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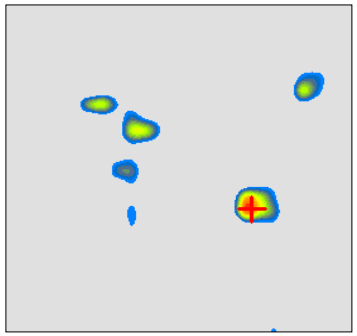
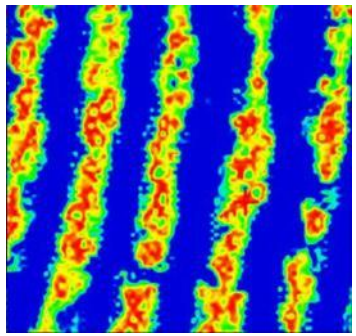
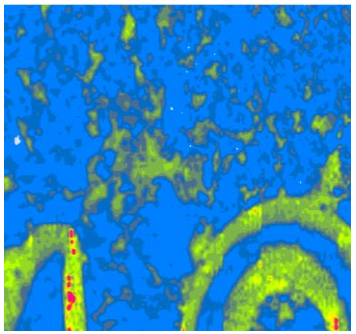
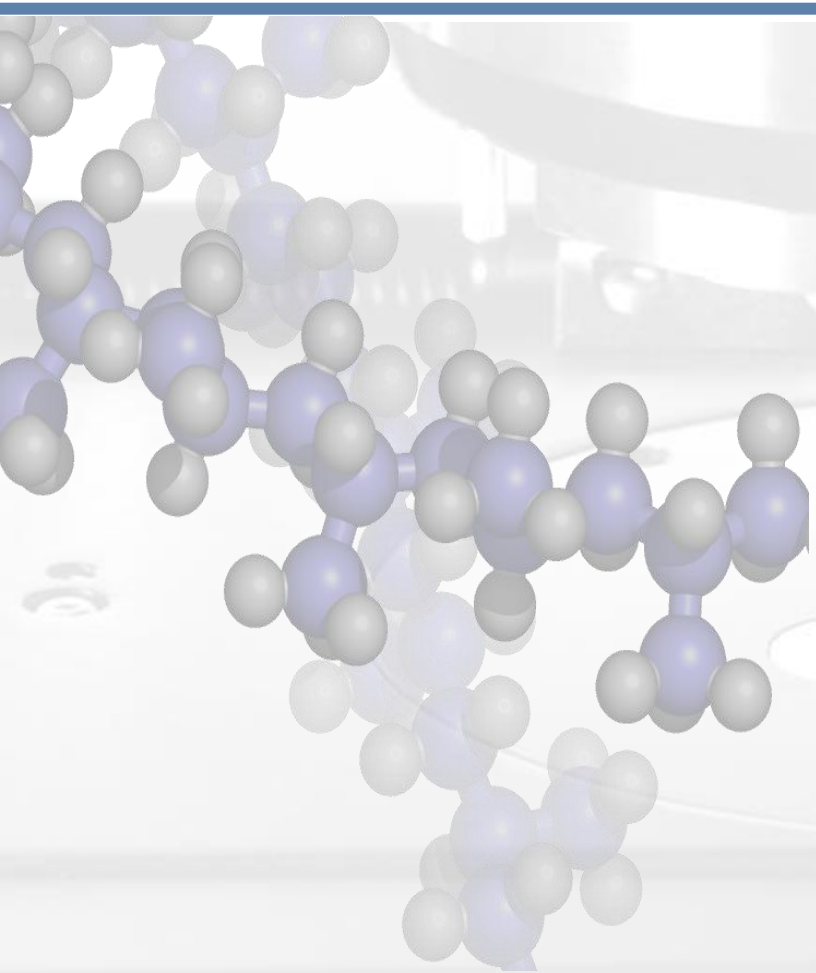
FT-IR Microscopy with Image-Guided Analysis: Polymer Applications







Agenda

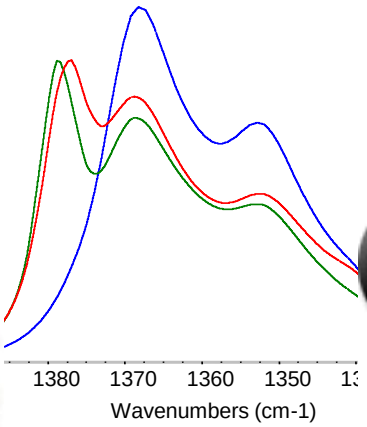


- Vocabulary and “anatomy” of infrared microscopy
- Visual tools for data collection and analysis
- Integrated Operations and Applications
- Q & A

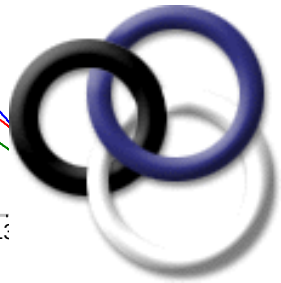
Infrared Microspectroscopy – An Indispensible Tool

- Light Microscopy + Spectroscopy =
  - Inspection & characterization using both visual and chemical contrast
- Chemical information of physically small samples, or small domains within heterogeneous specimens
  - How small? From barely visible to the eye down to diffraction limit (~λ)

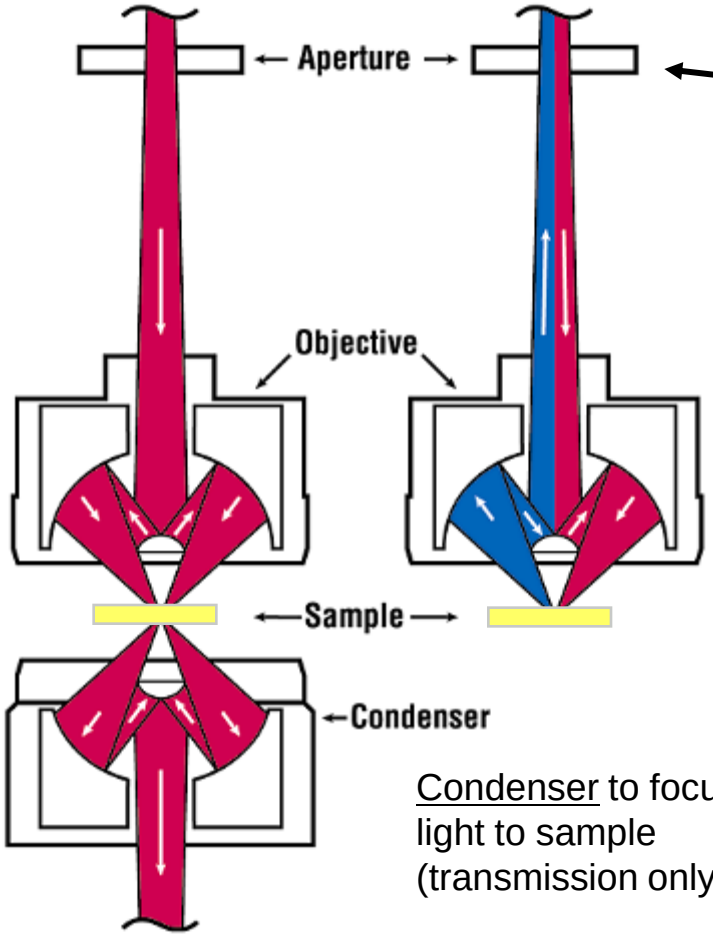
- Who needs it?
  - Analytical Services
  - Quality Control
  - R&D
  - Material scientists



- For what purpose?
  - Failure analysis
  - Reverse engineering
  - New products development



Generic Picture Infrared Microscopy Sampling Modes

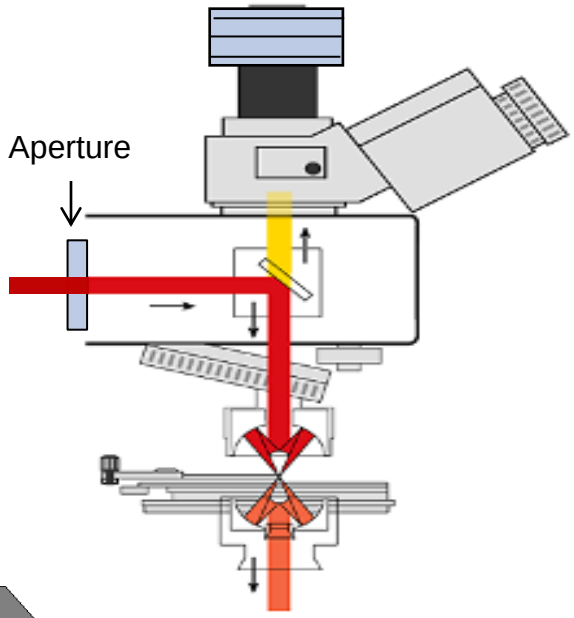


Apertures are a remote mask used to select region of specimen

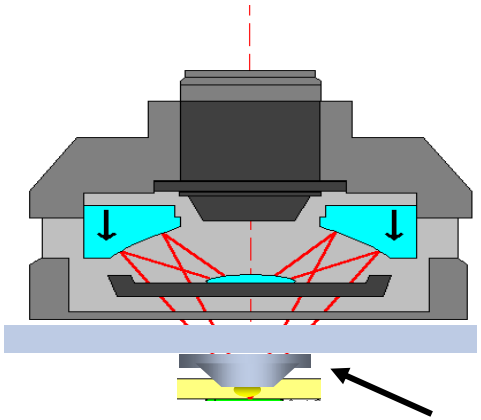
Objective to focus or collect infrared light to sample

Condenser to focus light to sample (transmission only)

Digital Video



Micro ATR



ATR Crystal for special case of Reflection mode

Thermo Scientific FT-IR Microscopes Product Line

- Nicolet iN10 and iN10 MX FT-IR imaging microscope
  - Ground breaking integration, software driven operations
  - Options for fast mapping and linear array detector
- Nicolet ContinuμmTM infrared microscope
  - The “Microscopist’s Microscope”
  - Flexible configuration for Infrared features
  - Outstanding visible light performance

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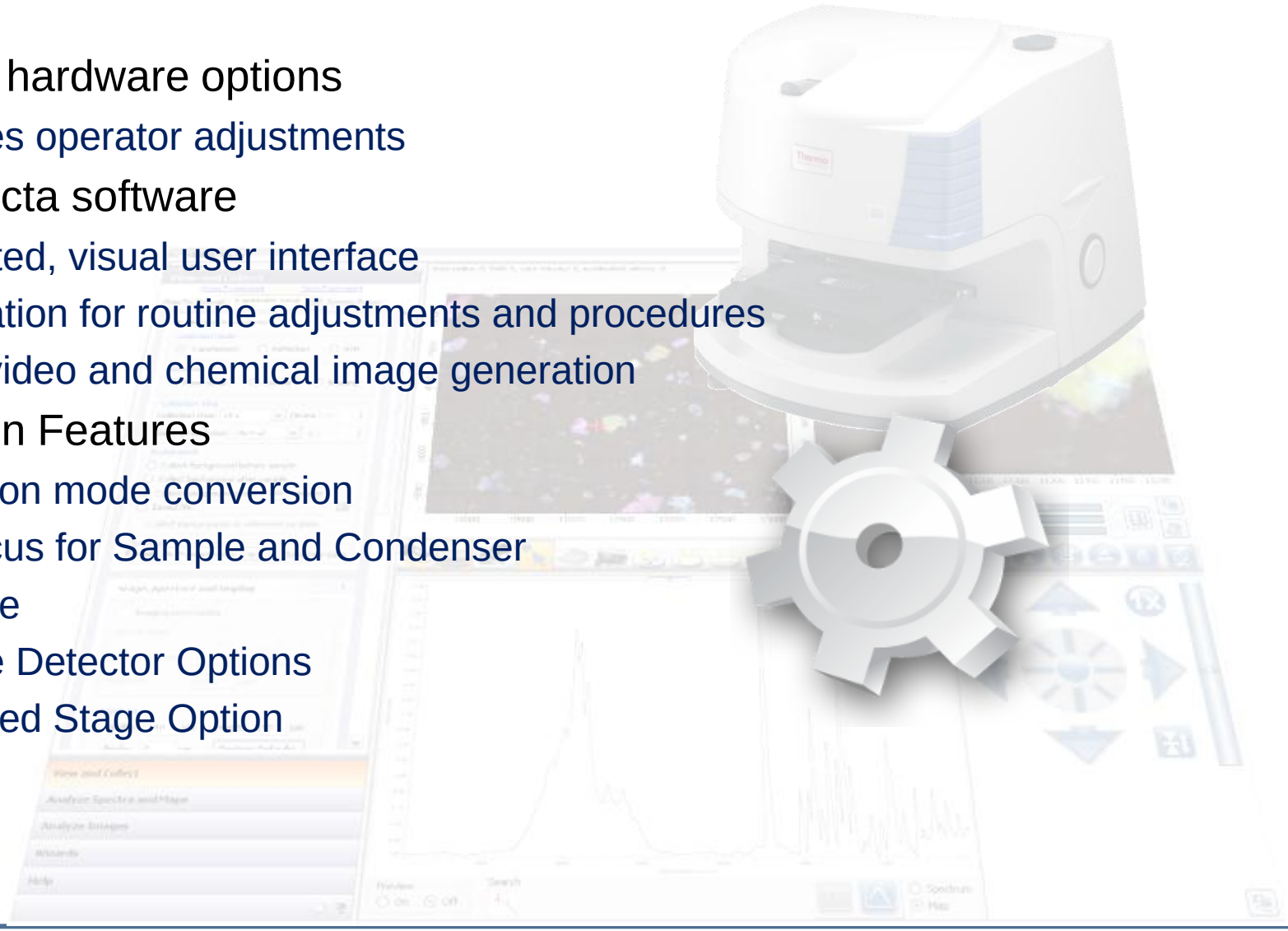
Nicolet iN10 FT-IR Microscope

