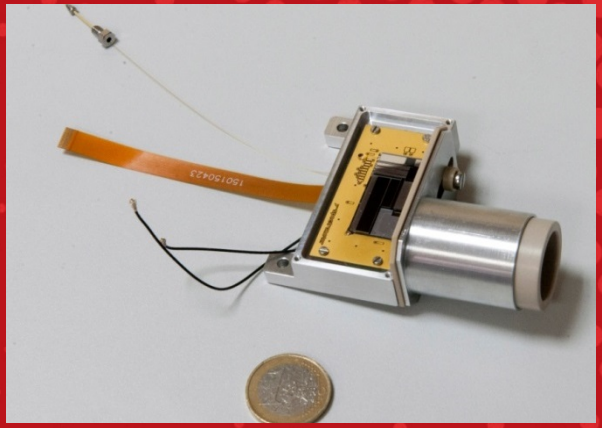


DE LA RECHERCHE À L'INDUSTRIE

Development of Micro-Time-Of-Flight mass spectrometer for *in situ* gas analysis

F. PROGENT, A. SONNETTE, J. TUPINIER, P.-E. BUTHIER, F. CHAUMONT, T. ALAVA, S. VIGNE

CEA, DAM, DIF, F-91297 Arpajon, France
Commissariat à l'énergie atomique et aux énergies alternatives - www.cea.fr

ON-SITE GAS ANALYSIS

Environment

(O_3, NOX, CH_4,…)

Large variety of chemicals to detect:
Need a fast, versatil, accurate, sensitive and portable analytical system

