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MELiSSA
ENGINEERING OF THE WASTE COMPARTMENT

ESA contract 15689/01/NL/ND

TECHNICAL NOTE 71.10.1

Life Test-Plan and Procedure

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ACRONYMES

COD : Chemical Oxygen Demand

CODs: COD soluble

CODt: COD total

DM: Dry Matter

EC: Electroconductivity

FU: Filtration Unit

GL: Gas Loop

MELiSSA : Micro Ecological Life Support System Alternative

NA: Non Applicable

Nt: Total Nitrogen

P&ID: Process and Instrumentation Diagram

RC: Reactor Content

VFA: Volatile Fatty Acids

1. INTRODUCTION

1.1 Purpose

The liquefying compartment of the MELiSSA loop is responsible for the biodegradation of human faecal material and other wastes (inedible parts of plant material) 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 CO_2 is supplied to the photoautotrophic compartment inoculated with the algal strain *Arthrospira platensis* and to the higher plants compartment.

At the pilot plant of the University of Barcelona, three compartments of the MELiSSA loop (photoheterotrophic compartment CII, nitrifying compartment CIII and photoautotrophic compartment CIVa) have already been 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.

After designing the compartment and building and testing an intermediate prototype, the pilot compartment is build. It is made of 3 frames, supporting the different pilot subsystems: bioreactor and influent tank, Filtration Unit (FU), Gas Loop (GL).

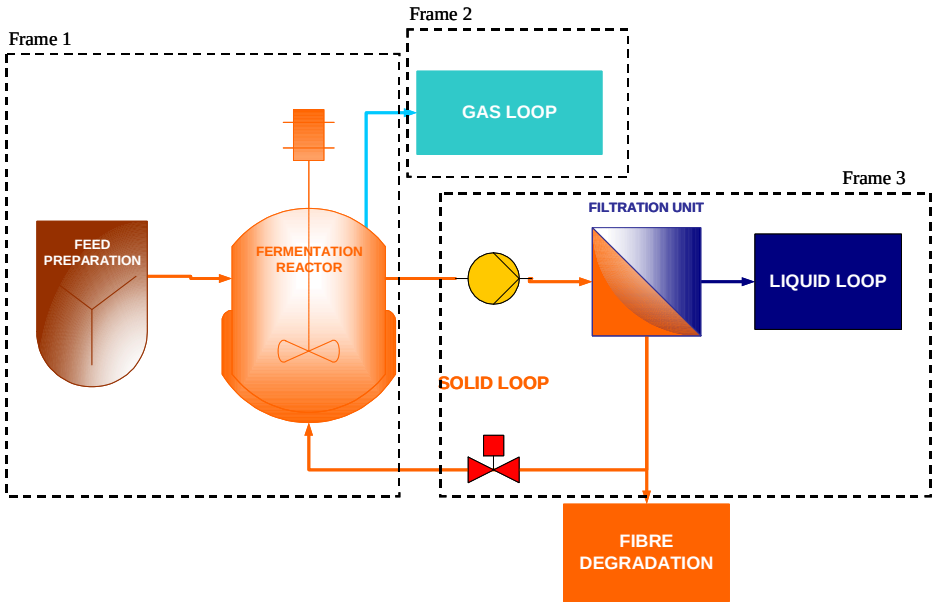

Figure 1. Concept of the pilot Compartment I

According to the Statement of Work, a Life Test Plan shall be defined in order to validate all levels of the selected hardware.

During the Life test period, the hardware validation (functional and operational tests) is realised and the process is studied in parallel. During this life test period, the reactor is operated in nominal mode.

This technical note proposes a test plan together with the corresponding procedures.

1.2 Testing Scope

The test plan will include the following topics:

1. **Hardware Tests**
   The hardware tests are performed on the instrumentation of each frame separately, after their delivery to EPAS, in parallel with the electricity and PLC connections and programming. They consist in checking that the instruments are in the required state to perform their function, and calibrating the sensors.

2. **Automation and Control Tests**
   These tests are also performed independently on the 3 frames, after the hardware tests and when the automation and control functions have been programmed in the PLC. The aim of these tests is to check that the system acts automatically like expected and that the control specifications are respected. The control can be optimised and validated based on these tests. The test of the complete system will be done during FU and process tests. Regarding automation and control, each procedure and control action is tested independently.

3. **Filtration Unit Tests**
   Because of its specific requirements, it is proposed to separate the FU tests from the others. The FU will be tested according to its requirements after being integrated in the compartment and tested from the hardware, control and automation point of view.

4. **Process Tests**
   These tests are dedicated to the study of the biological process itself. Therefore they are not real tests such as in the other sections: they are not submitted to a particular condition (test passes/ fails) but aimed to provide information about the process. The compartment requirements as defined in the early phase of the contract are fulfilled by the complete design of the system, including hardware selection, automation functions, control strategy, and operation. To validate the complete compartment, it is necessary to follow the installation at process level and to perform mass balance calculations. The process tests will moreover allow to define and optimize a tool for operating and following the process for the final user.

2. TEST ENVIRONMENT

The tests will be realised on the pilot compartment I, including:

- the Bioreactor and influent tank frame
- the Filtration Unit frame
- the Gas Loop frame
- the steam generator
- the PLC cupboard
- the computer interface.

The tests will be carried out in the EPAS laboratory. Extra material will be used when necessary, such as off-line sensors and analysers, material for bacterial counting...

The conceptual and detailed design of the compartment are described in the Design Report (see P&ID and Instrumentation list)

The results of the tests will be collected in technical not 71.10.3 and evaluated in technical note 71.10.4.. Problems and troubles will be clearly identified and corrective actions will be defined.

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3. Test plan

3.1 Hardware tests

The compartment is built in the form of 3 different frames supporting: Bioreactor and influent tank, Filtration Unit (FU) and Gas Loop (GL). Therefore the different frames are not built at the same time, but one after each other.
The hardware tests are performed on the instrumentation of each frame separately, after their delivery to EPAS, in parallel with the electricity and PLC connections and programming. They consist in checking that the instruments are in the required state to perform their function. These tests include e.g.:

- liquid/ gas tightness of tanks
- sensors calibration
- tanks volume calibration
- pumps flows calibration
- check of 3 way valves direction
- ...

Table 1 presents the detailed test plan for hardware tests.
Detailed tests procedures are described in section 4.1.

Table 1. Hardware Test Plan

Test case	Specifications/ Requirements	Instruments	Subsystem	Tag	Constraints	Acceptable (Test pass)	Moment	Duration	Test output
1	Liquid tightness	Tanks (mounted on frame with associated instrumentation), tubes	Bioreactor	R-V-01, R-R01, R-R-02, R-R-03		Total absence of leakage	Delivery and electrical integration of each frame	24 h	Check table
			FU	R-F-01					
			GL	R-G-01					
			Cleaning and sterilization	R-C-01, R-C-02, R-C-03					
2	Gas tightness (pressurized N2/air)	Tanks (mounted on frame with associated instrumentation), tubes	Bioreactor	R-V-01, R-R01, R-R-02, R-R-03		Total absence of leakage	Delivery and electrical integration of each frame	24 h	Check table
			FU	R-F-01					
			GL	R-G-01					
			Cleaning and sterilization	R-C-01, R-C-02, R-C-03					
3	Correct on-line measurements	Sensors	Bioreactor	LS-V-01, PI-V-01 PS-V-01, PS-V-02, PS-V-03, TS-V-01, TS-V-02, LS-R-01, LS-R-02, LS-R-03, pHS-R-01, pHS-R-02, PS-R-01, PS-R-02, TS-R-01, TS-R-02	Possibly: deviation range	Calibration performed	Delivery and electrical integration of each frame	Depending on sensor	Check table
			FU	FS-F-01, LS-F-01, LS-F-02, LS-F-03, PS-F-01, PS-F-02, PS-F-03, PS-F-04, PS-F-05, PS-F-06, PS-F-07, SS-F-01, TS-F-01, TS-F-02					
			GL	A-G-01, A-G-02, FS-G-01, FS-G-03, FS-G-04, FI-G-01, FI-G-02, FI-G-03, PI-G-01, PS-G-01, PS-G-02,PS-G-03					
			Cleaning and sterilization	PI-C, TS-C-01					
4	Correct volume measurement	Tanks	Bioreactor	R-V-01, R-R-01		Establishment of curve Volume =f(P)	Delivery and electrical integration of each frame; after tests 1, 2 and 3.	NA	Check table + calibration curve
			FU	R-F-01					
			GL						
			Cleaning and sterilization	R-C-01, R-C-02					
5	Correct flows	Pumps	Bioreactor	PMP-V-01, PMP-V-02, PMP-R-01, PMP-R-02, PMP-R-03		Establishement of set points	After Tests 1, 2, 3, 4	NA	Check table + set points
			FU	PMP-F-01, PMP-F-02, PMP-F-05					
			GL	PMP-G-01, PMP-G-02, PMP-G-03, PMP-G-04, PMP-G-05					
			Cleaning and sterilization	PMP-C-01, PMP-C02, PMP-C-03					
6	Correct 3-way valves position	Automated 3way valves	Bioreactor	V-V-03, V-V-04		Correct position	During construction phase of each frame	NA	Check table
			FU	V-F-02, V-F-03, V-F-04, V-F-05, V-F-07, V-F-08, V-F-14, V-F-15, V-F-16, V-F-17, V-F-18					
			GL	V-G-01, V-G-02, V-G-03					
			Cleaning and sterilization	V-C-12, V-C-13, V-C-14, V-C-15, V-C-16, V-C-17, V-C-18, V-C-19, V-S-02, V-S-03, V-S-04, V-S-05, V-S-06, V-S-07, V-S-08					

NA: Non applicable

The tests results will be presented in TN 71.10.2 in the form of tables. An example of empty results table is given in Table 2. When additional documents are made during the test (e.g. a volume calibration curve), they will be given in annex of the TN 71.10.2.

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Table 2. Example of Test result table

Test case	Specifications/ Requirements	Instruments	Subsystem	Tag	Date	Passed/ Failed	Remarks/ Extra documents
1	Liquid tightness	Tanks, tubes	Bioreactor	R-V-01			
				R-R-01			
				R-R-02			
				R-R-03			
			FU	R-F-01			
			GL	R-G-01			
			Cleaning and sterilization	R-C-01, R-C-02, R-C-03			
				R-C-02			
				R-C-03			

3.2 Automation and Control Tests

These tests are also performed independently on the 3 frames, after the hardware tests and when the automation and control functions have been programmed in the PLC. The aim of these tests is to check that the system acts automatically like expected and that the control specifications are respected. The control can be optimised and validated based on these tests.
The control and automated functions are defined by EPAS with support of SHERPA for control procedures. The corresponding test plan and test execution and interpretation will also be performed in interaction between EPAS and SHERPA, which expertise in control will allow to validate the compartment control.
Detailed tests procedures are described in section 4.2.

