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

COST Action TD1105

Fourth Scientific Meeting – Linköping, June 3 - 5, 2015

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

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

Plenary Session 2: Indoor Environment Quality Applications

FP7 SENSIndoor - Increasing Sensitivity and Selectivity of Gas Sensor-Systems by Using Micromachined Pre-Concentrators with MIP and MOF Layers

COST EUROPEAN COOPERATION IN SCIENCE AND TECHNOLOGY

UNIVERSITÄT DES SAARLANDES

Lehrstuhl für LMT Messtechnik

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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

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

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

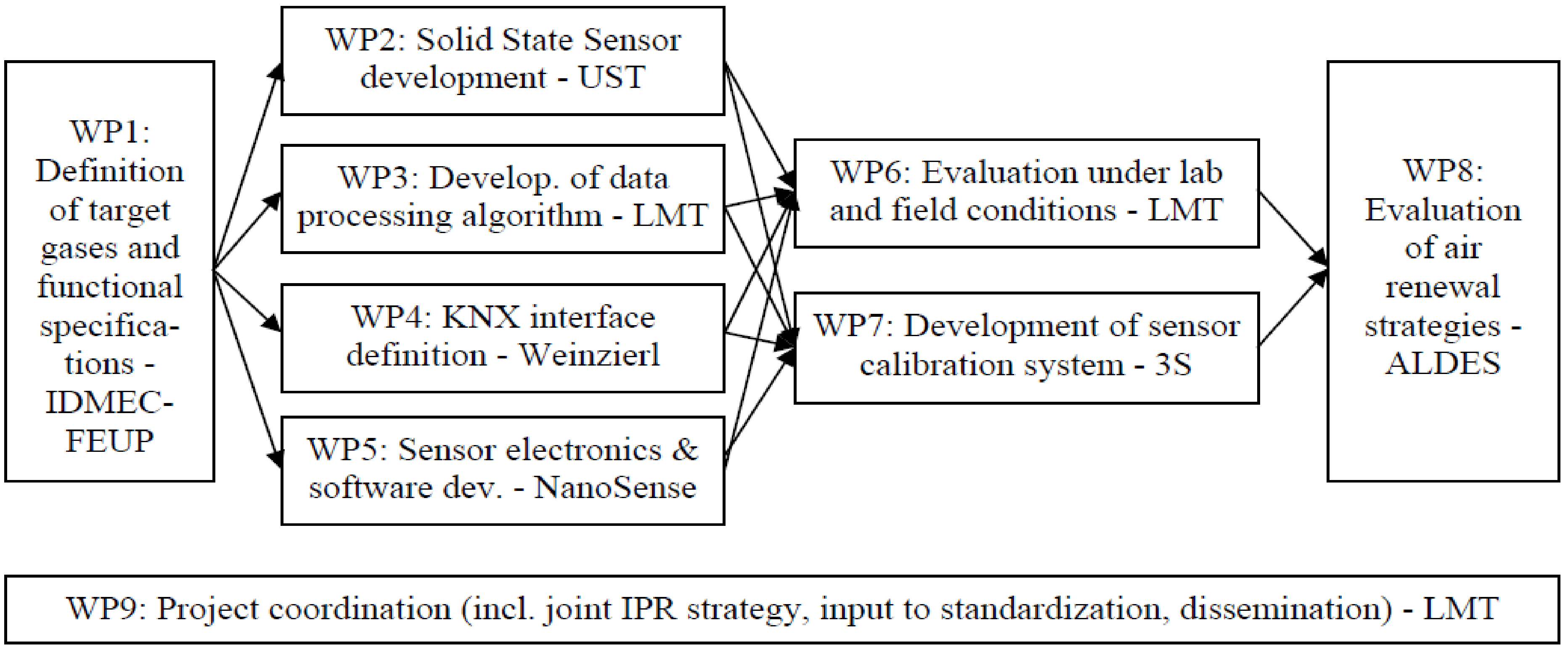
SENSIndoor: Nanotechnology based intelligent multi-SENsor System with selective pre-concentration for Indoor air quality control

- 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

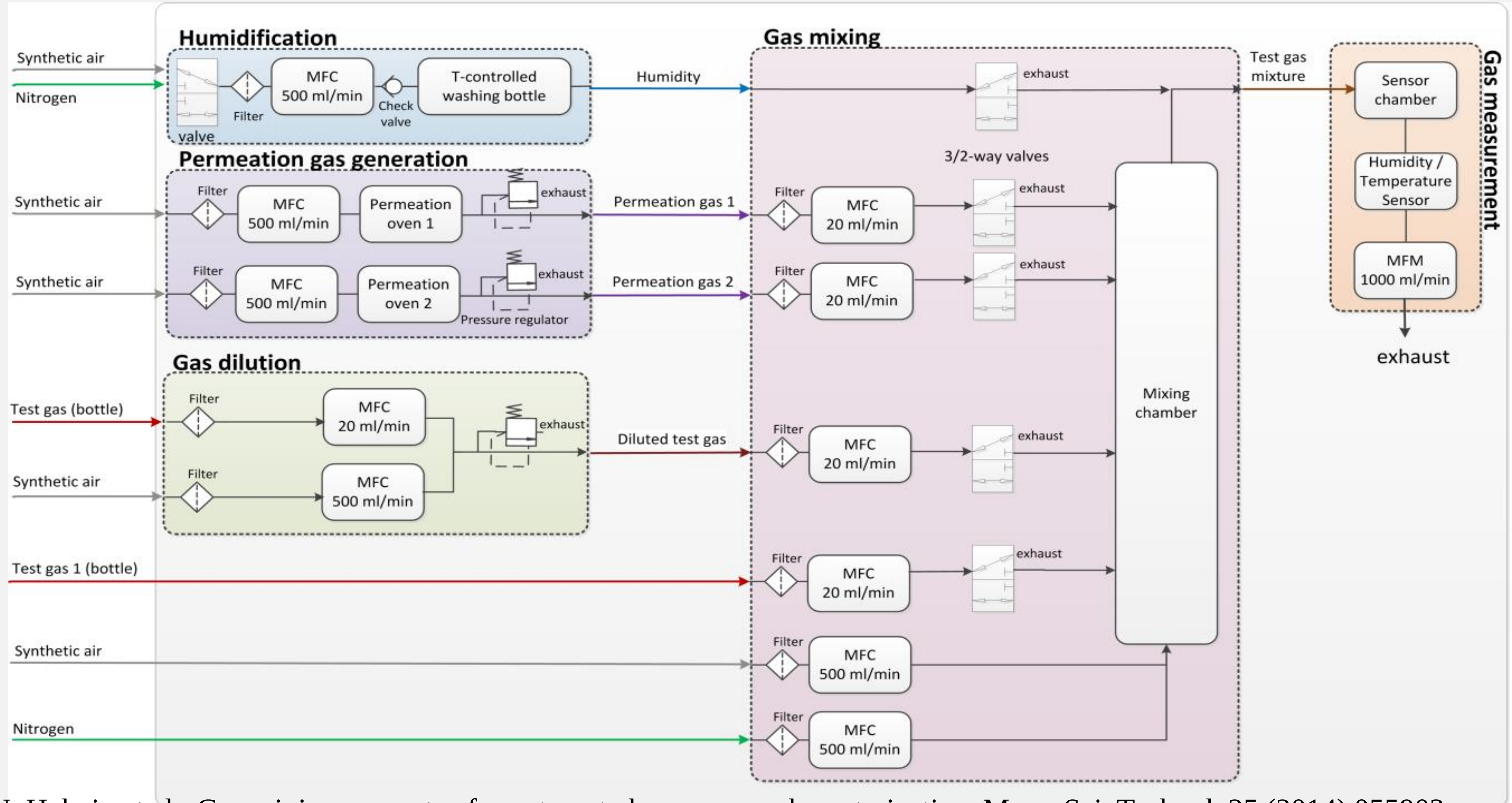
> Indoor Air Quality monitoring

MNT-ERA.net project VOC-IDS

- Volatile Organic Compound Indoor Discrimination Sensor
- Scenario specific detection of hazardous VOC
- Integration of sensor system into KNX building automation networks

