Dr. Tatiana Sakharova, CTO

art photonics

“Benefits of *in-line* fiber spectroscopy for industrial process-control & medical diagnostics”.

EPIC Meeting on Fiber Sensors at HBK FiberSensing, 19-20 April 2023 Porto, Portugal

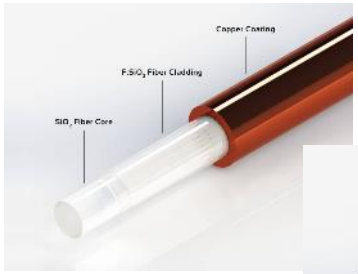
EPIC
EUROPEAN PHOTONICS INDUSTRY CONSORTIUM

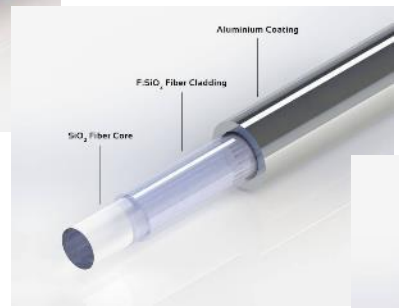
UNIQUE FIBER TECHNOLOGIES

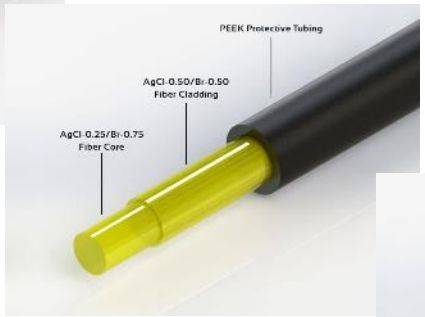
art photonics GmbH was founded in 1998 in Technopark Adlershof, Berlin, to transform unique fiber technologies accumulated by its team to broad variety of fiber solutions: high power laser cables, spectroscopy probes & coherent bundles for the broadest spectral range from 180nm to 18µm

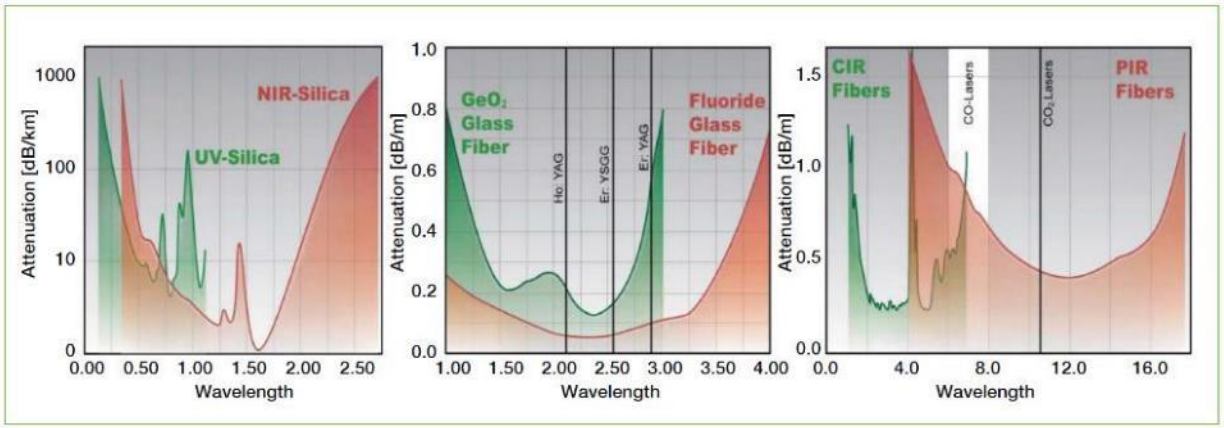
https://www.youtube.com/watch?v=PA1rMstzUJE

Fiber optic products for a broad spectral range with various fiber for NIR and MIR applications

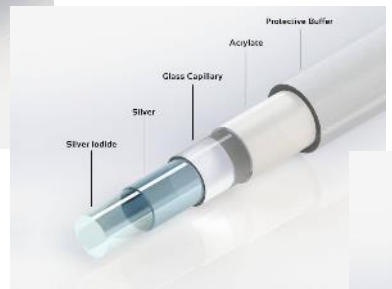


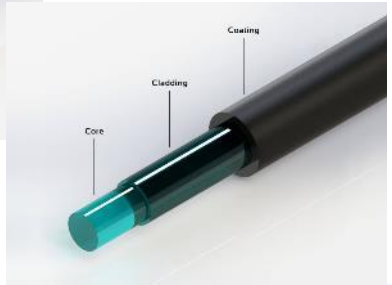






Complete cycle in-house production of Polycrystalline InfraRed fibers (PIR) is based on the patented technology.

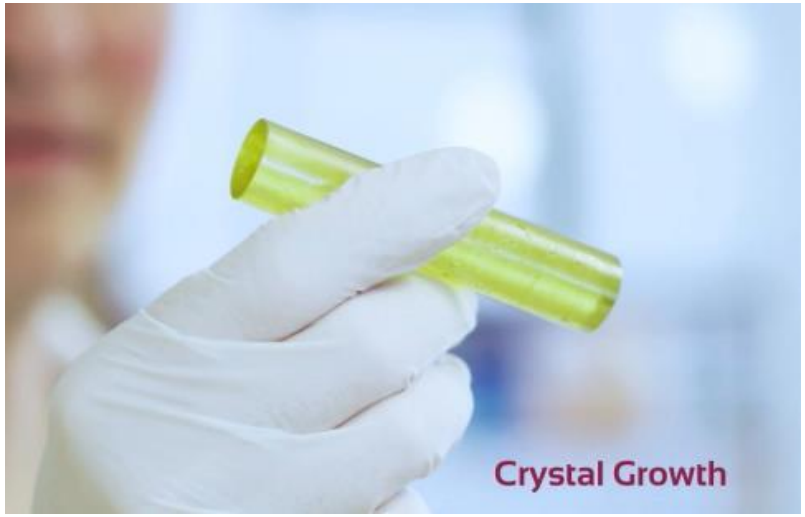




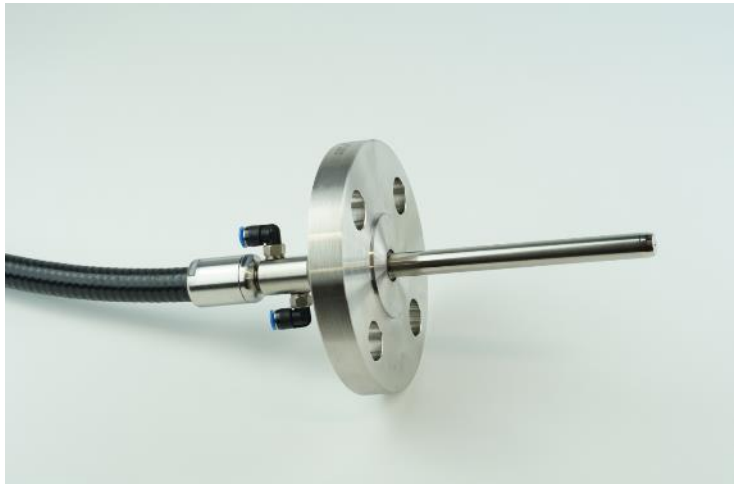
Alu- or Copper coating for Silica fibres to be used up to 350-600°C

