

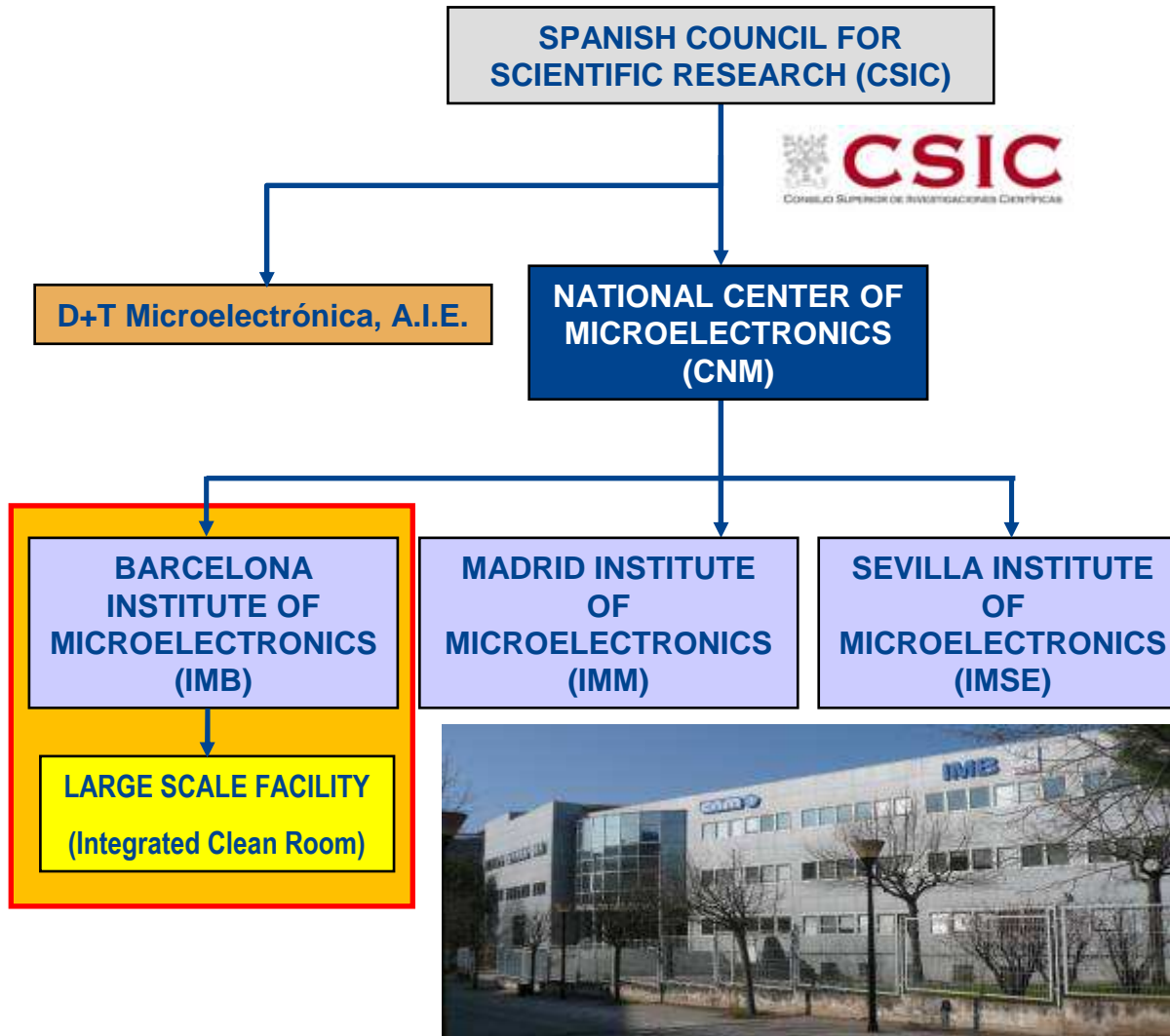
# Sistemes multisensors aplicats al control de qualitat dels vins

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# Barcelona Institute of Microelectronics (IMB-CNM), CSIC



2011 IMB Budget: 11.5 M€  
External funding: 50 %

IMB STAFF (2012)	
• Researchers	66
• Ph.D. Students	57
• Clean room	43
• Support services	28
• Admin. & general serv.	20
• Visitors	5
<b>TOTAL:</b>	<b>219</b>

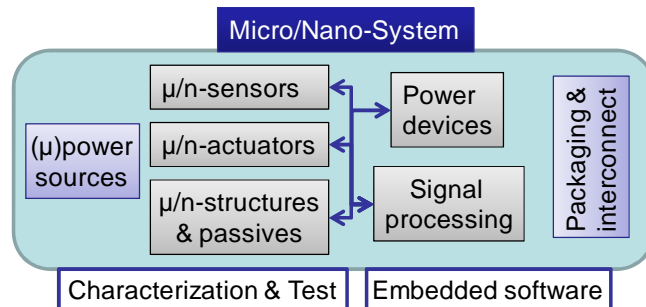


## Research at IMB:

- Micro – Nanotechnologies
- Nanofabrication and functional properties of nanostructures
- **Transducers for chemical and biochemical sensing**
- Micro-Nano-Bio systems
- Integrated circuits and systems
- Power devices and systems

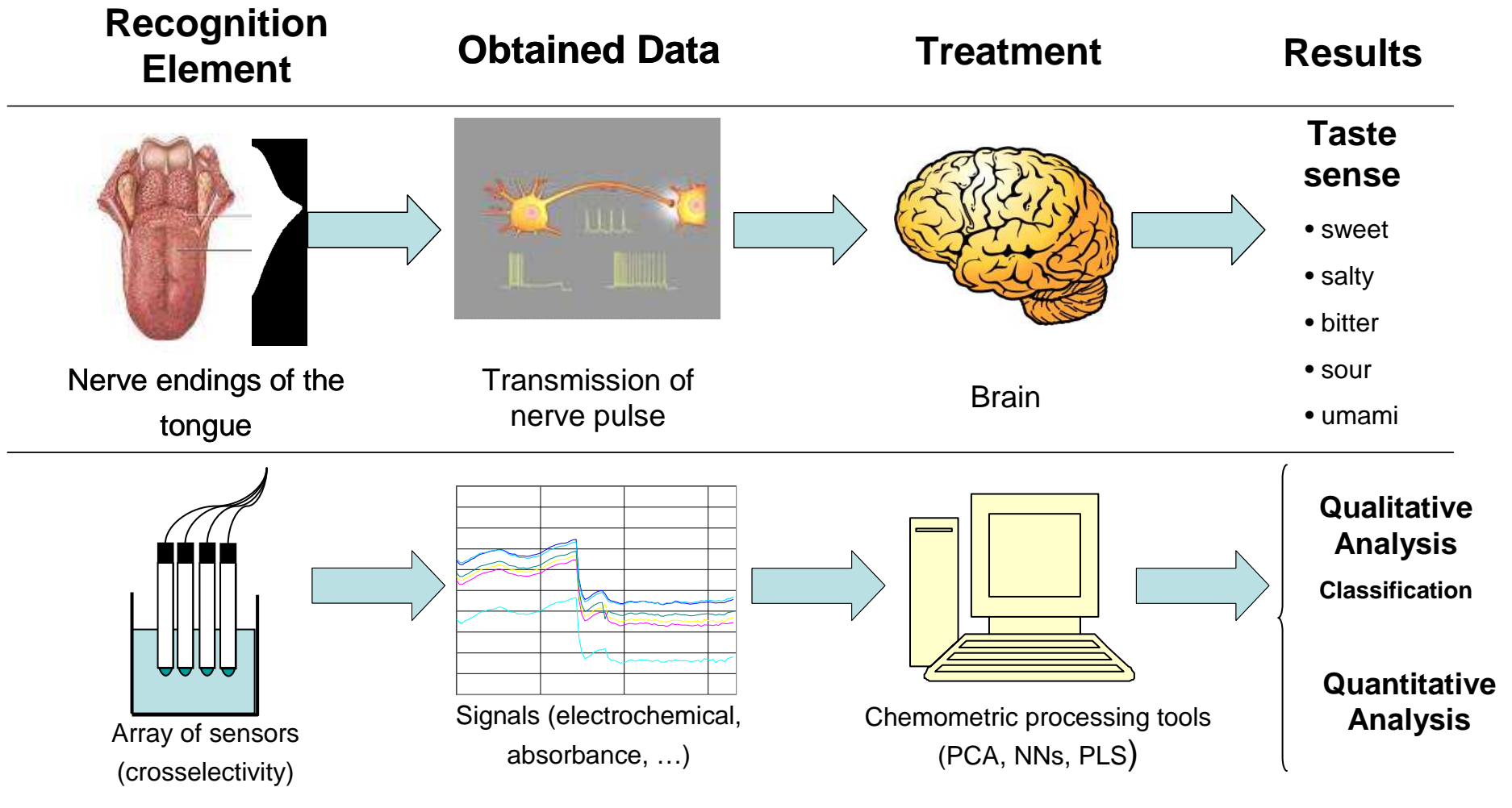


## Technology focus : MICRO & NANO INTEGRATED SYSTEMS





- Introduction to Multisensor systems or Electronic Tongues
- Microelectrodes used
  - ISFET based sensors
  - Thin film metal microelectrodes
  - Optical integrated systems
- Chemometric tools
- Results
  - Sensor signals & treatment of data
  - Classification of wines:
    - Grape variety/ vintage year/ geographical origin
  - Quantitative analysis
- Conclusions

