



**ThermoFisher**  
S C I E N T I F I C

## Home on the (Full) Range: Flexibility and Power in FT-IR

Michael S. Bradley

The world leader in serving science



- FT-IR to Discover
  - Thin Films, Light-weight materials, Polymer-based display panels
- FT-IR to Solve
  - Forensics, Oil and Gas, Analytical Laboratories
- FT-IR to Assure
  - Incoming material, Process Analytics

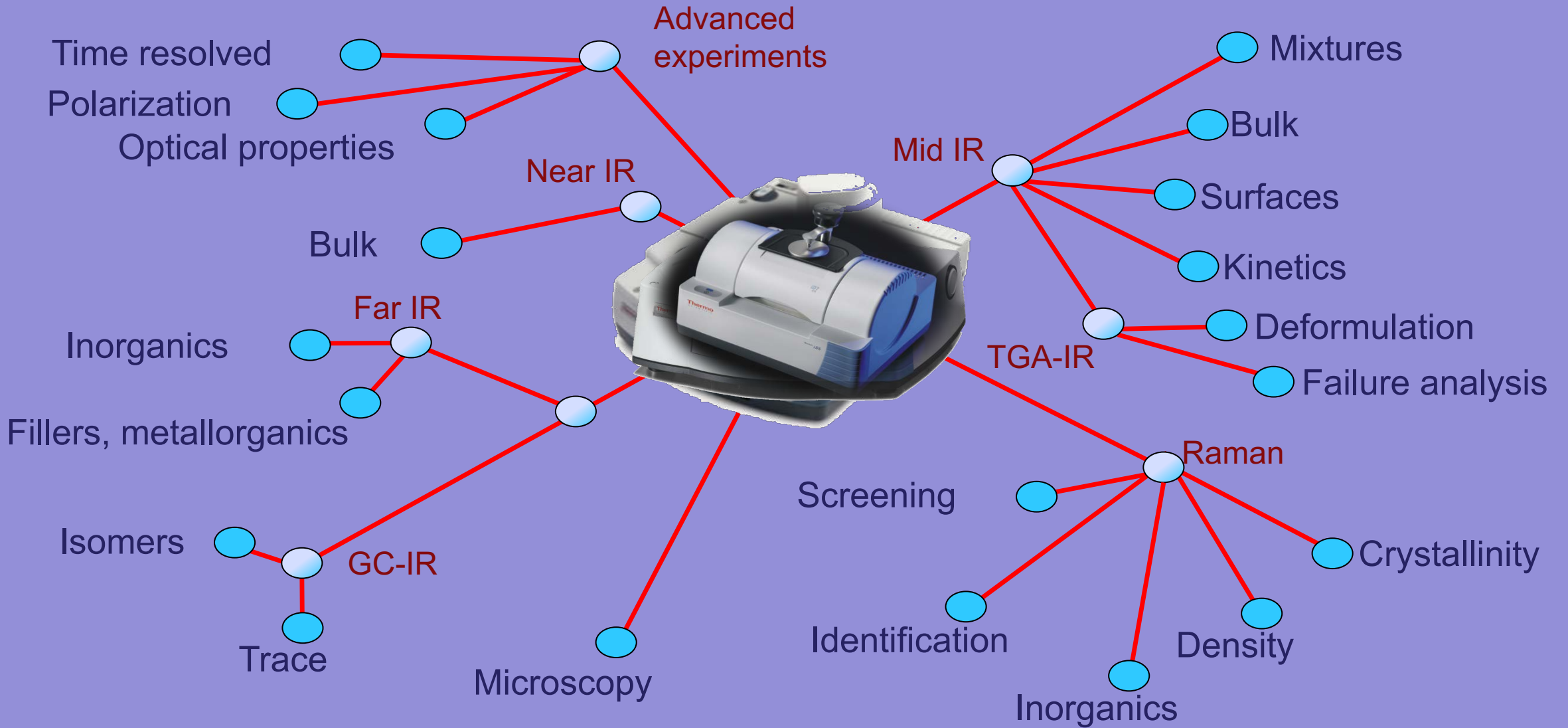
***FT-IR is the ultimate triage technique***

# FT-IR Triage: Stabilize the Situation by Getting Answers Fast.

<b>SYMPTOM</b> <i>What's your problem;                      what do you observe?</i>	<b>POSSIBLE CAUSES</b> <i>What could cause this problem?</i>	<b>SAMPLE TESTING PLAN</b> <i>How do you measure?</i>	<b>DATA ANALYSIS PLAN</b> <i>How do you identify the problem?</i>	<b>RECOMMENDED CONFIGURATION</b> <i>What to use?</i>
<b>Bloom</b>	Improper additive formulation – excess or un-reacted additive	1. Scrape material from surface 2. Measure by single-bounce ATR	1. Search libraries to identify the unknown material 2. Adjust formulation based on identified material	<ul style="list-style-type: none"> <li>Nicolet iS10 FT-IR spectrometer</li> <li>Smart iTR Diamond ATR accessory</li> <li>OMNIC Specta software for Polymer Labs</li> </ul>
<b>Hazing/streaking/incorrect color (white or black)</b>	Improper formulation: additives or fillers; contamination, poor mixing	1. Measure directly or excise outer or inner material from sample 2. Measure using diamond ATR Mid-IR or Far-IR for inorganic fillers	1. Compare to reference part data and search libraries to identify differences 2. Change formulation if appropriate	<ul style="list-style-type: none"> <li>Nicolet iS50 FT-IR spectrometer</li> <li>Built-in Diamond ATR accessory</li> <li>Solid-substrate beamsplitter</li> <li>OMNIC Specta software for Polymer Labs</li> </ul>
<b>Oily or tacky surface</b>	Improper additive formulation or contamination	1. Wipe or scrape surface to isolate material or direct analysis 2. Measure residue or sample surface on single bounce ATR 3. Measure reference part or sample with surface cut off	1. Search libraries to identify material 2. Adjust formulation or change process to avoid contamination	<ul style="list-style-type: none"> <li>Nicolet iS5 FT-IR spectrometer</li> <li>iD5 Diamond ATR accessory</li> <li>OMNIC Specta software for Polymer Labs</li> </ul>
<b>Inclusions, de-lamination, fish eyes (complex)</b>	Poor processing, contamination	1. Isolation of included contaminants 2. Sample cross-sectioning 3. Perform microscopic analysis a. FT-IR	1. Search libraries to identify contamination 2. Change process to avoid contamination	<ul style="list-style-type: none"> <li>Nicolet iN10 FT-IR microscope</li> <li>OMNIC Specta software for Polymer Labs</li> </ul> OR <ul style="list-style-type: none"> <li>DXR Raman microscope</li> <li>OMNIC Specta software for Raman Analytics</li> </ul>
<b>Roughness, speckles, mars, bubbles</b>	Contamination: surface or embedded processing problem (trapped gas)		1. Search libraries to identify contamination 2. Change process to avoid contamination	<ul style="list-style-type: none"> <li>Nicolet iS10 FT-IR spectrometer</li> <li>Smart iTR Diamond ATR accessory</li> <li>OMNIC Specta software for Polymer Labs</li> </ul>
<b>Brittle, cracking, weakness</b>	Oxidation, degradation, contaminant, incorrect material		1. Compare to reference part 2. Identify unexpected components 3. Ensure material is correct for conditions; change formulation as needed	<ul style="list-style-type: none"> <li>Nicolet iS10 FT-IR spectrometer</li> <li>Smart iTR Diamond ATR accessory</li> <li>OMNIC Specta software for Polymer Labs</li> </ul>
<b>Diminished</b>	Crystallinity, structure, polymorphism	Measure directly using	1. Search libraries using spectral region search to	Nicolet iS50 FT-IR spectrometer



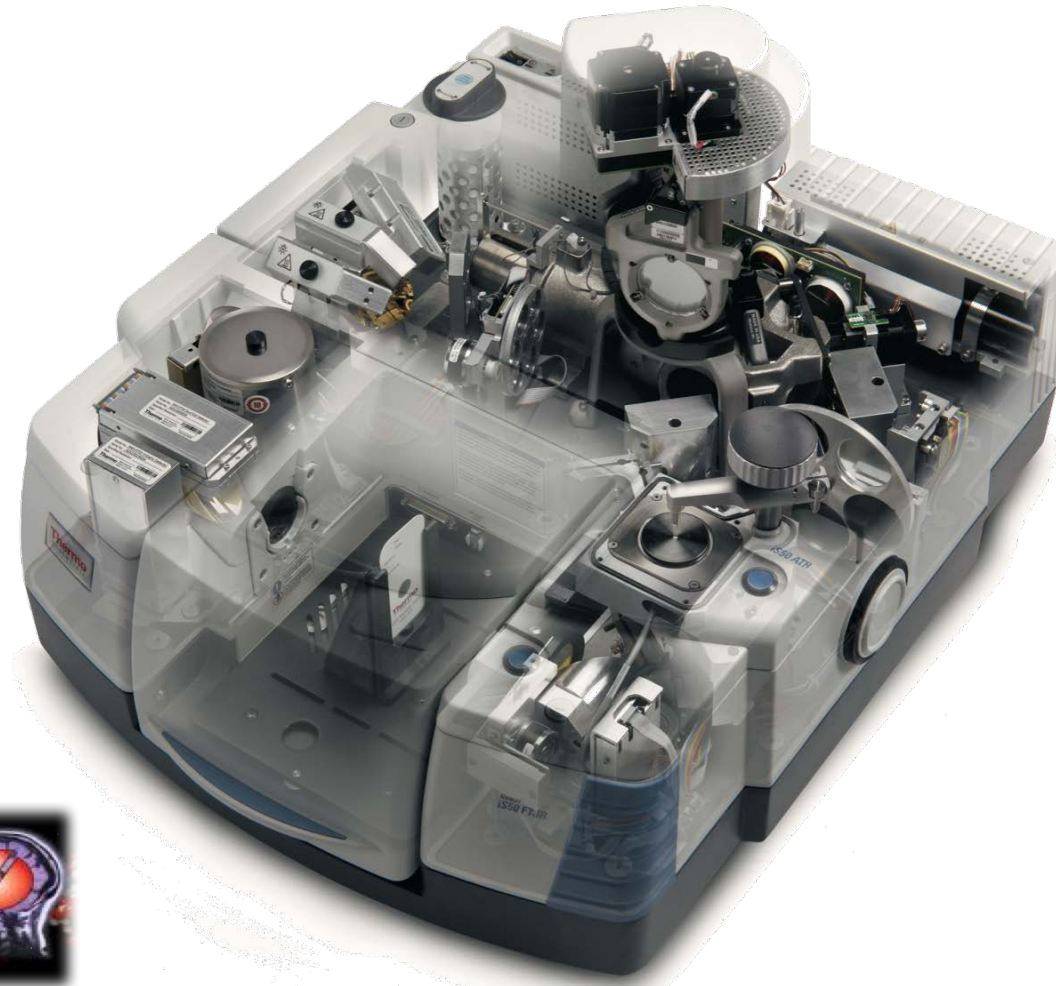
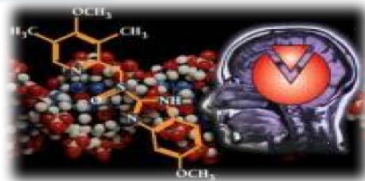
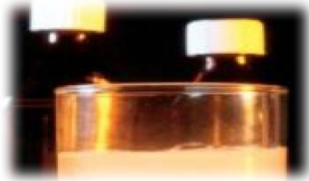
# Select the Right Tool



# Analytical Workstation: Discover, Solve and Assure on One Platform

## The Thermo Scientific™ Nicolet™ iS™ 50 FT-IR Spectrometer

Thinking done inside the Box!