Chemical Industry

(pesticid

Mass Spectrometry

PRINCIPLE OF A TOF-MS WITH REFLECTRON

① Gas injection from µ-GC or GC

② Ionization and molecule fragmentation by Electron Impact

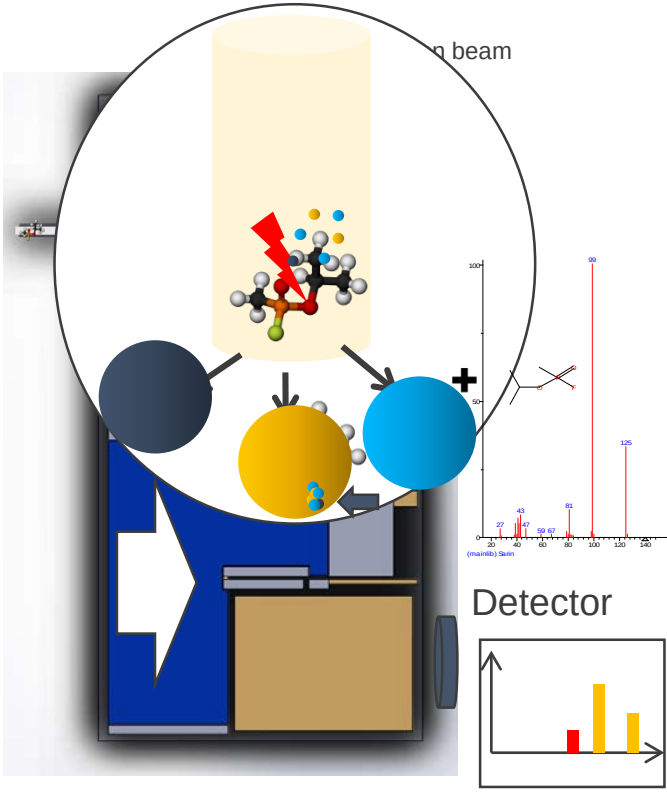
③ Ion extraction and focusing

④ Orthogonal injection

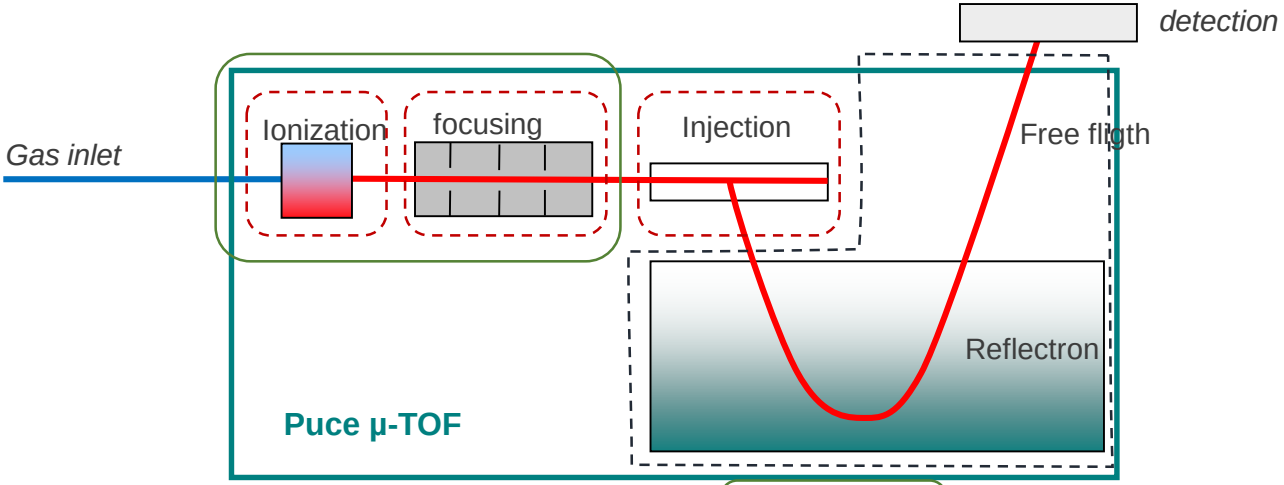
⑤ Energy focusing by reflectron

⑥ Ion free flight and detection

⑦ Comparison with spectra database and compound identification



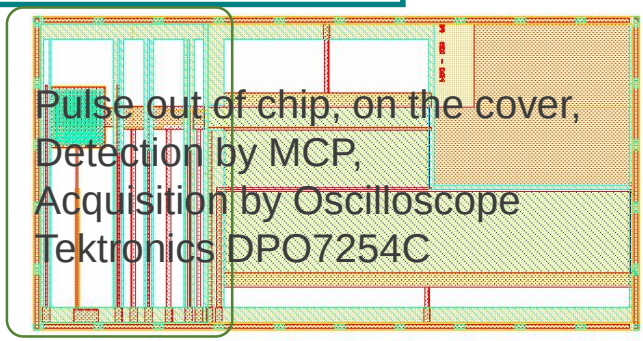
VALIDATION BY PART : LINEAR M-TOF



4 areas

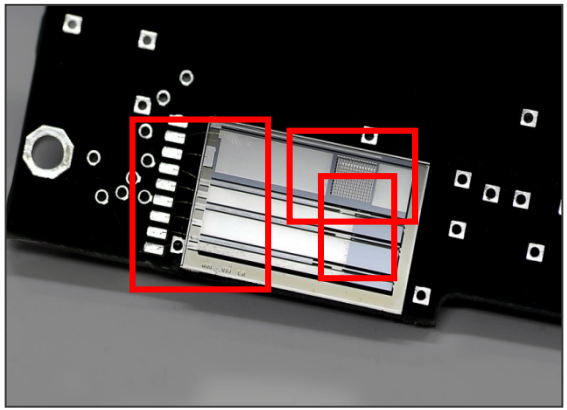
- **Ionization**
- **Ion extraction / focusing area**
- **Orthogonal Injection**
- **Free flight**

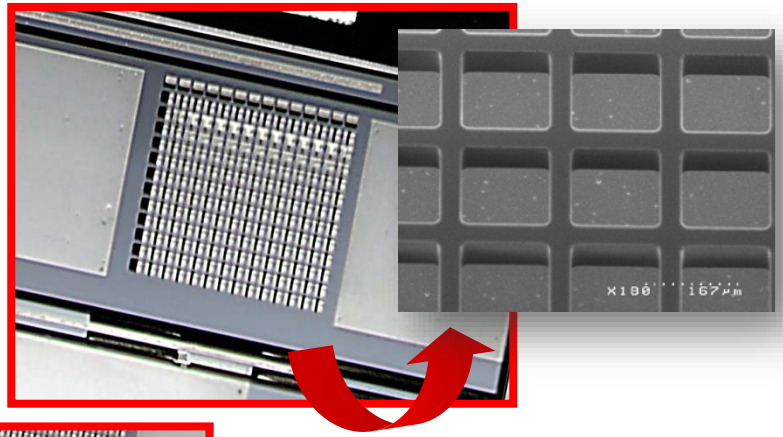
Linear micro-mass spectrometer

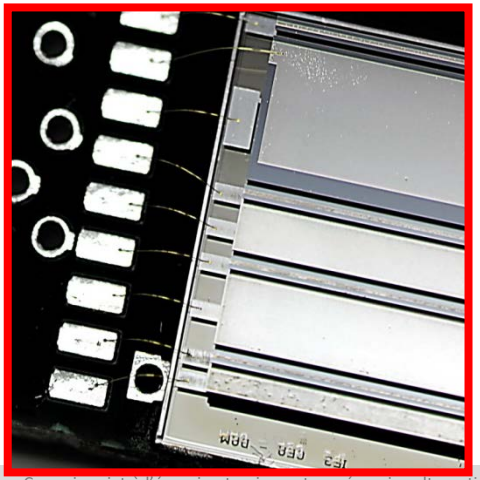


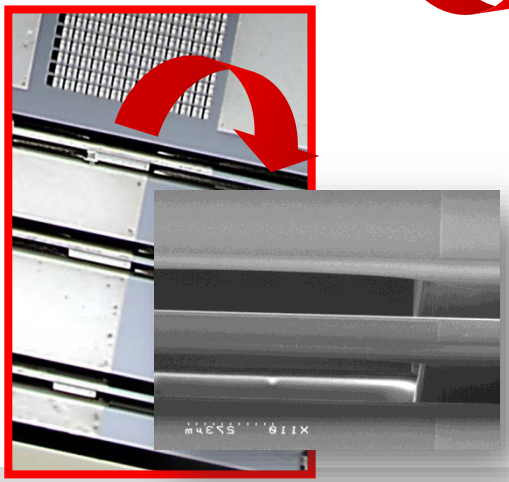
- Pulse out of chip, on the cover,
- Detection by MCP,
- Acquisition by Oscilloscope Tektronics DPO7254C

