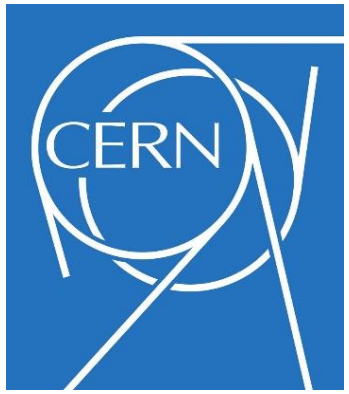
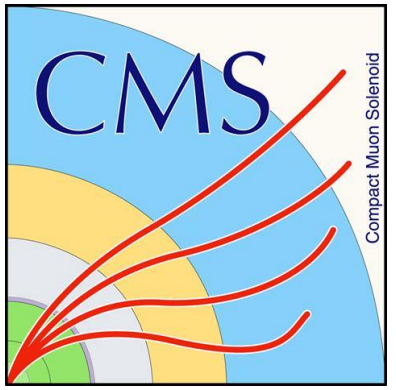


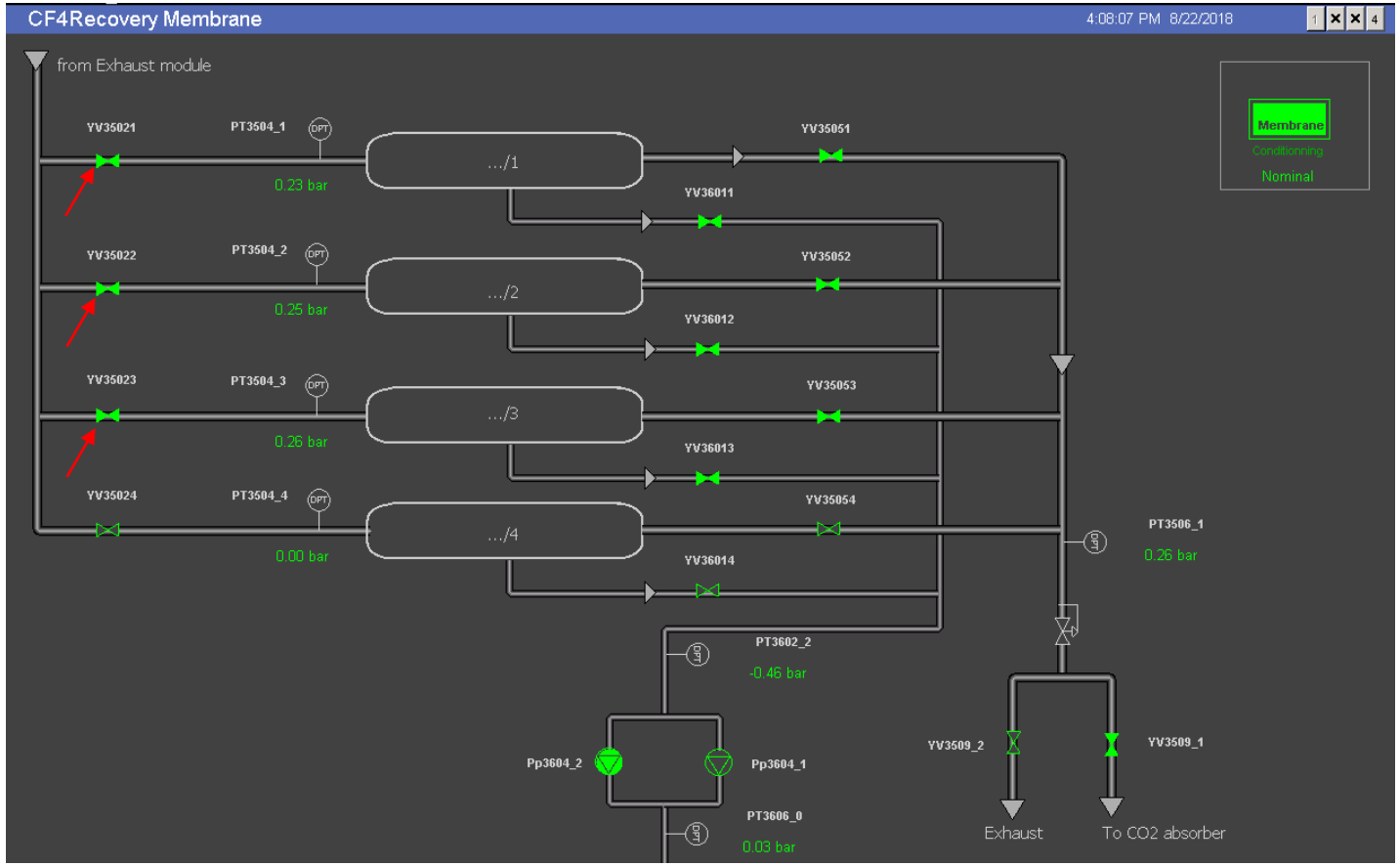


CF4 ANALYSIS

IMPROVEMENT AND CHANGING DONE AT CMS SITE

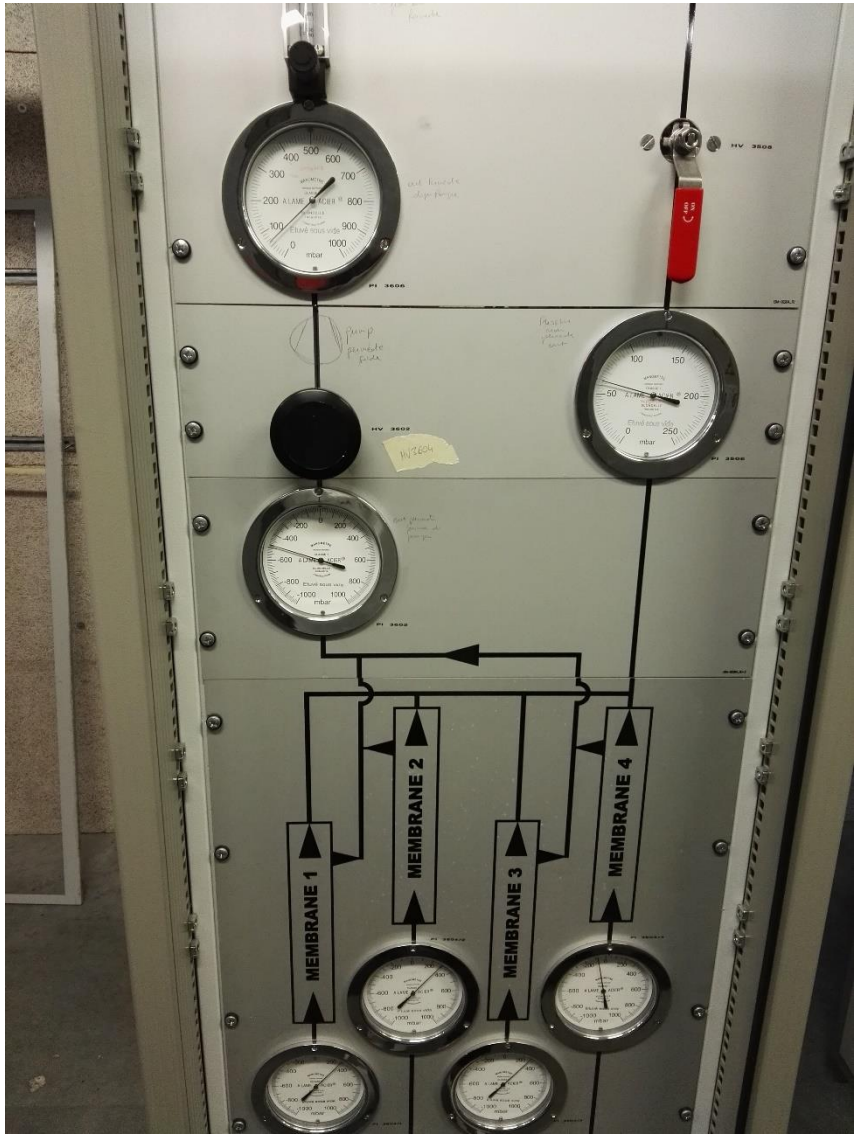
REWIEW BEFORE CHANGING PARAMETERS

Before changing parameters three membranes were involved and the flow was 600l/h NOT evenly divided between the three membranes because membrane1 had only 150l/h instead of 200l/h.



