European Network on New Sensing Technologies for Air Pollution Control and Environmental Sustainability - *EuNetAir* COST Action TD1105

> WGs and MC Meeting at ISTANBUL, 3-5 December 2014 Action Start date: 01/07/2012 - Action End date: 30/06/2016

Year 3: 1 July 2014 - 30 June 2015 (Ongoing Action)

### Outline of (ULg) Chemical Sensors Applications for IAQ Evaluation



### **A better IAQ?**

2 000 000 Healthy Life Years (3%) are lost every year due to indoor air Exposure in EU"

### Two approaches

#### **1. Source reduction**

Upstream: i.e. building materials with low emissions

Downstream: i.e. green ambulance

#### 2. Ventilation improvement

Energetic and CO<sub>2</sub>, RH considerations: recommended ventilation rate 4 L/s per person

15m<sup>3</sup>/h.pers (0.6 h<sup>-1</sup>) \* COEFFICIENT RELATED TO IAQ

(source: HealthVent project)

→ CE mark

### Evaluation with sensors ?

Devices already commercialised

electrochemical cells, NDIR, PID, "MOS" for CO<sub>2</sub>, TCOV, HCOH, O<sub>3</sub>, NH<sub>3</sub>, H<sub>2</sub>S, NO, NO<sub>2</sub>, SO<sub>2</sub>, PM







### **Outline of sensors applications**

### Continuous and real time monitoring

□ control systems:

ventilation systems + dirty, moulds indicators air treatment equipments intelligent materials (active paints)

□ air quality indicator for

Regulations: i.e. French decree on thresholds levels for HCOH, Benzene in public buildings (2013) Building Environmental efficiency: BREEAM, LEED, HQE

# HQE: NO<sub>2</sub> CO Benzene TVOC PM 2.5 PM 10 Radon Formaldehyde Punctual and real time monitoring (handheld devices)

- ❑ source identification
- □ fast diagnosis

### "Lab" analysis

❑ assessment of emissions from building materials →labelling

EURO <u>Regulation (EU) No 305/2011</u> laying down harmonised conditions for the marketing of construction products (89/106/EEC); standards EN 16516 and 16402 (2013)

Most frequent problems in buildings

SBS respiratory infection asthma skin and eye irritation headache cough nausea



Usual Evaluation:

quantity of viable spores (air, surface and building material, settled dust) results obtained after several days

New methods: detection of fungal components, mycotoxins

microbial volatile organic compounds (MVOCs)

Advantages:

these compounds can penetrate barriers not penetrable by spores

 $\rightarrow$  facilitating the detection of hidden moulds



Chemical sensors for source identification with respect to a MVOC pattern???

□ Selectivity?  $\rightarrow$  sensor array (« e-nose » principle) □ LOD?



- VOC for moulds (MVOC): typical of moulds contamination?
- MVOC pattern specific for specific moulds?
- > No confusion with VOC indoor air background?
- Concentration level is it detectable?

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> MVOC: typical of moulds + pattern specific of moulds? (Source: HEMICPD Belgian project)

study of VOC produced under defined lab conditions learning whether field measurements can identify MVOCs

Development of Cladosporium microorganisms over time



agar culture medium

- Cladosporium cladosporoides
- Aspergillus versicolor
- Penicillium purpurogenum
- Stachybotrys chartarum

Tenax TA cartridges on the output of the  $\mu\text{-}CTE$  under the following conditions:

- o Temperature: 23 °C ± 2 °C
- o Relative humidity:  $0\% \pm 5\%$
- o Sampling flow: 100 mL/min
- o Sampling duration: 30 minutes
- o Sampled volume: 3,0 L

TD-GC-MS (series iso 16000) Reference (substraction)

Lor, M., Vause, K., Dinne, K., Goelen, E., Maes, F., Romain, A.-C., Nicolas, J., Degrave, C., Horizontal evaluation method for the implementation of the Construction Products Directive – emissions from construction products into indoor air, Healthy Buildings Syracuse, NY USA, 2009.

