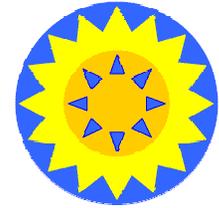


# How to run membrane experiments successfully

Dr Ir F.Petrus Cuperus

Pilot plant experiments v2



**SolSep BV**

Robust Separation Technologies

## How to run membrane experiments successfully

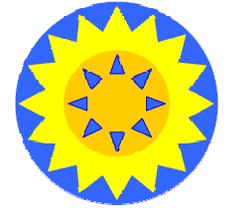
Dr Ir F. Petrus Cuperus

# Disclaimer



## **Disclaimer:**

**SolSep BV is not liable to any direct nor indirect damage that is claimed to be due to the use of materials or instruction like this manual.. All your work is done at your own risk! The experimenter is advised to follow and comply to all safety instructions that apply to the specific site.**



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## 1. Steps in a successful demo or pilot

### 1. Define objectives and understand the fluid

**Objectives** can be:

- screening for flux and selectivity

- fouling study

- scale-up data collection

- concentration

- effect of feed stock-changes on performance

- membrane life

**Fluid**

- Components to be separated

- Pre-treatment needs

- Viscosity (change)

- Particles/colloids

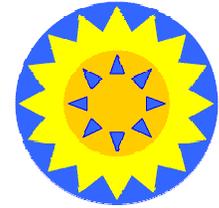
- Compositions

- Moleculare structures, polarity, etc.



## 2. Materials needed

1. Data sheet
2. Flow meter
3. Stop watch
4. Thermometer
5. Safety equipment
6. Samples bottle and labels
7. Cleaning chemicals/clean solvent
8. Storage vessel
9. Graph paper-computer
10. Viscosity info (vs temperature)



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### 3 - 4 Measurements - Fix “benchmark”

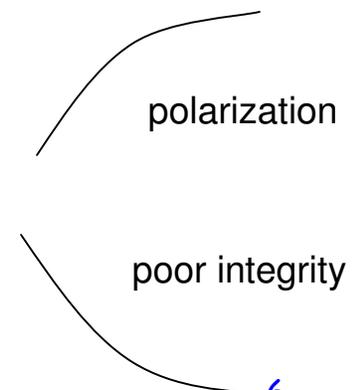
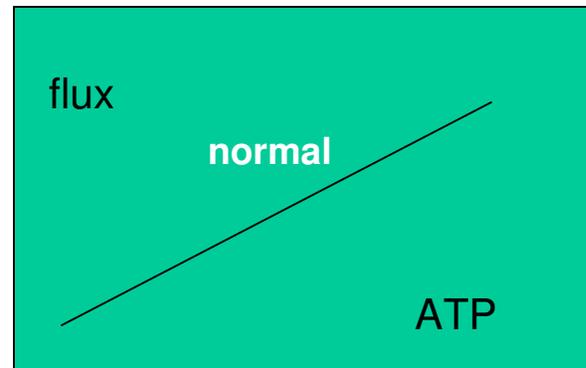
3. Flush systems with pure solvent, measure or estimate hold up volume

(note: in some membranes preservatives should be rinsed out )

4. Record solvent flow vs ATP (average trans membrane pressure)

4.1 @ some 4 pressures. (record temperature!)

4.2 construc plot





## 4 Measurements - Fix “benchmark”

“normal” straight behavior could be influenced by:

Wetting (not thru origin)

Swelling

Compaction

Temperature correction: (approximation)

