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ENGINEERING OF THE WASTE COMPARTMENT

ESA contract 15689/01/NL/ND

TECHNICAL NOTE 71.9.2

Hardware set-up and documentation

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7. SAFETY

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1. Overview

1.1 Introduction

The liquefying compartment of the MELiSSA loop is responsible for the biodegradation of human faecal material, toilet paper and inedible parts of plants generated by the crew. The volatile fatty acids and ammonia produced during the anaerobic fermentation process are fed to the second photoheterotrophic compartment inoculated with the bacterium *Rhodospirillum rubrum*. The produced CO₂ is supplied to the photoautotrophic compartment inoculated with the algal strain *Arthrospira platensis* and to the higher plant compartment.

At the pilot plant of the University of Barcelona, the three compartments of the MELiSSA loop (photoheterotrophic compartment CII, nitrifying compartment CIII and photoautotrophic compartment CIVa) are already connected at lab scale and will be validated at pilot scale. In order to validate the whole MELiSSA loop, it is necessary to construct the first compartment at pilot scale (fermentation reactor) for the primary degradation of the waste produced by the crew, in order to demonstrate the Melissa technology on Earth.

Before this phase, a prototype reactor was designed and build, allowing to test and optimise the selected hardware. This technical note includes a description of the prototype reactor operation and its associated instrumentation.

The prototype compartment performs the anaerobic digestion of a waste representing 25% of the waste produced by one man per day.

The prototype reactor is designed to be operated continuously, with a semi-continuous feeding of 2.5L/d of Melissa mix and a semi-continuous production of 2.5L/d of permeate generated by an associated ultra-filtration unit.

1.2 Concept of the prototype compartment

The prototype compartment has been built based on the design of the pilot compartment as it was defined in technical notes 71.1 to 71.5. It does not correspond to an exact copy of the pilot reactor: the aim of this prototype is to validate the design conceived for the pilot, it has thus been built with adaptable materials, in order to allow adaptations for optimisation of the design. The following schemes describe the concept of the prototype sub-units (reactor, filtration unit, gas loop).

