Meaningful Particle Analysis Begins with Light Microscopy

Bill Chapin
Sr. Research Microscopist

Webinar Overview

RE-INTRODUCTION OF LIGHT MICROSCOPY

A Time-proven, but often Forgotten, Analytical Technique that Augments Other Instrumental Analyses

- Equipment and Tools
  - Stereomicroscopy
  - Polarized Light Microscopy
  - Illumination Techniques for Maximized Effectiveness
  - Sample Handling Tools
- How it Helps
  - Discovery of Valuable Information that often Leads to Improved Sample Identifications
  - Provides a Means of More Effective Micro-sample Preparation for Other Instrumental Techniques

Stereomicroscope

Zoom or Click Magnification from ~10X to ~135X

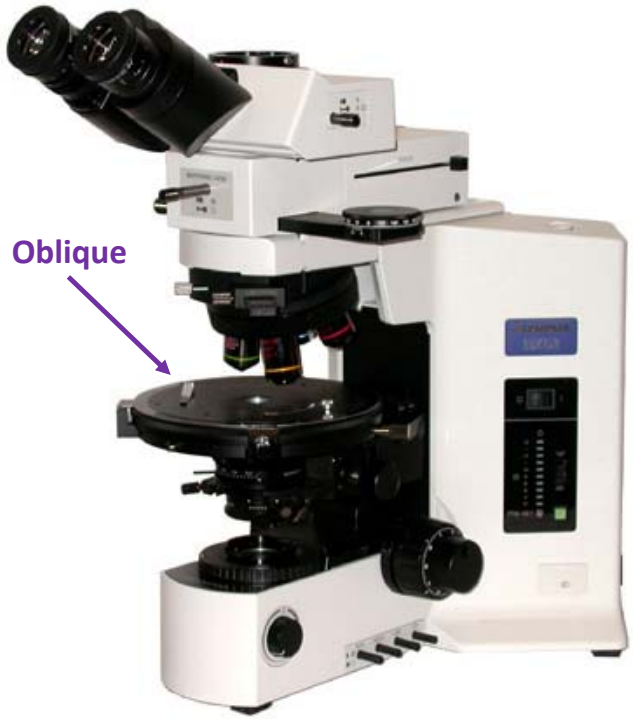
Polarized Light Microscope

"Set" Magnifications from 40X to 500X (1000X with Oil)