Table 3. Automation and control preliminary test plan

Test case	Function	Subsystem	Constraints	Acceptable (Test pass)	Moment	Duration	Test output
Control tests							
1	Temperature control	Influent & filtrate tanks	Set point: 4 °C (0,5<T<6)	T = 4 °C in R-V-01 and R-F-01	FU and influent subunits started	4h + 1/day	Check list
		Bioreactor	Set point: 55°C (54,5<T<55,5)	T = 55 °C in R-R-01	After inoculation	4h + 1/day	Check list
2	Pressure control	Bioreactor		P constant in R-R-01	After hardware tests GL	1,5h + 1/day	Check list
3	Volume control	Tanks		Constant volume	Subunits started	4h + 1/day	Check list
4	Gas flow control	Gas for analysis	Flow rate > 1L/min	Constant flow	GL started	1,5h + 1/day	Check list
5	pH control	Bioreactor R-R-01	5,1<pH<5,4	Constant	After inoculation	4h + 1/day	Check list
Safety tests							
6	Pressure safety	Bioreactor, influent tank, filtrate tank, GL, cleaning tanks	P<200 mbar	Gas is released when P > set point	After hardware tests	3 times	Check list
7	Level safety	Level switches in tanks		Corrective action when level > switch	After hardware tests	3 times	Check list
Other tests							
8	Cleaning procedure	FU	without stopping filtration	Respect of procedure	FU started	3 times	Check list
9	Sterilization procedure	FU	without stopping filtration	Respect of procedure	FU started	3 times	Check list
10	Mixing	Influent tank, bioreactor		Tanks content must be homogenous	After tank use started	3 times	Check list

3.3 Filtration Unit Tests

Because of its specific requirements, it is proposed to separate the FU tests from the others. The FU will be tested according to its requirements after being integrated in the compartment and tested from the hardware, control and automation point of view. The different tests will be therefore performed simultaneously in the last part of the Life test period. They will concern the following requirements:

- Barrier to bacteria
- Barrier to particles
- Selective products recovery

Detailed tests procedures are described in section 4.3.

Table 4. Filtration Unit Test Plan

Test case	Specifications/ Requirements	Subsystem	Constraints	Acceptable (Test pass)	Moment	Duration	Test output
1	Barrier for bacteria	FU	Production of a sterile filtrate	Filtrate sterile for same period as prototype without sterilization / continuously with sterilization	All subunits tested and started	4 weeks including sterilization procedures	Check list + graphs bacterial count in function of time
2	Barrier for particles	FU	Minimize particular material in filtrate	Proportion of particular COD retained in bioreactor equal or higher to the one obtained at prototype scale	All subunits tested and started	4 weeks	Check list + graphs particular COD in function of time
3	Selective products recovery	FU	Maximize VFA and NH4 concentrations in filtrate	Proportion of products recovery equal or higher to the one obtained at prototype scale	All subunits tested and started	4 weeks	Check list + graphs VFA/NH4 in filtrate compared to concentrations in R-R-01

3.4 Process Tests

These tests are dedicated to the study of the biological process itself. The compartment requirements as defined in the early phase of the contract are monitored by the complete design of the system, including hardware selection, automation functions, control strategy, and operation. To validate the complete compartment, it is also necessary to check that the requirements are respected at process level. The process tests will moreover allow to define and optimise a tool for operating and following the process for the final user.

The process will be followed through the total life test period, including the following periods:

- inoculation
- growing of inoculum
- stabilization (in batch)
- stabilization (in semi continuous)
- Start up of FU: accumulation of solid matter

The total period will be studied with a specific analysis plan. The test plan will allow to characterize the degradation process, the accumulation due to filtration, the production of VFA, NH4 and CO2, the amounts of minerals and elements... Mass balances will also be calculated.

The results will be compiled in a process database and presented in TN 71.10.2 in the form of graphs showing the evolution of the important process parameters and efficiencies in function of the time.

Detailed tests procedures are described in section 4.4.

Table 5. Process Test Plan

	Frequency (per week)			
Period: Growing of inoculum/ stabilization				
	Influent (/batch)	**Reactor content**	**Filtrate**	**Gas**
T	X	7	X	X
pH	1	7 (on line) 3 (off line)	X	X
DM	1	3	X	X
Ashes	1	3	X	X
EC	1	1	X	X
VFA	1	3	X	X
NH4+-N	1	3	X	X
N total	1	1	X	X
COD total	1	1/2weeks	X	X
COD soluble	1	1/2weeks	X	X
CHONSP	X	X	X	X
Minerals	X	X	X	X
CO_2, CH_4, H_2, O_2	X	X	X	3
Gas volume	X	X	X	7
Influent preparation	1/ 4 weeks			
Period: with FU & GL				
	Influent (/batch)	Reactor content	Filtrate	Gas

T	X	7 (in R-V-01, R-R-01, R-F-01)	7	X
pH	1	7 (on-line) 3 (off line)	3 (off line)	X
DM	1	2	2	X
Ashes	1	2	2	X
EC	1	1	3	X
VFA	1	3	*3*	X
NH4+-N	1	3	3	X
N total	1	1	3	X
COD total	1	1/2weeks	2	X
COD soluble	1	1/2weeks	2	X
CHONSP	2 (in total)	2 (in total)	2 (in total)	X
Fibres	2 (in total)	2 (in total)	2 (in total)	X
Minerals (Ag, As, Cd, Cr, Cu, Pb, Ni, Zn, Hg, Ca, Na)	2 (in total)	2 (in total)	2 (in total)	X
CO_2, CH_4				7 (on-line)
H_2, O_2				3 (off-line)
H_2S				1 (off-line)
Gas volume				7 (on-line)
Influent preparation	1 /week			

4. Test Procedures

The following section presents the procedures of the tests defined in the test plan. Objectives, conditions and output of the tests are also recapitulated.

4.1 Hardware tests

The following section presents the procedures to be used for the Hardware tests. The tests results will be compiled and presented in TN71.10.2.

Table 6. Test case 1: Liquid tightness

Test Case	**1**
Specification/ Requirement	**Liquid tightness (tanks, tubes)**
Tags	- R-V-01, V-V-01, V-V-04, TS-V-01, LS-V-02, V-V-05, V-V-09, PS-V-01, V-V-06, V-V-07, V-V-08, BL-V-01, No-C-01, LS-V-01, V-V-03 - R-R01, PS-R-02, LS-R-02, TS-R-01, pHS-R-01, pHS-R-02, V-R-01, V-R-02, V-R-03, V-R-08, V-R-20, V-R-09, V-R-10, V-R-13, V-R-14,V-R-16, V-R-11, V-R-15, V-R-17, V-R-05, No-C-02, V-R-04, BL-R-01, PS-R-01, V-R-07, V-R-06, V-R-19, V-R-18, LS-R-01 - R-R-02: PMP-R-01 - R-R-03, PMP-R-02 - R-F-01LS-F-02, V-S-08, V-F-10, V-G-21, TS-F-01, LS-F-01, V-F-12, LS-F-03, No-C-03 - R-G-01, V-G-26, V-G-05, V-G-06, LS-G-01, PI-G-01, PS-G-01 - R-C-01, V-C-10, PMP-C-01, LS-C-02, LS-C-01, V-C-11, V-C-08, V-C-09 - R-C-02, PMP-C-03, V-C-05, V-C-06, TS-C-01, LS-C-04, LS-C-03, V-C-07 - R-C-03, PMP-C-02
Moment / Duration	The test will be performed during 24 hours after delivery and integration of each frame.
Test objective	The test must allow to check the liquid tightness of the tanks.
Test procedure	1. Close the tested tank (valves, sensors...) 2. Fill the tank with water up to the maximum liquid level it can contain 3. Flush pressurized air up to a pressure of 2 bar (for R-R-01, R-V-01, R-F-01, R-G-01 and R-G-02) 4. Then let the tank for 24 h and control regularly the presence or absence of liquid leakages.
Acceptable (Test pass)	Total absence of leakage after 24 h.
Test output	Check table

Table 7. Test case 2: Gas tightness

Test Case	**2**
Specification/ Requirement	**Gas tightness (tanks, tubes GL)**
Tags	- R-V-01, V-V-01, V-V-04, TS-V-01, LS-V-02, V-V-05, V-V-09, PS-V-01, V-V-06, V-V-07, V-V-08, BL-V-01, No-C-01, LS-V-01, V-V-03 - R-R01, PS-R-02, LS-R-02, TS-R-01, pHS-R-01, pHS-R-02, V-R-01, V-R-02, V-R-03, V-R-08, V-R-20, V-R-09, V-R-10, V-R-13, V-R-14,V-R-16, V-R-11, V-R-15, V-R-17, V-R-05, No-C-02, V-R-04, BL-R-01, PS-R-01, V-R-07, V-R-06, V-R-19, V-R-18, LS-R-01 - - R-F-01, LS-F-02, V-S-08, V-F-10, V-G-21, TS-F-01, LS-F-01, V-F-12, LS-F-03, No-C-03 - R-G-01, V-G-26, V-G-05, V-G-06, LS-G-01, PI-G-01, PS-G-01 - R-G-02, V-G-07, PS-G-04, TS-G-01
Moment / Duration	The test will be performed during 24 hours after delivery and integration of each frame.
Test objective	The test must allow to check the gas tightness of the tanks.
Test procedure	1. Close completely the tested tank (valves, sensors...) 2. Flush pressurized air inside the tank (while following the inside pressure) up to around 2 bar 3. Then let the tank for 24 h and control regularly the pressure inside
Acceptable (Test pass)	Total absence of leakage: the inside pressure after 24 h must be the same as the initial inside pressure.
Test output	Check table

