Ústav fotoniky
a elektroniky

OPTICAL FIBERS FOR SENSORS
How to employ light for information harvesting?

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OUTLINE

- Optical fiber sensors - basic terms
- PCS fibers and ways for increasing the detection sensitivity
- Surface-plasmon fiber-optic sensors
- Gratings in optical fibers
- Fiber-optic tips and tapers

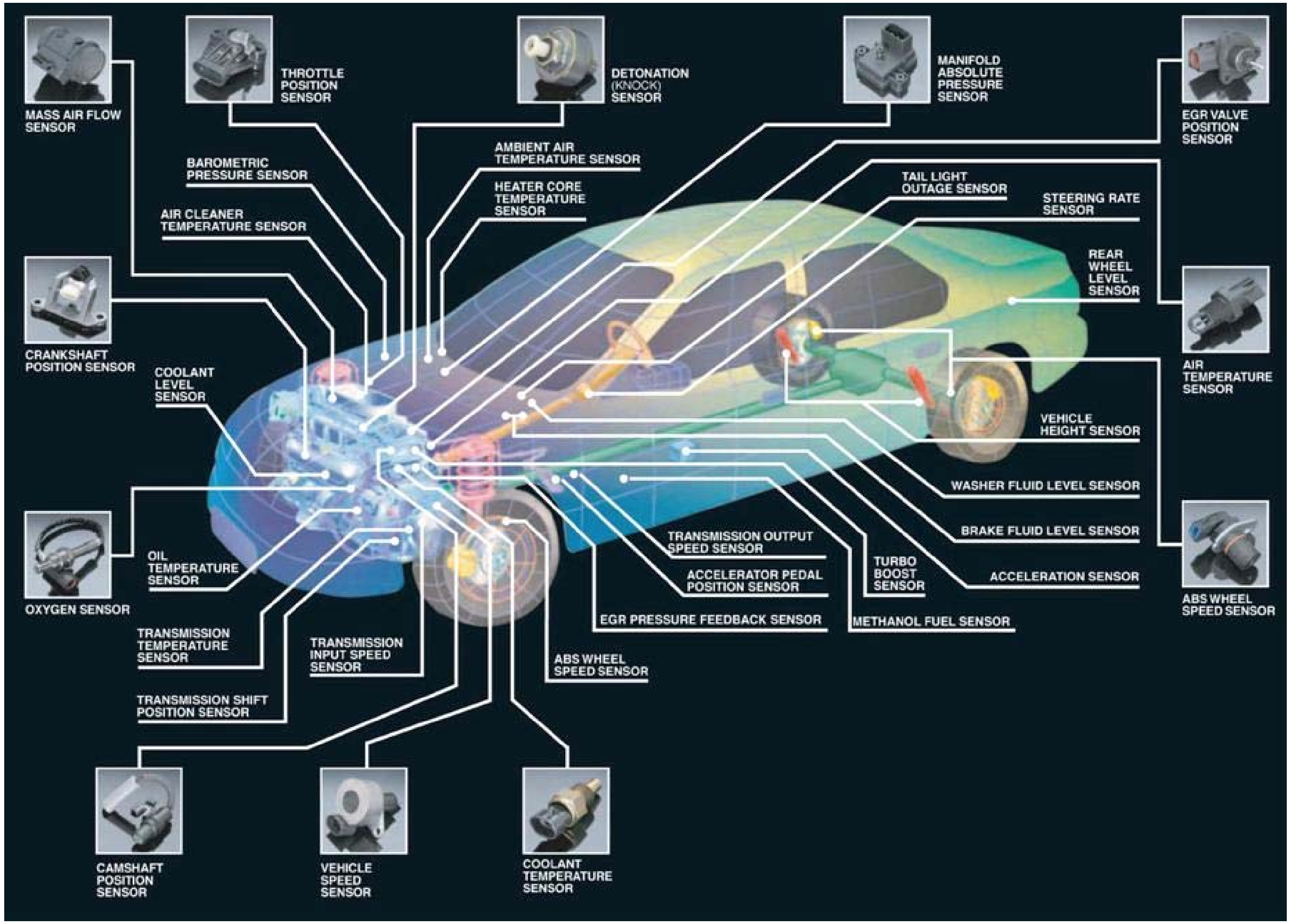
INTRODUCTION

Optical fiber sensitivity to surrounding changes

- Telecommunications – „in-sensitivity“ to changes in their surrounding (-40 – 50 °C)
- Sensors – controllable sensitive to changes in their surrounding

? Issues for development of special optical fibers sensitive (selectively) to surrounding changes

SENSORS IN AUTOMOBILE



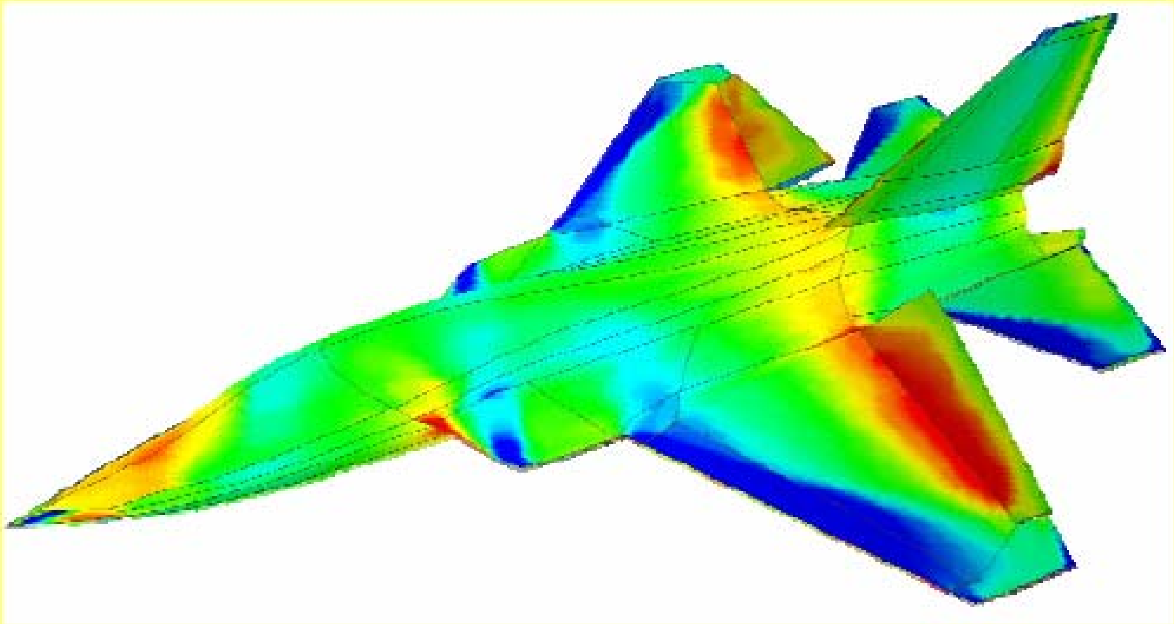
http://www.accurateautomotiveaz.com/index.php?option=com_content&view=article&id=84:com..

Detection of hydrogen leakages from hydrogen-based automobiles

SENSORS IN AIRPLANES

* Pressure–sensitive Paints (PSPs) and
* Temperature–Sensitive Paints (TSPs)

Used to "photograph" pressure (in fact oxygen) or temperature



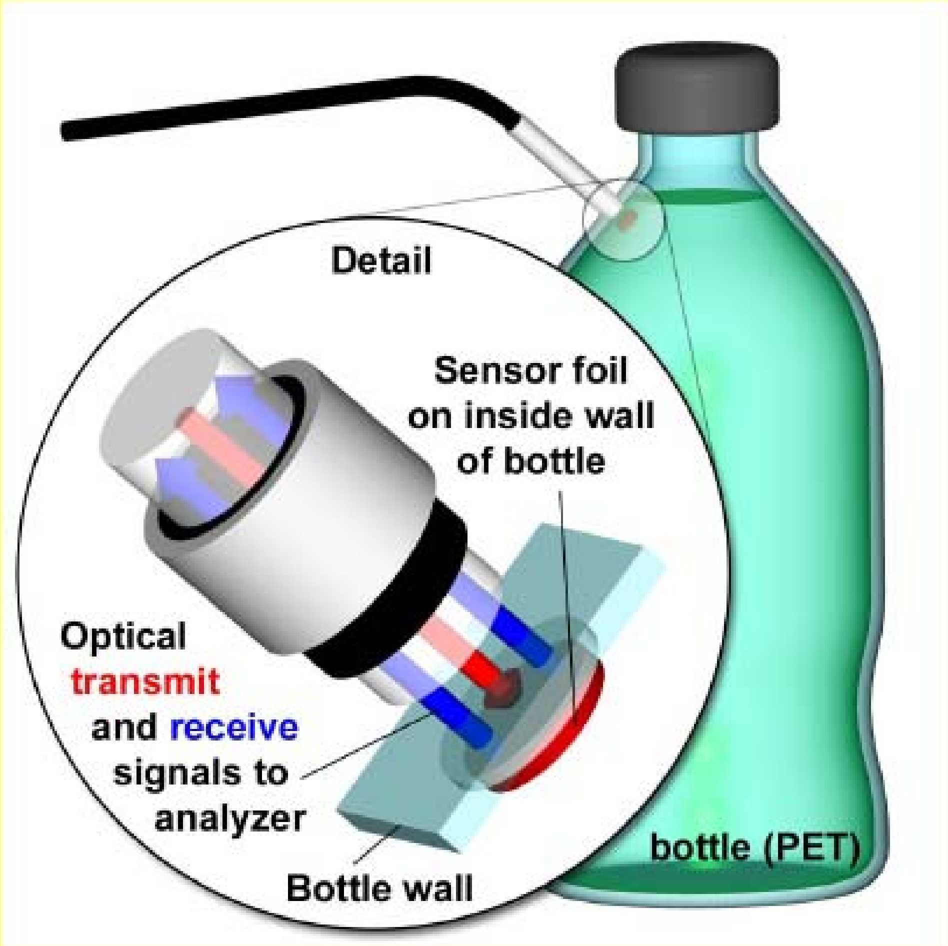
Book: T. Liu, J. P. Sullivan, *Pressure and Temperature Sensitive Paints*, 2005, Springer.

SENSORS IN FOOD PRODUCTION

PET is permeable to oxygen and CO_2

* CO_2 diffuses out
* O_2 diffuses in

Sensor Spots Placed Inside the Bottle



O.S. Wolfbeis, EUROPTRODE X, 2010, Prague

SENSORS IN PHARMACY

Product completeness **Product content**

http://www.keyence.com/products/vision/vision-sensor/iv/applications/application-02.jsp

WHAT IS OPTICAL SENSOR?

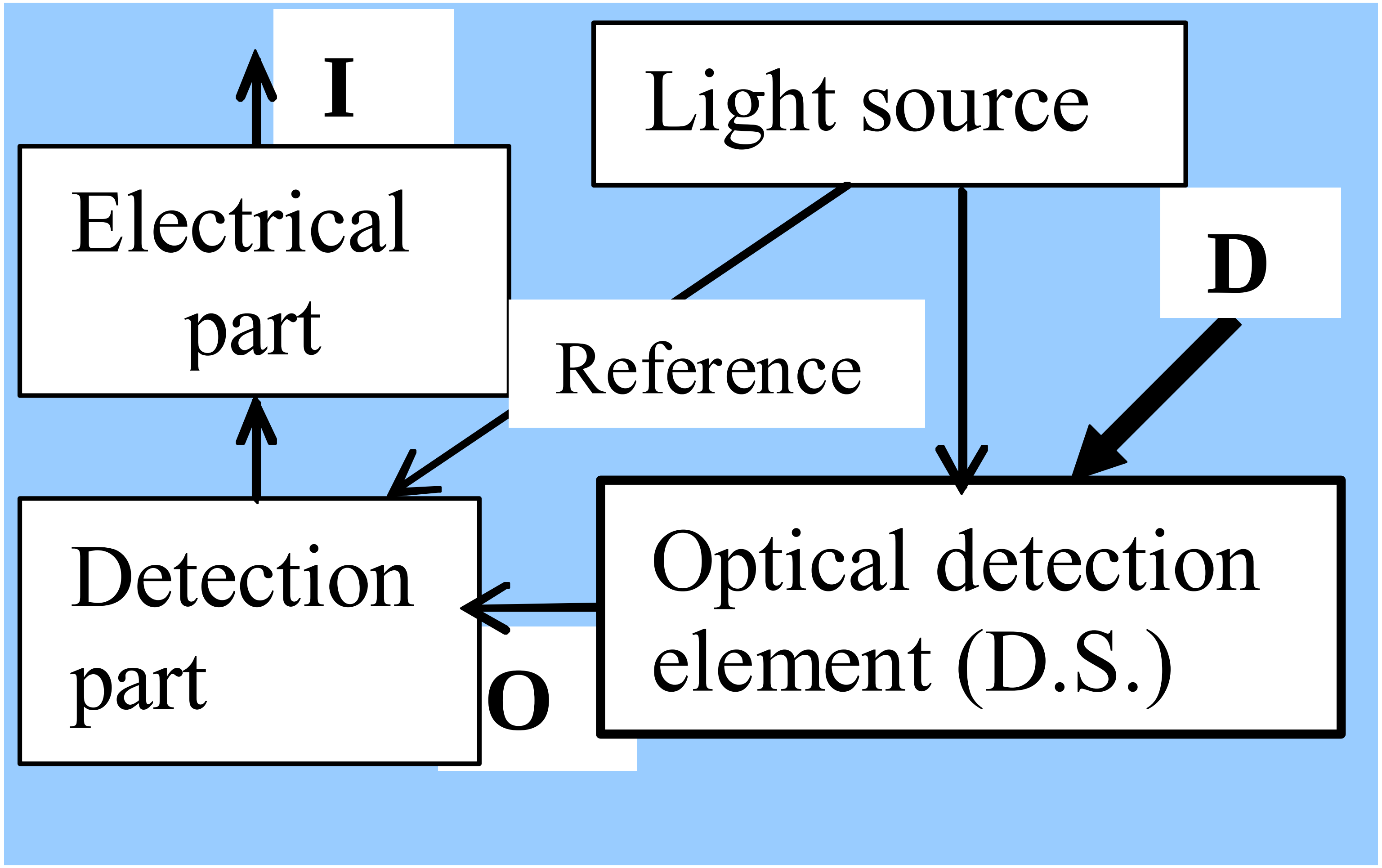
Def:

a small device for continuous and reversible determination of qualitative and quantitative changes

physical variables (temperature, pressure, etc.), chemical composition in its vicinity by employing light changes

(amplitude, phase, frequency, polarisation).

SET-UP OF OPTICAL SENSOR



D.S. – Detection site

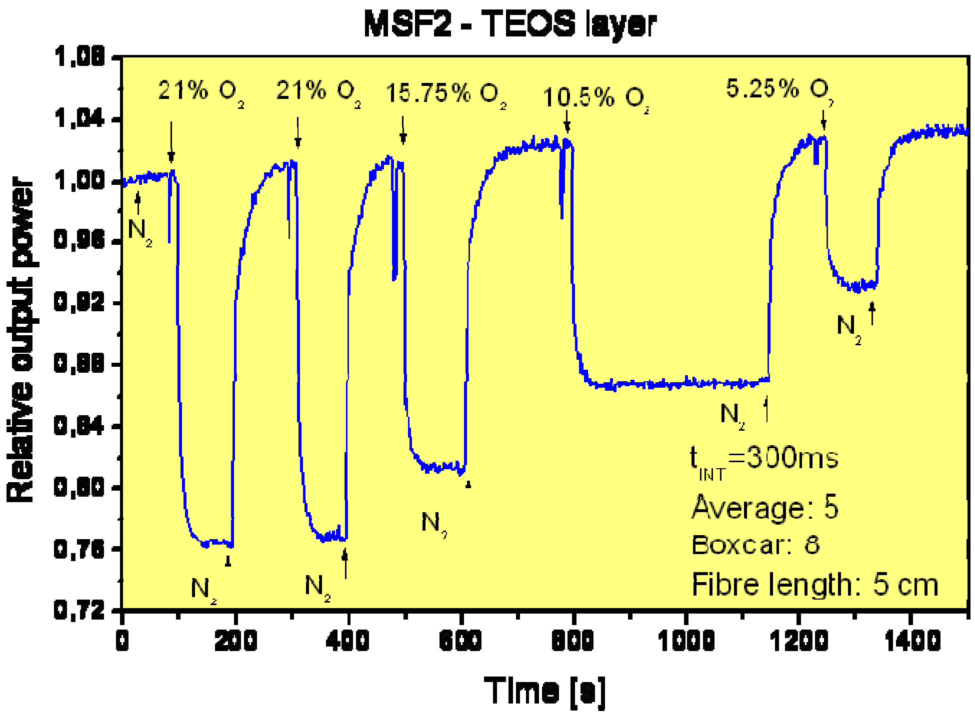
OPTICAL SENSOR

Obligatory parts of optical sensors

- Light source (halogen lamp, LED, LD)
- Detection part (waveguide, foil, prism, …)
- Detector (photodiode, photon counter, spectrometer)
- Optical signal treatment

All these parts are integrated in one systems

TIME-RESPONSE OF SENSOR



$$I = I_s\left(1 - \exp\left(\frac{-t}{\tau}\right)\right)$$

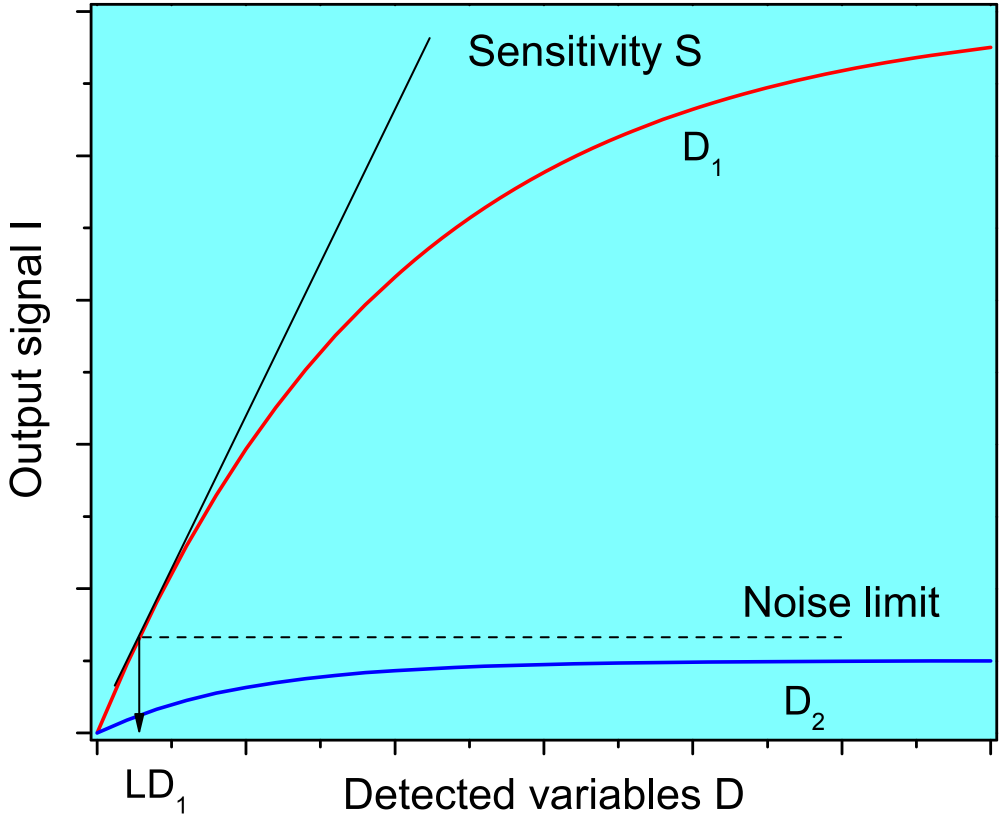
Oxygen detection by quenching fluorescence of Ru complex

I_s saturated value-

τ- time constant (for $I=0.63\ I_s$)

τ Range: s - min

SENSOR SENSITIVITY- CALIBRATION CURVE



Variable D_1 detected in an extent LD_1 →

Variable D_2 can' t be detected

SENSITIVITY AND SELECTIVITY

$$S_i = \left(\frac{\partial O}{\partial D_i}\right)\left(\frac{\partial I}{\partial O}\right)$$

Selectivity

S is high (for the detected variable D)

I< Noise limit or S $\rightarrow$0 (the other variables in the detection site)

OPTICAL CHEMICAL SENSORS

- D: Qualitative and quantitative detection of chemical substances, i.e. their presence and concentrations in detection site
- In detection site light interact with analyte and its characteristics are changed
- O: Light wave characteristics (**amplitude**, frequency, polarisation) employed for detection of D

WHAT VARIABLES ARE DETECTED?

Analyte comes to the detection site and change

Refractive index

Gases – n ~ 1

Water - n ~ 1,33; Ethanol - n ~ 1,37

Silicone polymers, fluorinated acrylates n ~ 1,4

Silica glass – n ~ 1,46

Toluene - n ~ 1,5

PMMA - n ~ 1,49, PVC – n ~ 1,54-1,56

Optical glass F2 – n ~ 1,51

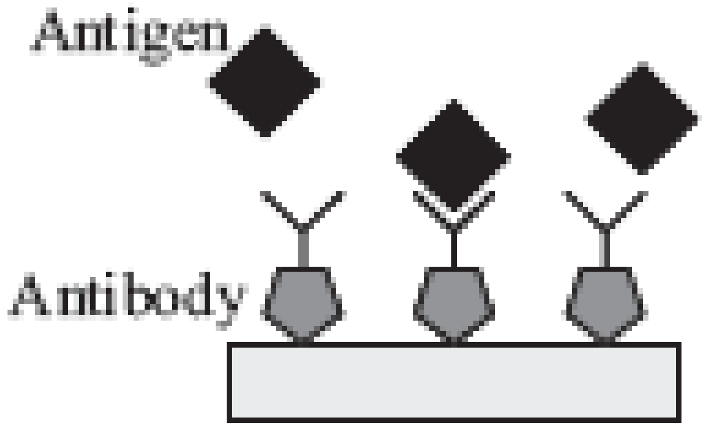
Histidine – n=1,7

REFRACTIVE-INDEX CHANGES

Refractive-index changes in the detection site are **nonspecific** **⇒ <u>transducer is necessary</u> that accept only detected substance (analyte)**

„label free“ imunosenzors interaction of antibody with antigene

Increased density in antibody chain → ***<u>n increase</u>***



ABSORPTION COEFFICIENT

Detected substance – analyte changes absorption coefficient

NIR spectral bands

(CH~1600-1700 nm, NH ~ 1500 nm, OH ~ 1400 nm
overtones of fundamental IR frequencies 2900-3600 cm^{-1})

Modified Lambert-Beer law absorbance A

UV spectral bands (250 nm) of aromatic compounds

$$A = \gamma L \sum_{i=1}^{N} \varepsilon_i c_i$$

γ < 0.01 if standard fibers without any change are used

Direct detection of cells with fiber tips or UV spectra of antibodies

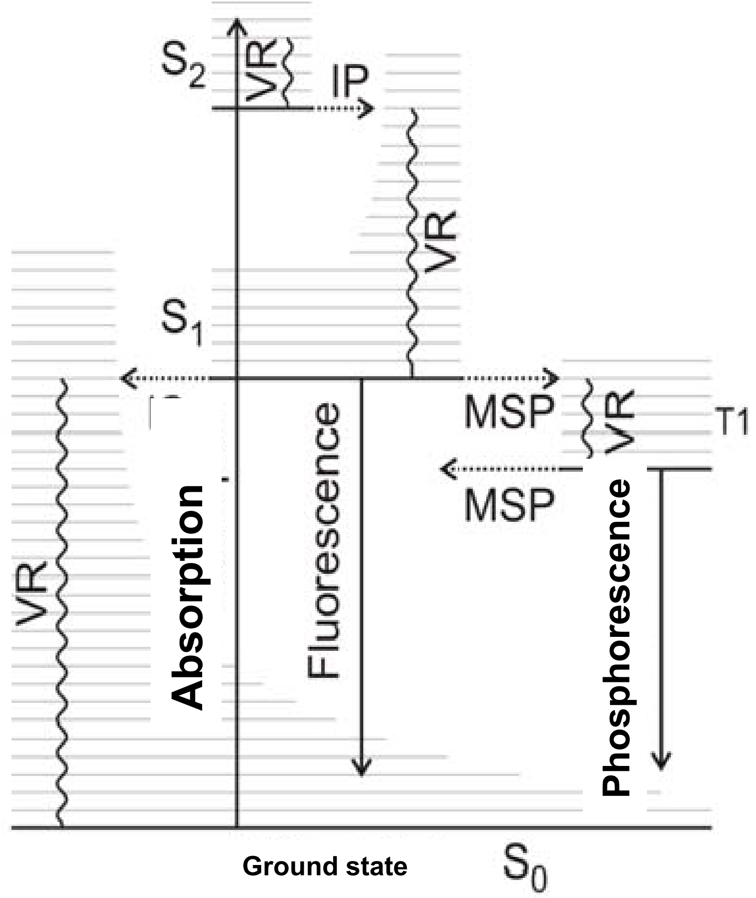
See e.g.:A. Leung et al. Sensors and Actuators B 125 (2007) 688–703

LUMINESCENCE

Luminescence =

Fluorescence or

Phosphorescence



IP	**Internal transition**	S_i --> T_j	**Nonradiative transition**
MSP	**Intersystem transition**	S_i --> S_j	**Radiative transition**
VR	**Vibration relaxation**		

LUMINESCENCE

<u>Fluorescence</u>: allowed energetical transition without spin change, rapid (lifetime ~ μs –ns)
<u>Phosphorescence:</u> spin change, not allowed, slow (lifetime ~ s – ms)

<u>Luminescence effects are related to:</u>

Aromatic rings, conjugated double bonds, Ru complexes

LUMINESCENCE INTENSITY

$$P \approx P_0 \varepsilon(\lambda) QcL$$

$\varepsilon(\lambda)$ – absorption coefficient ~ 10^5 l/mol/cm Q –quantum efficiency, c – concentration of luminescent analyte (luminofor)

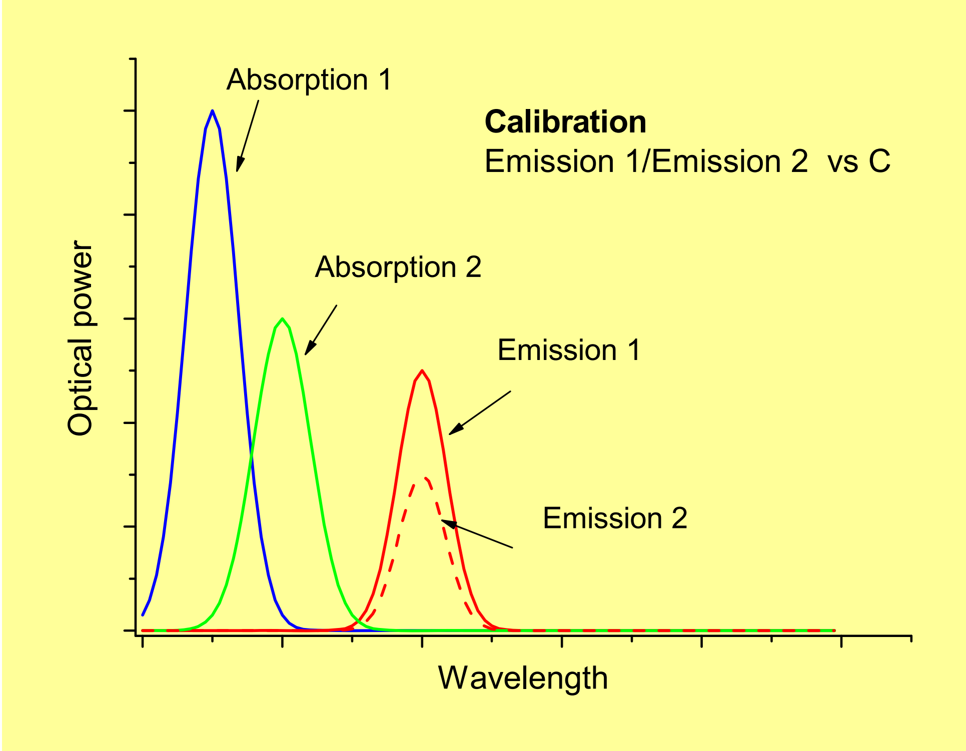
Excitation way: electroluminescence, photoluminescence, bioluminescence, chemoluminescence

See e.g.: C.L. Morgan et al. Clinical Chemistry 42, No. 2, 1996

REFERENCE METHODS

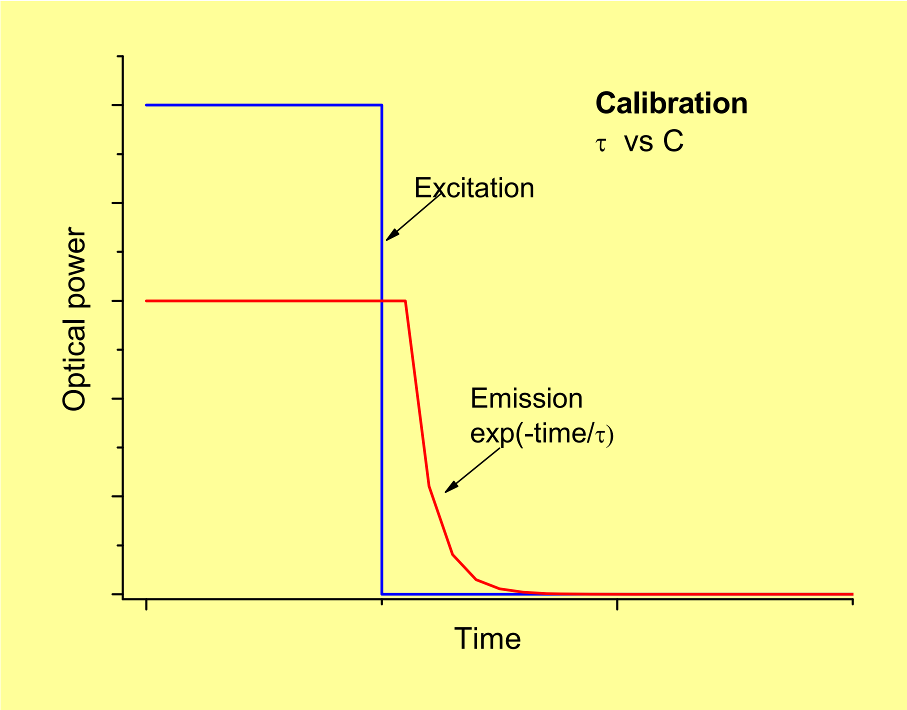
Luminescent intenzity P suffers from random fluctuations → Reference methods:

1. Excitation at two absorption wavelength, detection at one detection wavelength



LUMINESCENCE LIFETIME

2. Luminescence lifetime τ is practically independent of random fluctuations. Some analytes (oxygen, carbon dioxide) induce fluorescence quenching.



