Special session: **Chemical Sensors and New Technologies for Air-Pollution Control**
COST Action TD1105 – EuNetAir

Chemical Sensors for Indoor Applications

IMCS, May 20 – 23, 2012, Nuremberg

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

Outline

- Introduction: indoor applications
- Gas measurement systems – more than just sensors
  - The three “S”
  - Gas measurement systems
  - Signal processing and evaluation
  - Calibration and field testing
- Application examples
  - Fire detection
  - Indoor air quality
- Novel developments
  - Novel sensors: versatile GasFETs
  - System self monitoring
  - Multifunctional multisensor systems
- Conclusions

Introduction: indoor applications

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
  - 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

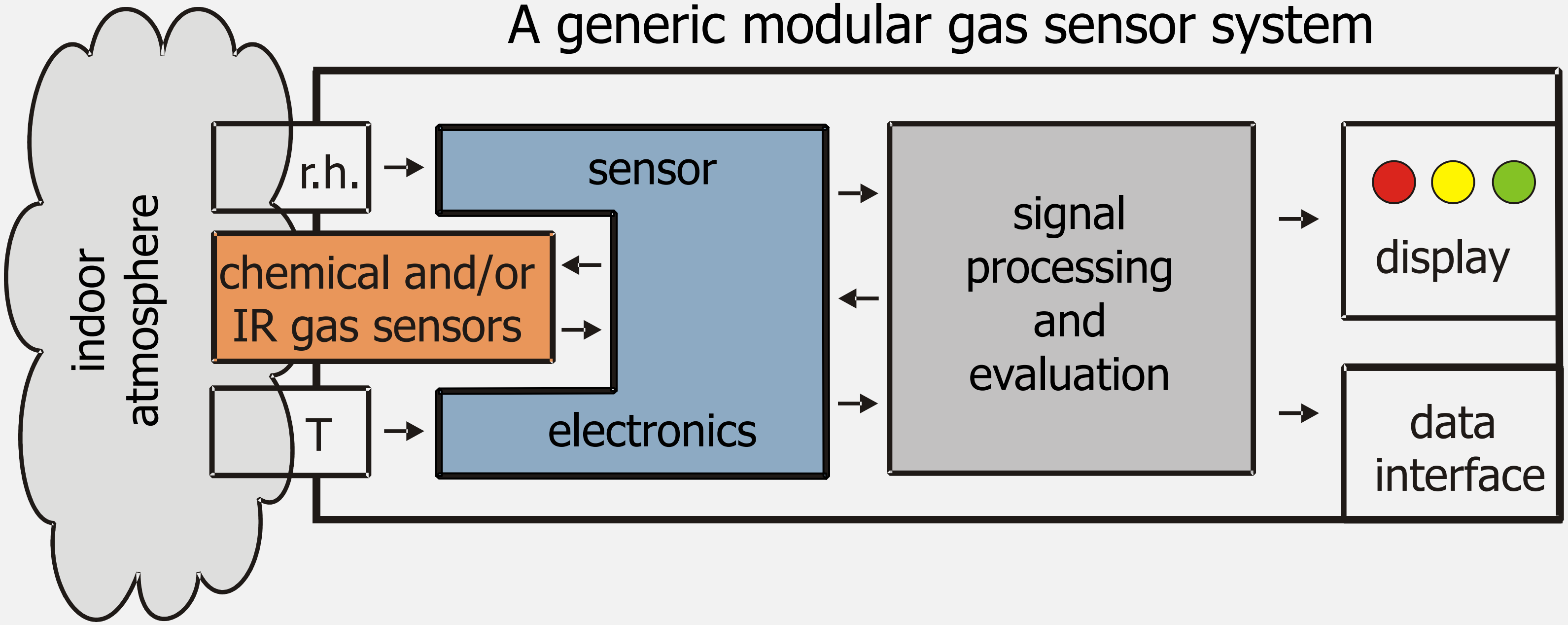
Which sensors are used today?

- Safety
  - Gas leak detection: pellistors (ind.), human nose (in Japan: MOS)
  - 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
  - VOCs: MOS (total VOC), GasFET (emerging)

The three "S"

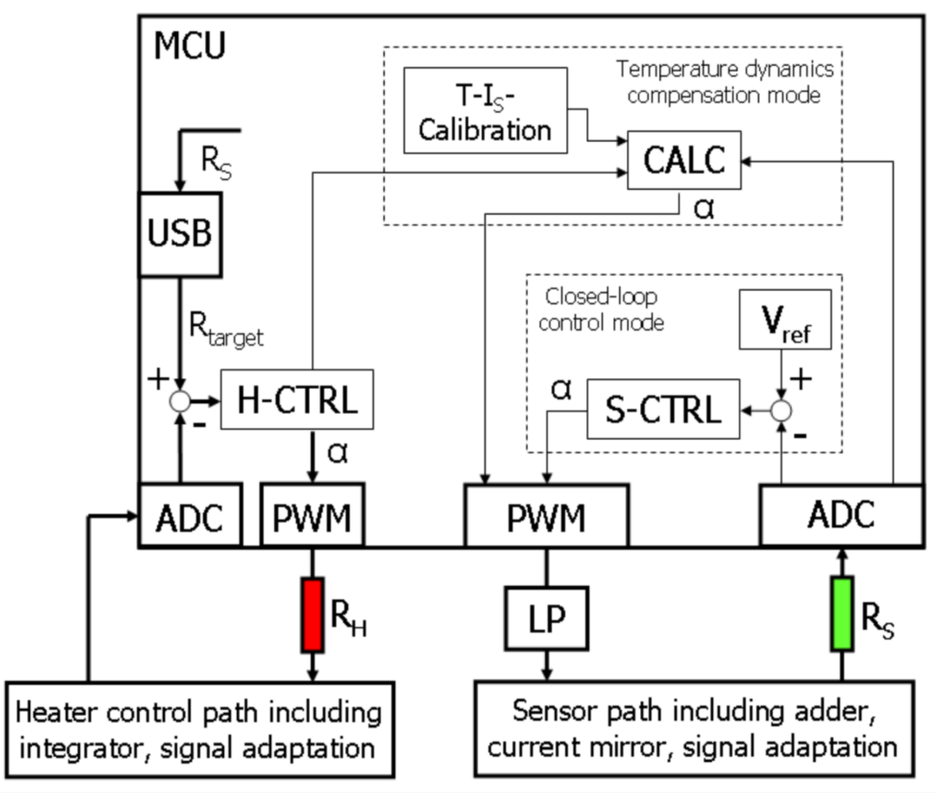
- Sensitivity
  - Broad spectrum
    from ppb (for malodors, ozone, hazardous VOCs)
    up to 1000 ppm (gas leak, CO_2)
- Selectivity
  - False alarms are primary concern for fire detection (ratio 10:1)
  - VOC detection: hazardous (formaldehyde) vs. neutral (alcohol vapor, cleaning agents) vs. wanted (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?

A generic modular gas sensor system



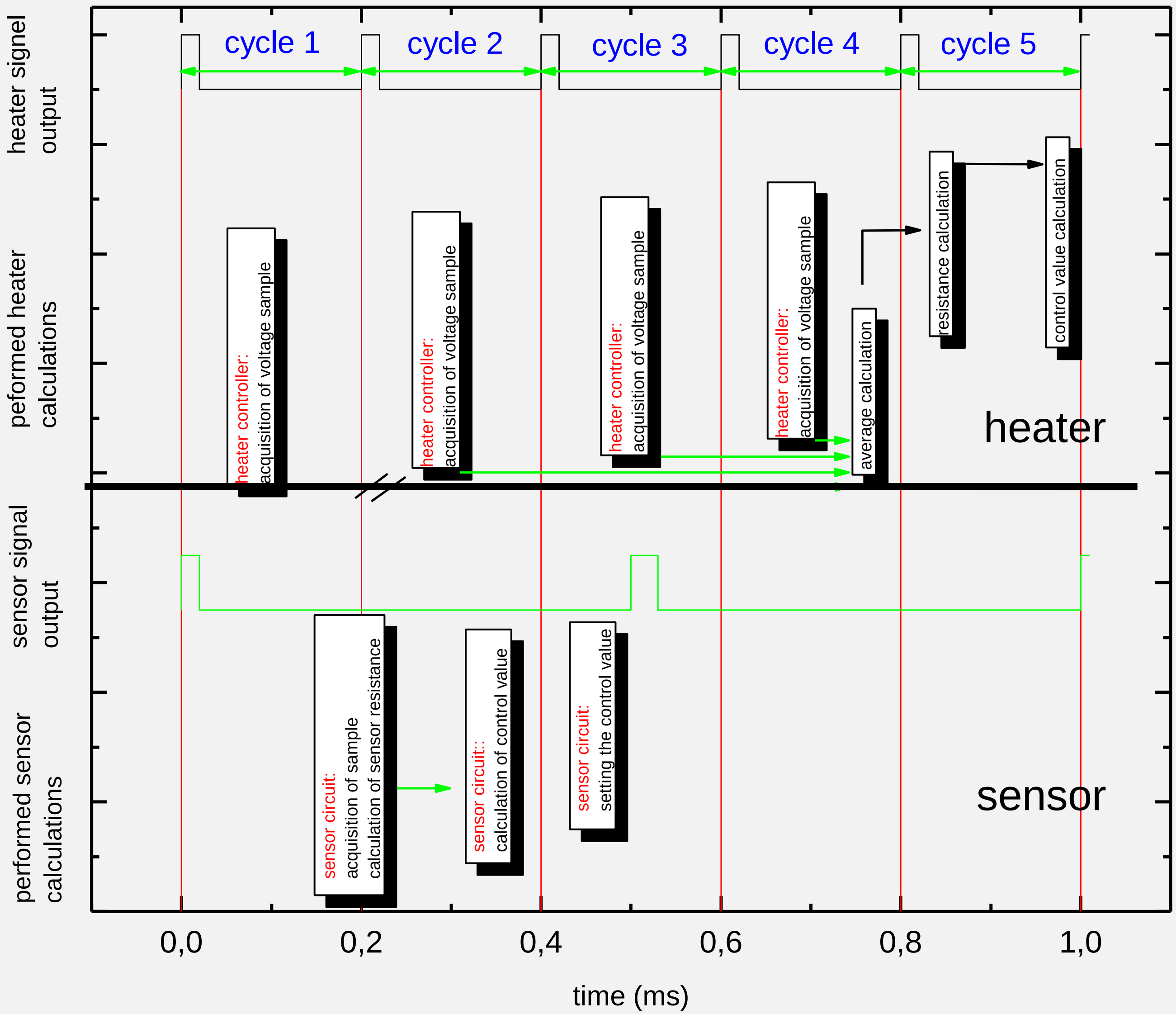
Hardware platform PuMaH for exact temperature control and large dynamic range data acquisition - Pulse-width-modulated Measuring and Heating Unit

- Heater temperature control
  Heater resistor $R_H(T)$ controlled at 5 kHz for exact temperature control of micro-hotplates
- Sensor resistance read-out
  Gas sensitive layer: R_S(gas)
  - Closed-loop control mode
    → constant voltage drop across R_S
  - Temperature dynamics compensation mode
    → large dynamic range of 26 bits
- Software controlled
- 16 bit PWM outputs used to apply signals to R_H and R_S