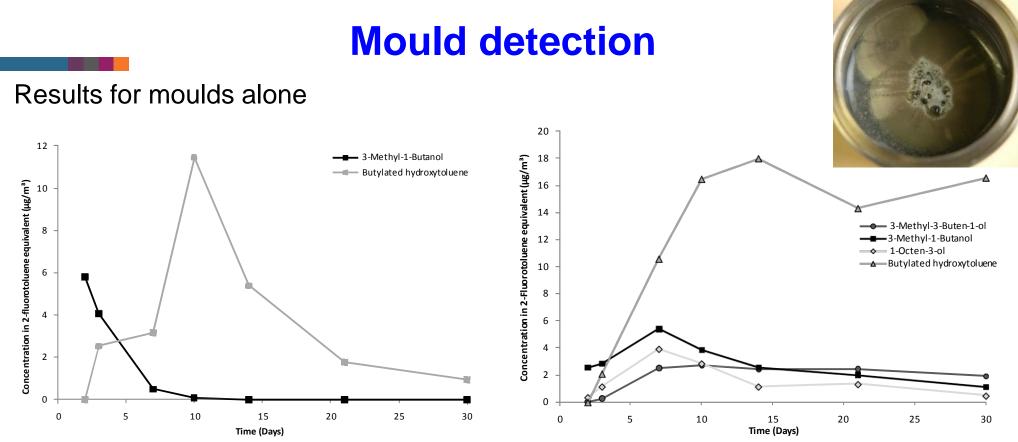


Figure 11 : Evolution of the principal MVOCs emitted by a strain of Cladosporium (strain 01)

Figure 18 : Evolution of the principal MVOCs emitted by a strain of Penicillium

- Complex emissions (several compounds)
- Two MVOCs always present and helped to highlight fungial activity: 3-methyl-1-butanol and butylated hydroxytoluene chemical markers

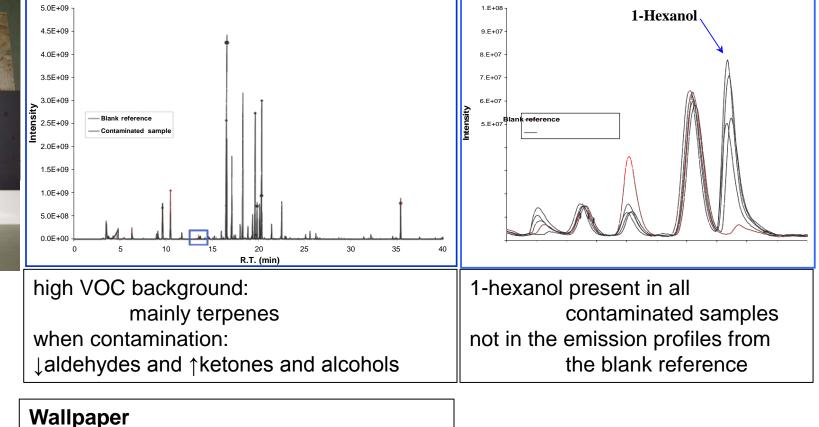


Source: HEMICPD Belgian project

Results for moulds on building materials

i.e. OSB (inoculation of Penicillium on 9 OSB samples)





low VOC background when contamination: ↓aldehydes and ↑alcohols

VOC for moulds (MVOC) typical of moulds contamination?
 MVOC pattern specific for specific moulds?
 No confusion with VOC indoor air background?
 Concentration level detectable?

Source: HEMICPD Belgian project

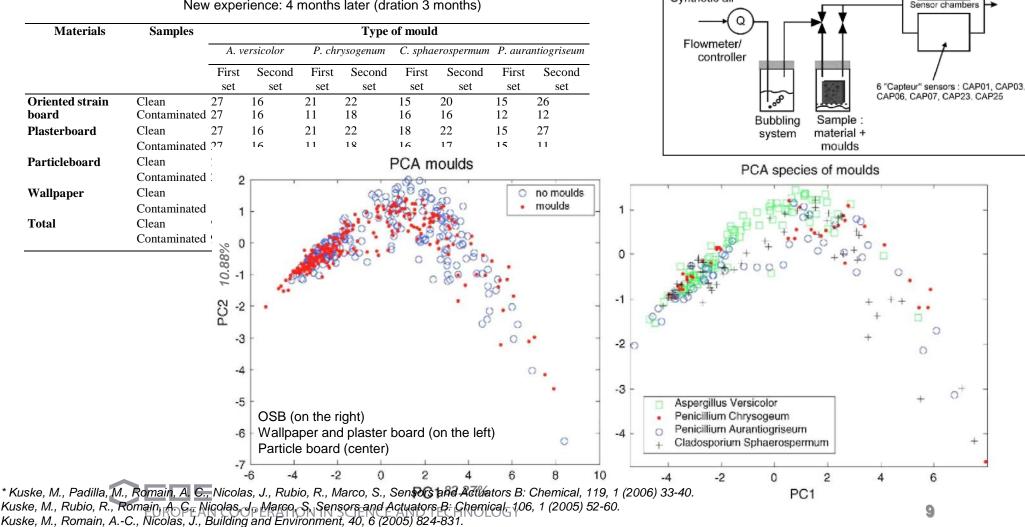
6 "Figaro" sensors : TGS2620, TGS2180, TGS825, TGS822,

