

Atmosf'air 2015

MPS

Mini Particle Sampler

Stéphane PERCOT, ECOMESURE sales engineer



maîtriser le risque |
pour un développement durable



ECOMESURE

COMPANY PROFILE

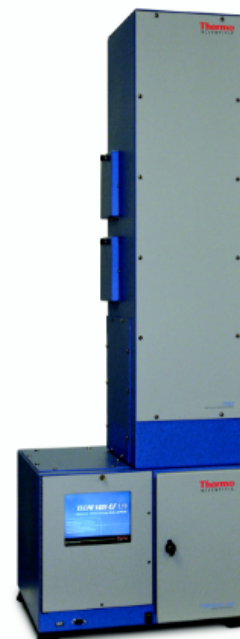
- ECOMESURE is specialized in metrology and instrumentation of air quality and aerosols
- 15 people – 4.2 M€ sales – 10% EBITDA
- Rated 3 at Banque de France
- Certified ISO 9001
- Present in the market for more than 20 years
- Taken over by Damien PELLETIER in October 2013 under LBO



OUR PRODUCTS

We provide our customers with complete and innovative solutions:

- Products for sampling, measuring and analysing air quality
- Integrated systems
- Added-value services





State-of-the-art
technologies from:

- USA
- Japan
- Europe
- Australia

ThermoFisher
SCIENTIFIC

particle technology
PALAS®

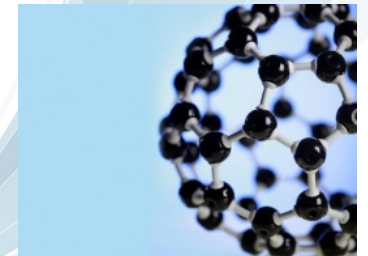
 **EIGENBRODT**®


GRIMM

APPLICATIONS



- Environment (ambient air)
- Emission
- In-door air
- Industrial hygiene
- Clean room
- Physics of aerosols



OUR CLIENTS



- **Air Quality Monitoring Networks**
- **Industry** : chemical, pharmaceutical, cosmetics, electronics, aerospace, food, environment, construction materials, etc.
- **Research laboratories** (climat, pollution, ...) and **test laboratories** (automobile, aerospace, ...)
- **Public Services** : Transportation, Energy, Hospitals, Police, Defense, etc.
- **Local collectivities**
- **Quality and Regulation Control Service Providers**

OUR TECHNOLOGY PARTNERS



MEMBERSHIPS



- A sampler developed by INERIS



- Manufactured and sold by ECOMESURE

EXISTINGS METHODS :

Real time analysis

- Mass concentration: TEOM
- Number concentration: CNC
- Size distribution: SMPS, ELPI, diffusion battery

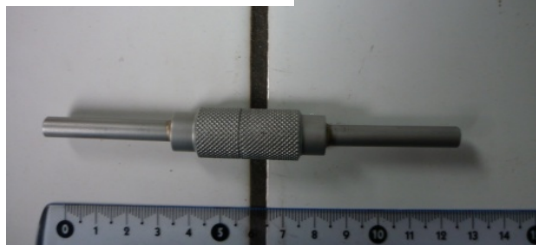
Analysis

- Quantification
- Morphology, size, elementary composition(TEM, SEM and EDX)

NEED :

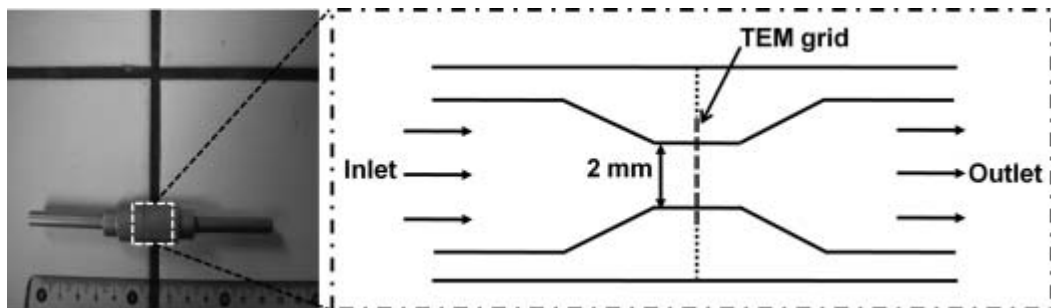
- Multipurpose tool, « low cost, portable, easy to use »

PRINCIPLE



(INERIS, 2011)

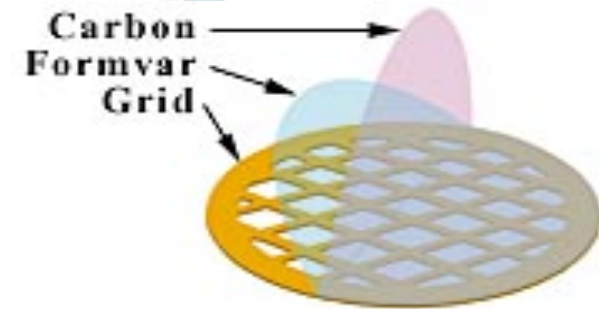
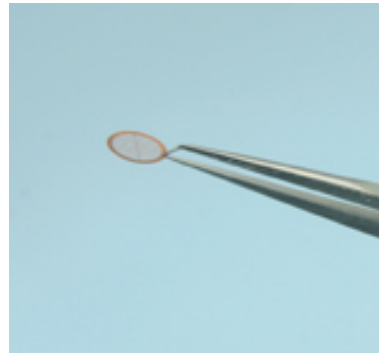
→ Ease of use, low cost, small and lightweight



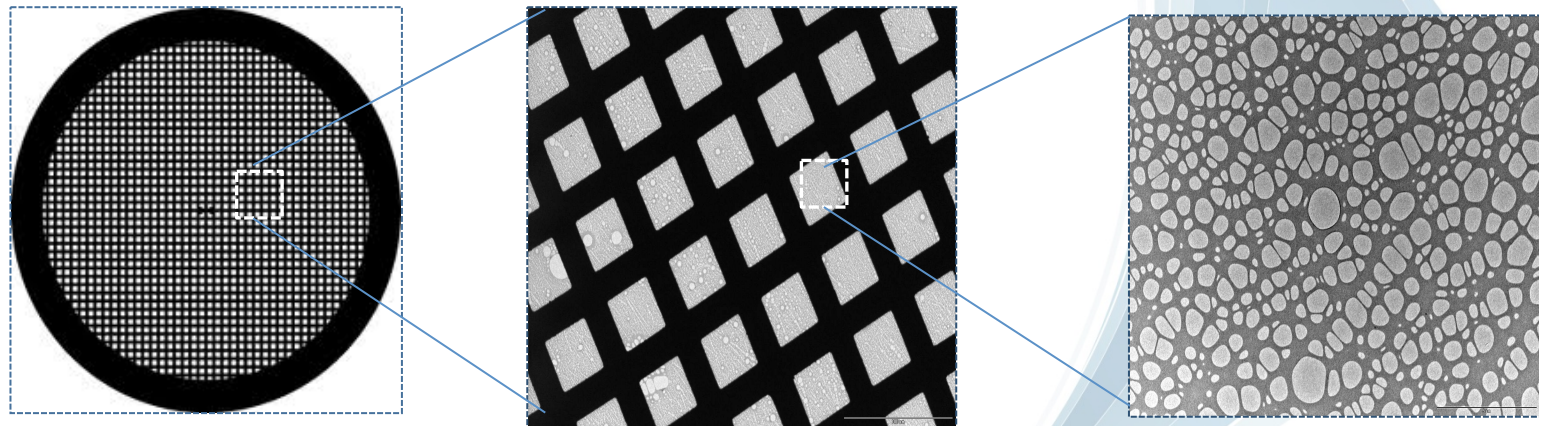
TEM GRID

(Lyyräinen et al. 2009)

