Methane Test - Week 1

Summary of the week

- Cartridge’s regeneration
- Methane Calibration with Gas Chromatograph
- Set up
- Methane Analysis

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Methane Test - Day 11.12.2023

Cartridge's regeneration

4 types of **molecular sieves** to be tested : materials with **different power of absorption**

Z3

Z4

Z5

Z10

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Cartridge's regeneration set up

Regeneration : **remove** the **gas** trapped in the **molecular sieve**

- Cartridges installed in the set up
- Flushed with Argon
- Heated up to 250°C
- Pressure set at 1.3 bar

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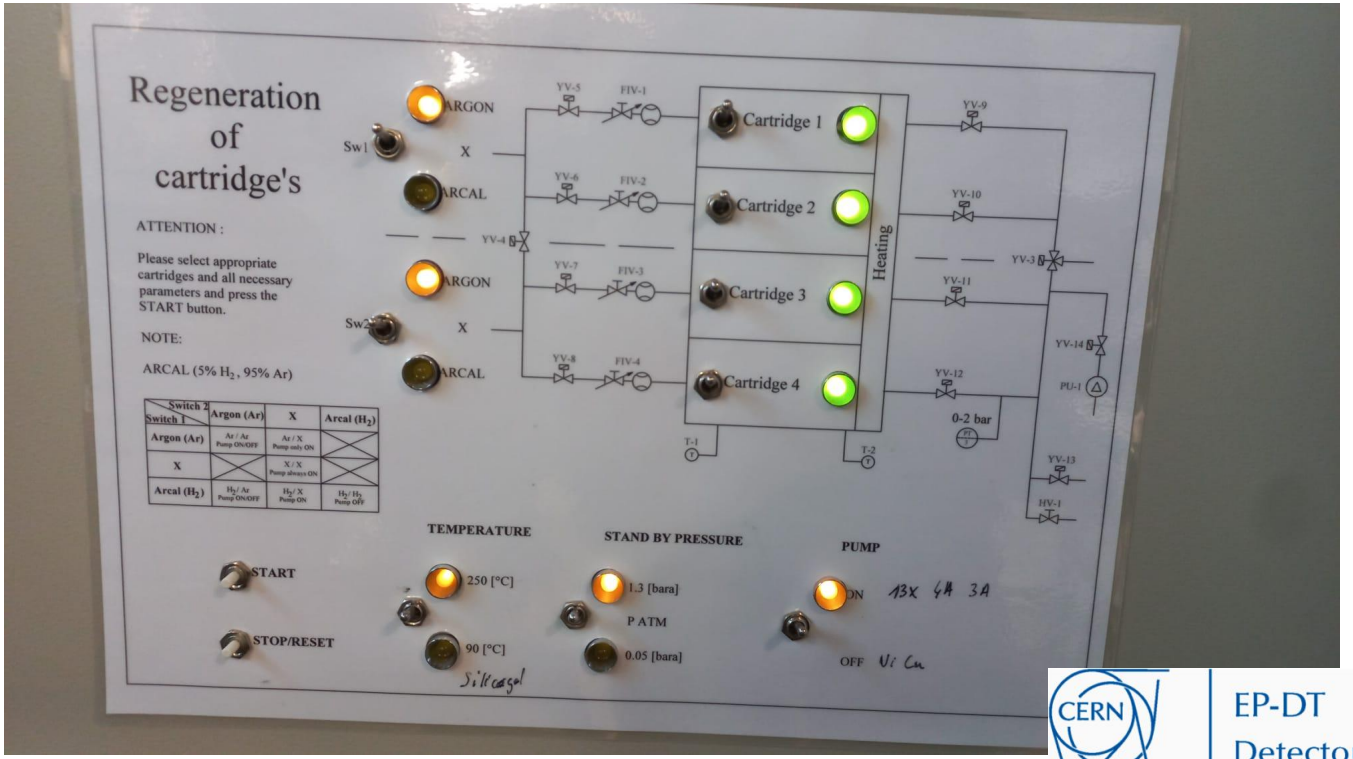
Cartridge's regeneration set up



