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Water reuse and waste water minimization

Reverse osmosis process on rinsing waters after the phosphating process for water and raw material recuperation

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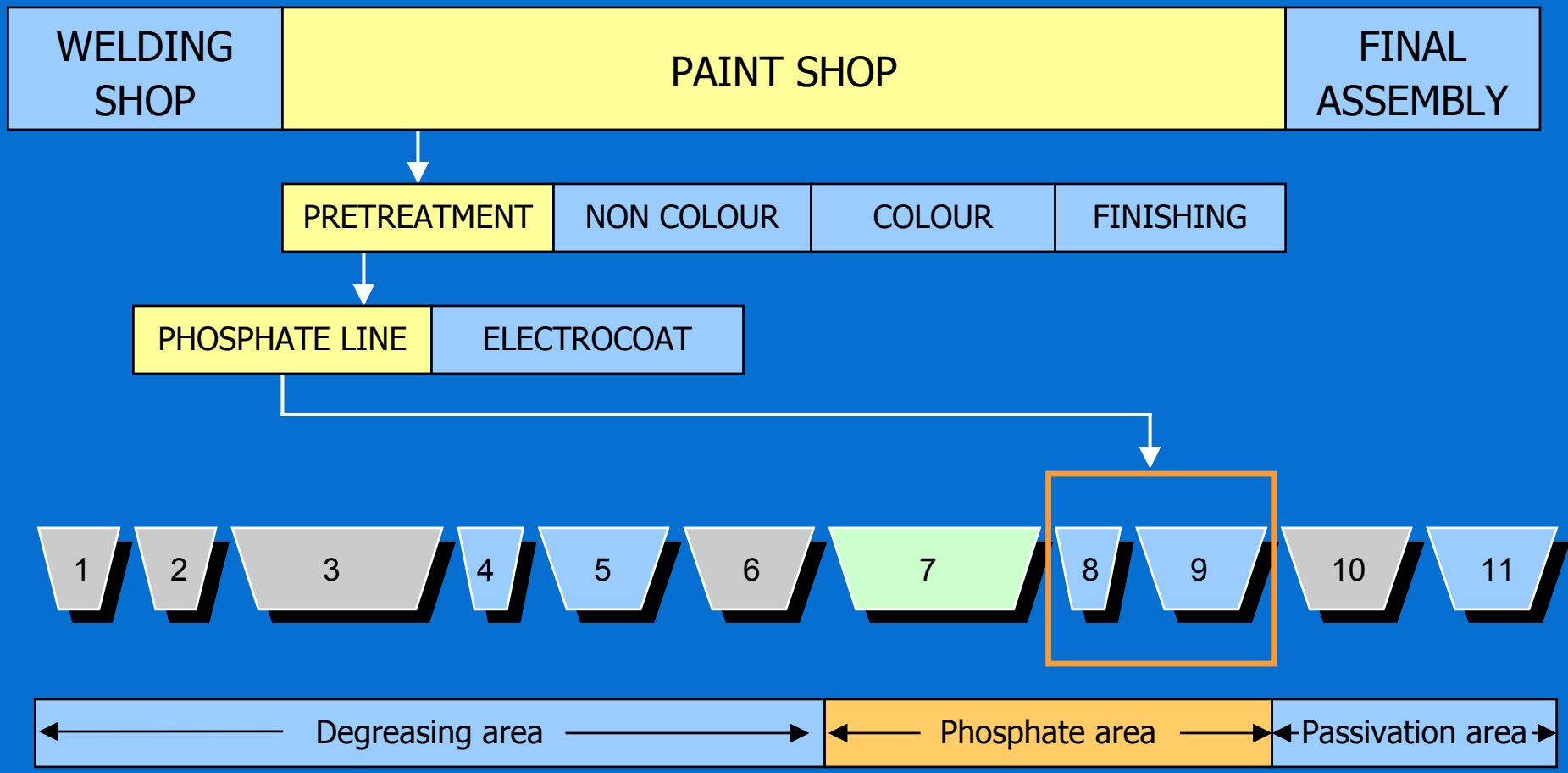
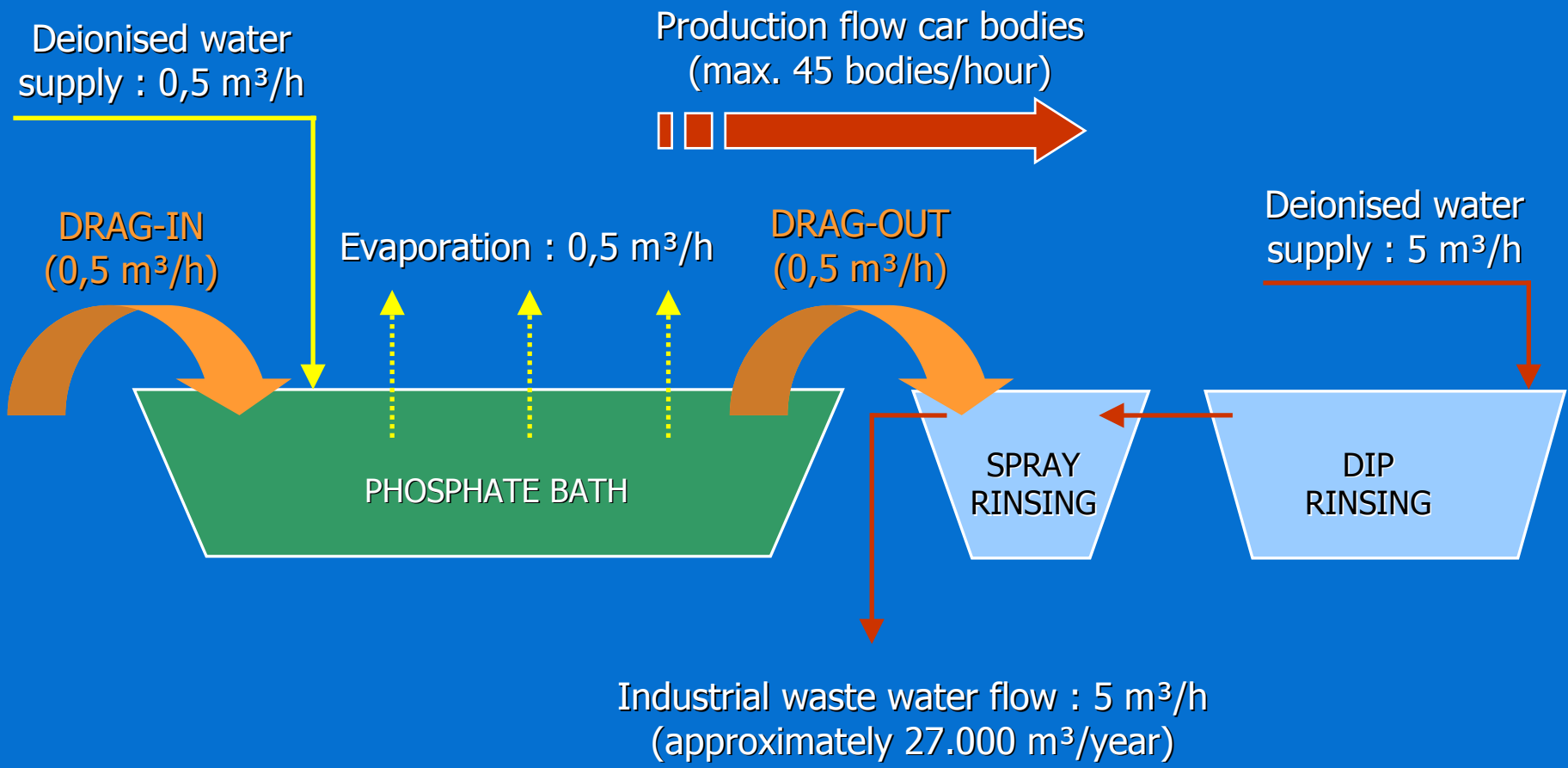
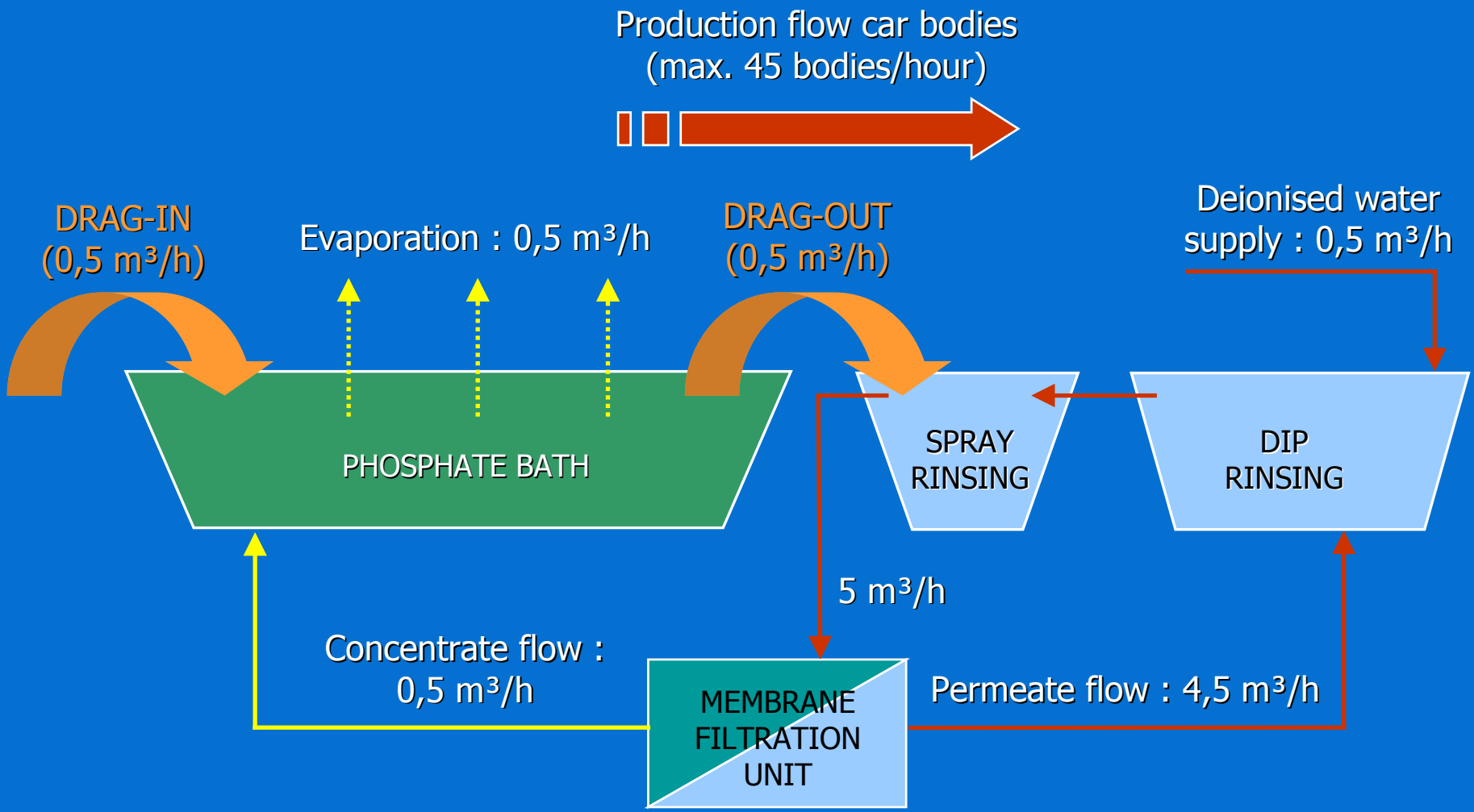
Production area of the RO project