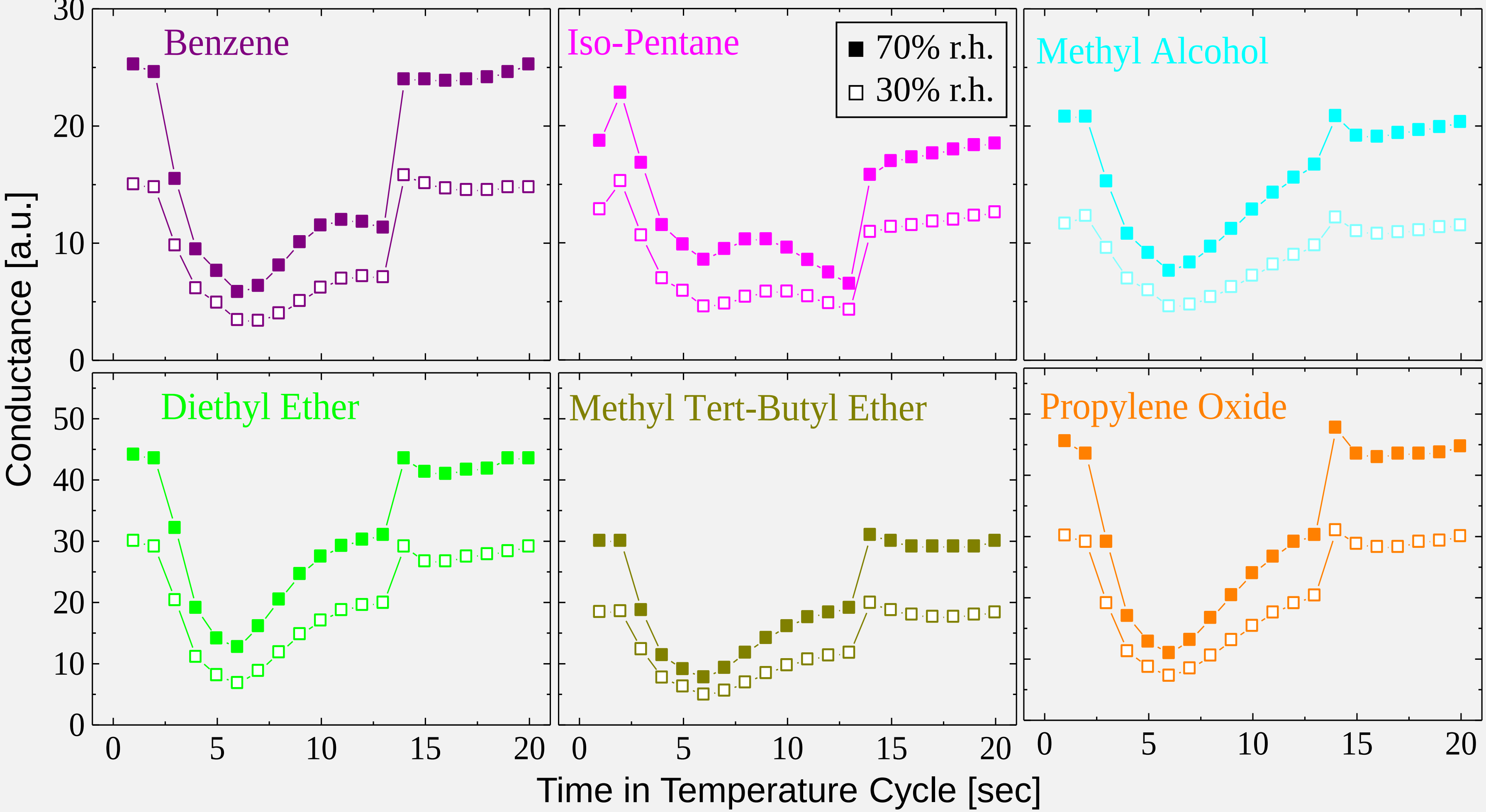


Th. Conrad et al., IEEE Sensors Conference 2005

Gas measurement systems – more than sensors



Th. Conrad et al., IEEE Sensors Conference 2005



A. Schütze, A. Gramm, T. Rühl
IEEE Sensors Journal, Vol. 4, No. 6, 2004

Signal evaluation:

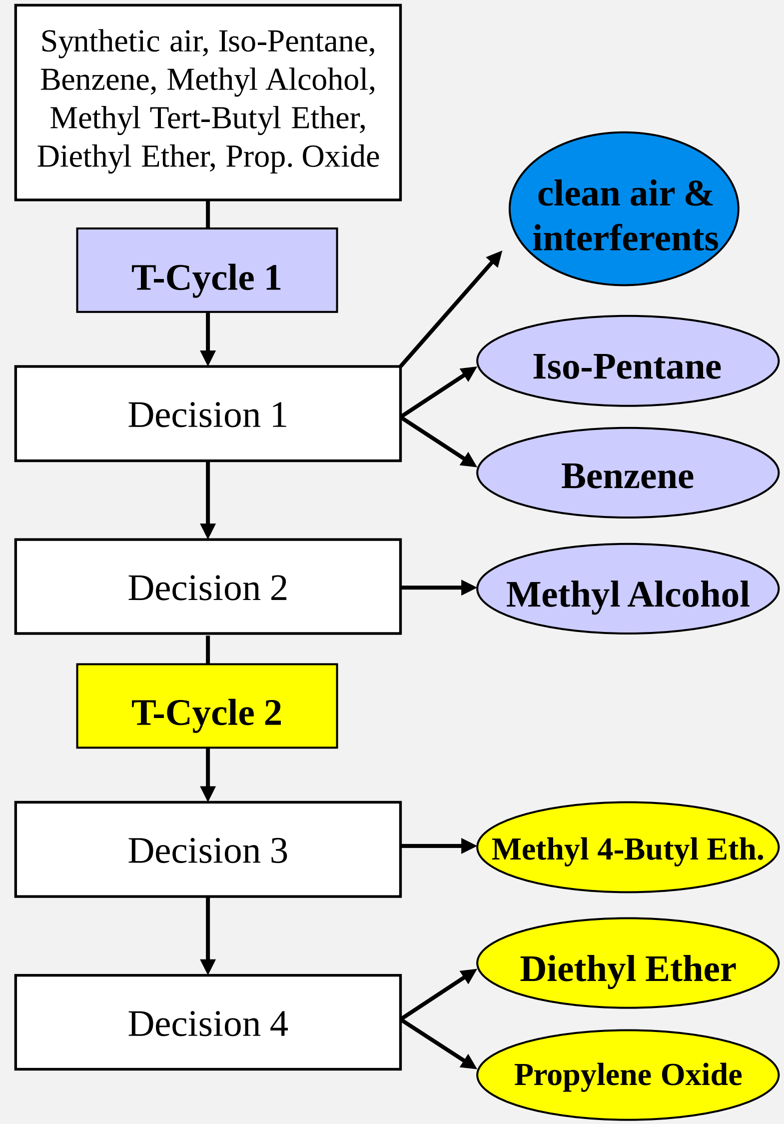
1. Normalization of the response curves ⇒ reduces sensor drift
2. Generation of secondary features, i.e. levels, slopes etc.
3. Suitable patterns are extracted for further evaluation

Evaluation of sensor data based on temperature cycling (example)
→ **Virtual multisensor**

Characteristic features of the curve shapes (i.e. ***slope at the end of the high temperature phase*** and ***curvature during the low temperature phase***) are evaluated, to discriminate between different gases in several steps.

Note: the decision tree reflects the chemical composition of the solvents starting with the alkane pentane and the aromatic benzene (both pure CH-compounds), then the alcohol (R-COH) and finally the three ether compounds (R1-O-R2). This indicates that an expansion might be possible to classify many different molecules.

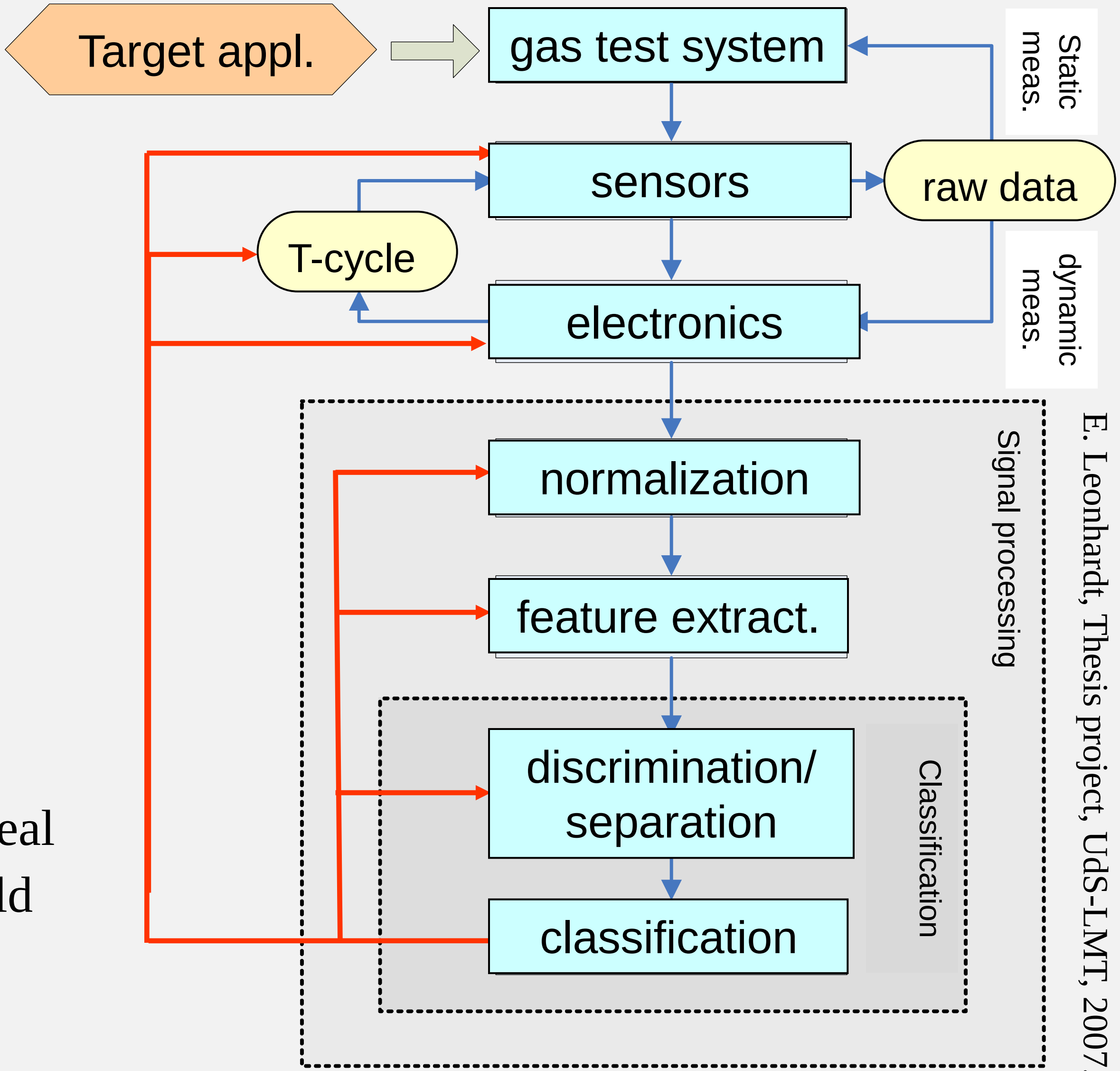
Source: A. Schütze, A. Gramm, T. Rühl
IEEE Sensors Journal, Vol. 4, No. 6, 2004



Many possibilities for optimization:

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

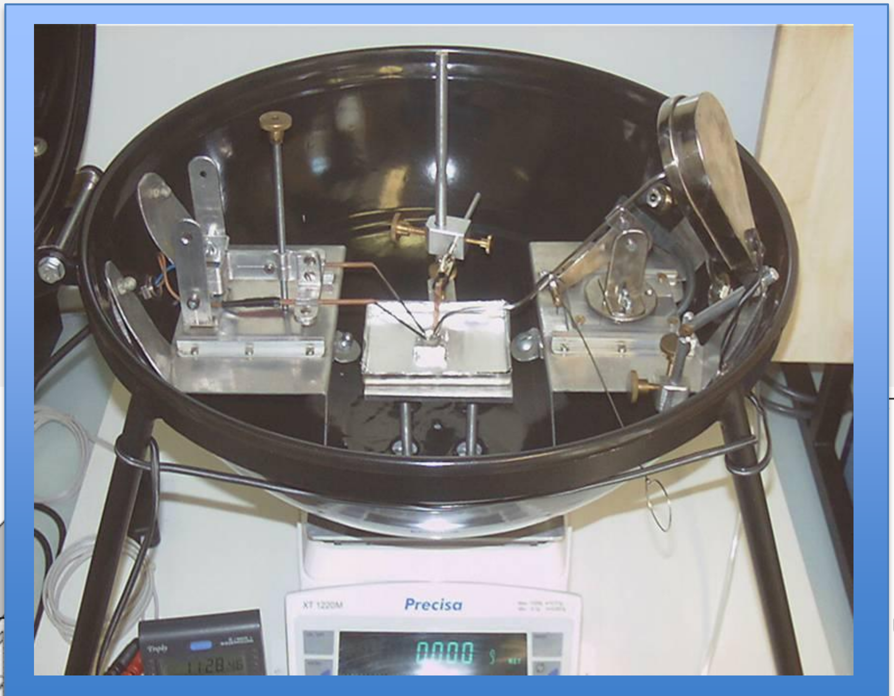
…and **always** testing under real application conditions (field testing)!

