Promising fiber solutions for molecular analysis in food industry

Alexander Novikov

EPIC Online Technology Meeting
11. December 2023

art photonics
broad spectra fiber solutions

EPIC
EUROPEAN PHOTONICS INDUSTRY CONSORTIUM

art photonics
broad spectra fiber solutions

art photonics GmbH is founded in Berlin, Germany in 1998 by Dr. Viacheslav Artyushenko

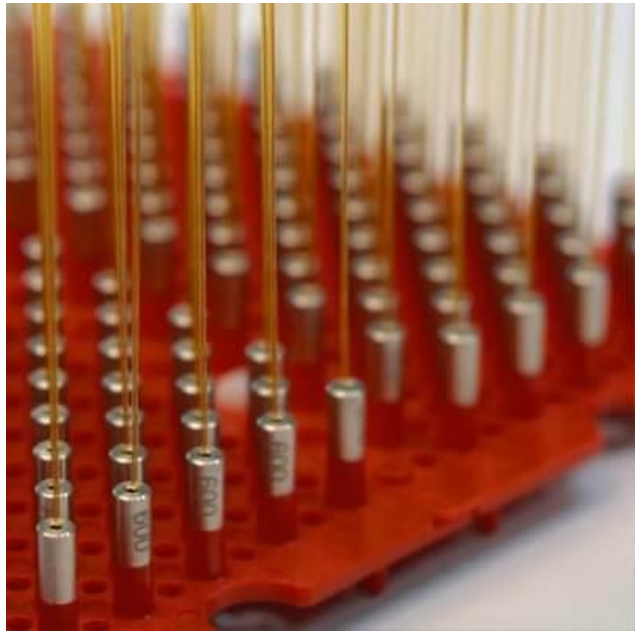
R&D and production of specialty **fiber products** for a broad spectrum range **0.3 – 16 µm**

Unique technologies of Polycrystalline Mid InfraRed (PIR-) fibers, Hollow waveguides (HWGs) and Metal coated Silica fibers

Spectroscopy probes for medical diagnostics and industrial process control

24% of our employees have a PhD

25 Years Anniversary



Made in Germany

Process monitoring and control. Why?

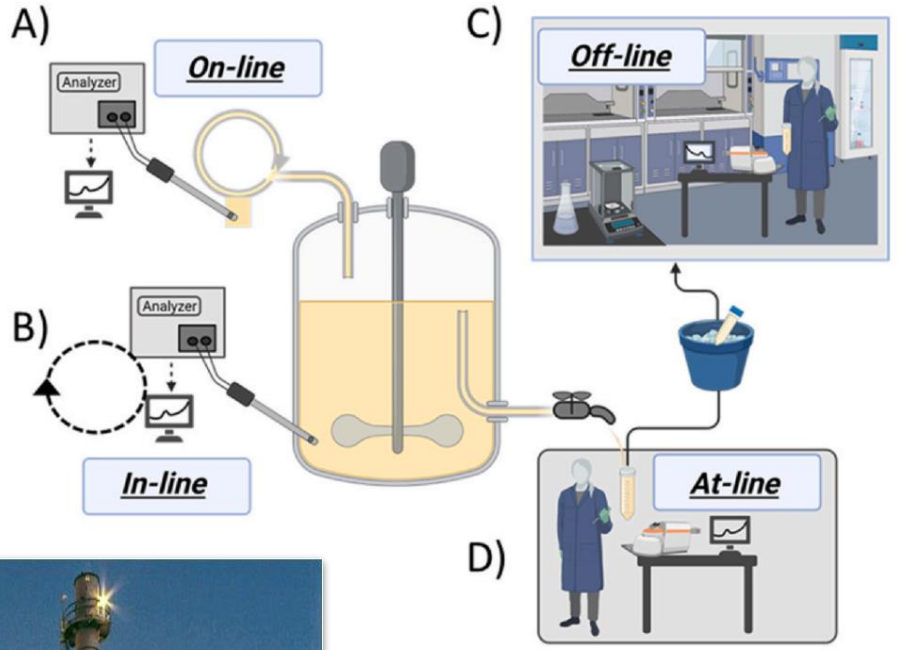
Economical benefit & Research

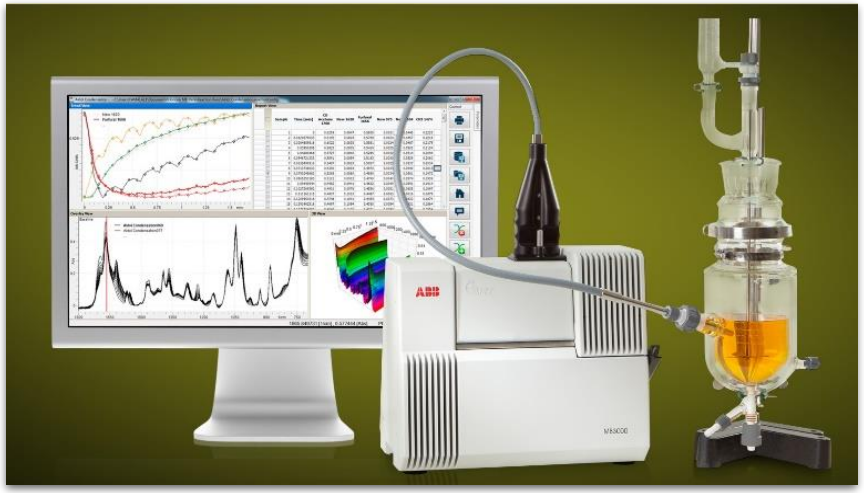
- Maintain **product quality** and consistency
- **Optimize** resource utilization and minimize waste
- Enhance process **efficiency** and productivity
- Obtaining **accurate and reliable data**
  for better understanding of the underlying mechanisms and dynamics of a process
- Ensure compliance with **regulatory requirements**
- Improve **safety** by identifying and mitigating potential hazards
- Return of investment (ROI) is less than 2.5 years
  for most PAT projects (30k – 300k $)*

*Dr. Martin Hajduk, “PAT in the Process Industry – Nice To Have or Efficient Cash Cow?”, Hellma PAT-Forum Webinar, 18.04.2023

Process monitoring and control. How?

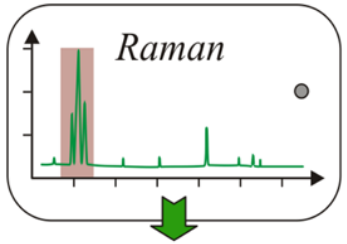
- Spectroscopy (more selective)
- Fiber-optics (more robust)
- In-line / On-line sampling for real-time monitoring (saves time and qualified human resources comparing to At-line and Off-line)

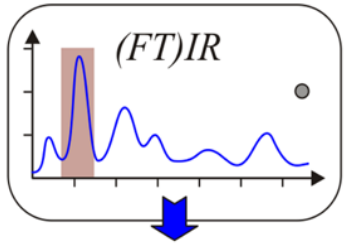


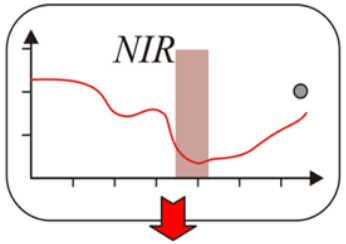


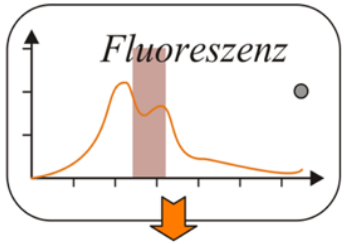
Gerzon, G., Sheng, Y., & Kirkitadze, M. (2022). Process Analytical Technologies–Advances in bioprocess integration and future perspectives. *Journal of Pharmaceutical and Biomedical Analysis*, *207*, 114379.