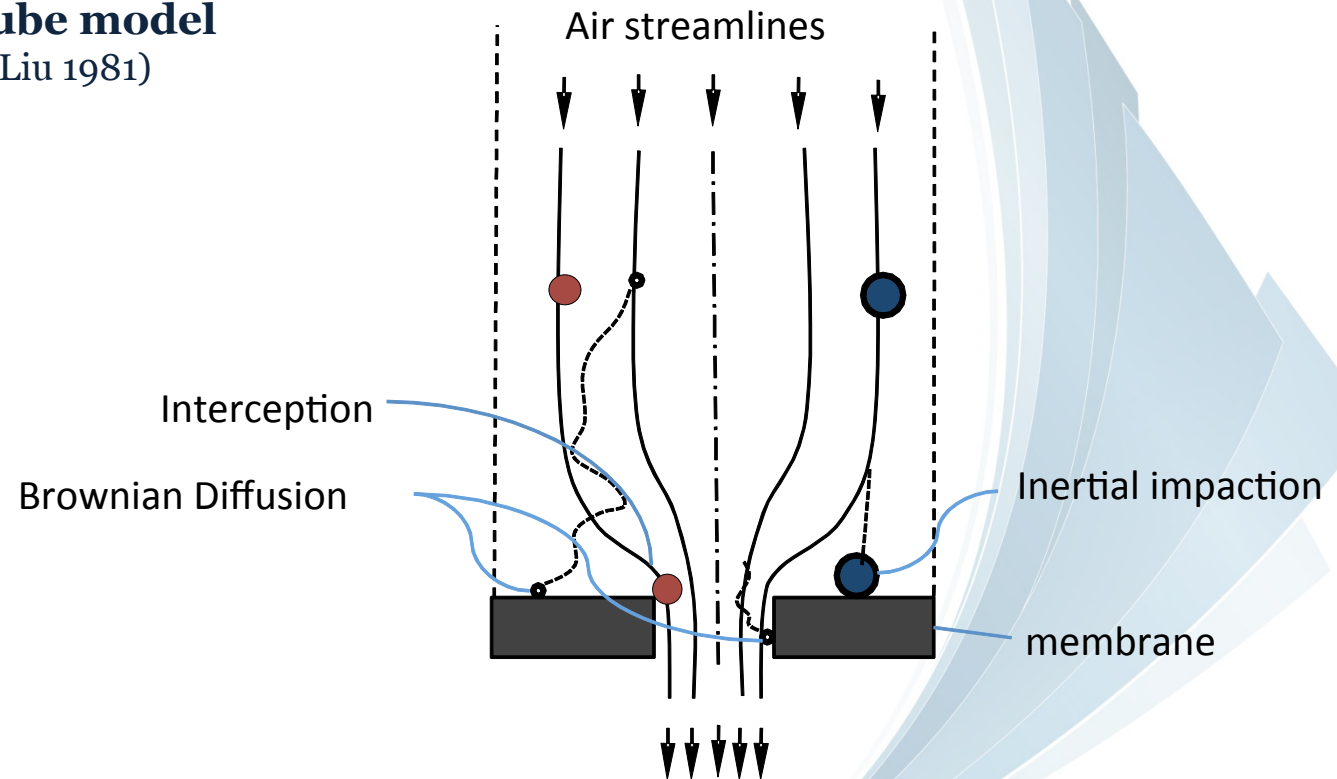
Potential / other technics:
simplicity



Example of a porous membrane TEM grid



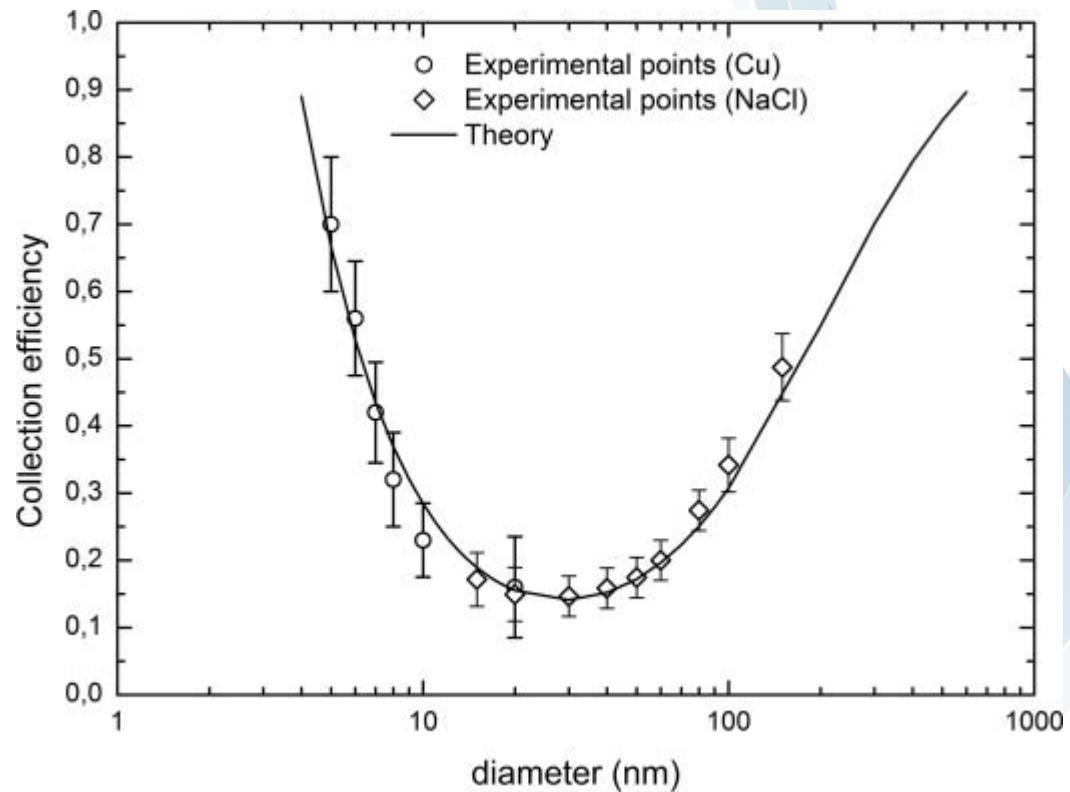
Capillary tube model (Rubow & Liu 1981)



