FTIR in FOOD

1. Composition Analysis:

- **Identification of Components**: FTIR helps in identifying various components in food products by detecting specific functional groups (like alcohols, esters, amines) based on their infrared absorption spectra.
- **Quantification**: It can be used for quantitative analysis of food constituents, such as moisture, fats, proteins, and carbohydrates.

2. Quality Control:

- **Adulteration Detection**: FTIR is effective in detecting food adulteration by comparing the spectra of pure and adulterated samples.
- **Freshness and Shelf-life**: It can monitor changes in food composition over time, aiding in assessing freshness and predicting shelf-life.

3. Rapid and Non-destructive Testing:

- **Speed**: FTIR provides rapid results, making it suitable for real-time monitoring in food production lines.
- **Non-destructive**: The technique often requires minimal sample preparation, preserving the integrity of the sample for further tests if needed.

4. Nutritional and Functional Analysis:

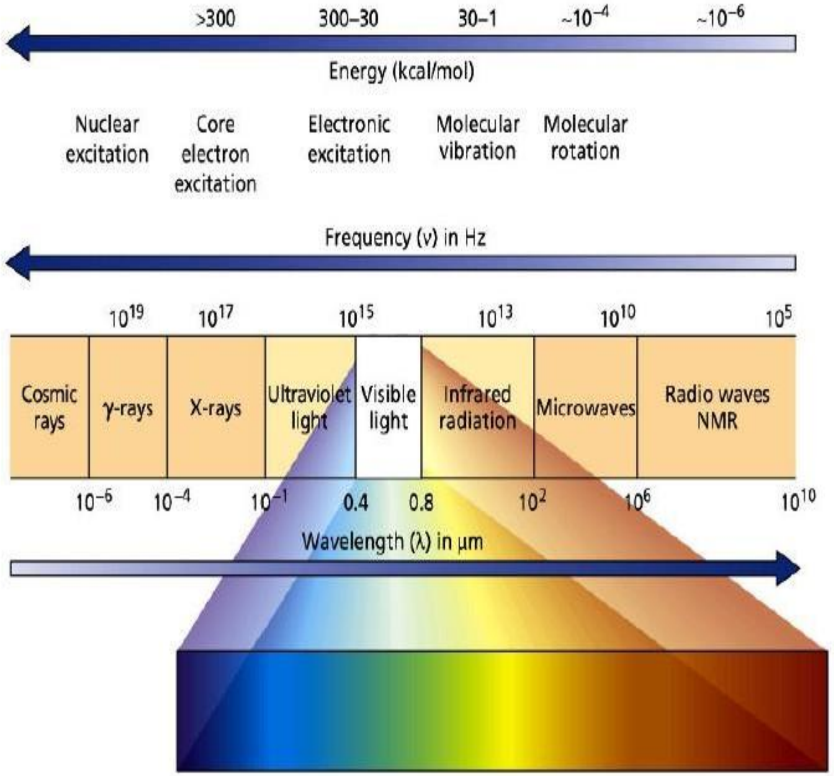
- **Nutrient Profiling**: FTIR helps in profiling the nutritional content of food products, which is essential for labeling and regulatory compliance.
- **Functional Properties**: It can assess the functional properties of food ingredients, such as emulsifiers and stabilizers, based on their molecular interactions.

5. Authentication:

- **Geographic and Botanical Origin**: FTIR can differentiate food products based on their geographic or botanical origin by identifying unique spectral fingerprints.
- **Organic vs. Conventional**: It aids in distinguishing between organic and conventionally produced food products.

6. Regulatory Compliance:

- FTIR supports regulatory compliance by ensuring that food products meet specified standards and do not contain banned or harmful substances.



INTRODUCTION OF IR SPECTROSCOPY

- Infrared spectroscopy is an important analytical technique for determining the structure of both inorganic & organic compounds. It is also known as vibrational spectroscopy
- IR radiations lies in the wavelength range of 0.7 - 400 μm.
- IR spectroscopy is based upon selective absorption of IR radiations by the molecule which induces vibration of the molecules of the compound.
- IR instruments are of 2 types namely, dispersive instruments (spectrophotometers) and Fourier transform IR instrument.
- The radiation sources used are incandescent lamp, Nernst glower etc., and the detectors used are thermal and photon detectors.

NATURE OF IR SPECTRA

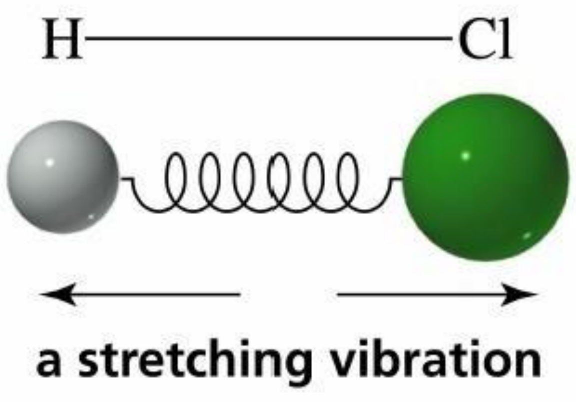
- IR spectrum is a graph of band intensities on ordinate versus position of band on abscissa.
- Band intensities can be given in terms of transmittance(T) or absorbance(A).
- Position of band can be expressed in terms of wave number (n) or wavelength(λ).
- In IR spectra, wave numbers (n) are used instead of wavelength (λ) for mentioning the characteristic peak as this unit has advantage of being linear with energy of radiation (E) .

$$E = hc/\lambda \quad \text{or, } E = hcn$$

[$n = 1/\lambda$, c= velocity of light, h= Planck's constant]

PRINCIPLE OF IR SPECTROSCOPY

- When the energy in the form of IR is applied and if the applied IR frequency = Natural frequency of vibration, the absorption of IR takes place and a peak is observed.
- Molecules are excited to the higher energy state from the ground state when they absorb IR radiation.
- When a compound is exposed to IR radiation, it selectively absorbs the radiations resulting in vibration of the molecules of the compound, giving rise to closely packed absorption bands, called as IR absorption spectrum.
- The bands correspond to the characteristic functional groups and the bonds present in a chemical substance. Thus, an IR spectrum of a compound is considered as the fingerprint for its chemical identification.



CHANGE IN DIPOLAR MOMENT OF THE MOLECULES MUST OCCUR!

