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

COST Action TD1105

Satellite event – Eurosensors XXIX, Freiburg, 09.09.2015

Action Start date: 01/07/2012 - Action End date: 30/06/2016

Year 4: 1 July 2015 - 30 June 2016 (*Ongoing Action*)

Integrated sensor systems for indoor applications: ubiquitous monitoring for improved health, comfort and safety

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UNIVERSITÄT DES SAARLANDES

Lehrstuhl für Messtechnik

COST EUROPEAN COOPERATION IN SCIENCE AND TECHNOLOGY

EuNetAir European Network on New Sensing Technologies for Air-Pollution Control and Environmental Sustainability

COST is supported by the EU Framework Programme

ESF provides the COST Office through a European Commission contract

Why worry about indoor air?

- Safety
  - Gas leak detection (combustible gases, e.g. CH_4)
  - Fire detection (various gases)
  - Hazardous gas detection (e.g. CO)
- Malodor detection (kitchen & bathroom ventilation)
- HVAC systems
  - Reduced air circulation for greatly reduced energy consumption
    - ➢ CO_2 monitoring for fresh air
  - Mold detection / prevention
  - Increased levels of VOCs lead to sick building syndrome
    - ➢ Selective (formaldehyde, benzene etc.) and sensitive (ppb level) detection
  - Systems have to be adapted to the specific room use scenario

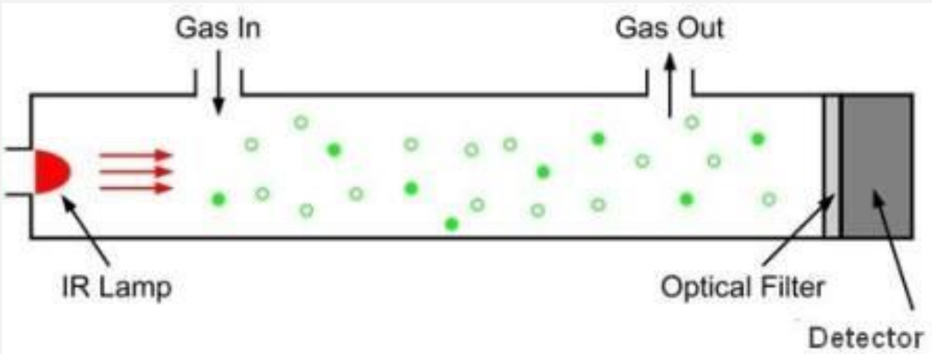
Sensor requirements

- Low cost
- Networked systems (in major buildings, but also private homes)
- Long lifetime: >10 years without maintenance for private homes

Which sensors are used today?

- Safety
  - Gas leak detection: human nose, Japan: MOS; pellistors: only industrial use
  - Fire detection: various sensors, mostly optical;
    gas sensor systems under development (EC, MOS, GasFET)
  - Hazardous gas detection: EC, MOS
- Malodor detection: MOS
- HVAC systems
  - CO_2 monitoring: NDIR (in major rooms/buildings), EC & GasFET (emerg.)
  - VOCs: MOS: total VOC and specific (emerging), GasFET (emerging)

Example: CO_2 monitoring based on NDIR sensors



http://www.co2meter.com

http://www.senseair.com

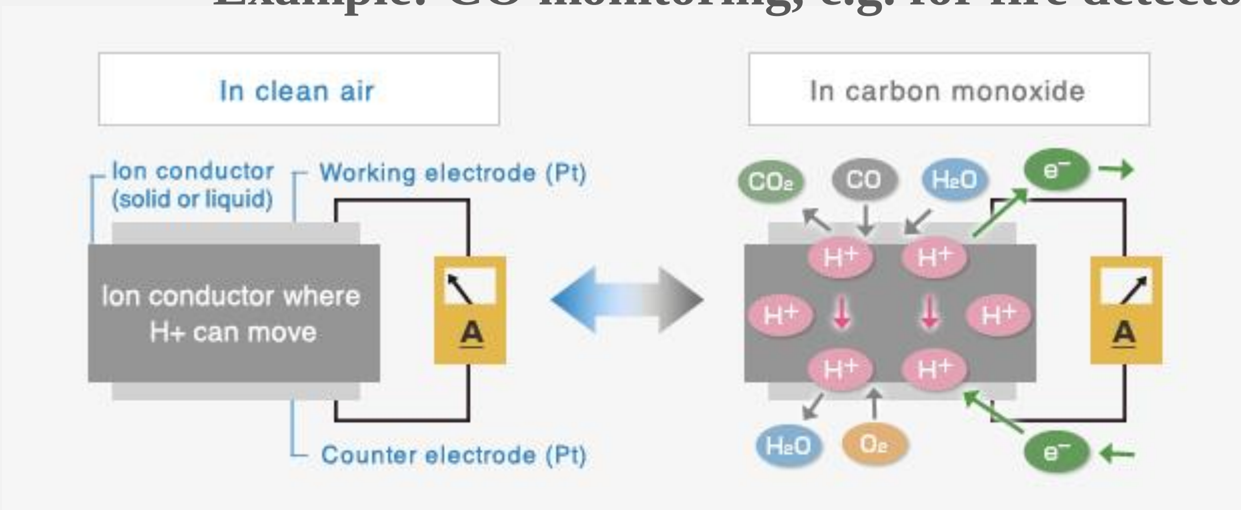
Physical sensor principle: very reliable, very exact, fairly costly and large

- long path length required for high sensitivity
- wavelength tuned for selectivity, for CO_2 excellent, other gases OK
- good stability due to physical sensing principle, improved with reference path

Alternatives: solid-state electrochemical sensor, work-function based sensors?

> Sensor applications for Indoor Air

Example: CO monitoring, e.g. for fire detectors, with EC sensors



http://www.figaro.co.jp

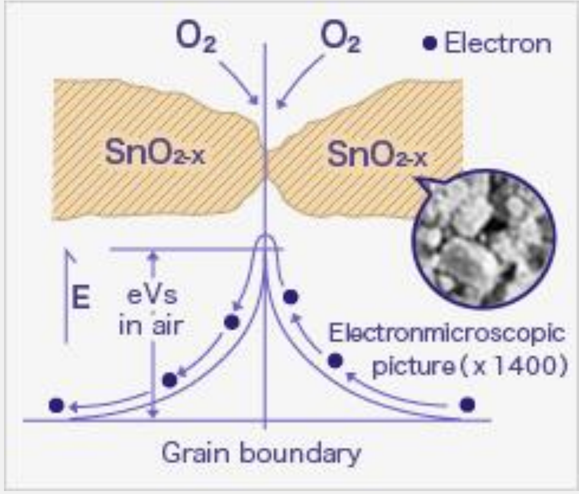
http://www.alphasense.com

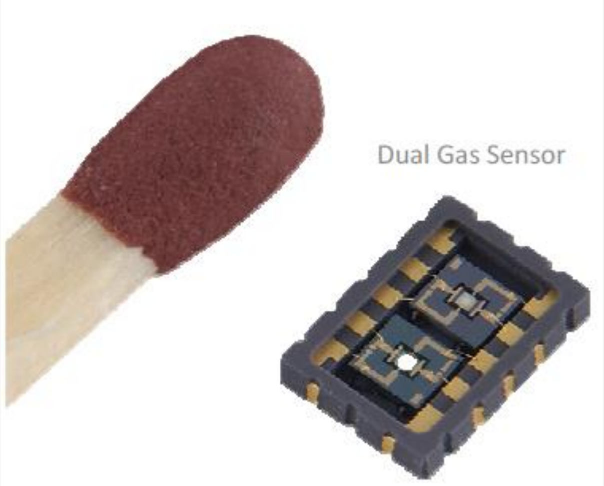
Electrochemical sensing principle: low power consumption, fairly costly and large

- Sensitivity in the ppm to ppb range depending on target gas
- Selectivity tuned with electrolyte, electrodes, operating voltage, generally OK
- Stability limited due to electrolyte consumption, especially difficult at low r.h.

Alternatives: CMOS based GasFET (e.g. mySENS by Micronas)

Example: gas leak detection with Metal Oxide (MOX) sensors





http://www.figaro.co.jp

http://www.sgxsensortech.com

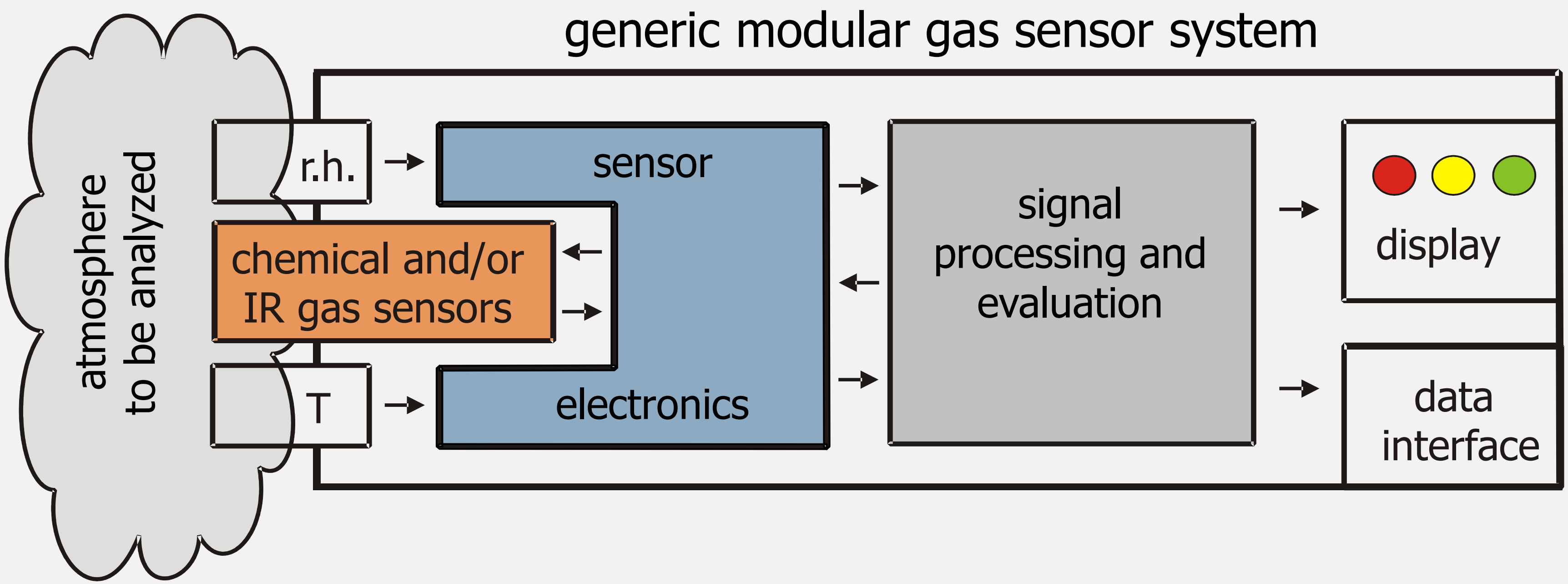
Resistance change due to grain boundary effect: low cost, very robust

- Sensitivity down to sub-ppb levels depending on target gas
- Selectivity depends on material (SnO_2, WO_3), doping, temperature, generally poor
- Stability limited due to electrolyte consumption, especially difficult at low r.h.