Original phosphating process VCG



Principle closed loop phosphating

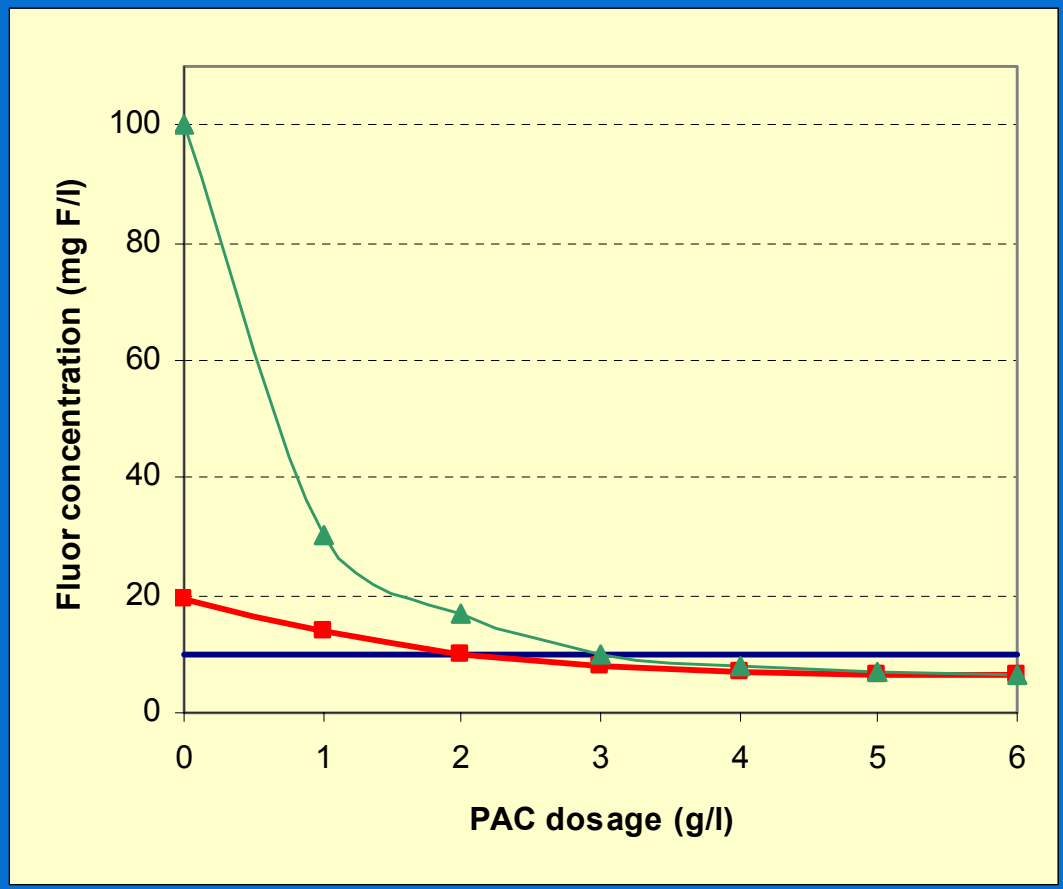


Project motivation (1)

- ➢ Introduction aluminium
  - ✓ Addition of free fluoride in phosphate bath
    - ↳ increased sludge production
    - ↳ higher fluor concentration

Significant increase of the fluor load to the waste water which makes fluor removal in the physico-chemical waste water treatment plant necessary !

Project motivation (2)



- **No aluminium**
  - ✓ 5 m³/h
  - ✓ 10 mg F/l
  - ✓ No PAC dosage
- **Alu without RO**
  - ✓ 5 m³/h
  - ✓ 20 mg F/l
  - ✓ PAC dosage 2 g/l (= 240 kg/d)
- **Alu with RO**
  - ✓ 1 m³/h
  - ✓ 100 mg F/l
  - ✓ PAC dosage 3 g/l (= 72 kg/d)

