Forschungszentrum Karlsruhe
in der Helmholtz-Gemeinschaft

Institut für Instrumentelle Analytik

Accident Warnings, Fire Detection and Air Quality Monitoring with the Karlsruhe Micronose KAMINA for Flight Compartments

Joachim Goschnick, C. Arnold, D. Häringer, I. Kiselev

- Principle considerations
- Gas analytical & other requirements for low cost air monitor
- Karlsruhe Micronose KAMINA and its gradient microarray
- Application examples for indoor air monitoring
- Summary and future prospects

The Nose: A Chemical State Monitor

Noses characterize gas ensembles in an integral manner

- A multitude of gases with appr. constant concentration relations is determined as an entity :
  The gas ensemble is characterized by quality (type) and quantity (concentration)
- To some extent the integral can be broken down to components
  But usually (complex mixtures) resolution of all chemicals cannot be achieved

The human nose is a versatile chemical screening instrument

- **Food:** checking freshness, cooking, frying and baking control
- **Fire:** prevention and detection
- **Health:** breath and skin odor indicate diseases
- **Air, Water and Soil:** pollution often releases smelling vapours
- **Solid/Liquid Products:** often emit volatiles used as a signature for quality

An E-Nose Can Be A Versatile Indoor Air Monitor

- **KAMINA: Online chemical condition monitors for intelligent systems**

- **Indoor air contains a complex gas ensemble**
  - **often continuously changing depending on usage**
  - **but usually keeping within certain limits = normal situation**
  - **deviation from normal may indicate accidents**
- **An E-Nose can continuously track indoor gas ensembles in quality & quantity to obtain information for air conditioning or accident management**
- **Air quality & odor comfort can be obtained for intelligent air conditioning**
- **Fires, pyrolytic degradations & gas leaks adds a characteristic bouquet of volatiles to room air indicating an accident even in advance allowing early counteractions**
- **KAMINA is developed to demonstrate how intelligent systems in industry & households can be supplied with detailed condition information simply, sensitively and at low cost**

Gas Analytical & Other Requirements for Mass Product Compatibility

- **Broad spectrum of detectable gas components**
- **High gas discrimination power**
- **High gas sensitivity, i.e. detection limits < 1 ppm**
- **Rapid enough for on-line measurements: response times < 10 sec**

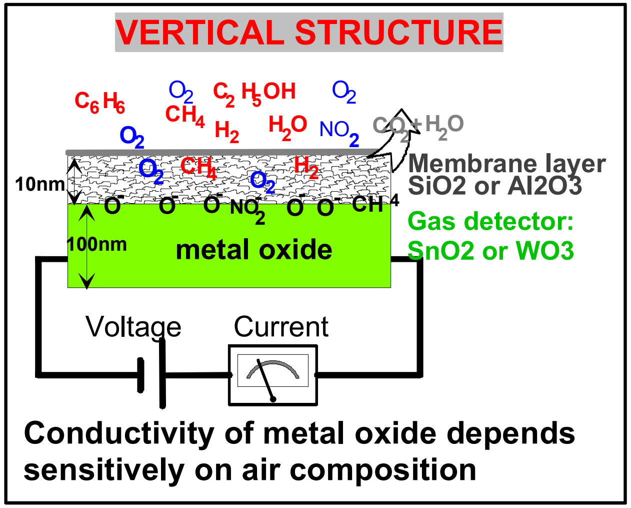
- **Affordability: price < 50 US Dollars**
- **Applicability: low size, low power, robustness, simple handling**
- **Durability: Functionality over terms of 10y or more**

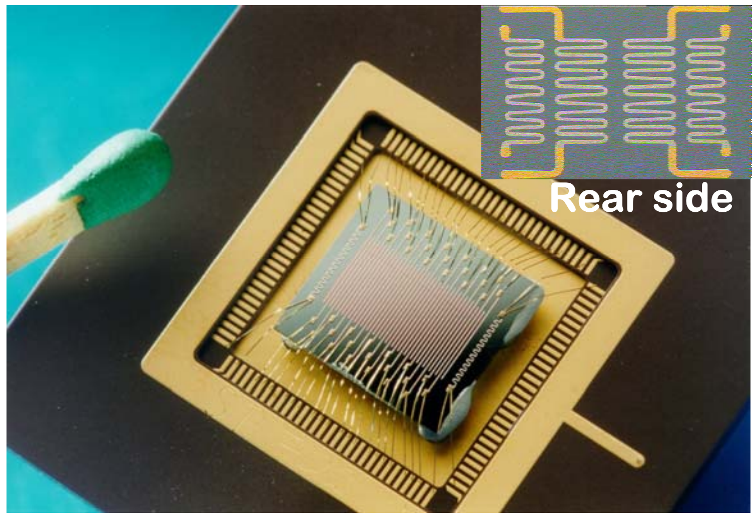
REALIZATION

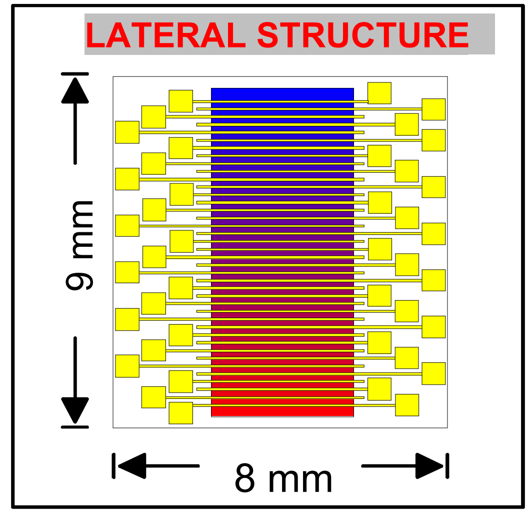
- **Electronic nose based on an array of metal oxide gas sensors is able to detect & discriminate many gases with extreme sensitivity even in rough environment**
- **Microsystems can provide low price, low size and robustness**

Forschungszentrum Karlsruhe
in der Helmholtz-Gemeinschaft
Institut für Instrumentelle Analytik

HIGHLY INTEGRATED GAS SENSOR MICROARRAY
Segmented Metal Oxide Film with Gradient Technique





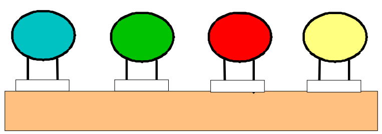


MICROARRAY= Segmented MOX film
STANDARD: 9x10 mm^2 $SiO_2/Si/SiO_2$ or Al_2O_3
39 Pt strips > 38 gas sensor segments
of SnO_2 or WO_3
2 temperature sensors for temp. control
Heating: 4 Pt heating meanders on rear side
Gas permeable membrane: SiO_2 or Al_2O_3

DIFFERENTIATION OF THE SENSOR SEGMENTS

- **2 gradients: chip temperature gradient and thickness gradien across the array differentiate the sensor segments sensitivity spectrum**
- **Conductivity patterns allow recognition and quantitative determination of gas ensembles**
- **Pattern analysis allows determination of gas components**

Innovation in Gas Sensor Array Structures

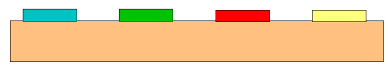


1st generation EN :
Classical macro-design

Separately housed sensors plugged onto a substrate

Expensive production, large, high energy consumption, mechanically sensitive

Chem. differentiation >> sensor-individual ageing

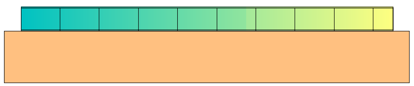


2nd generation EN :
Conventional micro-systems

Sensor pads & interconnection deposited on substrate

Less expensive, small, low power, enhanced mechanical stability

Chem. differentiation >> sensor - individual ageing



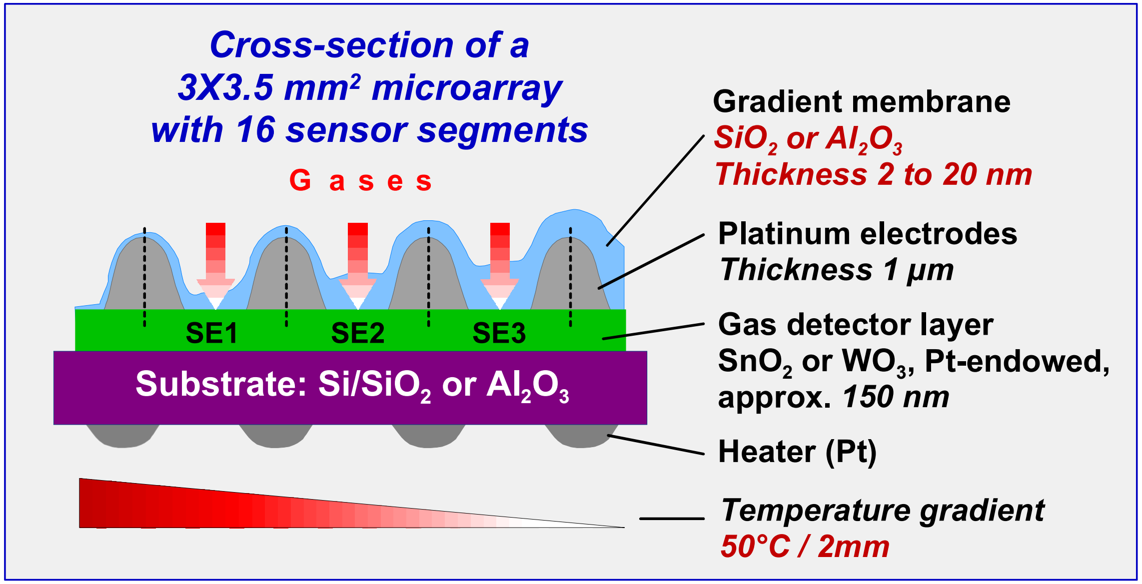
3rd generation EN KAMINA:
Segmented single metal oxide film