- Simplified hardware options
  - Reduces operator adjustments
- OMNIC Picta software
  - Dedicated, visual user interface
  - Automation for routine adjustments and procedures
  - Rapid video and chemical image generation
- Automation Features
  - Collection mode conversion
  - Autofocus for Sample and Condenser
  - Aperture
  - Multiple Detector Options
  - Motorized Stage Option

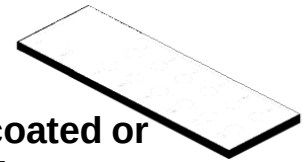


Sample Presentation for Infrared Microscopy

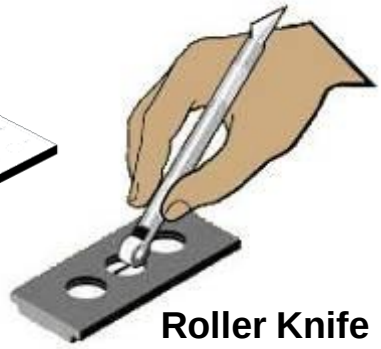
- Transmission analysis
  - Best quality but specimen must be THIN
  - Standard 13 mm infrared-transparent windows (salt or diamond)
- External reflectance and reflection-absorbance
  - Convenient, very fast data colletion
  - Reflective substrates boost sensitivity
  - Metal coated or low-e slides
- ATR – minimum prep
  - Contact sampling, surface sensitive
  - Fixed “path-length”

3-position window holder slide

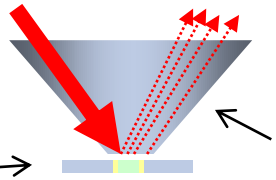
Compression Cell



Metal-coated or “low-e” IR reflective slides



Roller Knife

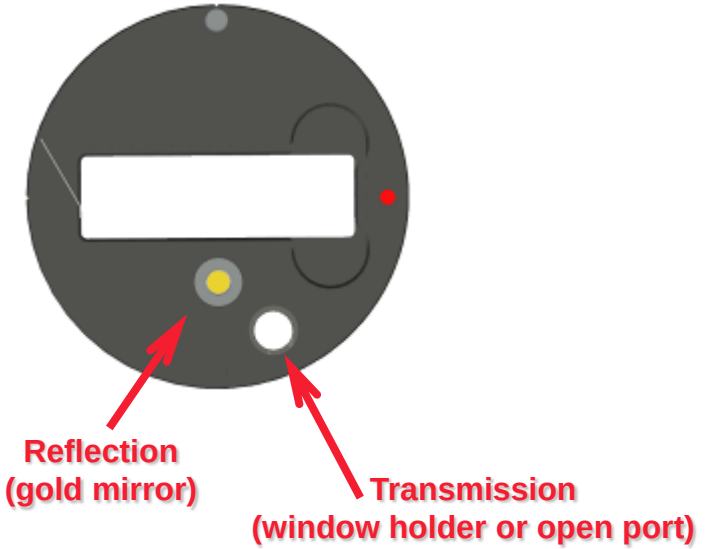


sample

IRE (germanium)

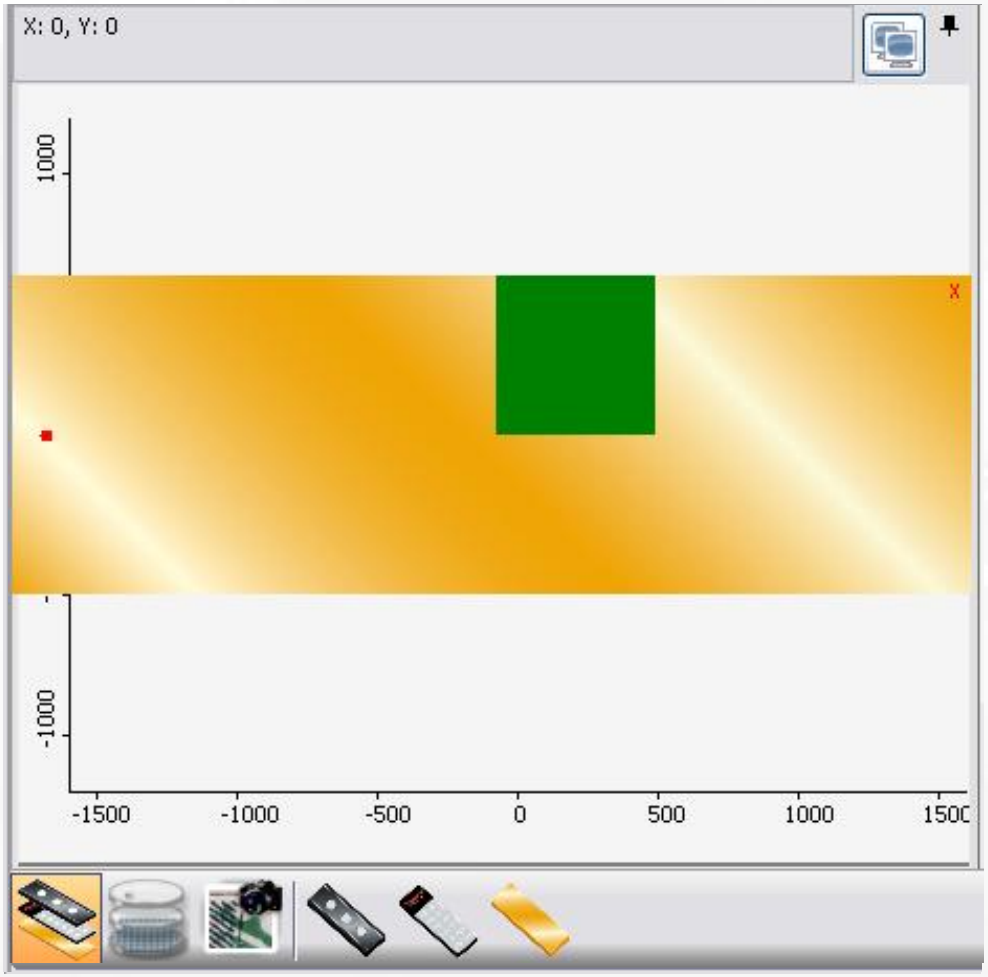
Nicolet iN10 – Fast Sample & Background Positioning

- Custom slide holder simplifies loading
- Standard background locations enable automatic background collection
- The background locations, X, Y and Z (focus) coordinates are stored for rapid and automatic background collection

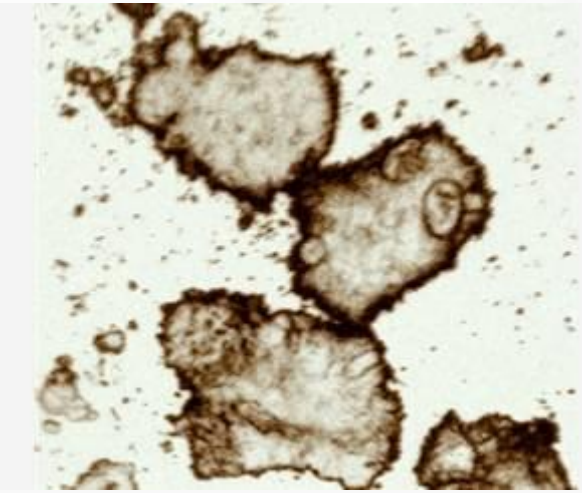


Background Locations

Slide View - Efficiently Locate Micro-samples for Analysis



- Slide View helps locate sample position
  - Select your slide type
  - Click on the slide where the sample is located...
- OMNIC Picta automatically moves the stage to sample location, X – Y, & Focus



The Mosaic – Expand your Field of View (FOV)

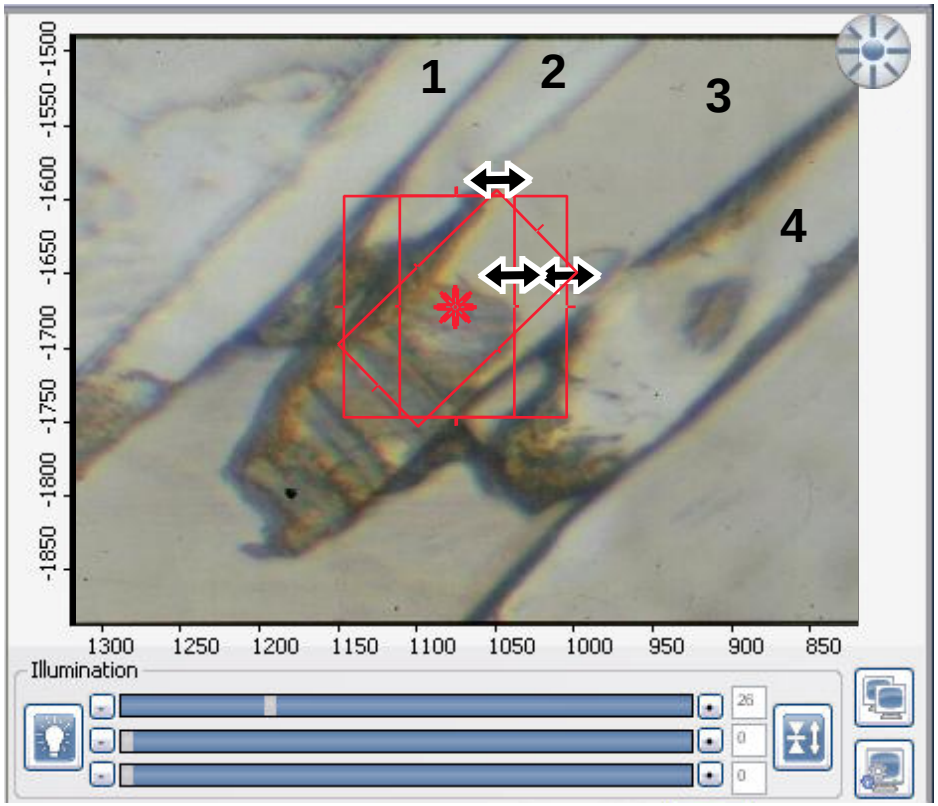
Static Mosaic ***Live Video***

- Even at low magnification, the field of view is small
- Use Automation to create a wide-field road map; a mosaic of multiple video frames

Aperture Control - Selecting the Analysis Region

- Interactive video image control selects region of FOV for exposure to Infrared beam

