



The Abdus Salam
International Centre for Theoretical Physics



SMR 1829 - 23

Winter College on Fibre Optics, Fibre Lasers and Sensors

12 - 23 February 2007

Spectroscopy-based
Fiber Optic Sensors

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Department of Optoelectronics and Photonics

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CNR-IFAC, Sesto Fiorentino (FI), Italy



Useful Books

- T. Okoshi "Optical Fibers", Academic Press Inc., London, 1982
- B.P. Pal "Fundamentals of Fiber Optics in Telecommunication and Sensor Systems", Wiley Eastern Ltd., New Delhi, 1992
- B. Culshaw, J.P. Dakin "Optical Fiber Sensors", Artech House, London
 - vol I: Principles and Components (1988)
 - vol II: Systems and Applications (1989)
 - vol III: Components and Subsystems (1996)
 - vol IV: Applications, Analysis and Future Trends (1997)
- O.S. Wolfbeis "Fiber Optic Chemical Sensors and Biosensors", Vol. I- II, CRC Press, Boca Raton FL, 1991
- K.T.V. Grattan, B.T. Meggit "Optical Fiber Sensor Technology", Chapman & Hall, London, 1995
- K.T.V. Grattan, B.T. Meggit "Optical Fiber Sensor Technology", Kluwer Academic Publishers, Dordrecht, The Netherlands
 - vol 3: Applications and Systems (1999)
 - vol 4: Chemical and Environmental Sensing (1999)
- D.A. Krohn "Fiber Optic Sensors, Fundamentals and Applications", ISA, Research Triangle Park, NC, 2000
- J.M. Lòpez-Higuera "Handbook of Optical Fibre Sensing Technology", John Wiley & Sons, Chichester, UK, 2001

On-line bibliography on optical fibers and sensors

- <http://www.sff.net/people/Jeff.Hecht/history.html>
- <http://electronics.howstuffworks.com/fiber-optic5.htm>
- <http://www.commspecial.com/fiberguide.htm>
- http://www.webopedia.com/TERM/f/fiber_optics.html

Outline

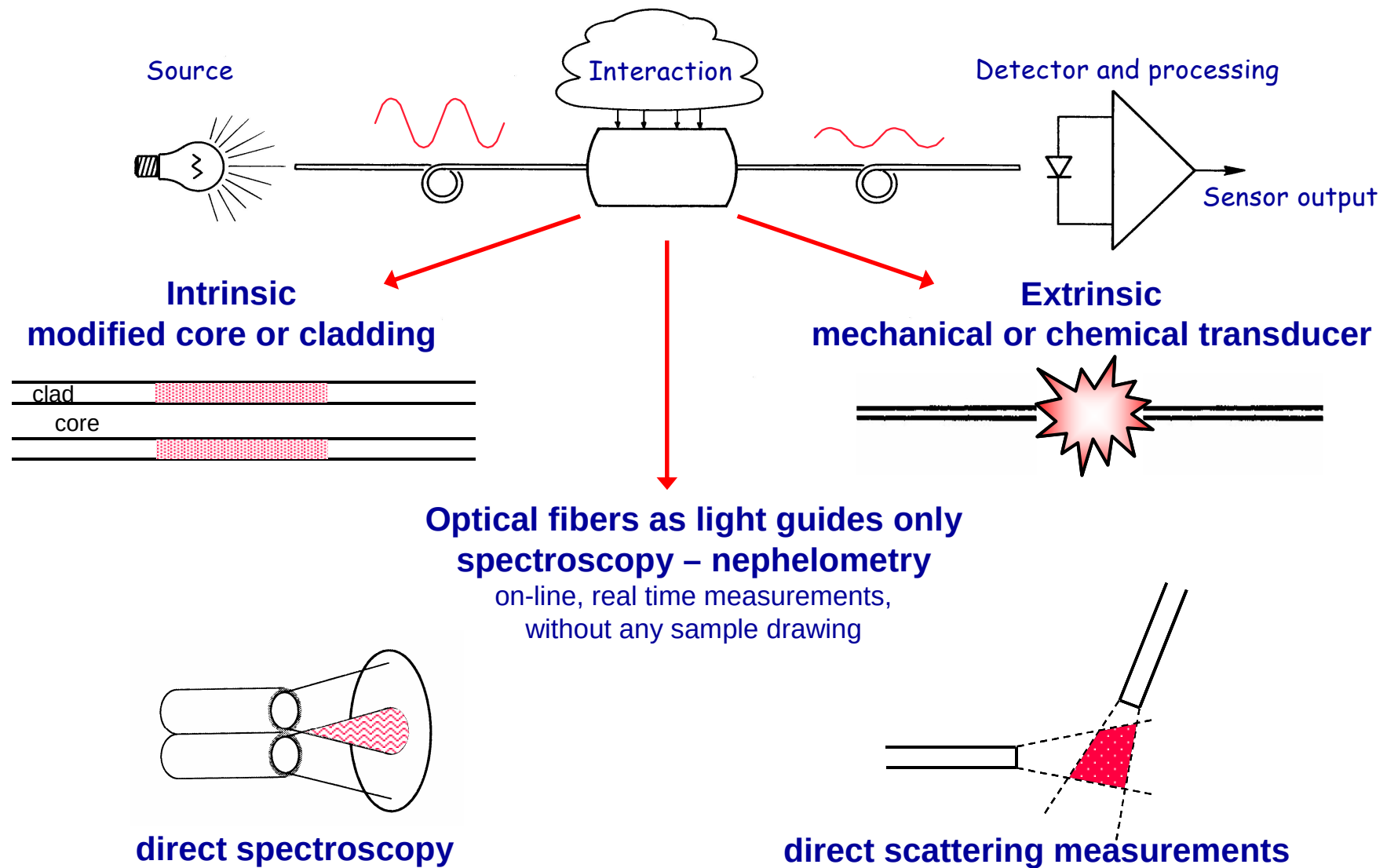
- **Optical fiber sensors: basics**
- **Colorimetry**
 - Absorption and reflection spectroscopy
 - Fiber optic spectrophotometers for general purpose applications – Direct / chemically-mediated
- **Scattered colorimetry**
 - Multi- λ and multi- α absorption spectroscopy
- **Hyperspectral colorimetry**
- **Fluorescence spectroscopy**

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Fiber optic sensors

Intensity modulation



Optoelectronic components of optical fiber sensors

Components:

- <http://www.fiberoptic.com/site/main/main.asp>
- <http://www.global-fiber-optics.com/>
- <http://www.geocities.com/SiliconValley/Circuit/8070/>
- <http://www.fiberopticeq.com/> (second-hand)

Optical Fibers:

- <http://www.corning.com/opticalfiber/> - <http://www.fiberguide.com/>
- http://www.3m.com/us/util_telecom/optical_oem/
- <http://www.artphotonics.de>

Sources, Filters and Optics, Detectors:

- <http://www.roithner-laser.com/>
- <http://www.edmundoptics.com/de/>
- <http://sales.hamamatsu.com/en/products.php?GLBSESSID=5400bd2542c1f8823a85d2055c655f4d>
- <http://www.oceanoptics.com/> - <http://www.controldevelopment.com/> - <http://www.stellarnet-inc.com/>
- <http://www.ezconn.com/en/og.htm>



ROITHNER LASERTECHNIK

- **Laser Diodes** 635 nm - 1.9 μ m
- **Optics For Laser Diodes**
- **Drivers For Laser Diodes**
- **Lasermodes** violet, blue, green, red, ir
- **Fiber Coupled Components**
- **VCSELs** 780 nm, 840 nm
- **Accessories For Laser Diodes**
- **Photodiodes** 130 nm - 3.6 μ m
- **Thermopiles** 5 μ m - 14 μ m
- **LEDs** 350 nm - 5.0 μ m
- **High Power LEDs** 370 - 1550 nm
- **White LED Torches**
- **UV-LED Pointers** 370 nm, 380 nm
- **LED Applications**
- **Laserpointers** 635 nm - 1.9 μ m
- **Green Laserpointers** 532 nm
- **Infrared Laserpointers**
- **Laser Crystals & Optics**
- **Datasheets**
- **COHERENT® Instruments**
- **Interference Filters**
- **IR - Detector Cards**
- **Superconductors**
- **Laser Safety Devices**
- **Where We Are**
- **Exhibitions, Fairs**
- **Pricelist, Links**
- **How To Order**

e-mail contact

mirror sites:
roithner-laser.com
roithner.mcb.at
laserfocus.de
chello.at
updated 2004-01-08

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Products

- Solid State Division
- Electron Tube Division
- System Division
- Laser Division



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Solid State Division

- Si Photodiode Series**
 - Si Photodiode
 - Si PIN Photodiode
 - Si Photodiode Array
 - With Preamp/Cooler
 - Si APD
 - X-ray Detectors
- Photo IC Series**
 - Photo IC
 - Photoreflector
 - Photointerrupter
 - Phototransistor
- Position Sensitive Detectors**
 - One-dimensional
 - Two-dimensional
 - Special Type
 - Signal Processing Circuit
- InGaAs PIN Photodiodes**
 - Standard Type
 - Long Wavelength Type
 - Image Sensor/Array
 - Infrared Detector Module
- Optical Communication Devices**
 - High-speed
 - Low-speed, Short-haul
 - Spatial Light Transmission

Electron Tube Division

- Detectors**
 - Photomultiplier Tubes (PMTs)
 - PMT Modules
 - Ion Detectors
 - Phototubes
 - Electron Multipliers
 - Microchannel Plates (MCPs)
 - Image Intensifiers
 - UV Sensors
 - Spatial Light Modulator
 - Accessories
- Light Sources**
 - Xenon Lamps
 - Mercury-Xenon Lamps
 - Xenon Flash Lamps
 - Deuterium Lamps
 - Hollow Cathode Lamps
 - Light Source Solutions
 - Accessories
 - UVSpot
- X-ray Products**
 - Microfocus X-ray Source (MFX)
 - X-ray Image Intensifiers
 - X-ray Tubes
 - Scintillators
- Others**

System Division

- Cameras**
 - High Resolution
 - High Sensitivity
 - Color
 - IR / UV / X-ray
 - High Speed
- Bio Imaging Instruments**
 - US Solutions
 - European Solutions
- Machine Vision**
- Ultra Fast**
 - Streak Systems
 - Fluorescence Lifetime Spectroscopy
 - Picosecond Light Sources
- Non-Destructive Testing**
- Semiconductor Industry**
 - Failure Analysis
 - Process Control & Monitoring
 - Yield Management
- Laser & Fibre Optic Measurement**
 - Beam Analysis
 - Optical Oscilloscopes
 - DMD Workstations
 - Picosecond Light Sources
 - Streak Systems
 - Real-Time IR Spectrometer

Laser Division

- CW Laser Diode & Array
- Pulsed Laser Diode
- Super Luminescent Diode
- MSM Photodetector

Outline

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- **Colorimetry**
 - Absorption and reflection spectroscopy
 - Fiber optic spectrophotometers for general purpose applications – Direct / chemically-mediated
- **Scattered colorimetry**
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- **Hyperspectral colorimetry**
- **Fluorescence spectroscopy**

Colorimetry:

- Fiber-optic spectrophotometers for absorption and reflection spectroscopy
 - Direct:
 - Gasoline blendings
 - Water color monitoring in a recycling plant
 - Chemically-mediated:
 - pH, pCO₂
 - Lighting monitoring in museums
 - Fiber optic multimeter for spectral interrogation of opto-chemical sensor arrays
 - Metal ions in water
 - Artificial olfactory perception of olive oil rancidity

Colorimetry:

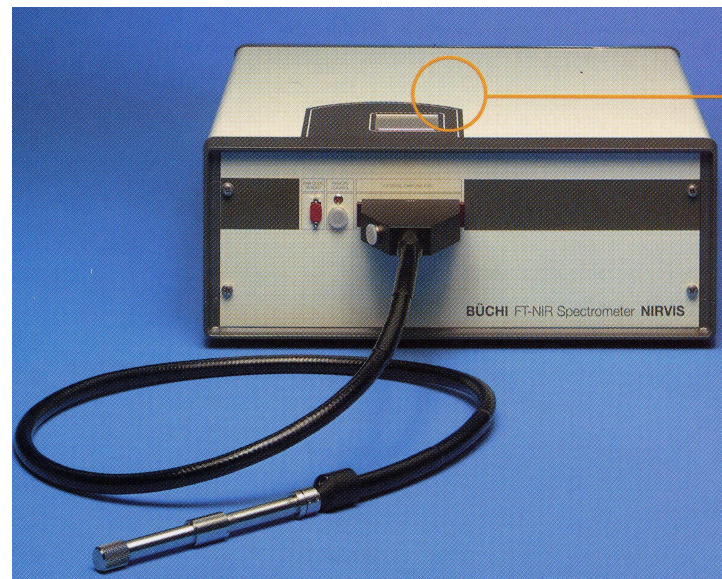
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Spectrophotometers equipped with optical fibers

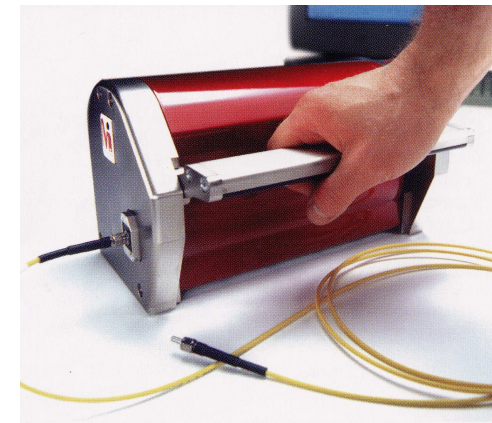
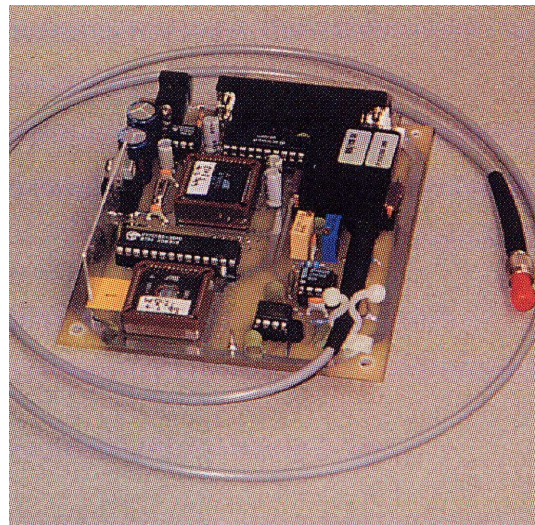
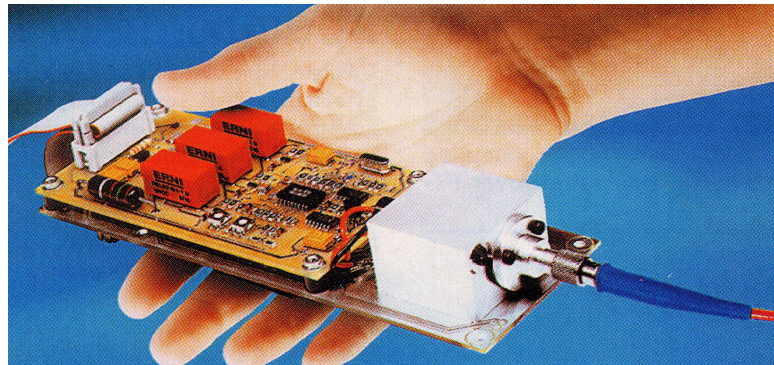
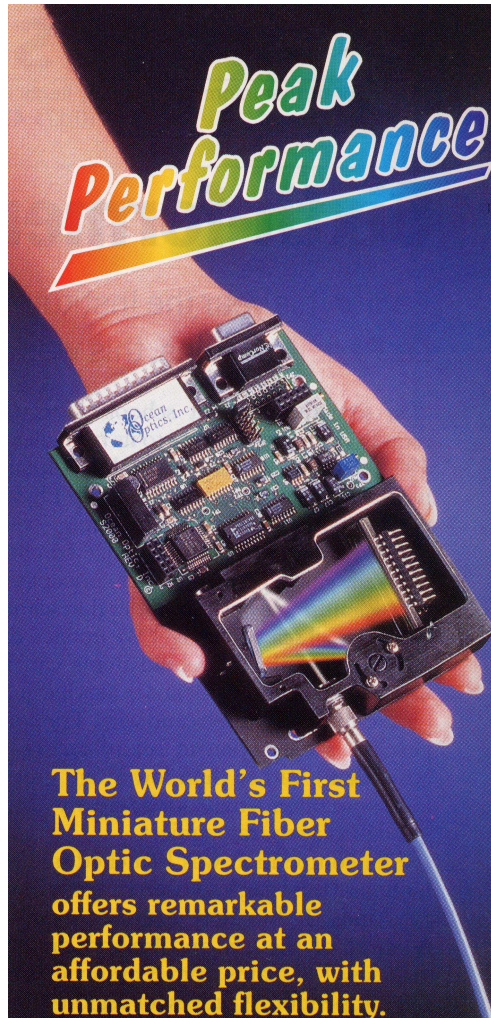


Drawbacks

- limited fiber length
- no multiplexing
- poor compactness



Optical Fiber Microspectrometers



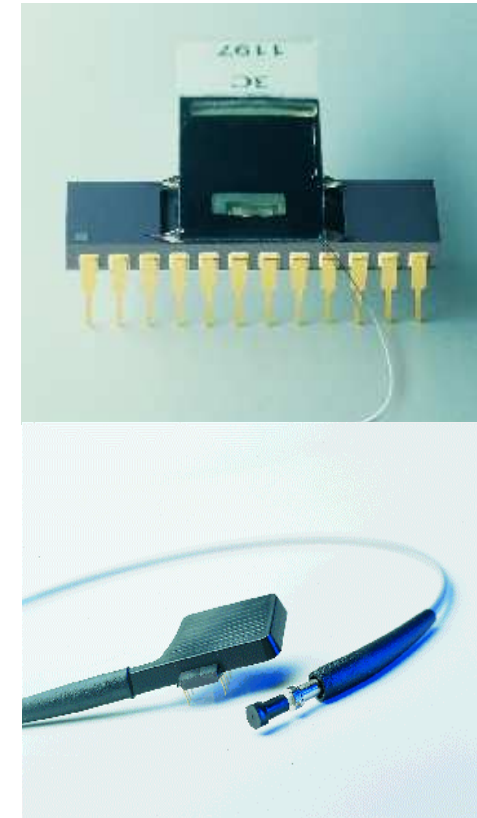
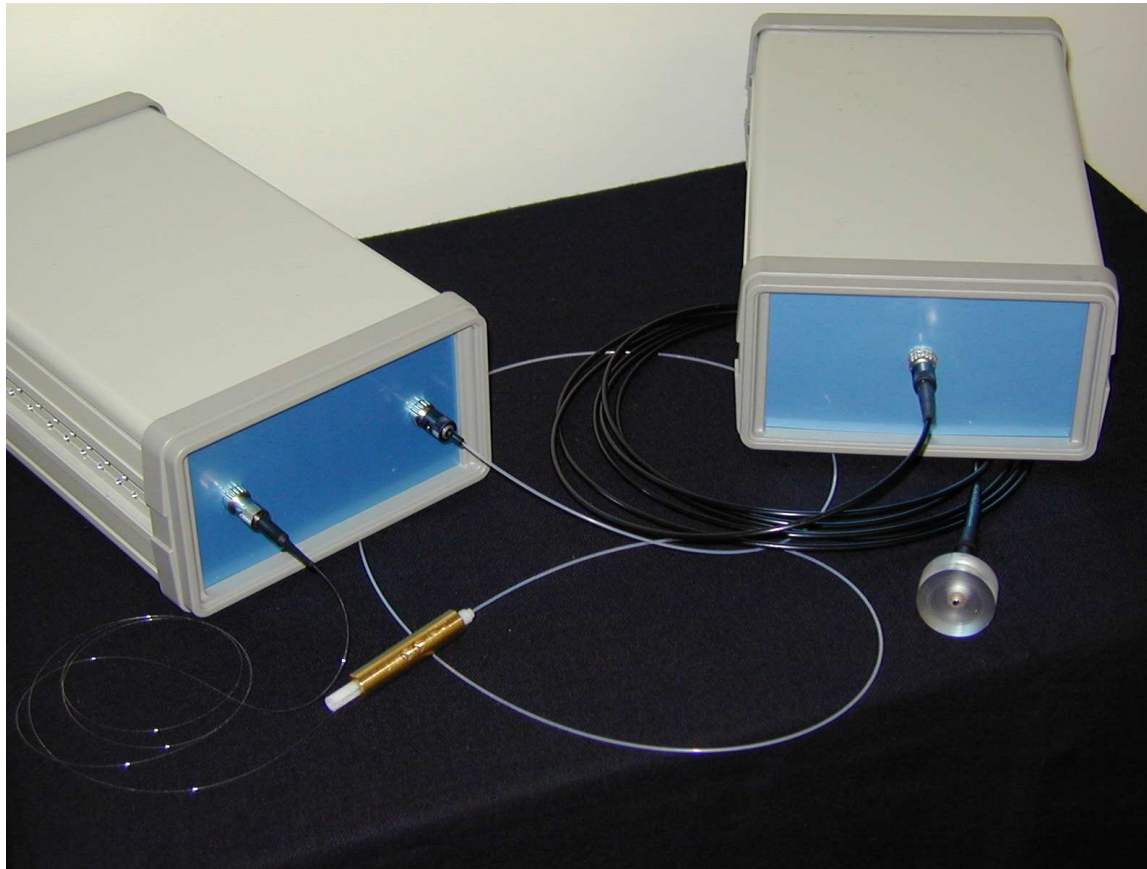
..... and many more

Absorption and reflection spectroscopy

Main requirements:

- High degree of compactness
 - Sources, detectors, and coupling optics must fit into a compact box
 - No mechanical alignments of the optical components
- Low cost optoelectronic components
 - LED sources
 - Fiber optic compatible spectrometer
 - Thermal stability
- Probe using a single optical fiber configuration, rather than bundle
- A flexible software interface friendly enough to be used by operators with little technical background

Compact and low-cost spectrophotometers for absorption and reflection spectroscopy

