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TECHNICAL NOTE 94.66

CI test protocol: validation of the filtration unit optimization

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CI test protocol: validation of the filtration unit optimization

I really don't know if we can keep this front page; if we do so, then even UAB is maybe not allowed to modify the document. We need to clarify this urgently.

After discussing, we understand that we can keep this page providing the front pages with the appropriate MELiSSA footer and header, and referencing there the present TM document

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Confidential

Filtration Unit Optimization

Technical Note 94.66

CI test protocol: validation of the filtration unit optimization

Distribution List

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Author	Issue	Date	Change
J.C. Lasserre L. Resses	1 Draft	02/04/2009	New Document
J.C. Lasserre L. Resses	1.1	26/01/2010	Updated with MPP comments
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MELISSA Pilot Plant

Technical Note 94.66

CI test protocol: validation of the filtration unit optimization

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1.Scope

This documents describes the tests protocols for the characterization and validation of the membrane filtration for the compartment CI.
Three kinds of tests are described:

- Water permeability test
- Membrane performing testing
- Limit flow research

Water permeability test allow to check the initial permeate water flow of the membrane and to compare it with that given by the manufacturer.

Membrane performing testing allow to validate the membrane performance with the biomass and filtration unit in the MPP laboratories. The results will be compared with those obtained during the filtration unit optimization study performed at TechnoMembranes (RD1).

Limit flow research allow to study the presence of a external reversible fouling or/and an internal fouling.

All the tests will be performed in the MPP facilities.

2.Reference and applicable documents

2.1. *Applicable documents*

AD1	19071/05/NL/CP	Memorandum of Understanding between MELiSSA Partners
AD2	OFR-ESA-03/07-UAB	MPP Proposal for Call Off Order 3 – C1 additional characterization,
AD3	TN94.5	CI Additional Characterisation Test Plan

2.2. *Reference documents*

RD1	TN94.42	Trade-off and selection of the best suited membranesRD1
RD2	TN94.43	Hardware procurement and upgrading activities
RD3	TN94.11	Integration of CI in the MELiSSA Pilot Plan
RD4	DP94.1	Compartment I Acceptance Review Control datapackage

RD5	MPP-PID-10-1001-B1	Compartment I PID
RD6	NTE-MCI-HB-012	CI HMI Software User manual

3.Acronyms and definitions

- PID: Piping and Instrumentation Diagram
- GN2: Gaseous Nitrogen
- COD: Chemical Oxygen Demand
- VFA: Volatile Fatty Acids

4.Test items

4.1. *Description (PID, technical drawings, user manual)*

- Filtration unit of compartment C1
- Document MPP : reference MPP-1000-A-001-A7
- Technical data sheet of KERASEP membranes.

4.2. *Hazards induced by test item and safety measures to be taken*

- Mechanical hazard (pump GP1201 01)
- Pressure hazard (compressed air and GN2 supplies : 6barg)
- Chemical hazards with cleaning agents (use of NaOH)

4.3. *Instructions for operation*

- Pump must be stopped
- Unit mustn’t be under pressure
- Pipes must be empty
- Check that gaskets (membrane and clamp) are not damaged
- Check that membrane doesn’t present signs of deterioration (cracks, fissures, notch on the tips, …)
- Check that membrane channel is not plugged and rinsed it with demineralized water if necessary

- During the demineralized water circulation, no leaks have to be observed on the clamps. If there are leaks, re-install the clamps or change joints.
- Install membrane into its module with care
- Install gaskets on the two tips of membrane
- Screw module tips straight and without forcing (not to break the membrane)
- Check alignment between tips and module
- Install module in the filtration unit skid : install gaskets and fix with clamps
- Do not force when mounting module on the filtration unit skid

4.4. *Instructions for maintenance*

- Check that gaskets (membrane and clamp) are not damaged
- Check that membrane doesn't present signs of deterioration (fissure, notch on the tips, …)
- Check that membrane channel is not plugged and rinsed it with demineralized water if necessary
- During water circulation, no leaks have to be observed on the clamps. If there are leaks, re-install the clamps or change gaskets.

5.Recall of test sequence

- Bring membrane out of its packing
- Install and fix it into the module
- Install module in the filtration unit
- For water permeability test: closed loop circulation of demineralized water in the membrane
- For membrane performance and limit flow tests, see the dedicated diagramme within the dedicated test procedure

6.Test protocol for Water Permeability Test

6.1. *Test procedure for Water Permeability Test*

- Set circulation flow at 200 L/h with demineralized water (tangential velocity of 2 m/s in the filtration loop)
- The temperature has to be around 25°C (± 5°C)
- Set the permeate flow at 6 L/h (±0,3 L/h) with the PP_1202_01 pump
- Wait until system stabilization (10 minutes)
- Record transmembrane pressure
- Realize a first measurement of permeate flow
- Wait 5 minutes
- Realize a second measurement of permeate flow

- Wait 5 minutes
- Realize a third measurement of permeate flow
- Calculate the average water permeability

6.2. *Features to be tested: functions, hardware, software*

- Mountability
- Leak tightness
- Water permeability

6.3. *Success/failure criteria*

- Axial alignment
- Absence of effort when mounting the module
- Absence of leaks
- The water permeability has to be superior to 1250 L/h.m².bar at 25°C.
- If the permeability is inferior to 1250 L/h.m².bar at 25°C the membrane has to be cleaned with a solution of NaOH at 10 g/L.

