UNIVERSITÀ DI PAVIA

INFN

CERN

EP-DT
Detector Technologies

ZEOLITES TESTS

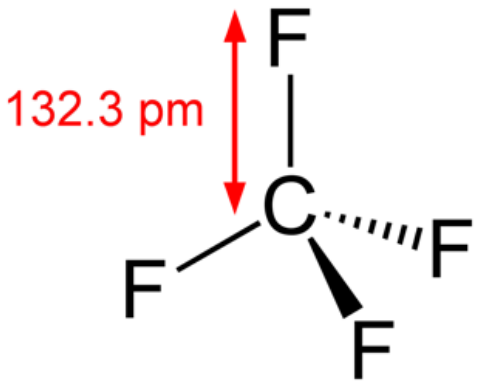
CH4rLiE Meeting 13/12/2024

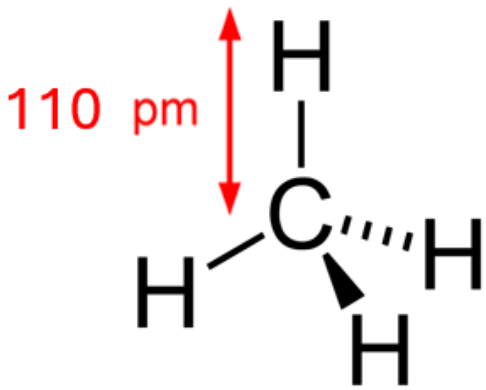
Francesco A. Angiulli, Maria Cristina Arena, Matteo Brunoldi, Simone Calzaferri, EP-DT Group

WHY ZEOLITES?

Zeolites are used at CERN to adsorb CF4

CF4 and CH4 are similar → same adsorbent as first trial step





PURPOSE

Evaluate which zeolite is the best at adsorbing CH4

4 possible Zeolites with different pores size:

- Z3 (0.3 nm) - Z4 (0.4 nm) - Z5 (0.5 nm) - Z10 (1.0 nm)

Evaluate best regeneration of material after adsorption:

- High Temperature
- Vacuum Pump

GAS CHROMATOGRAM ANALYSIS

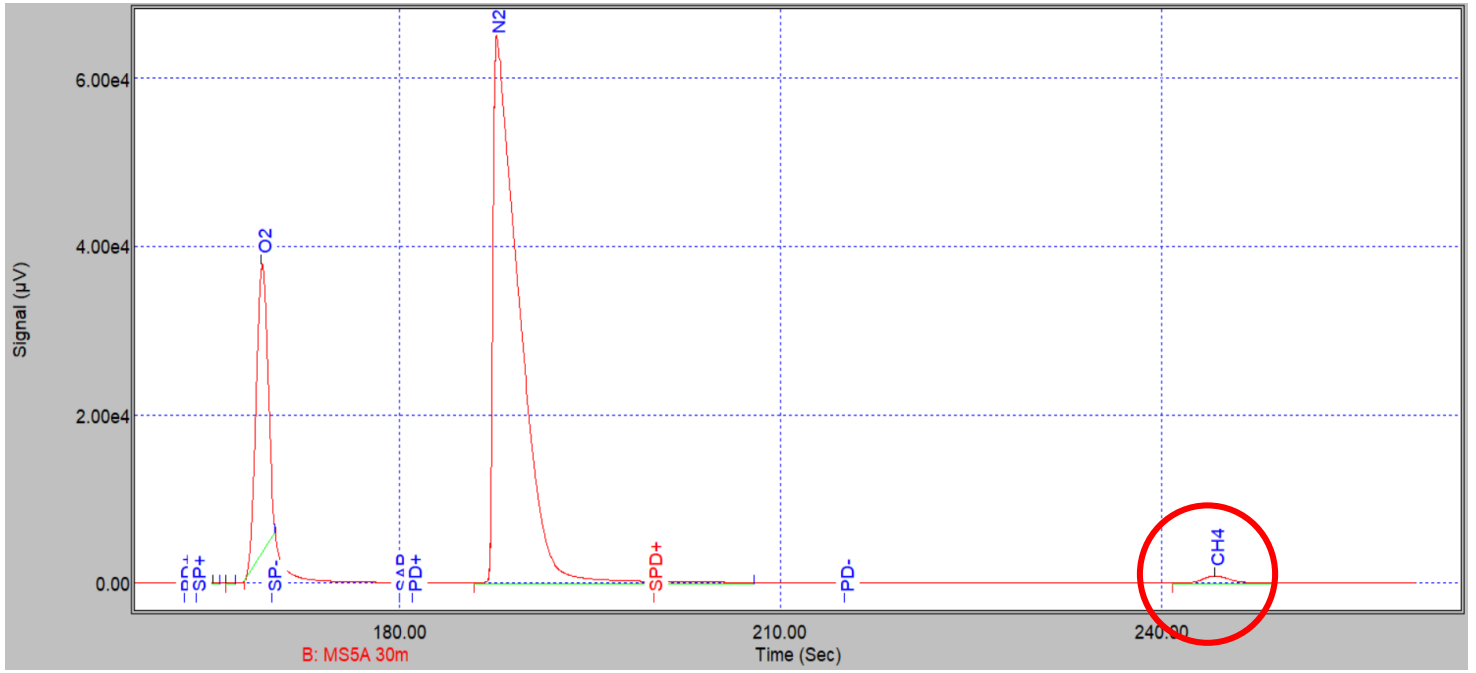
Gas Chromatograph:

Signal with peaks at different retention time for different gas components

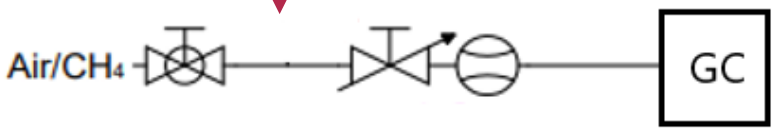
Integral of the peak is proportional to concentration of methane

RUN = Series of 5 minutes analysis

Output:



Calibration is required



$4509\ ppm$ in the bottle

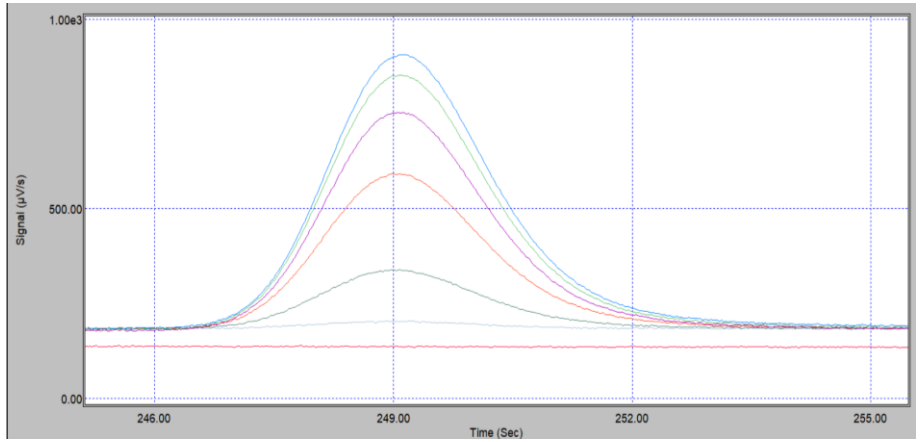
TEST

Idea of the test:

Air + CH4 (4509 ppm) → Adsorption [Zeolites] → Gas Chromatograph (**GC**) analysis of output mixture

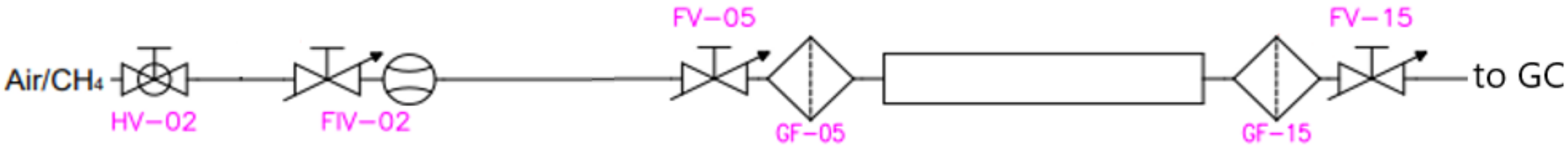
2 main phases:

- Zeolites adsorb all methane, there is no peak → **Total adsorption**, no methane escaping the cartridge
- Zeolites start to fill, methane survives → The **peak grows** until plateau saturation



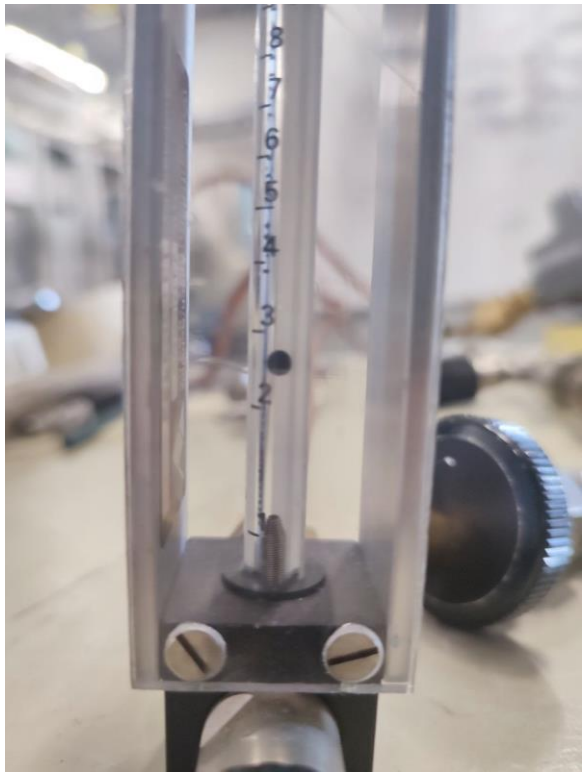
MAY MEASUREMENT SETUP

Setup during mission **29/04/24 – 13/05/24**:



Rotameter: range = $0 - 10\ L/h$ accuracy = $0.5\ L/h$

Average flow during analysis $2.5\ L/h$

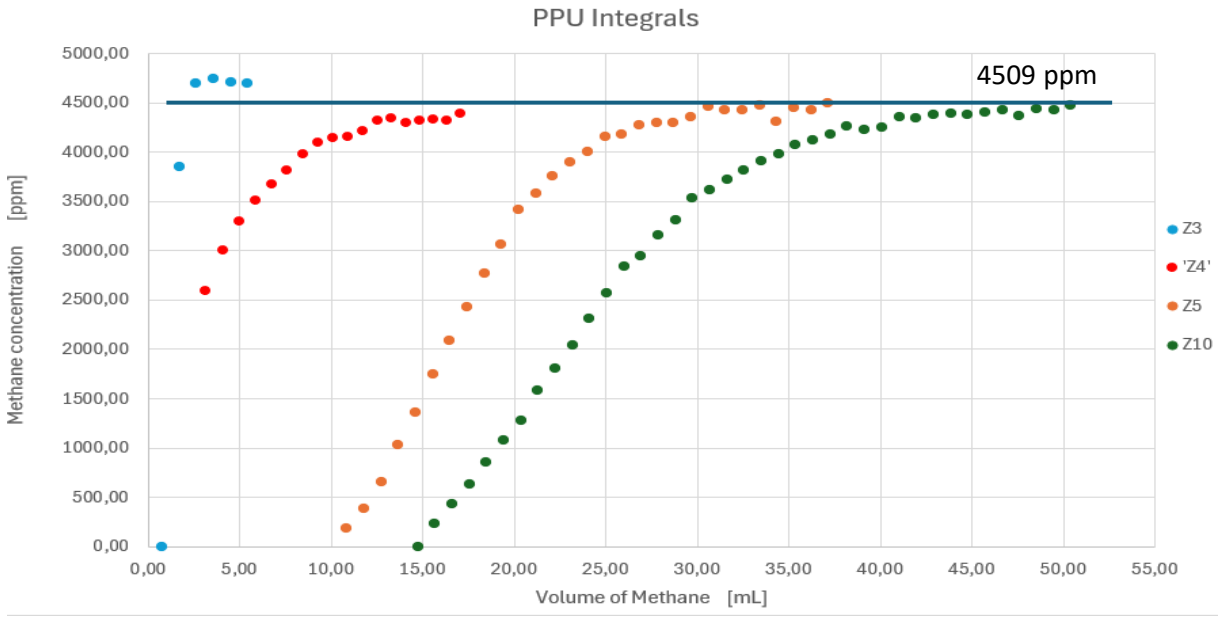


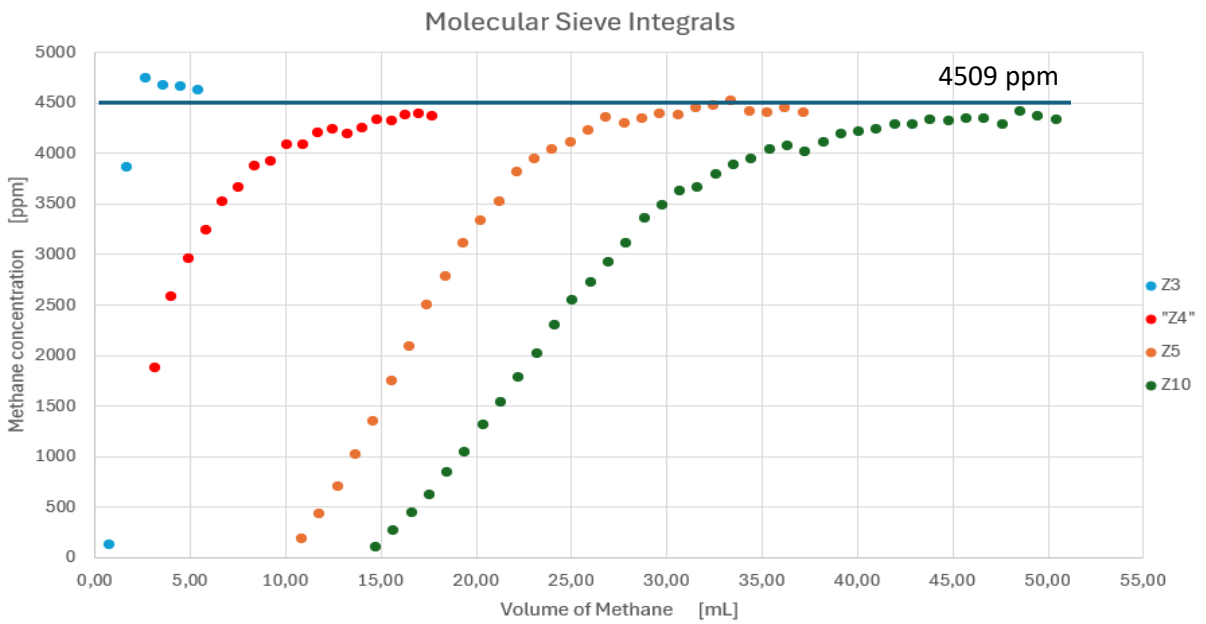
SATURATION CURVES

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

High temperature regenerated cartridges:





Adsorbed methan before peak appearance in the chromatogram:

$Z3 = 0.75\,mL$ $Z4 = 3.13\,mL$ $Z5 = 10.70\,mL$ $Z10 = 14.73\,mL$

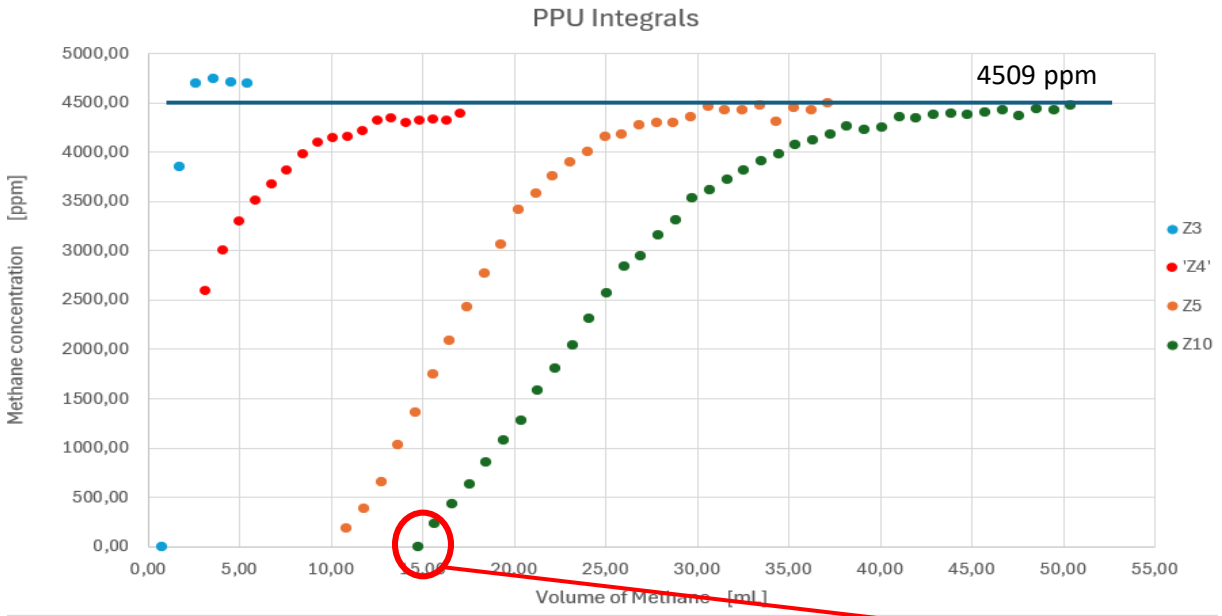
Overall error of 20% due to rotameter measures

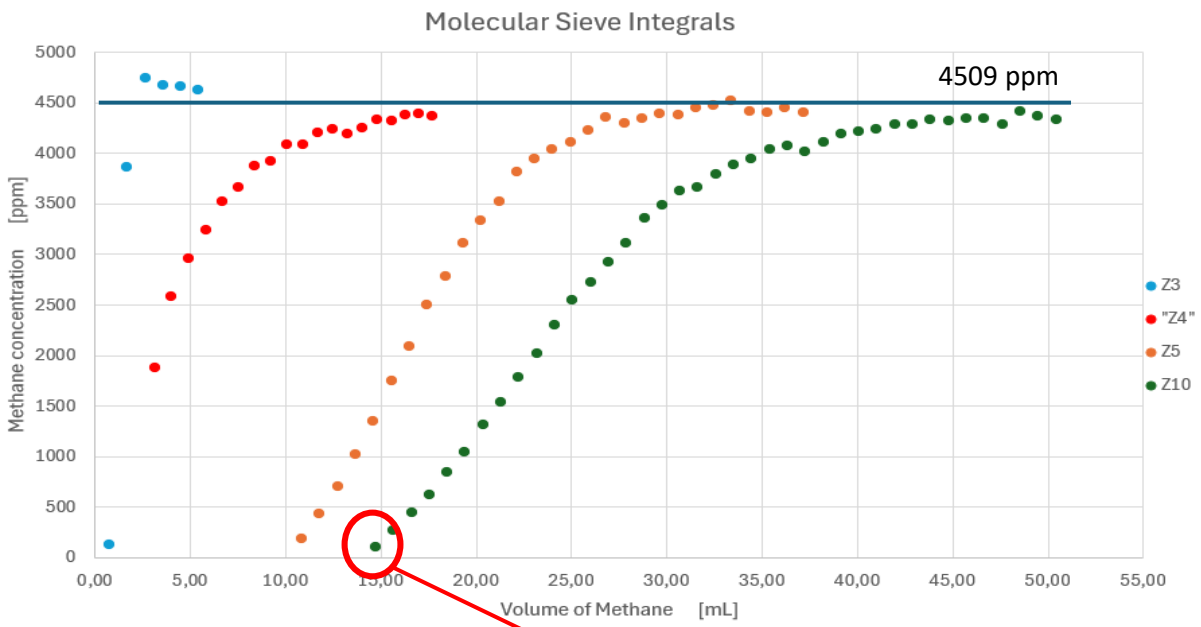
SATURATION CURVES

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

High temperature regenerated cartridges:





Adsorbed methan before peak appearance in the chromatogram:

$Z3 = 0.75\,mL$ $Z4 = 3.13\,mL$ $Z5 = 10.70\,mL$ $Z10 = 14.73\,mL$

Overall error of 20% due to rotameter measures

FIRST RESULTS 05/24

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

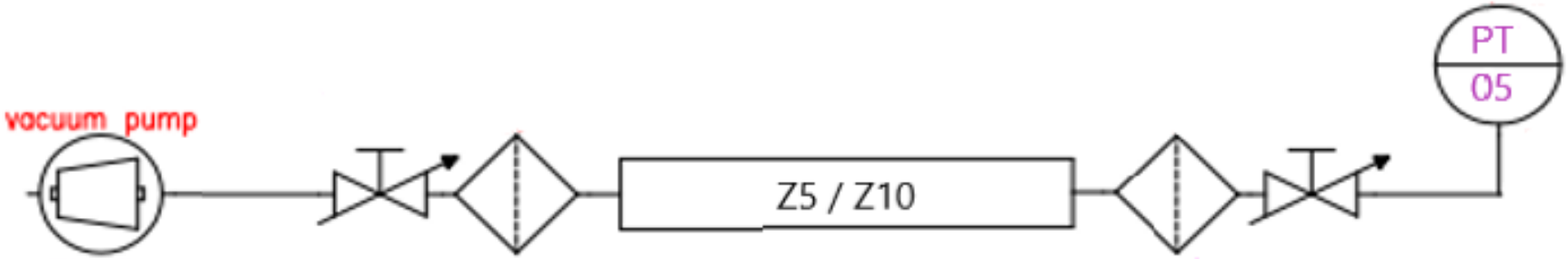
Z3, Z4 excluded from further measurements with CH4

Z3 has been used later as adsorbent of H2O

NEXT STEP:

Cycles with **Vacuum pump regeneration** trying different regeneration time for Z5/Z10 to evaluate regeneration efficiency

Setup for regeneration:



FIRST RESULTS 05/24

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Regeneration	Time of regeneration	Number of RUNS		Mean CH4 Adsorbed Z5	Mean CH4 Adsorbed Z10	Efficiency * Z5	Efficiency * Z10
High Temperature		**1**	Before first peak	10.0 ± 2.0 mL	14.5 ± 2.9 mL	100 %	100 %
			At saturation	17.6 ± 2.6 mL	24.6 ± 4.9 mL	100 %	100 %
Vacuum	LONG (> 2 hours) **	**3**	Before first peak	11.1 ± 2.2 mL	9.1 ± 1.8 mL	111 %	62.7 %
			At saturation	18.7 ±3.7 mL	17.6 ± 3.5 mL	105 %	91.1 %
	SHORT (~ 40 min)	**4**	Before first peak	8.3 ± 1,7 mL	6.3 ± 1.3 mL	83.1 %	43.6 %
			At saturation	17.0 ± 3,4 mL	15.9 ± 3.2 mL	95.5 %	66.2 %

* Efficiency is calculated with respect to the High Temperature regeneration results

** Overestimated flux due to inclined rotameter

FIRST RESULTS 05/24

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Regeneration	Time of regeneration	Number of RUNS		Mean CH4 Adsorbed Z5	Mean CH4 Adsorbed Z10	Efficiency * Z5	Efficiency * Z10
High Temperature		**1**	Before first peak	10.0 ± 2.0 mL	14.5 ± 2.9 mL	100 %	100 %
			At saturation	17.6 ± 2.6 mL	24.6 ± 4.9 mL	100 %	100 %
Vacuum	LONG (> 2 hours) **	**3**	Before first peak	11.1 ± 2.2 mL	9.1 ± 1.8 mL	111 %	62.7 %
			At saturation	18.7 ±3.7 mL	17.6 ± 3.5 mL	105 %	91.1 %
	SHORT (~ 40 min)	**4**	Before first peak	8.3 ± 1,7 mL	6.3 ± 1.3 mL	83.1 %	43.6 %
			At saturation	17.0 ± 3,4 mL	15.9 ± 3.2 mL	95.5 %	66.2 %

* Efficiency is calculated with respect to the High Temperature regeneration results

** Overestimated flux due to inclined rotameter

29/04/24 – 13/05/24 RESULTS

UNIVERSITÀ DI PAVIA

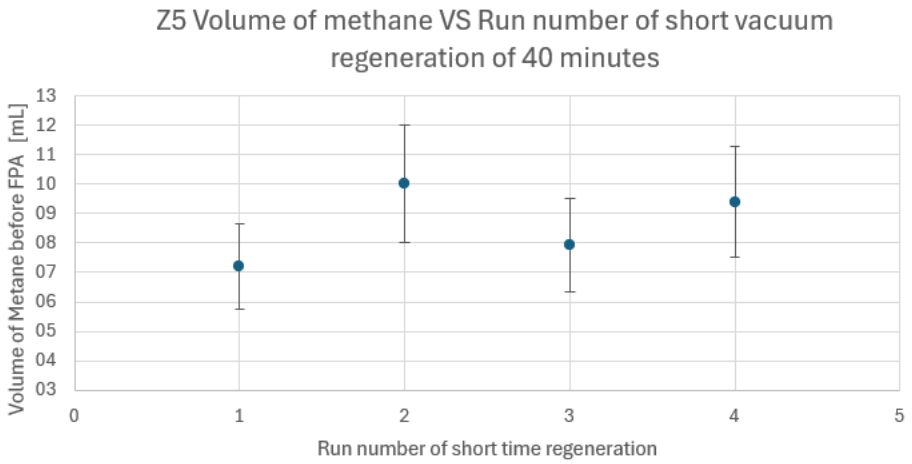
INFN CERN EP-DT Detector Technologies

Regeneration	Time of regeneration		Mean CH4 Adsorbed		Efficiency *	
			Z5	Z10	Z5	Z10
Vacuum	SHORT (~ 40 min)	Before first peak	8.3 ± 1,7 mL	6.3 ± 1.3 mL	83.1 %	43.6 %
		At saturation	17.0 ± 3,4 mL	15.9 ± 3.2 mL	95.5 %	66.2 %

- Z5 : the ratio is a little lower, but consistent **95.5 %** regeneration on total adsorbtion

- Z10 : irregular behaviour, the regeneration ratio

⇒ **Z5** chosen for further measurements



OSS:

All values have 20 % error due to rotameter resolution

03/11/24 – 24/11/24 MISSION

PURPOSE:

1. Confirm vacuum regeneration efficiency for **Z5**

   → Saturation cycles with 40 minutes vacuum pump regeneration

2. Test using Zeolites as humidity adsorbent

   → humidity adsorbed by **Z3** before gas enters the cartridge with **Z5**

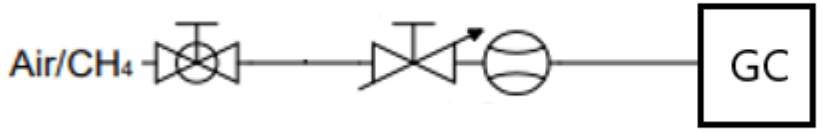
GC CLIBRATION FOR 4509 ppm METHANE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

NEW CALIBRATION:

Bottle of Air ($N_2 + O_2$) and Methane at 4509 ppm directly connected to Gas Chromatograph (GC)



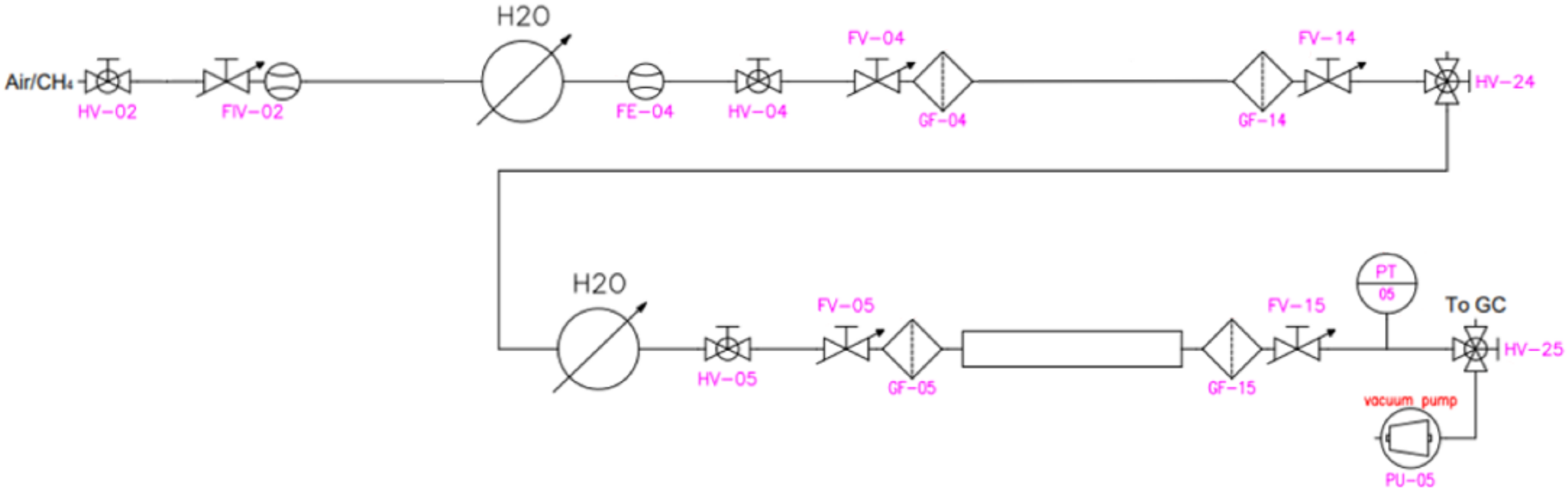
Bronkhorst pressure = 100 mbar

	PPU AREA	PPU to ppm CONVERSION FACTOR		MS AREA	MS to ppm CONVERSION FACTOR
AVERAGE	5149	0,874	AVERAGE	1958	2,304
DEV. STD	27	0,004	DEV. STD	13	0,017
AVG DEV. STD	10	0,002	AVG DEV. STD	5	0,006

FIRST MEASUREMENT SETUP

UNIVERSITÀ DI PAVIA
INFN CERN EP-DT Detector Technologies

Setup with **new sensors** to control humidity, pressure and flow



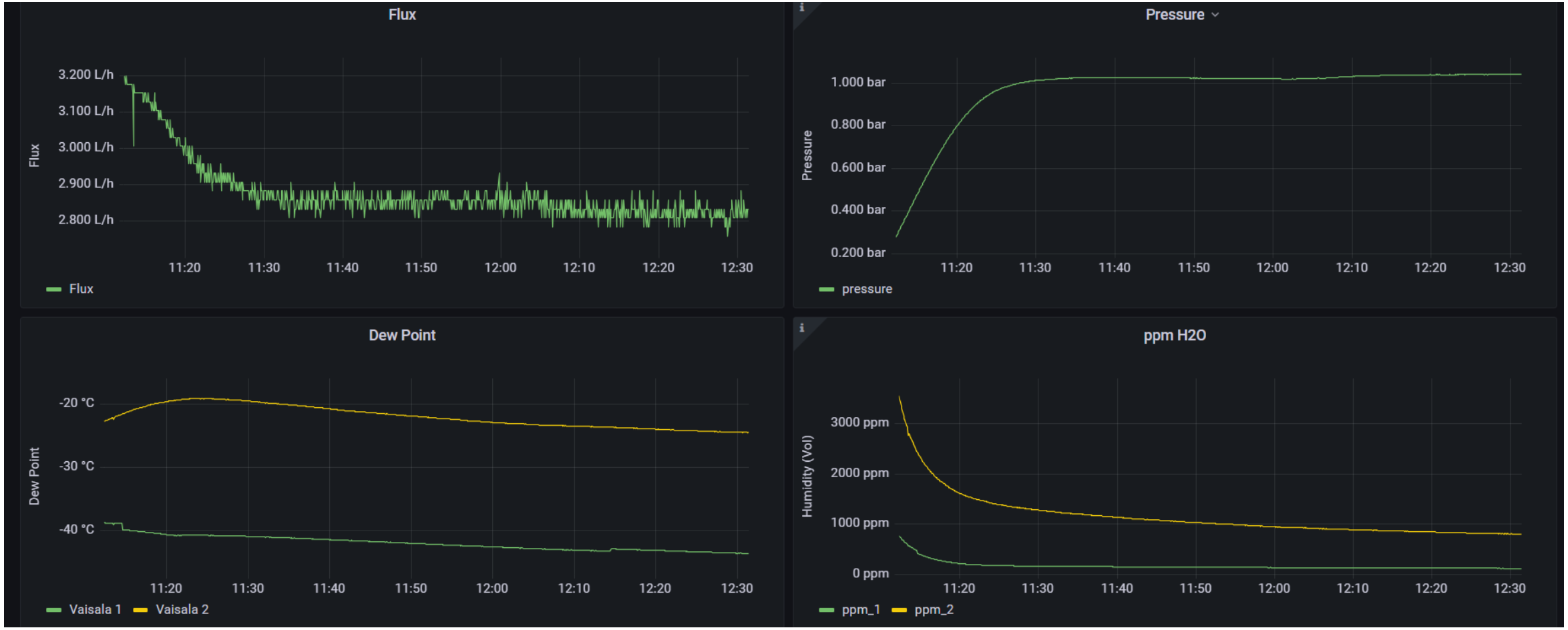
FIRST MEASUREMENT

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

10 cycles with the cartridge of Z5

Data during measurement:



FIRST MEASUREMENT

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

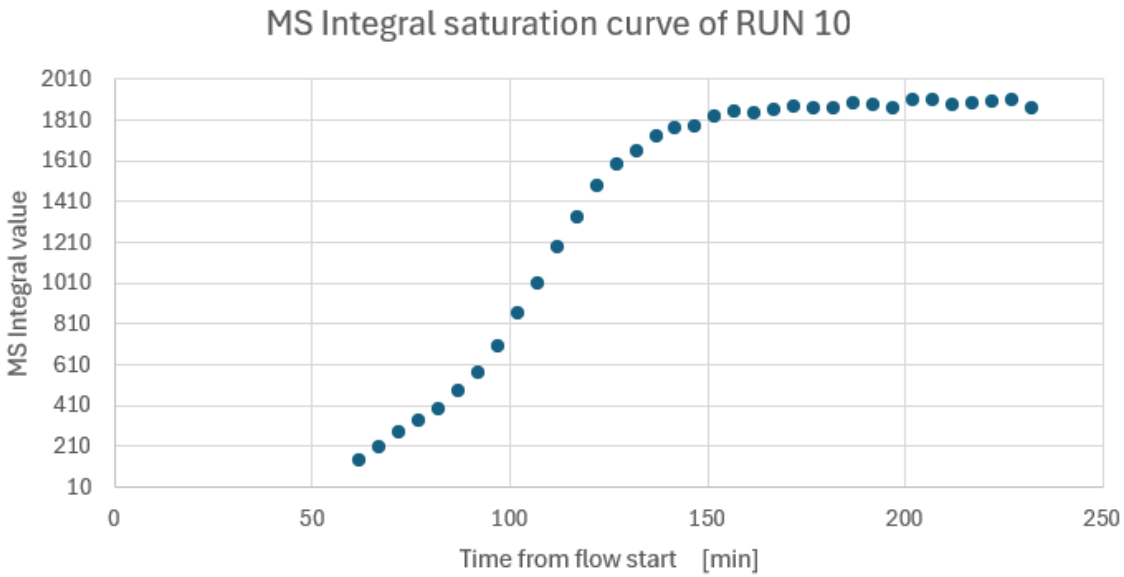
RUN = CH4 flows through cartridge filled with Z5 until saturation

Saturation = Integral of GC stops rising

Vacuum Regeneration Time = 40 min

RUN 3-10 with flow $\simeq 2.7 \div 3 \ \frac{L}{h}$

RUN 1 and 2 flow higher than 3 L/h and pressure over sensor range

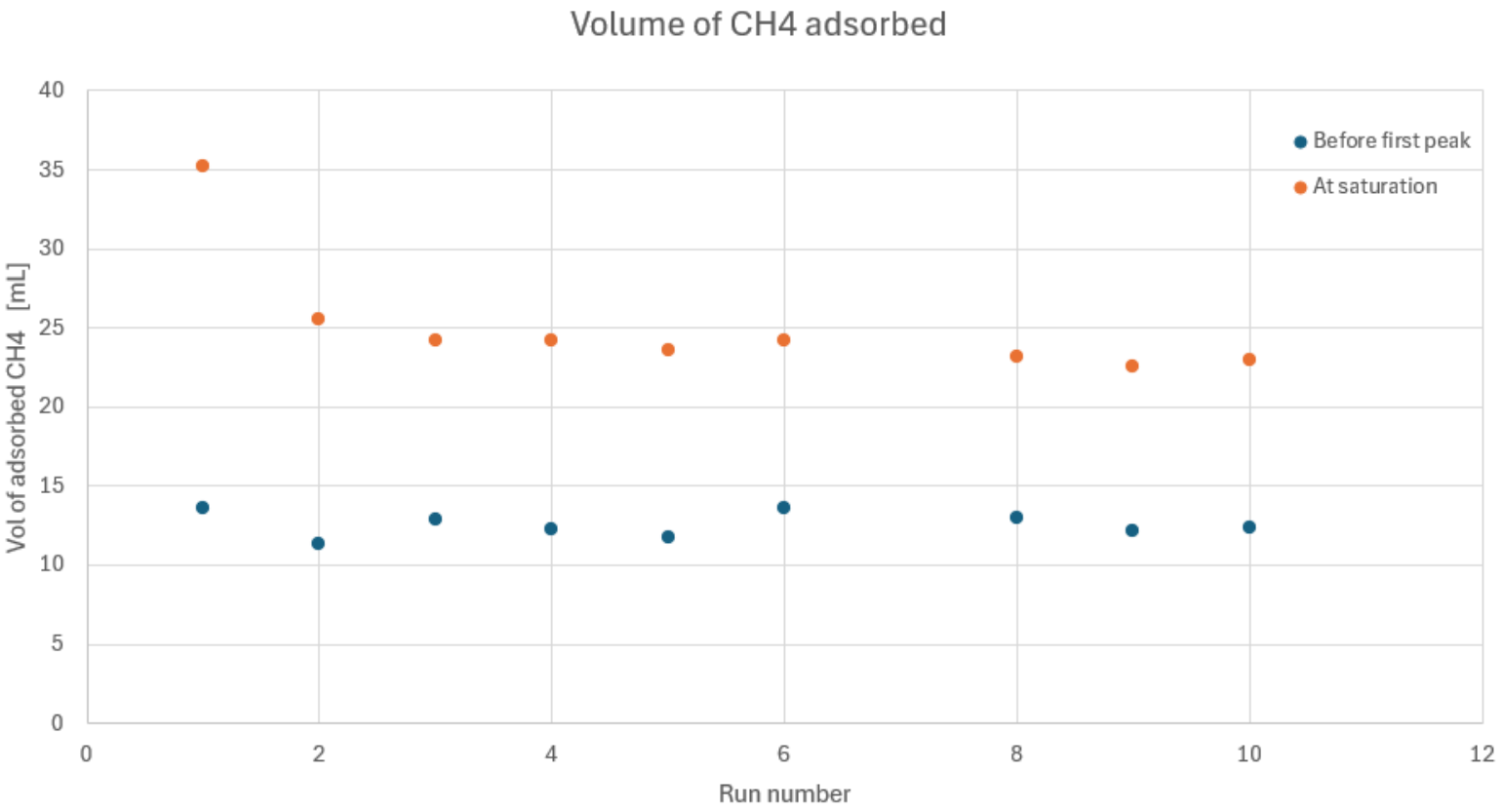


CH4 adsorbed	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6	RUN 8	RUN 9	RUN10
Before first peak	13.56	11.29	12.88	12.25	11.77	13.61	12.97	12.19	12.32
At saturation	35.20	25.53	24.17	24.22	23.60	24.21	23.21	23.13	22.98

PRELIMINARY RESULTS

RUN 7 has no sensors data due to raspberry problems

Results after 10 runs:



Mean Volume of CH4 adsorbed:

- at saturation:

$$23.6 \pm 0.6\ mL$$

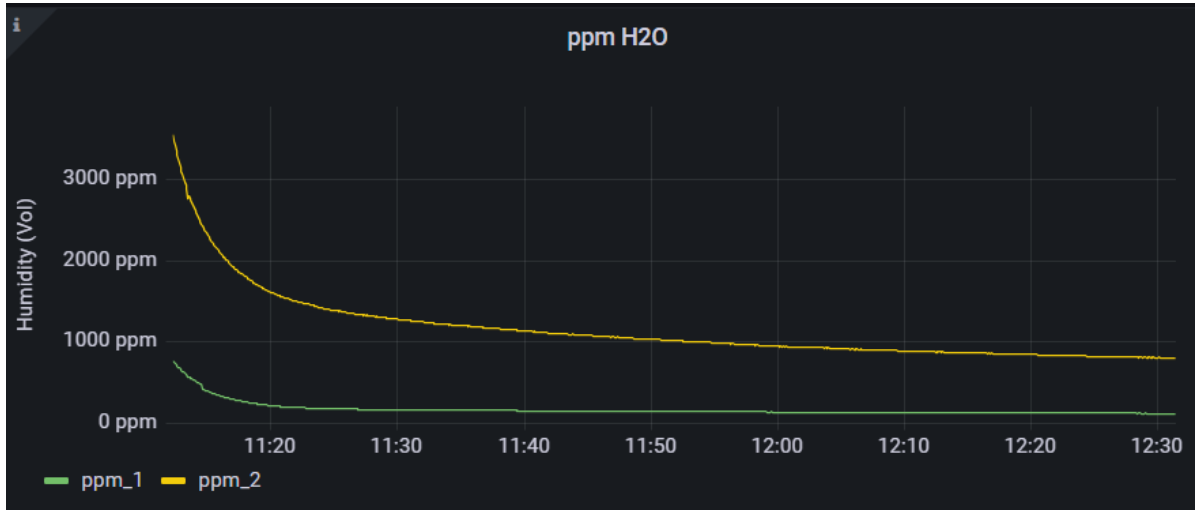
- before first peak:

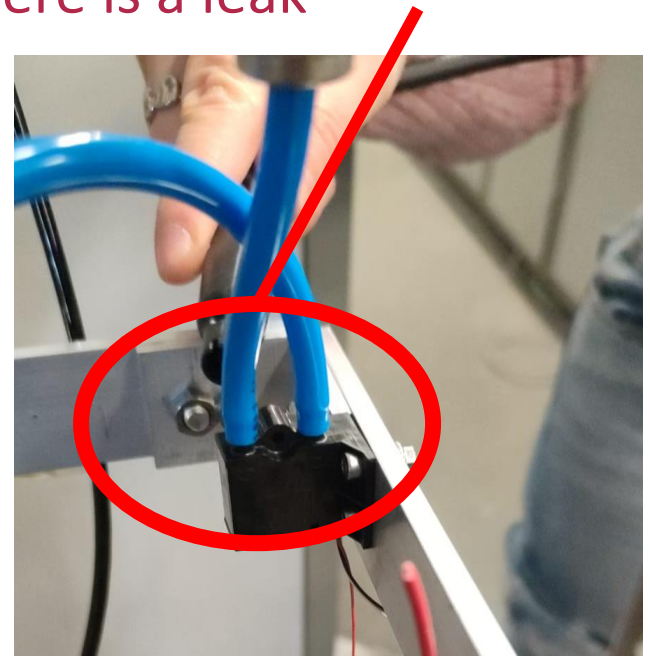
$$12.6 \pm 0.6\ mL$$

MEASUREMENT

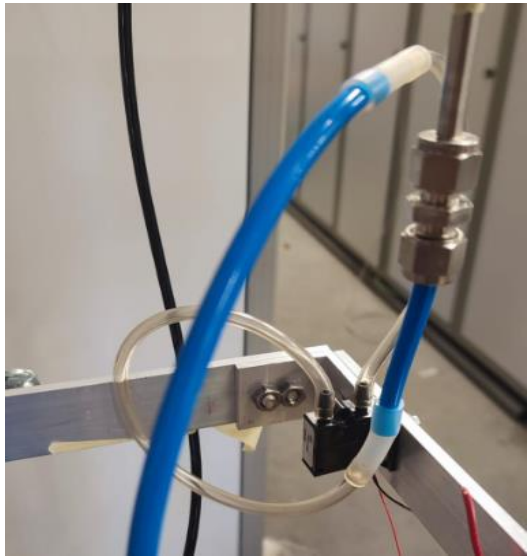
10 cycles with the cartridge of Z5

Vaisala 2 reads more humidity than Vaisala 1 → there is a leak



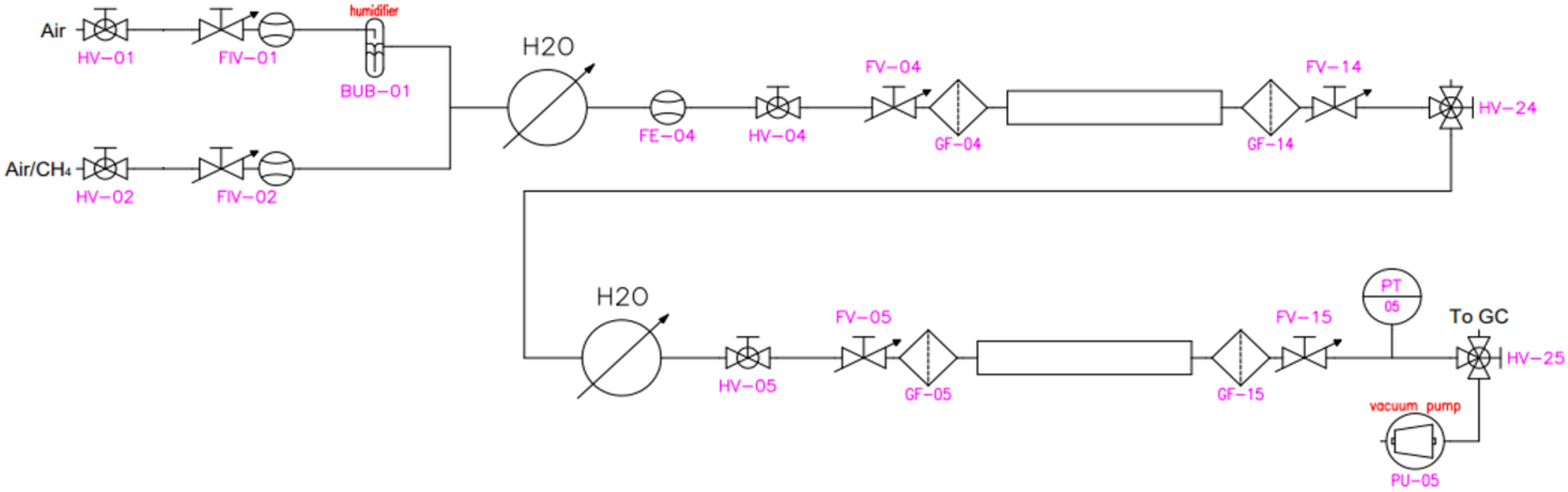


After 10 runs we changed plastic pipes:



SECOND MEASUREMENT SETUP

Setup for second week:



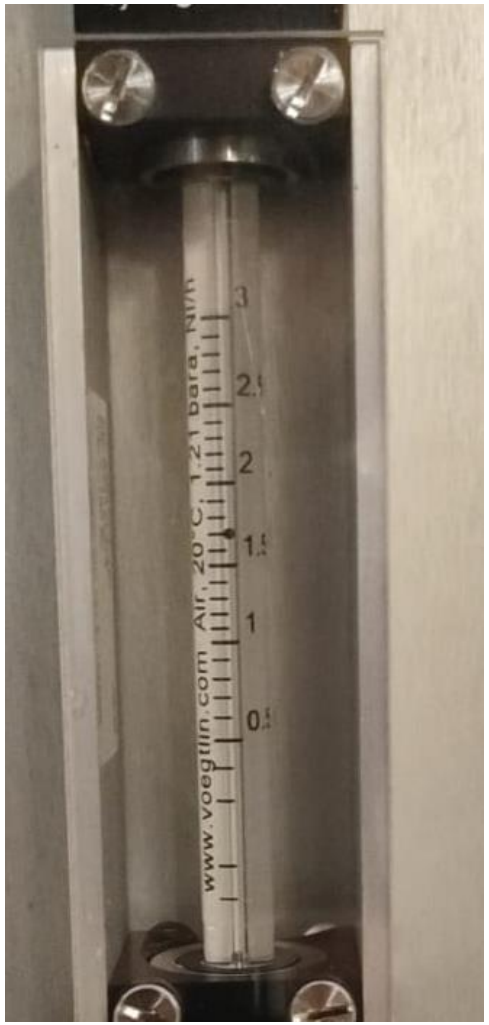
SECOND MEASUREMENT SETUP

UNIVERSITÀ DI PAVIA
INFN CERN EP-DT Detector Technologies

GOAL:

Measurements of adsorption with CH4 diluted with mixture of Air + H2O

Ratio of diluted CH4 set using rotameters at 50 % with respect to the CH4 bottle at 4509 ppm

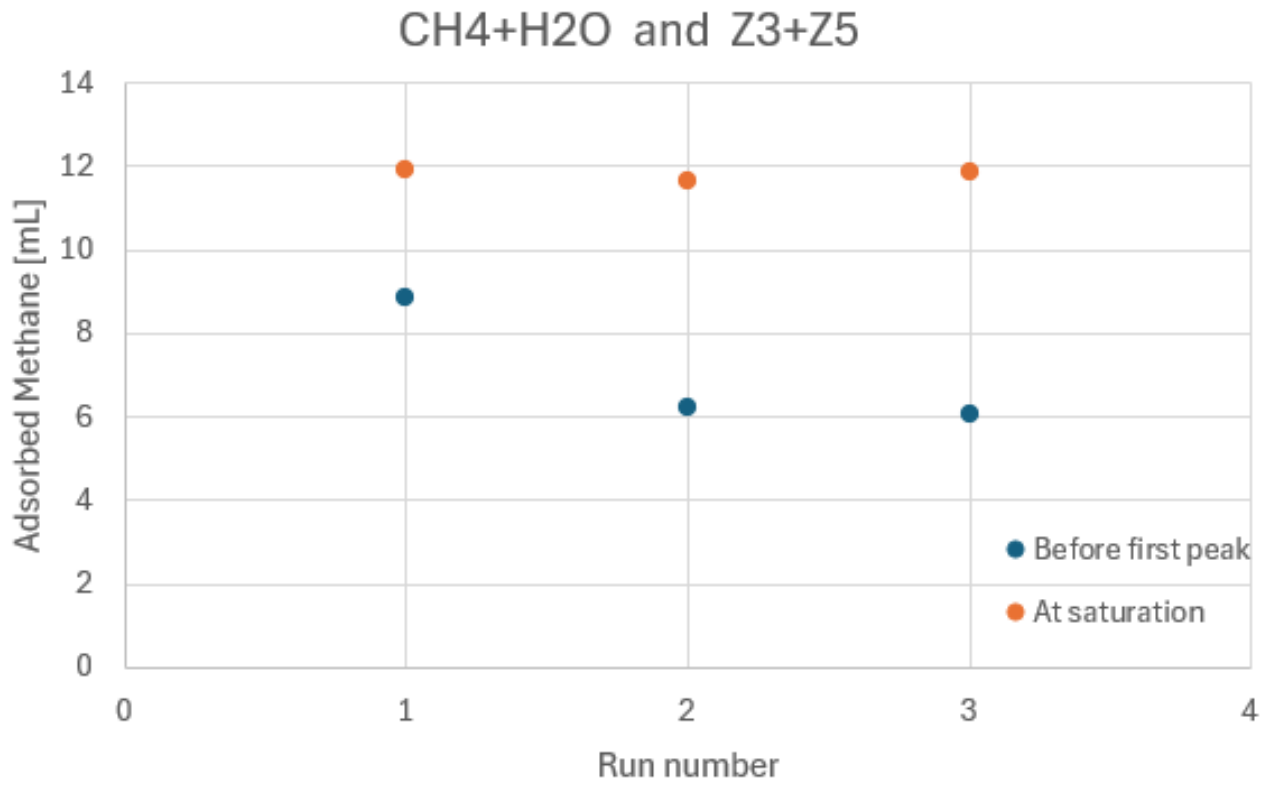


Air + H20

Air+CH4

SECOND MEASUREMENT

First results:



