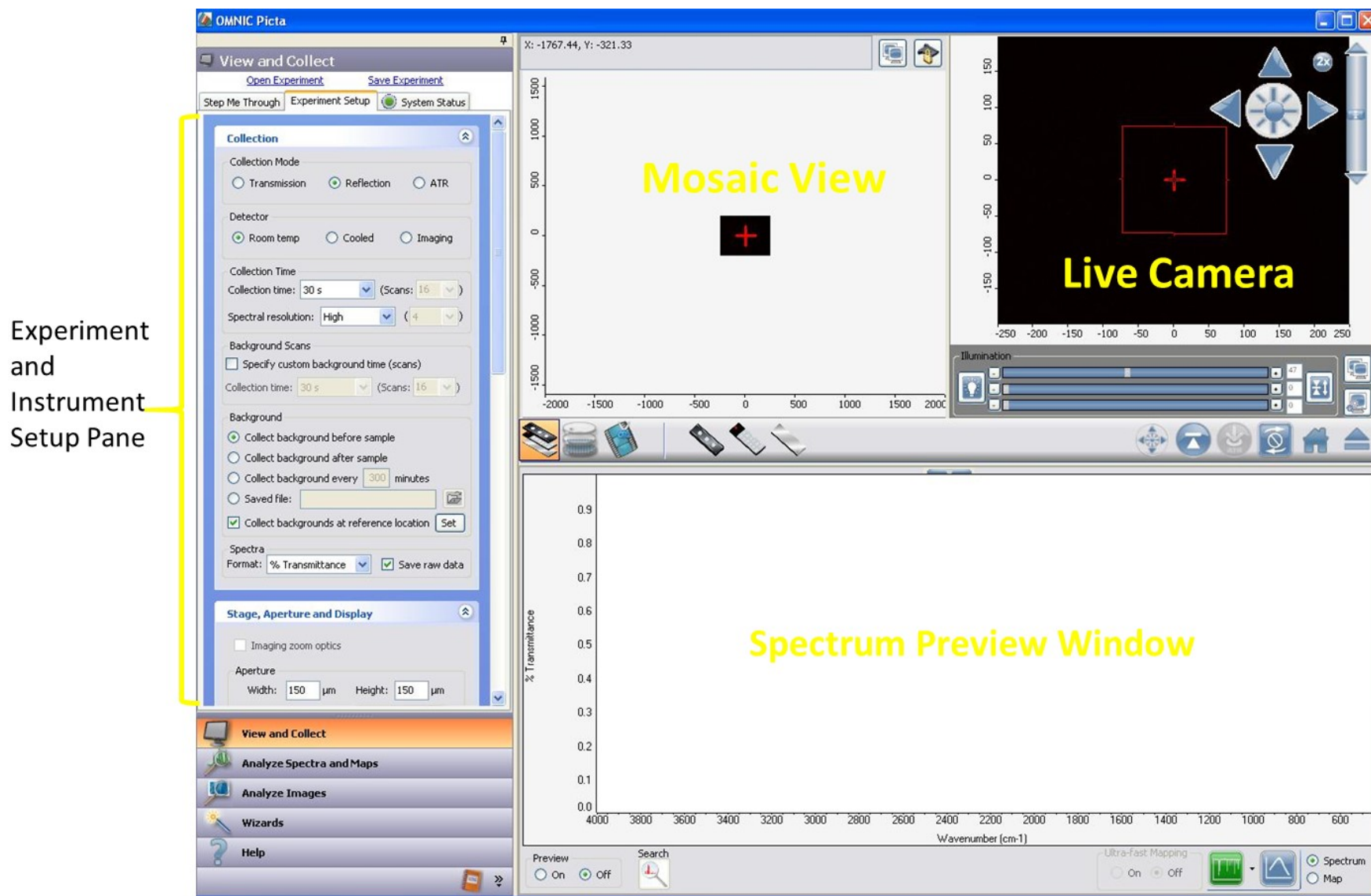


Figure 1: Typical start-up conditions

Thermo iN10MX FTIR Microscope Operating Procedures

Experimental Setup:

Collection Mode: This will usually be Reflection.