MICRO-SYSTEME TOF / SILICON CHIP





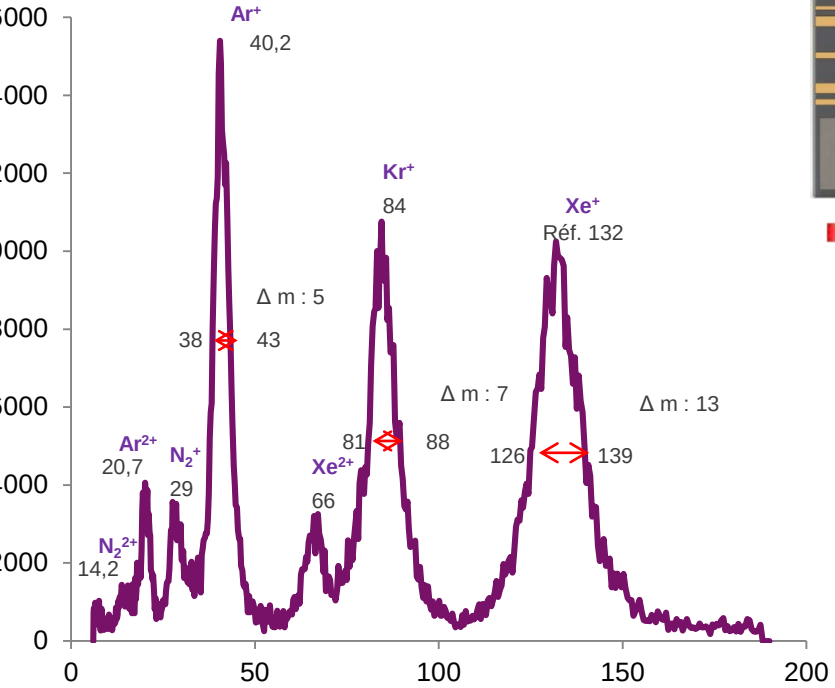




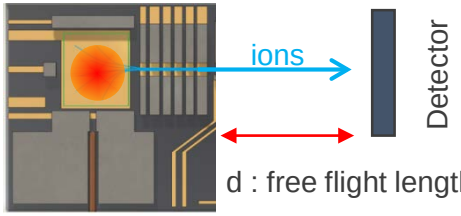
- Ionization Area
- Bonding
- Electrostatic lens

Chip check by SEM (Scanning Electron Microscopy)

MASS SPECTRUM WITH LINEAR M-TOF

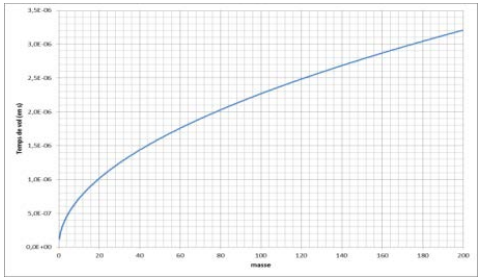


Ne, Ar, Kr, Xe 1% in helium, calibration on Xe^+ peak, 132 uma



- Pulse electron beam

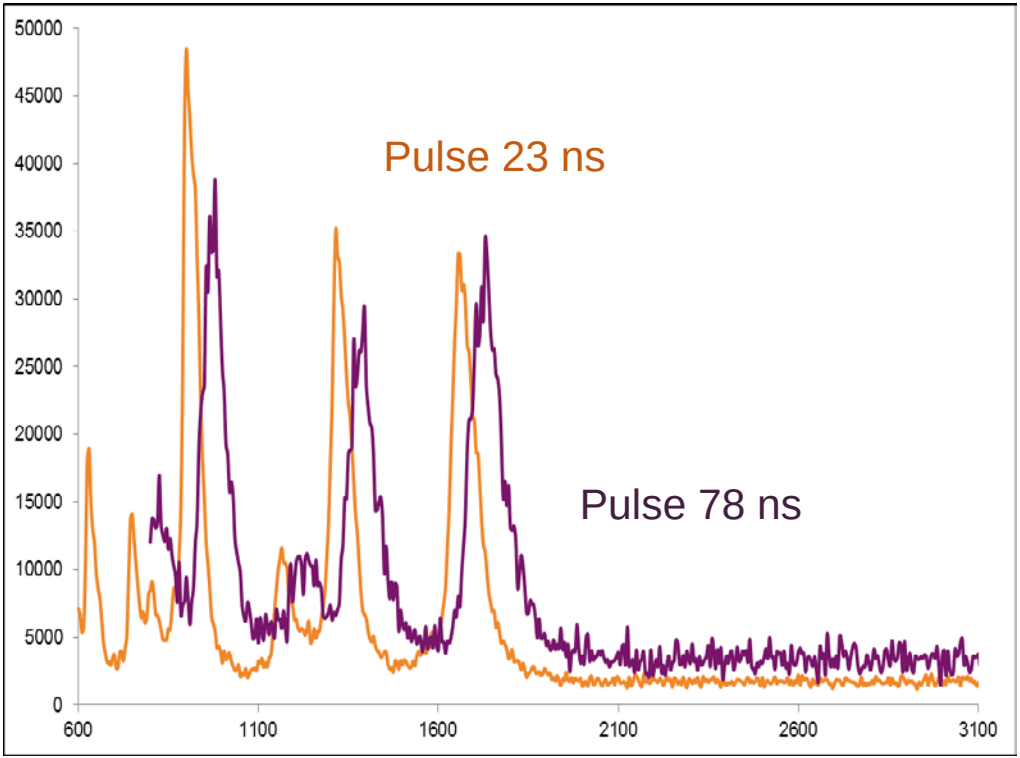
$$t_{flight} = \frac{d}{2\sqrt{U}}\sqrt{\frac{M_a M_p}{q}}$$



