

FTIR and NIR Spectroscopy for food applications Sofia, 25 October 2018

Steve Reynolds
Molecular Spectroscopy
Sales Manager, EEMEA



Thermo Scientific: Analytical Instruments

Materials and Structural Analysis

- Molecular spectroscopy
 - FT-IR spectrometers, software and accessories
 - FT-IR microscopy and imaging, software, accessories and consumables
 - FT-NIR analysers, software, accessories and consumables
 - Infrared gas analysers
 - Raman spectrometers, software and accessories
 - Vis and UV/Vis spectrophotometers, software, accessories
 - NMR



Food Quality FTIR Microscopy

Is your food safe?
What FTIR microscopy
can bring to the table.



Agenda

- Introduction into FTIR microscopy
- Point and shoot applications
- Advanced Visualization
- Imaging applications

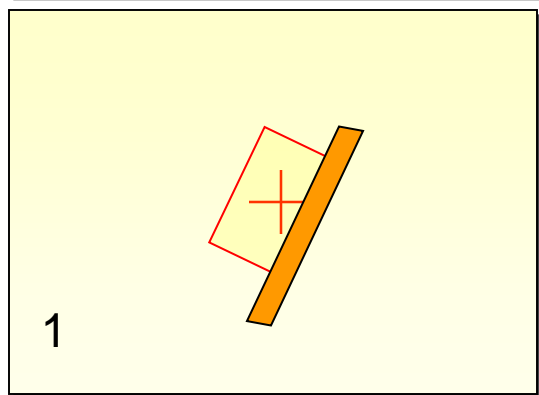
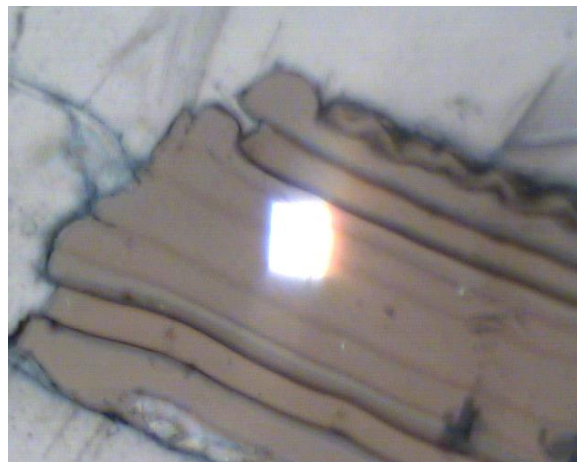
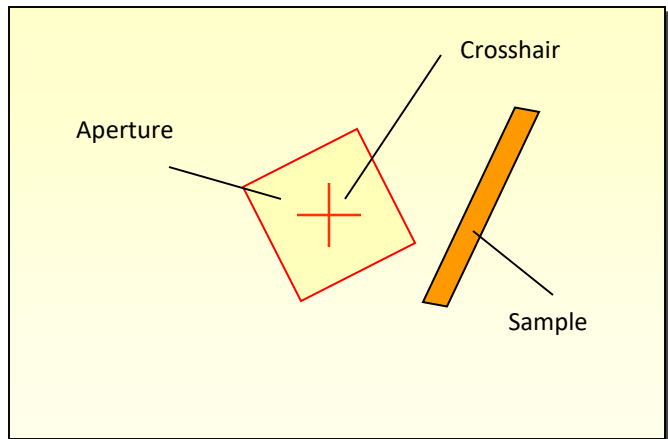


FT-IR Microscopy is useful for...

- Samples that are $<100\ \mu\text{m}$ in size
- Samples that can not be physically separated
- Combining visible and infrared information in one report format



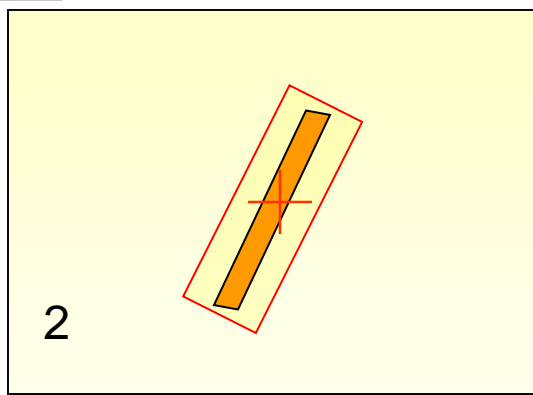
The Aperture : Selecting a specific area or domain for analysis



Sample not in the center



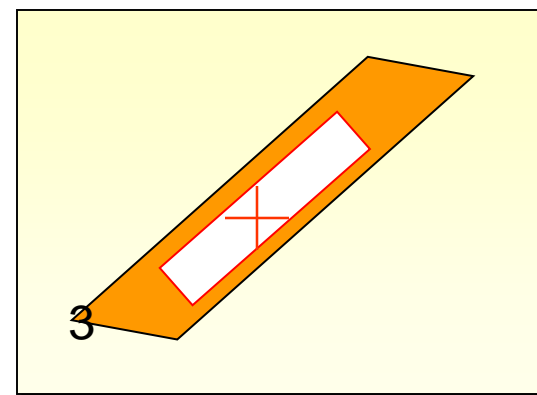
Aperture bigger than sample



Sample centered



Aperture bigger than sample



Sample centered



Aperture smaller or equivalent to sample size

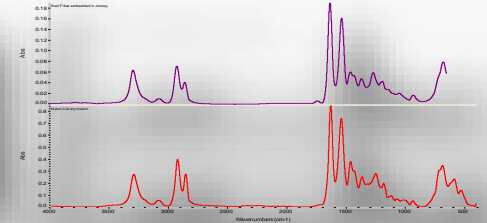


The Value of FT-IR Microscopy

Which FT-IR microscopy technique best fits your needs?

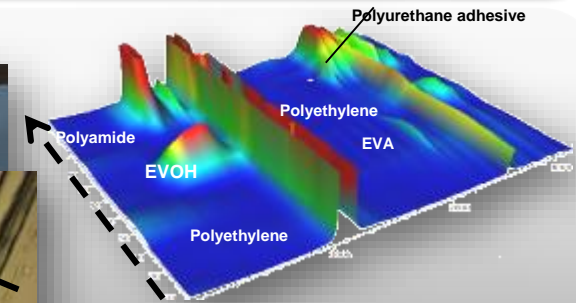
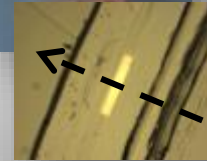
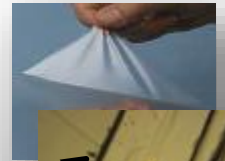
- Single point analysis for...

- Single specimen identification
 - Fibers, Particles, Inclusions
- Observe, get a spectrum, and identify



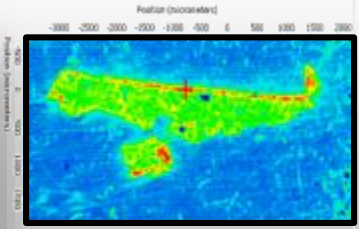
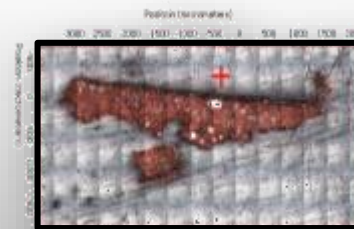
- Multi-point and line maps for...

- Sections and small area characterization
 - Multi-layer and other cross sections
 - Small area materials distribution studies
- Observe, get a series of spectra and identify / measure



- 2-D Imaging for...

- Large area characterization
- Large area surveys
- Observe, set an area, get an ordered array of spectra and extract chemical information

