# Applications of FT-NIR
in the Edible Oil Industry

Agenda

- What is Near-Infrared Spectroscopy?

- NIR for Quality Control of Incoming Oils

- Determination of Degradation Products in Frying Oils

- Verification of Olive Oil Quality

- Analysis of Oilseeds

- Process Applications

- Summary

The Discovery of Infrared Radiation
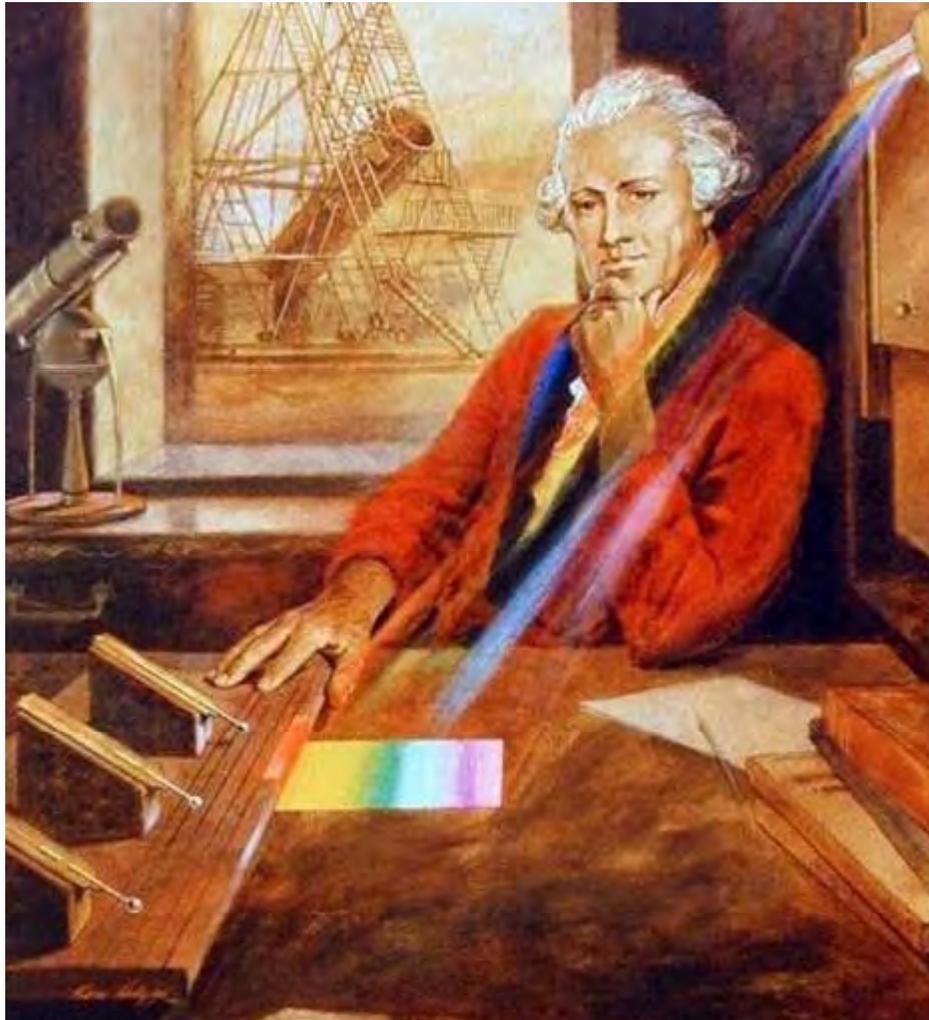
Herschel discovered the temperature rise of thermometers by radiation beyond the red light

↓

Infrared Radiation

Sir William Herschel (1800)

Bruker Optics

The Electromagnetic Spectrum
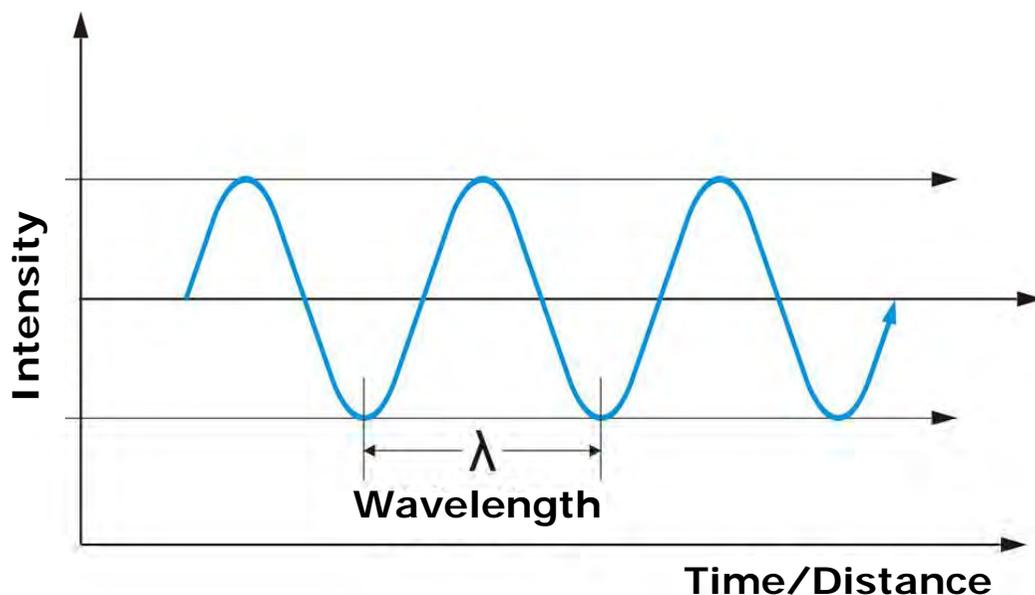
Characterisation:

Wavelength $\quad \lambda$ [nm]
Frequency $\quad \nu = c/\lambda$ [Hz]
Energy $\quad E = h\nu$ [J]

Wavenumber $\quad \nu = 1/\lambda$ [cm^{-1}]

(Diagram: Intensity vs. Time/Distance, sine wave with Wavelength λ labeled)

Intensity

λ

Wavelength

Time/Distance

Infrared light is like the visible light

electromagnetic radiation.