Unique Polycrystalline IR-Fibers for 3-18µm

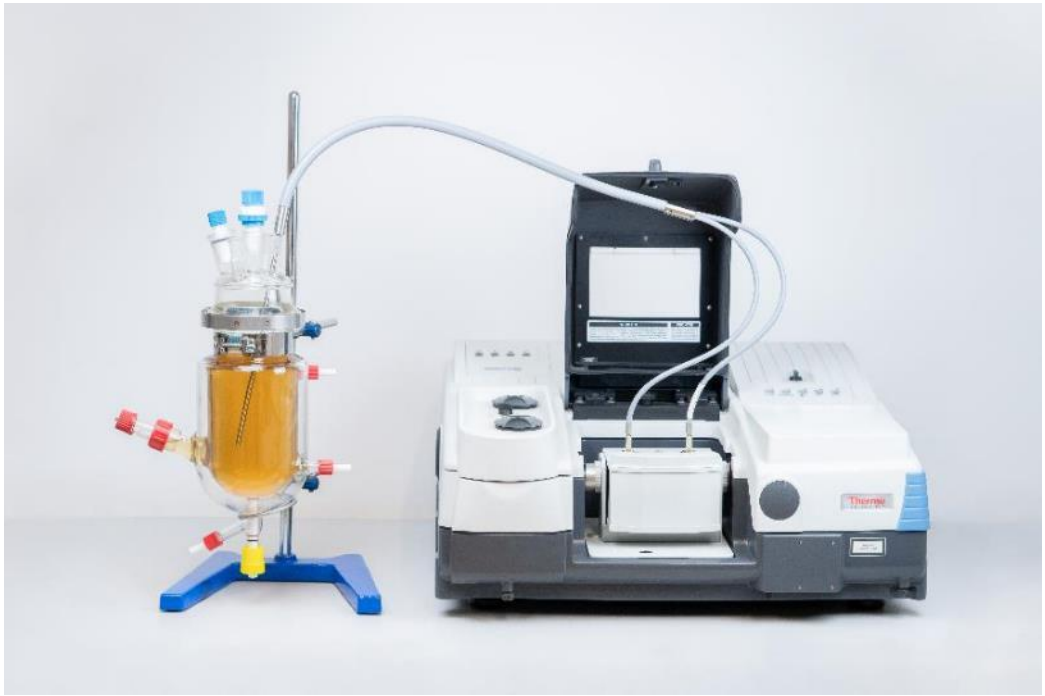
Complete production cycle

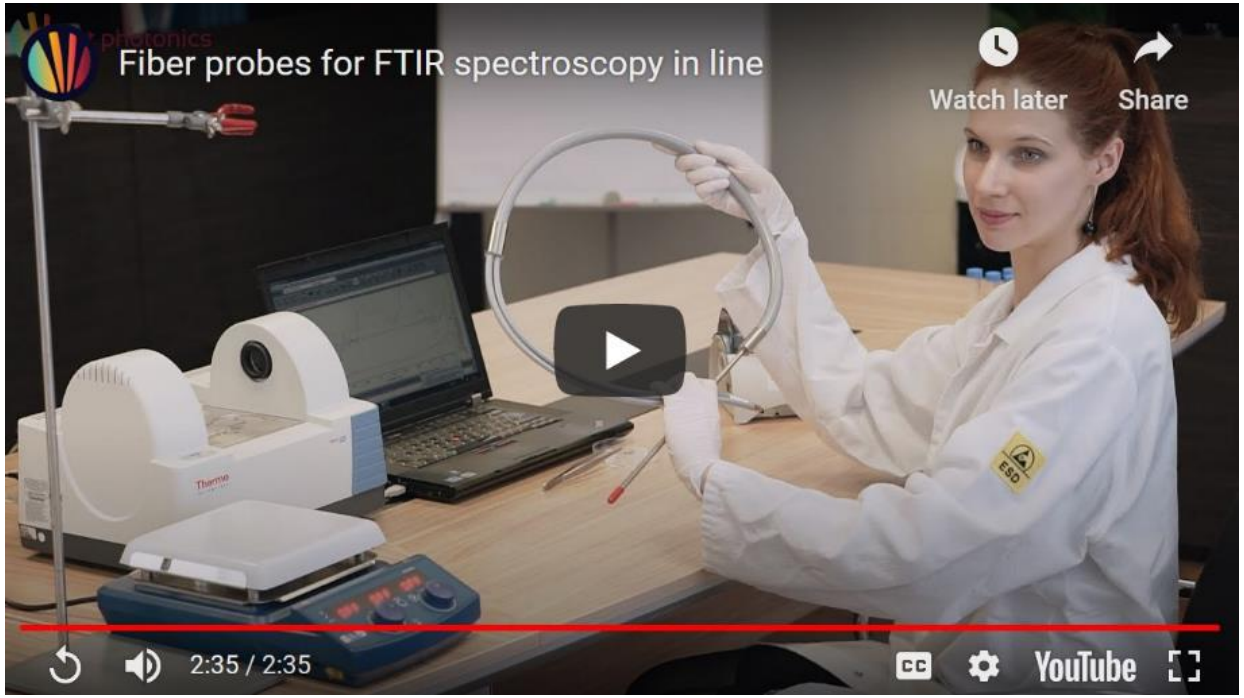


Fiber optic probes and couplers for *in-line* spectroscopy



FlexiSpec®-Duo: COUPLER + PROBE – enables to use bench FTIR for process-control in-line!





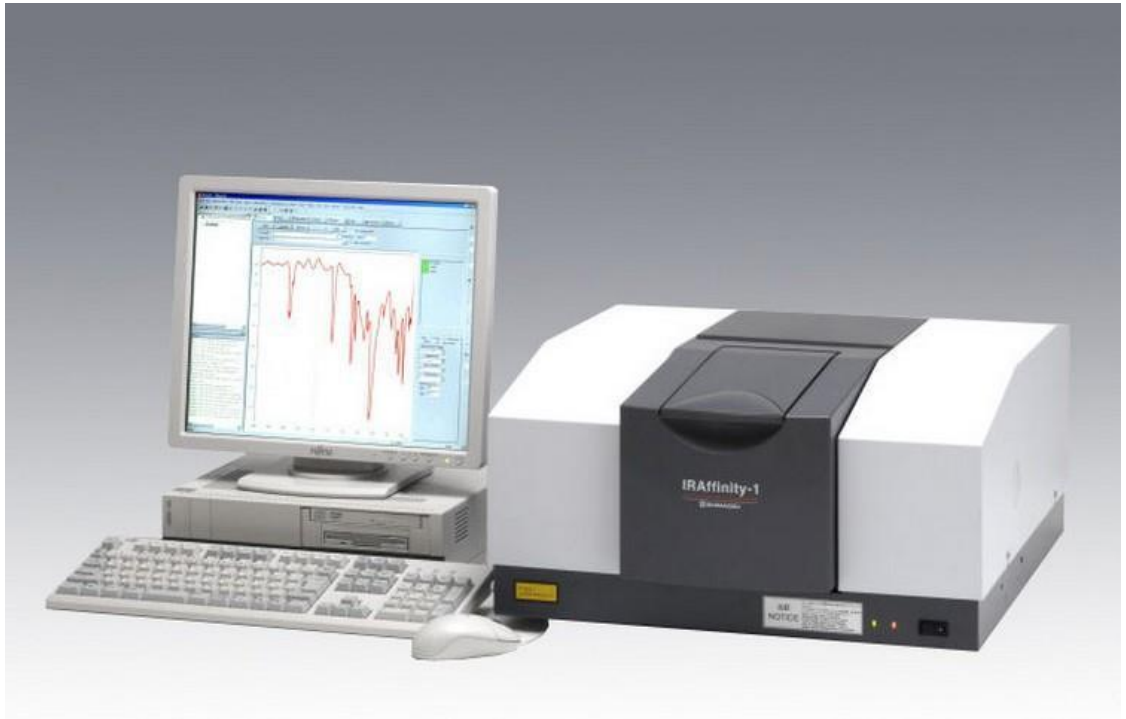
Reaction Initiation
Kinetics Determination
Reaction End-Point Determination
Identify Transient Intermediates

https://youtu.be/kfs9LByPEZg

In-line spectroscopy measurement versus sampling

Drawbacks of common spectroscopy tests for samples:

- **Lost of active components because of reaction with container material, atmosphere, etc.**
- **Change of sample composition during the storage or transporting**
- **Containment of a number of samples and utilizing after the measurement**



- **Storage and accounting of samples**
- **Maintenance of sampling documentation**
- **Human factor**
- **Delay in obtaining results**
- **Compilation and maintenance of measurement results for a number of samples**

Advantages of robust fiber probes for in-line spectroscopy

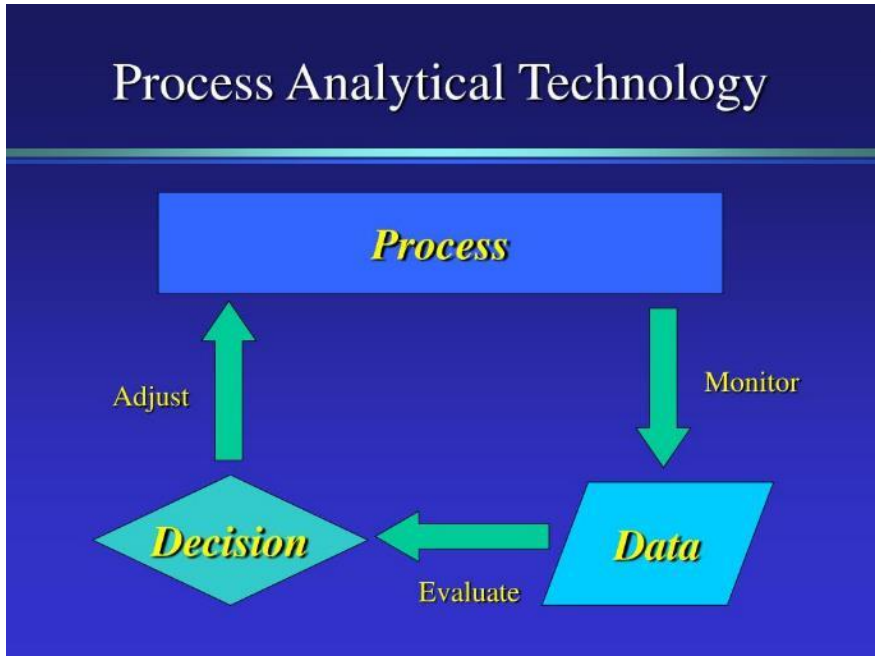
Remote sensing in real time with no sample preparation in lab and industry

- **for “hard to get to” samples (high temp, pressure, etc.)**
- **for air/moisture sensitive samples**
- **For hazardous samples (poisonous, corrosive, radioactive, etc.)**

In-situ real-time Reaction Monitoring at research, development, scale up and reaction optimization

- **Remote control of chemical reactions in-line**
- **Reduce production time**
- **Prevent rejection of batches**
- **Increase the yield & level of automation**
- **Save materials and energy**

Parameters to measure *in-line* in chemical processes



- **Process analytical technology** (**PAT**) = the measurement of critical process parameters and monitoring them in a timely manner thus improving the production process.

PAT goals:

- reduce production cycling time
- prevent rejection of batches
- enable real time release
- increase automation and control
- improve energy and material use
- facilitate continuous processing

PAT tools:

- in-line and on-line analytical instruments used to measure critical process parameters :
  - Spectroscopy - UV-Vis-NIR, Fluo, Raman, Mid-IR
  - Sensing – pH, humidity, conductivity, O_2, etc.
  - Turbidity – scattering
  - Chromatography
- multivariate data acquisition and data analysis tools
- knowledge management tools such as implementing and monitoring process improvement initiatives

Development, Specs and QC of spectroscopy probes

Meet requirements for industrial probes

- stability of characteristics over time
- high optical throughput and sensitivity
- operation in harsh process environment
  - temperature and pressure ranges,
  - chemical resistance
  - vibration, etc.
- different mounting options

<u>In particular:</u>

- ATEX standard
- 3A standard for food and drugs
- easy cleaning and calibration

Materials for building of fiber probes

- Probe immersible tip is made of Stainless steel or Hastelloy C22 to withstand the corrosive environment

- Robust PVC coated conduit protects fibers against overbending and break

- Sapphire and Diamond optical parts are stable in agressive chemicals

- Sealing with PTFE and Gold protects optics against the penetration of liquids under high pressure

Details of design to match the process demands

Process adapters like flanges,
Swagelok or InGold adapters

Standard A3 for food and drug

Multichannel probes

Sterilizable probes

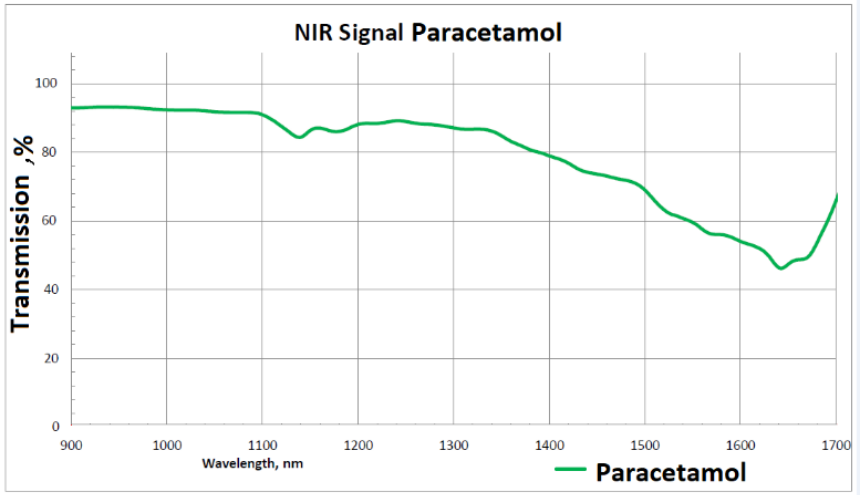
Polymer non-magnetic and
non-conductive design