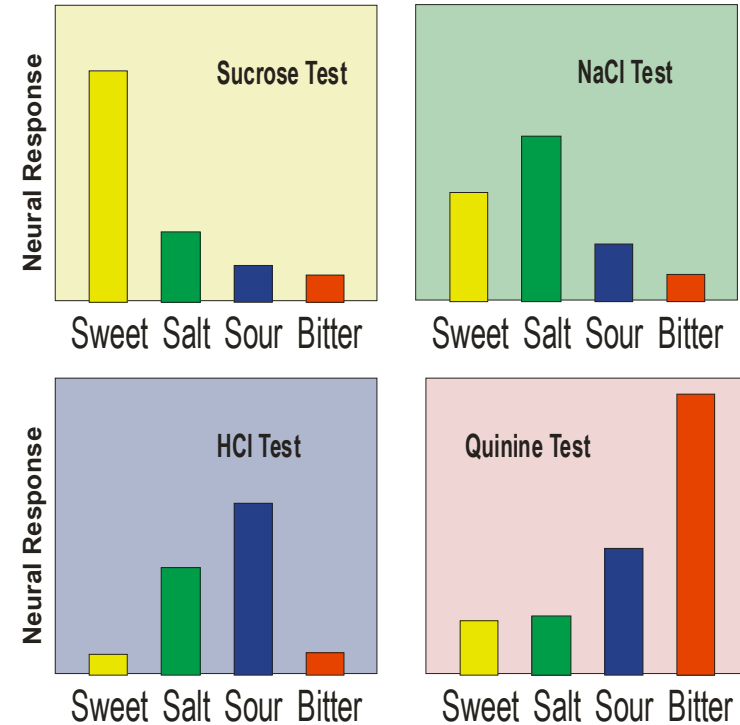
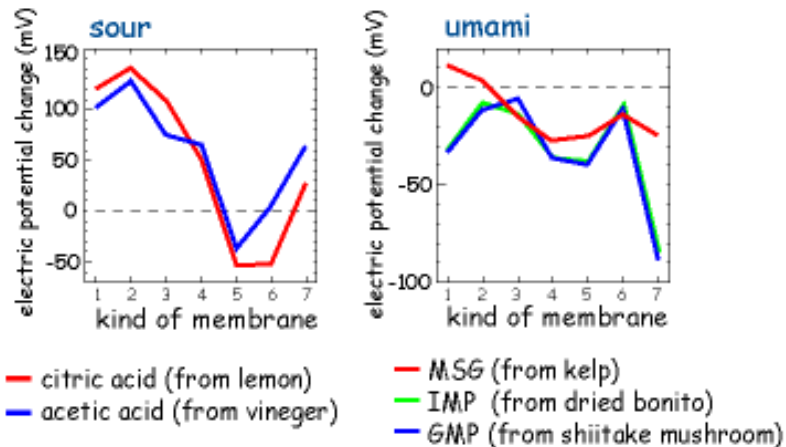


**Sabor: Sensación debida a conjunto de sustancias**

**Categorías:**

- **Salado:** NaCl
- **Dulce:** glucosa, fructosa
- **Amargo:** quinina, cafeína
- **Acido:** HCl, ácido acético
- **“Umami”:** glutamato monosódico (MSG)

**Respuesta de sistema gustativo a distintos compuestos**



Toko, K., *Taste sensor. Sensors and Actuators B-Chemical*, 2000. **64**(1-3): p. 205-215.

**Respuesta de conjunto de sensores a distintos compuestos**

- Introduction to Multisensor systems or Electronic Tongues
- **Microelectrodes used**
  - **ISFET based sensors**
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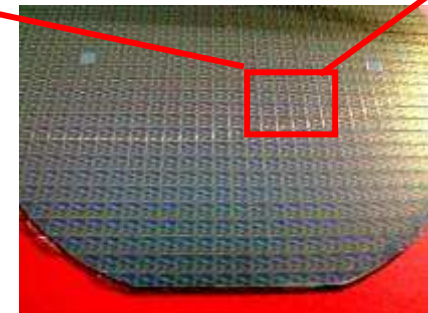
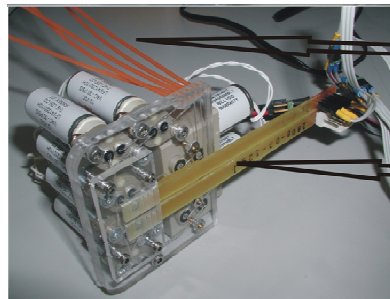
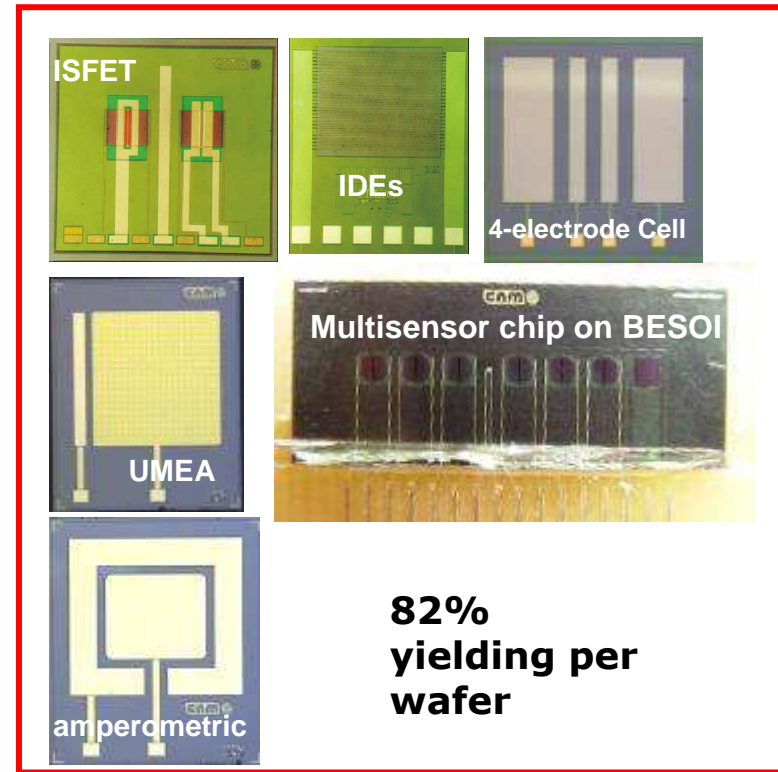
## Fabricated with microelectronic technology

### Advantages

- Low cost (mass produced)
- Low energy consumption
- Reproducible
- Miniaturization ( $\mu$ TAS or LOC)
- Circuit integration

### Type of transducers:

- **ISFET based sensors:** pH, ions Na, K, Ca, Cl, NO<sub>3</sub>, etc
- **Microelectrodes and UMEAs** (Voltamperometry): Cl<sub>2</sub> and COD, heavy metals, molecules
- **Interdigitated electrodes (IDEs):** conductivity, dielectric properties.
- **Multisensor chips** (compatibilization of fabrication technologies)

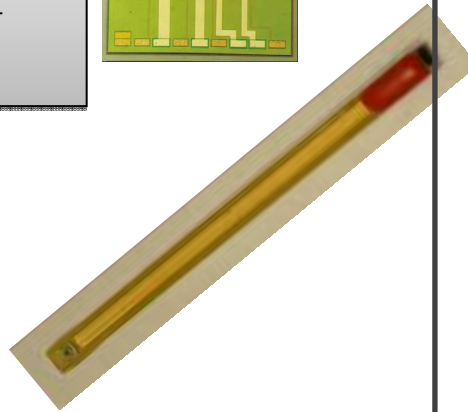
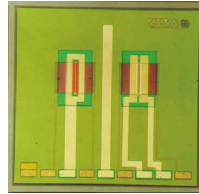




# ISFET and CHEMFETs

## Detection of:

- pH
- Cations  $\text{Ca}^{2+}$ ,  $\text{K}^+$ ,  $\text{Na}^+$
- Anions:  $\text{Cl}^-$ ,  $\text{CO}_3^{2-}$



**Product Information**

**ISFET pH Sensors**

Ion Sensitive Field Effect Transistors (ISFETs) are non-glass pH electrodes. Their fabrication with microelectronic technologies offers advantages like robustness, small size and low cost compared to standard pH glass electrodes.

IMB-CNM fabricates several designs of ISFETs. These contain  $\text{Si}_3\text{N}_4$  and  $\text{SiO}_2$  gate membranes for measuring pH and for depositing ion selective membranes respectively.

**Applications**

Biomedical analysis, environmental monitoring and industrial process control are attractive applications of ISFET chemical sensors.

**Technologies**

- Standard CMOS based technologies
- Fully CMOS compatible technology with on-chip circuit integration
- Biocompatible and Organic membranes for ion selective detection.
- Automatic packaging based on thermocurable and photocurable encapsulant polymers.

**Device features**

- Small size, adaptable to miniaturized systems
- Robustness due to non-glass and solid state nature
- Response time up to 10 times faster than glass electrode
- High long-term stability
- High reproducibility
- Low impedance output signal
- Low cost (mass production)
- Stores dry, no maintenance required

**Contact:**

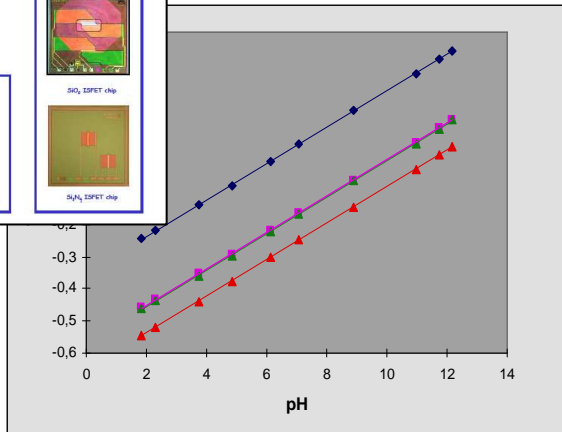
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Tel: +34 3 588 77 00  
Fax: +34 3 588 14 92  
e-mail: dimit@gtq.com  
http://www.gtq.com

**Detail of an encapsulated ISFET sensor**

**SiO<sub>2</sub> ISFET chip**

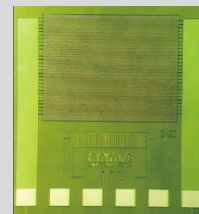
**Si<sub>3</sub>N<sub>4</sub> ISFET chip**



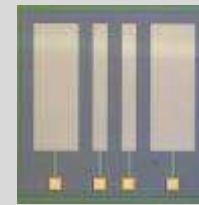
# Pt/Au Microelectrodes

- Conductivity/impedance
- Redox Potential (ORP)
- Dissolved Oxygen ( $\text{O}_2$ )
- Chloride ( $\text{Cl}_2$ )
- Heavy Metals
- Electrochemical oxygen demand (EOD)

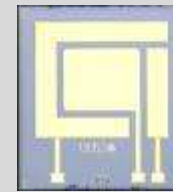
## Standard technology Si/SiO<sub>2</sub>/metal