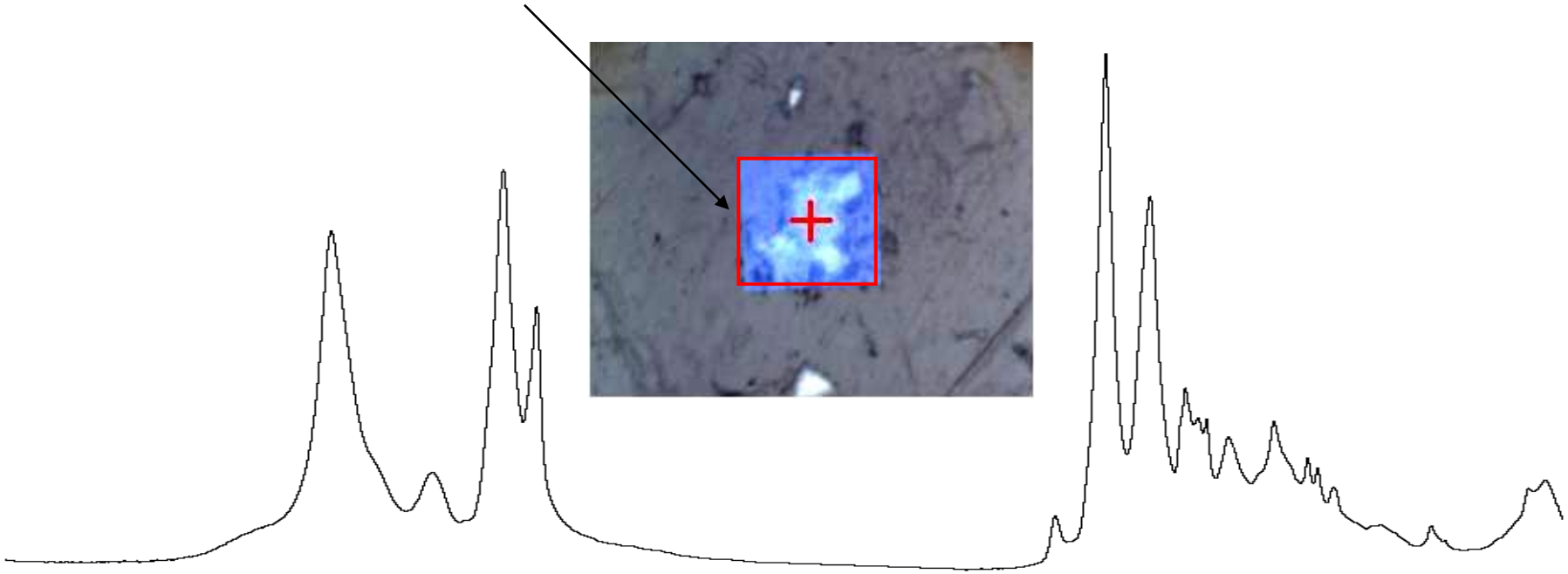
Click and drag on an edge to change the size of the aperture to match a sample dimension



Click on a corner and drag to change the angle of the aperture to match the sample

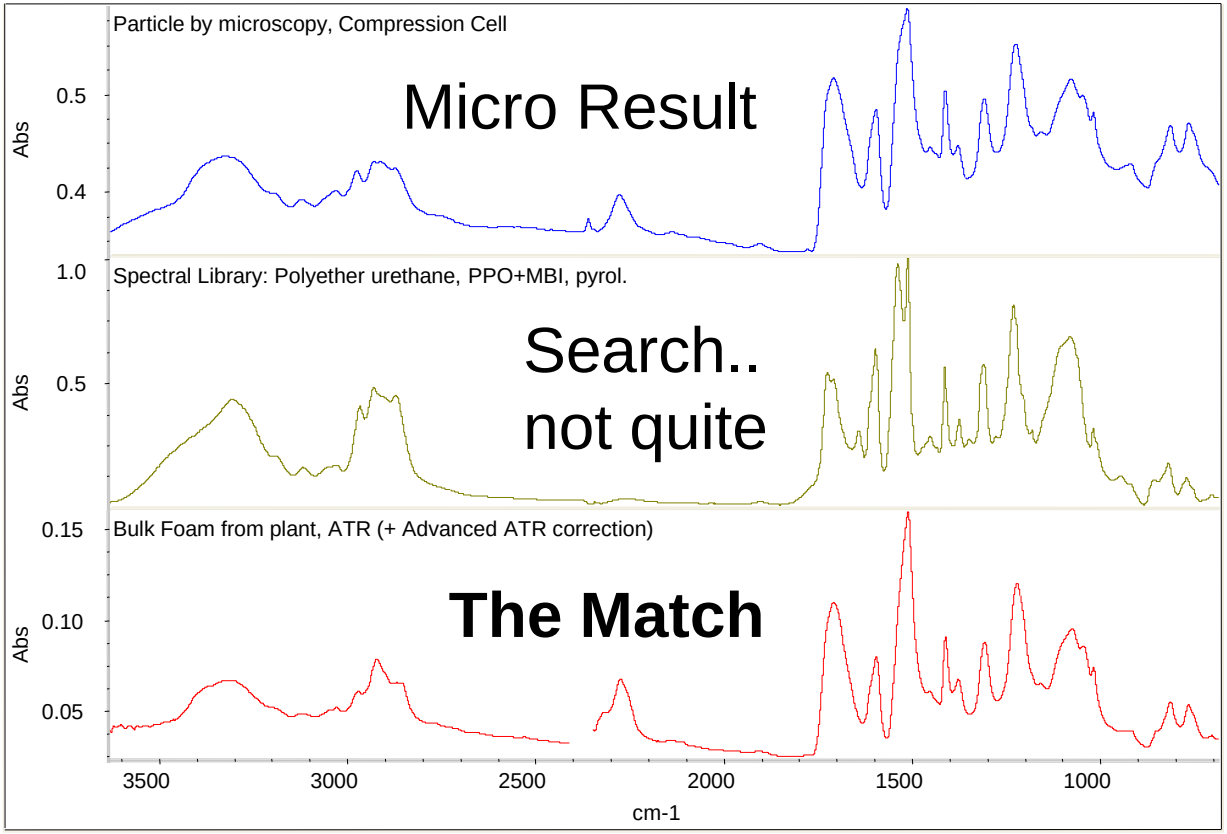
Verify the aperture position

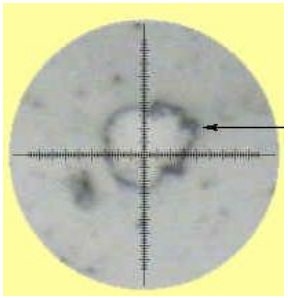
- Simultaneous collection of data while viewing the sample
  - TruView™ Simultaneous View And Collection
  - View a live IR spectrum while adjusting the aperture
  - Full view of the sample, even when masked
- Separate illumination source highlights the IR beam path



Single Point Analysis – Transmission

- Trouble shooting a customer complaint
  - Product being pushed back to supplier due to particles on the film
  - Technique: Isolate a particle in micro-transmission in compression cell





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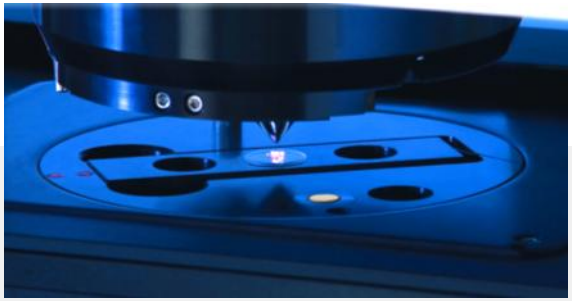
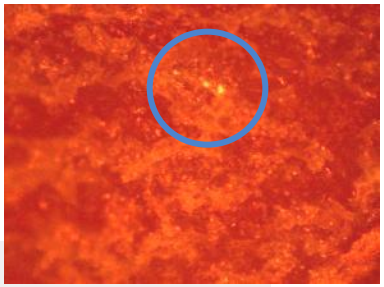
Polymer Surface Check – Micro ATR

- White crystal particle on red polymer feed beads – Source?
- Confirmed material excess antioxidant, DLTDP





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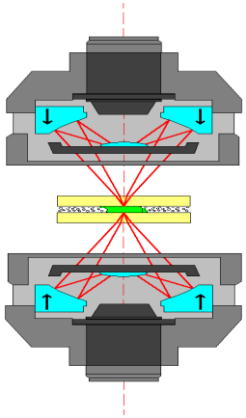
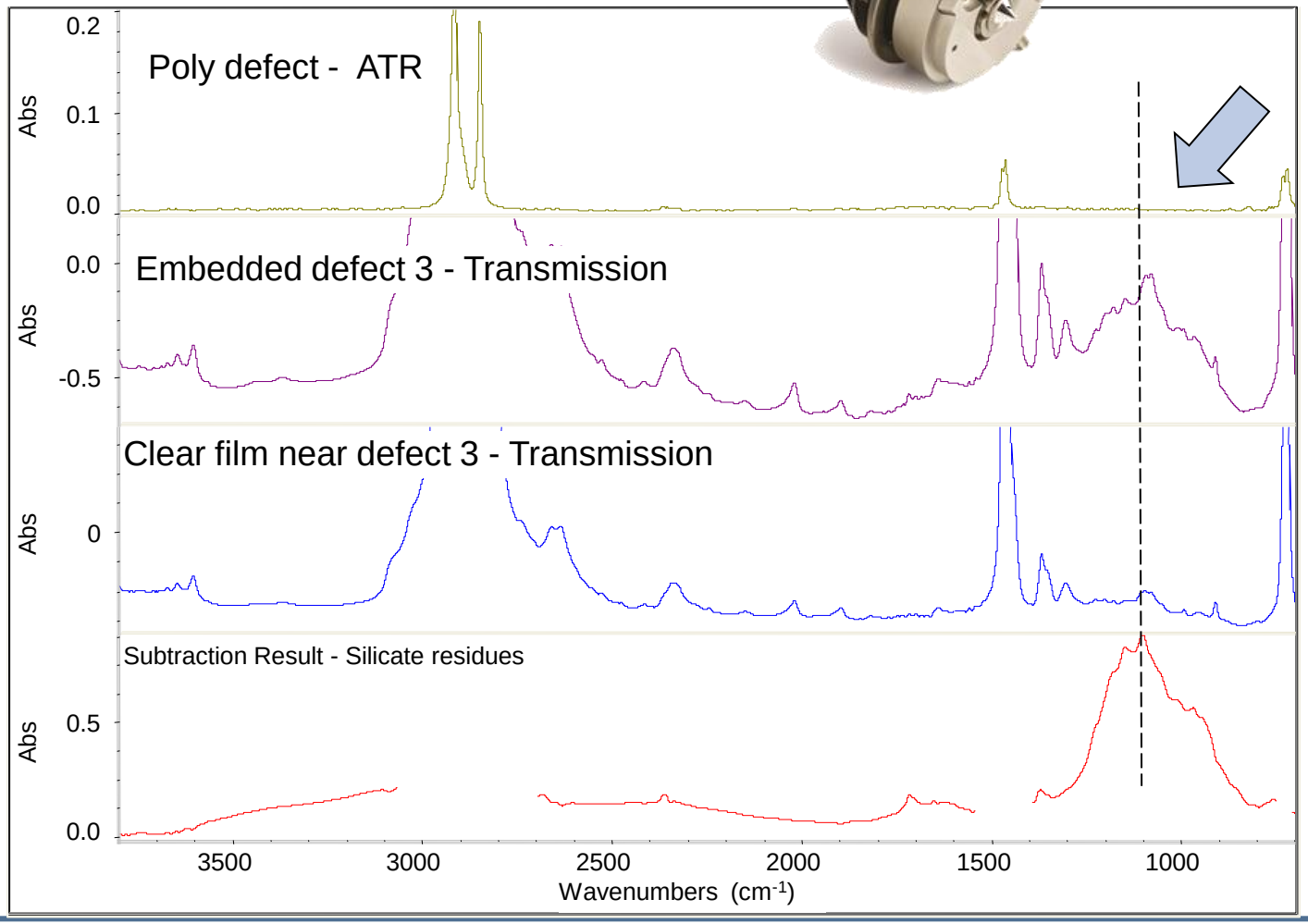
Transmission IR Microscopy for High Sensitivity