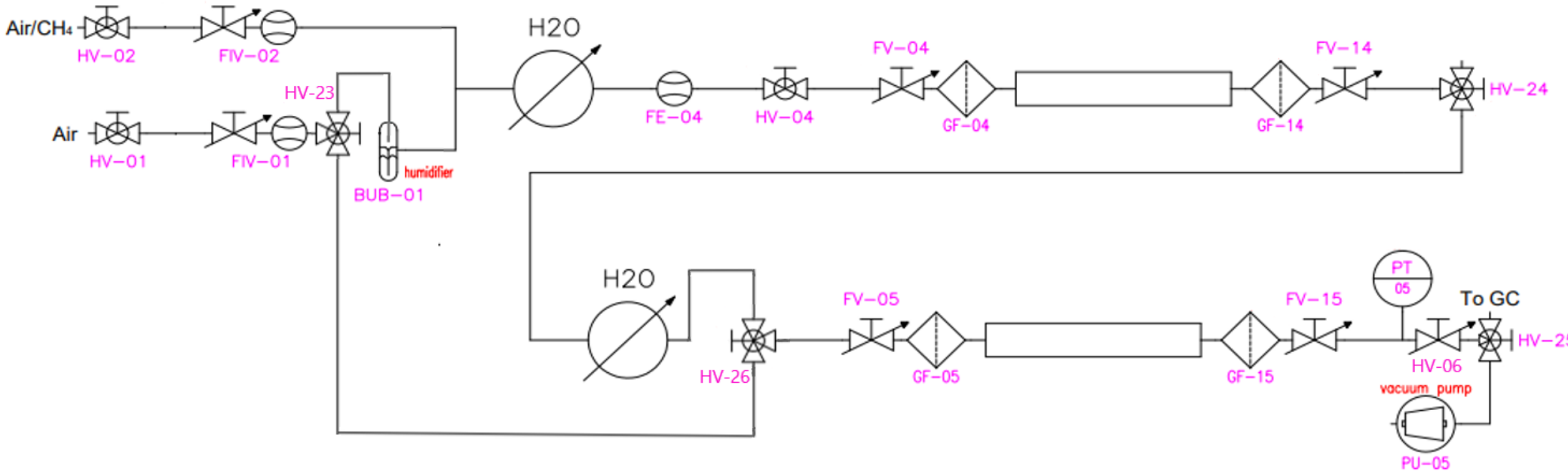
Unexpected beaviour → SOLUTION IMPLEMENTED: High temperature regeneration and bypass line after vacuum regeneration

MEASUREMENT WITH BYPASS

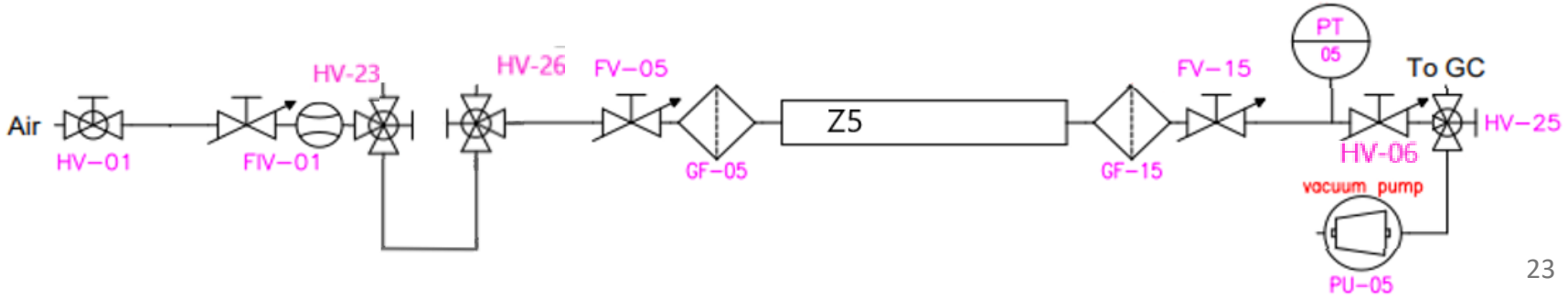
UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

New Setup:

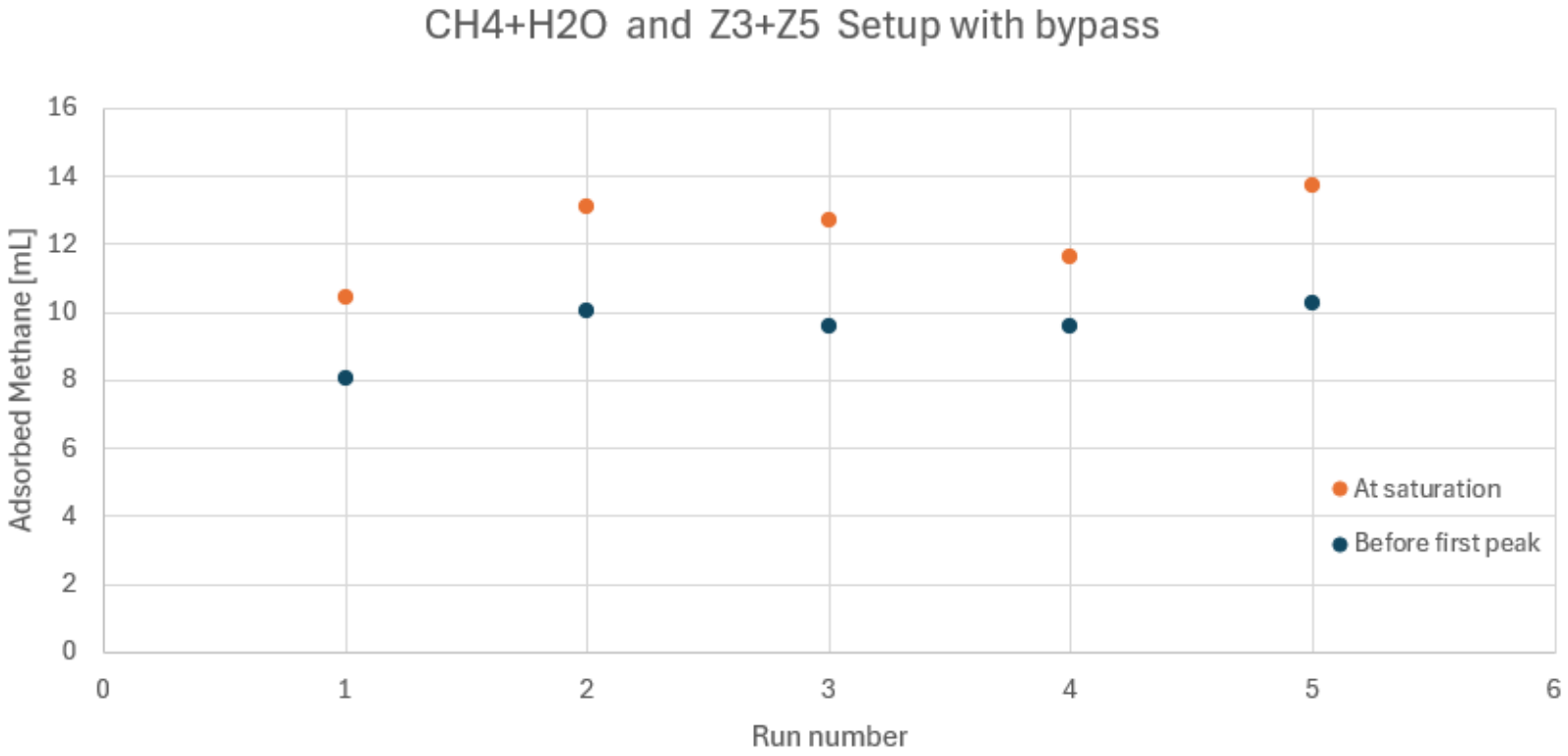


Bypass line to get atmospheric pressure inside the cartridge:



MEASUREMENT WITH BYPASS

New results:



Total of 5 runs, same behaviour

Mean Volume of CH4 adsorbed:

- At saturation: $12.32 \pm 1.3\ mL$
- Before first peak: $9.5 \pm 0.88\ mL$

DIFFERENT RESULTS

	Z5 - only CH4		Z3 + Z5 - H2O + CH4	
	At saturation	Before first peak	At saturation	Before first peak
Mean Volume of CH4 adsorbed	23.6 mL	12.6 mL	12.3 mL	9.51 mL

Total adsorbed CH4 when H2O is added almost half of the adsorbed CH4 with only CH4

Unexpected behaviour

CALCULATIONS

The volume of CH4 adsorbed before first peak is calculated as:

$$Vol_{CH_4\ 1st\ peak}[mL] = \frac{\phi\left[\frac{L}{h}\right]}{3.6} \cdot \Delta t[s] \cdot \frac{C_{saturation}[ppm]}{10^6}$$

Where:

$\phi\left[\frac{L}{h}\right]$ is the flux in the line measured by the *Mass Flow sensor*

$\Delta t[s]$ is the time between the beginning of the flow in the cartridge and the first analysis of the GC where the concentration is not zero (***first peak***)

$C_{saturation}[ppm]$ is the concentration of methane in the mixture seen at saturation at the end of the run

CALCULATIONS

The volume of CH4 adsorbed before first peak is calculated as:

$$Vol_{CH_4\ saturation}[mL] = Vol_{CH_4\ 1st\ peak}[mL] + \sum_i \frac{\phi_i\left[\frac{L}{h}\right]}{3.6} \cdot \Delta t[s] \cdot \frac{C_{saturation}[ppm] - C_{i-th\ analysis}[ppm]}{10^6}$$

Where:

$\phi_i\left[\frac{L}{h}\right]$ is the mean flux measured by the *Mass Flow sensor* during i-th analysis

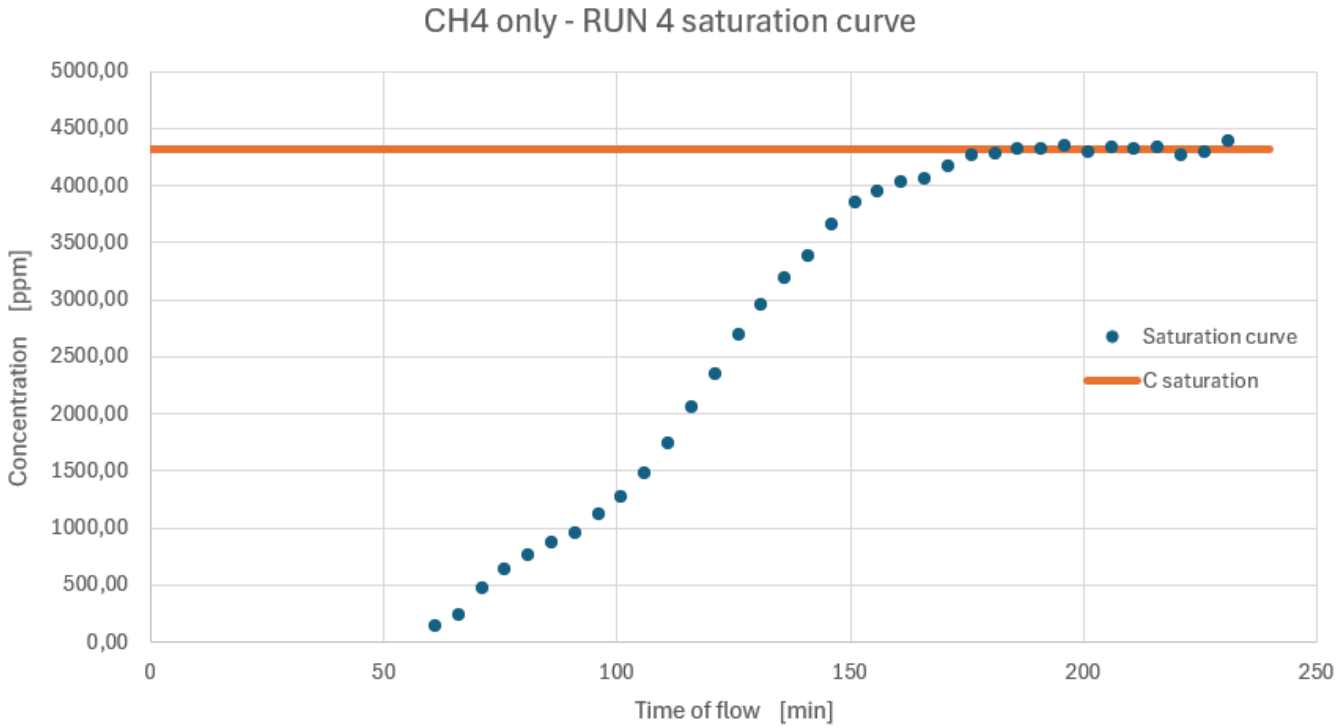
$\Delta t[s]$ is 5 minutes duration of the analysis of the GC

$C_{saturation}[ppm]$ is the concentration of methane in the mixture seen at saturation at the end of the run

$C_{i-th\ analysis}[ppm]$ is the concentration measured by the GC during the i-th analysis

EXAMPLE OF THE INTEGRAL

The volume of CH4 adsorbed before first peak is calculated as:

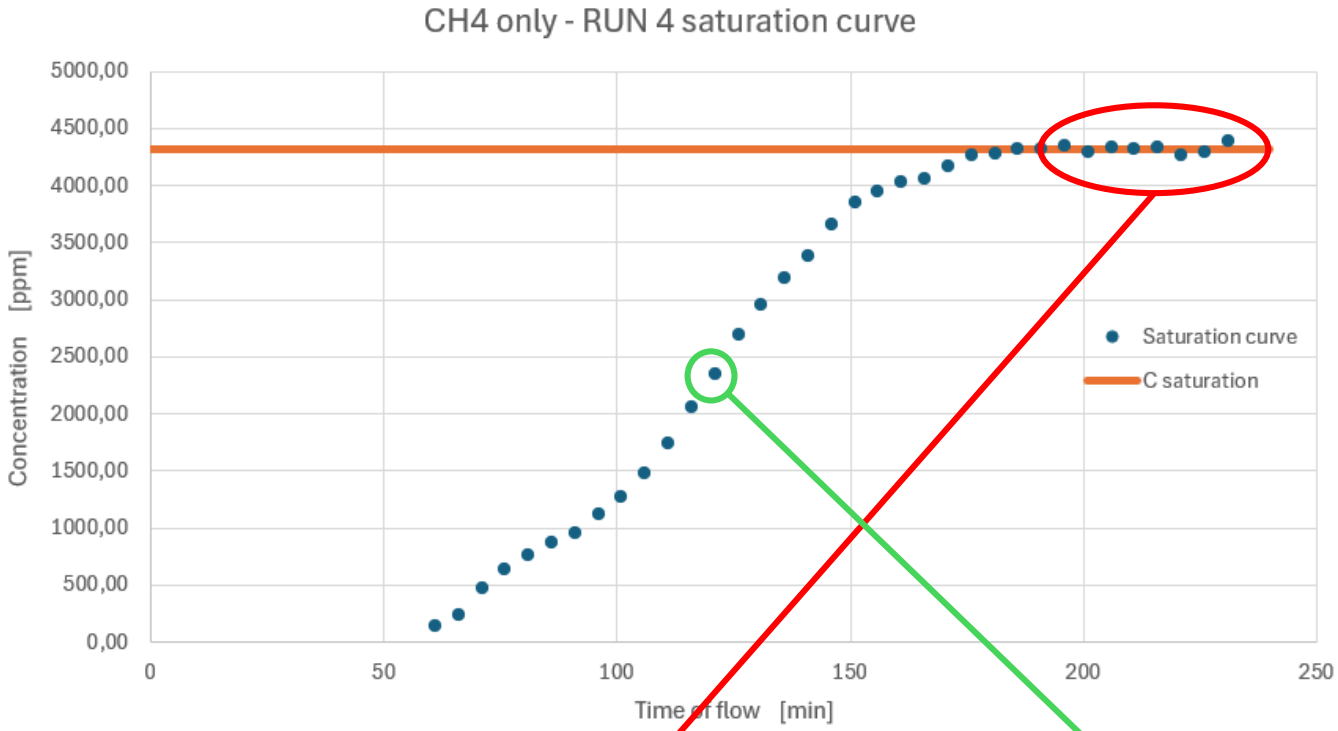


$$Vol_{CH_4\ saturation}[mL] =$$

$$= Vol_{CH_4\ 1st\ peak}[mL] + \sum_i \frac{\phi_i\left[\frac{L}{h}\right]}{3.6} \cdot \Delta t[s] \cdot \frac{C_{saturation}[ppm] - C_{i-th\ analysis}[ppm]}{10^6}$$

EXAMPLE OF THE INTEGRAL

The volume of CH4 adsorbed before first peak is calculated as:

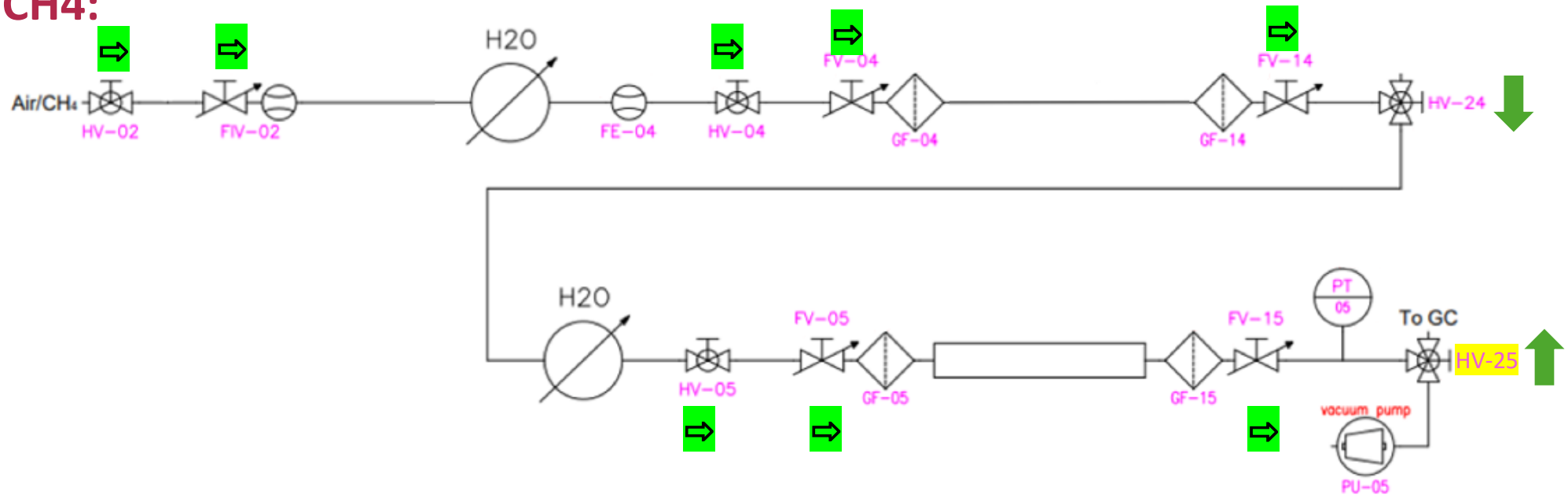


$$Vol_{CH_4\ saturation}[mL] =$$

$$= Vol_{CH_4\ 1st\ peak}[mL] + \sum_i \frac{\phi_i\left[\frac{L}{h}\right]}{3.6} \cdot \Delta t[s] \cdot \frac{C_{saturation}[ppm] - C_{i-th\ analysis}[ppm]}{10^6}$$

REGENERATION PROCEDURE

Measurement **only CH4:**



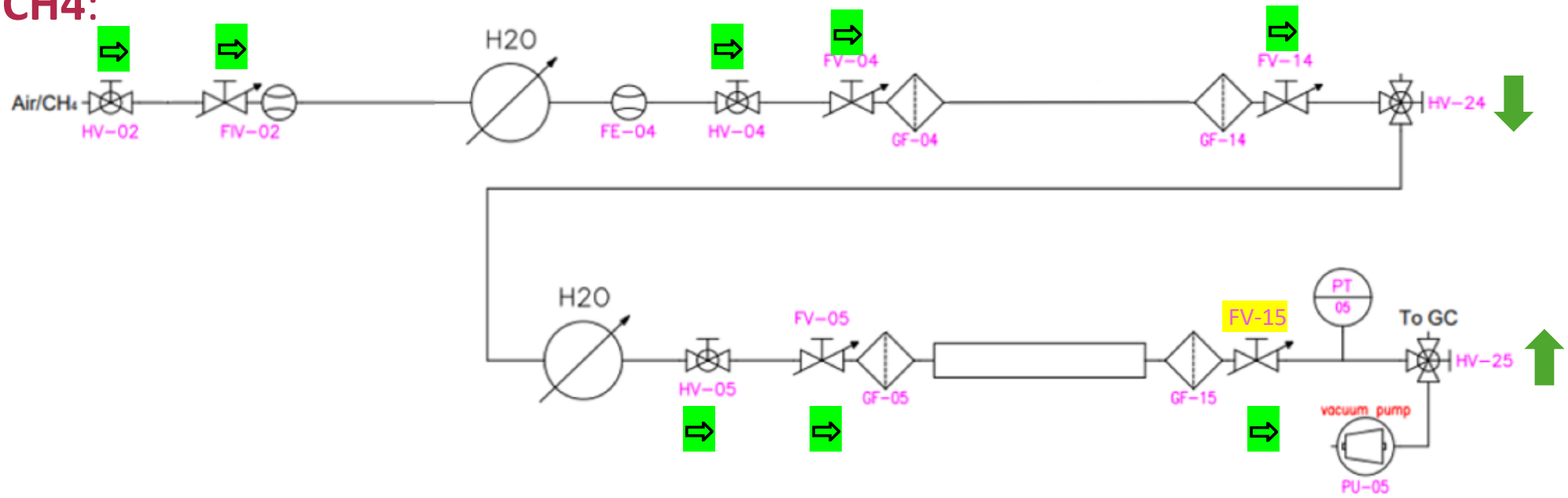
⇨ = open valve

X = close valve

During RUN all valves are open and the HV-25 3way is directed to the GC

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

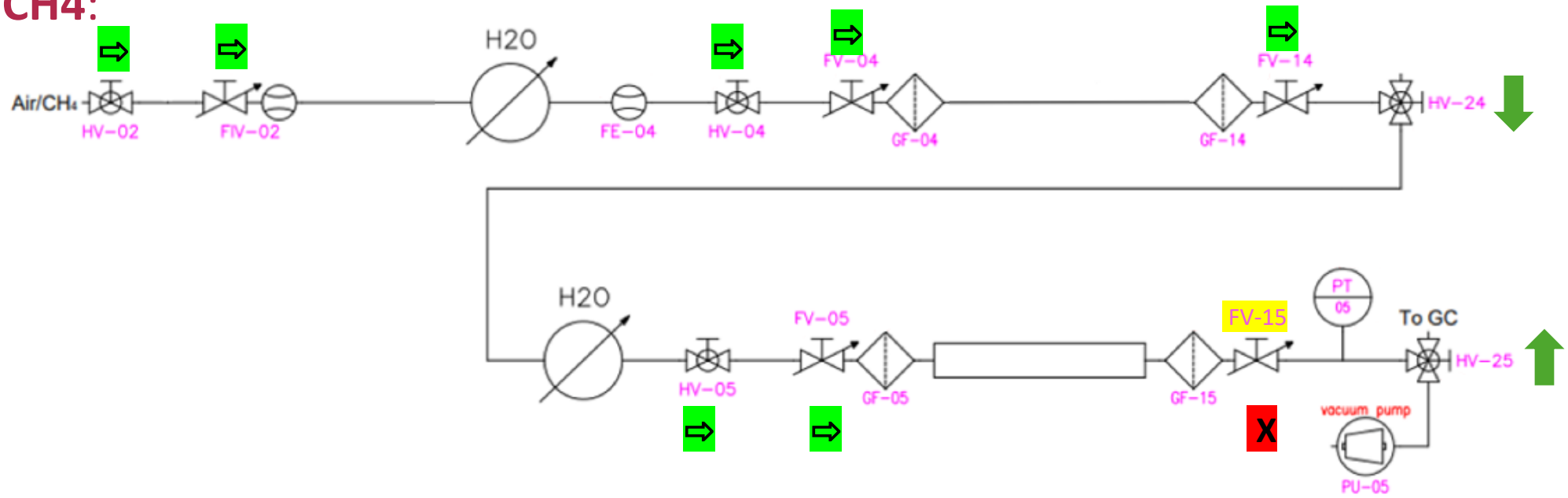
X = close valve

When saturation is reached, we closed all the valves going backwards starting from

FV-15

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

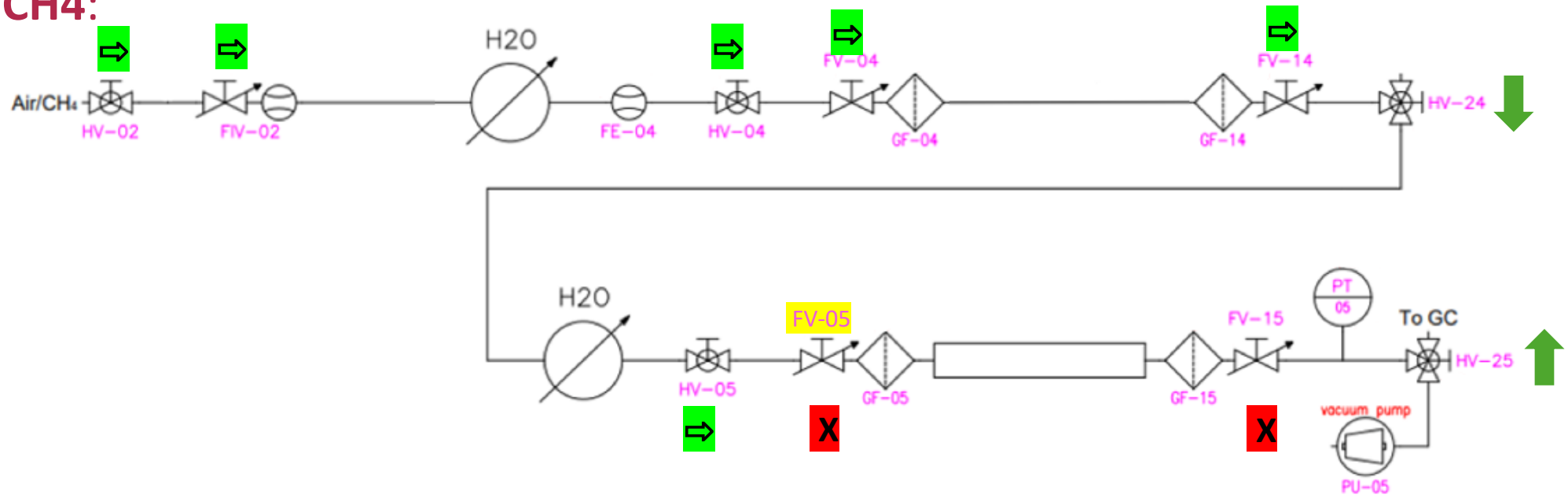
X = close valve

When saturation is reached, we closed all the valves going backwards starting from:

FV-15 →

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

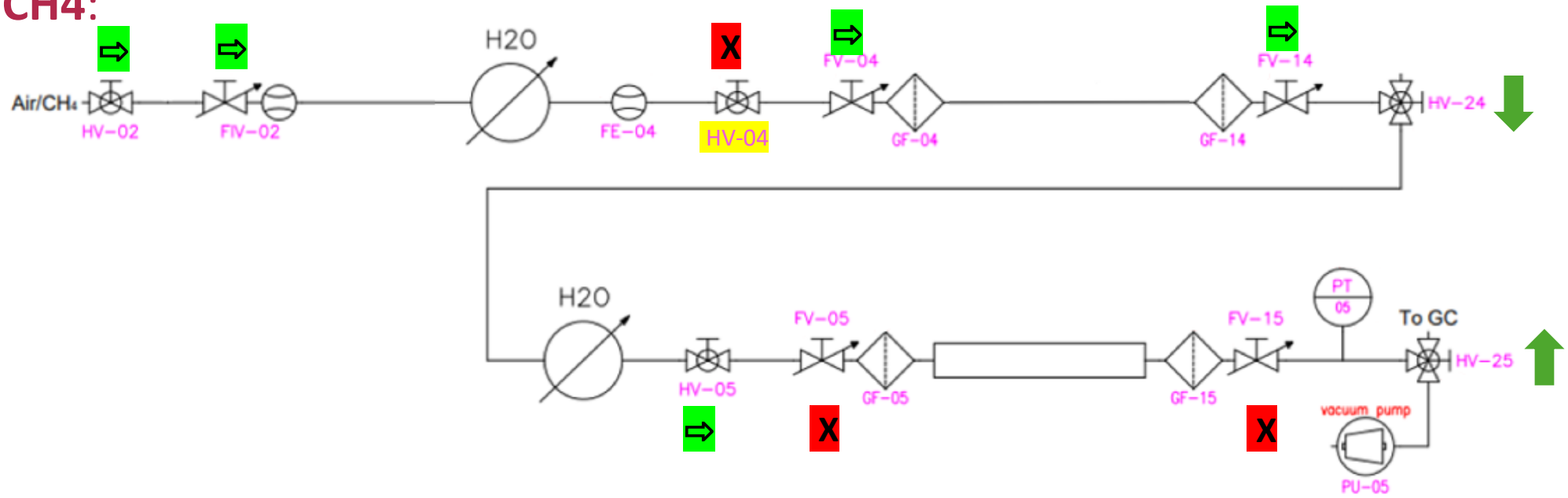
X = close valve

When saturation is reached, we closed all the valves going backwards starting from

FV-15 → FV-05 →

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

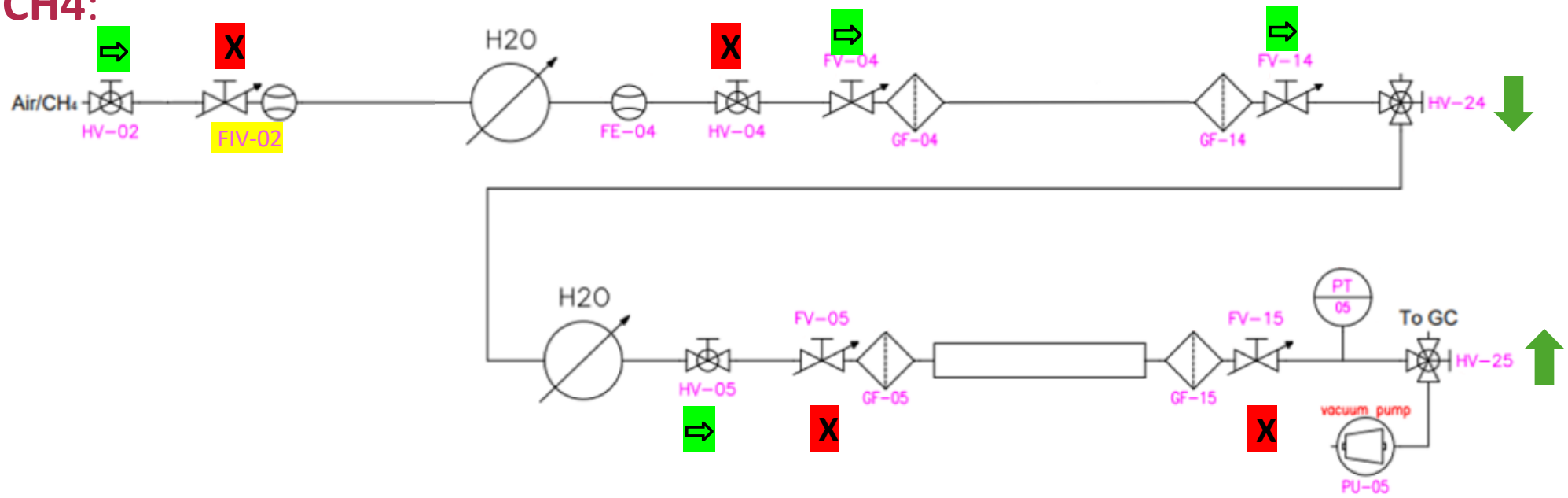
X = close valve

When saturation is reached, we closed all the valves going backwards starting from

FV-15 → FV-05 → HV-04 →

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

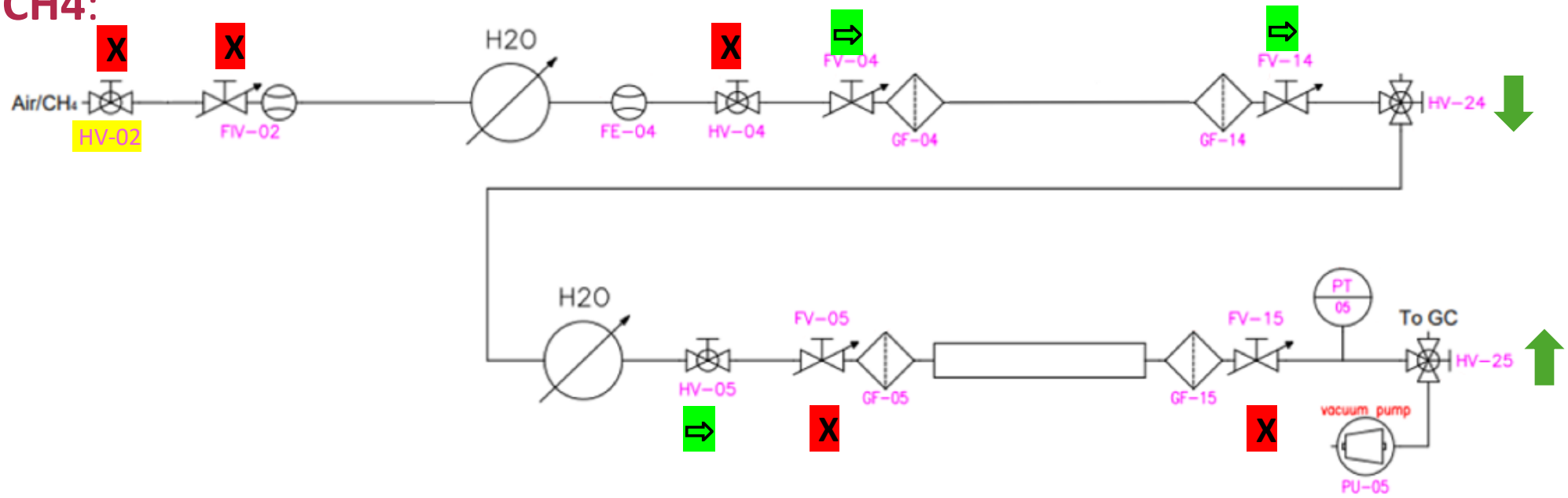
X = close valve

When saturation is reached, we closed all the valves going backwards starting from

FV-15 → FV-05 → HV-04 → FIV-02 →

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

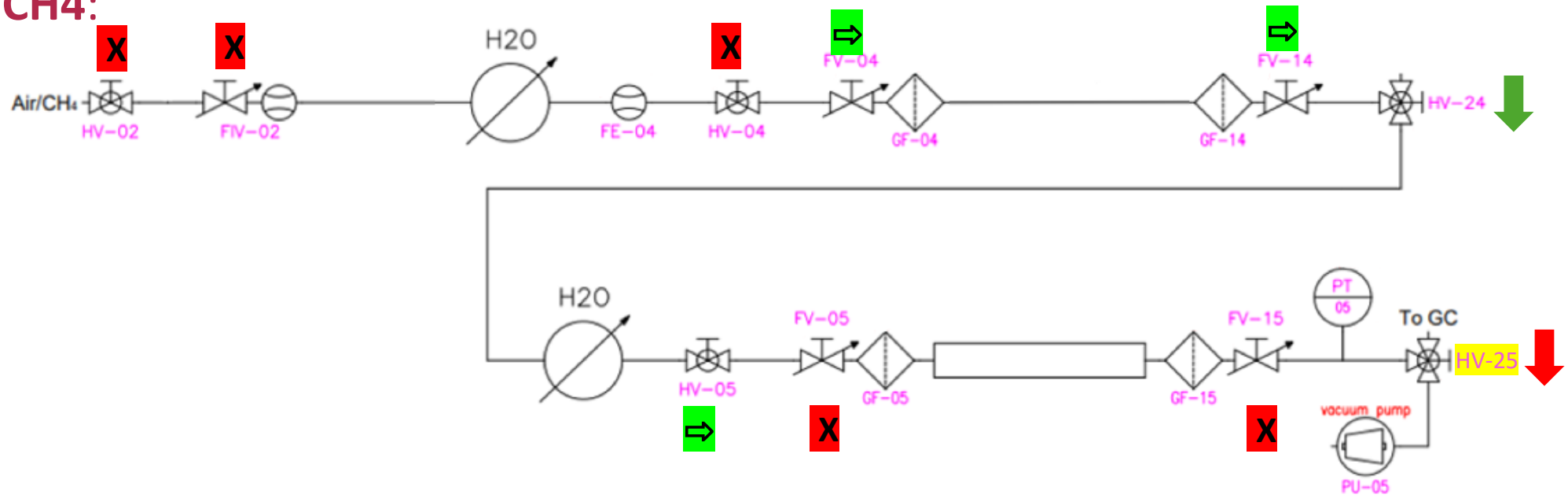
X = close valve

When saturation is reached, we closed all the valves going backwards starting from

FV-15 → FV-05 → HV-04 → FIV-02 → HV-02

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

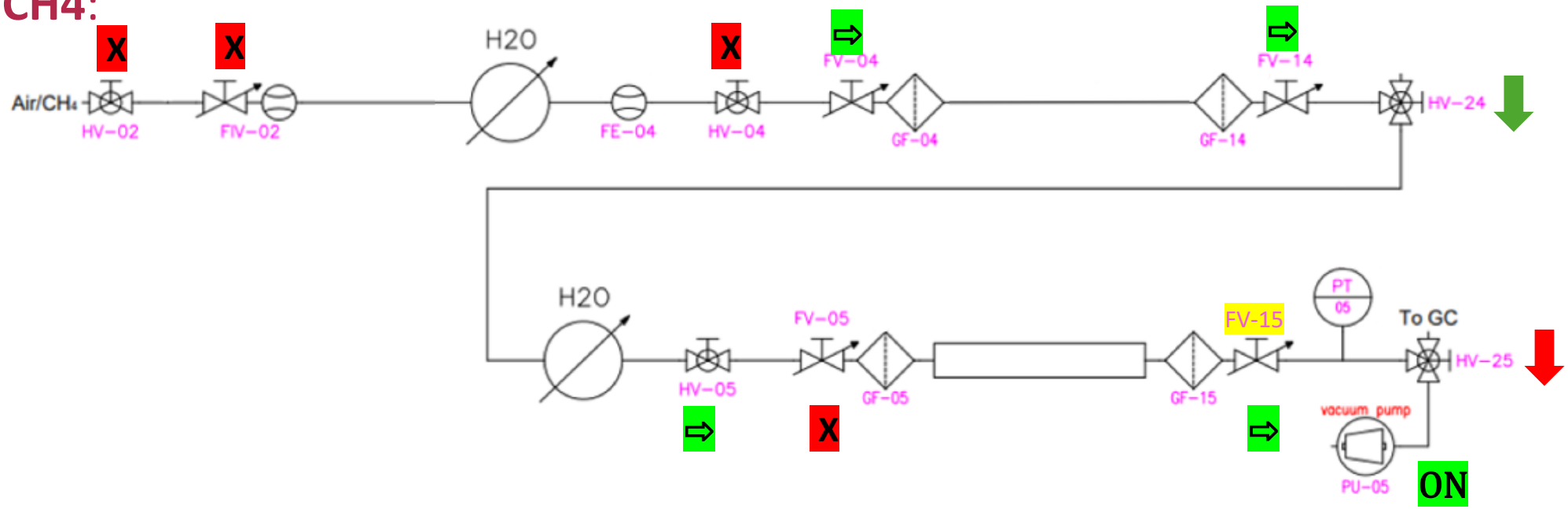
When saturation is reached, we closed all the valves going backwards starting from

FV-15 → FV-05 → HV-04 → FIV-02 → HV-02

Then we switch the 3way HV-25 going to the **vacuum pump** for regeneration

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

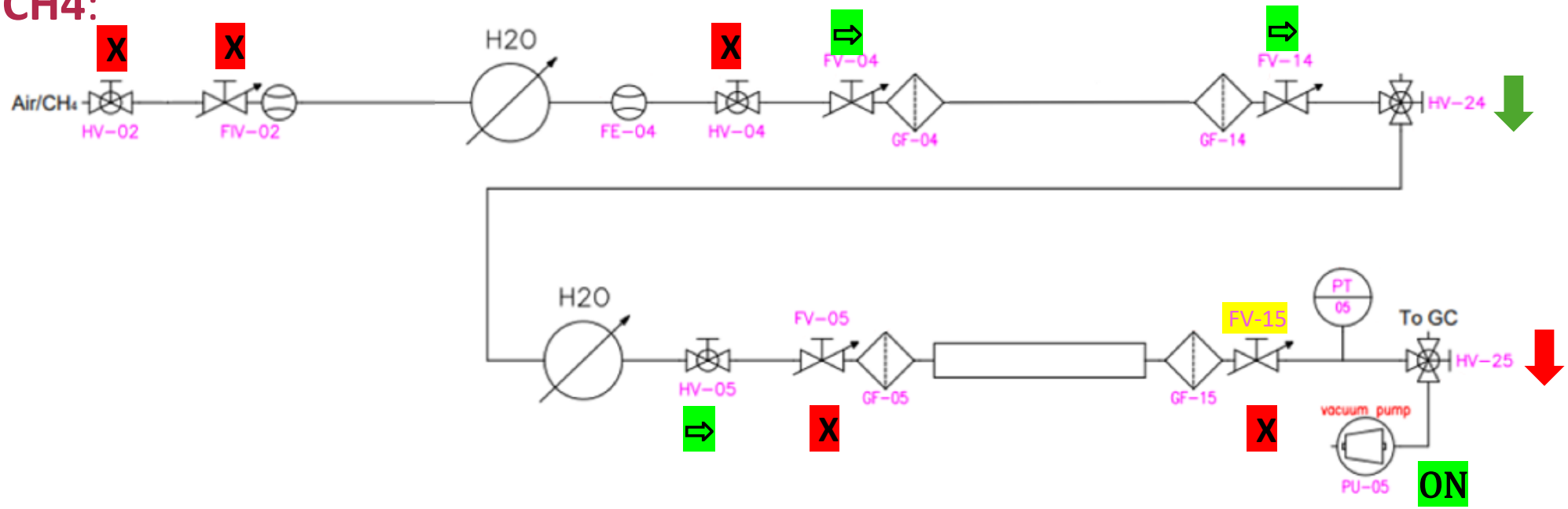
X = close valve

Now we open FV-15 and activate the pump ON.

40 minutes regeneration

REGENERATION PROCEDURE

Measurement **only CH4**:



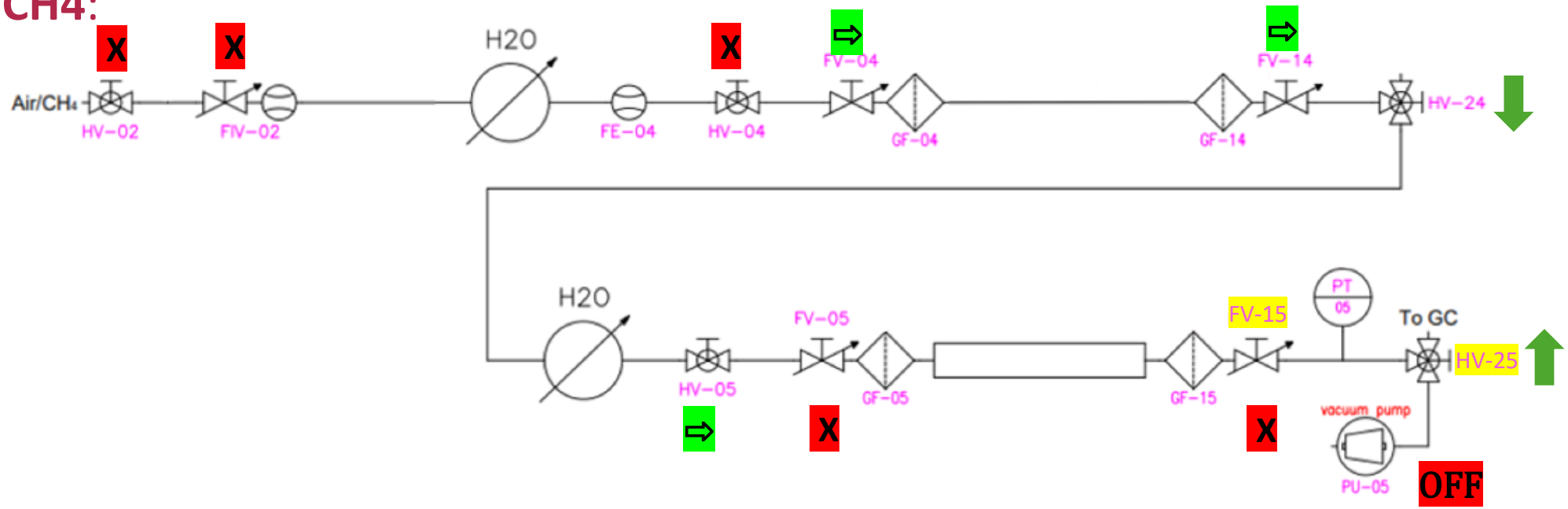
⇨ = open valve

X = close valve

After regeneration we close FV-15

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

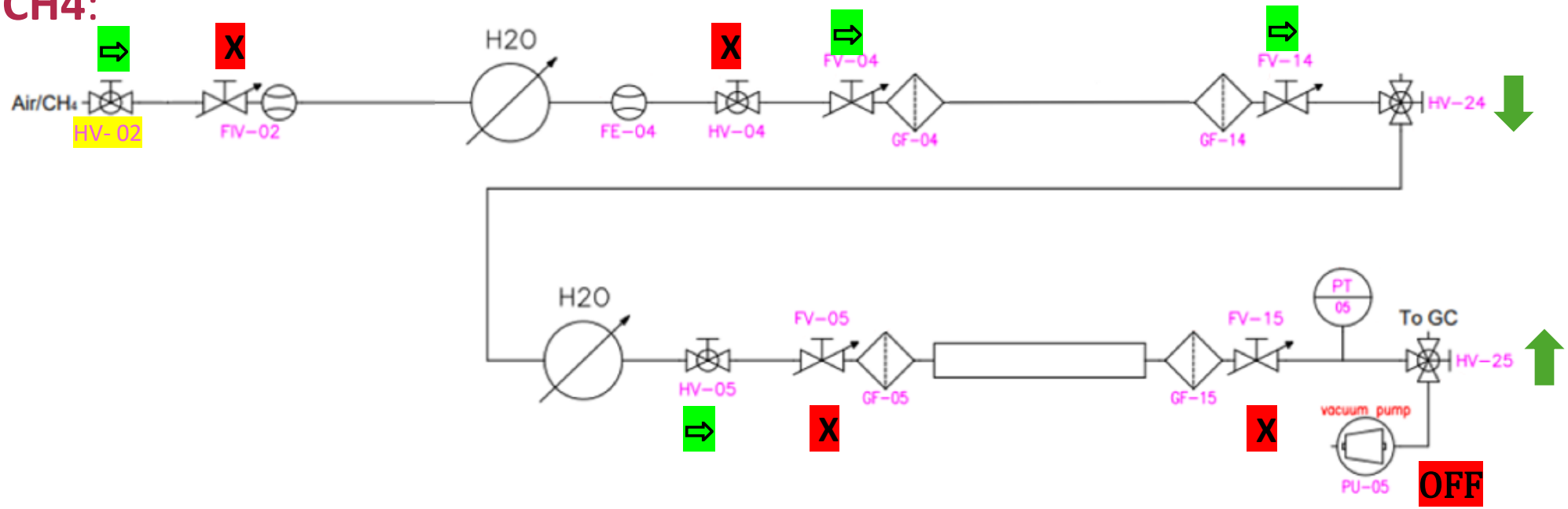
After regeneration we close FV-15

Then turn OFF the vacuum pump and redirect HV-25 to GC and we can prepare a new RUN

Next steps: open all valves starting from the bottle HV-02

REGENERATION PROCEDURE

Measurement **only CH4**:



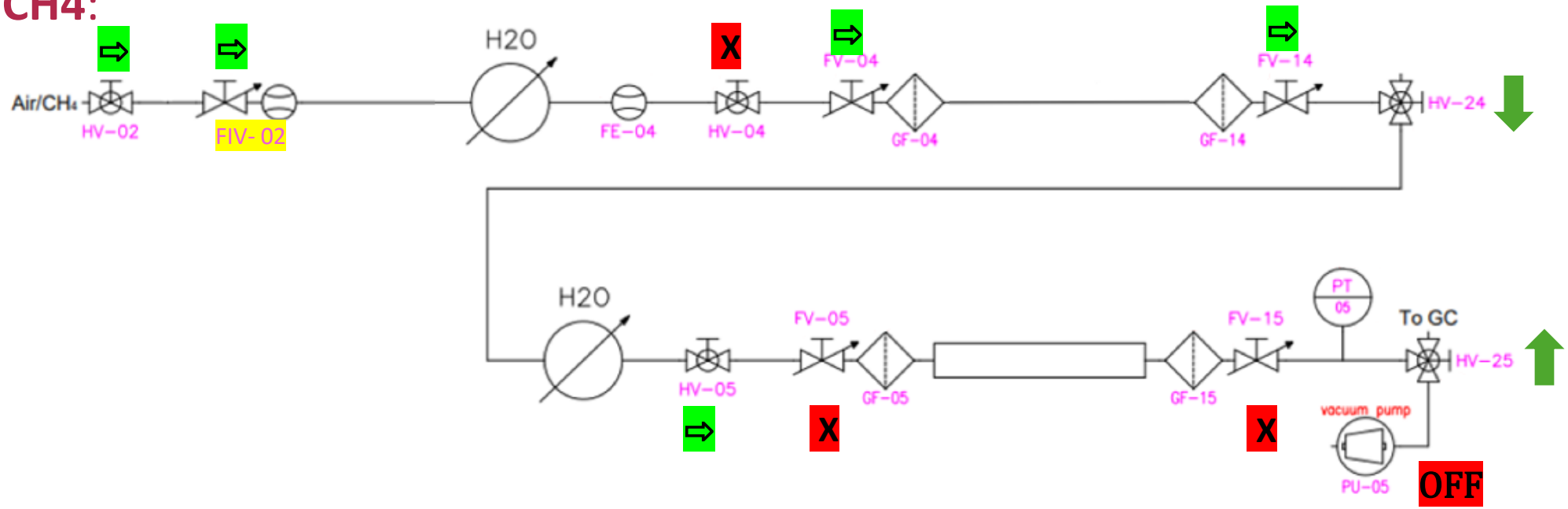
BEFORE NEW RUN: open all valves forward from the bottle

HV-02 →

Inside Z5 Cartridge there is vacuum $P = 2\,mbar$ (corresponds to sensor sensitivity)

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

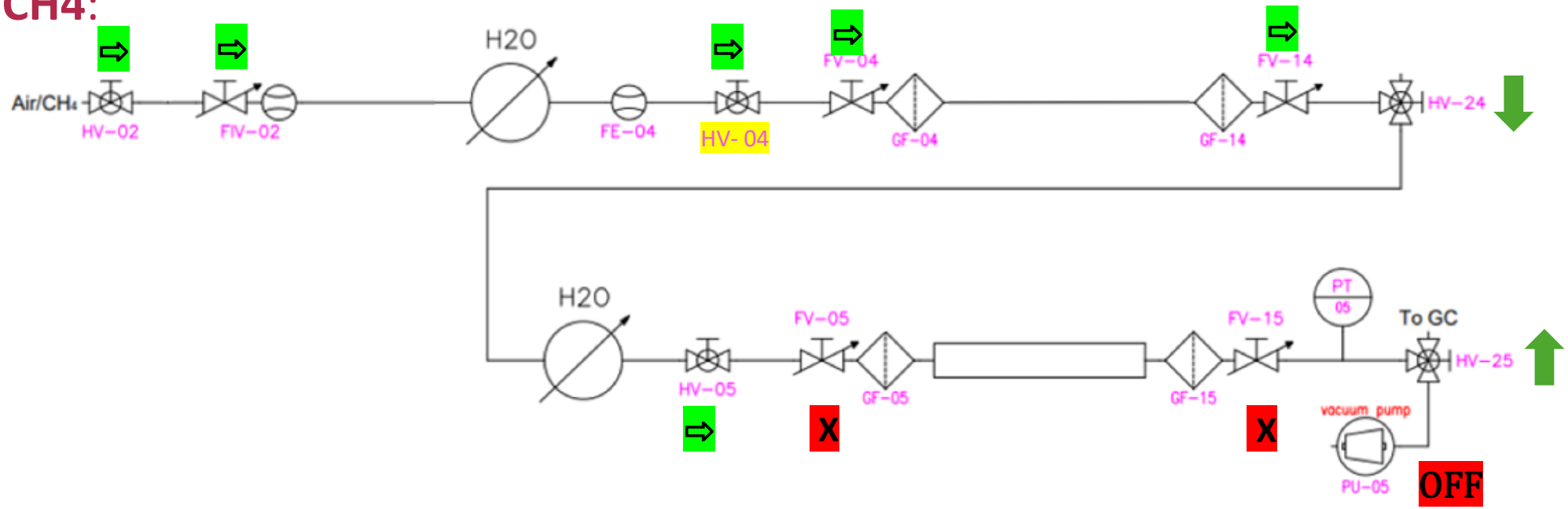
BEFORE NEW RUN: open all valves forward from the bottle