1.2.1 Prototype reactor

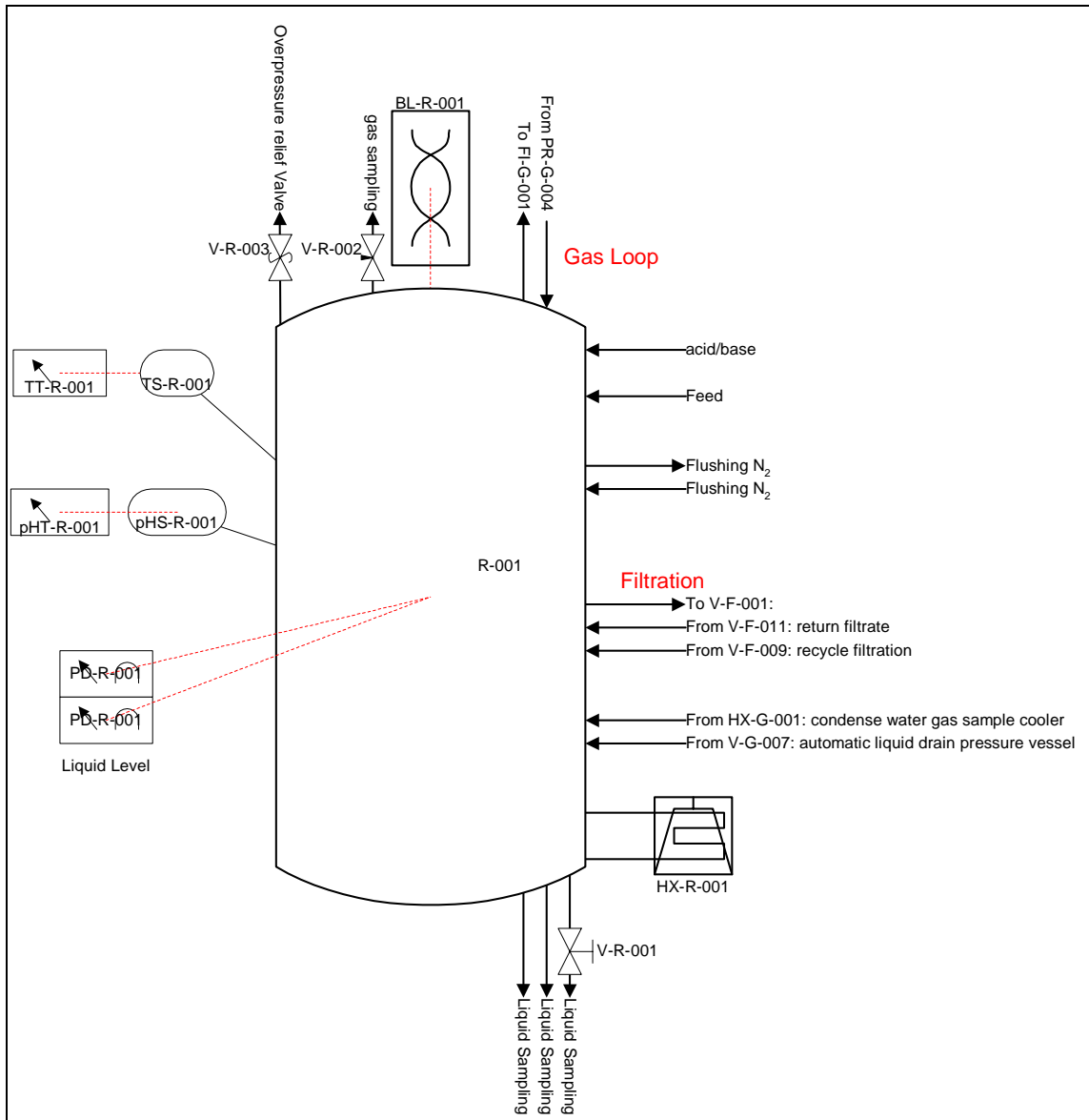


Figure 1. Prototype reactor

1.2.2 Prototype Gas loop

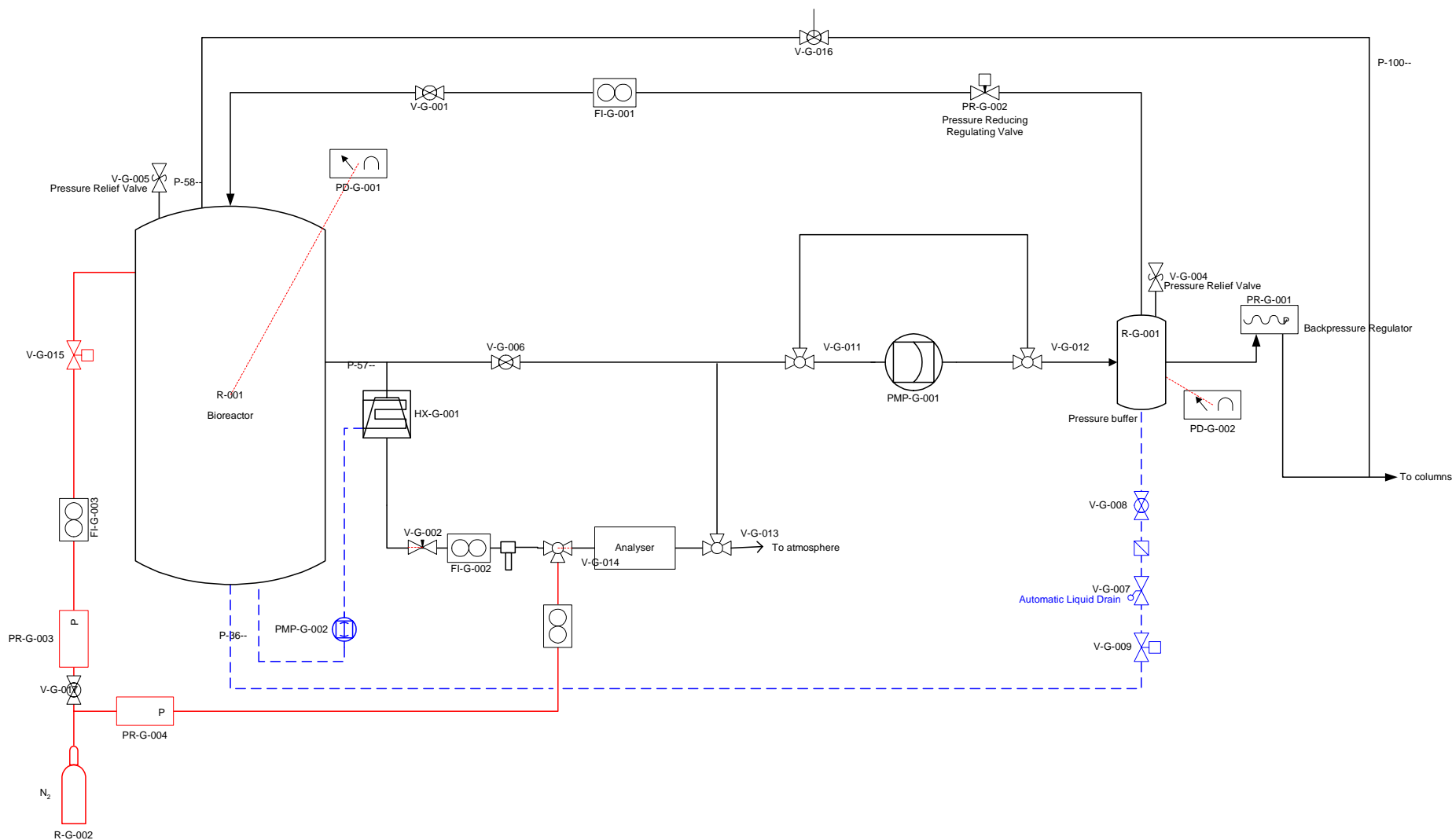


Figure 2. Prototype Gas Loop

1.2.3 Prototype Filtration Unit

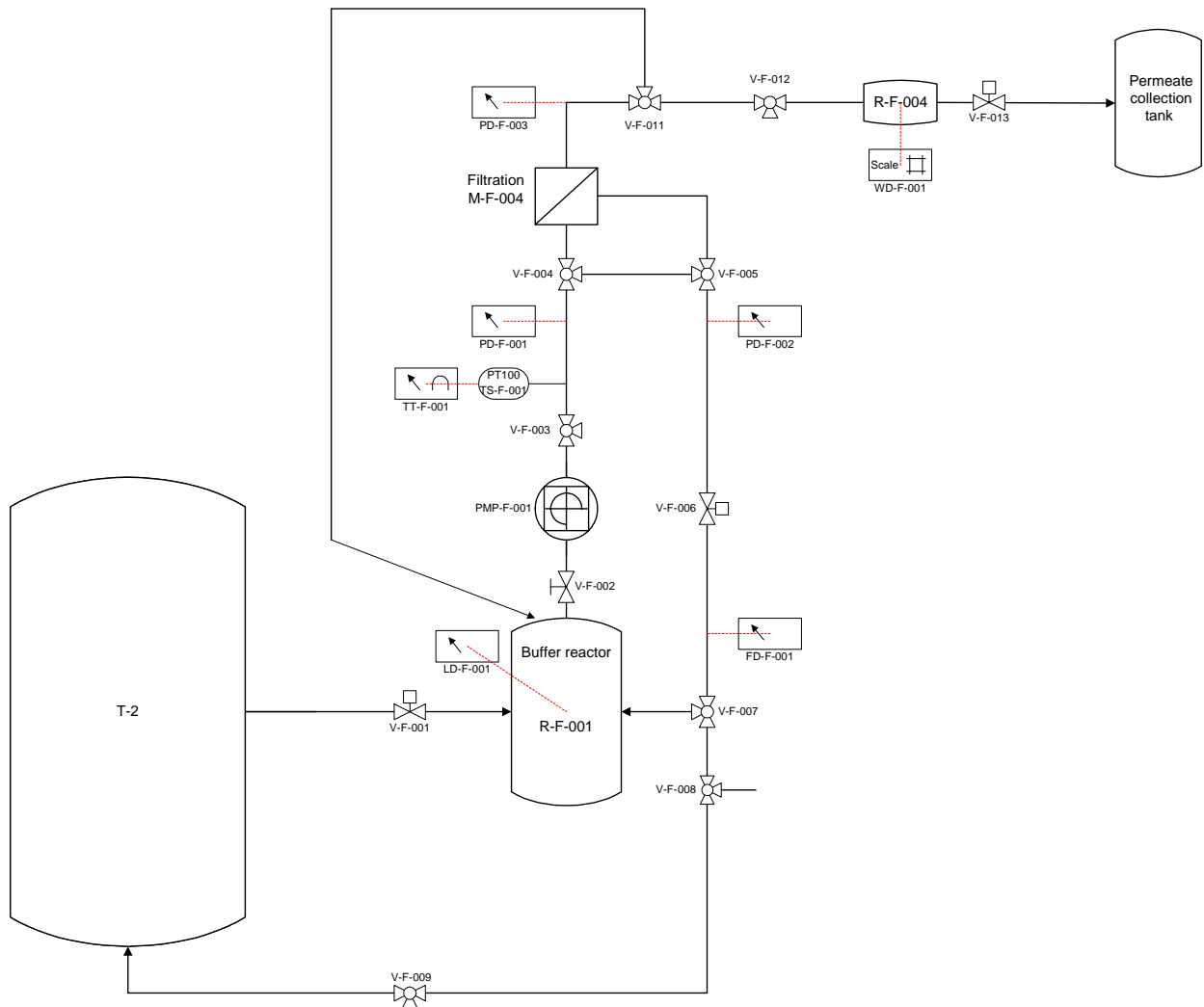


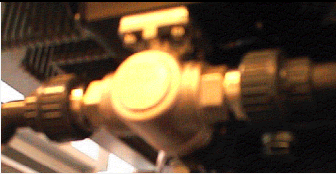


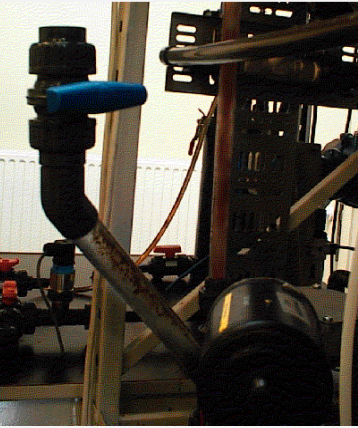
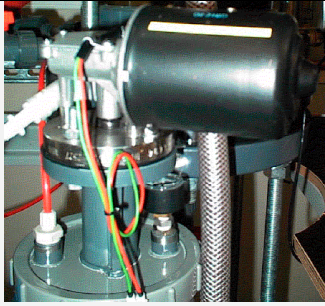


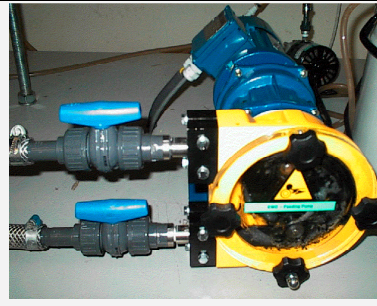
Figure 3. Prototype Filtration Unit

1.3 Principal instruments

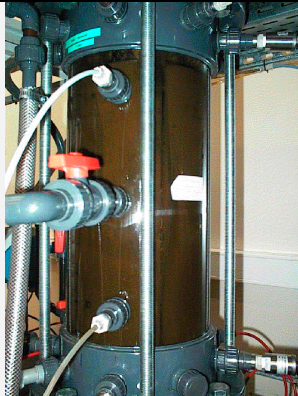
| General hardware | |
|---|--|
|  |  |
| <p>Electrical cupboard</p> | <p>Manual valves</p> |
|  | |
| <p>Electronic valve</p> | |
| Bioreactor | |
|  | |
| <p>Computer</p> | |
|  |  |
| <p>Influent buffer tank</p> | <p>Influent tap</p> |



Influent tank mixer



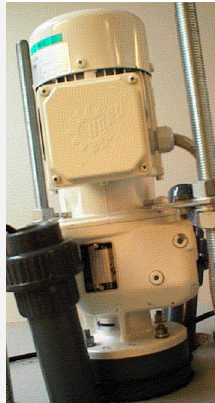
Influent feeding pump



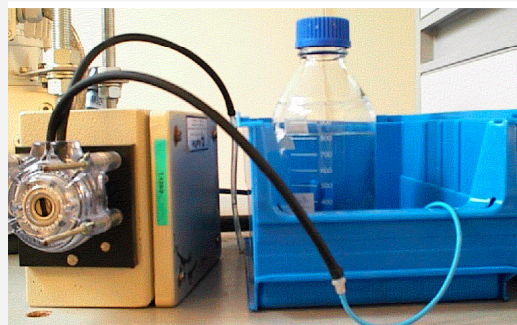
Bioreactor




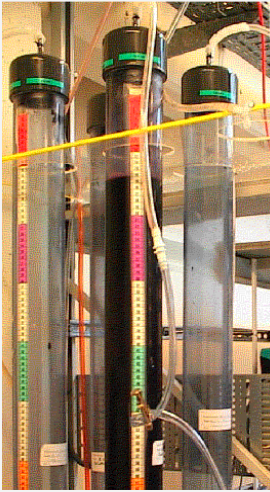
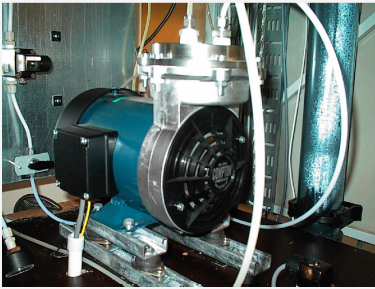
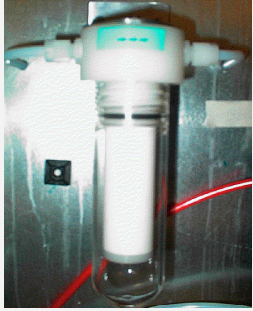