Table 8. Test case 3: Correct on-line measurement

Test Case	**3**
Specification/ Requirement	**Correct on-line measurement**
Moment / Duration	The test will be performed after delivery and integration of each frame.
Test objective	The test must allow to check that the sensors give correct measurements.
Tags	LS-V-01, PI-V-01 PS-V-01, PS-V-02, PS-V-03, TS-V-01, LS-R-01, LS-R-02, LS-R-03, pHS-R-01, pHS-R-02, PS-R-01, PS-R-02, TS-R-01, TS-R-02, FS-F-01, LS-F-01, LS-F-02, LS-F-03, PS-F-01, PS-F-02, PS-F-03, PS-F-04, PS-F-05, PS-F-06, PS-F-07, SS-F-01, TS-F-01, TS-F-02, A-G-01, A-G-02, FS-G-01, FS-G-03, FS-G-04, FI-G-01, FI-G-02, FI-G-03, PI-G-01, PS-G-01, PS-G-02,PS-G-03, PI-C, TS-C-01
Test procedure	1. Pressure sensors: - No calibration is required (already performed before delivery) - The pressure is measured by several sensors in the system, and double check can therefore easily be done 2. Temperature sensors: double check with a portable temperature analyser 3. pH sensors: - initial calibration procedure following manual, with 2 standard solutions at pH 4 and 7 - double check with a portable pH probe off-line on a sample of the bioreactor - double check: the pH is measured by 2 different probes in the bioreactor 4. Flow sensors: see test 5 5. SS sensor: initial calibration procedure following manual 6. Gas analysers: - initial calibration procedure following manual - double check with portable infra-red analyser (for CO2 and CH4) off line on a sample of the gas phase of the bioreactor
Acceptable (Test pass)	It is determined by the specific manual of each sensor.
Test output	Check table

Table 9. Test case 4: Correct volume measurement

Test Case	**4**
Specification/ Requirement	**Correct volume measurement (tanks)**
Tags	R-V-01, R-R01, R-F-01, R-C-01, R-C-02,
Moment / Duration	The test will be performed after delivery and integration of each frame. Also after tests 1, 2 & 3.
Test objective	The test must allow to provide a correct liquid volume measurement in the tanks.
Test procedure	1. Let one valve open so that the pressure inside the tank is atmospheric pressure. 2. Fill the tank with water, litre per litre, up to its maximum capacity. 3. After each litre filled, report the pressures measured at the 3 different levels of the tank: 4. Plot the calibration curve $P_{liq} = aV + b$ 5. The volume must be corrected with the differential gas pressure in the tank, using the following formula:

	$V = (P_{liq} - P_{gas}) \times \frac{1}{a} + \frac{b}{a}$ Where P_{liq} = pressure in liquid phase P_{gas} = pressure in gas phase V = volume
Acceptable (Test pass)	Establishment of a linear calibration curve and equation
Test output	Check table and calibration curve

Table 10. Test case 5: Correct flows

Test Case	**5**
Specification/ Requirement	**Correct flows (pumps)**
Moment / Duration	The test will be performed after tests 1, 2, 3, 4.
Test objective	The test must allow to provide set points for correct gas and liquid flows
Tags	Liquid pumps: PMP-V-01, PMP-V-02, PMP-R-01, PMP-R-02, PMP-R-03, PMP-F-01, PMP-F-02, PMP-F-05, PMP-G-03, PMP-G-04, PMP-C-01, PMP-C02, PMP-C-03
Test procedure	1. Fill the tank at inlet of the pump 2. Start the pump at the expected settings and report the volume pumped in function of the time. 3. Determine the right set point(s) based this function.
Tags	Gas pumps: PMP-G-01, PMP-G-02, pressure regulators
Test procedure	1. Flush some nitrogen gas in the loop of the tested pump 2. Check flows on flow indicators 3. Determine manually the right set points.
Acceptable (Test pass)	The expected flow is obtained.
Test output	Check table, list of set points.

Table 11. Test case 6: Correct 3-way valves position

Test Case	**6**
Specification/ Requirement	**Correct 3-way valves position**
Tags	V-V-03, V-V-04, V-F-02, V-F-03, V-F-04, V-F-05, V-F-07, V-F-08, V-F-14, V-F-15, V-F-16, V-F-17, V-F-18, V-G-01, V-G-02, V-G-03, V-C-12, V-C-13, V-C-14, V-C-15, V-C-16, V-C-17, V-C-18, V-C-19, V-S-02, V-S-03, V-S-04, V-S-05, V-S-06, V-S-07, V-S-08
Moment / Duration	The test will be performed during the construction phase of each frame.
Test objective	The test must allow to check that all 3-way valves are correctly positioned.
Test procedure	1. Establish tables with the right positions of the valves (based on P&ID drawing) (see example in addendum in section 6) 2. Check visually at constructor's site that the valves are in their right position. 3.
Acceptable (Test pass)	The valves are in the right position.
Test output	Check table

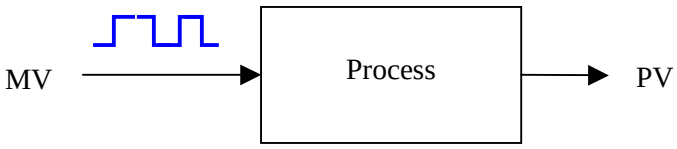
4.2 Automation and Control tests

4.2.1. Control tests

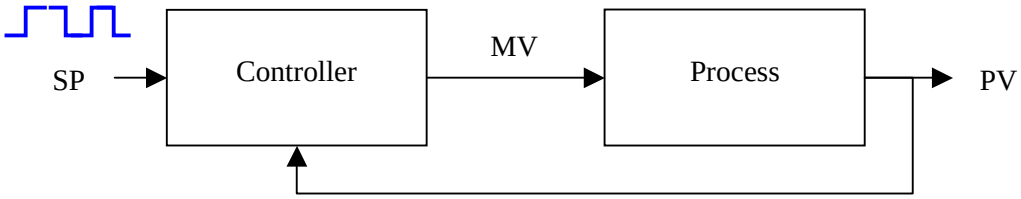
The objective of the study is to analyse the level 0 control behaviour for the waste compartment. For this, it is necessary to apply tests during normal functioning of the pilot. Each test requires therefore that the corresponding sub-unit is in operation. The tests will be performed in the real operation conditions, meaning with the inoculum.

Two possible ways of tests are possible:

1. Open loop test if no control is implemented
   In this case the test signal is applied on manipulated variables of the process

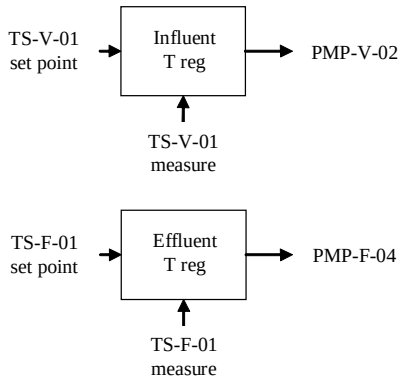
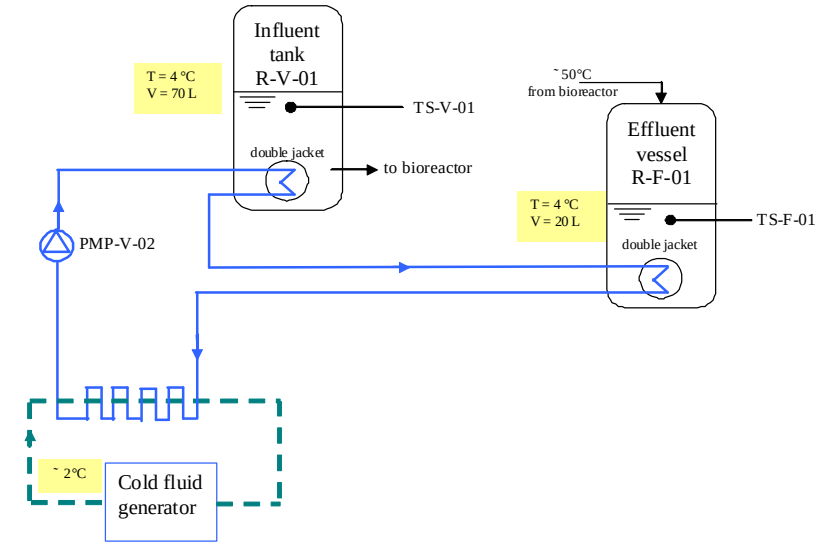


2. Closed loop test for the existing controllers.
   In this case the test signal is applied on the set point(s) of the controller.



Additional tests will be performed to check the safety procedures and Filtration procedures (cleaning, sterilization). There are no specific control loops in the Filtration Unit.

Table 12. Test case 1.1: temperature in the influent and effluent tanks

Test Case	**1.1**
Requirement	**Temperature control in influent and effluent tanks**
Control specification	1. Set point and constraints - Set point ≈ 4°C - Min constraint = 0.5 °C 2. Actuators and manipulated variables - Only the cooler can be steered on/off. 3. Sensors Both temperatures are measured: TS-V-01 and TS-F-01. 4. Disturbances The temperature of the incoming products.
Tags	TS-V-01, TS-F-01, TS-F-02, PMP-V-02, HX-V-01
 The same cooling fluid (glycol water) is used for both systems. The double jackets of the two vessels are put in series. There is only one pump: PMP-V-02. This pump runs constantly and cannot be steered by the PLC. Only the cooler can be steered on/off. .	 Volume Influent tank = 70 L The influent tank can be filled once a week with 70 L at ambient temperature.
Moment / Duration	The test will be performed when the control is already working. Test duration : 4 h
Test objective	The upward steps (last two hours) will show the influence of the thermal losses.
Test procedure	Protocol Test: Step change of the set points 1. Conditions of tests: - No influent injected into the bio reactor. - Nominal conditions for the tanks.