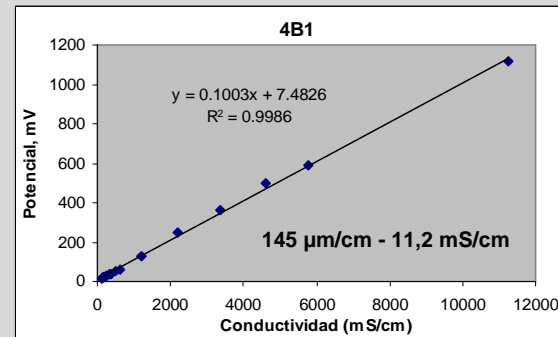
IDS



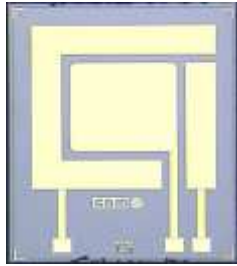
4 bars



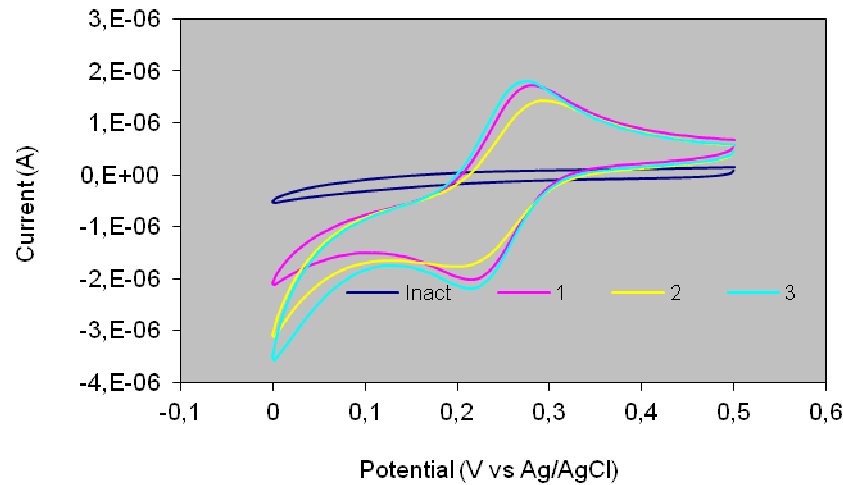
Au/Pt microelectrodes



Conductivity calibration curve



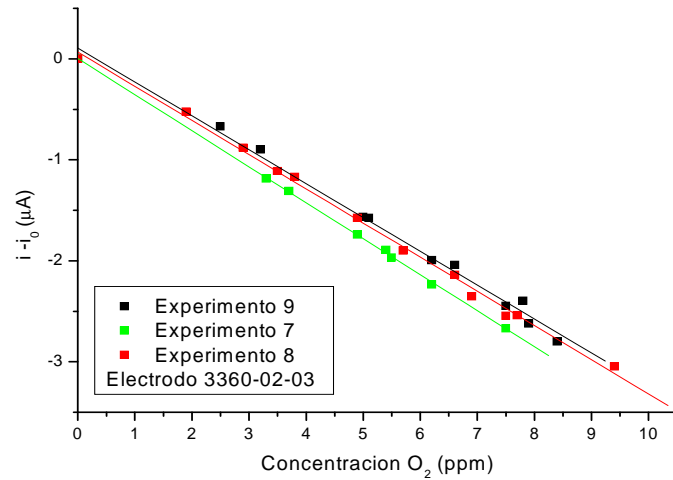
3-cell Au/Pt microelectrodes



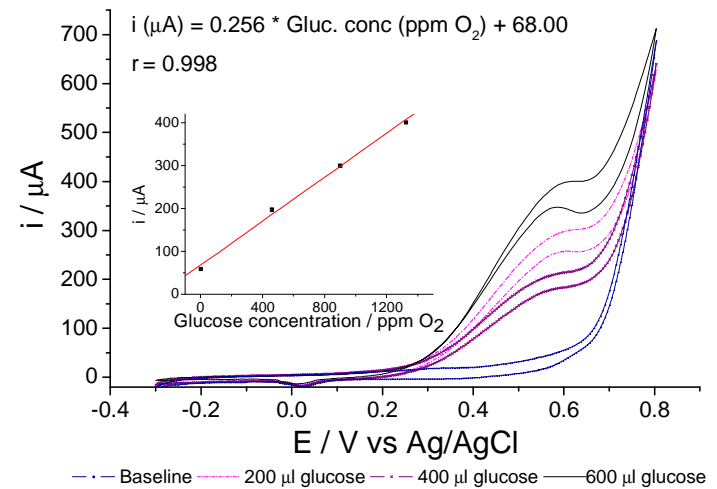
Oxidation- Reduction reactions:  $Fe^{2+}$

Eq. Randles  $I_p = K \times C$

## Disolved $O_2$ sensor

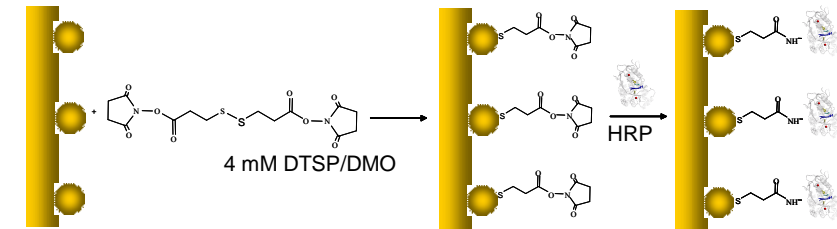


## Glucose sensor

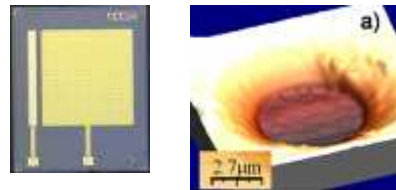


## UMEA modified with Au nanoparticles

### Peroxidase Biosensors



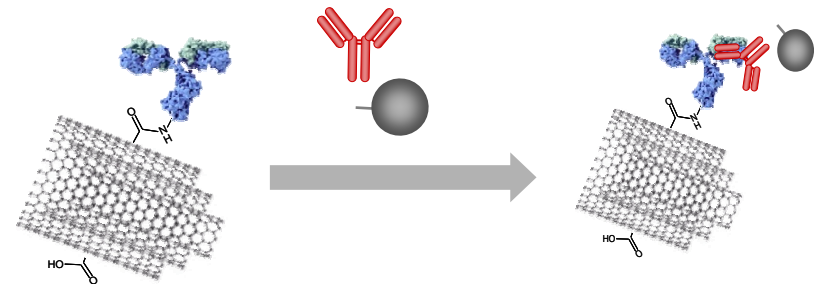
Gold UMEA



10 times higher sensitivity/ Au microelectrode

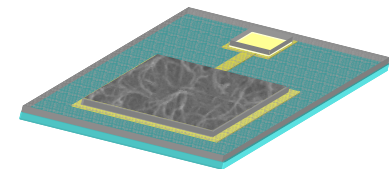
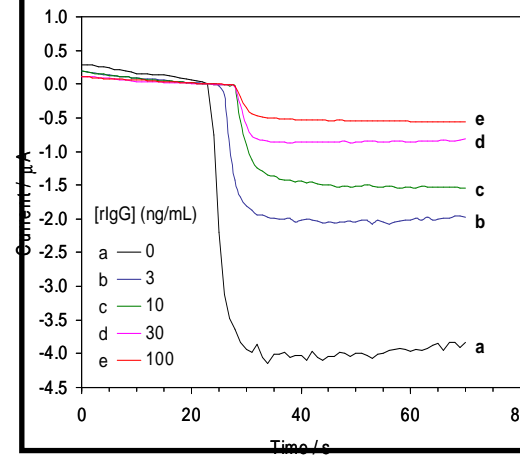
## Carbon nanotube-polystyrene composite electrodes

### Immunosensor approach for the detection of rabbit IgG



Covalent immobilization of rabbit IgG

Biological recognition event using anti-rabbit IgG peroxidase conjugate



Calibration:

- $I/\mu A = 0.44 \cdot \log[rIgG] - 2.47$
- $r = 0.99; n = 4$

Linear range:

- 3-100 ng/mL IgG rabbit

Limit of detection:

- 3 ng/mL

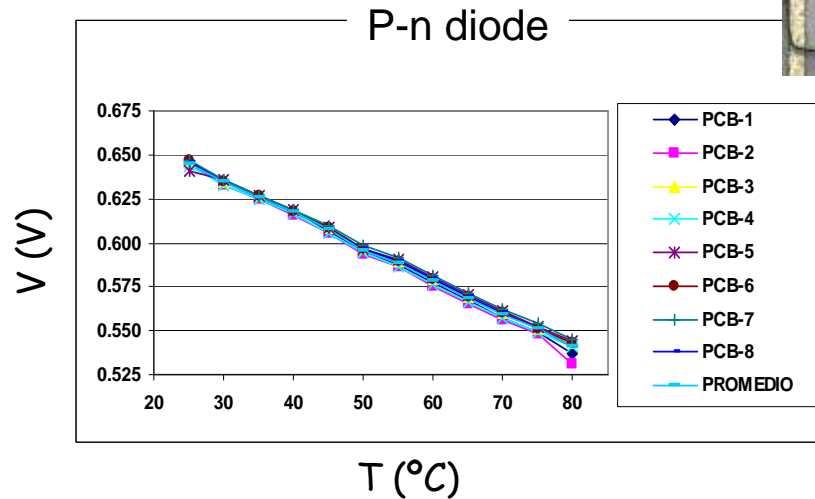
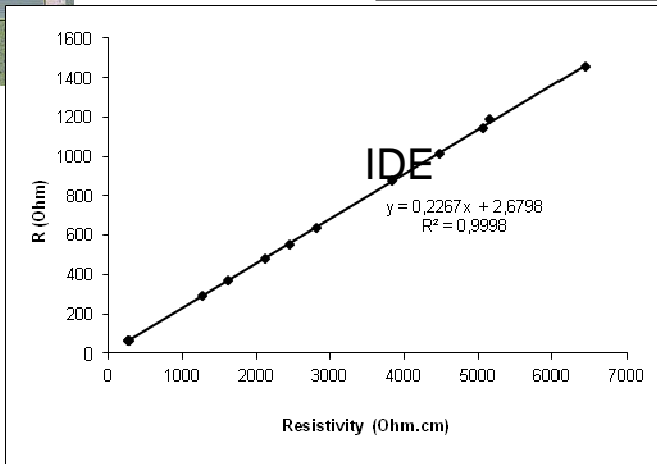
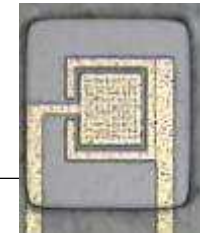
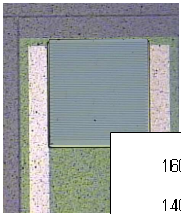
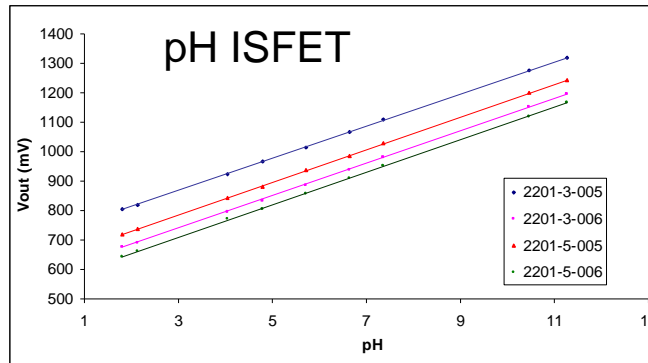
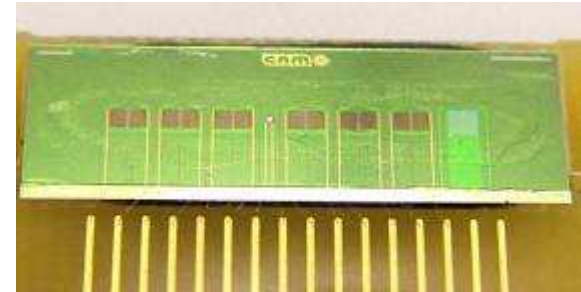
## Monolithic Integration on BESOI substrate