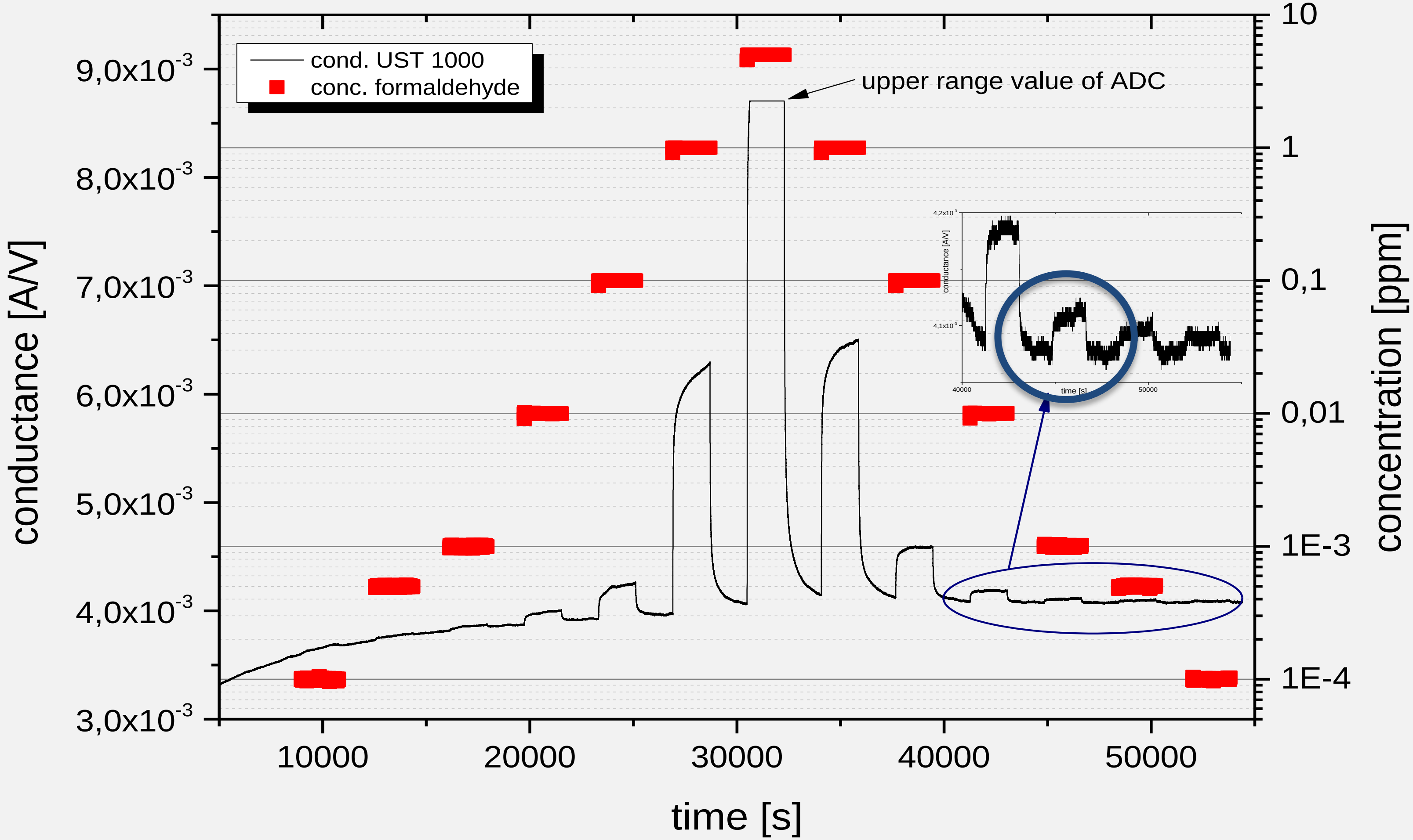


First step: novel gas mixing system for VOC testing/calibration @ (sub) ppb-level



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



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

Sensor reaction to 1 ppb formaldehyde

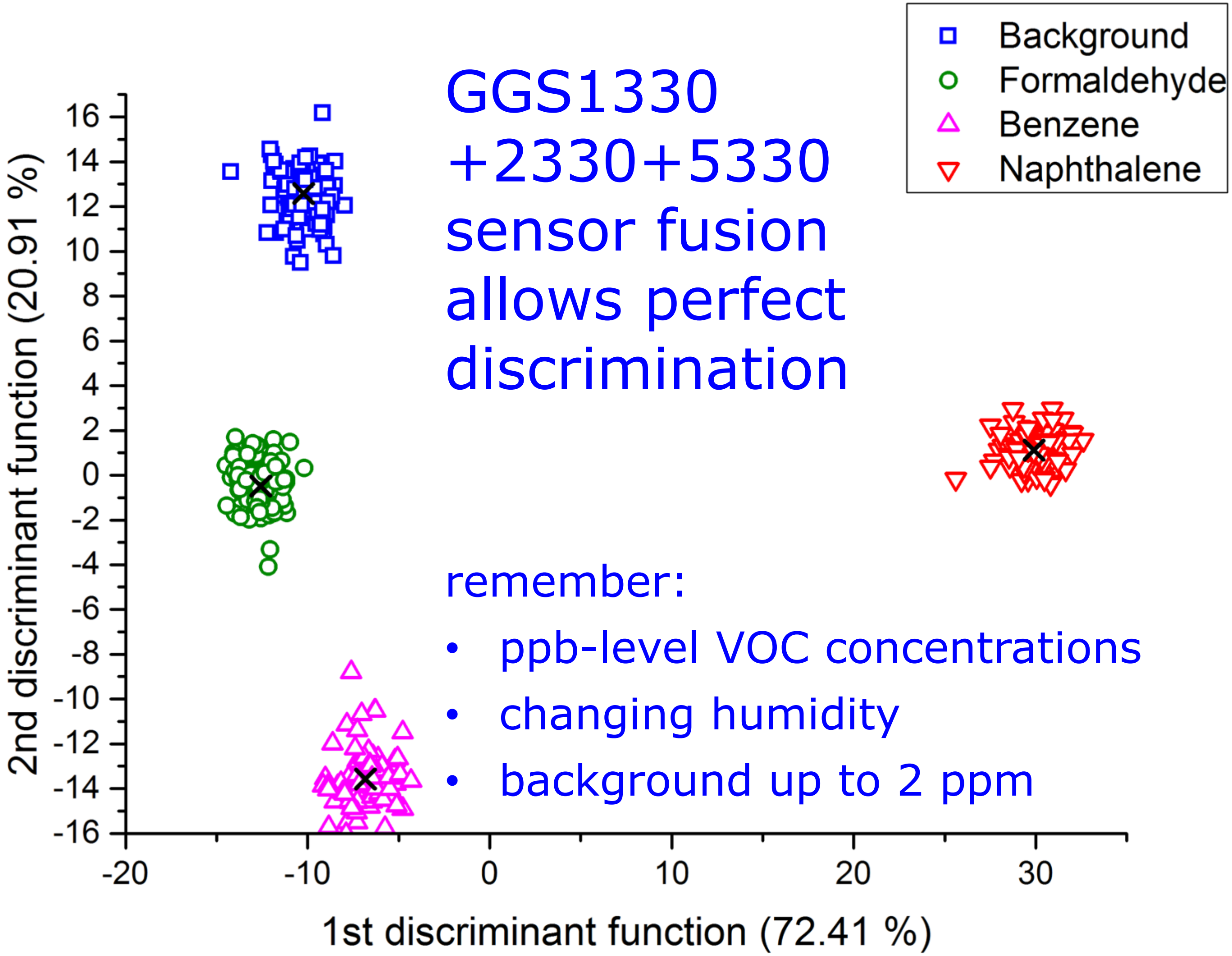
Relevance? Legal limits in France for indoor air: Formaldehyde 25 ppb in 2015; Benzene 0.6 ppb in 2016

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

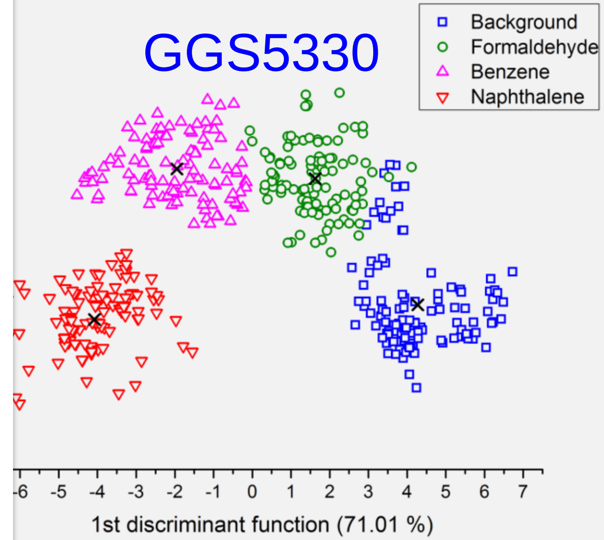


GGS1330 +2330+5330 sensor fusion allows perfect discrimination

remember:

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

GGS5330

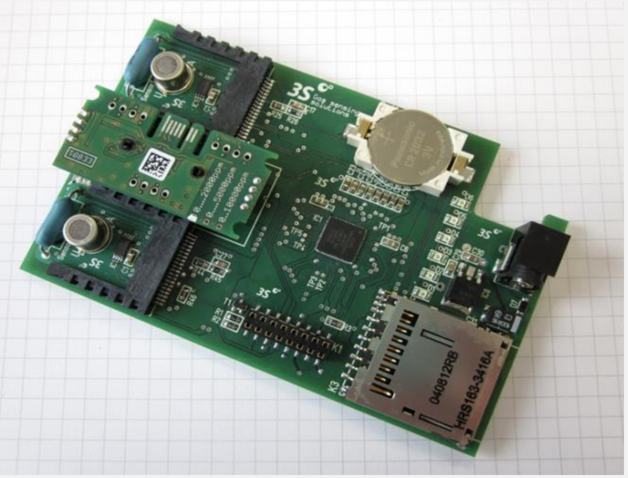


mnt-era.net

- 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!**

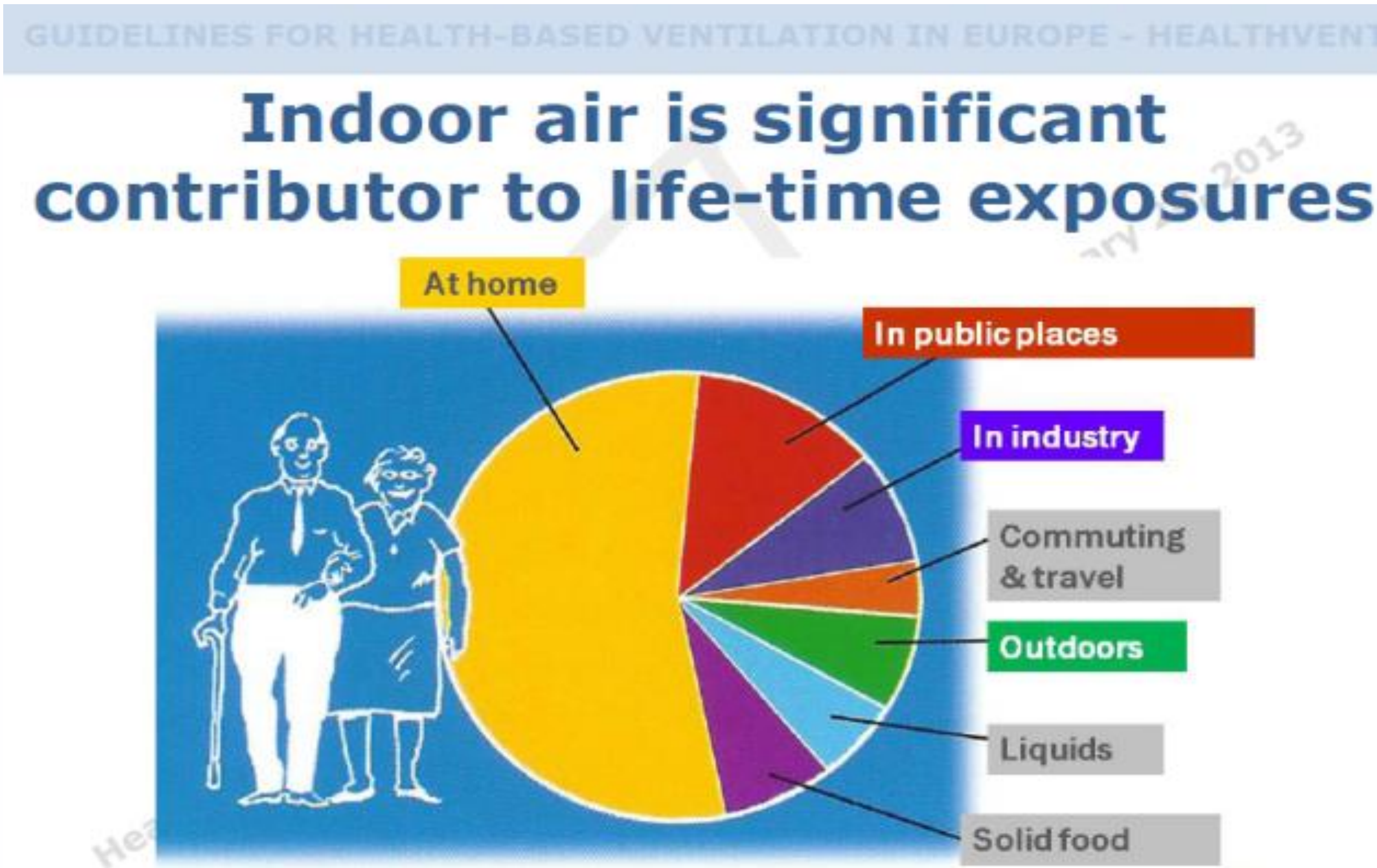
Setup for system calibration



© 3S GmbH, 2013

Motivation

- Core motivation for the SENSIndoor project

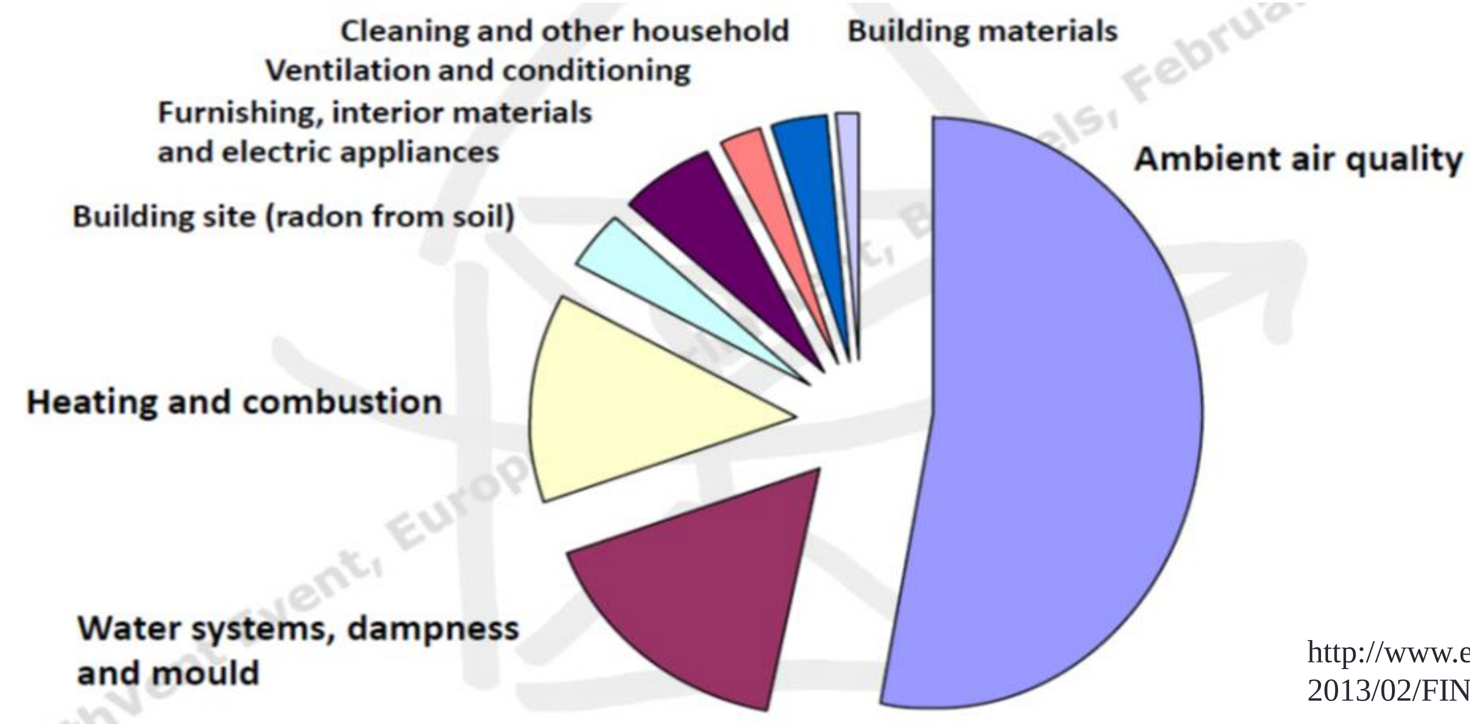


http://www.healthvent.byg.dtu.dk/

Motivation

➢ Core motivation for the SENSIndoor project

2 Mio healthy life years are lost every year in the EU due to indoor exposure according to an analysis in the EU project EnVIE



http://www.efanet.org/wp-content/uploads/2013/02/FINAL-Presentation-all.pdf

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**

SENSIndoor

Sensor system requirements 1

- Demand controlled ventilation to achieve optimal compromise between energy efficiency and health benefits adapted to specific application scenarios
  - ➢ **room-specific measurements required**
- Significant contribution to EU 20-20-20 goals and health aspects of IAQ targeted
  - ➢ **ubiquitous measurements required**

➢ **Core challenges addressed with micro- and nanotechnologies**

- ➢ **microtechnologies for low cost mass production (and low power consumption)**
- ➢ **nanotechnologies for unrivalled sensitivity and selectivity**

Sensor system requirements 2

- Detection of hazardous indoor air pollutants at relevant levels
  - key target pollutants are VOCs, i.e. formaldehyde, benzene,…
  - target concentrations are ppb and sub-ppb level

➢ **extremely high sensitivity required**

	2012							2013			2015			2016			2023		
	MAK work place (8 hours)		TRK work place (15 mn)		Domestic (MAK/3) 24h/24h			French decree n° 2011-1727 of 2/12/2011 long-term guide value for public buildings											
	ppm	mg/m3	ppm	mg/m3	ppm	ppb	mg/m3	ppm	ppb	µg/m3	ppm	ppb	µg/m3	ppm	ppb	µg/m3	ppm	ppb	µg/m3
Formaldehyde	0.5	0.615	1	1.23	0.17	166.67	0.21				0.024	24.39	30.00				0.008	8.13	10.00
Benzene	1	3.25	1	3.25	0.33	333.33	1.08	0.00154	1.54	5.00				0.00062	0.615	2.00			

