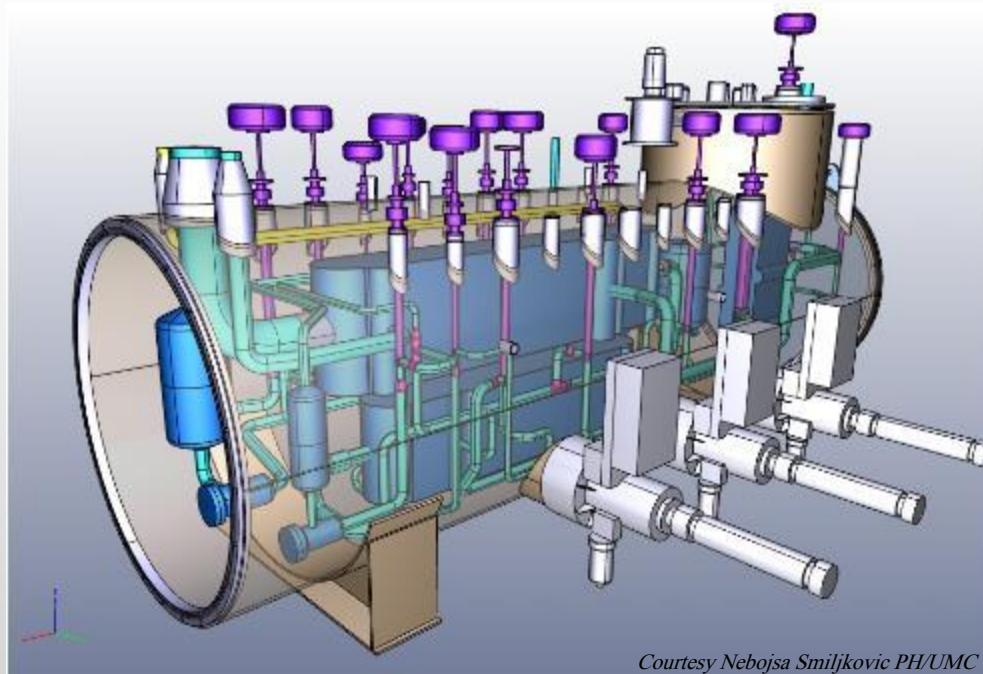


## *Chemistry lab contributions for the diagnostic and cleaning of the CMS cold box contaminated with Breox B35 oil*



*Courtesy Nebojsa Smiljkovic PH/UMC*

CHARVET Colette  
DENIS Maxime (IUT chimie de Besançon)  
TEISSANDIER Benoit

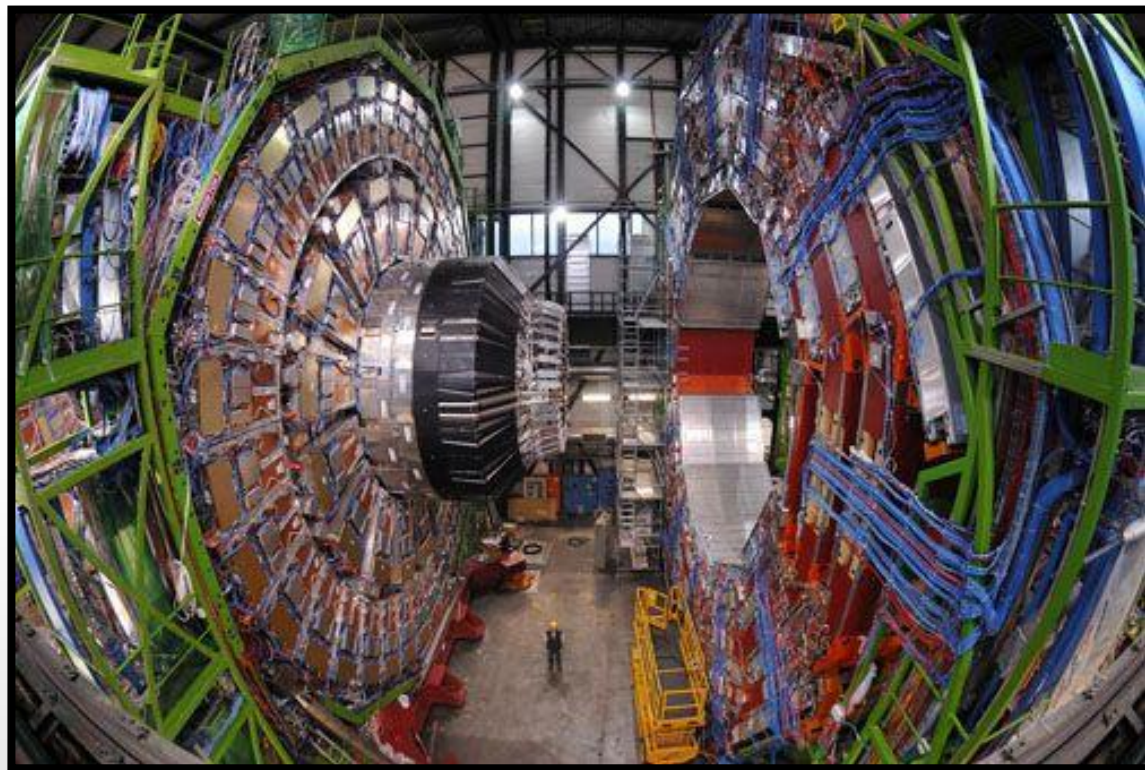
## Introduction – Motivation

## Problem understanding and cleaning strategy

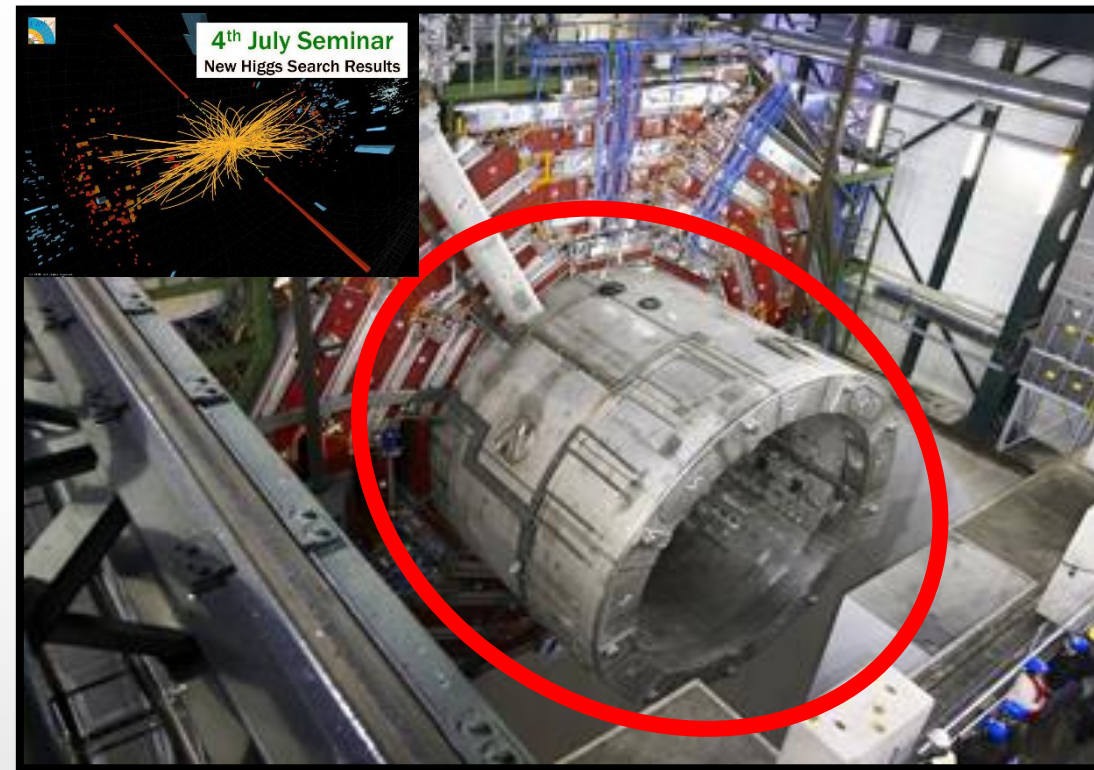
- Analysis for solvent validation
- Development of a cleaning quality control method
- Development of a drying quality control method
- Responsible for quality control during cleaning operation

**Chemistry lab  
contributions**

## Conclusions

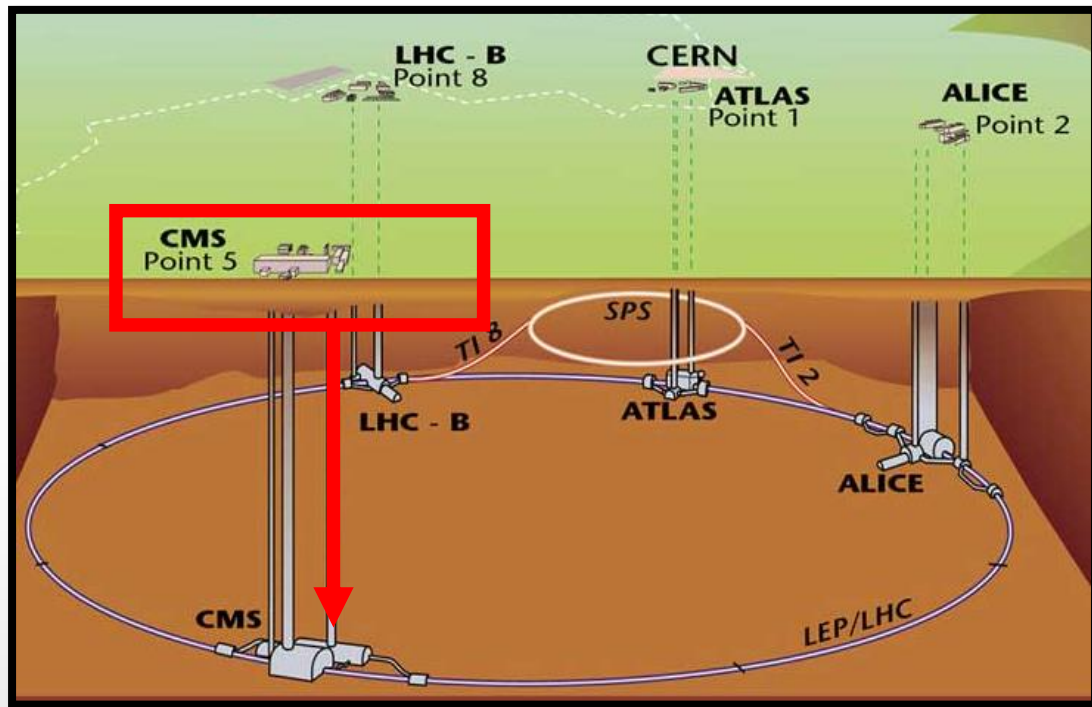


CMS experiment



Superconducting magnet (cold mass : **225 t**)  
Nominal operation at **4.5K** (-268.65 ° C)  
Cooled with liquid helium