Probes for extruders

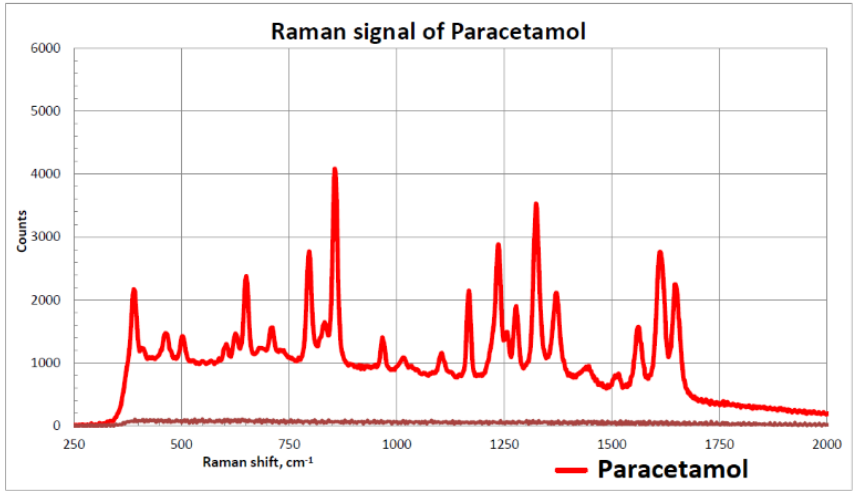
Air flow for cooling or heating
the probe interior

Side-looking Reflection Probe
for measurements of powder-fall

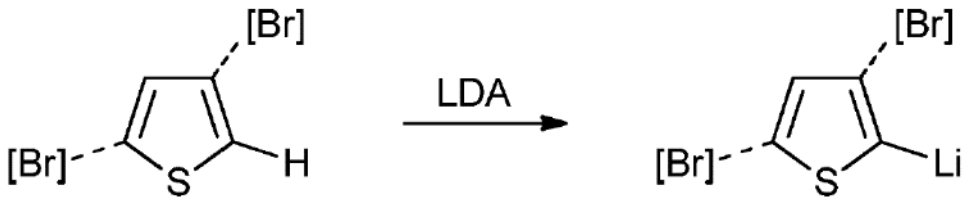
Development of multichannel probes Raman+ NIR

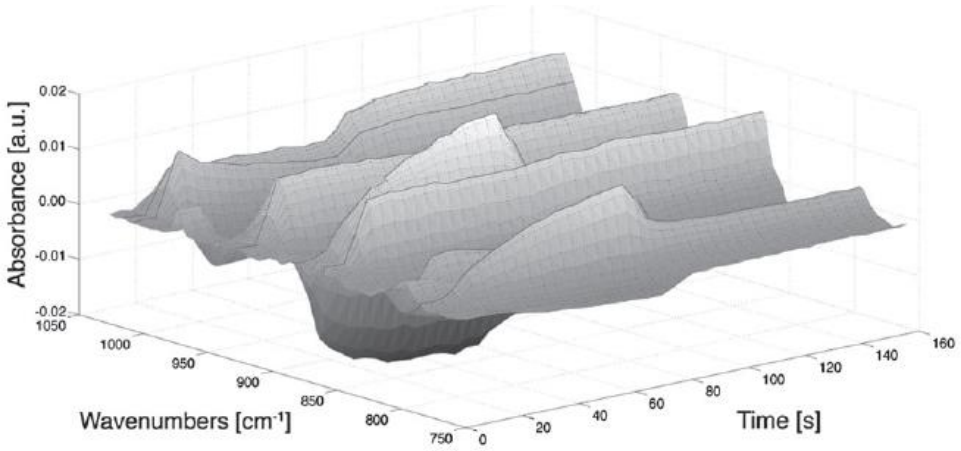


- Raman and Near IR diffuse reflectance channels (patent pending)
- Measurements of solids, powders or liquids.
- Data fusion from the simultaneous measurement for enhanced accuracy



Low Temperature Reactions of Lithiation





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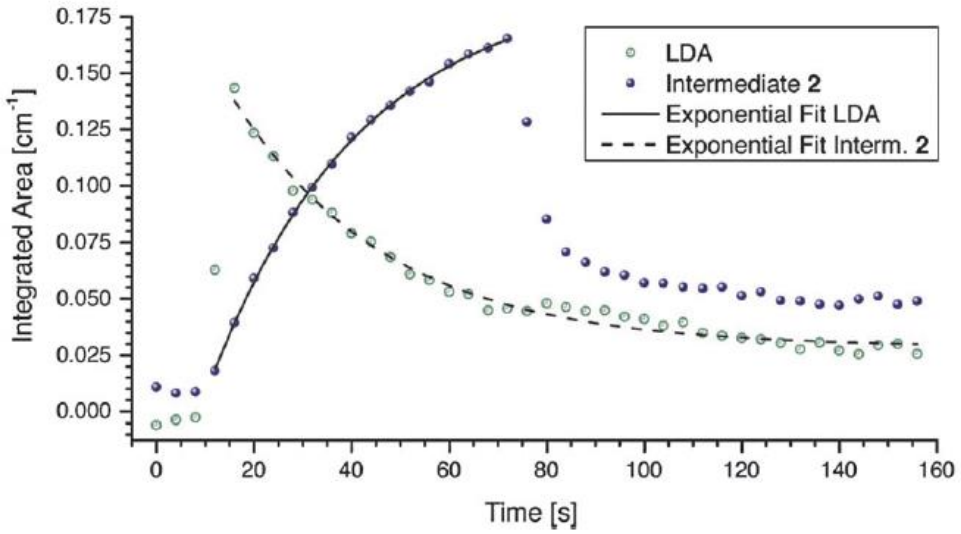
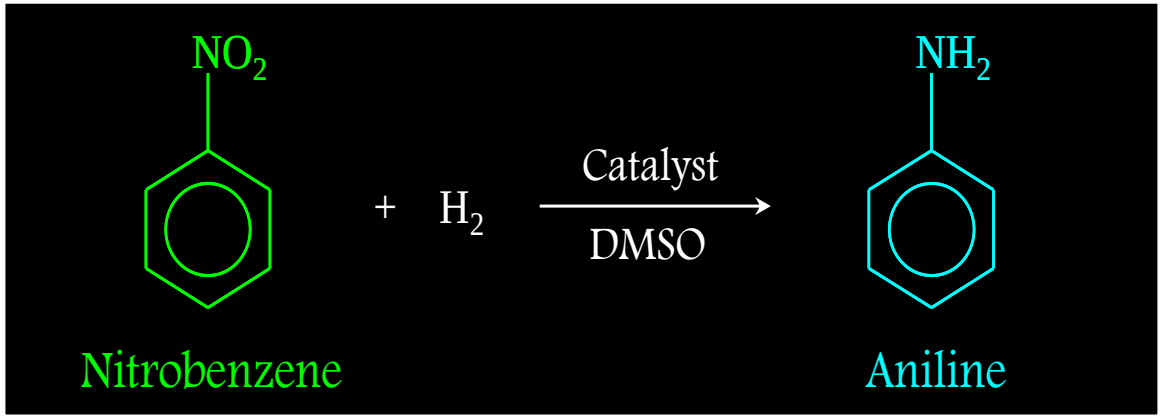