H—Cl

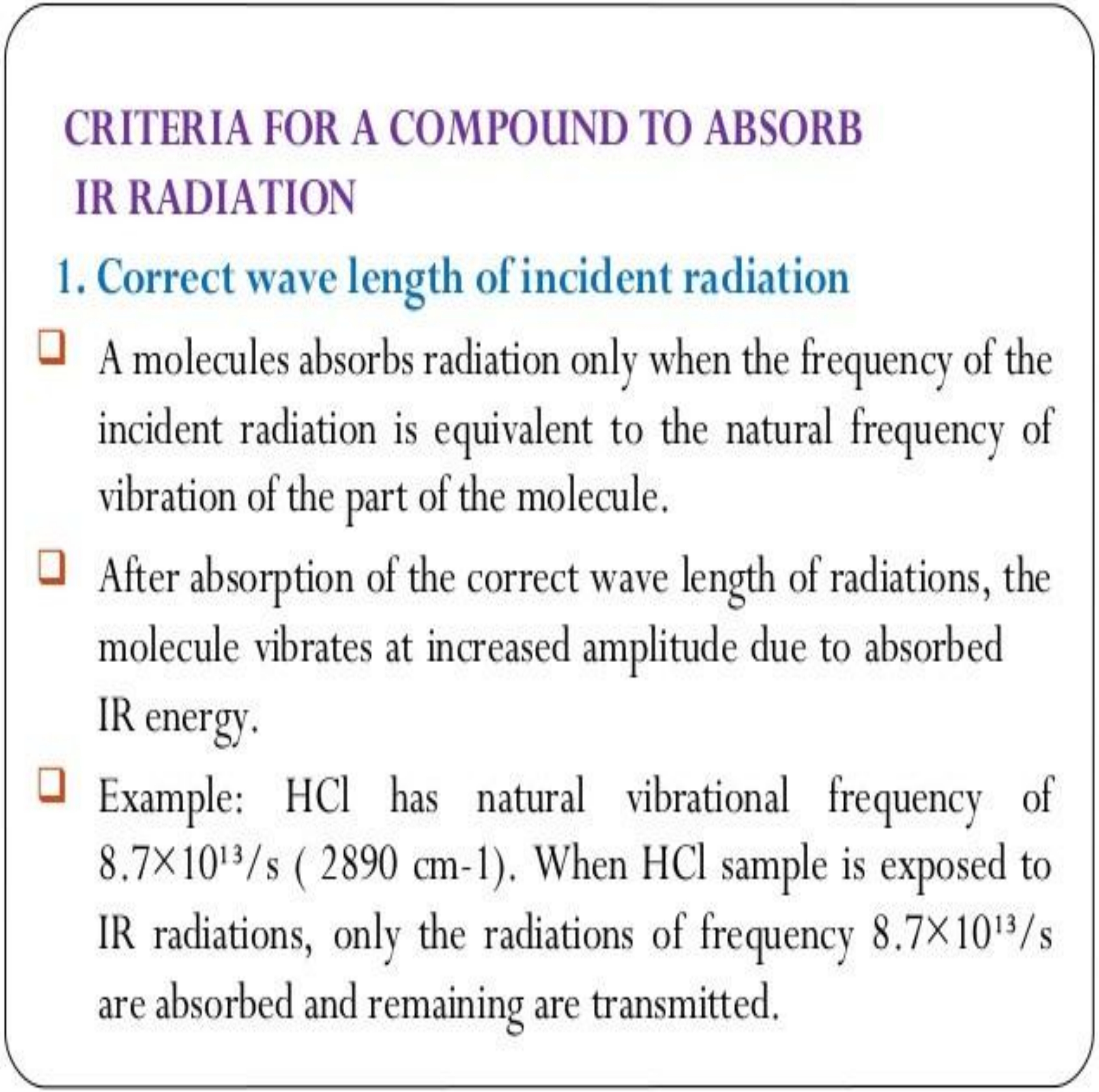
← ←
O=C=O

ACTIVE IR STRECHTING

H—H

← →
O=C=O

INACTIVE IR STRECHTING



CRITERIA FOR A COMPOUND TO ABSORB IR RADIATION

1. Correct wave length of incident radiation

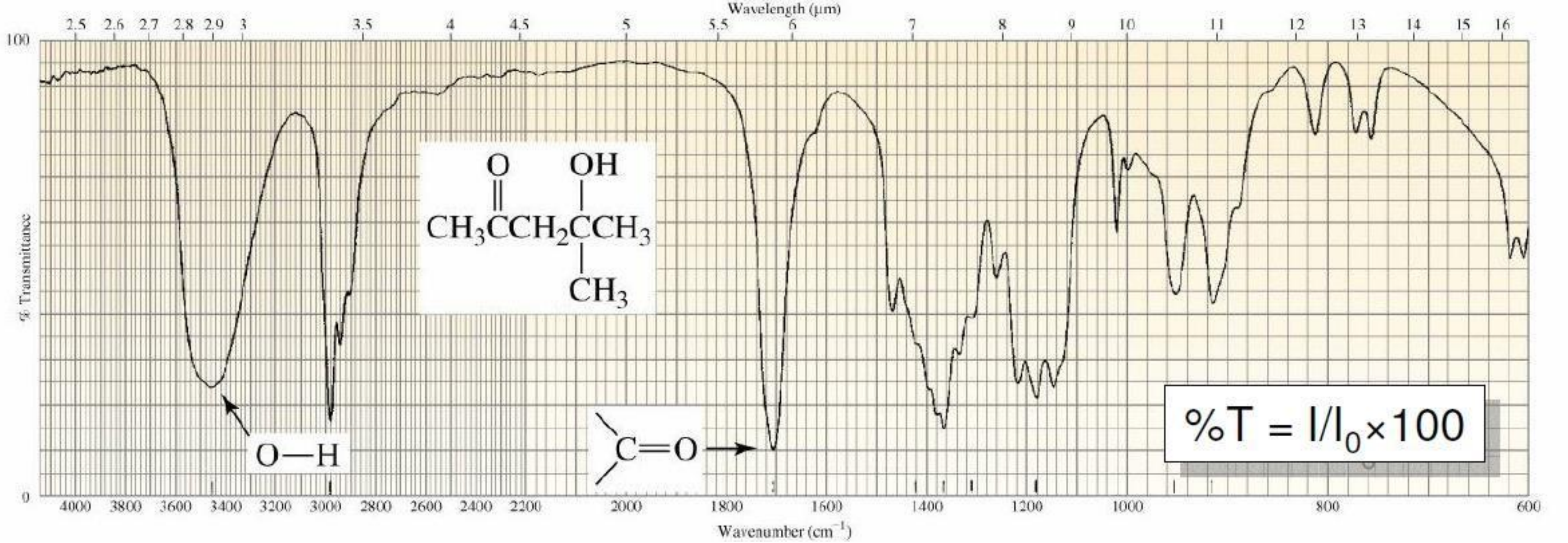
- ❑ A molecules absorbs radiation only when the frequency of the incident radiation is equivalent to the natural frequency of vibration of the part of the molecule.
- ❑ After absorption of the correct wave length of radiations, the molecule vibrates at increased amplitude due to absorbed IR energy.
- ❑ Example: HCl has natural vibrational frequency of 8.7×10^{13}/s (2890 cm-1). When HCl sample is exposed to IR radiations, only the radiations of frequency 8.7×10^{13}/s are absorbed and remaining are transmitted.