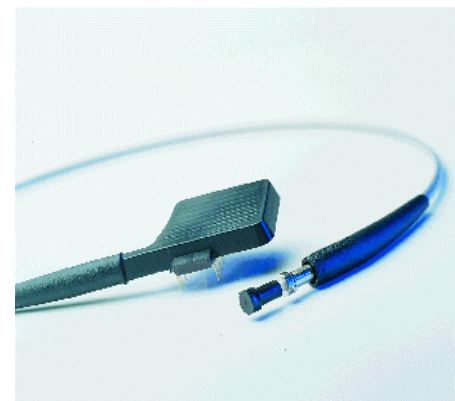
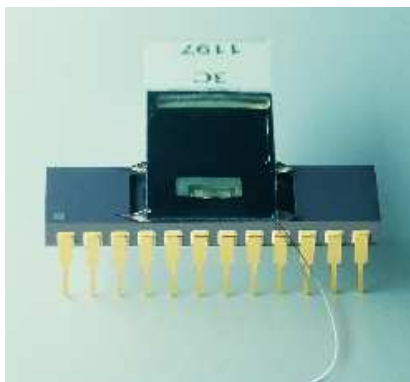
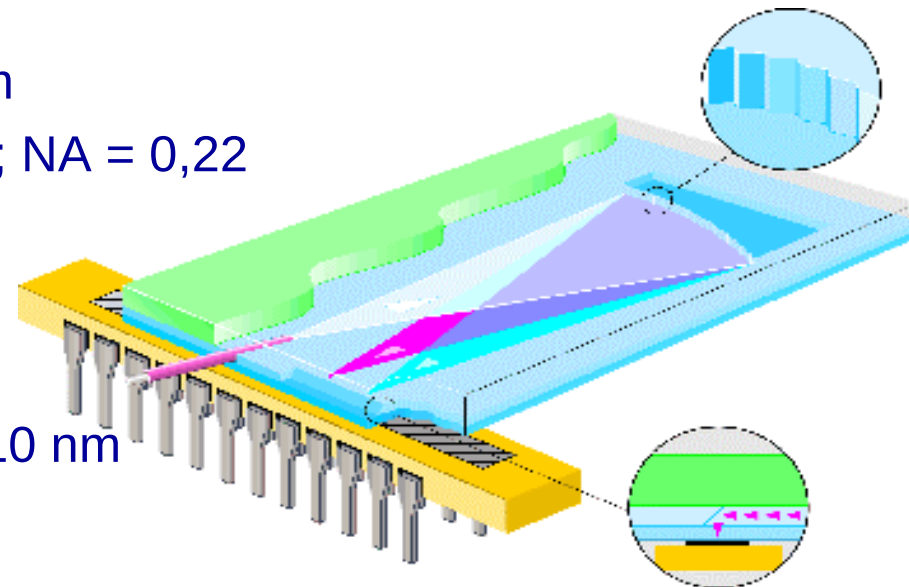


- ✓ spectral range: 400-700 nm
- ✓ resolution: 10 nm

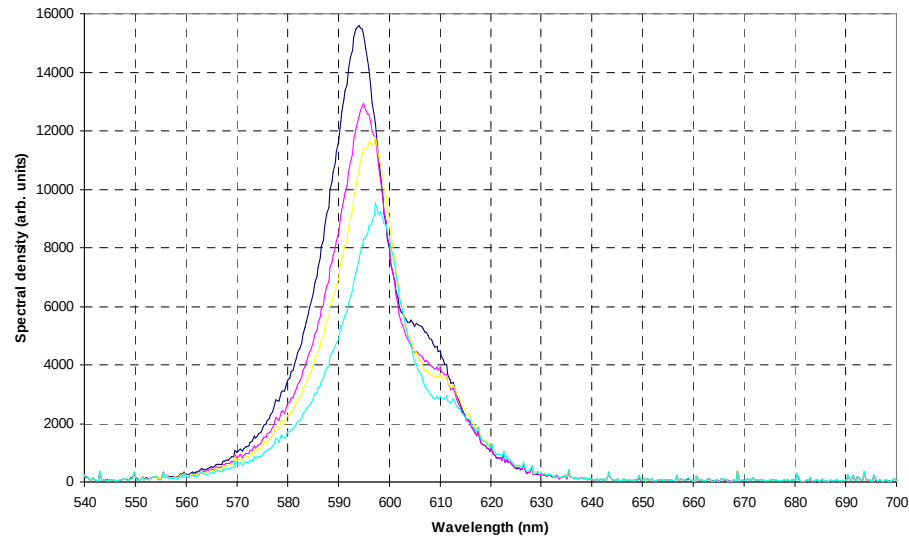
VIS-Microspectrometer

Technical Data:

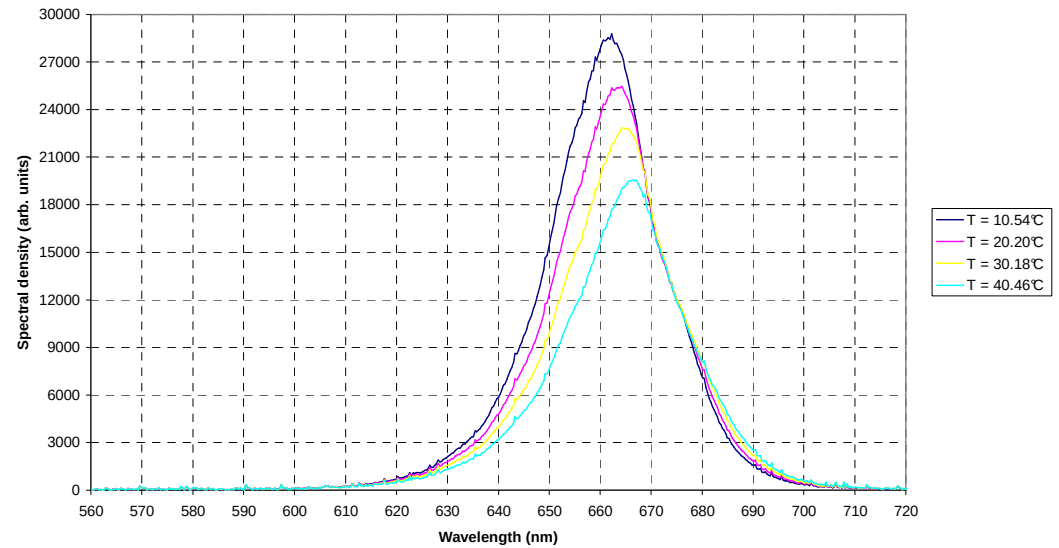
- Spectral range from 380 - 780 nm
- Fibre: 50 / 105 μm core diameter; NA = 0,22
- Blazed Grating: 600 lines / mm
- Dispersion: 2,9 nm / Pixel
- Throughput: 30%
- Spectral resolution FWHM : 7nm / 10 nm
- Dynamic ratio: > 10.000



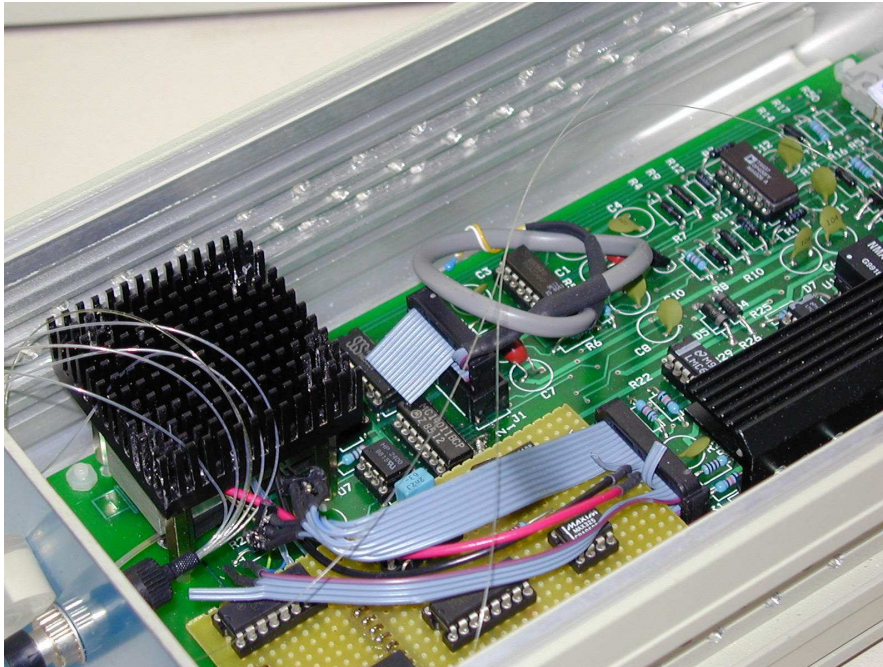
Spectrum of LED Ledtronics L200CWY3KB @ 590 nm
Modulation: Square Wave @ 440 Hz, 50% duty cycle - 0-25 mA



Spectrum of LED Ledtronics L200CWR5K @ 660 nm
Modulation: Square Wave @ 440 Hz, 50% duty cycle - 0-25 mA

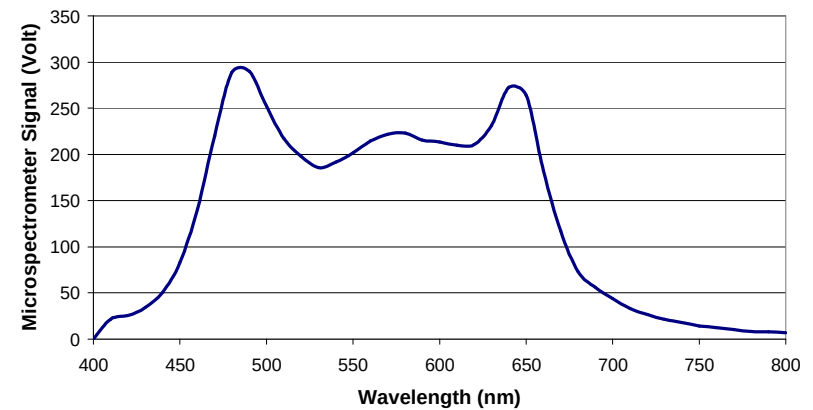


2-LED source



Thermal stabilization and microoptic joint
of the module for reflection spectroscopy

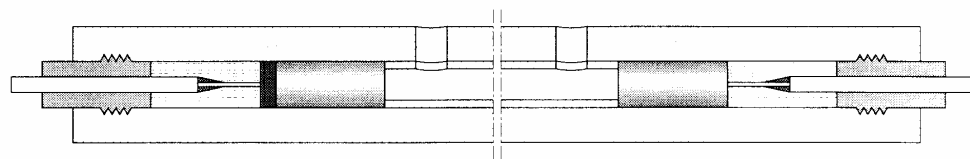
2-LED Source



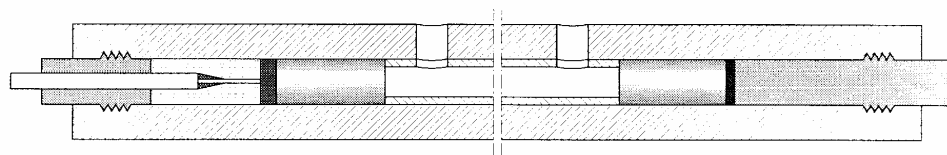
Optical fiber probe for reflection spectroscopy



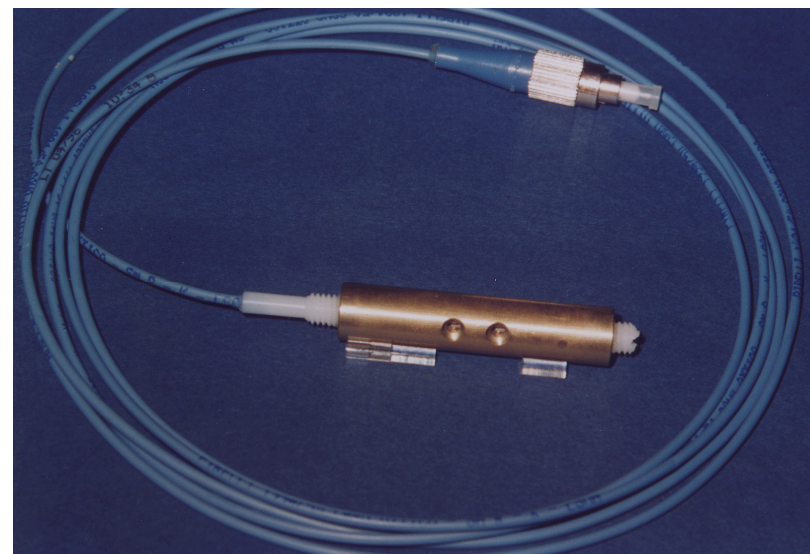
Probes for on-line absorption spectroscopy



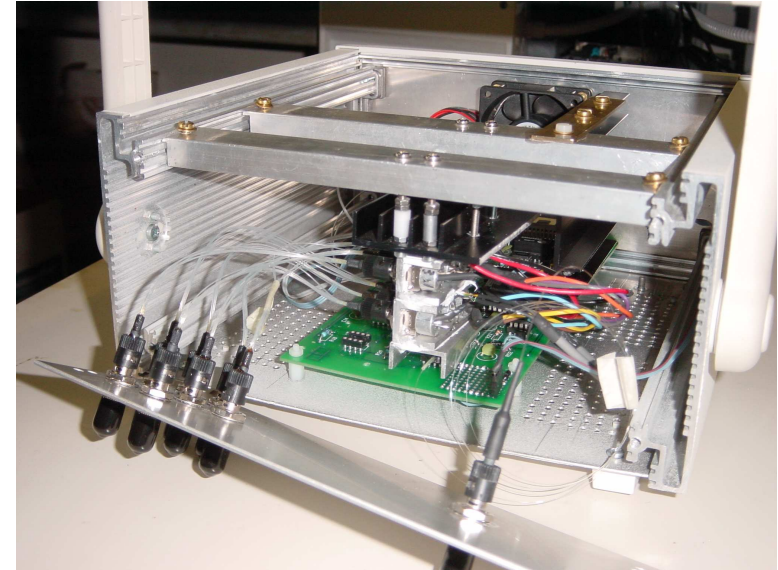
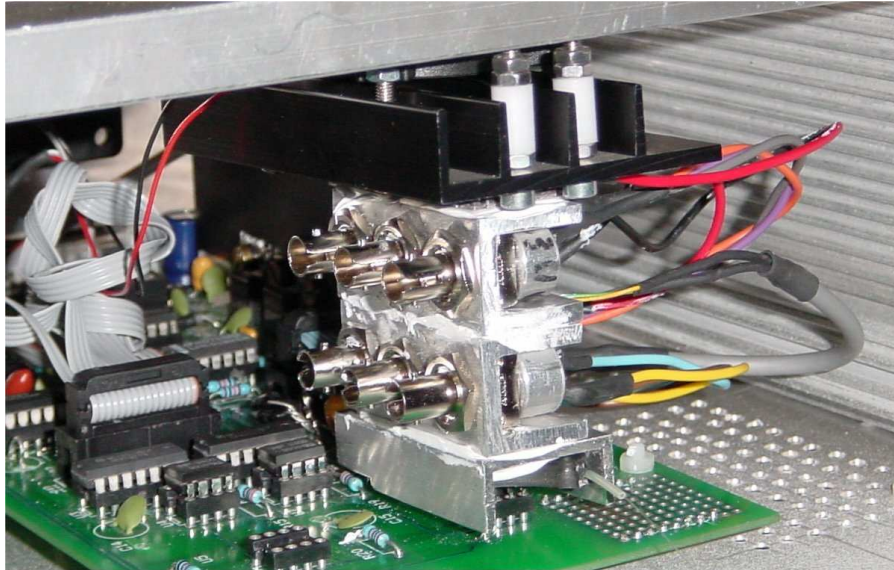
transmissive



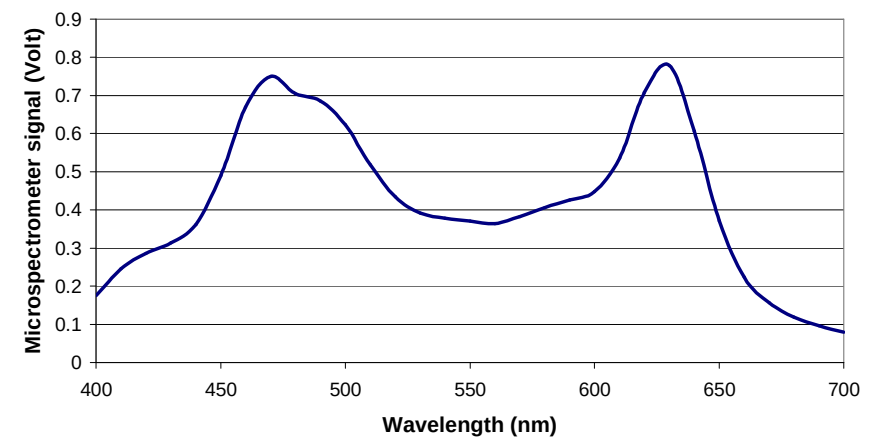
transmissive
folded path



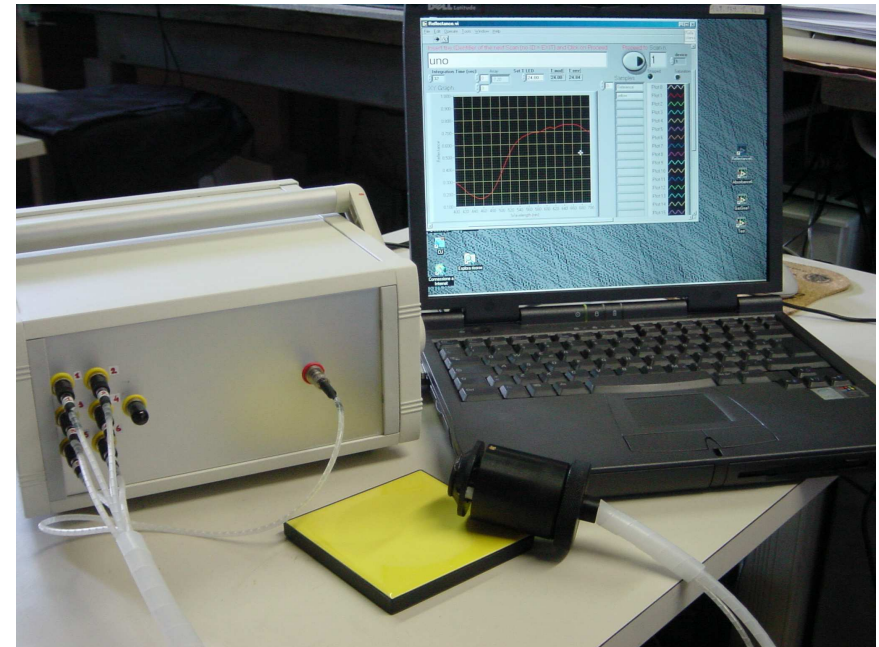
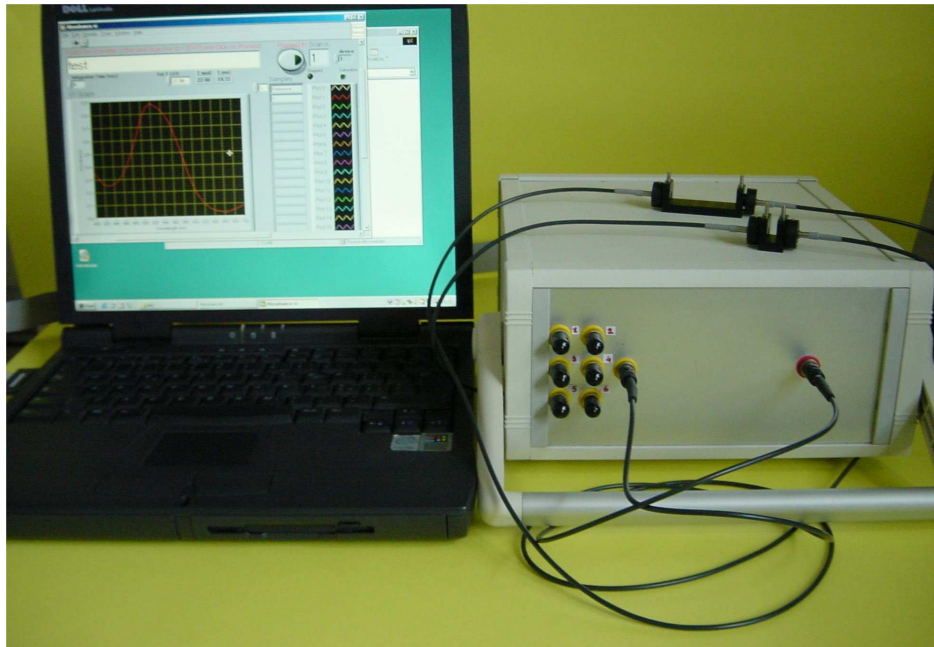
6-LED source



6-LED Source

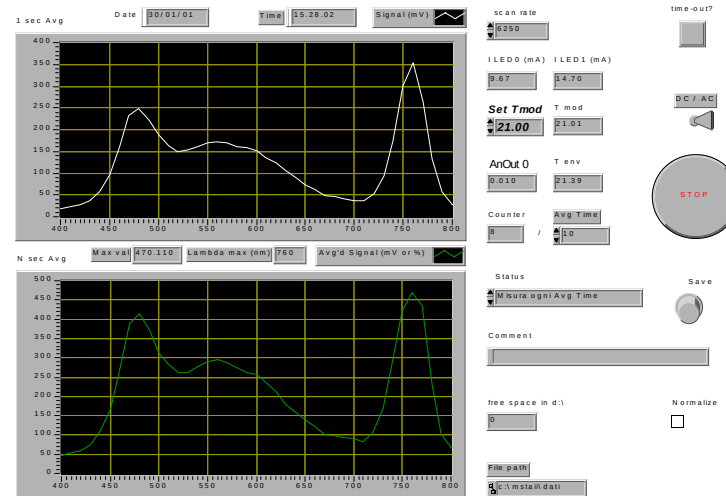


Spectrophotometer for absorption and reflection spectroscopy 6-LED source



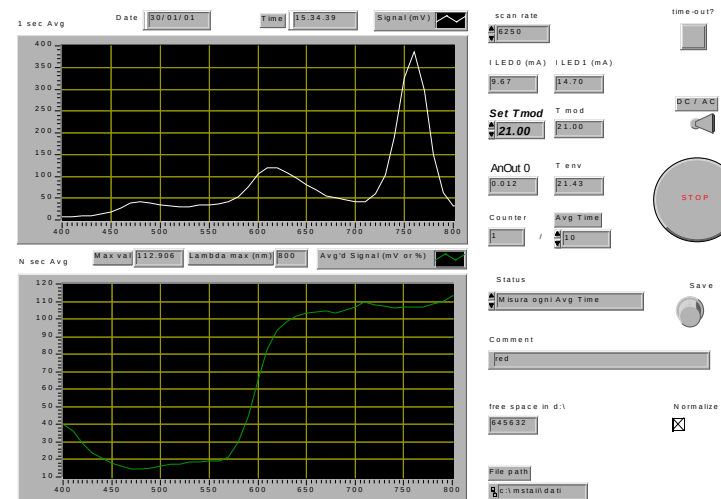
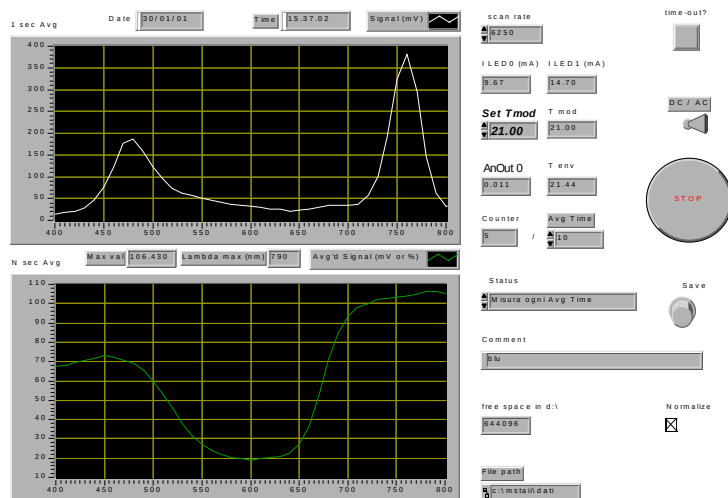
Laptop interface

DAQ1200 card + Labview software



For instructions select Show Vi info from the Windows menu.