$$\text{Flux T2} = \text{Flux T1} \times (\text{visc T1} / \text{visc T2})$$

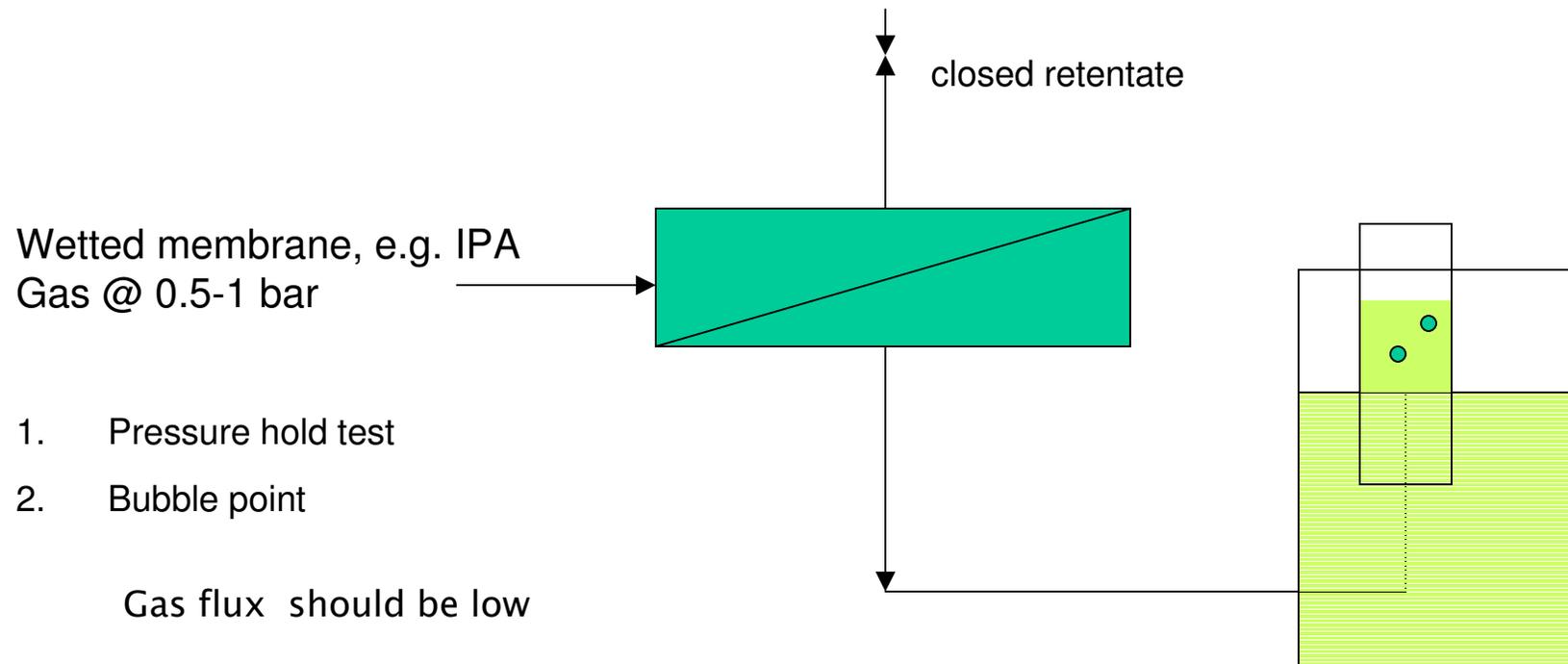
Clean solvent flux is reference to see whether

“something” happens to the membrane

(e.g. fouling, compaction, rupture)

## 5. Integrity test

If integrity is questionable: perform integrity test





## 6 “Real” measurements

Pre-condition membrane if necessary  
(swelling important for organic solvents)

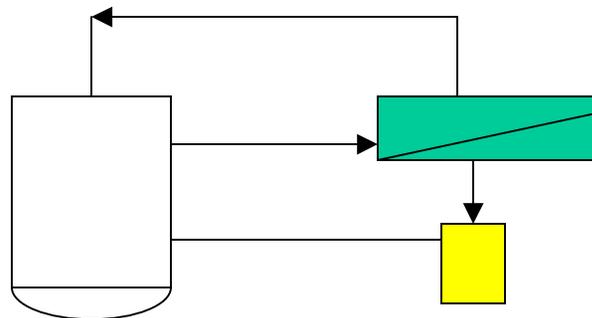
Start up process solvent:

at total recycling mode ( $P \sim 0$  bar)

Increase  $P$  slowly

Record  $T$

Save sample of  $t=0$  (starting material)

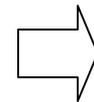


Total recycling:  
Permeate is fed back  
to feed vessel



## 7. Variables in real measurements

1. Record and calculate flux (not only V & time data!)
2. Determine concentration of target molecules (ASAP)
3. Measure filtrate flow in time (like #3) to see fouling conc. polarization (CP)
4. Try to see whether you can operate under stable flux operation (stable can also be gradually decrease =CP, fouling)  
(NB1 record T!)  
(NB2 use log – normal plots cause fouling normally will occur!)
5. Work @ different pressure and flow conditions: use an order like:
  - reduce velocity- measure @ 2-3 ATPs
  - increase velocity: repeat



