The University of Texas at Austin
Cockrell School of Engineering

SENSORHUT
Optical Chemical Sensing

# Self-Aligned Fiber-to-Waveguide Configuration for Enhanced Thermal Stability and Cost-Effective Production of Nanoporous Waveguides for Sensing

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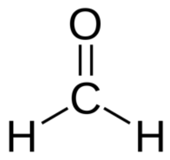
# Gas sensing

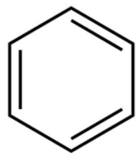
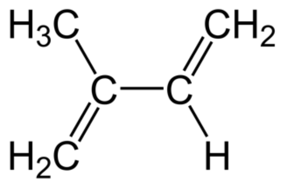
- Gases (CO, $NO_2$, $O_3$, $SO_3$, $H_2S$, NO)
- Particulate matter ($PM_{10}$, $PM_{2.5}$)

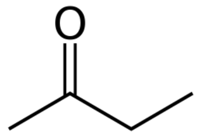
Legal concentrations are well defined
Sensors exist

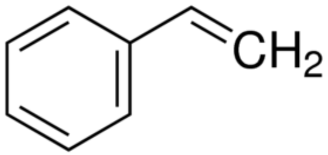
- Volatile organic compounds (VOCs)

Toxicity is still unknown for most VOCs
+
No affordable and accurate VOC sensors



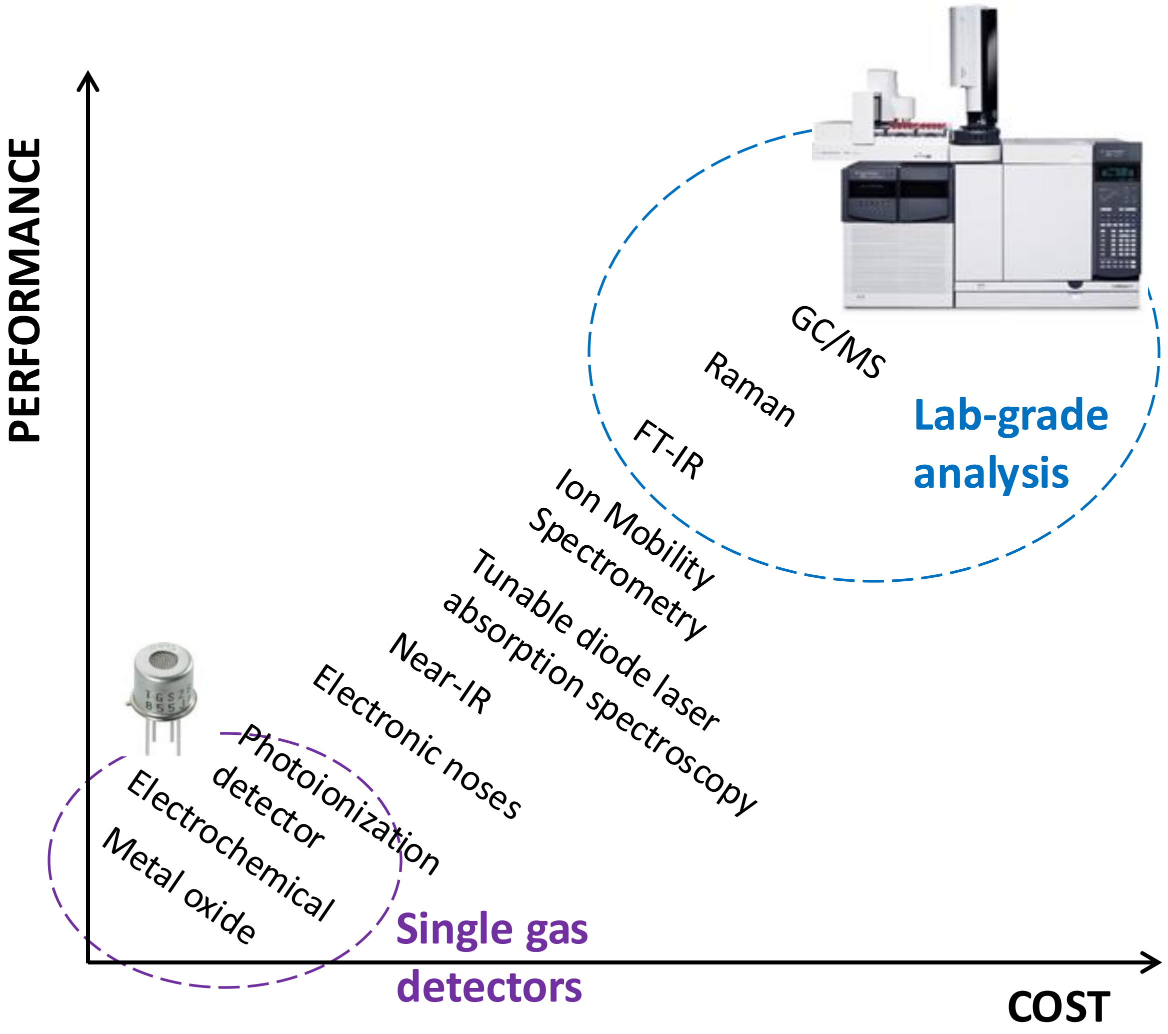





## Applications:

- Health and safety
- Air quality
- Chemical process
- Homeland security
- Medical diagnostics

# Volatile organic compounds (VOCs) sensing



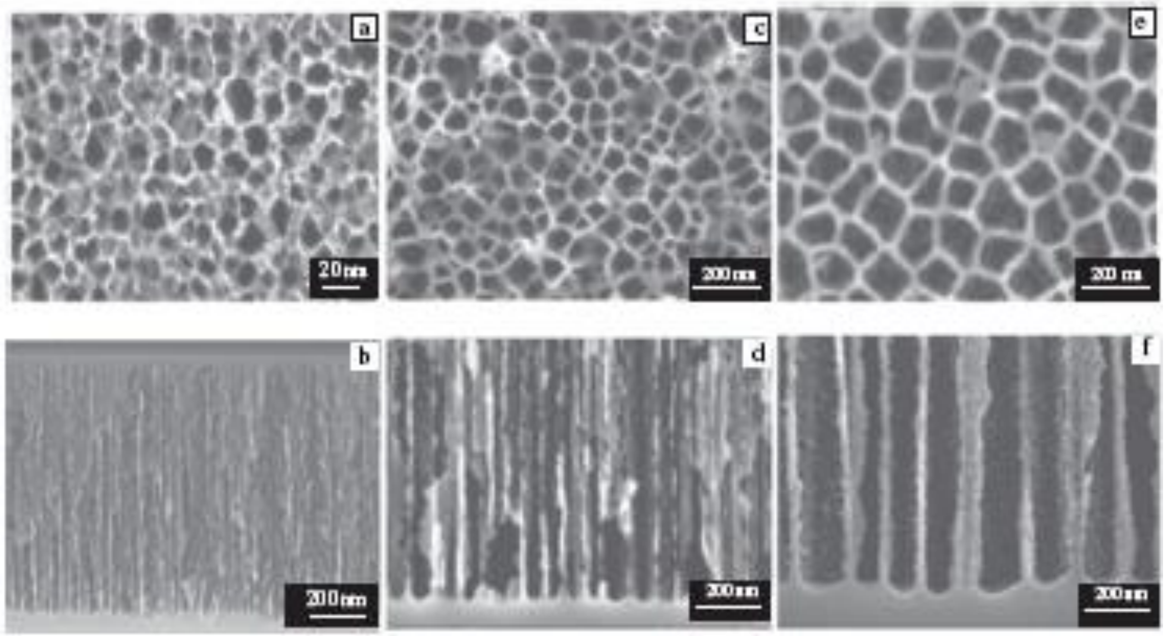

**Need for small low-cost VOCs sensors, able to distinguish between chemically similar compounds**

# Nanoporous materials

Large surface area → high sensitivity, miniaturisation

<u>Nano-porous silicon</u>

- Simplicity and repeatability of fabrication
- High surface area 200 - 1000 $m^2/cm^3$
- Pore sizes 30Å to 1µm and porosities of 10-90%
- Compatibility with Si-based microelectronics

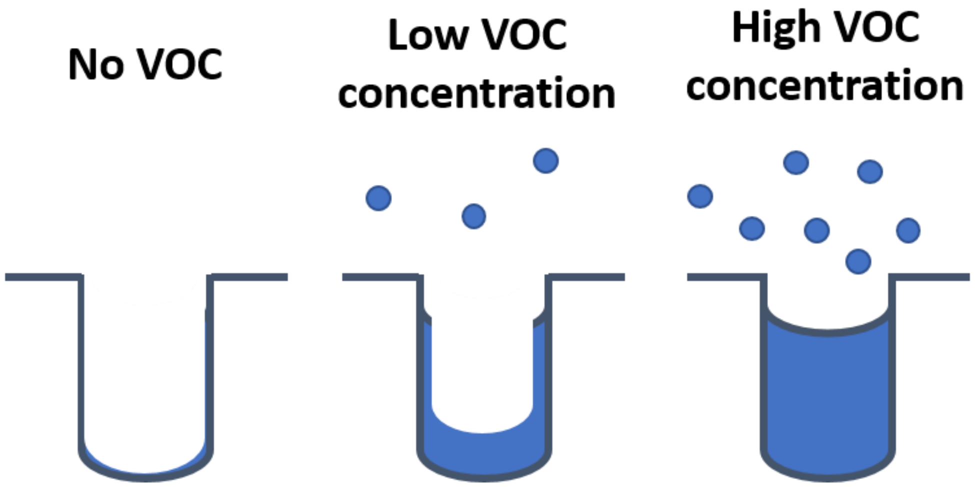



Adapted from: H. Ouyang *et al.*, Frontiers in Surface Nanophotonics, 2007

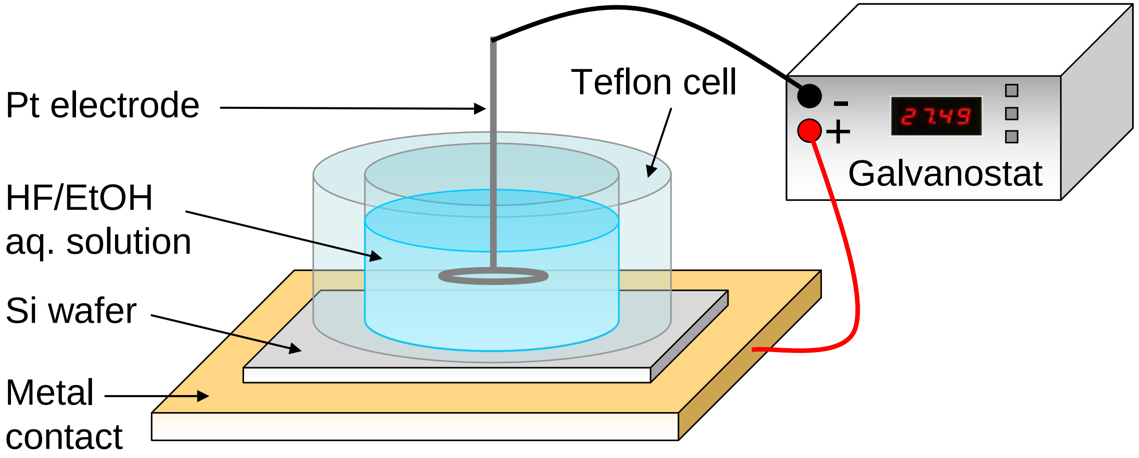
# Nanoporous materials

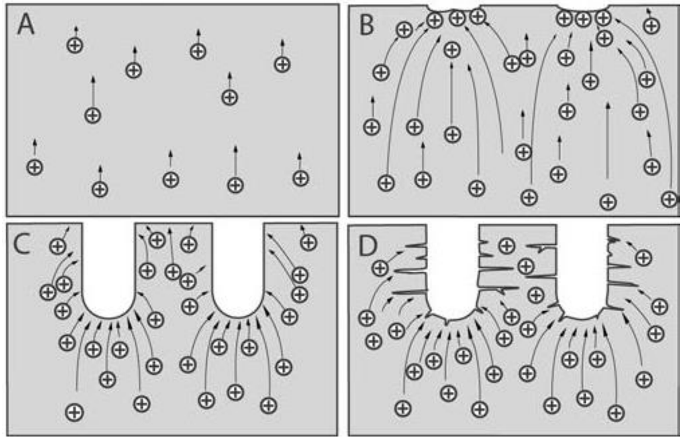
Large surface area → high sensitivity, miniaturisation

- **Capillary condensation**: condensation of vapors inside nano-pores well above their condensation temperature.
- Fully reversible.