MPS – Collection efficiency

- Sampling:

Collection efficiency
> 15%



B. R'mili , Olivier L. C. Le Bihan , C. Dutouquet , O. Aguerre-Charriol & E. Frejafon (2013):

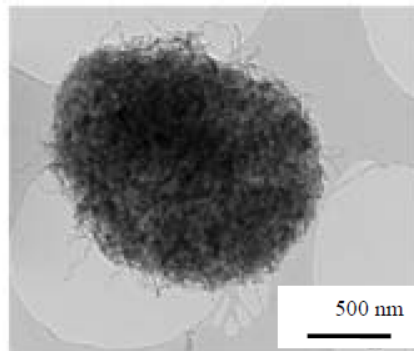
Particle Sampling by TEM Grid Filtration, Aerosol Science and Technology, 47:7, 767-775

MPS – Analysis

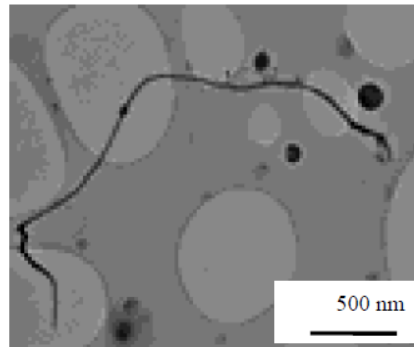
- Analysis with a TEM

Example :

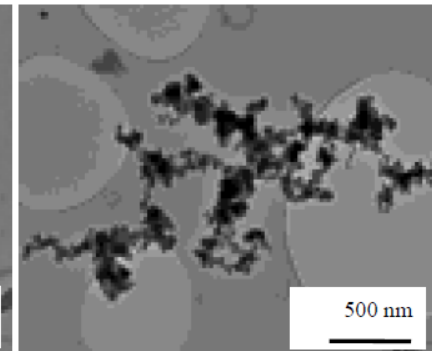
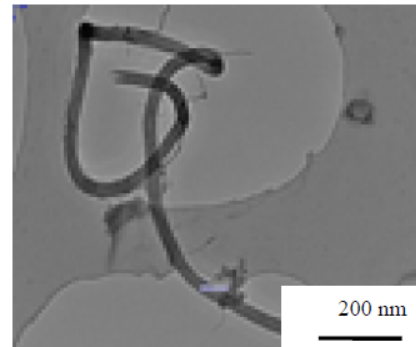
Analysis of particle release when handling CNT (Carbon NanoTube) powders (R'mili et al. 2011)



CNT bundle



Isolated fiber of CNT



Carbon agglomerates

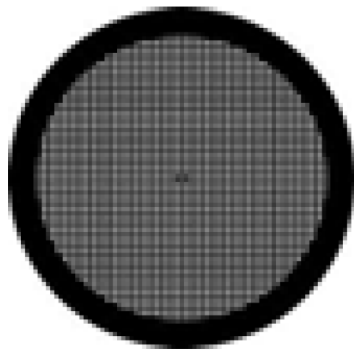
MPS – Demonstration

- MPS



MPS – Demonstration

- Support of sampling: standard TEM grid

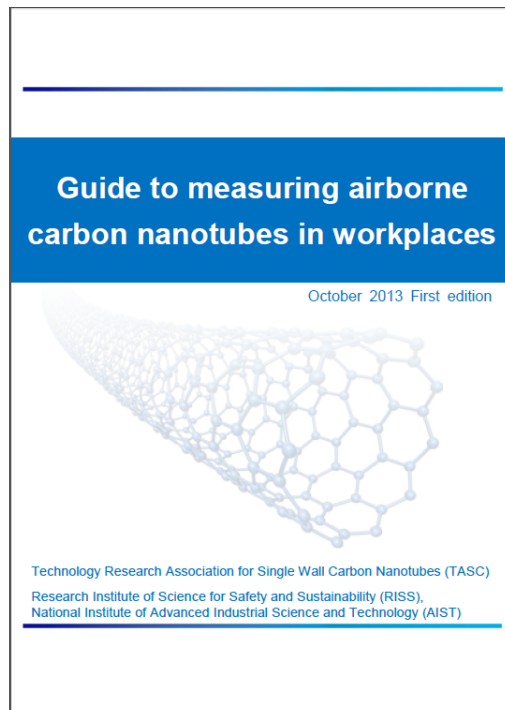


TEM grid 400 mesh



MPS – Recommendation

- Recommendation by the AIST (in Japan) for the observation of airborne carbon nanotubes in workplaces



3.2 模擬排出試験による粒径分布や形態の確認

CNT の飛散時の粒径分布を確認するために、簡易な試験管かくはん法(Maynard *et al.* 2004; Ogura *et al.* 2009)によって CNT を飛散させて(図 3.2)、走査型移動度粒径測定器(SMPS; model 3936L72, TSI Inc., USA)、エアロダイナミックパーティクルサイザ(APS; model 3321, TSI Inc., USA)、光散乱式粒子計数器(OPC; model 3330, TSI Inc., USA)によって、飛散粒子の個数濃度粒径分布を計測した(Hashimoto *et al.* 2013))。計測結果の例を図 3.3 に示す。粒径分布は、ナノサイズからミクロンサイズまでの広い範囲にわたる分布であった。