Common Illumination Techniques

Oblique –typical illumination for stereomicroscopy

Stereomicroscope



Polarized Light Microscope

Common Illumination Techniques

Transmission – typical illumination for PLM

Transmitted

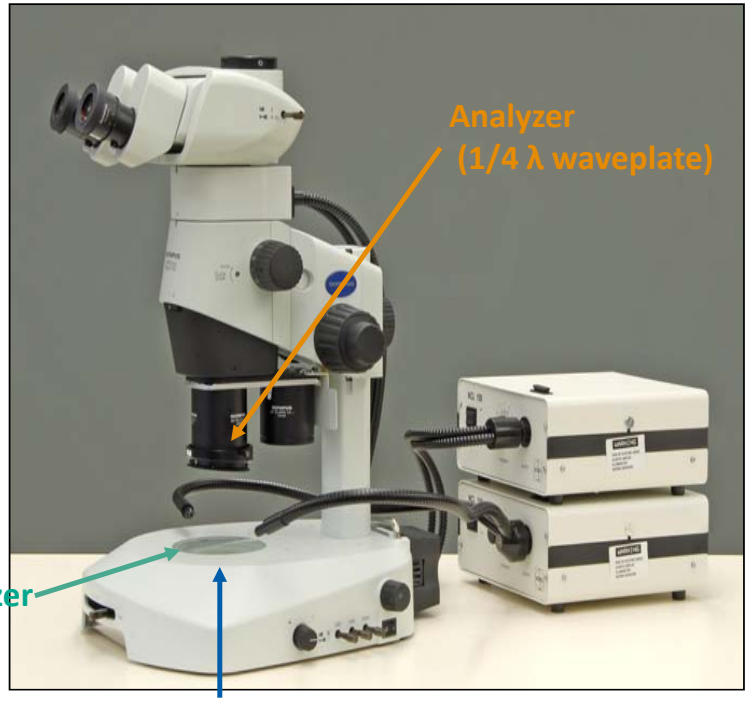
Stereomicroscope

Transmitted

Polarized Light Microscope

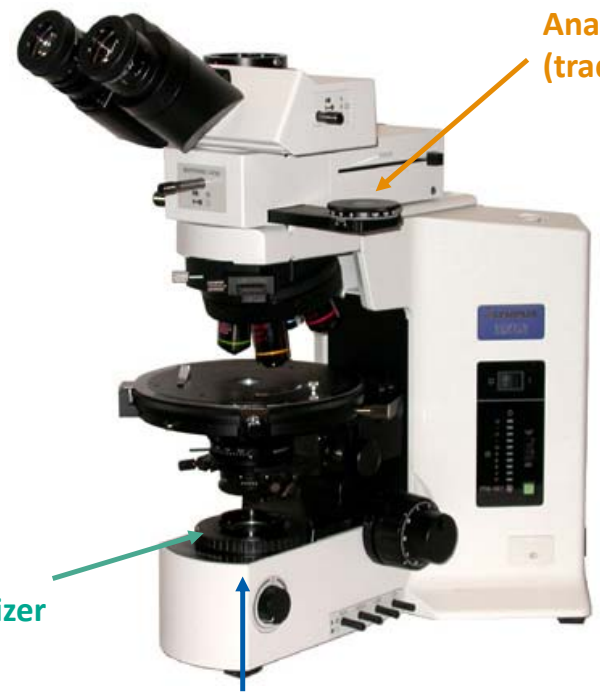
Common Transmission Illumination

Partially Crossed Polarizer



Stereomicroscope

Fully Crossed Polarizer



Polarized Light Microscope

Common Illumination Techniques

Reflection – powerful use on either Stereomicroscope or PLM

Coaxial

Coaxial Illumination on Stereomicroscope

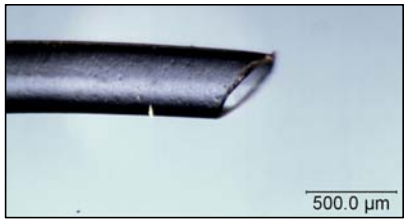
Episcopic

Episcopic Illumination on Polarized Light Microscope

Necessary Micro-Tools

Tungsten Needles
of
Various sizes

Micro-pipette
< 1 µl delivery

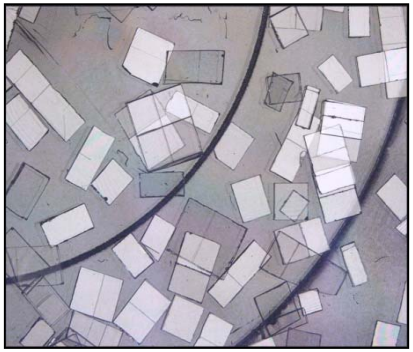


Tungsten-Carbide Scribe

Microscalpel

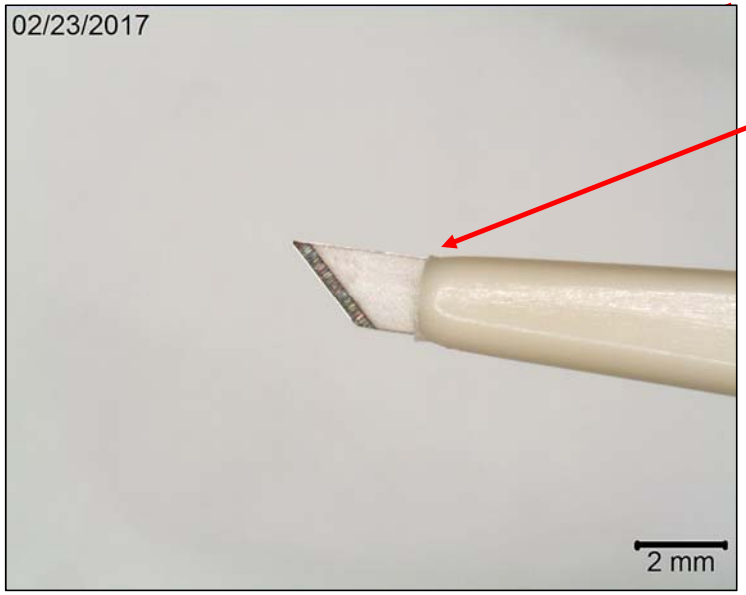
Tungsten Needles

Sharp Tweezers

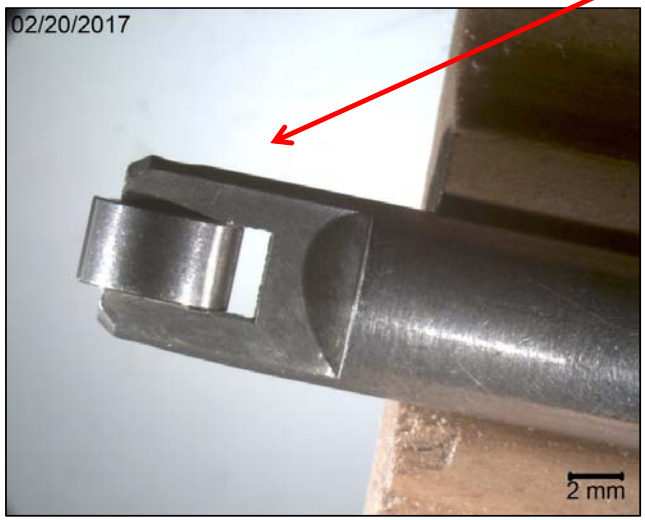


1-4 mm glass slides
Larger 5-7 mm are also useful
Made from cover slips

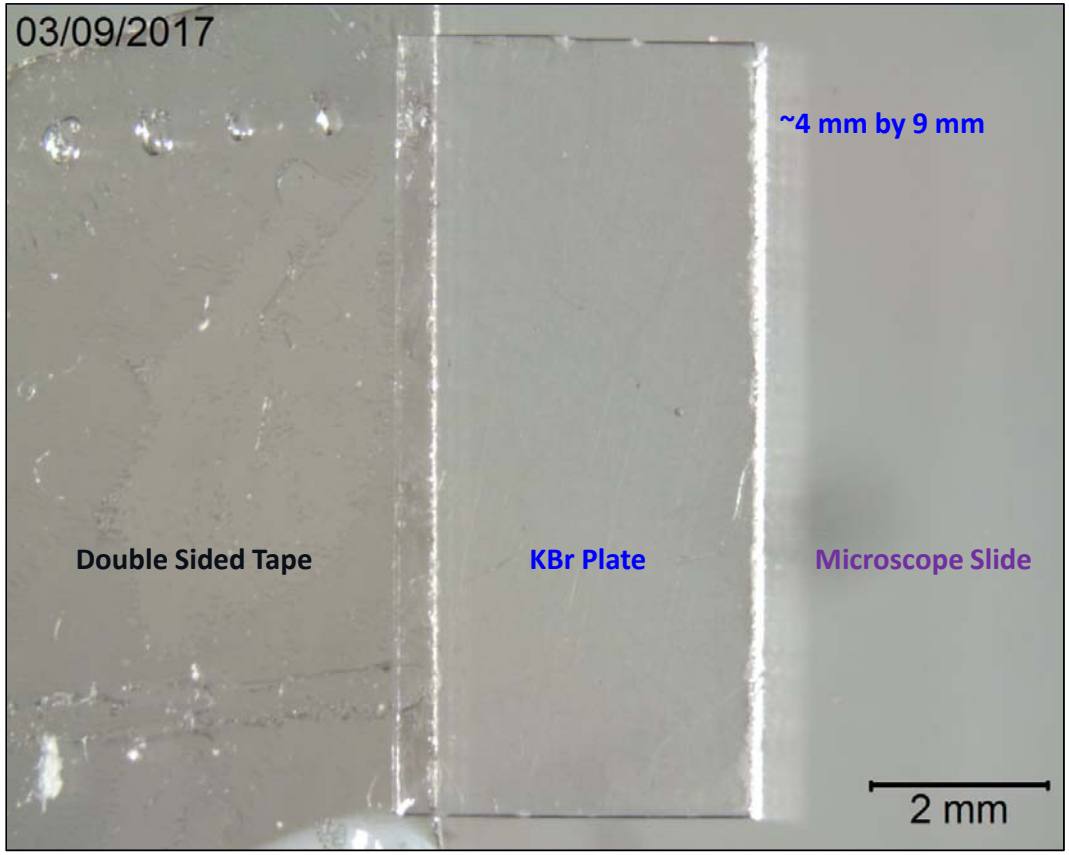
Micro-scalpel



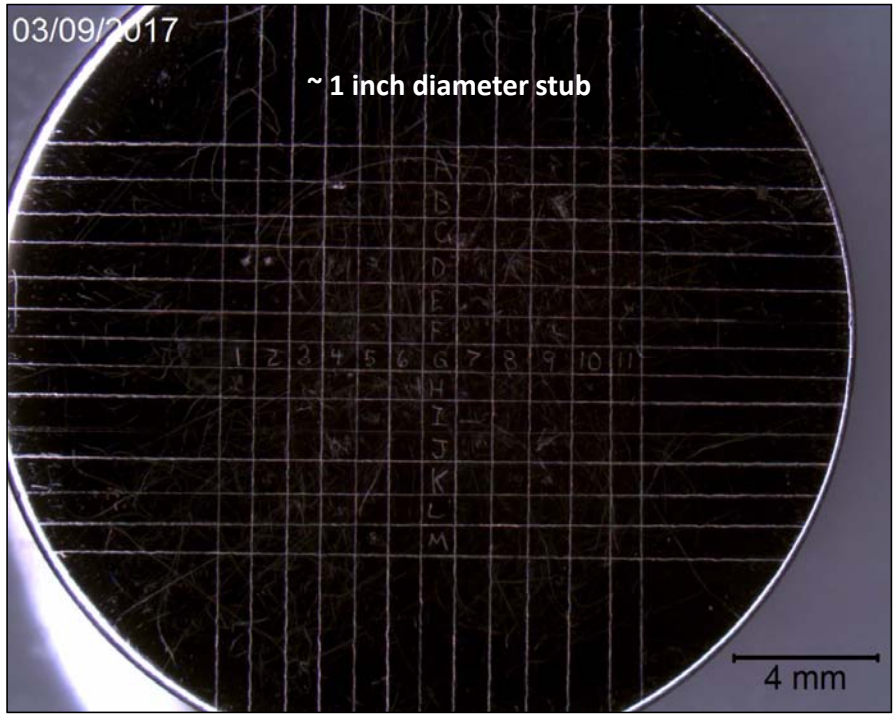
Roller Tool



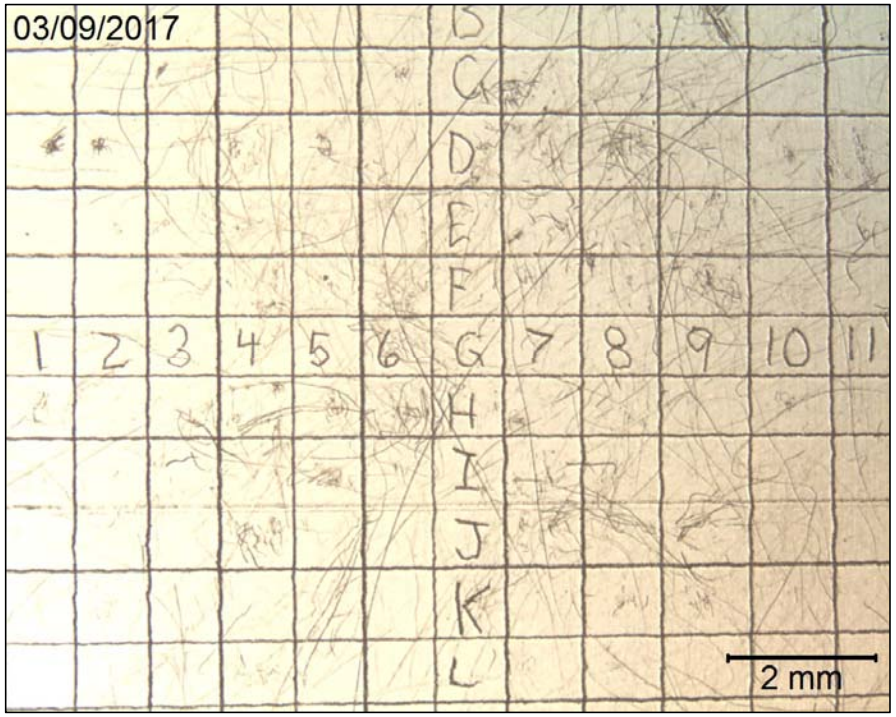
Potassium Bromide Plates for Transmission Micro-FTIR