PEAK POSITION

$$\tilde{\nu} = \frac{1}{2\pi c}\sqrt{\frac{k}{m^*}}$$

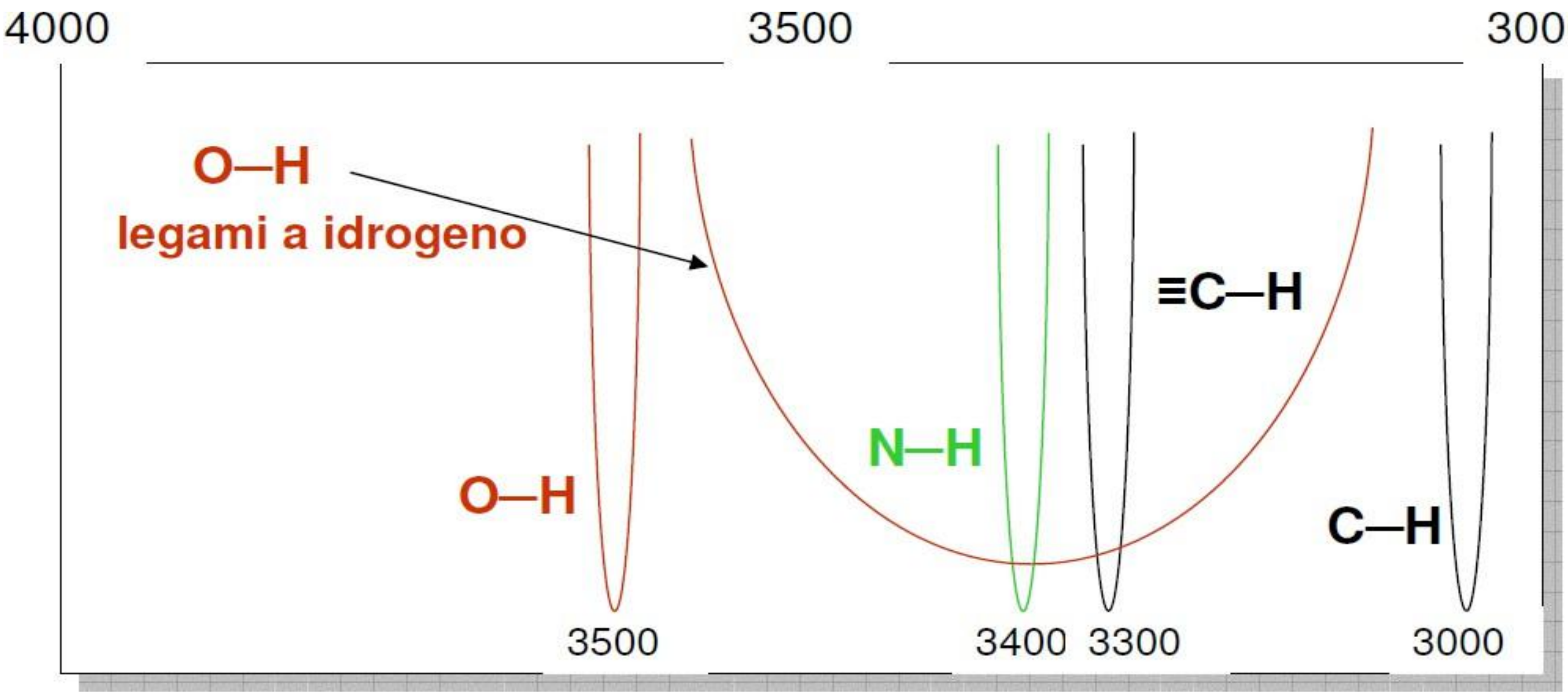
k = bond strenght

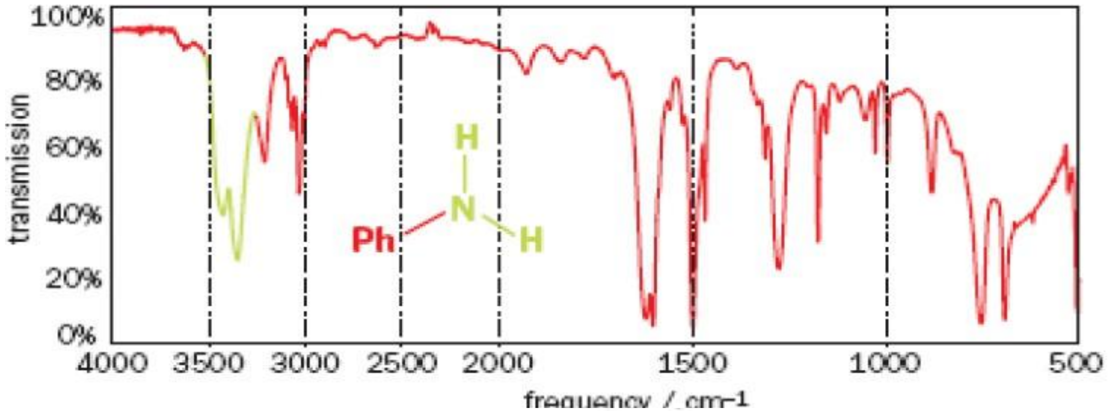
$m^* = m_A \, m_B \, / \, (m_A + m_B)$

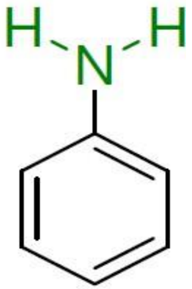


O—H > N—H > C—H

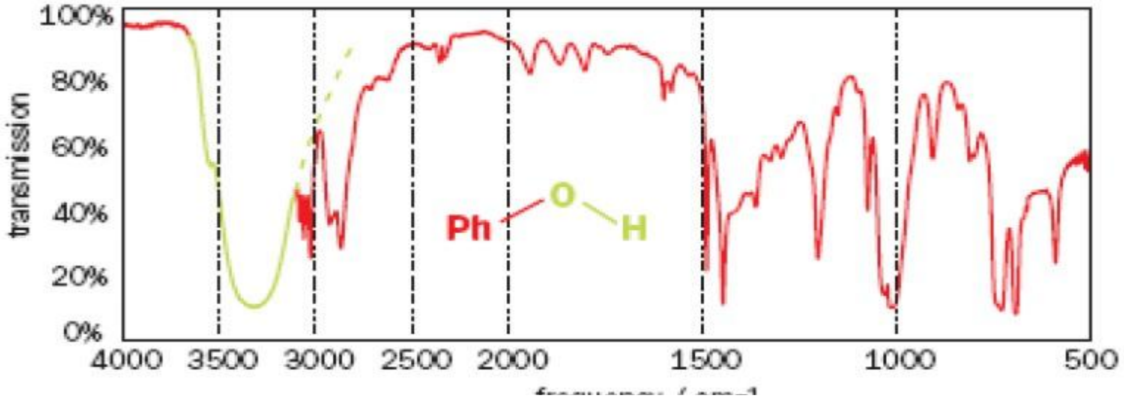
BOND STRENGHT

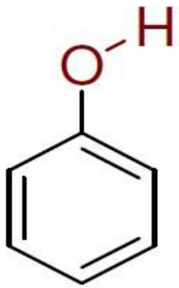




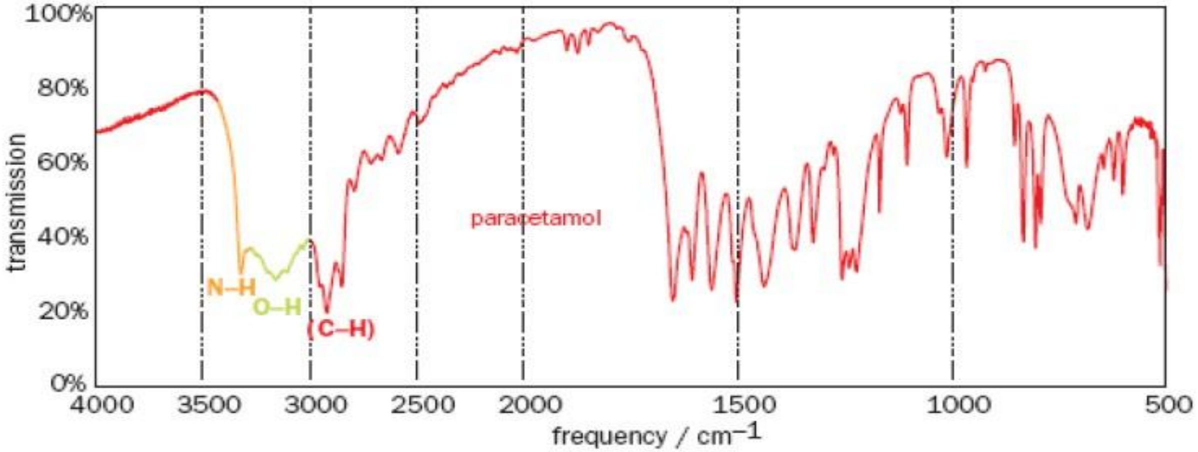


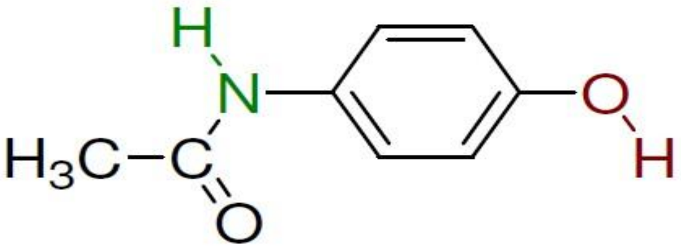
SIMMETRIC AND ASIMMETRIC STRETCHING





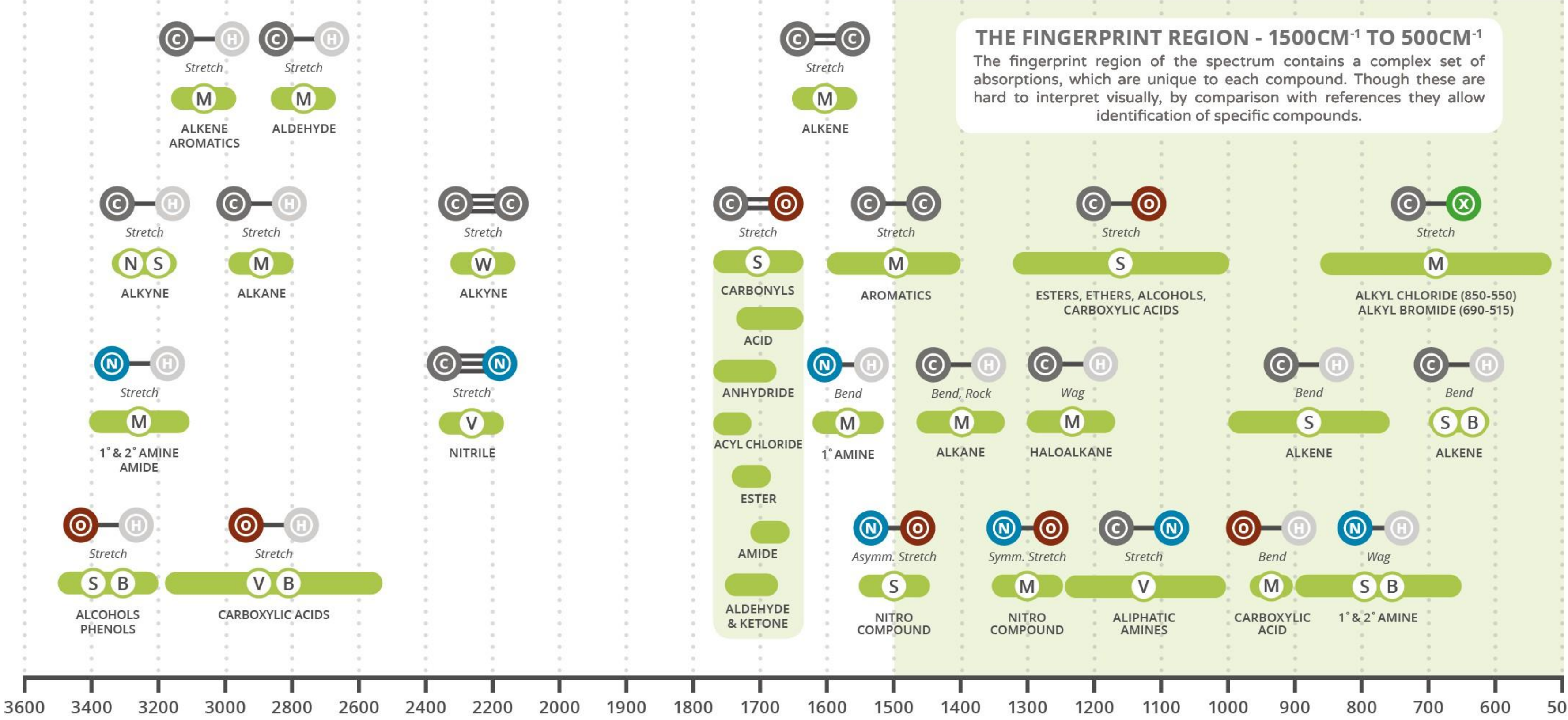
HYDROGEN BONDS





ANALYTICAL CHEMISTRY - INFRARED SPECTROSCOPY

Commonly referred to as IR spectroscopy, this technique allows chemists to identify characteristic groups of atoms (functional groups) present in molecules.



THE FINGERPRINT REGION - 1500CM^{-1} TO 500CM^{-1}

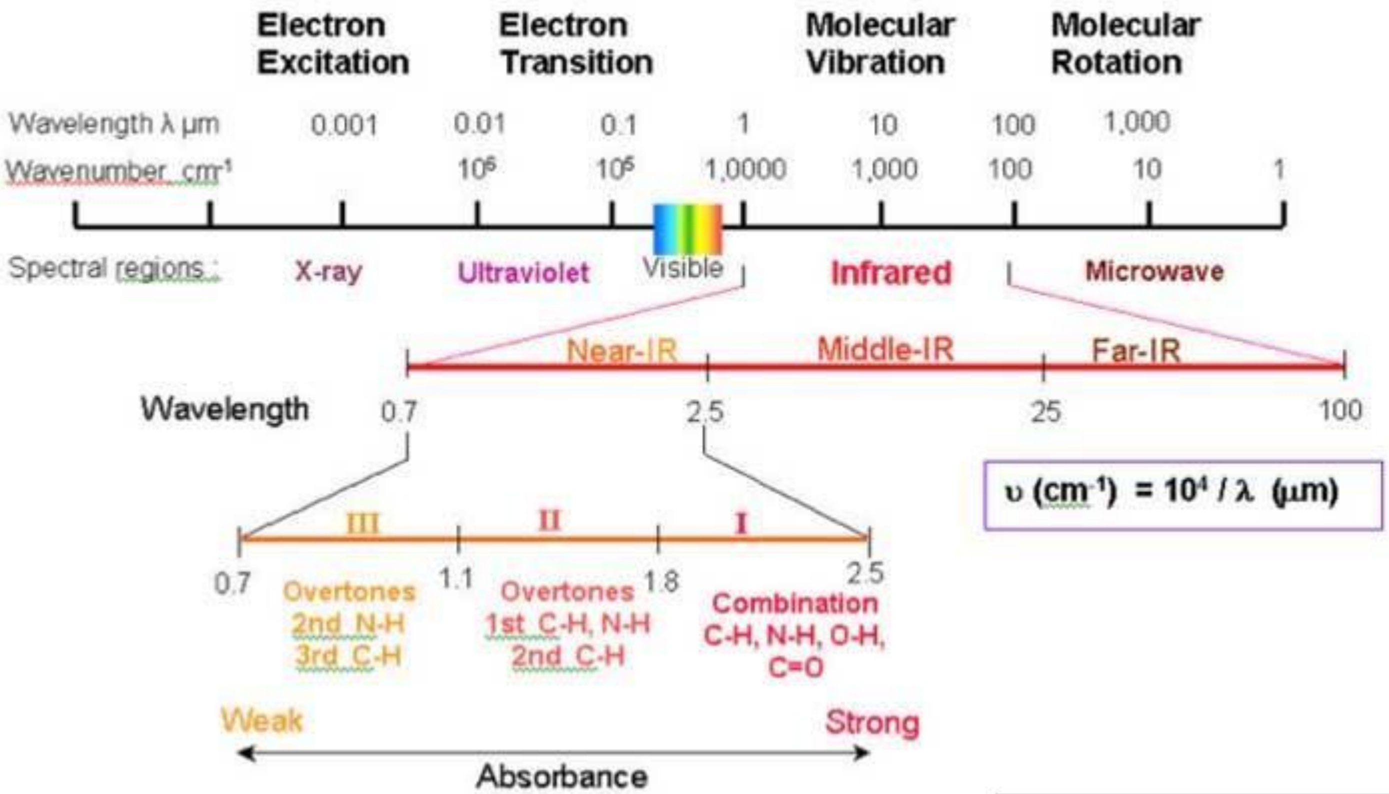
The fingerprint region of the spectrum contains a complex set of absorptions, which are unique to each compound. Though these are hard to interpret visually, by comparison with references they allow identification of specific compounds.