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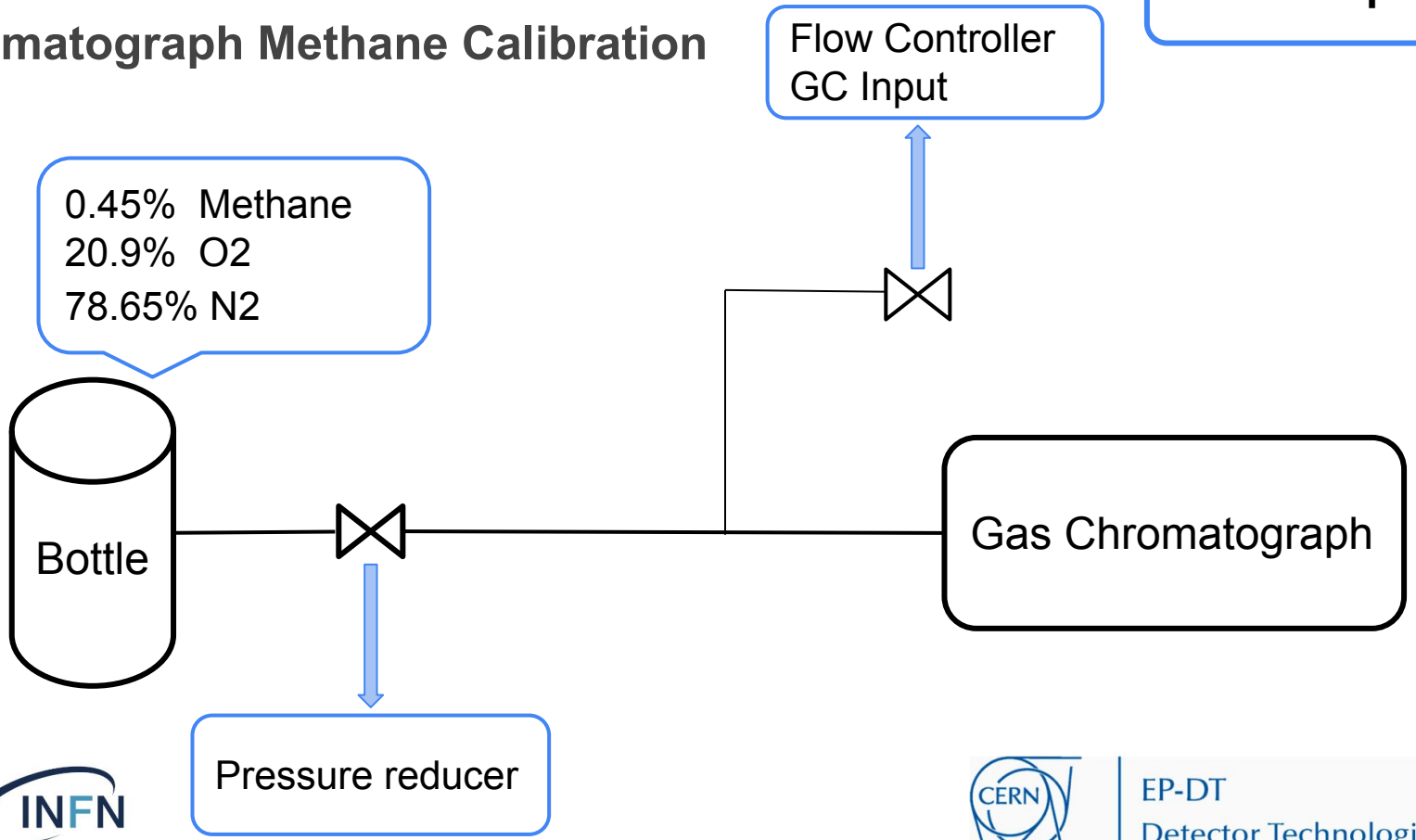
Methane Test - Day 12.12.2023

Gas Chromatograph Methane Calibration

Set up



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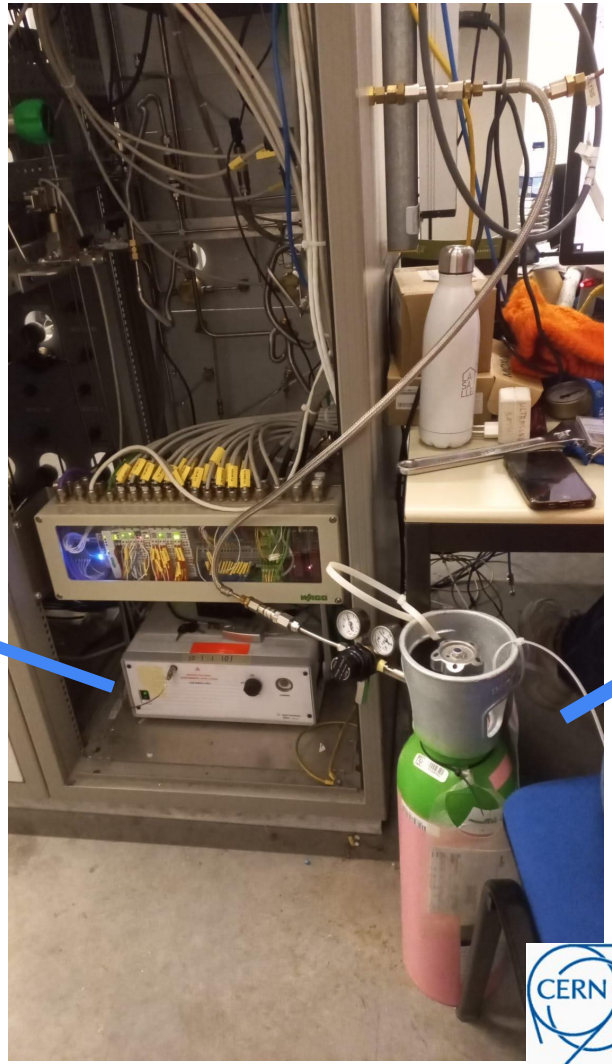
Gas Chromatograph Methane Calibration