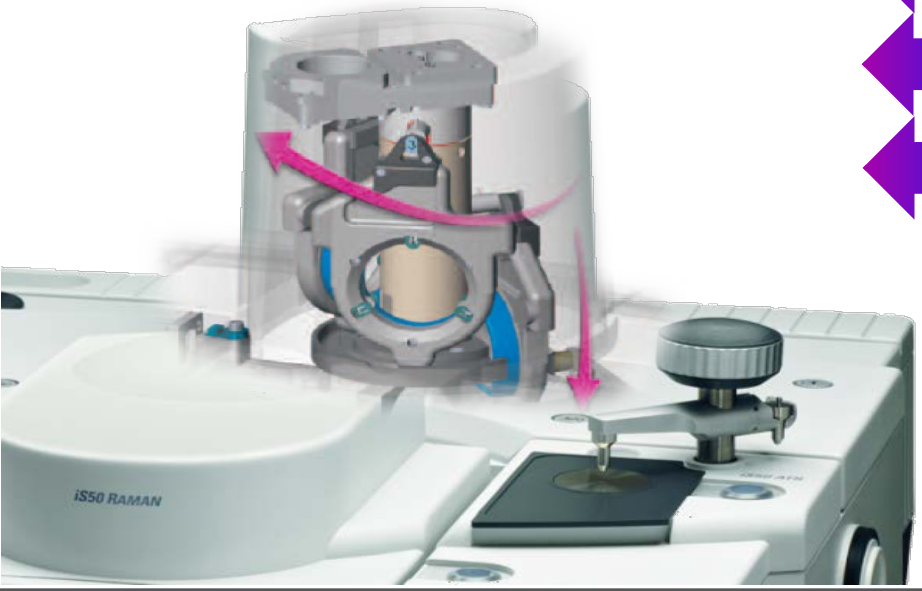
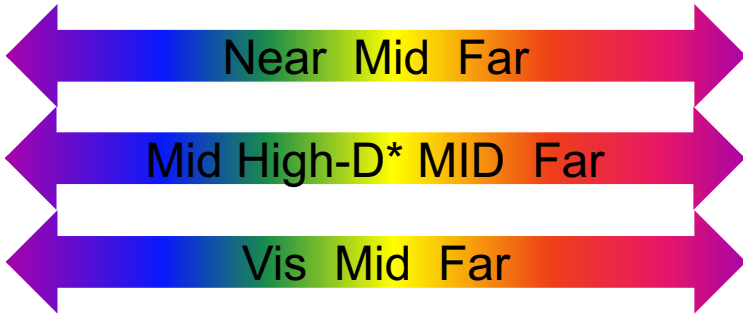


# Flexibility – Automated Multi-range

- iS50 ABX: Automatic Beamsplitter eXchanger

- Triple Detector Mount

- Automated Multi-range control



# Integration – ATR and FT-Raman Made Simple

- ATR and Transmission Simultaneously
  - Without needing to swap accessories
  - Always ready and purged/desiccated
  - Monolithic diamond: mid-IR and far-IR



- Sample compartment FT-Raman
  - Touch Point Access
  - Plug and Play
  - Cost effective

# Going Beyond – Multimodal Analysis

- Microbeads, chemical homogeneity
  - FT-IR Microscopy
- Chemical basis of viscoelastic properties
  - Rheometry – FT-IR
- Deformulation
  - TGA-IR
- Cannabinoids and Bath Salts
  - GC-IR module







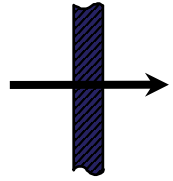
**ThermoFisher**  
S C I E N T I F I C

**Discover, Solve and Assure**

The Nicolet iS50 as a Solution Generator

The world leader in serving science

# Full Suite of Tools



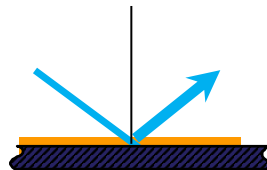
- Over 60% still use Transmission
  - The method for many analyses
  - Great sensitivity



- Over 70% use ATR
  - Simplicity in sampling
  - Sensitivity has greatly improved

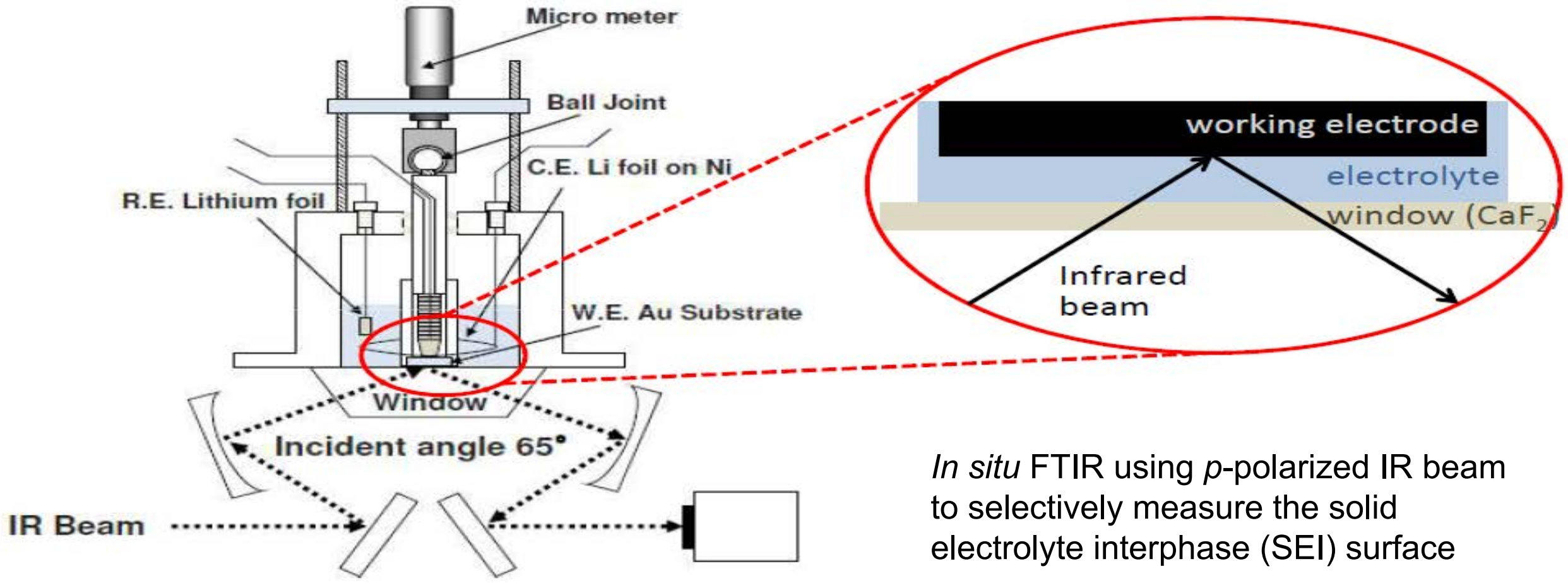


- Diffuse Reflectance still needed
  - Specified in some applications



- Specular Reflection has unique application
  - Thin Films, Reflection-Absorption



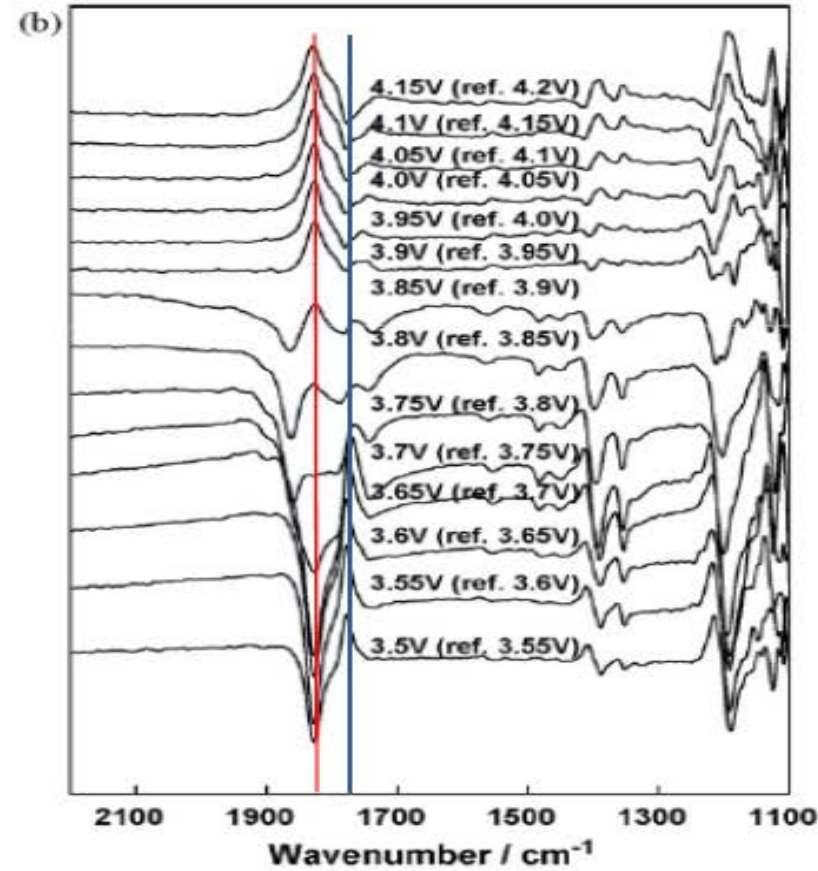
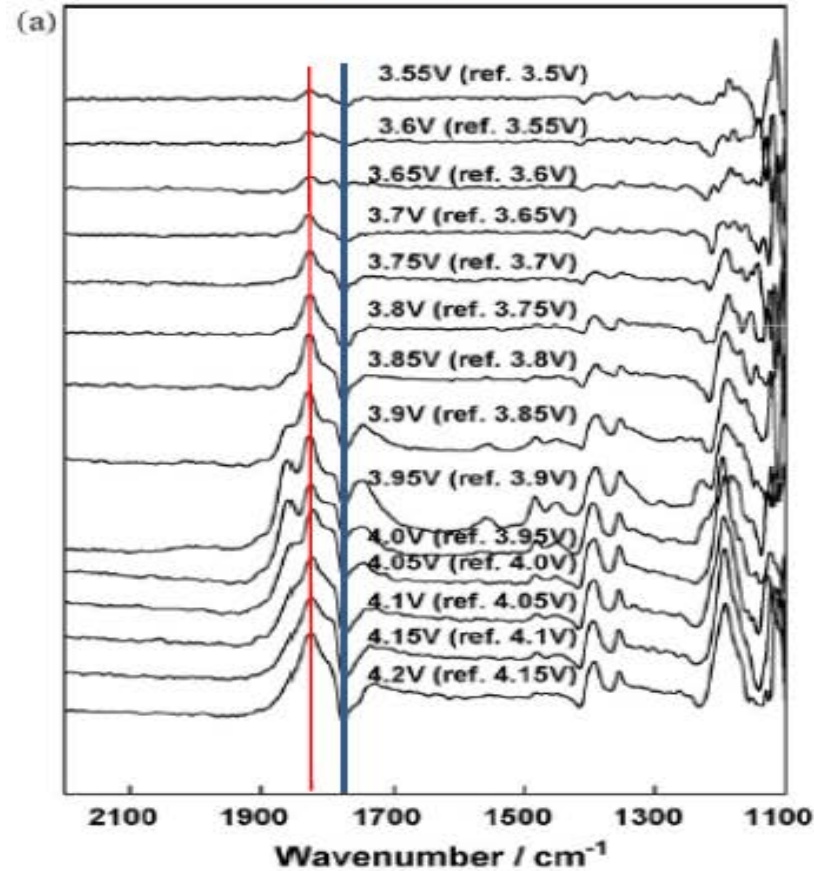


*In situ* FTIR using *p*-polarized IR beam to selectively measure the solid electrolyte interphase (SEI) surface

Dynamic behavior of surface film on LiCoO<sub>2</sub> thin film electrode.  
M. Matsui, K. Dokko, and K. Kanamura, Tokyo Metropolitan University  
*J Power Sources* 2008, **177**, 184.

charge

discharge



Peak assignment for *in situ* FTIR spectra for the electrochemical oxidation of propylene carbonate containing  $1.0 \text{ mol dm}^{-3} \text{ LiClO}_4$  on the  $\text{LiCoO}_2$  thin film

$\text{cm}^{-1}$	Upward peaks
1830	C=O stretching vibration in PC
1565	O-C-O bending vibration in PC
1485	CH <sub>2</sub> wagging vibration in PC
1455	CH <sub>3</sub> asymmetric bending in PC
1395	O-CH <sub>2</sub> wagging vibration in PC
1355	CH <sub>3</sub> symmetric bending vibration in PC
1190	C-O-C asymmetric stretching vibration in PC
Downward peaks	
1780	C=O symmetric stretching vibration in decomposition products
1420	CH <sub>2</sub> bending or CO <sub>2</sub> symmetric stretching vibration in decomposition products
1375	CH <sub>3</sub> symmetric bending vibration in decomposition products
1235	C-O-C asymmetric stretching vibration in decomposition products

