# OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2

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UNIVERSITAT ROVIRA i VIRGILI

# Optimisation of wastewater treatment at Ecoparc 2

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Supervised by Allan D. Mackie and Inmaculada Sanz

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Departament d'Enginyeria Química

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HAGO CONSTAR que el presente trabajo, titulado "OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2", que presenta MARIA ELOISA ALBACETE GARCIA para la obtención del título de Doctor, ha sido realizado bajo mi dirección en el Departamento de INGENIERIA QUÍMICA de esta universidad.

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# Nomenclature

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|---|---|
| ACA | Agència Catalana de l'Aigua |
| AMB | Àrea Metropolitana de Barcelona |
| ARC | Agència de Residus de Catalunya |
| BGS | Bacteria Growth Speed |
| BOD | Biological Oxygen Demand |
| BW | Black Water |
| C | Collector |
| $C_{ammonium}$ | Ammonium load |
| $C_{COD}$ | COD load |
| $C_m$ | Mass load |
| COD | Chemical Oxygen Demand |
| CP | Connection Points |
| CRT | Cellular Residence Time |
| $C_v$ | Volumetric load |
| CW | Clean Water |
| DM | Dry Matter |
| DO | Dissolved Oxygen |
| GD | Denitrification grade |
| GW | Grey Water |
| HRT | Hydraulic Residence Time |
| IDS | Inerts Dissolved Solids |
| IE | Input Effluents |
| IF | Inflow |
| IREC | Catalonia Institute for Energy Research |
| ISS | Inerts Suspended Solids |
| IWW | Industrial Wastewater |

| | |
|---|---|
| MBR | Membrane BioReactor |
| MW | Municipal Water |
| $N_2$ | Nitrogen gas |
| $N_{org}$ | Organic Nitrogen |
| $N_{total}$ | Total Nitrogen |
| $N\text{-}NH_4$ | Ammonium |
| $N\text{-}NO_2$ | Nitrite |
| $N\text{-}NO_3$ | Nitrate |
| OE | Output Effluents |
| OF | Outflow |
| PNT | Santandardised Work Procedures |
| PW | Process Water |
| RD | Royal Decree |
| Rel.F/M | Food/microorganism relation |
| SF | Solubilisation Factor |
| SVI | Sludge Volume Index |
| TDS | Total Dissolved Solids |
| TIS | Total Inerts Solids |
| TKN | Total Kjeldahl Nitrogen |
| TL | Tanks Level |
| TOC | Total Organic Carbon |
| TS | Total Solids |
| TSS | Total Suspended Solids |
| TVS | Total Volatile Solids |
| USW | Urban Solids Waste |
| UWWTS | Urban Wastewater Treatment Station |
| VDS | Volatile Dissolved Solids |
| VS | Volatile Solids |

| | |
|---|---|
| VSS | Volatile Suspended Solids |
| WDF | Waste Derived Fuel |
| WM | Water Meter |
| WWTP | Wastewater Treatment Plant |
| $\eta_{COD}$ | COD performance |
| $\eta_{N}$ | N performance |

# SUMMARY

Ecoparc 2 is an Urban Solid Waste (USW) treatment plant managed by UTE Ebesa which belongs to the Barcelona Metropolitan Area (AMB) and is located in Montcada i Reixac.

In its facilities the following RSU fractions are treated: packaging (yellow container), kitchen waste (brown container) and non-recyclable mixed waste (grey container), with the objective of transforming waste into compost, biogas and recovering materials that have the potential to be recycled.

The plant occupies an area of 11.7 ha and has a nominal capacity of 27,500 tons/year of packaging, 120,000 tons/year of kitchen waste and 160,000 tons/year of non recyclable mixed waste.

Ecoparc 2 has been in operation since 2004 and is constantly improving, innovating and optimising its processes.

As a result of the solid waste treatment, a large amount of process wastewater is generated which also requires treatment before being discharged. Ecoparc 2 has an MBR (Membrane Bio Reactor) wastewater treatment plant, which was initially designed to treat much lower concentrations of COD and ammonia than those that it is currently treating. Although the wastewater treatment plant has been expanded, its performance needs to be reviewed to ensure an optimal operation.

Anaerobic digestion contributes 75% to the total wastewater produced, and has a high COD and N-NH3 concentration (40.000 mg/l COD and 4.500 mg/l N-NH4).

This thesis arises from the need for an optimisation of the wastewater at Ecoparc 2 with regards to the quantity and quality of the discharge. To achieve these objectives, the study has been divided into 5 phases:

Phase 1. Characterisation of the wastewater

It is necessary to know in detail the characteristics and production of all the different wastewaters being produced.

There are 4 types of wastewater at Ecoparc 2:

a) Black water (or sanitary waters) (BW): those that come from the toilets (bathrooms, showers…).

b) Clean water (CW): rainwater that has been captured on the roofs and has not been in contact with the waste. These effluents are directed to the clean water tank and is used in the process, thus reducing the Municipal Water (MW) consumption.

c) Grey water (GW): rainwater that has been in contact with the waste and must be analysed before being discharged. These effluents are directed to the retention tank for analysis.

d) Process water (PW): waters produced in waste treatment processes such as composting PW or sludge from anaerobic digestion.

During this phase all possible effluents have been determined and codified at Ecoparc 2. In addition, all the tunnels through which the wastewater follows have been reviewed taking into account the type of wastewater that flows through them and their exact location. Water meters have also been installed in order to calculate the flow rates of all possible effluents. From this information, it has been observed that the areas that have a greater MW consumption are those from the wastewater treatment plant (WWTP), air treatment and anaerobic digestion.

35% of the MW consumed in the treatment plant is due to the refrigeration towers and the vacuum pump. At present, it has been possible to shut down one of the refrigeration towers and the vacuum pump thus reducing the MW consumption. The water discharged from the towers contains practically negligible COD and ammonium values, so that these effluents are collected in a deposit and after an analysis, are sent to the clean water tank to be able to reuse the water, thus avoiding higher MW consumption.

The grey water production is approximately 100 $m^3$/day, of which 26% comes from the air treatment area and accounts for an ammoniac load of 55% of the total grey water, and has the highest COD and ammonium concentration. In order to avoid mixing this effluent with the rest, a pipeline has been installed that channels this effluent directly to the anaerobic digestion area. Here it is reused for cleaning the centrifuge and preparation of the flocculant, thus reducing the MW consumption. It ends its journey in the PW tank to be treated in the treatment plant and, since it is not mixed with the rest of the grey water, improves the final discharge quality.

A homogenisation tank has also been implemented to avoid peaks in the discharge that collects the grey water from the retention tank and the permeate from the WWTP.

This tank help to maintain more homogeneous properties and a more stable and constant discharge flow.

Phase 2. Optimisation of the wastewater treatment plant

Several control parameters and analyses have been established in intermediate reactors in order to monitor the state of the WWTP. This allows the operators to observe variations in the process that can destabilise it, and help to minimise the response time needed to be able to restore normality.

Several flocculants have been tested to determine the one that obtains the best performance in the centrifuge, that is, obtaining lower solids concentrations in the liquid phase.

Equations have been made available for the operators that calculate the required dosage of phosphoric acid, sodium hydroxide and methanol and purge requirements. In this way reagent excesses or shortages are avoided by maintaining at good operation of the WWTP and by controlling the solids content inside the reactors, avoiding a high viscosity.

Finally, higher methanol dosing, aeration and pause periods have been established in the combined reactor, which is thought to favour the oxia and anoxia conditions for the nitrification and denitrification reactions, respectively.

Phase 3. PW solids separation

A high solids content inside the reactor produces an increase in the sludge viscosity hindering the necessary solubility and diffusion of the nutrients and dissolved oxygen required for the microbial degradation. In order to be able to reduce the PW solids quantity, its separation has been studied by using process equipment available in the company (centrifugation) and other equipment available commercially (membranes).

In the tests performed with the centrifuge it has been observed that a double cycle of the PW favours the PW quality, being able to obtain at present concentrations of less than 3% TS compared to the previous ones of less than 5% TS.

In the tests performed with commercial membranes, very good results have been obtained. The membranes in the microfiltration range are sufficient for the total solids removal, but the membranes in the ultrafiltration range obtains better results in relation to the fouling and recovery of the membrane permeability. There is a drawback in that

the effluent must be pretreated by screening at 100 microns to remove the coarser solids and diluted to 50% to decrease the viscosity. However, this dilution can be done by recycling part of the produced permeate..

Phase 4. New technologies: thermal hydrolysis and stripping

In order to increase the COD biodegradability and reduce the ammonium load, the possibility of incorporating a pretreatment treatment consisting of thermal hydrolysis and stripping has been studied.

In the thermal hydrolysis it was observed that COD biodegradability increases at higher temperatures and longer exposure time, obtaining a 24% increase in the COD solubility at a temperature of 75 ° C during a period of 120 minutes.

This study has focused on the PW solids separation since a LIFE project has been approved at the Ecoparc 2 where the stripping technology will be studied in more depth. According to the first stripping tests carried out, a 50 % ammonium reduction could be achieved.

Phase 5. New biological processes

Although the LIFE project will focus on new biological processes, the possibilities for improvement through Anamox biological processes have been reviewed in this phase.

An approximation of the improvements in the ammonia elimination can be calculated for Anamox processes, where 62.5% of the energy savings are found to be obtained due to a reduced need for dissolved oxygen and a lower cost since it does not need the contribution of carbon sources as methanol or acetic acid. However, a way to eliminate COD would have to be found, since this process does not eliminate such loads.

As a result of this study, the MW consumption has been reduced by 46.3 % in relation to 2009 and by 33.8 % in relation to the year 2013 when this study was started. The discharge amount has decreased by 41.2 % compared to the year 2014, which means more than 40,000 $m^3$ less discharge today. And, finally, the permeate obtained in the WWTP contains 39.3% less COD and 62.2% less ammonium in relation to 2008 and 38.0% less COD and 10.9% less ammonium compared to the year 2013.

# INTRODUCTION

Sustainable development is defined as that "*which meets the needs of the present without compromising the ability of future generations to meet their own needs*" (source: Brundtland report (1987)).

Industry and agriculture have always been blamed for the degradation of the environment. Although it is true that they are responsible for causing severe negative impacts, they are not the only activities that cause it. In addition, technology has advanced to the point that many possible environmental impacts can potentially be treated, thus facilitating sustainable development.

Sustainable development does not focus on the environment, but is a relationship between Social, Economic and Ecological ambits (figure 1).

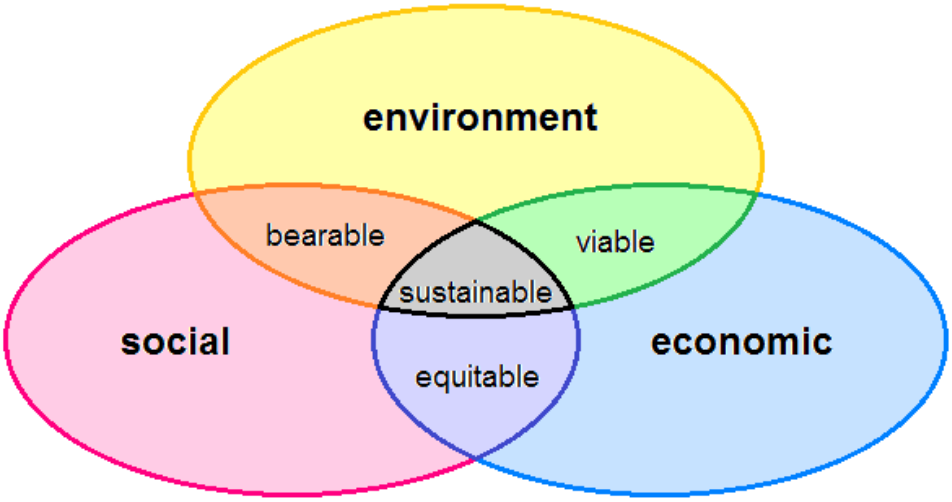



***Figure 1**: Sustainable development (image author: Eloisa Albacete Garcia)*

According to Conesa (1997, p.31) sustainable development has the following main characteristics:

- Maintains general life quality
- Allows continued access to natural resources
- Prevents lasting environmental damage

The materials cycle is basically composed of two parts. The first is the waste generation portion (production, use and consumption), while the second is waste management (collection, treatment and/or disposal) (figure 2).

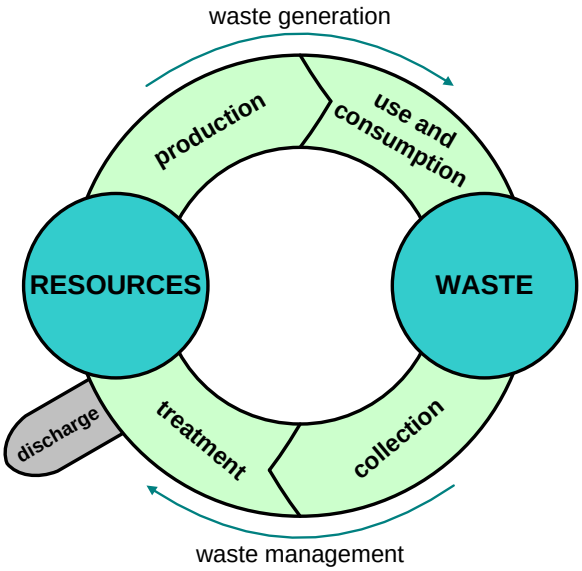



***Figure 2**: Materials cycle (image author: Eloisa Albacete Garcia)*

Since resources are limited, it is important to try to close this materials cycle reusing treated waste as secondary raw materials, and discharge the minimum possible waste to the environment. In figure 3 a hierarchy favouring sustainable development with regards to waste is given.

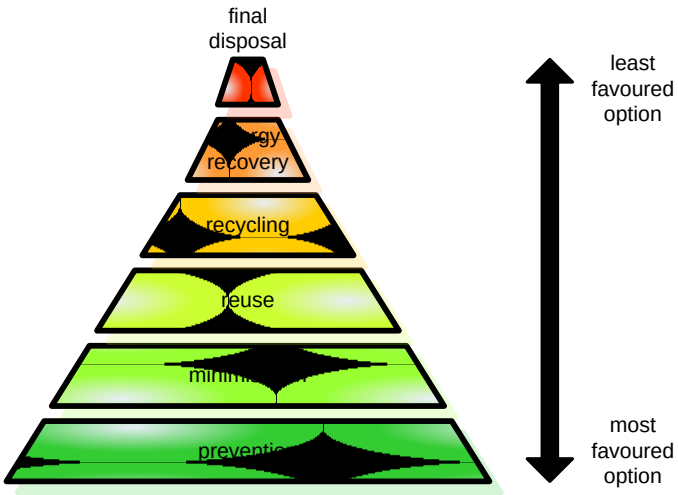



***Figure 3**: Waste hierarchy (image author: Eloisa Albacete Garcia)*

Water is essential for life. Its quantity and quality are directly related to the health of people and animals. Unfortunately, it is an increasingly scarce commodity because available water catchment sources for human consumption are becoming progressively limited. Additionally, the high human consumption of water is strong negative factor. Figure 4 shows the water distribution on earth according to the World Resources Institute.

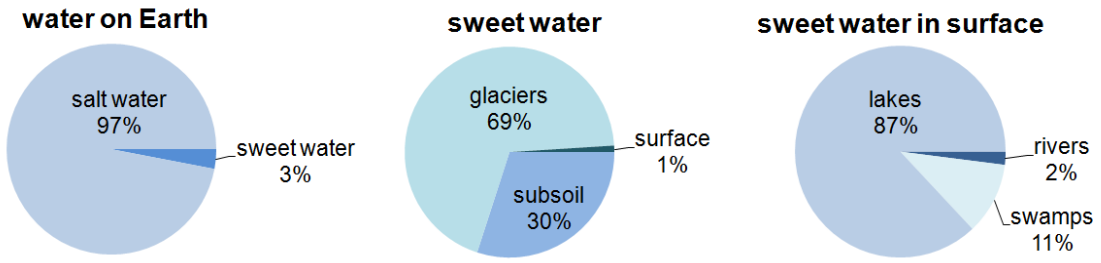



***Figure 4:*** *Water distribution on Earth (image author: Eloisa Albacete Garcia from source National Statistics Institute through World Resources Institute)*

The amount of water collected in relation to the water available for human consumption shows a potentially unfavourable scenario for humanity in the future. This strengthens the idea that all people should make an efficient use of water.

The ARC (Agència de Residus de Catalunya) is a public legislation entity inscribed in the European register of management and environmental auditing (EMAS). The ARC has competence on waste generated in Catalonia (figure 5) and those managed in its territory (source: [www.arc.cat](http://www.arc.cat)).


***Figure 5:*** *Catalonia location (image author: Evan Centanni)*

The ARC is responsible for the control and management of the following waste categories in Catalonia:

- Municipal waste
- Industrial waste
- Livestock waste
- Construction waste
- Sanitary waste

Each category poses specific challenges that require specific action policies and strategies adapted to their nature and difficulty. As a result, the following programmes have been developed with their corresponding objectives: municipal waste management programmes (PROGREMIC), industrials (PROGRIC) and construction (PROGROC).

Within municipal waste, there are subdivisions depending on the territory in charge of its management. In Catalonia, municipal waste is classified into 5 different fractions:

1. **Glass**. Composed of glass packings and bottles.
2. **Paper and cardboard**. Involves cardboard packagings, boxes, office paper, magazines and other paper class.
3. **Light packaging**. Plastic, ferric or aluminium packagings.
4. **Organic matter**. Biodegradable matter such as food leftovers or plants.
5. **Other**. Wastes that are not included in any of the previous sections.

The ACA (Agència Catalana de l'Aigua) is a public company responsible for planning and managing the integral water cycle in Catalonia. The ACA has developed the PSARI (Programa de Sanejament d'Aigües Residuals Industrials) that comply with the quality objectives that fixed the sanitation programs created from Directive 91/271/EEC of 21 May for urban wastewater treatment.

The PSARI establishes a rules system, relationships and infrastructures that allow for industrial discharge and natural environment restoration in compatible conditions with good ecological waters status according to the industrial sanitation model. Currently, there are 2 types of discharges:

a) **Discharges to the physical environment (channel or sea).** Industries contempled in this group must remove all incompatible pollutants for the environment, therefore, discharge limits are very demanding.
b) **Discharges to sanitation systems.** Since they will be treated by an Urban Wastewater Treatment Station (UWWTS), discharge limits are less restrictive.

Years ago, Industrial Wastewater (IWW) sanitation was based on the autonomous discharge by the industries to the natural environment. Currently, however, IWW has been integrated into the urban wastewater sanitation programme, so that the industrial waters discharge, through an industrial collector to an, urban wastewater treatment plant. Therefore, the discharge limits are less restrictive because the discharge is subsequently treated in the urban wastewater treatment plant.

Decree 130/2003, 13th May, by which sanitation public services Regulations are approved, sets the industrial wastewater discharge limits to a collector in Annex II:

*"These Annex limitations have been established in response to:*

*a) Public system sanitation capacity and use*
*b) Discharge limits setting by systems according to Directive 91/271/EEC*
*c) Directive 76/464 and other development directives and Royal Decree 995/2000*
*d) Receiving environment protection"*

Tables 1 and 2 show Decree 130/2003 Annex II blocks 1 and 2 which establish the discharge limits.

***Table 1:*** *Treatable parameters in UWWTS indicating a low impact on the associated environmental quality objectives*

| parameters | limit value |
|---|---|
| temperature | 40 ºC |
| pH (range) | 6 - 10 |
| SS (suspended substances) | 750 mg/L |
| $BOD_5$ | 750 mg $O_2$/L |
| DQO | 1.500 mg $O_2$/L |
| oils and fats | 250 mg/L |
| chlorides | 2.500 mg $Cl^-$/L |
| conductivity | 6.000 µS/cm |

| parameters | limit value |
|---|---|
| sulphur dioxide | 15 mg $SO_2$/L |
| sulphates | 1.000 mg $SO_4^{2-}$/L |
| total sulphides | 1 mg $S^{2-}$/L |
| dissolved sulphides | 0,3 mg $S^{2-}$/L |
| total phosphorus | 50 mg P/L |
| nitrates | 100 mg $NO_3^-$/L |
| ammonium | 60 mg $NH_4^+$/L |
| organic and ammonia nitrogen | 90 mg N/L |

***Table 2:*** *Pollutant parameters indicating intractability of UWWTS and with a significant impact on the associated environmental quality objectives and treated water potential usages*

| parameters | limit value | parameters | limit value |
|---|---|---|---|
| total cyanides | 1 mg $CN^-$/L | zinc | 10 mg Zn/L |
| phenols index | 2 mg $C_6H_5OH$/L | IS (inhibitory substances) | 25 equitox |
| fluorides | 12 mg $F^-$/L | colour | negligible dilution 1/30 |
| aluminium | 20 mg Al/L | noniphenol | 1 mg NP/L |
| antimony | 1 mg Sb/L | anionic surfactants | 6 mg LSS/L |
| barium | 10 mg Ba/L | total pesticides | 0,10 mg/L |
| boron | 3 mg B/L | polycyclic aromatic hydrocarbons | 0,20 mg/L |
| cadmium | 0,5 mg Cd/L | BTEX (benzene, toluene, ethylbenzene and xylene) | 5 mg/L |
| copper | 3 mg Cu/L | total triazines | 0,30 mg/L |
| hexavalent chromium | 0,5 mg Cr(IV)/L | hydrocarbons | 15 mg/L |
| total chromium | 3 mg Cr/L | AOX | 2 mg Cl/L |
| tin | 5 mg Sn/L | chloroform | 1 mg $Cl_3CH$/L |
| iron | 10 mg Fe/L | 1,2 dichloroethane | 0,4 mg $Cl_2C_2H_4$/L |
| manganese | 2 mg Mn/L | trichloroethylene (TRI) | 0,4 mg $Cl_3C_2H$/L |
| mercury | 0,1 mg Hg/L | perchloroethylene (PER) | 0,4 mg $Cl_4C_2$/L |
| nickel | 5 mg Ni/L | trichlorobenzene | 0,2 mg $Cl_3C_6H_3$/L |
| lead | 1 mg Pb/L | carbon tetrachloride | 1 mg $Cl_4C$/L |
| selenium | 0,5 mg Se/L | tributyltin | 0,10 mg/L |

The AMB (Àrea Metropolitana de Barcelona) is an institutional organisation that is formed by Barcelona and 35 surrounding municipalities (source: www.amb.cat) (figure 6). One of the AMB competences is the metropolitan territory waste management.


***Figure 6:*** *Municipalities that form the AMB (image author: www.amb.cat)*

The AMB has different infrastructures for municipal waste management:

- 1 bulky item treatment plant
- 13 mobile waste collectors
- 67 waste collection and storage depots
- 1 integral waste recovery plant
- 2 composting plants
- 3 sorting plants
- 4 Ecoparcks

Ecoparc 2 is one of the 4 AMB Ecoparcks, and occupies a surface area of 11 ha, 8 of which are constructed on (see figure 7). It has 150 workers performing different tasks of processing, cleaning, maintenance and management.

***Figure 7:*** *Ecoparc 2 aerial view (image author: Ecoparc 2)*

Ecoparc 2 has a nominal capacity of 27,500 light packaging annual tons, 100,000 organic matter annual tons and 160,000 residual fraction annual tons, and subjected them to different treatments depending on their type:

- Packagings: the different packaging types are classified by a mechanical separation. The separate types are then directed to different recyclers for further processing and return to the materials cycle. The rejection from this process is sent first to energy recovery as Waste Derived Fuel (WDF) and the rest goes to a final disposal.

- Organic matter: improper materials is extracted in a first treatment by manual and mechanical separation. The organic matter obtained is sent to an anaerobic digestor including cogeneration for energy recovery. The sludge from the digestor is composted for recycling as organic fertilizer. The rejection obtained goes to a final disposal.

- Other: additional materials classified as secondary products (cardboard, plastic, glass, etc) are sorted by manual and mechanical separation. The secondary products are sent to different recyclers for further processing and return to the materials cycle. The organic matter composting in tunnels and trenches obtaining a suitable material for construction and slopes rebuilding. The rejection obtained goes to a final disposal.

The Ecoparc 2 has a structure that includes different areas into which the plant is conceptually divided:

- 01 area → staff
- 02 area → reception
- 03 area → pretreatment
- 04 area → anaerobic digestion
- 05 area → cogeneration
- 06 area → composting tunnels
- 07 area → refining
- 08 area → wastewater treatment plant
- 09 area → air treatment
- 11 area → fire extinction
- 12 area → trenches
- 15 area → mobile machinery
- 99 area → others

- 99.1 area → waters
- 99.2 area → administration, visits and changing rooms
- 99.3 area → workshop
- 99.4 area → laboratory
- 99.5 area → road lighting
- 99.6 area → watering gardens
- 99.7 area → outside road
- 99.8 area → fuel station
- 99.9 area → common electrical installations
- 99.10 area → doors

# OBJECTIVES

Ecoparc 2 is an urban solid waste treatment company dedicated, in essence, to improving the environment. In line with this overall goal, this thesis aims to make a detailed analysis of the consumption of water in the plant and the optimisation of its use.

The **main objectives** of this thesis are:

- To reduce the municipal water consumption, optimising the current consumption and/or reusing water effluents where possible.

- To reduce the amount of water discharged into the collector.

- To optimise the wastewater treatment system already existing at Ecoparc 2 and to study possible pretreatment processes to maximise the overall process efficiency.

To accomplish these objectives, the study is divided into 5 phases:

A) Phase 1. Wastewater characterisation

   To have a solid basis on which to carry out the study, a characterization of all points related to water consumption and discharge at Ecoparc 2 is required. During this phase improvements are proposed to help fulfill the main thesis objectives.

   The phase 1 **specific objectives** are:

   - To characterise all wastewater at Ecoparc 2 in order to be able to have a solid basis for the project, and to act on the most problematic areas (in terms of both concentration and quantity), for which the flow, ammonium concentration, COD, total solids and volatile solids of different effluents are determined.

   - To decrease the municipal water consumption.

- To reuse any grey water, so that it can pass to the process water and finish its journey in the wastewater treatment plant.

B) Phase 2. Wastewater treatment plant optimisation

The wastewater treatment plant at Ecoparc 2 and its current operation is studied to optimize and improve its performance.

The phase 2 **specific objectives** are:

- To review the analytical procedures and replace them with standardised methods consistent with the analysed wastewater.
- To assess the wastewater treatment plant sampling point, both the analytical procedure and its periodicity.
- To check the well operation of the wastewater treatment plant and optimise its performance.

C) Phase 3. Process Water solids separation

Investigate the possibility of separating the Process Water solids prior to being fed to the wastewater treatment plant to improve the solubility and facilitate dissemination in the biological process. Only existing and accessible machinery at Ecoparc 2 should be considered.

The phase 3 **specific objectives** are:

- To check the effect of sending the Process Water to a centrifugation prior to the biological with regards to solids, COD and ammonium reduction.
- To determine the optimal and sustainable speed and time that the centrifuge must operate to remove the maximum possible COD load and solids content in the PW.
- To check the viability of implementation at Ecoparc 2.
- To study other alternatives for solids separation.

D) Phase 4. New technologies: thermal hydrolysis and stripping

Study the possibility of including a thermal hydrolysis processes and stripping prior to the wastewater treatment to increase the COD biodegradability and decrease the ammonia concentration in the PW.

The phase 4 **specific objectives** are:

- To increase organic matter biodegradability in effluent with higher volumetric weight by thermal hydrolysis.
- To find the optimal temperature for thermal hydrolysis.
- To reduce the ammonia concentration in the liquid effluent from the anaerobic digestor by aeration and agitation (stripping).

Initially, Phase 5 was considered as one of the objectives and this is contemplated, but at Ecoparc 2 a LIFE Project was approved that studied this subject at pilot scale and in depth. However, a bibliographic study has already been carried out and is attached in Annex I.

E) Phase 5. New biological processes

To study the possibility of wastewater treatment through new biological processes: Anammox or nitrate via.

The phase 5 **specific objectives** are:

- To consider other alternatives for process water treatment in Ecoparc 2.
- To achieve energy savings in water purifying.
- To reduce methanol consumption, previously required for water purifying in the current wastewater treatment plant.

# ANALYSES AND METHODS

## A. Analytical determinations

| | |
|---|---|
| Temperature | *Tools*: temperature sensor Orbisint CPS 11-2AA2ESA and CPK 9-NAA1A or mercury thermometer<br>*Method*: direct reading |
| pH | *Tools*: pH metre XS instruments pH310<br>*Method*: 4500-$H^+$B Standard Methods for the Examination of Water and Wastewater |
| Conductivity | *Tools*: conductivity instrument Endress+Hauser LIQUISYS M COM 253 and Syland Scientific 6000-B-PPC<br>*Method*: 2510B Standard Methods of the Examination of Water and Wastewater |
| Alkalinity | *Tools*: pH metre XS instruments pH310<br>*Method*: 2320B of Standard Methods for the Examination of Water and Wastewater |
| Dissolved oxygen (DO) | *Tools*: oxygen sensor Endress+Hauser LIQUISYS M COM 253 and Syland Scientific 6000-B-PPC<br>*Method*: direct reading |
| Chemical Oxygen Demand (COD) | *Tools*: NANOCOLOR 400D or HACH DR3900 photometer, NANOCOLOR VARIO COMPACT or HACH DRB200-1 heating block, and piston pipette with tips<br>*Method*:<br>- NANOCOLOR → DIN ISO 15705:2002 and ISO 6060 |

- HACH → ISO 6060-1989, DIN 38409-H41-H44

*Procedures*: annex II

Total Organic Carbon (TOC)

*Tools*: NANOCOLOR 400D or HACH DR3900 photometer, NANOCOLOR VARIO COMPACT or HACH DRB200-1 heating block, and piston pipette with tips

*Method*:

- NANOCOLOR → decomposition of the organic carbon and detection of the carbon dioxide formed by means of an indicator
- HACH → EN 1484, DIN 38409-H3

*Procedures*: annex II

Phosphorus (P)

*Tools*: NANOCOLOR 400D or HACH DR3900 photometer, NANOCOLOR VARIO COMPACT or HACH DRB200-1 heating block, and piston pipette with tips

*Method*:

- NANOCOLOR → photometric determination of the yellow phosphate-molybdate-vanadate complex afteracidic hydrolysis and oxidation at 100–120 °C
- HACH →

*Procedures*: annex II

Total Nitrogen ($N_{total}$)

*Tools*: NANOCOLOR 400D or HACH DR3900 photometer, NANOCOLOR VARIO COMPACT or HACH DRB200-1 heating block, and piston pipette with tips

*Method*:

- NANOCOLOR → DIN EN ISO 11905-1
- HACH → DIN EN ISO 11905-1

*Procedures*: annex II

Ammonium

*Tools*: NANOCOLOR 400D or HACH DR3900 photometer, and

| | |
|---|---|
| ($N\text{-}NH_4$) | piston pipette with tips<br>*Method*:<br>- NANOCOLOR → photometric determination as indophenol: at a pH value of about 12.6 ammonium reacts with hypochlorite and salicylate in the presence of sodium nitroprusside as catalyst to form a blue indophenol<br>- HACH → ISO 7150-1, DIN 38406 ES-1<br>*Procedures*: annex II |
| Nitrate ($N\text{-}NO_3$) | *Tools*: NANOCOLOR 400D or HACH DR3900 photometer, and piston pipette with tips<br>*Method*:<br>- NANOCOLOR → photometric determination with 2,6-dimethylphenol in sulphuric acid / phosphoric acid<br>- HACH → ISO 7890-1-2-1986, DIN 38405 D9-2<br>*Procedures*: annex II |
| Nitrite ($N\text{-}NO_2$) | *Tools*: NANOCOLOR 400D or HACH DR3900 photometer, and piston pipette with tips<br>*Method*:<br>- NANOCOLOR → photometric determination with sulphanilamide and N-(1-naphthyl)ethylenediamine<br>- HACH → EN ISO 26777, DIN 38405 D10<br>*Procedures*: annex II |
| Organic Nitrogen ($N_{org}$) | *Method*: calculation<br>*Calculations*:<br>$\mathrm{N_{org}} = \mathrm{N_{total}} - (\mathrm{N - NH_4^+}) - (\mathrm{N - NO_3^-}) - (\mathrm{N - NO_2^-})$ |
| Total Kjeldahl Nitrogen (TKN) | *Method*: calculation<br>*Calculations*: $\mathrm{TKN} = (\mathrm{N - NH_4^+}) + \mathrm{N_{org}}$ |

| | |
|---|---|
| Chloride ($Cl^-$) | *Tools*: NANOCOLOR 400D or HACH DR3900 photometer, and piston pipette with tips<br>*Method*:<br>- NANOCOLOR → DIN EN ISO 15682-D31<br>- HACH → DIN EN ISO 15682-D31<br>*Procedures*: annex II |
| Sulphate ($SO_4^{2-}$) | *Tools*: NANOCOLOR 400D or HACH DR3900 photometer, and piston pipette with tips<br>*Method*:<br>- NANOCOLOR → photometric determination as barium sulphate<br>- HACH → photometric determination as barium sulphate<br>*Procedures*: annex II |
| Sulphide ($S^{2-}$) | *Tools*: NANOCOLOR 400D or HACH DR3900 photometer, and piston pipette with tips<br>*Method*:<br>- NANOCOLOR → DIN 38405-D26/27<br>- HACH → ISO 10530-1991, DIN 38405-D26<br>*Procedures*: annex II |
| Solids | *Tools*: drying oven Memmert and muffle furnace Hobersal HD 280<br>*Method*: 2540 Standard Methods for the Examination of Water and Wastewater |

## B. Control parameters

| | |
|---|---|
| Flows | *Tools*: flowmeter Krohne Aquaflux 010 K and Endress+Hauser Promag 10 W<br>*Method*: direct reading |

Tanks level (TL)

*Tools*: level sensor WIKA S 11

*Method*: direct reading

Volume

*Method*: calculation

*Calculations*:

$$V = V_{maximum}(m^3) \cdot TL(\%)$$

Denitrification grade (ºD)

*Method*: calculation

*Calculations*:

$$ºD = \frac{(N - NH_4^+)_{input} - (N - NO_3^-)_{output}}{(N - NH_4^+)_{input}}$$

COD load

*Method*: calculation

*Calculations*:

$$\text{COD load}\left(\frac{\text{kg COD}}{m^3 \cdot \text{day}}\right) = \frac{COD_{PW}(mg/L) \cdot Q_{input}(m^3/day)}{1{,}000 \cdot V_{total}(m^3)}$$

$NH_4^+$ load

*Method*: calculation

*Calculations*:

$$NH_4^+ \text{ load}\left(\frac{\text{kg } NH_4^+}{m^3 \cdot \text{day}}\right) = \frac{{NH_4^+}_{PW}(mg/L) \cdot Q_{input}(m^3/day)}{1{,}000 \cdot V_{total}(m^3)}$$

COD/$NH_4$ ratio

*Method*: calculation

*Calculations*:

$$\frac{COD}{NH_4}\text{ratio}\left(\frac{\text{kg COD}}{\text{kg } NH_4^+}\right) = \frac{\text{COD load}\left(\frac{\text{kg COD}}{m^3 \cdot \text{day}}\right)}{NH_4^+ \text{ load}\left(\frac{\text{kg } NH_4^+}{m^3 \cdot \text{day}}\right)}$$

| | |
|---|---|
| Hydraulic Residence Time (HRT) | *Method*: calculation<br>*Calculations*:<br>$$\text{HRT (days)} = \frac{\text{V}(\text{m}^3)}{\text{Q}(\text{m}^3/\text{day})}$$ |
| COD performance | *Method*: calculation<br>*Calculations*:<br>$$\text{COD performance (\%)} = \frac{\text{COD}_{\text{input}} - \text{COD}_{\text{output}}}{\text{COD}_{\text{input}}} \cdot 100$$ |
| $NH_4^+$ performance | *Method*: calculation<br>*Calculations*:<br>$$\text{NH}_4^+\text{performance (\%)} = \frac{\text{NH}_4^+{}_{\text{input}} - \text{NH}_4^+{}_{\text{output}}}{\text{NH}_4^+{}_{\text{input}}} \cdot 100$$ |
| Food/microorganism relation (Rel.F/M) | *Method*: calculation<br>*Calculations*:<br>$$\text{Rel. F/M}\left(\frac{\text{kg COD}}{\text{kg SSV} \cdot \text{day}}\right) = \frac{\text{COD}_{\text{input}}(\text{mg/L})}{1{,}000 \cdot \text{SSV(g/L)} \cdot \text{HRT (days)}}$$ |
| Biology inhibition | *Method*: graphic display |
| Suldge Volume Index (SVI) | *Tools*: Imhoff cones<br>*Method*: 2710D Standard Methods for the Examination of Water and Wastewater |

## C. Phase 1 tests

To characterise the wastewater type, one of the items considered was to check how the manholes are connected. This was determined by the following parameters:

| | |
|---|---|
| Origin, destination and | *Tools*: hose connected to the municipal water network |

| | |
|---|---|
| water network | *Procedure*: Introduce water from the hose into one of the connection points and observe where the water appears at a different connection point. Continue the process until the final point is reached: rainwater tank, retention tank, wastewater treatment plant, or collector |
| Localization | *Tools*: metric measuring wheel<br>*Procedure*: measure the distance of each manhole centre with respect to 2 different plant references to determine the exact location of the manhole. |
| Dimensions | *Tools*: tape measure and meter laser pointer<br>*Procedure*: measure the manhole depth from ground level, including the depty of each of the ducts that go into the manhole, the manhole internal measurements and lid measurements. |

## D. Phase 3 tests

To check the solids, COD and ammonium decrease by PW solids separation, several tests have been performed at both the laboratory and industrial scale.

a) Laboratory scale

We used a centrifuge Digicen 20 with time limits between 1 – 90 minutes and speed limits between 400 – 9000 rpm (figure 8). Two tests were performed: one with fixed time and varying the centrifugation speed (test 1) and another with the speed fixed and varying the centrifugation time (test 2).

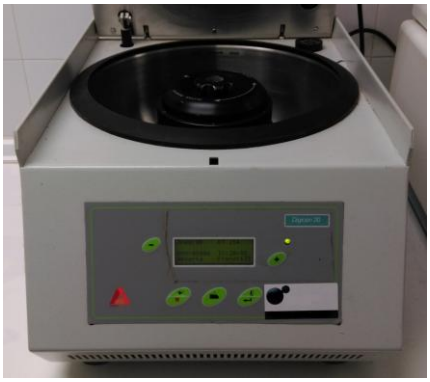


***Figure 8:*** *Centrifuge Digicen 20 (image author: Eloisa Albacete Garcia)*

**Test 1:** to determine the optimum centrifuge speed a fixed time of 20 minutes is set and the solids, COD and ammonium reduction were checked at speeds of 400, 700, 1000, 1250, 1500, 1750, 2000, 3000, 4000, 5000, 7000 and 9000 rpm.

**Test 2:** to determine the optimal centrifugation time a fixed speed of 9000 rpm is established (according to the results obtained in test 1) and the solids, COD and ammonia reduction were checked at times of 5, 10, 15, 20, 30, 45, 60 and 90 minutes.

b) Industrial scale

An Andritz centrifuge was used where various parameters can be regulated: speed (rpm), relative speed, sludge flow ($m^3$/h) and flocculant flow (L/h).

Different tests were performed by varying the centrifuge parameters and analysing the DM % in the liquid phase and the appearance of liquid and solid phases in order to determine the optimal values of the different parameters needed to obtain the lowest DM % value in the liquid phase while obtaining a good appearance in both the liquid and solid phases.

## E. Phase 4 tests

The purpose of these tests was to determine the optimal temperature and time to biodegrade PW COD in the thermal hydrolysis. For this reason, 3 tests were performed at different temperatures: 55, 65 and 75 ºC. In each test the solids, total and soluble COD and ammonium at different times were analysed to observe how they evolve as a function of time. The initial sample conditions were determined before subjecting it to the chosen temperature, and samples were analysed after reaching the test temperature (0 minutes) and after 30, 60, 90 and 120 minutes.

Figure 9 shows a picture of the assembly used for these tests. It consists of a cylindrical Pyrex reactor with a 5 litres capacity, introduced into a thermostatic bath.


***Figure 9:*** *Assembly for the thermal hydrolysis tests*

# PHASE 1. WASTEWATER CHARACTERISATION

## 1.1. Introduction

Annually, Ecoparc 2 consumes about 80,000 $m^3$ of municipal water and discharges approximately 50,000 $m^3$ to the collector that goes to the municipal wastewater treatment plant in Montcada i Reixac.

The waters produced at Ecoparc 2 have very different sources and completely different properties. Therefore, in the original Ecoparc 2 construction project, 4 different water networks were contemplated according to their origin where each networks goes to a different destination:

- **Black water (BW).** Is the water coming from the changing rooms, toilets and sanitary water in general. This water flows into the industrial estate collector.

- **Clean water (CW).** Arises from the rainwater that is collected from the building roofs and therefore have not been in contact with waste. This water goes to the rainwater pond, and it can be used in the processes, reducing the municipal water consumption.

- **Grey water (GW).** This is rainwater that have not been collected from the building roofs and, therefore, may have been in contact with waste and should be analysed. Water generated in the air treatment process (condensers, humidifiers and biofilters) is also included here. This effluent is collected in a retention tank, where it is checked to see if it meets the discharge limits. If so, it is sent to the collector, otherwise it goes to the wastewater treatment plant (WWTP) at Ecoparc 2.

- **Process water (PW).** This is water generated during the solid waste treatment and contains a high organic load and a high ammonium concentration. It needs to be treated in the wastewater treatment plant (WWTP) before being discharge to the collector.

Figure 1.1 shows a schematic diagram of the water system at Ecoparc 2 prior to starting the thesis.

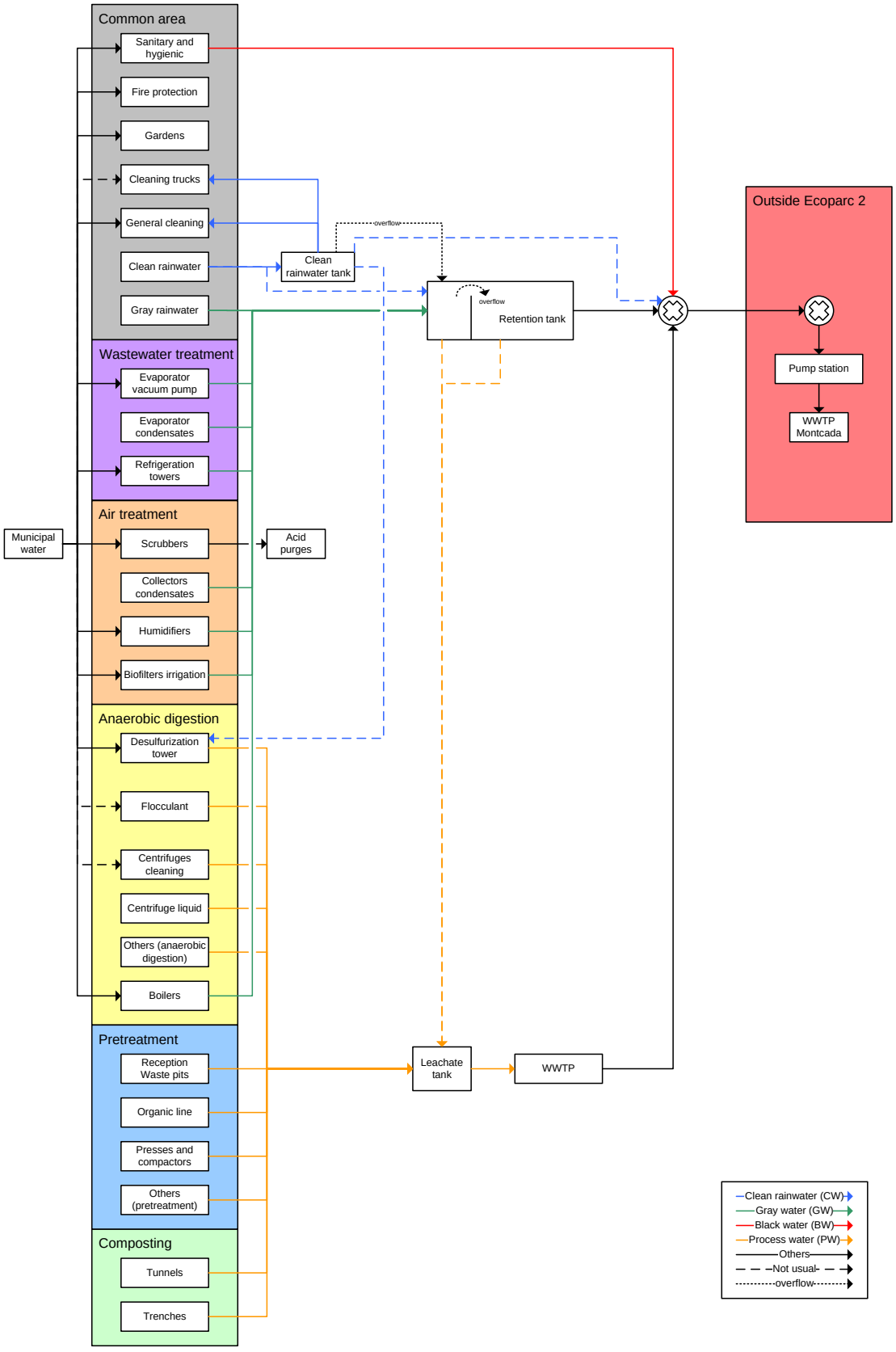



***Figure 1.1:*** *Water system at Ecoparc 2 before starting the thesis project*

The sanitary or black water (BW) from toilets and others sanitary facilities of the Ecoparc 2 installations is given in red, and goes directly to the discharge connection point into the collector (external water system).

The clean rainwater (CW) is given in blue and goes to the rainwater tank and is used for the general cleaning of the plant or for cleaning the trucks. Eventually, it can also be used in the desulphurisation tower. There is also the possibility to send this effluent to the retention tank or the discharge connection point if necessary.

The grey water (GW) is given in green, and goes to the first phase of the retention tank where it is analysed to be able to determine if it can be discharged or needs to be treated in the WWTP already mentioned.

The process water (PW) is given in yellow, and goes to the PW tank to be treated in the WWTP. Subsequently, the permeate obtained in the WWTP is discharged to collector.

The water from the collector is sent to a pumping station that collects wastewater from all of the companies in the area and pumps them to the WWTP at Montcada i Reixac where they are treated as urban wastewater.

During the thesis project, and in particular during the phase 1 tests, it was observed that the plant layout did not coincide exactly with the observations on the ground as far as water pipelines and connection points with manholes were concerned. Therefore, during this phase, a detailed analysis was carried out of the connections of the water system at Ecoparc 2.

## 1.2. Classification of effluents, connection points, water meters, and flows

### *1.2.1. Effluents*

It was determined if every possible effluents were currently operating or not. In table 1.1 all these effluents are listed, whether consumption (input effluents (IE)) or production (output effluents (OE)), depending on the area of origin, the water network is indicated to which they belong (municipal water (MW), clean water (CW), grey water (GW), process water (PW), black water (BW), or collector (C)), and their code.