Permanently Scribed Beryllium Stub for SEM-EDS



Ring Light Illumination at 5.25X Magnification

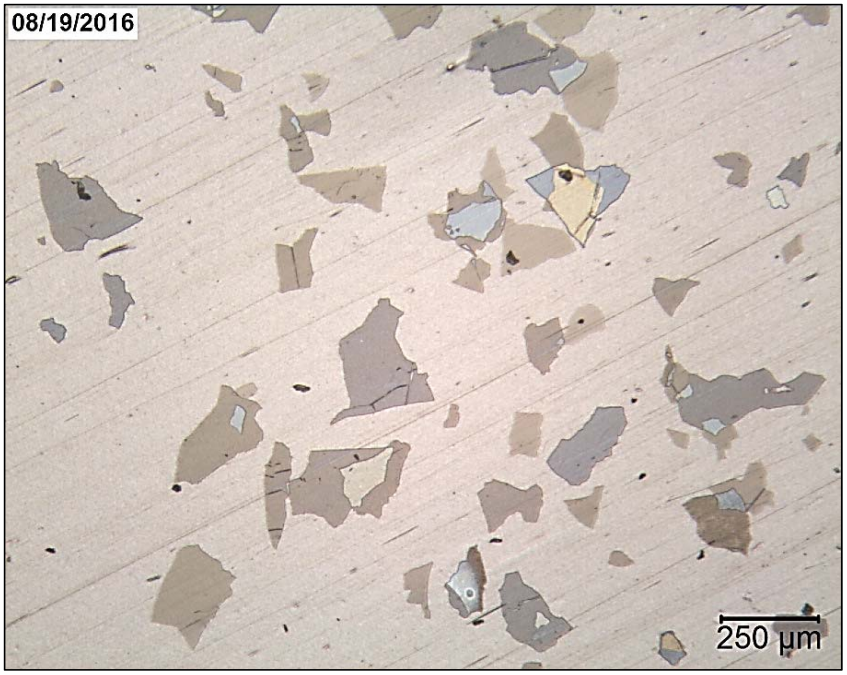


Coaxial Illumination at 10.5X Magnification

What is So Special about Reflection Techniques

Glass Delamination Flakes on Filter - Same Field-of-View for Both Images

Oblique Illumination,
Stereomicroscope, 60X Magnification
Glass Delamination on Filter

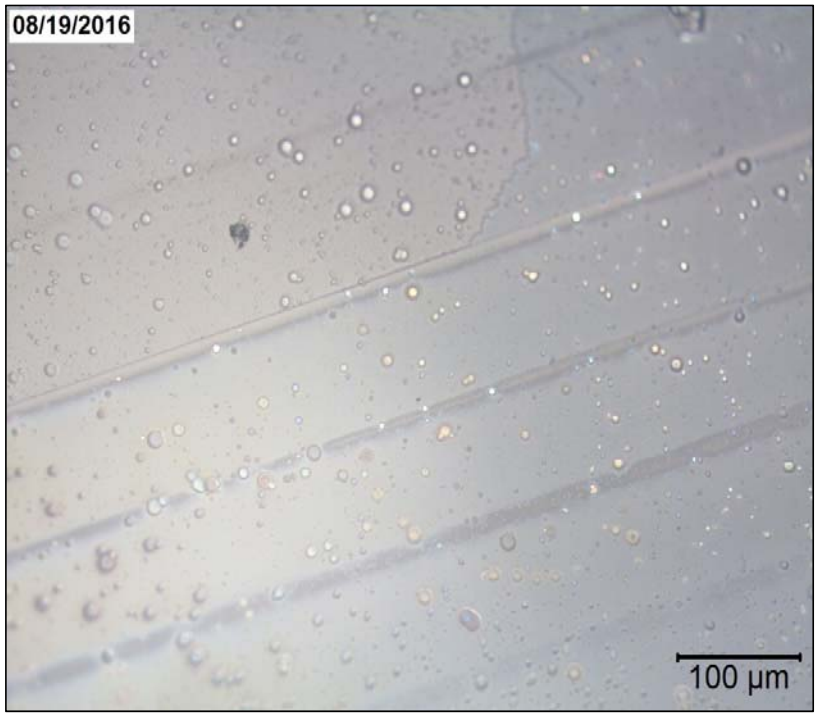


Coaxial Illumination,
Stereomicroscope, 60X Magnification
Glass Delamination on Filter

What is So Special about Reflection Techniques

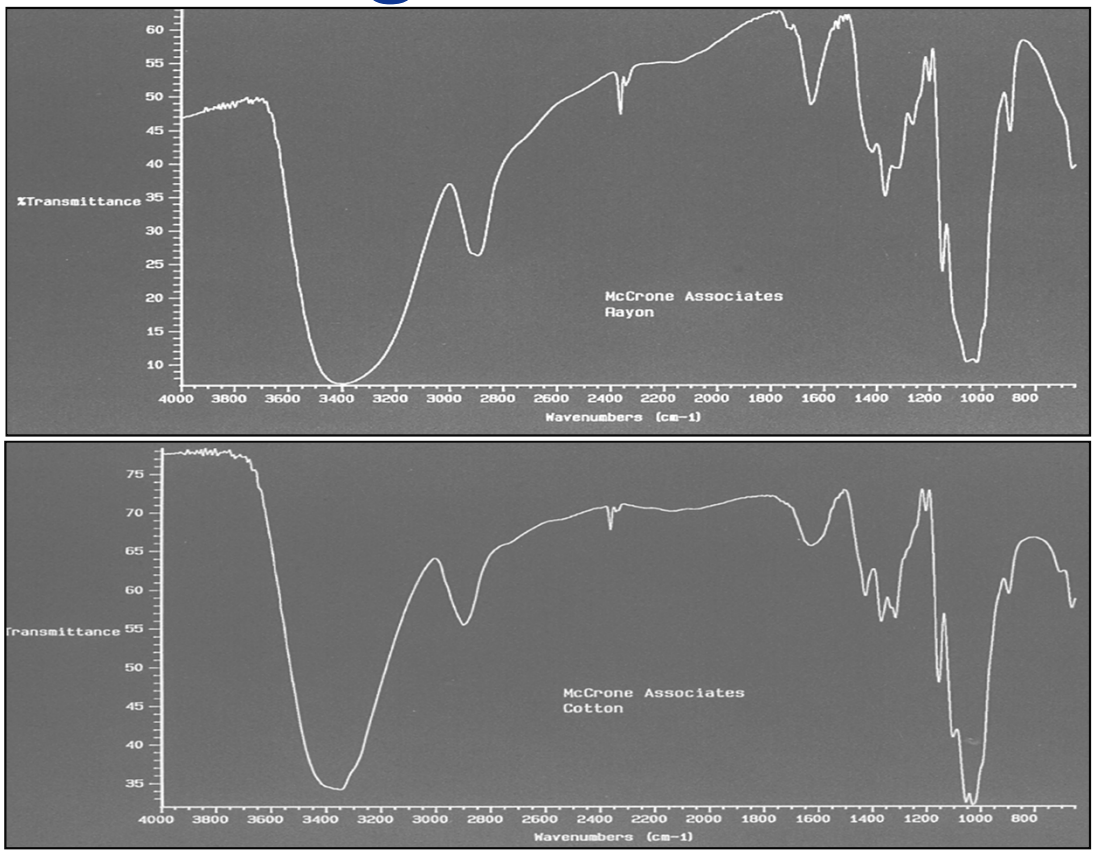
Glass Damage of Inner Vial Wall - Same Field-of-View for Both Images

Oblique Illumination,
Polarized Light Microscope, 200X Magnification
Inner Wall of Vial



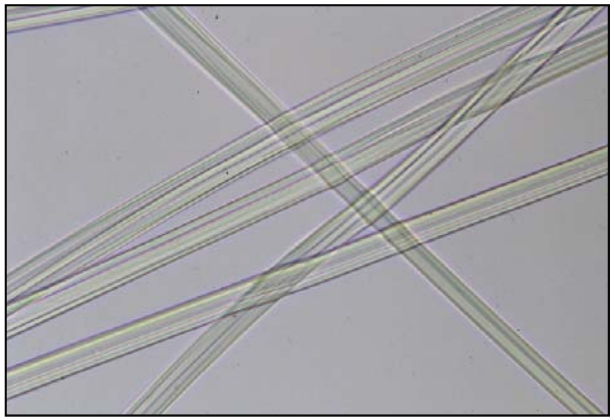
Episcopic Illumination,
Polarized Light Microscope, 200X Magnification
Inner Wall of Vial

Microscopy May Provide Information that Augments Infrared Spectroscopy



FTIR Spectra – Carbohydrates (Cellulose)

