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INFN

CERN

EP-DT
Detector Technologies

CAMPAGNA DI MISURE CERN 30/04 – 13/05

Francesco A. Angiulli, Maria Cristina Arena, Matteo Brunoldi, Simone Calzaferri, EP-DT Group

PURPOSE

Calibration at different Bronkhorst pressure values

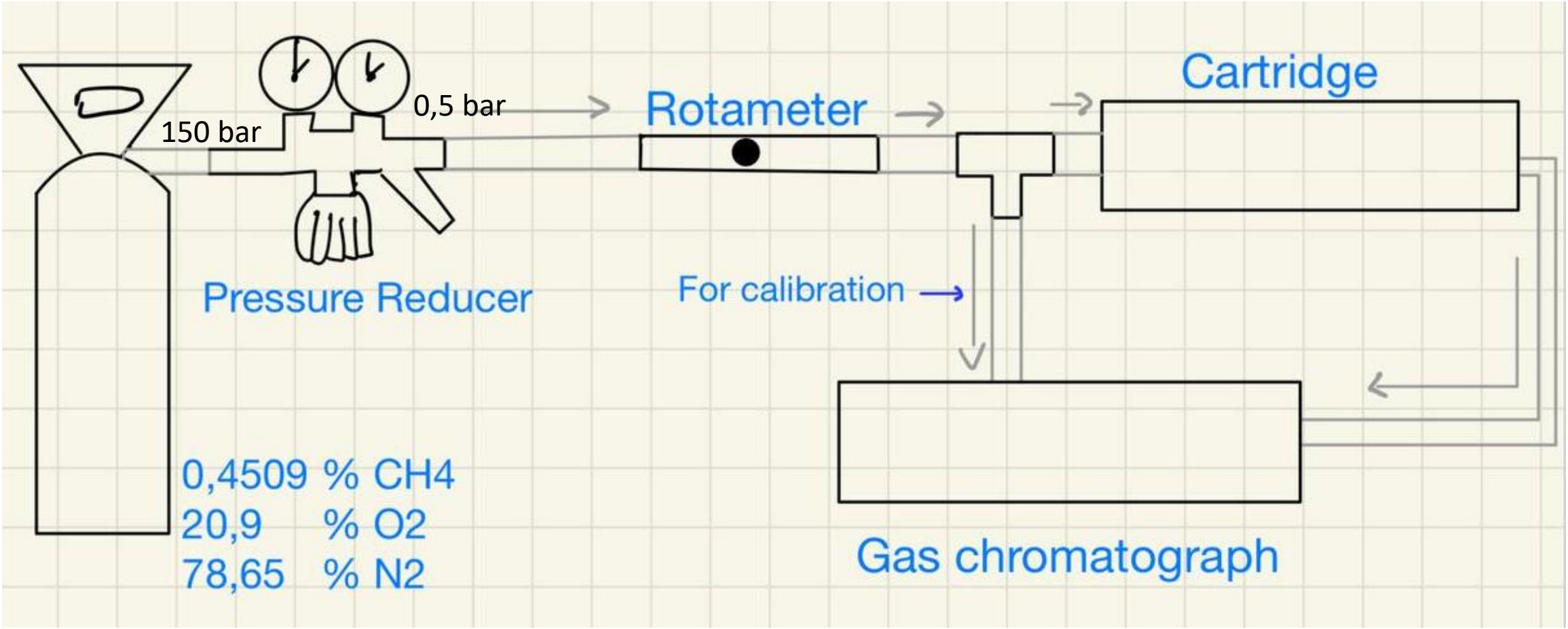
Evaluating different molecular sieves for capturing methane:

- Z3 - Z4 - Z5 - Z10

Performance after high temperature regeneration

Performance after regeneration with vacuum pump

MEASUREMENT SETUP



GC CLIBRATION FOR 4509 ppm METHANE

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Bronkhorst pressure = 100 mbar

	PPU AREA	PPU CONVERSION FACTOR		MS AREA	MS CONVERSION FACTOR
AVERAGE	5080	$8{,}875 \cdot 10^{-5}$	AVERAGE	1907	$2{,}365 \cdot 10^{-4}$
DEV. STD	37	$6{,}6 \cdot 10^{-7}$	DEV. STD	11	$1{,}3 \cdot 10^{-6}$
AVG DEV. STD	16	$2{,}9 \cdot 10^{-7}$	AVG DEV. STD	4,8	$5{,}9 \cdot 10^{-7}$

Bronkhorst pressure = 60 mbar

	PPU AREA	PPU CONVERSION FACTOR		MS AREA	MS CONVERSION FACTOR
AVERAGE	5091	$8{,}858 \cdot 10^{-5}$	AVERAGE	1894	$2{,}380 \cdot 10^{-4}$
DEV. STD	46	$8{,}0 \cdot 10^{-7}$	DEV. STD	15	$1{,}9 \cdot 10^{-6}$
AVG DEV. STD	20	$3{,}6 \cdot 10^{-7}$	AVG DEV. STD	6,7	$8{,}5 \cdot 10^{-7}$

Bronkhorst pressure = 30 mbar

	PPU AREA	PPU CONVERSION FACTOR		MS AREA	MS CONVERSION FACTOR
AVERAGE	5144	$8{,}766 \cdot 10^{-5}$	AVERAGE	1932	$2{,}333 \cdot 10^{-4}$
DEV. STD	48	$8{,}3 \cdot 10^{-7}$	DEV. STD	15	$1{,}9 \cdot 10^{-6}$
AVG DEV. STD	22	$3{,}7 \cdot 10^{-7}$	AVG DEV. STD	6,9	$8{,}4 \cdot 10^{-7}$

FIRST SERIES OF MEASUREMENTS

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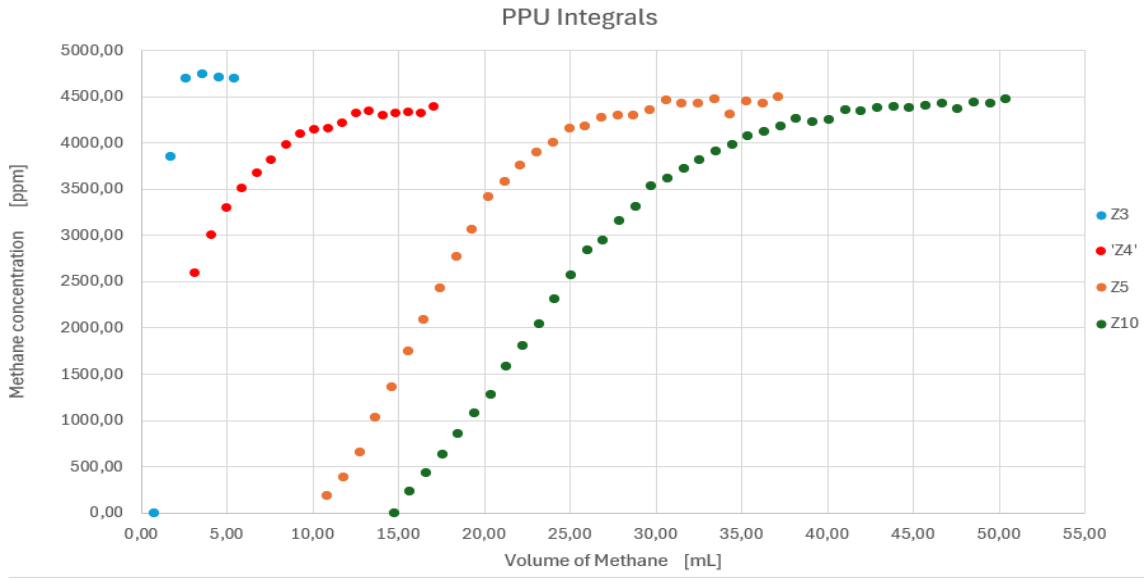
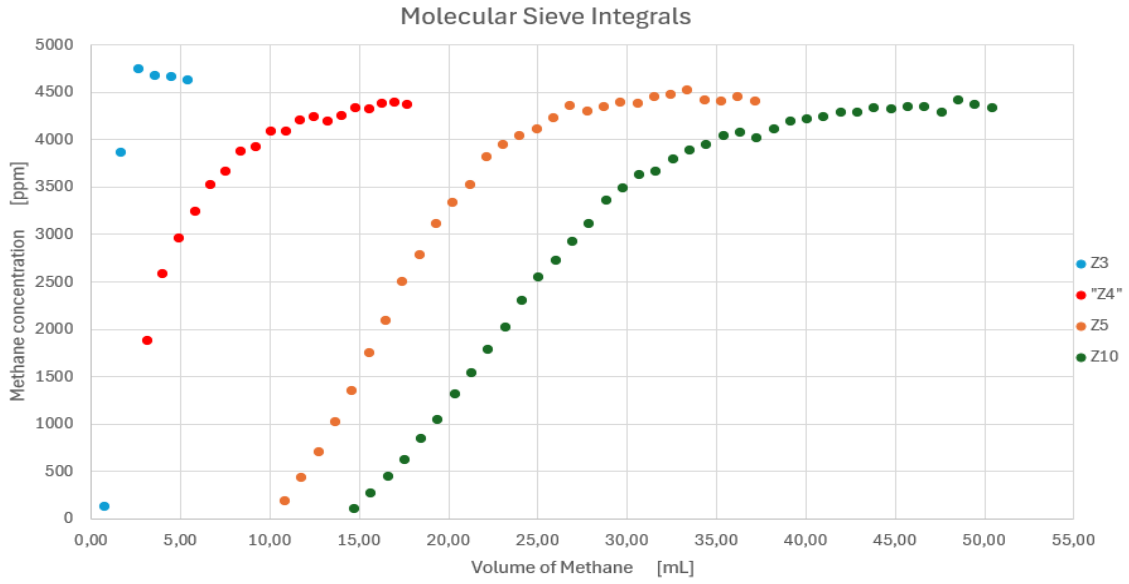
High temperature regenerated cartridges:



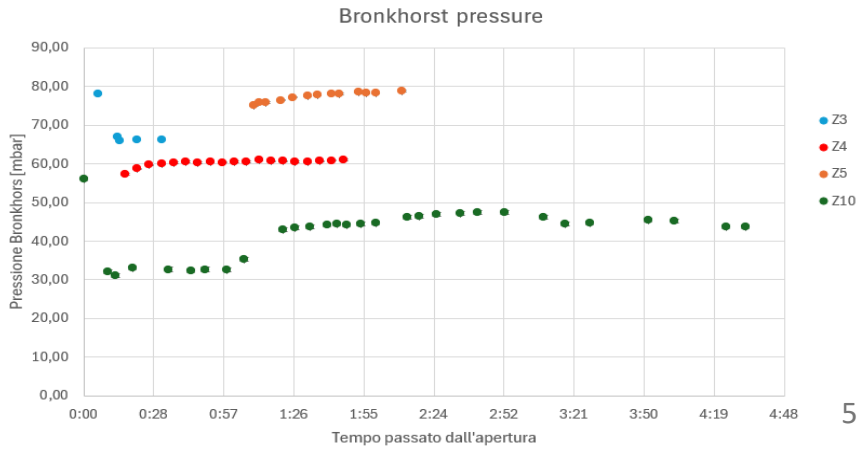


Absorbed methan before peak appearance:

$Z3 = 0{,}75\ mL$ $Z4 = 3{,}13\ mL$

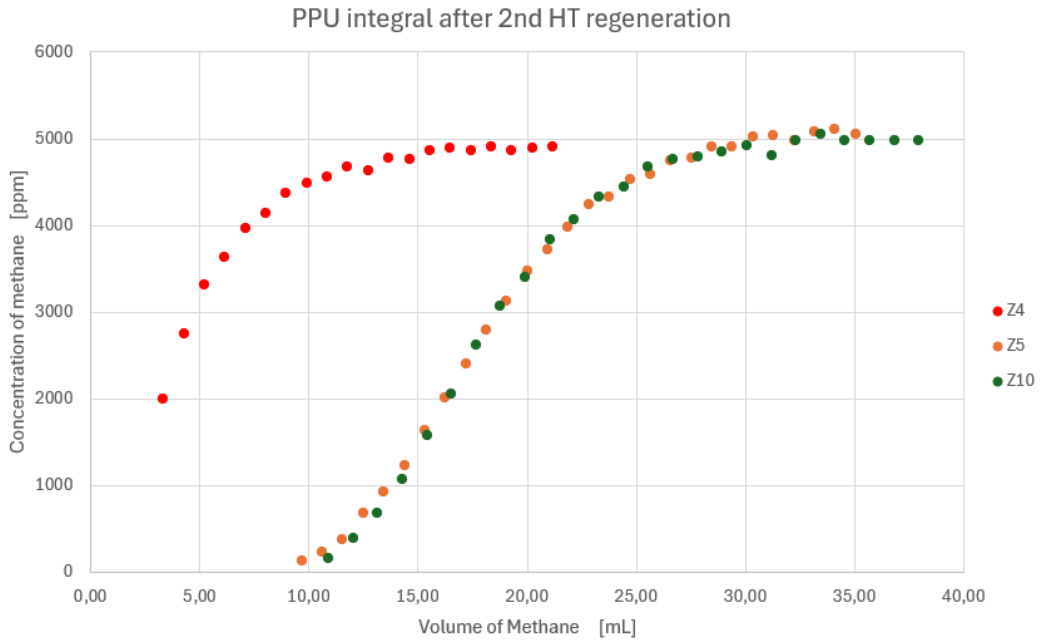
$Z5 = 10{,}86\ mL$ $Z10 = 14{,}73\ mL$

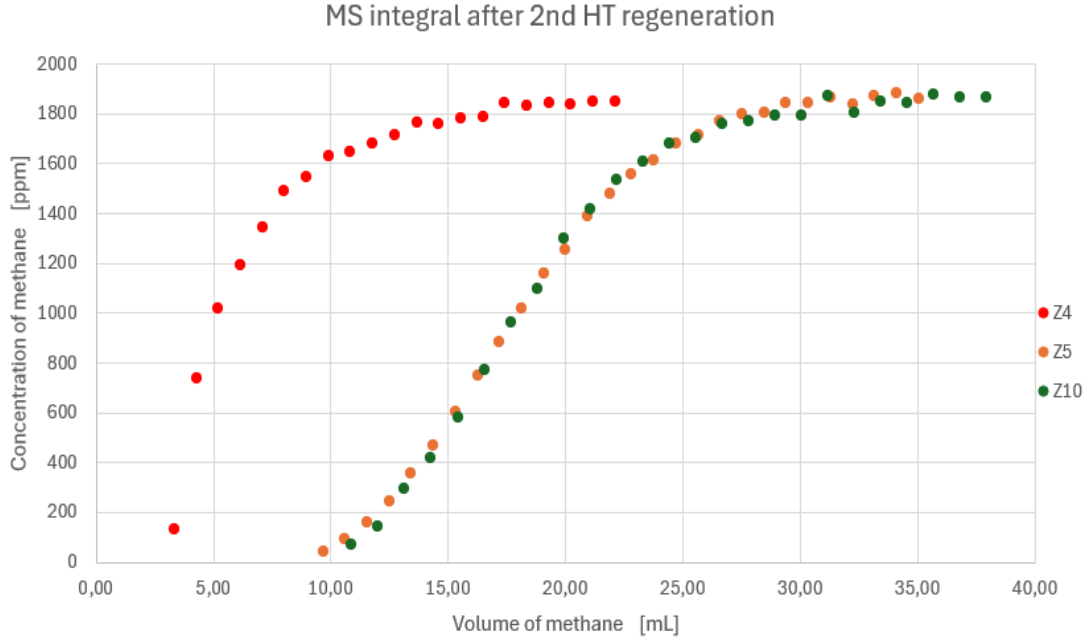
Overall error of 20% due to rotameter measures



SECOND SERIES OF MEASUREMENTS

High temperature regenerated cartridges:





Absorbed methan before peak appearance:

$Z3 = ---$ $Z4 = 3{,}33\ mL$

$Z5 = 9{,}68\ mL$ $Z10 = 10{,}90\ mL$ → Z10 not completely regenerated

Overall error of 20% due to rotameter measures

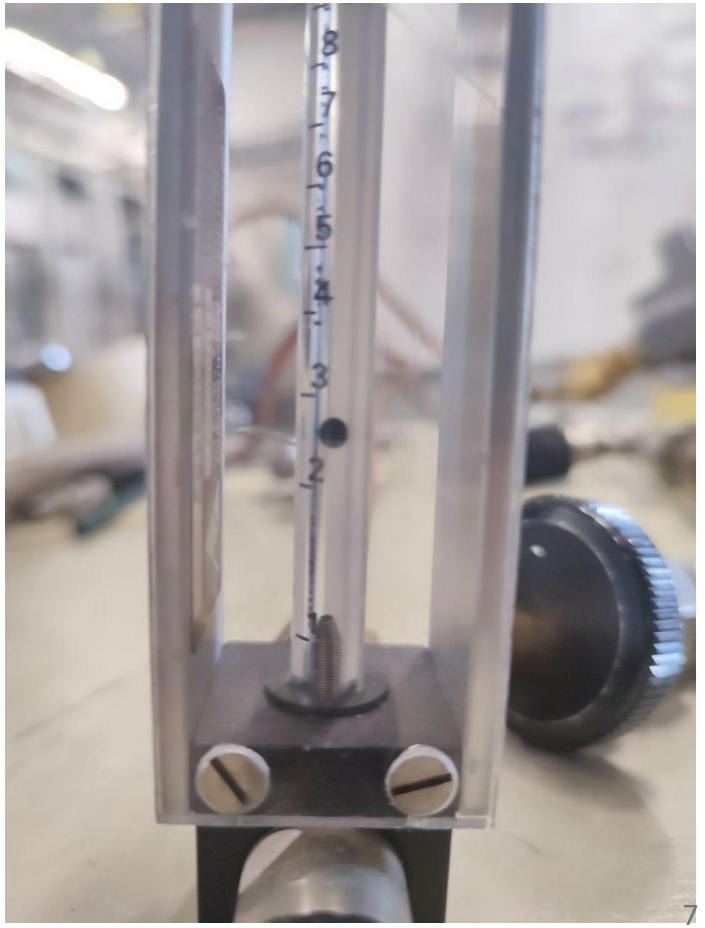
RESULTS OF FIRST WEEK

Performance of Z5, Z10 are much better than Z3, Z4

Z3, Z4 excluded from further measurements

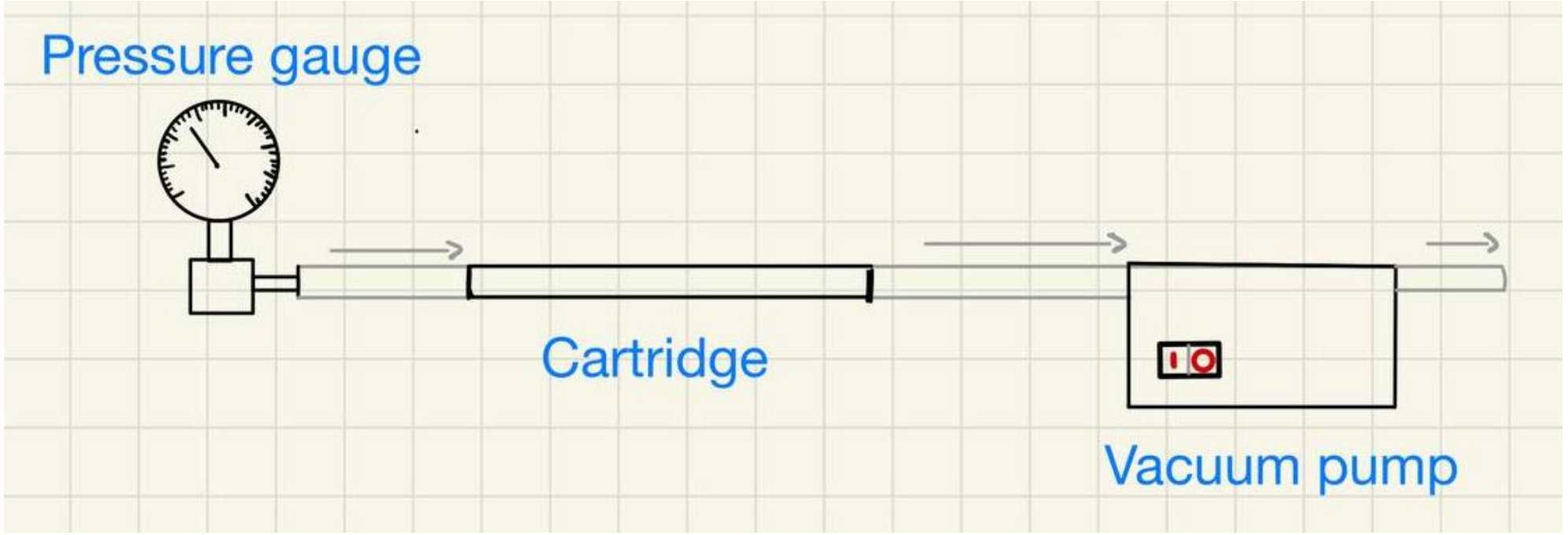
Overall error around 20% due to rotameter measures on flowed Methane.