***Table 1.1:*** *Ecoparc 2 effluents*

| area | | description | network | | | | | | code | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | MW | CW | GW | PW | BW | C | IE | OE |
| 01 | Staff | | | | | | | | | |
| 02 | Reception | drain trucks to WWTP | | | | x | | | | OE01 |
| | | drain trucks to evaporator | | | | x | | | | OE02 |
| | | reception waste pit to WWTP | | | | x | | | | OE03 |
| | | reception waste pit to evaporator | | | | x | | | | OE04 |
| 03 | Pretreatment | sanitary | x | | | | | | IE01 | |
| | | cleaning | x | | | | | | IE02 | |
| | | facilities roofs rainwater to CW pond | | x | | | | | | OE05 |
| | | sanitary to collector | | | | | x | | | OE06 |
| | | cleaning to retention tank | | | x | | | | | OE07 |
| | | cleaning to WWTP | | | | x | | | | OE08 |
| | | cleaning to evaporator | | | | x | | | | OE09 |
| | | line MWOF to WWTP | | | | x | | | | OE10 |
| | | line MWOF to evaporator | | | | x | | | | OE11 |
| | | presses and compactors to WWTP | | | | x | | | | OE12 |
| | | presses and compactors to evaporator | | | | x | | | | OE13 |
| 04 | Anaerobic digestion | sanitary | x | | | | | | IE03 | |
| | | cleaning | x | | | | | | IE04 | |
| | | | | x | | | | | IE05 | |
| | | | | | x | | | | IE06 | |
| | | washing centrifuges | x | | | | | | IE07 | |
| | | | | x | | | | | IE08 | |
| | | | | | x | | | | IE09 | |
| | | flocculant | x | | | | | | IE10 | |
| | | | | x | | | | | IE11 | |
| | | | | | x | | | | IE12 | |
| | | desulfurization tower | x | | | | | | IE13 | |
| | | | | x | | | | | IE14 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE14 |
| | | sanitary to collector | | | | | x | | | OE15 |
| | | cleaning to retention tank | | | x | | | | | OE16 |
| | | cleaning to WWTP | | | | x | | | | OE17 |
| | | washing centrifuges to WWTP | | | | x | | | | OE18 |
| | | centrifuge 1 liquid to WWTP | | | | x | | | | OE19 |
| | | centrifuge 2 liquid (digester) to WWTP | | | | x | | | | OE20 |
| | | centrifuge 3 liquid (purge) to retention tank | | | x | | | | | OE21 |
| 05 | Cogeneration | motors | x | | | | | | IE15 | |
| | | boiler | x | | | | | | IE16 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE22 |
| | | boiler to retention tank | | | x | | | | | OE23 |
| 06 | Tunnels | watering | x | | | | | | IE17 | |
| | | | | x | | | | | IE18 | |
| | | | | | x | | | | IE19 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE24 |
| | | PW zone 1 to WWTP | | | | x | | | | OE25 |
| | | PW zone 1 to evaporator | | | | x | | | | OE26 |
| | | PW zone 2 to WWTP | | | | x | | | | OE27 |
| | | PW zone 2 to evaporator | | | | x | | | | OE28 |
| 07 | Refining | sanitary | x | | | | | | IE20 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE29 |
| | | sanitary to collector | | | | | x | | | OE30 |
| 08 | Wastewater treatment plant | sanitary | x | | | | | | IE21 | |
| | | cleaning | x | | | | | | IE22 | |
| | | | | x | | | | | IE23 | |
| | | refrigeration tower 1 | x | | | | | | IE24 | |
| | | | | x | | | | | IE25 | |
| | | refrigeration tower 2 | x | | | | | | IE26 | |
| | | | | x | | | | | IE27 | |
| | | refrigeration tower 3 | x | | | | | | IE28 | |
| | | | | x | | | | | IE29 | |
| | | vacuum pump | x | | | | | | IE30 | |
| | | | | x | | | | | IE31 | |
| | | washing evaporator | x | | | | | | IE32 | |
| | | | | x | | | | | IE33 | |
| | | wash tank | x | | | | | | IE34 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE31 |
| | | sanitary to collector | | | | | x | | | OE32 |
| | | cleaning to WWTP | | | | x | | | | OE33 |
| | | cleaning to evaporator | | | | x | | | | OE34 |
| | | refrigeration tower 1 to CW tank | | x | | | | | | OE35 |
| | | refrigeration tower 1 to retention tank | | | x | | | | | OE36 |
| | | refrigeration tower 2 to CW tank | | x | | | | | | OE37 |
| | | refrigeration tower 2 to retention tank | | | x | | | | | OE38 |
| | | refrigeration tower 3 to CW tank | | x | | | | | | OE39 |

| area | | description | network | | | | | | code | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | MW | CW | GW | PW | BW | C | IE | OE |
| 08 | Wastewater treatment plant | refrigeration tower 3 to retention tank | | | x | | | | | OE40 |
| | | vacuum pump to CW tank | | x | | | | | | OE41 |
| | | vacuum pump to retention tank | | | x | | | | | OE42 |
| | | evaporator condensate to retention tank | | | x | | | | | OE43 |
| | | washing evaporator to WWTP | | | | x | | | | OE44 |
| | | washing evaporator to evaporator | | | | x | | | | OE45 |
| | | permeate to recirculation | | | | x | | | | OE46 |
| | | permeate to collector | | | | | | x | | OE47 |
| 09 | Air treatment | washers | x | | | | | | IE35 | |
| | | scrubber | x | | | | | | IE36 | |
| | | humidifier 1 | x | | | | | | IE37 | |
| | | humidifier 2 | x | | | | | | IE38 | |
| | | humidifier 3 | x | | | | | | IE39 | |
| | | humidifier 4 | x | | | | | | IE40 | |
| | | product dosage | x | | | | | | IE41 | |
| | | biofilter 1 | x | | | | | | IE42 | |
| | | biofilter 2 | x | | | | | | IE43 | |
| | | biofilter 3 | x | | | | | | IE44 | |
| | | biofilter 4 | x | | | | | | IE45 | |
| | | biofilter 5A | x | | | | | | IE46 | |
| | | biofilter 5B | x | | | | | | IE47 | |
| | | biofilter 6A | x | | | | | | IE48 | |
| | | biofilter 6B | x | | | | | | IE49 | |
| | | collectors condensate to GW pit | | | x | | | | | OE48 |
| | | humidifier 1 to GW pit | | | x | | | | | OE49 |
| | | humidifier 2 to GW pit | | | x | | | | | OE50 |
| | | humidifier 3 to GW pit | | | x | | | | | OE51 |
| | | humidifier 4 to GW pit | | | x | | | | | OE52 |
| | | biofilter 1 to GW pit | | | x | | | | | OE53 |
| | | biofilter 2 to GW pit | | | x | | | | | OE54 |
| | | biofilter 3 to GW pit | | | x | | | | | OE55 |
| | | biofilter 4 to GW pit | | | x | | | | | OE56 |
| | | biofilter 5A to GW pit | | | x | | | | | OE57 |
| | | biofilter 5B to GW pit | | | x | | | | | OE58 |
| | | biofilter 6A to GW pit | | | x | | | | | OE59 |
| | | biofilter 6B to GW pit | | | x | | | | | OE60 |
| 11 | Fire extinction | fire extinction tank | x | | | | | | IE50 | |
| | | cleaning to GW pit | | | x | | | | | OE61 |
| | | cleaning to retention tank | | | x | | | | | OE62 |
| | | cleaning to WWTP | | | | x | | | | OE63 |
| | | cleaning to evaporator | | | | x | | | | OE64 |
| 12 | Trenches | watering | x | | | | | | IE51 | |
| | | plenum | x | | | | | | IE52 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE65 |
| | | PW to WWTP | | | | x | | | | OE66 |
| | | PW to evaporator | | | | x | | | | OE67 |
| | | plenum to WWTP | | | | x | | | | OE68 |
| | | plenum to evaporator | | | | x | | | | OE69 |
| 15 | Mobile machinery | | | | | | | | | |
| 99.1 | Waters | grey rainwater to GW pit | | | x | | | | | OE70 |
| | | grey rainwater well overflow to retention tank | | | x | | | | | OE71 |
| | | grey rainwater to retention tank | | | x | | | | | OE72 |
| | | wall to WWTP | | | | x | | | | OE73 |
| | | wall to collector | | | | | | x | | OE74 |
| 99.2 | Administration, visits, and changing rooms | sanitary | x | | | | | | IE53 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE75 |
| | | sanitary to collector | | | | | x | | | OE76 |
| 99.3 | Workshop | sanitary | x | | | | | | IE54 | |
| | | facilities roofs rainwater to CW tank | | x | | | | | | OE77 |
| | | sanitary to collector | | | | | x | | | OE78 |
| 99.4 | Laboratory | sanitary | x | | | | | | IE55 | |
| | | sanitary to collector | | | | | x | | | OE79 |
| 99.5 | Road outside lighting | | | | | | | | | |
| 99.6 | Watering gardens | watering zone 1 | x | | | | | | IE56 | |
| | | watering zone 2 | x | | | | | | IE57 | |
| 99.7 | Road outside | cleaning | x | | | | | | IE58 | |
| | | cleaning to GW pit | | | x | | | | | OE80 |
| | | cleaning to retention tank | | | x | | | | | OE81 |
| | | cleaning to WWTP | | | | x | | | | OE82 |
| | | cleaning to evaporator | | | | x | | | | OE83 |
| 99.8 | Fueling station | | | | | | | | | |
| 99.9 | Common electrical installations | | | | | | | | | |
| 99.10 | Doors | | | | | | | | | |

### *1.2.2. Connection points (CP)*

At Ecoparc 2 there are different connection points with manholes that carry different output water types. After 13 years of operation and after several modifications, it has been observed that the plant process drawings do not reflect what is actually observed on the ground. This is because some of the manholes may not be localized correctly or are incorrectly placed and are not well characterized with respect to their origin and destination.

As a result, a review of the wastewater connections was needed to update the process drawings with regards to the observations from the plant.

To update the process drawings to the reality of the entire Ecoparc 2 surface the following parameters of each manhole were determined: origin, destination and water network, localisation and dimensions.

The tubes that carry rainwater from the facility roofs, and the grids that collect the vials waters are also contemplated.

### *1.2.3. Water meters (WM)*

At Ecoparc 2, there were already several water meters to control water consumption and some discharges. Nevertheless, during this study it has been possible to install several more water meters so that control more water flows and observe key points which had not been covered so far.

Figures 1.2 and 1.3 show the water meters that are installed for the monitoring of consumption and discharge respectively. In green appears the water meters that were already installed and work correctly. In yellow appears the water meters that were already installed but did not work, and it was necessary to repair or replace them with new ones. Finally, in red appears the new water meters that were installed as part of this thesis with particular regards to key points.

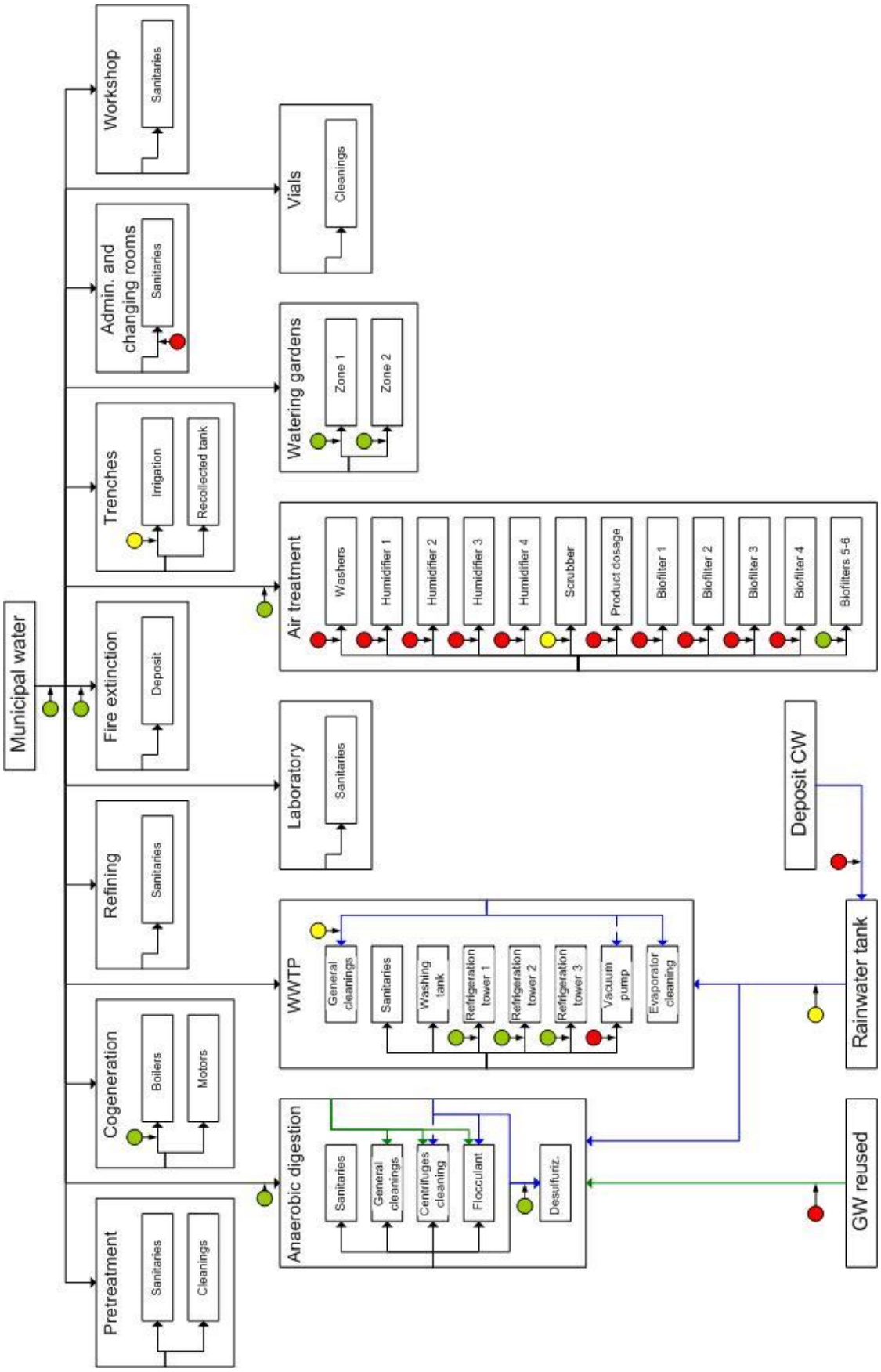



***Figure 1.2:*** *Water meters for consumption control*

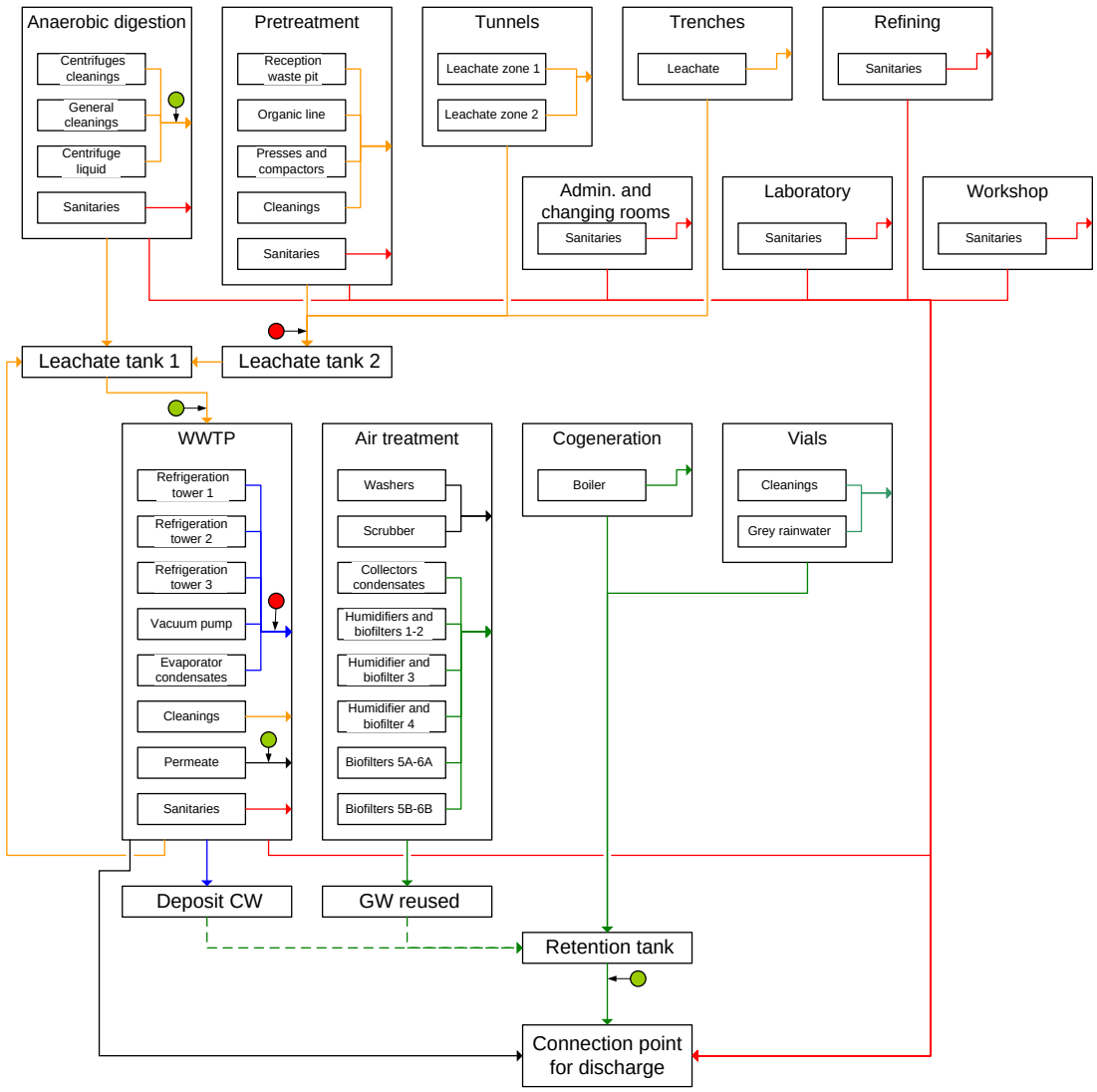



***Figure 1.3:*** *Water meters for discharge control*

In table 1.2 are listed all the water meters that currently available in Ecoparc 2 by areas, and the code that has been given to each one.

***Table 1.2:*** *Water meters code*

| area | | description | code |
|---|---|---|---|
| 01 | Staff | | |
| 02 | Reception | | |
| 03 | Pretreatment | | |
| 04 | Anaerobic digestion | methanation general | WM01 |
| | | desulfurization tower | WM02 |
| | | liquid tank (centrifuges + cleanings) | WM03 |
| 05 | Cogeneration | boiler | WM04 |
| 06 | Tunnels | | |
| 07 | Refining | | |
| 08 | Wastewater treatment plant | wastewater treatment plant general | WM05 |
| | | refrigeration tower 1 | WM06 |
| | | refrigeration tower 2 | WM07 |
| | | refrigeration tower 3 | WM08 |
| | | vacuum pump | WM09 |
| | | refrigeration towers + vacuum pump to CW pond | WM10 |

| area | | description | code |
|---|---|---|---|
| 08 | Wastewater treatment plant | input PW to WWTP (filters after) | WM11 |
| | | total permeate (to recirculation and to collector) | WM12 |
| | | permeate to collector | WM13 |
| 09 | Air treatment | air treatment general | WM14 |
| | | air treatment washers | WM15 |
| | | scrubber | WM16 |
| | | humidifier 1 | WM17 |
| | | humidifier 2 | WM18 |
| | | humidifier 3 | WM19 |
| | | humidifier 4 | WM20 |
| | | product dosage | WM21 |
| | | biofilter 1 | WM22 |
| | | biofilter 2 | WM23 |
| | | biofilter 3 | WM24 |
| | | biofilter 4 | WM25 |
| | | biofilter 5A + biofilter 6A | WM26 |
| 11 | Fire extinction | fire extinction | WM27 |
| 12 | Trenches | | |
| 15 | Mobile machinery | | |
| 99.1 | Waters | Ecoparc 2 general | WM28 |
| | | rainwater pond | WM29 |
| | | double connection point | WM30 |
| | | GW reused | WM31 |
| | | GW discharge to collector | WM32 |
| | | total discharge to collector | WM33 |
| 99.2 | Administration, visits, and changing rooms | changing rooms | WM34 |
| 99.3 | Workshop | | |
| 99.4 | Laboratory | | |
| 99.5 | Road outside lighting | | |
| 99.6 | Watering gardens | watering zone 1 | WM35 |
| | | watering zone 2 | WM36 |
| 99.7 | Road outside | | |
| 99.8 | Fueling station | | |
| 99.9 | Common electrical installations | | |
| 99.10 | Doors | | |

These water meters include one or more effluents that have already been listed in table 1.1. In table 1.3 are listed all the water meters available to Ecoparc 2, with respect to the effluents.

***Table 1.3:*** *Relationship between effluents and water meters*

| | WM01 | WM02 | WM03 | WM04 | WM05 | WM06 | WM07 | WM08 | WM09 | WM10 | WM11 | WM12 | WM13 | WM14 | WM15 | WM16 | WM17 | WM18 | WM19 | WM20 | WM21 | WM22 | WM23 | WM24 | WM25 | WM26 | WM27 | WM28 | WM29 | WM30 | WM31 | WM32 | WM33 | WM34 | WM35 | WM36 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| IE01 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE02 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE03 | x | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE04 | x | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE05 | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE06 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| IE07 | x | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE08 | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE09 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| IE10 | x | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE11 | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE12 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| IE13 | x | x | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE14 | | x | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE15 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE16 | | | | x | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE17 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE18 | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE19 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| IE20 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE21 | | | | | x | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE22 | | | | | x | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE23 | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE24 | | | | | x | x | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE25 | | | | | | x | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE26 | | | | | x | | x | | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE27 | | | | | | | x | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE28 | | | | | x | | | x | | | | | | | | | | | | | | | | | | | x | | | | | | | | | |
| IE29 | | | | | | | | x | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE30 | | | | | x | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | | | |

| | WM01 | WM02 | WM03 | WM04 | WM05 | WM06 | WM07 | WM08 | WM09 | WM10 | WM11 | WM12 | WM13 | WM14 | WM15 | WM16 | WM17 | WM18 | WM19 | WM20 | WM21 | WM22 | WM23 | WM24 | WM25 | WM26 | WM27 | WM28 | WM29 | WM30 | WM31 | WM32 | WM33 | WM34 | WM35 | WM36 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| IE31 | | | | | | | | | x | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE32 | | | | | x | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE33 | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE34 | | | | | x | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE35 | | | | | | | | | | | | | | x | x | | | | | | | | | | | | x | | | | | | | | |
| IE36 | | | | | | | | | | | | | | x | | x | | | | | | | | | | | x | | | | | | | | |
| IE37 | | | | | | | | | | | | | | x | | | x | | | | | | | | | | x | | | | | | | | |
| IE38 | | | | | | | | | | | | | | x | | | | x | | | | | | | | | x | | | | | | | | |
| IE39 | | | | | | | | | | | | | | x | | | | | x | | | | | | | | x | | | | | | | | |
| IE40 | | | | | | | | | | | | | | x | | | | | | x | | | | | | | x | | | | | | | | |
| IE41 | | | | | | | | | | | | | | x | | | | | | | x | | | | | | x | | | | | | | | |
| IE42 | | | | | | | | | | | | | | x | | | | | | | | x | | | | | x | | | | | | | | |
| IE43 | | | | | | | | | | | | | | x | | | | | | | | | x | | | | x | | | | | | | | |
| IE44 | | | | | | | | | | | | | | x | | | | | | | | | | x | | | x | | | | | | | | |
| IE45 | | | | | | | | | | | | | | x | | | | | | | | | | | x | | x | | | | | | | | |
| IE46 | | | | | | | | | | | | | | x | | | | | | | | | | | | x | x | | | | | | | | |
| IE47 | | | | | | | | | | | | | | x | | | | | | | | | | | | | x | | | | | | | | |
| IE48 | | | | | | | | | | | | | | x | | | | | | | | | | | | x | x | | | | | | | | |
| IE49 | | | | | | | | | | | | | | x | | | | | | | | | | | | | x | | | | | | | | |
| IE50 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE51 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE52 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE53 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | x | | | |
| IE54 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE55 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| IE56 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | x | | |
| IE57 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | x | |
| IE58 | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | | |
| OE01 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE02 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE03 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE04 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE05 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE06 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | x | | | | |
| OE07 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE08 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE09 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE10 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE11 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE12 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE13 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE14 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | |
| OE16 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE17 | | | x | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE18 | | | x | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE19 | | | x | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE20 | | | x | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE21 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE22 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE23 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE24 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE25 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE26 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE27 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE28 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE29 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE30 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | |
| OE31 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE32 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | |
| OE33 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE34 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE35 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE36 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE37 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE38 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE39 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE40 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE41 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE42 | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE43 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE44 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE45 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE46 | | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | | | | | | | |
| OE47 | | | | | | | | | | | | x | x | | | | | | | | | | | | | | | | | | | x | | | | |
| OE48 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE49 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE50 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE51 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE52 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE53 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE54 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE55 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE56 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE57 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE58 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE59 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE60 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE61 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

| | WM01 | WM02 | WM03 | WM04 | WM05 | WM06 | WM07 | WM08 | WM09 | WM10 | WM11 | WM12 | WM13 | WM14 | WM15 | WM16 | WM17 | WM18 | WM19 | WM20 | WM21 | WM22 | WM23 | WM24 | WM25 | WM26 | WM27 | WM28 | WM29 | WM30 | WM31 | WM32 | WM33 | WM34 | WM35 | WM36 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| OE62 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE63 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE64 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE65 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE66 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE67 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE68 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE69 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE70 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE71 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE72 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE73 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE74 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | x | | | | |
| OE75 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE76 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | |
| OE77 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE78 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | |
| OE79 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | |
| OE80 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE81 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| OE82 | | | | | | | | | | | x | | | | | | | | | | | | | | | | | | x | | | | | | | |
| OE83 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | x | | | | | | | |

### *1.2.4. Flows*

From the water meters we can determine (directly or indirectly) several important flow rates for the monitoring and control of the water consumption (inflow (IF)) and production (outflow (OF)). Table 1.4 gives the different flow rates as a function of their area, location, water network, and equation used to calculate their values.

***Table 1.4:*** *Flow rates calculations*

| area | | description | network | | | | | | code | | equation (mm=month ; yy=year) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | MW | CW | GW | PW | BW | C | IF | OF | |
| 01 | Staff | | | | | | | | | | |
| 02 | Reception | | | | | | | | | | |
| 03 | Pretreatment | | | | | | | | | | |
| 04 | Anaerobic digestion | MW anaerobic digestion general | x | | | | | | IF01 | | $WM01_{mm/yy}$ - $WM01_{(mm/yy-1)}$ |
| | | desulfurization tower | x | x | | | | | IF02 | | $WM02_{mm/yy}$ - $WM02_{(mm/yy-1)}$ |
| | | liquid tank (centrifuges + cleanings) | | | | x | | | | OF01 | $WM03_{mm/yy}$ - $WM03_{(mm/yy-1)}$ |
| 05 | Cogeneration | MW cogeneration general (boiler descalcifier) | x | | | | | | IF03 | | $WM04_{mm/yy}$ - $WM04_{(mm/yy-1)}$ |
| 06 | Tunnels | | | | | | | | | | |
| 07 | Refining | | | | | | | | | | |
| 08 | Wastewater treatment plant | MW WWTP general | x | | | | | | IF04 | | $WM05_{mm/yy}$ - $WM05_{(mm/yy-1)}$ |
| | | refrigeration tower 1 | x | x | | | | | IF05 | | $WM06_{mm/yy}$ - $WM06_{(mm/yy-1)}$ |
| | | refrigeration tower 2 | x | x | | | | | IF06 | | $WM07_{mm/yy}$ - $WM07_{(mm/yy-1)}$ |
| | | refrigeration tower 3 | x | x | | | | | IF07 | | $WM08_{mm/yy}$ - $WM08_{(mm/yy-1)}$ |
| | | refrigeration towers (1 to 3) | x | x | | | | | IF08 | | $IF05_{mm/yy}$ + $IF06_{mm/yy}$ + $IF07_{mm/yy}$ |
| | | vacuum pump | x | x | | | | | IF09 | | $WM09_{mm/yy}$ - $WM09_{(mm/yy-1)}$ |
| | | refrigeration towers + vacuum pump | | x | x | | | | | OF02 | $WM10_{mm/yy}$ - $WM10_{(mm/yy-1)}$ |
| | | input PW to WWTP (filters after) | | | | x | | | | OF03 | $WM11_{mm/yy}$ - $WM11_{(mm/yy-1)}$ |
| | | total input to WWTP (liquid tank 04AREA + double CP) | | | | x | | | | OF04 | $OF01_{mm/yy}$ + $OF07_{mm/yy}$ |
| | | total permeate (to recirculation and to collector) | | | | x | | x | | OF05 | $WM12_{mm/yy}$ - $WM12_{(mm/yy-1)}$ |
| | | permeate discharge to collector | | | | | | x | | OF06 | $WM13_{mm/yy}$ - $WM13_{(mm/yy-1)}$ |
| 09 | Air treatment | MW air treatment general | x | | | | | | IF10 | | $WM14_{mm/yy}$ - $WM14_{(mm/yy-1)}$ |
| | | washers | x | | | | | | IF11 | | $WM15_{mm/yy}$ - $WM15_{(mm/yy-1)}$ |
| | | scrubber | x | | | | | | IF12 | | $WM16_{mm/yy}$ - $WM16_{(mm/yy-1)}$ |
| | | humidifier 1 | x | | | | | | IF13 | | $WM17_{mm/yy}$ - $WM17_{(mm/yy-1)}$ |
| | | humidifier 2 | x | | | | | | IF14 | | $WM18_{mm/yy}$ - $WM18_{(mm/yy-1)}$ |
| | | humidifier 3 | x | | | | | | IF15 | | $WM19_{mm/yy}$ – $WM19_{(mm/yy-1)}$ |
| | | humidifier 4 | x | | | | | | IF16 | | $WM20_{mm/yy}$ - $WM20_{(mm/yy-1)}$ |
| | | humidifiers (1 to 4) | x | | | | | | IF17 | | $IF13_{mm/yy}$ + $IF14_{mm/yy}$ + $IF15_{mm/yy}$ + $IF16_{mm/yy}$ |

| area | | description | network | | | | | code | | equation (mm=month ; yy=year) |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | x | | | | | IF20 | | |
| 09 | Air treatment | product dosage | x | | | | | IF18 | | $WM21_{mm/yy}$ - $WM21_{(mm/yy-1)}$ |
| | | biofilter 1 | x | | | | | IF20 | | $WM22_{mm/yy}$ - $WM22_{(mm/yy-1)}$ |
| | | biofilter 2 | x | | | | | IF21 | | $WM23_{mm/yy}$ - $WM23_{(mm/yy-1)}$ |
| | | biofilter 3 | | | | | | | | $WM24_{mm/yy}$ - $WM24_{(mm/yy-1)}$ |
| | | biofilter 4 | x | | | | | IF22 | | $WM25_{mm/yy}$ - $WM25_{(mm/yy-1)}$ |
| | | biofilters (1 to 4) | x | | | | | IF23 | | $IF19_{mm/yy}$ + $IF20_{mm/yy}$ + $IF21_{mm/yy}$ + $IF22_{mm/yy}$ |
| | | biofilter 5A + biofilter 6A | x | | | | | IF24 | | $WM26_{mm/yy}$ - $WM26_{(mm/yy-1)}$ |
| 11 | Fire extinction | MW fire extinction general | x | | | | | IF25 | | $WM27_{mm/yy}$ - $WM27_{(mm/yy-1)}$ |
| 12 | Trenches | | | | | | | | | |
| 15 | Mobile machinery | | | | | | | | | |
| 99.1 | Waters | MW Ecoparc 2 general | x | | | | | IF26 | | $WM28_{mm/yy}$ – $WM28_{(mm/yy-1)}$ |
| | | rainwater pond (CW) | | x | | | | IF27 | | $WM29_{mm/yy}$ – $WM29_{(mm/yy-1)}$ |
| | | double connection point | | | x | | | | OF07 | $WM30_{mm/yy}$ - $WM30_{(mm/yy-1)}$ |
| | | GW reused | | | | x | | IF28 | | $WM31_{mm/yy}$ - $WM31_{(mm/yy-1)}$ |
| | | GW discharge to collector | | | | | x | | OF08 | $WM32_{mm/yy}$ - $WM32_{(mm/yy-1)}$ |
| | | BW discharge to collector | | | | | x | | OF09 | $OF10_{mm/yy}$ - $OF06_{mm/yy}$ - $OF08_{mm/yy}$ |
| | | total discharge to collector | | | | | x | | OF10 | $WM33_{mm/yy}$ - $WM33_{(mm/yy-1)}$ |
| 99.2 | Administration, visits, and changing rooms | MW changing rooms general | x | | | | | IF29 | | $WM34_{mm/yy}$ - $WM34_{(mm/yy-1)}$ |
| 99.3 | Workshop | | | | | | | | | |
| 99.4 | Laboratory | | | | | | | | | |
| 99.5 | Road outside lighting | | | | | | | | | |
| 99.6 | Watering gardens | MW watering gardens general | x | | | | | IF30 | | $IF31_{mm/yy}$ + $IF32_{mm/yy}$ |
| | | watering zone 1 | x | | | | | IF31 | | $WM35_{mm/yy}$ - $WM35_{(mm/yy-1)}$ |
| | | watering zone 2 | x | | | | | IF32 | | $WM36_{mm/yy}$ - $WM36_{(mm/yy-1)}$ |
| 99.7 | Road outside | | | | | | | | | |
| 99.8 | Fueling station | | | | | | | | | |
| 99.9 | Common electrical installations | | | | | | | | | |
| 99.10 | Doors | | | | | | | | | |

## 1.3. Results obtained

### *1.3.1. Grey water*

In September 2013, we decided to perform a thorough study of the grey water to propose changes that could be expected to improve the situation at that time.

Four tests were carried out:

- Test 1: Grey water global study
- Test 2: Grey water partial study
- Test 3: Grey water manhole tracking
- Test 4: Ammonia nitrogen reduction by stripping

Table 1.5 summarizes the observations, results discussion, conclusions and improvement proposals of the results obtained in each test.

***Table 1.5:*** *Grey water air treatment study observations, results and discussion*

| | Observations | Results discussion | Conclusions | Improvement proposals |
|---|---|---|---|---|
| **Test 1** | The 35% of MW consumption in WWTP comes from refrigeration towers.<br>COD and ammonia concentrations in the GW produced in the WWTP are very low.<br>Air treatment area has a high MW consumption.<br>GW production was about 100 $m^3$/day, and approximately the 26% of GW come from air treatment.<br>Air treatment ammonia nitrogen and COD concentration is more than twice the concentration in the last point before discharge. | The 26% of GW come from air treatment, which have a higher ammonium and COD concentration, it gets diluted with GW from WWTP area (refrigeration towers, condensate evaporator, and vacuum pump). | The wastewater produced in the air treatment area represent the 26% of GW that are discharged, but are most concentrated in ammonium and COD. | Install a tank to collect the GW from refrigeration towers and vacuum pump to go to rainwater pond or wall if it is necessary.<br>Study in greater depth the air treatment area to minimize the GW production and improve water dilution is going to collector.<br>Install a holding tank below the wall which increase the residence time before discharge to collector by such smoothing the peaks based on the effluents state. |
| **Test 2** | The discharged water through the water spray in the humidifiers is 7,8 $m^3$/day.<br>GW due to biofilters irrigation is approximately 4,3 $m^3$/day. | The humidifiers and biofilters 1, 2 and 3 produced 3 to 5 times more wastewater than the biofilters rest. | Of the GW 26.4 m3/day from air treatment area, 30% is directly related to the water spray humidifiers and 16% to biofilters irrigation. | Act humidifiers drift eliminators that may be affected, so decreasing the water entrained amount by the air.<br>Regulate the periodicities and irrigation times to minimize the wastewater. So the ideal humidity biological bed is maintained, minimizing the water consumption and its discharge. |
| **Test 3** | The COD and ammonia concentration in the first manholes (air treatment area) is very high and decreases as they move the connection points (be mixed with GW from other sources). | Ammonium high concentration that comes of air treatment area that decreases as mixed with other effluents, especially those generated in WWTP. | The 26% from GW containing the 55% of ammonium discharge. | Install a tank that collects water coming from air treatment area for reuse for flocculant preparation, centrifuges washing and other equipment, so as to minimize the MW consumption and end in the WWTP as PW. |
| **Test 4** | The COD and ammonia concentration, to laboratory level and standard conditions, does not vary depending on the stripping time. | To observe significant difference in reducing ammonium concentration must be higher concentrations or by changing the temperature and pH. | Although beyond the ammonium discharge limits, do not contain sufficient concentration to reduce its by stripping. | Not interested in testing altering the pH and temperature. |

### *1.3.2. Process water*

In December 2013 we started a study about the process water. Unfortunately the connection points were found to flooded and an external company was required to clean them. As a consequence the samples collected are not representative.

In January 2014 we collected samples from each process water connection point and analysed them for COD and ammonium concentrations. Figure 1.4 shows the location of each connection point that was analysed and the results obtained are shown in table 1.6.


***Figure 1.4:*** *Locations of the analysed process water connection points*

***Table 1.6:*** *Results of the analysed process water connection points*

| Connection point | COD (mg $O_2$/L) | Ammonium (mg N/L) |
|---|---|---|
| 1 | 35.000 | 3.050 |
| 2 | 25.500 | 2.350 |
| 3 | >75.000 | 4.350 |
| 4 | 58.500 | 4.450 |
| 5 | 70.500 | 5.100 |
| 6 | 68.500 | 4.850 |
| 7 | 69.000 | 4.750 |
| 8 | 42.500 | 3.000 |

The first connection point collects the process water from the trenches area, which has a high organic load. The second connection point has a lower concentration are contains mixed effluents coming from the pretreatment area of the cleanings zone. In the third connection point the organic load reaches the maximum value and it is clear that it has been mixed with the PW produced in the tunnels area, which contain a high COD and ammonium concentration. In the fourth connection point it is mixed with additional pretreatment effluents that decrease the COD concentration, however the ammonium concentration remains unchanged. In the fifth connection point the concentrations increase again, since it is the where the mixture is returned with the PW from the discharge of the waste pits which have a high load. In the sixth connection point there are no significant differences since no new effluents haves been added. The same happens in the seventh connection point, where it has not been mixed with any new effluents, so the concentrations remain very similar. However, in the last connection point, where the PW of the whole company are collected with the exception of the anaerobic digestion, a decrease of both concentrations is observed, due to the dilution effect with PW at this point.

Since the PW flow rates are not continuous, the process water load is highly variable depending on what is considered or not as PW from trenches, tunnels and discharge waste areas.

### *1.3.3. Flow rates*

The flow rates of various effluents (MW/CW/GW consumption and wastewater production) can be determined from the existing and newly installed water meters. Since not all the water meters were installed at the same time, the results are represented in the periods that it was possible to quantify.

#### 1.3.3.1. MW/CW consumption: Global evaluation

Figure 1.5 shows the total MW consumption at Ecoparc 2 since January 2007 to May 2017.

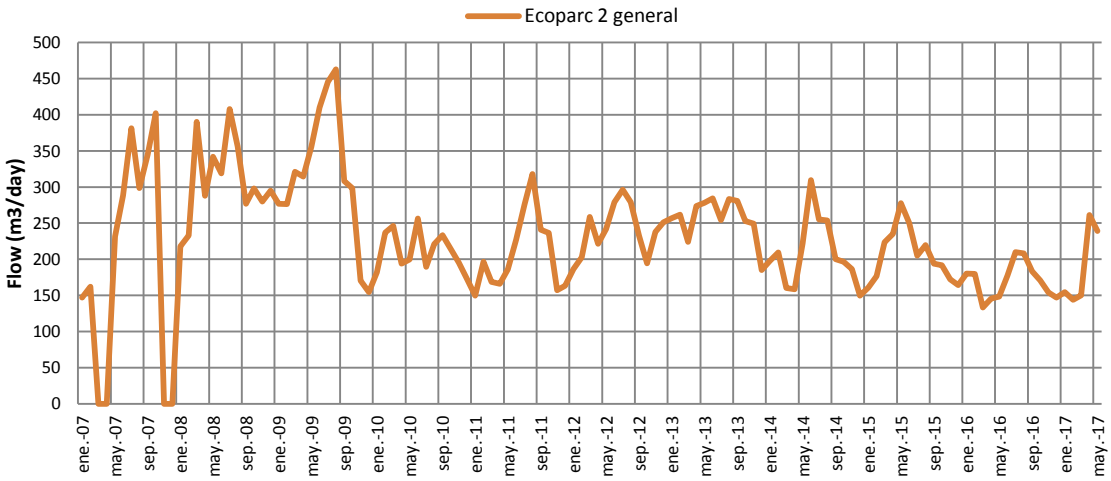



***Figure 1.5:*** *MW consumption at Ecoparc 2 since January 2007 to May 2017*

The general MW consumption at Ecoparc 2 peaked in the middle of 2009. Since then, it has dropped significantly, considering the normal seasonal fluctuations (higher consumption in the warmer months and lower consumption in the colder months). Although the processes have changed significantly in recent years, it has managed to maintain or even reduce the MW consumption.

To make a more specific evaluation, the consumption of each one of the areas in which it is possible to evaluate the flow rate is given. Figure 1.6 shows the data obtained.

The MW consumption with greater weight is included in the WWTP, air treatment and anaerobic digestion areas.

Much of the MW consumption in the WWTP is due to the refrigeration towers. However, throughout the study the WWTP treatment capacity has been increased without the use of the evaporator, thus dispensing with the MW consumption in this.

On the other hand, there is an increase in MW consumption in the air treatment area due to modifications made in the biofilters, in which the fluidized bed has been changed and has been controlled by an external company.

An increase in MW consumption is also observed in the anaerobic digestion area, due to tests that have been carried out with the flocculant. Better results have been

obtained with a greater flocculant dilution, separating more solids and improving the PW to be treated in the WWTP.

Regarding the CW consumption, its use has been optimised whenever possible, thus reducing MW consumption.

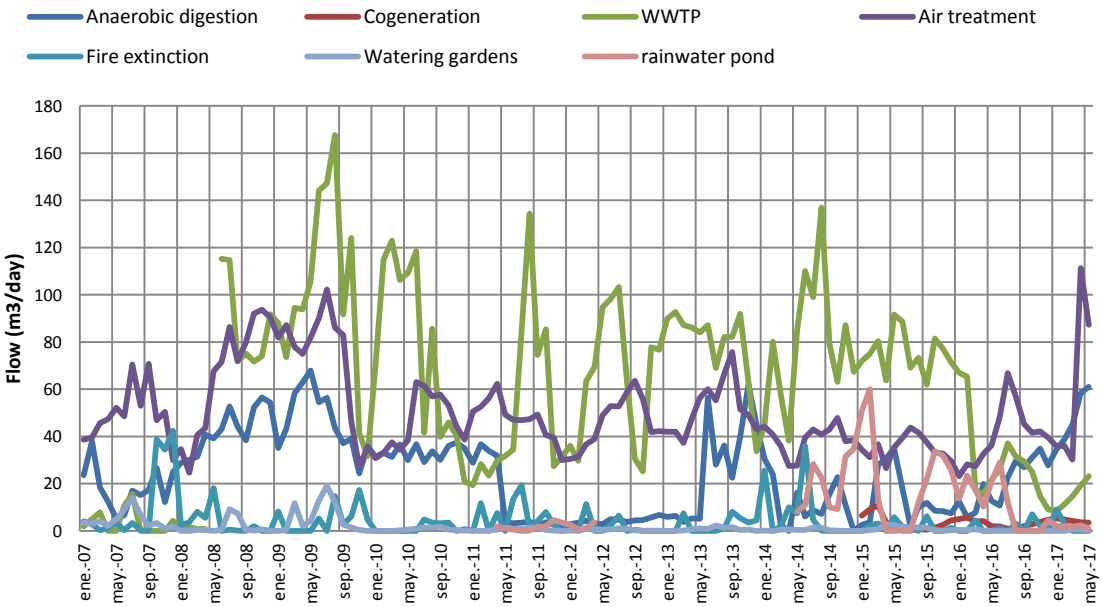



***Figure 1.6:*** *MW consumption in different areas of Ecoparc 2 from January 2007 to May 2017*

### 1.3.3.2. MW/CW consumption: Evaluation of the different areas

In this section, each area is studied separately, to be able to detect those points with greater consumption and to act on them if possible.

#### a. Anaerobic digestion and cogeneration

Figure 1.7 shows the MW consumptions in the anaerobic digestion and cogeneration areas and the MW or CW consumption of the desulphurisation tower.

Much of the MW consumption in anaerobic digestion is due to the desulphurisation tower. However, in recent months it has been observed that the consumption of mains water has increased significantly without significant differences in the desulphurisation tower. This is due to modifications in the process, in which different flocculants and dilutions have been tested, with which better results has been observed with greater dilution (this will be seen in more detail in phase 2 of the project).

However, the PW production in anaerobic digestion has remained in the same line, without appreciating a significant increase due to the greater MW consumption. Even so, it is true that the PW obtained in this area currently has better characteristics for treatment in the WWTP.

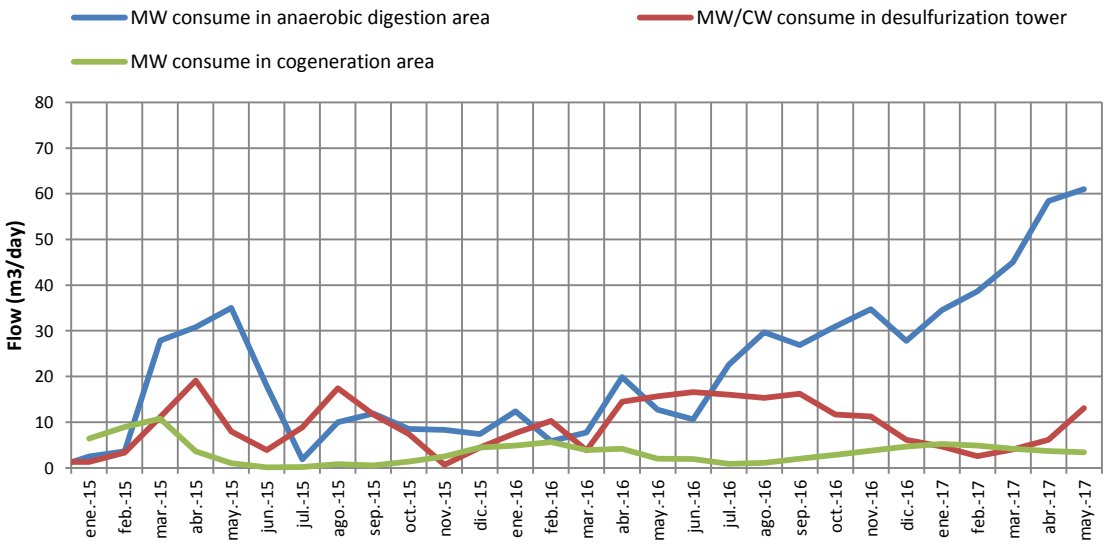



***Figure 1.7:*** *MW consumption in anaerobic digestion and cogeneration areas and MW/CW consumption in the desulphurisation tower*

b. Wastewater treatment plant

Figure 1.8 shows the MW consumption of refrigeration towers and the evaporator vacuum pump from the WWTP area.

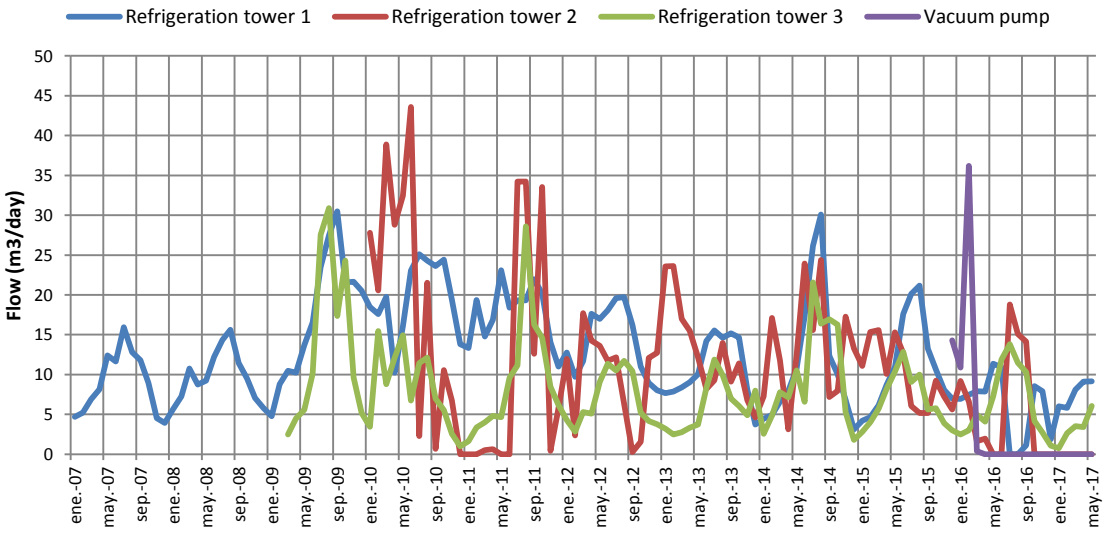



***Figure 1.8:*** *MW consumption of refrigeration towers and evaporator vacuum pump from WWTP area*

Initially, the WWTP was designed to treat a PW with an organic load of COD of 10,000 mg/L. However, throughout the operation it was observed that the PW arriving at the WWTP contained COD 40,000 mg/L, which inevitably lead to the expansion of the WWTP by installing a new nitrification reactor of 900 $m^3$ with its corresponding refrigeration tower. This new addition was made in 2009, which has since increased the MW consumption due to the new refrigeration tower.