## CMS surface cryogenic systems

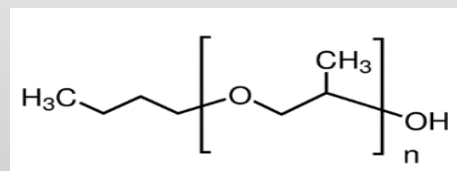


## Helium compressor station (on surface)



### Compression of helium gas

(Adding "Breox B35" lubricant during compression)



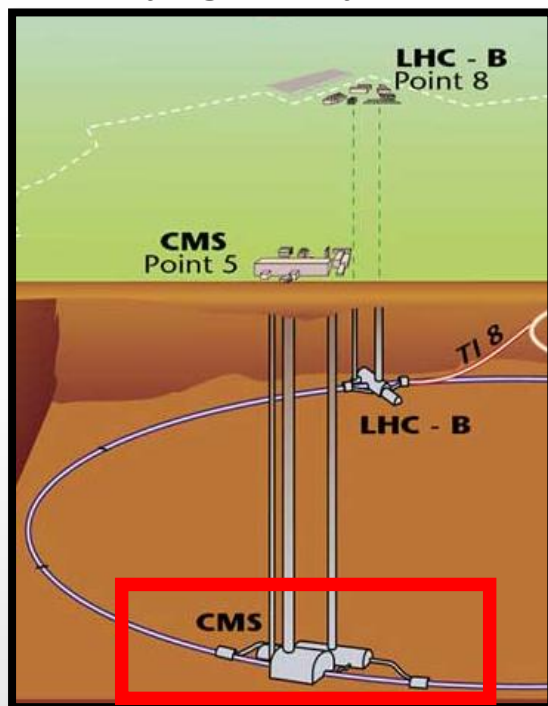
### Separation Breox B35 / helium

Gravity separator, coalescers, activated carbons  
Acceptable Breox concentration in helium lower than 10 ppb