0,5 L/h error on a 2,5 L/h measure



VACUUM PUMP REGENERATION

SETUP:



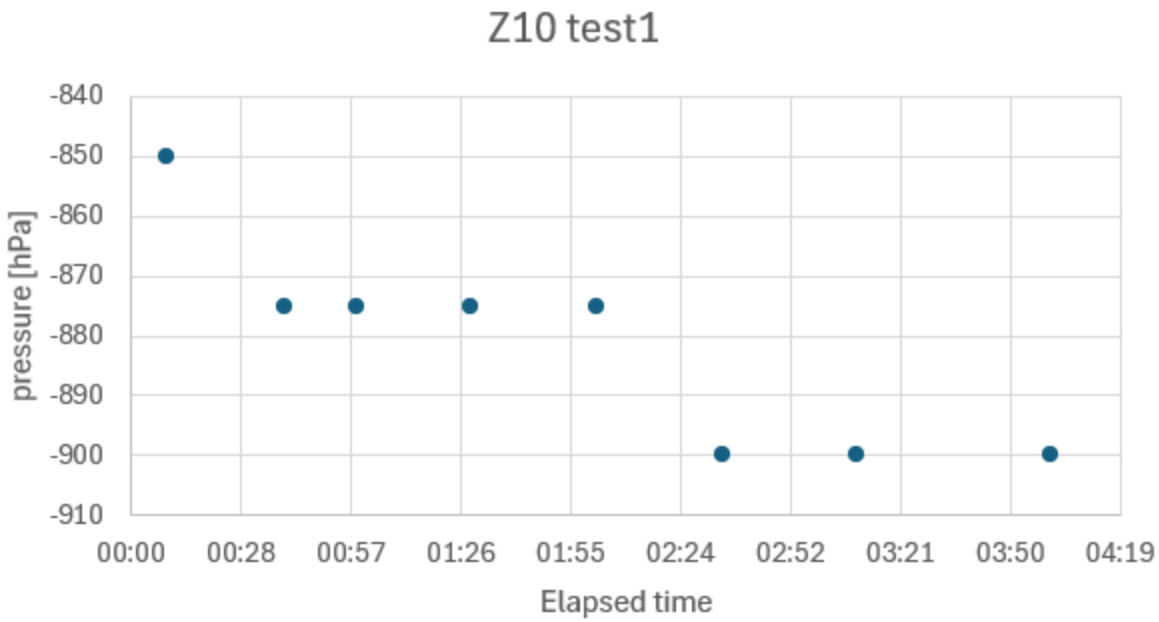
VACUUM PUMP REGENERATION

After the activation of the pump the pressure goes from a *difference* of 0 with respect to the atmosphere pressure to a value between **-970 mbar** and **-960 mbar**

At the beginning the pressure gauge was not enough sensitive to measure this changes.

Pressure range was (-1, +5)

From 09/05 we had new pressure gauge that measures in the range (-1,1) with a sensitivity of *10 mbar*



Measurements with 1° pressure gauge

LONG VACUUM REGENERATIONS

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FPA = First Peak Appearance of CH4 in the chromatogram

	Cartridge	Regeneration time	Methane before FPA	Time before FPA	Saturation methane	Saturation time	Bronkhorst pressure
*	Z5	19 h	14,6 mL	46 min	36 ÷ 40 mL	2 h 51 min	35 → 44 mbar
	Z10	17 h	11 mL	35 min	35 ÷ 39 mL	2 h 41 min	38 → 49 mbar
	Z5	4 h	13 mL	45 min	36 ÷ 39 mL	2 h 56 min	37 → 48 mbar
→	Z10	4 h	8,4 mL	30 min	30 ÷ 34 mL	2 h 45 min	38 → 49 mbar
	Z5	3 h	10 mL	40 min	30 ÷ 34 mL	2 h 50 min	38 → 48 mbar
	Z10	3 h	8 mL	40 min	30 ÷ 34 mL	2 h 45 min	45 ÷ 47 mbar
→	Z5	2h	11 mL	55 min	29,5 ÷ 33,5 mL	2 h 55 min	44 → 52 mbar

*measurements corrupted by interruptions during the flow

→ Last Z10 analysis before valve replacemente

→ Last Z10 analysis before valve replacemente

LONG VACUUM REGENERATION

With long vacuum regeneration:

- Z5 : seems to have the same performance, but the value of methane may be overestimated (*rotameter inclined → higher flux*)

- Z10 : is not completely regenerated, it absorbs less than whaht it did in previous measurements

$$Z5\ long\ vacuum\ regeneration\ efficiency = \frac{12{,}5}{10{,}86} \approx 112\ \%$$

$$Z10\ long\ vacuum\ regeneration\ efficiency = \frac{9{,}1}{14{,}73} \approx 64\ \%$$

SHORT VACUUM REGENERATION

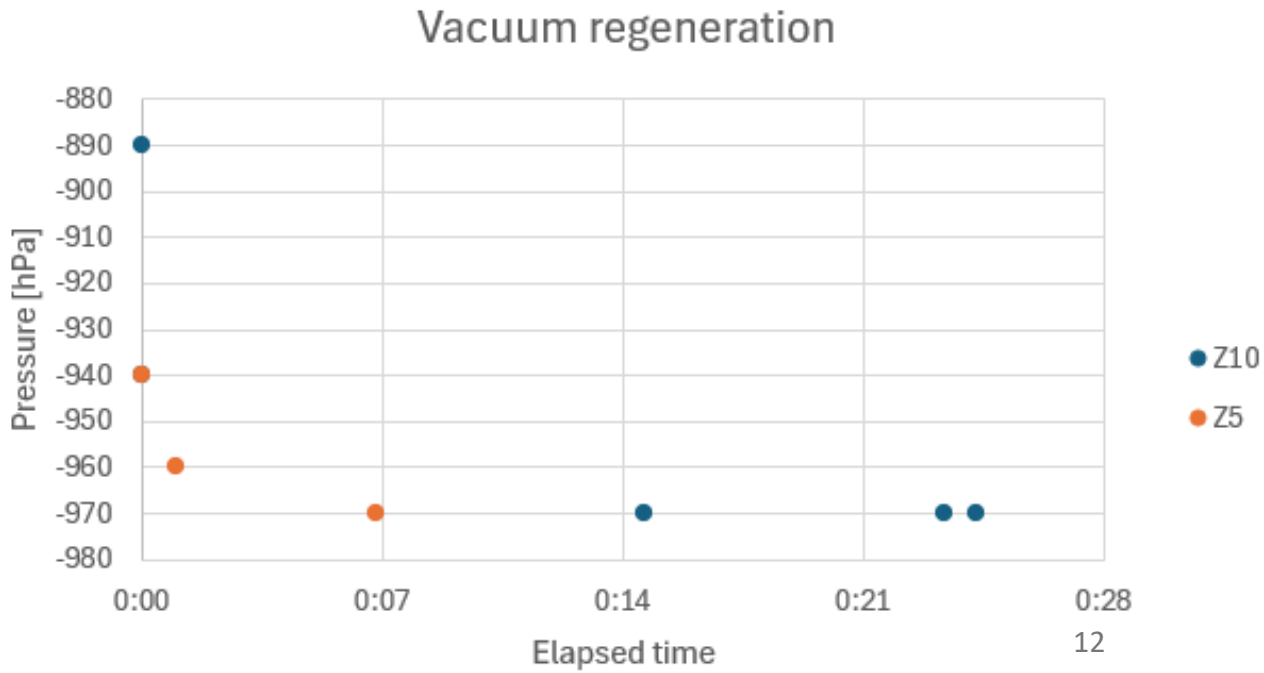
Next approach: stopping regeneration when pressure in the cartridge gets to the minimum value

Minimum value is measured with no cartridge in the setup.

Target value for pressure is between **-970 mbar** and **-960 mbar**

This target value is reached pretty soon for bothcartridges.

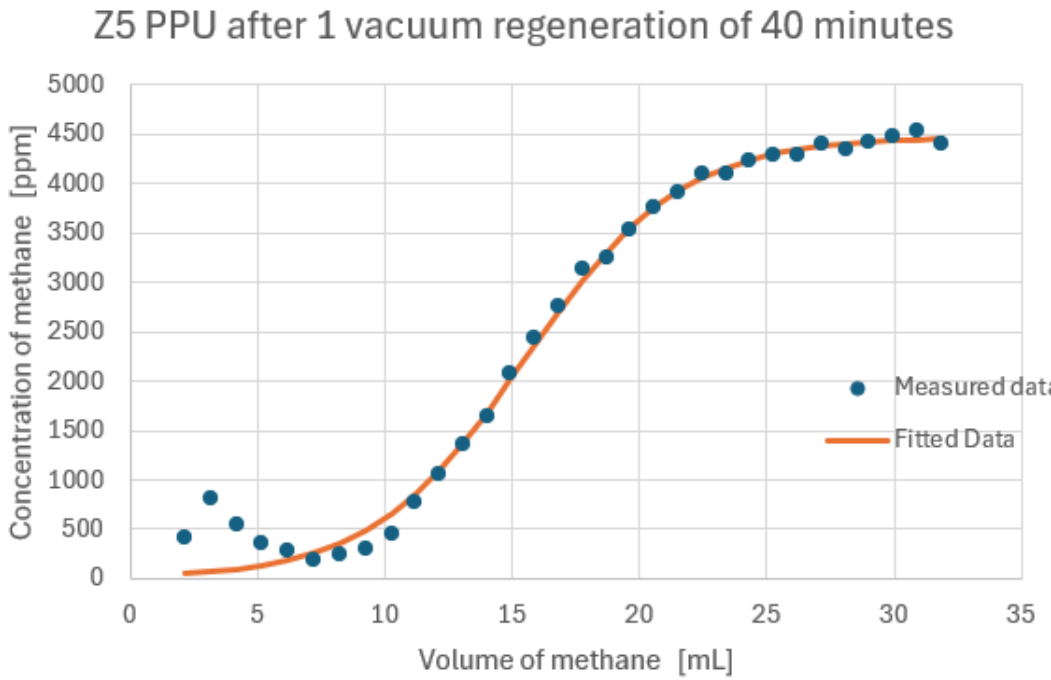
It also depends on the presence of leaks

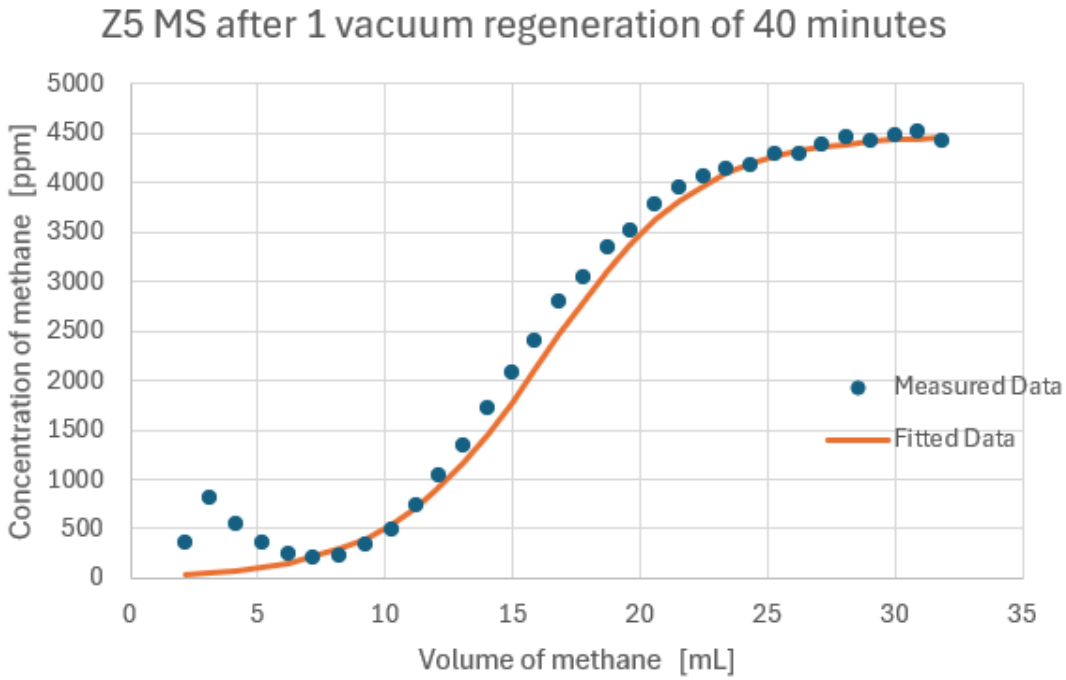


Z5 SHORT VACUUM REGENERATIONS

Z5 reaches the target value of pressure in 40 minutes, then it gets filled with the gas mixture