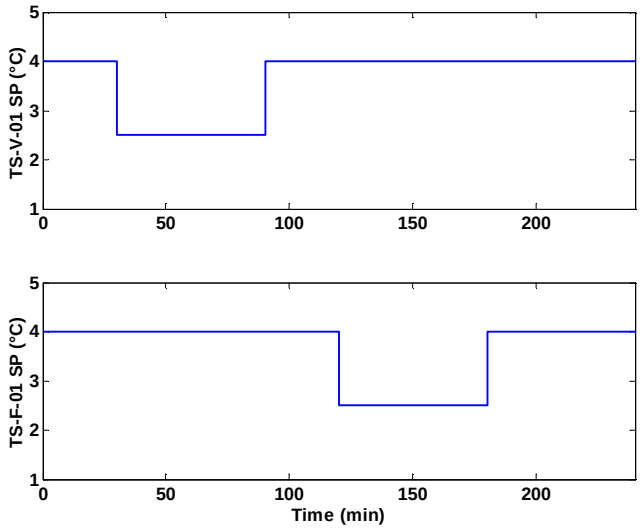
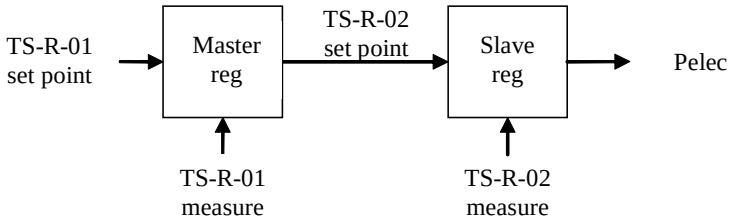
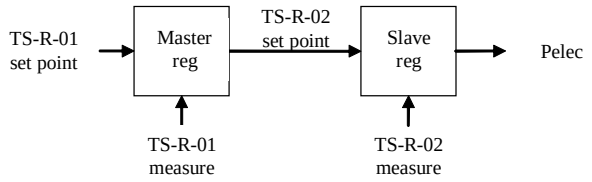
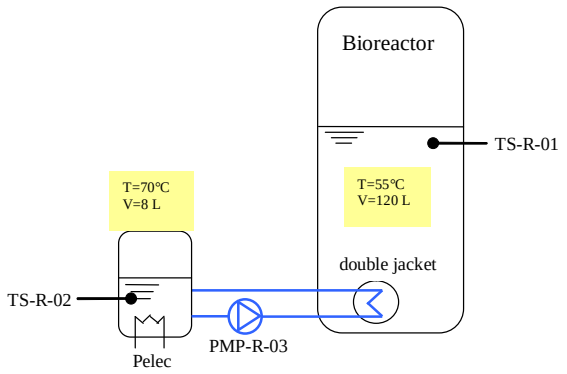
	 2. Variables to be recorded: - TS-V-01 set point and measurement - TS-F-01 set point and measurement - PMP-V-02 pump state (on/off). - PMP-F-04 pump state (on/off). - Cooling fluid temperature if it exists. - V-V-03 valve position (influent input) - V-F-08 valve position (effluent side)
Acceptable (Test pass)	The output of the test can be used to optimize the control.
Test output	Log file

Table 13. Test case 1.2: Temperature control in the bioreactor

Test Case	**1.2**
Requirement	**Temperature control in bioreactor**
Control specifications	1. Set point and constraints - Max constraint = 60 °C - Steady state set point = 55 ± 0.5°C - Start-up set point = ramp from 20 to 55 °C (max ramp to be defined) 2. Actuators and manipulated variables - Heating via hot fluid (water): o The temperature of the hot fluid (≈ 70 °C in steady state condition) is controlled by an electric resistance o Its flow rate is constant o No cooling device. 3. Sensors - The temperature in the bioreactor is measured: TS-R-01. - The temperature of the warm water bath is measured: TS-R-02 4. Disturbances - Temperature of the influent product (≈ 4 °C) which is fed semi-continuously - Temperature of the recirculation product from filtration - Fluctuation of the ambient temperature - Thermal losses 5. Current control strategy A cascaded loop strategy is implemented in PLC Quantum (concept language): 
Tags	TS-R-01, TS-R-02, PMP-R-03, HX-R-01
	
Moment / Duration	The test will be performed when the control is already working. Test duration : 4 h
Test objective	The downward step (last two hours) will show the influence of the thermal losses

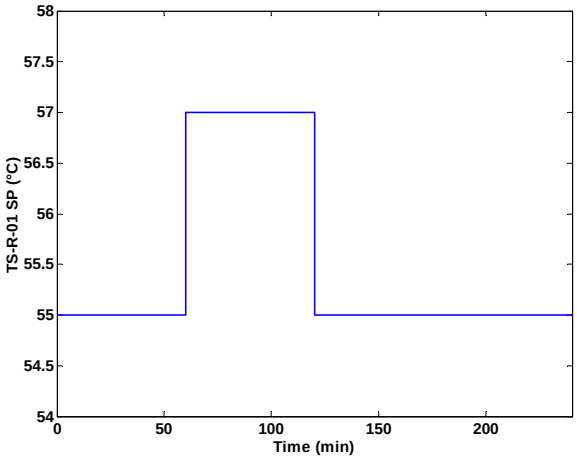
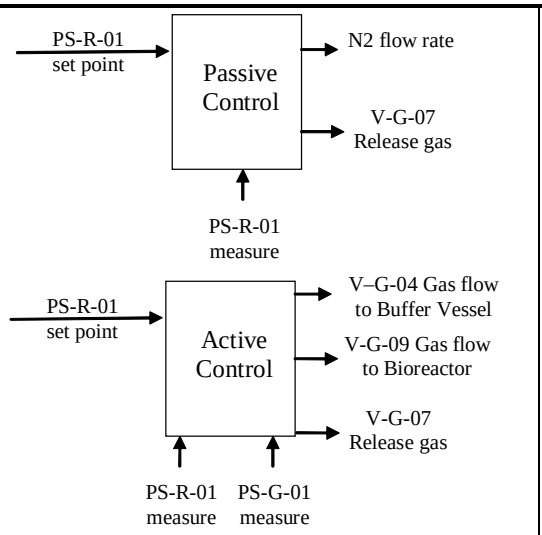
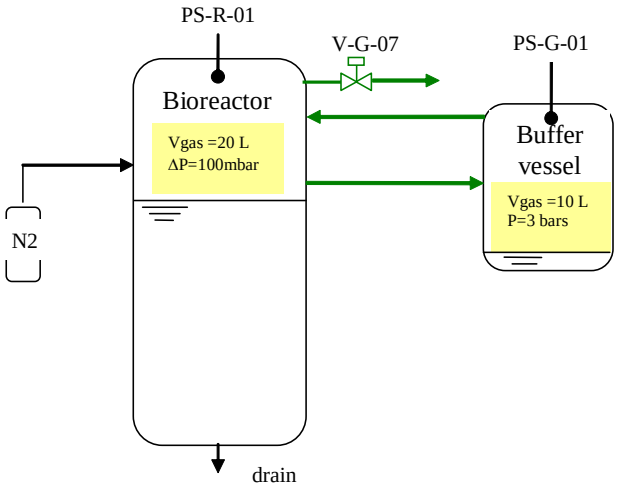
Test procedure	Protocol Test: Step change of the set points 1. Conditions of tests (first test case): - No influent injected into the bio reactor. - Nominal conditions for the reactor (pressure, volume and pH) and for the hot water tank. 2. Conditions of tests (second test case) - FU connected - Influent injected - Temperature set-point constant in the bioreactor  3. Variables to be recorded (in both test cases): - TS-R-01 set point and measurement - TS-R-02 set point and measurement - Pelec: Electrical power of the heating source. - V-V-03 valve position (influent input) - PMP-F-02 pump speed (effluent). - V-F-08 valve position (effluent side)
Acceptable (Test pass)	The output of the test can be used to optimize the control.
Test output	Log file

Table 14. Test case 2: Pressure control in bioreactor

Test Case	**2**
Requirement	**Pressure control in bioreactor**
Control specifications	1. Set point and constraints - Set point: $P_{gaz} = P_{atmospheric} + 100$ mbar - Constraints: $P_{atmospheric} < P_{gaz} < P_{atmospheric} + 500$ mbar 2. Actuators and manipulated variables - Release of gas outside the reactor with valve V-G-07 - Flush of N2 into the reactor - Gas flow rate: • from the reactor to the pressurized gas buffer vessel (by mean of a compressor PMP-G-01) • or from the gas buffer vessel to the reactor via regulating valve V-G-09 3. Sensors - The pressure in the gas phase of the bioreactor is measured: PS-R-01 - The pressure in the gas phase of the gas buffer tank is measured: PS-G-01 4. Disturbances - Draining which decreases the pressure (most important disturbance) - Gas generated by the reaction - Difference between influent and effluent flow rates - Remark: influent is fed semi-continuously (about 400 mL each hour) while effluent is taken continuously. 5. Current control strategy Two strategies are considered: - Passive control: • If $\Delta P > \Delta P_{set\text{-}point}$, the valve V-G-07 is opened to release gas • If $\Delta P < \Delta P_{set\text{-}point}$, the valve V-G-07 is closed and N2 is flushed in the bioreactor - Active control: an internal gas recirculation loop is used between the bioreactor and a pressurized gas buffer vessel: • If $\Delta P < \Delta P_{set\text{-}point}$, circulation of gas from the buffer to the bioreactor • If $\Delta P > \Delta P_{set\text{-}point}$: • circulation of gas from the bioreactor to the buffer if $P_{buffer} < 3$ bars • opening of the valve V-G-07 to release gas if $P_{buffer} > 3$ bars o Drawback of passive control: consumption of N2 (open loop) o Drawback of active control: condensation of water in the buffer and the compressor, which raises problems at start-up, noises and vibrations.
Tags	PS-R-01, V-G-07, RS-G-01, R-R-01, R-G-01