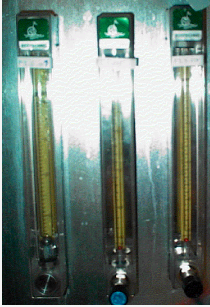

Warm water bath



Bioreactor mixer



pH controller

| Gas Loop | |
|---|--|
|  |  |
| <p>Pressurized buffer vessel</p> | <p>Gas column</p> |
|  |  |
| <p>Compressor</p> | <p>Gas filter</p> |
|  |  |
| <p>Gas analyser</p> | <p>Gas cooler</p> |
|  |  |
| <p>Flow indicators</p> | <p>Backpressure regulator</p> |




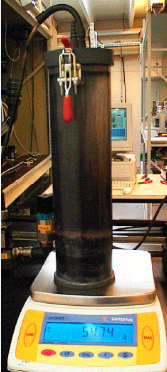
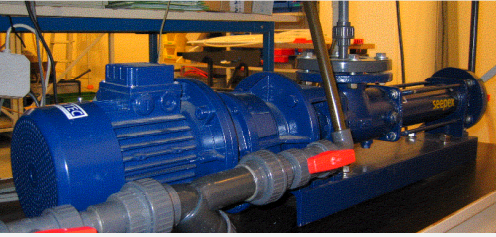

| | |
|---|--|
| Filtration Unit | |
|  |  |
| Computer | Membrane module |
|  |  |
| Buffer tank | Permeate tank |
|  |  |
| Progressive cavity pump | Flowmeter |

Figure 4 Prototype instrumentation pictures

1.4 Description of user interfaces

1.4.1 Interface Bioreactor

Figure 1 shows the corresponding user interface. The different parameters that are measured on-line can be read on this interface. Also actions can be performed remotely from the interface: start-up of Gas Loop compressor, feeding system and blender.

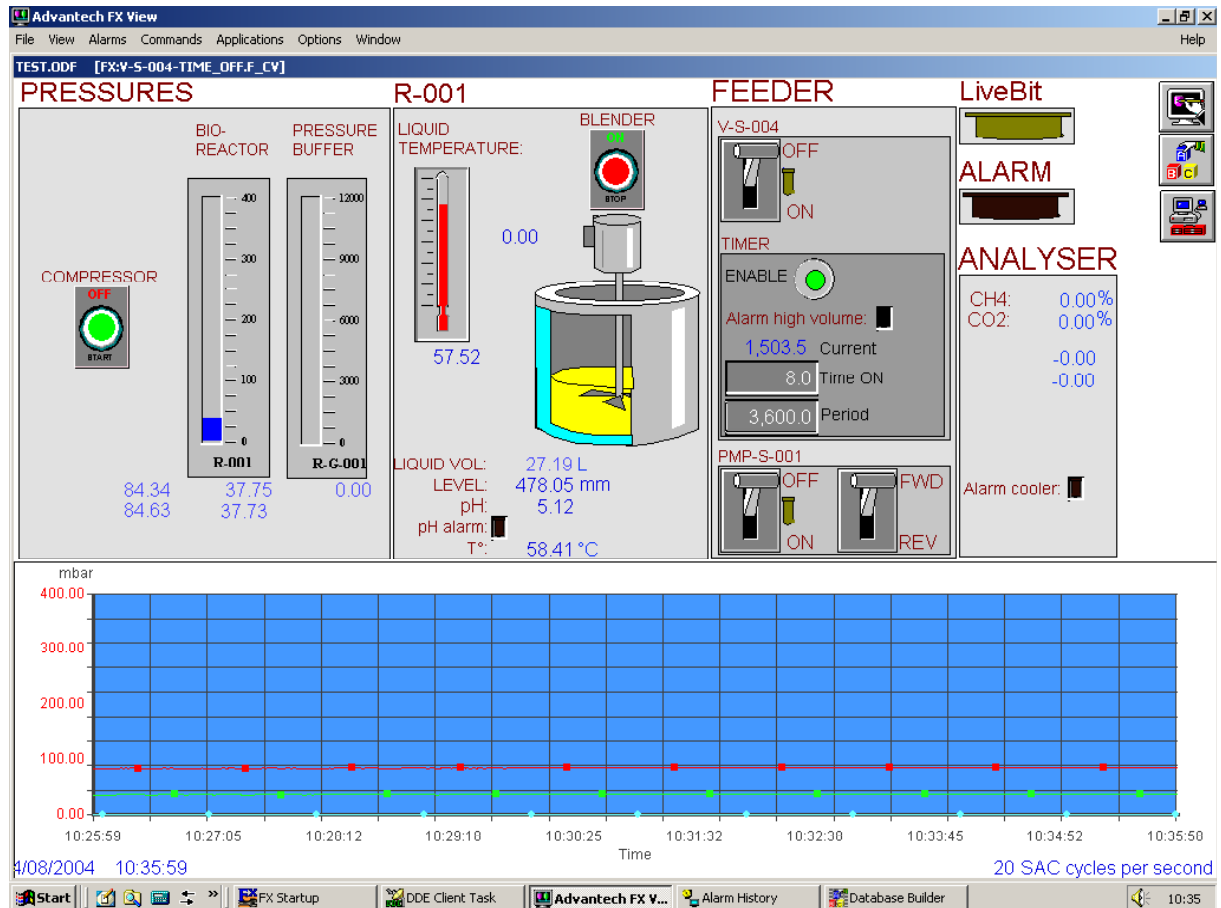


Figure 5. Bioreactor PC interface

1.4.2 Interface Filtration Unit

1.4.2.1 Program window

This is the window that appears when the program is opened. In this window, it is possible to select the type of module, and to load a program to run the unit.

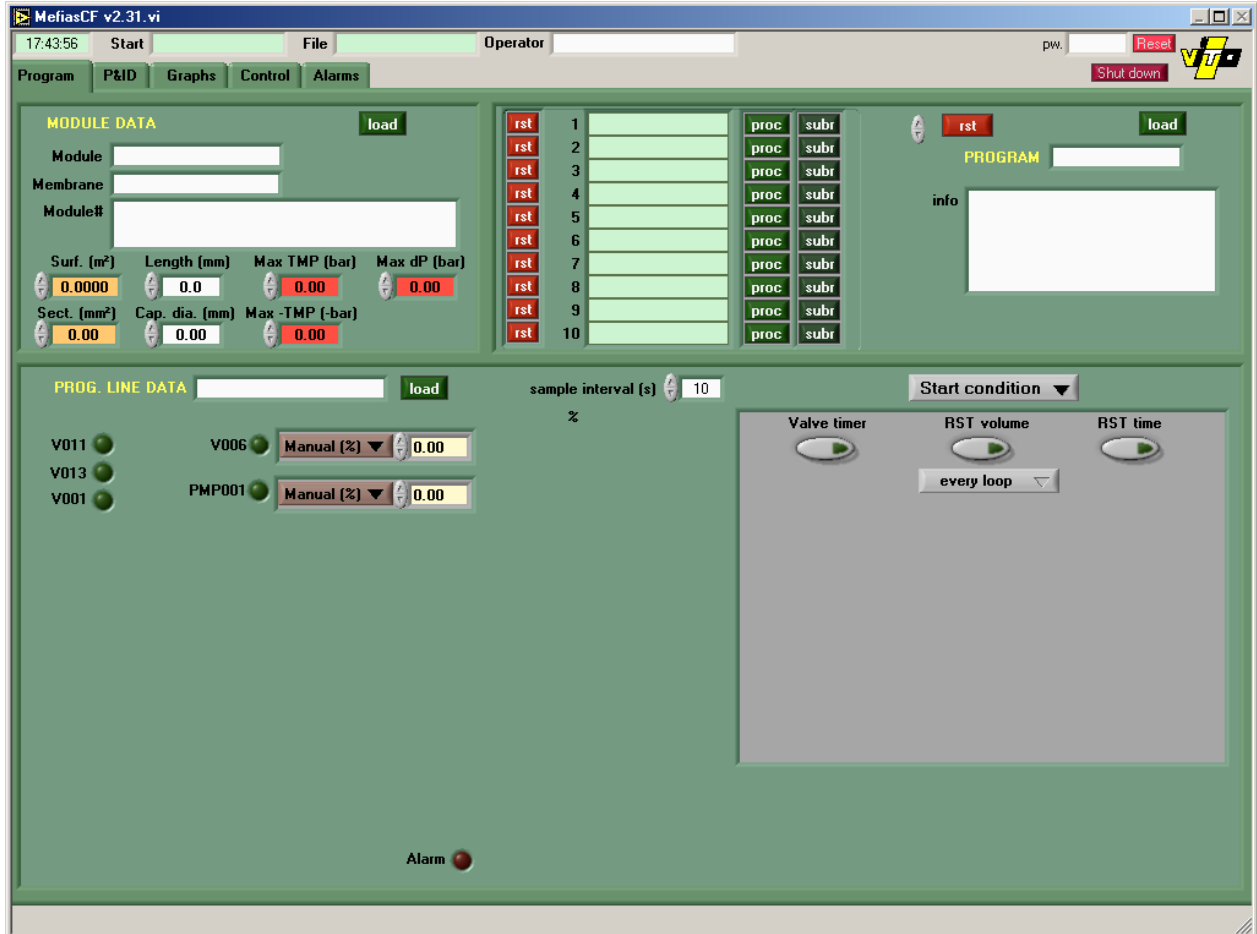


Figure 6. FU Program window

1.4.2.2 P&ID window

This window allows to visualise on-line the data measured on a conceptual scheme of the unit.