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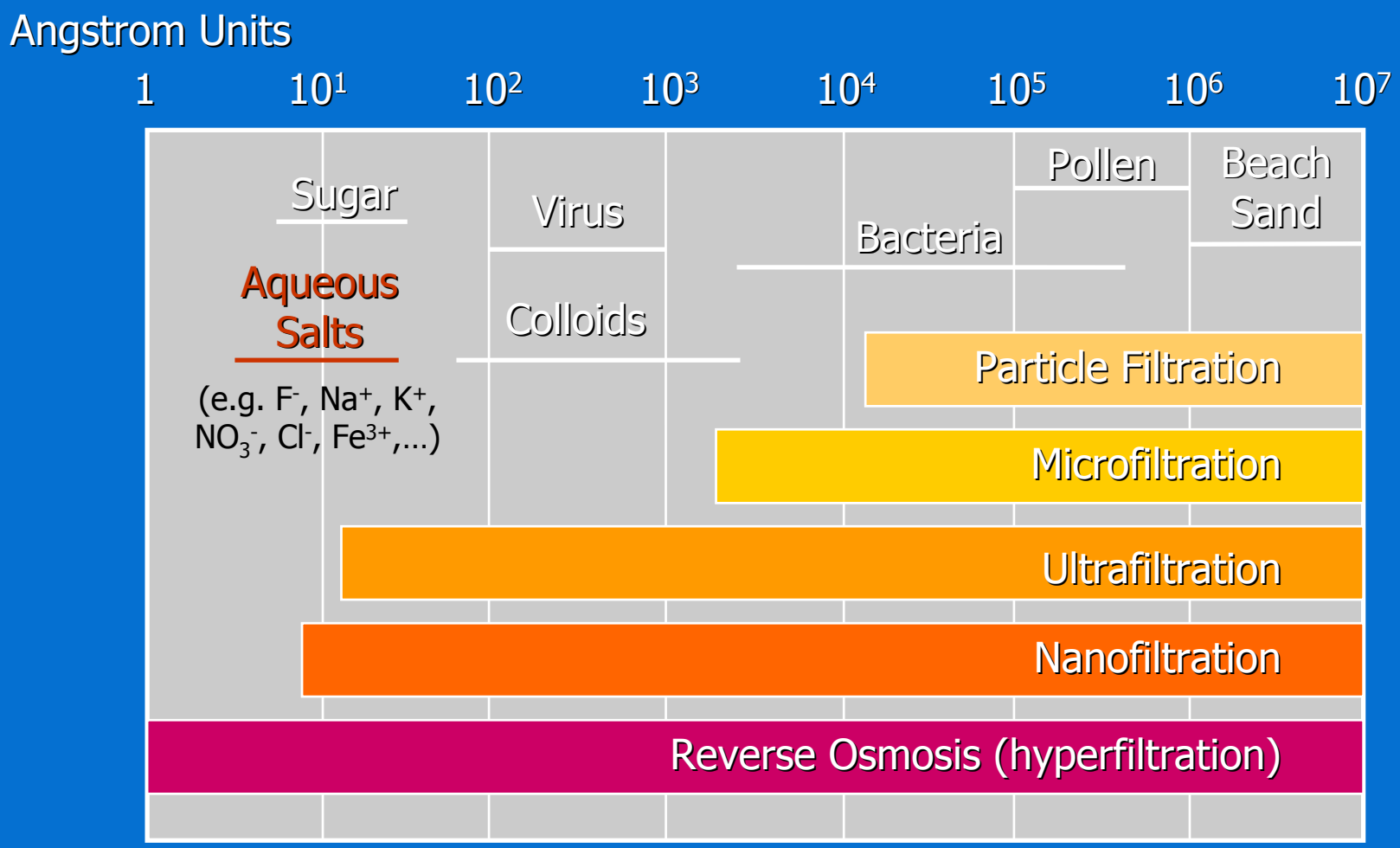
Advantages of closed loop phosphating

- Reduction of the water consumption
- Reduction of the amount of industrial waste water
- Lower running cost in the waste water treatment
  - ✓ less waste water to treat
  - ✓ higher fluor concentration, resulting in a higher efficiëncy of the PAC dosage for fluor removal
- Reduction of the emission of heavy metals (Zn, Ni, Mn,…) and nutrients (N, P) to the environment
- More capacity free in the waste water treatment for future projects (e.g. increased production capacity)

The Filtration Spectrum



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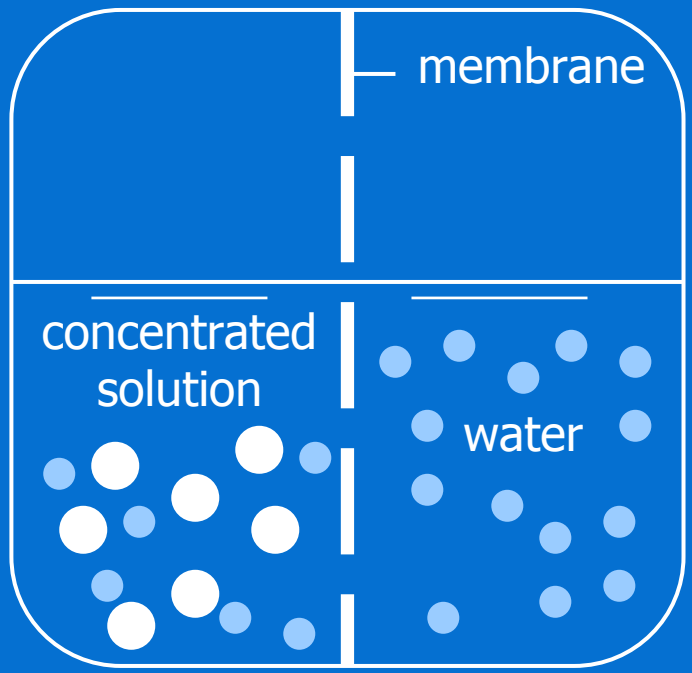
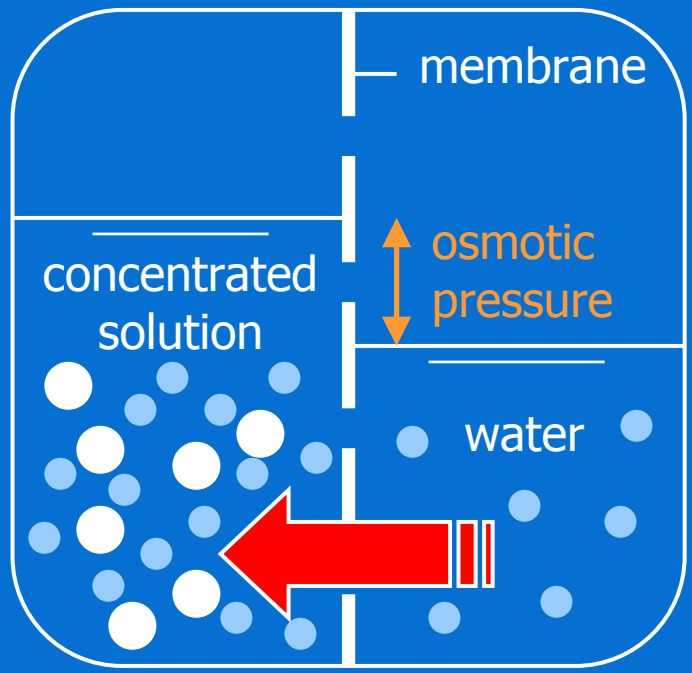
Principle of osmosis

Start situation



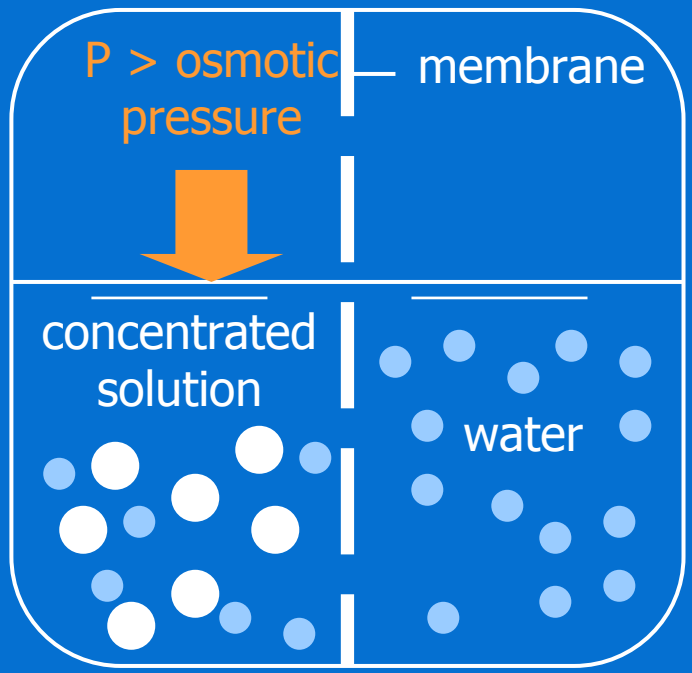
Equilibrium



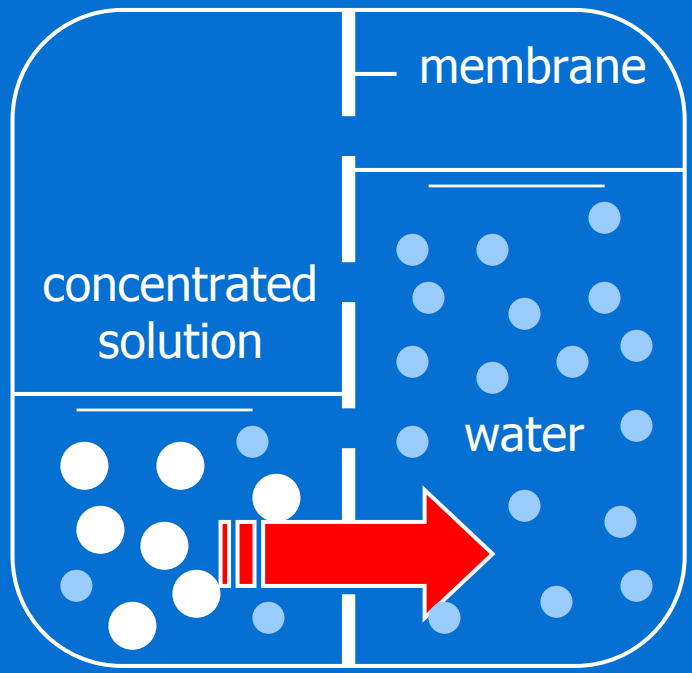
Water is passing through the membrane from the water compartment to the concentrated solution compartment