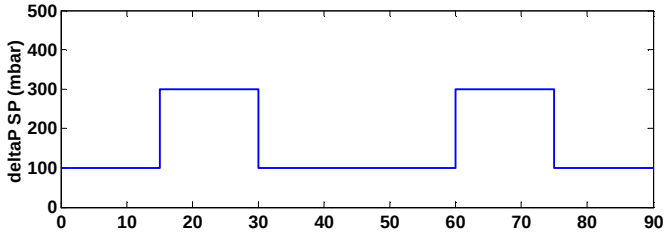
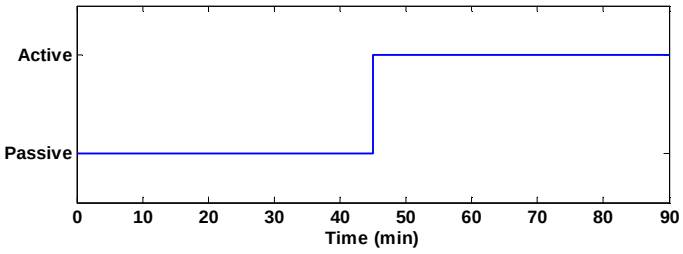
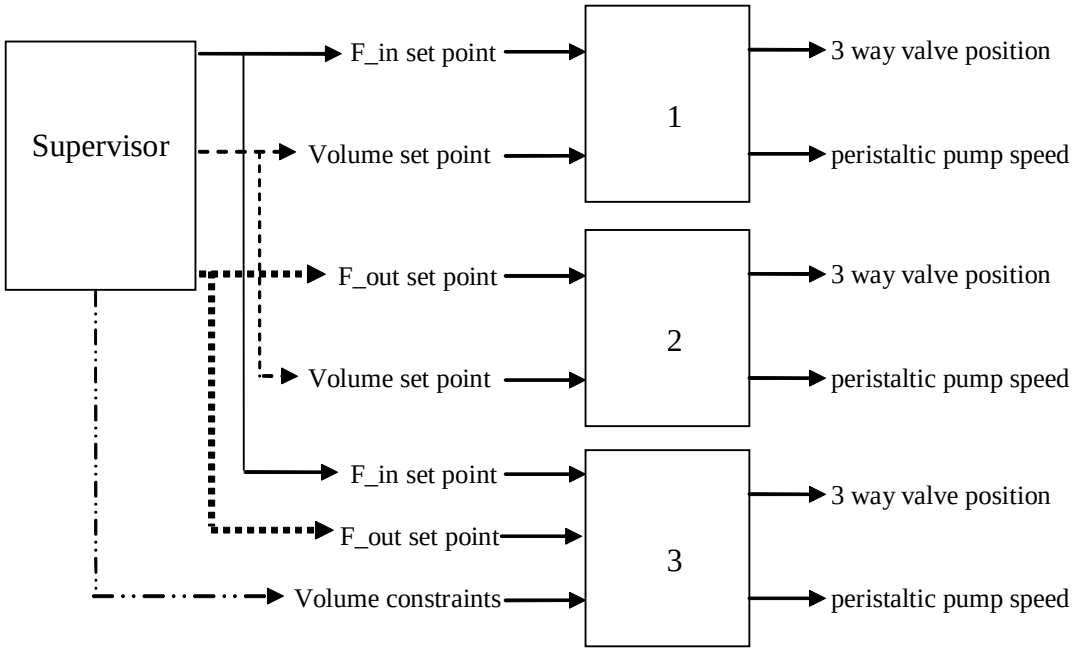
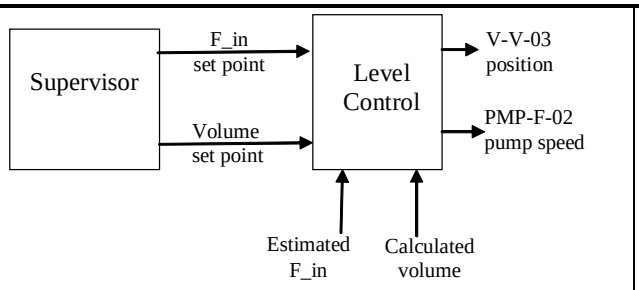
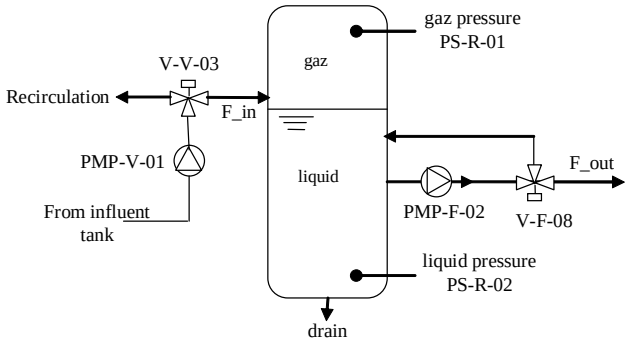
Moment / Duration	The test will be performed when the control is already working. Test duration : 1.5 h
Test objective	Optimisation of the control
Test procedure	Protocol Test: Step change of the set point 1. Conditions of tests:   2. Variables to be recorded: - PS-R-01 and PS-G-01 pressure measurement - V-G-04, V-G-07 and V-G-09 valves position - N2 flow rate into the bioreactor
Acceptable (Test pass)	The output of the test can be used to optimize the control.
Test output	Log file

Table 15. Test case 3: Bioreactor Volume control

Test Case	**3**
Requirement	**Bioreactor volume control**
Control specifications	1. Set point and constraints - Nominal set point = 100 L - Max constraint = 110 L - Min constraint = defined by program 2. Actuators and manipulated variables - The level is controlled by acting on the volumetric influent or/and effluent flow rates: - The input flow F_in is manipulated via an on/off 3 way valve V-V-03. - The output flow F_out is controlled by a peristaltic pump PMP-F-02. 3. Sensors - The level (and volume) of liquid in the bioreactor is calculated from the pressure measurements. - No flow is measured. 4. Disturbances - The variation of incoming flow F_in, as the feeding is done semi-continuously. - Draining happens rarely. 5. Current control strategy The flow rate set point is defined by a supervisor, typically 10 L/day. Three strategies can be considered (the current one is the first one): 
Tags	V-V-03, PMP-F-01, PMP-V-01





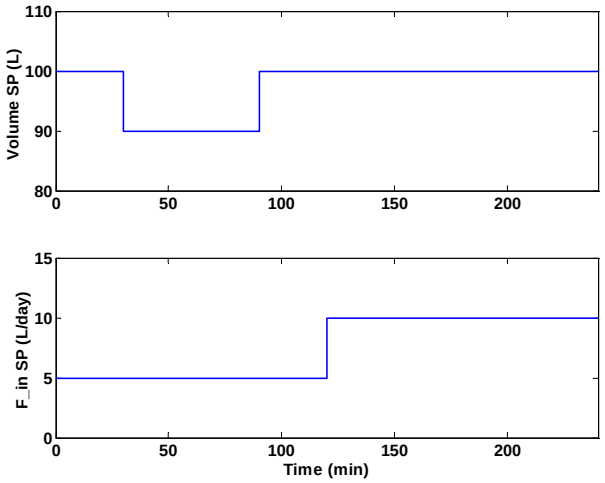
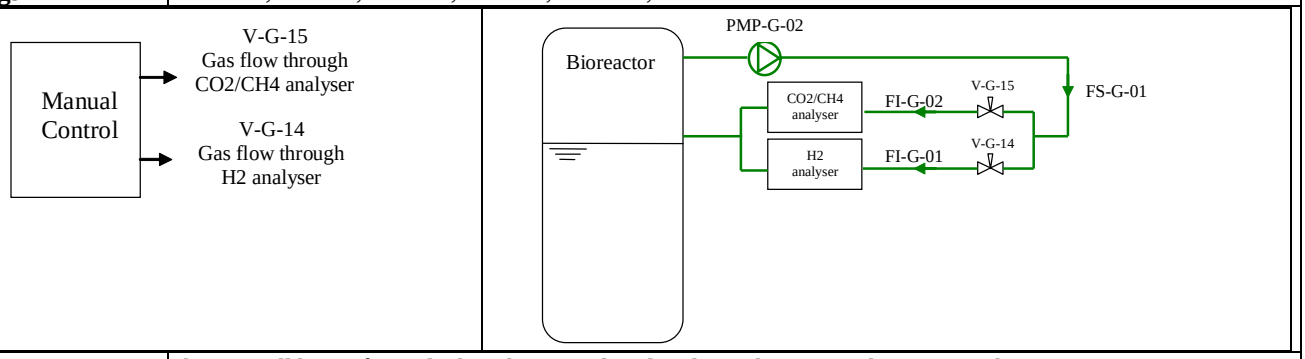
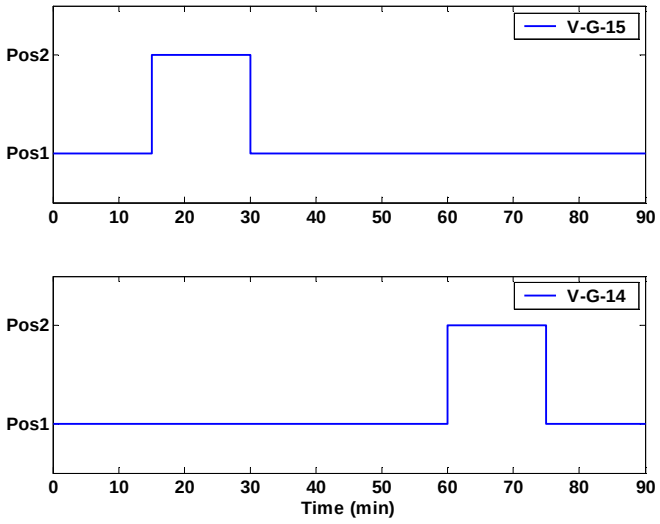
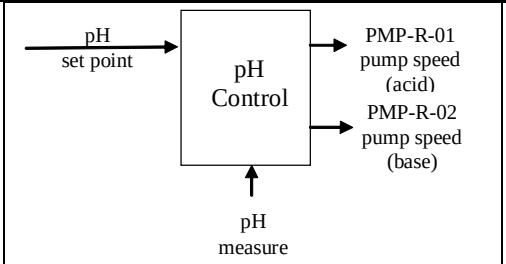
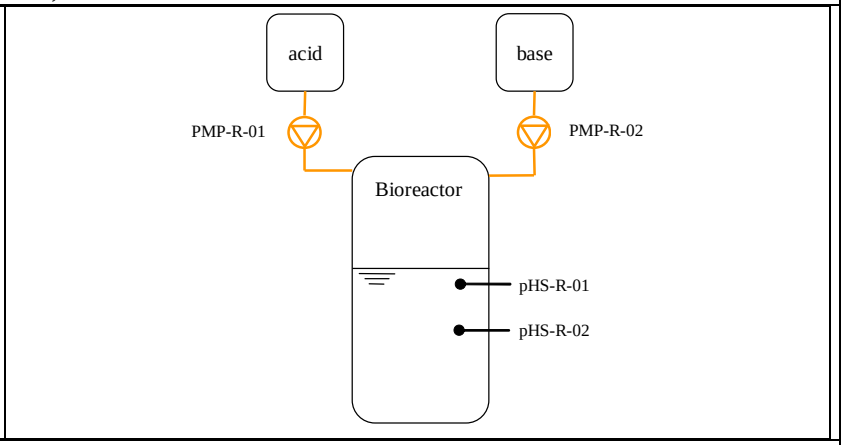
Moment / Duration	The test will be performed when the control is already working. Test duration : 4 h
Test objective	Optimisation of the control
Test procedure	Protocol Test: Step change of the set point 1. Conditions of tests: - No drain - No action on the influent flow (constant) - Increase of effluent flow  2. Variables to be recorded: - PS-R-01 gas pressure measurement - PS-R-02 liquid pressure measurement - Calculated Level and Volume of the bioreactor - V-V-03 valve position (influent input) - Calculated or measured influent flow rate F_in - PMP-F-02 pump speed (effluent). - V-F-08 valve position (effluent side) - Calculated or measured effluent flow rate F_out
Acceptable (Test pass)	The output of the test can be used to optimize the control.
Test output	Log file

