



VYSOKÁ ŠKOLA
CHEMICKO-TECHNOLOGICKÁ
V PRAZE



NIR spectrometry

Pavel Matějka

NIR spectrometry

- ❖ molecular absorption/reflection spectrometry
- ❖ non-destructive method used in process analysis, QC/AC
- ❖ practical method that can replace more expensive, more time-consuming and more laboured methods – GC, HPLC, titrimetry
- ❖ relatively fast method for a routine use in technological applications

NIR spectrometry

- ❖ qualitative information – **NIR libraries** –
identification of pure substances and/or
check of pre-defined mixtures
 - pharmaceuticals, polymers etc.

- ❖ quantitative analysis – multivariate
calibration models
 - **multi-component analysis** - organic, inorganic

NIR spectrometry

NIR

- ❖ from
 - $14\ 000\ \text{cm}^{-1}\ 714\ \text{nm}$
 - $12\ 500\ \text{cm}^{-1}\ 800\ \text{nm}$
 - $12\ 000\ \text{cm}^{-1}\ 833\ \text{nm}$

- ❖ to
 - $4\ 000\ \text{cm}^{-1}\ 2\ 500\ \text{nm}$

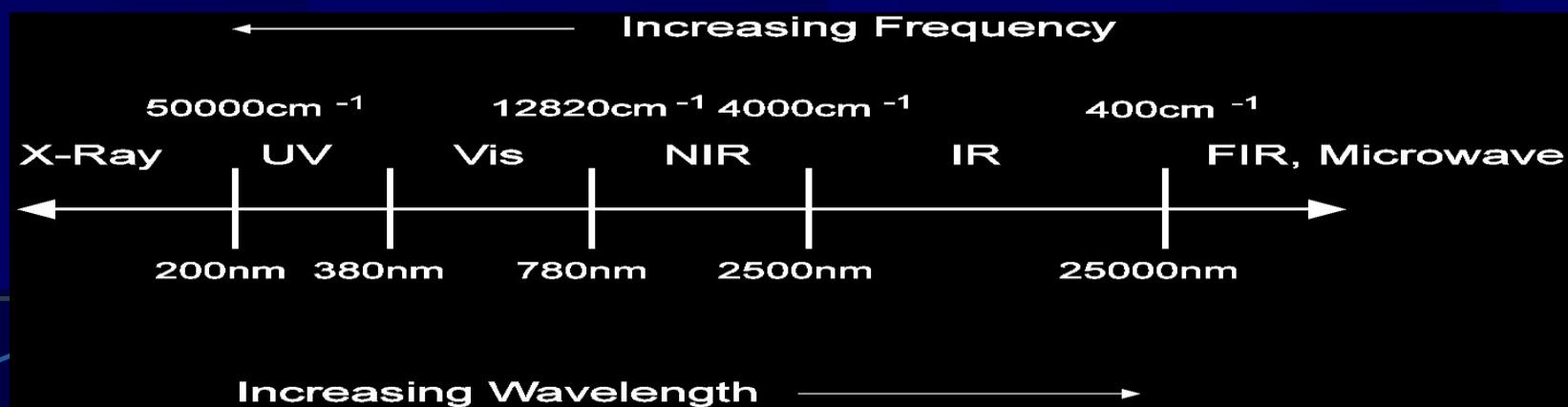
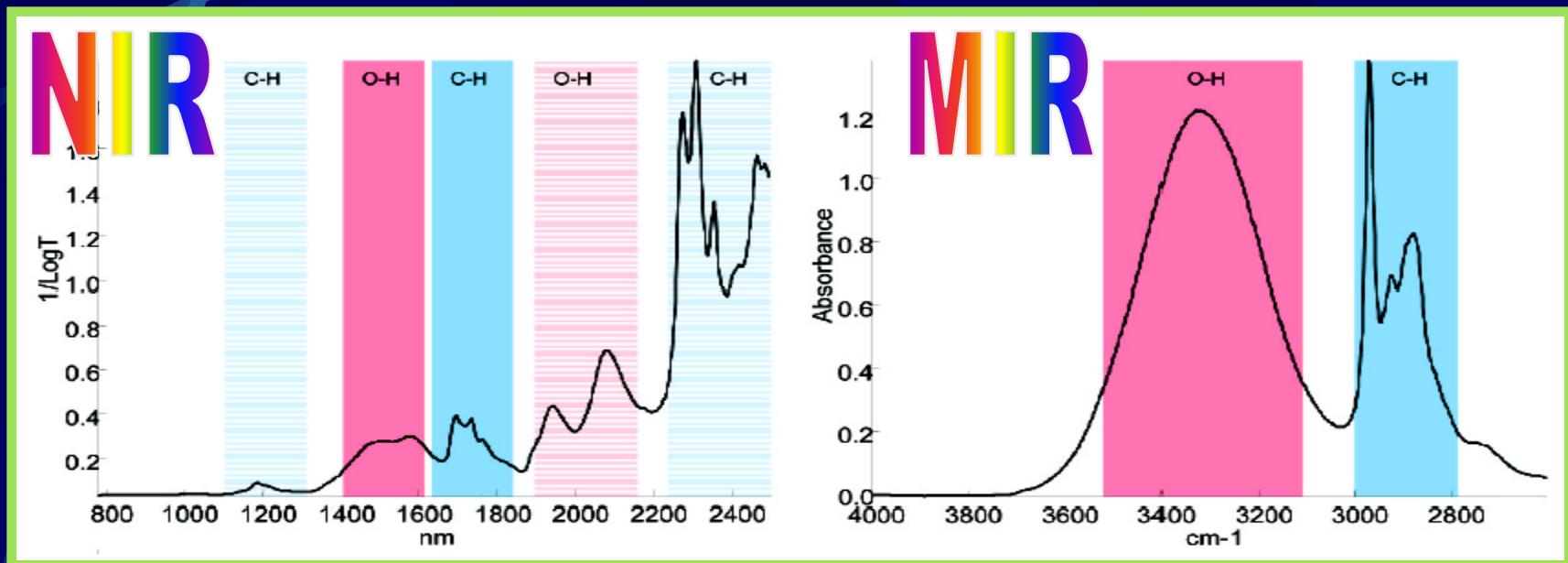
MIR

- ❖ from
 - $4\ 000\ \text{cm}^{-1}\ 2\ 500\ \text{nm}$

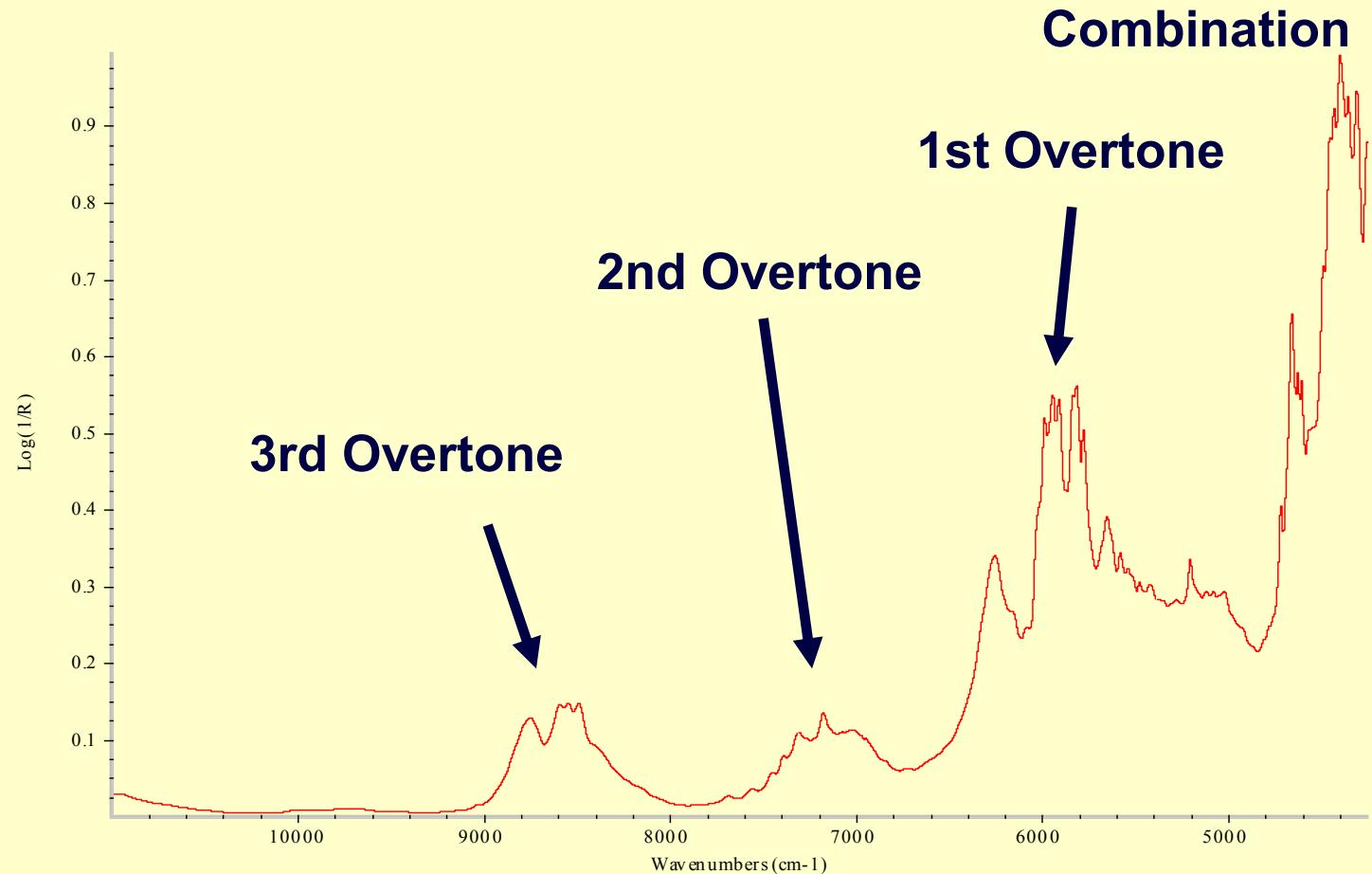
- ❖ to
 - $400\ \text{cm}^{-1}\ 25\ 000\ \text{nm}$
 - $200\ \text{cm}^{-1}\ 50\ 000\ \text{nm}$

NIR spectrometry

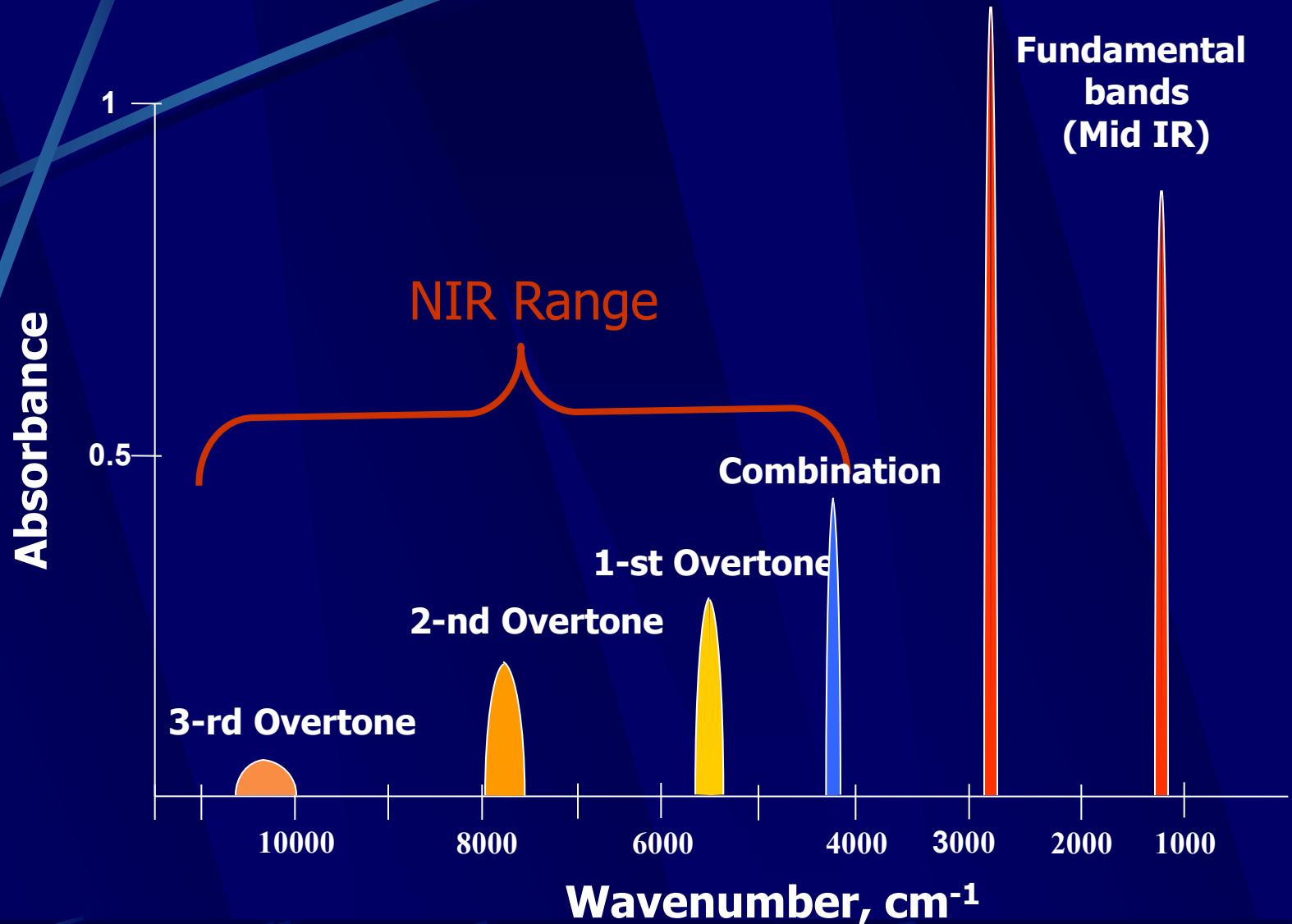
- ❖ relatively broad bands – overtones and combination bands

