Theory and Utility of FT-NIR Spectroscopy

An Introduction to Nicolet Antaris FT-NIR Analyzers

Analyze • Detect • Measure • Control™

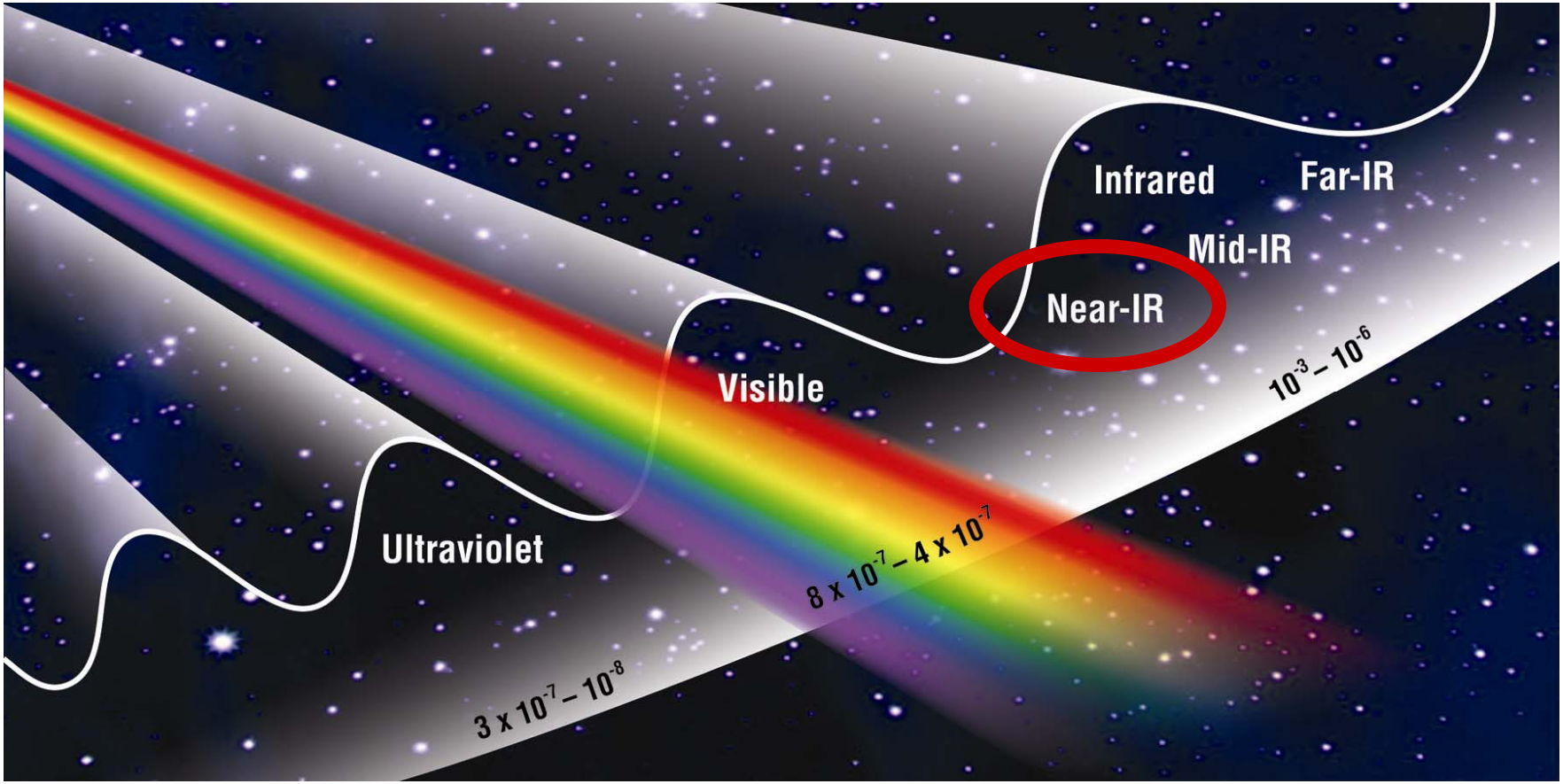
Thermo ELECTRON CORPORATION

MOLECULAR SPECTROSCOPY

The study of the interaction of electromagnetic waves and matter

(Includes UV, visible, NIR, IR, Raman etc.)

Transitions Guide to the EM Spectrum



SPECTRAL ABSORPTIONS

Radiowave	NMR
Microwave	Rotation of molecules, ESR
IR	Fundamental molecular vibrations
NIR	Overtones and combinations of Mid-IR
UV / Visible	Electronic transitions, energy of electrons raised to an excited state
X-Ray	Core electronic transitions in atom

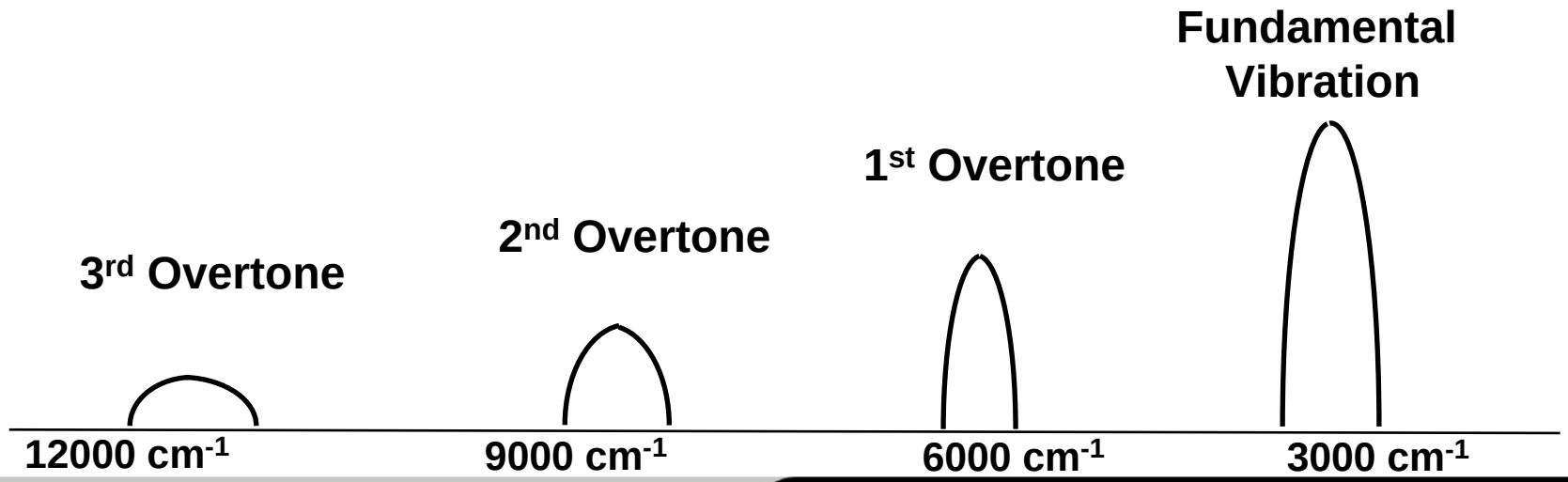
Vibrational Spectroscopic Regions

- Mid-IR Spectral Region
  - 4,000 - 400 cm^{-1} (wavenumber)
  - 2,500 - 25,000 nm (wavelength of light)
- NIR Spectral Region
  - 12,820 - 4,000 cm^{-1} (wavenumber)
  - 780 - 2,500 nm (wavelength of light)

To convert between nm and wavenumbers, simply take the inverse of the number in nm and multiply by 10^7. For example, to convert 1000 nm into wavenumbers, take 1000, invert it and multiply by 10^7. This gives 10,000 cm^{-1}.

What Information Comes From the NIR ?

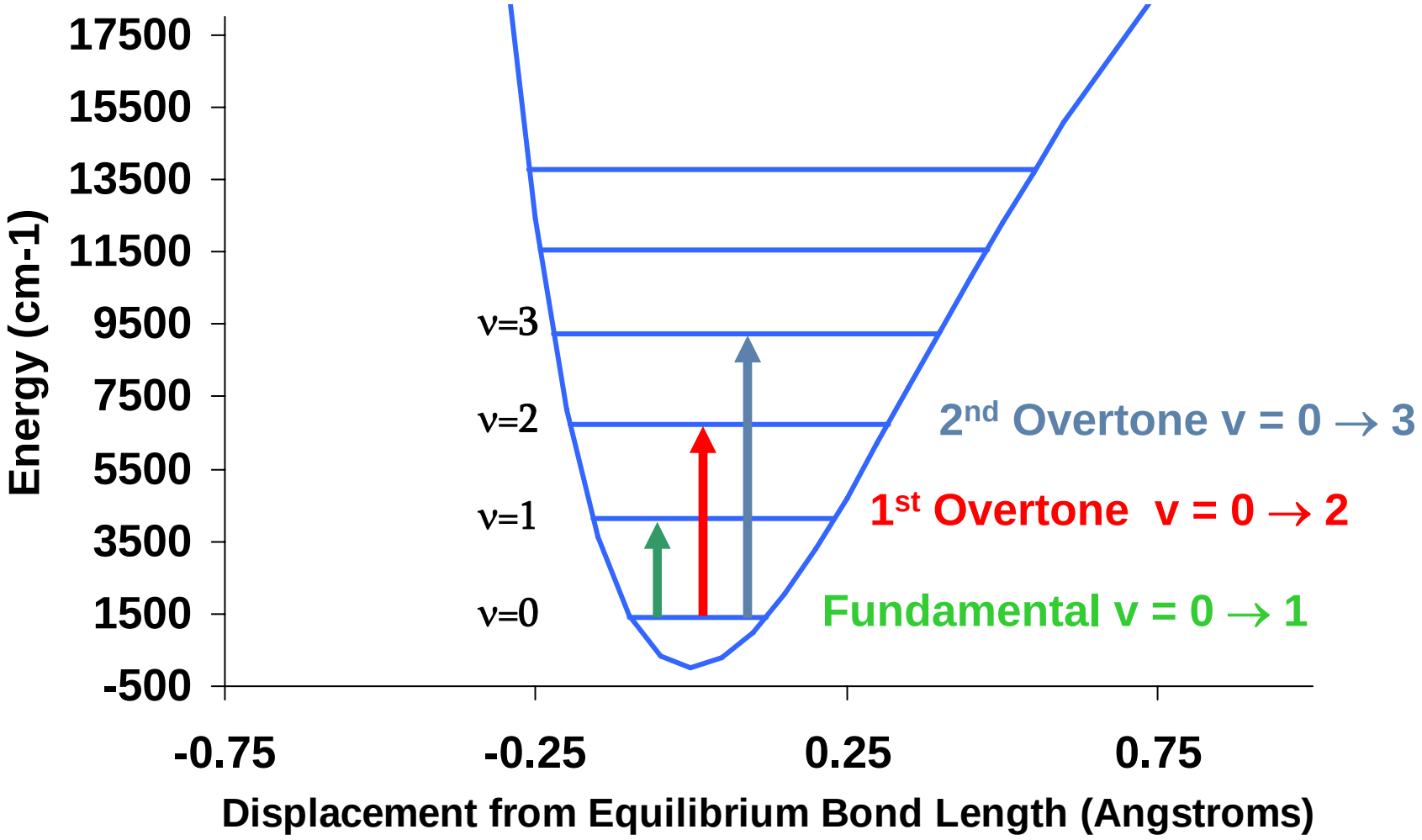
- Mid-IR spectroscopy makes use of *fundamental* vibrations
- Near-IR spectroscopy uses *Overtones* and *Combination Bands*
- Overtones and Combination Bands are types of vibrations related to the fundamental vibrations seen in the Mid-IR



Nature of Overtones

- Musical instruments like guitars and pianos make use of fundamentals and overtones just like in spectroscopy
- Overtones occur at about 2 and 3 times the frequency of the fundamental vibration. Absorption intensity decreases with increasing overtones
- Band overlap increases with increasing overtones

Fundamental and Overtones – A Quantum View



Overtones

Occur Close to
Integer Multiples Of Fundamental Bands.

For example: C-H Overtones Will Occur Near:

First Overtone

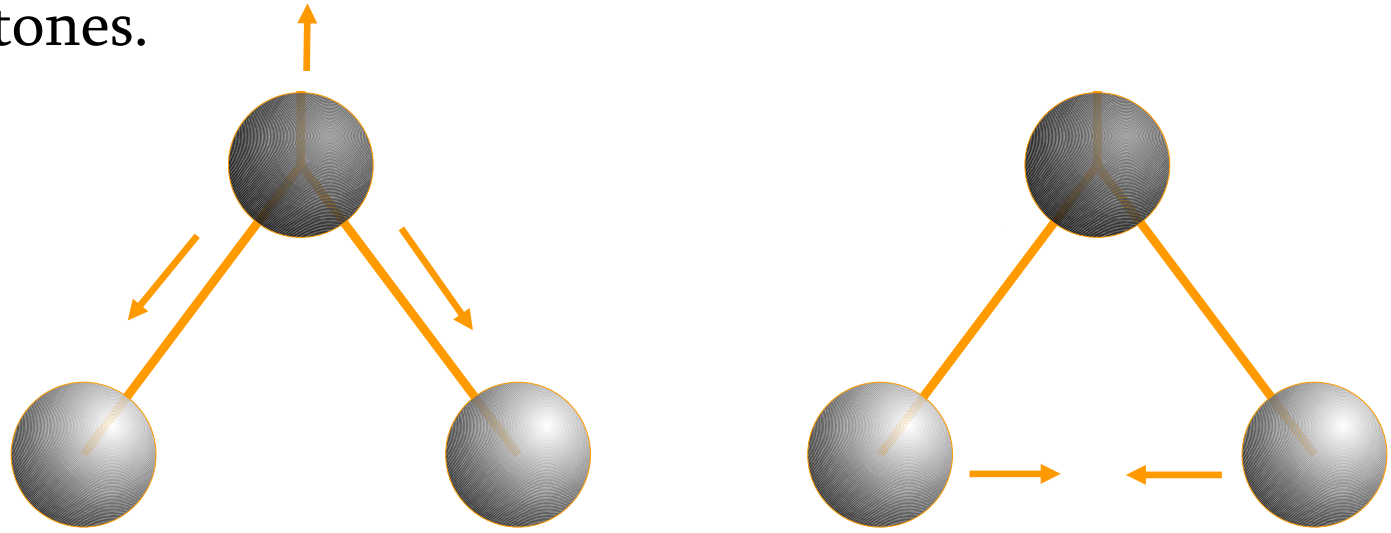
2960cm-1 (C-H Stretch) * 2 = 5920 cm^{-1}
3378 nm (C-H Stretch) * 1/2 = 1689 nm

Second Overtone

2960cm-1 (C-H Stretch) * 3 = 8880 cm^{-1}
3378 nm (C-H Stretch)* 1/3 = 1126 nm

Combination Bands

- There are vibrations in the Near-IR other than overtones of fundamentals. These are called Combination Bands.
- COMBINATION Bands are the sum of several fundamentals from **different** vibrations. One photon of light excites two vibrations.
- Combination Bands are typically found at lower energies than overtones.