ATR Accessory

Transmission configuration





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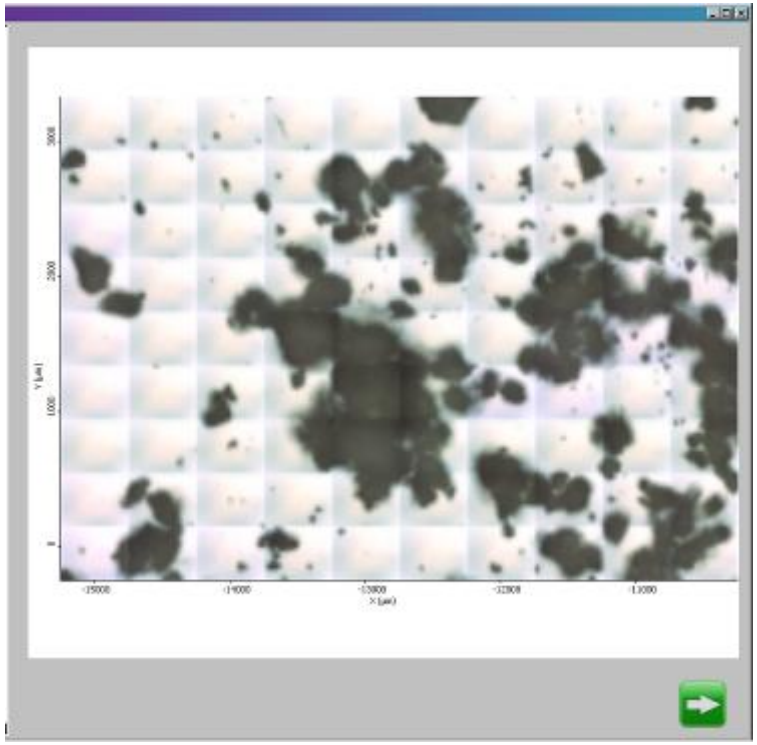
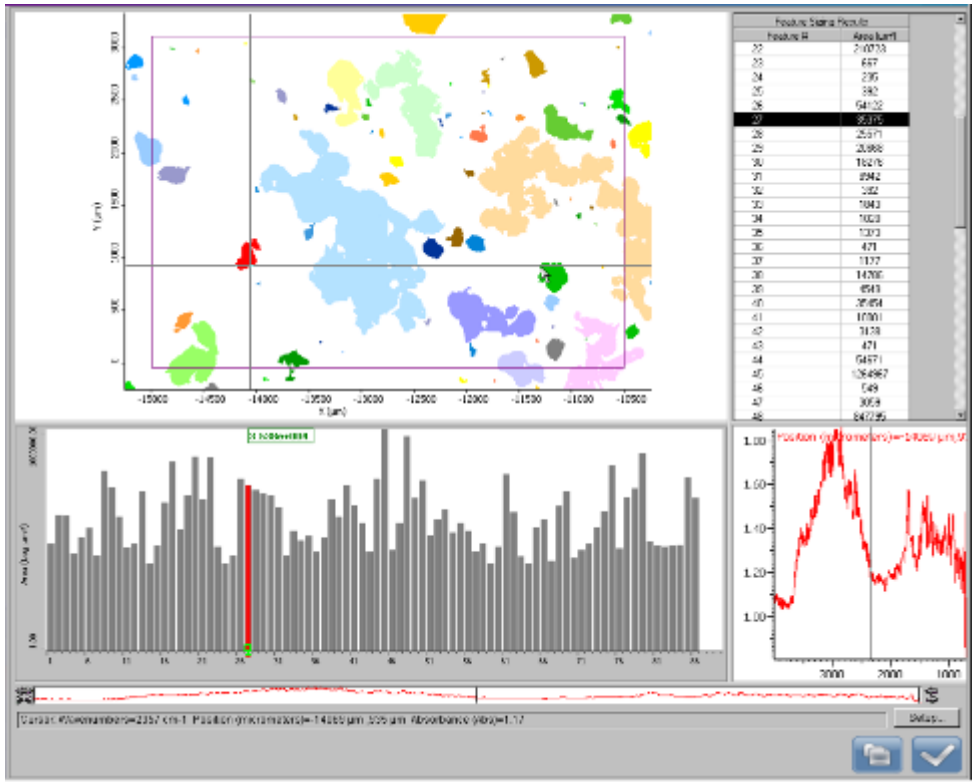
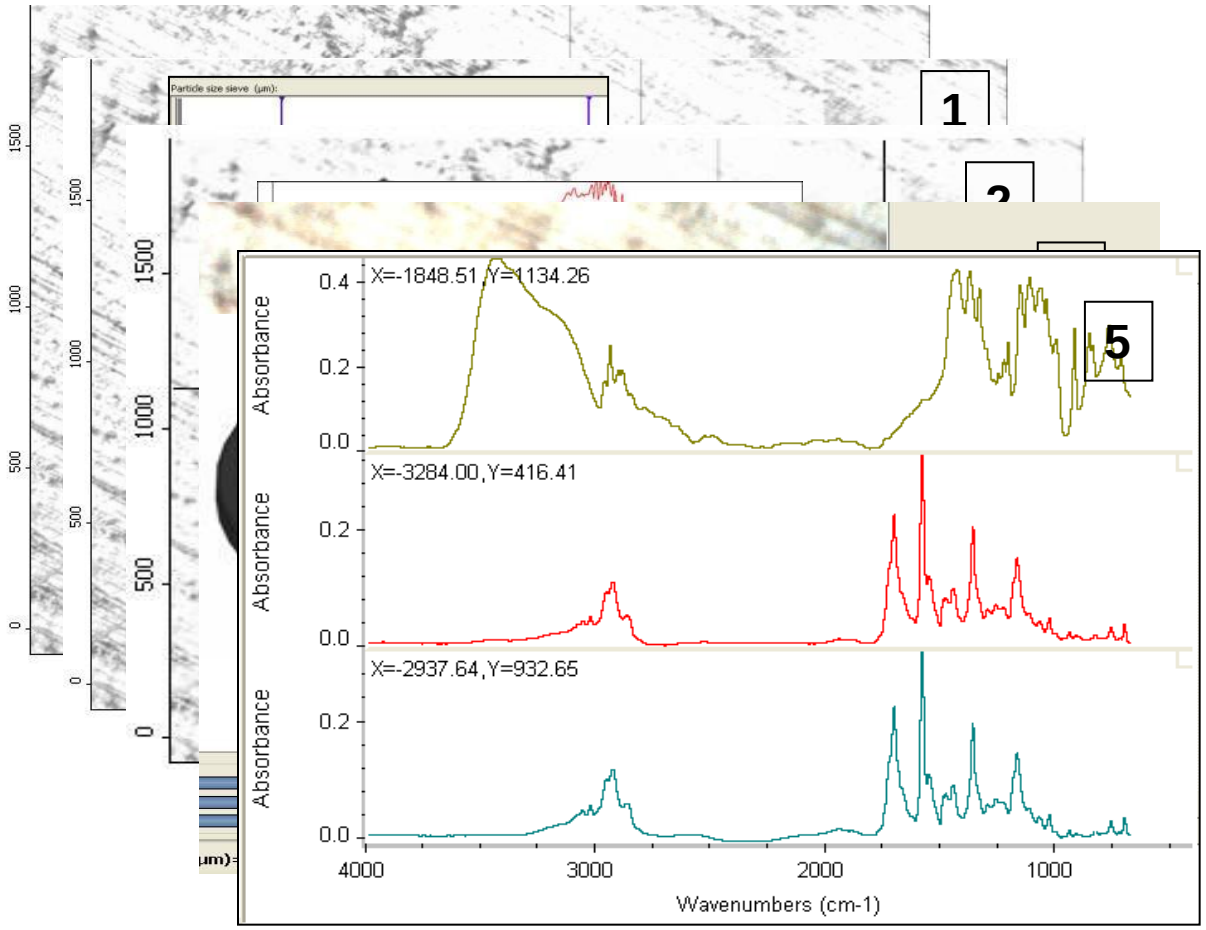
Built-in Image Analysis – Video or Chemical Images

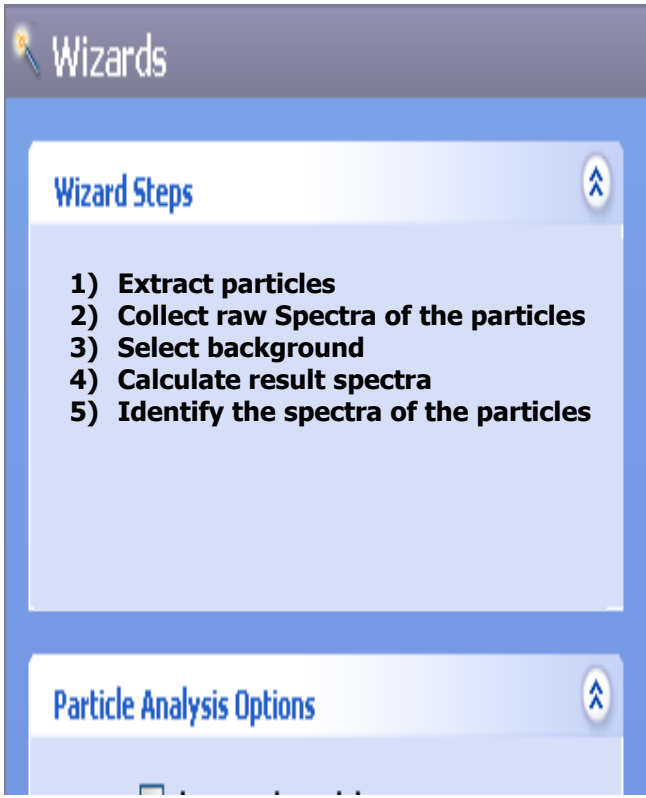
- Standard Features of Omnic Picta and Atlus Software
- Particle size and distribution determinations
- Enables new automation features for microspectroscopy





OMNIC Picta Particle Wizard – Step by Step





Automation of Single-Point Reflectance-Absorbance Experiment

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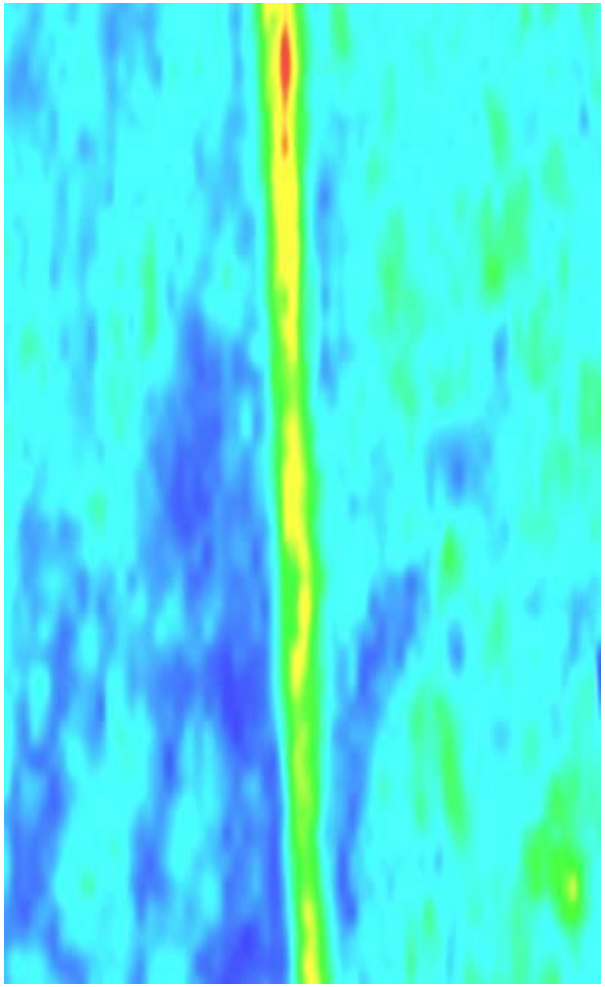
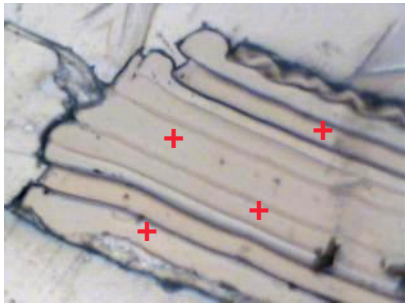
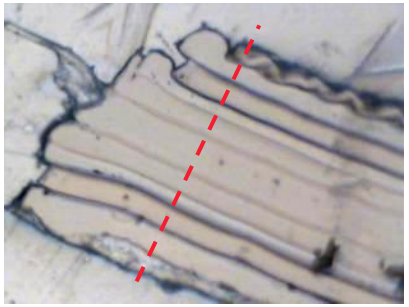
Beyond “Point & Shoot”: Chemical Maps and Images



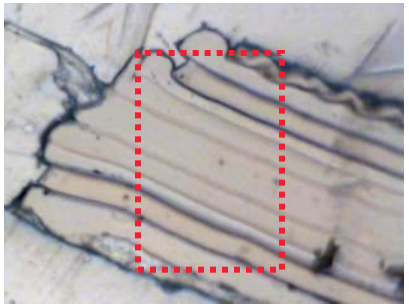
- Discrete points Sampling
  - Autosampling