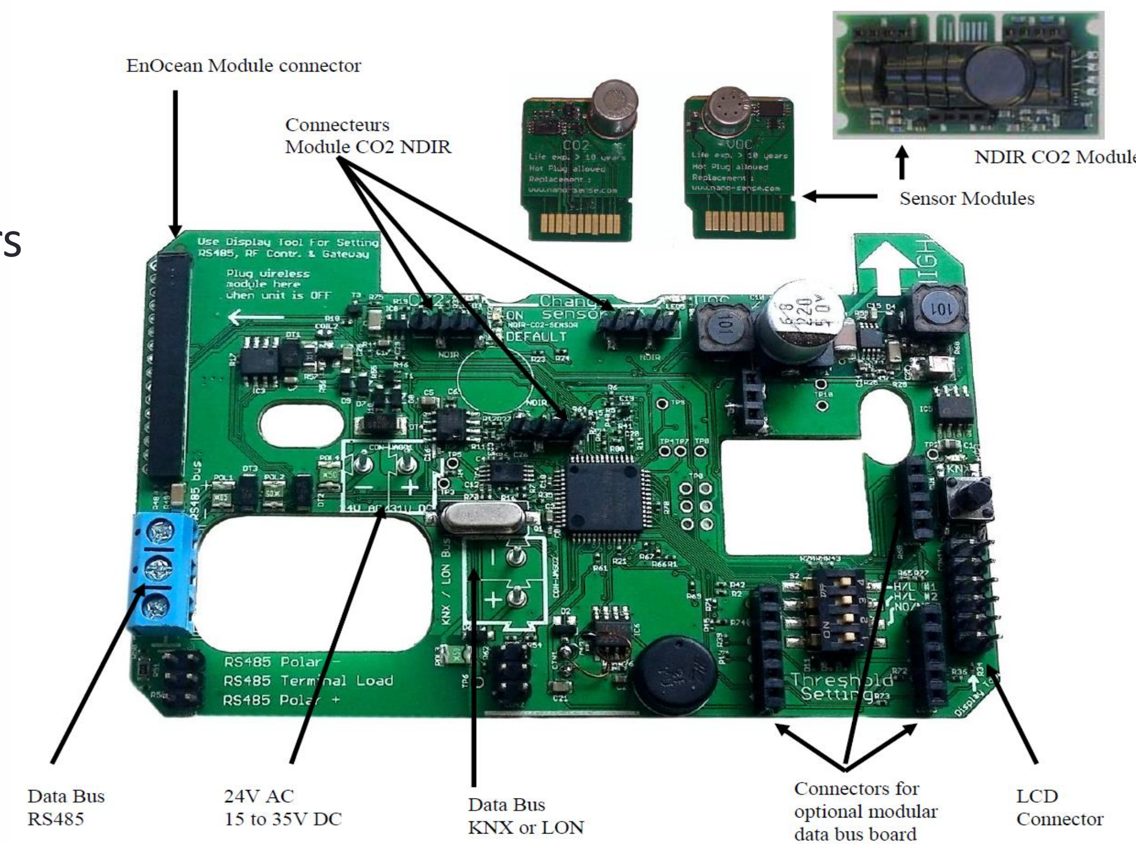
- In addition: many other interfering gases/VOCs
  - benign, e.g. ethanol, air freshener (much higher concentrations!)
  - unpleasant, but not hazardous, e.g. isovaleric acid
  - other background gases, e.g. CO, O_3, NO_x, ammonia, …

➢ **extremely high selectivity required**

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)



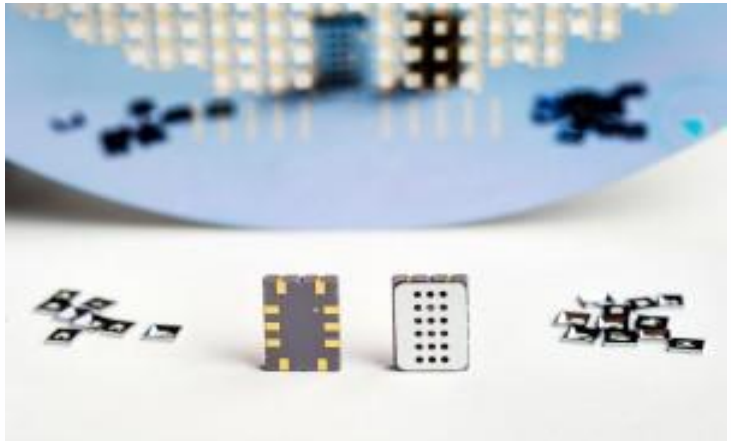
SENSIndoor

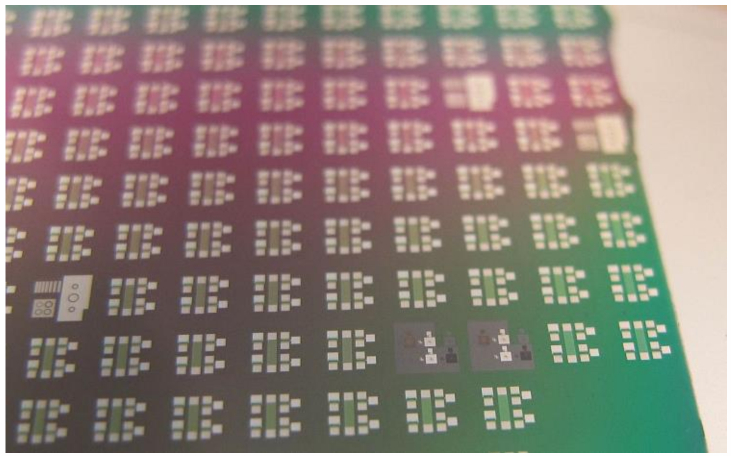
SENSIndoor origins

- **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

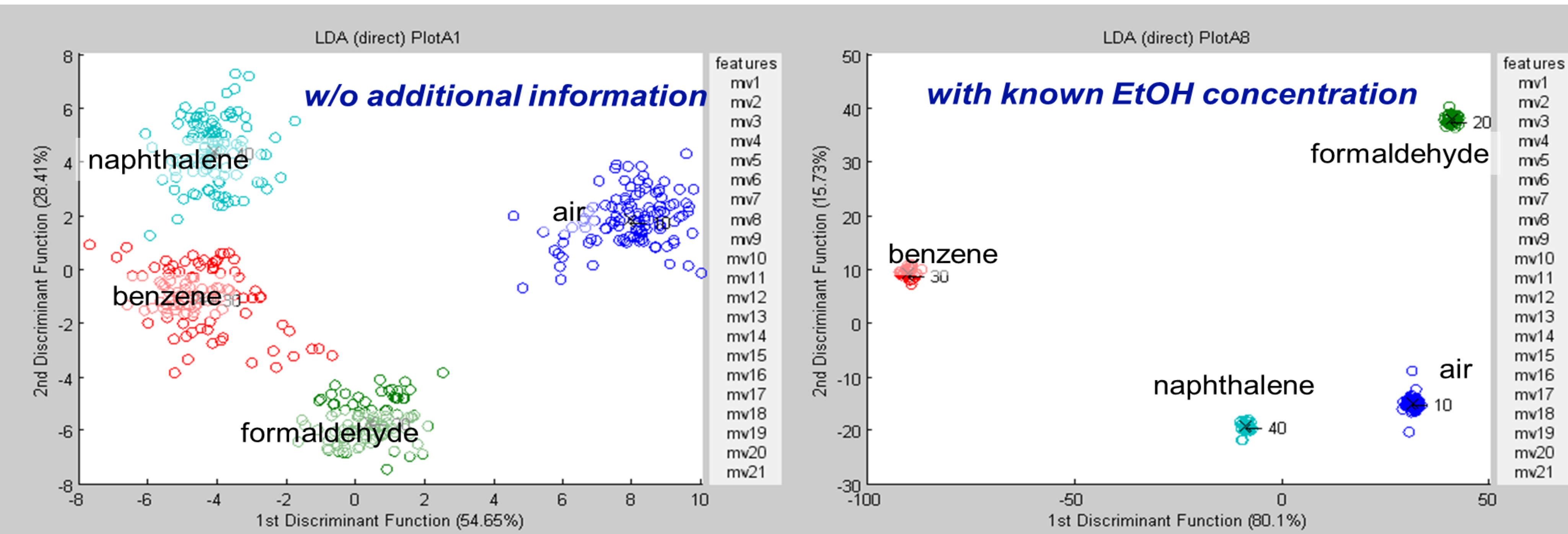
- Sensor technologies
  - **MOS – Metal oxide semiconductor** (*SGX Sensortech, USAAR-LMT*)
    - well known for high sensitivity and robustness @ low-cost
    - MEMS technology for mass production and low power consumption
  - **GasFET – Gas-sensitive Field Effect Transistors** (*LiU, SenSiC*)
    -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