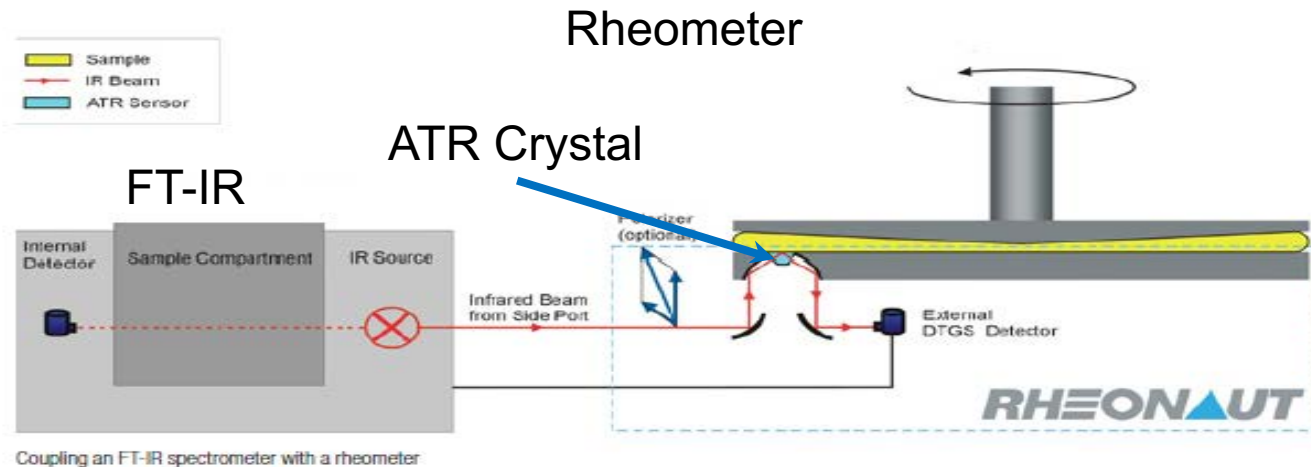
Differential spectra:

Positive band: species decrease

Negative peak: species increase

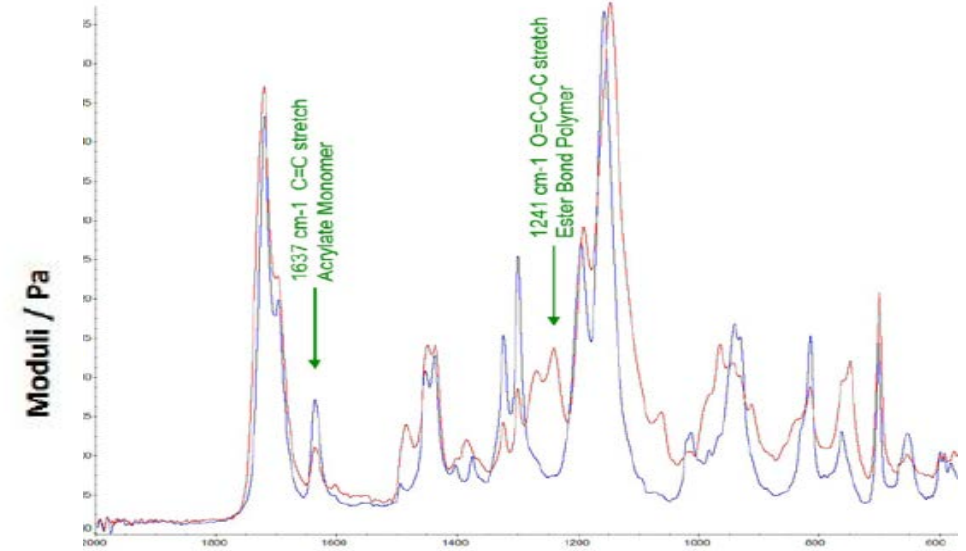
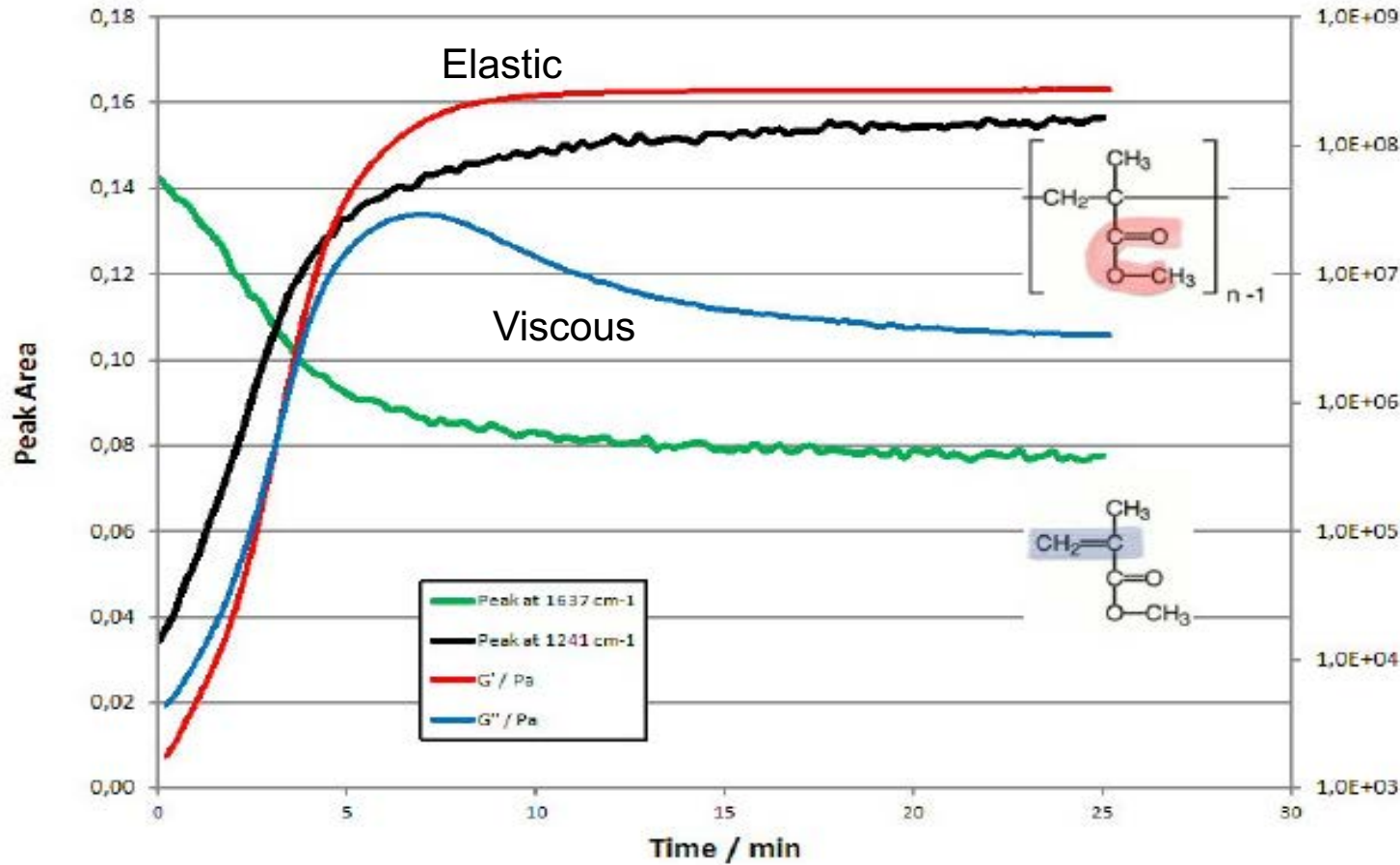
→ reversible surface film formation

- Steady-state and time dependent viscoelastic properties
  - As a function of stress or strain
  - Measure  $G'$  (shear storage modulus) and  $G''$  (shear loss modulus)
- The viscoelastic response depends upon the molecular properties
  - Which is what FT-IR and Raman probe!
- Obtain physical and chemical information simultaneously