Key spectroscopy methods





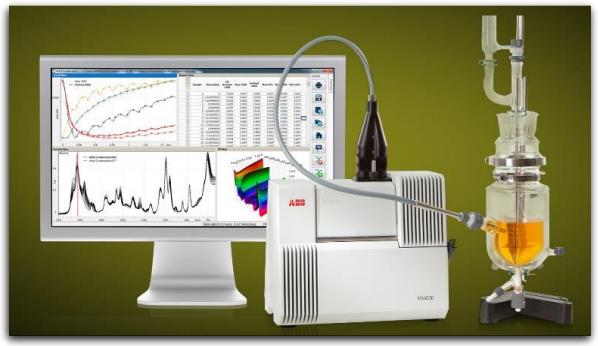




Method\Factor	Raman	FT-IR ATR	Vis/NIR	Fluorescence
Selectivity	high	high	low	low
Sale price	high	high	small-medium	small-medium
Penetration depth	small	small	high	medium
The main disadvantages	too weak Raman signals	measurement slowness by FT-IR	Water absorption	low information content

Advantages of fiber-coupled sensors / probes

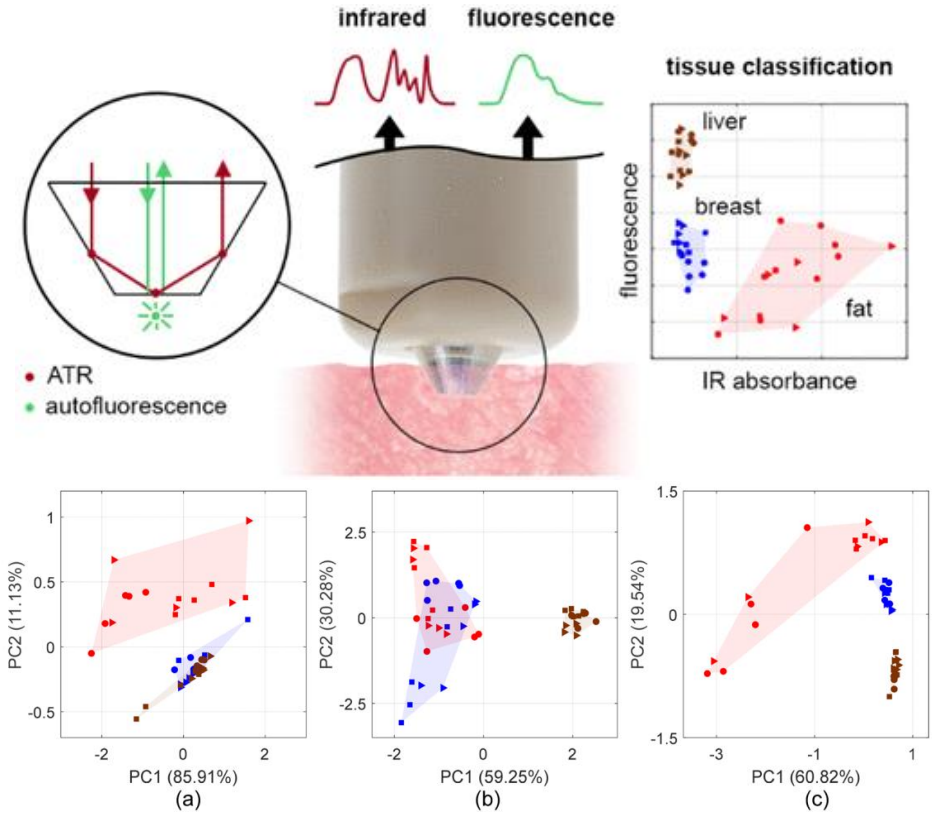
- ✓Easy & Safe spectroscopy with no sample preparation
- ✓In-line / in situ online process monitoring, in vivo measurements
- ✓Remote sensing for „hard-to-get“ samples
- ✓Single-use / disposable sensor tips
- ✓**Sterilizable sensor tips (using autoclave, radiation, plasma)**
- ✓**Reaction initiation / end-point / kinetics determination**
- ✓**Hazardous conditions (high temp, pressure, pH, vibration, etc.)**
- ✓Aggressive / toxic media
- ✓**Air / Moisture sensitive samples**



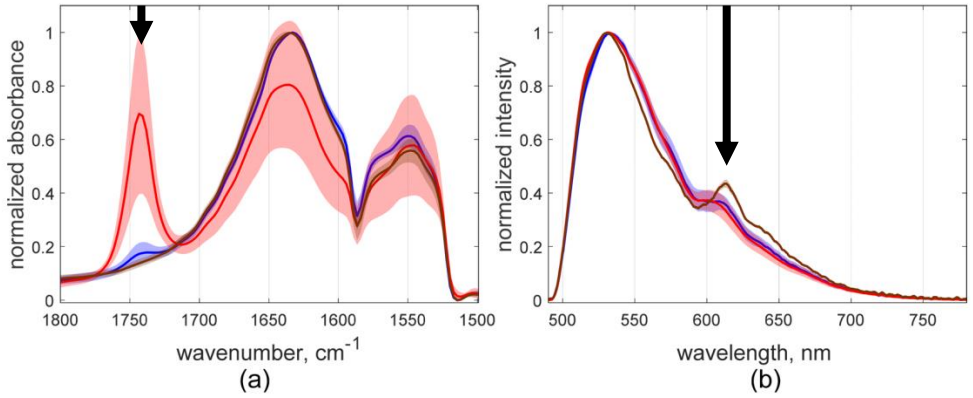
Adding combination to bring synergy

From our research:

- **Mid-IR + Fluorescence: *Better distinguish*** kidney RC-carcinoma tumor
- **Near-IR + Fluorescence: *Higher sensitivity*** in discrimination between malignant and benign colorectal tissue
- **Near-IR + Mid-IR: *Increasing the accuracy*** of abdominal cancer detection



- Combi-probe for Fluorescence & ATR (mid-IR)
- Experiments with **chicken tissues: liver, breast and fat**
- Same measurement point in real time
- 1st Dual wavelength Combi Fiber sensor
- **Full separation of 3 different tissues using only 2 spectral variables: 5.74 µm (1743 cm-1 Mid-IR) + 613 nm (Fluo)**



Bogomolov, A., et al. *Sensors*, *17*(11), 2548. https://doi.org/10.3390/s17112548