- Gas identification / few air gas
- Broad peaks

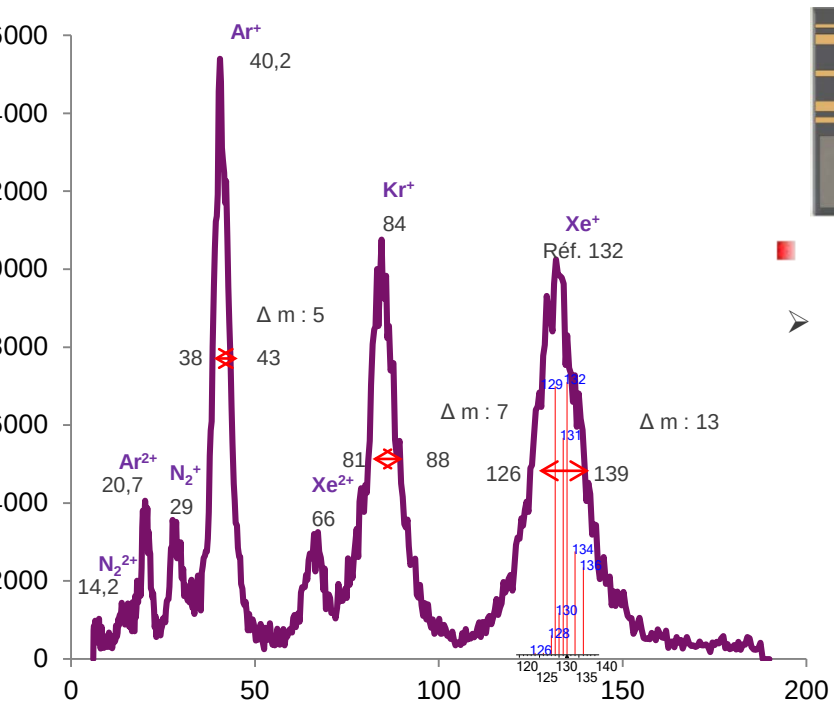
→ Pulse length

PULSE LENGTH & RESOLUTION



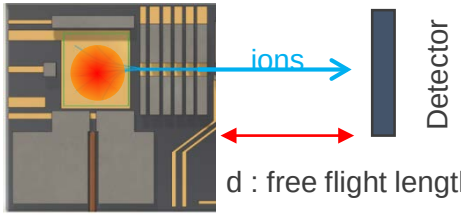
- Light improvement of resolution
- Lower background (change of pulse generator)

MASS SPECTRUM WITH LINEAR M-TOF

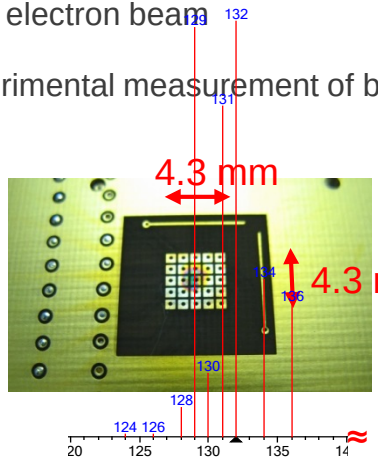


Ne, Ar, Kr, Xe 1% in helium, calibration on Xe^+ peak, 132 uma

- Broad peaks
  - Consistent with geometrical broadening / ionisation area
  - Underlying mass isotopic group



- Pulse electron beam
  - ➢ Experimental measurement of beam size

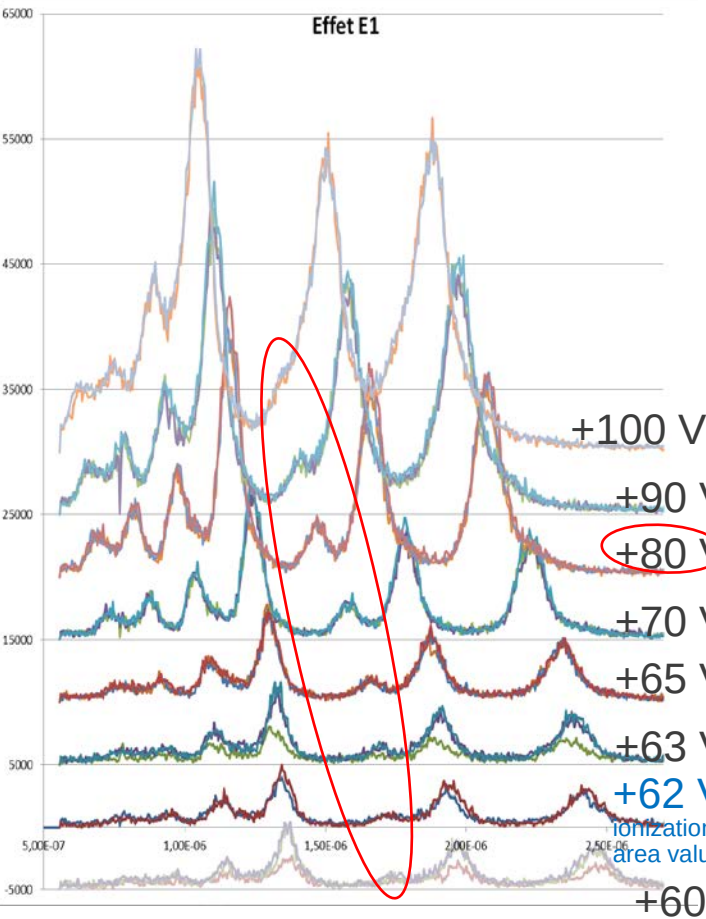


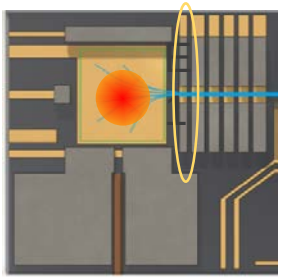
Theroetical mass spectrum of Xe, between 124 et 136 u, by E.I., NIST