Principle of reverse osmosis

Start situation

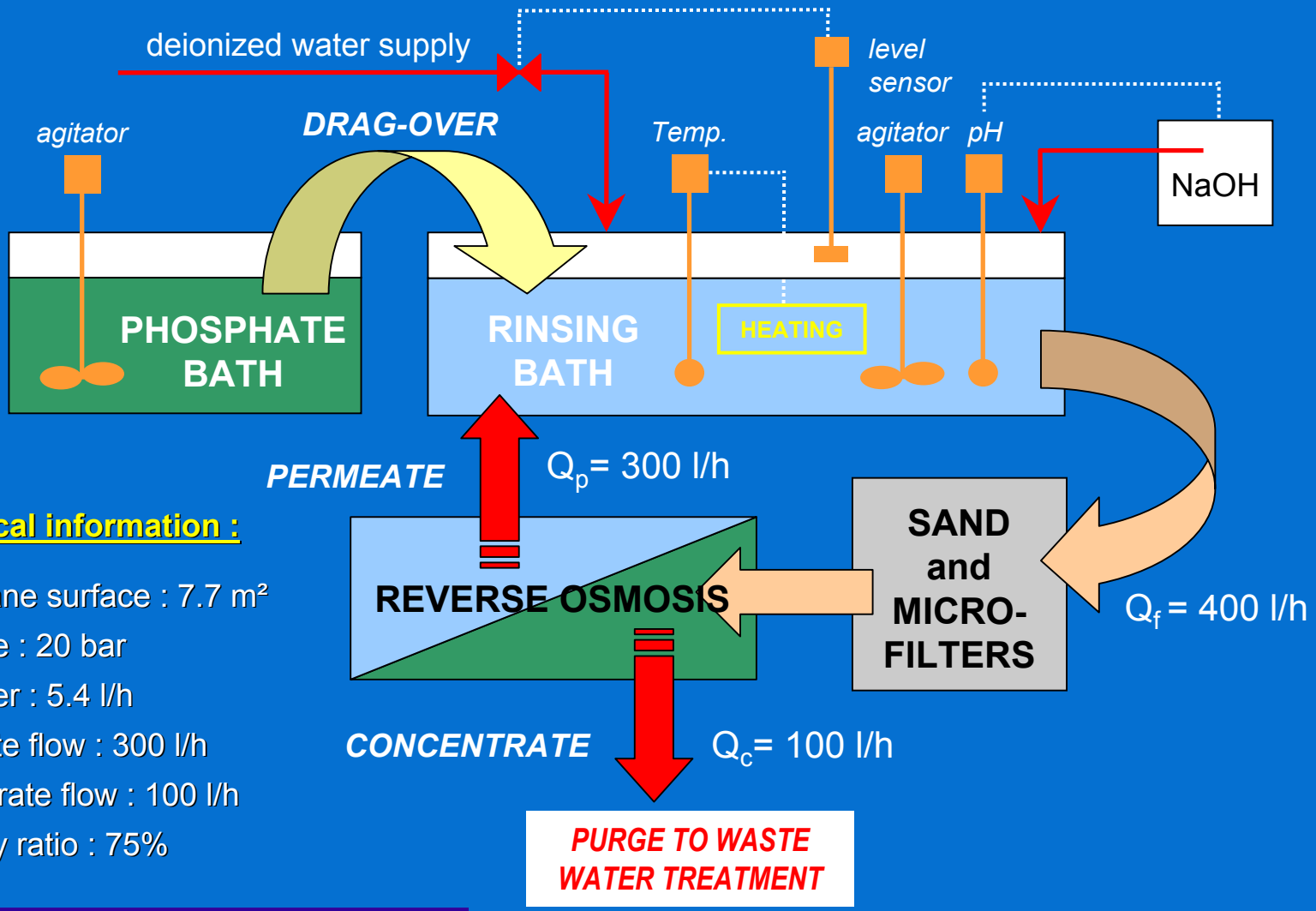


Equilibrium



Water is passing through the membrane from the concentrated solution compartment to the water compartment

Principle flow diagram test-unit (1 stage)



❑ **Technical information :**

- ✓ membrane surface : 7.7 m²
- ✓ pressure : 20 bar
- ✓ drag over : 5.4 l/h
- ✓ permeate flow : 300 l/h
- ✓ concentrate flow : 100 l/h
- ✓ recovery ratio : 75%

Recovery (symbol S)

Definition

The recovery ratio is the relation between the permeate flow rate and the feed flow rate (sum of permeate and concentrate flow).

Formula

$$S = \frac{Q_p}{Q_f} \times 100\ \%$$

Recovery during tests

✓ adjusted to obtain an average recovery of 75%

(80% at start, 70% at end)

Rejection (symbol R)

Definition

The reductions of components were calculated by comparing the content in the permeate with the content in the feed.

Formula

$$R = 1 - \left(\frac{C_p}{C_f} \right) \times 100\ \%$$

Theoretical values

- ✓ NaF 98%
- ✓ $NaNO_3$ 93%
- ✓ $NiSO_4$ 99%
- ✓ SiO_2 98%

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Flux (symbol J)

➢ Definition

The flux is the unit membrane throughput, usually expressed in volume per unit time per area, such as liters per hour per m².

➢ Formula

$$J = \frac{Q_p}{A}$$

➢ Theoretical values

✓ Nominal active surface of the FilmTec membrane : 41 m²

✓ Product water flow rate : 44 m³/d

⇨ Flux : 45 l/m².h

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Technical data FilmTec RO membrane

➢ Membrane characteristics

- ✓ Supplier : FilmTec Corporation (Dow Chemical Company)
- ✓ Type : spiral wound thin-film composite polyamide

➢ Operating limits

- ✓ Maximum operating pressure : 69 bar
- ✓ Maximum operating temperature : 45 °C
- ✓ Free chlorine tolerance : < 0.1 mg/l
- ✓ pH range during operation : 2-11
- ✓ pH range during cleaning (short-term 30 min.) : 1-12