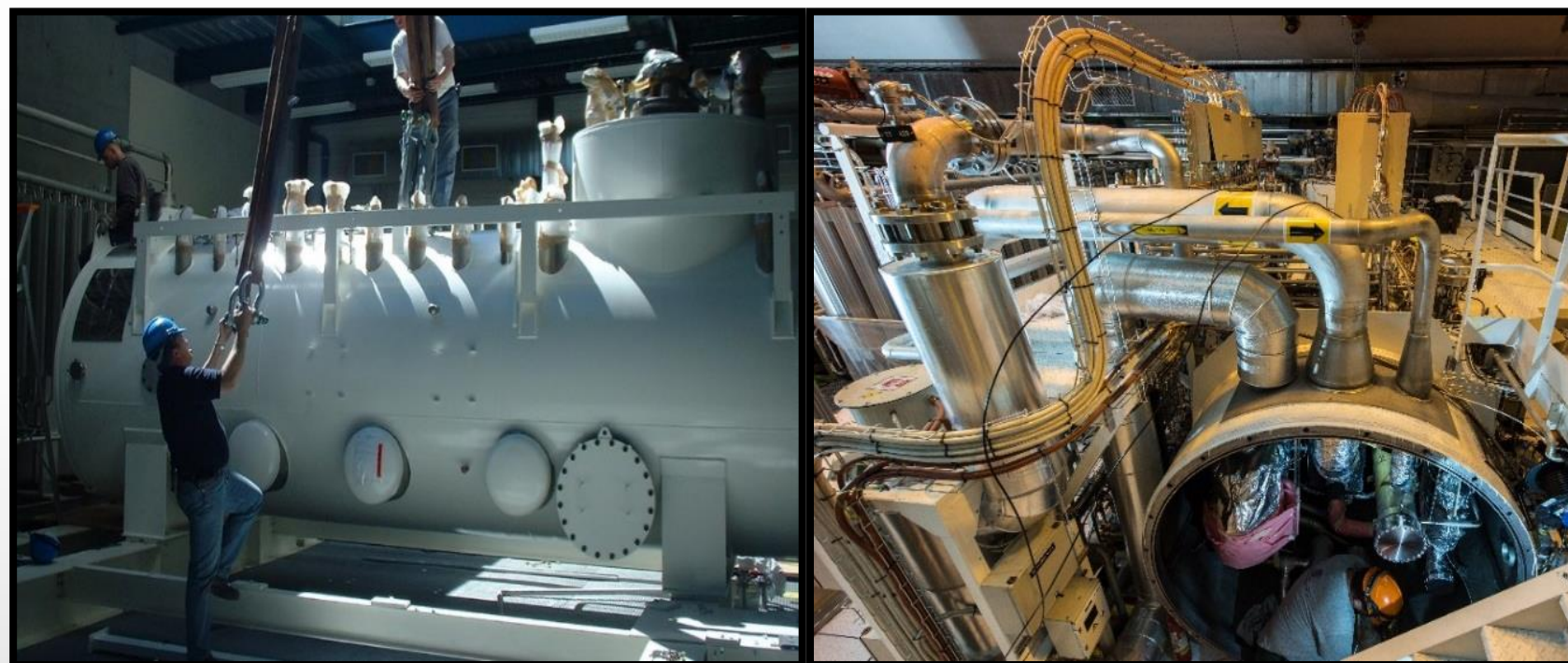


### Helium injection to the underground cold box

## CMS underground cryogenic systems

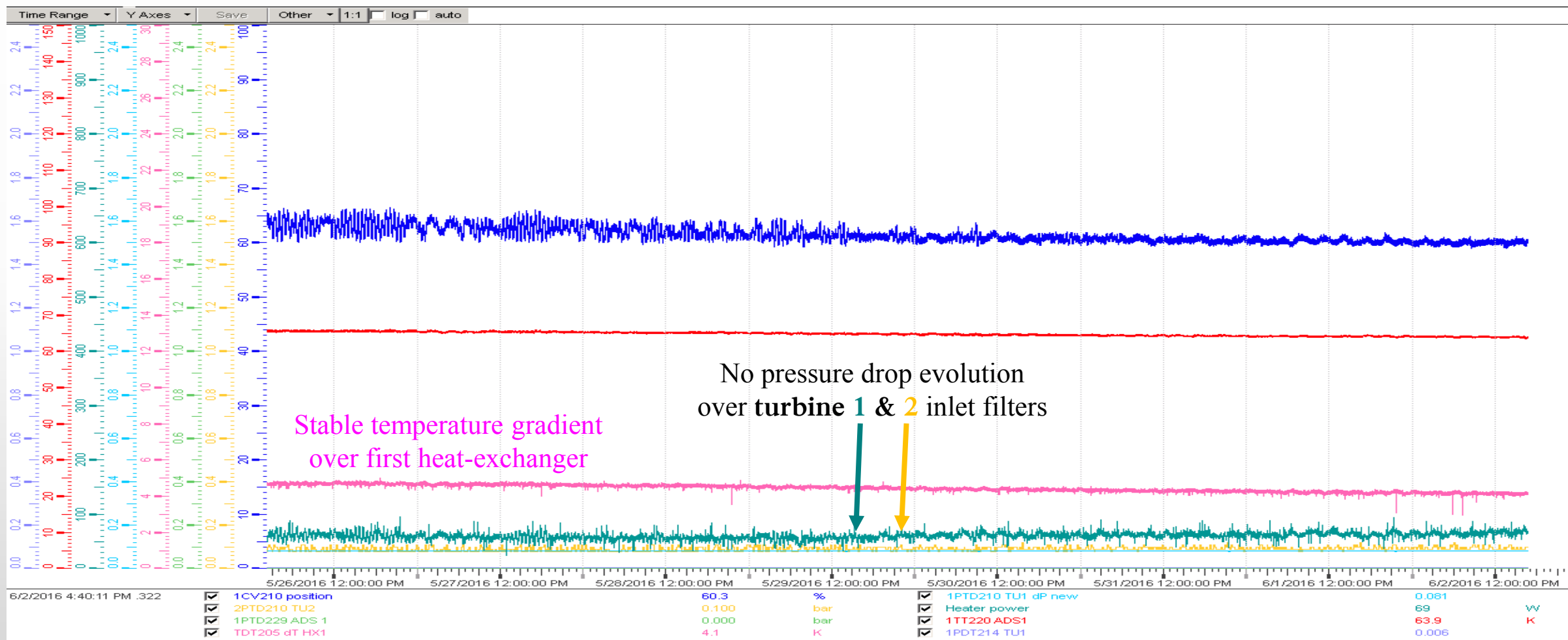


## The CMS Cold-Box

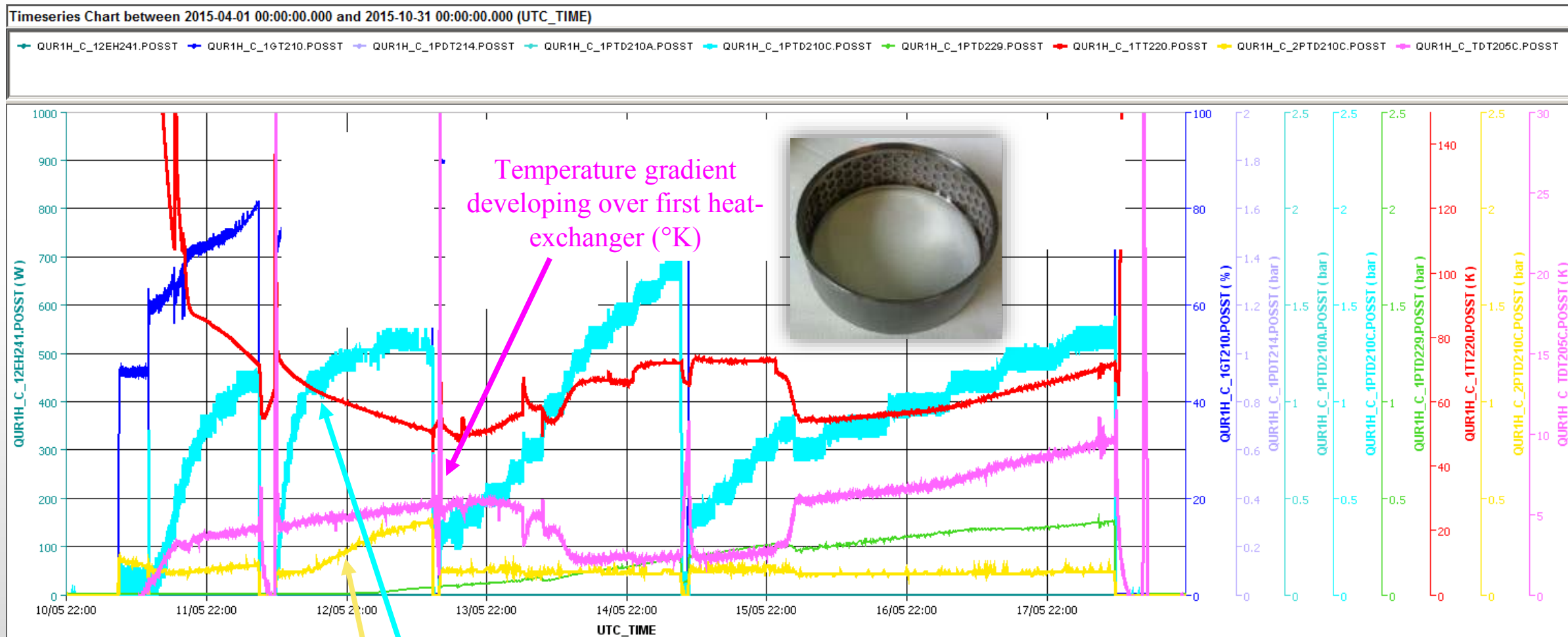


Composed of turbines, heat exchangers, filters, etc.  
Feeding the superconducting magnet with helium 4.5K

## CMS cold box with nominal behaviour



## 2015 CMS cold box run: clogging filters and loss of cold box efficiency



CMS detector **performance is degraded** in absence of the magnetic field provided by its solenoid magnet.  
Thermal cycles can affect the life time of CMS magnet



Need to quickly and clearly **identify the Cold-Box problem**  
**Find a solution** to solve it



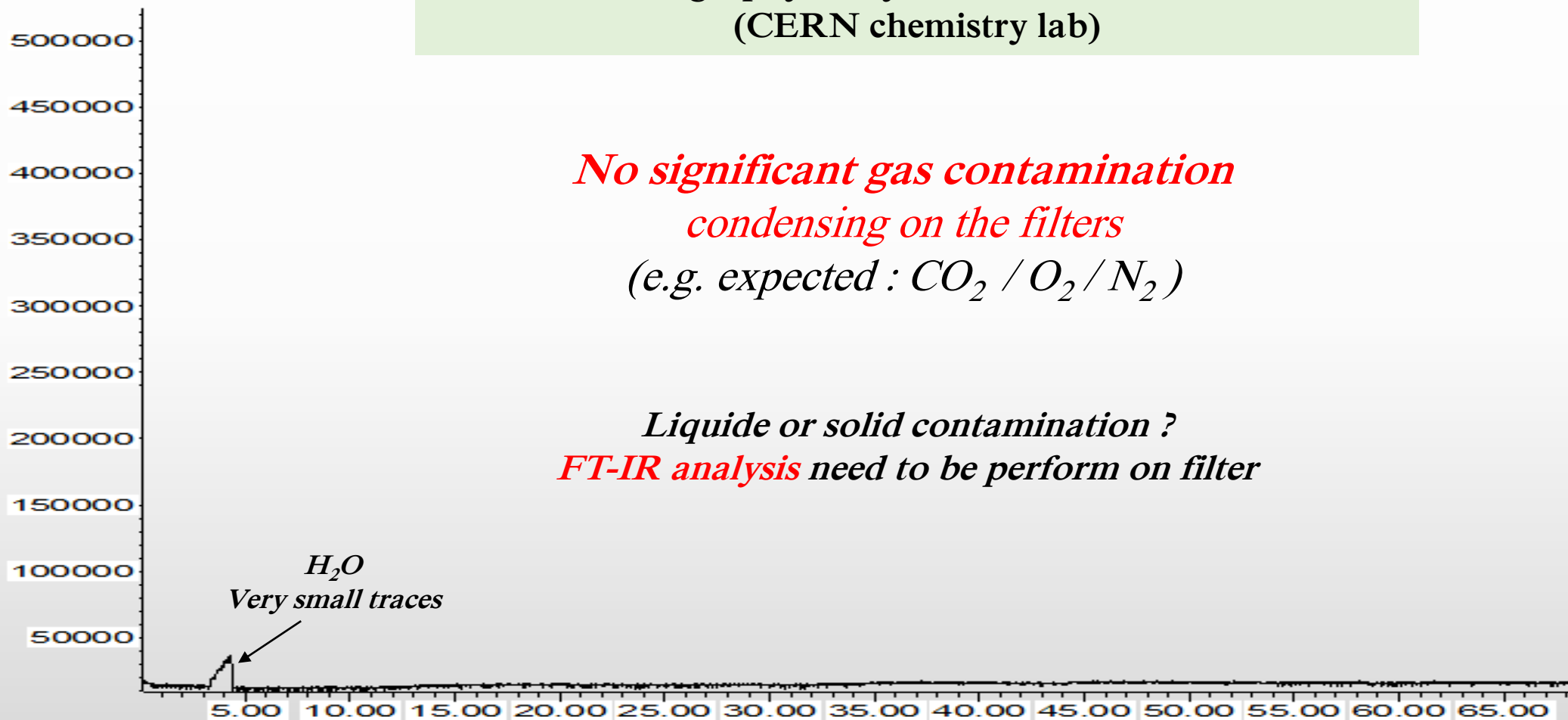
# *Problem understanding - cleaning strategy*

Gas chromatography analysis on helium CMS Cold-Box  
(CERN chemistry lab)

*No significant gas contamination  
condensing on the filters  
(e.g. expected :  $CO_2$  /  $O_2$  /  $N_2$ )*

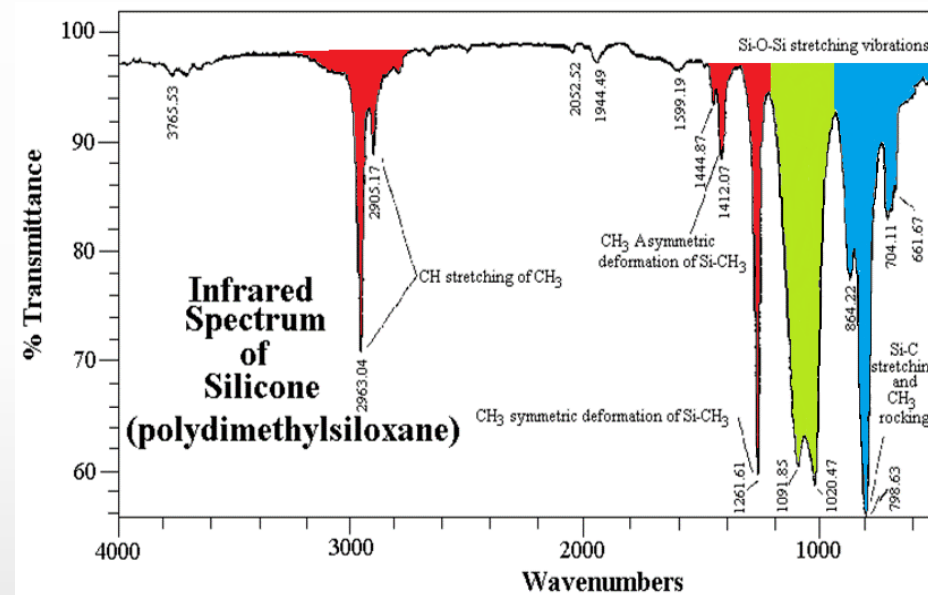
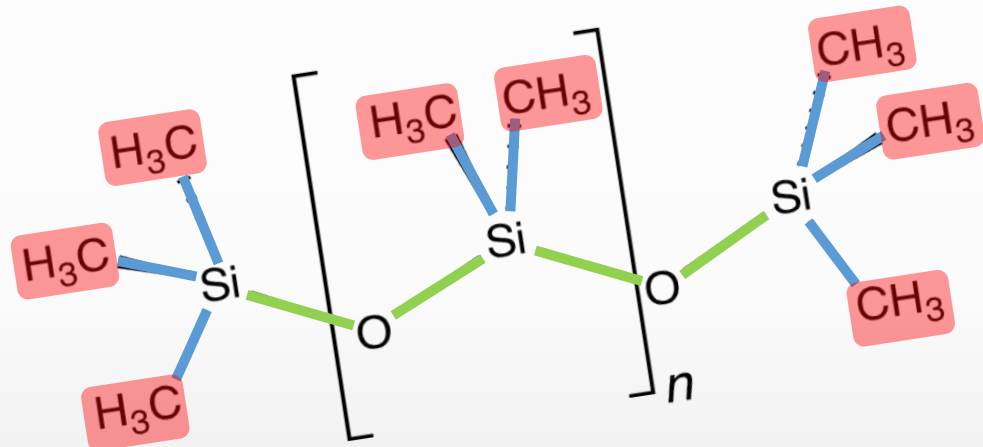
*Liquide or solid contamination ?  
FT-IR analysis need to be perform on filter*