The calculation of the permeability is performed according with the following formula:

Jp (L/h.m².bar)= [Qp (L/h) / A (m2) / TMP (bar)] x Kt
where:

Qp: measured permeate flow
A: membrane surface (0,0113 m2)
TMP: Average retentate pressure (inlet+outlet/2) – permeate pressure
Kt: permeability coefficient (depending on temperature)

6.4. *Resources for the test*

6.4.1. *Personnel: staff qualification and training needs*

MPP Technician with TechnoMembranes personnel

6.4.2. *Personnel Protective Equipments*

- Safety shoes
- Laboratory coat
- Gloves and goggles

6.4.3. *Hardware: instruments, specific part, hardware for software operation, calibration certificates*

- Millwright work (screwdriver, pipe-wrench, …)
- No specific tools are needed
- Pressure sensors, permeate and retentate flowmeters, temperature sensor as already mounted on the skid
- A specific permeate flowmeter has to be mounted with the following measurement range : maxi 10 L/h, mini 0.1 L/h
- All sensors are calibrated with certificates

6.4.4. *Software: verification of software, backup needs*

All acquisitions have been validated
PLC is connected to the acquisition server

6.4.5. *Test conditions*

- Demineralised water quality: a conductivity value of less than 50 µS/cm of the demineralised water is required for the rinsing of the microfiltration membranes.
- Demineralised water supply: in order to perform the water permeability test directly on Compartment I skid, the retentate pipeline should be disconnected from the bioreactor inlet and outlet, and replaced by a demineralised water supply, using clean hoses to avoid any influence of contamination in the test results.
- Additional flowmeter and permeate pump maybe required to perform the tests, according to the specified flows.

6.5. *Measurement and data sampling*

6.5.1. *Data logfile*

Filtration unit test 1.date.dat

The acquired parameters are at least the following ones:

MPP Tag	*Description*
CP_1207_01	Pump
PP_1209_01	Peristaltic pump
CP_1207_02	Pump

PP_1202_01	Filtrate pump
GP_1201_01	Pump
TT_1200_01	Temperature sensor
TT_1200_02	Temperature sensor
TT_1200_03	Temperature sensor
FT_1201_01	Flow meter
AT_1201_01	Turbidity sensor
GP_1201_01	
PP_1202_01	
PT_1203_01	Pressure transducer
PT_1203_02	Pressure transducer
PT_1203_03	Pressure transducer
PT_1203_04	Pressure transducer
PT_1203_05	Pressure transducer
PT_1203_06	Pressure transducer
PT_1203_07	Pressure transducer
PT_1203_08	Pressure transducer
TT_1205_01	Temperature sensor
LSH_1206_01	Level switch
LSH_1206_02	Level switch
LT_1206_01	Level sensor
PI_1207_01	Pressure indicator
PI_1207_02	Pressure indicator
TT_1208_01	Temperature sensor : REPLACED BY FT_1202_01
LSH_1209_01	Level switch
LSL_1209_01	Level switch
LSH_1209_02	Level switch

LSL_1209_02	Level switch
FT_1202_01	Additional flowmeter on permeate

6.5.2. *Special requirements if any (frequency, duration, synchronization)*

Every minute for all instrumentation.

6.6. *Reporting of status for a test*

The test sequence is performed by MPP personnel, under the expertise and advice of TechnoMembranes personnel.

The final status of the test (passed/fail) is decided at the end of the test in agreement between TechnoMembranes expert and MPP management.

6.7. *Deviations and non conformances*

In case the test sequence cannot be performed as planned or some results are out of their expected range, a deviation is opened and appended to the test record. The process to fill out the deviation form is identical to the one to fill out the NCR as per the Quality Assurance Procedure for the control of non conformities MPP-QAP-07-0002.

This deviation is discussed between TechnoMembranes and MPP in order to decide how to address it. If necessary, on the basis of a given deviation, MPP can decide to open a NCR as planned by the Quality Manual and the Quality Assurance Procedure for the control of non conformities MPP-QAP-07-0002.

The discussion of all deviations is made before the final decision of the status for the test.

6.8. *Record for the test procedure with the various steps*

MELiSSA	**MELiSSA Pilot Plant**			UAB Universitat Autònoma de Barcelona
TEST RECORD SHEET		**Type**	**Chrono**	**Page :**
		MPP-REC	**0 -**	/

Compartment : C1 Test Phase : 1

Test title : Membrane Permeability Test

Objectives:

Applicable test plan and test protocols

Hardware:

Person responsible for the test :

Test prerequisites :

tep No.	Action description	Expected results / Nominal behaviour	Date / Hour	Observed results / calculated / remarks – ref. of Deviation	C/NC	Initials
1	**Membrane mounting**	**a.Good alignment between tips and module** **b.No forcing for mounting**				
2	**Demineralized water circulation**	**Circulation flow in FT_1201_01 : 200 L/h** **No leaks**				
3	**Correct acquisition of specified test parameters**	**Pressures** **Temperatures** **Flows** **Chemical sensors**				
4	**Stabilisation**	**10 minutes** **Stable pressures and flows**				

MELiSSA	**MELiSSA Pilot Plant**	UAB Universitat Autònoma de Barcelona

TEST RECORD SHEET	**Type**	**Chrono**	**Page :**
	MPP-REC	**0 -**	/

Compartment : C1 Test Phase : 1

5	**Permeate flow setting**	**FT_1202_01 : 6 L/h - 10 minutes**				
6	**Stabilisation**	**Stable pressures and flows**				
7	**Permeate flow measurement**	**FT_1202_01 reading**				
8	**Waiting**	**5 minutes**				
9	**Permeate flow measurement**	**FT_1202_01 reading**				
10	**Waiting**	**5 minutes**				
11	**Permeate flow measurement**	**FT_1202_01 reading**				
13	**Calculation of average water permeability**	**>1250 L/h.m².bar at 25°C**				

Conclusion for the Test	*Name*	*Signature*	*Date*
☐ Passed ☐ Failed			
- **Number of deviations attached to the document :** - **All deviations have been justified or corrected ? YES / NO**			
Comments			
Checked by **TechnoMembranes** **MELiSSA Pilot Plant**	*Name*	*Signature*	*Date*

Appendix 1 - record of implied personnel

Name	ORGANIZATION	Function	Initials

Appendix 2 - record of calibration certificates for the test instruments

Instrument description	Inv. Number	Calibration record reference	Date of calibration	Calibration valid until	Signature