No individual sensor elements

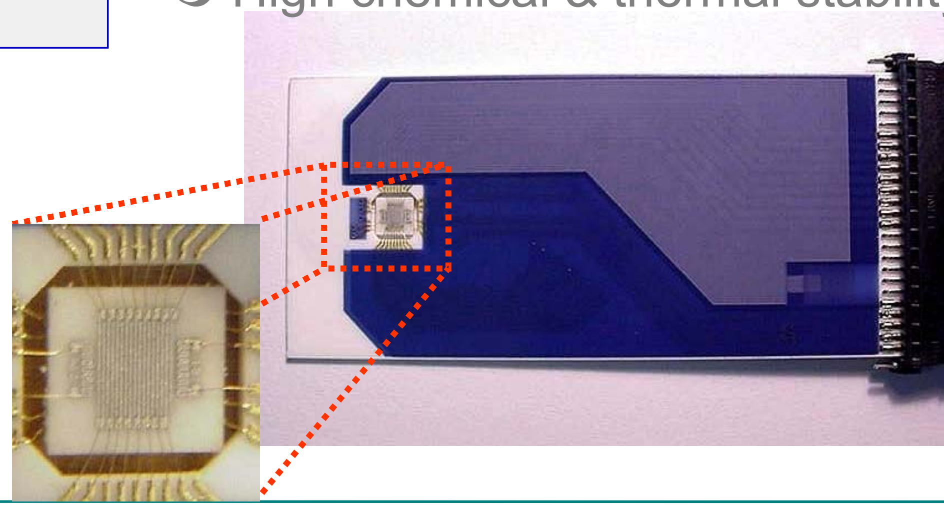
Inexpensive, small, excellent mechanica
stability, low power

Phys. differentiation >>
collective ageing
Gradient technique: Reliability checks
repairs, Noise reduction without extended measuring time

KAMINA Microarray Dwarf Chip

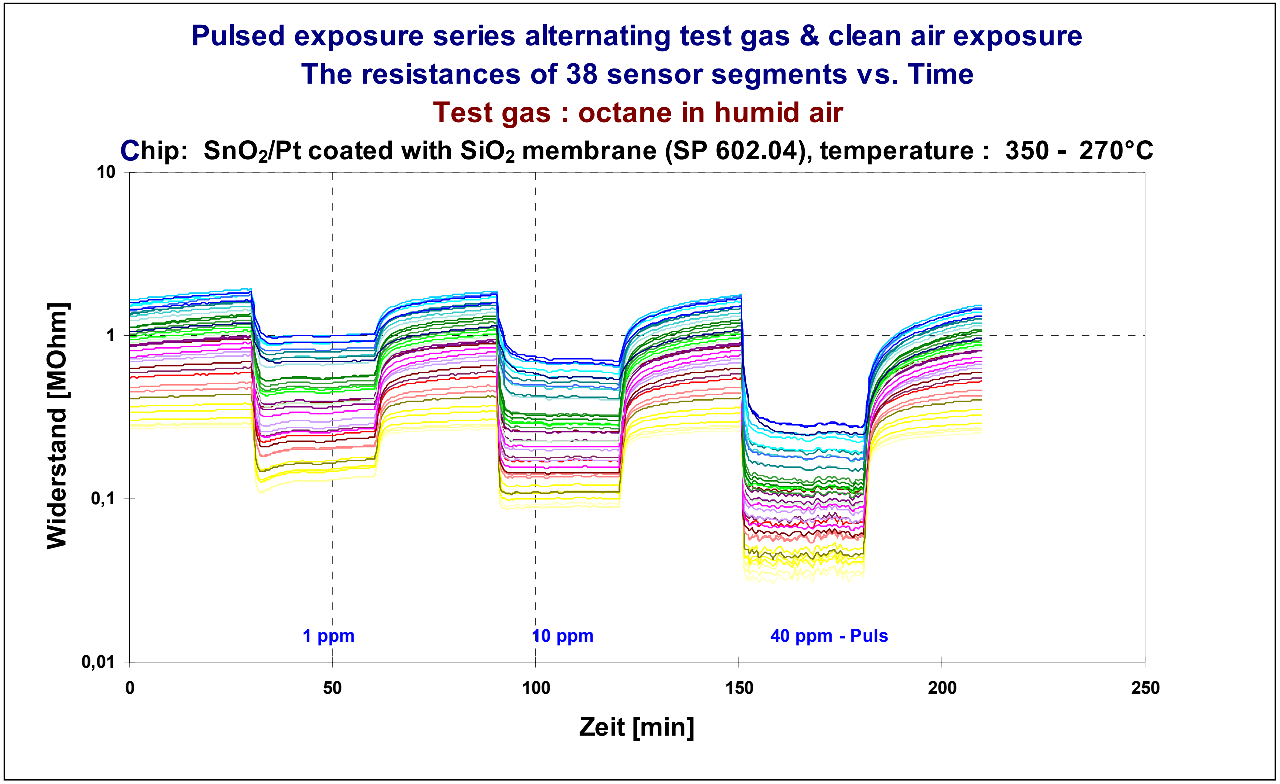


- Substrates: SiO_2/Si, Al_2O_3
- Metal oxide layer: SnO_2/Pt, WO_3/Au
- Gradient membrane layer consisting of SiO_2 or Al_2O_3
- Detection limits < 1ppm
- Power consumption < 1 Watt
- High chemical & thermal stability



- Microarray chip fixed only by bond wires within clearance of ceramic carrier card
- Chip corners attached to edges of the opening achieves vertical stability

Raw Data Obtained in Pulsed Test Gas Exposure Series

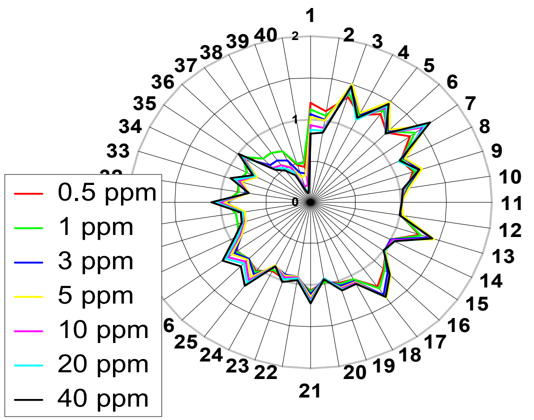


- High sensitivity, usual detection limits < 1ppm
- Vast range of detectable gases
  only inert gases such as rare gases, nitrogen cannot be detected

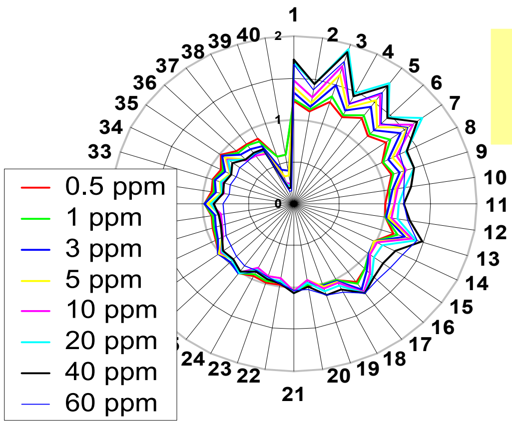
Polar diagrams of normalized signal patterns

Signals of the sensor segments displayed as deviation from median

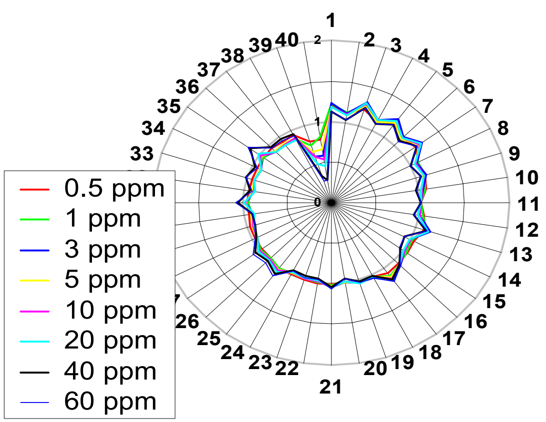
Octane
Sensor signal S=DG/G normalized to median



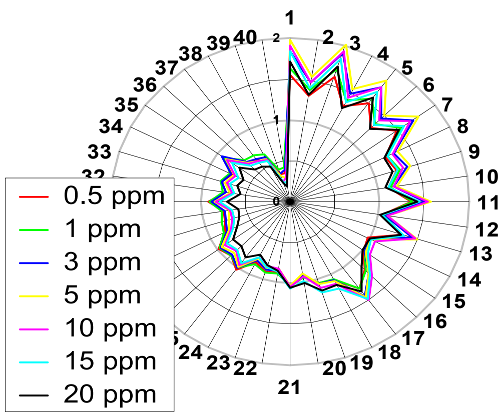
Xylene
Sensor signal S=DG/G normalized to median