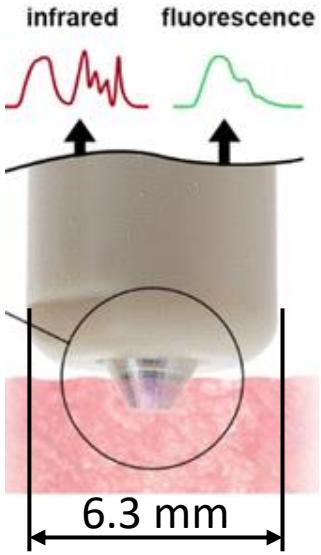
Ehlen, L., et al. journal of surgical research (2019), 242, 349-356. https://doi.org/10.1016/j.jss.2019.05.011

Hocotz, T., et al. Sensors, 20(22), 6706 https://doi.org/10.3390/s20226706

Bogomolov, A., et al. *Analytical Chemistry (2021)*, *93*(15), 6013-6018. https://doi.org/10.1021/acs.analchem.1c00080

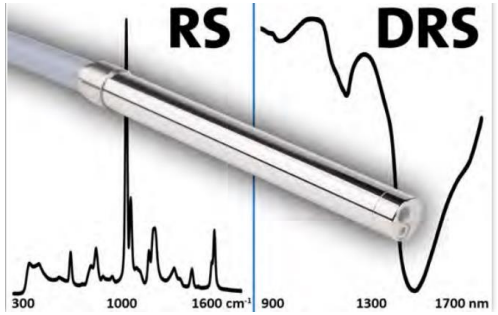
Our compact combi probes for enhanced monitoring and control

ATR + Fluo + Raman



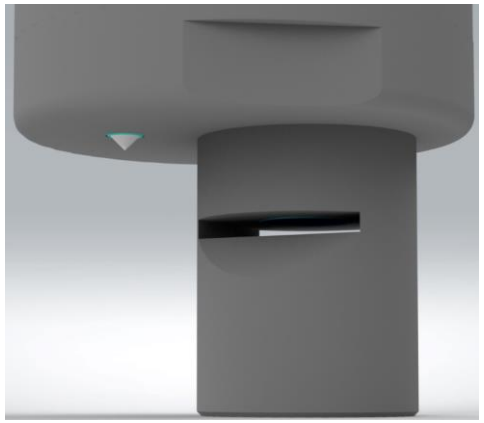
- 2-3 modalities
- Cost-effective lab design

NIR-Diffuse reflectance + Raman



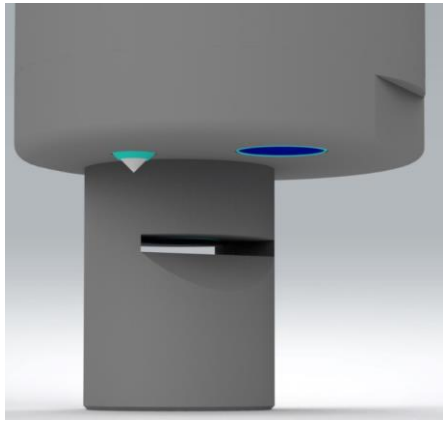
- 19 mm diameter
- NIR & Raman channels
- Straylight < 1%
- Heated shaft to prevent condensation
- Designed for **bio-pharma**

ATR + NIR



- 25 mm diameter
- ATR-FTIR channel
- NIR Transflex channel

ATR + NIR + Raman



- 25 mm diameter
- ATR-FTIR channel
- NIR Transflex channel
- Raman channel

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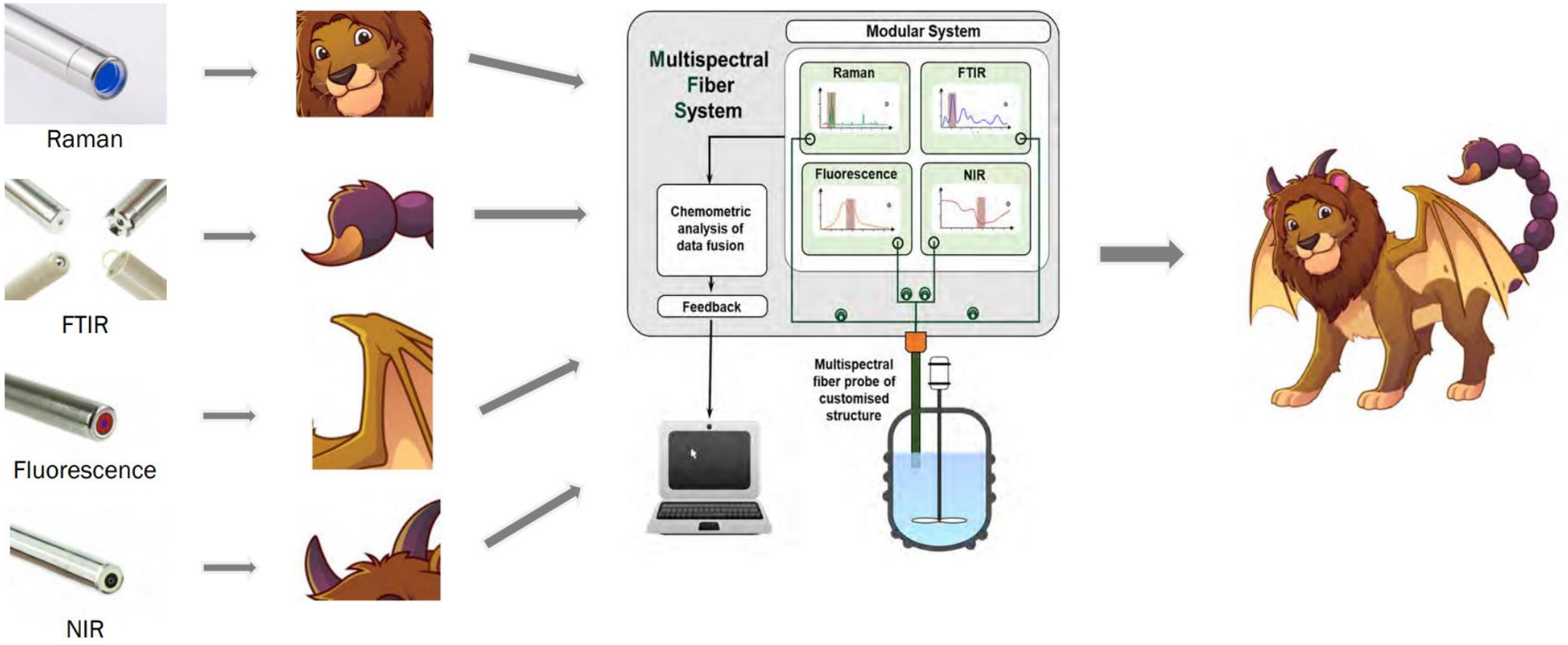
Multispectral Combi-Fiber Probes