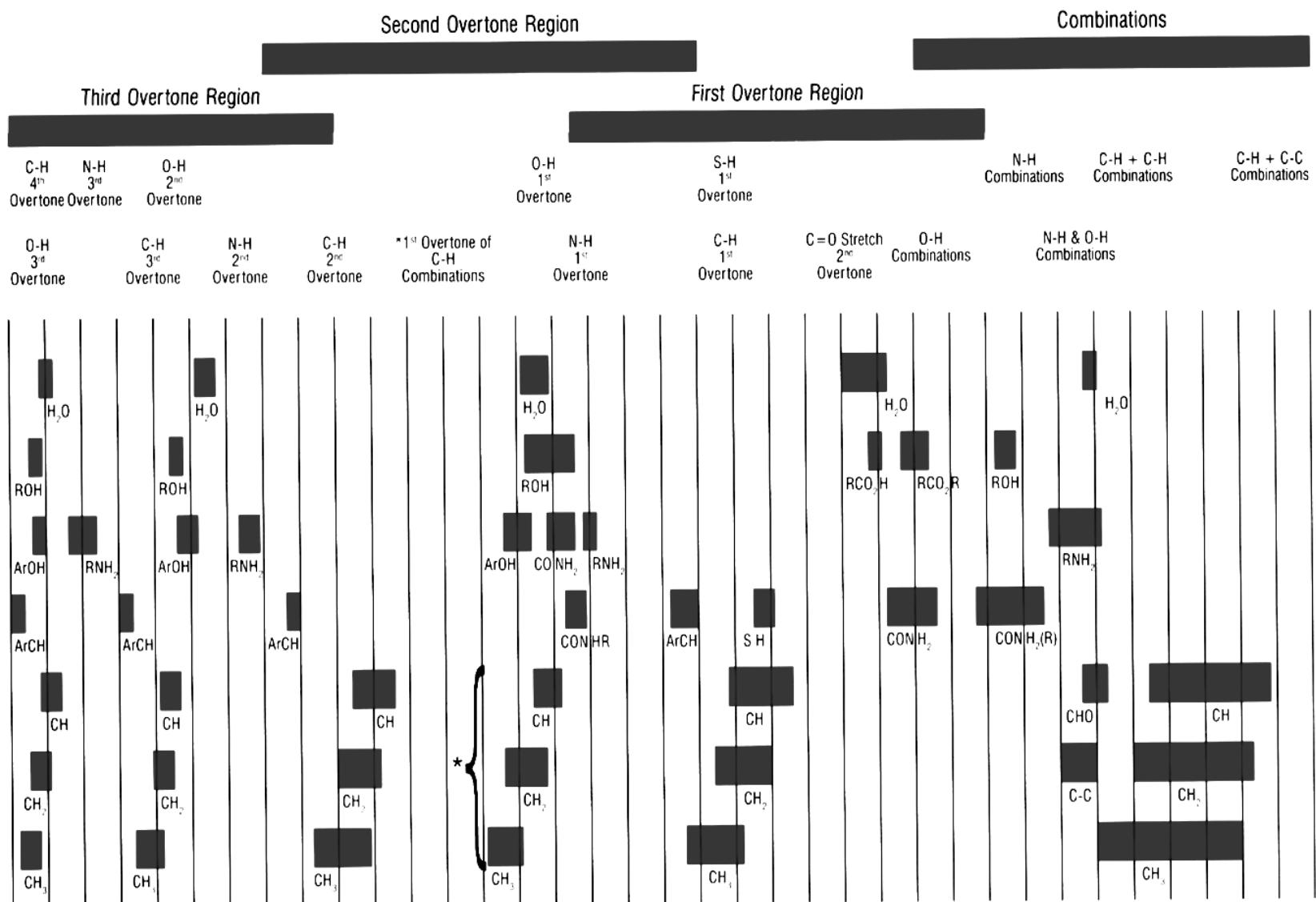


NIR spectra



NIR spectra

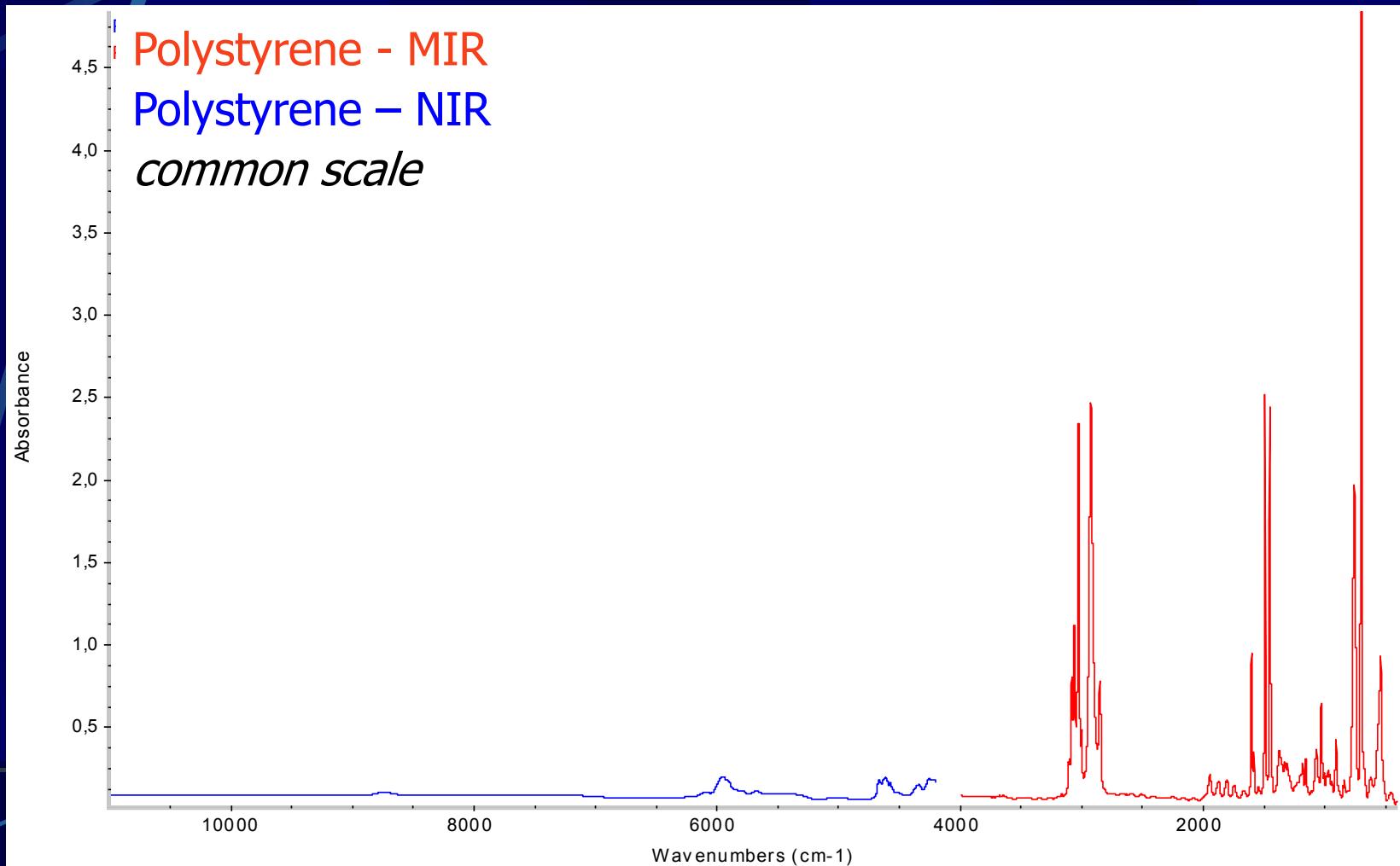




Wavelength λ nm	700	800	900	1000	1100	1200	1300	1400	1500	1600	1700	1800	1900	2000	2100	2200	2300	2400	2500
Wavenumber ν cm ⁻¹	14286	12500	11111	10000	9091	8333	7692	7143	6667	6250	5882	5556	5263	5000	4762	4545	4348	4167	4000

NIR/MIR spectra intensities

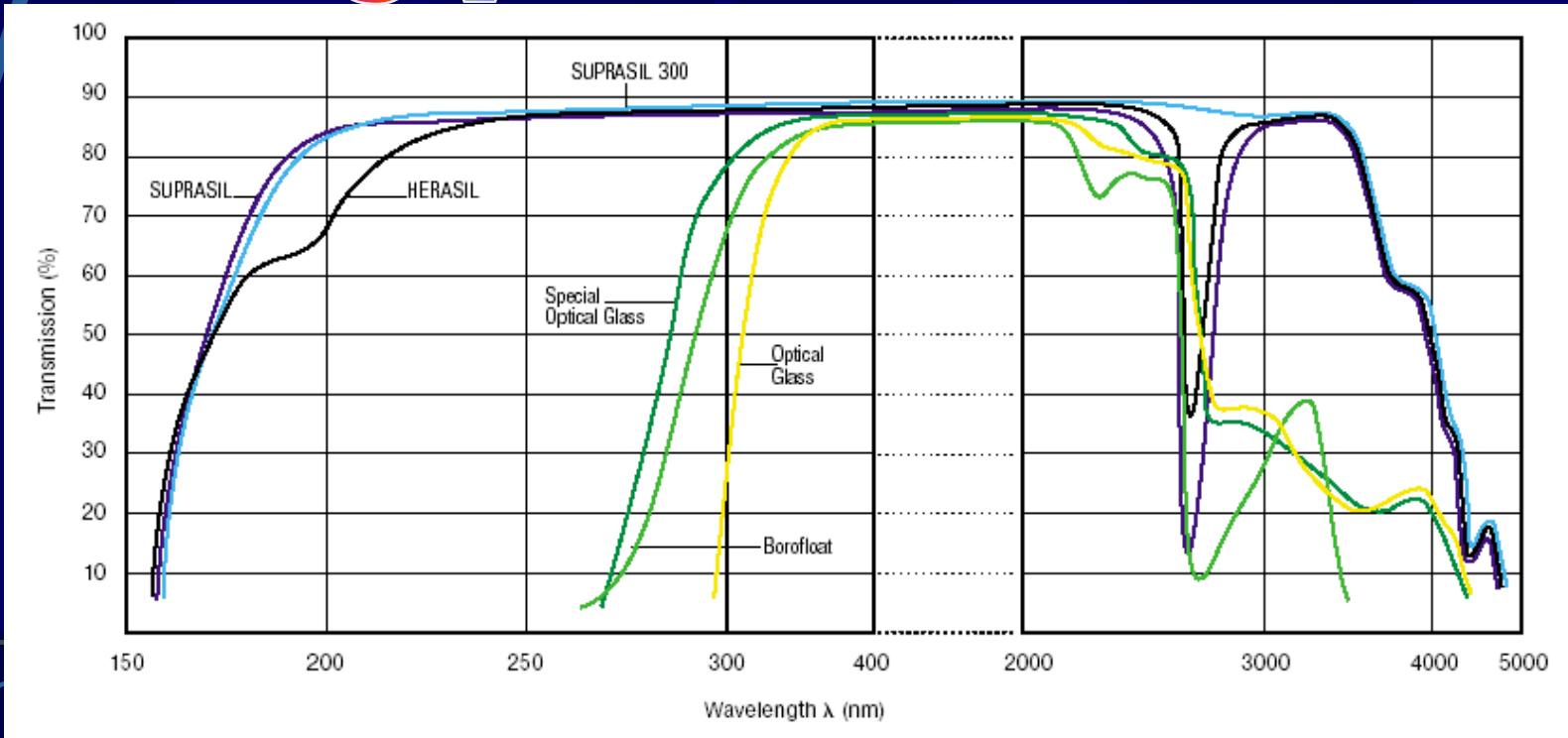
- ❖ the intensities are decreasing with increasing frequency / wavenumber