Alternatives: wide material range, e.g. polymers, nanowires, disposable sensors

The three "S"

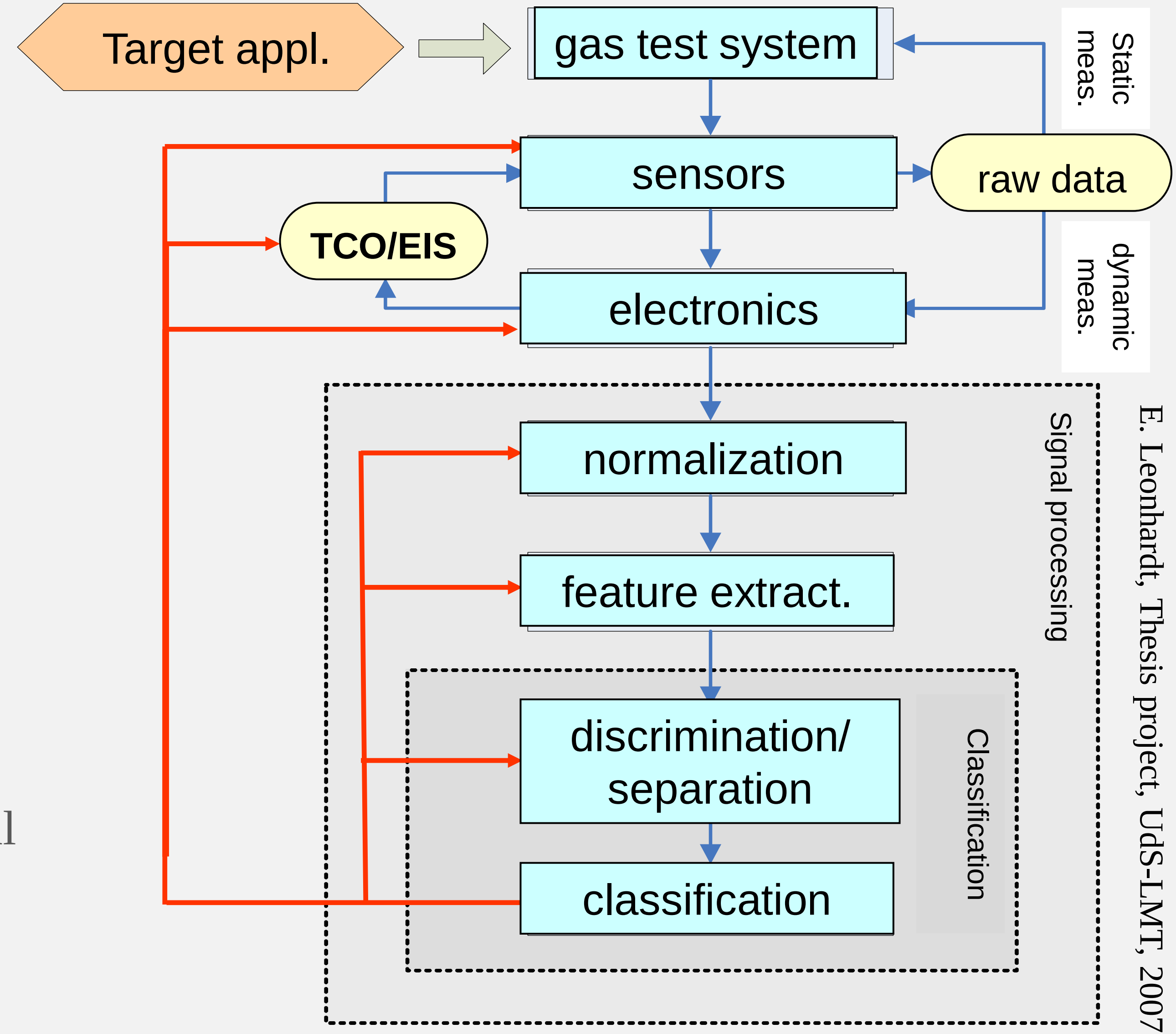
- Sensitivity
  - Broad spectrum
    from below ppb (for malodors, ozone, hazardous VOCs)
    up to 1000 ppm (gas leak, CO_2)
- Selectivity
  - False alarms are primary concern for fire detection (ratio 10:1)
  - VOC detection: hazardous (formaldehyde) vs. neutral (alcohol vapor, cleaning agents) vs. wanted (odorants)
- Stability
  - Industrial applications: maintenance interval < 6 months
  - Public buildings: annual or bi-annual tests (if that)
  - Private homes: 10 years lifetime w/o regular maintenance?


generic modular gas sensor system
atmosphere
to be analyzed
r.h.
T
chemical and/or
IR gas sensors
sensor
electronics
signal
processing and
evaluation
display
data
interface

Many possibilities for optimization:

- Sensor selection
- Operating mode
  - TCO
  - EIS
  - GBCO
- Data acquisition
- Signal preprocessing
- Feature extraction
- Separation
- Classification

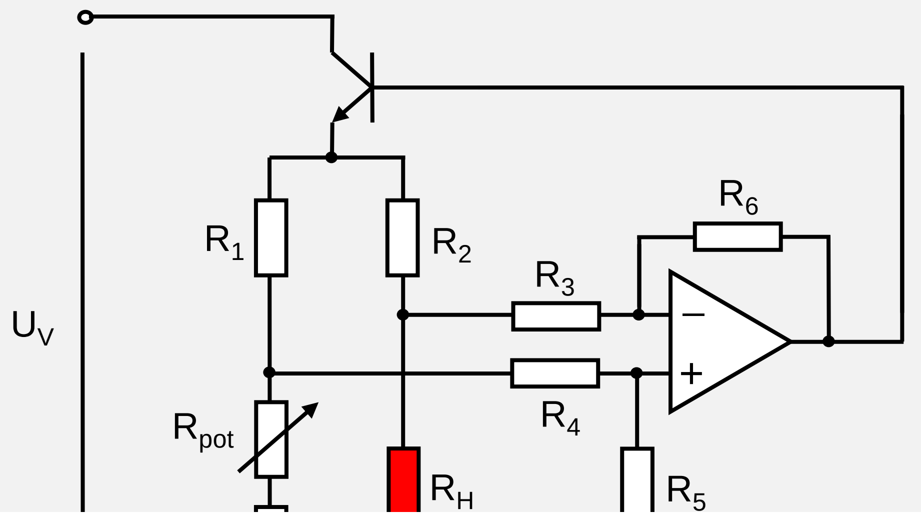
…and **always** testing under real application conditions (field testing)!



E. Leonhardt, Thesis project, UdS-LMT, 2007

Hardware platform GasTON for exact temperature control and large dynamic range data acquisition – Gas sensor T-cycle Operating uNit

- Heater temperature control
  Heater resistor $R_H(T)$ controlled for exact temperature control of (micro-)hotplates
- Sensor read-out with large dynamic range
  for MOS, GasFET and pellistor type sensors



➢ **now commercialized "OdorChecker"**
by 3S GmbH (spin-off of LMT)

VOC-IDS: Volatile Organic Compound Indoor Discrimination Sensor

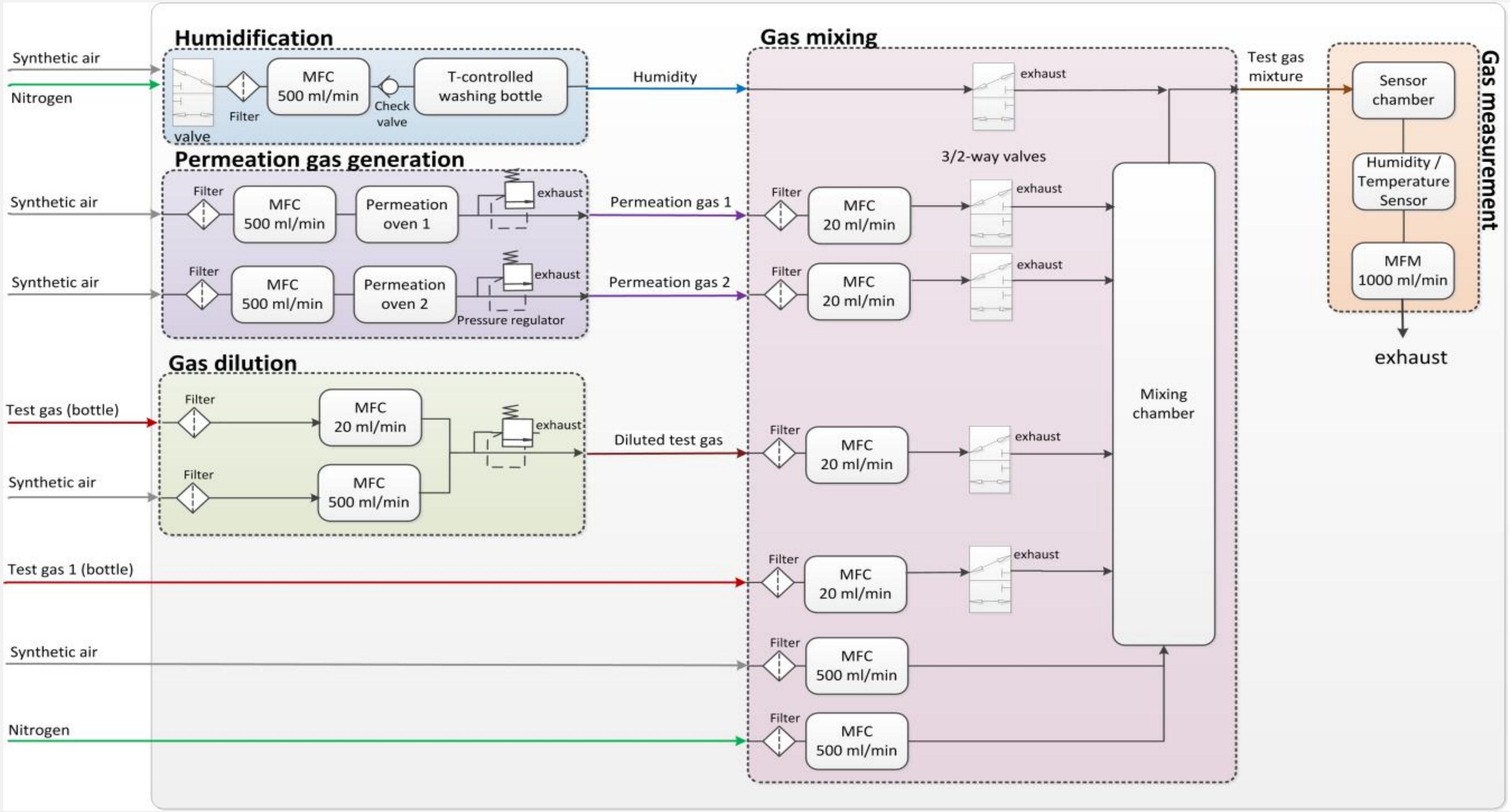
- Transnational project funded within MNT-ERA.net
- Selective VOC detection, primarily formaldehyde, benzene
- Novel ceramic nanomaterial metal-oxide semiconductor gas sensors
- Intelligent signal processing based on temperature cycling
- Networked systems connected to KNX bus

SENSIndoor: Nanotechnology based intelligent multi-SENsor System with selective pre-concentration for Indoor air quality control

- EU-FP7 project NMP.2013.1.2-1:
  Nanotechnology-based sensors for environmental monitoring
- Microtechnology based approach for MOS and SiC-GasFET sensors
- Pre-concentration to boost sensitivity and selectivity
- Integrated multi-sensor approach
- Application specific priorities and field tests