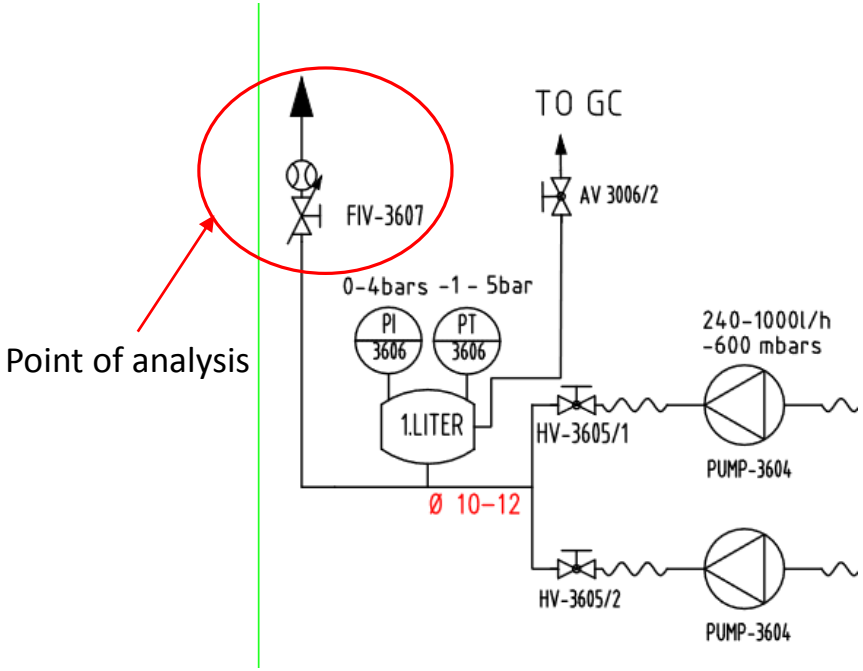
Three membranes involved

Initial flow exhaust



Situation at rack1

GC ANALYSIS WITH 3 MEMBRANES



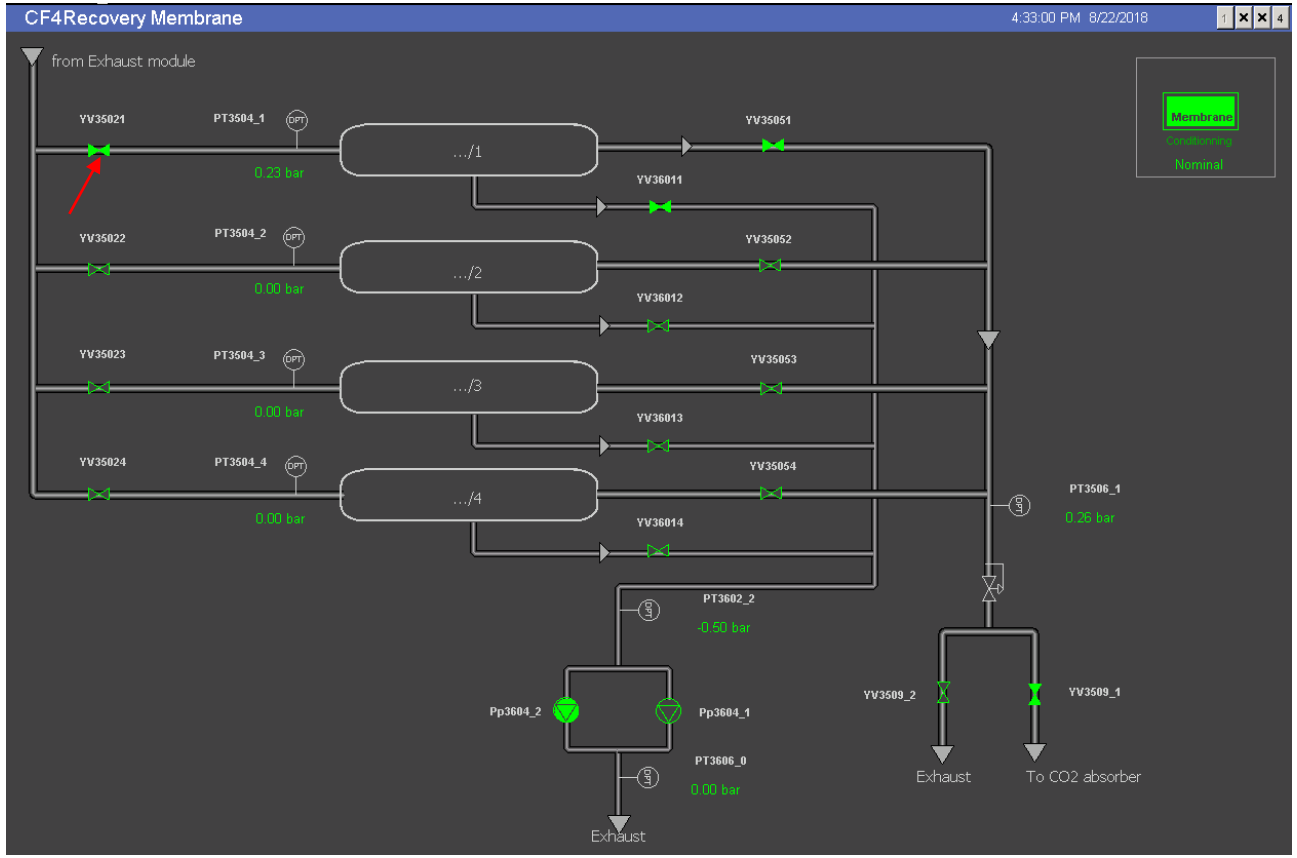
Membrane 1, 2, 3

GAS	QUANTITY
Ar	42.47%
CF4	1.50%
CO2	54.47%
O2	123 ppm
N2	7616 ppm

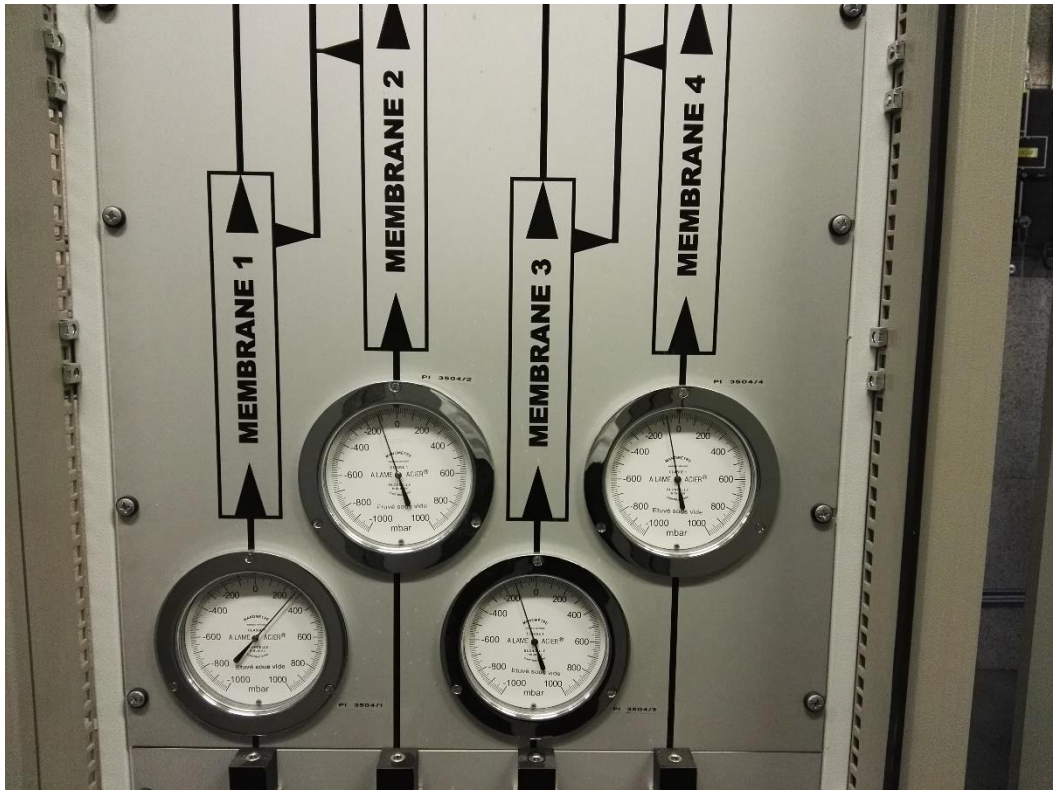
MEMBRANE 1

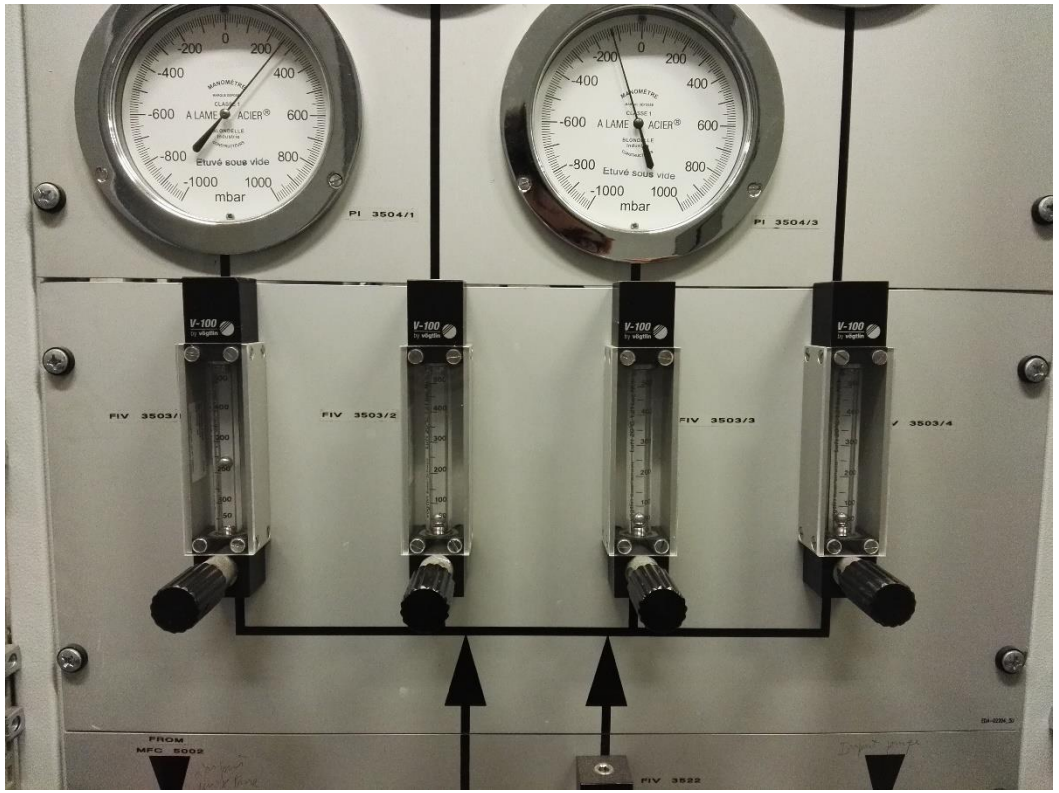
First step ---> 22/08/2018

✓ Close membrane 2 and 3. Leave only membrane one working. Then reduce flow from 600 l/h to 200 l/h to pass the same flow of when all three membranes were active.

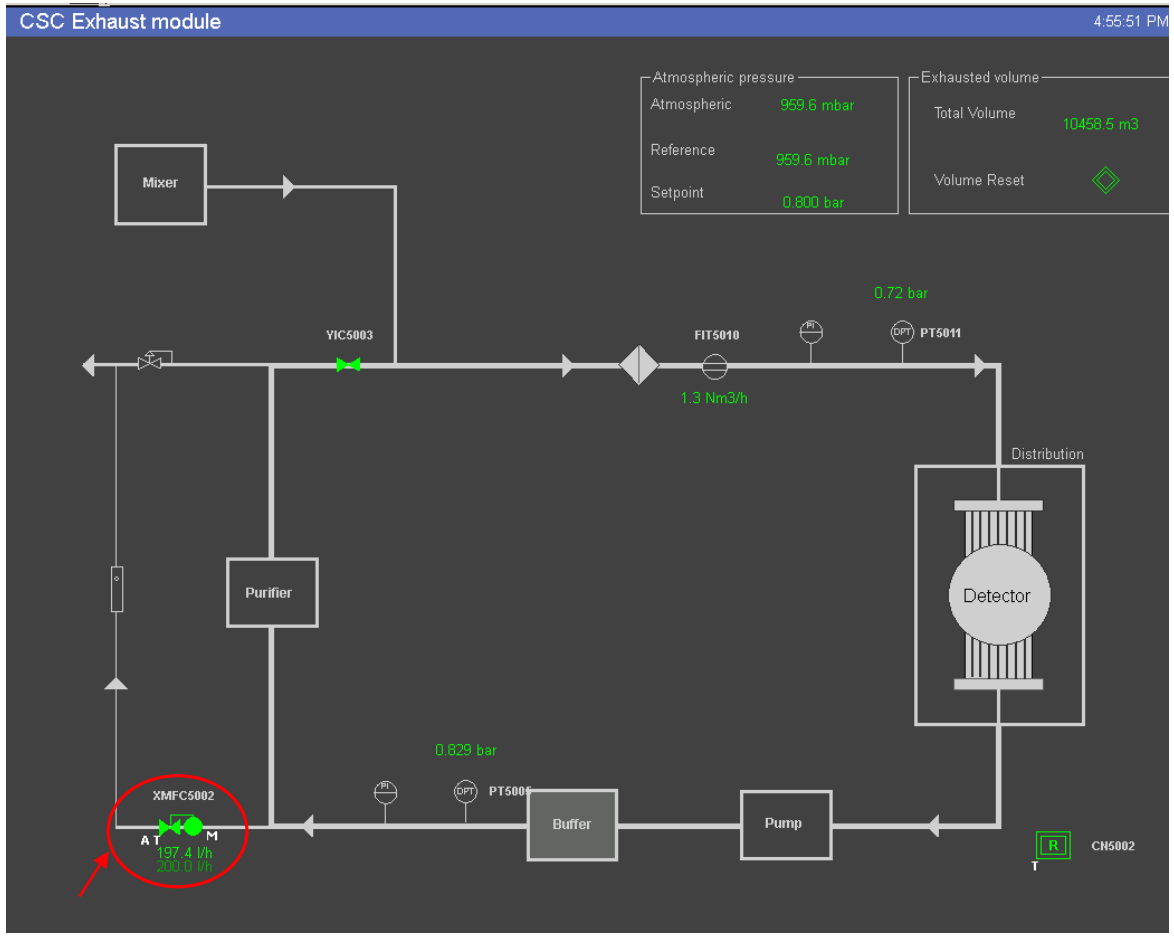


Membrane1 involved





Rack1 after changing. Only membrane 1 with flow



Flow 200l/h at exhaust

Decreasing of flow between 4pm and 5pm

GC ANALYSIS WITH M1

✓Check the efficiency of the membrane with gas chromatograph.