The WWTP also has an evaporator to pretreat the PW, to ensure that the inlet to the biological treatment plant contains a lower load and facilitate the purification. In practice, the evaporator was used to treat part of the PW, thus reducing the flow to be treated in the biological treatment plant since it was working at full capacity (142 $m^3$/day). The evaporator is not always in operation, only in those cases where it is necessary to relieve the biological treatment plant. We only have data on the consumption of this refrigeration tower (number 2) since 2010.

In general terms, it can be observed that MW consumption increases in the hotter months and decreases in the colder ones. Even so, a decrease of consumption with the time in both nitrification reactors (towers 1 and 3) can be observed. Note the absence of the evaporator tower consumption (tower 2) and the vacuum pump, because at present it is not necessary to start the evaporator to increase the WWTP performance.

### c. Air treatment

Figure 1.9 shows the consumption of different air treatment equipment: washers, scrubber, product dosage, humidifiers and biofilters.

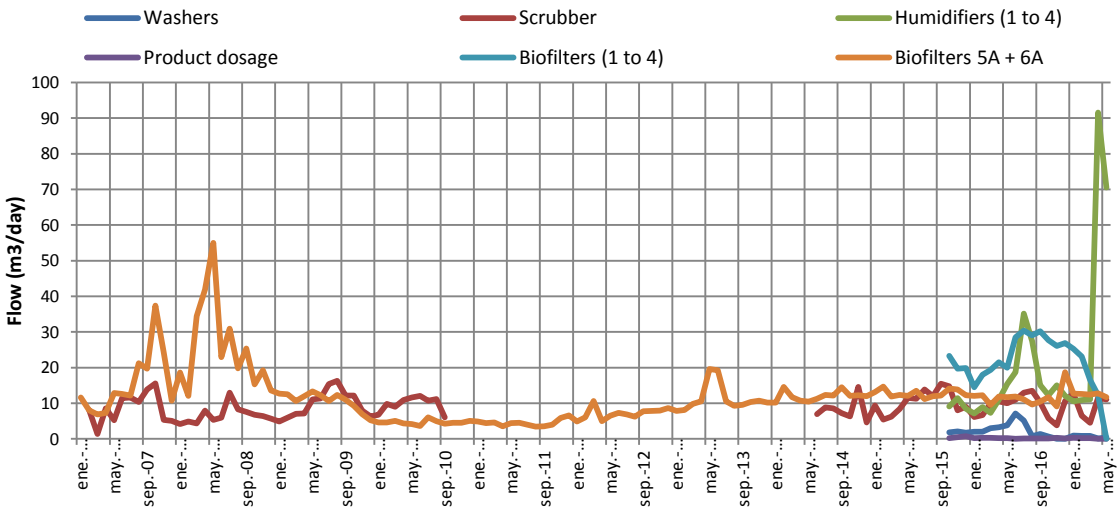



***Figure 1.9:*** *MW consumption in different process equipments from the air treatment area*

Initially it was believed that the product dosing equipment had a high MW consumption. However, following the installation of water meters in the different process equipment we were able to make sure that this was not he case.

The largest MW consumption in air treatment is due to humidifiers/scrubber and biofilters. Figure 1.10 shows the flow rates since October 2015, when the water meters were already fully installed.

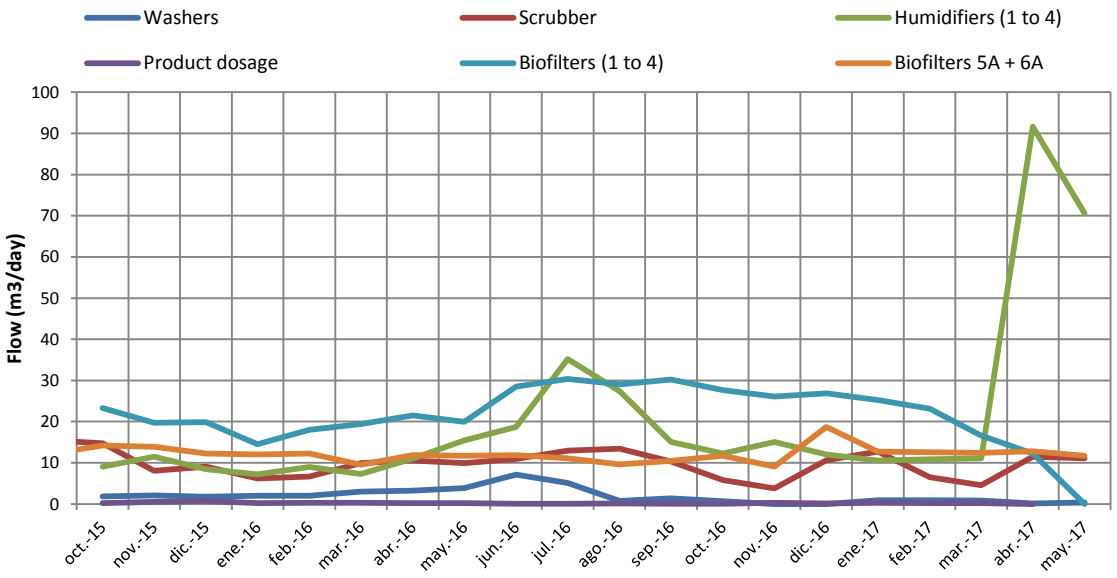



***Figure 1.10:*** *MW consumption in different process equipment from air treatment since October 2015*

On a closer examination, it is observed that the largest MW consumption is due to the biofilters, which are pending to regulate the irrigation to optimize MW consumption and produce GW. In April 2017, an external service company started changing the biofilters fluidised bed to control them, and a decrease of MW consumption has already been observed.

In the case of the humidifiers, the consumption remains stable around 10 m3/day except for a few months in which it was observed that the drop separator had to be repaired. This fault caused the water to be incorrectly retained and prevented its recirculation with a consequently larger MW consumption.

Due to the delicate nature of this area, the Ecoparc 2 has contacted an external company, expert in this type of process equipment, to carry out the maintenance and ensure their optimal state. In the meantime, we cannot perform any tests in this area until the process become stable.

Figure 1.11 shows the MW consumption of each of the humidifiers.

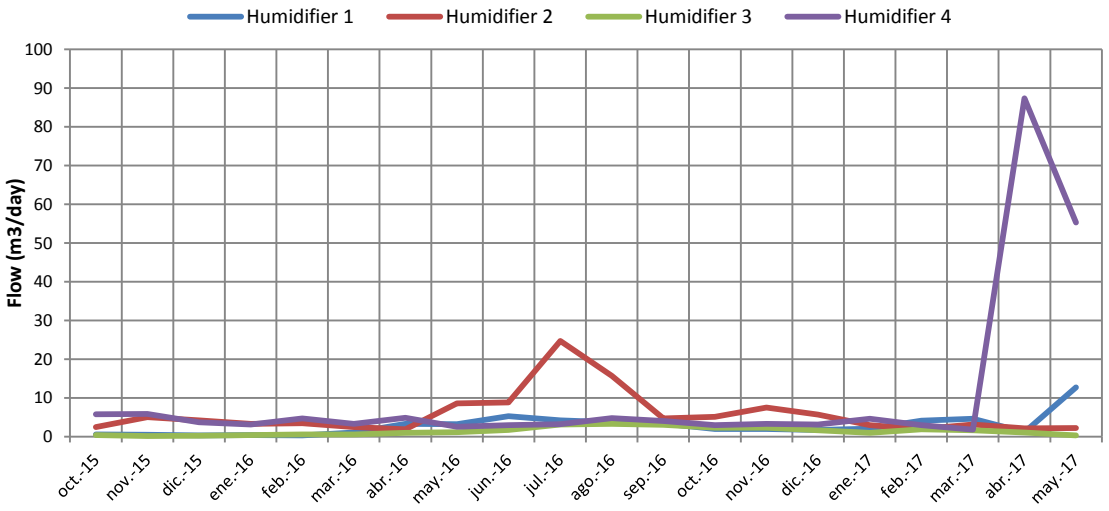



***Figure 1.11:*** *Humidifiers MW consumption*

As mentioned above, the humidifiers MW consumption is usually constant, except for the already mentioned case of the peak due to the state of the drop separator.

In the case of humidifier 2, it is observed that in May and June 2016 consumption increased and in July 2016 it reached a maximum, in which maintenance operations were carried out to return it to its previous state, being normalised in September 2016. The same happened with humidifier 4, where a big peak is observed in April 2017, in which the maintenance operations were carried out in time and it was possible to reduce consumption for the following month. In May 2017 humidifier 1 maintenance was started.

Figure 1.12 shows the consumption of each biofilter.

The biofilters MW consumption varies between 4 and 8 $m^3$/day normally. This process equipment has resulted to be difficult to maintain stable with a homogeneous humidity at all points of the fluidised bed. That is why it was decided to hire the external company, experts in this type of process equipment, to carry out a correct maintenance and control. For this reason, data are not available since February in biofilter 4 and since April in the rest of the biofilters, since the process equipment is not accessible for the reading the water meter.

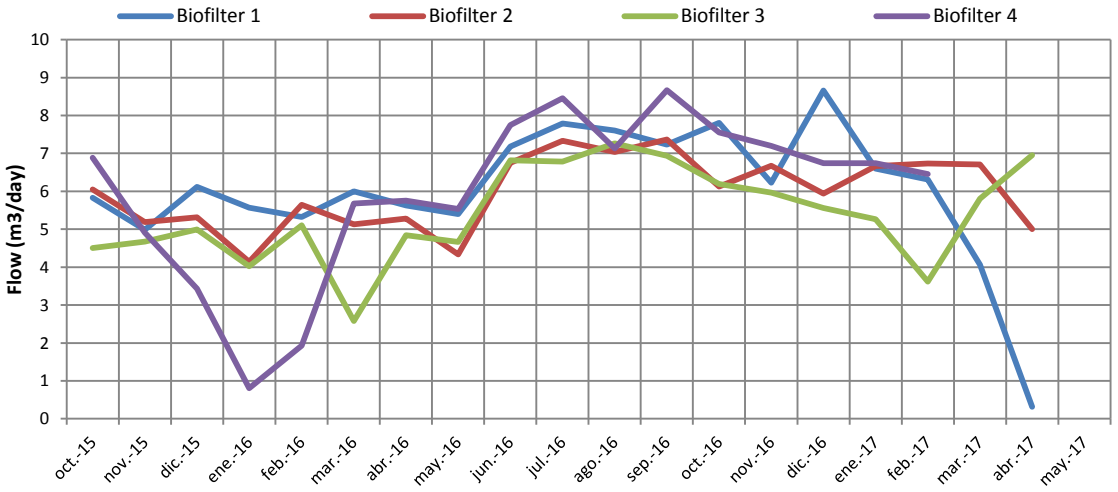



***Figure 1.12:*** *Biofilters MW consumption*

Figure 1.13 shows the biofilters 5 and 6 MW consumption. These biofilters were built in an expansion of the company's facilities in which 24 composting trenches were added, which assumed a high load in the air to be treated and caused a change for the air treatment area, so a scrubber was built followed by biofilters 5 and 6.

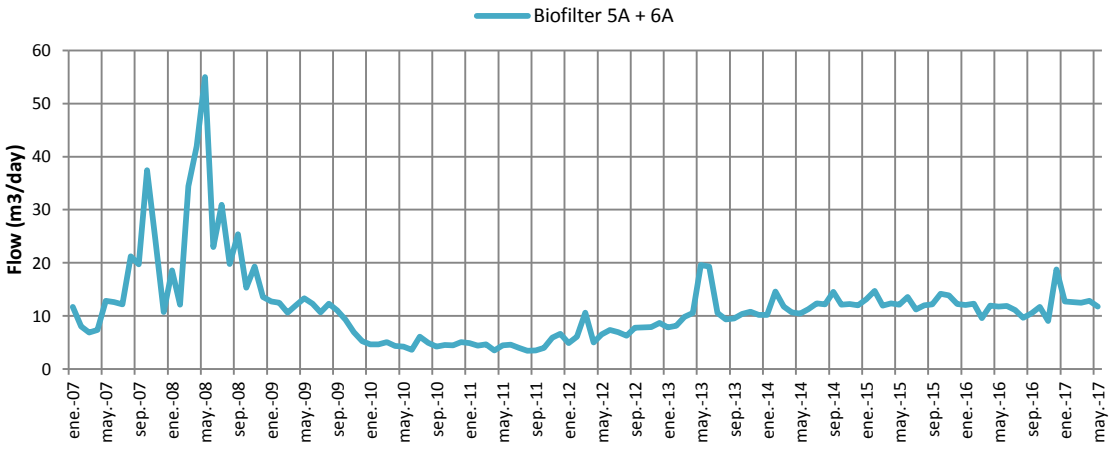



***Figure 1.13:*** *Biofilters 5 and 6 MW consumption*

The MW consumption in these biofilters has become much higher than biofilters 1-4 initially, however since 2010 it has been possible to significantly reduce consumption. The consumption of these 2 biofilters together varies between 9-18 $m^3$/day. As in biofilters 1-4, these will be controlled by the external company. So the irrigation regularization is outwith the scope of this current thesis project.

d. Watering gardens

Figure 1.14 shows MW consumption due to watering the gardens.

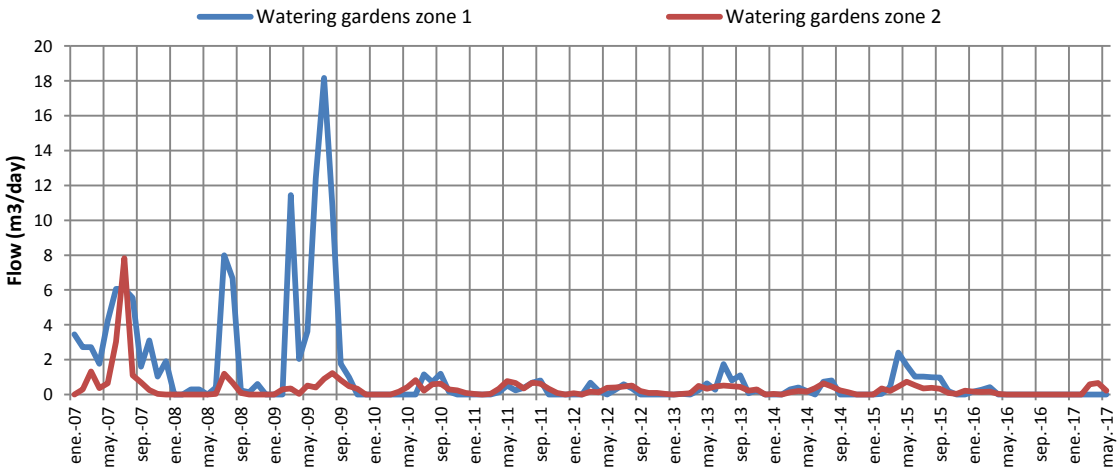



***Figure 1.14:*** *Watering gardens MW consumption*

The consumption of MW for watering the gardens was initially very high, so it was decided to change the irrigation system and eliminate some areas. In this way it was possible to maintain current consumption at < 1 $m^3$/day.

### 1.3.3.3. PW production: Global evaluation

Figure 1.15 shows the PW produced in the anaerobic digestion against the PW collected from the rest.

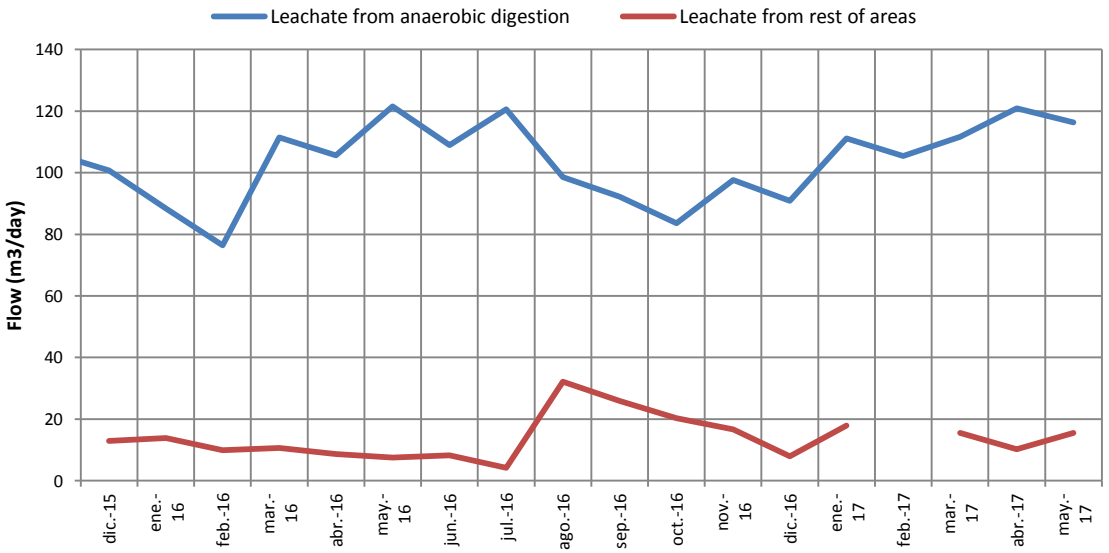



***Figure 1.15:*** *PW produced in the anaerobic digestion area and PW produced in the rest*

The PW production in both areas is unstable, there are variations from 75 to 122 $m^3$/day in PW produced in the anaerobic digestion area and from 4 to 33 $m^3$/day in the rest.

Table 1.7 shows PW percentage month to month, as well as the variation in percentage of PW production in relation to the previous month.

***Table 1.7:*** *PW percentage in the anaerobic area and the rest*

| Date | PW from anaerobic digestion | | | PW from rest of areas | | |
|---|---|---|---|---|---|---|
| | quantity | production (%) | variation (%) | quantity | production (%) | variation (%) |
| Dec-15 | 100,7 | 88,6% | | 12,9 | 11,4% | |
| Jan-16 | 88,4 | 86,5% | -12,3% | 13,8 | 13,5% | 7,0% |
| Feb-16 | 76,4 | 88,6% | -13,5% | 9,9 | 11,4% | -28,5% |
| Mar-16 | 111,4 | 91,3% | 45,7% | 10,6 | 8,7% | 7,1% |
| Apr-16 | 105,6 | 92,5% | -5,2% | 8,6 | 7,5% | -18,6% |
| May-16 | 121,6 | 94,2% | 15,1% | 7,5 | 5,8% | -13,0% |
| Jun-16 | 108,9 | 93,0% | -10,4% | 8,2 | 7,0% | 9,3% |
| Jul-16 | 120,6 | 96,6% | 10,7% | 4,2 | 3,4% | -48,5% |
| Aug-16 | 98,6 | 75,4% | -18,2% | 32,2 | 24,6% | 661,8% |
| Sep-16 | 92,2 | 78,0% | -6,5% | 25,9 | 22,0% | -19,4% |
| Oct-16 | 83,6 | 80,5% | -9,3% | 20,3 | 19,5% | -21,7% |
| Nov-16 | 97,7 | 85,4% | 16,8% | 16,7 | 14,6% | -18,0% |
| Dec-16 | 90,9 | 92,0% | -6,9% | 7,9 | 8,0% | -52,7% |
| Jan-17 | 111,1 | 86,1% | 22,2% | 17,9 | 13,9% | 126,9% |
| Feb-17 | 105,5 | | -5,1% | | | |
| Mar-17 | 111,7 | 87,8% | 5,9% | 15,5 | 12,2% | |
| Apr-17 | 120,9 | 92,2% | 8,3% | 10,2 | 7,8% | -34,4% |
| May-17 | 116,3 | 88,2% | -3,8% | 15,5 | 11,8% | 52,1% |
| **Minimum** | **76,4** | **75,4%** | **-18,2%** | **4,2** | **3,4%** | **-52,7%** |
| **Maximum** | **121,6** | **96,6%** | **45,7%** | **32,2** | **24,6%** | **661,8%** |
| **Average** | **103,5** | **88,1%** | **2,0%** | **14,0** | **11,9%** | **40,6%** |

Both the PW production in anaerobic digestion and the rest are very unstable, have constant variations, some of which are very pronounced.

It is also observed that the large PW percentage of input to WWTP comes from the anaerobic digestion, since they represent between 75.4 and 96.6 %.

Since the WWTP is biological, the process is sensitive to variations, since it needs a time to adapt to the new conditions. This indicates a clear need to retain the PW in a homogenisation tank prior to entering the WWTP, where abrupt changes in flow and organic and ammonia loads are minimised since the effluent properties of the

anaerobic digestion differ from the rest. Adding a homogenisation tank to the input would allow a more stable flow with an organic load with less abrupt variations, this giving stability to the biological process and increasing its performance.

Figure 1.16 shows the total PW treated in the WWTP against the permeate obtained.

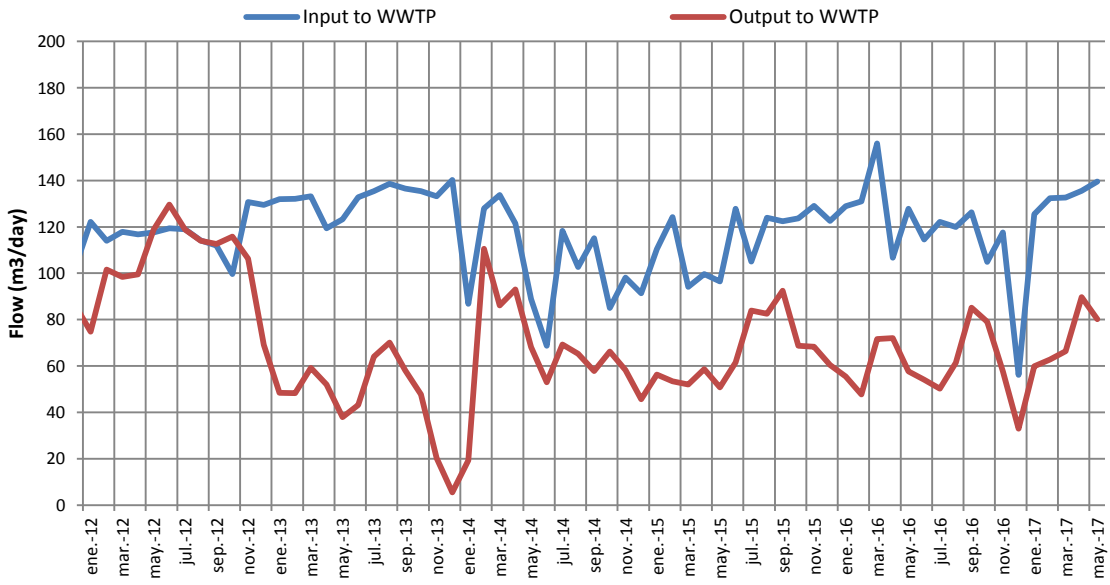



***Figure 1.16:*** *Input against output to the WWTP*

The input to the WWTP is very unstable, suffering abrupt variations month to month. This produces instability in the biological processes of WWTP and, consequently, lower purification efficiency.

However, much of the PW ends the purification process and exits as permeate, the rest are $N_2$ process emissions and purges of dead matter. There have been times when the process barely managed to clean PW and it was necessary to perform a large number of purges. Again, this shows that it is necessary to stabilize the WWTP input to stabilise the process and obtain a good performance.

Figure 1.17 shows the MW against CW consumption. Although the CW tank is used, it is only a small amount of the MW consumed in Ecoparc 2. Even so, it is necessary to prioritize the CW consumption to reduce, as far as possible, the MW consumption.

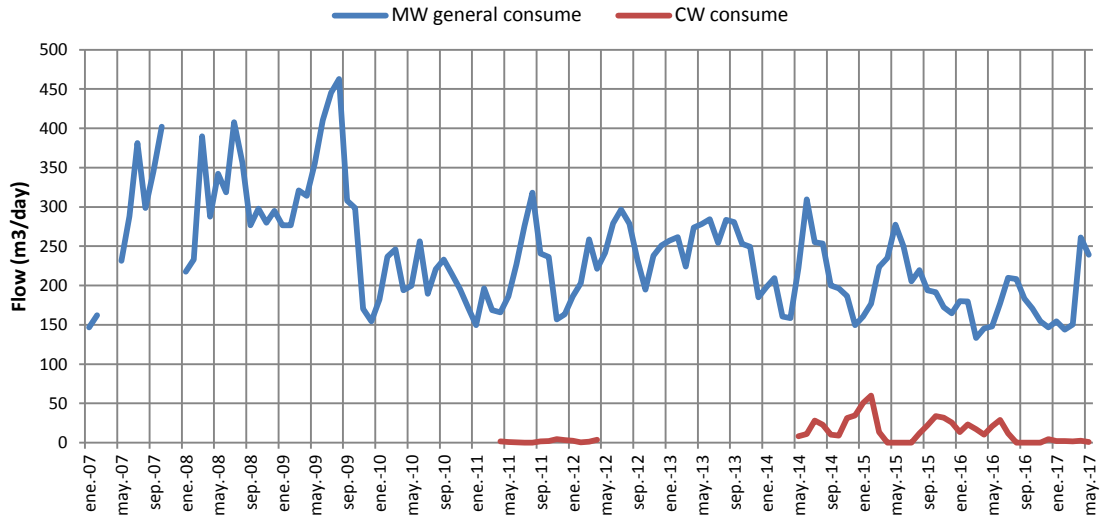



***Figure 1.17:*** *MW against the CW consumption*

Figure 1.18 shows the permeate obtained in the WWTP versus the GW discharged to the collector.

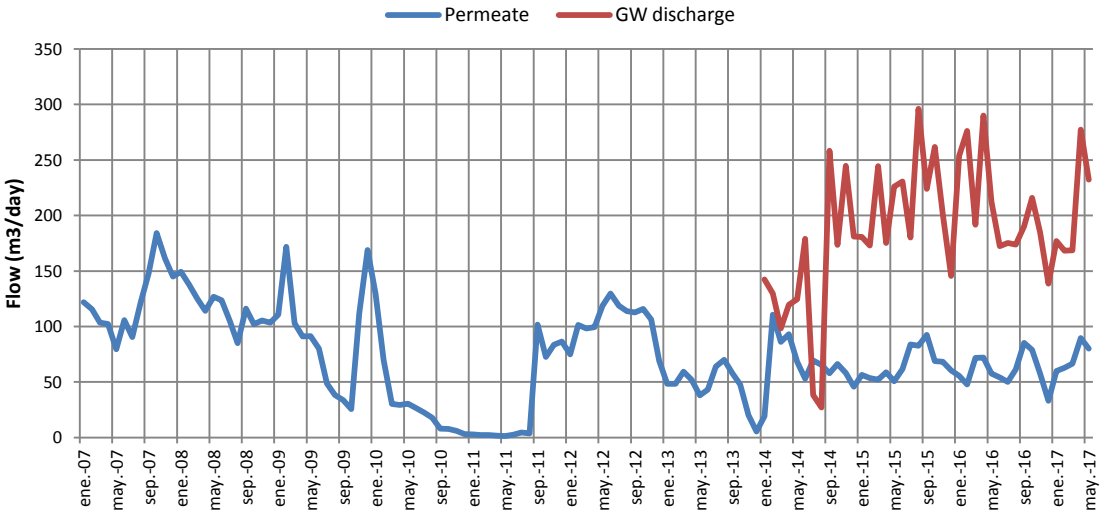



***Figure 1.18:*** *Permeate obtained in WWTP versus GW discharged to the collector*

The amount of permeate is less than the GW discharge. The treatment of the GW in the purifier would allow a discharge of greater quality to be obtained. Therefore, it is advisable to treat all wastewater, either PW or GW, in the WWTP before sending them to the collector. In this way it would be possible to reduce significantly the nitrogen forms ($NH_4^+$, $NO_3^-$ and $NO_2^-$) and organic loads (COD), obtaining a discharge of higher quality. In order to achieve this, the performance and effectiveness of the WWTP must

be improved so that its treatment capacity increases and all wastewater can be sent to the WWTP thus improving the discharge.

Figure 1.19 shows the annual MW and CW consumption of Ecoparc 2.

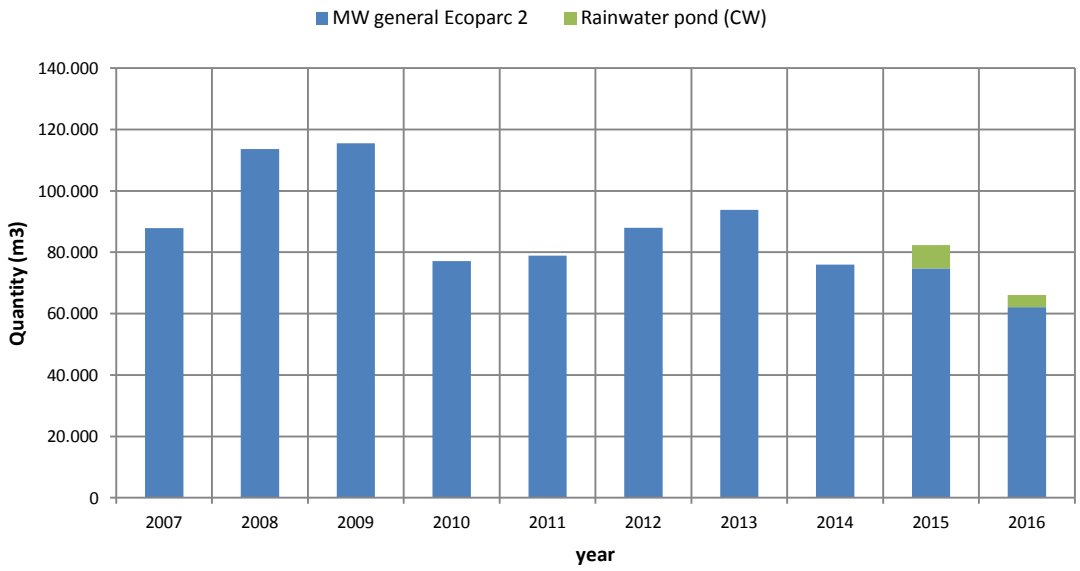



***Figure 1.19:*** *Annual MW and CW consumption of Ecoparc 2*

Water consumption at Ecoparc 2 has actually declined to almost half the consumption of 10 years ago. Even considering modifications and extensions to which the company has been subjected, it has been possible to optimise water consumption, being significantly lower iver the last 7 years, and reaching the minimum consumption of the company last year.

Figure 1.20 shows the GW amount discharged and the total permeate obtained in the WWTP, either discharged or recirculated.

The GW amount discharged in 2014 was around 100,000 $m^3$. Thanks to changes in the process in which GW can be reused at specific points in the process, it has been possible to reduce the discharge in 2015 to 66,000 $m^3$ and to 58,000 $m^3$ in 2016. Therefore, it has been possible to improve significantly the discharge amount to the collector, having decreased by more than 37% in relation to the 2014 data. When reusing the GW in process points, not only the MW consumption decreases but these

waters are destined to the WWTP as PW and end their journey as already purified permeate, with which we also improve the discharge quality.

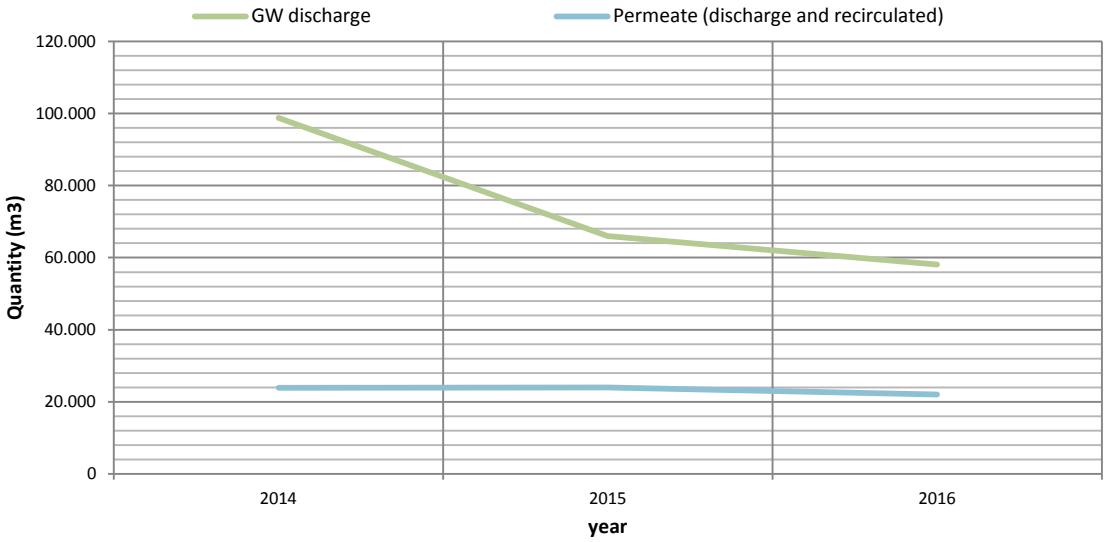



***Figure 1.20:*** *GW discharge versus permeate produced*

The amount of permeate obtained in the WWTP does not change significantly over the years. This because the WWTP input does not increase by including the GW but rather it is received in place of MW consumed years ago.

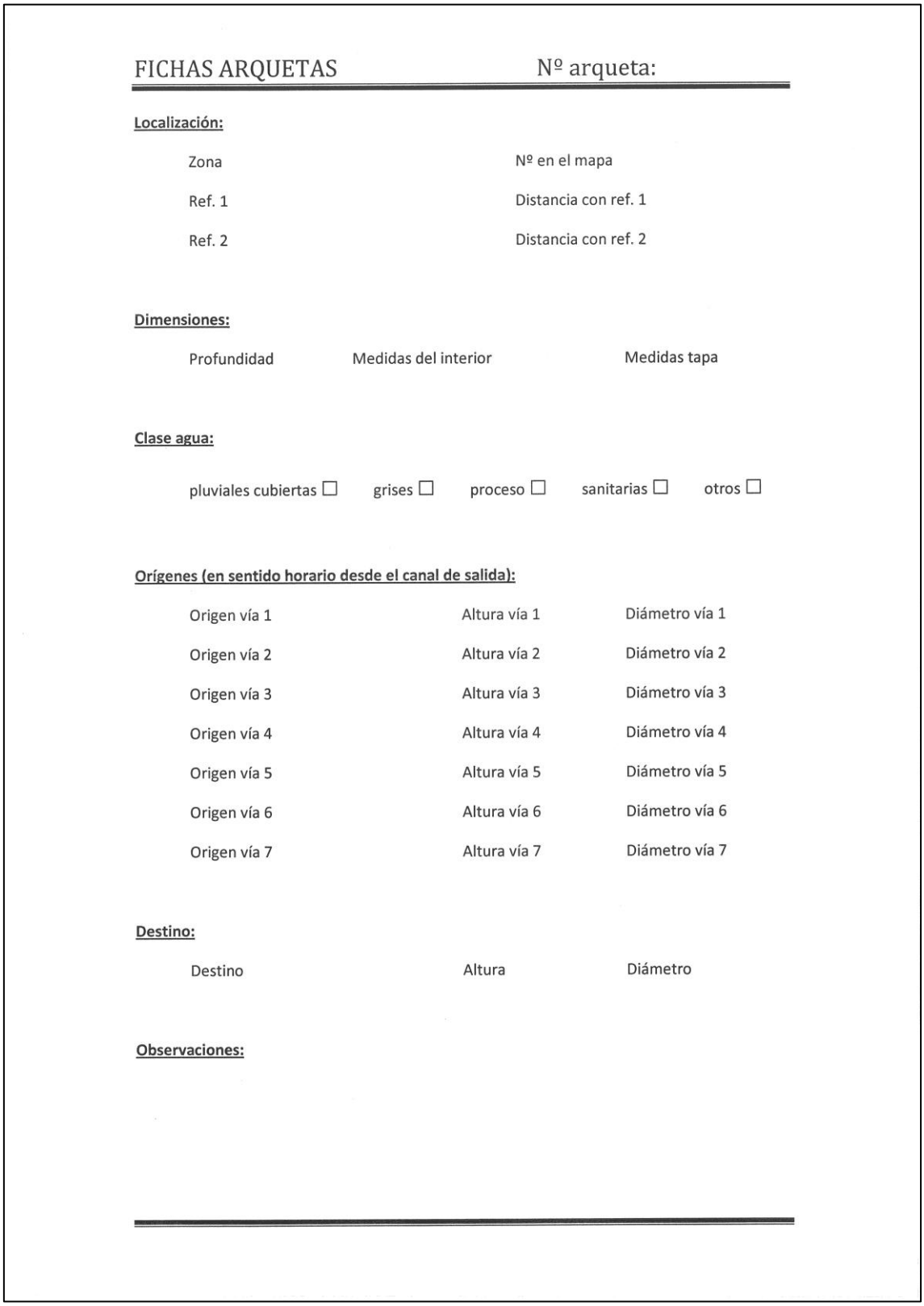
## 1.4. Changes made in the plant during the thesis

Throughout the thesis, and particularly at this phase, there have been several changes that have made a significant improvement in the usage of wastewater at Ecoparc 2, both with regards to the consumption and production as well as the wastewater quality that goes to the collector, and also in the control of these waters.

### *1.4.1. Connection points and drawings*

Each connection points of Ecoparc 2 has been identified with all the necessary data to be able to update the drawings in relation to completing a file for each of them as shown in figure 1.21.

In the header has been entered the connection point number that has been assigned following the company codification.



FICHAS ARQUETAS Nº arqueta:

**Localización:**

| | |
|---|---|
| Zona | Nº en el mapa |
| Ref. 1 | Distancia con ref. 1 |
| Ref. 2 | Distancia con ref. 2 |

**Dimensiones:**

| | | |
|---|---|---|
| Profundidad | Medidas del interior | Medidas tapa |

**Clase agua:**

pluviales cubiertas ☐ grises ☐ proceso ☐ sanitarias ☐ otros ☐

**Orígenes (en sentido horario desde el canal de salida):**

| | | |
|---|---|---|
| Origen vía 1 | Altura vía 1 | Diámetro vía 1 |
| Origen vía 2 | Altura vía 2 | Diámetro vía 2 |
| Origen vía 3 | Altura vía 3 | Diámetro vía 3 |
| Origen vía 4 | Altura vía 4 | Diámetro vía 4 |
| Origen vía 5 | Altura vía 5 | Diámetro vía 5 |
| Origen vía 6 | Altura vía 6 | Diámetro vía 6 |
| Origen vía 7 | Altura vía 7 | Diámetro vía 7 |

**Destino:**

| | | |
|---|---|---|
| Destino | Altura | Diámetro |

**Observaciones:**

***Figure 1.21:*** *File to be completed with each connection point data*

In the “Localización” section, the area where it is located has been identified and 2 reference points (always of the company facilities) have been entered including the

distance whit respects to them to be able to reflect in the drawing the exact point at which the connection point is located.

In the “Dimensiones” section has been identified the depth, interior measurements and cover of each connection point with a manhole.

The “Clase agua” section identifies the water type it contains taking into account the different possibilities (CW, GW, PW, BW or others).

The “Orígenes” section identifies all the connections that connection points have, determining where they come from, at what height they are in the connection point and the pipe diameter that carries this effluent.

The “Destino” section identifies where this effluent is directed to and at what height and diameter does the pipe leave of the connection point with a manhole.

Based on the data collected from each connection point, the water system drawings have been updated with the exact location and dimensions.

### *1.4.2. Water meters*

Ecoparc 2 already had water meters at various points to account for MW consumption or wastewater production. However, we were able to install additional meters to be able to quantify the most significant global and partial flows in Ecoparc 2.

Knowing that the areas that involve a greater MW consumption are from air treatment and WWTP, water meters have been installed in the process equipment of these areas to be able to quantify each one and to be able to act in the most relevant points such as humidifiers and biofilters from air treatment or vacuum pump from the WWTP.

### *1.4.3. GW reused*

The GW produced in Ecoparc 2 contributes a large amount to the discharge and should be minimised as much as possible. Although they comply with the discharge parameters, it is ideal that all these waters pass through the WWTP in a way that decreases the concentration thus improving the discharge quality.

For this reason, a change has been proposed that implies an improvement in discharge quantity and quality: namely to capture those GW effluents that contain higher concentrations and to use them in the possible processing points for cleaning process

equipment or as a flocculant mixture for centrifuges. In this way it is possible to reduce the MW consumption, this water ends up into the WWTP being treated as a PW without having to increase the WWTP capacity and obtaining the same permeate quantity.

- In the WWTP area there are three refrigeration towers and one evaporator vacuum pump, which produce large GW quantities with practically nonexistent total and volatile solids, COD and ammonia concentrations. According to these properties, in November 2013 a deposit was installed that collects these effluents. The captured water is analysed and it is determined whether it is possible to conduct them to the CW tank by contributing to MW consumption minimization or should go directly to the retention tank (figure 1.22).

***Figure 1.22:*** *GW deposit*

- In December 2013, a test was carried out in which a pit was built that captured the GW from the air treatment area and used it for anaerobic digestion for cleaning process equipment and the centrifuge flocculant mixture, but the amount of solids that accumulated in the pit made it difficult for the pump to work, so it had to be stopped. A proposal was made to change to a screw pump instead of the centrifugal pump and make a sediment trap in the previous connection point, but it is not clear if it will work correctly. At present, there is still the option of reusing these effluents, pending the construction of a system capable of doing so without problems caused by the retained solids (figure 1.23).

***Figure 1.23:*** *GW pit in works*

### *1.4.4. Biofilters irrigation*

In the study explained in point 1.3.1, tests were carried out to determine the amount of GW produced, which collection points were the most significant ones in its production, and the COD, ammonium and total and volatile solids values of the different collection points.

In this study it was observed that the effluents with the highest concentration came from the air treatment area, namely from the water captured from the biofilters outlet. Figure 1.24 shows a schematic diagram of this effluent production.

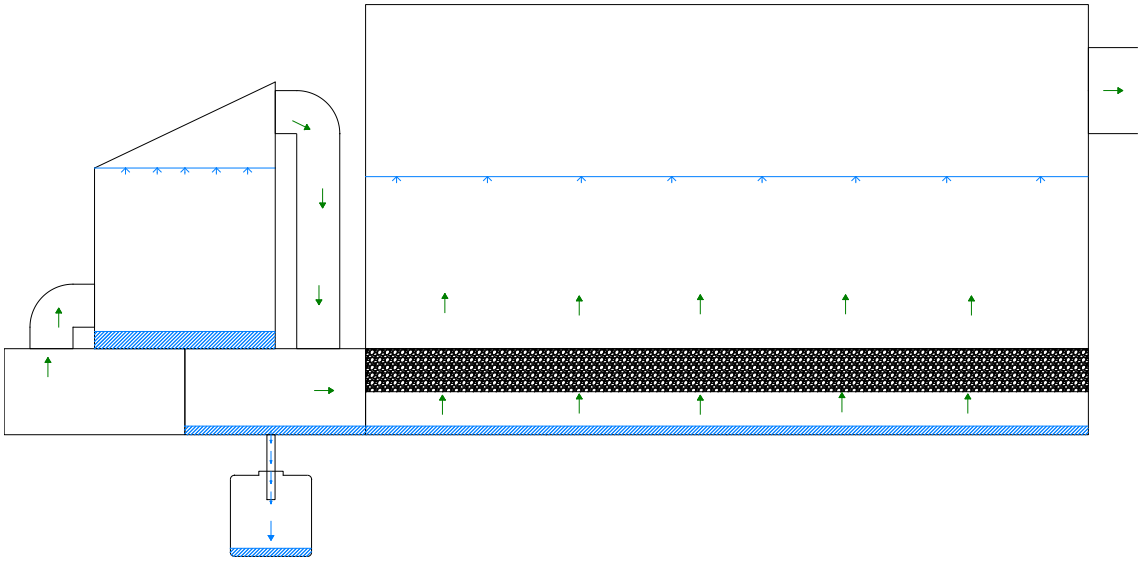


***Figure 1.24:*** *GW production in biofilters from the air treatment area*

The air to be treated enters the humidifier bottom, which is moistened by the sprinkler. The humidified air exits through the top and is led to the biofilters bottom, where it passes through 120 cm of fluidized bed and exits the top towards the chimney to be emitted to the atmosphere.

In relation to the water, in the upper part of the humidifier there is a drop separator that retains the water that is entrained by the air to be treated and returns it to the humidifier bottom, where it captures the spray system that is used to humidify the air. Regarding the biofilters, an irrigation system is available to maintain an adequate humidity of the bed avoiding for it to be dried by the treated air. The surplus water is collected in the biofilters bottom and is channelled through various connection points until it reaches the GW tank.

To reduce the GW amount at this point it was proposed to adjust the irrigation frequency of the biofilters and time until the optimum sequence is obtained that maintains an adequate bed humidity without producing surplus water. However, due to the current delicate state of the biofilters, we have not been able to act at this point. This task remains pending for when the state of the biofilters improves sufficiently.

### *1.4.5. Homogenisation tank*

The discharge to the collector contains the GW, BW and permeate, which have very different characteristics and there are peak times in which they are produced in very different quantities and frequencies. As a result, the discharge is constantly subject to peaks both in quantity and in concentration of different parameters.

A homogenisation tank was created that increases the wastewater retention time and allows for a smooth discharge in quantity. In this way damage to the industrial collector pumps is avoided, and the concentration is stabilised with a more constant discharge (figures 1.25 and 1.26).

***Figure 1.25:*** *Homogenisation tank view 1*

***Figure 1.26:*** *Homogenisation tank view 2*

### *1.4.6. CW in refrigeration towers*

Since the refrigeration towers have a high MW consumption, the idea was to use CW for this process equipment in order to reduce MW consumption. For this reason, a company was consulted and to install the necessary equipment. After consultation with the company, following factors were taken into consideration:

- The input water to refrigeration towers must contain a conductivity of less than 2,000 µS/cm.
- If water contains volatile COD, ammonia and others volatiles odours can be produced.
- Suspended solids can clog the refrigeration tower landfiller.
- If water contains fouling salts it can override the landfill over time.
- It is more probable that with the same conditions the discharge might contain Legionella in breach of the RD 1620/2007.

In case the test is carried out the following should be taken into account:

- If the refrigeration tower decreases its performance, it is a sign that the water is affecting the landfill and would have to be changed with a cost of 4,000-5,000 €.
- It would be necessary to previously incorporate a filter to retain the solids with a pore size of 100-200 µm, which would cost about 1,000-2,000 €.

In order to maintain the correct working conditions in the refrigeration towers and vacuum pump it was decided not to use CW in this process equipment.

## 1.5. Conclusions

Throughout this phase and the associated changes, given in the previous point, the following conclusions can be made:

- All possible effluents at Ecoparc 2 have been identified, each connection point has been studied, new water meters have been installed and all possible flows have been determined. From all this information, it has been possible to fully characterise the water system at Ecoparc 2 and the new information has been incorporated into the update plant drawings.

  Areas with the highest MW consumption are those from air treatment and the WWTP.

  In air treatment, the equipment that consumes the most are the humidifiers and biofilters. It has also been observed that this area is the one that produces the most GW. The consumption of the GW needs to be regulated in order to minimise to MW consumption and decrease the mount of GW produced. In humidifiers it is necessary have a good maintenance of the drop separator to avoid a high consumption. In the biofilters it is needed to regulate the irrigation so that it is optimal and maintains the bed humidity but is not excessive and produces a surplus. However, due to the delicate state of the biofilters, Ecoparc 2 has contracted an external for the maintenance of this equipment. As a consequence, it is not possible to carry out any tests at this time.