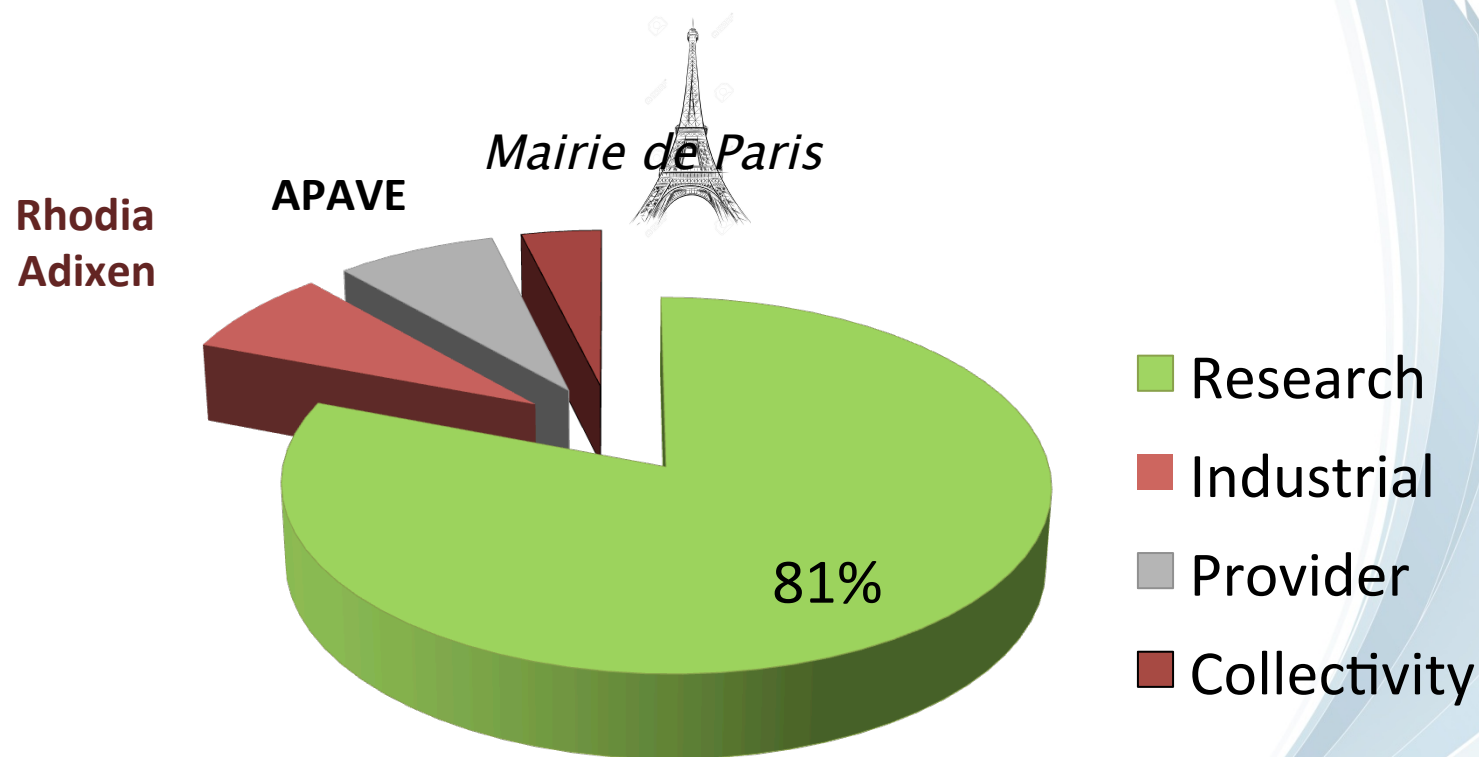
さらに、CNT の飛散時の形態を確認するために、あらかじめ白金パラジウムを約 2 nm の厚さで蒸着したポリカーボネートフィルタ(ニュークリポアメンブレン、孔径 80 nm、孔密度 6×10^8 個/cm²、φ 25 mm)をステンレス製フィルタホルダ(有効ろ過面積 3.7 cm²)に入れて、流量 0.5 L/min で飛散 CNT を捕集した。SEM による観察写真の例を図 3.4 に示す。また、多孔 TEM グリッド(Quantifoil R0.6/1, 孔径 0.6 μm (実際は若干大きい)、孔密度 3.9×10^7 個/cm²、φ 3.05 mm)を銅リング(内径 2 mm、φ 3.05 mm)とともにステンレス製の専用ホルダ(Mini-Particle Sampler: MPS®, Ecomesure, Janvry, France)に入れて、流量 0.3 L/min で飛散 CNT を捕集した。TEM による観察写真の例を図 3.5 に示す。多くは、サブミクロンからミクロンサイズに凝集した粒子であった。CNT はその種類やチューブ径によって見え方が異なり、チューブ径の細い単層 CNT はネット状や綿状、チューブ径の細い多層 CNT は羊毛状、チューブ径の太い多層 CNT は棒状の形態であった。