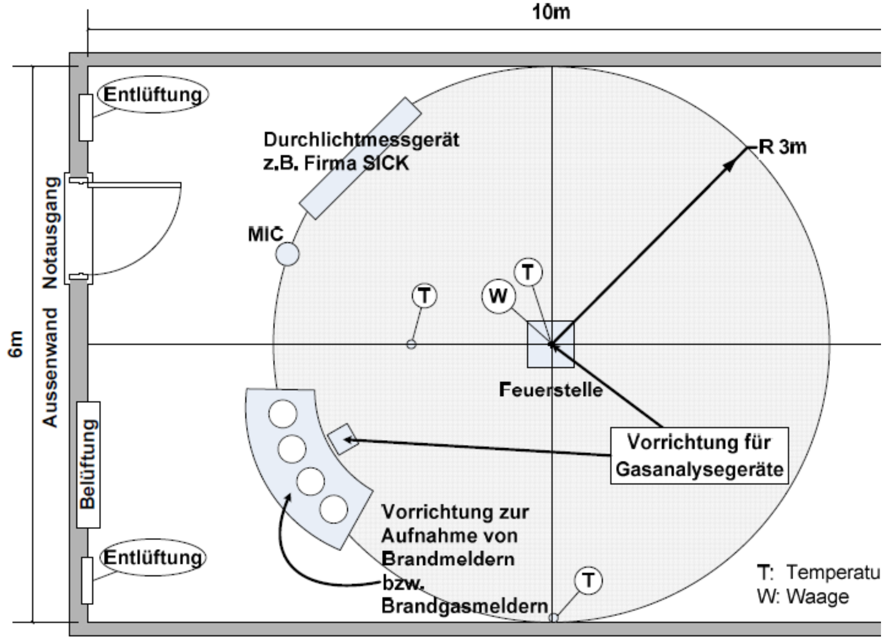


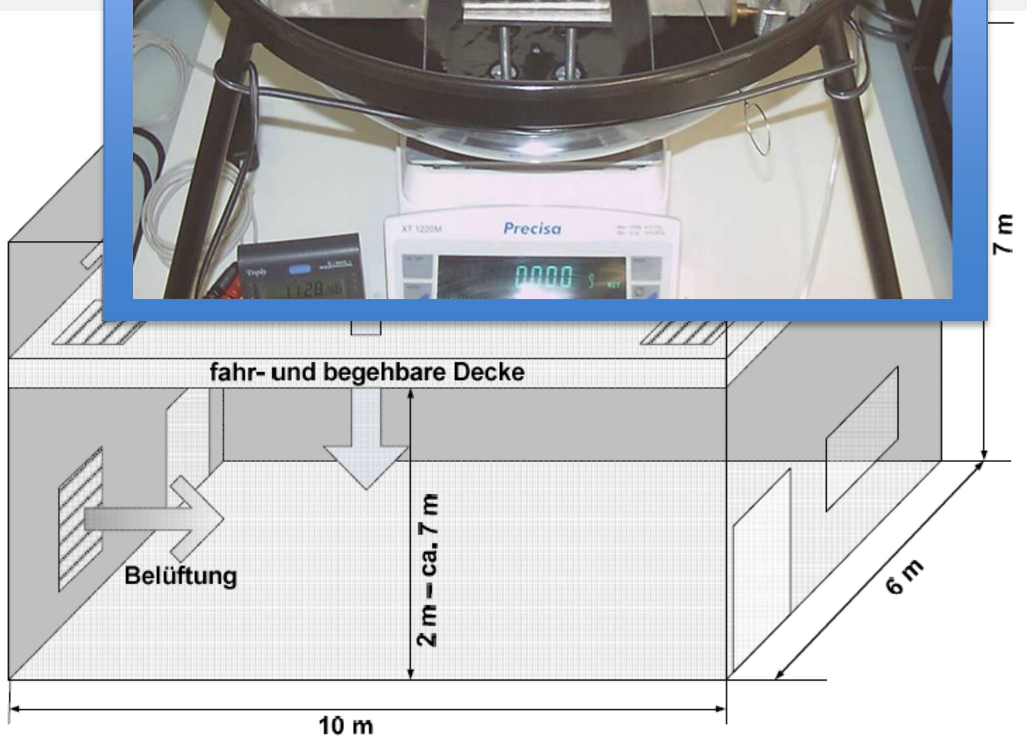
E. Leonhardt, Thesis project, UdS-LMT, 2007.

Calibration and field testing

- Gas mixing systems
- "Live tests" e.g. for fire detection
- Miniaturized test systems for faster, more efficient evaluation







Z. Ankara, PhD thesis, UdS-LMT, 2008.

Application examples – fire detection

Goal: a new type of fire detector with

- improved sensitivity for smoldering fires and
- reduced false alarm rate

Approach: integration of a gas sensor in conventional smoke/temperature detectors

Requirements

→ Low cost (approx. 2-5 $ per unit!)
  - achievable using mass produced microstructured gas sensors

→ Low power consumption (max. 10 mW, best below 1 mW)
  - Miniaturization combined with intermittent operation (250 ms on, 15 sec off)

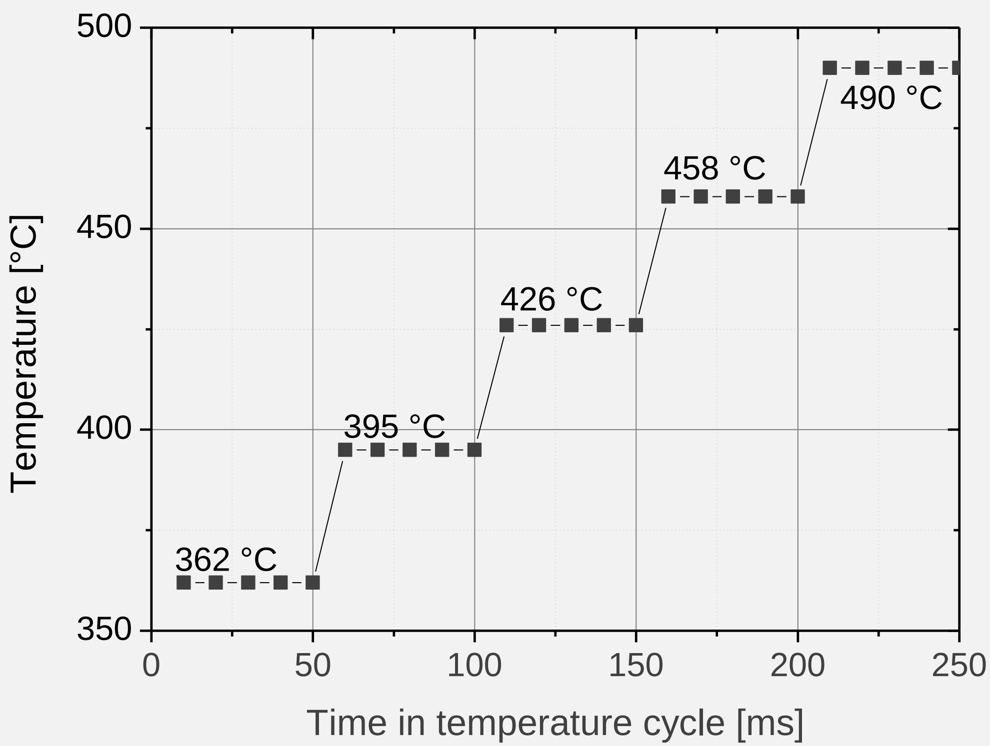
→ Good discrimination between harmful fires and background
  - Virtual multisensor approach (temperature is changed anyway for low power)

→ 10-year lifetime without maintenance???

Temperature cycle for ultra low power

→ Five temperature levels during 250 msec cycle followed by 14.75 sec pause

→ Average power consumption 1.33 mW



Sensor-Type	1 mW-Cycle			
	U_H [V]	R_H [Ω]	P [mW]	T [°C]
MiCS 5131	3.20	109.3	93.69	490
	3.00	106.8	84.27	458
	2.90	104.4	80.56	426
	2.75	102.1	74.07	395
	2.60	99.7	67.80	362
$P_{average}$	~ 80 mW			
$P_{average}$	with 14.75 s heating pause → 1.33 mW			

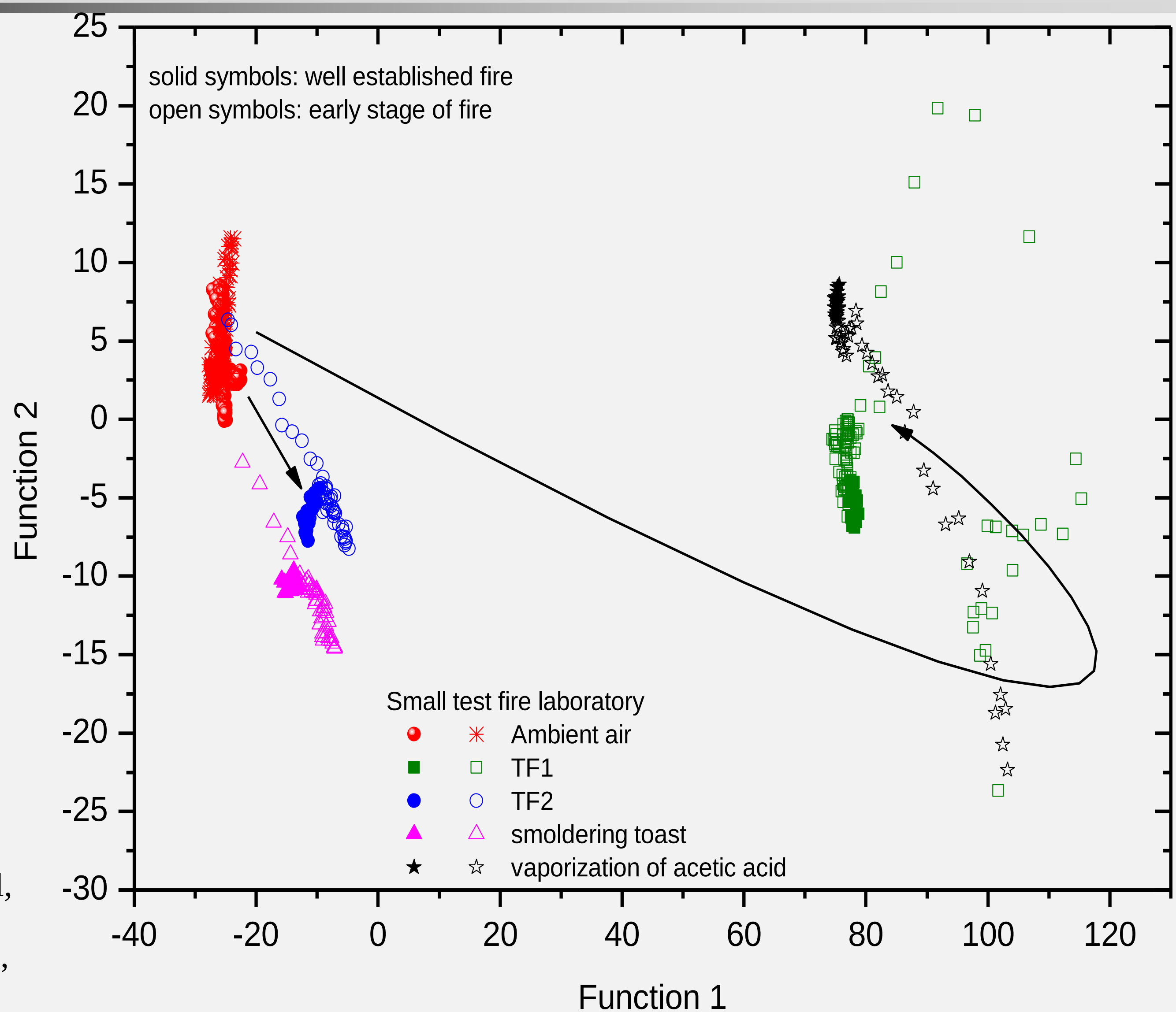
Z. Ankara, A. Schütze
EUROSENSORS 2008 Conference, Dresden