The Electromagnetic Spectrum
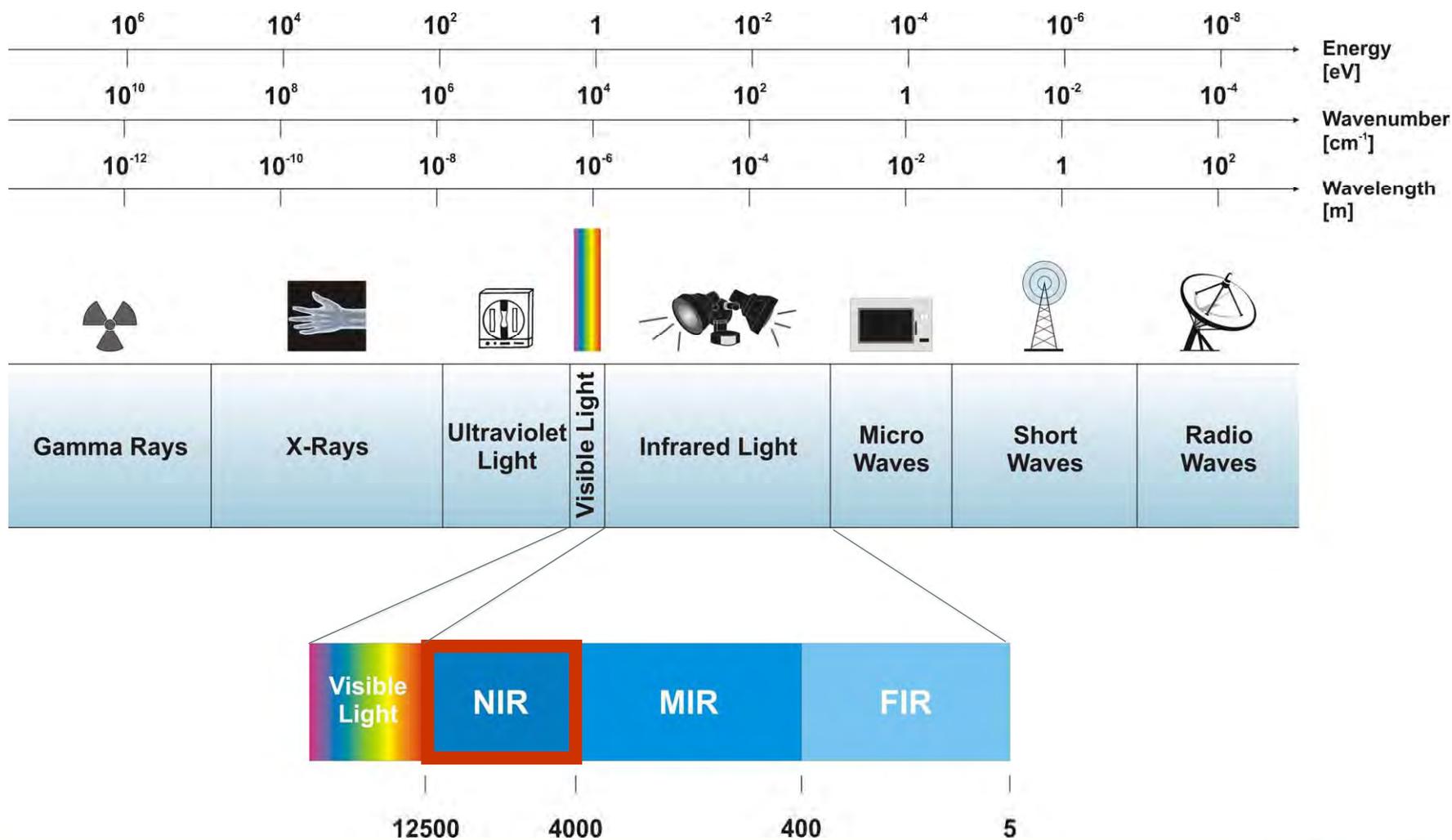
The Infrared(IR)-Spectrum
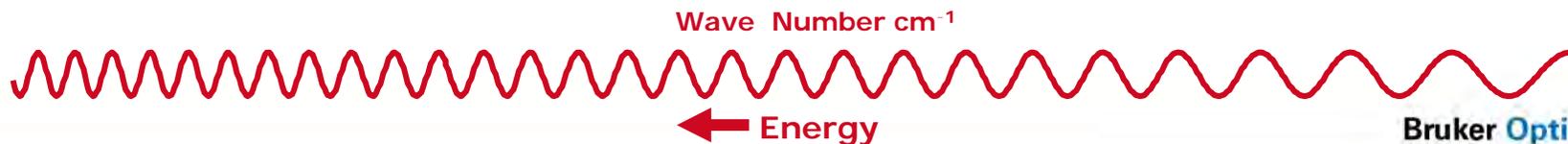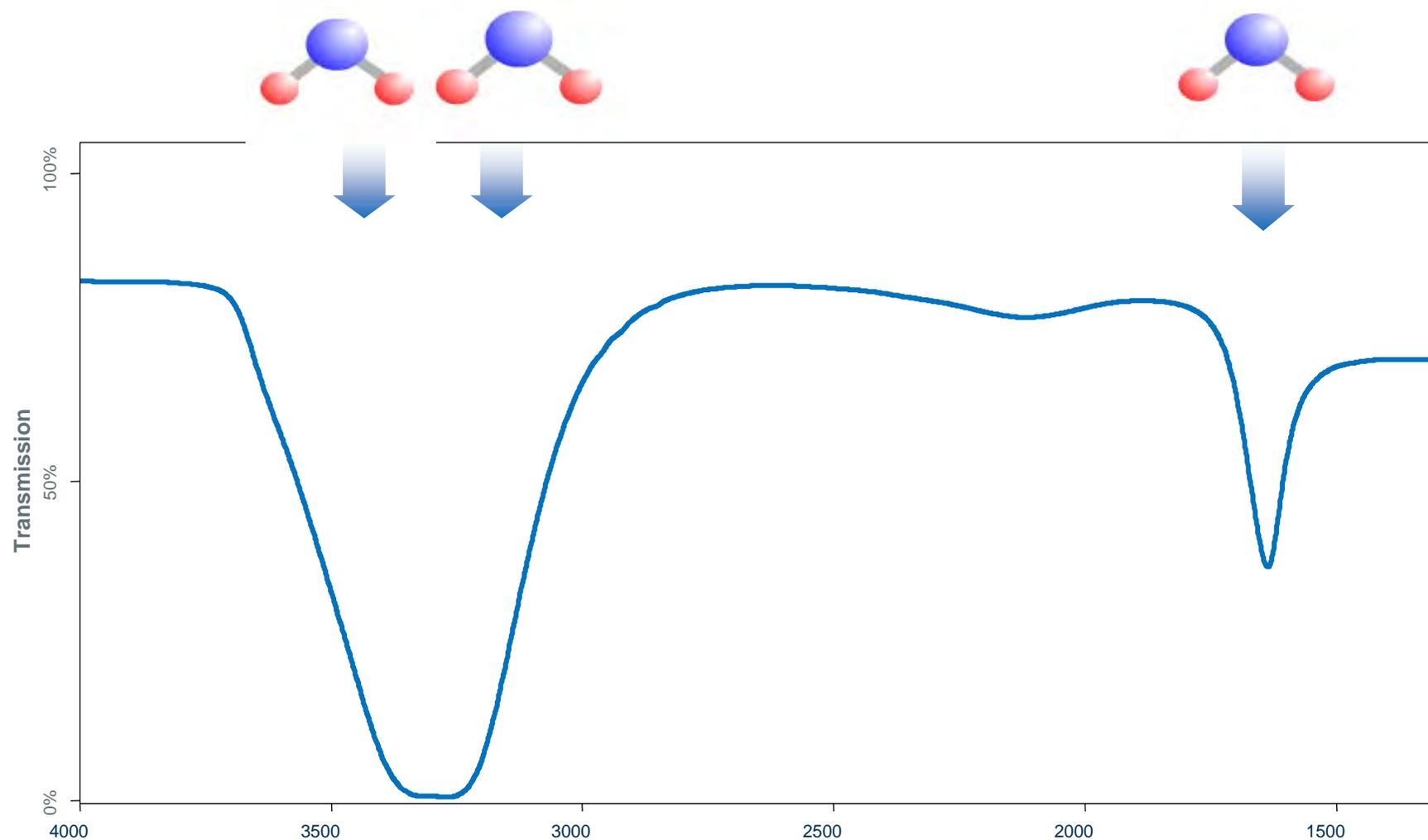
Principle of Oil Measurements