Set up



Gas Chromatograph

Bottle Air-Methane

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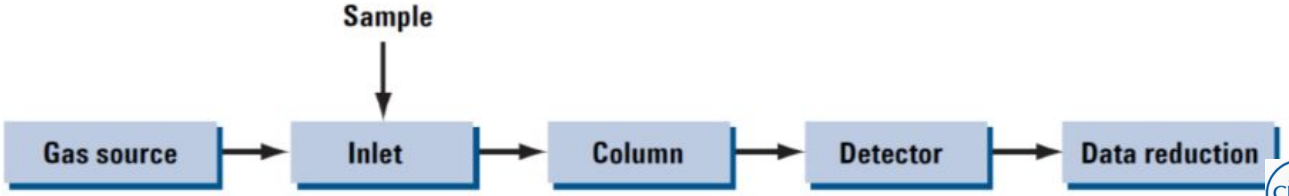
Fundamentals of Gas Chromatography

- It separates gas mixtures into individual components.
- GC creates a time separation of the components.
- The mixture passes through a column containing a material that retards some components more then others → *separation*.
- The components are detected by the TCD (thermal conductivity detector).
- The output of the GC is a gas chromatogram (µV versus time).
- The different components are identified by their retention time (time at which the peak appears in the chromatogram).
- Their concentration is given by the peak area.



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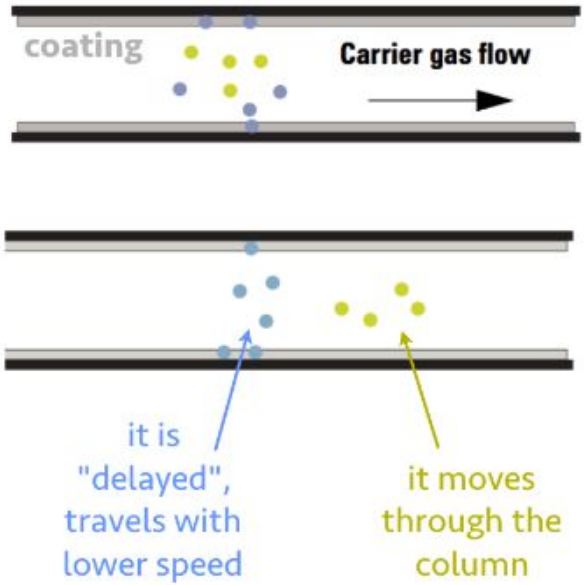
The carrier gas

- It is a pure gas (Ar, He, ..)
- It moves the sample through the GC
- It serves as a reference for the TCD

The column

- Different types of columns, depending on the coating material.
- The coating inside the column separates the components.
- Some components are more attracted than others to the coating.



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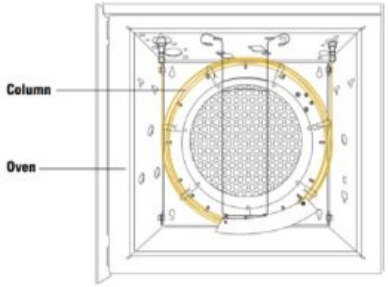
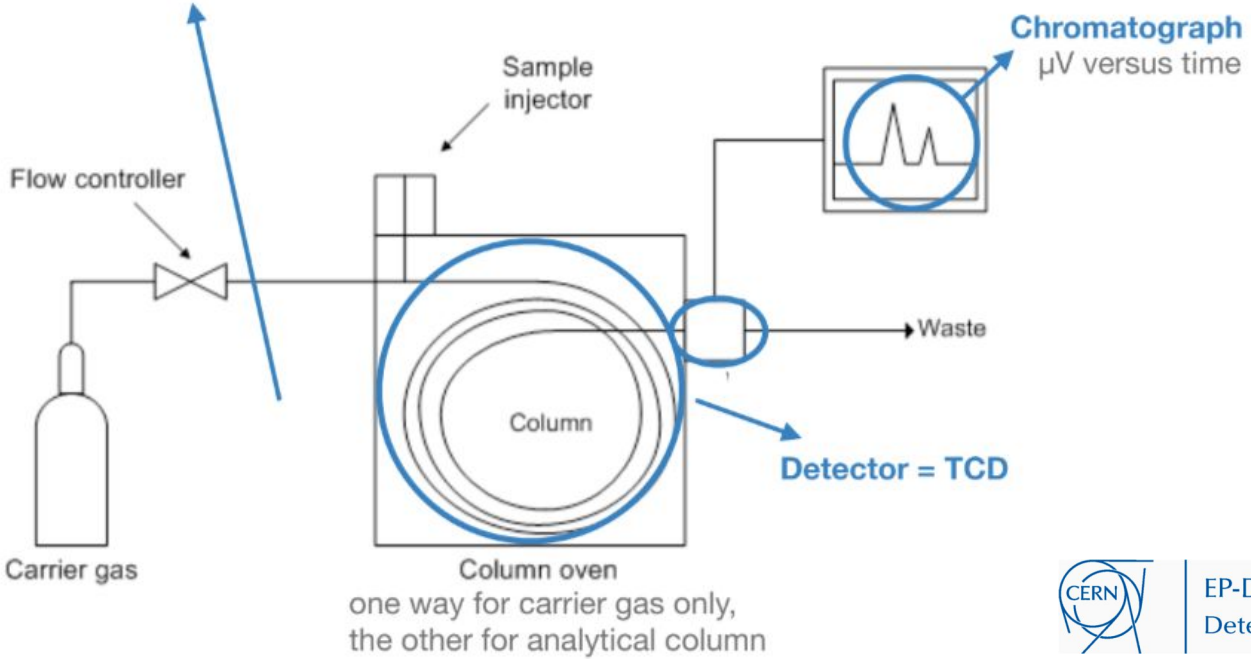