  In the WWTP, the largest MW consumption is due to the refrigeration towers and the evaporator vacuum pump. We have studied the possibility of used CW in the refrigeration towers, but not test has been carried out due to the lack of a minimum conductivity and content in suspended solids so as not to deteriorate the process equipment. However, as will be seen in phase 2, it has been possible to optimize the WWTP, so that the evaporator has been able to stop treating all the effluents in the WWTP. In this way it has been possible the stop refrigeration tower 2 and vacuum pump, thus significantly reducing MW consumption.

  Connection points with PW were analysed. We have observed that the effluents with higher COD and ammonium concentrations come from the composting areas (tunnels and trenches) and from the waste discharge pits. However, at the end point, where the PW accumulate to be treated, COD and ammonium values are diluted and decreased. COD maximum values exceed 75,000 mg

$O_2$/L and ammonium 5,000 mg N/L, however the PW tank has 40% lower concentrations (42,500 and 3,000 mg/L respectively).

It has also been observed that more than 75% of the PW treated in the WWTP comes from the anaerobic digestion area, which also has very different properties from the rest of PW. Taking into account the discontinuity of effluents and different properties of each one of them, it is necessary to carry out a homogenisation to avoid destabilising the WWTP biological process.

An extensive GW study has also been carried out, determining that the most concentrated effluents come from the air treatment area, and it is diluted as the effluent progresses and is mixed with other GW effluents.

- It has been proposed to avoid mixing the more concentrated GW effluents, using these at any point in the process where possible. In this way, it is possible to reduce MW consumption and, moreover, the more concentrated GW end up in the WWTP to be treated as PW. The GW effluent contains much lower concentrations than the previous ones since mixing with effluents from air treatment area is avoided. For this reason, a pit was built in a GW connection point, where a pump was installed that sends the effluent to a deposit which feeds the anaerobic digestion area for the cleaning water and centrifuge flocculant mixtures. However, many solids sedimented and prevented the pump from working properly, which stopped this test. At present, this effluent idea is maintained, in the hope of incorporating a system that avoids these difficulties.

- A deposit has also been incorporated that collects the water produced in the refrigeration towers and the evaporator vacuum pump, which are similar to CW. Periodically the effluent properties are analysed to determine if it is should be sent to the CW tank to reuse the water in the process and reduce the MW consumption, or to the retention tank to discharge along with the GW.

  It has not been possible to perform the required tests in the MW consumption reduction in the biofilters irrigation and this task is pending for when the state of this process can allow it.

  The option to use CW in refrigeration towers has also been studied. However, this idea has been dismissed given the need to maintain this process equipment in good condition.

  On the basis of all these measures it has been possible to reduce MW consumption by 33.8% in relation to 2013 when this project was started.

Looking at the data, MW consumption in 2009 was 115,000 $m^3$ compared to the 62,000 $m^3$ consumed in 2016.

It has also been possible to reduce the discharge to the collector which was 98,000 $m^3$ GW and 24,000 $m^3$ permeate in 2014 compared to 58,000 $m^3$ GW and 22,000 $m^3$ permeate in 2016, implying a 34,7% discharge lower than in 2014.

# PHASE 2. WASTEWATER TREATMENT PLANT OPTIMISATION

## 2.1. Introduction

Ecoparc 2 has a wastewater treatment plant with a MBR system (Membrane Bio Reactor) to treat wastewater before being discharged to the collector, in order to reduce the organic load (COD) and nitrogen (ammonium, nitrate and nitrite) in compliance with the discharge parameters established in the Decree 130/2003.

The WWTP unit operations at Ecoparc 2 are:

- Primary treatments
    - Flocculation → only the PW from the anaerobic digestion area is subjected to flocculation and phase separation by a centrifuge. As we have seen in phase 1 this effluent accounts for more than 75% of the PW to be treated.
    - Filtration → The feed pump picks up the PW from the tank and directs it to one rotating and two vibrating filters with a pore size of 800 µm (figure 2.1).

***Figure 2.1:*** *Vibrating filters at Ecoparc 2*

- Secondary treatments:
  - Aerobic biological reactor (oxic): nitrification tank → Oxidation of ammonium to nitrite and nitrite to nitrate, releasing $H^+$ ions and thereby acidifying the medium. Reactions:

$$NH_4^+ + 1{,}5\ O_2 \xrightarrow{nitrosomonas} NO_2^- + H_2O + 2\ H^+ \qquad \Delta E = -352\ kJ/mol$$

$$NO_2^- + 0{,}5\ O_2 \xrightarrow{nitrobacter} NO_3^- \qquad \Delta E = -73\ kJ/mol$$

$$NH_4^+ + 2\ O_2 \longrightarrow NO_3^- + H_2O + 2\ H^+ \qquad \Delta E = -425\ kJ/mol$$

    In the case of exothermic reactions it is necessary to use refrigeration to maintain the temperature in the reactor. The necessary oxygen is obtained by two blowers injecting air under pressure into the reactor bottom. Since $H^+$ ions are produced, the reactions acidify the medium which, if the alkalinity is not sufficient, can be regulated with the addition of sodium hydroxide.

  - Aerobic biological reactor (anoxic): denitrification tank → Reduction of nitrate to nitrogen gas consuming carbon compounds (COD) and $H^+$ ions, therefore making the medium more basic. Reactions:

$$4\ NO_3^- + 4\ H^+ + 5\ [C] \xrightarrow[bacteries]{facultative\ heterotropic} 2\ N_2 + 5\ CO_2 + 2\ H_2O$$

    A carbon source is needed that acquires COD content, if this is insufficient, methanol or acetic acid can be dosed to satisfy this need. As $H^+$ it consumed, the reactions produce an increase in pH which, if necessary, can be regulated by adding phosphoric acid. An agitator is required to avoid the sedimentation of solids and to release the gases produced ($N_2$ and $CO_2$).

  - Combined biological reactor: denitrification/nitrification tank → It is possible to operate either as denitrification, using methanol (carbon source) and stopping the aeration (anoxic conditions), or as nitrification airing (oxic conditions). A supply of methanol as a carbon source is necessary for the denitrification reactions.
- Tertiary treatments:
  - Ultrafiltration → Eight multilayer tubular ultrafiltration modules are available where suspended solids and bacteria are retained, allowing passage to water and ions (figure 2.2).

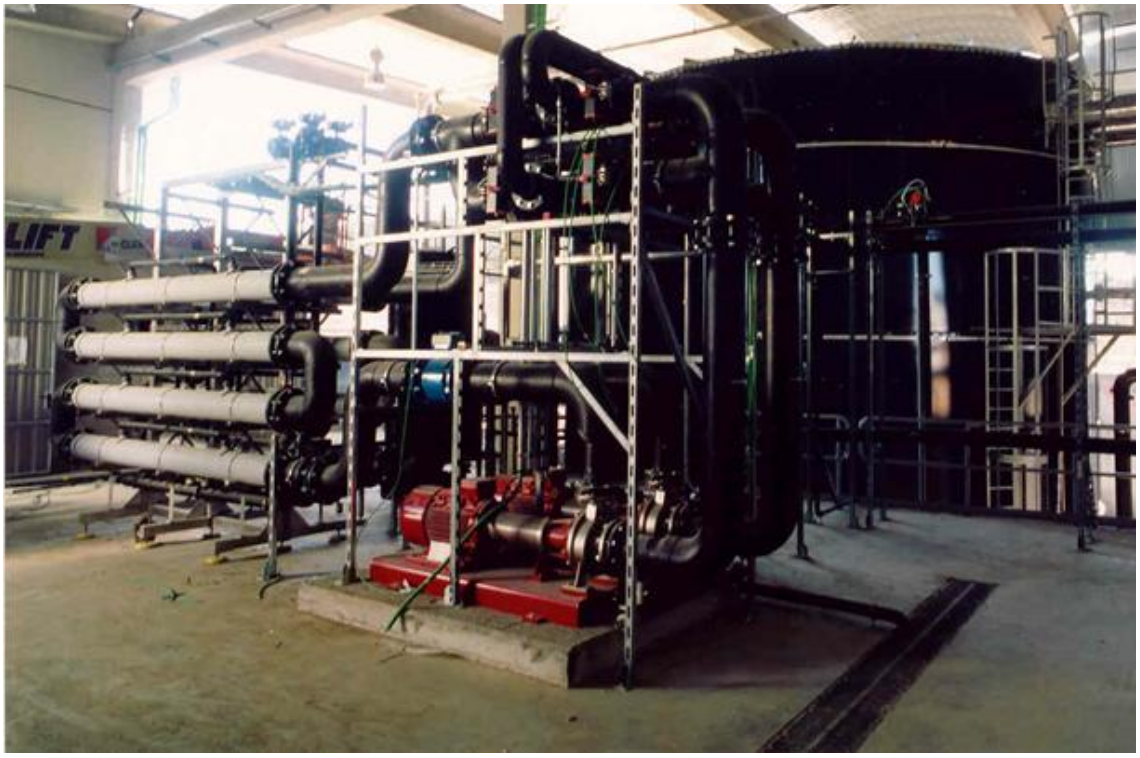


***Figure 2.2:*** *Ultrafiltration at Ecoparc 2*

- Other techniques:
  - Evaporation → An evaporator was initially installed to treat PW that did not come from the anaerobic digestion area at such times as is necessary. However, it has been possible to shut down this equipment and treat all the PW in the MBR WWTP, thus reducing energy and water consumptions.

The initial design consisted of a nominal capacity of 142 $m^3$/day with a COD of 10,000 mg/L, and involved three reactors: denitrification tank, nitrification tank and combined tank.

In January 2009, the plant had to be expanded by installing a second nitrification tank, so that it could handle 40,000 mg/L COD from the input. Figure 2.3 shows an upo to date Ecoparc 2 WWTP process scheme.

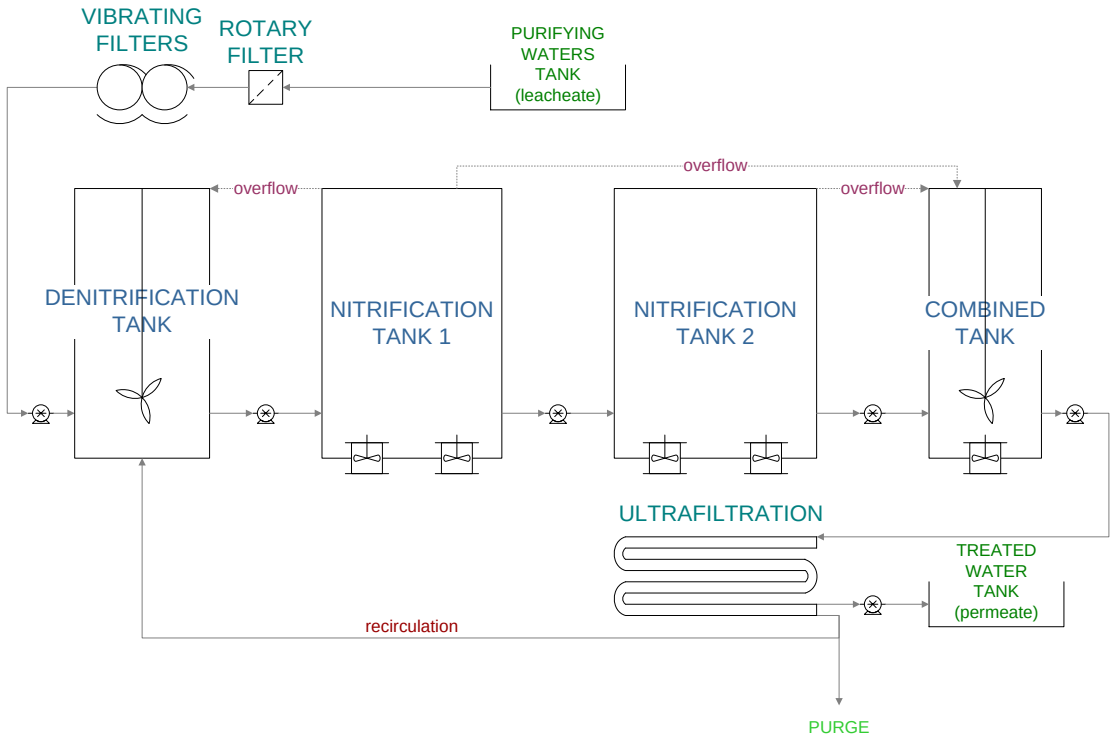



***Figure 2.3:*** *Ecoparc 2 WWTP process scheme (image author: Eloisa Albacete Garcia)*

## 2.2. Applied studies

### *2.2.1. Analytical*

Before this study, the analytical planning of the WWTP monitoring was the one presented in table 2.1.

***Table 2.1:*** *Former WWTP analytical planning*

| | PW | Denitrification tank | Nitrification tank 1 | Nitrification tank 2 | Combined tank | permeate |
|---|---|---|---|---|---|---|
| **dry material** | daily | 3 times at week | daily | daily | 3 times at week | daily |
| **pH** | daily | daily | daily | daily | daily | daily |
| **temperature** | daily | | | | | daily |
| **conductivity** | daily | | | | | daily |
| **dissolved $O_2$** | | | daily | daily | | |
| **COD** | daily | | | | | daily |
| **ammonium** | daily | | | | | daily |
| **alkalinity** | 3 times at week | | | | | 3 times at week |
| **nitrites** | | | | | | twice at week |
| **nitrates** | | | | | | daily |
| **Total phosphorus** | | | | | | twice at week |
| **chlorides** | | | | | | twice at week |

As seen in the table, only a few chemical parameters were determined at the WWTP input and output, and only the intermediate reactors dry material and pH were included. In some case the parameters exceeds the periodicity and in others the measurements were not being made.

Since the Ecoparc 2 WWTP basically removes COD and nitrogen, it was necessary to determine these parameters in each of the reactors, so the evolution could be observed and for it to be possible to quickly determine whether any of the reactors is malfunctioning.

Therefore, we decided to make the following changes.

#### 2.2.1.1. Sampling points

All the sample capture points have been established so that they are always the same in order to be able to compare the results obtained over time, bibliography dates and other similar WWTP dates.

#### 2.2.1.2. Procedures

In order to update the analytical procedures with standardised methods, we studied first whether the analytical procedure was correct and, if not, tests were performed with other methods and adapted to the laboratory routine. Finally, we decided to change the following procedures:

- *Nitrates and nitrites*: currently determined by NANOCOLOR or HACH tests that are based on standardized methods with internationally approved as DIN, EN, ISO, EPA and APHA.
- *Alkalinity*: determined according to the procedure 2320B of Standard Methods for the Examination of Water and Wastewater.

On the other hand, new analytics have been proposed to determine important parameters at specific points, thus obtaining a greater process control. The new analytics incorporated in the planning are the following:

- *Viscosity*: phase 3 will show the importance of this parameter, as both viscosity and solubility directly affect the WWTP biological process. For this reason a rotating viscometer has been acquired to determine this parameter.

- *Total nitrogen*: the different forms that nitrogen can be present are elemental nitrogen ($N_2$), organic nitrogen ($N_{org}$), ammonium ($N\text{-}NH_4^+$), nitrites ($N\text{-}NO_3^-$) and nitrates ($N\text{-}NO_2^-$) and their transformations obey the following order.

  $N_{org} \rightarrow N\text{-}NH_4^+ \rightarrow N\text{-}NO_2^- \rightarrow N\text{-}NO_3^- \rightarrow N_2$

  In the analytics performed in the laboratory only ammonium, nitrites and nitrates are determined. The PW also contains organic nitrogen that will later be transformed into the different components following the previous order, so it is necessary to know the $N_{org}$ concentration in the PW. By including the total nitrogen analytic and taking into account the ammonium, nitrite and nitrate data, the organic nitrogen can be calculated by calculating the difference between these values.
- *Sulphides:* is one of the discharge parameters that appears in the Decree 130/2003, for this reason it was decided to incorporate this analytic for the permeate obtained in the WWTP.
- *Total and volatile solids*: until now only the dry matter was determined by a humidity analyser but the solids distribution in suspended/dissolved and volatile/inerts was unknown. Therefore, total and volatile solids analytics were incorporated for the sludge and permeate, obtaining the results of the total and soluble samples and allowing for the calculation of the nine types of solids (figure 2.4).

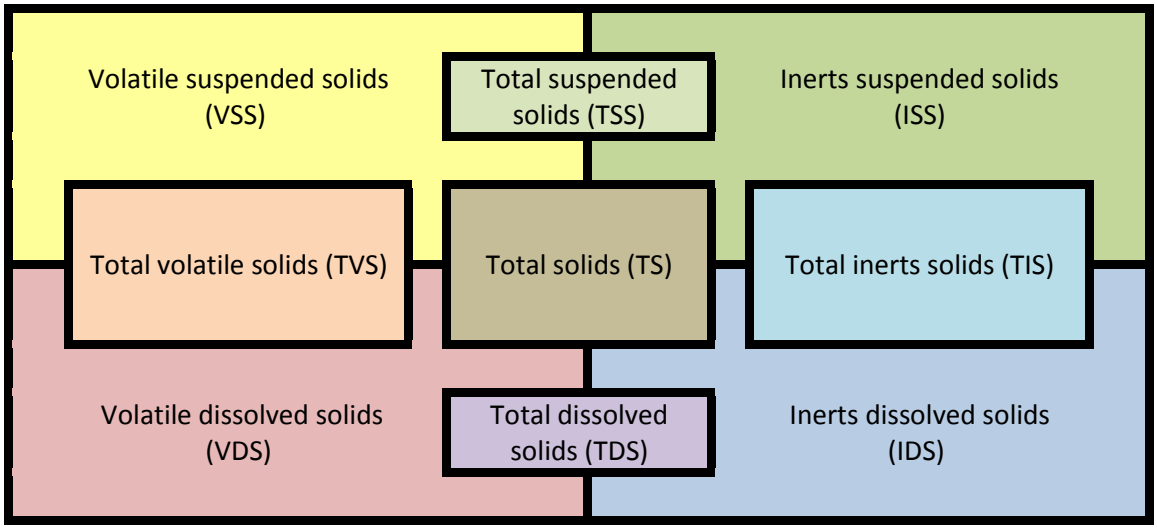



***Figure 2.4:*** *Solids distribution (image author: Eloisa Albacete Garcia)*

#### 2.2.1.3. Periodicity

It has been observed that there are analytics that are done too frequently and in contrast others that are considered vital are rarely done or even nonexistent. For this reason, the periodicity was modified taking into account the need to know the different parameters and response time to changes in the process. For this, a proposal was made to the different departments involved and adapted to the need of each one obtaining a suitable planning for all. Table 2.2 shows the current planning.

***Table 2.2:*** *Current WWTP analytical planning*

| | PW | Denitrification tank | Nitrification tank 1 | Nitrification tank 2 | Combined tank | permeate | purge |
|---|---|---|---|---|---|---|---|
| **dry material** | daily | weekly | daily | weekly | weekly | daily | daily |
| **temperature** | daily | daily | daily | daily | daily | daily | |
| **pH** | daily | daily | daily | daily | daily | daily | |
| **conductivity** | daily | weekly | weekly | weekly | weekly | daily | |
| **dissolved $O_2$** | weekly | weekly | daily | daily | weekly | | |
| **COD** | daily | weekly | weekly | weekly | weekly | daily | |
| **TOC** | weekly | | | | | weekly | |
| **Total nitrogen** | weekly | weekly | weekly | weekly | weekly | weekly | |
| **ammonium** | daily | weekly | weekly | weekly | weekly | daily | |
| **nitrites** | weekly | weekly | weekly | weekly | weekly | daily | |
| **nitrates** | weekly | weekly | weekly | weekly | weekly | daily | |
| **Total phosphorus** | weekly | | | | | weekly | |
| **Sulphates** | | | | | | weekly | |
| **sulphides** | | | | | | weekly | |
| **chlorides** | | | | | | weekly | |
| **Total solids** | | | weekly | | | weekly | |
| **Volatile solids** | | | weekly | | | weekly | |
| **Viscosity** | weekly | weekly | weekly | weekly | weekly | | weekly |
| **alkalinity** | weekly | | | | | weekly | |

#### 2.2.1.4. Analytics transmission

In order to reduce the response time to a process alteration, the analytical data transmission time has been minimised. At present, as soon as the analytics are finished, the information is transferred to an existing Excel record in which coloured indicators have been added to make them more visual, so that on the same day small deviations can be seen and acted on before they increase, thus minimising the impact.

### *2.2.2. Feed to the WWTP*

As we have seen previously, the PW produced at Ecoparc 2 has a lot of diversity both in properties and in the quantity produced. Since the WWTP is biological, it is important

to avoid sudden changes that destabilise the process and create alterations in the biology. For this reason it is necessary to minimize PW variations as far as possible.

#### 2.2.2.1. Flocculant performance improvement

One of the major problems in the biological process is the presence of flocculant in the PW. The presence of flocculant, hinders the biological reactions inside the reactors since a polyelectrolyte excess causes an increase in the viscosity and a decrease in the oxygen transference. One measure taken in this phase has been the optimisation of flocculant use to improve its performance and decrease the amount present in the PW. For this purpose, a flocculant supplier has been contacted and different flocculants have been tested.

*A. Test 1*

This test is performed with a liquid flocculant with emulsion (FLOPAM EM 840 HIB). Table 2.3 shows the results obtained.

***Table 2.3:*** *Results obtained in test 1 with FLOPAM EM 840 HIB*

| Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | Liquid phase TS (%) | Solid phase TS (%) | consumption (kg/Tn DM) |
|---|---|---|---|---|---|
| 1300 | 3.5 | 40 | | | 17.77 |
| 2300 | 3.5 | 40 | | | 31.44 |
| 3500 | 3.5 | 40 | 1.02 | 35.61 | 47.85 |
| 3500 | 5.0 | 40 | 1.37 | 35.47 | 33.49 |
| 3500 | 7.0 | 40 | 2.09 | 34.74 | 23.92 |
| 3500 | 9.0 | 40 | 2.82 | 33.54 | 18.61 |
| 3500 | 9.0 | 60 | 2.72 | 32.13 | 18.61 |

Figure 2.5 shows a graph of sludge flow versus total solids in the phase liquid and the polyelectrolyte consumption.

The TS in the liquid phase increases as the sludge flow increases, while the polyelectrolyte consumption decreases.

In the test the following results were obtained: TS 1.02 - 2.82 % with a consumption of 18.61 – 47.85 kg/Tn DM. During normal centrifuge operation, a flow rate of 9 $m^3$/h is found giving values of ST 2.22 – 2.90 % in the liquid phase with a Zetag 8185 polyelectrolyte consumption of 7.0 kg/Tn DM. It is also true that the polymer flow rate normally is 1,000 – 1,500 L/h compared to the 3,500 L/h that was consumed in the test.

This is an important factor to evaluate since, despite obtaining better results, this implies a greater consumption.

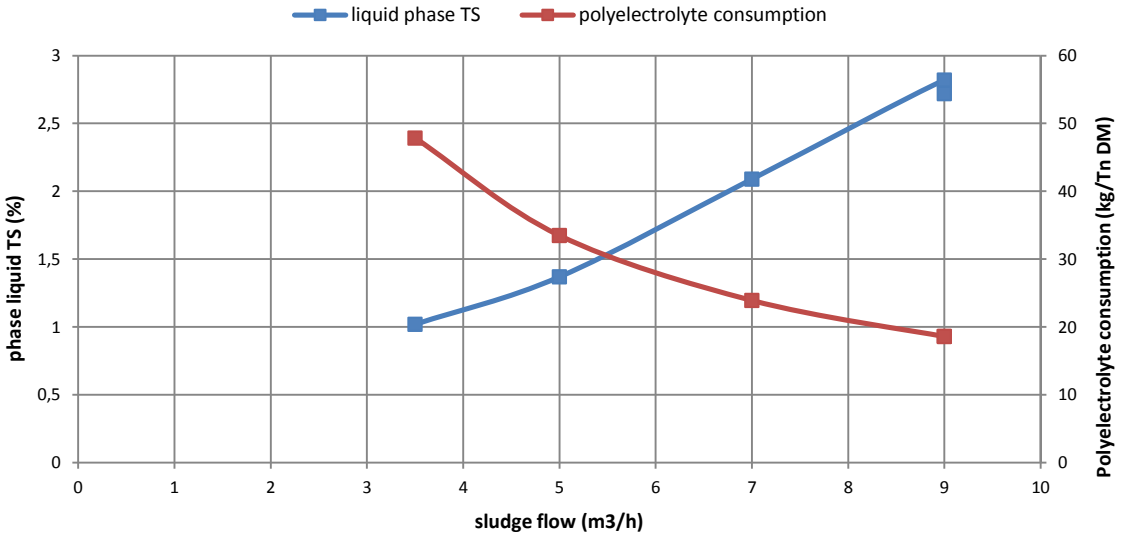



***Figure 2.5:*** *Liquid phase TS and polyelectrolyte FLOPAM EM 840 HIB consumption versus sludge flow (test 1)*

In this test liquid phase samples were taken and some parameters were determined. Table 2.4 shows the results obtained.

***Table 2.4:*** *Results obtained in the test 1analyses with flocculant FLOPAM EM 840 HIB*

| Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | COD (mg/L) | N-$NH_4^+$ (mg/L) | N-$NO_2^-$ (mg/L) | N-$NO_3^-$ (mg/L) | $N_{org}$ (mg/L) | $N_{total}$ (mg/L) | TS (%) | VS (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| 3500 | 5.0 | 40 | 13400 | 2680 | 2.8 | 34 | 50.7 | 2767.5 | 1.44 | 45.86 |
| 3500 | 7.0 | 40 | 19000 | 3120 | 5.5 | 50 | 479.5 | 3655 | 2.08 | 53.45 |
| 3500 | 9.0 | 40 | 35500 | 3950 | 10.0 | 96 | 141.5 | 4197.5 | 2.81 | 58.15 |
| 3500 | 9.0 | 60 | 30000 | 3550 | 8.5 | 82 | 314.5 | 3955 | 3.01 | 58.9 |

From the results obtained, we can observe that the higher the TS quantity, the higher are the COD concentrations and the forms of nitrogen. In order to obtain a PW with more optimal conditions for input to the WWTP, it is necessary to decrease the solids %. This is possible by lowering the sludge flow and increasing the polyelectrolyte consumption. However, it is necessary to take into account the space needs and economic cost due to higher energy and flocculant consumption.

### *B. Test 2*

This test is performed with a solid powder flocculant (FLOPAM TH 4650 VHM). In this test, two centrifuge stages are used, so the flocculant is divided between the two

stages. Tables 2.5 and 2.6 show the results obtained in the first and second stages respectively.

***Table 2.5:*** *Results obtained in test 2 with flocculant FLOPAM TH 4650 VHM in the first centrifuge stage*

| Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | Liquid phase TS (%) | Solid phase TS (%) | Consumption (kg/Tn MS) |
|---|---|---|---|---|---|
| 1100 | 8.1 | 25 | 3.23 | | 4.59 |
| 1100 | 8.1 | 25 | 4.59 | | 4.59 |
| 1100 | 8.1 | 25 | 4.01 | 30.1 | 4.59 |
| 1100 | 8.1 | 25 | 3.56 | | 4.59 |

***Table 2.6:*** *Results obtained in test 2 with flocculant FLOPAM TH 4650 VHM in the second centrifuge stage*

| Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | Liquid phase TS (%) | Solid phase TS (%) | Consumption (kg/Tn MS) |
|---|---|---|---|---|---|
| 102.7 | 6.5 | | 2.36 | | 2.05 |
| 513.5 | 6.5 | | 1.43 | 31.2 | 10.27 |
| 513.5 | 6.5 | | 2.28 | | 10.27 |
| 513.5 | 6.5 | | 2.05 | | 10.27 |

In the first stage, under equal conditions of polymer and sludge flow, several samples were taken at different times to determine the TS, obtaining values of 3.23 - 4.59 % TS, which are very high. However, in the second stage, values of 1.43 – 2.36 % TS with a total consumption (first and second stage) of 6.64 – 14.86 kg/Tn DM.

### *C. Test 3*

This test is performed with a solid cationic flocculant (FLOPAM TE 4650 SH). Table 2.7 shows the results obtained.

***Table 2.7:*** *Results obtained in test 3 with flocculant FLOPAM TE 4650 SH*

| Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | Liquid phase TS (%) | Solid phase TS (%) | Consumption (kg/Tn MS) |
|---|---|---|---|---|---|
| 2000 | 7.72 | 5 | 0.92 | 12.2 | 9.3 |
| 2000 | 8.8 | 7 | 0.76 | 15.01 | 9.3 |
| 1800 | 7.9 | 7 | 0.82 | 16.9 | 8.37 |
| 2000 | 9.2 | 9,1 | 0.59 | 17.01 | 9.3 |
| 2000 | 9.2 | 5 | 0.77 | 11.9 | 9.3 |

Figure 2.5 shows a graph of the sludge flow versus TS in the liquid phase and the polyelectrolyte consumption.

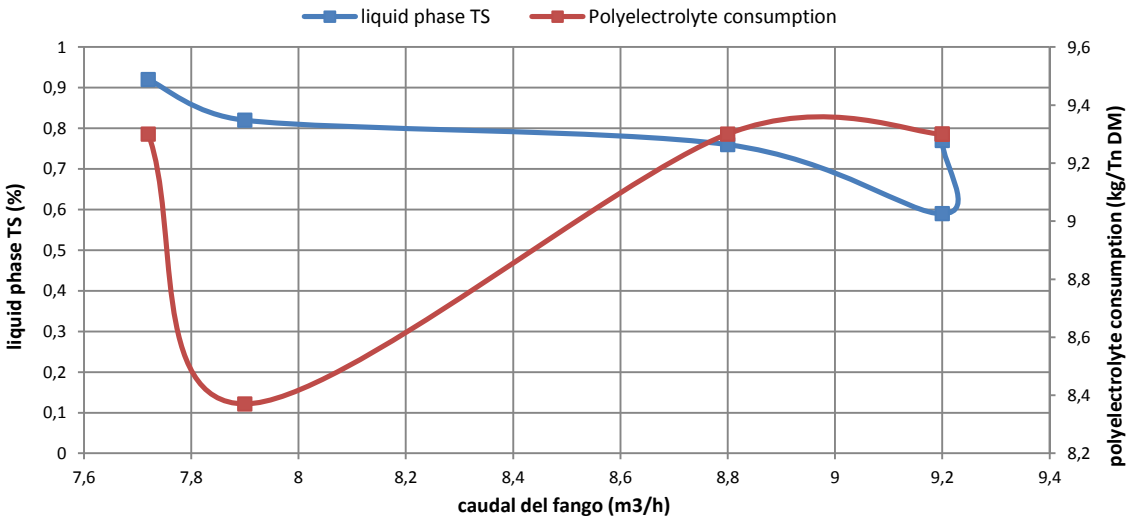



***Figure 2.6:*** *Liquid phase TS and polyelectrolyte FLOPAM TE 4650 SH consumption versus sludge flow (test 3)*

In this case there is no trend as clear unlike in test 1. However, it is the test with the best results obtained in liquid phase solids concentrations and flocculant consumption where at a flow of 9.2 $m^3$/h (similar to normal operation) we obtained concentrations of TS 0.59 - 0.77% in the liquid phase with relative velocities of 9.1 and 5 respectively and a consumption of 9.3 kg/Tn DM. Figure 2.7 shows a photograph of the liquid phase obtained in this test.

***Figure 2.7:*** *Liquid phase aspect obtained with flocculant FLOPAM TE 4650 SH (test 3)*

However, the test was repeated with this same flocculant several days later and very different results were obtained. Table 2.8 shows the results.

***Table 2.8:*** *Results obtained in a repetition of test 3 with flocculant FLOPAM TE 4650 SH*

| Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | Liquid phase TS (%) | Solid phase TS (%) | Consumption (kg/Tn MS) |
|---|---|---|---|---|---|
| 2100 | 8.6 | 5 | 2.06 | 30 | 7.4 |
| 2100 | 8.6 | 7 | 2.62 | 30 | 7.4 |
| 2100 | 8.6 | 8 | 2.16 | 30 | 7.4 |

At present a repeat of the test is pending to resolve the doubts with this flocculant.

D. Results comparative

Considering that, during to space requirements in the tanks the centrifuge flow must be 8 – 10 $m^3$/h, table 2.9 shows the results obtained in the tests that comply with this premise, as well as the values obtained in the normal centrifuge operation with Zetag 8185 and Hyfloc XT-653 that were used before beginning the tests, the sludge TS is also shown in order to calculate the obtained performance.

***Table 2.9:*** *Results comparative with different flocculants*

| Flocculant | Satges | Sludge TS (%) | Polymer flow (L/h) | Sludge flow ($m^3$/h) | Relative speed | Liquid phase TS (%) | Performance (*) | Consumption (kg/Tn DM) |
|---|---|---|---|---|---|---|---|---|
| ZETAG 8185 | 1 | 12.13-13.74 | 1000-1500 | 9.0 | 40 | 2.83-4.56 | 2.7-4.9 | 7.0 |
| | 2 | 12.37-13.24 | | | 20 | 2.22-2.90 | 4.6-5.6 | 7.0 |
| HYFLOC XT-653 | 1 | 11.91 | 2100 | 8.6 | 5 | 2.06 | 5.8 | 7.4 |
| FLOPAM EM 840 HIB | 1 | 13.66 | 3500 | 9.0 | 40 | 2.82 | 4.8 | 18.61 |
| | | | | | 60 | 2.72 | 5.0 | 18.61 |
| FLOPAM TH 4650 VHM | 2 | 14.02 | 1202.7 | 8.1 | 25 | 2.36 | 5.9 | 6.64 |
| | | | 1613.5 | | 25 | 1.43-2.28 | 6.1 -9.8 | 14.86 |
| FLOPAM TE 4650 SH | 1 | 11.91 | 2100 | 8.6 | 5 | 2.06 | 5.8 | 7.4 |
| | | | | | 7 | 2.62 | 4.5 | |
| | | | | | 8 | 2.16 | 5.5 | |

* Performance = % TS of input sludge / % TS of the liquid phase

Taking into account the TS % in the liquid phase, the best result were obtained with flocculant FLOPAM TH 4650 VHM with 1.43% and a consumption of 14.86 kg/Tn DM. However, this test was performed with a double centrifuge stage, which gives better results as will be seen in phase 3. To be able to compare this flocculant with the others a single stage centrifuge must be performed, or the other flocculants in two stages. It is possible, however, to compare it with the flocculant Zetag 8185, where it is observed that the new flocculant obtains better results but with a much higher consumption. In

this case it is necessary to evaluate if the cost increase in relation to the obtained improvement is justified.

If we consider only flocculants tested with a single stage, the best result is obtained with 2.06% TS in the liquid phase with the flocculant HYFLOC XT-653 and FLOPAM TE 4650 SH with the same flow, relative speed and consumption conditions, but as discussed in the previous point, we are still waiting to repeat test 3 due to having obtained very different results under similar conditions. In this case, the consumption of both flocculants is 7.4 kg/Tn DM, which is one of the lowest consumptions achieved so far.

We propose to repeat the tests with double centrifuge stage since it has been observed that the performance is superior, obtaining results < 3% TS compared to the previous ones to these test that were < 5% TS.

#### 2.2.2.2. PW tank sealing

There are 3 feed tanks in the WWTP area. Tank 1 receives the PW collected in tank 2 and takes it to the initial filtration, before the input to the WWTP. Tank 2 receives, normally, the liquid tank effluent from the anaerobic digestion area. Tank 3, normally, contains the PW from other areas. Tanks 2 and 3 are connected by an overflow.

On April 2014 it was observed that the levels in tanks 2 and 3 were not in line with the PW received, and that they had an extra contribution. It was suspected that there were infiltrations into the feed tanks, therefore we proceeded to perform a tank sealing testing.

To perform the test, the following flows were measured: input to the WWTP (F1), the anaerobic digestion area liquid tank (tank 2) (F2), the increase in volume in the tanks (F3) and, by differences, the PW from other areas (tank 3) (F4). Table 2.10 shows the results.

***Table 2.10:*** *Feed tanks sealing test*

| start date | start time | end date | end time | F1 | F2 | F3 | F4 |
|---|---|---|---|---|---|---|---|
| 23/04/2014 | 20:00 | 24/04/2014 | 8:00 | 5,41 $m^3/h$ | 3,50 $m^3/h$ | 0,67 $m^3/h$ | 2,58 $m^3/h$ |
| 24/04/2014 | 16:30 | 25/04/2014 | 8:30 | 5,25 $m^3/h$ | 3,58 $m^3/h$ | -0,43 $m^3/h$ | 1,24 $m^3/h$ |
| 25/04/2014 | 8:00 | 28/04/2014 | 8:00 | 4,95 $m^3/h$ | 3,50 $m^3/h$ | -0,18 $m^3/h$ | 1,27 $m^3/h$ |
| 29/04/2014 | 20:00 | 02/05/2014 | 8:00 | 5,43 $m^3/h$ | 3,50 $m^3/h$ | -,035 $m^3/h$ | 1,58 $m^3/h$ |
| 01/05/2014 | 11:00 | 02/05/2014 | 8:00 | 5,45 $m^3/h$ | 4,13 $m^3/h$ | 0,33 $m^3/h$ | 1,65 $m^3/h$ |
| 02/05/2014 | 8:00 | 05/05/2014 | 7:00 | 5,65 $m^3/h$ | 4,36 $m^3/h$ | 0,11 $m^3/h$ | 1,40 $m^3/h$ |
| 05/05/2014 | 7:00 | 07/05/2014 | 7:00 | 4,67 $m^3/h$ | 3,54 $m^3/h$ | 0,31 $m^3/h$ | 1,44 $m^3/h$ |

Note that the flow calculated in tank 3 (F4) was higher than expected, but could not be demonstrated. Therefore, we proceeded to perform a second test: to determine the flow into the evaporator (F5) capturing the tank 3 PW. Table 2.11 shows the results obtained.

***Table 2.11:*** *Feed tank 3 sealing test*

| start date | start time | end date | end time | F5 | F2 | F4 | difference |
|---|---|---|---|---|---|---|---|
| 02/05/2014 | 12:00 | 07/05/2014 | 7:00 | 80 $m^3$ | 7 $m^3$ | 55 $m^3$ | 18 $m^3$ |

The evaporator treated 80 $m^3$, of which 55 were from tank 3 and 7 were from the overflow of tank 2 to tank 3. This gives a difference of 18 $m^3$ which is unaccounted for. In conclusion it was determined that the tank 3 has an infiltration, so it needed to be emptied and repaired.

To solve this, we proposed to empty the tanks and to cover them with impermeable material. Due to the space required to retain the PW, in May 2017 this measure was started.

#### 2.2.2.3. PW tanks homogenisation

It is known that, compared to biological treatment, it is essential to avoid abrupt changes, and give sufficient time to allow the system to adapt to any change.

The PW properties fed to the WWTP depends on several factors: the process undergone, the source from which they come, the quantity treated, etc. These are factors that we are unable to control because the USW (Urban Solids Waste) received in the Ecoparc 2 have seasonal changes in both quantity and type.

Therefore, it was decided that the best point to act was in the WWTP feed tanks. By homogenising the tanks input to the WWTP by agitation, the PW properties become more constant, and changes that require a rapid response in the biological treatment are avoided.

### *2.2.3. Biological purification process improvement*

Once the analysis procedures and the feed to WWTP have been studied, we have reviewed the biological process to improve the WWTP performance and thus improve the discharge quality.

### 2.2.3.1. Control parameters

We introduced several control parameters calculated from the analyses, so as to make good use of the results obtained and interpret the data. In this way we are able to observe changes depending on the PW introduced. An example is the COD/N ratio, which can be observed if a good proportion holds in the nutrients to the WWTP biology, although the PW may have fluctuations.

In order to determine the control parameters, the WWTP has been assumed to be a biological reactor comprising of the nitrification, denitrification and combined tanks and a decanter which in this case is ultrafiltration. Figure 2.8 shows a schematic of the system where Q refers to the flow, X to the VSS content and S to the organic mater content (COD).

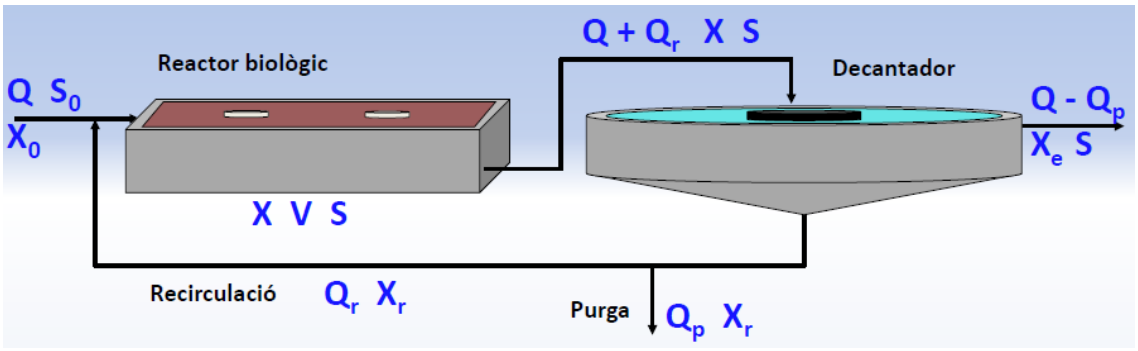



***Figure 2.8:*** *WWTP system considered for control parameter calculation*

The control parameters considered are:

- *Nutrients COD:N:P* → normally $BOD_5$:N:P is determined to be 100:5:1 respectively but since the $BOD_5$ is not analysed the ratio calculated is COD:N:P.

$$A = \frac{COD\ (mg\ O_2/L)}{P_{total}\ (mg\ P/L)} \quad ; \quad B = \frac{N_{total}\ (mg\ N/L)}{P_{total}\ (mg\ P/L)} \quad ; \quad \boxed{A:B:1}$$

- *Mass load ($C_m$)* → ratio between the input COD amount per unit time and the microorganisms mass in the reactor.

$$C_m\left(\frac{kg\ COD}{kg\ VSS \cdot day}\right) = \frac{Q\ (m^3/day) \cdot S_0\ (kg\ COD/m^3)}{V\ (m^3) \cdot X\ (kg\ VSS/m^3)}$$

- COD load ($C_{COD}$) → daily input COD amount.

$$C_{COD}(kg\ COD/day) = \frac{COD_{leachate}\ (mg\ O_2/L) \cdot Q\ (m^3/day)}{1.000}$$

- Ammonium load ($C_{ammonium}$) → daily input N-$NH_4^+$ amount.

$$C_{ammonium}(kg\ N/day) = \frac{N-NH_4^+{}_{leachate}\ (mg\ N/L)\cdot Q\ (m^3/day)}{1.000}$$

- *Volumetric load ($C_v$)* → ratio between the input COD amount per unit time and reactor volume.

$$C_v(kg\ COD/day) = \frac{Q\ (m^3/day)\cdot S_0\ (kg\ COD/m^3)}{V\ (m^3)}$$

- *COD performance ($\eta_{COD}$)* → ratio between eliminated organic mater and input organic mater.

$$\eta_{COD}\ (\%) = \frac{COD_{leachate}\ (mg\ O_2/L) - COD_{permeate}\ (mg\ O_2/L)}{COD_{leachate}(mg\ O_2/L)}\cdot 100$$

- *N performance ($\eta_N$)* → ratio between eliminated nitrogen and input nitrogen.

$$\eta_N\ (\%) = \frac{N_{total_{leachate}}\ (mg\ N/L) - N_{total_{permeate}}\ (mg\ N/L)}{N_{total_{leachate}}\ (mg\ N/L)}\cdot 100$$

- *Denitrification grade (DG)* → ratio between biologic reactor input ammonium and output nitrates.

$$DG\ (\%) = \frac{N-NH_4^+{}_{input}\ (mg\ N/L) - N-NO_3^-{}_{output}\ (mg\ N/L)}{N-NH_4^+{}_{input}\ (mg\ N/L)}\cdot 100$$

- *Hydraulic Residence Time (HRT)* → calculates how long the PW remains inside the reactor.

$$HRT\ (h) = \frac{V_{reactor}\ (m^3)}{Q\ (m^3/h)}$$

- *Cellular Residence Time (CRT)* → ratio between reactor sludge mass and extracted sludge mass (purge) per unit of time.

$$CRT\ (days) = \frac{V\ (m^3)\cdot X\left(\frac{kg\ VSS}{m^3}\right)}{Q_p\left(\frac{m^3}{day}\right)\cdot X_r\left(\frac{kg\ VSS}{m^3}\right) + \left(Q\left(\frac{m^3}{day}\right) - Q_p\left(\frac{m^3}{day}\right)\right)\cdot X_e\left(\frac{kg\ VSS}{m^3}\right)}$$

- *Sludge Volume Index (SVI)* → the volume (mL) occupied by SS 1 g after decanting for 30 minutes in a conus Imhoff.

$$V_{30} = \frac{V_{sludge}\ (mL)}{V_{total}\ (L)} \quad ; \quad IVS\ (mL/g) = \frac{V_{30}\ (mL/L)}{X\ (g/L)}$$

It was not possible determine this parameter due to the large amount of suspended solids contained the Ecoparc 2 PW, we can see this in figure 2.9.

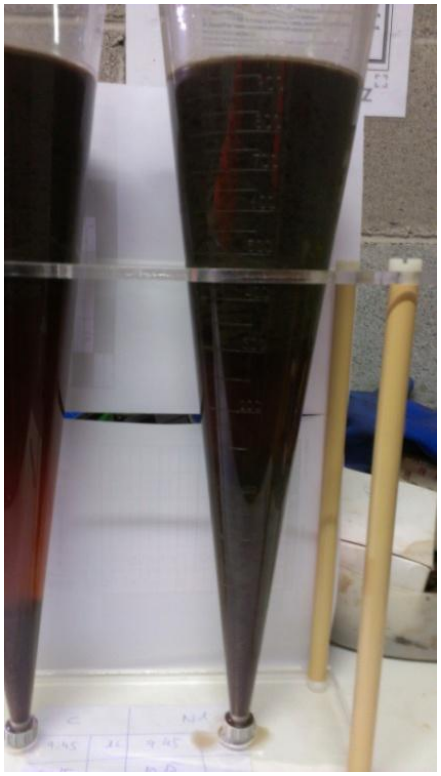


***Figure 2.9:*** *Test to determine the SVI control parameter*

- *Biology inhibition* → it is necessary to take into account the ammonium and nitrites concentrations since they can inhibit the nitrobacters and nitrosomonas bacteria as a function of medium pH. Figures 2.10 and 2.11 show the inhibition graphs by ammonia and nitrous acid respectively.