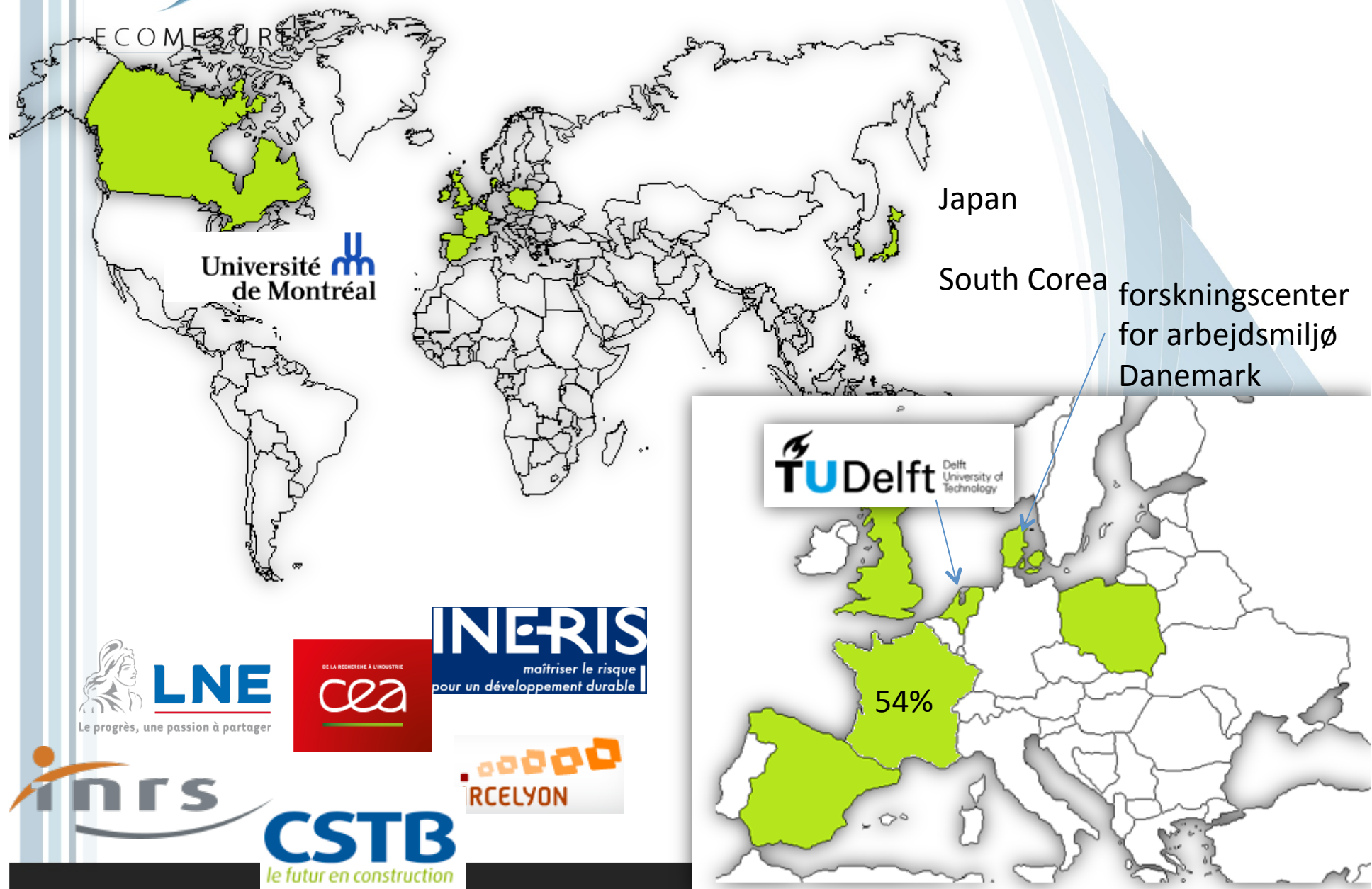
Application examples

MPS Ecomesure

Repartition by sector of activity



Research laboratories



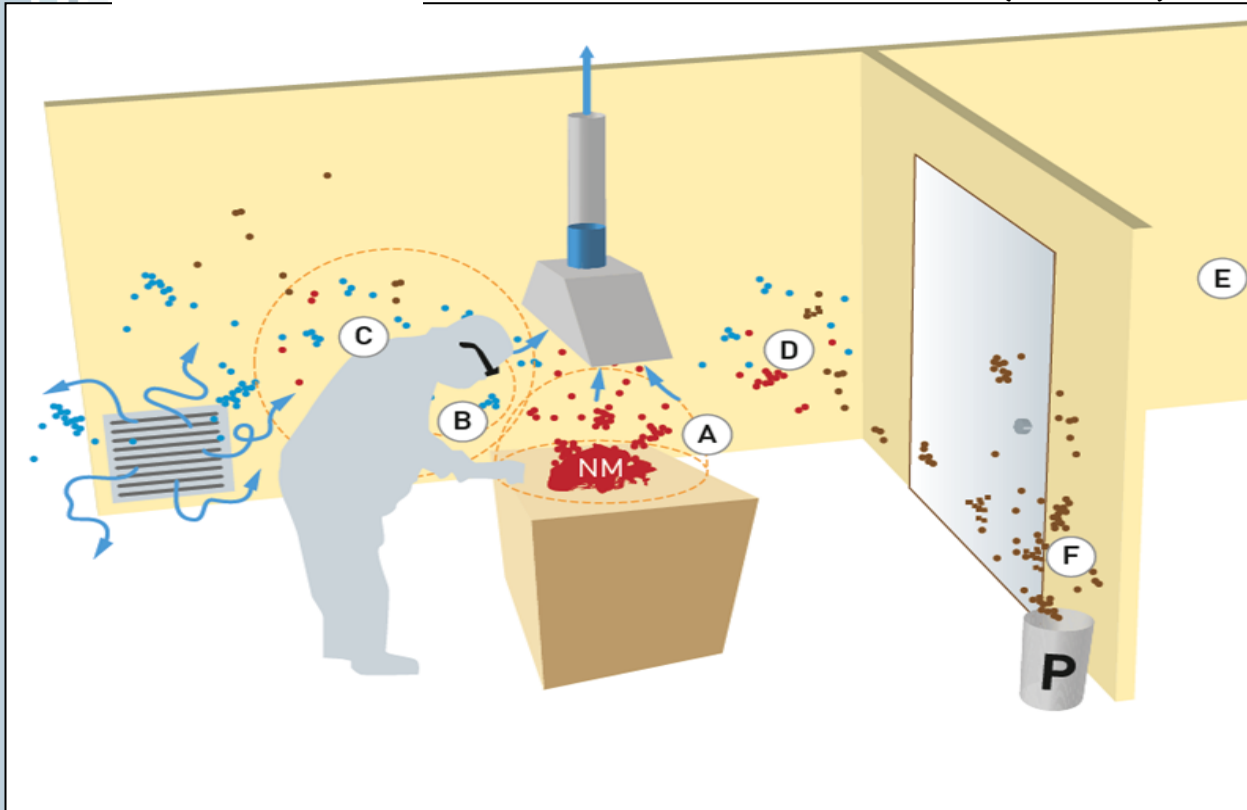
Customers applications

- In line use: Research laboratories and industries.
 - Emission: soot particles.
 - Industry: Microelectronic component. Sources characterisations.
 - Clean room: Nanomaterials manipulation.
- Indoor air quality: personnal exposition.
 - Asbestos fiber.
 - Mural degradation.
 - Grid analysis by INERIS.



Workstation measurement

STAKE: morphology, elementary composition, different contribution (ex. NMs)



Witschger, Le Bihan et al.,
Hygiène Santé au travail, avril
2012.

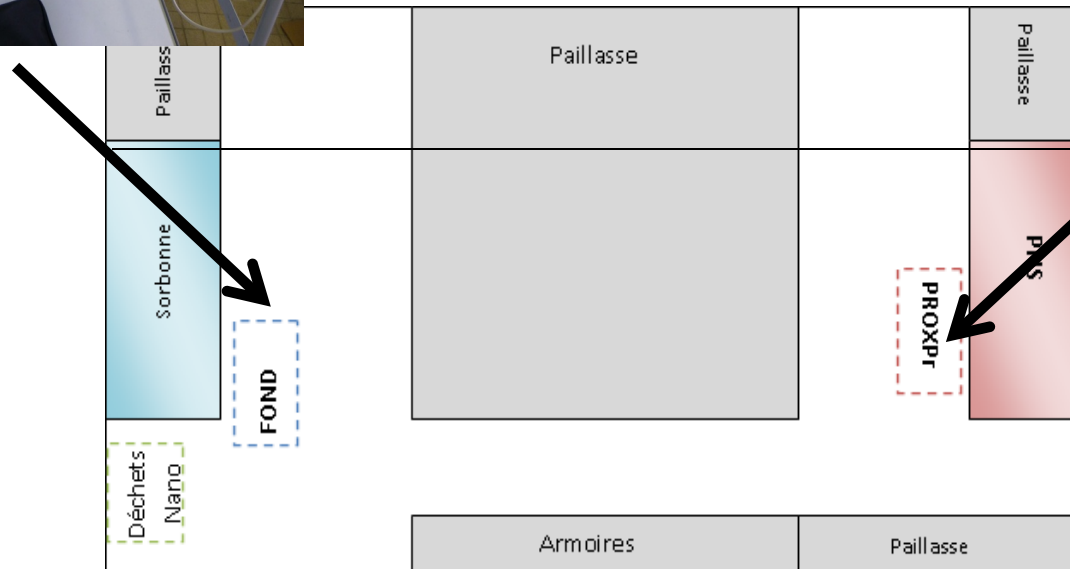
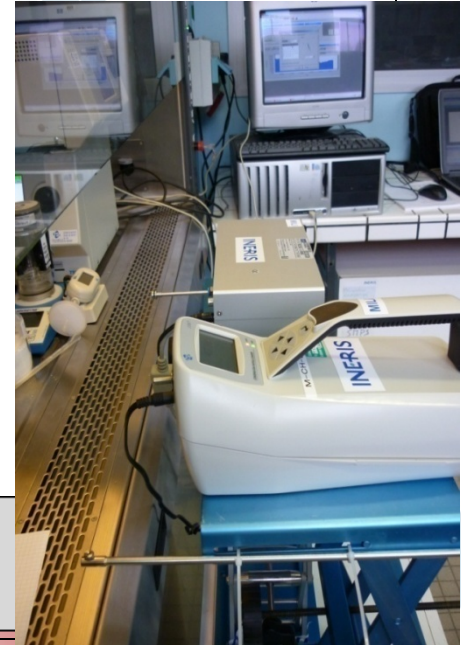
Illustration of different sampling positions in a place where an operator is working with nanomaterial (NM).