➢ Geometrical dispersion obtained by peak enlargement

1,8 mm

EXAMPLE EXTRACTING & FOCUSING ELECTRODES



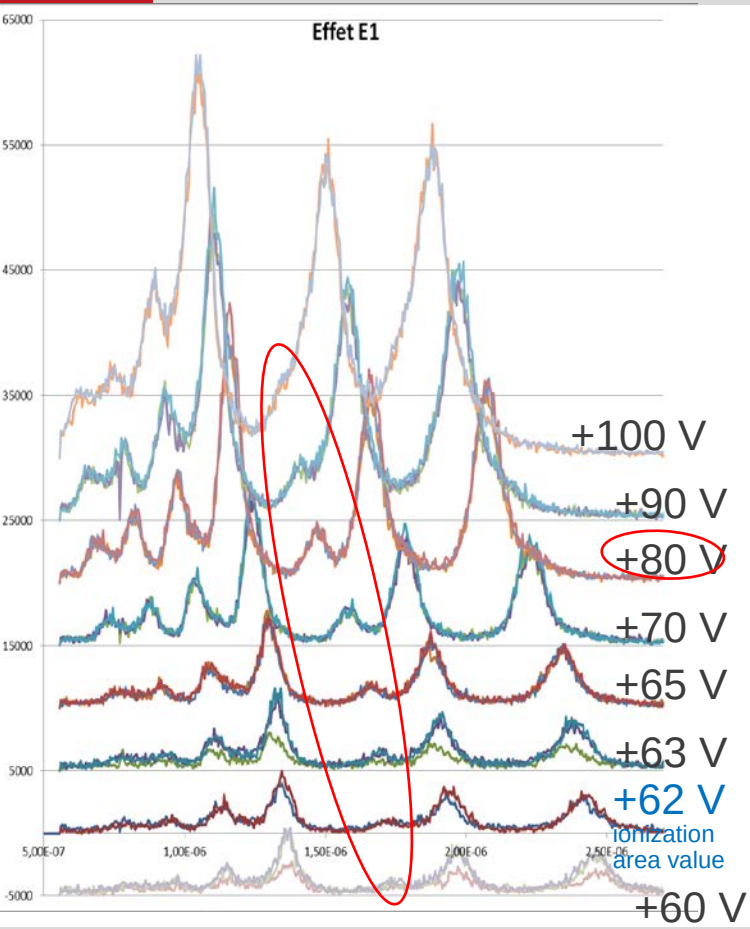


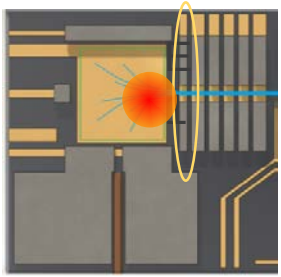
E1: extracting (& focusing)

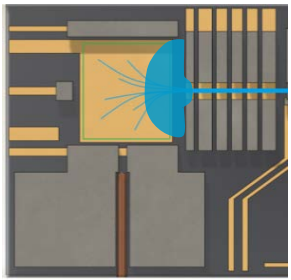
~~By simulation, a E1 voltage similar or slightly higher than ionization area is promising~~

- Ion acceleration
  - Higher acceleration gradient, dramatical modification of accelerate voltage
- Increasing intensities
  - Displacement of e beam and ionization area?
- Enhancement of peak separation
  - Better focusing, or…

EXAMPLE EXTRACTING & FOCUSING ELECTRODES





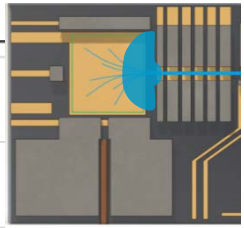


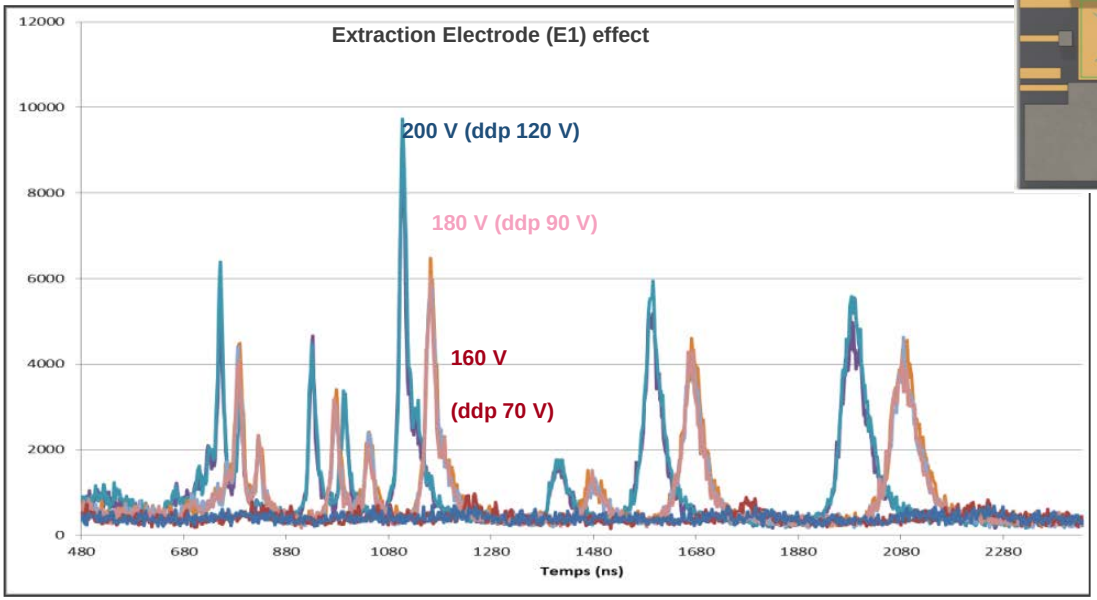
E1: extracting (& focusing)

- Reduced ionization area
  - Reduced geometric band broadening

EXAMPLE EXTRACTING & FOCUSING ELECTRODES

- Effect on intensity?



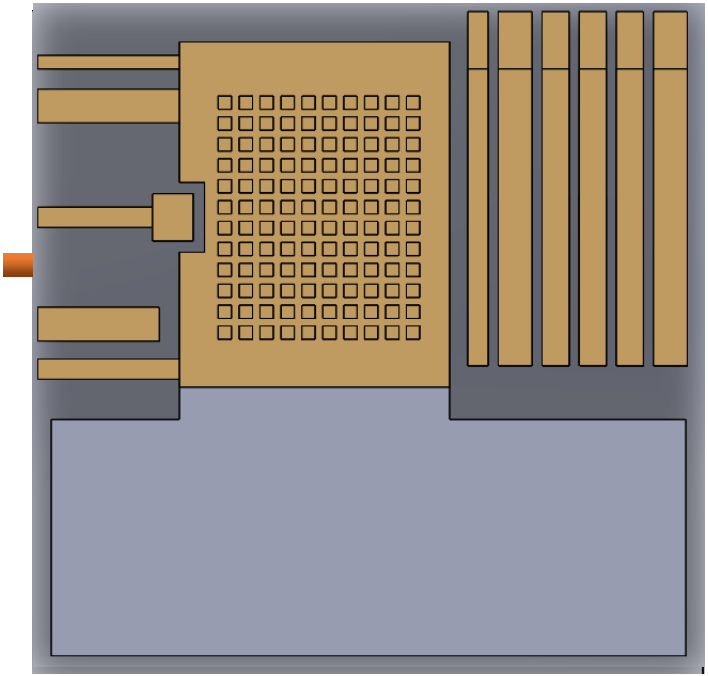


- Improvement of signal intensity

Gas inlet ?

