Meaningful Particle Analysis Begins with Light Microscopy

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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
Microscope

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

Polarized Light

“Set” Magnifications from 40X to 500X (1000X with Oil)
Common Illumination Techniques

Oblique – typical illumination for stereomicroscopy
Common Illumination Techniques

Transmission – typical illumination for PLM

Transmitted
Stereomicroscope

Transmitted
Polarized Light Microscope
Common Transmission Illumination

Partially Crossed Polarizer

Fully Crossed Polarizer

Analyzer (traditional)

Polarizer

Transmitted
Stereomicroscope

Polarizer

Transmitted
Polarized Light Microscope

Analyzer (1/4 λ waveplate)
Common Illumination Techniques

Reflection – powerful use on either Stereomicroscope or PLM

Coaxial Illumination on Stereomicroscope

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

Double Sided Tape  KBr Plate  Microscope Slide

~4 mm by 9 mm

2 mm

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Permanently Scribed Beryllium Stub for SEM-EDS

Ring Light Illumination at 5.25X Magnification

Coaxial Illumination at 10.5X Magnification

~ 1 inch diameter stub
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

More Appropriate Microscopic Magnification
(Same Sample Preparation)
Deposition and Sampling Information

Indicates a spattered drop of organic material that has “boiled”. Infrared data may only indicate “degraded organic material”.

Shows degraded and non-degraded locations. Indicates a heat “zone”. Should produce an identifiable infrared spectrum.
Creation of a Homogeneous Sample from Heterogeneous Material

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

Sample in refractive index liquid under a cover slip.

Spectrum 1

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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

Possible Additional Layer between the Black Layer and the Thick Red Layer

Stereomicroscope, Oblique Illumination
Area of Interest Carved From Whole and Rolled

Stereomicroscope, Oblique Illumination

Red Layer 1

Red (Plum) Layer 2
Both Paint Layers Prepared for FTIR Analysis

Plum-colored Layer

Red-colored Layer

100 µm

65 µm

40 µm

75 µm
FTIR Spectra of Both Layers

Yellow 1/2 layer paint - plum layer between red layer and black layer

Yellow 1/2 layer paint - red layer over plum layer over black layer

Wavenumbers (cm⁻¹)
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.

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