Detector: Room Temp, Cooled, and Imaging

Room Temperature detector produces inferior quality spectra, but is good for a quick identification measurement. Runs from 4000 – 550 cm^{-1} .

Cooled: This detector is liquid nitrogen cooled. This is a single point detector. This detector produces good quality spectra from 4000 – 700 cm^{-1} . Please cool the detector before use if the System Status indicator is yellow.

Imaging: This is the array detector and it is also liquid nitrogen cooled and runs from 4000 – 700 cm^{-1} . This detector is used for mapping. Please cool the detector before use if the System Status indicator is yellow.

Collection Time: Collection time will increase with selected resolution and number of scans.

Room Temperature and Cooled Detector: Normally I run at spectral resolution 4 with 16 scans.

Mapping Detector: You do not readily have control over the collection parameters. Run with defaults.

Background: The background scans should be equal to the number of spectral collection scans.

All detectors: This should be set to take a background before every sample (or map) for optimum quality.

Liquid Nitrogen Cooling Procedure:

Liquid Nitrogen safety notes: **Liquid Nitrogen is extremely cold, an asphyxiating agent, and expands to a large quantity of gas. HANDLE WITH CARE. See further precautions at the bottom of this operating procedure.**

I. Fill the Liquid Nitrogen Dewars:

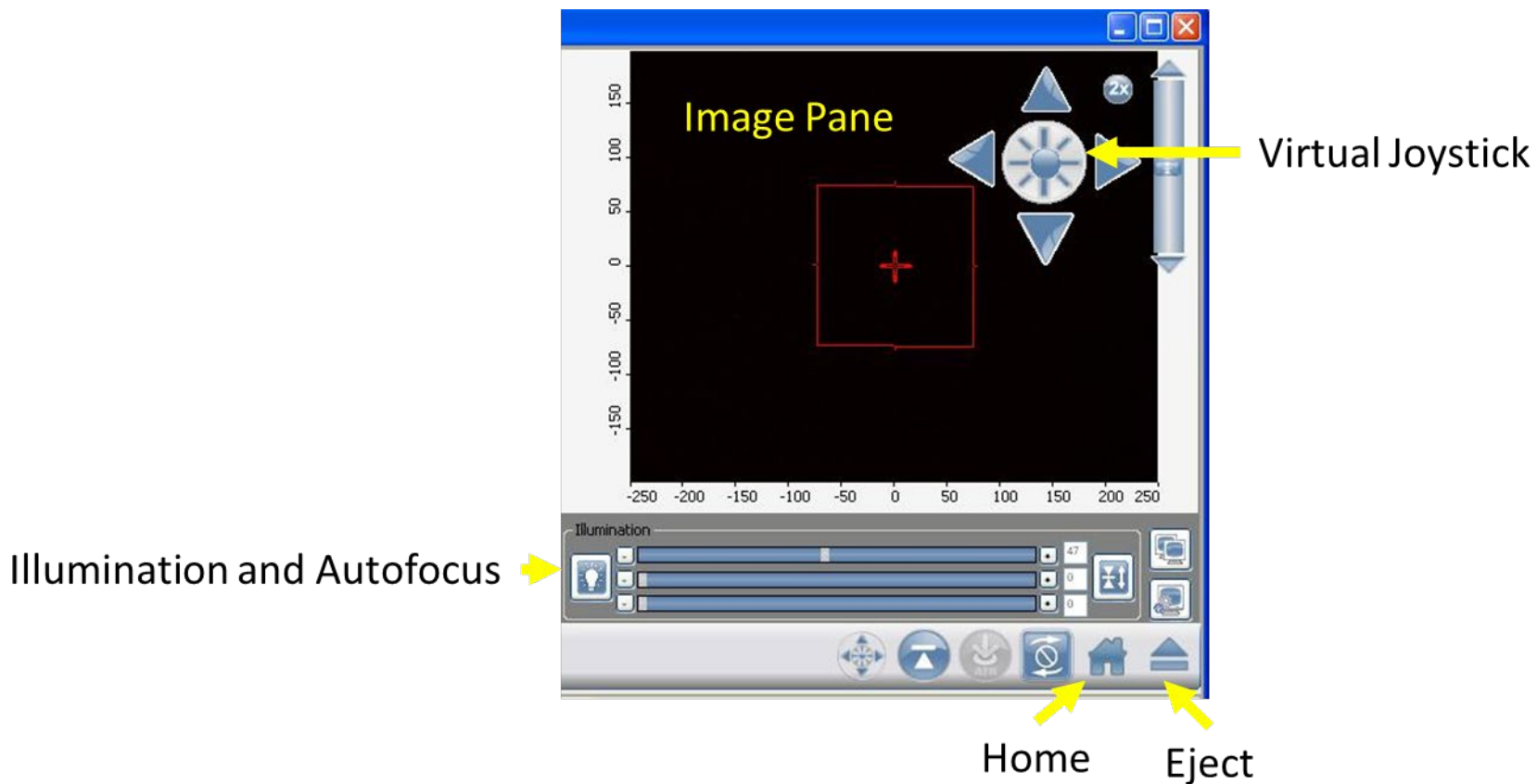
***Warning** – It is important to follow the chilling process listed below, or the detectors will break.