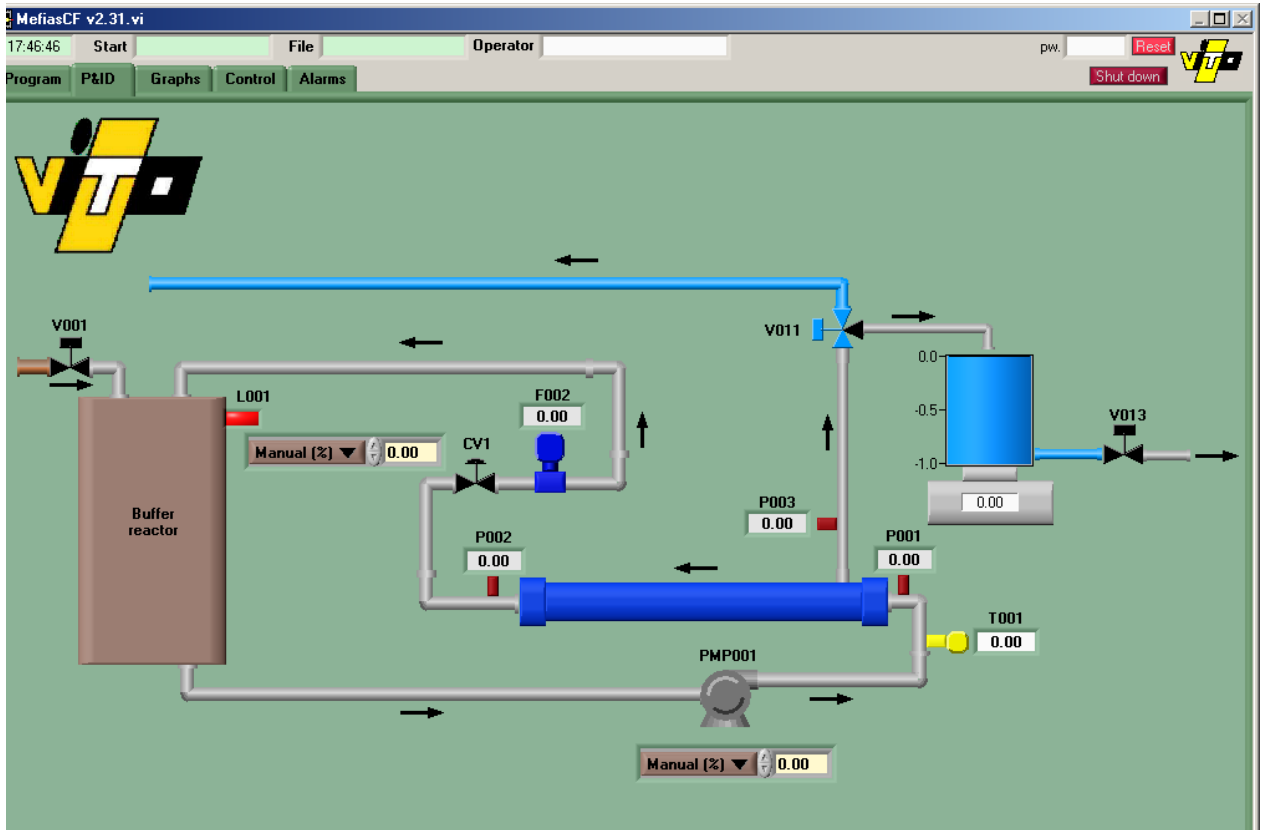


Figure 7. FU P&ID window

1.4.2.3 Graphs window

This window allows to visualise the evolution in the time of all the parameters measured on-line on the unit.

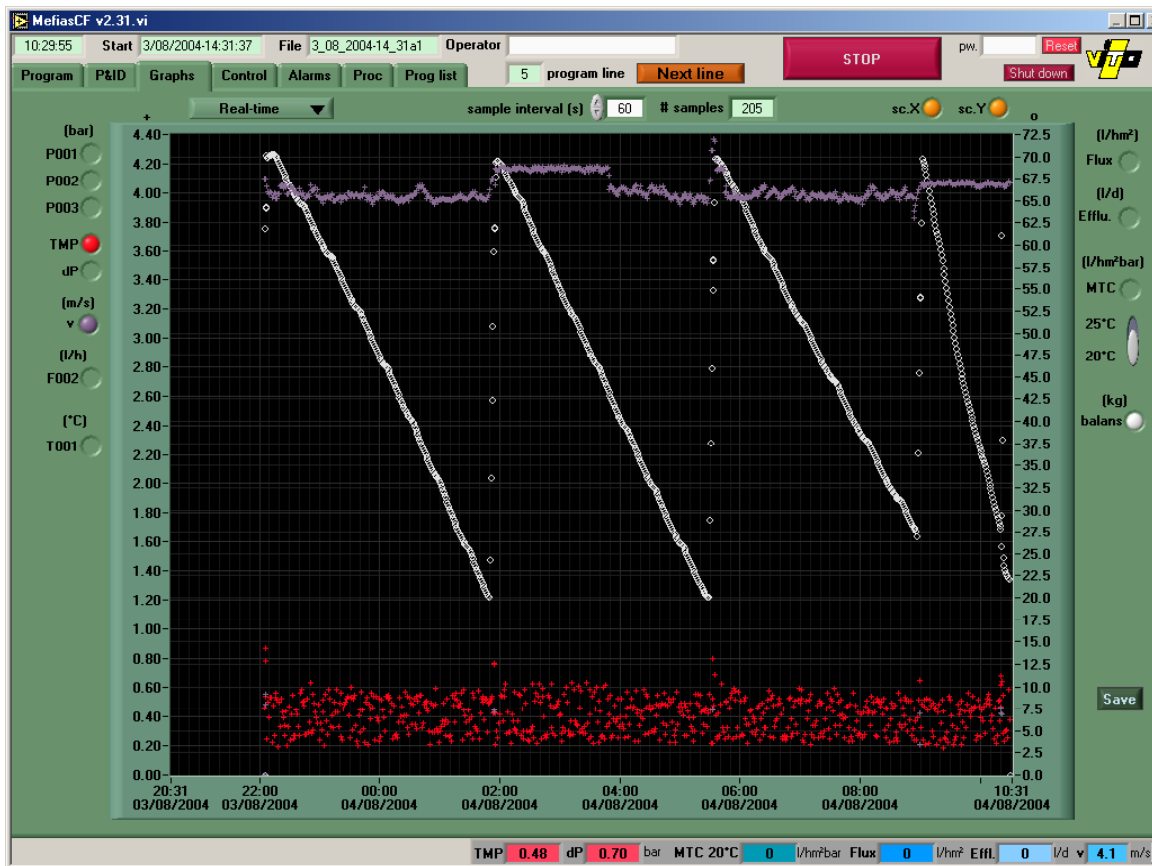


Figure 8. FU Graphs window

1.4.2.4 Proc window

This window allows to modify definitely the settings of a running program (overflow period, TMP (Trans Membrane Pressure), mass of permeate to produce...)