After 1° regeneration:





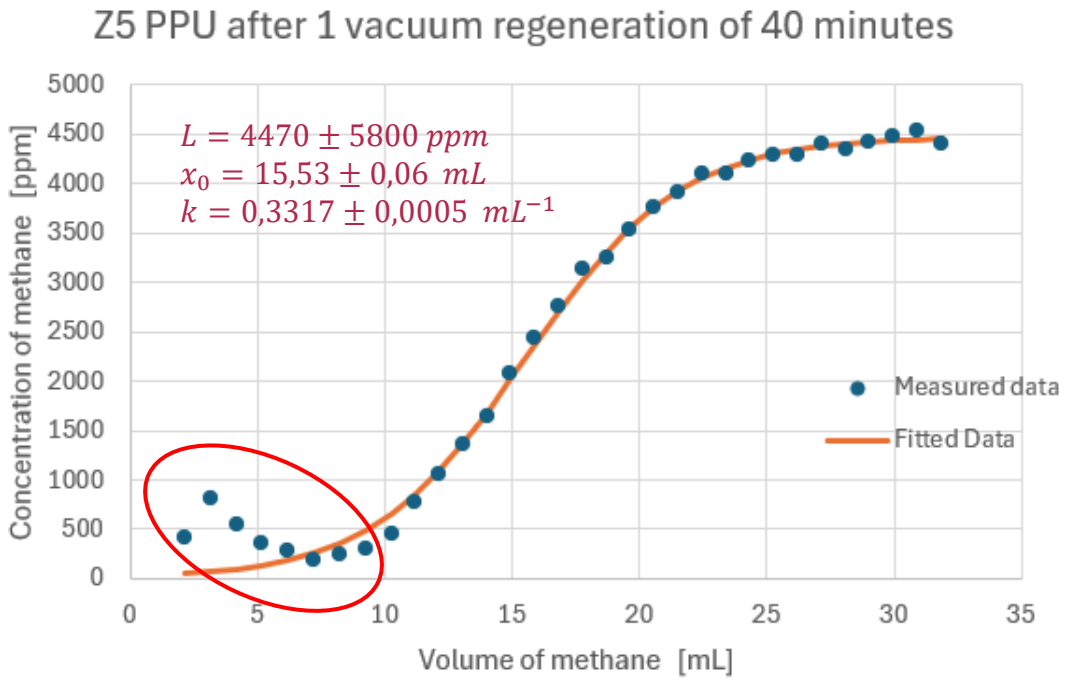
Fit function: $sigmoid = \frac{L}{1+e^{-(x-x_0)\cdot k}}$

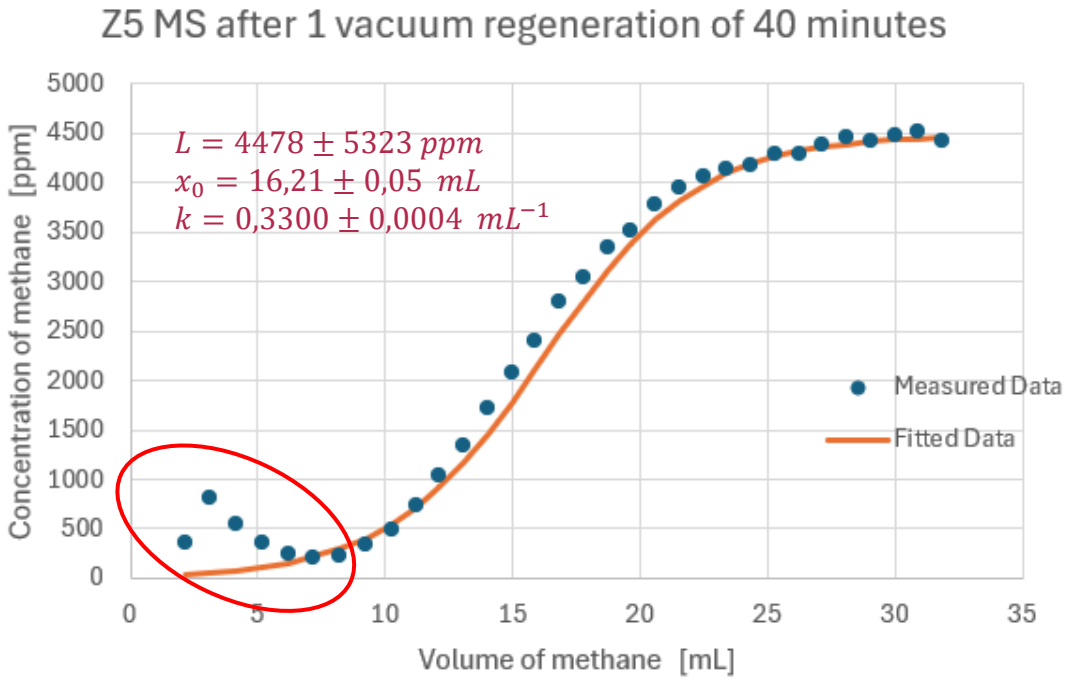
L = saturation level x_0 = x value to reach $\frac{L}{2}$ k = slope of the curve

Z5 SHORT VACUUM REGENERATIONS

Z5 reaches the target value of pressure in 40 minutes, then it gets filled with the gas mixture

After 1° regeneration:



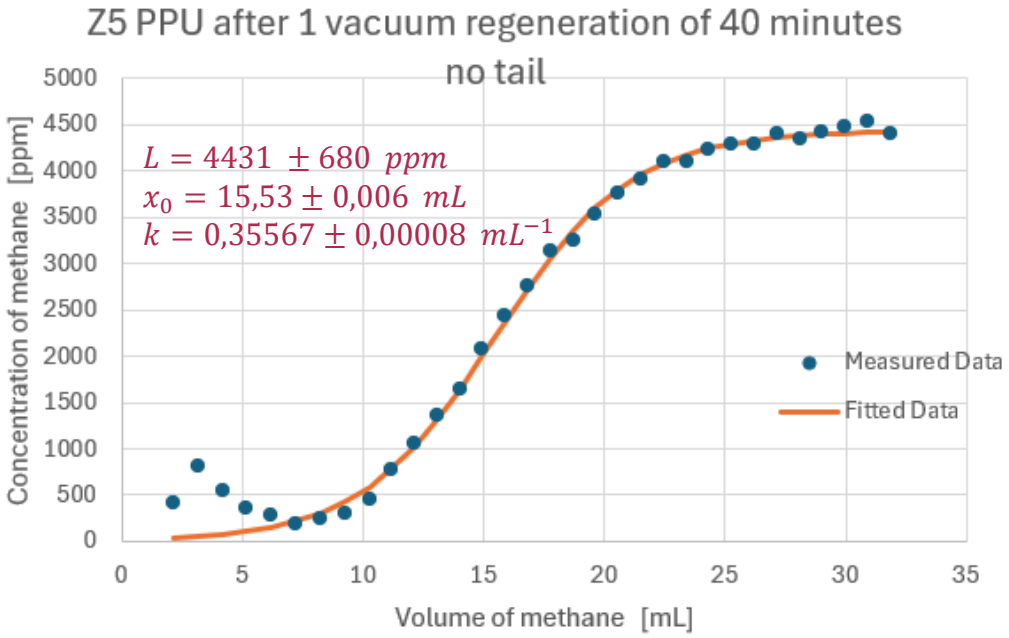


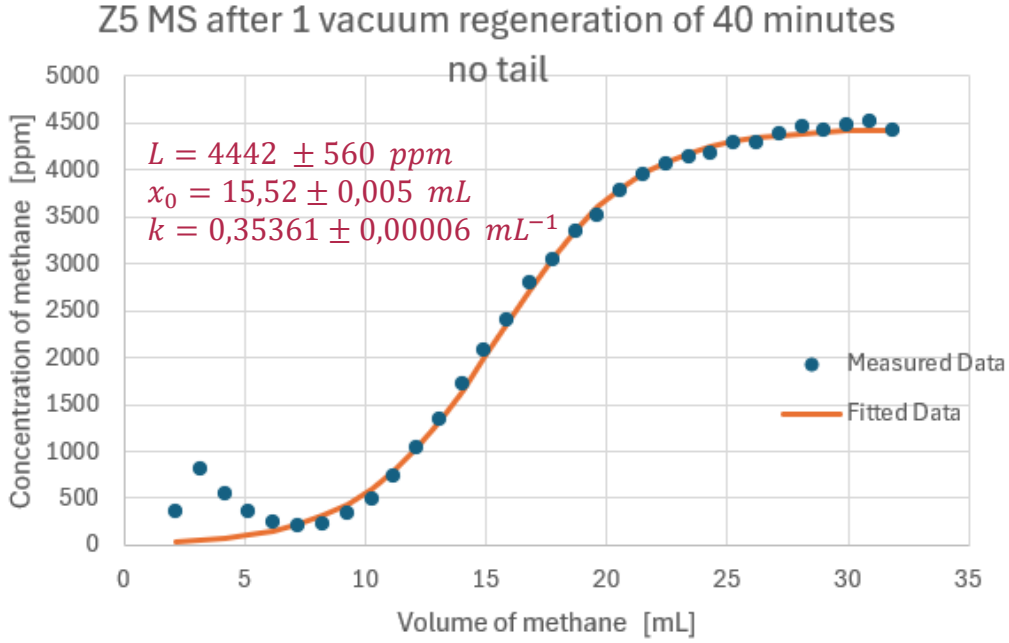
Fit function: $$\boldsymbol{sigmoid = \frac{L}{1+e^{-(x-x_0)\cdot k}}}$$