NIR spectrometry – transmission measurement

- ❖ cells - various types of glass
 - INFRASIL, SUPRASIL (critical part $\sim 4000 \text{ cm}^{-1}$)

UV VIS-NIR MIR

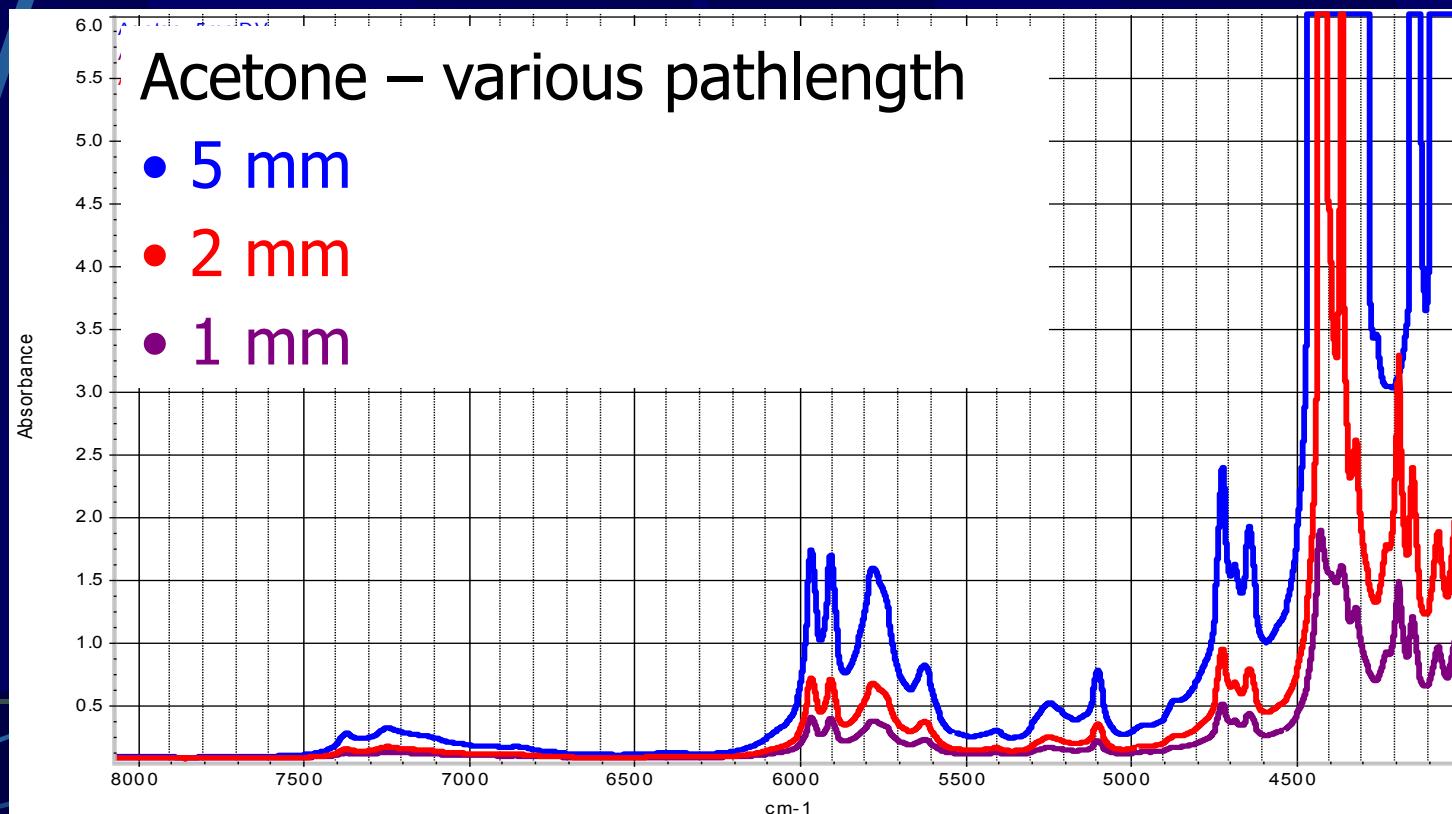


NIR spectrometry – transmission measurement

- ❖ cells - various types of glass
 - pathlength 1 – 5 (10) mm
 - effect of solvent absorption
 - effect of selected subregion (combination bands, order of overtones)
 - effect of concentrations of analytes studied
- ❖ fiber optics probes
 - fixed pathlength or adjustable pathlength

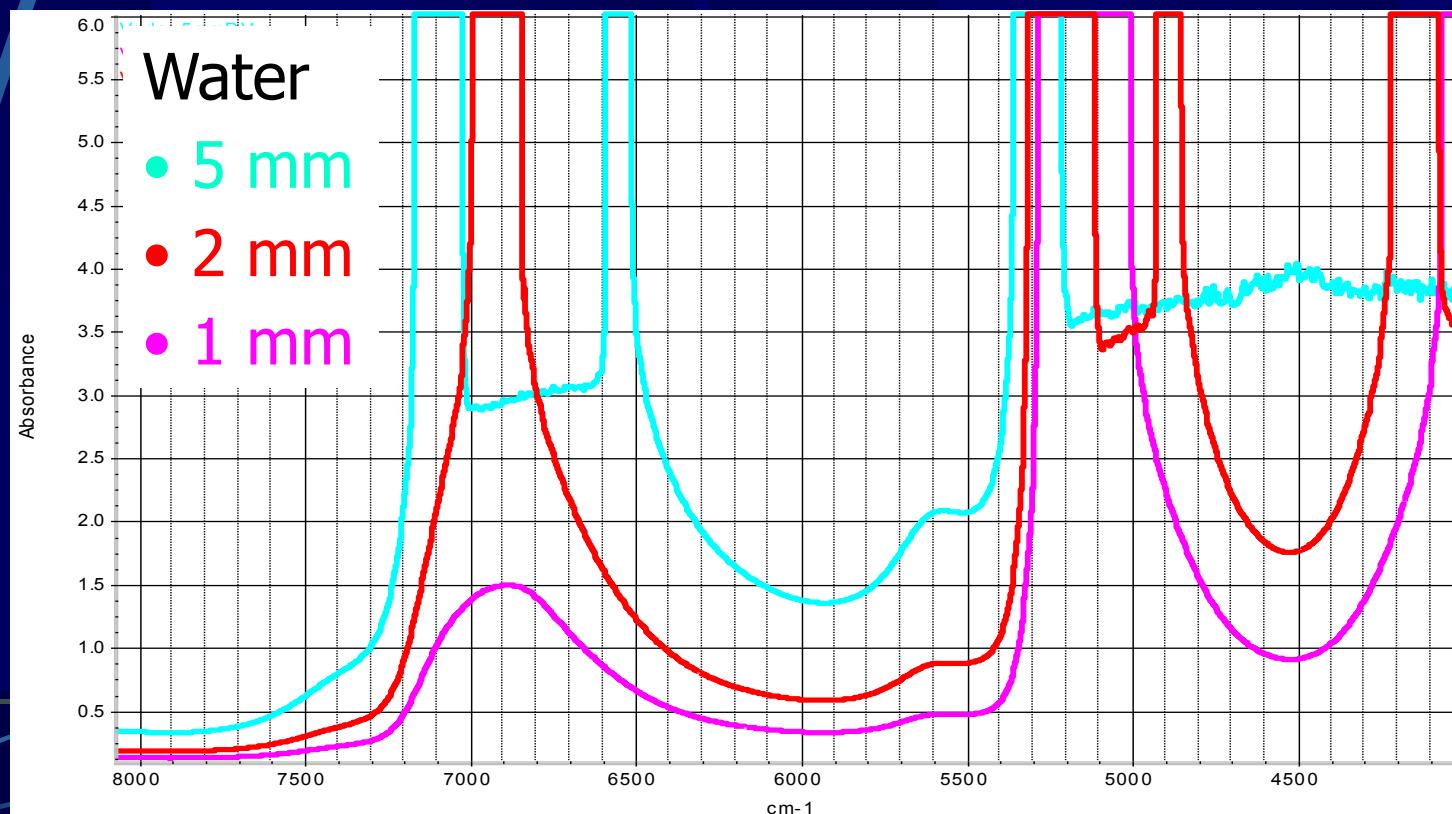
NIR spectrometry – transmission measurement

- ❖ cells – both polar and non-polar samples
 - organic liquids (oils, petroleum)
 - aqueous solutions (drinks – content of sugars, ethanol)



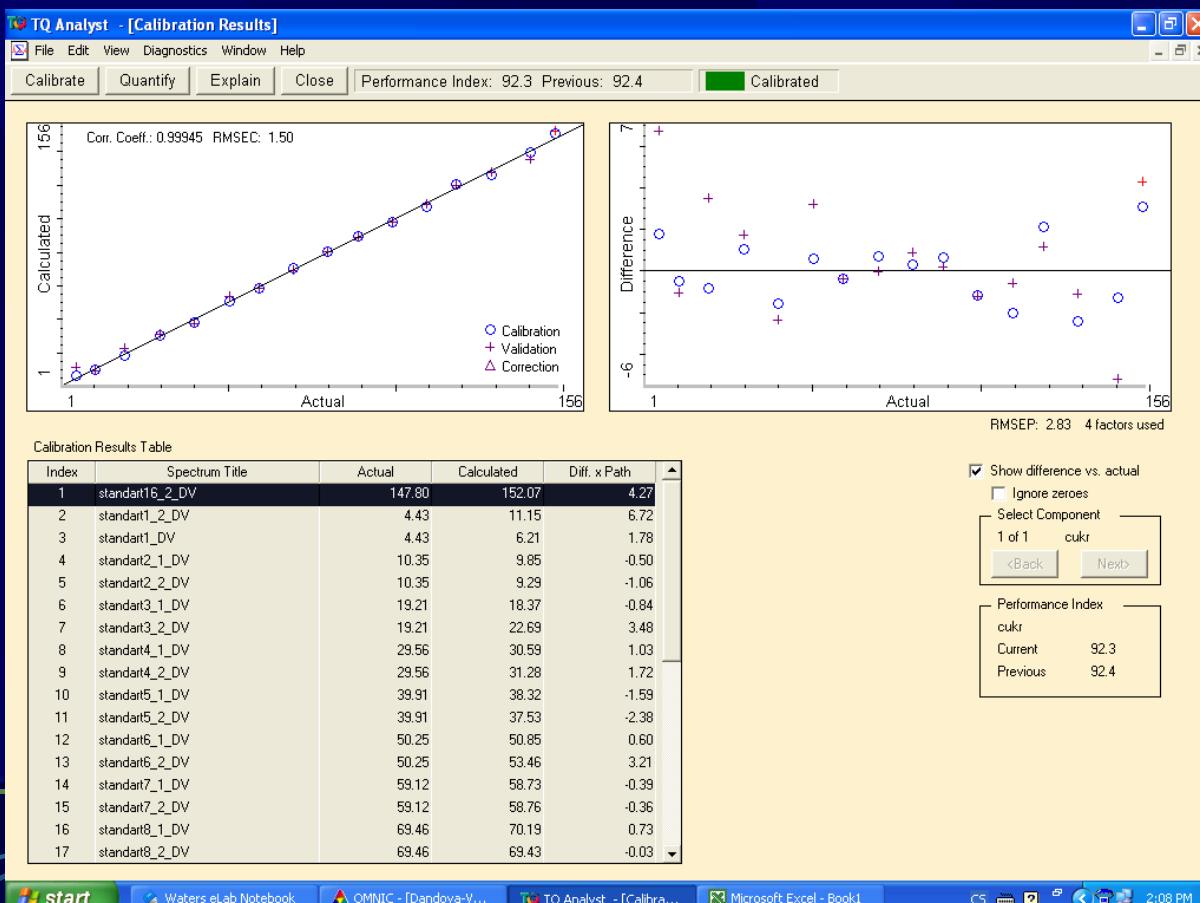
NIR spectrometry – transmission measurement