Rheonaut with Thermo Scientific™  
Nicolet™ iS™ 10 FT-IR

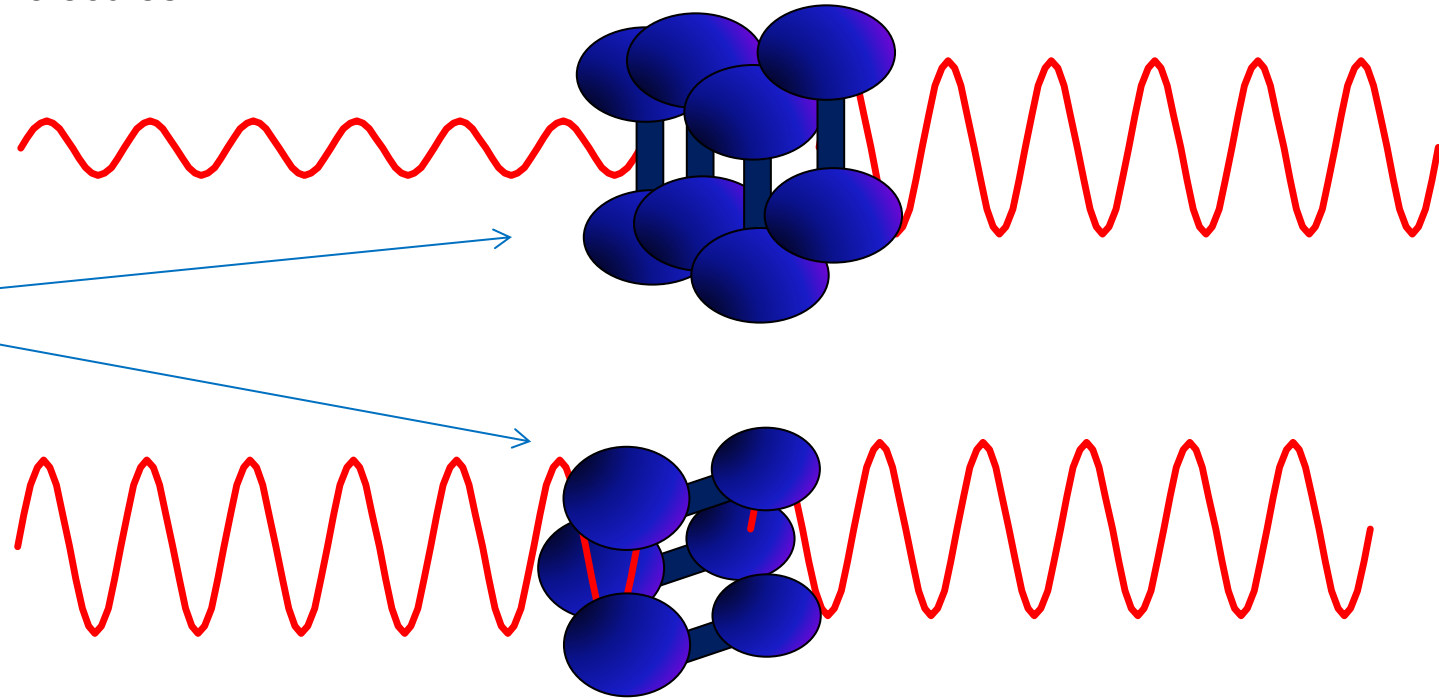


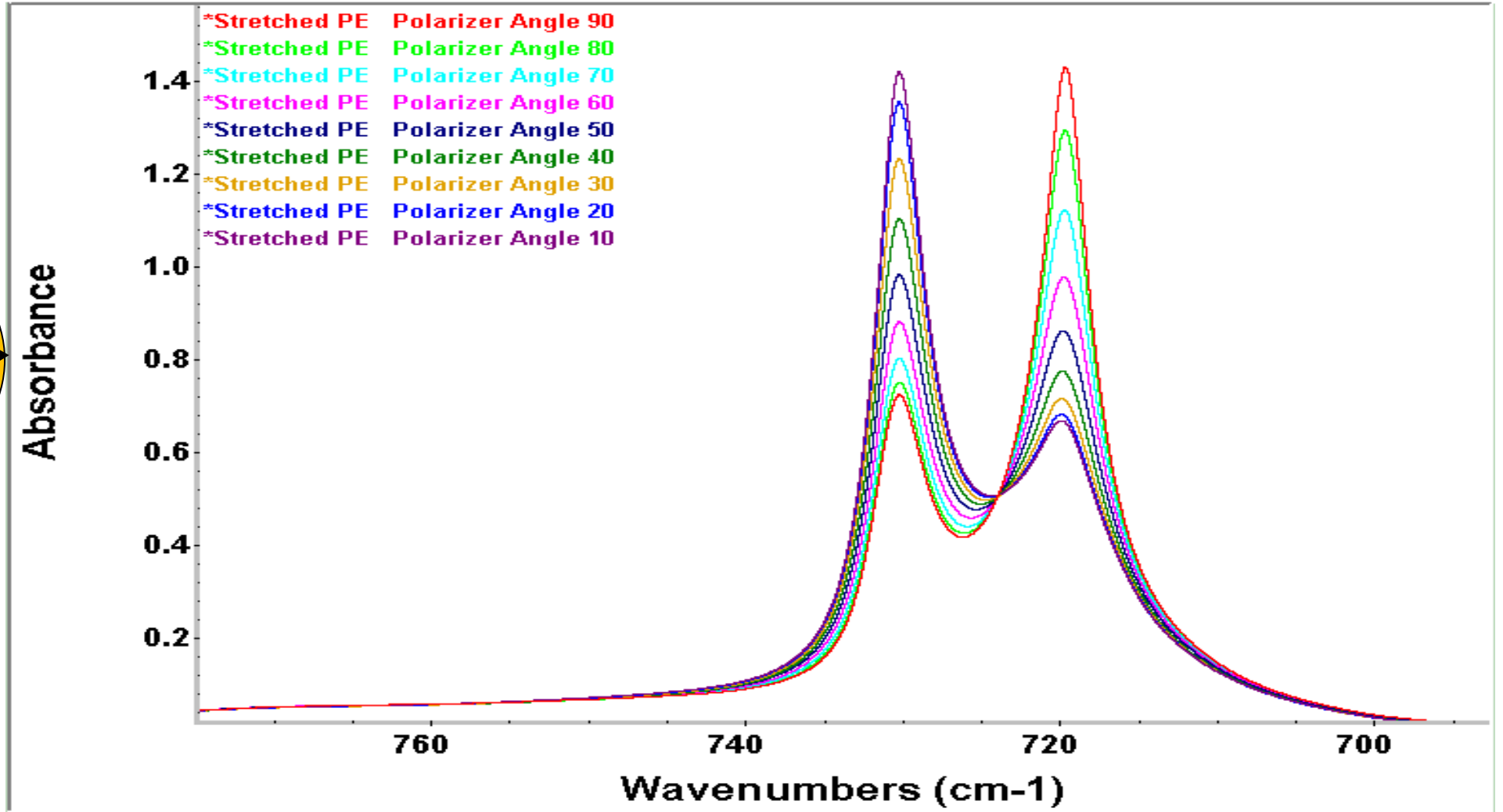
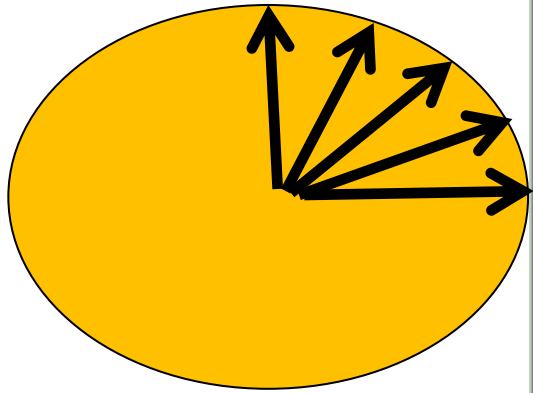


- Correlating FT-IR Spectra to G' and G''
- When G' = G'', Gel Point for cure
- Monomer: Green (dropping), Ester: Black (rising)

- Polymers are often stretched along an axis
  - IR absorption is sensitive to the orientation of the molecules

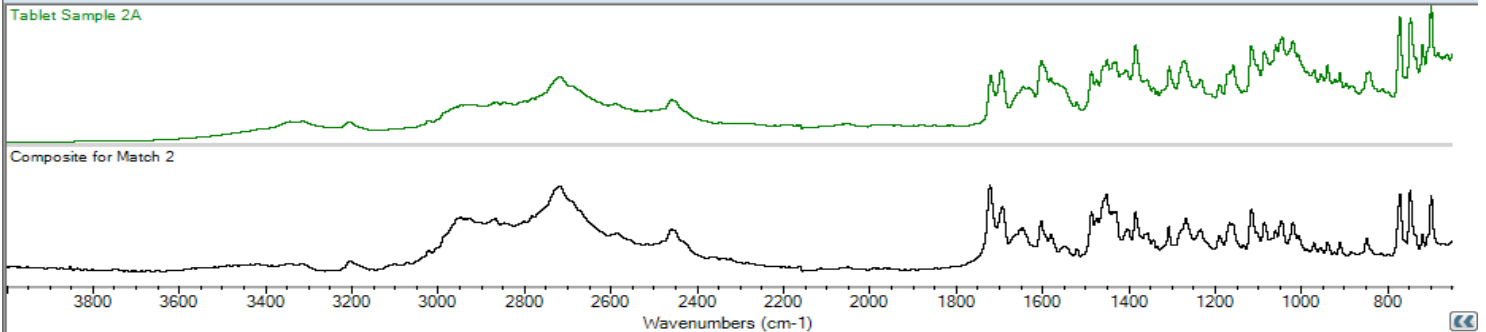
- Spectra depend upon bond orientation



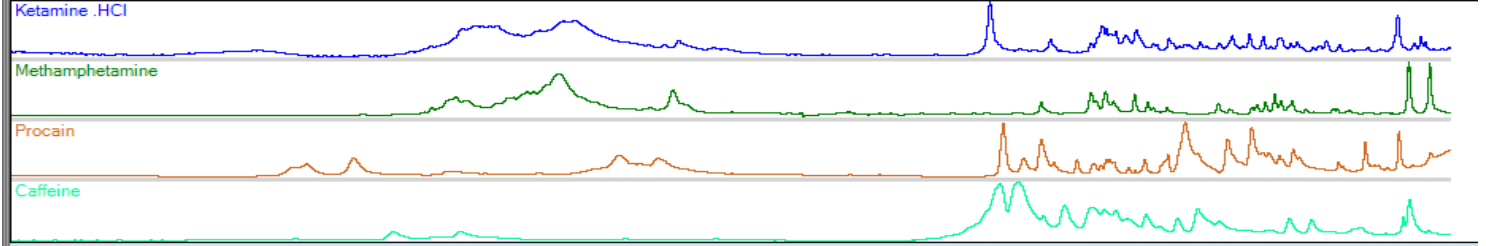




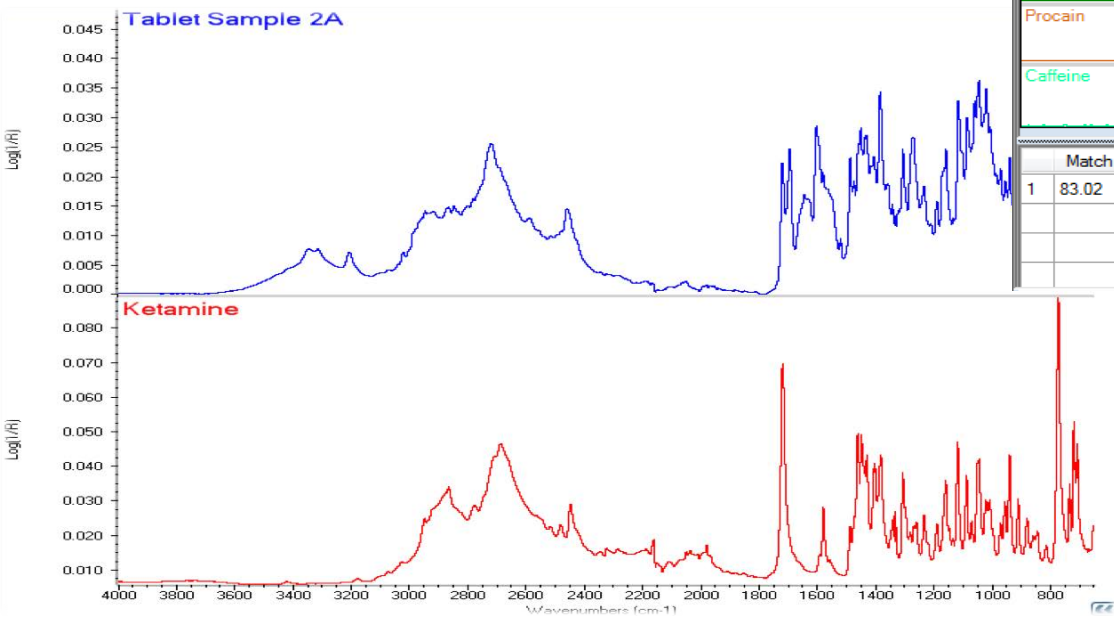
# Drug Identification in the Forensics Laboratory