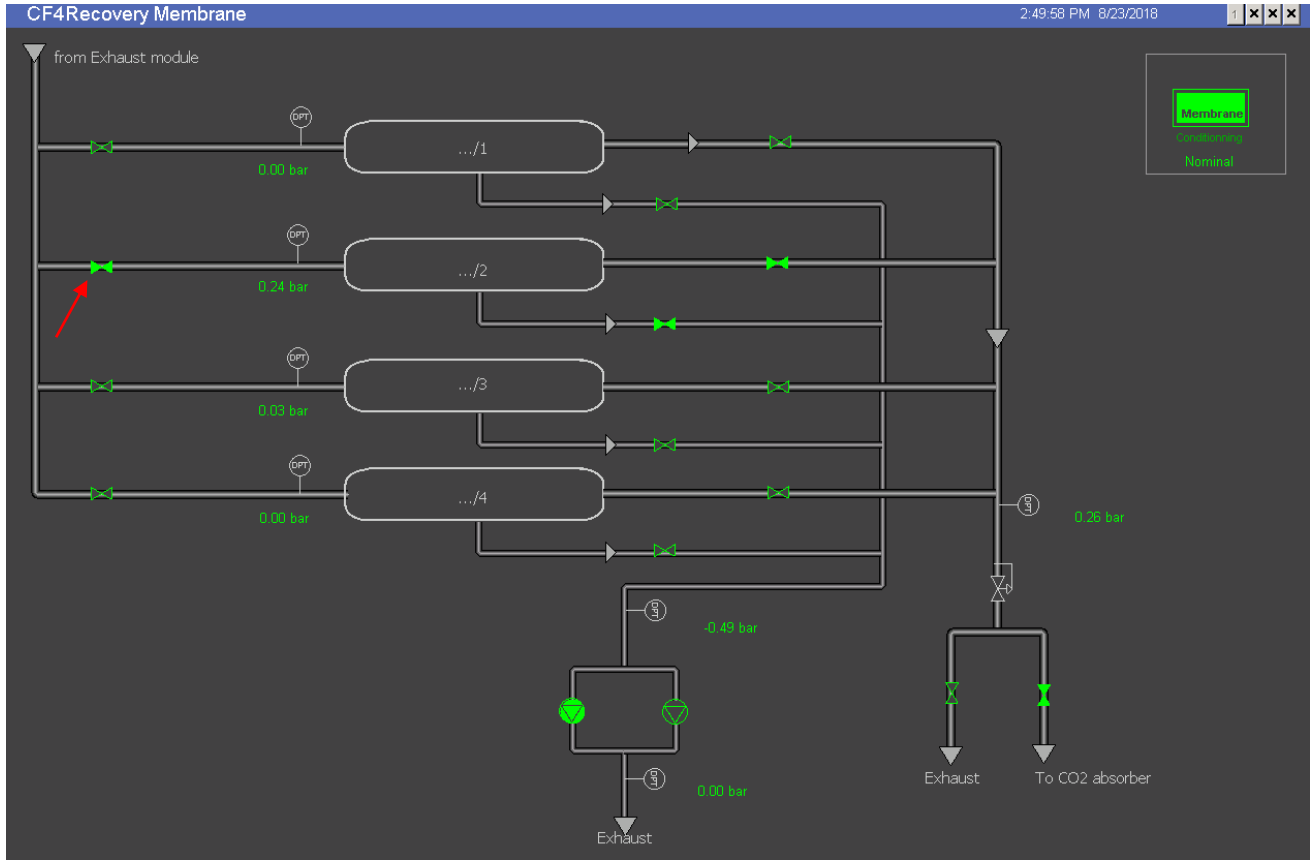
Membrane 1

GAS	QUANTITY
Ar	42.51%
CF4	2.54%
CO2	52.72%
O2	113 ppm
N2	7680 ppm

MEMBRANE 2

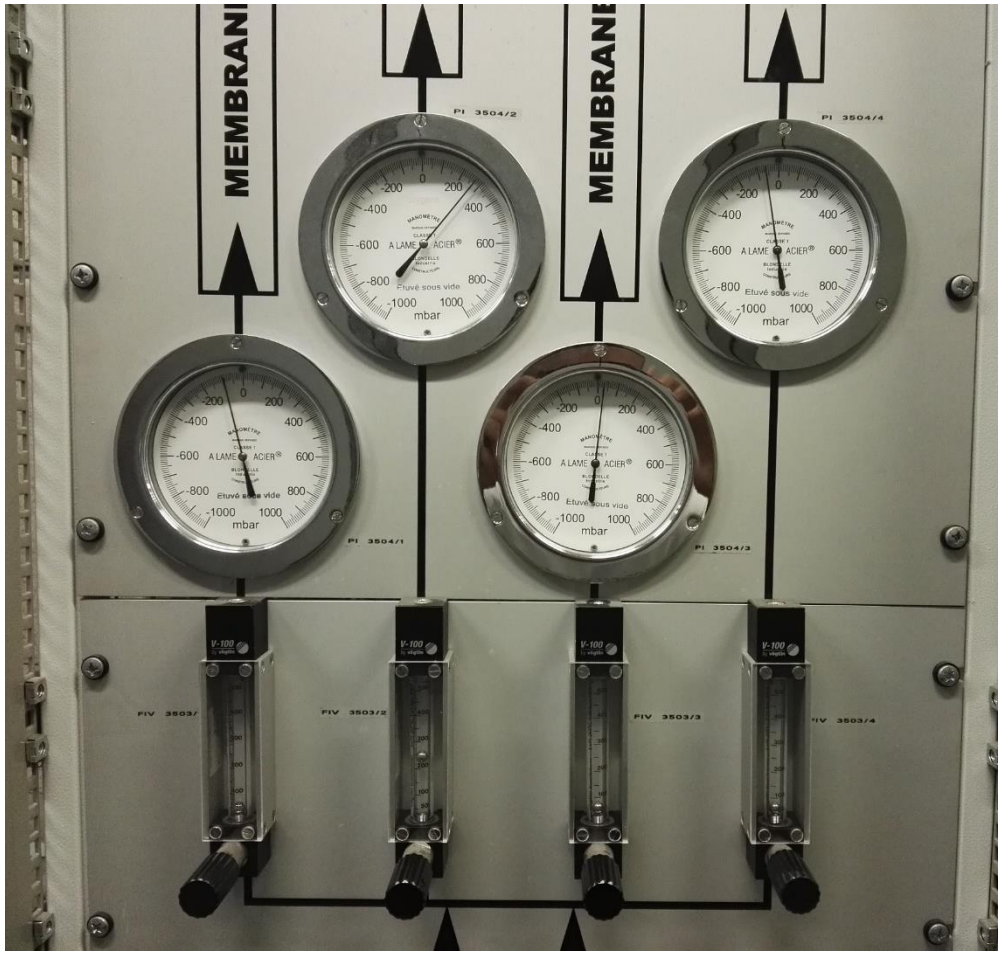
Second step ---> 23/08/2018

✓ Close membrane 1 and 3. Leave only membrane two working. Then reduce flow from 600 l/h to 200 l/h to pass the same flow of when all three membranes were active. The same process of M1.

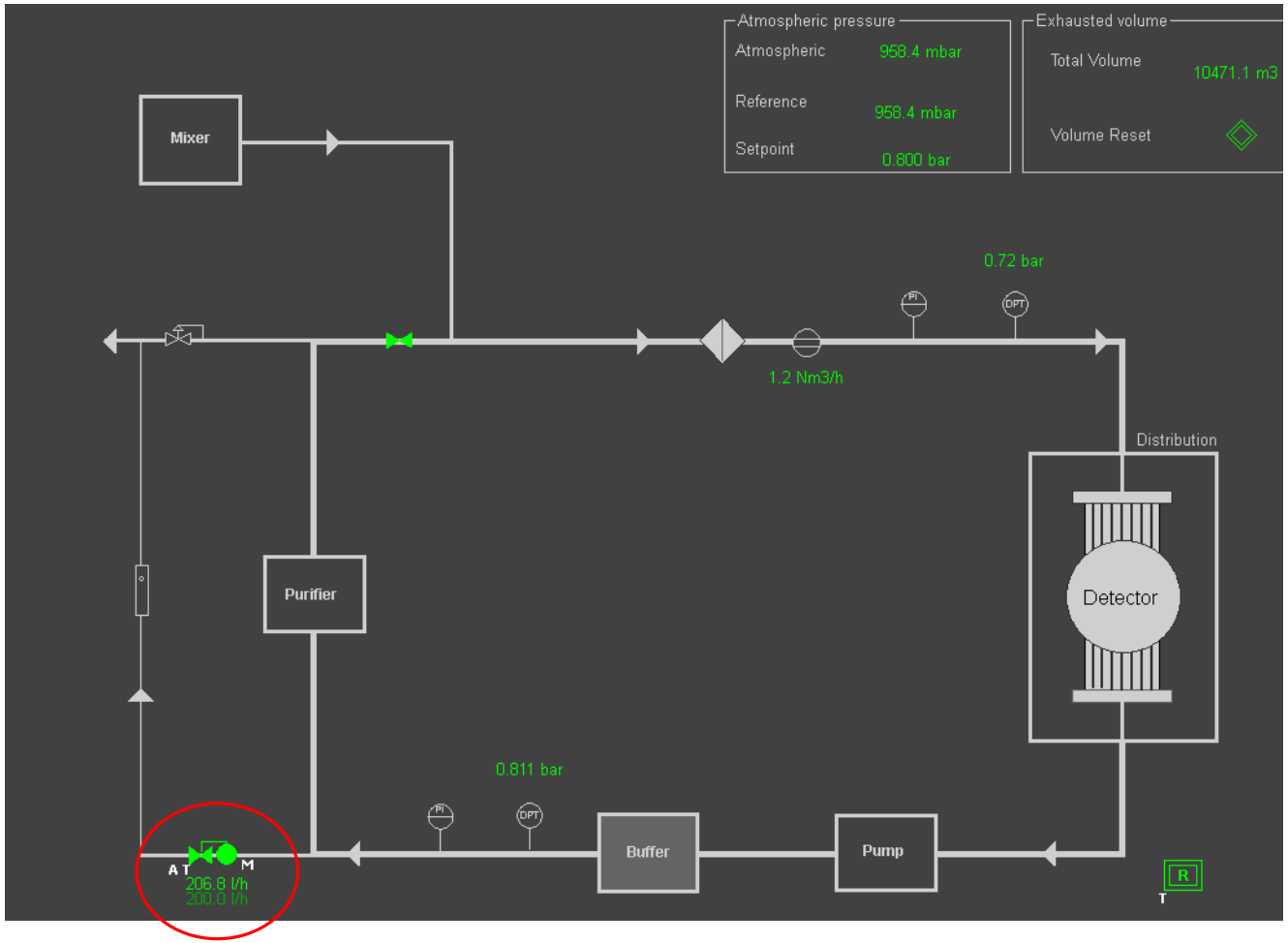


Membrane2 involved

Only membrane2 in function.