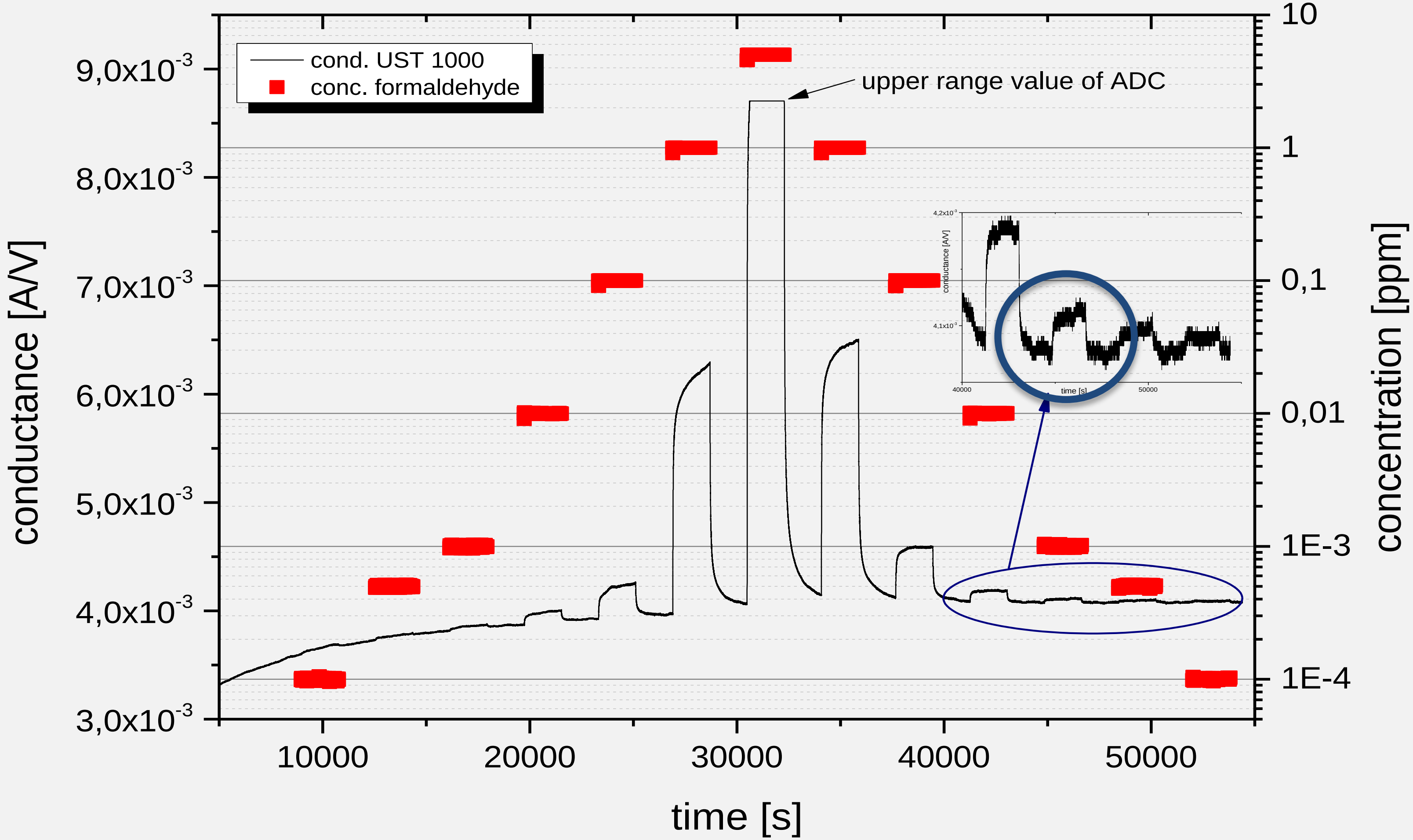
Further EU projects with focus on indoor air quality:
IAQSense, MSP

First step: novel gas mixing system for VOC testing/calibration @ (sub) ppb-level



N. Helwig et al.: Gas mixing apparatus for automated gas sensor characterization, Meas. Sci. Technol. 25 (2014) 055903

Novel gas mixing system: results of first sensor tests



Sensor reaction to 1 ppb formaldehyde

Relevance? Legal limits in France for indoor air: Formaldehyde 25 ppb in 2015; Benzene 0.6 ppb in 2016

WHO limits similar

N. Helwig et al.: Gas mixing apparatus for automated gas sensor characterization, Meas. Sci. Technol. 25 (2014) 055903

MNT-ERA.net project VOC-IDS

- Example for selective detection of VOCs in interfering background
- Classification of formaldehyde, benzene, naphthalene in the presence of ethanol

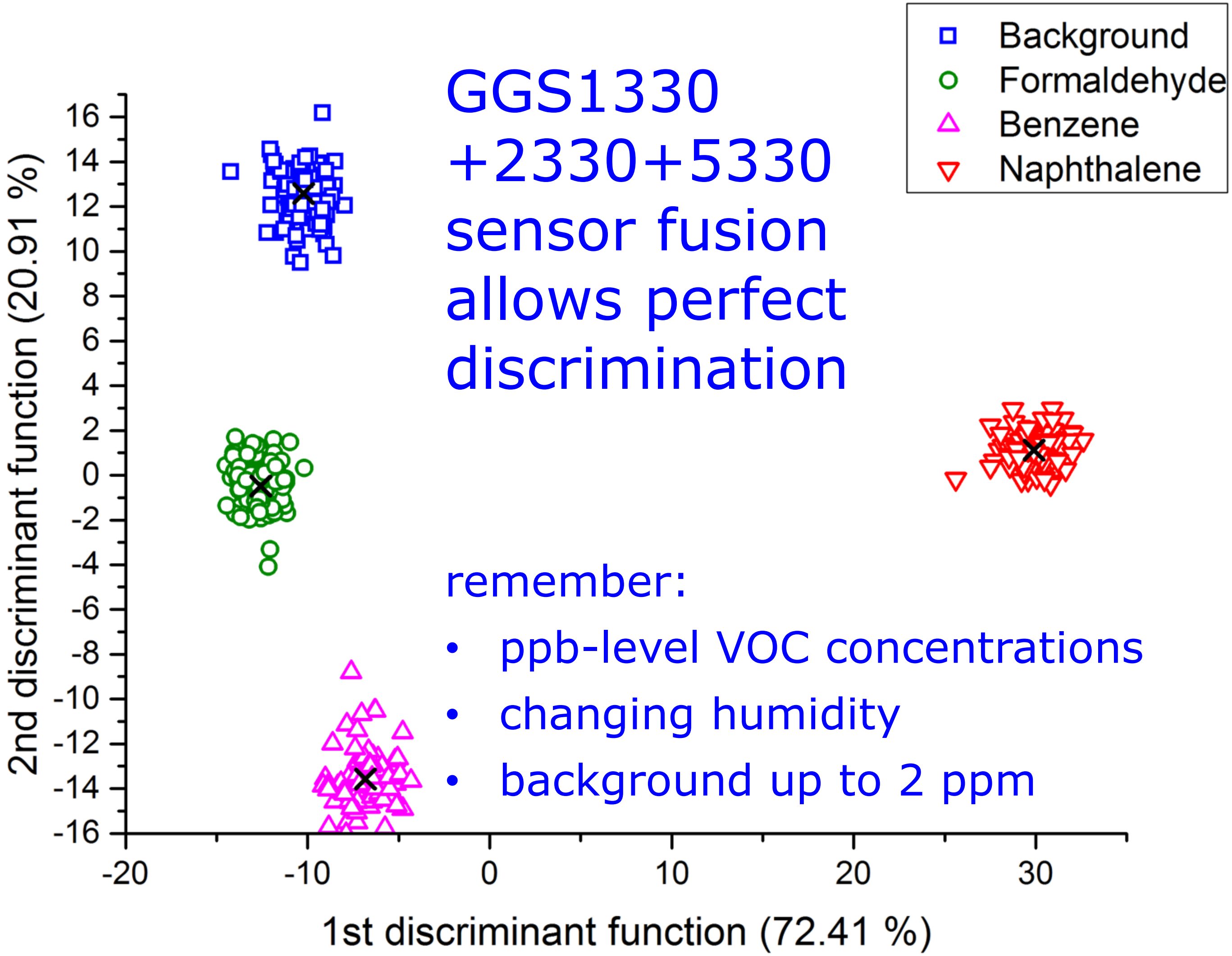
target gas	Concentration (ppb)	humidity	Interferents (EtOH ppm)
Air	NA	40%, 60%	none, 0.4, 2
Formaldehyde	10, 100	40%, 60%	none, 0.4, 2
Benzene	0.5, 4.7	40%, 60%	none, 0.4, 2
Naphthalene	2, 20	40%, 60%	none, 0.4, 2

Classification target	interferent concentrat.	relative humidity	number of LDA steps for charac.	Estimated # of LDAs
generalized classification	0, 0.4, 2	40%, 60%	1	1
classification w known r.h.	0, 0.4, 2	known	1 (2)	(1+) 5*1
classification w known EtOH	known	40%, 60%	2	1+10(?)*1

LEHRSTUHL FÜR MESSTECHNIK

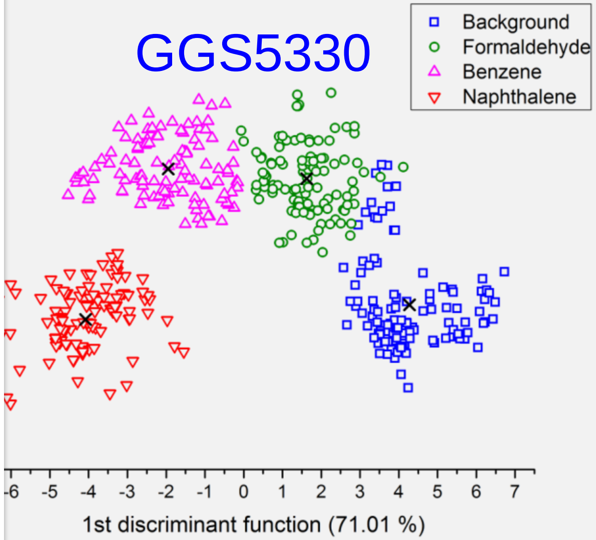
mnt-era.net

GGS1330 +2330+5330 sensor fusion allows perfect discrimination



remember:

- ppb-level VOC concentrations
- changing humidity
- background up to 2 ppm



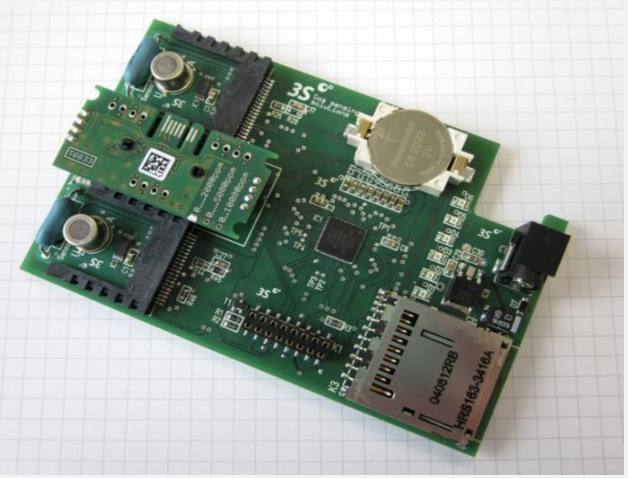
M. Leidinger et al.: Selective Detection of Hazardous Indoor VOCs Using Metal Oxide Gas Sensors, EUROSENSORS 2014, talk C1L-B02