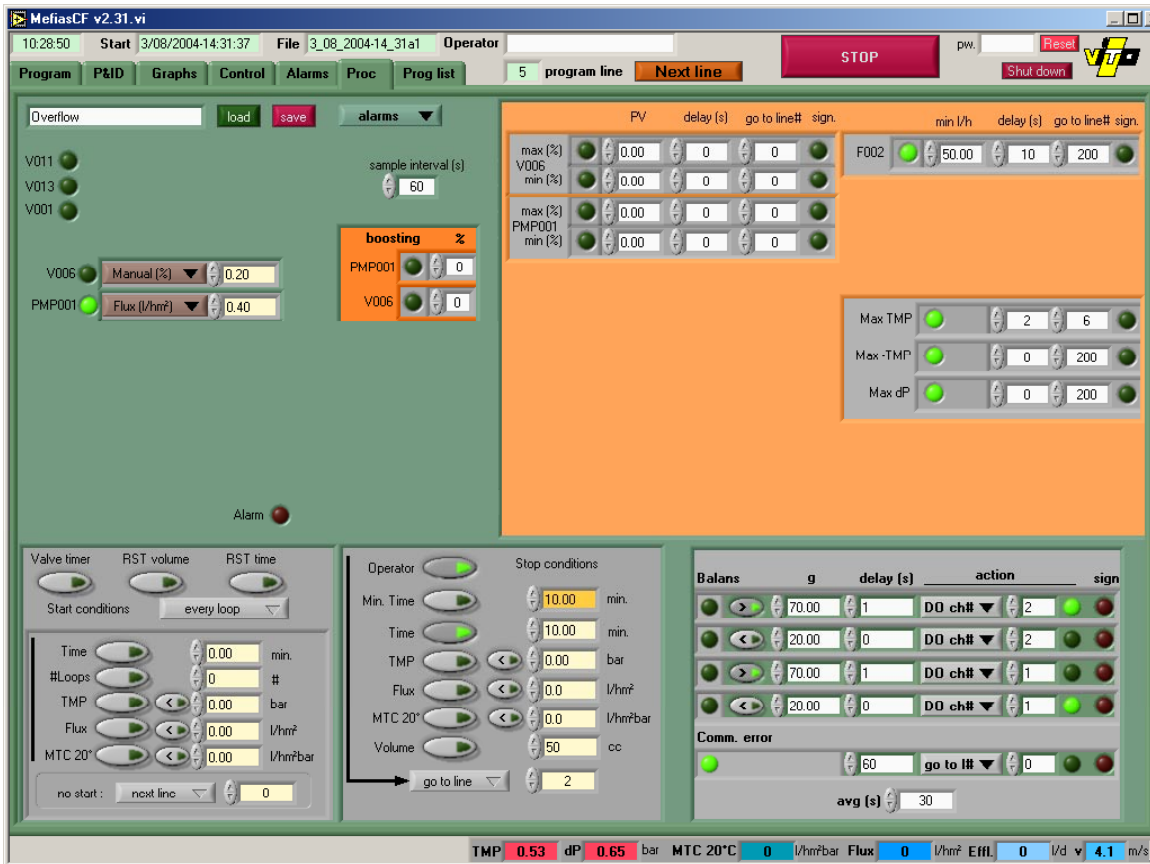


Figure 9. FU Proc window

1.4.2.5 Control window

This window allows the user to modify the settings of alarms of one specific program.

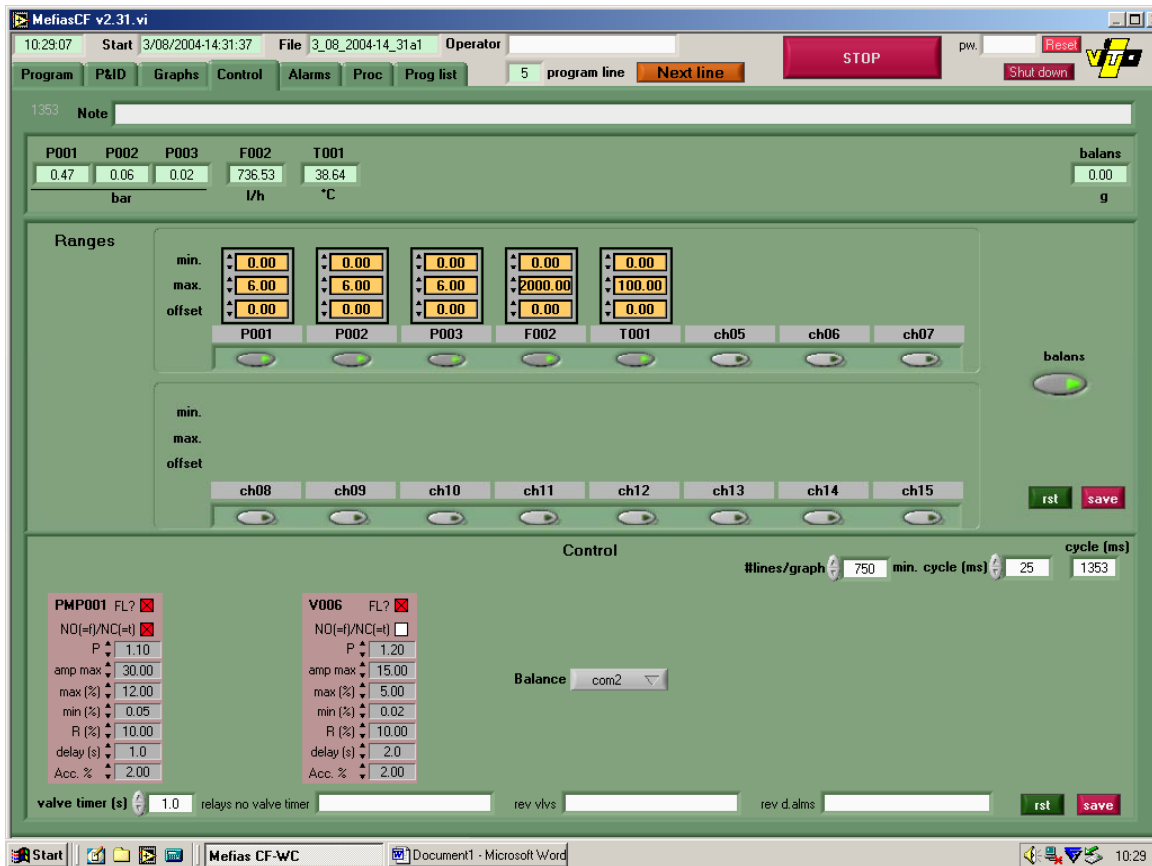


Figure 10. FU Control window

1.4.2.6 Alarm window

This window shows the alarm messages delivered by the hardware.

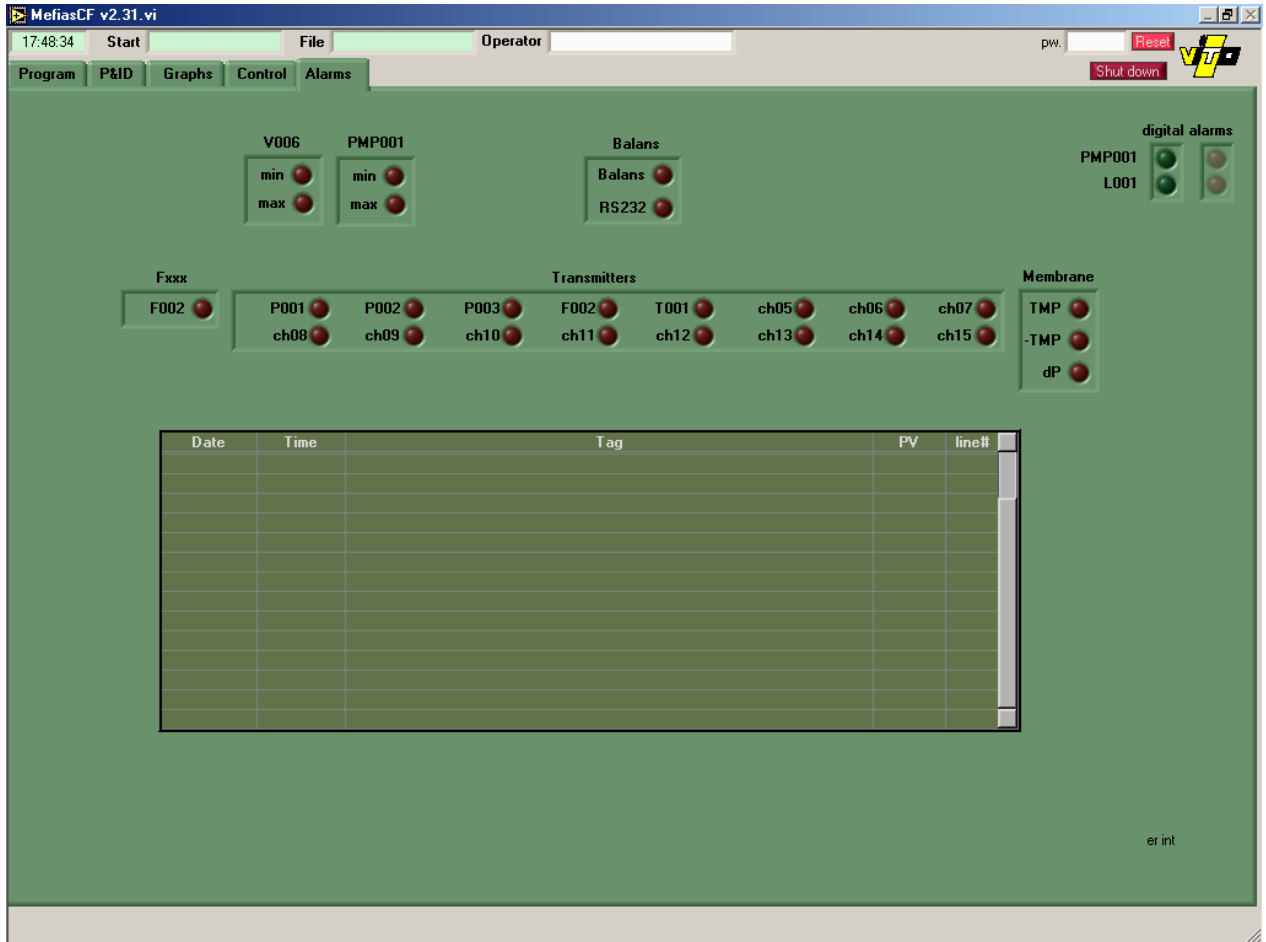


Figure 11. FU Alarm window

2. Specific operation of the instruments

2.1 Gas cooler

The gas cooler cools down the gas and dries it by condensation. It can be activated by plugging it in. The cooler is effective at its optimal temperature of 5°C when the arrow is in the green zone on the screen.