Exhaust with M2



GC ANALYSIS WITH M2

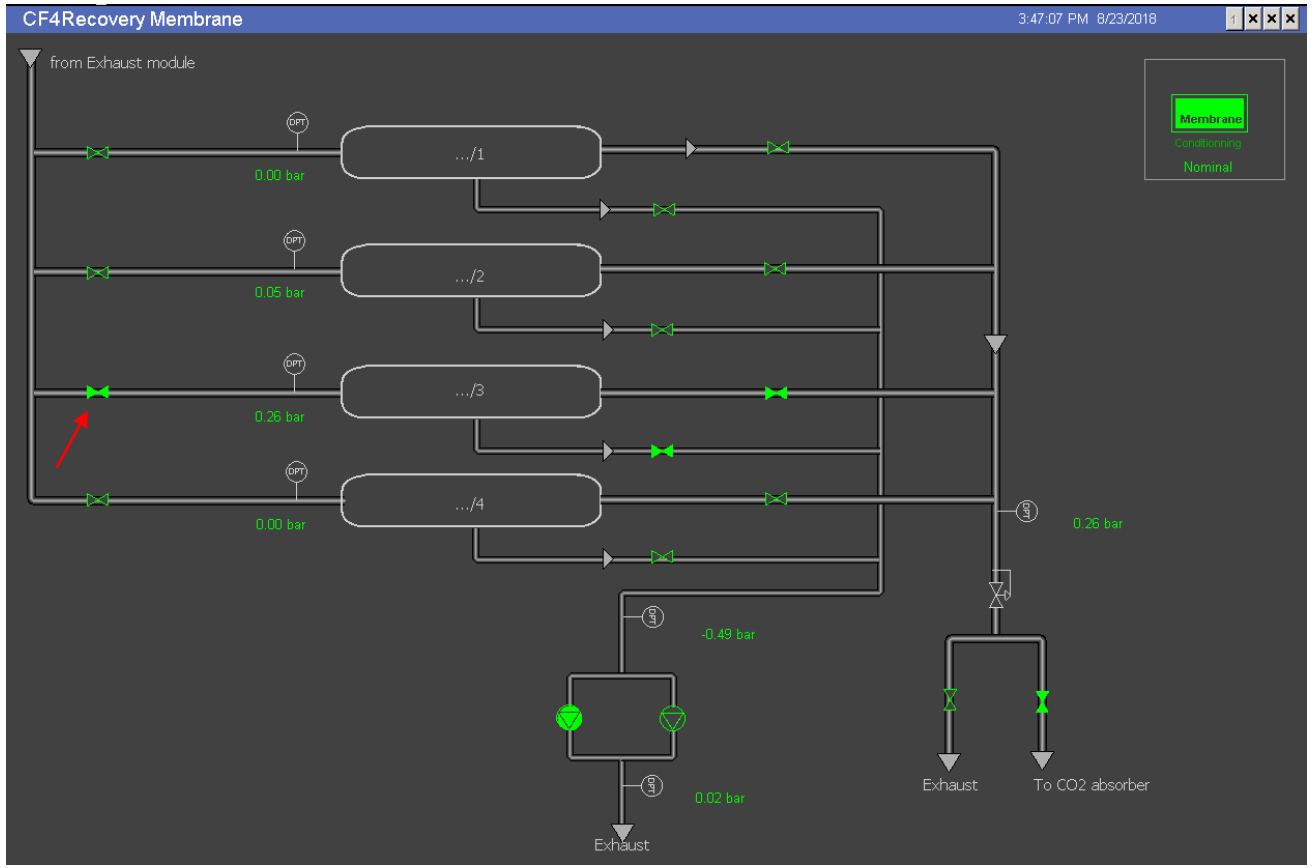
Membrane 2

GAS	QUANTITY
Ar	43.08%
CF4	0.60%
CO2	55.70%
O2	127 ppm
N2	7620 ppm

MEMBRANE 3

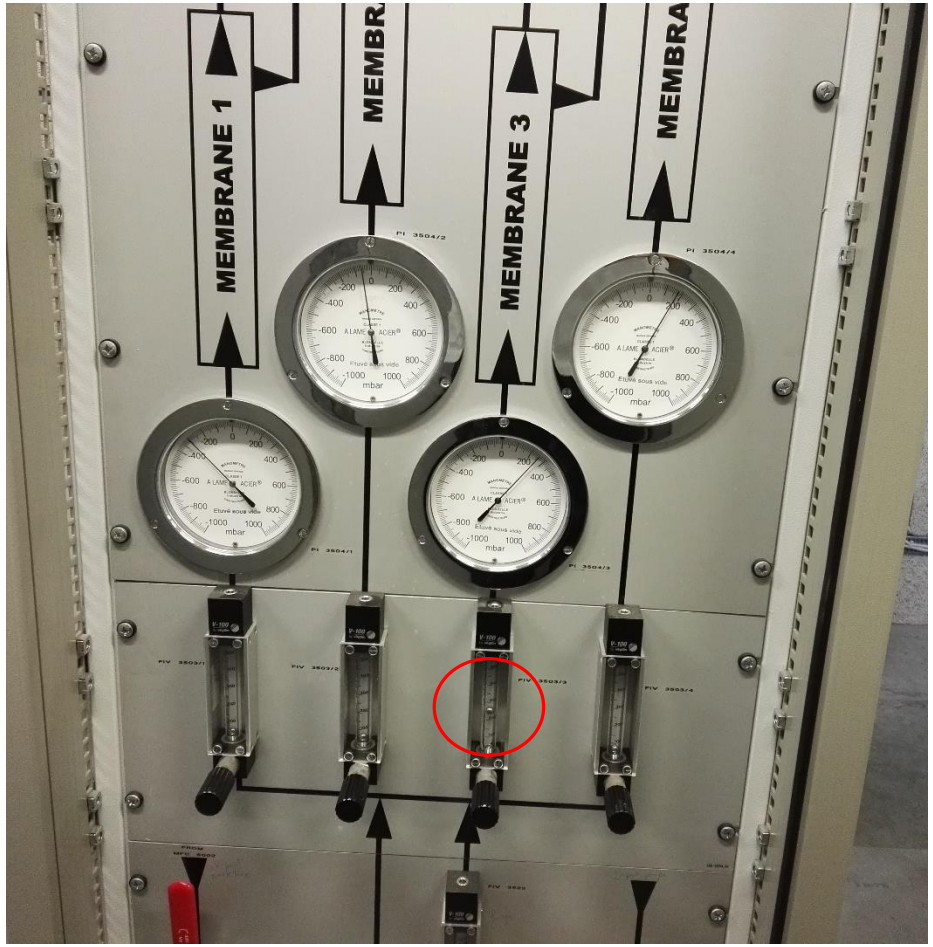
Third step ---> 23/08/2018

✓ Close membrane 1 and 3. Leave only membrane three working. Then reduce flow from 600 l/h to 200 l/h to pass the same flow of when all three membranes were active.

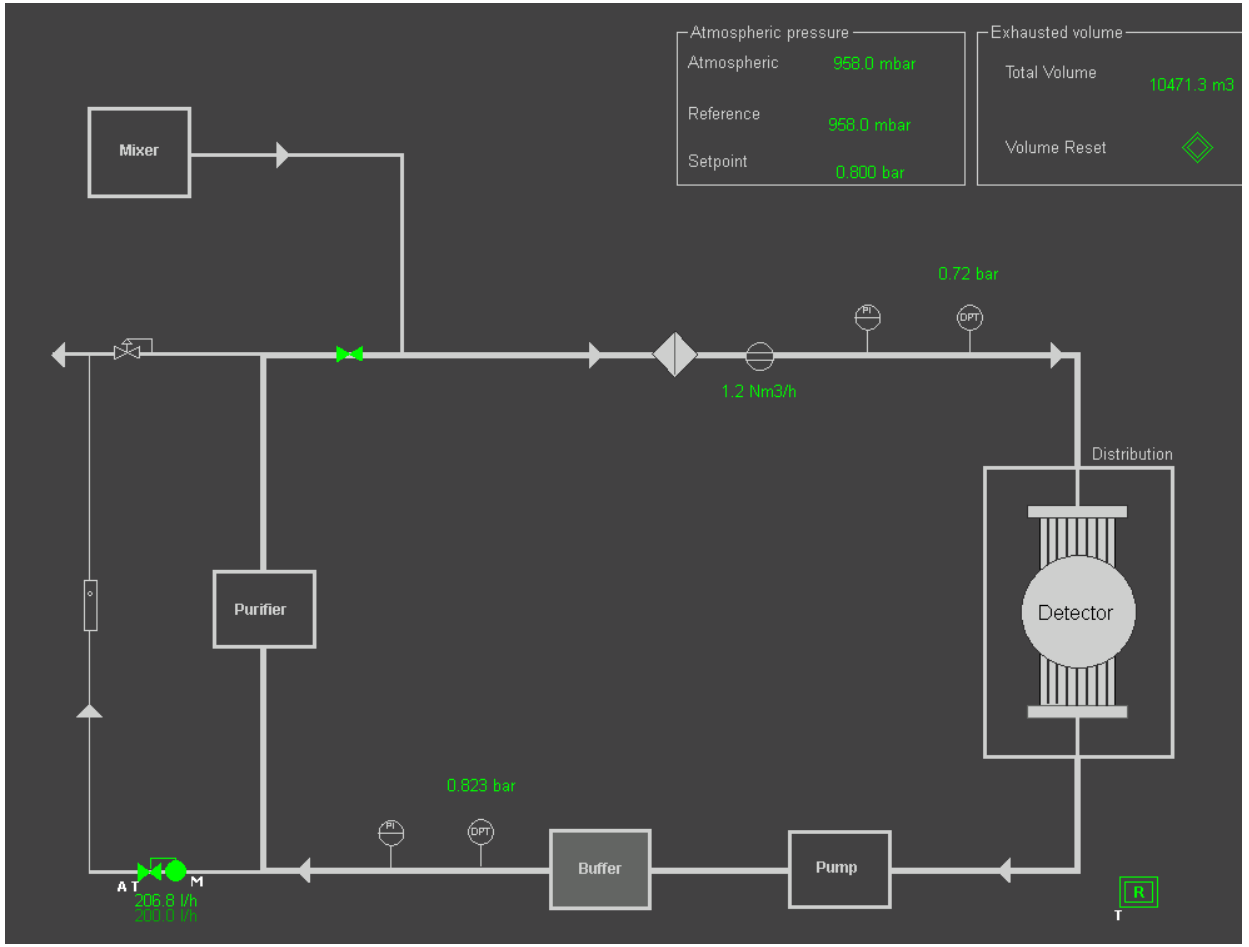


Membrane3 involved

Only membrane3 in function.