Stern-Volmer equation

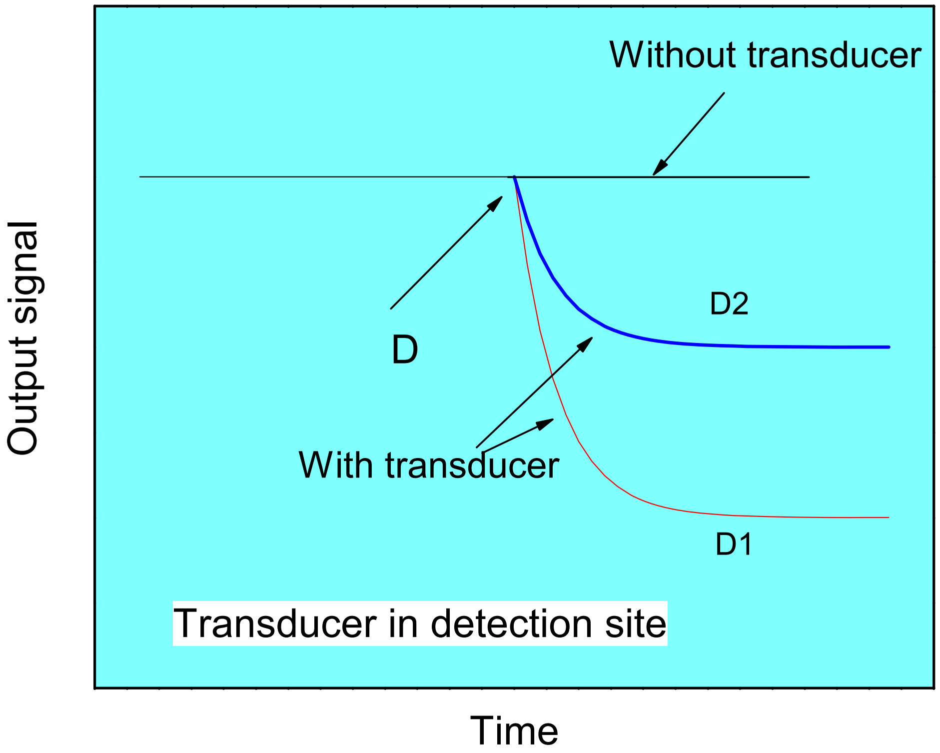
$$\frac{\tau_0}{\tau} = 1 + K_{SV}[Q]$$

Q j- quencher concentration

http://www.jh-inst.cas.cz/~fluorescence/support/Lectures/UFCH_fluor04.pps

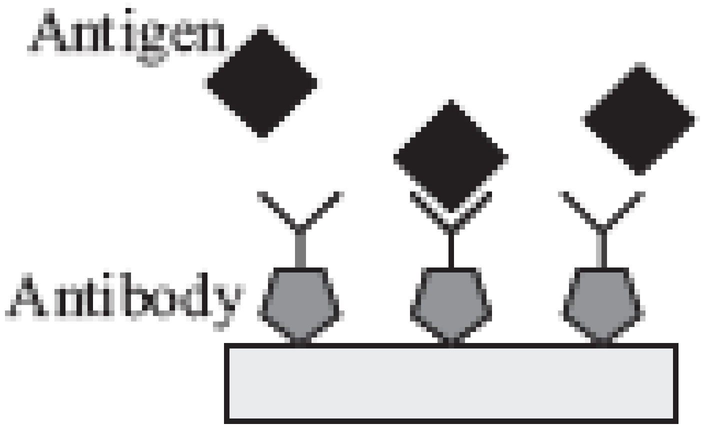
OPTICAL TRANSDUCERS

If analyte has no optical absorption, luminescence,
only it changes refractive index → **transducer** has to be added that changes its optical properties due to interaction with analyte



REFRACTIVE INDEX - IMMUNOTRANSDUCERS

Based on immunoreaction (affinity reaction) at which antibody (protein) specifically capture analyte (antigen) → refractive-index change



Gamaglobulins Ig
5 types – G,M,A,E,D

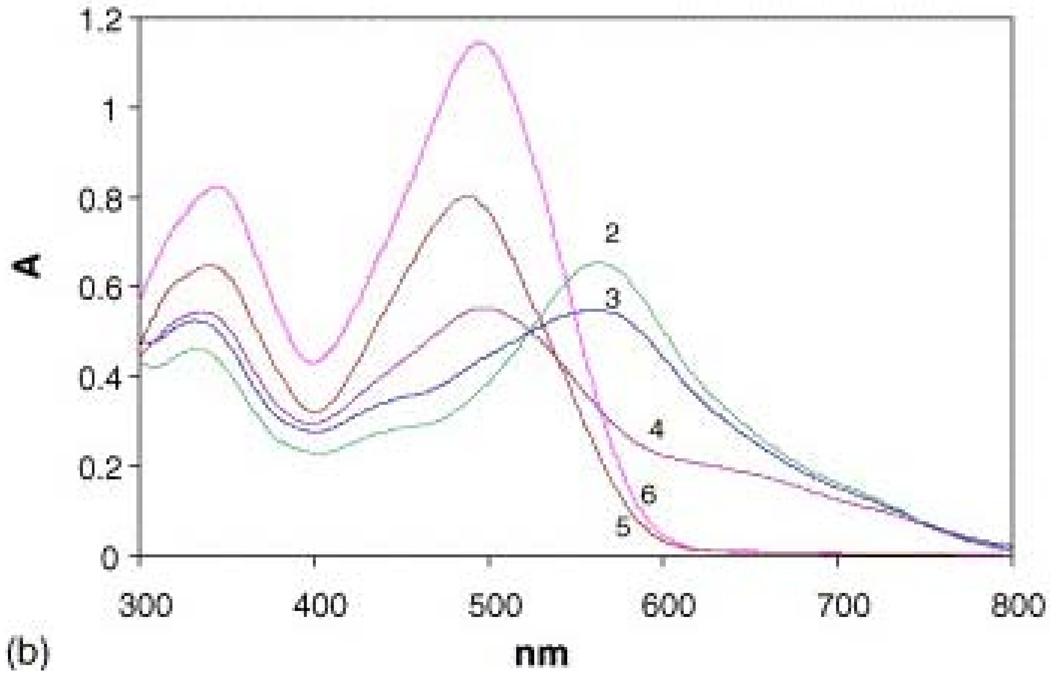
Usually IgG

See e.g.: D.W.G. Morrison et al., „Clinical application of micro- and nanoscale biosensors“, Biomedical Nanostructures, Ed. K.E. Gonsalves,C.L. Laurencin, C.R.Halberstadt, L.S. Nair, 433-453, Ch. 17, 2008

ABSORPTION TRANSDUCERS

Absorption coefficient

pH indicators: **$BInd + H_3O^+ \rightarrow AInd + H_2O$**



below pH 3.0 *above pH 5.2*

3.0 ↔ **5.2**

Absorption spectra of „Congo red“ for different pH

P. Hashemi et al., Sens. Act. B115, 49-53 (2006)

ENZYMATIC TRANSDUCERS

Enzymes = biocatalysts

<u>Enzymatic reaction</u>

Substrate (analyte) $\rightarrow^{enzym}$ Product

Analytes = glucose, pesticides, urine

Glucose + oxygen $\rightarrow^{glucoseoxydase}$ Gluconic acid + hydrogen peroxide

Product can be detected by luminescence or absorption spectroscopy or other transducer is added (Ru complex for detection of oxygen, pH indicator for detection of gluconic acid)

DETECTION OF ORGANOPHOSHOROUS COMPOUNDS

Enzymatic transducer + pH transducer

Cholinesterase + methyl red

Acetylcholine + water→ cholin + acetic acid

Reaction takes part at nerve end and enables registration of external changes. <u>Organophosphorous compounds stop the reaction of acetylcholine (inhibition).</u>
Detection of nervous paralytic welfares (Sarin, Soman).
Inhibition of cholinesterase ⇒ no pH change.

Army detector CHP71

LUMINESCENT TRANSDUCERS

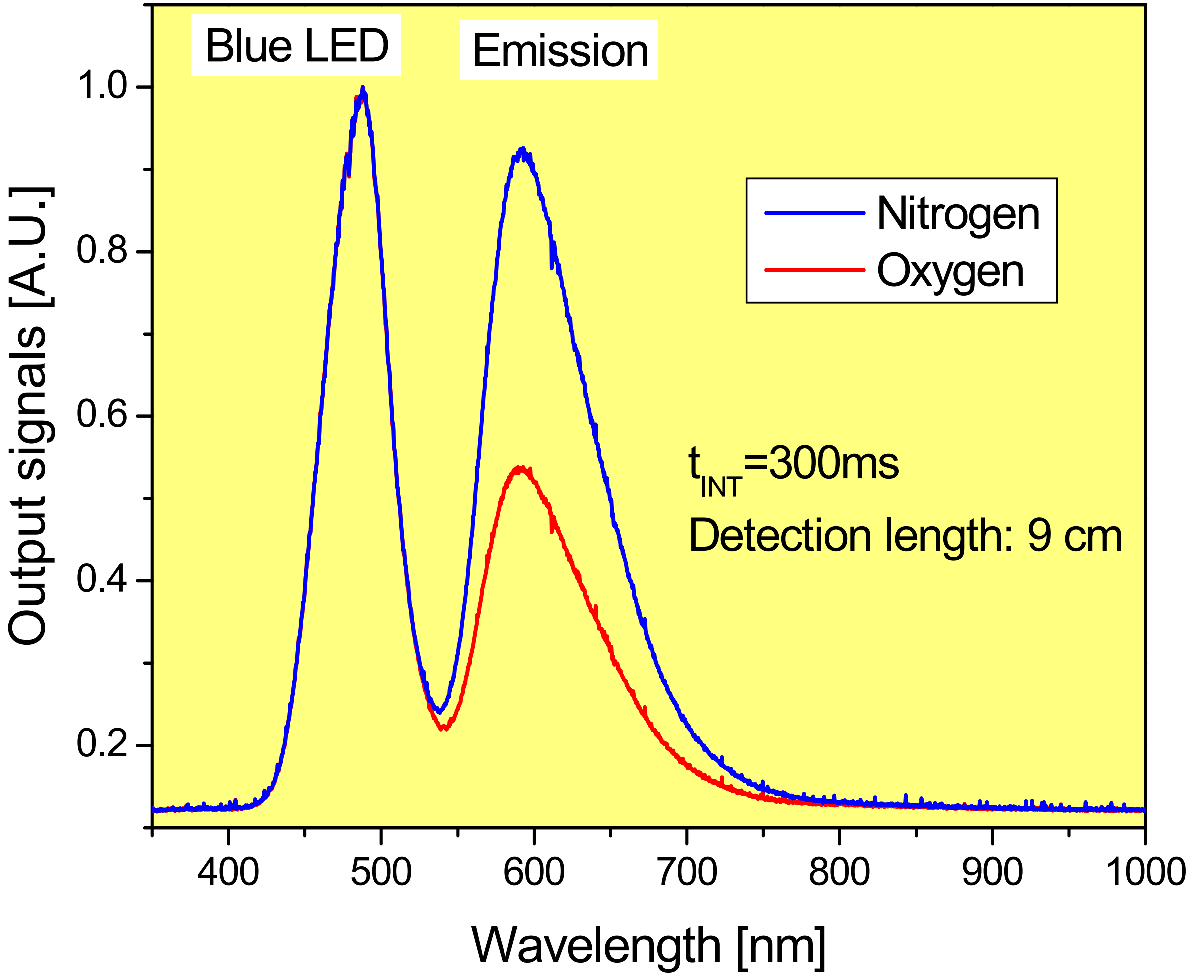
- Fluorescence marks on IgG, enzymes, DNA chains

- Ru complexes with fluorescence quenched by oxygen

- Fluorescence pH indicators – BCECF, HPTS used in biosensor.

DETECTION OF OXYGEN

Oxygen ↔ $Ru(phen)_2Cl_2$ ⇒ fluorescence quenching



Function

1. *Control the refractive index in the detection site*
2. *Increase concentration of analyte in the detection site* (partition coefficient K_P)

$$K_p = \frac{C_{site}}{C_{external}}$$

DETECTION MEMBRANES

$K_p \rightarrow \infty$ (for analyte)

$K_p \rightarrow 0$ (other substances in sample)

3. Immobilize optical transducers in the detection site

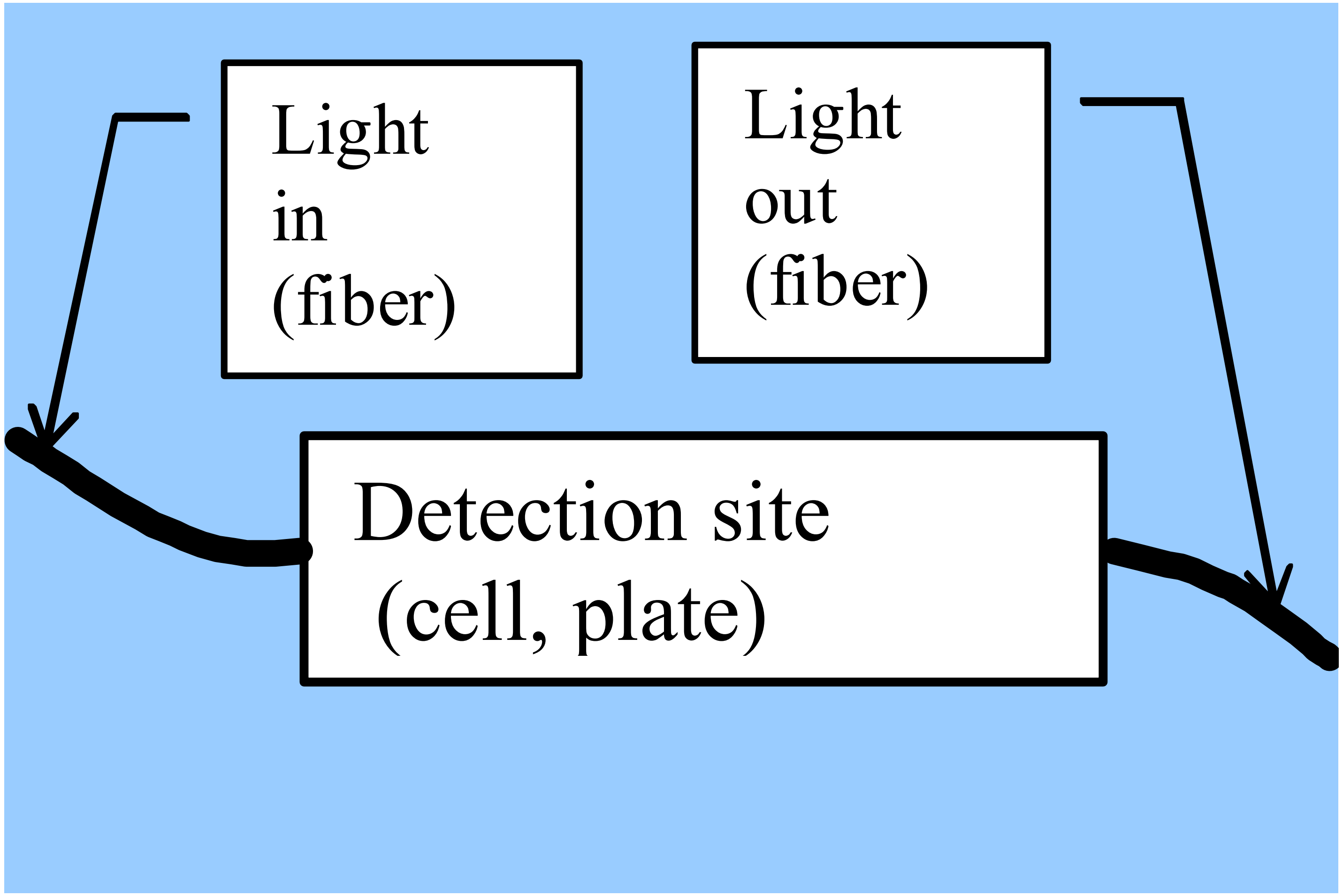
FIBER-OPTIC CHEMICAL SENSORS (FOCS)

- They are used for transmission of light to and from detection site (extrinsic sensors)

- Detection sites can be created in their cladding (intrinsic sensors)

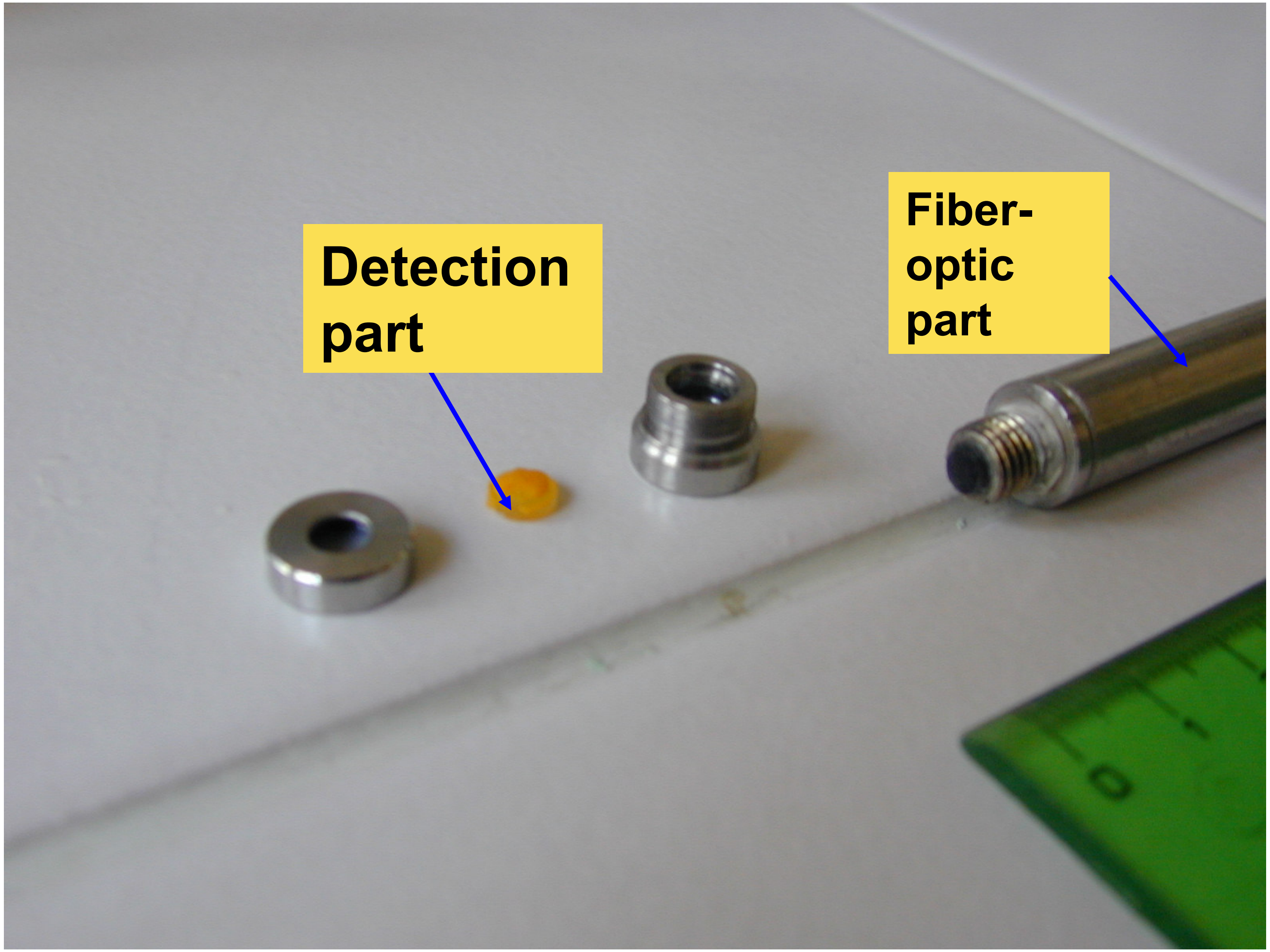
- Detection membranes and optical transducers are applied onto fibers, creating the detection site

EXTRINSIC FOCS

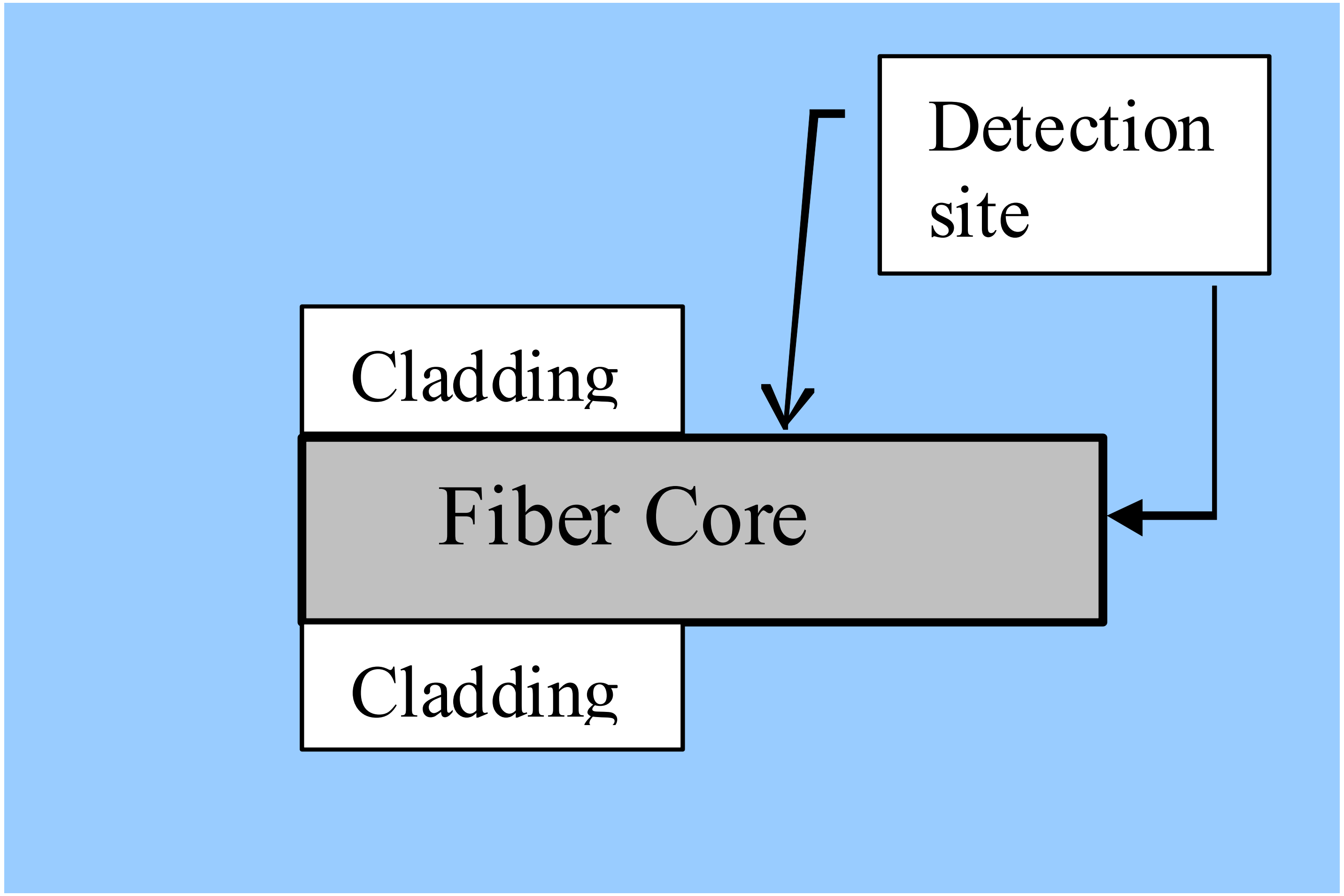


Detection site is outside the fiber (cell, plates)

IPE - EXTRINSIC FOCS OF OXYGEN

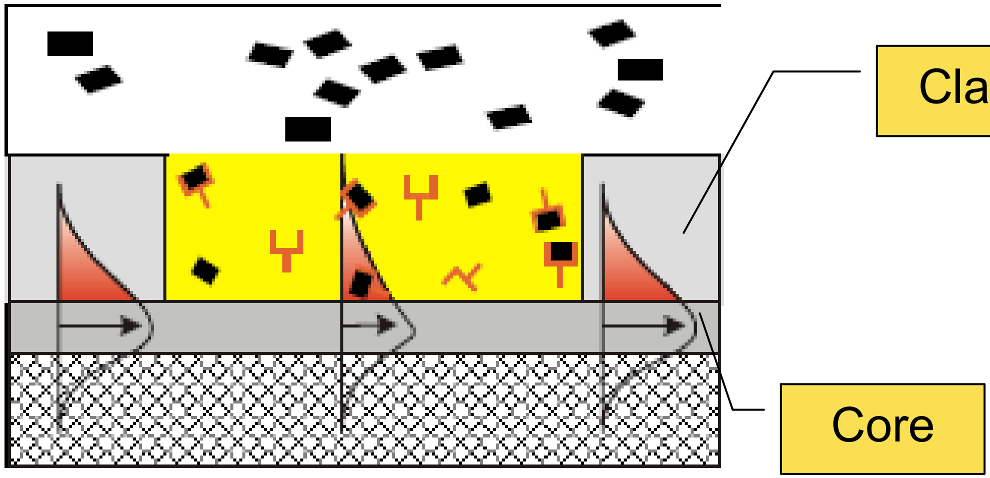


INTRINSIC FOCS



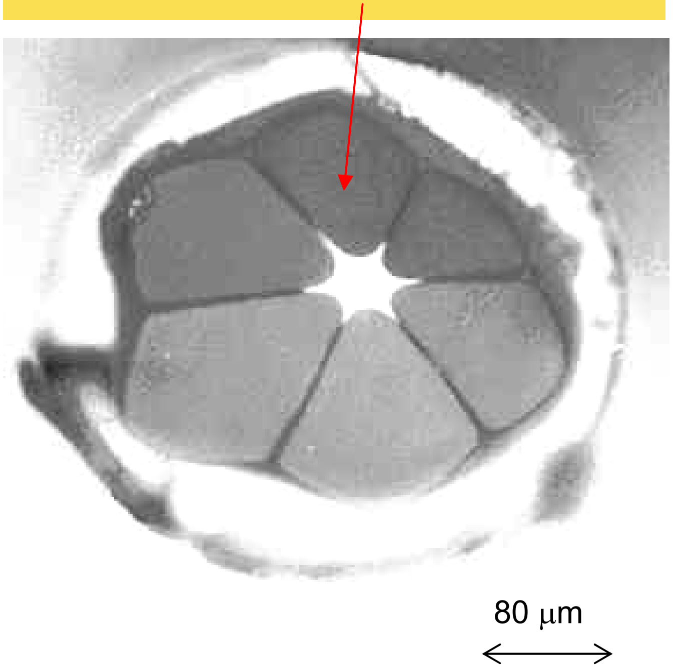
Detection site is in the fiber structure (cladding, core)

INTRINSIC FOCS



Detected substance (gas or solution) in direct contact with guided modes

Detection site in cladding holes



ADVANTAGES OF FOCS

- Small dimensions, long detection lengths
- Distributted and remoted detection

Disadvantages of FOCS

- Special instrumentation of chemical parts
- Competition with other sensor types (electrical, mechanical)
- Parazitic signals, fawling of detection sites

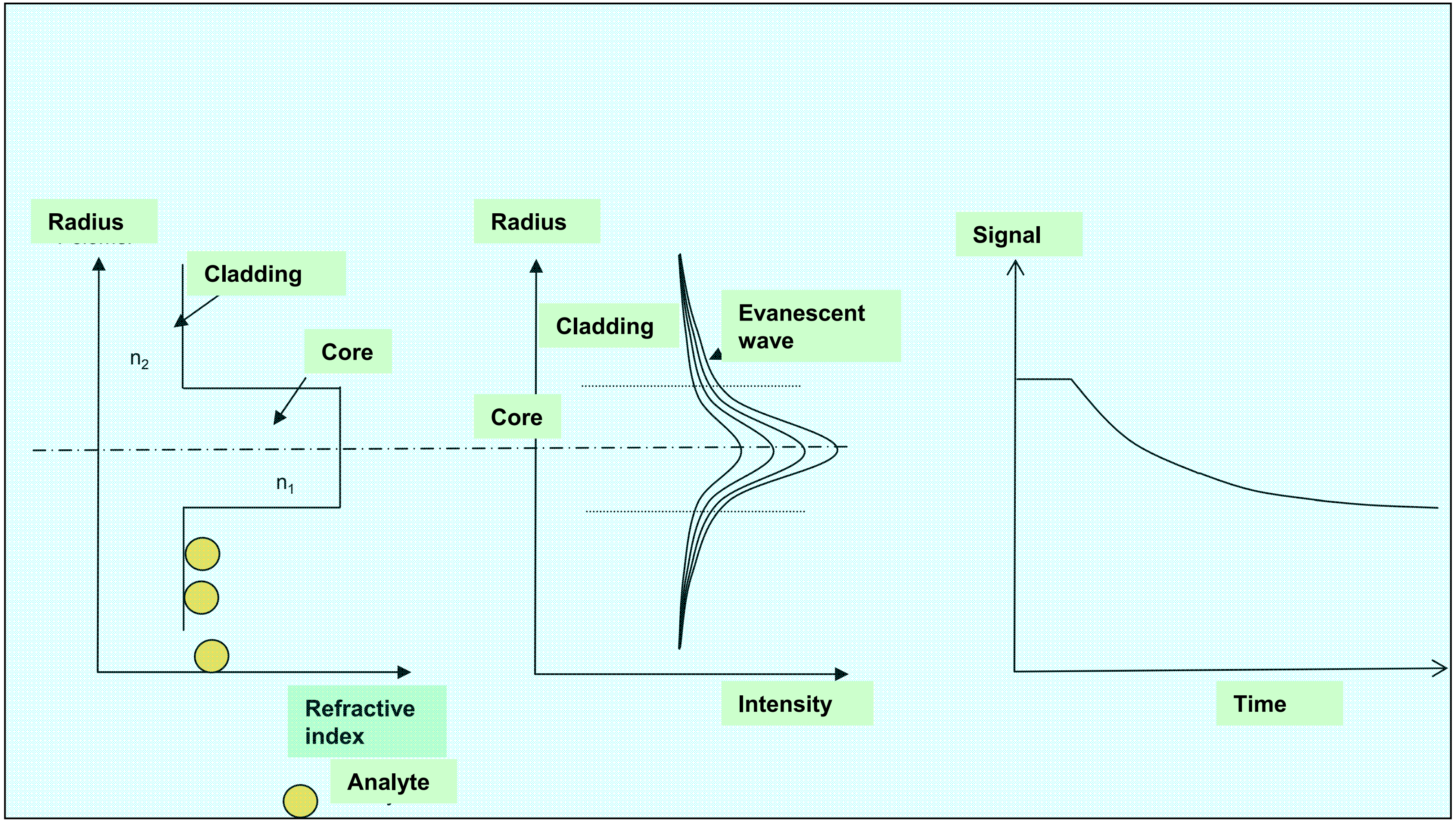
POTENTIAL APPLICATIONS OF FOCS

- **in remote and dangerous spaces** (petrol tanks, rafineries, workshops with flammable and explosive gases, depots of radioactive substances)
- **In medicine** for continuous monitoring of concentrations of gases, pH, in blood or in organs such as stomach or brain.