Appendix 3 - deviations list

DEV. FORM #	Deviation:		Criticality Low Medium High	
	Corrective action:		Resp.	Due date
	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date	

DEV. FORM #	Deviation:		Criticality Low Medium High	
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	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date	

DEV. FORM #	Deviation:		Criticality Low Medium High	
	Corrective action:		Resp.	Due date

	Corrective action performed and checked: **Ref. of retests:**	**Checked / approved by**	**Closing Date**

7.Test protocol for Stage 1: Membrane Performance testing

7.1. *Test procedure for Membrane performance testing*

1- The Kerasep membrane (60 cm, 0.1 µm) is installed into its module connected to the filtration unit skid and a demineralized water permeability test has to be done (see part II.1).
If the permeability is inferior to 1250 L/h.m².bar at 25°C (according to NOVASEP technical specifications, see TN94.43 for further details) the membrane has to be cleaned with a solution of NaOH at 10 g/L and the permeability has to be measured again until it is above 1250 L/h.m².bar at 25°C.

2- As the biomass is between 30 and 40 g/L of dry matter, the biological suspension characteristics are analysed (soluble COD, VFA, Suspended solid, Volatil Suspended Solid), pH, temperature (55°C).
If the characteristics measured (biological suspension characteristics, pH, temperature) are similar to those of the suspension studied in Montpellier, the Kerasep membrane (60 cm, 0.1 µm) will be installed into the filtration loop.

3- The circulation without filtration (biomass has to pass inside the channel of the membrane, permeate valve has to be closed) is set at 200 L/h (tangential velocity of 2 m/s in the filtration loop) at least 12h before filtration starts to mitigate all the floc structure changes linked to shearings (due to putting into circulation which generally involves at the beginning an increase of the clogging soluble COD and a change of floc size).

4- After the first 12h filtration starts for 12h with a constant permeate flow equal to 30 L/h.m², that is to say 0.33 L/h.
The following are measured and recorded:
- Inlet module pressure;
- Longitudinal loss of pressure (difference between inlet and outlet module pressure);
- Evolution of transmembrane pressure during filtration time (dP/dT).

5- If the inlet module pressure is superior to 0.6 bar for a circulation velocity of 2 m/s, it is necessary to make sure that the membrane channel is not obstructed and the suspension does not contain too many big particles which could obstruct the circulation channel. In the opposite case see following step.

6- If the increase in transmembrane pressure during these 12h is superior to 0.3 bar, the circulation velocity has to be increased by 1 m/s and filtration with a permeate

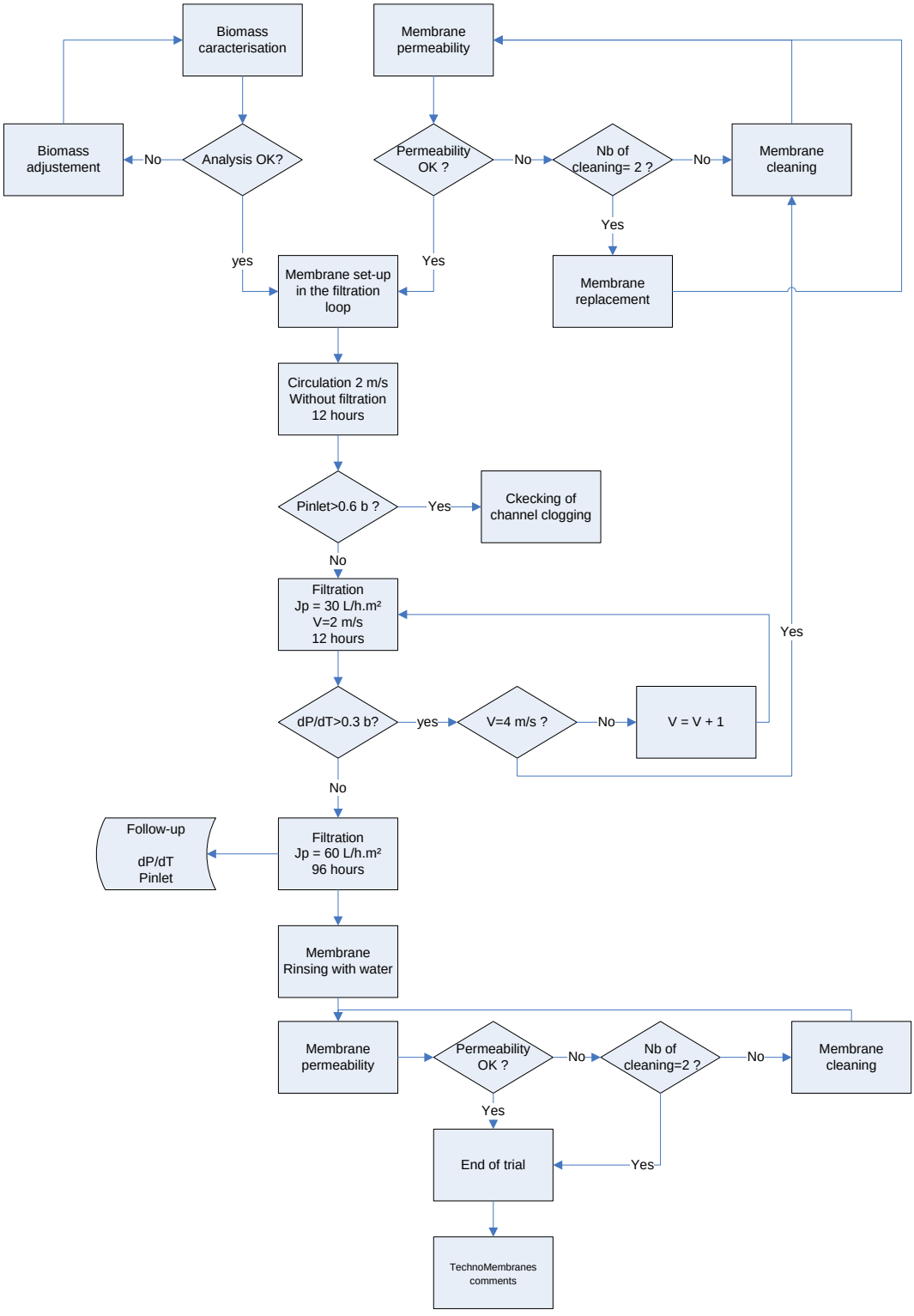
flow of 30 L/h.m² will continue during these 12h. If the transmembrane pressure does not decrease after 2h, the circulation velocity is increased until 4 m/s, maximum recommended value. Without filtration improvement, the membrane has to be cleaned with chemicals. Having regained a permeability value close to that of the new membrane (±10%), a new test can be done with a circulation velocity directly fixed at 4 m/s for 12h. If the transmembrane pressure does not evolve and remains low the circulation velocity is gradually decreased to 2 m/s. If the system remains stable it is possible to set following conditions.