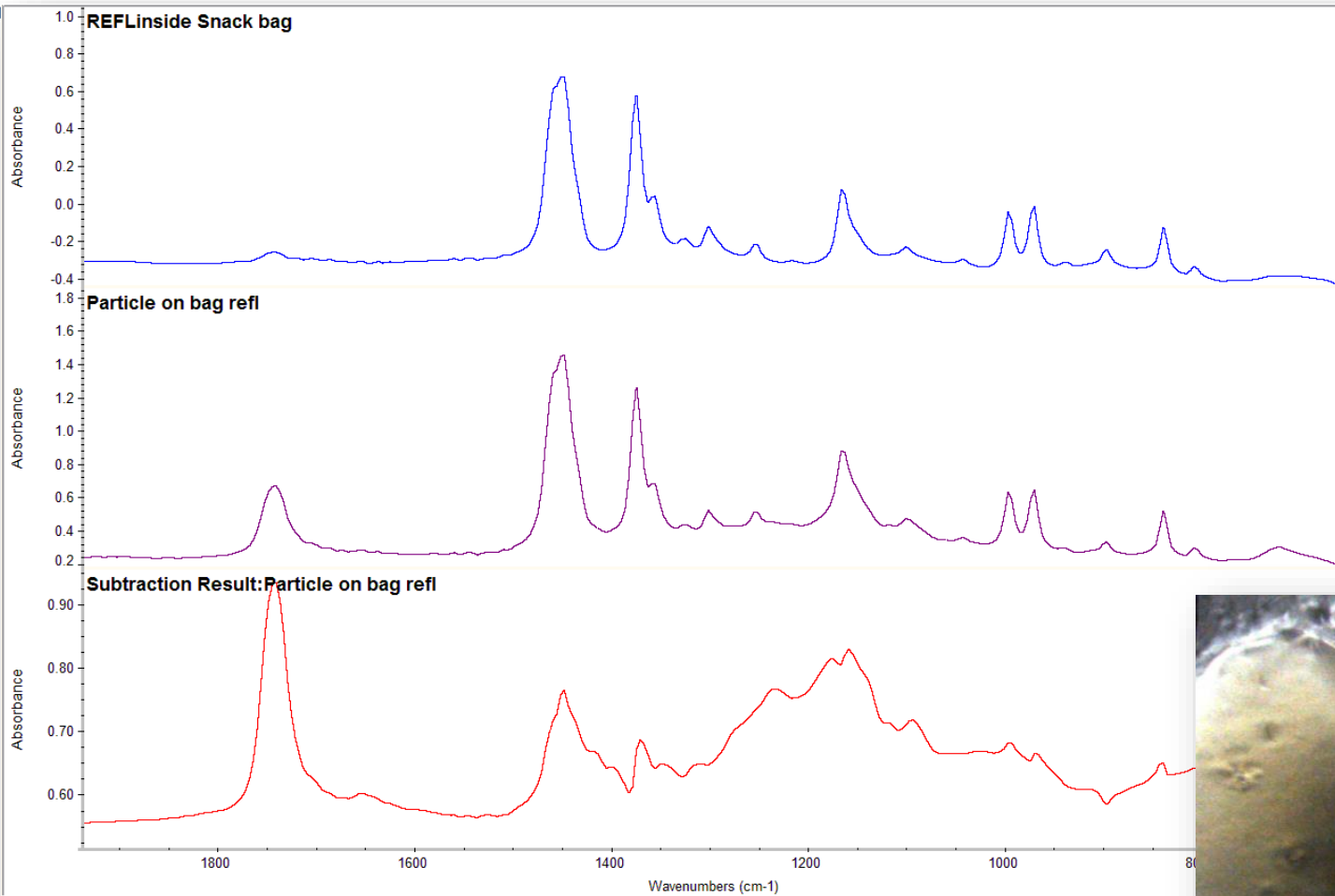


Nicolet iN5 FTIR Microscope

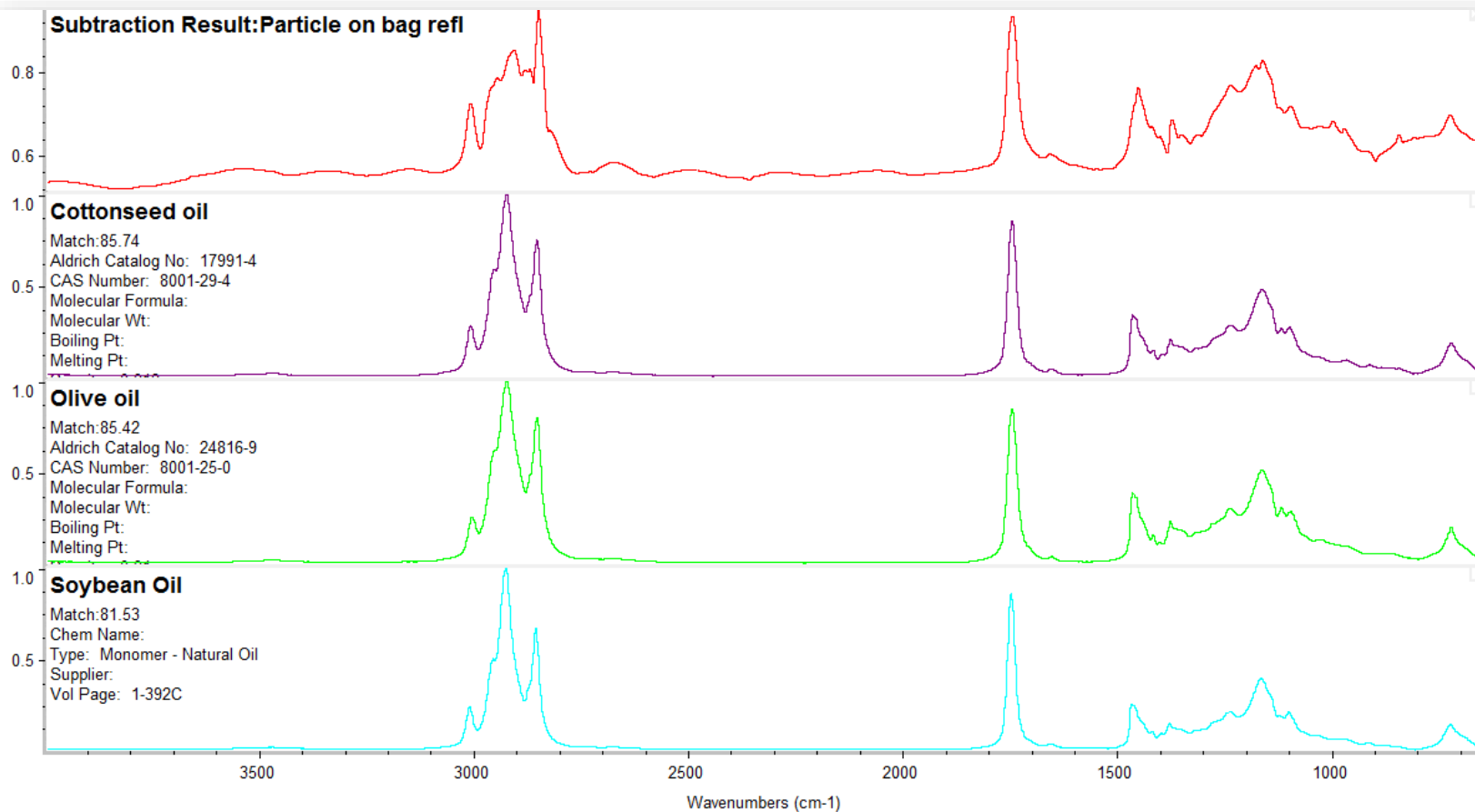
- Simple walk up and use design
- Completely manual operation
 - Fixed and variable apertures available
- Detectors
 - No Fuss room temperature
 - High sensitivity Liquid nitrogen cooled
- 10x fixed objective



Applications: Food Packaging



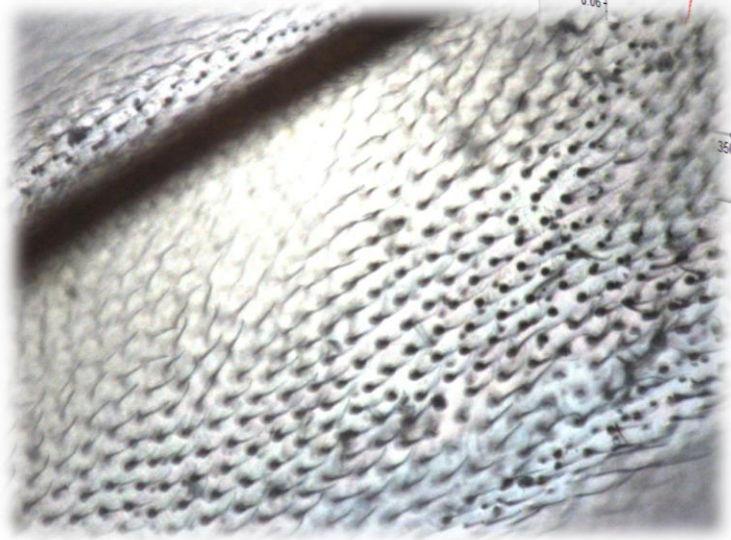
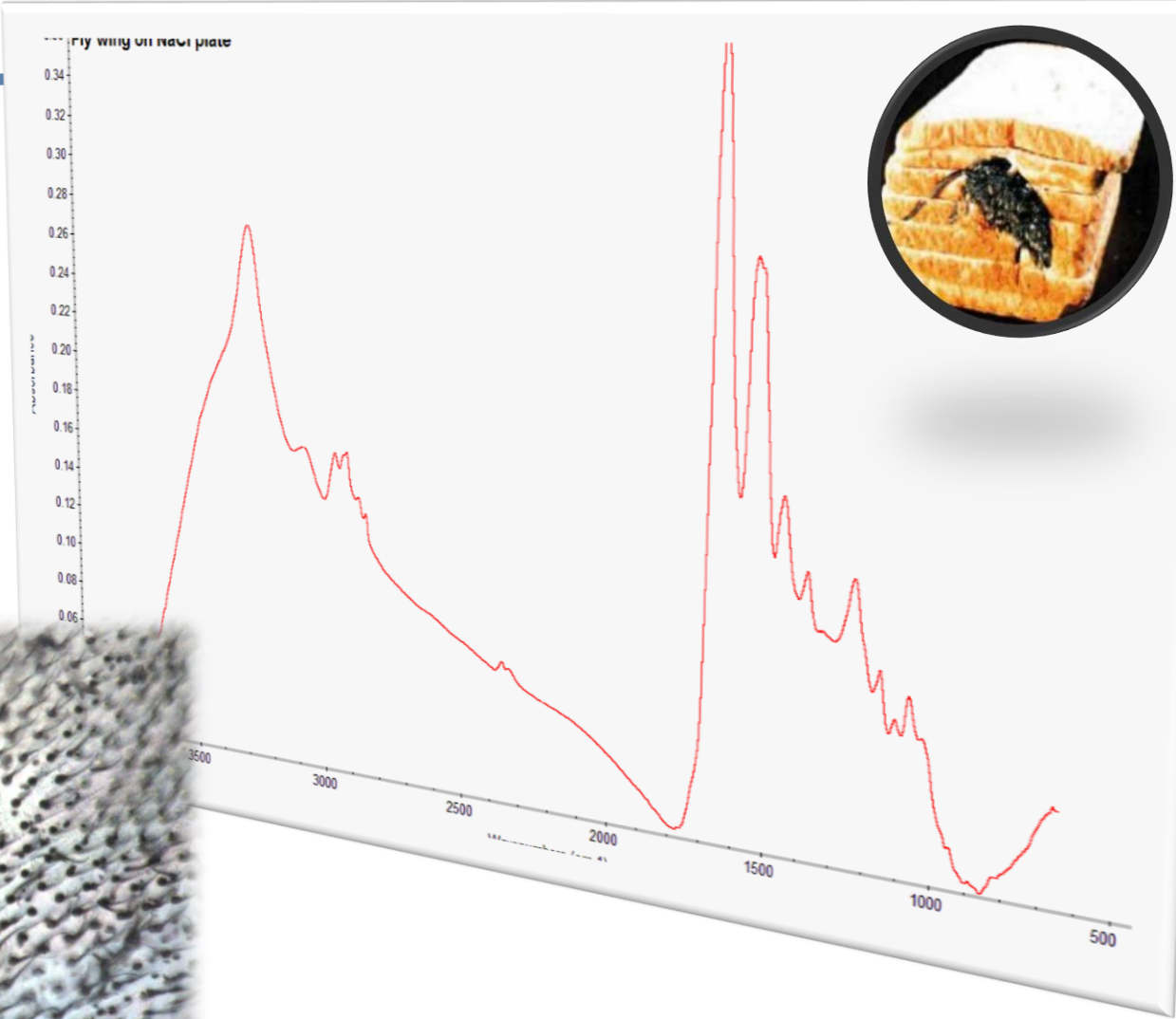
Applications: Food Packaging