SENSIndoor technologies 2

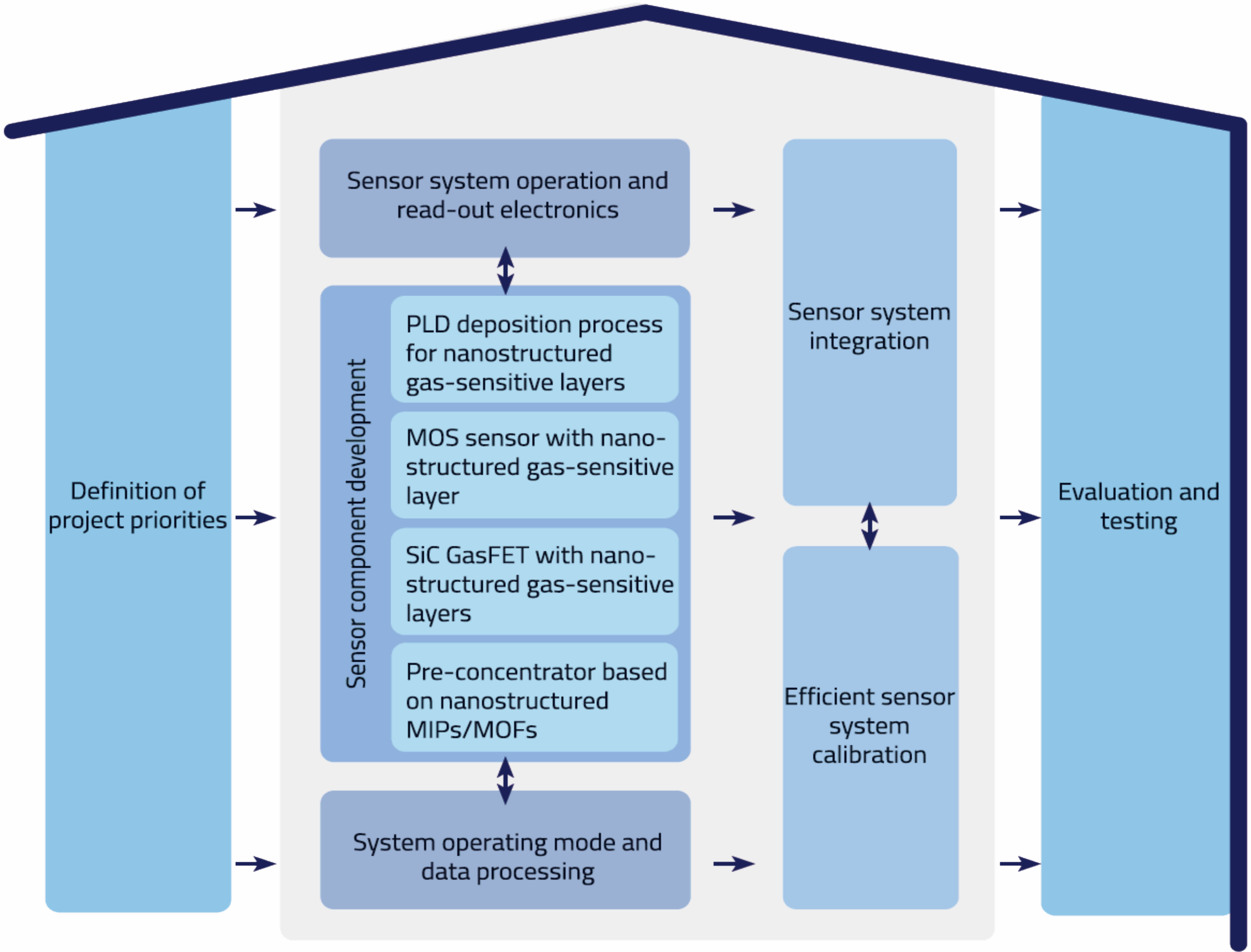
- Dynamic operation and intelligent signal processing
  - **Temperature Cycled Operation** (*USAAR-LMT, NanoSense, 3S*) to increase selectivity ("virtual multisensor") and stability



SENSIndoor overview

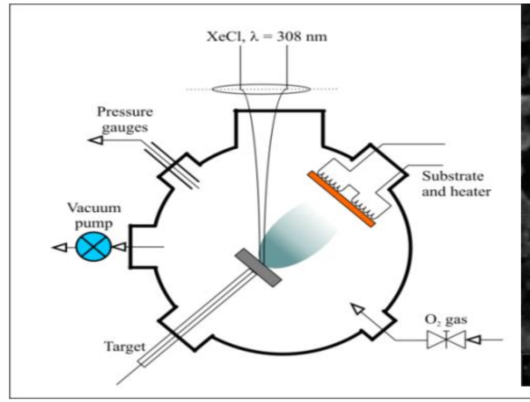
Project structure:

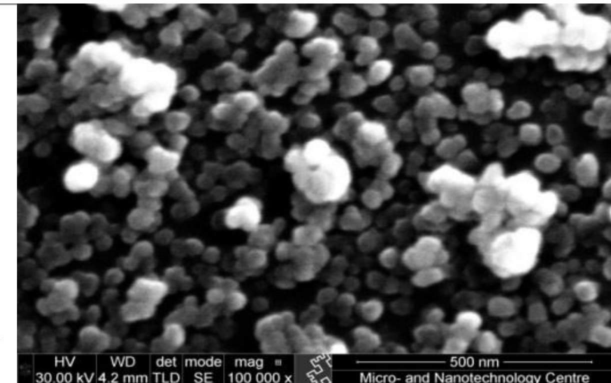
A clear road from application requirements to field evaluation



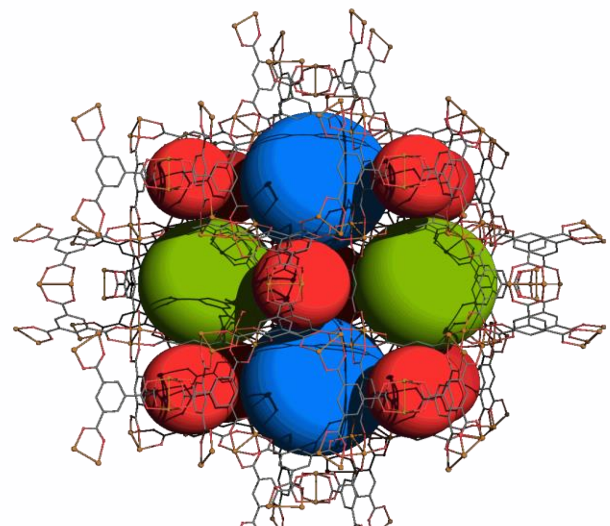
SENSIndoor technologies 3

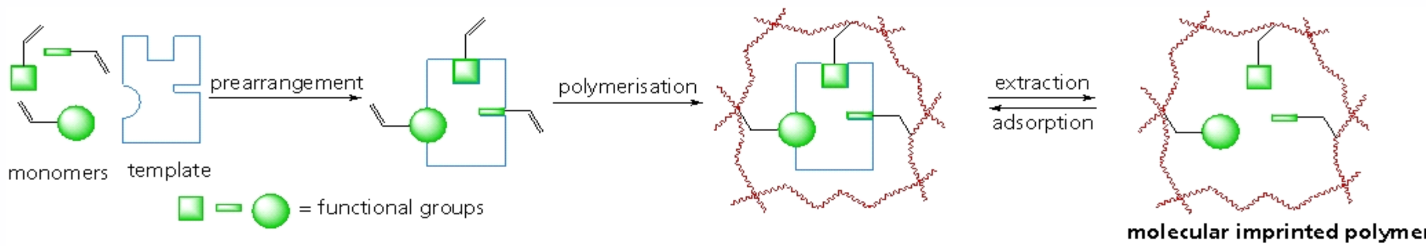
- Nanotechnology for improved sensor elements
  - **Pulsed Laser Deposition** *(U Oulu, Picodeon)* for novel, highly sensitive gas-sensitive layers suitable for wafer level mass production