TGS2600, TGS2602

Synthetic air

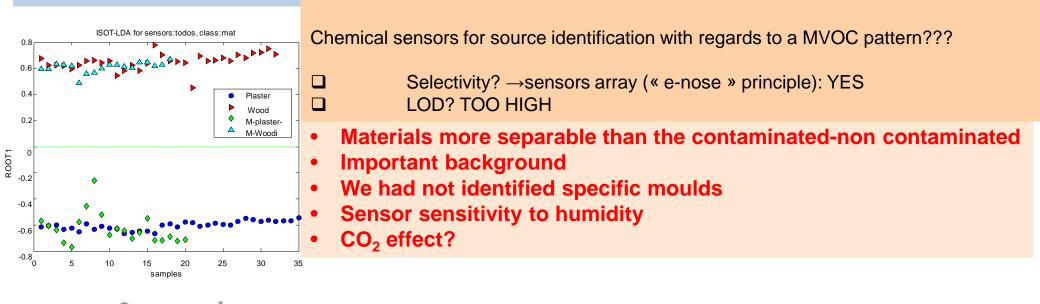
#### Sensor tests\*

Duration: 4 months Sampling: randomly First set: 1 month for data processing algorithm development Second set: last month (4) for classifier testing (posterior classification New experience: 4 months later (dration 3 months)



Kuske, M., Rubio, R., Nicolas, J., Marco, S., Romain, A. C., Prooceedings of ISOEN'03, Riga, Latvia, June 25-28, 2003, 2003

 ✓ VOC for moulds (MVOC) typical of moulds contamination? YES
 ✓ MVOC pattern specific for specific moulds? YES AND MARKERS COMPOUNDS
 No confusion with VOC indoor air background? INTERFERENCES
 MVOC PATTERNS RELATED TO SUBSTRATE (MATERIALS), ENVIRONMENTAL CONDITIONS, SPECIES
 Concentration level : is it detectable?
 < 10µg/m³ per MVOC...</li>



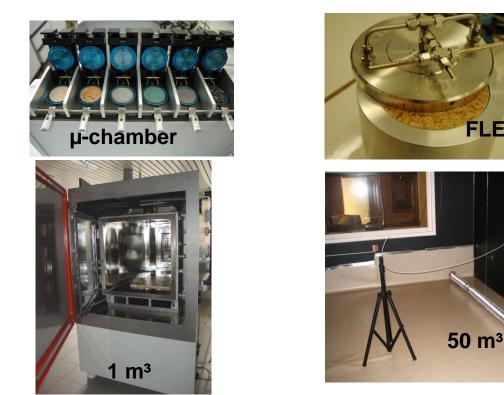
### **Building material emissions**

#### HEMICPD project (2007-2010)

Horizontal evaluation method for the implementation of the Construction Products Directive"-Emissions to indoor air concerned the marking of building materials