# Nanoporous silicon fabrication



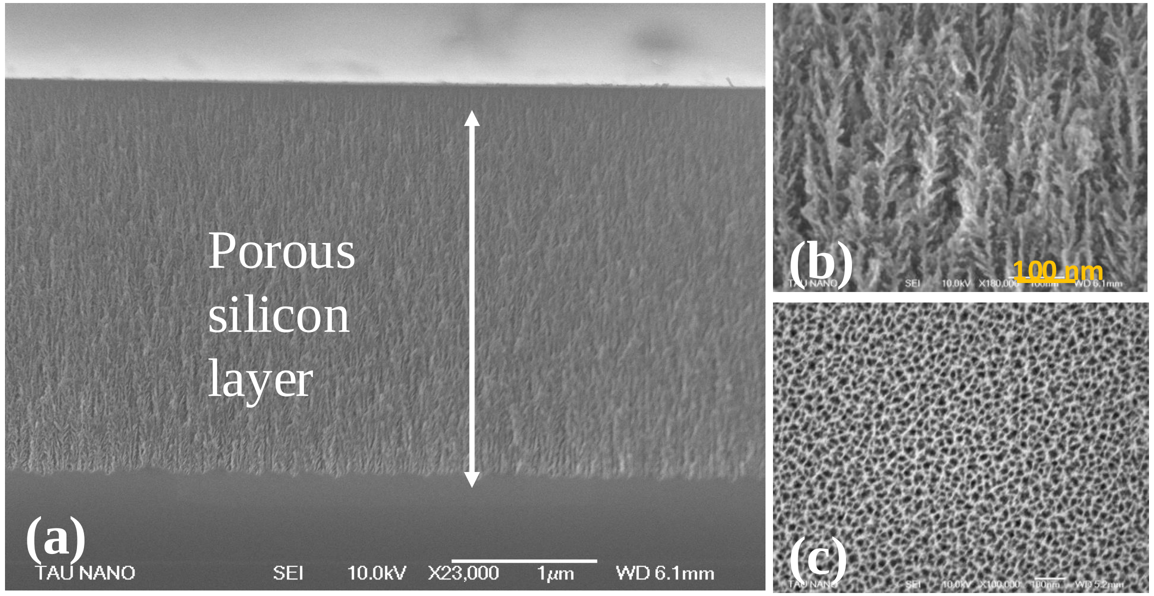

**Parameters:**

- Etch rate
- Porosity
- Pore diameter

Controlled by…

**Etching conditions:**

- Wafer type
- Temperature
- Resistivity
- HF concentration
- Current density
- Crystallographic orientation

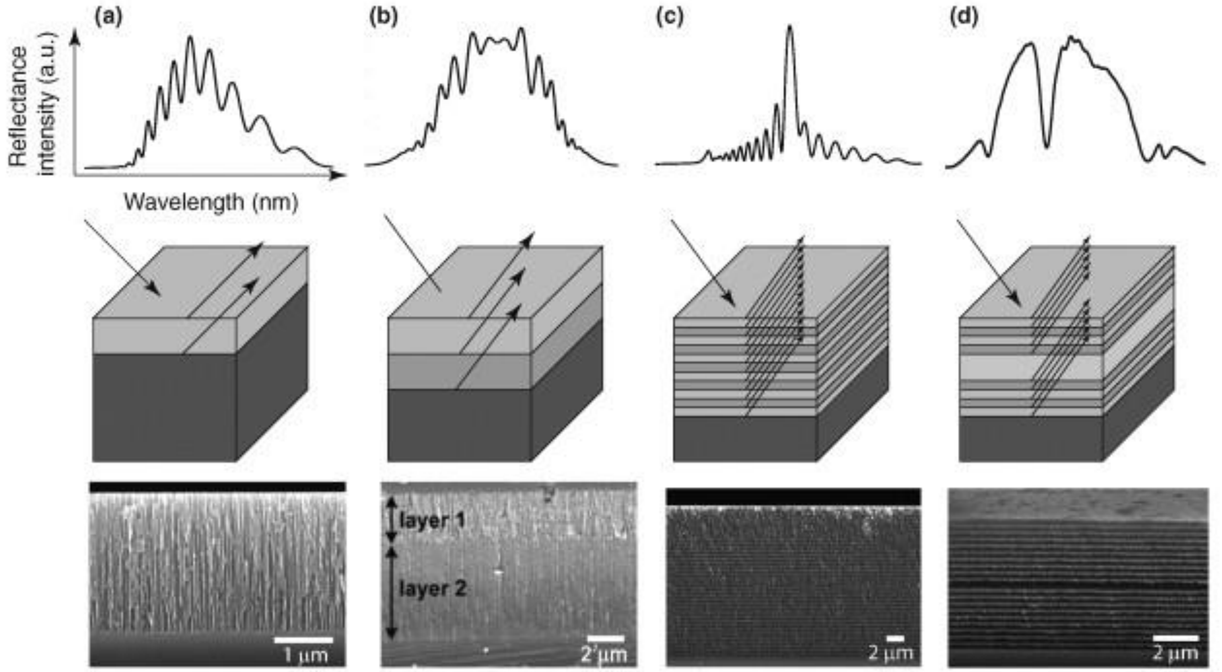


# Optical properties

- Current density ∝ Porosity ∝ Refractive index
- Brueggemann Effective Medium Approximation