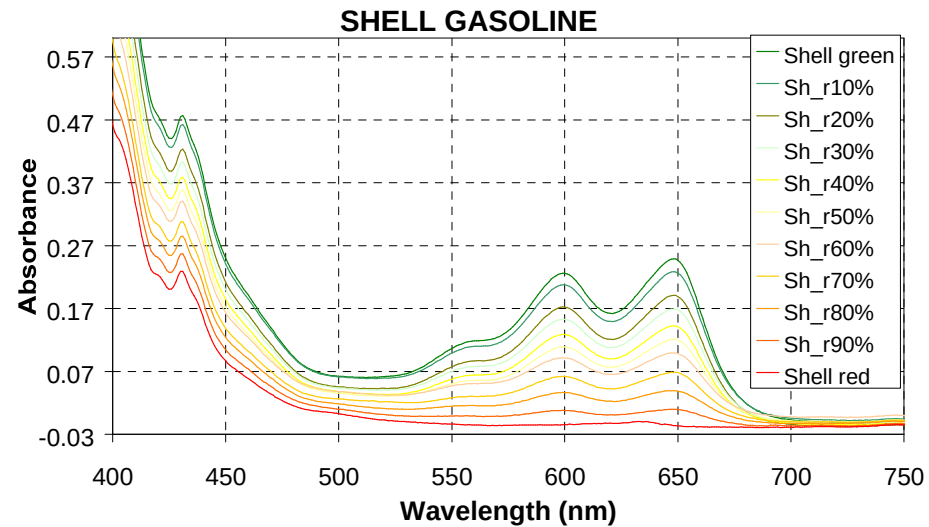
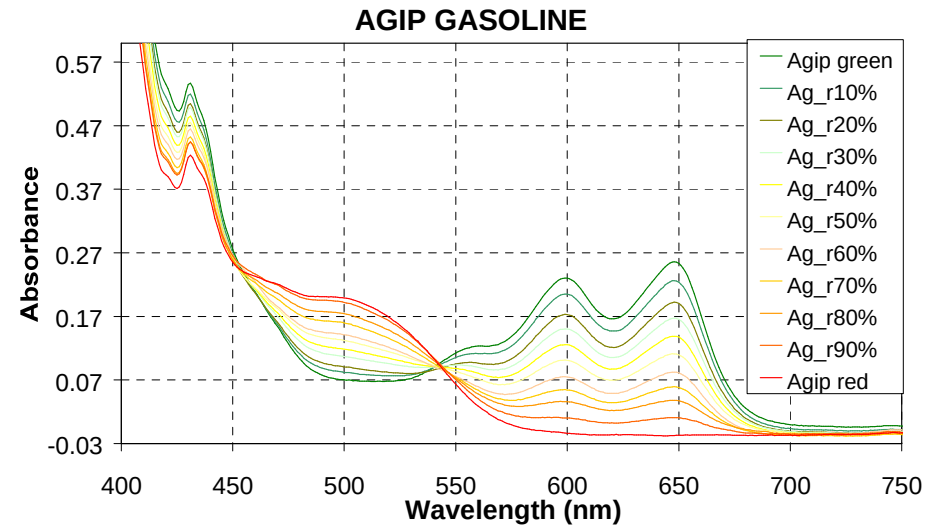
For instructions select Show Vi info from the Windows menu.



Colorimetry:

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Recognition of gasoline blending transition in a pipeline



Application of absorption spectroscopy

Water color monitoring in a recycling plant

- The water recycling process at the IDRA plant in Prato makes use of reservoirs for water storage after each recycling step. At present, water samples from reservoirs are taken and analysed in the laboratory, so as to monitor the operating efficiency of the recycling process.
- A fiber optic spectrophotometer was used to perform online water color measurements, proving the feasibility of online monitoring by exploiting fiber optic technology.

On-line absorption spectroscopy: water color monitoring in a recycling plant

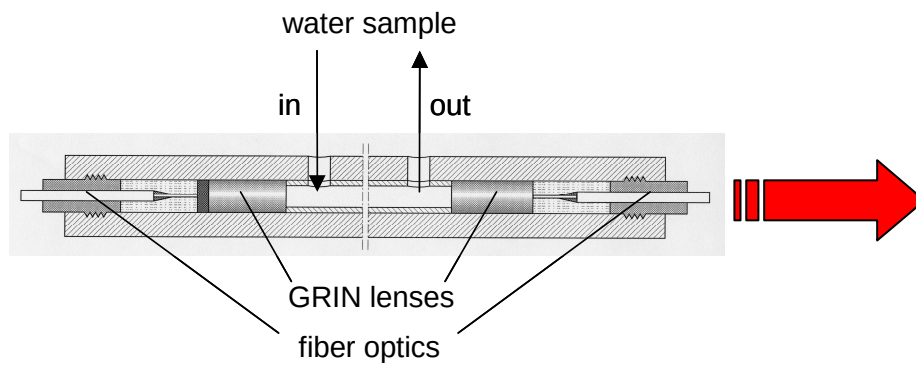
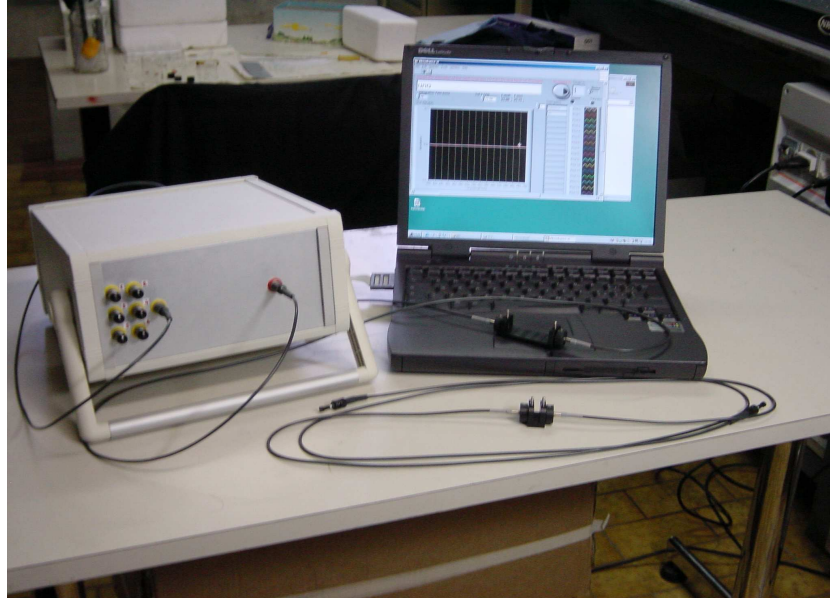


On-line absorption spectroscopy: water color monitoring in a recycling plant



Application of absorption spectroscopy

Water color monitoring in a recycling plant

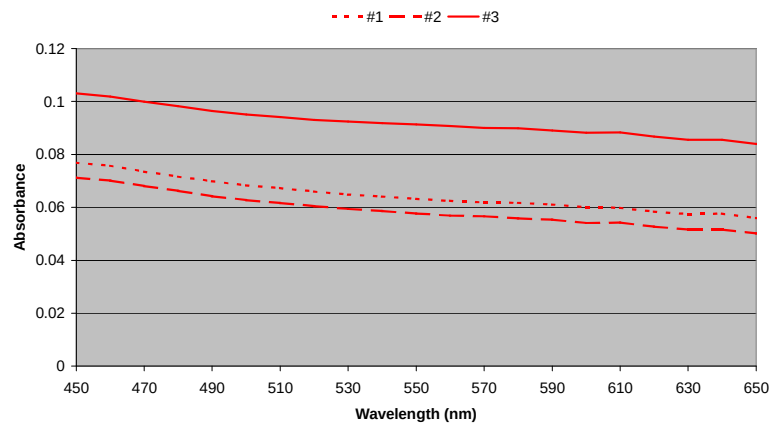


Application of absorption spectroscopy

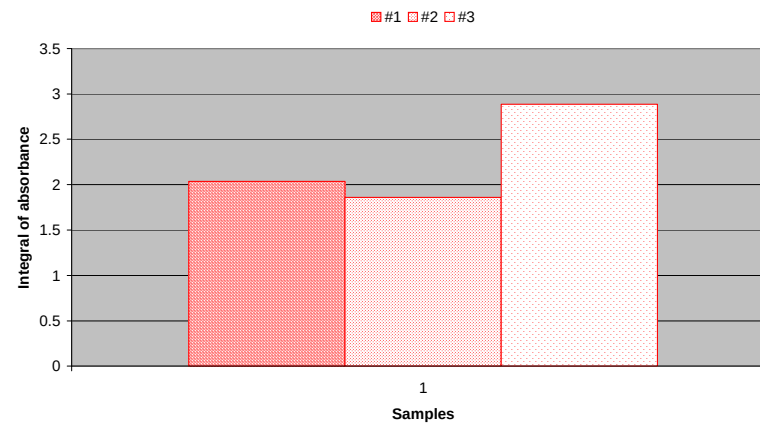
Water color monitoring in a recycling plant



09/10/2003 - around 12:00 - online

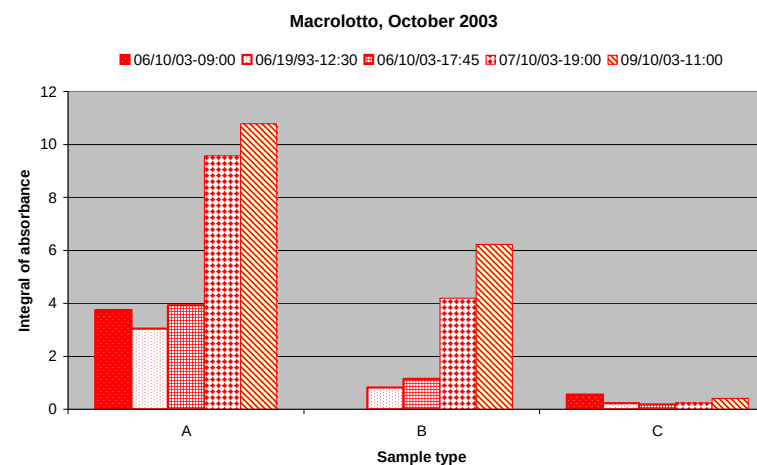
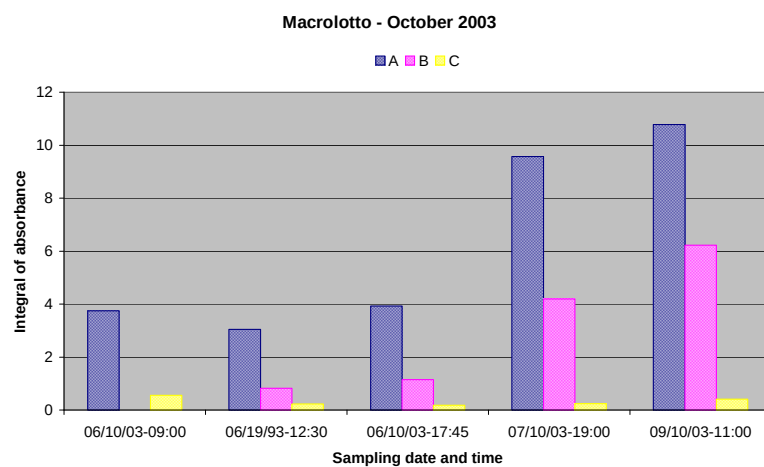


Macrolotto - 09/10/2003 - online



Application of absorption spectroscopy

Water color monitoring in a recycling plant



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Application of reflection spectroscopy: equivalent-light dosimetry inside museums

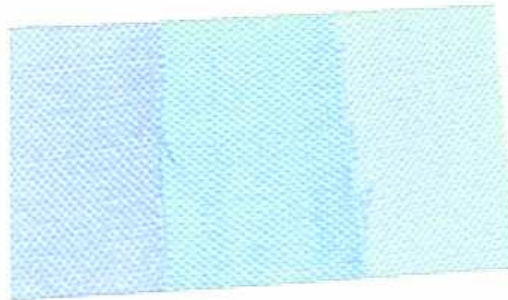
- All organic materials such as papers, textiles, resins and leathers can be damaged by excessive or unsuitable lighting, because of the light-induced photo-oxidation
- Photo-oxidation is a cumulative and irreversible effect; consequently, artistic material remains permanently damaged by the wrong lighting
- Lighting is not the only cause of color fading. The presence of pollutants and unsuitable temperature and humidity conditions can amplify and accelerate photo-alteration processes

Firenze, Italy: the Uffizi Gallery

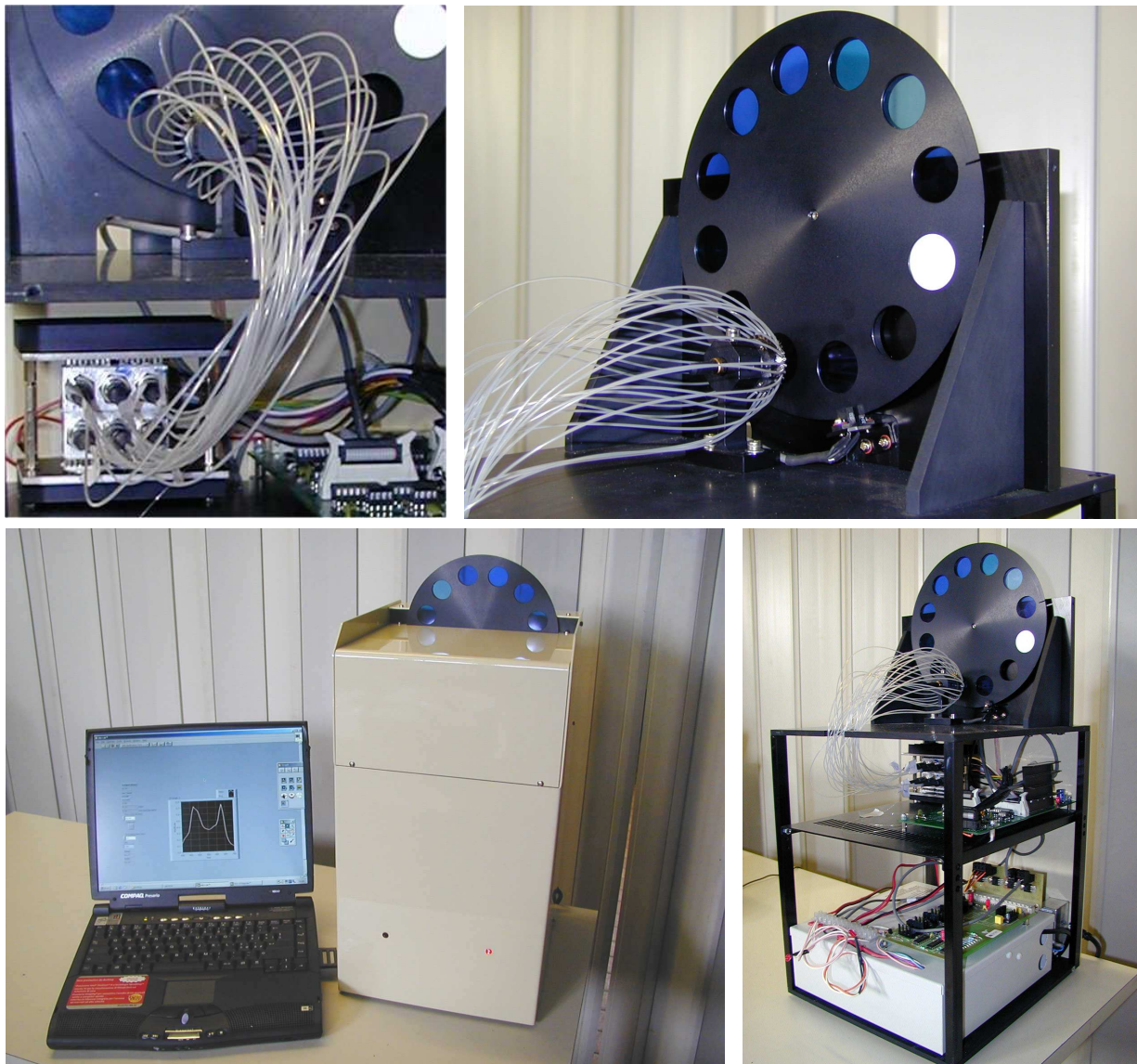


Equivalent-light dosimetry inside museums

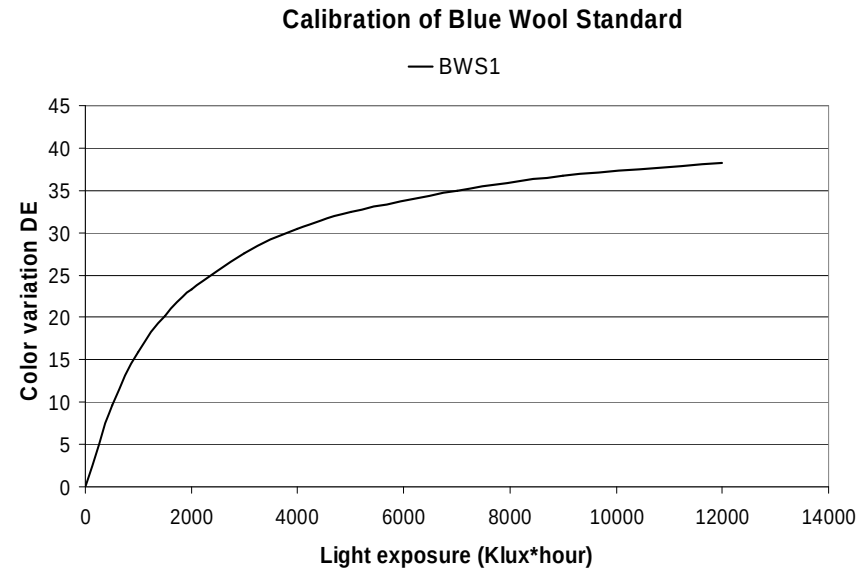
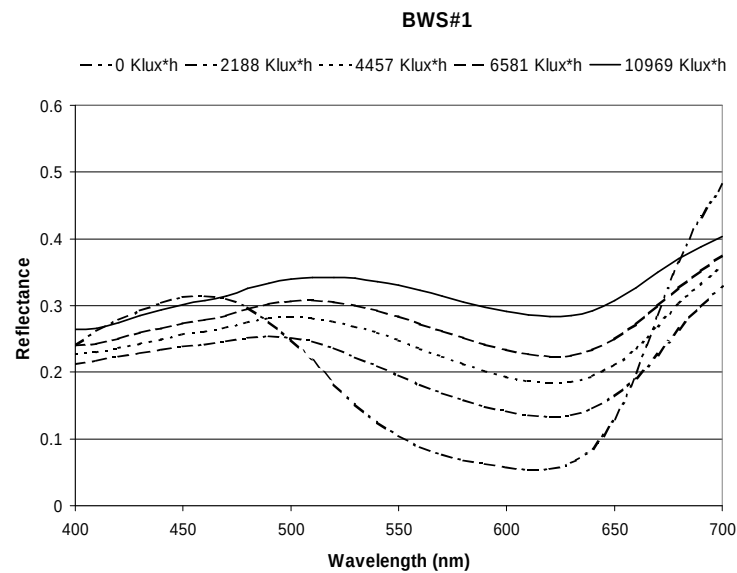
- **‘Equivalent-light dose’:** cumulative impact of light and environmental conditions
- Blue Wool Standard
 - Suitable for light-equivalent dosimetry since exposure to environmental conditions induces a progressive color fading
 - Two series of ‘Blue-Wool Standard’ samples can be used
 - one series can be exposed to environmental conditions
 - the other can be kept un-exposed
 - comparative colorimetry between exposed and un-exposed samples can be achieved by reflection spectroscopy, thus measuring the ‘equivalent-light’ dose