2.2 Warm water bath

Fill the bath with demineralised water. Push the on/off button and set the temperature (55°C) by turning the button on top of the thermometer until the red line is on the right temperature.

2.3 Stirrers (bioreactor and influent tank)

Both stirrers must be switched on/off from the reactor electrical cupboard. Their speeds can be regulated from there.

2.4 Centrifugal pump for influent

It can be switched on/off from the screen located under the reactor electrical cupboard. The circulation sense and speed can also be adapted there. The pump cannot be controlled from the computer interface. The influent feeding can however be controlled from the interface by setting the timer of the valve allowing the feeding.

2.5 Gas analyser

It can be switched on/off from behind. The gas cooler must always be installed before the analyser and must work since at least a few hours before to switch the analyser on. The screen displays directly the composition in terms of CO₂ and CH₄.

3. Specific settings and calibrations

3.1 Centrifugal pump for influent

Before usage, calibrate the pump with the appropriate tubing. Therefore a chronometer and a graduated cylinder are needed. Make sure the tubing is completely filled with liquid. Measure the flow for 5 different speeds and set up the calibration curve. The desired speed can be selected from the calibration curve, knowing that the influent shall be fed once per hour and that a higher speed is preferred for the pump since it allows also a homogenisation of the influent tank. Once the speed is selected, the corresponding frequency of feeding/ non feeding must be set on the screen of the reactor computer. Click on the frequency box under "Influent tank" and enter the appropriate periods (Time ON and period). Then click on "save", and "replace".

3.2 Overflow setting on Filtration Unit

The filtration unit operates alternatively in 2 phases:

- Filtration: the filtrate produced is sent to a permeate tank where it is weighted before to be released out of the system; one filtration phase corresponds to the production of 50mL
- Overflow: the filtrate produced is sent back to the buffer tank of the filtration unit.

The overflow period is set to allow the wanted production of filtrate per day (2.5L/d). It can vary with the age and degree of foulness of the membrane and should be regularly and manually adapted. To set it, open the "overflow" procedure on the "Proc" window of the FU computer. Then enter the right period in the box called "time". Then click on "save", and "replace".

3.3 pH control

pH is measured by an electrode inside the reactor and a transmitter built into the front door of the electrical cabinet. This transmitter is equipped with functionality to also control pH by means of two electrical relay contacts REL1 and REL2 that can be used to add acid or base. Each of them can be linked to a different control function.

In this setup both REL1 and REL2 are put in series so that addition of acid or base will happen only if both contacts are closed. REL1 is configured for PID control. REL2 is configured as a limit contactor that enables the function of the PID control only beyond a certain deviation from the set point. An "alarm" contact is again put in series with REL1 and REL2 to disable acid or base addition when the controller does not achieve in keeping the deviation of the process variable from the setpoint to a reasonable value (for instance pH 0.2) for a longer time (for instance 1 minute), because of an abnormality like a leak in tubing or a wrong measurement caused by a faulty electrode.



Figure 12. pH transmitter

Altering setpoint and control direction can be done in the function group RELAIS that can be accessed by pressing "E", entering code 22 and then pressing "+" or "-" until "RELAIS" is found. Then just press "E" again. Both configurations for REL1 configured as PID controller and REL2 configured as Limit contactor need to be revised if set point and/or control direction need to be changed. Please refer to the manual pages 43 to 46.

- PID controller: First choose REL1 in field R1 of function group RELAIS. This leads to field R2 which should be set to "PID". Most sub fields of R2 should remain unaltered.
 - To change set point: enter new set point (SP) in field R232.
 - To change control direction: switch R236. dir (direct) is to be chosen in case of acid addition; inv (inverted) for base addition.
- Limit contactor: see Figure 13 and the manual paragraph 5.5.1 page 38 for information on the operation of the limit contact function. First set Relais REL to limit contact functionality by choosing "REL2" in field R1 of function group RELAIS. This leads to field R2 which should be set to "LC PV" (Limit Contactor for Process Value).

The setting of switch-on and switch-off points will directly lead to the appropriate setting regarding the control direction (i.e. minimum or maximum). If the switch-on point is lower than the switch-off point then the limit contactor will work in minimum function (see Figure 13, B). In this mode the relais contact is closed when the process value (pH) is too low. Because this relais contact is wired in series with the contact of REL1 – which is controlled by the PID controller - it will allow the PID controller's output when the pH is lower than the set point. Therefore this control direction is to be used in case of base addition.

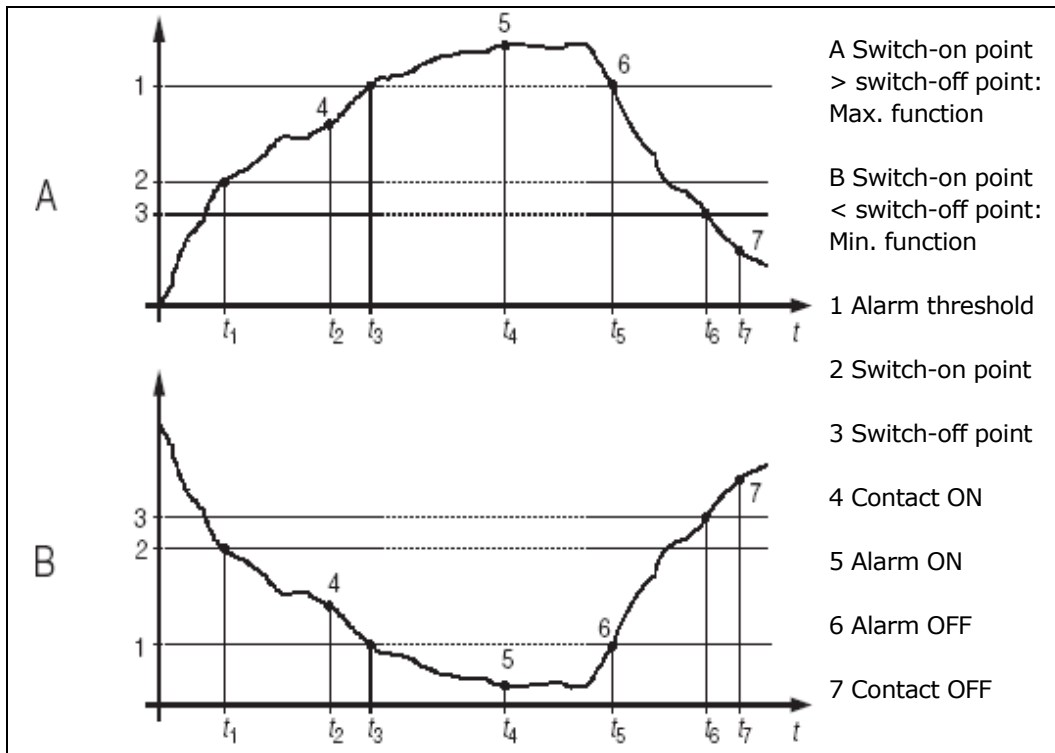


Figure 13. alarm and limit functions

- In case of control with base addition the limit contactor REL2 has to be closed to enable the PID control by REL1 if the process value is lower than the set point (SP). It can be opened again if the process value is rising above the set point. Therefore the on value (R212) is set pH 0.05 lower than the set point entered for the PID controller (SP) in R232. The off value (R213) is set equal to SP. Enter in R216 an alarm threshold lower than the switch off point in order to keep the limit contactor closed between its limits. R217 should show MIN which means that the minimum function was selected.
- In case of control with acid addition REL2 has to be closed to enable PID control if the process value is higher than the set point. It can be closed again if the process value is falling below the set point. Therefore the on value (R212) is set pH 0.05 higher than the set point entered for the PID controller (SP) in R232. The off value (R213) is set equal to SP. Enter in R216 an alarm threshold higher than the switch off point in order to keep the limit contactor closed between its limits. R217 should show MAX which means the maximum function was selected.
- The alarm contact is also configured in the Limit contact function. An alarm threshold (in case of base addition pH 0.2 lower than the set point and pH0.2 higher in case of acid addition) is set in R216. The alarm delay is set in another function group. Please go to function group "Alarm" and enter an alarm delay of 60 seconds in F3. The transmitter will switch the alarm contact when both the alarm threshold is reached and the alarm delay ($t_4 - t_3$ in Figure 13. alarm and limit functions) has expired.