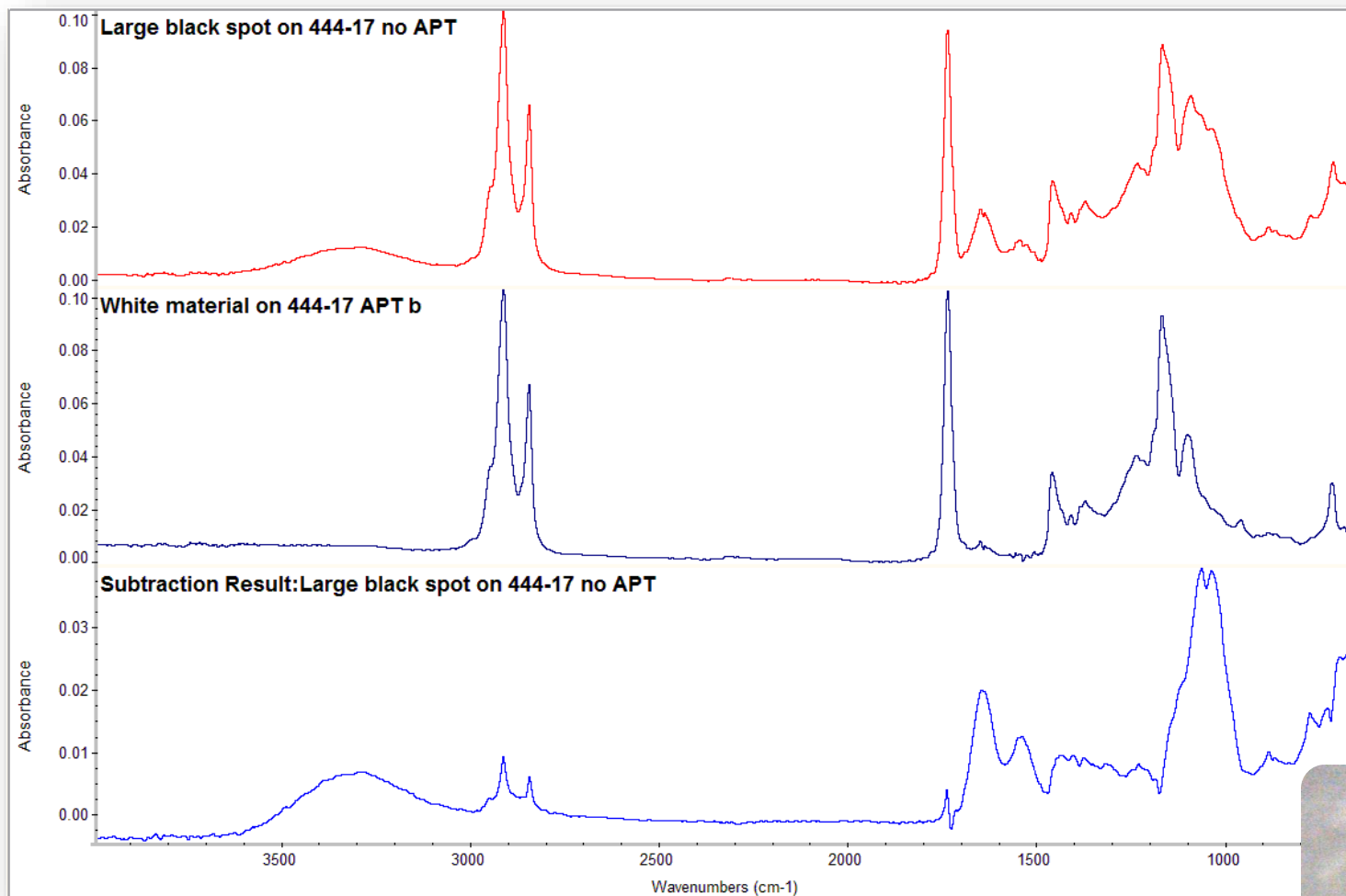
Index	Match	Compound Name	Library Name
3894	85.74	Cottonseed oil	HR Aldrich FT-IR Collection Edition II
3895	85.42	Olive oil	HR Aldrich FT-IR Collection Edition II
870	81.53	Soybean Oil	HR Coatings Technology
3896	80.68	Castor oil	HR Aldrich FT-IR Collection Edition II

U.S. Food and Drug Administration

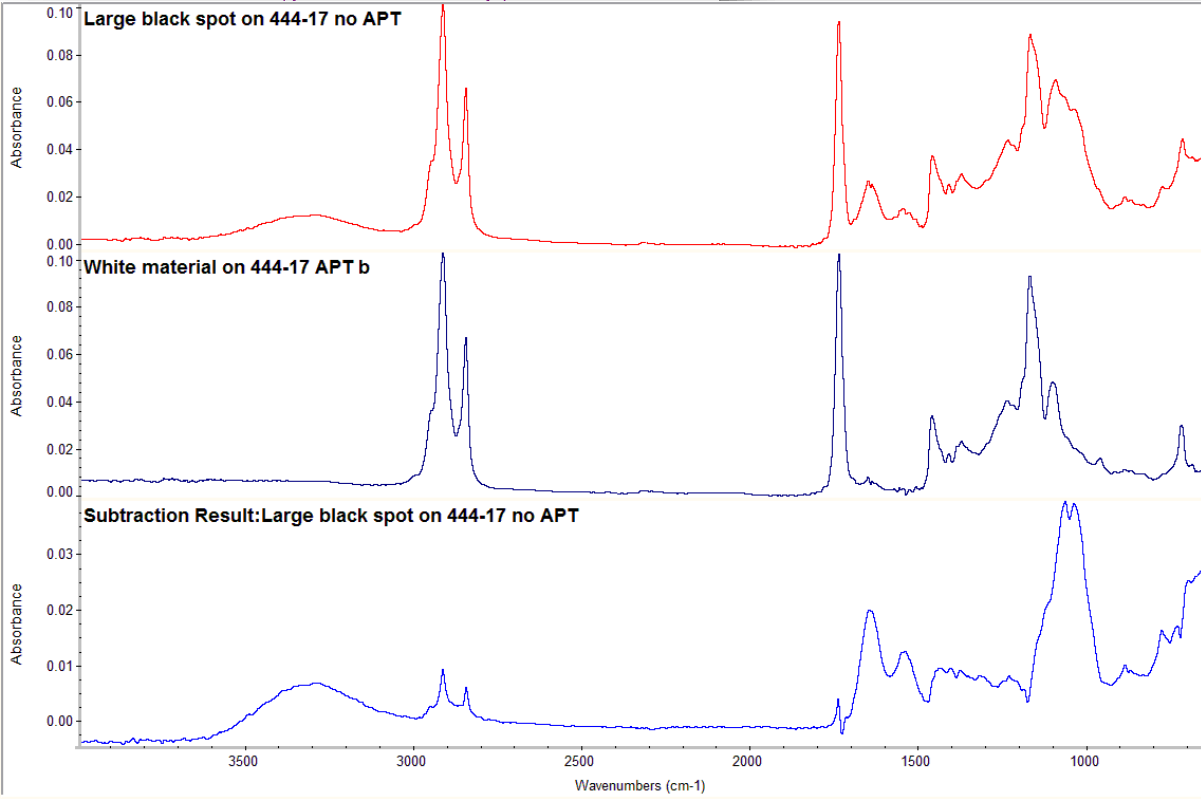
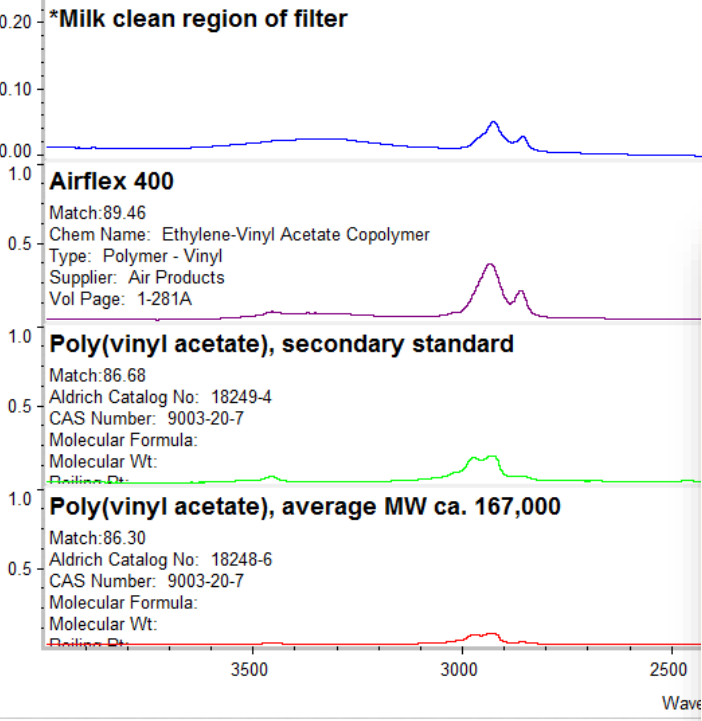
- *food defect action level list*
- Acceptable level in each type of food
- Economically unfeasible to eliminate