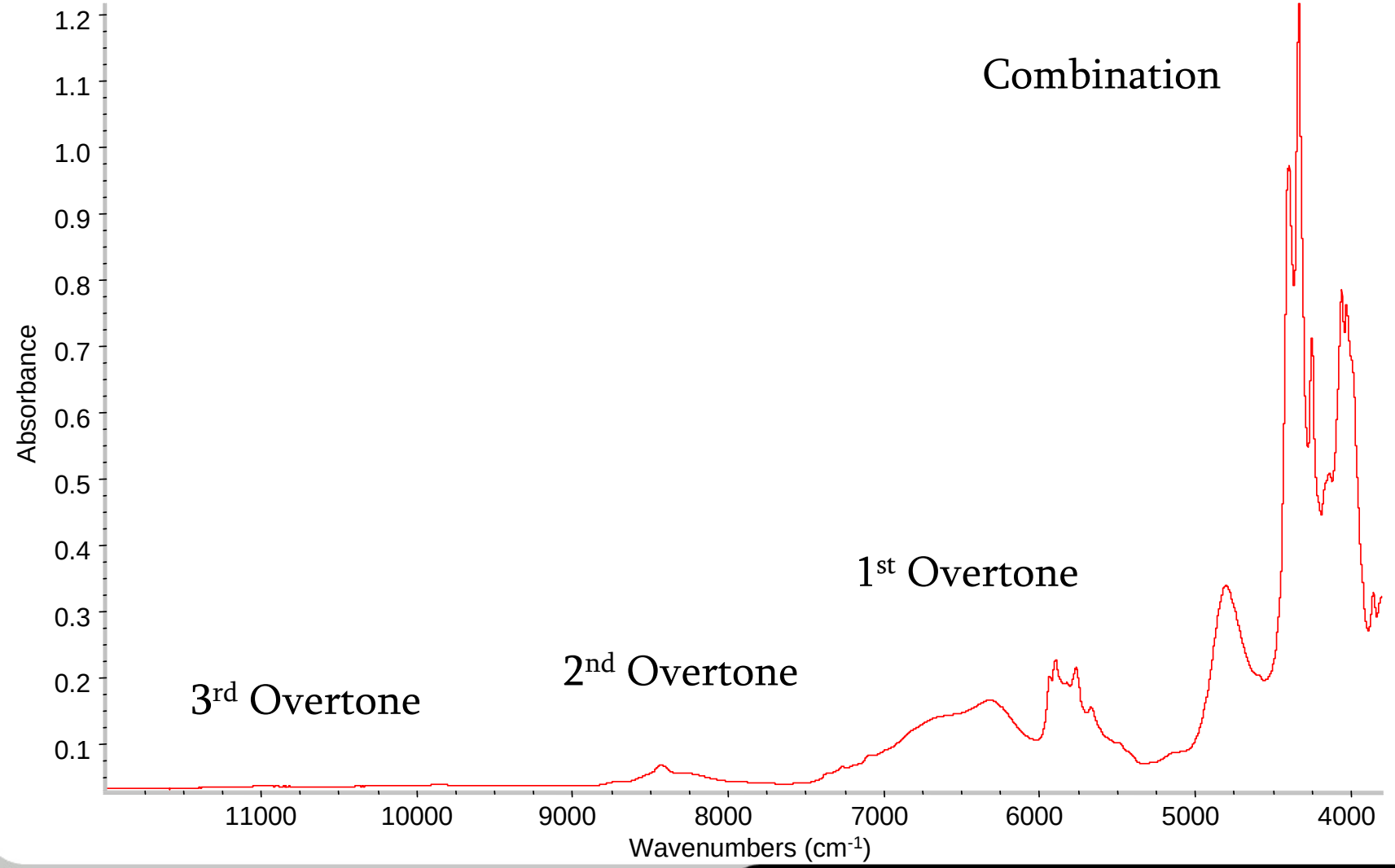
Combination Bands

COMBINATION Bands Appear Near The Sum Of Two Or Three FUNDAMENTAL Bands

For Example: A C-H Combination Will Occur Near…

2960cm^{-1} (C-H Stretch) + 1460cm^{-1} (C-H Bend)
= 4420cm^{-1}
= 2262nm

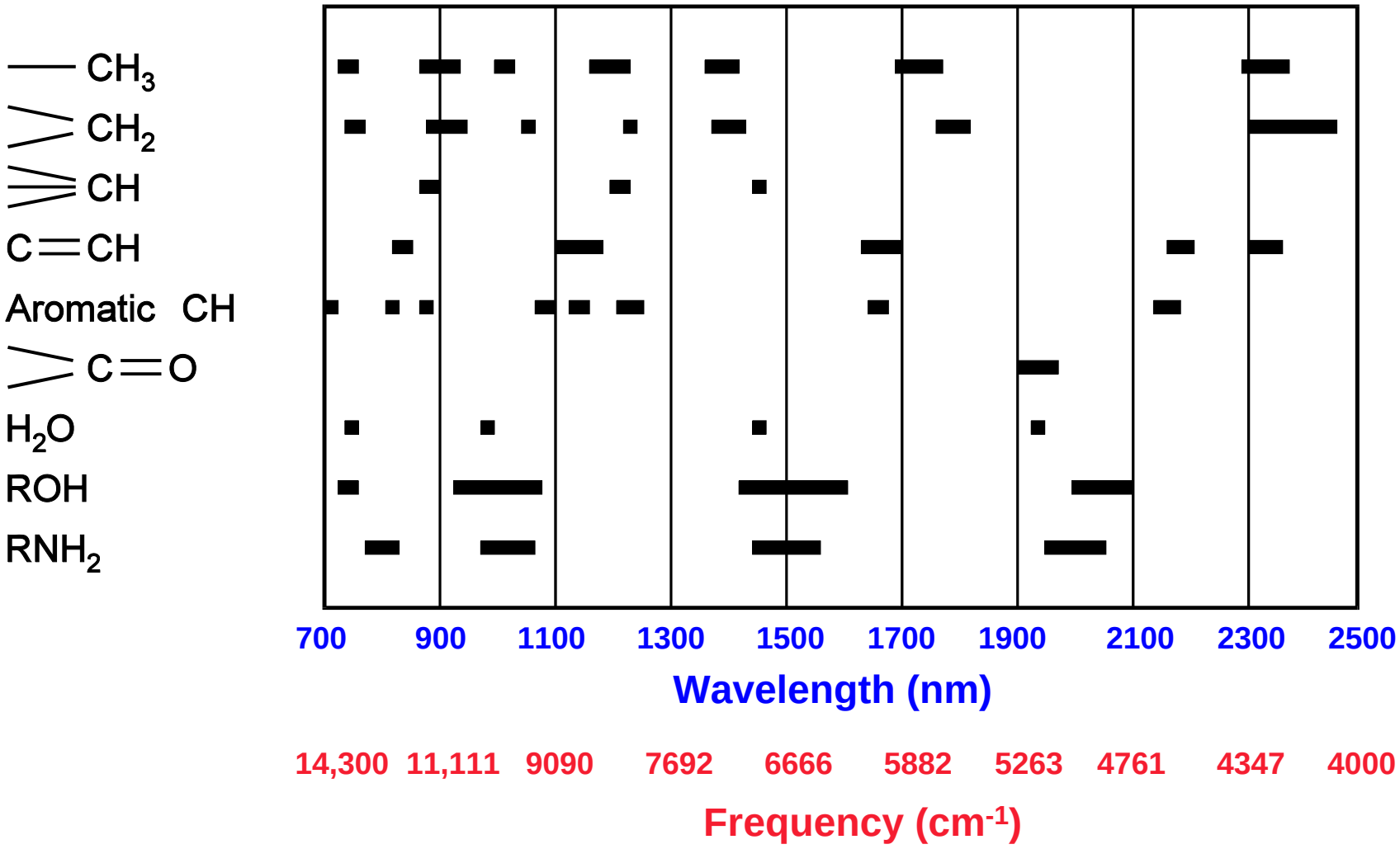
Example NIR Spectrum



NIR Characteristics

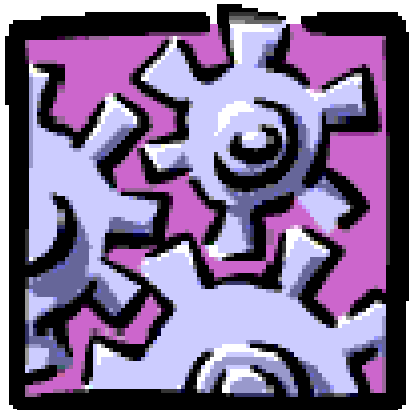
- R-H groups have the strongest overtones due to hydrogen's degree of anharmonicity.
- O-H, N-H, C-H, S-H bonds etc. strong NIR absorbers
- H_2 and O_2 has no change in dipole moment, hence no NIR absorption
- R-H stretch or R-H stretch/bend form most NIR bands

Typical Absorptions



Typical FT-NIR Measurements

- Qualitative and quantitative analysis
  - *Identification*
  - *Qualification and quantitation*
  - *Determination of change*
  - *Process monitoring and control*



- Can measure all sample types

Solids	*Softgels*	*Films*
Liquids	*Grains*	*Suspensions*
Tablets	*Pastes*	*Syrups*
Powders		

Uses for NIR Spectroscopy

- NIR spectroscopy finds widespread use as an excellent QA/QC analysis tool in industrial applications:
  - *Pharmaceutical*
  - *Chemical*
  - *Petrochemical*
  - *Food and Beverage*
  - *Polymer*
  - *Agriculture*
- NIR is a proven and popular technique because it is rapid, accurate, non-destructive and can go through glass and plastic to analyze the material inside

The FT-NIR Analysis Advantage

- Large pathlength
  - *Analyze more sample*
- Sampling *in situ* - through glass, packaging materials
  - *No sample preparation required*
- Accurate and precise
- Fast - Results in seconds
- No Hazardous reagents
- Non-destructive
- Remote sampling capabilities

Regulatory Acceptance for Near IR

U.S. Pharmacopeia
The Standard of Quality℠

- ASTM NIR Method E 1944
- USP Chapter <1119>
- European Pharmacopeia (Ph.Eur.) 2.2.40 1997
  (European Directorate for the Quality of Medicines)

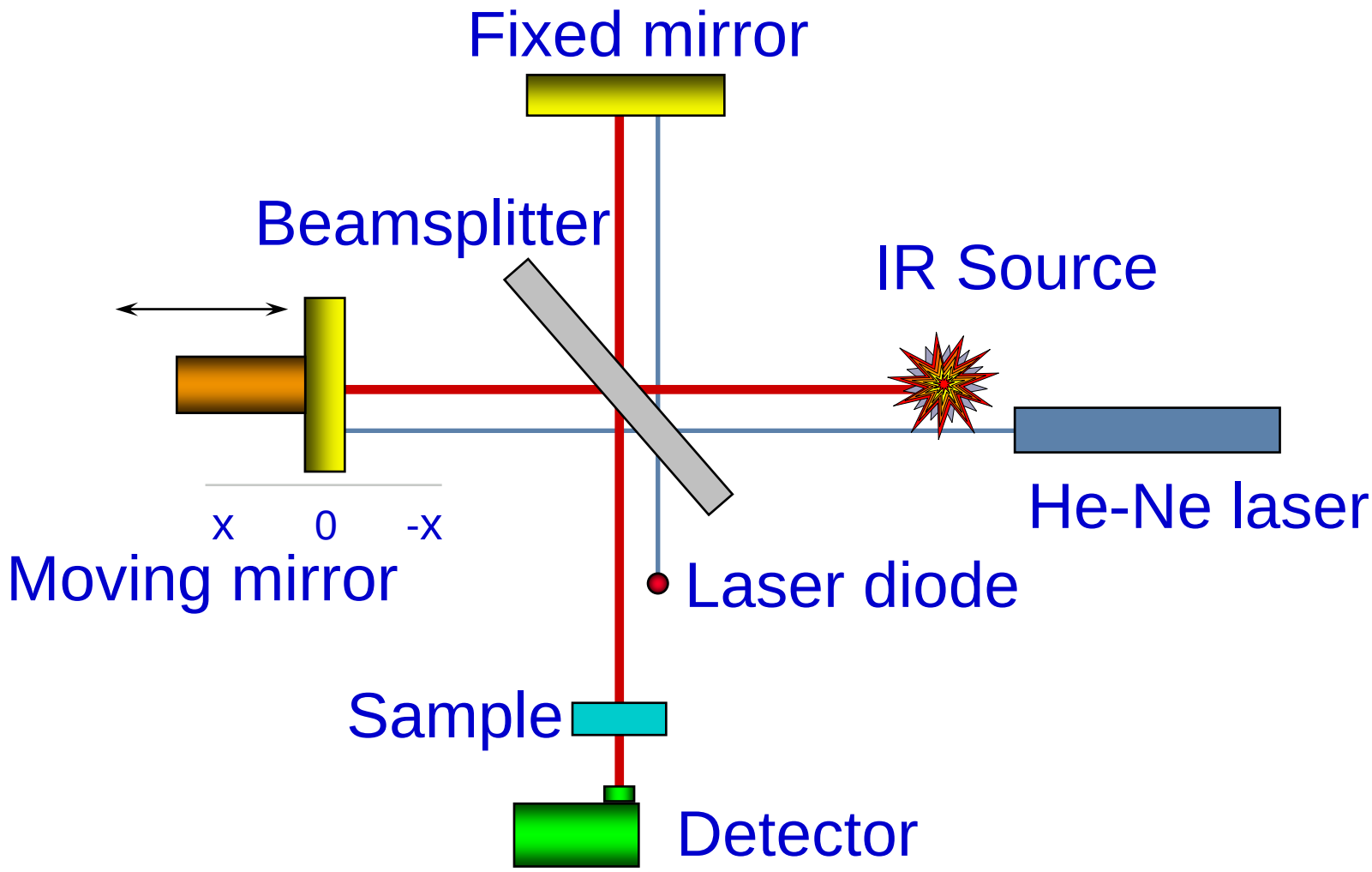
Types of Near IR Instrumentation

- Filter – Wavelength filters used to select specific wavelengths for analysis
- Dispersive – Gratings or prisms in combination with slits
- Fourier Transform (Nicolet Antaris) – Interferometer and internal HeNe laser

Nicolet Antaris™ FT-NIR Analyzer

- A complete analyzer for the pharmaceutical, food & beverage, chemical, and polymer industries

FT-NIR Spectrometer

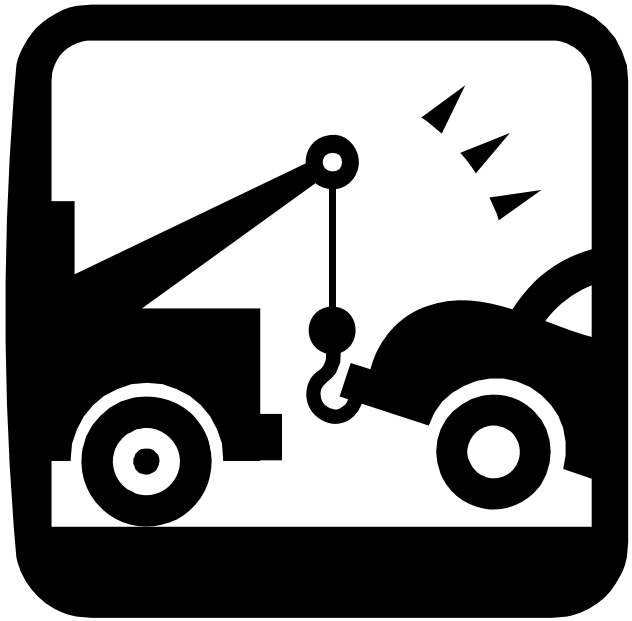


FT Technology for Near Infrared

- Based on the same interferometer technology used for mid-infrared and FT-Raman
- Utilizes all the advantages of FT based instruments
  - *Internal calibration is derived from He-Ne laser (precision = 0.01 cm^{-1})*
  - *High resolution (4 cm^{-1})*
  - *All wavelengths are measured simultaneously*
  - *Higher energy throughput (larger apertures)*
  - *Excellent stability and reproducibility*
  - *Dynamic alignment*

