

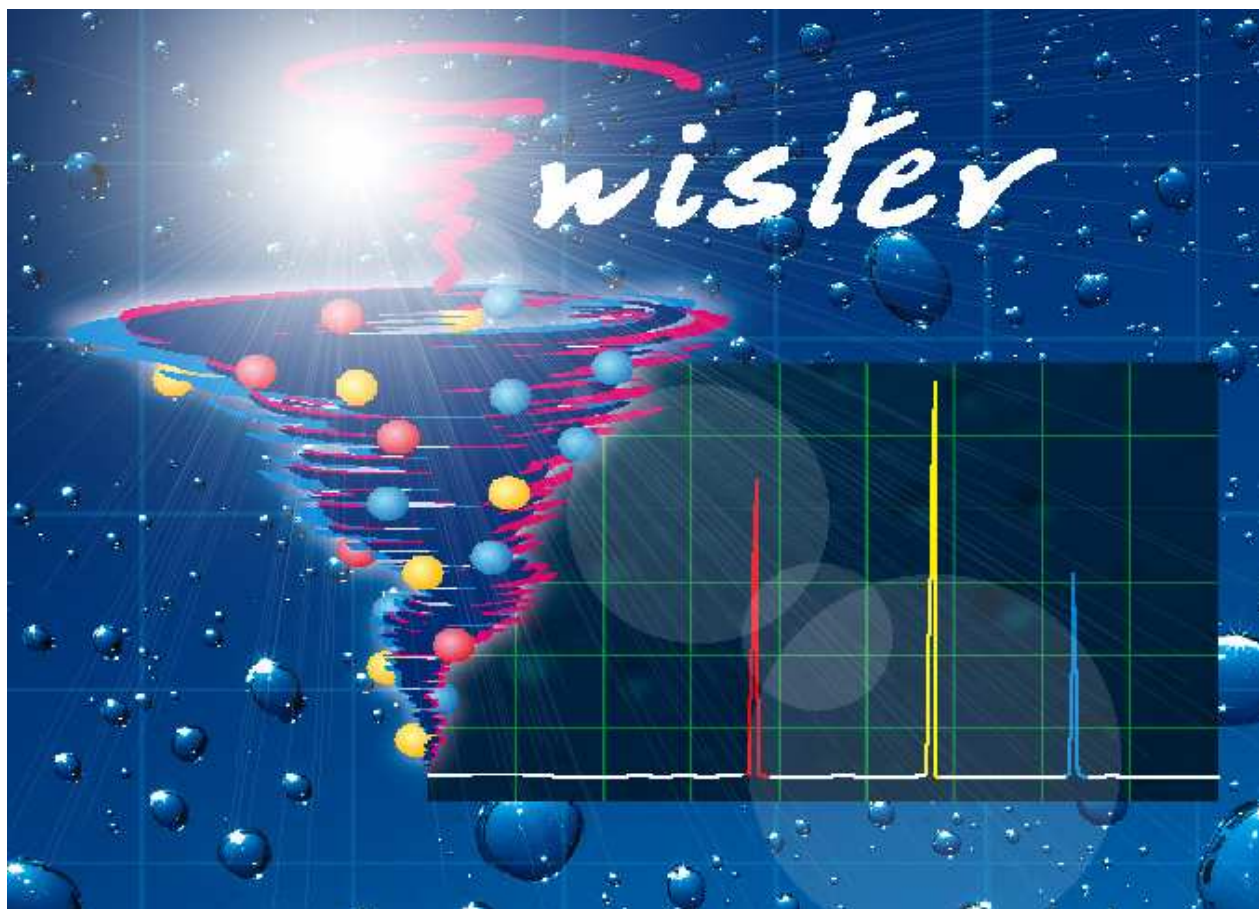
# ***2001-2011 : application de la SBSE pour la caractérisation et l'analyse des matrices environnementales***

David BENANOU,  
Veolia Environnement Recherche & Innovation



20/01/2011 ) STIR BAR SORPTIVE EXTRACTION 1<sup>st</sup> Technical Meeting

# 2001: SAMPLE PREP REVOLUTION

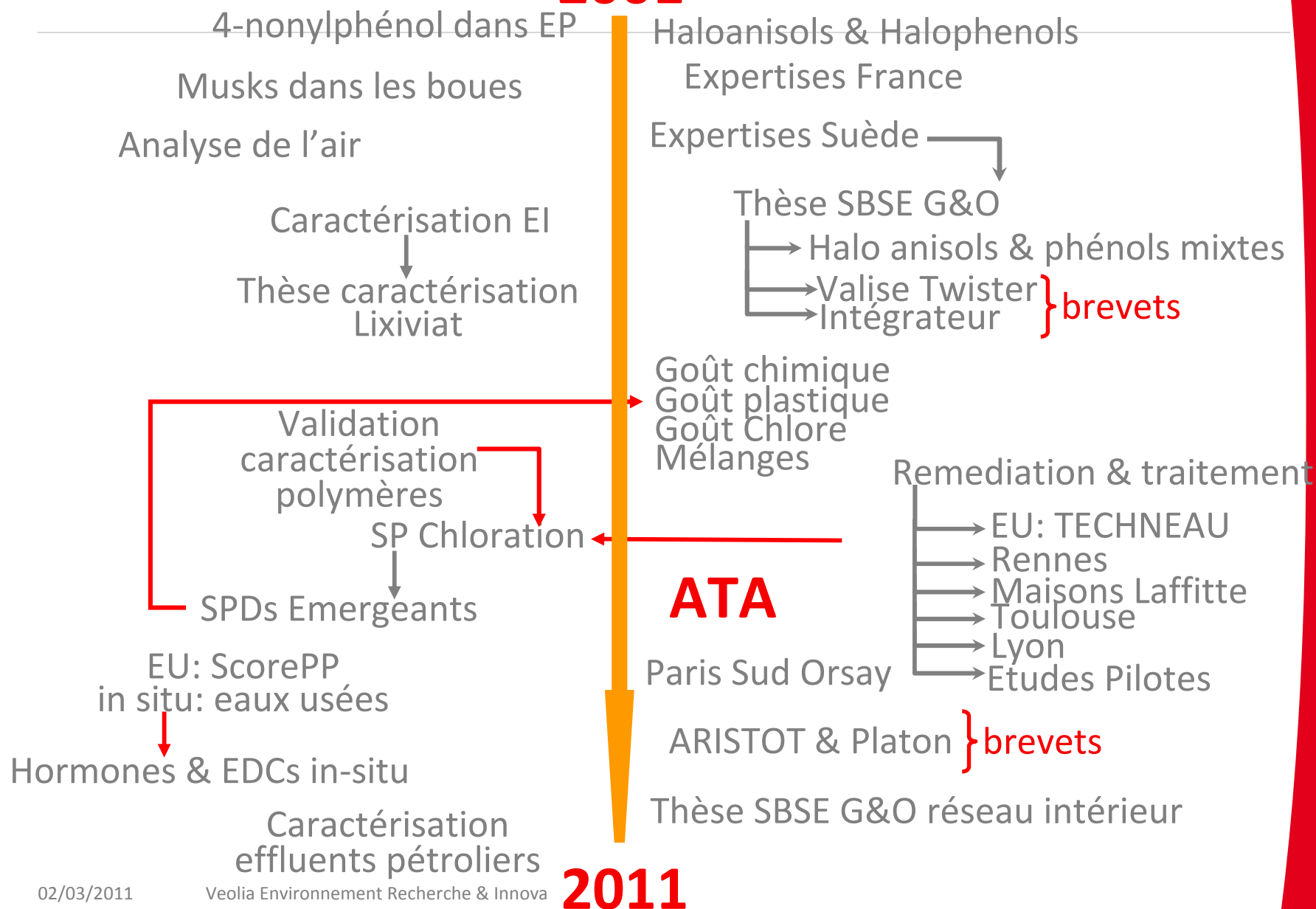


02/03/2011

# De la Genèse à la maturité



**2001**

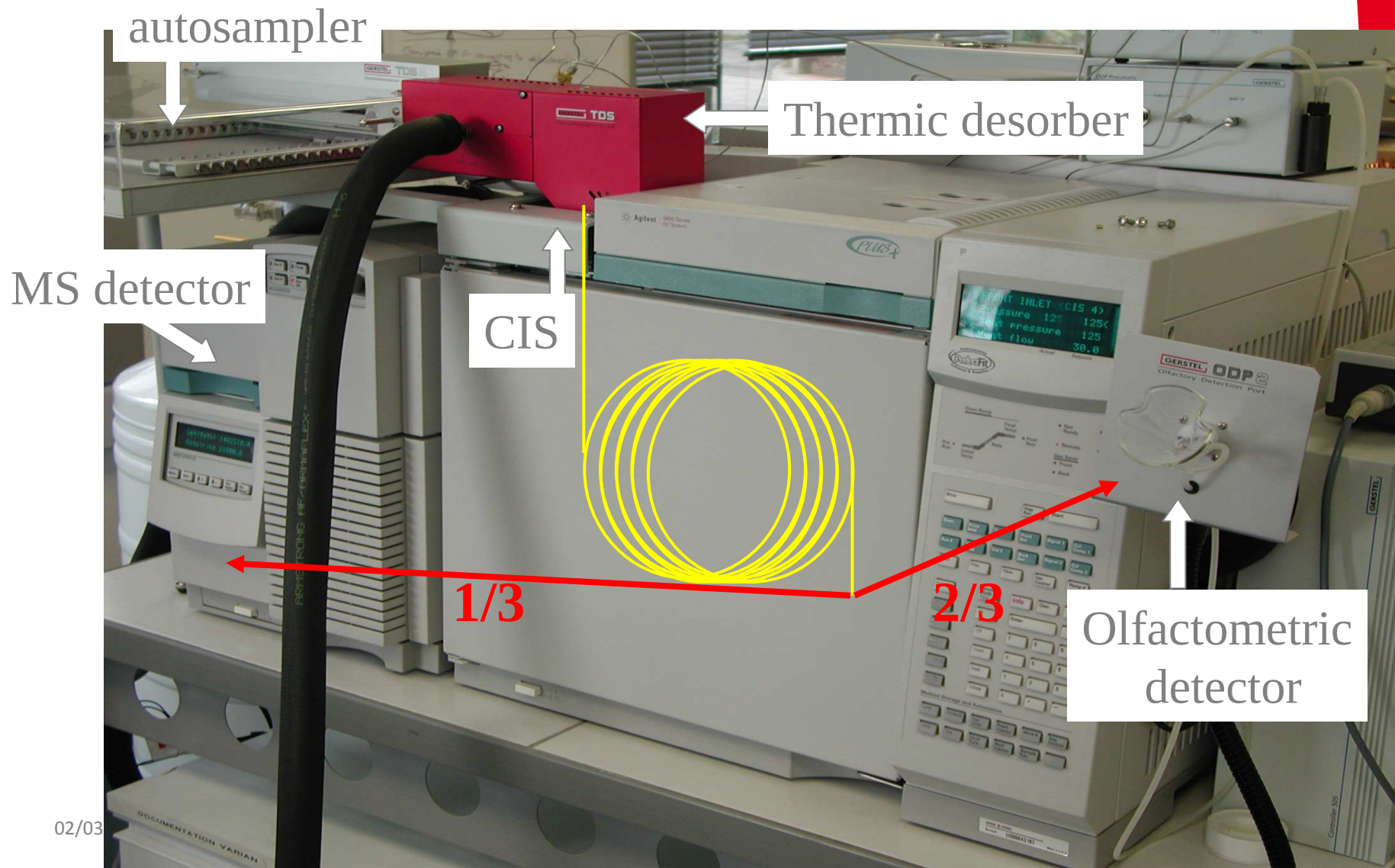


# Protocole général

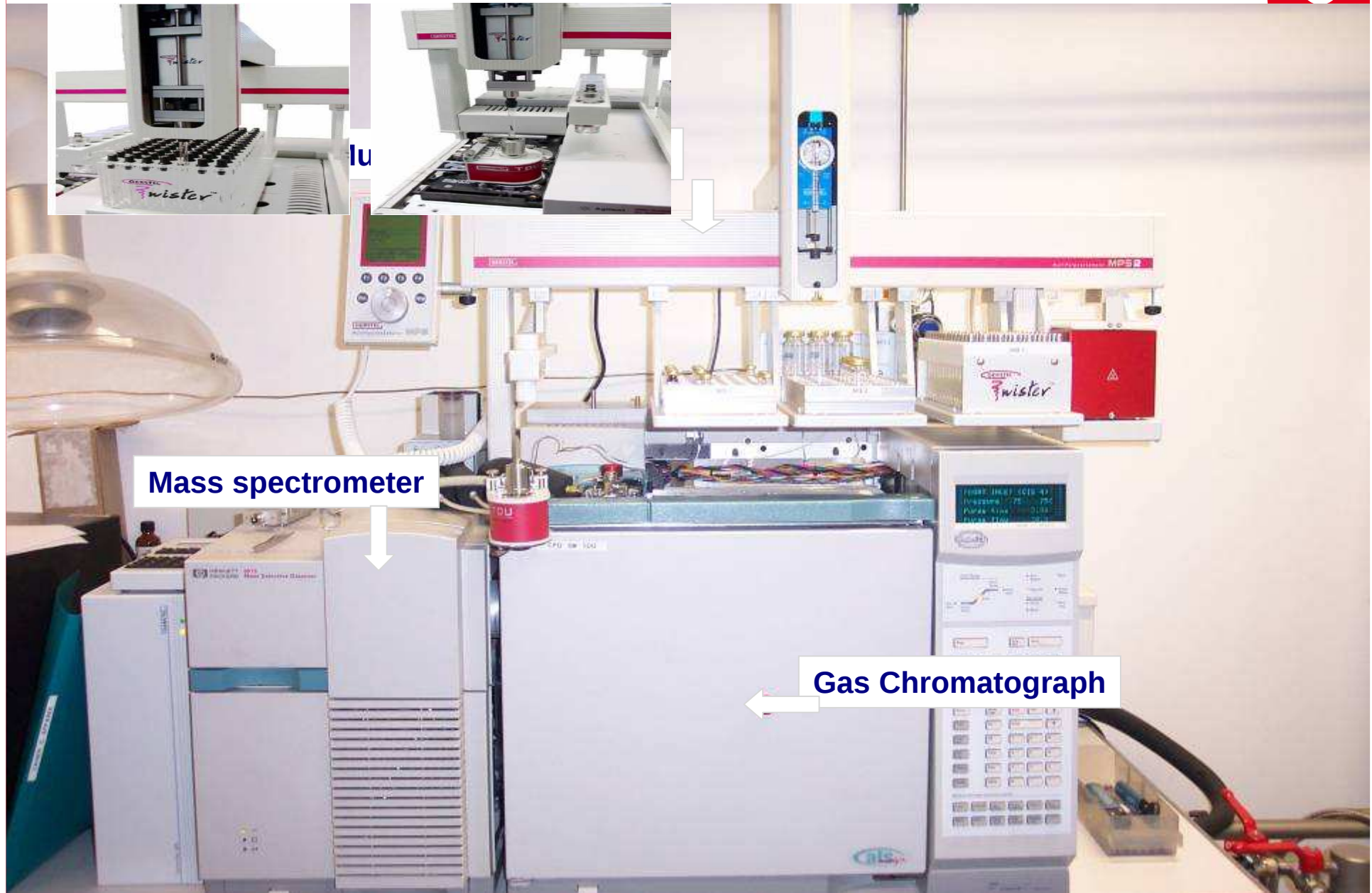


- **Introduce twister into water**
- **Stirring extraction**
- **Take twister out**
- **Rinse it with distilled water**
- **Dry it with clean wipes**
- **Introduce into a desorption tube**
- **Put the Tube into autosampler**
- **Thermic desorption**
- **Analysis**