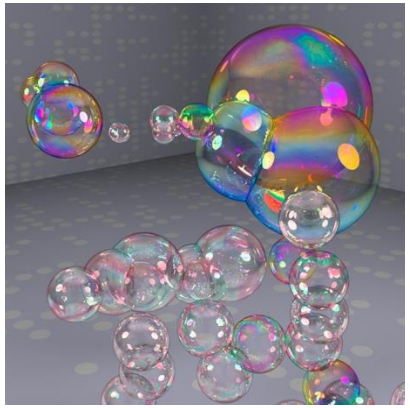
  Pores diameter << wavelength of light

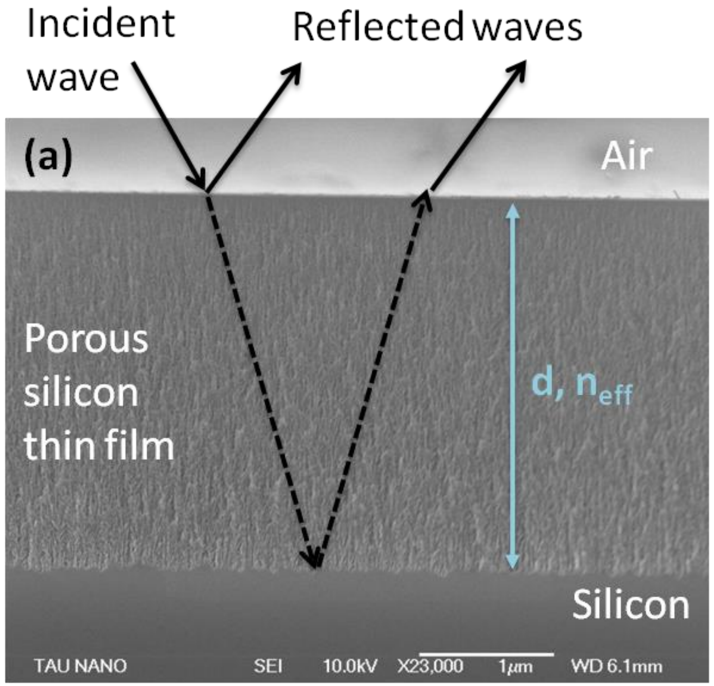
- Filters / reflectors




A.Jane et al. Trends in Biotechnology 27(4) 230 - 239, 2009

# Thin-film interference

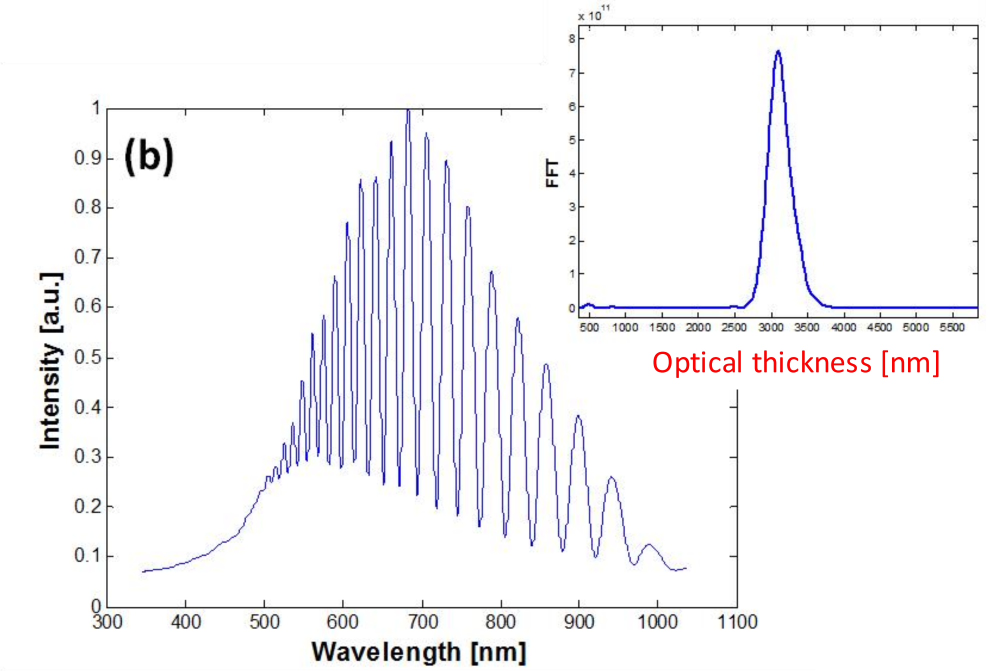





$$m \cdot \lambda_m = 2 \cdot n_{eff} \cdot d \cdot \cos\theta$$

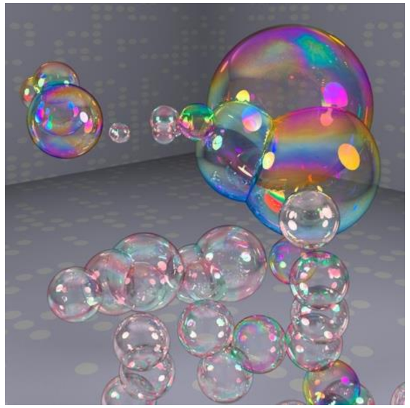
Optical thickness

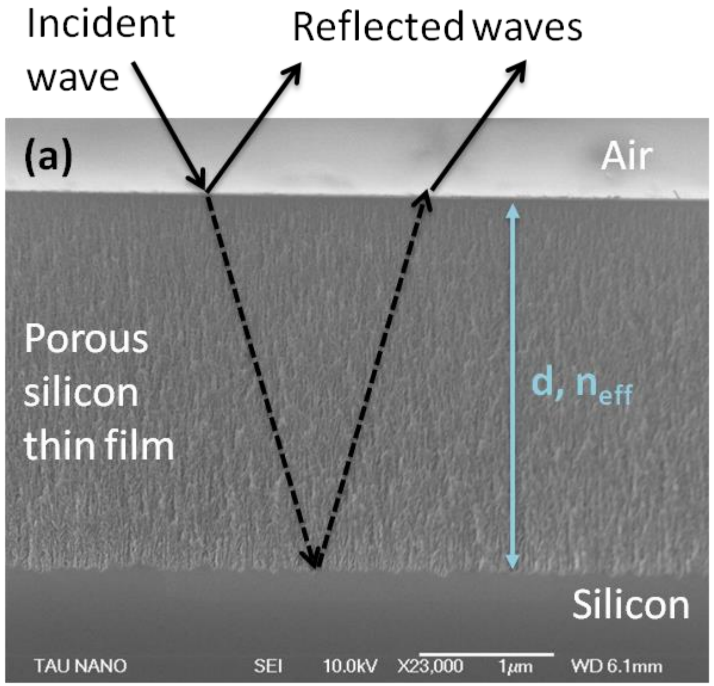
Fourier-transform

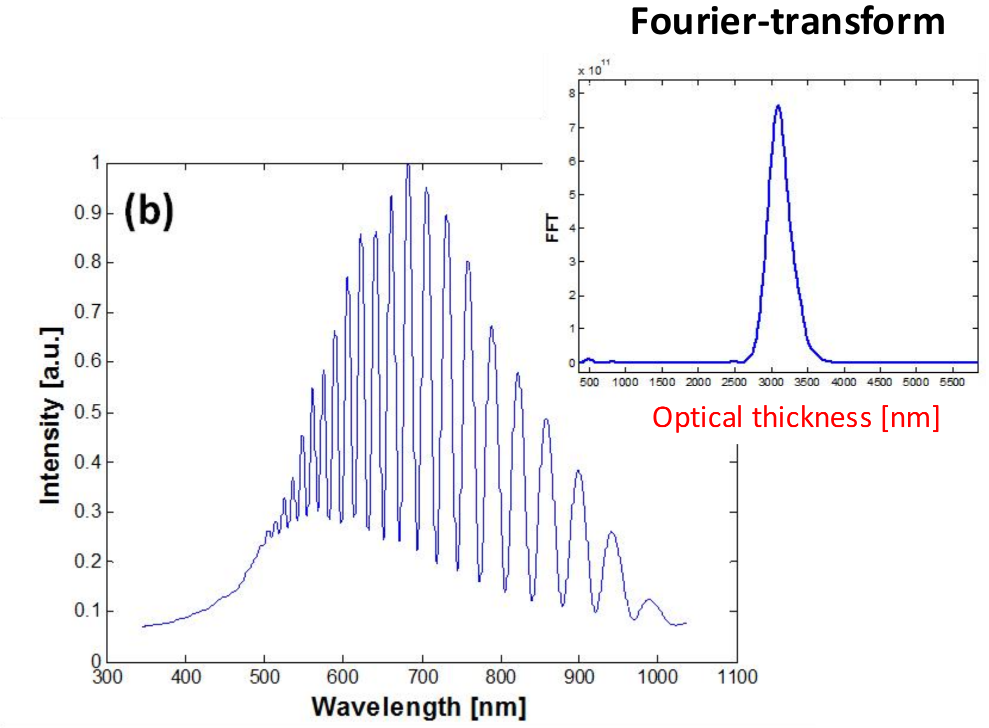



**Reflectance spectrum** – constructive and destructive interference maxima and minima

# Thin-film interference






$$m \cdot \lambda_m = 2 \cdot n_{eff} \cdot d \cdot \cos\theta$$

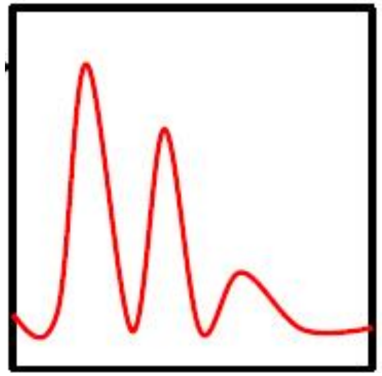
Optical thickness

$$\underbrace{(1-p)\frac{n_{Si}-n_{eff}}{n_{Si}+2n_{eff}}}_{Silicon} + \underbrace{(p-V)\frac{n_{air}-n_{eff}}{n_{air}+2n_{eff}}}_{Air} + \underbrace{V\frac{n_{vap}-n_{eff}}{n_{vap}+2n_{eff}}}_{Vapor} = 0$$

# Sensing with a porous silicon layer

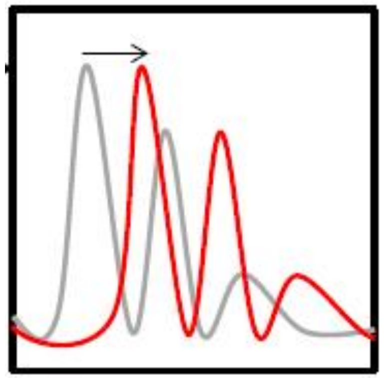
<u>Before gas exposure</u>

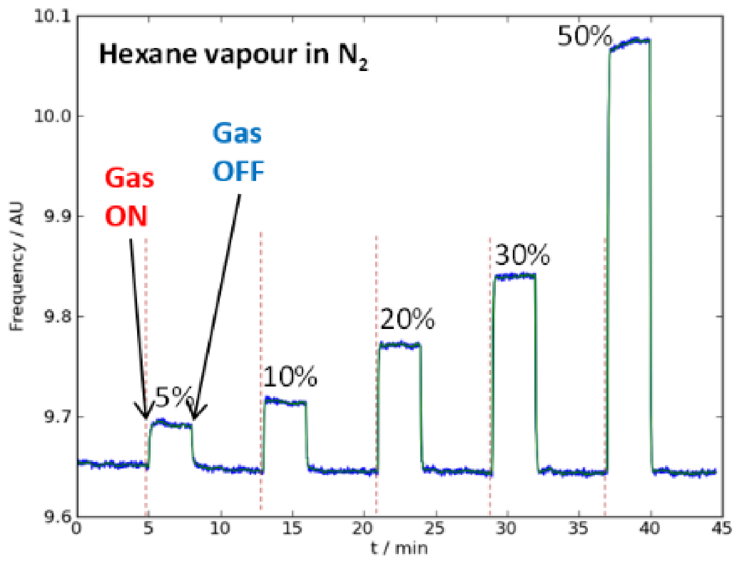
- empty pores

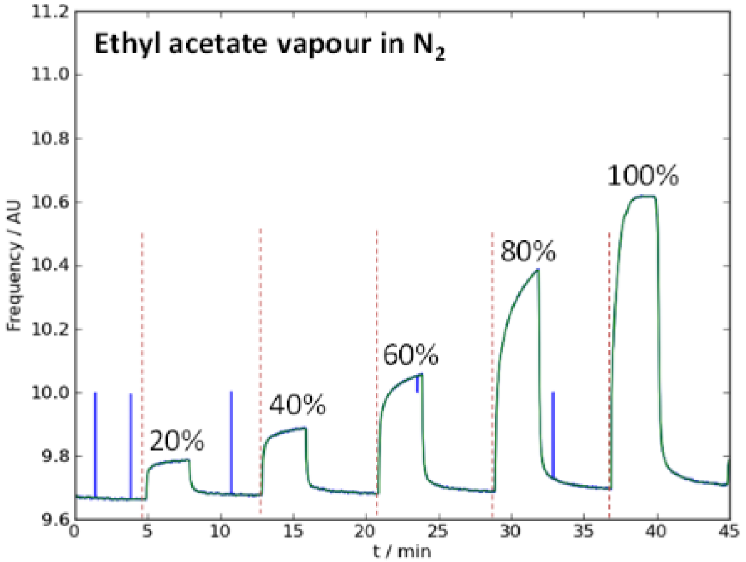



<u>Upon gas exposure</u>

- increase in the refractive index



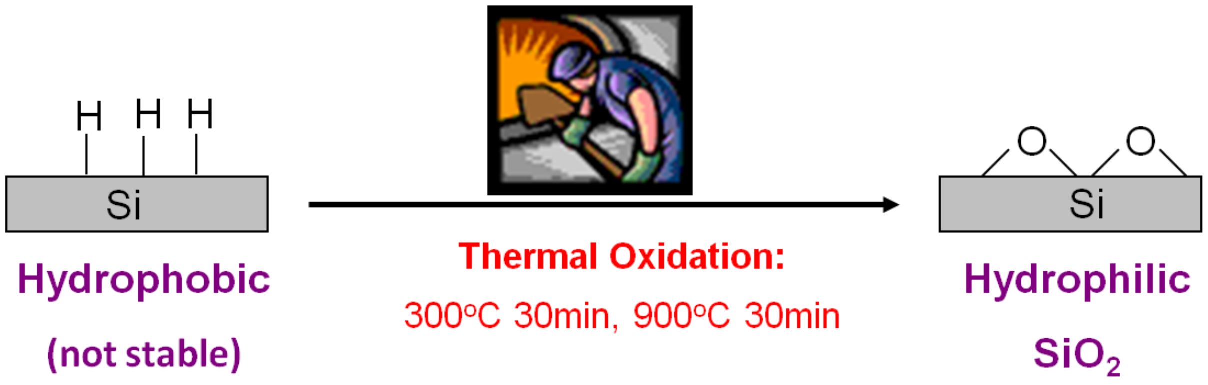




- Real-time monitoring
- Fast response
- Fully reversible
- Stable baseline

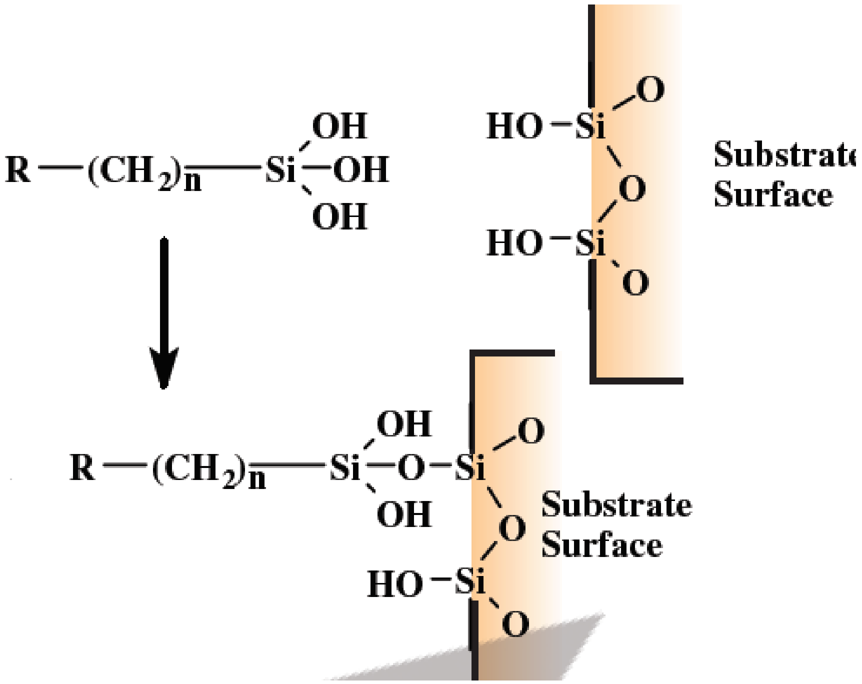
**Selectivity!**

# Chemical surface functionalisation

- Oxidation
  - Thermal, ozone, chemical

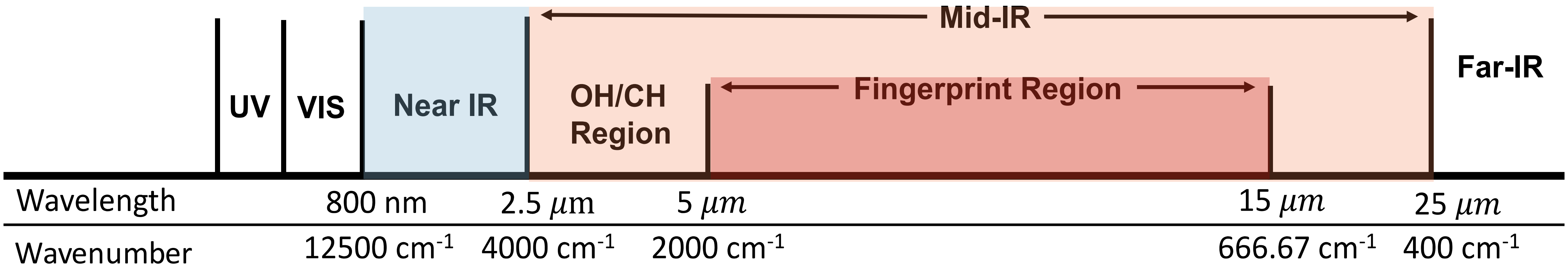



- Silane chemistry
  - Known chemistry of attaching various functional groups to Si surface, including biological groups




Good for bio-sensing with specific bio-receptors, but only partial selectivity for VOCs!