In blue : background aerosol from the outside,
in maroon : Background aerosol from a punctual source P of particles,
In red the target aerosol from the nanomaterial.

Measurement point at the source (A), in the breathing zone (B), near field of the operator (C), far field of the operator (D) outside the local zone (E), close to the punctual source P (F).

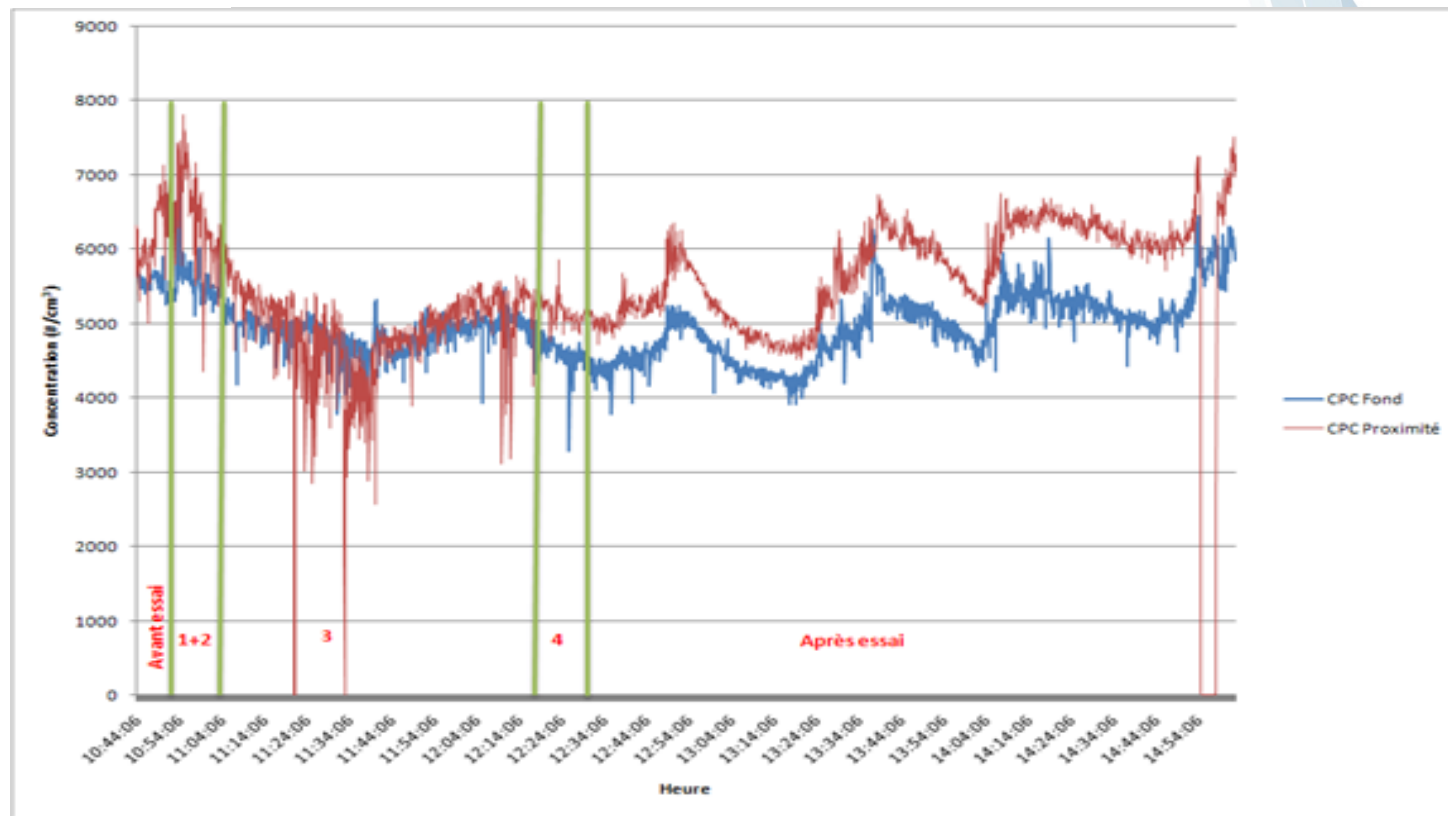


Positionning example



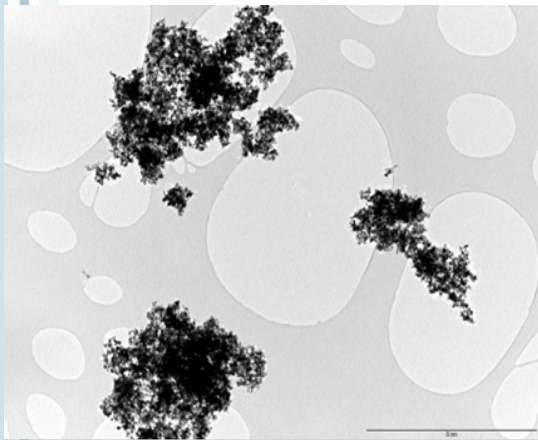


ECOMESURE



Sources studies

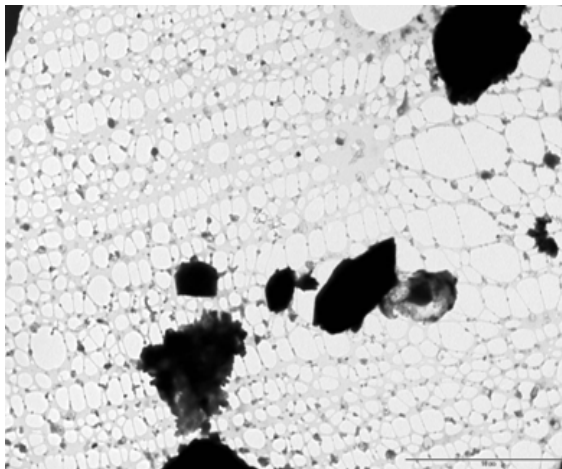
- Process : TiO₂ nano-structuré



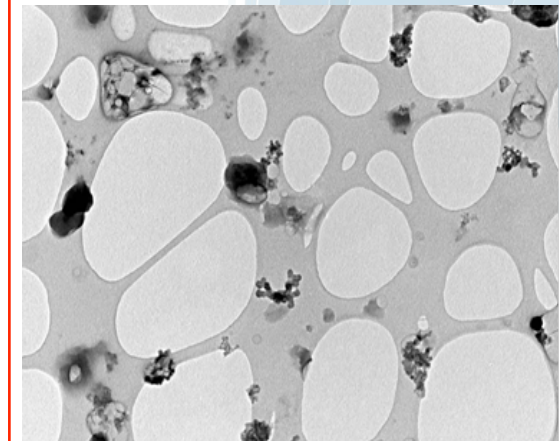
Measurement : Terrigenous coarse particle and soots

Coarse particle with various composition : silicium, limestone, sodium chloride, silicoaluminates, polymer fragments.

