Operation of the Nicolet iN10 MX Infrared Microscope

Instrument Summary:

      The Thermo Scientific Nicolet iN10 MX is an infrared imaging microscope that offers the ability to rapidly acquire microscopic images and simultaneously collect infrared spectra of solid specimens. It allows analysis of the distribution of chemicals within a sample through both point based analysis and comprehensive spectral mapping. The microscope is equipped with a cooled MCT detector which facilitates ultra-fast imaging and spectral mapping of large areas within minutes. Additional attachments include a MicroTip ATR for contact sampling, a nitrogen flow chamber, and a motorized stage. This instrument is ideal for the analysis of coatings, contaminant identification, and the mapping of chemicals in printed media.
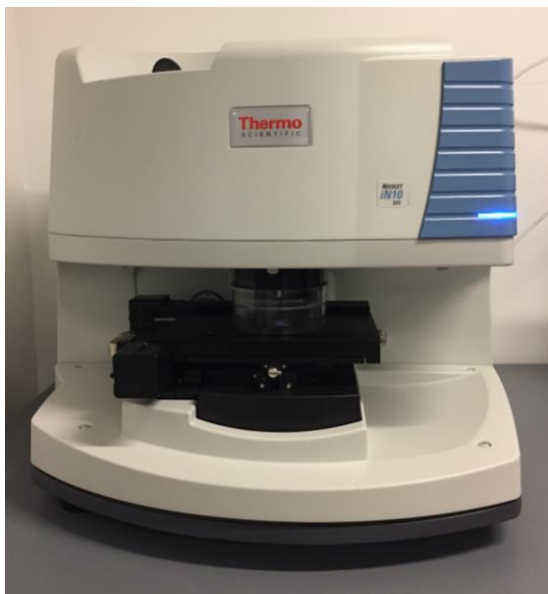

Figure 1. The Thermo Scientific Nicolet iN10 MX infrared imaging microscope.

Safety and Precautions:

- Inspect the stage before moving it. Ensure that the stage, the ATR attachment, and your sample will not crash into the objective.
- **Warning –** Do not allow loose sample or particles to fall into the space under the stage.
- **Warning –** Follow the proper procedure utilized to cool the detectors.
- **Warning –** Do not move the stage when the ATR tip is in contact with a sample.
- **Warning –** Do not touch the tip of the ATR crystal.

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.

II. Select Settings in Picta:

1. **Log into the Instrument**
2. **Open Picta –** Picta controls the microscope's imaging and FTIR capabilities.
3. **Select a Collection Mode –** Select a collection mode from the "View and Collect" tab.
   a. **Transmission –** For transparent or translucent samples, or samples on a microscope slide.
   b. **Reflection –** For solid, opaque samples, and for surface analysis.
   c. **ATR (Attenuated Total Reflectance) –** For samples requiring contact based spectra. Requires an additional attachment equipped with a germanium crystal.
4. **Select an IR Detector –** There are three detectors with varying acquisition speeds and capabilities.
   a. **Room Temperature –** For general analysis of samples from 4000-400 cm^{-1}.
   b. **Cooled Detector –** Facilitates point, line, and area analysis of samples from 4000-675 cm^{-1}. Detects less noise than the room temperature detector.
   c. **Imaging Detector –** For rapidly acquiring line and area scans which span large areas. Does not facilitate point scans. Detection range from 4000-715 cm^{-1}.
5. **Select a Resolution –** Select a spectral resolution, higher resolution allows the detection of more peaks but results in noisier spectra.
6. **Select Number of Background Scans –** The background is a coadded from multiple scans.
7. **Select Background Frequency and Type –** Backgrounds can be taken before or after analysis of each sample or at regular time intervals.
8. **Select a Spectra Format –** The data can be formatted into multiple types:
   a. Transmittance
   b. Absorbance
   c. Single Beam
   d. Interferogram
   e. Kubelka-Monk
9. **Enter Aperture Size –**The default aperture setting is 100 x 100 μm. The dimensions of the aperture can be programed from 5-400 μm.
10. **Enter Number of Points –** The number of points can be tailored for area and line scans. Increasing the number of points will cause the instrument to collect more spectra from more locations across your sample.