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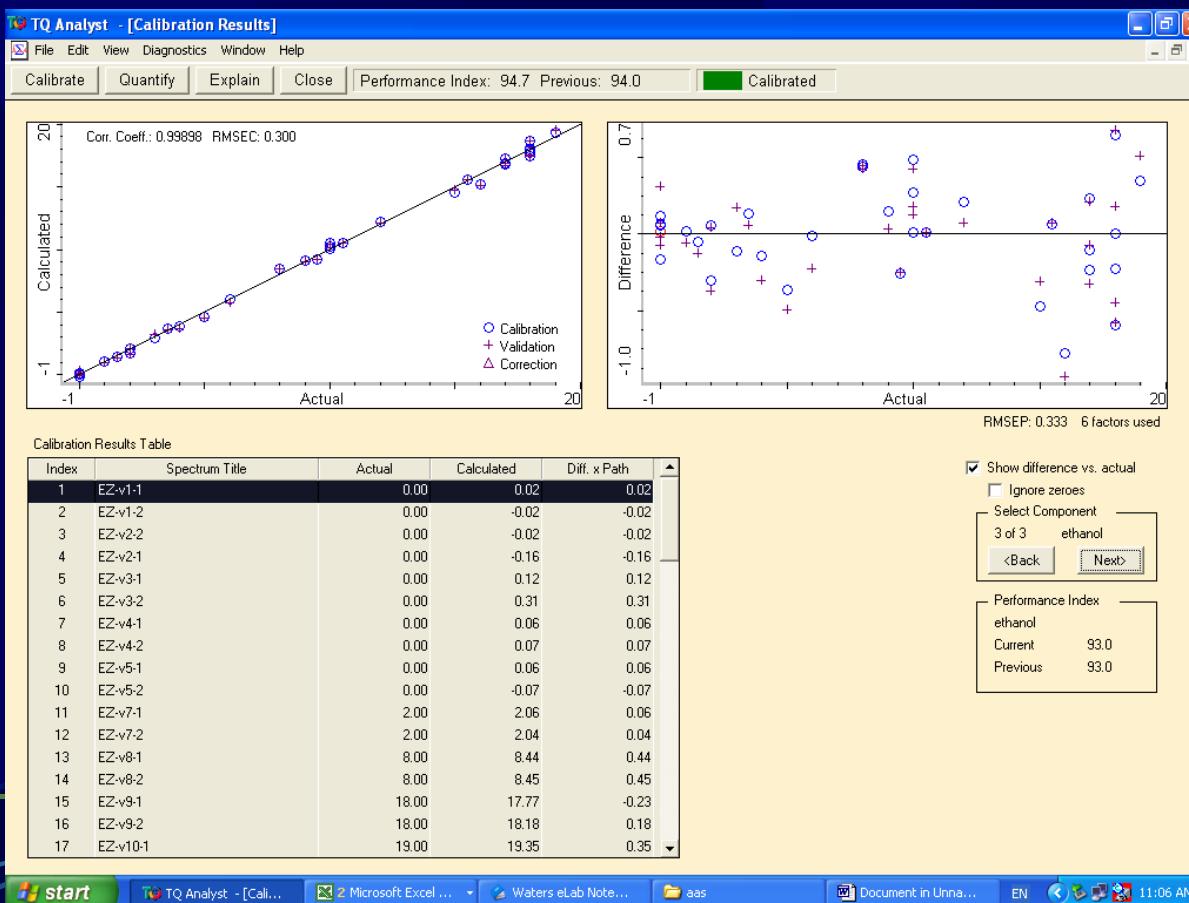
NIR spectrometry – transmission measurement

- ❖ aqueous solutions
 - calibration model for sugar content in soft drinks



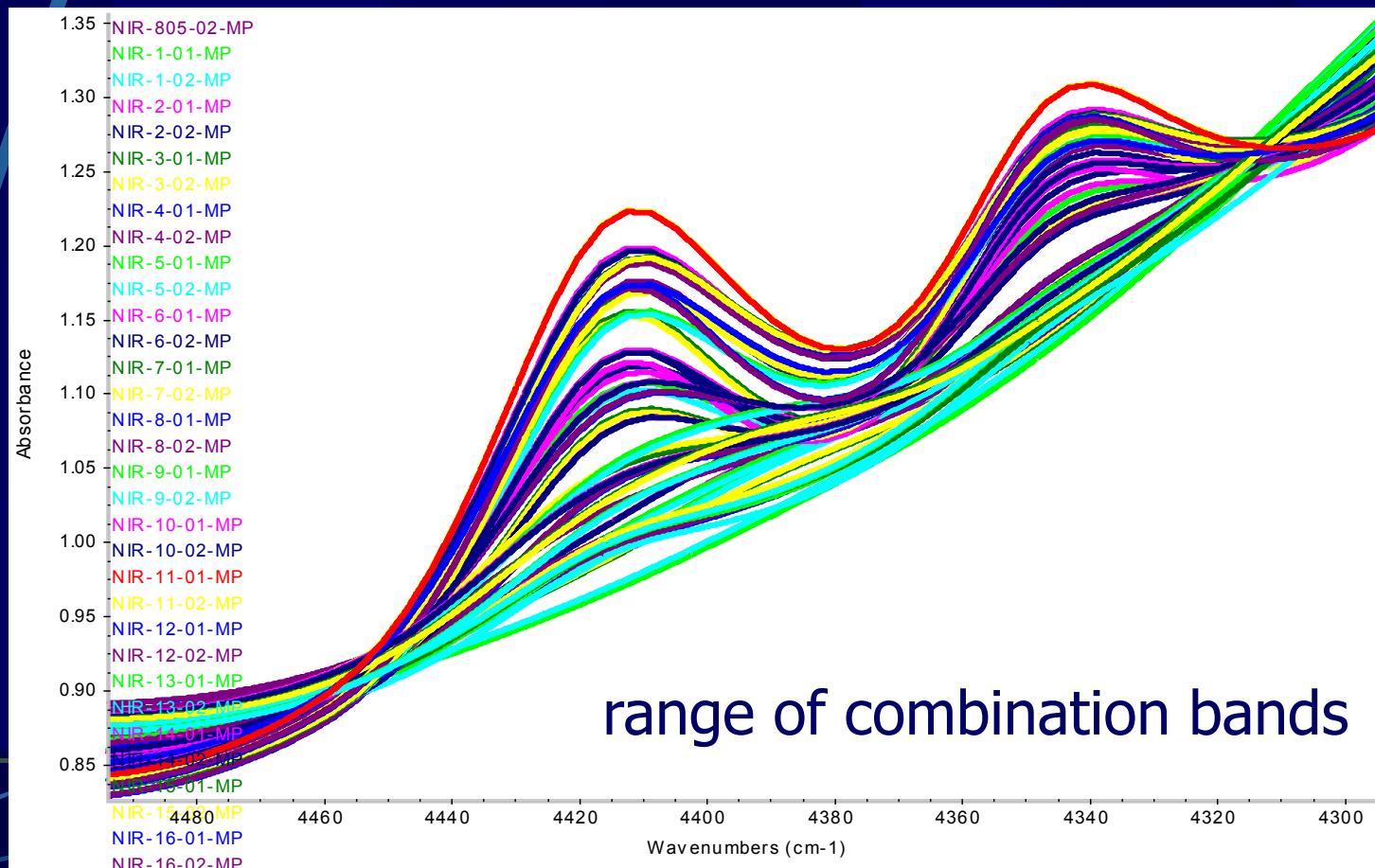
NIR spectrometry – transmission measurement

- ❖ aqueous solutions
 - calibration model for alcoholic drinks



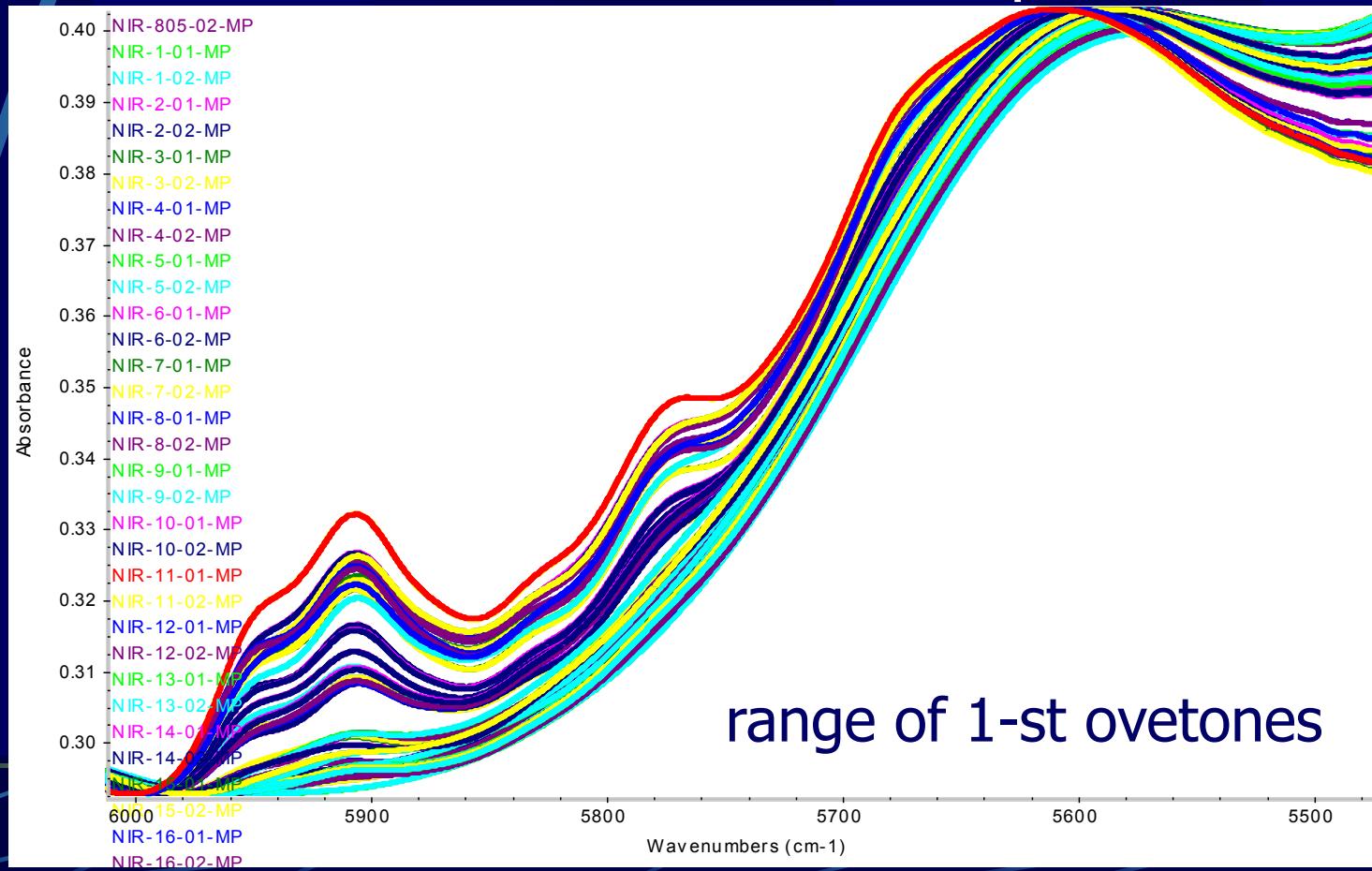
NIR spectrometry – transmission measurement