Tetrachloroethene
Sensor signal S=DG/G normalized to median



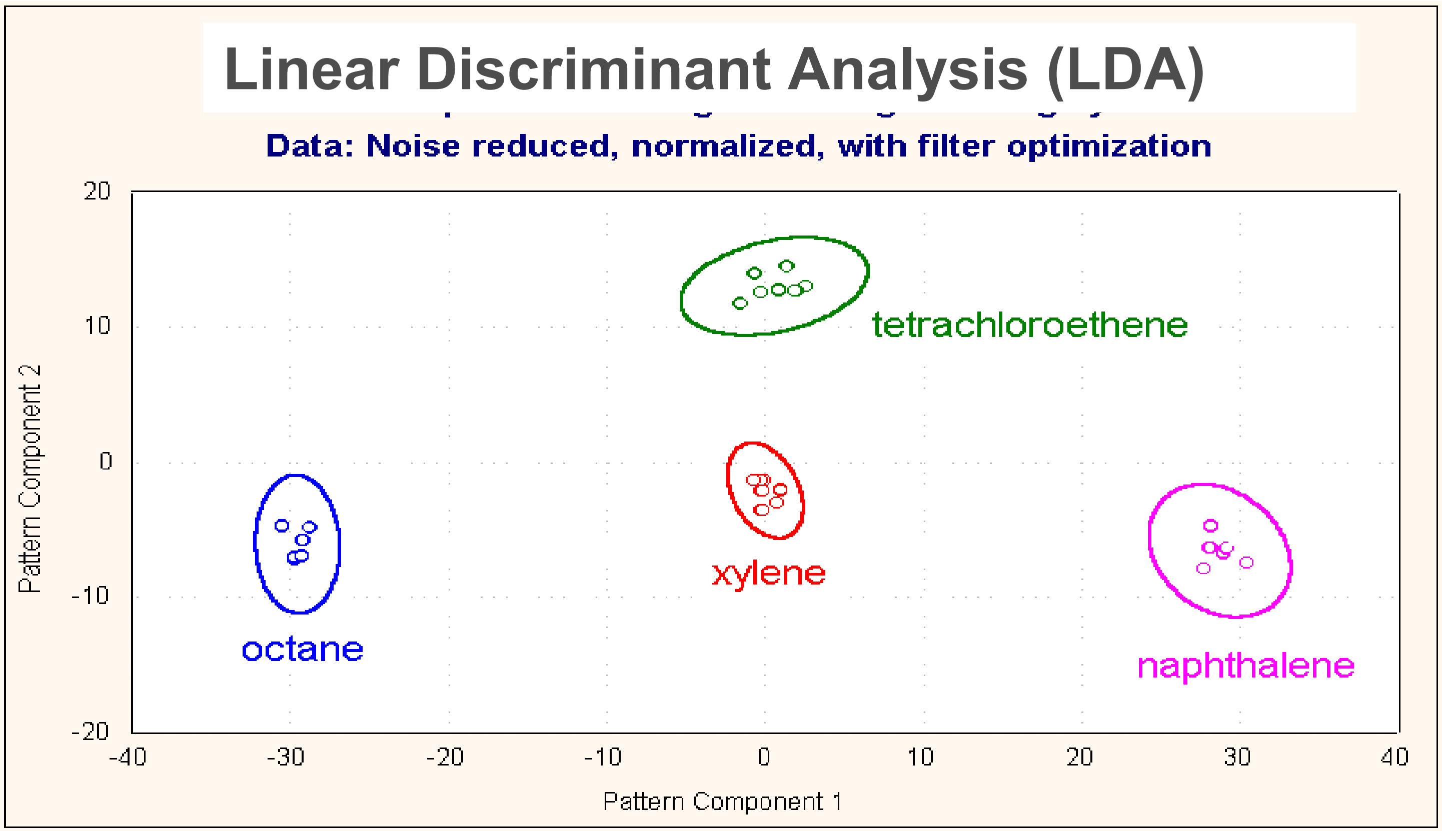
Naphthalene
Sensor signal S=DG/G normalized to median



- **Gas characteristic signal patterns independent from concentration**

Signal Pattern Analysis: Training And Recogintion

- Original signal patterns exist in 38 dimensional signal space
- Training assigns pattern areas belonging to gases or gas ensembles
- Measured signal patterns are classified for coincidence: gas recognition or “unknown”)



LDA is the optimum projection to show the differences of the trained classes

KAMINA Module

The KAMINA module combines gas sampling, microarray chip and µP-controlled operating electronics

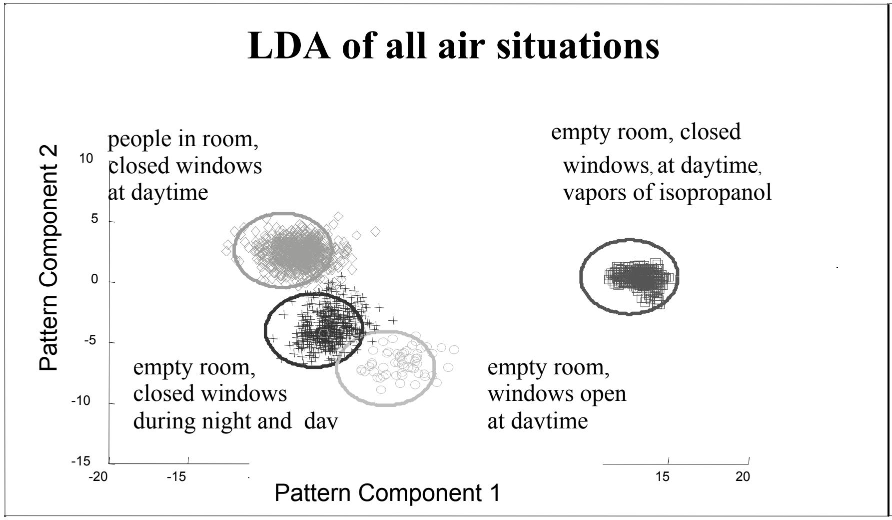
The electronics provide for:
- measurement of sensor segments at 1 Hz
- supplies chip heating & gas sampling
- serial data interface > computer control
- relais outputs

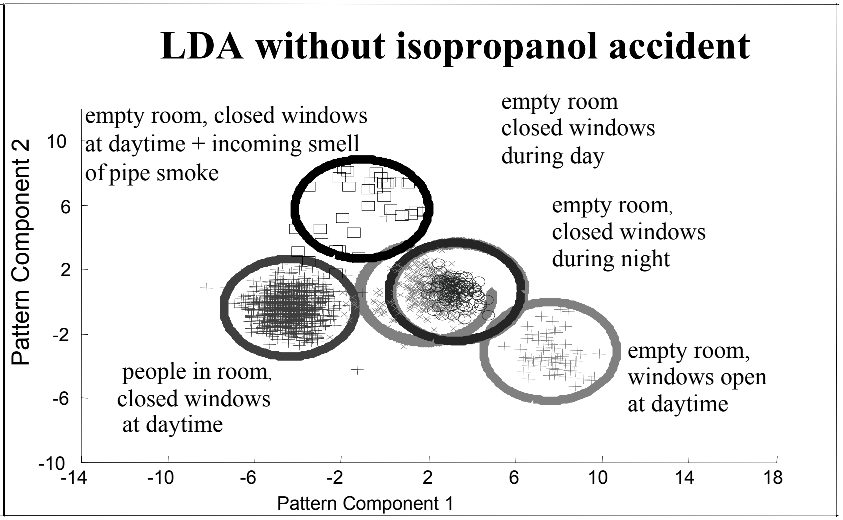
Gas sampling via ventilator or micro-pump

Indoor Air Monitoring

Linear Discrimination Analysis of signal patterns obtained in a conference room for about 30 people:

- Measurements carried out in empty room day and night 1 m above the floor
- 2 meetings of 3 hours each (second meeting held a week later) with approx. 20 people
- Hazard simulation: isopropanol on paper tissue approx. 0.8 m from point of measurement



