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IN-LINE MEASUREMENT WITH NIR – ADVANTAGES AND LIMITATIONS

Håkan Wedelsbäck, FOSS, hwk@foss.dk

Measurement of Moisture in Materials – Trible M Conference

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CONTENT

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- Typical NIR food/feed applications
- FOSS NIR Instruments
- Molecules, vibrations, overtones
- Spectral regions
- Instrument design
    - Sample presentation
    - Spectrometer configurations
- In-line moisture measurement example
- How to verify performance
- NIR advantages and disadvantages

THE BEGINNING

Sir William Herschel
1800

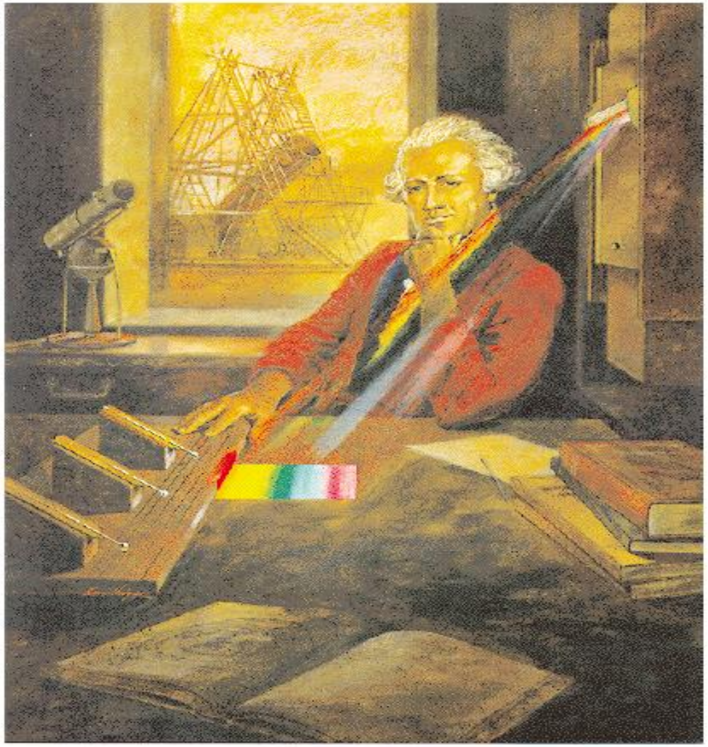


Figure 1. Portrait of Sir William Herschel, who discovered the existence of infrared radiation in 1800. (Picture credit: NASA/IPAC; http://www.ipac.caltech.edu/Outreach/Edu/herschel.gif)

HISTORY

Karl Norris
USDA ARS Beltsville

1800 The First NIR Spectrum recorded (Herschel)

1950 - Potential of NIR quantitative analysis was recognized (Kaye)

1960 - Research program at USDA for NIR analysis of agricultural commodities (Norris)

1970 - First commercial NIR Instruments – Reflection, optical filters, basic mathematics
Improvements in optics and electronics
Introduction of computerized instruments

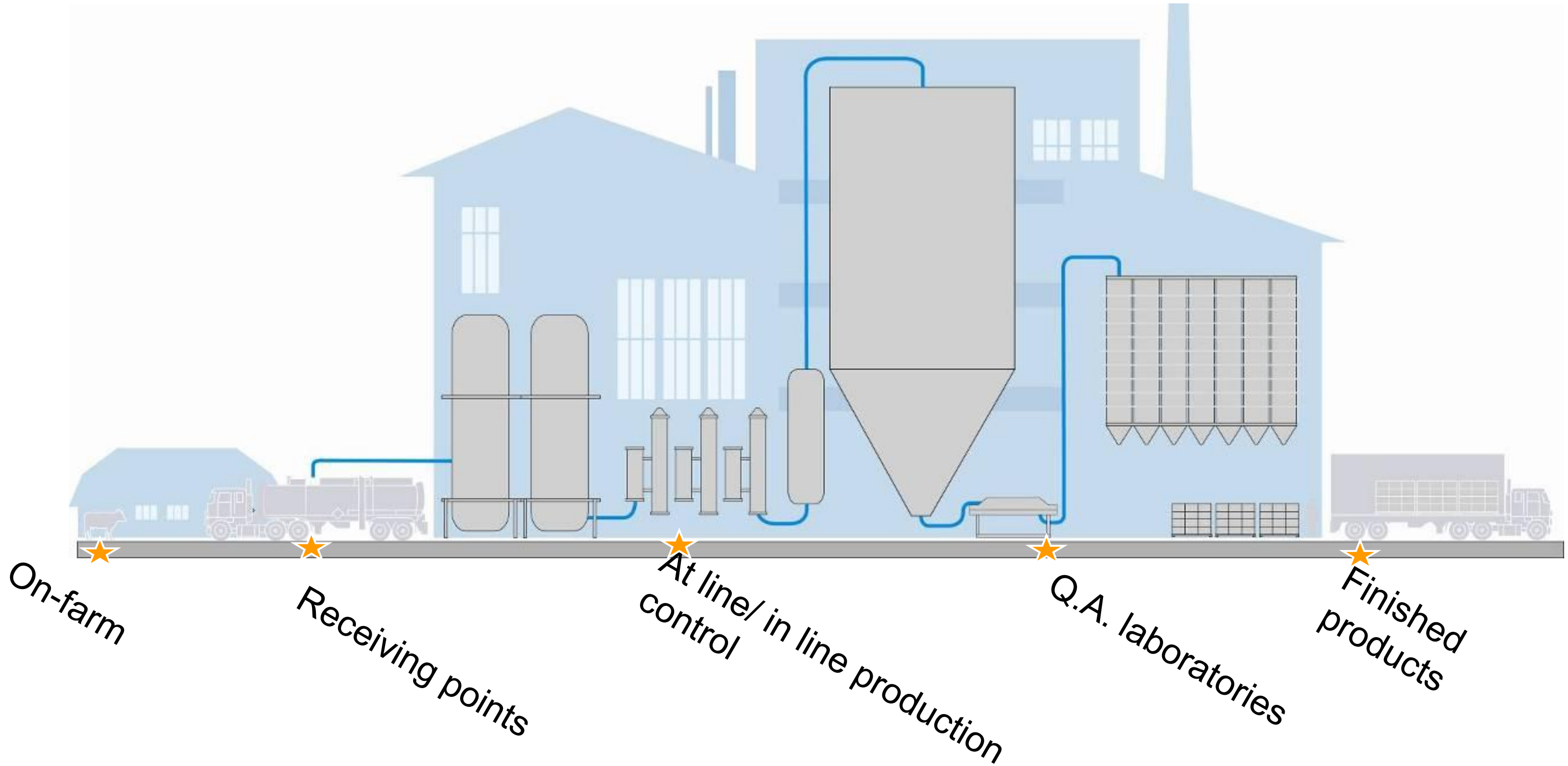
1980 - Continuously scanning spectrophotometer
New calibration techniques (PLS)