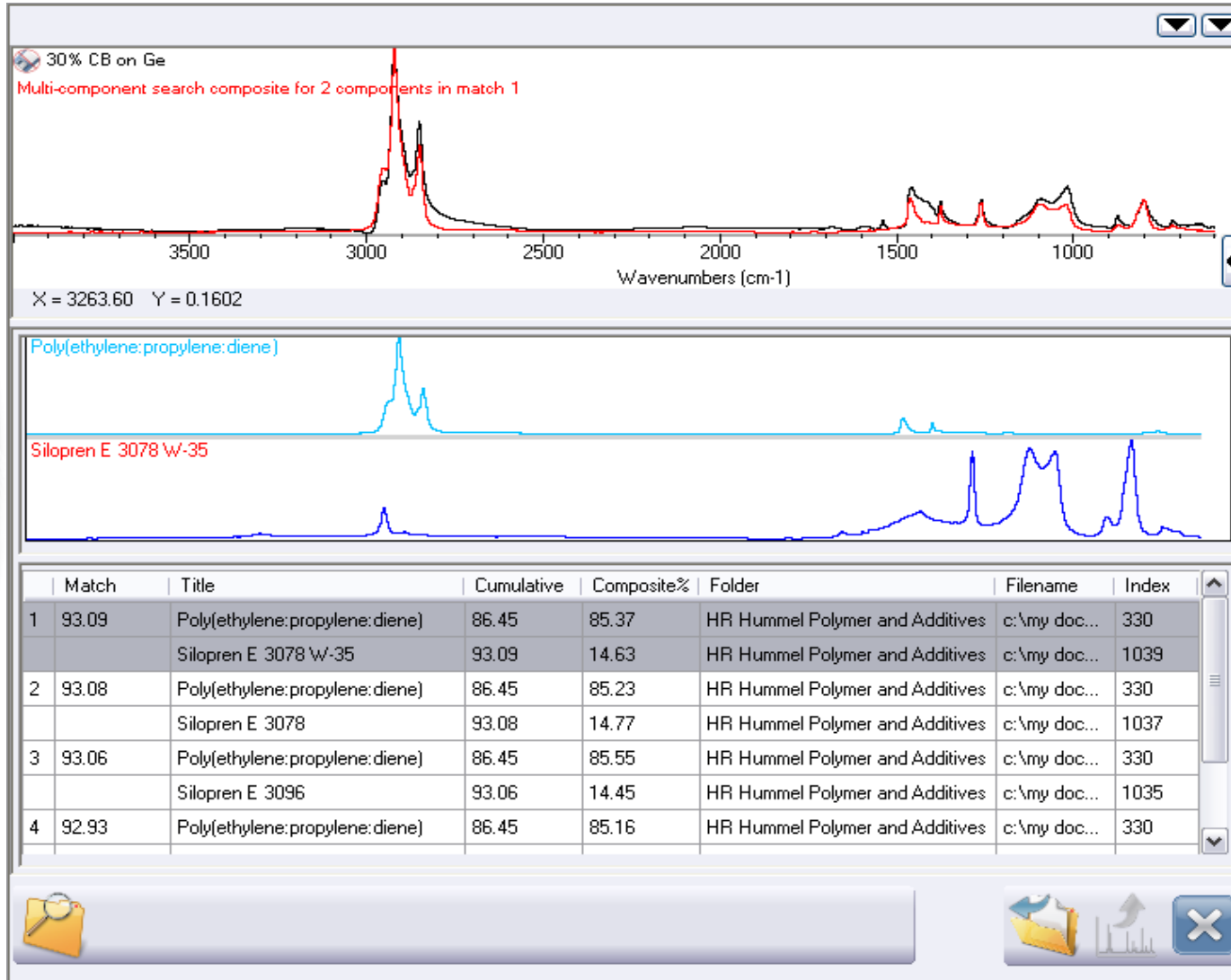


X = 3981.41 Y = 0.0230

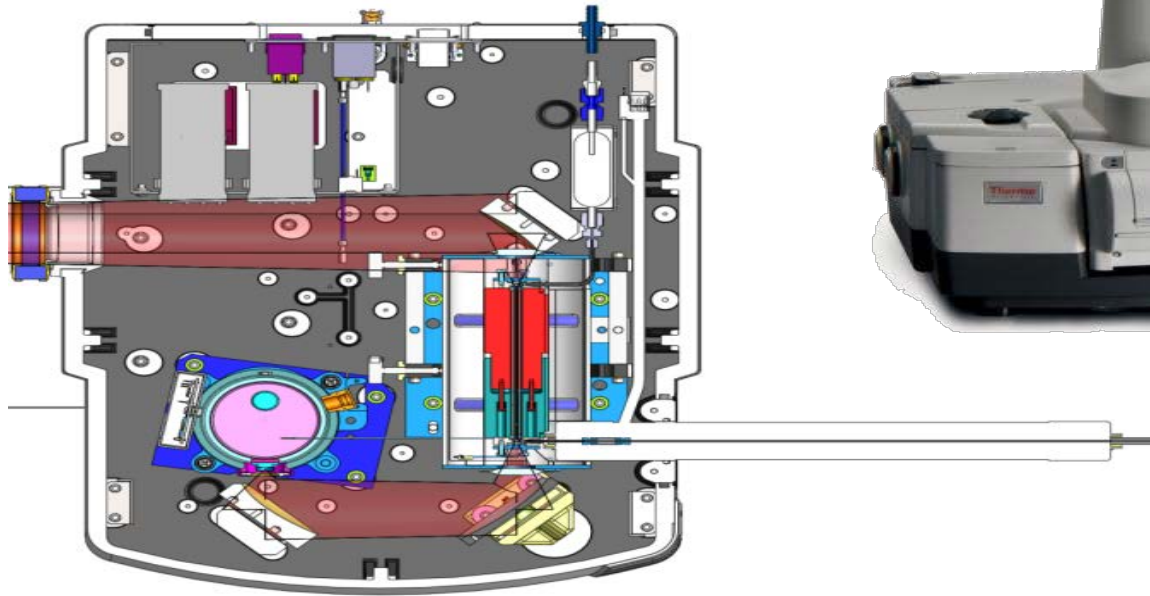


Match	Title	Cumulative	Composite%	Folder	Filename	Index
1	Ketamine .HCl	35.47	2.94	HR Georgia State Forensic Drugs	c:\my documen...	310
	Methamphetamine	68.73	58.41	Drug Search	c:\my documen...	
	Procain	80.87	7.08	Drug Search	c:\my documen...	
	Ketamine	83.02	31.58	Drug Search	c:\my documen...	



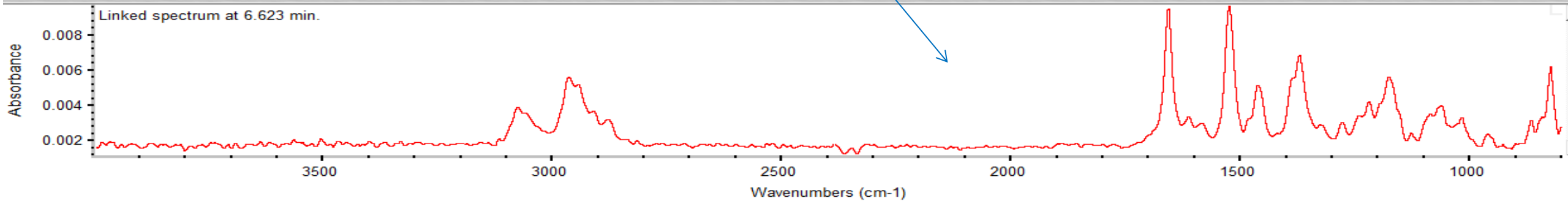
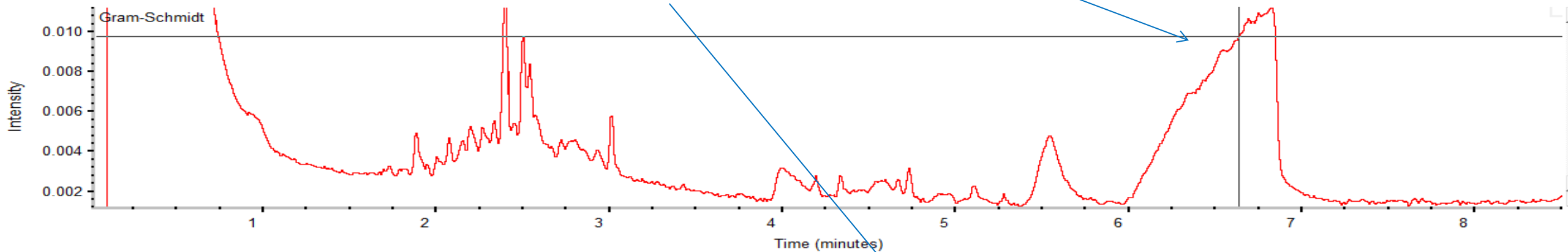


- Carbon black has high index
  - Ideal for Ge-ATR
- Multiple components
  - Base polymer
  - Silane slip-aid



- Separate and Identify
- Examine molecules while intact
- Stereo-isomers, Positional isomers

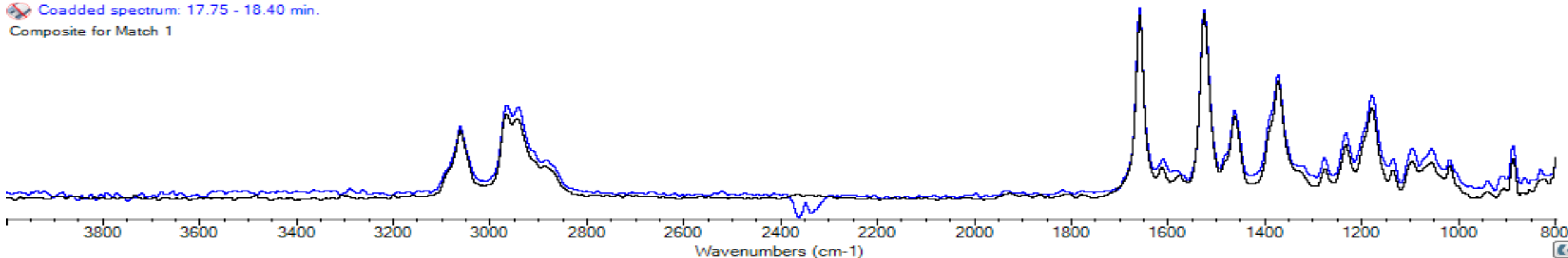
- Rapid elution: Short column
- Cannabinoid peaks co-elute
- Spectrum is a mixture



# Thermo Scientific OMNIC Spectra: Analyze Mixture

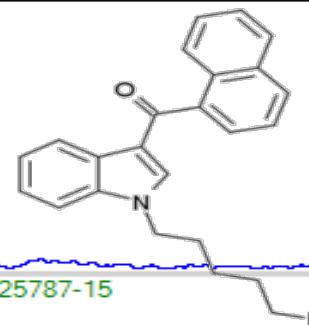
S

Coadded spectrum: 17.75 - 18.40 min.  
Composite for Match 1

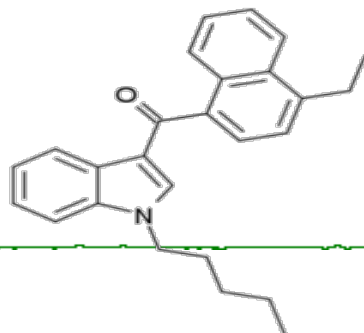


X = 2710.06 Y = -0.0006

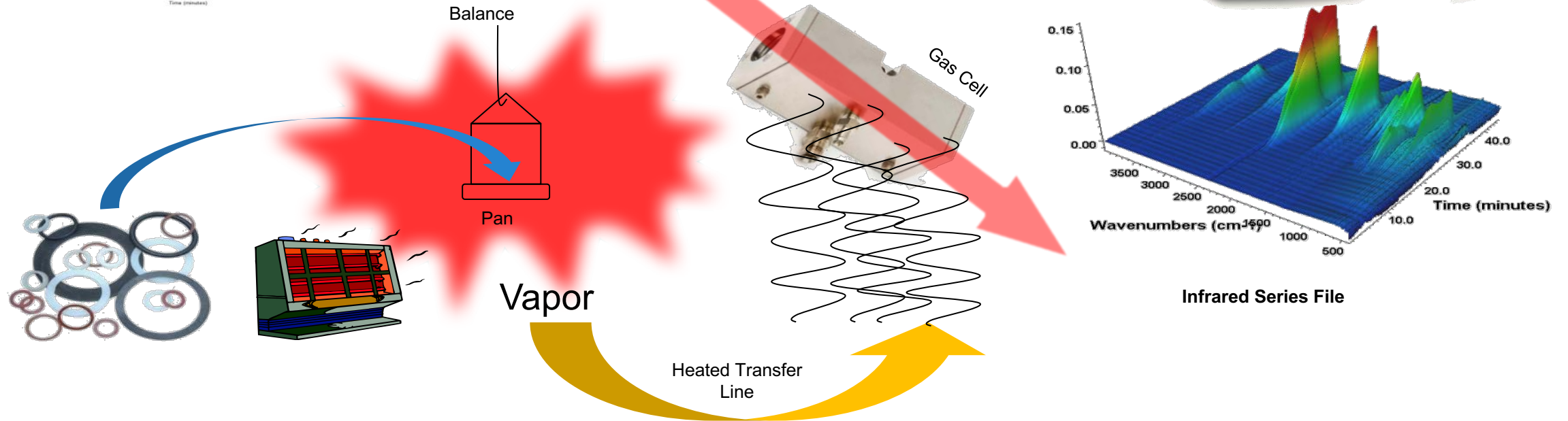
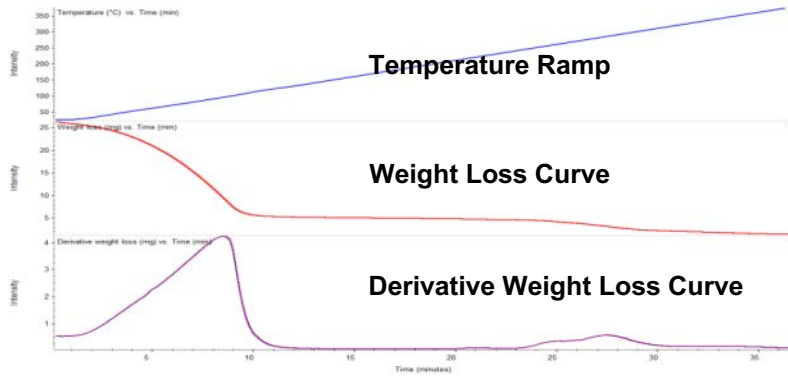
AM2201 CAYMAN CHEMICAL LOT 0427135-10 120-270