3.4 pH electrode calibration

The "wet calibration" method is used. For this method two buffered calibration solutions with known pH (usually pH 7 and pH 4) and temperature are used. When these are set out, first of all the "CAL" button must be pushed and code 22 must be entered as described in paragraphs 4.5.1 and 4.5.2 (pages 20 and 21) in the manual. Then please perform steps C11 to C19 as described in paragraph 5.8.1 of the manual, pages 54 and 55. It may be useful to know that there is no potential matching line, and consequently it doesn't have to be connected to the buffer solution.

3.5 Gas analyser calibration

To determine the zero point when calibrating the analyser, pure N₂ must flow through the analyser. The direction of the valves is depicted in Figure 14. The compressor can keep on running. After calibration V-G-013 and V-G-014 can be switched back into normal position (Figure 15).

On the screen of the analyser, select Main menu -> calibration -> manual procedure.

Select then the Zero gas 1 which corresponds to N₂. Press Enter to start the internal procedure. After the start, a span delay time runs down, indicated by Wait..., then the real values are measured. Wait until End: Enter appears on the display and press Enter. Press again Enter to have the analyser compensate the drifts. Then press line 7: Calibr. cuvette. There the test gas is delivered instead of the nitrogen. Press as for the zero gas twice on Enter to complete the procedure.

The calibration of the gas analyser shall be performed once a week.

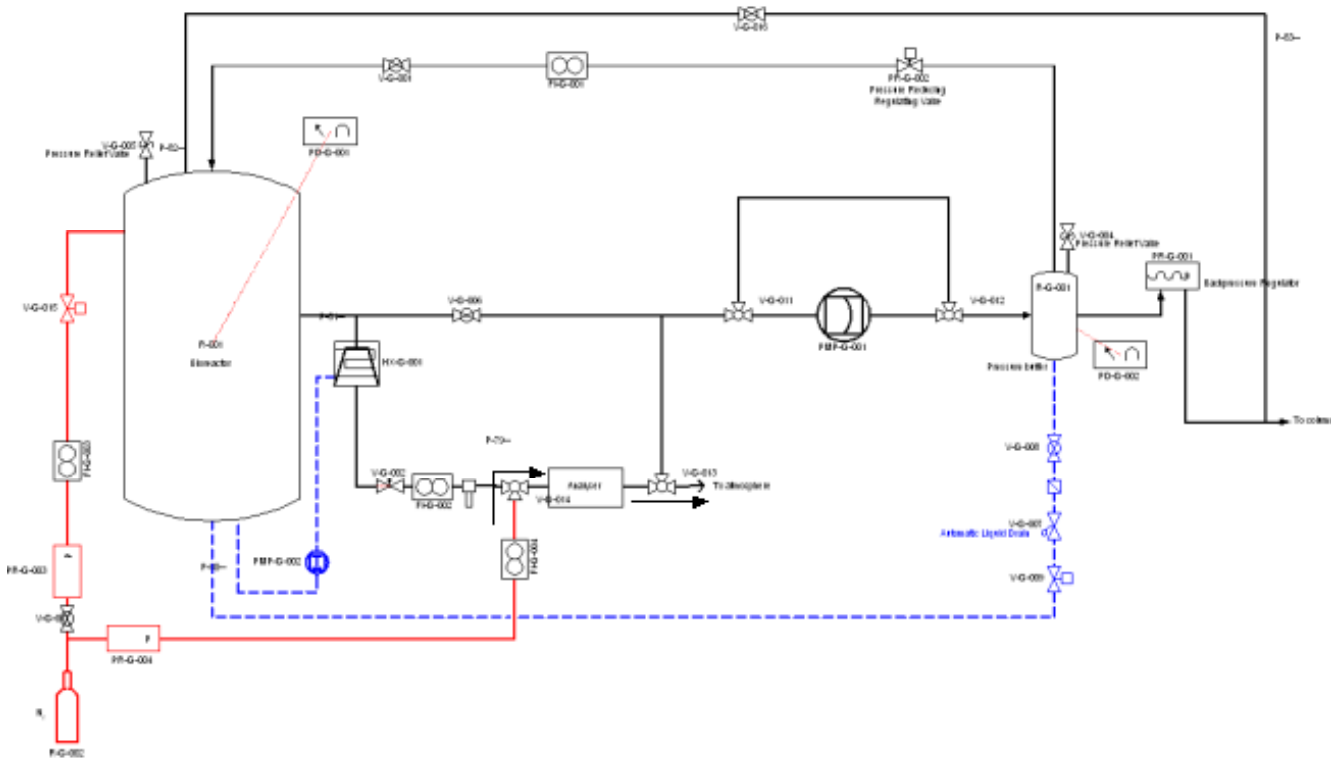


Figure 14. Calibration of the analyser

4. Working principles

The whole installation is operated automatically except for a few specific actions (addition of influent in influent tank, removal of effluent, removal of gas, calibrations).

4.1 Start up

4.1.1 Bioreactor

The first step consists to introduce the inoculum inside the bioreactor. Then the reactor must be closed and flushed with some nitrogen gas. Then the associated instrumentation (warm water bath, pH, T, P probes) can be started.

The computer interface is opened when starting the computer. Once opened, the user must enter settings for the influent feeding, and click on the stirrer on button to start the stirrer.

Data can be saved on-line by opening the icon "EWC logger 01.exe" on the desk of the computer screen. Then the user can select a name for the log file, and a frequency for saving data and saving the file.

4.1.2 Gas Loop

4.1.2.1 Cold start-up

Before the initial start-up of the gas loop, no overpressure is present in the gas loop. This is called "cold start-up".

The compressor is started by clicking the compressor on button on the computer interface. The direction of the valves is depicted in Figure 15. Valves without an arrow must be closed.

All the gas entering the buffer vessel is compensated by a same amount of gas leaving it to the bioreactor. Since an underpressure in the bioreactor must be avoided, a reasonable amount of gas must be added into the buffer reactor (e.g. N₂ or CO₂). This must be done by opening the needle valve V-G-015. This valve is succeeding a regulating valve PR-G-003 set at a pressure a bit higher than 100 mbar. This is to enable flushing agent to be pushed in the bioreactor while it is at its nominal pressure.

Note that by adding N₂ or CO₂, the concentration of O₂ is lowered (if O₂ is present). To obtain a minimal concentration of O₂, valve V-G-013 can be set with its output to the outer atmosphere to release all the oxygen.

If valve V-G-013 is switched back to its normal position the gas loop is entirely closed. The loop is put under pressure. While the compressor extracts gas from the bioreactor to pressurise the pressure buffer vessel, the bioreactor is kept at its nominal pressure by flushing gas through PR-G-003.

As the pressure buffer vessel gets at its nominal pressure the surplus of gas is sent to compartment II. To avoid this V-G-015 must be closed before this happens. At this point the gas loop is running. Note that at this point the gas loop contains nothing but flushing agent.

High flow rates through the analyser must be avoided (< 2 l/min). The flow through the analyser is indicated by FI-G-002 and can be regulated using V-G-002 and V-G-006.

4.1.3 Filtration Unit

When starting the filtration unit, the computer shall be switched on and the Mefia program opened. A password is necessary to use the program:

- the password: "Level1" allows the operation of the unit without modifying the program
- the password: "Level2" allows the operation of the unit with modifications of the program.

