Développement de micro-préconcentrateurs pour l'analyse de traces de gaz et explosifs.

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Outline

Micro-preconcentrators

- **Context / Principle**
- **Design and fabrication**
- **Adsorbent characterization / Parameters**
- **Applications**
  - VOC preconcentration test, GC analysis
  - Explosive detection
- **Conclusions**

Introduction: context

Challenge : Gas trace detection with sensor or analyzers with limited sensitivity
Miniaturized system for in situ detection

- Conventional trap (preconcentration tube)

Reduce dead volume and thermal mass

↓

MEMS technology
Microfabricated preconcentrator

- Miniaturized analyzer : Micro-GC module

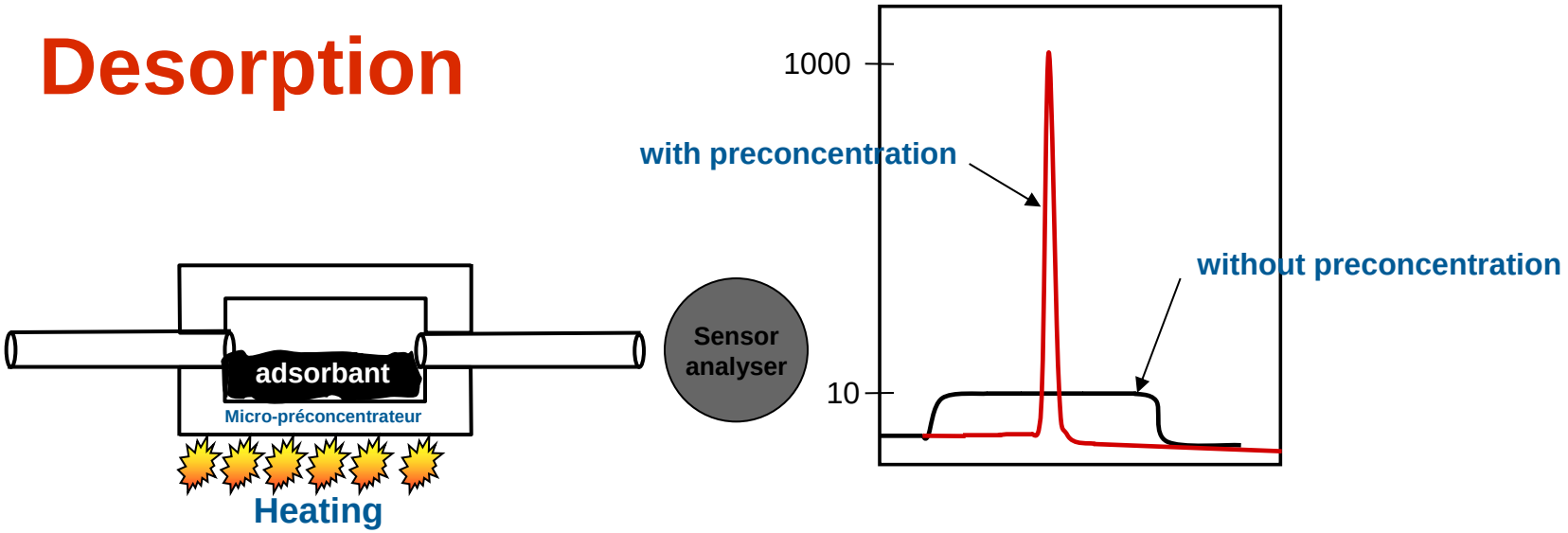
Miniaturized system : Micro-GC /Micro-preconcentrator coupling

Introduction: preconcentration principle

Process : Accumulate a target gas, then desorbed it by a temperature pulse

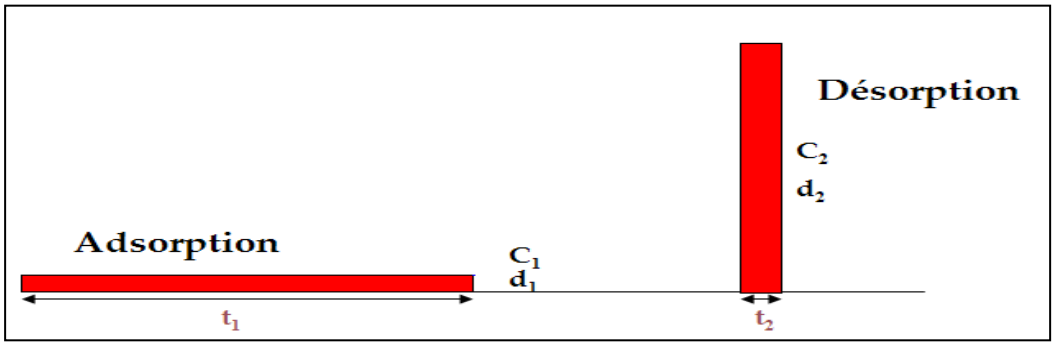
Gas pre-conditioning is of importance for the detection of chemical substances in trace level.