- Choice and thickness of column coating.
- Column length and diameter.
- Choice of carrier gas and flow rate.



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Gas Chromatograph Methane Calibration

The used Gas Chromatograph contains to columns with 2 different coating material that separates the components

PPU

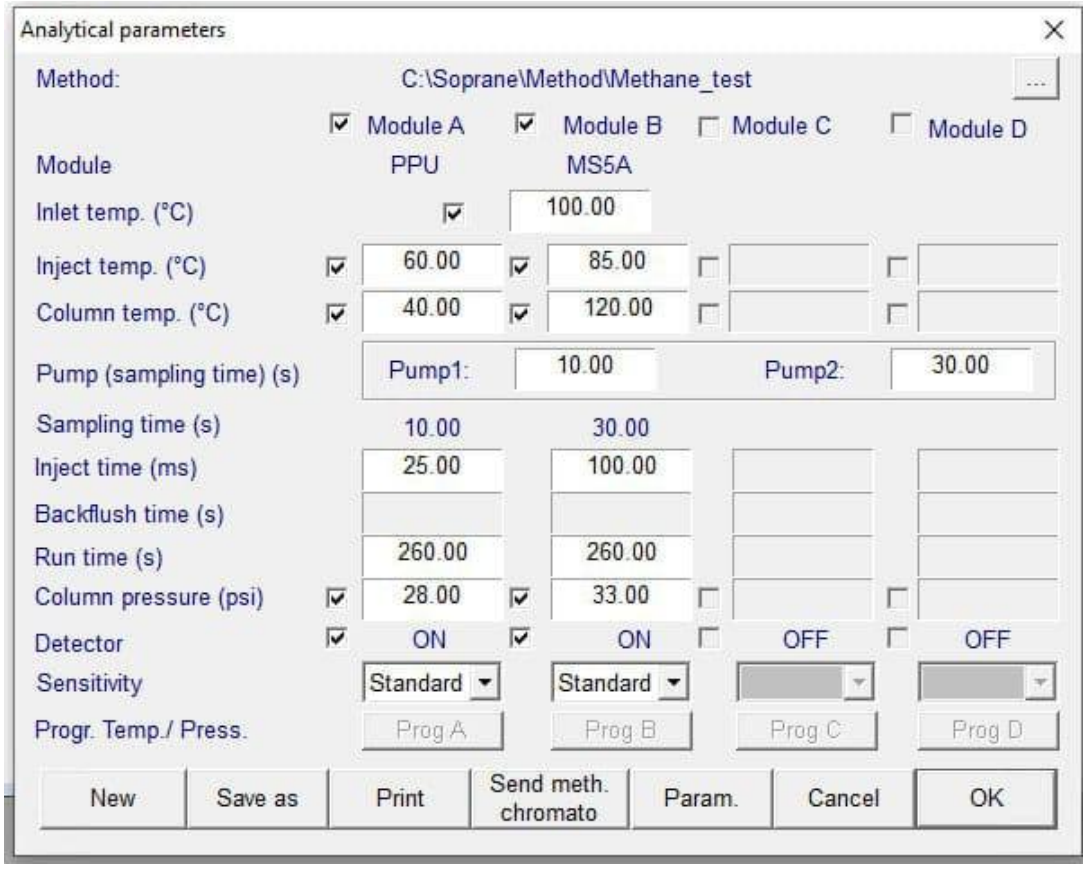
Shorter Methane retention time

Molecular Sieve

Longer Methane retention time

Methane Test - Day 12.12.2023

Gas Chromatograph Methane Calibration Method



Parameters to set to find the right calibration factor of the tested gas

Last of 1 analysis : 4,30 min

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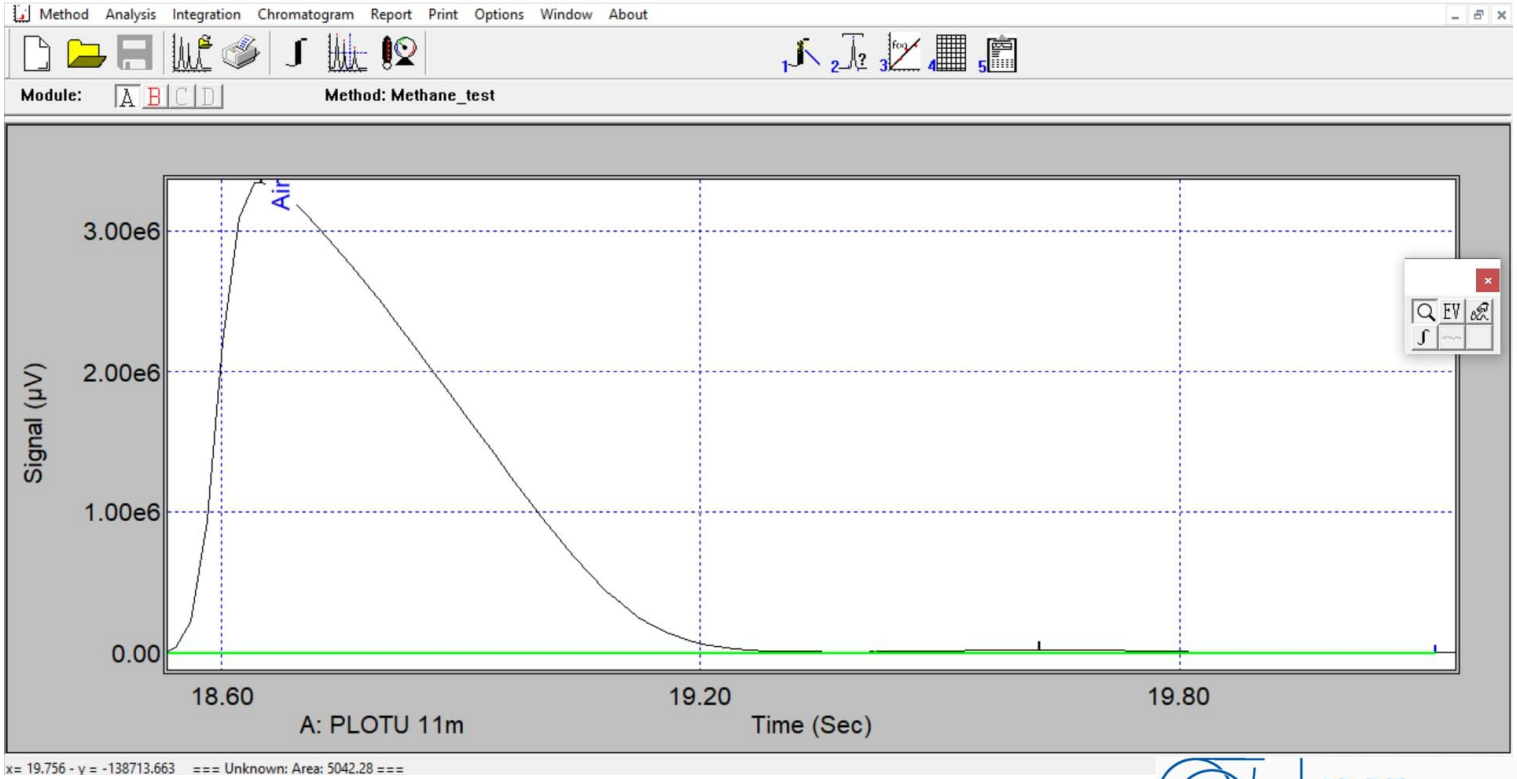
Gas Chromatograph Methane Calibration Chromatogram PPU