Abundance

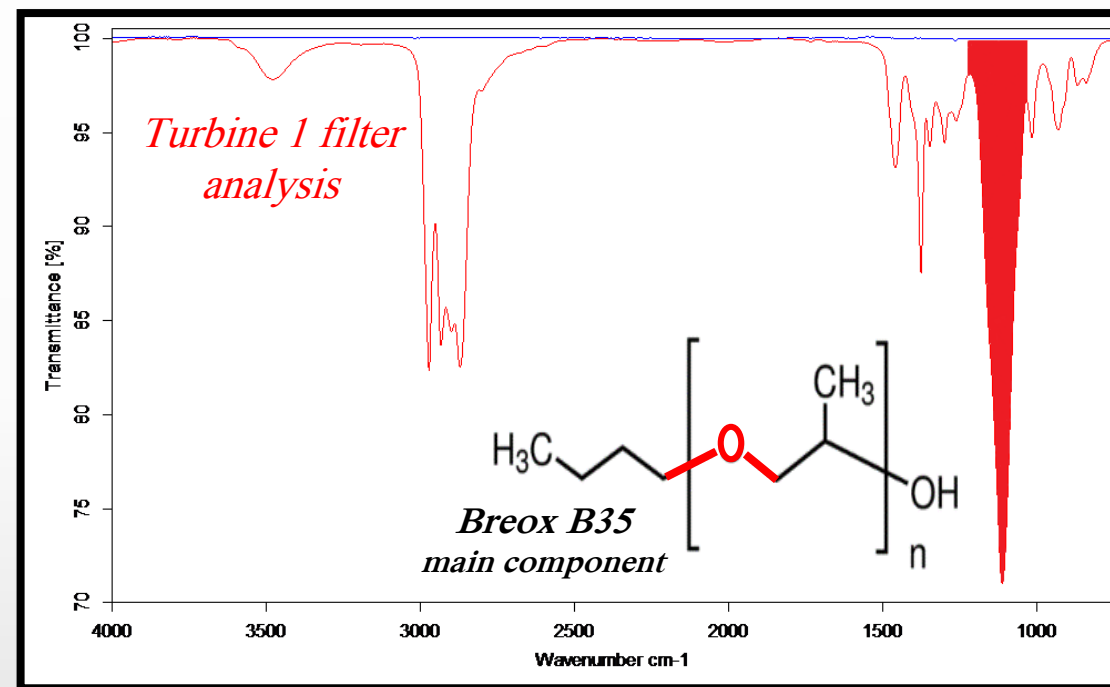


Time →

## Basic FT-IR spectroscopy analysis

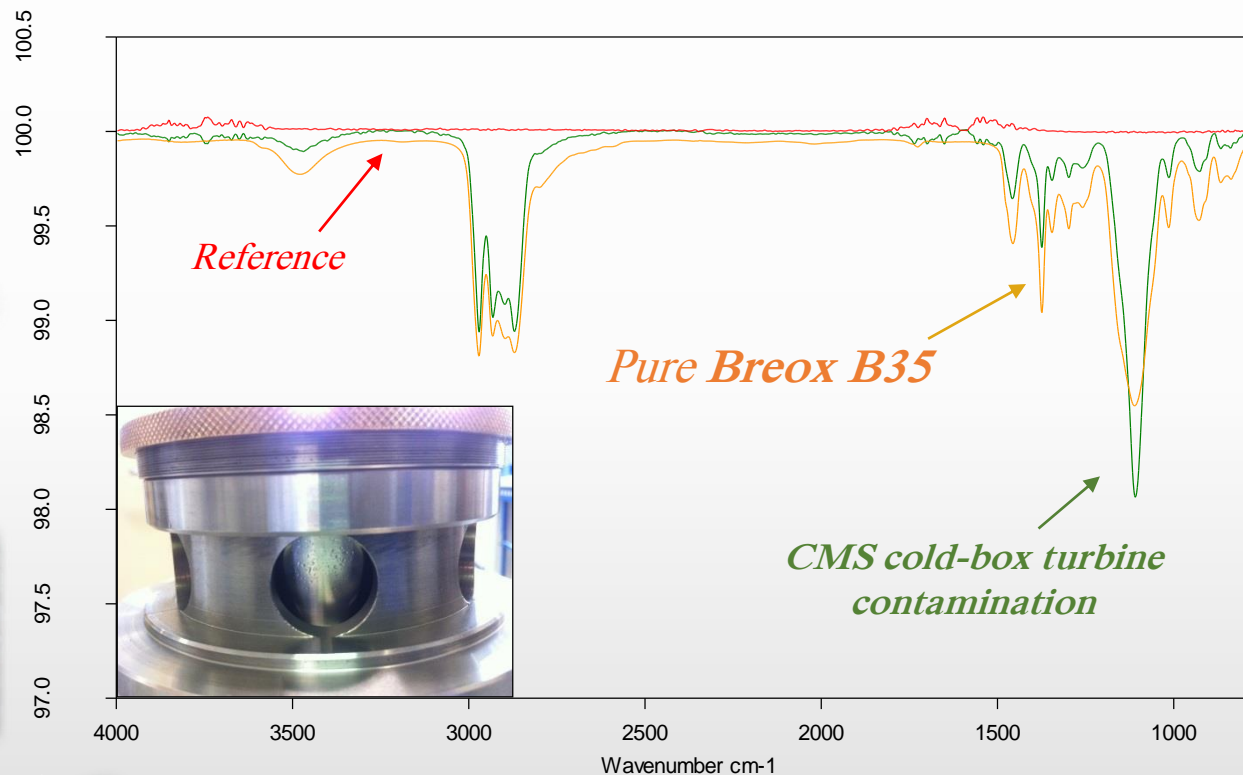


*FT-IR analysis on turbine filter 1: identification of the responsible for the pressure drop*



***100 mg** of Breox B35 on filter generate the cold-box perturbation*

*New specific analysis methods were developed  
Around **50** complete analyses ([EDMS reports](#))*



Contamination was clearly identified  
**everywhere Breox B35 oil**

First meeting : 19<sup>th</sup> august 2015

Clean the cold-box on site underground by solvent circulation during YETS (in January 2016)

Task force : CMS - TE

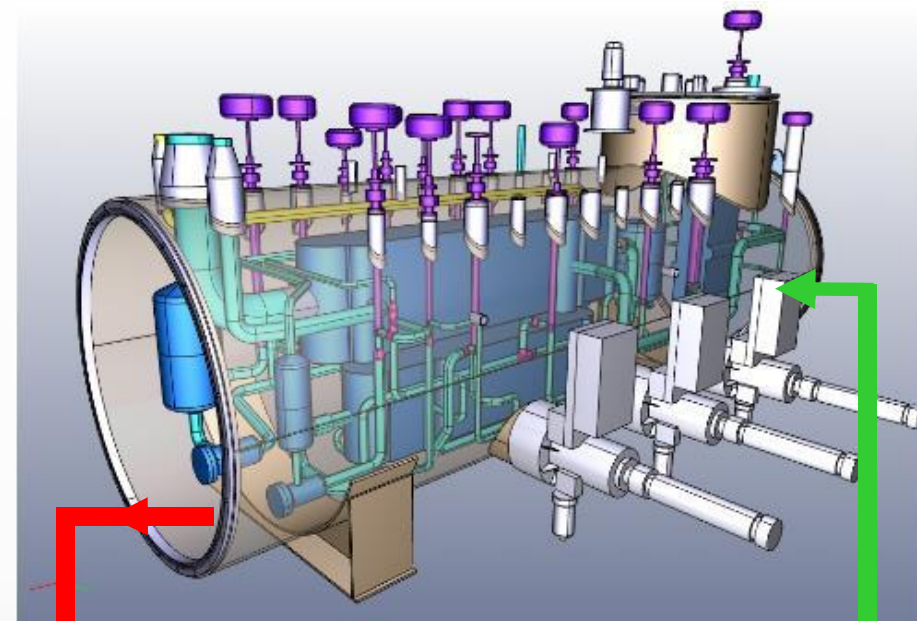
TE-VSC-SCC mandate :

Leonel Ferreira :

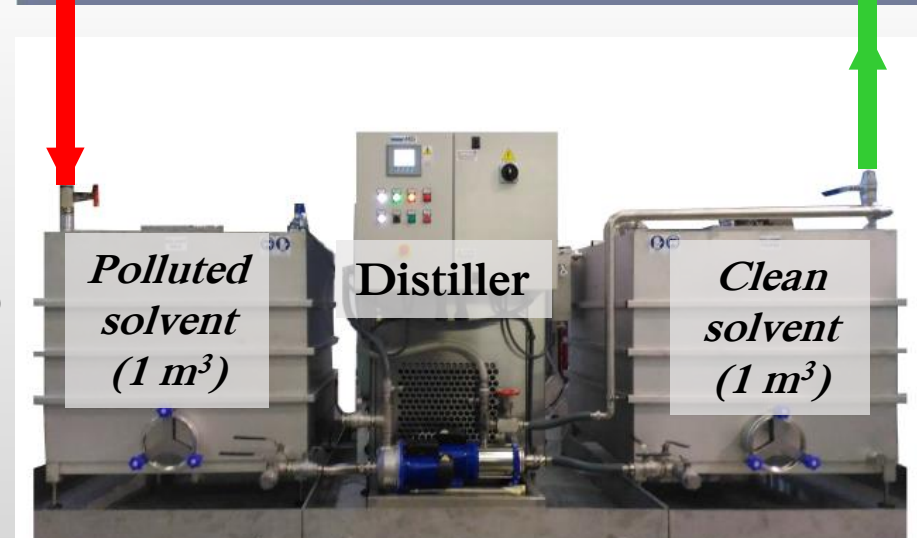
- Design, select and buy the **cleaning machine**
- Select and validate an **adapted solvent** (efficient, safe, compatible)
- Responsible for **cleaning machine operation**

Chemistry lab :