Desorption



Introduction: Preconcentration factor

Preconcentration factor : ratio of outlet to inlet concentration

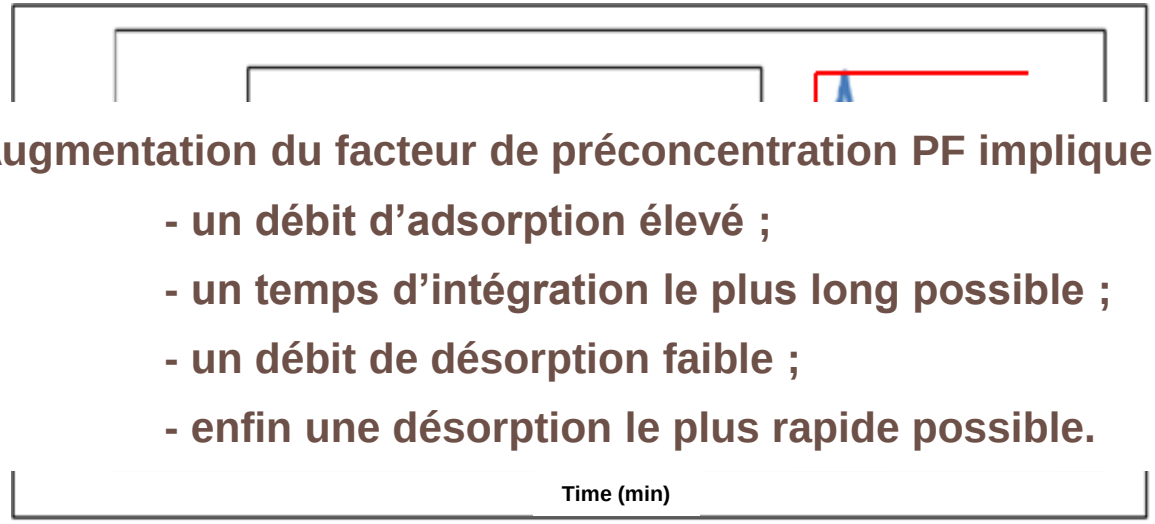


Si t_1 < temps de perçage, conservation de la matière :

$$C_1 . t_1 . f_1 = C_2 . t_2 . f_2$$

C: concentration
t: time
f: flow

Preconcentration factor: $PF = \frac{t_1 f_1}{t_2 f_2} = \frac{C_2}{C_1}$

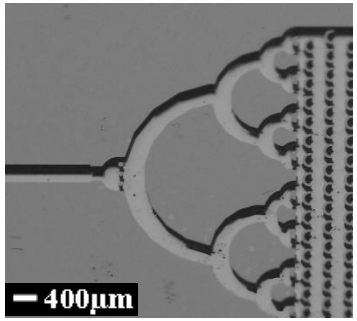


Augmentation du facteur de préconcentration PF implique :

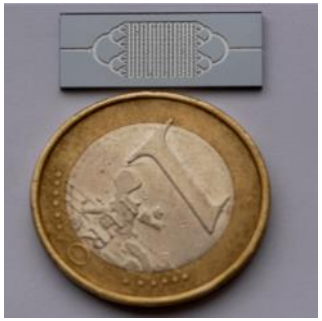
- un débit d'adsorption élevé ;
- un temps d'intégration le plus long possible ;
- un débit de désorption faible ;
- enfin une désorption le plus rapide possible.

Design

Preconcentrator design

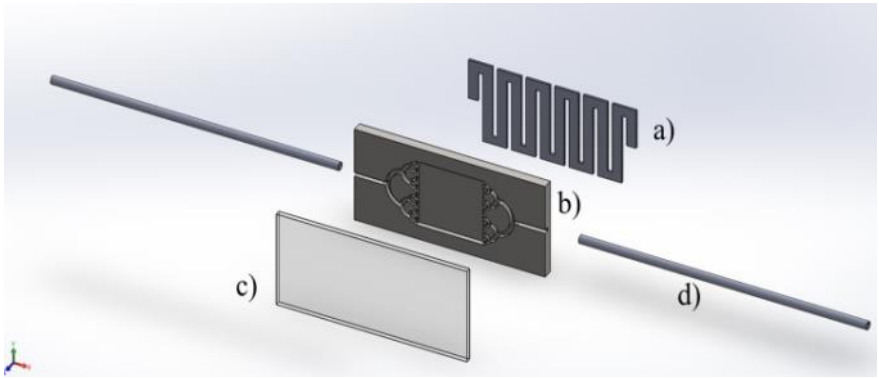


Inlet/outlet channels



preconcentrator/ 1euro

Characteristics	Values
Depth	500µm
Chamber volume	14 µL
External diameter capillary	500 µm
Internal diameter capillary	385 µm
Resistance value	10 Ω



Design and schematic device composition:

a) platinum heater,
b) etched silicon wafer
c) glass cover
d) capillaries

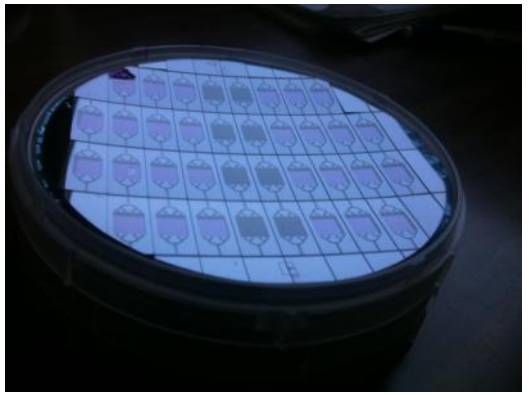
Preconcentrator fabrication

Microfabrication at EPFL, Neuchâtel

Deep Reactive Ion Etching (DRIE)

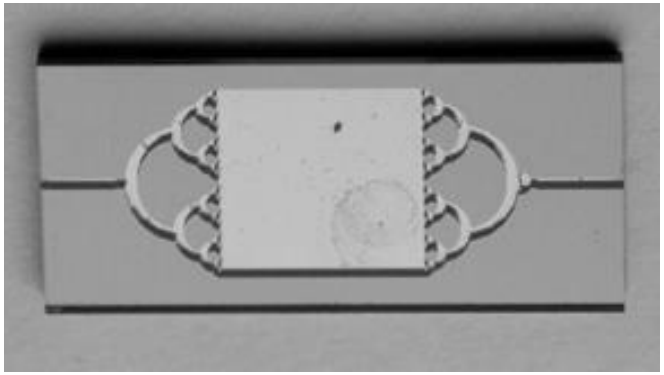
Wafer bonding (Si/Pyrex glass)