- ❖ aqueous solutions
 - calibration for alcoholic drinks - spectra



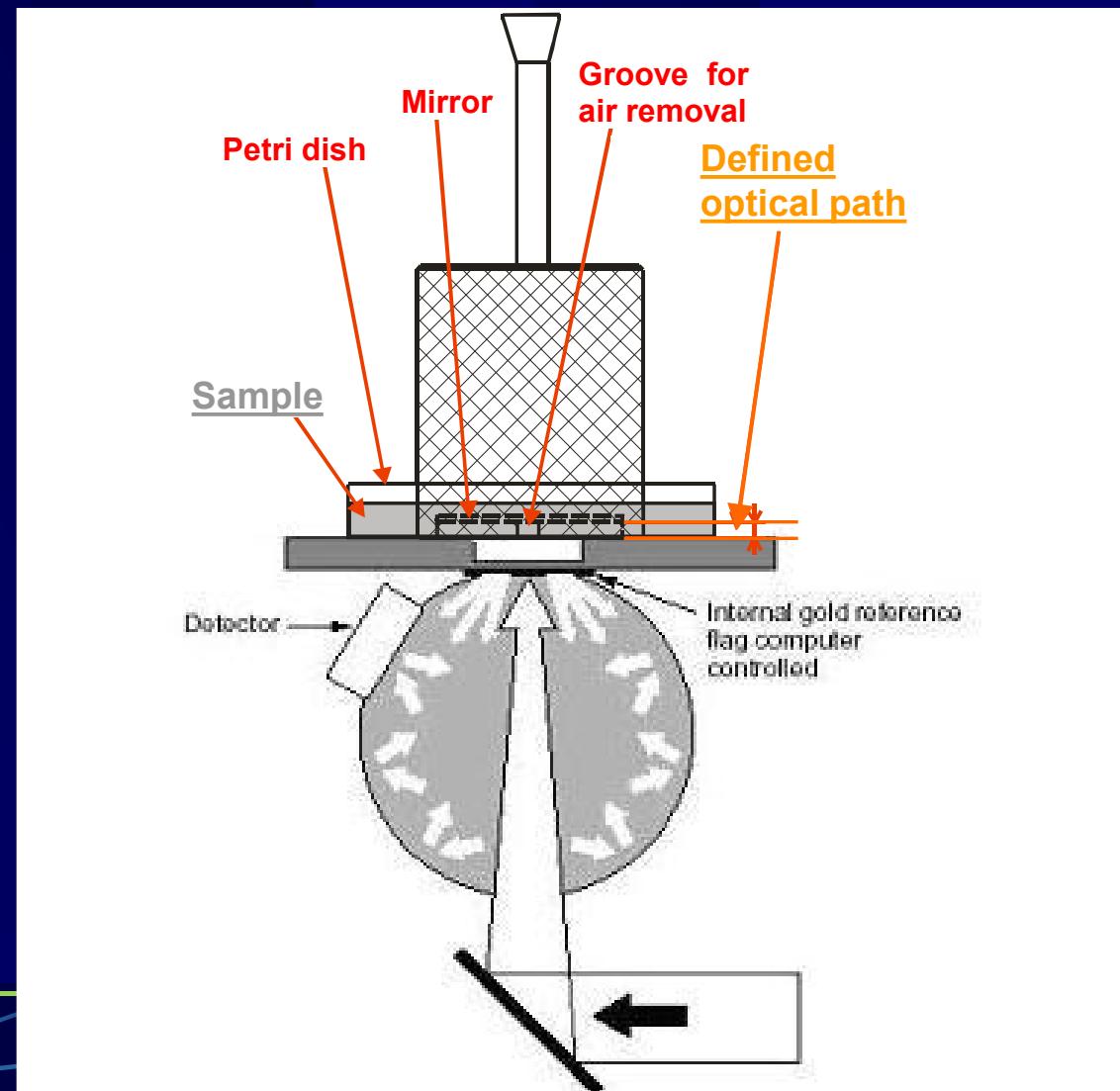
NIR spectrometry – transmission measurement

- ❖ aqueous solutions
 - calibration for alcoholic drinks - spectra



NIR spectrometry – transreflectance measurement

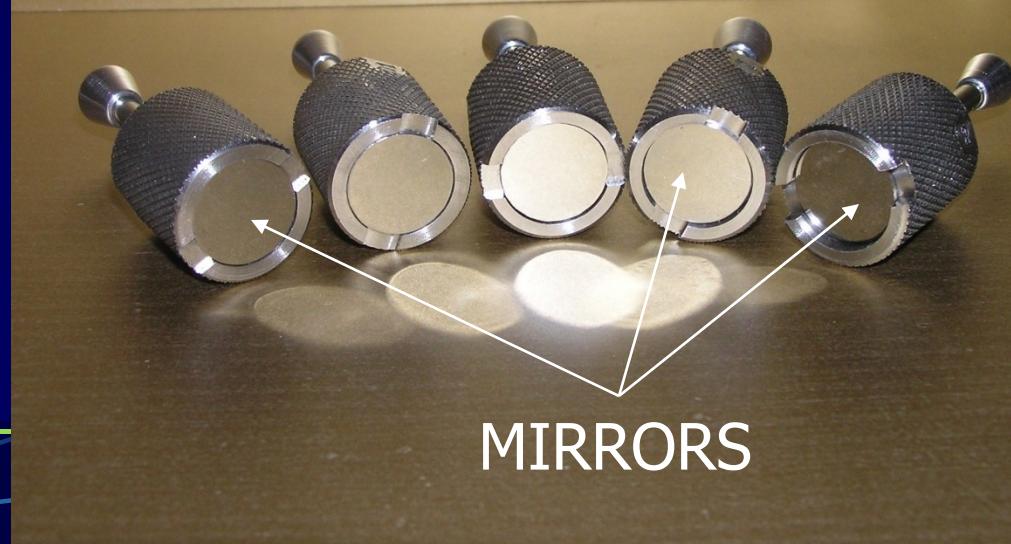
- ❖ transreflectance cells
 - defined pathlength
 - transmission/reflection
 - viscous liquids, pastes



NIR spectrometry – transreflectance measurement

- ❖ transreflectance cells

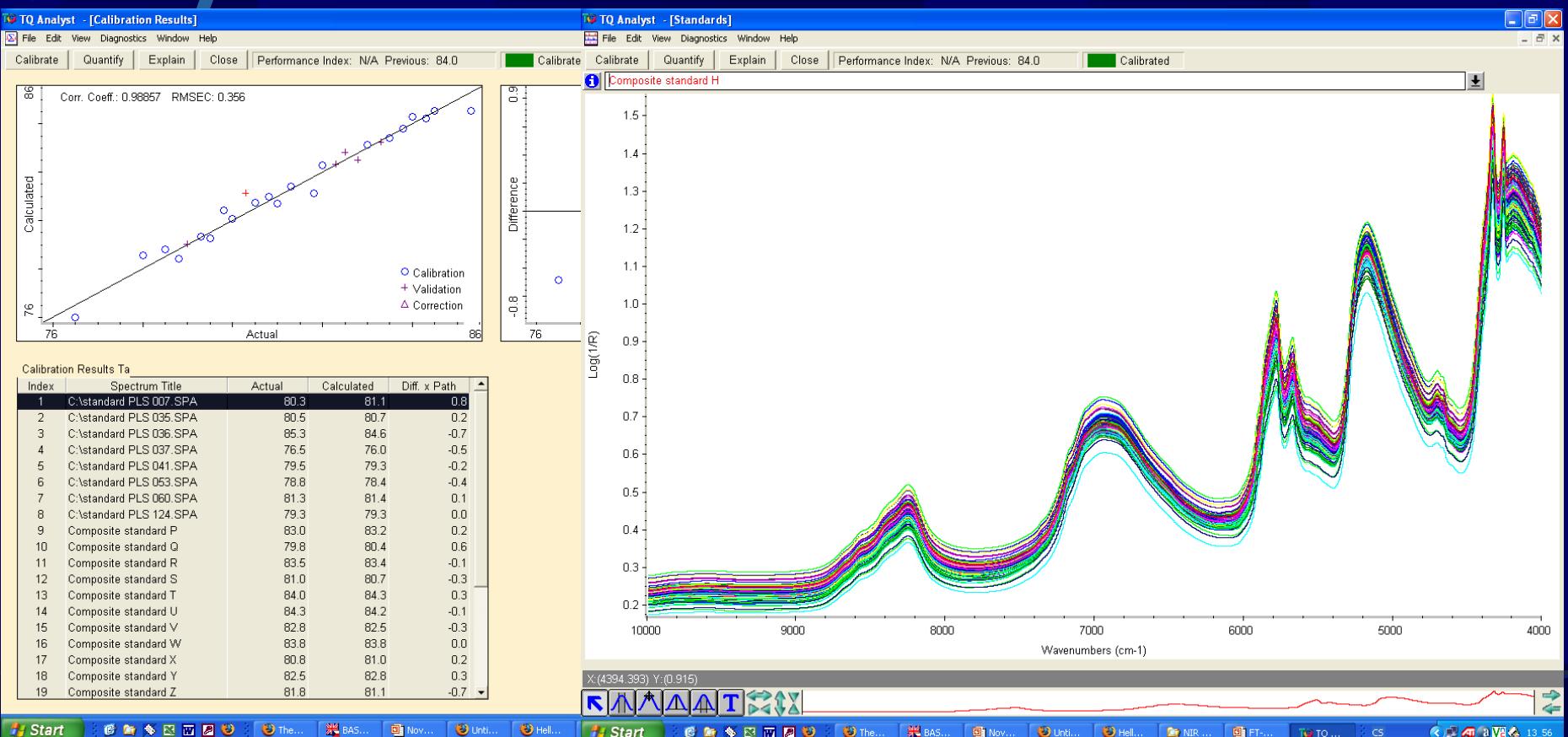
- various pathlengths
- transmission/reflection
- viscous liquids, pastes