# SYSTÈME ANALYTIQUE 1 puis...



# SYSTÈME ANALYTIQUE 2 à 12



# Procédure Analytique

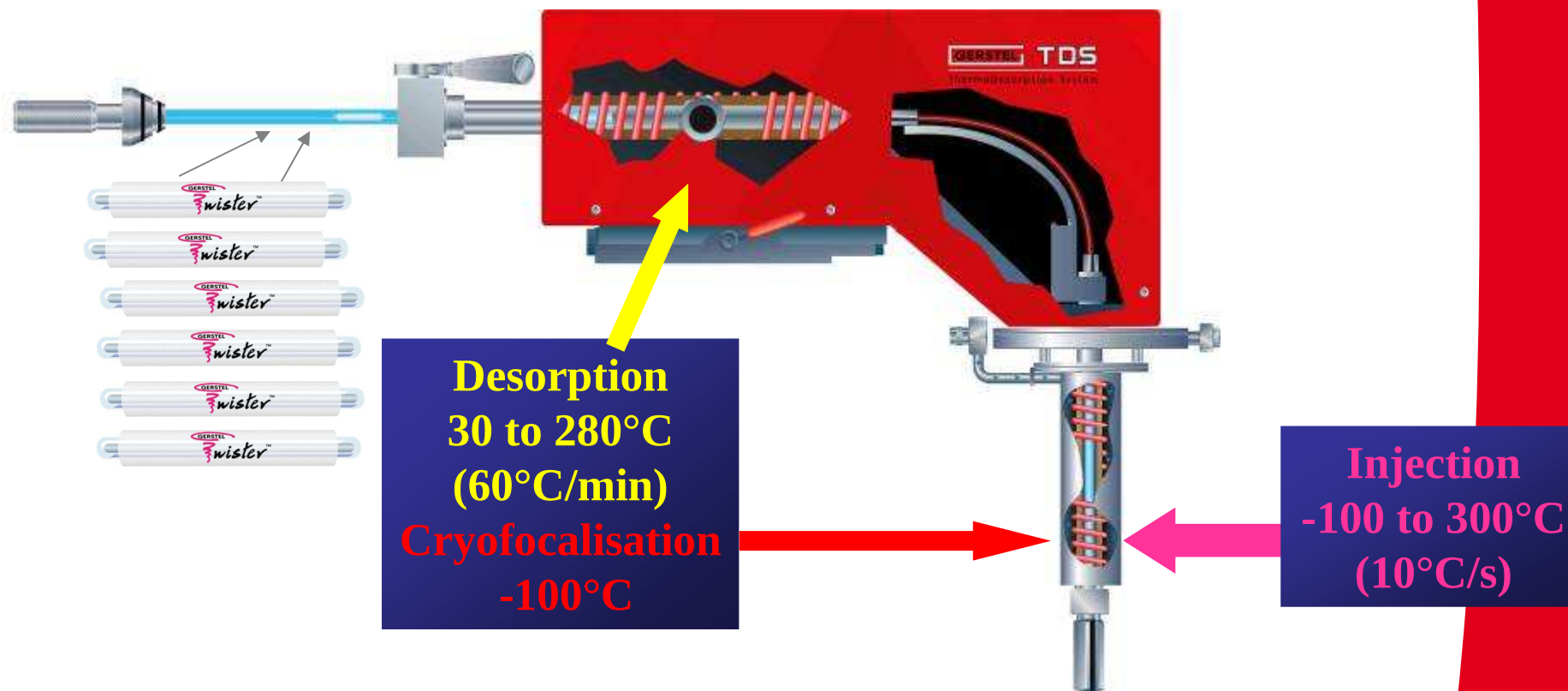


Sample volume : n x 100 ml

Extraction time : 2 h

Twister size : 2 cm

Desorption



# Synoptique de développement



-  **Optimisation du volume échantillon**
-  **Optimisation du temps d'extraction**
-  **Réactifs de dérivation in-situ : composés polaires**
-  **Stabilité du Twister après extraction**
-  **Utilisation d'isotopomères pour la quantification:**