Instrumentation for indoor equivalent-light dosimetry inside museums



Equivalent-light dosimetry inside museums: calibration



Equivalent-light dosimetry inside museums: preliminary tests

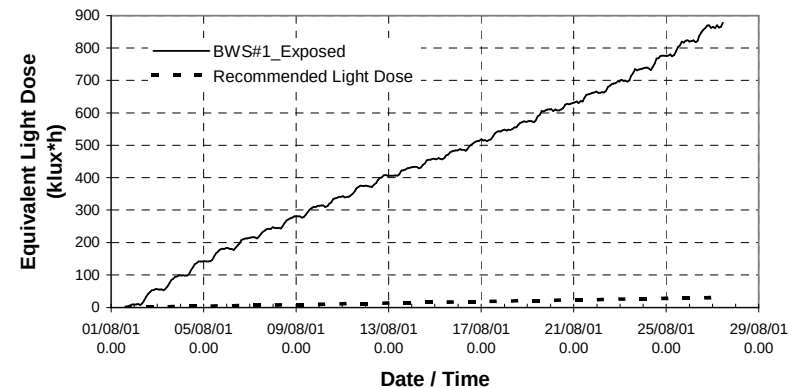
CNR-IFAC laboratory
Summer 2001



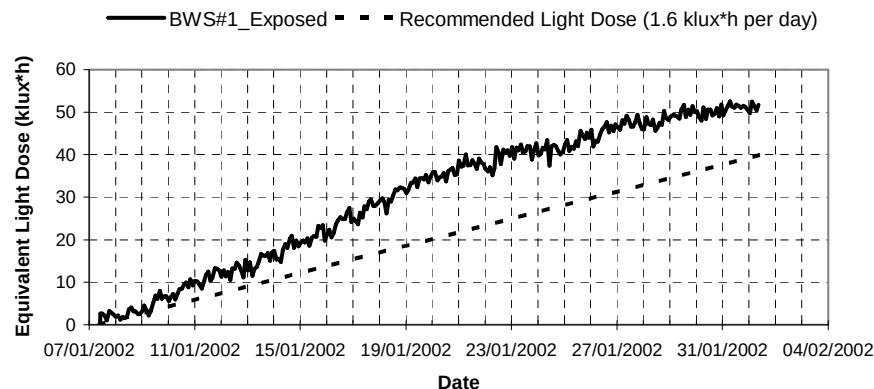
Uffizi Gallery
room of Geographic Maps
Winter 2002



Florence, Italy - Exposure from 1 to 27 August 2001
South-East Exposure - Strong illumination in the morning



Uffizi Gallery, Florence, Italy - Room of 'Geographic Maps'
south-east exposure - from 7 to 31 January 2002



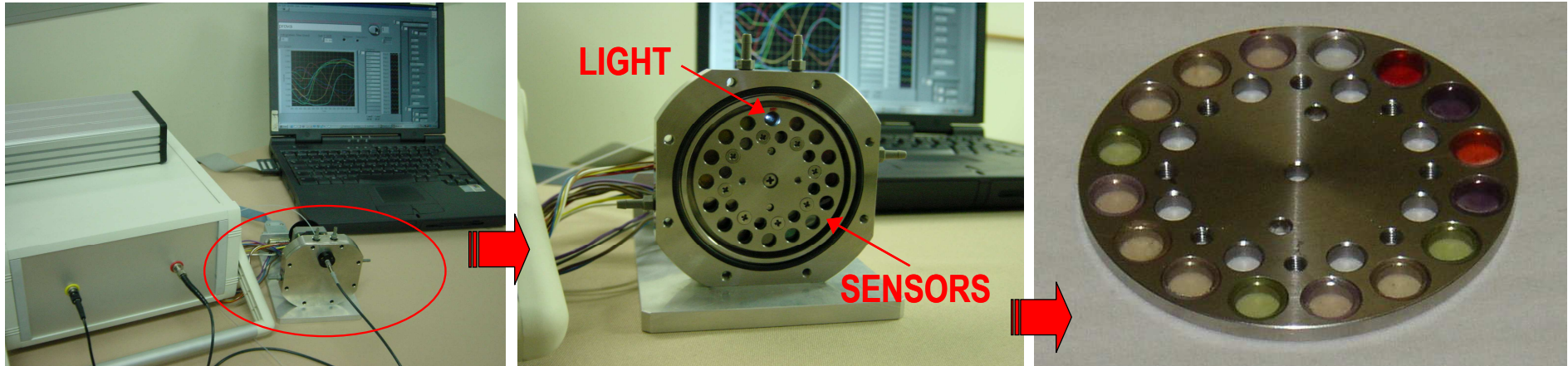
AGM - ICTP - Feb'07



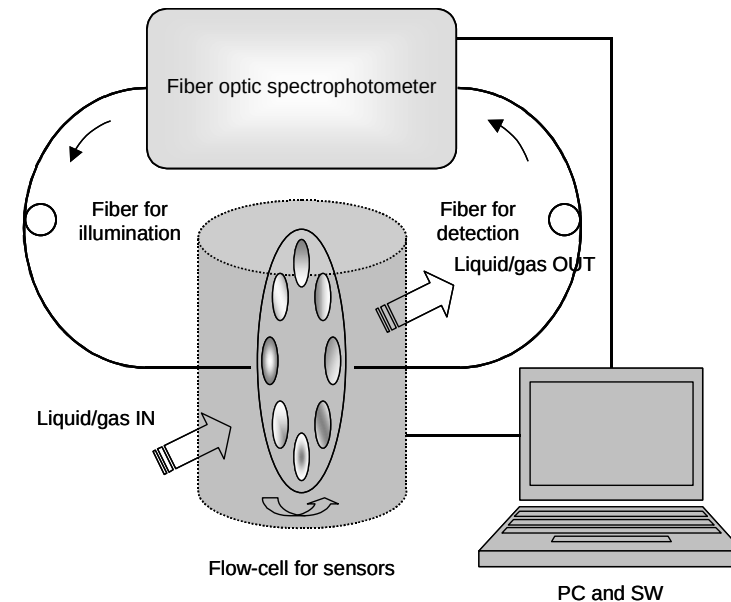
Equivalent-light dosimetry at Palazzo Vecchio, Firenze, Italy



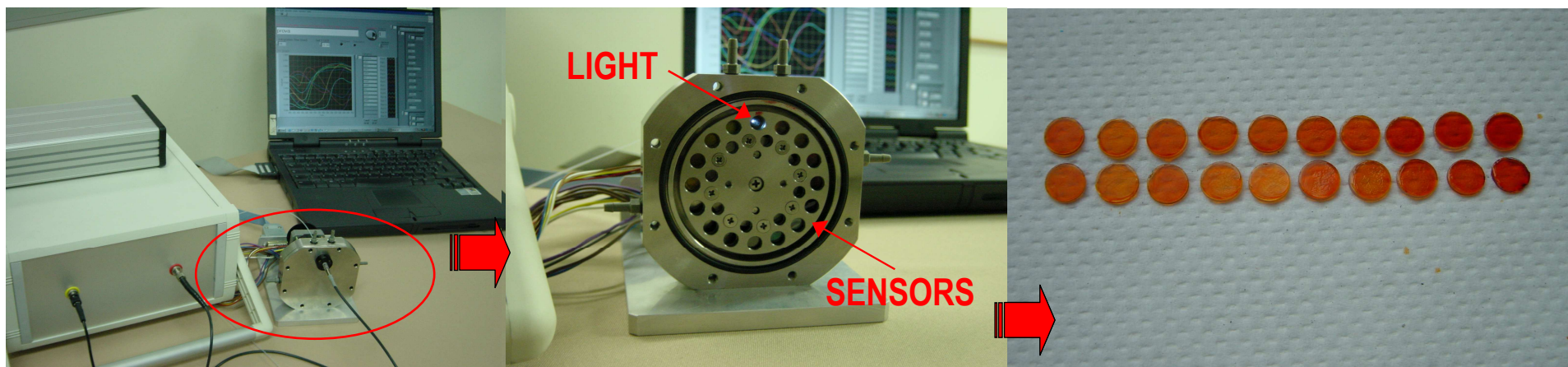
Fiber optic multimeter for spectral interrogation of an opto-chemical sensor array



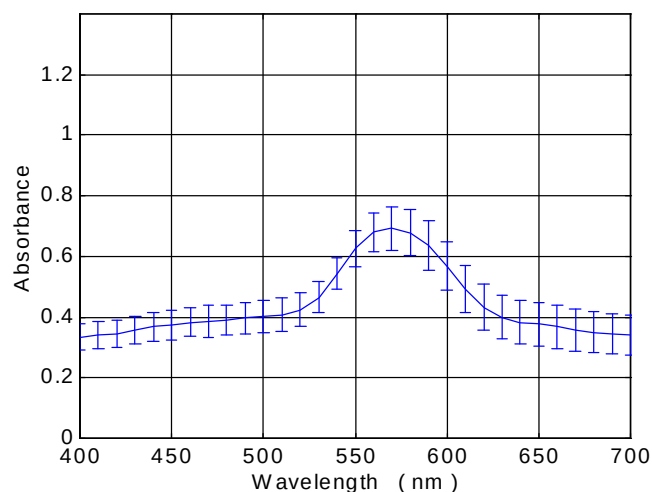
A single electro-optic unit capable of interrogating a multiplicity of sensors
evident added value supplied by
identical hardware and software at
the service of a sensor array



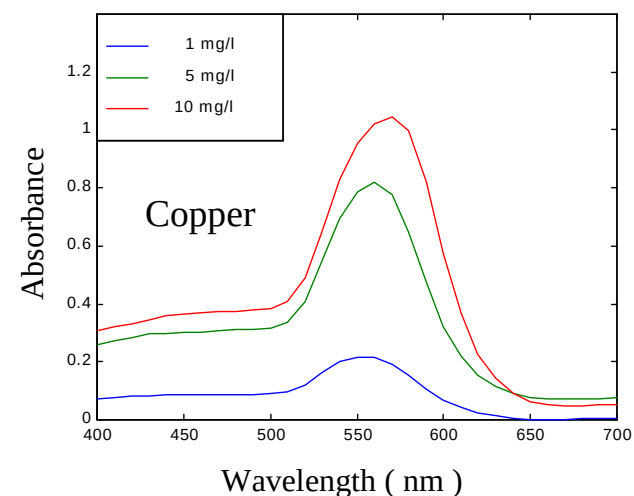
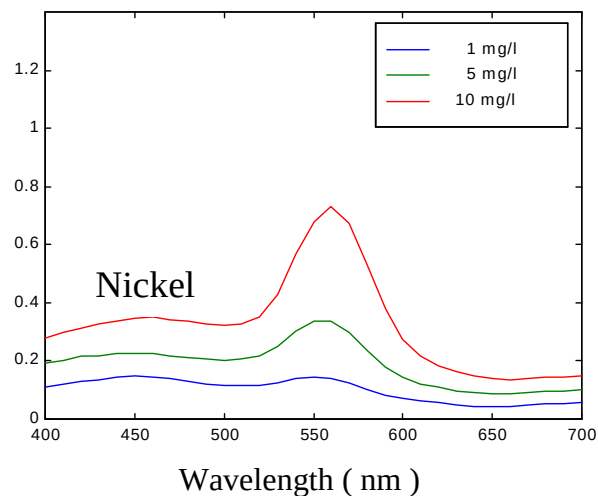
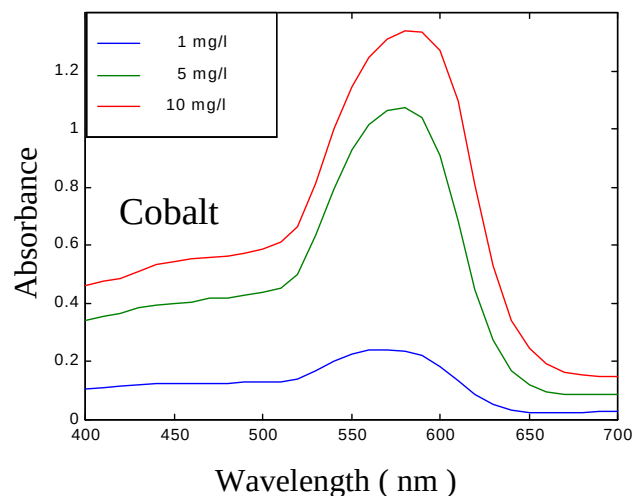
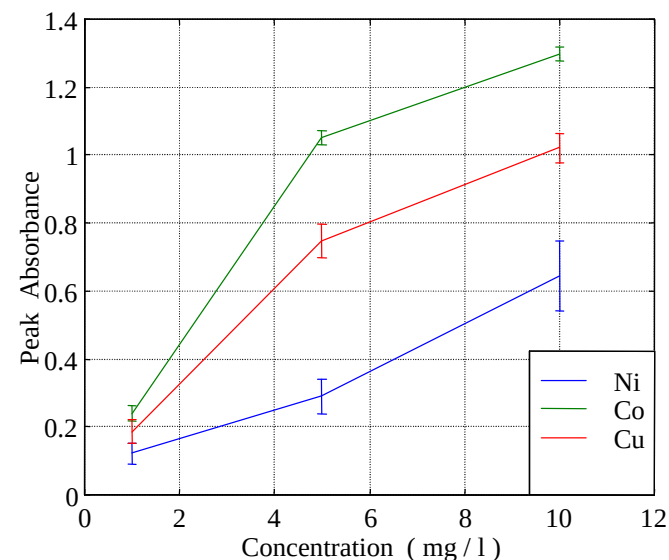
Fiber optic multimeter for spectral interrogation of an opto-chemical sensor array: validation of metal ion dosimeters



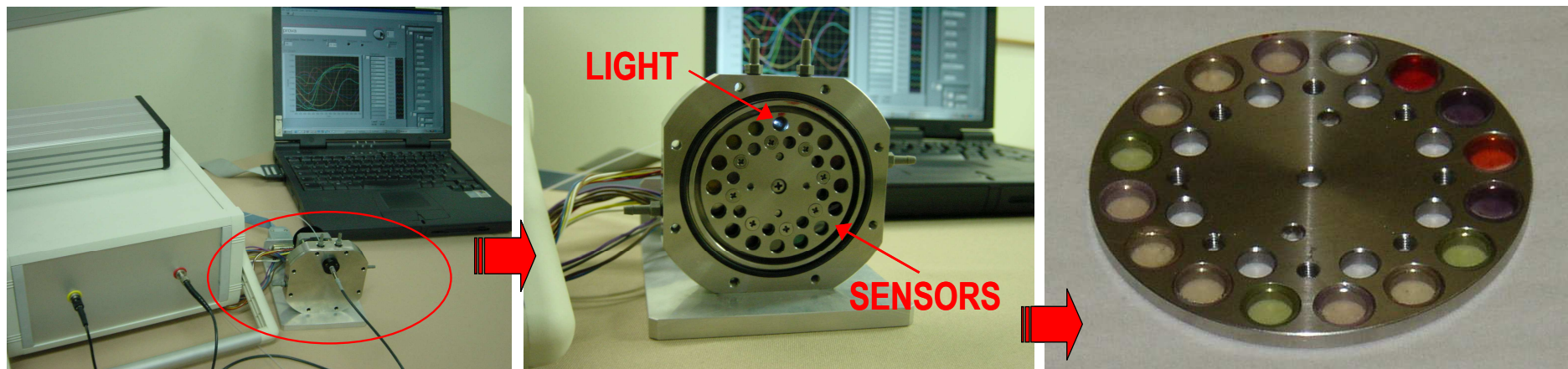
SENSORS: azo dye species, 2-(5-Bromo-2-pyridylazo)-5-diethylaminophenol (BrPADAP), together with an ionophore and plasticizer, immobilized in a PVC film and deposited onto a glass disc



Fiber optic multimeter for spectral interrogation of an opto-chemical sensor array: validation of metal ion dosimeters



Fiber optic multimeter for spectral interrogation of an opto-chemical sensor array: artificial olfactory perception of toxic gases and vapors



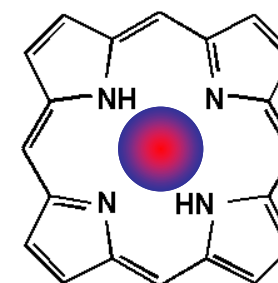
SENSORS: blend of metallo-porphyrins and metallo-ftalocyanins

Structure name

Zinc-5,10,15,20-tetrakis (4-Heptiloxyphehyl)Porphyrin
Cobalt-5,10,15,20-tetrakis (4-Hexadodecyloxyphenyl)Porphyrin
Manganese-5,10,15,20-tetrakis (4-Hexadodecyloxyphenyl)Porphyrin
Copper--5,10,15,20-tetrakis (4-Dodecyloxyphenyl)Porphyrin
Zinc-Tetraphenyl-Porphyrin-PVC
Copper-TetraphenylPorphyrinPVC

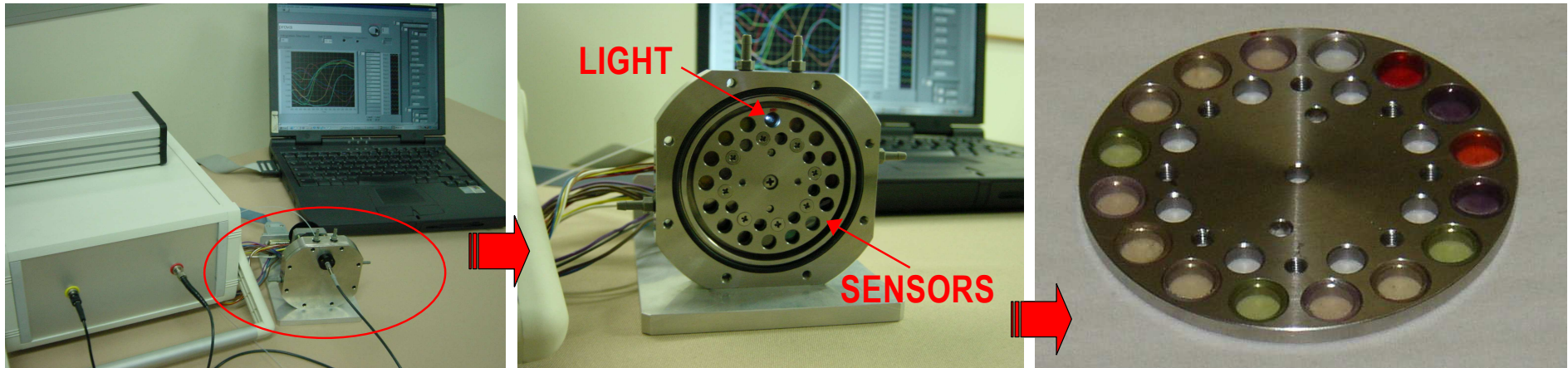
Acronym

ZnTHEPP (3)
CoTEXPP (3)
MnTEXPP (3)
CuTDPP (2)
ZnTPPPVC (2)
CuTPPPVC (2)



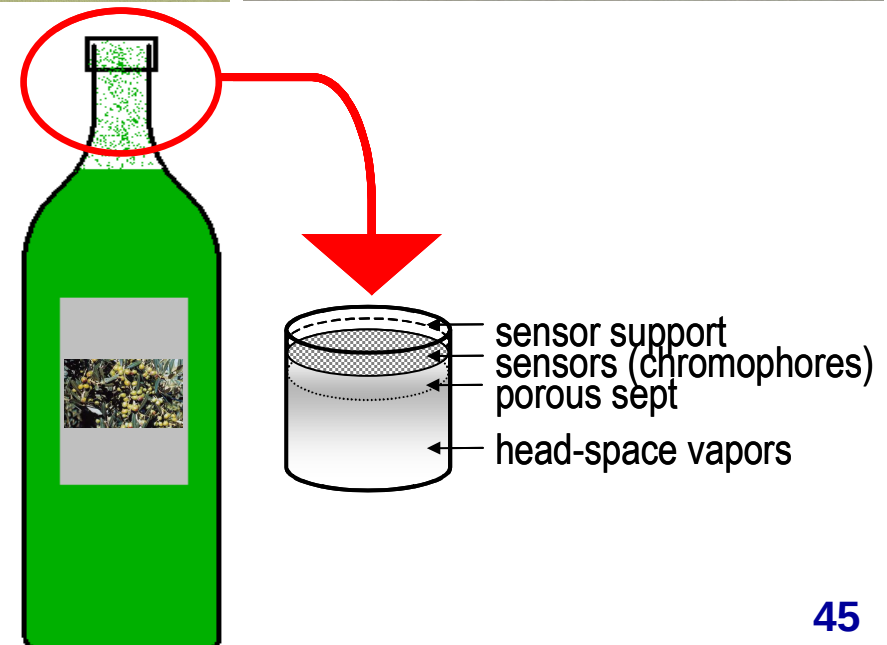
Different metals coordinated
to the macrocycle

Fiber optic multimeter for spectral interrogation of an opto-chemical sensor array: artificial olfactory perception of olive oil rancidity

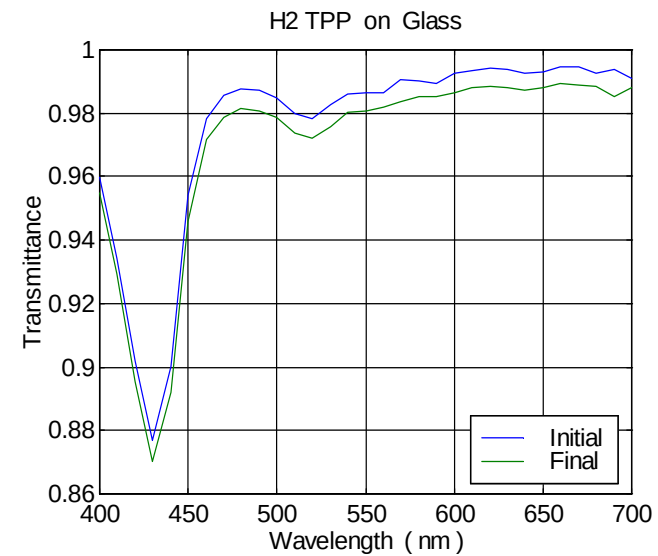
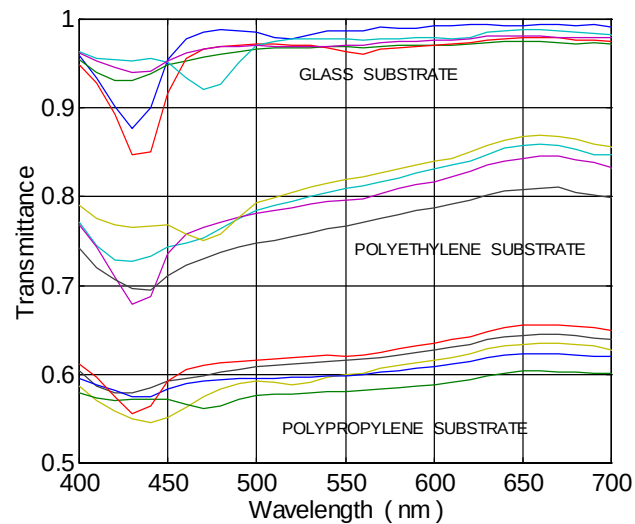
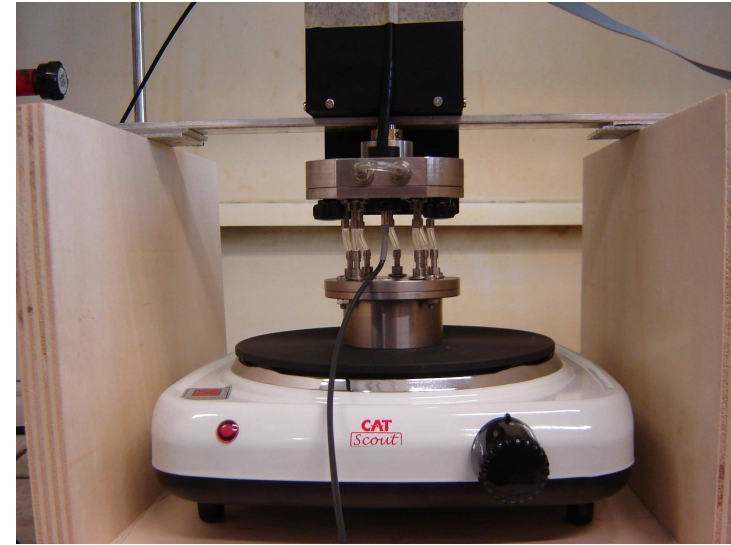
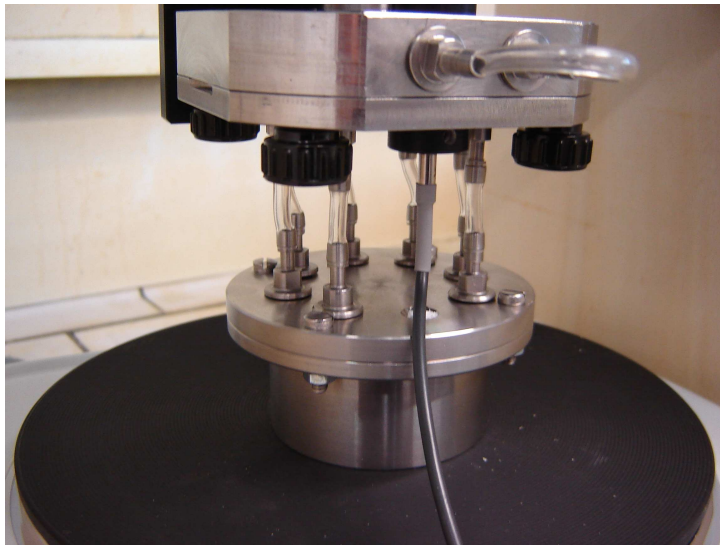