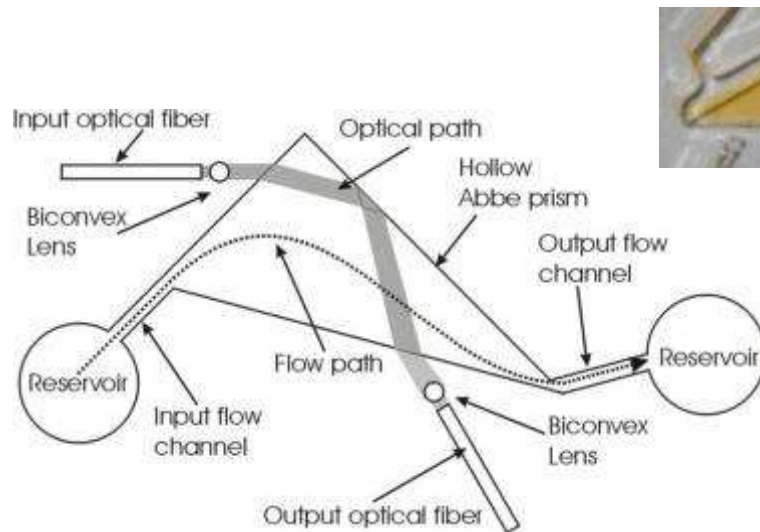
6 NMOS ISFET

1 IDEs

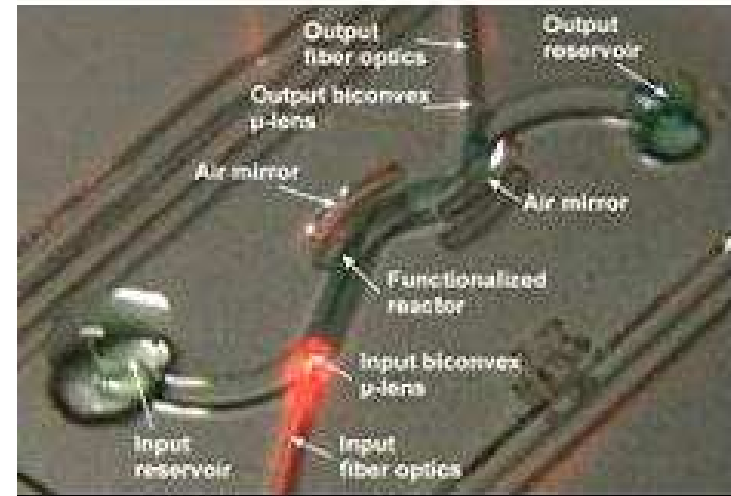
1 Temperature Diode

Electrically Isolated by Trench (SOI Substrate)

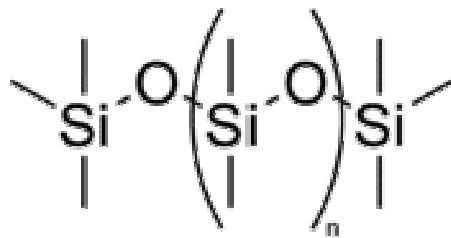




**Prism configuration**



**MIR configuration**



PDMS

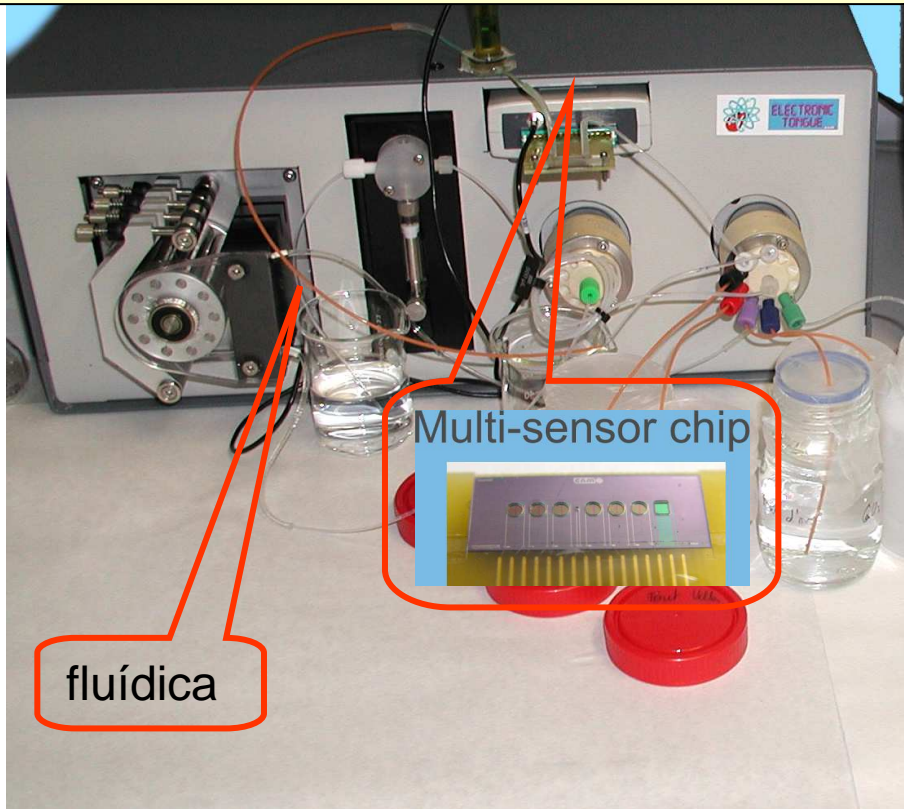
Polydimethylsiloxane

**Characteristics:**

- Method of fabrication is fast and easy.
- Monolithic integration ( $\mu$ TAS).
- Compatible with aqueous media.
- Biocompatible, chemically stable and non-toxic.
- Absorbance between 200 and 1000 nm



- Instrumento analítico para control de calidad de alimentos
- Formado por:
  - Sistema de muestreo
  - Conjunto de sensores químicos
  - Sistema de procesamiento de datos: reconocimiento de patrones o calibración multivariante (PCA, Redes Neuronales)

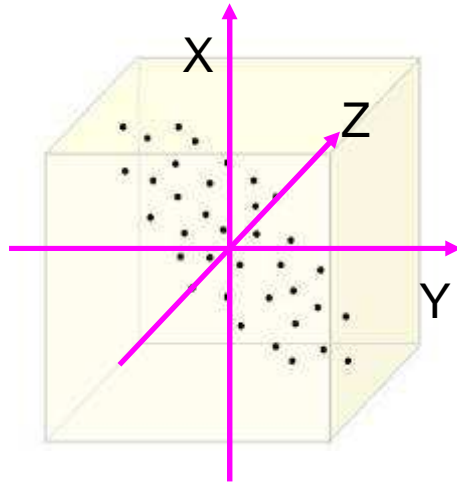


**Software para:**  
**control de bombas/válvulas**  
**Visualización señal**  
**Tratamiento señal**

- ✓ **Aplicaciones**  
 Alimentación, bebidas, cosmética, farmacéutica y medio ambiente
- ✓ **Control de calidad y fraude**
- ✓ **Selección de tipos de alimentos**
- ✓ **Determinación de D.O. en vinos**
- ✓ **Análisis cuantitativo**

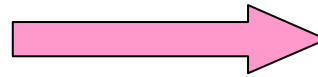
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Original Data