# Rendement versus Volume Echantillon

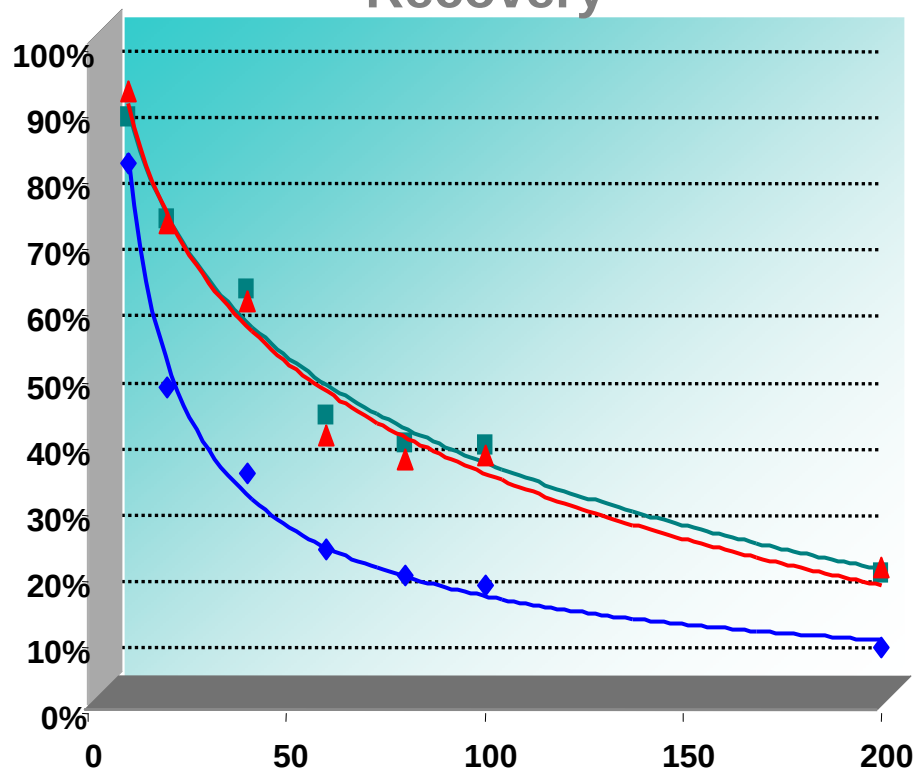


◆ MIB

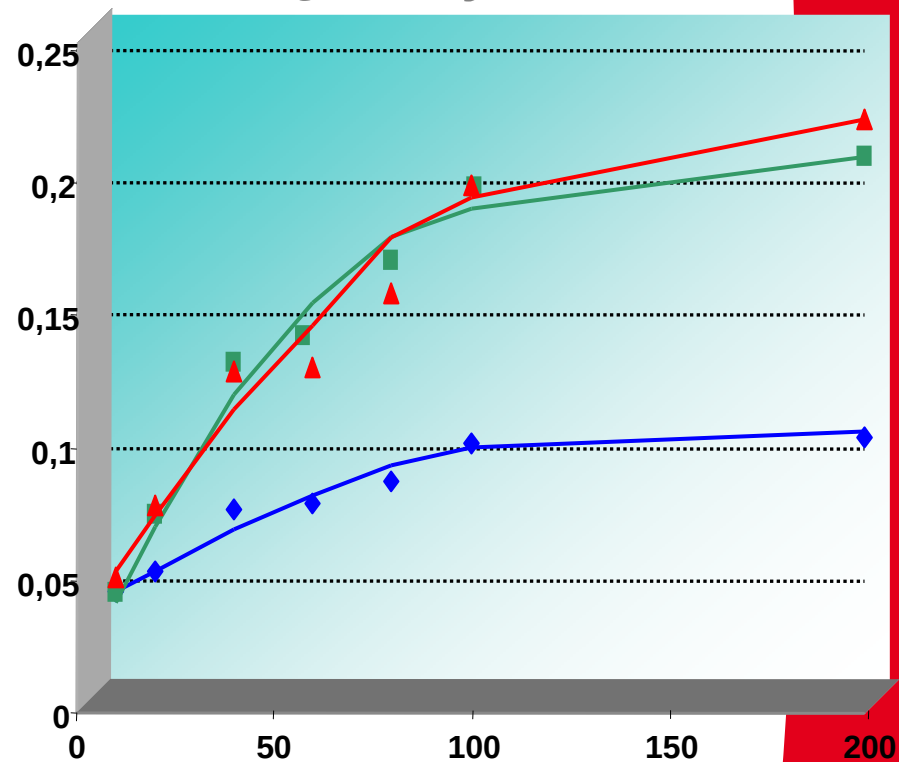
▲ 246 TCA

■ Geosmine

Recovery

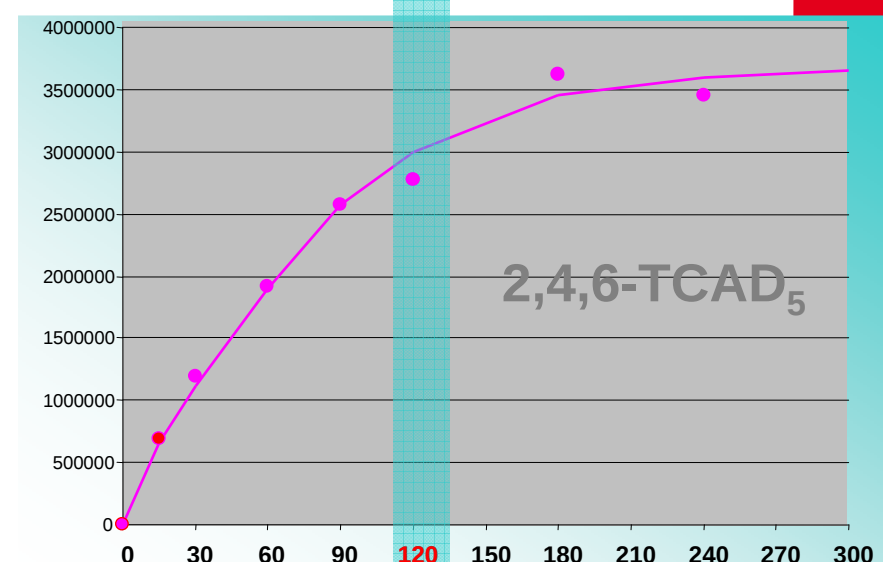
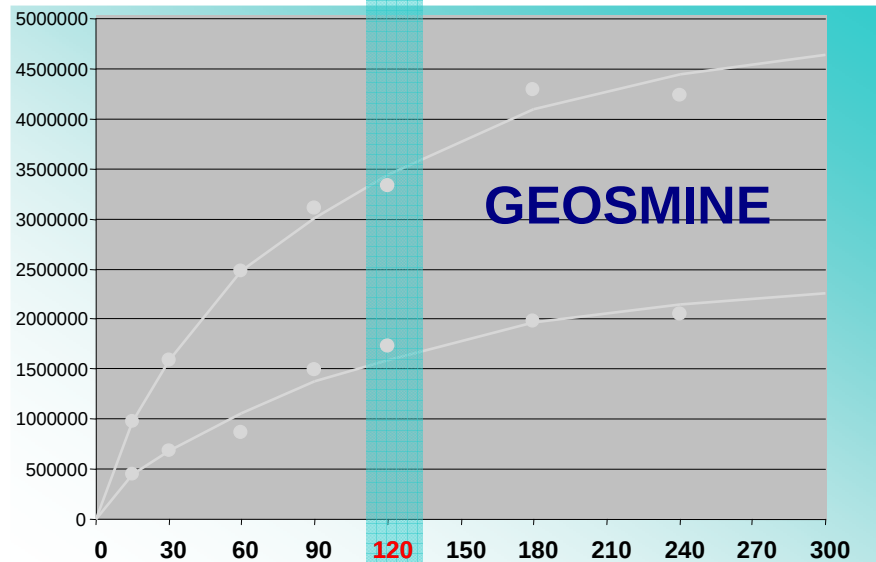
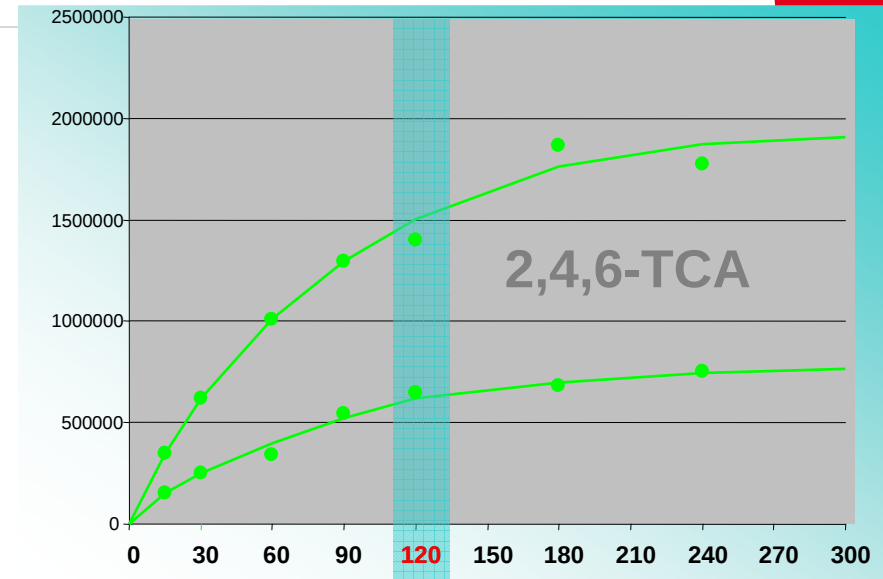
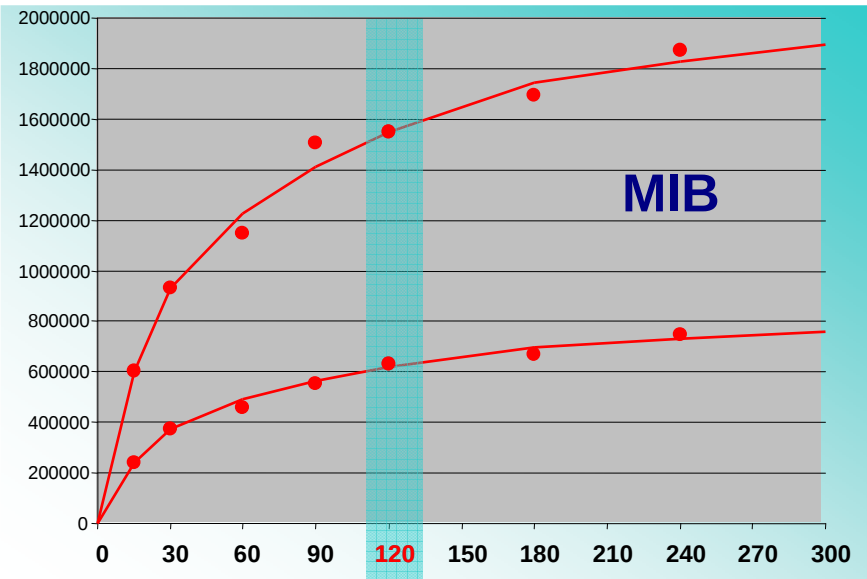


Quantity extracted



Sample volume

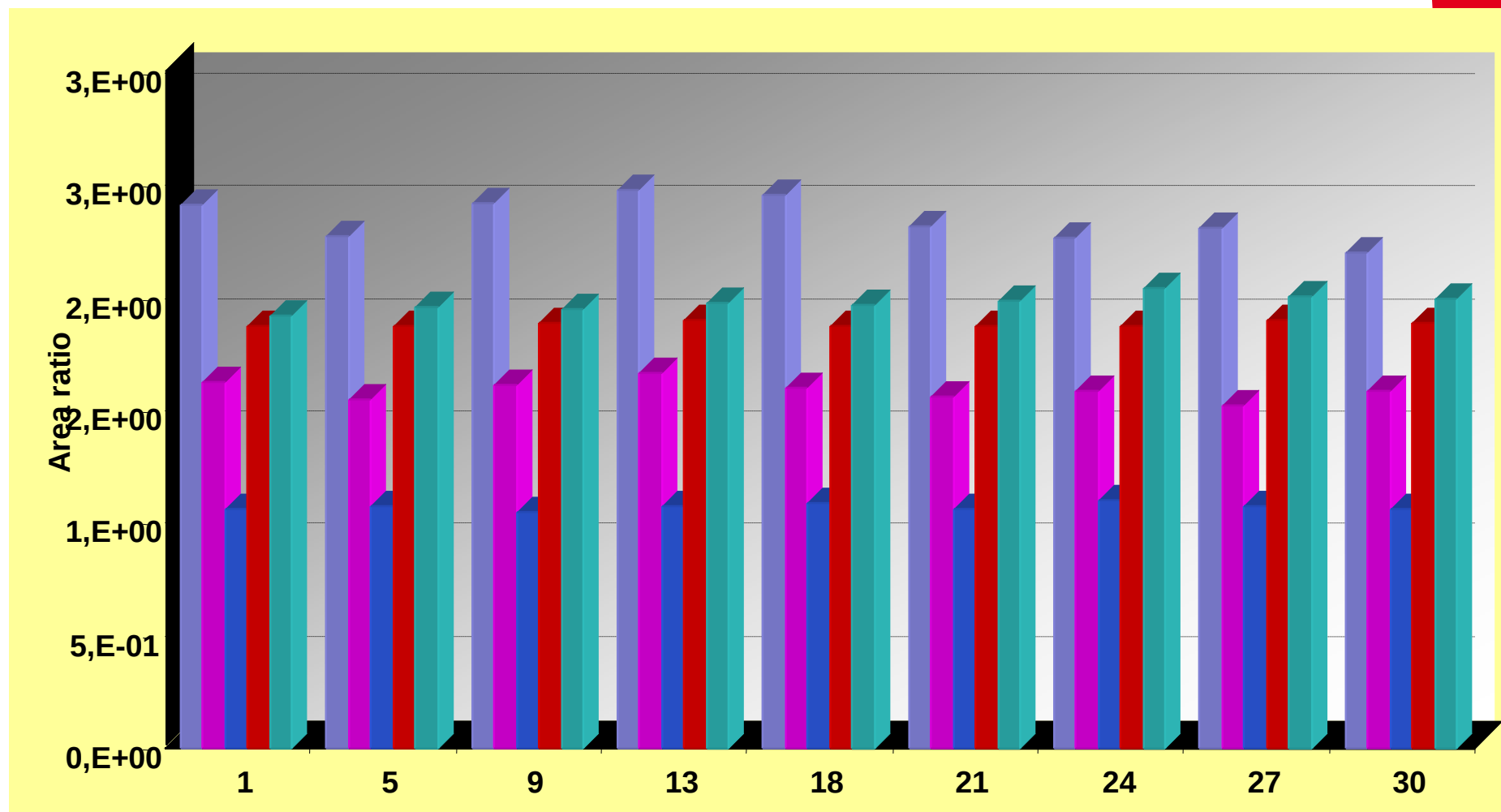
# Optimisation du temps d'extraction



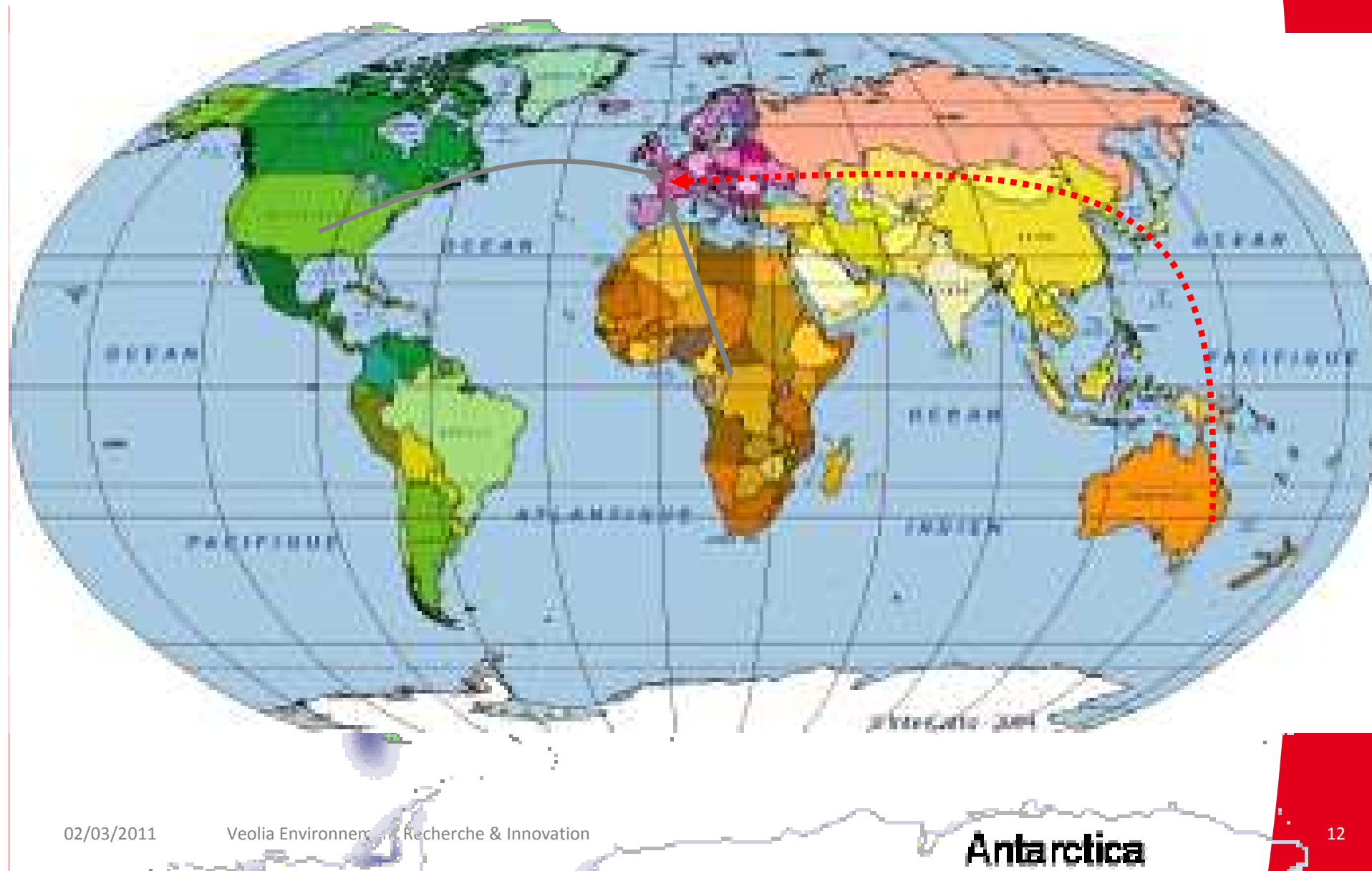
# Incidence du stockage après enrichissement



TBP 4-CPhenol 23diCphenol 246triCphenol PCP



# Laboratoires Twister: enrichissement in-situ



# Les outils...



- Utilisation conventionnelle
  - Goût récurant: piégeage au laboratoire
- Extraction in-situ
  - Être au plus près de la problématique
- Extraction chez le particulier
  - valise Twister (brevet)
- Extraction en continue sur le réseau
  - Intégrateur (brevet)
- Extraction en continue chez particulier
  - Robinet Twister (brevet)



## Récurrence...laboratoire



02/03/20

# Procedures pour:

## haloanisols

- 100 mL of water
- 40 µL of Labelled IS mixtures
- Extraction for 2 h
- One shot desorbtion
  - TDS 30 to 280
  - CIS – 50°C
  - Column RXI 5 MS
  - Up to 340°C



