



**Óbuda University**  
**Doctoral school on Materials**  
**Sciences and Technologies**

**1<sup>st</sup> Semester progress presentation**  
**Spring 2016/17**

**by : Larbi Eddaif**

*May 25 2017*

# **Presentation Outlines:**

## **➤ Fullfiled tasks:**

- Results of the 1st semester.**
- Literature survey.**
- Research topic evaluations.**

## **➤ Future planed tasks.**

## **➤ Conculsions.**

# Fullfiled tasks:

- **Results of the 1<sup>st</sup> semester:**
- **Literature Survey:**
  - Topic of the research: *introduction, definition and importance,*
  - Heavy metals: hazardous and detection,
  - Sensors and Transducers: *definition and types.*
- **Research topic evaluations:**
  - Sensing materials and methods: *Polymers, methods of measurement,*
  - Immobilization of materials on Transducer,
  - Sensing layer characterization.

# Completed courses.

## Completed doctoral subjects

Courses & Neptun codes	Parts	Lecturer
Chemical Sensors: Methods and Applications. (OATEMAS1ND)		Dr. Shaban Abdul
Selected chapters about material testing methods. (OATVFAM1ND)	AFM and SEM techniques	Prof. Telegdi Judit
	IR and FTIR	Dr. Takacs erzsebet

# Fullfiled tasks: *in 1<sup>st</sup> semester*

- Results of the 1<sup>st</sup> semester:
- **Literature Survey:**
  - **Topic of the research: *introduction, definition and importance,***
  - **Heavy metals: hazardous and detection**
  - **Sensors and Transducers: *definition and types,***
- **Research topic evaluations:**
  - **Sensing materials and methods: *Polymers, methods of measurement.***
  - **Immobilization of materials on Transducer,**
  - **Sensing layer characterization.**

## Ph. D. Research Topic:

“Application of sensors in the detection of heavy metal ions in the environment”

## Experimental work at:

MTA TTK AKI

Hungarian Academy of Sciences

Research Centre for Natural Sciences

Institute of Materials and Environmental  
Chemistry



**Supervisor:**

Dr. Shaban Abdul



# Why we choose this topic?



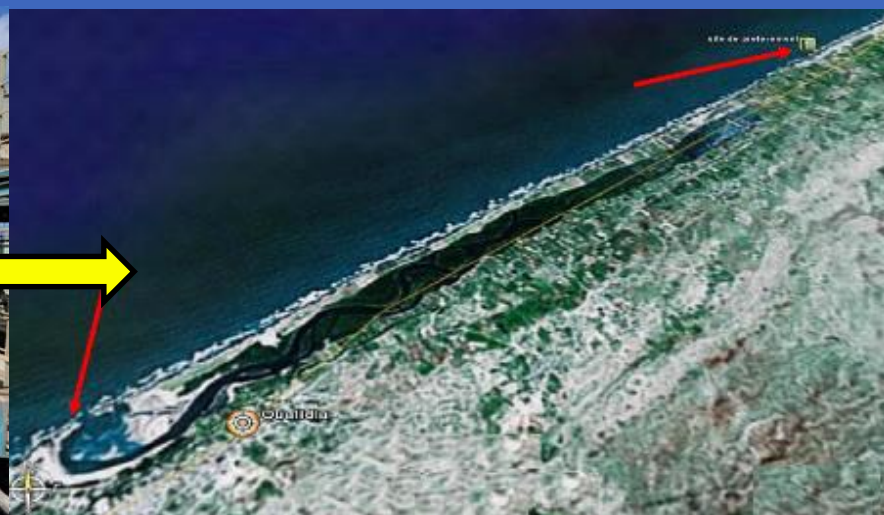
Tannery in Fès



Sebou river



OCP Jorf Lasfar



Atlantic Ocean

# Fullfiled tasks: *in 1<sup>st</sup> semester*

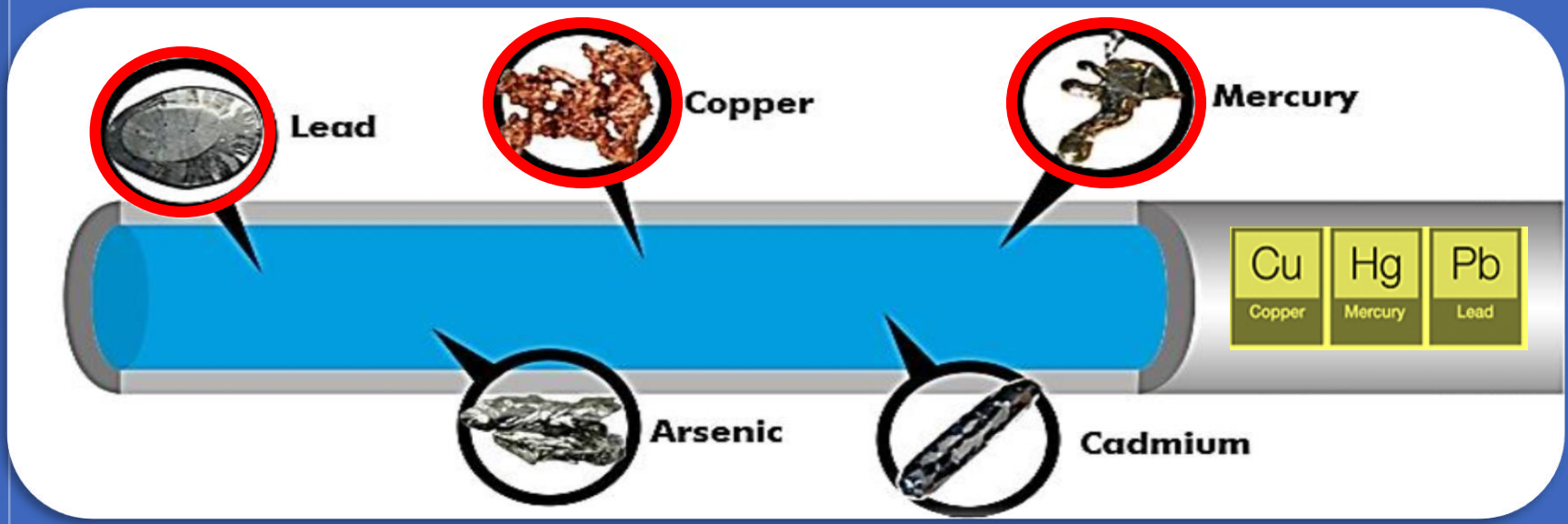
- Results of the 1<sup>st</sup> semester:
- **Literature Survey:**
  - Topic of the research: *introduction, definition and importance,*
  - **Heavy metals: hazardous and detection,**
  - Sensors and Transducers: *definition and types.*
- Research topic evaluations:
  - Sensing materials and methods: *Polymers, methods of measurement,*
  - Immobilization of materials on Transducer,
  - Sensing layer characterization.