**and compare !**

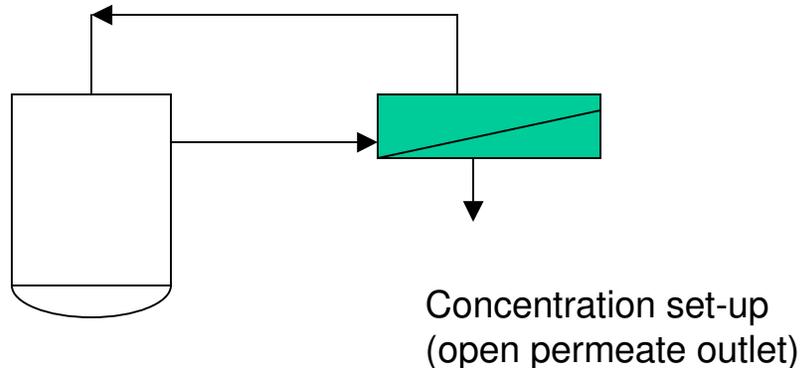
[www.SolSep.com](http://www.SolSep.com) return to original: repeat



## 8. Concentration measurements

1. Select best set of  $v$  & ATP, start batch concentration
2. Record flow and temp at  $Y$ (=recovery/conc factor) range  
adjust for volume removed
3. Return to total recycle (#6) at higher  $y$  and repeat measurements  
always return to original flow and ATP
4. Collect final cumulative samples in feed and permeate!

Always calculate fluxes during experiments: it is easier to see changes then!





## 9. Finalization

Flush system and if necessary  
perform cleaning with minimum permeate rate

Re-measure clean solvent flux and compare to initial data

If you want to use the same sample:

Store membrane wet

Repeat experiments – long(er) term experiments

Draw conclusions

Consider further experiments

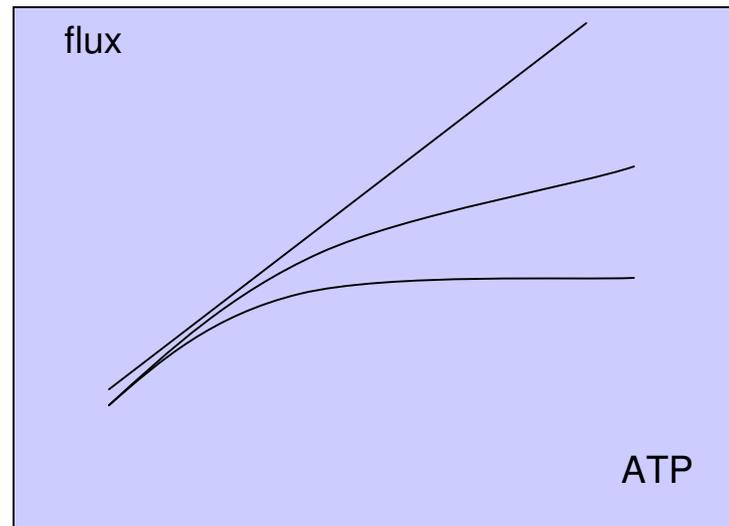


## Remarks on phenomena 1

In organic solvents swelling can be very extensive and prolonged  
Exposure could be necessary to yield stable operation.  
You might consider pre-swelling the material.