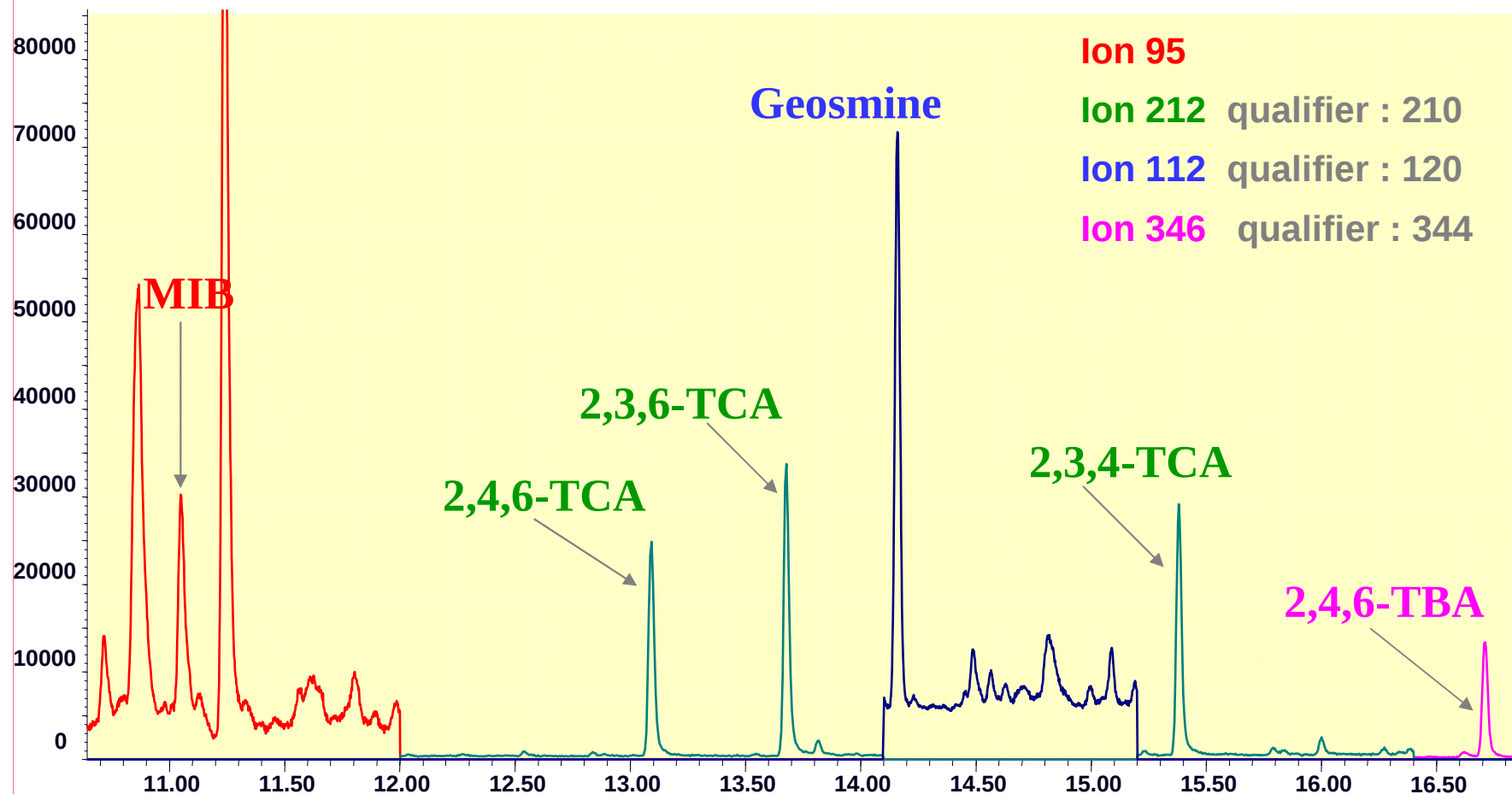
## halophénols

- 100 mL of water
- 1g potassium Carbonate
- 500 µL acetic anhydride
- 40 µL of Labelled IS mixtures
- Extraction for 1 h
- One shot desorbtion
  - TDS 30 to 280
  - CIS – 50°C
  - Column RXI 5 MS
  - Up to 340°C

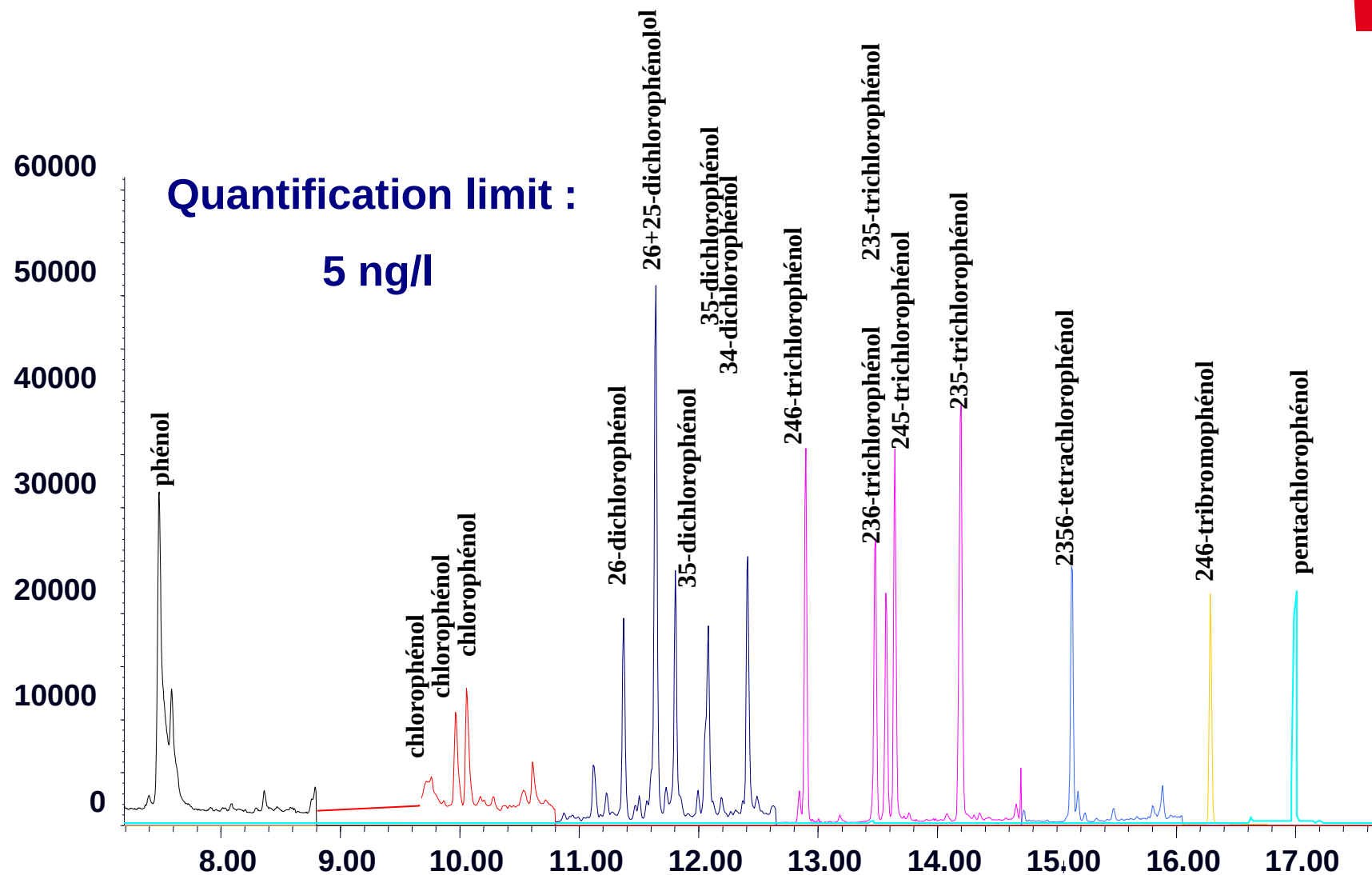
# Haloanisols: GC/MS EI (Single Ion Monitoring)



**Tap water spiked with 2 ng/l**



# Halophénols: GC/MS EI (Single Ion Monitoring)



# Very First Twister Lab

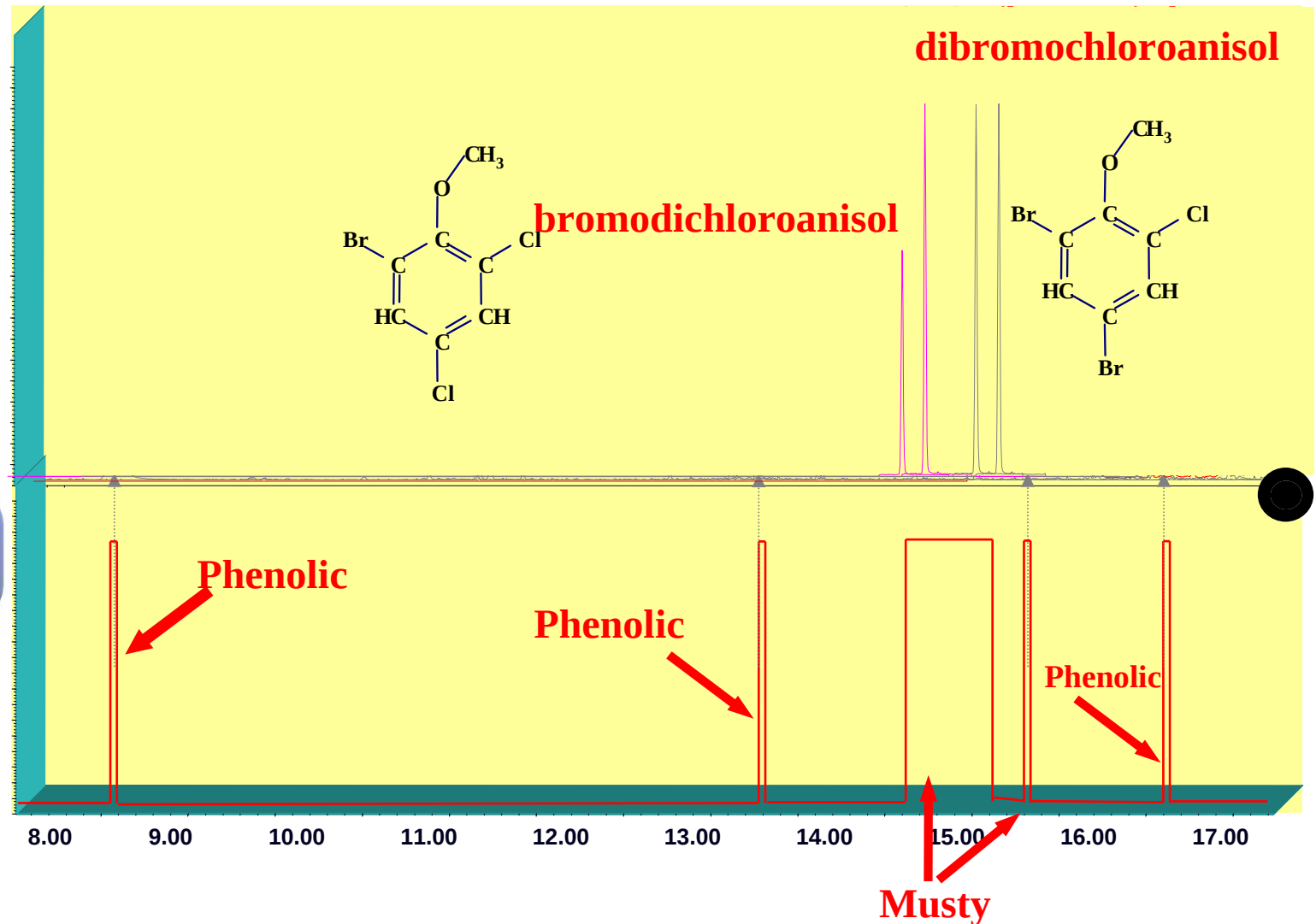


# Eau du robinet Suédoise



**MS  
detection**

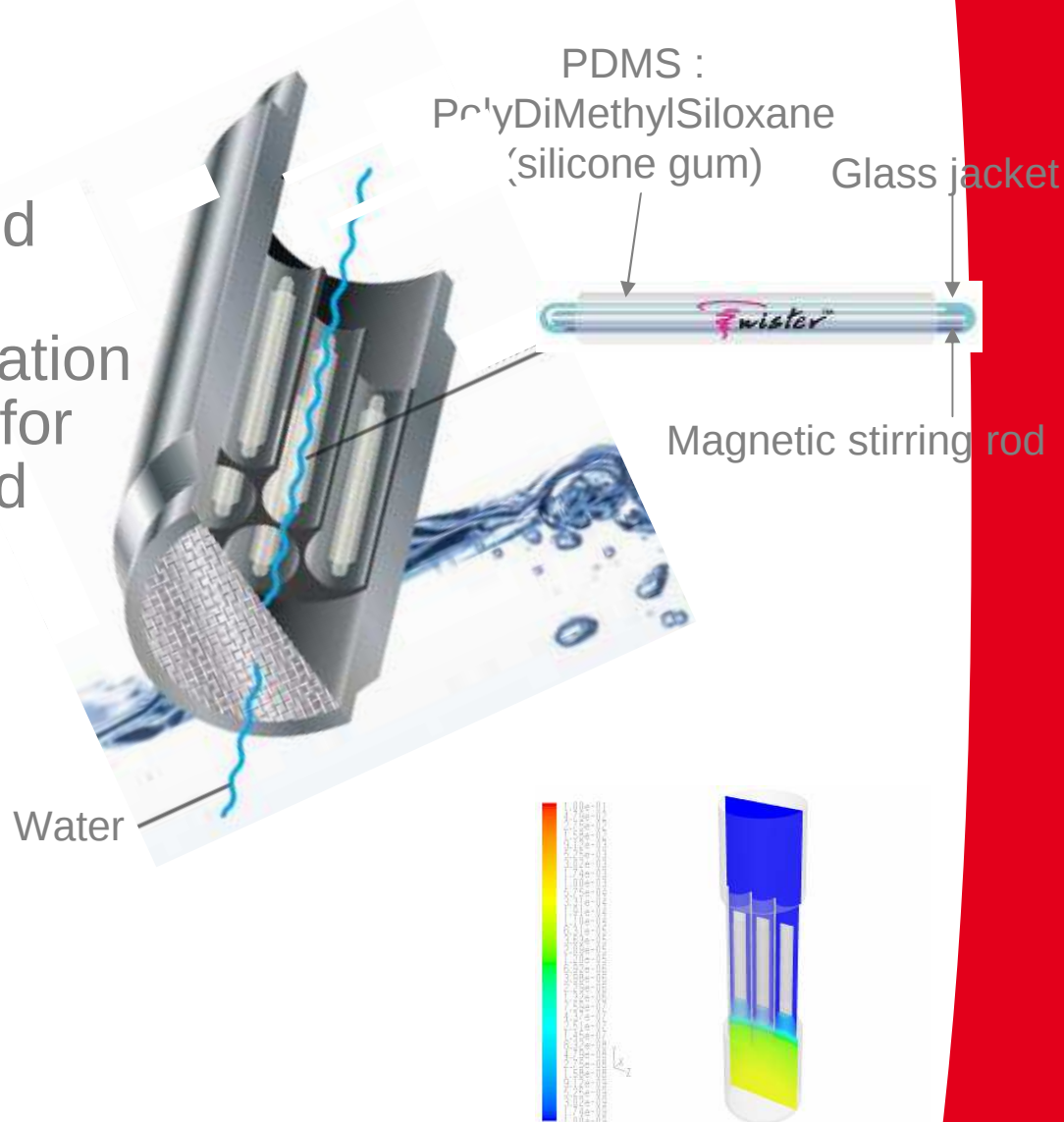
**sniffing  
detection**



# ARISTOT – Un enrichissement au robinet



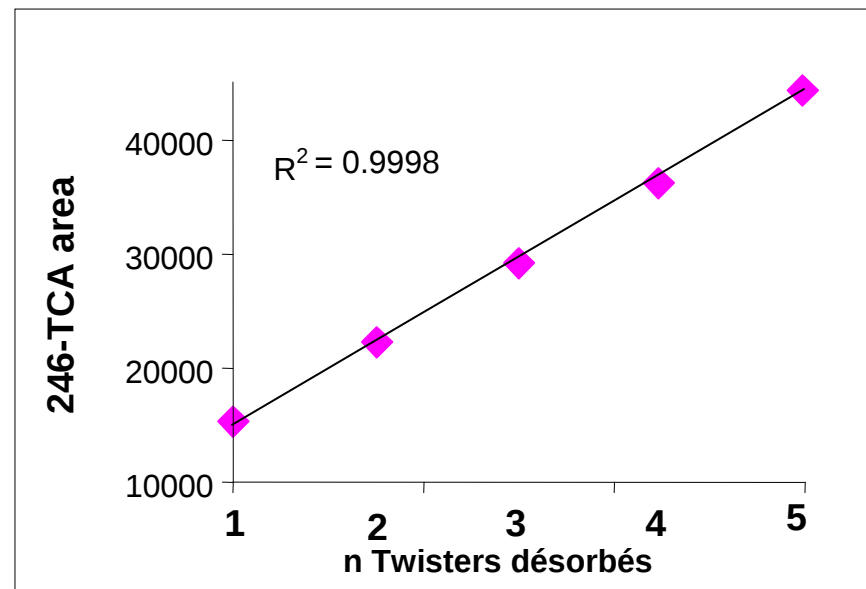
Advanced relevant investigation sampler for taste and odor at tap