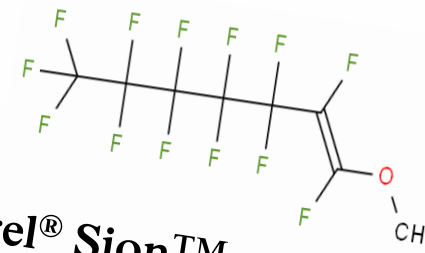
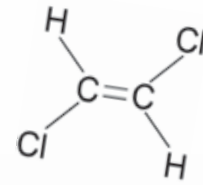
- Analysis for **solvent validation**
- Develop a **cleaning and drying quality control methods**
- **Responsible for quality control** during cleaning operation



Cleaning machine



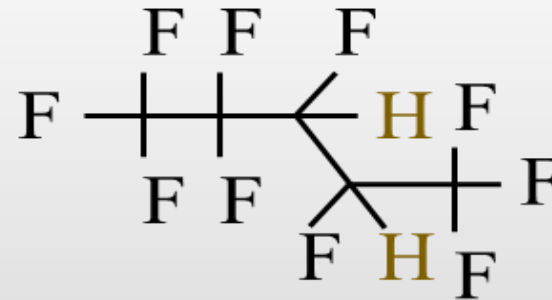
Promosolv™ 71PA



Vertrel® Sion™

## *Analysis for solvent validation*

1,1,1,2,2,3,4,5,5,5-Decafluoropentane



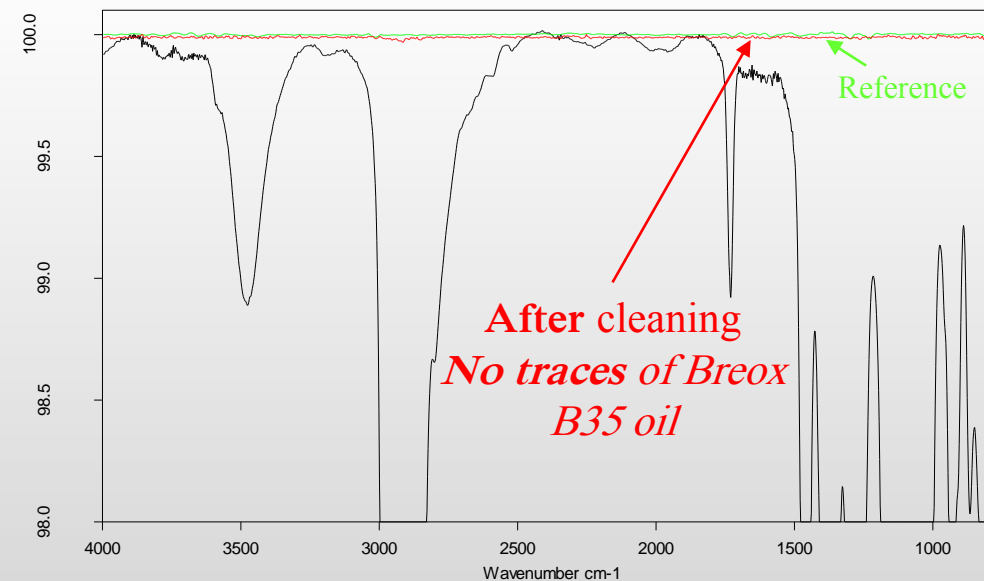
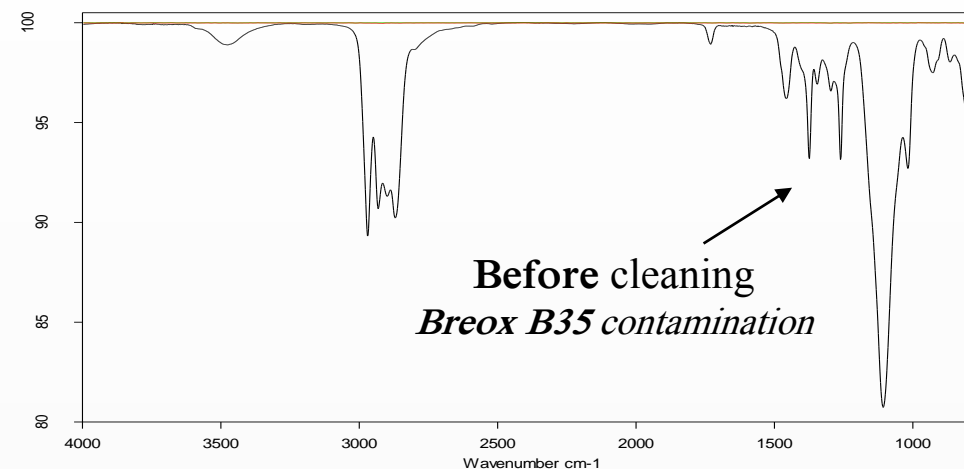
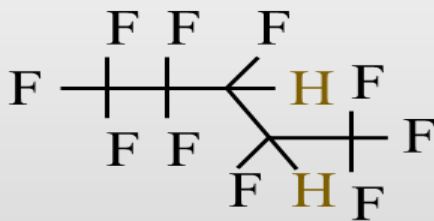
Vertrel™ XF



## Cleaning efficiency tests of **Vertrel XF** against Breox contamination

- Stainless steel samples were **contaminated** with **Breox oil**  
Test procedure (EDMS [997350](#))
- Samples were **cleaned** with solvent
- **FT-IR analysis** was performed to evaluate the solvent **efficiency**  
Results and report (EDMS [997350](#))

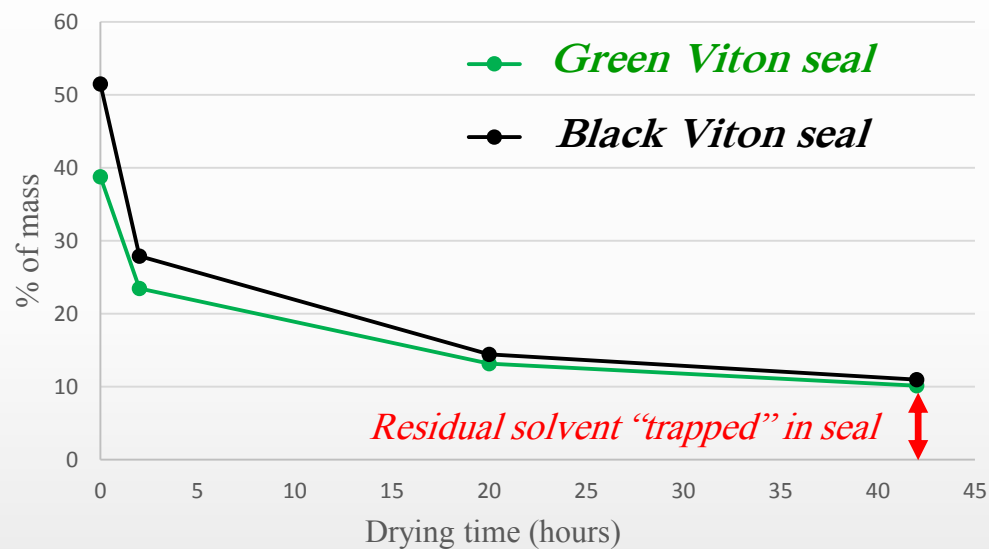
**Vertrel™ XF is efficient**





## Examples of material compatibility tests

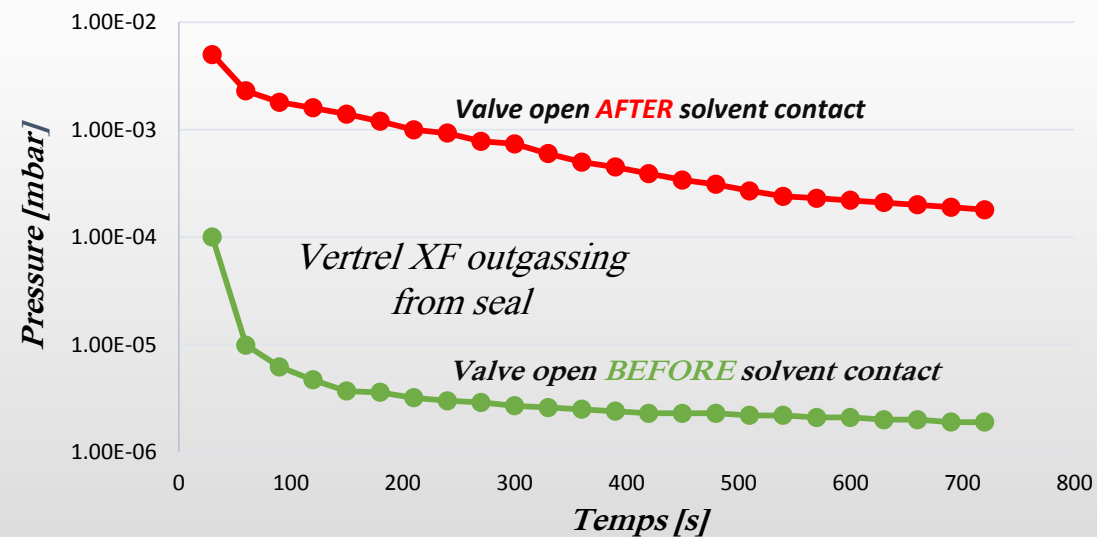
Drying speed (in air) of Viton seals after contact with Vertrel XF for 24h



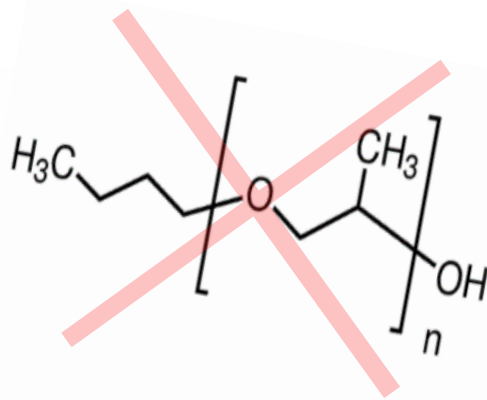
**All Viton seals need to be replaced after cleaning**