Limitation of Old Dispersive Technology

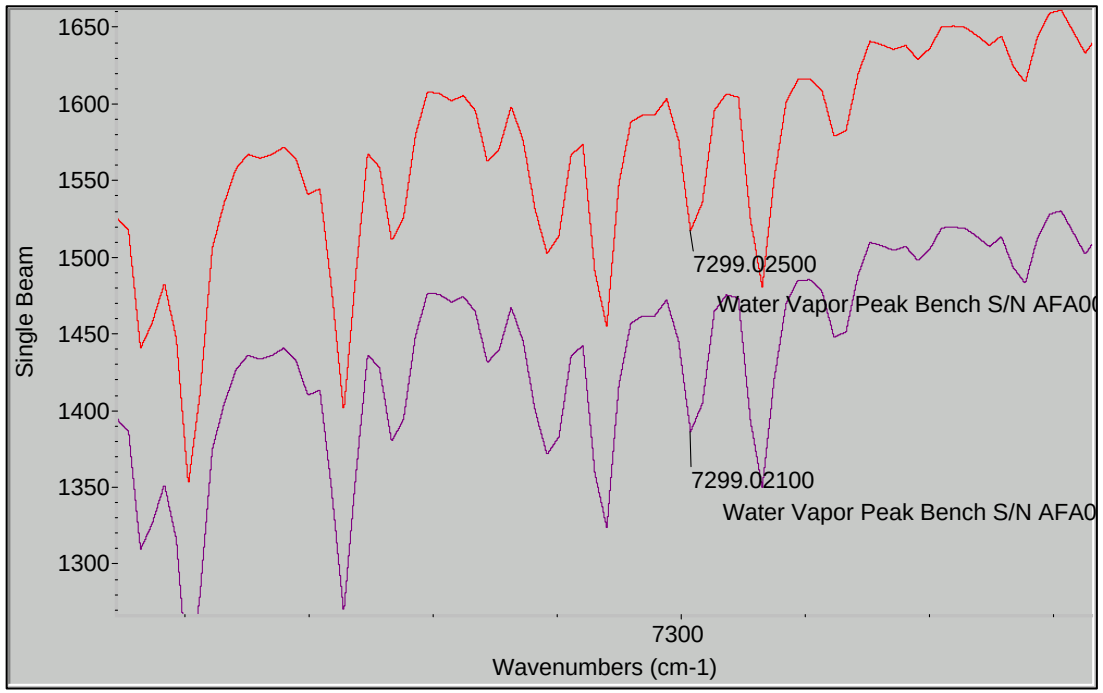
- Mechanically complicated
  - Grating filter and chopper
- Poor resolution
  - 16 cm^{-1} or worse
  - Loss of spectral information
- Wavelength inaccuracy
  - Due to prism or grating
- Stray light effects
- Difficult to transfer methods due to wavelength inaccuracy



Traditional NIR Problems

Poor Wavelength Accuracy and Precision

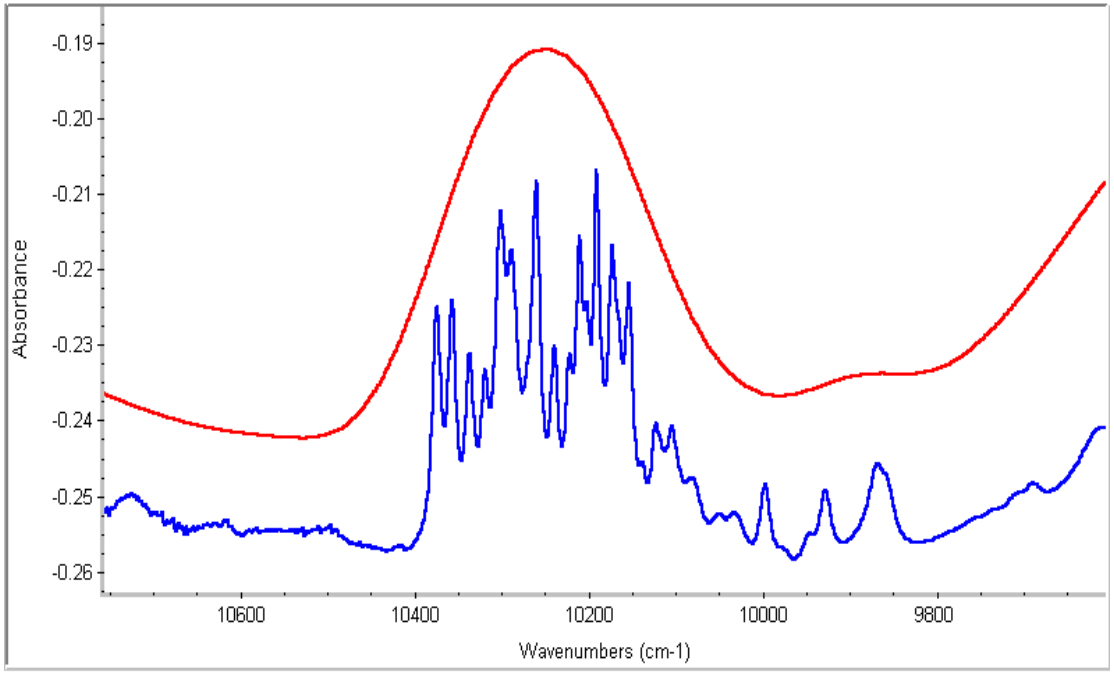
- FT-NIR advantage: data repeatability and method reliability due to superior wavelength accuracy and precision
  - *Internal frequency reference with HeNe laser*



Traditional NIR Problems

Low Resolution

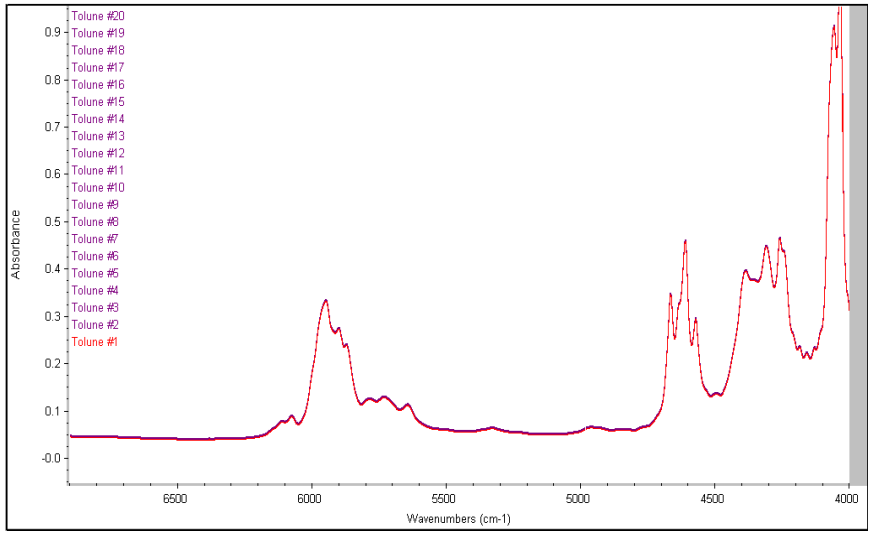
- Information can be hidden at lower resolution
  - *Need resolution to distinguish two closely related materials*
- FT-NIR advantage – simpler models with fewer calibration spectra possible with greater specificity

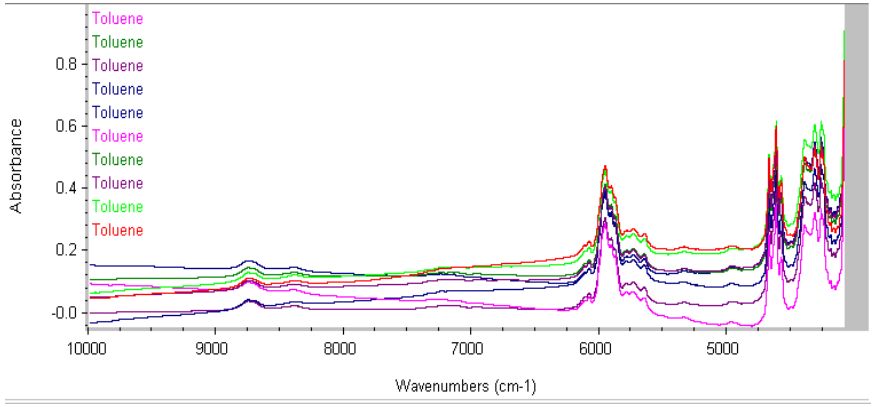


Traditional NIR Problems

Poor System Stability and Artifacts

- Stability – consistency in measurement results
- Artifacts – peaks or other anomalies in the spectrum not due to the sample
- Problems eliminated with Nicolet Antaris design





Sampling with an FT-NIR Analyzer

Analyze • Detect • Measure • Control™

Thermo
ELECTRON CORPORATION

Measurement Techniques

- Transmission
  - *Liquid analysis*
    - Quantitative
    - Qualitative
  - *Films*
  - *Tablets*
  - *Semi-solids*

- Diffuse reflectance
  - *Powders*
  - *Solids*
  - *Tablets*
  - *Fibers*
  - *Pastes*
  - *Suspensions with high solids contents*
  - *Opaque semi-solids*

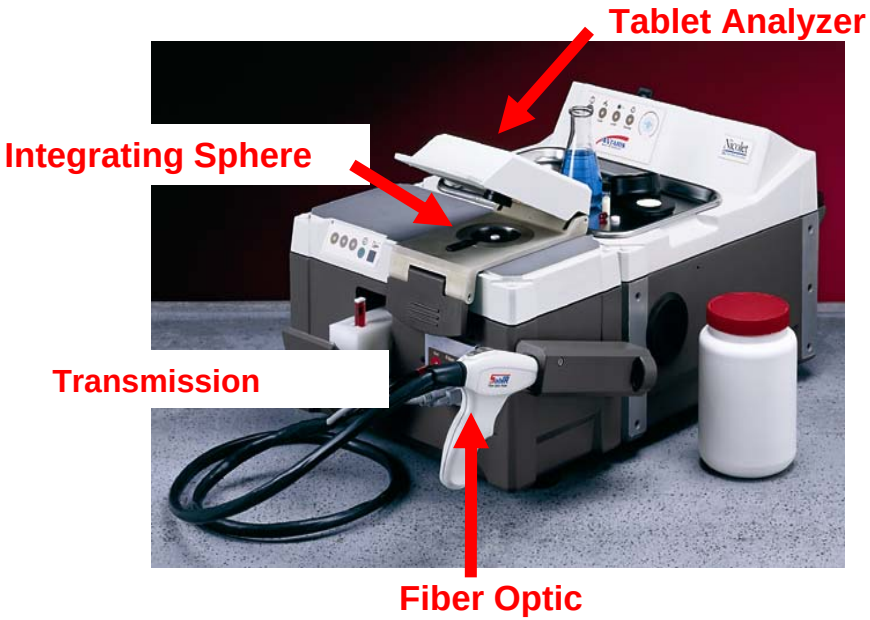
Nicolet Antaris FT-NIR Analyzer - Overview

Method Development Sampling System (MDS) shown above

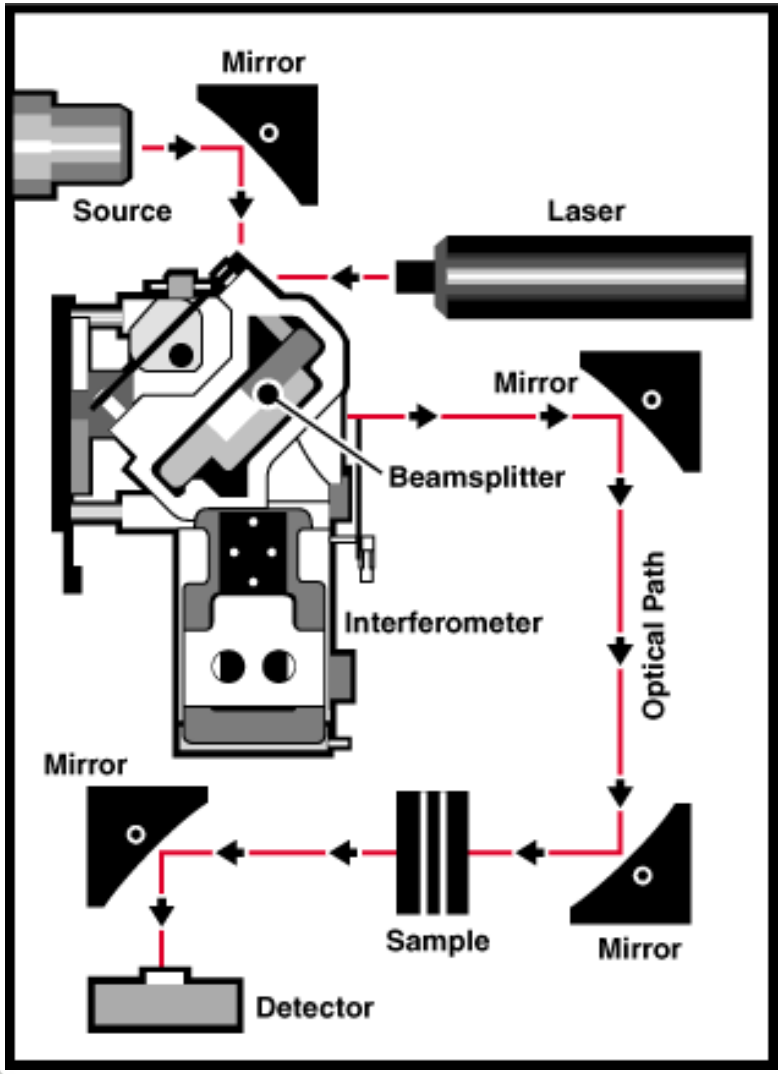
Dedicated configurations also available for specific analyses

Nicolet Antaris FT-NIR Analyzers

- MDS System
  - *All techniques on one system*
- Other Dedicated Systems
  - *RTS System*
  - *Liquid Analyzer*
  - *Solid Sampling System*
  - *Tablet Analyzer*
  - *Fiber Optic System*
  - *Fiber Optic Multiplexer*



Nicolet Antaris Optical Bench



- Rugged industrial platform
- Dynamic Alignment
- User-replaceable and pre-aligned consumables
- Modular design flexibility with stability of fixed beampaths
- Integrated validation (Built to meet or exceed USP monograph)

Near IR Quartz Halogen Source

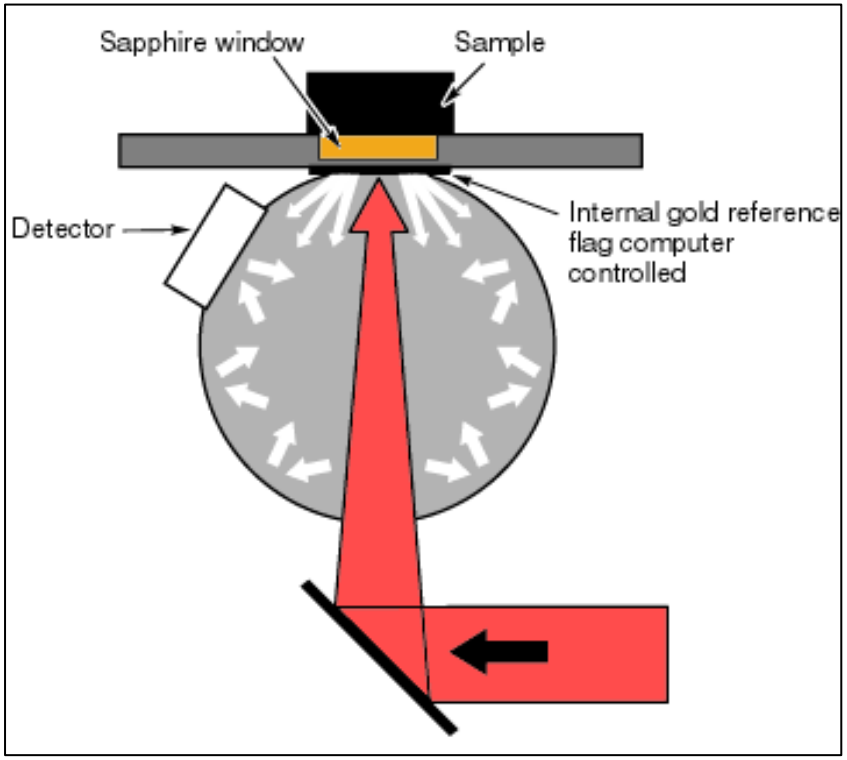
- External replacement with only 3 screws
- No wires
- Source change in seconds
- Pinned-in-place aluminum housing for lock-and-key fit
- High throughput