Source   Sample   Detector

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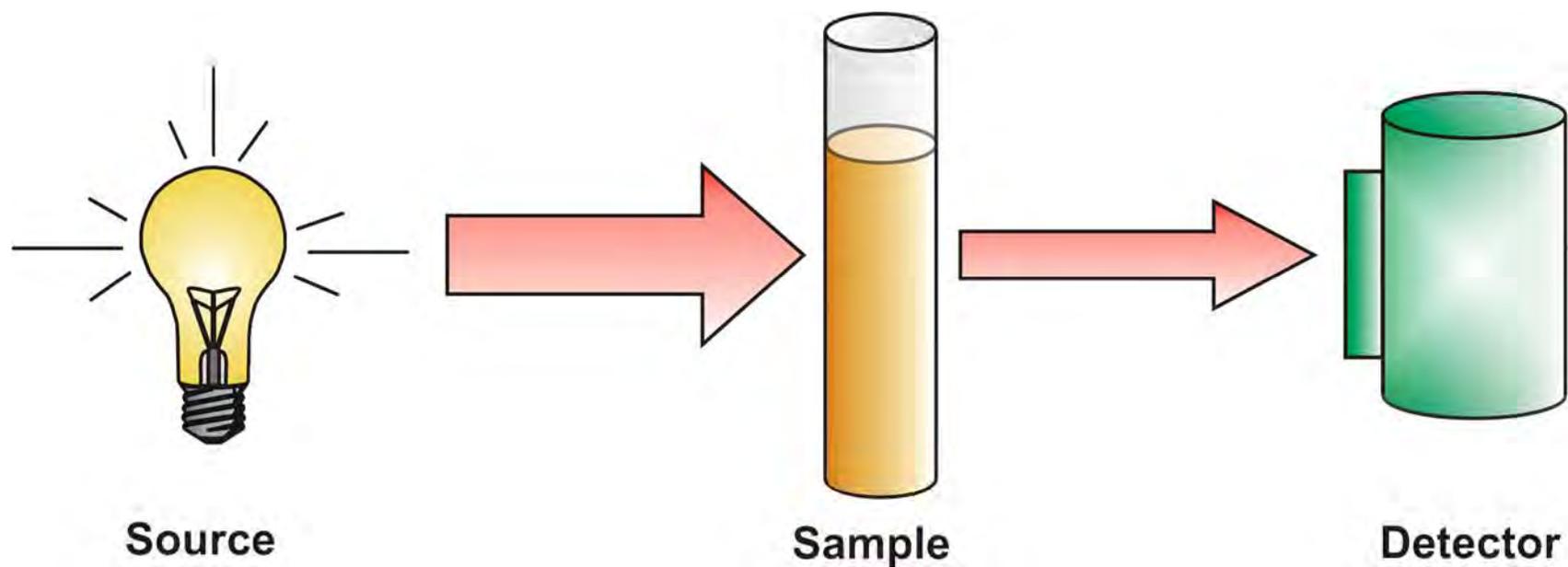
Near Infrared Band Assignment Table

Second Overtone Region

Combinations

Third Overtone Region

First Overtone Region
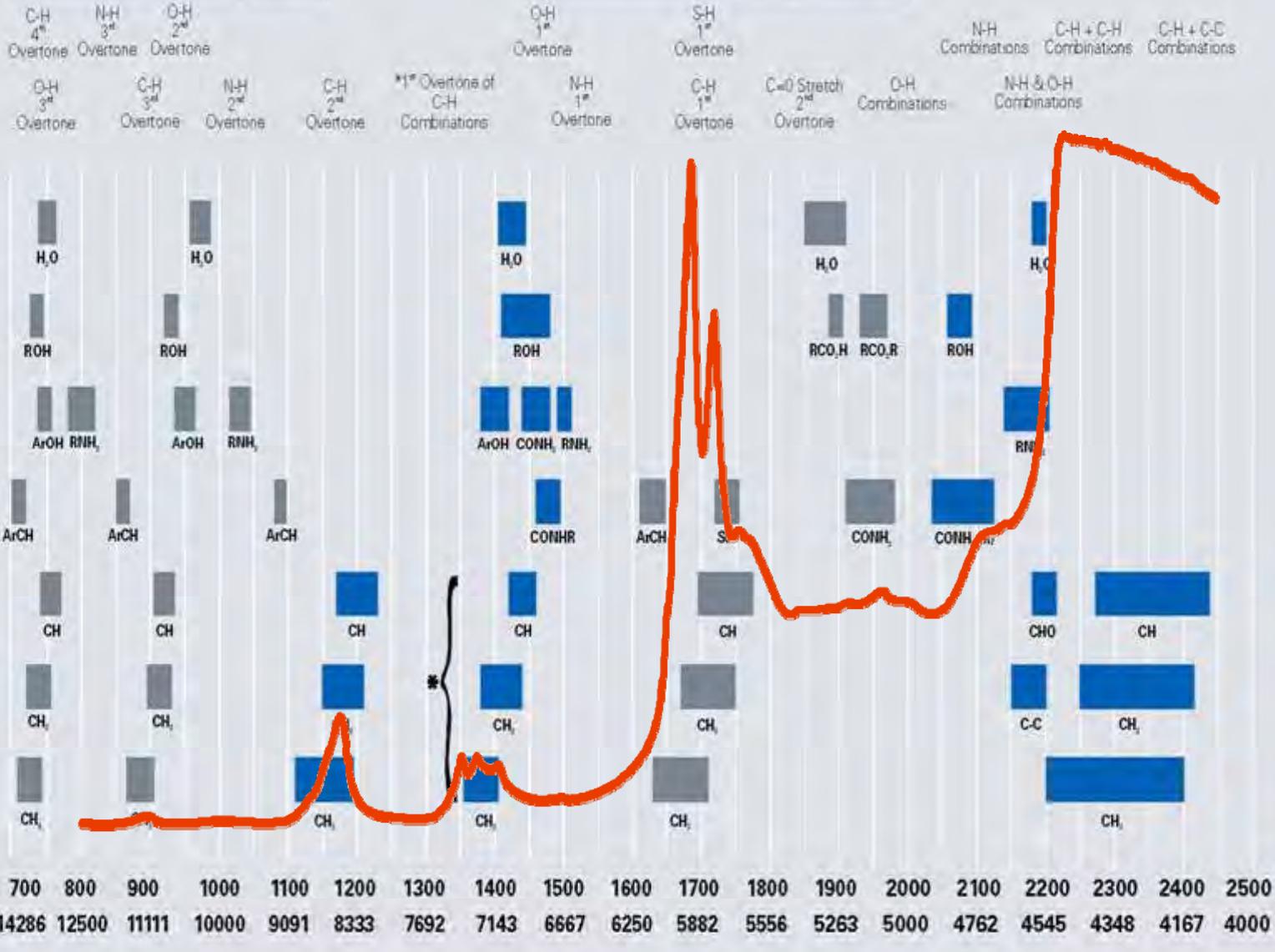
FT-NIR Measurement of Oils