JWH 210 CAYMAN CHEMICAL CO. 120-270C MeOH RTW 5M COLUMN 5.60 MINUTES LOT 0425787-15

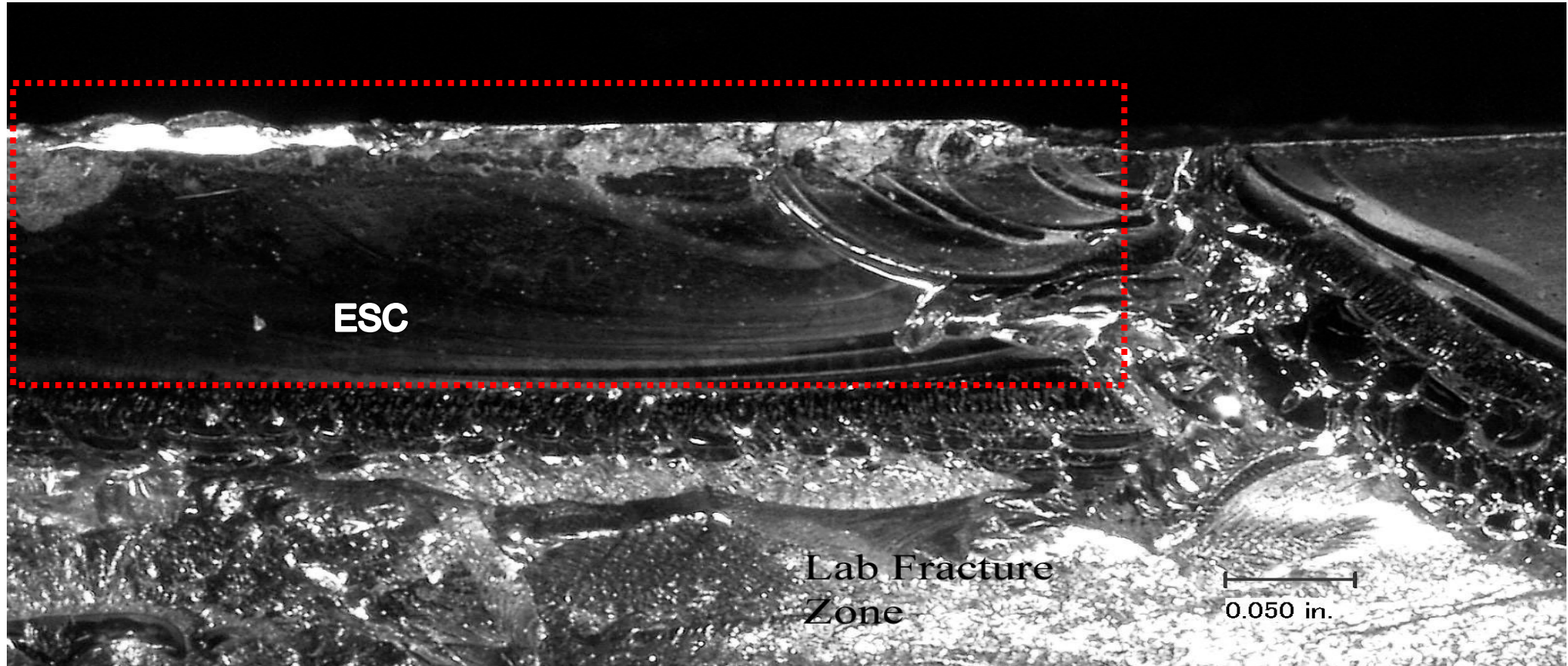


# TGA-IR: The Basics



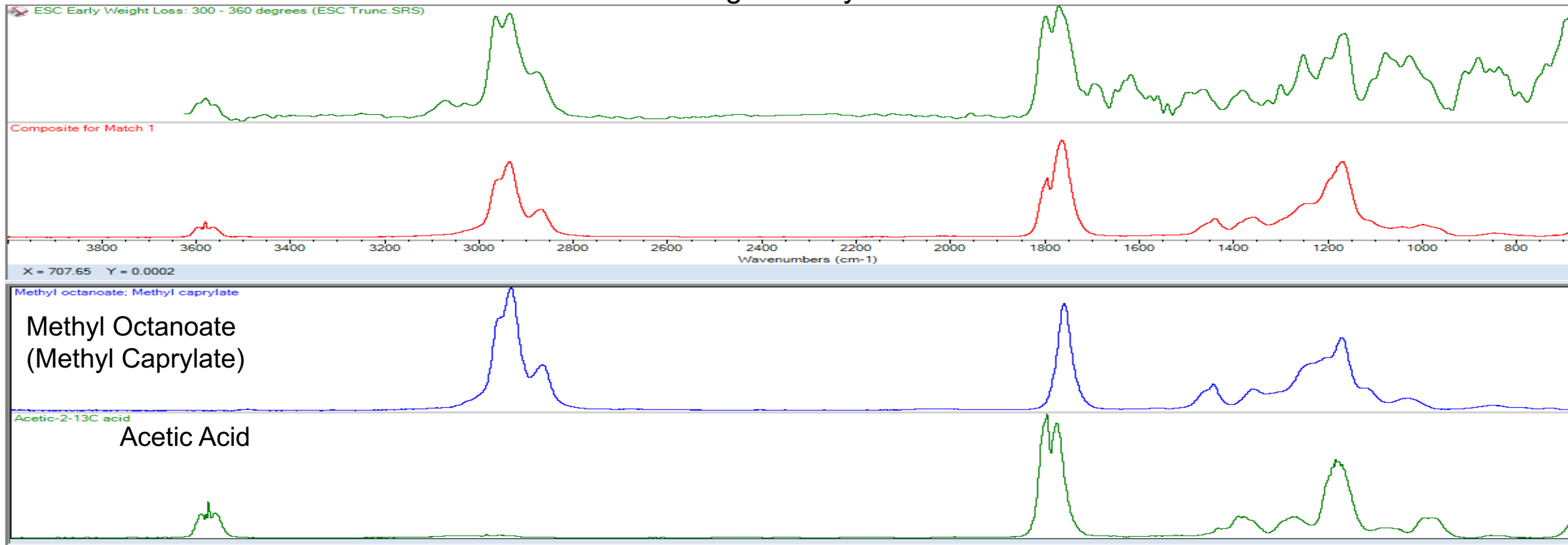
Quantitative: T "G" A

Qualitative: FT-IR



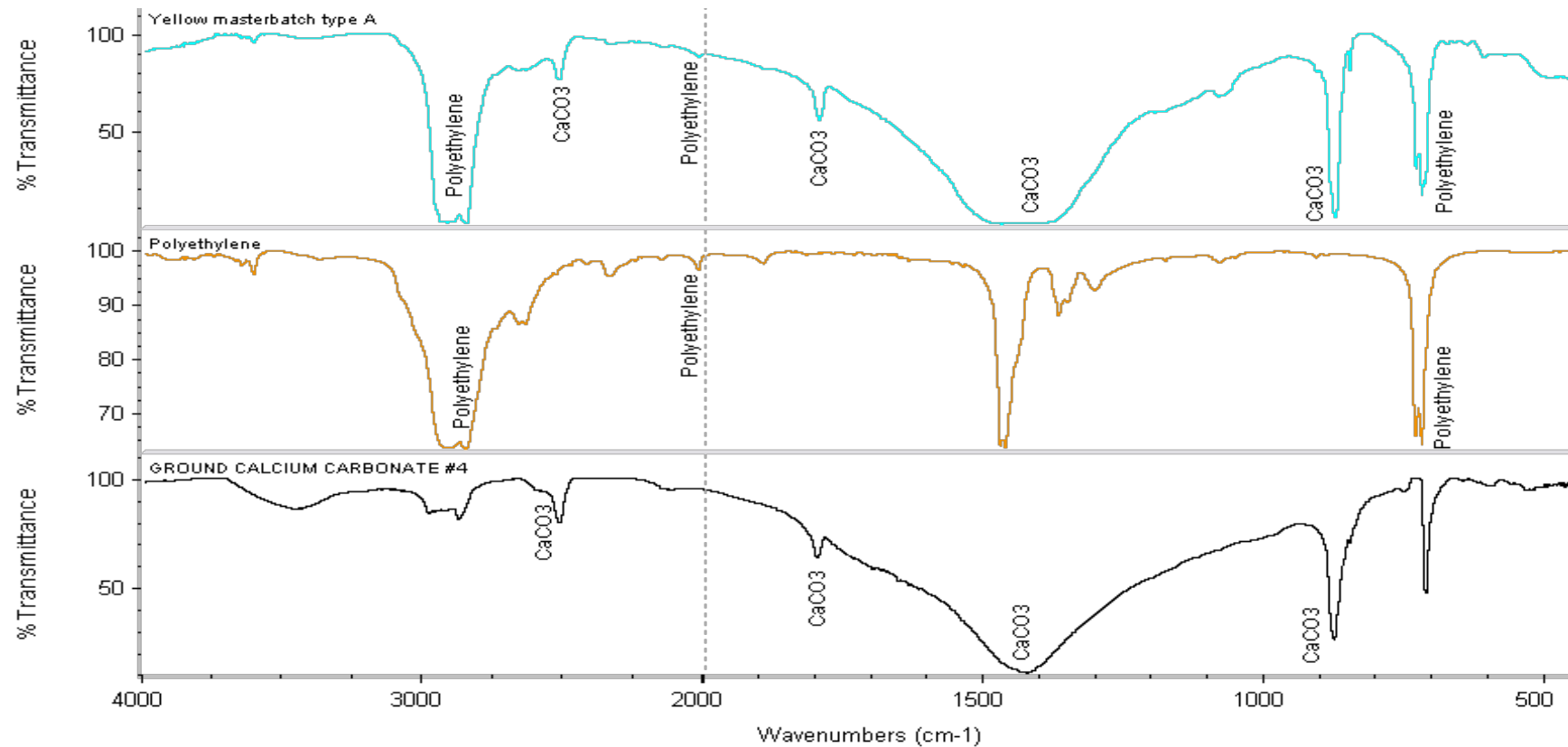
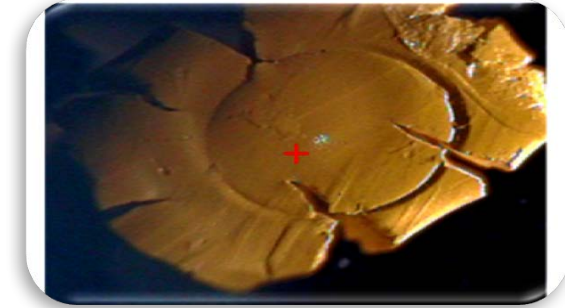
- TGA-IR Analysis:

- Early weight loss area 300 – 360 C
- Extracted spectrum subjected to OMNIC Spectra Multi-Component Search
- Identified acetic acid and medium-chain length methyl ester