4.1.3.1 Cold Start up of the liquid loop

The liquid loop can be started up only when the bioreactor is filled with enough liquid. Initially all connections with gas loop and bioreactor are still closed in order to avoid oxygen to enter (V-G-016, V-F-001 and V-F-007). V-G-016 connects the buffer reactor's headspace to the atmosphere. The different regulating mechanisms are put in action one by one as follows:

- Level control in the buffer vessel is activated. At this moment V-F-001 is opened until R-F-001 is full.
- The other valves (except for V-G-016 and V-F-007) are put in their normal positions to produce filtrate. V-F-006 is opened entirely. It is used to regulate the TMP only when the nominal flow is reached.
- R-F-004 is emptied by opening V-F-013. The latter is closed again when the scale's output doesn't change anymore. This weight is kept in a variable of the control system in order to be able to calculate the amount of filtrate in R-F-004.
- The pump PMP-F-001 is not intended to run dry because friction forces between stator and rotor must be lowered by lubrication with the pumped liquid. However it is impossible to avoid this when the loop has never run before. This is not a problem if the pump runs dry only for only a short time (1 minute) and friction forces are kept low by keeping a low rotational speed. The liquid can be brought into the entrance part of the pump chamber by opening the screw bolt that is dedicated for this purpose. This bolt is closed again when or right before liquid starts running out of it. Then the pump is started at 20% of its full speed to progressively lubricate the rest of the pump house. Once fluid is leaving the pump at the outlet, it is regulated to the nominal flow rate. If the liquid loop is restarted the pump house is already lubricated and the pump can be started right away to reach and maintain the nominal flow rate.
- Most of the air in the loop is pushed through V-G-016 into the atmosphere. The level in R-F-001 decreases and is topped up by the level control. When the loop is completely filled with liquid material V-G-018 can be opened to get out the rest of the oxygen in the head space of R-F-001. Then V-G-016 can be switched to establish a connection with the gas loop, and V-G-018 can be closed.
- Then the TMP control can start to act on V-F-006 once the nominal flow rate has been reached.

4.1.3.2 Warm Start up of the liquid loop

Normally no oxygen is in the loop and the pump is lubricated. Start up can now happen fully automated.

- Level control in the buffer vessel is activated to make sure it is full.
- The other valves are put in their normal positions to produce filtrate. V-F-006 should be entirely open.

- V-F-013 is opened until the scale's output doesn't change anymore in order to empty R-F-004. This weight is kept in a variable of the control system in order to be able to calculate the amount of filtrate in R-F-004.
- The pump is started and its speed is set to the nominal flow rate.

TMP control is activated.

4.2 Nominal operation

4.2.1 Feeding

The influent tank shall be filled every day with cold influent. After reading the influent consumption and calculating the flow, the influent buffer tank can be filled with new influent. This can be done with the following procedure:

- Take new influent bottle.
- Open blue valve at top of influent buffer tank.
- Introduce the new influent with help of a funnel, until 4 to 5.5L.
- Flush influent tank with nitrogen: open the nitrogen tap and the valve on the Gas Loop labelled "Flush influent tank". Let flush for 1-2 minutes. Then close the two valves.
- Close blue valve and report new influent volume

4.2.2 Filtrate removal

After reading the filtrate volume on the filtrate tank, transfer the filtrate to a storage box or in a multibox for (for treatment) and rinse the tank. The filtrate stored is kept at -18°C in order to be possibly used in other Melissa experiments.

4.2.3 Sampling

4.2.3.1 Influent

When it is wanted, an influent sample can be taken directly on the influent after preparation, or in the storage bottles.

4.2.3.2 Reactor content

Samples can be directly taken on the bioreactor, from valve V-R-001.

4.2.3.3 Filtrate

Samples can be directly taken in the filtrate tank, at the output of the system.

4.2.3.4 Gas

Gas samples can be taken directly on the gas loop, from valve V-G-013, by connecting a syringe on the valve and open it before sampling.

4.2.4 Manual homogenization of liquid between bioreactor and filtration unit

This operation is not automated on the prototype compartment. It should be done several times a day.

To proceed, open first valve V-F-008 behind FU buffer tank and then close valve V-F-007. Let the sludge circulating for around 1 min. Then open first valve V-F-007 and then close valve V-F-008.

4.2.5 Calibrations

4.2.5.1 pH

The pH probe should be calibrated regularly (recommended frequency: once a week). However its calibration requires an opening of the bioreactor and thus a stopping of the Gas Loop and a perturbation of the gas phase equilibrium. Therefore, number of openings must be minimized as much as possible. pH can be measured off-line on reactor content samples with a calibrated probe. The regular comparison with on-line measurement allows to detect the real moment when a calibration is required.

When a calibration is required, stop the gas loop by clicking on the compressor stop button on PC interface and close valves V-G-001, V-G-006, V-G-014 and V-G-016. Then start calibration procedure on the pH interface of electrical cupboard and take out the pH probe from the top of the reactor. Then follow calibration procedure as described in section 3.4.

4.2.5.2 Gas analyser

The gas analyser should be calibrated once a week. A parallel loop was made to close the analyser from the reactor gas phase and to allow an independent calibration (see section 3.5).

5. Maintenance

Some instruments require regular specific actions:

- Influent pump: the inner tubing shall be changed every 6 months
- Feeding tank: tank and tubings shall be rinsed with tap water every 15 days
- Gas analyzer: the calibration cuvette shall be calibrated once a year
- Membrane: it shall be replaced when the flux is below 4 L/m².h

6. Troubleshooting

Specific problems can occur during the nominal operation of the system. This section describes the actions that can be taken when it occurs.

Table 1. Troubleshooting

| Action | Procedure | Special requirement | Possible problem | Check list | Troubleshooting |
|---------------------------------|--|---|-------------------------------------|---|--|
| Parameters /measurements | | | | | |
| 1. Temperature measurement | Read T on interface on screen of prototype PC, check enough water in water bath under reactor | T \approx 55°C (T \in [45-58]) | T \notin [45-58] | -check presence of water in the warm water bath under the reactor | -add distilled water to the water bath |
| | | | | -check that water bath+pump are running | |
| | | | | -check tuning of T on water bath thermometer | -adjust T set by turning the wheel at top of thermometer |
| 2. pH measurement | Read pH on interface on screen of prototype PC, measure base volume in bottle on the upper rack | 5.0<pH<6.0 (pH is controlled by addition of base) | pH<5 | -check presence of NaOH in bottle connected to reactor (upper rack) | -Add NaOH |
| | | | | -check that the pH probe extremity is in the liquid | |
| | | | | pH>6 | -check that the pH probe extremity is in the liquid |
| 3. Liquid volume measurement | Read volume on interface on screen of prototype PC | Volume can fluctuate slightly, no control/action is required. | | | |
| 4. Gas composition measurement | Read percentages of CO ₂ and CH ₄ on the interface on screen of the prototype PC | CH ₄ <2% | Problem on inhibition | -check pH (must be <6) | -cf pH measurement |
| 5. Gas volume measurement | Read volume on the column 1 next to the freezer + column 3, then adjust liquid level to 0 with the pump on the rack next to the column (NB: columns 1& 2 are connected together to the bioreactor, with the same level => measure on 1 only; column 3 is | | -lack of liquid in the columns bath | | -add tap water in the bath |

| | | | | | |
|---------------------------------|---|-------------------------------|-----------------------|--|---|
| | connected to buffer of FU) | | | | |
| Hardware | | | | | |
| 6. Mixing | Check visually that the reactor is mixed | reactor mixed | -reactor not mixed | -check that the "on"-button of the stirrer is present on interface of prototype pc | -if not: click on it |
| 7. Running of filtration Unit | Check that it is working (STOP must be present on the screen of the FU PC, on the FU frame) | Buffer tank must not be empty | -Buffer tank empty | | -stop FU by pushing the STOP button on screen of FU computer |
| | | | -FU not working | | -try to push START button on screen of FU computer; if it stops again, let it stopped. |
| 8. Homogenisation of FU content | Behind FU buffer tank, open orange valve on the left (V-F-008). Then close orange valve V-F-007 and let the sludge circulating for around 1min. Then open first valve V-F-007 and then close valve V-F-008. | | | | |
| Influent/ Effluent | | | | | |
| 9. Filtrate | Check volume and flow of filtrate produced | Permeate 2.5L/d | Permeate <2.5L/d | -check flux value | -adapt setting of overflow on the FU PC screen -clean or replace membrane |
| 10. Feeding influent | Open blue valve on top of influent tank and introduce influent (bottle in the fridge) with help a funnel (on the bioreactor rack). Fill the tank slowly to 5L. | | -clogging of the pump | | -pump briefly in the opposite sense (push the reverse button and then the start button on the driver to inverse the sense of pumping) -check maintenance of pump (replace tubing if necessary) |
| 11. Flushing influent tank | Open nitrogen tap on the wall behind the prototype to reach a few hundreds of mbar. Open the valve of the gas loop labelled "influent vessel flush" and let flushing for 1-2min. Then close valve on gas loop and blue valve on top of influent tank. Don't forget to close nitrogen tap | | | | |

7. Safety

When working with the Prototype Compartment, the following measures should be taken into account:

The entire manual and the manuals of the parts should be reviewed before operating the installation.

The use, maintenance or repairing is only to be performed by qualified persons.

When removing or opening any parts from the system, the installation should be unplugged from the main power.

The installation should be inspected regularly on correct functioning by qualified persons.

General laboratory safety conditions should be applied : wear safety goggles, laboratory gloves, lab coat, safety shoes.

An enclosure must be placed around the installation with a board : "restricted area, only for authorized persons" (in the local language).