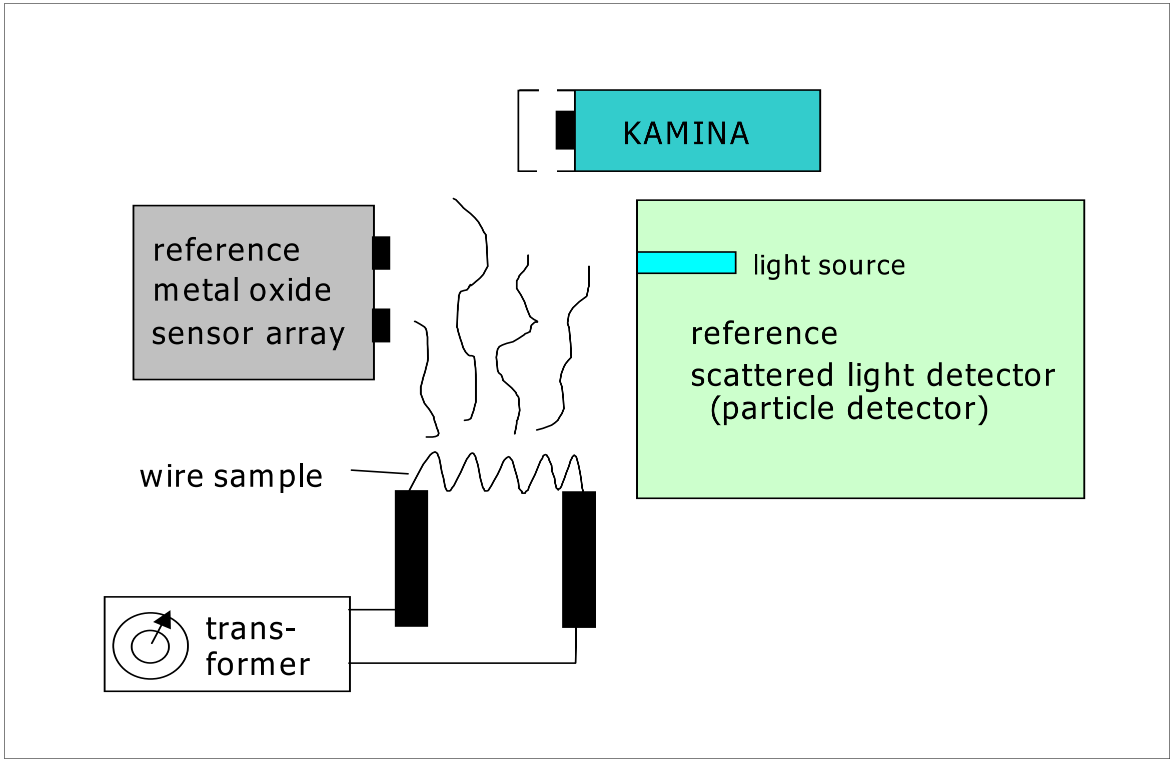


Decreasing air quality caused by the presence of people can be detected
Dangers caused by gases can be realized

Set-up to detect overheated wire insulations

Joined Project with NASA/Kennedy Space Flight Center:

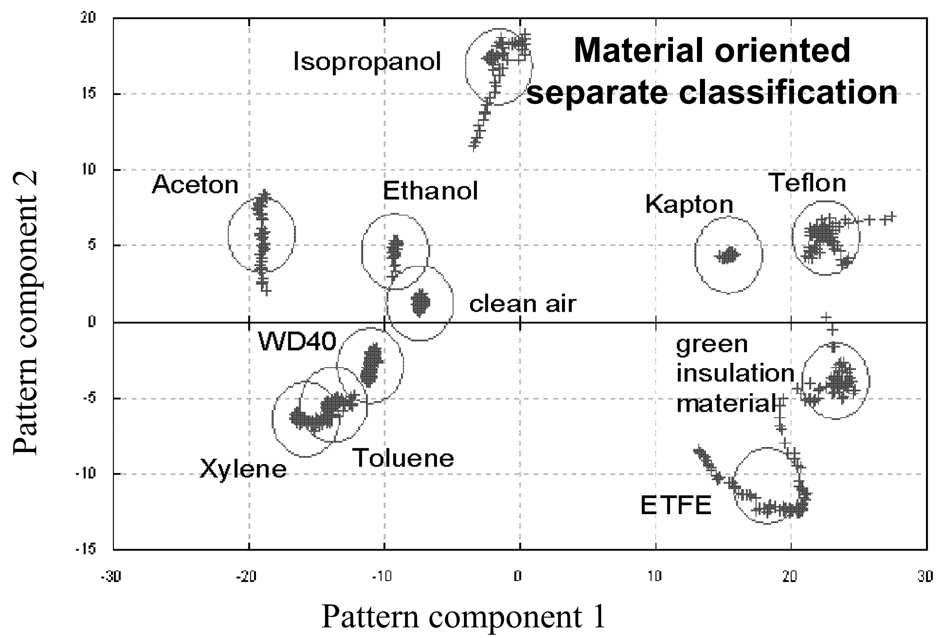
Early detection of cable smouldering in Space Shuttle and International Space Station

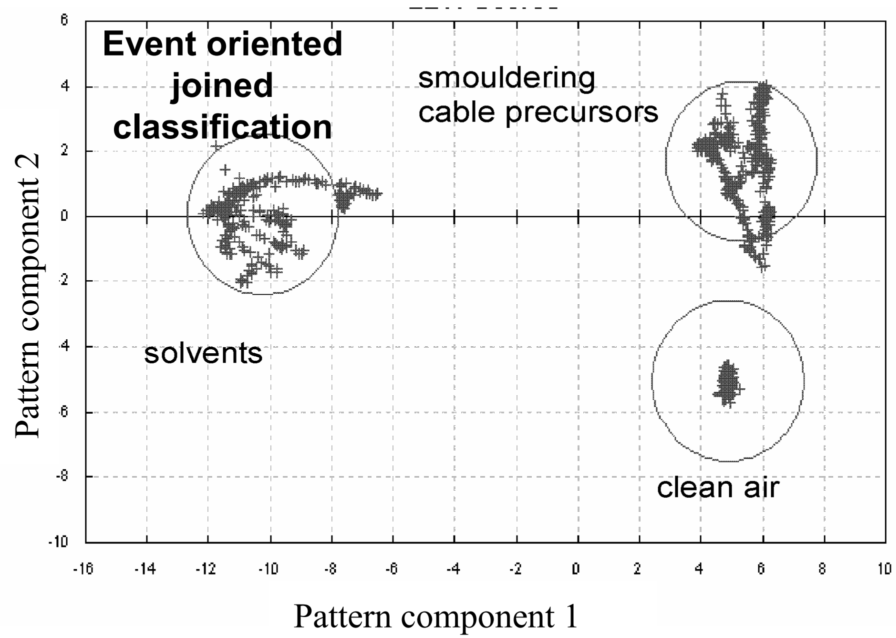


- Wires with different insulation electrically overheated in closed box
- Vapor exposures from tissues soaked with solvents as possible interferants
- Reference analysis: Conventional sensor array with separated sensors and scatterd light detector

Detection of Overheated Cable Insulation to Prevent Smouldering Fires

Joined Project with KSC/NASA: Fire prevention in Space Shuttle and the Intern. Space Station





- All insulations can be distinguished by the gas release during heat up
- All overheated insulations can be classified together as smouldering event and discriminated against normal air or solvent release events

Indoor Air Monitoring
Distinctive & Early Detection of Fire Accidents

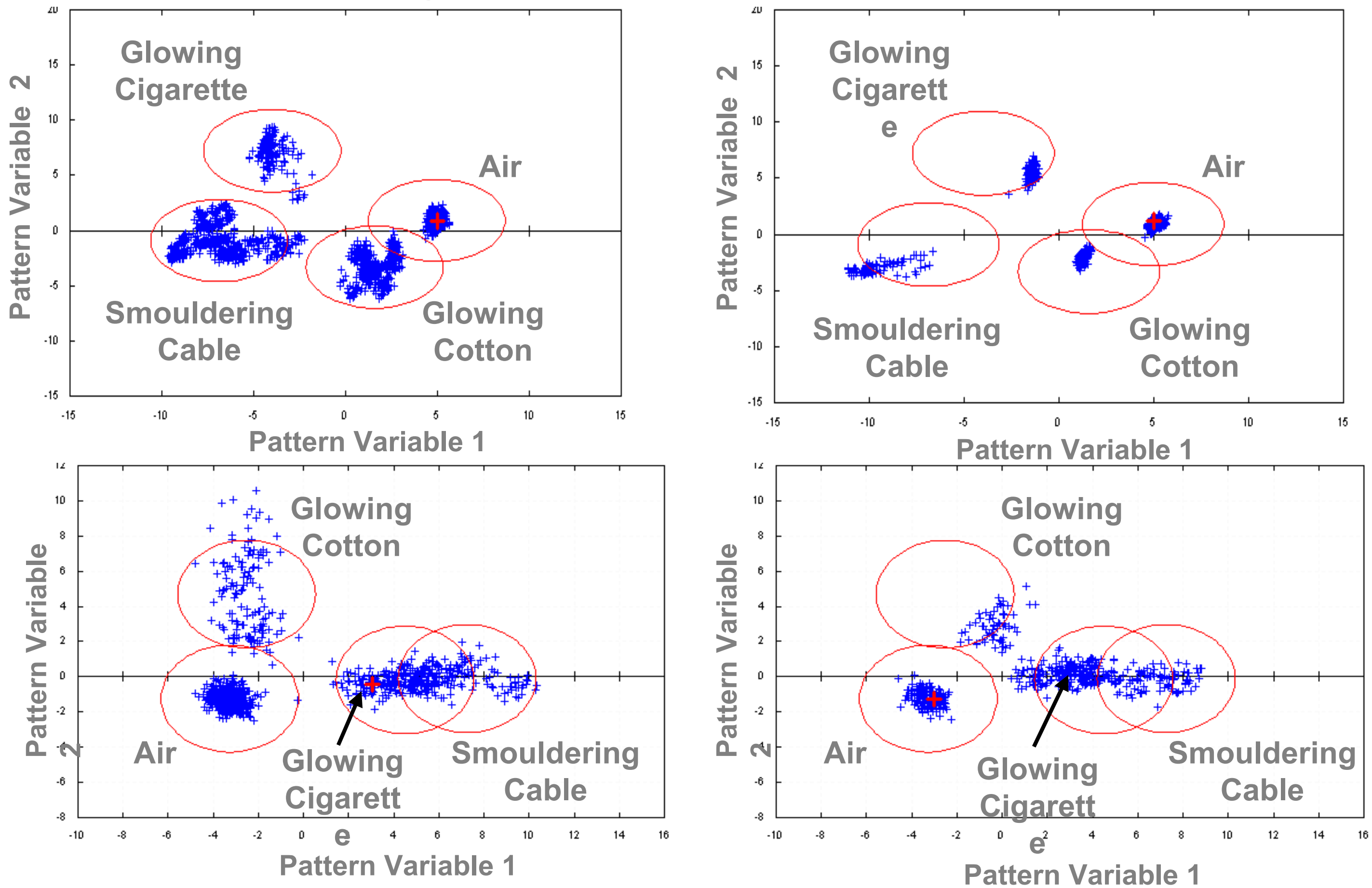
3X3.5 mm^2 Microarray with 16 sensor segments
SnO$_2$/Pt detector layer and
Al_2O_3 gradient membrane