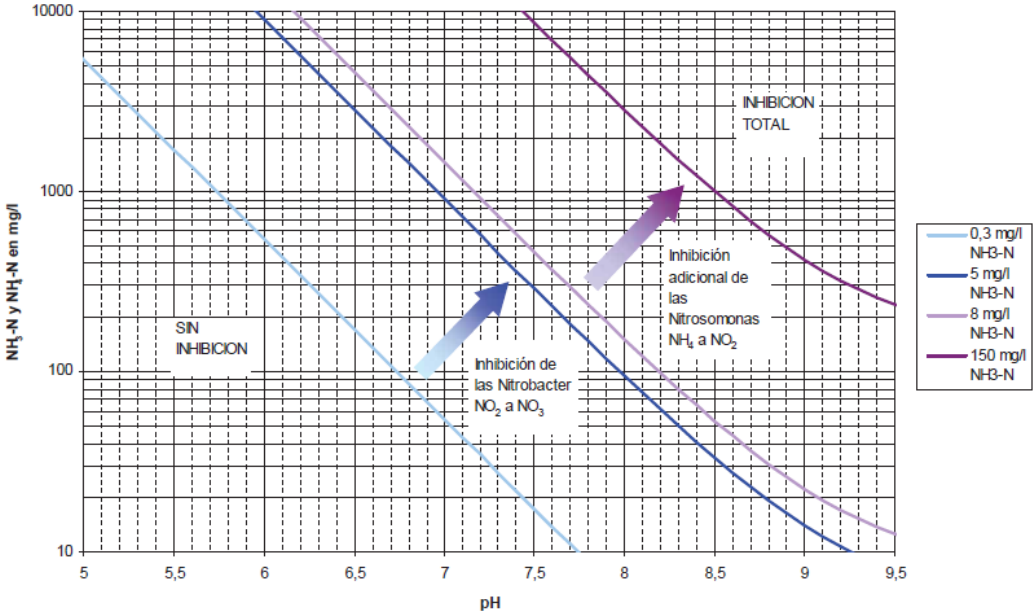



***Figure 2.10:*** *Nitrobacter and nitrosomonas inhibition due to ammonium concentration (image author: Wehrle Medio Ambiente)*

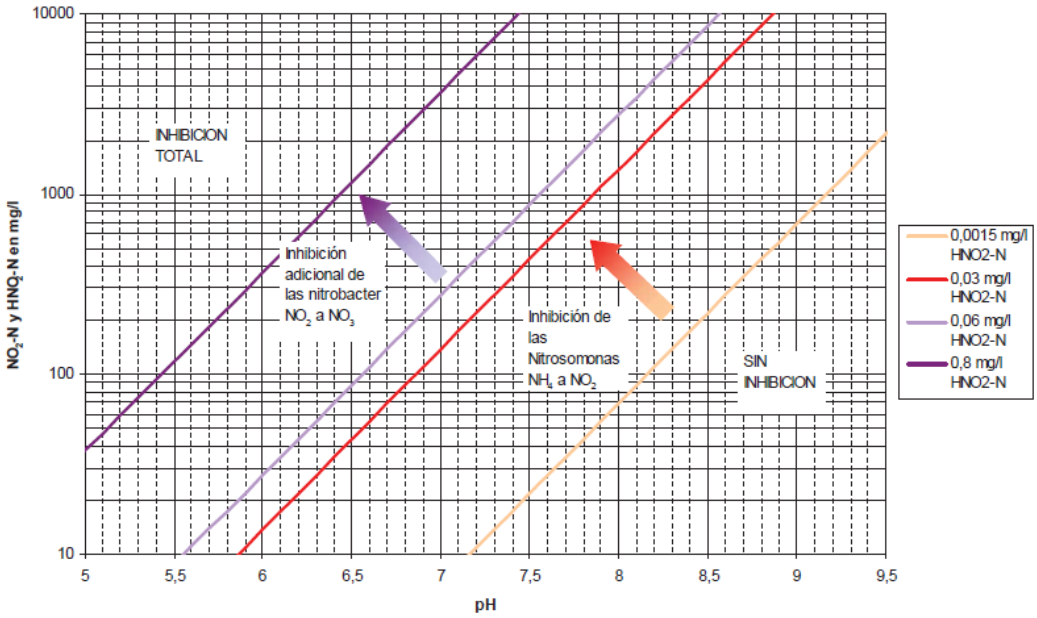



***Figure 2.11:*** *Nitrobacter and nitrosomonas inhibition due to nitrite concentration (image author: Wehrle Medio Ambiente)*

### 2.2.3.2. Chemical dosing

In the cases where there is a WWTP destabilisation chemical products can be used. When this happens, it is necessary to state which chemicals need to be dosed by specifying the parameter that can be adjusted and how to calculate the dose to be introduced.

- *Phosphoric acid:* helps to adjust the nutrient proportion by a good purification ($BOD_5$:N:P → 100:5:1). In the Ecoparc 2 process the nutrient that is always in excess is the N, and it is possible to regulate the phosphorus with phosphoric acid dosage and $BOD_5$ with methanol dosage if necessary. To determine the required phosphoric acid dose we take as a reference PW total nitrogen and a 5:1 ratio with total phosphorus. The way we have calculated the phosphoric acid required is:

- Calculate the real proportion between PW total nitrogen and total phosphorus (A).

$$A(mg\ N/mg\ P) = \frac{N_{total}\ (mg\ N/L)}{P_{total}\ (mg\ P/L)}$$

If A ≤ 5 a dose is not required.

If A > 5 go to the next point.

- Calculate the proportion that needs to be contributed (B).

$$B\ (mg\ P/L) = \frac{N_{total}\ (mg\ N/L)}{5} - P_{total}\ (mg\ P/L)$$

- Calculate the flow at which the phosphoric acid must be dosed (C), knowing that:

PM (P) = 30,97 g/mol ; PM ($H_3PO_4$) = 98,00 g/mol ; p = commercial phosphoric acid purity (g $H_3PO_4$ / 100 g comercial phosphoric acid) ; ρ = commercial phosphoric acid density (g/L)

$$C\ (L/h) = \frac{B\ (mg\ P/L) \cdot Q_{leachate}\ (m^3/h)\ \cdot\ 98{,}00/30{,}97}{p\ \cdot \rho}$$

Equation:

$$C\ (L/h) = \frac{\left(N_{total}\ (mg\ N/L) - 5\ \cdot\ P_{total}\ (mg\ P/L)\right) \cdot Q_{leachate}\ (m^3/h)\ \cdot\ 98{,}00/30{,}97}{5 \cdot\ p\ \cdot \rho}$$

- *Sodium hydroxide:* the nitrification reactions produce $H^+$ ions which implies a acidification of the medium. Thanks to the alkalinity contained in the PW, it is possible to maintain the pH at the appropriate values. But sometimes the alkalinity is not sufficient to cope with it. In these cases it is necessary to dose with $OH^-$ ions (for example NaOH) to compensate the protons excess and avoid a quick pH decrease, which would produce an inhibition of the biology and destabilization of the WWTP. The way we have calculated the sodium hydroxide required is:

We know from Wehrle, the engineering company which designed the WWTP, that a biological treatment disposes of sufficient alkalinity when the system output shows a residual alkalinity greater than 5 mmol HCl/L.

- Calculate the required alkalinity (A).

$$A = \frac{N - NH_4^+{}_{input}\ (mg\ N/L) - N - NO_3^-{}_{output}\ (mg\ N/L)}{14.01\ (mg\ N/mmol)} \cdot \frac{1\ mmol\ HCl}{1\ mmol\ N} + 5\ mmol\ HCl/L$$

If A ≥ alkalinity, not dosing is required.

If A < alkalinity go to the next point.

- Calculate the alkalinity needed to be supplied to the system (B).

$$B\ (mmol\ HCl/L) = A\ (mmol\ HCl/L)\ -\ alkalinity\ (mmol\ HCl/L)$$

- Calculate the concentration at which it is equivalent (C) knowing that:

PM (NaOH) = 40,00 g/mol

$$C\ (mg\ NaOH/L) = B\ (mmol\ HCl/L) \cdot \frac{1\ mmol\ NaOH}{1\ mmol\ HCl} \cdot \frac{40{,}00\ mg\ NaOH}{1\ mmol\ NaOH}$$

- Calculate the flowrate at which the sodium hydroxide needs to be dosed (D), knowing that:

p = comercial sodium hydroxide purity (g NaOH / 100 g comercial sodium hydroxide) ; ρ = comercial sodium hydroxide density (g/L)

$$D\ (L/h) = \frac{C\ (mg\ NaOH/L) \cdot Q_{leachate}\ (m^3/h)}{p\ \cdot \rho}$$

Equation:

$$D\ (L/h) = \frac{\left(\frac{N - NH_4^+{}_{input} - N - NO_3^-{}_{output}}{14.01} + 5\ -\ alkalinity\right) \cdot 40{,}00 \cdot Q_{leachate}}{p\ \cdot \rho}$$

In December 2015 we observed that in spite of dosing NaOH no process improvement was obtained. After several checks it was detected that the pump that was being used for the NaOH dosing was the one designed for the phosphoric acid dosifing. For this reason the NaOH was being introduced into the denitrification tank.

As we saw in the introduction of this phase, the reaction that occurs in the denitrification tank is:

$$4\ NO_3^- + 4\ H^+ + 5\ [C] \xrightarrow{facultative\ heterotropic\ bacteries} N_2 + 5\ CO_2 + 2\ H_2O$$

Added NaOH reacts with the $H^+$ ions, avoiding that the denitrification reaction takes place. This causes an additional disadvantage to the purification process. As a result of this observation, a pump conduit to the nitrification tank 1 was installed to allow for the sodium hydroxide to be dosed correctly.

- *Methanol:* a carbon source is necessary in the anoxic reactors to perform denitrification. This carbon source is obtained from the sludge COD, but as the wastewater treatment progresses, the available COD becomes depleted. This requires an external carbon to be supplied, in this case methanol, to reduce nitrates to nitrogen gas ($N_2$).

  The methanol dosage has always been made based on action-reaction principles (introduction/response). What is proposed here is based on actual WWTP metering and current status. From stoichiometric calculations the methanol amount necessary to achieve our purpose can be determined, without exceeding the methanol consumption. A methanol excess implies a greater COD removal before discharge. A calculation of the required methanol dose thus allows performance to be optimised and minimises the cost of both consumables and in the process. The way we have calculated the methanol required is as follows:

We know from Wehrle, the engineering company which designed the WWTP, that:

Each methanol kg is equal to 1.5 $BOD_5$ kg ; each N-$NO_3$ kg to denitrify needs $BOD_5$ 4 kg ; $BOD_5$/COD ratio at Ecoparc 2 is approximately 0.4

- Calculate the nitrates generated in nitrification (A).

$$A\ (kg\ N-NO_3^-/h) = \frac{Q_{leachate}\ (m^3/h) \cdot N-NH_4^+{}_{input}(mg\ N/L)}{1000}$$

- Calculate the $BOD_5$ necessary to denitrify to a certain denitrification degree GD (B).

$$B\ (kg\ BOD_5/h) = A\ (kg\ N-NO_3^-/h) \cdot \frac{4\ kg\ BOD_5}{1\ kg\ N-NO_3^-} \cdot GD$$

- Calculate the $BOD_5$ present in the PW (C).

$$C\ (kg\ BOD_5/h) = \frac{Q_{leachate}\ (m^3/h) \cdot COD_{input}(mg\ O_2/L)}{1000} \cdot \frac{0{,}4\ kg\ BOD_5}{1\ kg\ COD}$$

- Calculate the BOD5 which is necessary to provide externally (D).

$$D\ (kg\ BOD_5/h) = B\ (kg\ BOD_5/h) - C\ (kg\ BOD_5/h)$$

- Calculate the metanol amount to be dosed (E), knowing that:

p = comercial metanol purity (g methanol / 100 g comercial methanol) ; ρ = comercial metanol density (g/L)

$$E\ (L/h) = \frac{D\ (kg\ BOD_5/h) \cdot \frac{1\ kg\ methanol}{1{,}5\ kg\ BOD_5} \cdot 1000}{p \cdot \rho}$$

Equation:

$$E\ (L/h) = \frac{Q_{leachate} \cdot (N - {NH_4}^+_{input} \cdot 4 \cdot GD - COD_{input} \cdot 0.4)}{1.5 \cdot p \cdot \rho}$$

### 2.2.3.3. Purge

In the activated sludge process there are some key parameters such as the Bacteria Growth Speed (BGS), the Hydraulic Residence Time (HRT), the Cell Residence Time (CRT), recirculation, and purge.

An inconsistency in these parameters may involve the destabilisation of the correct WWTP operation, as shown in figure 2.12:

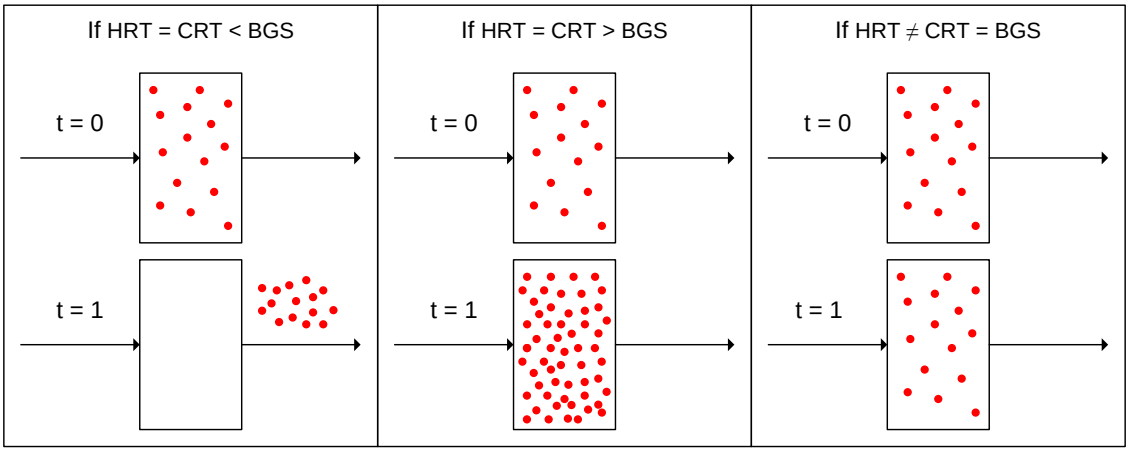



***Figure 2.12:*** *Relation between HRT, CRT and BGS (image author: Eloísa Albacete García)*

1. If HRT = CRT < BGS → all biology from reactor will be lost
2. If HRT = CRT > BGS → many bacteria will be available, these bacteria will not have sufficient nutrients and will end up dying, causing a solids excess that make it difficult for the bacteria to live.

3. If HRT ≠ CRT = BGS → bacteria content in the reactor remains constant and the process has good results.

In summary, it is necessary to maintain the CRT = BGS. This is achieved by recirculating and purging.

*A. Purge calculation*

Increased CRT is achieved through the recirculation, while at the same time purging the excess bacteria and solids, that hinder the biological process.

To determine the sludge amount that must be purged, we require to know the sludge input and output (HRT) and the dry matter generated leading to the COD removal, nitrogen removal, as by ISS (Inert Solids in Suspension) introduced. We calculate the sludge amount to be purged from the following equation:

We know from Wehrle, the engineering company which designed the WWTP, that: the growth rate of COD eliminator bacteria is: 0,15 – 0,25 kg DM / kg COD removed ; the growth rate of N eliminator bacteria is: 0,05 – 0,10 kg DM / kg N removed ; ISS accumulation in the wastewater is: 1 kg DM / kg ISS

- Calculate the eliminated COD (A).

$$A\ (kg\ COD/h) = \frac{Q_{leachate}(m^3/day) \cdot (COD_{input}(mg\ O_2/L) - COD_{output}(mg\ O_2/L))}{1000}$$

- Calculate the COD provided by methanol (B), knowing that:

p = comercial metanol purity (g methanol / 100 g comercial methanol) ; ρ = comercial metanol density (g/L) ; 1 kg methanol ≈ 1 kg COD

$$B\ (kg\ COD/day) = \frac{Q_{methanol}(L/h) \cdot 24 \cdot \rho \cdot p}{1000}$$

- Calculate the total COD eliminated (C).

$$C\ (kg\ COD/day) = A\ (kg\ COD/day) + B\ (kg\ COD/day)$$

- Calculate the DM generated by COD elimination (D).

$$D\ (kg\ MS/day) = C\ (kg\ COD/day) \cdot \frac{0{,}2\ kg\ DM}{1\ kg\ COD}$$

- Calculate the ammonium removed (E).

$$E\ (kg\ N/day) = \frac{Q_{leachate}(m^3/day) \cdot (N - {NH_4}^+_{input}(mg\ N/L) - N - {NH_4}^+_{output}(mg\ N/L))}{1000}$$

- Calculated the DM generated by ammonium removed (F).

$$F\ (kg\ MS/day) = E\ (kg\ N/day) \cdot \frac{0{,}075\ kg\ DM}{1\ kg\ N}$$

- Calculate the ISS introduced into the reactor (G).

$$G\ (kg\ ISS/day) = Q_{leachate}(m^3/day) \cdot ISS\ (g/L)$$

- Calculate the DM generated by the ISS introduced (H).

$$H\ (kg\ MS/day) = G\ (kg\ ISS/day) \cdot \frac{1\ kg\ DM}{1\ kg\ ISS}$$

- Calculate the total DM generated (I).

$$I\ (kg\ MS/day) = D\ (kg\ MS/day) + F\ (kg\ MS/day) + H\ (kg\ MS/day)$$

- Calculate the theoretical purge taking into account the DM inside the reactor (J).

$$J\ (m^3/day) = \frac{I\ (kg\ MS/day)}{DM\ (g\ MS/L)}$$

Equation:

$$J = \frac{Q_{PW} \cdot \left(\frac{COD_{in} - COD_{out}}{1000} \cdot 0.2 + \frac{N - NH_4^+{}_{in} - N - NH_4^+{}_{out}}{1000} \cdot 0.075 + ISS\right) + \frac{Q_{metanol}(L/h) \cdot 24 \cdot \rho \cdot p}{1000} \cdot 0.02}{DM}$$

*B. Continuous purge*

As already mentioned, this is a biological process and sudden changes can alter the process. For this reason it is interesting to consider a continuous purge to keeps the solids proportion stable and provide a more homogeneous process. As a reference, the results of USW (Urban Solid Waste) treatment plant in Madrid (*las Dehesas*), where a continuous purge is used, shows that they succeeded in stabilising the process with regards to dry matter.

### 2.2.3.4. Combined tank operation

The combined tank has the peculiarity of being able to operate as a denitrificator or nitrificator. The system for regulating as one or the other consists of three parameters:

- *Methanol operation time* → regulates the time in minutes that methanol must be dosed at the indicated flow.
- *Air operation time* → regulates the time in minutes to be aerated inside the tank.
- *Pause time* → regulates the time in minutes that operations must be stopped (methanol dosing and aeration).

Figure 2.13 shows the sequence that the tank follows.

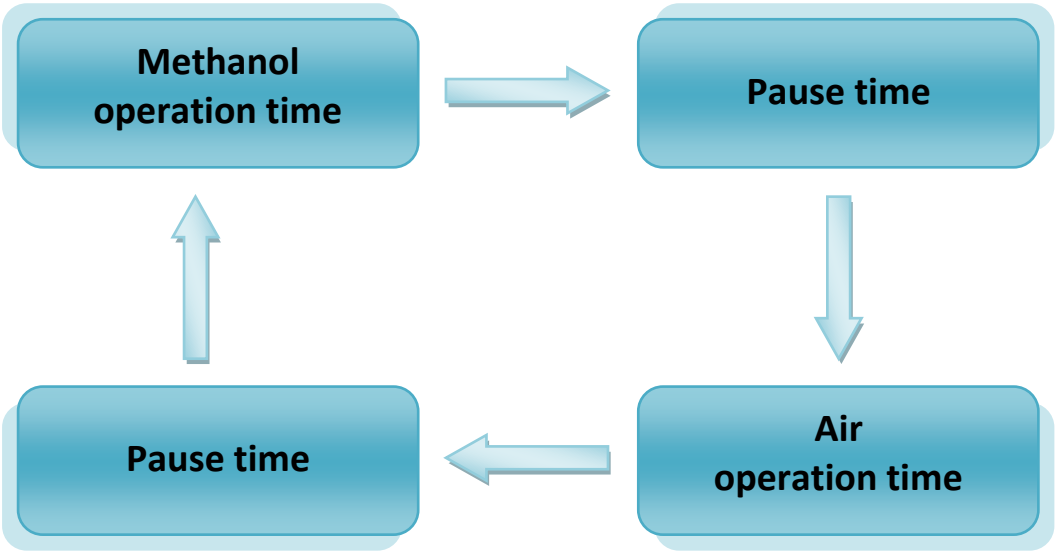



***Figure 2.13:*** *Combined tank sequence (image author: Eloísa Albacete García)*

First, in “methanol operation time”, methanol is used as the carbon source to carry out the denitrification reactions by reducing $NO_3^-$ to $N_2$. Next, a “pause time” is given to exhaust the methanol and proceed to aeration, where oxic aerobic conditions are present to oxidize the ammonium to nitrites and the nitrites to nitrates. Again a “pause time” is carried out to exhaust the dissolved oxygen and to obtain anoxic conditions before redosing methanol.

At the beginning of the Project, the combined tank practically always worked as a nitrificator. Sometimes it was actually operated as a combination with the following parameters:

- Methanol operation time = 20-25 minutes
- Pause time = 5 minutes
- Air operation time = 20 minutes

With such small time periods it is difficult to achieve optimum oxic/anoxic conditions for biological reactions.

It is also true that a good performance has been observed in the ammonium and COD reduction, but the nitrates concentration limits the process, so it is more favourable that the combined reactor works as a denitrificator or with superior denitrification times to help the total nitrates reduction. At present, the parameters are established at:

- Methanol operation time = 60 minutes
- Pause time = 10 minutes
- Air operation time = 60 minutes

And in case the nitrate value is not low enough, the pause and air operation times parameters are left at 0, so that the tank is always functioning as a denitrator.

## 2.3. Changes made in the plant during the thesis

Throughout this phase different changes have occurred that give a process improvement and, therefore, a performance improved of the WWTP at Ecoparc 2.

### *2.3.1. WWTP area project upgrading*

As a result of several changes over the time the plant has been operating, different projects have been carried out for the same area. The WWTP project has been unified into one considering the following points:

- Operation manuals: the WWTP expansion (nitrator 2), modifications (vibrating filters, etc) and the evaporator have taken into account.
- Working instructions: this is the work plan, under normal conditions, to be carried out in the WWTP area.
- Analytical monitoring: sampling points description, analyses to be performed, their frequency and the established analytical procedures.
- Drawings: the drawings have been updated eliminating all the equipment that no longer exists and adding new ones, and the tubes that connect them.
- Listings: lists all the equipment, valves, sensors, pumps and existing pipelines in the WWTP area.
- Equipment documentation: a compilation of all the technical documentation of equipment, valves and sensors that are currently working.

### *2.3.2. Analytical*

Throughout this phase many important changes have been made regarding the analytics that have allowed for a greater knowledge about the process and a better control of the WWTP state. To reach this point the following has been considered:

- Sampling points: the samples collection points have been established so that the results obtained at the same point can be compared with the historical, bibliographic data and other similar WWTP dates.
- Intermediate reactors: before beginning the project WWTP PW and permeate were being analysed. Only DM and pH were determined in the reactors. Therefore, the WWTP effectively worked as a “black box” where only the input and output were known without having information about the conditions occurring inside the process. Any failure in the process could only be detected by alterations in the reactor pH and high parameters in the permeate.

  At present, the COD and nitrogen forms are analysed within each reactor, so that any alteration can be detected in each individual reactors and acted directly on. Moreover, it facilitates the visualization of whether or not the reactors are operating correctly without waiting for permeate values to be affected.
- Analytical Procedures: Procedures have been modified by standarised methods, so that the results are more reliable and comparable to bibliographic data where necessary.
- New analytics: new analytics have been incorporated for a greater process control, obtaining useful information to be able to act quickly in case of a destabilisation of the biological purification process.
- Analytical frequency: the analytical planning has been updated in all departments involving necessity criteria.
- Data transmission: the speed of transmission of the analytical dates to Excel have been increased and can be visualised by the different departments involved to control the proper biological process functioning. Coloured indicators have been incorporated that make the small alterations in the different parameters more visible.
- WWTP state scheme: a scheme with the WWTP state has been incorporated into the Excel in the summary of the average weekly parameters, with alerts, so as to facilitate the WWTP state visualisation and warn if there is any altered parameter (figure 2.14). This scheme is updated automatically with the date in the moment that it is visualised. Also, the control parameters (figure 2.15) and the possible biology inhibition appear (figures 2.16 and 2.17).

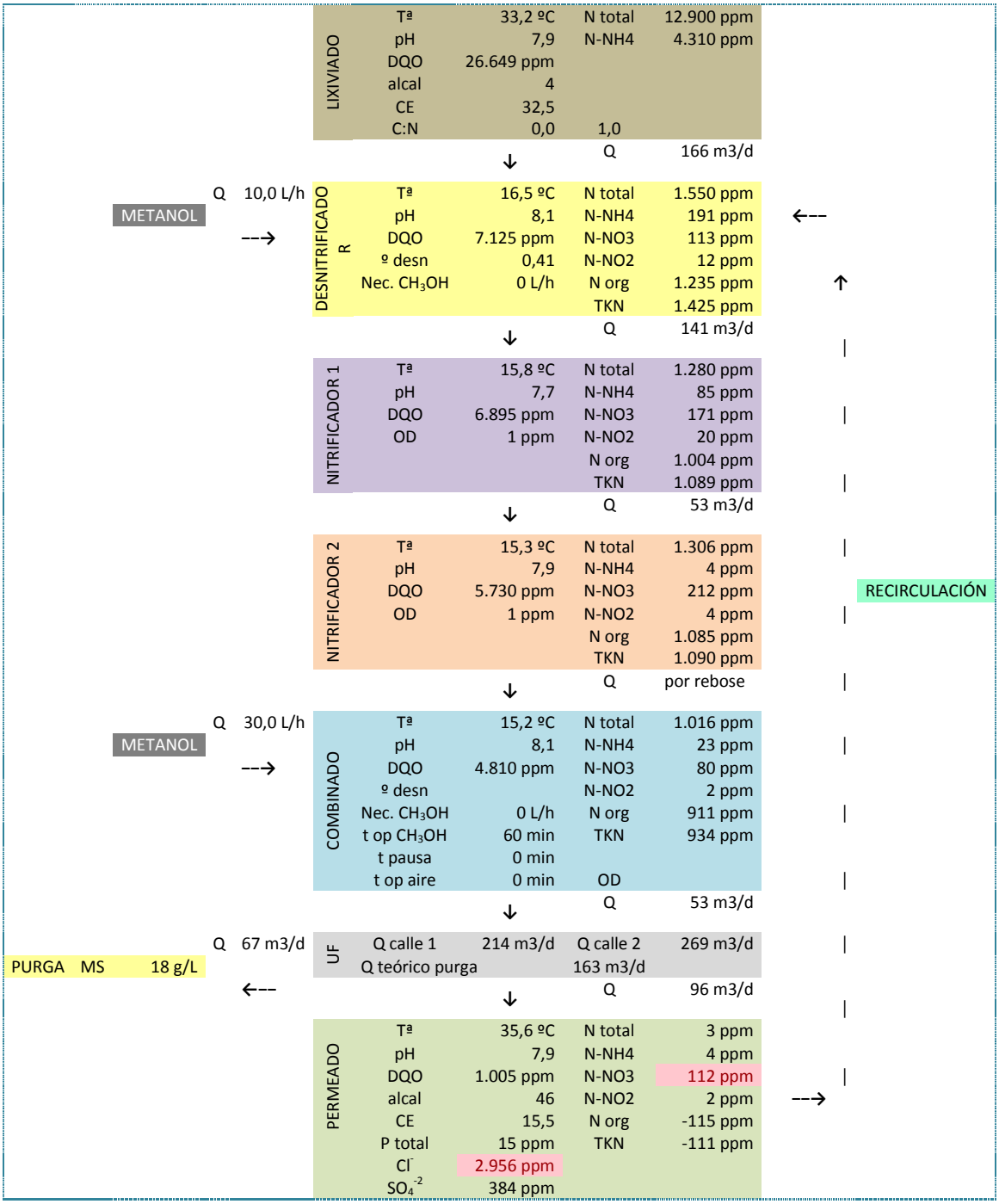



***Figure 2.14:*** *WWTP state scheme at Ecoparc 2 (part 1) – analytical parameters*

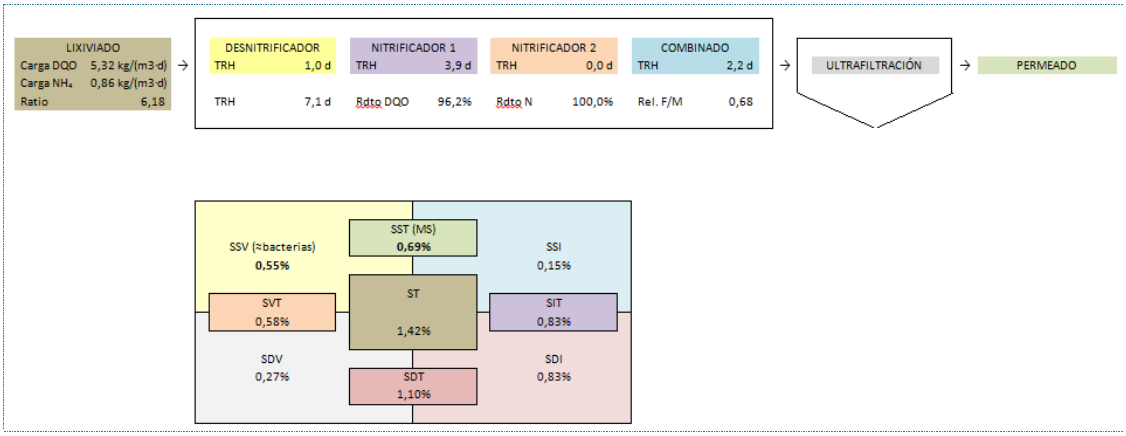



***Figure 2.15:*** *WWTP state scheme at Ecoparc 2 (part 2) – control parameters*

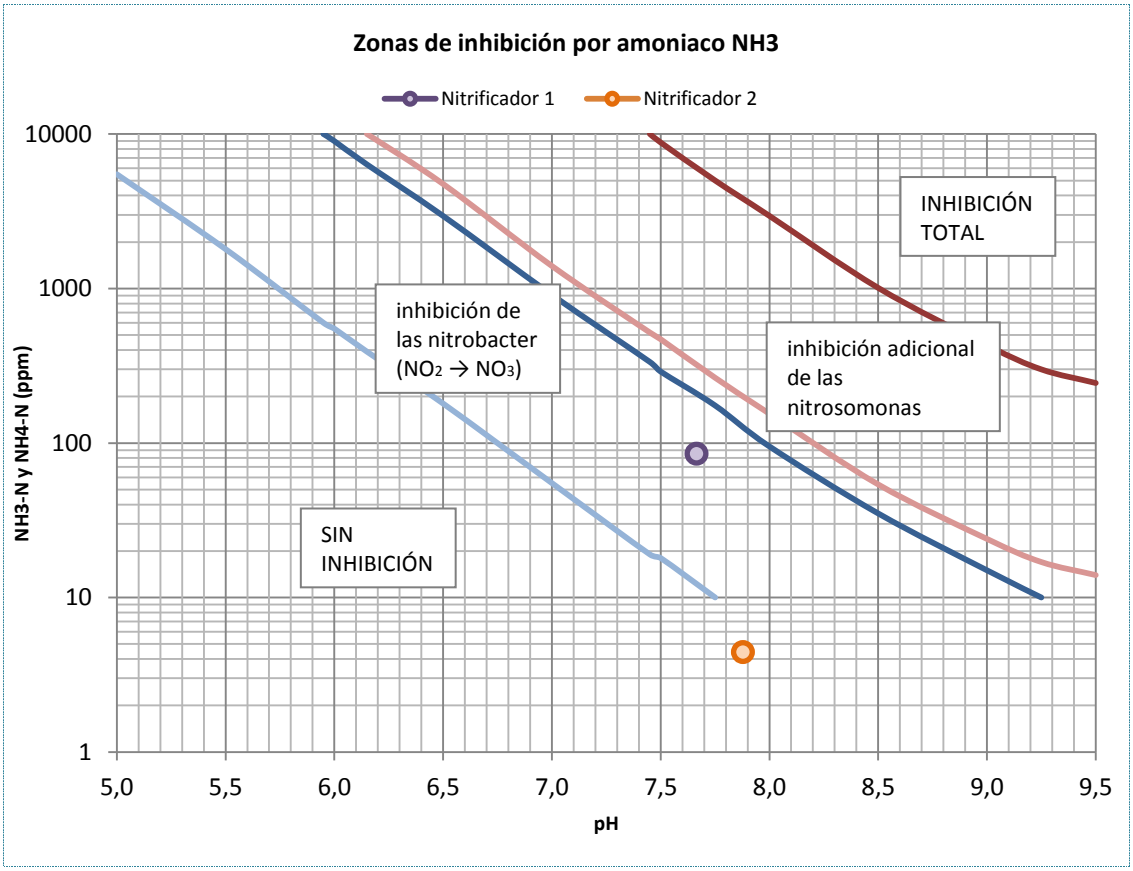



***Figure 2.16:*** *WWTP state scheme at Ecoparc 2 (part 3) – biology inhibition by ammonia (ammonium)*

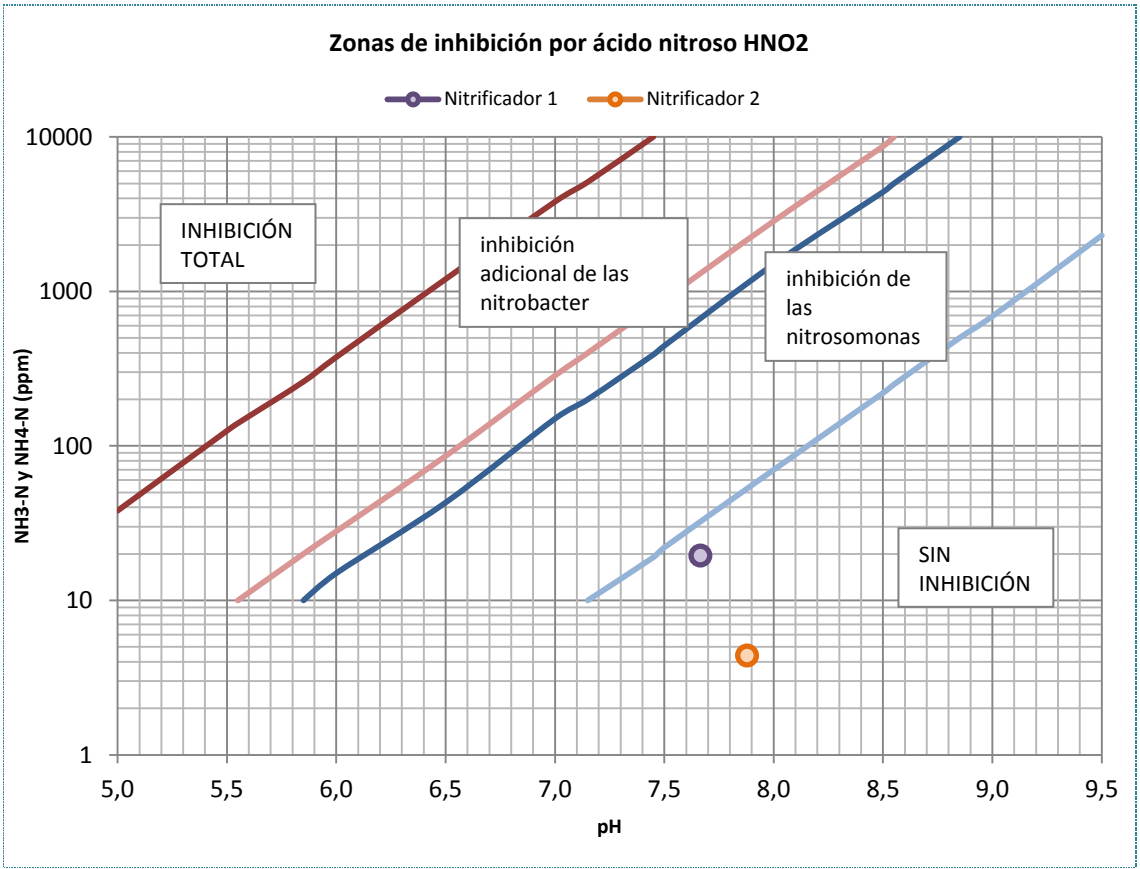



***Figure 2.17:*** *WWTP state scheme at Ecoparc 2 (part 4) – biology inhibition by nitrous acid (nitrites)*

- Formation: staff has been trained bythe engineering company which designed and built the WWTP,. This training facilitates the understanding of the data and the calculations that are required to be performed. Also, the laboratory staff has been trained to perform minimise analytical errors (dilutions, material use, good zone state, etc). In the near future a course will be given on analytical techniques in the Ecoparc 2 installations for the laboratory personal.
- Analysis Procedures Guide: a guide has been written for the laboratory staff where all necessary information is collected about the analyses to be carried out in all samples at Ecoparc 2 (not only the WWTP). The points that are taken into account in the guide are:
    - *Periodic analytics* → summarises the analytics and periodicity to be performed in each area: Pretreatment, Anaerobic digestion, Composting, Air treatment and WWTP
    - *Analysis procedures* → specify the instruccions for performing the different analytic phases.

- Reagents preparation. In the case solution from standards for acid-base titrations need to be preapred.
- Sampling. Indicates how to perform each sample and specific points.
- Sample preparation. In case of prior preparation as well as aqueous dilutions or extractions.
- Analytical procedures. Santandardised Work Procedures (PNT) applied to Ecoparc 2 have been drafted, which are followed wherever standardised analytical methods are possible.

- *Summary table:* A table has been made which indicates for each sample and analysis to make the references of: person responsible for analytics, periodicity, determinations number (if necessary in triplicate or not), necessary reagents, sampling, sample preparation and procedure (PNT).

- Analytical timetable: an analysis timetable has been carried out with the previous dates.
- Reagents, material and laboratory equiments: inventory of reagents, materials and laboratory equipments required to carry out the analytics mentioned.
- Calibrations: details the equipment to be calibrated, the periodicity and the method.
- Stock control: taking into account the consumption speed and consumables delivery times, a weekly stock register with indicators has been made to determine the need to order new material on time.

### *2.3.3. WWTP feed*

Studies have been carried out to deplete the flocculant entering the WWTP and obtain a more stable PW to avoid destabilisation in the biological process, increase its performance, avoid infiltration and homogenize the input to the WWTP.

#### 2.3.3.1. Flocculant performance improvement

Several tests have been performed with different flocculants in order to: optimise their performance, obtain a liquid phase with lower solids concentration, and deplete flocculant so that it does not reach the WWTP creating viscosity problems and a decrease in $O_2$ transfer. For this, the TS % has been determined at different sludge and

flocculant flows. Five flocculants have been analysed, two of which were previously used.

At present, tests are being carried out to determine which flocculant is the most suitable for Ecoparc 2 sludge, obtaining a higher yield in the solids retention without increasing flocculant consumption.

#### 2.3.3.2. PW tanks sealing

Following phase 1 where it was possible to determine the different effluents flow, we observed that the WWTP treated more PW than was reflected in the water meters. For this reason, we suspected that there were infiltrations in the feed tanks since this is where the difference in flow between the PW production and the input to the WWTP was found.

Thanks to the tests carried out (explained in section 2.2.2.2) we determined that tank 3 had runoff water infiltration and had to be repaired.

At present, the PW tanks have been emptied and are being repaired.

#### 2.3.3.3. PW tanks homogenization

As has already been mentioned on several occasions, it is essential to homogenise the WWTP input in order to avoid sudden changes that destabilise the biological process, alter bacterial growth and affect WWTP performance.

Currently, since the tanks have been emptied to repair them and to avoid infiltrations, we have built an agitator which homogenises the PW content in the feed tanks, so that any changes are reduced in the input which improves the wastewater treatment process (figure 2.18). A rotatory filter will also be incorpored to help retain the PW solids that do not come from the anaerobic digestion (figure 2.19).

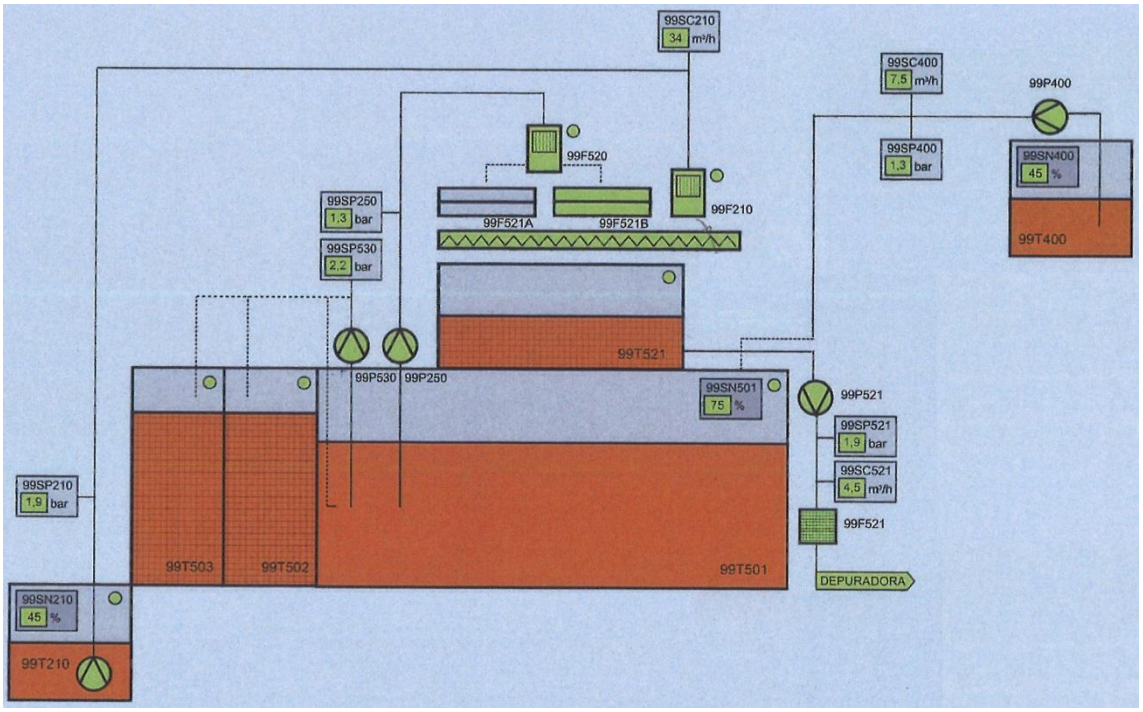



***Figure 2.18:*** *Modifications in the feed tanks to homogenise the PW*

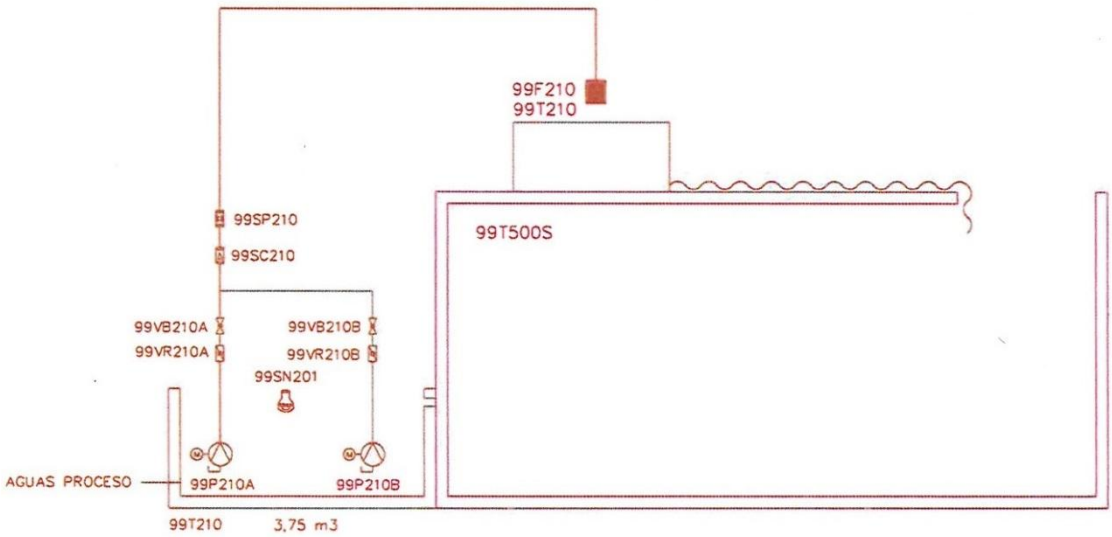



***Figure 2.19:*** *Rotatory filter incorporation to retain PW solids*

### *2.3.4. Biological purification process improvement*

In addition to analytical and input WWTP, changes have also been made in the biological purification process with the aim of improving the WWTP performance by obtaining a higher quality permeate.

#### 2.3.4.1. Control parameters

We have added the calculation of various control parameters to observe if the state of the WWTP remains constant or is altered by the different ratios. There are changes that cannot be seen at first glance, and it is necessary to determine the key parameters.

These parameters have been added in the Excel record so that they are automatically calculated from theentered data of control checks and performed analytics.

#### 2.3.4.2. Chemicals dosing

In order to maintain WWTP functioning correctly it is necessary to dose some chemicals that overcome any shortages the PW. For this reason, an equation has been established that calculates the quantity needed to be dosed depending on the conditions in which the PW is found. In this way, it is ensured that the necessary quantity is added without any excess that can disfavour the process and cause a different type of destabilisation and even an increase in the economic cost. For example, an excess methanol dosage causes an increase in permeate COD.

#### 2.3.4.3. Purge

It is necessary to maintain a correct cell residence time to maintain the biology in the reactors without accumulating large quantities that in the absence of nutrients end up dying and increasing the DM inside the reactor, hindering the biological reactions of the microorganisms. This parameter can be controlled from the recirculation and purge. It is therefore necessary to calculate correctly the amount needed to purge, so that it does not alter the CRT by emptying the biology tank or creating a solids excess.

*A. Purge calculation*

An equation has been established which calculates the exact amount to be purged depending on the inlet and outlet conditions from the WWTP. This equation has been added to the Excel record so that it is calculated directly from the control and analytical data.

### *B. Continuous purge*

In order to try to maintain the optimal WWTP conditions, without major alterations that do not allow working at a stationary point, it is advisable to purge continuously. Working in batch causes increases and decreases in solids concentration and performance differences throughout the day (figure 2.20), while in continuous bleaching these destabilizations do not occur and allows the process to work with a more stable performance (figure 2.21).

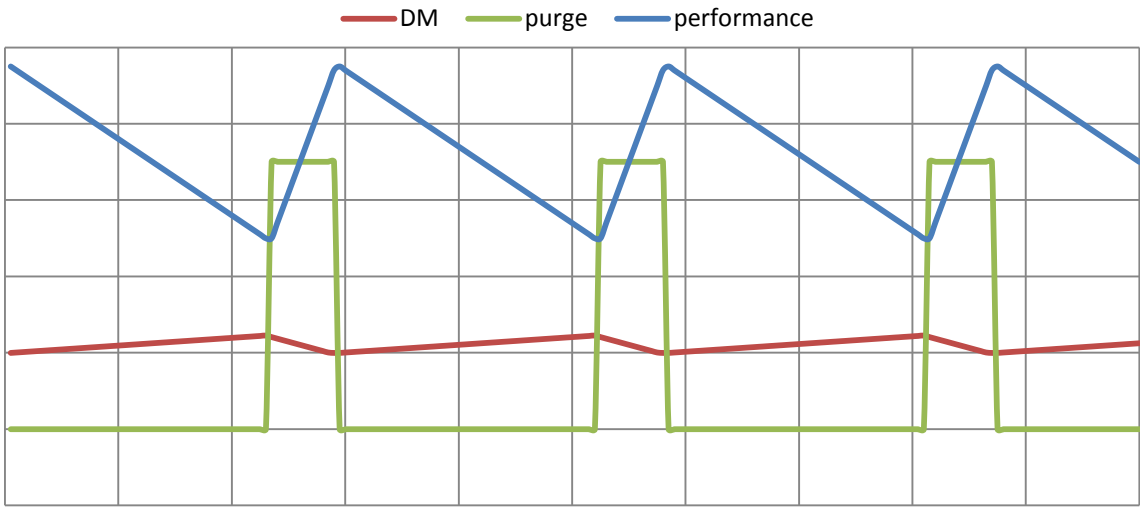



***Figure 2.20:*** *DM and performance effect when purging in batch*

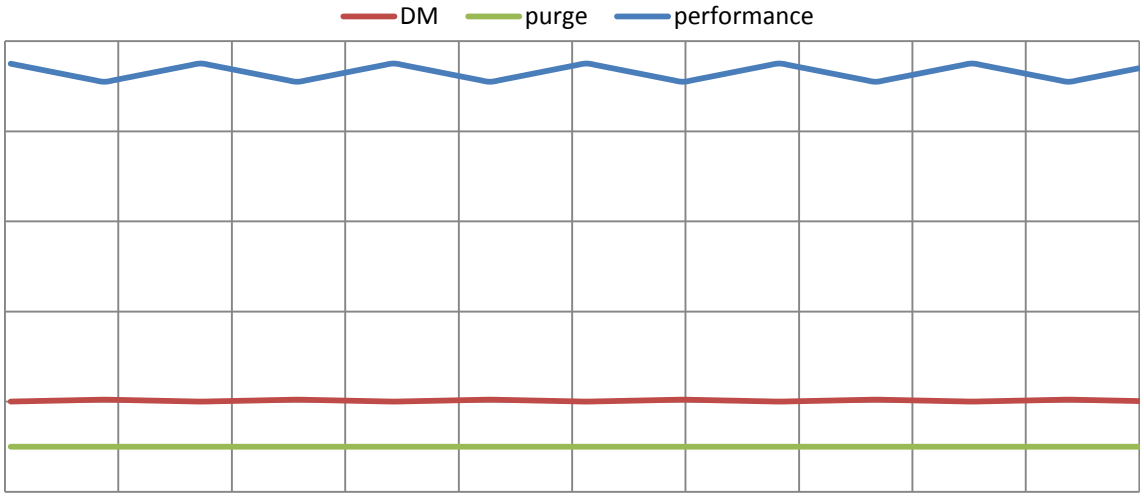



***Figure 2.21:*** *DM and performance effect by continuous purging*

A study has also been carried out to determine the best purge flow rate for continuous operation to maintain the WWTP properties unchanged taking into account that the

purge would be destined to a centrifuge in which the liquid phase would be recirculated to the first WWTP tank.