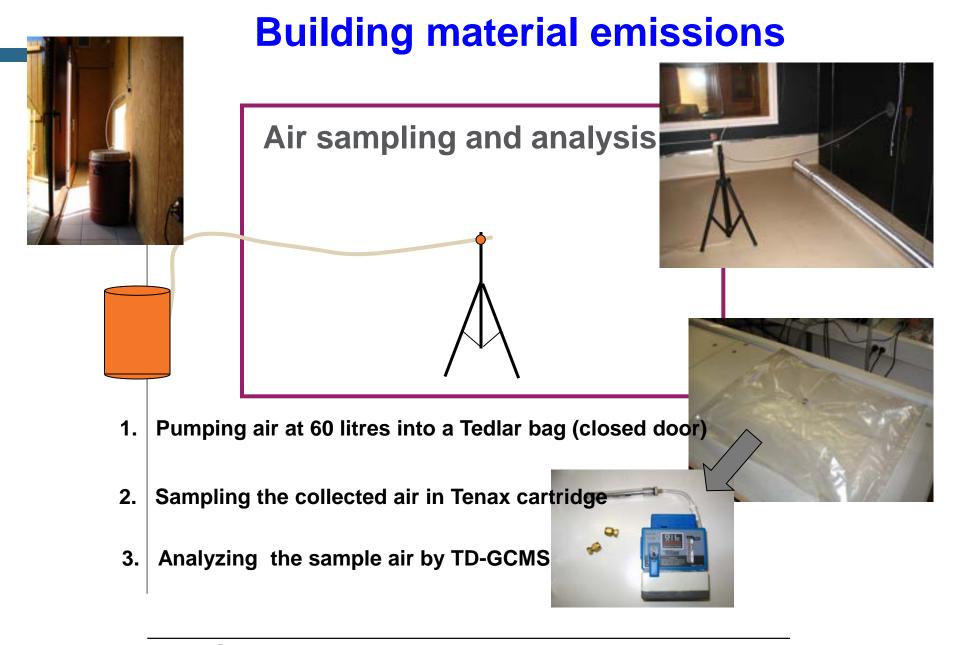
- VOC TD-GC-MS in a big chamber
- Odour-Emission relationships
- Test of the sensors array principle to monitor the compounds emanating from different building materials during 28 days, in emission test chambers



FLEC

Romain, A.-C., Degrave, C., Nicolas, J., Lor, M., Vause, K., Dinne, K., Maes, F., Goelen, E., Olfactory, chemical and e-nose measurements to characterize odors emission of construct materials for the implementation of the European construction products directive (CPD) on a Belgian level, OLFACTION AND ELECTRONIC NOSE: Proceedings of the 13th International Symposium on Olfaction and Electronic Nose, Brescia (Italy), 2009

Lor, M., Vause, K., Goelen, E., Maes, F., Romain, A.-C., Nicolas, J., Implementation of health aspects (ER N°3) in the Construction Products Directive (CPD) regarding emissions to indoor air, 11th International Conference on IAQ and Climate Denmark 17th-22nd August 2008





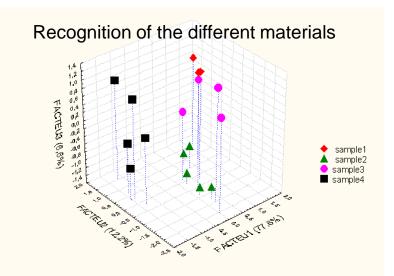
### **Building material emissions**

#### 6 Floor coverings were tested:

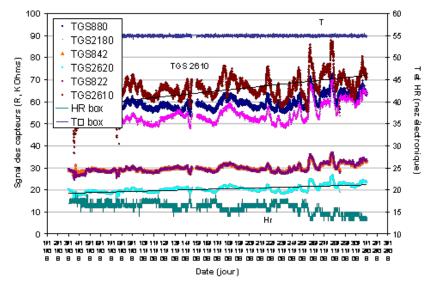
- 2 PVC floor coverings
- 2 linoleum floors
- 1 carpet
- 1 versatile rubber flooring







#### Continuous material emissions monitoring during 28 days



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3 industrial projects (2010-2012)

wallpapers with active reagent

to either oxidise (Titane oxide+uv) or capture VOC (molecular chelating agents)

#### Detection of odour-non odour with the chemical sensors (MOS)?

- Test of sensor array in real conditions:
- chip shop (100-800  $uo_E/m^3$  with a max 3000 above the fryer)
- company toilet (100-600  $uo_{E}/m^{3}$ )
- garbage room (200 and 500  $\,$  uo\_{\rm E}/m^3)

- + GC-MS analyses
- + olfactometry measurements



#### Results

- Identification of odour and non odour ambiance
- × Not always able to correlate "odour"-"compounds concentration"
- Not able to discriminate the different odours
- Odour concentration has to be > 100 uo<sub>E</sub>/m<sup>3</sup>

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#### Efficiency of the product pulverised on the walls?