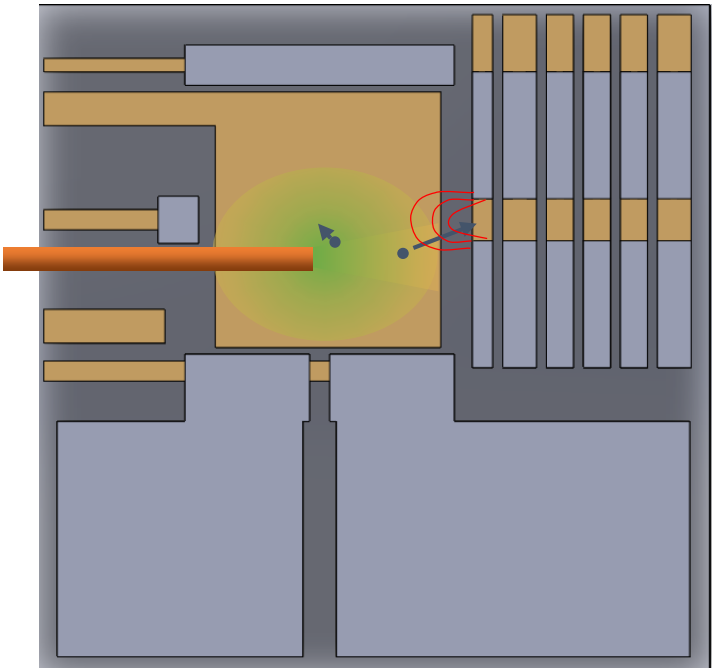
Flow Type?

- Micro-system
- 10^{-5} / 10^{-6} mbar (rarified gas)
  - Free molecular flow (impacts with walls / mean free path larger than the chamber size)
  - Supersonic flow (preferential direction)



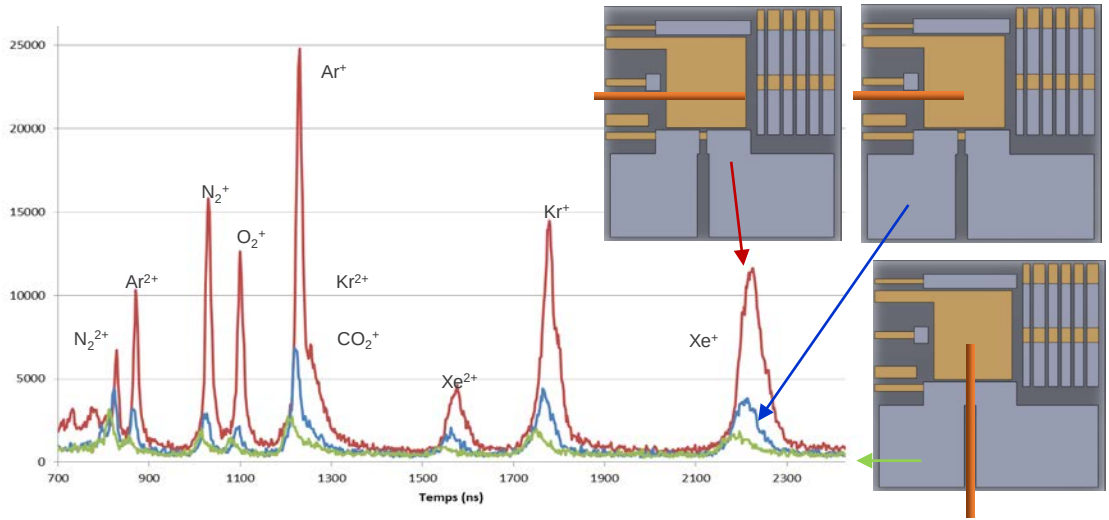
Flow Type?

- Micro-systeme
- 10^{-5} / 10^{-6} mbar (rarified gas)
  - Free molecular flow (impacts with walls / mean free path larger than the chamber size)
  - Supersonic flow (preferential direction)
- Issue involves:
- Concentration
- Geometrical and speed band broadening…



Flow Type?