#### 2.3.4.4. Combined tank operation

The operation of the combined tank has been modified, further spacing of the operation time with aeration and methanol and the pause between one phase and another. In this way, it is possible to improve the oxia and anoxia conditions for the biological nitrification and denitrification reactions respectively.

It has also been chosen to use it more as a denitrificator since in the WWTP there are already two nitrificator tanks with good performance, and a single denitrificator that needs to be extended to reduce all of the nitrates to elemental nitrogen.

## 2.4. WWTP evolution

In this section the evolution of some of the parameters is given from January 2008 to June 2017 in order to observe the WWTP improvement and optimisation.

### *2.4.1. Flows*

Figure 2.22 shows the PW, permeate and purge flows from January 2008 to June 2017.

PW flow at present is slightly more stable than in the first year that it was analysed. However, it is still necessary to continue to work to stabilise and homogenise the input to the WWTP. It is true that, since 2013, we have been working at flow rates close to the design limit value (142 $m^3$/day), so the WWTP performance is optimised, but the process efficiency to improve the quality of the spill. It should also be noted that since the beginning of 2016 the evaporator has been shut down, causing a lower MW and energy consumption, and all the PW has been treated in the biological WWTP.

Regarding the purge, the flow has been very unstable, having values from 6 to 134 $m^3$/day. Therefore it is necessary to apply the purge equation, to avoid abrupt changes and to ensure a constant DM inside the tanks. Since May 2016, an automatic purge calculation has been started, purging approximately 90 $m^3$/day compared to the previous 60 $m^3$/day. Once the DM was lowered into the tanks, the purge calculation decreased to the current 70 $m^3$/day.

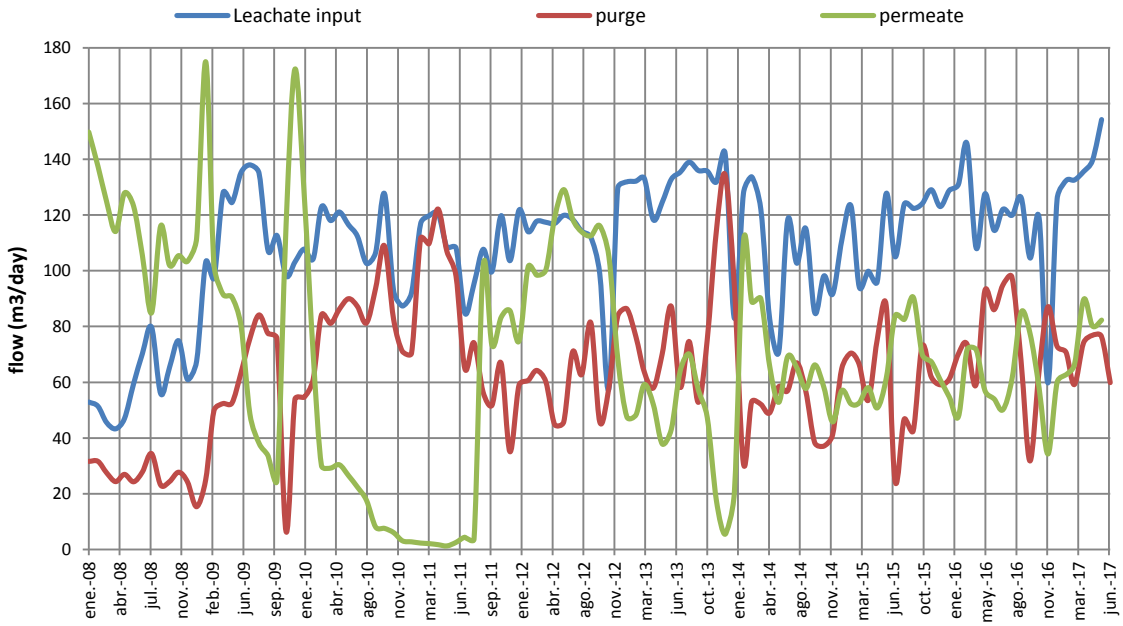



***Figure 2.22:*** *PW, permeate and purge flows since January 2008 to June 2017*

### *2.4.2. Dry maters*

Figure 2.23 shows the quarterly evolution of the PW, permeate and four WWTP tanks DM.

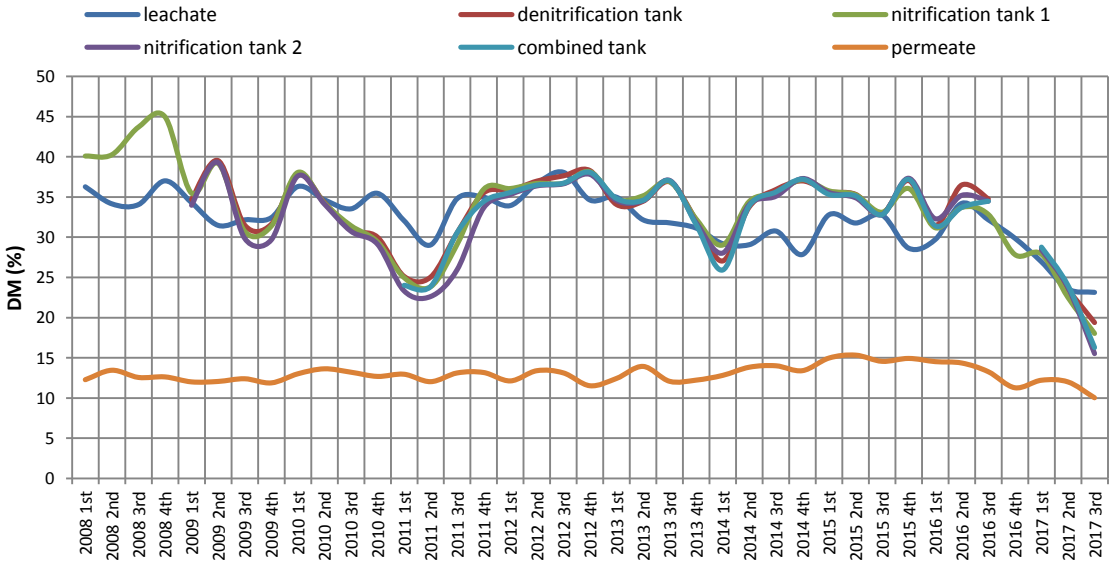



***Figure 2.23:*** *Quaterly average of PW, permeate and four WWTP tanks DM*

The PW DM is very variant, so it was decided that the incoming PW should be homogenised as much as possible to allow a greater biological process stabilisation in

the WWTP. The value in tanks is very similar, but with pronounced variations that can destabilise the WWTP process. In the permeate case, variations are also observed, but they are smaller because large solids amounts are retained in the ultrafiltration and are purged or recirculated to the process.

There is also a decrease in DM in the PW and all reactors from 2016 to the present. The last period of these data is shown in figure 2.24.

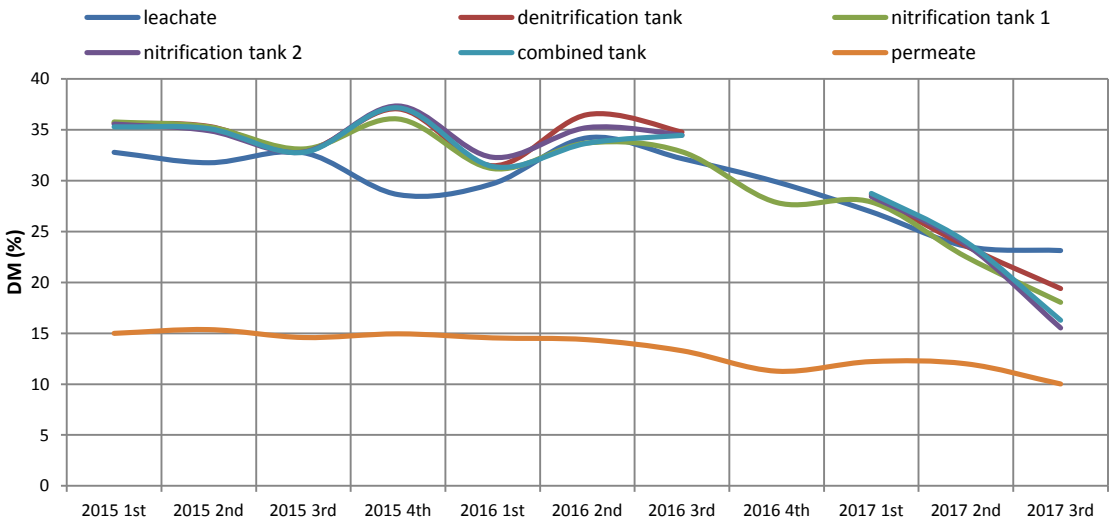



***Figure 2.24:*** *Quaterly average of PW, permeate and four WWTP tanks DM in the last period*

This decrease in DM is due to the fact that in May 2016 the purge calculation was started and more sludge was purged to ensure the DM within the process. In December 2016, the tanks were emptied to reinforce them, and they were thoroughly cleaned and the deposited solids were removed. In spite of having tested the double centrifuge cycle, in March 2017 the second centrifugation cycle was started as usual, so that the PW DM is 22 % compared to the 35-40 % previously obtained. In this way it has been possible to reduce the TS amount in the sludge to be treated in the WWTP and to facilitate the dissolved oxygen transfer and decrease the viscosity within the tanks.

### *2.4.3. COD and ammonium*

Figure 2.25 shows the PW and permeate COD and ammonium concentrations from January 2008 to June 2017.

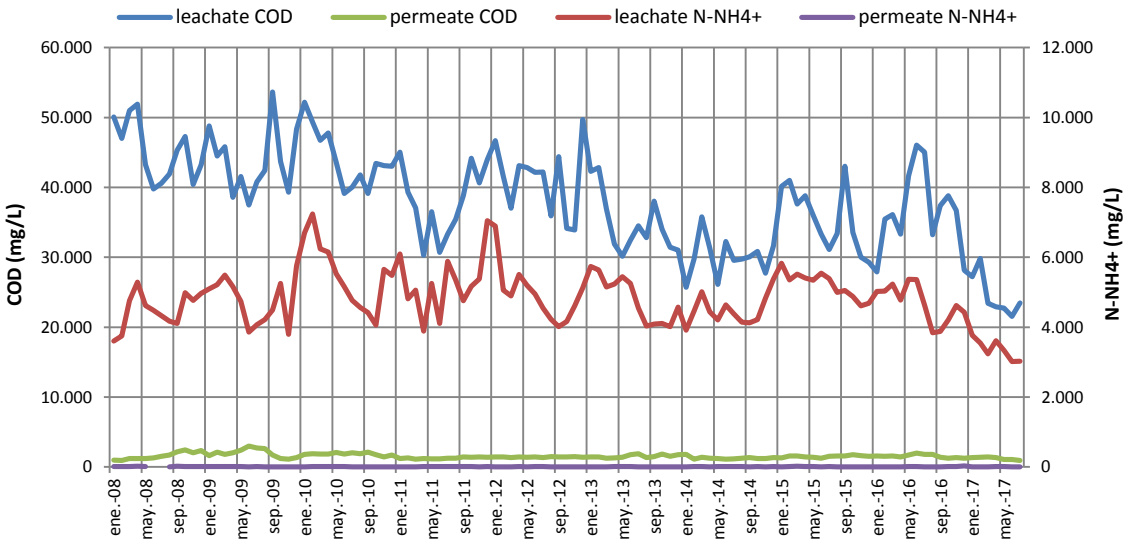



***Figure 2.25:*** *PW and permeate COD and ammonium concentrations*

The COD has decreased in large quantities over this period, from values above 53,000 mg $O_2$/L in September 2009 to values below 22,000 mg $O_2$/L in June 2017, which represents a decrease of 59.85 % COD at the input to the WWTP.

In the ammonium case, it was also possible to decrease the concentration in the PW from values higher than 7,000 mg N/L to values of 3,000 mg N/L, assuming a 58.27% ammonium reduction at the input to the WWTP.

The second centrifugation cycle has proved to be key to eliminate a large part of the PW organic and ammoniacal load, thus subjecting the WWTP to a smoother process.

There has also been observed an improvement in the permeate case where the values continue to meet the discharge limits but with lower oscillations and having subjected the biology to a smoother process than before.

### *2.4.4. Combined tank parameters*

Figure 2.26 shows the average methanol dosage, aeration and pause parameters of the combined tank.

The air, pause and methanol times were very short, so that the process did not really reach oxic or anoxic conditions for the sludge nitrification or denitrification. Only occasionally did the reactor work with methanol dosing, so the process was subjected to nitrification without denitrification in this tank. Since 2016 the times have been extended to improve the oxia and anoxia conditions in this tank and methanol dosage

periods were established to reduce nitrates to elemental nitrogen, but it is in the year 2017 when this became more accentuated.

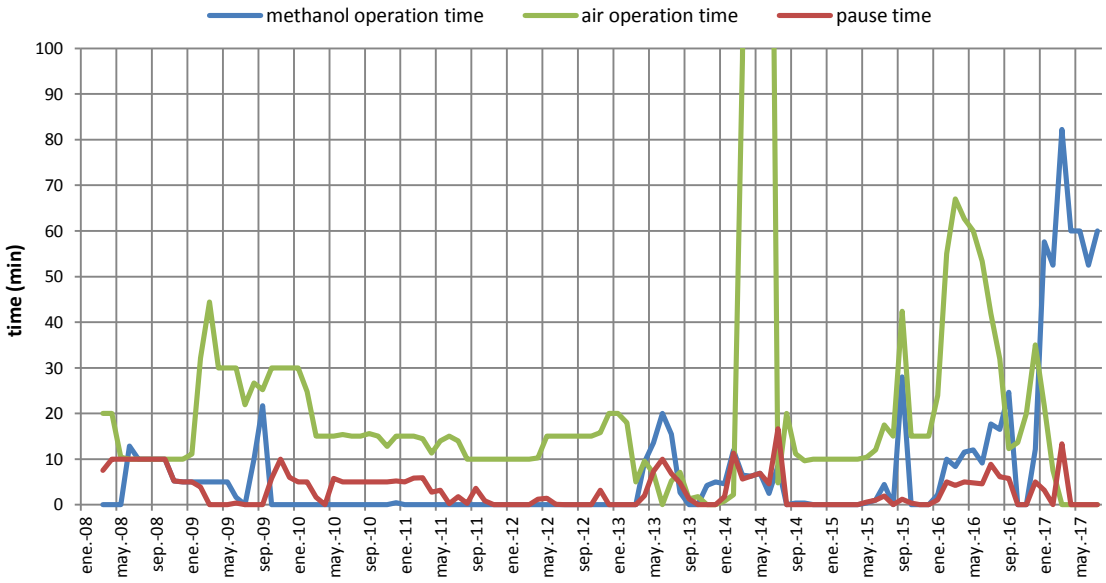



***Figure 2.26:*** *Combined tank methanol dosage, aeration and pause time parameters*

### *2.4.5. Methanol dosification*

Figure 2.27 shows the methanol dosage in the denitrification and combined tank from January 2008 to June 2017.

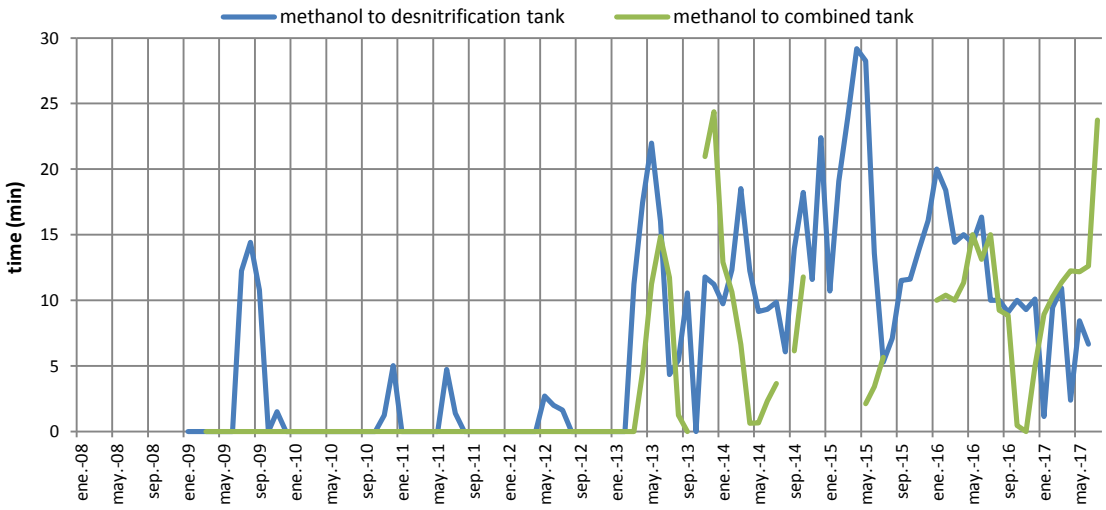



***Figure 2.27:*** *Methanol dosage in the denitrification and combined tank*

In the beginning, methanol was not dosed to any of the two tanks in which the denitrifications reactions occur. From 2009 to 2012, this practise was performed occasionally. Since 2013 the dosage has been established without completely eliminating it given that greater stability was observed in the biological process.

However, methanol dosing is only necessary in the case of a carbon source shortage (low COD) since the biology involved in the process reacts with methanol earlier than with the COD present in the sludge since methanol is easier to biodegrade. It is not necessary to dose methanol in the denitrification tank since it is the first WWTP tank which contains a higher organic load. In the combined tank it is necessary only in those cases where the COD present in the sludge is not enough for the process. Consequently, calculation equations have been established for this methanol dosage that determine if dosification is necessary and, if so, with what flow.

Therefore, it is necessary to eliminate the methanol dosage in the denitrification tank and decrease the combined tank flow. But it is necessary to take into account that the purification process is biological and cannot be subjected to abrupt changes in biology, so that the methanol dosage must gradually decrease until it is eliminated, giving the biology sufficient time to get accustomed to the new conditions and keeping the process stable. This is a change on which we are still working today.

## 2.5. Conclusions

Throughout this phase, including the changes made during the project given in the previous section, the following conclusions have been obtained:

- Particular attention has been paid to the analytical procedures carried out at Ecoparc 2 in such a way that they are carried out according to standardised methods which ensure good practice and enable us to compare values with bibliographic dates or other similar WWTP.
  Virtually all analytics are performed with analytical kits which are based on standardised internationally approved methods such as DIN, EN, ISO, EPA and APHA. However, nitrates and nitrites followed other procedures, therefore they have been modified to DIN, EN and ISO methods. The remaining parameters that were not performed with analytical kits have been found to follow the Standard Methods for the Examination of Water and Wastewater and the alkalinity procedures has been modified to be consistent with this.

- To know the process state and the permeate quality, new analytics have been incorporated to determine the viscosity, total nitrogen, sulfides and solids parameters. After all these additions a new periodicity has been established taking into account the needs and the reponse time to correct any alteration. For example, the DM is no longer determined daily in the nitrificator 2, however, the process solids are determined.

  At the beginning of the thesis project only the pH and DM tanks were considered and the rest of the analyses were carried out to the PW and permeate, so that if the process did not work correctly it was difficult to detect where the process was failing. However, weekly COD and nitrogen forms analytics have now been incorporated to determine the state of each reactor and to be able to determine if each process is performing correctly.

  The response time due to a process alteration has been reduced since the data transmission time to the Excel record that the different departments control has been minimised. Colored indicators have also been incorporated to more easily detect if a parameter deviates from the set interval, so it can be quickly checked without having to waiting for the purifying process to become destabilised.

  Furthermore, we have also looked at which analytics are performed at each point and their periodicity, and a guide has been established for analytical procedures where everything related to analytics is indicated, from periodicity analysis, points sampling and procedures, including the analytical timetable and an inventory, calibrations and stock control, up until the interpretation of the obtained results .

- Finally, new work forms have been established and some changes have been made to ensure the proper functioning of the WWTP and to optimise its performance and effectiveness. To this end, measures have been applied both in the PW prior to the WWTP entry and in the process itself.

  Firstly, tests have been carried out to optimise and deplete the flocculant used in the centrifuge of the PW that arising from the anaerobic digestion, since this represents more than 75 % of the PW treated in the WWTP. The tests were performed with one and with two centrifugation stages. In the single stage tests, the best results were obtained with the flocculant HYFLOC XT-653 and FLOPAM TE 4650 SH with a value of 2.06 % TS in the liquid phase and a consumption of 7.4 kg/Tn DM. However, in the tests performed with two centrifugation stages better results have been obtained, where the most

effective flocculant is FLOPAM TH 4650 VHM with a result of 1.43 % TS in the liquid phase and a consumption of 14.86 kg/Tn DM. Compared with the Zetag 8185 flocculant used earlier, better results are obtained but with a higher consumption that must be taken into account for its titration.

We have observed that the feed tanks to the WWTP had runoff water infiltrations, so that the flow to be treated in WWTP was larger than necessary. At present, work is being carried out in which the tanks have been emptied and are being treated to avoid external infiltrations. A rotary screen is also being incorporated to retain the solids coming from the Ecoparc 2 PW, except for those coming from the anaerobic digestion and composting which are directed to PW tank 1. Tank 1 has an agitator than mixes and homogenises all the PW to be treated previously to the feed filters of the WWTP.

On the other hand, control parameters have been incorporated to observe the WWTP state together with an Excel record of analytical parameters. Several schemes that facilitate a display of the WWTP state by way of both analytical values and control parameters that is automatically updated according to the consultation day.

Concerning the chemicals dosing, an equation has been established for each product (phosphoric acid, sodium hydroxide and methanol) which directly calculates the flow to be dispensed of each product from data in an Excel register. An equation has also been established which calculates the amount to be purged daily from COD, methanol dosing, ammonium and ISS input values. The possibility of continuous purging is currently being studied to maintain the process stability and to avoid abrupt changes which might affect the biology.

Finally, more extended combined tank operation periods have been established, so that the nitrification and denitrification operations are improved due to better oxia and anoxia conditions in the tank.

Thanks to all these changes, improvements have been observed in the biological purification process, which has made it possible to treat all the PW in the WWTP without needing to use the evaporator since 2016.

The calculation of the daily amount to be purged has allowed the reduction of DM inside reactors and to maintain it within required values, avoiding a high viscosity that would affect the biological process and the dissolved oxygen transfer. The flocculant and double centrifuge cycle optimisation has also caused a significant change, and has allowed to reduce PW DM to be treated in

values higher than 38 % in 2012 to values lower than 24 % in 2017, leading to a 39.26 % DM reduction to the WWTP.

The changes with the centrifuges and flocculant have also led to improvements in the organic and ammoniacal loading at the WWTP, giving 59.85 % and 58.27 % less respectively. This provides the biology to a smoother process, facilitating the purification and improving the efficiency. In this way, smaller and more stable permeate values have been obtained than prior to this project.

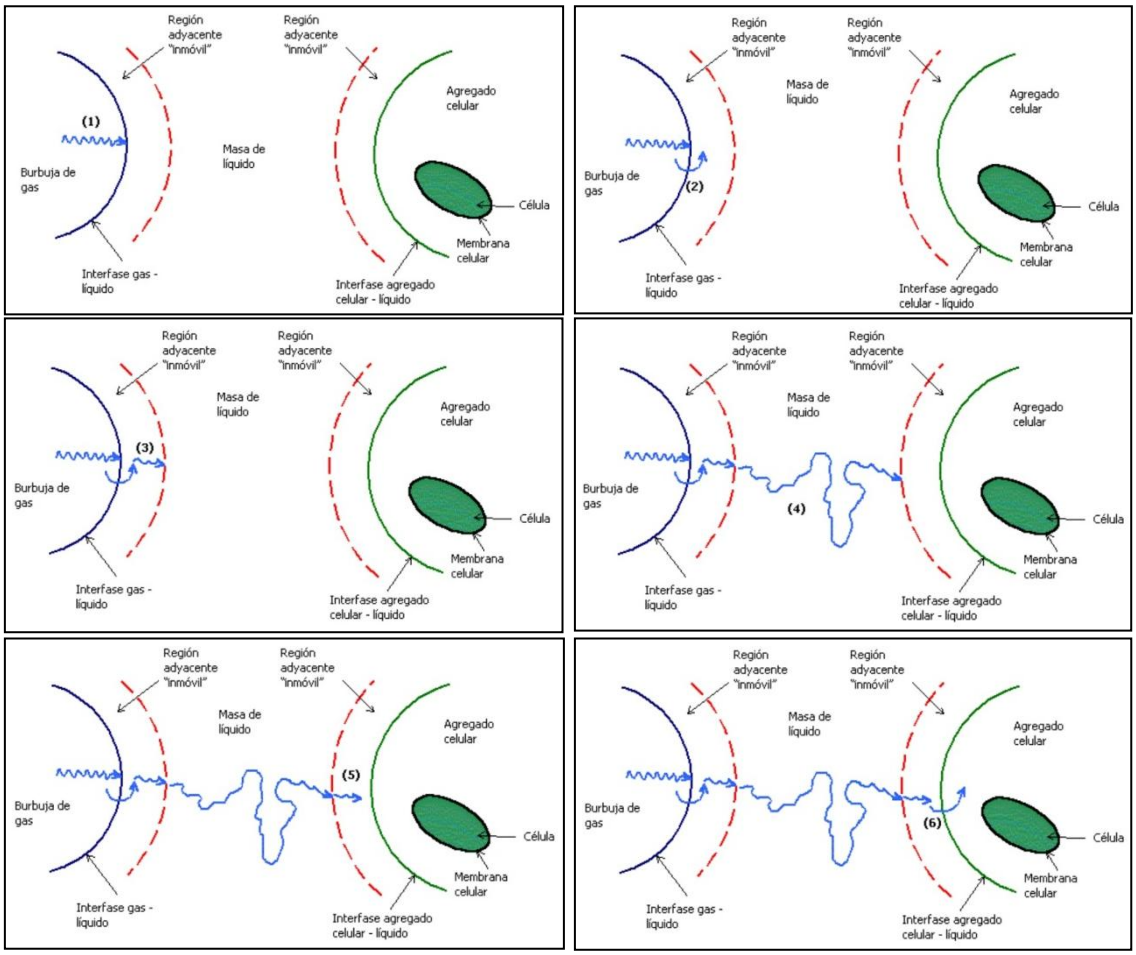
In the case of the methanol dosage, it has been observed that it is necessary to eliminate it in the denitrification tank, but this should be done progressively so as not to subject the biology to modifications that can affect the process. In this sense, we are still working on this reduction, in order to reach a methanol dosage in denitrification equal to 0 and in the combined tank equal to that indicated from the relevant equation each day.

# PHASE 3. PROCESS WASTEWATER SOLIDS SEPARATION

## 3.1. Introduction

According to UGR 1995, the $O_2$ transfer is the controlling step in anaerobic fermentation processes. Therefore it is necessary to have suitable methods for the measurement of transfer parameters, specific interfacial areas and bubble size distributions.

Both the nutrients and dissolved oxygen consumption by process biology is done by transferring from one phase to another, giving rise to biological reactions within the cell membrane. Figure 3.1 shows the phases that an oxygen molecule follows once it is dissolved until it is inside the cell membrane.



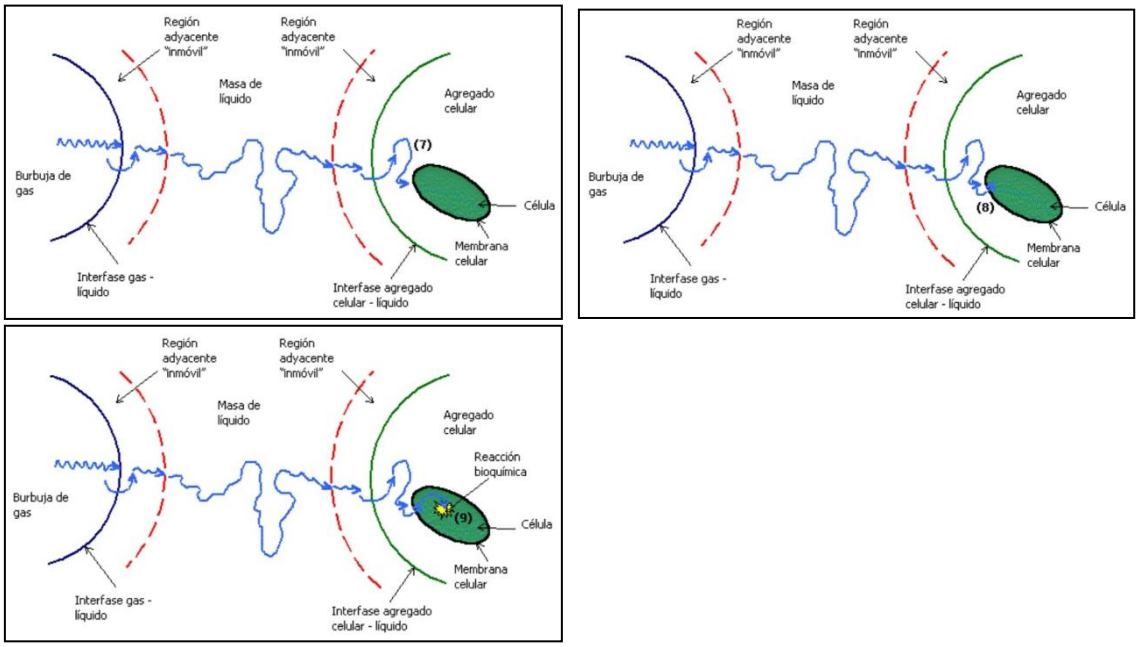



***Figure 3.1:*** *Oxygen transfer phases (image author: [http://procesosbio.wikispaces.com](http://procesosbio.wikispaces.com))*

The phases to which each image belongs are:

(1) Diffusion of gas bubble to the gas–liquid interface.
(2) Transfer through gas-liquid interface.
(3) Dissolution of solute through the layer of immobile apparent liquid adjacent to the gas bubble.
(4) Transport of solute through the liquid mass to the apparently immobile liquid adjacent to the cellular aggregate.
(5) Transport of solute through the layer adjacent to the cellular aggregate.
(6) Diffusion within the cellular flocculus, mycelium or soil particle.
(7) Transport to the cellular envelope.
(8) Passage through the cellular envelope.
(9) Transport of solute to the site of intracellular biochemical reaction.

There are several factors that influence the oxygen and nutrients solubility such as temperature, pressure and presence of solutes. Biological reactions occur within the cell, so the oxygen and nutrient particles must be able to enter the cell, so a high suspended solids content or high viscosity hamper access to the cell and decrease the process yield.

As we have seen in the previous phases, the Ecoparc 2 process waters contain a large amount of solids that hinders the WWTP biological process. The WWTP biology has a more difficult access to nutrients and dissolved oxygen due to high solids contents in the reactor and the high medium viscosity. Therefore, the oxygen and nutrients diffusion and solubilization is more complex.

Throughout the study there have been several tests, including different analytical methods for each parameter listed in the WWTP state.

One test was to decide if the analytical trials were made from the collected sample or if they should be centrifuged before performing the analysis, to minimise any possible interference in the spectrophotometer caused by colour or turbidity. The problem was that the exact parameter was not determined. However this observation was important for this study. The centrifugation could retain a significant amount of solids so that the COD could be minimises in very significant values, a 44.4% average. It proved to be such a high value that we considered the feasibility of performing a PW solids separation prior to the WWTP. For this, the available and accessible machinery at Ecoparc 2 was examined for its possible implementation.

COGERSA, S.A.U. is a Company for Solid Waste Management in Asturias that, since the 80s, has has tried to find the most appropriate solution to waste treatment in Asturias.

COGERSA has a PW treatment plant with a process very similar to Ecoparc 2. This plant has undergone several extensions due to PW changes and needs, from having a treatment capacity of 100 $m^3$/day to 700 $m^3$/day. Figure 3.2 shows the COGERSA PW treatment process scheme.

COGERSA found that PW occasionally contained high amounts of suspended solids that were concentrated in reactors and caused a collapse. To prevent this problem in 2007 they incorporated a physical-chemical treatment to coagulate, precipitate and centrifuge the solids ensuring the PW quality to be treated and the installation capacity.

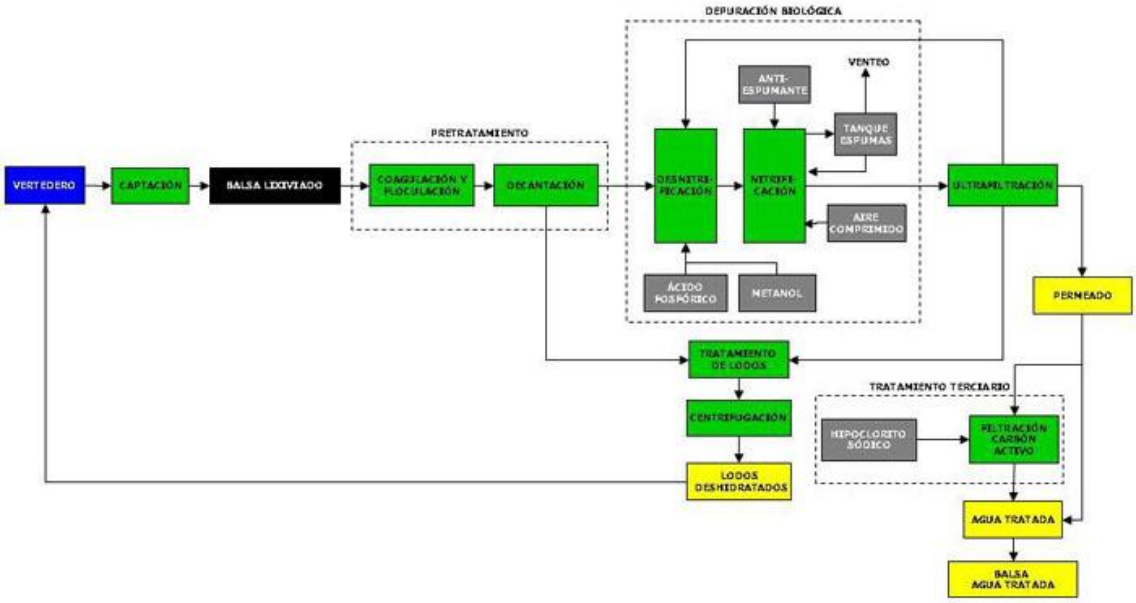



***Figure 3.2:*** *COGERSA landfill PW treatment process scheme (image author: COGERSA, S.A.U.)*

*Landfill PW treatment case studies:* is a study carried out in France where they characterised three different landfill PW's and determined the reduction of COD, BOD5, TKN, SS and metals of each one after subjecting them to cogulation/flocculation, membrane reactors and ozonation processes (figures 3.3 – 3.5).

In all three cases, the treatment with the best reduction results in all measured parameters is the treatment with ozone, followed by membrane bioreactors and, finally, coagulation/flocculation.

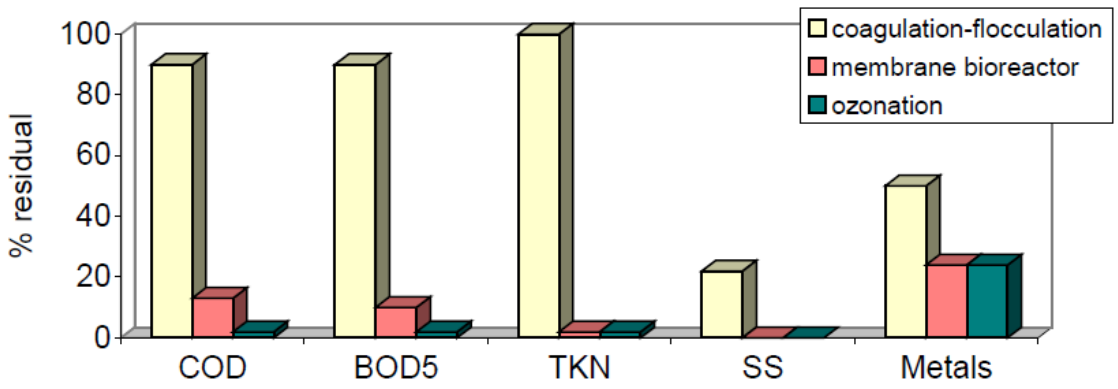



***Figure 3.3:*** *Residual percentage in PW 1 (author: S. Baig)*

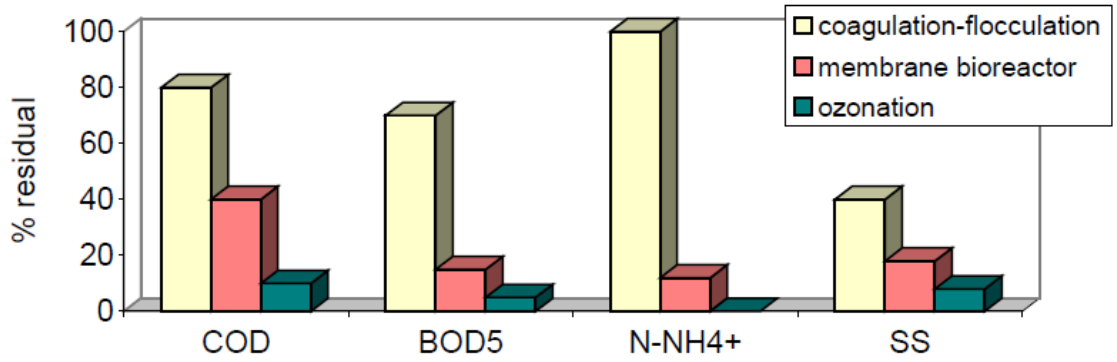



***Figure 3.4:*** *Residual percentage in PW 2 (author: S. Baig)*

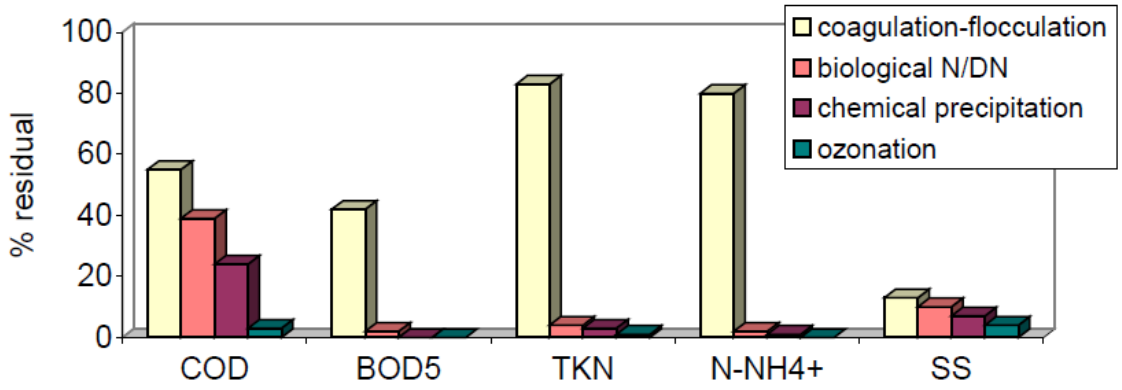



***Figure 3.5:*** *Residual percentage in PW 3 (author: S. Baig)*

Flocculation treatments with centrifuges tests can be performed by using the equipment available at Ecoparc 2, but not separation by membranes or ozone treatments. For this reason, IREC (Catalonia Institute for Energy Research) was contacted, as they had all the necessary equipments to carry out membrane separation tests and to determine the results with commercial membranes.

## 3.2. Solids separation using available machinery

A sludge is obtained as a result of anaerobic digestion that goes through a dehydration process by a press followed by a centrifuge. The anaerobic digestion area has 3 centrifuges, 2 of which are operational and 1 is available (figure 3.6). So that one can be used for PW solids separation in the case that we get good results after the appropriate testing.

***Figure 3.6:*** *Anaerobic digestion area centrifuges*

### *3.2.1. Laboratory scale tests*

To determine the optimum and viable speed and time to which the centrifuge should operate, 2 different tests were made according to the procedures detailed in “Phase 3 tests” of chapter “Analysis and methods”.

- *Test 1*: to calculate the solids, COD and ammonium decrease at different speeds setting centrifugation 20 minutes for each cycle.

- *Test 2*: to calculate the solids, COD and ammonium decrease at different times with the same speed (determined as the best option in test 1).

#### 3.2.1.1. Test 1: Varying speed centrifugation

Table 3.1 shows the results obtained in test 1 and figure 3.7 presents the same results graphically.

***Table 3.1:*** *Solids, COD and ammonium reduction depending on centrifugation speed during 20 minutes centrifugation*

| Time (min) | Speed (rpm) | TS reduction (%) | VS reduction (%) | COD reduction (%) | N-$NH_4$ reduction (%) |
|---|---|---|---|---|---|
| 20 | 400 | 7.5% | 14.0% | 1.8% | -5.0% |
| 20 | 700 | 10.0% | 15.6% | 1.8% | -2.0% |
| 20 | 1000 | 12.0% | 16.8% | 3.6% | -4.0% |
| 20 | 1250 | 13.4% | 20.9% | 5.4% | -7.0% |
| 20 | 1500 | 14.3% | 20.7% | 7.1% | -4.0% |
| 20 | 1750 | 14.8% | 23.3% | 8.9% | -7.0% |
| 20 | 2000 | 15.7% | 25.6% | 8.9% | -6.0% |
| 20 | 2500 | 16.5% | 22.4% | 10.7% | -5.0% |
| 20 | 3000 | 17.3% | 19.1% | 14.3% | -3.0% |
| 20 | 4000 | 19.4% | 26.0% | 17.9% | -5.0% |
| 20 | 5000 | 18.9% | 24.8% | 24.1% | -9.0% |
| 20 | 7000 | 25.4% | 38.8% | 25.9% | -12.0% |
| 20 | 9000 | 26.3% | 43.4% | 37.5% | -5.0% |

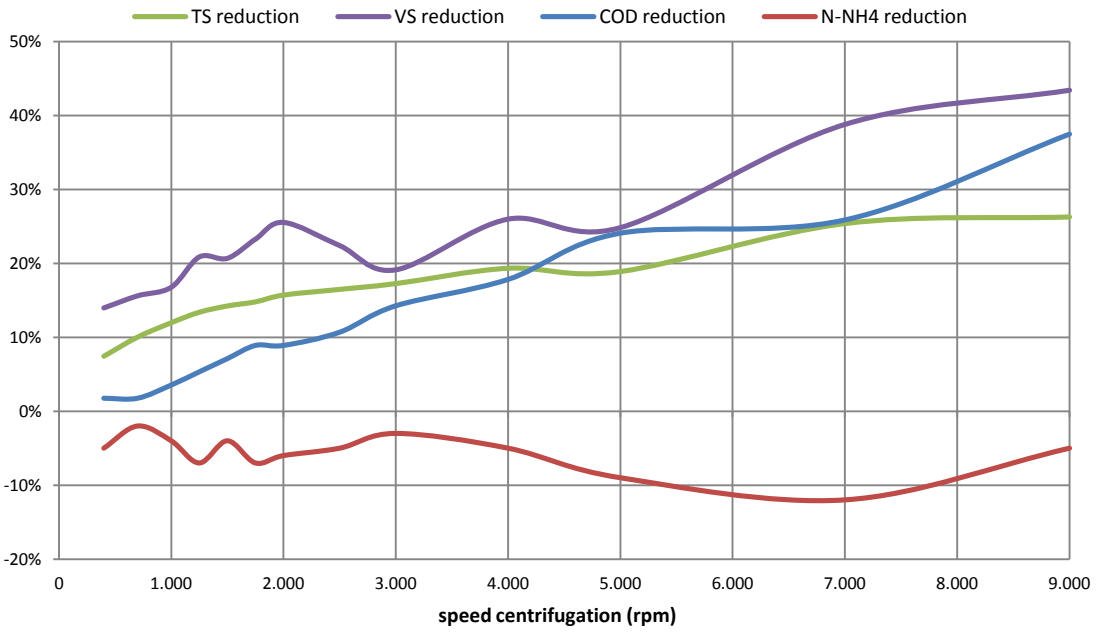



***Figure 3.7:*** *Solids, COD and ammonium reduction depending on centrifugation speed (rpm) during 20 minutes centrifugation*

The most sensitive parameters are VS and ammonium because they vary in time rapidly due to side reactions and equilibrium with liquid phase and air. This is reflected in the results obtained in these parameters as fluctuations without a constant tendency are observed. However, it is observed that the VS decrease with increasing the centrifugation speed, while the ammonium is not affected by the centrifugation, as no decrease is seen. The ammonium increase, without direct relation with the centrifugation speed, can be due to side reactions, in which the organic nitrogen changes its form to ammonium. Regarding the TS and COD decreased, a direct relation can be seen with the centrifugation speed, the higher the speed the better the results obtained.

Given that the centrifuges available at Ecoparc 2 can reach up to 3000 rpm, with the results obtained we expected a TS and COD decrease of 17 and 14% respectively.

#### 3.2.1.2. Test 2: Varying centrifugation time

Table 3.2 shows the results obtained in test 2 and figure 3.8 presents the same results graphically.

***Table 3.2:*** *Solids, COD and ammonium reduction depending on centrifugation time at 9000 rpm*

| Time (min) | Speed (rpm) | TS reduction (%) | VS reduction (%) | COD reduction (%) | N-$NH_4$ reduction (%) |
|---|---|---|---|---|---|
| 5 | 9000 | 23.5% | 40.2% | 33.1% | 1.3% |
| 10 | 9000 | 25.8% | 41.6% | 35.6% | -3.8% |
| 15 | 9000 | 26.0% | 42.4% | 37.3% | -3.8% |
| 20 | 9000 | 26.3% | 43.4% | 37.5% | -5.0% |
| 30 | 9000 | 29.5% | 49.2% | 39.8% | -2.5% |
| 45 | 9000 | 30.8% | 52.8% | 43.2% | -1.3% |
| 60 | 9000 | 33.3% | 54.3% | 47.5% | -2.5% |
| 90 | 9000 | 34.2% | 56.4% | 49.2% | -6.3% |

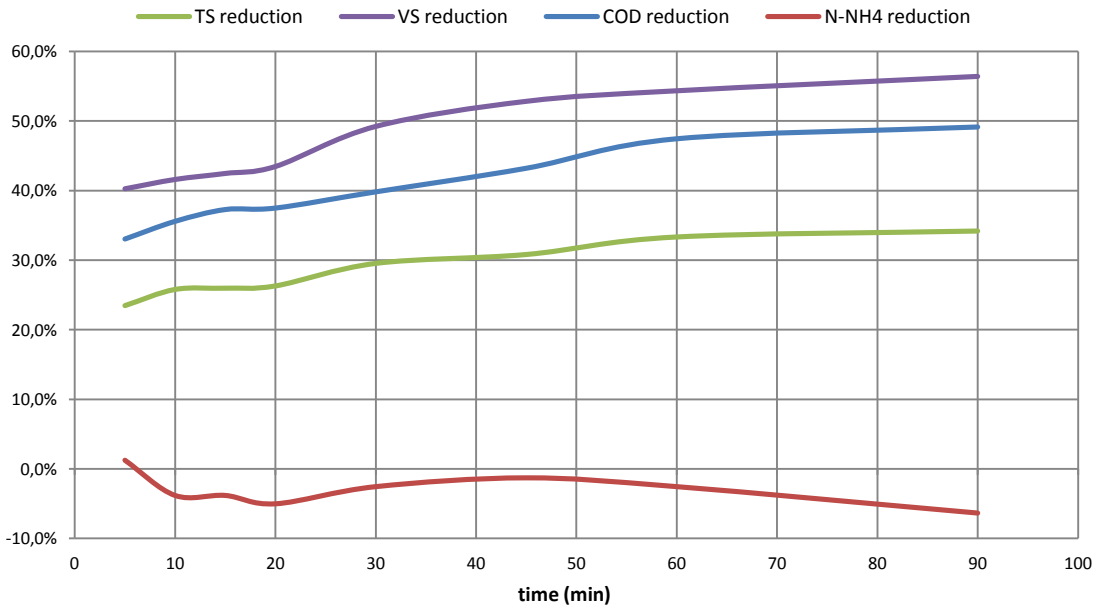



***Figure 3.8:*** *Solids, COD and ammonium reduction depending on centrifugation time (minutes) at 9000 rpm*

In the same way as in test 1, we did not see a direct relation between ammonium decrease and centrifugation time. However, the solids and COD decrease are influenced by time, being higher the percentage reduction as the centrifugation time is prolonged. However, the only slight difference between 60 and 90 minutes does not appear to hjustify lengthening the process.