Application examples – fire detection

Lab validation

- → Tested with real fires in small fire test chamber scaled from IEC defined fire test
- → False alarms tested with typical situations based on experience of major producer (i.e. smoldering toast, acetic acid from cleaning agent)

Z. Ankara, T. Kammerer, M. Engel, H. Nagel, A. Schütze SENSOR+TEST 2005 Conference, Nuremberg

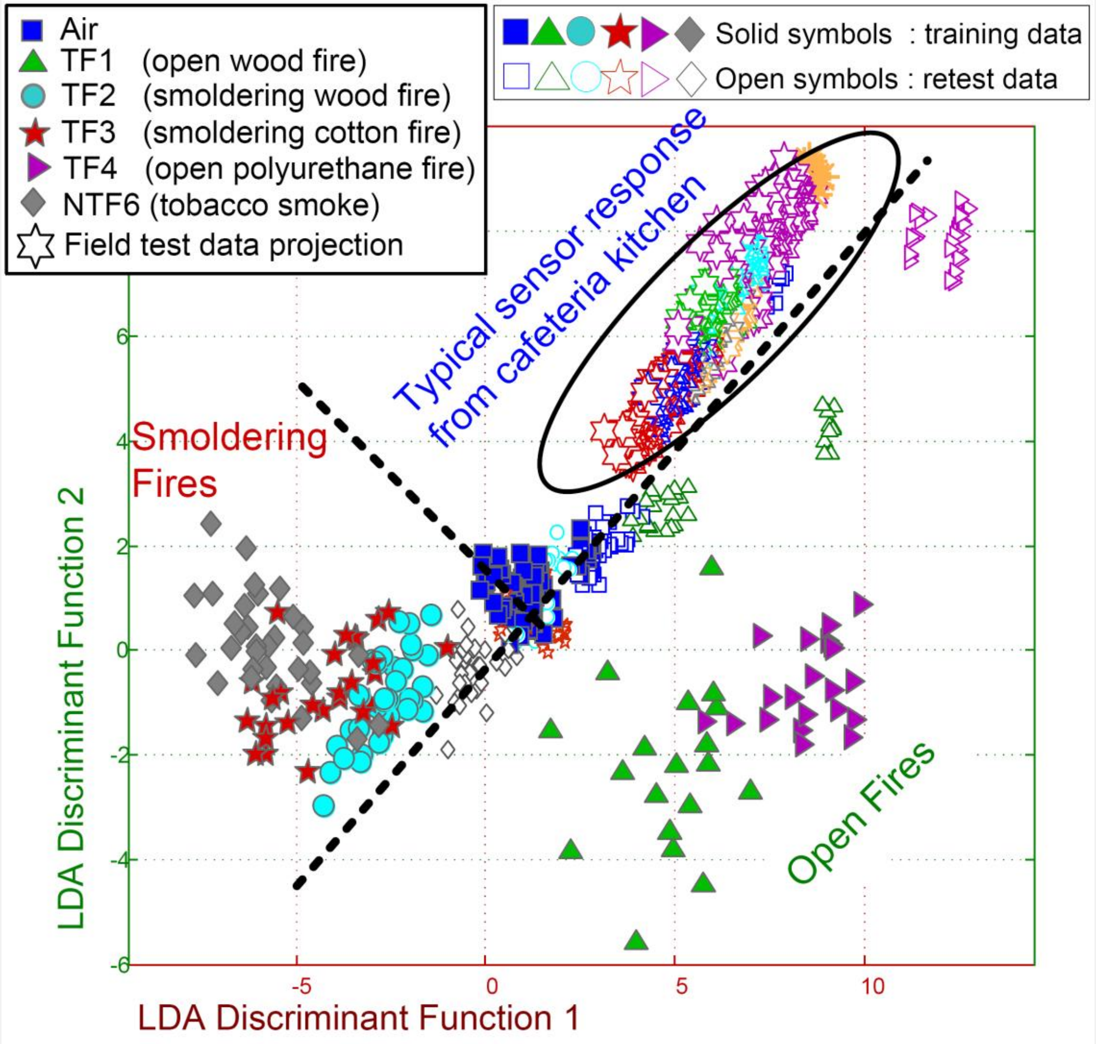


Application examples – fire detection

Field evaluation

- → Gas sensor strongly responded to typical cooking odors
- → However, discriminant analysis shows that cooking odor patterns are not confused with trained open or smoldering fires
- → Stable performance of the sensors over several months of operation even in kitchen atmosphere (grease, high humidity, high temperatures)

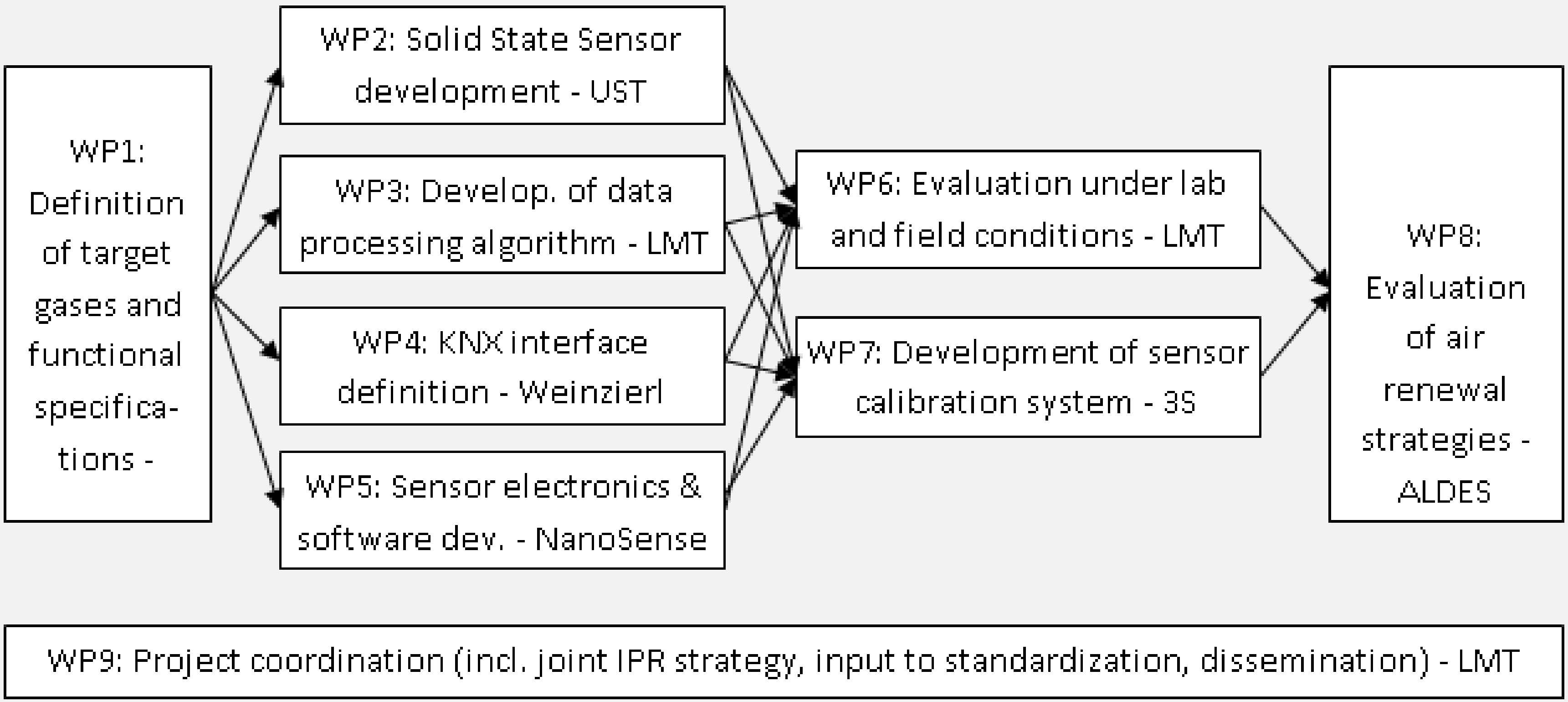
Z. Ankara, A. Schütze
EUROSENSORS 2008 Conference, Dresden



Application examples – indoor air quality

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



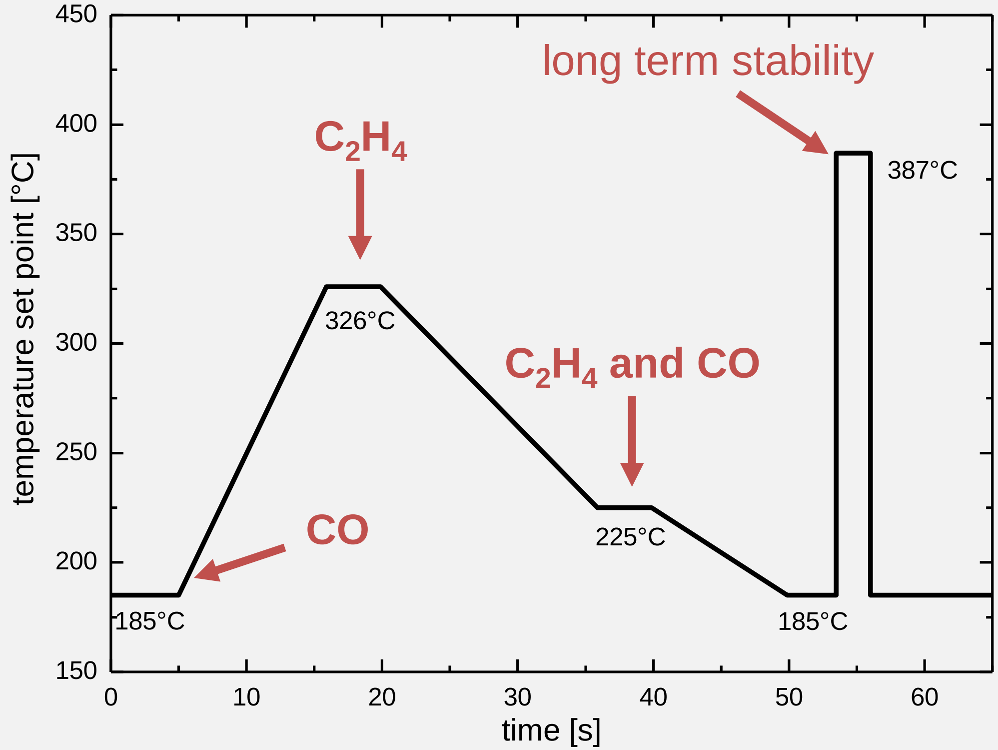
Application examples

VOC-IDS: ppb-level detection of VOC against background gases?