SENSORS: metallo-porphyrins

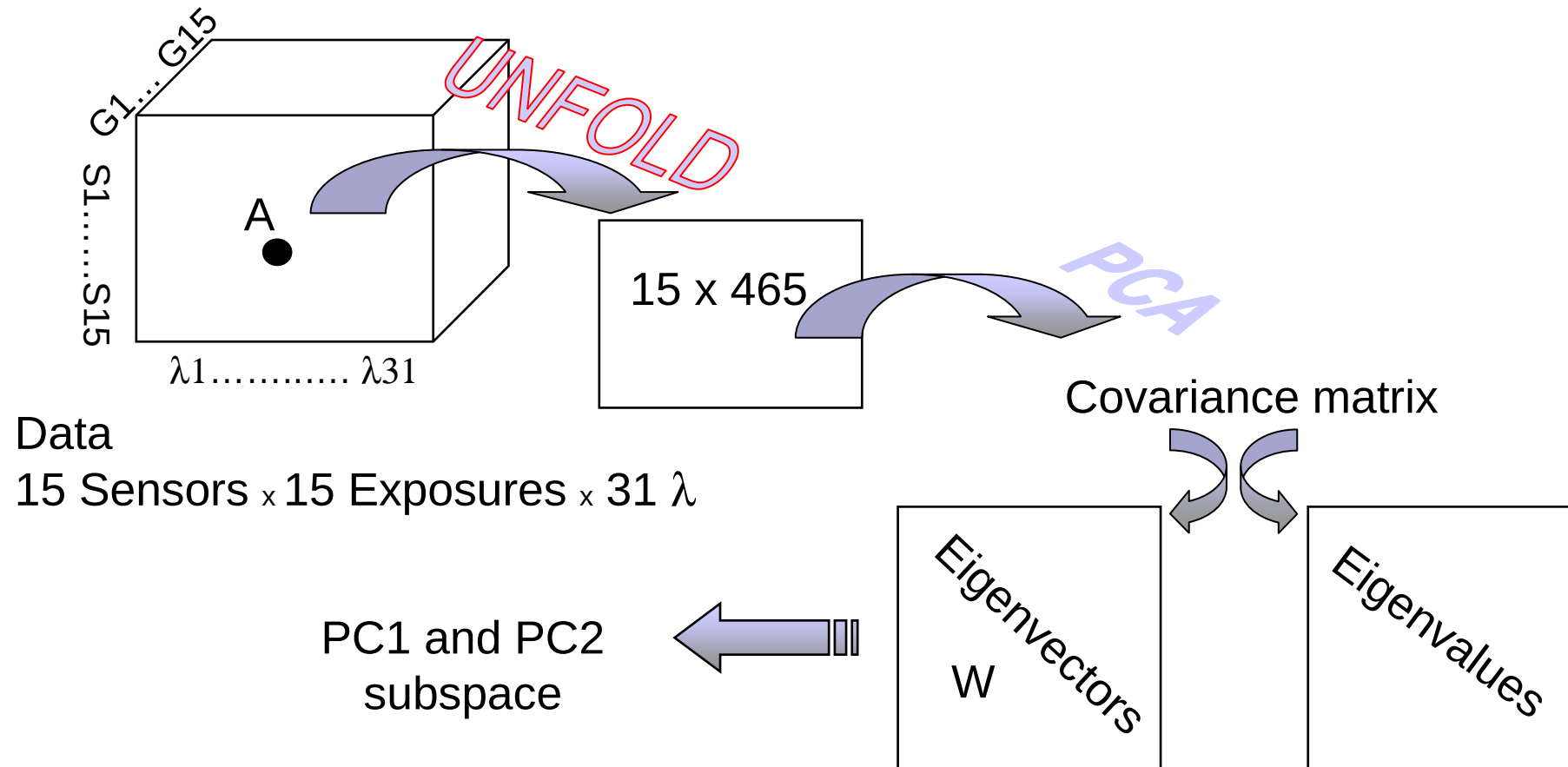
Smart cap for non-destructive
testing of oil quality (rancidity) by
analyzing the vapor in the bottle
head-space during the shelf life



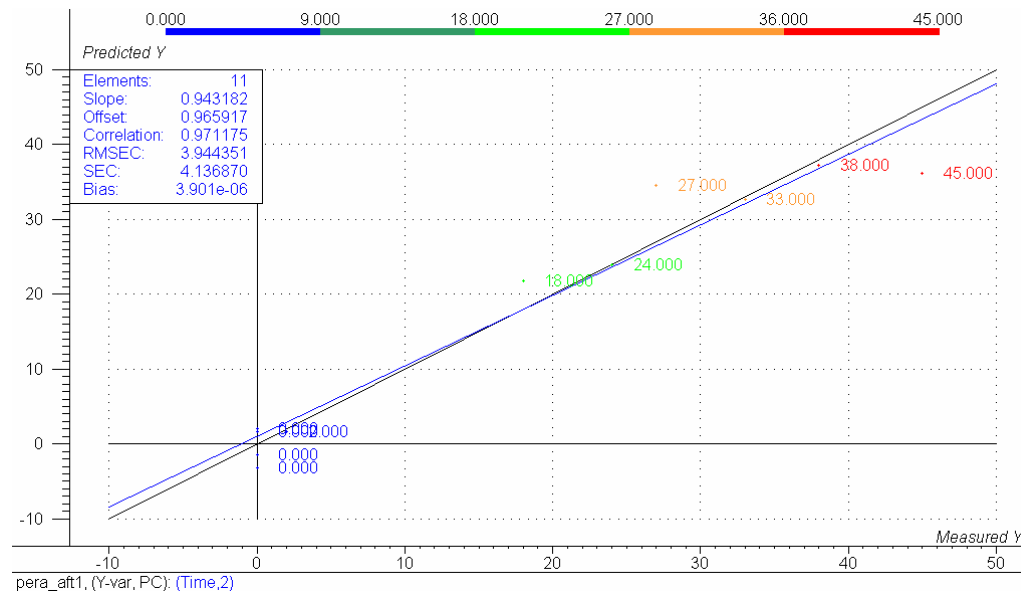
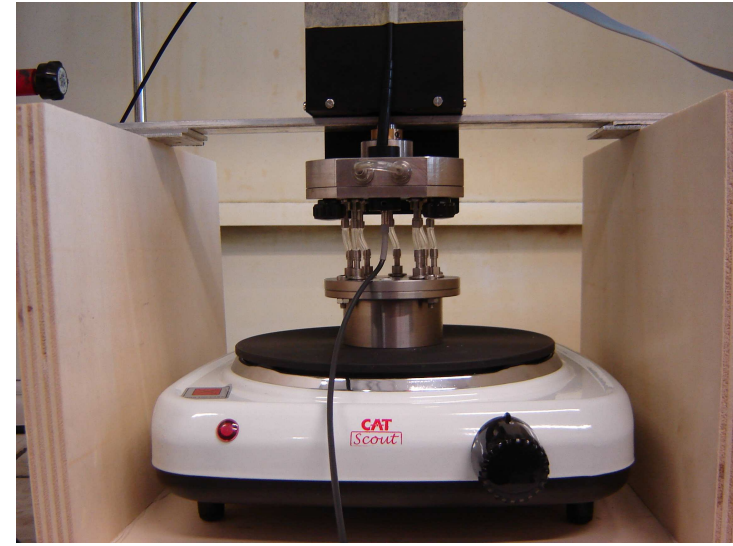
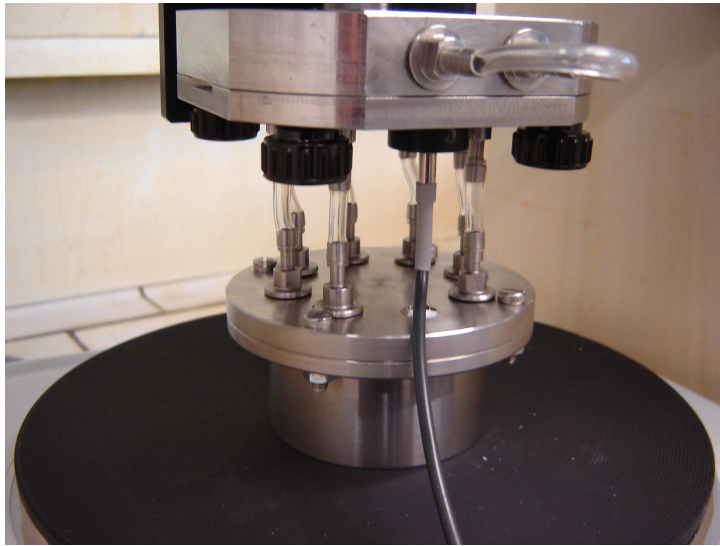
Simulation of a smart cap for artificial olfactory perception of olive oil rancidity



PARAFAC and PCA signal processing



Simulation of a smart cap for artificial olfactory perception of olive oil rancidity



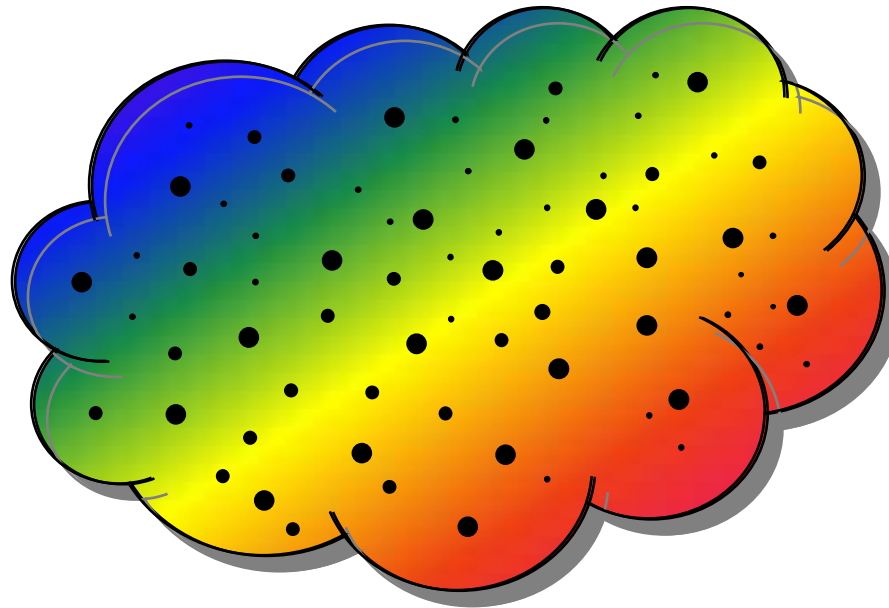
Outline

- **Optical fiber sensors: basics**
- **Colorimetry**
 - Absorption and reflection spectroscopy
 - Fiber optic spectrophotometers for general purpose applications – Direct / chemically-mediated
- **Scattered colorimetry**
 - Multi- λ and multi- α absorption spectroscopy
- **Hyperspectral colorimetry**
- **Fluorescence spectroscopy**

..... Liquids

- ✓ Moderately colored
- ✓ Moderately turbid
- ✓ Appearance
- ✓ Mapping
- ✓ Comparing and classifying

The appearance of a liquid: what a liquid looks like



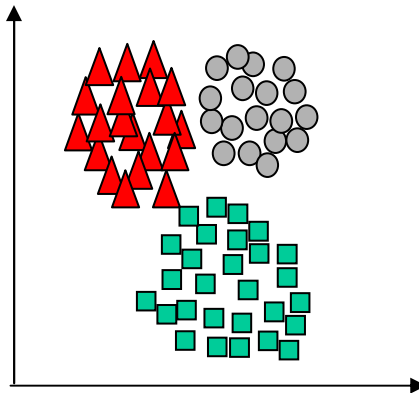
color + turbidity = visual perception

- ✓ quality
- ✓ mixtures: components

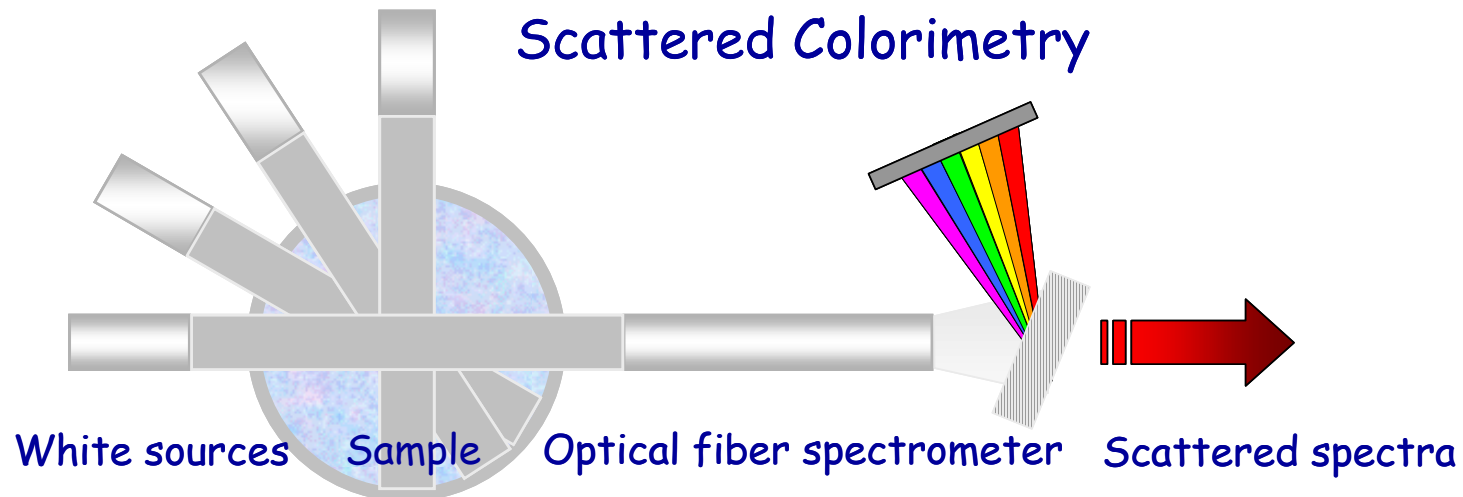
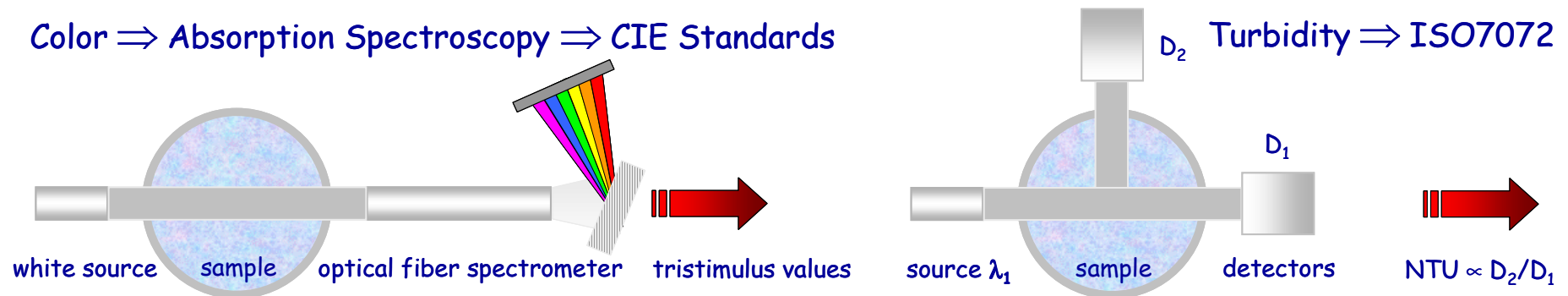
Scattered colorimetry + multivariate data processing

- Integrated and global measurement of the color and turbidity = appearance of liquids
 - Appearance is determined objectively by means of a measuring instrument
-
- Liquid mapping = digital 'footprint'

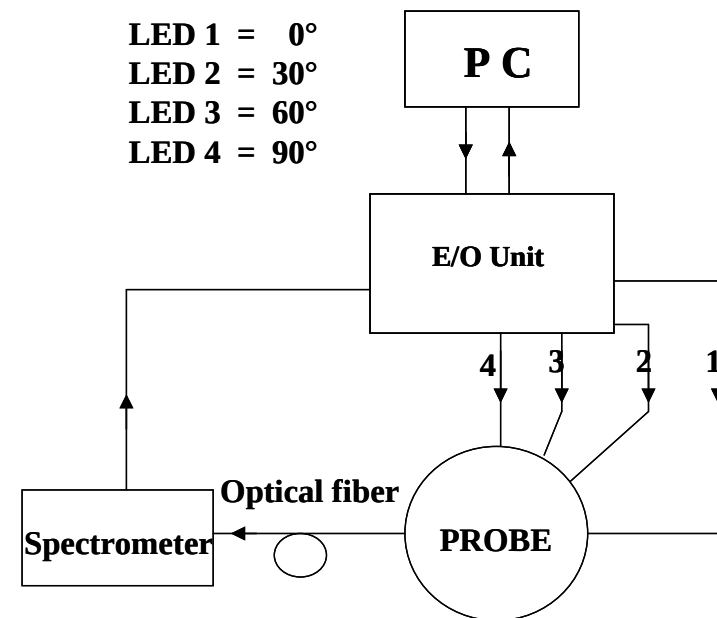
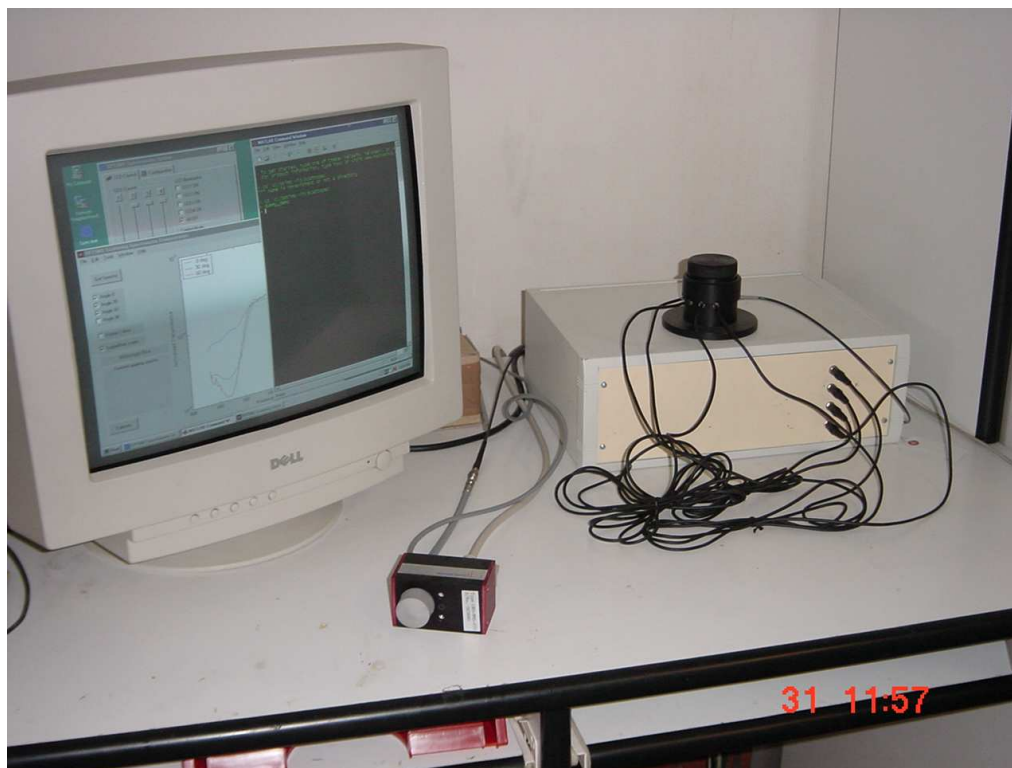
- Clustering



Scattered colorimetry multi- λ and multi- α absorption spectroscopy in the visible spectral range