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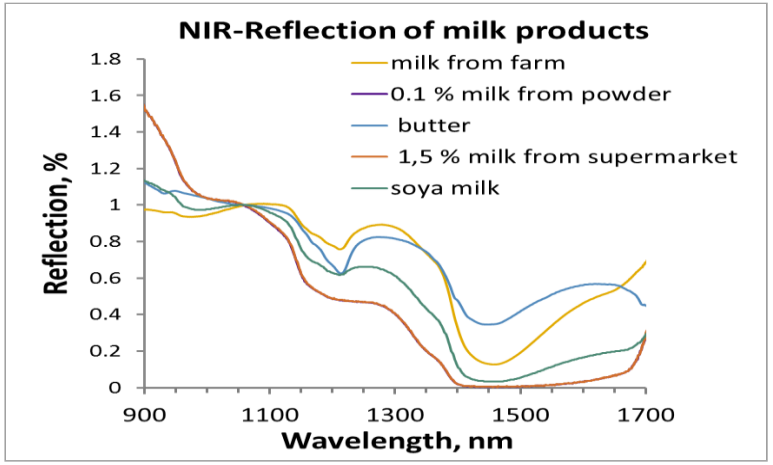
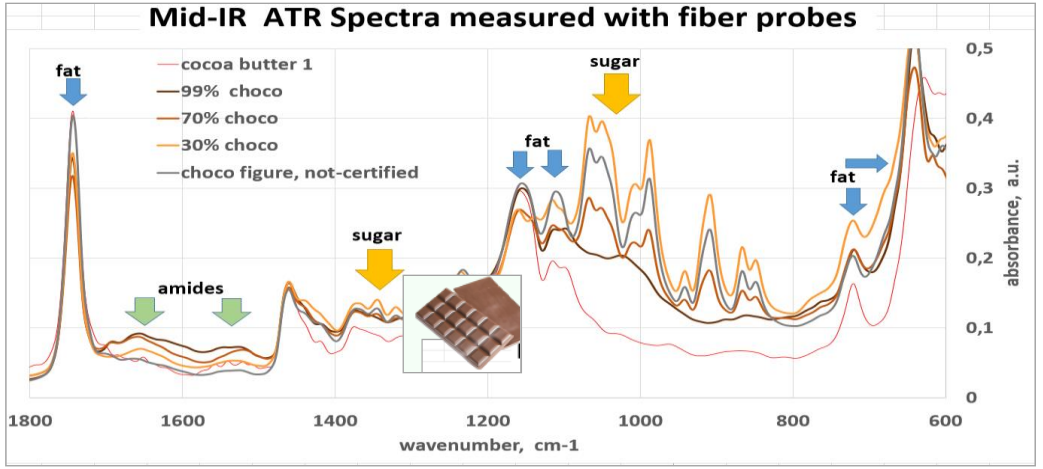
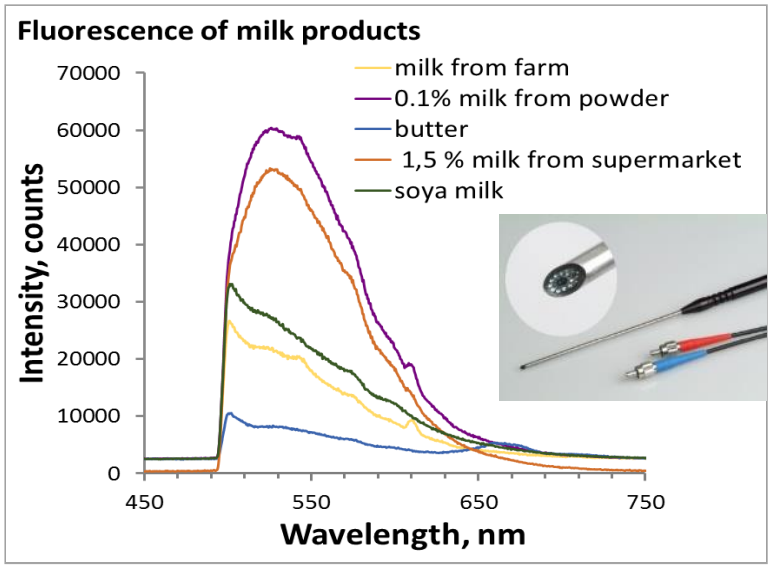
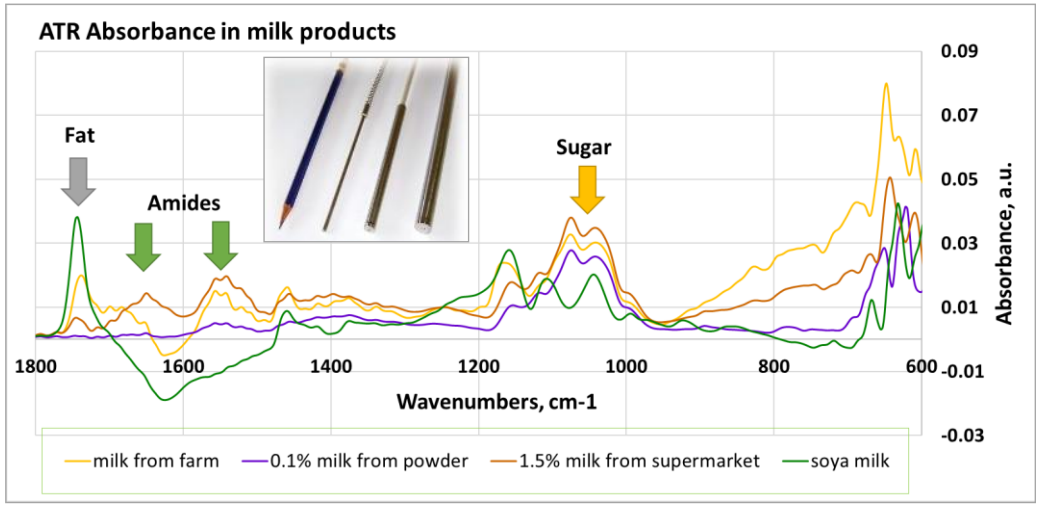
Multi-spectral Analysis of Milk Products

Different spectroscopy methods provides complimentary information in real time on milk and chocolate composition & quality









Multi-spectral system at art photonics lab

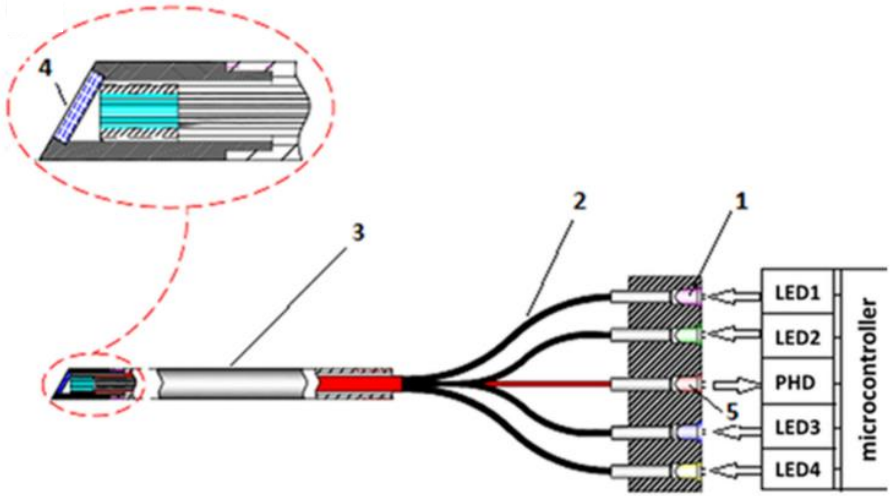
Optical multisensor systems (OMS)