> IAQ monitoring: field test systems

- Stand-alone field test systems by 3S GmbH (Saarbrücken, Germany)
- 2 MOS gas sensors (+ CO_2 + humidity) with independent temperature control
- Data storage on SD card

➢ **Conclusion of field tests: even better sensitivity and selectivity required!**

Setup for system calibration



Motivation

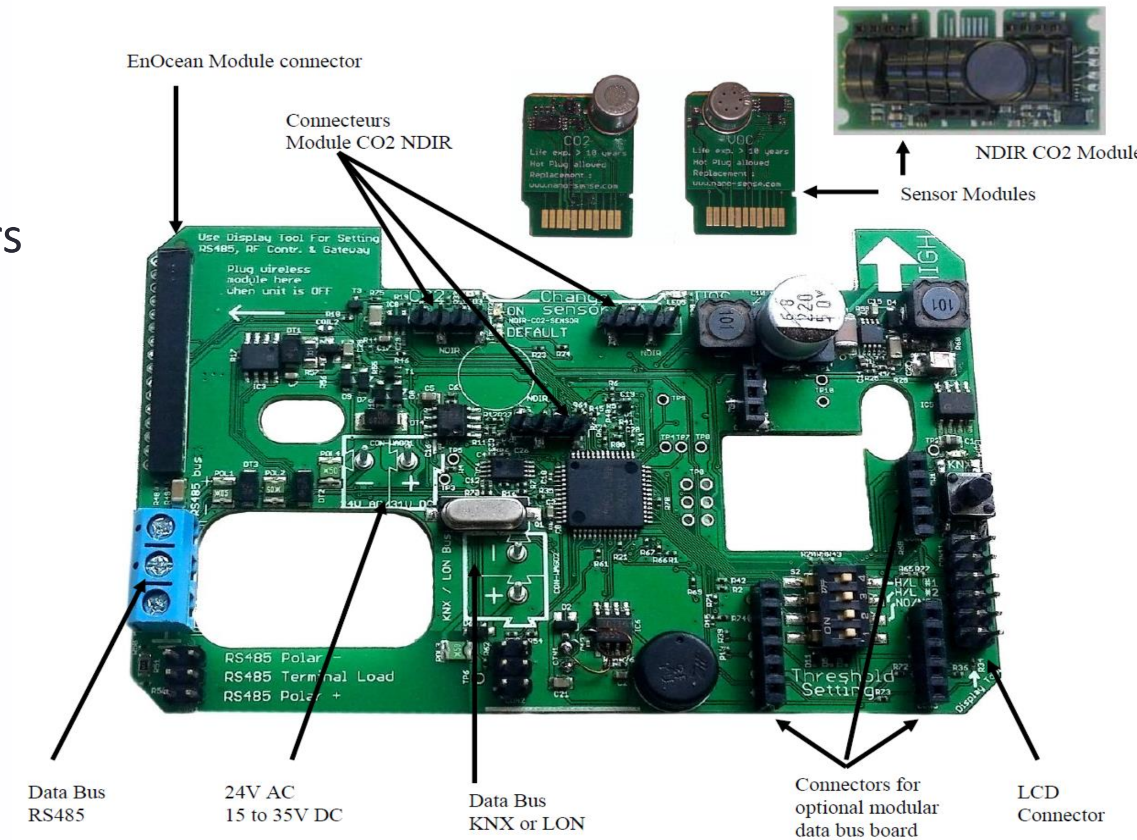
- People spend more than 80 % of their time indoors where fresh air exchange is increasingly limited to reduce energy consumption.
- Indoor air pollution contributes significantly to the global burden of disease.
- Continuous ventilation would greatly increase energy consumption for HVAC (heating, ventilation, air conditioning) systems.
- Low-cost sensor systems are required to provide ubiquitous Indoor Air Quality (IAQ) monitoring.

  ➢ **Core motivation for the SENSIndoor project**

State-of-the-art

- Demand controlled ventilation today
  - mostly CO_2 monitoring, at best total VOC (TVOC)
  - CO_2 based on IR absorption or solid state electrolyte
  - TVOC based on metal oxide semiconductor (MOS) sensors

E4000 Air Quality Probe
(NanoSense SARL)

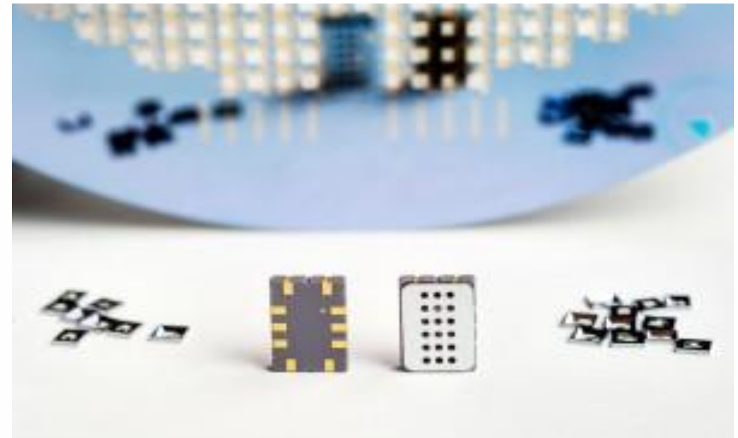


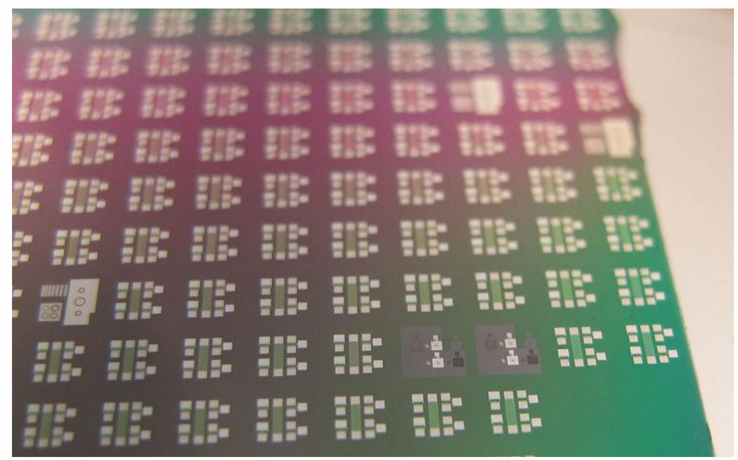
SENSIndoor origins

- **VOC-IDS** (MNT-ERA.net collaborative project)
  Volatile Organic Compound Indoor Discrimination Sensor
  - Partners: USAAR-LMT, IDMEC-FEUP - Instituto de Engenheria Mecânica, University Porto (P), UST Umweltsensortechnik GmbH (D), 3S GmbH (D), NanoSense SARL (F), Weinzierl Engineering GmbH (D), CIAT - Compagnie Industrielle d'Application thermique S.A. (F), ALDES Aéraulique S.A. (F)
- COST action TD1105 **EuNetAir**
  European Network on New Sensing Technologies for
  Air-Pollution Control and Environmental Sustainability
  - Partners: U Linköping (A Lloyd Spetz: vice chair of action), U Oulu, USAAR, 3S GmbH, SenSiC AB, SGX Sensortech S.A.
  - Several topics identified to be addressed in call **NMP.2013.1.2-1 Nanotechnology-based sensors for environmental monitoring**

SENSIndoor technologies 1

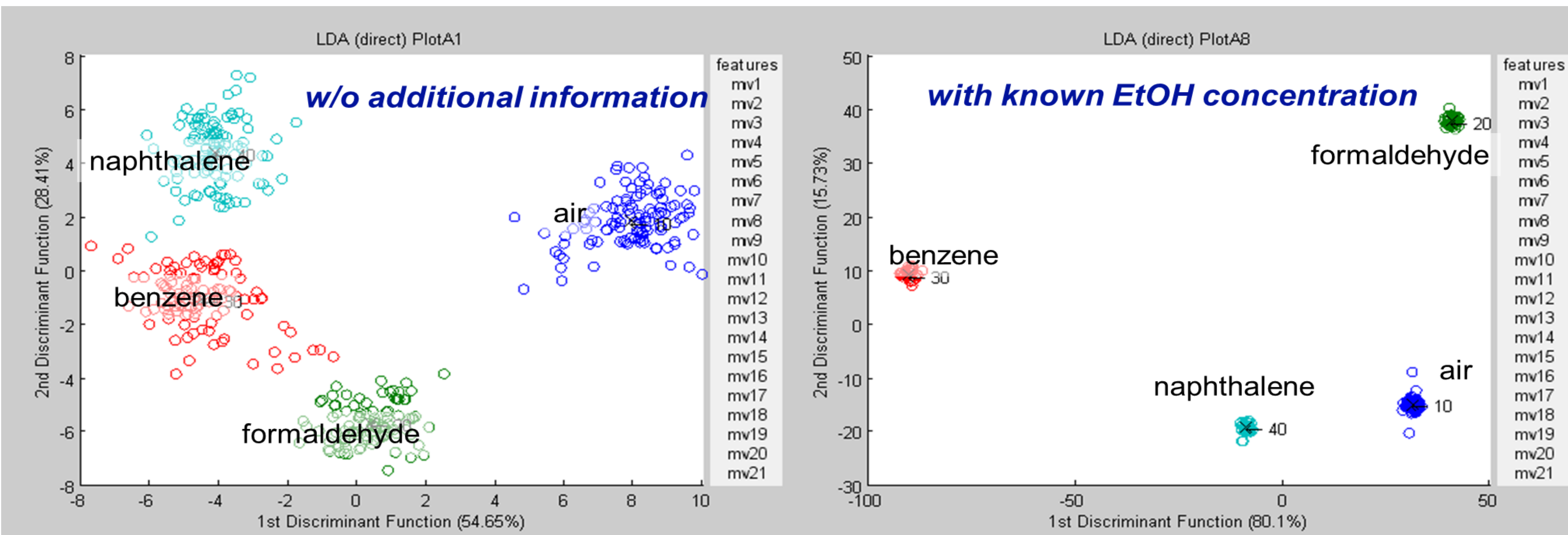
- Sensor technologies
  - **MOS – Metal oxide semiconductor** (*SGX Sensortech, USAAR-LMT*)
    - well known for high sensitivity and robustness @ low-cost
    - MEMS technology for mass production and low power consumption
  - **GasFET – Gas-sensitive Field Effect Transistors** (*LiU, SenSiC*)
    - complementary technology (polarity ⇔ reaction)
    - SiC technology for chemical robustness and high operating temperatures