# Du *multishot* à la Multidesorbtion



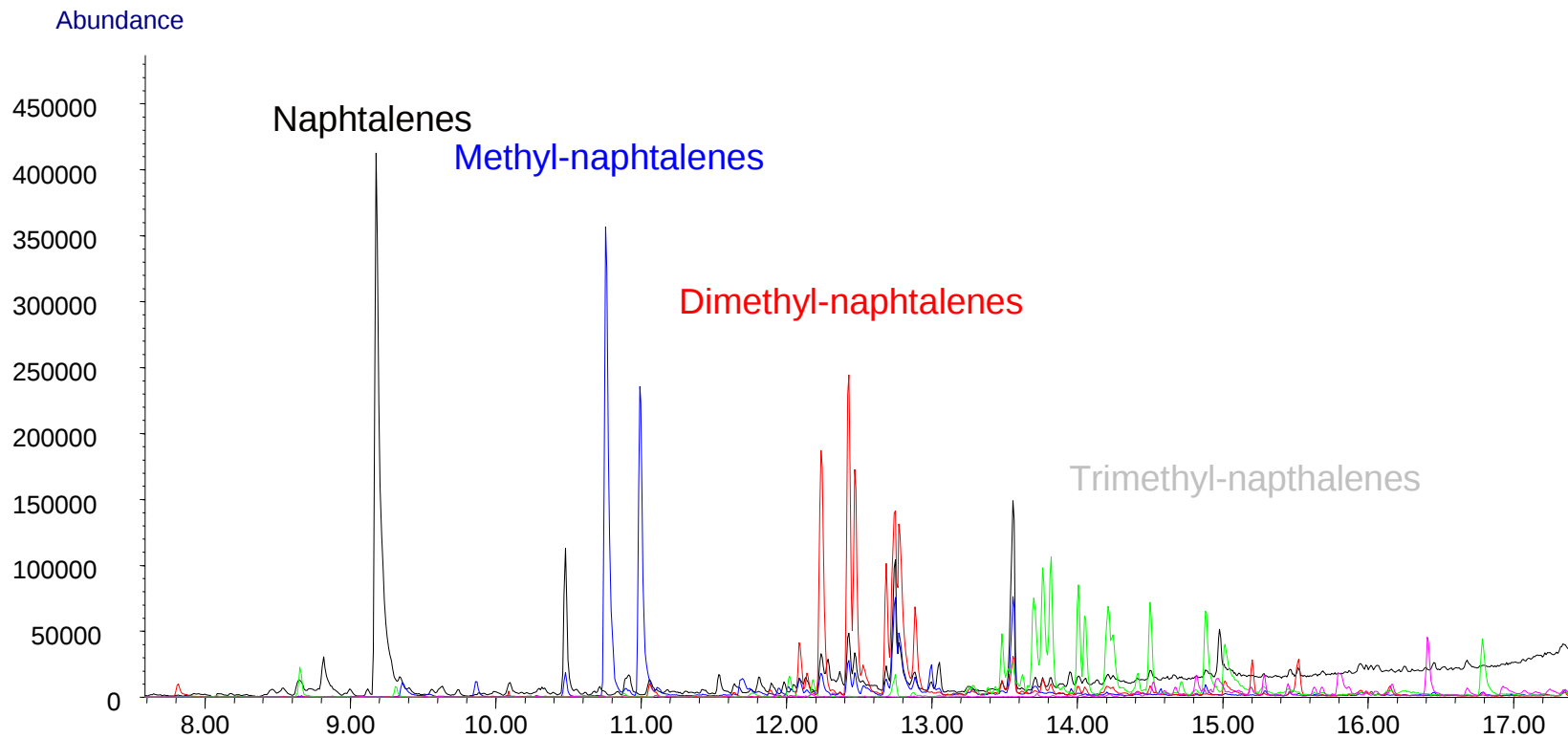
- Désorbtion successive de 5 Twister avant 1 seule analyse chromatographique
  - constant cryocooling ( $-70^{\circ}\text{C}$ )
  - no run (0.1 min)
  - jusqu'à 5 désorbctions ( $5 \times 147 \mu\text{L}$  PDMS et  $-50^{\circ}\text{C}$ )
  - 1 seule analyse chromatographique
  - **Haute sensibilité**



# T&O episode



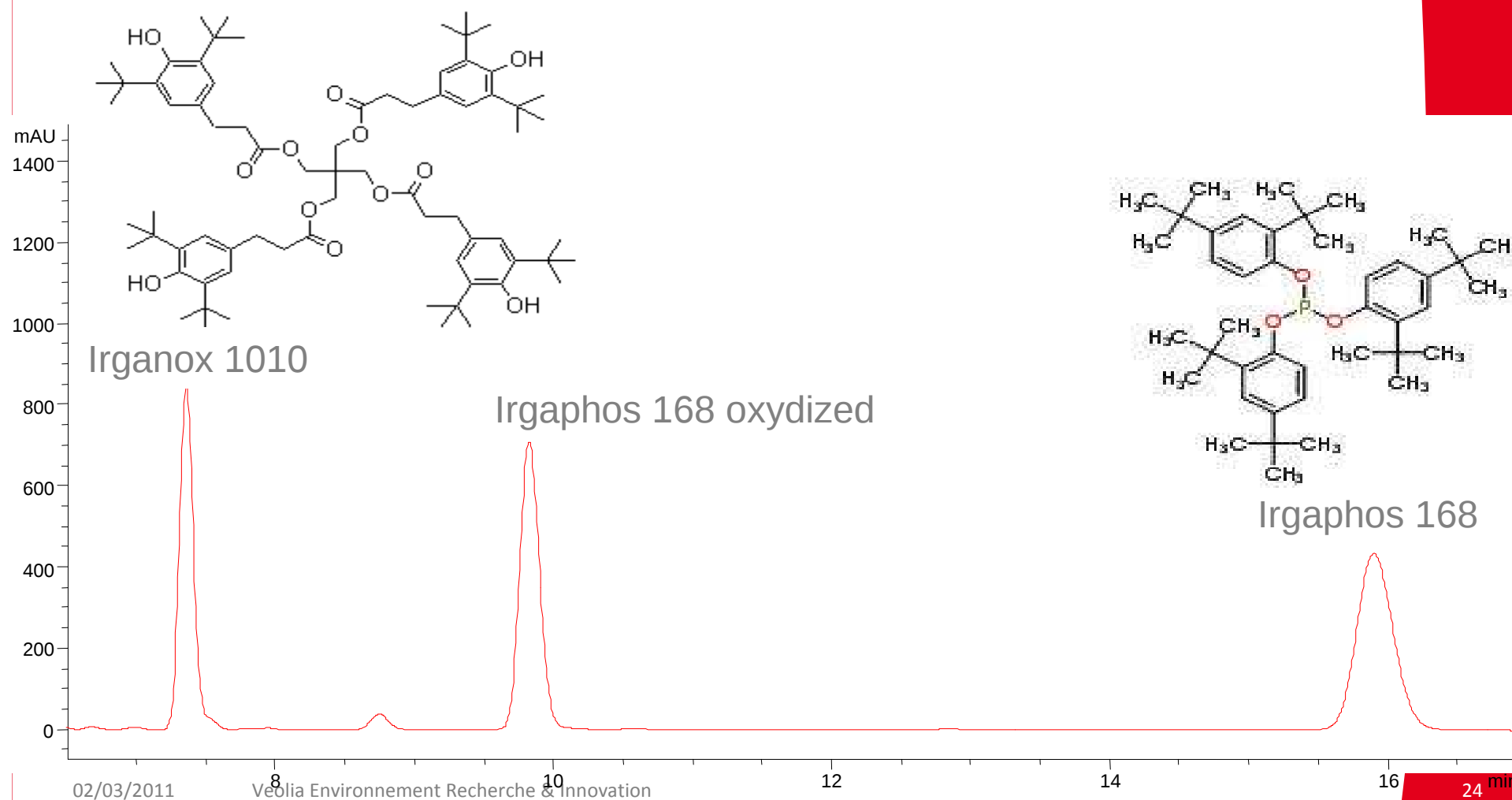
- Case 3
  - odeur épisodique chimique
  - appartement
  - Somme des HAPs: 2086 ng/L



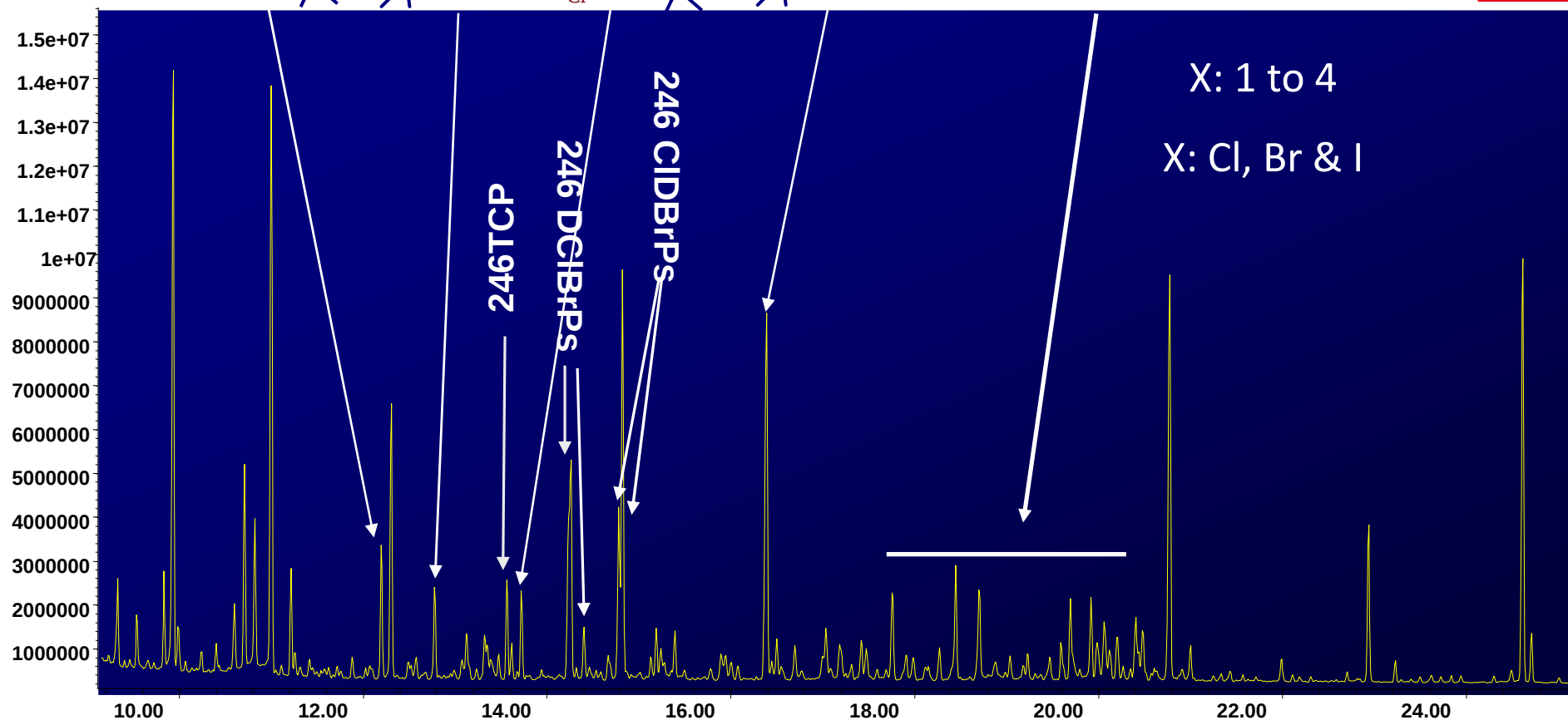
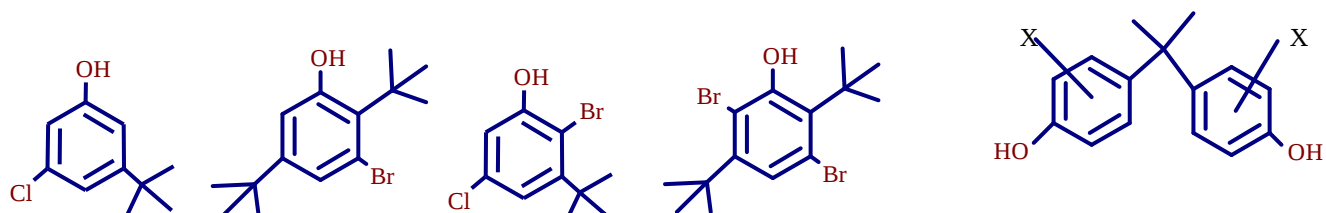
# Caractérisation des polymères