Internal Referencing For All Collection Types

- Protected internal reference material simplifies background collection
  - *Spectralon*
  - *Diffuse Gold Flag*
- Eliminates errors due damaged reference materials
- Collect backgrounds with sample already loaded
- Easy to use and automated

Reflection Sampling – Integrating Sphere

- Ideal collection environment for diffusely reflected light
- High efficiency collection
- Internal gold background material
- Sample simply needs to be placed on the instrument for data collection

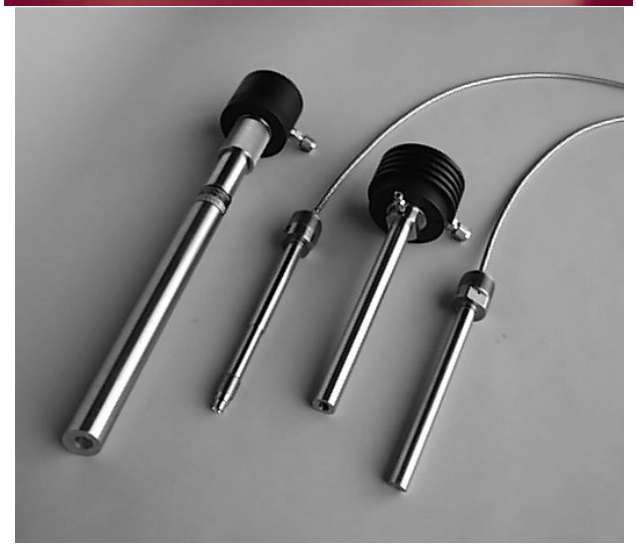


Liquid Sampling - Transmission Compartment

- Collect data on liquid, film or paper
- Accommodates vials, tubes, cuvettes and card holders
- Temperature control
- Automatic internal background position in transmission cell
- Reproducible positioning

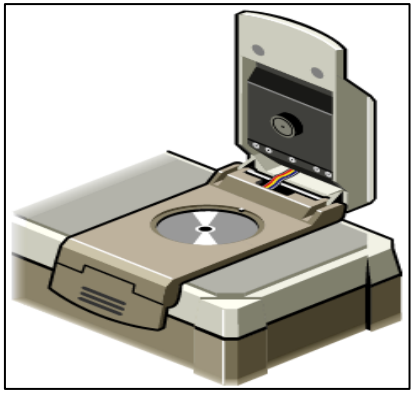
Fiber Optic Sampling – SabIR and 3rd Party Probes

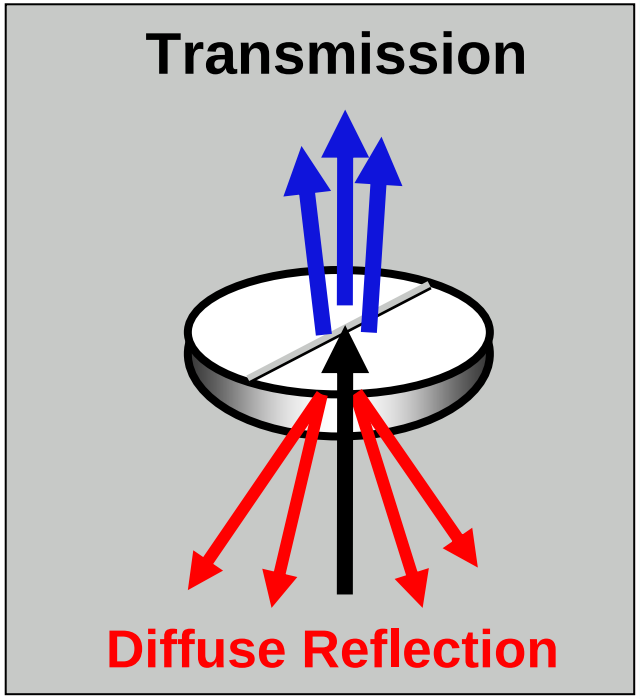
- Transflectance or reflection sampling
- Fiber optic port uses standard SMA connectors
- Remote sampling capability for At-line, near-line, on-line
- Ideal for incoming raw material identification and in-process analysis



Solid Transmission Sampling – Tablet Analyzer

- Thick, opaque tablets give quantitative results for
  - *Content uniformity*
  - *API or excipient concentrations*
  - *Physical properties like hardness*
- Integrating sphere reflection data gives tablet coating information
- Collect reflection and transmission data without removing the tablet
- Dedicated softgel detector





Antaris II

- Method Development System - All sampling types for NIR
  - *Transmission, tablets, softgels, diffuse reflection, fiber optics*
  - *Automatic sampling of solid dosage forms - 40 tablets at once*
  - *Simultaneous reflection and transmission measurement of tablets*
- Configurable base system with selection of sampling modules

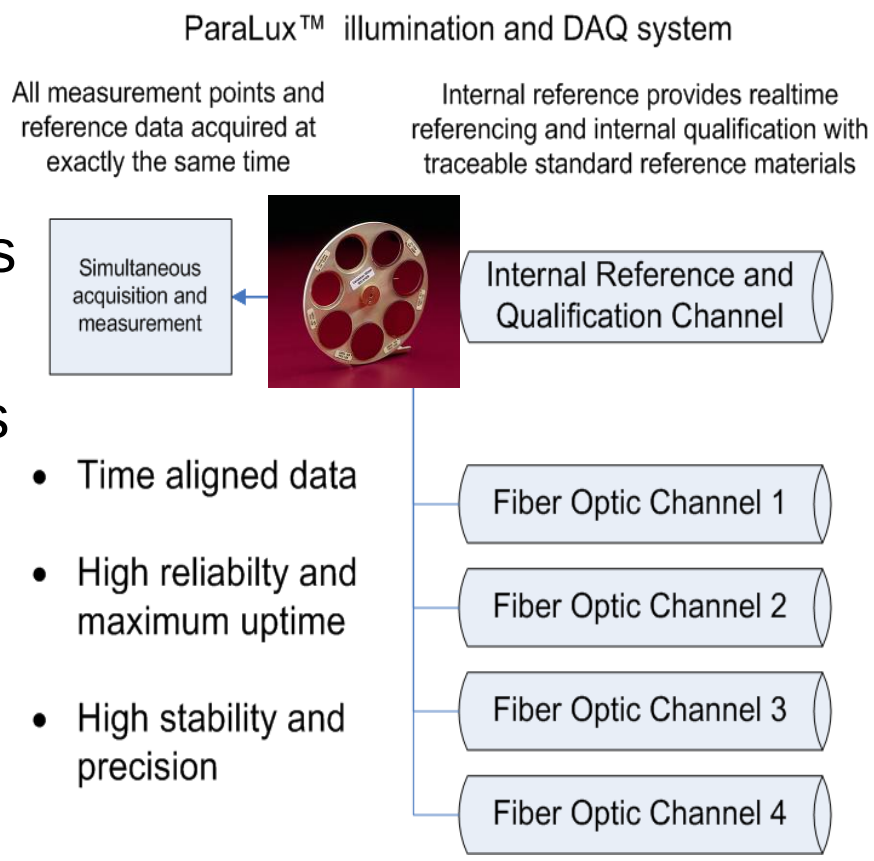
Remote Sampling – Fiber Optics – Antaris MX

- Simultaneous multi-channel measurement
- Simultaneous internal backgrounds
- No mechanical multiplexing
- Dual trigger probe capability
- Programmable analog/digital I/O options with RESULT Interface
- Spectral corrections adjust for fiber optic differences

Antaris MX

Analyzer performance verification

- Traditionally done with probe pulled off process
- Internal reference channel can qualify operation while probe is inline
- Traceable reference standards for spectroscopic performance checks according to USP <1119>

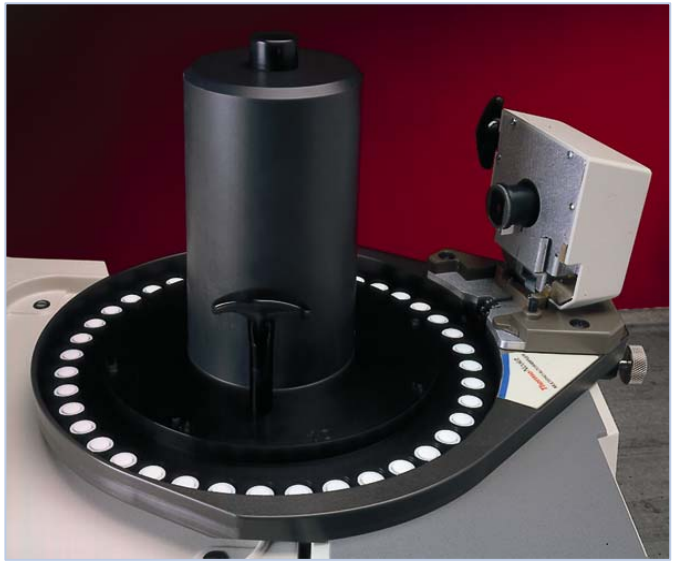


Fiber optic probes

- High quality probes with proprietary window mounts in custom configurations
- Specialty probes
  - *Fermentation – dual mode reflection and transmission*
  - *Retractable dryer probe with self cleaning tip and wash chamber*

High Throughput Sampling – Autosampler RS and Multipro Autosampler

- Multiple samples with no operator interaction
- Eliminates sample-to-sample operator error
- Useful for analyzing tablets, softgels, solids or powders in vials
- Transfer already existing methods for seamless scale-up



Heterogeneous Sampling – Sample Spinners

- Obtain spectra of heterogeneous samples like

  Polymer pellets

  Coarse powders

  Granular solids

  Crystalline materials

  Grains

  Silage

  Corn

- Eliminate the need to collect multiple spectra on the same sample

Antaris EX: A complete process analyzer

- Hazardous environments
- Integrated computing
- Integrated process communications
- Same sampling technology and advantages as MX
  - *Simultaneous measurements with internal referencing and qualification*
- Not a box that you have to put inside of another box!
- Complete new lineup of probes

Hazardous environments

- Three EX models:
  - *EX-Z1 for Class I Division 1/Zone 1 Environments*
  - *EX-Z2 for Class I Division 2/Zone 2 Environments*
  - *EX-IP65 for*
- Enclosures are:
  - *Hose washable, Stainless steel, corrosion proof*
  - *Individually certified*
- Vortex cooling provides operating temperature range of -20 to 40 C
- Purge systems (different for each environment) linked to power of system

On Blender Analysis

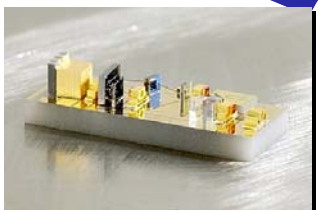
Blending in Development – Multiple sizes

- Instrumentation must work with a wide range of blender sizes
- Issues of scale with equipment size and weight

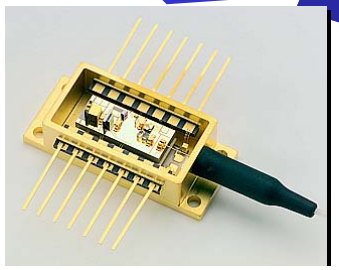
Monitoring the Blend with NIR

- By scanning the sample (pharmaceutical powder blend) through a window in the modified lid, the progression of mixing and concentration of components can be measured
- Data is triggered when bin is upside down (powder in contact with sampling window)
- Powered by rechargeable battery pack
- Diffuse reflection sampling
- Wireless communications to remote PC with RESULT software for control/DAQ
- Standard deviation measurement can be used to determine when mix is fully achieved

The Spectroscopy Technology



Optical Bench



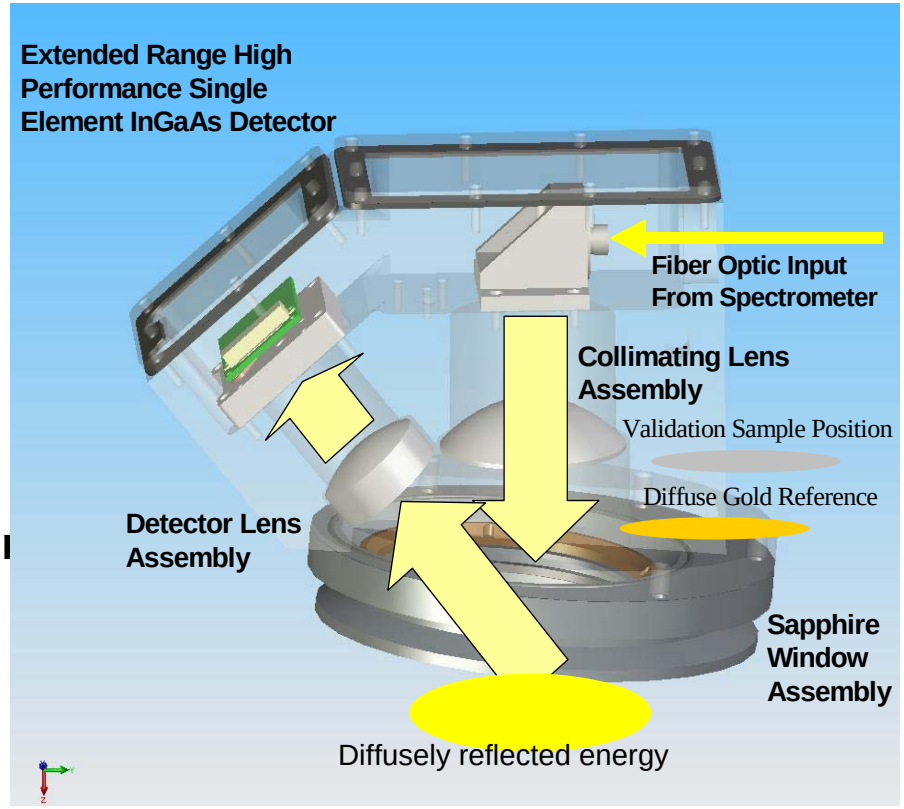
Optical Module Sub-Assembly

Spectrometer Assembly

- Source – Semiconductor based NIR tunable laser
  - *Not a light bulb/globar – does not go "out" or fade*
  - *Rated for a 25 year lifetime (Telcordia Spec.)*
- Wavelength selection - High resolution (2 nm / 8cm-1)
- Fabry-Perot tunable filter
  - *Tuning range of hundreds of nm*
  - *Scans full range in tens of milliseconds*
  - *Insensitive to vibration*
- Internal Wavelength and Amplitude Reference
  - *Ensures optimum wavelength calibration over time*

Instrumentation and Sampling Interface

- **Detector: Single element InGaAs photodiode**
  - **Optimized detector element sized to collect highest solid angle for maximum efficiency**
- **Baseline s/n of 0.0002**
- **Wavelength ranges available**
  - **1350-1550 nm, 1550-1800 nm and 1800 – 1970 nm**
- **Fast Scanning (~80 ms / scan)**
- **Sample sensing area of 40 mm (coincides with ~600 mg sample size)**
- **Smaller spot size available (~10 mm) for "intra-dosage" uniformity investigation**
- **Wireless communication (~15 meters)**
- **Battery operation (2 – 3 hours)**



Summary

- On Blender Real-time analysis provides a rapid and calibration free method of getting blend information and performing experiments
- With the correct type of optical interface the instrumentation can be quickly moved between blender sizes and types for scale-up development
- Instrumentation is process ‘friendly’
  - *Small*
  - *Lightweight*
  - *Easy to set up*
  - *Long battery life*
  - *Robust*