Exhaust with M3



GC ANALYSIS WITH M3

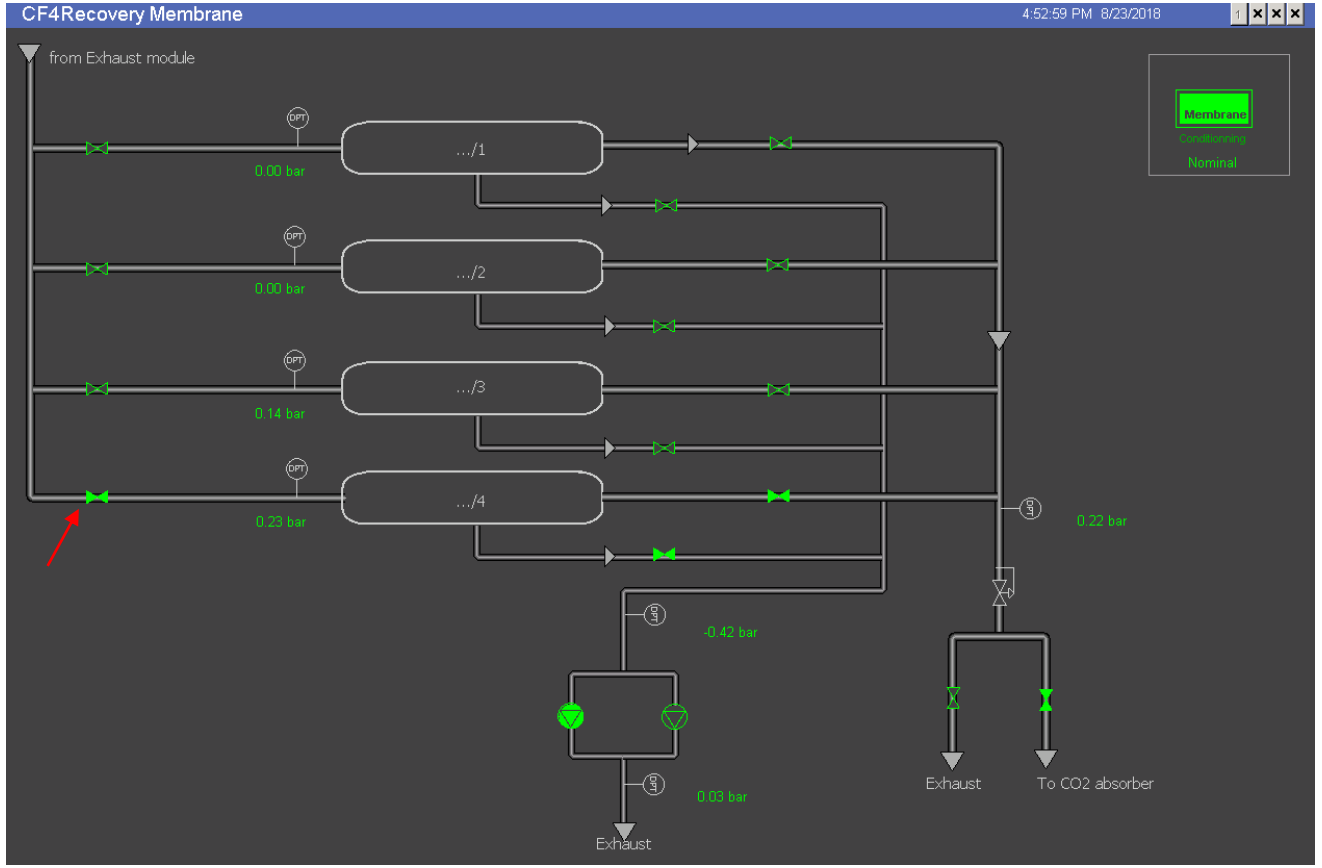
Membrane 3

GAS	QUANTITY
Ar	42.52%
CF4	0.61%
CO2	55.27%
O2	117 ppm
N2	7434 ppm

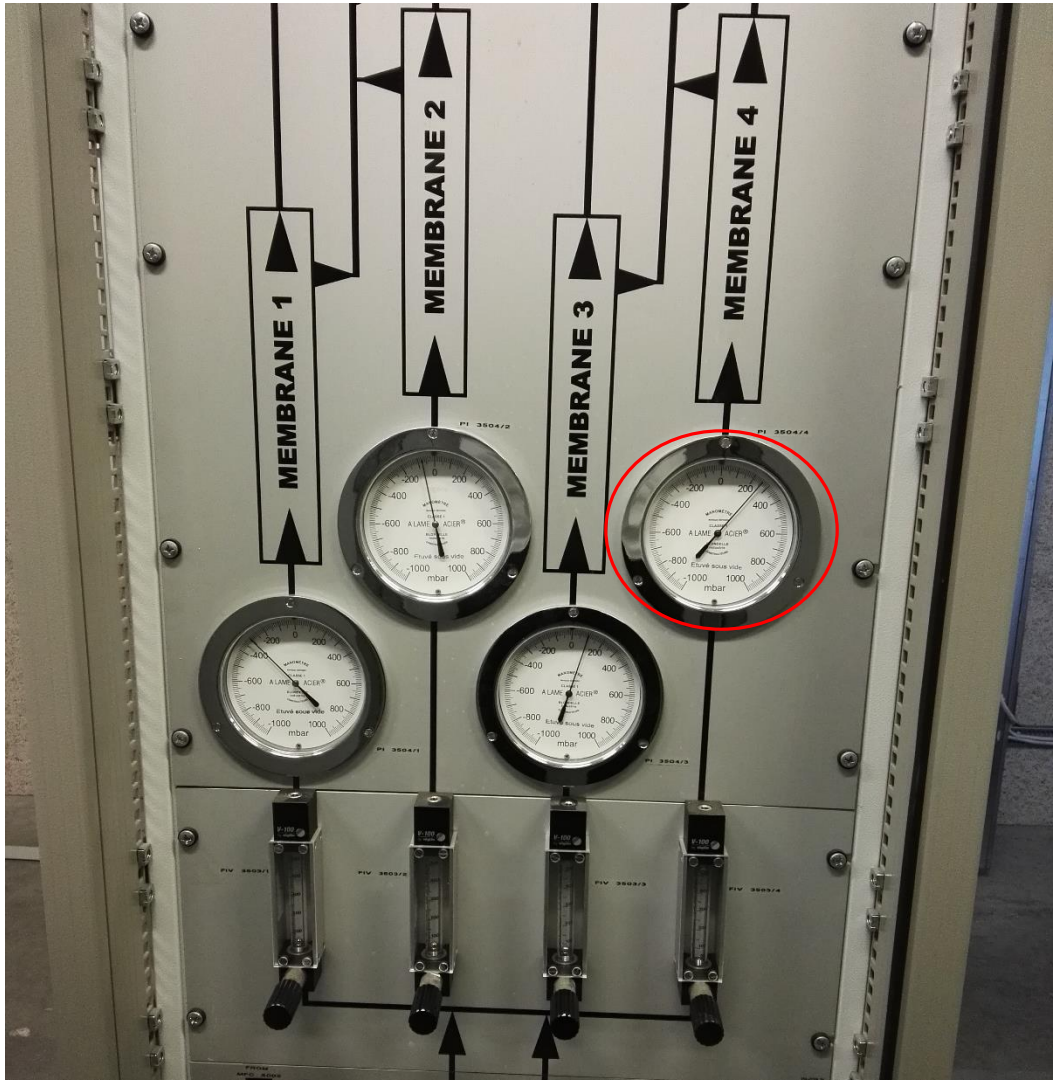
MEMBRANE 4

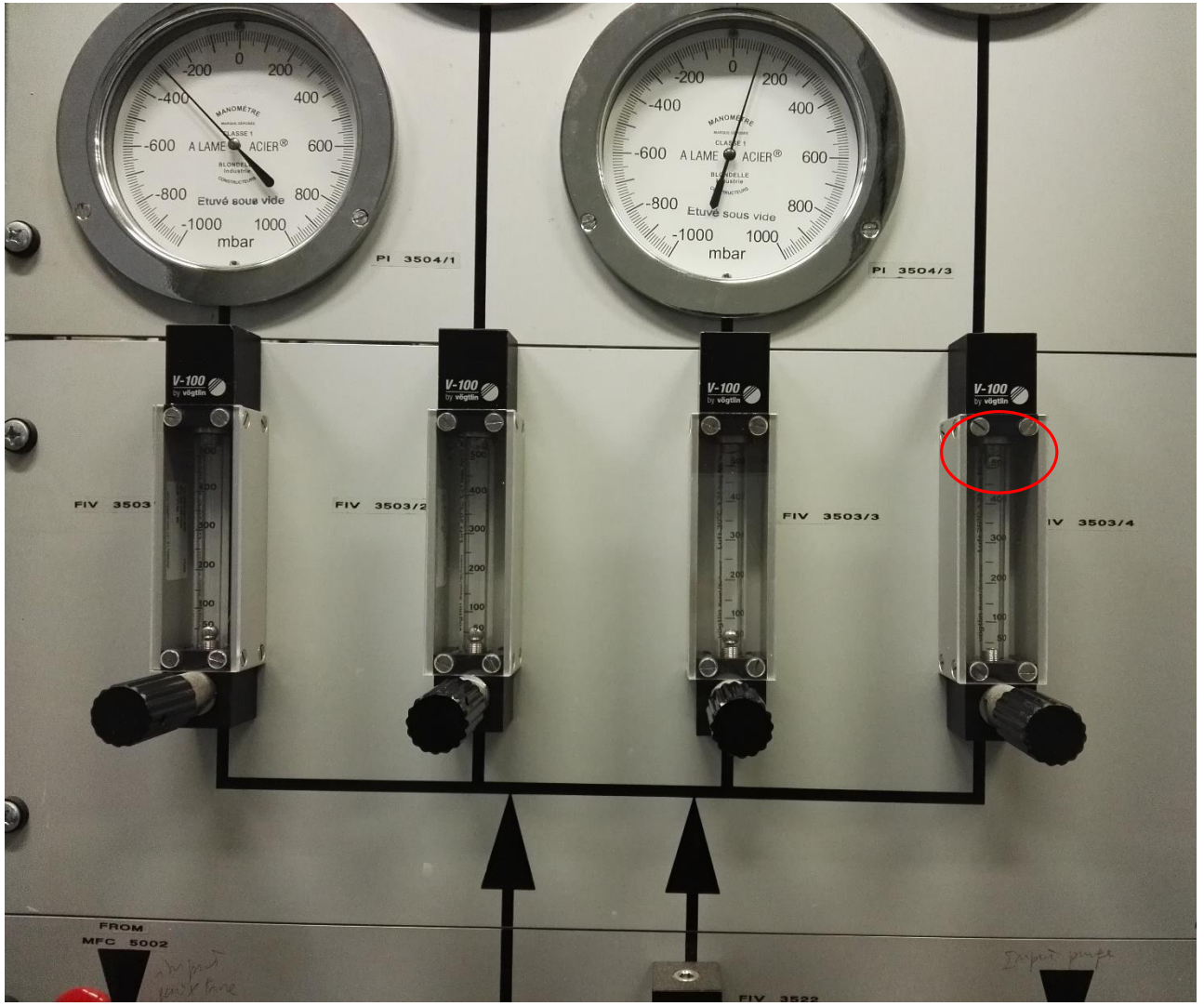
Fourth step ---> 23/08/2018

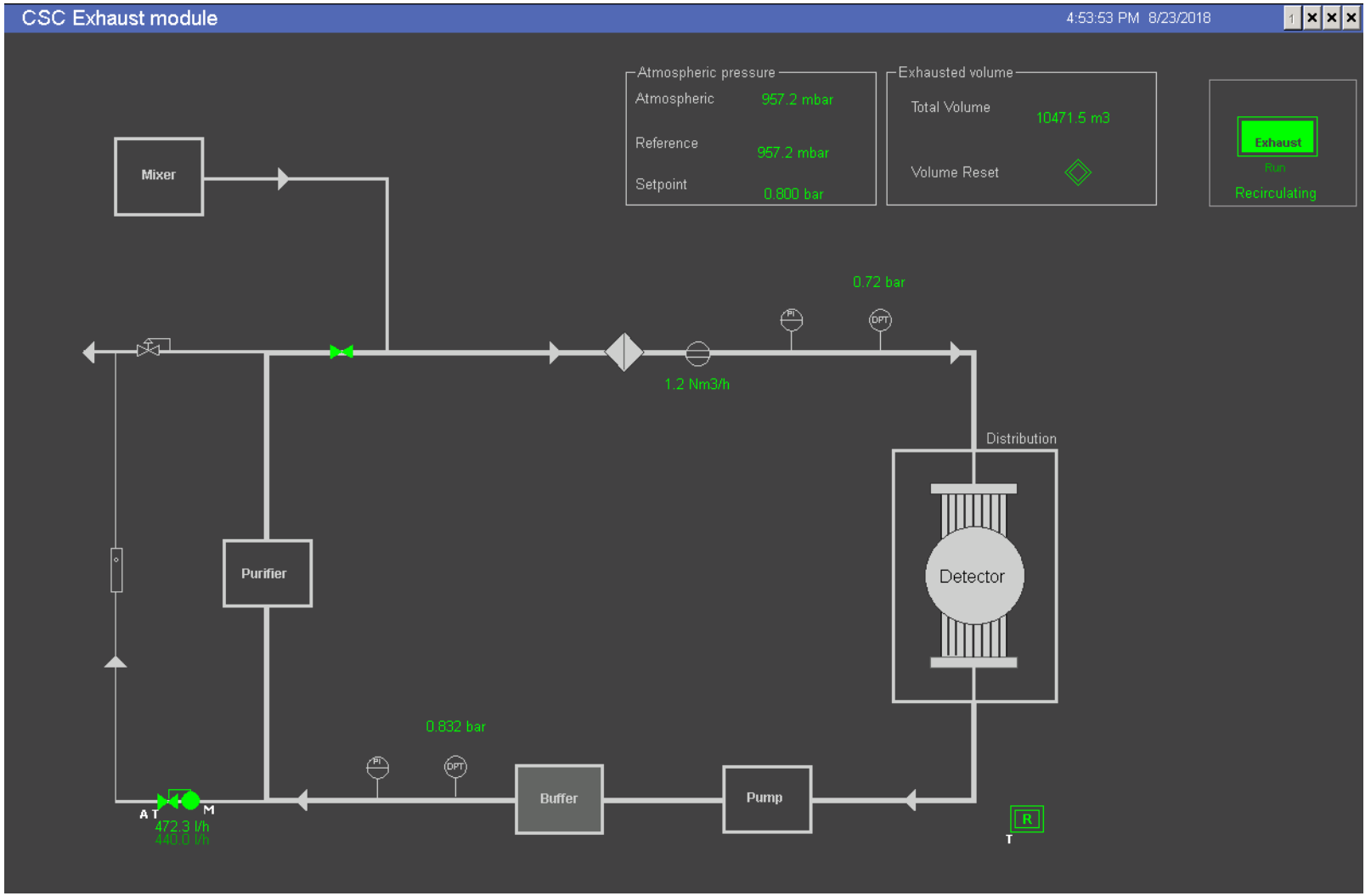
✓ Close membrane 1, 2 and 3. Leave only membrane four working. Then reduce flow from 600 l/h to 440 l/h because this membrane is bigger than the others. Flow has to be 2.2 times higher.