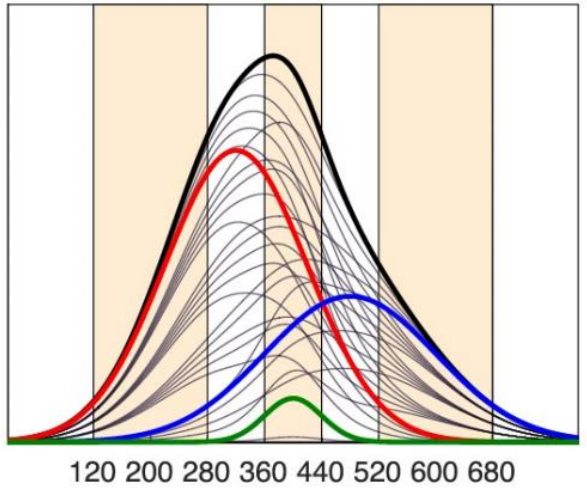
- Analytical device with at least 2+ optical chemical sensors
- Optimized for the specific application

Different from conventional spectroscopic analysis:

Parameter of Analyzer	Spectrometer	OMS
Application area	Universal	Specialized
Selectivity	High	Low
Application of Chemometrics	Recommended	Requried

- Relatively **low cost**
- Compact (**portability**)
- High measurement **speed**
- **Autonomous** realization
- Unlimited spectral range
- User may not be an analyst





Optimization of OMC channels on full-spectrum experiment data.

Bogomolov, A. (2021). Developing multisensory approach to the optical spectral analysis. *Sensors*, *21*(10), 3541.

Bogomolov, A., et al. (2023). New approaches to data processing and analysis in optical sensing. TrAC Trends in Analytical Chemistry, 116950.

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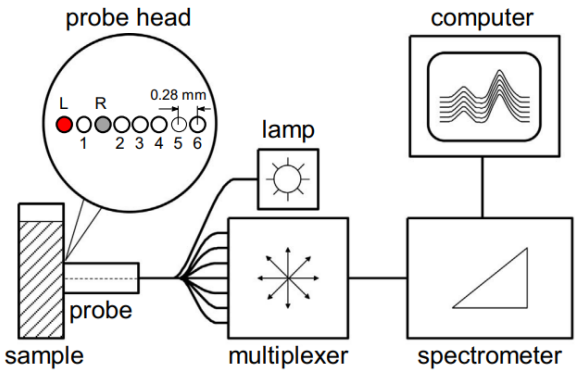
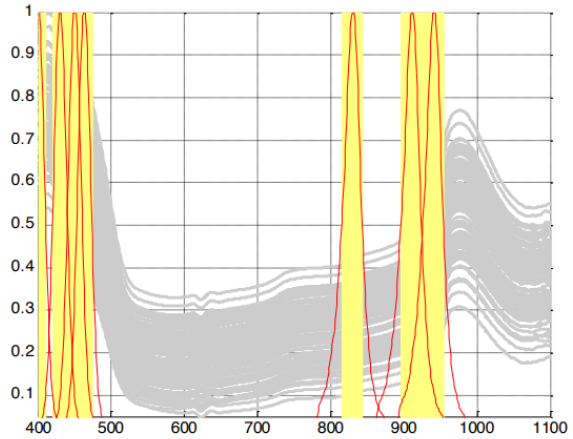
Optical multisensor systems (OMS)

- Common opinion *„Sensor system is not a spectrometer, but at least cheaper“* **is not correct**!
- A properly built OMS can and must be **better than a spectrometer** due to:
  - Less non-relevant information/signals, less noise
  - Simplified mathematical model
  - No need to study wavelengths one by one, expanding the capabilities of the spectral method!
  - Ability to use the whole spectral range, combine different physical techniques
- Application case: **determination of fat and protein in milk**:
  - Visible and NIR region
  - Optimization of amount and parameters of LEDs from spectral data
  - 7 LEDs
  - Graduation statistics **are noticeably better** than for the model on full-spectral data

- Application case with ATR probes and pyroelectric MIR sensor for in-line process monitoring in biotechnology (Biomass/Glucose/Ethanol):
  - Good enough results with much more affordable MIR sensors





%fat model	Full spectra	LED-Sensor
RMSEC	0.1040	0.0846
RMSECV	0.1158	0.0947
R^2C	0.9746	0.9832
R^2CV	0.9685	0.9790

Bogomolov, A., et al. (2017). Reference-free spectroscopic determination of fat and protein in milk in the visible and near infrared region below 1000 nm using spatially resolved diffuse reflectance fiber probe. Talanta, 167, 563-572.

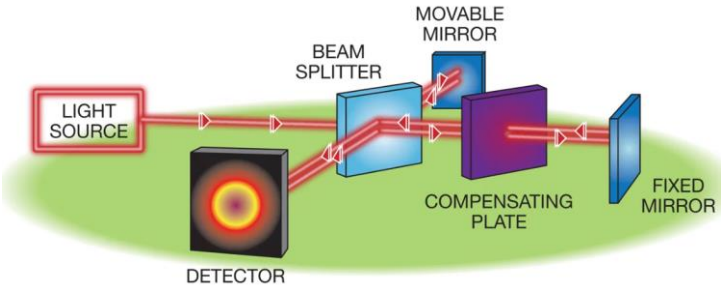
Bogomolov, A., et al. (2015). Development and testing of mid-infrared sensors for in-line process monitoring in biotechnology. Sensors and Actuators B: Chemical, 221, 1601-1610.

Surkova, A., et al. (2023). LED-Based Desktop Analyzer for Fat Content Determination in Milk. Sensors, 23(15), 6861.

NIR spectrometers / sensors

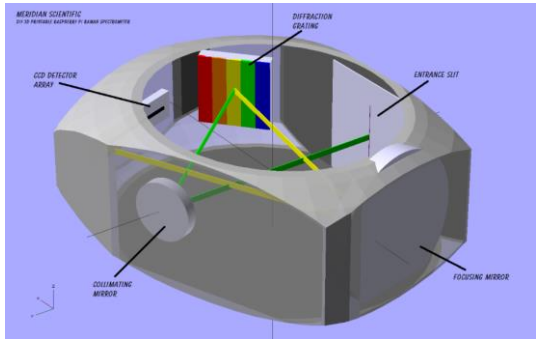
FTIR spectrometer:

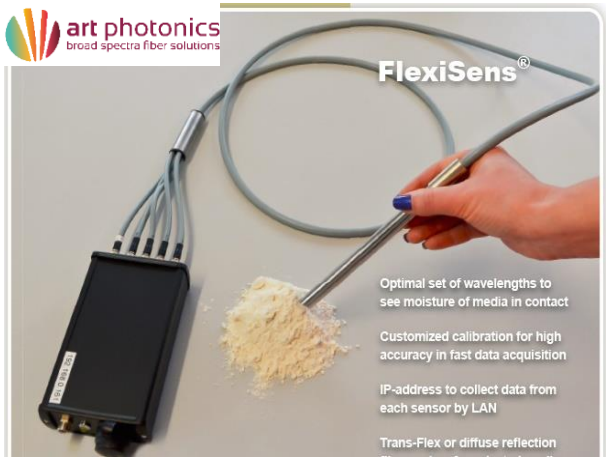
- Broad spectral range 850 – 2500 nm
- High resolution, but
- Expensive
- Slow
- Sensitive to vibrations