- Silicon
- Photoresist
- Pyrex glass



Structured silicon wafer

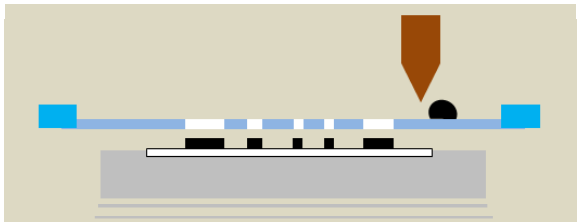
Silicon material
-good thermal conductiviy,
-suitable material for DRIE)

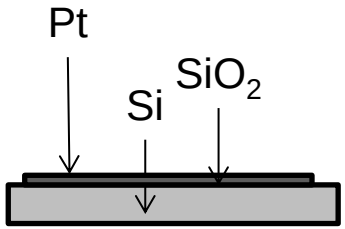


MEMS preconcentrator

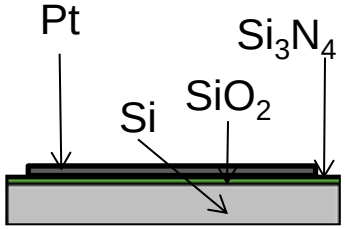
Heater deposition

Heater deposition by screen printing

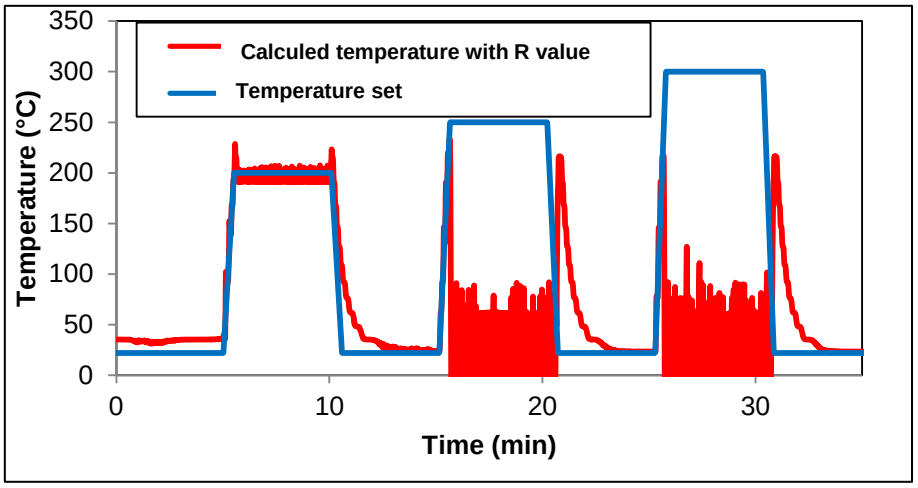


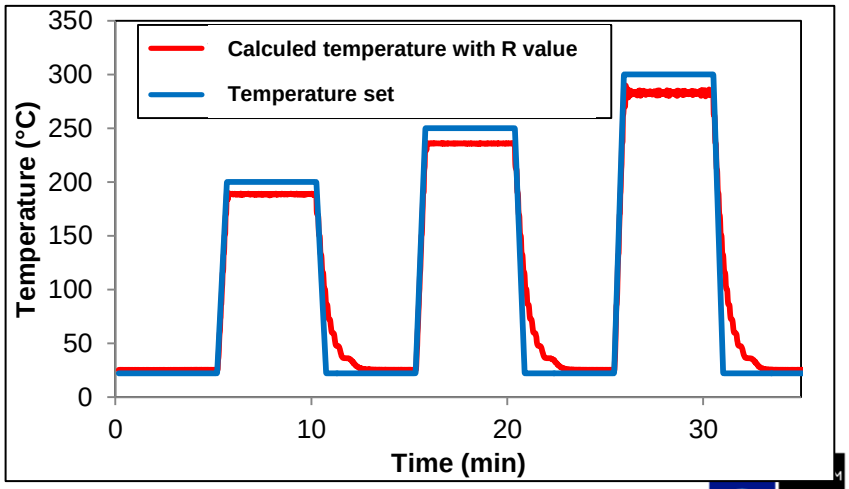


Short circuit due to the silicon conductivity



Better insulation with a layer of silicon nitride

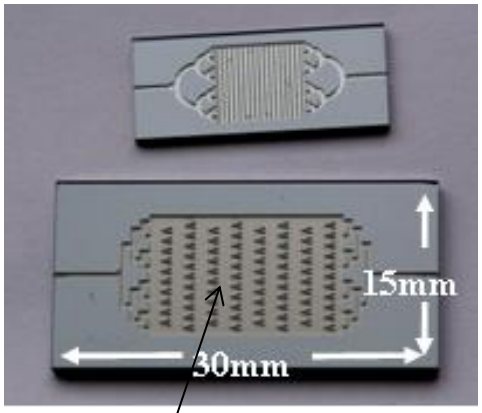




Thèses M. CAMARA (2010)
F James (2015)