DETECTION STRUCTURES OF FOCS

- **Evanescent FOCS**
- **Reflection FOCS**
- **Surface plasmon (SPR) FOCS**
- FOCS based on optical gratings (LPG)
- FOCS based on fiber tips, tapers

EVANESCENT-WAVE SENSOR PRINCIPLE



Analyte penetrate into the cladding, changes optical properties there → changes in transmission of evanescent wave and signal

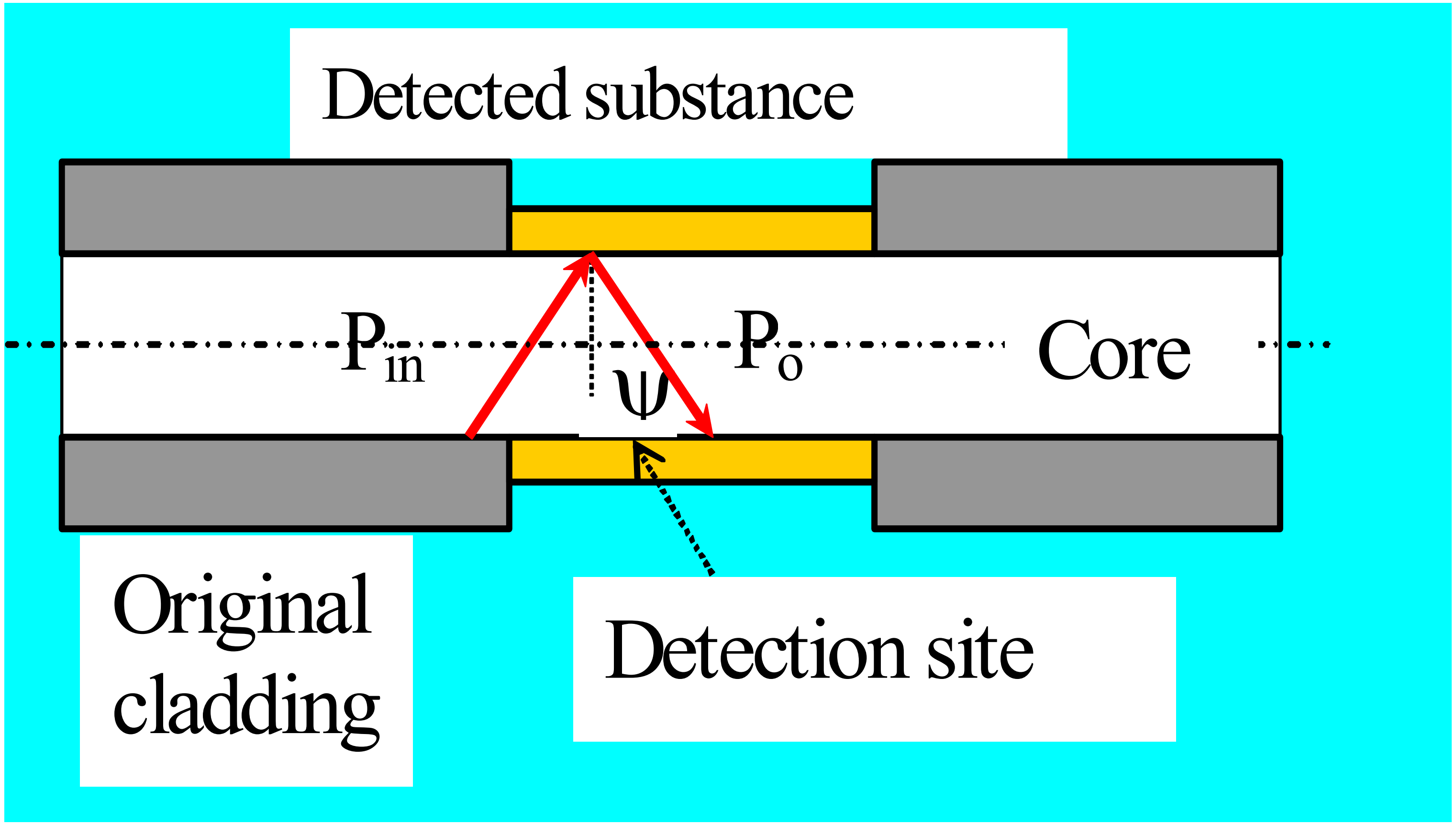
PRINCIPLE OF EVANESCENT FOCS

Analyte changes transmission of evanescent wave in the cladding (a decrease of its amplitude) due to:

- Refractive index – refraction sensors
- Optical absorption - attenuated total reflection sensors
- Luminescence – luminescence sensors

Consequently amplitude of part of optical mode propagating in the core (evanescent wave is a part of optical mode) changes which is detected as change of the output signal.

RAY-OPTIC MODEL OF EVANESCENT-WAVE SENSOR



$$P_o = P_{in} R$$

R – power reflection coefficient <u>$R <= 1$</u>

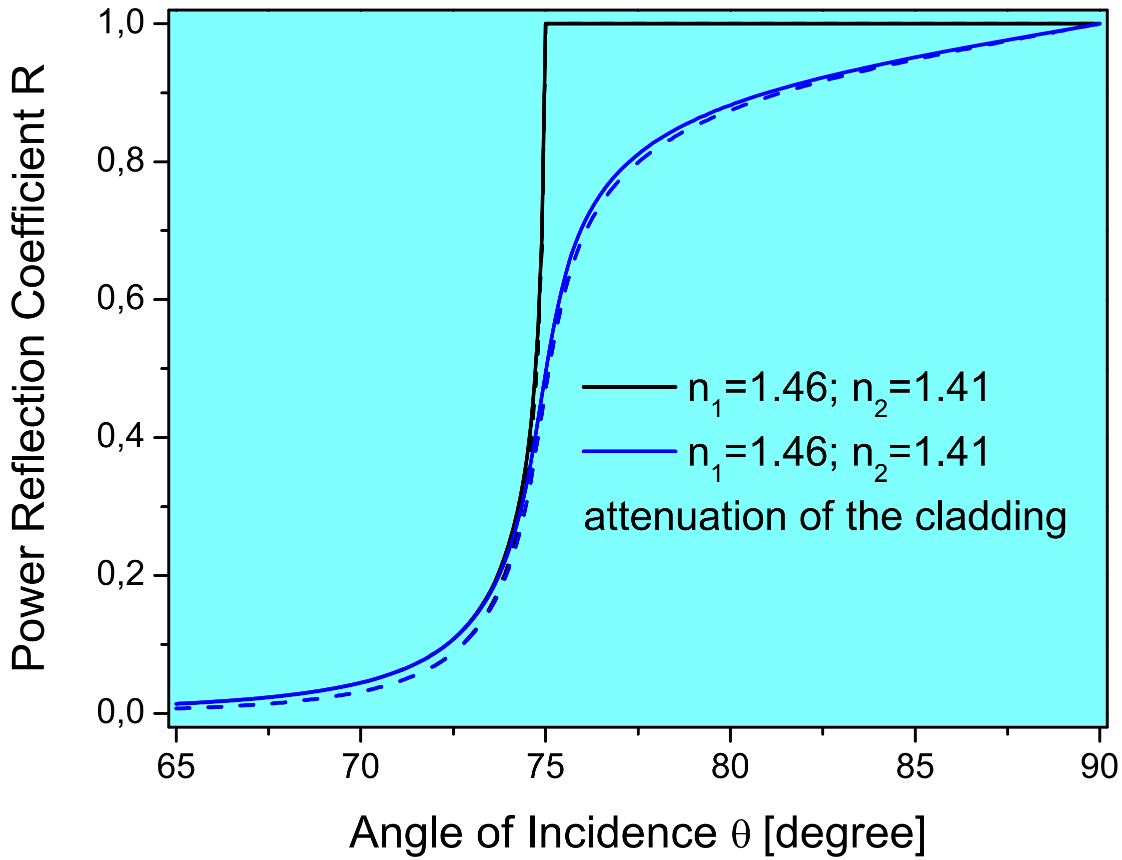
OPTICAL POWER TRANSMITTED BY RAY

$$P_i = P_{0i} R^{N_i}$$

$N_i \sim L/d$ – number of reflections

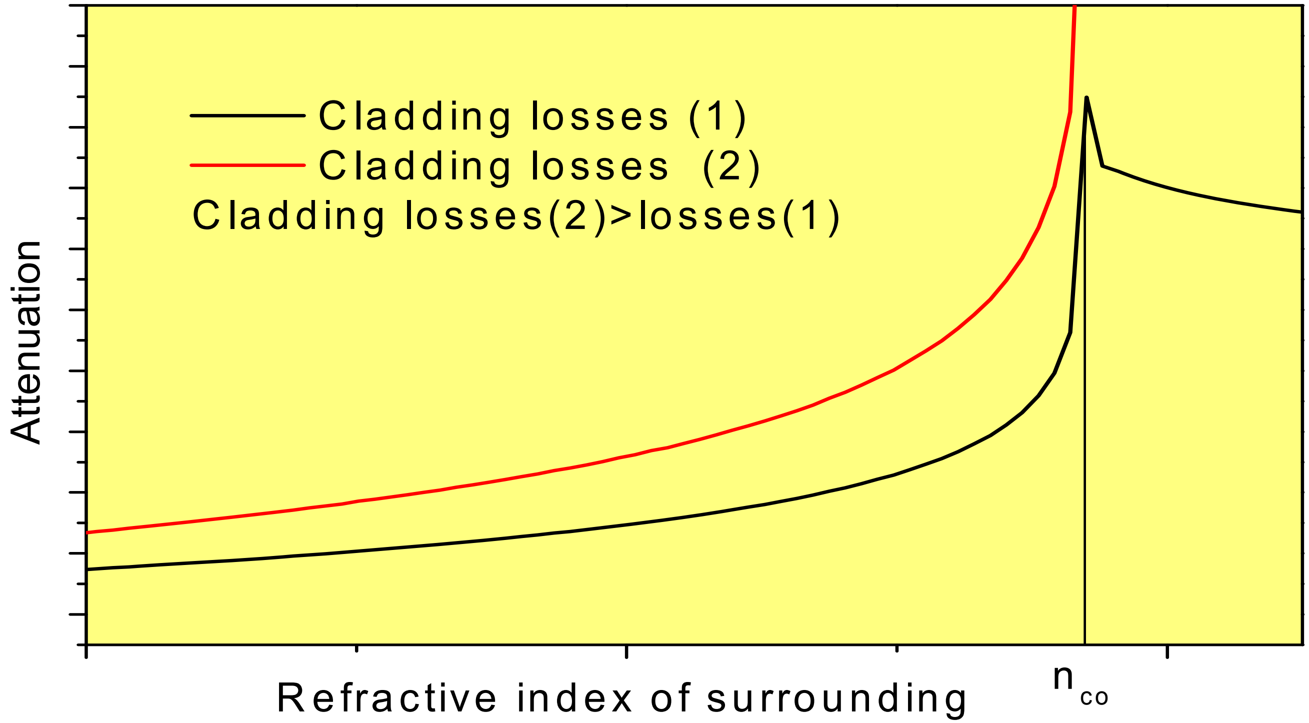
P_{0i} – optical power launched into i-th ray on the input face of the fiber

REFLECTION COEFFICIENT- REFLECTIVITY R



$\theta = 90 - \Psi$ – axial angle complementary to reflection angle

TRANSMITTED POWER ↔ RI AND CLADDING LOSS



Attenuation = 10 log(Input power/Transmitted power)

Attenuation ↑↔ Transmitted power ↓

TRANSMITTED POWER ↔ ATR SENSORS

Meridional rays – Approximate formula

$$P_i = P_{0i}\,\exp(-\gamma\ \ L)$$

$$\gamma \approx \varepsilon_{cl}\,\frac{\lambda}{\pi d\sqrt{n_{co}^2 - n_{cl.}^2}}\,\frac{\left(\frac{\theta}{\theta_c}\right)^2}{\sqrt{1-\left(\frac{\theta}{\theta_c}\right)^2}}$$

$\theta = \pi/2 - \Psi$ – complementary angle to the angle of reflection Ψ **(ATR – Attenuated Total Reflection)**

LIMITS OF EVANESCENT-WAVE SENSORS

Sensitivity

~1% of total power transmitted in the cladding of standard optical fibers → low sensitivity to optical changes in cladding.

$\gamma<0.01$

Detection depth

Intensity of evanescent wave exponentially decreases from the core/cladding boundary

Penetration depth d_p – place in the cladding where intensity = 1/e intensity on the boundary

$d_p \sim 0.1 – 1\ \mu m$

$$d_p = \frac{\lambda}{2\pi\sqrt{n_1^2 \sin^2 \Psi - n_2^2}}$$

WAYS FOR SENSITIVITY INCREASE

Higher sensitivity ↔ Lower value of transmitted power P_i

1. Increase of detection length L
2. Increase of bulk absorption coefficient ε
3. Decrease of core diameter d
4. Approaching $n_{cl} \rightarrow n_{co}$
5. Approaching $\theta \rightarrow \theta_c$

These approaches are used for multimode fibers

EVANESCENT-WAVE SENSORS ON MULTIMODE FIBERS

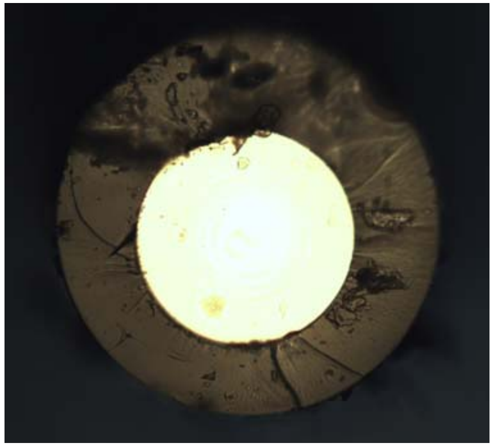
- **Polymer-Clad Silica (PCS) fibers**

  Low prices, good availability, robustness, simple modification , losses 0.01 dB/m

  Core: silica n=1.46

  d ~ 0.2-1 mm,

  Cladding polydimethylsiloxane (PDMS), fluorinated acrylate (n=1.41)



- **Polymer-Clad Glass (PCG) fibers** with expensive, commercially hardly available, losses ~ dB/m

  core: optical glass (n~1.5-1.6)

  d~ 0.4 – 0.6 mm

  cladding: PDMS, UV acrylate

EVANESCENT-WAVE SENSORS ON MULTIMODE FIBERS

- **Polymeric fibers**

cheap, commercially available, flexible, higher optical losses ~ dB/m

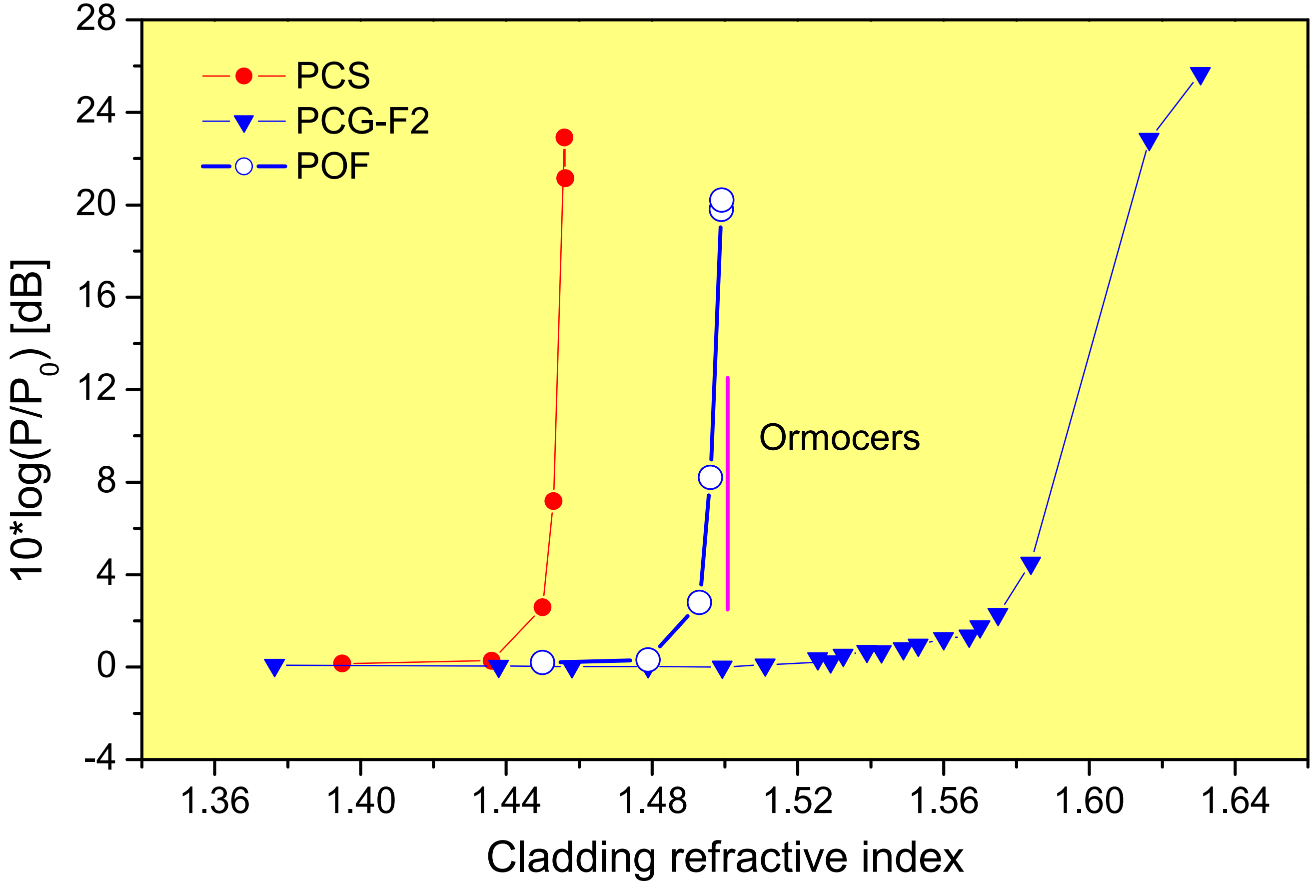
core: polymethylmetacrylate (PMMA) n~1.49
d ~ 1 mm

cladding: less dense PMMA or other polymers

In evanescent-wave sensors cladding is usually removed on a length of several cm → making thus core/cladding boundary accessible for analytes

SENSITIVITY INCREASE: $n_{cl} \rightarrow n_{co}$

Callibration curve

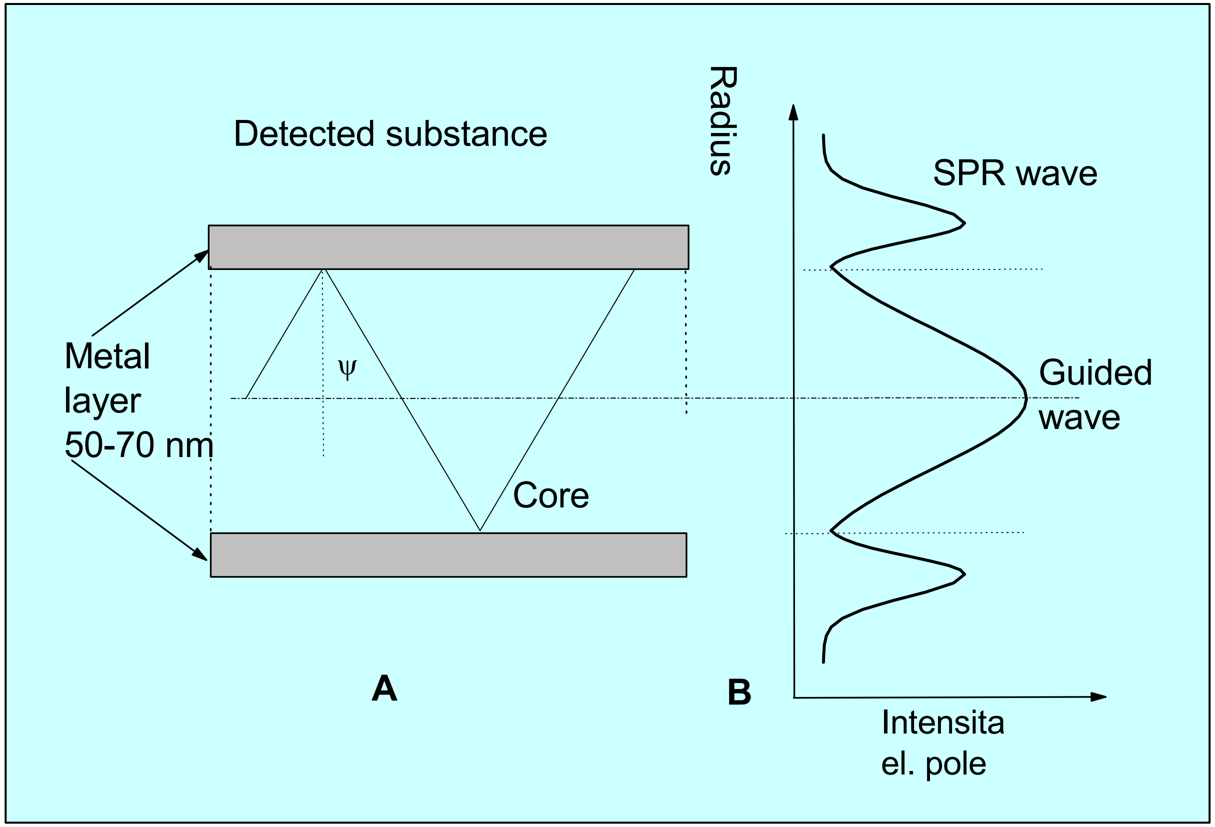


Sensitivity is high only for analyte refractive indices approaching to n_{co}

SURFACE PLASMON RESONANCE (SPR) SENSORS

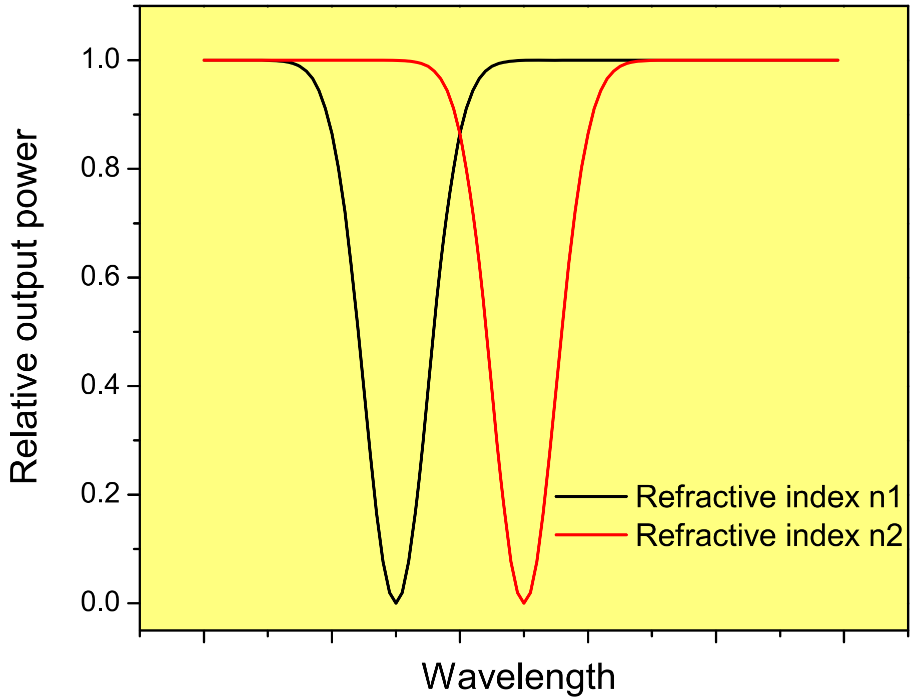
- a thin layer Au or Ag (30-50 nm) applied onto the core/cladding boundary
- evanescent wave excites free electrons in the layer to a higher energy level
- at proper wavelength, propagation constant, refractive index of surrounding energy from the wave is transferred to electrons → output power decreases (surface plasmon resonance - SPR)

SCHEME OF SPR FOCS



SPR wave penetrate into analyte on the layer boundary (100nm) and can be used for detection of analyte refractive index

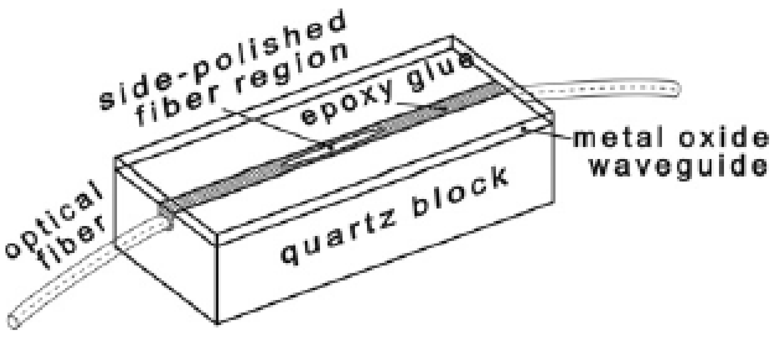
SP RESONANCE

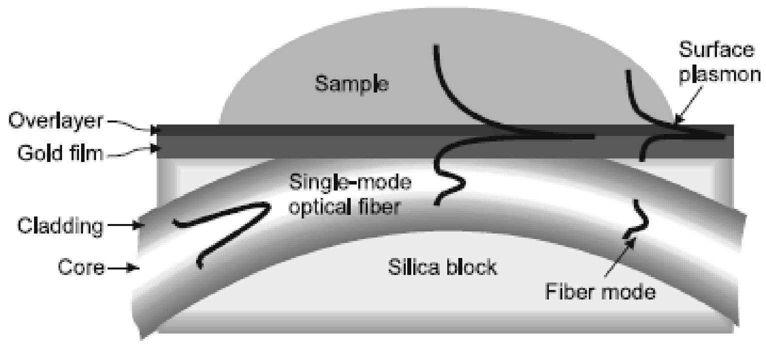


SP resonance= dip observed at the output power spectrum, position depends on wavelength and analyte refractive index. Can be used also in aqueous solutions even with silica fibers.