- Sample preparation:
  fill in 8mm disposable vials

- Temperature control at 50°C

- Measurement time: approx. 20 sec

- Display of results on the screen, as
  PDF or print out

Benefits of FT-NIR Spectroscopy

- rapid measurement,
  high sample throughput

- measurements on liquid, solid or
  paste-like samples

- little sample preparation

- no additional reagents necessary

- sample measurements through glass

- simultaneous determination of
  different components

Quality Control of incoming Oils

Quality Control of incoming Oils

FT-NIR spectroscopy can help to:

- Identify the incoming oil

- Assess the quality of the oil

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NIR Analysis of Parameters TFA, FFA and IV

Property		Data Set			Performance		
Name	Unit	n	Min	Max	Rank	R^2	RMSEP
TFA (Trans Fatty Acids)	%	659	0	60.4	14	99.9	0.61
TFA low range	%	265	0	2.9	18	96.4	0.11
FFA (Free Fatty Acids)	%	790	0.1	6	9	99.8	0.09
IV (Iodine Value)	IV	612	0.4	133	11	99.9	0.61

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Typical Performance for NIR Analysis of Fatty Acids in Edible Oils

| Property | | Data Set | | | Performance | | |
Name	Unit	n	Min	Max	Rank	R^2	RMSEP
C16:0	%	608	4	16	10	95.2	0.50
C18:0 all Oils	%	673	1.7	92	19	99.3	0.98
C18:0 Palm Oil	%	251	3.7	17.8	10	99.3	0.36
C18:0 Soya Oil	%	46	3,7	19.5	6	99.5	0.36
C18:0 Sunflower Oil	%	73	2.5	16.5	6	99.6	0.26
C18:1 all Oils	%	800	0	85.2	17	99.9	0.65
C18:2 all Oils	%	673	0	63.2	17	99.9	0.39
C18:3 all Oils	%	415	0	9.2	16	99.5	0.14

Monitor Frying Oil Quality

Monitor Frying Oil Quality

FT-NIR analysis for the critical parameters:

- **Acid Value**

 value for measuring acidic groups in fats and oils formed by hydrolytical and oxidative reactions

- **p-Anisidine Value**

  measuring aldehydes and ketones as secondary oxidation products

- **Total Polar Material**

  measuring all polar compounds in frying fats and oils

- **Polymerized Tri-Acylglyceroles**

  generated during frying as a result of oxidative and thermal degradation

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Total Polar Material

Spectra
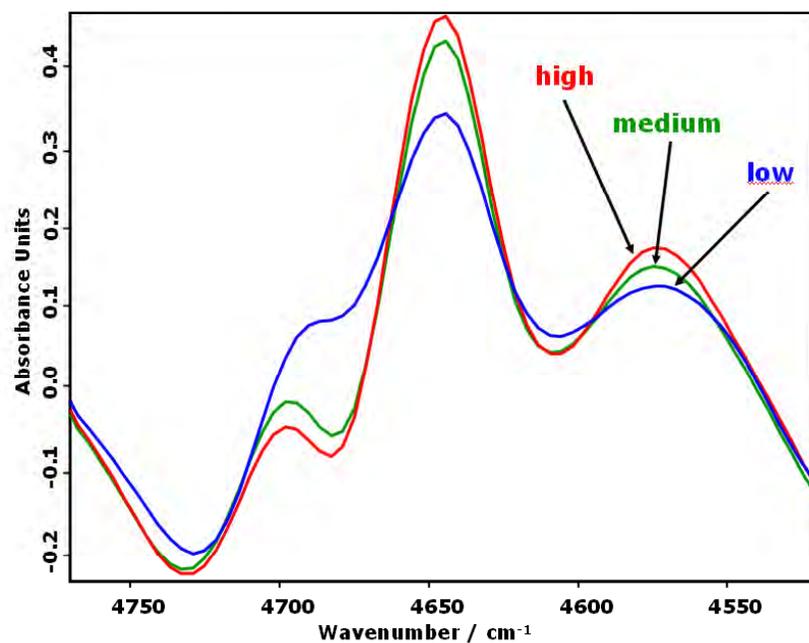
Calibration Plot
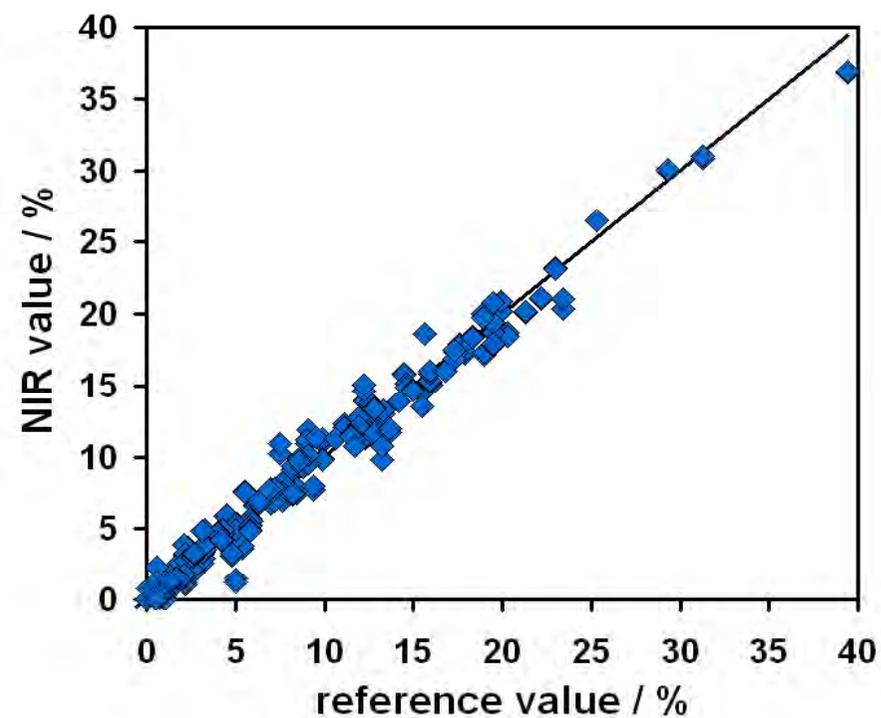
NIR Calibration Data

Property		Data Set			Performance		
Name	Unit	n	Min	Max	Rank	R^2	RMSECV
Acid Value	%	556	0.05	5.7	12	96.1	0.17
p-Anisidine Value	-	451	0.1	178	13	90.6	6,84
Total Polar Material	%	540	1.8	50.3	10	95.7	1.52
polymerized TAGs	%	663	0.1	39.4	9	96.7	0.56

Screening analysis of used frying fats and oils using FT-NIR spectroscopy (C-VI 21 (2013))

The Art of Frying...