Applications: Filter contamination

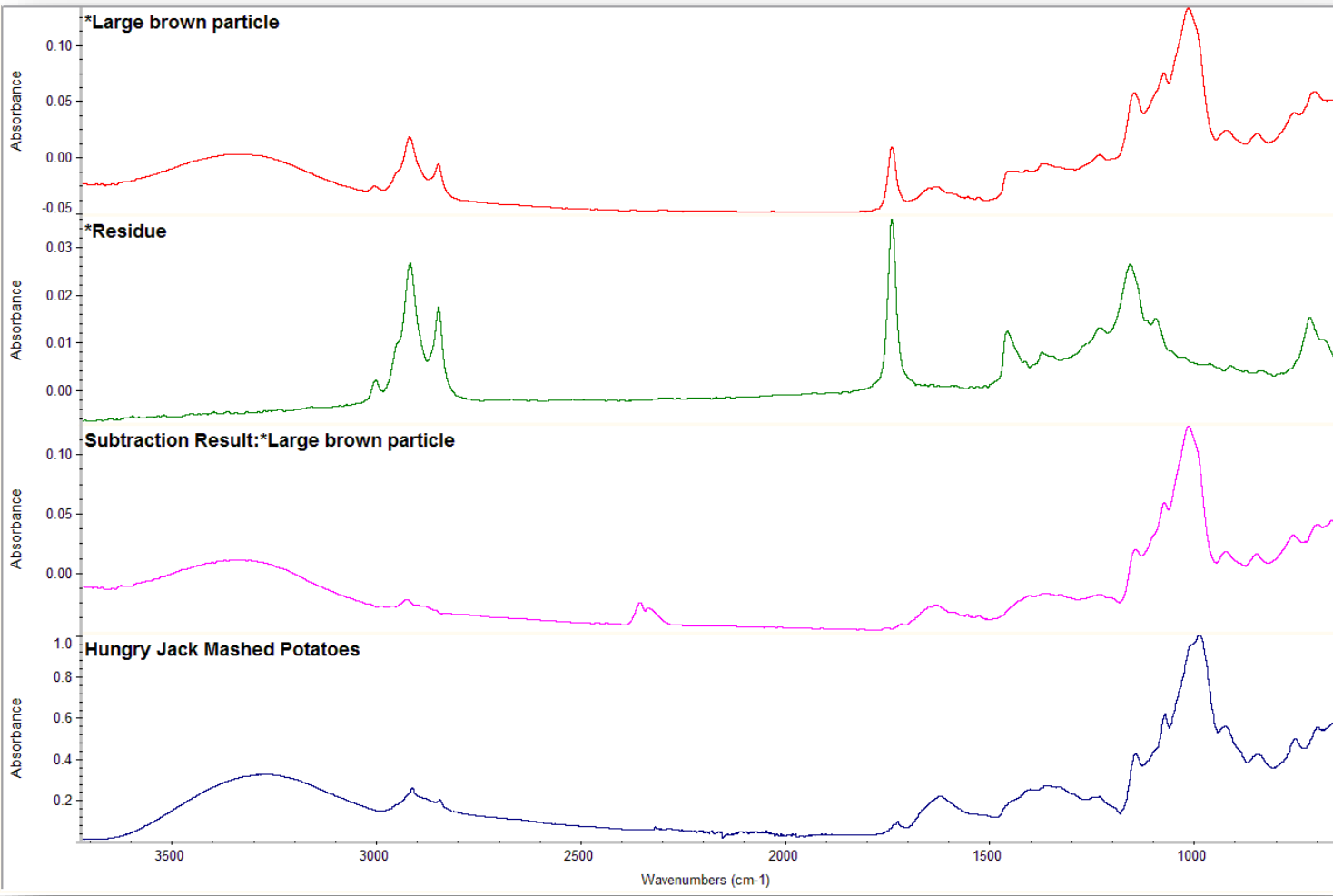


Applications: Filter contamination

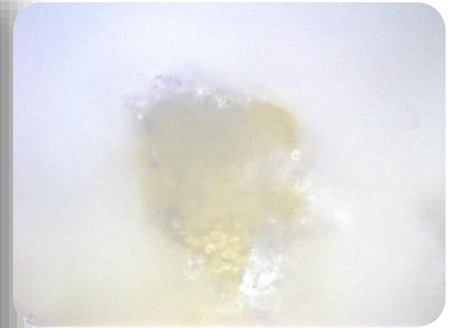
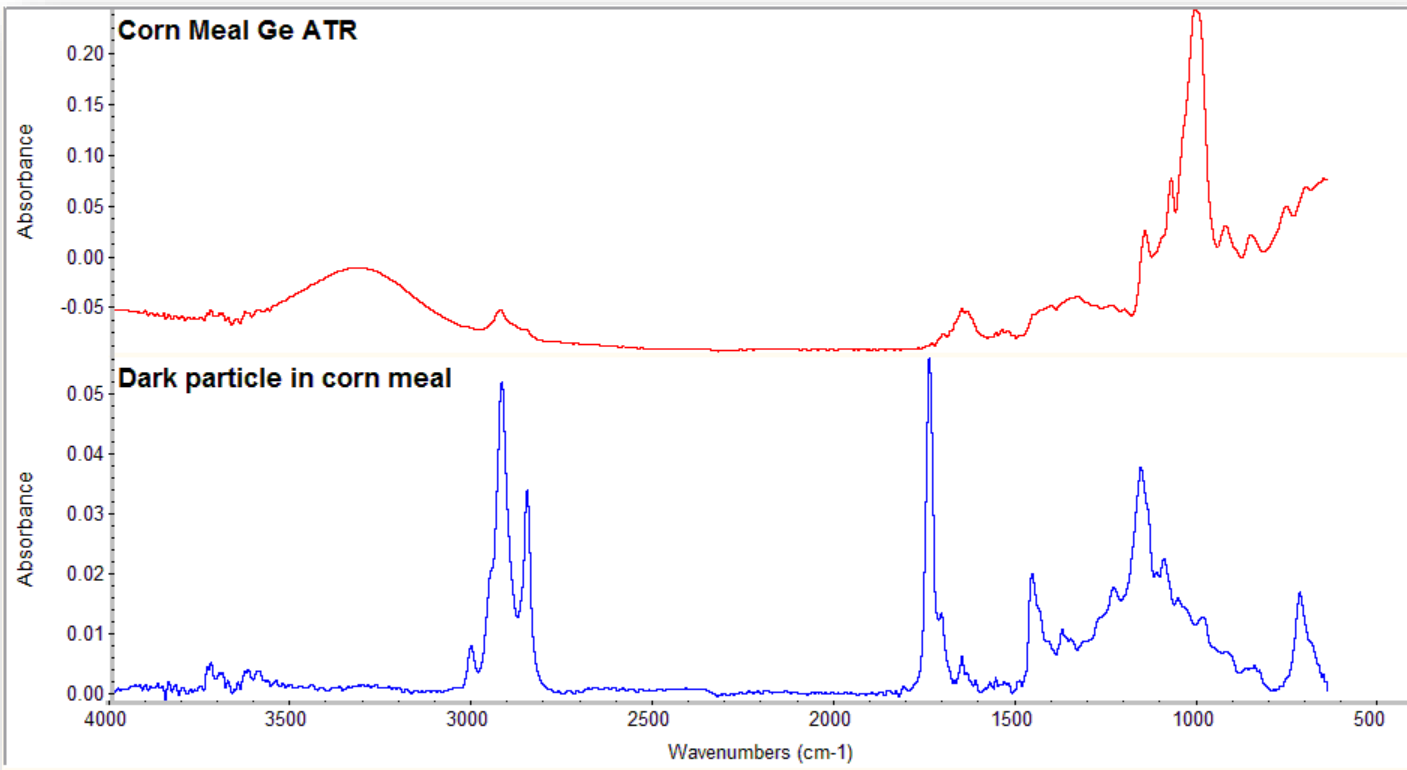


Index	Match	Compound Name
535	89.46	Airflex 400
18055	86.68	Poly(vinyl acetate), secondary standard
18058	86.30	Poly(vinyl acetate), average MW ca. 167,000

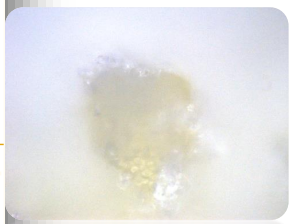
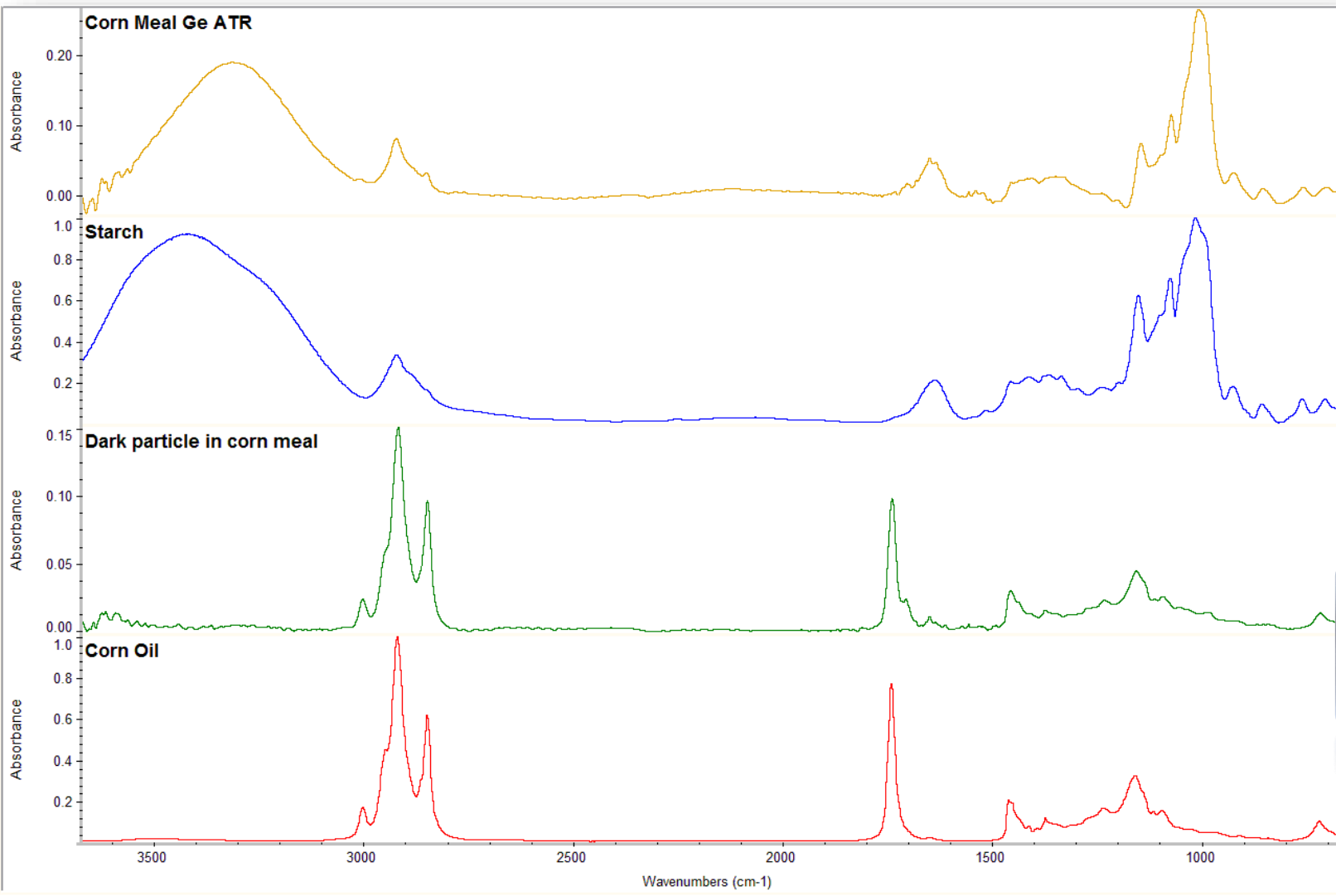
Applications: Potato chip bag by Ge ATR