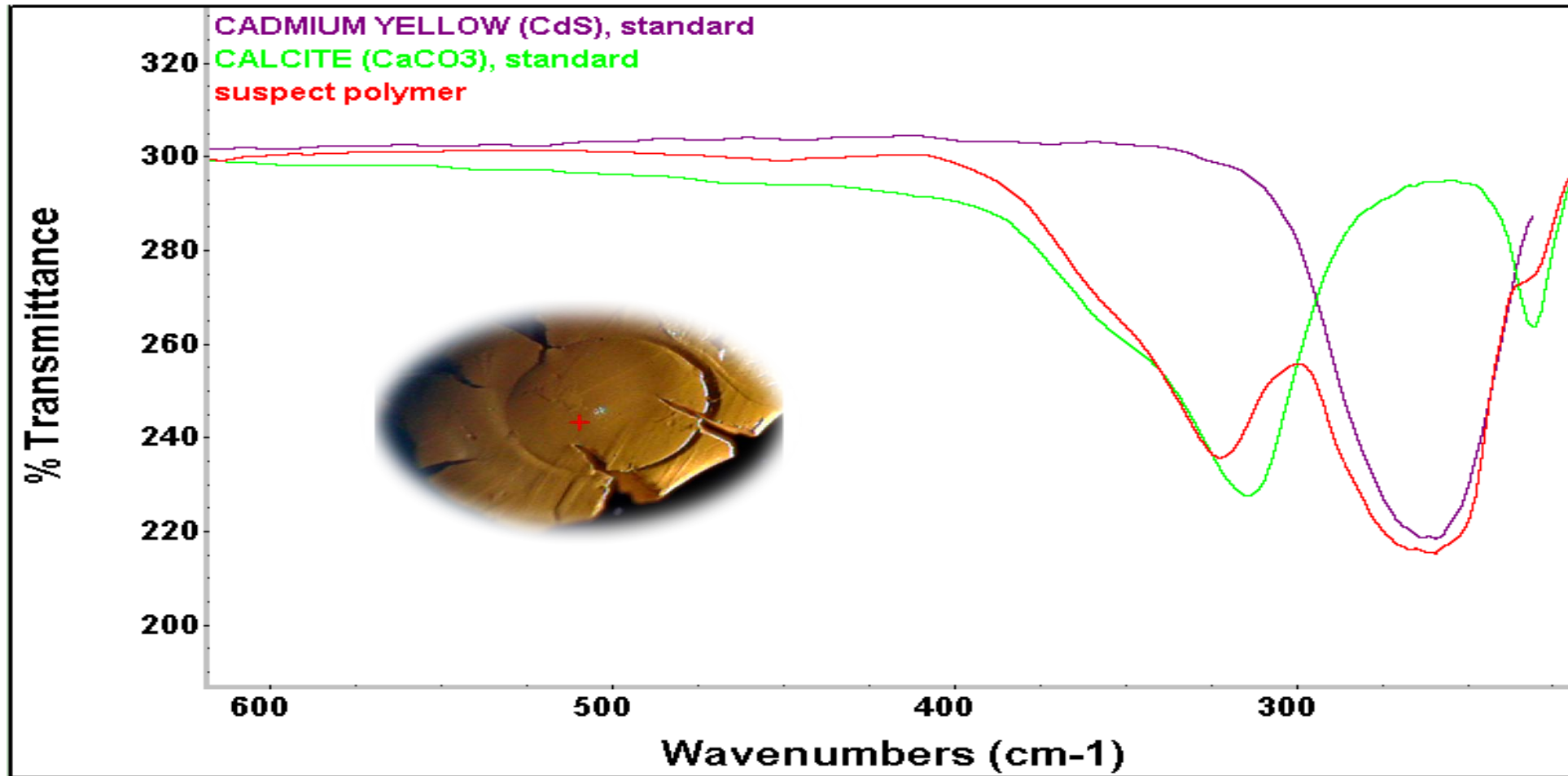


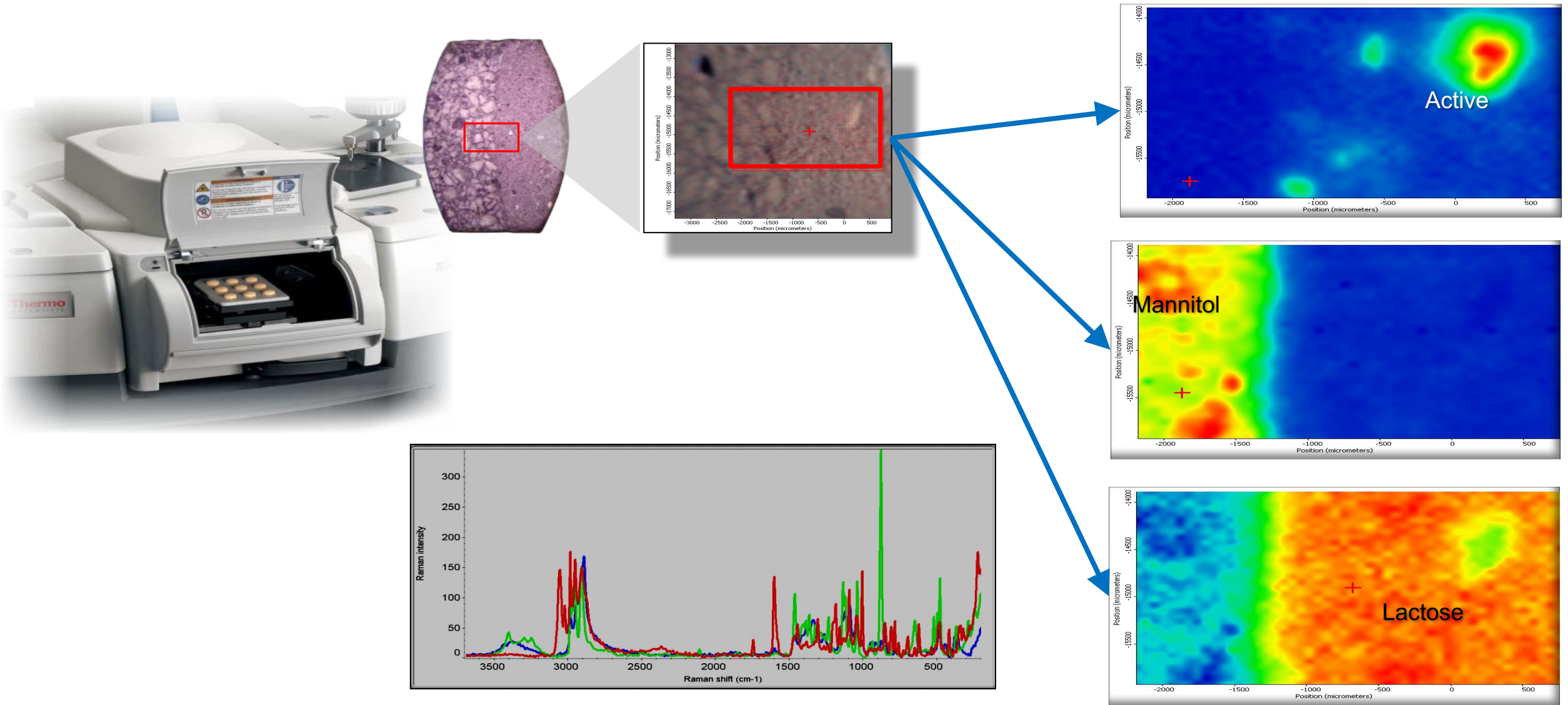


- Does this polymeric 'masterbatch' contain heavy metals?
  - Mid-IR spectrum inconclusive – no sign of cadmium compound
  - Far-IR required due to heavier molecular weight

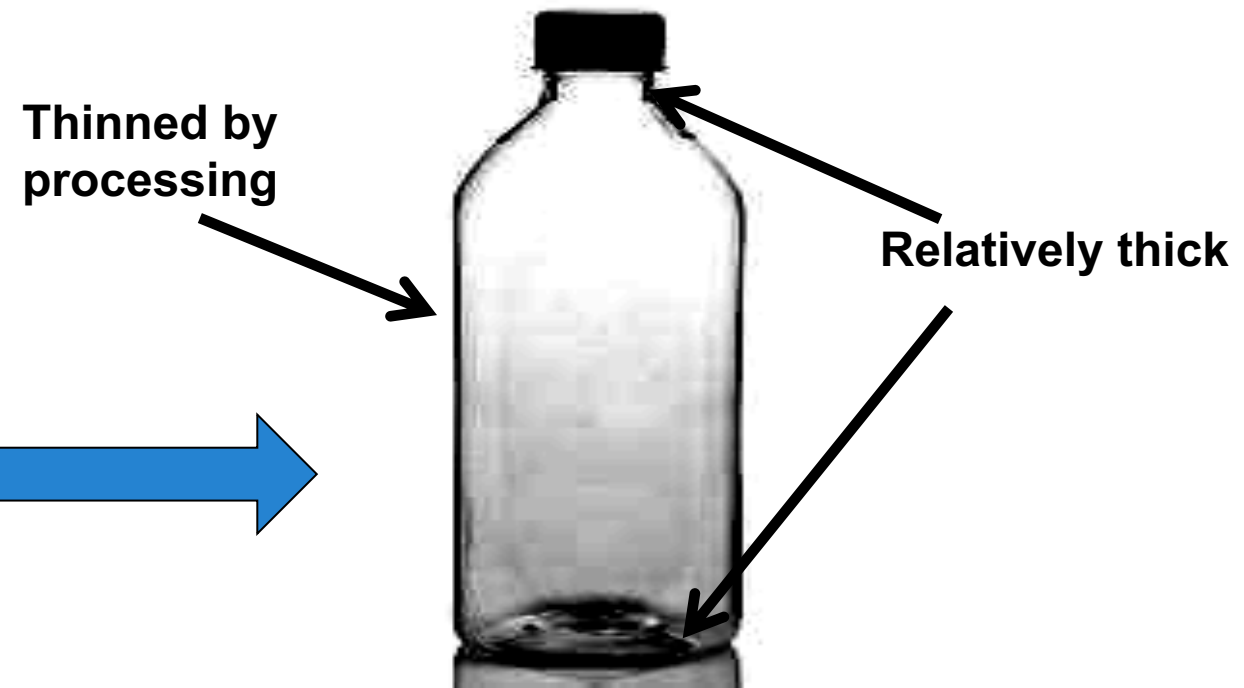
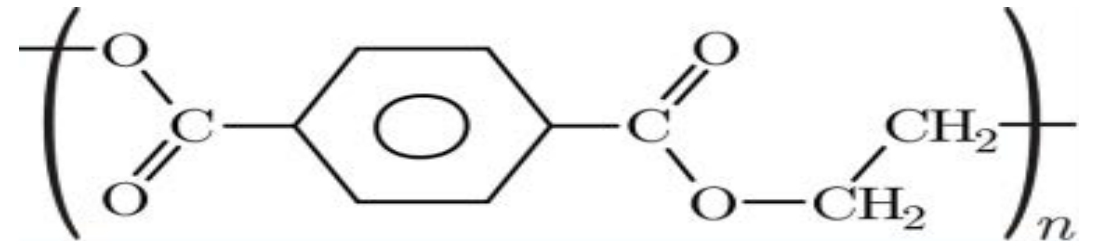


- Spectrum of polymer shows two peaks in far-IR
- One is  $\text{CaCO}_3$
- Second one is Cadmium Yellow ( $\text{CdS}$ )

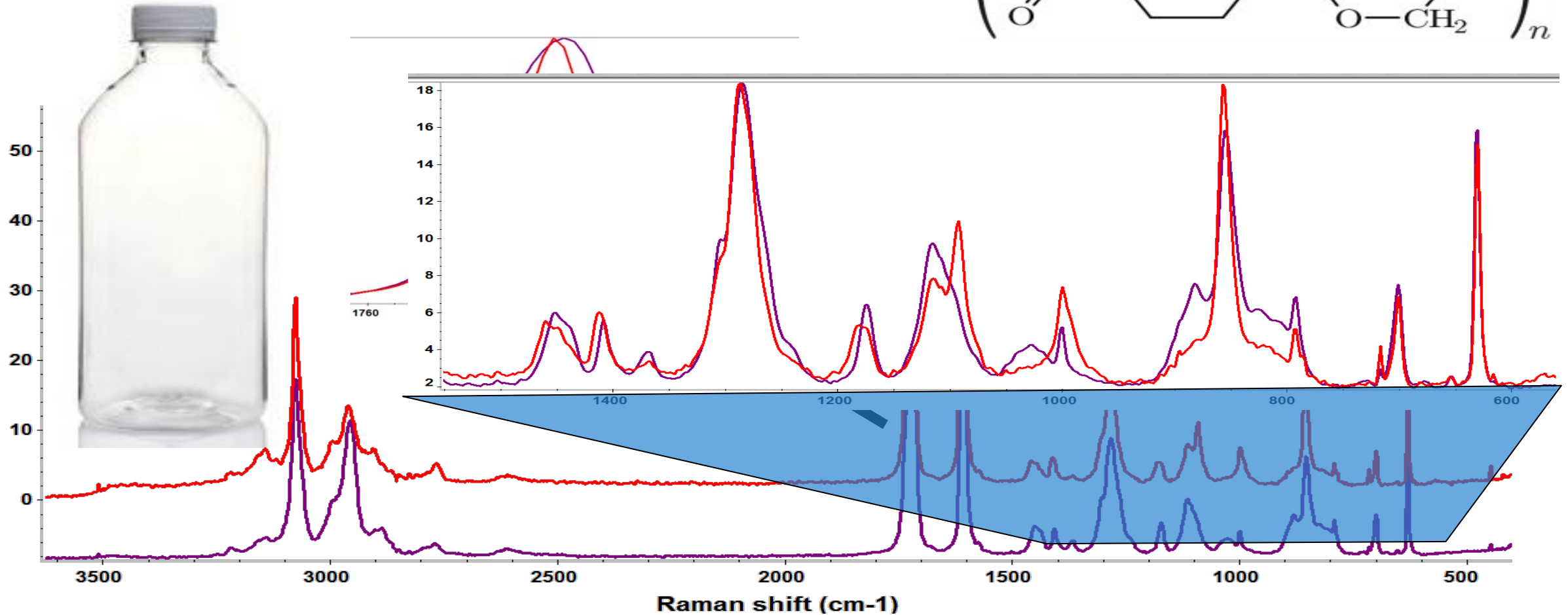
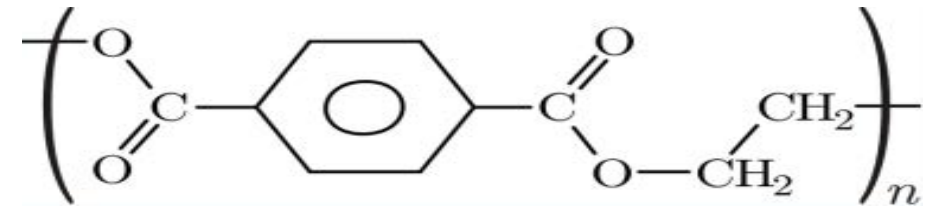