# Heavy metals

## Heavy metals

- Metals exist naturally in the earth's crust but in varying concentrations.
- Heavy metals are omnipresent in the environment where they are harmless to living organisms



# Effects on Human Health

<b>Metal</b>	<b>Main source</b>	<b>Health effects</b>	<b>Maximum Perm. Limit (mg/L of water)</b>
<b>Lead (Pb)</b>	<b>Natural deposits, plumbing of old households...</b>	<b>Poor physical growth and learning disabilities in children, kidney problems, and high blood pressure in adults</b>	<b>0.015</b>
<b>Mercury (Hg)</b>	<b>Electrical industry, dentistry (dental amalgams), and in nuclear reactors...</b>	<b>Affects the central and peripheral nervous systems, digestive and immune systems, lungs and kidneys, and may be fatal.</b>	<b>0.002</b>
<b>Copper (Cu)</b>	<b>Household plumbing materials and industrial manufacture...</b>	<b>Gastro-intestinal distress and in the long run, experience liver or kidney damage.</b>	<b>1.300</b>

# Detection Techniques

## Conventional methods for heavy metals detection:



**Inductively coupled  
plasma/atomic emission  
spectrometry (ICP-AES)**



**Inductively coupled  
plasma/mass  
spectrometry (ICP-MS)**



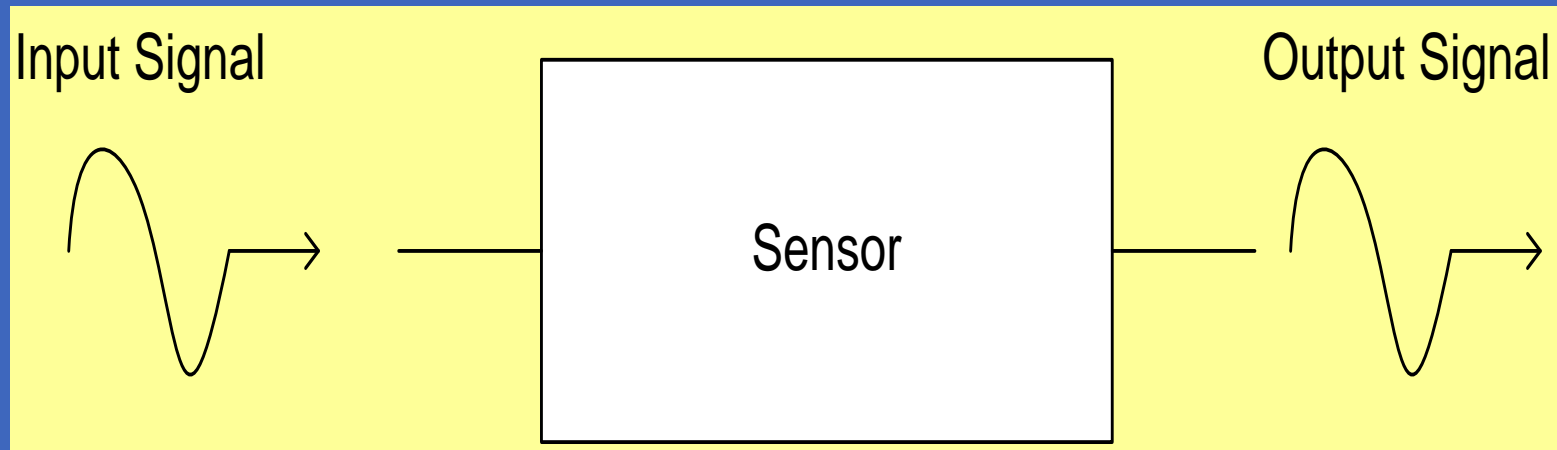
**Atomic absorption  
spectroscopy (AAS)**

# Fullfiled tasks: *in 1<sup>st</sup> semester*

- Results of the 1<sup>st</sup> semester:
- **Literature Survey:**
  - Topic of the research: *introduction, definition and importance,*
  - Heavy metals: hazardous and detection,
  - **Sensors and Transducers: *definition and types.***
- Research topic evaluations:
  - Sensing materials and methods: *Polymers, methods of measurement.*
  - Immobilization of materials on Transducer,
  - Sensing layer characterization.

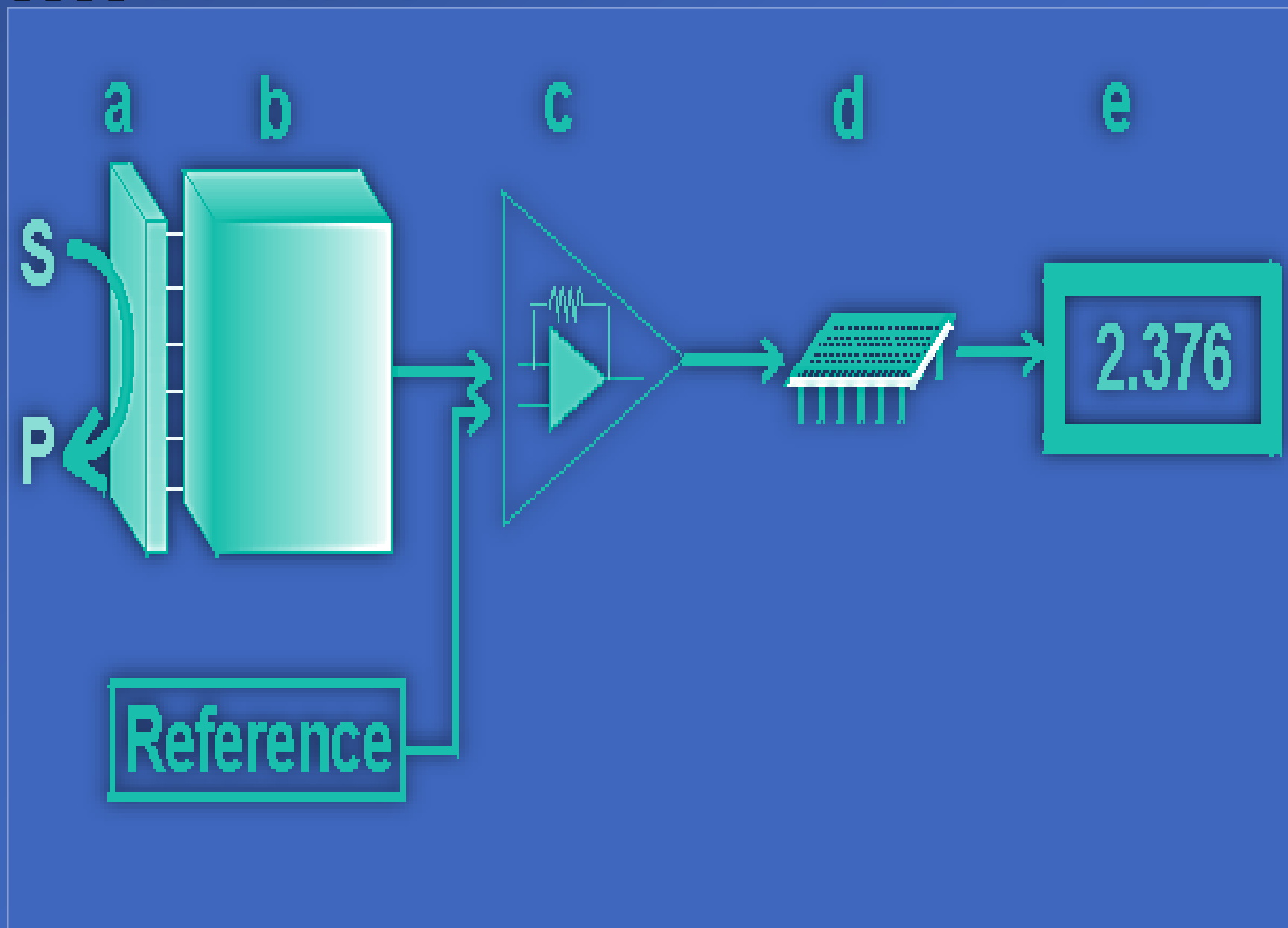
# Sensors and transducers

- A sensor acquires a physical parameter and converts it into a signal suitable for processing (e.g. optical, electrical, mechanical)





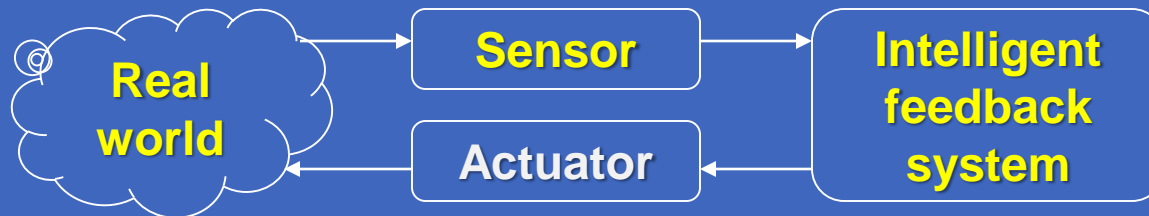
# Sensors



# Sensors and transducers

## ➤ Transducer:

- ✓ A device that converts a primary form of energy into a corresponding signal with a different energy form. Primary Energy Forms: mechanical, thermal, optical, chemical, electromagnetic, etc....