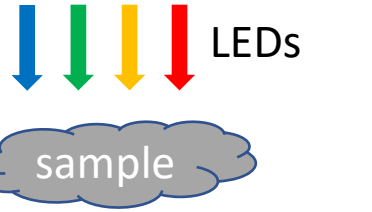


Diode array / Diffraction grating spectrometer:

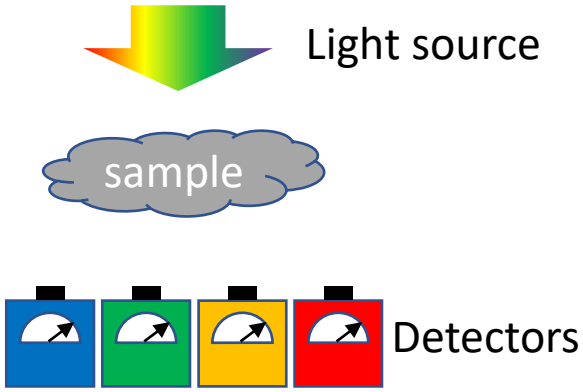
- Spectral range 900 – 1700 nm
- Low resolution
- Low cost
- Fast
- Robust







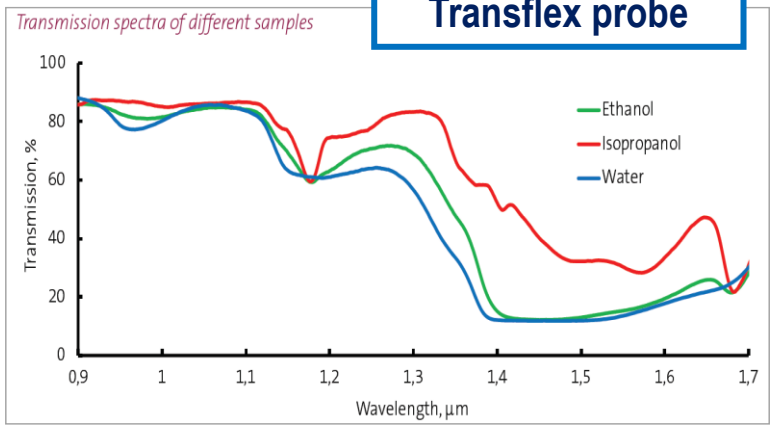
NIR Moisture sensor from art photonics GmbH
4 narrow light sources - 1 broad detector



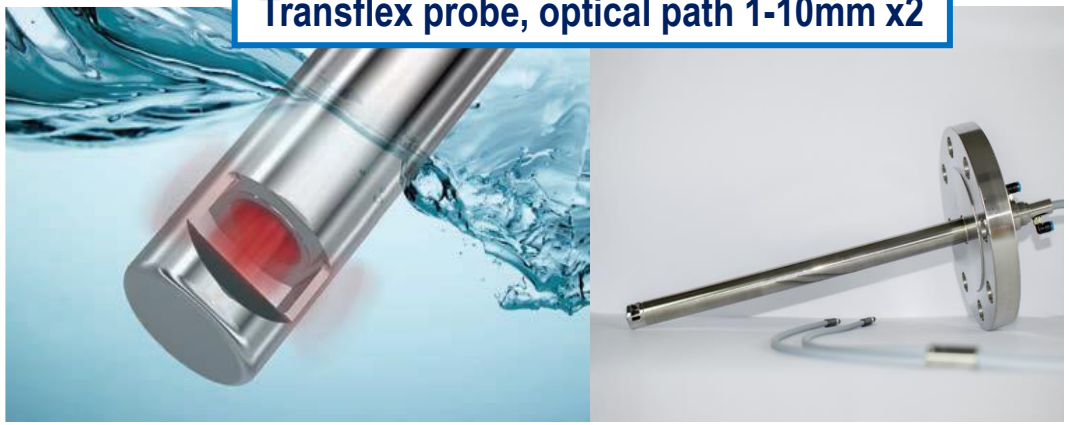
NIR sensor from Mantispectra with a probe from art photonics
1 broad light source - 16 detectors with various filters

NIR transmission / transflection fiber probes

Transflex probe

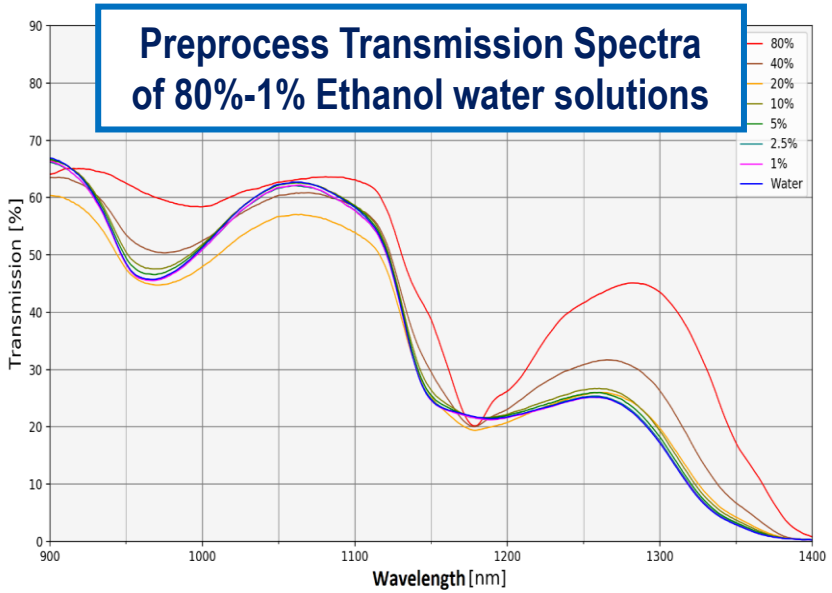


Transflex probe, optical path 1-10mm x2

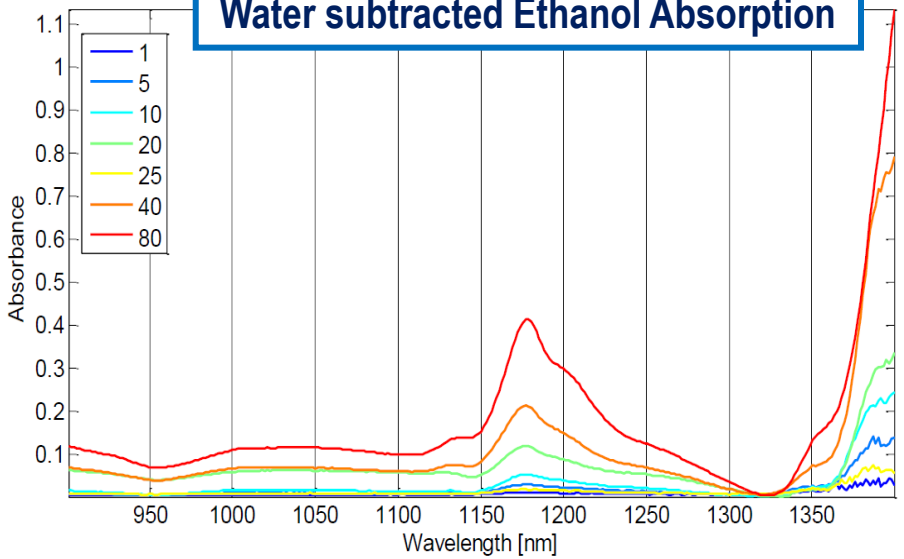


Hygienic transflex probe (3-A standard)