RESULT Software Suite for the Nicolet Antaris Analyzer

Analyze • Detect • Measure • Control™

Thermo
ELECTRON CORPORATION

RESULT Software Suite for Near IR

- RESULT is the software package made for collecting Near IR data with the Nicolet Antaris Analyzer

- Provides
  - *Real time data acquisition*
  - *Sample prediction using developed models*
  - *Chemometrics tools for method development*
  - *Full suite of compliance tools*

RESULT Provides Tools for all Users

- RESULT Operation allows users with no science training to run routine analyses
  - *Minimal training time and one-click use*

- RESULT Integration provides the tools to develop workflows with integrated SOPs for workroom deployment
  - *Design simple workflows like Raw Material ID*
  - *Create sophisticated experiments with logical gates, delays and repeats*

RESULT Provides Tools for all Users

- TQ Analyst is a powerful chemometrics package
  - *Easy to use*
  - *Fully integrated with spectral collection software*
  - *No secondary programs needed*
- Administrator Mode
  - *Creates new users*
  - *Maintains system integrity*
  - *Sets workflow access privileges for all users*

ValPro Instrument Qualification Platform

- Design qualification (DQ) documentation provided
- Detailed Installation Qualification (IQ) procedure
- USP-based Operational Qualification (OQ) tests provided
  - *Traceable & serialized internal validation wheel*
- Tools for Performance Qualification (PQ)

21 CFR Part 11 Compliance with RESULT

- Full Suite of Part 11 Tools
  - *Full operation auditing*
  - *SOP enforcement and automation*
  - *Digital signatures*
  - *Security, access control, and privilege and workflow assignment*

Key ID	Date	PC	Operator Name	Workflow Name	File Name	File Type	Item Name	Run Mode
275	08-24-2000 08:23:29	PC_DRAPER	Draper, Carla	Precision Constraint	C:\RESULT Data\Archive\precision 51731739-79C1-11D4-A31E-0050DA5D91D9.htm	Report	statistics report	Production
273	08-24-2000 08:22:21	PC_DRAPER	Draper, Carla	Precision Constraint	C:\RESULT Data\Archive\ethanol\ethanol 51731736-79C1-11D4-A31E-0050DA5D91D9.spa	Spectra	precision test	Production
272	08-24-2000 08:22:20	PC_DRAPER	Draper, Carla	Precision Constraint	C:\RESULT Data\Archive\ethanol\ethanol 51731735-79C1-11D4-A31E-0050DA5D91D9.spa	Spectra	precision test	Production
271	08-24-2000 08:22:20	PC_DRAPER	Draper, Carla	Precision Constraint	C:\RESULT Data\Archive\ethanol\ethanol 51731734-79C1-11D4-A31E-0050DA5D91D9.spa	Spectra	precision test	Production
270	08-24-2000 08:22:20	PC_DRAPER	Draper, Carla	Precision Constraint	C:\RESULT Data\Archive\ethanol\ethanol 51731733-79C1-11D4-A31E-0050DA5D91D9.spa	Spectra	precision test	Production
269	08-24-2000 08:22:19	PC_DRAPER	Draper, Carla	Precision Constraint	C:\RESULT Data\Archive\ethanol\ethanol 51731732-79C1-11D4-A31E-	Spectra	precision test	Production

Software Validation

- TQ Analyst Algorithms
  - *Fully Documented*
  - *Externally reproduced for verification in Excel*
  - *Demonstrates internal validation*

Calibration

The following steps are performed in a PCR calibration for dimensions $h = 1, 2, \ldots, a$:

1. Initialize a matrix $\mathbf{U}$ that has r rows and has $h = 0$ columns.
2. Increase h by 1 and select the column of $\mathbf{X}$ (actually $\mathbf{X_h}$ since it depends on the value of h) with the greatest sum of squares. This is a first estimate for the principal component scores (or latent variables). Call this vector $\mathbf{u_h}$. $\mathbf{u_h}$ is of size r x 1.
3. Compute the squared norm of $\mathbf{u_h}$.

   $$u_h^2 = \mathbf{u_h}' \mathbf{u_h}$$

4. Calculate the row vector $\mathbf{b_h}$ as $\mathbf{b_h}' = \mathbf{u_h}'\mathbf{X} / u_h^2$. $\mathbf{b_h}$ is of size p x 1.

Process Analytical Technology (PAT)

- PAT is a system for designing, analyzing, and controlling manufacturing through timely measurements (i.e., during processing) of critical quality and performance attributes of raw and in-process materials and processes with the goal of ensuring final product quality
- Seamless migration of FT-NIR technology from the laboratory to the manufacturing floor

Time for a Paradigm Shift - PAT

- In-process testing that currently exists in pharmaceutical manufacturing consists of
  - *Physical measurements*
  - *Results from samples sent to the lab while processes were halted or continued "blindly"*
- Current testing is not real-time, nor "whole batch"
- Non-optimal conditions can still occur (over-blending for example)
- Testing and waiting for results is costly

Typical FT-NIR PAT Applications

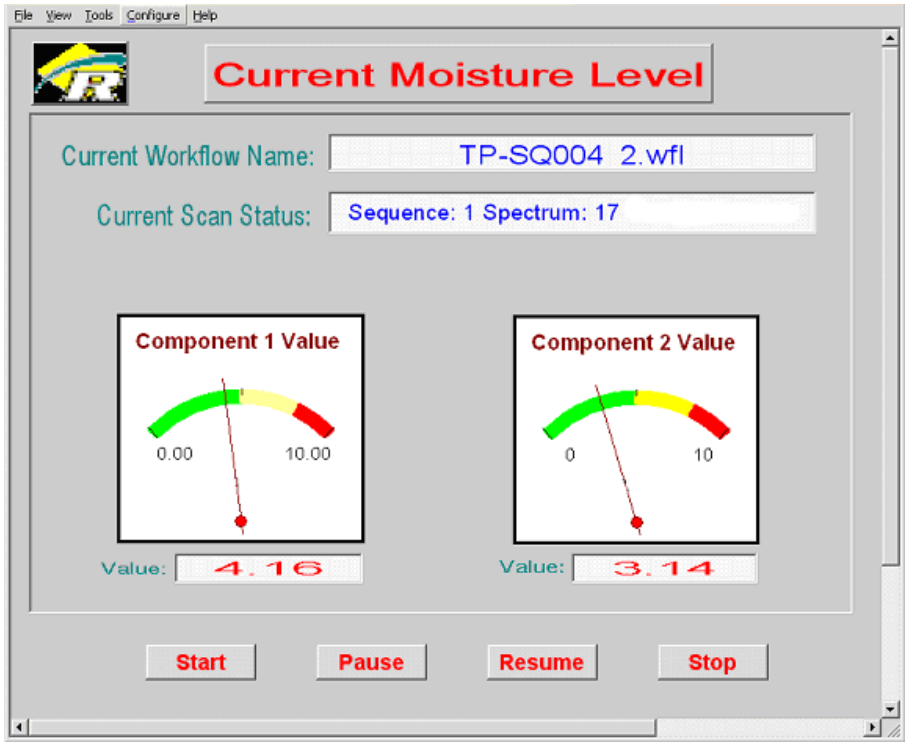
- Blending and mixing
- Reaction monitoring
- Fermentation
- Drying
- Post tableting tests

NIR in the Manufacturing Process

- Traditionally, optimal mixing time was determined during process development and scale-up
- Avoids problems of incomplete or over-blending (de-blending)
- Improves efficiency of production processes and equipment usage
- Assures product uniformity
- Goals
  - *Establish adequacy of mixing*
  - *Eliminate thieving/process interference*
  - *Eliminate error and recipe approach*
  - *Accurate endpoint determination*

Integration with manufacturing systems

- RESULT OPC (OLE for Process Control) Server facilitates powerful communication with manufacturing systems
- Send and receive commands automatically
- Trigger runs, endpoints, valves, etc.

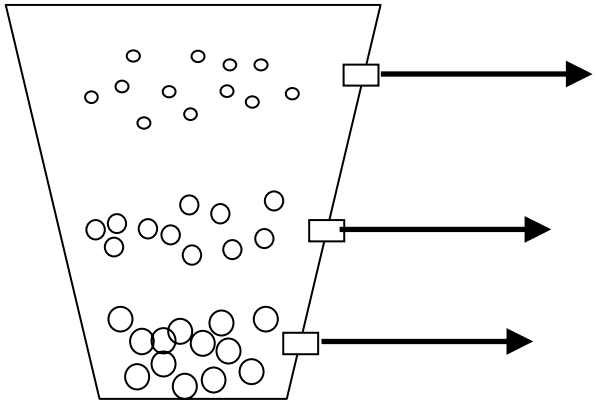


Communication is Essential to Process Control

- Communication system must be able to bridge
  - *Disparate technologies from various manufacturers*
  - *Compatibility with both high and low level interfaces*
  - *Opposing computer platforms*
  - *Geographic separation from target*
  - *Interface with business software to expand the process control loop*
- Data communication architecture must be inclusive, not exclusive
- Existing and modern facilities

OPC in Process: Dryer Monitor

Dryer Data Collection → Analyzer Data Processing / OPC Server Output → SCADA or DCS OPC Input



Typical FT-NIR Pharmaceutical Applications

- Raw Material ID
- Moisture Content
- Content Uniformity
- Tablet API
- Coating Thickness
- Process Analytical Technology (PAT)

Pharmaceutical Application: Raw material ID

- Apply NIR to inspect incoming raw materials quickly at the loading dock
- Equip the receiving area with an NIR to be used by technicians who are not trained as scientists
- Confirm the identity of each container of material that is received, without lab wet chemistry techniques that are time-consuming and expensive

Library Raw Material Samples

- One lot each of the following substances was provided for the construction of the raw material library

d-Glucose
d-Fructose
Sucrose
d-Mannitol
d-Sorbitol
α-d-Lactose Monohydrate
Acetylsalicylic Acid
Acetaminophen
L-Ascorbic Acid
Citric Acid

Validation Samples

d-Glucose - Second lot from a different manufacturer

d-Fructose - Second lot from a different manufacturer

Acetaminophen - Second lot from a different manufacturer

Salicylic Acid - Degradation product of Acetylsalicylic Acid

2-acetamidophenol - Constitutional isomer of acetaminophen

α-d-Lactose anhydrous - Dehydrated form of library material

Black items are positive challenge samples (Should Pass)

Red items are negative challenge samples (Should Fail)

External Challenge Validation Results

Compound	Score	Result
Glucose (Lot #2)	99.9	Pass
Fructose (Lot #2)	98.0	Pass
Acetaminophen (Lot #2)	100.0	Pass
Salicylic Acid	55.2	Fail
2-Acetamidophenol	19.3	Fail
Lactose Anhydrous	68.9	Fail

FT-NIR Success stories

- Customer had a plant with diverse array of incoming raw materials that needed to be classified accurately.
- Implemented NIR testing for over 200 materials
- Based upon technician time savings alone, paid back the investment in about 7 months
- Needed to identify a small number of materials at very high volume composing the bulk of their test load
- Implemented 100 % inspection for ID and qualification
- Dropped their testing time on each incoming shipment of raw material by 68 %

Lyophilized Material Analysis

- Lyophilized materials are an excellent application for analysis by NIR
- Packaging cannot be opened without corrupting the product inside
- Can apply to food, pharmaceutical, fine chemical or biotech industries

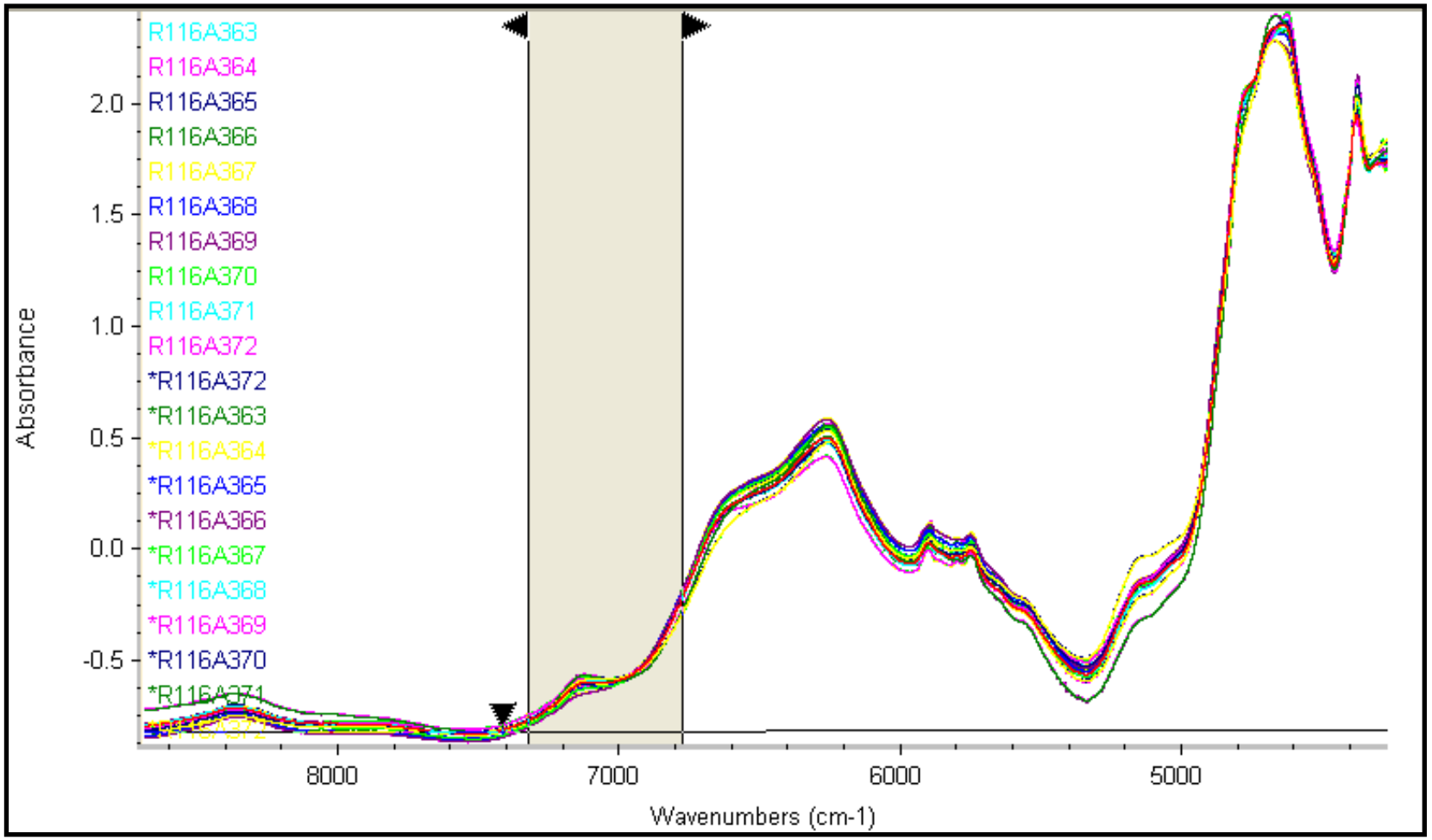
Current Testing for Lyophilized Products

- Current testing methods like Karl Fisher titration, LOD or GC
  - *cumbersome, time consuming and costly*
  - *destructive*
- NIR does not need solvents or carrier gases
- NIR calibrations for lyophilized materials have been shown to be as accurate as the reference method
- Other components can also be measured using the same NIR spectrum (Buffer, API, Cryoprotectant)