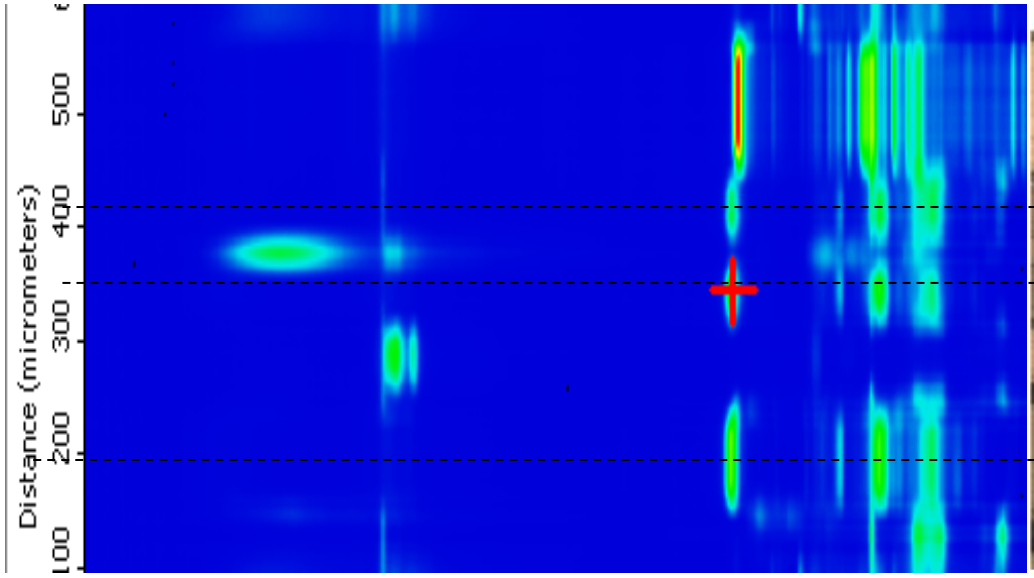
- 1-D Line maps
  - Cross-Sections

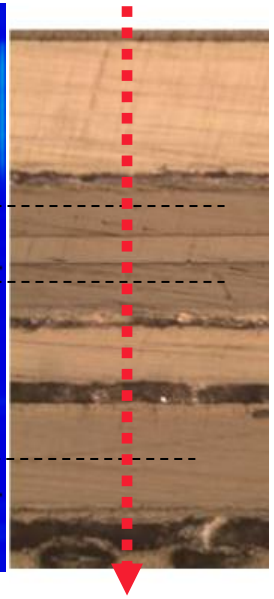


- 2-D Area maps
  - Chemical Images



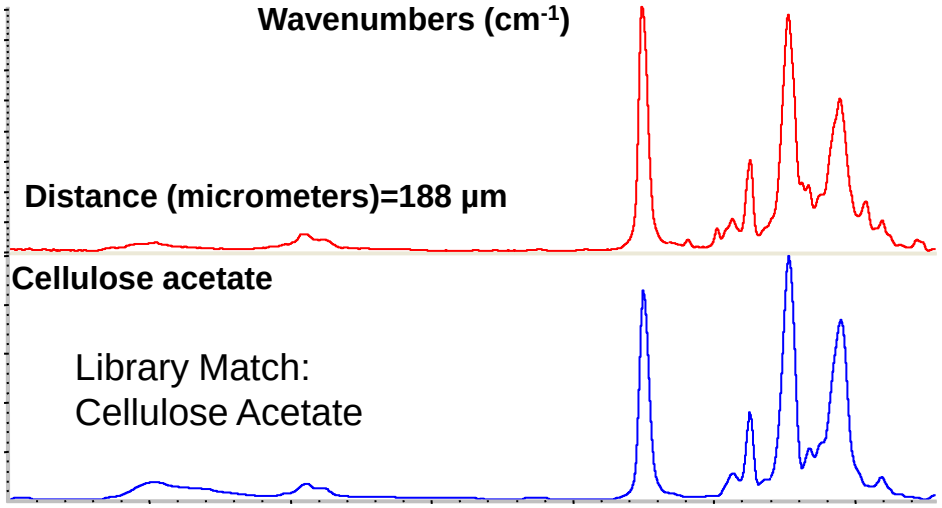
1-D Line Mapping - LCD Polarizer Film Elements





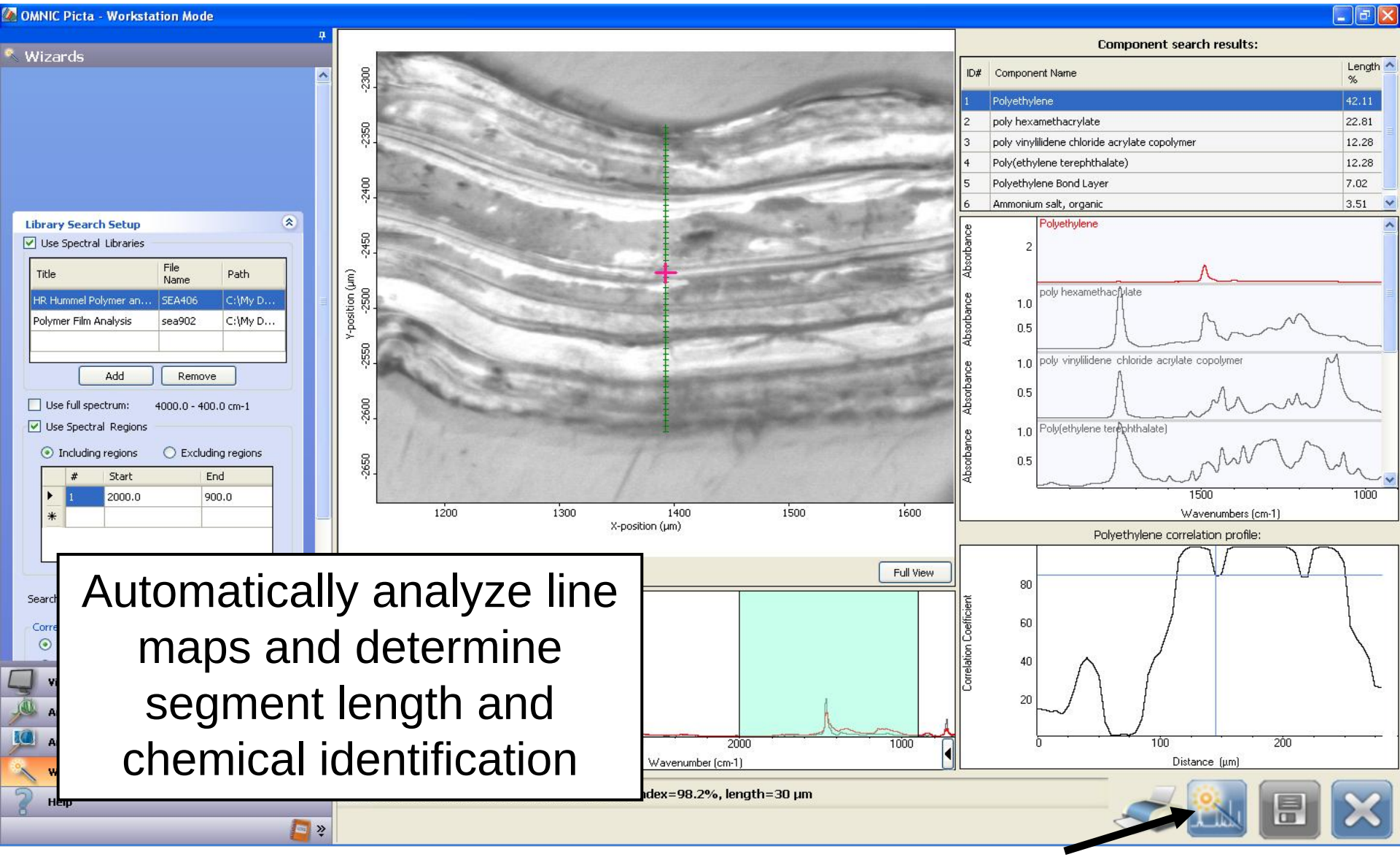
Identical Components

Data Collection



- Silicone Adhesive
- Cellulose Acetate
- Silicone Adhesive
- Poly(2-vinylnorbornane)
- Silicone Adhesive
- Cellulose Acetate
- PVA Copolymer
- Cellulose Acetate
- Silicone Adhesive
- Polyester

Picta Cross Section Analysis Wizard



Automatically analyze line maps and determine segment length and chemical identification

Report Tool

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One Button Report Generation

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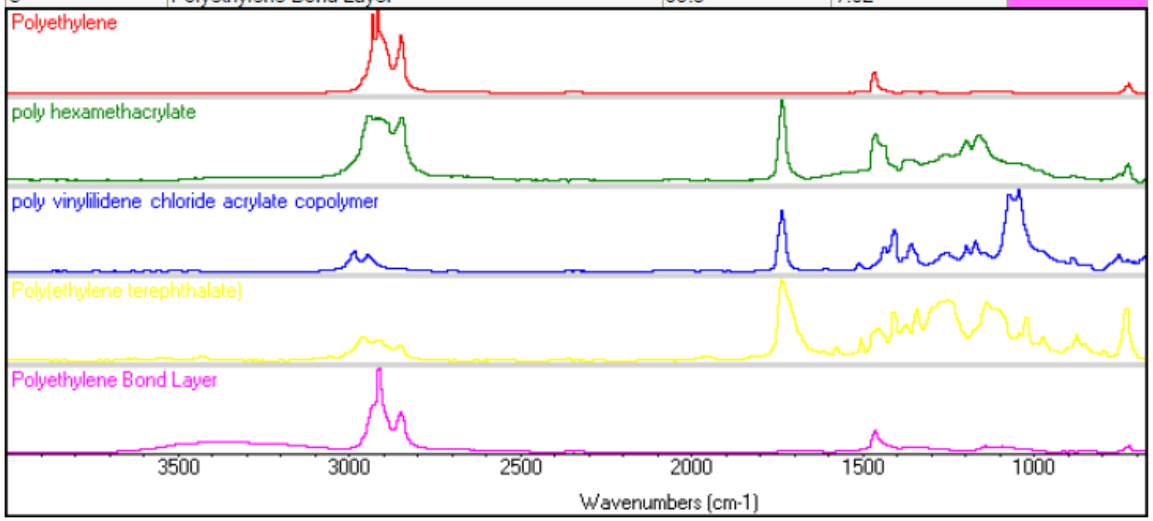
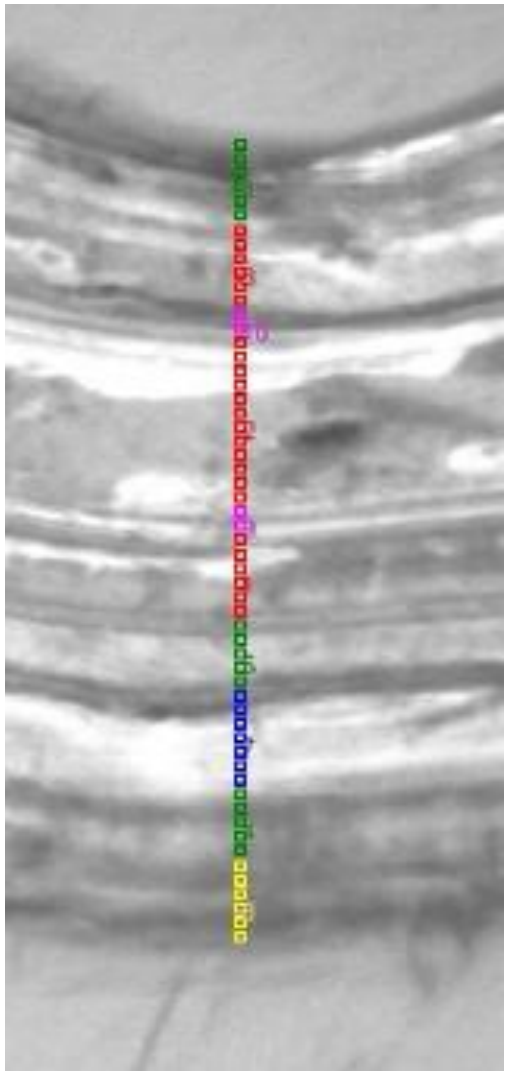
Identified Components