L = saturation level x_0 = x value to reach $\frac{L}{2}$ k = slope of the curve

Z5 SHORT VACUUM REGENERATIONS

Fit without the tail points





Huge error on L

Besides the anomaly, the minimum corresponds to **7,20 mL** of methane

Z5 SHORT VACUUM REGENERATIONS

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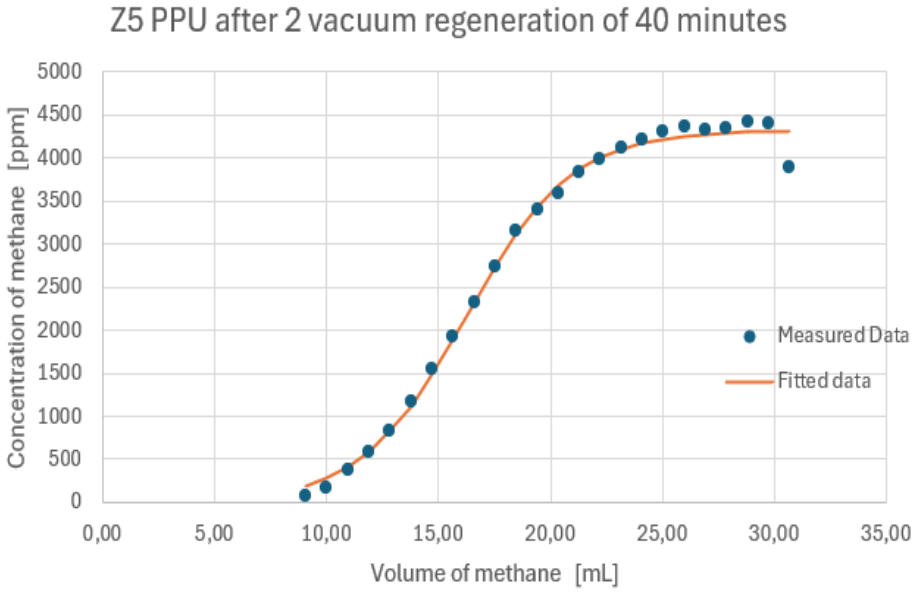
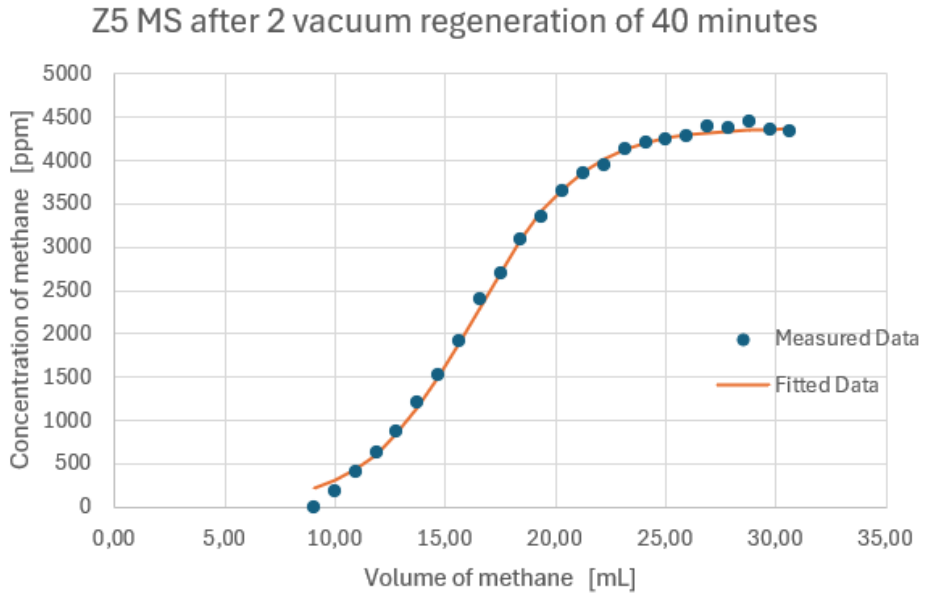
2° regeneration of 40 minutes:





$x_{0\,PPU} = 16{,}214 \pm 0{,}014\ mL$

$x_{0\,MS} = 16{,}303 \pm 0{,}005\ mL$

$$FPA = 10{,}01\ mL$$

Z5 SHORT VACUUM REGENERATIONS

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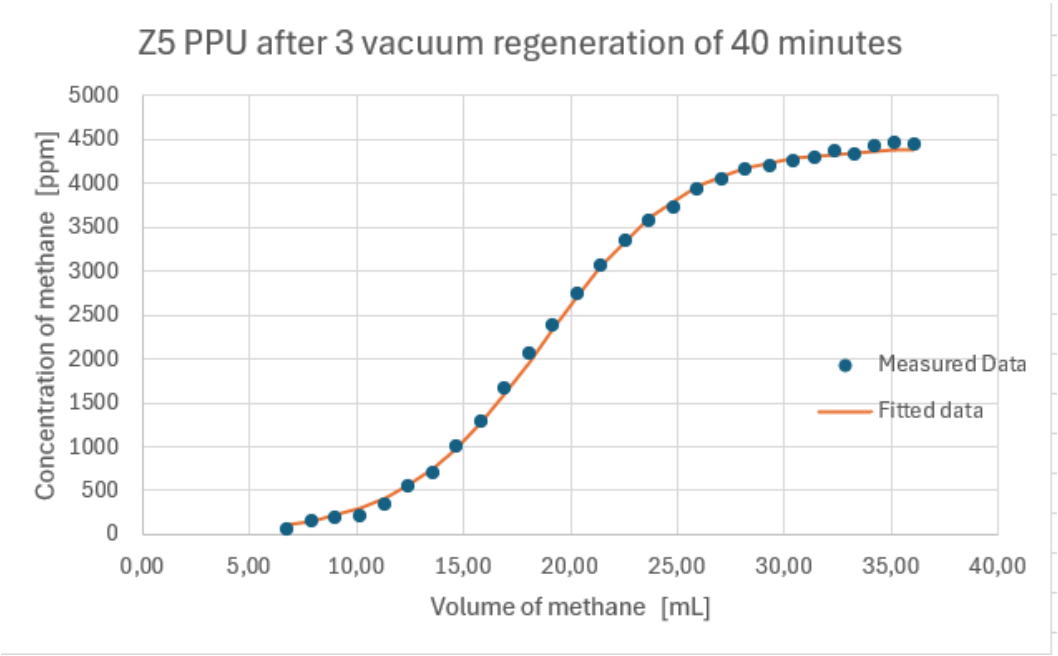
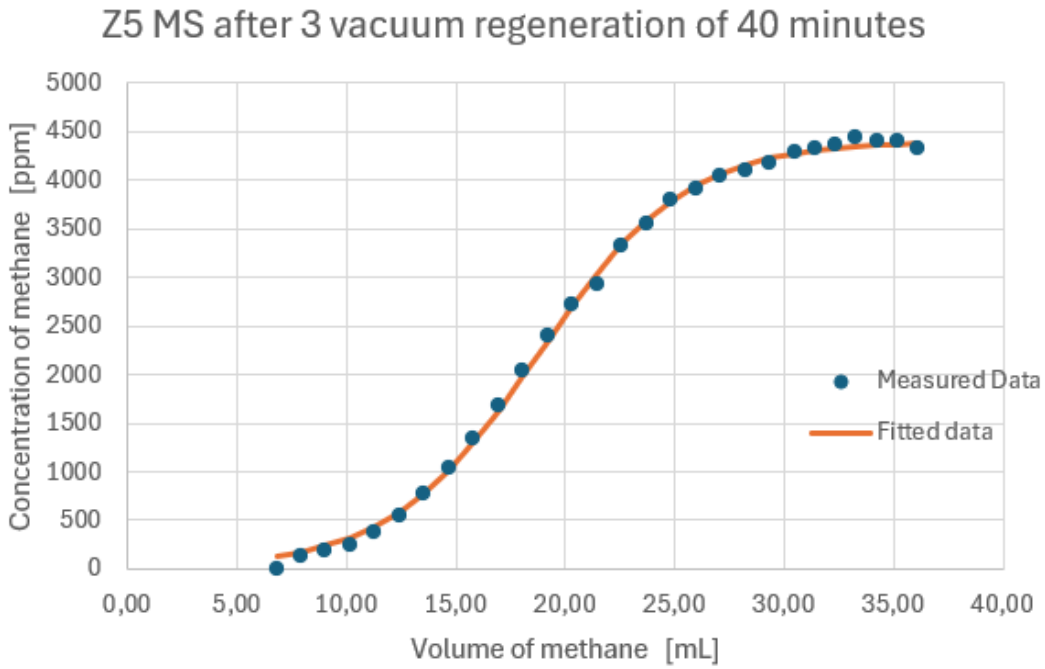
3° regeneration of 40 minutes:





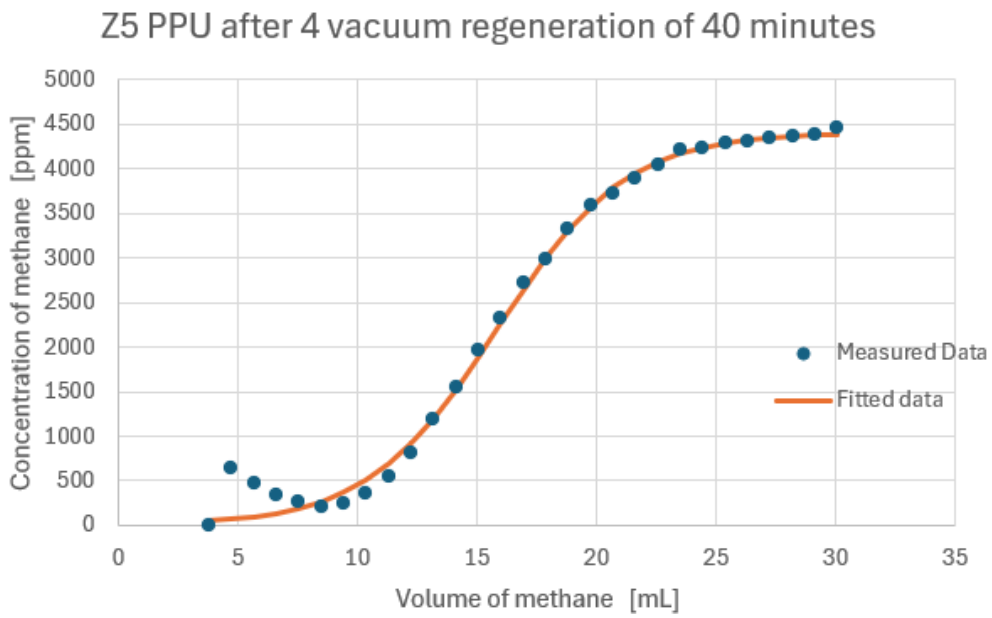
$x_{0\,PPU} = 18{,}774 \pm 0{,}005\ mL$

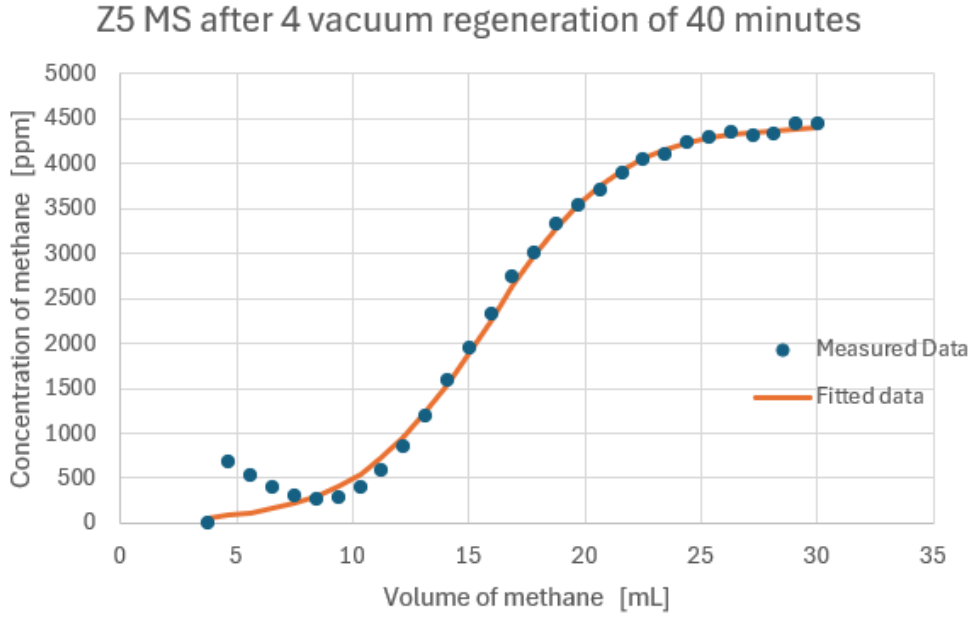
$x_{0\,MS} = 18{,}759 \pm 0{,}007\ mL$

$$FPA = 7{,}93\ mL$$

Z5 SHORT VACUUM REGENERATIONS

4° regeneration of 40 minutes:





$x_{0\,PPU} = 15{,}82 \pm 0{,}03\ mL$

$x_{0\,MS} = 15{,}82 \pm 0{,}04\ mL$

$$minimum = 9{,}40\ mL$$

Z5 SHORT VACUUM REGENERATIONS

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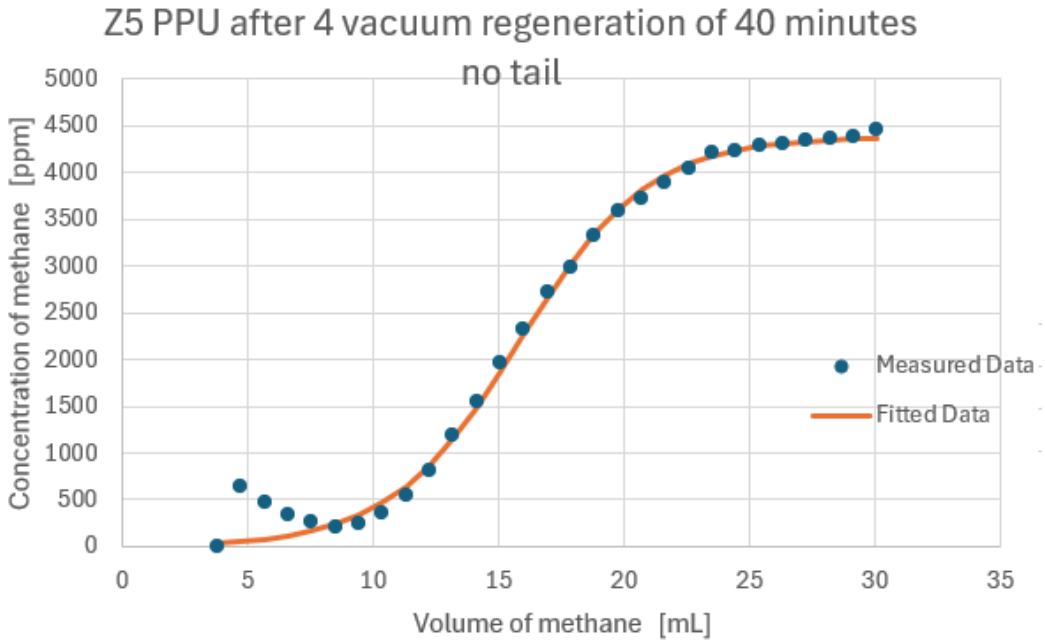
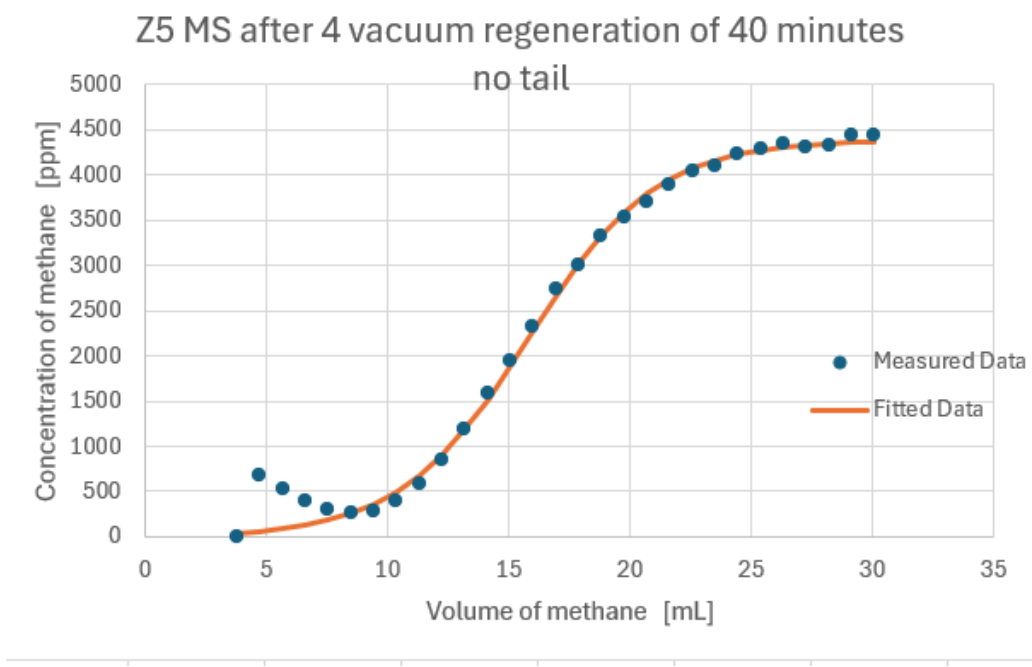
4° regeneration of 40 minutes **without tail**:





$x_{0\ PPU} = 15{,}798 \pm 0{,}006\ mL$

$x_{0\ MS} = 15{,}781 \pm 0{,}005\ mL$

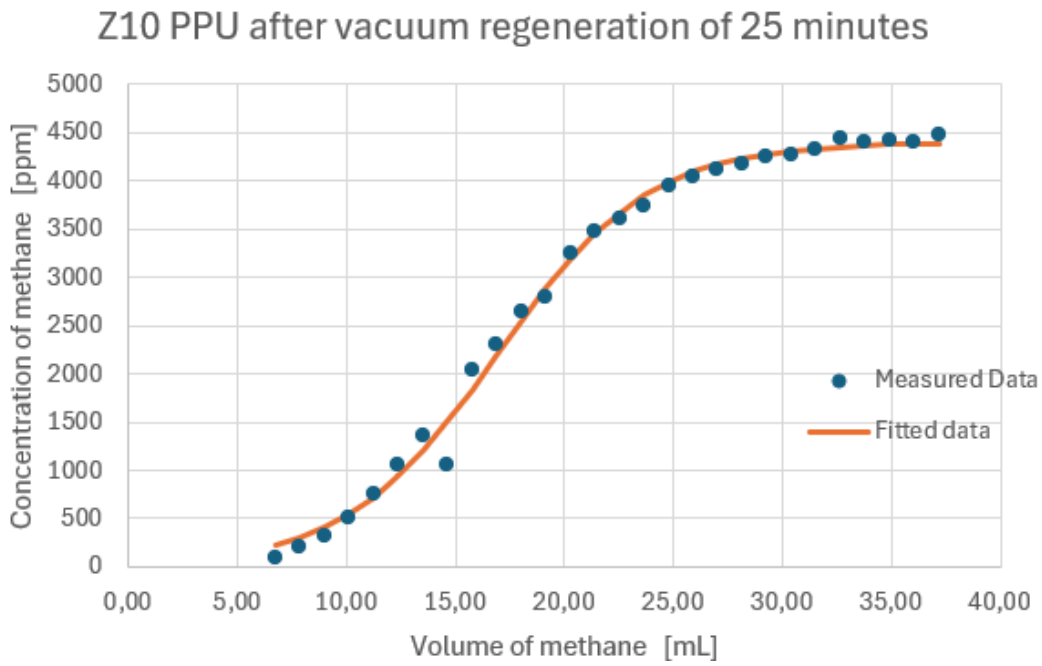
$$minimum = 9{,}40\ mL$$

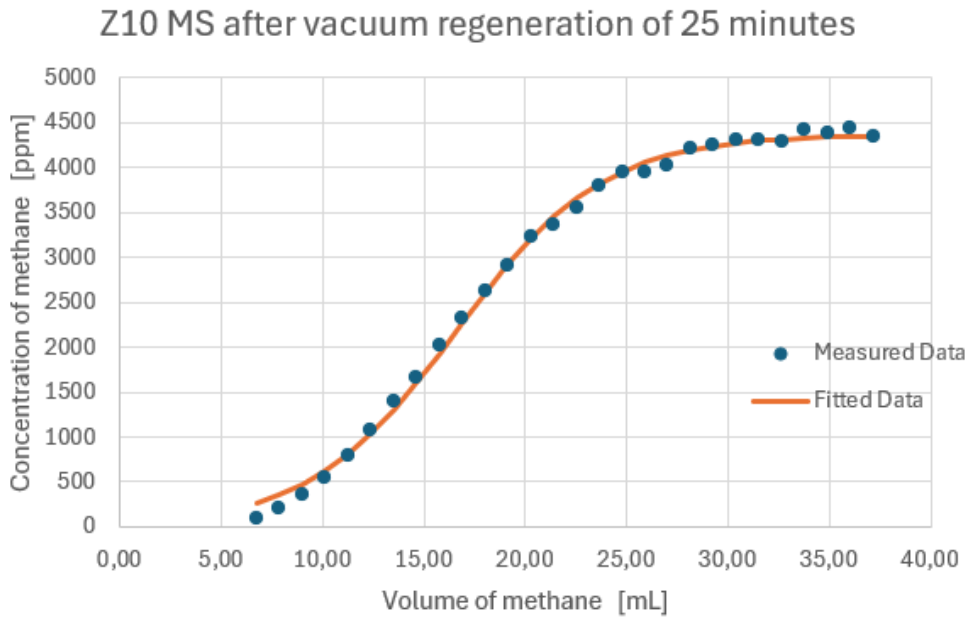
Z5 SHORT VACUUM REGENERATIONS

Regeneration	FPA / minimum	$x_{0\,PPU}$ [mL]	$x_{0\,MS}$ [mL]
1	7,20 mL	$x_0 = 15{,}53 \pm 0{,}06$	$x_0 = 16{,}21 \pm 0{,}05$
		$x_0 = 15{,}53 \pm 0{,}006$	$x_0 = 15{,}52 \pm 0{,}005$
2	10,01 mL	$x_{0\,PPU} = 16{,}214 \pm 0{,}014$	$x_{0\,MS} = 16{,}303 \pm 0{,}005$
3	7,93 mL	$x_{0\,PPU} = 18{,}774 \pm 0{,}005$	$x_{0\,MS} = 18{,}759 \pm 0{,}007$
4	9,40 mL	$x_{0\,PPU} = 15{,}82 \pm 0{,}03$	$x_{0\,MS} = 15{,}82 \pm 0{,}04$
		$x_{0\,PPU} = 15{,}798 \pm 0{,}006$	$x_{0\,MS} = 15{,}781 \pm 0{,}005$

Z10 SHORT VACUUM REGENERATIONS

1° regeneration, 25 minutes :