Similar requirements: Fire detection in coal mines

- CO/C_2H_4 mixtures at 10 ppm/100 ppb respectively
- Background: up to 1% methane + interfering gases: r.h., CO, H_2, NO_2 etc.

P. Reimann, PhD thesis, UdS-LMT, 2011



different temperature levels for the target gases

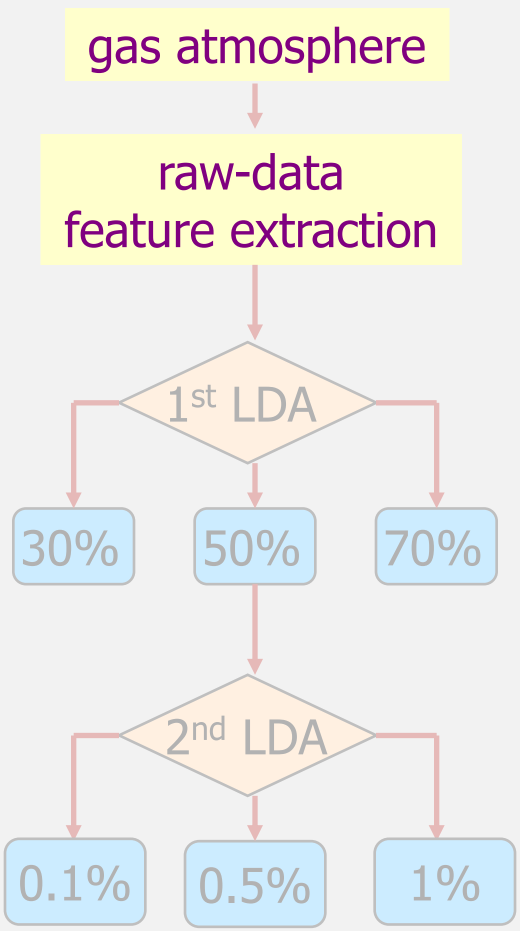
ramps between these levels allow max. reproducibility

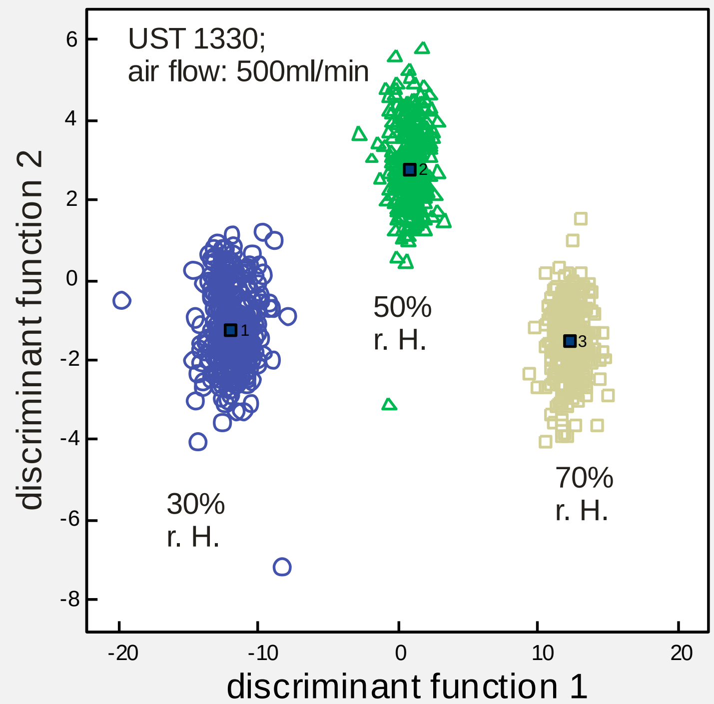
↓

feature extraction

slopes and mean values from these sections

Application examples



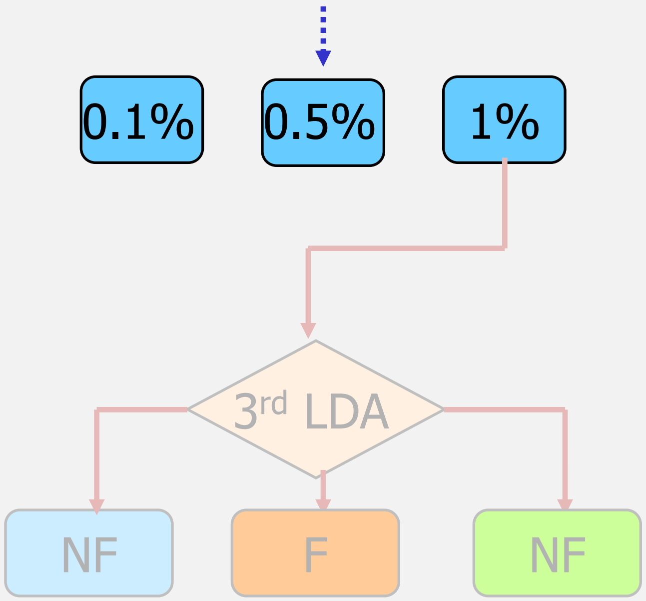


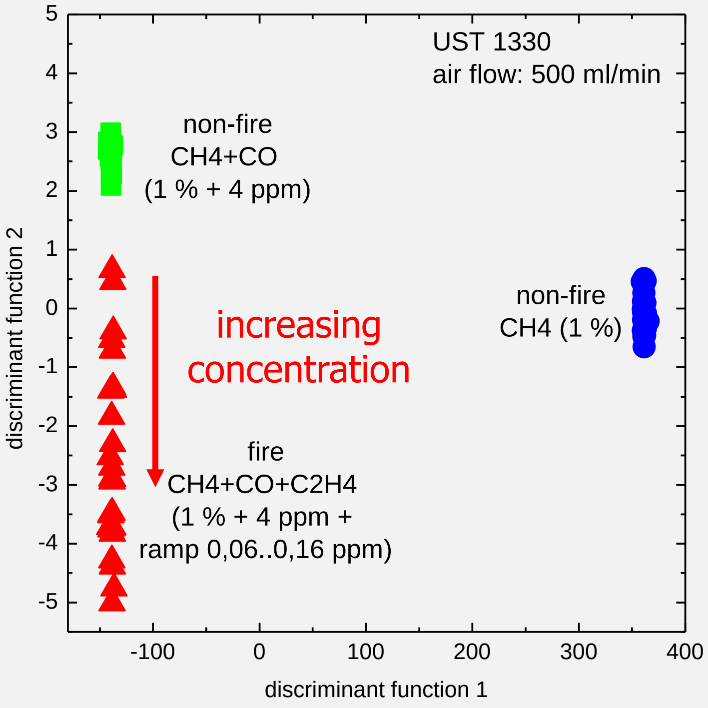
- discrimination along function 1
- similar for CH_4

P. Reimann, A. Schütze: Sensor Review Vol. 32 Iss: 1, 2012

Application examples

P. Reimann, A. Schütze:
Sensor Review Vol. 32 Iss: 1, 2012

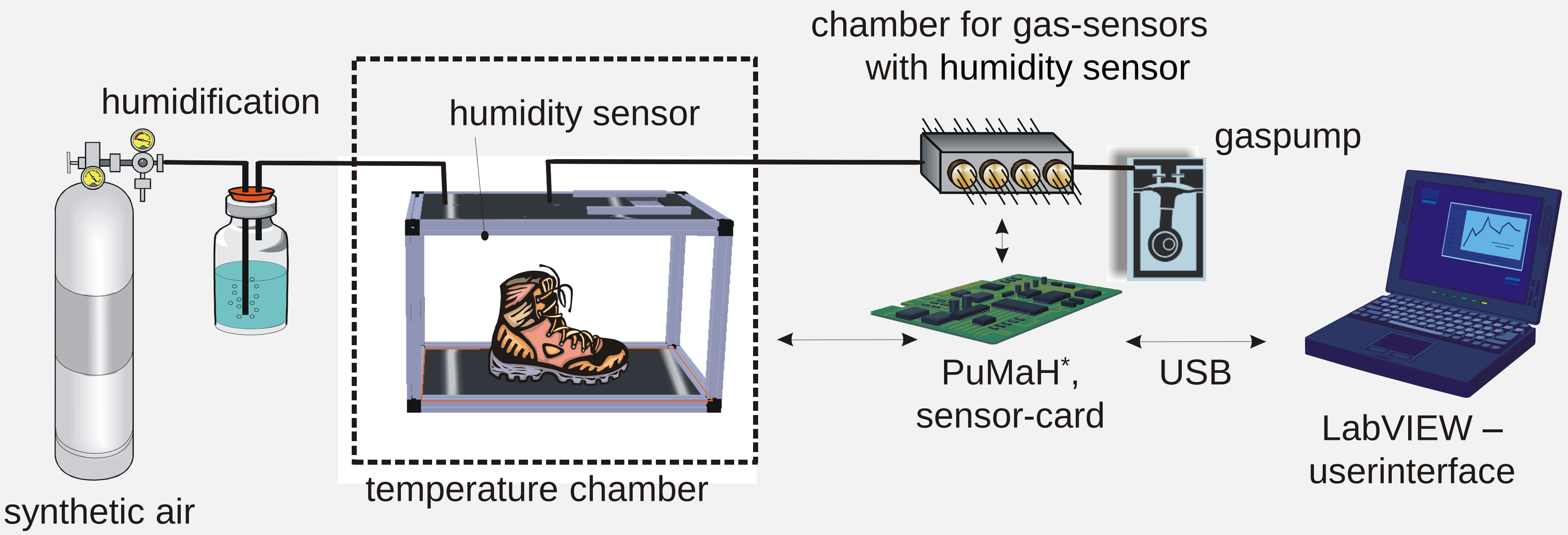




- alarm decision
- discrimination along function 1 & 2
- additional step(s) for interfering gases

Field test: correlation with existing sensors shown, stability needs to be improved