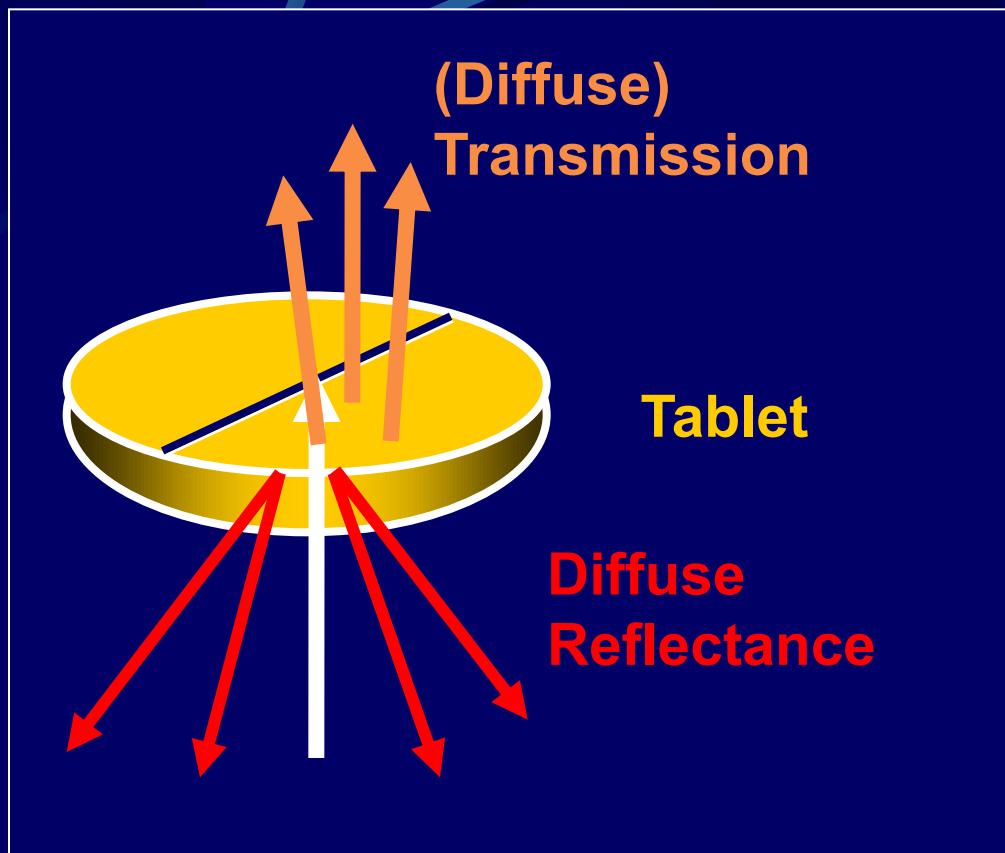
MIRRORS

NIR spectrometry – transreflectance measurement

- ❖ transreflectance cells
 - fat in the butter

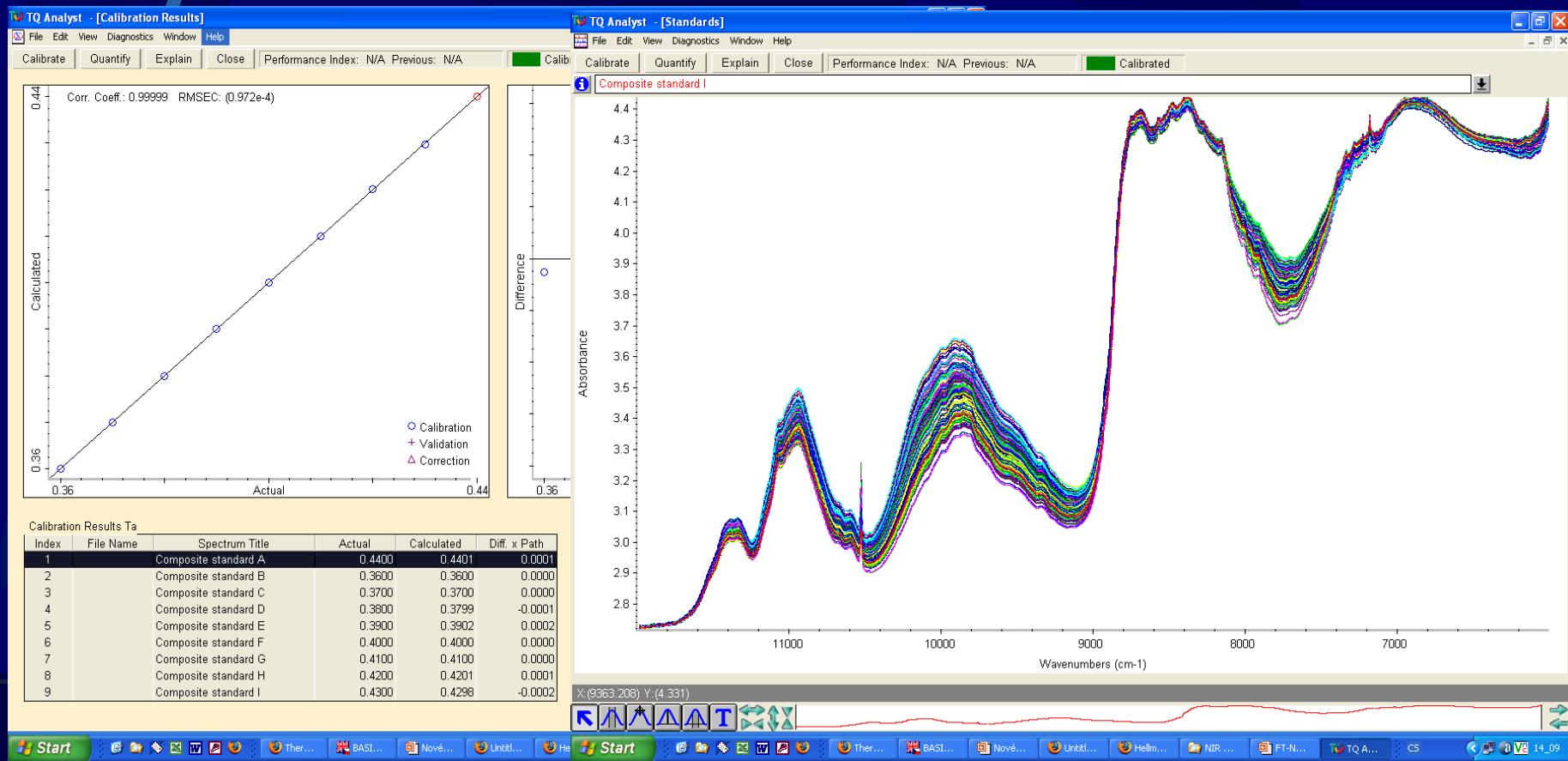


NIR spectrometry – TABLET Analyzer



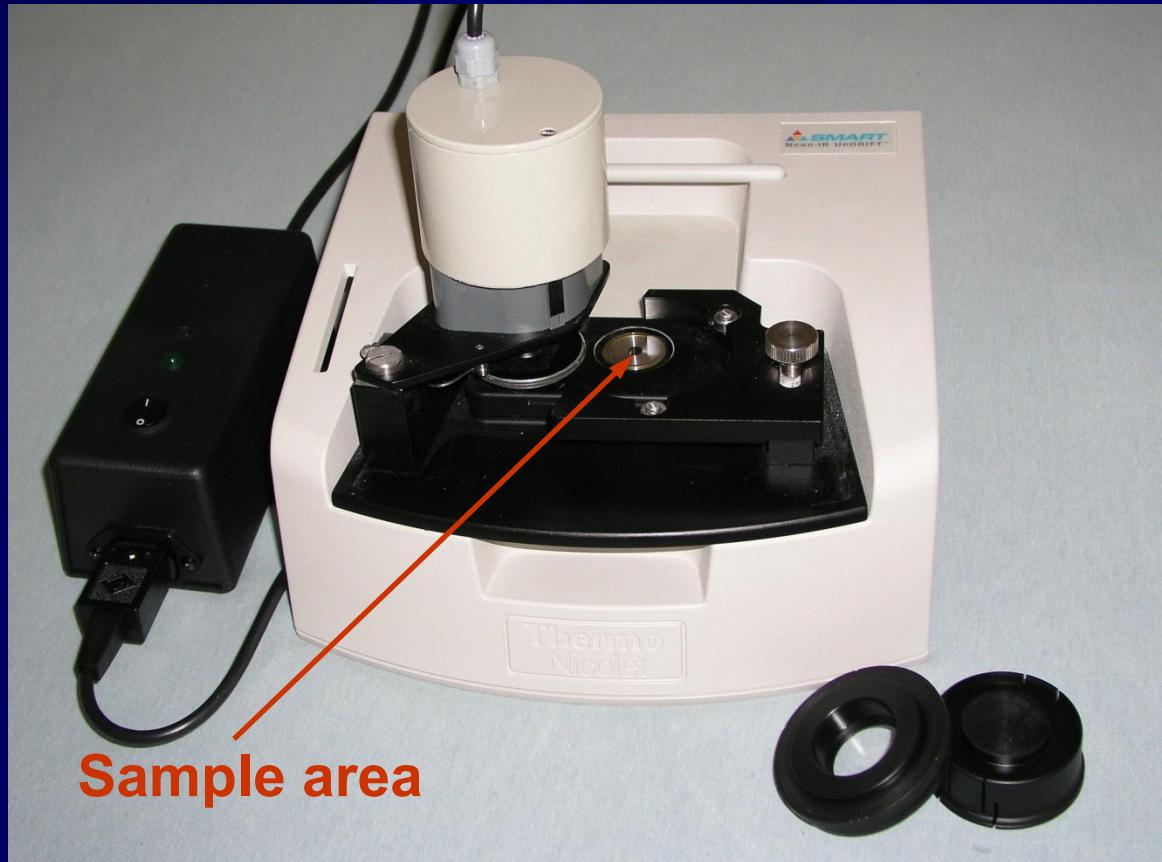
NIR spectrometry – TABLET Analyzer

- ❖ tablet analyzer
 - determination of active substance in a capsule



NIR spectrometry – diffuse reflectance measurement

- ❖ UpDRIFT
- ❖ absorption and reflections on irregular particles
 - reflected radiation collected



NIR spectrometry – diffuse reflectance measurement

❖ UpDRIFT

- background measurement with Spectralon (ceramics)
- direct measurement of pellets, powders ...
- measurement in rotational cell (glass bottom) – powders, granular materials, pulps ...



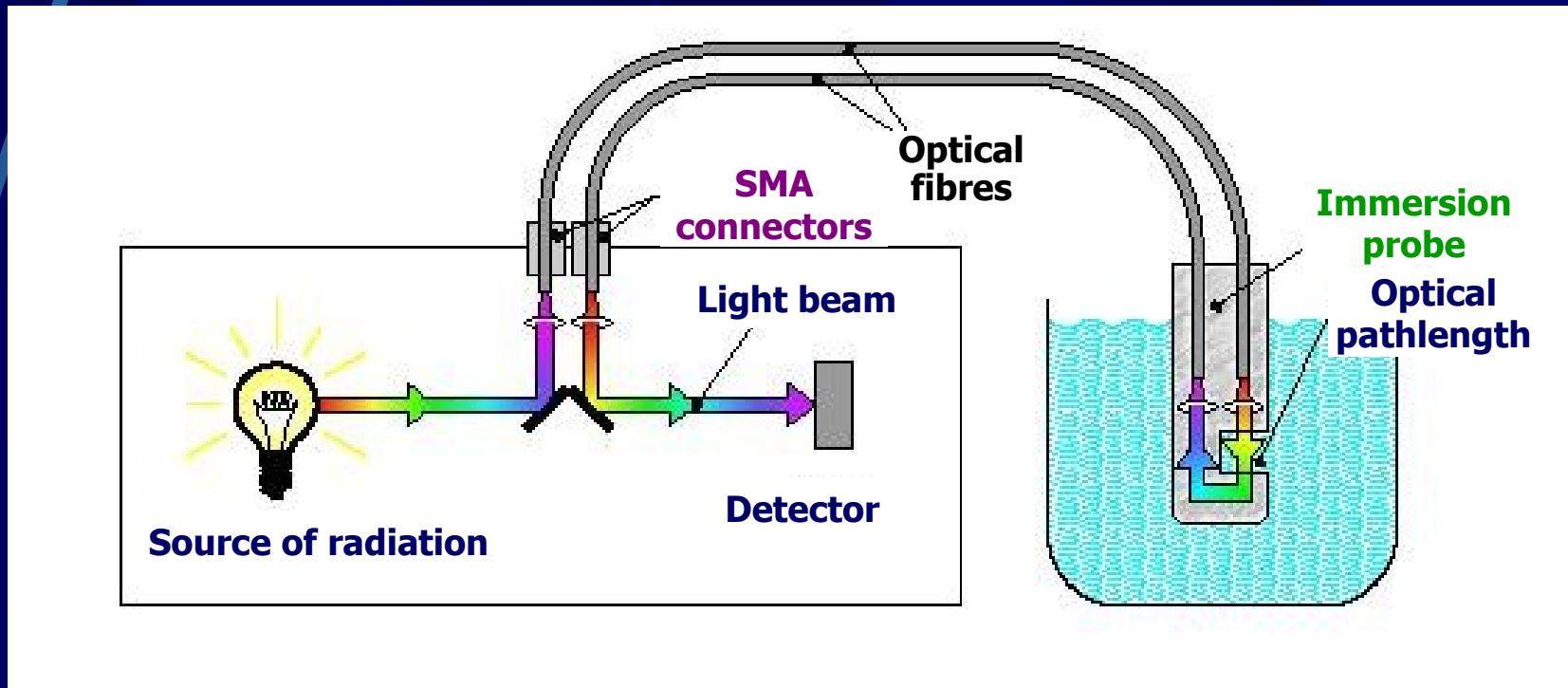
NIR spectrometry – fibre optics probes

- ❖ remote sensing



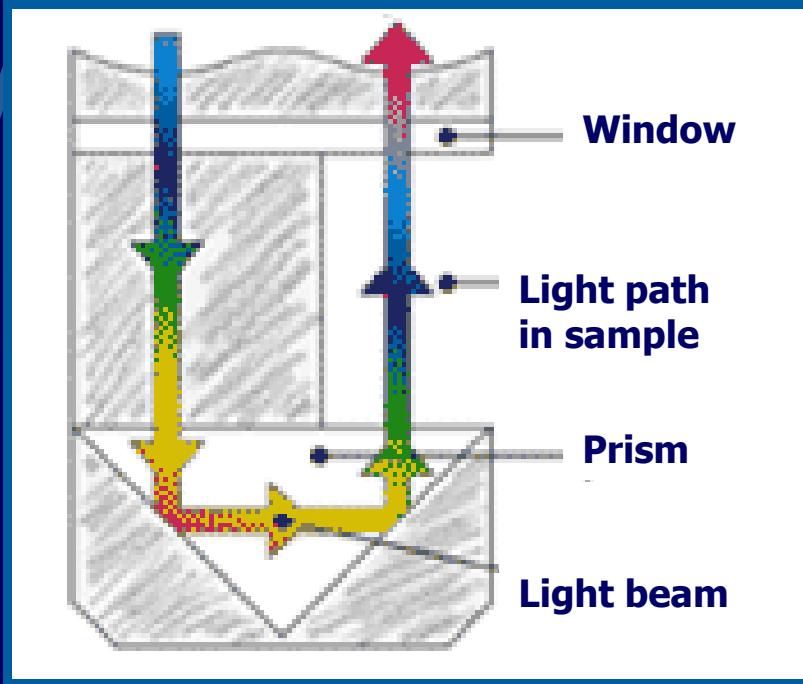
NIR spectrometry – fibre optics probes

- ❖ remote sensing



NIR spectrometry – fibre optics probes

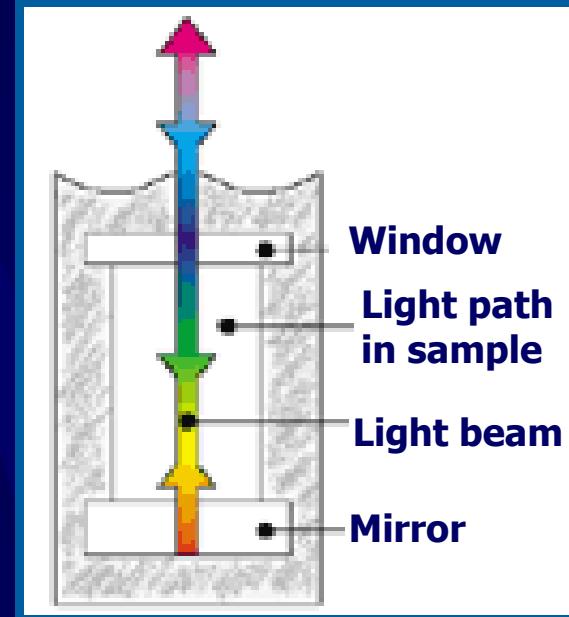
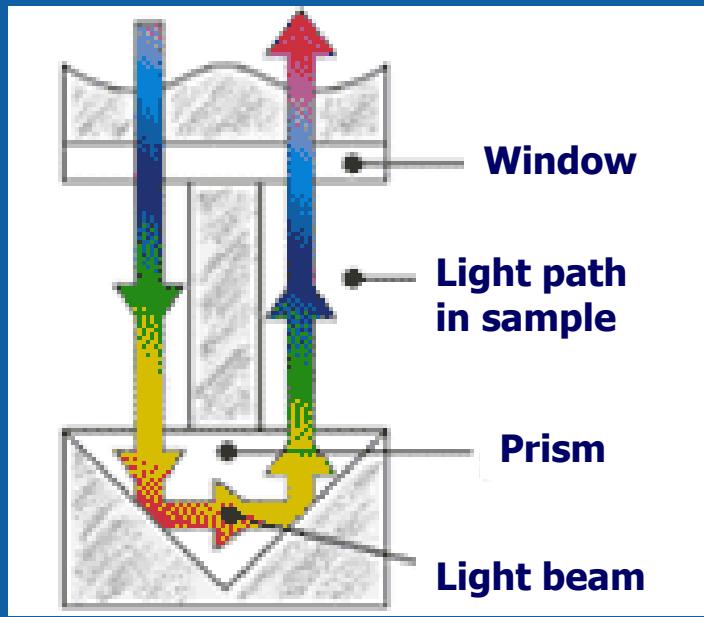
- ❖ IMMERSION PROBES
 - standard type