III. Load and Locate Your Sample:

1. **Raise the N₂ Chamber –** Lift and rotate the plastic chamber to remove it from the stage.
2. **Inspect the Stage –** Check to make sure the stage and objective are free of obstructions.
3. **Eject the Stage –** Press the "Eject" button to make the stage more accessible.
4. **Insert your Sample –** A sample can be placed on a microscope slide, the 3-hole slide, or the 12-spot slide. Clip the slide onto the stage to prevent movement.
5. **Locate and Focus your Sample –** Use the joystick, virtual joystick, autofocus, or the keyboard arrow keys to move the stage into position and focus on your sample.

    Autofocus;   Virtual Joystick

IV. Capture an Image of your Sample:

1. **Select an Image Type –** Press the "Map View" button. The detector can collect point, line, and area images.
2. **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.
3. **Collect an Image –** Press the "Capture Mosaic" button to collect an image.

    Map View;   Point Tool;   Line Tool;   Area Tool;   Capture Mosaic

V. Collect a Background Spectrum:

1. **To Automatically Collect a Background –** Select "Collect Backgrounds at Reference Location". The sample holder has preset positions for collecting background spectra.
    a. **Gold Disk –** For Reflection Mode
    b. **Open Hole –** For Transmission Mode
2. **To Manually Collect a Background –** Move your sample to an area of interest, focus the image, press the "IR Energy" button and then the "Background" button.
3. **Collect the Background –** After a location has been marked, press the "Collect Background" button.

    IR Energy;   Background;   Collect Background

VI. Collect a Spectra:

1. **Focus the Sample –** Use the joystick or virtual joystick to bring the image into focus.
2. **Select a Spectra Tool –** Either a point, line, or area scan can be produced.
3. **Select a Spectra Type –** Either a single spectra or a map can be produced by selecting the appropriate bubble in the bottom right hand corner.
4. **Ultra-Fast Mapping –** Press "Ultra-Fast Mapping" for a fast scan with increased noise.
5. **Collect Spectra –** Optimize the "IR Energy" with the IR energy tool then press "Collect Spectra" button in the bottom-right corner.

    Collect Spectra;   IR Energy

VII. Data Analysis and Library Searching:

1. **Set up a Library –** In the "Analyze Spectra and Maps" tab scroll down to "Library Set-Up" and "Select Library".
2. **Select Libraries to Include –** Highlight libraries related to your sample and press "Add >>", Press "Ok", scroll down to the analyze section and press "Search".
3. **Analyze Spectra –** Picta also allows spectral mapping of your sample and 3D Mapping. This is accomplished by selecting a peak of interest in the spectral window.
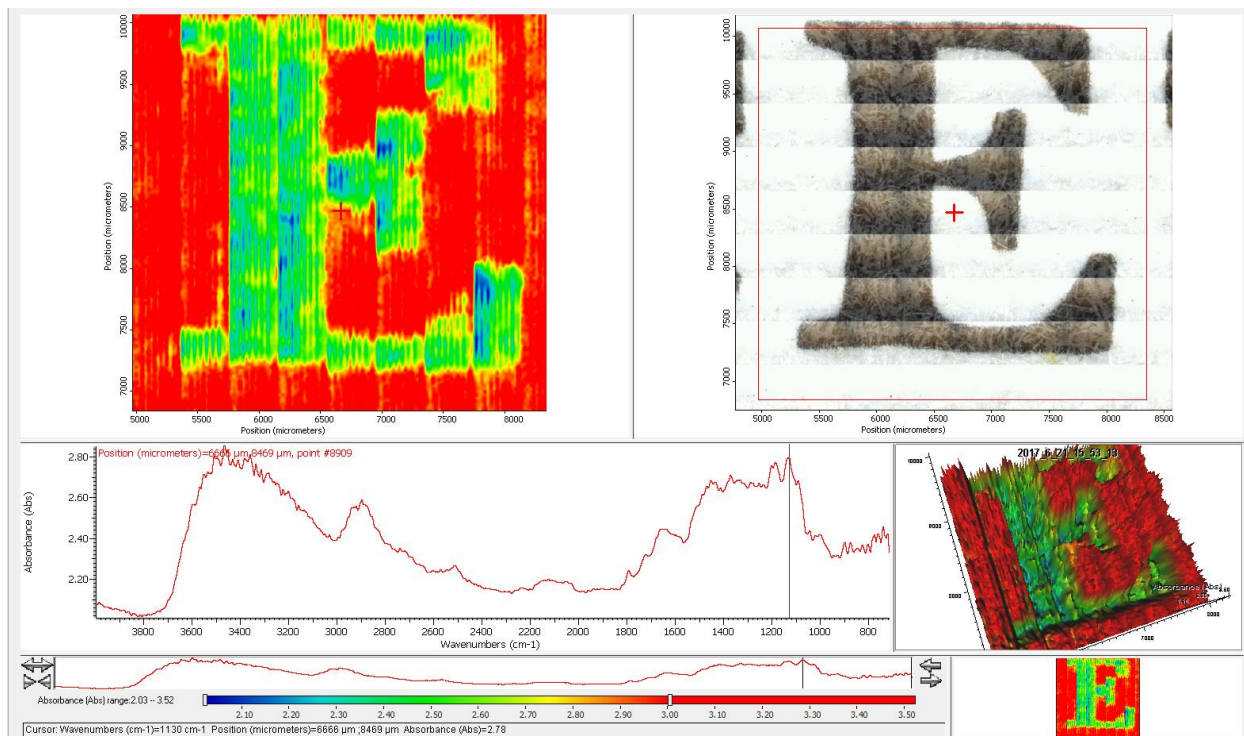