➢ **The Al_2O_3 membrane allows better gas discrimination**

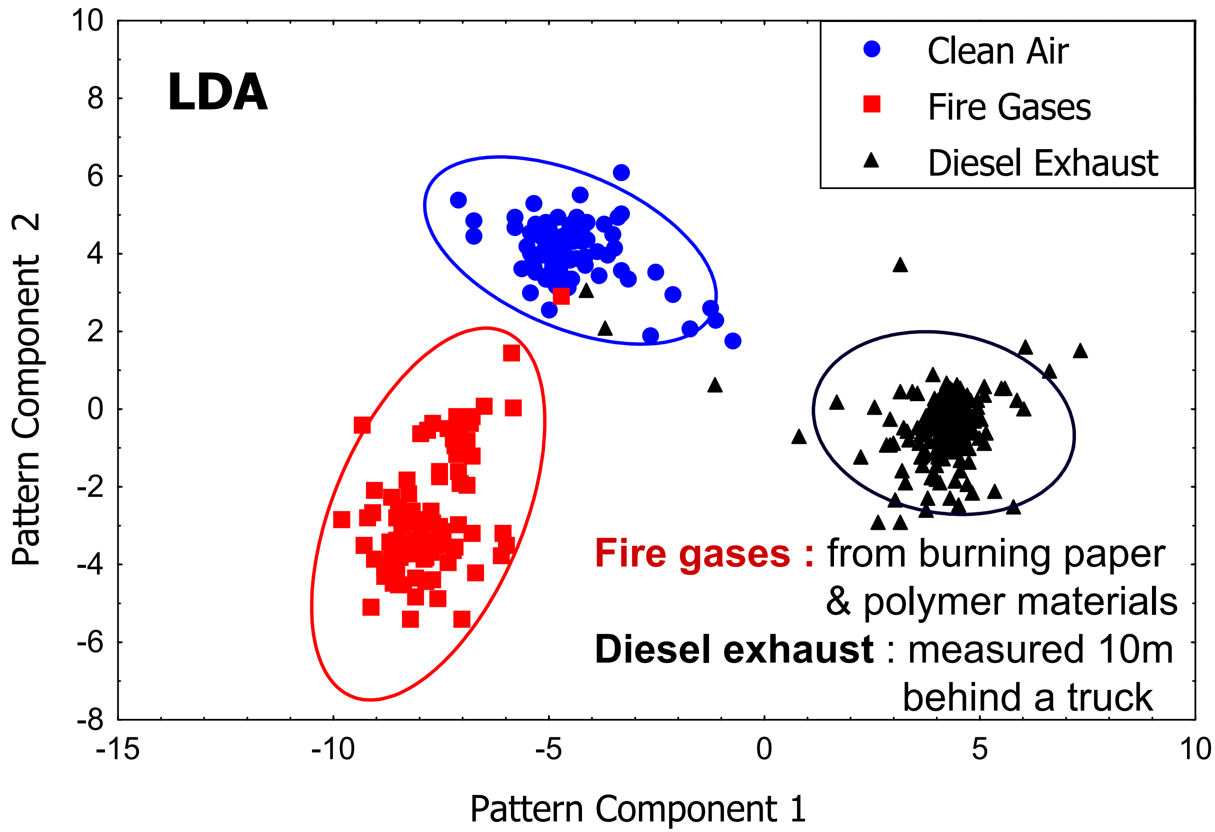
8X9 mm^2 Microarray with 38 sensor segments
SnO$_2$/Pt detector layer and
SiO_2 gradient membrane

Modelling

Model test



Fire detection: High discrimination power in fire detection



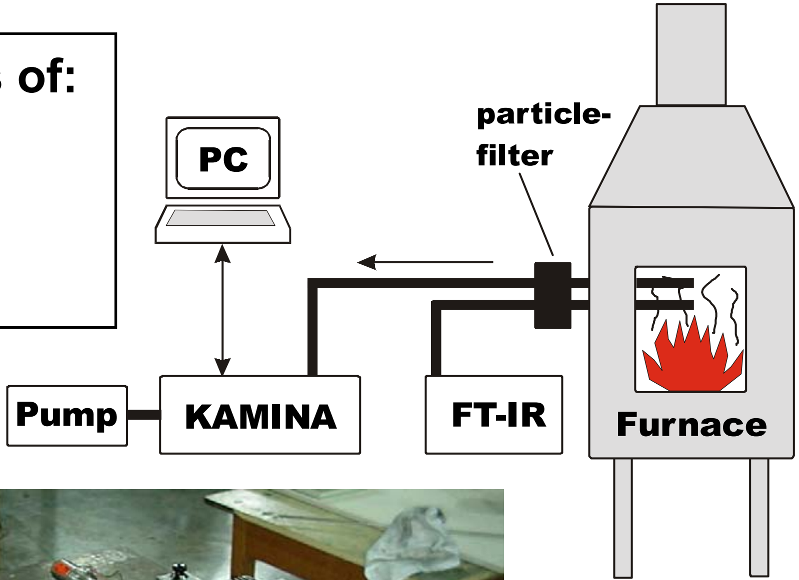
- Diesel exhaust contains CO, NO_2 and soot particles

> False Alarm: Often classical fire detectors are cheated by Diesel exhaust

- Fire gas is clearly recognized
- Diesel exhaust can be well separated from fire gas

Experimental setup for tests at model fires

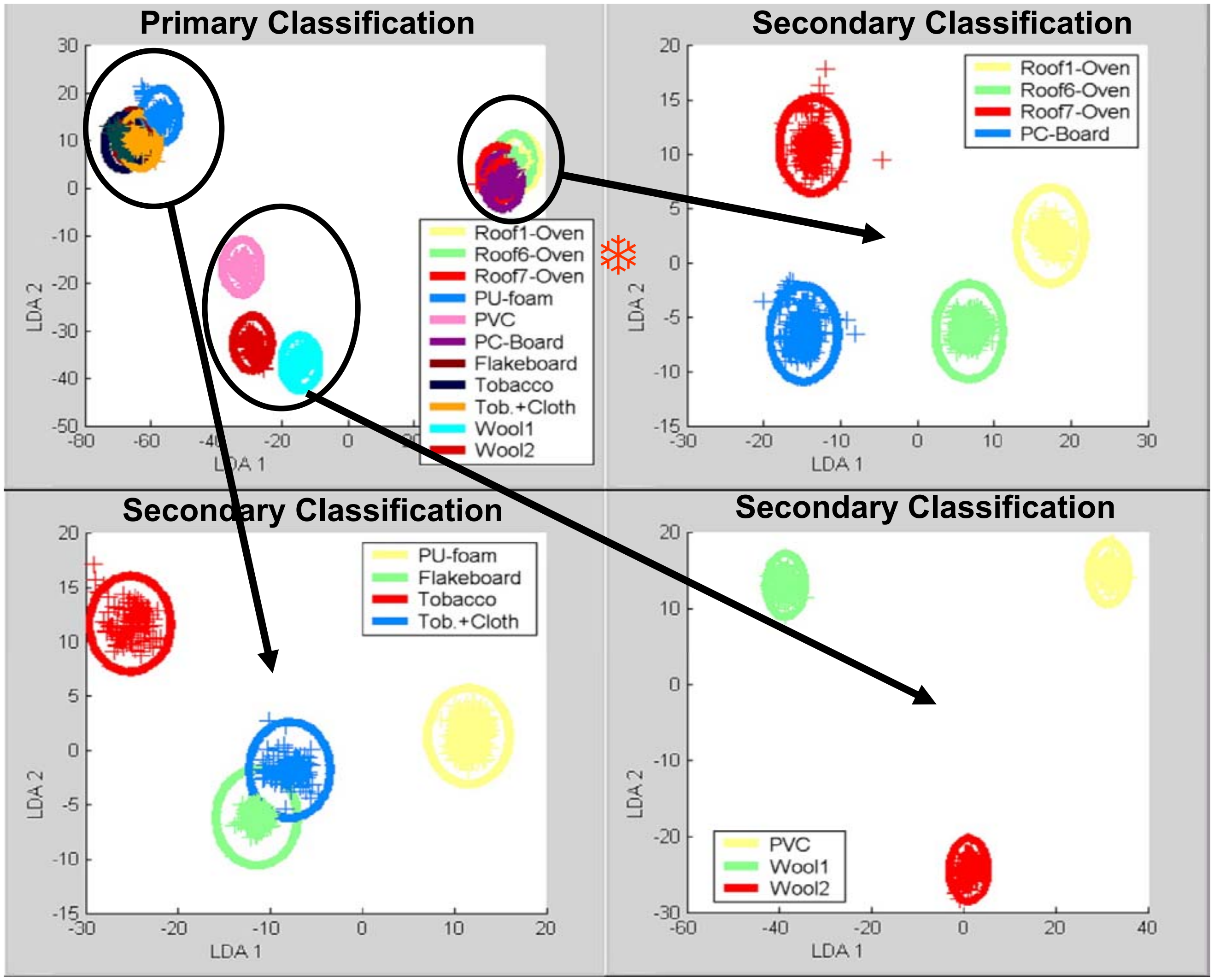
Burning & pyrolysis of:
wood, plastics,
printed circuit board,
tobacco, wool, roof
materials.