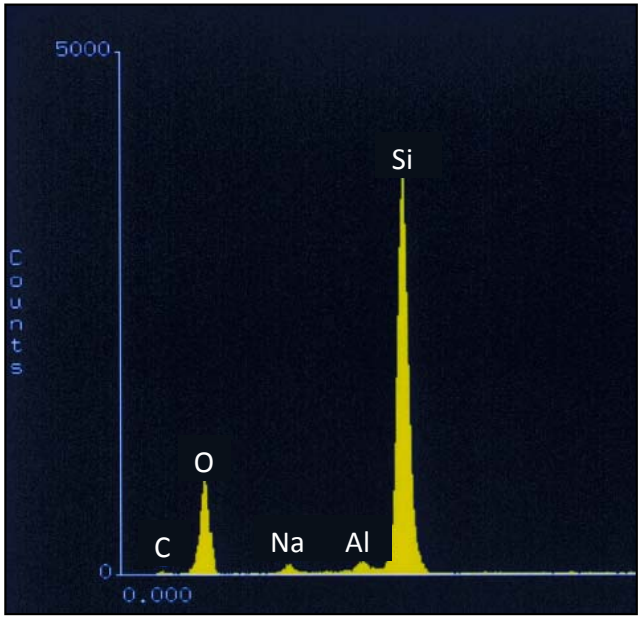
Rayon

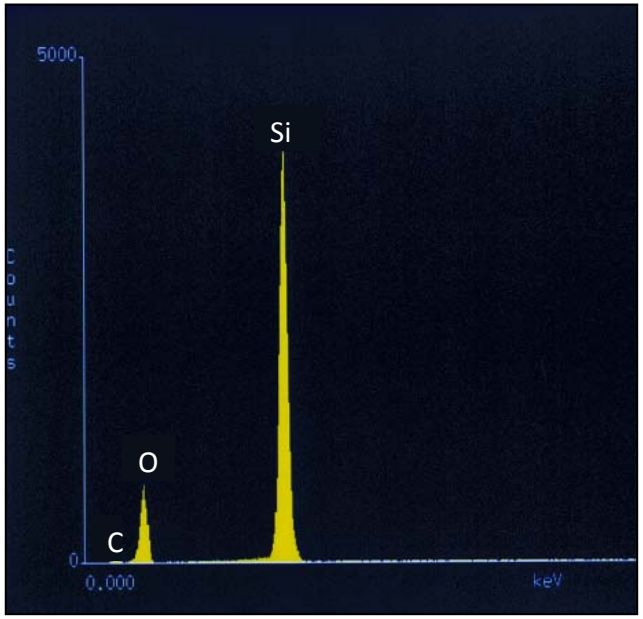


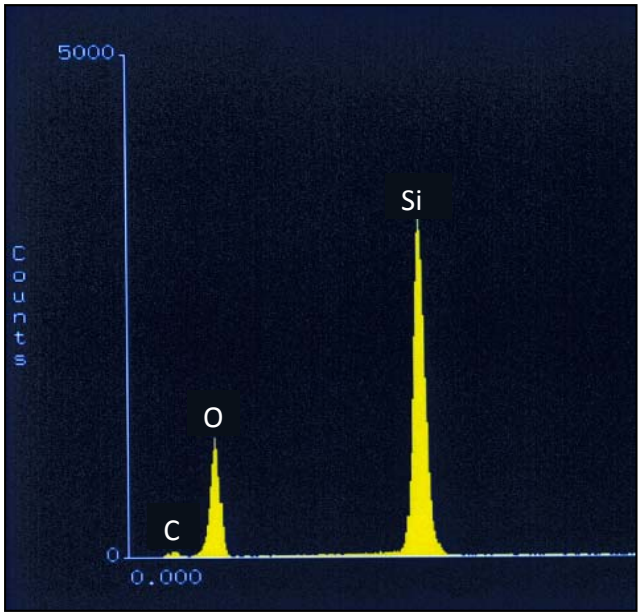
Cotton

Polarized Light Microscopy

Is All SiO_x from the Same Source ?

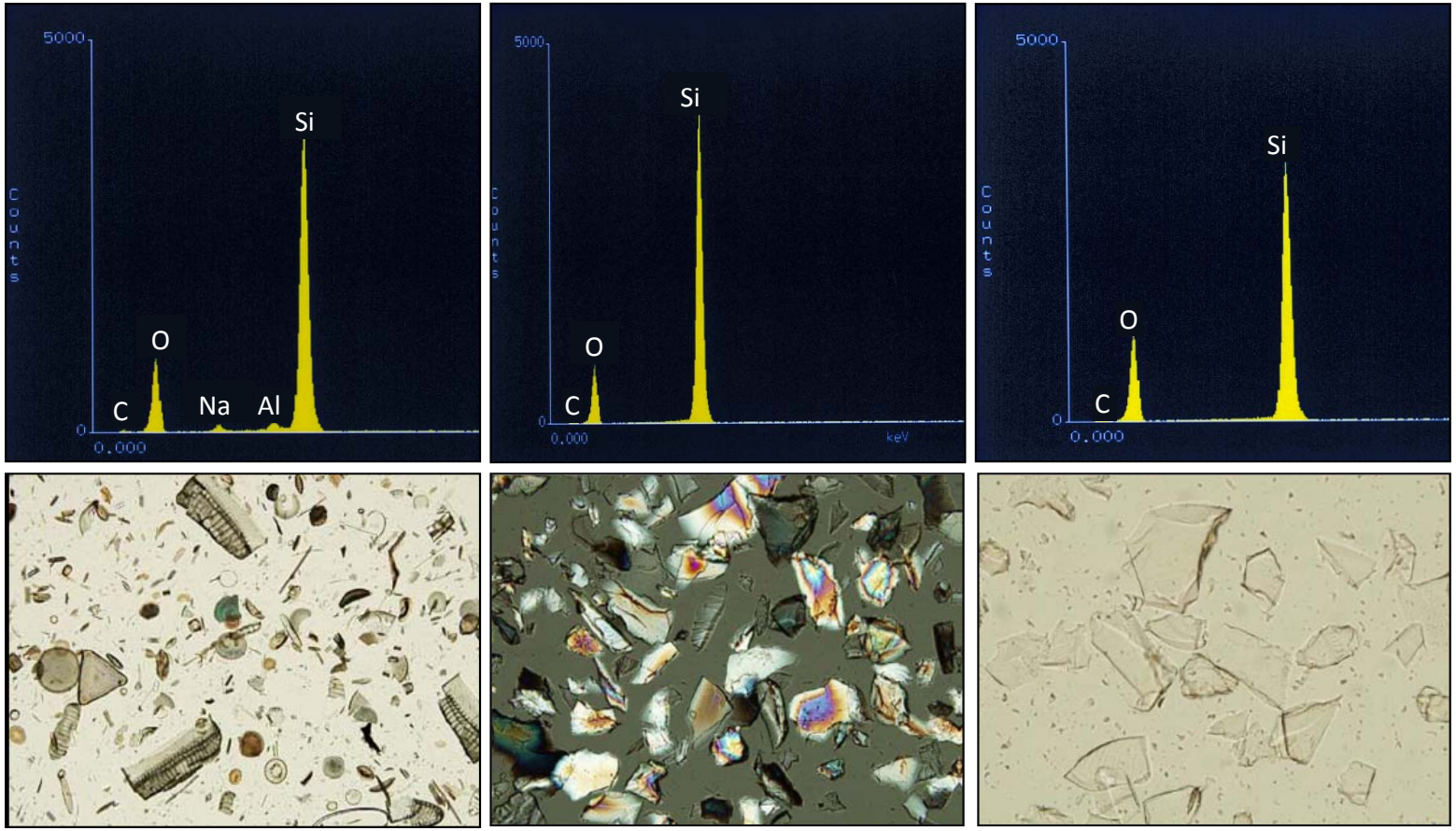






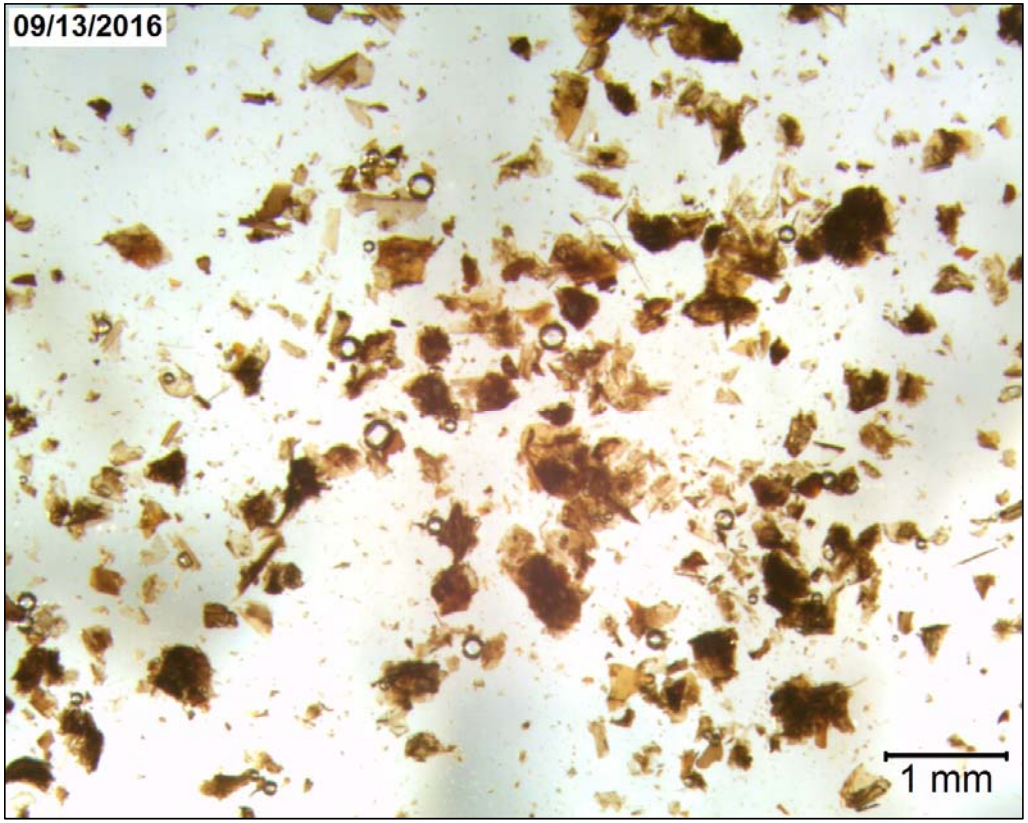
Elemental Analysis – JEOL, JSM-6480LV with Oxford AZtec EDS