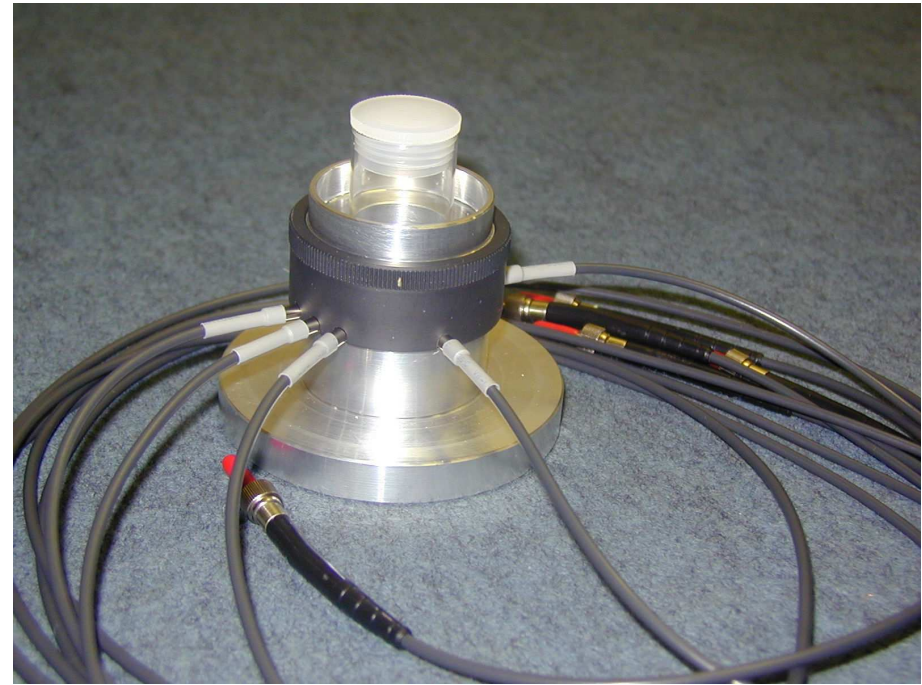
Instrumentation for scattered colorimetry



Probes for scattered colorimetry



without optical fibers



with optical fibers

Scattered colorimetry: analysis of port wine



OPTIMO
Optical Technology for Intelligent Monitoring Online



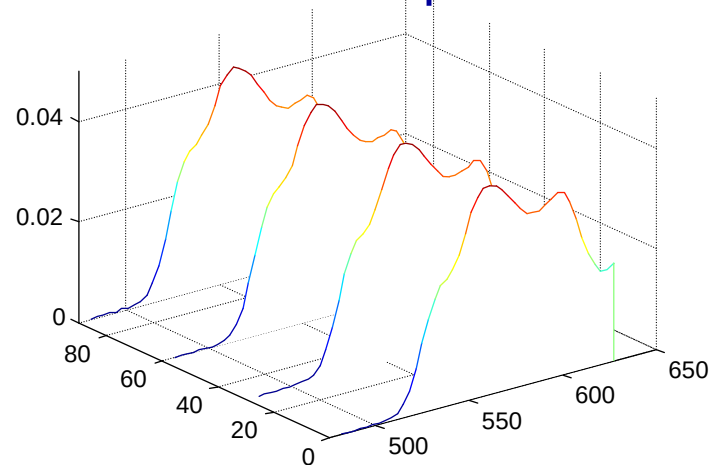
Courtesy of Sandeman, Porto, Portugal

AGM - ICTP - Feb'07



Scattered colorimetry: the spectral data

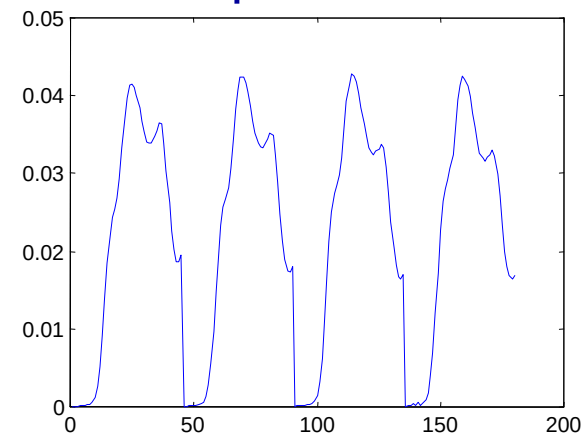
Measured Spectra



46 wavelengths x 4 angles

Unfolding

Sample Pattern

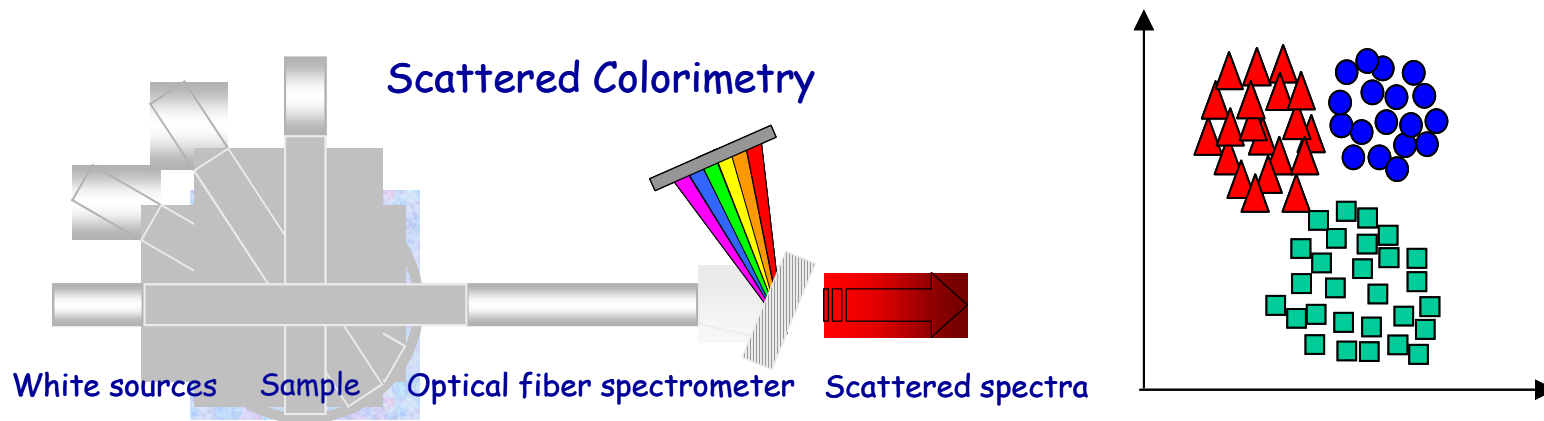


..... 184 variables
dimensionality reduction is needed



MULTIVARIATE DATA PROCESSING
Principal Component Analysis
Linear Discriminant Analysis

Scattered colorimetry + multivariate data processing



- Mapping
- Clustering
- Comparing

Successful applications

➤ Food

- Mapping of extra virgin olive oils according to geographic area of origin
- Mapping of frying oils according to degree of degradation and substance being fried
- Mapping of beers according to types

➤ Non-food

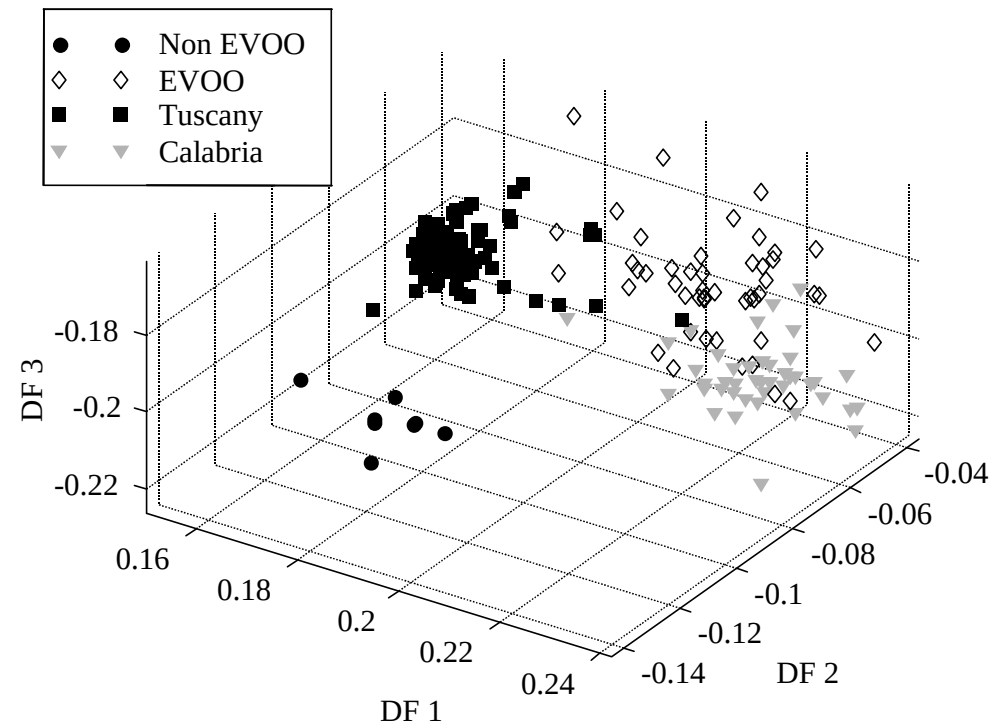
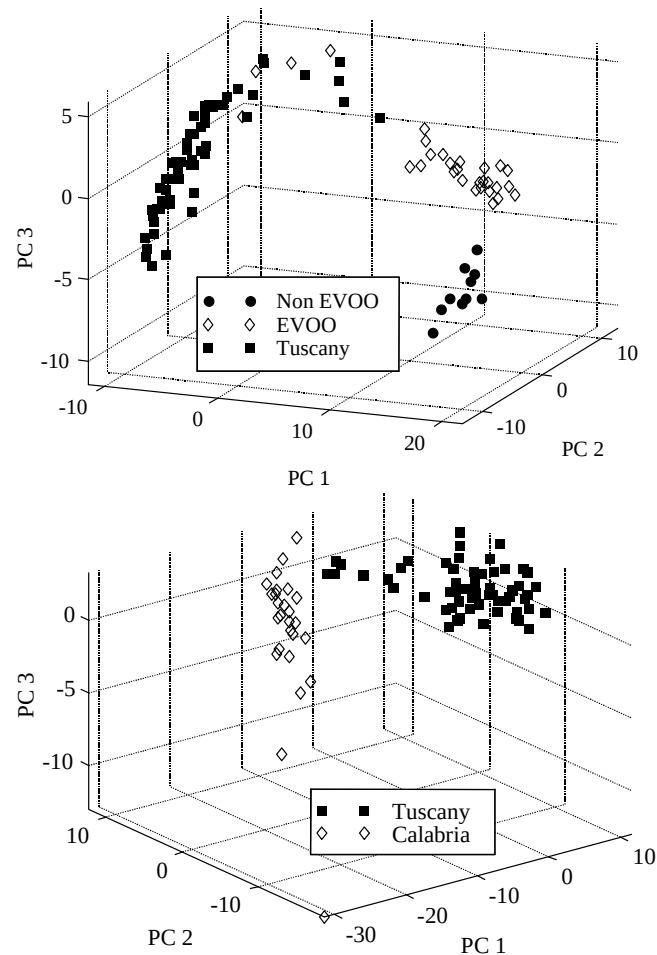
- Mapping of lubricant oils according to the degree of degradation

Mapping of extra virgin olive oils according to geographic area of origin

- 115 Tuscan and 53 Calabrian extra virgin olive oils produced by traditional methods
- 68 oils (58 extra virgin and 10 non extra virgin) purchased from retailers



Mapping of extra virgin olive oils according to geographic area of origin



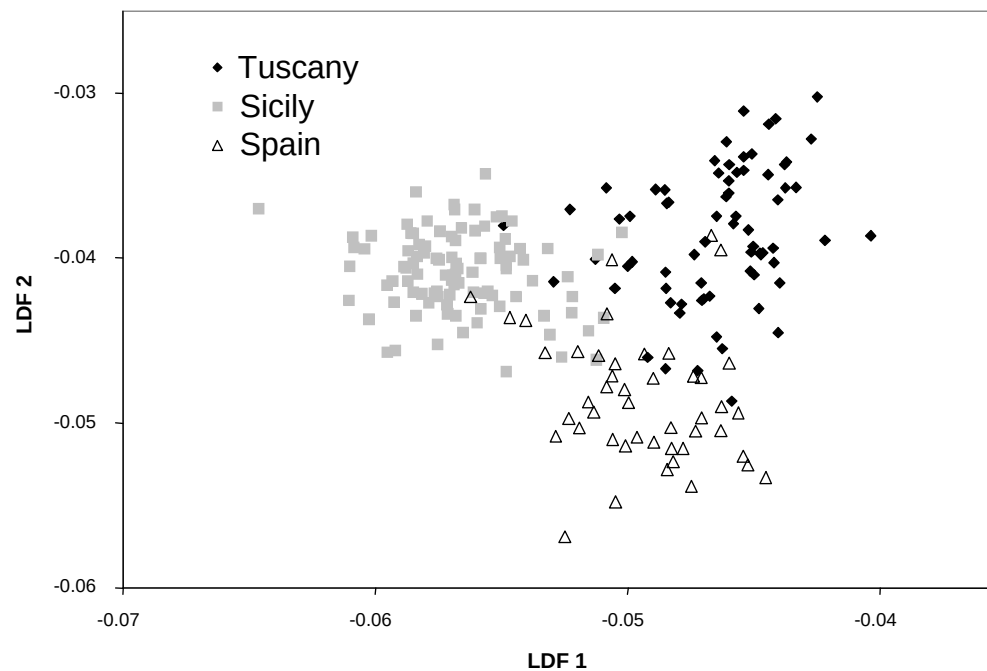
Mapping of extra virgin olive oils according to geographic area of origin

- 213 Italian oils (90 Tuscan and 123 Sicilian)
- 57 Spanish oils



- Because of the intrinsic differences due to different harvesting time (weather conditions, harvest times, etc.), a new map had to be created

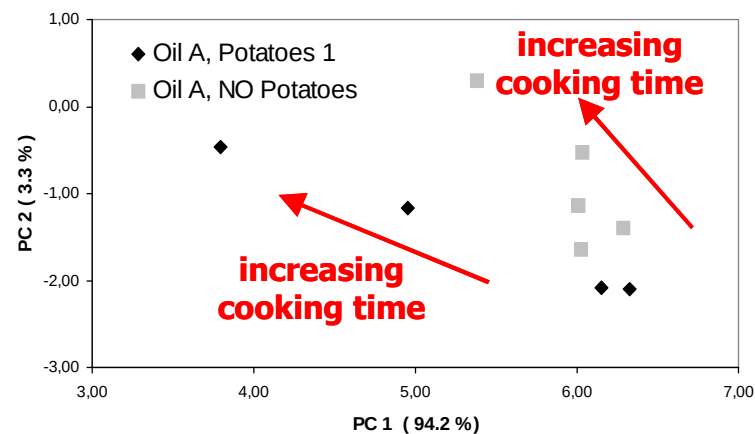
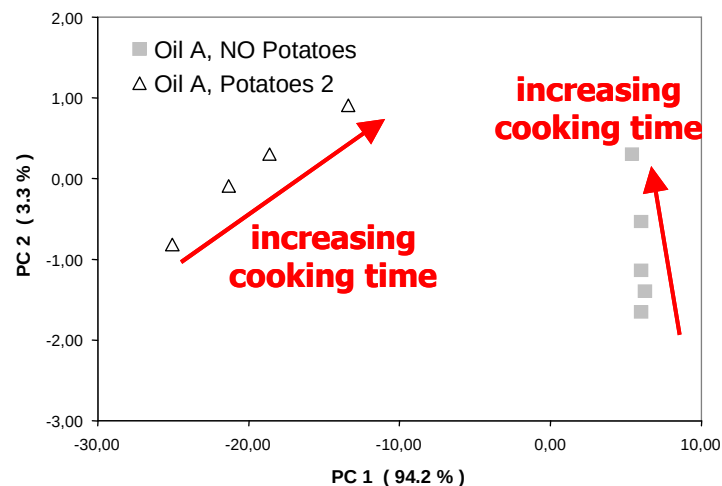
Mapping of extra virgin olive oils according to geographic area of origin



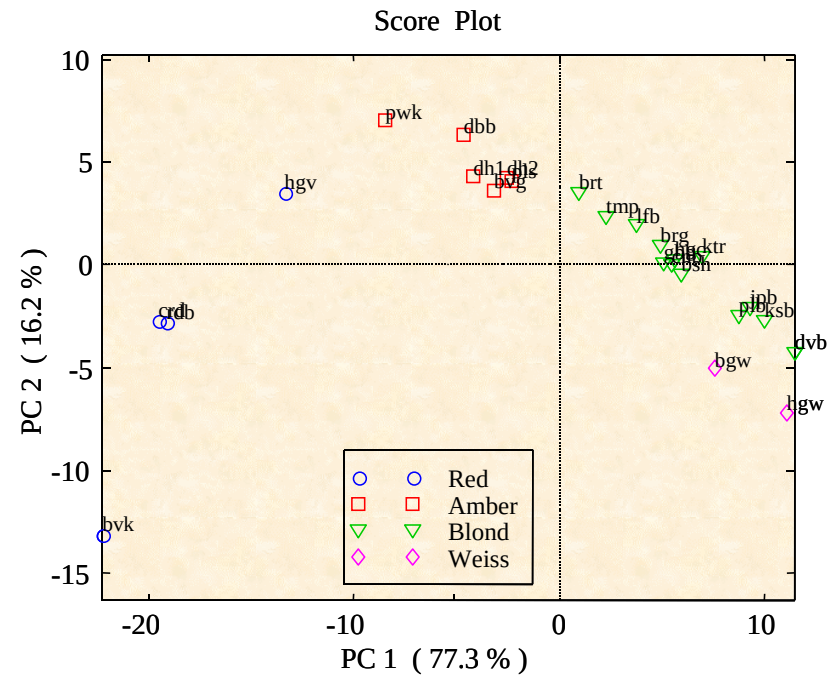
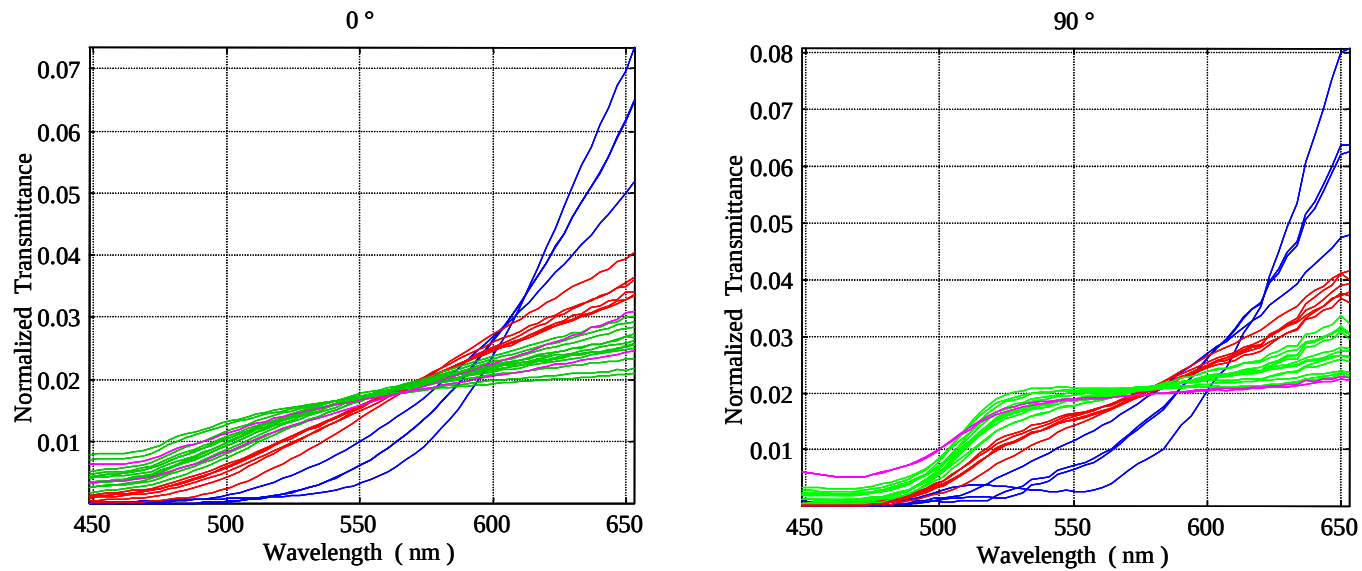
Mapping of frying oils according to degree of degradation and substance being fried

- Industrial fryers
- During frying, oil changes color, turning darker and more turbid, from the food being fried leaves residues in the oil
- Variation in color and turbidity are thus indicators of the oil's degree of degradation
 - Experiment: different types of potatoes for different cooking times ranging from 4 to 16 hours

Mapping of frying oils according to degree of degradation and substance being fried



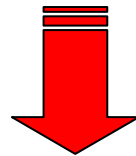
Mapping of beers according to types



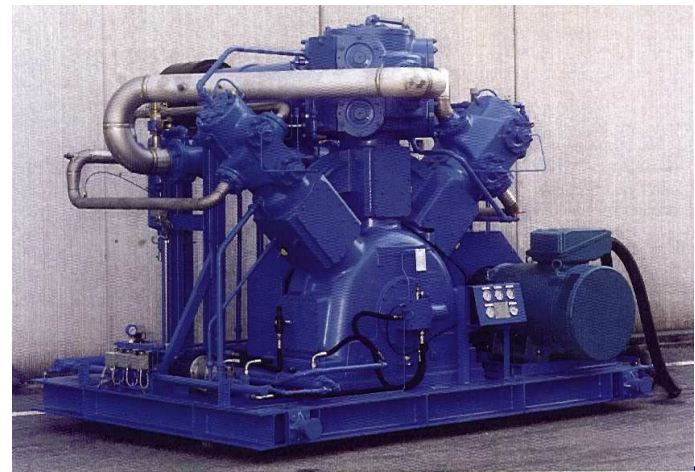
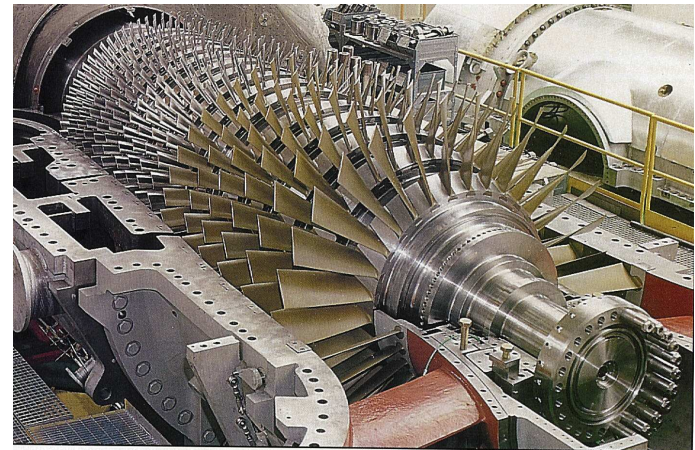
Lubricant oils: the machineries



high temperature
metals
particles
dusts



oil degradation



Metals in lubricant oil as indicators of wrong functioning



Regularly-programmed sampling and conventional laboratory analysis



- viscosity
- FTIR
- ferrography
- acidity

score



chemical-physical status:

- ✓ 1: good
- ✓ 2: acceptable
- ✓ 3: change suggested
- ✓ 4 and 5: change mandatory

On-line diagnosis of early wrong functioning

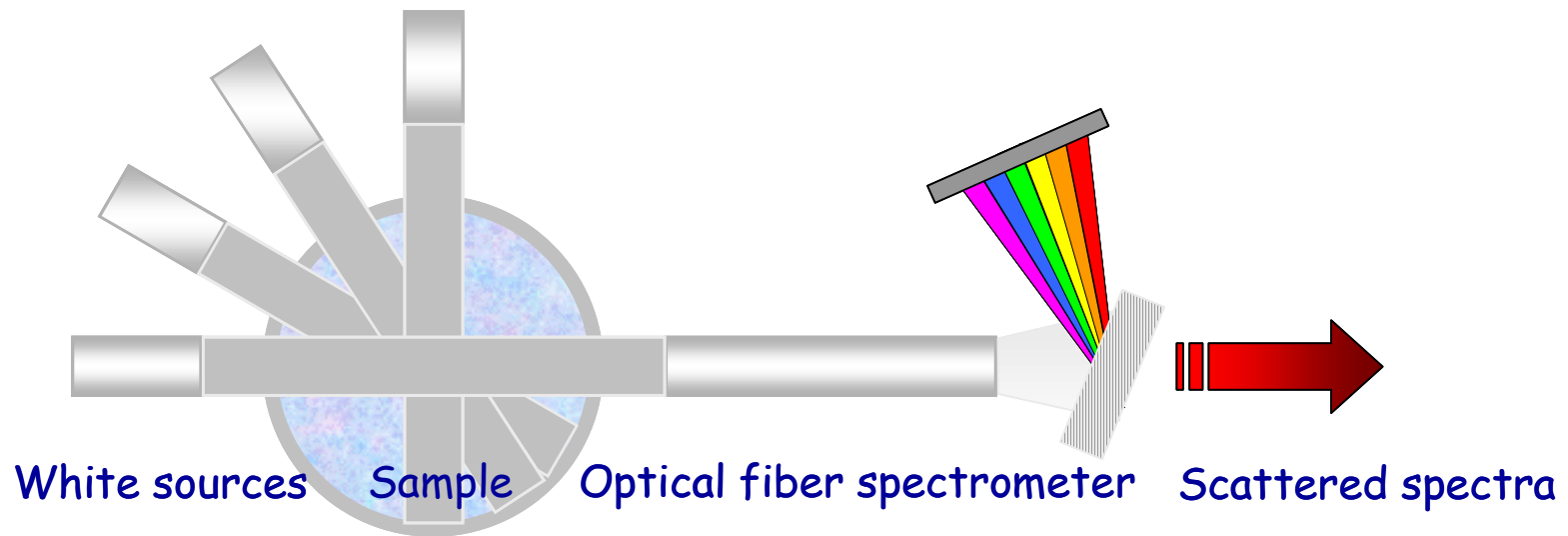
- Capable of distinguishing different chemical-physical status
- Low-cost system
- Immune to EMI

The lubricant oils



Degree of degradation = Different color and turbidity content

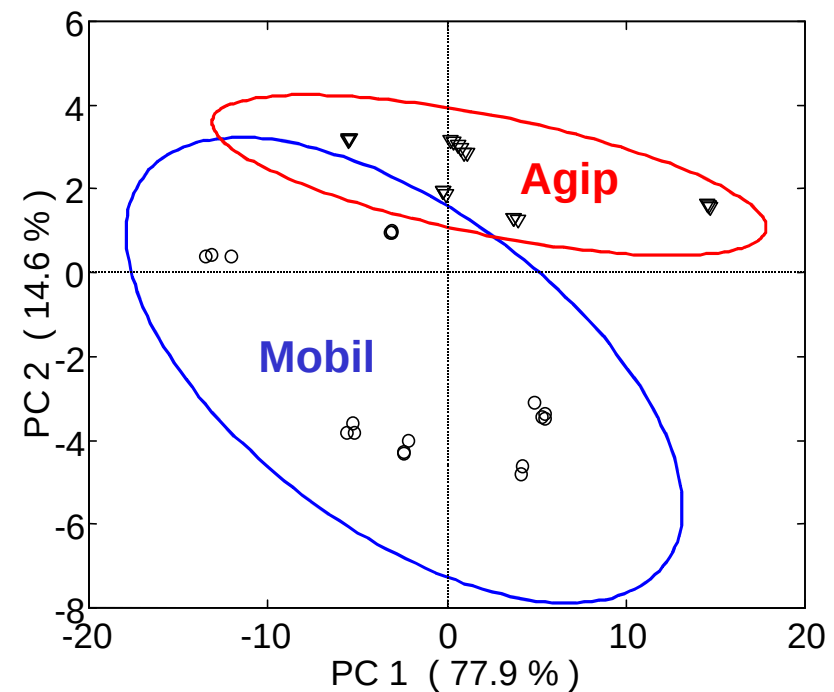
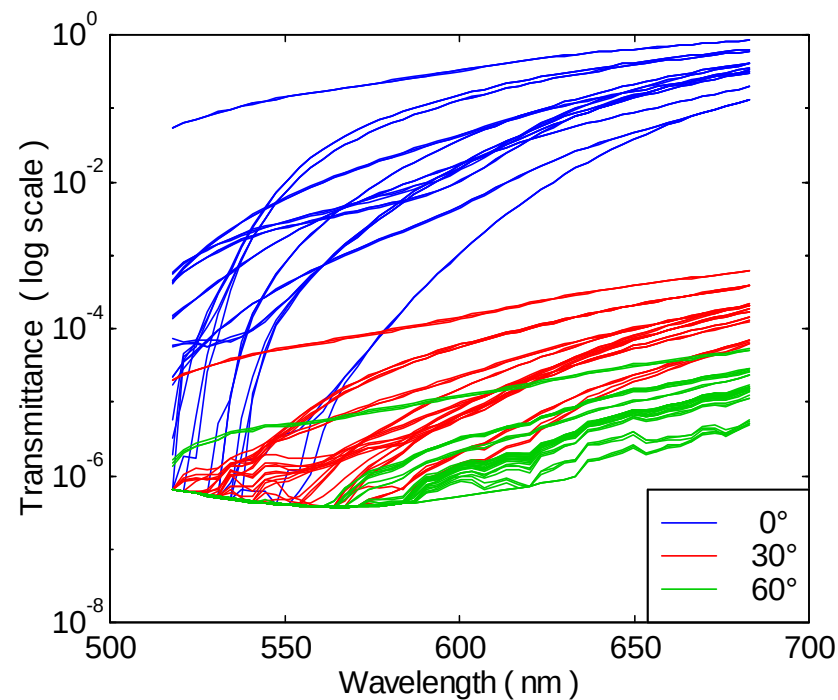
Scattered colorimetry multi- α and multi- λ absorption spectroscopy



- 184 spectral data for each sample
- Multivariate data analysis (PCA-PLS) for dimensionality reduction and sample classification

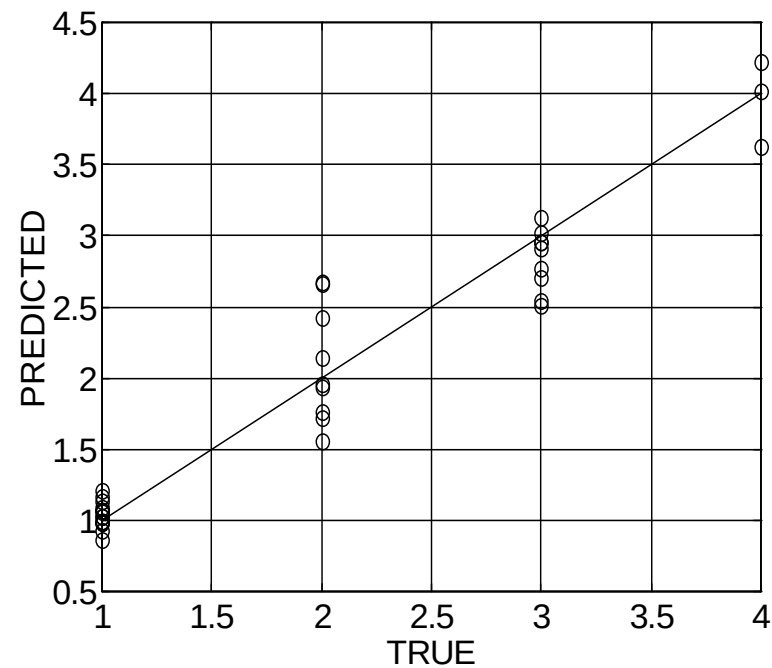
Lubricant oils: Experimental results

- Mobil Jet II polyester-synthetic oil from a gas turbine
- Agip OTE 46 mineral-paraffin oil from a steam turbine



Lubricant oils: Experimental results

Correlation between the spectral data and the chemical-physical status of the lubricant oil (score 1-4)



RMSECV = 0.35 R = 0.967

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Hyperspectral colorimetry as an optical fingerprint - optical signature -

UV-VIS-NIR absorption spectroscopy
(200-1700 nm)

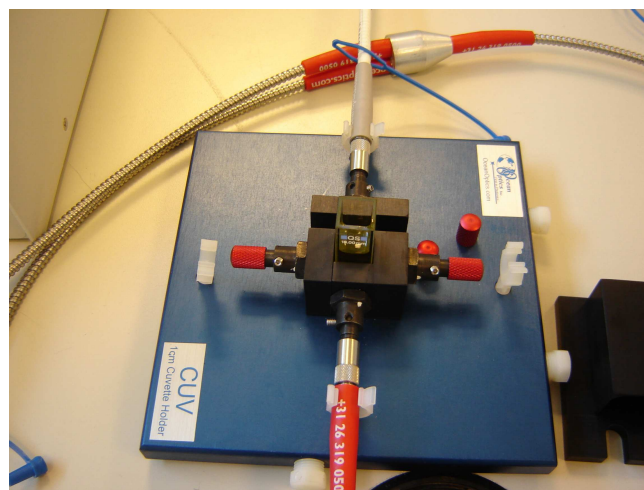
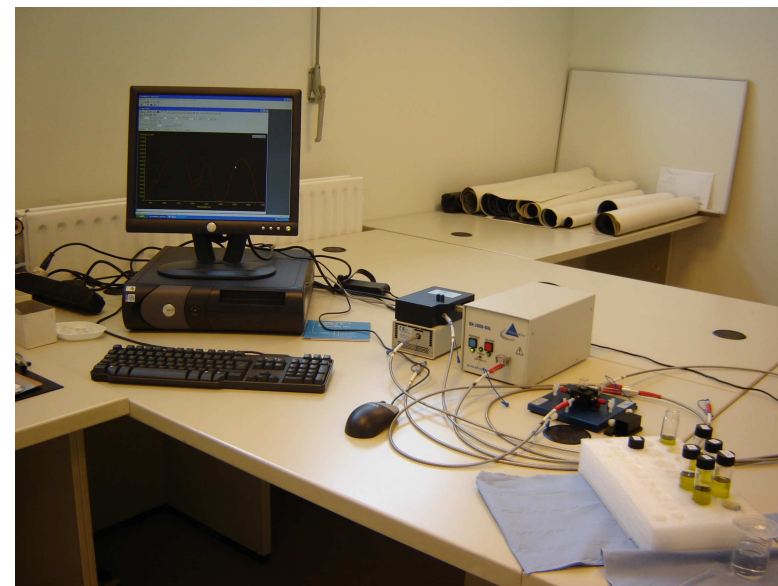
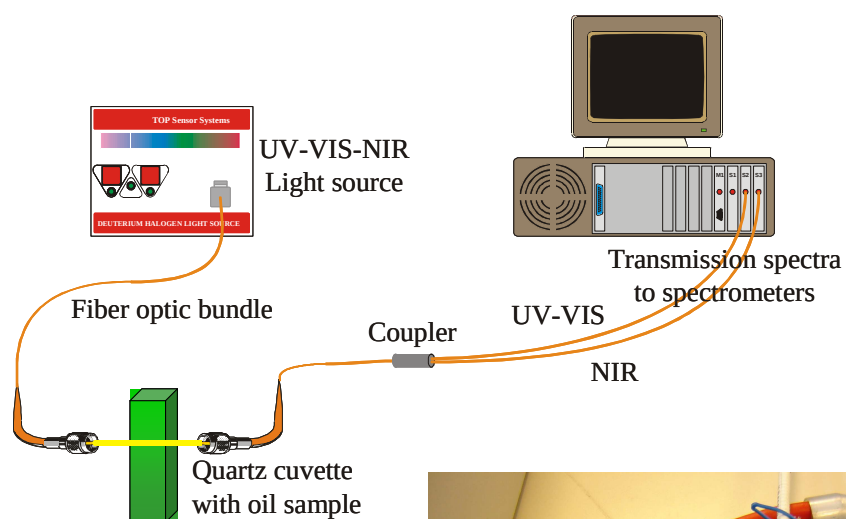
+

Multivariate data processing and classification
(PCA, LDA, PLS, KNN)

=

- 2D map clustering the oils according to similarities
- Correlation of optical parameters with chemical characteristics (fatty acid content)

The instrumentation for optical fingerprint



Extra virgin olive oils

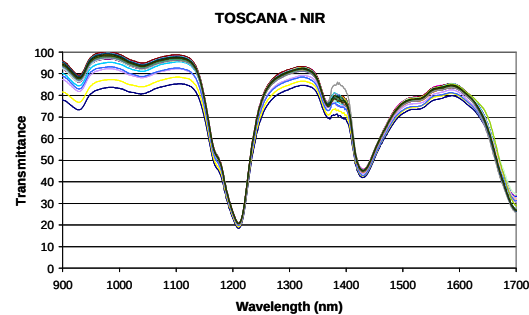
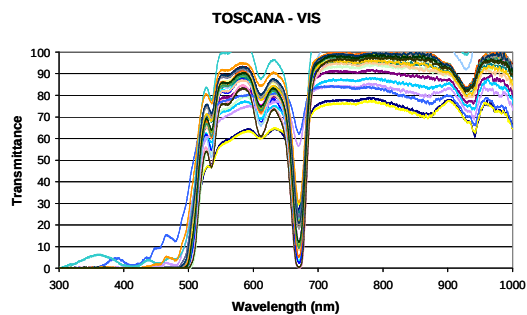
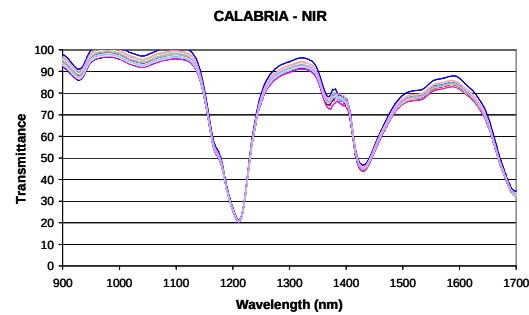
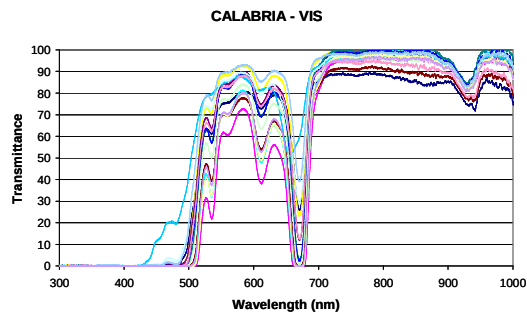
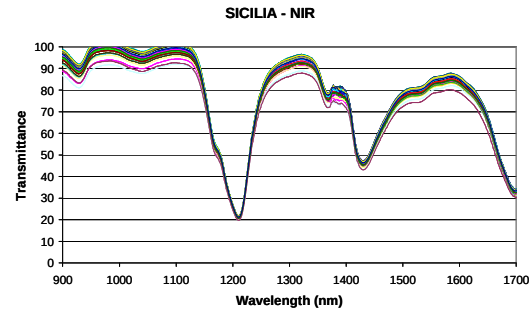
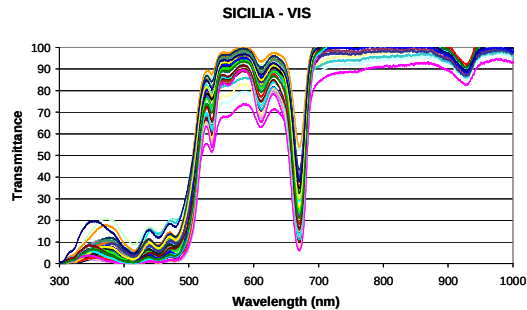
- The library: 85 samples -

- 28 samples from Toscana
 - 15 samples from Calabria
 - 42 samples from Sicilia
-
- ✓ Different cultivar
 - ✓ Different weather conditions
 - ✓ Different harvesting time
 - ✓ Different production systems
 - ✓ All artesanally produced



The measured spectra

- The optical signature -



UV-VIS: 200-1100 nm

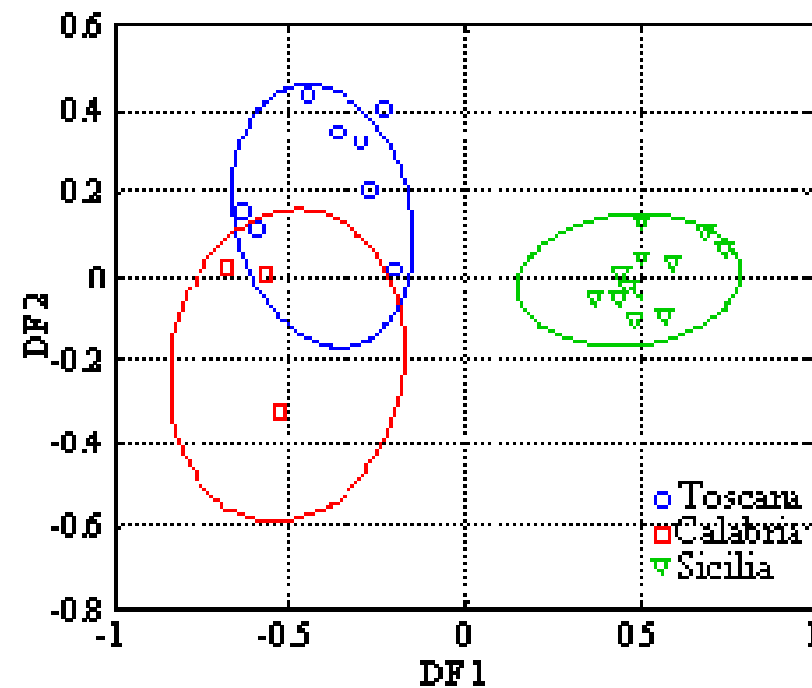
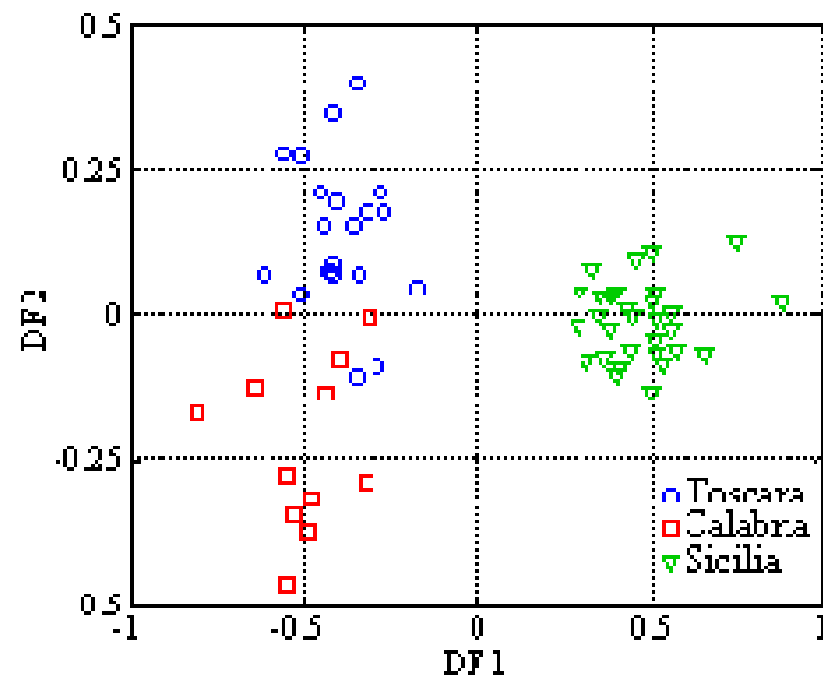
DI: 0.23 nm