1990 - Non-linear calibration methods (ANN)
Instrument Networking

2000 - Improved Instrument Standardisation
High performance Detector Diode Array

2010 - Spectral standardization methods

WHERE ARE NIR INSTRUMENTS USED ?



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TYPICAL NIR
FOOD AND FEED APPLICATIONS

Commodities

- Cereal grain
- Flour
- Meat
- Dairy food
- Feed
- Forage

Often inhomogeneous samples

Both absorption and light scatter attenuate light.

Constituents

- Protein
- Moisture
- Starch
- Fat
- Fibre
- Sugar
- Collagen

% range concentrations

NH, OH, CH absorption

SOME DIFFERENT FOSS NIR/NIT INSTRUMENTS

Infratec™ Nova Grain Analyzer

FoodScan™ Food Analyzer

NIRS DS 2500

XDS PA

Infratec™ Sofia Grain Analyser

MeatScan

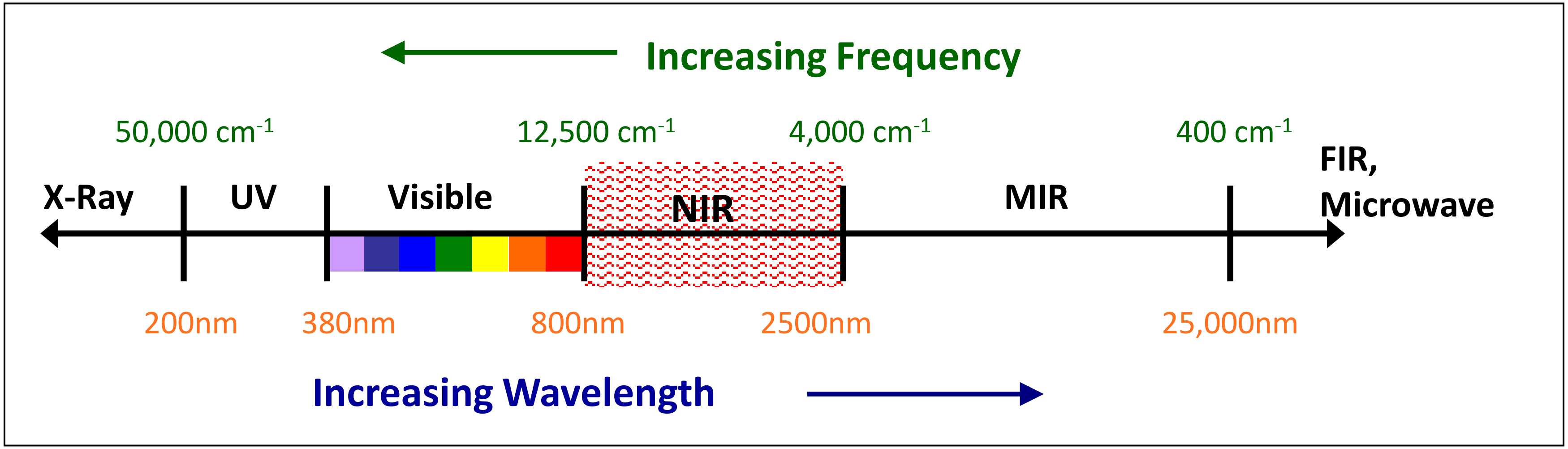
NIRS DA 1650

ProFoss

SPECTRAL REGIONS

NIR just above visible region of the electromagnetic spectrum

~ 750 to 2500 nm



Frequency = 1 / wavelength

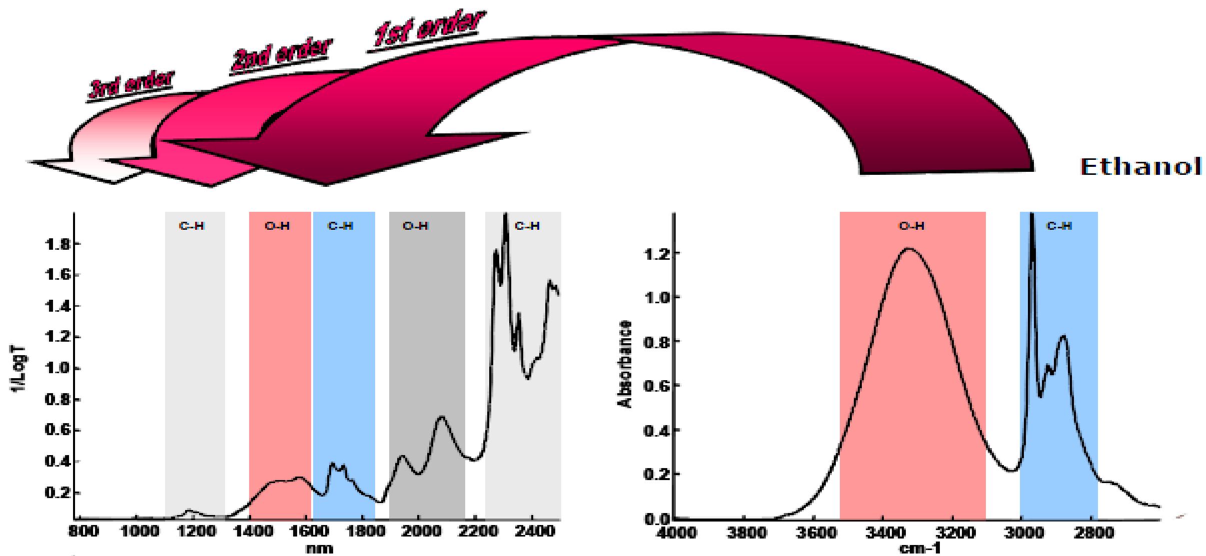
ABSORBANCE BANDS

The absorbance bands observed in the Near Infrared region arise mainly from vibrations of molecules with bound hydrogen atoms.

- CH	FAT
- OH	Moisture
- NH	Protein

NIR SPECTRUM

NIR is repeating IR



Reproduced from D.K. Pedersen, and S.B. Engelsen, Monitoring Industrial Food Processes Using Spectroscopy & Chemometrics, *New Food*, 2 (2001), 9-13

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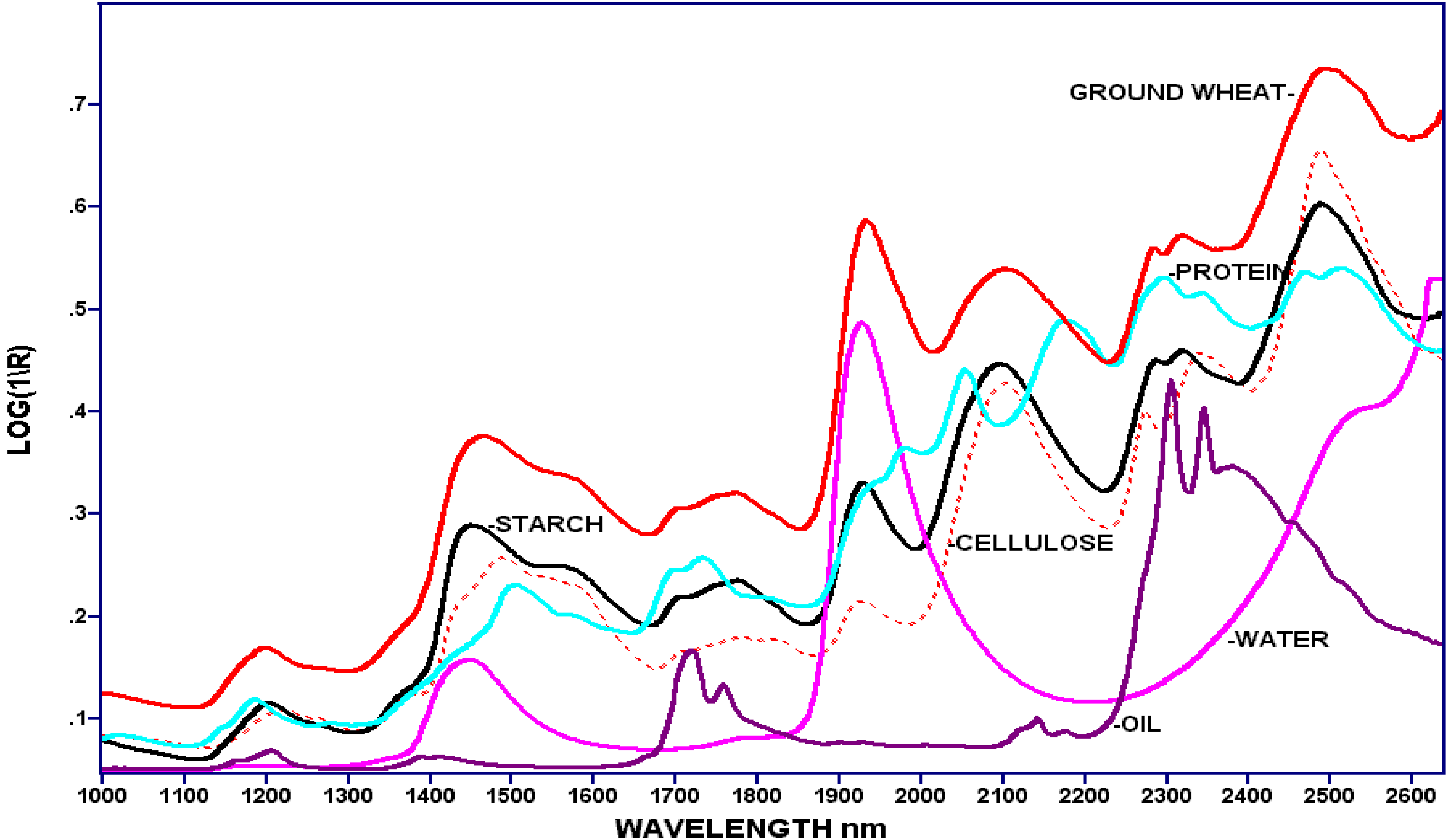
DIFFUSE REFLECTANCE NIR SPECTRA