Of Course it Isn't



DIATOMS **QUARTZ** **SILICA GEL**

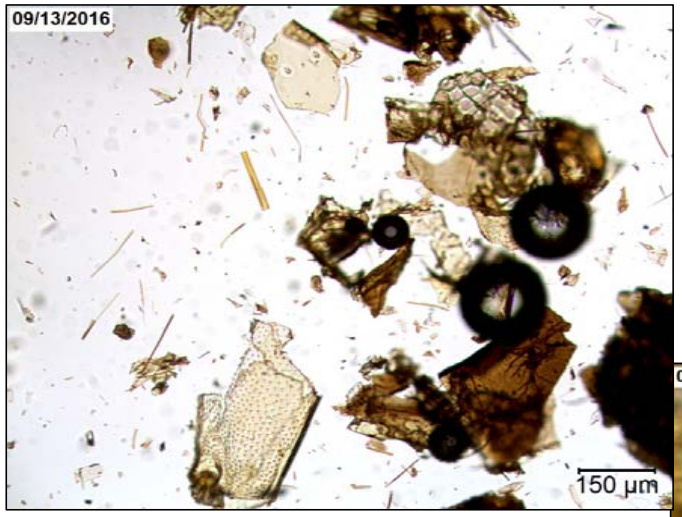
Use of the Correct Microscope May Lead to Sample Identification



Stereomicroscope – 15X

Insufficient Magnification for Sample Identification

Use of the Correct Microscope May Lead to Sample Identification



Polarized Light Microscope – 100X

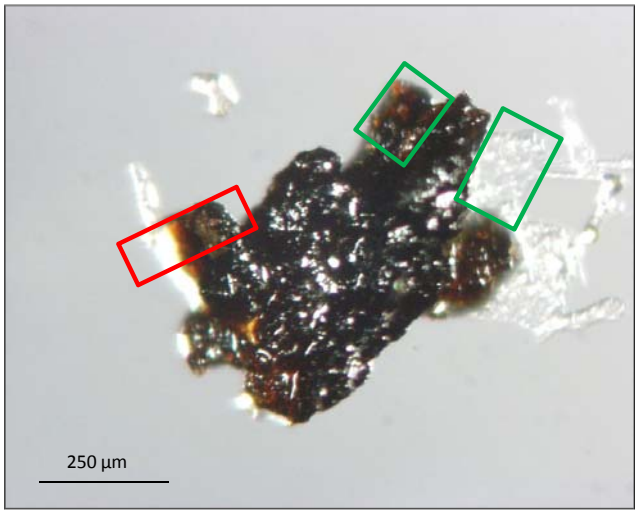
More Appropriate
Microscopic Magnification

(Same Sample Preparation)

Polarized Light Microscope – 400X

Polarized Light Microscope – 400X

Deposition and Sampling Information

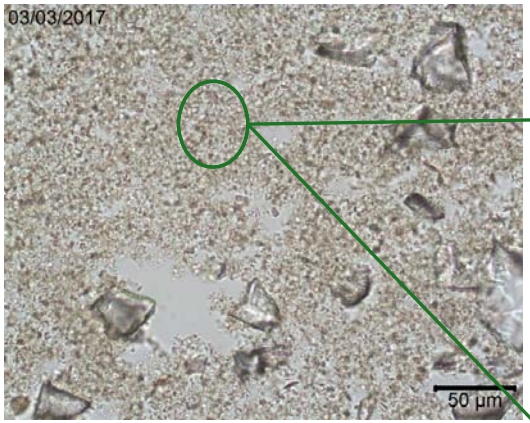


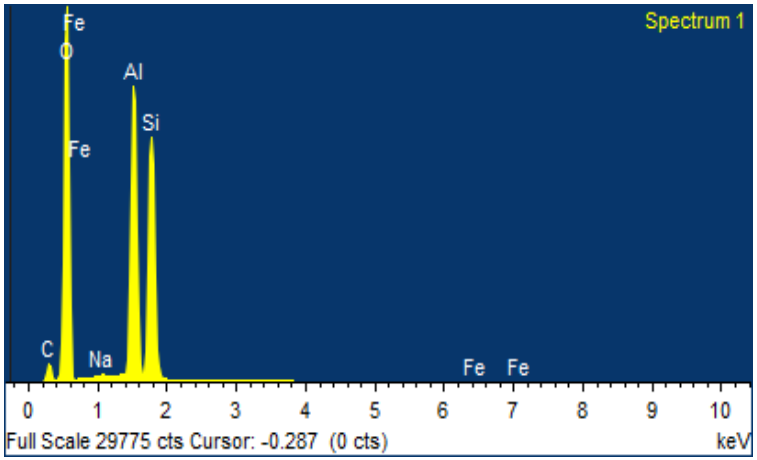
Shows degraded and non-degraded locations. Indicates a heat "zone". Should produce an identifiable infrared spectrum.

Indicates a spattered drop of organic material that has "boiled". Infrared data may only indicated "degraded organic material".

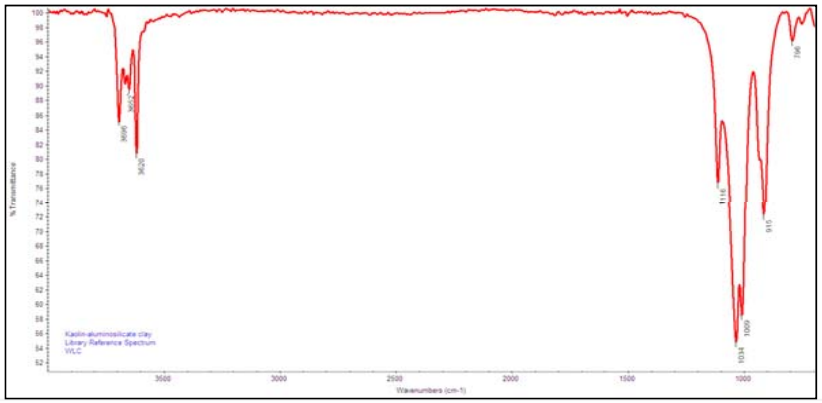
Creation of a Homogeneous Sample from Heterogeneous Material

Sample in refractive index liquid under a cover slip.