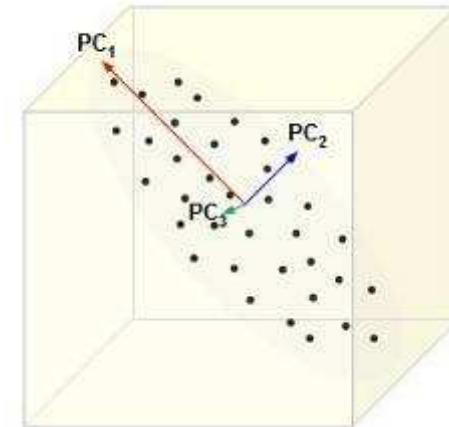
Method for pattern recognition

**PCA**



Change of the axes

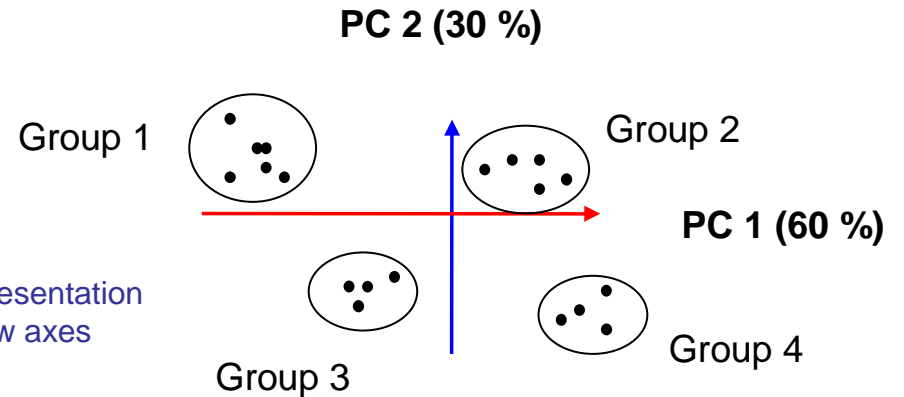
Directions of maximum variation, PCs



Reduction of variables

Representation in two dimensions

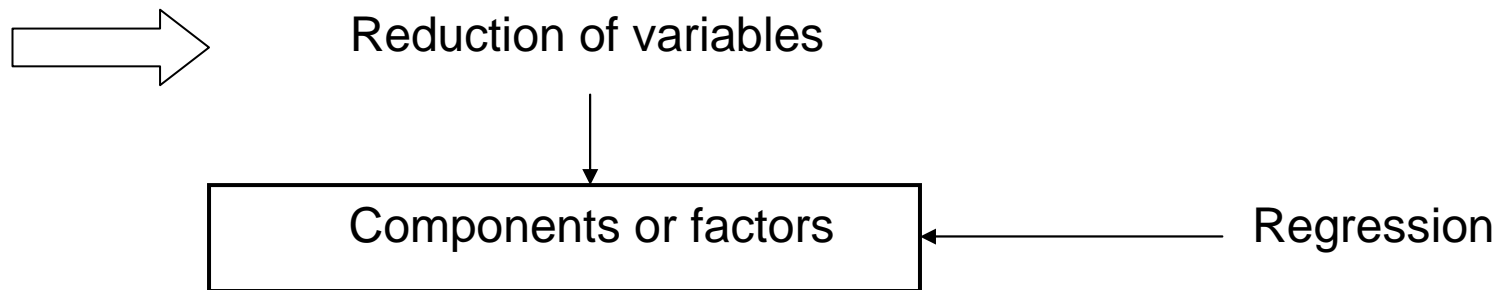
Graphic representation in the new axes



➔ Standard Method in Chemometrics.

➔ Method of multivariate calibration. Linear equation expressed generally like this:

$$y = b_0 + \sum_{k=1}^k b_k x_k$$

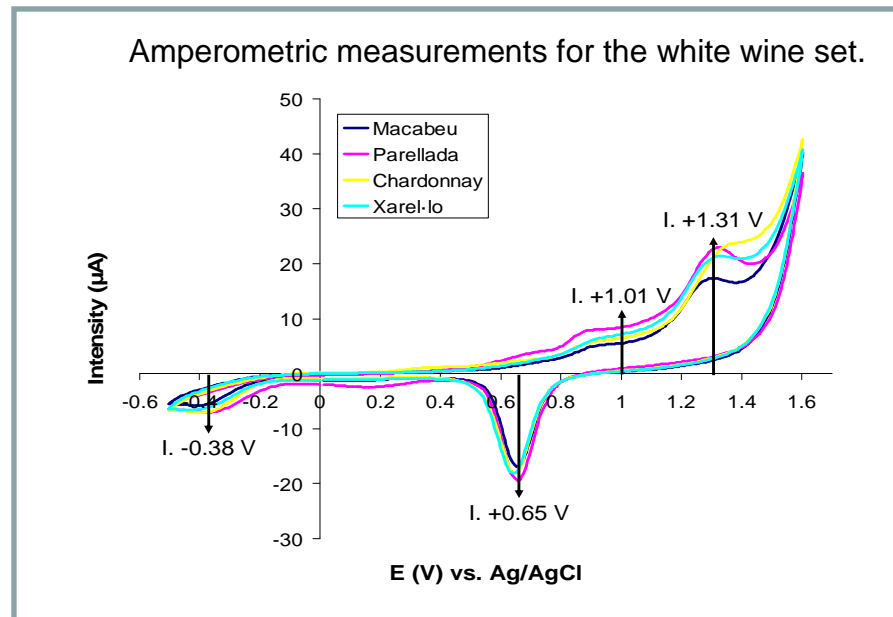
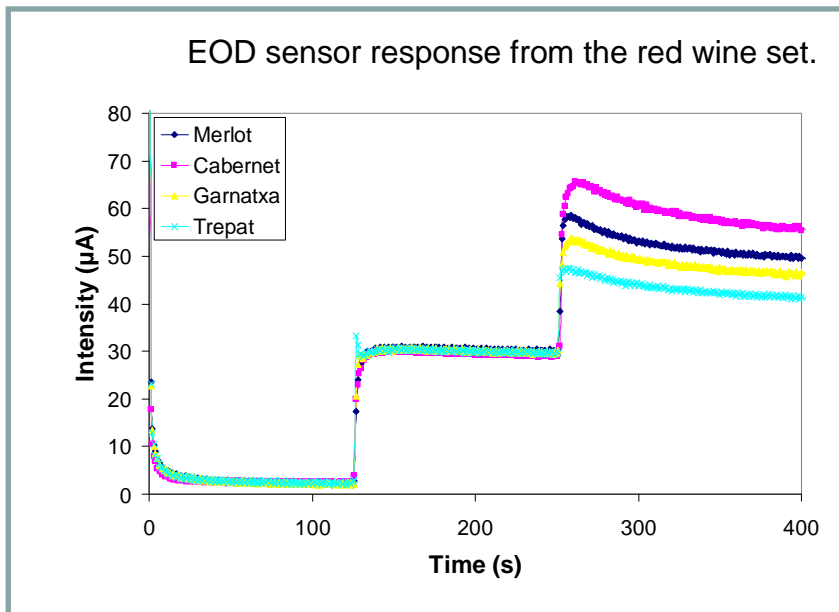
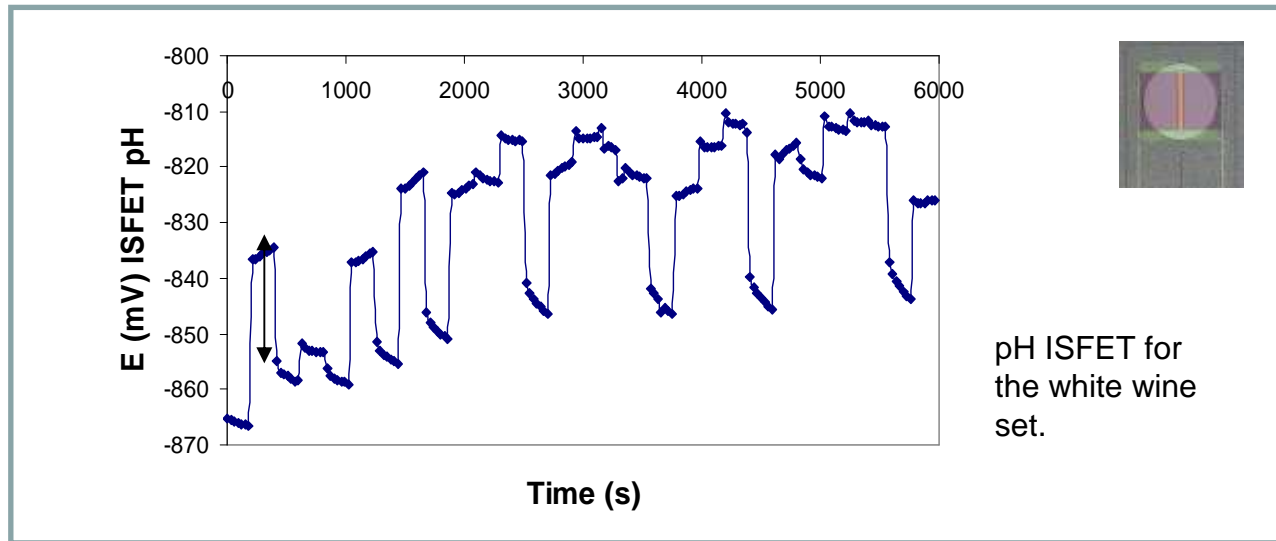


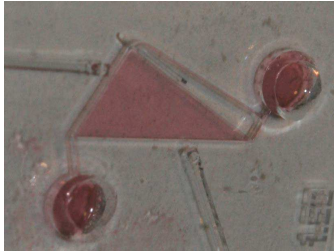
➔ Wine Samples {

- Calibration set:** 2/3 of the total number of samples
- Prediction set:** the rest 1/3 of samples. At least, one sample of each variety

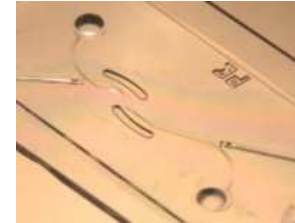
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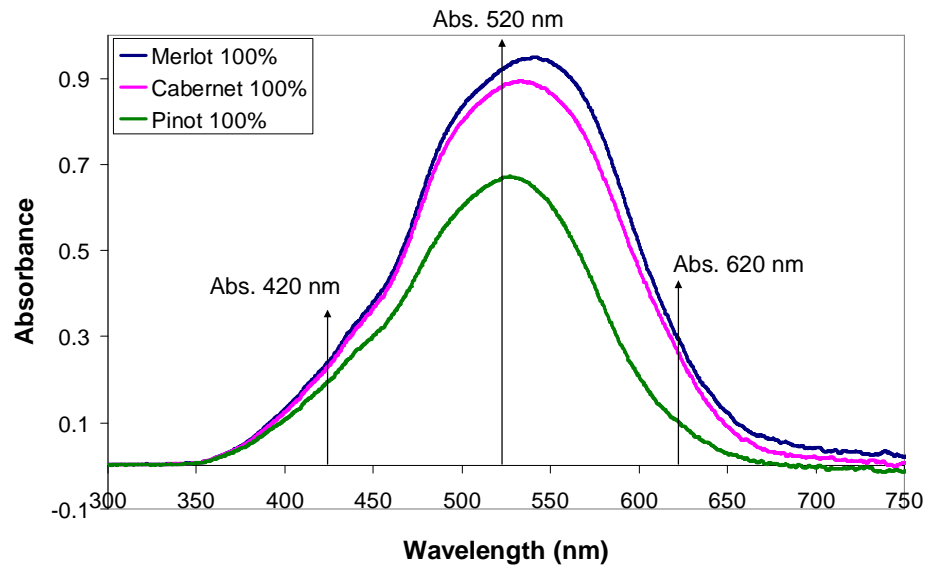