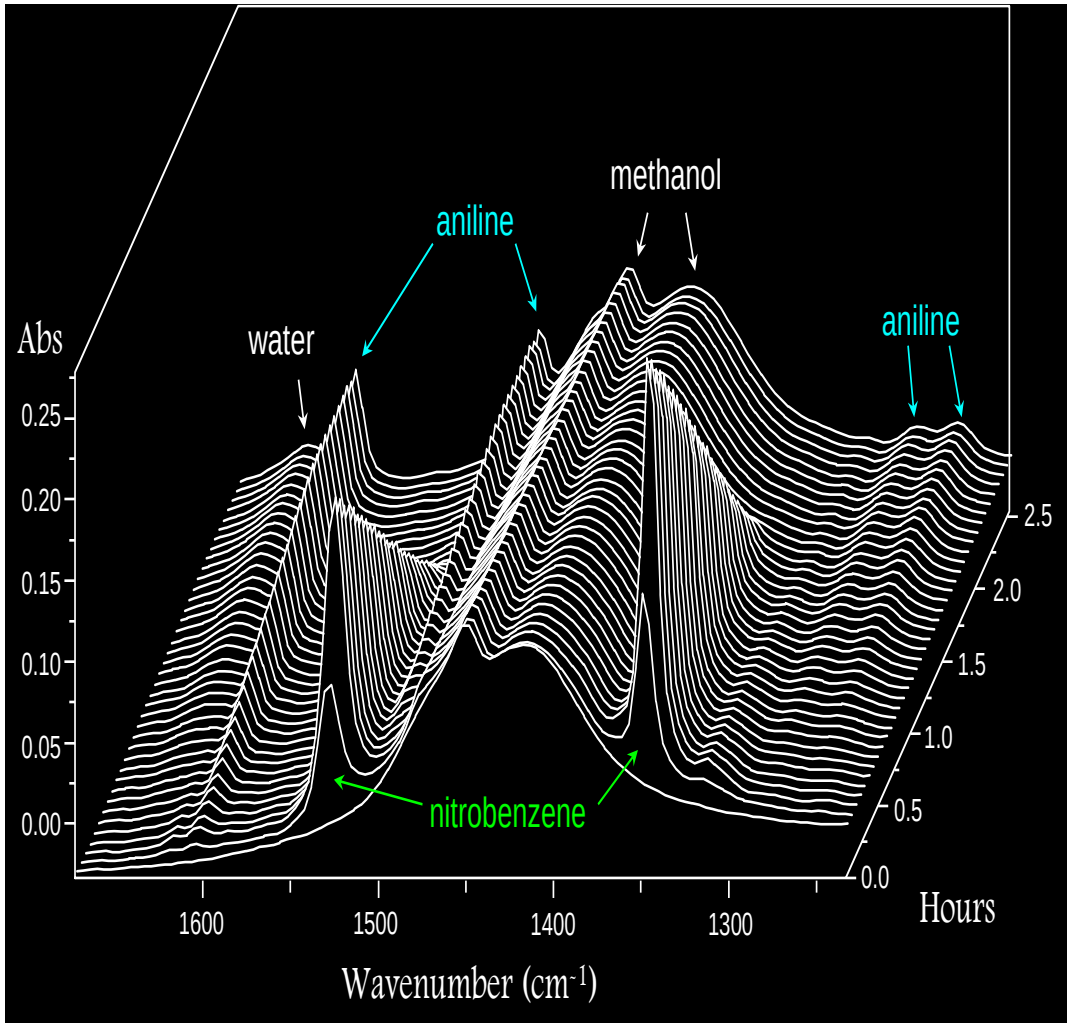
*Fibre optic ATR-IR spectroscopy at cryogenic temperatures: in-line reaction monitoring on organolithium compounds.
D. Lumpi etc. Chem. Commun., 2012, 48, 2451–2453 DOI: 10.1039/c2cc16016a

Reduction of Nitro to Amine



Monitoring Objectives

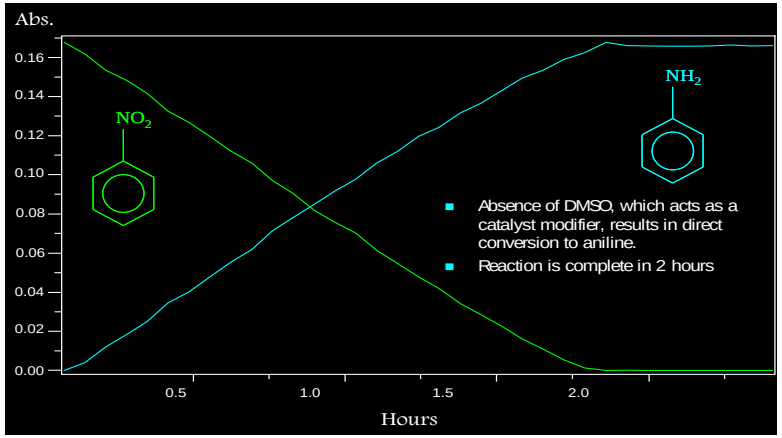
- Track nitrobenzene consumption and aniline formation
- Determine effect of modifying catalyst (DMSO) on reaction mechanism
- Define reaction end-point using aniline formation
- Eliminate oxygen contamination and hazards associated with grab sampling
- Reduce the dependency on time consuming and potentially hazardous “grab sample” analytical methods



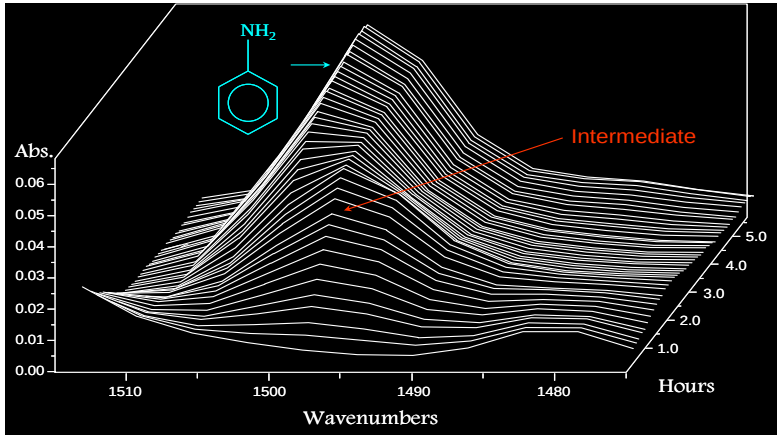
*Presented at The Cortona Conference, September 2010 Mettler-Toledo AutoChem

Reduction of Nitro to Amine

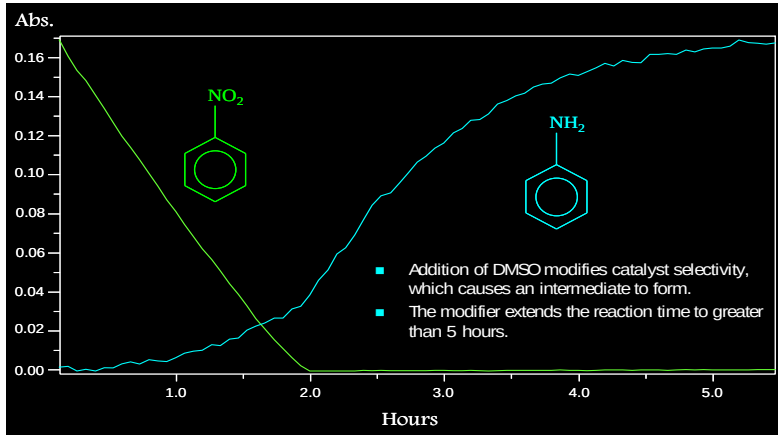
Infrared Profiles Indicate Direct Conversion to Aniline