$x_{0\,PPU} = 16{,}92 \pm 0{,}03\ mL$

$x_{0\,MS} = 16{,}605 \pm 0{,}014\ mL$

$$FPA = 6{,}74\ mL$$

Z10 SHORT VACUUM REGENERATIONS

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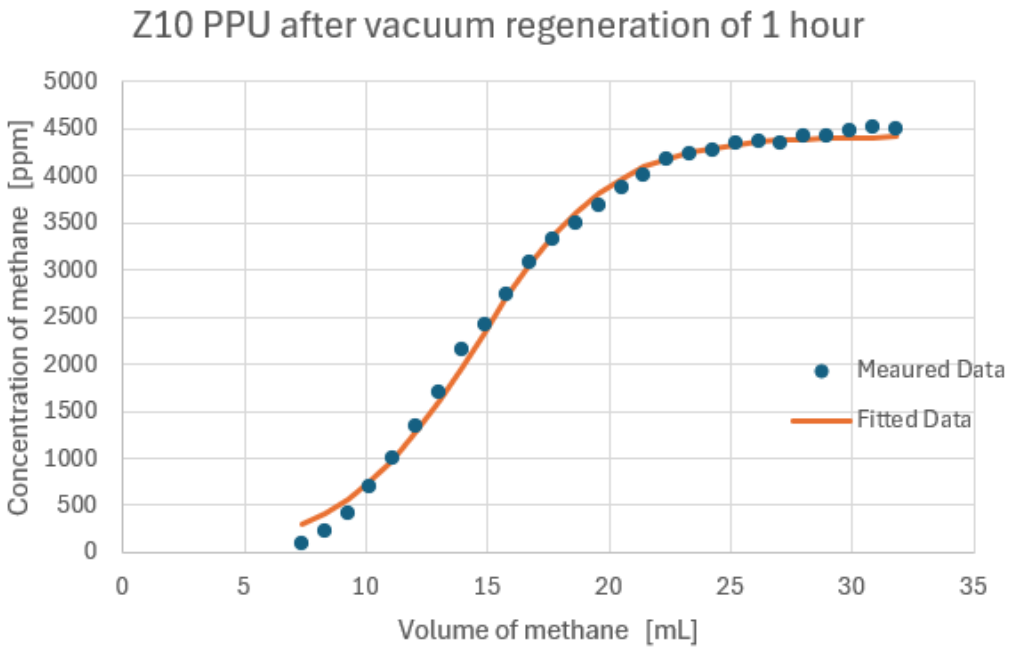
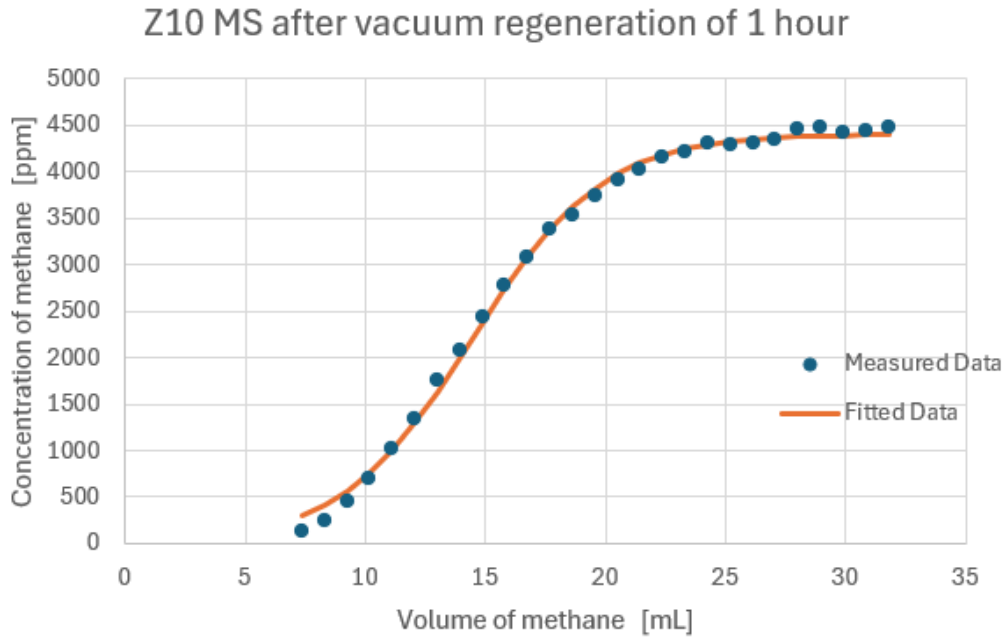
2° regeneration, 1 hour :





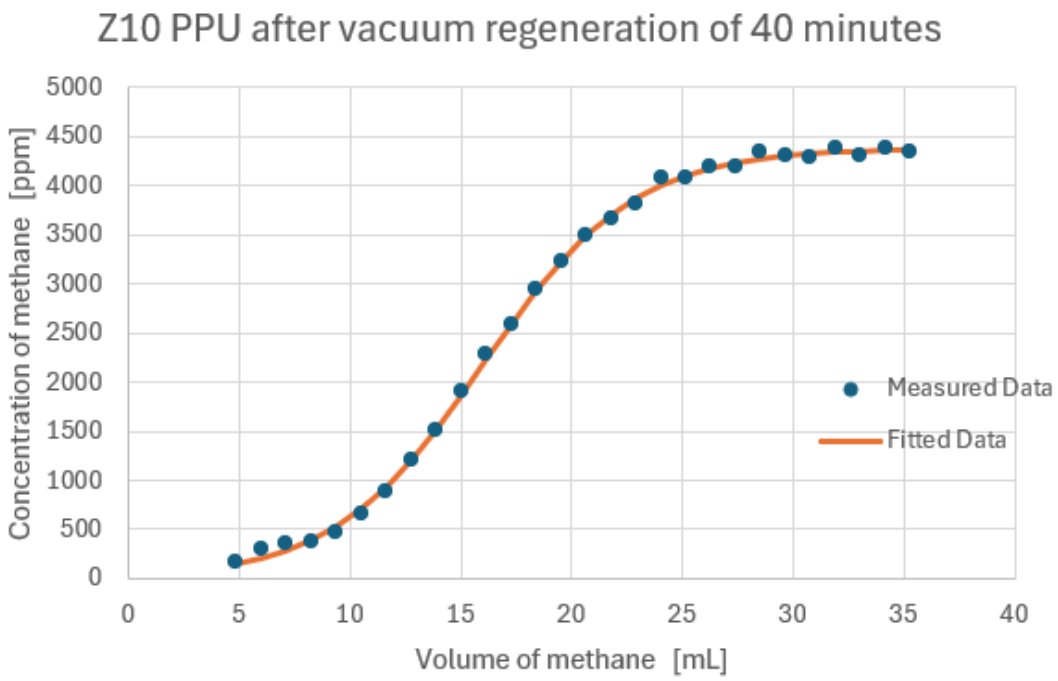
$x_{0\,PPU} = 14{,}549 \pm 0{,}011\ mL$

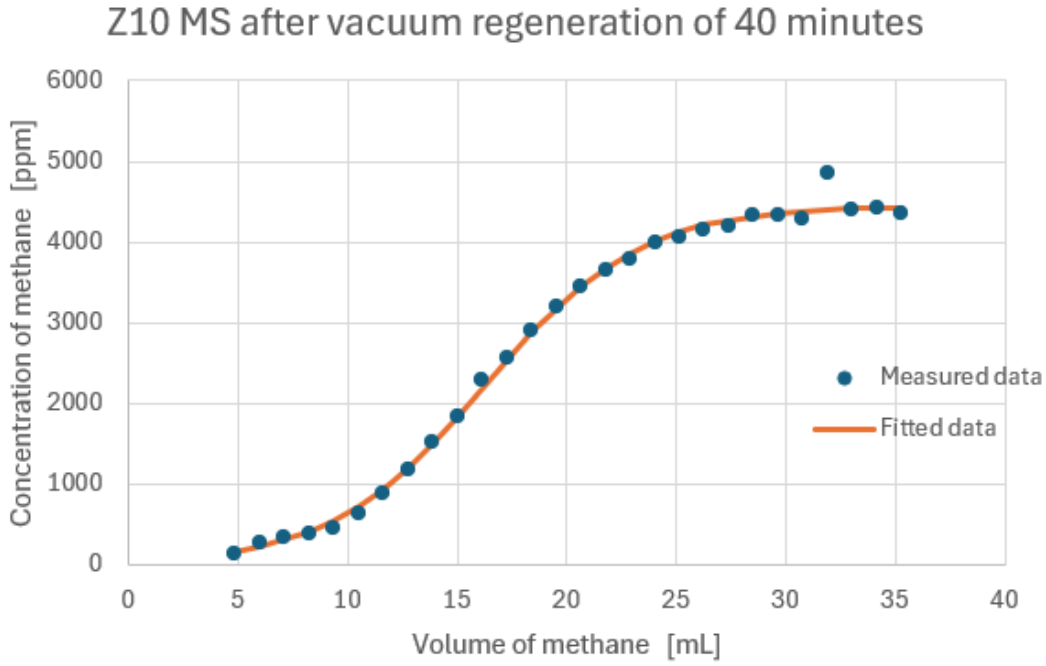
$x_{0\,MS} = 14{,}456 \pm 0{,}008\ mL$

$$FPA = 7{,}40\ mL$$

Z10 SHORT VACUUM REGENERATIONS

3° regeneration, 40 minutes :





$x_{0\,PPU} = 16{,}017 \pm 0{,}004\ mL$

$x_{0\,MS} = 16{,}281 \pm 0{,}023\ mL$

$FPA = 4{,}90\ mL$

Z10 SHORT VACUUM REGENERATIONS

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	Regeneration	FPA / minimum	$x_{0\,PPU}$ [mL]	$x_{0\,MS}$ [mL]
	1 (25 min)	6,74 mL	$x_{0\,PPU} = 16{,}92 \pm 0{,}03$	$x_{0\,MS} = 16{,}605 \pm 0{,}014$
*	2 (1 hour)	7,40 mL	$x_{0\,PPU} = 14{,}549 \pm 0{,}011$	$x_{0\,MS} = 14{,}456 \pm 0{,}008$
	3 (40 min)	4,90 mL	$x_{0\,PPU} = 16{,}017 \pm 0{,}004$	$x_{0\,MS} = 16{,}281 \pm 0{,}023$

* Longer regeneration time due to a leak found in the pressure gauge connection to the cartridge

COMBINED SHORT VACUUM REGENERATIONS

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	Regeneration	FPA / minimum	$x_{0\,PPU}$ [mL]	$x_{0\,MS}$ [mL]
Z5	1 (40 min)	7,20 mL	$x_0 = 15,53 \pm 0,06$	$x_0 = 16,21 \pm 0,05$
			$x_0 = 15,53 \pm 0,006$	$x_0 = 15,52 \pm 0,005$
	2 (40 min)	10,01 mL	$x_{0\,PPU} = 16,214 \pm 0,014$	$x_{0\,MS} = 16,303 \pm 0,005$
	3 (40 min)	7,93 mL	$x_{0\,PPU} = 18,774 \pm 0,005$	$x_{0\,MS} = 18,759 \pm 0,007$
	4 (40 min)	9,40 mL	$x_{0\,PPU} = 15,82 \pm 0,03$	$x_{0\,MS} = 15,82 \pm 0,04$
			$x_{0\,PPU} = 15,798 \pm 0,006$	$x_{0\,MS} = 15,781 \pm 0,005$
Z10	1 (25 min)	6,74 mL	$x_{0\,PPU} = 16,92 \pm 0,03$	$x_{0\,MS} = 16,605 \pm 0,014$
	2 (1 hour)	7,40 mL	$x_{0\,PPU} = 14,549 \pm 0,011$	$x_{0\,MS} = 14,456 \pm 0,008$
	3 (40 min)	4,90 mL	$x_{0\,PPU} = 16,017 \pm 0,004$	$x_{0\,MS} = 16,281 \pm 0,023$

SHORT VACUUM REGENERATION

With long vacuum regeneration:

- Z5 : seems to have the same performance

- Z10 : irregular behaviour, but efficiency drops.