# Specifications of Sensor

- **Accuracy:** error between the result of a measurement and the true value being measured.
- **Resolution:** the smallest increment of measure that a device can make.
- **Sensitivity:** the ratio between the change in the output signal to a small change in input physical signal. Slope of the input-output fit line.
- **Repeatability/Precision:** the ability of the sensor to output the same value for the same input over a number of trials

# Specifications of Sensor

- **Accuracy:** error between the result of a measurement and the true value being measured.
- **Resolution:** the smallest increment of measure that a device can make.
- **Sensitivity:** the ratio between the change in the output signal to a small change in input physical signal. Slope of the input-output fit line.
- **Repeatability/Precision:** the ability of the sensor to output the same value for the same input over a number of trials

# Specifications of Sensor

- **Accuracy:** error between the result of a measurement and the true value being measured.
- **Resolution:** the smallest increment of measure that a device can make.
- **Sensitivity:** the ratio between the change in the output signal to a small change in input physical signal. Slope of the input-output fit line.
- **Repeatability/Precision:** the ability of the sensor to output the same value for the same input over a number of trials

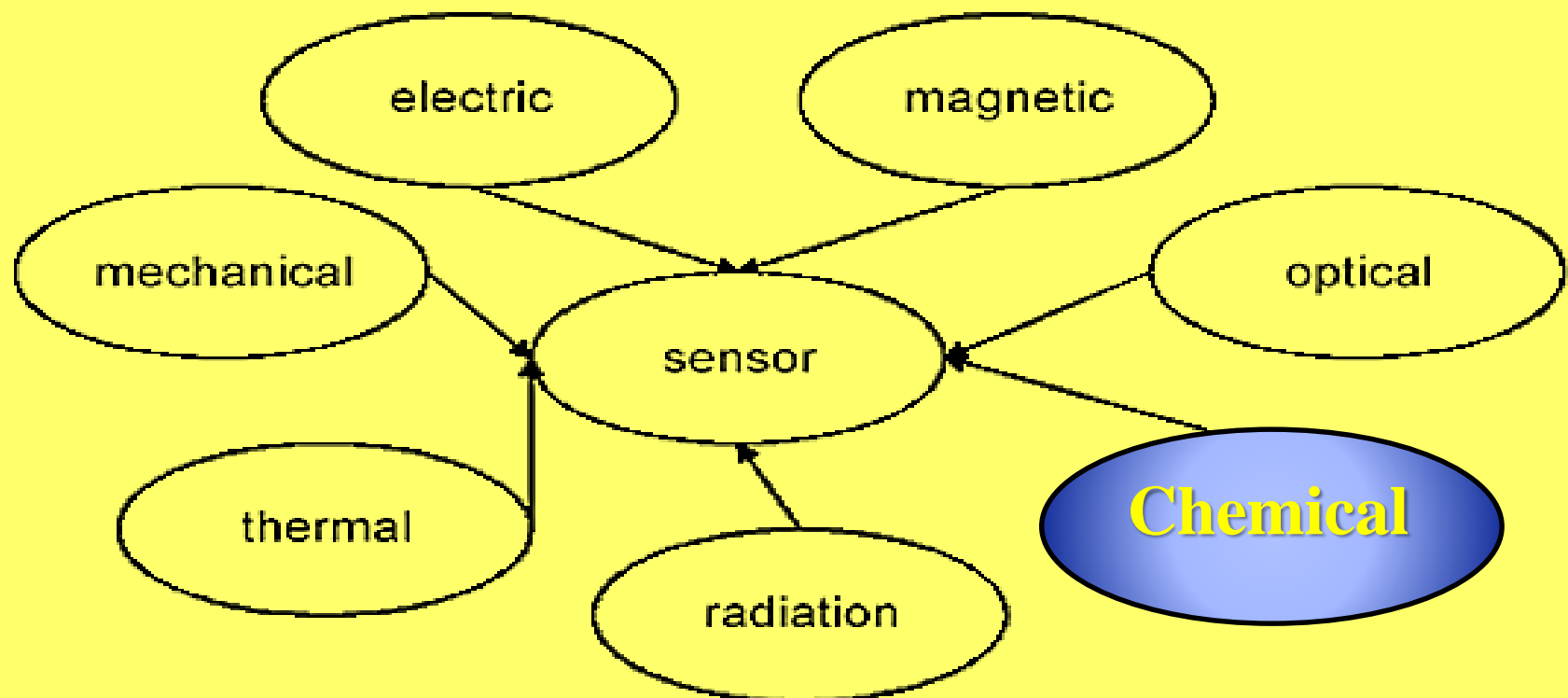


# Specifications of Sensor

- **Accuracy:** error between the result of a measurement and the true value being measured.
- **Resolution:** the smallest increment of measure that a device can make.
- **Sensitivity:** the ratio between the change in the output signal to a small change in input physical signal. Slope of the input-output fit line.
- **Repeatability/Precision:** the ability of the sensor to output the same value for the same input over a number of trials

# Sensors types:

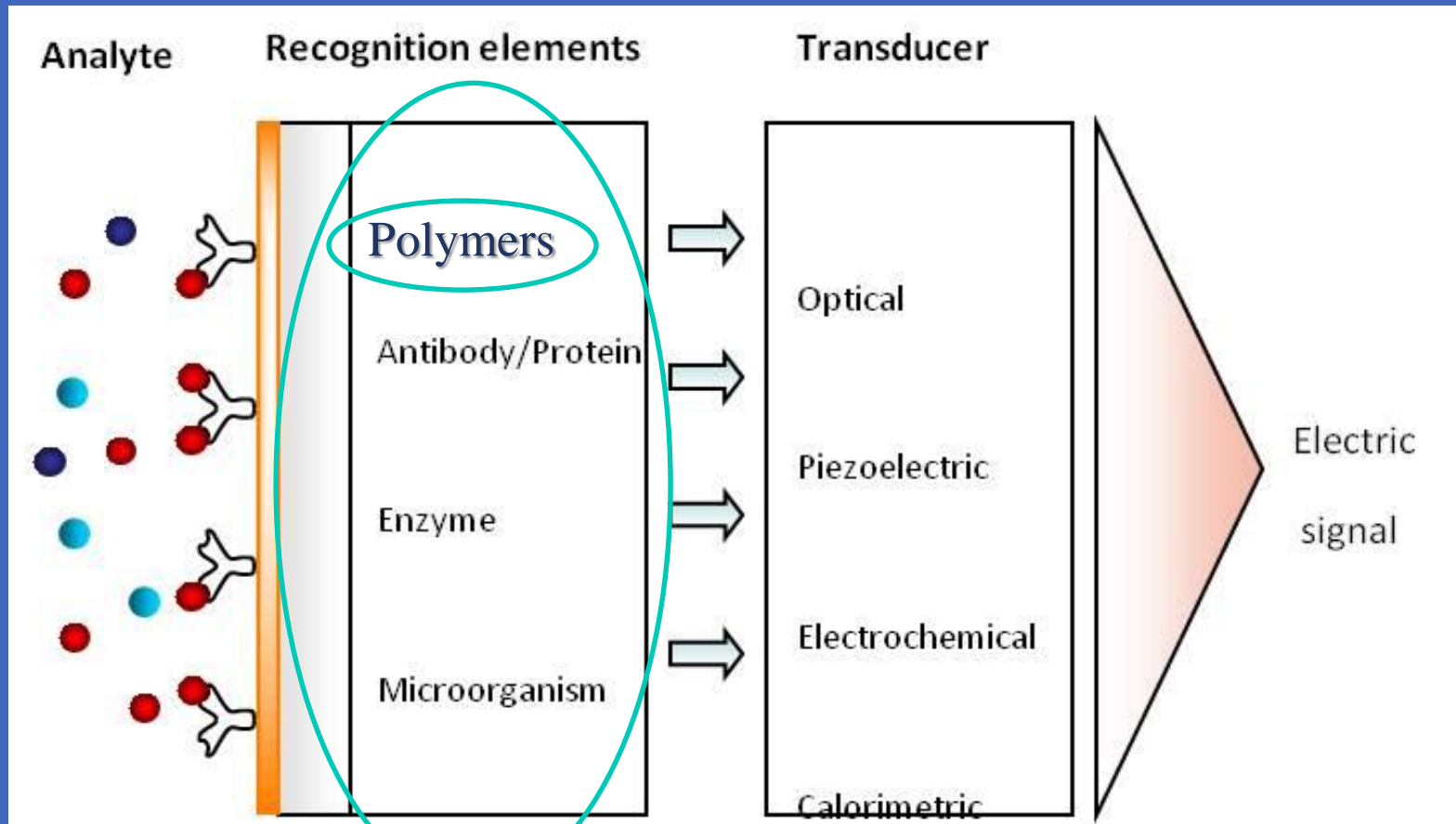
It is very difficult to classify sensors under one criterion and hence different criteria may be adopted for the purpose. Some of these include: Transduction principles, primary input quantity, material and technology, applications & properties.