Evaluation of the smell of sweat with a temperature cycled MOS sensor



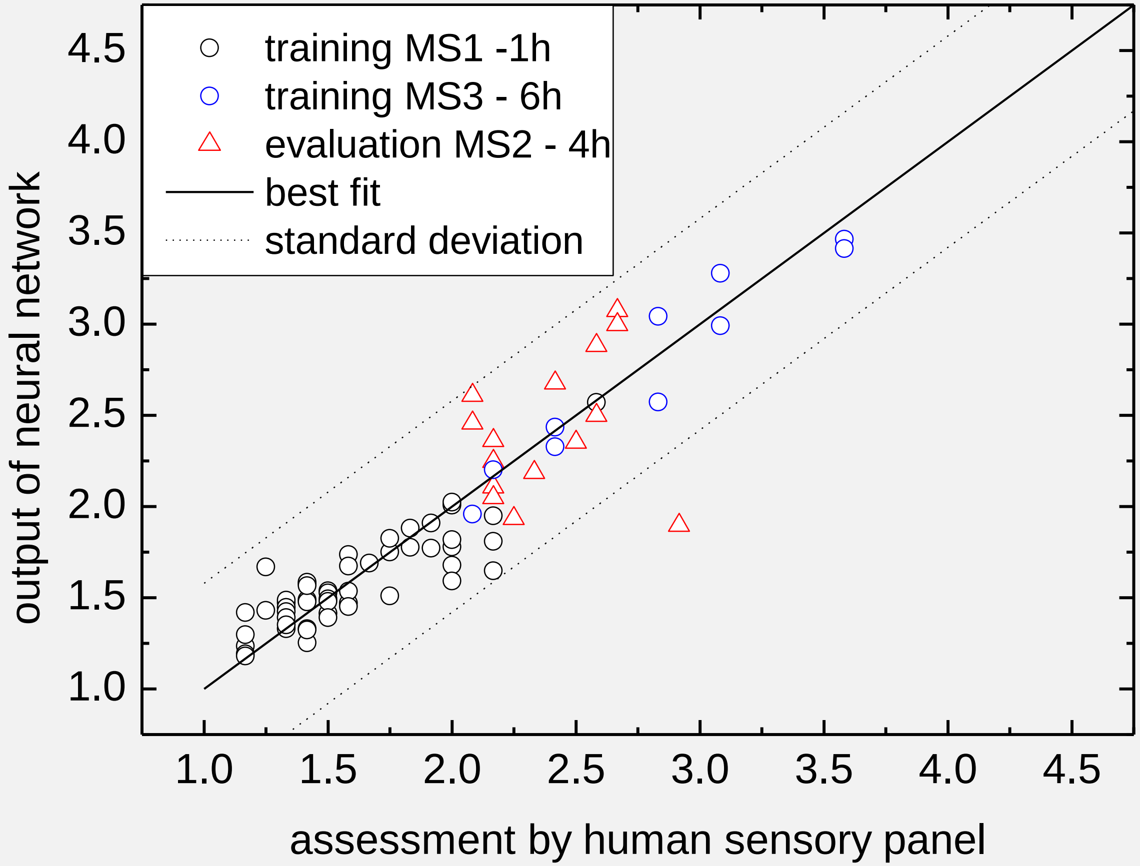
S. Horras et al.: Evaluation of an E-nose system for objective smell assessment of shoe/socks systems by comparison with a sensory panel, SENSOR+TEST Conference, 2009.

Application examples – odor assessment

Evaluation of the smell of sweat with a temperature cycled MOS sensor

- Correlation with Artificial Neural Network (ANN)
- 3 measurement series (MS) – all shoes measured one day after wear

S. Horras et al.: Correlation of an E-Nose system for odor assessment of shoe/socks systems with a human sensory panel, ISOEN Conference 2009.



sweat odor intensity
1: no sweat odor
5: very strong sweat odor

three layer ANN
(8 – 8 – 1 neurons)
in feedforward topology

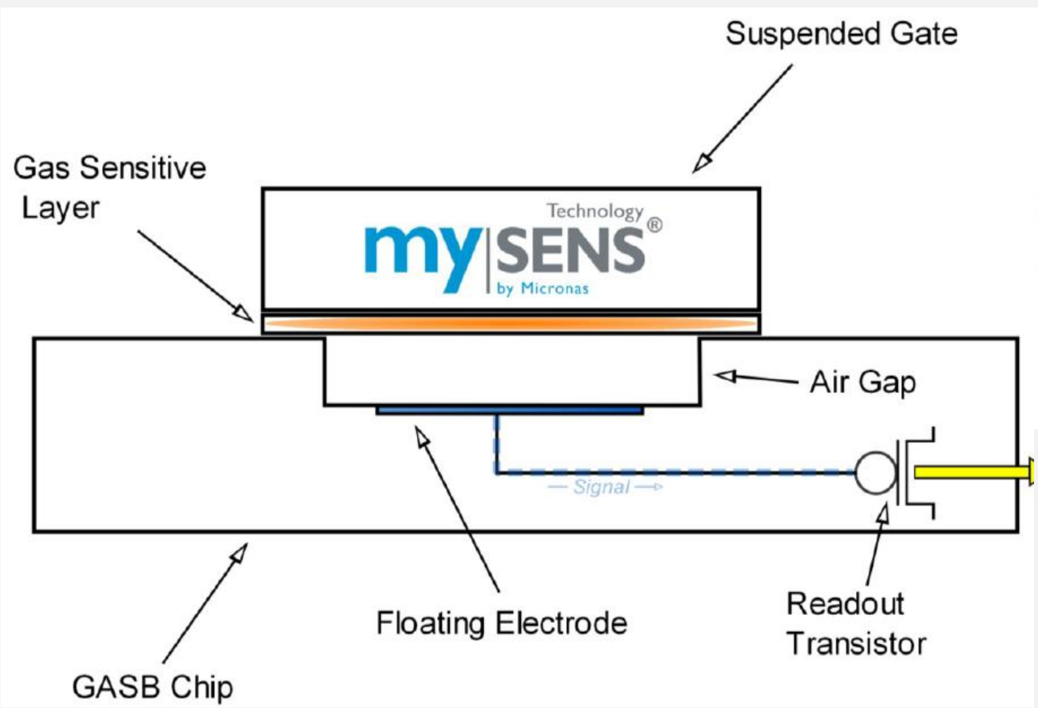
mean values of all temperature cycles per measurement are plotted

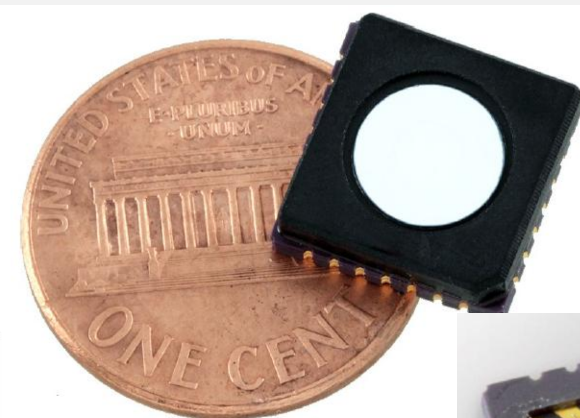
- **Stable operation over several months**
- **Sensor can eliminate odor of shoe material (leather etc.)**

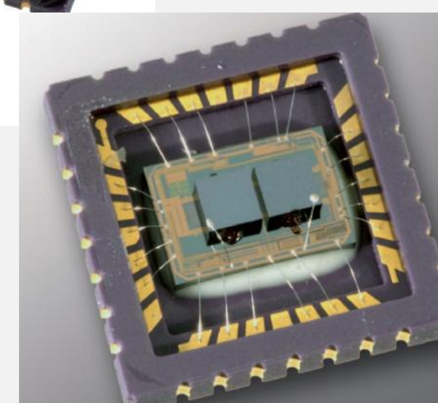
Novel developments

Novel sensor materials and transducer principles

- Nanotechnology for improved gas sensitive layers
- Novel transducer principles
  - Gas ionization (can be electronically controlled!)
  - GasFETs commercially available