SAMPLE PENETRATION DEPTH

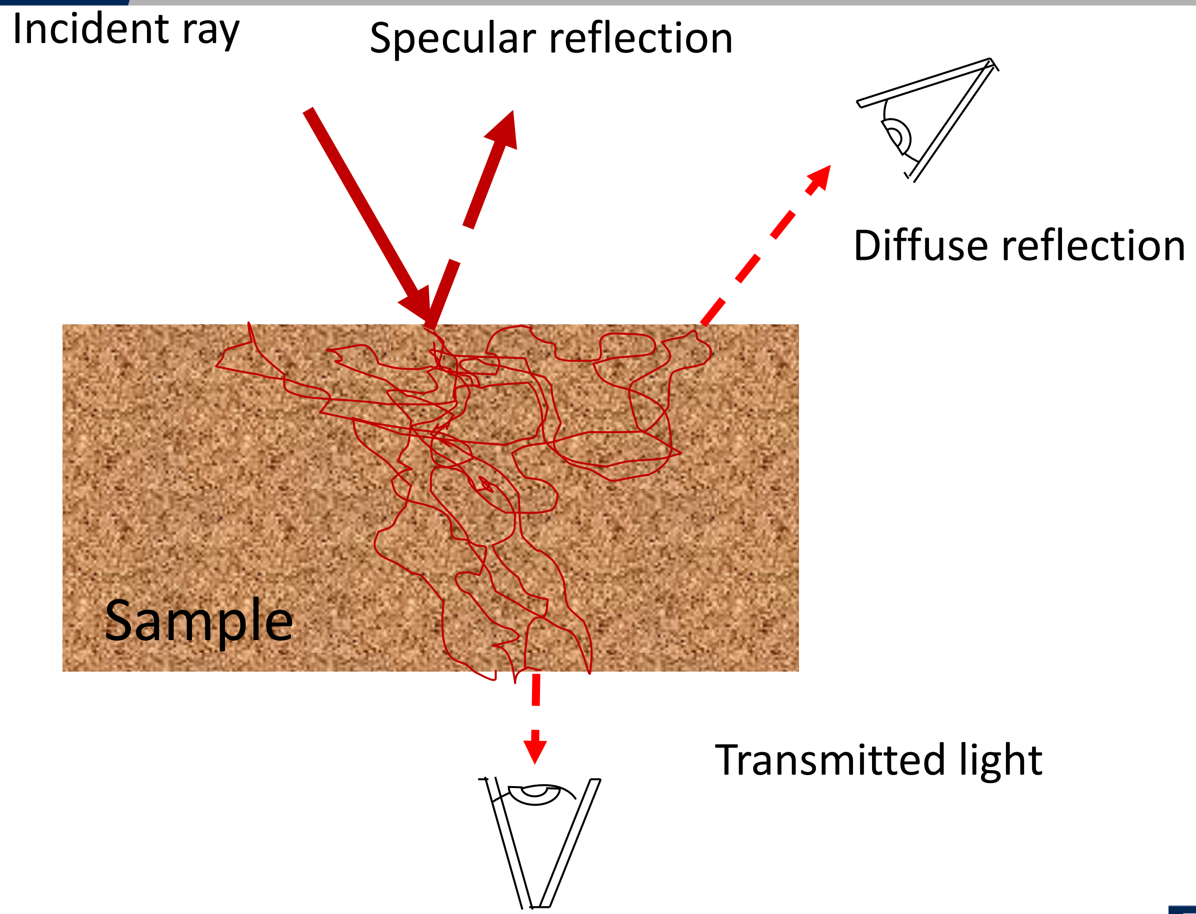
	Wavelength range	Rel. ε	Pathlength
IR	2500 - 25000 nm	1	μm
1'st overtone	1500 - 2000 nm	1/20	mm
2'nd overtone	1100 – 1600 nm	1/500	mm
3'rd overtone	700 – 1200 nm	1/7500	cm

LIGHT SAMPLE INTERACTION

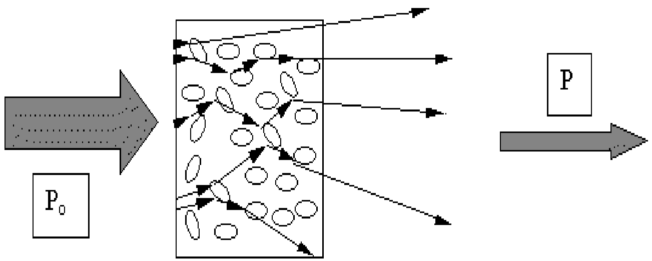
Absorption

AND

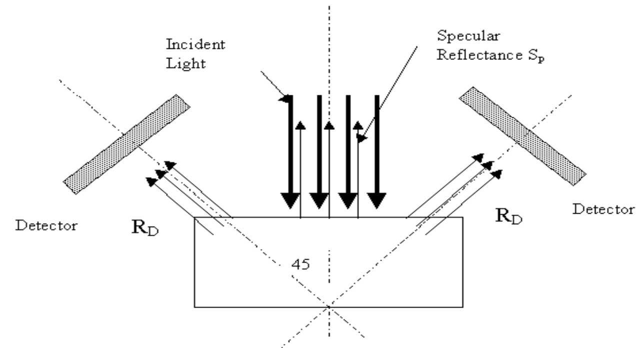
Light Scatter



NIR/NIT SAMPLE INTERFACE



Transmission
6-30mm cell width



Diffuse reflectance

(Transflectance with diffusing mirror behind)

WAVELENGTH SELECTION TECHNOLOGIES

Filter

- Interference filters
- Acousto-Optic Tunable Filter (AOTF)
- Scanning Fabry-Perot Filter
- Linear variable filter