Micro-Preconcentrators development

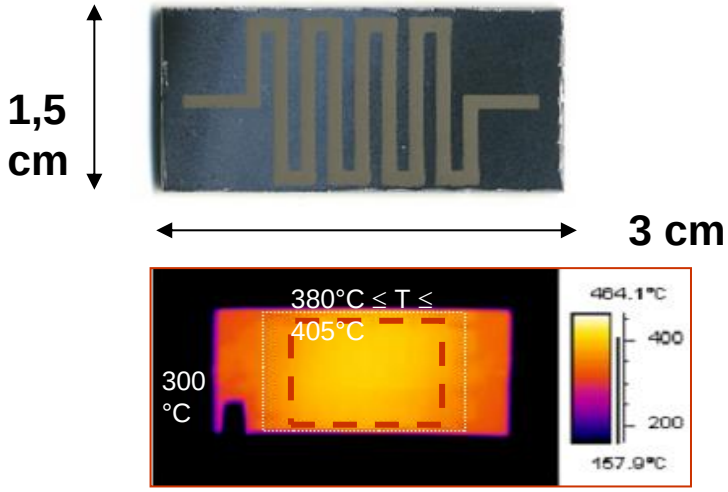
Etched Silicon Microcomponent



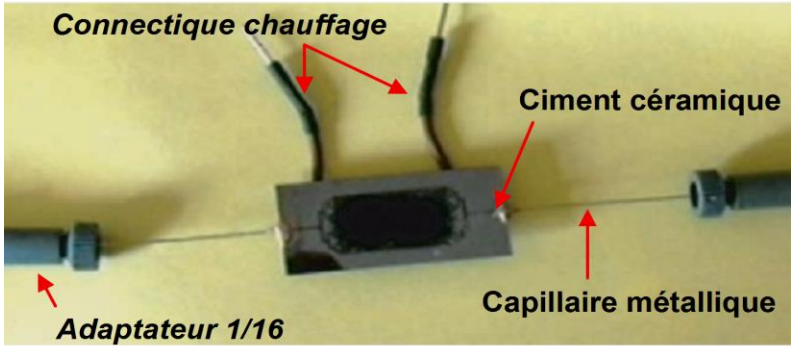
Adsorbent chamber

Metallic capillaries sealed with ceramic cement, with 1/16” connectors.

Platinum heater deposition by screen-printing.



Heating element and temperature distribution

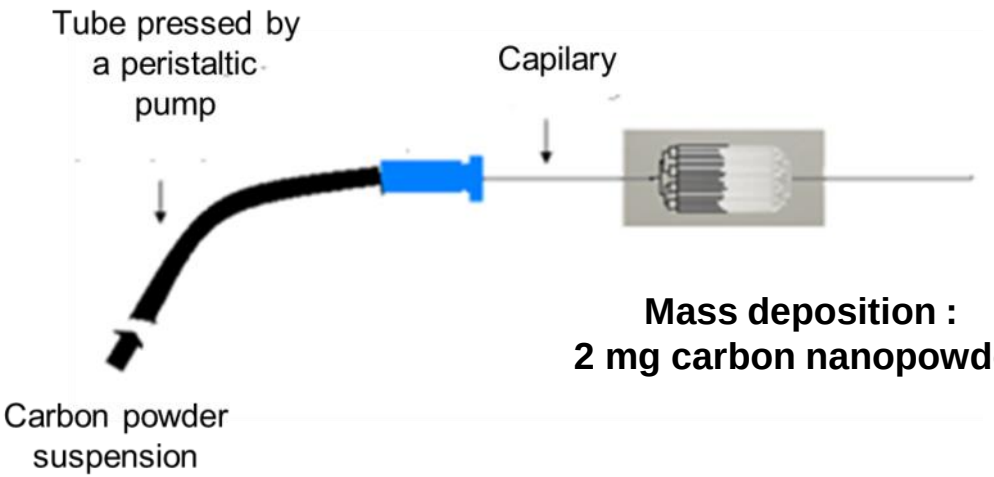


Micro-préconcentrateur device

Capillary : $Ø_{interne}$ 220 µm for 325µm deep IMT micro component .

Sorbent deposition

Carbon Nanopowder deposition by microfluidic method



Mass deposition :
2 mg carbon nanopowder

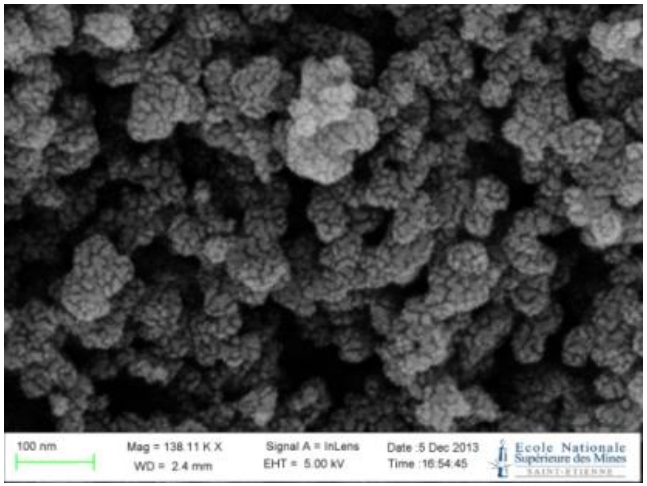
Characterizations	Carbon	Tenax TA
Particule diameter	100 nm	200 µm
Surface Area	95 m^2/g	20 m^2/g