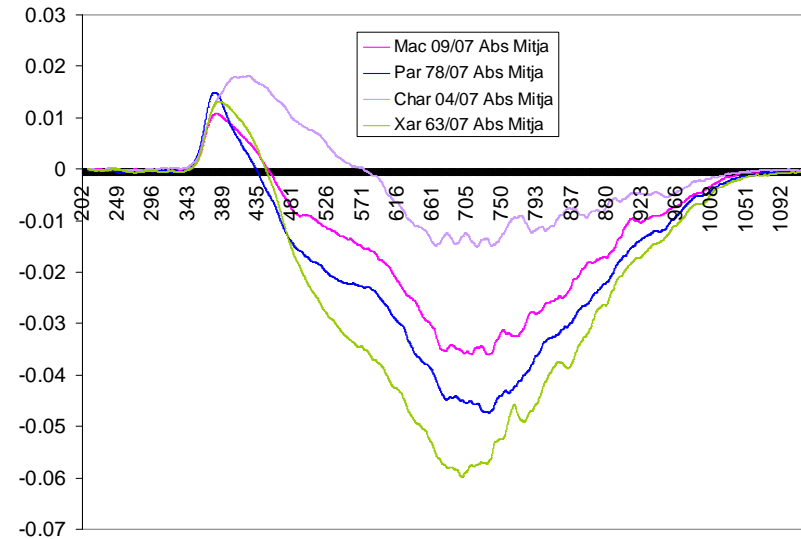
Absorbance spectra obtained with the optical sensor using DI water as reference



### Red wines



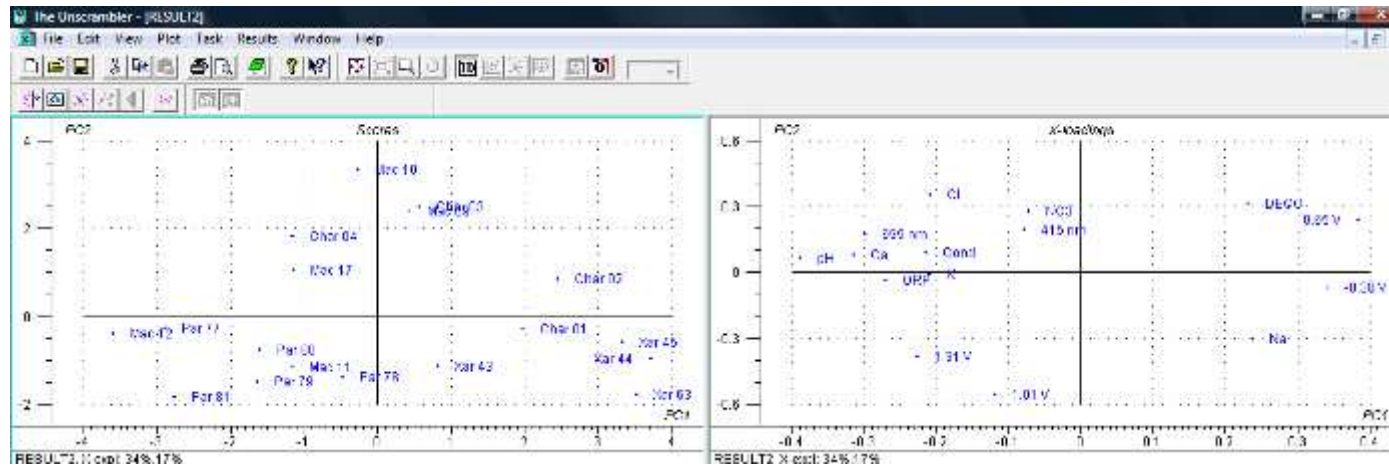
### White wines



## PCA analysis

	pH	Na	K	Ca	Cl	NO3	Cond	ORP	DECO	-0.38 V	1.01 V	1.31 V	0.65 V	415 nm	695 nm	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
Par 78	7	30.3700	-8.9800	13.8800	-2.1400	10.0500	7.3200	1.4400	226.8900	1.6641	-5.5664e-06	9.2957e-06	2.0761e-05	-1.8646e-05	2.8874e-03	-4.5269e-02
Par 79	8	29.9100	-4.7300	17.2500	1.6800	19.3700	9.6100	1.4700	215.2100	1.6525	-6.7413e-06	8.6914e-06	2.1118e-05	-1.9254e-05	2.8752e-03	-3.4705e-02
Par 80	9	34.3400	-8.5500	17.5500	-0.9200	6.1000	7.1800	1.5000	217.8900	1.6401	-6.7810e-06	8.3191e-06	2.0007e-05	-1.8295e-05	9.1073e-03	-3.4944e-02
Par 81	10	31.8900	-8.2200	14.9600	-1.5200	9.8000	7.4800	1.4100	221.5100	1.5988	-7.0251e-06	8.6060e-06	2.2946e-05	-1.9434e-05	1.3250e-02	-2.1667e-02
Char 01	11	24.1100	-7.4400	13.2700	-2.7400	20.0600	6.2500	1.3900	165.6100	1.6983	-6.2012e-06	7.8705e-06	2.0163e-05	-1.7053e-05	-1.8728e-03	-4.8208e-02
Char 02	12	22.7400	-6.7800	11.6000	-3.8200	20.3200	1.9800	1.4500	173.6100	1.6700	-6.0486e-06	5.9998e-06	1.8573e-05	-1.6751e-05	7.4086e-03	-4.2942e-02
Char 03	13	24.4200	-6.9100	8.7000	-0.4600	22.7800	10.5300	1.7100	202.9000	1.7442	-6.8817e-06	5.5725e-06	1.9794e-05	-1.7807e-05	1.9684e-02	-4.7415e-02
Char 04	14	28.2300	-12.5000	12.6700	-1.9800	23.0400	6.5600	1.4100	177.5700	1.7071	-6.9977e-06	6.6284e-06	2.2064e-05	-1.8027e-05	1.8202e-02	-1.2793e-02
Xar 43	15	23.9500	-7.1700	11.9000	-0.4600	8.9200	0.9200	1.1800	219.8300	1.6629	-6.3019e-06	7.3486e-06	2.1390e-05	-1.8015e-05	1.4846e-02	-4.7104e-02
Xar 44	16	23.9500	-7.5900	10.8400	-5.0300	-3.0500	3.9700	1.1400	170.7100	1.7088	-5.4504e-06	7.4453e-06	1.9650e-05	-1.7209e-05	5.7254e-03	-4.8429e-02
Xar 45	17	24.4200	-5.3000	9.0000	-4.4200	-2.9800	8.9500	1.1800	230.8500	1.7259	-5.2521e-06	7.4036e-06	1.9635e-05	-1.7136e-05	1.2306e-02	-5.1342e-02
Cupatge 1	18	34.1800	-10.1900	12.2100	0.0000	13.5200	4.8900	1.3300	228.4900	1.6818	-6.2653e-06	8.1879e-06	2.0975e-05	-1.8225e-05	3.4651e-03	-3.7414e-02
Cupatge 2	19	29.4500	-11.6700	15.1100	-2.6300	20.5000	-0.9200	1.3800	197.3700	1.7125	-5.8380e-06	6.8481e-06	1.8295e-05	-1.6385e-05	-1.4966e-03	-5.1209e-02
Cupatge 3	20	31.8900	-10.4500	15.7200	0.3100	18.7500	14.9600	1.4500	236.9400	1.6412	-7.5623e-06	6.0546e-06	2.3459e-05	-2.0163e-05	7.1376e-03	-3.1644e-02
Cupatge 4	21	25.3300	-6.4700	9.3100	-1.3700	11.0300	-5.6500	1.2100	207.6800	1.6945	-5.8885e-06	6.7413e-06	1.9055e-05	-1.7532e-05	1.2880e-02	-4.9674e-02
Cupatge 5	22	30.5200	-6.9800	11.9000	-1.8300	13.4100	7.9300	1.3200	210.9000	1.6959	-6.1157e-06	6.7555e-06	2.0508e-05	-1.8152e-05	1.1742e-02	-3.8200e-02
Cupatge 6	23	30.6700	-9.8400	16.3200	-1.5200	19.0900	13.7300	1.4100	248.2500	1.7147	-6.7963e-06	6.8420e-06	1.9296e-05	-1.8567e-05	9.2301e-03	-5.9665e-02
Cupatge 7	24	32.2000	-2.2800	13.4300	-2.7400	16.0900	13.5800	1.2800	228.4600	1.7841	-6.1096e-06	7.1808e-06	2.0825e-05	-1.8555e-05	9.5100e-03	-5.1037e-02
Grasevina	25	5.1900	-4.8900	1.6300	-17.7000	25.5400	-7.7800	1.9100	4.7000	1.6728	-8.8492e-06	1.0101e-05	2.3621e-05	-1.8158e-05	1.2607e-02	-5.3398e-02
Zelenac	26	3.9700	-2.7500	4.5800	-3.6600	27.8700	-3.5100	1.5600	134.6600	2.0923	-5.6610e-06	7.9956e-06	2.2562e-05	-1.8048e-05	2.5051e-02	-4.4232e-02
Xar 63	27	17.8500	-3.0500	12.8100	-1.3700	9.2100	4.1200	1.3600	176.6400	1.7358	-4.8920e-06	8.7677e-06	2.1173e-05	-1.7404e-05	7.5472e-03	-5.9336e-02

- Matrix of Data
- Variables vs samples
- We perform a PCA: all samples are used for the **training**
- Weight of samples: all are normalized to 1/sdv