SENSIndoor technologies 2

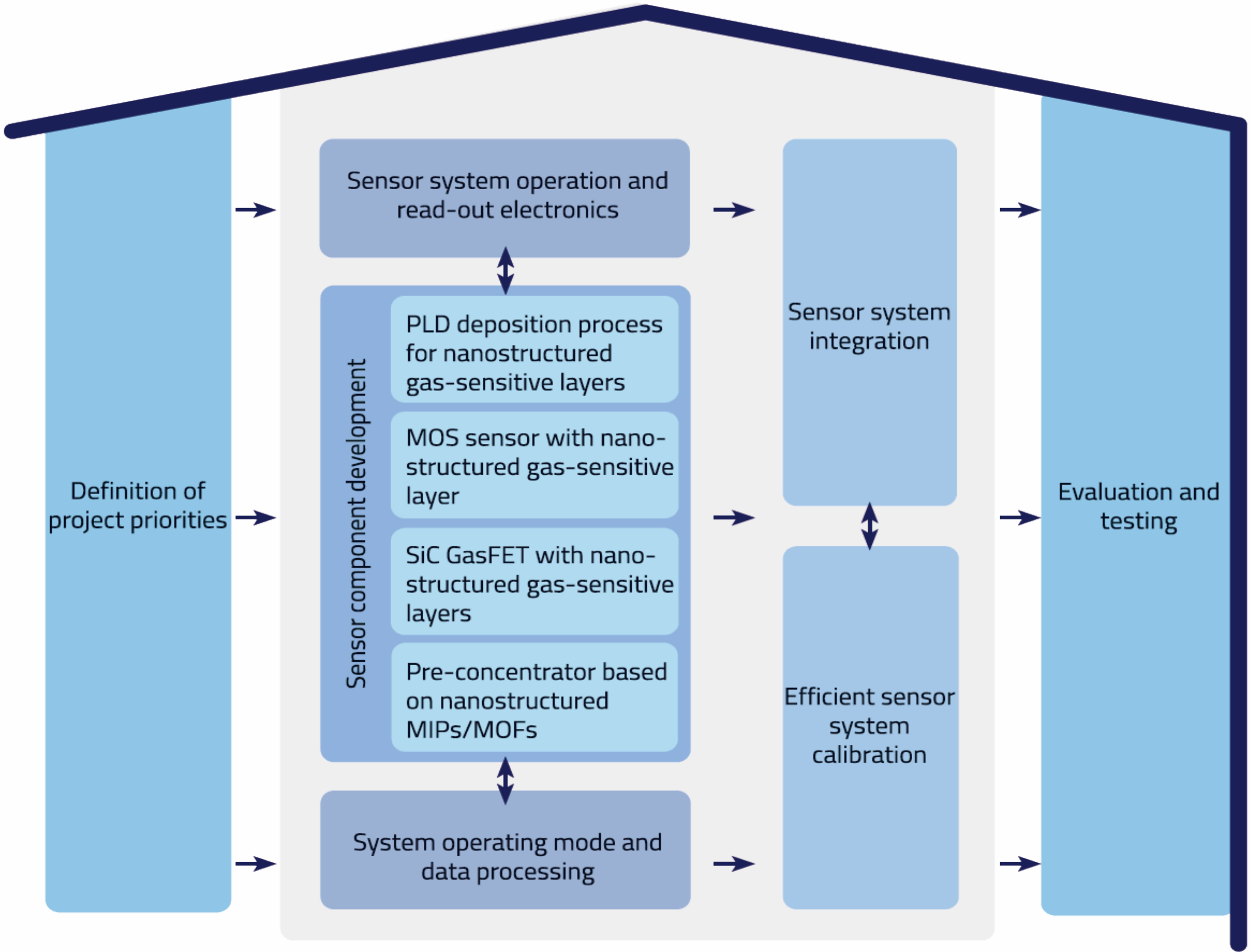
- Dynamic operation and intelligent signal processing
  - **Temperature Cycled Operation** (*USAAR-LMT, NanoSense, 3S*)
    to increase selectivity ("virtual multisensor") and stability



SENSIndoor overview

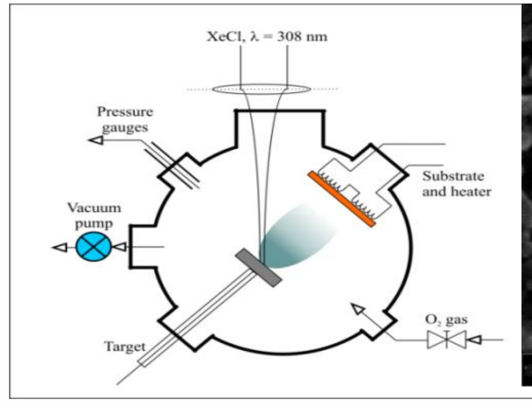
Project structure:

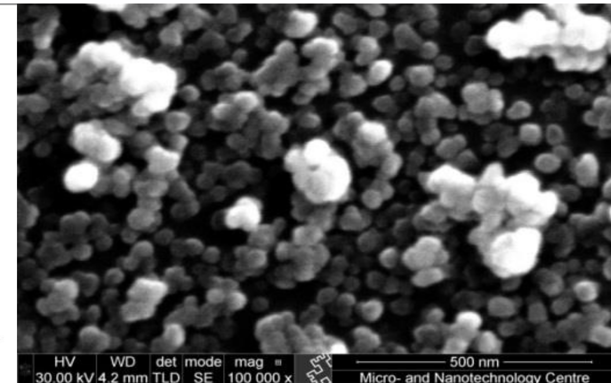
A clear road from application requirements to field evaluation



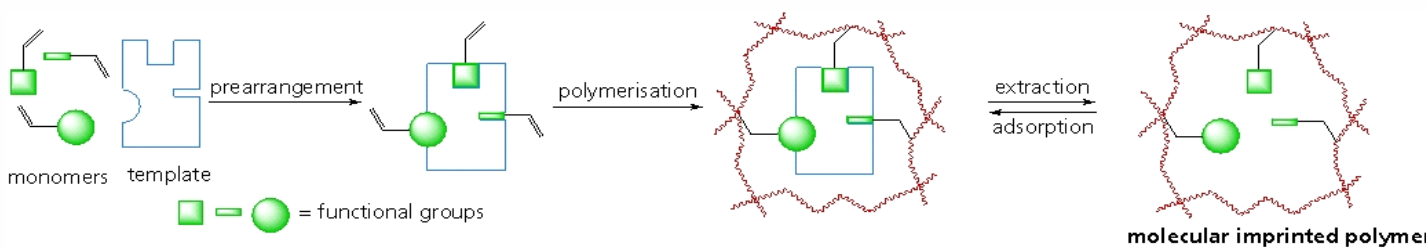
SENSIndoor technologies 3

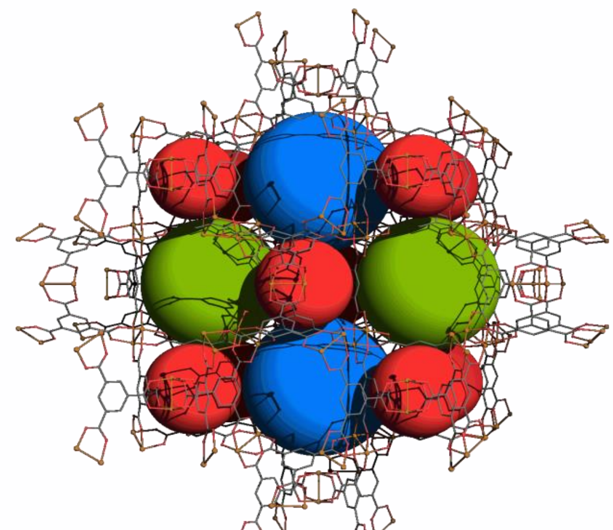
- Nanotechnology for improved sensor elements
  - **Pulsed Laser Deposition** *(U Oulu, Picodeon)* for novel, highly sensitive gas-sensitive layers suitable for wafer level mass production





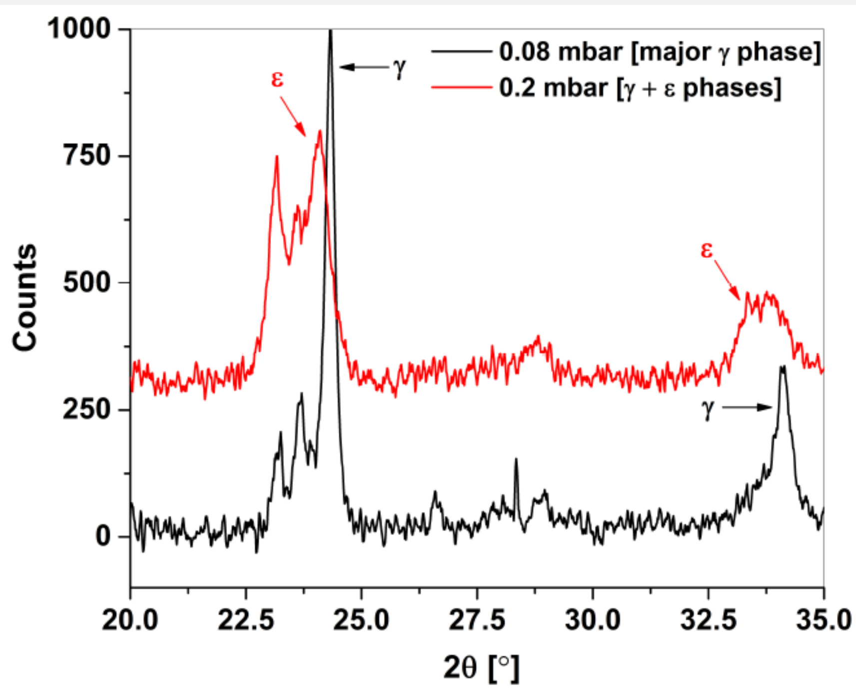
  - **Selective pre-concentration** *(FhG-ICT)* based on MOFs (metal-organic frameworks) → and MIPs ↓ (molecular imprinted polymers)

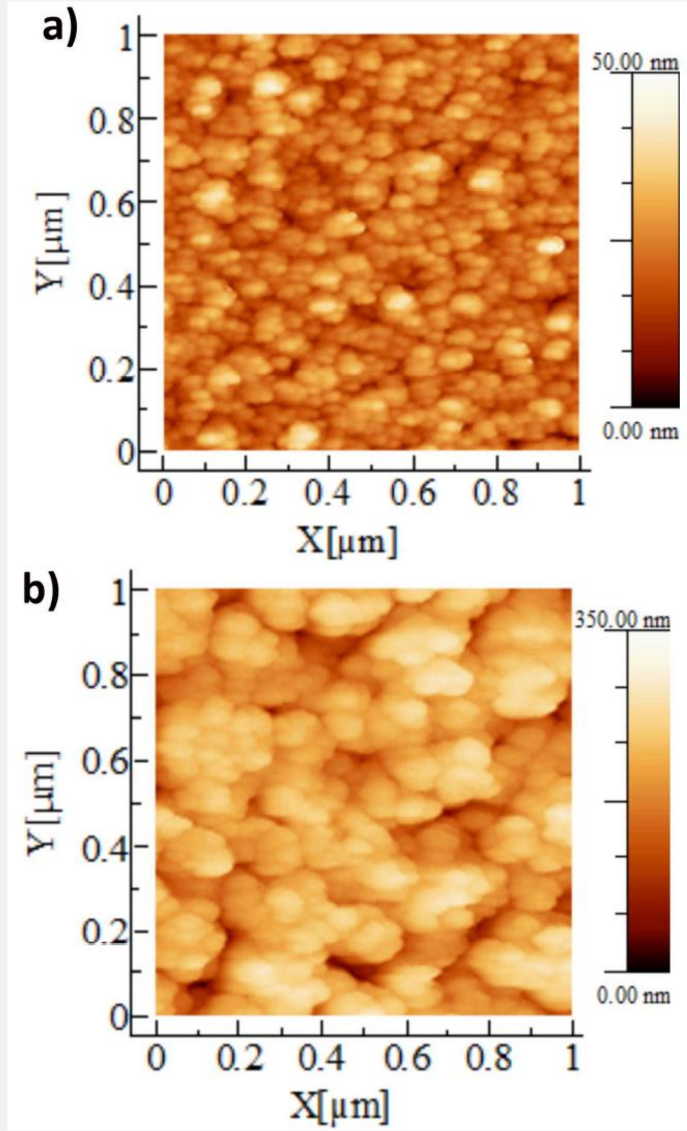




Depositions of WO_3 layers
(Oulu University w 25 ns laser)