Applications: Black Particle in Corn Meal



Applications: Black Particle in Corn Meal

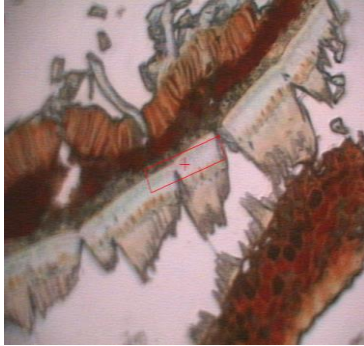


Nicolet Continuum Microscope

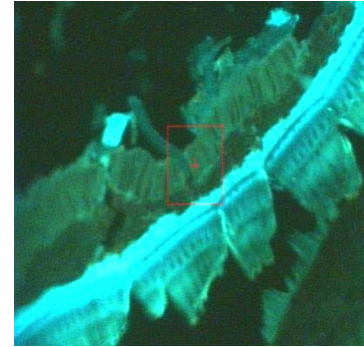


- Best spectral purity
 - Dual aperture
 - Infinity corrected optics
 - Dual detectors
 - Four-place nosepiece
- Best spatial resolution
 - Better than 5 microns, Slide-On Tip ATR
- Simultaneous viewing and collection
- Best viewing capabilities
 - Infinity corrected, Reffachromat™
- Ideal choice for demanding applications
- Contrast enhancement options
 - Polarized light
 - Fluorescence
 - Differential Interference Contrast

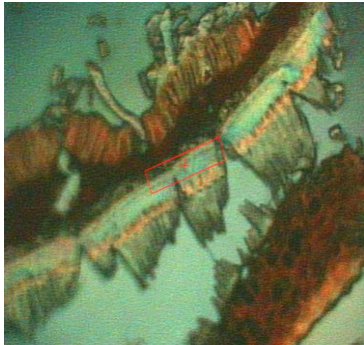
Contrast Enhancement Techniques



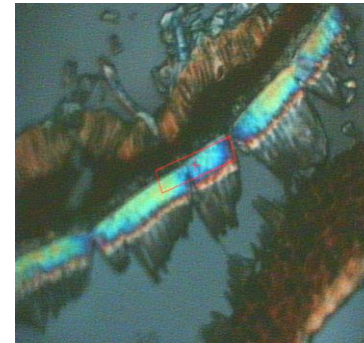
Brightfield/darkfield
Traditional contrast techniques for most samples



Fluorescence
Reveals small sample details not normally seen



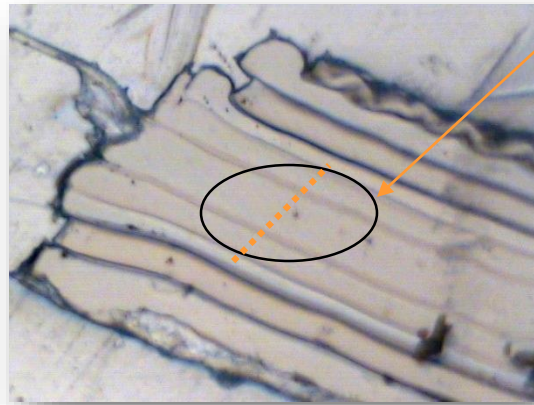
Differential interference contrast (DIC)
Imparts color & 3D to isotropic samples



Polarized light
Offers insight to sample anisotropy or thickness

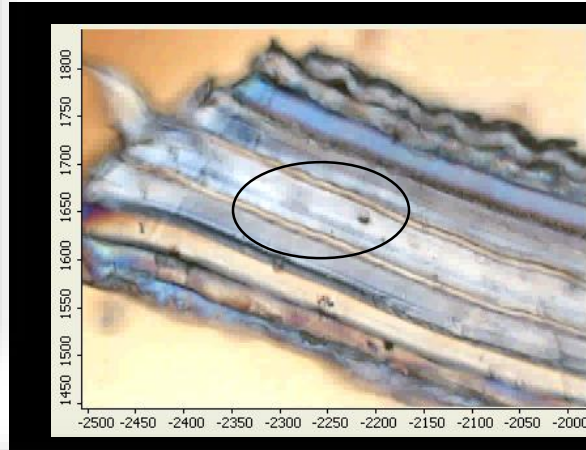
Cotton seed coat images courtesy of Dave Himmelsbach USDA-ARS-Russell Research Center

DIC Reveals More Details



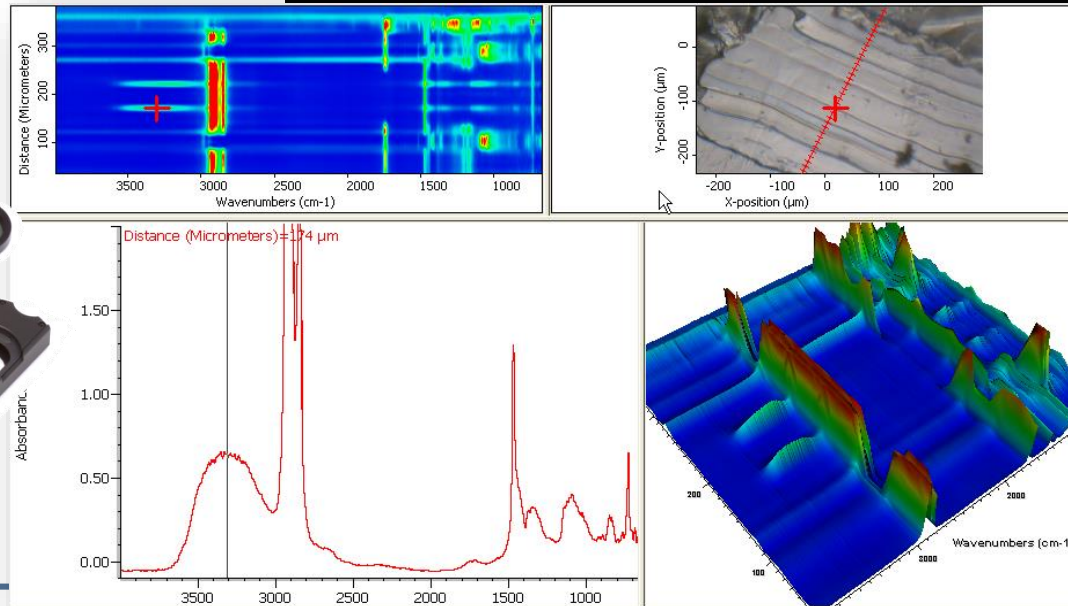
Polymer laminate film

- How many layers do you see here?



How many now?

- The two thin lines in the center are not just boundaries
- They show different refractive index hence, they are layers of a different material

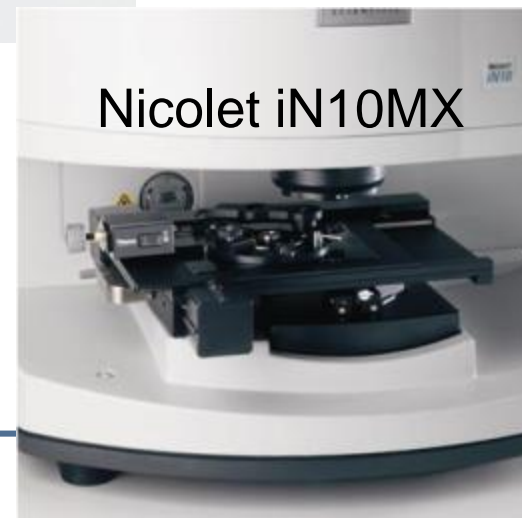
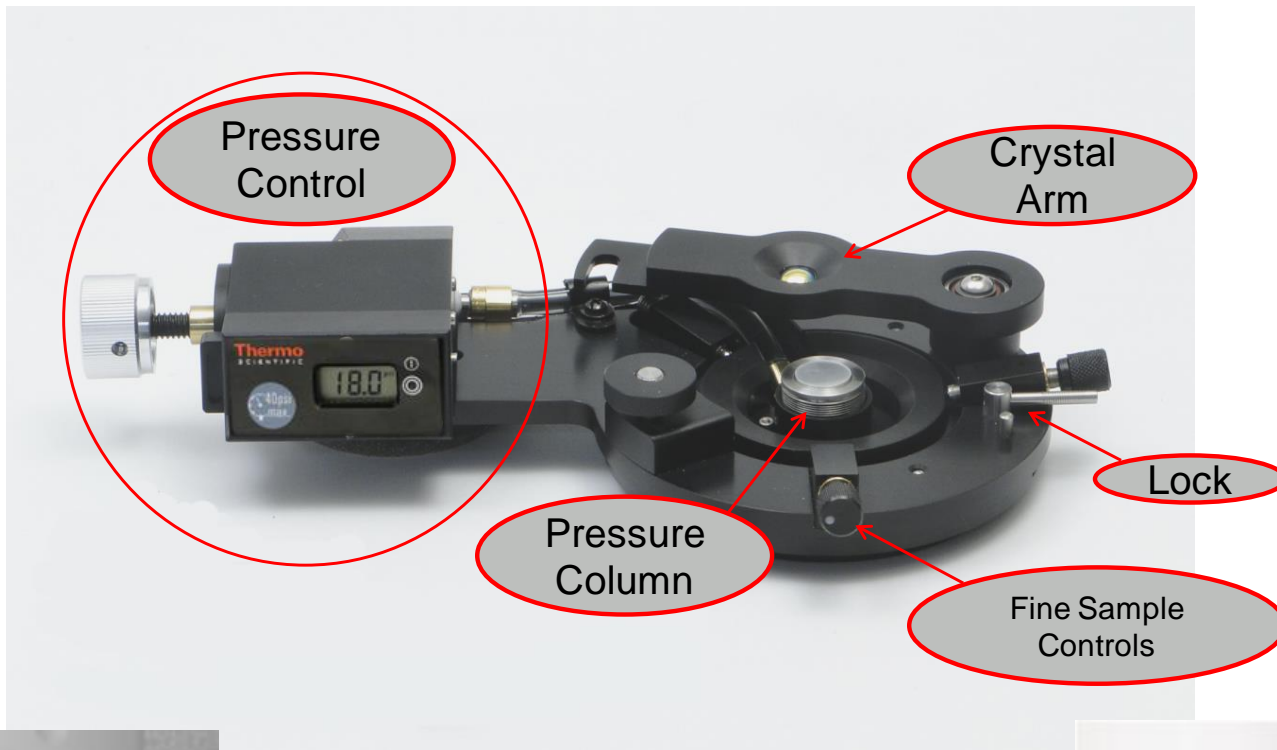