7- If the transmembrane pressure evolution remains low at 30 L/h.m² filtration velocity is gradually increased to reach 60 L/h.m² (optimal condition observed by TechnoMembranes). This operation is maintained for 96 hours with a constant permeate flow equal to 60 L/h.m².
The following are measured and recorded:

- Inlet module pressure;
- Longitudinal loss of pressure (difference between inlet and outlet module pressure);
- Evolution of transmembrane pressure during filtration time (dP/dT).

8- At the end of the filtration period the membrane is rinsed with demineralized water and its permeability is controlled (see demineralized water permeability test protocol).

9- Data are recorded during these tests and sent to TechnoMembranes for comment. The values will be compared with the results obtained during this first campaign at TechnoMembranes.



7.2. *Features to be tested: functions, hardware, software*

Validation of membrane filtration performance
Validation of cleaning sequence

7.3. *Success/failure criteria*

No plugging has to be observed.
Flux equal to 60L/h.m^2 with an inlet pressure < 0.6 bar for a circulation velocity of 2m/s.
Stability of transmembrane pressure for the fixed permeate flow.

7.4. *Resources for the test*

7.4.1. *Personnel: staff qualification and training needs*

MPP Technician with TechnoMembranes personnel

7.4.2. *Personnel Protective Equipments*

- Safety shoes
- Laboratory coat
- Gloves and goggles

7.4.3. *Hardware: instruments, specific part, hardware for software operation, calibration certificates*

- Millwright work (screwdriver, pipe-wrench, …)
- No specific tool are needed
- Pressure sensors, permeate and retentate flowmeters, temperature sensor as already mounted on the skid
- A specific permeate flowmeter has to be mounted: 0.33 – 0,66 L/h
- Additional permeate pump, compatible with the range 0,33-0,66 L/h
- All sensors are calibrated with certificates

7.4.4. *Software: verification of software, backup needs*

- All acquisition have been validated
- PLC is connected to the acquisition server

7.4.5. *Test conditions*

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- On one side, as indicated in Section 7.1, some conditions of the culture broth are required for the performance of the test: biomass between 30 and 40 g/L of dry matter, and broth characteristics (soluble COD, VFA, Suspended solid, Volatil Suspended Solid), pH, temperature (55°C) similar to those of the

suspension studied in the trade-off and selection campaign performed in Technomembranes facility.

- On the other side, additional flowmeter and permeate pump maybe required to perform the tests, according to the specified flows, as indicated in Section 7.4.3.

7.5. *Measurement and data sampling*

7.5.1. *Data logfile*

Filtration unit test2.date.dat

The acquired parameters are at least the following ones:

MPP Tag	*Description*
CP_1207_01	Pump
PP_1209_01	Peristaltic pump
CP_1207_02	Pump
PP_1202_01	Filtrate pump
GP_1201_01	Pump
TT_1200_01	Temperature sensor
TT_1200_02	Temperature sensor
TT_1200_03	Temperature sensor
FT_1201_01	Flow meter
AT_1201_01	Turbidity sensor
GP_1201_01	
PP_1202_01	
PT_1203_01	Pressure transducer
PT_1203_02	Pressure transducer
PT_1203_03	Pressure transducer
PT_1203_04	Pressure transducer

PT_1203_05	Pressure transducer
PT_1203_06	Pressure transducer
PT_1203_07	Pressure transducer
PT_1203_08	Pressure transducer
TT_1205_01	Temperature sensor
LSH_1206_01	Level switch
LSH_1206_02	Level switch
LT_1206_01	Level sensor
PI_1207_01	Pressure indicator
PI_1207_02	Pressure indicator
TT_1208_01	Temperature sensor : REPLACED BY FT_1202_01
LSH_1209_01	Level switch
LSL_1209_01	Level switch
LSH_1209_02	Level switch
LSL_1209_02	Level switch
FT_1202_01	Additional flowmeter on permeate

7.5.2. *Special requirements if any (frequency, duration, synchronization)*

Every minute for all instrumentation

7.6. *Reporting of status for a test*

The test sequence is performed by MPP personnel, under the expertise and advice of TechnoMembranes personnel.
The final status of the test (passed/fail) is decided at the end of the test in agreement between TechnoMembranes expert and MPP management.

7.7. *Deviations and non conformances*

In case the test sequence cannot be performed as planned or some results are out of their expected range, a deviation is opened and appended to the test record. The process to fill

out the deviation form is identical to the one to fill out the NCR as per the Quality Assurance Procedure for the control of non conformities MPP-QAP-07-0002.
This deviation is discussed between TechnoMembranes and MPP in order to decide how to address it. If necessary, on the basis of a given deviation, MPP can decide to open a NCR as planned by the Quality Manual and the Quality Assurance Procedure for the control of non conformities MPP-QAP-07-0002.
The discussion of all deviations is made before the final decision of the status for the test.