- Characterized w AFM → and XRD





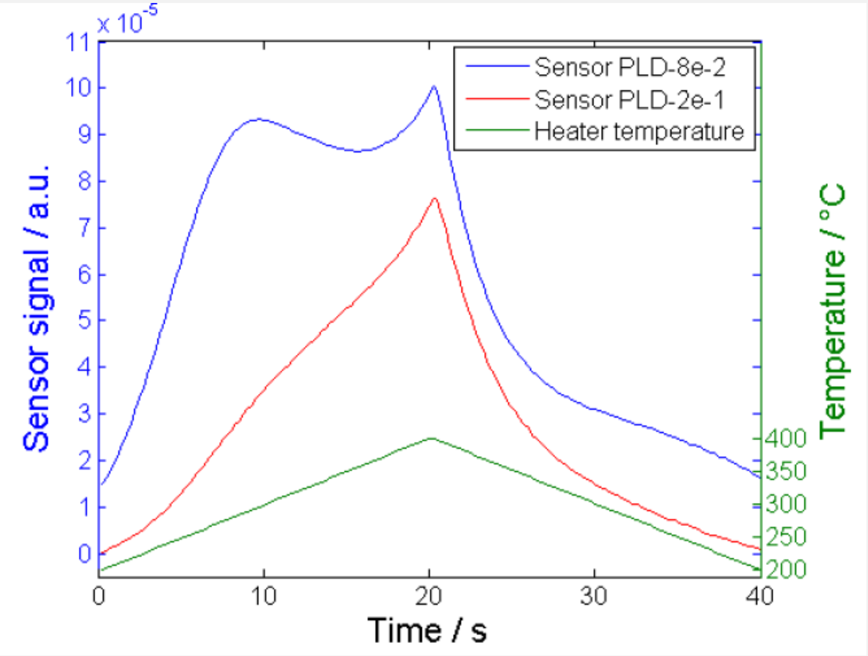
SENSIndoor

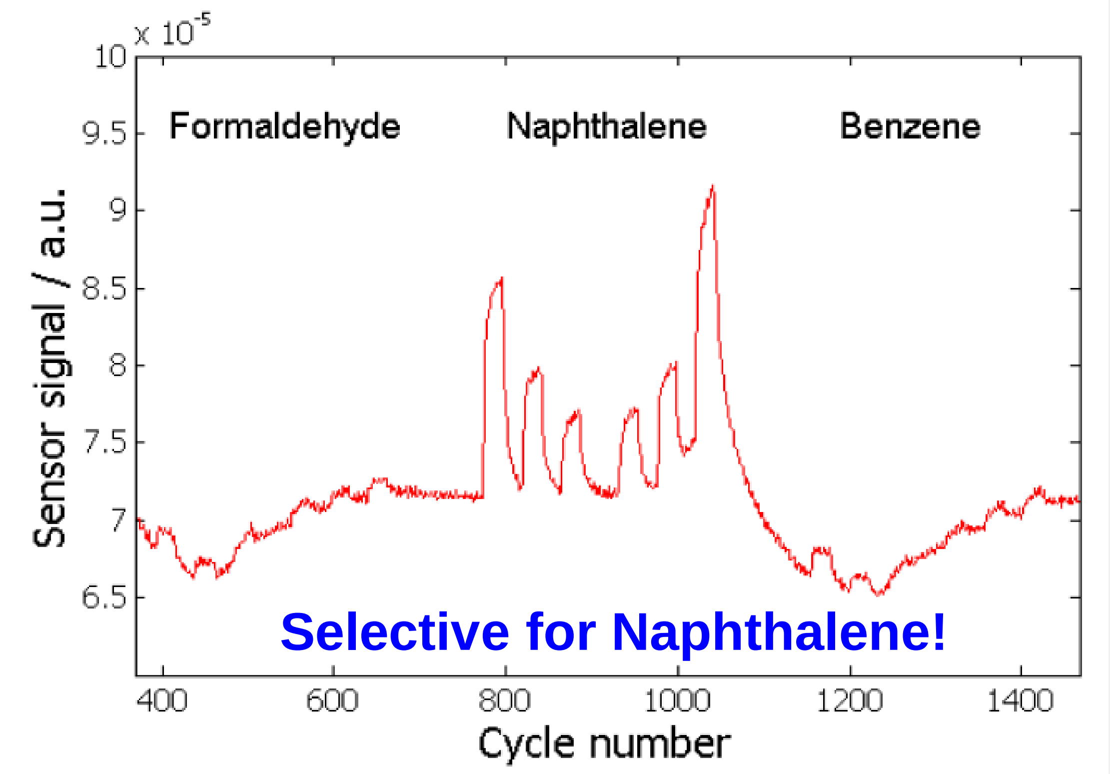
M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze:
Nanostructured WO_3 Semiconductor Gas Sensor for Selective Detection of Naphthalene,
SENSOR 2015, Nuremberg, May 19 – 21, 2015.

See also:
Joni Huotari et al.: Pulsed laser deposition of metal oxide nanoparticles, agglomerates, and nanotrees for chemical sensors
EUROSENSORS XXIX
Poster: TP-I09

Testing with ppb level VOCs
(Saarland University)

- Simple temperature cycle →
- Quasi-static signal @ max. temp.

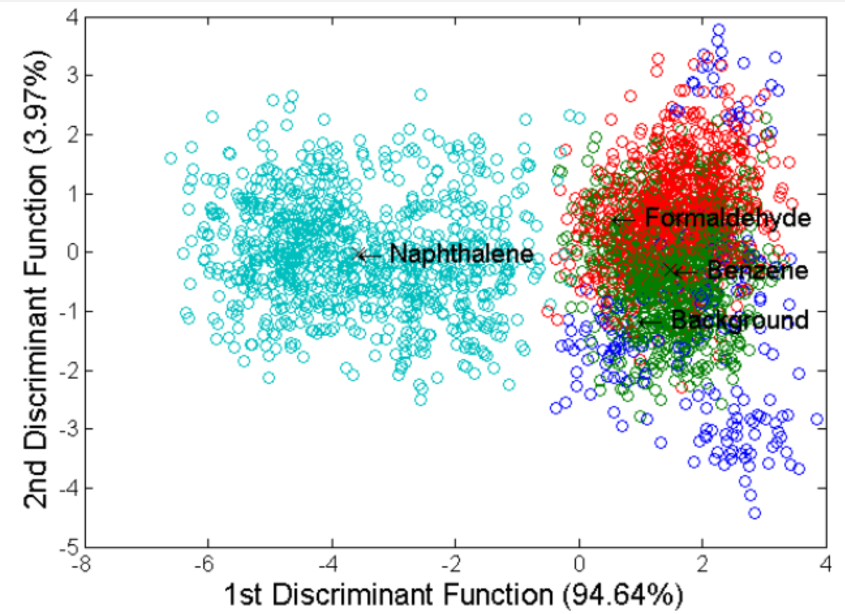


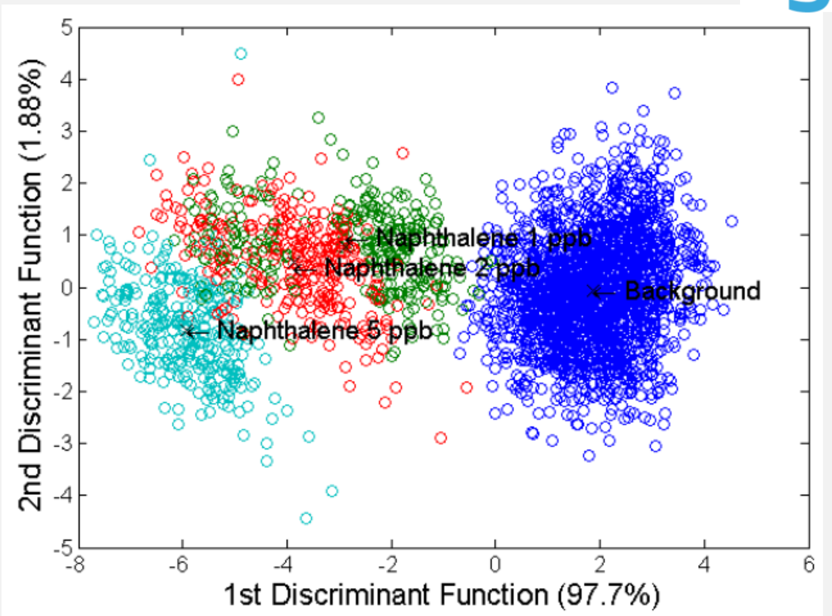


M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze: Nanostructured WO_3 Semiconductor Gas Sensor for Selective Detection of Naphthalene, SENSOR 2015, Nuremberg, May 19 – 21, 2015.

Identification and Quantification using LDA
(Saarland University)

- Sensor deposited at 0.2 mbar O_2 pressure





LOOCV: Naphthalene 99% background + 5 ppb: 95%

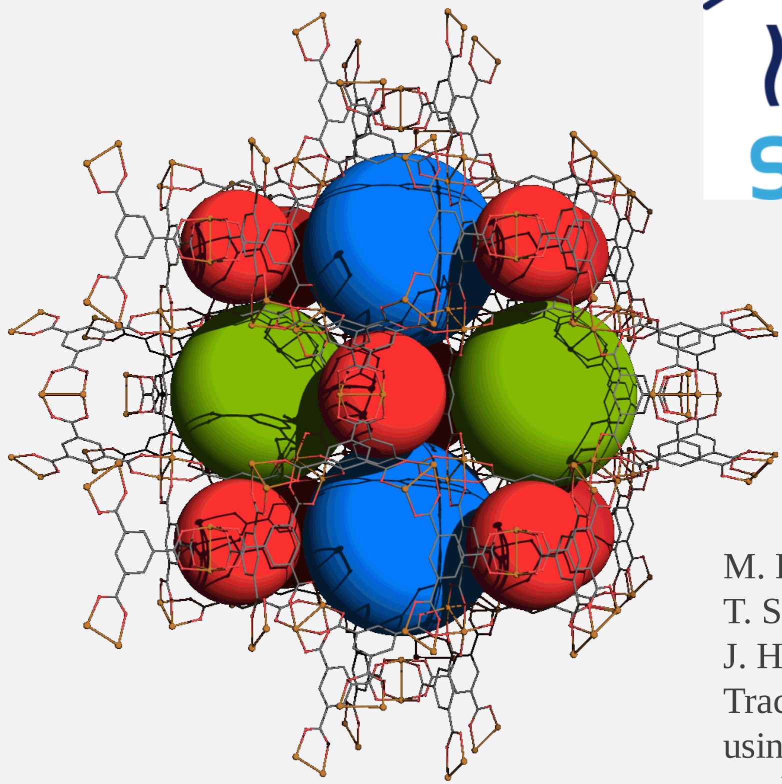
Note: data include **changing r.h.** and **ethanol** (up to 1 ppm)

M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze: Nanostructured WO_3 Semiconductor Gas Sensor for Selective Detection of Naphthalene, SENSOR 2015, Nuremberg, May 19 – 21, 2015.