NIR: 900-1700 nm

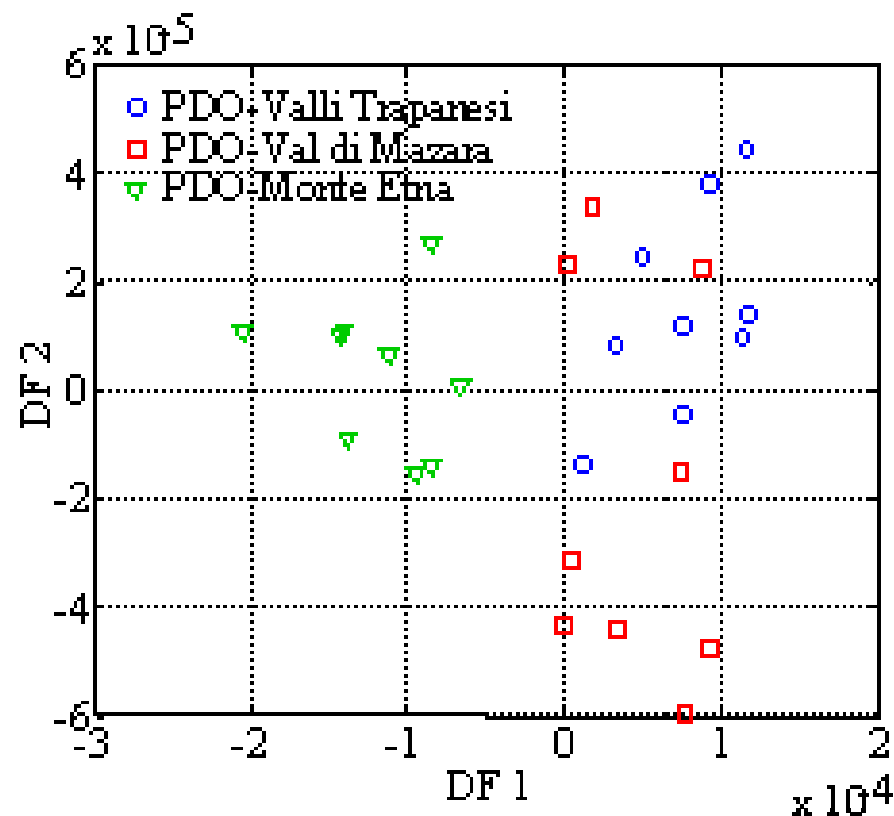
DI: 1.7 nm

Each sample
4160 spectral data

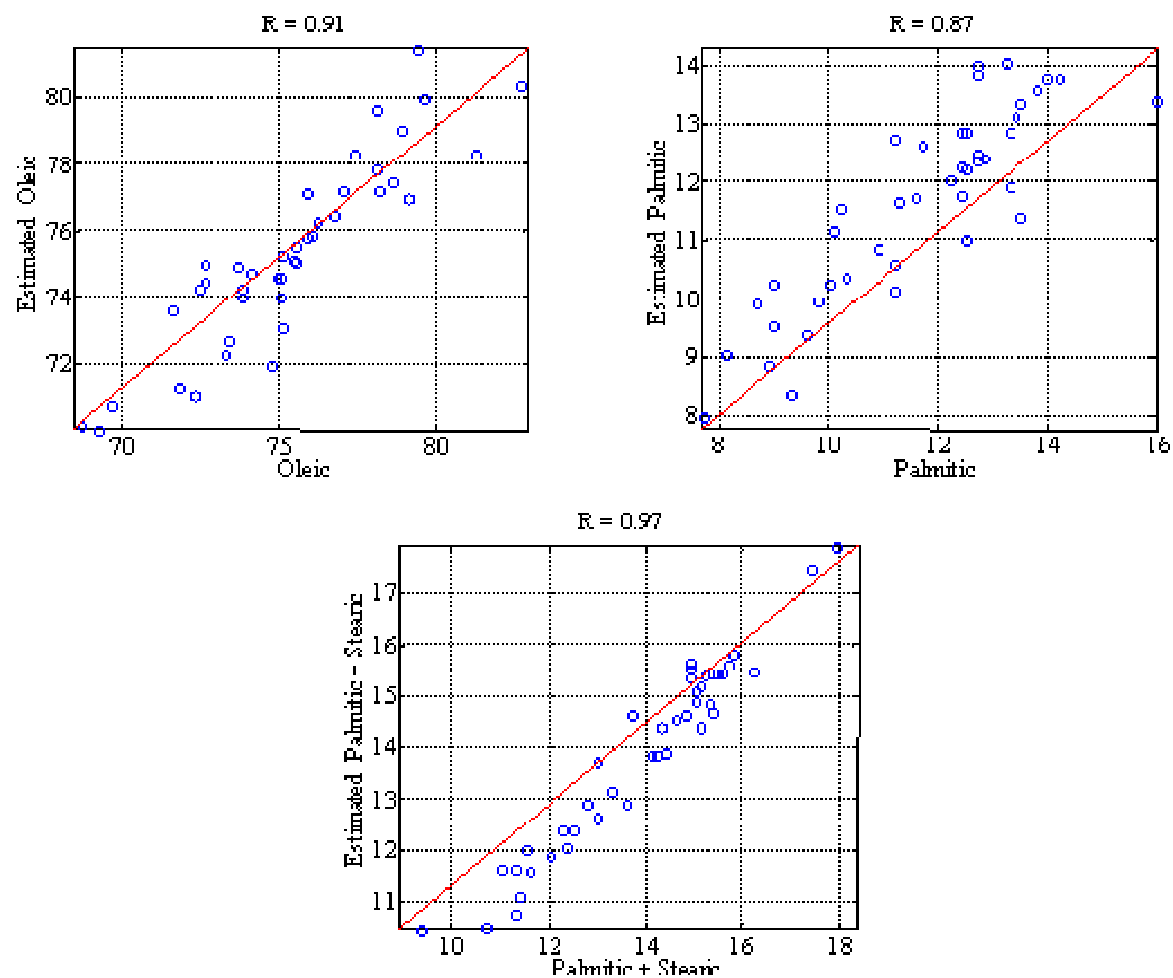
Discrimination of extra virgin olive oils from Sicilia, Calabria, and Toscana



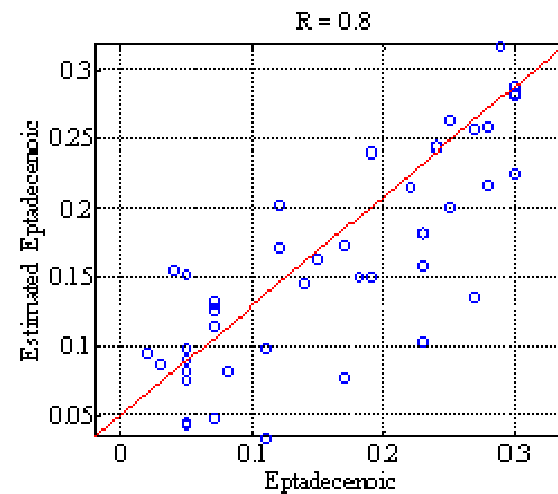
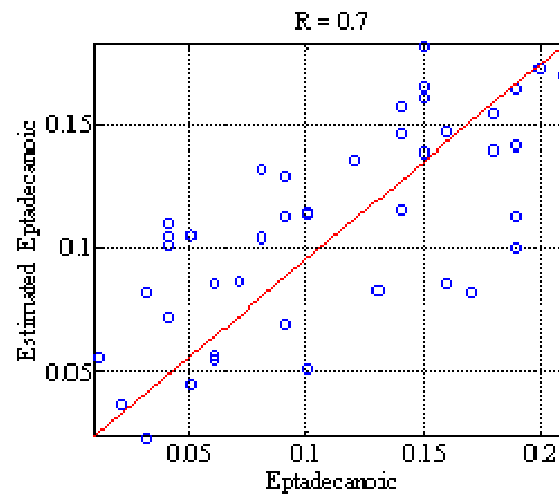
More thorough analysis of extra virgin olive oils from Sicilia: - Discrimination according to PDO -



More thorough analysis of extra virgin olive oils from Sicilia: - Spectral data and fatty acids -



More thorough analysis of extra virgin olive oils from Sicilia: - Spectral data and fatty acids -

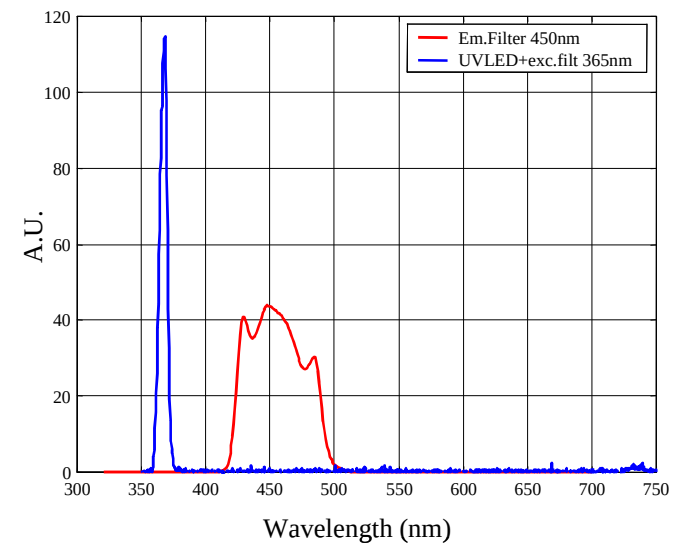
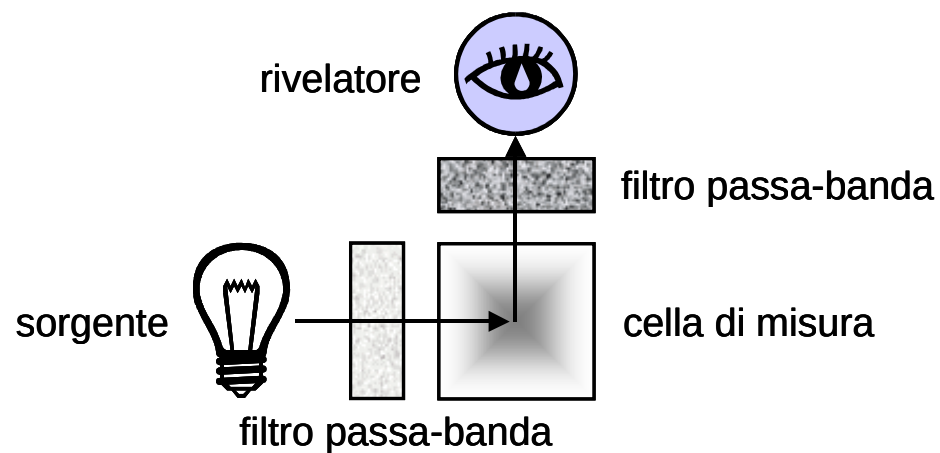


Outline

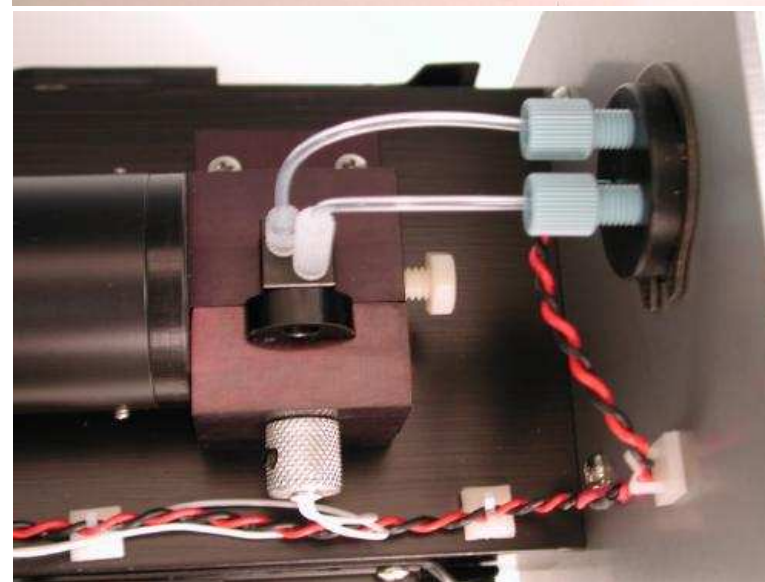
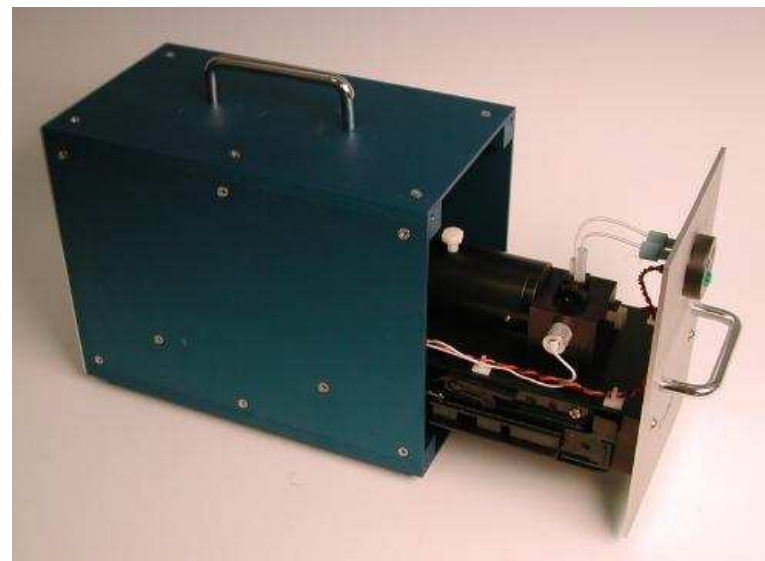
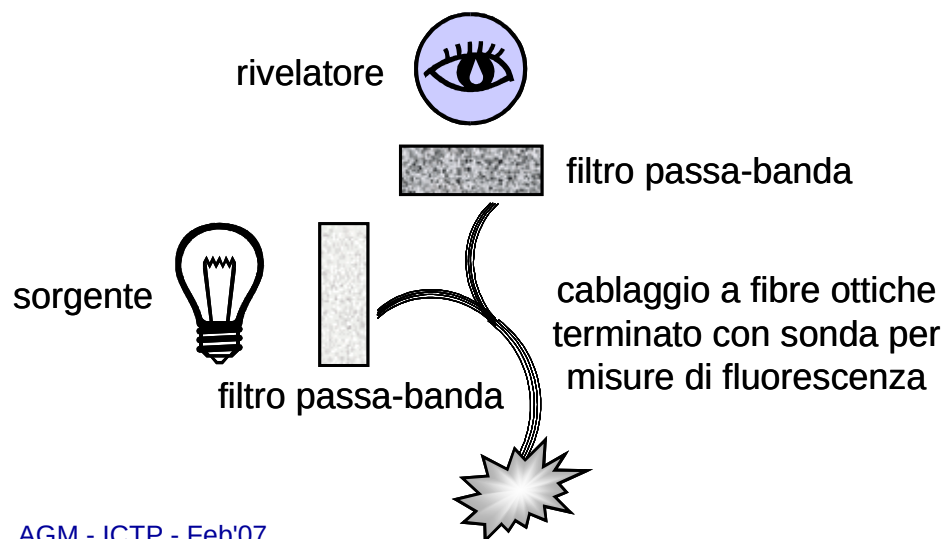
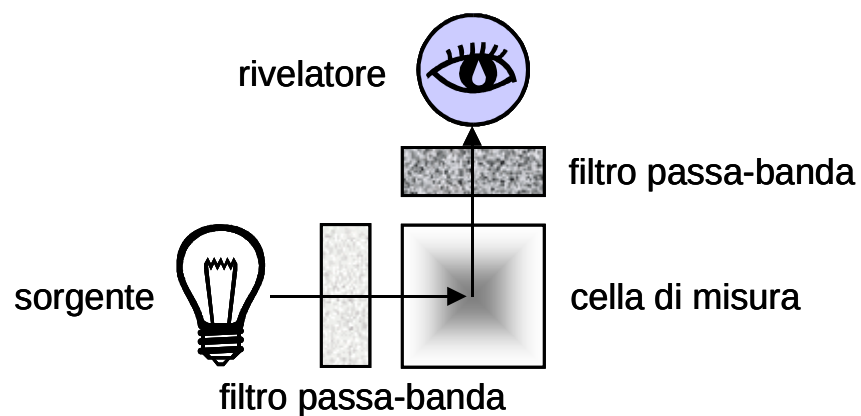
- **Optical fiber sensors: basics**
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- **Hyperspectral colorimetry**
- **Fluorescence spectroscopy**
 - **M1 aflatoxin monitoring**

Fluorescence spectroscopy for M1 aflatoxin detection

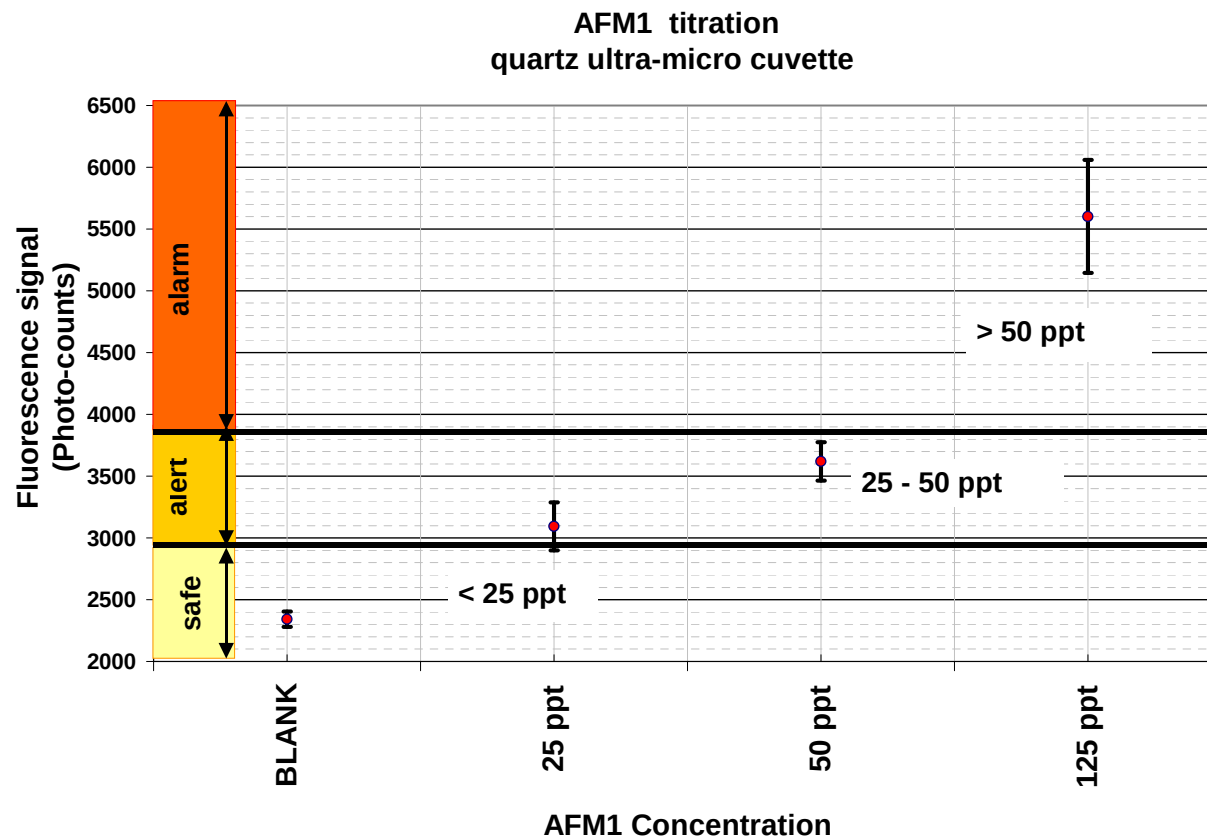
- LED: Roithner Lasertechnik, UVLED365-10 ($\lambda=365$ nm) and high-sensitivity photomultiplier
- Band-pass filters
- Micro-cells



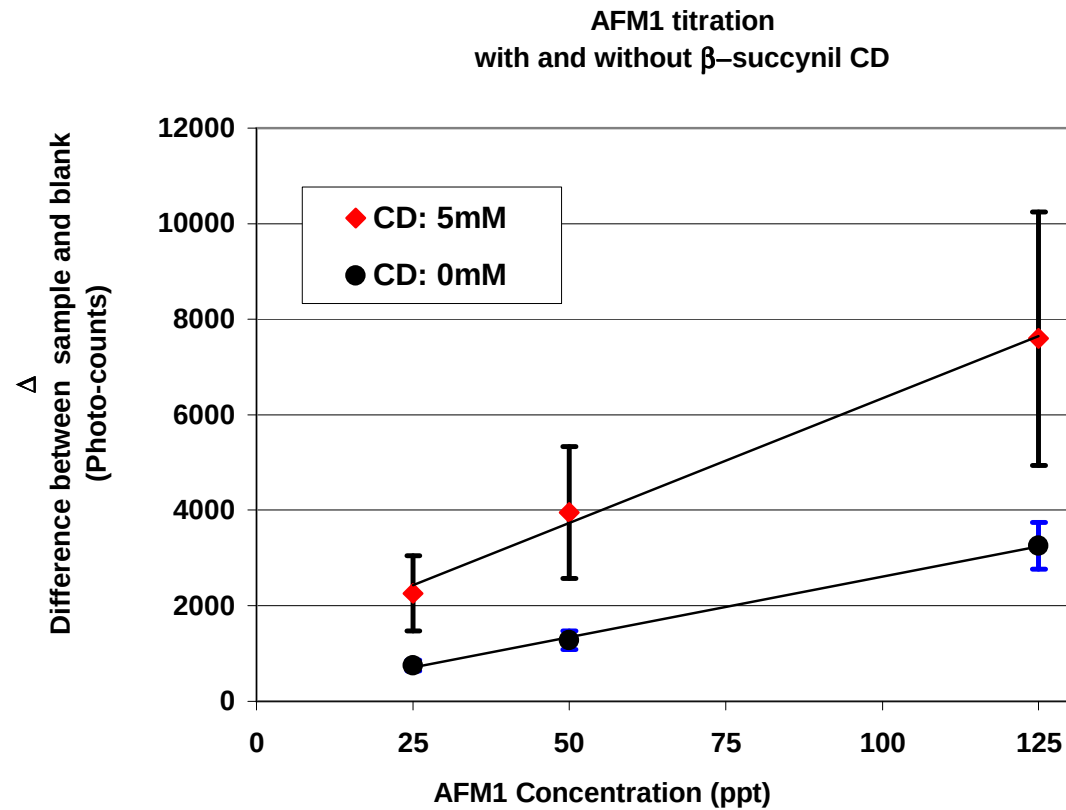
Fluorescence spectroscopy for M1 aflatoxin detection – the instrumentation



Fluorescence spectroscopy for M1 aflatoxin detection – the results



Fluorescence spectroscopy for M1 aflatoxin detection – the results



Acknowledgements

- ✓ European Commission: 'RAMFLAB' project (contract #ERBIC18CT970171)
 - ✓ European Commission: 'OPTIMO' project (contract #ERBIC18CT970171)
 - ✓ European Commission: Network of Excellence on Micro-Optics
 - ✓ CNR-MADESS II: 'OptoSpec' project
 - ✓ Regione Toscana: 'RIS+', 'SERQUA' and 'CARABIOTEC' projects
 - ✓ MIUR: FISR 'ARGO' project and FIRB contract #RBNE01KZZM
 - ✓ CNR-Short Term Mobility Programme
-
- ✓ Andrea Mencaglia
 - ✓ Patrizia Bizzarri
 - ✓ Leonardo Ciaccheri
 - ✓ Franco Cosi