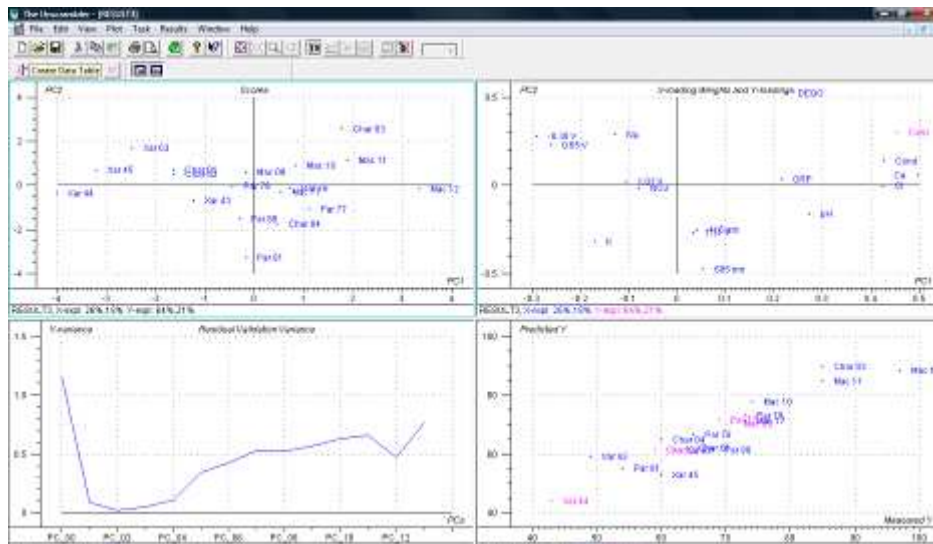


- Scores distribution of all the samples all through the 2 C
- Loadings distribution of the variables (weight of each variable)

## PLS analysis

	DRG0	-3.26 V	1.91 V	1.31 V	0.85 V	475 nm	495 nm	DR%	DR	AcTemp	Slope	AcCoef	Intercept	Temperature	Quadrant	Current	Magneta	Calc
Mac09	1.8201	-4.8518e-04	5.0719e-04	1.7271e-03	-1.8258e-03	3.2207e-03	-2.1701e-03	0.0090	0.0000	0.0000	0.1200	0.0000e+00	0.7770	0.0000	0.0000	0.0000	0.0000	0.0000
Mac13	1.7271	-4.8306e-04	5.1343e-04	1.7008e-03	-1.7005e-03	1.8258e-03	-1.8007e-03	0.0090	0.0000	0.0000	0.1900	0.0000e+00	0.6890	0.0000	0.0000	0.0000	0.0000	0.0000
Mac11	1.8908	-4.8056e-04	5.2889e-04	2.2704e-03	-1.8161e-03	1.1218e-03	-1.8104e-03	0.0090	0.0000	0.0000	0.2800	7.4000e-02	0.7330	0.0000	0.0000	0.0000	0.0000	0.0000
Mac12	1.8976	-7.3911e-04	0.0847e-03	2.3738e-03	-1.9219e-03	1.1807e-03	-2.3928e-03	0.0090	0.0000	0.0000	0.4900	0.0000e+00	0.2890	0.0000	0.0000	0.0000	0.0000	0.0000
Mac17	1.7128	-5.4971e-04	7.4529e-04	2.2078e-03	-1.8088e-03	1.8508e-03	-2.3001e-03	0.0090	0.0000	0.0000	0.1900	0.0000e+00	0.2890	0.0000	0.0000	0.0000	0.0000	0.0000
Par17	1.9078	-4.8306e-04	5.2889e-04	2.1249e-03	-1.8087e-03	1.1986e-03	-1.8073e-03	0.0090	0.0000	0.0000	0.5000	0.0000e+00	0.7770	0.0000	0.0000	0.0000	0.0000	0.0000
Par18	1.9041	-5.5004e-04	5.2957e-04	2.0701e-03	-1.8548e-03	2.8074e-03	-1.5208e-03	0.0090	0.0000	0.0000	1.2200	0.0000e+00	0.6890	0.0000	0.0000	0.0000	0.0000	0.0000
Par19	1.8525	-4.7013e-04	5.0919e-04	2.1119e-03	-1.8254e-03	2.8752e-03	-1.4705e-03	0.0090	0.0000	0.0000	0.2800	0.0000e+00	0.7290	0.0000	0.0000	0.0000	0.0000	0.0000
Par20	1.8801	-4.7919e-04	5.2191e-04	2.2007e-03	-1.8205e-03	1.8073e-03	-1.8884e-03	0.0090	0.0000	0.0000	0.3500	0.0000e+00	0.2790	0.0000	0.0000	0.0000	0.0000	0.0000
Par21	1.9068	-7.3251e-04	0.0809e-03	2.2948e-03	-1.8424e-03	1.2025e-03	-2.1857e-03	0.0090	0.0000	0.0000	0.4000	0.0000e+00	0.6890	0.0000	0.0000	0.0000	0.0000	0.0000
Char01	1.8963	-6.3012e-04	7.6225e-04	2.1010e-03	-1.7023e-03	1.4709e-03	-1.8208e-03	0.0090	0.0000	0.0000	0.1400	7.9000e-02	0.1420	0.0000	0.0000	0.0000	0.0000	0.0000
Char02	1.8708	-4.8869e-04	5.0999e-04	1.8373e-03	-1.8701e-03	7.4028e-03	-1.4924e-03	1.9890	1.1300	0.0000	0.4000	0.1300	7.0000e-02	0.2720	0.0000	7.9000	0.0000	0.0000
Char03	1.7842	-4.8917e-04	5.0725e-04	1.8794e-03	-1.7907e-03	1.8984e-03	-1.7815e-03	1.2790	1.8900	0.0000	0.1900	0.1200	0.6590	0.0000	7.0000	0.0000	0.0000	0.0000
Char04	1.7071	-4.8917e-04	5.0725e-04	2.2064e-03	-1.8027e-03	1.8203e-03	-1.2709e-03	1.0390	1.1200	7.9000	0.0000	0.1300	0.0000e+00	0.4580	0.1300	0.0000	0.0000	0.0000
Par45	1.9028	-4.3016e-04	7.3489e-04	2.1208e-03	-1.8015e-03	1.8944e-03	-1.7104e-03	0.1800	1.2800	0.0000	0.2000	0.5800	0.1110	2.5170	0.1300	0.0000	0.0000	0.0000
Par44	1.7068	-4.4504e-04	7.4482e-04	1.9084e-03	-1.7208e-03	1.7254e-03	-1.8629e-03	0.0090	1.3400	5.7000	0.0000	0.0300	0.0000e+00	0.0890	0.0000	0.0000	0.0000	0.0000
Par45	1.7258	-4.5201e-04	7.4839e-04	1.9025e-03	-1.7158e-03	1.2306e-03	-1.5142e-03	0.0090	1.1900	0.0000	0.4000	0.0900	0.1110	2.6170	0.0000	7.9000	0.0000	0.0000
Par03	1.7358	-4.8928e-04	5.1877e-04	2.1173e-03	-1.7604e-03	1.5472e-03	-1.8328e-03	0.1820	1.1200	0.0000	0.2000	0.3300	0.0780	2.1790	0.0000	7.9000	0.0000	0.0000

- PLS 1. One model for each variable
- Comparison of our data with standard method

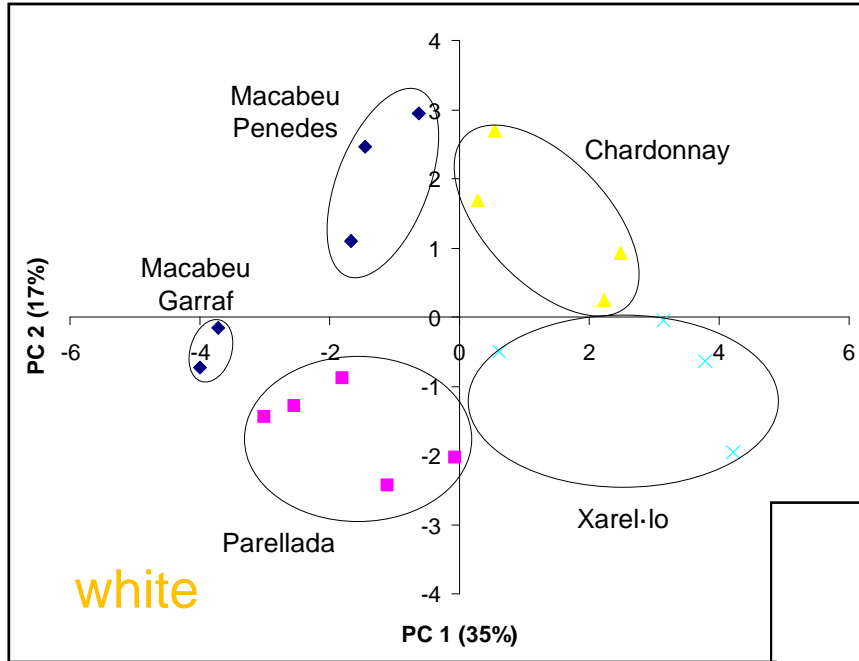


	Calca	Sampl...	PC 04	Calca	Sampl...	PC 04
Mac 09	72.000	=	72.000	69.954	=	
Mac 10	74.000	=	74.000		=	
Mac 11	88.000	=	88.977	88.000	=	
Mac 12	97.000	=	98.888	97.000	=	
Mac 17	78.000	=	78.067	78.000	=	
Par 77	80.000	=	80.000	73.736	=	
Par 78	80.000	=	72.240	80.000	=	
Par 79	70.000	=	71.470	70.000	=	
Par 80	80.000	=	80.112	80.000	=	
Par 81	84.000	=	82.353	84.000	=	
Char 01	84.000	=	81.289	84.000	=	
Char 02	88.000	=	88.000	80.951	=	
Char 08	88.000	=	86.982	88.000	=	
Char 09	80.000	=	88.926	80.000	=	
Par 48	48.000	=	48.488	48.000	=	
Par 44	48.000	=	48.000	42.185	=	
Par 45	80.000	=	87.927	80.000	=	