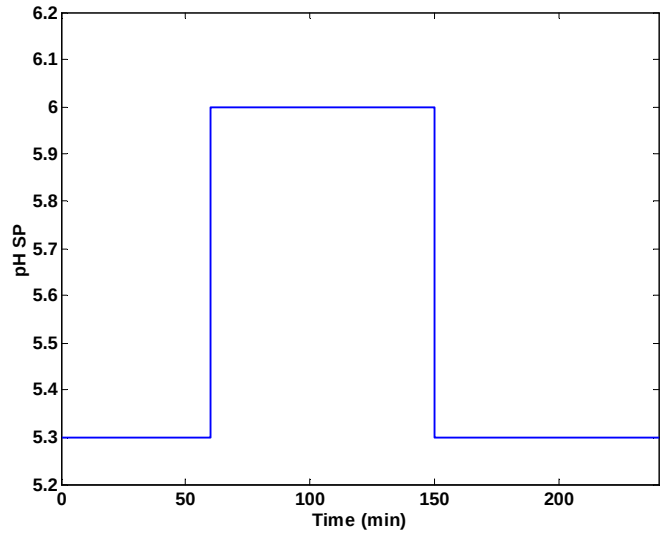
Table 16. Test case 4: Gas flow control

Test Case	**4**
Requirement	**Gas flow control**
Control specifications	1. Set point and constraints - Minimum flow rate = 1 L/min 2. Actuators and manipulated variables - Manual valves: o V-G-15 to regulate the gas flow through the CO2/CH4 analyser o V-G-14 to regulate the gas flow through the H2 analyser 3. Sensors - Measure of gas flow through the analysers: FS-G-01 4. Actual control strategy - Manual control
Tags	V-G-G14, V-G-15, FI-G-01, FI-G-02, FS-G-01, PMP-G-02
	
Moment / Duration	The test will be performed when the control is already working. Test duration : 1.5h
Test objective	Optimisation of the control
Test procedure	Protocol Test: Step change of the set point 1. Conditions of tests: 

	2. Variables to be recorded: - FS-G-01, FI-G-01 and FI-G-02 flow rate measurement - V-G-14 and V-G-15 valves position - PMP-G-02 speed
Acceptable (Test pass)	The output of the test can be used to optimize the control.
Test output	Log file

Table 17. Test case 5: pH control

Test Case	**5**
Requirement	**pH control**
Control specifications	1. Set point and constraints - Constraints: o 5 < pH < 7.5 o -Δ < dpH/dt < Δ - Set point: 5.1 < pH < 5.4 (in steady state or transient conditions) 2. Actuators and manipulated variables - Acid or base can be added in the bioreactor: peristaltic pumps PMP-R-01 and PMP-R-02. 3. Sensors - pH is measured continuously by means of two pH probes: pHS-R-01 and pHS-R-02 (redundancy). 4. Disturbances o The product in the reactor becomes naturally acid: pH decreases 0.1 a day. o Influent flow rate (either acid or basic) 5. Current control strategy The main issues are: o the validation of the measures o the accuracy of the sensors - The PLC generate an alarm when the values of the two pH measurements become too different.
Tags	PHS-R-01, pHS-R-02, PMP-R-01, PMP-R-02
	
Moment / Duration	The test will be performed when the control is already working. Test duration : 4h
Test objective	Optimisation of the control
Test procedure	Protocol Test: Step change of the set point 1. Conditions of tests: - No influent injected into the bio reactor.

	 2. Variables to be recorded: - pHS-R-01 and pHS-R-02 pH measurement - PMP-R-01 pump speed (acid) and PMP-R-02 pump speed (base) - V-V-03 valve position (influent input).
Acceptable (Test pass)	The output of the test can be used to optimize the control.
Test output	Log file

4.2.2. Safety tests

Table 18. Test case 6: Pressure safety

Test Case	**6**
Requirement/ Specification	**Pressure safety**
Tags	R-V-01, R-R-01, R-F-01, V-G-16, V-V-07, V-V-08, V-R-04, V-R-19, V-F-12, V-C-11, V-C-07
Moment / Duration	The test will be performed when the control is already working. Test repeated 3 times.
Test objective	Check that the gas is released when the pressure increases above the set point of the vents
Test procedure	1. Close the concerned loop or tank 2. Flush pressurized air or nitrogen and monitor the pressure evolution
Acceptable (Test pass)	The pressure shall not increase above the set point.
Test output	Check table, list of set points

Table 19. Test case 7: Level safety

Test Case	**7**
Requirement/ Specification	**Level safety**
Tags	R-V-01, LS-V-02, R-R-01, LS-R-01, R-C-02, LS-C-03, R-C-01, LS-C-01
Moment / Duration	The test will be performed when the control is already working. Test repeated 3 times.
Test objective	Check that an alarm is given when the liquid level increases above its limit
Test procedure	1. Increase the volume in the tested tank: a. R-V-01: with influent b. R-R-01: with influent (same flow – wait to drain manually) c. R-C-01 and R-C-02: with water 2. Monitor the volume evolution
Acceptable (Test pass)	An alarm must be given when the level increases above the set point (and possibly activation of the corrective action)
Test output	Check table, list of set points

4.2.3. Others

Table 20. Test case 8: Cleaning procedure

Test Case	**8**
Requirement/ Specification	**Cleaning procedure**
Tags	FU and cleaning system
Moment / Duration	The test will be performed when the control is already working. Test repeated 3 times.
Test objective	Check that the procedure is respected
Test procedure	1. Run manually the cleaning procedure from the PLC (FU filled with water) 2. Check the actions
Acceptable (Test pass)	The actions happen according to the written procedure
Test output	Check table

Table 21. Test case 9: Sterilization procedure

Test Case	**9**
Requirement/ Specification	**Sterilization procedure**
Tags	FU and sterilization system
Moment / Duration	The test will be performed when the control is already working. Test repeated 3 times.
Test objective	Check that the procedure is respected
Test procedure	1. Run manually the sterilization procedure from the PLC (FU filled with water) 2. Check the actions
Acceptable (Test pass)	The actions happen according to the written procedure
Test output	Check table

Table 22. Test case 10: Mixing

Test Case	**10**
Requirement/ Specification	**Mixing**
Tags	R-R-01, BL-V-01, PMP-V-01, R-V-01, BL-R-01
Moment / Duration	The test will be performed when the control is already working. Test repeated 3 times.
Test objective	The mixing must allow to have an homogenous product in the tank.
Test procedure	1. Take samples from the tank at 2 different locations 2. Analyse DM (Dry Matter) concentration
Acceptable (Test pass)	The samples shall not be statistically different.
Test output	Check table, analysis results

4.3. Filtration Unit tests procedures

The following tables describe the procedures for the specific Filtration Unit tests.

Table 23. Filtration Unit Procedure Test Case 1

Test Case	**1**
Specification/ Requirement	**Barrier for bacteria: production of a sterile filtrate**
Moment / Duration	The test will be performed during 4 weeks when all subunits are started.
Test objective	The test will allow to check that the FU can produce a sterile filtrate and that the cleaning and sterilization procedures are adapted to allow a sterile production of filtrate. Also the duration of filtrate production without contamination will allow to set the frequency of cleaning/ sterilization.
Test procedure	1. the sterilization procedure must be initially applied. 2. Samples of filtrate will be taken in sterile conditions: - at t = 0 (start-up of FU after sterilization procedure) - at t = 1 day - at t = 2 days - at t = 3 days - at t = 7 days - at t = 15 days - at t = t_{-1} + 7 days When a contamination is found, the sampling process will be stopped. 3. The filtrate samples will be plated on petri dishes and incubated: - for total aerobic count: on medium for total aerobic count (APHA) at 25 °C - for total anaerobic count: on Shaedler sheep blood agar at 44 °C 4. Once a contamination is found, a cleaning procedure followed by a sterilization procedure of the FU filtrate line will be applied. 5. Then new samples of filtrate will be taken in sterile conditions: - at t = 0 (start-up of FU after sterilization procedure) - at t = 1 day - at t = 2 days - at t = 3 days - at t = 7 days - at t = 15 days - at t = t_{-1} + 7 days When a contamination is found, the sampling process will be stopped.
Acceptable (Test pass)	- First phase without cleaning/ sterilization: a sterile filtrate shall be produced for at least the same period as obtained at prototype scale (3 days for anaerobic bacteria) - Second phase with cleaning/ sterilization: after applying the cleaning and sterilization procedure, a sterile filtrate shall be obtained.
Test output	Results of bacteria counts.

Table 24. Filtration Unit Procedure Test Case 2

Test Case	**2**
Specification/ Requirement	**Barrier for particles: minimize particular material in filtrate**
Moment / Duration	The test will be performed during 4 weeks when all subunits are started.
Test objective	The test must allow to check the efficiency of the membrane to retain the solid material inside the bioreactor
Test procedure	During the 4 weeks of test: 1. Take one sample of reactor content and one sample of filtrate 3 times per week 2. Analyse on each sample total and soluble COD (see procedure in Table 33) 3. Calculate the particular COD concentration in the reactor and in the filtrate for each sample: $COD_{Part} = COD_t - COD_s$ 4. The proportion of particular COD is calculated as: $\% = \frac{COD_{Part/Reactor}}{COD_{Part/Reactor} + COD_{Part/Filtrate}}$
Acceptable (Test pass)	The proportion of particular COD (COD_{Part}) retained in the bioreactor must be as high as possible and equal or higher than the one obtained during prototype tests (average > 99 %)
Test output	Graph showing the evolution of COD_{Part} in reactor and filtrate, and the proportion of particular COD retained in the reactor, in function of the time. The average proportion will also be calculated.

Table 25. Filtration Unit Procedure Test Case 3

Test Case	**3**
Specification/ Requirement	**Selective products recovery: maximize VFA and NH4 concentrations in the filtrate**
Moment / Duration	The test will be performed during 4 weeks when all subunits are started.
Test objective	The test must allow to check the efficiency of the membrane to recover the important degradation products in the filtrate.
Test procedure	During the 4 weeks of test: 1. Take one sample of reactor content and one sample of filtrate 3 times per week 2. Analyse on each sample VFA and NH4-N (see procedures in Table 29 and Table 32) 3. The proportion of products recuperation is calculated as: $\% = \frac{VFA/NH4_{Filtrate}}{VFA/NH4_{Retentate}}$
Acceptable (Test pass)	The proportion of recovery for VFA and NH4 shall be as high as possible and equal or higher than the ones obtained during prototype tests (91 % of recuperation of VFA, 91 % of recuperation of NH4).
Test output	Graph showing the evolution in the time of the proportion of recuperation of VFA and NH4. The average proportion will also be calculated.