Membrane4 involved


MEMBRANE 1
MEMBRANE 2
MEMBRANE 3
MEMBRANE 4


PI 3504/1
PI 3504/3
Etuvé sous vide
mbar
V-100
FIV 3503
FIV 3503/2
FIV 3503/3
FROM
MFC 5002

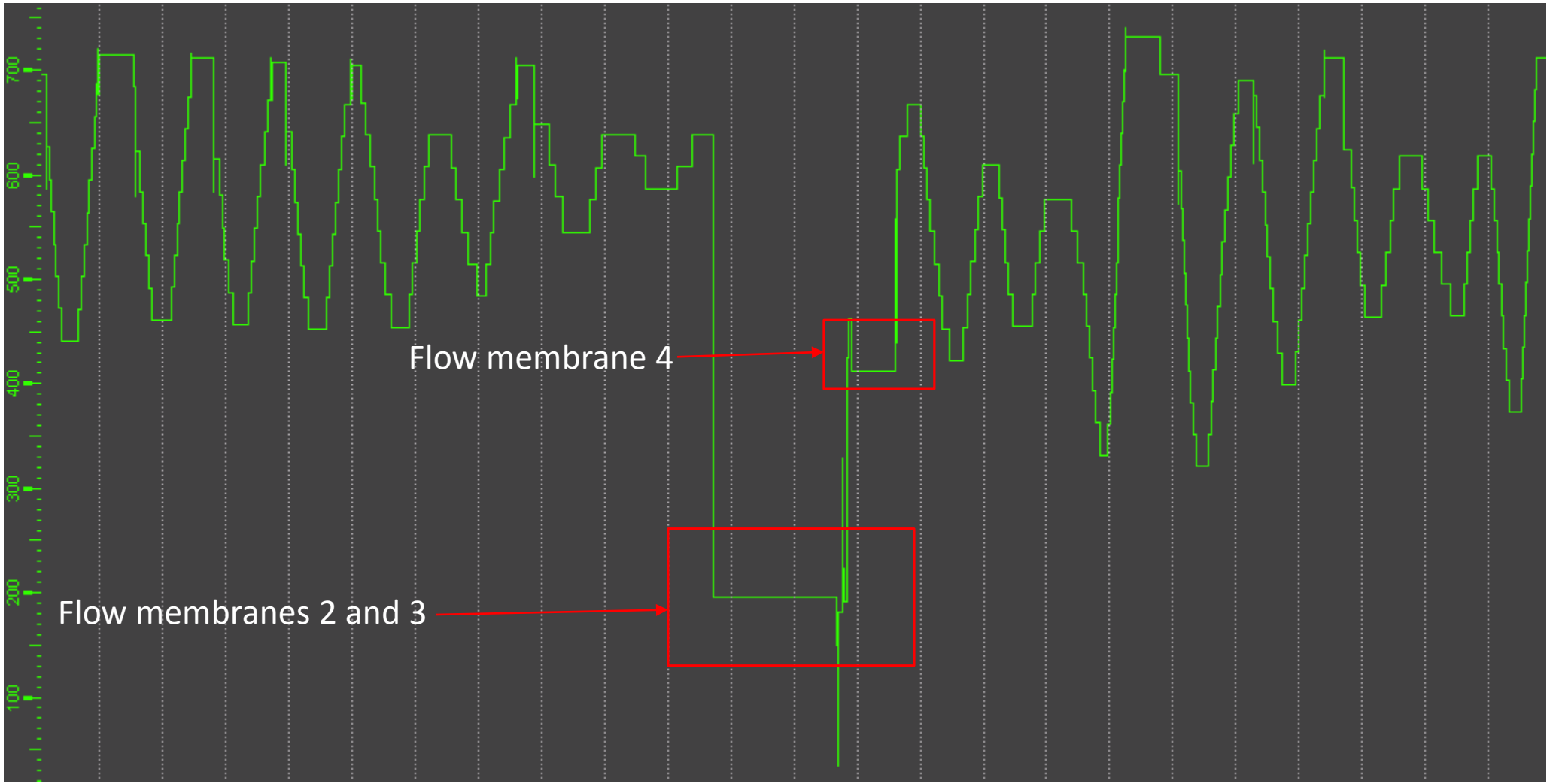


440 l/h of exhaust

GC ANALYSIS WITH M4

Membrane 4

GAS	QUANTITY
Ar	42.52%
CF4	0.59%
CO2	55.17%
O2	120 ppm
N2	7523 ppm


700
600
500
400
300
200
100
Flow membrane 4
Flow membranes 2 and 3

MEMBRANE 1,2,3

Fifth step ---> 24/08/2018

- ✓ Redo other analysis with three membranes involved but with the flow evenly divided into M1,M2 and M3 (200, 200, 200). This because the first analysis were done with different flow between the three membranes (150, 300, 300). With an equal flow in M1, M2 and M3 we expected an higher concentration of CF4 because we increase flow in M1 which is the less efficient membrane but CF4 percentage decrease. This probably because we didn't check correctly the flow during first analysis.

GC ANALYSIS WITH M1, M2 AND M3

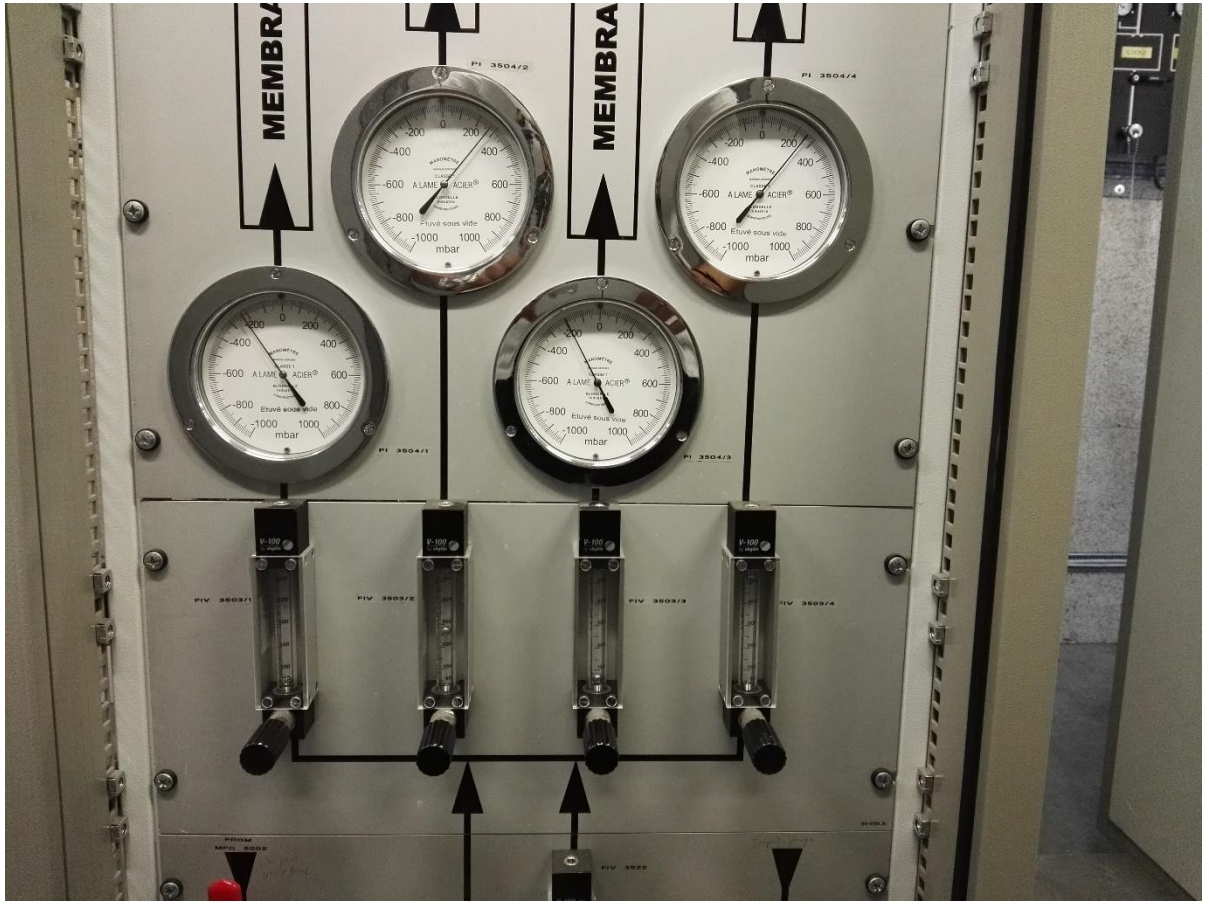
Membrane 1, 2, 3

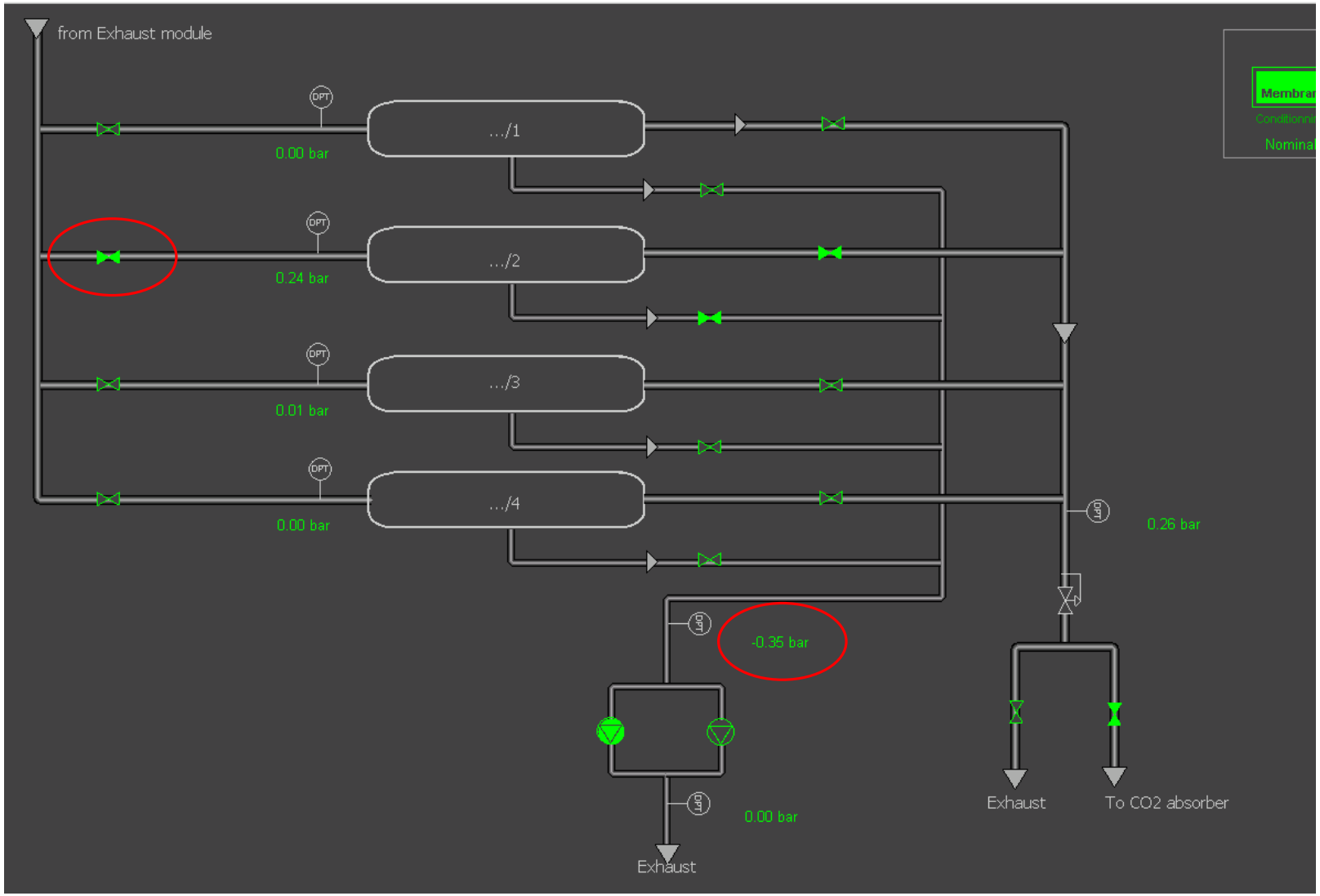
GAS	QUANTITY
Ar	43.46%
CF4	1.31%
CO2	56.47%
O2	130 ppm
N2	8087 ppm