Figure 2. FTIR mapping of printed media.

VIII. 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 IAC's 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.
7. **Sign the Logbook**
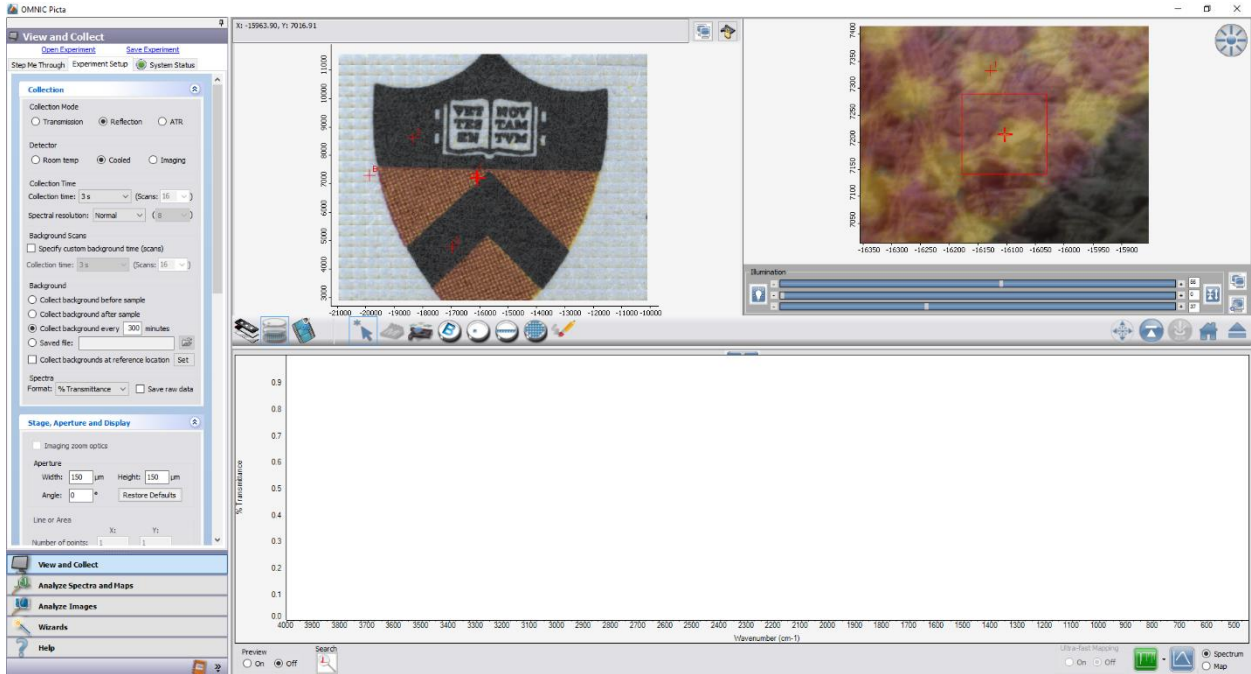
Appendix
Omnic Picta Software

Figure 3. Omnic Picta Software.

 Slide View

 Map View

 Virtual Joystick

 Mosaic Image

 Background Button

 Sample Point Tool

 Line Tool

 Area Tool

 Eraser Tool

 Autofocus

 X-Y Stage Joystick Control

 IR Energy

 Return Stage Home

 Eject Stage

 Collect Spectra

 Collect Background

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:
1.   Contact IAC Staff.
2.   If unsure, leave system as is.
3.   Try to power down the system.

Audible/Siren Emergency Alerts:
Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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