4.4. Process tests procedures

The following section presents the procedures to be used for process follow-up during the Process Tests. The tests results will be compiled and processed in a general process database. The most significant results of this database will be presented in TN 71.10.2 under the form of graphs (parameters evolution in the time). These results will also be used to make further efficiency calculations

Table 26. Temperature

Parameter	**Temperature**
Sample	**Reactor content**
Moment / Frequency	The temperature has to be monitored once a day during all period the installation is operative.
Acceptable work range	The temperature in the reactor should be 55°C. The acceptable work range is between 54.5 – 55.5 °C for the optimization of acidogenesis and inhibition of pathogens.
Sample	**Influent and Filtrate**
Moment / Frequency	The temperature has to be monitored once a day during all period the FU is operative.
Acceptable work range	The temperature in the filtrate tank and the influent tank should be 4°C to prevent bacterial growth and quality degradation.
Test objective	The temperature has to remain constant and in the defined range, to ensure the optimal activity of the thermophilic bacteria and inhibit the possible pathogens.
Test procedure	- It is measured with an online temperature sensor (Pt100 sensors) and can be read directly on the PC interface (TS-R-O1). - The T variations will be analysed

Table 27. pH

Parameter	**PH**
Sample	**Influent**
Moment / Frequency	The pH of the influent is measured off-line with the consort C835 once per batch during all the period when the installation is operative.
Expected range	The expected work range is between 5 – 6,7.
Sample	**Reactor content**
Moment / Frequency	The online pH has to be monitored every day. Furthermore the pH will be checked 3 times a week with an offline pH meter in order to verify the similarity between the 2 measurements. This is performed during all period the installation is operative
Acceptable work range	In order to avoid metanogenisis the pH has to be lower than 6. The pH has to range between 5,1 and 5,6.
Sample	**Filtrate**
Moment / Frequency	The pH of the filtrate is measured off-line 3 times a week during all period the installation is operative.
Acceptable work range	The expected work range is between 5,1 and 5,6. It has to be more or less the same as in the bioreactor.
Test objective	The pH has to remain constant and in the defined range, to insure the optimal activity of the liquefying bacteria and inhibit the methanogenic ones.
Test procedure	***Online pH measurement*** It is measured with two online glass electrodes. The value can be directly read of the displays (pHT-R-O1 and pHT-R-O2) ***Offline pH measurement*** The pH measurement is performed with the consort C835. The pH is linked with the temperature of the sample. When measuring the pH the temperature sensor should be used at all time to correct the pH. During measurements the sample has to be stirred. Before and after the measurement the electrodes have to be rinsed with demineralized water. 1. switch on the consort C835 with the ON/OFF - switch and wait until the apparatus is started up (1 min). 2. Use the MODE - button to select the wanted measurement (pH). 3. Rinse the pH-probe and temperature sensor with demineralized water 4. Put the pH-probe together with the temperature sensor in the sample you wish to measure and wait until the value on the display is stable. (the dot on the display stops flickering) 5. After each measurement rinse the pH-probe and temperature sensor with demineralized water and when finished measuring put them back in the KCl-solution.

Table 28. Electroconductivity

Parameter	**Electroconductivity (EC)**
Sample	**Influent**
Moment / Frequency	It is measured once per batch with the consort C835 during all period the installation is operative.
Expected range	The EC in the influent is expected to be in the range 2 – 4 mS/cm
Sample	**Reactor content**
Moment / Frequency	It is measured 1 time a week with the consort C835 during all period the installation is operative.
Acceptable work range	The EC in the reactor is expected to be around 5 mS/cm and may not be higher than 18 mS/cm (see J.F. Malina, Jr.F.G. Pohland, Design of anaerobic processes for the treatment of industrial and municipal wastes, Water Quality Management Librairy, 1992).
Sample	**Filtrate**
Moment / Frequency	It is measured 3 times a week with the consort C835 during all period the installation is operative..
Acceptable work range	The EC in the filtrate is expected to be around 5 mS/cm and may not be higher than 18 mS/cm.
Test objective	The EC has to remain in the defined range. Above a limit of around 18 mS/cm, the electroconductivity indicates a salts concentration that corresponds to a high die-off of the bacteria.
Test procedure	The EC is linked with the temperature of the sample. When measuring the EC the temperature sensor should be used at all time to correct the EC. During measurements the sample has to be stirred. Before and after the measurement the electrodes have to be rinsed with demineralized water. 1. switch on the consort C835 with the ON/OFF - switch and wait until the apparatus is started up (1 min). 2. Use the MODE - button to select the wanted measurement (EC). 3. Put the EC-probe together with the temperature sensor in the sample you wish to measure and wait until the value on the display is stable. (the dot on the display stops flickering) 4. After each measurement rinse the EC-probe and temperature sensor with demineralized water and when finished measuring put them back in the demineralized-solution.

Table 29. VFA

Parameter	**VFA (Volatile Fatty Acids)**
Sample	**Influent**
Moment / Frequency	The VFA production will be measured once per influent batch.
Expected range	The expected range depends on the composition. There is a difference in range with (800 – 900 mg/L) or without (< 100 mg/L) the presence of faecal material.
Sample	**Reactor content**
Moment / Frequency	The VFA production in the reactor will be measured three times a week during all period the installation is operative.
Acceptable work range	The VFA concentration in the reactor should be higher than 3000mg/L and as high as possible without inhibiting the process
Sample	**Filtrate**
Moment / Frequency	The VFA production in the reactor will be measured three times a week during all period the installation is operative.
Acceptable work range	The amount of VFA in the filtrate should be about the same as in the reactor (see test FU section).
Test objective	The VFA production has to be as high as possible. In the filtrate there has to be a maximum recuperation of VFA from the reactor content ($VFA_{filtrate}/VFA_{reactor\ content}$ ~1). The VFA production is a direct indicator of the efficiency of the digestion process.
Test procedure	Preparation of the sample A standard is added to each batch for control. 2 ml of standard solution is brought in a plastic tube in stead of filtered sample and treat as the samples. Take 2 ml filtered sample in a plastic tube Add 0,5 ml ½ thinned sulphuric acid Add a pinch of Sodium chloride in each tube Add 0,4 ml internal standard Add 2 ml diethyleter Close the tubes with a cap an vortex the tubes during 2 minutes. Then centrifuge during 3 minutes by 3000 rpm After centrifugation the upper layer of ether is transferred in a glass receiver by pipette meant for the carousel of the GC. Close the glass recipient with a screw cap. Measurement of VFA concentration with the GC Measurement of volatile fatty acids in liquid solutions are performed using a Gas chromatograph type SHIMADZU-benelux Instrument GC 17 AA with FID and autosampler. The carrier gas used is Helium. The sample is detected by a temperature of 260°C and injected by a temperature of 250°C. The VFA is measured using special software. The procedure followed is described within an utility instruction**.**

Table 30. Dry matter and ashes

Parameter	**Dry Matter (DM) and Ashes**
Sample	**Influent**
Moment / Frequency	The amount of dry matter and ashes will be measured once per influent batch
Expected range	There is a difference in range with or without the presence of faecal material.
Sample	**Reactor content**
Moment / Frequency	The amount of dry matter and ashes will be measured three times a week in the reactor during all period the installation is operative.
Acceptable work range	After the installation of the filtration unit, the amount of dry matter in the reactor will be stabilized around 40 g/l to avoid the increase of dry matter.
Sample	**Filtrate**
Moment / Frequency	The amount of dry matter and ashes will be measured three times a week in the filtrate during all period the installation is operative
Acceptable work range	The dry matter should be constant in range and lower than the amount in the reactor.
Test objective	Making sure the amount of dry matter in the reactor stays in the region of 40 g/l (when using the FU); following the parameters, which was defined in the prototype tests as the optimal concentration. Also some information can be learned on the drain frequency and amount.
Test procedure	**Dry matter** Dry a crucible or beaker during 2 hours by 105°C till constant weight, cool in a dessicator and tare(=x1 g). In that you pipette V ml well mixed sample (usually 20 ml) and dry for minimum 12 hours till constant weight by 105 °C, cool down in a dessicator and then weight (=x2 g). measurement: $DM = \frac{x_2 - x_1}{V} \times 1000(g/L)$ **Ashes** Take the crucible where you measured the dry matter and put it in the oven for 2 hours at 600 °C. Then cool it down in a dessicator and weight it (=x3 g). measurement: $AS = \frac{x_3 - x_1}{V} \times 1000(g/L)$

Table 31. Total nitrogen

Parameter	**Total Nitrogen (Nt)**
Sample	**Influent**
Moment / Frequency	The amount of total nitrogen will be measured once per influent batch.
Expected range	The total nitrogen concentration can vary and is expected to remain between 300 and 600 mg/L.
Sample	**Reactor content**
Moment / Frequency	Total nitrogen of the reactor content is measured once a week during all period the installation is operative.
Expected range	The total nitrogen concentration can vary between 300 and 600 mg/L without use of FU (with FU it will be accumulated up to a maximum of 3 g/L)..
Sample	**Filtrate**
Moment / Frequency	The amount of total nitrogen will be measured 3 times a week during all period the installation is operative.
Expected range	The total nitrogen concentration will increase up to a maximum of 1.5 g/L
Test objective	To follow the concentration of total nitrogen and therefore the evolution and degradation of proteins
Test procedure	The analyses can be performed with the following test kits (Isis 6000 from Dr. Lange): LCK 338, LCK 238 en LCK 138 with a measurement range of respectively 20-100, 5-40 and 1-16 mg/L. 1. The sample has to be diluted to make sure the amount of nitrogen is within the measuring range of the test kit and to avoid interferences with p.e. chlorine. 2.To avoid interferences the sample has to be filtered 3. In a test tube an amount (dependant on the kit) of sample and caustic soda is pipetted. 4. A tablet of potassiumperoxodisulphat (oxidant) is added. 5. The test tube is put in the destructor during 1 hour by 100°C 6. After cooling down till room temperature one micro cap (lyophilisaat) is added. 7. Shake until the lyophilisaat is completely dissolved and is divided homogeneous. 8. Off this mixture 0.5 mL is pipetted in a cuvet, which contains 60% sulphuric acid and 33% phosphoric acid 9. Pippet 0.2 mL dimethylfenol solution in the cuvet 10. Wait 15 minutes and then measure the absorbance of the solution with the spectrophotometer, Isis 6000. Total nitrogen is measured with a wavelength of 360 nm. Together with every bath of samples a standard is measured. The result is displayed immediately in mg/l. Off course the performed dilutions have to be taken into account .