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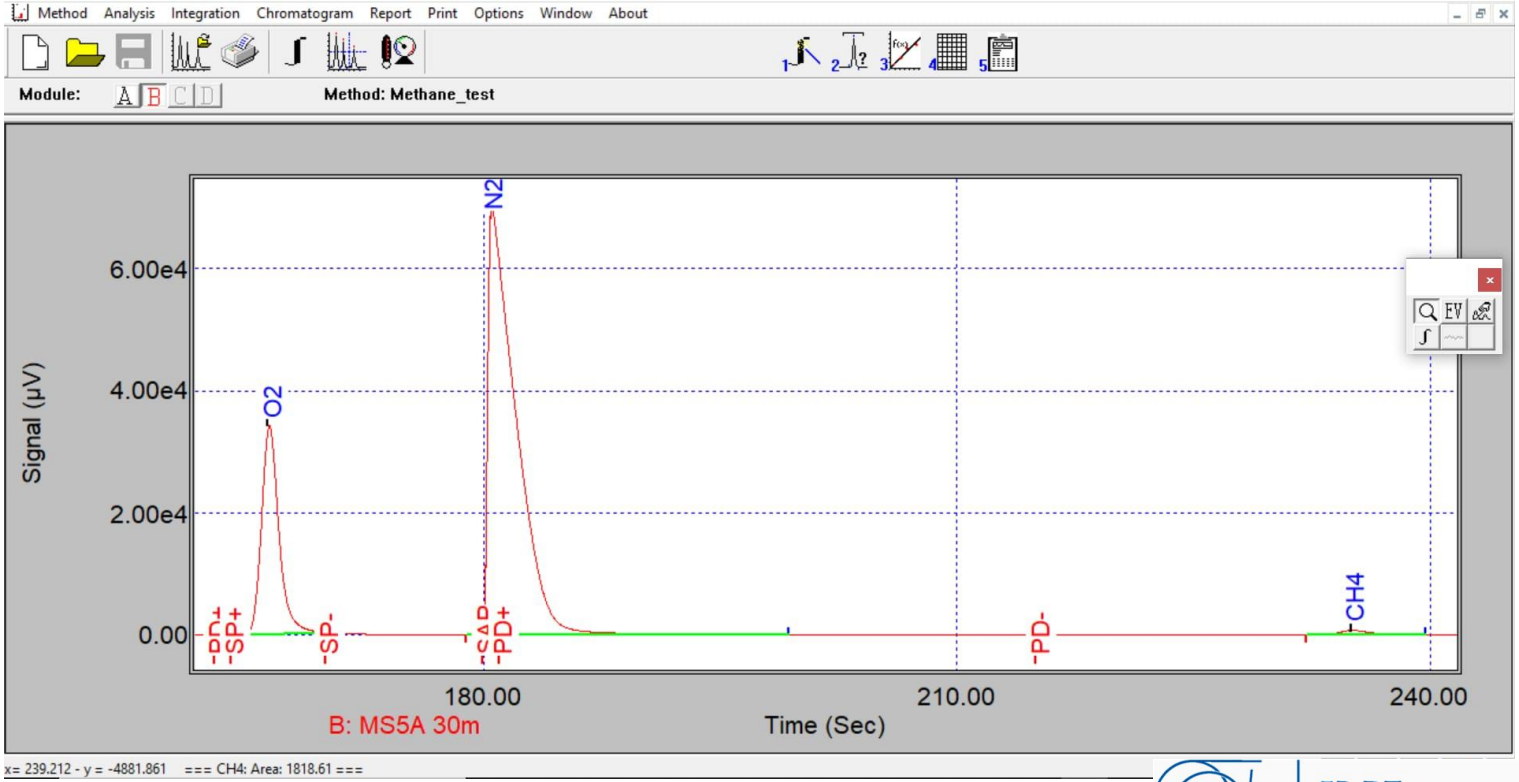
Gas Chromatograph Methane Calibration Chromatogram Mol Sieve



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Methane