NIR spectrometry – fibre optics probes

❖ IMMERSION PROBES

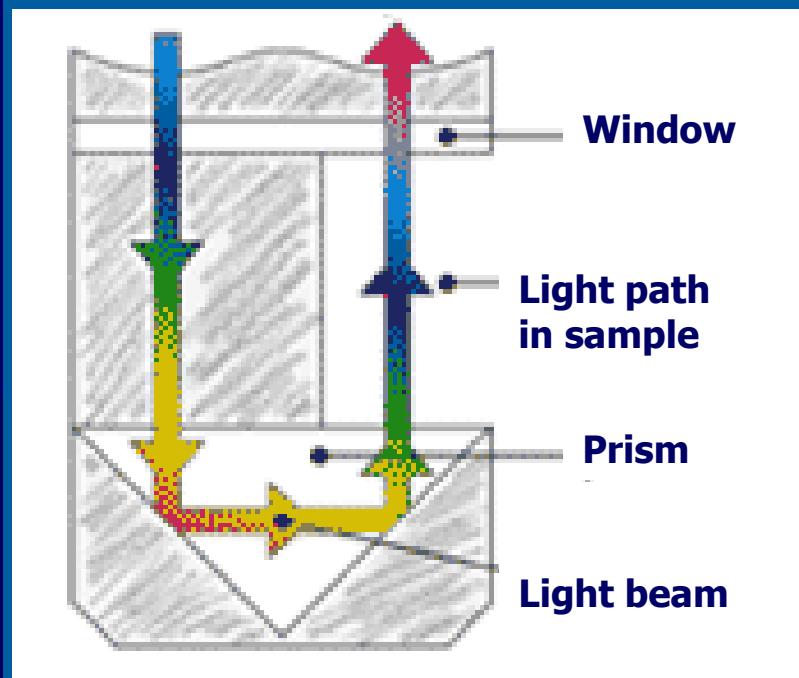
- types for small sampling volumes



NIR spectrometry – fibre optics probes

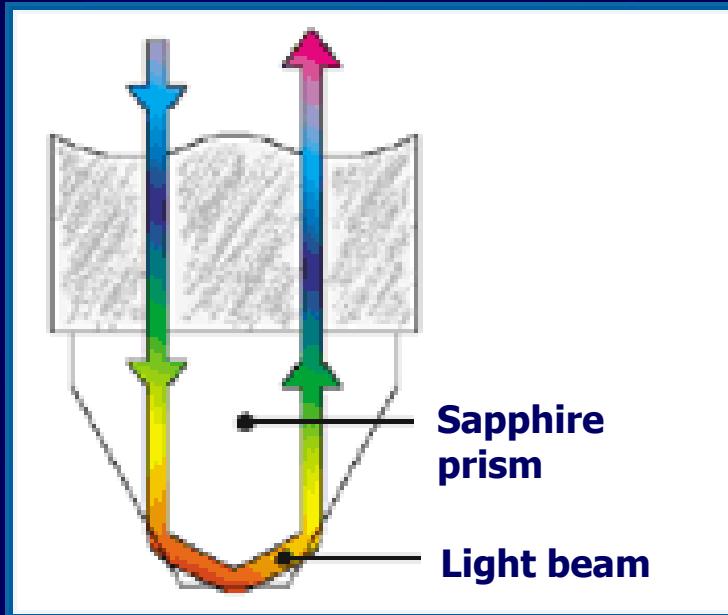
❖ IMMERSION PROBES

- types for process analysis



NIR spectrometry – fibre optics probes

- ❖ IMMERSION PROBES
 - ATR probe



NIR spectrometry – fibre optics probes

❖ Multiplexer System



NIR spectrometry – some practical applications

❖ ANALYSIS OF FOODS

- MILK, CHEESES, SOFTDRINKS, WINES etc.

❖ ANALYSIS OF MEDICAMENTS

- active substances in tablets, plant extracts etc.

❖ ANALYSIS OF POLYMERS

- quality control of products, additives etc.

❖ ANALYSIS OF PETROCHEMICAL PRODUCTS

- content of aromates, octane number etc.

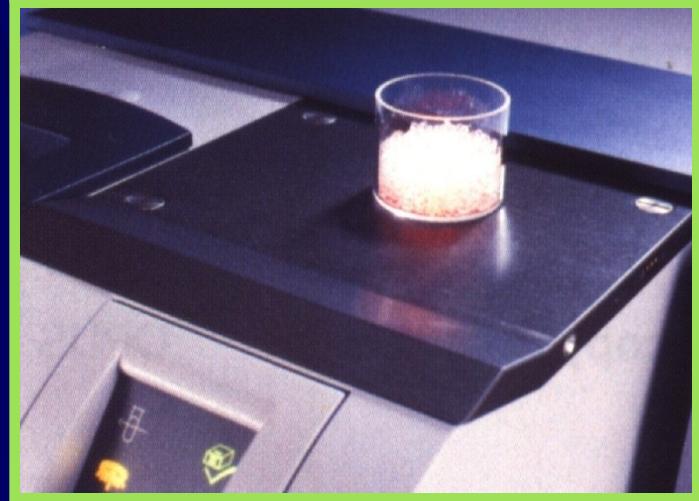
NIR spectrometry – instrumentation Nicolet



ANTARIS

NIR spectrometry – instrumentation Bruker

MPA - multi-purpose analyzer



NIR spectrometry – instrumentation

various probes for technologies

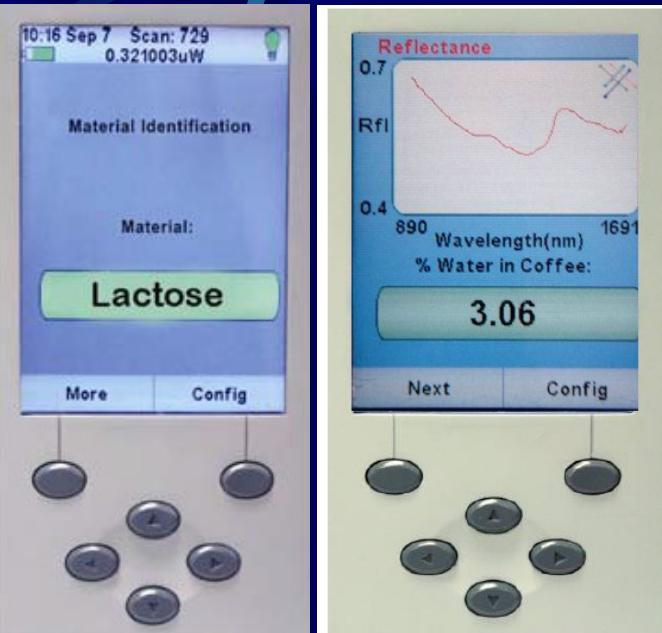




microPHAZIR Rx

Handheld Pharmaceutical Material Analysis

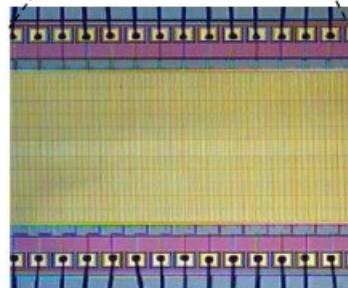
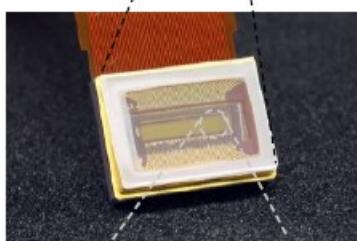
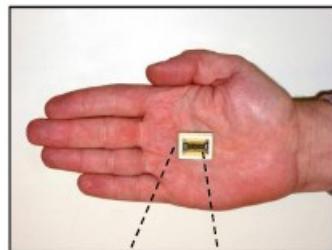
The microPHAZIR™ Rx is the world's first handheld analysis system designed for rapid on-site pharmaceutical material identification and analysis.

- 
- Diffuse reflectance, optional adapters for liquids
 - Tungsten light bulb, safe for operators and sample integrity
 - Measurement time – several seconds
 - 1600 – 2400 nm (cca 6250 – 4160 cm^{-1}) including the complete near-infrared combination region and first overtone region) – wolfram bulb – light source
 - **Resolution** – 8 nm per **pixel** / 12 nm optical

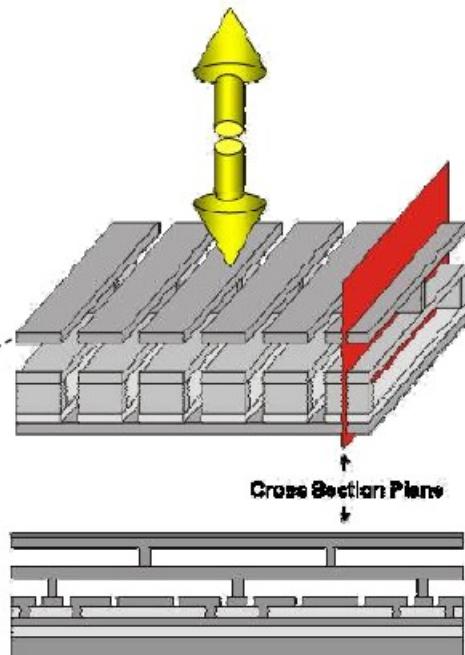


microPHAZIR Rx

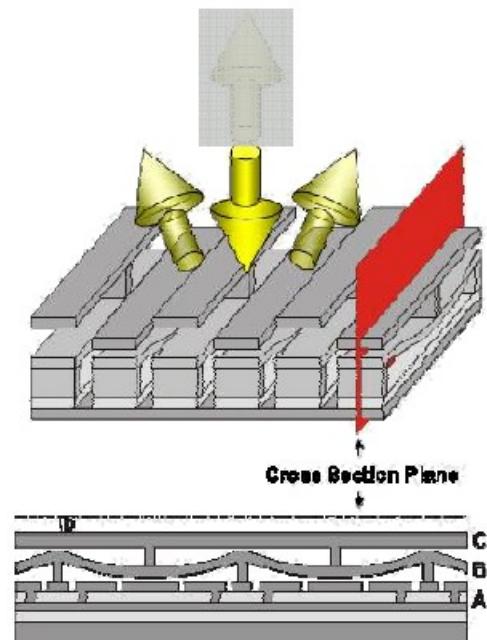
Miniature Spectrometer-on-chip spectrometers