M2 (-350mbar)

Sixth step ---> 24/08/2018

- ✓ After done analysis with different membrane settings we changed permeate pressure from -480mbar to -350mbar and do GC analysis to understand if percentage of CF4 decrease. Less CF4 percentage means more efficiency.





M2 on. Permeate pressure at -350mbar

GC ANALYSIS WITH M2 (-350mbar)

Membrane 2

GAS	QUANTITY
Ar	40.61%
CF4	0.56%
CO2	58.19%
O2	123 ppm
N2	7178 ppm

M2 (-200mbar)

Seventh step ---> 24/08/2018

- ✓ After done analysis with different membrane settings we changed permeate pressure from -350mbar to -200mbar and do GC analysis to understand if percentage of CF4 decrease. This solution seems the best as we can see from the next slide but N2 ppm decrease a lot so there are many consideration to do.

GC ANALYSIS WITH M2 (-200mbar)

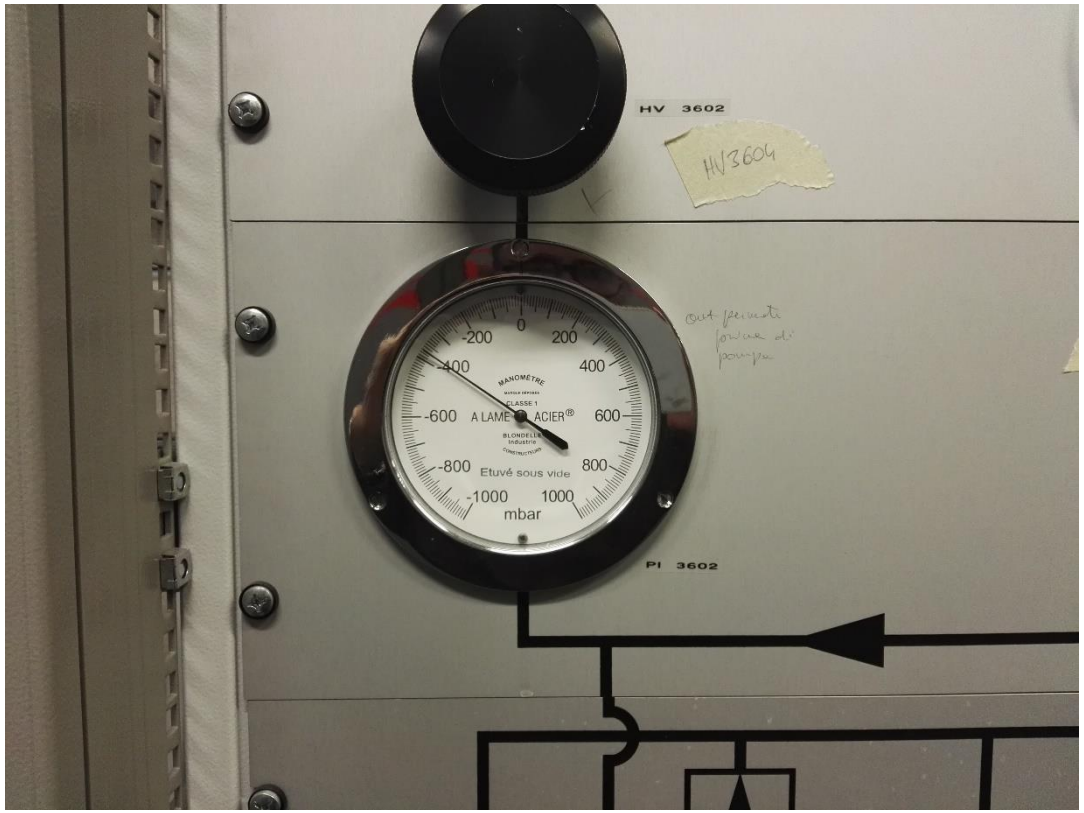
Membrane 2

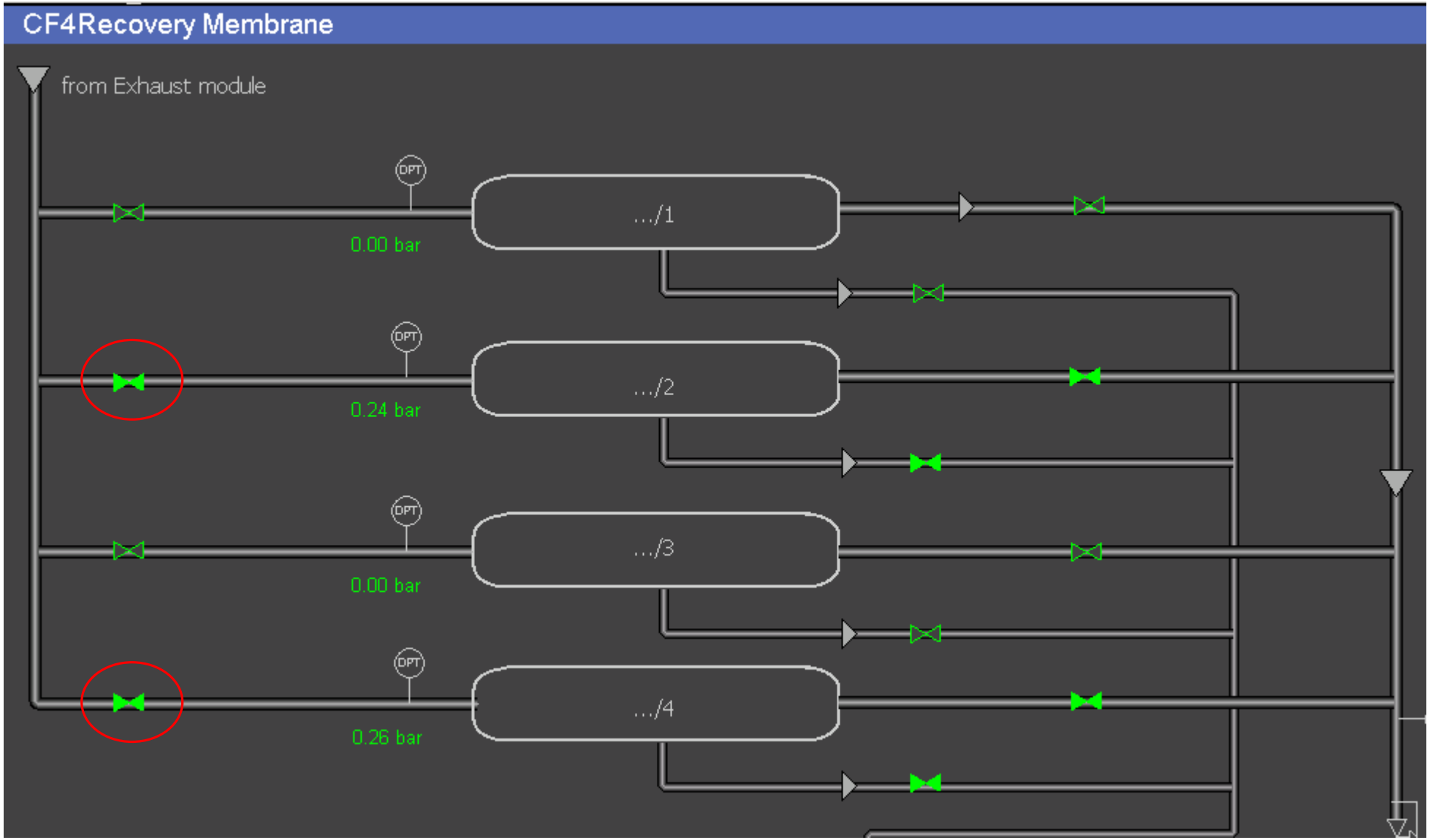
GAS	QUANTITY
Ar	38.91%
CF4	0.51%
CO2	60.37%
O2	120 ppm
N2	6685 ppm

MEMBRANE M2 AND M4

Eight step ---> 27/08/2018

- ✓ After done analysis with different pressure we return at -360mbar and do GC with M2 and M4 involved (not M1 because we calculated it is the membrane with the lower efficiency).




CF4Recovery Membrane
from Exhaust module
DPT
0.00 bar
.../1
DPT
0.24 bar
.../2
DPT
0.00 bar
.../3
DPT
0.26 bar
.../4

M2 and M4 on

GC ANALYSIS WITH M2 AND M4

Membrane 2 and 4

GAS	QUANTITY
Ar	43.00%
CF4	0.60%
CO2	56.72%
O2	145 ppm
N2	7401 ppm

FLOW WITH DIFFERENT MEMBRANE SET UP

M1	M2	M3	M4	Permeate Pressure	Flow
Rotameters Value				(mbar)	(l/min)
150	300	300	0	-480	9.60
250	250	250	0	-480	10.06
250	0	0	0	-480	3.15
0	250	0	0	-480	3.28
0	0	250	0	-480	3.15
0	0	0	>500	-480	6.96
0	250	0	0	-350	2.96
0	250	0	0	-200	2.75
0	250	0	>500	-360	9.30

CF4 ABSORBER

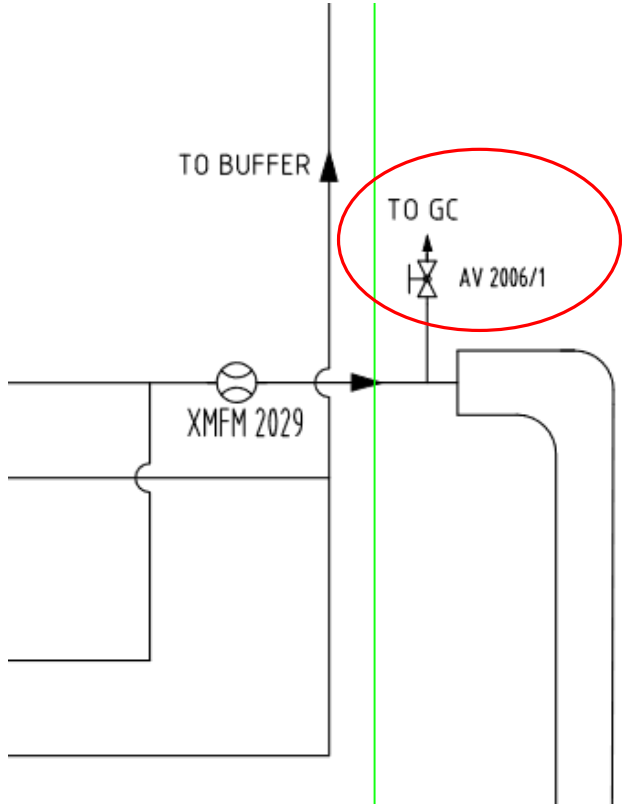
Ninth step ---> 27/08/2018

✓ Analysis done at CF4 absorber. Valve AV 2006-1. Membrane 2 on with a flow of 200 l/h and membrane 4 with 440 l/h.