KAMINA: Data acquisition with 1 Hz
0.4 lit/min gas flux

FTIR: TEMEL/Finland Type GASMET
Spectrum span 800-4000cm^{-1}
Spectrum duration 20 or 60sec
3 lit/min gas flux

Discriminative power for material recognition



Materials pyrolized:

- Bituminous roofing felts:
  7 types with different fire retarding additives
- Polyurethane foam
- Polyvinylchloride rods
- Computer circuit board
- Wood composite (Flakeboard)
- Cigarette tobacco
- Cigarette tobacco .+ textile:
  to simulate "smoking in bed"
- Wools: 2 Wools with different additives

Stepwise LDA based Classification

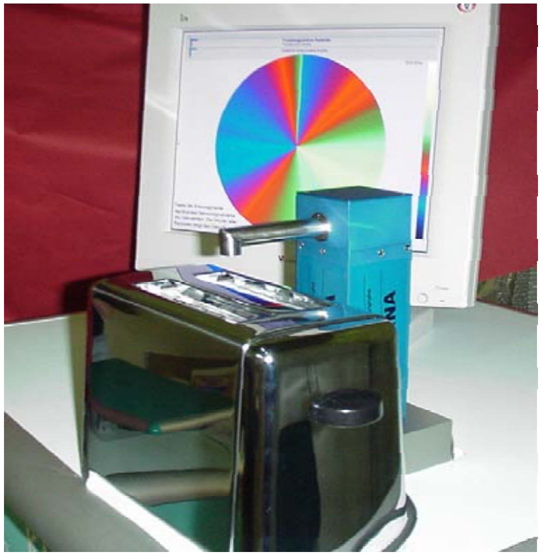
1st step: Primary Classification
into 3 major material classes

2nd step: Secondary Classification
LDAs of each class

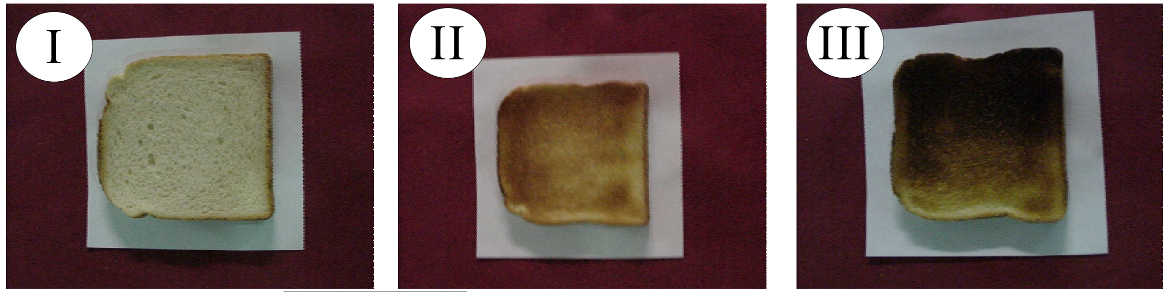
⇨ detailed recognition

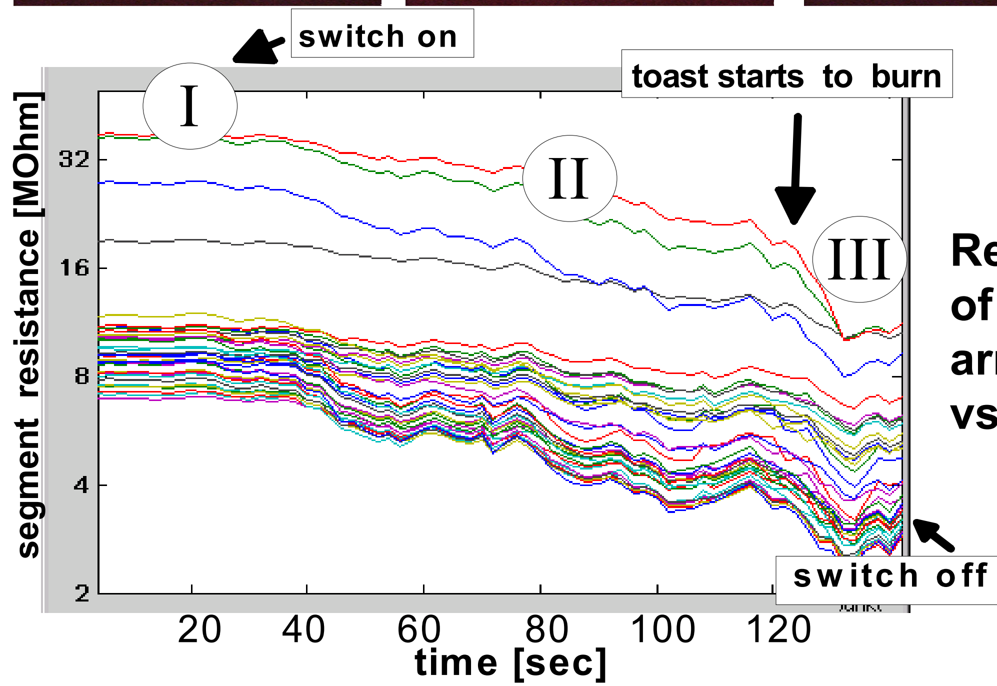
➢ **Detailed Classification of pyrolized materials feasible**

Controlling a Toaster



Toaster with KAMINA



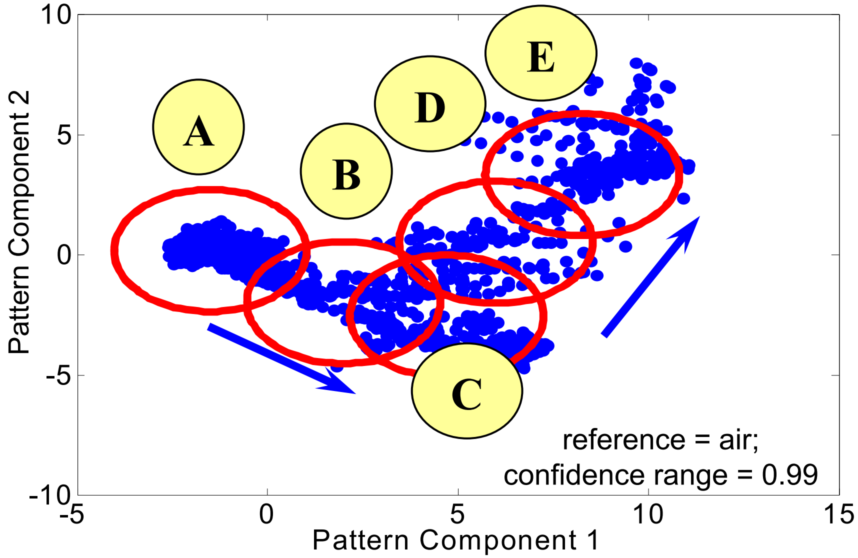


Resistances of the micro-array segments vs. process time

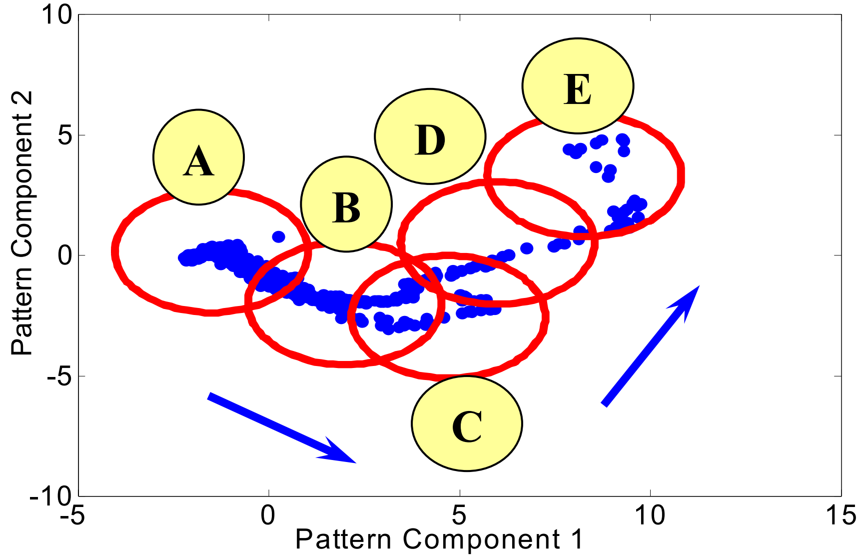
Controlling the Toaster Process

Linear Discrimination Analysis (LDA) of Median & Reference Normalized Data

Advancement classes of process states:
A= raw bread B= light brown C= medium brown D= dark brown E= burned