Sorbent material study

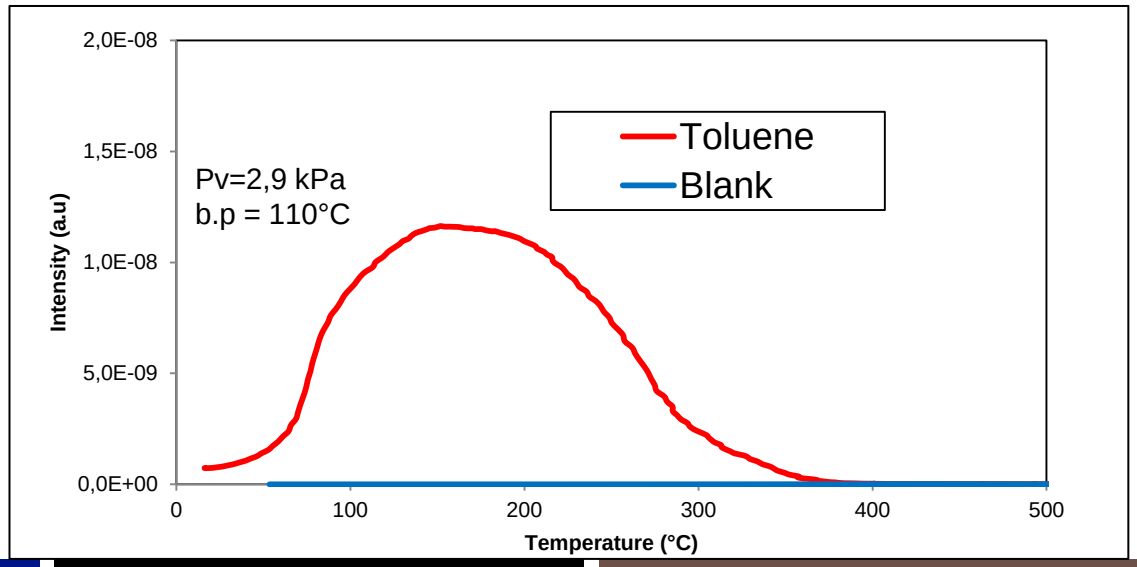
Temperature Programmed Desorption

Thermal desorption of gas species analyzed by mass spectrometry

Carbon nanopowder



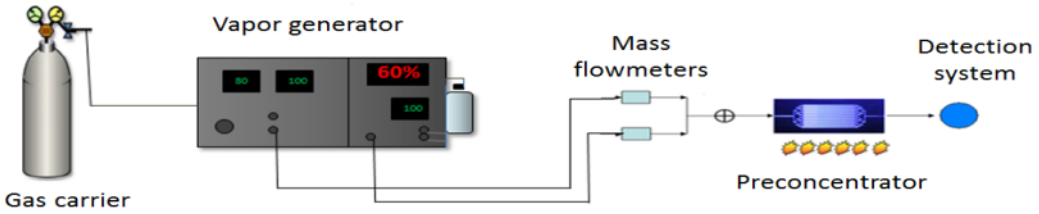
Vapor exposure :
1 ppm of Toluene
during1 hour at 10L/h

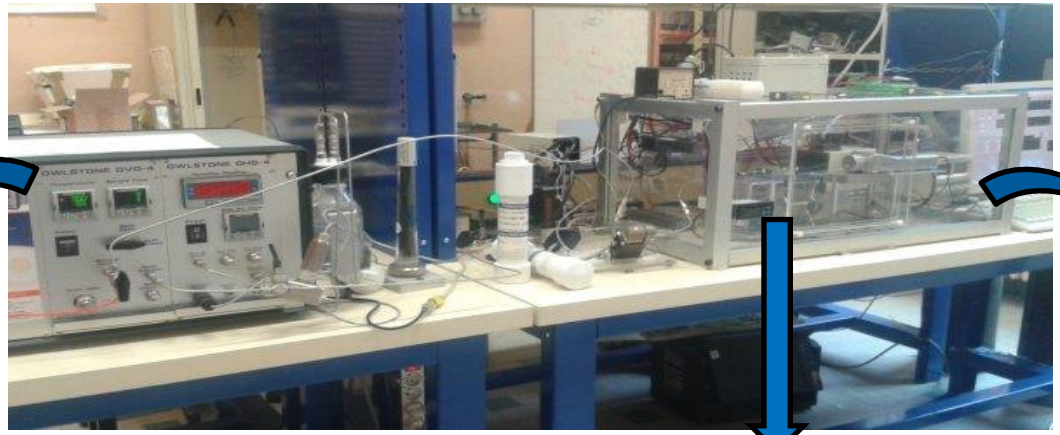


Thermal desorption:
Desorption
temperature at 250-
300°C

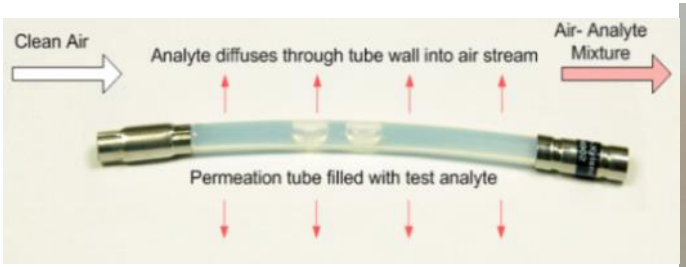
Preconcentration test. Laboratory test bench