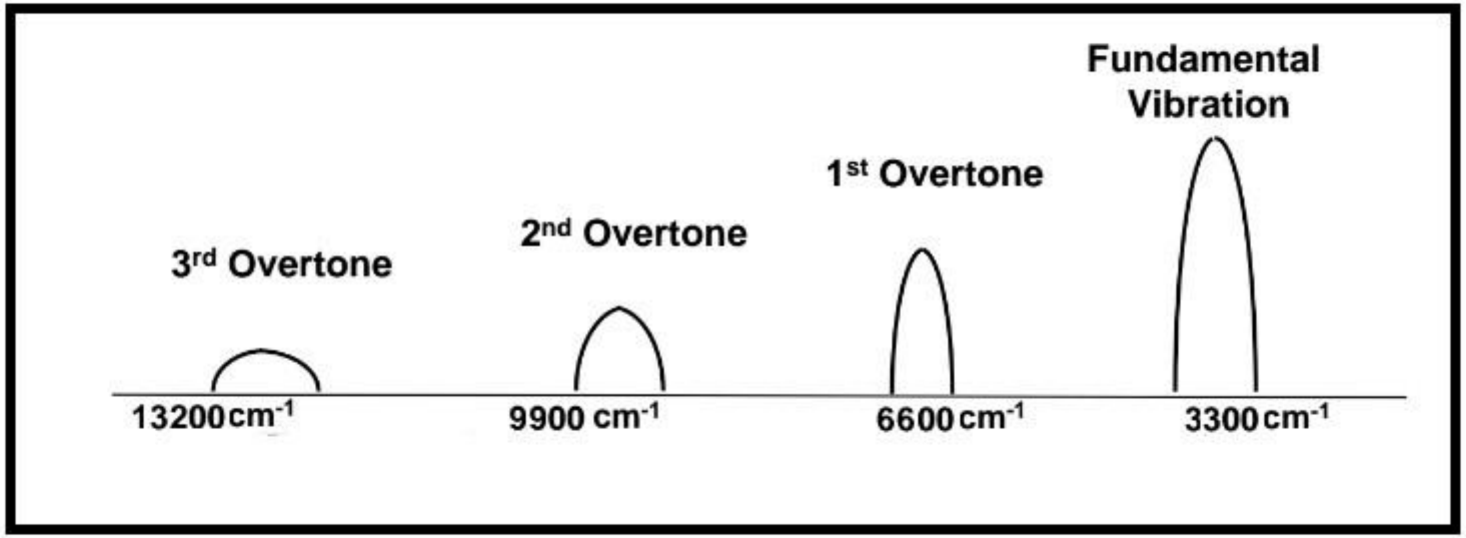
FREQUENCY/WAVENUMBER OF ABSORPTION (CM^{-1})

Key: S STRONG M MEDIUM W WEAK B BROAD N NARROW V VARIABLE

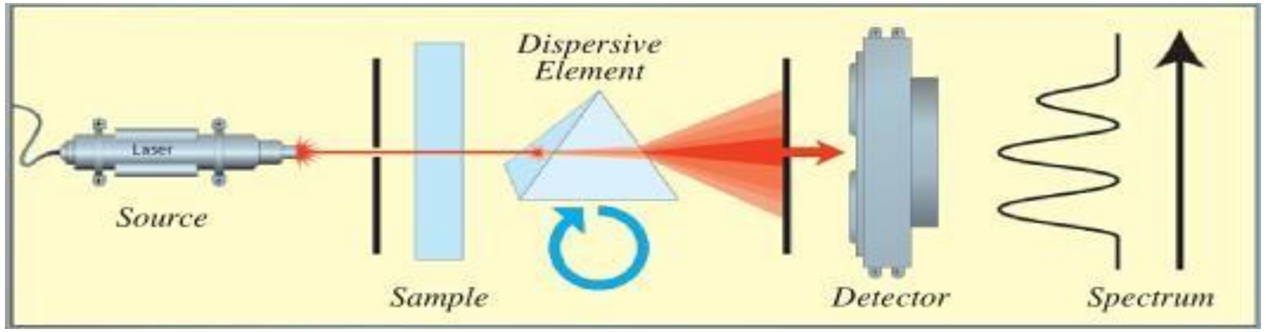
Infrared frequencies make up a portion of the electromagnetic spectrum. If a range of infrared frequencies are shone through an organic compound, some of the frequencies are absorbed by the chemical bonds within the compound. Different chemical bonds absorb different frequencies of infrared radiation. There are a number of characteristic absorptions which allow functional groups (the parts of a compound which give it its particular reactivity) to be identified. This graphic shows a number of these absorptions.

ELECTRO-MAGNETIC SPECTRUM

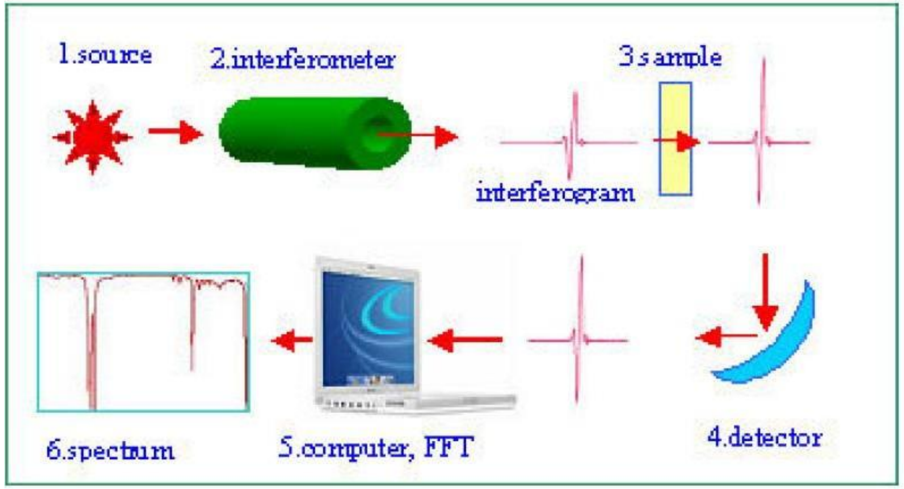




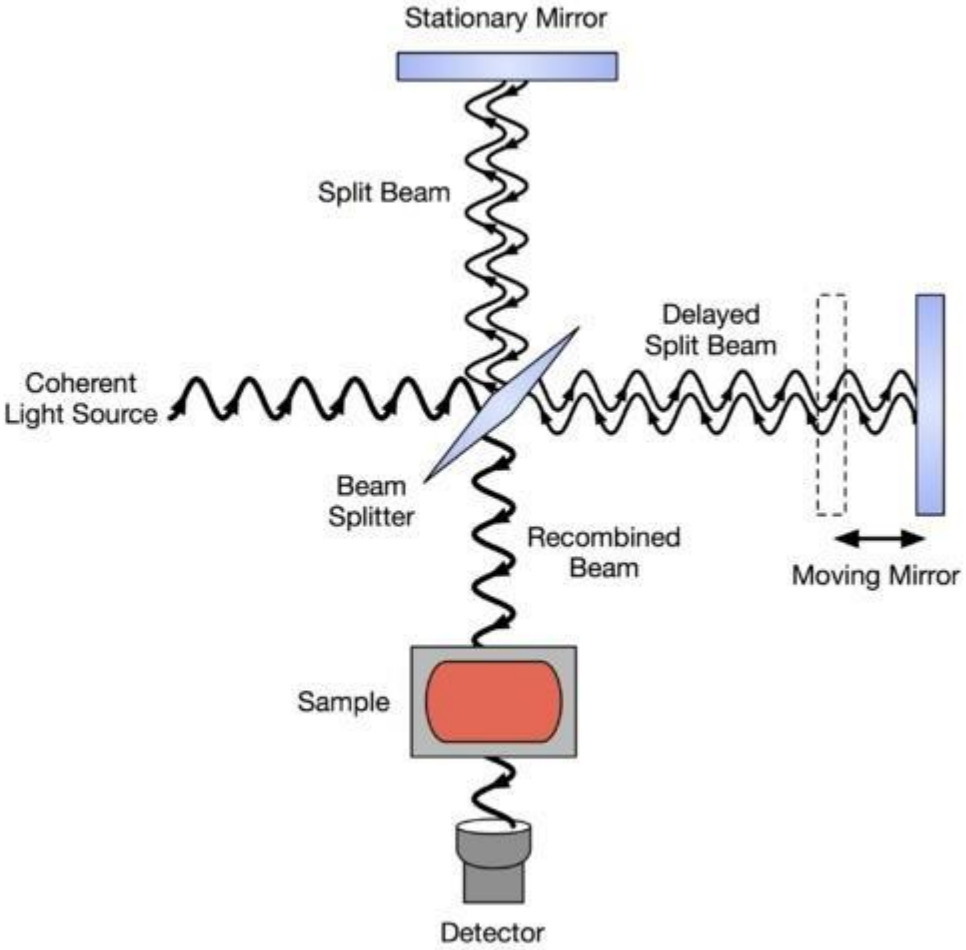
SPETTROMETRI IR E FTIR (O FT-NIR)