- Polyethylene terephthalate (PET) used in making bottles
- Thick walled blanks are heated and blown into a mold
- The PET molecules in different regions of the bottle are in different environments



**Bottle side: Linearly aligned**  
**Bottle top: Amorphous**



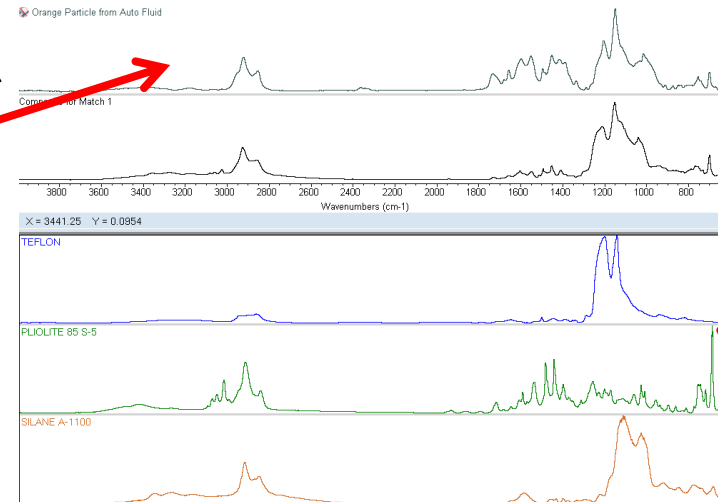
- Thermo Scientific™ Nicolet™ iN™5 FT-IR Microscope
  - Manual Control
  - Excellent Visuals
    - Camera and Eyepieces
  - Fast to Learn, Simple to use



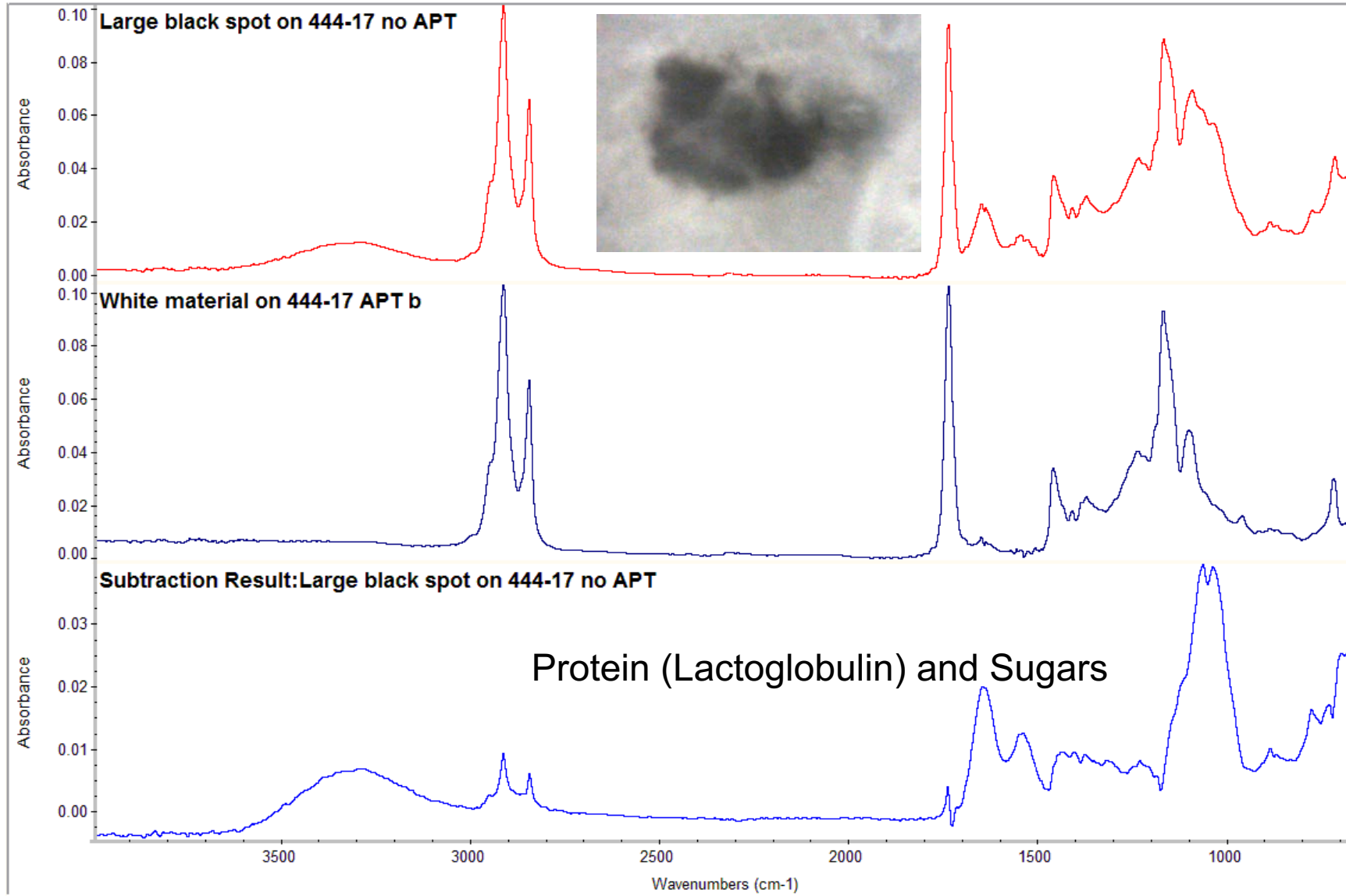
Locate



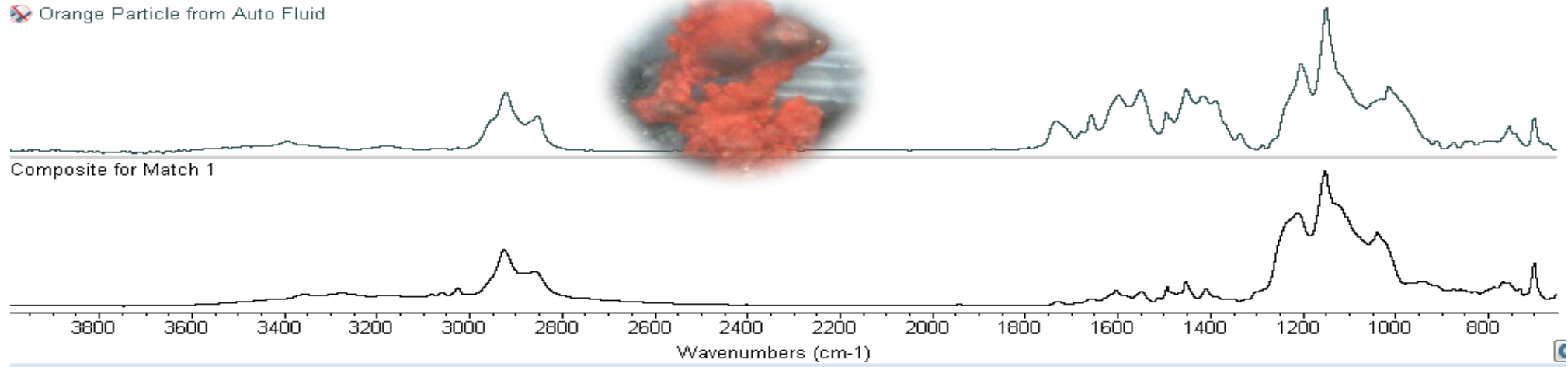
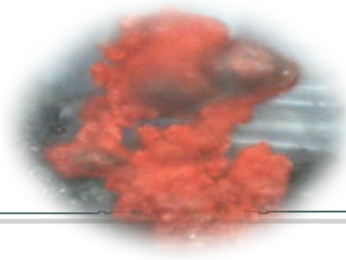
Collect



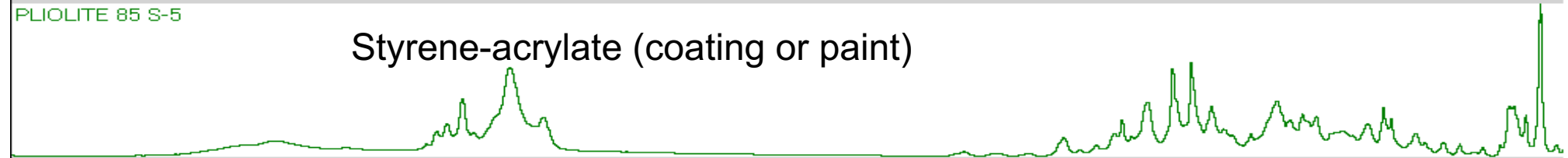
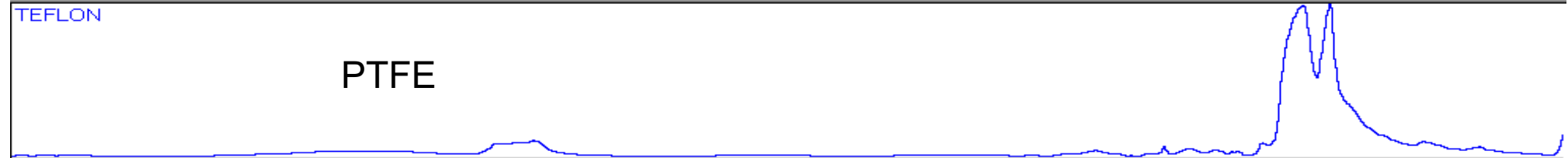
Identify



Orange Particle from Auto Fluid



X = 3441.25 Y = 0.0954

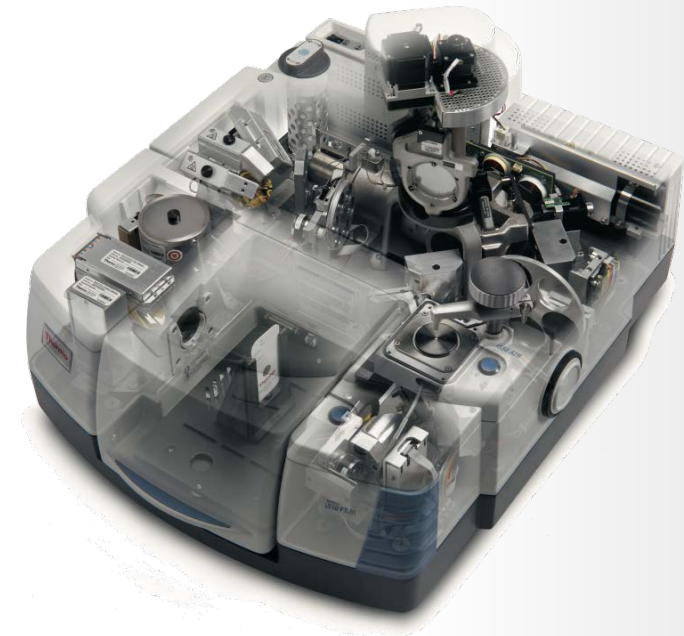




- Discover. Solve. Assure.
  - Research to Routine:
    - Advanced FT-IR with Touch Point Simplicity
    - Multimodal Analysis
    - Flexibility in the box



- Applications
  - Particles to Bulk
  - Far-IR to Visible
  - Multi-component Analysis





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**f** fisher scientific

**unity** lab services

Please join me in the  
**Materials Identification**  
section of our booth where I'll  
address comments and questions.