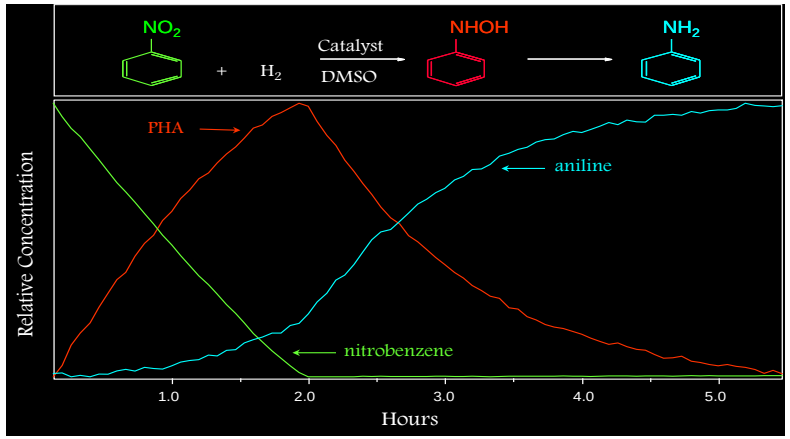
An Infrared Band for Aniline Reveals a Reaction Intermediate



Infrared Profiles Suggest a Intermediate or Side Products



Reactive Intermediate Phenylhydroxylamine



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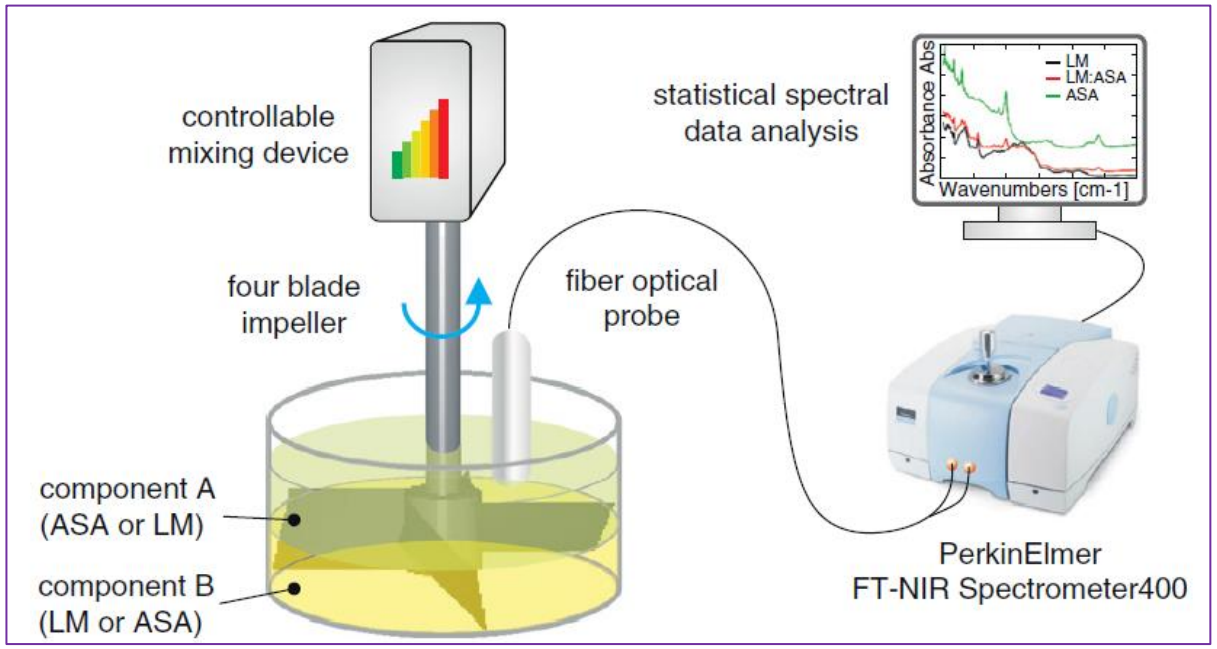
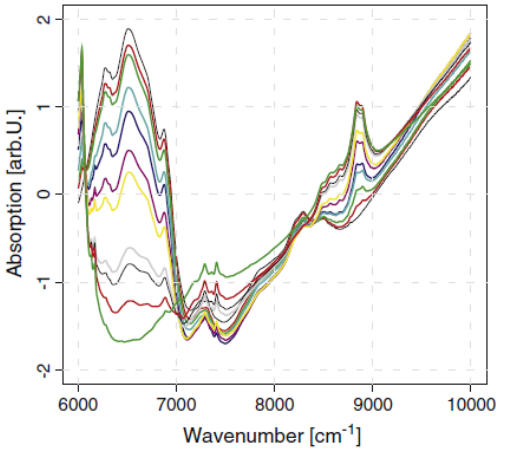
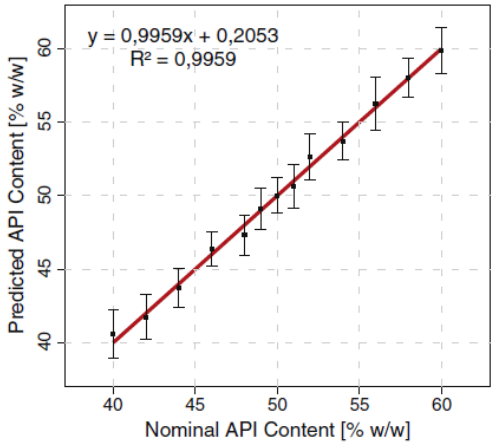
Blending process monitoring with NIR Reflection Probe

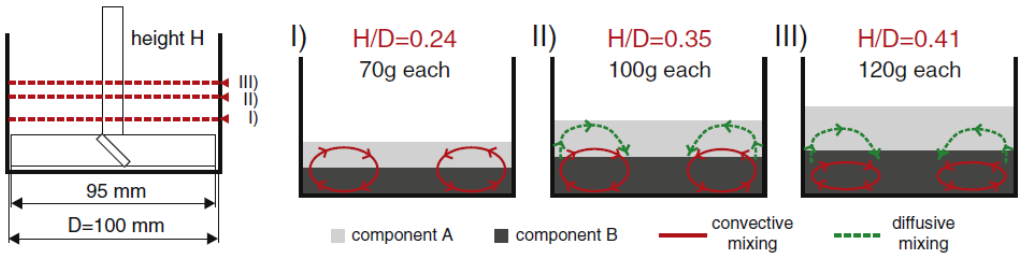




NIR spectra of the first calibration model (0–100%) after data processing with SNV and 4th order detrending.



Model performance plot for 40–60% API content



Schematic illustration of the mixer geometry with fill levels: I) H/D = 0.24, II) H/D = 0.35 and III) H/D = 0.41.