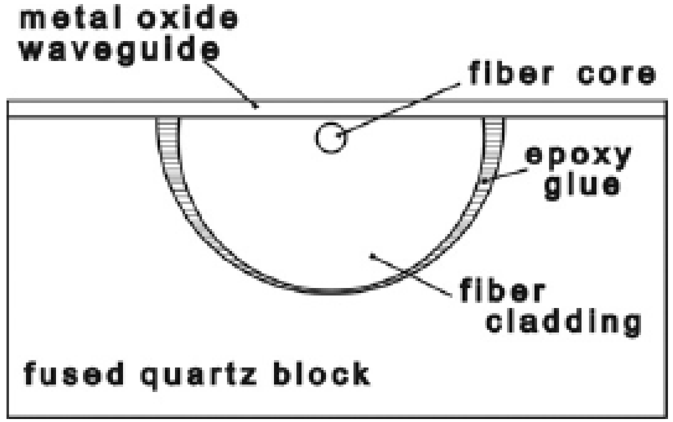
IPE SPR FOCS – SIDE POLISHED FIBER

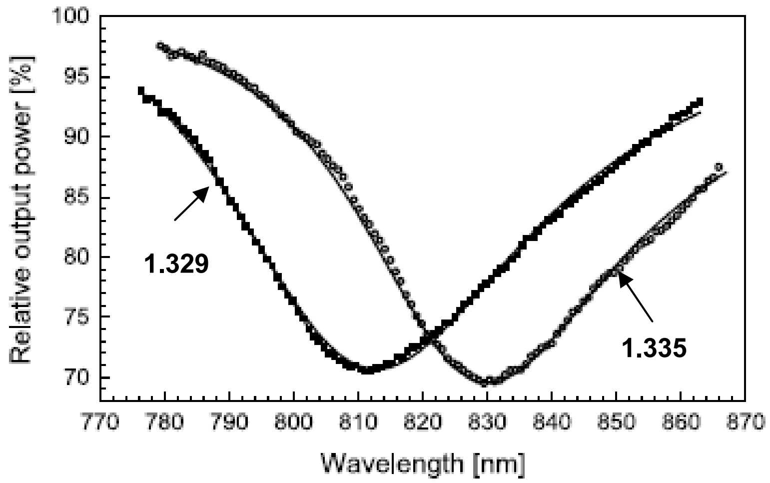
(a)





(b)

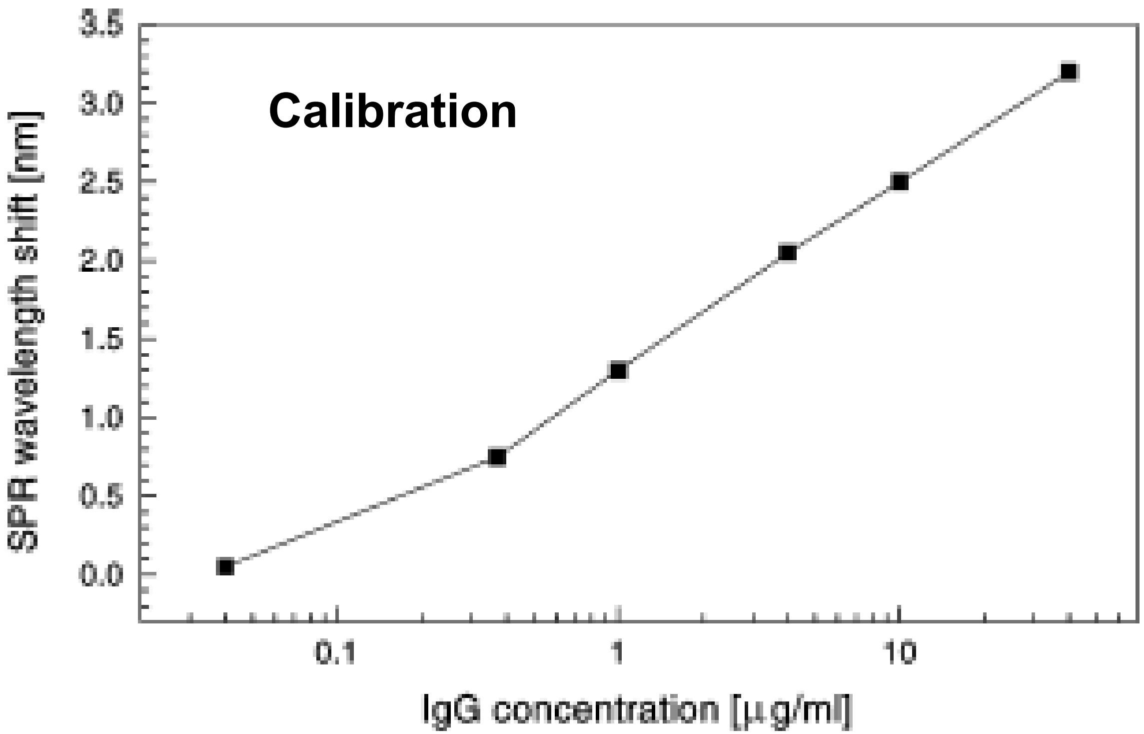




R. Slavik et al., Novel spectral fiber optic sensor based on surface plasmon resonance, *Sens. Actuators B*74, 106-111 (2001)

SPR FIBER-OPTIC BIOSENSOR

SM fiber side-polished, Au layer 30 nm; immunotransducer antigen to IgG fixed on the fiber , detection of IgG in a buffer solution (PBS) a BSA.

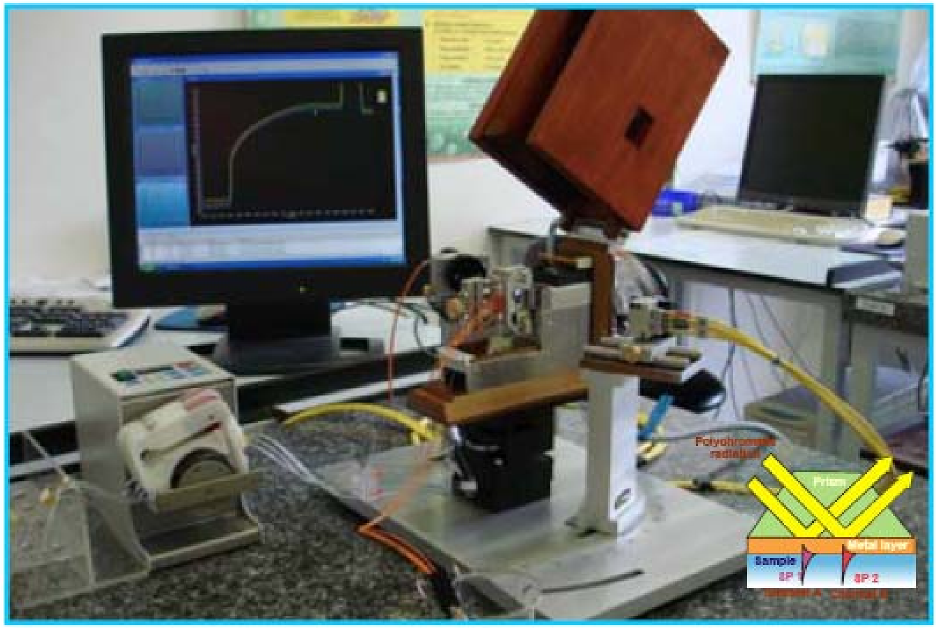


PBS – Phosphate Buffered Saline (pH=7,4), BSA-Bovine Serum Albumine

IPE LABORATORY OF SPR SENSORS

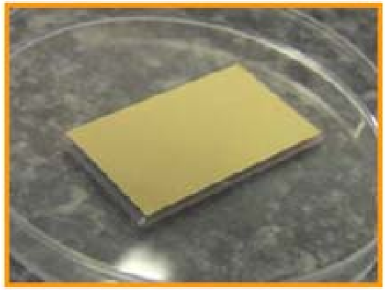
Employs usually optical prism for SP excitation (details www.ufe.cz)

- Spectroscopy of surface plasmons.
- Four sensing channels, (flow chamber volume 0.5 μL per channel)
- Temp. stabilization (stability < 0.02°C)



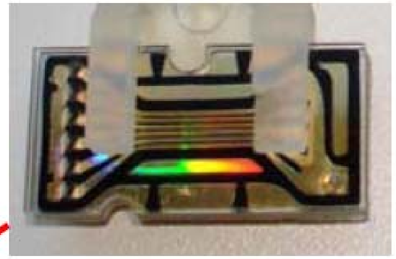
RI RESOLUTION:
< 2×10^{-7} RIU
OPERATING RANGE:
1.32-1.45 RIU

Four-channel SPR sensor and (top) and detail of an SPR chip (right).



IPE PROTOTYPE OF SPR SENSOR

Based on Au layers excited by optical grating



SENSITIVITY:
615 nm/RIU

RESOLUTION:
< 2×10^{-7} RIU

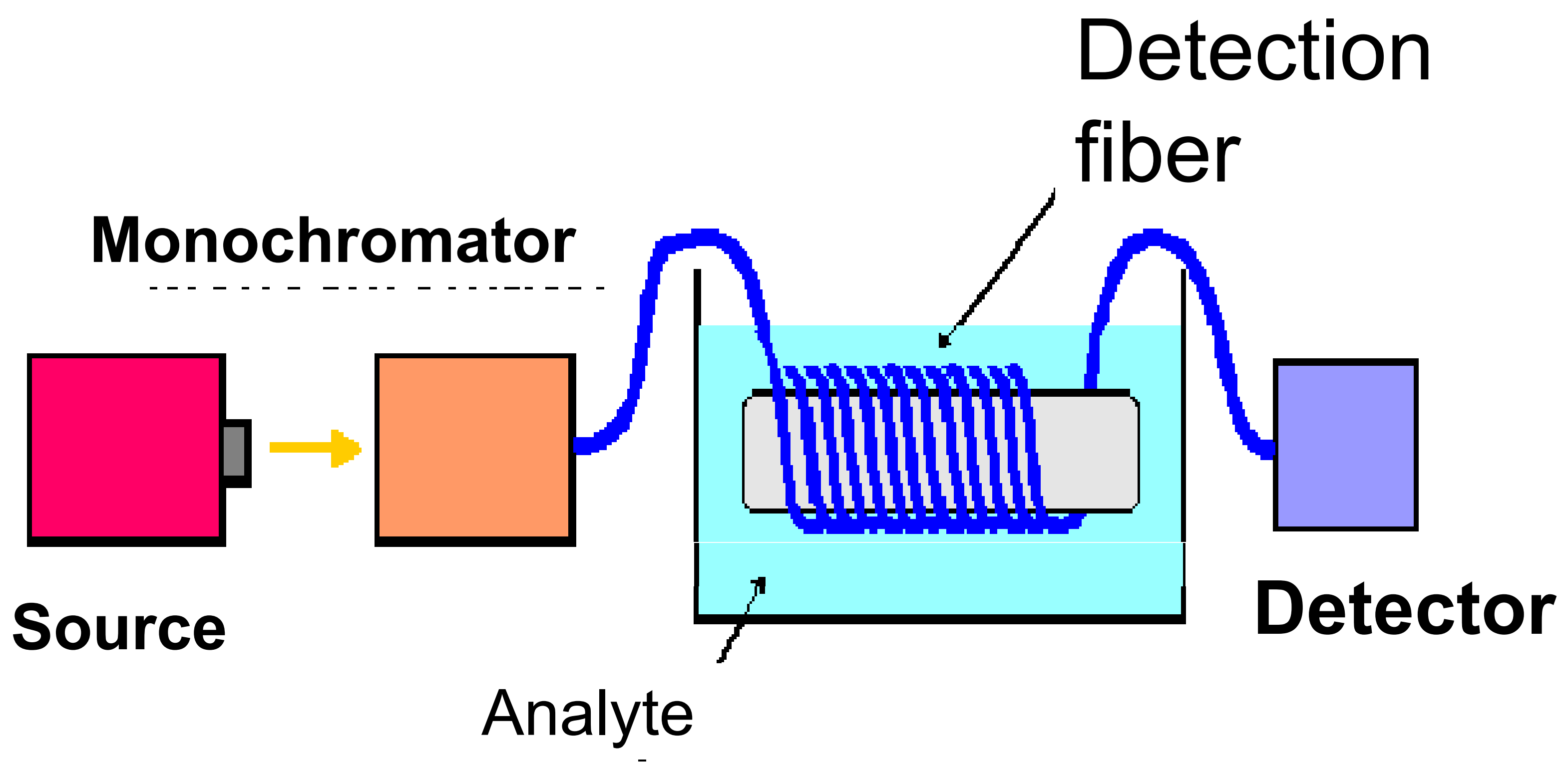
OPERATING RANGE:
1.33-1.35 RIU

Laboratory prototype of 6-channel SPRCD sensor.

Used in > 5 countries, Hungary, Turkey, USA Federal Drug Agency

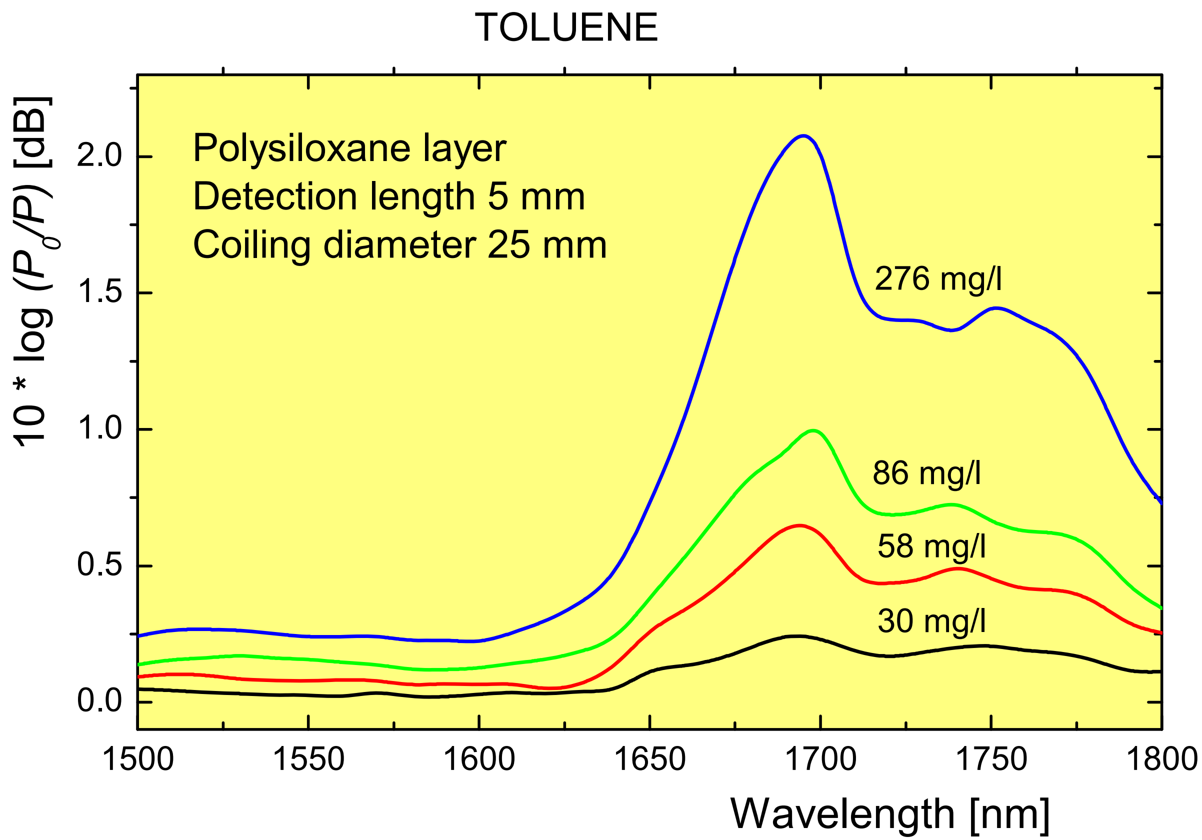
M. Piliarik, M.Vala, I. Tichý, J. Homola, *Biosens. Bioelectr.* 24, 3430–3435 (2009).

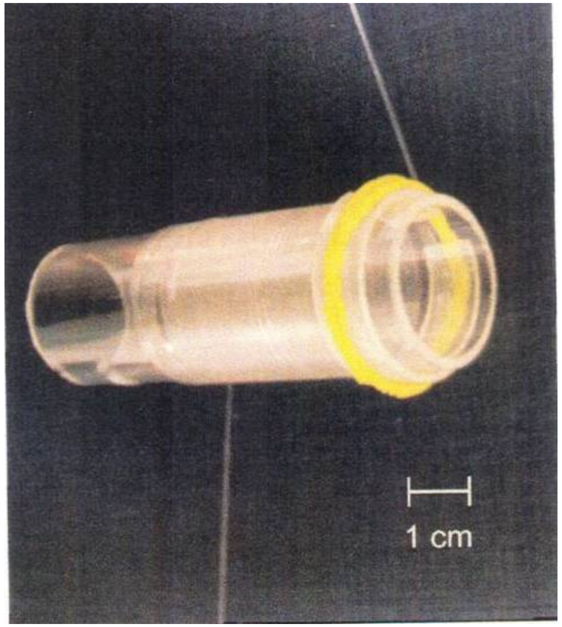
SENSITIVITY INCREASE: L↑



Up to 10 m of PCS fiber (core diameter 0.2 mm) coated with hydrophobic siloxane polymer coiled onto a glass rod (a diameter of 25 mm)

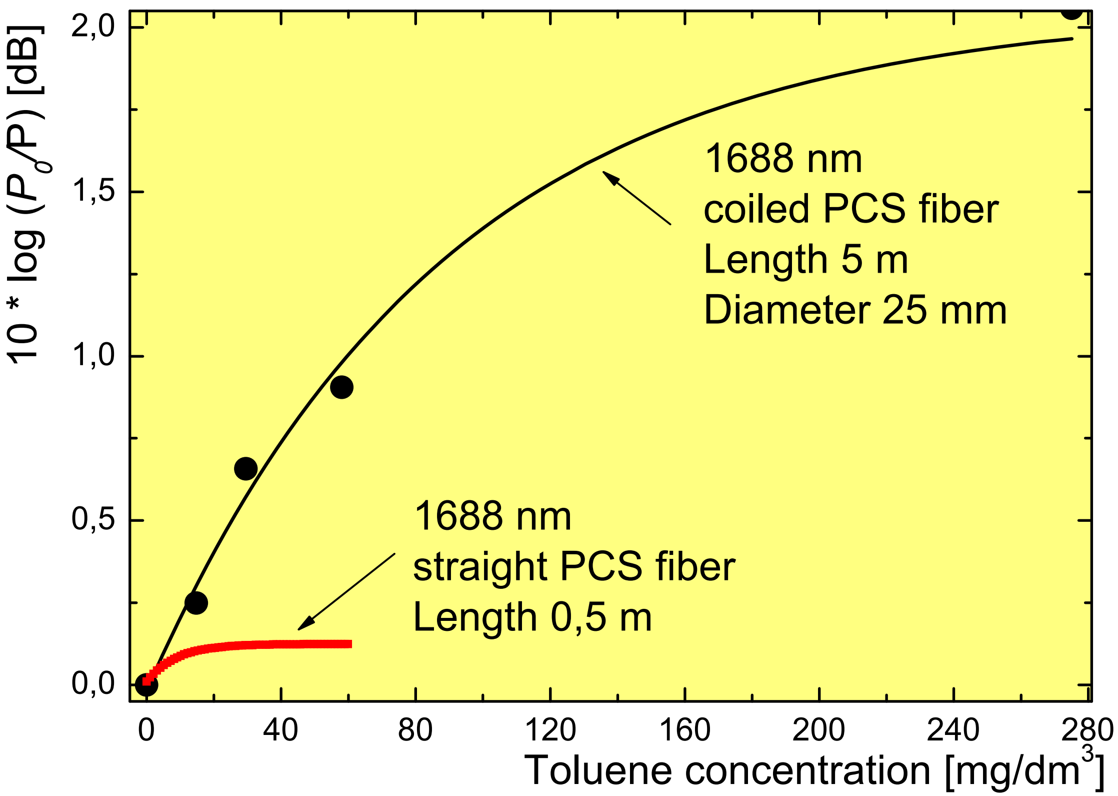
SPECTRAL DETECTION OF TOLUENE IN WATER





PCS fiber 0.2 mm, cladding PDMS

CALIBRATION CURVES



Effects: L, n_{cl}, ↑ and θ↓

SENSITIVITY INCREASE: d↓

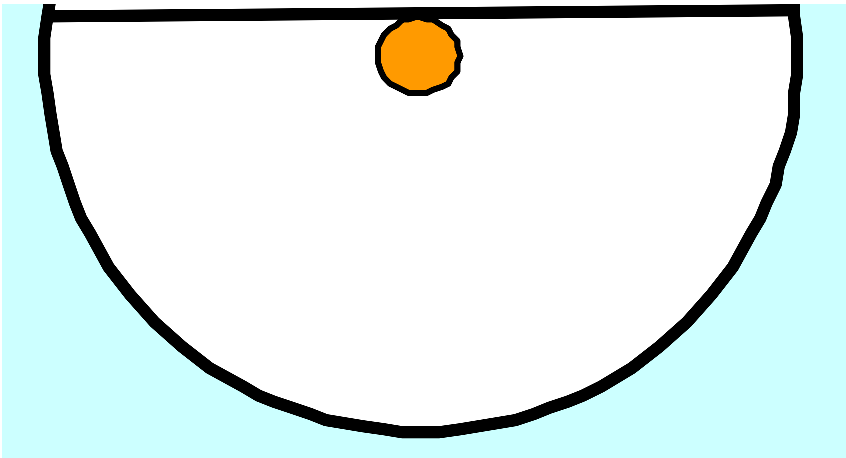
Single-mode and GI fibers can't be directly used for detection because the core is surrounded by glass (thickness 30-50μm)

Outer glass tube and cladding have to be removed to <u>open core/cladding boundary</u> for analytes

D-shaped fibers or s fibers developed

D-FIBERS

D-fibers (B. Culshaw-UK-1985)



D-fiber prepared from a D-polished preform

Or

by polishing SM fibers

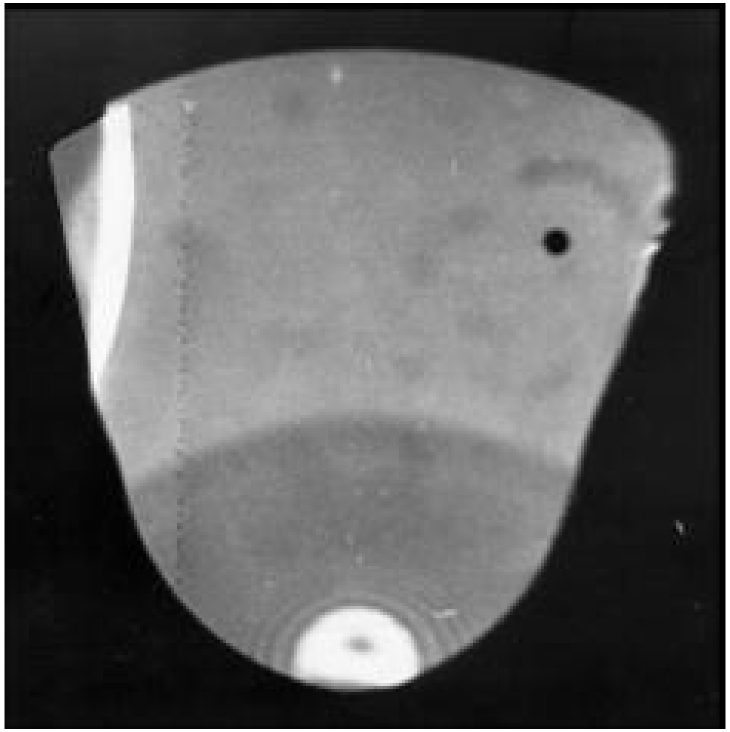
Applied for CH_4, detection, SPR biosensors

G. Stewart, W. Jin, B. Culshaw, Sens. Act. B 38, 42-47, 1997

R. Slavik et al., *Sens. Actuators B*74, 106-111 (2001)

IPE SECTORIAL (s) FIBERS

Similar to D fiber with easy access of analyte to the core/surrounding boundary; enables decrease of core diameter d__ .



S fiber is robust ↔ large sectorial part (0.3 mm)

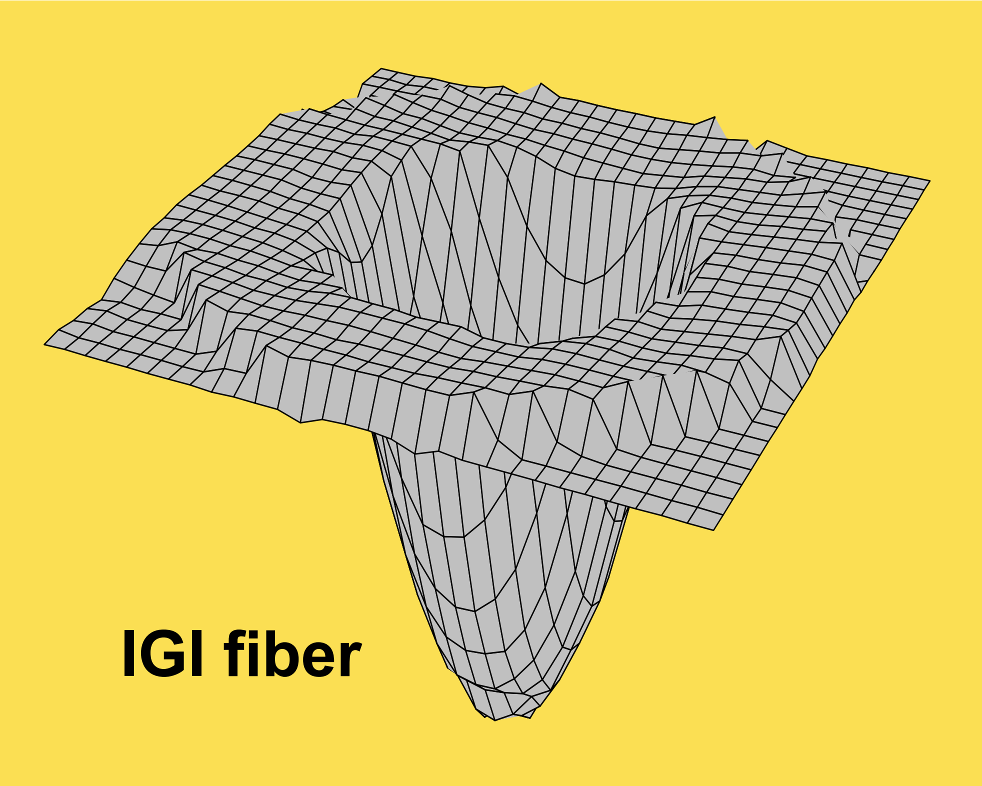
Core diameter - 30μm (2-5x increase of detection sensitivity compared to PCS fiber d=0.3 mm)