GC ANALYSIS IN CF4 ABSORBER

CF4 Absorber

GAS	QUANTITY
Ar	24.42%
CF4	73.81%
CO2	0.02%
O2	28 ppm
N2	10098 ppm



GC ANALYSIS IN CF4 ABSORBER 2

CF4 Absorber

GAS	QUANTITY
Ar	23.22%
CF4	78.09%
CO2	0.04%
O2	61 ppm
N2	31688 ppm

Three times higher than the measure before. Too much N2.

GC ANALYSIS IN CF4 ABSORBER 3

Tenth step ---> 04/09/2018

✓ Analysis at CF4 absorber were remade to check why during the previous test N2 concentration was 30 000 ppm.

The results:

CF4 Absorber

GAS	QUANTITY
Ar	68.70%
CF4	30.08%
CO2	0.0064%
O2	64 ppm
N2	10267 ppm

BATTERY

Eleventh step ---> 04/09/2018

✓ Analysis at the battery. Battery B connected directly to the GC. Battery analysis can be done once battery per time because there is only one channel.

In the next slides is reported the procedure to follow for battery analysis.

1) 1- HV 4813 is located in the mixer rack

2) HV 4810 is located after HV 4813

3) HV 4811 for the GC

4) HV 4812 for the GC

5) Valve for the battery. When this valve is open is necessary to reduce pressure under 1 bar to not damage GC

GC ANALYSIS IN THE BATTERY

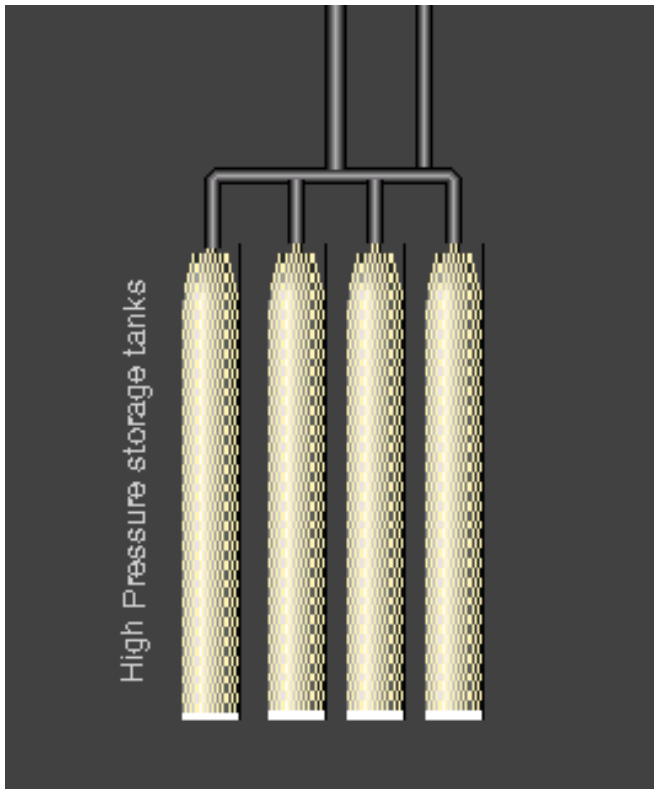
CF4 Battery1

GAS	QUANTITY
Ar	29.50%
CF4	76.10%
CO2	0.0026%
O2	97 ppm
N2	12629 ppm

CF4 INJECTION

Twelfth step ---> 12/09/2018

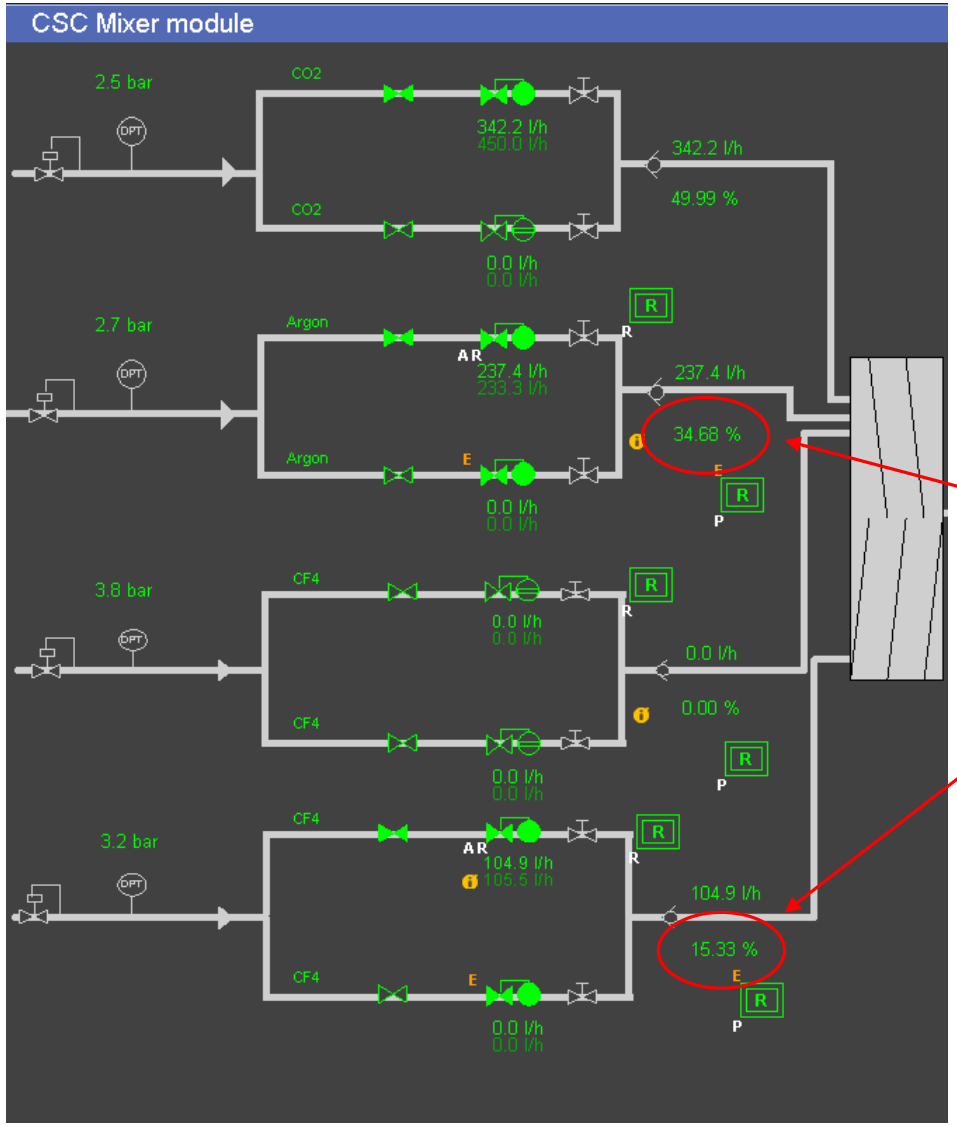
- ✓ Start injection from battery B. The pressure of the battery is 99.49 barg.
- ✓ Expected recuperation time ~2 months.



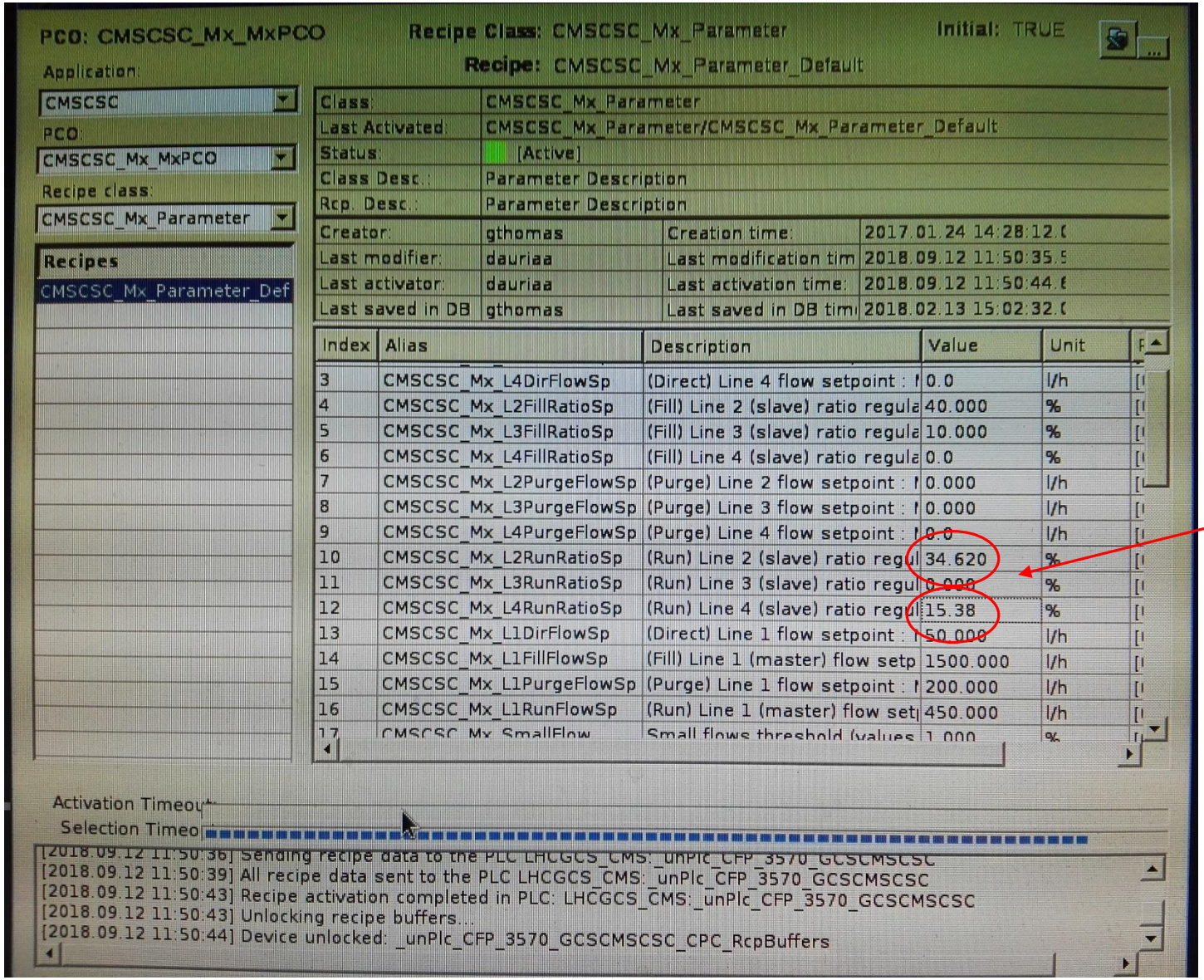
INJECTION PROCEDURE

First: open valve HV4813

Second: Increase pressure in the mixer (CF4 part) up to 3 bar



Percentage of Argon and CF4 to start injection



Percentage of Argon and CF4 to start injection, view from control panel

PROBLEM AT CF4 ABSORBER

On 16th September there was a problem with YV2103 at CF4 abs because the valve was set in manual due to automatic. So it did not open until we putted it in automatic the day after.

Efficiency for 2 days has been conditioned by this error.