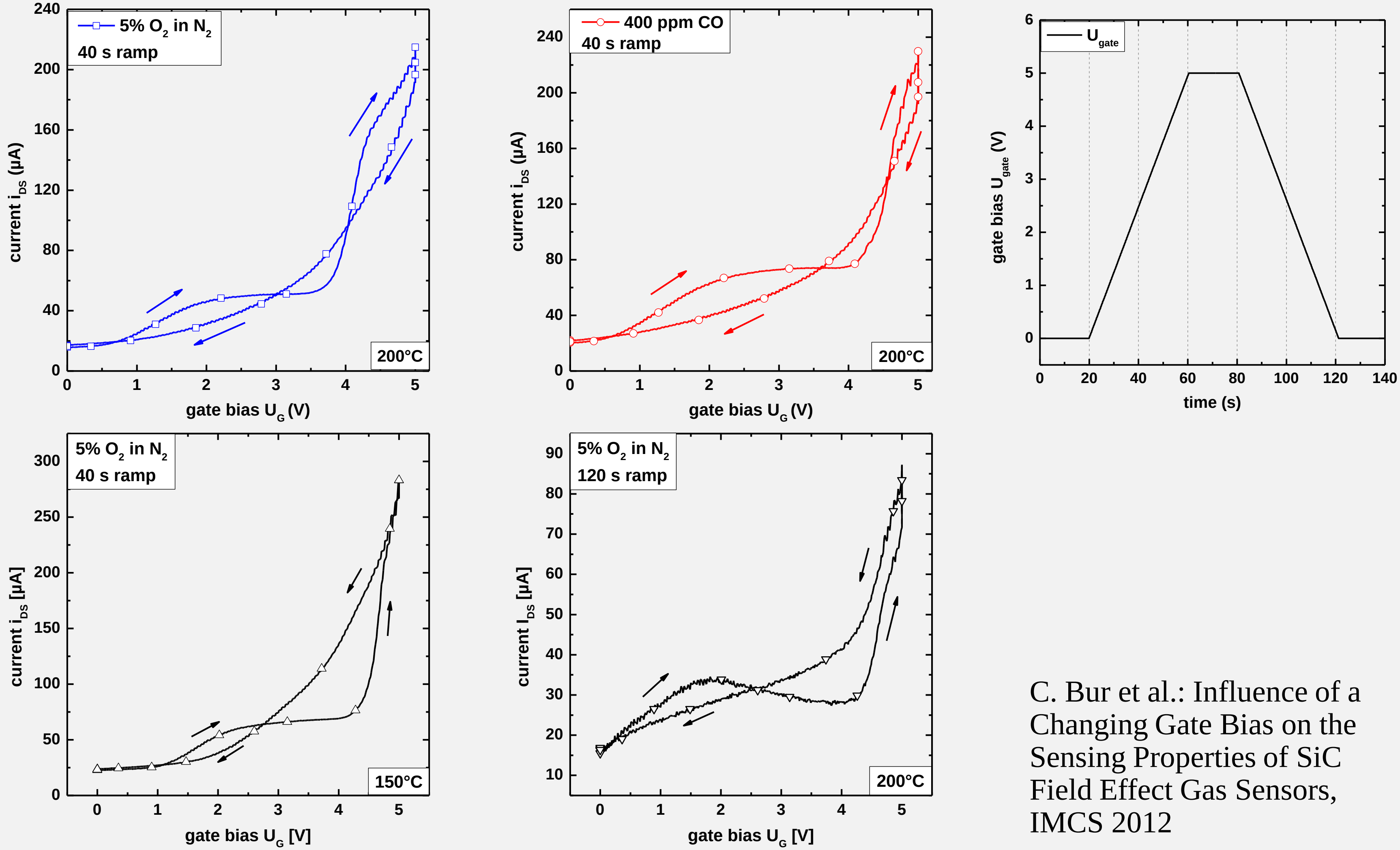






Source: Micronas

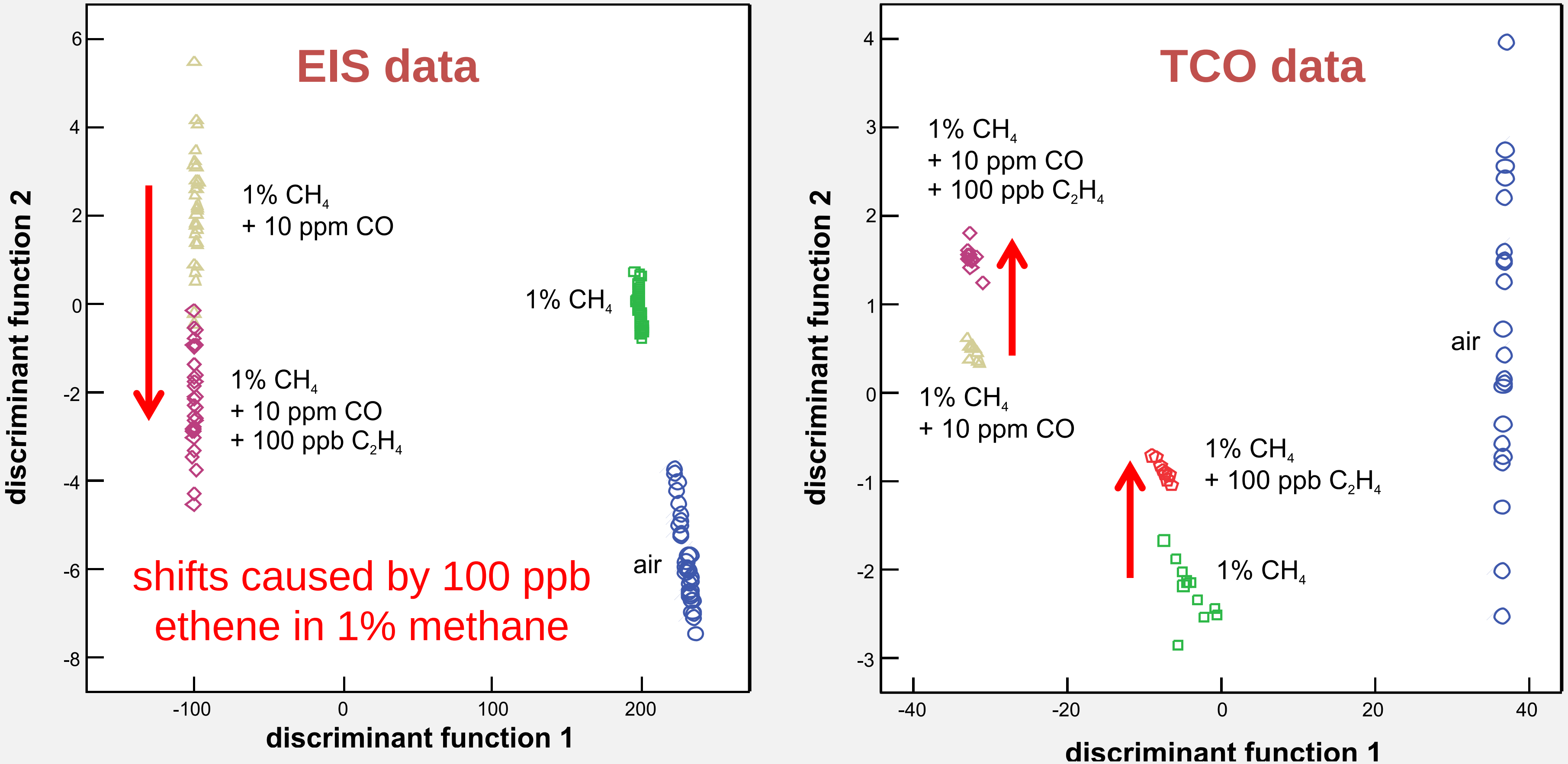
System integration: temperature and gate bias variation for SiC-GasFETs



C. Bur et al.: Influence of a Changing Gate Bias on the Sensing Properties of SiC Field Effect Gas Sensors, IMCS 2012

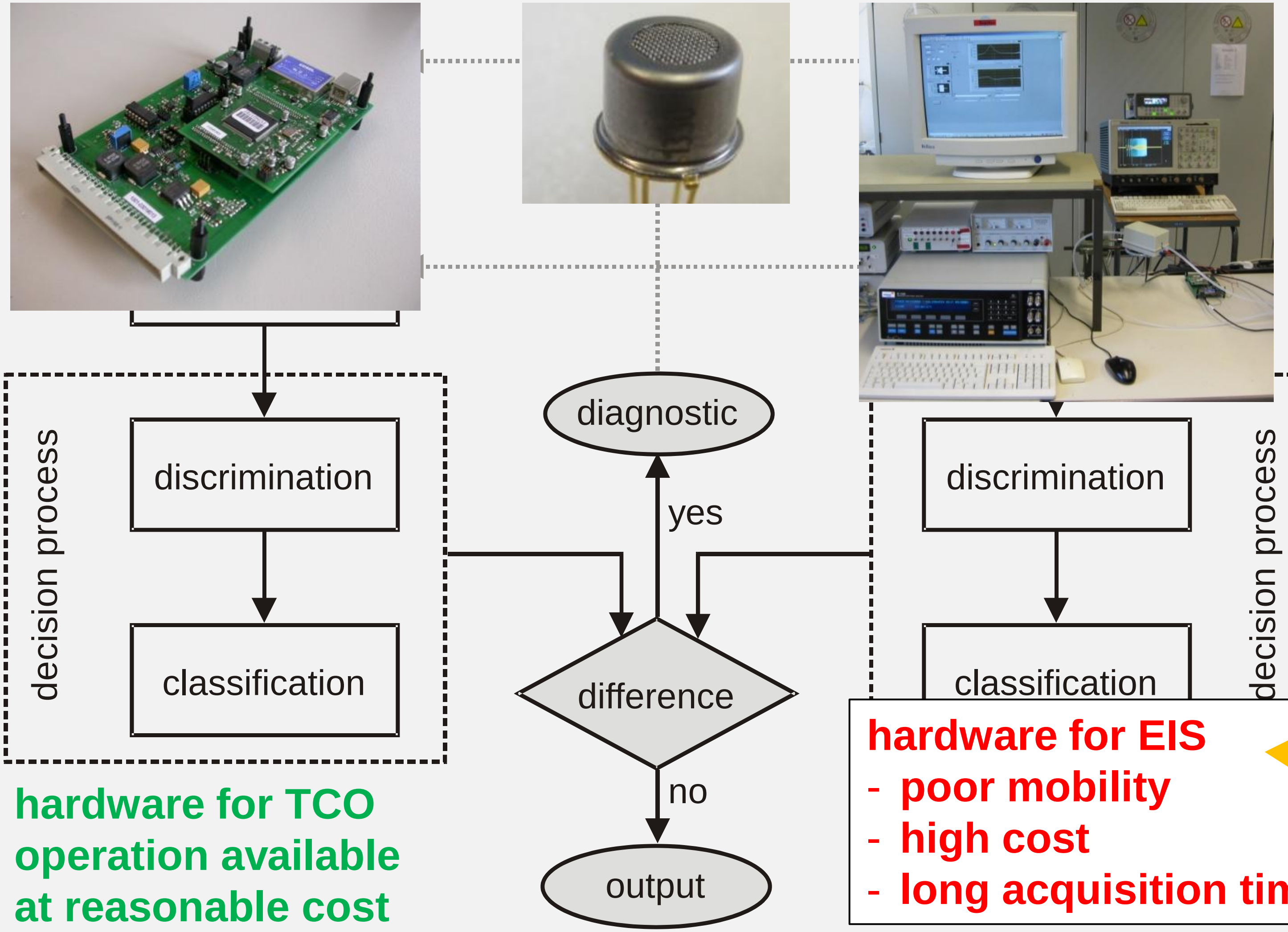
Novel developments

- Electrical Impedance Spectroscopy yields similar improvement in selectivity as Temperature Cycled Operation (but time scale is completely different)



A. Schütze et al.: Improving MOS Virtual Multisensor Systems by Combining Temperature Cycled Operation with Impedance Spectroscopy, ISOEN 2011

Sensor self-monitoring with combination of TCO and EIS



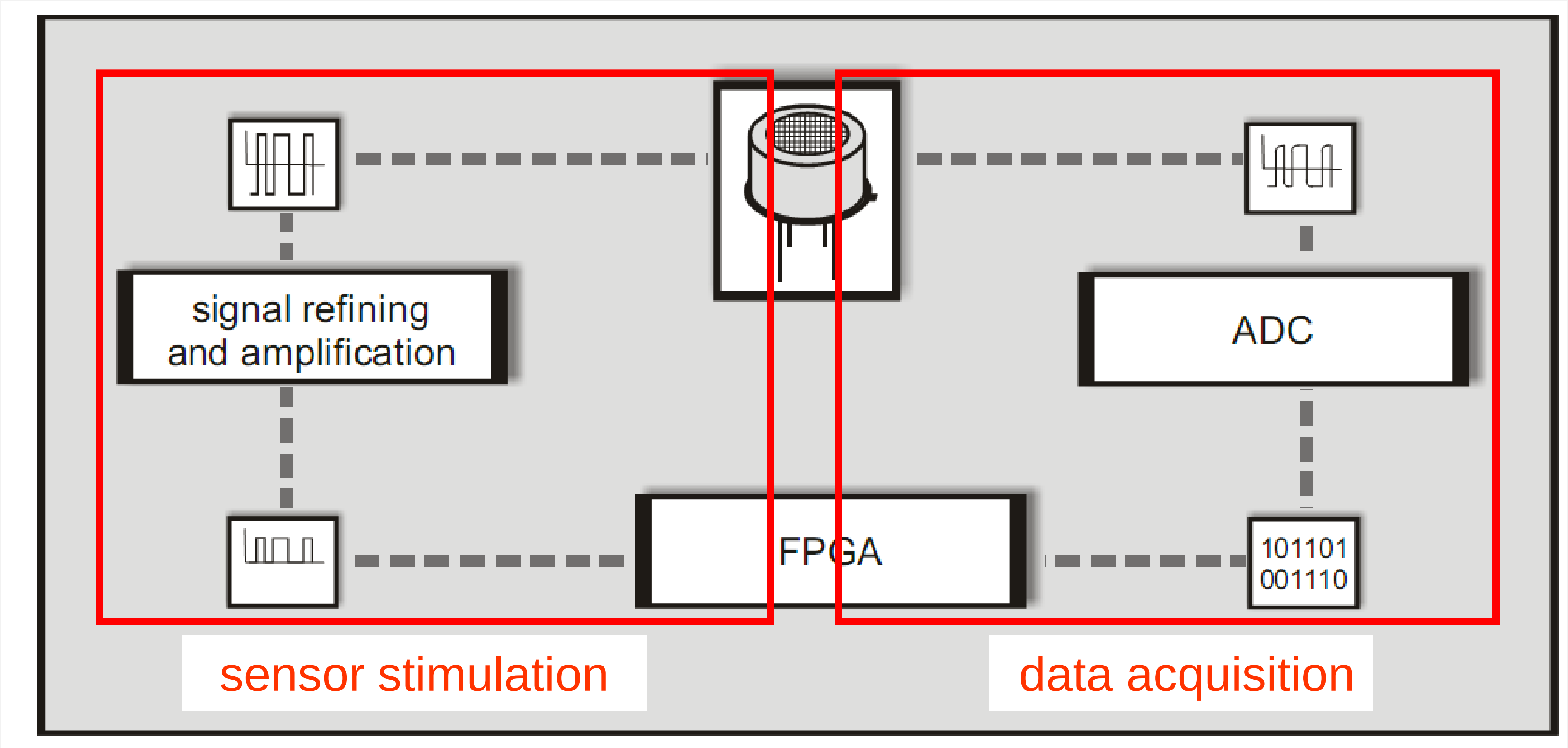
hardware for TCO operation available at reasonable cost

hardware for EIS
- **poor mobility**
- **high cost**
- **long acquisition time**

A. Schütze et al.: Improving MOS Virtual Multisensor Systems by Combining Temperature Cycled Operation with Impedance Spectroscopy, ISOEN 2011

Low cost EIS hardware realization: general approach

- Implementation using an FPGA (field programmable gate array)
- MLS signal refined with variable amplification using dedicated circuit
- Data acquisition using high speed ADC (sample rate 200 MHz)