Test - Day 12.12.2023

Gas Chromatograph Methane Calibration

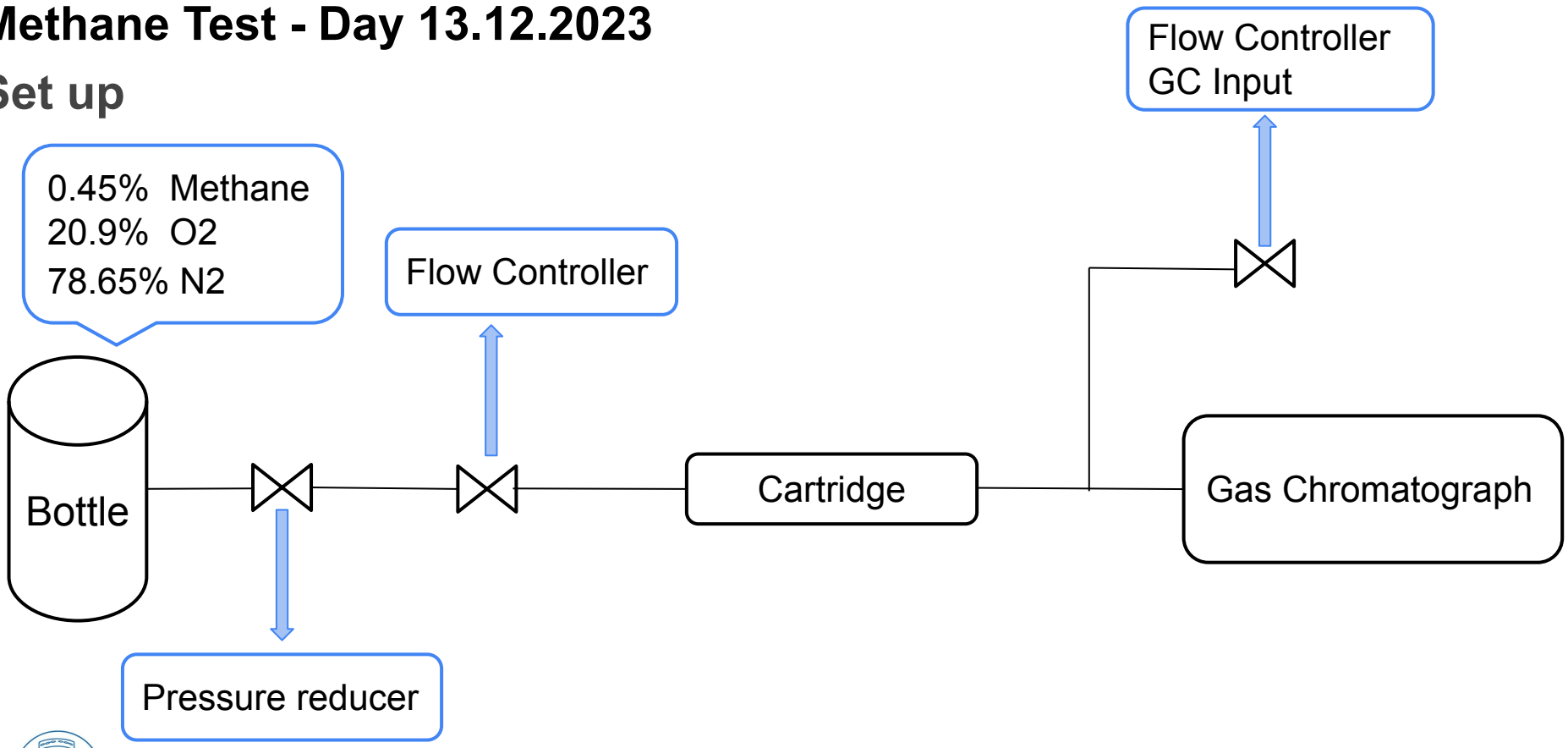
The calibration was performed using both the columns **PPU** and **Molecular Sieve**