# Spectral infrared (IR) regions




**Near-infrared (NIR)**

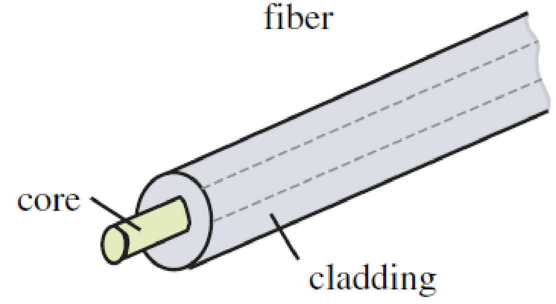
- Broad and weak absorption peaks
- Low sensitivity and selectivity
- Low-cost and compact components

**Mid-infrared (MIR)**

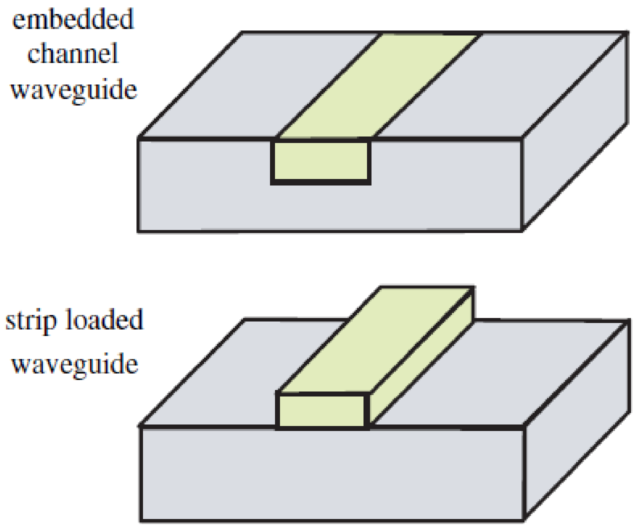
- Strong and sharp absorption peaks
- High sensitivity and selectivity
- Expensive and bulky components

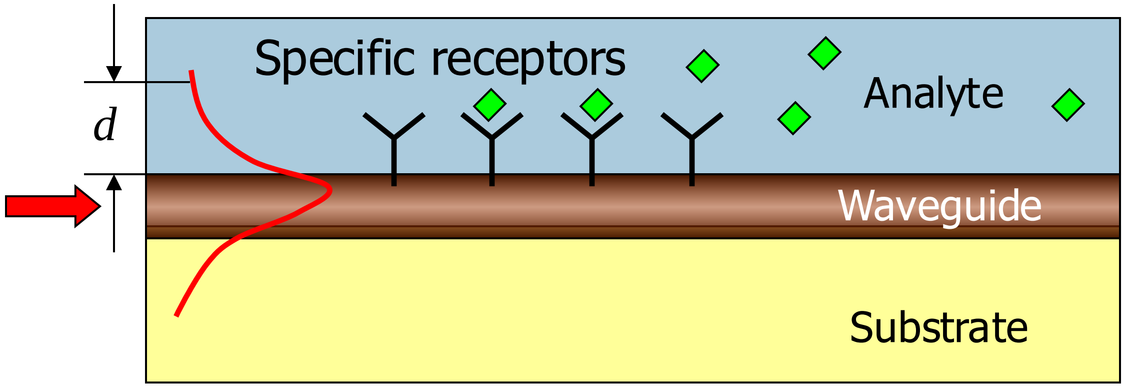
**Mid-IR offers good specificity of detection, however lacks sensitivity in the low-cost NIR region.**

# Evanescent field fiber/waveguide sensors




$n_{core} > n_{cladding}$



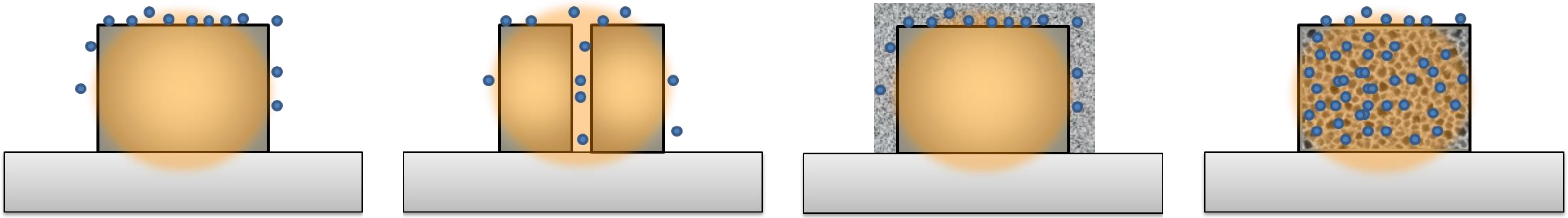




*d* - Evanescent field penetration depth

- Widely used for liquid-phase sensing
- Not sensitive in gas-phase

  gas is a lower-density matrix, resulting in a small number of molecules at the fibre/waveguide surface at any given time

# Waveguide gas sensors




**Evanescent-field sensing**
Short light-gas interaction
*Low sensitivity*

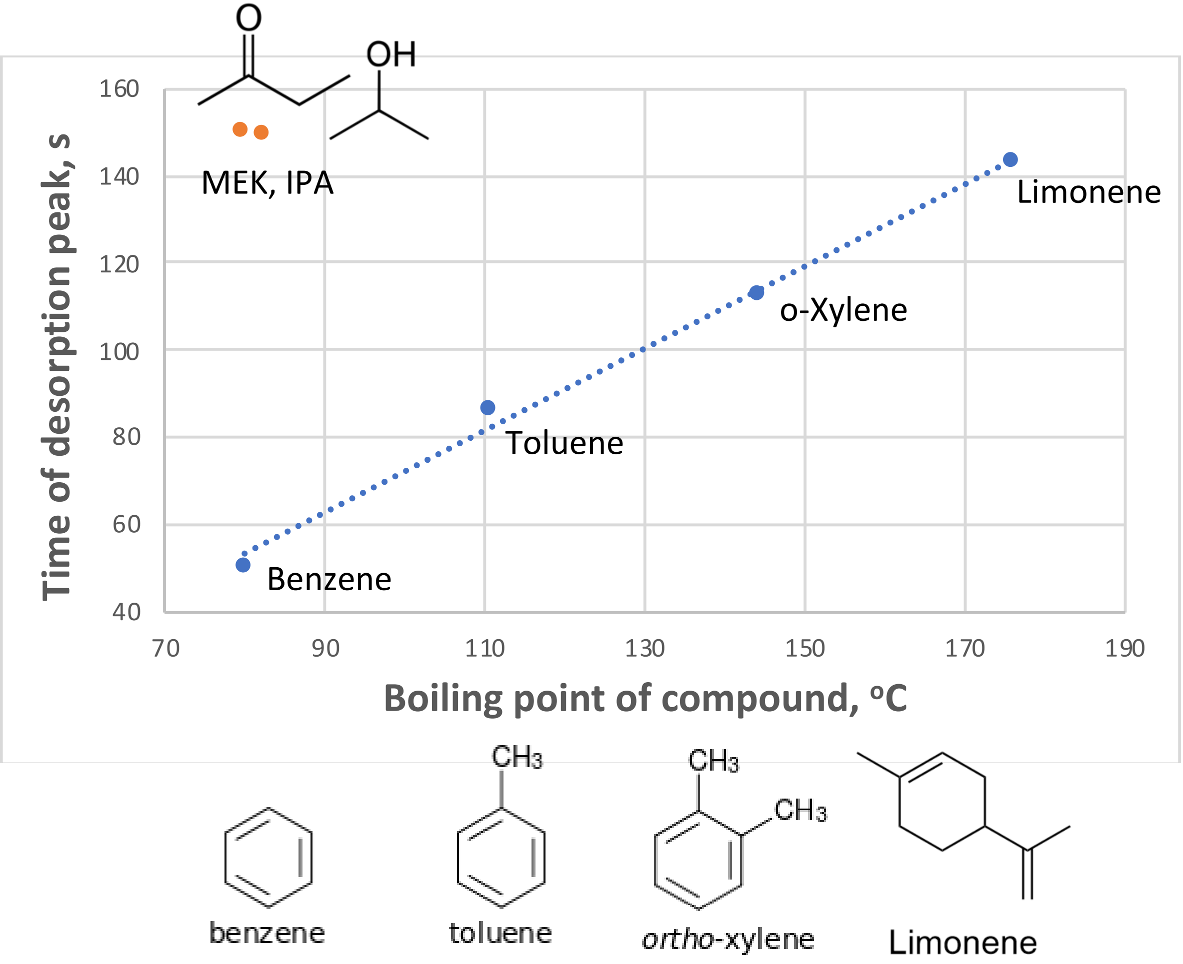
**Slot-waveguide sensing**
Nano-sized slot along the waveguide length
*Medium sensitivity*

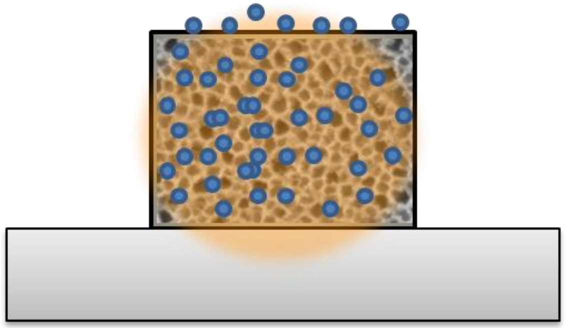
**Nanoporous-cladding waveguide sensing**
Upper cladding facilitates gas sorption
*Medium sensitivity*

**Nanoporous-core waveguide sensing**
Waveguide core facilitates gas sorption
*High sensitivity*

# Waveguide gas sensors

## Temperature of the porous material governs adsorption-desorption



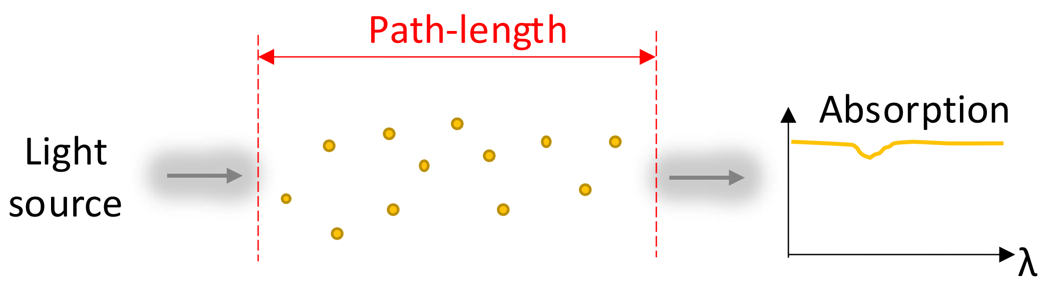



**Nanoporous-core waveguide sensing**

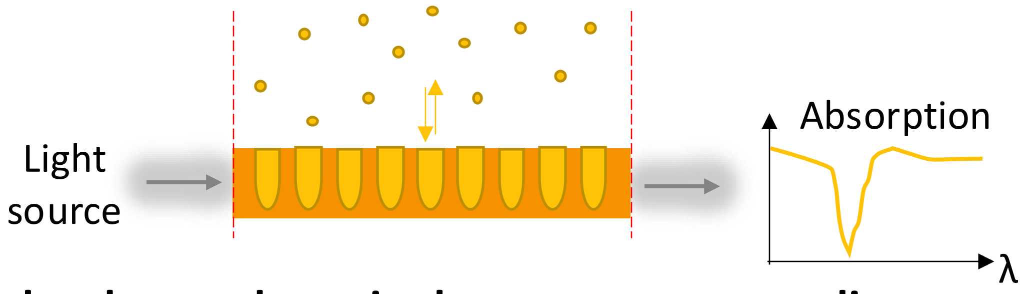
Waveguide core facilitates gas sorption

*High sensitivity*

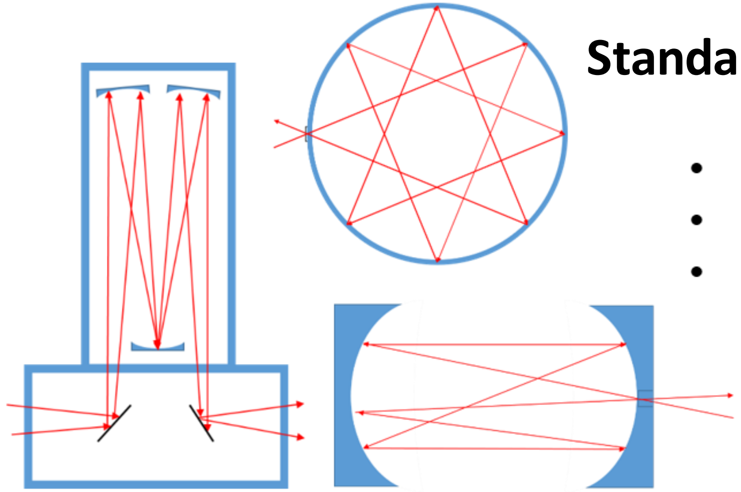
# IR spectroscopy: standard free-space vs porous waveguide