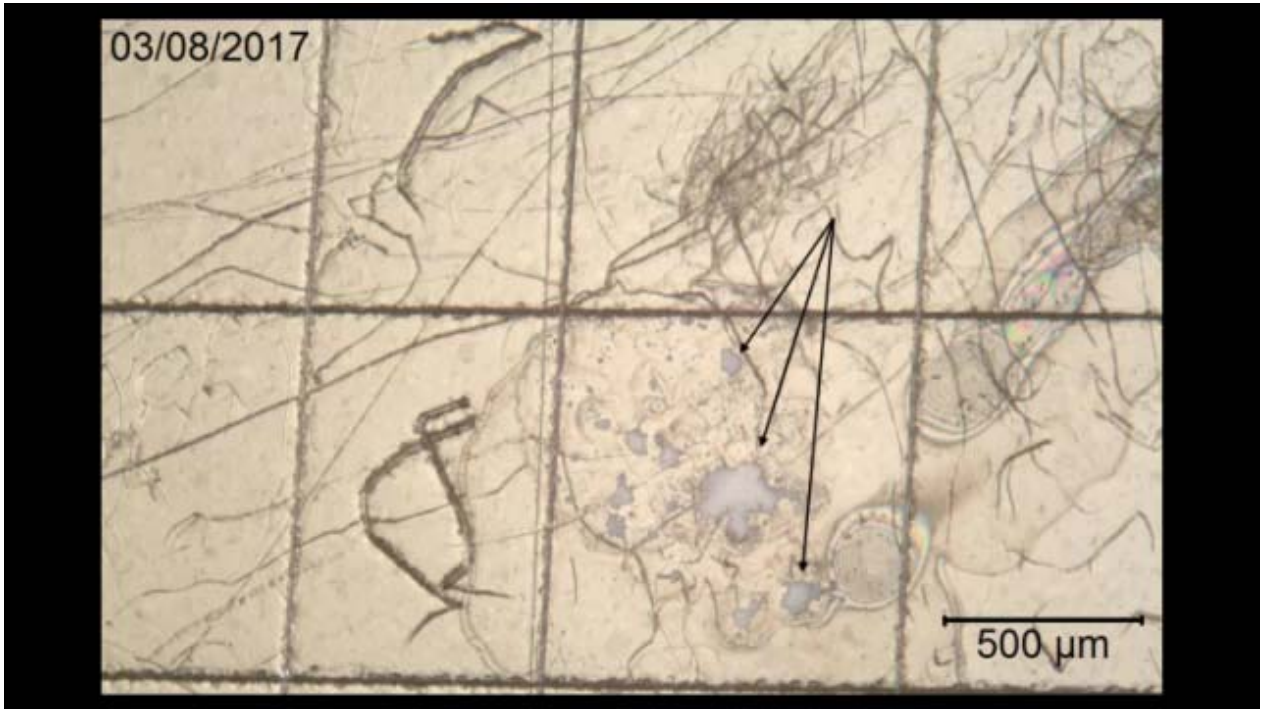




1. Slide the cover slip aside
2. Use tungsten needle to isolate the target sample
3. Use amyl acetate to wash away RI liquid
4. Mount the sample for analysis

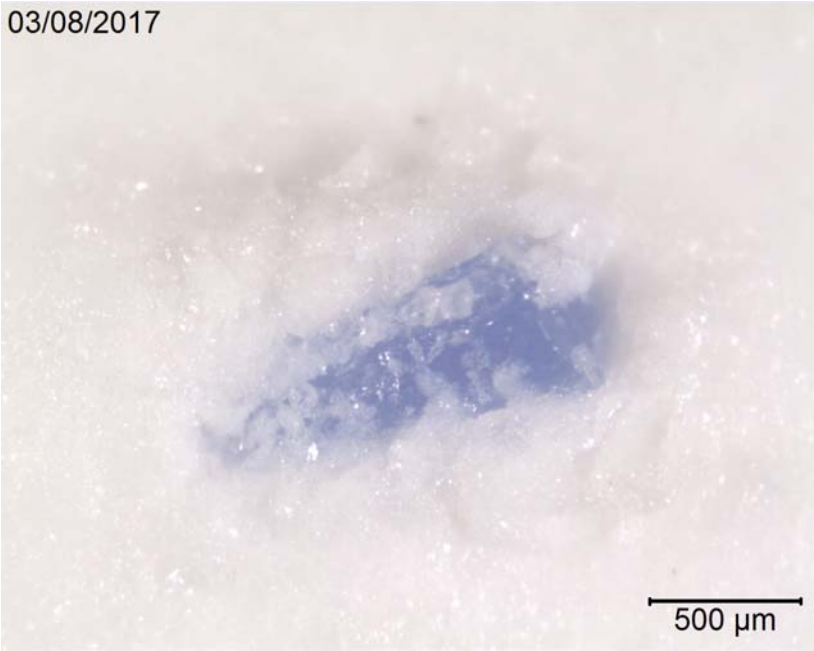


Isolation of Homogeneous Clay Sample and Preparation for SEM-EDS Analysis



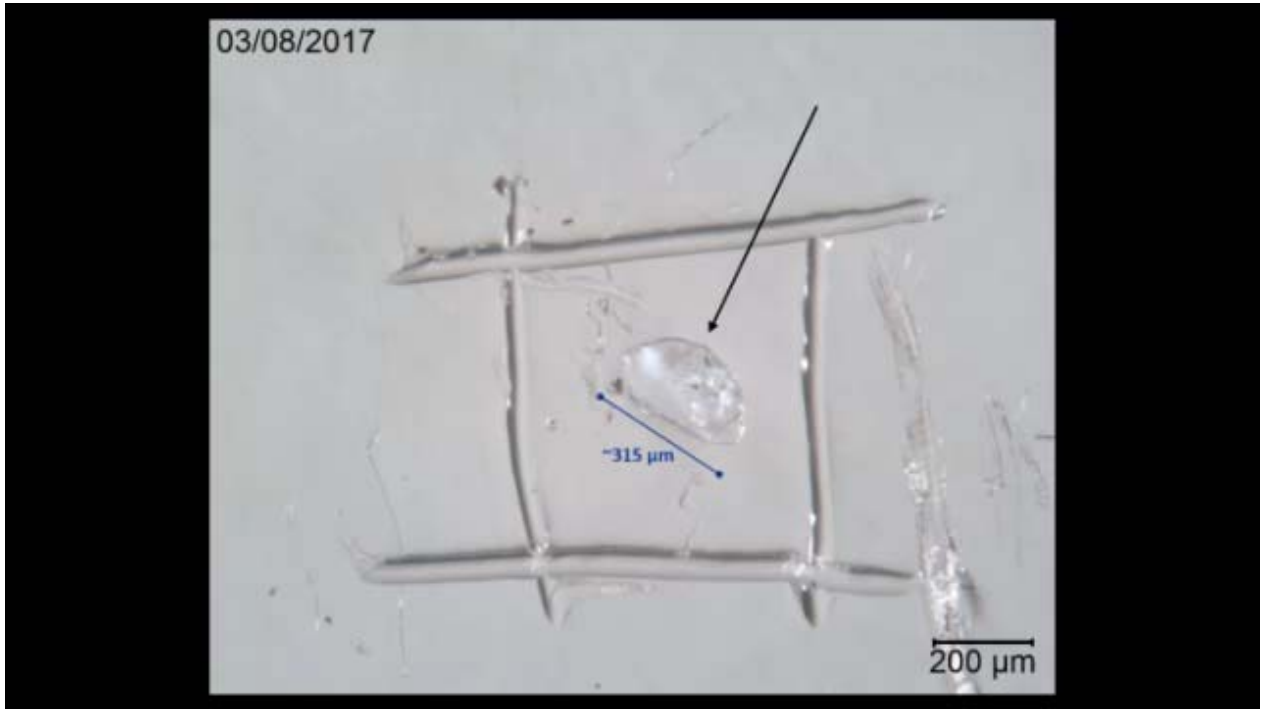
Particle Isolation and Preparation for FTIR Analysis