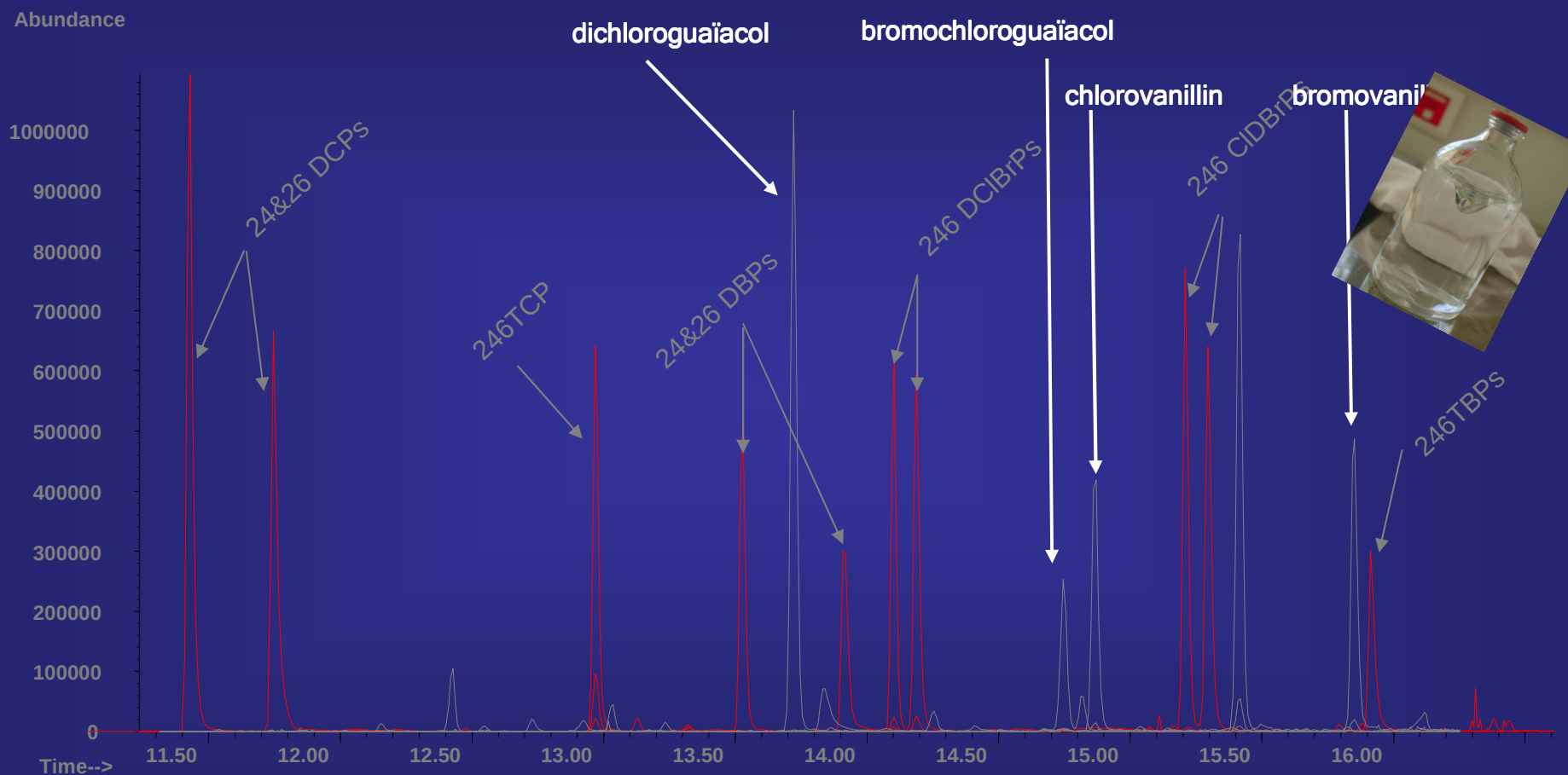
# LC-MS après lixiviation



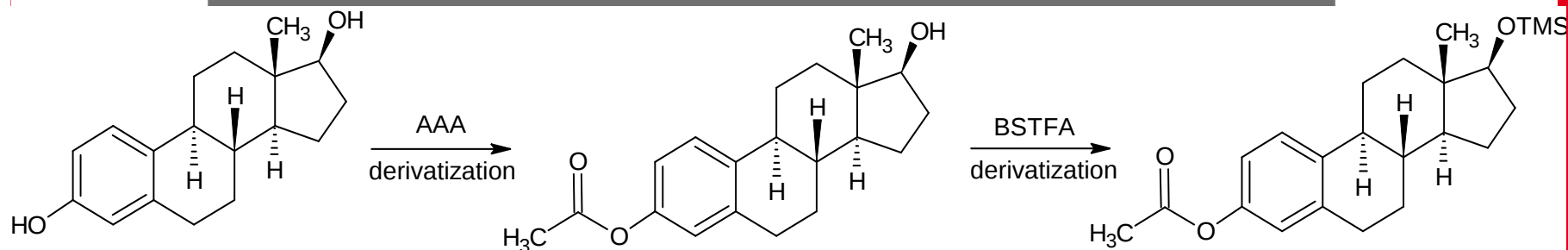
# TD-GCMS: stirring extraction with PDMS stir bar



# Screening approfondi: nouveaux SPDs



# Hormones & EDCs dans les eaux usées



# ISOCRATE – le ROBOT Twister » et les eaux usées



100 mL outlet Waste Water

5 mL of  $K_2CO_3$

500  $\mu$ L of Acetic Anh

Twister: 2 cm X 1 mm

Stiring Extraction: 2h

In  
Situ  
Off-flavor's  
Capture by  
Recurring and  
Automated  
Twisters  
Extraction



# Hormones & EDCs avec SBSE GC-MSMS.

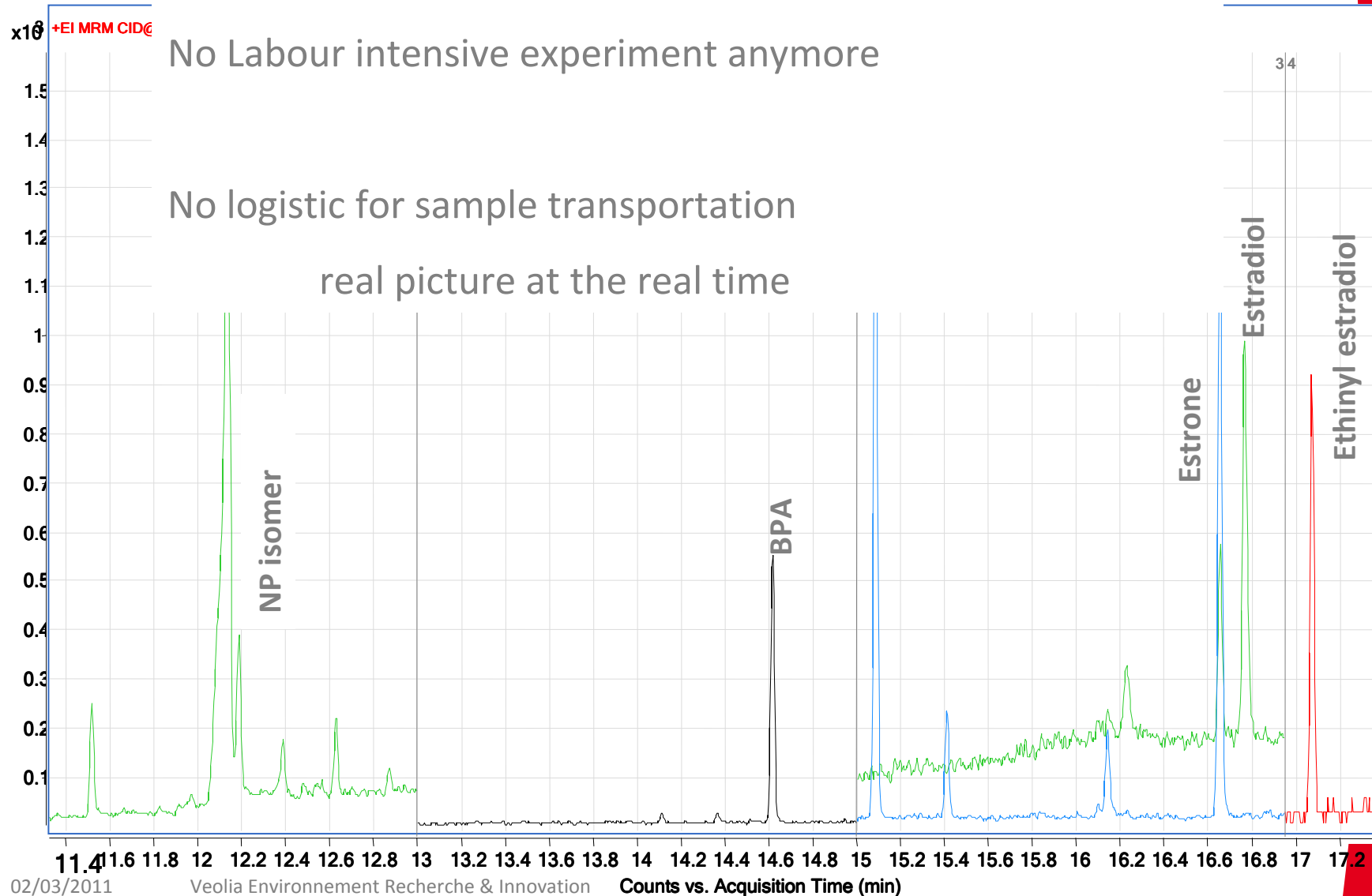


Low Coast Investment compare to LC System

No Labour intensive experiment anymore

No logistic for sample transportation

real picture at the real time



# SBSE – GC Comprehensive



## ● GCxGC-TOF LECO PEGASUS IV

Multipurpose autosampler

- Liquid injection
- Large Volume injection
- Thermo-desorption
- Gas injection (HS)
- Pyrolysis (soon)