Nicolet iN10 FT-IR Integrated Microscope

- Stand-alone microscope
 - Incorporates all FT-IR critical components: interferometer, source, laser and detectors
 - Does not require an external spectrometer
- Microscopy simplified
 - Single Aperture
 - Fixed focal length
 - 15X Objective
 - Three detector options
- Unparalleled advantages
 - High signal to noise performance
 - Room temperature microscopy
 - Small footprint, stand alone unit
 - High performance array detector

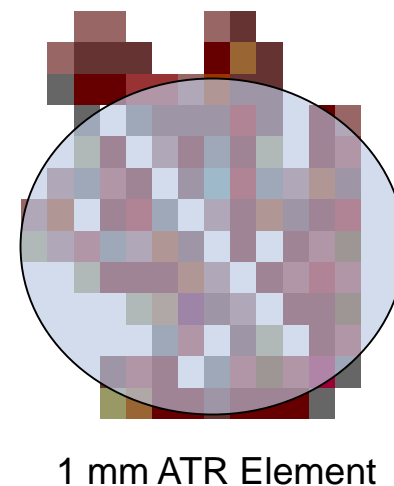
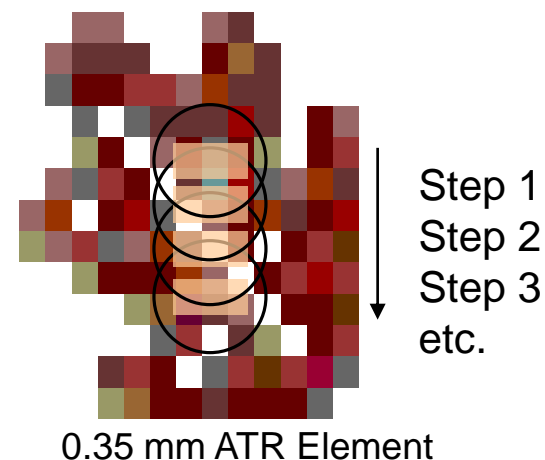


Thermo Scientific Imaging ATR Accessory



Major difference between Slide-on ATR?

- Slide-on: Crystal is attached to the objective
 - Each sample point is a separate contact
 - Contact cycles synchronized with stage movement
 - Requires breaking and re-establishing contact
 - Good for rough (on micro scale) surfaces
- Imaging ATR Accessory: Crystal is attached to the STAGE
 - Sample is compressed only once against the ATR element
 - Stage moves the sample with the ATR element freely *under* the objective – up to 10x faster

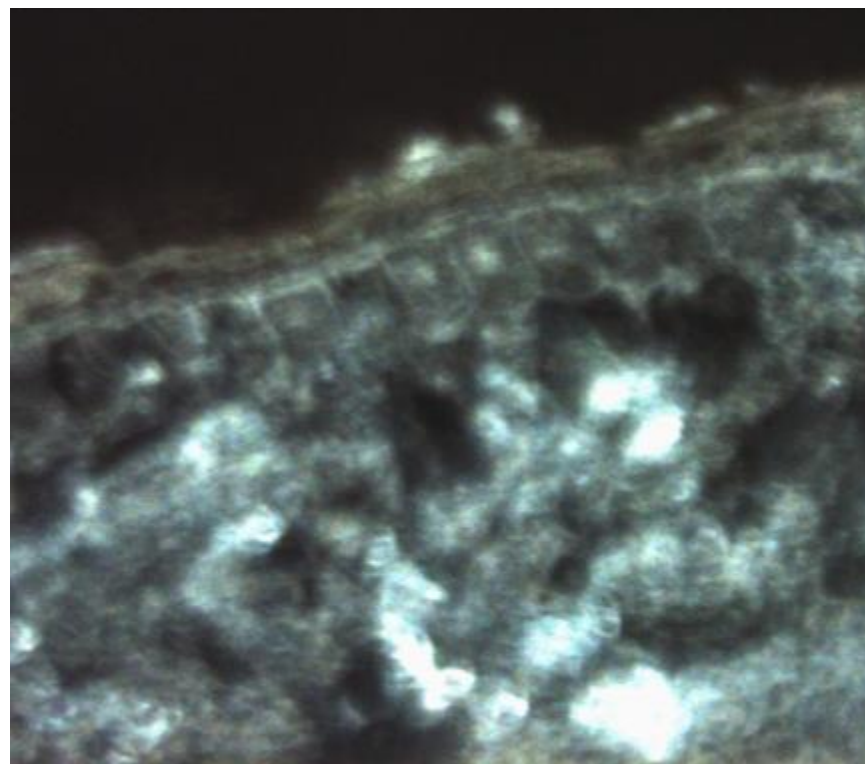


Preparing a Cereal and Oil Seeds for Imaging

Fix specimen to the center of a sample holder with an appropriate cement (5-min epoxy in this case)



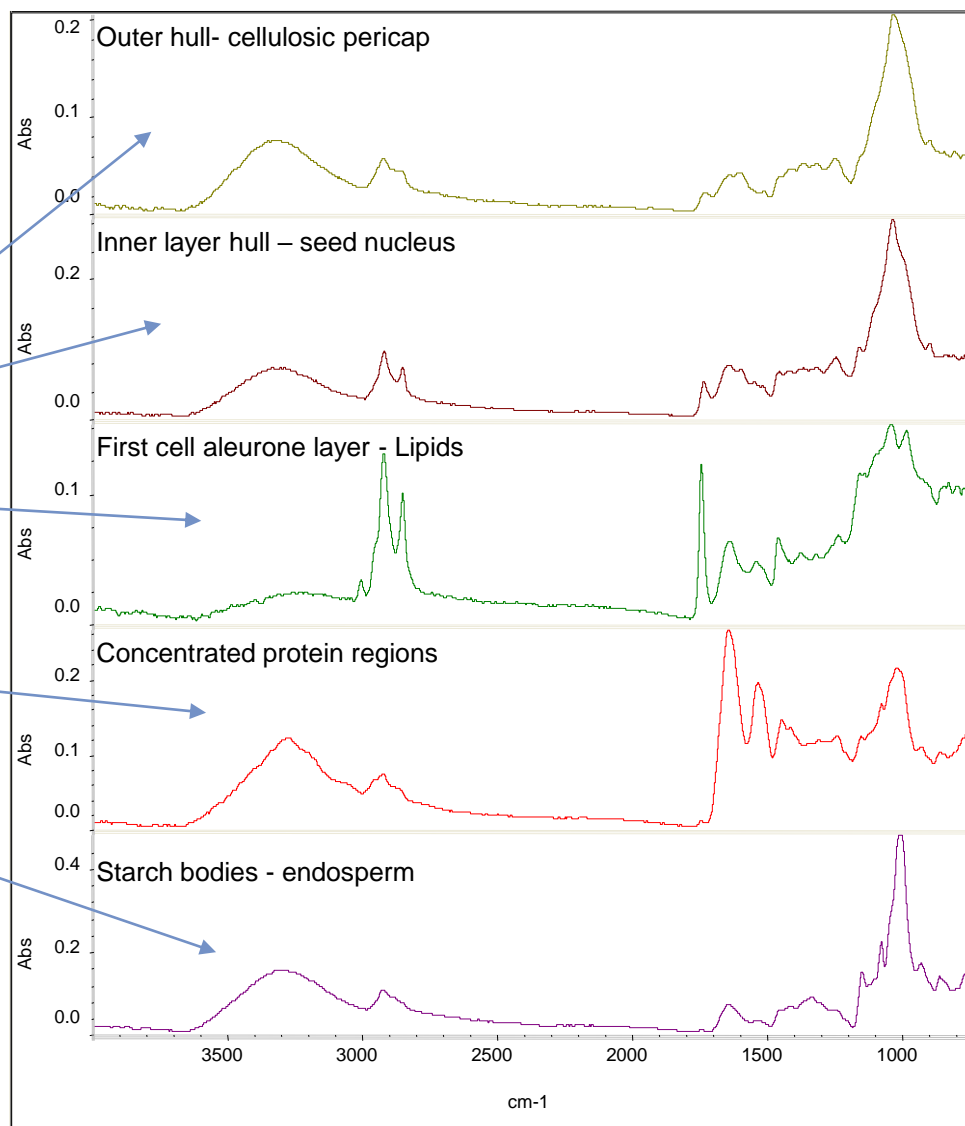
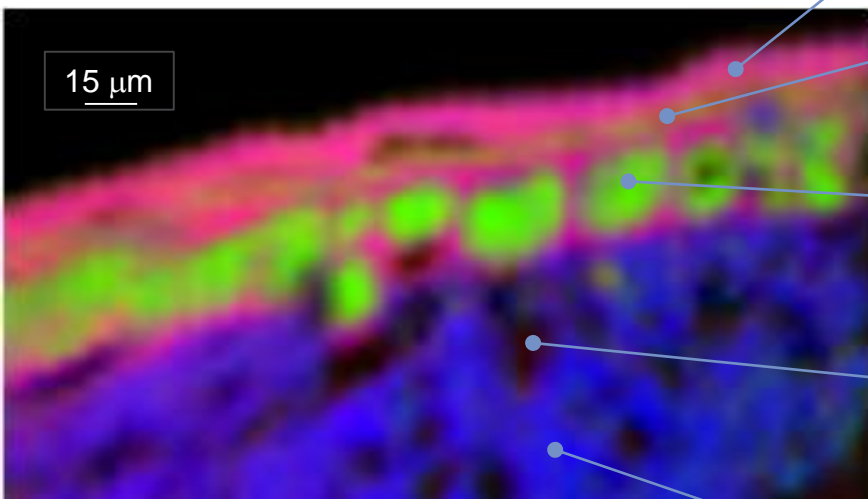
Cut parallel to Sample holder surface with simple microtome with disposable blade. Adjust feed rate and blade angle for smooth cut.