Test of the sensor array in real conditions:

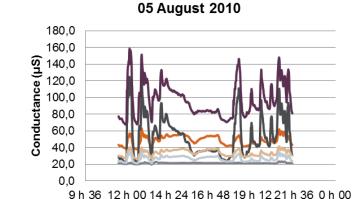
- French fries shop (100-800 uo<sub>E</sub>/m<sup>3</sup>)
- Garbage room (200  $uo_{E}/m^{3}$  and 500  $uo_{E}/m^{3})$

#### **Results with «pulverised » walls:**

In the French fries shop:

odour reduction (around 35%) VOC reductions (around 30%)

- ★ Lab-built « e-noses »: no proofs of a significant reduction of « odours » during the day
- ✓ But-faster reduction of odour between days (fryers ON) and nights (fryers OFF)



Time

✓ Garbage room:

			-
Classes	Odourless	Odorous	
Non pulverised	25%	75%	
observations			
Pulverised observations	41%	<b>59%</b>	] ,
	•		

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+ GC-MS analyses

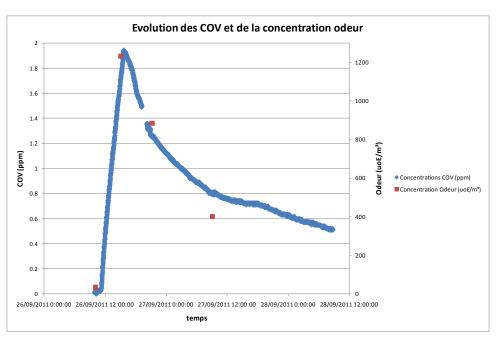
+ olfactometry measurements

#### Methodology development

Tests in emission chamber (50m<sup>3</sup>): fryer in the middle of the chamber

PID (VOC) Olfactometry (concentration + intensity)

#### Results with «pulverised » walls :





date	time	Fryer state	Odour concentration (uo <sub>F</sub> /m <sup>3</sup> )	VOC (PID) (ppb)
26/09/20 11	10 h 10	Fryer OFF	33	10
26/09/20 11	15 h 03	Fryer On at 11h00 and OFF at 15h40	1.232	1720
26/09/20 11	21 h 15	06 hours after switc off	883	1250
27/09/20 11	9 h 08	18 after swich off	401	800

Mainly: 1-Penten-3-ol, Octane, 2-Heptenal, (Z)-, 2,4-Heptadienal, (E,E)-, Nonanal, 2-Decenal, (E)- et 2,4-Decadienal, (E,E)-

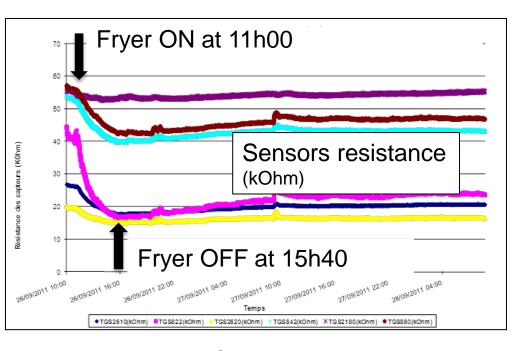
EUROPEAN COOPERATION IN SCIENCE AND TECHNOLOGY

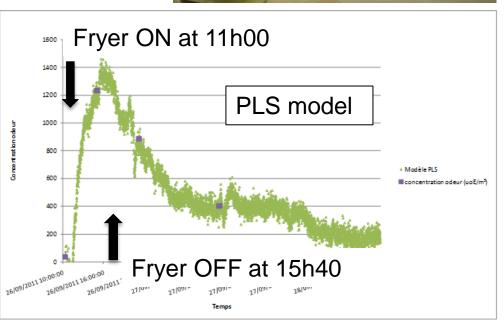
#### Efficiency of the product pulverised on the walls

Tests in emission chamber (50m<sup>3</sup>): fryer in the middle of the chamber MOS sensor array PID (VOC) Olfactometry (concentration + intensity)

Panellings were pulverised outside the chamber and installed in the early morning before the tests