15% TPM / AnV 45

RANCID TASTE!
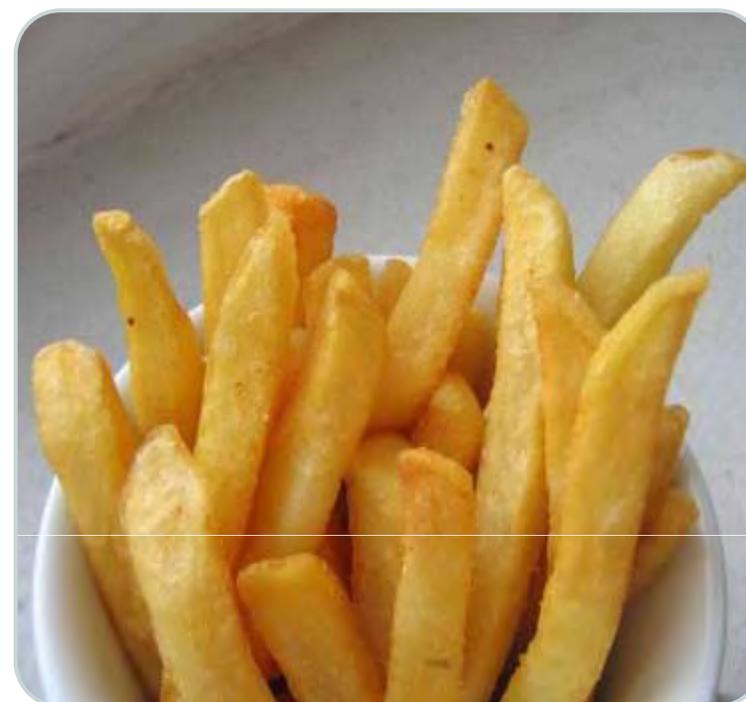
20% TPM / AnV 90

**OPTIMAL TASTE!**

Verification of Olive Oil Quality

Methods to Check the Quality of Virgin Olive Oils
(proposed 2005 by Ch. Gertz)

- **Determination of 1.2 di- and 1.3-diacylglycerol**

  (DGF-Standard Method C-VI 16(06) (ISO 29822:2009)

- **Determination of degradation products of chlorophyll A in virgin olive oils (PPP)**

  (DGF Standard Method C-VI 15(06) (ISO 29841:2009)

Statistical Evaluation of 960 Olive Oils in Germany (Gertz: 2005-2010)
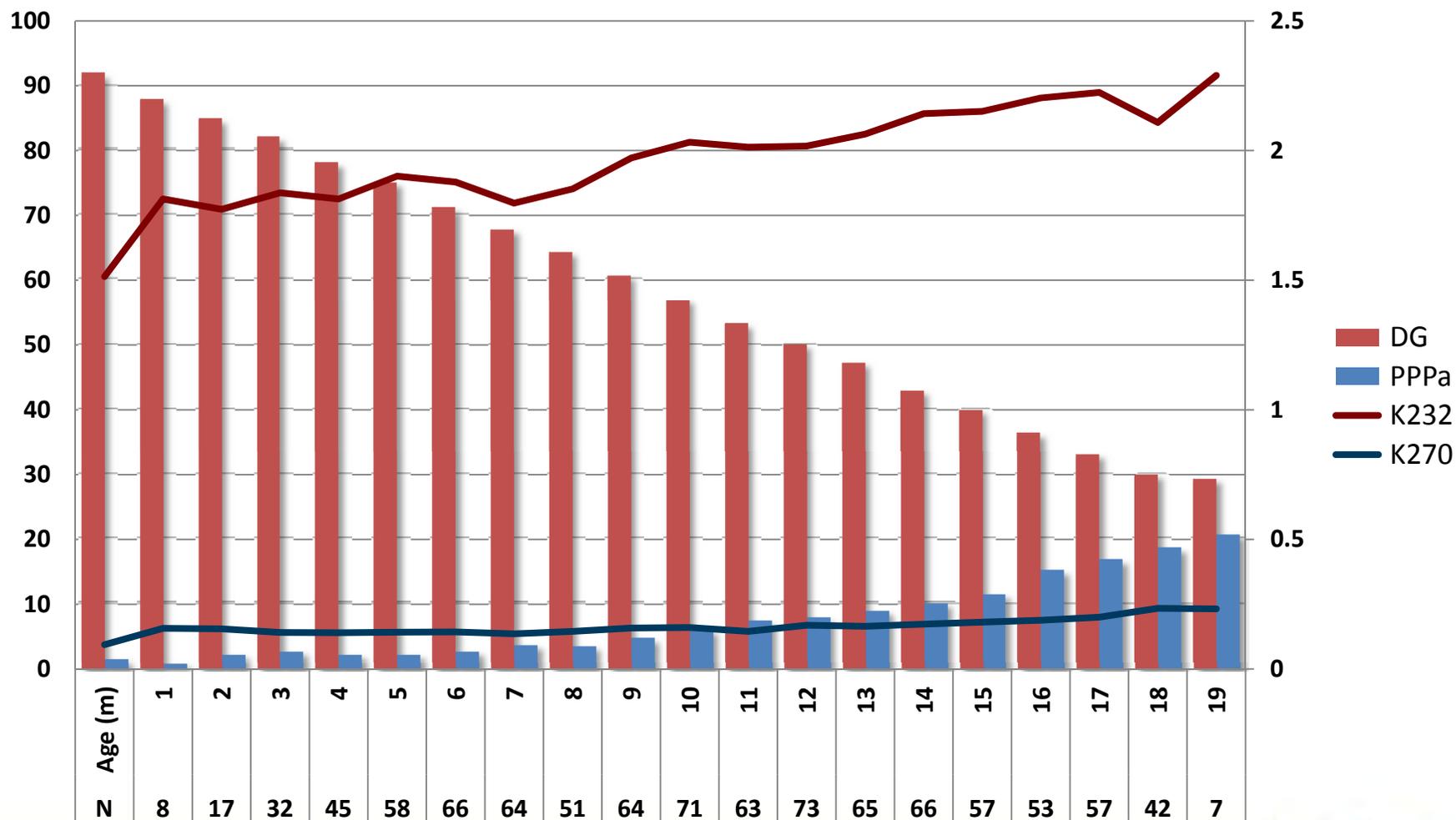
Information of selected Analytical Criteria