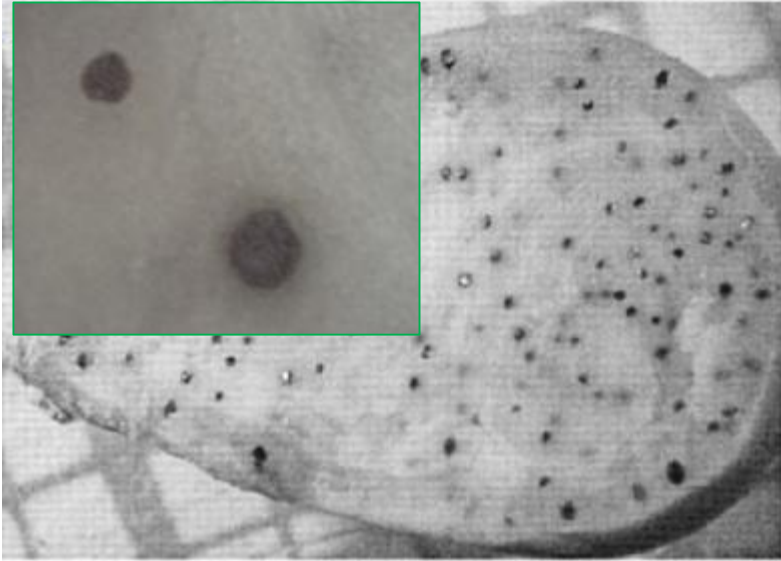
Cross section view of seed coat and aleurone layer

Key chemical domains of wheat seed

ATR Chemical Image



Pigment Glands in Cotton Seeds

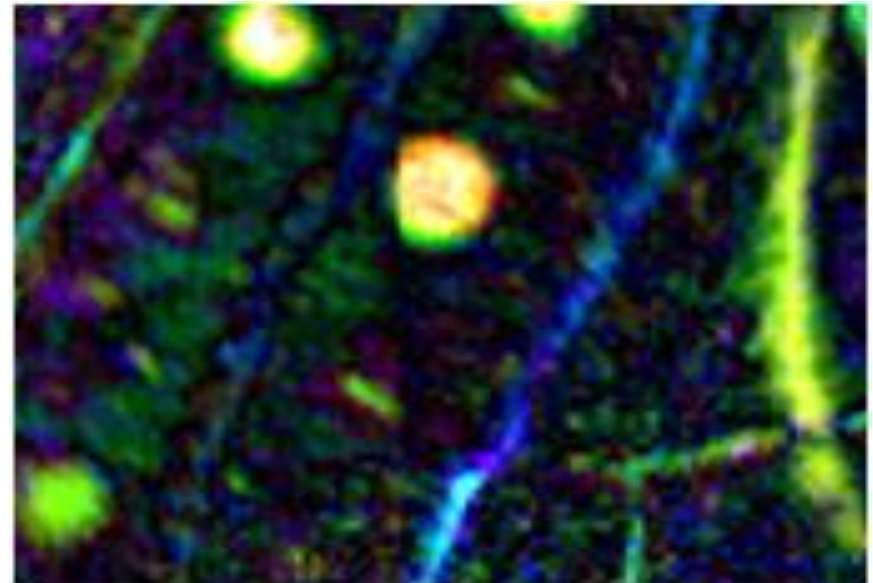


Video at different magnifications
(Inset: FOV of Nicolet iN10, 500 microns)

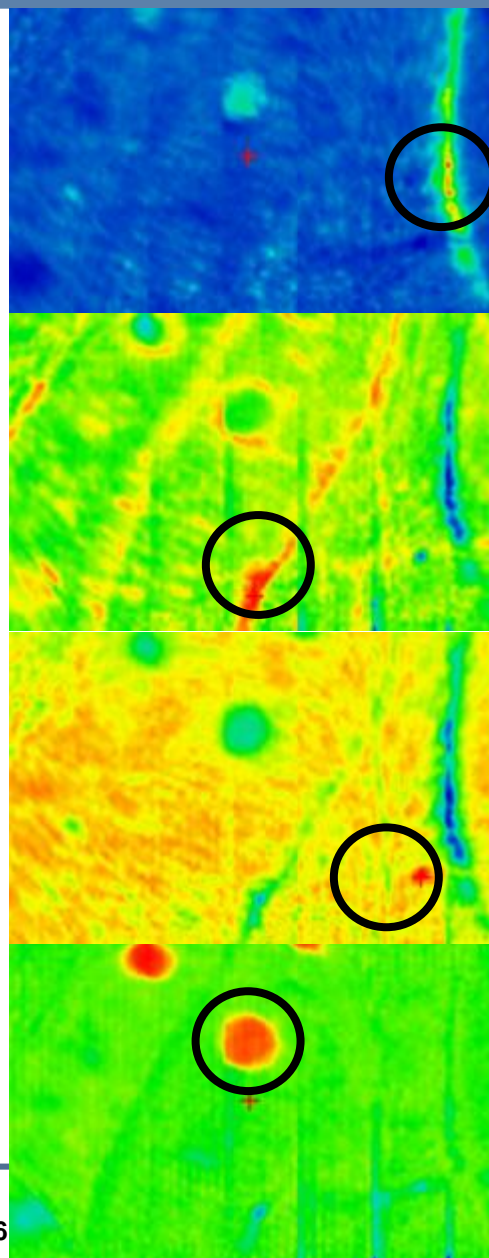
H. Benbouza, G. Lognay, R. Palm, JP. Baudoin,
and G. Mergeai, *Crop. Sci.*, **42**, 1937 (2002)

- Cotton seeds contains variable amount of toxic gossypol
- Chemical imaging by ATR shows the glands and elongated structures
- 6.25 micron pixel, ~10,000 spectra

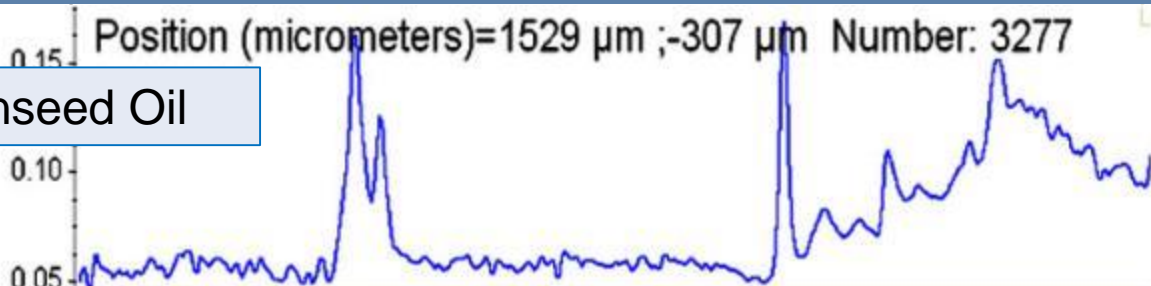
ATR Chemical Image



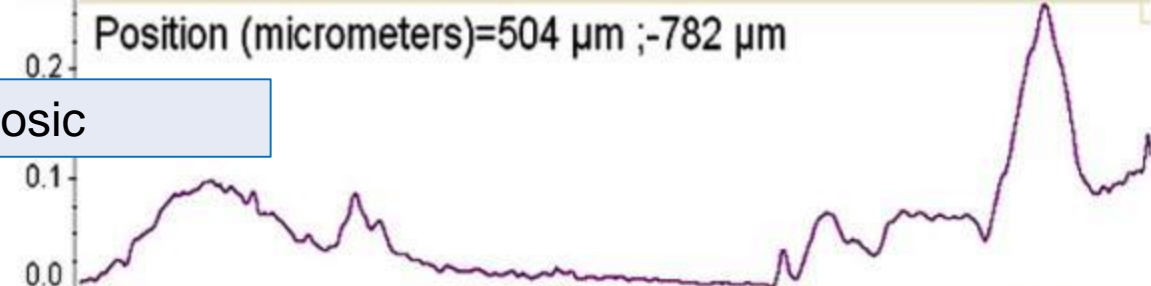
Correlation Profiles – Highlight Specific Domains



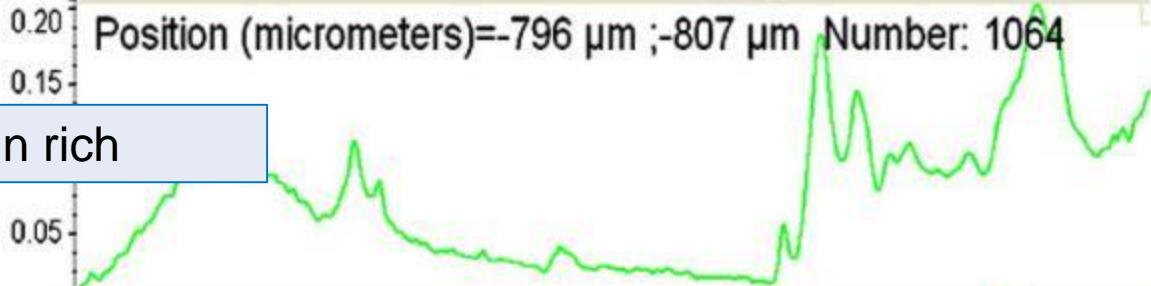
Cottonseed Oil



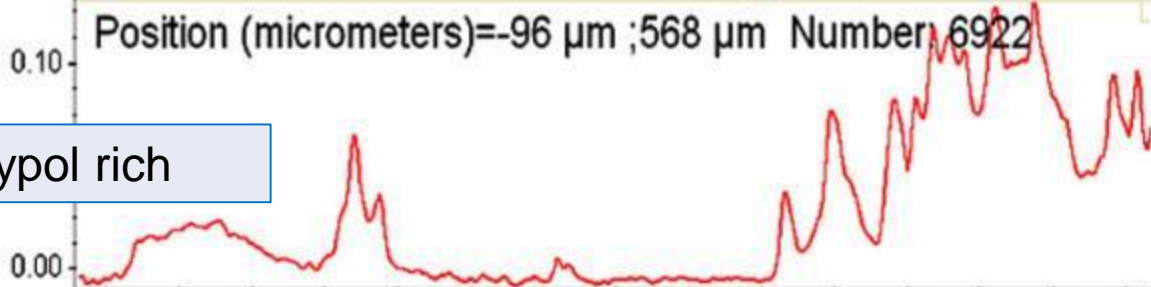
Cellulosic



Protein rich



Gossypol rich



Wavenumbers (cm^{-1})