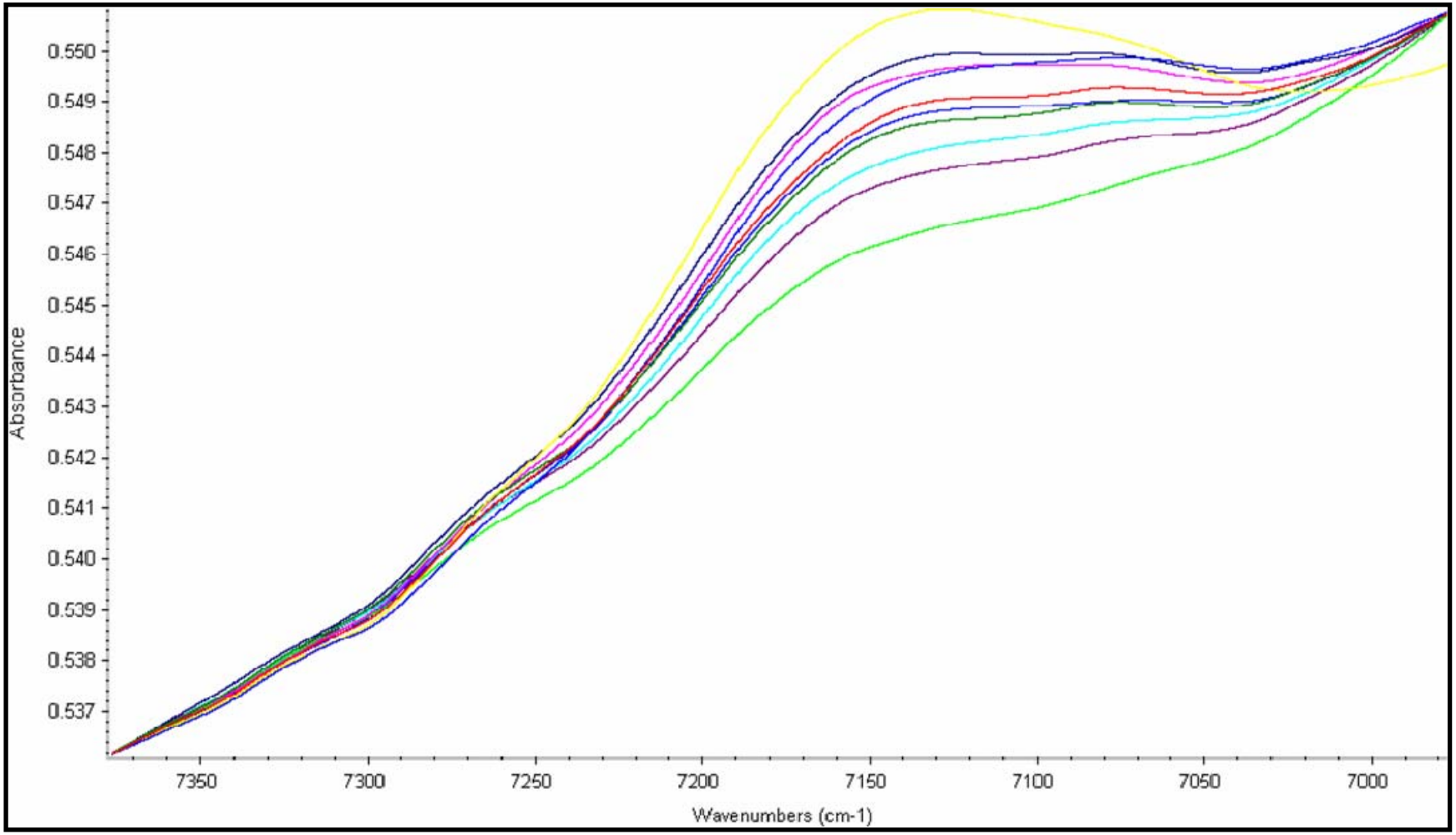
Case Study For Lyophilized Sample Analysis

- Primary component of interest in any lyophilized materials is moisture (0.5% - 2.0% typically)
- Sample is Thrombin, a topical coagulant used in the medical and dental fields
- Thrombin loses its efficacy if exposed to high heat or wet conditions. Current application will look at moisture and potency in Thrombin

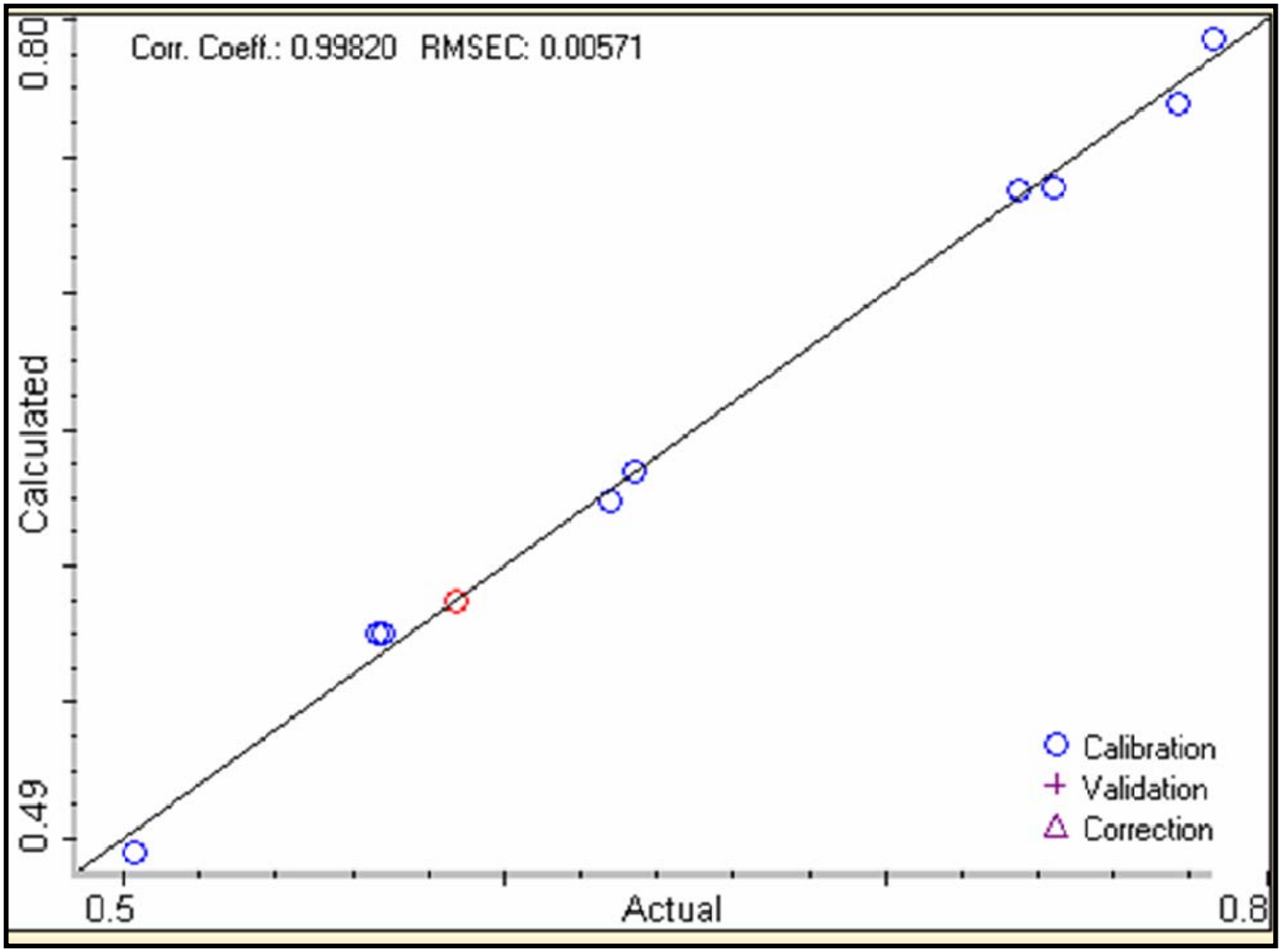
Moisture First Overtone Region



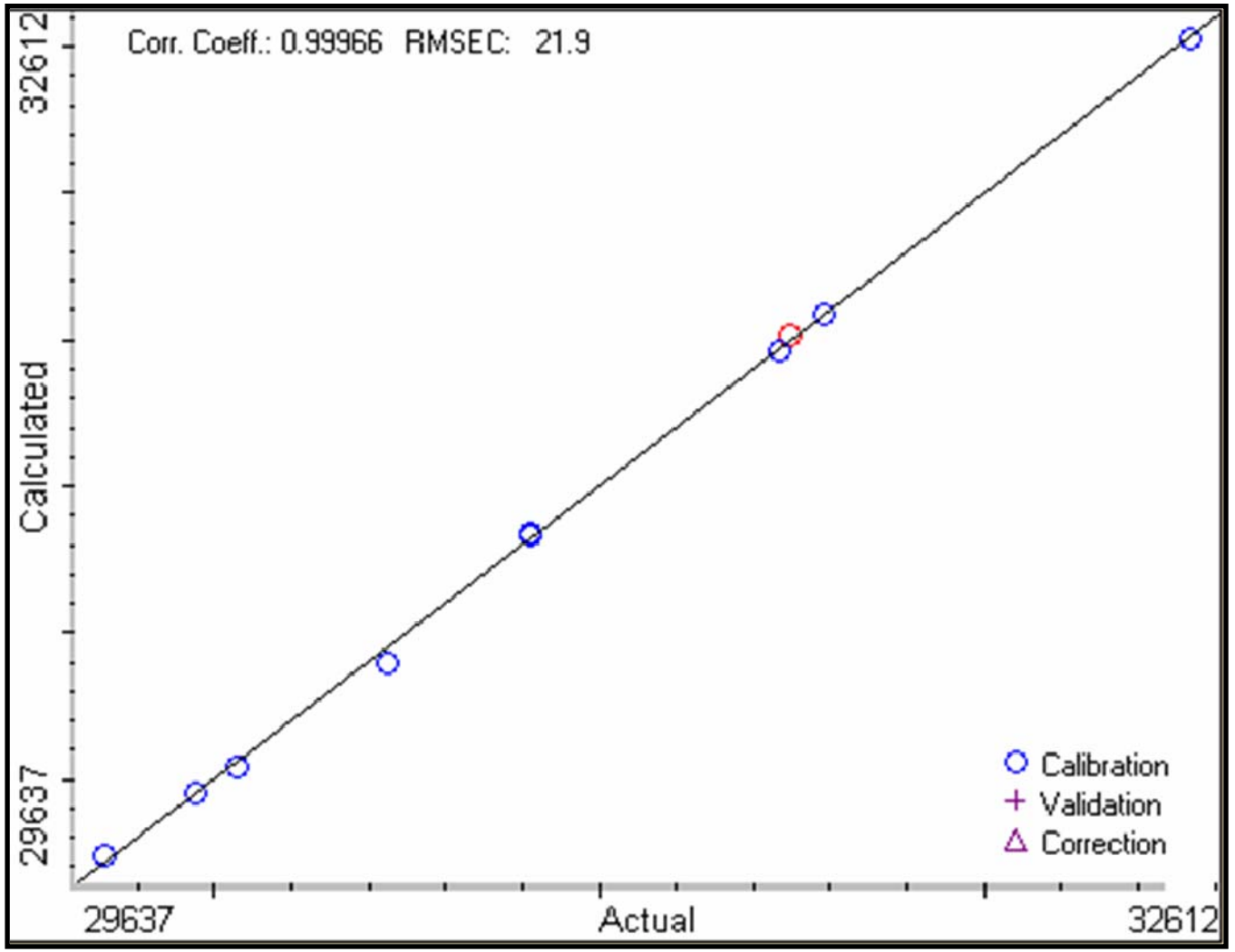
Close-up of First Moisture Overtone Region