HV-02 → FV-02 →

Inside Z5 Cartridge there is vacuum $P = 2\,mbar$ (corresponds to sensor sensitivity)

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

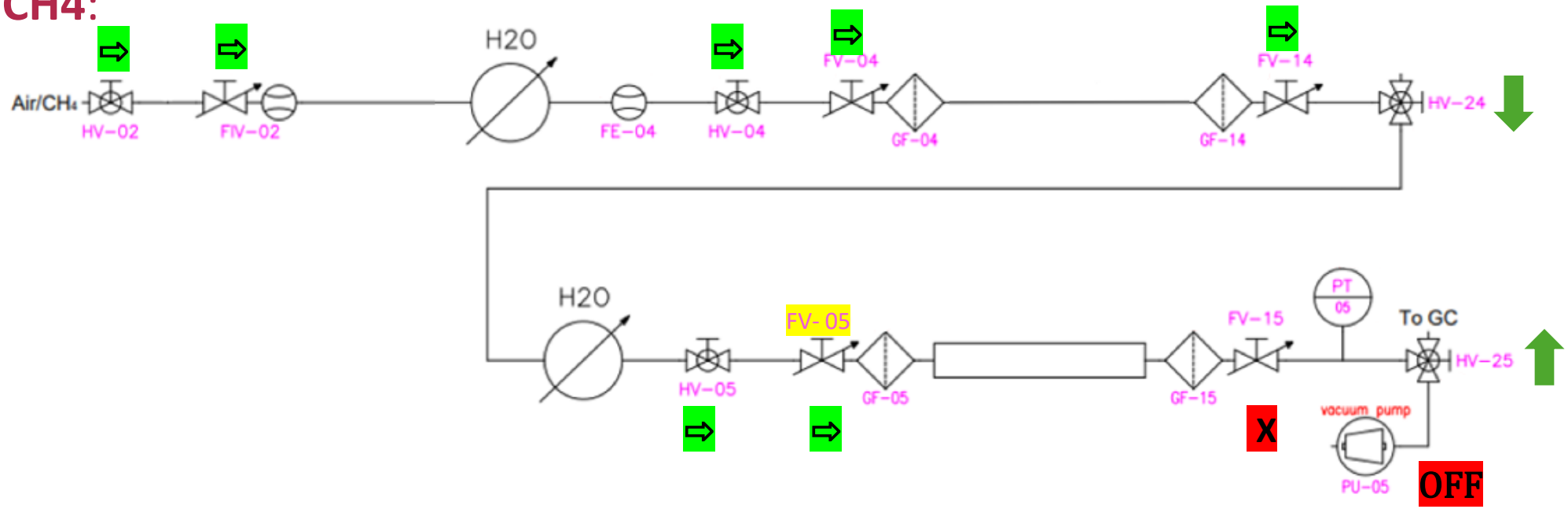
BEFORE NEW RUN: open all valves forward from the bottle

HV-02 → FV-02 → HV-04 →

Inside Z5 Cartridge there is vacuum $P = 2\,mbar$ (corresponds to sensor sensitivity)

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

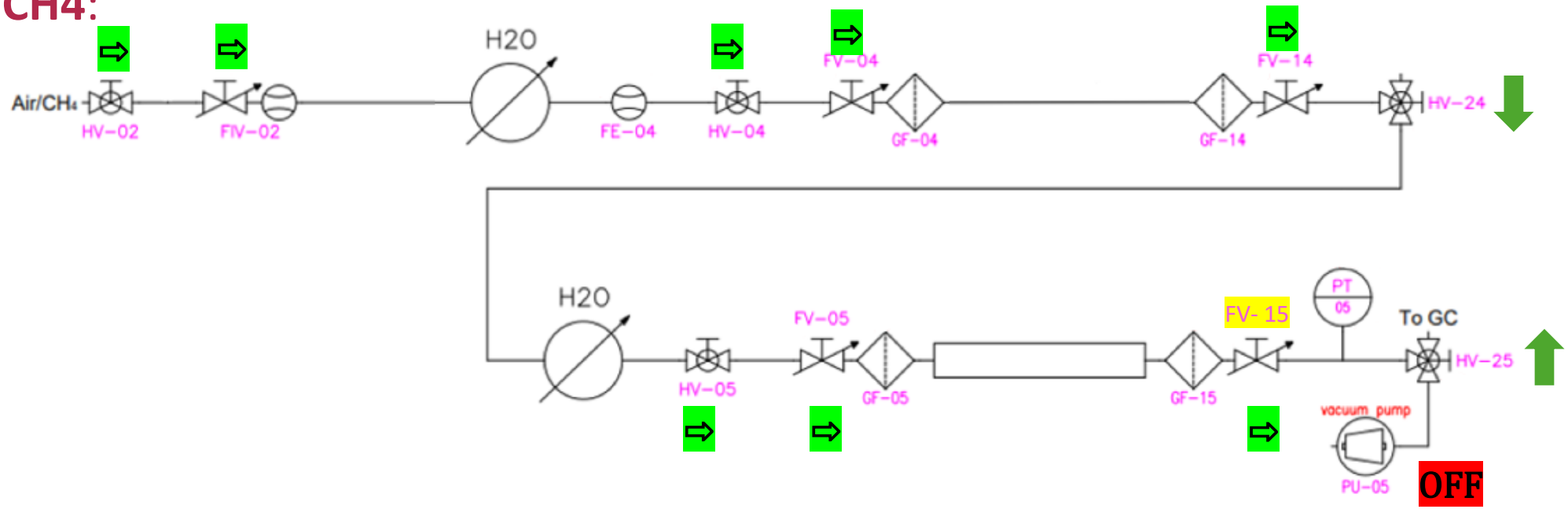
BEFORE NEW RUN: open all valves forward from the bottle

HV-02 → FV-02 → HV-04 → FV-05 →

Pressure inside the cartridge increases rapidly

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

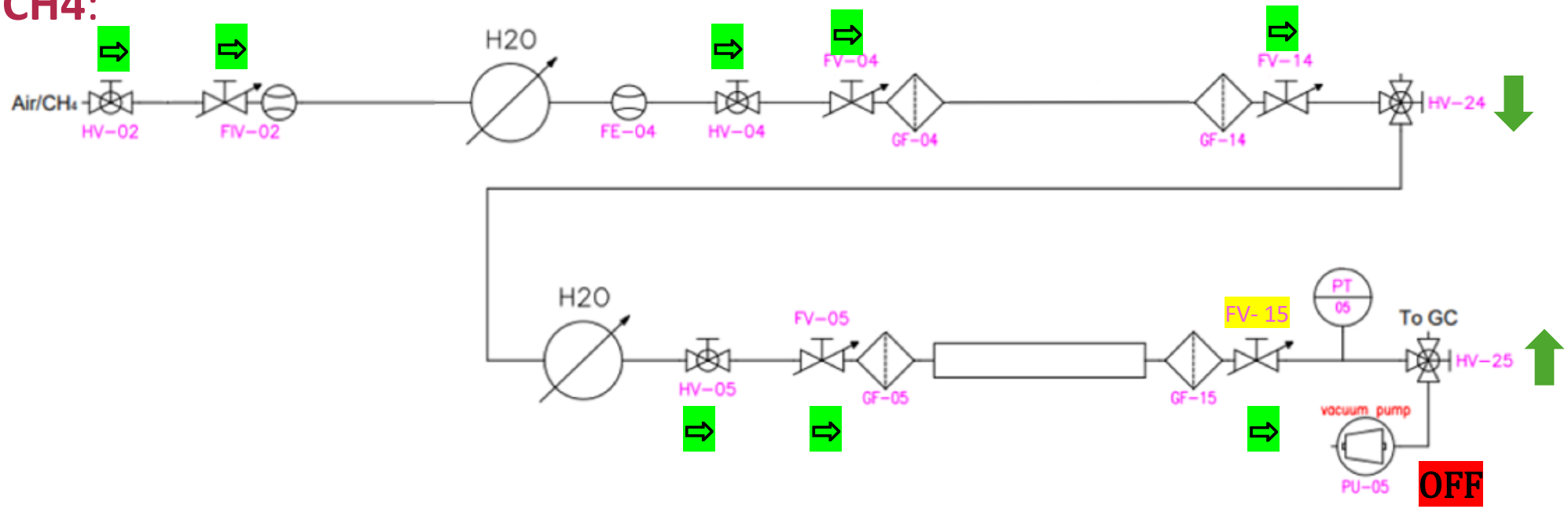
BEFORE NEW RUN: open all valves forward from the bottle

HV-02 → FV-02 → HV-04 → FV-05 → FV-15

Pressure inside the cartridge increases rapidly

REGENERATION PROCEDURE

Measurement **only CH4**:



⇨ = open valve

X = close valve

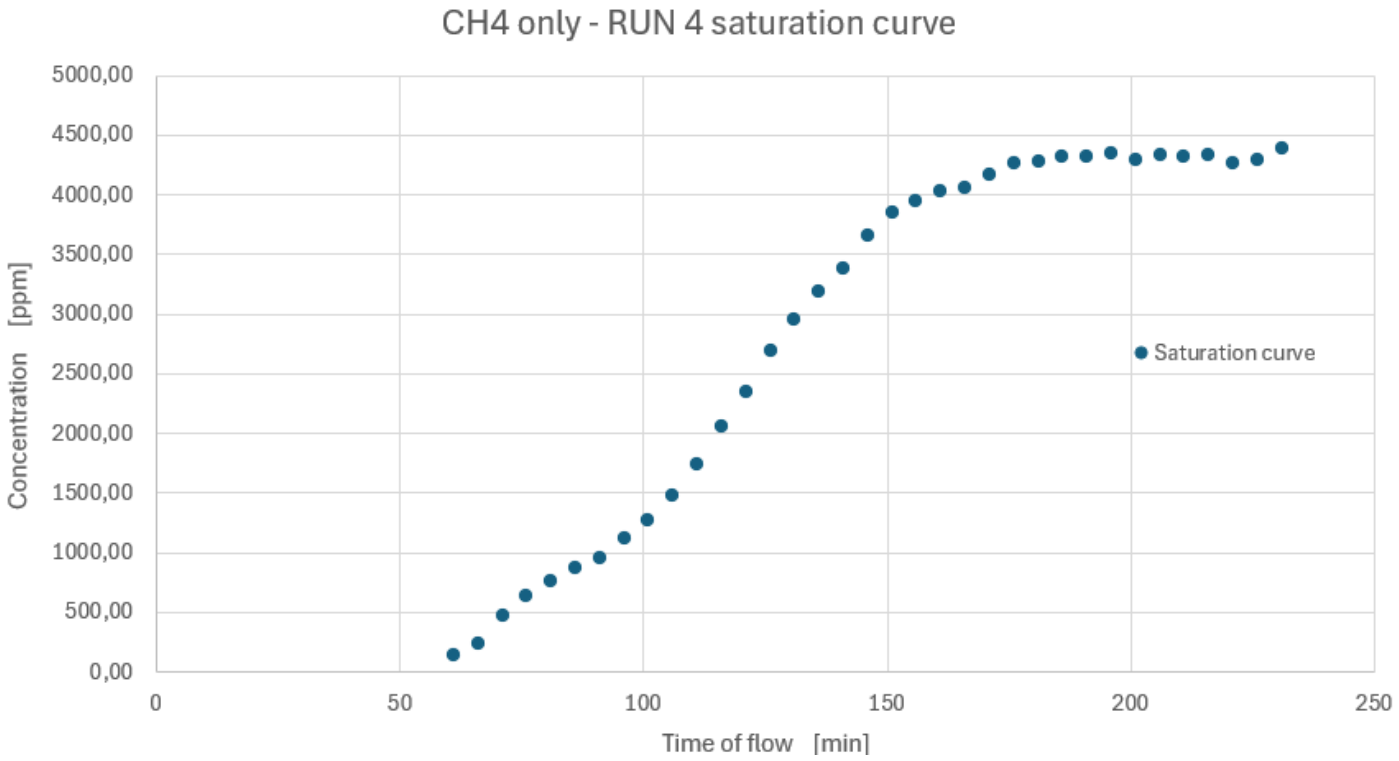
BEFORE NEW RUN: open all valves forward from the bottle

HV-02 → FV-02 → HV-04 → FV-05 → FV-15 → GC analysis start

NEW RUN until saturation

REGENERATION PROCEDURE

Example of saturation curve with this method:



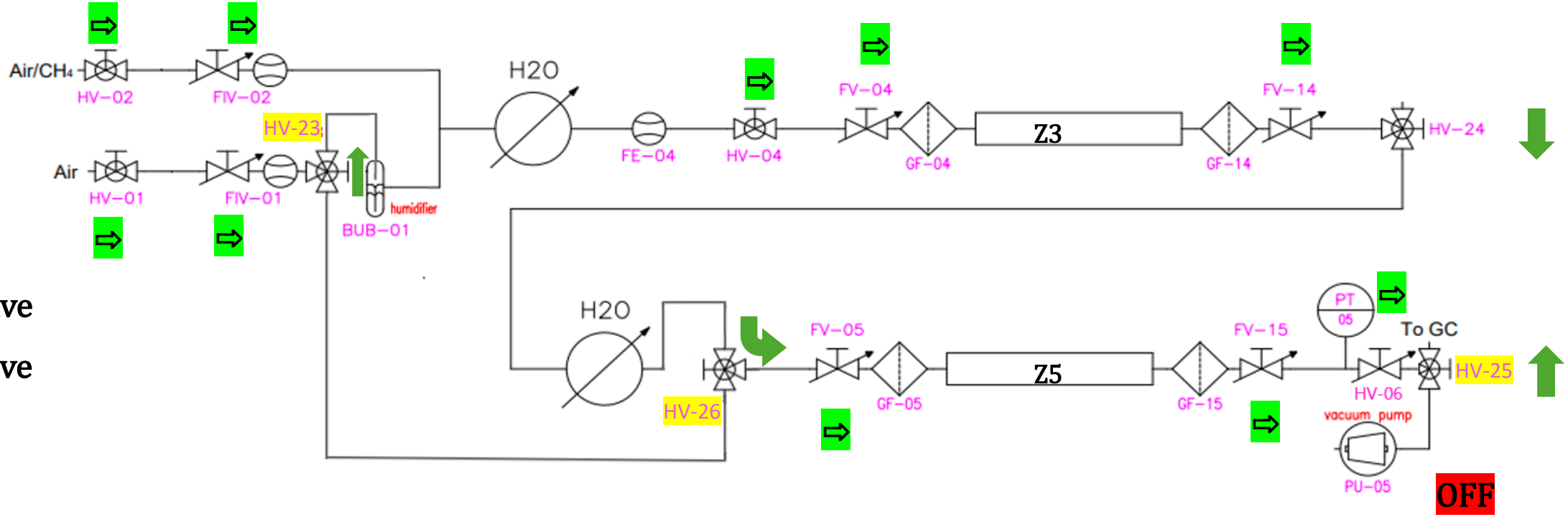
Chromatogram value was zero in the first analysis

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

During RUN all valves are open and:

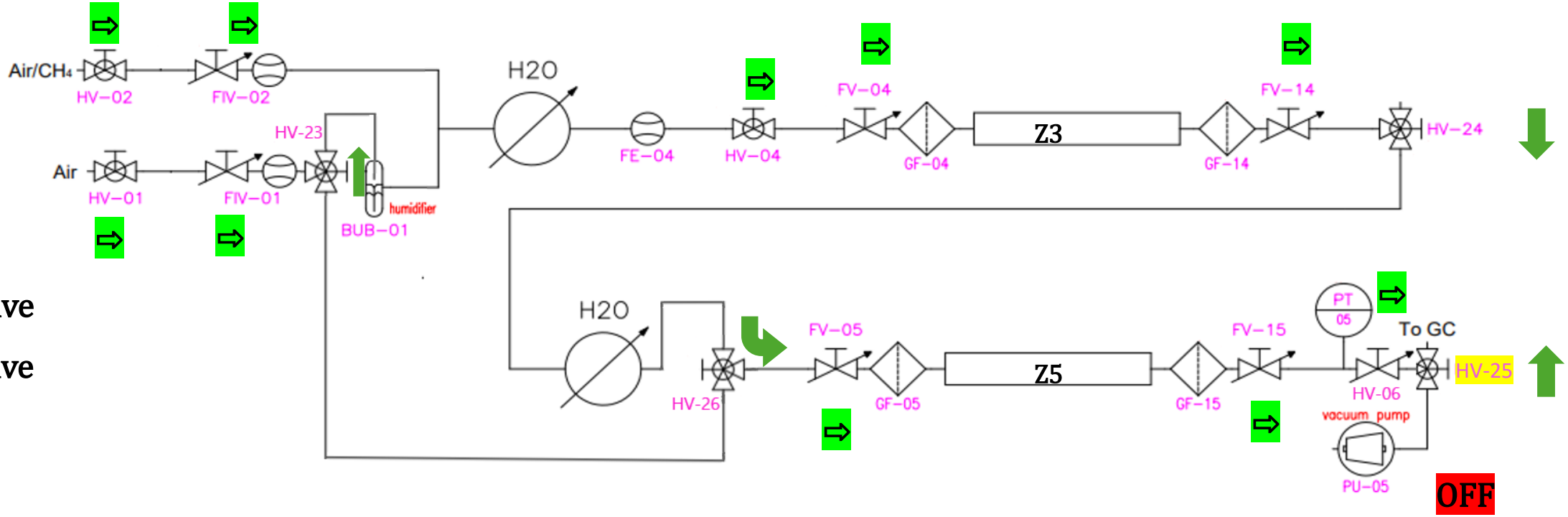
- HV-23 3way-valve is directed to the main line with the cartridges
- HV-26 3way-valve has the main line as input
- HV-25 3way-valve is directed to the GC

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

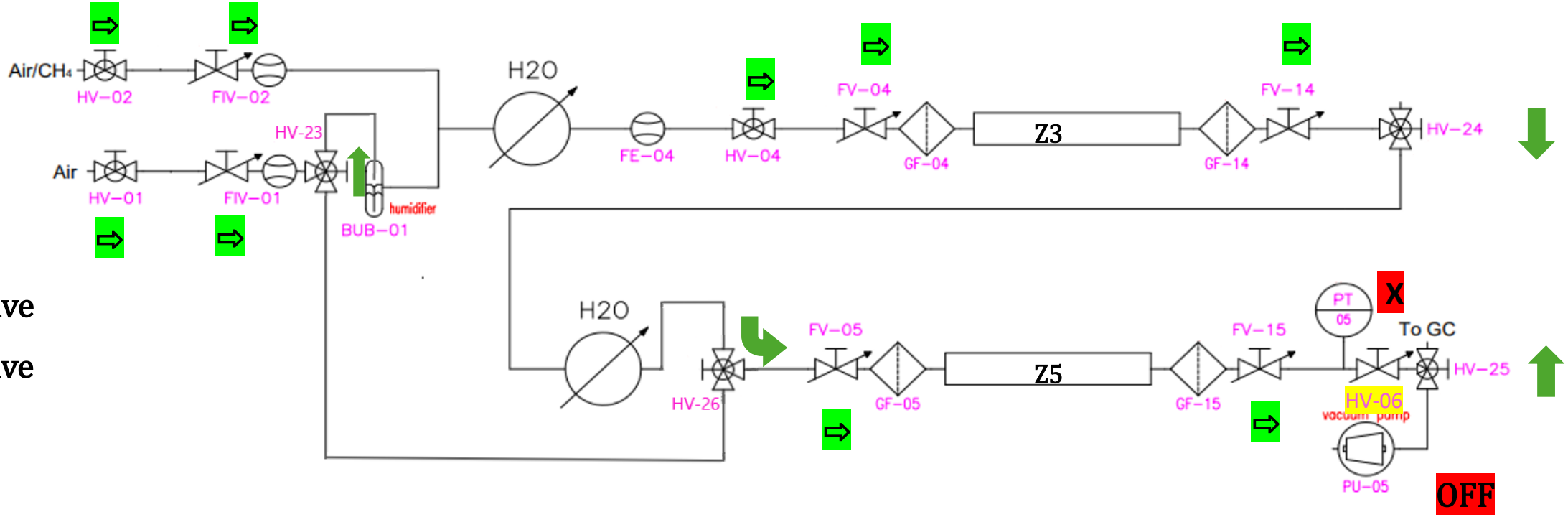
When saturation is reached we close backwards all valves

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

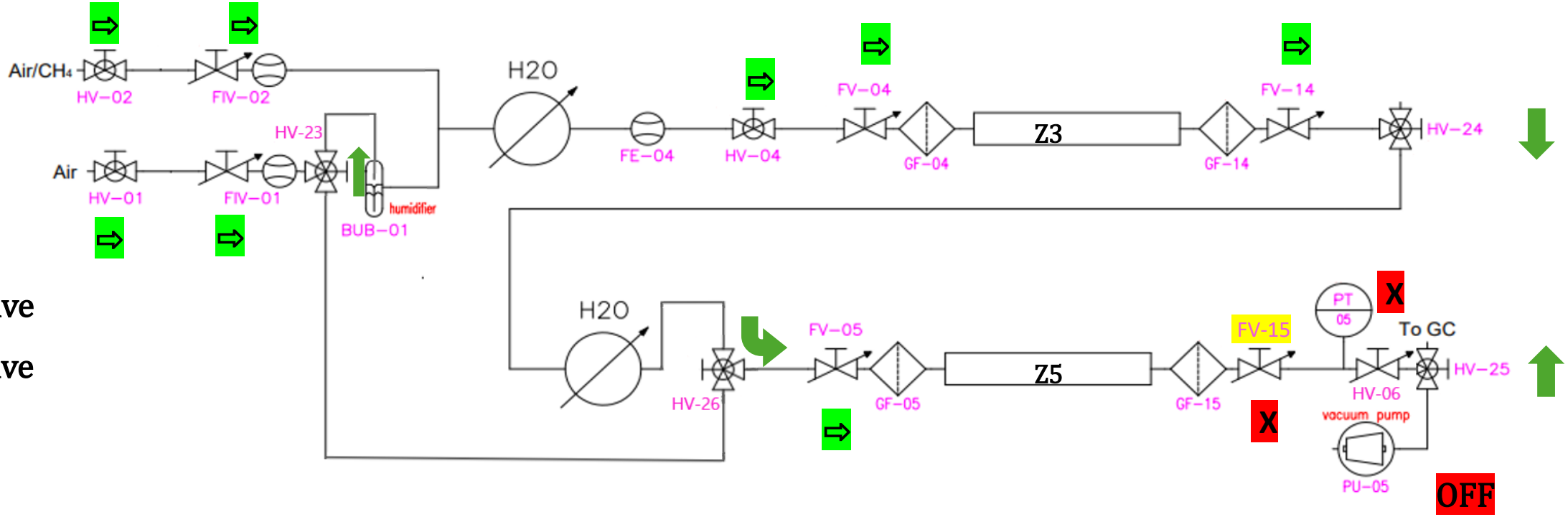
HV-06 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

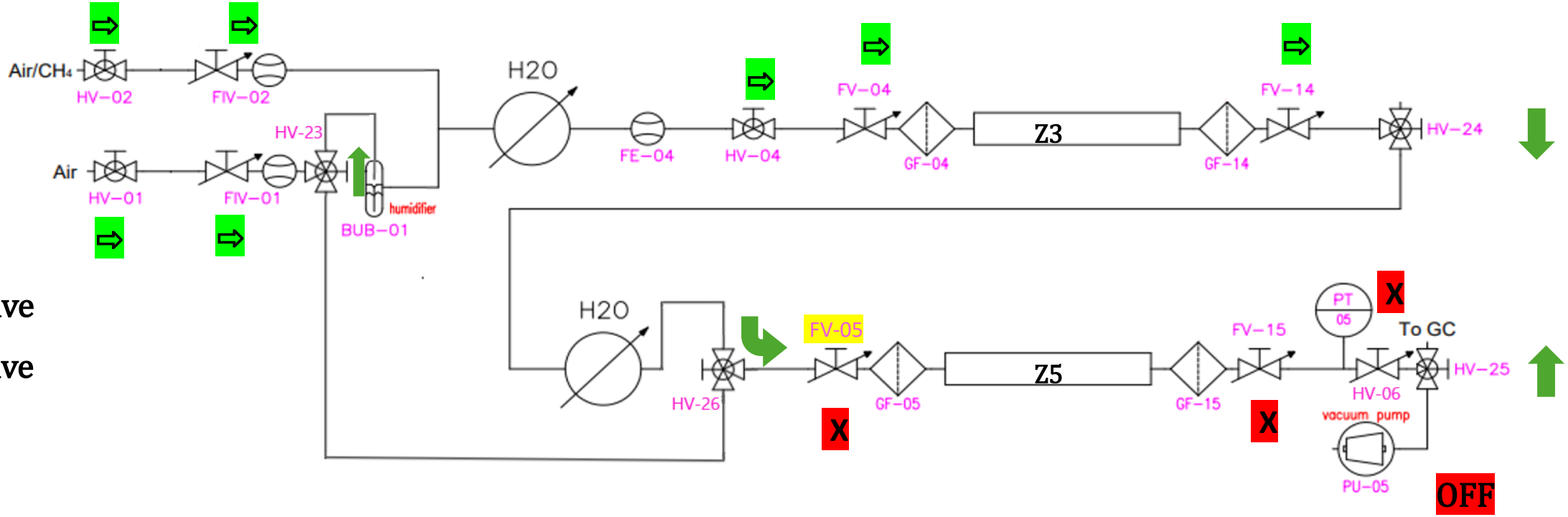
HV-06 → FV-15 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