- **1,2-Diglycerides:**
  - Quality of harvesting and treatment before pressing
  - Age of the oil

- **FFA:**
  - Another criterium of good manufacturing practice
  - Independent from 1,2-Di-Content

- **Pyropheophytin:**
  - Age, Oxidation
  - Quality of package
  - Thermal treatment

- **K232/K270:**
  - Oxidation
  - Thermal treatment

Validation of NIR Data

| Property		Data Set			Performance
Name	Unit	n	Min	Max	RMSECV
1,2-Diglycerides	%	209	29.8	96.5	**0.17**
Pyropheophytins	%	128	0.1	14.5	**6,84**
K232	–	138	1.73	3.91	**1.52**
K270	–	138	0.13	0.75	**0.56**

Measurement of Oilseeds

Integration Sphere
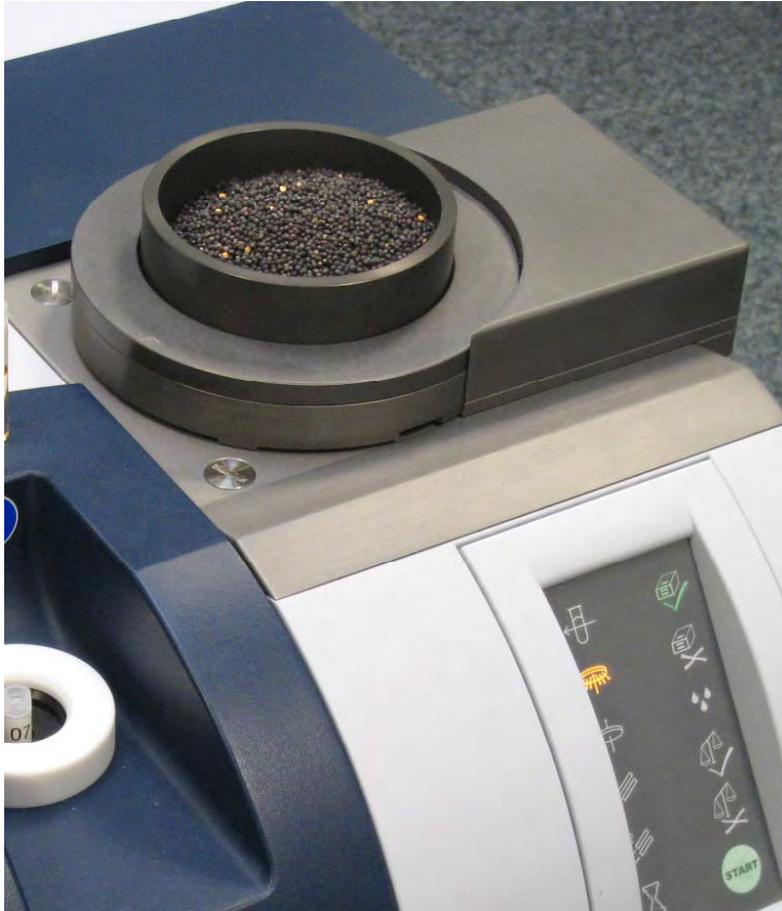
- For measurements in diffuse reflection

- Ideal for seeds

- Guarantees high reproducibility with heterogeneous samples

# Integration Sphere

Sample

Gold coated Integration Sphere

Detector

NIR beam

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Sample Spinners for the Integrating Sphere
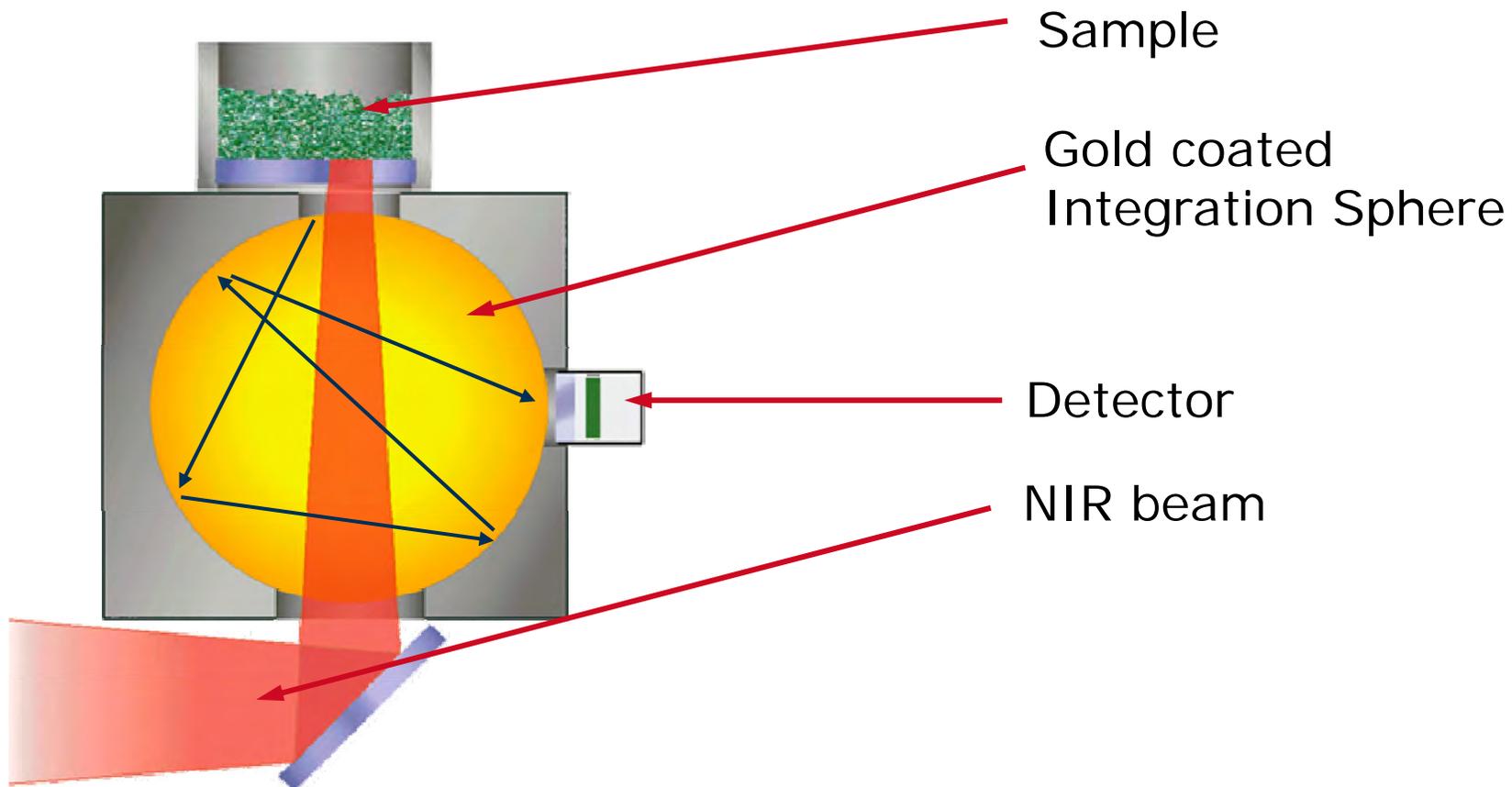
Measurement of heterogeneous Samples