→ Terrigenous sources or industrial



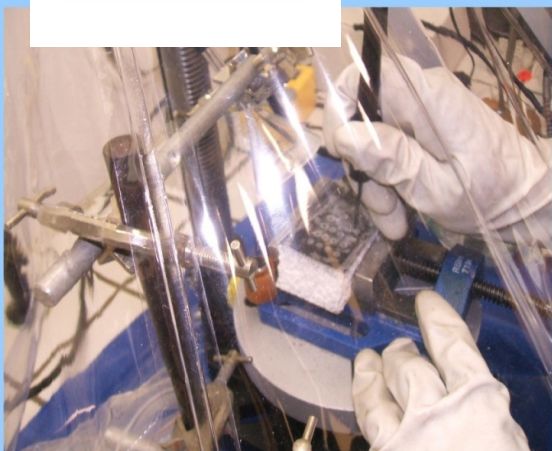
Soot particles aggregates
→ Combustion process



Degradations studies

Le Bihan et al.,
Advances in
Nanoparticles (ANP),
2013.

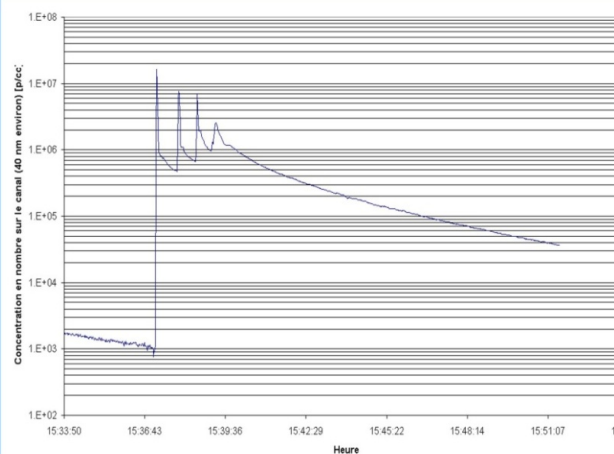
ECOMESURE



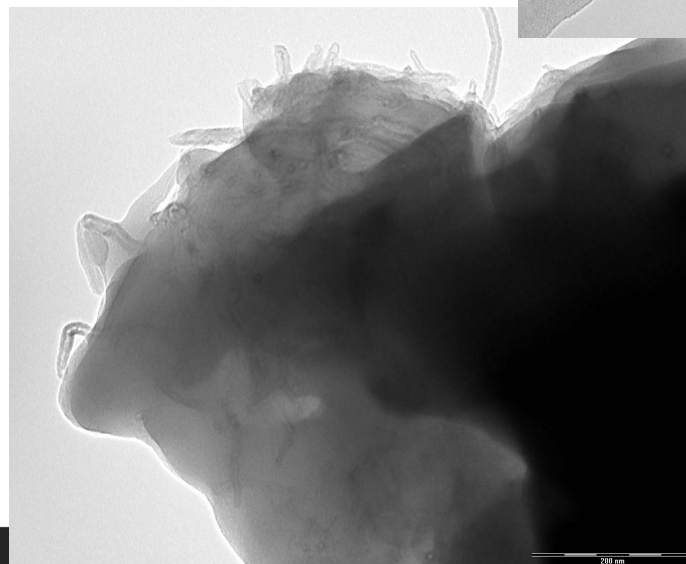
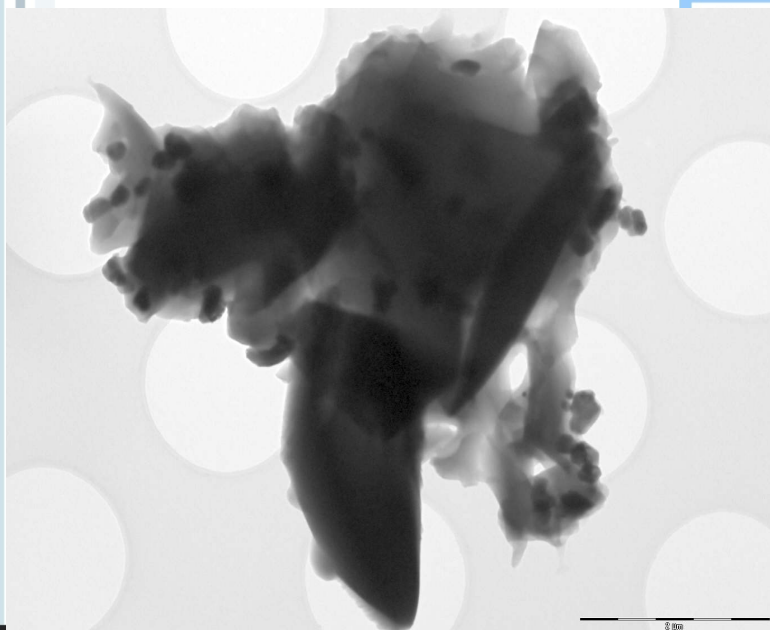
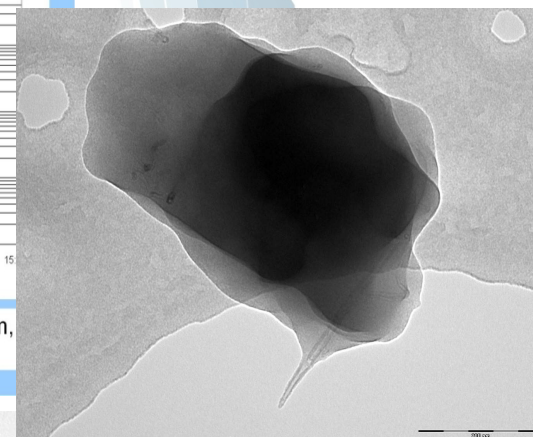
Picture n° 1: drilling in the glove-box.