Nicolet FT-IR Microscopes Product Line

- Nicolet iN5™ infrared microscope
 - Simple and reliable “point and shoot” solution
- Nicolet Continuum™ infrared microscope
 - Incomparable for research and analytical service labs
- Nicolet iN10 and iN10 MX FT-IR imaging microscope
 - Microscopy simplified, with the speed of array imaging



Nicolet iN5
microscope



Nicolet Continuum
microscope



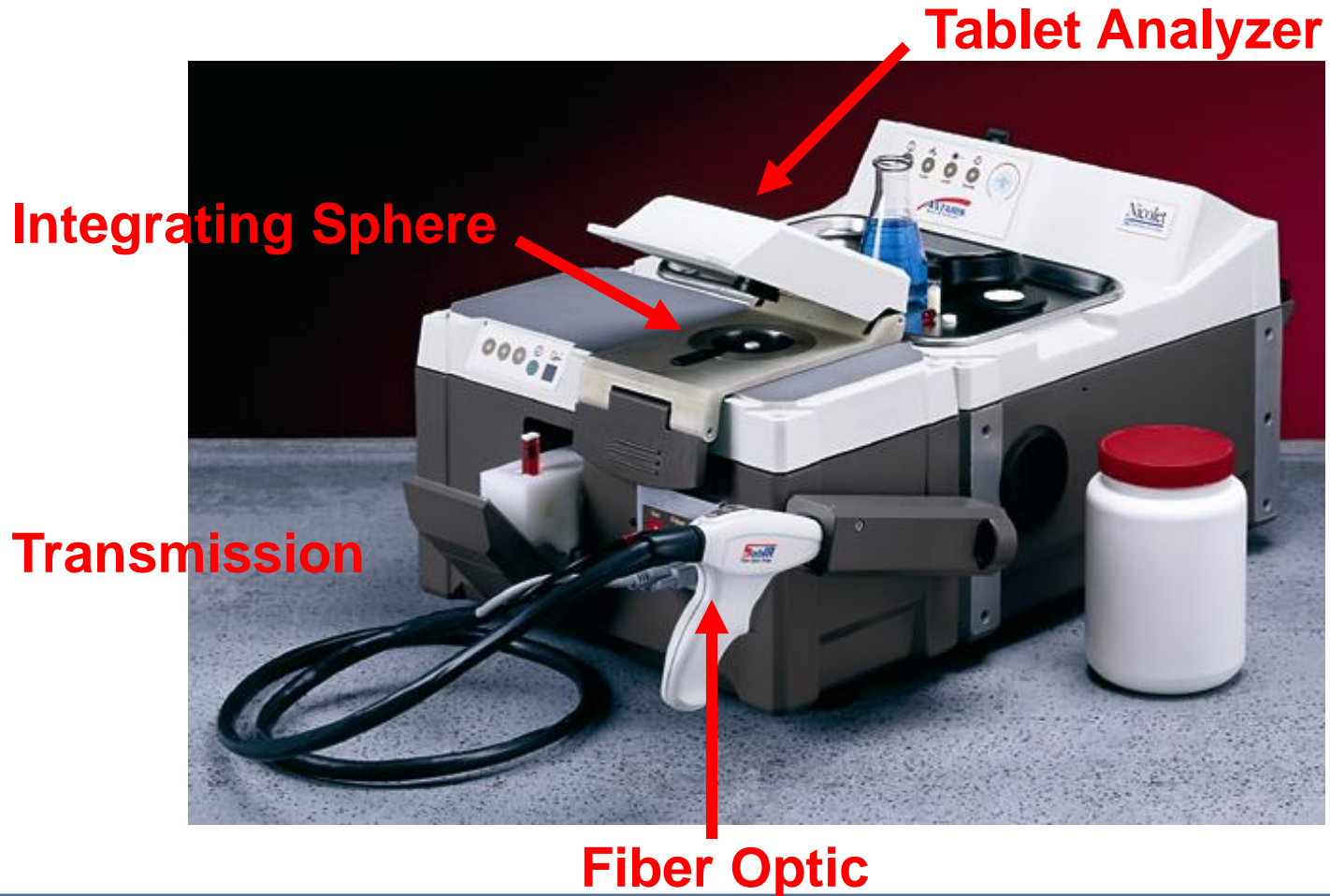
Nicolet iN10
microscope



Nicolet Antaris NIR Analyser



All Sampling on One Analyzer



Typical FT-NIR Sample Types

- Can measure all sample types

Solids

Softgels

Films

Liquids

Grains

Suspensions

Tablets

Pastes

Syrups

Powders

Pellets

- Analysis Types

- Quantification – Multiple Component %
- Identification
- Qualification – class segregation of compounds
- Determination of change – trending, control charts
- Process monitoring and control



Common FT-NIR Applications in Food and Agriculture

- Multi-component analysis of sweeteners and carbohydrates
- Fat, starch, moisture, protein and ash
- Food additives
- Fermentations
- Tobacco
- Feed and feed ingredient nutritional parameters



Food and Agriculture Industry Segments

Food and Agriculture Segments	Analysis by NIR
Confectionary	YES
Animal Feed and Ingredients	YES
Pet Food	YES
Flour	YES
Grains and grain milling	YES
Oilseeds and oilseed milling	YES
Meat	YES
Dairy	YES
Vegetable oil and fat	YES
Cereal	YES
Snack Food	YES
Baking and baking premixes	YES
Wine, beer and alcoholic beverages	YES

Antaris II DR and AuNIR pre-calibrations

- The Antaris II DR is the recommended instrument for use with AuNIR calibrations.
- AuNIR (Ingot) calibrations are issued as Unscrambler calibrations
- The Unscrambler software is not required. OLUP and/or OLUC are required.
- OLUC and OLUP are intermediary applications that allow a model created in The Unscrambler to be interfaced with RESULT
 - 840-180100 The On-Line Classifier for The Unscrambler (OLUC)
 - 840-180200 The On-Line Predictor for The Unscrambler (OLUP)
 - 840-180000 Complete Unscrambler Routine, OLUC and OLUP

AUNIR



[Forages](#)



[Animal by-products](#)



[Plant breeders](#)



[Speciality Markets](#)



AusScan Online

■ Giving you access to *in vivo* energy calibrations for poultry, swine and ruminants.

■ Introducing a calibration to measure reactive lysine in soya and canola.



[Animal Feed](#)



[Flour & Milling](#)



[Pet Food](#)



[Aqua Feed](#)



NIR Analysis in the Baking Industry

NIR Suitability for Analysis in the Baking Industry

- Moisture in dough is a critical parameter for controlling quality of bread
- Moisture analysis of bread needed for feedback control of the baking process
- Moisture analysis by NIR
 - Lowest detection limit of any compound
 - Accurate and precise for sample $<0.1\%$
- NIR analysis in baking industry
 - Raw material – Flour, Yeast
 - Starting material – Dough
 - Final Product - Bread





Quantitative Analysis of Fructose in Corn Syrup

Quantitative Analysis of Fructose in Corn Syrup

- Fructose corn syrup is used in the soft drink and food industries as a direct replacement for sucrose (table sugar) since it has a similar sweetness.
- Fructose corn syrup has many advantages over sucrose including longer shelf life, ease of transportation, ease of blending a liquid vs. a powder, and lower cost in areas where corn is plentiful.
- The most common grades of high fructose corn syrup (HFCS) contain 42% and 55% fructose.
- If the process for making HFCS is disrupted, even for a short period of time, thousands of pounds of out-of-specification product can be produced.

PLS Model for Fructose in Corn Syrup

- 47 corn syrup calibration spectra were collected on an Antaris II FT-NIR spectrometer using the temperature controlled transmission module
 - 1 mm pathlength cuvettes, 60 °C to mimic process conditions
 - HPLC primary analytical method
 - Concentration range 51 to 62% fructose
 - 4 cm⁻¹ resolution, averaging 50 scans (~40 sec)
- 10 validation spectra were collected under the same conditions
- Spectra were pre-processed with 1st derivative and Norris filter smoothing function
 - Segment length 15, gap between segments 0



FT-NIR Advantages for Analysis of Corn Syrup

- Replace HPLC analysis
 - Analysis time 10 minutes or more
 - Consumables and maintenance labor costs
- HPLC CANNOT be used to trend a production process in real-time
- Replace refractometer tests for Dry Substance or Brix
 - Temperature dependent
 - Subjective test by QA analyst
- FT-NIR is a very precise techniques allowing for easy detection of process upsets

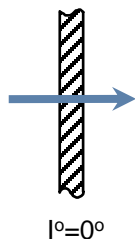


Problem Solving with FT-IR, NIR and Raman

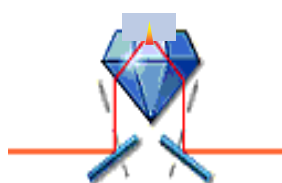
- What kind of sample do you have?
 - Solids
 - Coatings
 - Liquids
 - Gases
 - Mixtures
- How much sample?
 - Bulk
 - Particles
- Throughput?
 - Few samples
 - Many samples
- Do you need to identify or quantify?



Choosing the Optimal Sampling Method



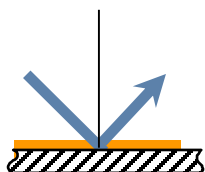
- **Transmission**
 - Preferred method for quantitative analysis
 - Sample mixed and pressed; good sensitivity



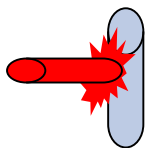
- **Attenuated total reflectance (ATR)**
 - The sample must contact the crystal
 - Simple and fast, generally non-destructive



- **Diffuse reflectance (DRIFTS)**
 - Dilute sample in a matrix (like KBr)
 - Simple, but requires mixing



- **Specular reflectance**
 - Sample must be reflective or on a mirror
 - Signals can be very weak



- **Raman Scattering**
 - Simple sample preparation – often none!
 - Useful in aqueous environments

Connect With Us!

- **Find us on the web:**

- Thermo Scientific FTIR product line: www.thermofisher.com/ftir
- Thermo Scientific FTIR microscopy product line: www.thermofisher.com/FTIRmicroscopes
- Thermo Scientific spectroscopy products: www.thermofisher.com/spectroscopy
- Spectroscopy videos: <https://www.youtube.com/thermoscientificspec>
- FTIR Spectroscopy Academy: www.thermofisher.com/FTIRAcademy

- On-demand Spectroscopy Webinars: www.thermofisher.com/spectroscopywebinars

Thank you