7.8. *Record for the test procedure with the various steps*

MELiSSA	MELiSSA Pilot Plant	UAB Universitat Autònoma de Barcelona

TEST RECORD SHEET	Type	Chrono	Page :
	MPP-REC	0 -	/

Compartment : C1 Test Phase : 2

Test title : Membrane Perfomance test

Objectives:

Applicable test plan and test protocols

Hardware:

Person responsible for the test :

Test prerequisites :
Permeability test was passed

tep No.	Action description	Expected results / Nominal behaviour	Date / Hour	Observed results / calculated / remarks – ref. of Deviation	C/NC	Initials
1	**Demineralised water permeability**	**>1250 L/h.m².bar at 25°C**				
2	**Analysis of biological suspension**	**Similar characteristics to those of the suspension studied in Montpellier** **Dry Matter : 30g/L to 40g/L**				
3	**Setting of circulation without filtration**	**200 L/h – 12h**				

MELiSSA Pilot Plant						UAB Universitat Autònoma de Barcelona
TEST RECORD SHEET			Type	Chrono	Page :	
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Compartment : C1 Test Phase : 2						
4	Filtration starting for 12h	30 L/h.m² (0.33 L/h)				
5	If the increase in TMP>0.3 bar and test duration<12h, increase circulation velocity by 1 m/s and filtration at 30 L/h.m² will continue	Decrease of TMP				
6	If no decrease of TMP, increase velocity until 4 m/s	Filtration improvement				
7	If permeability value close to that of the new membrane, new filtration test with circulation velocity at 4 m/s	No evolution of TMP TMP remains low				
8	If the TMP does not evolve, gradually decrease circulation velocity to 2 m/s	System stable				
9	If TMP evolution remains low at 30 L/h.m², gradually increase filtration velocity to 60 L/h.m² (0.66 L/h)	Duration of filtration at 60 L/h.m²: 96h				
10	Rinsing with demineralized water	No plug				

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TEST RECORD SHEET	Type	Chrono	Page :
	MPP-REC	0 -	/

Compartment : C1 Test Phase : 2

11	Demineralised water permeability	>1250 L/h.m².bar at 25°C				

Conclusion for the Test	*Name*	*Signature*	*Date*
☐ Passed ☐ Failed			
1- Number of deviations attached to the document : 2- All deviations have been justified or corrected ? YES / NO			
Comments			
Checked by TechnoMembranes MELiSSA Pilot Plant	*Name*	*Signature*	*Date*

Appendix 1 - record of implied personnel

Name	ORGANIZATION	Function	Initials

Appendix 2 - record of calibration certificates for the test instruments

Instrument description	Inv. Number	Calibration record reference	Date of calibration	Calibration valid until	Signature

Appendix 3 - deviations list

DEV. FORM #	Deviation:		Criticality Low Medium High
	Corrective action:		Resp. / Due date
	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date

DEV. FORM #	Deviation:		Criticality Low Medium High
	Corrective action:		Resp. / Due date
	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date

DEV. FORM #	Deviation:	Criticality Low Medium High
	Corrective action:	Resp. / Due date

	Corrective action performed and checked: **Ref. of retests:**	**Checked / approved by**	**Closing Date**

8.Test protocol for Limit Flow Research Test

8.1. *Test procedure for Limit Flow Research*

To determine the outer limits of filtration from an intensification perspective, the tests in several increasing stages will be conducted according to the following procedure.

1- The Kerasep membrane (60 cm, 0.1 µm) is installed into its module connected to the filtration unit skid and a demineralized water permeability test has to be done (see part II.1).
If the permeability is inferior to 1250 L/h.m².bar at 25°C the membrane has to be cleaned with a solution of NaOH at 10 g/L and the permeability has to be measured again until it is above 1250 L/h.m².bar at 25°C.

2- As the biomass is between 30 and 40 g/L of dry matter, the biological suspension characteristics are analysed (soluble COD, VFA, Suspended solid, Volatile Suspended Solid), pH, temperature (55°C).
If the characteristics measured (biological suspension characteristics, pH, temperature) are similar to those of the suspension studied in Montpellier, the Kerasep membrane (60 cm, 0.1 µm) will be installed into the filtration loop.

3- The circulation without filtration (biomass has to pass through the membrane) is set at 200 L/h (tangential velocity of 2 m/s in the filtration loop) at least 12h before filtration starts to mitigate all the floc structure changes linked to shearings (due to putting into circulation which generally involves at the beginning an increase of the clogging soluble COD and a change of floc size).

4- Filtration starts for 30 minutes with a constant permeate flow equal to 30 L/h.m² (0.33 L/h).
The following are measured and recorded:
- Inlet module pressure;
- Longitudinal loss of pressure (difference between inlet and outlet module pressure);
- Evolution of transmembrane pressure during filtration time (dP/dT).

5- After these first 30 minutes filtration is continued for 30 minutes with a constant flow equal to 60 L/h.m² (0.66 L/h).
The following are measured and recorded:
- Inlet module pressure;
- Longitudinal loss of pressure (difference between inlet and outlet module pressure);
- Evolution of transmembrane pressure during filtration time (dP/dT).

6- Filtration is then carried on for 30 minutes with a lower permeate flow equal to the reference value of 30 L/h.m² (0.33 L/h) (for which no clogging must be observed).