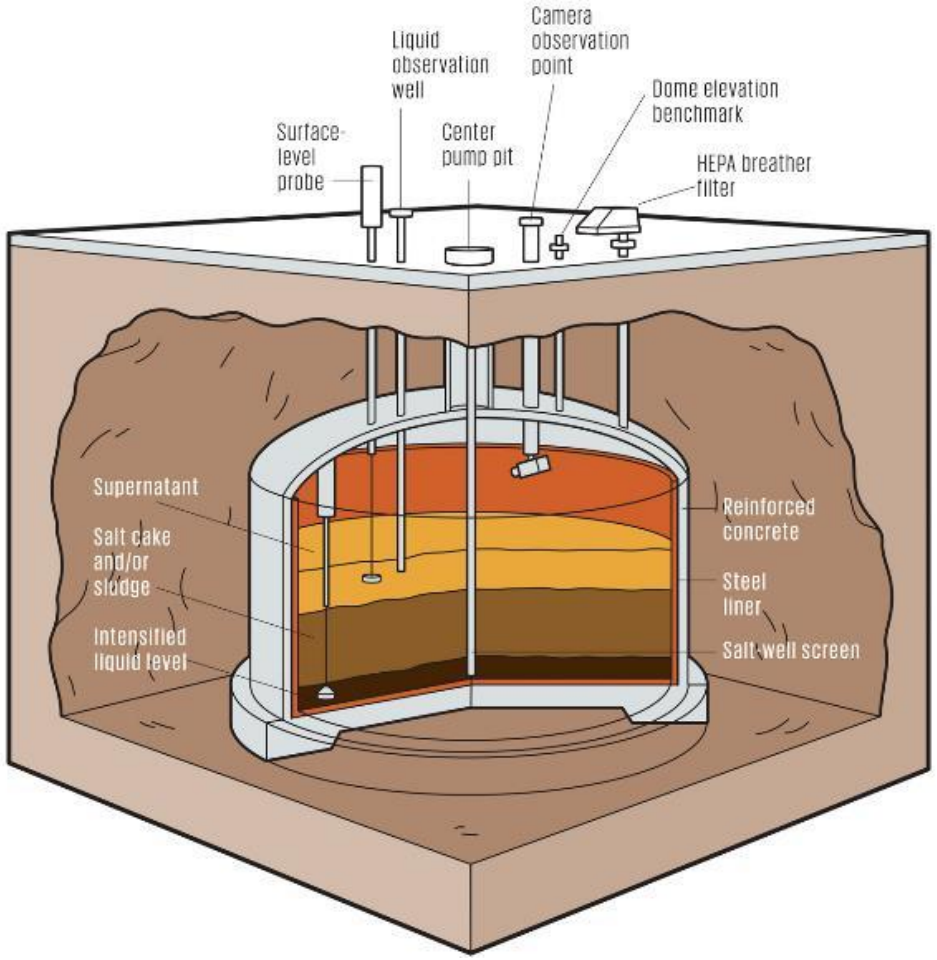
*Continuous quantitative monitoring of powder mixing dynamics by near-infrared spectroscopy.

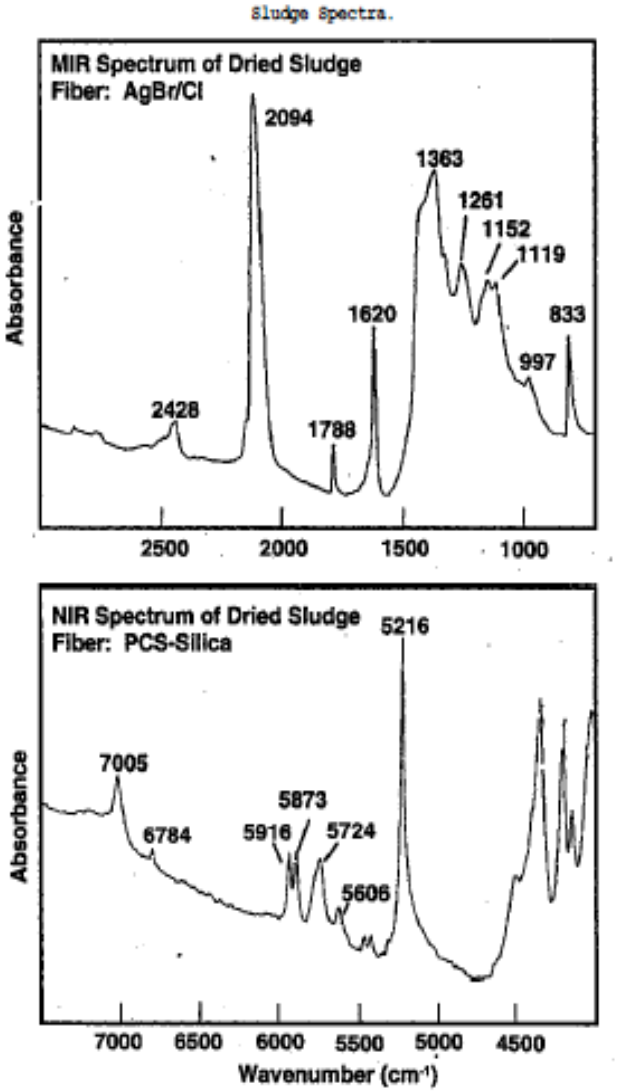
D.M. Koller, A. Posch, G. Hörl, C. Voura, S. Radl, N. Urbanetz, S.D. Fraser, W. Tritthart, F. Reiter, M. Schlingmann, J.G. Khinast

Powder Technology 205 (2011) 87–96

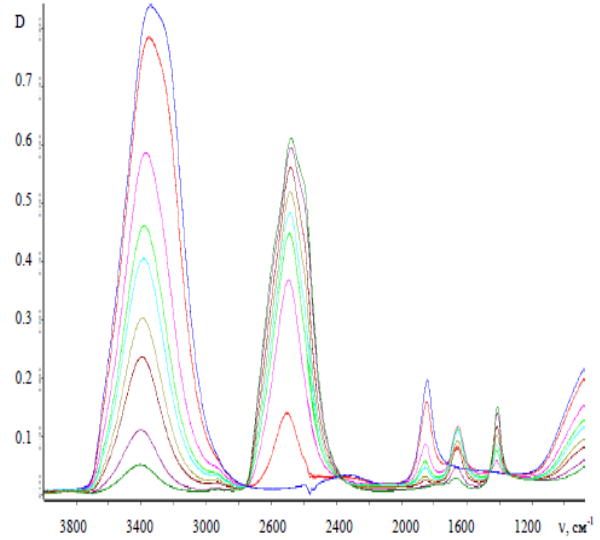
In-situ Characterization of Waste Sludge

Double-Shell Tank





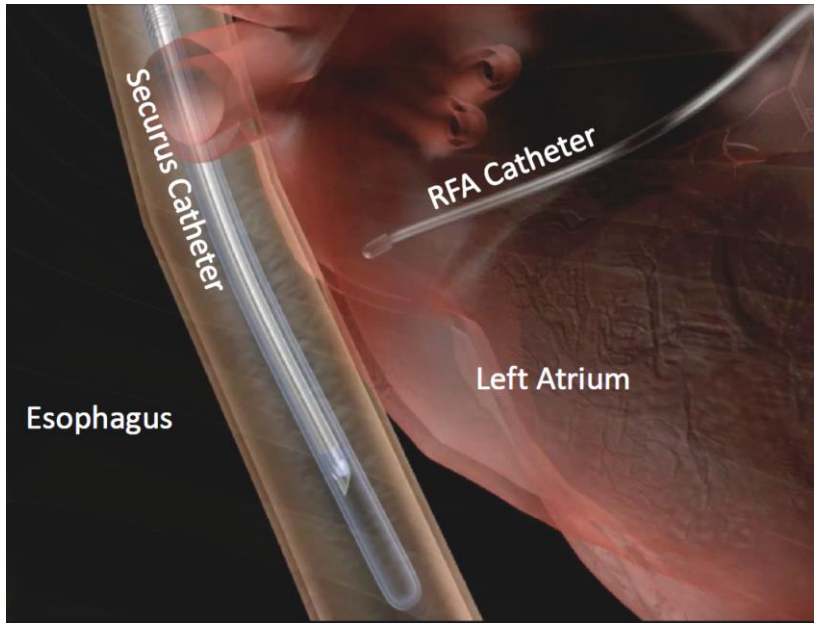
IR-attenuation of Heavy Water solutions in Water