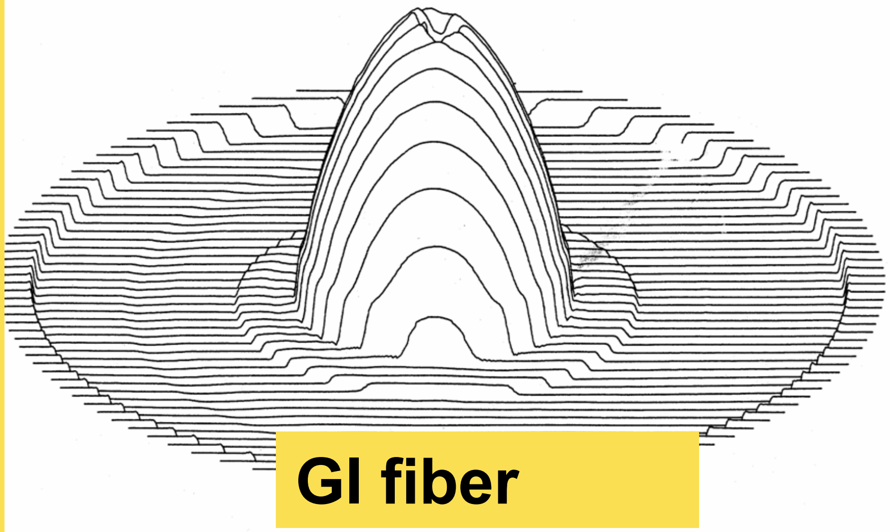
S fiber tested for pH detection

V. Matejec et al., Sens. Actuators B 38-39 (1997) 334-338

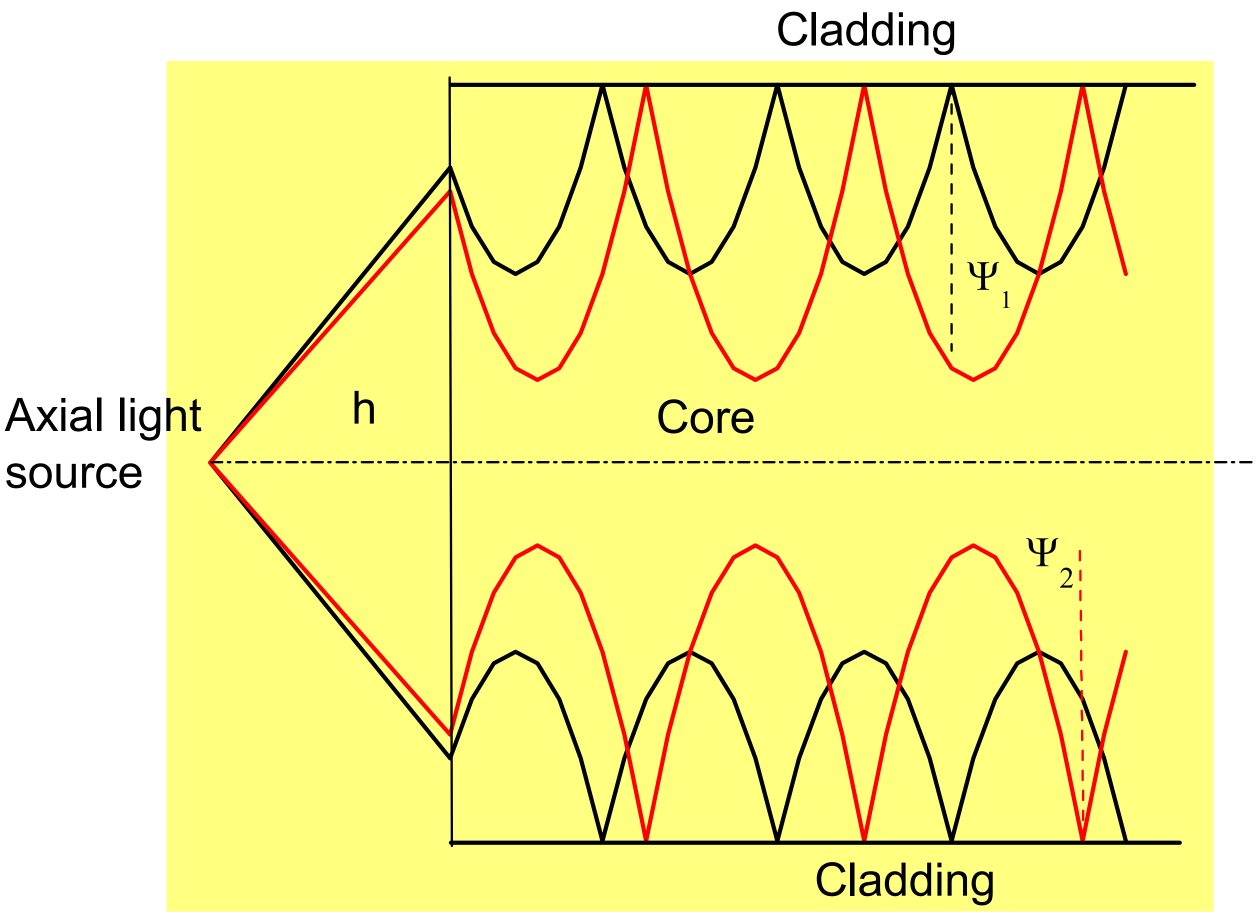
CONTROL OF ANGLE θ

Multimode fibers with inverted graded-index (IGI) refractive-index profile; Enable control of θ on the core/cladding boundary





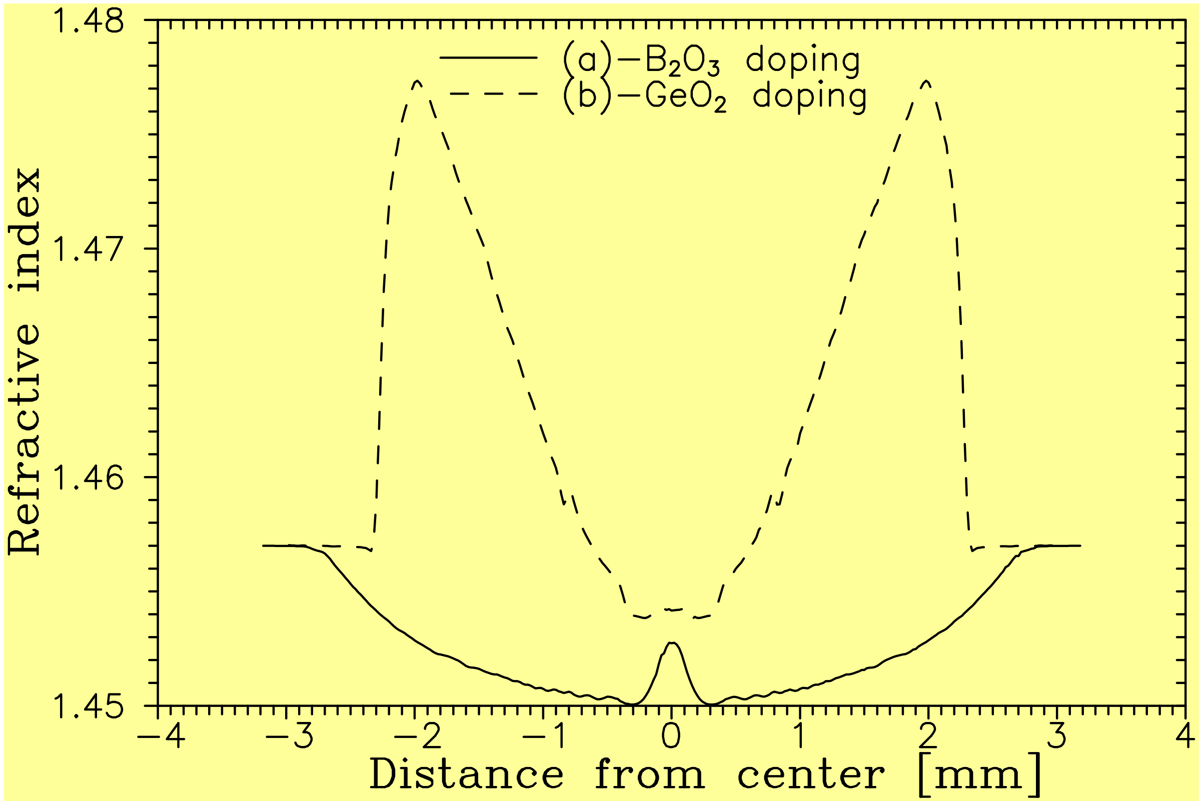
IGI FIBER RAY TRAJECTORIES



Nearly the same angle incident angle Ψ on the core/cladding boundary for different rays → the same sensitivity

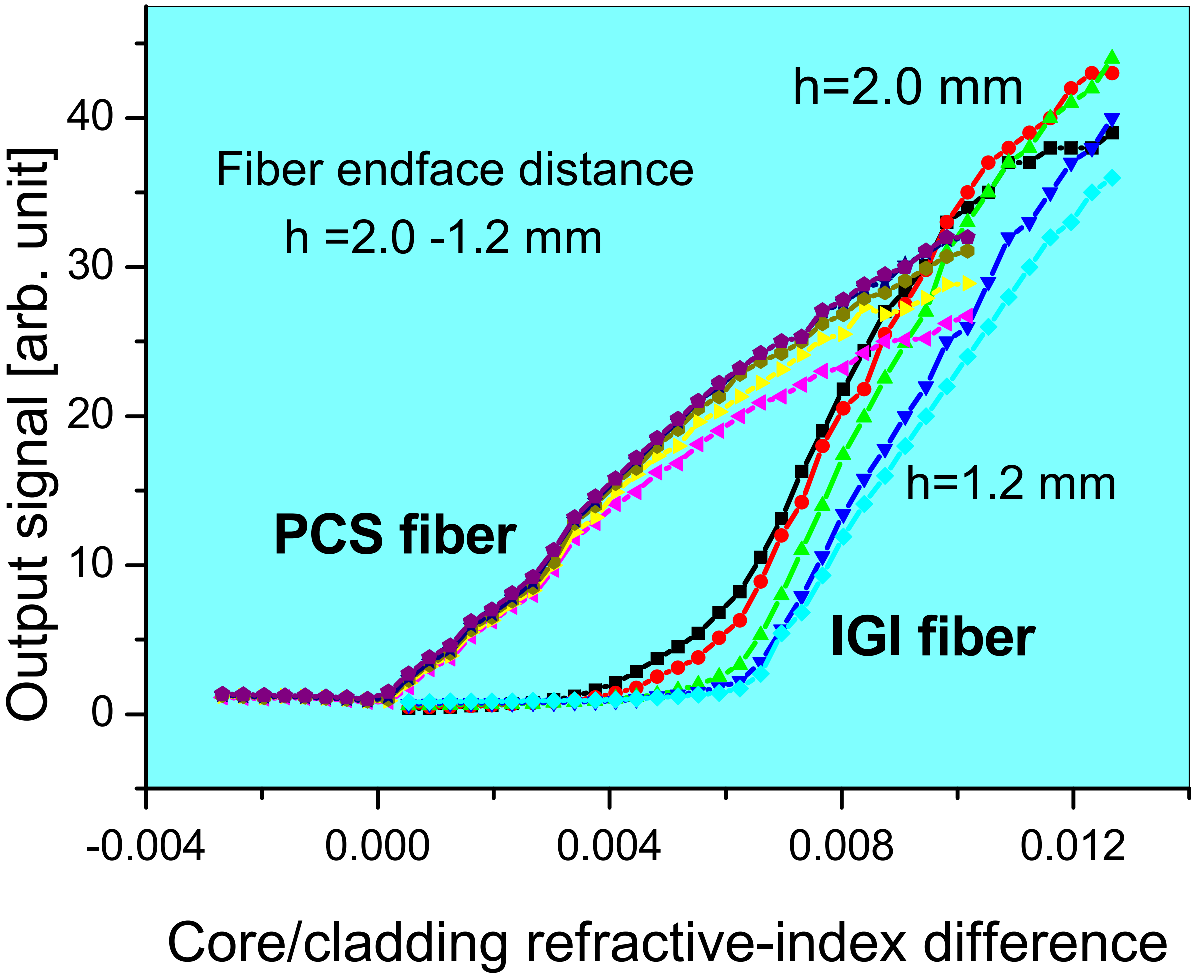
PREPARATION OF IGI FIBERS

MCVD method, boron oxide or germanium oxide silica doping; Sensitivity to refractive index can be controlled by doping in a range 1.45-1.51. Fiber diameter 100-500μm



Minimum RI determines detected refractive index

IPE IGI FIBER – BORON OXIDE DOPED



Sensitivity to RI changes for n=1.45

M. Chomat et al., Sens. Act B 87, 257-268 (2002)

USING OF IGI FIBERS

IGI fibers tested in SPR biosensors, SPR sensors for detection of toluene in water - Collaboration with Ecole Centrale de Lyon

IGI fiber for toluene detection using SPR sensor, detection length 1,5 cm, Au layer 50 nm coated with detection membrane;

Refractive-index changes of the membrane due to toluene penetration in it detected (limit of detection mg/l)

F. Bardine, These de la Universite Claude Monnet, Saint Etienne, 2004

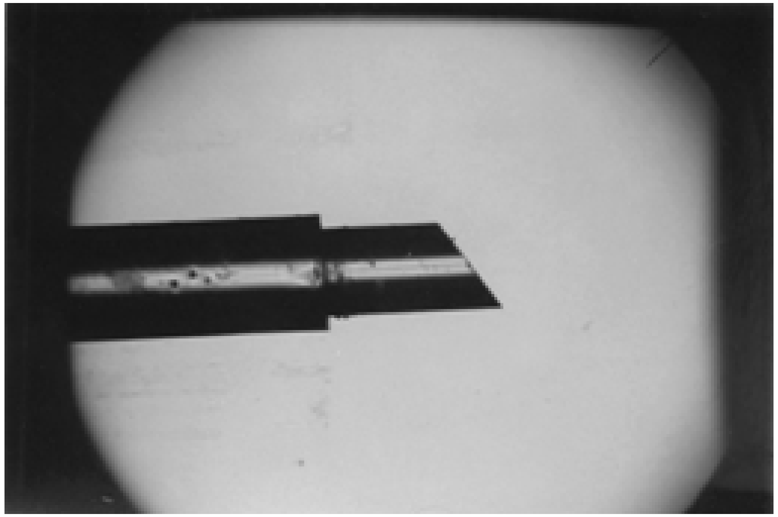
OTHER IPE APPROACHES FOR SENSITIVITY IMPROVING

Control of θ

Excitation by an inclined collimated beam

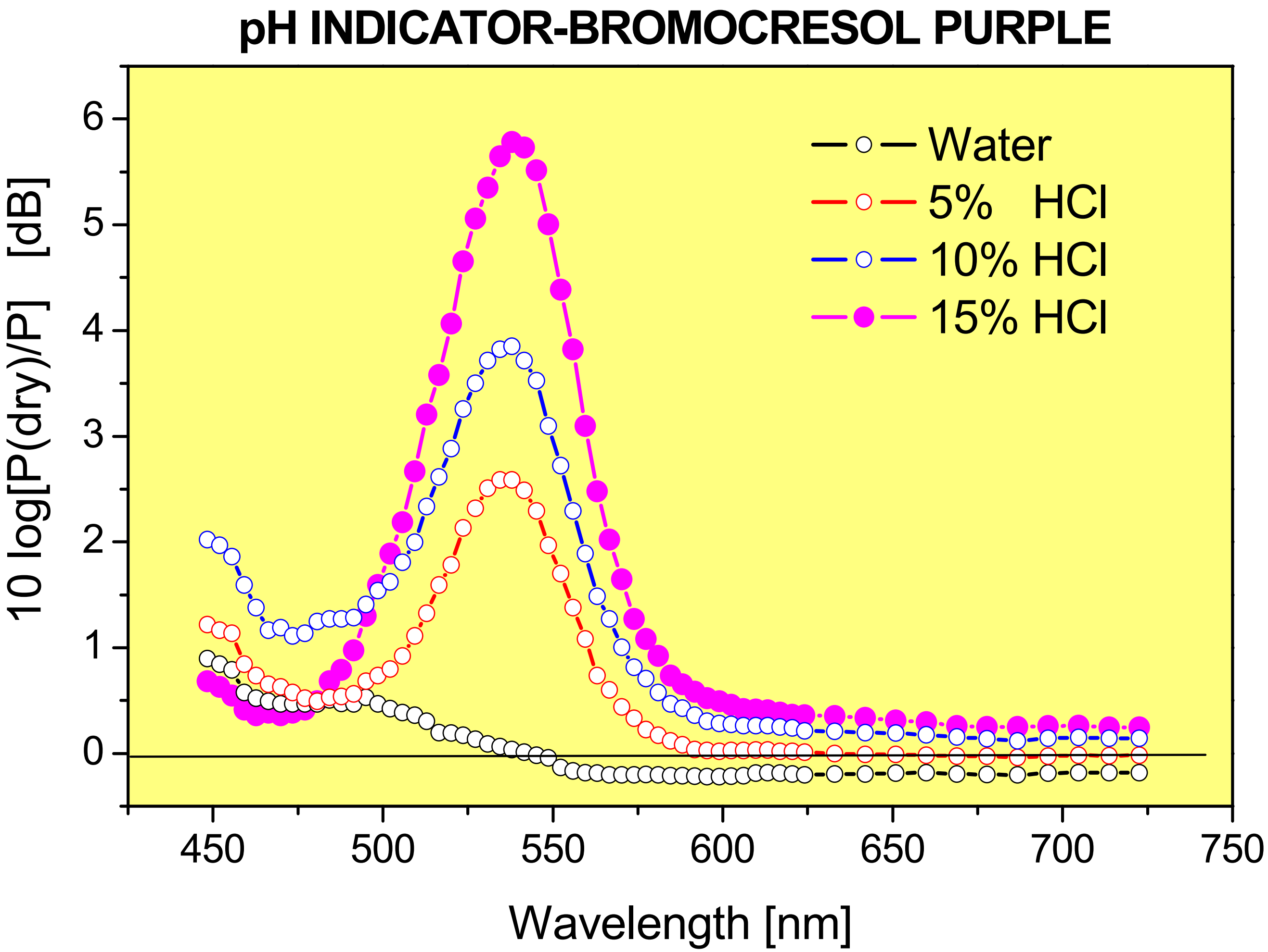
Beveled fibers

Output mode filters



Beveled fiber

pH MEASUREMENTS WITH PCS FIBERS



pH transducer in a porous layer applied onto PCS fiber

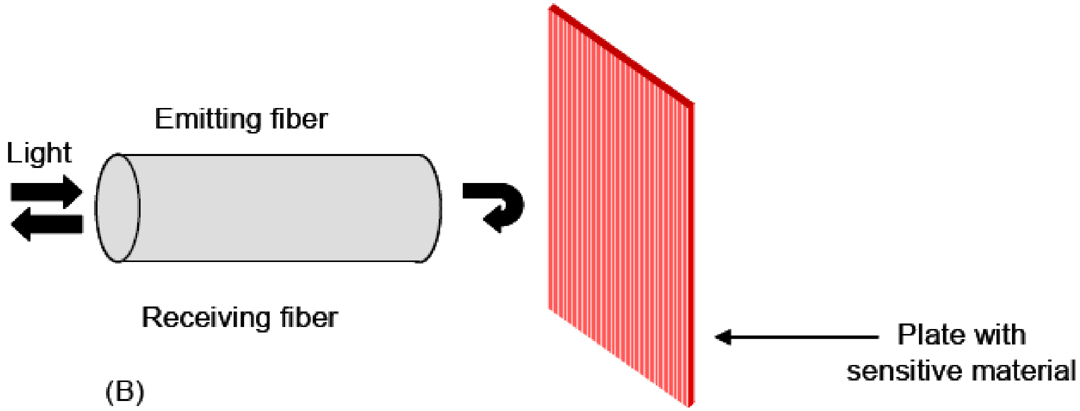
SENSORS OF TOLUENE ON PCS FIBERS

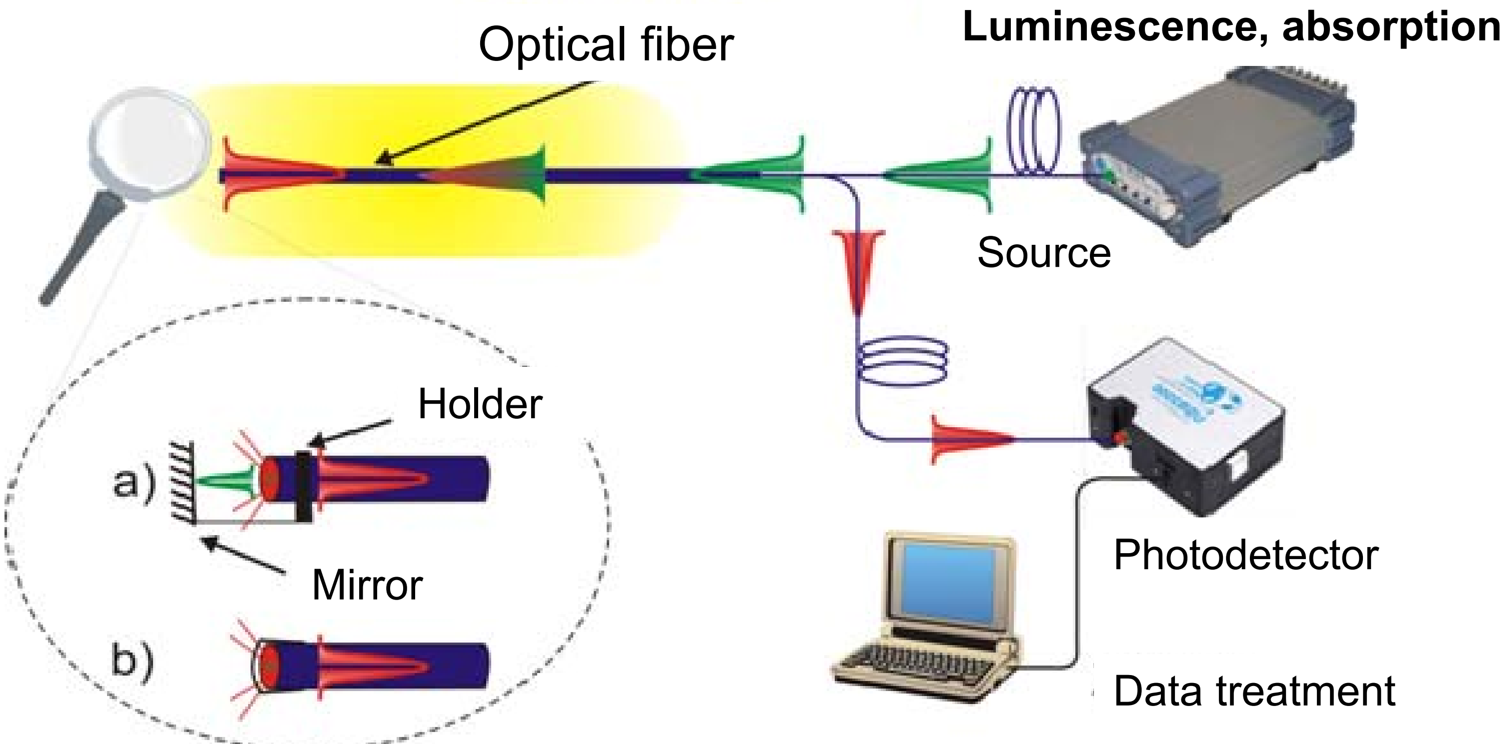
- Gaseous toluene – 0.003%
- Toluene in water – 1-2 mg/l

Practical use – detection of leakages of petrol from tanks, petrol lines

Control of quality of waste water from petrol refinery

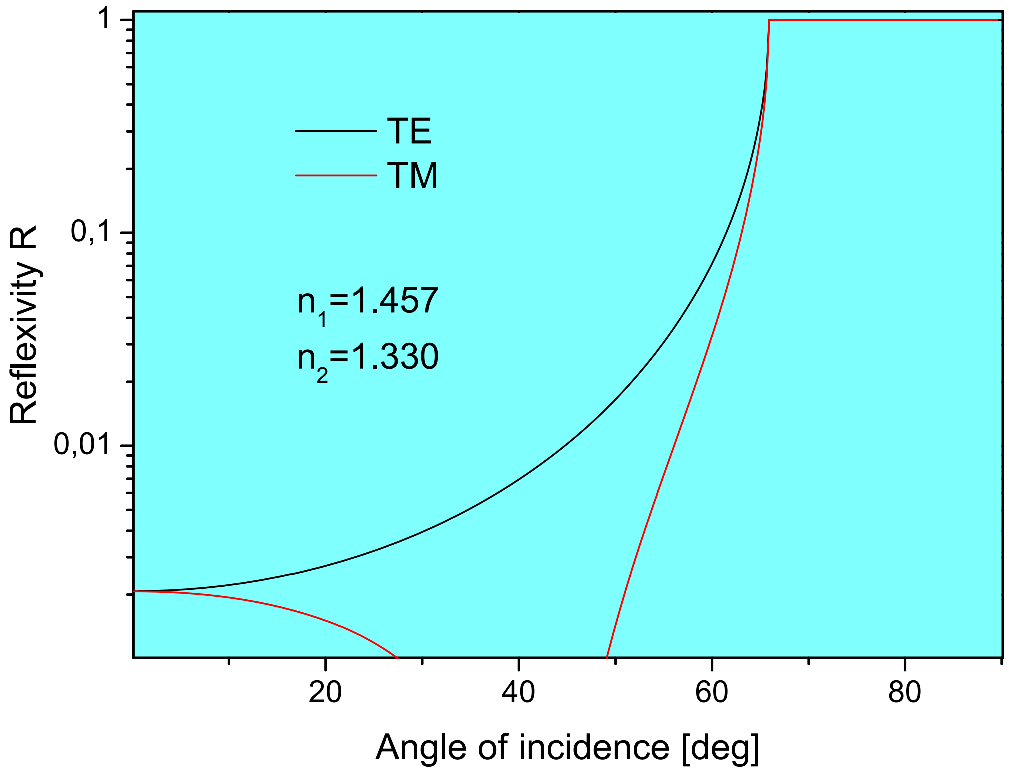
REFLECTION FOCS - PRINCIPLE





REFLECTION SENSOR - LIMITATIONS

Low reflectivity due at small angle θ (%) – necessary to use approaches for R increase



Evanescent sensor
θ > 80deg, R>0.9

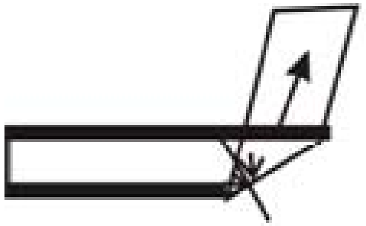
Reflection sensor
θ ~ 0 deg, R = 0.04

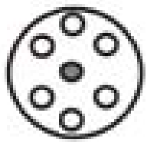
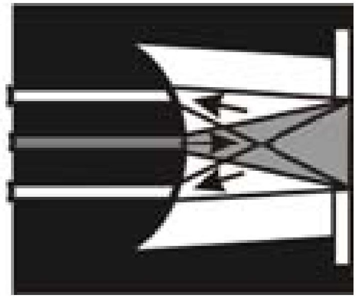
$$R = \left(\frac{n_1 - n_2}{n_1 + n_2} \right)^2$$

IMPROVEMENT OF REFLECTIVITY

Small R + Small reflection area → low intensity of reflected light

Different approaches are used for improving intensity of reflected light – bebled fiber ends, fiber bundles, mirrors

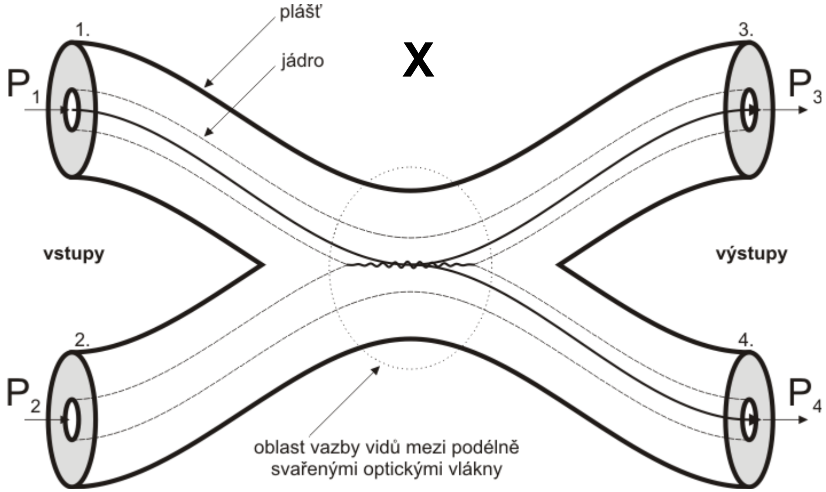


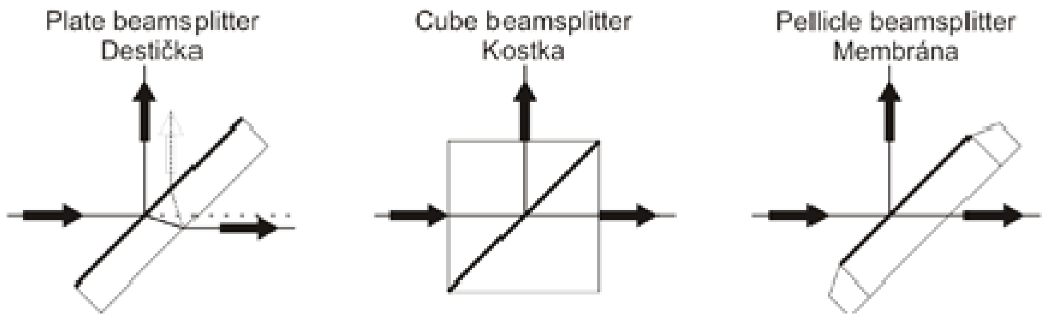



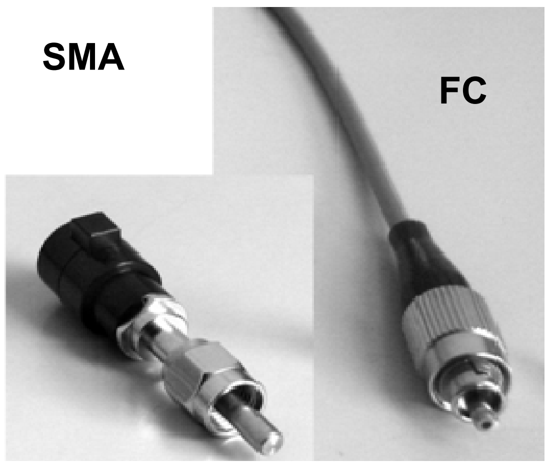
U. Utziger et al., „Fiber-optic probes for biomedical optical spectroscopy“, J. Biomedical Optics 8(1) (2003) 121–147