**Molecules dispersed in gas phase**
Small absorption peak




**Molecules condense in the nano-porous medium**
High 'effective' interaction length
Strong absorption peak

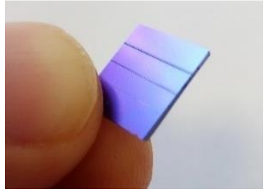


## Standard trace-gas cells

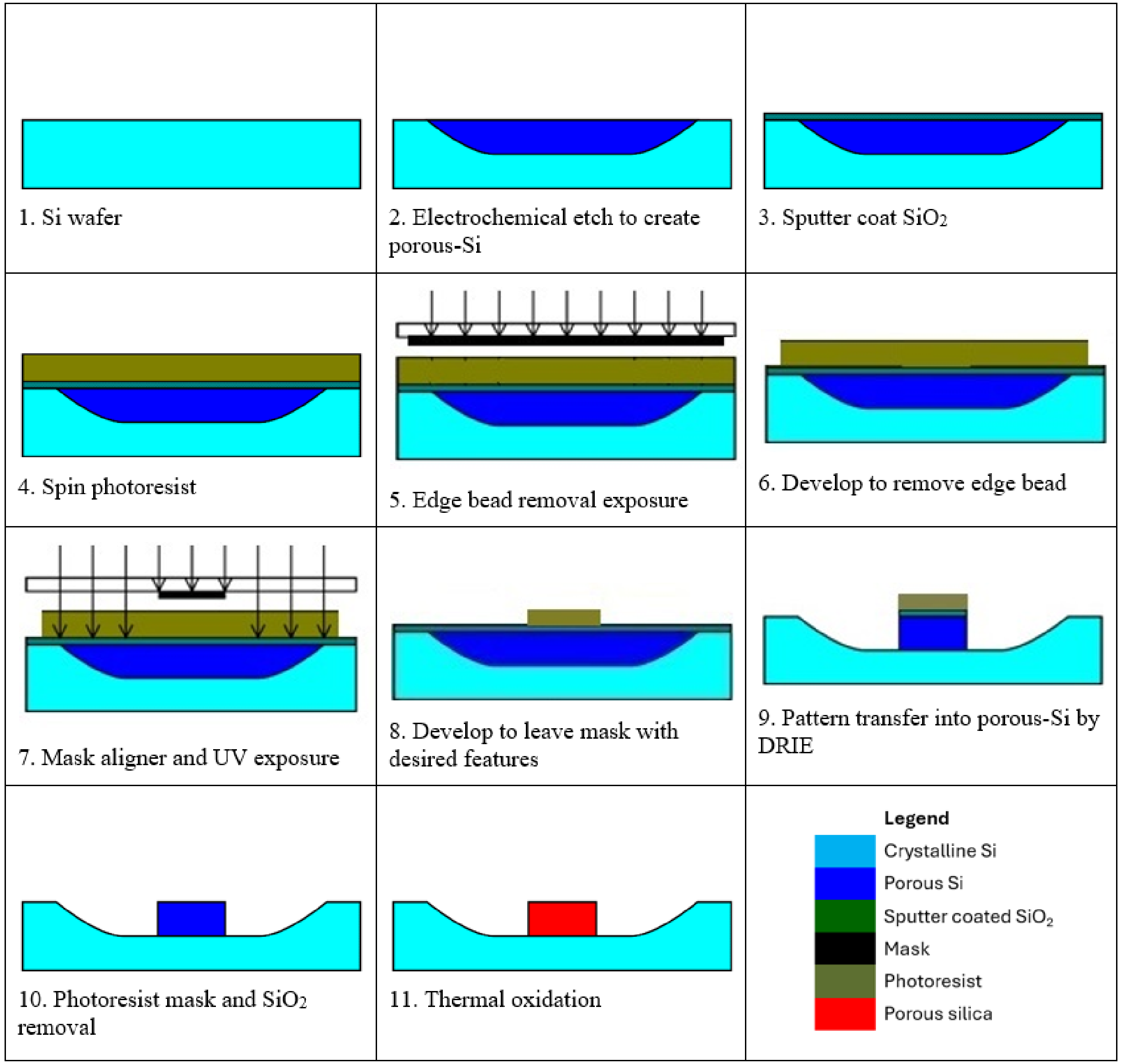
- Optical alignment
- Bulky
- Expensive

## Optical nanoporous waveguide

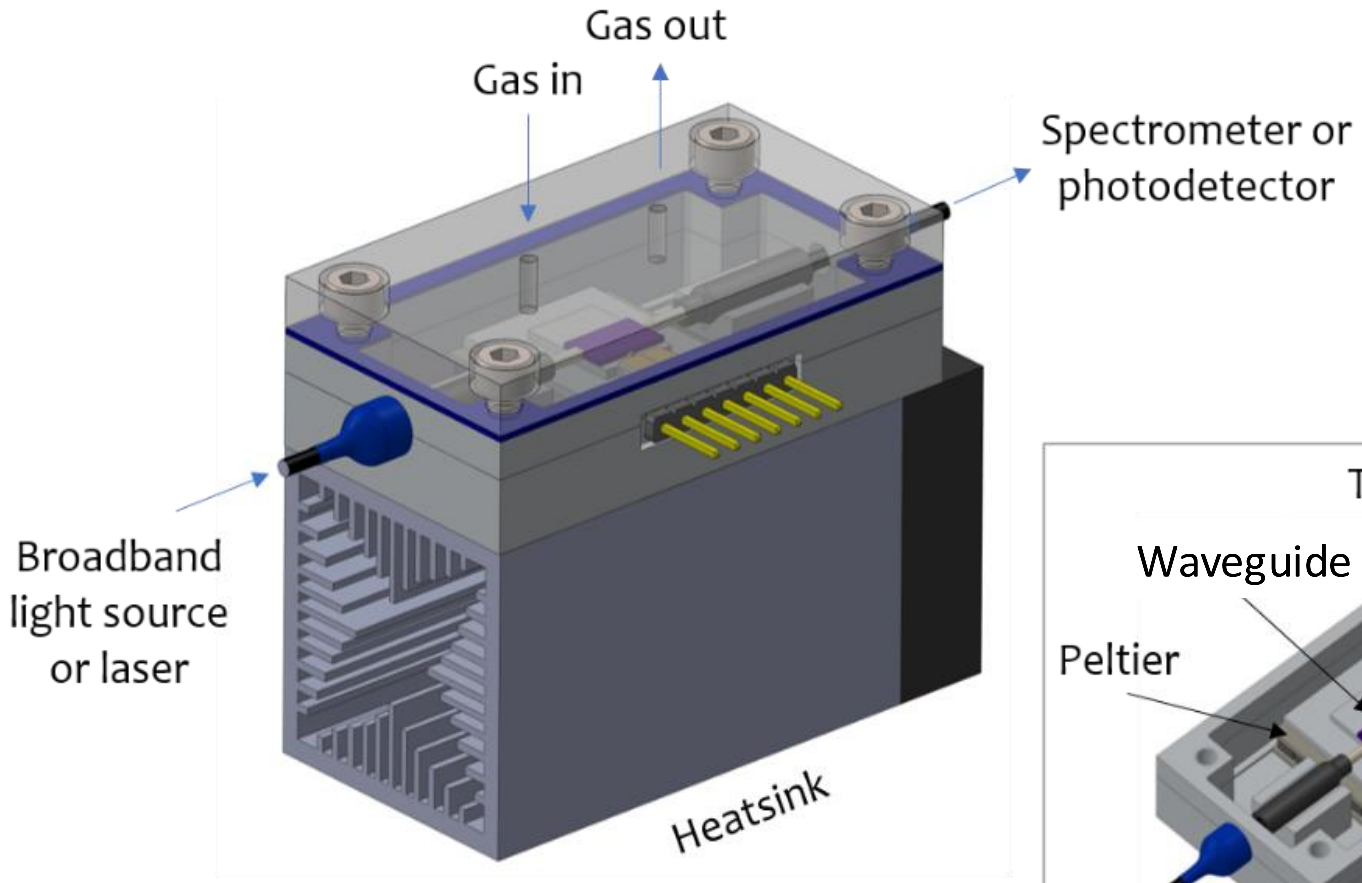
- Robust
- Small
- Cheap

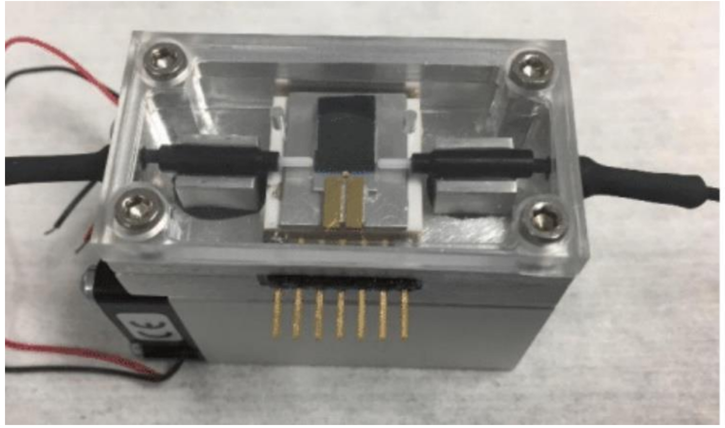


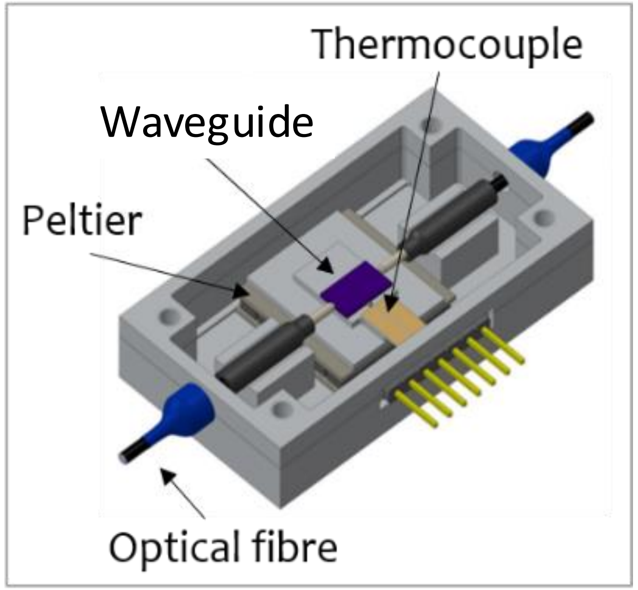
# Waveguide fabrication



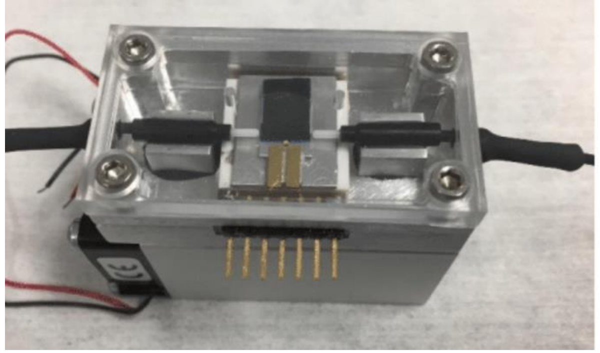
# Waveguide sensor assembly



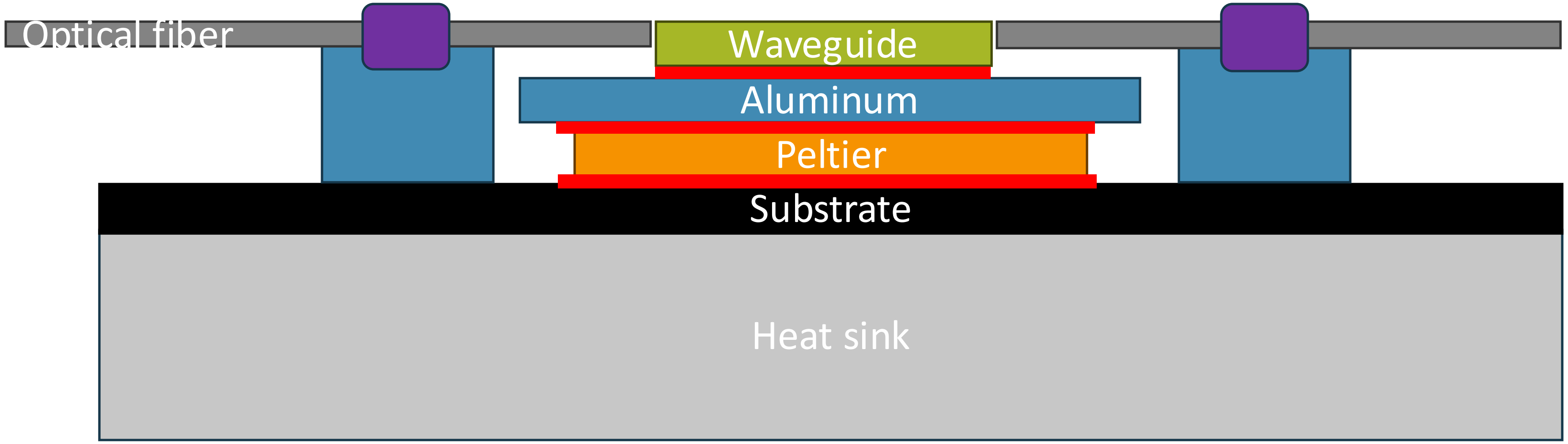





# Waveguide sensor assembly



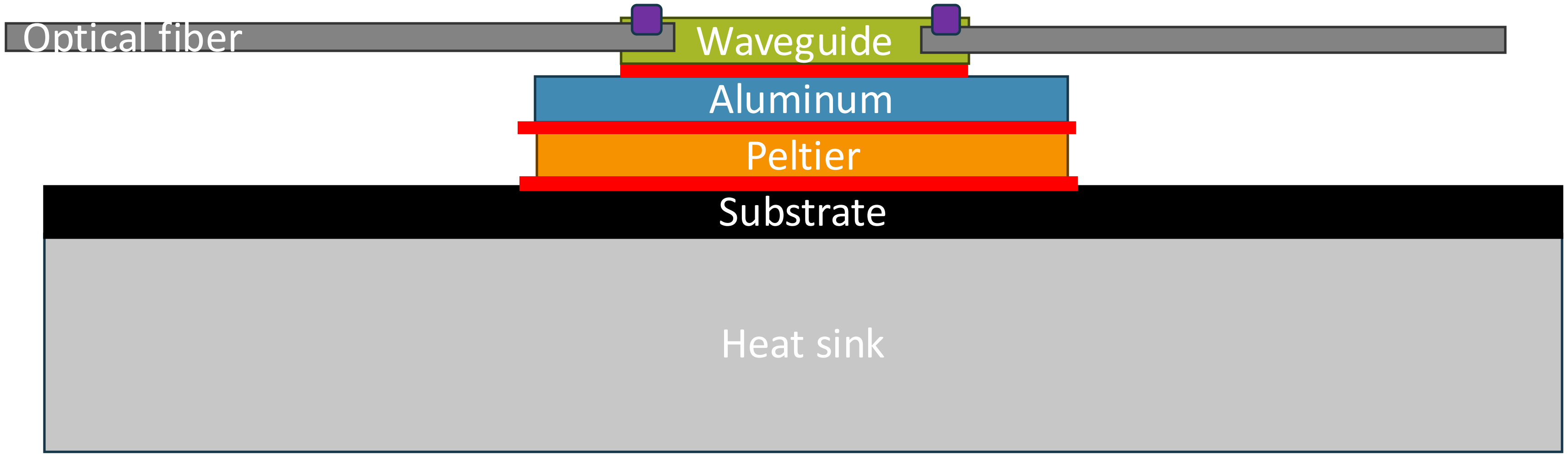
Active optical alignment
Followed by an adhesive to secure in place