Thermodesorbor

- Twisters / solids analysis

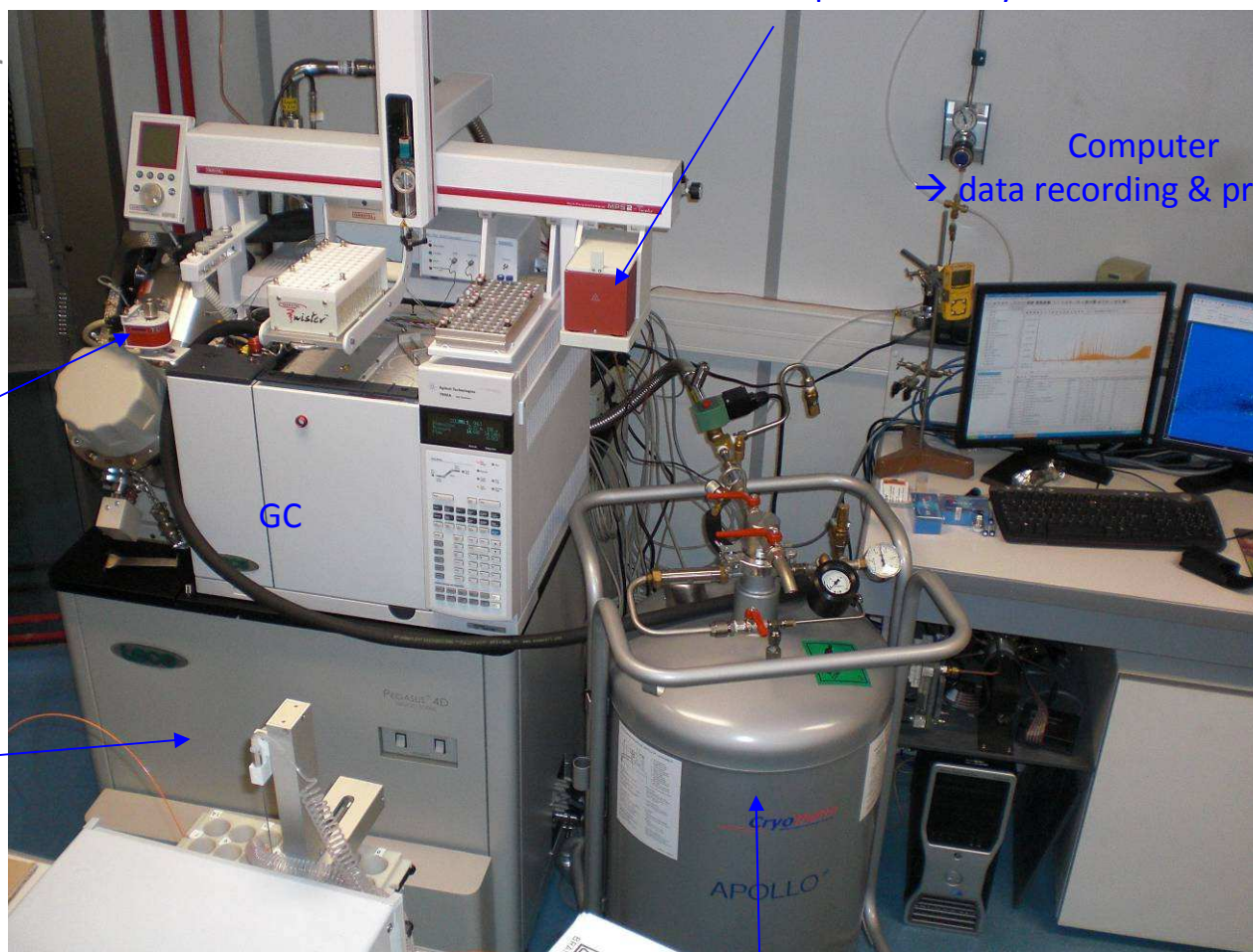
Time Of Flight  
Mass Spectrometer

HeadSpace module

- Volatiles compounds analysis

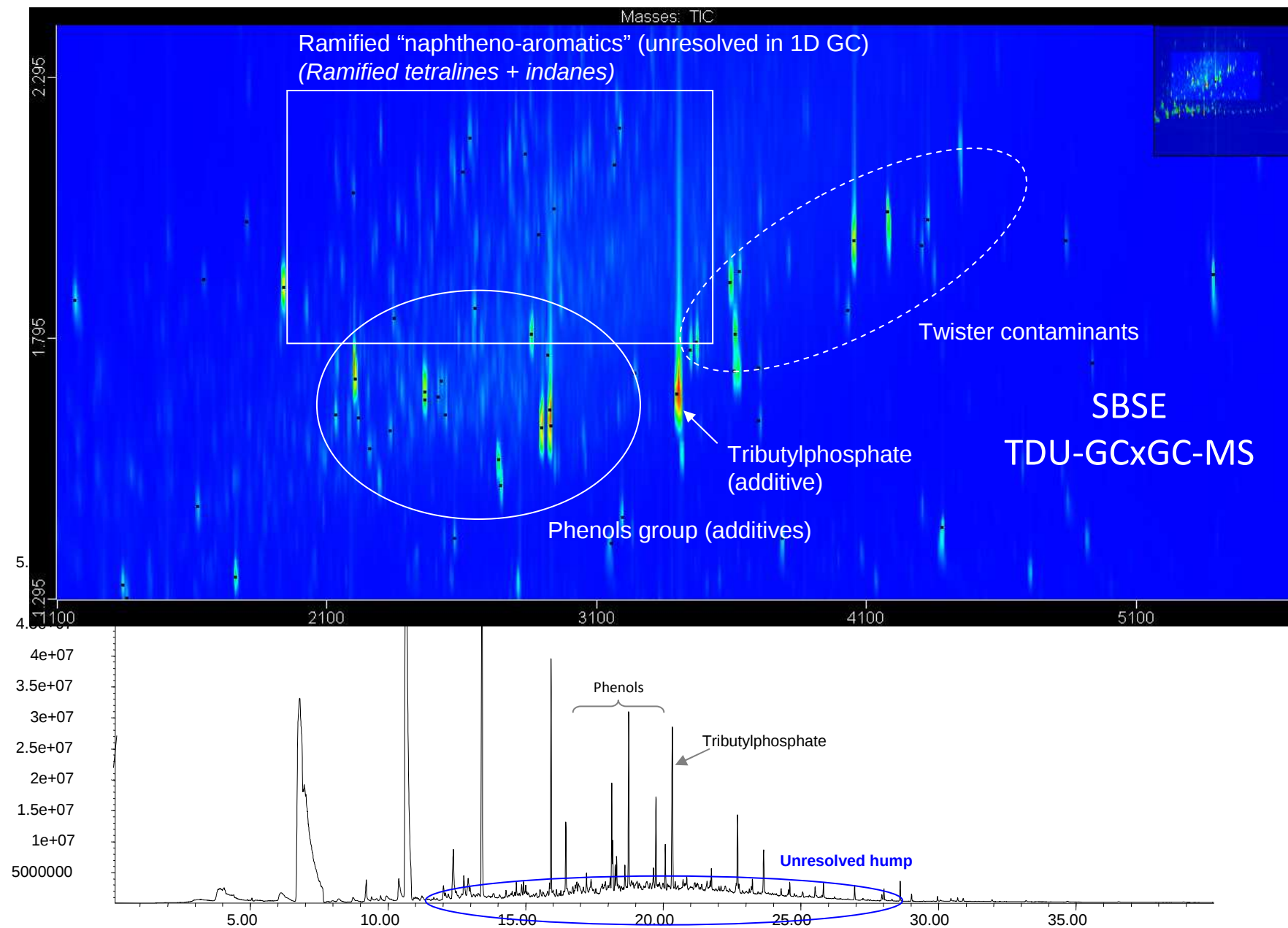
Computer

- data recording & processing

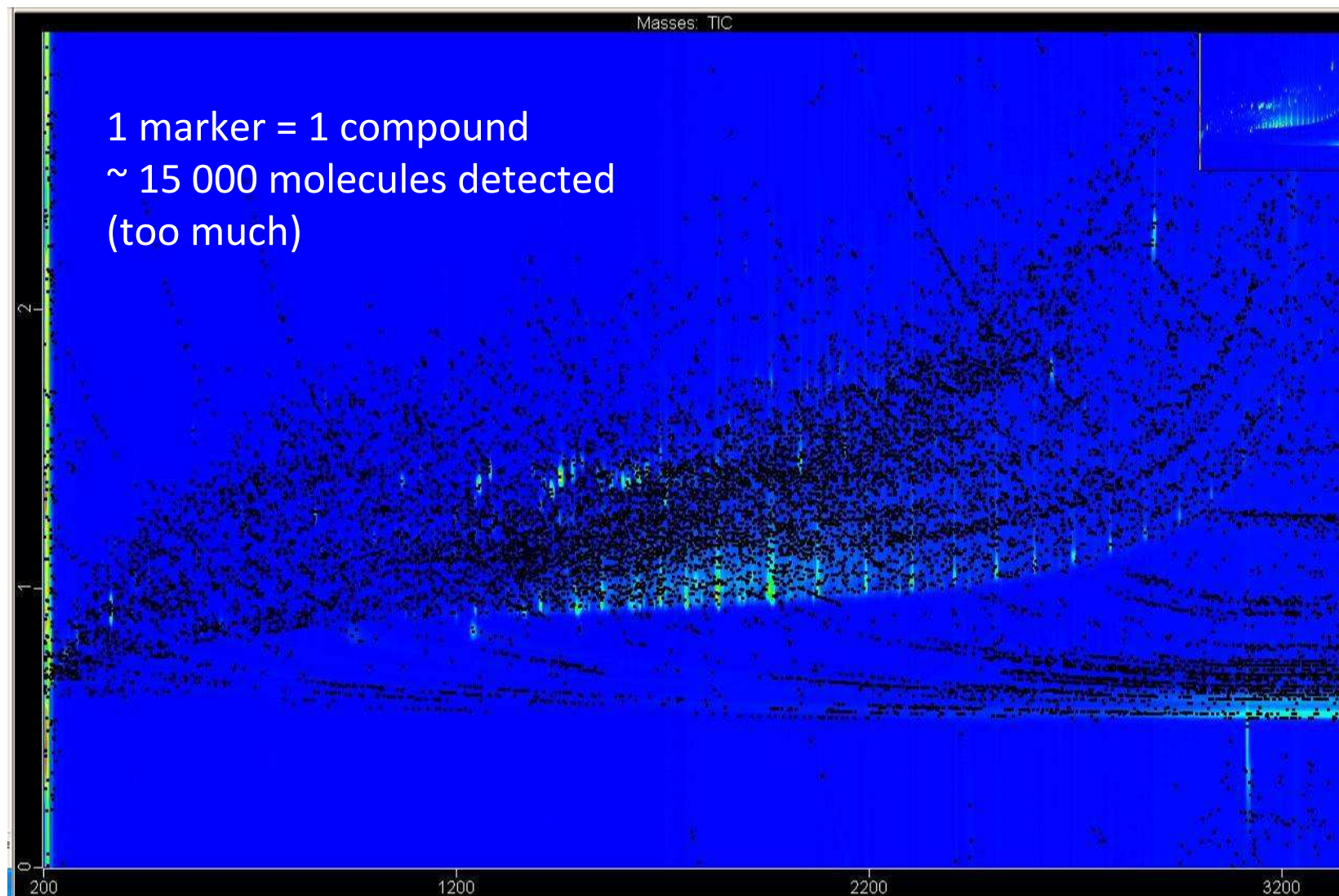


Liquid Nitrogen tank  
→ Cryogenic modulator

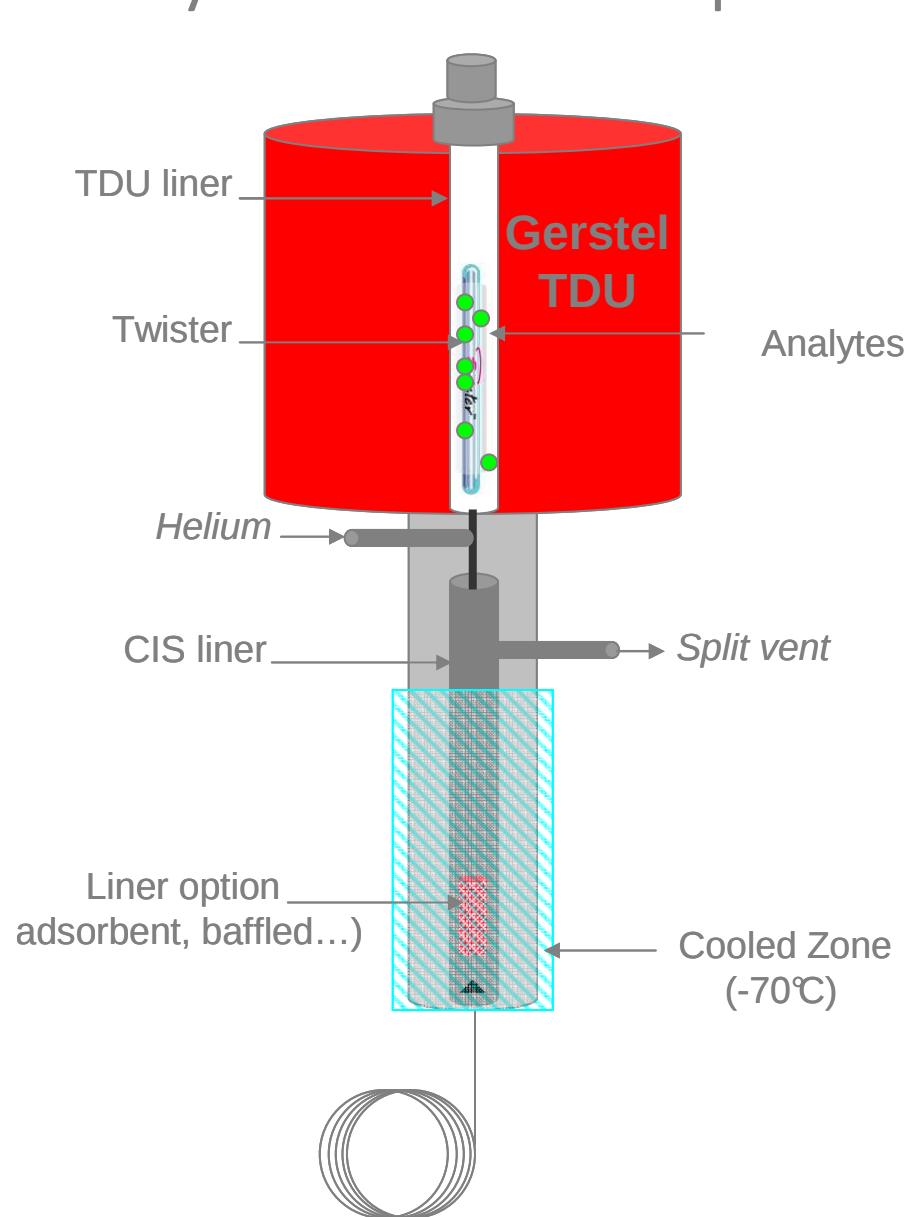
# SBSE- Effluent Pétrolier Sortie de STEP