# Fullfiled tasks: *in 1<sup>st</sup> semester*

- Results of the 1<sup>st</sup> semester:
- Literature Survey:
  - Topic of the research: *introduction, definition and importance,*
  - Heavy metals: hazardous and detection,
  - Sensors and Transducers: *definition and types.*
- **Research topic evaluations:**
  - Sensing materials and methods: *Polymers, methods of measurement,*
  - Immobilization of materials on Transducer,
  - Sensing layer characterization.

# Sensing materials:



# Sensing materials

## Sensing materials

- A **polymer** is a large molecule, or macromolecule, composed of many repeated subunits called **monomers** .
- In this project we'll use:
  - Cyclodextrine (CD)
  - Calixarenes.



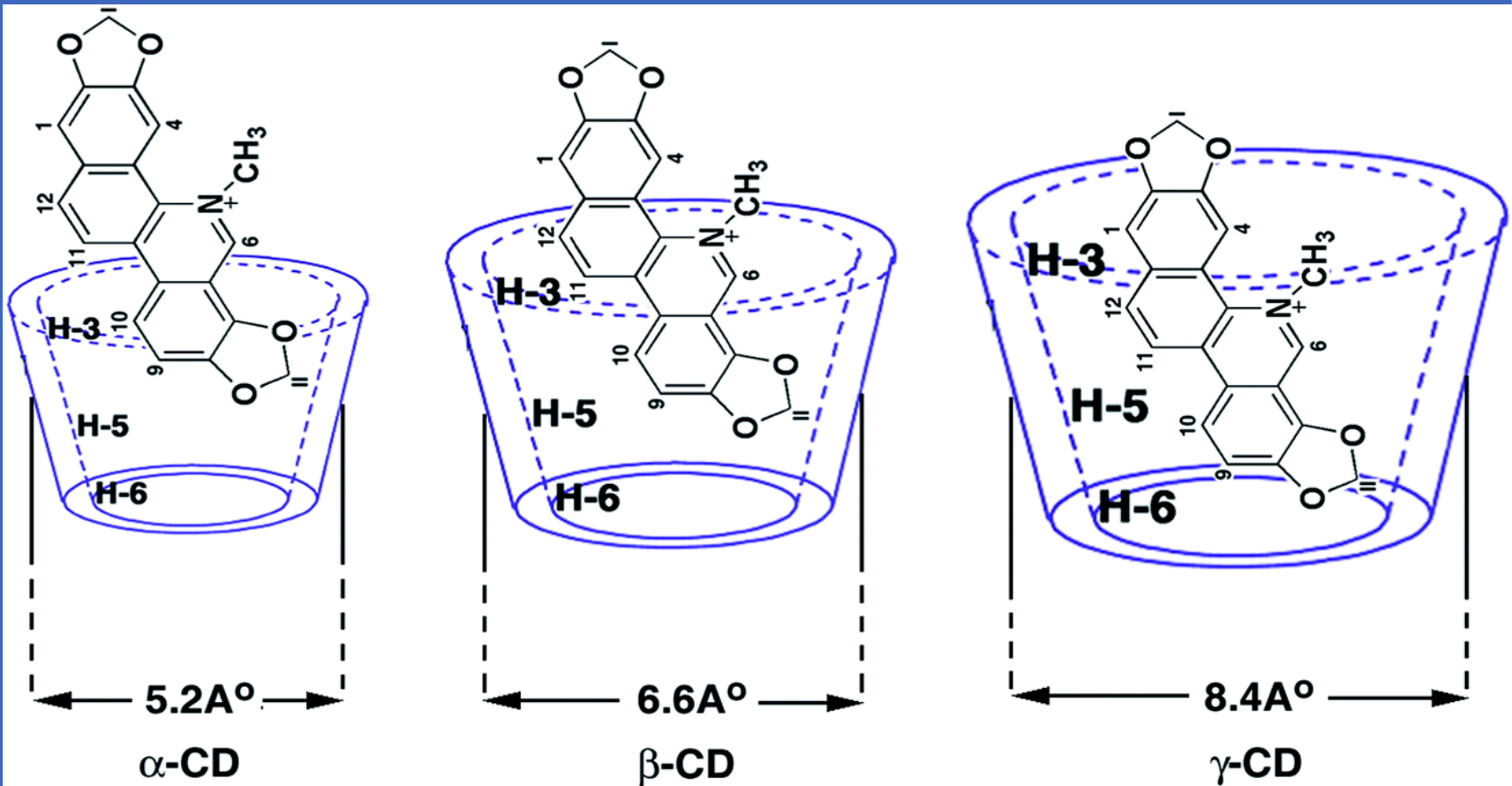
# Sensing materials

There are 3 types of CD:

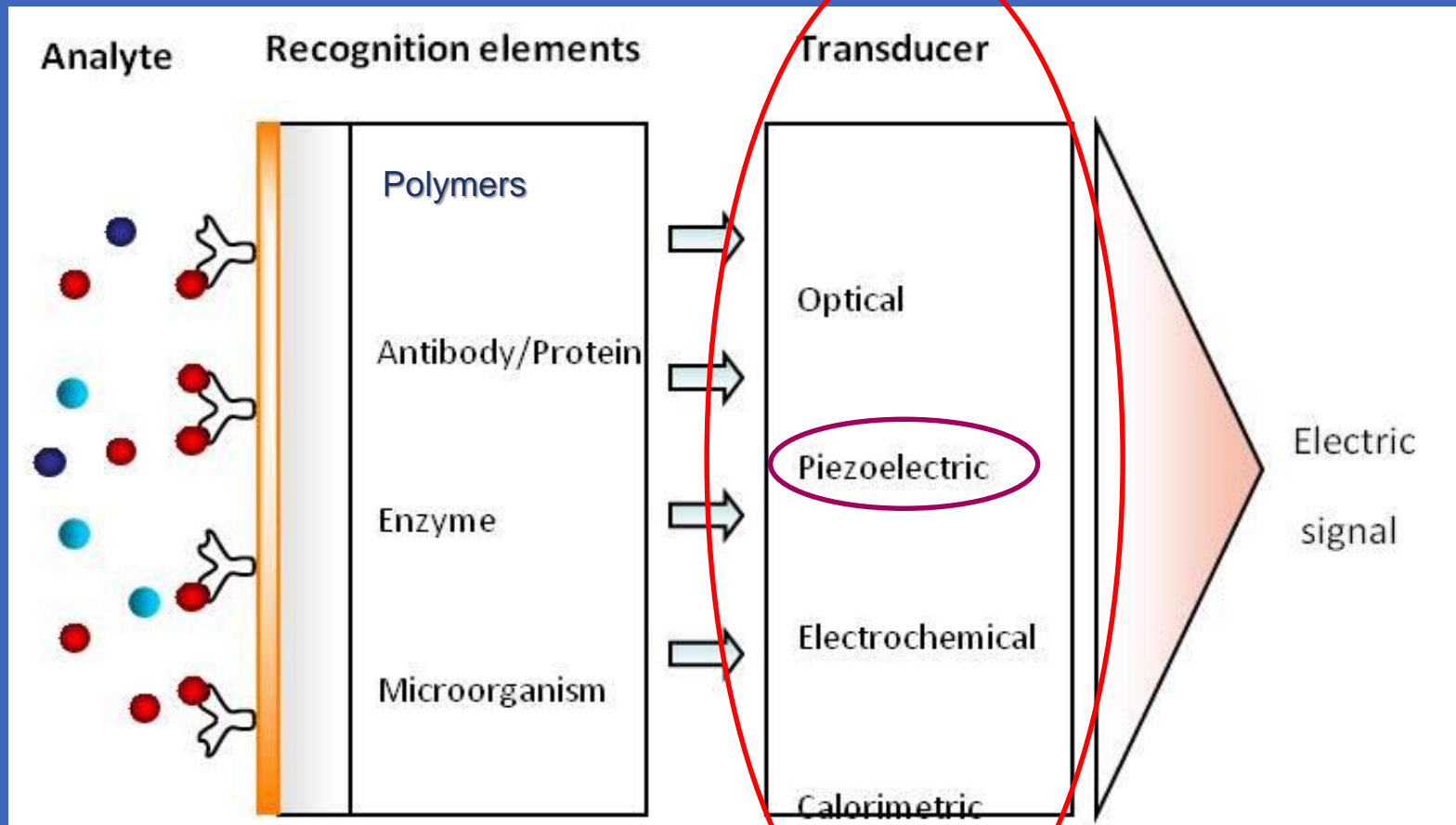
$\alpha$  (alpha)-cyclodextrin: 6-membered sugar ring molecule