Comp #	Component Name	Match %	Length %	Color
1	Polyethylene	99.4	42.11	
2	poly hexamethacrylate	90.7	28.07	
3	poly vinylilidene chloride acrylate copolymer	97.0	12.28	
4	Poly(ethylene terephthalate)	90.8	10.53	
5	Polyethylene Bond Layer	99.3	7.02	



Identified Segments

Segm #	Comp #	Component Name	Match %	Length (µm)
1	1	Polyethylene	99.1	30.00
2	1	Polyethylene	99.7	60.00
3	1	Polyethylene	98.9	30.00
4	2	poly hexamethacrylate	96.1	25.00
5	2	poly hexamethacrylate	92.9	25.00
6	2	poly hexamethacrylate	84.5	30.00
7	3	poly vinylilidene chloride acrylate copolymer	97.0	35.00
8	4	Poly(ethylene terephthalate)	90.8	30.00
9	5	Polyethylene Bond Layer	99.6	10.00
10	5	Polyethylene Bond Layer	99.1	10.00



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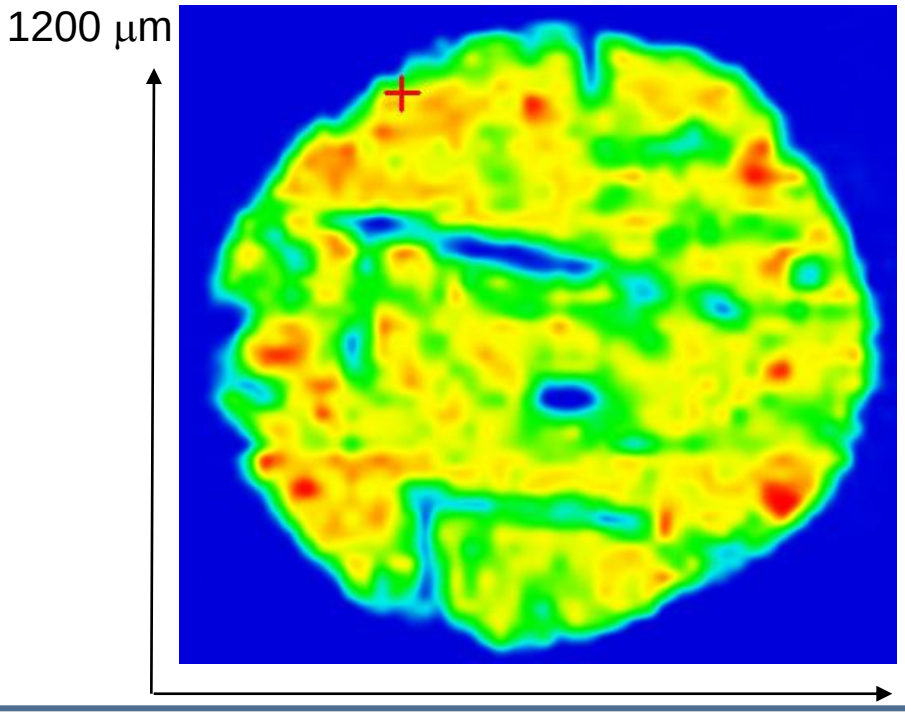
2-D Imaging Hardware Options

- Random Points and 1-D Maps, Speed not a factor
- 2-D Maps can be time intensive, investment pays back
  - Similar analytical problems may be solved with all
    - Times for relative comparison
    - (*area, resolution, averaging time changes absolute collection time*)

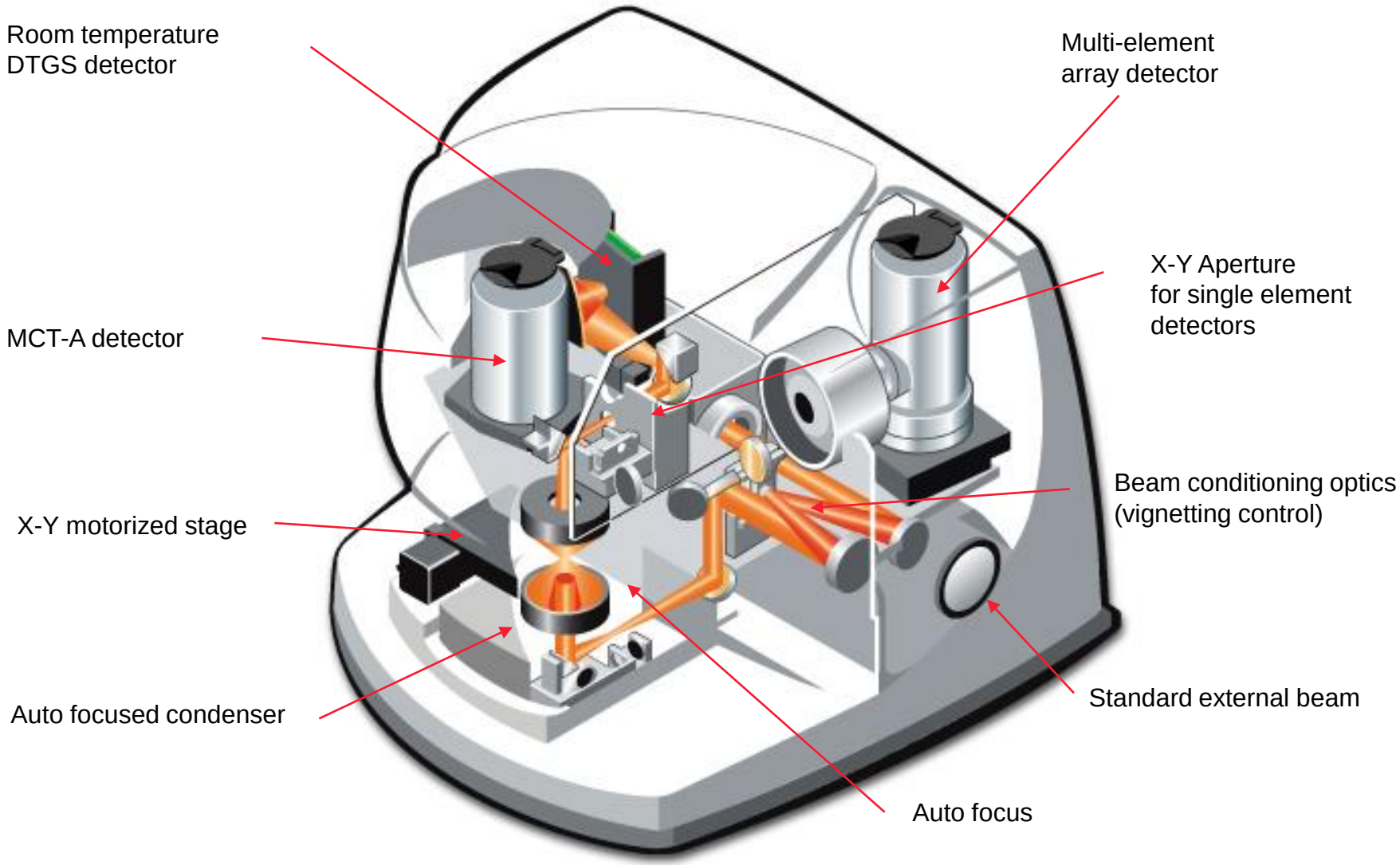


Conventional Mapping
2700 seconds (45 minutes)

Nicolet iN10 MX Ultra-Fast mapping
270 seconds (4.5 minutes)

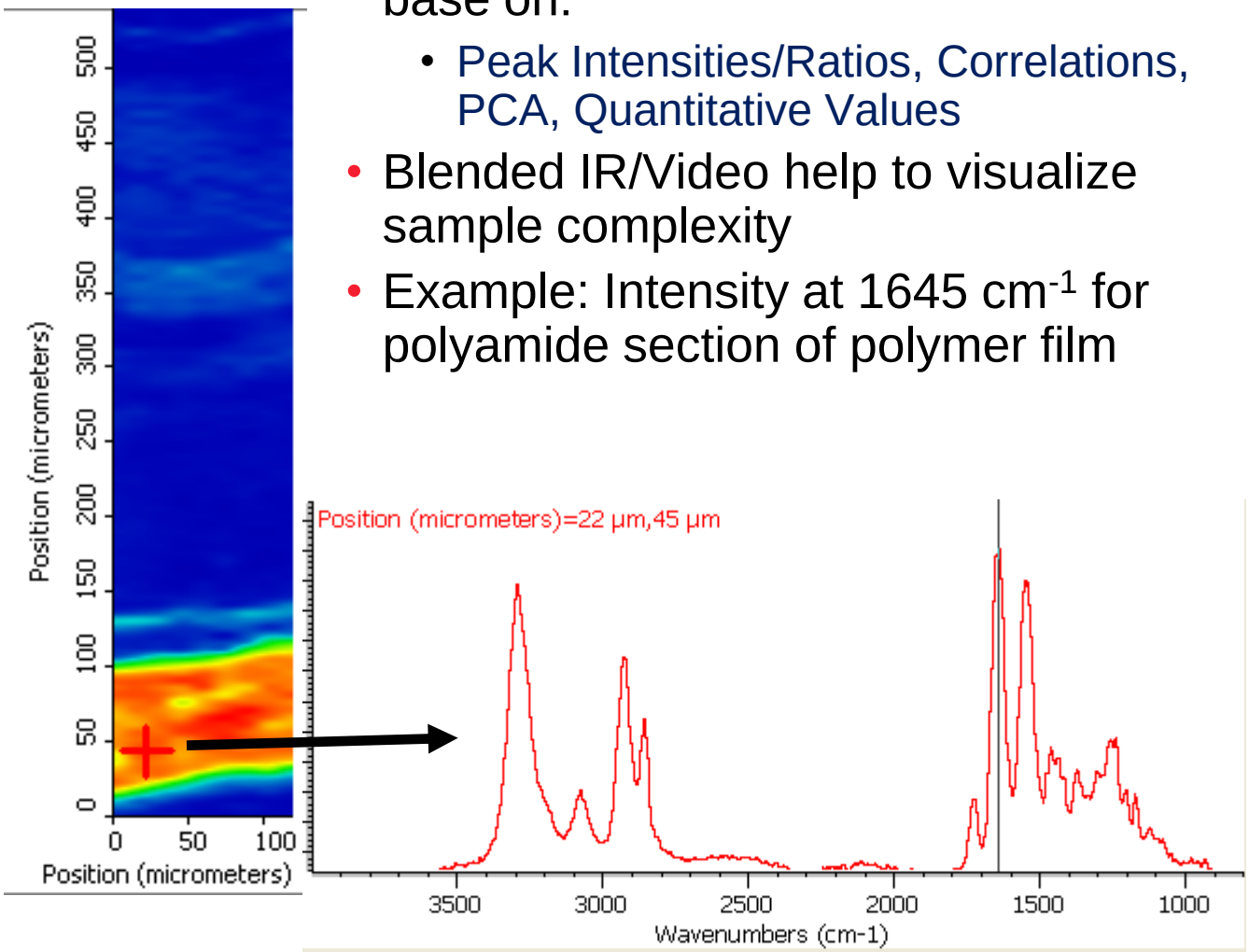
Nicolet iN10 MX Imaging
20 seconds (0.3 minutes)

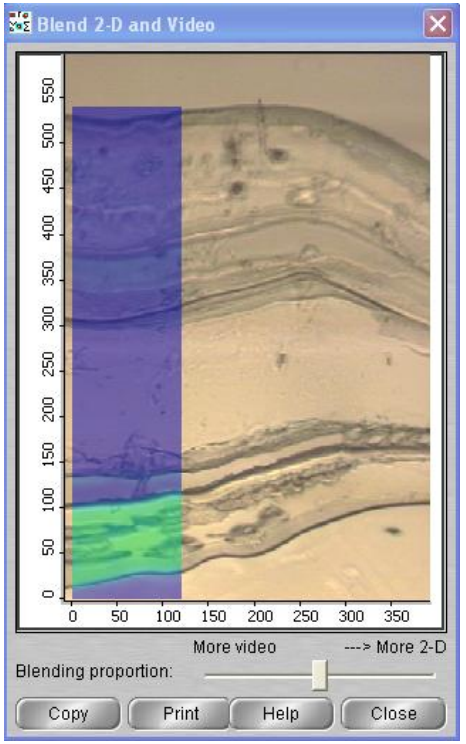
Nicolet iN10 MX – Three Detectors Configuration



2-D Chemical Imaging – Selecting Response Metric

- Intensity indicates specific materials base on:
  - Peak Intensities/Ratios, Correlations, PCA, Quantitative Values
- Blended IR/Video help to visualize sample complexity
- Example: Intensity at 1645 cm^{-1} for polyamide section of polymer film