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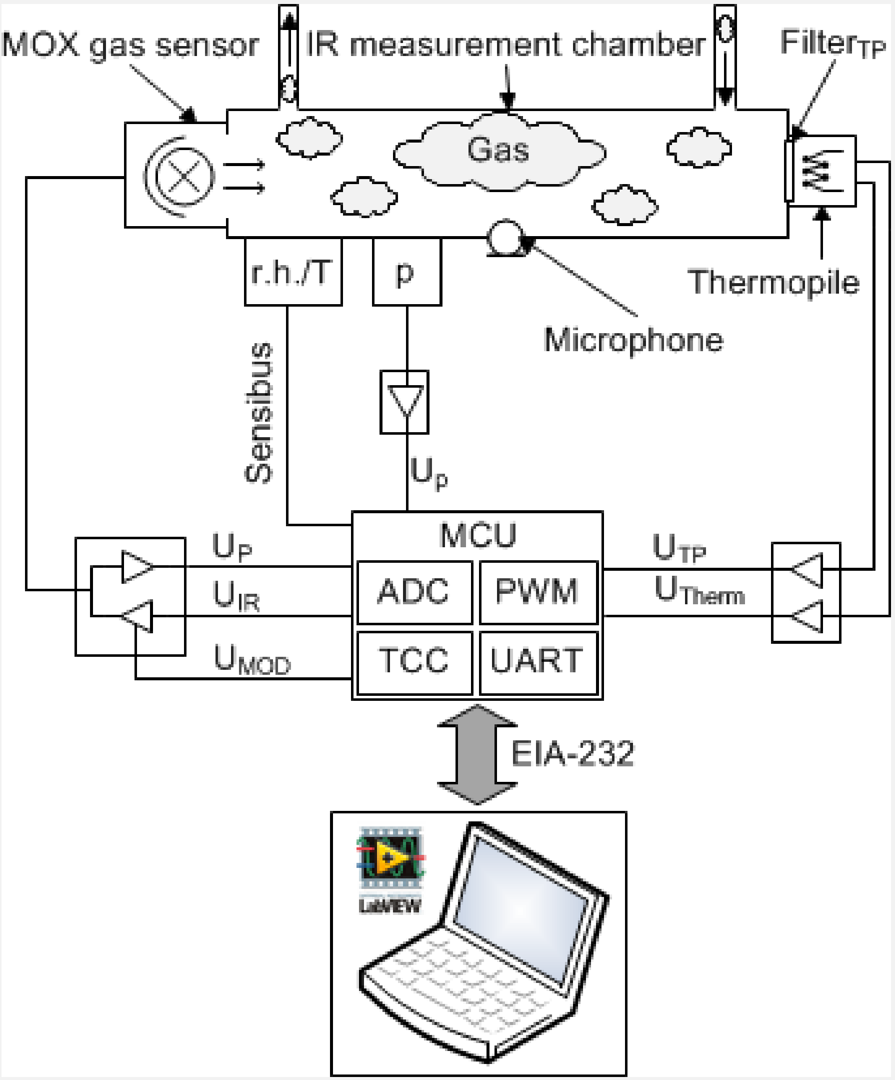
Novel developments



MOS-IR measurement system:

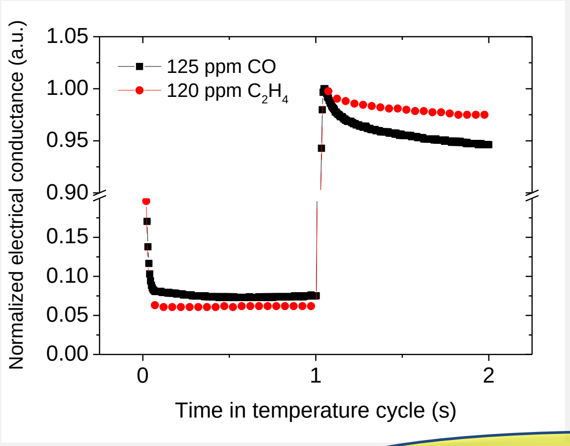
- Gas filled chamber (9 cm length)
- MOS gas sensor (MICS 5131, e2v)
- Transmission: Thermopile (HIS A21 F4.26, Heimann Sensors)
- Ambient condition monitoring (p, r.h./T)
- Electronics controlled by a microcontroller
- Configuration settings set by a GUI (LabVIEW)
- Data evaluation offline using Matlab

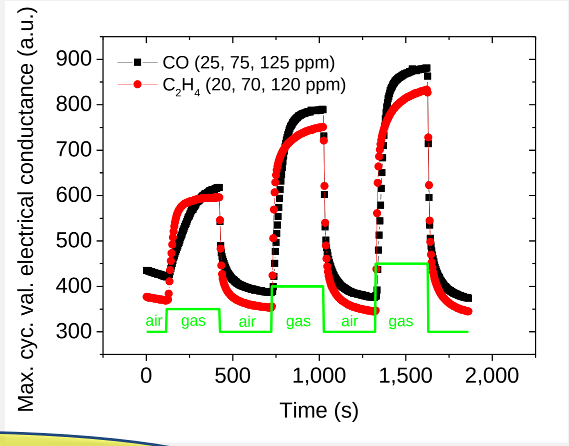
K. Kühn et al.: Investigations on a MOX Gas Sensor as an Infrared Source for an IR-based Gas Sensing System, IMCS 2012

Novel developments

MOS signal:

Raw data DC resistance → Shape of the response curve from temperature cycle

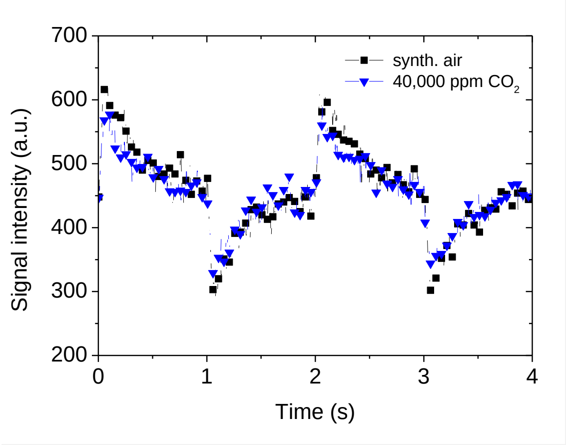


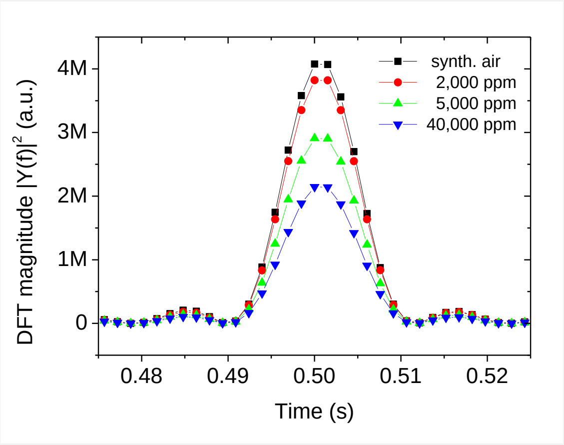


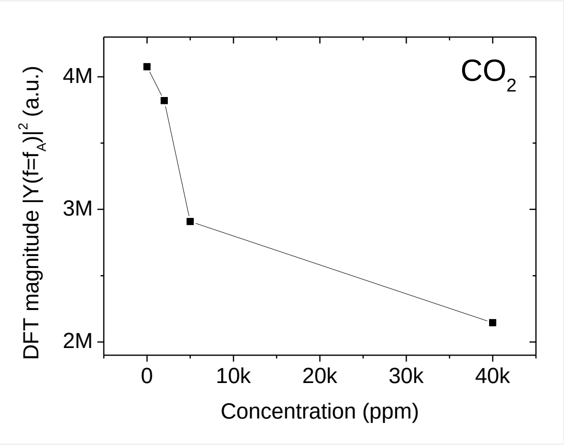
K. Kühn et al.: IMCS 2012

Transmission signal:

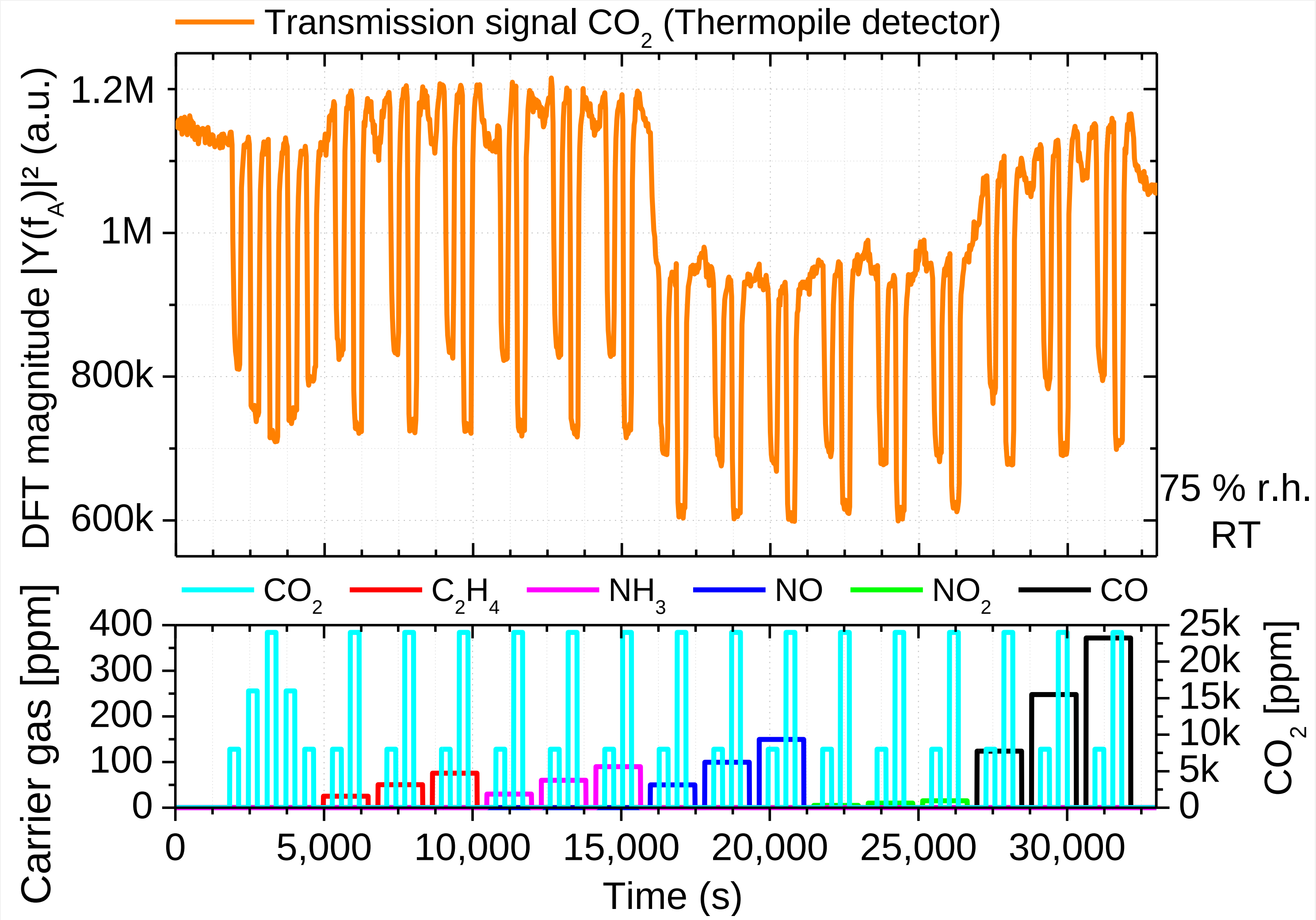
Raw signal (thermopile) → DFT analysis of raw data (90 ON/OFF cycles) → $|Y(f=f_A)|^2(c)$







Transmission signal, f_A = 6 Hz square wave mod. of the MOS gas sensor (MICS 5131, e2v)



K. Kühn et al.: Investigations on a MOX Gas Sensor as an Infrared Source for an IR-based Gas Sensing System, IMCS 2012

Conclusion

- **Indoor applications are of increasing interest, especially for improving energy efficiency**
- **Gas measurement systems are more than just sensors**
- **Multifunctional, intelligent multisensor systems can address emerging applications**
- **Application specific development still required**
- **Field testing is an absolute must for any chemical sensor system**
- **Chemical sensor systems can become ubiquitous in modern building environments**

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Thank you for your attention.

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