To determine the optimal time at which the process should be done an industrial scale test needs to be peformed.

### *3.2.2. Industrial scale tests*

The Humboldt centrifuge is the one used in the sludge dehydration and the Andritz centrifuge is the one that remains on standby and has been used in the test to realise the second centrifuge stage.

Initially, a preliminary test was performed in which the liquid obtained in the Humboldt was subjected to centrifugation in the Andritz without adding flocculant in order to exhaust the amount remaining in the liquid phase. As a result, it was observed that the centrifugal product was not towards cake formation and thus lack of retention of the solids did not vary the concentration in the liquid obtained in the second stage.

After consulting with a centrifuge expert, he informed us that the Andritz has a capacity of 1,000 kg/h while we were working with a load of 240 kg/h. To be able to obtain good results we needed to work at a greater flow or concentration. Therefore, a second test was performed in which the same flocculant amount was added as in the first stage centrifugation, but in this case it was divided into two stages. So, with a lower flocculant dosing in the first stage, the liquid phase would have higher concentration and better results were expected in the second stage in which we would also have flocculant contribution to help retain solids. On this occasion, if positive results were observed, it was decided to establish a test schedule to determine the appropriate conditions for a second centrifuge stage, establishing a premise and a modification:

- Premise: objective is to find second centrifuge stage liquid phase with the lowest DM concentration possible.
- Modification: reduce polymer concentration in half, from 5 g/L to 2.5 g/L, decreasing the dosing time.

The centrifuge technician advised us to calculate the VS in the centrifuges inlet and outlet as it is the liquid part that cannot be separated by centrifugation, so that if we have a concentration at the entrance of DM 4 g/L and VS 3 g/L, the maximum result that we can obtain is a decrease of 1 g/L.

Table 3.3 shows the results obtained in 13 tests carried out, taking into account that in all tests HYFLOC XT-653 flocculant was used at a concentration of 2.5 g/L.

***Table 3.3:*** *Results obtained in double centrifuge stage industrial tests*

| Test | Speed (rpm) | Relative speed | PAR (%) | Input concentration (g/L) | Sludge flow ($m^3/h$) | Input DM (kg/h) | Flocculant flow (L/h) | Consumption (kg/Tn DM) | Liquid phase TS (%) | Liquid phase aspect | Solid phase aspect |
|---|---|---|---|---|---|---|---|---|---|---|---|
| A | 2,000 | 5 | 4.6 | 95.8 | 8.5 | 814.3 | 2,600 | 7.93 | 2.69 | regular | **good** |
| B | 2,000 | 5 | 14.3 | 95.8 | 8.5 | 814.3 | 2,600 | 7.93 | 2.15 | **good** | good/regular |
| C | 2,000 | 7.4 | 6 | 95.8 | 8.5 | 814.3 | 2,600 | 7.93 | 2.22 | **good** | good/regular |
| D | 2,000 | 7.4 | 5.9 | 95.8 | 8.5 | 814.3 | 2,150 | 6.60 | 2.54 | **good** | good/regular |
| E | 2,000 | 5 | 11.5 | 95.5 | 8.5 | 814.3 | 2,150 | 6.60 | 2.55 | **good** | good/regular |
| **F** | 2,000 | 5 | 4.5 | 95.8 | 7.5 | 718.5 | 2,300 | 8.00 | 1.39 | **good** | **good** |
| **G** | 2,000 | 5 | 11.3 | 95.8 | 7.5 | 718.5 | 2,600 | 9.04 | 1.67 | **good** | **good** |
| H | 1,800 | 5 | 4.5 | 95.8 | 7.5 | 718.5 | 1,300 | 4.52 | 3.17 | regular | regular |
| I | 1,600 | 5 | 3.6 | 95.8 | 7.5 | 718.5 | 1,300 | 4.52 | 3.01 | regular | regular |
| **J** | 1,600 | 5 | 6 | 95.8 | 7.5 | 718.5 | 2,600 | 9.04 | 1.97 | **good** | **good** |
| **K** | 1,900 | 5 | 16.1 | 133 | 8 | 1,064 | 2,600 | 6.10 | 2.26 | **good** | **good** |
| **L** | 1,900 | 5 | 15.7 | 133 | 8 | 1,064 | 2,150 | 5.04 | 2.52 | **good** | **good** |
| M | 2,000 | 5 | 8.5 | 88.6 | 7.5 | 664.5 | 1,300 | 4.88 | 2.96 | good/regular | **good** |

Taking into account the good appearance of both phases (solid and liquid), the five tests that have proved effective are F, G, J, K and L, in which results have been obtained of 1.39 – 2.52 % TS, with a consumption of 5.04 – 9.04 kg/Tn DM. Figure 3.9

shows the results obtained from TS % and flocculant consumption of tests that have proven to be effective.

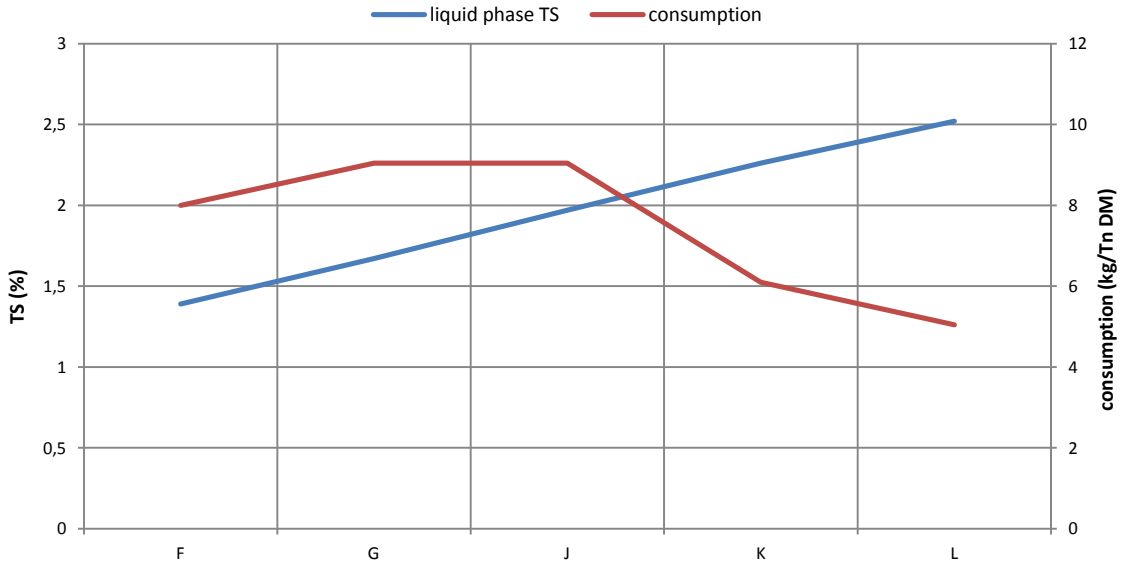



***Figure 3.9:*** *Results obtained in double centrifuge stage effectivity tests*

Taking into account the low flocculant consumption, the results obtained are 2.26 – 2.52 % TS. However, in test F a very favourable result was obtained with an average consumption with values of TS 1.39 % in the liquid phase with a consumption of 8 kg/Tn DM. Therefore, the following working conditions were established as the most favourable for double centrifuge stage with HYFLOC XT-653:

- Speed = 2,000 rpm
- Relative speed = 5
- PAR = 4.5 %
- Sludge flow = 7.5 $m^3$/h
- Flocculant flow = 2,300 L/h
- Flocculant concentration = 2.5 g/L

### *3.2.3. Implementation*

As seen in phase 2, since March 2017 the second centrifugation stage has been implemented as a normal working procedure with remarkable results in WWTP process where a substantial improvement in process performance has been observed and discharge quality.

Table 3.4 shows the COD, ammonium and solids data obtained in liquid phase of first and second centrifuge stages. And table 3.5 shows the working intervals in which centrifuges are operated since the second stage has been implemented as usual.

***Table 3.4:*** *Results obtained in de la double centrifuge stage implementation*

| | Centrifuge stage 1 liquid phase | Centrifuge stage 2 liquid phase |
|---|---|---|
| COD (mg $O_2$/L) | 62.350 | 25.765 |
| N-$NH_4^+$ (mg N/L) | 4.500 | 3.465 |
| TS (%) | 2.78 | 1.08 |

By dividing the flocculant between 2 centrifuge stages, the results of the first stage are good, however in the second stage they are very good obtaining a 1.08% TS and COD and ammonia loads (25,700 and 3,400 mg/L respectively), with a reduction between the first and second stages of TS 61.2 %, COD 58.7% and ammonium 23.0%.

The second centrifuge stage was very positive as COD, ammonium and TS values were much lower than the historical data (graphs of project phase 2), where the input COD to WWTP was 35,000 – 50,000 mg/L and is currently 25,000 mg $O_2$/L; in the case of ammonium there has been a reduction of 4,500 – 6,000 mg/L and is currently 3,000 mg N/L; and in the case of TS at the moment values of approximately 2% are being obtained versus 4% of the centrifuge liquid phase previously. Therefore, a PW with lower COD and ammonium loading is currently being treated, which facilitates the purification process and lower TS contents that help the nutrients and oxygen solubility and decrease the viscosity, obtaining a better performance of the biological degradation.

***Table 3.5:*** *Parameter intervals in the double centrifuge stage implementation*

| | Minimum value | Maximum value |
|---|---|---|
| Flocculant type | Hyfloc, Zetag or mixed | |
| Flocculant concentration (g/L) | 2.5 | 2.5 |
| Sludge flow centrifuge 1 ($m^3$/h) | 8.2 | 9.8 |
| Relative speed centrifuge 1 | 20 | 20 |
| DM liquid phase centrifuge 1 (%) | 6.98 | 10.13 |
| Sludge flow centrifuge 2 ($m^3$/h) | 7.6 | 9.3 |
| Relative speed centrifuge 2 | 5 | 5 |
| DM liquid phase centrifuge 2 (%) | 1.76 | 3.26 |

Figure 3.10 shows the DM daily content since its implementation in both first and second centrifuge stages.

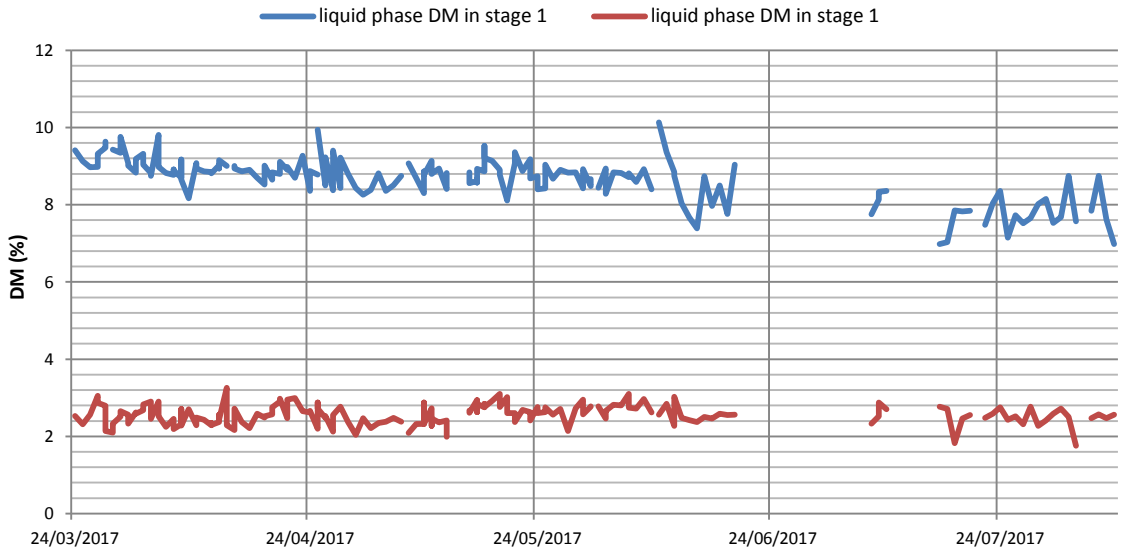



***Figure 3.10:*** *Liquid phase DM in centrifuge stage 1 and stage 2*

The double centrifugation stage process has been implemented as a standard procedure recently, so it is still in a process of adaption to find the most favourable working parameters. Over the last few days we have observed that the lowest liquid phase DM values in both stages are being reached.

## 3.3. PW solids separation by membranes

In parallel, PW solids separation was examined by other technologies not available at Ecoparc 2. The chosen technology was solids separation by membranes with different ranges (micro and ultrafiltration).

In order to carry out these tests, a collaboration with IREC (Catalonia Institute for Energy Research) was needed. The IREC had access to all the equipment necessary for this study.

### *3.3.1. Study approach*

It was decided to conduct a study on membranes separation based on two phases. First, the suspended particle size distribution was to be studied in order to determine which membrane type is best suited for the suspended solids contained in our effluent.

As a second phase, the separation of 12 different commercial membranes was studied in order to determine which of the 12 membranes was the most adequate, taking into account transmembrane pressure and fouling effects.

### *3.3.2. Suspended particle size distribution*

In the first step, the sample nature was observed under a microscope, observing two main distributions: one of approximately 1 micron range microorganisms (figure 3.11) and another of heterogeneous solids with a range from 10 microns to millimetres (figure 3.12).

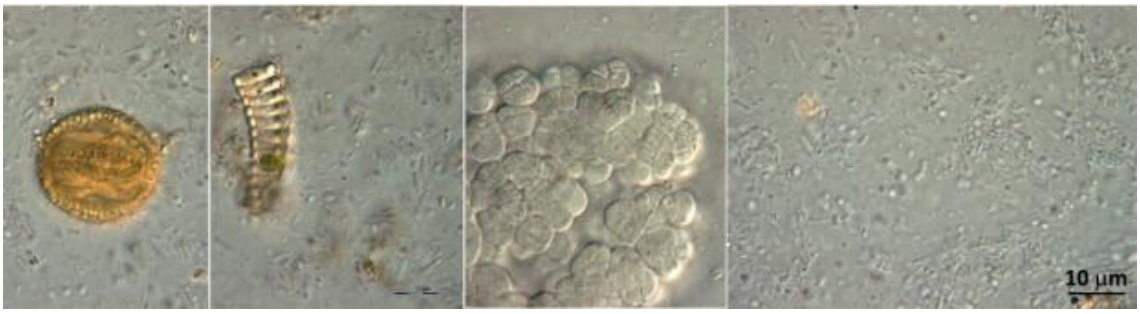



***Figure 3.11:*** *One micron range microorganisms*

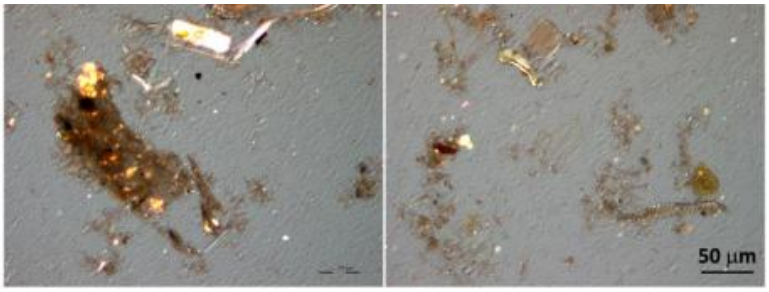



***Figure 3.12:*** *10 microns to millimetre range heterogeneous solids*

Next, the particle size distribution was studied with specialised equipment, where 2 main distributions were observed: one centered at 0.6 microns and one with a peak at 40 microns. Tests were performed with and without sonication and no significant differences were observed (figure 3.13), which assumes that there are no aggregates. It was also observed that there are solids larger than 100 microns which can be separated by sieving (figure 3.14). The sieve test was performed and it was observed that this pretreatment did not alter the size distribution unlike sonication application in this new sample (figure 3.15).

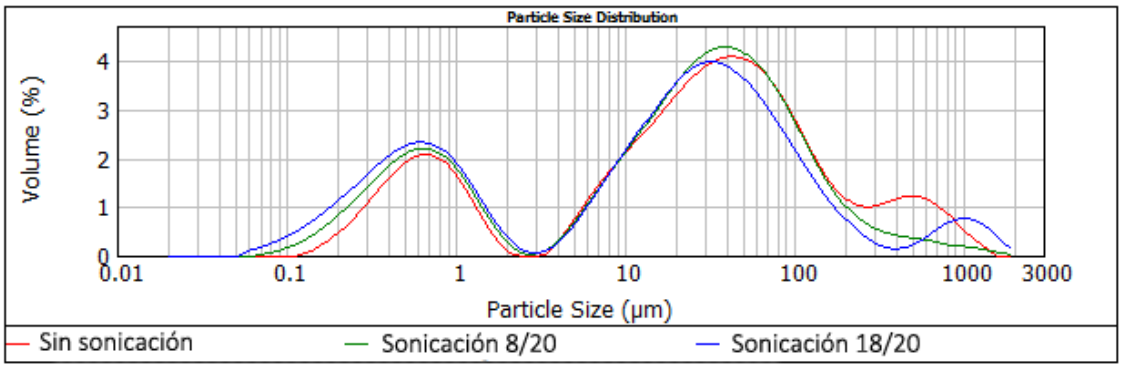



***Figure 3.13:*** *Particle size distribution with and without sonication*

***Figure 3.14:*** *Sieved sample*

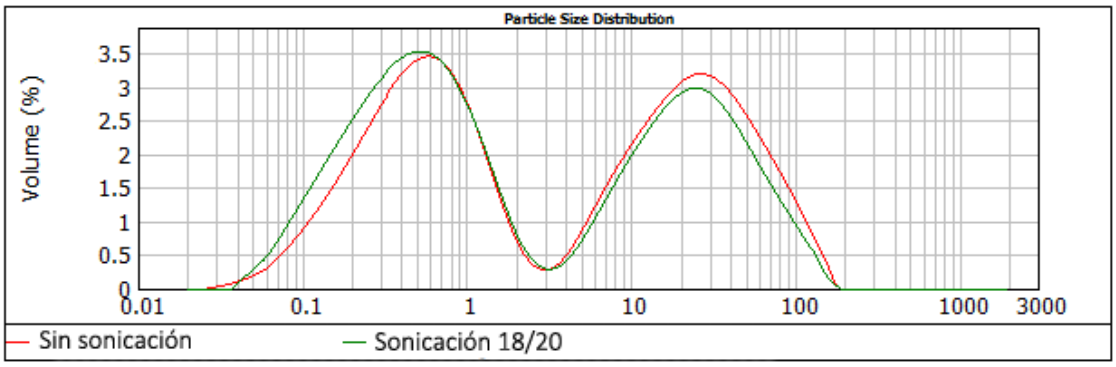



***Figure 3.15:*** *Sieved sample particle size distribution with and without sonication*

Another pretreatment that was studied was the dilution effect, noting that sample dilution did not alter the particle size distribution (figure 3.16).

Other parameters that were measured were the density and viscosity, where it was observed that the density was similar to water (1.009 g/L) but, however, the viscosity was about 17 times higher than water (16.92 cP). The sample viscosity was diluted to 50 and 25 % and a significant decrease in viscosity was observed (3.83 and 2.11 cP respectely).

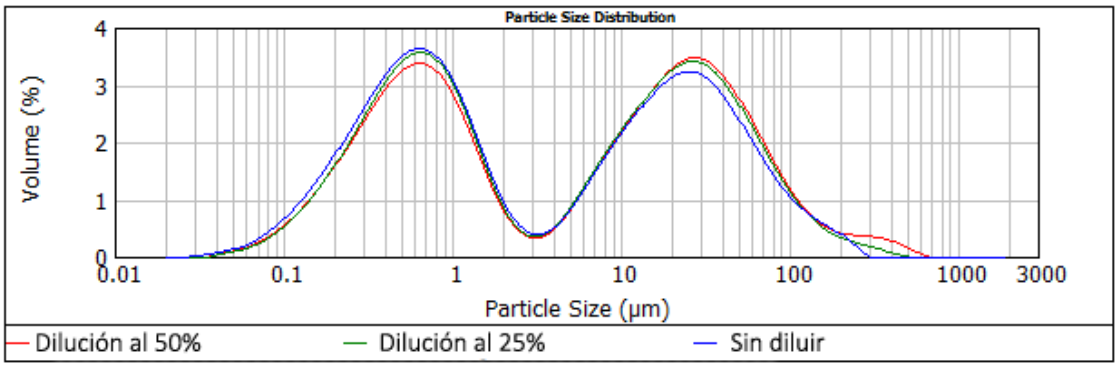



***Figure 3.16:*** *Sieved simple and diluted particle size distribution with and without sonication*

Finally, turbidity and concentration were studied. The sample turbidity sieved at 100 microns was found to be very high, with a concentration of 22.39 g/L of dry sample ashes free versus 38.11 g/L of total dry weight.

In conclusion, it was observed that sonication does not affect the sample particle size distribution. The initial sample contains particles greater than 100 microns that can be separated by a sieve and has a high viscosity that it was significantly reduced by way of dilution. Neither process was found to alter the particle size distribution. In addition, the permeate can be used to for the dilution , without needing to consume MW. Since the unsieved and undiluted samples do not allow membranes to be used, during the second phase of this study the samples were subjected to a sieving at 100 microns and a 50 % dilution pretreatment.

From the results obtained in this first phase it was decided to study the 12 membranes that appear in table 3.6. In figure 3.17 we can see a particle size distribution graph and the point at which each membrane is located.

***Table 3.6:*** *Membranes chosen for the study*

| Membrane | | Pore size |
|---|---|---|
| **M1** | PS35 | 0,003 µm |
| **M2** | PV400 | 0,010 µm |
| **M3** | UC100 | 0,013 µm |
| **M4** | PA400 | 0,015 µm |
| **M5** | PAN400 | 0,027 µm |

| Membrane | | Pore size |
|---|---|---|
| **M6** | MP005 | 0,050 µm |
| **M7** | 0.05Freud | 0,050 µm |
| **M8** | 0.1PE | 0,100 µm |
| **M9** | PV400R | 0,100 µm |
| **M10** | PV500 | 0,160 µm |
| **M11** | PES MF | 0,200 µm |
| **M12** | 1.0 PE | 1,000 µm |

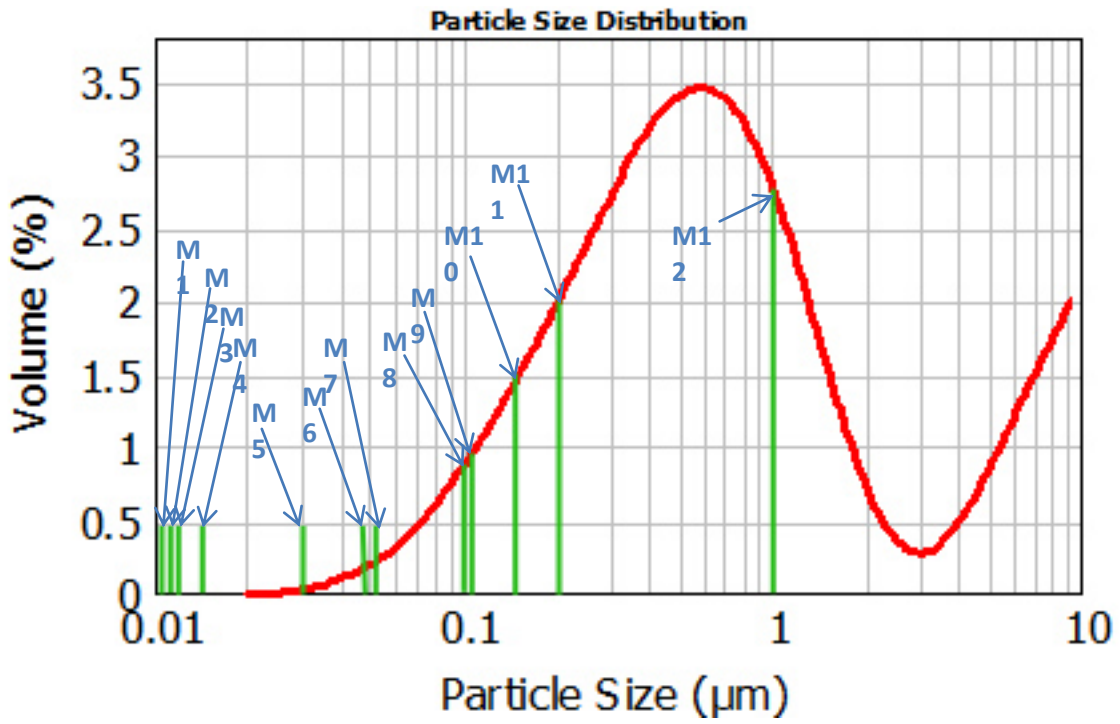



***Figure 3.17:*** *Particle size distribution with the point where each membrane is located*

### *3.3.3. Separation by different commercial membranes types*

In the second phase, six samples were analysed, which belong to three different points.

The first sampling point was the same one that was studied in the first phase: the centrifuge liquid (LC) (figure 3.18).

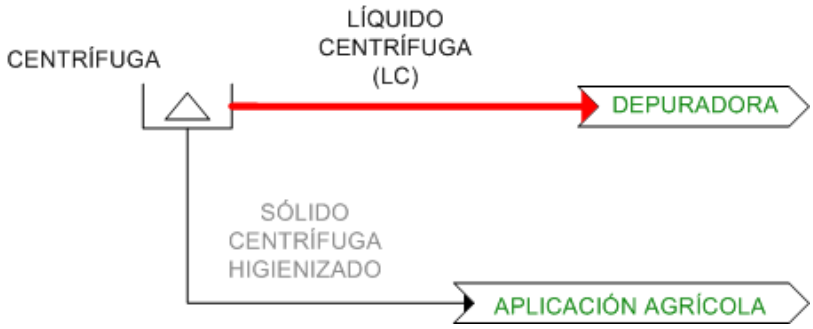



***Figure 3.18:*** *Centrifuge liquid sample*

As the study progressed, it was decided to modify the process by subjecting the effluent to a double centrifuge stage, so that the second sampling point was the liquid obtained from the second centrifuge stage (LC-DC) (figure 3.19).

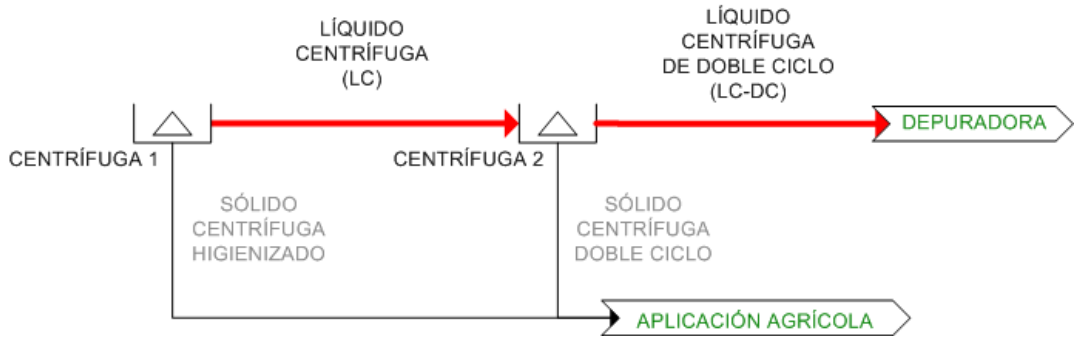



***Figure 3.19:*** *Centrifuge liquid from the second centrifuge stage sample*

As mentioned in phase 1, the centrifuge liquid accounts for between 75 and 97 % of the PW in the WWTP, so the third sampling point consisted of the PW which is introduced into the WWTP after sieving at 800 microns (LIX) (figure 3.20).

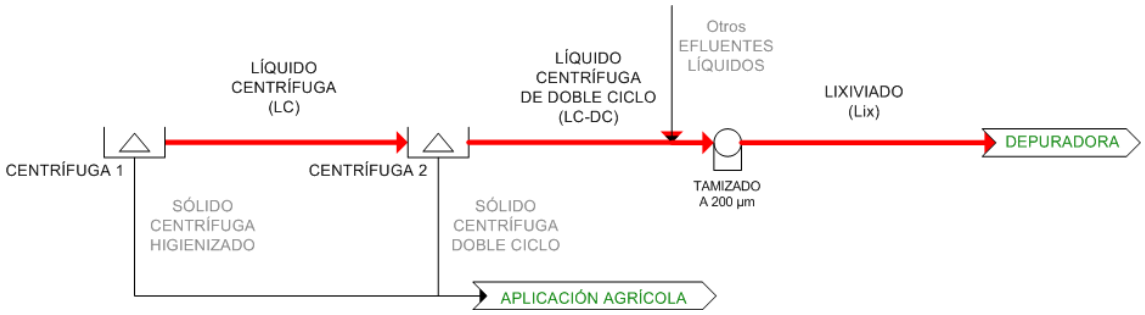



***Figure 3.20:*** *PW sample*

Therefore, the results obtained in different membranes are not comparable to each other because they are tested with different samples.

### 3.3.3.1. Membrane 1: PS35

Table 3.7 shows the results obtained in membrane 1, which was tested with the centrifuge liquid sample.

***Table 3.7:*** *Results obtained in membrane 1*

| Parameter | Value |
|---|---|
| **Pore size** | 0.003 µm |
| **Sample** | 2 – 20161017 (LC) |
| **Permeability with water in virgin membrane** | 353 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.69 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 5.39 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,800 mL |
| **Concentration factor** | 1.31 |
| **Initial turbidity** | 11.34 |
| **Detained turbidity** | 12.99 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.25 |

Figure 3.21 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has a peak at 0.6 and 15 microns.

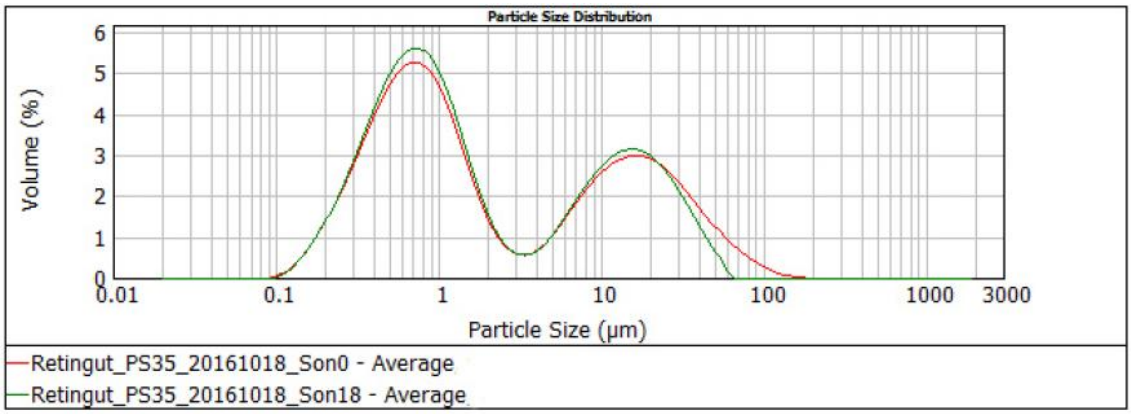



***Figure 3.21:*** *Particle size distribution with and without sonication from membrane 1 sample*

Figure 3.22 shows the decrease in permeability as a function of time, which is 1.69 L/h/m$^2$/bar, after cleaning a permeability of 5.39 L/h/m$^2$/bar can be recovered with water.

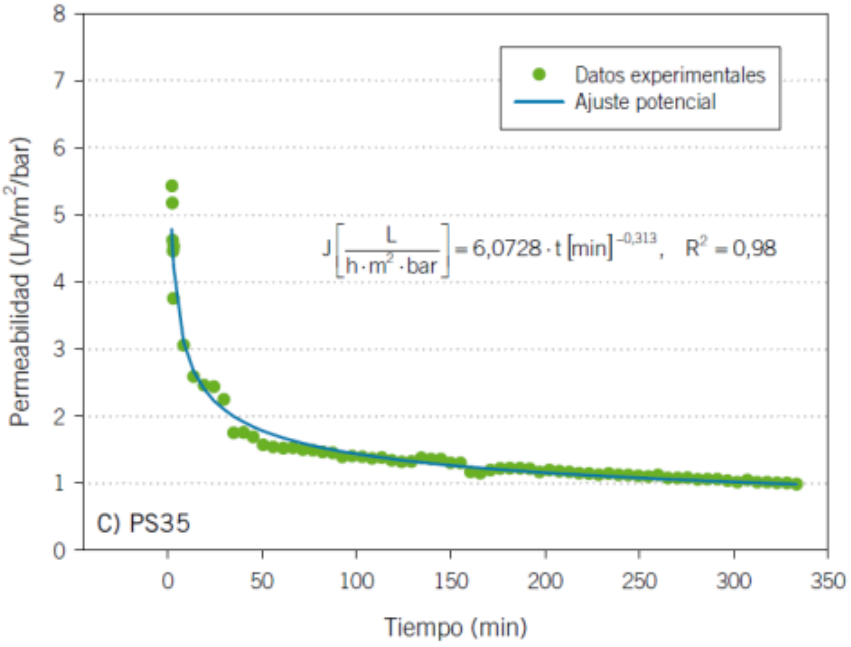



***Figure 3.22:*** *Permeability graph with membrane 1*

Figures 3.23 and 3.24 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate appear to be free of suspended solids, with a turbidity of 0.03.


***Figure 3.23:*** *Photograph of membrane 1 after test*


***Figure 3.24:*** *Retained and permeate samples from membrane 1 test*

Figure 3.25 shows retained particle size distribution. Compared with initial sample, no significant differences were observed.

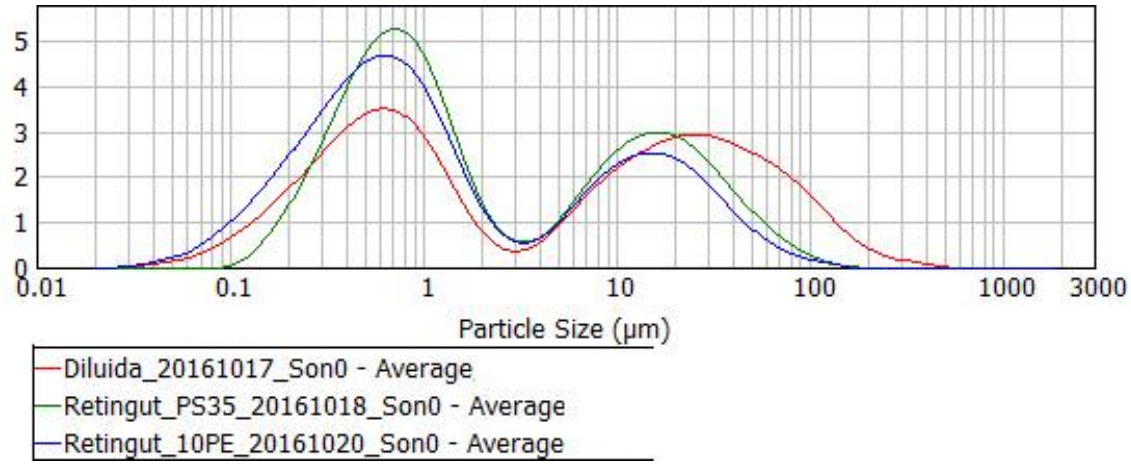



***Figure 3.25:*** *Retained particle size distribution from membrane 1 test*

### 3.3.3.2. Membrane 2: PV400

Table 3.8 shows the results obtained in membrane 2, which was tested with the centrifuge liquid of double stage sample.

***Table 3.8:*** *Results obtained in membrane 2*

| parameter | value |
|---|---|
| **Pore size** | 0.010 µm |
| **Sample** | 3 – 20161024 (LC-DC) |
| **Permeability with water in virgin membrane** | 405 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.87 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 40.30 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,750 mL |
| **Concentration factor** | 1.41 |
| **Initial turbidity** | 6.49 |
| **Detained turbidity** | 8.59 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.30 |

Figure 3.26 shows the sieved and diluted sample particle size distribution with and without sonication. In this case, the distribution is different than we expected with two peaks at 0.15 and 0.7 microns and one less pronounced peak at 15 microns. With this we observe that the particle size distribution does vary with the second centrifuge stage.

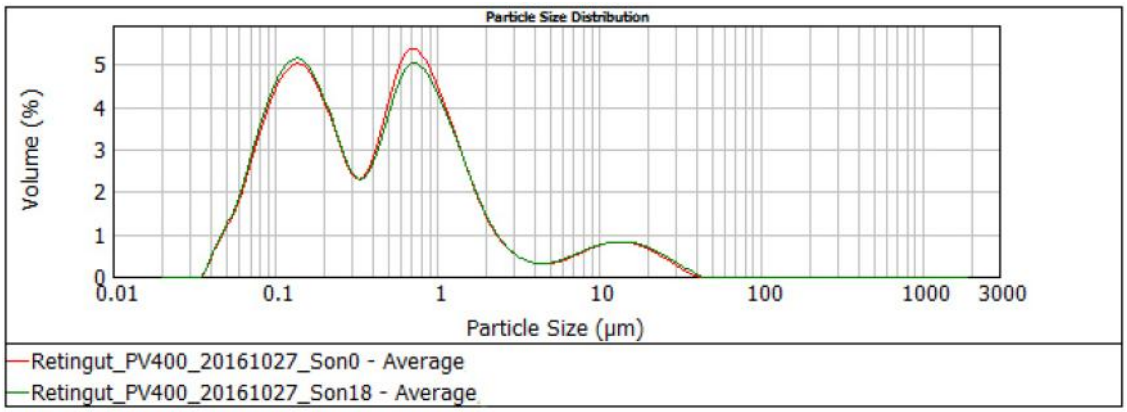



***Figure 3.26:*** *Particle size distribution with and without sonication for membrane 2 sample*

Figure 3.27 shows the decrease in permeability as a function of time, which is 1.87 L/h/m$^2$/bar, after cleaning a permeability of 40.30 L/h/m$^2$/bar can be recovered with water.

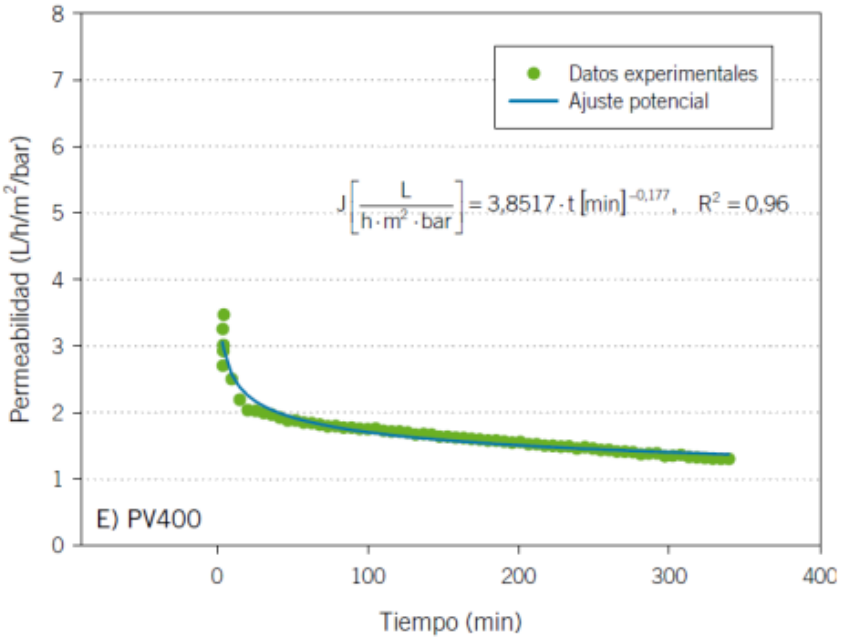



***Figure 3.27:*** *Permeability graph for membrane 2*

Figures 3.28 and 3.29 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.03.


***Figure 3.28:*** *Photograph of membrane 2 after test*

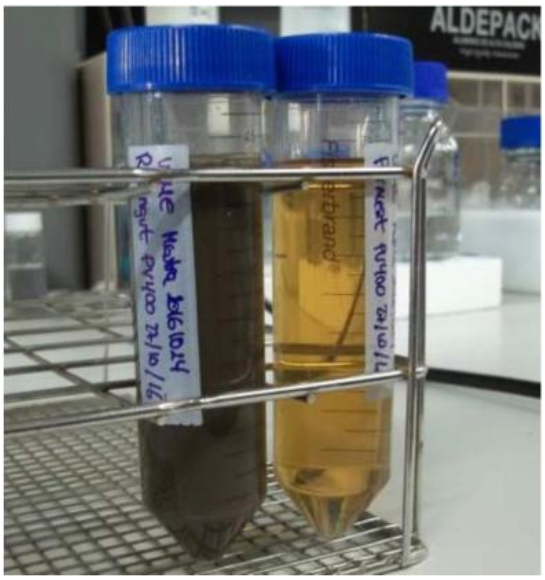



***Figure 3.29:*** *Retained and permeate samples from membrane 2 test*

Figure 3.30 shows the detained particle size distribution. Compared with the initial sample, no significant differences were observed.

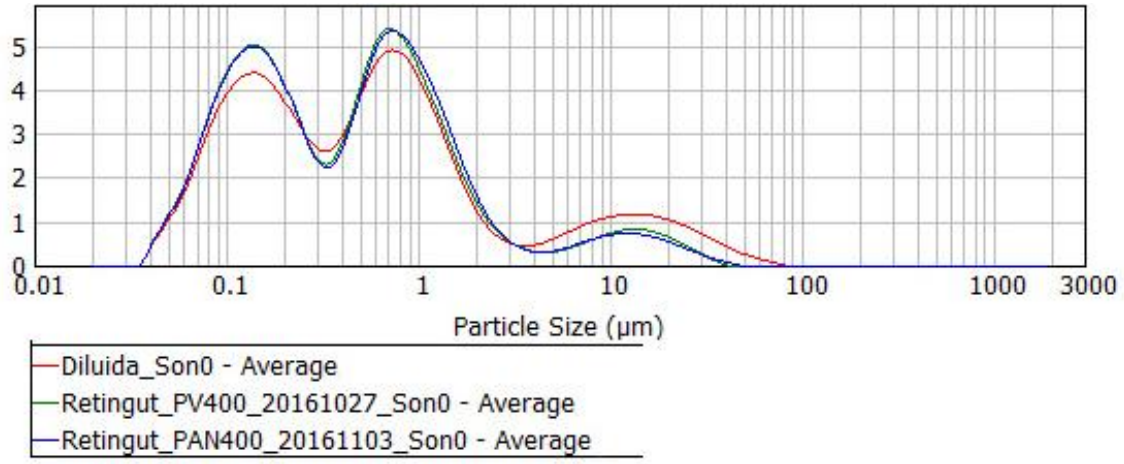



***Figure 3.30:*** *Retained particle size distribution in membrane 2 test*

### 3.3.3.3. Membrane 3: UC100

Table 3.9 shows the results obtained for membrane 3, which was tested with the centrifuge liquid sample.

***Table 3.9:*** *Results obtained for membrane 3*

| parameter | value |
|---|---|
| **Pore size** | 0.013 µm |
| **Sample** | 5 – 20161121 (LC) |
| **Permeability with water in virgin membrane** | 149 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.27 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 7.18 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,750 mL |
| **Concentration factor** | 1.21 |
| **Initial turbidity** | 7.67 |
| **Detained turbidity** | 7.95 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.35 |

Figure 3.31 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has a peak at 0.6 and 15 microns.

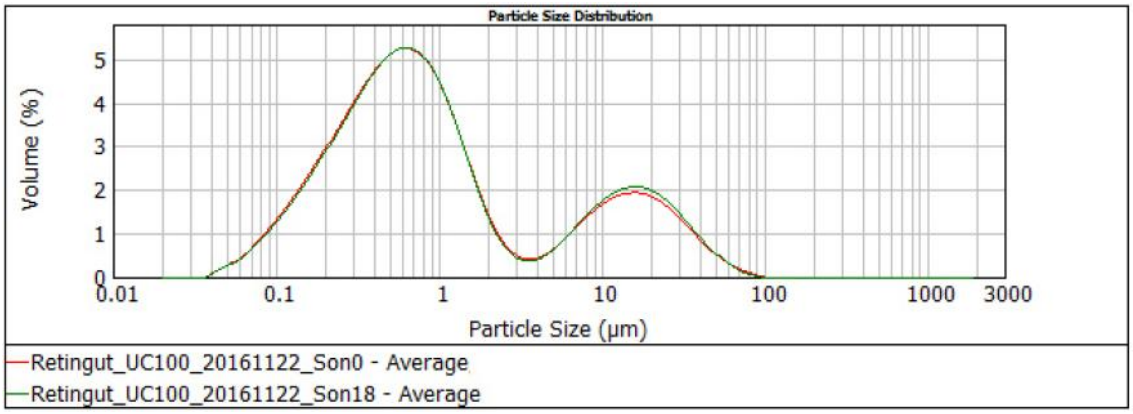



***Figure 3.31:*** *Particle size distribution witht and without sonication for membrane 3 sample*

Figure 3.32 shows the decrease in permeability as a function of time, which is 1.27 L/h/m$^2$/bar, after cleaning a permeability of 7.18 L/h/m$^2$/bar can be recovered with water.

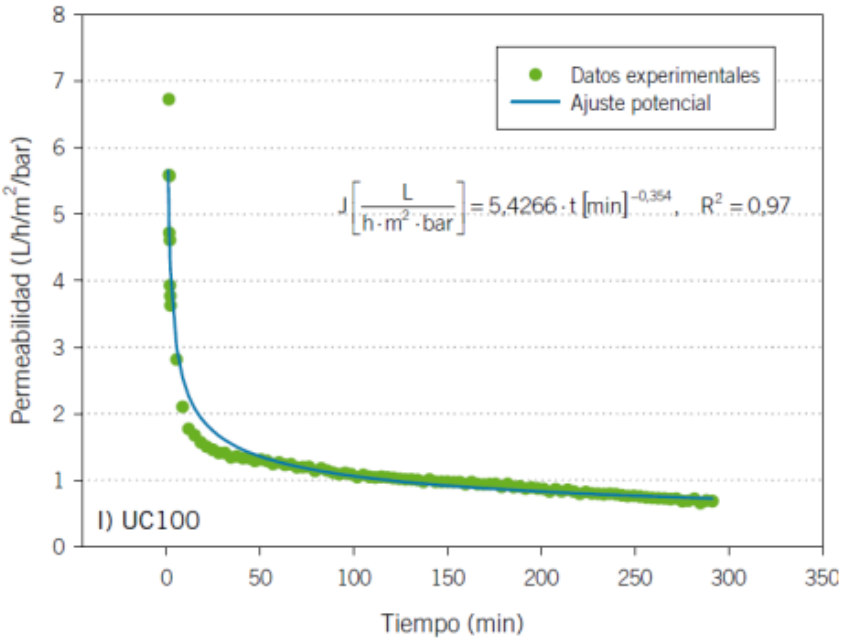



***Figure 3.32:*** *Permeability graph for membrane 3*

Figures 3.33 and 3.34 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.03.