**Failure after only 2-3 thermal cycles!**

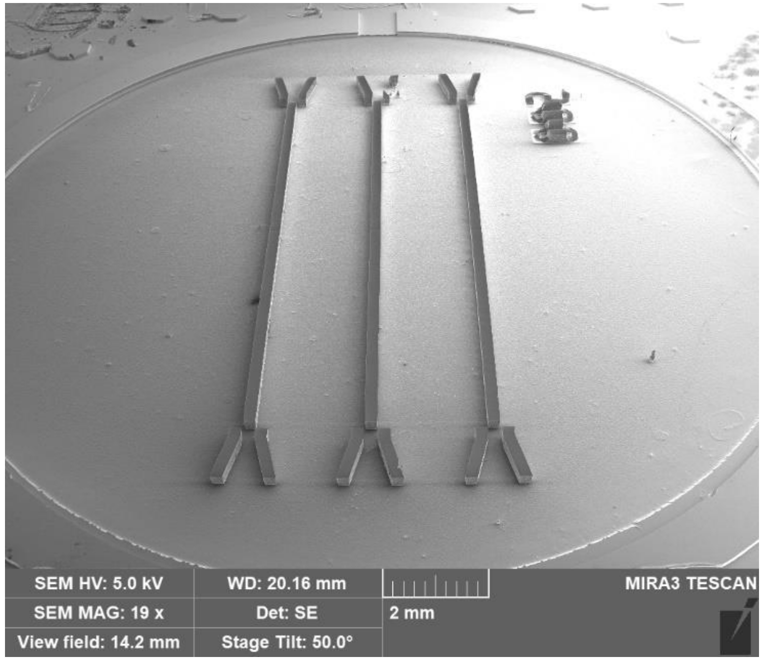
# Waveguide sensor assembly with on-chip alignment features

- No optical alignment, fibers ‘snap’ into position
- Waveguide and fibers on the same substrate, experience same thermal cycling

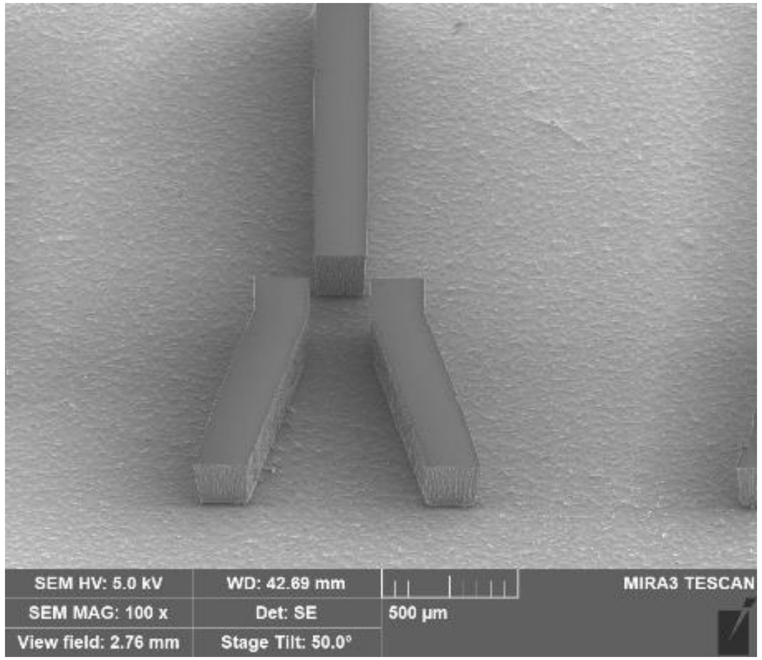



Patents: US10732093B2, GB2525854B, EP3137880B1

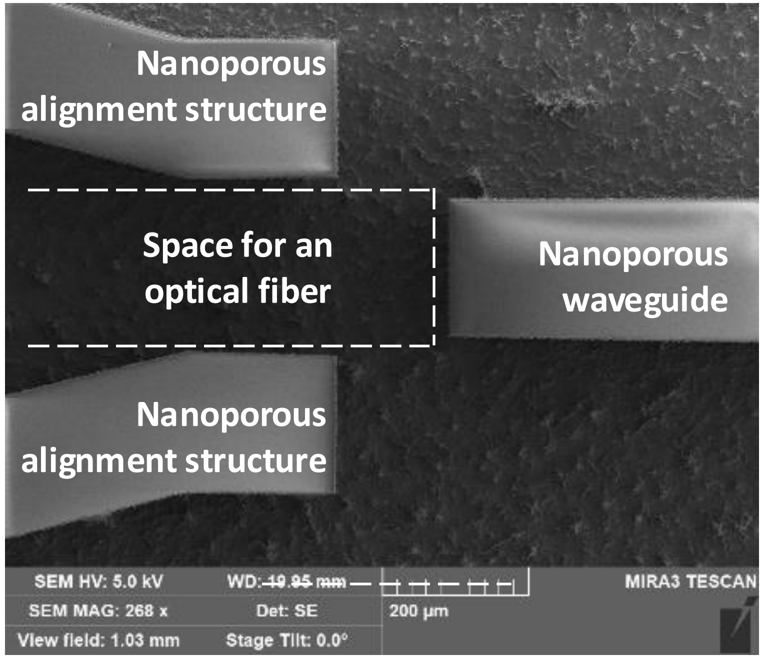
# Waveguide sensor assembly with on-chip alignment features