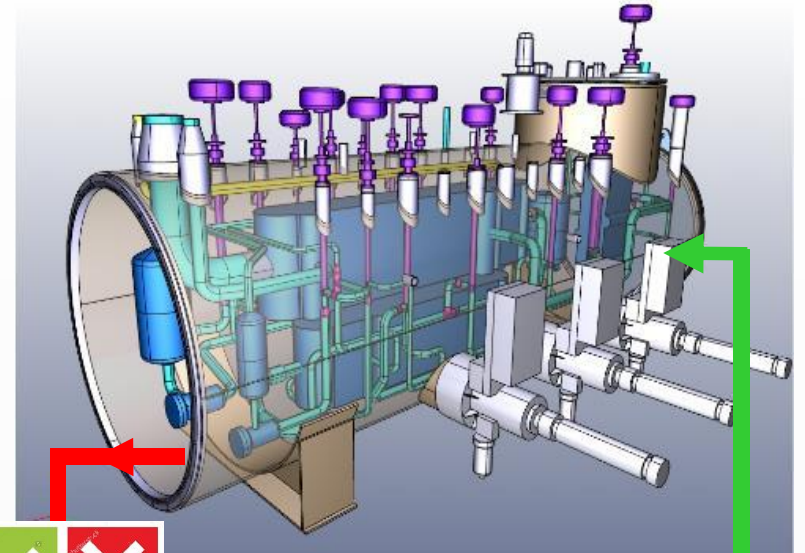
Pumpdown of valve placed in contact with Vertrel XF for 24h



Antonio Mongelluzzo (TE-VSC)



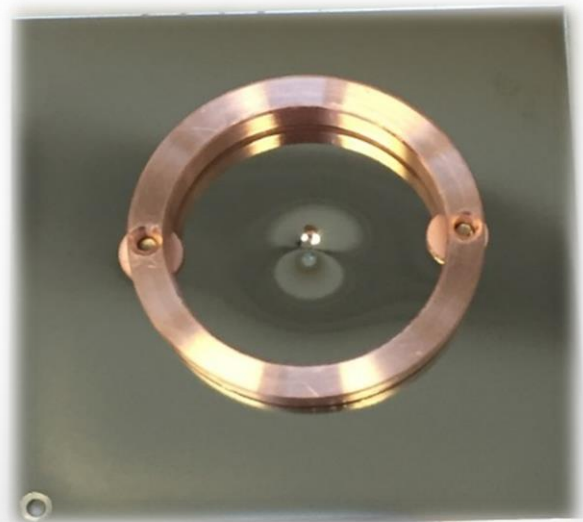
*Develop a cleaning quality control method (online)*