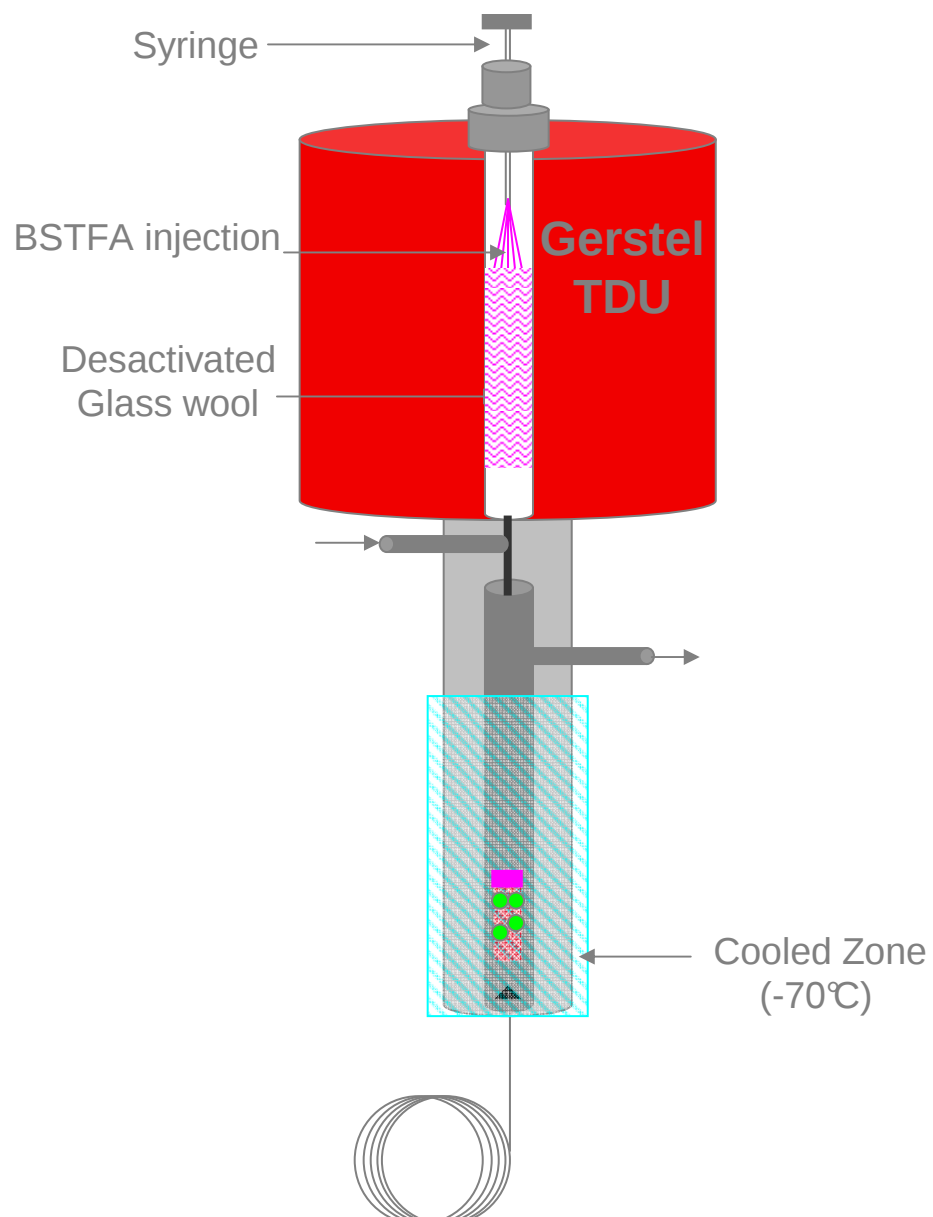
# Ineos – Bio Inlet – Data Processing



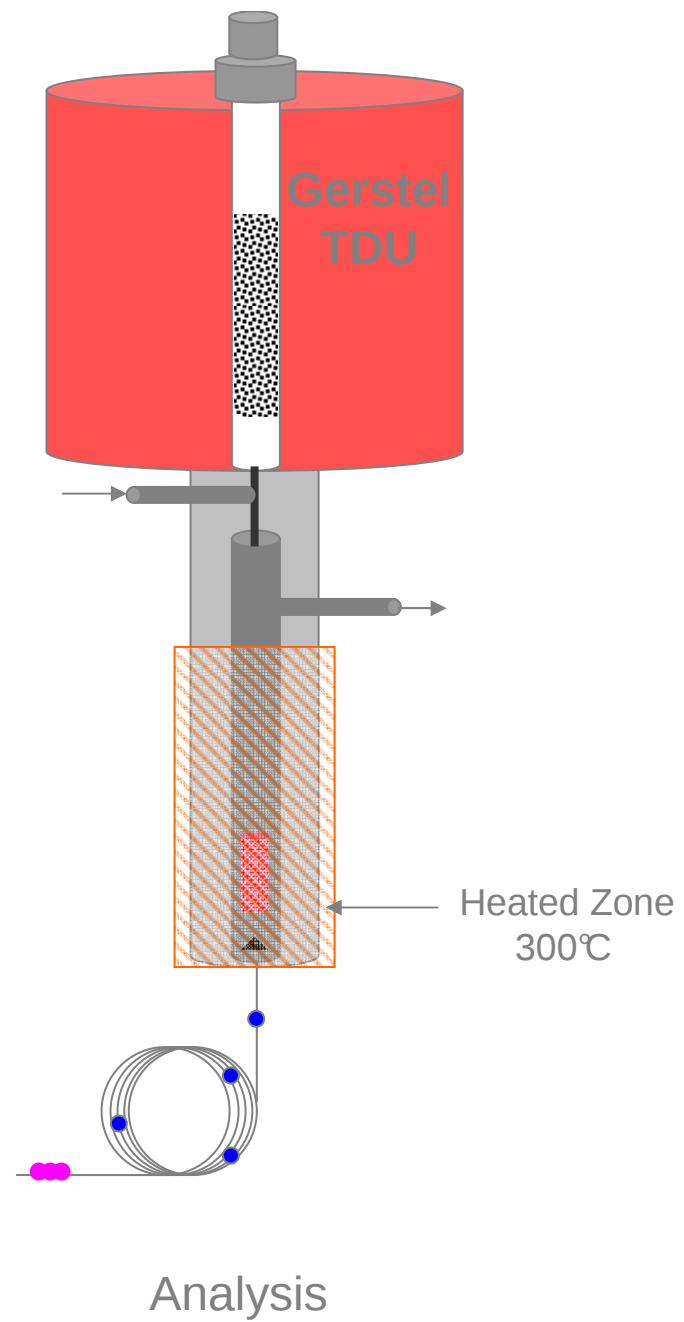
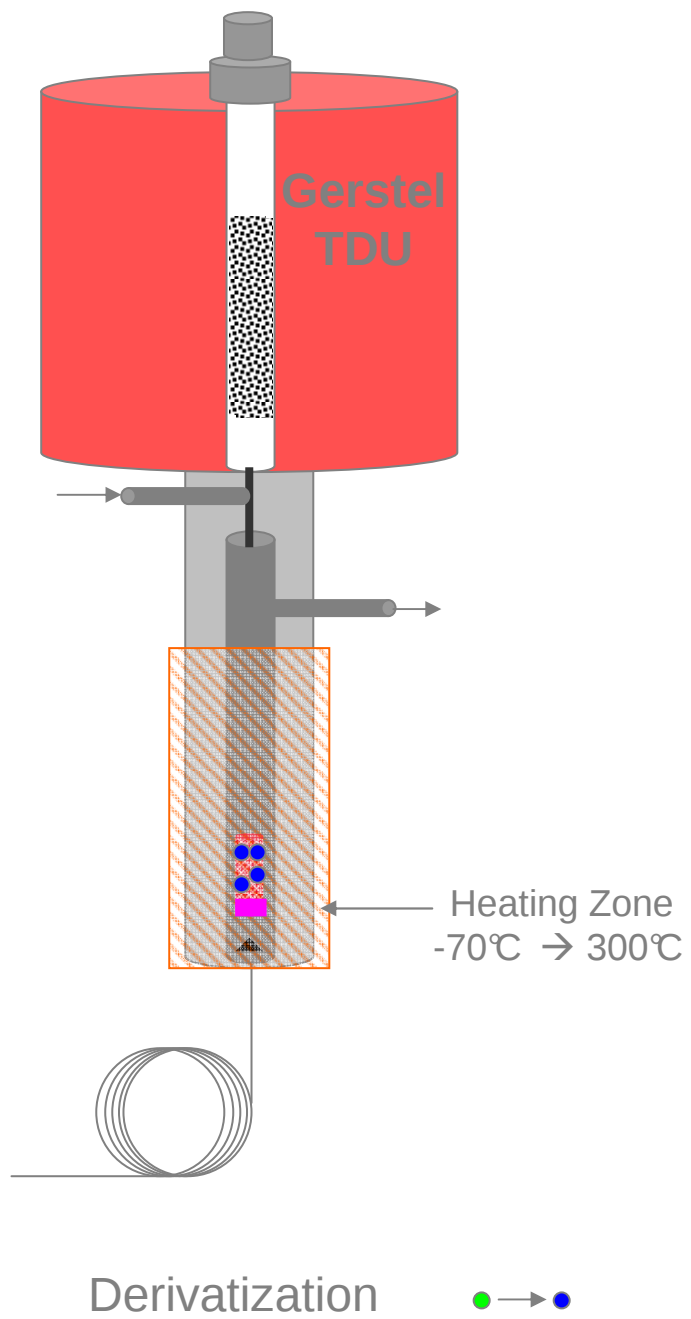
# Analyse des acides Naphteniques: dérivation in-liner



Thermo-desorption & focalization



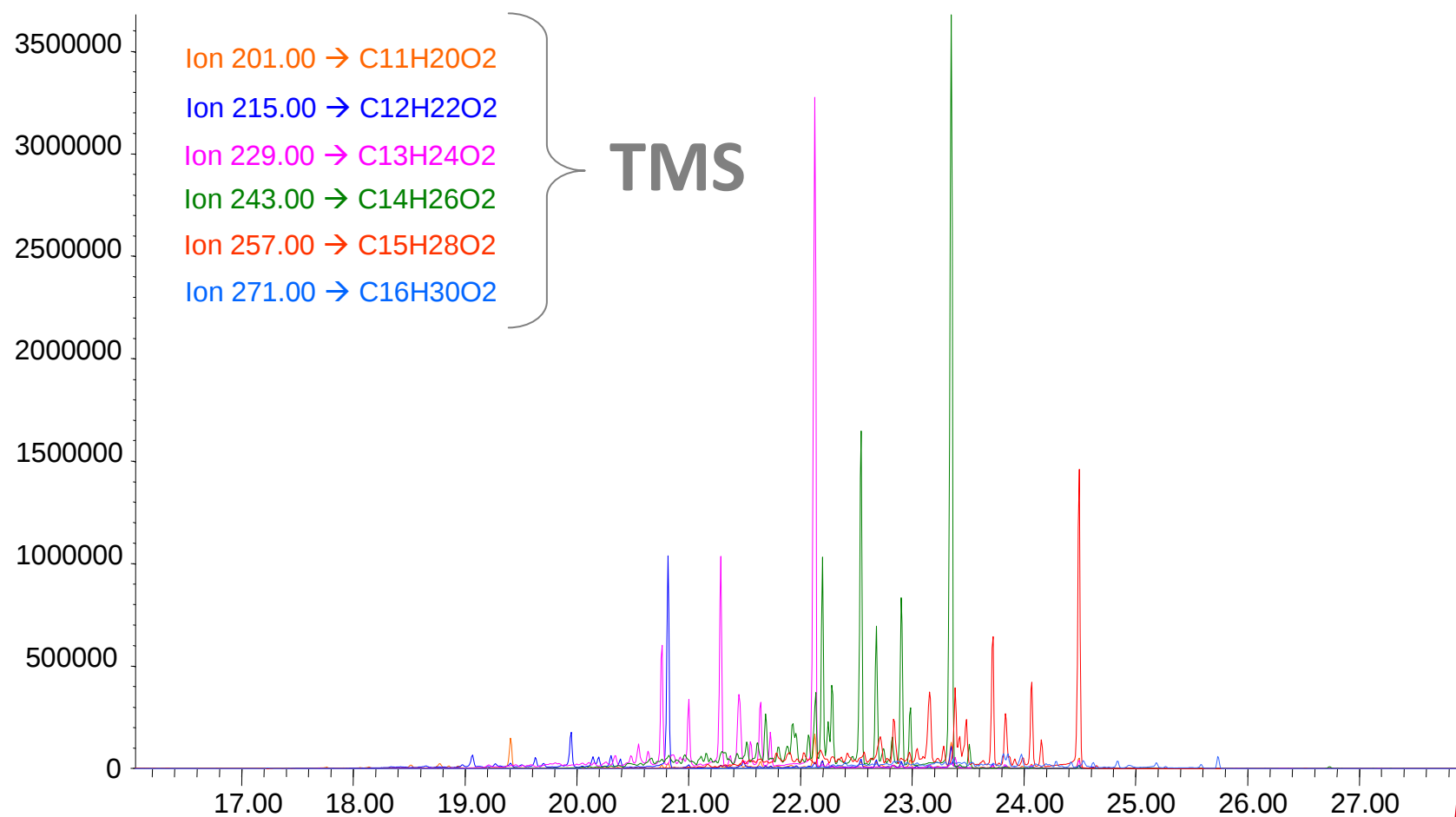
BSTFA injection & focalization



# Acides Naphténiques TMS



Abundance



# *Celles et ceux que je tiens à remercier*



Françoise Acobas  
Véronique Boireau  
Elise Corbi  
Dalel Benali  
Valerie ,Jacquemet  
Christophe Tondelier  
Aurore Choquet  
Stéphanie  
Emilie Manessier  
Pierre Lavaud  
Les Delphines  
Fanny Bodin  
Le chasseur  
Mélanie  
Amélie  
Emmanuel  
Pascal Roche  
Gaella Leroy

Sophie Gaborit  
Dorothée Lenes  
Aurélie Van de Mert  
Laurent Badoil  
Team Paris Sud 2010  
Team Paris Sud 2011  
Kemi Seriki  
Benjamin Maillet  
Faten Belhadj-Kaabi  
Sterenn Gernigon  
David Carteau  
Minh  
Solenn  
Yoann  
Celine  
Bochra  
Vanessa  
Etienne Basset

Sophie Debruyne  
Laetitia Maillet  
Sebastien Casanova  
Stéphanie Guilhermes  
Alexandra  
Emmanuel Roug



thank you for your attention ... Questions?

hvala vam na pažnji ... Pitanja?

ご清聴ありがとうございました...質問ですか？

obrigado pela sua atenção ... Dúvidas?

dank u voor uw aandacht ... Vragen?

أشكركم على اهتمامكم... أسئلة؟

תודה על תשומת הלב שלך ... שאלות?

İlginiz için teşekkür ederim ... Sorular?

grazie per la vostra attenzione ... Domande?

σας ευχαριστώ για την προσοχή σας ... ερωτήσεις;

merci de votre attention...des questions?

謝謝您的關注 ...什麼問題嗎？

dziękuję za uwagę ... Pytania?

Köszönöm a figyelmet ... Kérdések?

Vielen Dank für Ihre Aufmerksamkeit ... Fragen?

tak for Deres opmærksomhed ... Spørgsmål?