$\beta$  (beta)-cyclodextrin: 7-membered sugar ring molecule

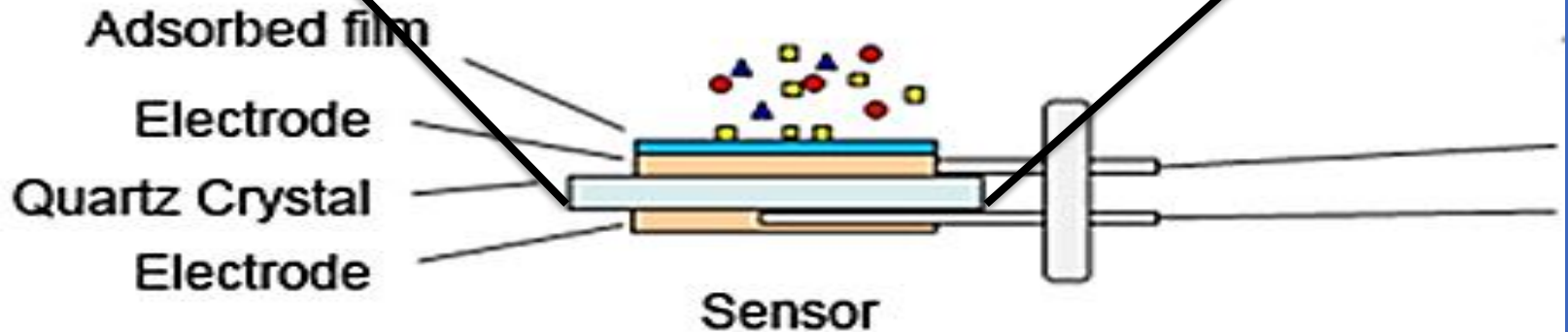
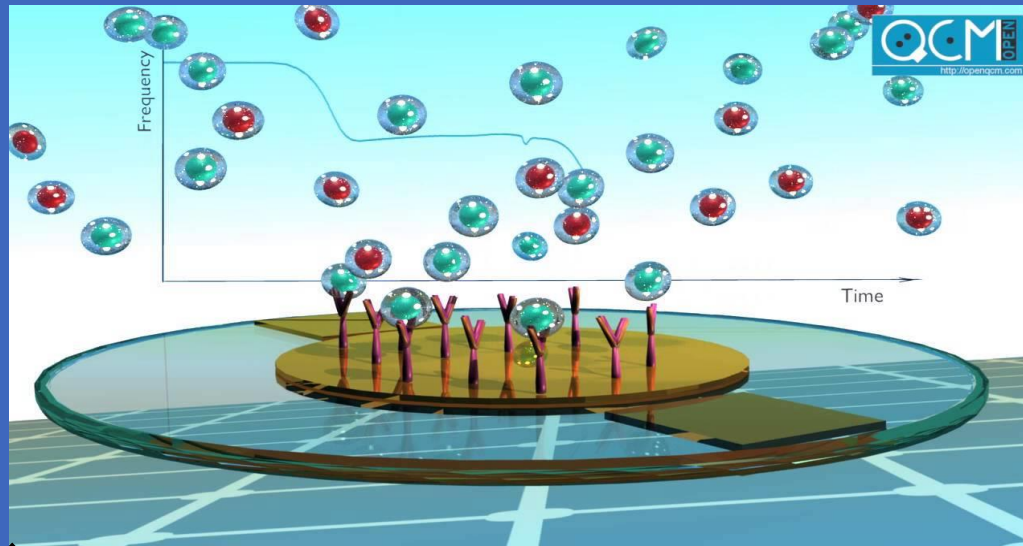
$\gamma$  (gamma)-cyclodextrin: 8-membered sugar ring molecule