ECOMESURE

INERIS



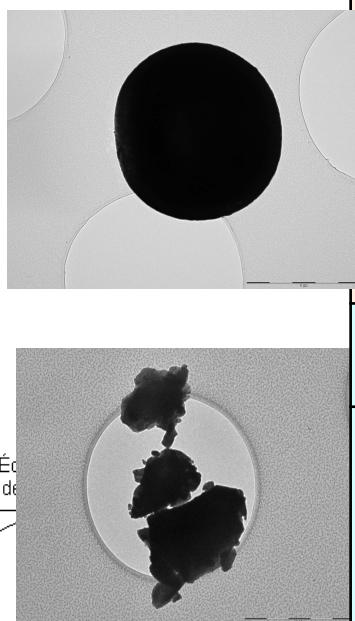
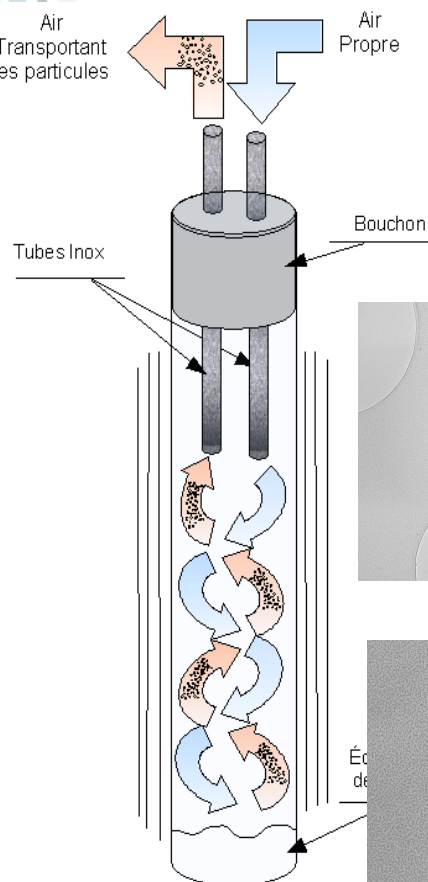
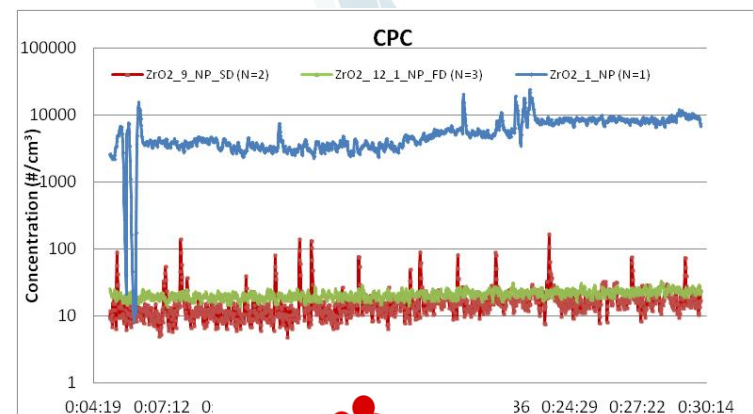
1: time variation of number concentration around 40 nm, grinding - example.



Nanomaterial conception

Morgeneyer et al. 2013
Le Bihan et al. , 2014
Nanosafe 2014.

Better understand
powder emissivity → ex.
control banding



Modified powders	Emissivity of modified powders (normalized to pristine powder)	Size range, nano-structure	CONCLUSION Improvement with respect to pristine powder
ZrO ₂ _9_NP_SD	Divided by 100	Spherical particles, of supra-micrometric sizes	Yes
ZrO ₂ _12.1_NP_FD	Divided by 100	"Polyhedral" particles of various sizes ranging from less than 1 μm to 7 μm. A few rare nanometric objects	Yes
PL_ZrO ₂ _PVP	Multiplied by 3	Particles of various shapes and sizes	No





New technologies

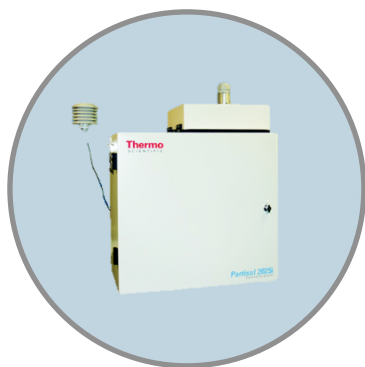
Naneos partector



- LDSA measurement, time resolution of 1 sec
- Wide concentration range, from 1 - 20'000 $\mu\text{m}^2/\text{cm}^3$
- Wide particle size range, from 10 nm - 10 μm
- Flow: 0.45 lpm
- Sampling efficiency: $\sim 3\%$ at 50nm.

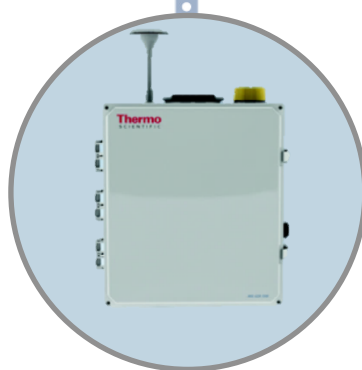
ECOMBOX

Universal solution to control instruments
remotely



Partisol 2025i

**1. Connexion to
samplers and
analysers**



ADR 1500



**2. Data on secured
and dedicated
web server**



**3. Access on tablets,
smartphones, PC ...**

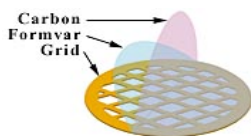


Questions?

THANKS FOR YOUR ATTENTION

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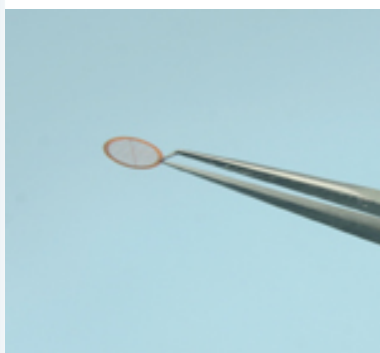
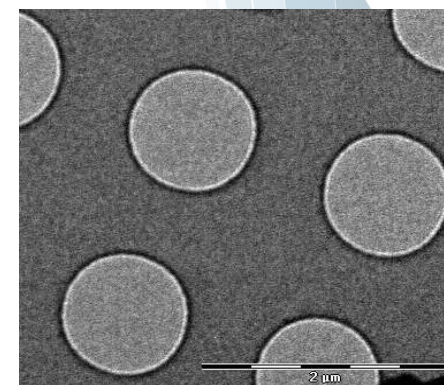
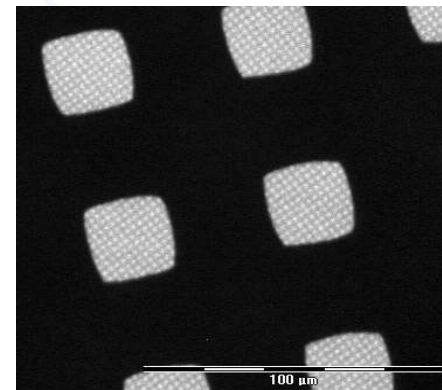
•Sampling for TEM analysis:



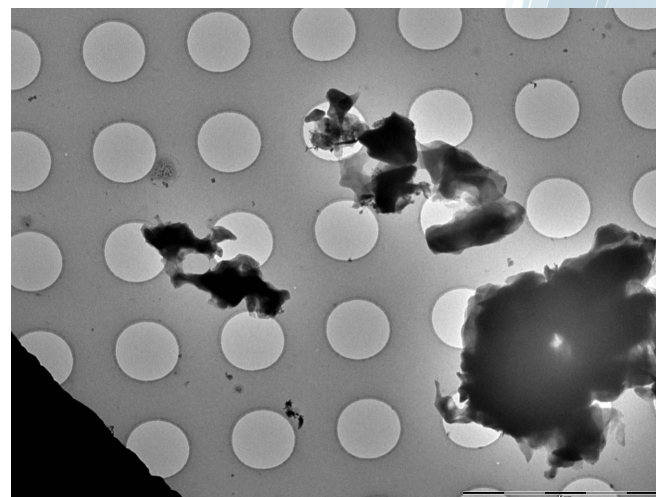
Formvar Carbon Grid
Tedpella



Mesh sizes : 400 Mesh = 37 μm
Tedpella



Sampling pump
Gilian



TEM grid
observation
Incineration
effluent of Black
Carbon
Size 5 μm