Cleaning can be often done by using clean solvent

Permeation rate

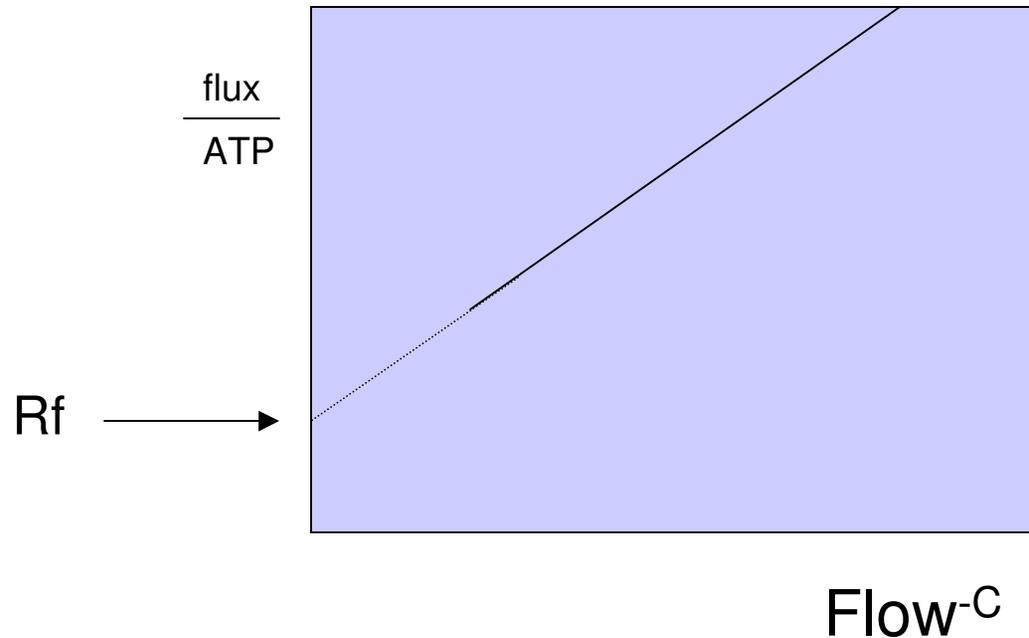


Linear-not polarized

Slighter polarization

Polarized-NB irreversible  
fouling could occur

## Remarks on phenomena 2



$$\text{Flux} = k * \text{flow}^C$$
$$C < 1 \text{ (theory 0.33)}$$

See how Rf increases at consecutive filtration

=

Fouling

=

See how it decreases after cleaning

## Finding the optimum



**With flat sheets this optimum is hardly only to be estimated!!**

1. Pressure
  1. Until plateau – stay out of plateau
  2. No plateau
    - pump limit (+ energy costs)
    - module limit (max P)
- Flow
  - If small pump limit-membrane limit
  - If big: cost=limit (area vs energy)

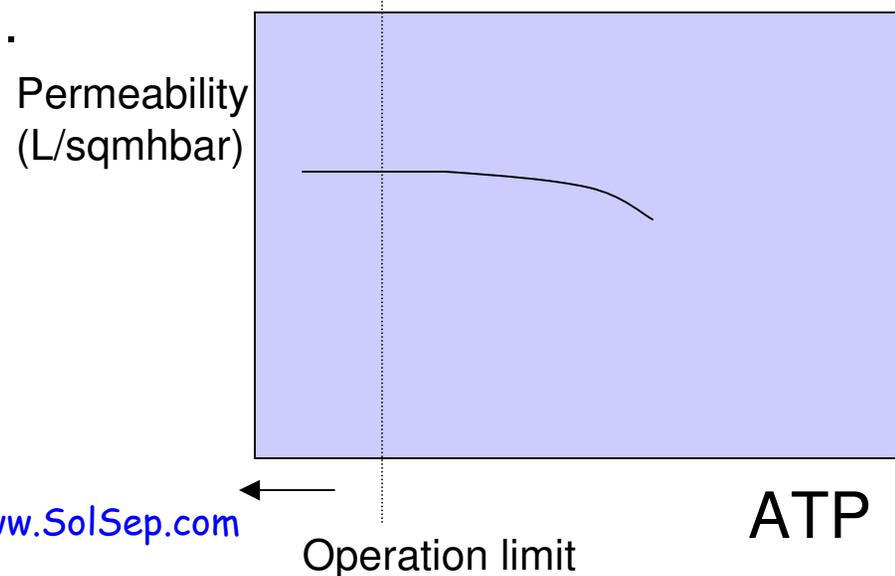
## Finding the optimum – limited flux operation



Theory:

Performance under limiting flux takes that at beneath certain flux – pressure – CP and fouling does NOT occur

In practice: one operates under (very) low pressures if the membrane is cheap (MF/UF, some RO).



Area mostly large(r): membrane costs vs cleaning/operation costs. For SRNF cleaning costs are mostly relatively low, and membrane costs drives processes to higher pressures



**We highly recommend you to stay in touch with SolSep BV on the performance of your membranes and the application development work you are planning.**

**SolSep BV is well-experienced in the application of membranes in non-aqueous environments.**

**We have seen a lot of solvents...!**