# Sensing methods:

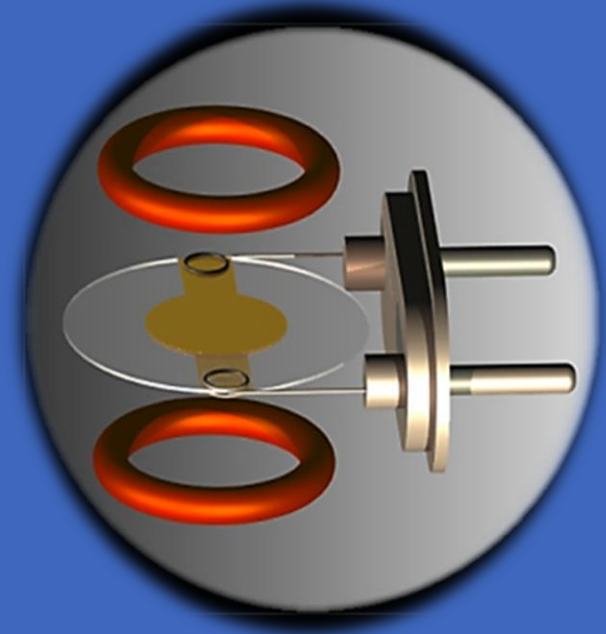
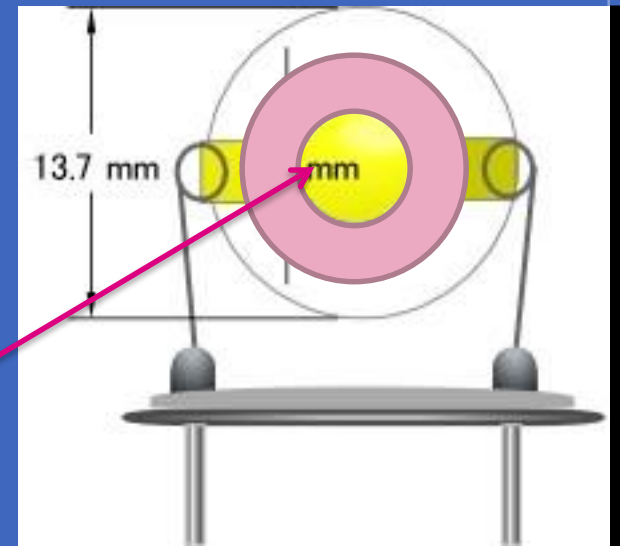
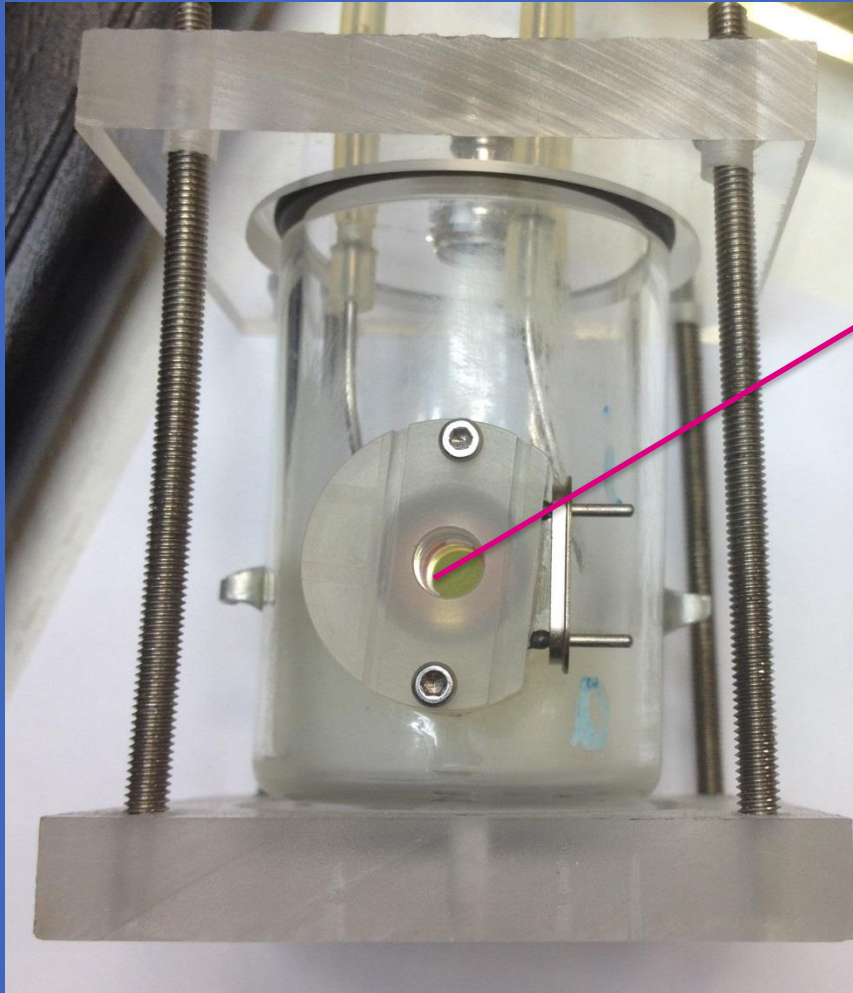


# Sensing methods QCM



Frequency changes according to adsorption of chemicals on the surface of QCM sensor.