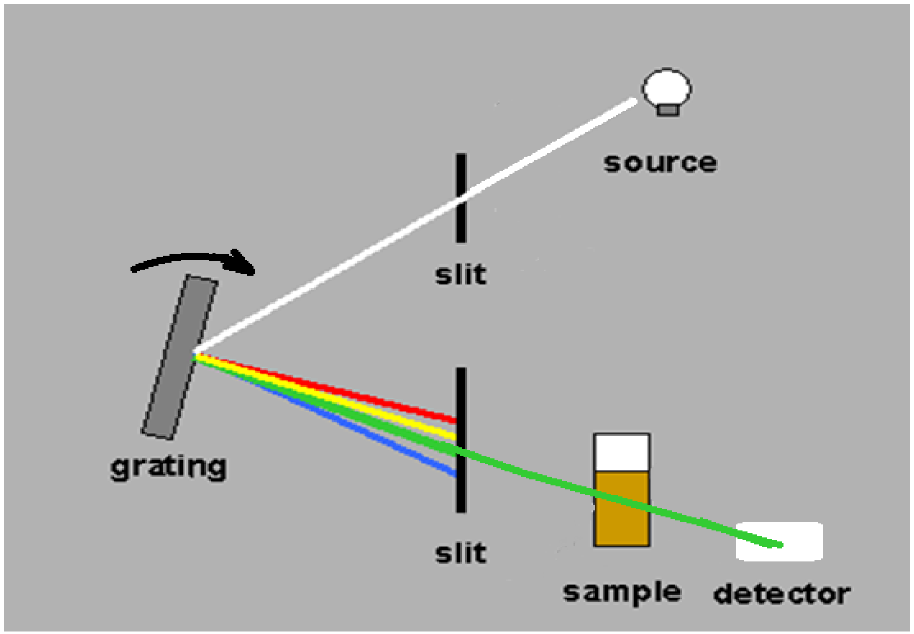
LED/Laser array

Dispersive

- Grating ← Used in FOSS NIR instruments
- Prism

Interferometric (FT-NIR)

PRE DISPERSIVE SCANNING MONOCHROMATOR



- Single Detector
- Rotating Grating
- Monochrome sample illumination

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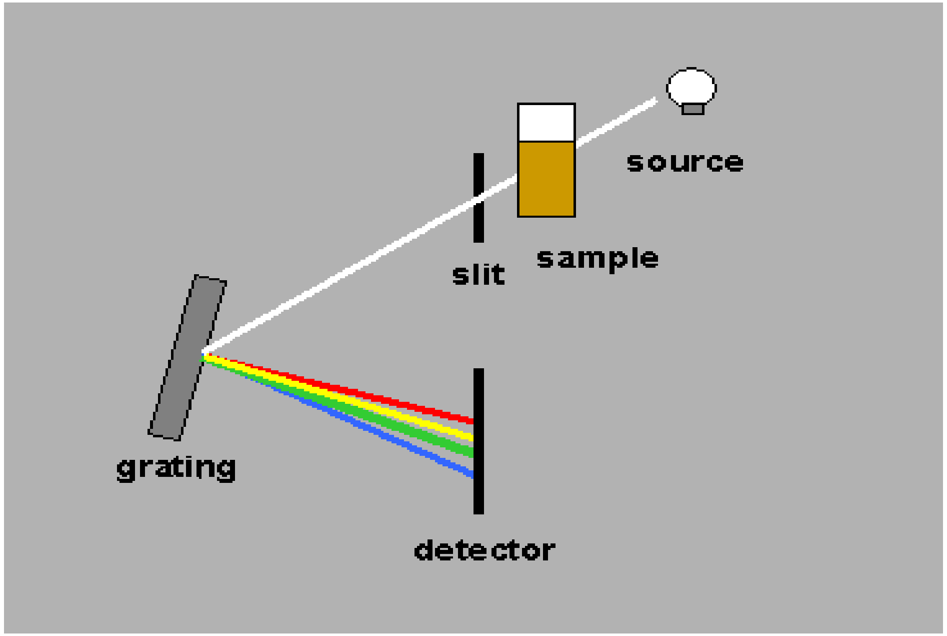
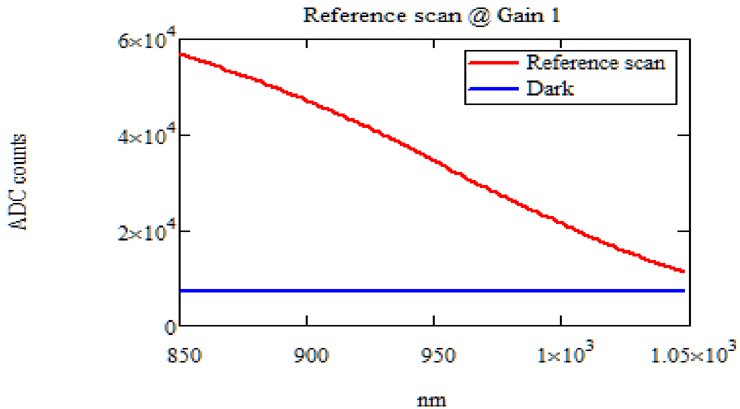
POST DISPERSIVE DETECTOR DIODE ARRAY