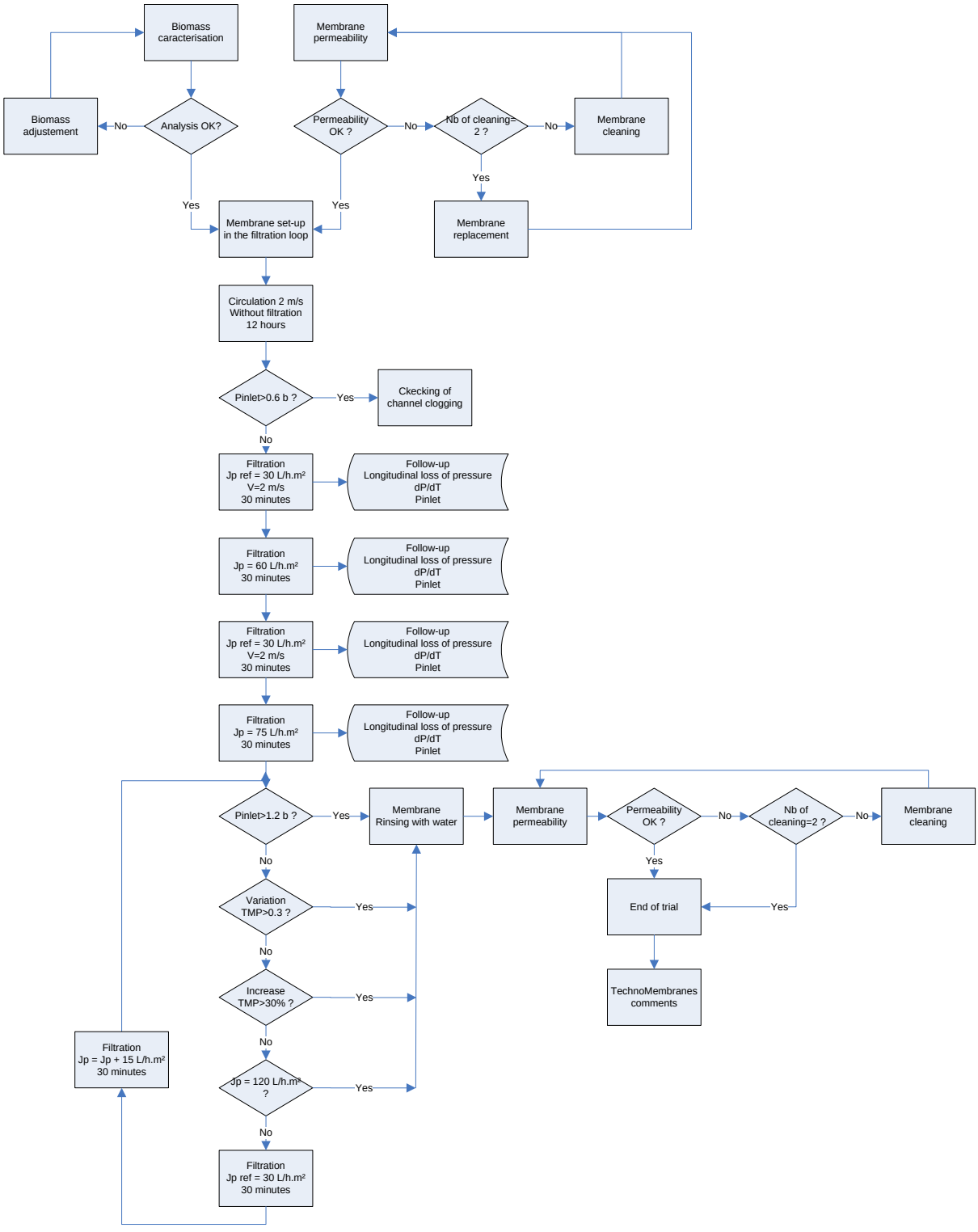
This lapse of time can permit a hydraulic regeneration of the membrane by removing a possible surface deposit.
The following are measured and recorded:
- Inlet module pressure;
- Longitudinal loss of pressure (difference between inlet and outlet module pressure);
- Evolution of transmembrane pressure during filtration time (dP/dT).

7- Then filtration is restarted for 30 minutes with a constant permeate flow equal to the permeation flow from step 3 with an increment of 15 L/h.m², that is to say 75 L/h.m² (0.83 L/h).
The following are measured and recorded:
- Inlet module pressure;
- Longitudinal pressure drop(difference between inlet and outlet module pressure);
- Evolution of transmembrane pressure during filtration time (dP/dT).

8- Steps 4 and 5 are then reproduced until a permeate flow equal to 120 L.h.m² (1.32 L/h) is obtained (that is to say 90 L/h.m² (0.99 L/h), 105 L/h.m² (1.16 L/h)), unless one of the following conditions achieved:
- Inlet pressure of module superior to 1,2 bar;
- Variation of transmembrane pressure measured during a filtration stage is superior to 0,3 bar;
- Increase of transmembrane pressure, measured after 10 minutes, between two filtration stages at 30 L/h.m² is superior of 30%.

If one of these conditions is achieved the test is stopped and steps 8 and 9 from stage 1 are performed.

All the results are communicated to TechnoMembranes to be interpreted.



8.2. *Features to be tested: functions, hardware, software*

Validation of the outer limits of filtration from an intensification perspective.

8.3. *Success/failure criteria*

No plugging has to be observed.
Flux equal to 60, 75, 90, 105 and 120 L/h.m^2 with an inlet pressure < 1.2 bar for a circulation velocity of 2m/s.
Stability of transmembrane pressure for a permeate flow fixed.

8.4. *Resources for the test*

8.4.1. *Personnel: staff qualification and training needs*

MPP Technician with TechnoMembranes personnel

8.4.2. *Personnel Protective Equipments*

- Safety shoes
- Laboratory coat
- Gloves and goggles

8.4.3. *Hardware: instruments, specific part, hardware for software operation, calibration certificates*

- Millwright work (screwdriver, pipe-wrench, …)
- No specific tool are needed
- Pressure sensors, permeate and retentate flowmeters, temperature sensor as already mounted on the skid
- A specific permeate flowmeter has to be mounted: 0.33 – 1.32 L/h
- Additional permeate pump, compatible with the range 0,33-1,32 L/h
- All sensors are calibrated with certificates

8.4.4. *Software: verification of software, backup needs*

- All acquisition have been validated
- PLC is connected to the acquisition server

8.4.5. *Test conditions*

- On one side, as indicated in Section 8.1, some conditions of the culture broth are required for the performance of the test: biomass between 30 and 40 g/L of dry matter, and broth characteristics (soluble COD, VFA, Suspended solid, Volatil Suspended Solid), pH, temperature (55°C) similar to those of the suspension studied in the trade-off and selection campaign performed in Technomembranes facility.