Table 32. Ammonium (NH4-N)

Parameter	**Ammonium nitrogen NH4-N**
Sample	**Influent**
Moment / Frequency	The amount of ammonium nitrogen will be measured once per influent batch.
Expected range	The expected range depends on the composition of the influent. There is a difference in range with or without the presence of faecal material (between 10 and 50 mg/L).
Sample	**Reactor content**
Moment / Frequency	Ammonium nitrogen of the reactor content is measured three times a week during all period the installation is operative
Expected range	The ammonium concentration in the reactor should be as high as possible (200 – 300 mg/L).
Sample	**Filtrate**
Moment / Frequency	Ammonium nitrogen of the reactor content and filtrate is measured tree times a week during all period the installation is operative.
Acceptable work range	The amount of NH4-N in the filtrate should be the same as the amount in the reactor.
Test objective	Making sure the NH4-N production in the reactor is as high as possible. The NH4 production is a direct indicator of the efficiency of the digestion process.
Test procedure	Ammonium-nitrogen is measured with Isis 6000 (Dr. Lange) The Test kit LCK 304 with a measuring range of 0.015 till 2 mg/L and LCK 303 with a measurement range of 2 till 47 mg/L can be used. The cuvets are already filled with the necessary reaction products. 1.The sample has to be diluted to make sure the amount of nitrogen is within the measuring range of the test kit and to avoid interferences with p.e. chlorine. 2.To avoid interferences the sample has to be filtered 3. 0.2 mL of the filtered sample is added to the cuvet. 4. immediately afterwards a dosicap with lyophilisaat is screwed on the cuvet 5. Shake the cuvet so the reaction products are well shaken with the sample 6. Wait 15 minutes and then measure the absorbance of the solution with the spectrophotometer, Isis 6000 with a wavelength of 695 nm. Together with every bath of samples a standard is measured. The result is displayed immediately in mg/l. Off course the performed dilutions have to be taken into account .

Table 33. COD total

Parameter	**Total Chemical Oxygen Demand (CODt)**
Sample	**Influent**
Moment / Frequency	The CODt will be measured once per influent batch.
Expected range	The expected range depends on the composition of the influent. There is a difference in range with or without the presence of faecal material (between 15 and 30 g/L).
Sample	**Reactor content**
Moment / Frequency	CODt of the reactor content is measured once every two weeks during all period the installation is operative.
Expected range	The CODt is expected to increase when using the FU up to a maximum of about 55 g/L.
Sample	**Filtrate**
Moment / Frequency	CODt of the filtrate is measured two times a week during all period the installation is operative.
Acceptable work range	The CODt of the filtrate should be about the same as the CODs, since the FU retains the solids.
Test objective	To follow CODt.
Test procedure	The total chemical oxygen demand is measured with Isis 6000 (Dr. Lange) he test kit LCK 514, with a measurement range of 100 till 2000 mg O_2/L is used. 1.The sample has to be diluted to make sure the amount of nitrogen is within the measering range of the test kit and to avoid interferences with p.e. chlorine 2. In a tube, already filled with mercurysulphat, 90% sulphuric acid potassiumdichromat, 2 ml sample is pippeted. Shake the mixture. 3. Then the tube is put into a destructor with a temperature of 150°C for two hours. 4. Cool the tube down till room temperature. Measure the absorbance of the yellow color with the spectrophotometer, Isis 6000. Together with every bath of samples a standard is measured. The result is displayed immediately in mg/l. Off course the performed dilutions have to be taken into account .

Table 34. COD soluble

Parameter	**Soluble Chemical Oxygen Demand (CODs)**
Sample	**Influent**
Moment / Frequency	The CODs will be measured once per influent batch.
Expected range	The expected range depends on the composition of the influent. There is a difference in range with or without the presence of faecal material (between 6 and 10 g/L).
Sample	**Reactor content**
Moment / Frequency	CODs of the reactor content is measured once every two weeks during all period the installation is operative.
Expected range	The CODs is expected to remain around 10 g/L
Sample	**Filtrate**
Moment / Frequency	CODs of the filtrate is measured two times a week during all period the installation is operative.
Acceptable work range	The CODs of the filtrate should be about the same as the CODs of the reactor content and the CODt of the filtrate, since the FU retains the solids.
Test objective	To follow CODs.
Test procedure	The soluble chemical oxygen demand is measured with Isis 6000 (Dr. Lange) The same method is used as for CODt, except the sample has to be filtered if CODs is determined.

Table 35. CHONSP

Parameter	**CHONSP**
Sample	**Influent**
Moment / Frequency	Two samples are taken on two different influent batches and the amount of CHONSP is measured by an independent lab when all the subunits of the compartment are working.
Sample	**Reactor content**
Moment / Frequency	Two samples are taken at one week of interval and the amount of CHONSP is measured by an independent lab when all the subunits of the compartment are working.
Sample	**Filtrate**
Moment / Frequency	Two samples are taken at one week of interval and the amount of CHONSP is measured by an independent lab when all the subunits of the compartment are working.
Test objective	To have preliminary information on the molecular composition of the compartment flows
Test procedure	The test is performed by an independent lab.

Table 36. Fibres

Parameter	**Fibres**
Sample	**Influent**
Moment / Frequency	Two samples are taken on two different influent batches and the amount of total fibres as well as cellulose, hemicellulose and lignin are measured by an external lab when all the subunits of the compartment are working, .
Sample	**Reactor content**
Moment / Frequency	Two samples are taken at one week of interval and the amount of total fibres as well as cellulose, hemicellulose and lignin is measured by an external lab when all the subunits of the compartment are working.
Sample	**Filtrate**
Moment / Frequency	Two samples are taken at one week of interval and the amount of total fibres as well as cellulose, hemicellulose and lignin is measured by an external lab when all the subunits of the compartment are working.
Test objective	To have preliminary information on the fibres composition of the compartment flows
Test procedure	The test is performed with the Van Soest method.

Table 37. Minerals

Parameter	**Minerals**
Sample	**Influent**
Moment / Frequency	Two samples are taken on two different influent batches and the amount of minerals ((Ag, As, Cd, Cr, Cu, Pb, Ni, Zn, Hg, Ca, Na) is measured by an independent lab when all the subunits of the compartment are working.
Sample	**Reactor content**

Moment / Frequency	Two samples are taken at one week of interval and the composition of minerals is measured by an independent lab when all the subunits of the compartment are working.
Sample	**Filtrate**
Moment / Frequency	Two samples are taken at one week of interval and the composition of minerals is measured by an independent lab when all the subunits of the compartment are working.
Test objective	To have preliminary information on the minerals composition of the compartment flows and the role of the FU regarding minerals.
Test procedure	The amount of minerals is determined by an independent lab. The determine the amount of minerals, the samples initially undergo a sour destruction with a microwave. This is followed by an analyses with ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy, an emission spectrophotometric technique, exploiting the fact that excited electrons emit energy at a given wavelength as they return to ground state.)

Table 38. Gas production

Parameter	**Gas production**
Sample	**Gas**
Moment / Frequency	The volume of gas in the 4 columns is measured once a day. When the GL is working, the flow of gas produced will also be measured and written once a day.
Test objective	To evaluate the gas production and therefore the digestion activity of the reactor
Test procedure	- Measure the gas volume produced in the columns - When the GL is working: check once a day the average flow of gas produced on the computer interface.

Table 39. Gas composition

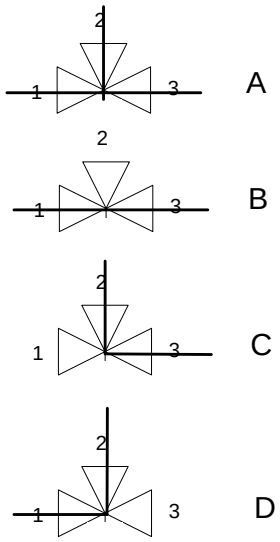
Parameter	**Gas composition**
Sample	**Gas**
Moment / Frequency	- 3 times a week with off-line analyser - once a day with on-line analyser when the GL is working
Expected work range	The CO_2 percentage should be as high as possible (around 80 % when the influent and effluent are taken continuously). The CH_4 concentration level is max 1 %. The O_2 concentration should be as low as possible and may not be higher than 1.5% (to not inhibit the anaerobic bacteria). Hydrogen Sulphide (H_2S) is a toxic and flammable gas, which is expected at traces level.
Test objective	To evaluate the gas composition and therefore the digestion activity of the reactor
Test procedure	**Offline analysis** The offline infrared gas analyser of Geotechnical instruments is used to measure the production of CO_2, O_2, H_2, CH_4. **Online analysis** The online gas analyses are performed with the online gas analyser of SICK-MAIHAK S710. The production of CO_2 and CH_4 is measured by an online analyser. (A-G-O2) The production of H_2S is measured by an online analyser. (A-G-O1)

5. CONCLUSIONS

The present test plan and test procedures document will allow to test the complete pilot Compartment I and verify that it fulfils the requirements as they were defined in previous phases of the project. In addition to these specific tests, the whole compartment will be evaluated through its continuous operation. Especially, a troubleshooting list will allow to carefully trace the encountered problems and the actions taken.

6. Addendum 1: Position of 3-way valves in FU

Legend:



Tag	Description	Article	Open	Gesloten
V-V-02	Manual 3-way valve	S57DBW, 1"	B	C
V-V-03	Powered 3-way valve	S57DBW, 1", AP3S en stuurventiel	D	C
V-V-04	Powered 3-way valve	S57DBW, 1", AP3S en stuurventiel	C	B
V-F-02	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-F-03	Powered 3-way valve	S57D-1/2"BW-L + AP2S + VTB950/230V + VTB755	D	C
V-F-04	Powered 3-way valve	S57D-1/2"BW-L + AP2S + VTB950/230V + VTB755	D	C
V-F-05	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-F-14	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-F-15	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-F-16	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-F-17	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-F-18	Powered 3-way valve	S57D-1/2"BW-L + AP2S + VTB950/230V + VTB755	D	C
V-C-14	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-C-15	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-C-16	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-C-17	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-C-18	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-C-19	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-S-02	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B
V-S-03	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-S-04	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	C	B
V-S-05	Powered 3-way valve	S57D-1/2"BW-T + AP2S + VTB950/230V + VTB755	D	B

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