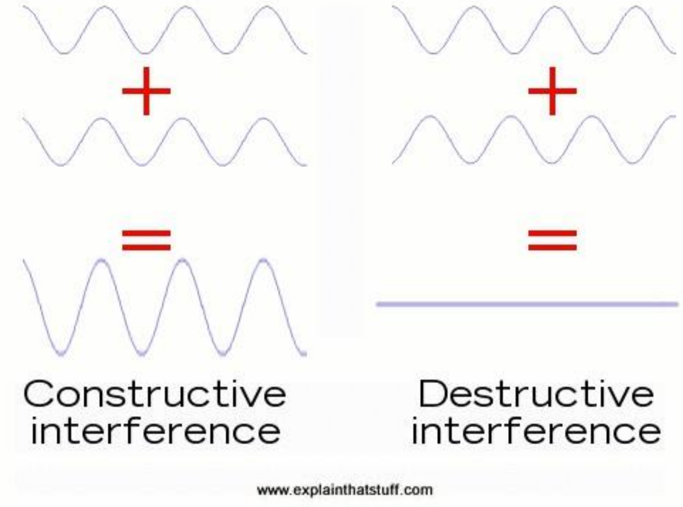


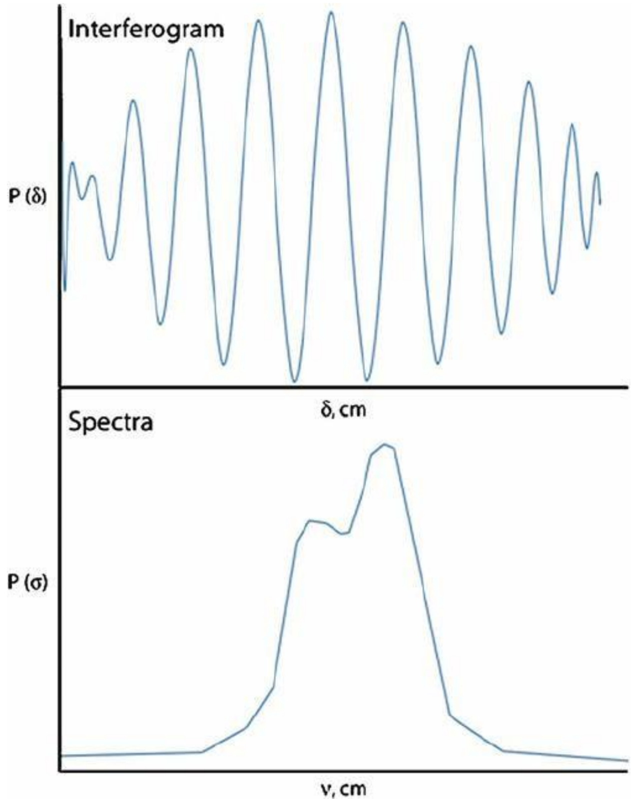
FT-IR



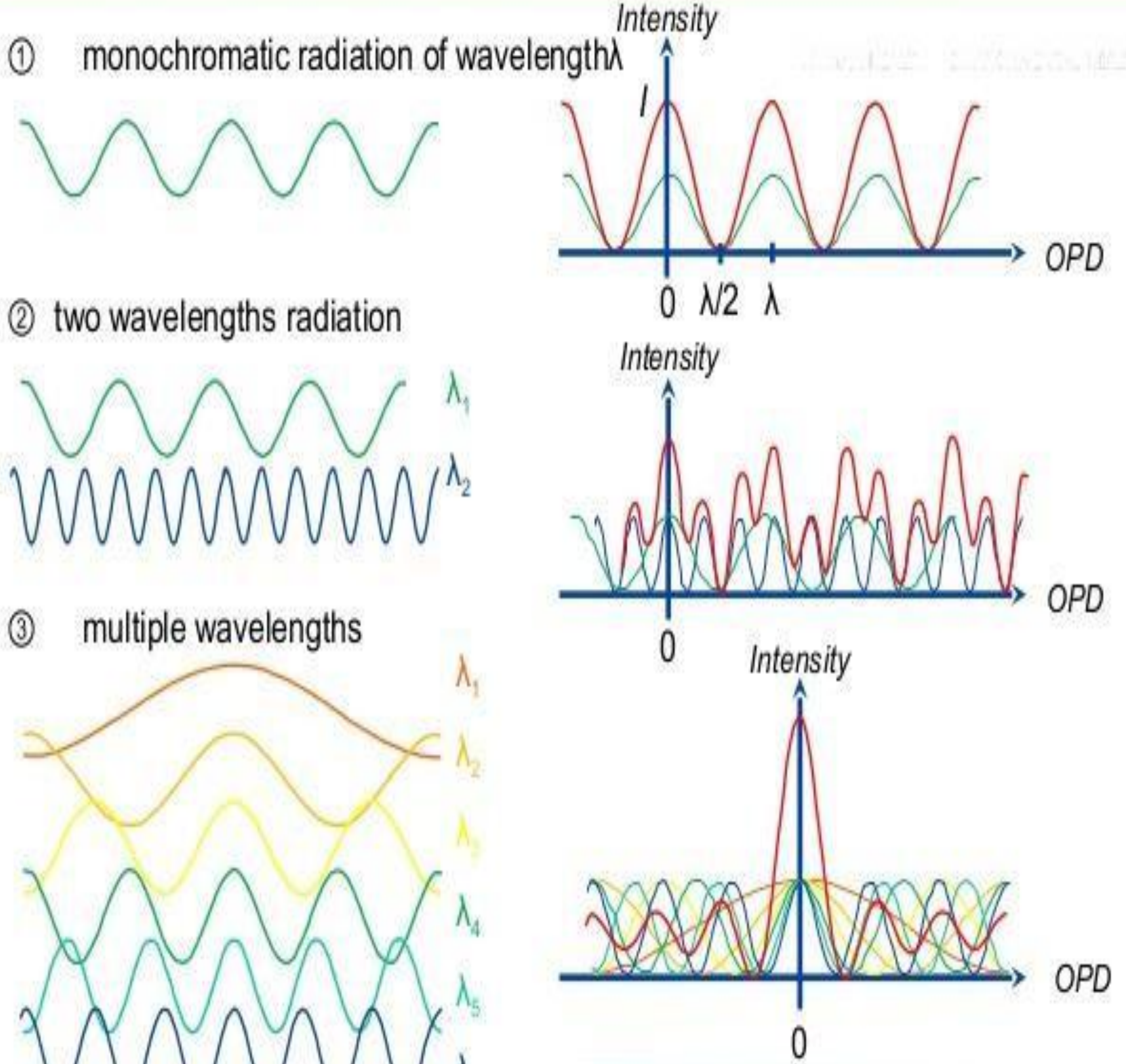
MICHELSON INTERFEROMETER







Light interference of multiple wavelengths(interferogram)



① monochromatic radiation of wavelengthλ

② two wavelengths radiation

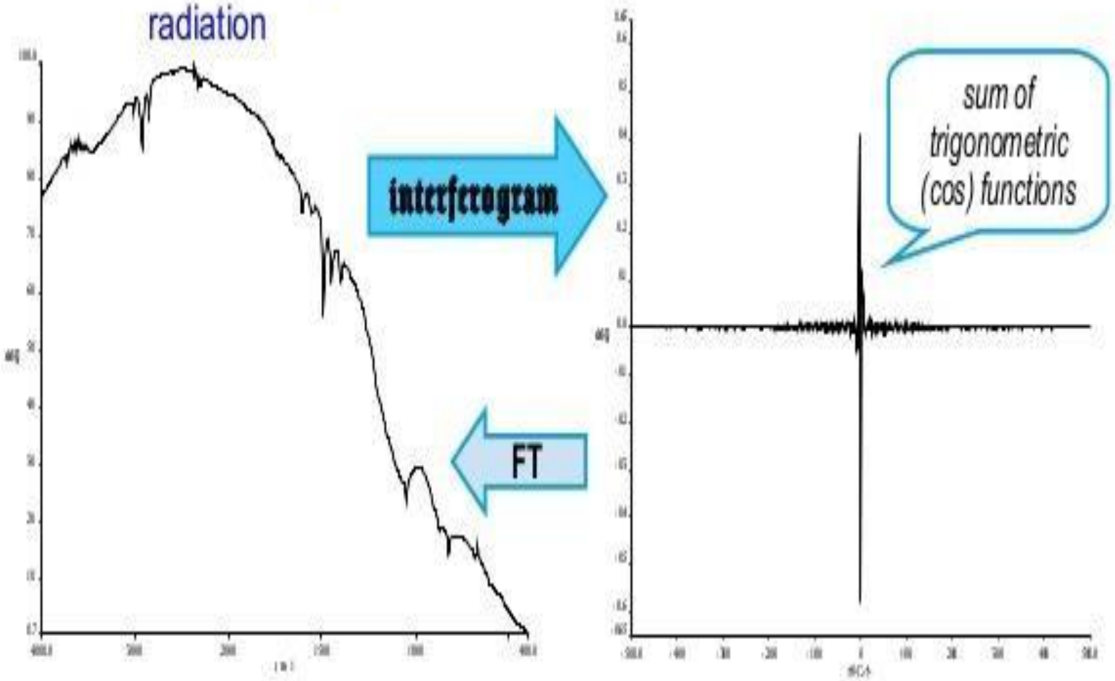
③ multiple wavelengths

intensity is strongest at 0 optical path difference

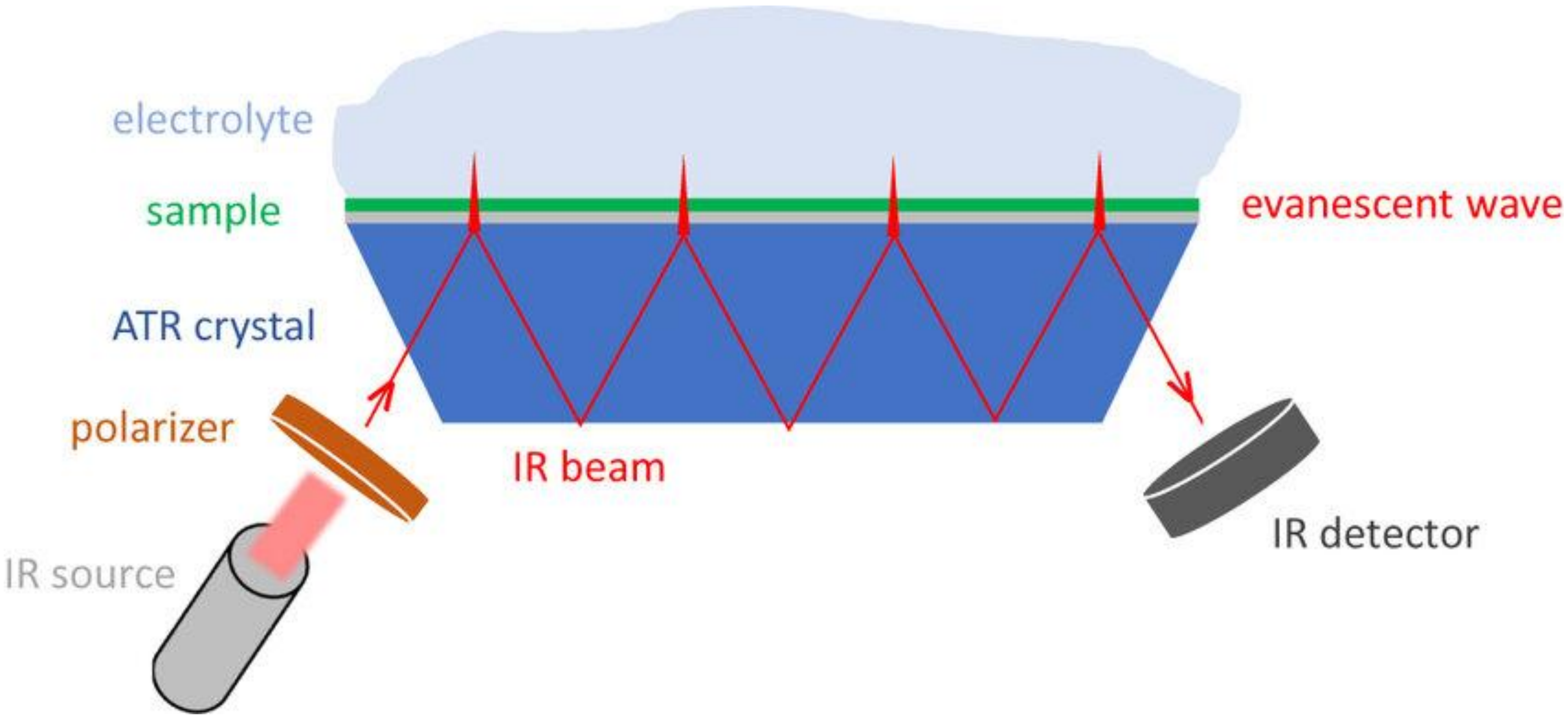
Energy in infrared region and interferogram