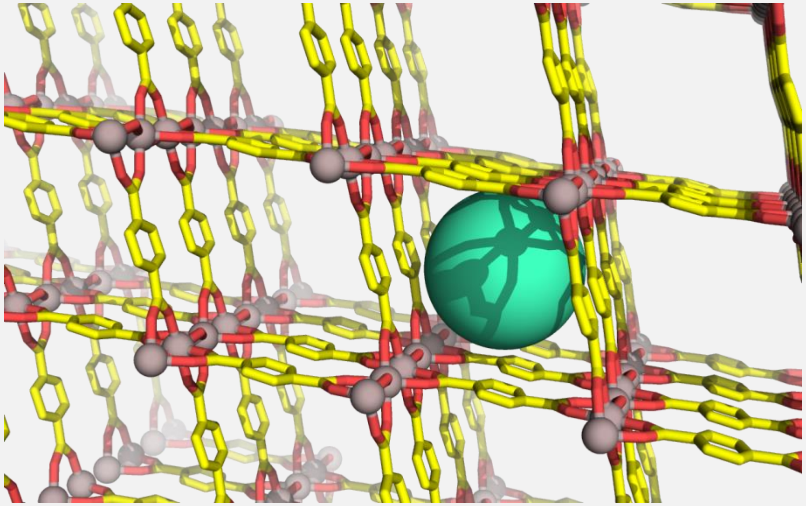
SENSIndoor

MOF layers deposited
(Fraunhofer ICT)

HKUST-1



MIL-53

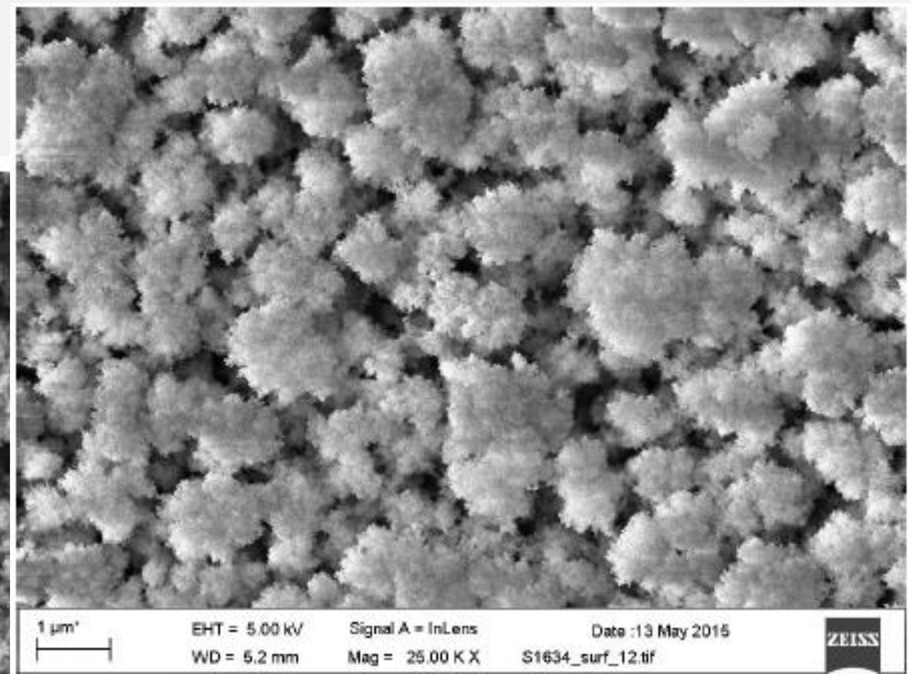


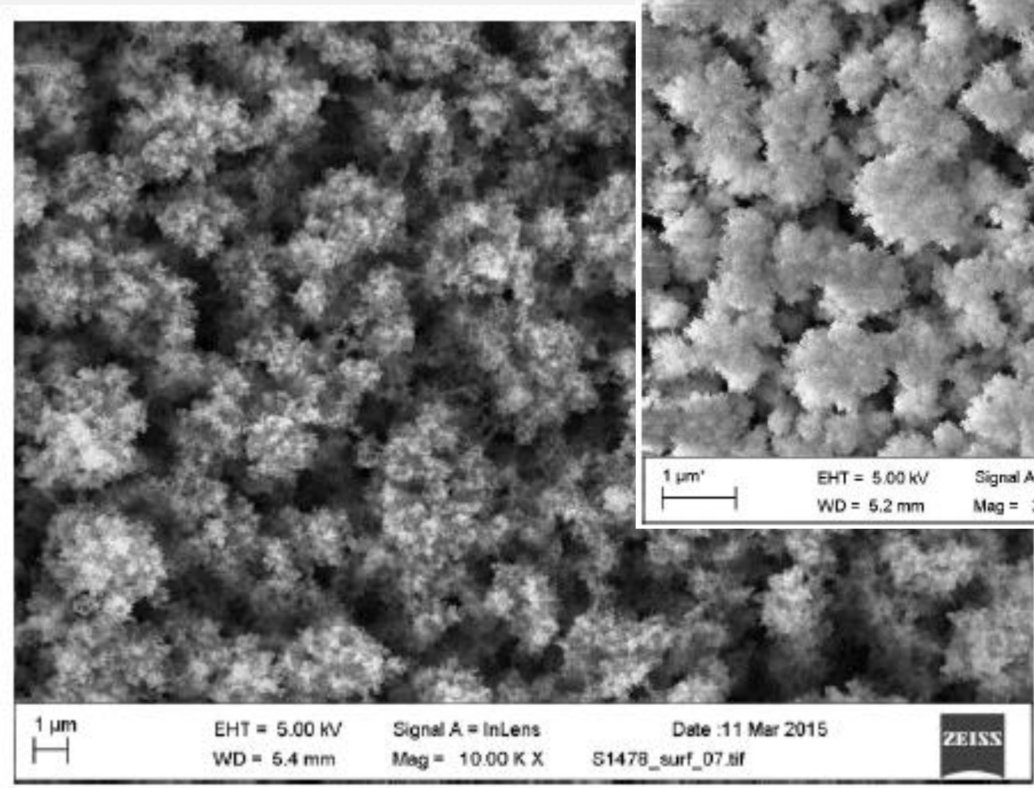
M. Leidinger, M. Rieger, T. Sauerwald, M. Nägele, J. Hürttlen, A. Schütze: Trace gas VOC detection using metal-organic frameworks micro pre-concentrators and semiconductor gas sensors, EUROSENSORS XXIX, Poster TP-E01

Next generation: SnO_2 and WO_3 w catalyst layers

(Picodeon w ps laser)

- SnO_2 →
- WO_3



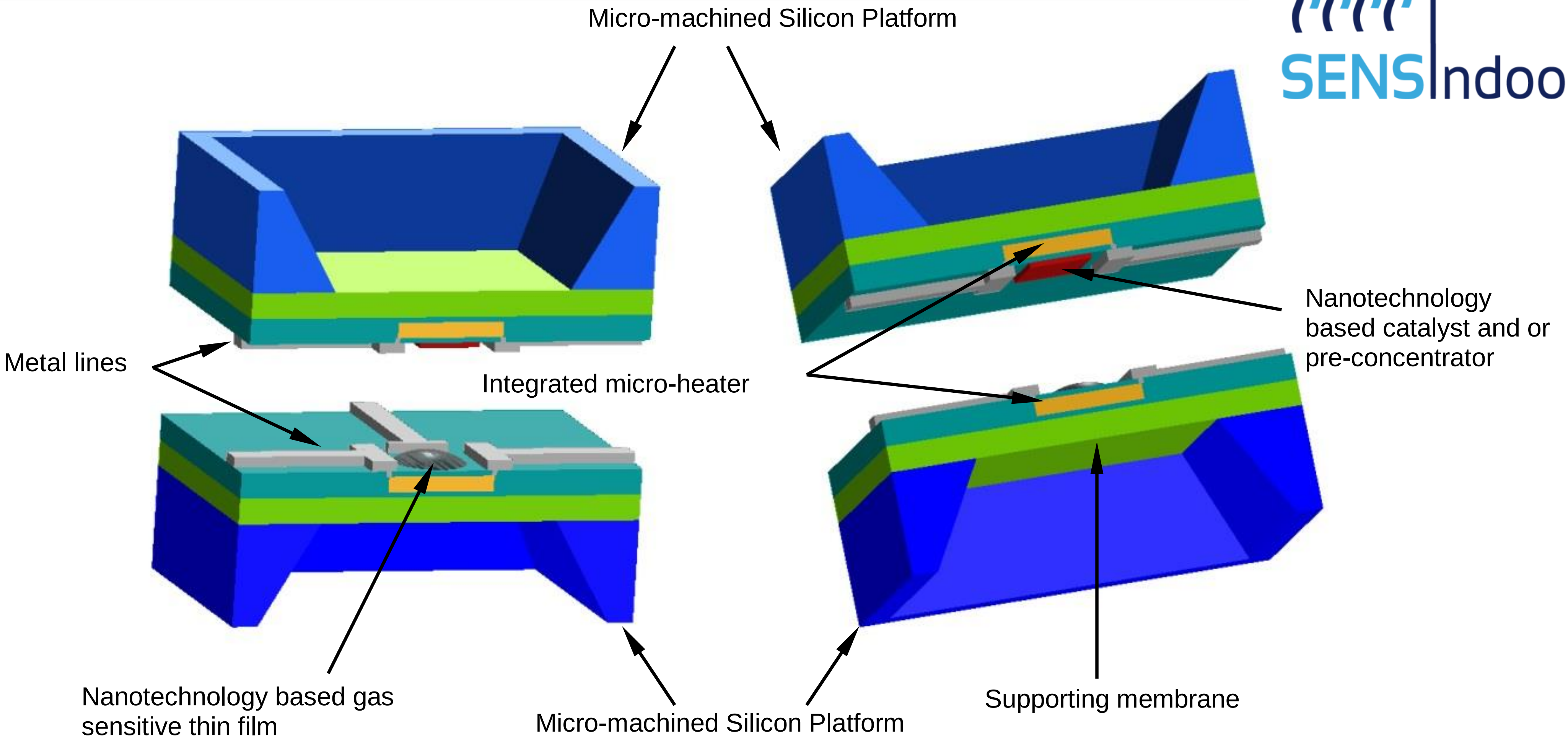


SENSIndoor

V. Kekkonen, J. Liimatainen, S. Chaudhuri, T. Sauerwald, A. Schütze: Engineered metal and metal oxide gas sensor layers by pulsed laser deposition technology, IEEE SENSORS 2015, Busan, Nov. 2015, accepted.