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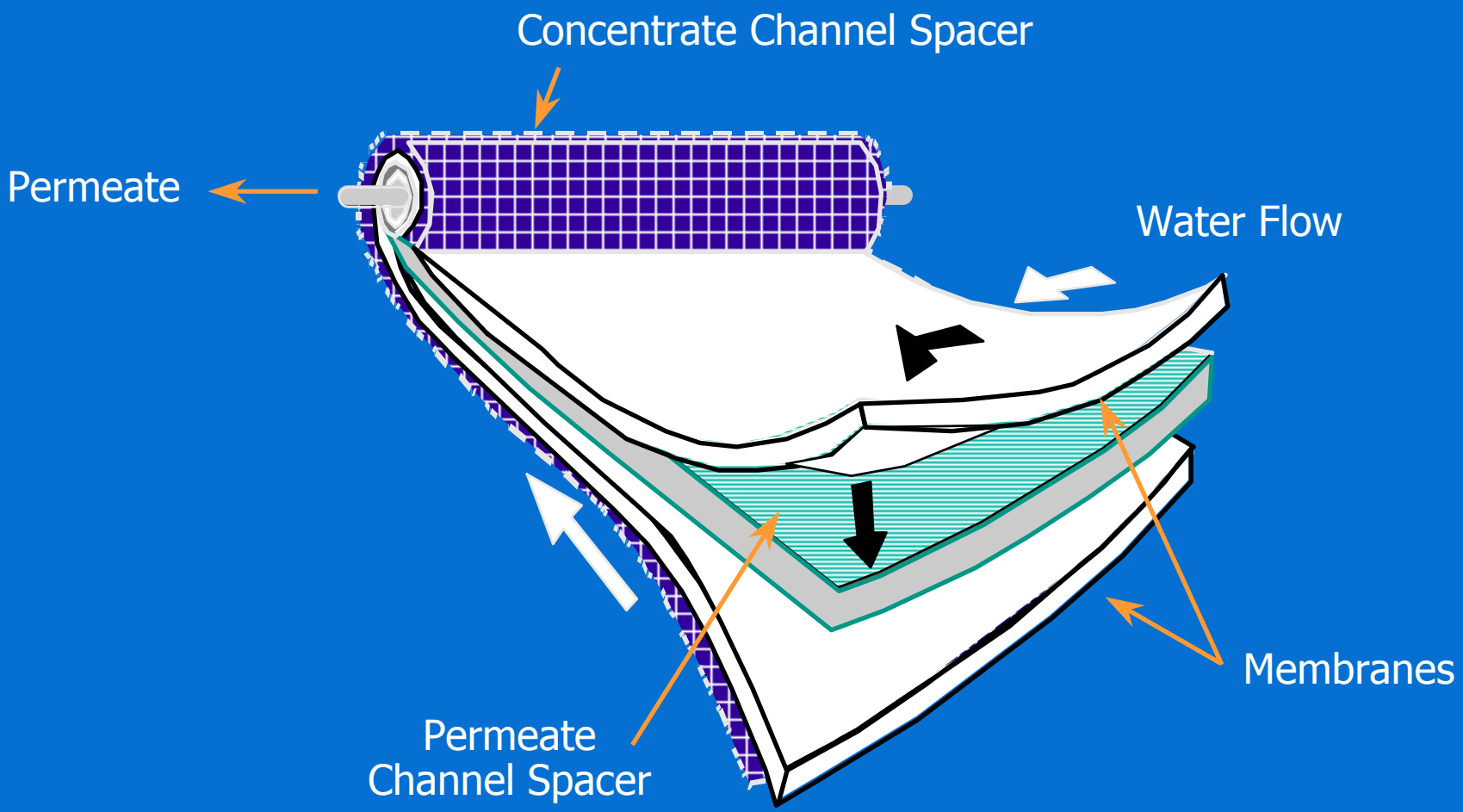
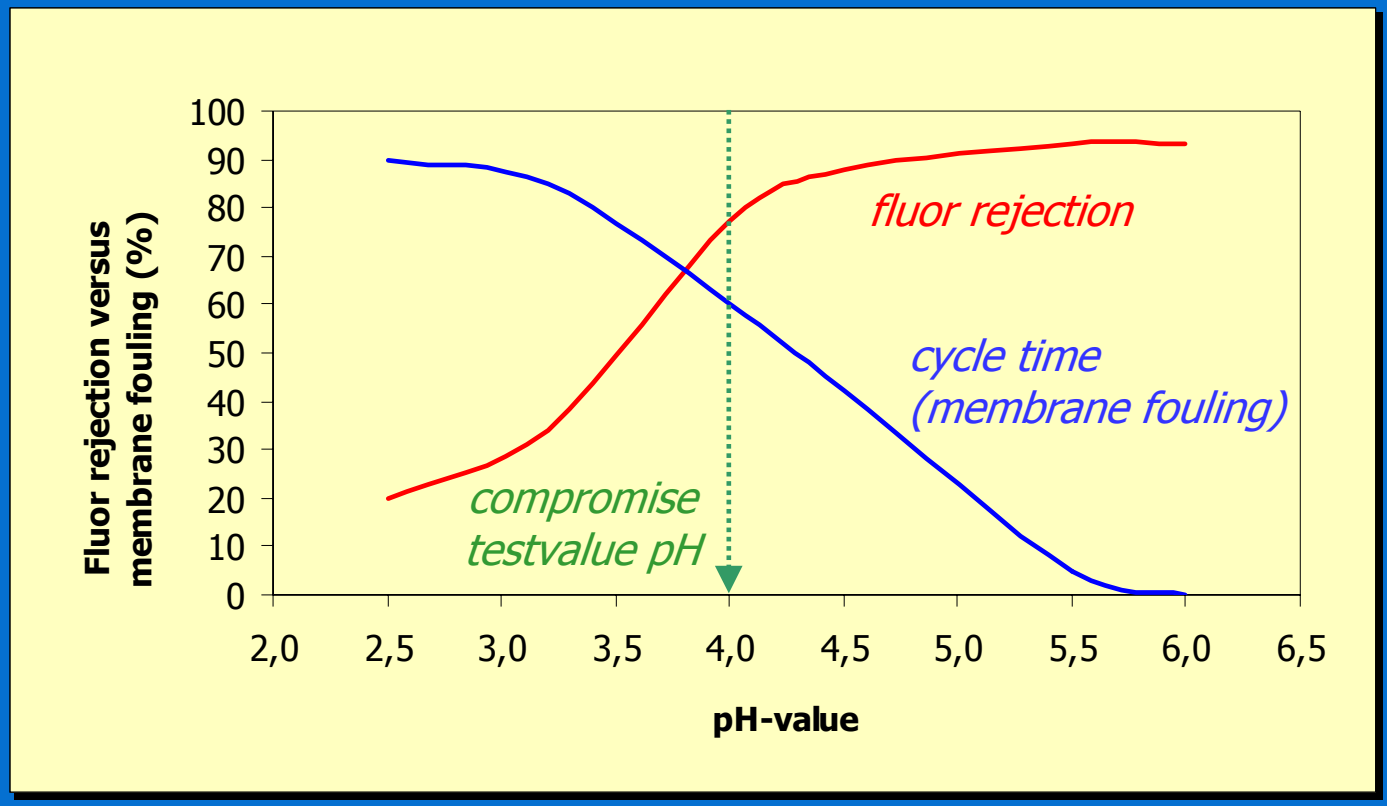
Spiral Wound RO Membrane