continuous wavelength

radiation



ATR attenuated total reflectance



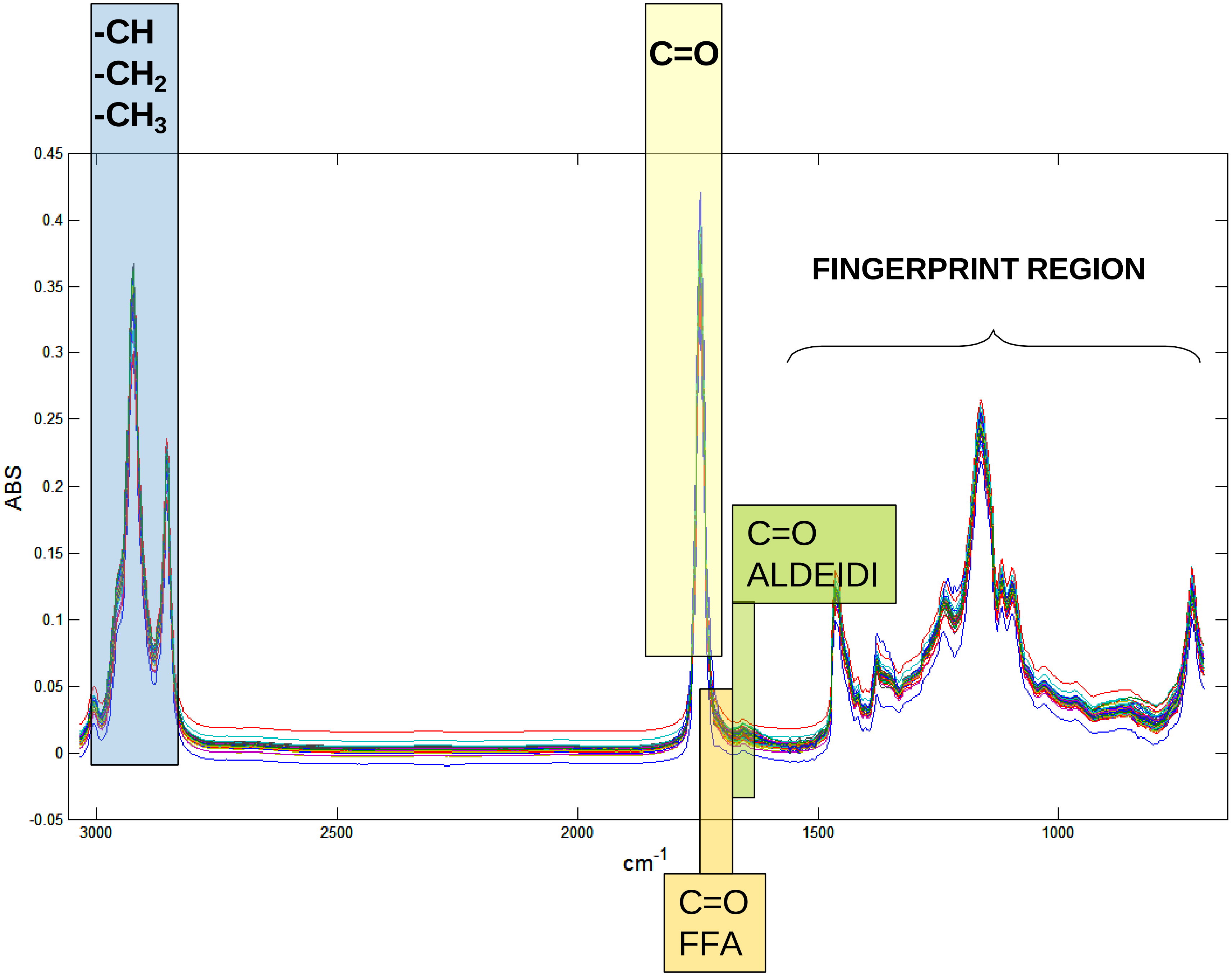
Quantitative analysis of :
- Oleic acid
- linoleic acid
- Saturated Fatty acids (SFA)
- Monounsaturated fatty acids (MUFA)
- Polyunsaturated fatty acids (PUFA)
- Peroxides value

86 samples of extravirgin olive oil from Abruzzo, Marche e Puglia (2006 e 2007)

- Chemical analysis (FA, PV, spectrophotometric indices)
- Determinaton of fatty acids (GC)

FTIR spectra acquired with Tensor 27™ FTIR (Bruker Optics, Milan, Italy), inteferometer Rocksolid™ and detector DigiTect™ with ATR. ATR (Specac Inc., Woodstock, GA, USA) had ZnSe crystal.

Spectra (32 scans/sample) acquired in the 600 to 4000 cm^{-1} range with a resolution of 4 cm^{-1}.

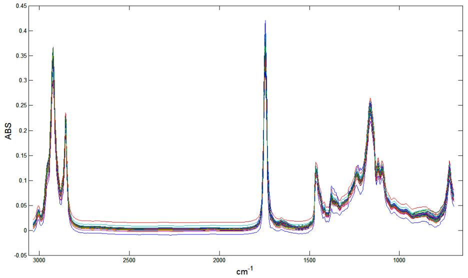

-CH
-CH_2
-CH_3
C=O
FINGERPRINT REGION
C=O
ALDEIDI
C=O
FFA
ABS
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
3000
2500
2000
1500
1000
cm^{-1}

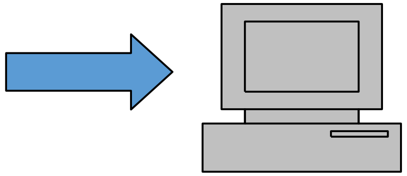
Data processing and calibration models

Data exported as ASCII file with OPUS 6.0 software and processed with a PLS routine (Partial least squares) run on Matlab (Mathworks Inc., Natick, MA, USA).

For each parameter a PLS model has been built starting from a training set and taking as true value the data obtained using GC or chemical analysis

Spectra initially processed entirely have been reduced using a "moving-windows" strategy with a Matlab routine.





Property	MUFA	PUFA	SFA
Calibration			
Spectral range (cm^{-1})	700-3033	700-3033	700-3033
Linear range (% in VOO)	64 - 81	13 - 20	6 - 16
Number of factors (LVs)	14	15	13
Number of training samples (*N*)	61	61	61
PRESS[a]	10.59	2.52	6.11
Root mean square deviation (RMSD, %)	0.42	0.20	0.32
Relative error in calibration (REC %)	0.56	2.23	1.95
r^2	0.9883	0.9941	0.9557
Selectivity	0.1734	0.1988	0.1378
Sensitivity (SEN)	0.0009	0.0015	0.0020
Analytical sensitivity, [γ= (SEN/σ_o)]	0.17	2.07	0.32
Minimum concentration difference	6.0	0.48	3.17
Limit of detection (LOD, % in VOO)	3	0.28	1.3
Limit of quantification (LOQ, % in VOO)	10	0.94	4.5
Validation			
Number of validations samples	25	25	25
Recovery rates (%)	100	103	98
Relative error in Prediction, (REP, %)	1	4	6
r^2	0.8884	0.9816	0.7099
y_o	5 ± 5	0.4 ± 0.2	5 ± 1
Slope	0.93 ± 0.07	0.98 ± 0.03	0.7 ± 0.1

Results I: oleic and linoleic acid

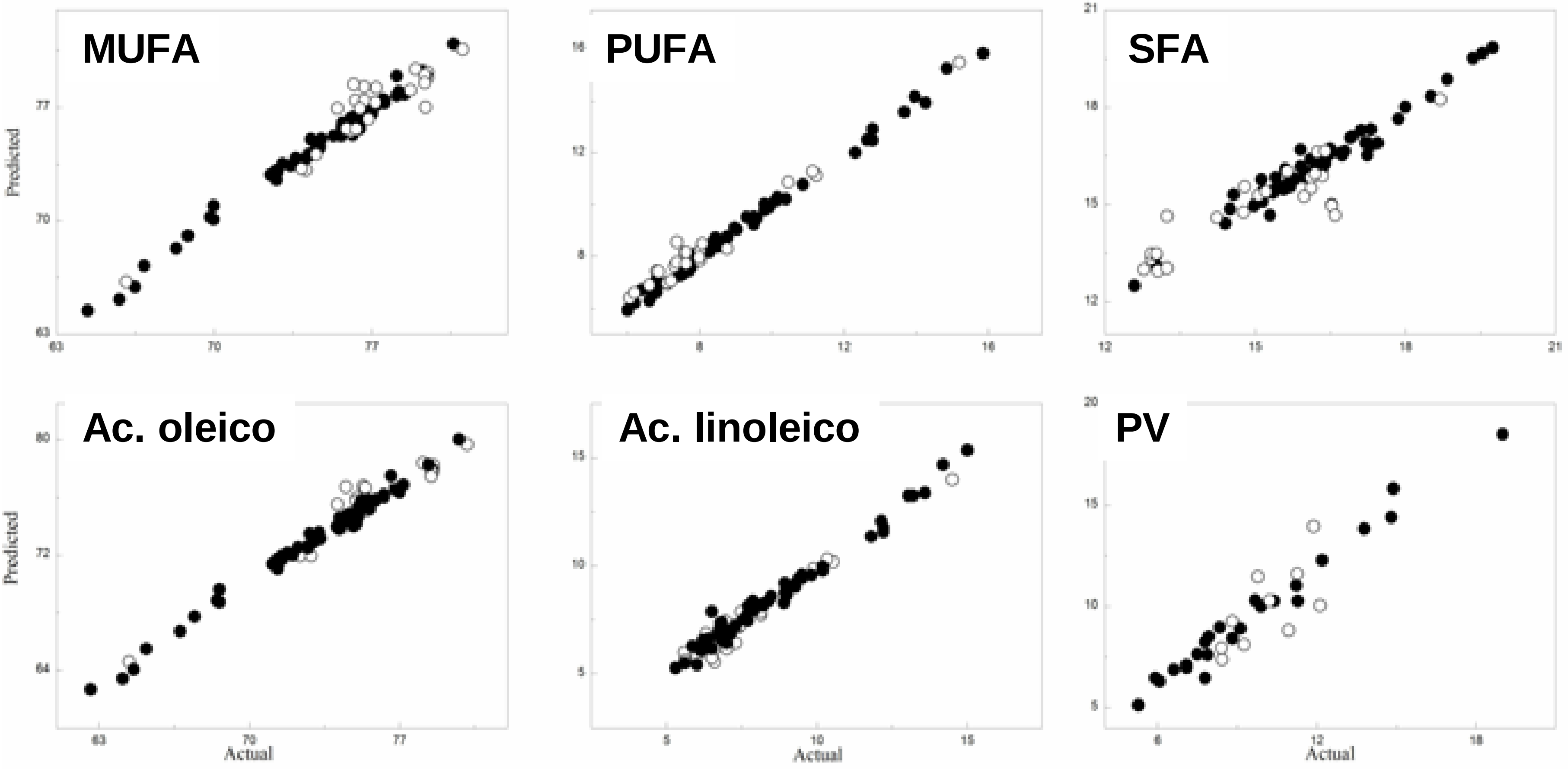
Property	Oleic Acid	Linoleic Acid
Calibration		
Spectral range (cm^{-1})	700-3033	700-3033
Linear range (% in VOO)	62 - 80	5 - 15
Number of factors (LVs)	14	13
Number of training samples (*N*)	61	61
PRESS[a]	10.88	9.33
Root mean square deviation (RMSD)	0.42	0.39
Relative error in calibration (REC %)	0.51	4.64
r^2	0.9886	0.9773
Selectivity	0.1785	0.1988
Sensitivity (SEN)	0.0009	0.0016
Analytical sensitivity, [γ= (SEN/σ_o)]	0.18	1.17
Minimum difference (%)	5.6	0.9
Limit of detection (LOD, % in VOO)	3	0.5
Limit of quantification (LOQ, % in VOO)	10	1.7
Validation		
Number of validation samples	25	25
Recovery rates (%)	100	98
Relative error in Prediction (REP %)	1	7
r^2	0.9232	0.9444
y_o	4 ± 4	0.1 ± 0.4
Slope	0.94 ± 0.06	0.96 ± 0.05