- 1. Preliminary Cooling of the Detectors** – Add two funnels worth of liquid N_2 to each dewar.
- 2. Allow the Detectors to Cool** – Wait 3 minutes for the detectors to cool.
- 3. Fill the Detectors with Liquid N_2** – Fill both dewars. Do not allow excessive LN_2 overflow.
- 4. Allow Further Cooling** – Wait 20 minutes before operating the instrument.

Thermo iN10MX FTIR Microscope Operating Procedures

Data Collection: Single Point - Reflection

Collecting a single point spectrum:



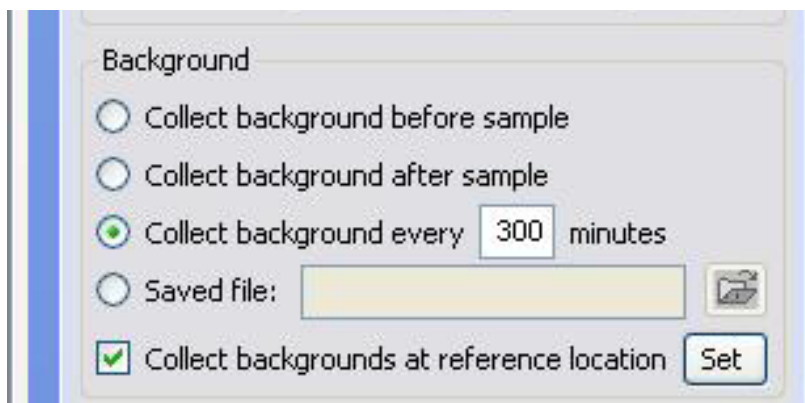
Thermo iN10MX FTIR Microscope Operating Procedures

Eject the stage. Remove slide holder/base. Place small sample into one of the wells of the slide. Tell stage to go to home position. Then tell the stage to move to the appropriate well on the sample slide using the slide selection.



Slide Selection

Set background point to a place on the gold reflector on the slide holder/base by selecting the set button next to the set background at reference location button.



Focus on gold region of slide. Adjust illumination if necessary. Say OK. The stage will return to the sample point.

Focus on your sample or the edge of the slide well. Select sampling location.

Select collect spectrum at bottom of the screen.

Hit the green collect spectrum button.

The FTIR will first collect its background spectrum, then return to the sampling location and collect the spectrum at the indicated location. After collection, the spectrum will appear in a window under Analyze spectra and maps. File – Save As and save your spectrum as .spa.

Thermo iN10MX FTIR Microscope Operating Procedures

Data Collection: Map – Reflection

Set detector selection to Imaging.