[Breox] < 10 mg.l<sup>-1</sup>



## FT-IR In-Situ measurement of Breox B35 in Vertrel XF

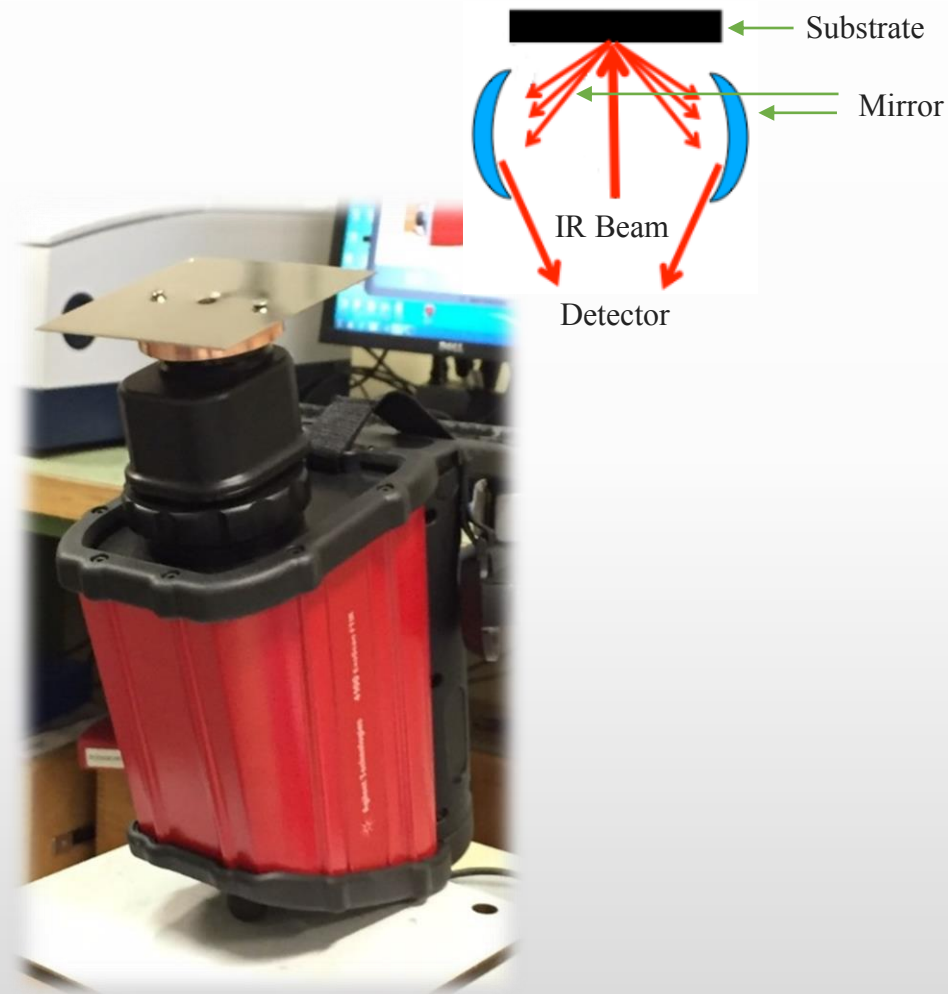


**20  $\mu$ l**  
Breox B35 + Solvent

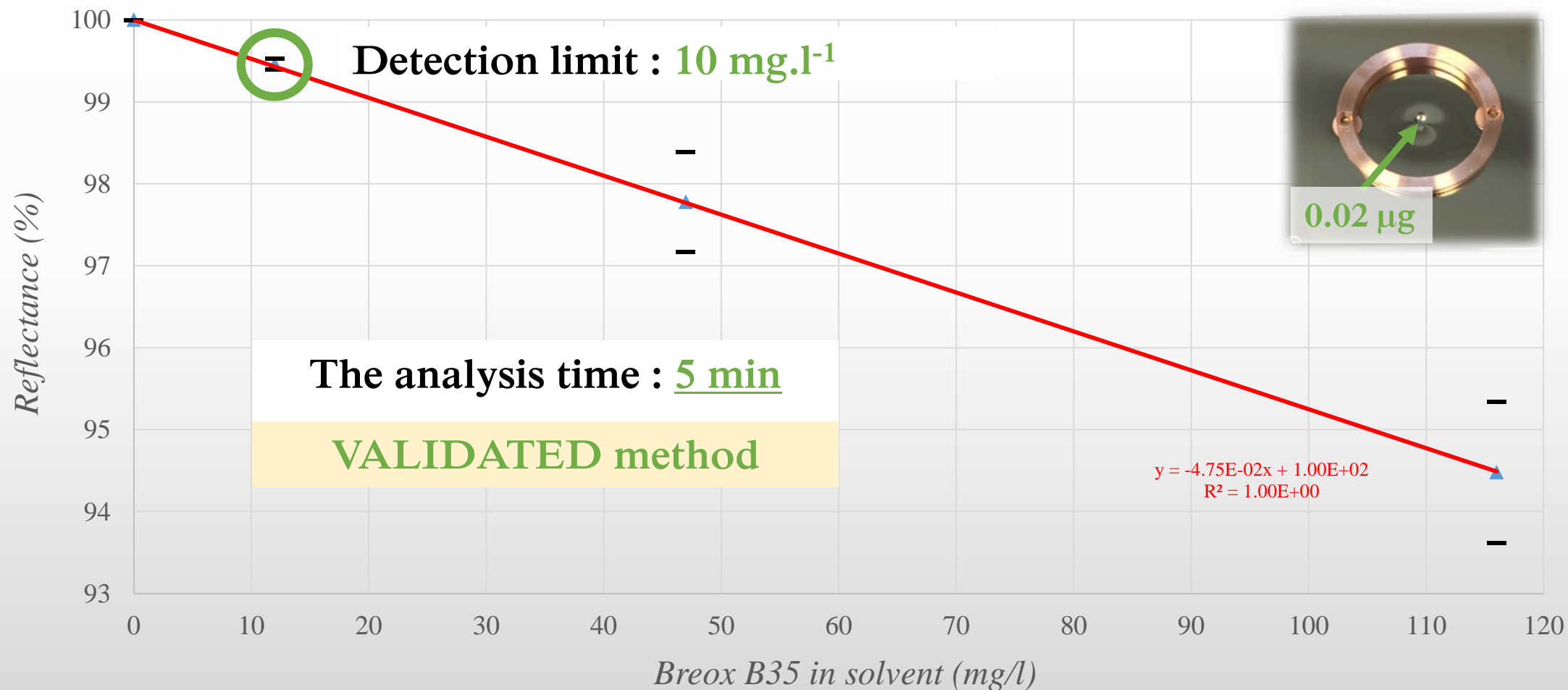


Solvent  
Evaporation

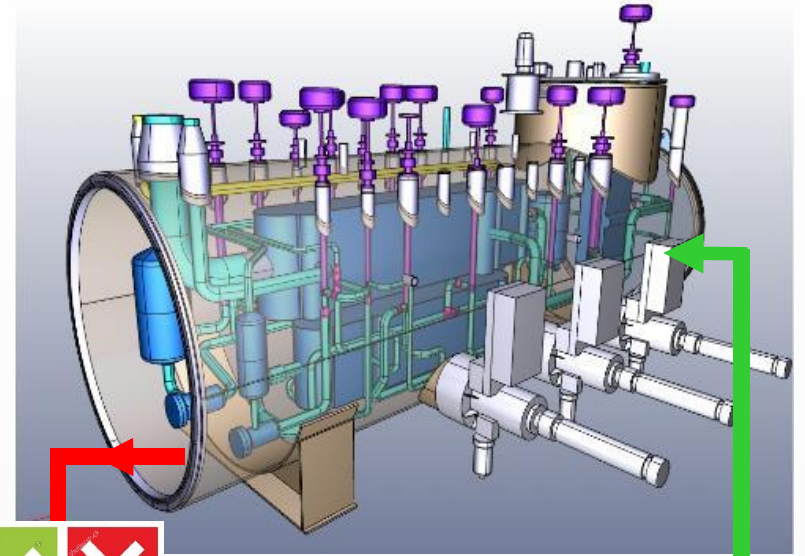
**Breox B35**



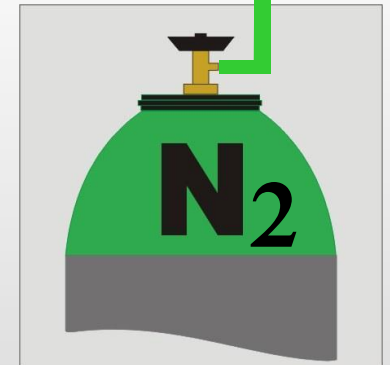
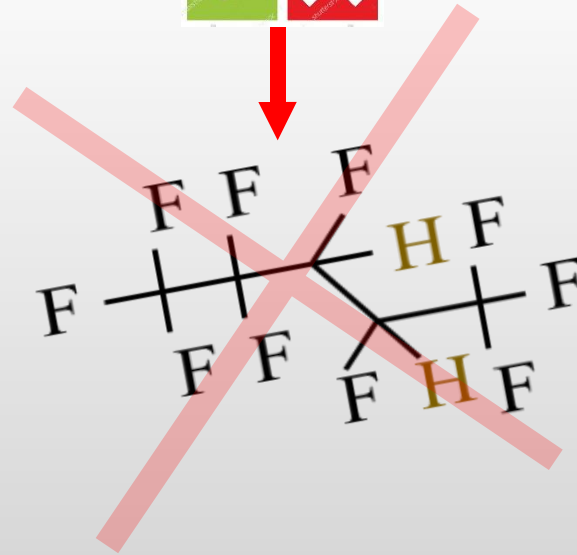
## FT-IR Calibration curve of Breox B35 in Vertrel XF



*Develop drying quality control method (After cleaning)*

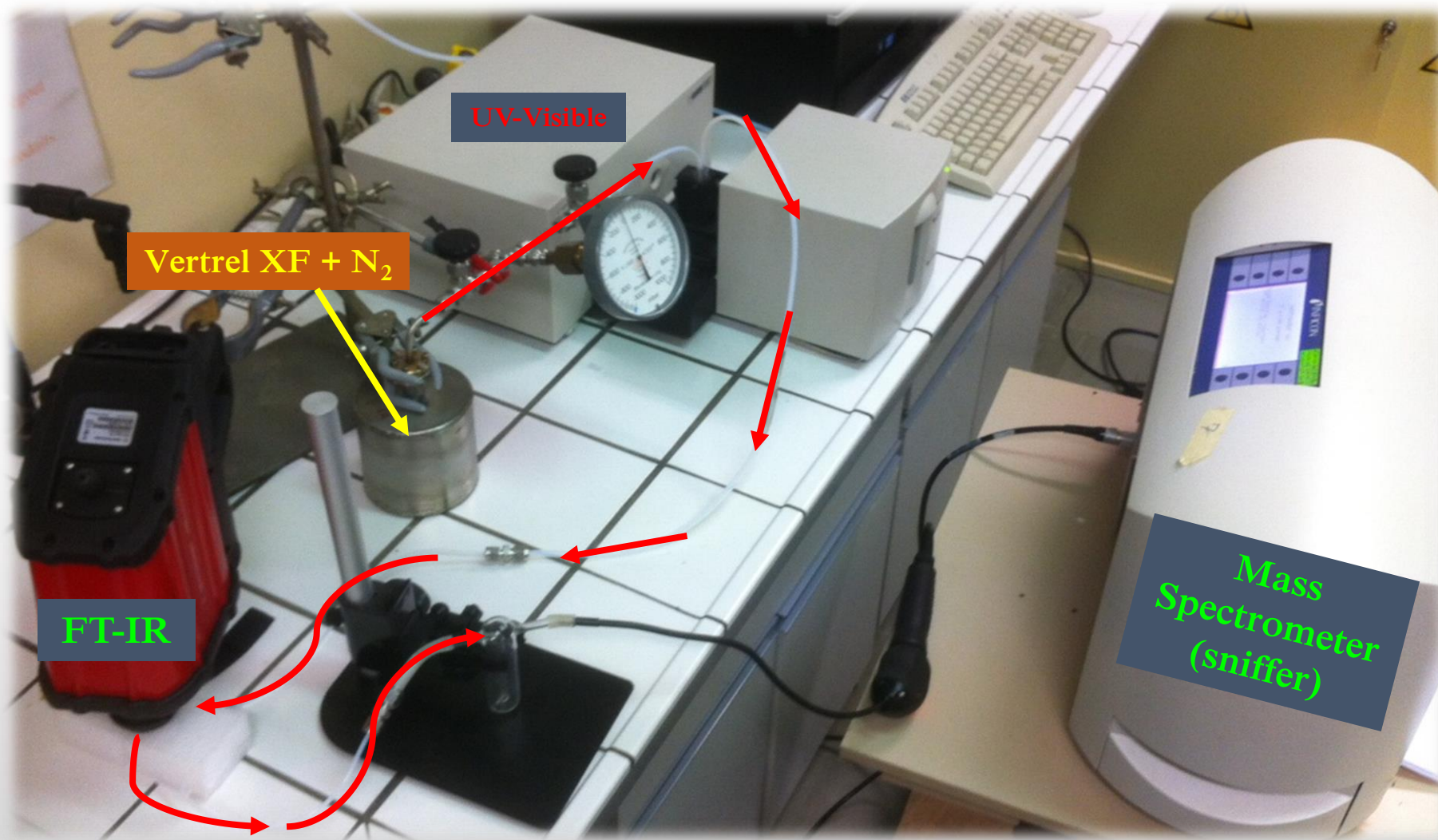


Vertrel XF < 10 ppm (v/v)



# Develop a drying quality control method

Lab setup to measure traces of different solvents in nitrogen: 3 methods

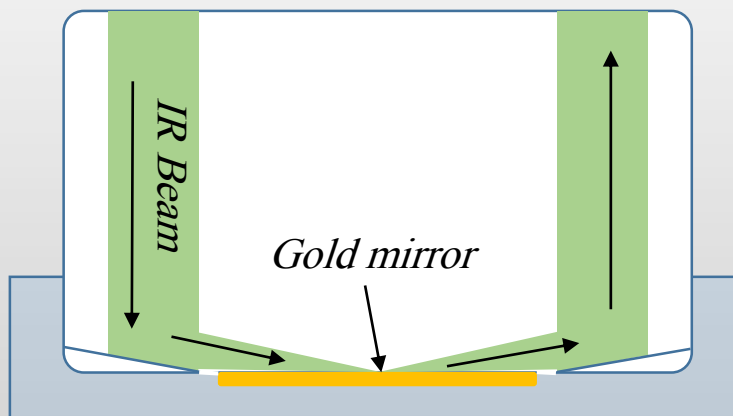
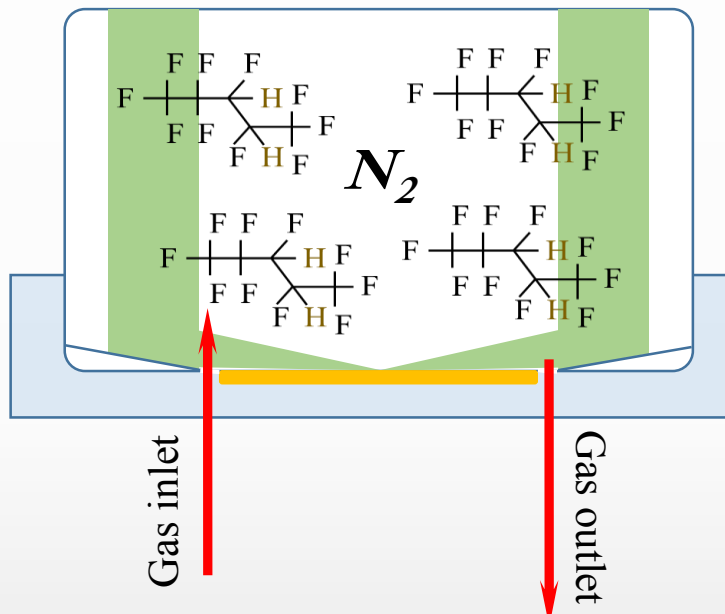
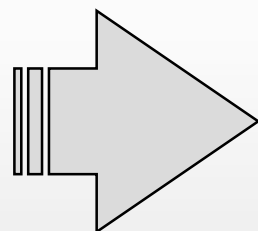