Results II: MUFA, PUFA, SFA

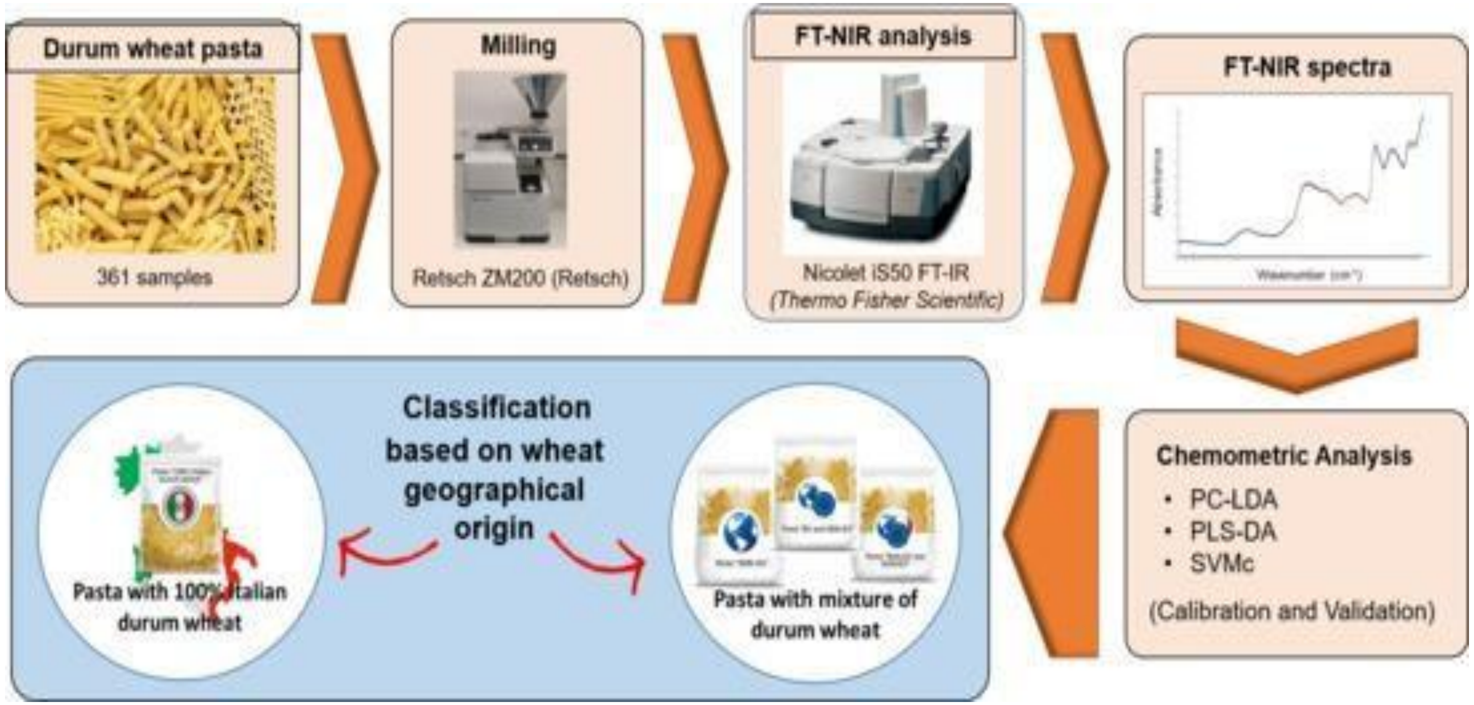
Property	MUFA	PUFA	SFA
Calibration			
Spectral range (cm^{-1})	700-3033	700-3033	700-3033
Linear range (% in VOO)	64 - 81	13 - 20	6 - 16
Number of factors (LVs)	14	15	13
Number of training samples (*N*)	61	61	61
PRESS[a]	10.59	2.52	6.11
Root mean square deviation (RMSD, %)	0.42	0.20	0.32
Relative error in calibration (REC %)	0.56	2.23	1.95
r^2	0.9883	0.9941	0.9557
Selectivity	0.1734	0.1988	0.1378
Sensitivity (SEN)	0.0009	0.0015	0.0020
Analytical sensitivity, $[\gamma = (SEN/\sigma_o)]$	0.17	2.07	0.32
Minimum concentration difference	6.0	0.48	3.17
Limit of detection (LOD, % in VOO)	3	0.28	1.3
Limit of quantification (LOQ, % in VOO)	10	0.94	4.5
Validation			
Number of validations samples	25	25	25
Recovery rates (%)	100	103	98
Relative error in Prediction, (REP, %)	1	4	6
r^2	0.8884	0.9816	0.7099
y_o	5 ± 5	0.4 ± 0.2	5 ± 1
Slope	0.93 ± 0.07	0.98 ± 0.03	0.7 ± 0.1

Results III: PV

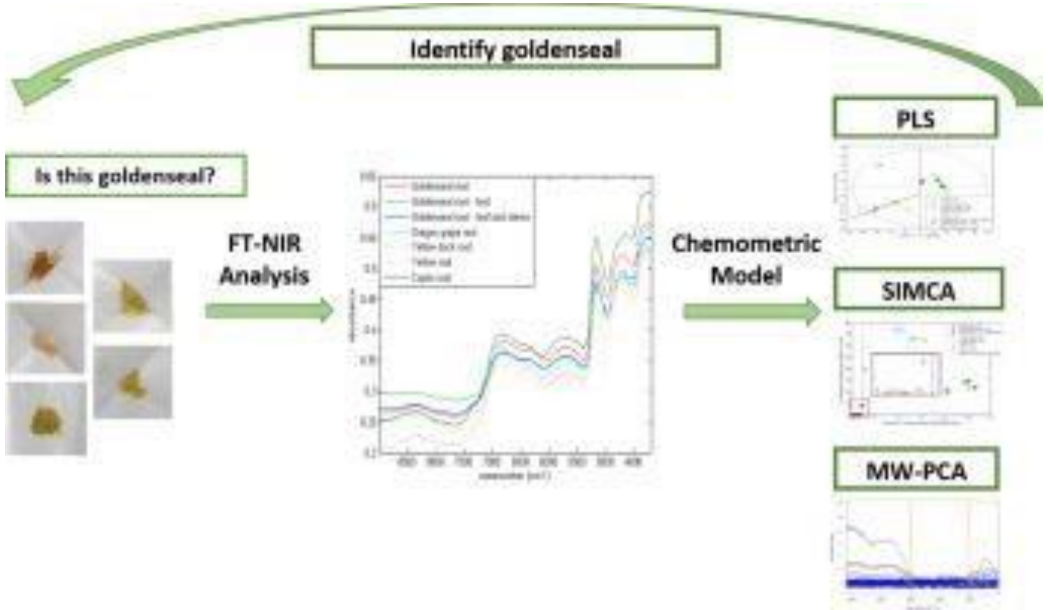
Property	D^{o}	D’	D”
Spectral range (cm^{-1})	4000-700	4000-700	4000-700
Calibration range (meqO$_2$ kg^{-1}oil)	5.7-15.7	5.7-15.7	5.7-15.7
Number of factors (LV)	5	10	7
Number of training samples	23	24	24
PRESS[a] (unidades)	174.66	152.35	191.32
Root mean square deviation, RMSD (unidades)	1.4302	0.6933	0.9482
Relative error in calibration, REC (%)	15.6	7.2	9.9
r^2	0.8040	0.9759	0.9446
Selectivity	1.0	0.35	0.55
Sensitivity (SEN)	0.0044	0.0001	0.0001
Analytical sensitivity, [γ= (SEN/σ_o)]	1.2	1.1	1.1
Minimum concentration difference (unidades)	0.8	0.9	0.9
Limit of detection (LOD) (unidades)	3.1	1.0	1.6
Limit of quantification (LOQ) (unidades)	10.3	3.4	5.2
Number of validations samples	10	10	10
Recovery rates (%)	74.7	97.7	96.0
Relative error in Prediction, REP (%)	23.7	13.6	12.2



calibration set (●) and trainingset (○)