INSTRUMENTATION

Fiber couplers (X, Y), beam splitters, connectors



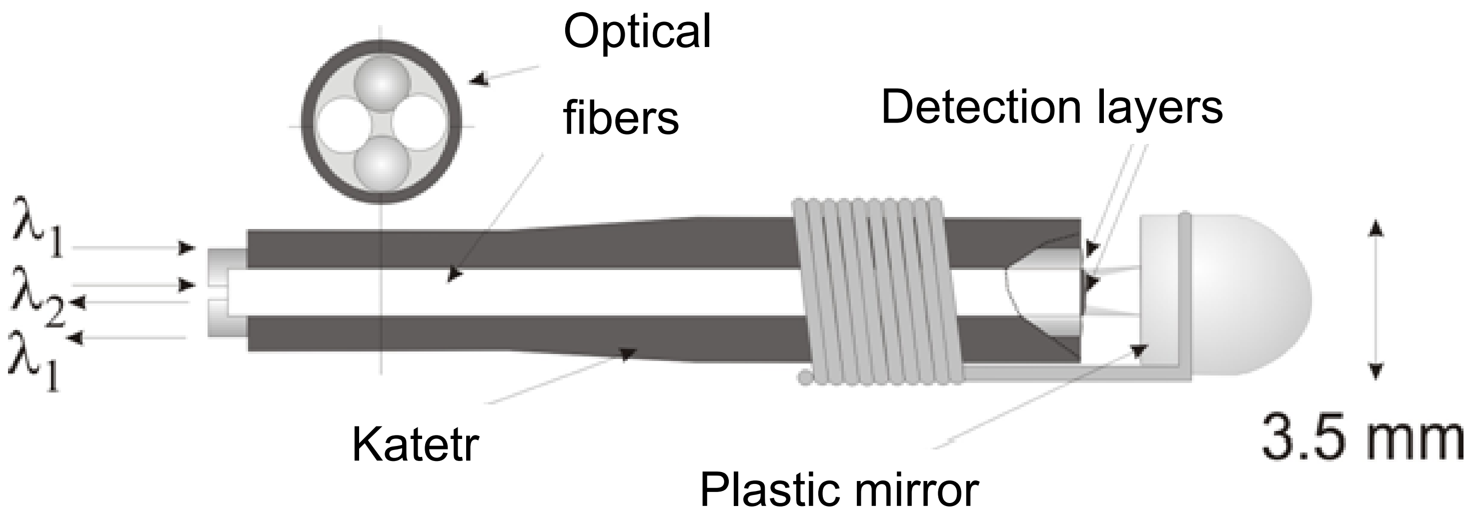




Commercially available

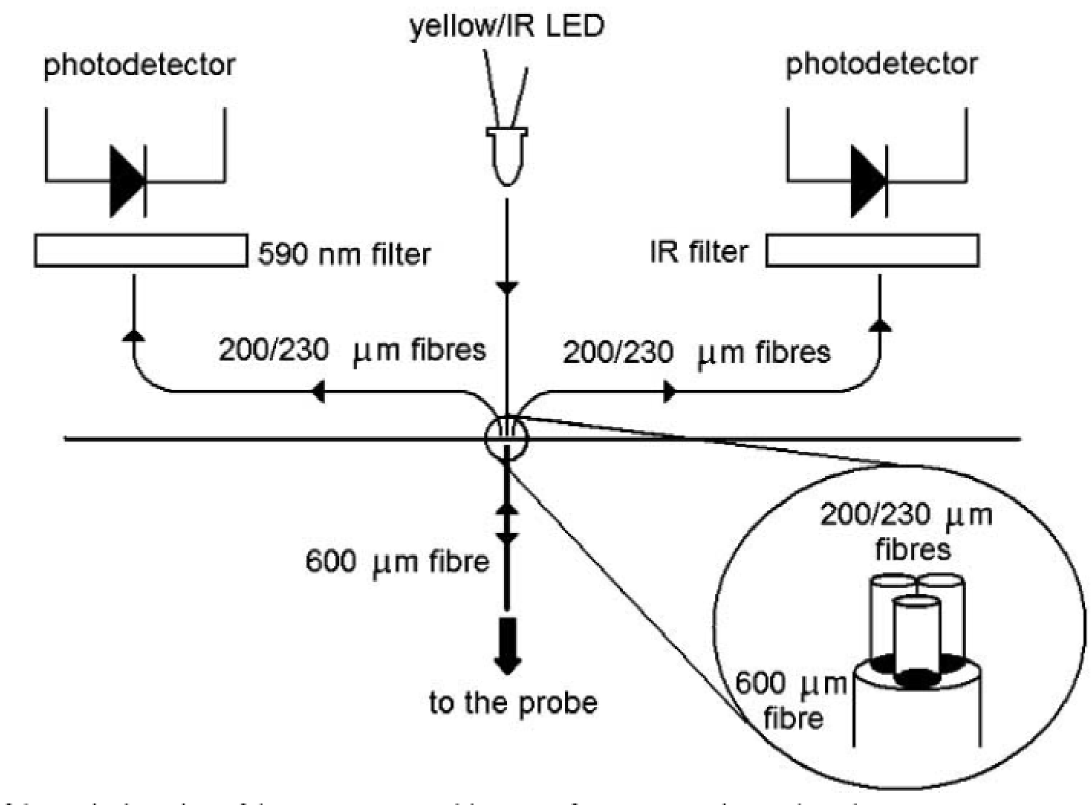
REFLECTION SENSOR FOR pH IN STOMACH

Absorption pH transducers bromophenol blue and thymol blue (pH range 1-8)



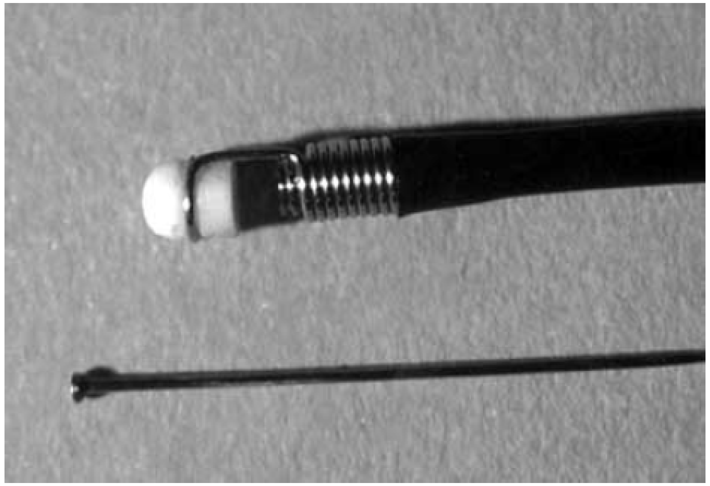
CNR Florence, Italy, F. Baldini, A. N. Chester, J. Homola, S. Martelluci: „*Optical chemical sensors*“, Springer (2006)

REFLECTION SENSOR OF BILIRUBIN



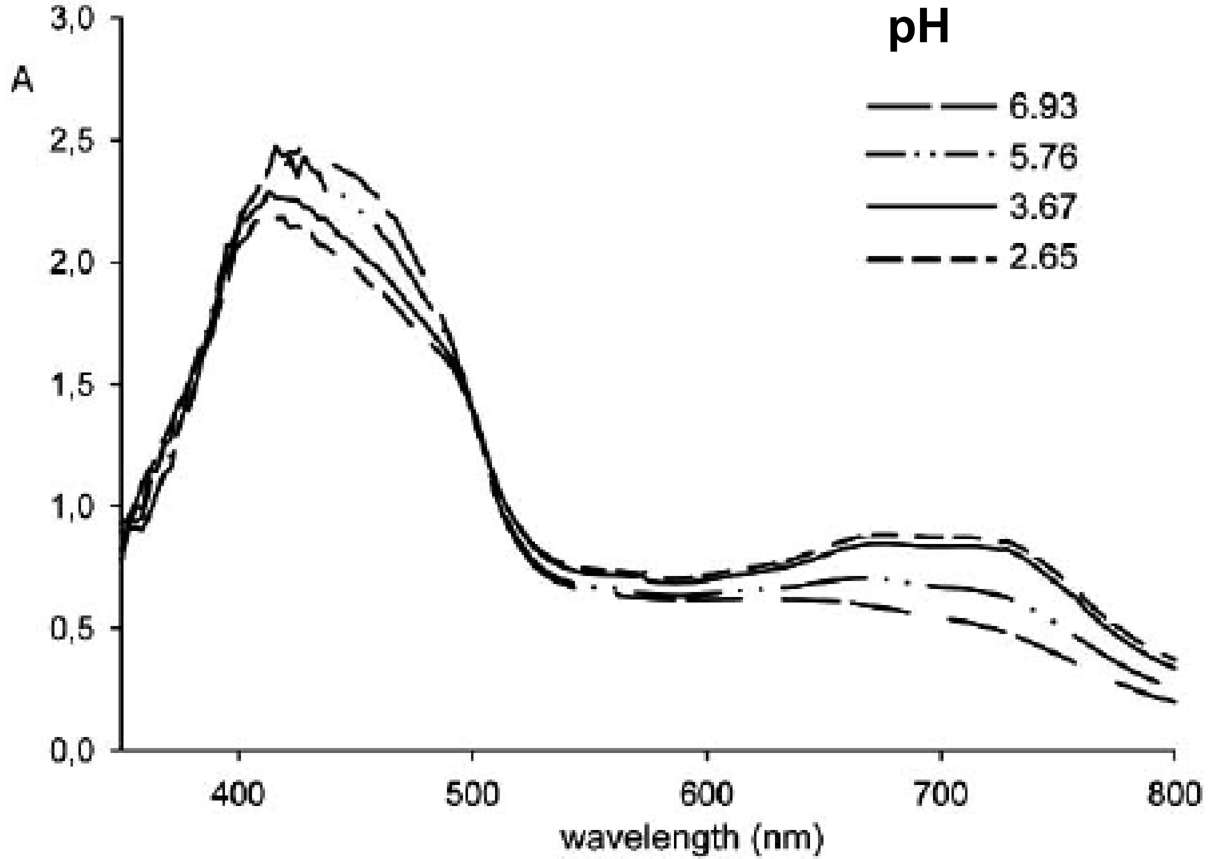
Measurement of absorption spectra of bilirubin

No transducer



F. Baldini et al., *Current Analytical Chemistry,* 2008, *4,* 378-390 (for detection of carbon dioxide in stomach)

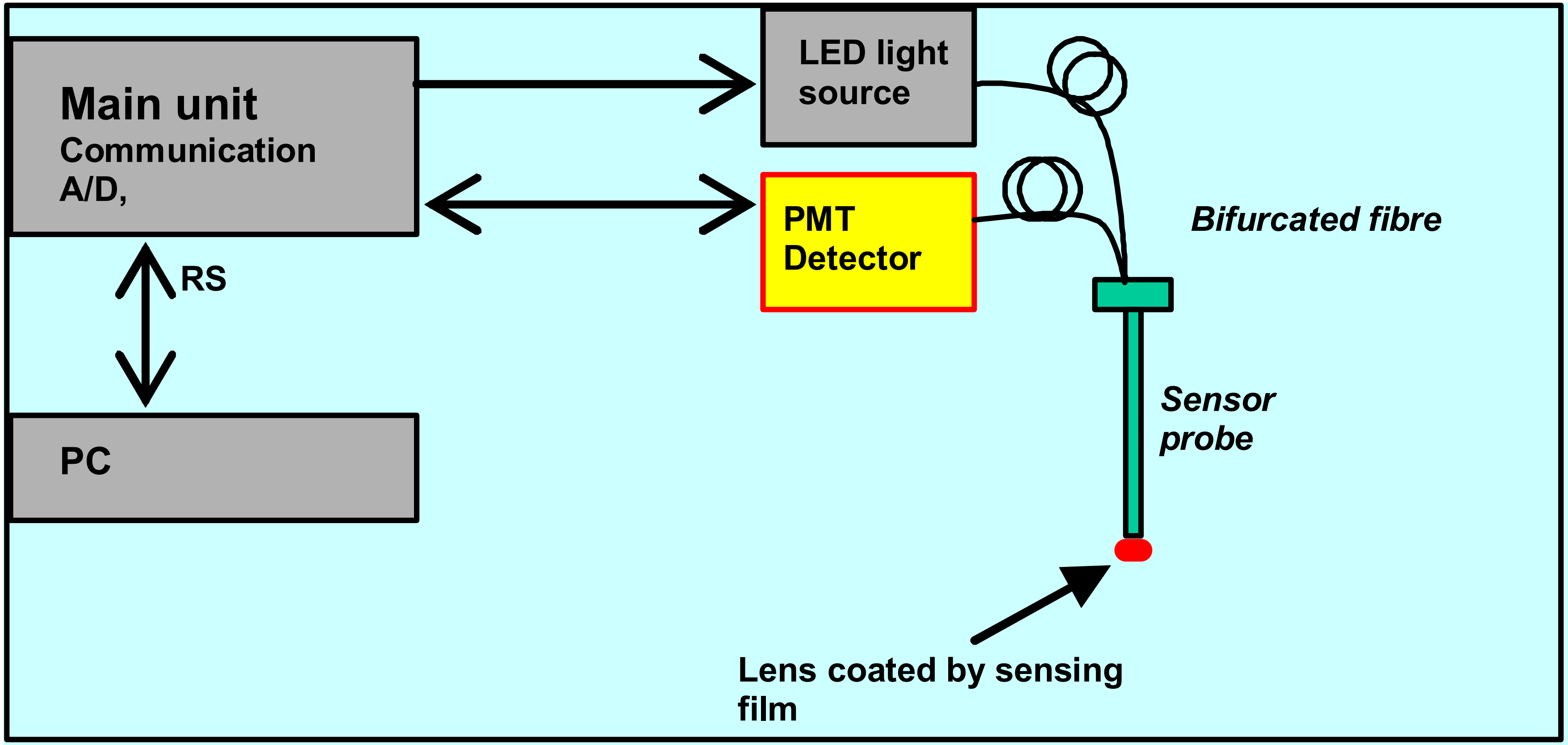
ABSORPTION SPECTRUM OF BILIRUBIN



Calibration:

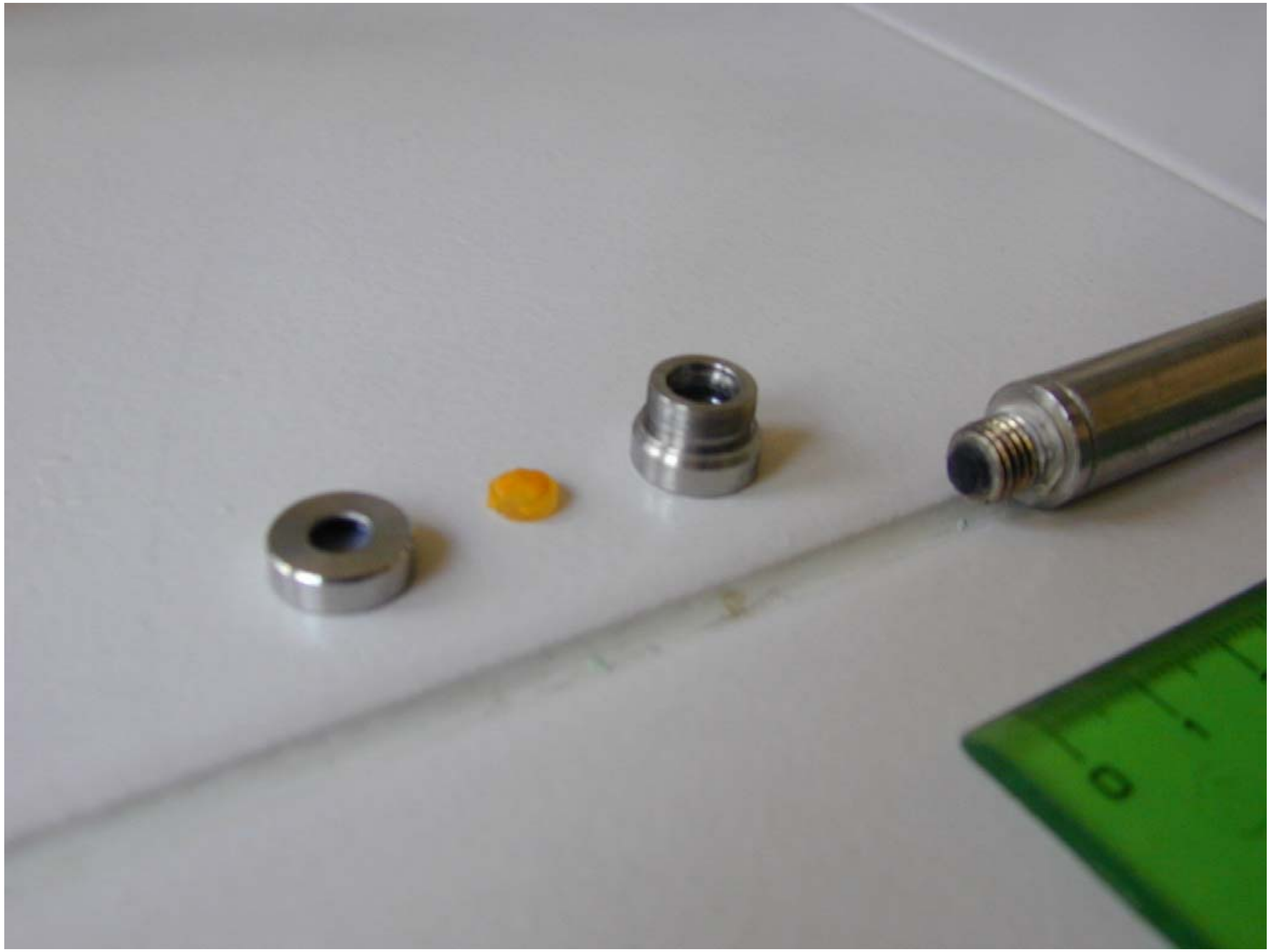
absorbance 470nm (measurement)/ 590 nm (reference)

IPE REFLECTION SENSOR OF OXYGEN, GLUCOSE



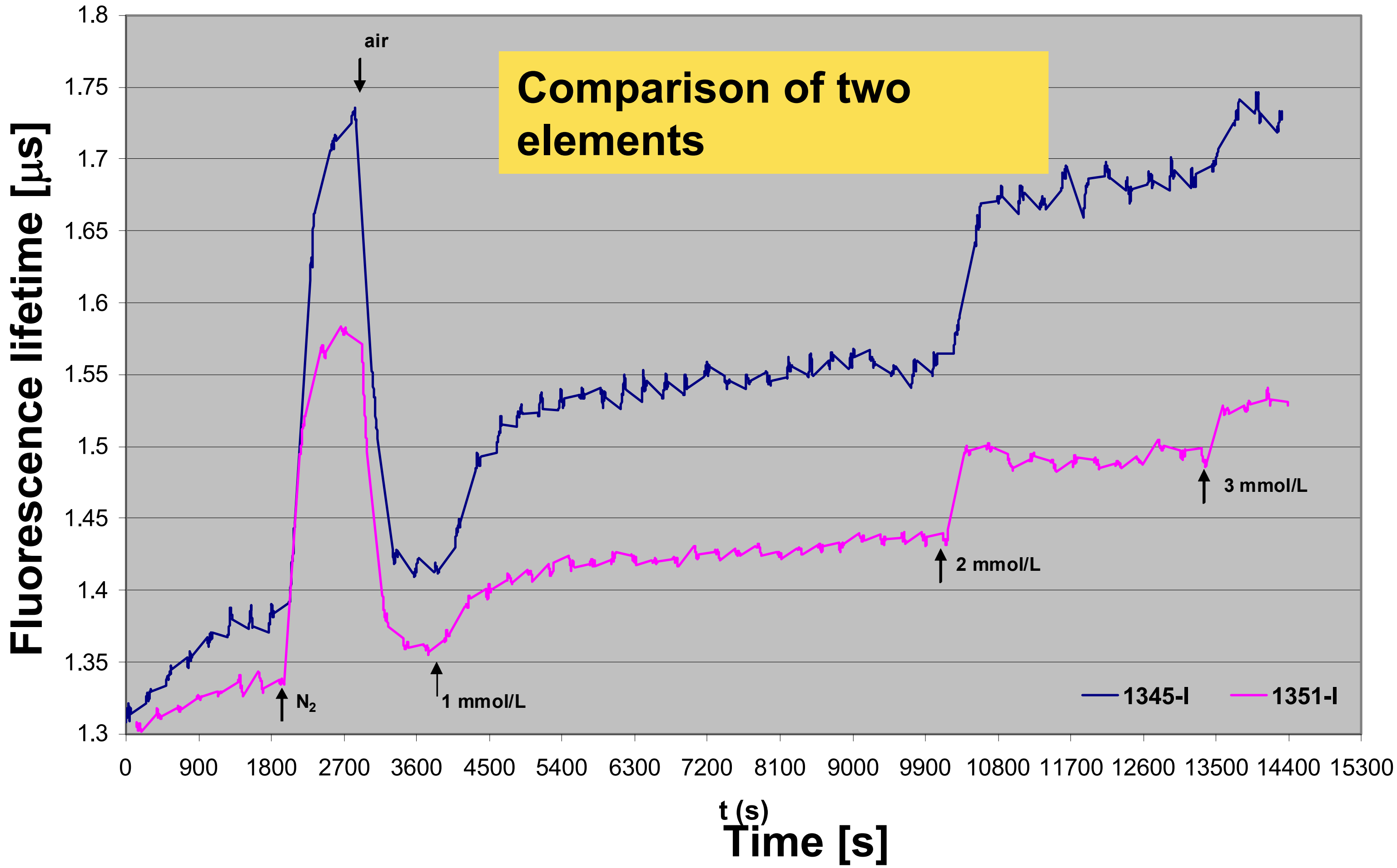
Oxygen – quenching of fluorescence Ru complex
Glucose – oxygen detection in enzymatic reaction

SENSING HEAD AND DETECTION LENS



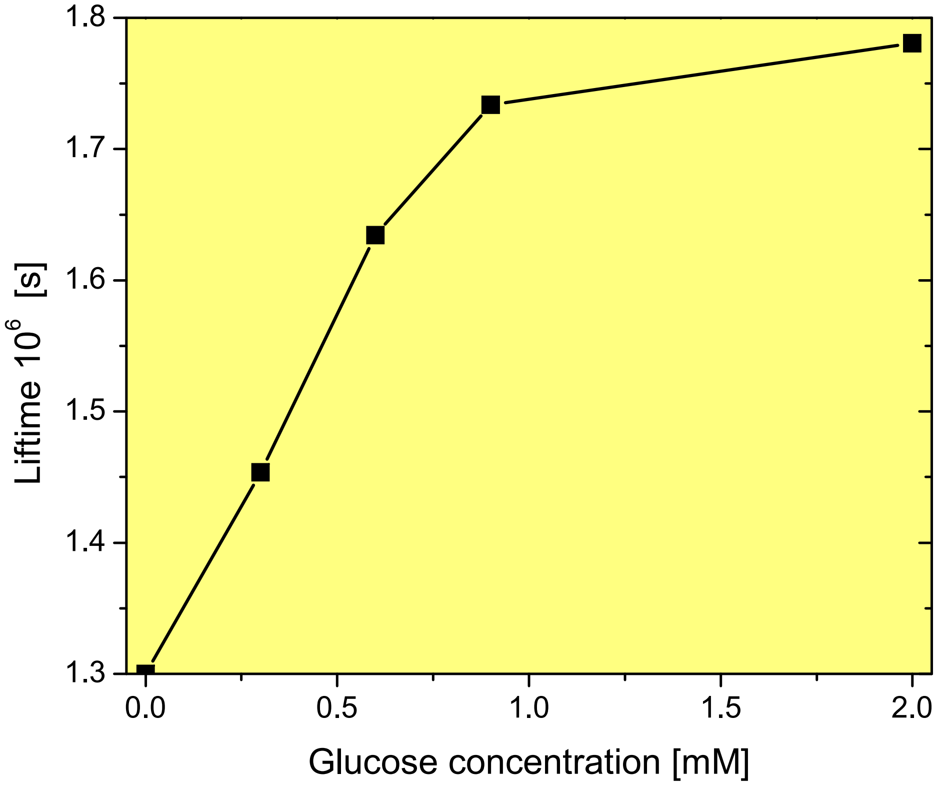
Fluorescence lifetime measurements

TIME RESPONSE TO OXYGEN AND GLUCOSE



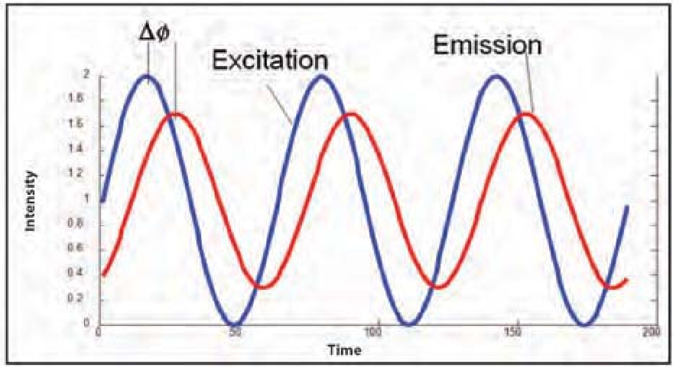
Glocose + oxygen $\longrightarrow^{\text{glucose oxidase}}$ gluconic acid + peroxide

GLUCOSE SENSOR - CALIBRATION CURVE



Detection limit 0.2 mM

COMMERCIAL OXYGEN SENSORS



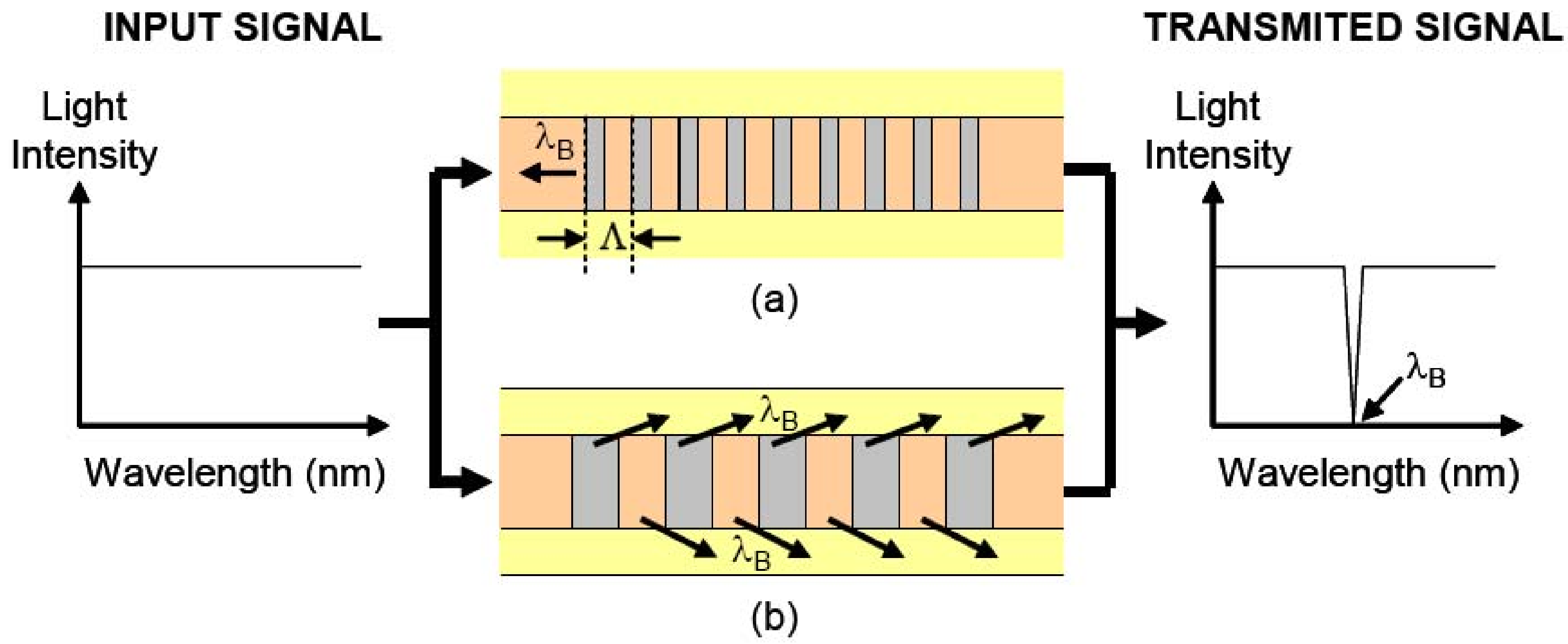
Fluorescence lifetime measurements, phase modulation

Detection elements

www.oceanoptics.com/Products

Detection of oxygen in plants

OPTICAL GRATINGS IN FIBERS



Longitudinal periodic changes of refractive index in fiber → light wave is reflected on grating at resonance condition:

(a) Bragg gratings (FBG) $\lambda_B = 2n_{eff}\Lambda_B$ **($\Lambda_B << \lambda$)**

(b) „Long Period Gratings (LPG)“ $\lambda_{LPG} = (n_{CO} - n_{cl}^{m})\Lambda_{LPG}$ **($\Lambda \sim 10^2$ μm)**