#### **Results with «pulverised » walls:**







Efficiency of the product pulverised on the walls

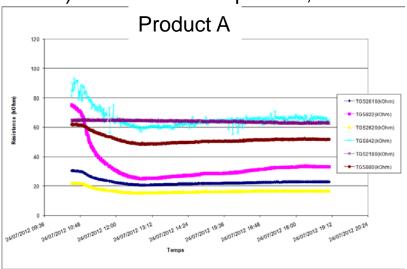
Tests in emission chamber (50m<sup>3</sup>) during 5 continuous days (july 2012) fryer in the middle of the chamber

Day 1 : without product Day 2 : with product A Day 3 : with product B Day 4 : with product C Day 5 : without product Panellings were pulverised outside the chamber and installed in the early morning before the tests

#### **Results:**

Product A: faster decrease of the odour (one night is needed) than without the product,

but low recovery of the sensors Product B: significant odour decrease after 06h Product C: also reduction for high odour level





### Take home message

- Push for development of new sensing technologies related to the IAQ evaluation
  - arrival of European and National laws,
  - emergence of the Environmental Assessments of buildings (Breeam, HQE,...),
  - increased awareness of the impact of indoor air quality on health
- Various interesting applications for chemical sensors and different Stakeholders' requirements
- Previous ULg studies highlighted several MOS limitations
  - high LOD
  - Lack of selectivity
  - Interferences
  - Low recovery time
  - (drift, humidity, ...)

#### WAIT and SEE

- Emergence of new sensing materials and new measurement principles (IMS)

Perspectives are again opened!

We plan to pursue again these previous projects



### **Chemical Sensor technologies in the world of IAQ**

#### ✓ What we need

#### $_{\rm O}$ Low cost sensors

to install several devices in the same room

#### ○ Long life time

to avoid costly development of classification and quantification models

#### Low drift and robustness

resistant to harsh environmental conditions

- Lower LOD (or preconcentration devices)
- o Selectivity (sensor array, T Modulation) ; cross-sensitivities
- o Low interferences
- Low humidity effects (T is not as important)

#### What we don't need

o Accurate output of chemical concentration:

alarm, presence-absence are often adequate

o Battery is not always required



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- o Jacques Nicolas (my predecessor, now retired)
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  - VITO: Eddy Goelen, Frederick Maes
    CSTC: Marc Lor (VITO), Karla Dinne, Kevin Vause



And my team SAM:



Gilles ADAM Noémie MOLITOR Laurent COLLARD Catherine HEYMAN



### International Symposium on **Olfaction and Electronic Nose**

# 28 June 2015 Dijon France





isoen

International Symposium on Olfaction and Electronic Nose



International Society for Olfaction and Chemical Sensing

# International Symposium on Olfaction and Electronic Noses

- ran by the International Society for Olfaction and Chemical Sensors
  - ✓15<sup>th</sup> issue, since 1994

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- ✓ Scientific Committee since 1999
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  - ✓General Assemblies



16<sup>th</sup> International Symposium Olfaction & Electronic Noses ISOEN 2015

"20 years of E-nose, time to take stock"



# Topics

### Solid State Sensor Technologies

**Gas sensors** 

✓ Biosensors

**✓Ion Selective Electrodes** 

✓Other gas detection technologies

### System level

✓ Gas Sensor Testing Systems

✓ Sampling Techniques

✓Instrumentation Software/Hardware design

✓ Data Processing

### > Odor / aroma generation

✓ Olfactometers✓ Olfactory displays & shooters



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"20 years of E-nose, time to take stock"





### Olfaction

- ✓ Biological Principles of Olfaction
- ✓Odor Intensity Measurement, cross
- modalities
- ✓ Bio-inspired sensing
- ✓ Computational models of the olfaction and Bio-inspired Algorithms

### > Analytical techniques

✓ Correlations with references, Experimental Design, Process Control, Calibration, Transfer

### > Applications

✓Industrial, Environment, Automotive, Breath, Medical, Consumer,...

✓Mobile robots based on gas sensing



16<sup>th</sup> International Symposium Olfaction & Electronic Noses ISOEN 2015

"20 years of E-nose, time to take stock"



## Thanks for your attention



ULg-Faculty of Sciences Department of environmental sciences Sensing of Atmospheres and Monitoring Arlon (Belgium)

> www.campusarlon.ulg.ac.be/ 0032 63 23.08.59

> > acromain@ulg.ac.be