Training: Model built from 9 measurements done at Karlsruhe



Model testing: 2 measurements done at HOMETEC fair in Berlin one week after training

Application Areas Of The KAMINA Under Development and Test

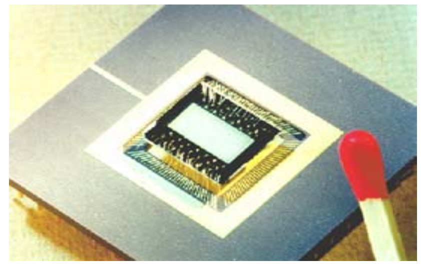
- **Household Appliances**
  Food processing, Laundry screening
- **Commercial Food Process Control**
- **Building Technology**
  Indoor air monitoring: pollution and odor determination, Heating system control
- **Automobil Equipment**
  Air conditioning, Onboard exhaust gas monitoring
- **Space Ship Technology**
  Cable smoldering, Air lock surveillance
- **Criminal Investigations**
- **Environmental Analytics**
  Air, Water, Soil
- **Biogas-Monitoring**
- **Medical Diagnosis**
  Breath and sweat odor Analysis
- **Fire Protection Technology**
  Early detection, material recognition, fire gas dissemination
- **Metal Processing**
  Biocide monitoring of lubricants
- **Textile Processing**
  Fabric recognition, impregnation degassing

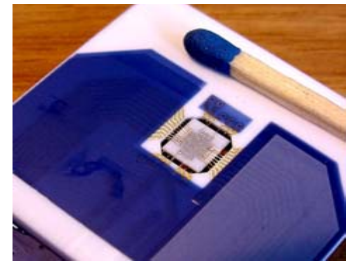
Electronic Nose Microsystems
Have the potential to be used as
Indoor air monitors applied for

- prenatal fire detection
- open fire detection
- burning material recognition
- air quality measurements
- odor comfort monitoring

KAMINA shows how
Low cost, low size
Can be combined with
High gas analytical power

Status & Prospect





Fabrication of the KAMINA at present in small series production for development & demonstration purposes.

Estimated cost in mass production:
KAMINA-Chip < 5€
Operating electronics < 20€

Set-up of commercial production underway
Variety of application projects in work

Questions

?

?

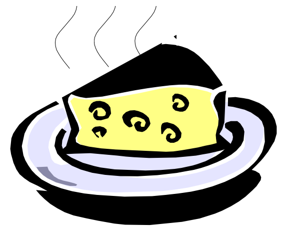
?

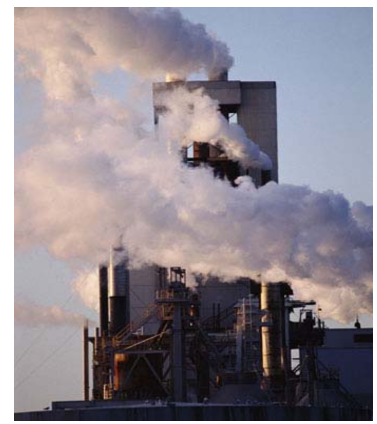
Widespread Applications

Size & price

Off-line applications

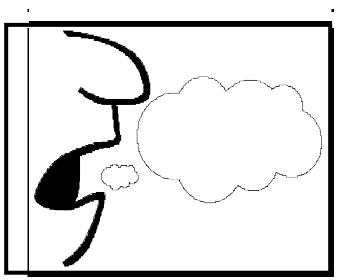
Product (food) quality control, scientific applications





Monitoring Moduls For Industrial & Environmental

Online production control, Work place monitoring
Environmental monitoring (e.g. air quality) > onsite

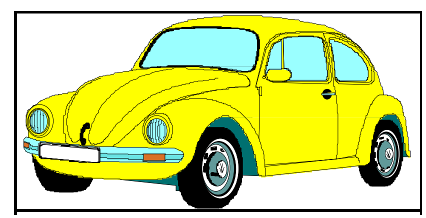


Medical Products & Diagnosis

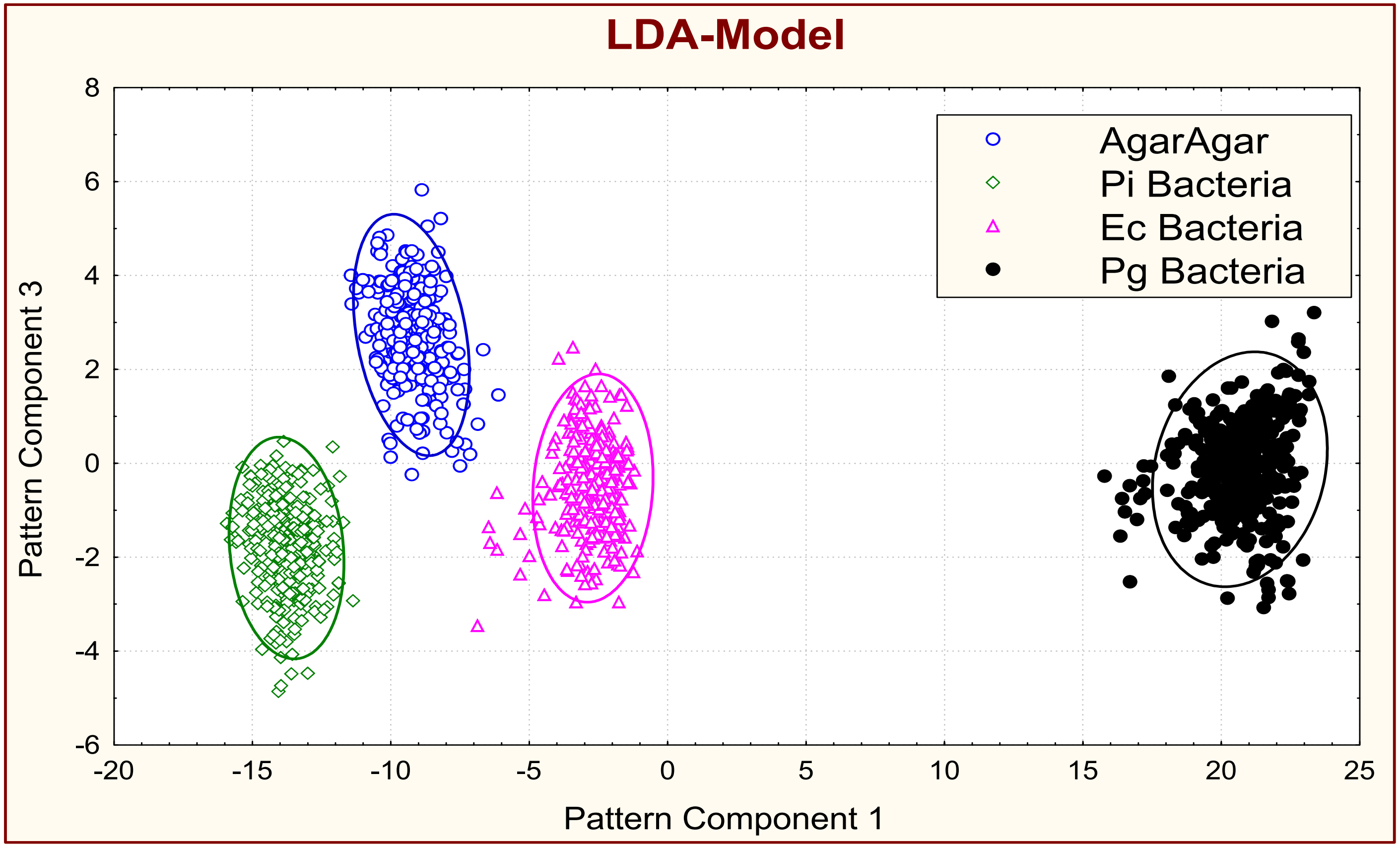
Analysis of breath, skin odor, and body fluids, pharma products

Mass Products

Automobiles, building technology, household appliances

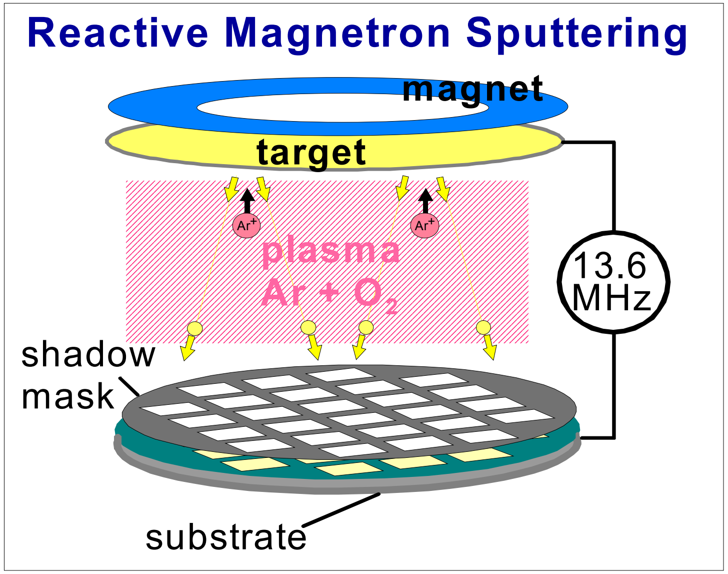


Discrimination of Paradonditis Bacteria For Medical Diagnosis



- **Even slight differences in the gas release of similar bacteria can be distinguished allowing the distinction of the bacteria**