Setup





Permeation tube



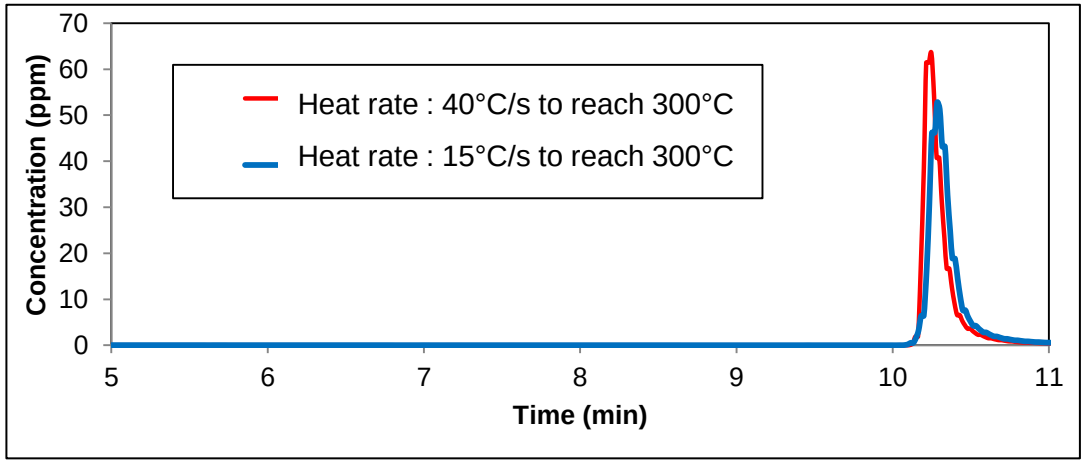
Carbon Preconcentrator

Photo Ionisation Detector (PID) for Vapour Organic Compounds (VOCs)

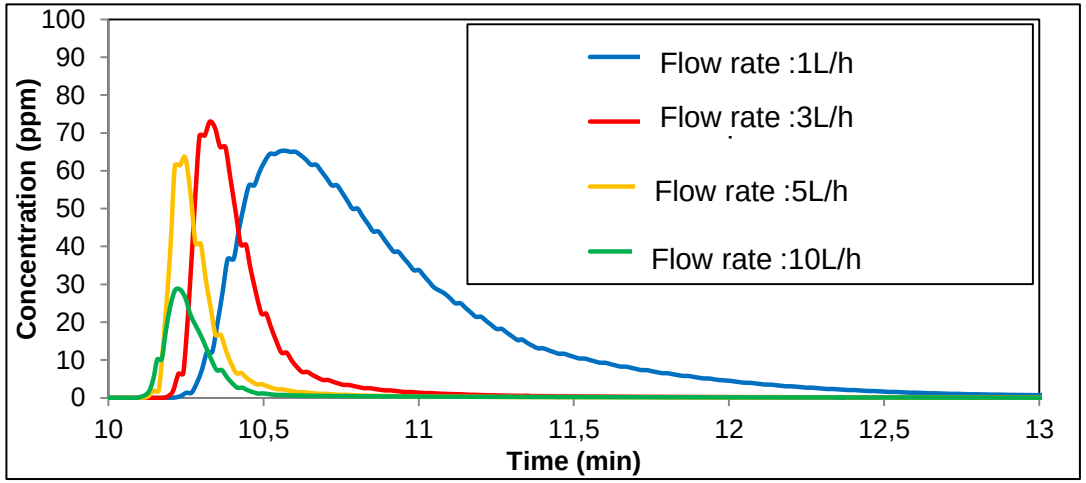
Preconcentration test. Parameter influence