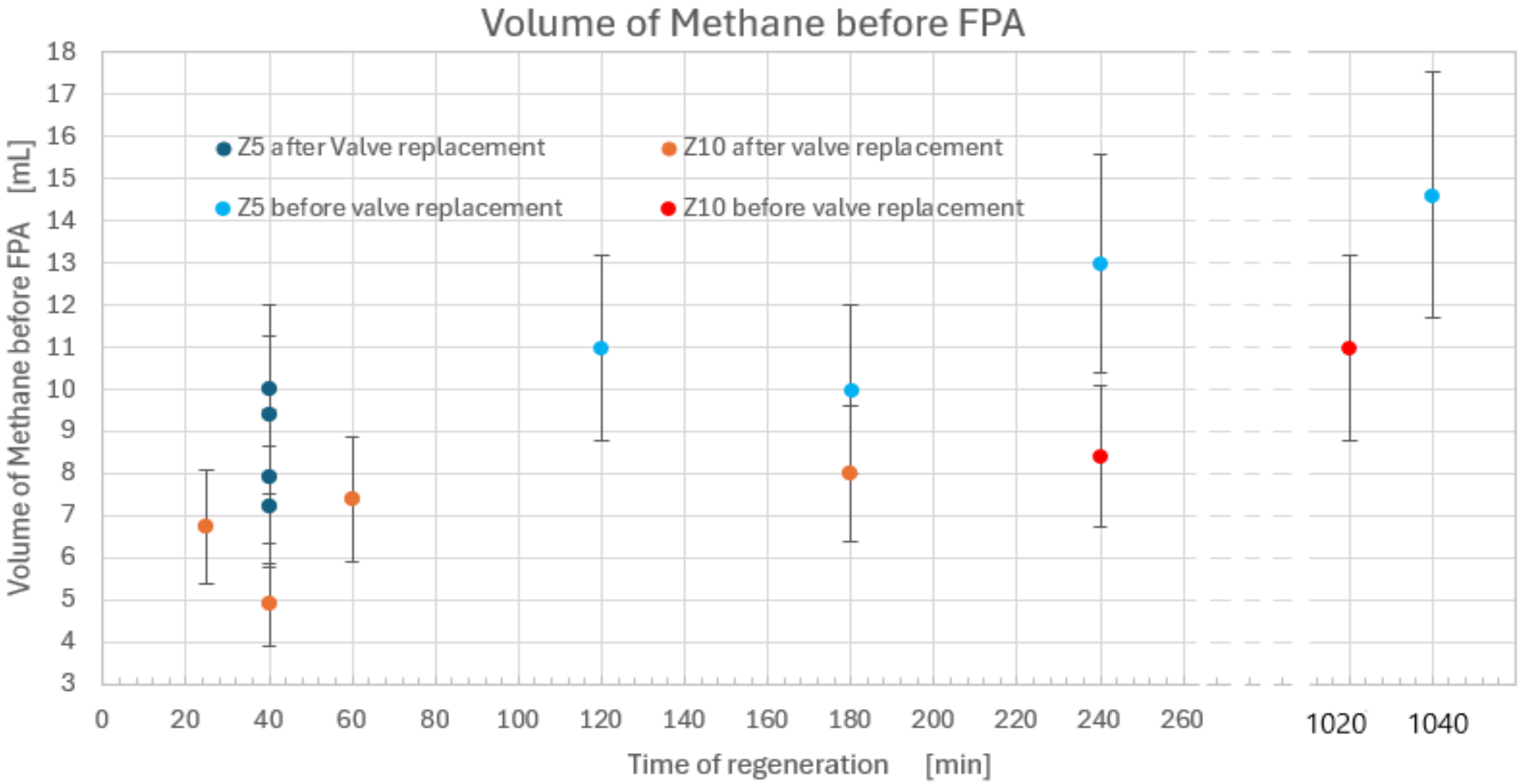
$$Average\ Z5\ short\ vacuum\ regeneration\ efficiency = \frac{8{,}64}{10{,}86} \approx 80\ \%$$

$$Average\ Z10\ short\ vacuum\ regeneration\ efficiency = \frac{6{,}35}{14{,}73} \approx 43\ \%$$

(10,86 mL and 14,73 mL are respectively the values of methane before first peak appearance in the chromatogram for Z5 and Z10 after full high temperature regeneration)

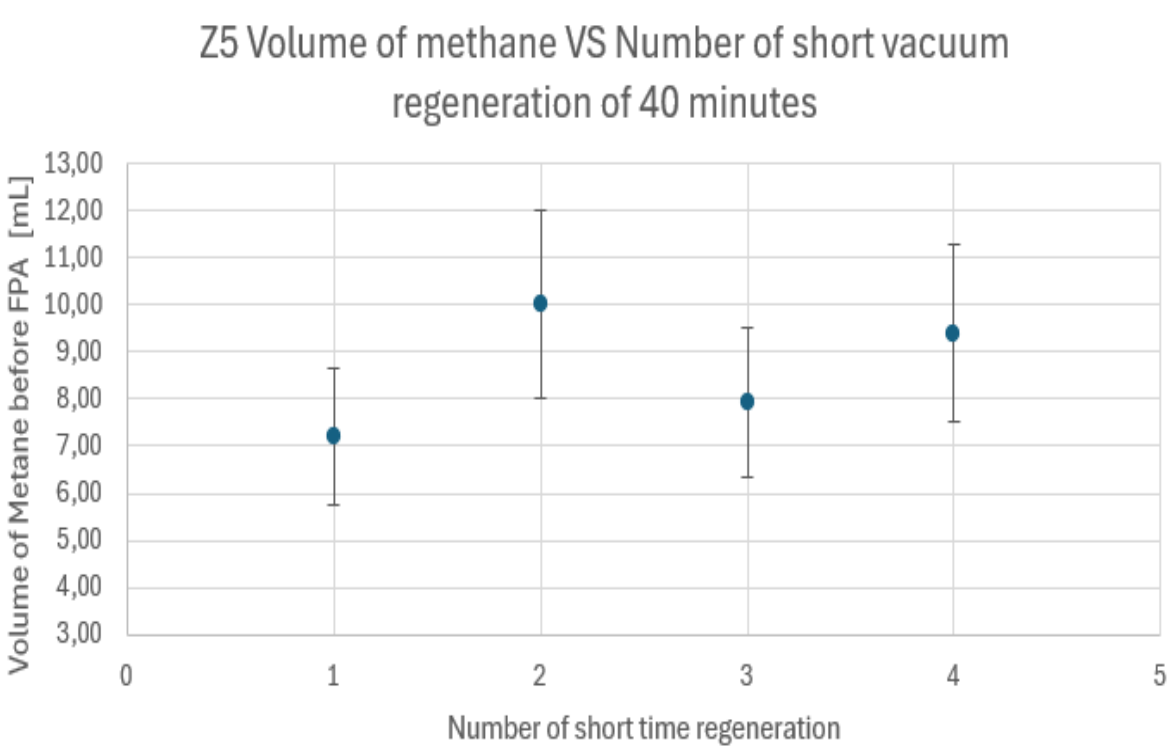
VACUUM REGENERATION REVIEW

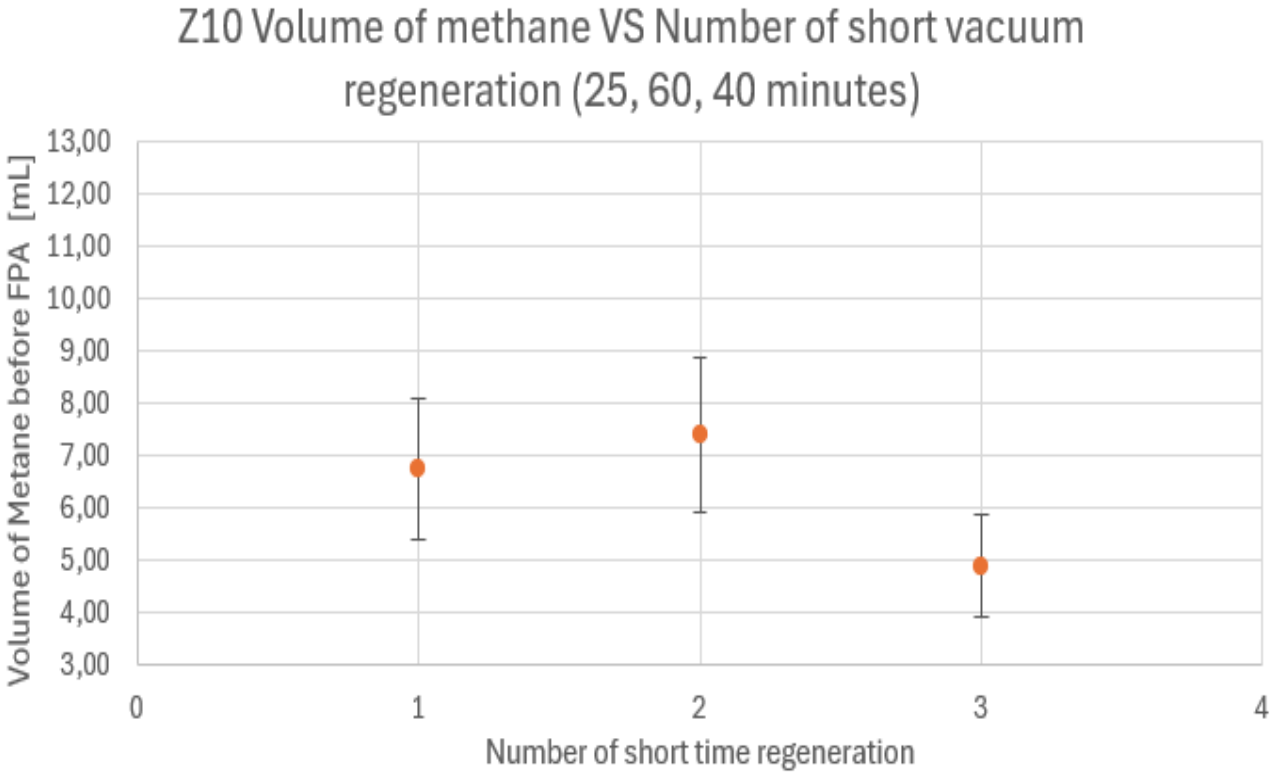
FPA = First Peak Appearance of CH4 in the chromatogram



VACUUM REGENERATION REVIEW

FPA = First Peak Appearance of CH4 in the chromatogram





VALVE REPLACEMENT

10/05: Replacement of one valve for both Z5 and Z10

Z5 : valve replaced between the 2 hours vacuum regeneration and the first 40 minutes vacuum regeneration

Z10: valve replaced between the 4 hours vacuum regeneration and the 3 hours vacuum regeneration