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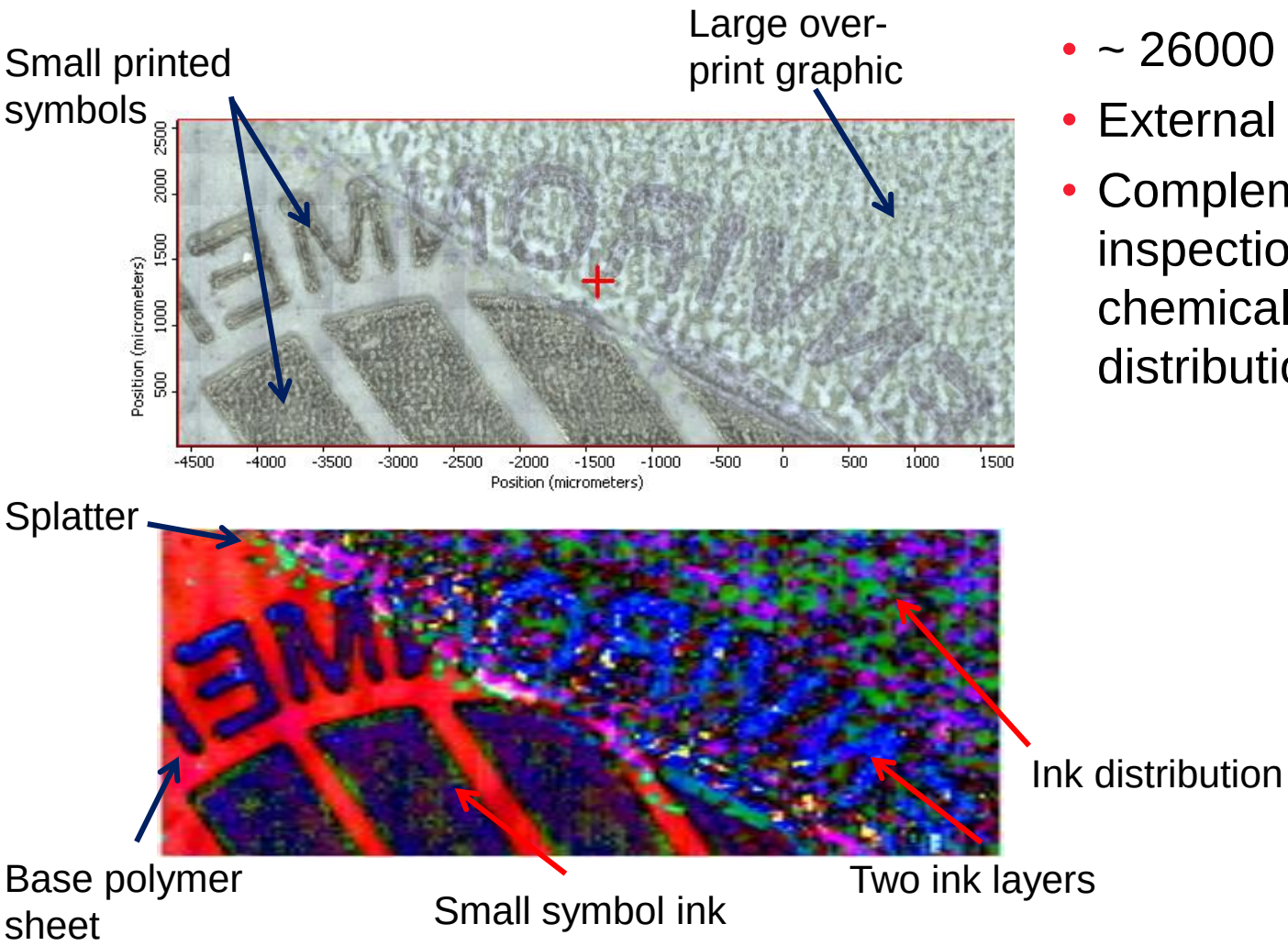
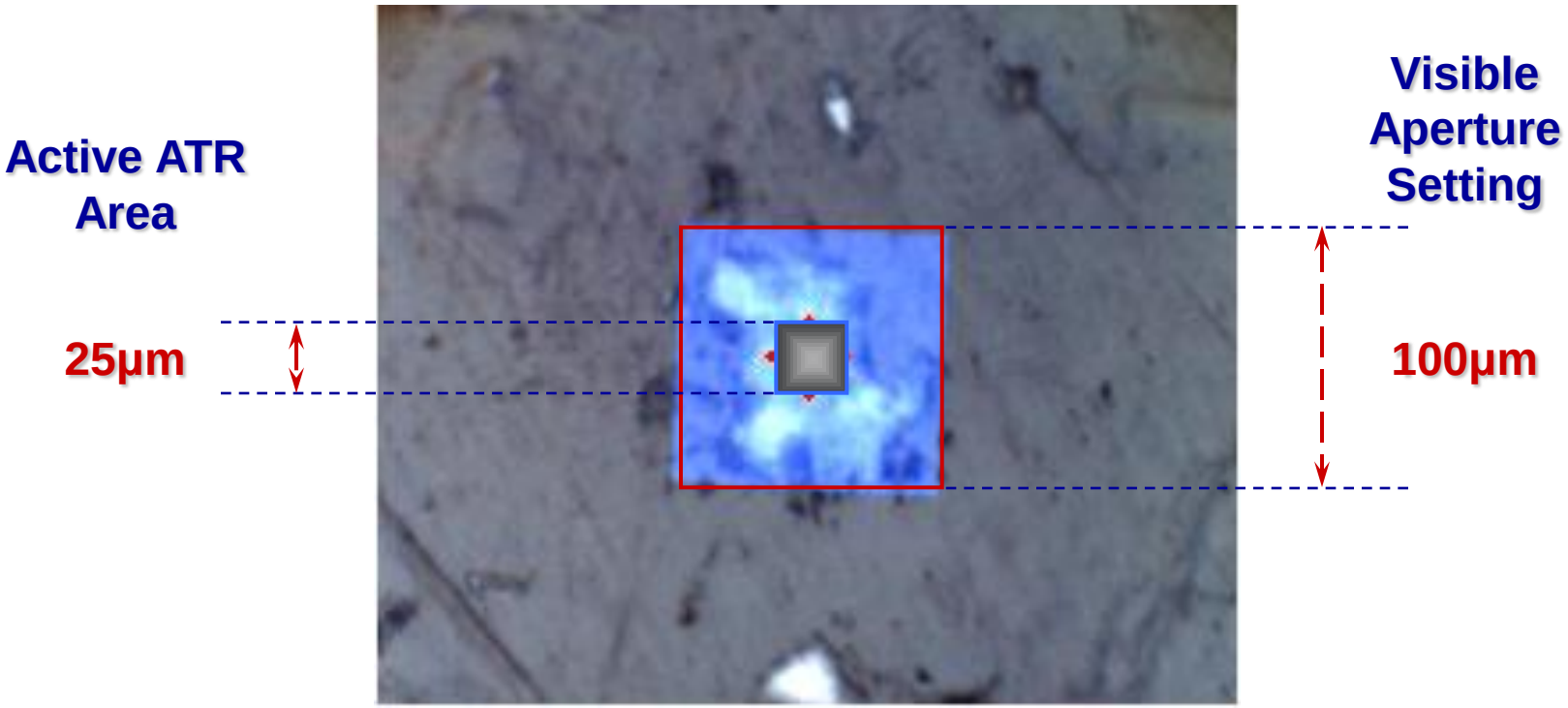
PCA Chemical Imaging of Print



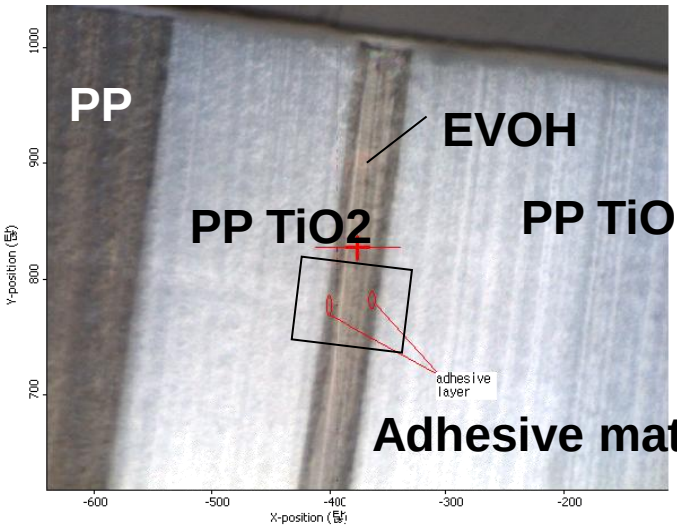
- ~ 26000 spectra
- External reflectance
- Complement visual inspection with chemical derived distribution of inks

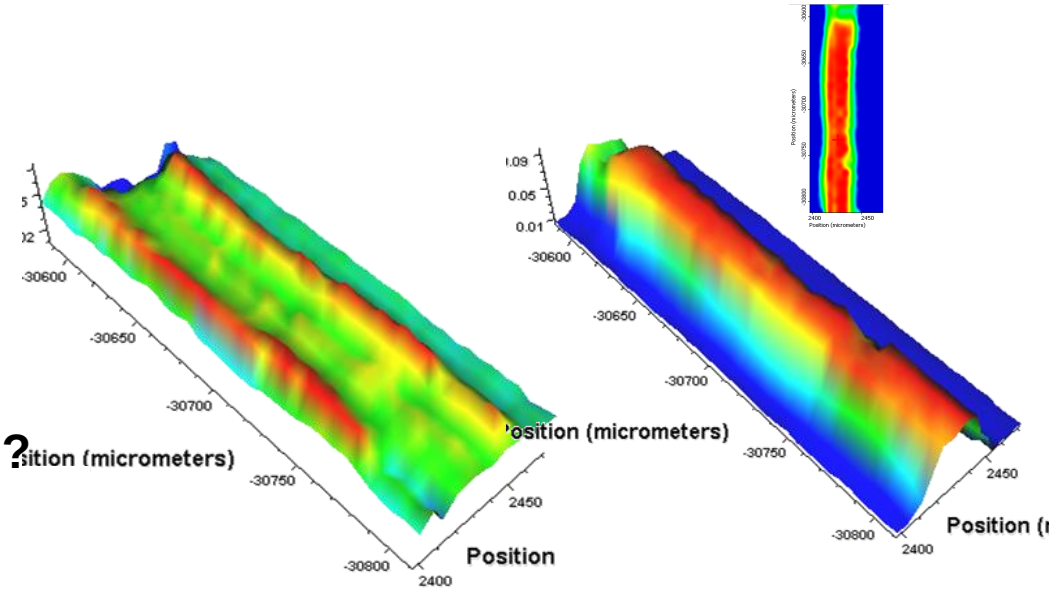
Effective Analysis Area with Micro-ATR

- The design of the Micro Tip ATR projects an image of the aperture on the active internal reflectance surface of the crystal
- Germanium (n = 4) introduces an additional **4X** magnification, focusing the aperture to smaller dimensions



Thin Adhesive layers observed – Tip ATR Imaging

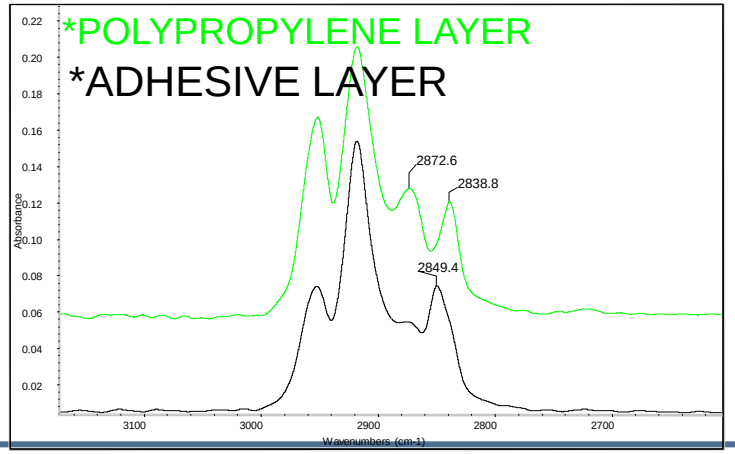




Tie layers

EVOH

- Measured area size: 210 x 70 micron
  - Collection time: less than 10 minutes
  - Resolution 8 cm-1
  - Time per step: 0.7 sec
  - ATR step – 6 microns