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

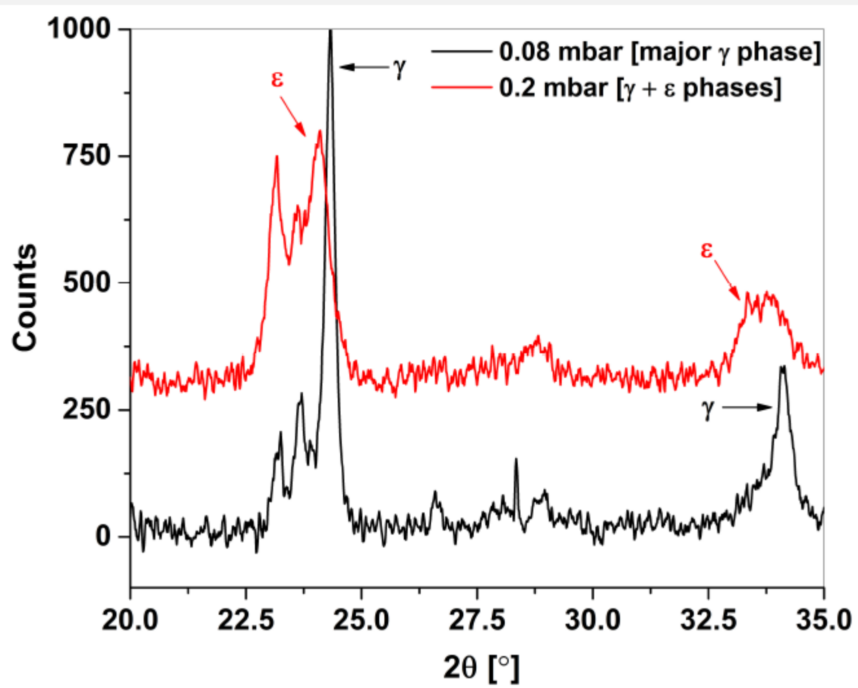


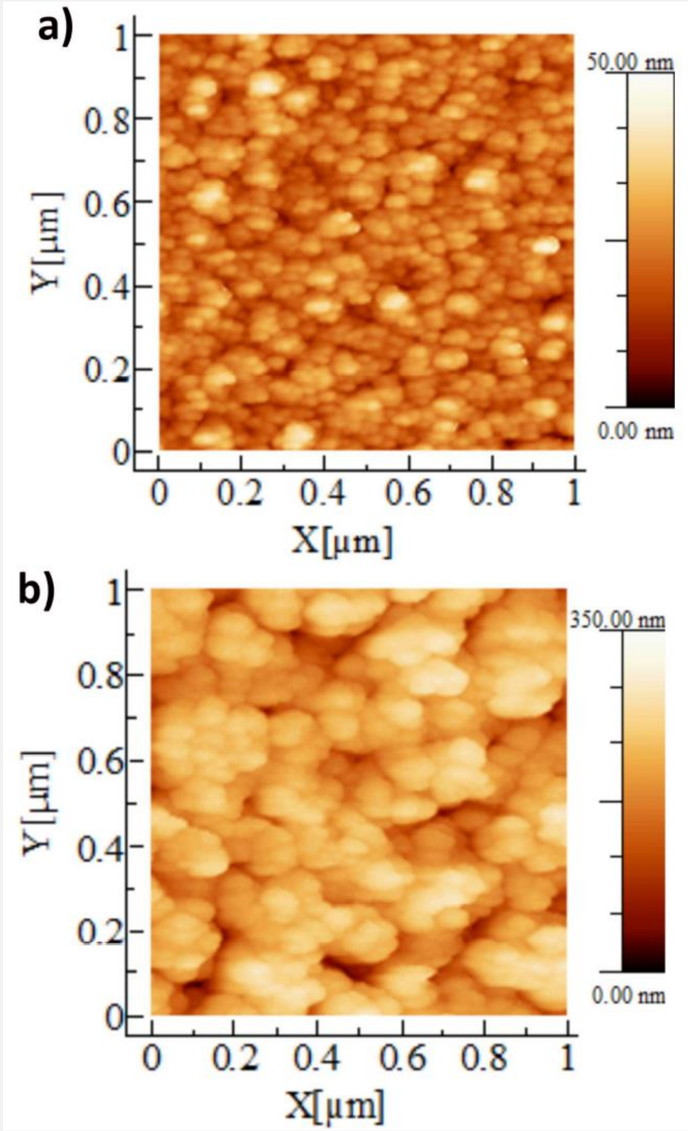


> PLD for well controlled sensing layers

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

- Characterized w AFM → and XRD

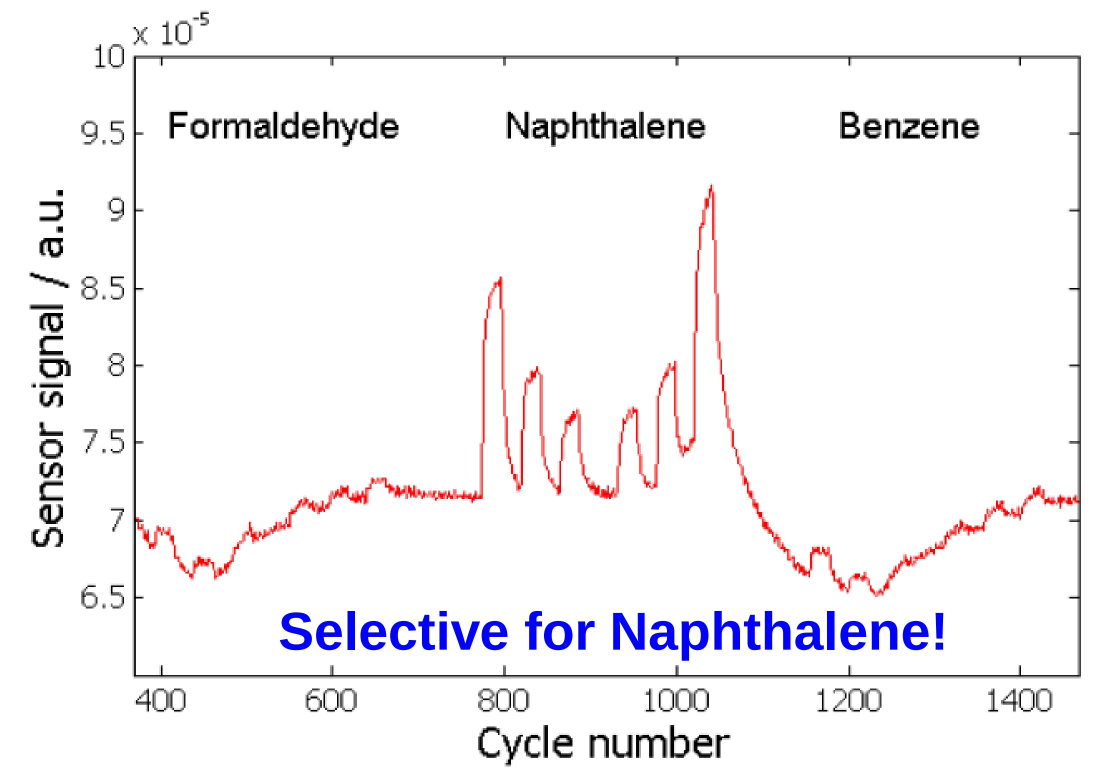


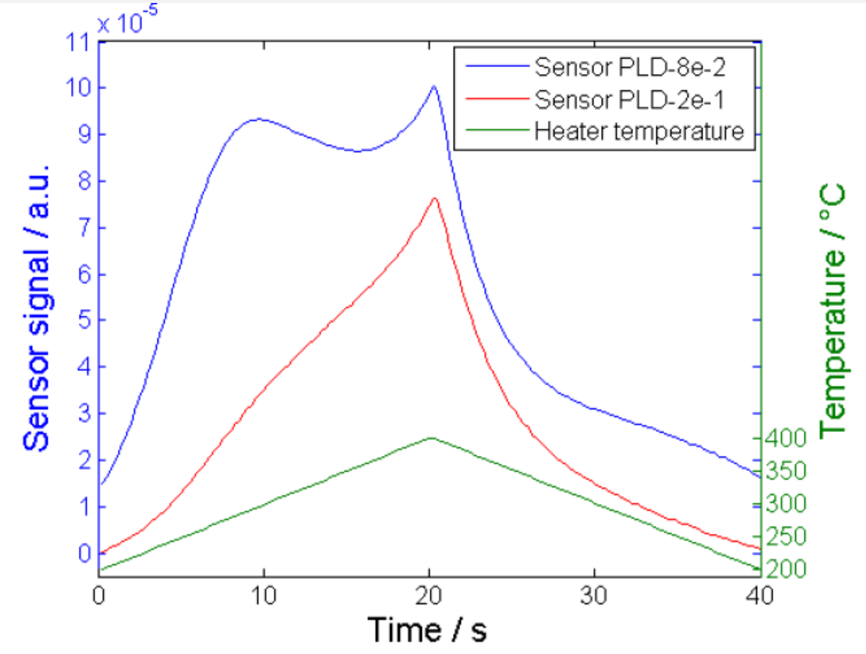


M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze: Nanostructured WO_3 Semiconductor Gas Sensor for Selective Detection of Naphthalene, SENSOR 2015, Nuremberg, May 19 – 21, 2015.

Testing with ppb level VOCs
(Saarland University)

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





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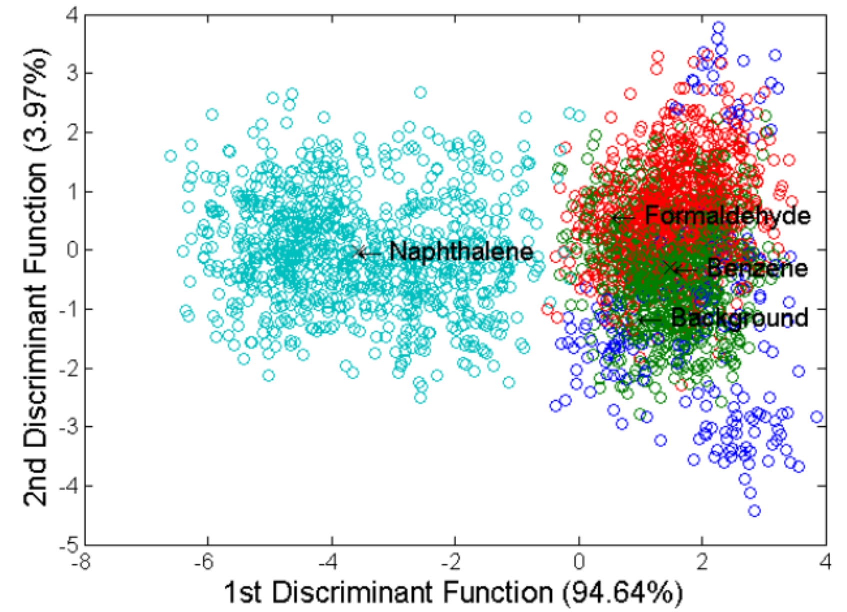
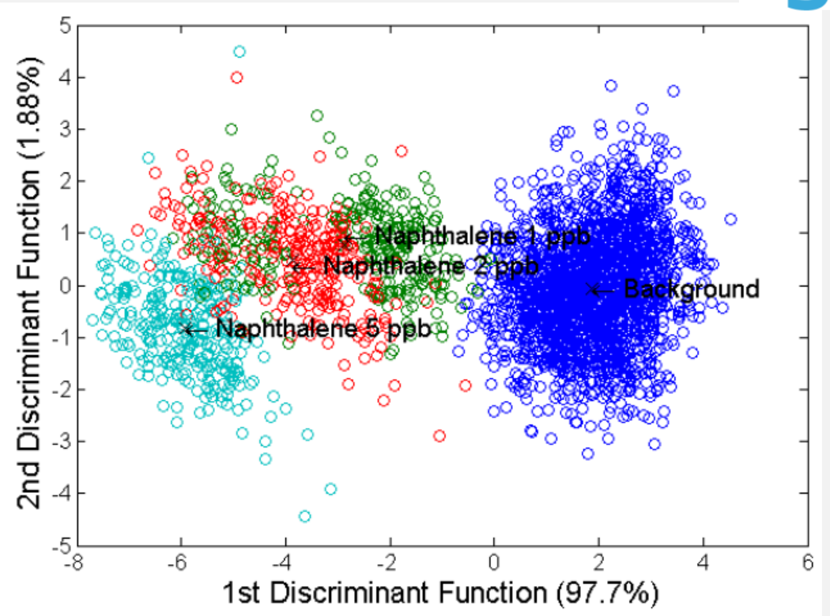
Identification and Quantification using LDA