Calibration Factor : Ratio between gas concentration and peak area

	Peak Area	**% Methane**	**Conversion Factor**
PPU	5197,406	0,4509	2,38 exp(-4)
MOL SIEVE	1896,988	0,4509	8,68 exp(-5)

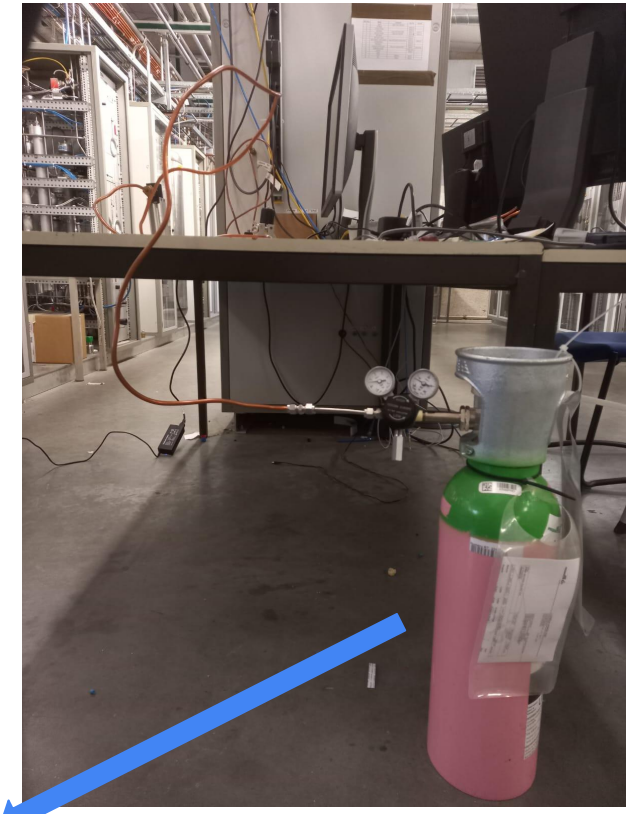
Methane Test - Day 13.12.2023

Set up



Methane Test - Day 13.12.2023

Set up



Cartiridge

Bottle
Air-Methane

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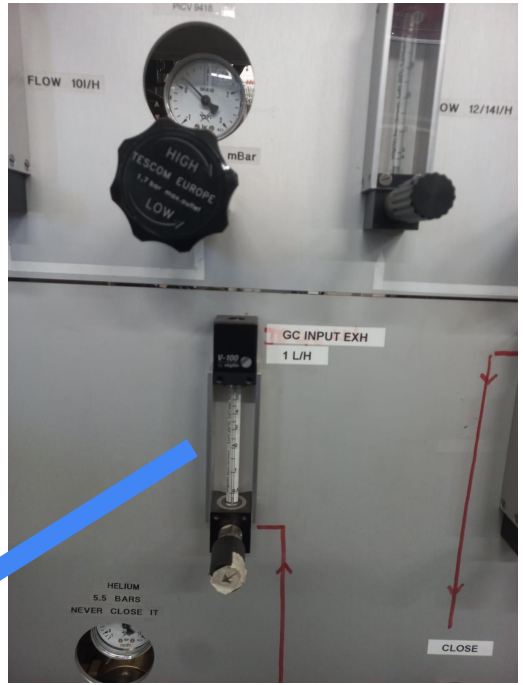
Methane Test - Day 13.12.2023

Set up



GC INPUT flux

Gas Chromatograph (GC)

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Methane Test - Day 14.12.2023

PROBLEMS!!!