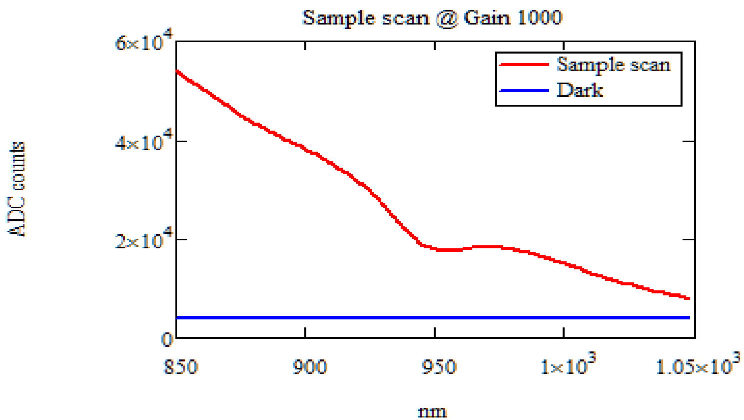


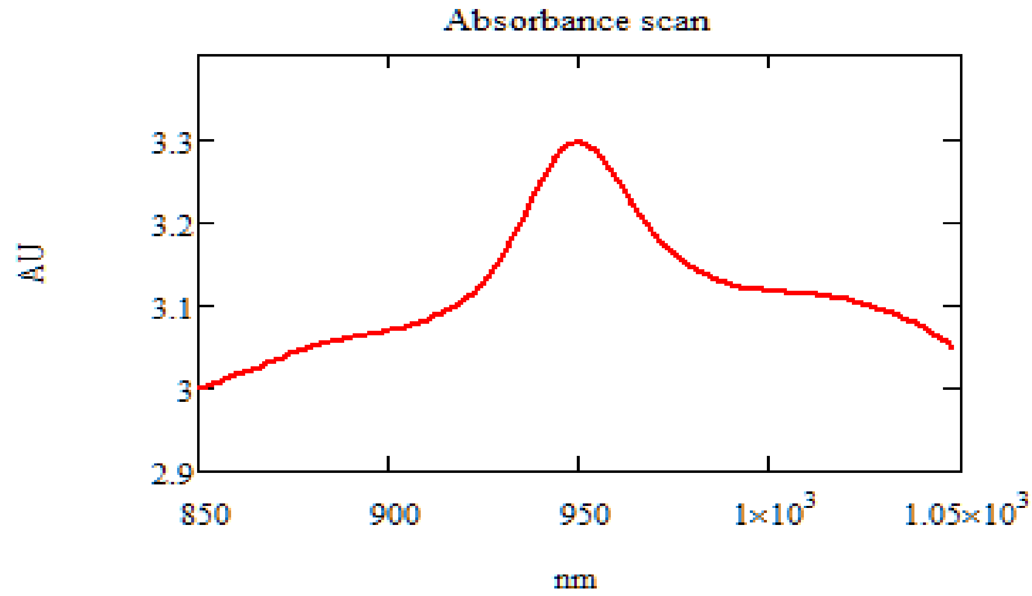
- Multiple Detectors
- Fixed Grating
- Polychrome sample illumination

ABSORBANCE SCAN CALCULATION

$$\text{Absorbance} = \log(\,(\text{Ref} - \text{Ref\_offset}) / (\text{Sample} - \text{Sample\_offset})\,) + \log(\text{Sample\_gain} / \text{Ref\_gain})$$







The reference scan correct for source, detector and grating characteristics and compensate for thermal y-axis drift.

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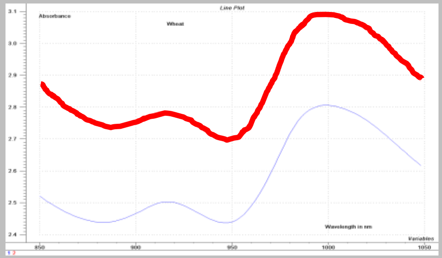
A DEDICATED SOLUTION

Instrument

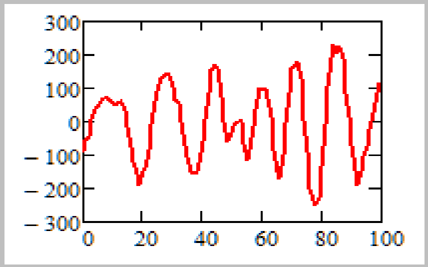
Result

Sample Presentation

Spectrometer



Prediction model
(Calibration)



x% Moisture

$$x=b_0+A_{\lambda 1}*b_1+ A_{\lambda 2}*b_2+ A_{\lambda 3}*b_3+ \ldots\ldots A_{\lambda n}*b_n$$

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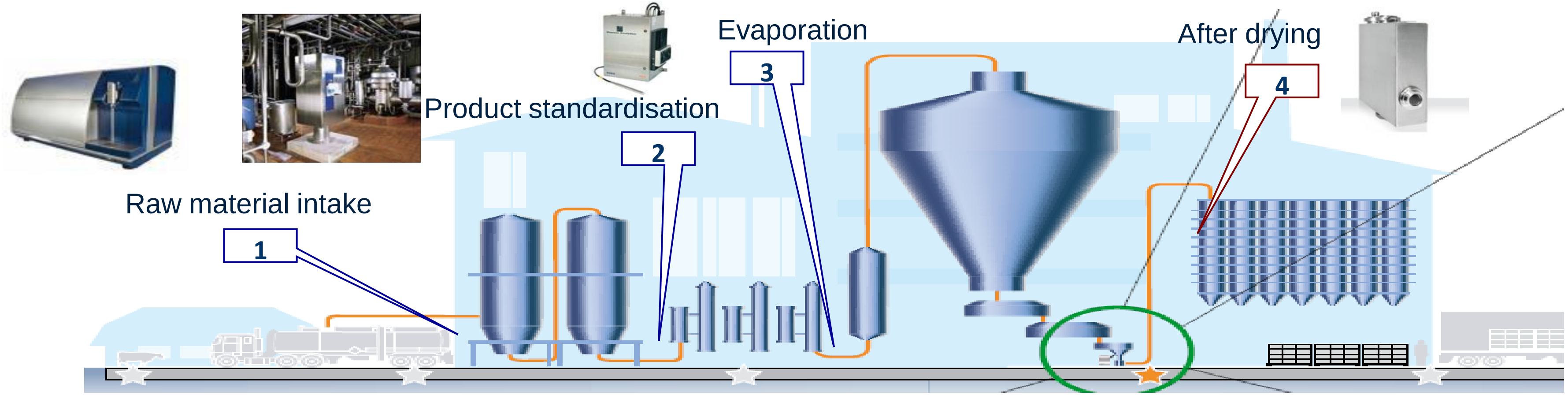
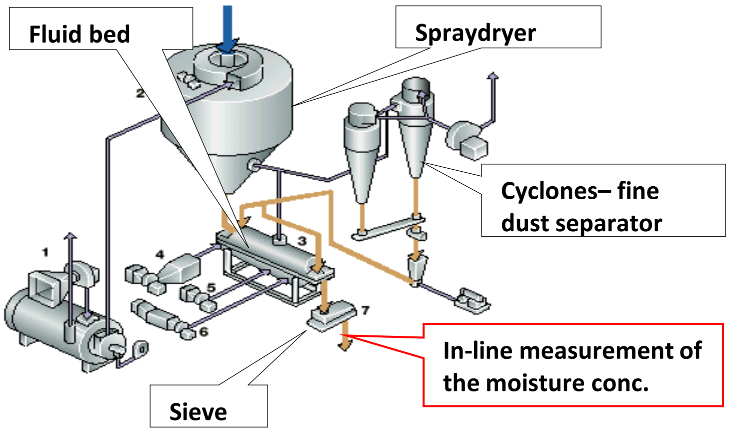
EXAMPLE: MOISTURE MEASUREMENT IN DAIRY POWDER PRODUCTION