(a) Three waveguides on one chip

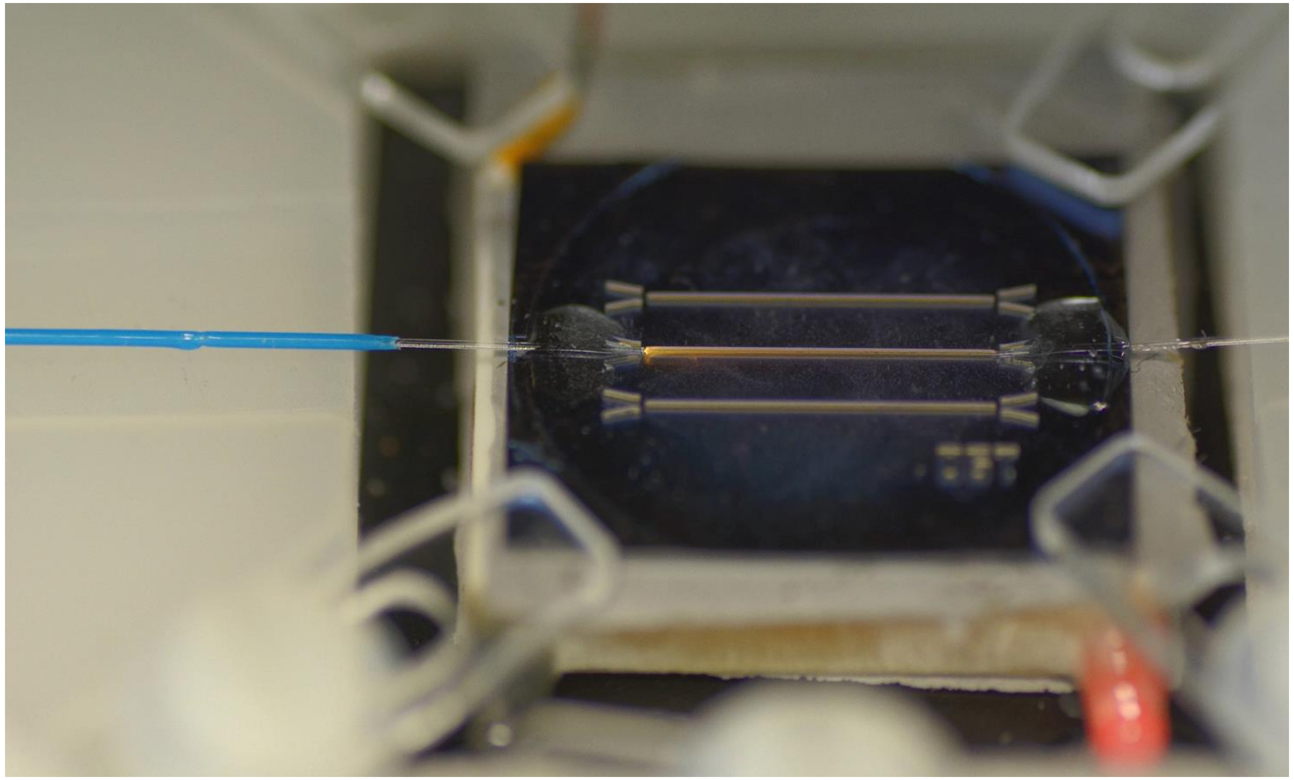



(b) Alignment features, side view

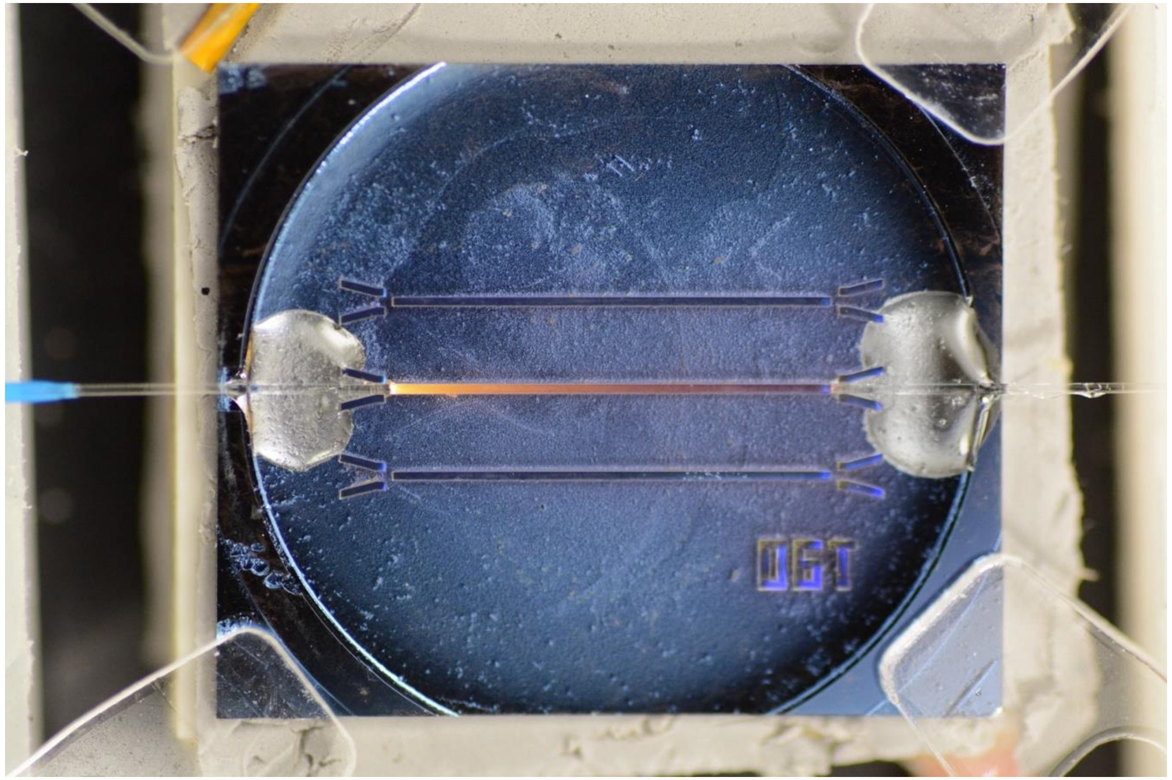



(c) Alignment features, top view

# Waveguide sensor assembly with on-chip alignment features

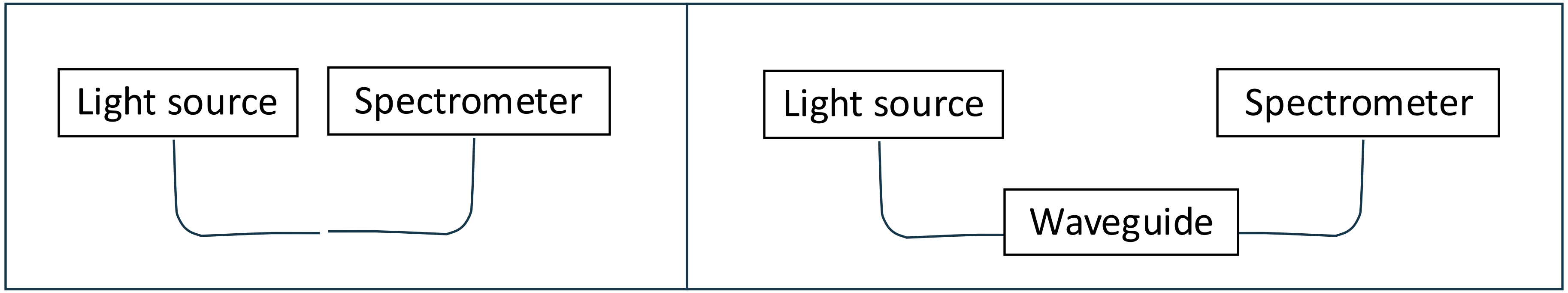


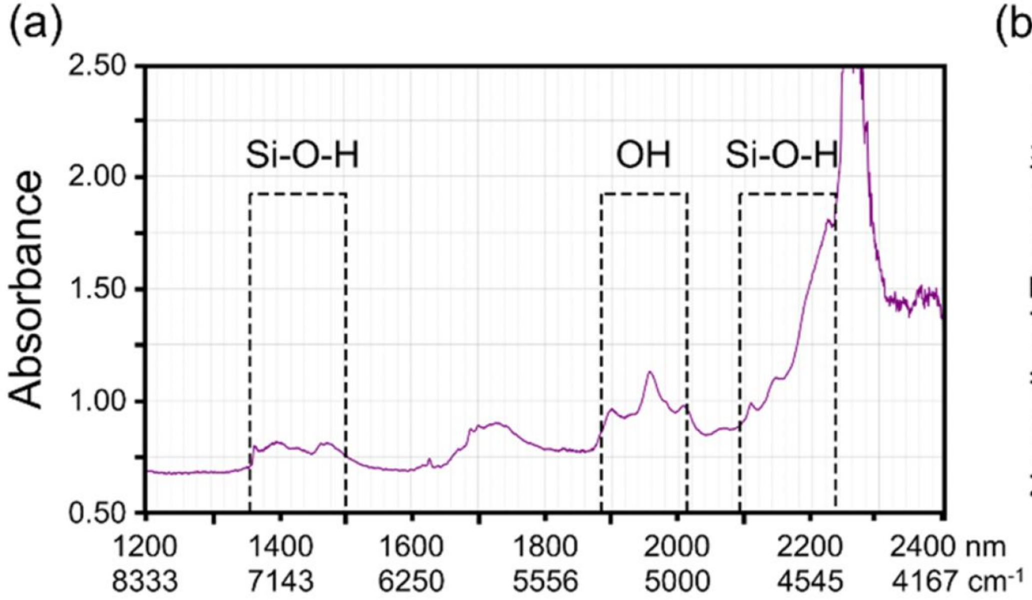
(a) Alignment features, side view



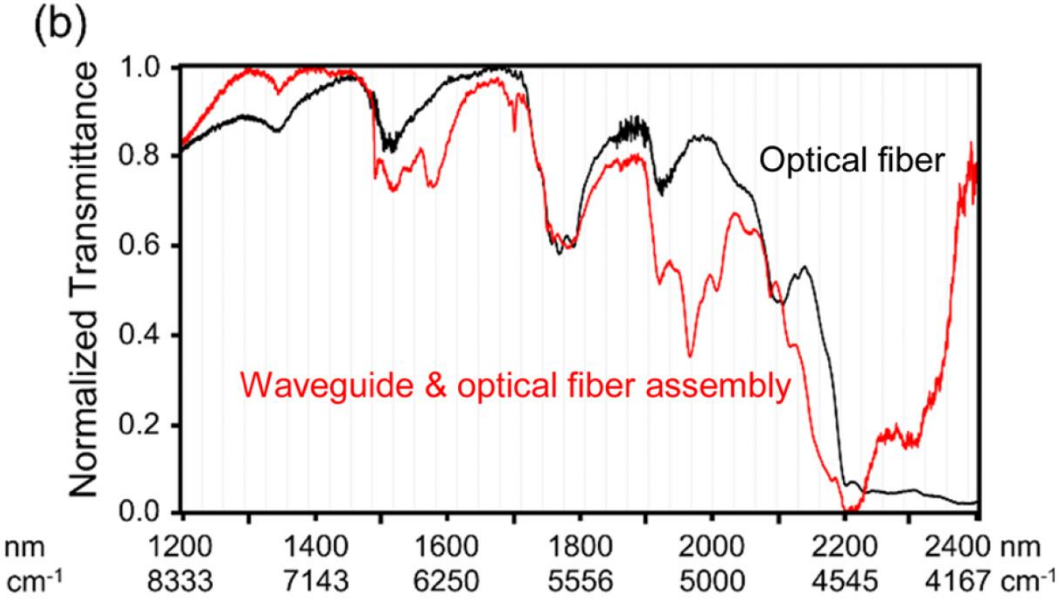
(b) Alignment features, top view

# Optical transmission and properties



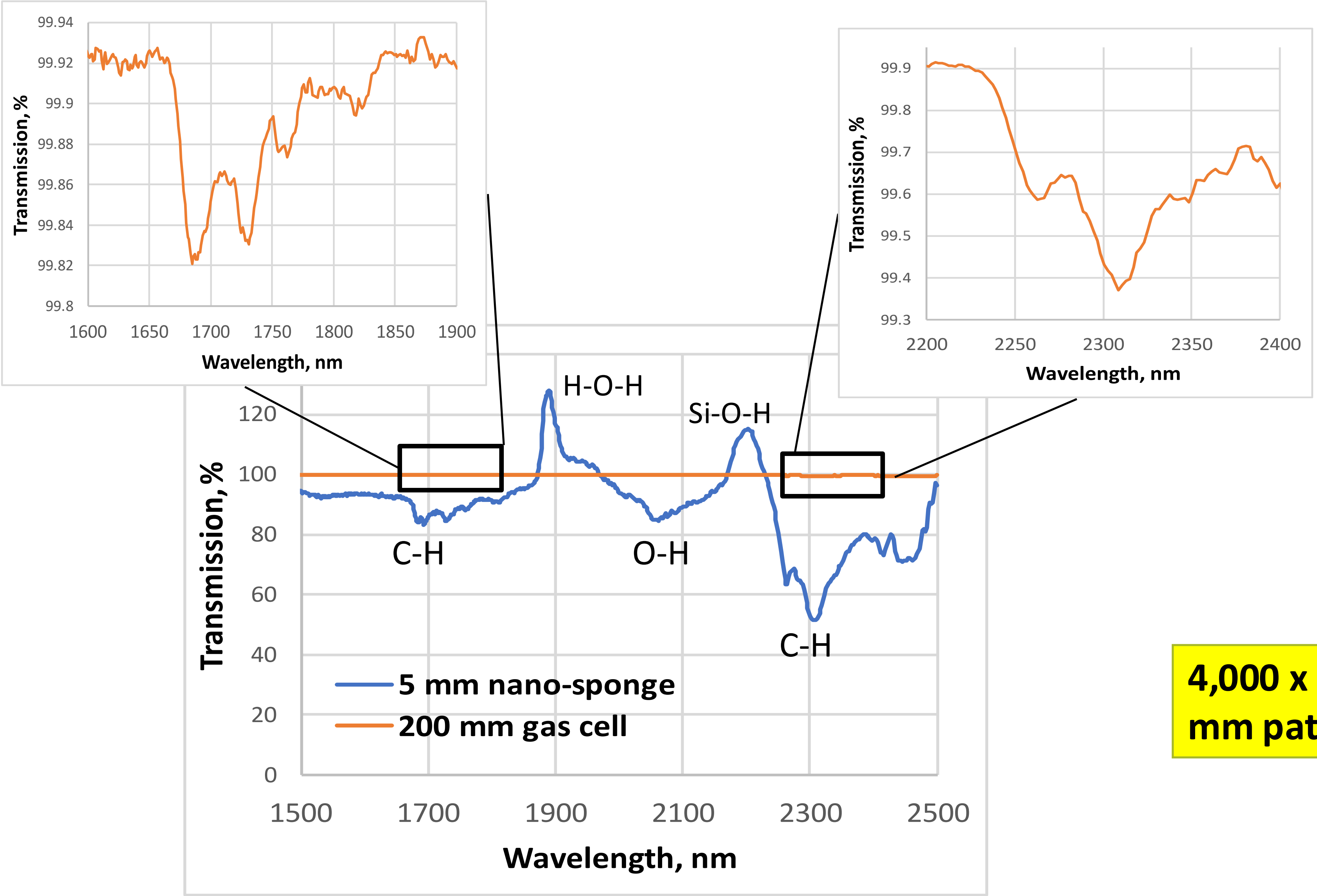

(a) Waveguide absorbance spectrum



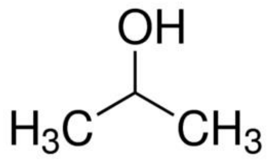

(b) Transmitted spectra though optical fibers with and without the waveguide

N. Barua et al., Spectral comparison of nanoporous silica-adsorbed organic molecules with gaseous and liquid states using a new waveguide technology, Vibrational Spectroscopy 125, 103496, 2023