Standard Microfabrication in 4 Phases On 3“ Or 6“ Si Substrates



I. HF Sputter Deposition With Shadow Masks

- Gas detecting MOx layer
- Electrode Pattern
- Heating elements

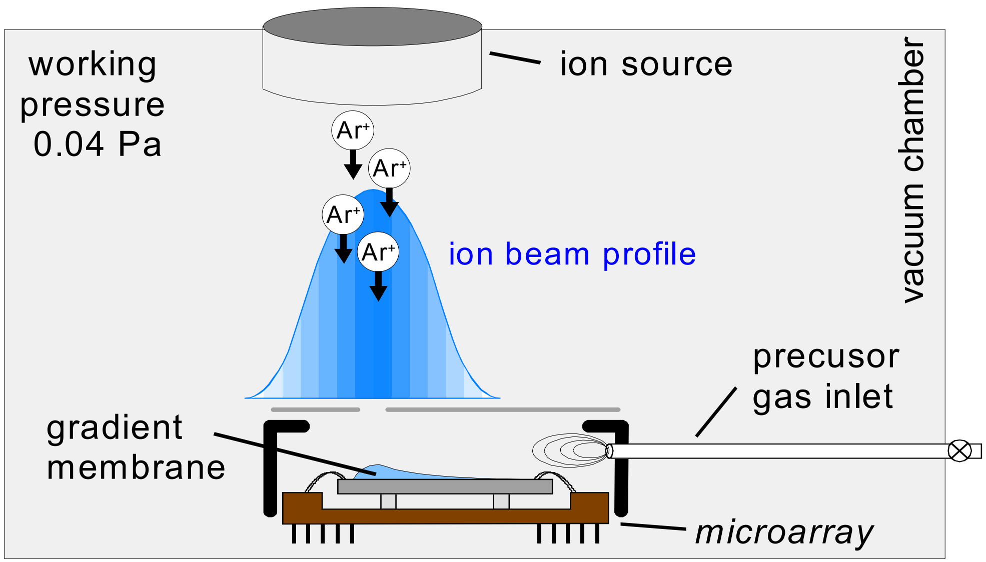
Detection limits 0.05- 10ppm
Response times ca. 1 min

II. Dicing & Assembly

- Separation of the chips
- Mounting on carrier
- Electrical contacts by wire bonds

III. Ion Beam Assisted Deposition (IBAD) To Deposit Inhomog. Membrane Coatings

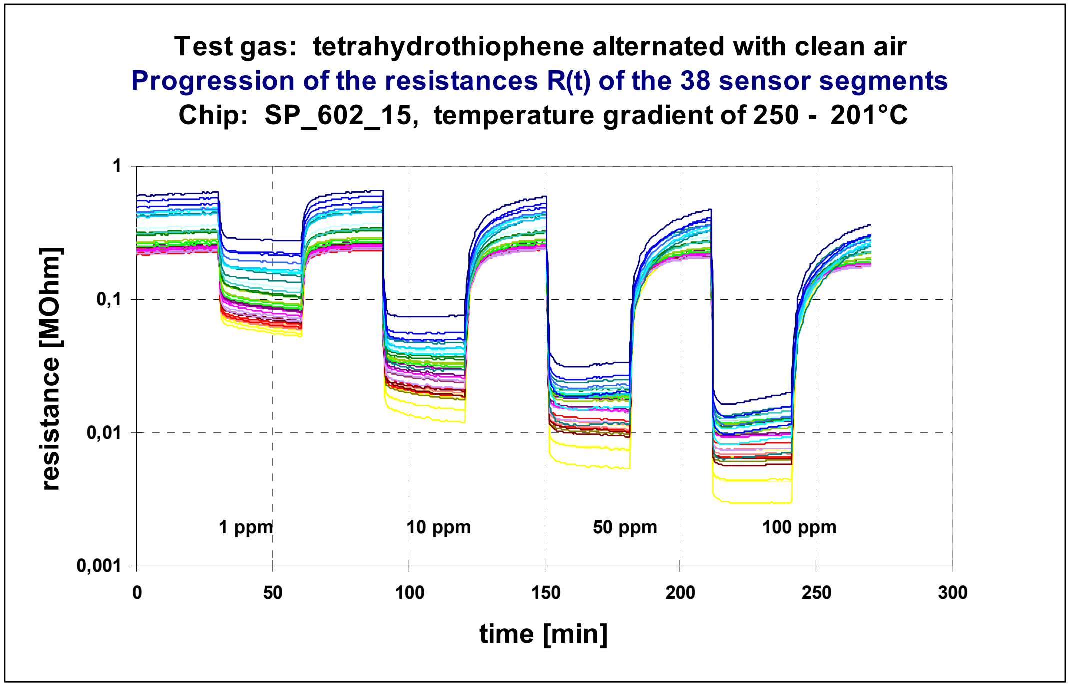
Ion beam converts at RT condensed process gas phenyl-TEOS to some nm thick Si,O,C film



IV. Final Conditioning

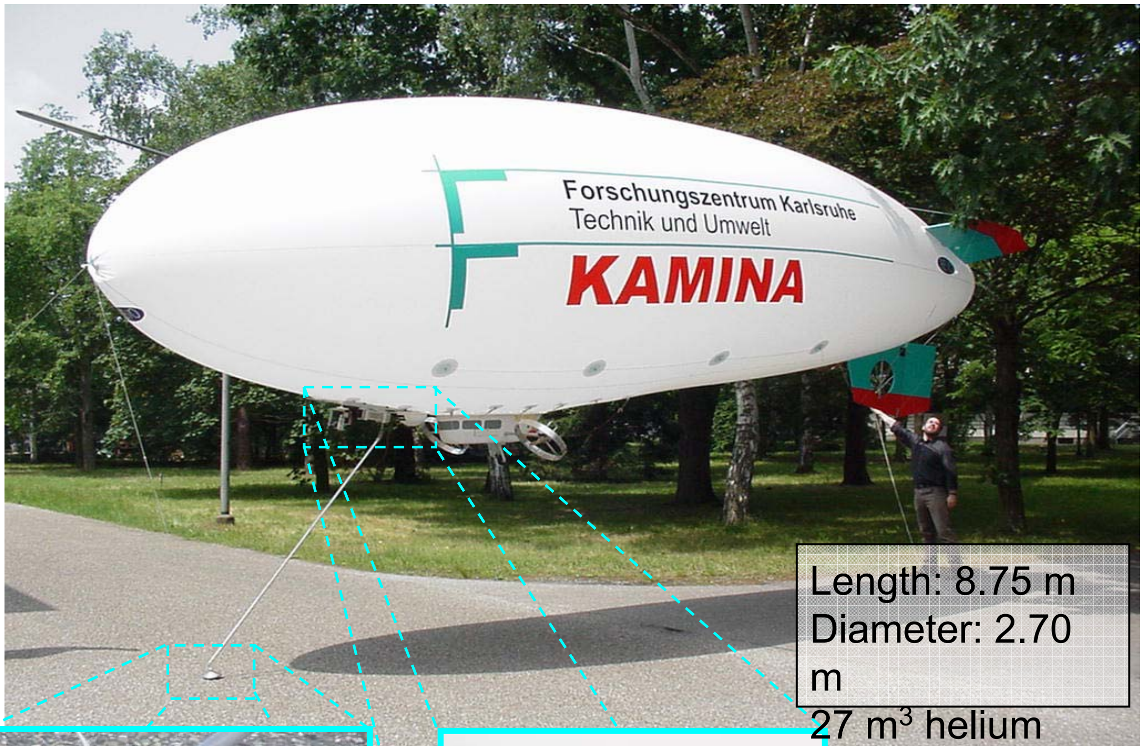
Annealing to give pure gas permeable SiO_2 by removal of residual amounts of C und stabilisation of the morphology

Raw Data Obtained in Pulsed Test Gas Exposure Series

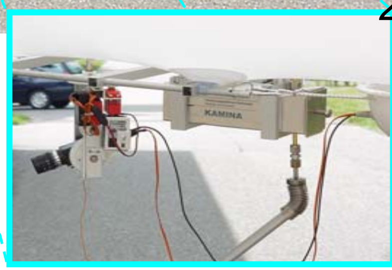


- **High sensitivity, usual detection limits < 1ppm**
- **Vast range of detectable gases**
  only inert gases such as rare gases, nitrogen cannot be detected

LENA: Airship Equipped with an Electronic Nose



Length: 8.75 m
Diameter: 2.70 m
27 m^3 helium



- **Mobile Electronic Nose combined with positioning option provides powerful analytical tool:**

⇨ **Determination of spatial gas distributions**
Pollutant gas ensembles from motor traffic, odor clouds, fire gases

⇨ **Localization of gas sources on the ground**
Gas leaks in industrial facilities, smelling objects on landfill sites, odor sources in agriculture, infestated areas of plant deseases, land mine detection

Operational data: electrical drive with 3 motors, 37 MHz radio control within sight, max. lateral speed: 60 km/h, payload: 4 kilograms

Instrumentation: KAMINA, video camera, GPS for current positioning and board computer with radio data transmission to ground control ne

ik


DGA
CENTRE D'ESSAIS
AERONAUTIQUE
DE TOULOUSE
The Fourth Triennial
International Aircraft Fire and Cabin Safety
Research Conference