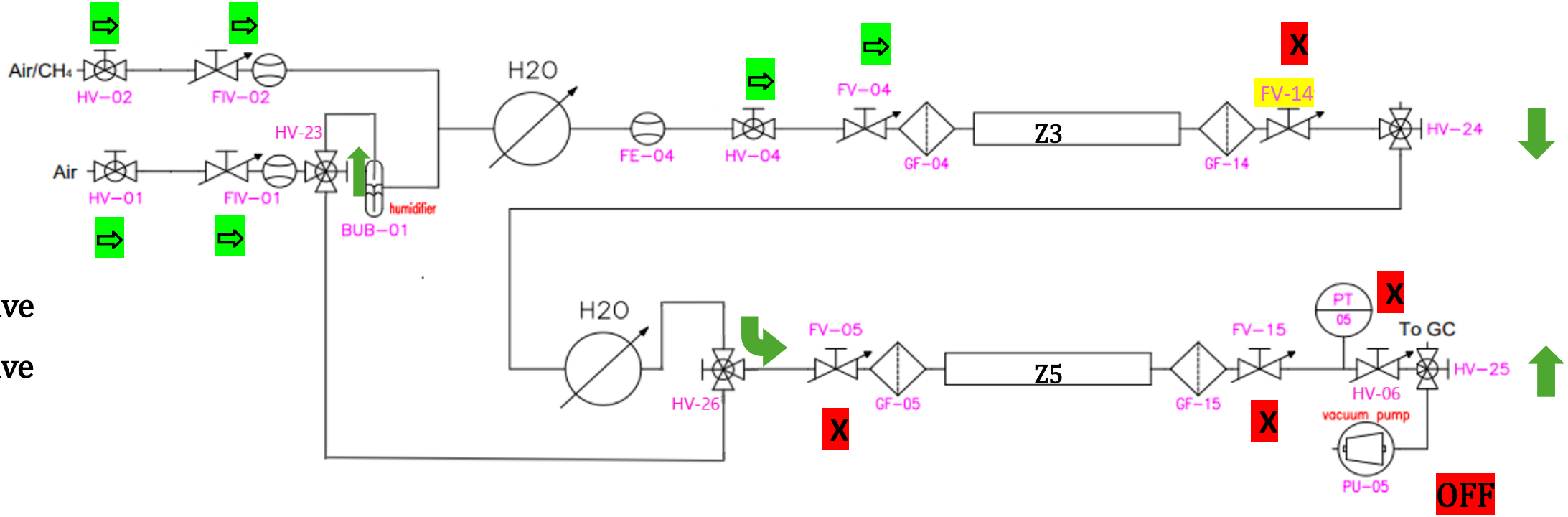
HV-06 → FV-15 → FV-05 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

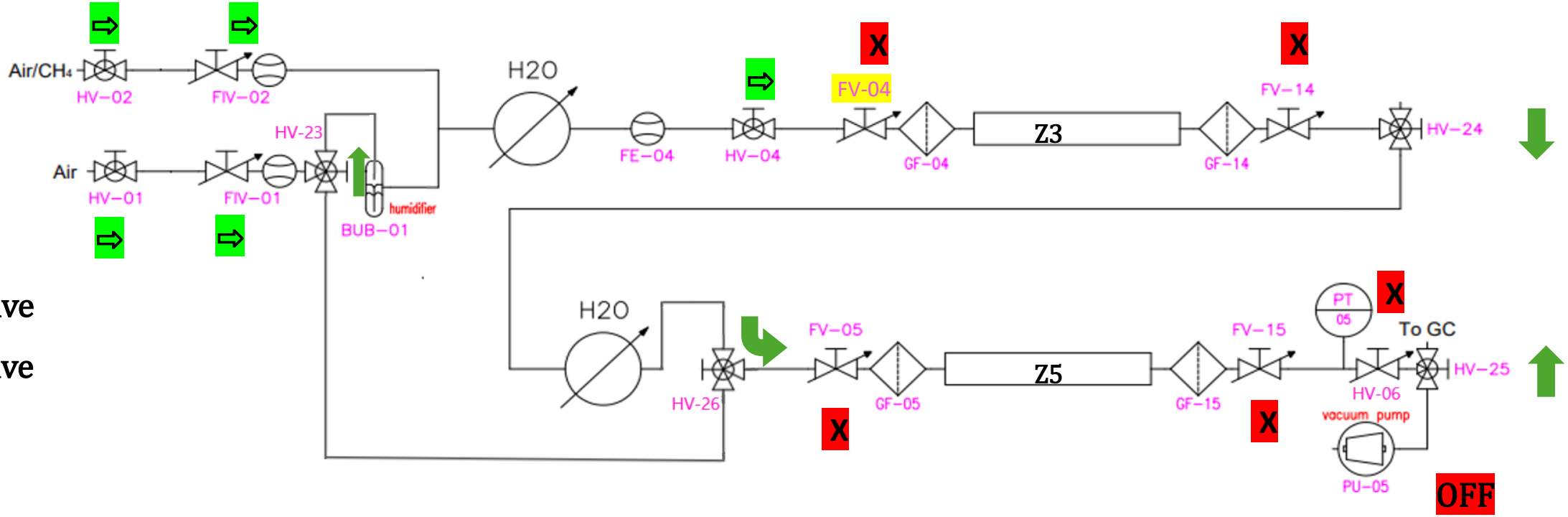
HV-06 → FV-15 → FV-05 → FV-14 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

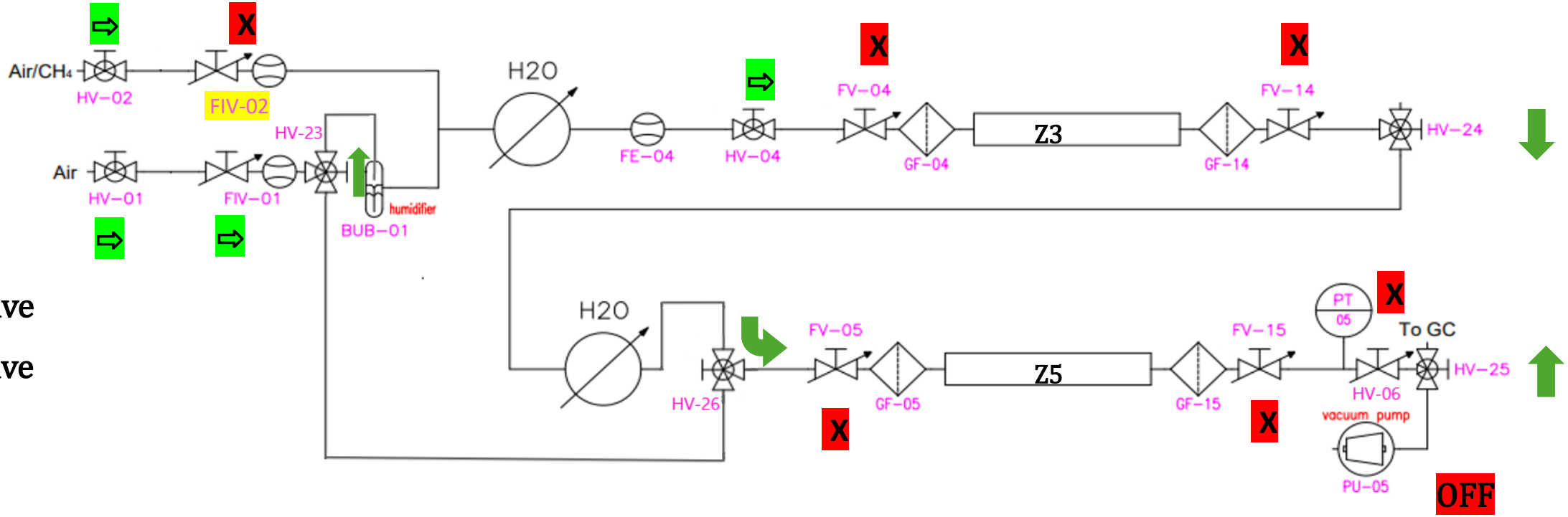
HV-06 → FV-15 → FV-05 → FV-14 → FV-04 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

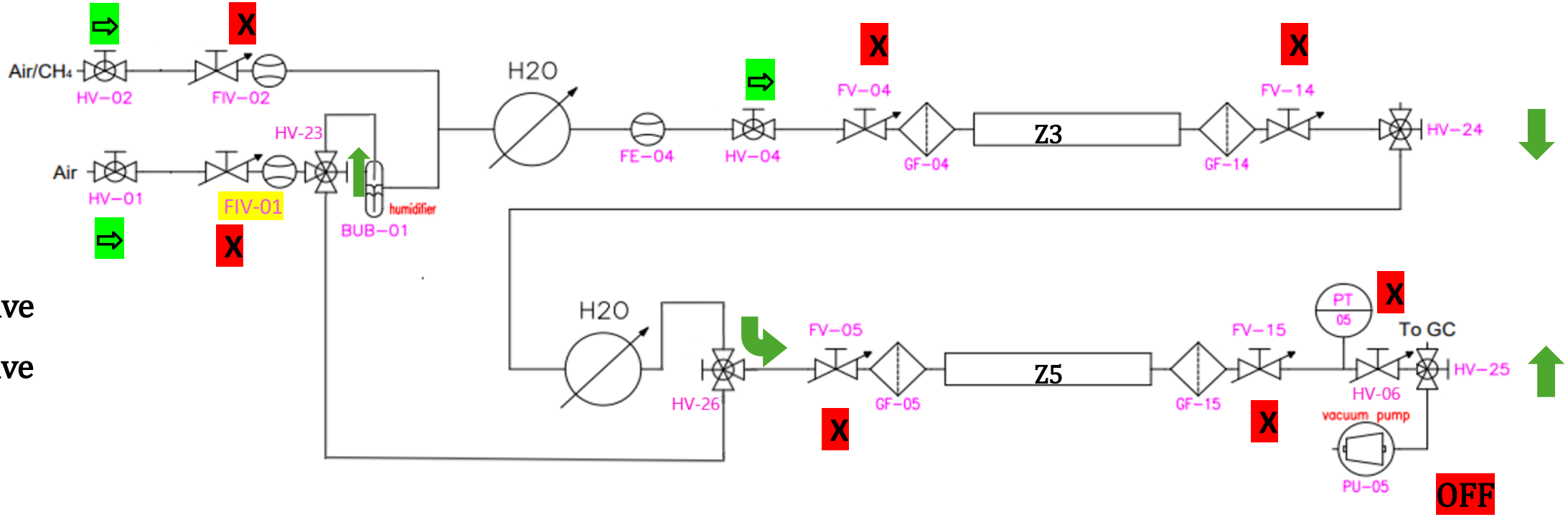
HV-06 → FV-15 → FV-05 → FV-14 → FV-04 → FIV-02 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



When saturation is reached we close backwards all valves

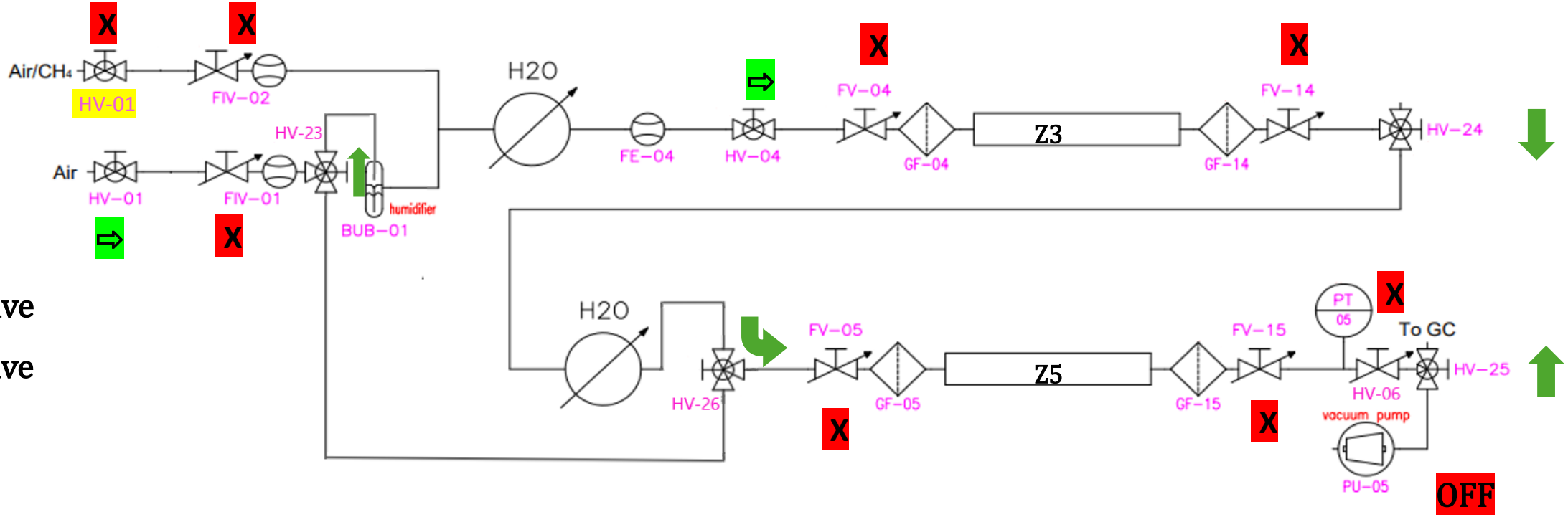
HV-06 → FV-15 → FV-05 → FV-14 → FV-04 → FIV-02 → FIV-01 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

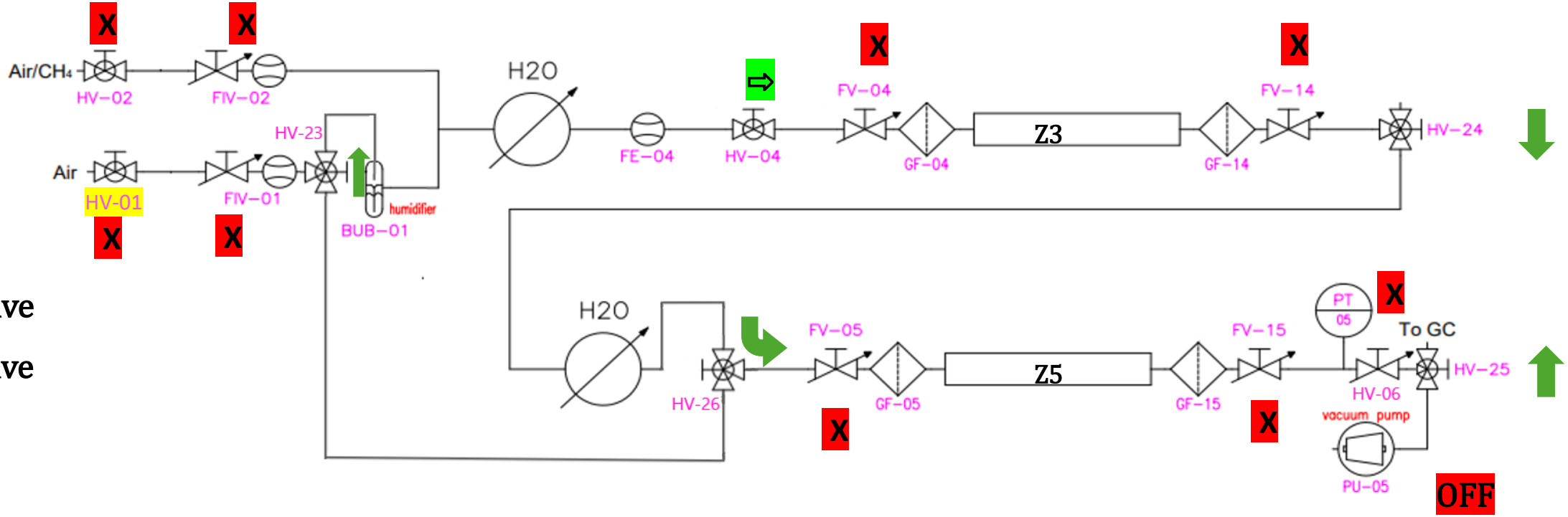
HV-06 → FV-15 → FV-05 → FV-14 → FV-04 → FIV-02 → FIV-01 → HV-02 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When saturation is reached we close backwards all valves

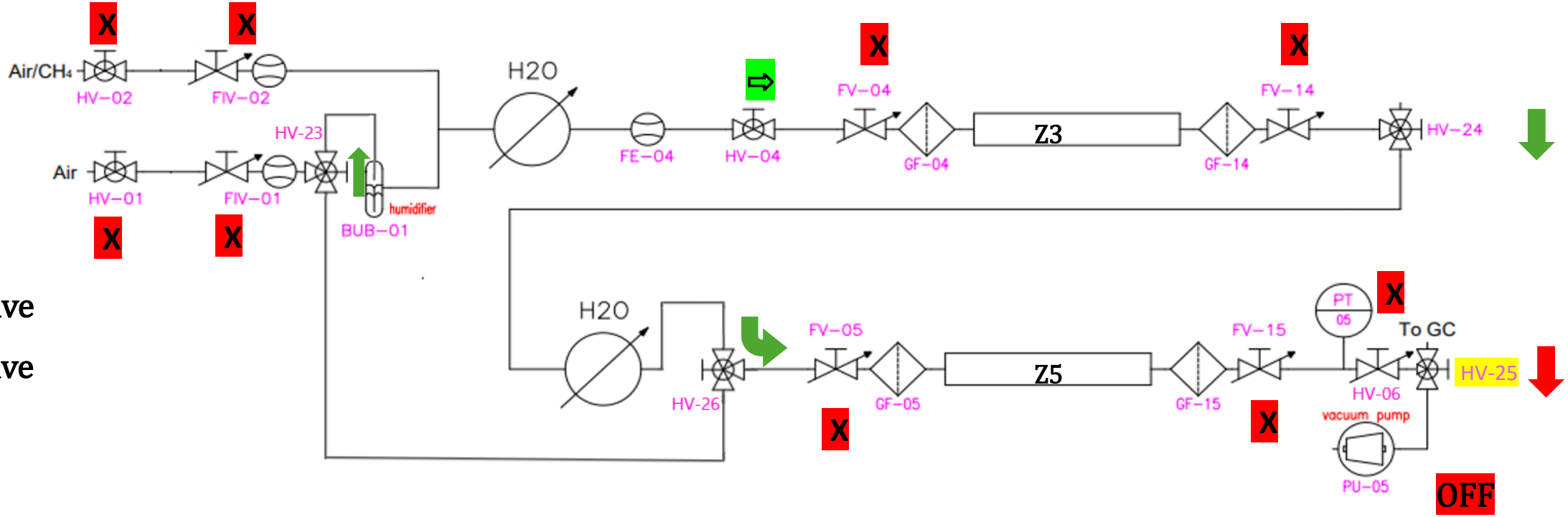
HV-06 → FV-15 → FV-05 → FV-14 → FV-04 → FIV-02 → FIV-01 → HV-02 → HV-01

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

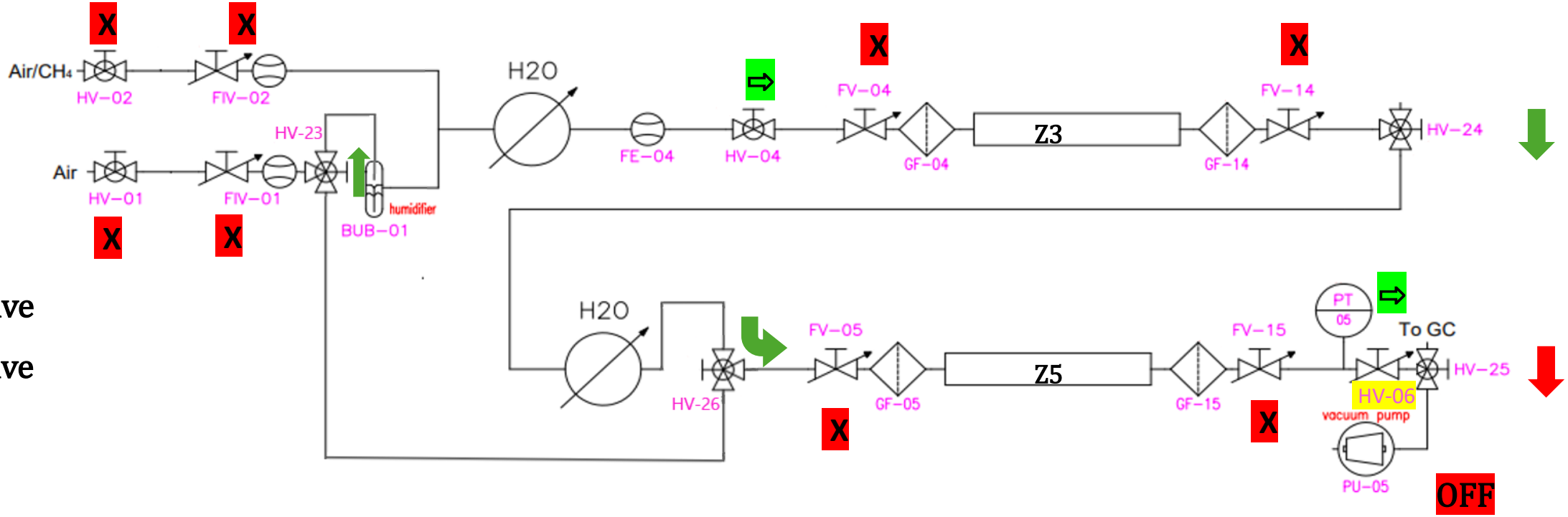
Now we prepare for regeneration switching the HV-25 to the vacuum pump

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

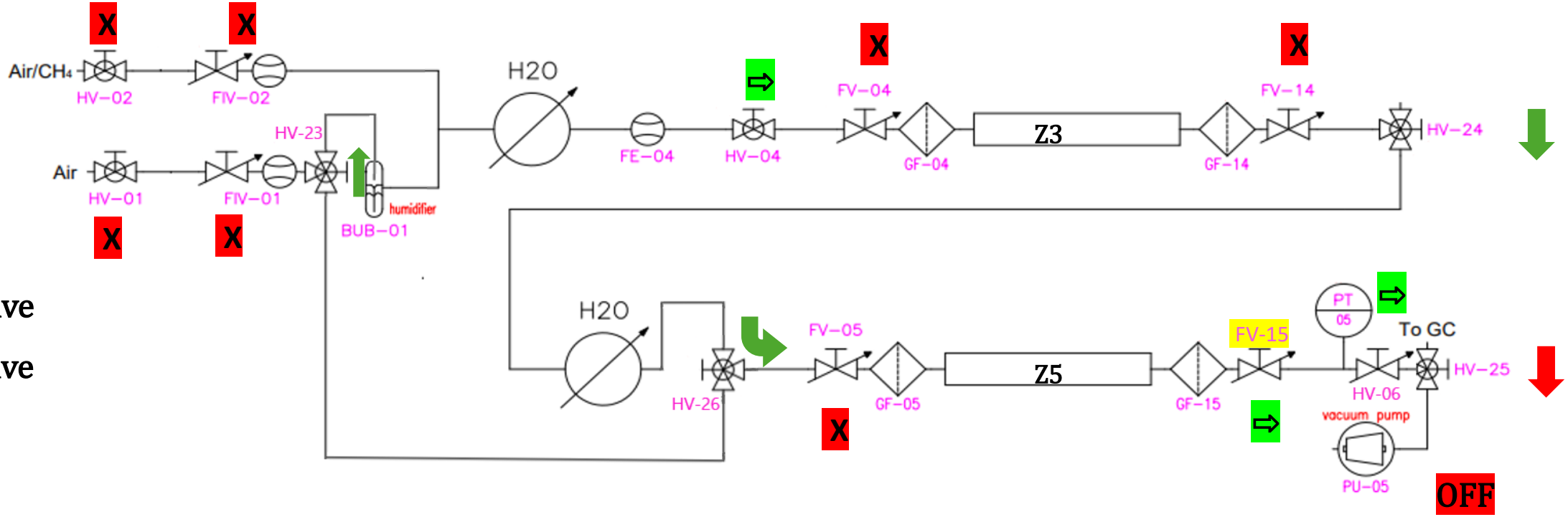
X = close valve

Now we prepare for regeneration switching the HV-25 to the vacuum pump

Then we can open the HV-06

REGENERATION PROCEDURE

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

Now we prepare for regeneration switching the HV-25 to the vacuum pump

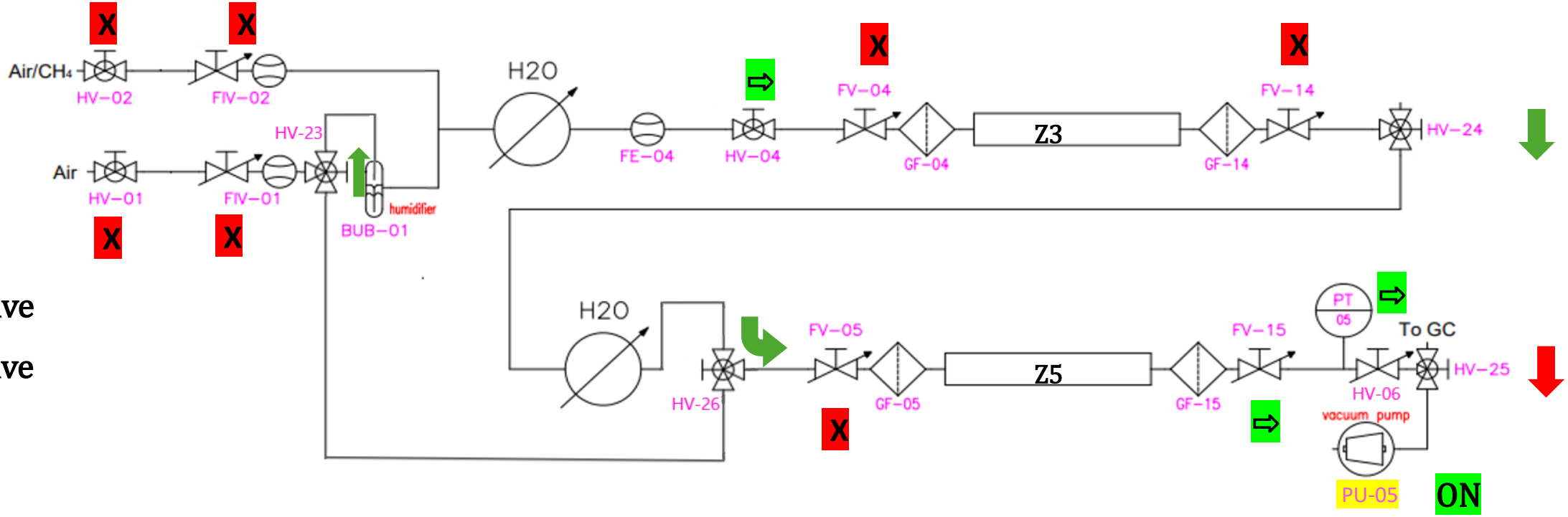
Then we can open the HV-06 and FV-15

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We are ready for **vacuum regeneration** and we turn ON the pump