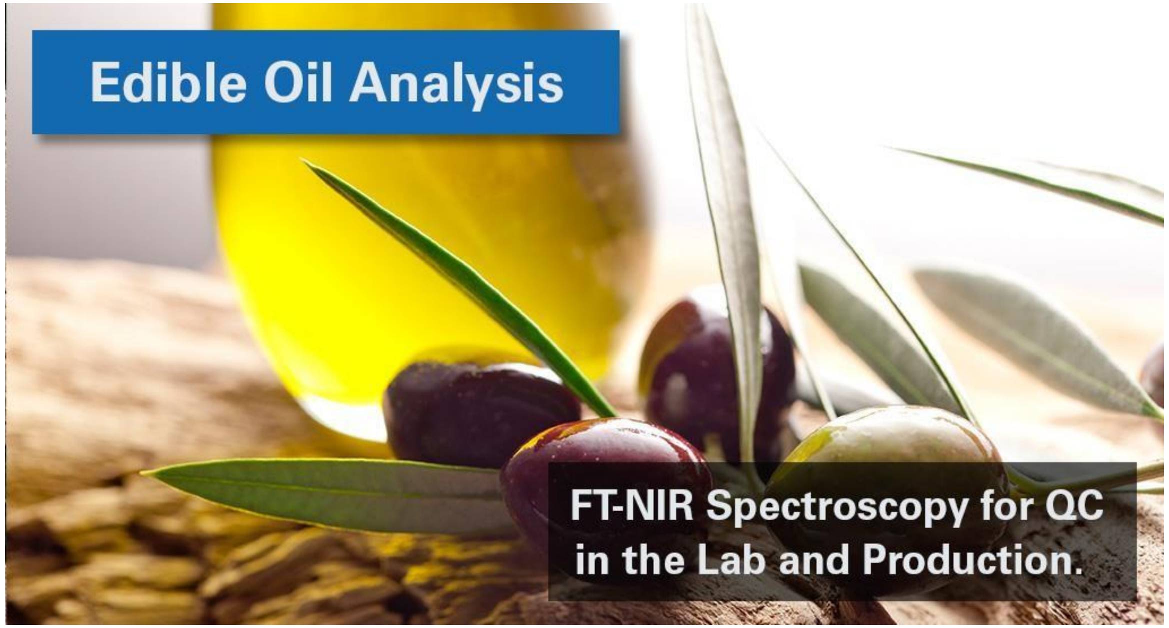
De Girolamo A, Cervellieri S, Mancini E, Pascale M, Logrieco AF, Lippolis V. Rapid Authentication of 100% Italian Durum Wheat Pasta by FT-NIR Spectroscopy Combined with Chemometric Tools. *Foods*. 2020; 9(11):1551. https://doi.org/10.3390/foods9111551

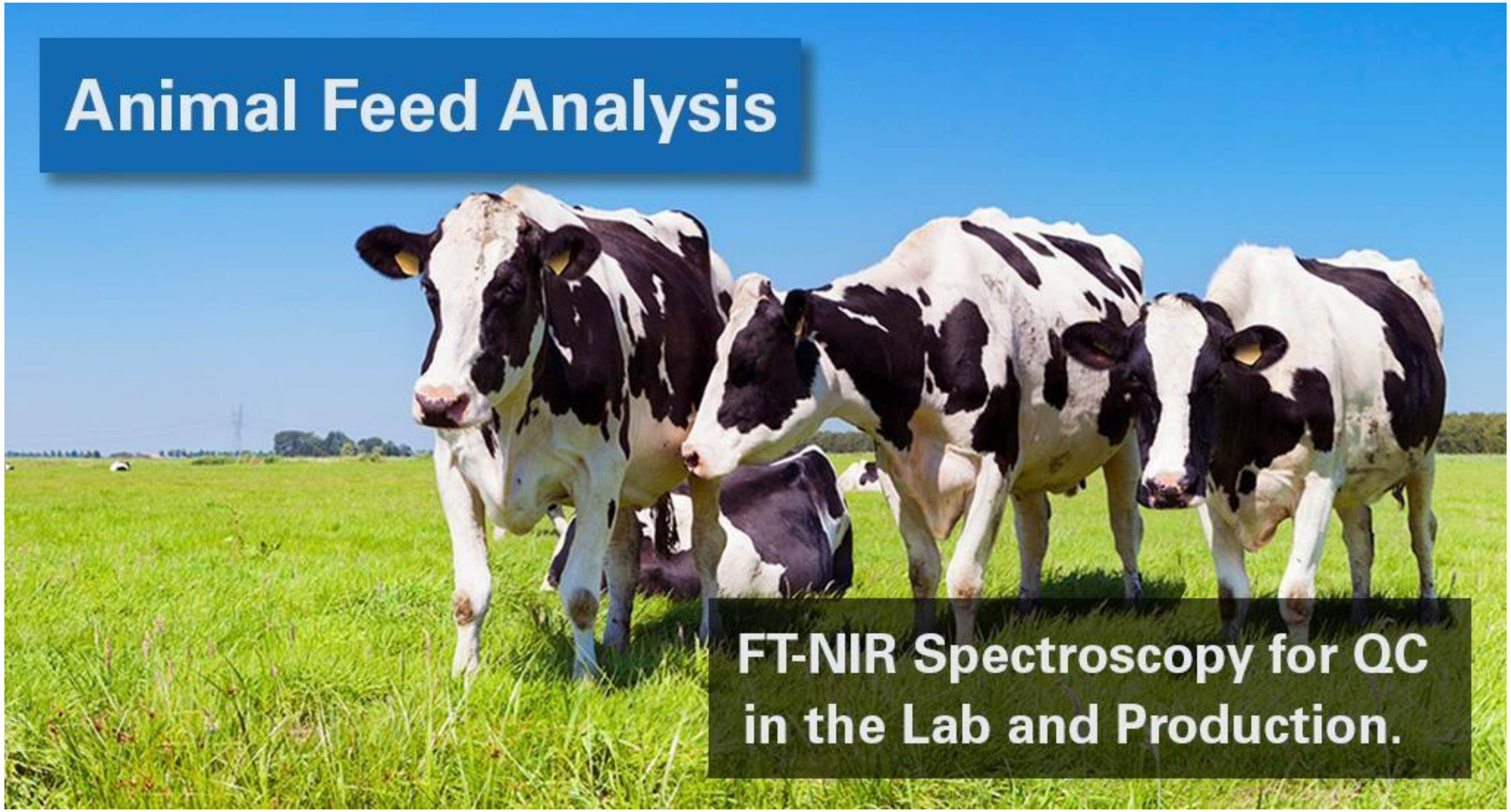


Ying Liu, Jamie Finley, Joseph M. Betz, Paula N. Brown, FT-NIR characterization with chemometric analyses to differentiate goldenseal from common adulterants, Fitoterapia, Volume 127,2018, Pages 81-88,


Final Grind


Food Analysis
FT-NIR Spectroscopy
for QC in the Lab and Production.

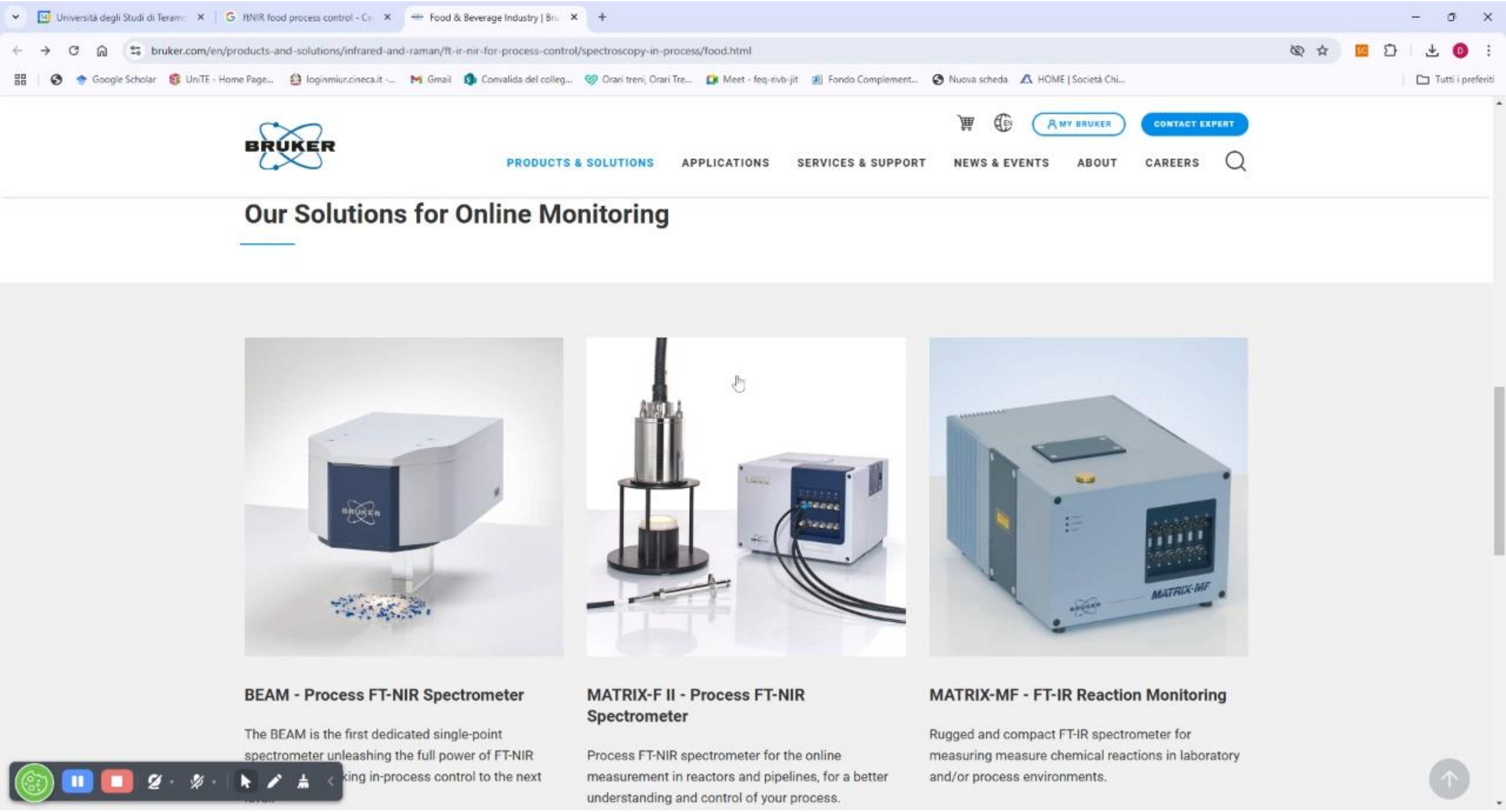

Edible Oil Analysis
FT-NIR Spectroscopy for QC
in the Lab and Production.



Animal Feed Analysis

FT-NIR Spectroscopy for QC
in the Lab and Production.

- Application Note 52269. The Antaris FT-NIR analyzer provides a rapid solution to accurately quantify the key flour components of moisture, protein, and ash. TQ Analyst offers easy and intuitive PLS calibration optimization and development through its visual and interactive diagnostic tools, such as Statistical Spectra, Residual, PRESS, and 2D and 3D PC scores plots.



Our Solutions for Online Monitoring

BEAM - Process FT-NIR Spectrometer

The BEAM is the first dedicated single-point spectrometer unleashing the full power of FT-NIR king in-process control to the next

MATRIX-F II - Process FT-NIR Spectrometer

Process FT-NIR spectrometer for the online measurement in reactors and pipelines, for a better understanding and control of your process.

MATRIX-MF - FT-IR Reaction Monitoring

Rugged and compact FT-IR spectrometer for measuring measure chemical reactions in laboratory and/or process environments.