Calibration Curve for Moisture



Calibration for Potency

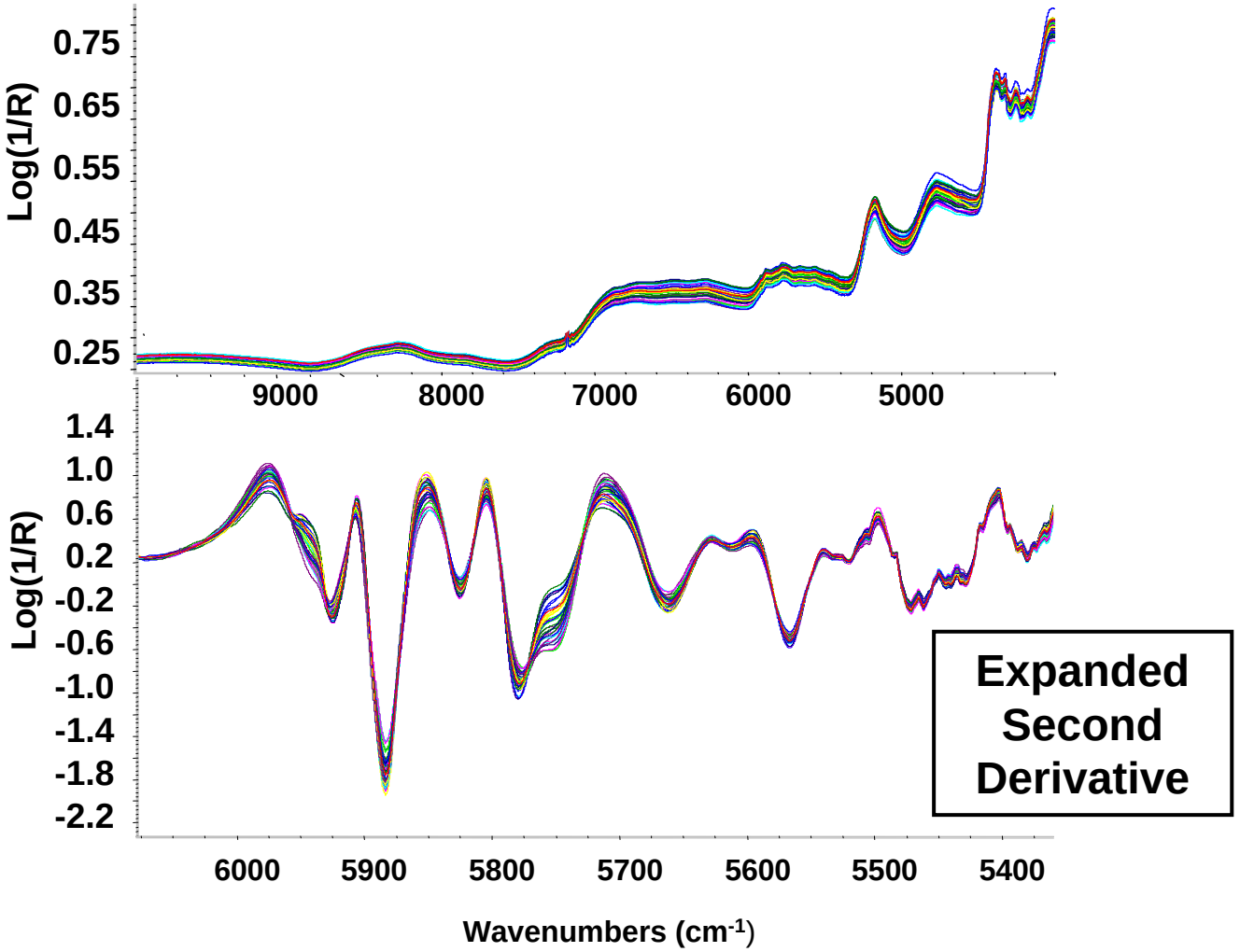


Quantification of Active Pharmaceutical Ingredient (API) in Tablets

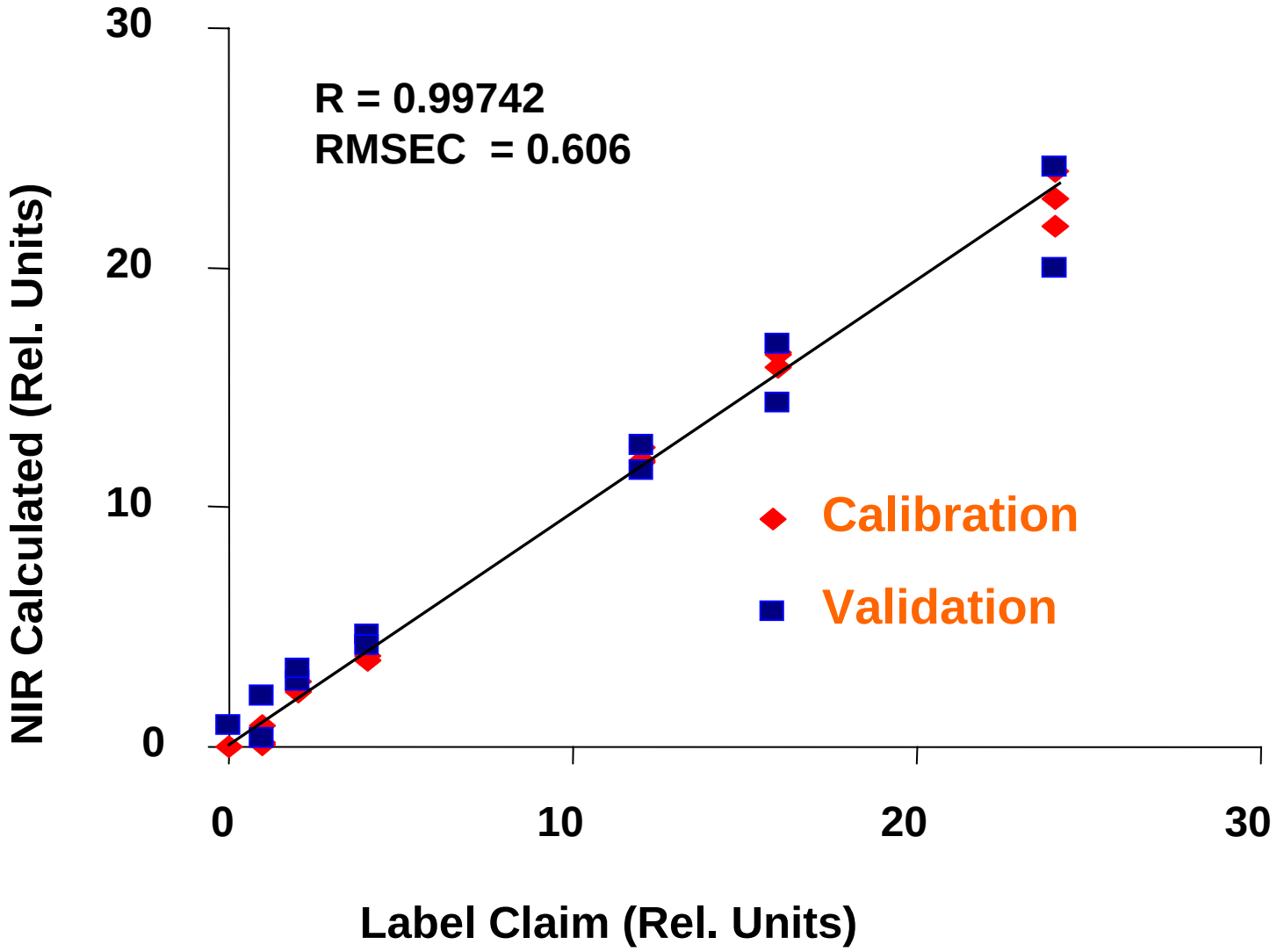
Show Concentration Differences in Tablets

- Distinguish different clinical tablet formulations by amount of active ingredient
- Finished product is a tablet and manufacturing protocols need independent verification of amount of active ingredient
- Understand the differences between transmission and reflection analysis of tablets

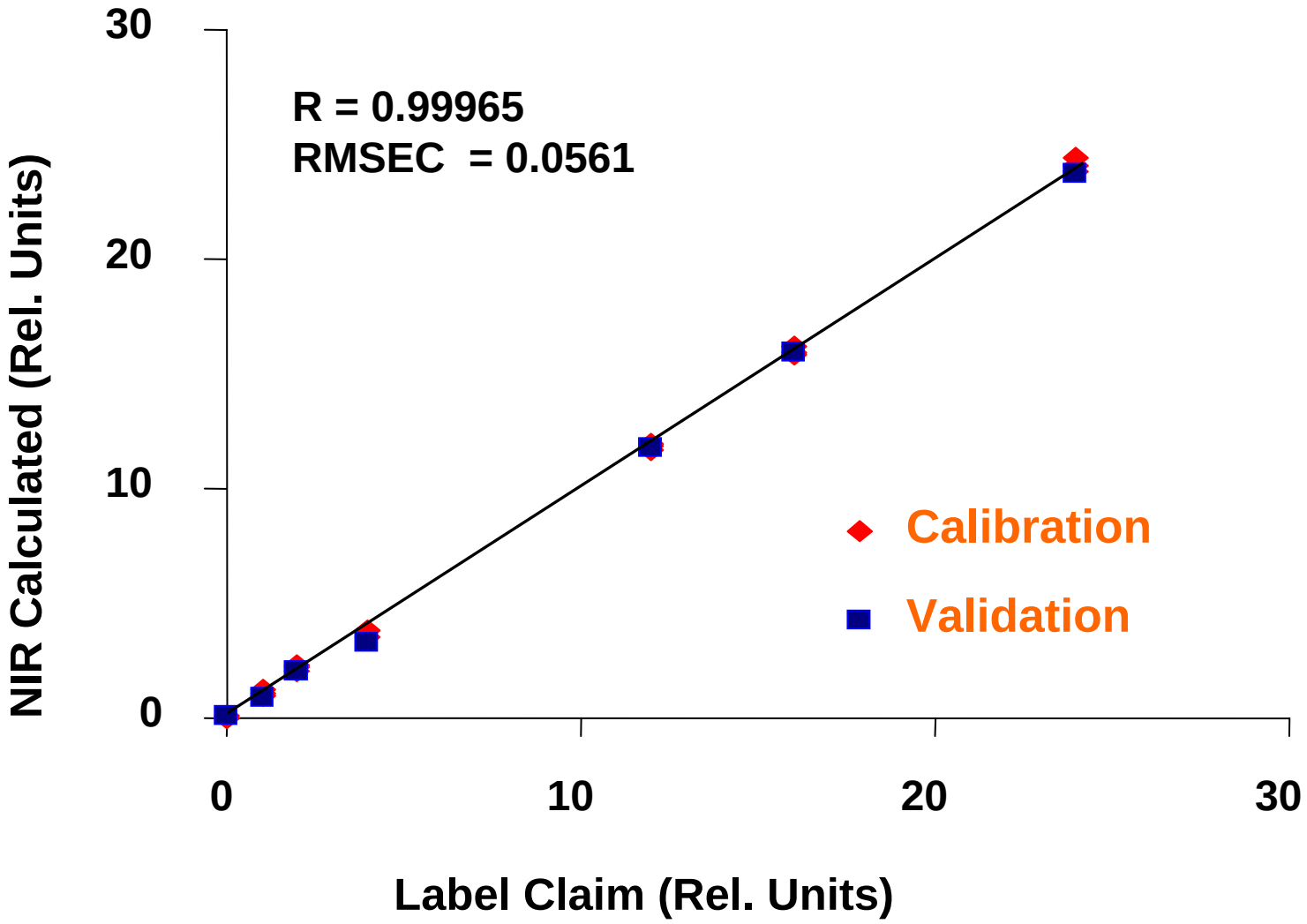
Reflectance Spectra for Clinical Tablets



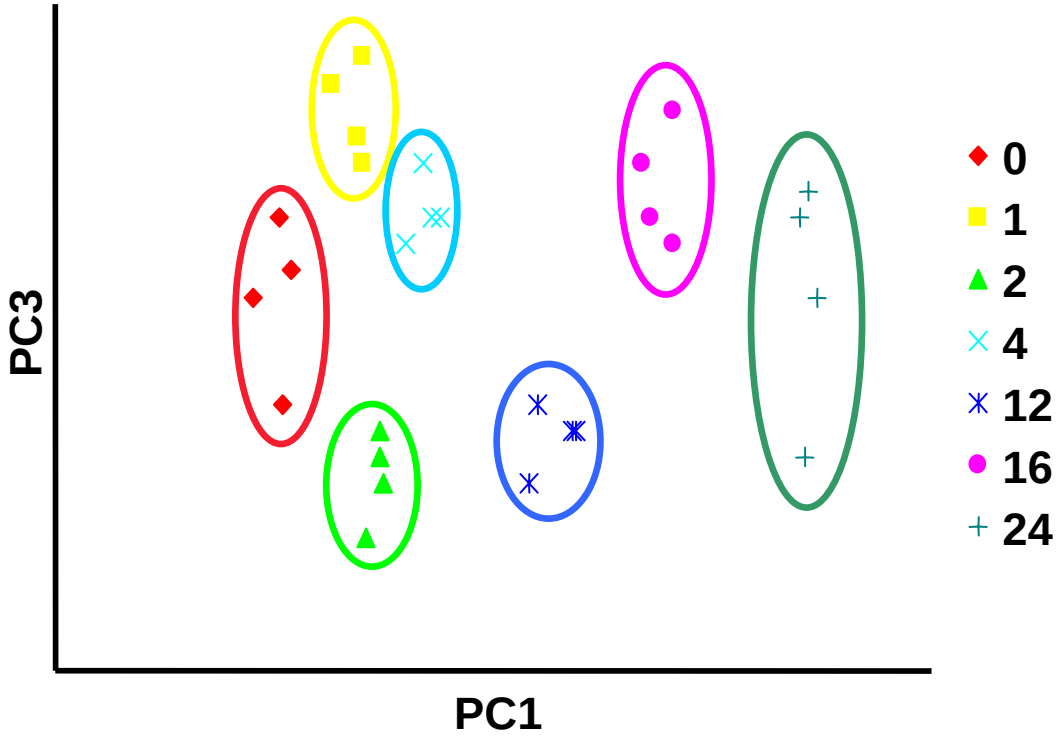
Tablet Reflection Results



Tablet Transmission Results



API Quantification Can Also Be Done Using Qualitative Methodology - Discriminant Analysis



- Principal component scores plot reveals quantitative method may not be necessary
- The required information is obtained with excellent results using qualitative analysis (Discriminant)

NIR in Pharmaceuticals - Summary

- NIR has proven useful in the following applications:
  - *Raw Material ID*
  - *Raw Materials - Particle Size*
  - *Analysis of Parenteral Drugs - Lyophilized Materials*
  - *Quantification of API in Tablets*
  - *Blend Homogeneity*
  - *Lactose Polymorph Screening*
  - *SoftGel Analysis*
  - *Enzyme Activity*

Near IR in the Chemical Industry

- Near IR analysis has proven worth across the chemical industry
  - *Fine chemicals*
  - *Polymers*
  - *Petrochemicals*
  - *Paints, coatings and adhesives*
  - *Soaps and cleaners*
  - *Synthetic materials*
- At-line, near-line and on-line

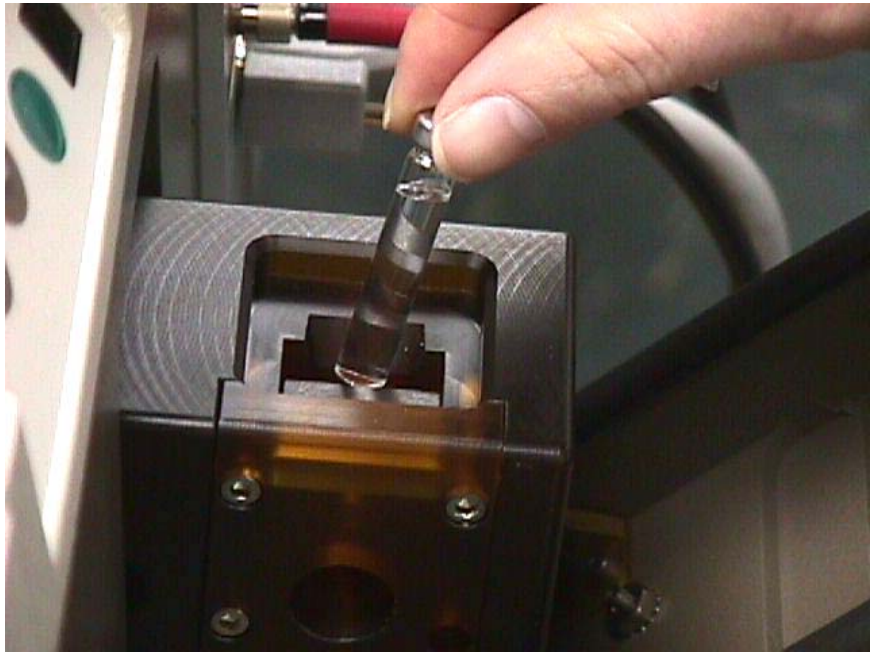
Polymerization Monitoring: Acid Value and Hydroxide

HoVal Analysis in NIR - Purpose

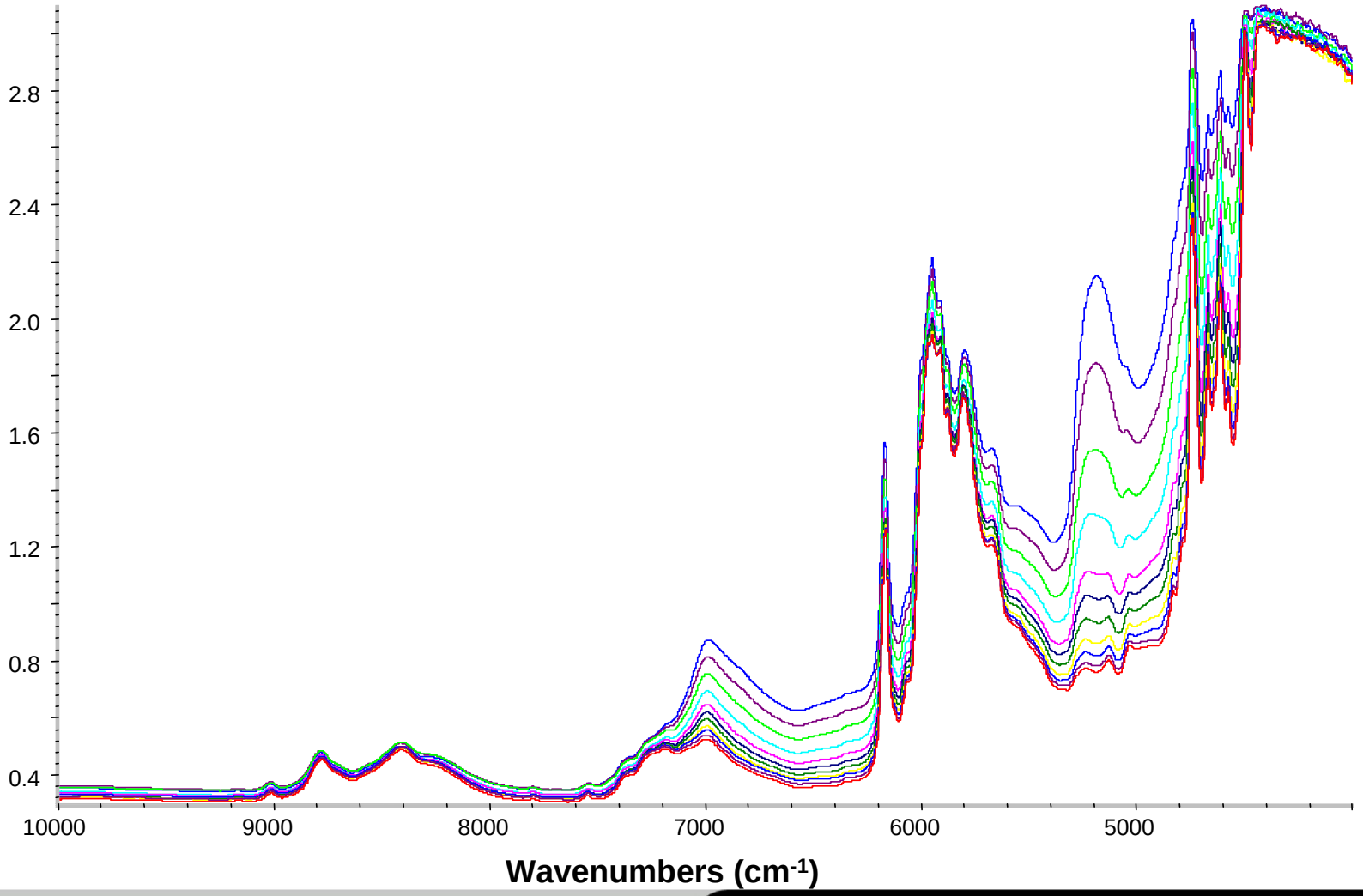
- HoVal is short for **H**ydr**o**xyl **Val**ue, an important metric in polymerization reactions
- Measures both starting materials (Acid and OH) simultaneously from the same spectra
- Condensation polymers (Polyesters) show decrease in acid and alcohol concentration with time
- Use NIR to monitor this decrease will show when polymerization is finished