See also: EUROSENSORS XXIX, late news poster TP-N16

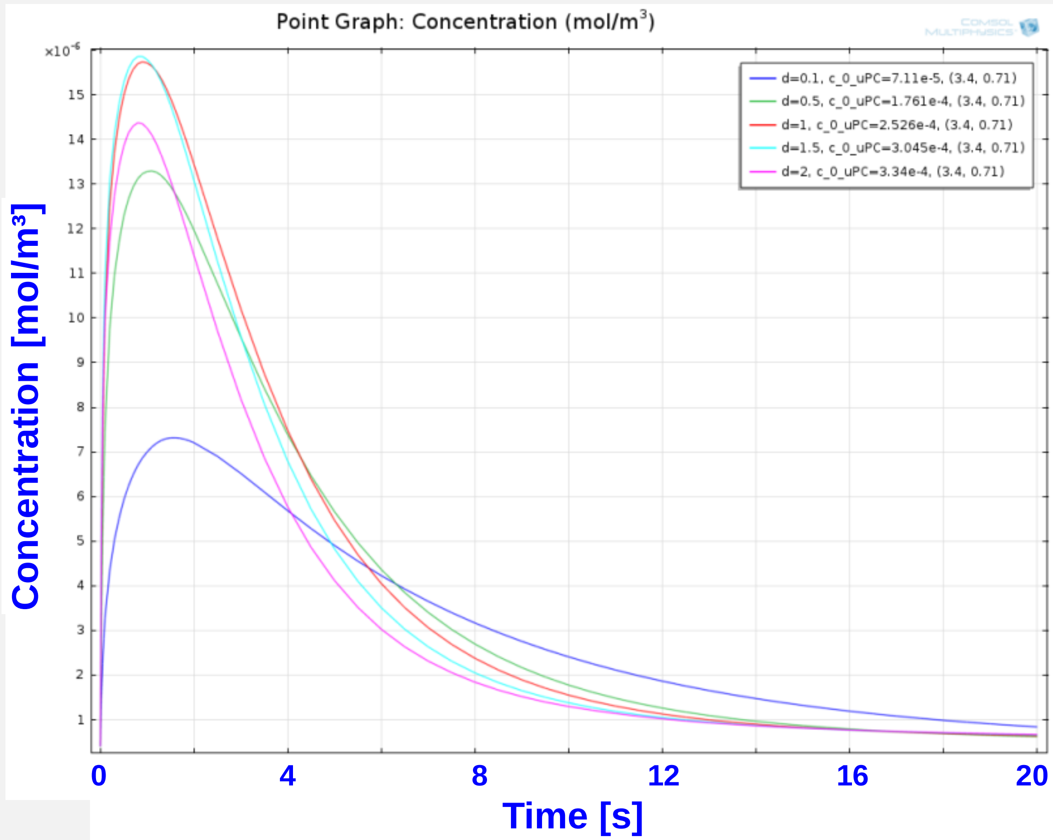
Miniaturization (concept): µ-hotplate for pre-concentrator



Simulations w Comsol Multiphysics
(Saarland University)

- Different configuration: side-by-side, face-to-face
- Strong increase in concentration achieved **(~ x50)**
- Short pulses **~ 1-4 sec**

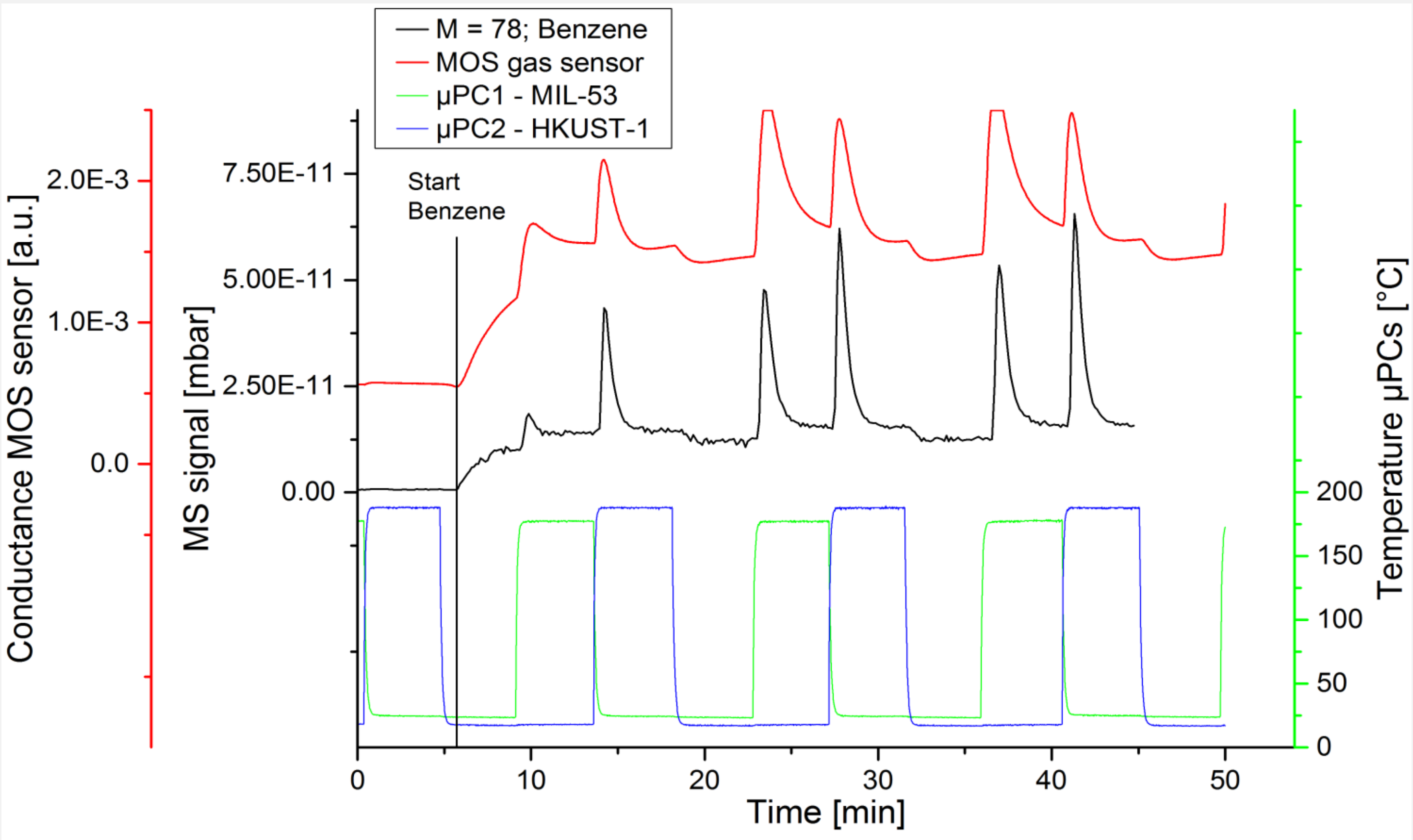
→ exact timing required for evaluation



A. Schütze, M. Leidinger, M. Rieger, B. Schmitt, T. Sauerwald: A novel low-cost pre-concentrator concept to boost sensitivity and selectivity of gas sensor systems, IEEE SENSORS 2015, Busan, Nov. 2015, accepted.

Experimental validation (MOF on ceramic heater)
(Saarland University and Fraunhofer ICT)

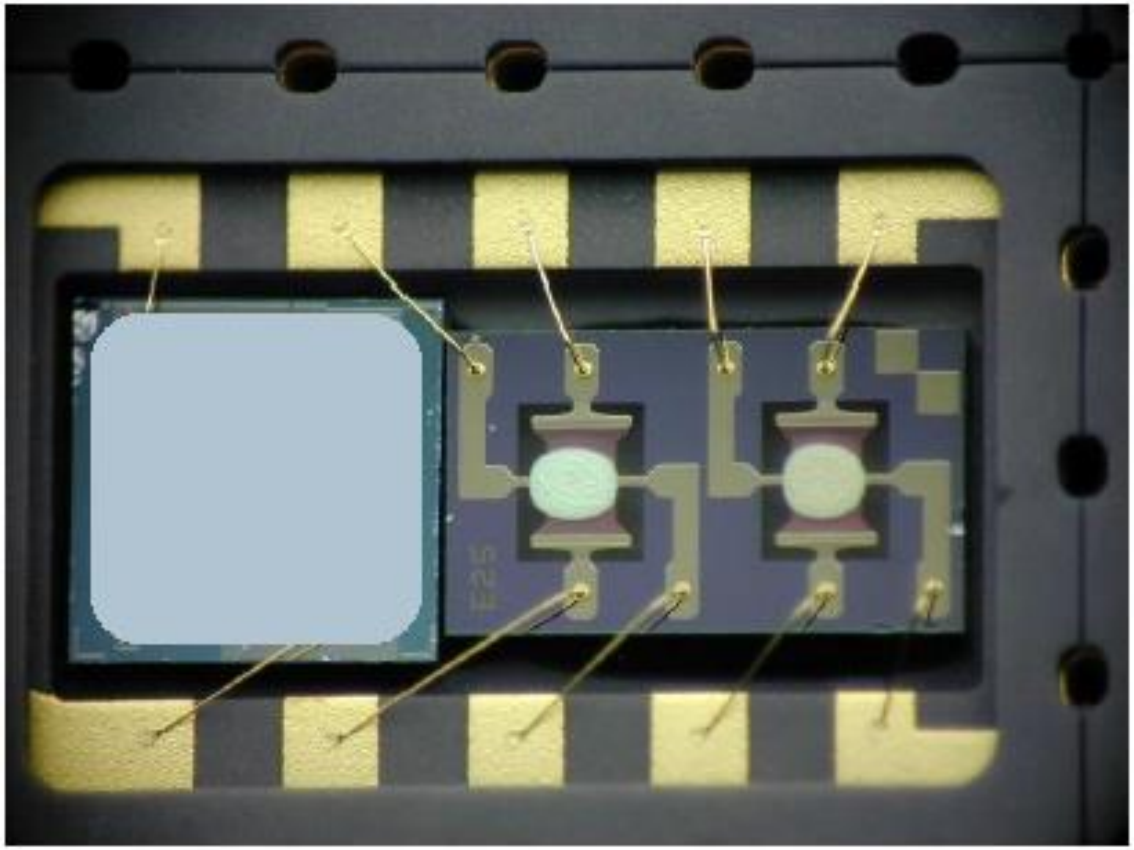
- Tested with gas sensor and mass spectrometer



M. Leidinger, M. Rieger, T. Sauerwald, M. Nägele, J. Hürttlen, A. Schütze: Trace gas VOC detection using metal-organic frame-works micro pre-concen-trators and semiconductor gas sensors, EUROSENSORS XXIX, Poster TP-E01

Target solution
(Integration: SGX)

- µ-pre-concentrator packaged with 2 MOS microsensors



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CONCLUSIONS

CONCLUSIONS:

- Indoor applications often underestimated
- Wide range of sensor technologies employed already today
- Sensor systems are more than just sensor elements

OUTLOOK:

- Indoor applications spectrum growing strongly
  - Indoor vs. outdoor air for optimum ventilation
  - Particle sensors for PM_{10}, $PM_{2.5}$ and UFP for indoor air
  - Sensors for air treatment systems
  - Fire detection with gas sensors
  - Mold detection (selective VOC detection? biochemical sensors?)
- Integration of ubiquitous sensors in smartphones

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Satellite event – Eurosensors XXIX, Freiburg, 09.09.2015

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Year 4: 1 July 2015 - 30 June 2016 (*Ongoing Action*)

Integrated sensor systems for indoor applications: ubiquitous monitoring for improved health, comfort and safety

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UNIVERSITÄT DES SAARLANDES

Lehrstuhl für Messtechnik

COST EUROPEAN COOPERATION IN SCIENCE AND TECHNOLOGY

EuNetAir
European Network on New Sensing Technologies for Air-Pollution Control and Environmental Sustainability

COST is supported by the EU Framework Programme

European Science Foundation — ESF provides the COST Office through a European Commission contract