(Saarland University)

- Sensor deposited at 0.2 mbar O_2 pressure





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

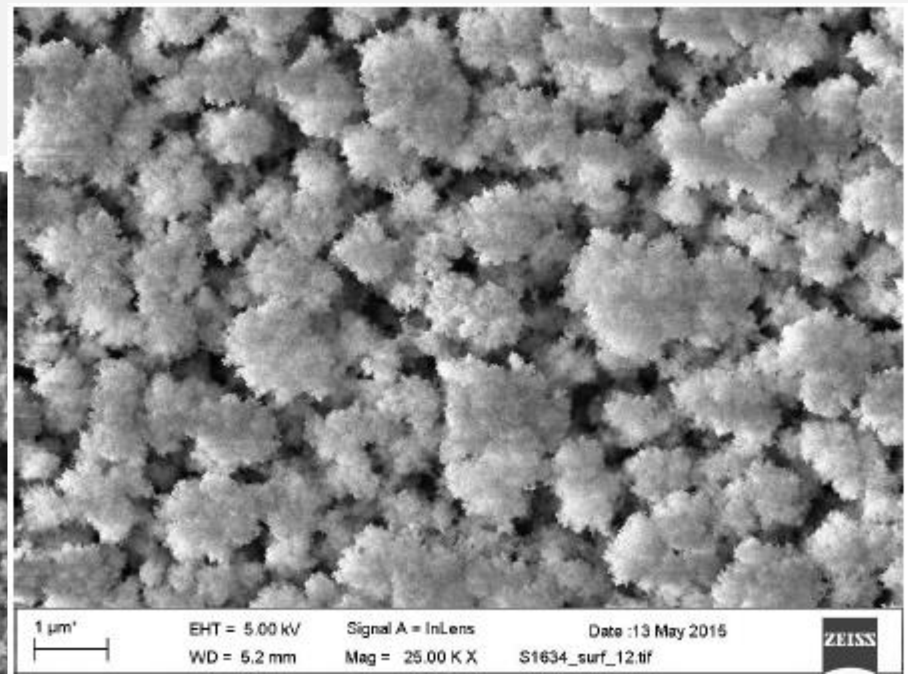
Note: data include **changing r.h.** and **ethanol** (up to 1 ppm)

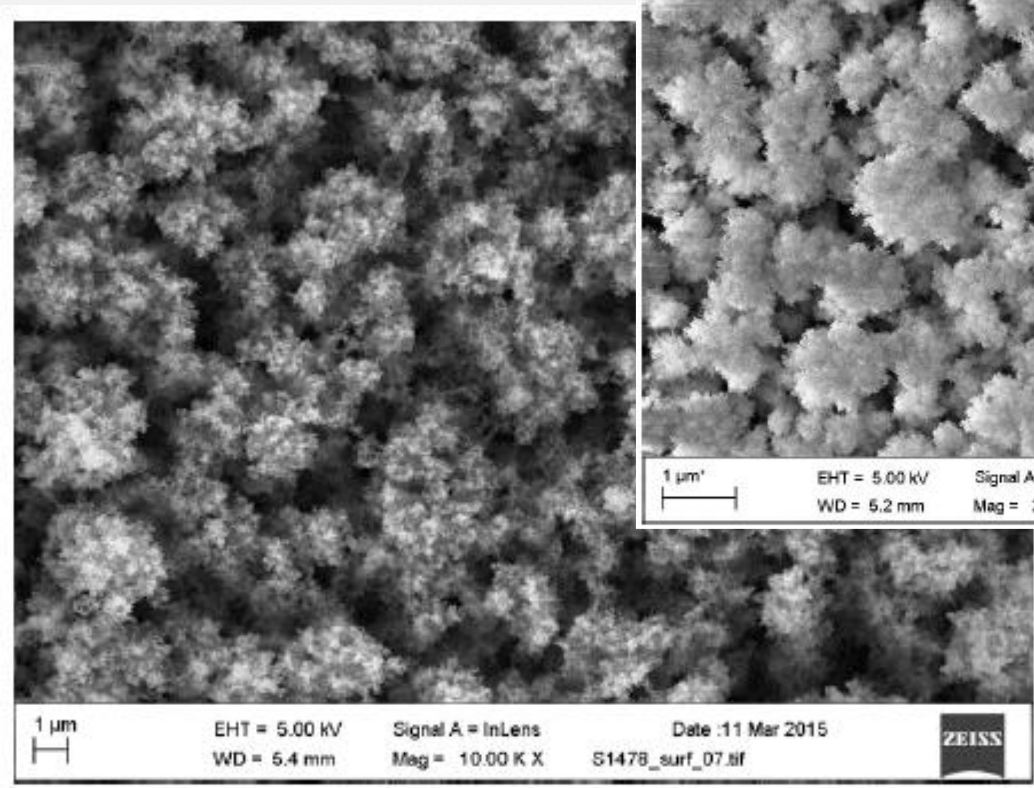
M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze: Nanostructured WO_3 Semiconductor Gas Sensor for Selective Detection of Naphthalene, SENSOR 2015, Nuremberg, May 19 – 21, 2015.

Next generation: SnO_2 and WO_3 w catalyst layers

(Picodeon w ps laser)

- SnO_2 →
- WO_3

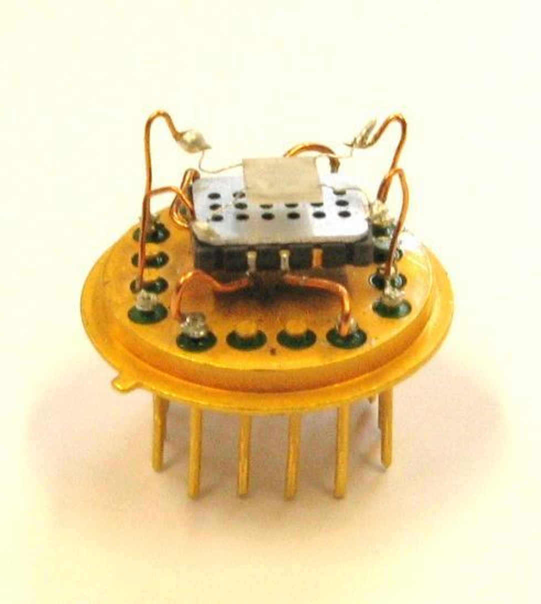


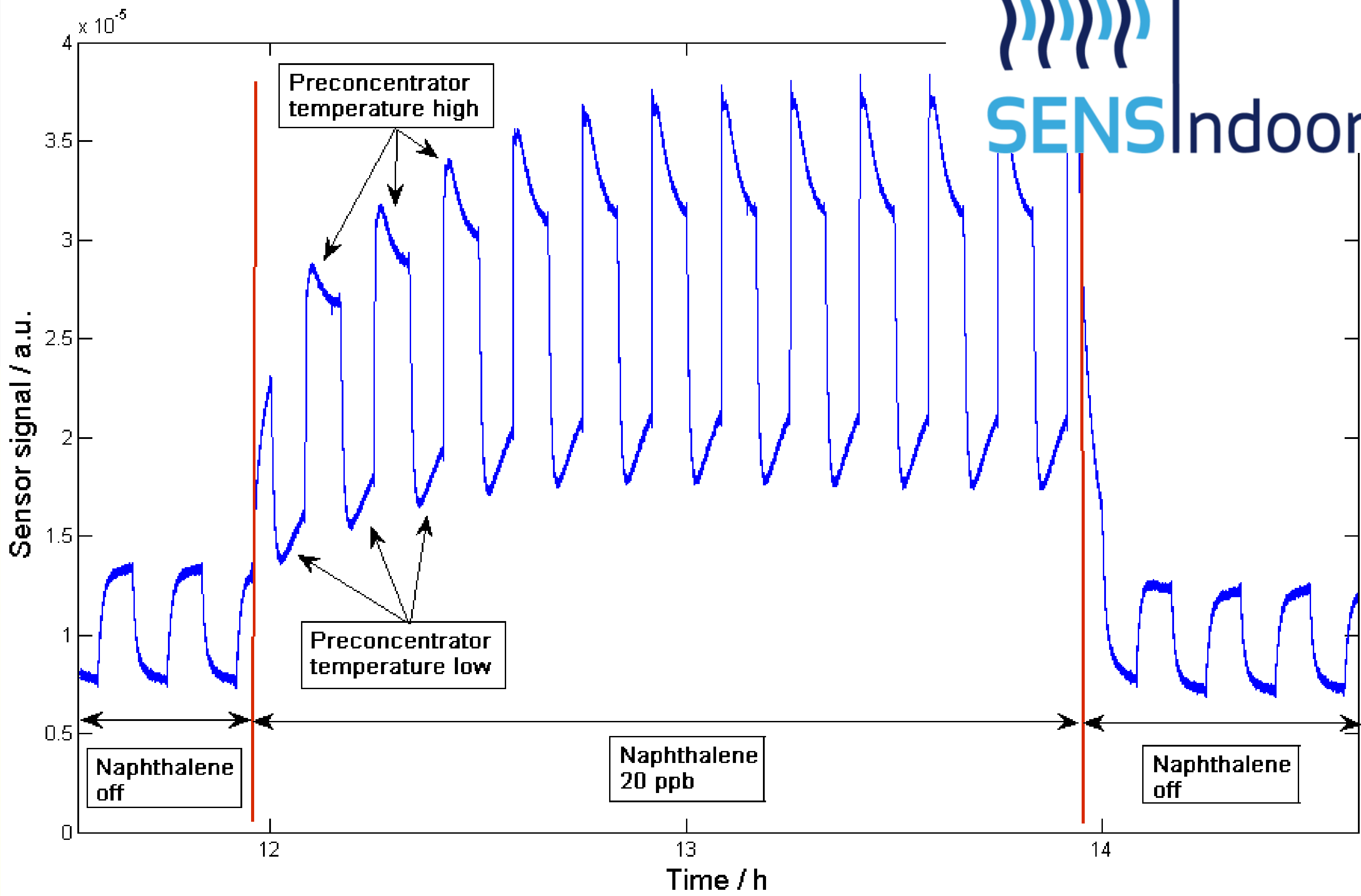


V. Kekkonen, J. Liimatainen, S. Chaudhuri, T. Sauerwald, A. Schütze: Engineered metal and metal oxide gas sensor layers by pulsed laser deposition technology, IEEE SENSORS 2015, Busan, Nov. 2015, submitted.