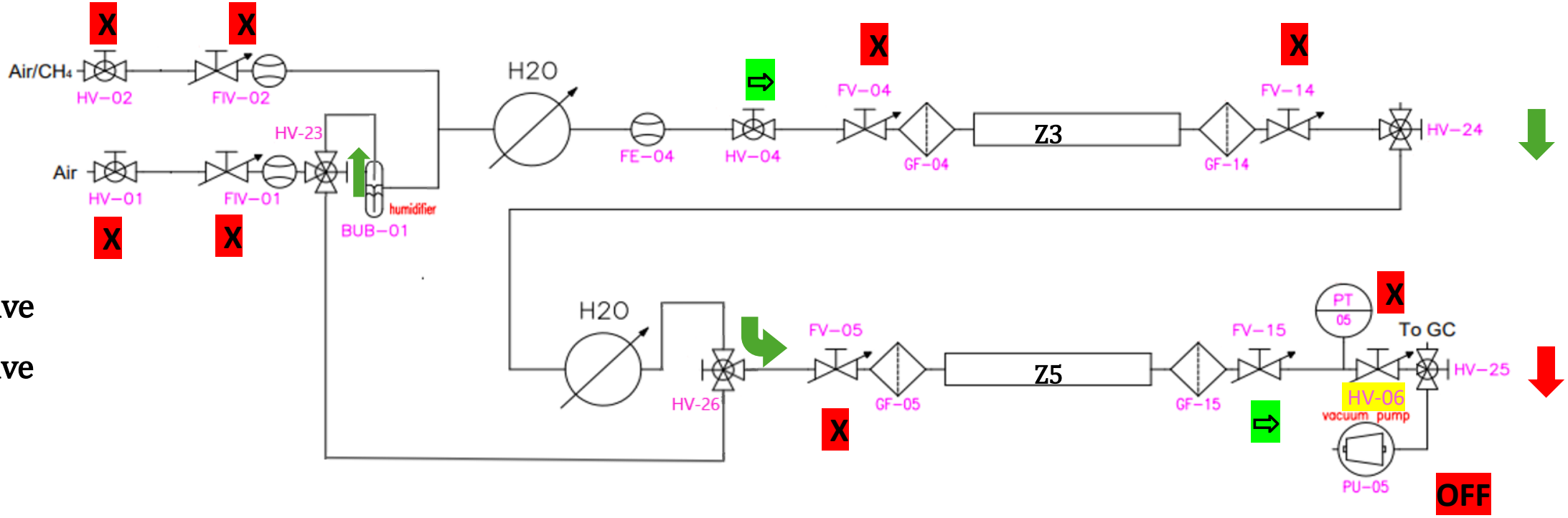
40 minutes regeneration

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

After regeneration we close the HV-06 and turn OFF the vacuum pump

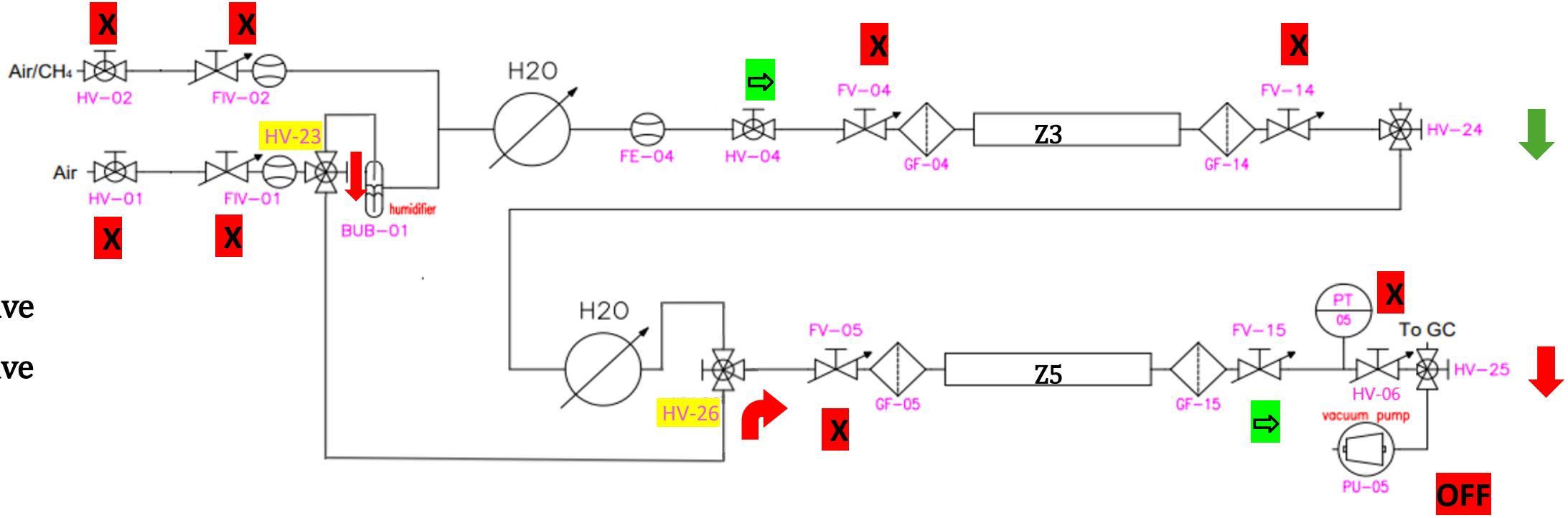
Pressure inside the Z5 cartridge $\mathrm{P} = \ 2\ mbar$

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We set the bypass line to flow Air inside the cartridge to get atmospheric pressure.

Switch HV-26 and HV-23

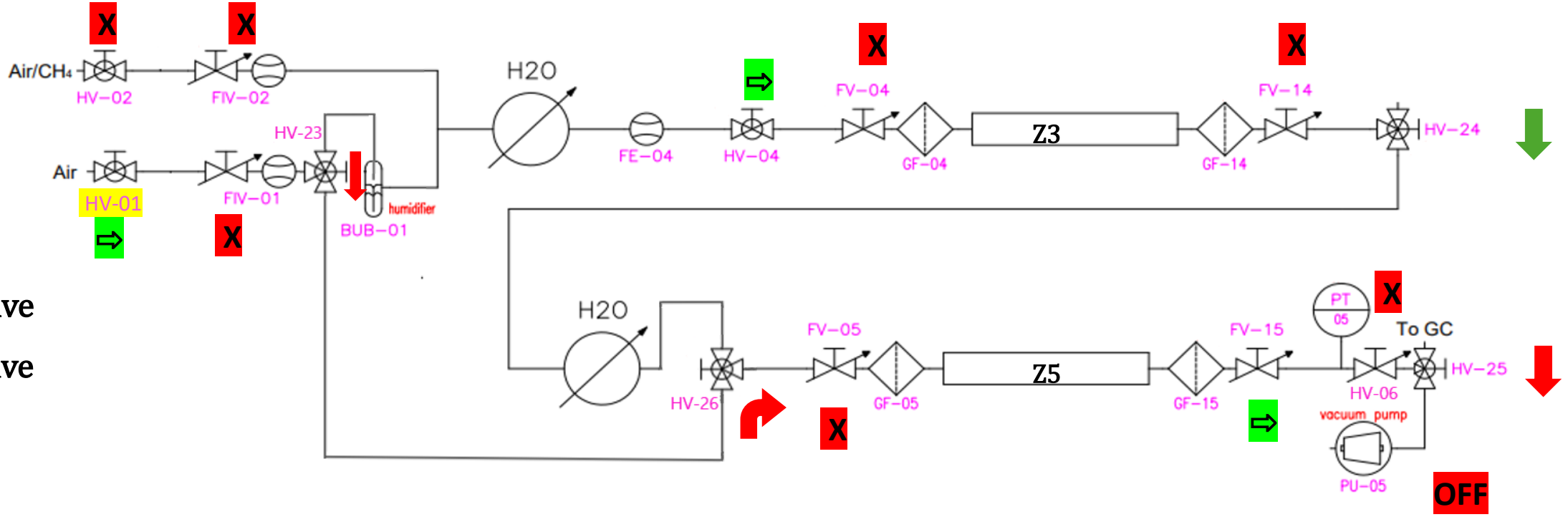
Pressure inside the Z5 cartridge $\mathrm{P} = \ 2\ mbar$

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

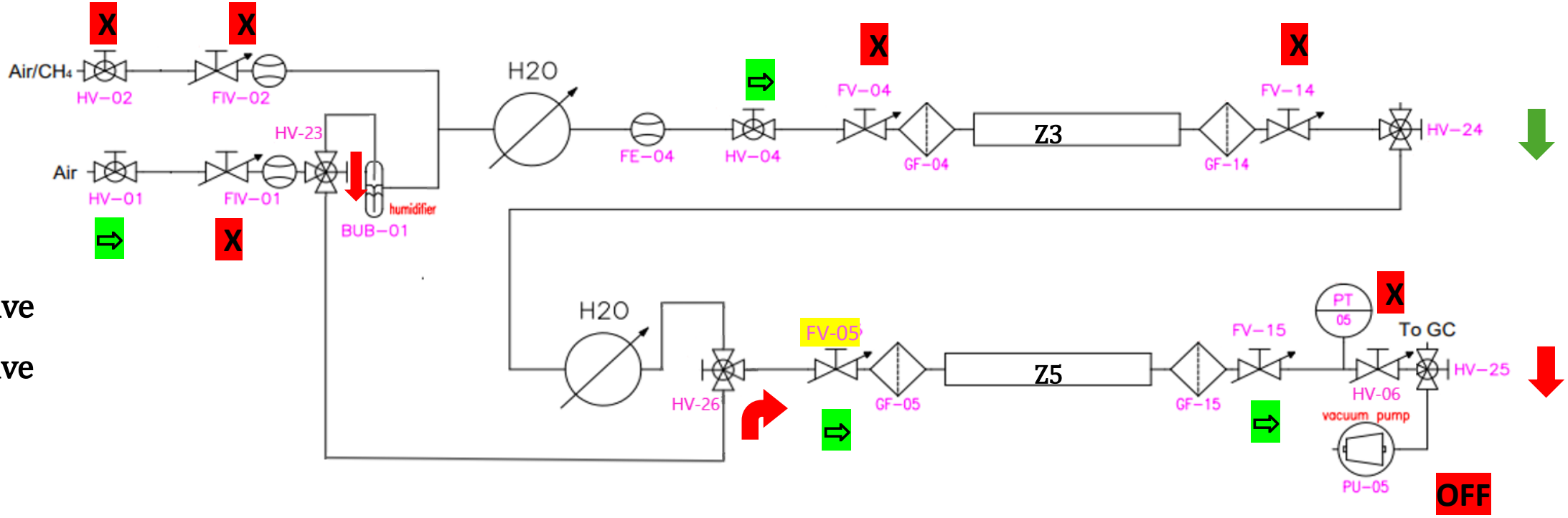
Now we open the valves between the Z5 cartridge and the air bottle

We start from the bottle HV-01 →

Pressure inside the Z5 cartridge $\mathrm{P} = 2\ mbar$

REGENERATION PROCEDURE

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

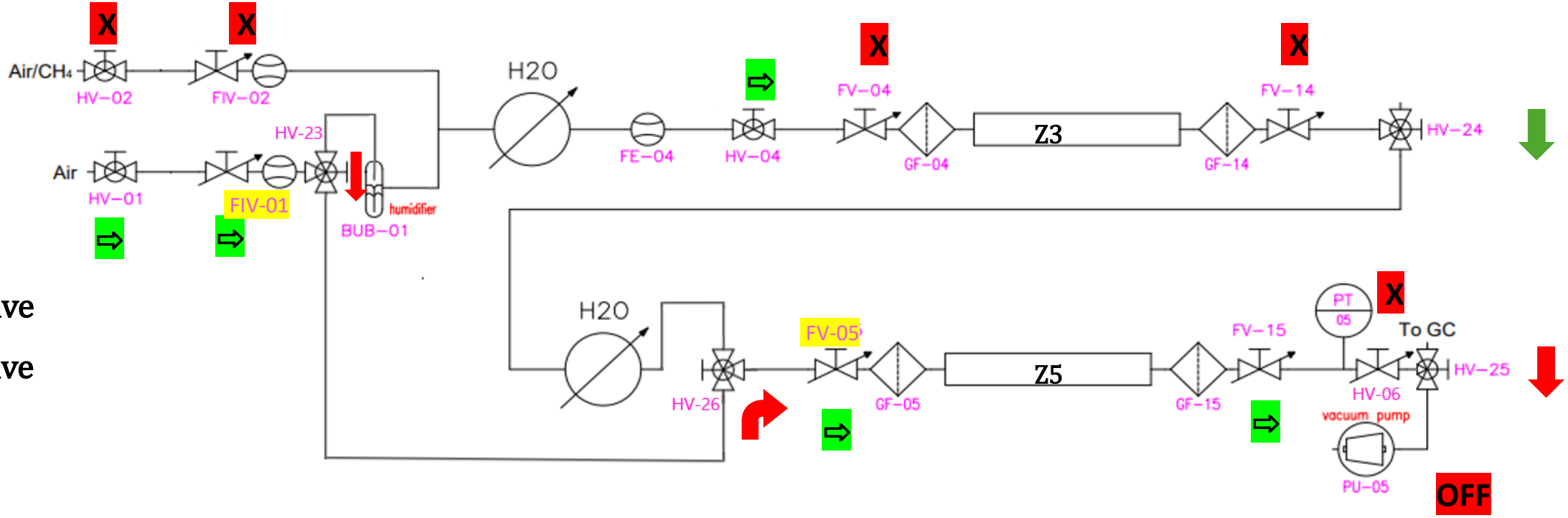
Now we open the valves between the Z5 cartridge and the air bottle

We start from the bottle HV-01 → FV-05 →

Pressure inside the Z5 cartridge $\mathrm{P} = 2\,mbar$

REGENERATION PROCEDURE

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

Now we open the valves between the Z5 cartridge and the air bottle

We start from the bottle HV-01 → FV-05 → FIV-01

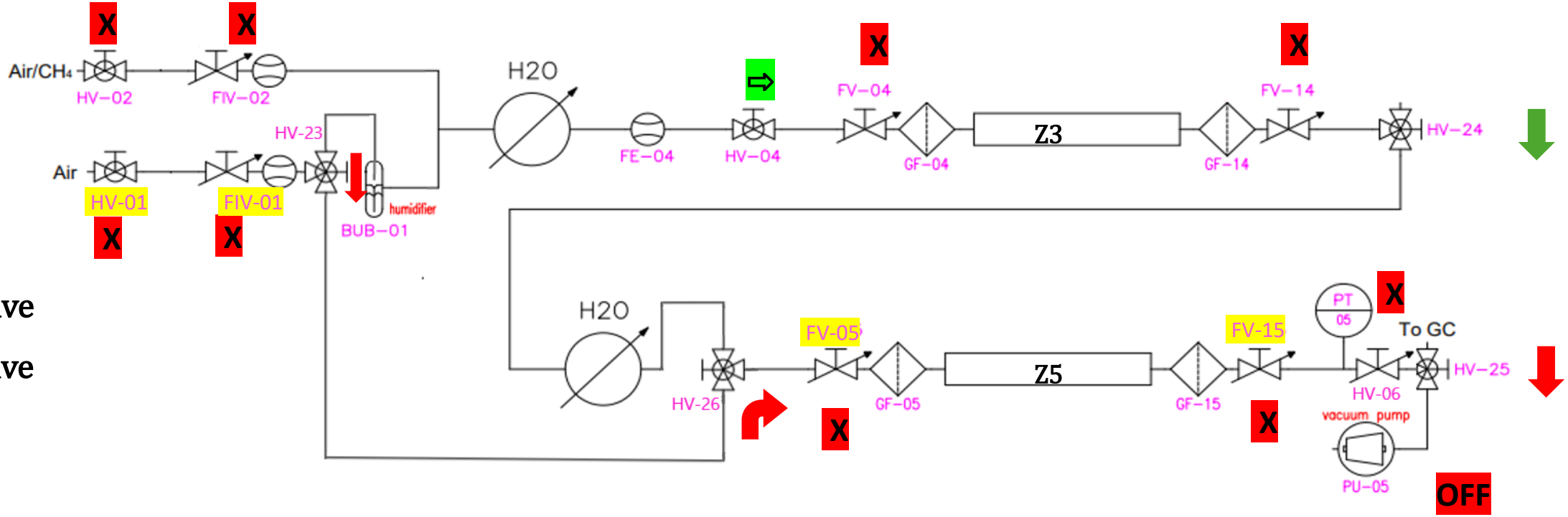
Pressure inside the cartridge rises in ~ 15 minutes to atmospheric pressure

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When target pressure is reached, we close all valves:

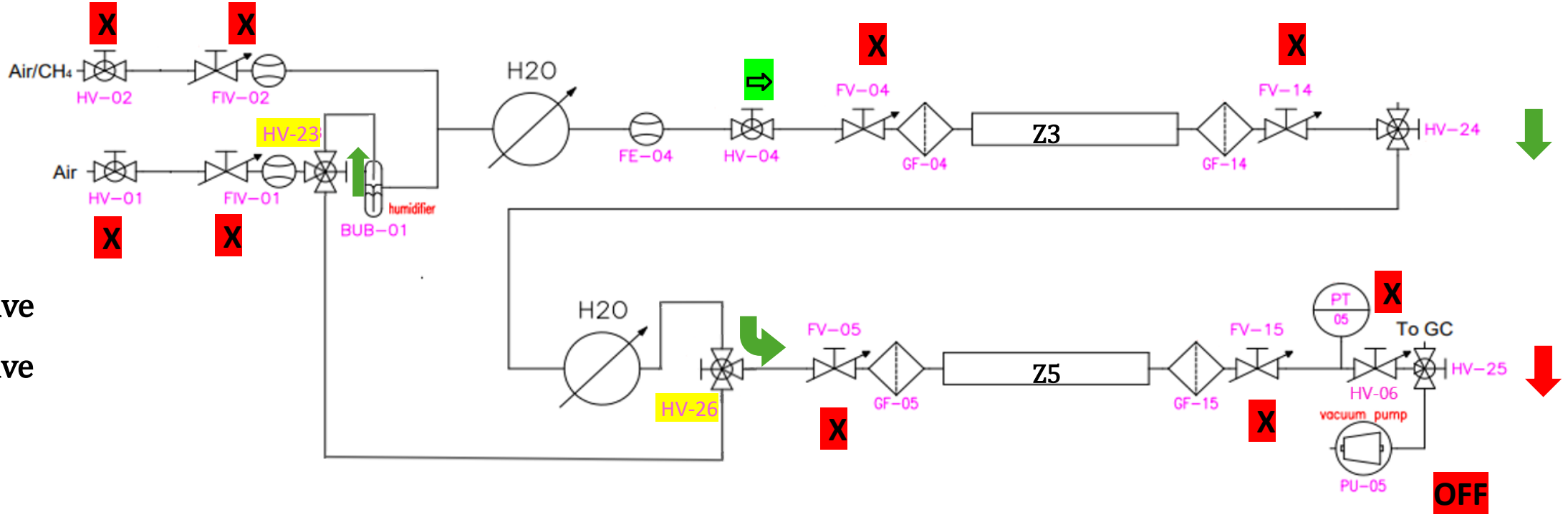
FV-05 → FV-15 → FIV-01 → HV-01

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

When target pressure is reached, we close all valves:

FV-05 → FV-15 → FIV-01 → HV-01

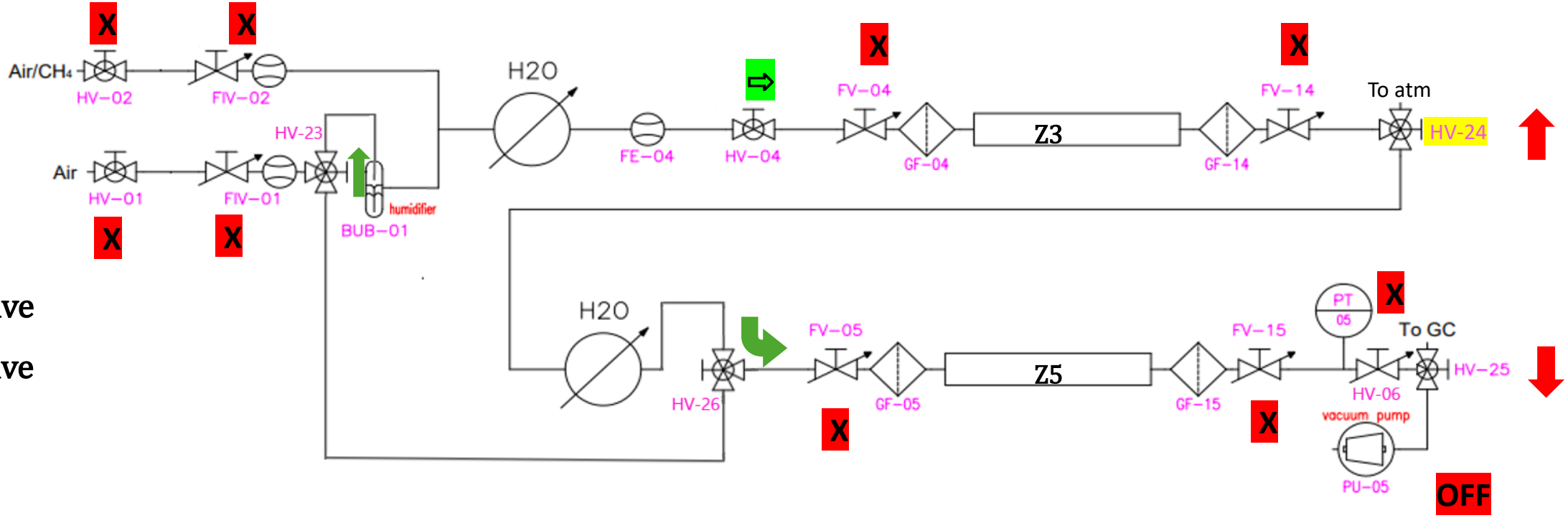
We switch the HV-23 and HV-26

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

Before starting new run we ensured the correct composition of the mixture **H2O+CH4**

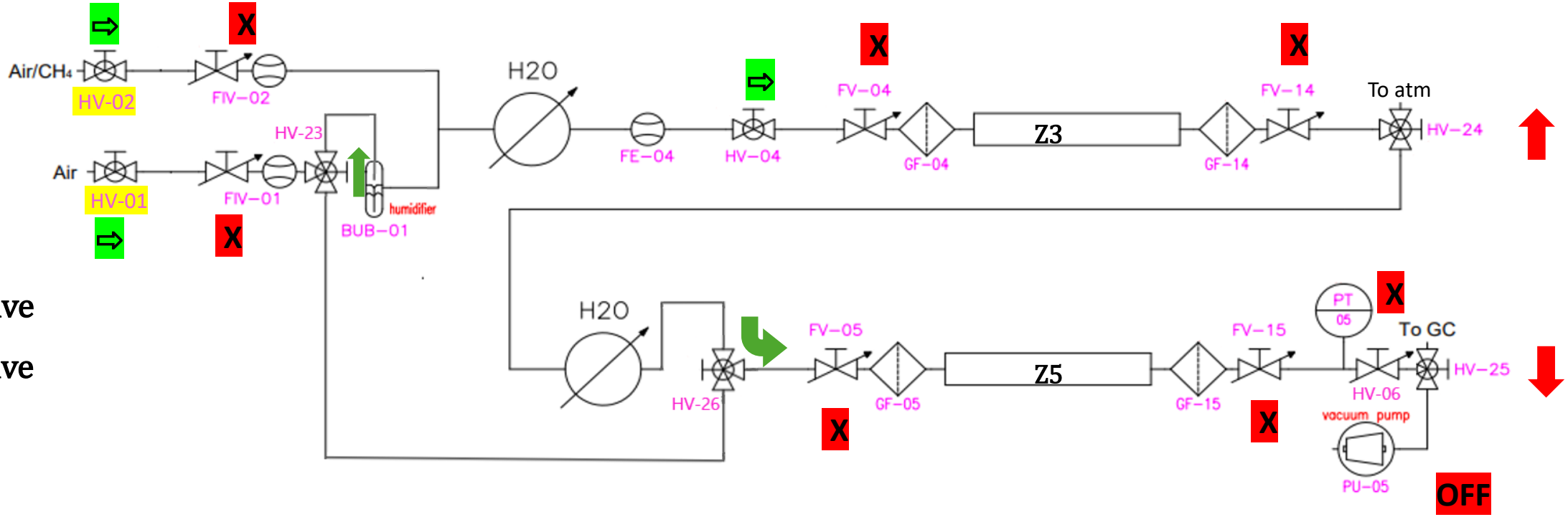
We switch HV-24 to atmosphere and then we open the valves to release the mixture after the Z3 cartridge

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



We open the bottles and the cartridge to obtain a mixture about 50/50 of H2O and CH4