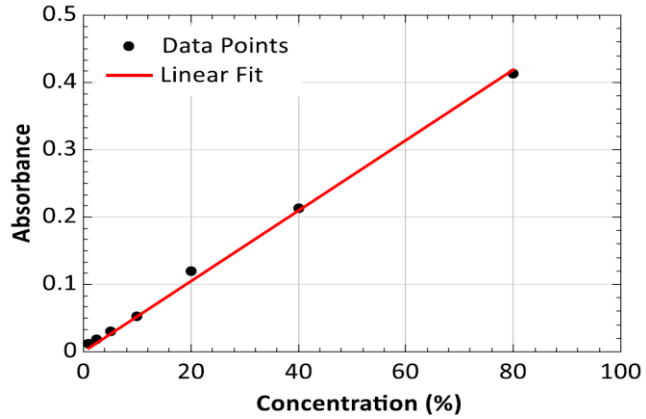
Preprocess Transmission Spectra of 80%-1% Ethanol water solutions



Water subtracted Ethanol Absorption

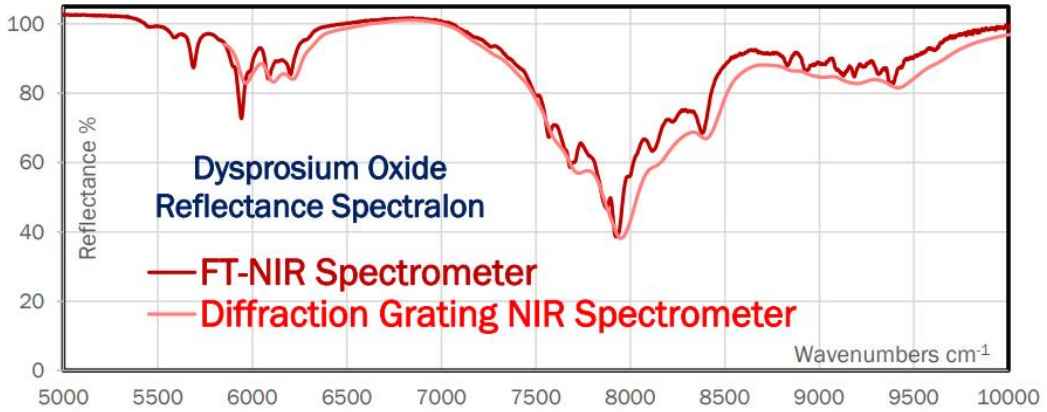


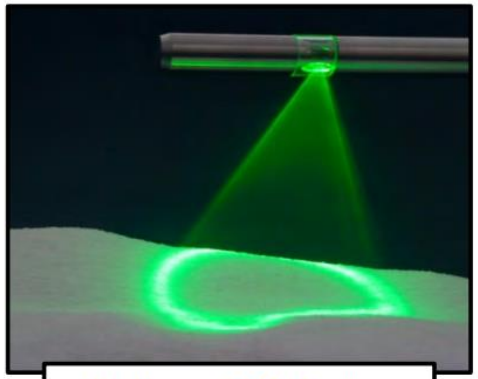
Ethanol peak decay at 1177nm



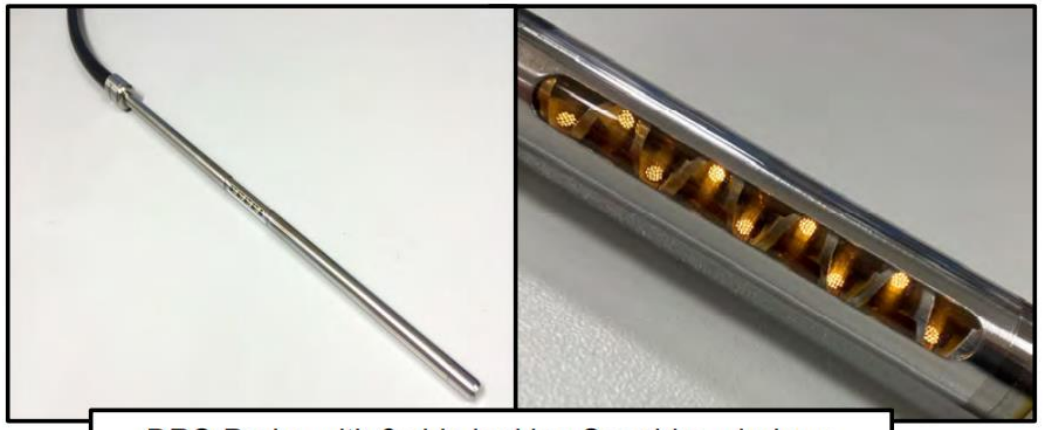
NIR Reflection fiber probes

12mm Reflexion Probe



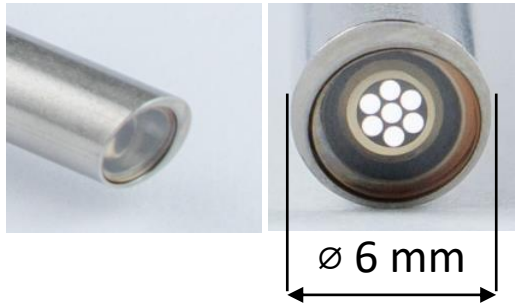


Fish-eye DRS-Probe
with 2 side windows

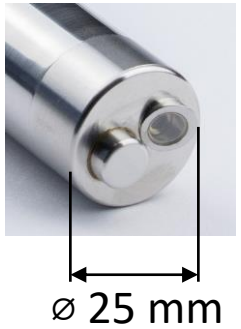


DRS-Probe with 9 side looking Sapphire windows

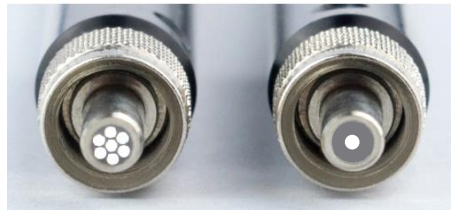
Angled optical window to reduce straylight

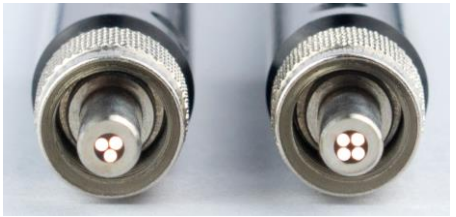


Rinse system to keep the window clean



To use with **diffraction grating** the output bundle should fit to the **slit size**.