Experimental

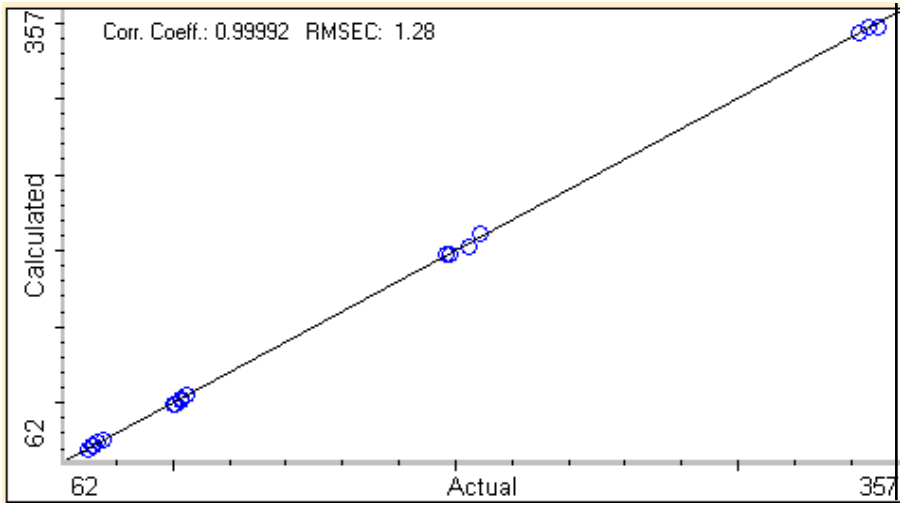
- Samples were placed into 6 mm disposable vials
- Analysis using Nicolet Antaris Transmission module



Spectral Data for HoVal Analysis

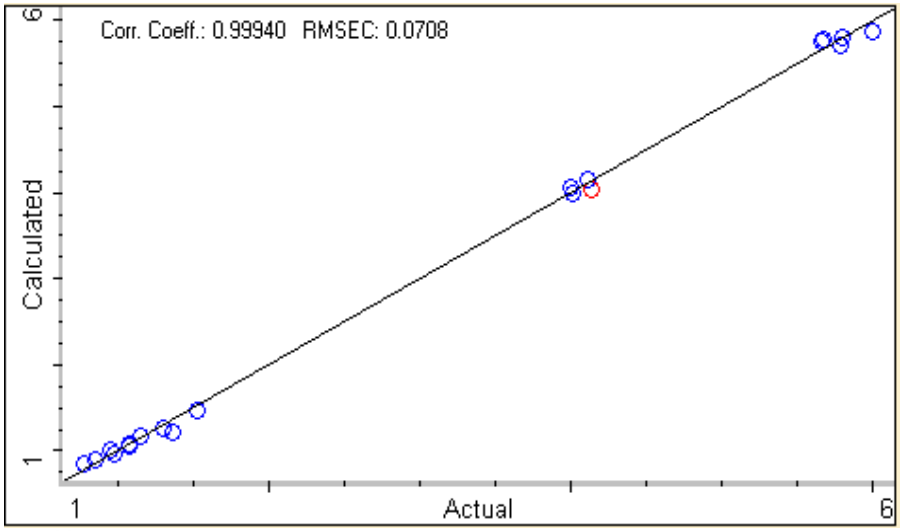


NIR Replaces Two Wet Chemical Methods



Hydroxyl Value Correlation

Acid Number Correlation



Plasticizer Content in PVC

Plasticizer Content in PVC

- Plasticizers are common additives to polymers.
- The quantity of plasticizer determines the properties of the polymer.
- The plasticizer contents of polymers differ depending on the application.
- Primary methods often involve tedious and time-consuming extractions.

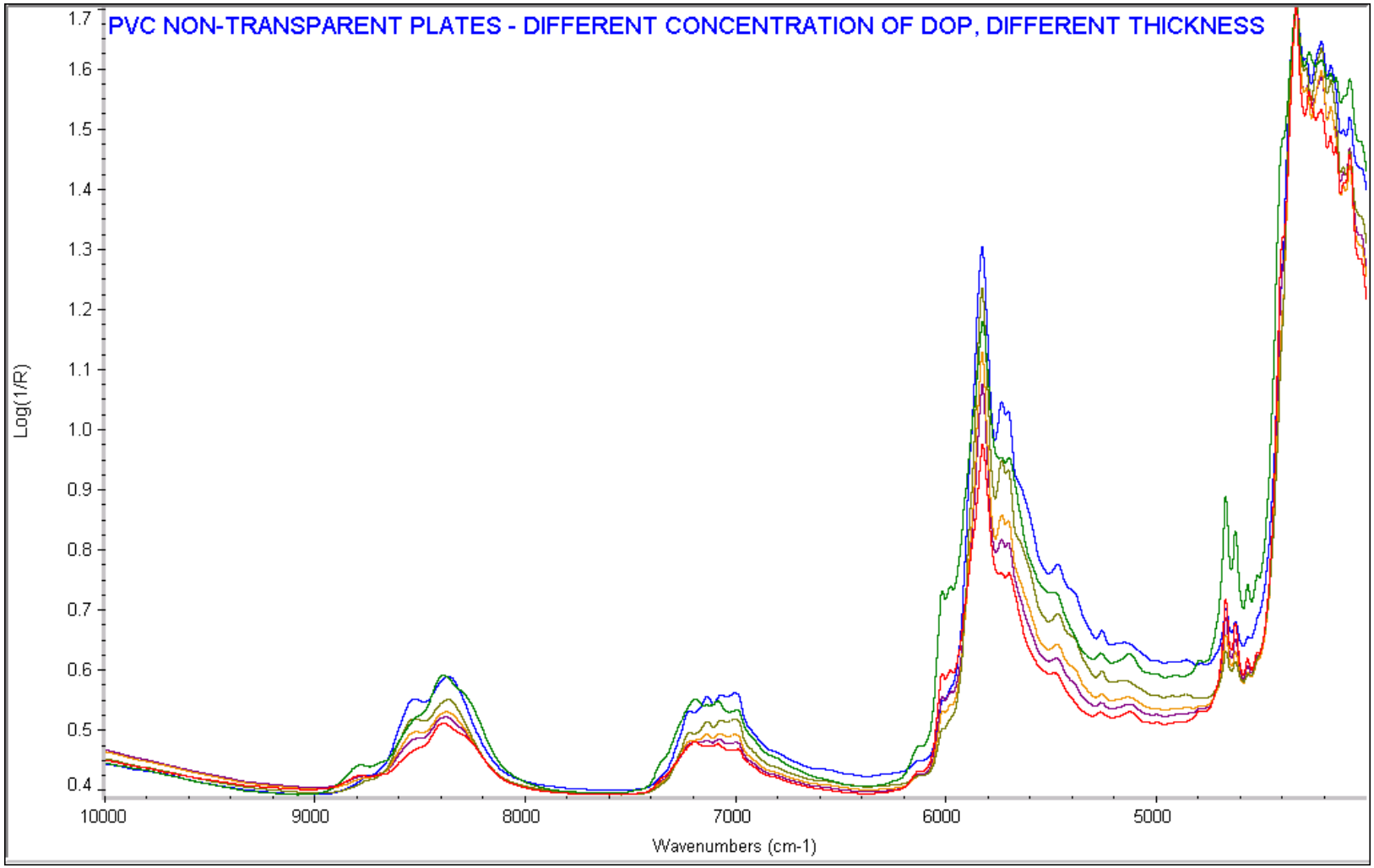
Samples

- Samples of polyvinyl chloride (PVC) with varying amounts of dioctylphthalate (DOP) as plasticizer
- Three types of samples:
  - *Opaque PVC plates with 5 - 50% DOP*
  - *Transparent PVC plates with 5 - 50% DOP*
  - *PVC Films with 9 - 40% DOP*

FT-NIR Measurements

- *As is* measurements (no sample preparation or modification).
- Data collected from 4,000 cm^{-1} to 10,000 cm^{-1}
- 4 cm^{-1} resolution.
- 90 scans
- One (1) minute collection time.
- All measurements done on Integrating Sphere

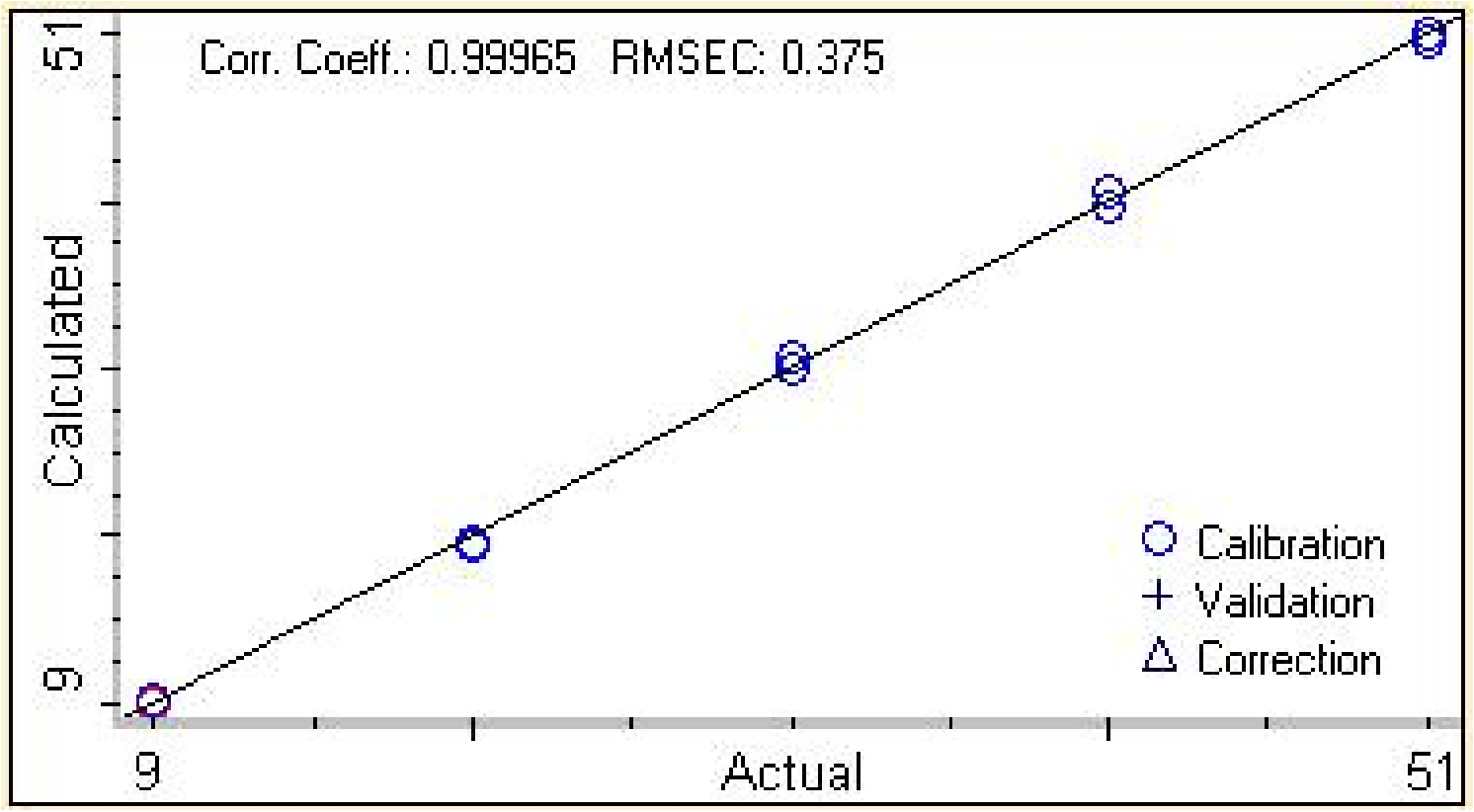
Non-Transparent Plates



Calibration Parameters

	Factors	Data Format	Smoothing	Region (cm^{-1})	Pathlength
Opaque	4	2^{nd} Derivative	Norris 9,0	8982-3920	MSC
Transparent	4	2^{nd} Derivative	Norris 5,5	4772-4520 5935-5322	MSC
Films	3	2^{nd} Derivative	Norris 5,5	4125-3946 4296-4263	MSC

Non-Transparent Plates



Calibration Results

	Calibration		Cross Validation	
	Corr. Coeff.	RMSEC	Corr. Coeff.	RMSECV
Opaque	0.999	0.375	0.995	1.49
Transparent Plates	0.999	0.591	0.996	1.45
Films	0.999	0.174	0.999	0.407

Conclusions on NIR of Plasticizer Content

- Additives such as plasticizers can be measured in various forms of plastic materials
- These materials can be finished or unfinished
  - *Measurements can be in-process or QC measurements*
- The analyses can be done easily, rapidly and non-destructively
- Current methodology for extraction of matrix elements in polymers is extremely time consuming

Near IR and Food

- Near IR analysis of food and food products has found extensive use in all segments of the industry
  - *Processed foods*
  - *Beverages*
  - *Grain*
  - *Fruit*
  - *Meat*

- Common components like protein, moisture, ash, starch and fat are measured simultaneously without destroying the sample

FT-NIR As A Process Analytic in Food

- Develop methods for any type of food product
- High resolution advantage of FT technology keeps number of standards low
- Many different sampling accessories for food products
  - *Sample spinner*
  - *SabIR Fiber Optic Probe*
  - *Cheese and Viscous Liquid Samplers*
  - *Softgel detector (Gum)*
- OPC Output for analyzer incorporation into Process Control Systems

NIR Analysis in the Food Industry

- Moisture and Protein in Barley
- Viscous Liquid Analysis
- Oil in Rice Powder
- Amylose Levels in Corn
- Multi-component Analysis of Wine
- Fat and Dry Matter in Cheese
- Discrimination of Jellybeans
- Choline Chloride Levels in Silage

Nicolet Antaris Product Line – Complete Solution

- FT-NIR Nicolet Antaris Spectrometer Platform

- RESULT Analyzer Software Platform

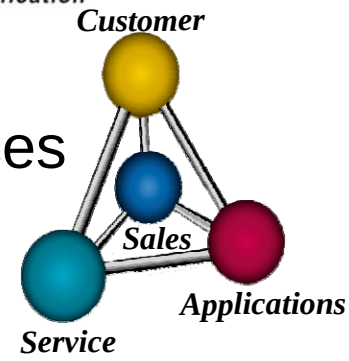
RESULT™ Integration Software

- TQ Analyst Chemometric Software Platform

- ValPro Validation/Qualification Platform

ValPro™ System Qualification

- Support “Pyramid of Support” products and services



The FT-NIR Advantage

The Bottom Line:

Save Time and Money.

Use FT-NIR to its maximum capability.