- On the other side, additional flowmeter and permeate pump maybe required to perform the tests, according to the specified flows, as indicated in Section 8.4.3.

8.5. *Measurement and data sampling*

8.5.1. *Data logfile*

Filtration unit test3.date.dat

The acquired parameters are at least the following ones:

MPP Tag	*Description*
CP_1207_01	Pump
PP_1209_01	Peristaltic pump
CP_1207_02	Pump
PP_1202_01	Filtrate pump
GP_1201_01	Pump
TT_1200_01	Temperature sensor
TT_1200_02	Temperature sensor
TT_1200_03	Temperature sensor
FT_1201_01	Flow meter
AT_1201_01	Turbidity sensor
GP_1201_01	
PP_1202_01	
PT_1203_01	Pressure transducer

PT_1203_02	Pressure transducer
PT_1203_03	Pressure transducer
PT_1203_04	Pressure transducer
PT_1203_05	Pressure transducer
PT_1203_06	Pressure transducer
PT_1203_07	Pressure transducer
PT_1203_08	Pressure transducer
TT_1205_01	Temperature sensor
LSH_1206_01	Level switch
LSH_1206_02	Level switch
LT_1206_01	Level sensor
PI_1207_01	Pressure indicator
PI_1207_02	Pressure indicator
TT_1208_01	Temperature sensor : REPLACED BY FT_1202_01
LSH_1209_01	Level switch
LSL_1209_01	Level switch
LSH_1209_02	Level switch
LSL_1209_02	Level switch
FT_1202_01	Additional flowmeter on permeate

8.5.2. *Special requirements if any (frequency, duration, synchronization)*

Every minute for all instrumentation

8.6. *Reporting of status for a test*

The test sequence is performed by MPP personnel, under the expertise and advice of TechnoMembranes personnel.
The final status of the test (passed/fail) is decided at the end of the test in agreement between TechnoMembranes expert and MPP management.

8.7. *Deviations and non conformances*

In case the test sequence cannot be performed as planned or some results are out of their expected range, a deviation is opened and appended to the test record. The process to fill out the deviation form is identical to the one to fill out the NCR as per the Quality Assurance Procedure for the control of non conformities MPP-QAP-07-0002.
This deviation is discussed between TechnoMembranes and MPP in order to decide how to address it. If necessary, on the basis of a given deviation, MPP can decide to open a NCR as planned by the Quality Manual and the Quality Assurance Procedure for the control of non conformities MPP-QAP-07-0002.
The discussion of all deviations is made before the final decision of the status for the test.

8.8. *Record for the test procedure with the various steps*

MELiSSA	MELiSSA Pilot Plant	UAB Universitat Autònoma de Barcelona

TEST RECORD SHEET	Type	Chrono	Page :
	MPP-REC	0 -	/

Compartment : C1 Test Phase : 3

Test title : Limit flow research

Objectives:

Applicable test plan and test protocols

Hardware:

Person responsible for the test :

Test prerequisites

tep No.	Action description	Expected results / Nominal behaviour	Date / Hour	Observed results / calculated / remarks – ref. of Deviation	C/NC	Initials
1	**Demineralized water permeability**	**>1250 L/h.m².bar at 25°C**				
2	**Analyse of biological suspension**	**Similar characteristics to those of the suspension studied in Montpellier**				
3	**Setting of circulation without filtration**	**2 m/s – 12h**				
4	**Filtration starting**	**0.33 L/h (30 L/h.m²)** **30 minutes**				

MELiSSA Pilot Plant			UAB Universitat Autònoma de Barcelona
TEST RECORD SHEET	Type	Chrono	Page :
	MPP-REC	0 -	/

Compartment : C1 Test Phase : 3

5	Filtration	0.66 L/h (60 L/h.m²) 30 minutes				
6	Filtration	0.33 L/h (30 L/h.m²) 30 minutes				
7	Filtration	0.83 L/h (75 L/h.m²) 30 minutes				
8	Checking of thresholds defined in 3.3.5.8	Pinlet<1.2 bar Variation TMP<0.3 bar Increase TMP<30 %				
9	Filtration	0.33 L/h (30 L/h.m²) 30 minutes				
10	Filtration	0.99 L/h (90 L/h.m²) 30 minutes				
11	Checking of thresholds defined in 3.3.5.8	Pinlet<1.2 bar Variation TMP<0.3 bar Increase TMP<30 %				
12	Filtration	0.33 L/h (30 L/h.m²) 30 minutes				

MELiSSA	MELiSSA Pilot Plant		UAB Universitat Autònoma de Barcelona
TEST RECORD SHEET	Type	Chrono	Page :
	MPP-REC	0 -	/

Compartment : C1 Test Phase : 3

13	Filtration	1.16 L/h (105 L/h.m²) 30 minutes				
14	Checking of thresholds defined in 3.3.5.8	Pinlet<1.2 bar Variation TMP<0.3 bar Increase TMP<30 %				
15	Filtration	0.33 L/h (30 L/h.m²) 30 minutes				
16	Filtration	1.32 L/h (120 L/h.m²) 30 minutes				
17	Rinsing with demineralized water	No plug				
18	Demineralized water permeability	>1250 L/h.m².bar at 25°C				

Conclusion for the Test	*Name*	*Signature*	*Date*
☐ Passed ☐ Failed			
3- Number of deviations attached to the document : **4- All deviations have been justified or corrected ? YES / NO**			
Comments			
Checked by **TechnoMembranes** **MELiSSA Pilot Plant**	*Name*	*Signature*	*Date*

Appendix 1 – record of implied personnel

Name	ORGANIZATION	Function	Initials

Appendix 2 – record of calibration certificates for the test instruments

Instrument description	Inv. Number	Calibration record reference	Date of calibration	Calibration valid until	Signature

Appendix 3 - deviations list

DEV. FORM #	Deviation:		Criticality Low Medium High	
	Corrective action:		Resp.	Due date
	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date	

DEV. FORM #	Deviation:		Criticality Low Medium High	
	Corrective action:		Resp.	Due date
	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date	

DEV. FORM #	Deviation:		Criticality Low Medium High	
	Corrective action:		Resp.	Due date

	Corrective action performed and checked: Ref. of retests:	Checked / approved by	Closing Date

SECTION 2 :

Comments

CI test protocol : validation of the filtration unit optimization

General comments

Please insert the MELiSSA Header and footer on each page
MELiSSA Header and footer on all the pages

We miss some information, according to the MPP template for Test protocol, and as well the overall logic of the test plan, which should have been provided as input to TN 94.5.
I would like to see this document following the template you proposed to us.
The content of the document has been splitted in two parts: what is relevant to the Test Plan has been removed to be included in TN96.5. The rest has been reorganized and completed according to the MPP Test Protocol template within this TN.