DAIRY POWDER PROCESS



1. Milkoscan: Compositional analysis of incoming raw milk
2. ProcesScan: Standardization of fat and protein concentration in liquid phase
3. XDS PA: Analysis of concentrate - TS is as high as possible to save energy
4. ProFoss: Analysis of dry powder – Moisture measurement for process control, Fat & Protein for monitoring

THE MEASURING POINT



Measuring Point

- Installed in the outlet of the fluid bed & sieve in a place where the powder is free falling.
- Measurements are made with a reflectance spoon probe installed directly into the hopper or pipe.

Purpose of measurement

- Measure moisture conc. to control both the Fluidbed and the Spray dryer drying process to avoid “over drying”(increase moisture in final product - yield).
- Monitor fat and protein – not used for process control. Fat and protein standardization is done before the evaporation and spray drying process.

TRUE IN-LINE ANALYSIS WITH A DEDICATED DAIRY POWDER SAMPLER

- An optical fiber “Powder probe” is installed in a pipe or hopper right after the secondary drying step (fluid bed/sieve) where powder is free falling – no complicated sample bypass or automatic sampling system.
- The Powder probe is casted in a polymer and has no glass window in between the sample and the optical fiber.
- An air purge system ensures that the sample is completely removed before collecting a new sample for analysis.

DIARY POWDER ACCURACY EXAMPLE

The calibration is based on skim milk powder data collected in-line at the outlet of the fluid bed and sieve. 367 samples was used for the moisture calibration.
N number of independent samples was used for validation.

Component	Model	N	Acc.	Min	Max	RSQ
Moisture	PLS	65	0.10	2.1 %	4.0 %	0.96
Protein	PLS	59	0.27	32.6 %	37.4 %	0.97

N: Number of independent samples in the validation set.
Acc.: Independent test set accuracy expressed as Standard Error of Prediction (SEP) corrected for bias (1 SD absolute)*.
Min.: Minimum reference value.
Max.: Maximum reference value.
RSQ: Linear correlation between ProFoss result and reference result.

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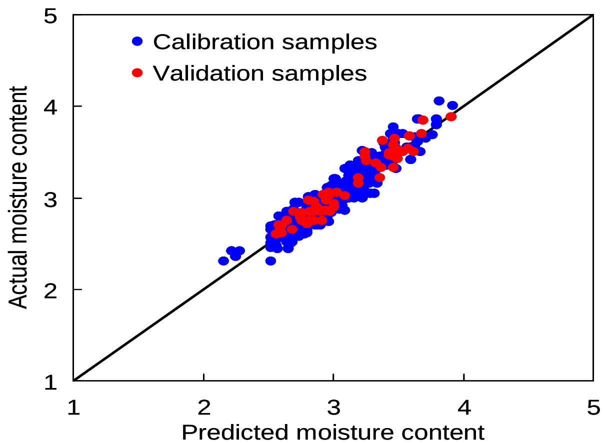
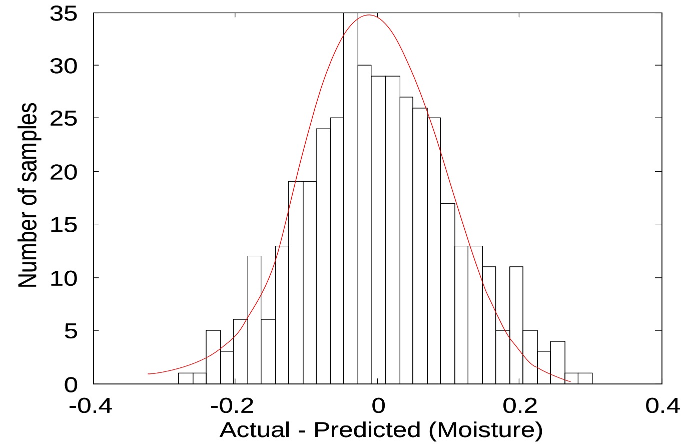
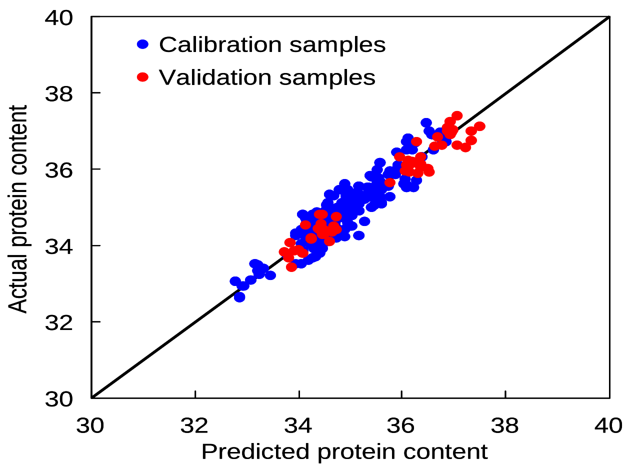
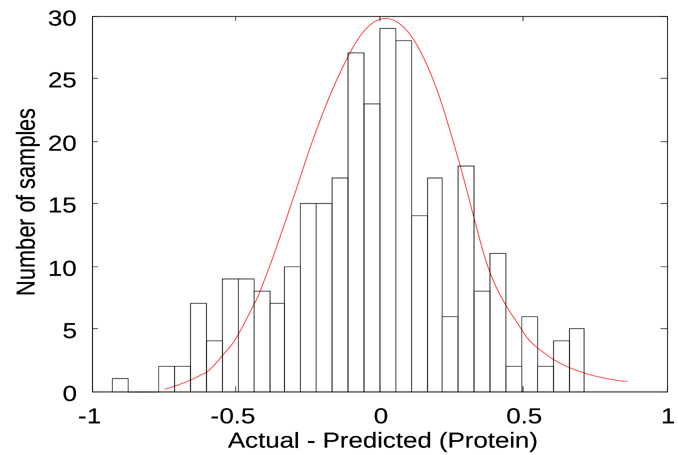
DIARY POWDER ACCURACY EXAMPLE