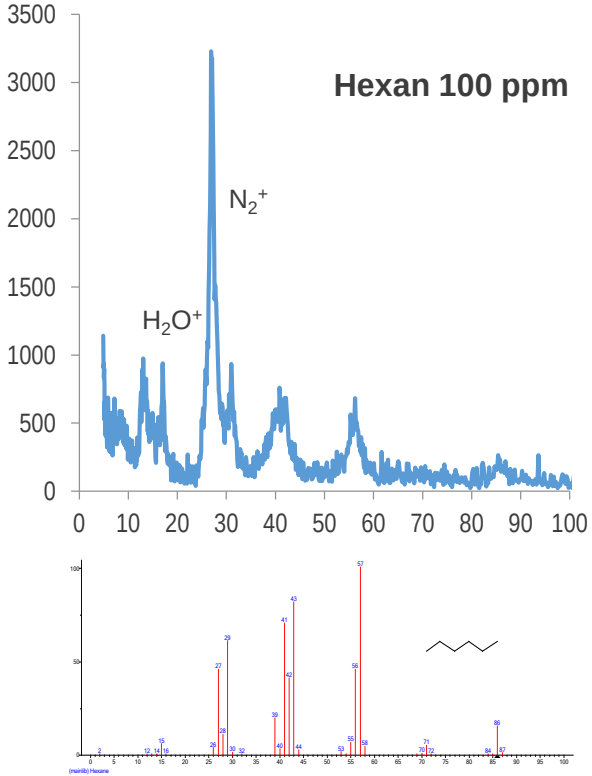
- Some test on capillary position

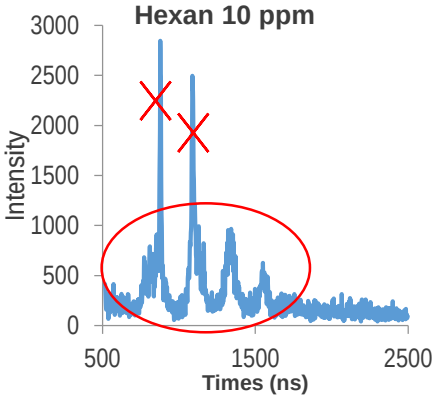


Ar, Kr, Xe 1000ppm (v/v) dans l'hélium, 5,96 10^{-6} mbar

- Support supersonic flow hypothesis

EXAMPLE OF ALKANE ANALYSIS



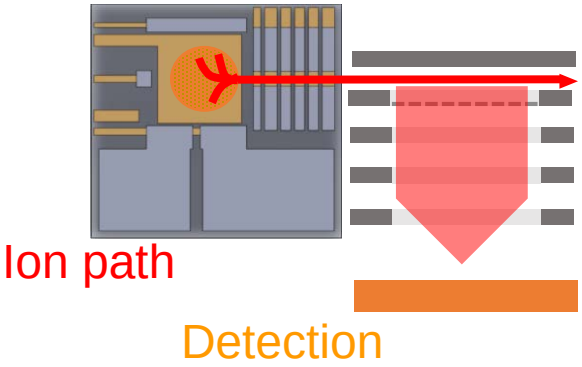


- Stabilisation of settings
- Resolution: est. 1-2 u. (masse 40)
- LD 100 ppm noble gas
- LD 10 ppm for hexane

Use as ion source

Linear µ-TOF used as ion source

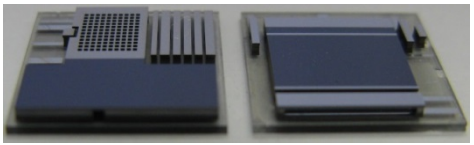
- Orthogonal injection on metallic electrode assembly



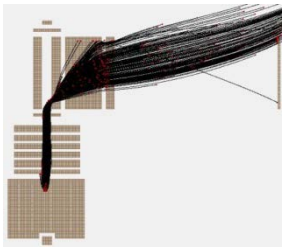
Metallic electrodes

- More versatil (design, voltage…)
- Protect orthogonal Injection chip

- Orthogonal Injection on chip



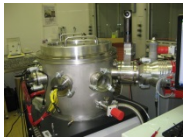
Chip assembly

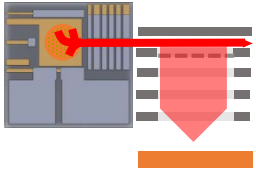


Simulations

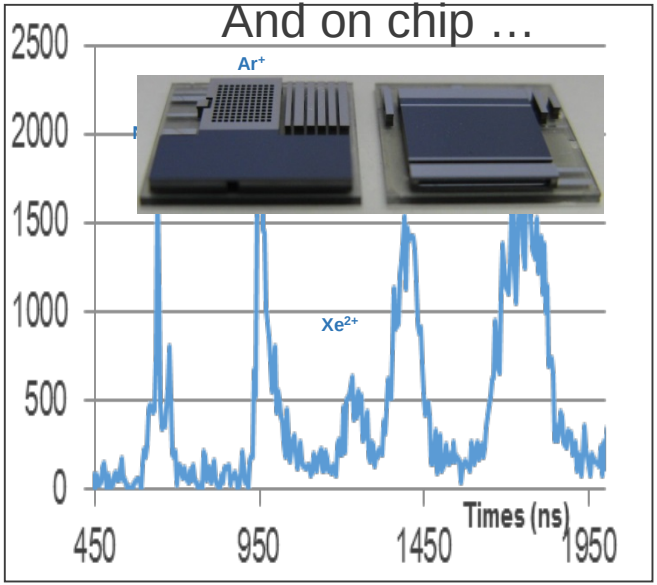
M-TOF WITH ORTHOGONAL INJECTION

Linear µ-TOF used as ion beam source

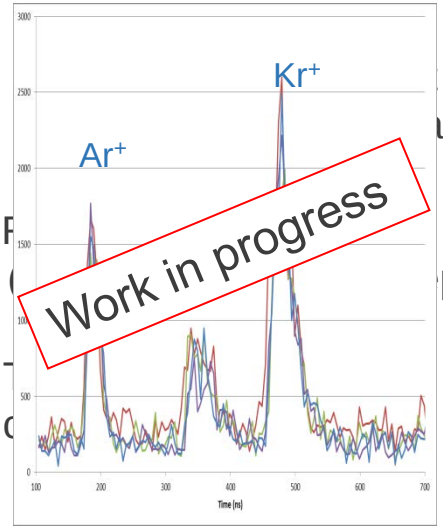




- Mass Spectra ⟺ Experimental informations on the different settings



- …
- …
- …



…s)

…paration?)