1. No access to the pc connected to the Gas Chromatograph

2. Start using another Gas Chromatograph
   - It needed to be calibrated
   - Changed and moved the set up
   - After 8 analysis it stopped to work properly

3. Different tests were performed to try to solve the problems

Methane Test - Day 15.12.2023

Analysis Cartridge Z4

- The second Gas Chromatograph wasn’t working
- Access again to the first Gas Chromatograph
- Moved again the set up
- Started to analyse the cartridge Z4

Methane Test - Day 15.12.2023

Analysis Cartridge Z4

- 32 analysis were performed, during which the pressure was controlled and regulated
- In the last 40 minutes of analysis it wasn't check and in the end both the pressure and the flux on the GC rotameter were found to be zero
- The analysis 16th to 20th were considered to calculate the methane concentration at the output of the cartridge

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Methane Test - Day 15.12.2023

Analysis Cartridge Z4

Has the Cartridge already been saturated or is it not absorbing CH4 at all?

Results from the 5th stable analysis, before the pressure and flux falled down

	Peak Area	% Methane	% Variation (0.4509 %)
PPU	4936,594	0,4283	5
MOL SIEVE	1783,222	0,4239	6

No considerable variation of methane concentration : **Z4 cartridge doesn't absorb methane**

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Methane Test - Week 2

Summary

To Test :

- Z3
- Z5
- Z10

Z3 - Z4 To rigenerate

Z10 - Z5 Test Ongoing

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Methane Test - Day 18.12.2023

Analysis Cartridge Z10

- At the beginning of the analysis there in no CH4 at the output of the cartridge, so it's **absorbing methane**
- Then the CH4 % start to increase
- **After 50 min = 1,7 l of gas** at the cartridge's output there is **0,02%** of **CH4**, so it starts to saturate
- The **last Z10 analysis** resulted in **0,4% of CH4**

$$g_{(CH4\ absorbed)} = \frac{Vol\ CH4}{24\ mol/l} \cdot M_{mol(CH4)} = 0{,}51\ g$$

Methane Test - Day 18.12.2023

Analysis Cartridge Z5

- At the beginning of the analysis there in no CH4 at the output of the cartridge, so it's **absorbing methane**
- Then the CH4 % start to increase
- **After 50 min = 1,7 l of gas** at the cartridge's output there is **0,03%** of **CH4**, so it starts to **saturate**

$$g_{(CH4\ absorbed)} = \frac{Vol\ CH4}{24\ mol/l} \cdot M_{mol(CH4)} = 0{,}51\ g$$

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Methane Test - Day 19.12.2023

Analysis Cartridge Z5

- End of the Z5 analysis regenerated with heating at lab 256
- The **last Z5 analysis** resulted in **0,3% of CH4**
- Meeting : it was decided to **regenerate Z5** cartridge using the **pump**
- **Z5** connected to the **pump** for **2h 30min**
- Z5 cartridge was connected in the set up and flushed at 2 l/h to reach the atmospheric pressure inside the cartridge

Methane Test - Day 20.12.2023

Analysis Cartridge Z5 - pump regeneration

- Z5 cartridge was flushed at 2 l/h and the analysis with the gas chromatograph started
- 16 analysis were performed
- The **first Z5 analysis** regenerated by **pump** resulted in **0,077% of CH4**, with an increasing trend

Z5 cartridge was not completed regenerated in 2h 30 min of aspiration from pump

Methane Test - Day 20.12.2023

Analysis Cartridge Z10 - pump regeneration

- **Z10** connected to the **pump** for **5h,** then set in the set up and flushed at 2 l/h to reach the atmospheric pressure
- 14 analysis were performed
- The **first Z10 analysis** regenerated by **pump** resulted in **0,03% of CH4**, with an increasing trend
- The **last Z10 analysis** resulted in **0,33% of CH4**

Z10 cartridge was not completed regenerated in 5h of aspiration from pump

Methane Test - Day 21.12.2023

Analysis Cartridge Z10 - pump regeneration (repeated analysis with the same steps from the day before)

- **Z10** connected to the **pump** for **5h**, then set in the set up and flushed for 30 min at 2 l/h to reach the atmospheric pressure, so the **cartridge's volume is 1 l**
- At the beginning of the analysis there in no CH4 at the output of the cartridge, so it was **completed regenerated**
- **After 25 min = 0,83 l of gas** at the cartridge's output there is **0,018%** of **CH4**, so it starts to **saturate**

$$g_{(CH4\ absorbed)} = \frac{Vol\ CH4}{24\ l/mol} \cdot Mmol_{(CH4)} = 0{,}25\ g$$

Methane Test

Results

From **Z3-Z4 cartridges** analysis they **don't absorb methane.** They were tested in the preliminary phase. Need to be **tested again to confirm the results**

Z5 cartridge was **not completed regenerated** in **2h 30 min** of aspiration from **pump**. It needs be c**onnected to the pump longer** and be **tested again**

Z10 cartridge was **not firstly completed regenerated** in **5 h** of aspiration from **pump**. The measurement was repeated.

Methane Test

Results

Parameters before saturation	Z5 (Heating)		Z10 (Heating)		Z10 (Pump 2)
Time	50 min		50 min		25 min
CH4 litres	0,76 l		0,76 l		0,37 l
g CH4 absorbed	0,51 g		0,51 g		0,25 g

Z10 cartridge was secondly completed regenerated in 5h of aspiration from pump