To use with **FTIR** the output bundle should fit to the **detector** size.

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Thank you for your attention!

25 Years Anniversary

Any questions?
Alexander Novikov, e-mail: an@artphotonics.com

Welcome to visit us!

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Rudower Chaussee 46
Technologiepark Adlershof
12489 Berlin, Germany

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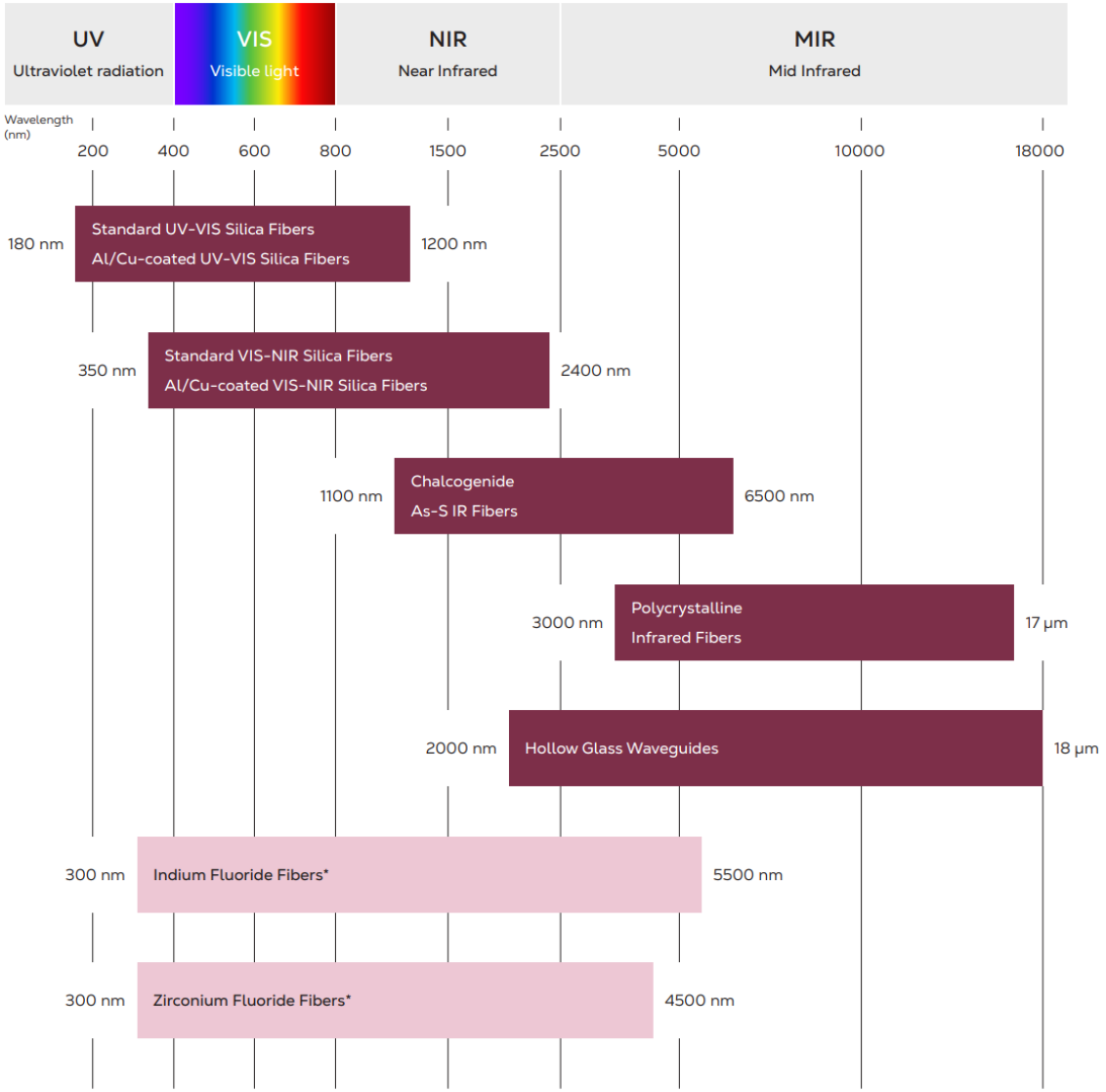
Optical fibers for spectroscopy applications



Fiber Type	Advantages	Drawbacks	Applications
Silica fiber Pure silica core & F-doped cladding	■ 0.2 – 2.4 µm ■ Non- toxic ■ Non-hygroscopic ■ Stable up to 600°C ■ Photonic crystals !	■ Brittle without coating	■ Telecom ■ Spectroscopy probes ■ Imaging bundles ■ Laser power delivery up to 10 kW ■ Illumination
POF fiber plastic optical fiber from PMMA, CYTOP, polystyrol, etc.	0,4 – 0,8 µm High flexibility Low cost Easy installation Photonic crystals !	■ Limited transmittance ■ Limited range of temperature: –55°C / +85°C	■ Illumination ■ Local telecom ■ Light control in auto, plane, etc. ■ Fiber sensing for various parameters
Sapphire fiber single-crystalline	■ 0.5 – 3.4 µm range ■ Non toxic ■ Stable up to 2000°C	■ No cladding ■ Stiff & Brittle	■ High power delivery for Er:YAG & Er:YSGG laser ■ Surgery ■ Spectroscopy probes
CIR-fiber chalcogenide IR glasses: As-S or Ge-As-Se-Te chalco-halide glasses Te-Ge-I	■ Transmittance in 0.7 – 6 µm (As-S) or 2 – 10 µm (GeAsSeTe) ■ Stable for 250 – 400 K ■ Non-hygroscopic	■ Fragile ■ Toxic ■ Low T_g (450 K) ■ High dn/dT ■ High N	■ Spectroscopy probes for gases & liquids ■ Flexible radiometry ■ IR-imaging bundles for endoscopy
PIR-Fiber polycrystalline IR crystals from silver halide solid solutions	■ Transmittance in wide 3 – 18 µm range ■ Non-brittle ■ Non toxic ■ Non-hygroscopic ■ Stable in 5 – 600 K	■ High scattering from 0.6 to 3 µm ■ UV-sensitive ■ Corrosive in contact with some metals	■ Spectroscopy ■ Probes for gases & liquids ■ Flexible radiometry ■ IR-imaging bundles ■ Power delivery (50 W) for CO-/CO_2-lasers
Hollow waveguides silica, silver or polymer tubes with reflective inner coating	■ High transmittance ■ Low divergence ■ High damage ■ Threshold (> 2 kW) for Er:YAG / CO_2-lasers ■ Rugged & durable	■ Sensitive to bending (3 dB at 10 cm radius) ■ High losses for transmission of NA > 0.1	High laser power delivery (> 2 kW) for Er:YAG / CO_2-lasers Spectral sensors for gas flow through hollow guide cell

Artyushenko, V., et al. "Mid-infrared Fiber Optics for 1—18 µm Range: IR-fibers and waveguides for laser power delivery and spectral sensing." Optik & Photonik 9.4 (2014): 35-39.