Detailed comments

Page/paragraph	Comment
1/Title of the TN	« Input for Technical Note 94.66 » Please update. OK, updated : «TECHNICAL NOTE 94.66 »
1/Title of the TN	« Test Plan » According to COO3, TN 94.66 is called CI Test Protocol : validation of the filtration unit optimization. It should not be called 'test plan' as the test plan is foreseen in TN 94.5, and TM should provide its inputs to this TN as well OK, title amended : « CI test protocol : validation of the filtration unit optimization »
5/ TM document front page	I really don't know if we can keep this front page; if we do so, then even UAB is maybe not allowed to modify the document. We need to clarify this urgently.

	After discussing, we understand that we can keep this page providing the front pages with the appropriate MELiSSA footer and header, and referencing there the present TM document.
9/Text box under index of TM document	« The data outlined in this document is strictly confidential. The technical and economical figures presented in this document are intended for the exclusive information of the customer.» We need to consult a contract officer on this matter. Because of this mention, UAB is maybe even not allowed to send the info to ESA. We have kept it, waiting for your feedback, but maintaining in the front pages the MELiSSA footer and header
11/Section 4.1	« Technical data sheet » Which one ? Phrase completed : « Technical data sheet of KERASEP membranes »
11/Section 4.2	« GN2 » Meaning ? Gaseous Nitrogen ; to be included in the acronyms
12/Section 4.3	« Check that membrane channel is not plugged and rinsed it with demineralized water if necessary» It would be good to precise a conductivity applicable for all tests OK, defined in the Test conditions
12/Section 5	Recall of test sequence : According to the MPP template, this paragraph should have been placed before entering the detail of each test, recalling the overall logic of test sequence. Correct, amended accordingly (one general sequence is provided as there is a detailed specific protocol for each of the tests in Section 6); maybe it would be better to split the three test sequences.
12/Section 5	« For membrane performance and limit flow tests, see the dedicated diagramme within the dedicated test procedure

	According to the MPP template, this paragraph should have been placed before entering the detail of each test, recalling the overall logic of test sequence ». Not understood why this sequence is mentioned here, as membrane performance testing should be performed only after water permeability? Correct, maintained only for the general test sequence, not in each phase
12/Section 6.1	Test procedure for Water Permeability Test: Isn't it actually the recall of the test plan or some inputs to TN 94.5 ? To be clarified The procedure is transferred to the Test Plan but it is maintained as well in the Test protocol -as it is quite detailed-, however moved to the section 6 of each protocol
13/Section 6.1	« Set the permeate flow at 6 L/h » +/- ? +/-5% is considered adequate, and it is compatible with the pump specifications; precised in the text : « 6 L/h (±0,3 L/h) »
13/Section 6.3	« The water permeability has to be superior to 1250 L/h.m^2.bar at 25ºC » +/- ? +/-10%(to be checked with TM). In fact, the range is 1250-2000, so it should be higher than 1250.
24/Section 7.1, bullet1	« If the permeability is inferior to 1250 L/h.m².bar at 25°C … » Why this value : according to the supplier technical datasheet, recommendations ? please precise, or include a reference; can you indicate some acceptable deviation? Reference to TN94.43 included in the text: in page 13 (Hardware definition) of this TN the technical specifications of the membrane are provided
24/Section 7.1, bullet 6	« If the increase in transmembrane pressure during these 12h is superior to 0.3 bar, the circulation velocity has to be increased by 1 m/s and filtration with a permeate flow of 30 L/h.m² will continue

	during these 12h. » To be rephrased apparently some part of the sentence is missing No, we think it is correct.
24/Section 7.1, bullet 6	"Having regained a permeability value close to that of the new membrane ..." Can you provide a range ? +/- 10%would be OK; precised in the text
24/Section 7.1, bullet 7	« If the transmembrane pressure evolution remains low at 30 L/h.m² filtration velocity is gradually increased to 60 L/h.m² … » Do you mean 'to reach 60 L/h.m2 ? Yes, precised : « …increased to reach 60 L/h.m² … »
27/Section 7.4.4	« All acquisition have been validated » Do you mean must / have to be validated ? It means that the data acquisition system (PLC+SCADA) has been already validated in MPP for CI
27/Section 7.4.5	Test conditions : This paragraph is a copy/paste of page 3 and 4, I don't see the point of this. OK, included only on Section 6 of the document
37/Section 8.1	Test procedure for Limit Flow Research: Isn't it actually the recall of the test plan or some inputs to TN 94.5 ? To be clarified The procedure is transferred to the Test Plan but it is maintained as well in the Test protocol -as it is quite detailed-, however moved to the section 6 of each protocol
37/Section 8.1, bullet 1	"If the permeability is inferior to 1250 L/h.m².bar ..." +/- ? +/-10% (to be checked with TM). In fact, the range is 1250-2750, so we are at the minimum

38/Section 8.1, bullet 8	"If one of these conditions is achieved the test is stopped and the two last steps from stage 1 are performed." i.e step 8 and step 9? Yes, correct; text amended accordingly.
40/Section 8.4.4	« All acquisition have been validated » See previously Answered in comment on Section 7.4.4
41/Section 8.4.5	Test conditions : See previously Answered in comment on Section 7.4.5