# QCM measuring cell





# QCM-I: Quartz Crystal Microbalance with Impedance Measurement

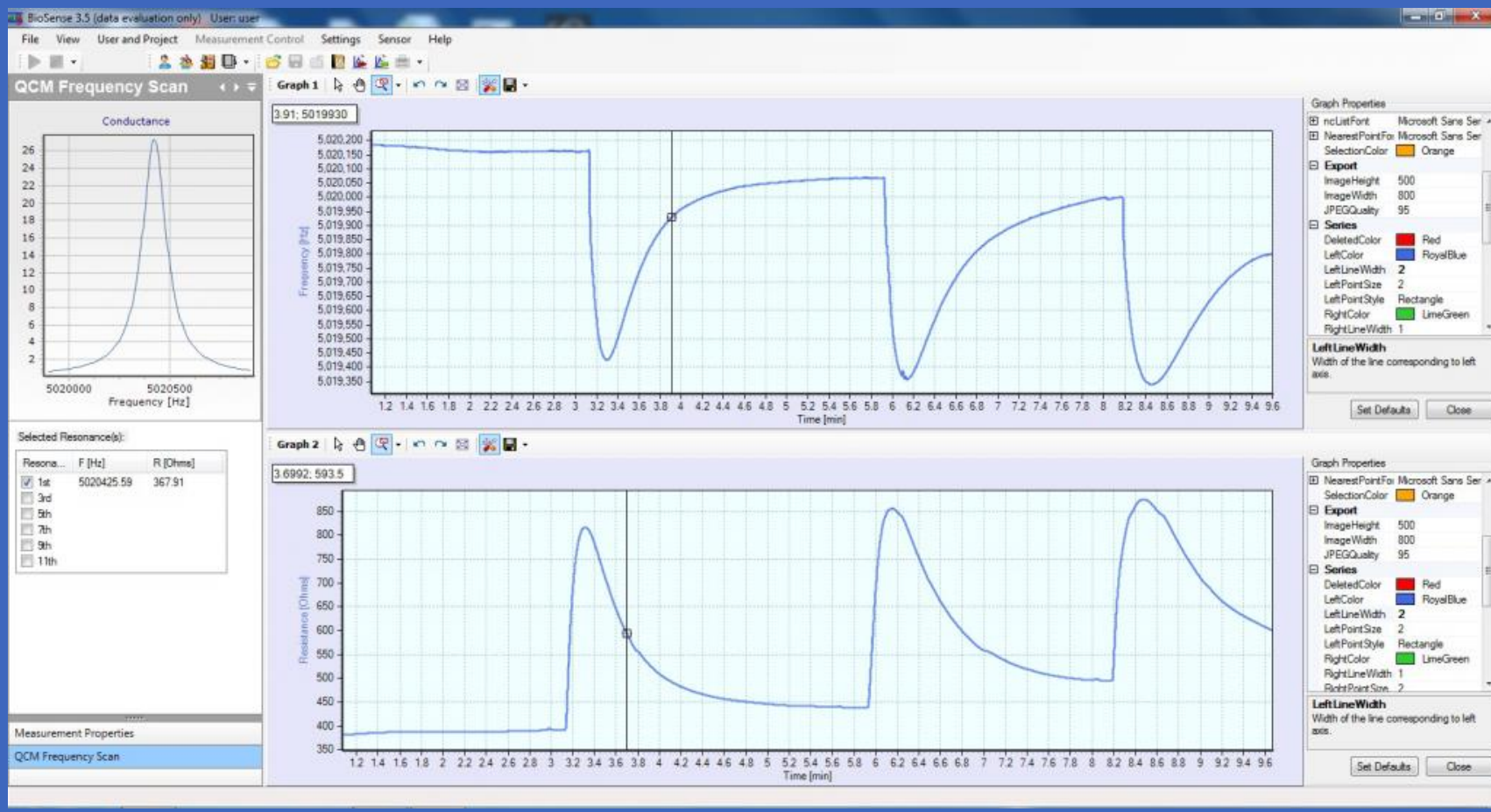




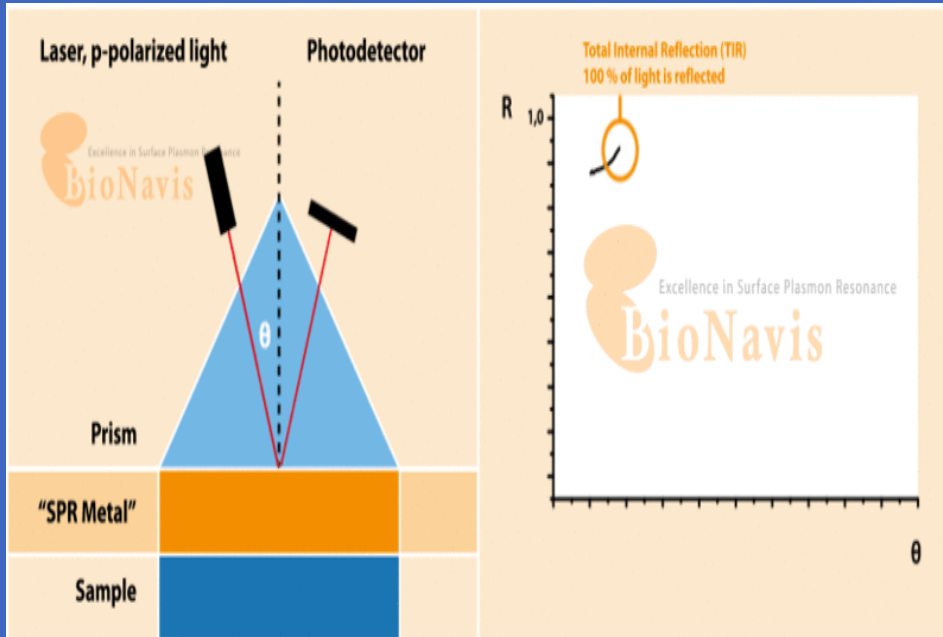
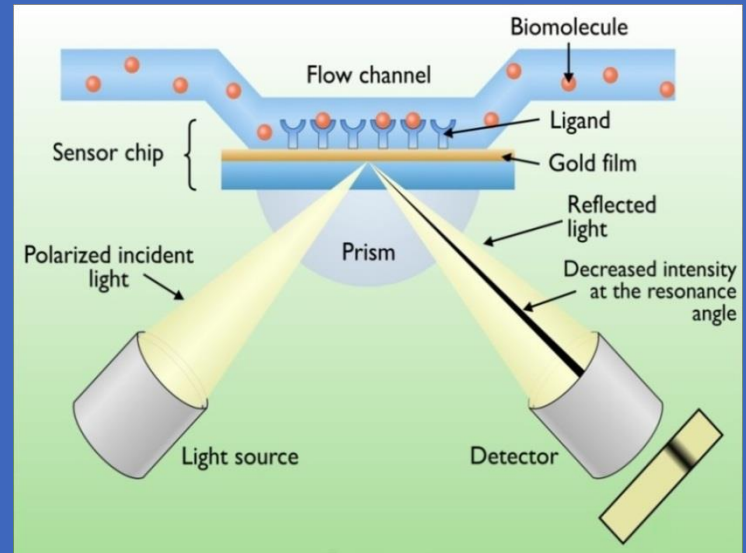
# QCM-I: Quartz Crystal Microbalance with Impedance Measurement

We can measure the layer properties for example:

Changes of frequency (mass), dissipation energy, charge, and resonance frequency at different overtones: for 5 Mhz crystal: 5, 15, 25 MHz.



# Sensing methods: Surface plasmon resonance (SPR)



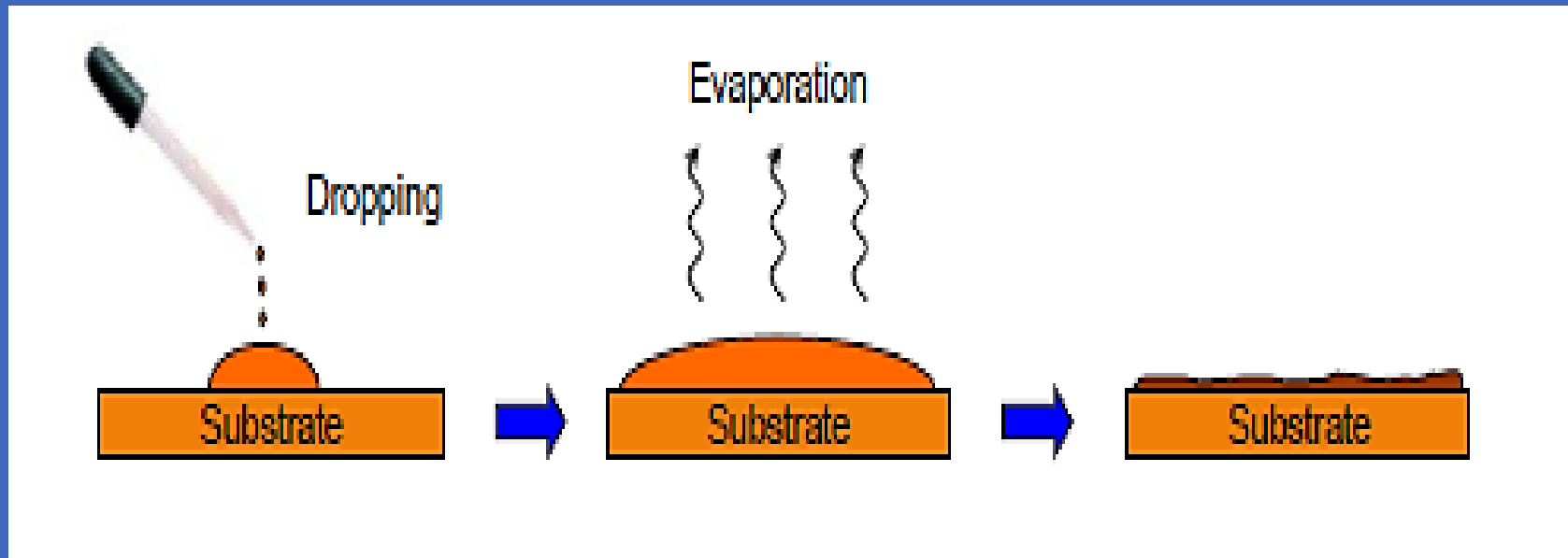
# Fullfiled tasks: *in 1<sup>st</sup> semester*

- Results of the 1<sup>st</sup> semester:
- Literature Survey:
  - Topic of the research: *introduction, definition and importance,*
  - Heavy metals: hazardous and detection,
  - Sensors and Transducers: *definition and types.*
- **Research topic evaluations:**
  - Sensing materials and methods: *Polymers, methods of measurement,*
  - **Immobilization of materials on Transducer,**
  - Sensing layer characterization.

# Immobilization methods:

## Drop coating

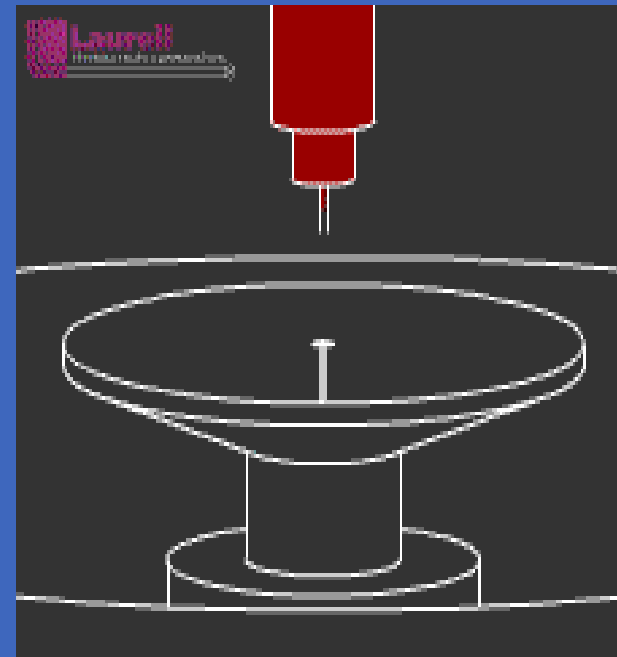
Dropping of solution and spontaneous solvent evaporation



# Immobilization methods:

## Spin coating

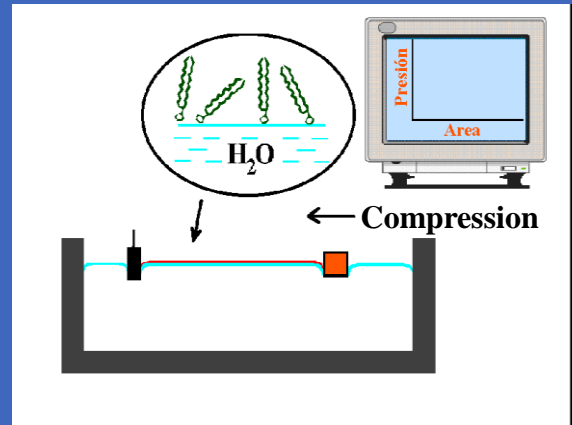
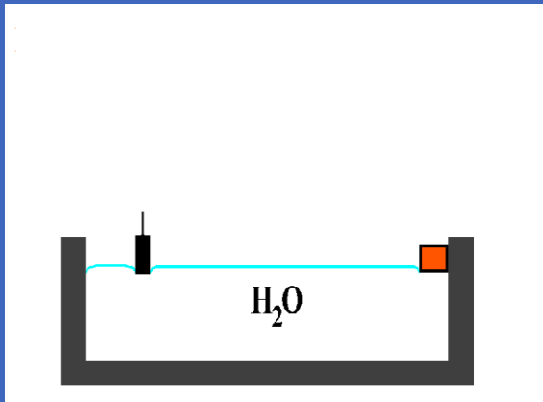
### Dropping on spinning substrate