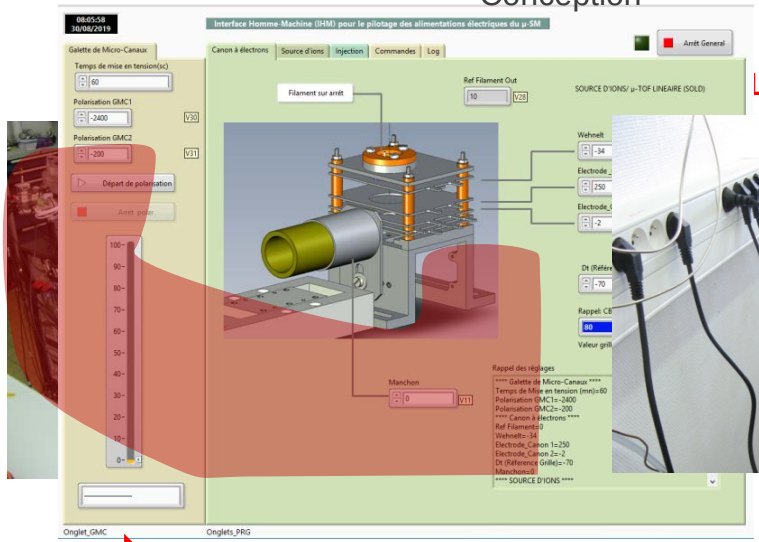
…similar to

FROM CHIP TO DEVICE: GLOBAL MINIATURIZATION

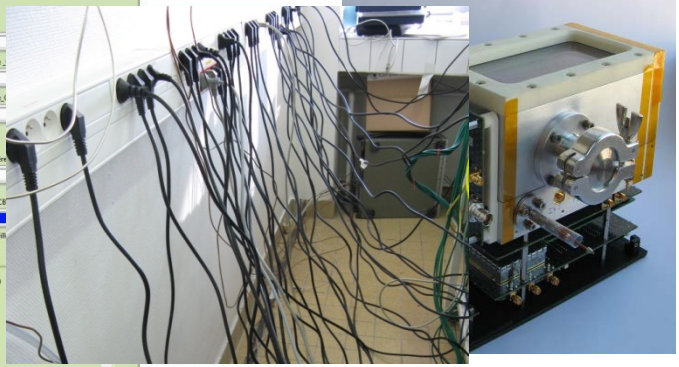
Chips Environment differentiates a source of academic curiosity and a efficient on-site device…

- Power supplies / Electrostatic & Pulse control

Conception



Manufacturing & Test & burn in



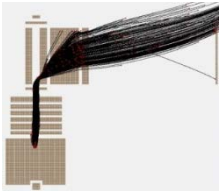
L8 cm

Test on linear µ-TOF

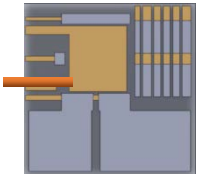
System is operational

Electrostatic & fluid Mechanics studies

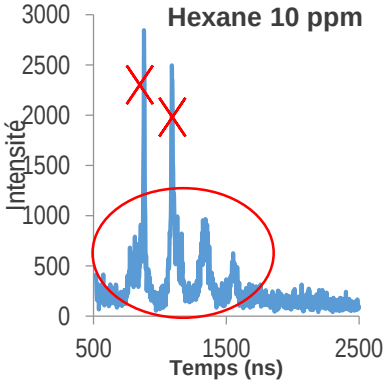
Simulations



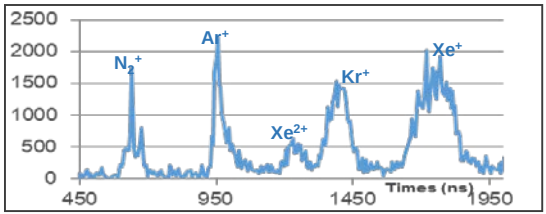
Experimental evaluation



Spectra acquisition



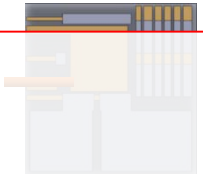
- More accurate set-up and adjustment, improvement of extraction and focusing performances
- Study of band broadening….



- Electrostatic & fluid Mechanics studies

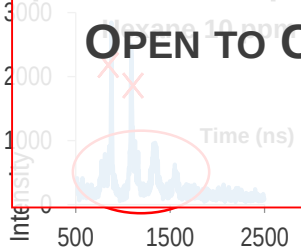
Simulations

Experimental evaluation



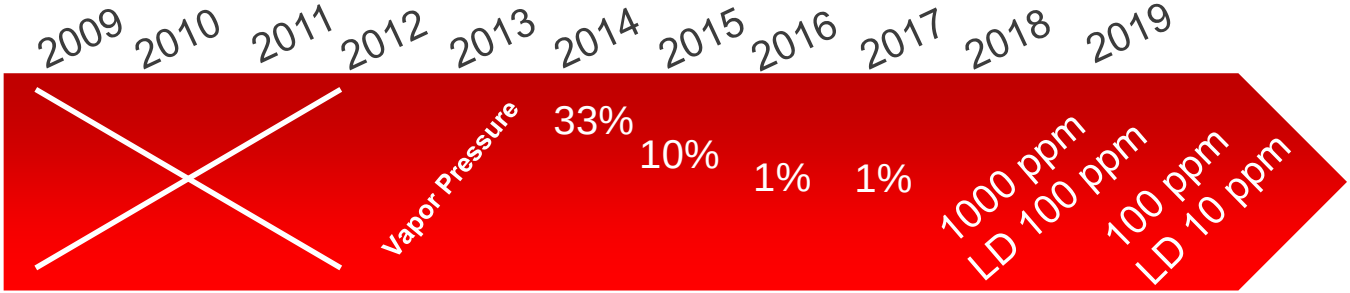
WORK IN PROGRESS…

- Spectra acquisition

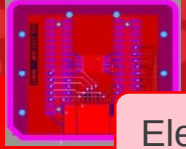


- More accurate set-up and adjustment, improvement of the ejection and focusing performances
- Study of band broadening….

OPEN TO COLLABORATION, EXTERNAL PARTNERSHIP…

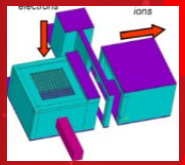


MEMS



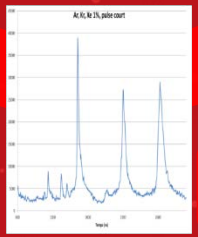
Electronic

Design & Simulations



***M-TOF IN SITU* DETECTION**

Vacuum & fluid's mechanical



Signal acquisition & processing

Electrostatic

DE LA RECHERCHE À L'INDUSTRIE

Thank you for your attention

frederic.progent@cea.fr