Heating rate and flow rate influence

Vapor exposure : 1 ppm of Toluene during 5 min at 10L/h



High heating rate for high peak detection

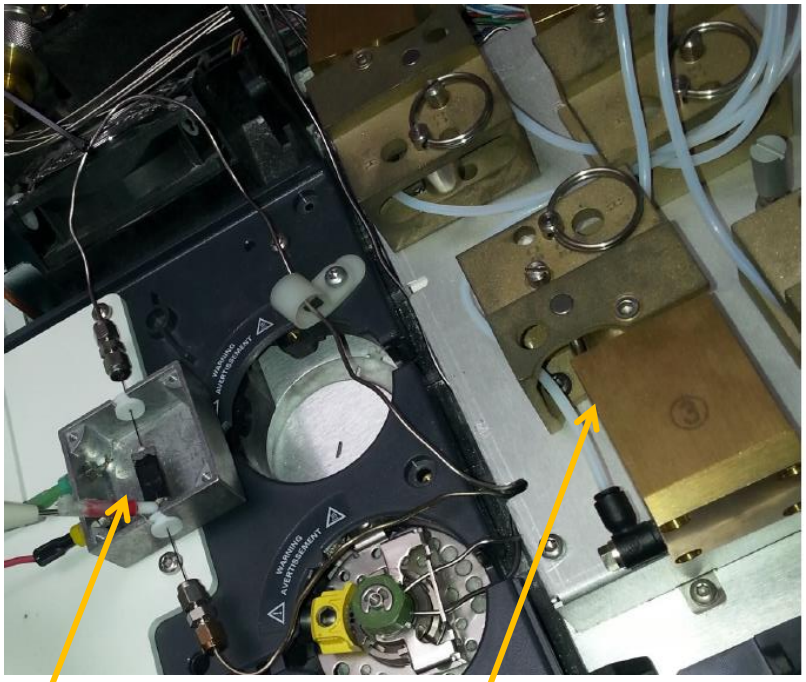


Low flow rate for high peak detection

$$C_{out} = \frac{Number\ \ of\ \ desorbed\ \ particles}{flow\ \ rate}$$

1rst application : GC Analysis. Industrial Setup

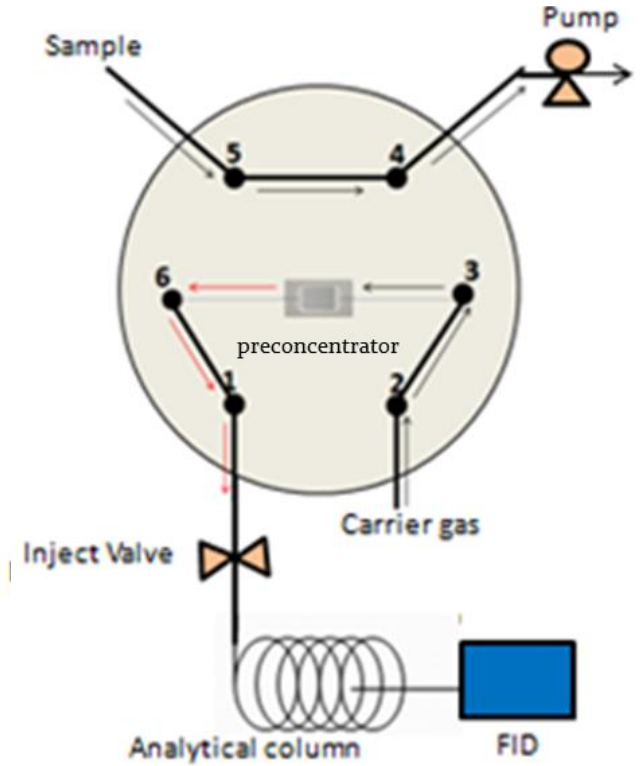
Setup and GC process



Preconcentrator

6 ways valve

Heating and injection



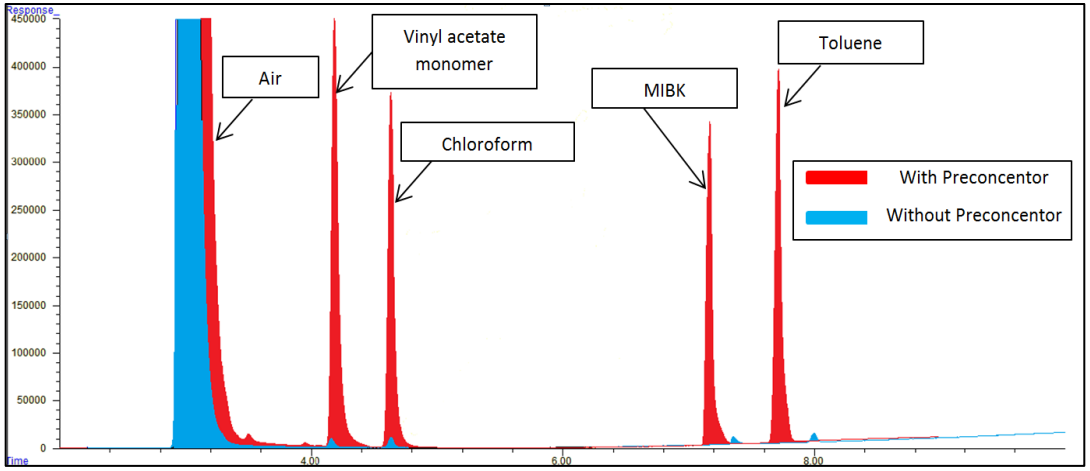
Preconcentrator as a sample loop

1rst application : GC Analysis

Chromatograms

Industrial applications:
Gas mixture analysis (Toluene,Methylisobutyketone, Chloroforme, Vinyl acetate)

ppm trace level analysis

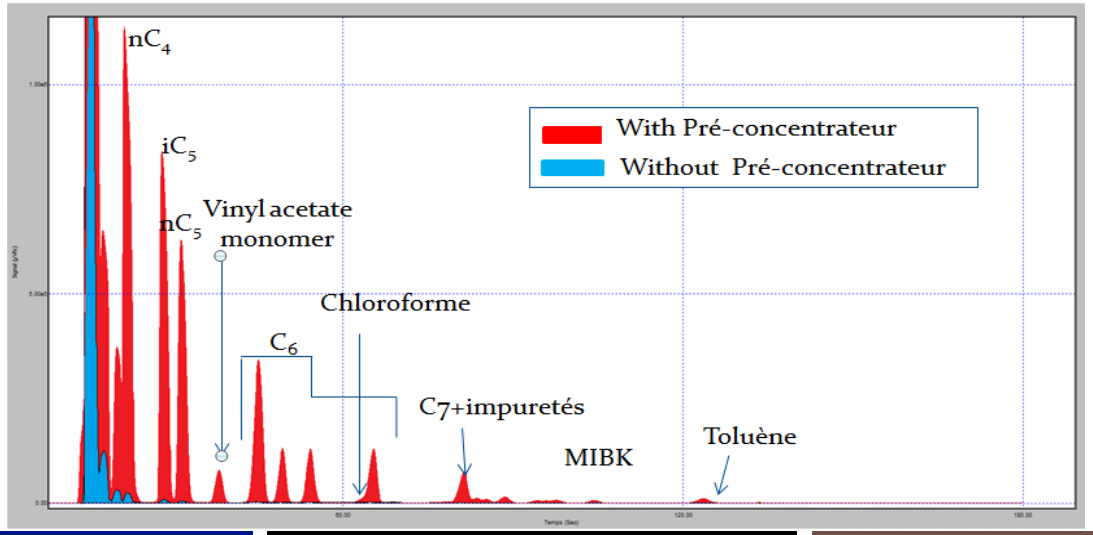


$$PF = \frac{A_2}{A_1}$$

A : peak area

Target Gas	Initial Concentrations	Preconcentration factors (PF)
Toluene	8 ppm	689
Vinyl acetate monomer	9 ppm	658
Chloroform	11 ppm	438
MIBK	7 ppm	576

ppb trace level analysis with interferences (natural gas)



Target Gas	Initial Concentrations	Preconcentration factors (PF)
Toluene	37 ppb	200
Vinyl acetate monomer	39 ppb	800
Chloroform	46 ppb	600
MIBK	35 ppb	200