GRATING PREPARATION AND PROPERTIES

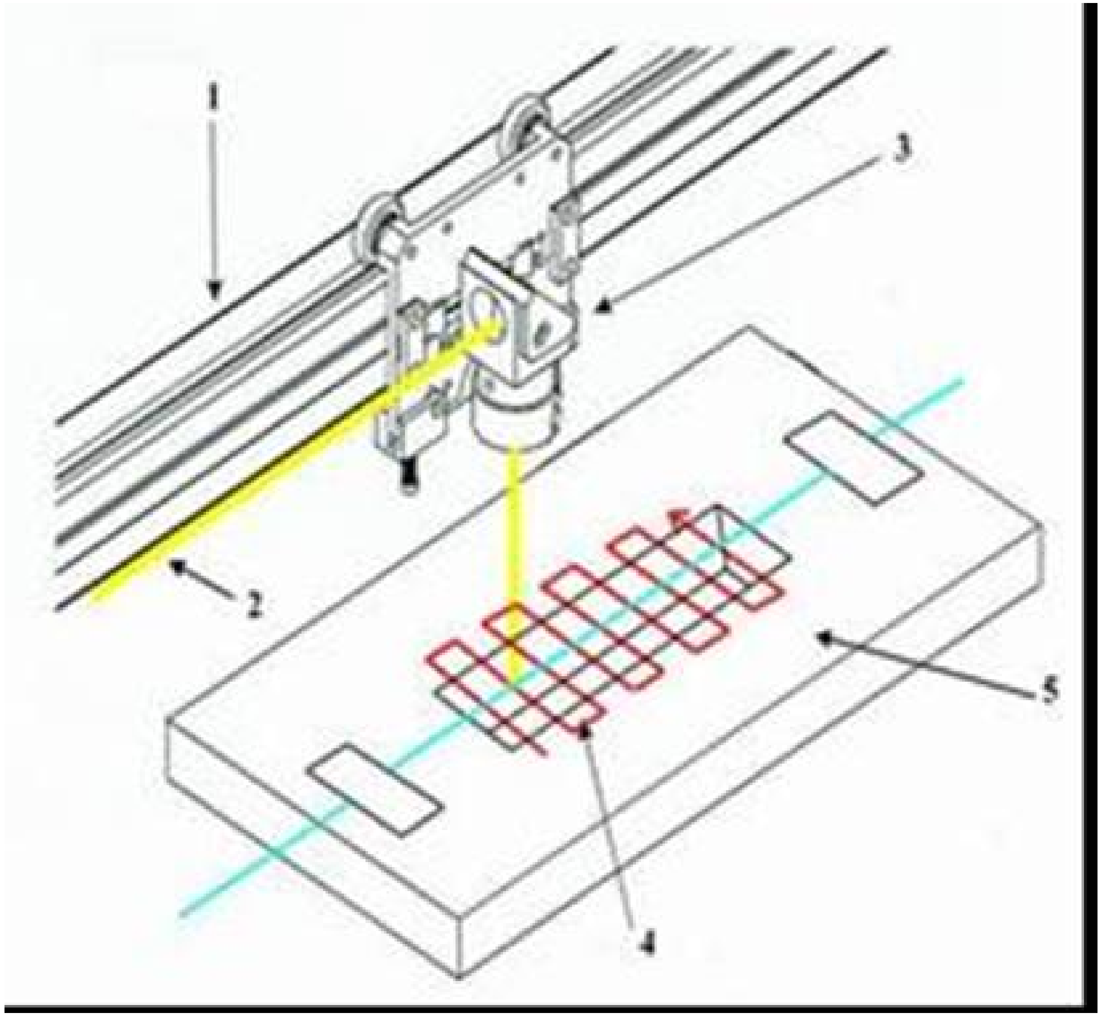
- FBGs - inscription by UV lasers into fibers saturated by hydrogen

  Sensitive to **effects influencing the core** – temperature, strains
- LPGs - inscription by CO_2 laser

  Sensitive to **effects influencing both core and cladding** – temperature, stresses, refractive index – chemical detection

K.O. Hill,Gerald Meltz, J. Lightwave Technol. 15, 1263-1276, 1997
J. Canning, J. Sensors 2009, Article ID 871580, 17 stran - vlákna
I. J. G. Sparrow, J. Sensors 2009, Article ID 607647, 12 stran - planární

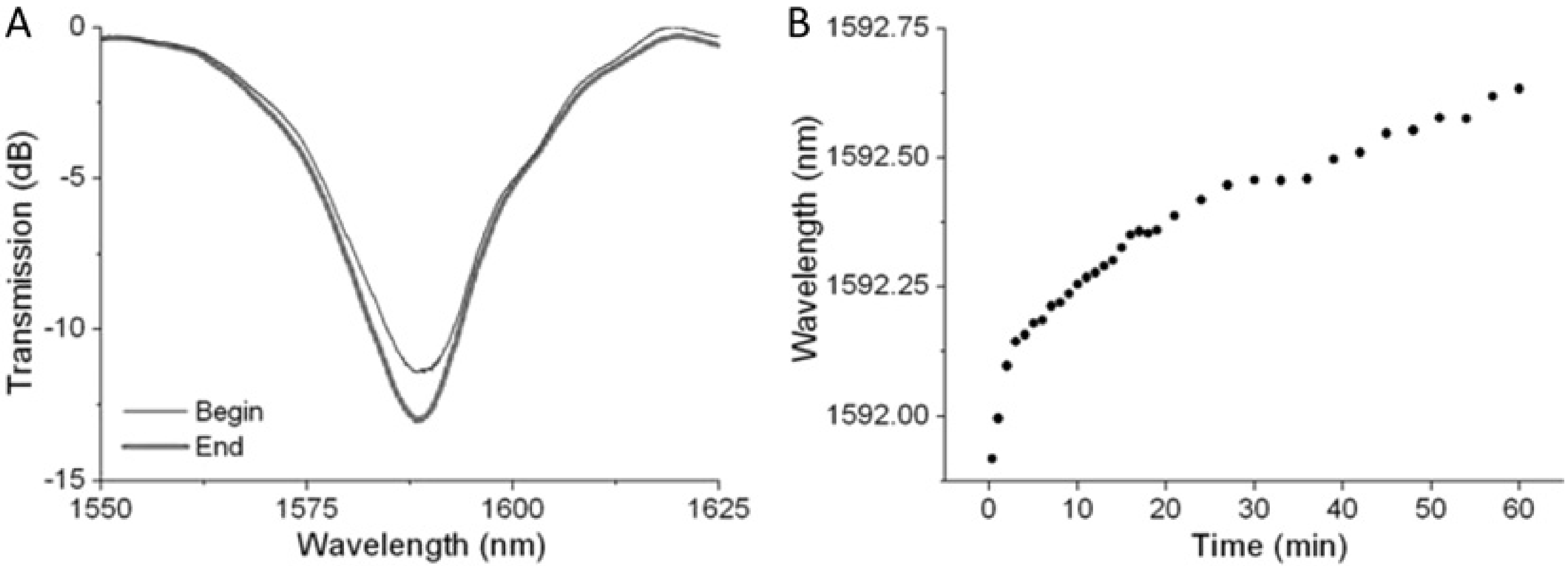
IPE PREPARATION OF LPGs



CO_2 laser

Beam crossing through the fiber heats up the fiber → changes of mechanical stresses → change of RI due to photoelasticity

DNA DETECTION BY LPG IN SM FIBER

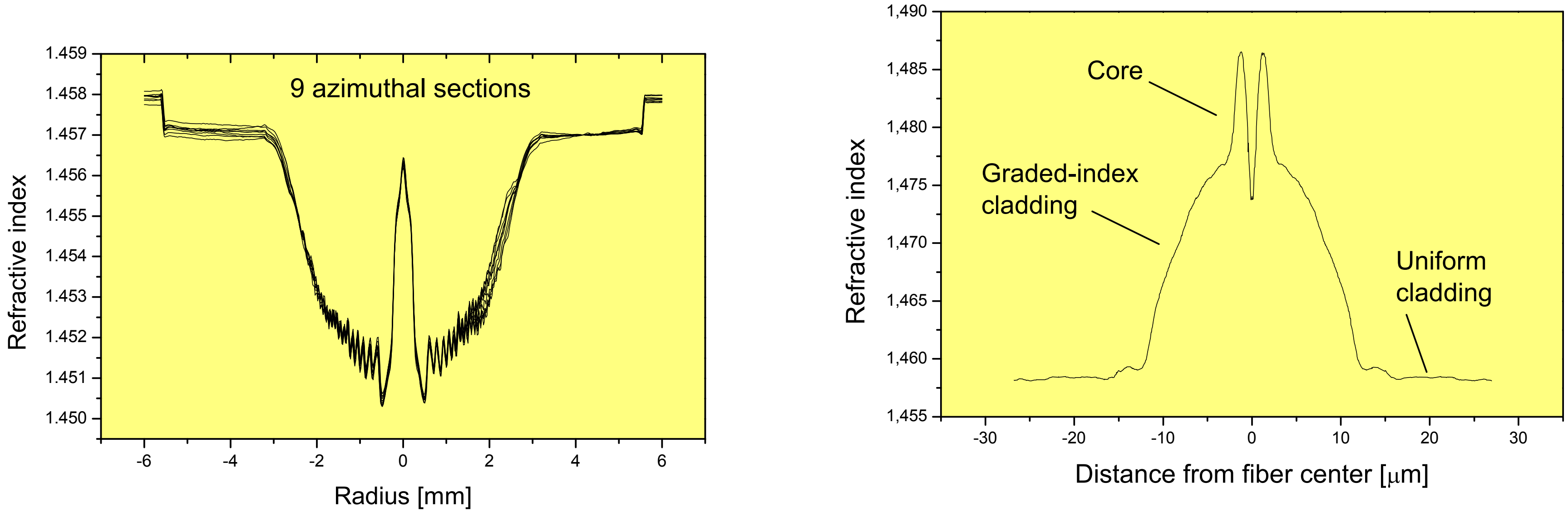


Λ=161 μm

1 μM ssDNA (5-GCACAGTCAGTCGCC-NH2-3) in PBS buffer

A.V. Hine et al., Biochem. Soc. Trans. (2009) 37, 445–449

IPE LPGs IN GI AND IGI FIBERS

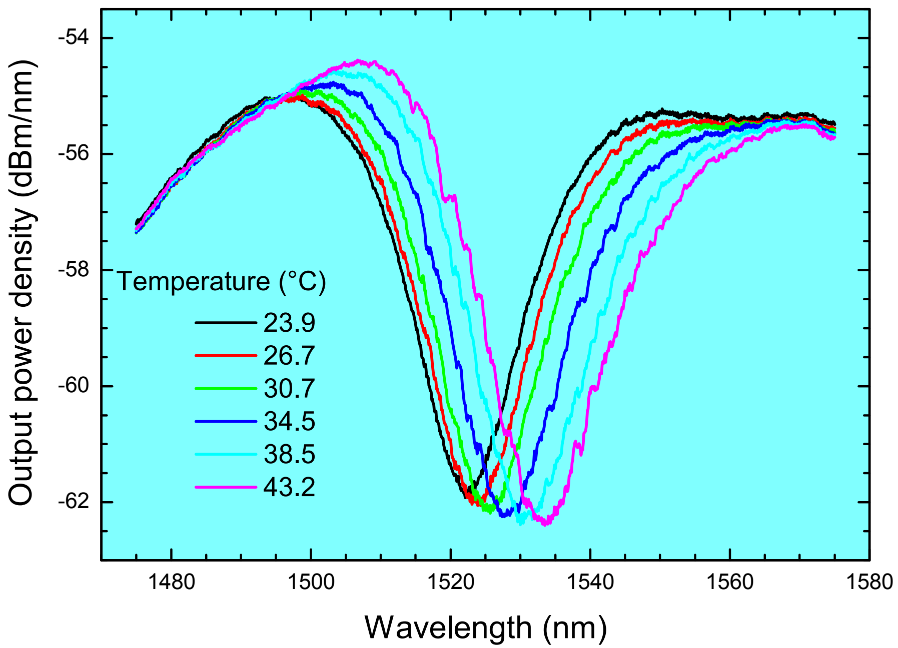


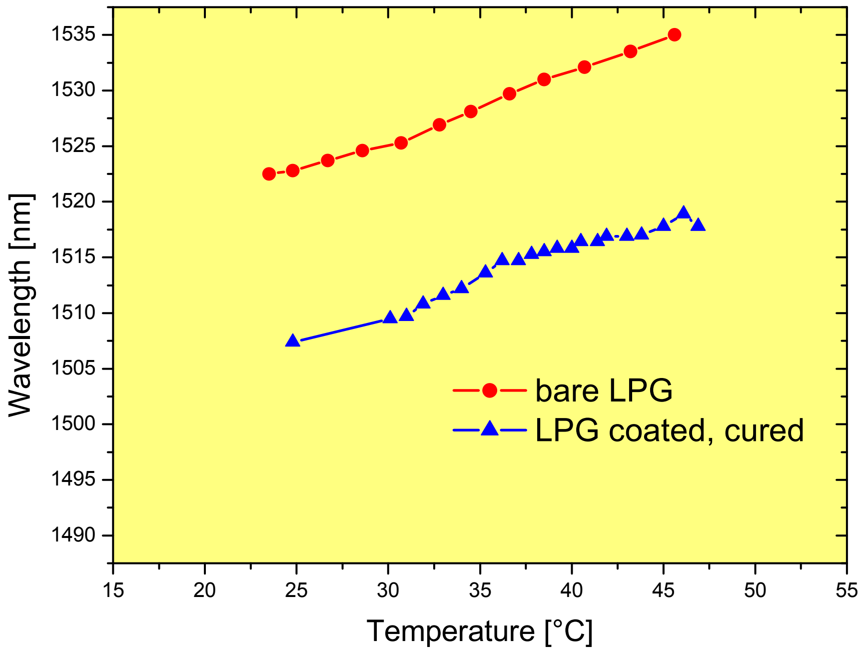
SM core, IGI, GI cladding allow to control detection selectivity to temperature, stresses

LPGs in standard SM fibers sensitive to a number of effects

F. Todorov et al., Sensor Lett. 7, 979-983 (2009)

LPG TEMPERATURE SENSITIVITY



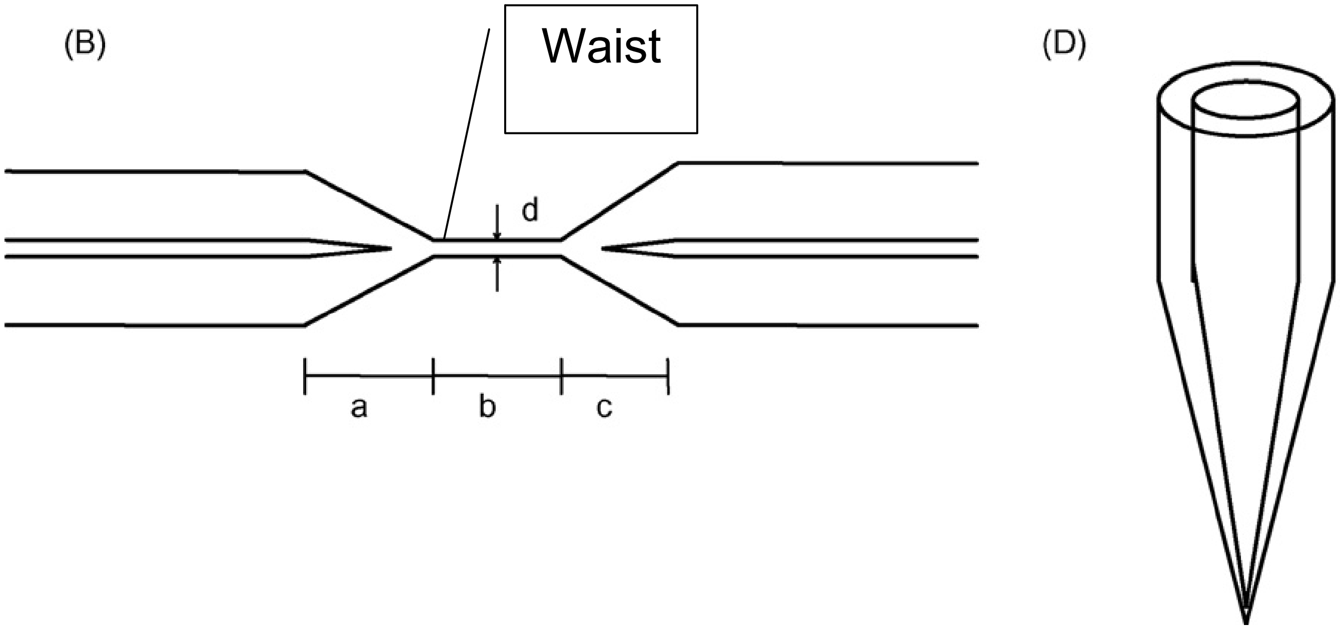


Λ_B=500 μm; Sensitivity 0,5 nm/˚C

OPTICAL MICROSENSORS

- **Fiber-optic tapers**
- **Optical nanoparticles – PEBBLE -** *„probes encapsulated by biologically localized embedding“;*
- **Microresonators with whispering gallery modes (WGM)**

FIBER-OPTIC TAPERS

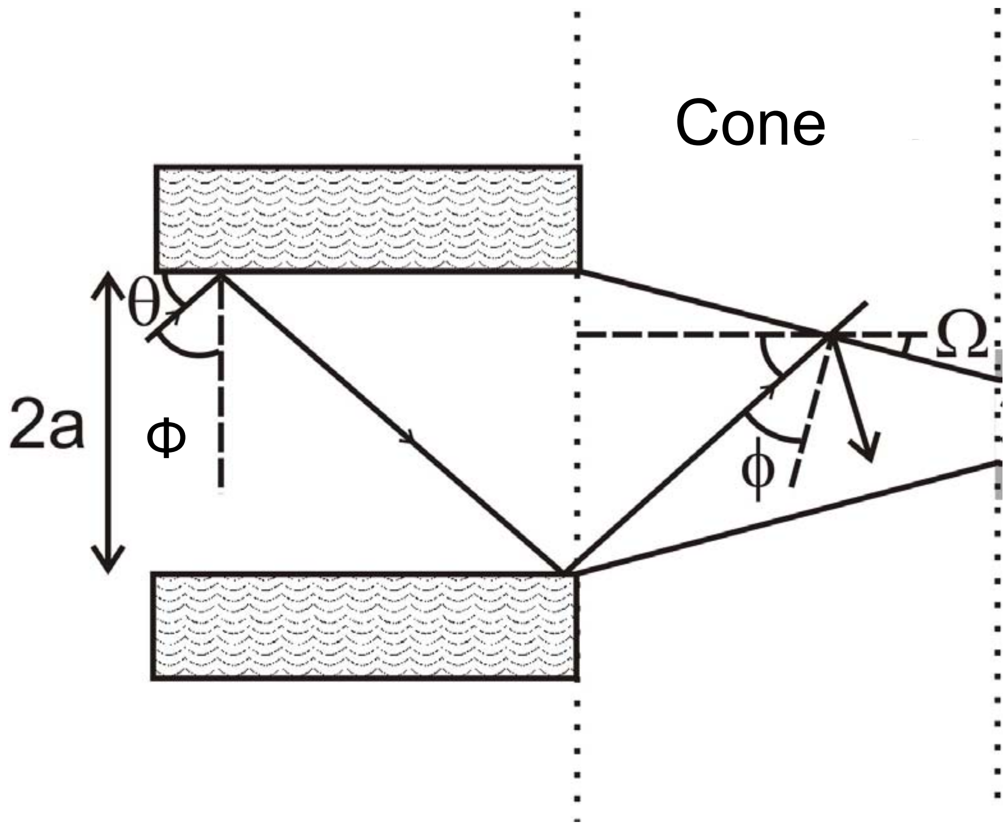


Make possible to decrease core diameter and angle θ on the core/cladding boundary → sensitivity increase.

Tapers with two cones (B) –"biconical tapers"

Tapers with one cone (D)- "fiber tips"

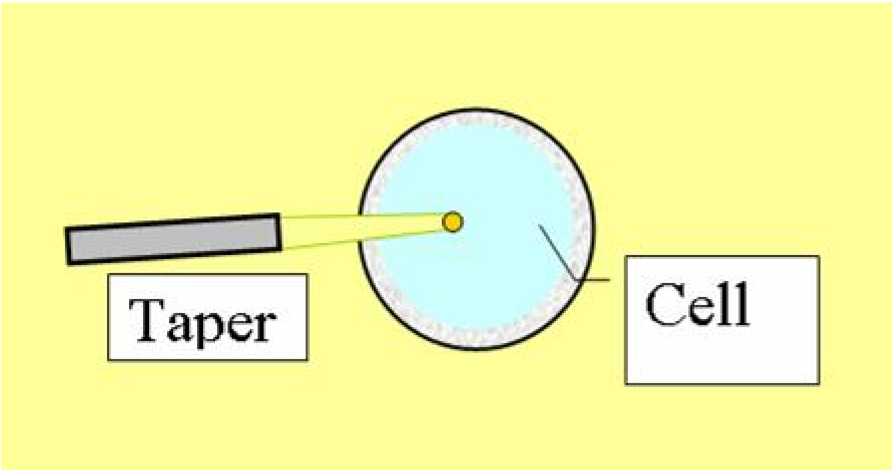
TAPER – PRINCIPAL SCHEME



d<2a, $\Phi < \theta \rightarrow$ increased power in evanescent wave

SENSORS ON FIBER TIPS

Optical fiber elongated into a tip that is coated with optical transducer



Tips sensitive to potassium in mouse oocyte cells (dimension 100μm)

S.M. Buck et al. , Talanta 63 (2004) 41

SENSORS ON FIBER TIPS - REFERENCES

- **R. Kopelman et al. (USA)**

E. J. Park et al., J. Mater. Chem. **15** (2005) 2913 – oxygen detection
M.R. Shortreed et al., Sens. Actuators **B 38-39** (1997) 8 – K detection

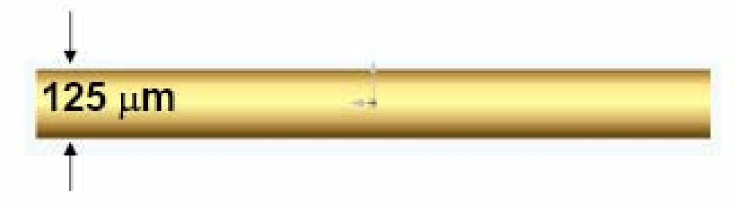
-T. Vo-Dingh et al., (USA)

T. Vo-Dingh et al., J. Nanoparticle Research **2** (2000) 17-
B.M. Cullum et al., Tibtech September **18** (2000) 388-review
T. Vo-Dinh et al., Anal Bioanal Chem **382** (2005) 918 -review
B. M. Cullum et al., Analytical Biochemistry **277** (2000) 25– benzo[a]pyrene tetrol in cancer cells

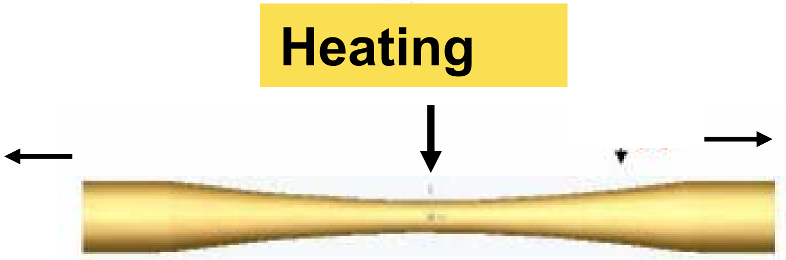
Optical sensors

O.S. Wolfbeis, J. Mater. Chem. **15** (2005) 2657– fluorescence sensors

FIBER TIP PREPARATION – THERMAL ELONGATION



Input optical fiber



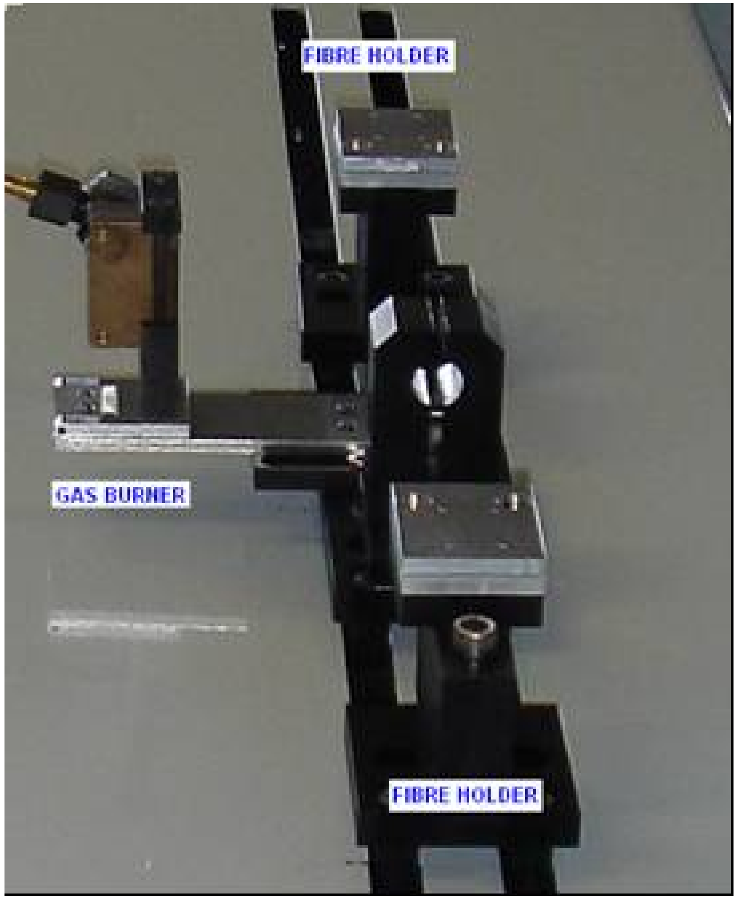
Heating up and elongation of fiber by a burner or CO_2 laser

Coating the taper with a metallic protective layer

Precise cutting

T. Martan et al., Proc. SPIE 7138 (2008), Article 71380Z DOI: 10.1117/12.818000

IPE TAPERIN APPARATUS



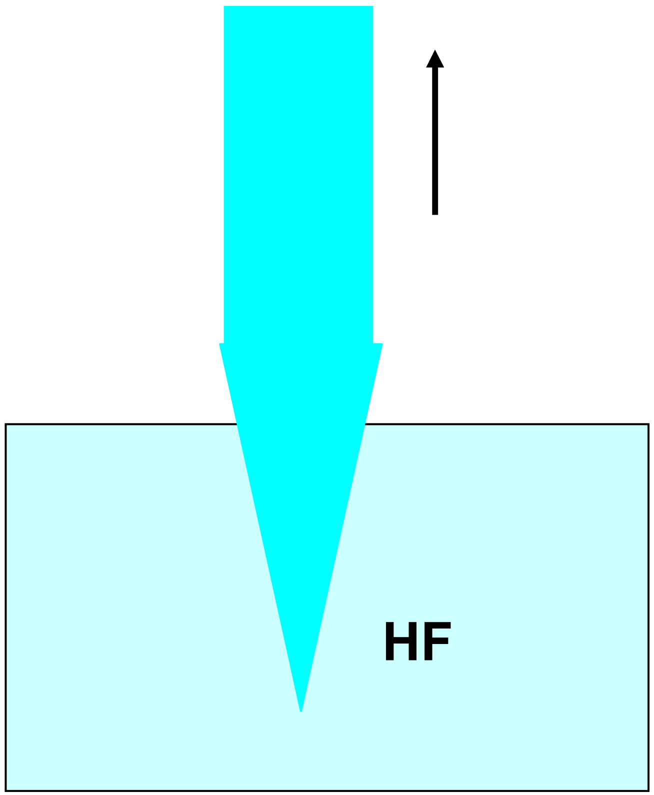
Gas burner

PC-controlled elongation

Al coating prepared by spattering

T. Martan et al., Proc. SPIE 7138 (2008), Article 71380Z DOI: 10.1117/12.818000

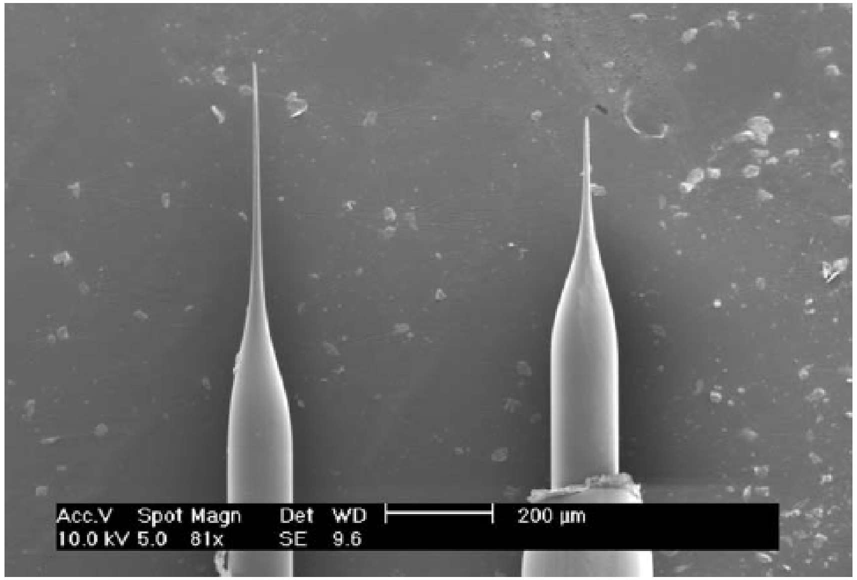
FIBER TIPS PREPARED BY CHEMICAL ETCHING



Immersing silica fiber in a solution of hydrofluoric acid and its slow withdrawal a tip at the fiber end is formed