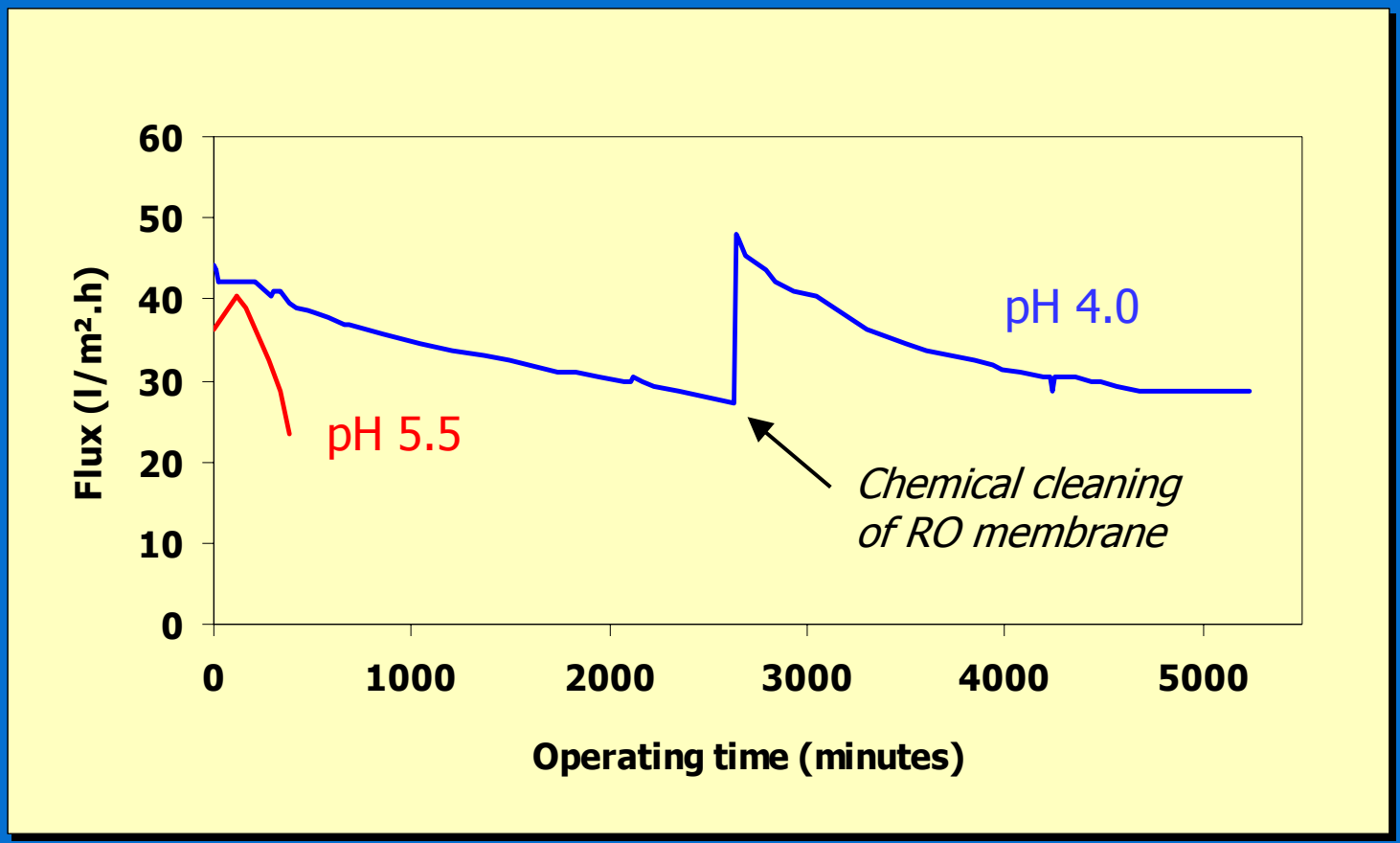
Photos FilmTec RO elements

Influence of the pH on the RO process

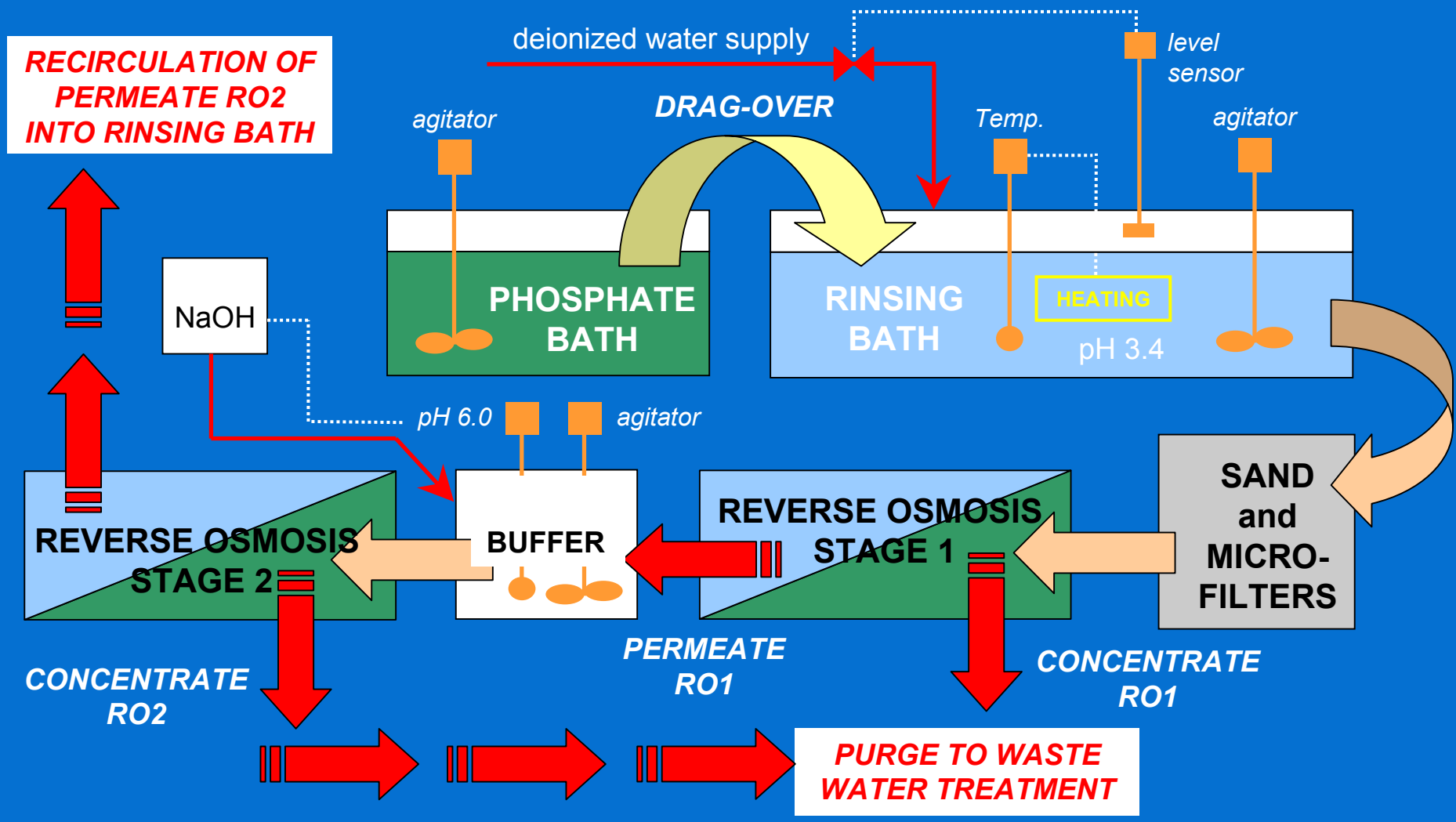


➠ Fluor rejection and membrane fouling are strongly pH dependant !