Select Map instead of Spectrum at the lower right.

Collect a mosaic, narrow area down to region of interest.

Capture an Image of your Sample:

Select an Image Type – Press the “Map View” button. The detector can collect point, line, and area images.

Select an Area – Use the scroll button to determine the size of the area being analyzed. Draw a box, line, or point where you would like to collect an image.

Collect an Image – Press the “Capture Mosaic” button to collect an image.

Enable Ultra-fast mapping.

Narrow the red box to the region of interest on the collected mosaic. Then, hit the green Collect map button.

Analysis: select peak of interest on the spectrum to get a special map of that functional group within your sample.

Don't forget to save your map.

Instrument Shutdown:

- 1. Inspect the Stage** – Check to make sure the stage is free of obstructions.
- 2. Eject the Stage** – Press the “Eject” button: to remove the stage from the objective.
- 3. Remove your Sample** – Remove your sample from the holder. Place the slides in their appropriate container.
- 4. Return the Stage Home** – Press the “Home” button: to return the stage.
- 5. Turn off the Illumination** – Use the illumination sliders to lower the brightness to zero.
- 6. Log off the Instrument** – Close Picta, remember to log off of the instrument in the hallway.

Thermo iN10MX FTIR Microscope Operating Procedures

Liquid Nitrogen Safety

Properties of Liquid Nitrogen (LN2)

1. It is extremely cold: $77.3\text{K} = -196\text{C} = -320\text{F}$ at atmospheric pressure. This can cause **severe frost bite**.
2. On vaporization it expands by a factor of 700; one liter of liquid nitrogen becomes 24.6 cubic feet of nitrogen gas. This can cause **explosion** of a sealed container, or it can displace oxygen in the room and cause **suffocation without warning**.
3. It can become oxygen enriched and cause ordinarily noncombustible materials to burn rapidly.

Precautions when handling liquid nitrogen

1. Treat liquid nitrogen and any object cooled with liquid nitrogen with respect.
2. Take care not to allow liquid nitrogen to be trapped in clothing near the skin.
3. Wear safety glasses or a face shield when transferring liquid nitrogen.
4. Wear gloves when touching any object cooled by liquid nitrogen. Gloves should be loose fitting, so they could be thrown off if liquid were to pour inside them
5. Use only approved unsealed containers. Never pour it into a coffee thermos. Never seal it in any container (it will explode).
6. Never dip a hollow tube into liquid nitrogen; it may spurt liquid.
7. Never use in a small poorly ventilated room, and never dispose of liquid nitrogen by pouring it on the floor. It could displace enough oxygen to cause suffocation. Nitrogen gas is colorless and odorless--the cloud that forms when you pour liquid nitrogen is condensed water vapor from the air, not nitrogen gas.
8. Do not store liquid nitrogen for long periods in an uncovered container (on the other hand, never totally seal a container). Because the boiling point of oxygen, 90.1K , is above that of nitrogen, oxygen can condense from the air into the liquid nitrogen. If the air over the nitrogen circulates, this liquid oxygen can build up to levels which may cause violent reactions with organic materials; even materials which are ordinarily nonflammable. For example, a severe clothing fire could result from ignition in the presence of liquid oxygen.

Thermo iN10MX FTIR Microscope Operating Procedures

First Aid

- Suffocation: If person seems to become dizzy or loses consciousness while working with liquid nitrogen, move to a well-ventilated area immediately. If breathing has stopped, apply artificial respiration. If breathing is difficult, give oxygen. Call a physician. Keep warm and at rest.
- Frost bite: If exposed to liquid or cold gas, restore tissue to normal body temperature, 98.6F (37C), followed by protection of the injured tissue from further damage and infection. Remove or loosen clothing that may constrict blood circulation to the frozen area. Call a physician. Rapid warming of the affected part is best achieved by using water at 106F (42C). Under no circumstances should the water be over 112F (44C), nor should the frozen part be rubbed either before or after rewarming. The patient should neither smoke, nor drink alcohol.