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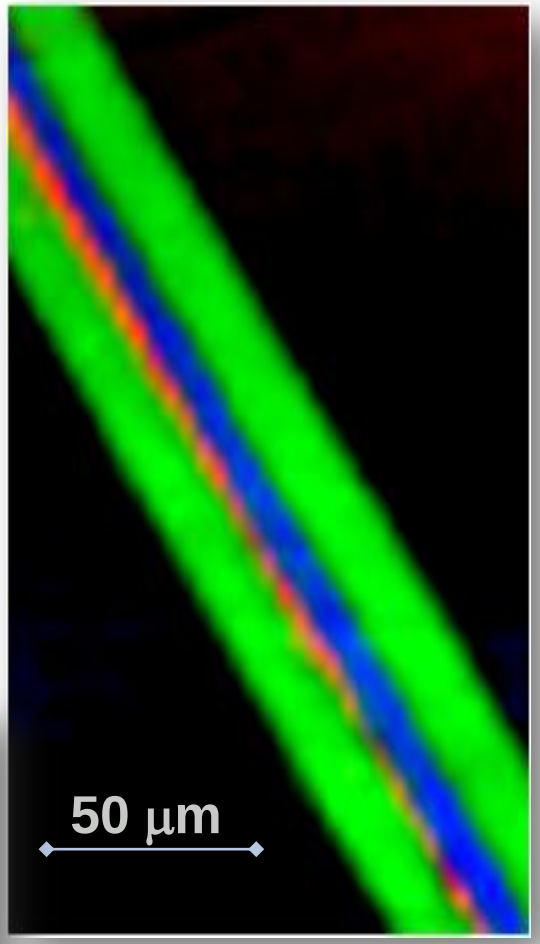
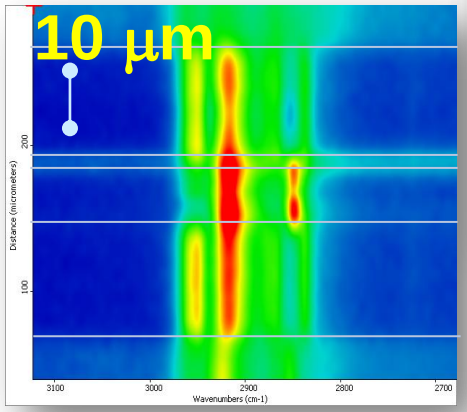
Hemispherical-ATR Imaging of Multi-layer Film

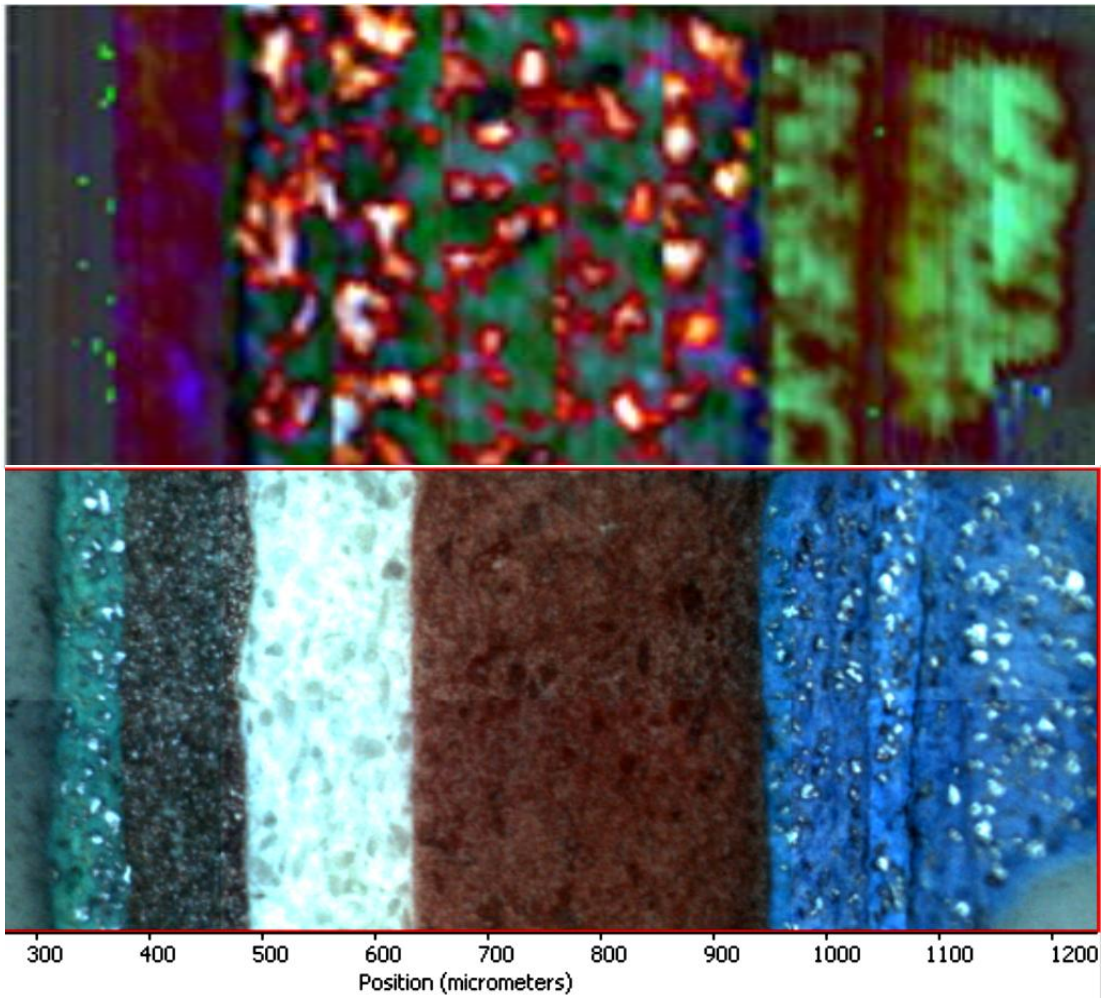
- Area Maps
  - **Classic multilayer film analysis** demonstrates high spatial resolution
  - 4 layers, PP/PE + print layer
  - **4 x 4 micron** pixel in Ge-ATR
- Line Maps of multilayer films have all the information needed
  - Chemical variation is in only one dimension





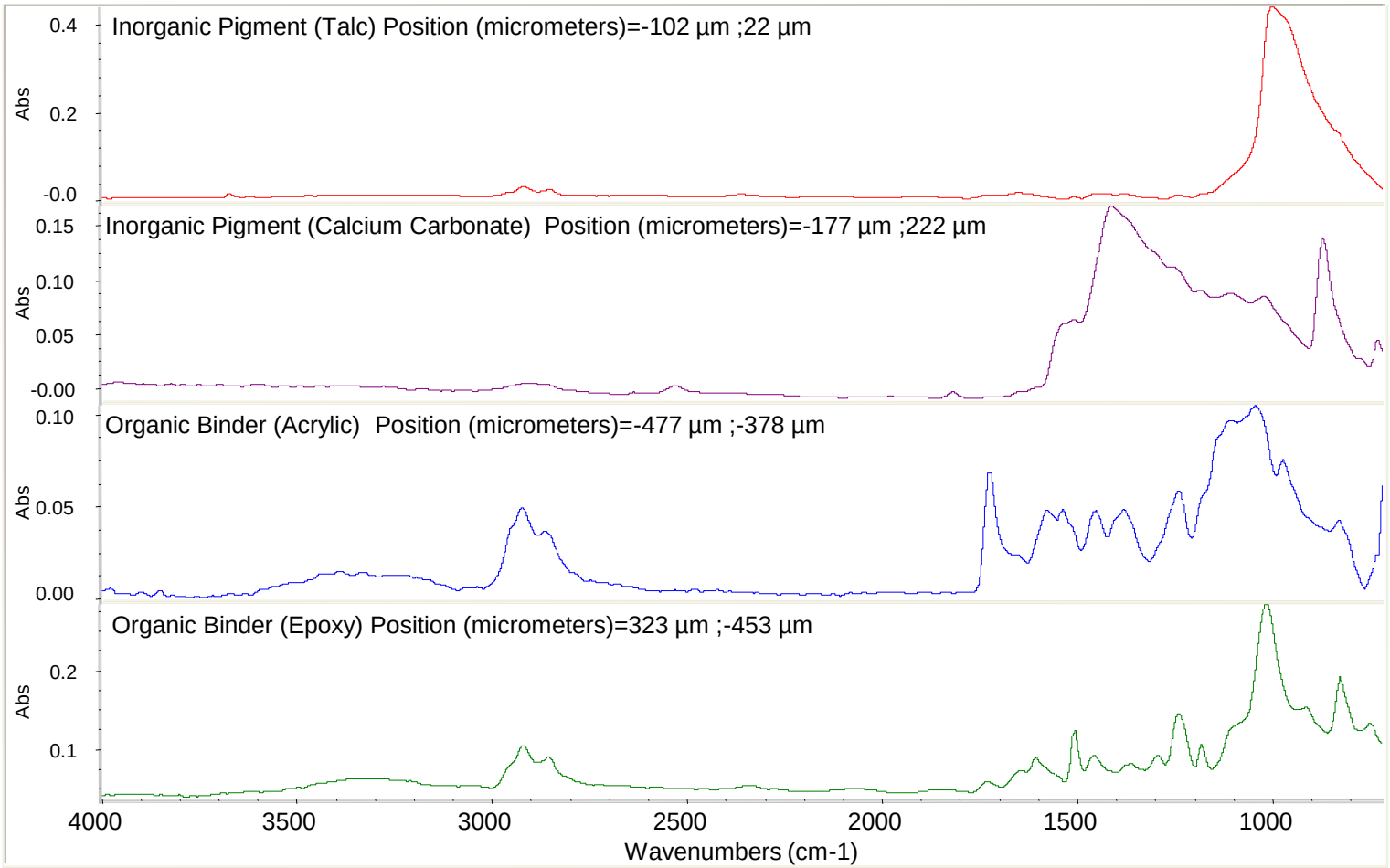
Hemispherical ATR Imaging of Automotive Coating

- Sample has layers
  - 1-D variation
- Sample has variation *within* layers
  - 2-D variation
- ATR Imaging shows *both* sources of variability



Spectra from Paint Chip Image

- Each spectrum each 8 scans (1 second), 6.25 micron spatial resolution



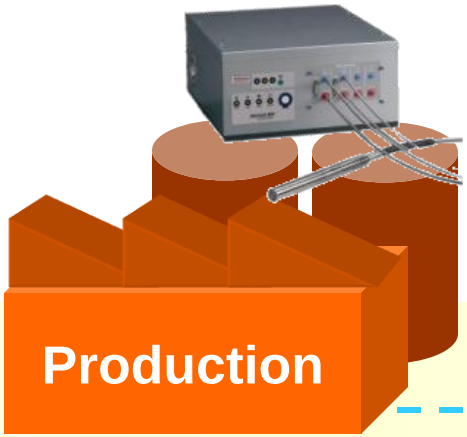
Guided Analysis with OMNIC Picta's Visual Tools

- Video based interface
  - With wide-view navigation and aperture control
- Graphical Slide View tools
  - position specimen for analysis quickly
- Multiple chemical mapping and imaging options
- Image driven analysis tools
  - Autosampling particles
  - Automatic Report Generation of cross sections and random mixtures samples

Thermo Scientific Molecular Spectroscopy

Process NIR

NIR



Instruments for all phases of the manufacturing process

Production

Receiving

Shipping

QC

R&D

FT-IR

FT-IR Microscopy

Raman Microscopy

UV-Vis

Upcoming Thermo Scientific Webinars

- For future and on-demand Webinars, VISIT :
  - www.thermoscientific.com/spectroscopywebinars

- FT-IR
  - Upcoming “Smarter FT-IR Spectroscopy series of webinars”
  - Series starts May 10 and follows every other Thursday through July 12
- Raman
  - Raman Spectroscopy Advances in the Analysis of Carbon Nanotubes, Graphene and Graphene Oxide - May 1
- Near-infrared
  - Introduction to NIR Method Development - Thursday, April 26
- FT-IR/UV-Vis
  - Infrared and UV-Vis Spectroscopy in Pharmaceutical QC - Part 1: Regulatory Consideration - May 17, 2012

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Thank you for your Attention!

Questions?