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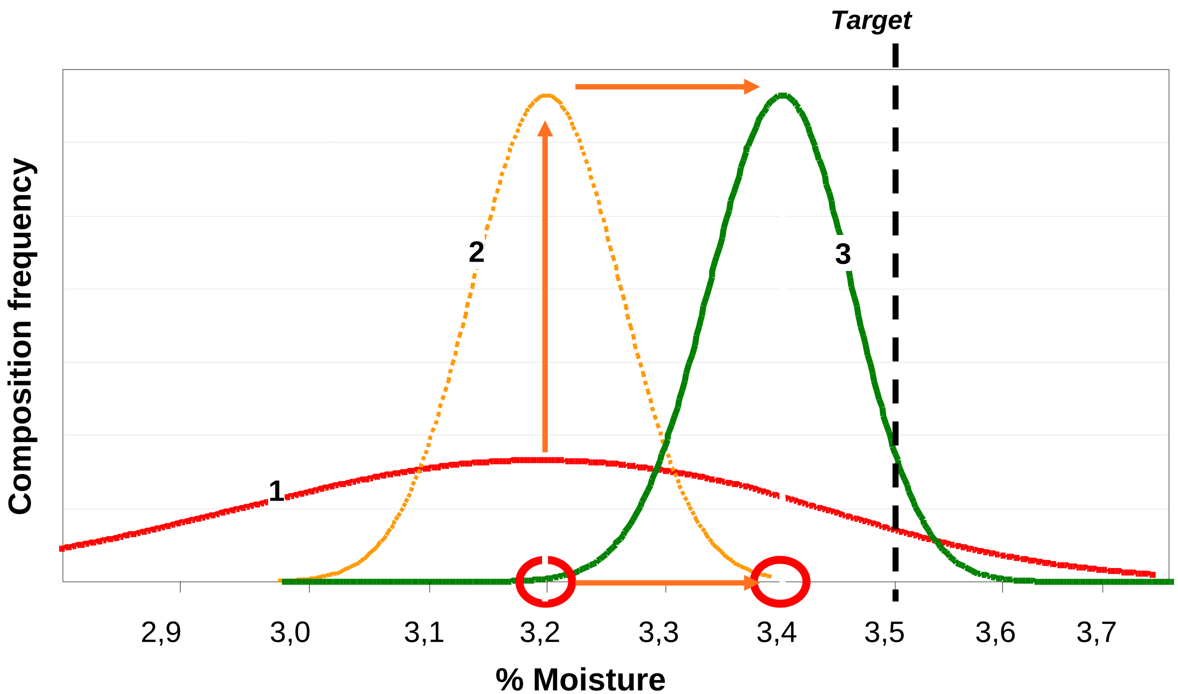
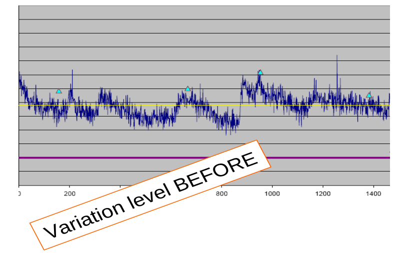
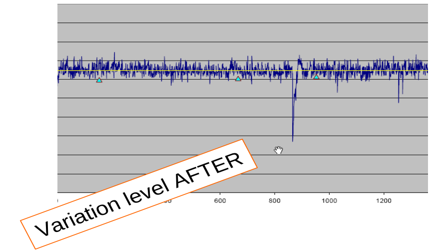
PRODUCING CLOSER TO THE TARGET SPECIFICATION

In-line NIR measurement allows you to run production much closer to your specification limits thus giving both increased yield and improved final product quality.







HOW TO CHECK PERFORMANCE

Independent test set to be analyzed according to the following methods:

Validation ISO21543/IDF 201: 2006.

Milk products – Guidelines for the application of near infrared spectrometry.

Reference methods

Moisture: ISO 5537:2004 / IDF 26 (2004),

Dried milk – Determination of Moisture content (Reference method)

Protein: ISO 8968-1:2001 / IDF 20-1 (2001),

Milk -- Determination of Nitrogen content -- Part 1: Kjeldahl method

ISO 8968-2:2001 / IDF 20-2 (2001),

Milk -- Determination of Nitrogen content -- Part 2: Block-digestion method

NIR
ADVANTAGES AND LIMITATIONS

ADVANTAGES

- High continous sampling rate enables automatic process control.
- Non destructive measurement.
- Good accuracy.
- No sample preparation.
- Can be used with sample presentation units for almost any liquid or solid sample.
- Can measure other sample constituents in addition to moisture (fat/oil, protein, sugar, fiber, etc).
- Can be made insensitive to product temperature.
- Simple to install and maintain.

LIMITATIONS

- Not a primary method.
- Needs to be initially calibrated (generally against a primary method).
- Measurement is made close to sample surface due to limited light penetration depth. To work properly, there must be a relationship between the surface moisture and total moisture of the product.
- A good sampling procedure is essential.
- Optical Path must be kept clean.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