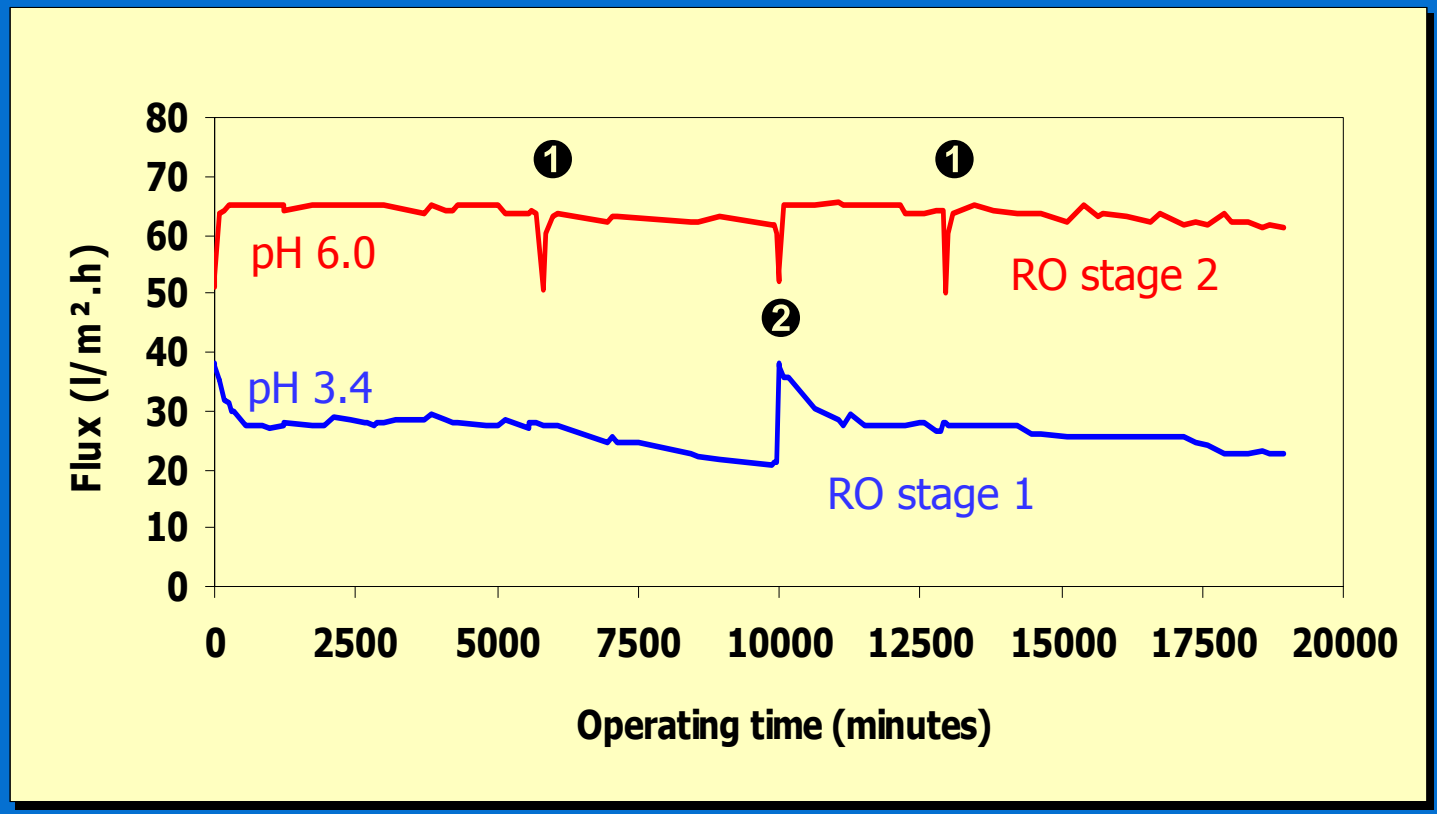
Evolution flux RO test in 1 stage



Principle flow diagram test-unit (2 stages)



Evolution flux RO test in 2 stages



1 *Lower temperature after weekend*

2 *Chemical cleaning of RO membranes*

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Test results analysis RO - stage 1

		Dipphosphatation area		Reverse osmosis – stage 1 -		
Parameter	Unit	Phosphate bath	Rinsing bath	Permeate RO1	Concentrate RO1	Rejection RO1
Flow	l/h	5.4	325	225	100	-
pH	-	3.4	3.6	3.3	3.8	-
Conductivity	µS/cm	18220	402	192	771	52 %
Zinc	mg Zn/l	1580	17	0.21	49	99 %
Nickel	mg Ni/l	628	7.5	< 0.1	18	> 99 %
Manganese	mg Mn/l	660	7.4	< 0.2	18	> 97 %
Phosphate	mg P/l	5680	63	0.13	169	> 99 %
Nitrate	mg N/l	1690	18	3.7	44	80 %
Nitrite	mg N/l	1.8	0.03	< 0.015	0.06	> 56 %
Ammonium	mg N/l	250	2.6	0.44	6.4	98 %
Fluoride	mg F/l	1700	21	9.5	48	55 %
Sodium	mg Na/l	4970	59	0.6	138	99 %

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Test results analysis RO - stage 2

		Dipphosphatation area		Reverse osmosis – stage 2 -		
Parameter	Unit	Phosphate bath	Buffer tank	Permeate RO2	Concentrate RO2	Rejection RO2
Flow	l/h	5.4	540	495	45	-
pH	-	3.4	6.3 *	6.6	59	-
Conductivity	µS/cm	18220	79	25	440	68 %
Zinc	mg Zn/l	1580	< 0.2	< 0.2	0.76	-
Nickel	mg Ni/l	628	< 0.1	< 0.1	0.23	-
Manganese	mg Mn/l	660	< 0.2	< 0.2	0.23	-
Phosphate	mg P/l	5680	0.09	< 0.05	0.38	-
Nitrate	mg N/l	1690	3.8	0.90	24	76 %
Nitrite	mg N/l	1.8	0.02	< 0.015	0.10	-
Ammonium	mg N/l	250	0.04	0.02	0.16	-
Fluoride	mg F/l	1700	9.5	2.9	55	69 %
Sodium	mg Na/l	4970	18	4.9	95	73 %

* Neutralization to pH 6.0 by dosing NaOH

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Test results analysis RO - stage 1 & 2

		Dipphosphatation area		Reverse osmosis – stage 1 & 2 -		
Parameter	Unit	Phosphate bath	Rinsing bath	Buffer tank	Permeate RO2	Rejection RO1 & RO2
Flow	l/h	5.4	325	540	495	-
pH	-	3.4	3.6	6.3 *	6.6	-
Conductivity	µS/cm	18220	402	79	25	94 %
Zinc	mg Zn/l	1580	17	< 0.2	< 0.2	> 99 %
Nickel	mg Ni/l	628	7.5	< 0.1	< 0.1	> 99 %
Manganese	mg Mn/l	660	7.4	< 0.2	< 0.2	> 97 %
Phosphate	mg P/l	5680	63	0.09	< 0.05	> 99 %
Nitrate	mg N/l	1690	18	3.8	0.90	95 %
Nitrite	mg N/l	1.8	0.03	0.02	< 0.015	> 95 %
Ammonium	mg N/l	250	2.6	0.04	0.02	> 99 %
Fluoride	mg F/l	1700	21	9.5	2.9	86 %
Sodium	mg Na/l	4970	59	18	4.9	92 %

* Neutralization to pH 6.0 by dosing NaOH

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Chemical cleaning of RO membranes

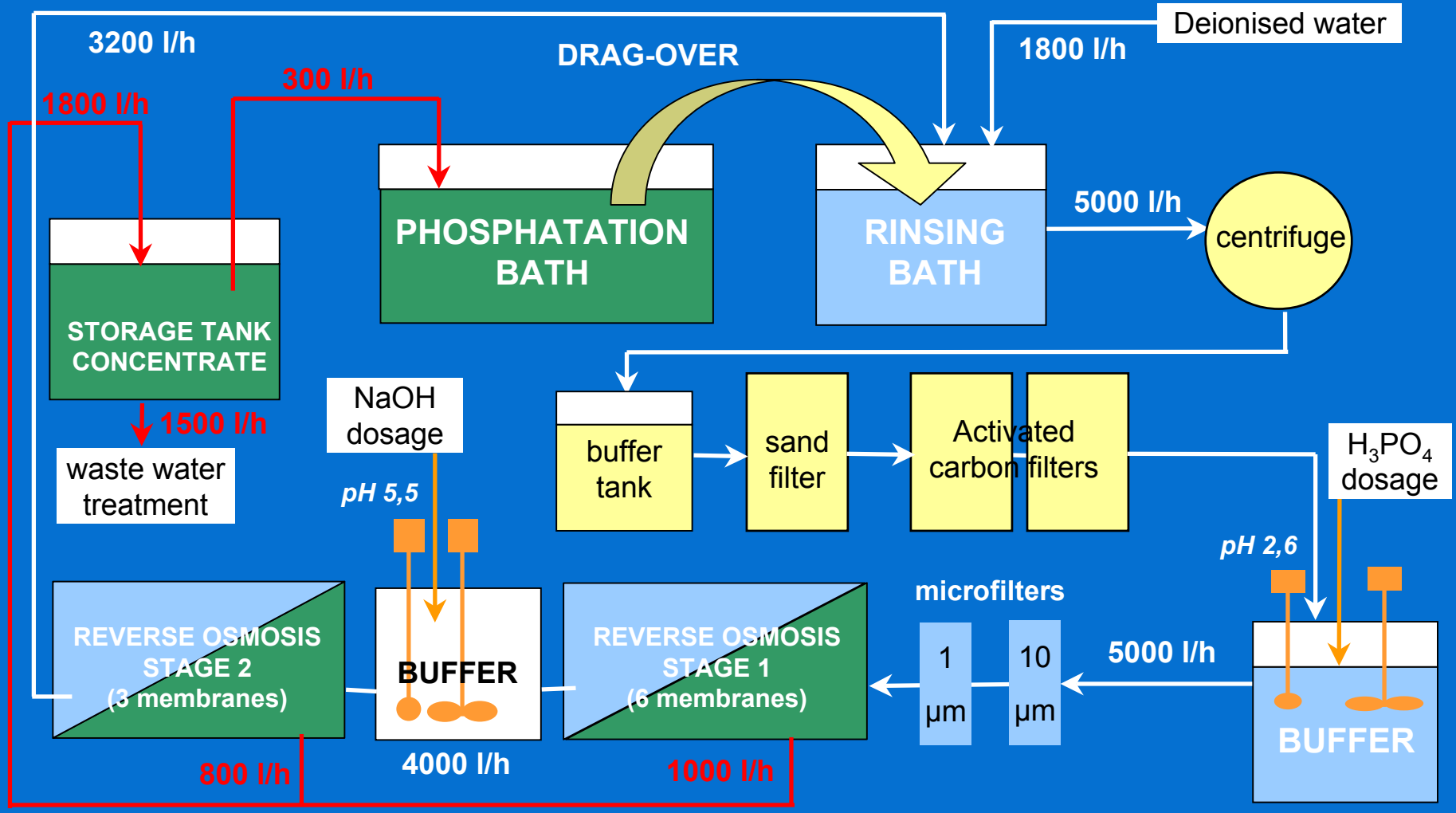
➢ Acid cleaning

- ✓ product name : P3-ultrasil 75 (Henkel Ecolab)
- ✓ composition : based on nitric acid (max. 30% HNO_3)
- ✓ dosage : 0.6% (6 ml/l or 8.4 g/l in deionized water)
- ✓ temperature during cleaning : operating temperature + 5°C
- ✓ duration : 30' cleaning with product; 3 x 10' rinsing with deionized water