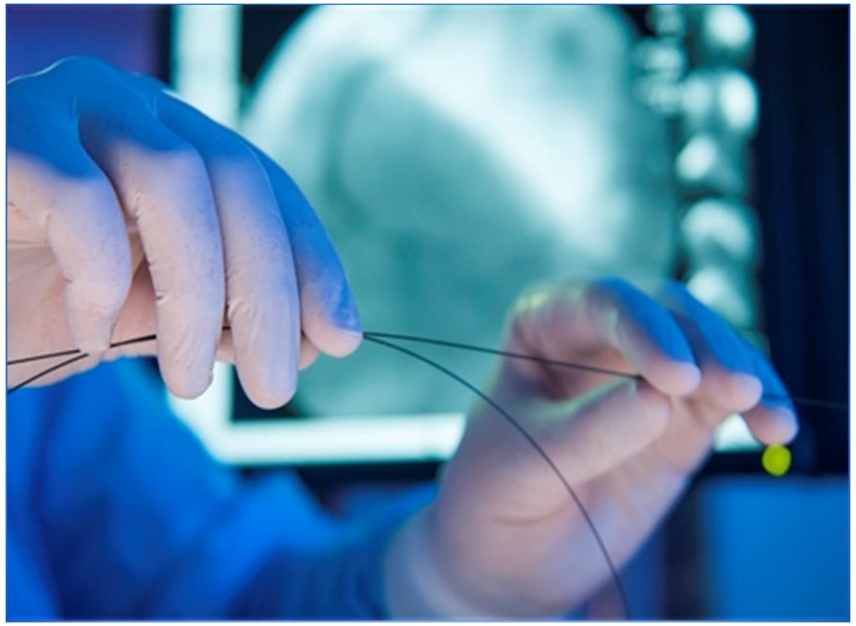


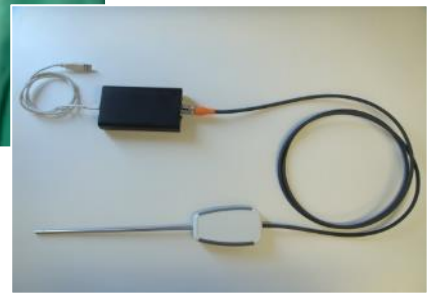
Constituent	Concentration, M
Na_2SO_4	0.23
Na_3PO_4	0.27
$NaNO_2$	1.5
$NaNO_3$	4.5
$CsNO_3$	0.00013

Note: No radionuclides

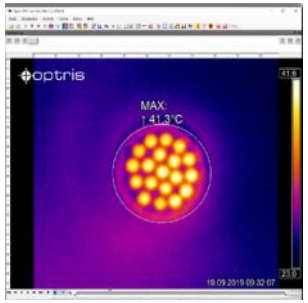
Fiber probes in medicine diagnostics



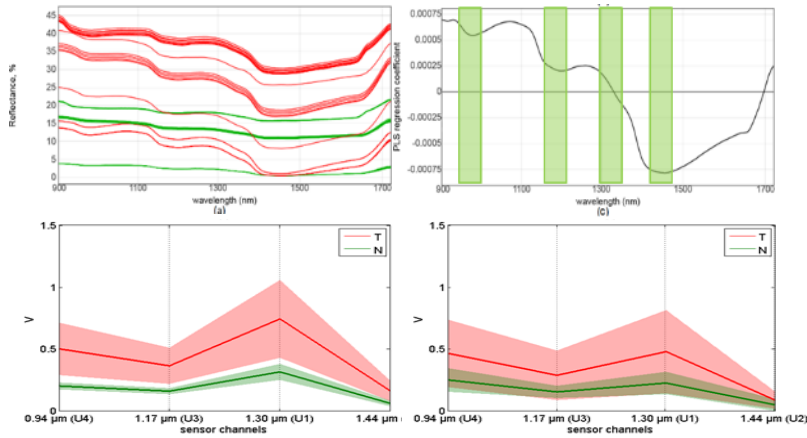




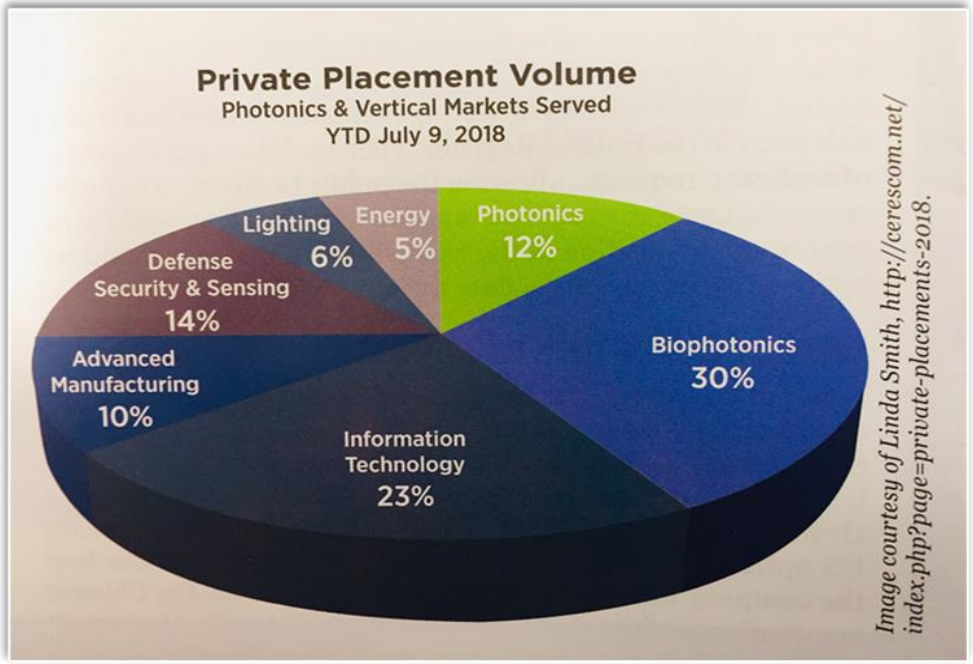
PIR-Fiber IR-imaging Endoscope



Tiny fiber probes enables spectral analysis of tissue inside human body in-vivo – vs common biopsy. Single fiber Raman probe with OD<200µm is used for it in 1s for 1cm depth!



Market for Photonics Sensors & Detectors ($ MILLIONS)



Application Type	2015	2016	2021	CAGR% 2016-2021
Military	2,709	3,051	5,694	13.3
Homeland security	980	1,126	2,279	15.1
Industrial process	739	861	1,868	16.8
Factory automation	623	730	1,635	17.5
Civil structure	645	740	1,498	15.1
Transportation	566	661	1,510	18
Biomedical	462	540	1,183	17
Microfluidics	412	483	1,084	17.5
Bio-environmental	260	309	737	19
Wind-energy turbines	226	269	648	19.2
Oil and gas	254	295	619	16
Others	174	204	445	16.9
TOTAL	8,050	9,269	19,200	15.7

- Spectral fiber sensors must be developed for customized applications in industrial process control – to be installed in critical points of reactor for low cost and in-line control of reagents composition. Sensors should transfer data by WiFi to iCloud for real time data treatment – to enable process control and its automatization matching the IoT & Industry4.0 concepts

Thank you for your attention !

https://www.youtube.com/watch?v=PA1rMstzUJE

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