Surface enlarged x 18

Window of the
Integration Sphere
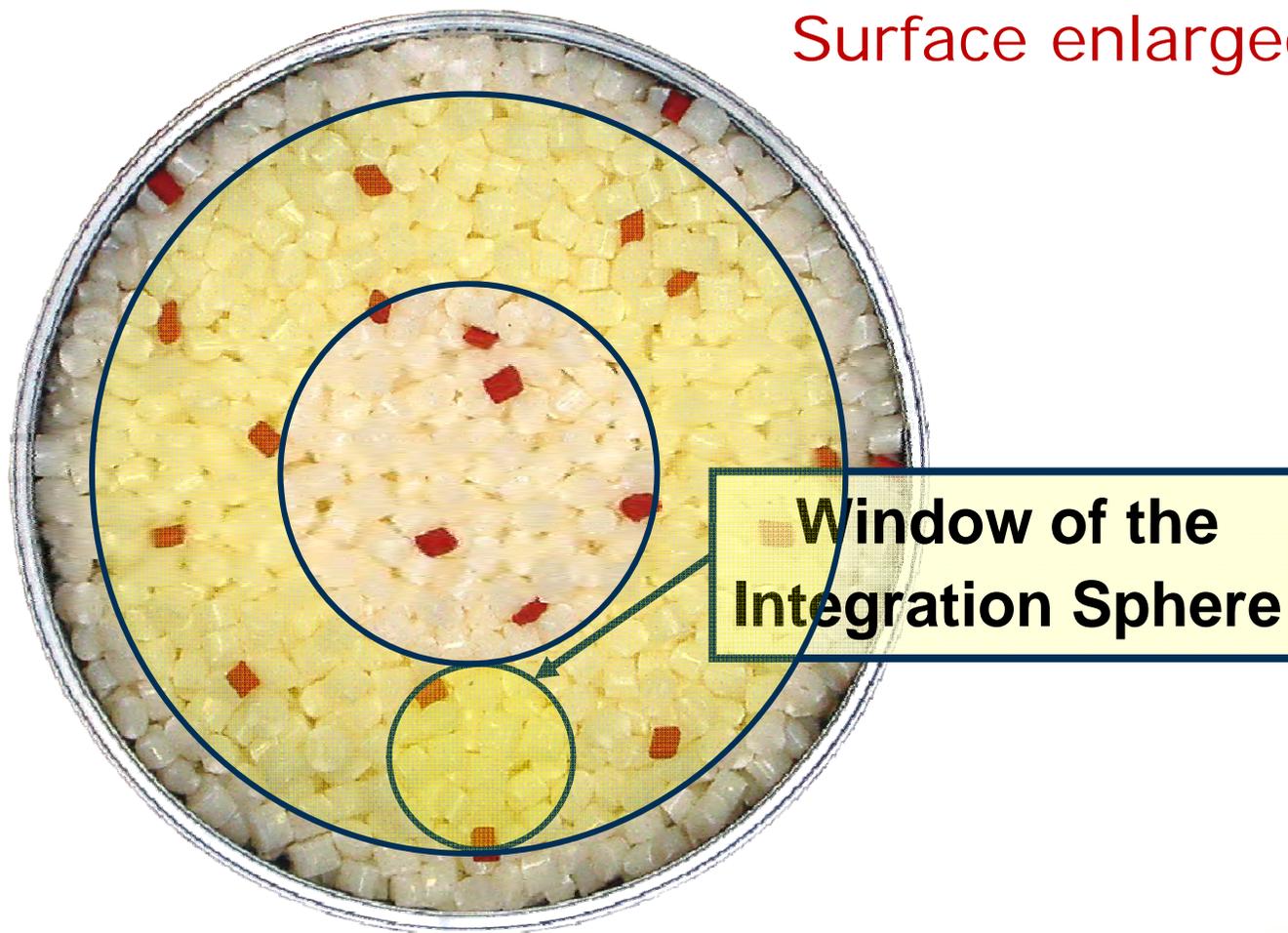
Measurement of heterogeneous Samples

Acessory	Area	Factor
Integration Sphere	1.1 cm^2	1
Small Spinner	6.8 cm^2	6
Large Spinner	19.6 cm^2	18

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Example: Rapeseed

Example: Rapeseed

- **for Oil Mills:**

  Parameters like oil content and moisture for the optimization of the pressing process

- **for Breeders:**

  Glucosinolate, Erucic Acid content

Plant Breeders Rapeseed

|	Minimum	Maximum	Calibration Error	Validation Error	Correlation
Dry Matter	85.0%	97.0%	0.35	0.39	0.880
Protein	10.0%	34.0%	0.56	0.61	0.960
Fat	25.0%	60.0%	0.81	0.78	0.950
Glucosinolate	1.4 umol/gram	160.0 umol/gram	3.73	5.44	0.940
Glucosinolate 30	1.4 umol/gram	30.0 umol/gram	2.94	2.21	0.820
Erucic	0.0%	60.0%	2.12	2.54	0.990
Linoleic	8.0%	30.0%	1.62	1.87	0.740
Linolenic	1.3%	16.0%	1.05	1.01	0.860
Oleic	10.0%	80.0%	3.08	3.02	0.970
Palmitic	2.0%	7.0%	0.29	0.30	0.840
Stearic	0.2%	3.0%	0.20	0.22	0.760

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Example: Olives

Measuring Olive Paste

Oil Content:

- before Pressing
    - → Value of Olives

- after Pressing
    - → Performance of Presses
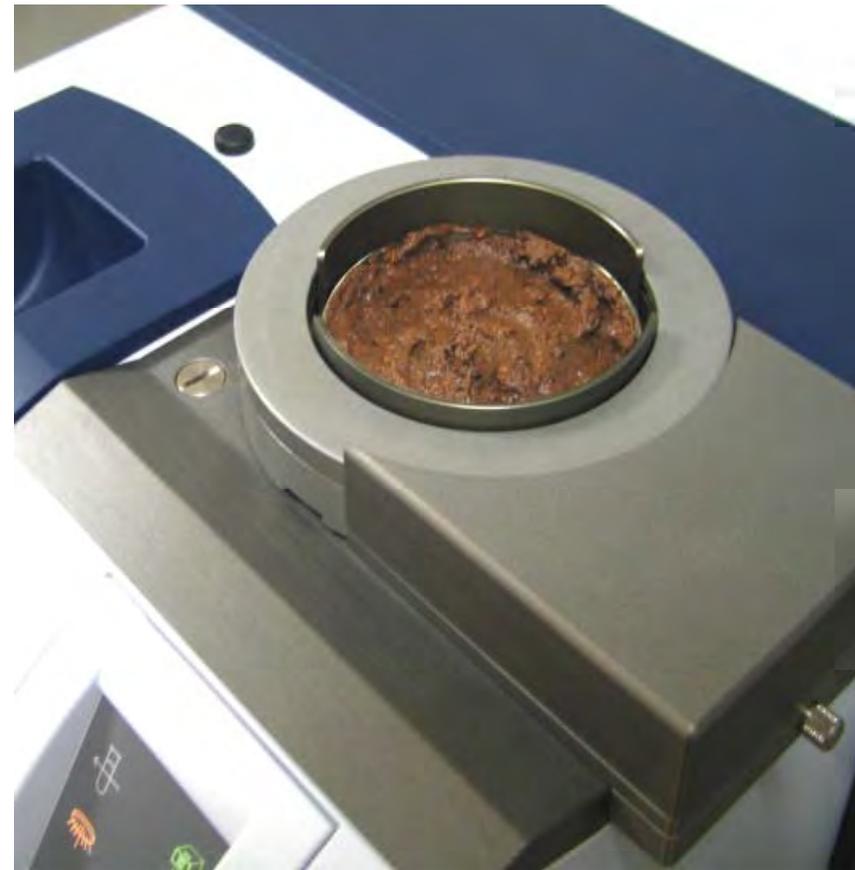
Advantages of FT-NIR Spectroscopy

- Only one system for oils and seeds!

Online analysis of FFA & Polars in Frying Oils

Fast loops with oils from 80°C – 220°C
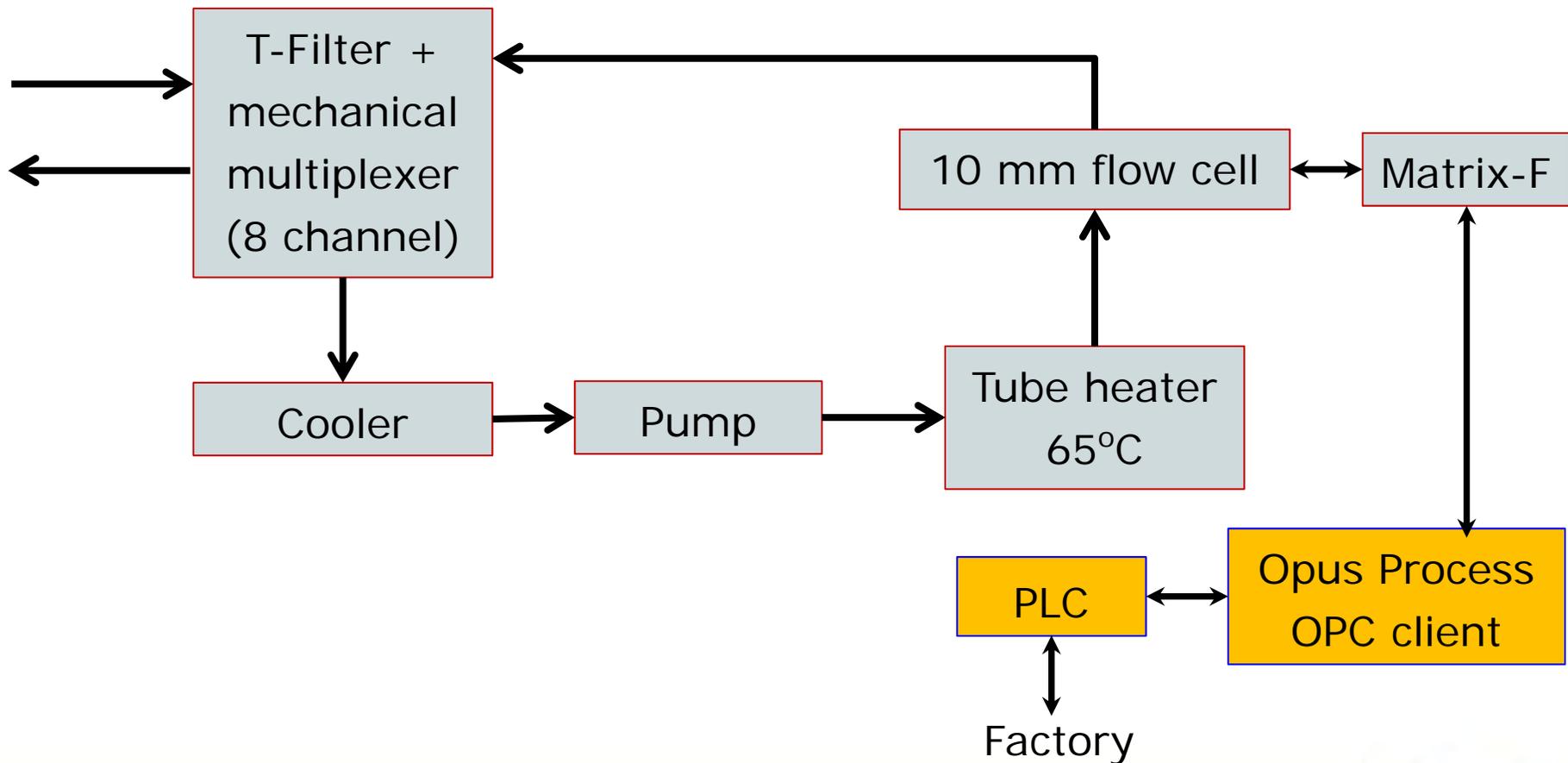
Sampling station for liquid samples

MATRIX-F online system in cabinet

# Contactless measurements of solids

Summary

Advantages of FT-NIR Spectroscopy

- very fast analysis method (< 30 seconds)

- low running costs

- parallel measurement of multiple parameters

- no use of chemicals, solvents or gases

- can substitute various analyses methods like titration and GC

- untrained staff can carry out analyses

- operator errors almost impossible

- applicable in the production area

Thank You for Your Attention!
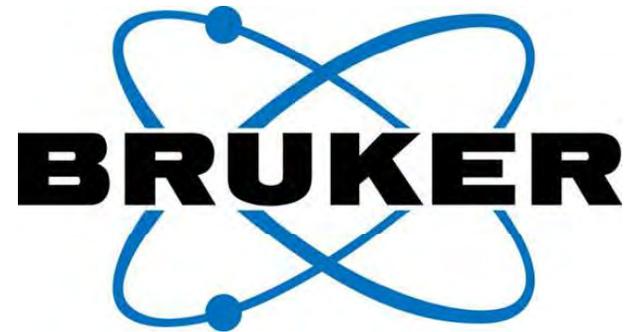
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