Stereomicroscope, 10.5X – Oblique Illumination

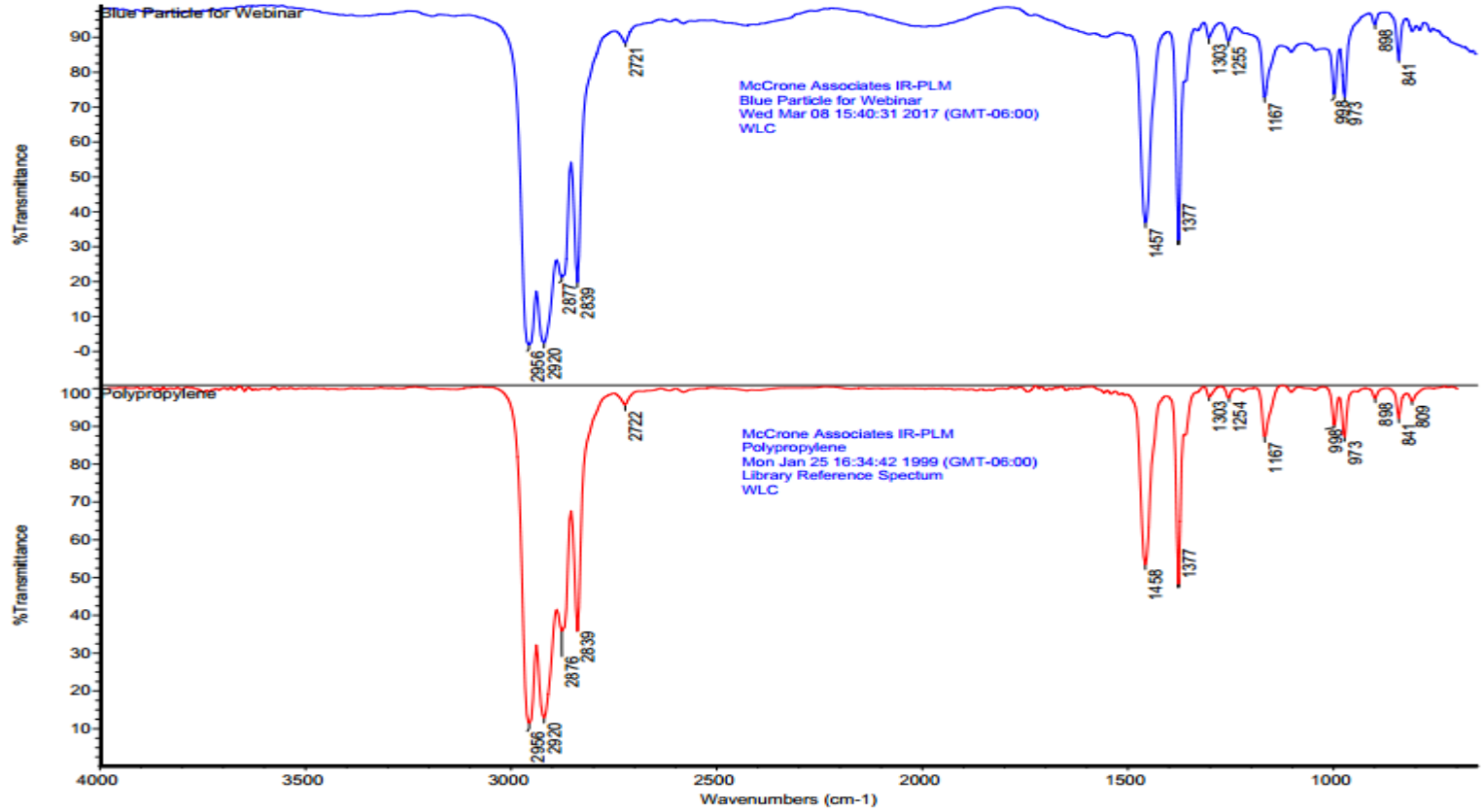


Stereomicroscope, 48X – Oblique Illumination

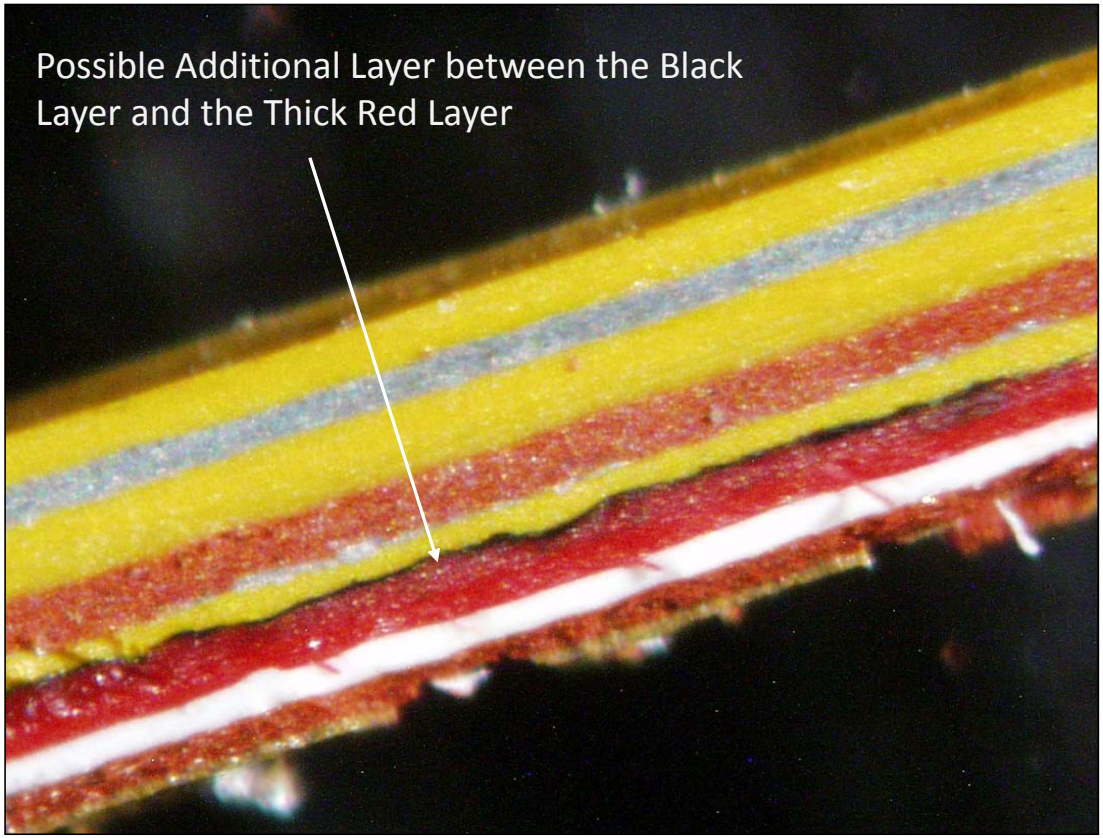
Isolation of Particle on Tablet and Preparation for Transmission micro-FTIR