## FT-IR accessory modification to measure solvent vapour traces in nitrogen

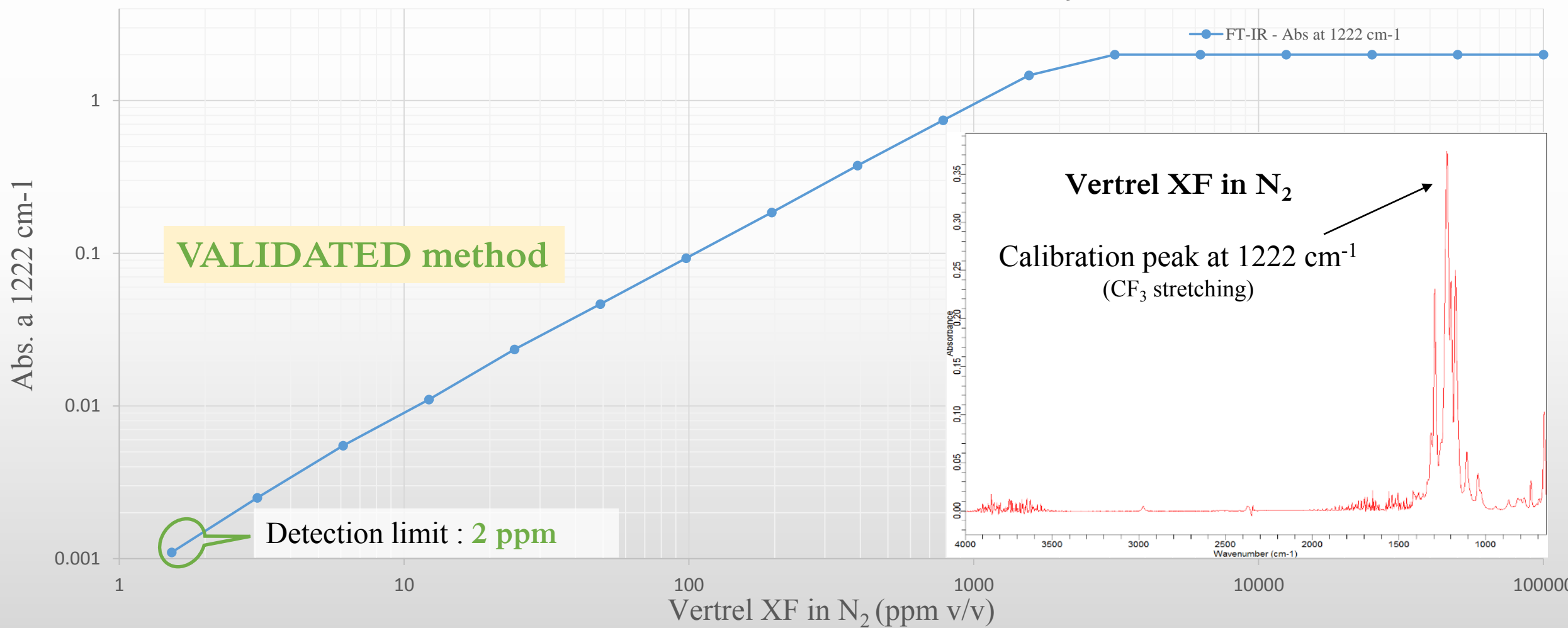
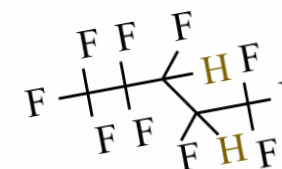


Handling FT-IR

Grazing angle module  
(82°) with gold mirror

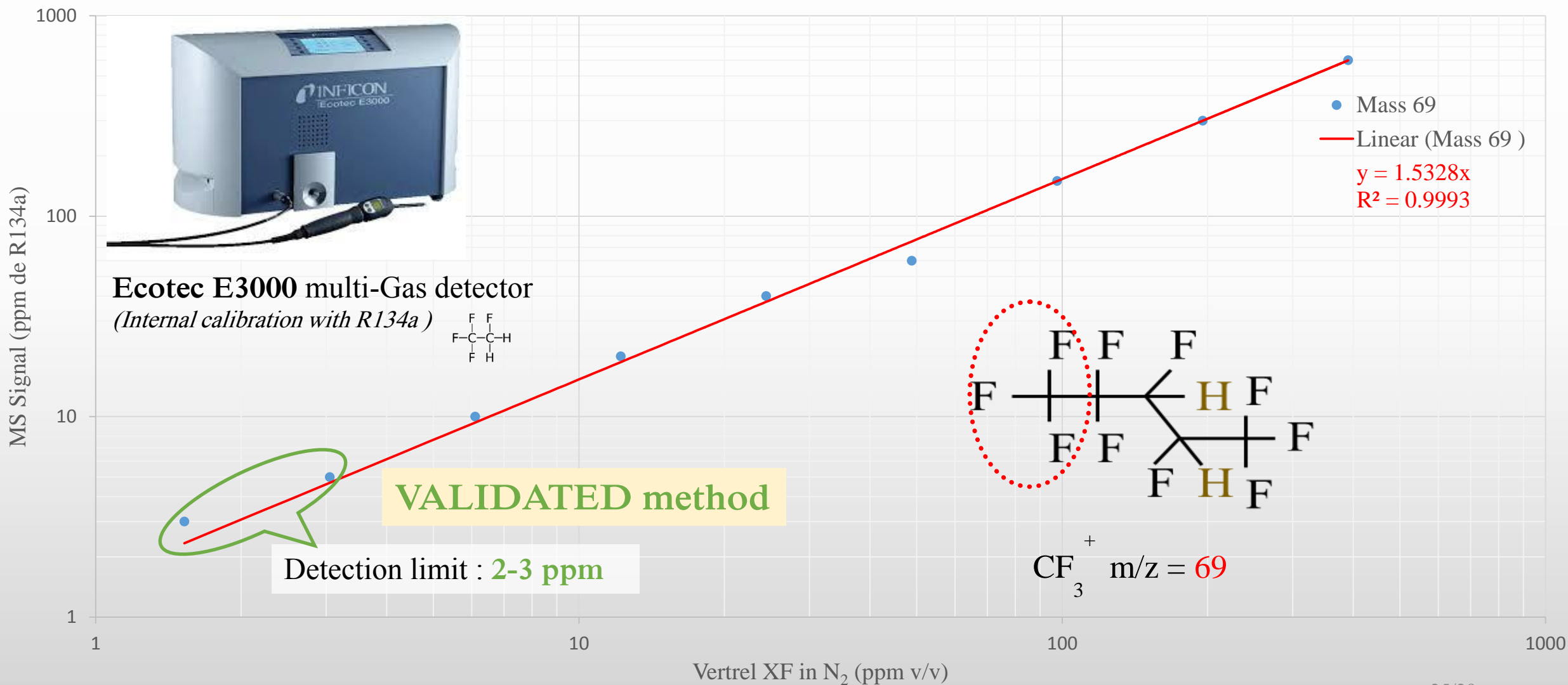


## FT-IR calibration curve: Vertrel XF in nitrogen





## Sniffer (Mass spectrometer) calibration curve: Vertrel XF in nitrogen



*Cleaning and drying operations  
in CMS cavern*



Chamonix 25 Jan 2016 AB

← Austin Ball (CMS Technical Coordinator)

## Moment of truth: is there Breox in the solvent?

This morning: 10am

Taking samples from full circuit



Preparing IR analysis



Awaiting result.....



Bingo! strong Breox signature....

Looks like we are on the right track



Team work during **1 month**

**4 circuits** were cleaned by solvent circulation

~ **400 g of Breox** oil were removed

Equivalent to **4000 clogged turbines filters**

More than **100 in-situ analyses** were performed by chemistry lab team

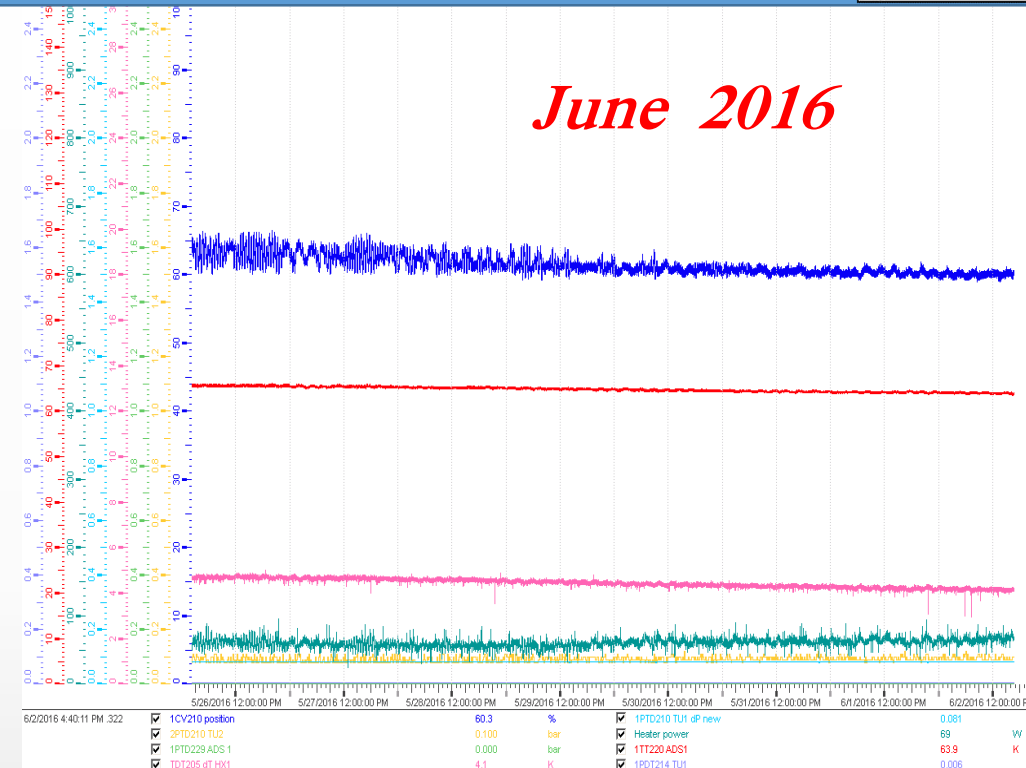
**Less than 10 ppm** (v/v) of residual vapour solvent in the cold-box left after drying

June 2016

*CMS cold-box works very well !!*

*Successful project for CMS, CERN, TE department*

*Very nice team work !!*



On going at the chemistry lab :

*- Breox B35 content analysis in activated carbon (filters) is developed*

*- Breox B35 analysis by Gas Chromatography (GC/MSD) will be developed*

*If needed, Dream team of the Chemistry lab is ready to repeat it somewhere else at CERN*



*Thanks to the  
Cleaner's*

*C. Charvet - TE/VSC  
C. Fabre - TE/CRG  
D. Majournal - TE/CRG  
J. Bremer - TE/CRG  
J. Gremion - TE/VSC  
L. Bardo - EP/ADO  
L. Ferreira - TE/VSC  
M. Taborelli - TE/VSC  
P. Maurin - TE/VSC  
R. Consentino - TE/CRG*

*Thanks for your  
attention !*