training set  
test set

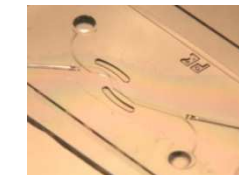
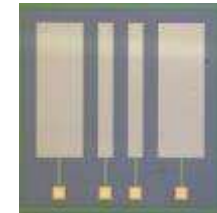
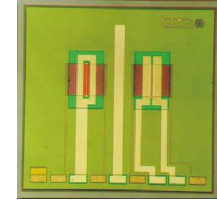
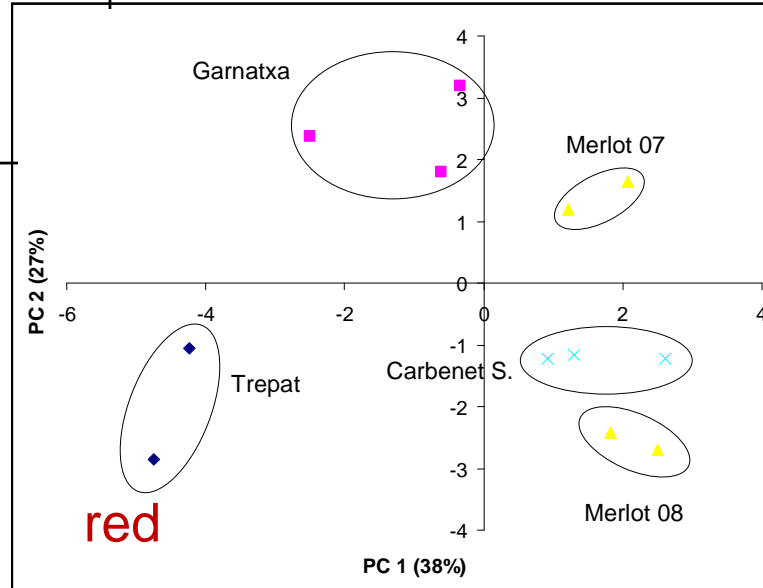
- One model for Ca detection
- Error ~ 0 for 2 PC
- Regression treatment

## PCA for monovarietal wines



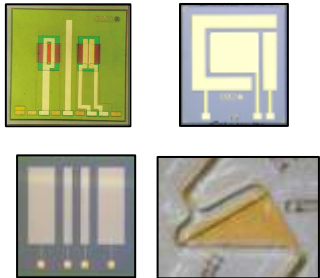
Classification according to:

- Grape variety
- vintage year
- geographical origin

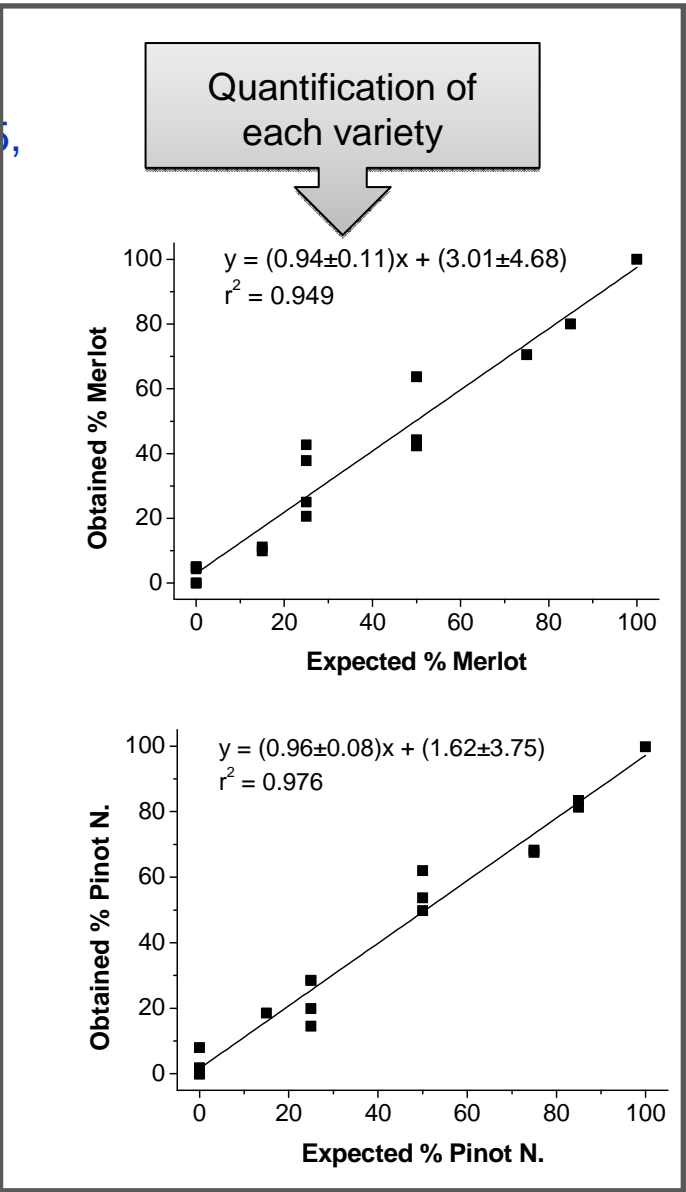
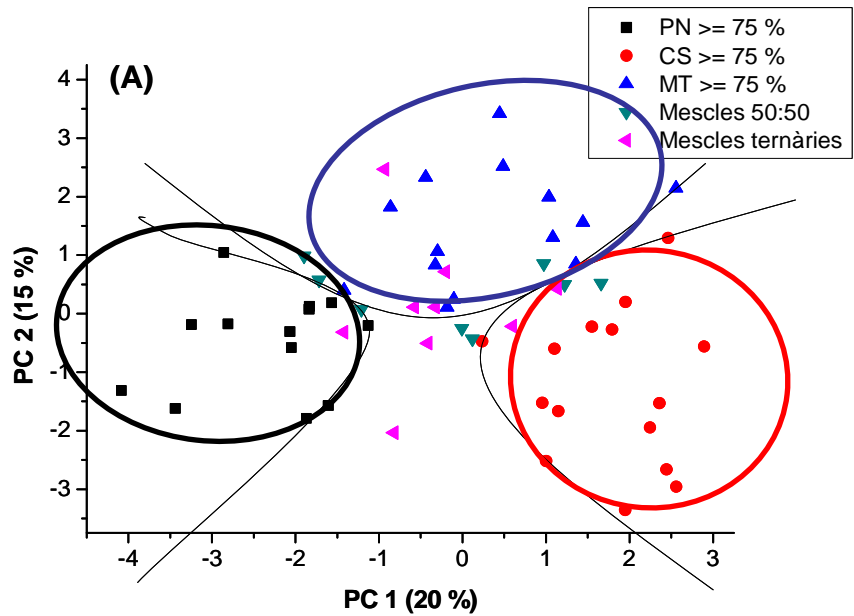


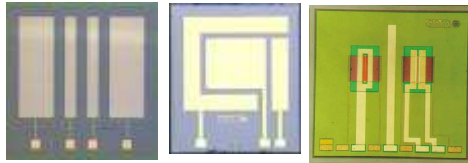


60 mixtures of Cabernet Sauvignon, Pinot Noir and Merlot 0, 25, 50, 75, 85, 100%



Wine classification according to the percentage of grape variety





## PLS for Chemical Wine Parameters

Relative errors comparing with standard methods of analysis:

### White wine

Sample	VAD	Total acidity	pH	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Glycerol
Macabeu 09/07	2.82	-3.7	-0.79	-0.7	-4.9	6.0
Parellada 77/07	0.07	9.5	-3.55	5.1	-0.1	2.0
Chardonnay 02/07	3.32	-2.7	1.16	5.7	-2.2	-4.3
Xarel-lo 44/07	-0.62	1.0	-4.76	4.0	-2.8	12.3

Error < 5 %

Error < 10 %

### Red wine

Sample	VAD	Total acidity	pH	Ca <sup>2+</sup>	K <sup>+</sup>	Glycerol
118/07 Trepas	4.45	0.6	-2.20	-5.1	-4.0	8.8
95/07 Garnatxa	-4.05	-7.7	1.34	-6.1	6.7	2.3
49/07 Merlot	-2.31	-2.6	-0.91	3.9	-0.7	3.8
45/08 Merlot	-6.52	8.0	2.03	-2.4	-3.2	-5.6
105/07 Cabernet	0.99	-9.0	-4.01	8.7	-8.5	-2.7

VAD: volatil alcoholic degree

## PLS for Optical Wine Parameters

Relative errors comparing with standard methods of analysis:



### White wine

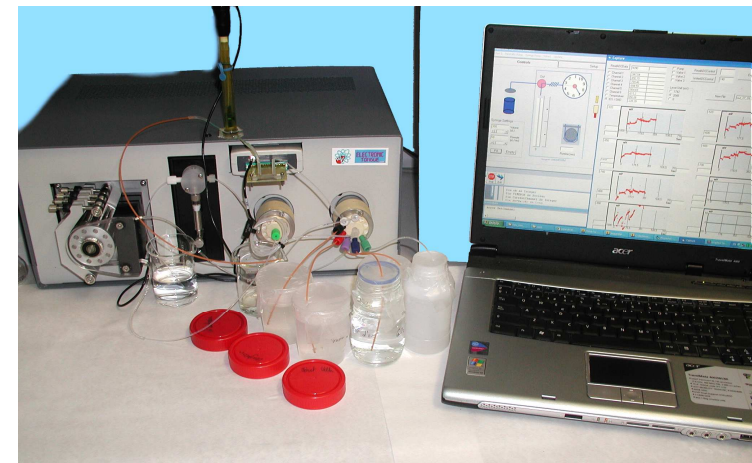
Sample	Intensity of color	Tonality
Macabeu 09/07	4.67	1.09
Parellada 77/07	4.56	3.66
Chardonnay 02/07	0.87	2.48
Xarel-lo 44/07	7.29	-4.23

Error < 10 %

### Red wine

Sample	Intensity of color	Tonality	Ciel ab a*
118/07 Trepat	-89.74	2.20	-5.16
95/07 Garnatxa	-44.57	-3.14	7.90
49/07 Merlot	-32.31	4.30	8.98
45/08 Merlot	17.43	3.17	-7.16
105/07 Cabernet	-10.22	-5.65	3.41

- A multisensor system with different kind of sensors: potentiometric, voltamperometric, conductimetric, optical → **hybrid electronic tongue**
- The system is capable of discriminating wines samples not only according to the **grape variety**, but also to the origin and even the vintage.
- The system is able to quantify several parameters. The relative errors obtained are **below 10%**.
- The ET could be applied in the cellars as a system for **rapid detection, close to the production** and as an **alarm system**.



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## Sistemas multisensors aplicats al control de qualitat dels vins

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