Transmission Micro-FTIR of Blue Particle and Reference Polypropylene

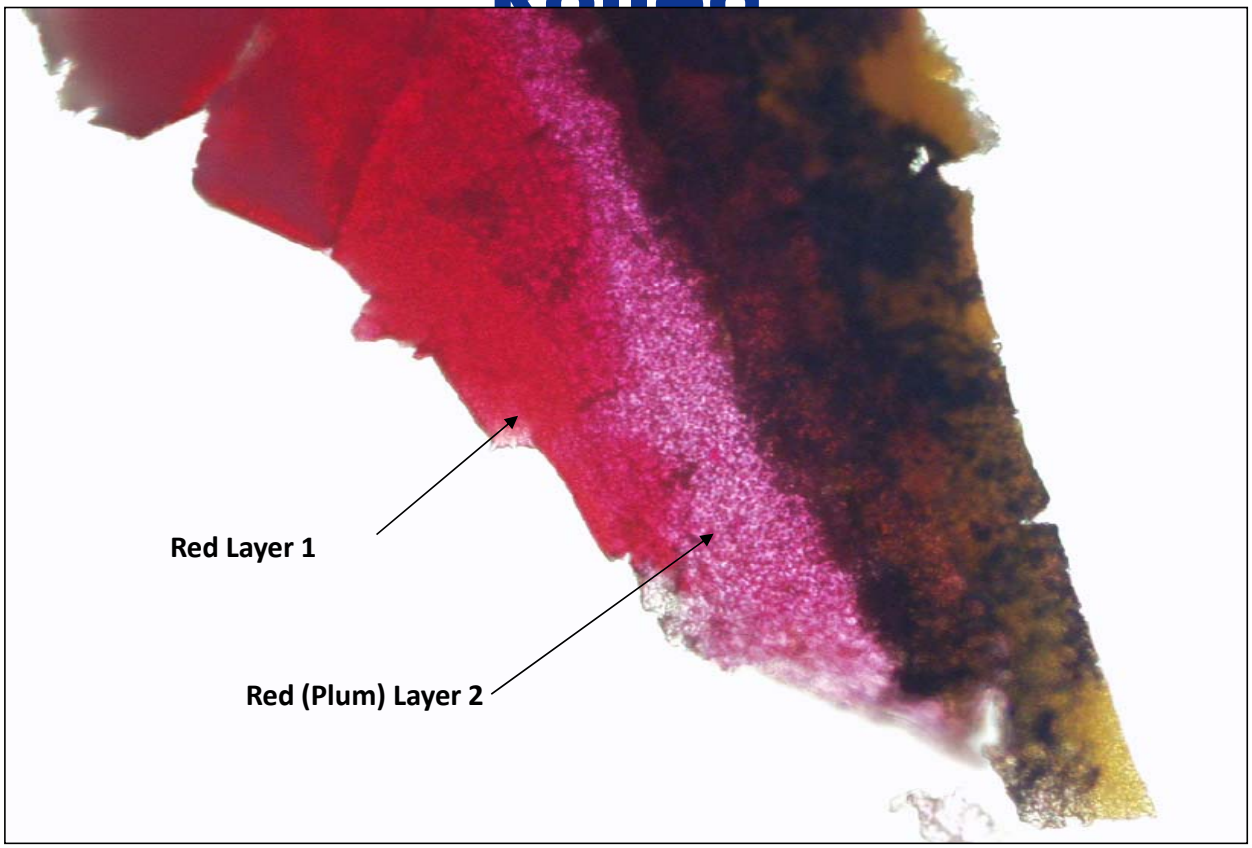


Unusual Vehicular Paint



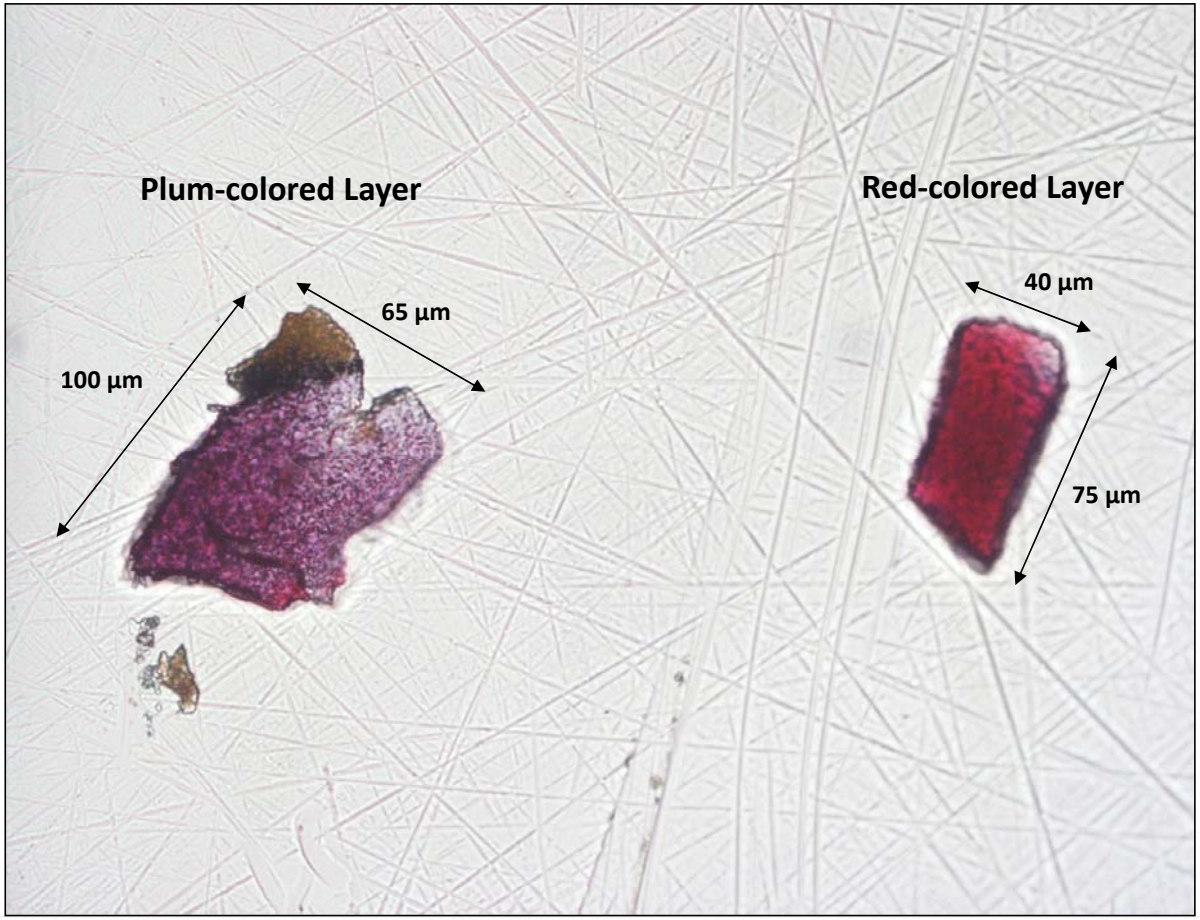
Stereomicroscope, Oblique Illumination

Area of Interest Carved From Whole and Rolled

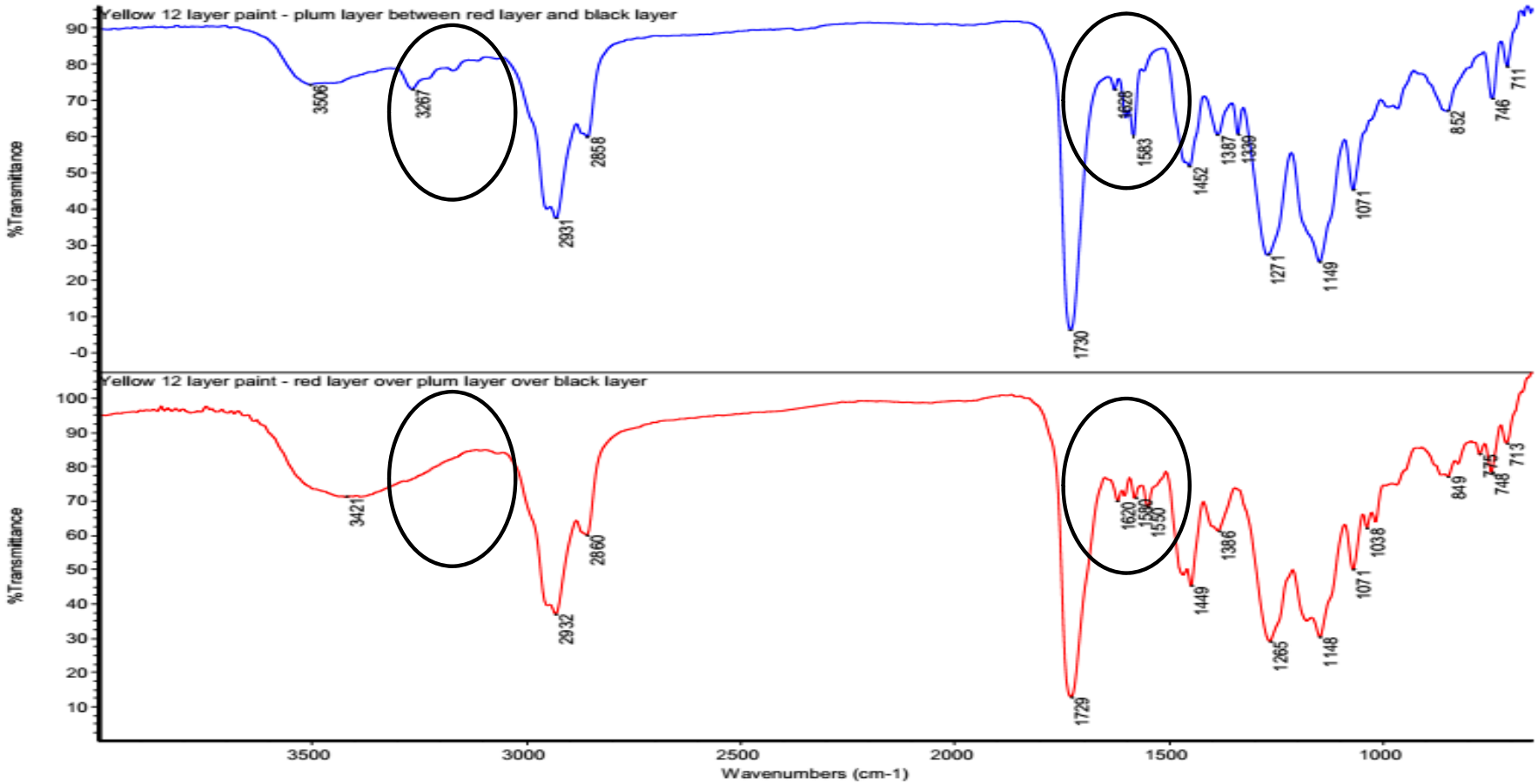


Stereomicroscope, Oblique Illumination

Both Paint Layers Prepared for FTIR Analysis



FTIR Spectra of Both Layers



Advantages That Justify Light Microscopy

- Increases understanding of the general nature of samples
- Assists in creation of homogeneous samples for analysis from more complex heterogeneous materials
- Assists in creation of improved sample preparations for instrumental analyses
- In some samples, valuable information is obtained that is not available by other instrumental techniques
- Some sample problems may be solved through microscopic observation without further analyses

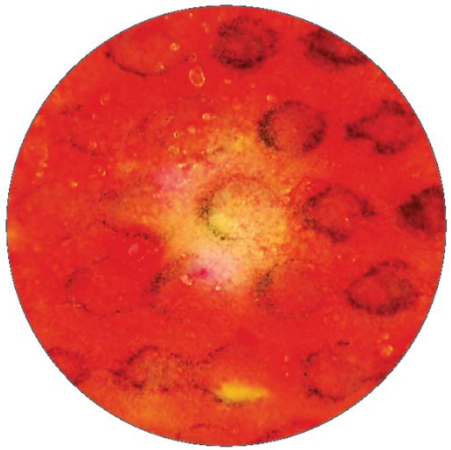
Questions?

Upcoming Courses at Hooke College

- Gunshot Residue Identification
  - April 4-6
- Freeze Drying Course
  - April 10-12
- Microscopic Particle Handling: Sample Preparation
  - April 18-20
- Forensic Hair Analysis
  - May 1-5
- Pharmaceutical Materials and Contaminants
  - May 15-19
- Polarized Light Microscopy
  - June 12-16

Upcoming Webinars

Using XPS for Industrial Problem Solving



Thursday, April 27, 2017 • 1:00 p.m.

Presenter: Kent Rhodes

Senior Vice President and Technical Director and Senior Research Microscopist, McCrone Associates

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Thank you for joining us.

Bill Chapin

Sr. Research Microscopist

wchapin@mccrone.com • (630) 887-7100