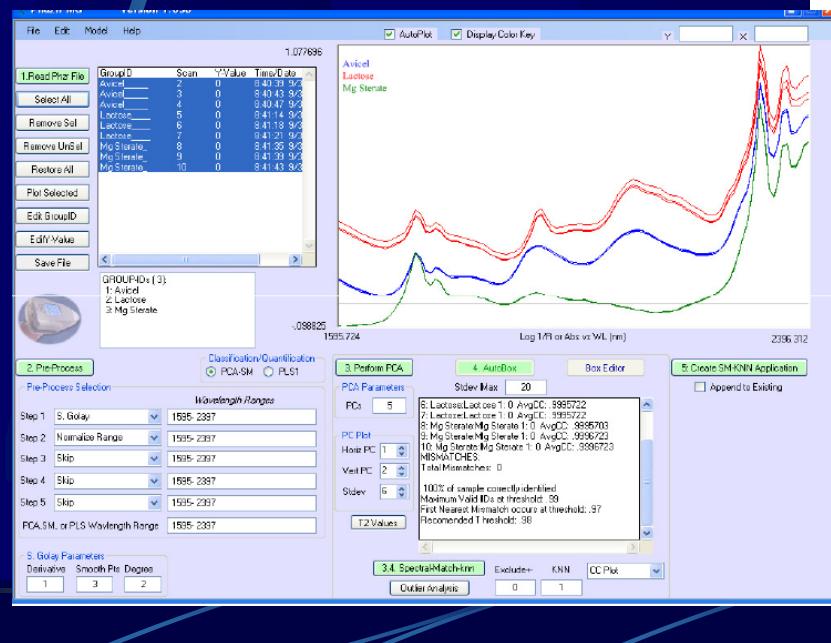
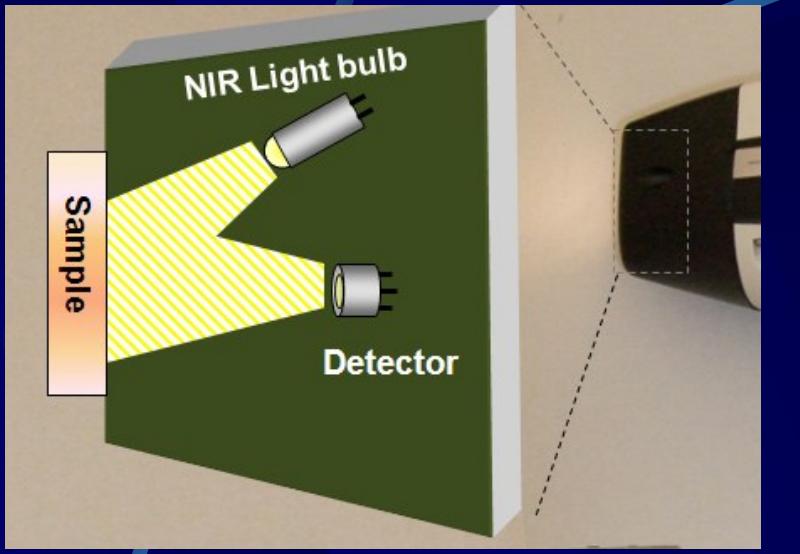
Unactuated: incident light reflected



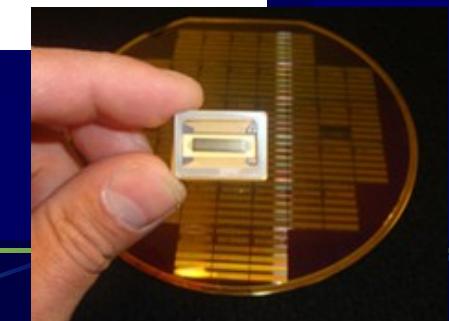
Actuated: programmable filtering
at 1/4- wavelength displacement



microPHAZIR Rx



- Handheld NIR for Pharmaceutical RMID
- Used in 17 of the top 22 largest pharmaceutical manufacturing companies
- Deployed in over 25 countries
- Applications for at-line analysis
 - Blending
 - Drying
 - Coating
 - Tableting
 - Dispensing



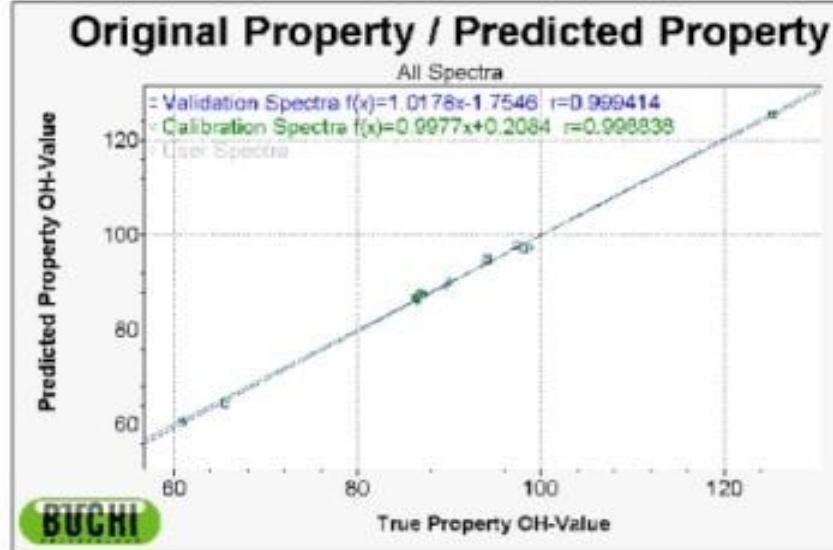
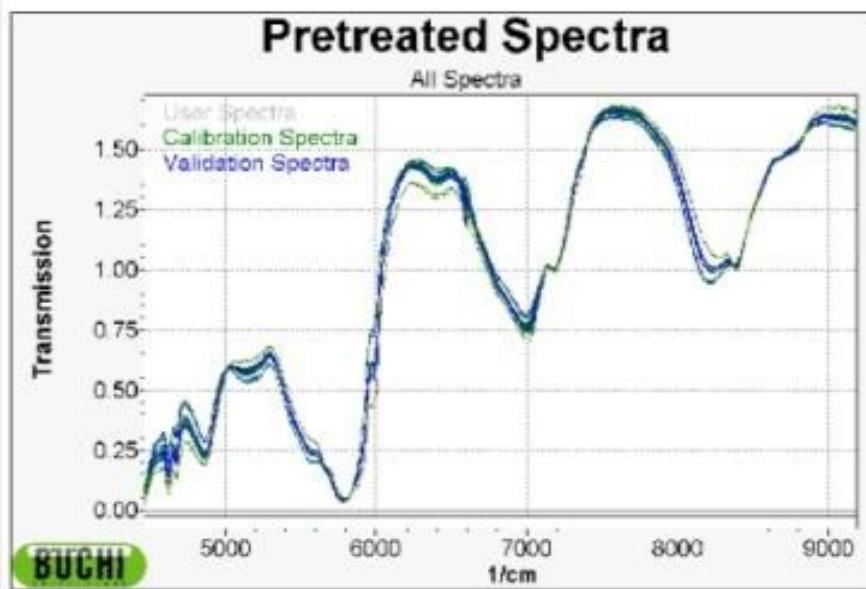
NIR spectrometry – practical examples

Determination of the OH value of Petrochemicals

nirvis

Task:

Determination of the OH value in petrochemical products.



Result

Application works with SEP of 0.79

Measurement Technique

Transmittance: GC vials in the kuvette channel,
3 Scans.

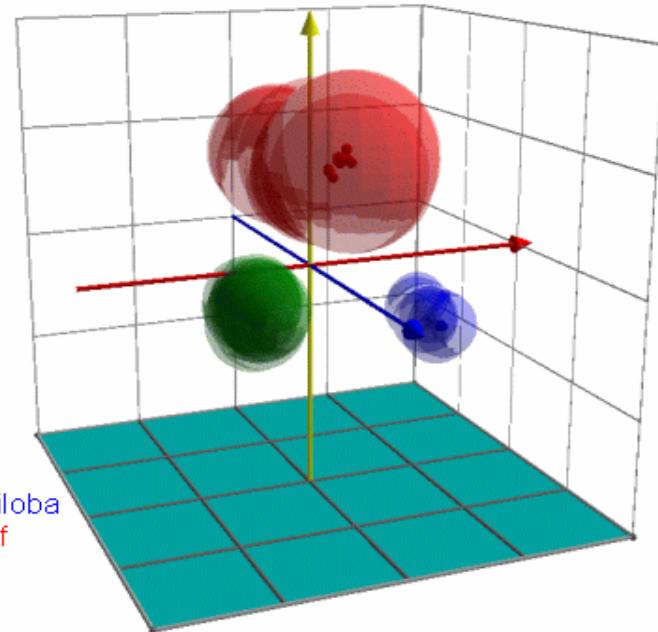
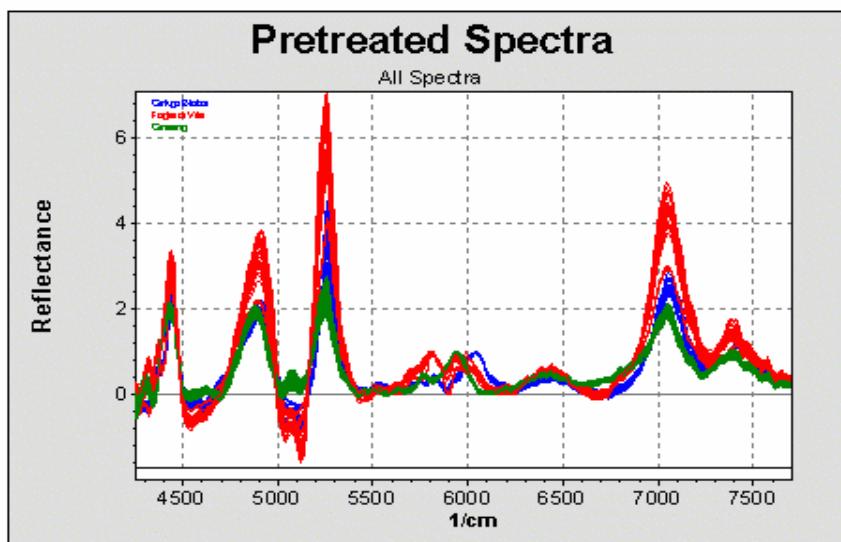
NIR spectrometry – practical examples

Raw Material Testing: Plant Extracts

NIRFlex N-400

Task

Plant Extracts are used as ingredients in natural care products. For QC purposes it is necessary to identify the incoming dried plant extracts of Gingko Biloba, Ginseng and Wine Leaf. One of the expected difficulties is to identify products from different harvests as one property.



Result

Reliable identification of plant extracts possible. Even the products coming from a broad variety of different harvests and regions can be projected into one cluster that is well distinguished from the other products. Therefore NIR can be used to perform a quick QC of Plant Extracts in the warehouse.

Measuring principle
Diffuse Reflectance, 6 Scans

BUCHI

NIR spectrometry – practical examples

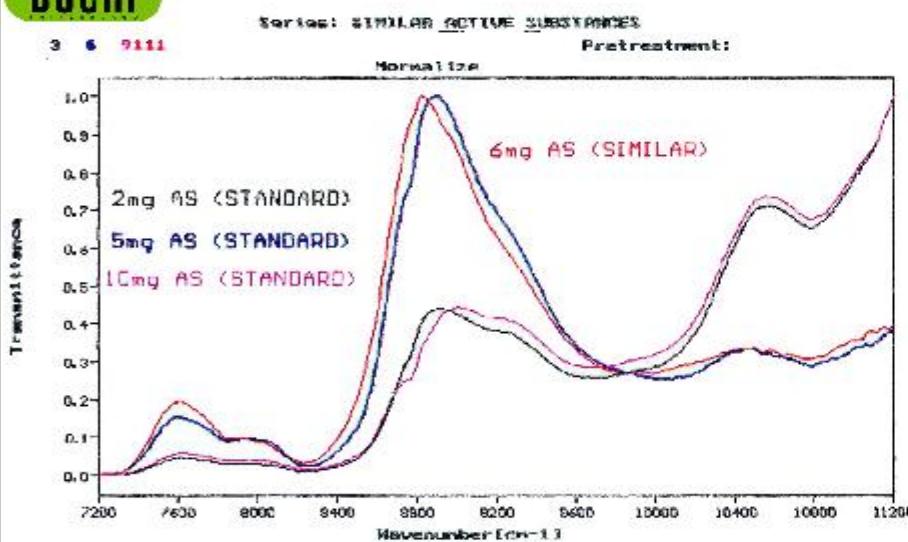
Tablets: active substance

NIRTAB

Task:

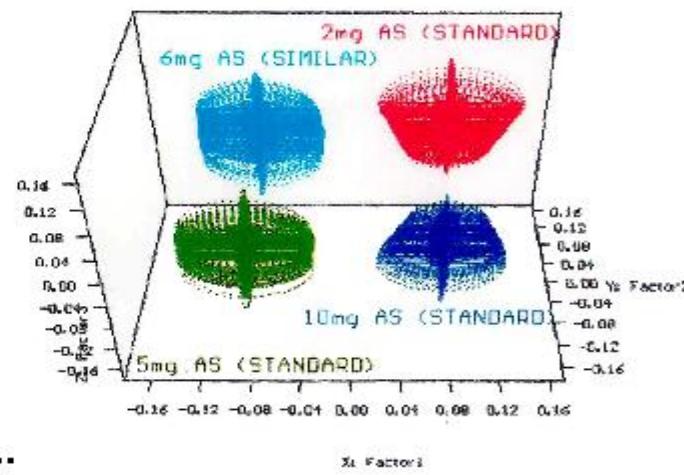
Distinction of tablets with different contents of active substance.

BUCHI



BUCHI

S-Factor Plot for Qualitative Model of SIMILAR ACTIVE SUBSTANCES
Displayed are the 150 spectra in the series



Result:

The distinction is possible.

Measuring principle:

Diffuse transmission (20 scans).

BUCHI