The composition is set by rotameters FIV-01 and FIV-02

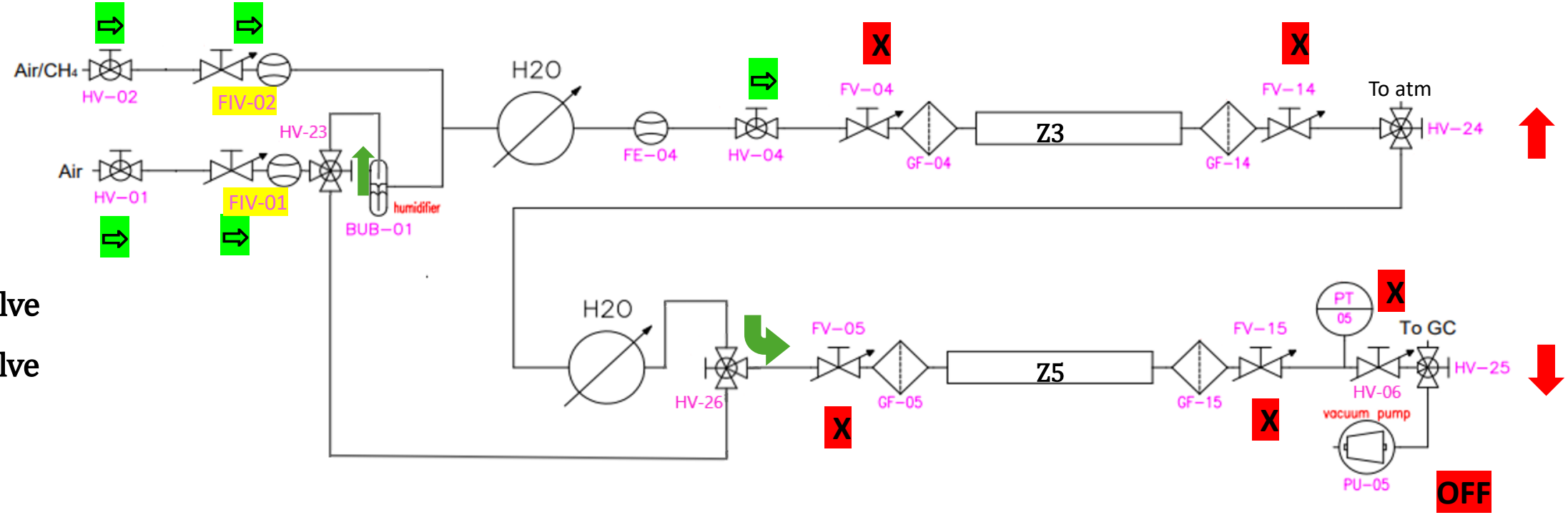
HV-02 → HV-01 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We open the bottles and the cartridge to obtain a mixture about 50/50 of H2O and CH4

The composition is set by rotameters FIV-01 and FIV-02

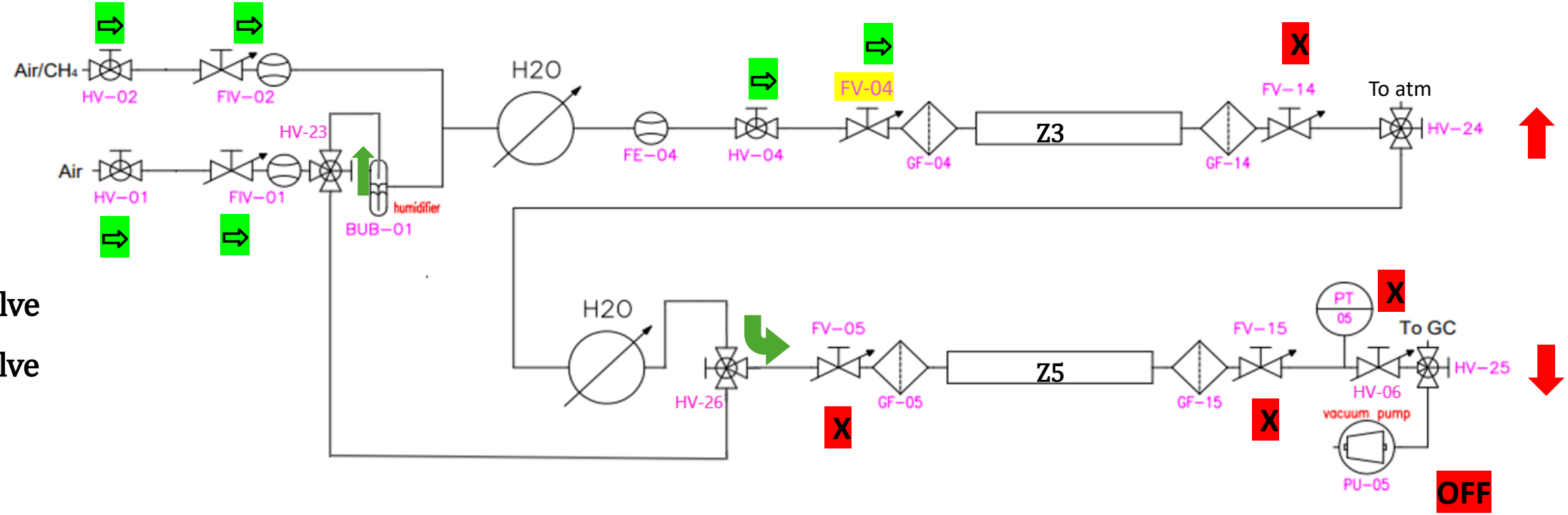
HV-02 → HV-01 → FIV-01 → FIV-02 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We open the bottles and the cartridge to obtain a mixture about 50/50 of H2O and CH4

The composition is set by rotameters FIV-01 and FIV-02

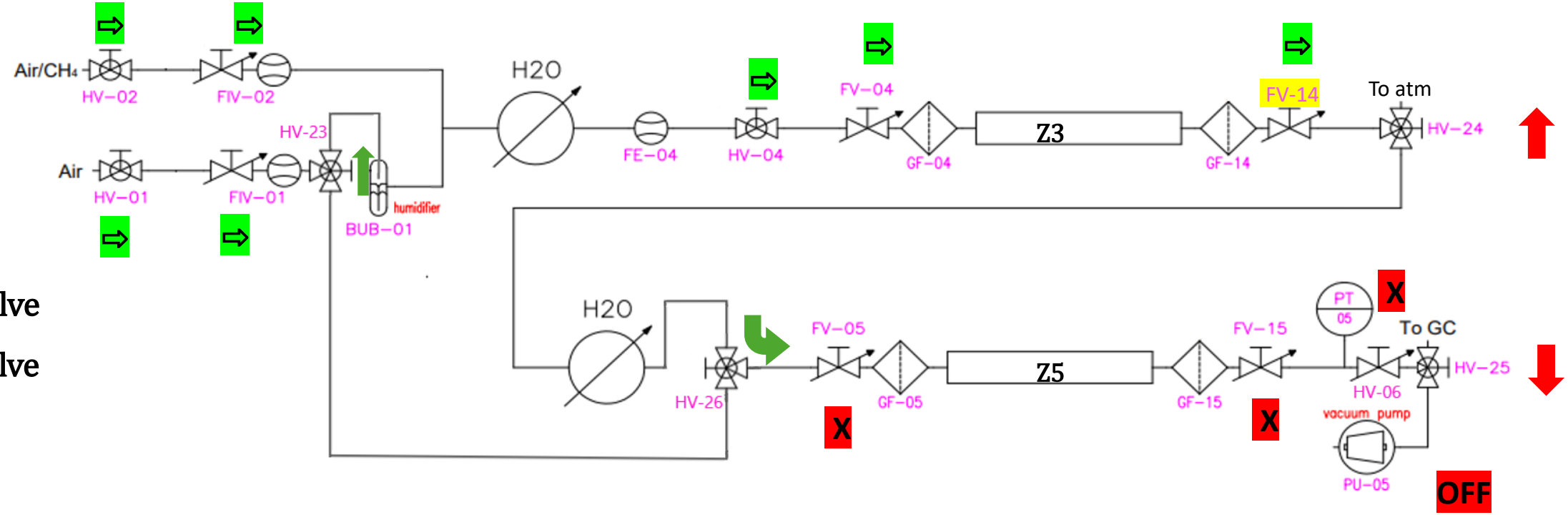
HV-02 → HV-01 → FIV-01 → FIV-02 → FV-04 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We open the bottles and the cartridge to obtain a mixture about 50/50 of H2O and CH4

The composition is set by rotameters FIV-01 and FIV-02

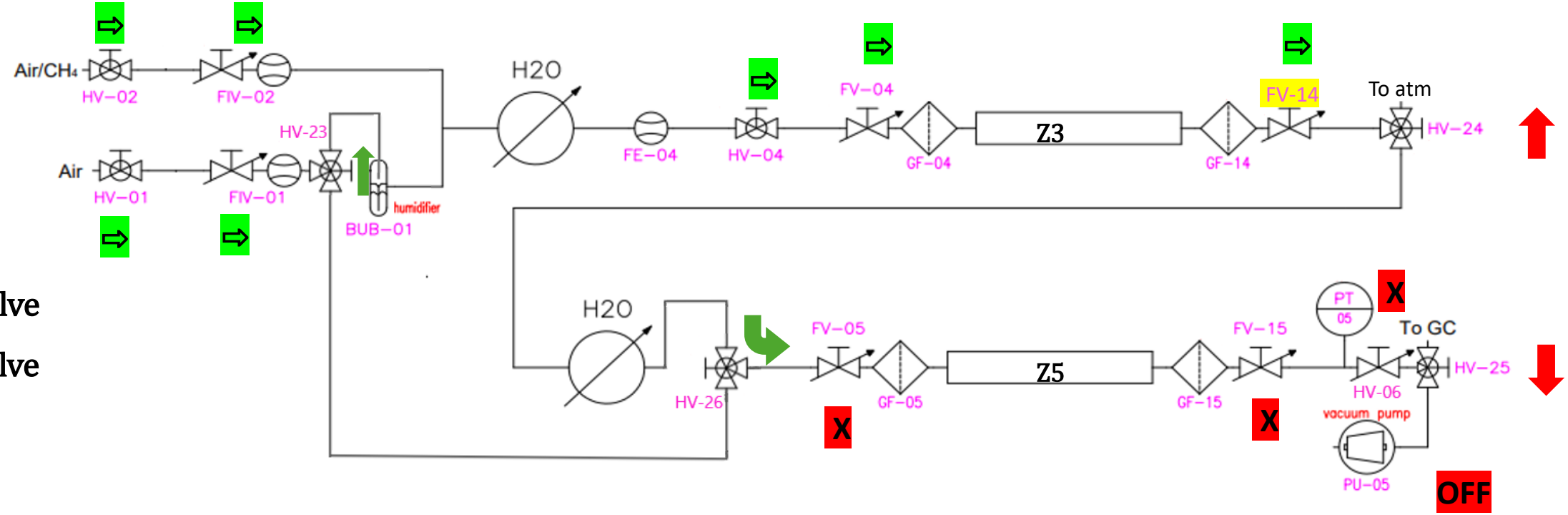
HV-02 → HV-01 → FIV-01 → FIV-02 → FV-04 → FV-14

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

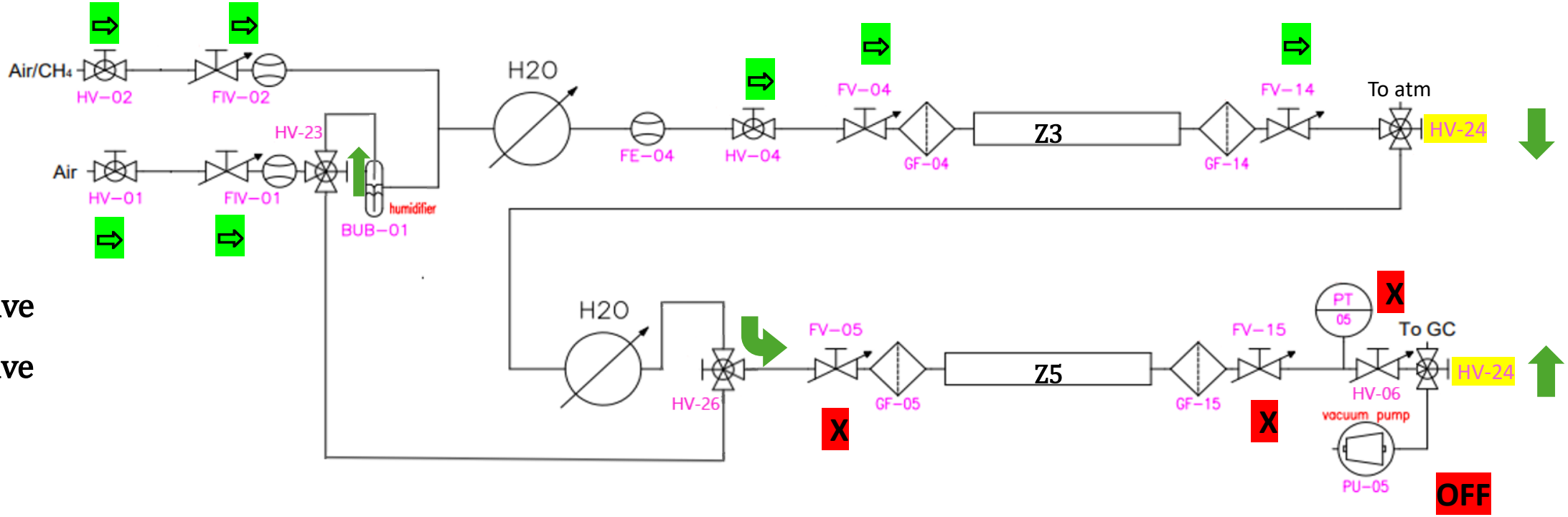
We open the bottles and the cartridge to obtain a mixture about 50/50 of H2O and CH4

The composition is set by rotameters FIV-01 and FIV-02

HV-02 → HV-01 → FIV-01 → FIV-02 → FV-04 → FV-14

REGENERATION PROCEDURE

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

Once the mixture is obtained we switch HV-24 and HV-25

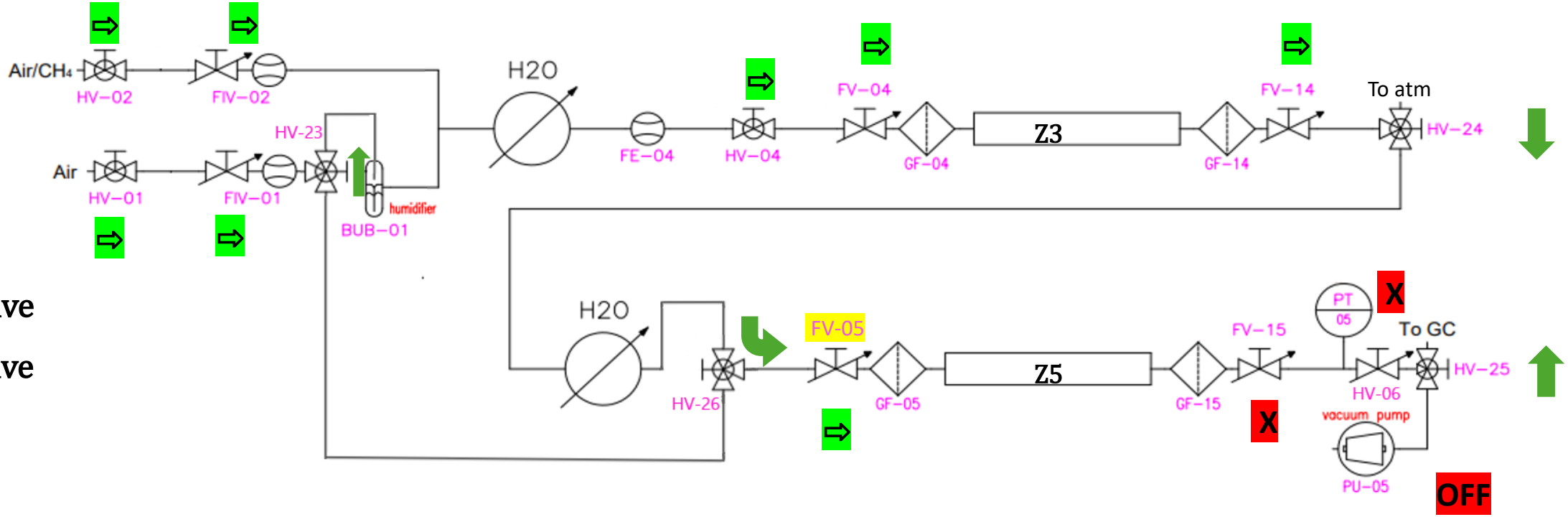
Then we can open the valves of the Z5 cartridge and begin to flow CH4 into it

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We now open the valves:

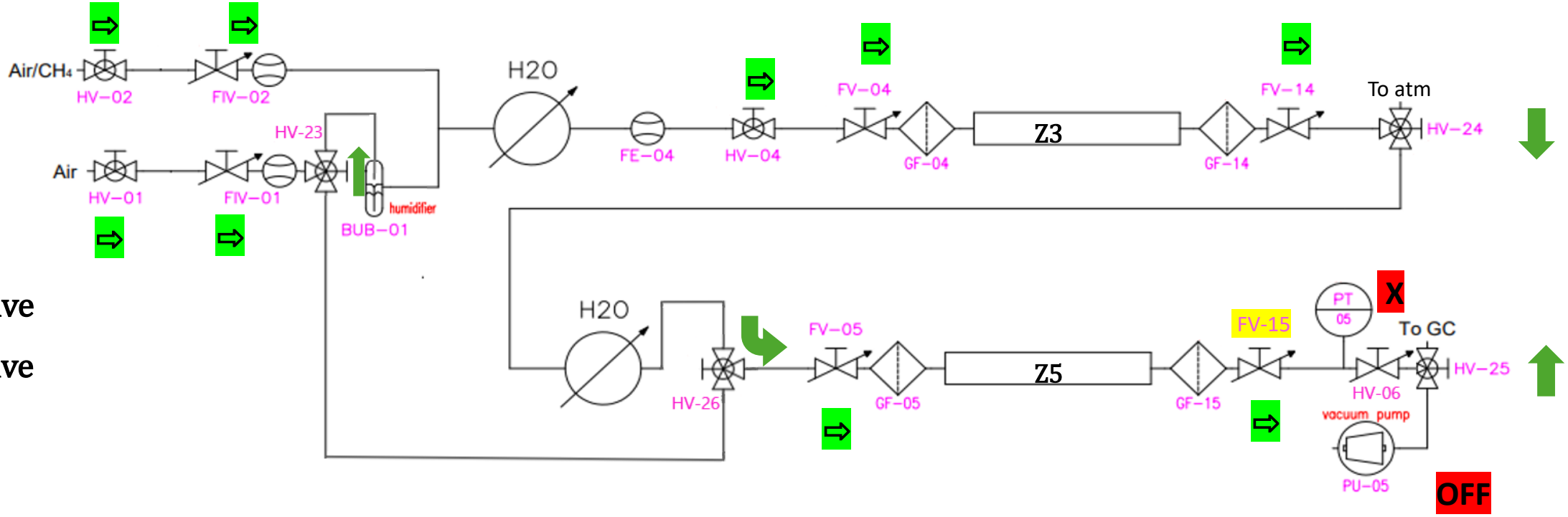
FV-05 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

We now open the valves:

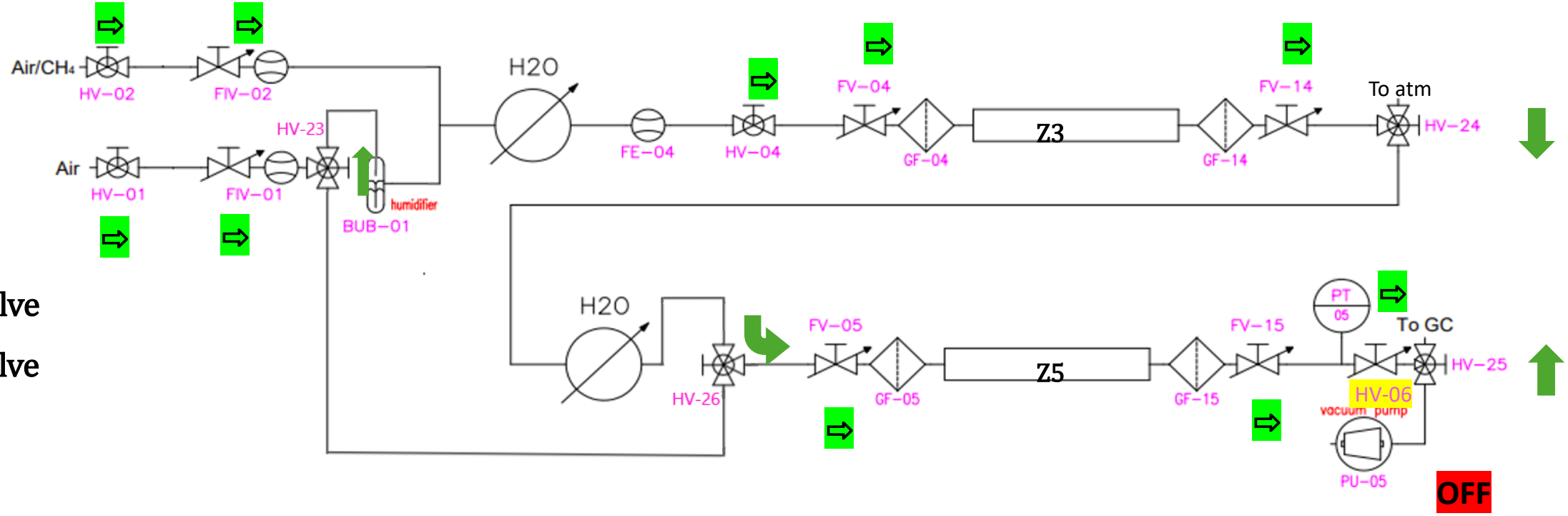
FV-05 → FV-15 →

REGENERATION PROCEDURE

UNIVERSITÀ DI PAVIA

INFN CERN EP-DT Detector Technologies

Measurement **H2O+CH4 and Z3+Z5:**



⇨ = open valve

X = close valve

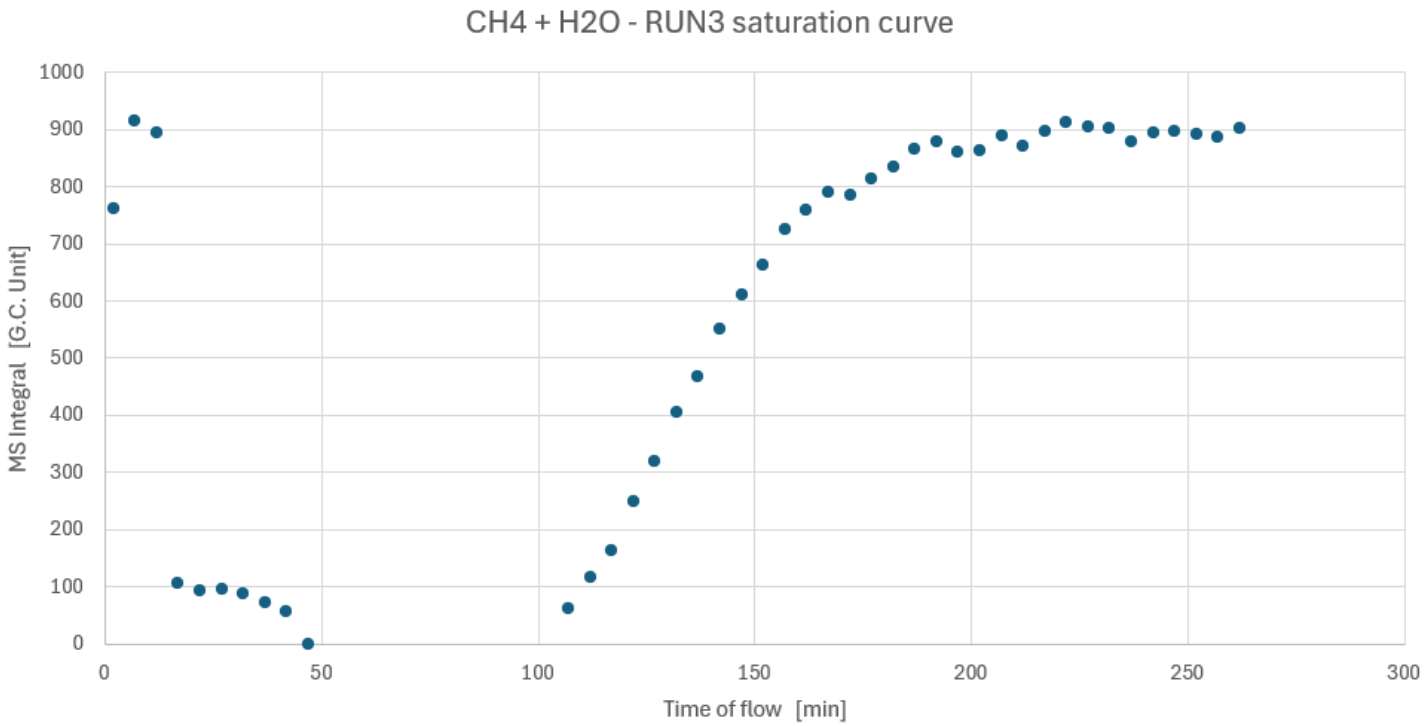
We now open the valves:

FV-05 → FV-15 → HV-06 → GC analysis

New RUN started

REGENERATION PROCEDURE

Example of saturation curve with this method:



First 3 analyses:

CH4 left in the line between the cartridge and GC

Adsorbed methane is calculated ignoring the first analyses

RESULTS EXPLANATION ?

	Z5 - only CH4		Z3 + Z5 - H2O + CH4	
	At saturation	Before first peak	At saturation	Before first peak
Mean Volume of CH4 adsorbed	23.6 mL	12.6 mL	12.3 mL	9.51 mL

We are not able to explain this difference

No time shift or delay in GC analyses

Possible explainations:

- Effects that were not considered (?)
- Adsorbtion property change with concentration (?)
- Calculations suppose adsorbtion is linear during time
- Flow is measured only before the Z3 cartridge

QUESTIONS AND DOUBTS

MORE DOUBTS:

- How does the GC works exactly ?
- Results of one GC analysis is before or during the 5 minutes interval ?
- What error on the pressure sensor ?

  (Datasheet: 0.03 % FSO/°C → 8 mbar at 20 °C)