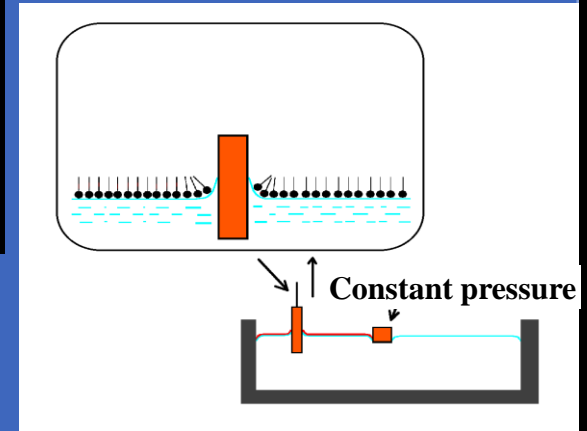
# Immobilization methods:

## Langmuir layers

The substrate is dipped into the solution and then withdrawn at a controlled speed.



Transition of mono- and multimolecular layers





# Fullfiled tasks: *in 1<sup>st</sup> semester*

- Results of the 1<sup>st</sup> semester:
- Literature Survey:
  - Topic of the research: *introduction, definition and importance,*
  - Heavy metals: hazardous and detection,
  - Sensors and Transducers: *definition and types.*
- **Research topic evaluations:**
  - Sensing materials and methods: *Polymers, methods of measurement.*
  - Immobilization of materials on Transducer,
  - **Sensing layer characterization.**

# Layer characterization : AFM

## Qualitative analysis

The AFM offers visualization in three dimensions. Resolution in the vertical, or Z, axis is limited by the vibration environment of the instrument, whereas resolution in the horizontal, or X-Y, axis is limited by the diameter of tip utilized for scanning.

Typically, AFM instruments have vertical resolutions of less than 0.1 nm and X-Y resolutions of around 1 nm.

# Layer characterization : ESEM

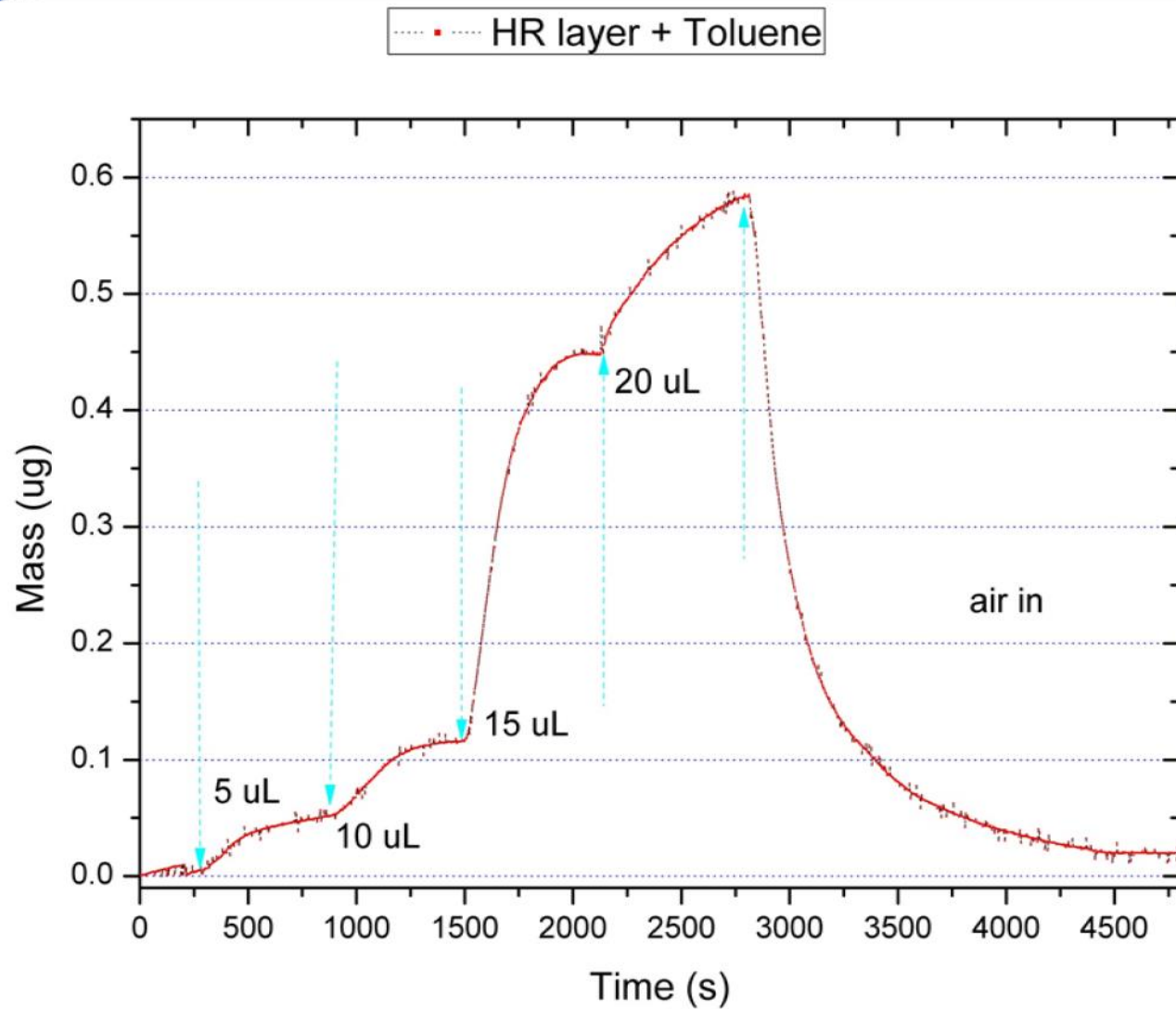
## Environmental SEM

In ESEM, samples can be looked at in a low pressure gas environment. While using ESEM it is not necessary to make nonconductive samples conductive. Materials samples do not need to be desiccated and coated with gold-palladium, for example, and thus their original characteristics may be preserved for further testing or manipulation. The GSED is set up to collect secondary electrons very efficiently.

# QCM measurements.

- Detection of toluene by a sensing layer of  $\beta$ -CD, code named HR.
- Volume of the polymer layer: 20 uL.
- Drop coating.
- Regenerating media: air.
- Temperature: room temp.

# QCM measurements.





# **Future planned tasks:**

- ✓ **Modification of sensor surface which enables us to obtain surface-modified electrodes for detecting heavy metals.**
- ✓ **Immobilization of polymers on sensing element surface and perform measurements using the QCM, QCM-I and SPR.**
- ✓ **2 courses related to the topic of polymers will be taken in the coming semester in order to strengthen the background.**



# Conclusions:

- ✓ Literature survey is well underway.
- ✓ New tool for detection of heavy-metals will be studied.
- ✓ These new tools offer new opportunities with many advantages.
- ✓ So we wish ourselves a lot of success for this topic to be one of the new trends in heavy metal sensing.



**Last but not the least:**

**I would like to thank the “Doctoral School Office” for the support and understanding of my circumstances during the past semester.**

**Köszönöm a figyelmet**

Thanks  
شكراً  
Shukran