# IR spectroscopy: standard vs nanoporous waveguide



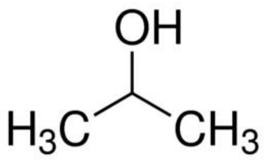

Isopropyl alcohol,
900 ppm



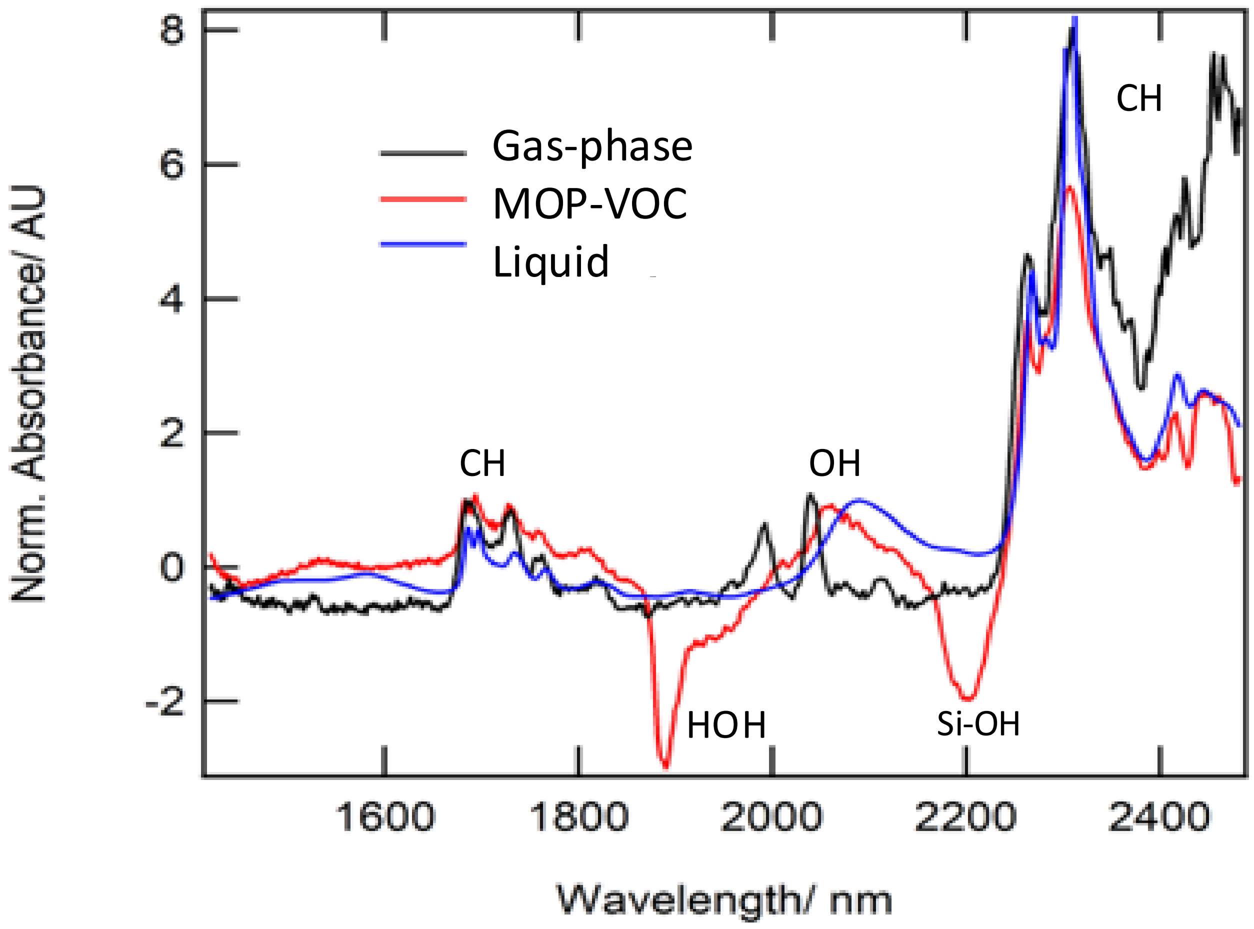

**4,000 x increase** in the sensitivity **per mm path-length** compared to free-space

# Gas, liquid and MOP-VOC

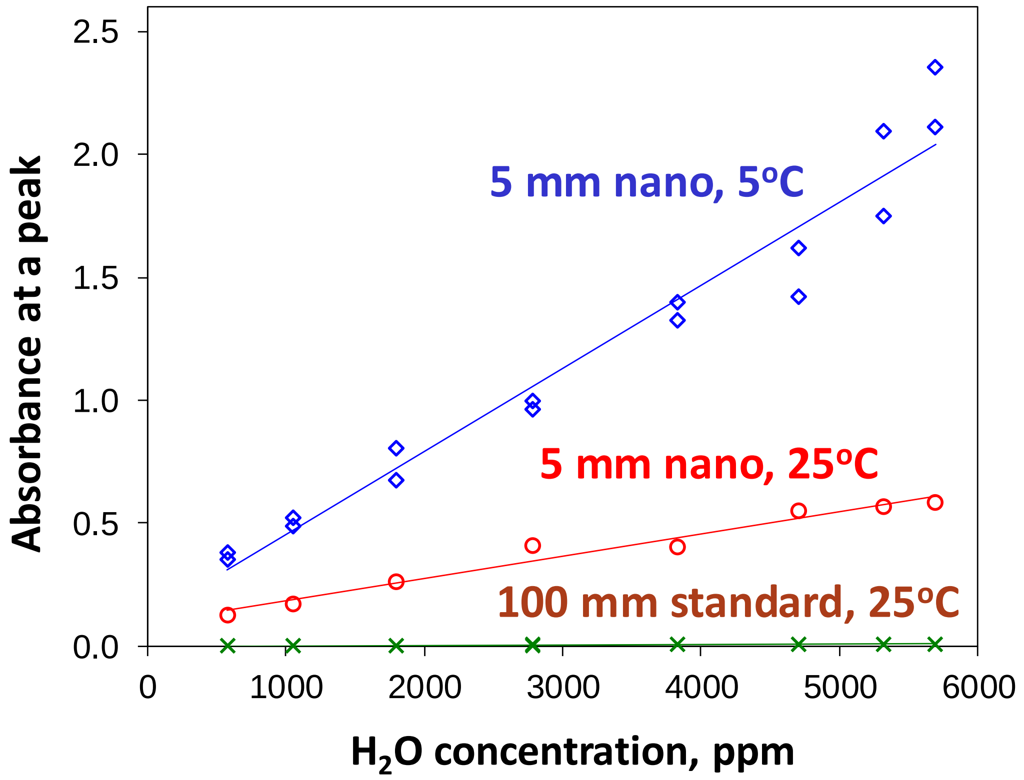
Isopropyl alcohol,
900 ppm

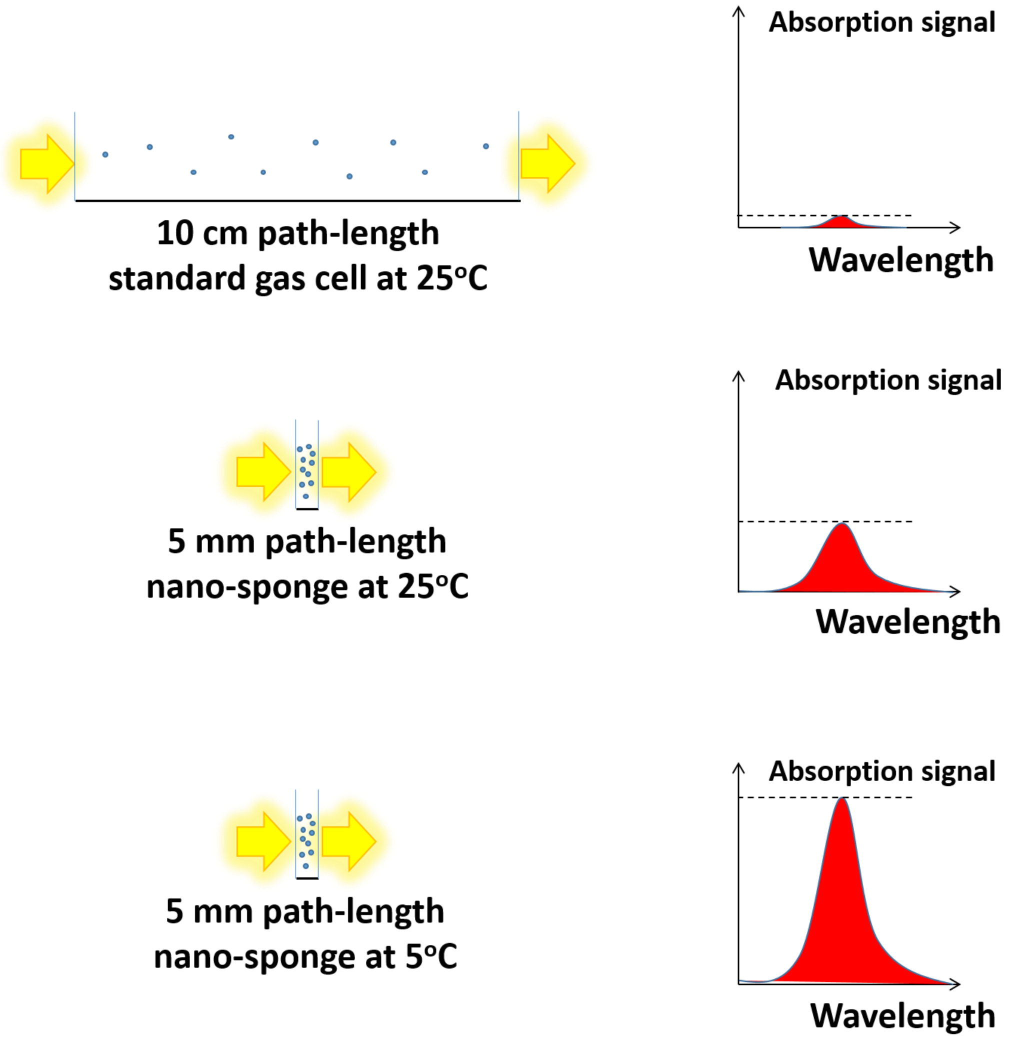



When compounds are concentrated in nanopores, they adopt a liquid-like phase.



# IR spectroscopy: standard vs MOP-VOC



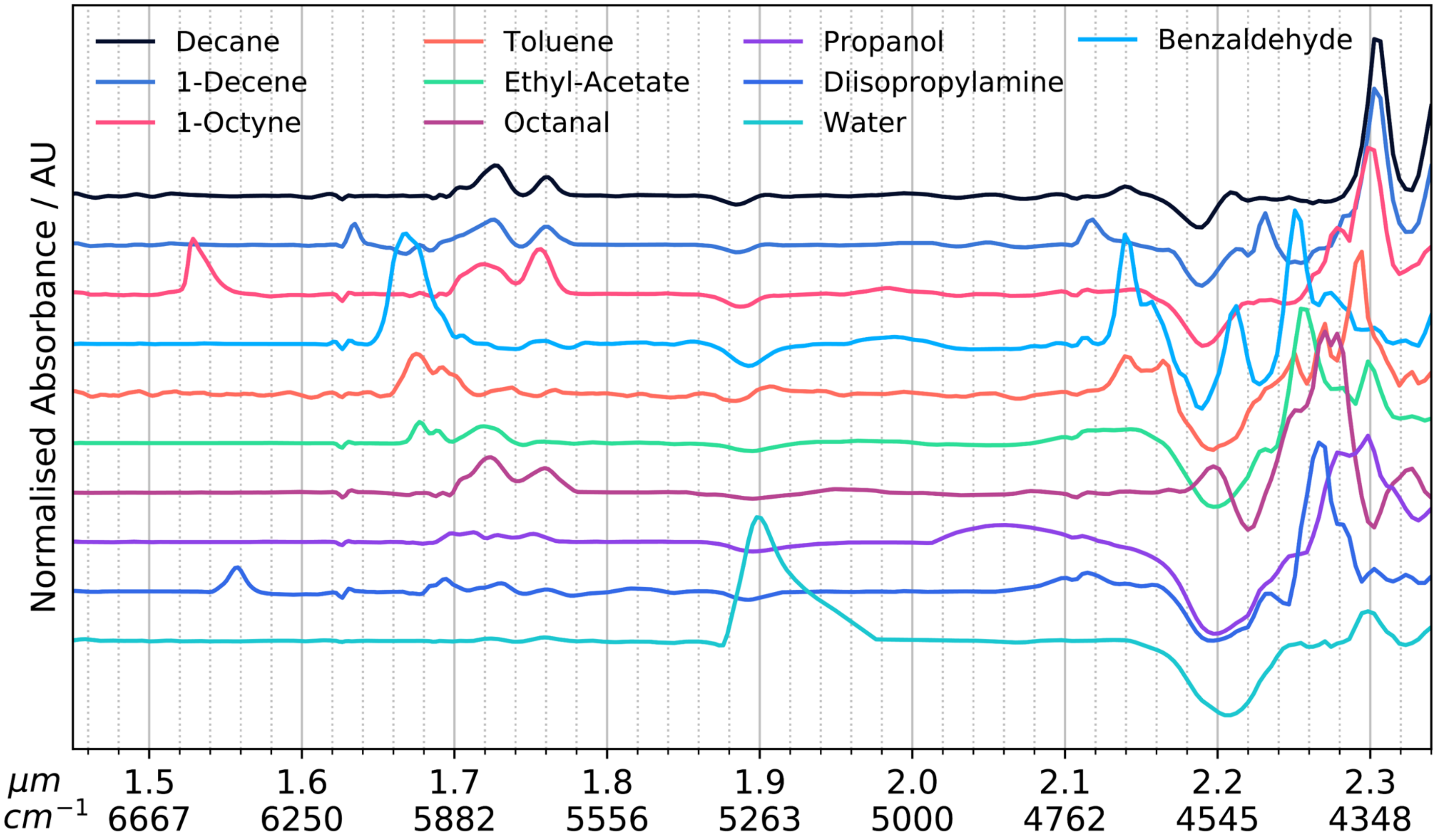




**5,000 x increase** in the sensitivity **per mm path-length** compared to free-space

@ 1055 ppm $H_2O$ and λ = 1900 nm

# Classification of VOCs in NIR using MOP-VOC

<u>Examples for:</u>
Alkene
Alkane
Alkyne
Aromatic
Ester
Aldehyde
Alcohol
Amine
Water



# Conclusions

- Nano-porous materials improve sensor sensitivity by adsorbing VOCs
- New nanoporous waveguide technology
  - Three orders of magnitude increase in sensitivity compared to standard free-space gas detection
  - Possible to distinguish between key chemical bonds using NIR (1-2.4 µm), therefore enabling selective VOC detection
  - Temperature modulation offers additional selectivity and ability to ’clean’ the waveguide between measurements
- Positioning of the porous waveguide, the porous alignment structures, and the silica optical fibers on the same substrate
  - No optical alignment required
  - Stable over thousands of thermal cycles

# Thank you

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