2nd application : Explosive detection

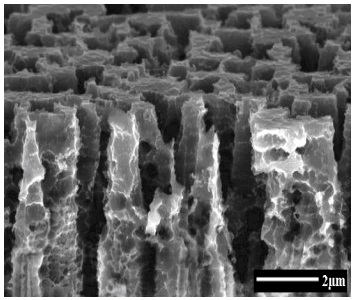
Explosive detection

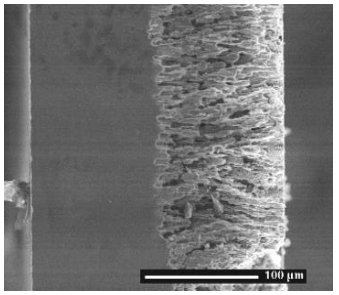
Micropreconcentration of DNT
(Dinitrotoluène)

ppb trace level analysis

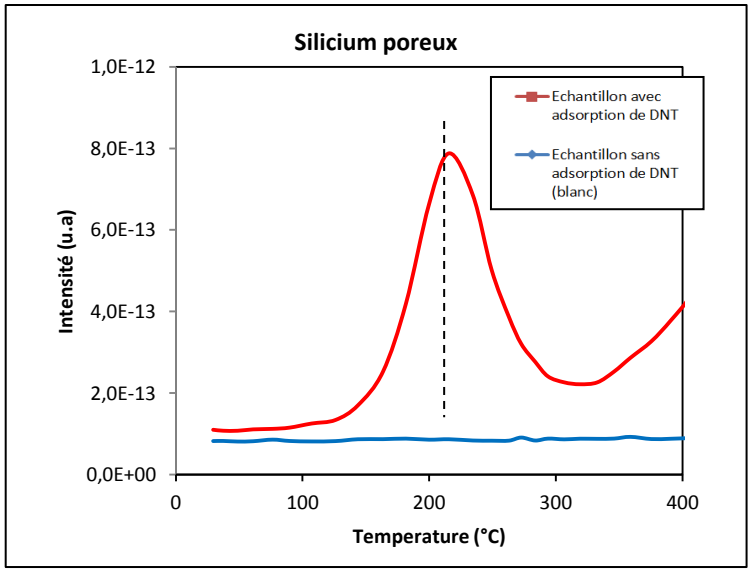
Use of porous silicon (composant wall) as adsorbant

pore size Ø : 1-2 µm and thickness: 90-110 µm

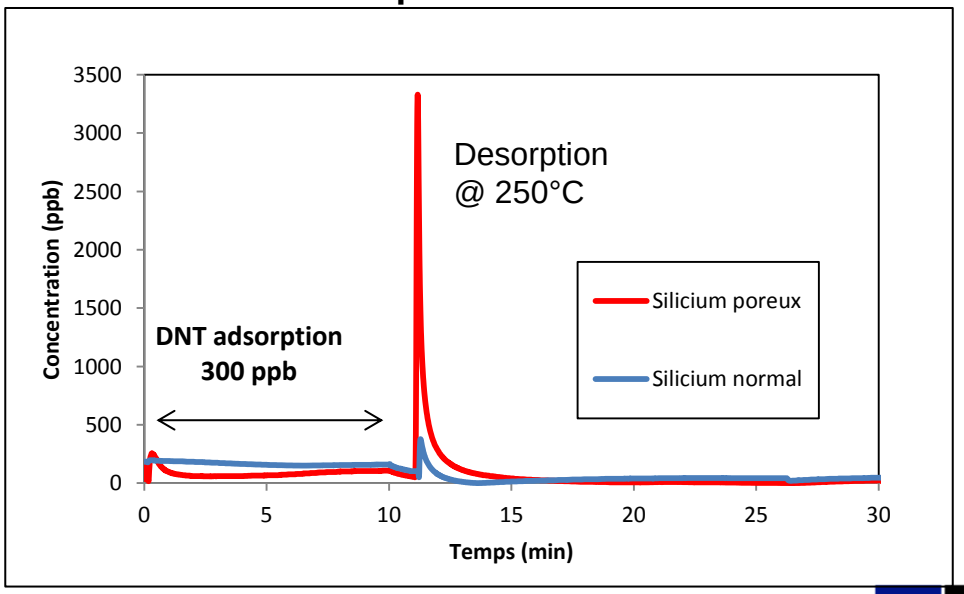




Thermal Programmed Desorption of DNT on porous silicon



Preconcentration test of DNT on porous silicon



Conclusions

- ✓ **Microfabricated MEMS preconcentrator**
  - Low volume :14 µL
  - Fast Heating rate :40°C/s
  - Flow rate : 1 to 20 L/h

- ✓ **Various adsorbent material : Carbon nanopowder or nanotubes, Tenax, porous silicon , …**
  - Good adsorption capacity for large range of VOCs, explosives, drugs…

- ✓ **Wide range of applications**
  - Coupling micropreconcentrator/µ-GC or with other analyzer
  - Air quality monitoring (VOCs)
  - Security applications (explosives, drugs)
  - Any need of traces detection…

Acknowledgments

MINES Saint-Étienne

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

IN nov AL

AXELERA catalyseur de croissance durable

L'analyse en ligne au coeur des procédés

SNIFFER

EC project on Explosive detection
FP7-SEC-2011-1 N° 285203

cea

MANCHESTER 1824 The University of Manchester

ARMINES

Università degli Studi di Padova

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EADS

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

CSSC Centre for Science, Society and Citizenship

TraceTech Security

GTP

משטרת ישראל

ST[SI]²

Thank you for attention