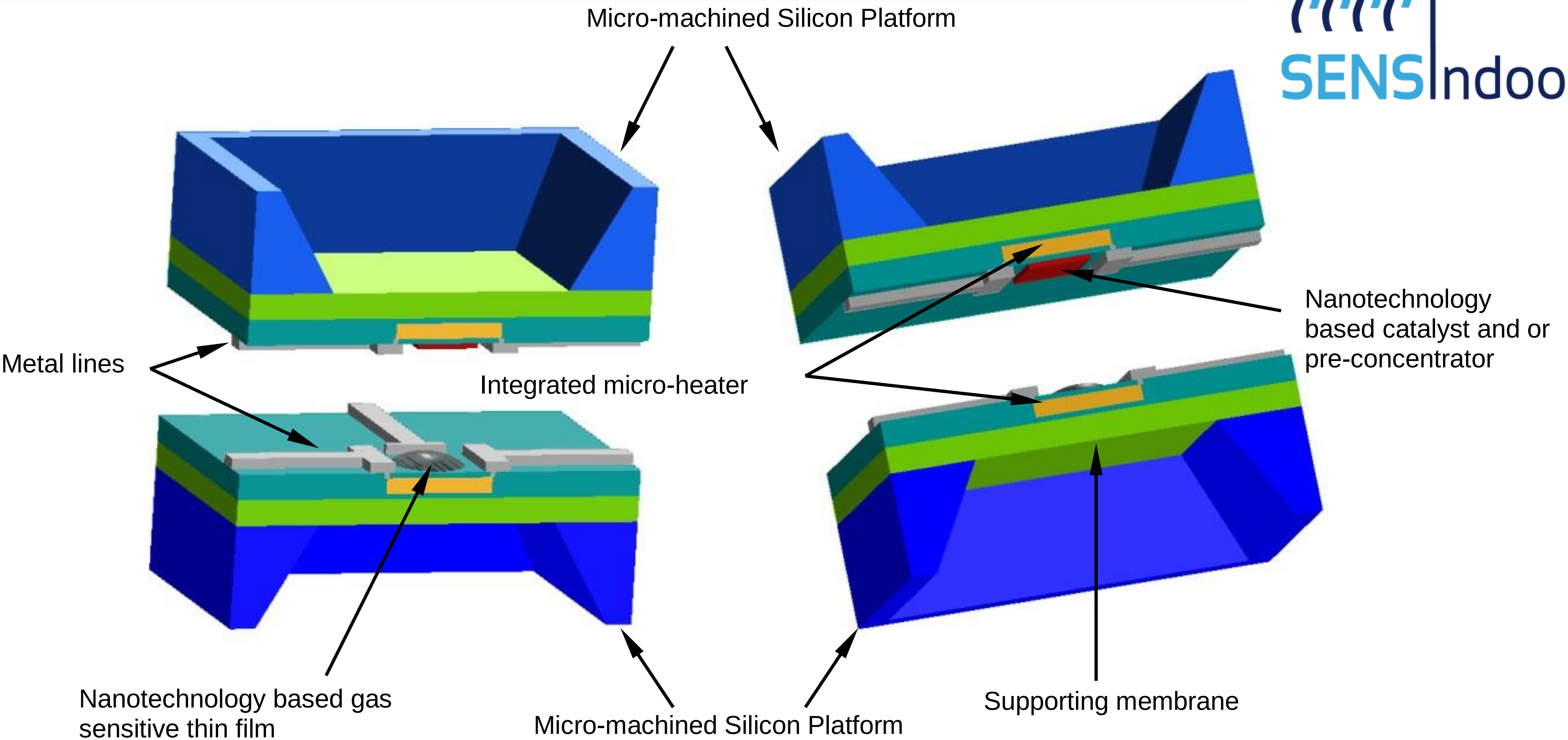
First proof-of-principle measurements (end 2014)

- preconc. off: signal low, then increases
- preconc. on: signal high, then decreases



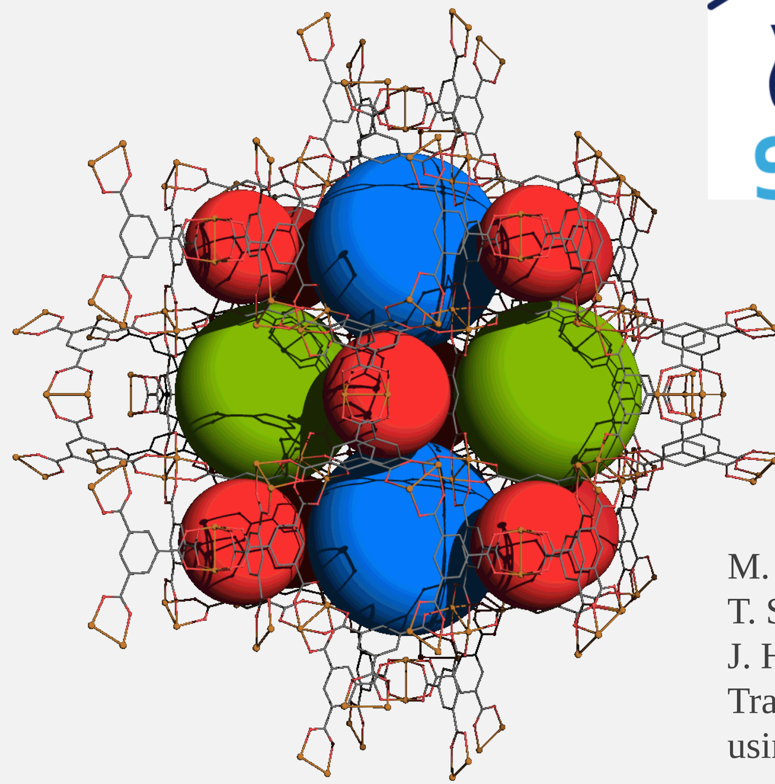


Miniaturization (concept): µ-hotplate for pre-concentrator

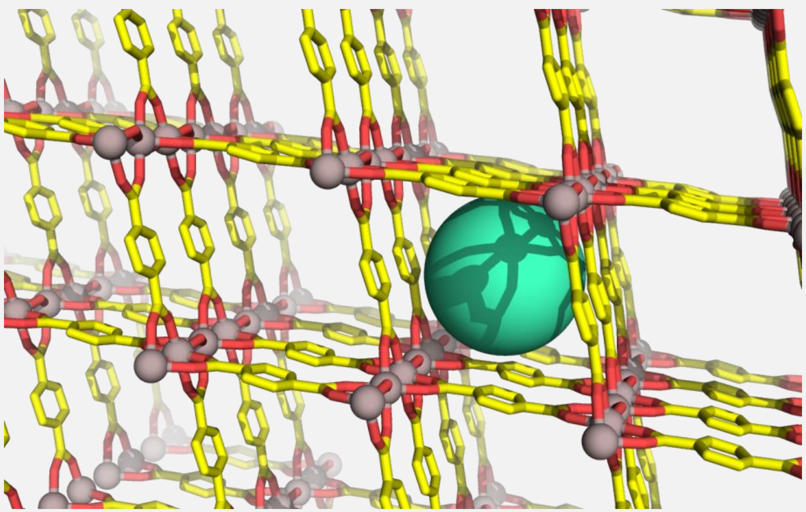


MOF layers deposited
(Fraunhofer ICT)

HKUST-1



MIL-53



M. Leidinger, M. Rieger, T. Sauerwald, M. Nägele, J. Hürttlen, A. Schütze: Trace gas VOC detection using metal-organic frame-works micro pre-concen-trators and semiconductor gas sensors, EUROSENSORS 2015, Freiburg, Sep.. 2015, submitted.

CONCLUSIONS

- **CONCLUSIONS:**
- Both MOS and GasFET sensors highly sensitive for VOC
- TCO allowing discrimination and quantification
- Micro- and nanotechnologies for excellent functionality at low cost
- µ-pre-concentrator: promising concept for IAQ applications
- Ubiquitous low-cost sensor systems for IAQ realistic

- **OUTLOOK:**
- Extensive field testing required: calibration and reliable operation
- Priority application scenarios: schools/kindergarten, refurb. homes

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