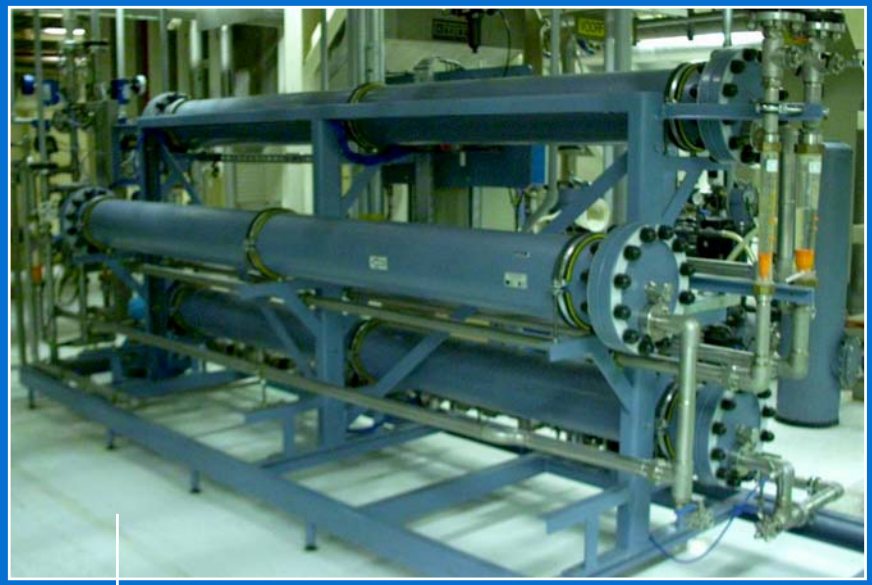
➢ Alkaline cleaning

- ✓ product name : P3-ultrasil 11 (Henkel Ecolab)
- ✓ composition : based on sodium hydroxide (max. 30% NaOH)
- ✓ dosage : 0.5% (5 g/l in deionized water)
- ✓ temperature during cleaning : operating temperature + 5°C
- ✓ duration : 30' cleaning with product; 3 x 10' rinsing with deionized water

Final reverse osmosis installation VCG



Reverse osmosis installation VCG



Membrane modules

Cleaning unit

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Savings reverse osmosis VCG

Water consumption

✓ Consumption of deionised water decreased from 5,0 to 1,8 m³/h per phosphate line

↳ 32.400 m³/year or 0,12 m³/car

↳ 64.800 EUR/year or 0,24 EUR/car

Running cost waste water treatment

✓ Industrial waste water flow decreased from 5,0 to 1,5 m³/h per phosphate line

↳ 35.100 m³/year or 0,13 m³/car

↳ 175.500 EUR/year or 0,65 EUR/car

TOTAL SAVINGS WATER & WASTE WATER
240.300 EUR/year or 0,89 EUR/car

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Pay-back period reverse osmosis VCG

➢ Estimation operating cost per year

✓ power consumption	8.000 EUR
✓ cleaning membranes and chemicals	10.000 EUR
✓ replacing membranes	30.000 EUR
✓ manpower	6.000 EUR

➠ TOTAL OPERATING COST RO
54.000 EUR/year or 0,20 EUR/car

➢ Estimation pay-back period

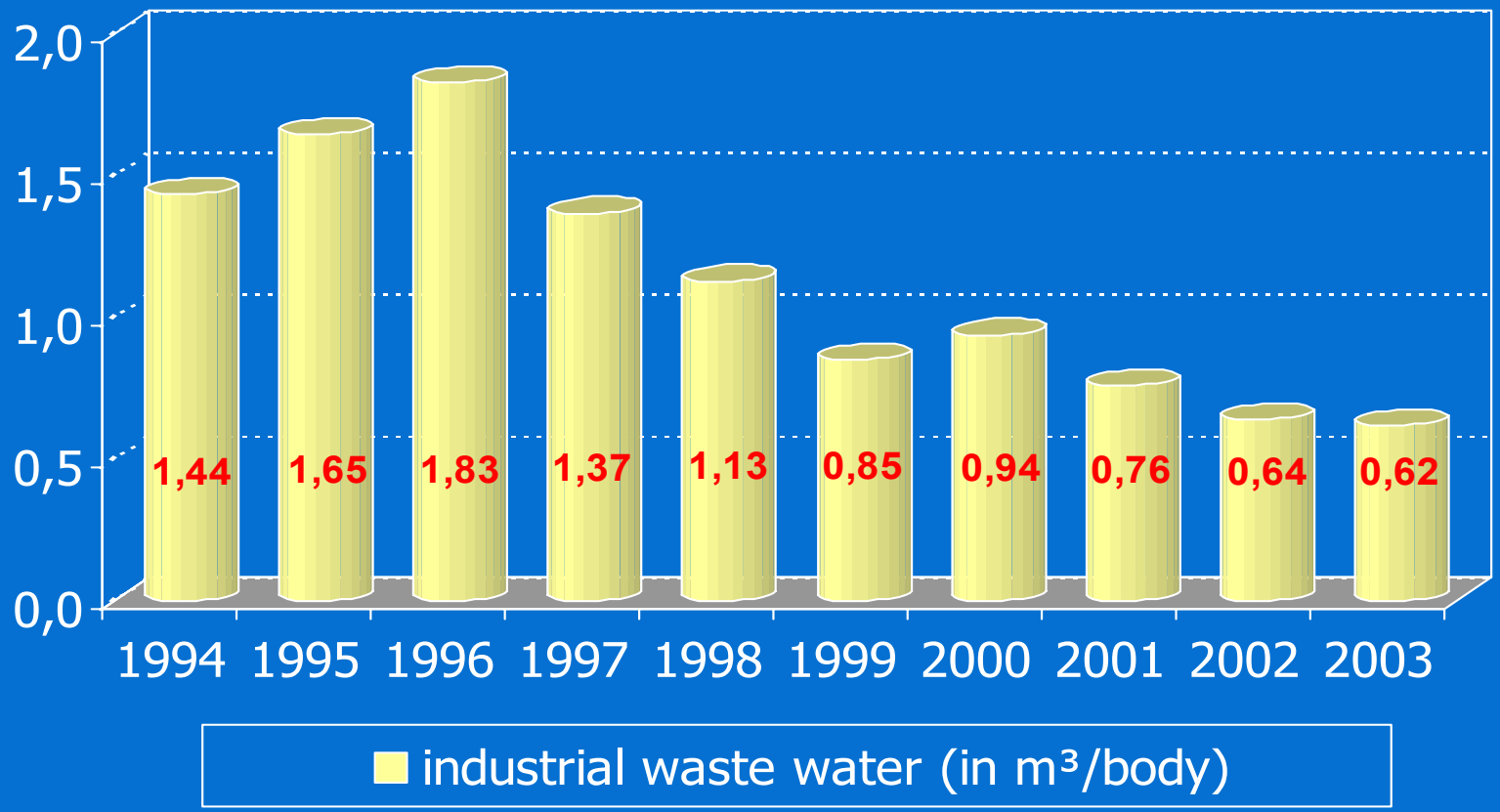
✓ netto saving	186.300 EUR
✓ total investment cost	750.000 EUR

➠ ESTIMATED PAY-BACK PERIOD RO
4 YEARS

Extra information RO results VCG

- Significant decrease of water consumption and amount of industrial waste water to "World Class" level
- Fluor concentrations in effluent far below discharge standard
- Project nominated for the European Environmental Press (EEP) Award 2003 (Pollutec, Paris)

Evolution waste water production VCG



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