N. Nath et al., J. Anal. Toxicology 23 (1999), 460-467

TIP PHOTOS - LITERATURE



Tip diameter 50 nm

B. Cullum et al., Tibtech September 18 (2000) 388-review

E. J. Park et al., J. Mater. Chem. 15 (2005) 2913 – detection of oxygen

APPLICATION OF DETECTION LAYER ON TIP

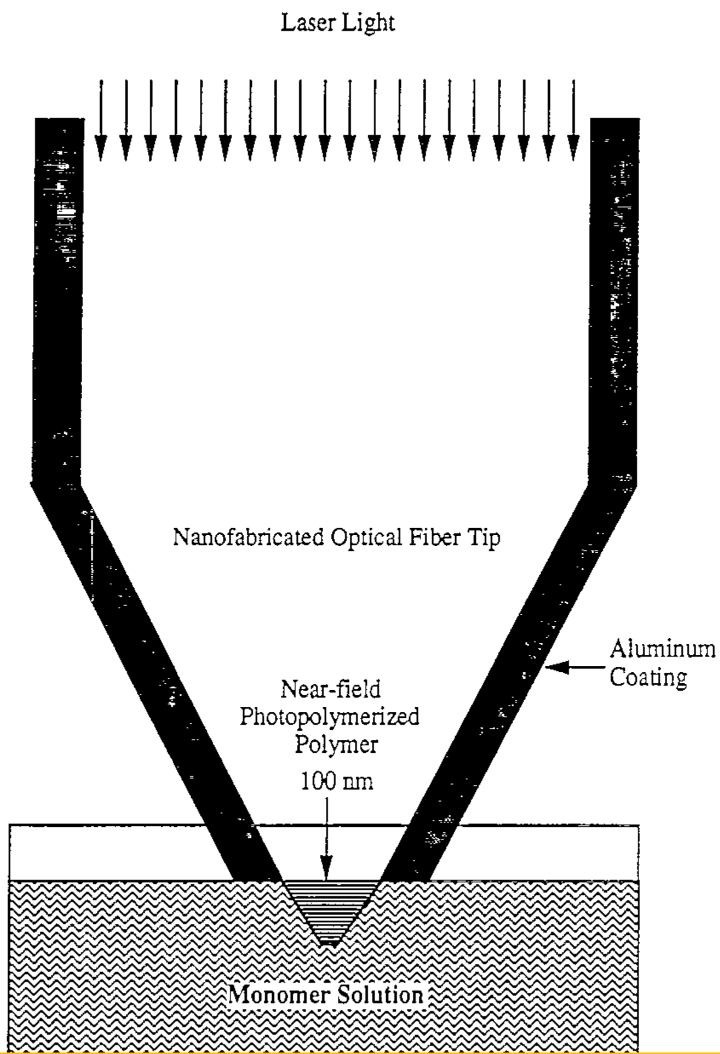
Photopolymerisation

Monomer (UV acrylate) solution containing transducer

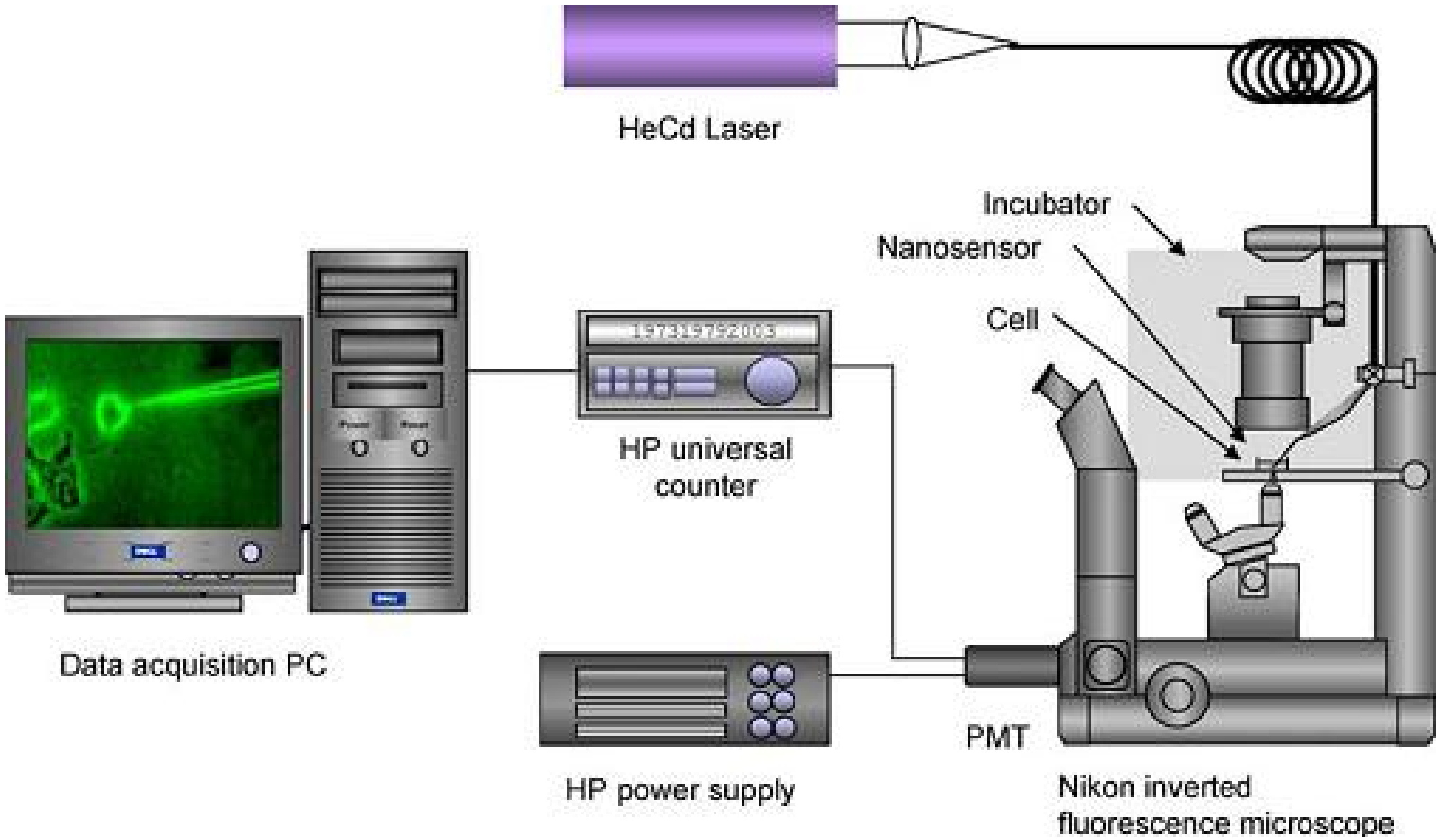
Launching UV radiation into the fiber a small amount of monomer is cured at the tip end

Only end of the tip coated

W. Tan et al., Sens. Actuators B28 (1995) 157 - pH

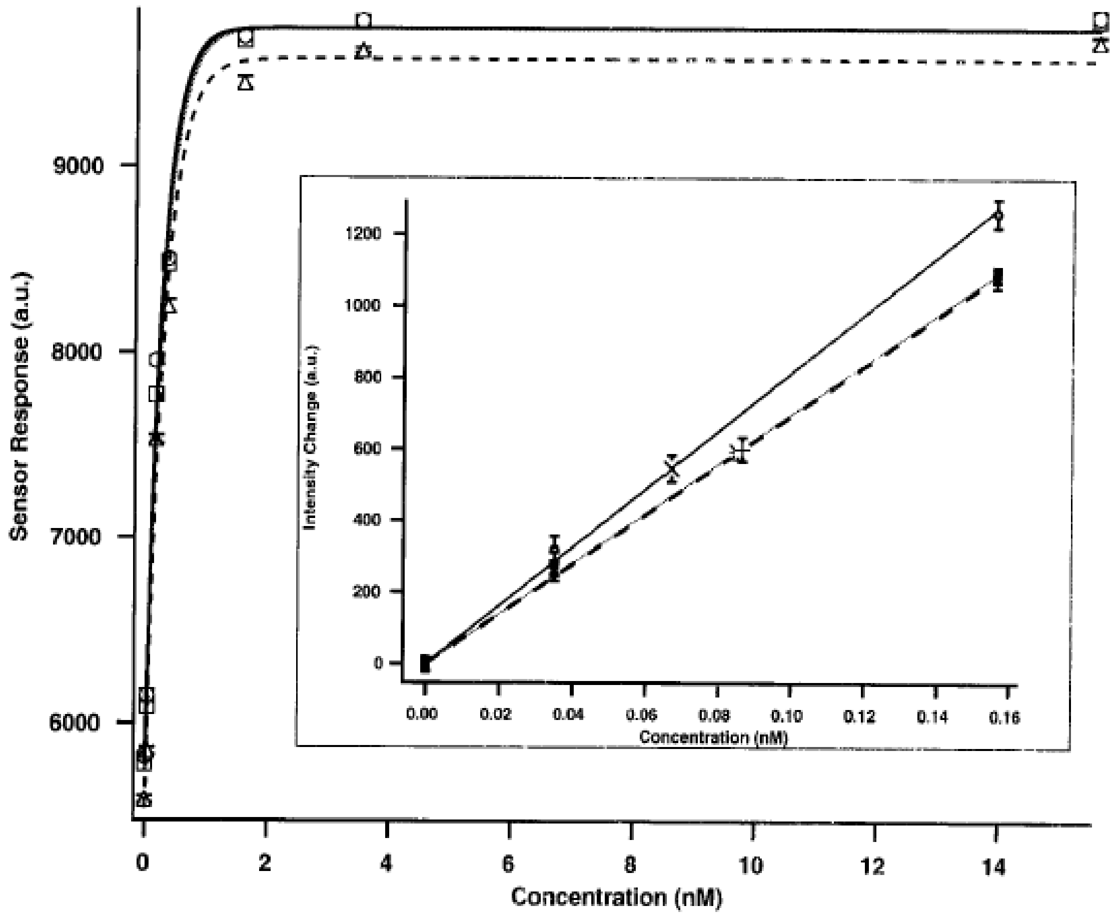


Inverted fluorescence microscope



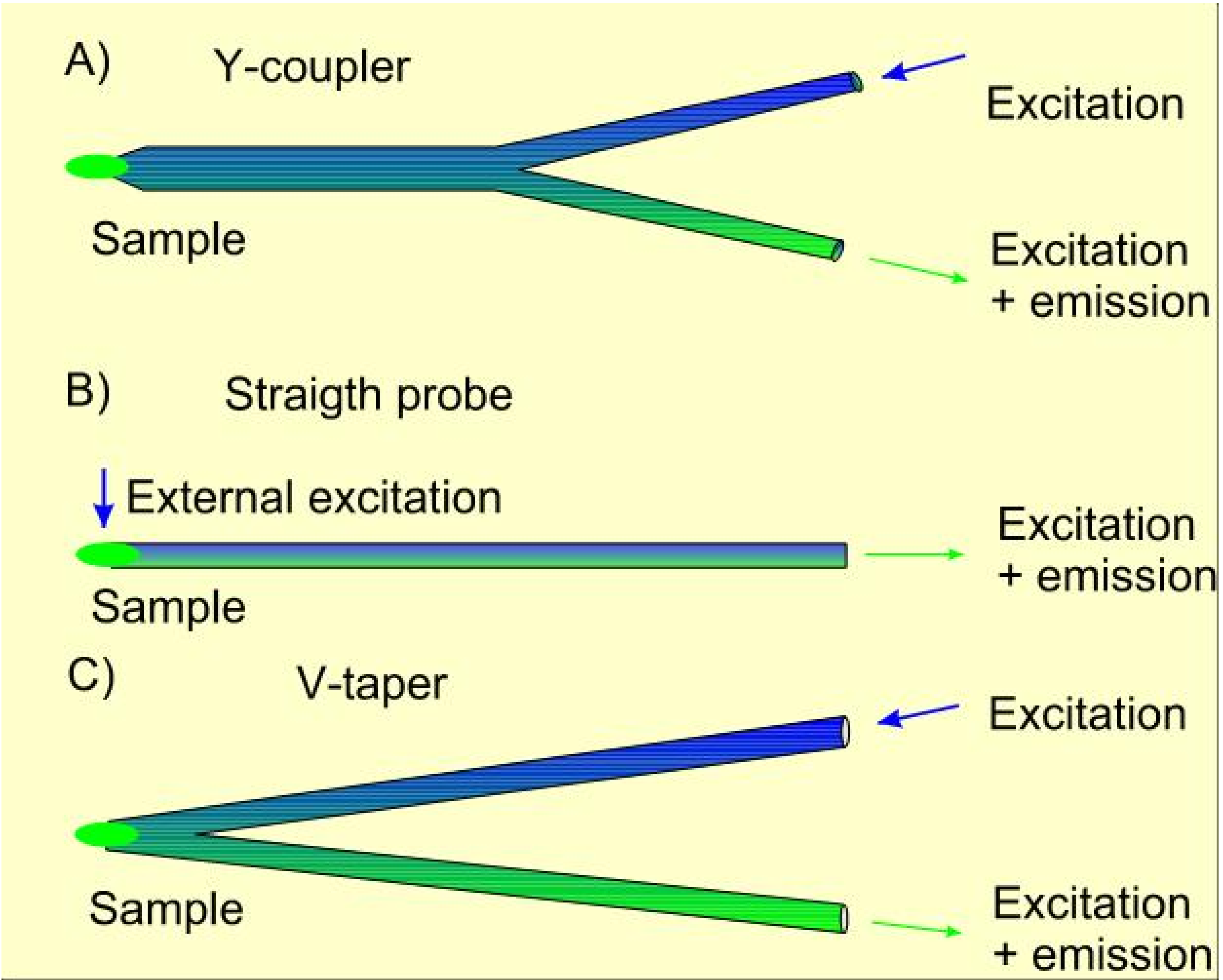
T. Vo-Dinh et al., Anal Bioanal Chem 382 (2005) 918

DETECTION OF CANCER MARKERS



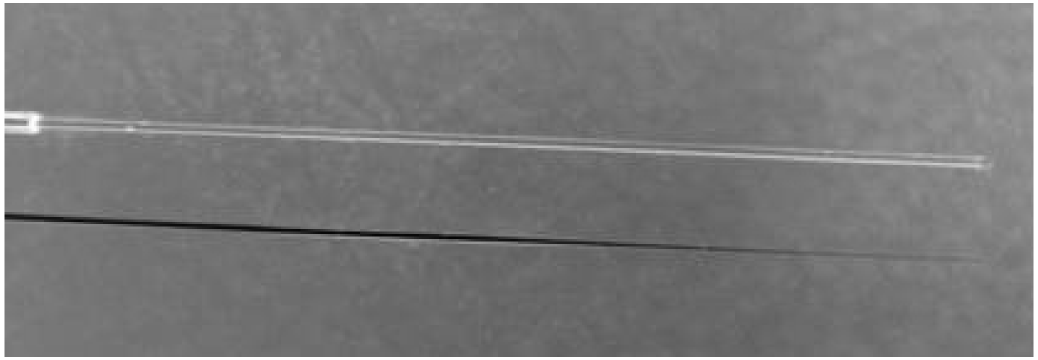
B. M. Cullum et al., Analytical Biochemistry 277 (2000) 25 – Auto fluorescence of benzo[a]pyrene tetrol in rat liver cell

IPE APPROACH - REFLECTION ARRANGEMENT

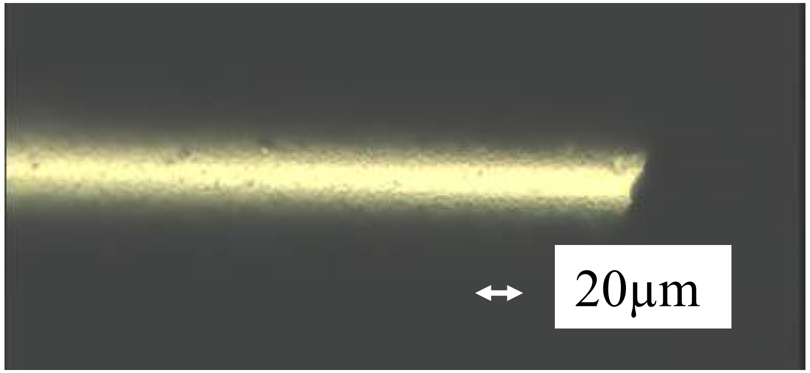


IPE TAPERS

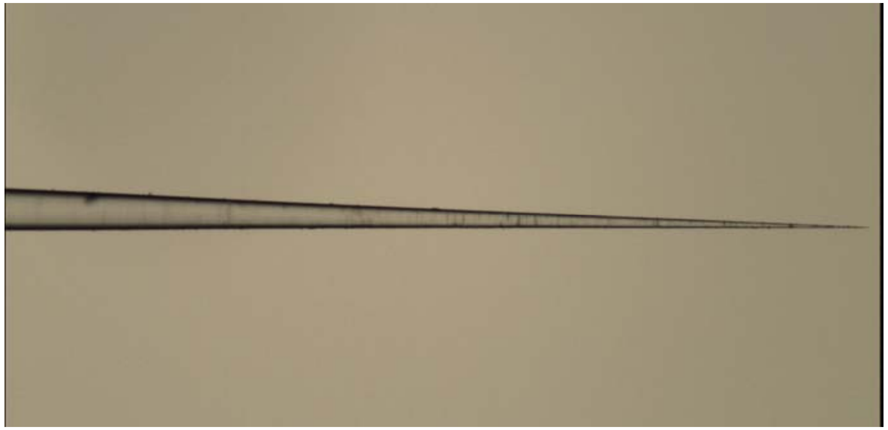
Taper coated with ITO coating and input fiber



Taper coated with Al



Taper coated with ITO, tip diameter 2 µm



pH DETECTION MEMBRANES APPLIED ON TIP

Porous membrane with fluorescence pH transducers BCECF or HPTS

HPTS= 8-hydroxypyrene-1,3,6-trisulfonic acid trisodium salt (λ_{exc} =430 nm, λ_{em} = 480 nm)

BCECF=2',7'-Bis(2-carbonylethyl)-5(6)-carboxyfluorescein (Aldrich 14560) (λ_{exc} =473 nm, λ_{em} = 540 nm)

<u>Porous membrane</u>

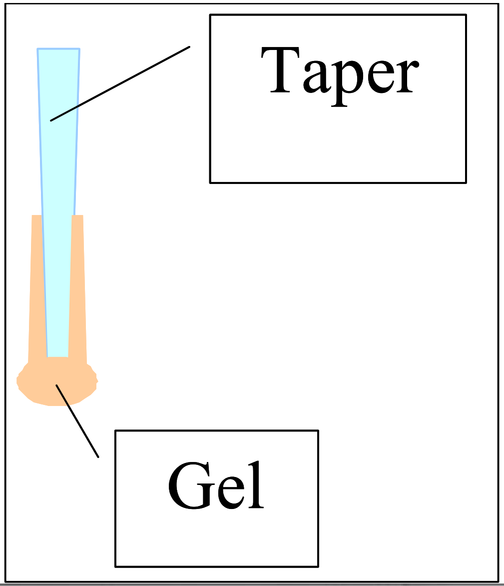
– TEOS, HCl, R_W=2, BCECF

– Propyltriethoxysilane + (3-glycidoxy)trimethoxysilane, HCl, R_W=2, HPTS, Ru phenantroline chloride

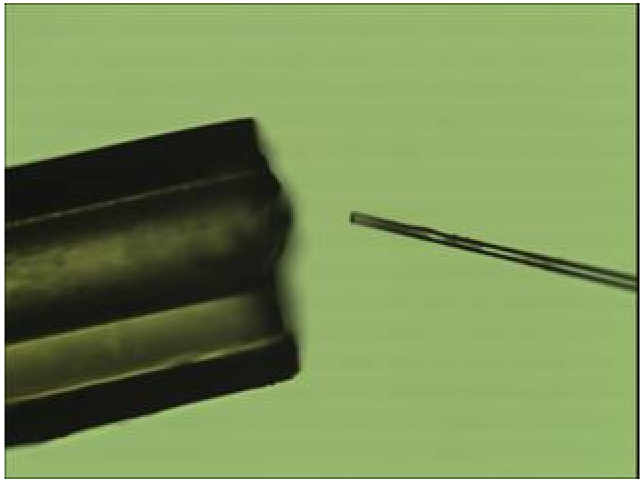
Gel layers dried at 70 ºC, 24 h

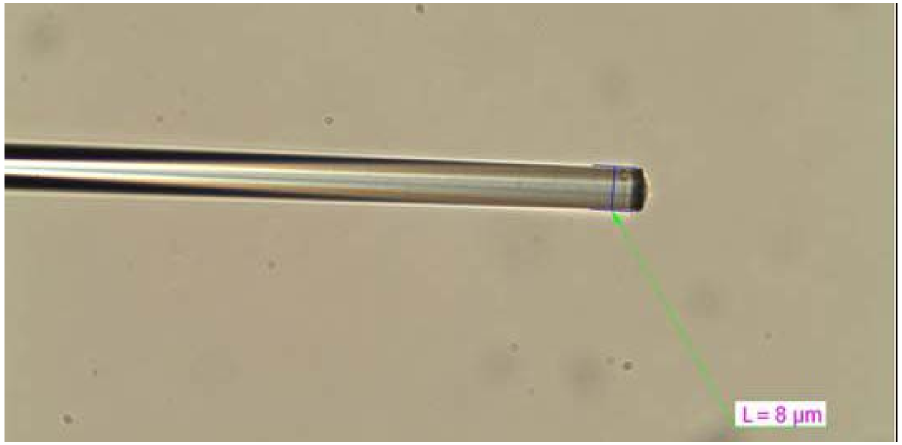
APPLICATION OF INPUT SOLUTIONS ON TIPS

Dip Coating

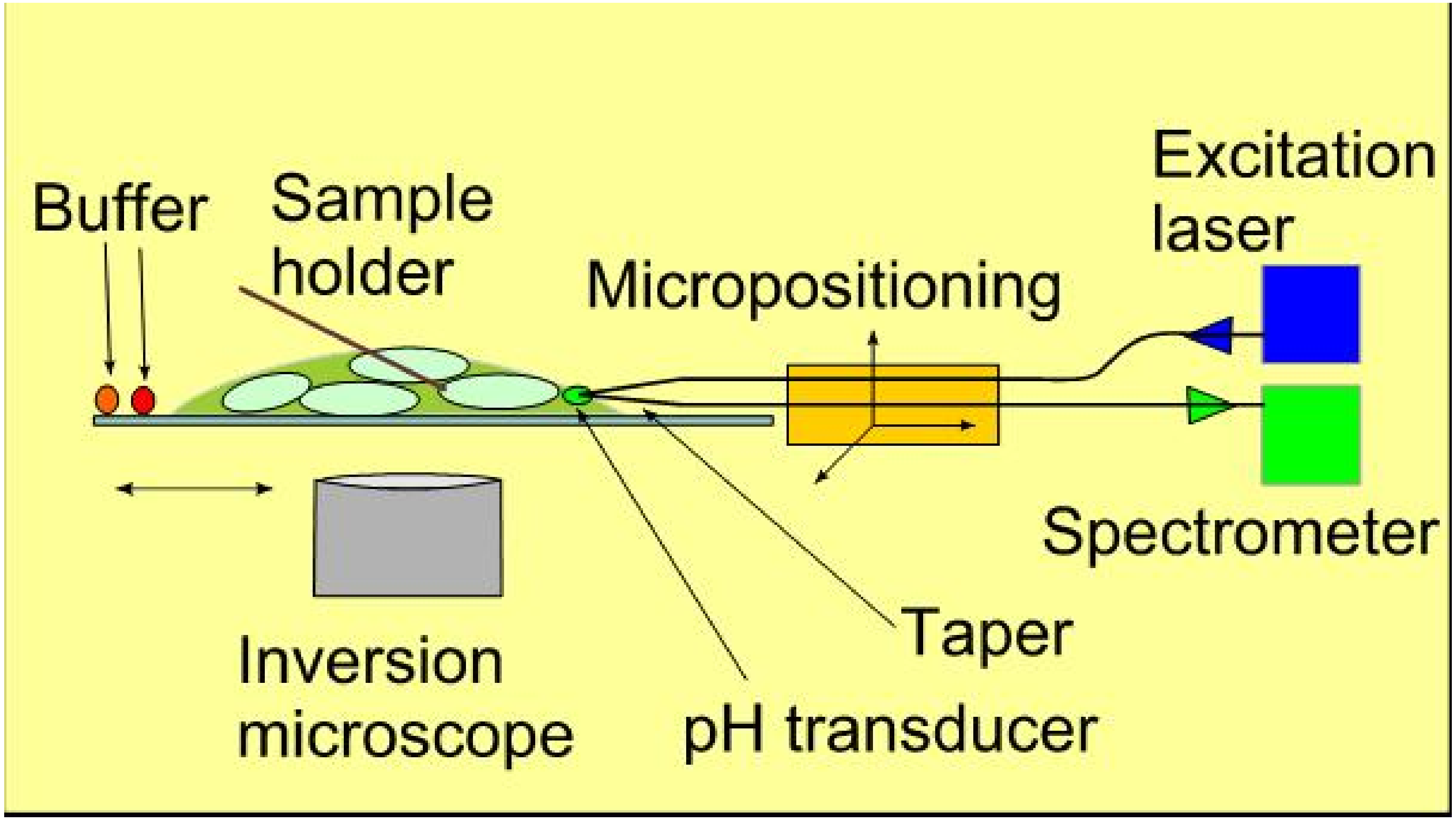


Capillary effects



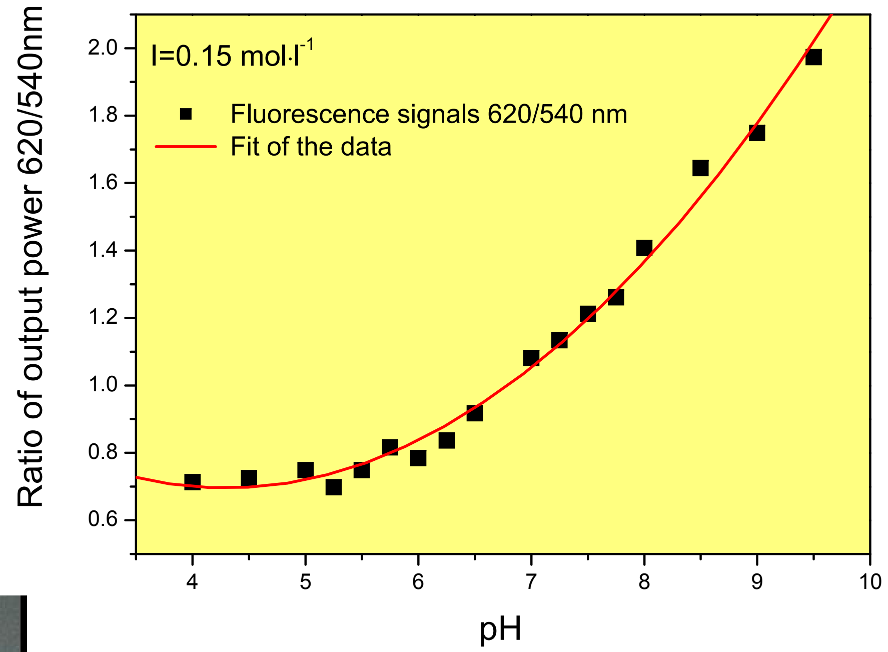


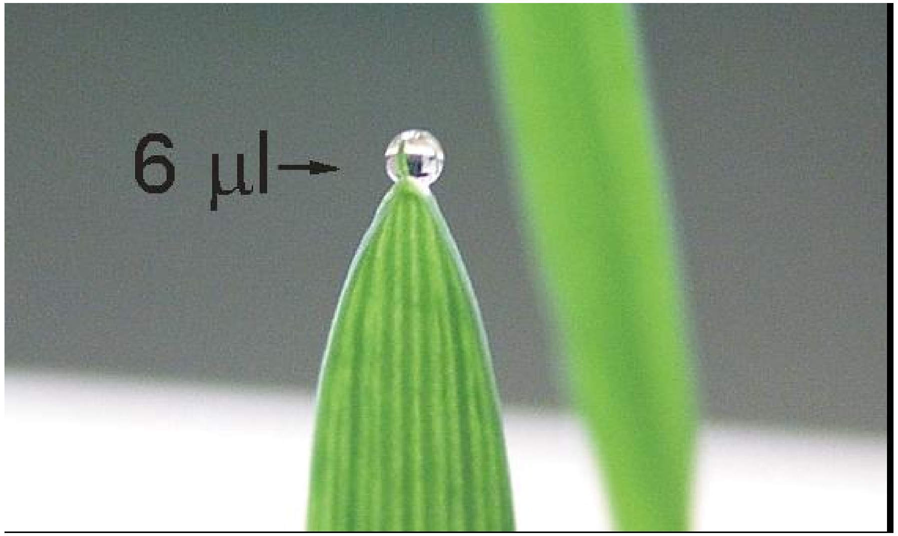
SCHEME OF SET-UP IPE



MEASUREMENTS

Change of fluorescence intensity of transducers due to pH changes





Drops of exudate from tobacco leaves

pH MEASUREMENTS RESULTS

	Exudate from leave tip	Exudate from a leave center	Exudate from leave basis
Average	5.0	5.6	5.5
St. deviation	0.3	0.3	0.1
Electrochem.	5.4	5.4	6.0

No pH gradient through leaves

I. Kašík et al., Anal. Bioanl. Chem. 398 (2010) 1883-1889

CONCLUSIONS

- **Several original approaches** for increasing the **detection sensitivity** of multimode PCS (PCG) or plastic fibers to chemical developed in IPE.
- Optical fiber sensors can be used for detection in small volumes (cells), in remote or hardly accessible places
- They can be used for distributed detection - useful for monitoring integrity of buildings, bridges, airplanes, preventing leakages of dangerous substances into environment