***Figure 3.33:*** *Photograph of membrane 3 after test*

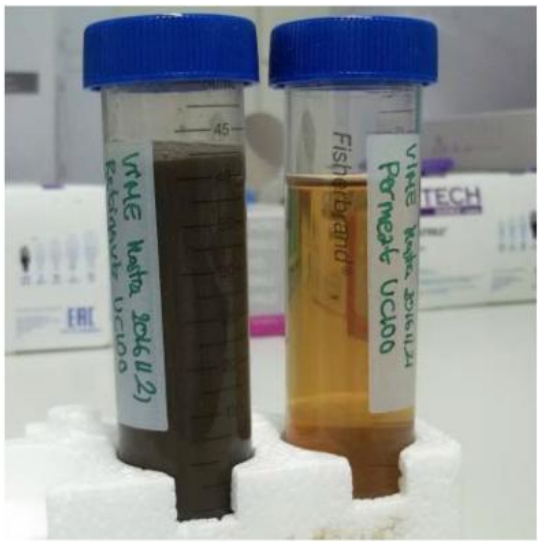



***Figure 3.34:*** *Retained and permeate samples from membrane 3 test*

Figure 3.35 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

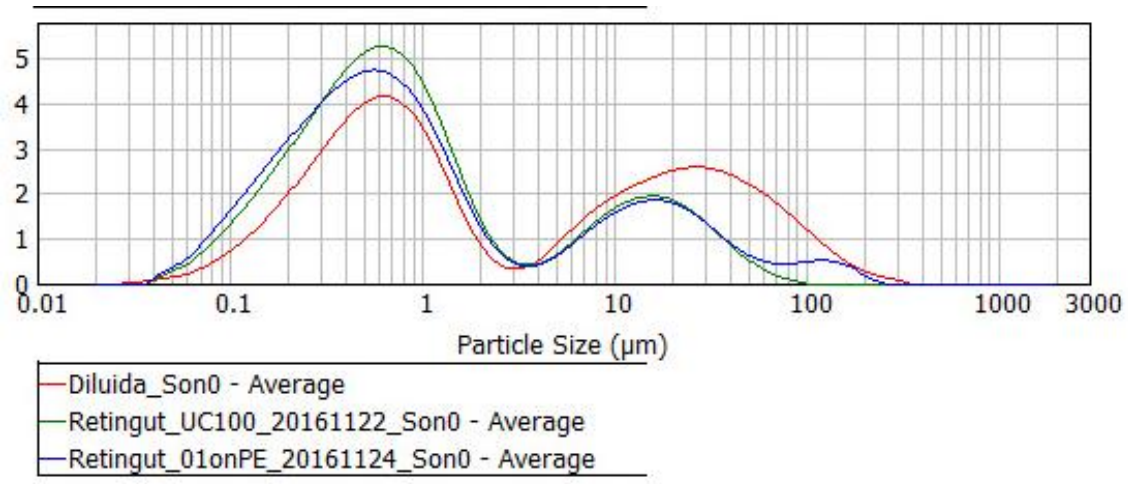



***Figure 3.35:*** *Retained particle size distribution in membrane 3 test*

### 3.3.3.4. Membrane 4: PA400

Table 3.10 shows the results obtained for membrane 4, which was tested with the centrifuge liquid sample.

***Table 3.10:*** *Results obtained for membrane 4*

| parameter | value |
|---|---|
| **Pore size** | 0.015 µm |
| **Sample** | 1 – 20161003 (LC) |
| **Permeability with water in virgin membrane** | 806 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.69 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 4.48 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,800 mL |
| **Concentration factor** | 1.29 |
| **Initial turbidity** | 10.77 |
| **Detained turbidity** | 11.78 |
| **Permeate turbidity** | 0.04 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.40 |

Figure 3.36 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has a peak at 0.6 and 15 microns.

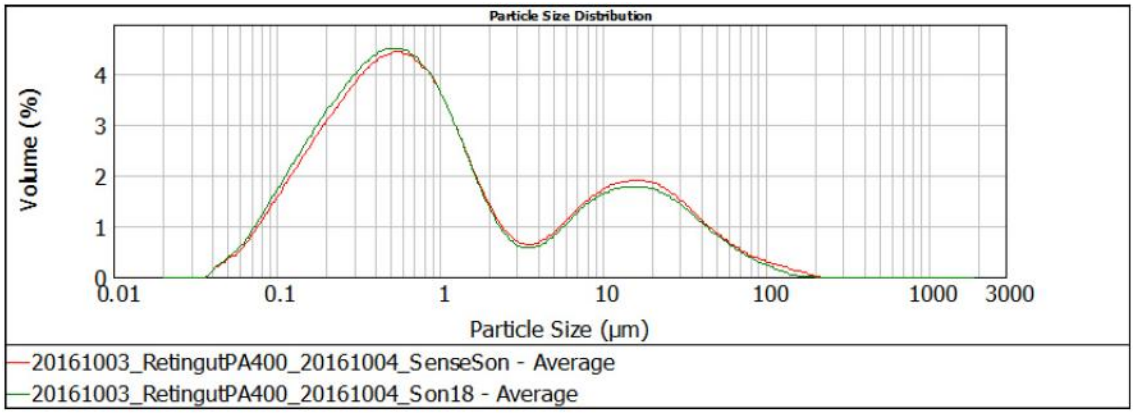



***Figure 3.36:*** *Particle size distribution with and without sonication for membrane 4 sample*

Figure 3.37 shows the decrease in permeability as a function of time which is 1.69 L/h/m$^2$/bar, after cleaning a permeability of 4.48 L/h/m$^2$/bar can be recovered with water.

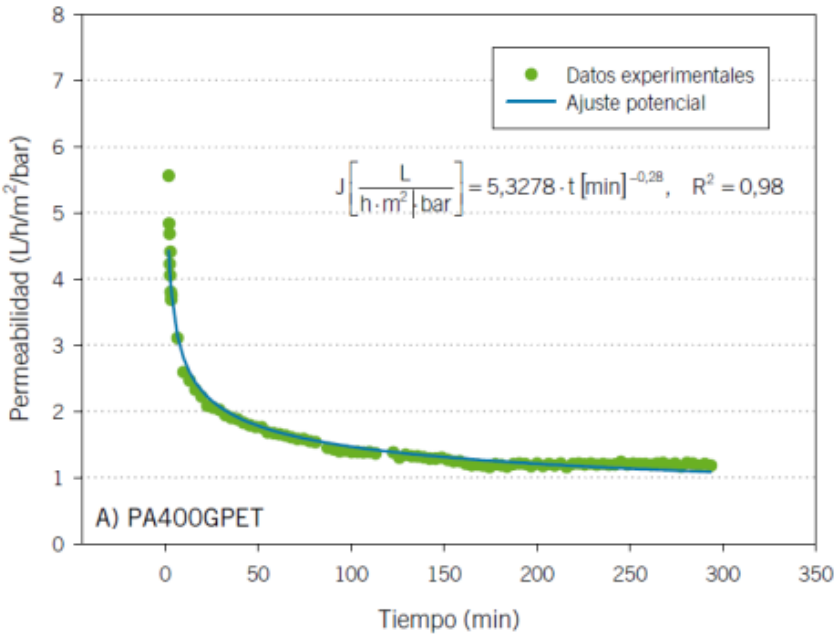



***Figure 3.37:*** *Permeability graph for membrane 4*

Figures 3.38 and 3.39 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.04.


***Figure 3.38:*** *Photograph of membrane 4 after test*

***Figure 3.39:*** *Retained and permeate samples from membrane 4 test*

Figure 3.40 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

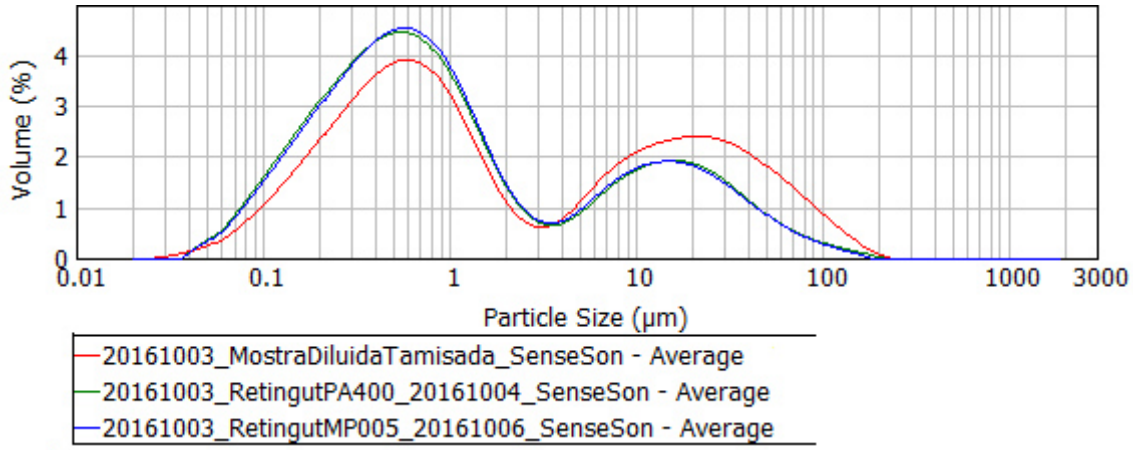



***Figure 3.40:*** *Retained particle size distribution from membrane 4 test*

### 3.3.3.5. Membrane 5: PAN400

Table 3.11 shows the results obtained in membrane 5, which was tested with the centrifuge liquid of double stage sample.

***Table 3.11:*** *Results obtained in membrane 5*

| parameter | value |
|---|---|
| **Pore size** | 0.027 µm |
| **Sample** | 3 - 20161024 (LC-DC) |
| **Permeability with water in virgin membrane** | 48 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 2.07 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 29.04 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,750 mL |
| **Concentration factor** | 1.55 |
| **Initial turbidity** | 6.49 |
| **Detained turbidity** | 10.27 |
| **Permeate turbidity** | 0.02 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.45 |

Figure 3.41 shows the sieved and diluted sample particle size distribution with and without sonication. In this case, the distribution is different than we expected, with two peaks at 0.15 and 0.7 microns and one less pronounced peak at 15 microns. Here we observe that particle size distribution does vary with the second centrifuge stage.

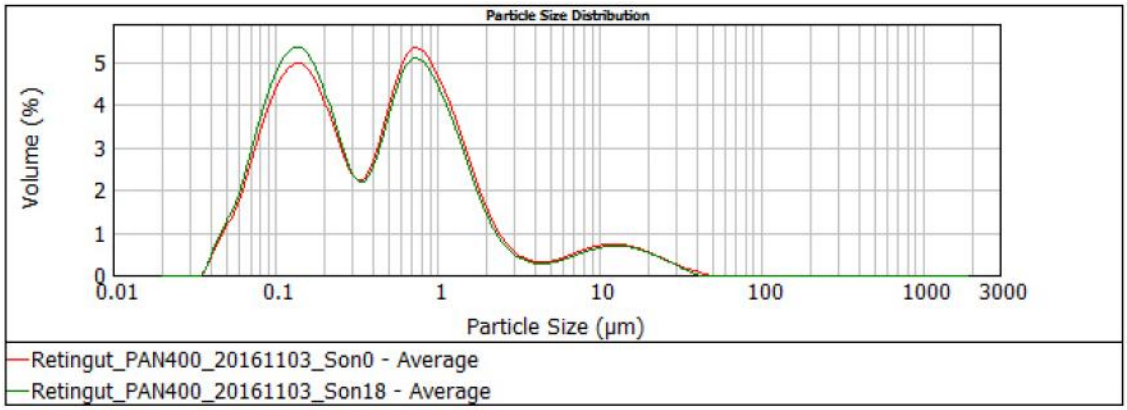



***Figure 3.41:*** *Particle size distribution with and without sonication for membrane 5 sample*

Figure 3.42 shows the decrease in permeability as a function of time which is 2.07 L/h/m$^2$/bar, after cleaning a permeability of 29.04 L/h/m$^2$/bar can be recovered with water.

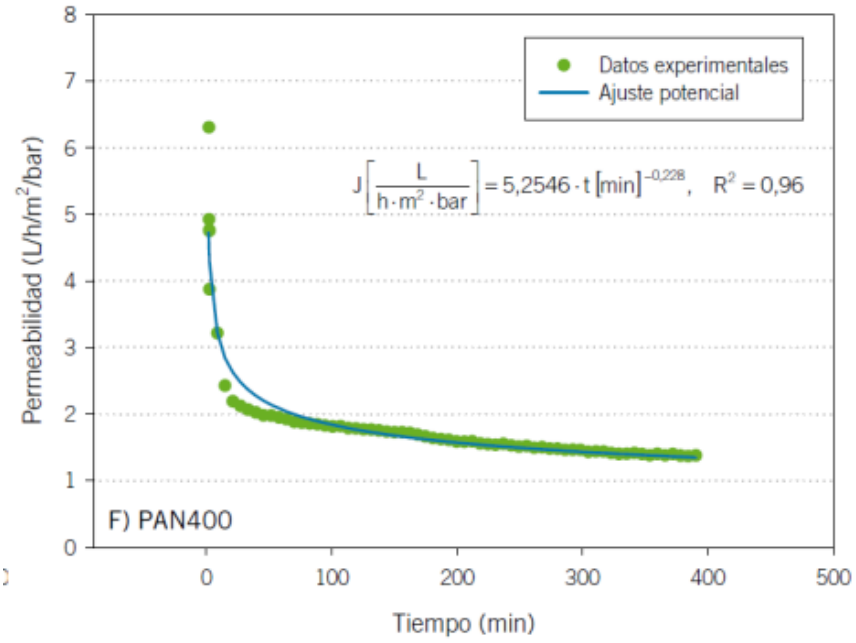



***Figure 3.42:*** *Permeability graph for membrane 5*

Figures 3.43 and 3.44 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.02.


***Figure 3.43:*** *Photograph of membrane 5 after test*

***Figure 3.44:*** *Retained and permeate samples in membrane 5 test*

Figure 3.45 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

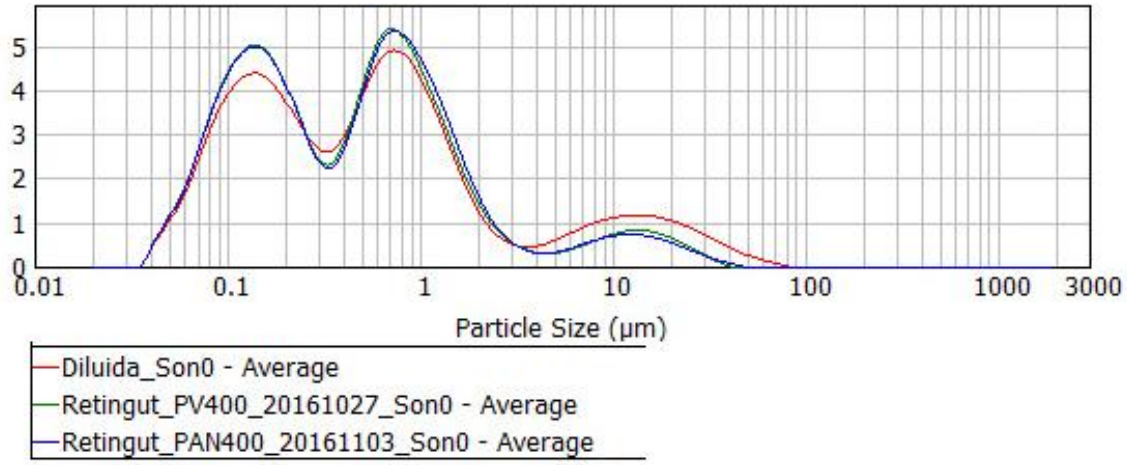



***Figure 3.45:*** *Retained particle size distribution in membrane 5 test*

### 3.3.3.6. Membrane 6: MP005

Table 3.12 shows the results obtained for membrane 6, which was tested with the centrifuge liquid sample.

***Table 3.12:*** *Results obtained for membrane 6*

| parameter | value |
|---|---|
| **Pore size** | 0.050 µm |
| **Sample** | 1 - 20161003 (LC) |
| **Permeability with water in virgin membrane** | 172 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.82 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 8.32 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,725 mL |
| **Concentration factor** | 1.39 |
| **Initial turbidity** | 10.77 |
| **Detained turbidity** | 14.17 |
| **Permeate turbidity** | 0.04 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.50 |

Figure 3.46 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has a peak at 0.6 and 15 microns.

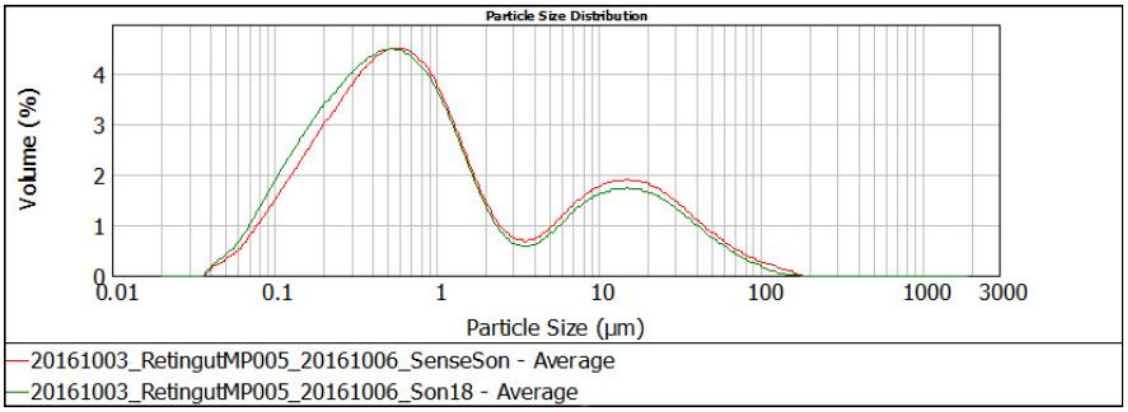



***Figure 3.46:*** *Particle size distribution with and without sonication for membrane 6 sample*

Figure 3.47 shows the decrease in permeability as a function of time, which is 1.82 L/h/m$^2$/bar, after cleaning a permeability of 8.32 L/h/m$^2$/bar can be recovered with water.

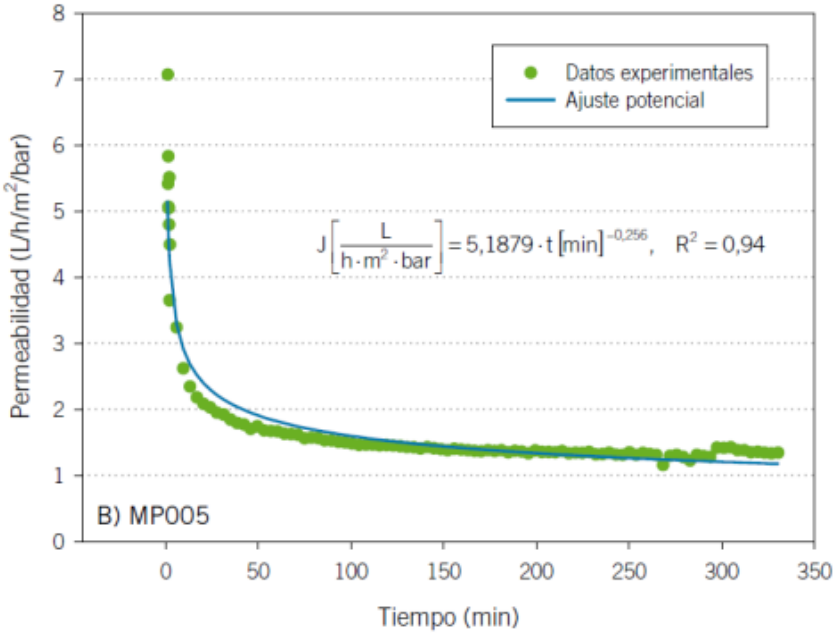



***Figure 3.47:*** *Permeability graph for membrane 6*

Figures 3.48 and 3.49 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.04.


***Figure 3.48:*** *Photograph of membrane 6 after test*

***Figure 3.49:*** *Retained and permeate samples for membrane 6 test*

Figure 3.50 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

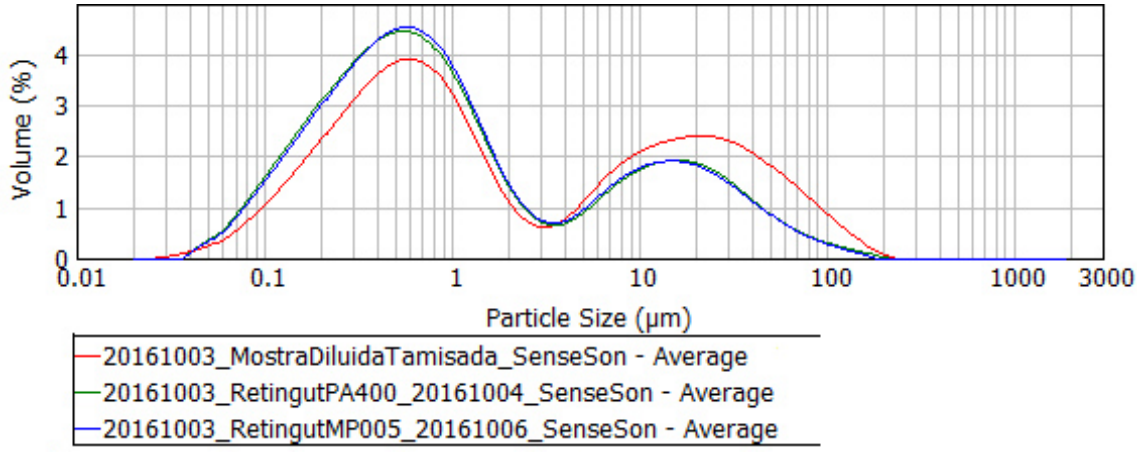



***Figure 3.50:*** *Retained particle size distribution for membrane 6 test*

### 3.3.3.7. Membrane 7: 0,05 on Freud

Table 3.13 shows the results obtained in membrane 7, which was tested with the centrifuge liquid sample.

***Table 3.13:*** *Results obtained in membrane 7*

| parameter | value |
|---|---|
| **Pore size** | 0.050 µm |
| **Sample** | 4 - 20161107 (LC) |
| **Permeability with water in virgin membrane** | 936 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.18 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 3.40 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,850 mL |
| **Concentration factor** | 1.23 |
| **Initial turbidity** | 11.18 |
| **Detained turbidity** | 12.33 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.55 |

Figure 3.51 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has peaks at 0.6 and 15 microns.

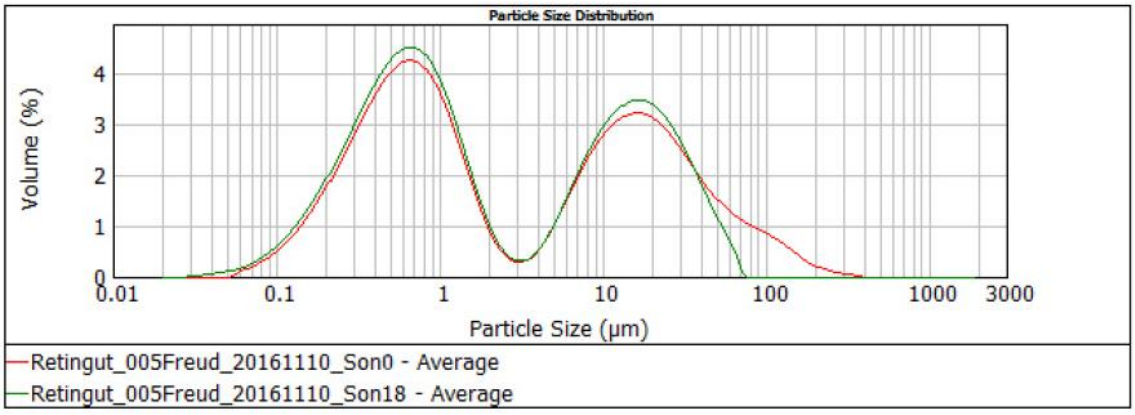



***Figure 3.51:*** *Particle size distribution with and without sonication for membrane 7 sample*

Figure 3.52 shows the decrease in permeability as a function of time which is 1.18 L/h/m$^2$/bar, after cleaning a permeability of 3.40 L/h/m$^2$/bar can be recovered with water.

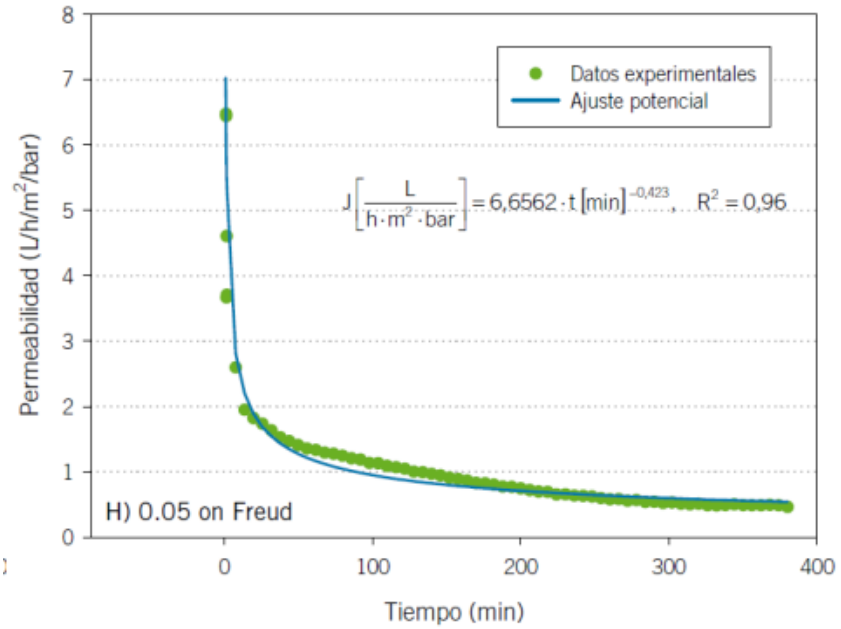



***Figure 3.52:*** *Permeability graph of membrane 7*

Figures 3.53 and 3.54 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.03.


***Figure 3.53:*** *Photographs of membrane 7 after test*

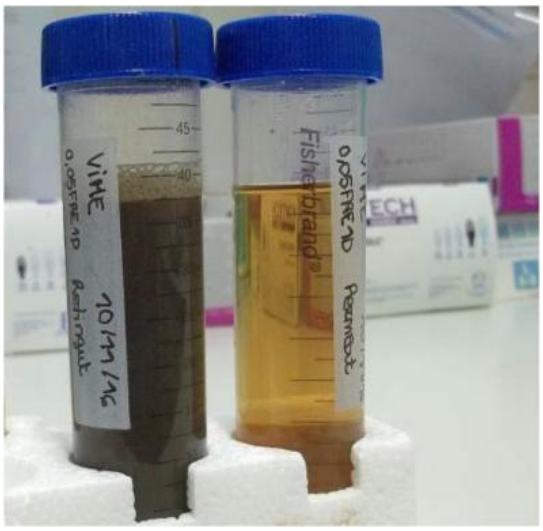


***Figure 3.54:*** *Retained and permeate samples for membrane 7 test*

Figure 3.55 shows the retained particle size distribution. Compared with the initial sample, small differences are observed in which the dilution assumes a decrease of 1% of solids with a size of 0.6 microns and a displacement of the peak of 15 microns to 25.

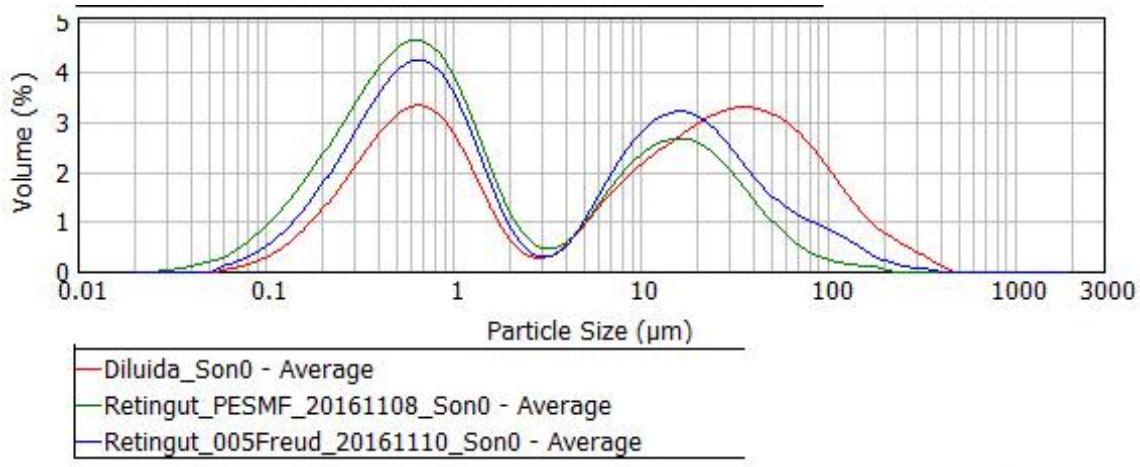



***Figure 3.55:*** *Retained particle size distribution for membrane 7 test*

### 3.3.3.8. Membrane 8: 0.1 on PE

Table 3.14 shows the results obtained for membrane 8, which was tested with the centrifuge liquid sample.

***Table 3.14:*** *Results obtained for membrane 8*

| parameter | value |
|---|---|
| **Pore size** | 0.100 µm |
| **Sample** | 5 – 20161121 (LC) |
| **Permeability with water in virgin membrane** | 1,176 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.43 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 22.99 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,800 mL |
| **Concentration factor** | 1.29 |
| **Initial turbidity** | 7.67 |
| **Detained turbidity** | 8.56 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.60 |

Figure 3.56 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has peaks at 0.6 and 15 microns.

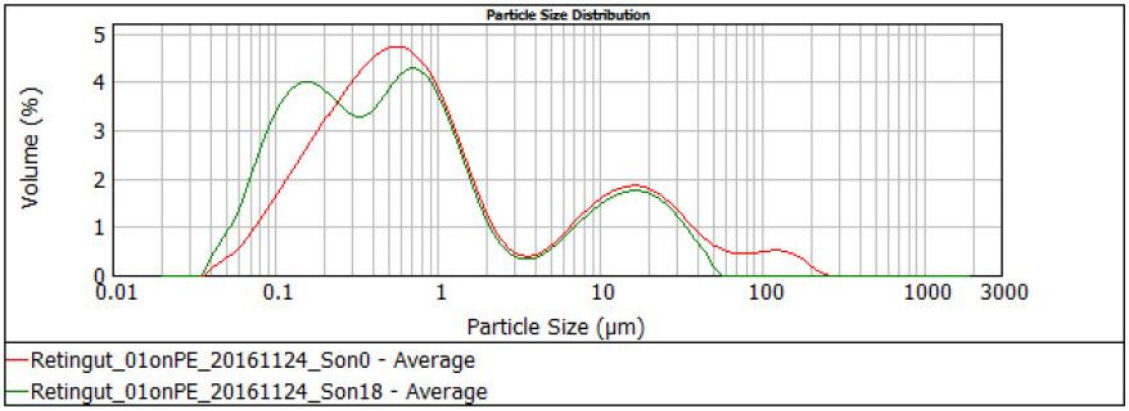



***Figure 3.56:*** *Particle size distribution with and without sonication for membrane 8 sample*

Figure 3.57 shows the decrease in permeability as a function of time which is 1.43 L/h/m$^2$/bar, after cleaning a permeability of 22.99 L/h/m$^2$/bar can be recovered with water.

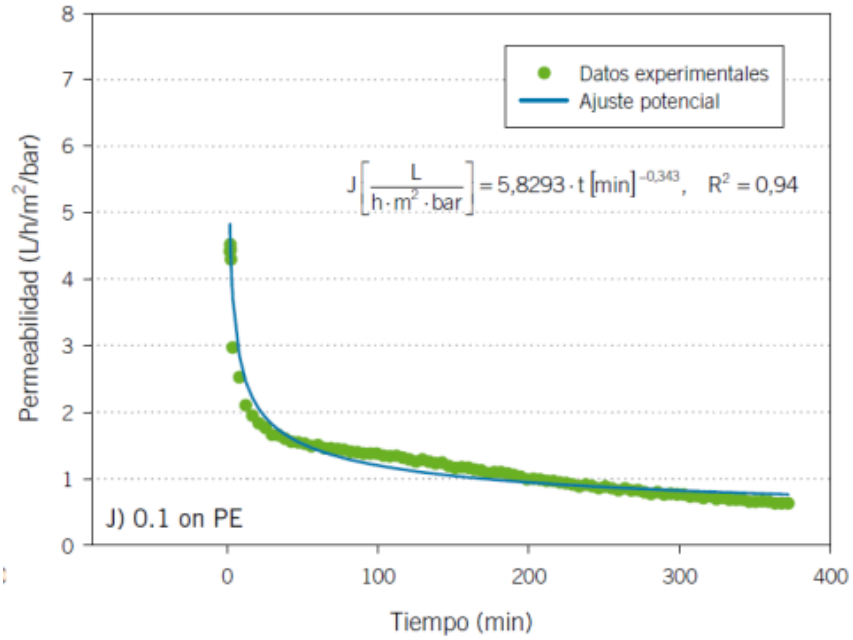



***Figure 3.57:*** *Permeability graph for membrane 8*

Figures 3.58 and 3.59 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.03.


***Figure 3.58:*** *Photograph of membrane 8 after test*

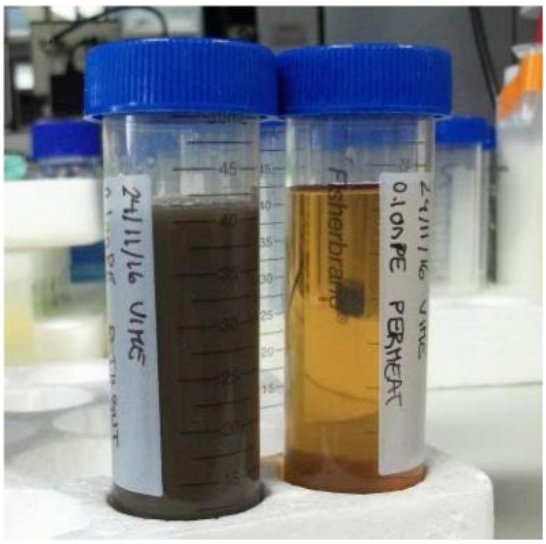



***Figure 3.59:*** *Retained and permeate samples for membrane 8 test*

Figure 3.60 shows the retained particle size distribution. Compared with the initial sample, small differences are observed in which the dilution supposes a decrease of 0.5% of solids with size of 0.6 microns and a displacement of the peak of 15 microns to 25 with an increase of 0.6%.

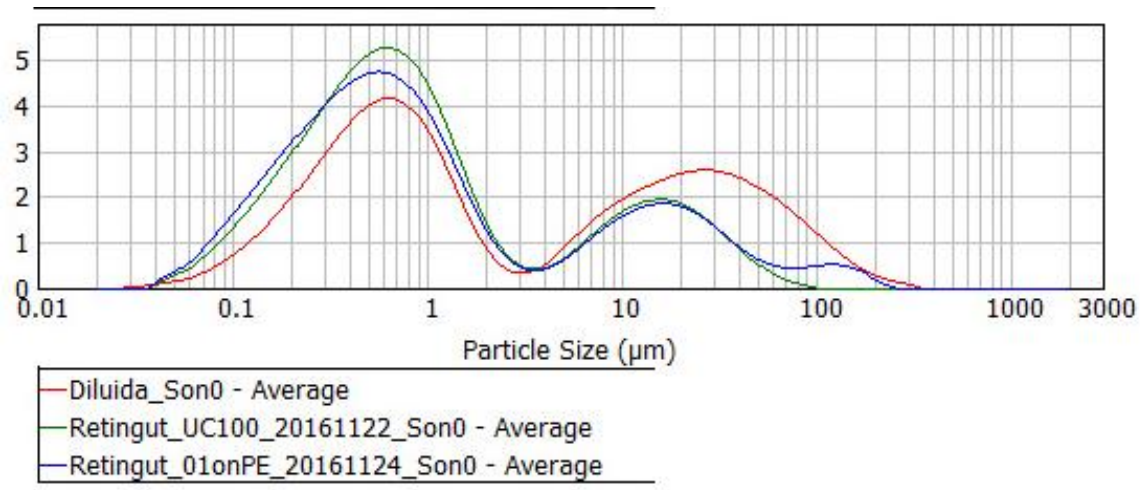



***Figure 3.60:*** *Retained particle size distribution for membrane 8 test*

### 3.3.3.9. Membrane 9: PV400R

Table 3.15 shows the results obtained for membrane 9, which was tested with the PW sample.

***Table 3.15:*** *Results obtained for membrane 9*

| parameter | value |
|---|---|
| **Pore size** | 0.100 µm |
| **Sample** | 6 - 20161128 (LIX) |
| **Permeability with water in virgin membrane** | 856 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.37 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 0.55 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,850 mL |
| **Concentration factor** | 1.28 |
| **Initial turbidity** | 12.24 |
| **Detained turbidity** | 16.38 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.65 |

Figure 3.61 shows the sieved and diluted sample particle size distribution with and without sonication. In this case two peaks are observed at 0.6 and 10 microns, the one at 10 microns being more pronounced than in the previous cases.

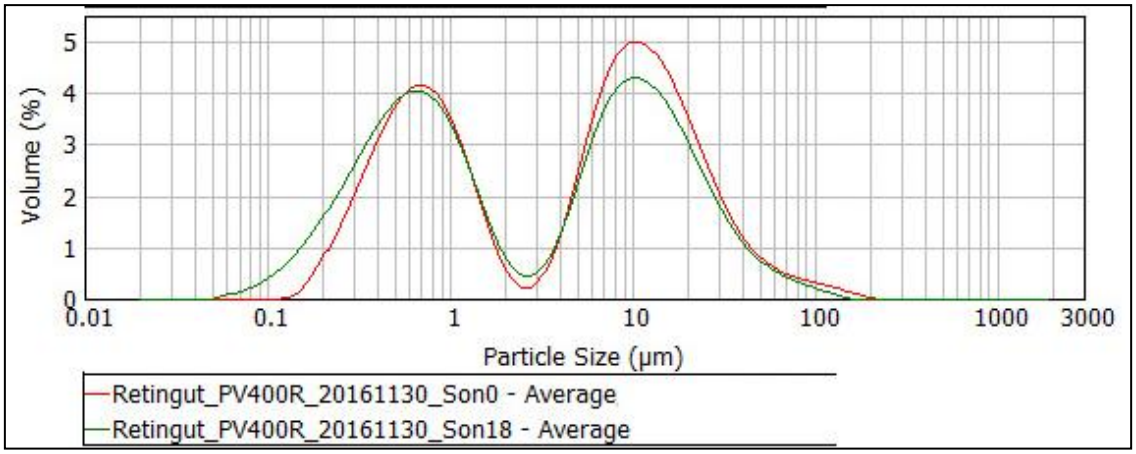



***Figure 3.61:*** *Particle size distribution with and without sonication for membrane 9 sample*

Figure 3.62 shows the decrease in permeability as a function of time which is 1.37 L/h/m$^2$/bar, after cleaning a permeability of 0.55 L/h/m$^2$/bar can be recovered with water, lower than the sample permeability.

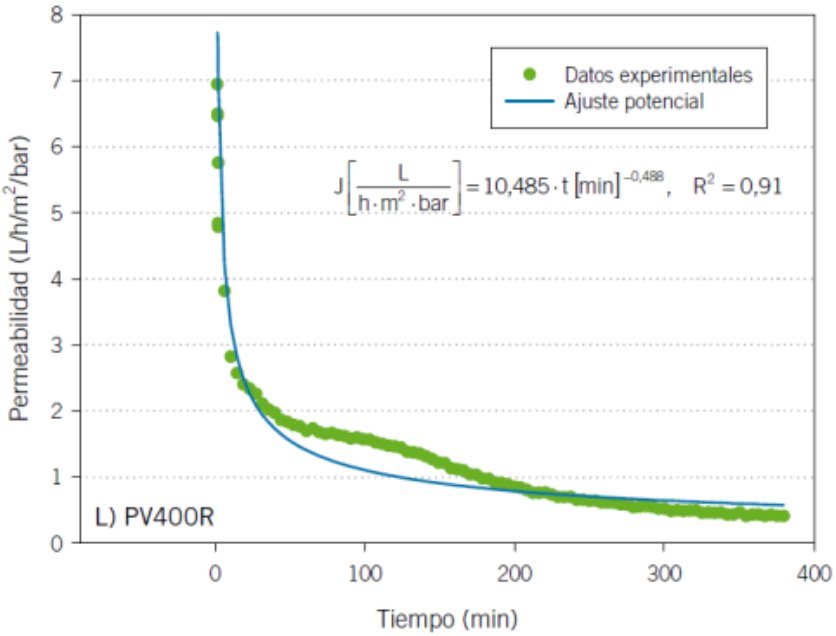



***Figure 3.62:*** *Permeability graph for membrane 9*

Figures 3.63 and 3.64 show the membrane after the test and the retained and permeate samples. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.03.


***Figure 3.63:*** *Photograph of membrane 9 after test*


***Figure 3.64:*** *Retained and permeate samples for membrane 9 test*

Figure 3.65 shows the detained particle size distribution. Compared with the initial sample, no significant differences were observed.

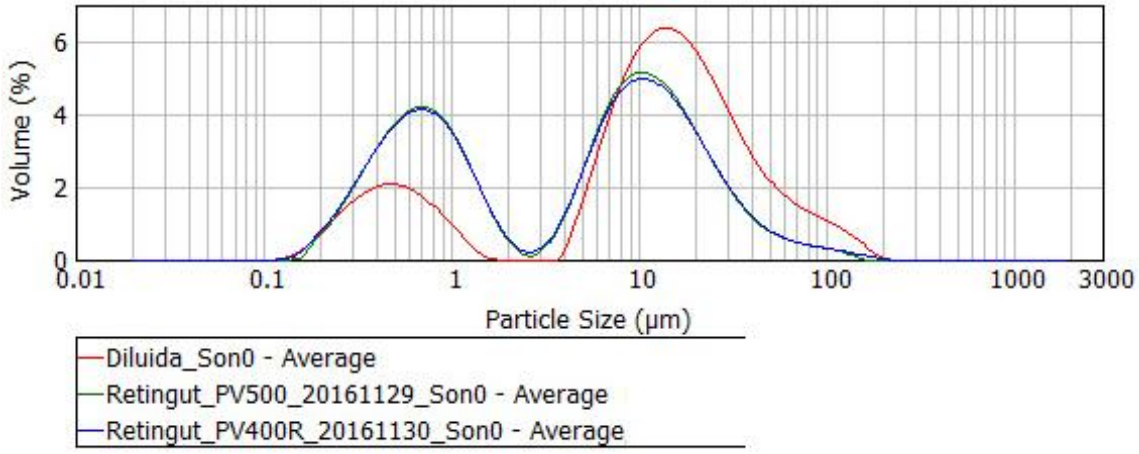



***Figure 3.65:*** *Retained particle size distribution for membrane 9 test*

### 3.3.3.10. Membrane 10: PV500

Table 3.16 shows the results obtained in membrane 10, which was tested with the PW sample.

***Table 3.16:*** *Results obtained in membrane 10*

| parameter | value |
|---|---|
| **Pore size** | 0.160 µm |
| **Sample** | 6 - 20161128 (LIX) |
| **Permeability with water in virgin membrane** | 562 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.42 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 0.68 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,800 mL |
| **Concentration factor** | 1.29 |
| **Initial turbidity** | 12.24 |
| **Detained turbidity** | 16.38 |
| **Permeate turbidity** | 0.02 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.70 |

Figure 3.66 shows the sieved and diluted sample particle size distribution with and without sonication. In this case two peaks are observed at 0.6 and 10 microns, the one at 10 microns being more pronounced than in the previous cases.

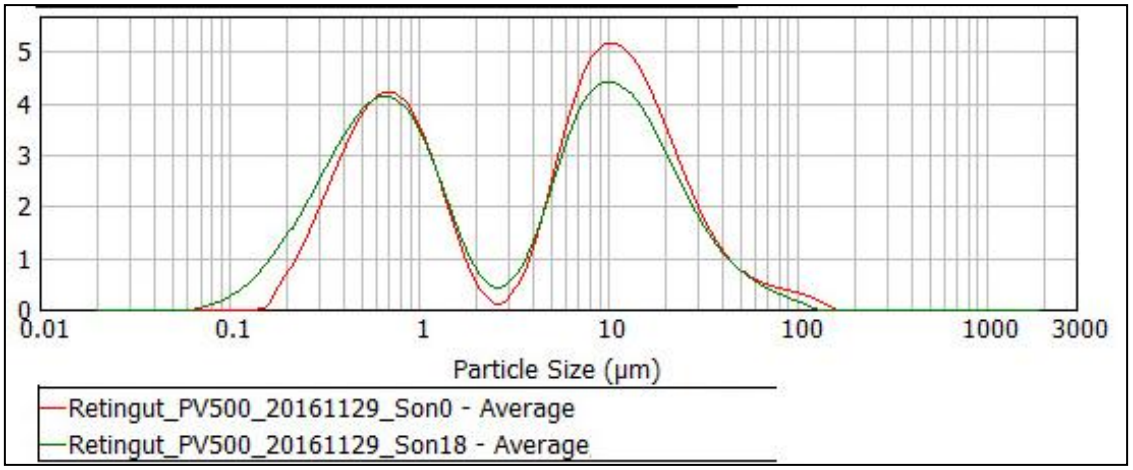



***Figure 3.66:*** *Particle size distribution with and without sonication for membrane 10 sample*

Figure 3.67 shows the decrease in permeability as a function of time which is 1.42 L/h/m$^2$/bar, after cleaning a permeability of 0.68 L/h/m$^2$/bar can be recovered with water, lower than the sample permeability.

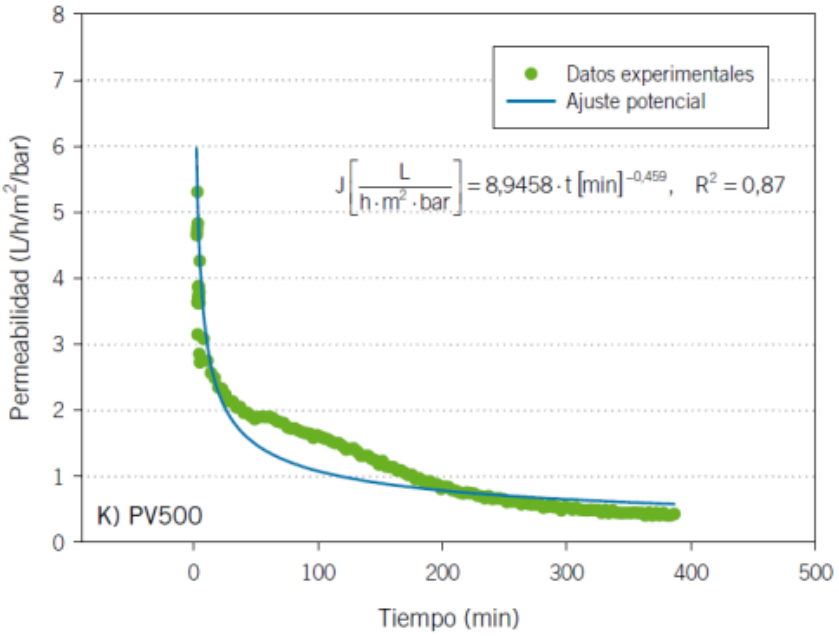



***Figure 3.67:*** *Permeability graph for membrane 10*

Figures 3.68 and 3.69 show the membrane after the test and the retained and permeate samples. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.02.


***Figure 3.68:*** *Photograph of membrane 10 after test*


***Figure 3.69:*** *Retained and permeate samples for membrane 10 test*

Figure 3.70 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

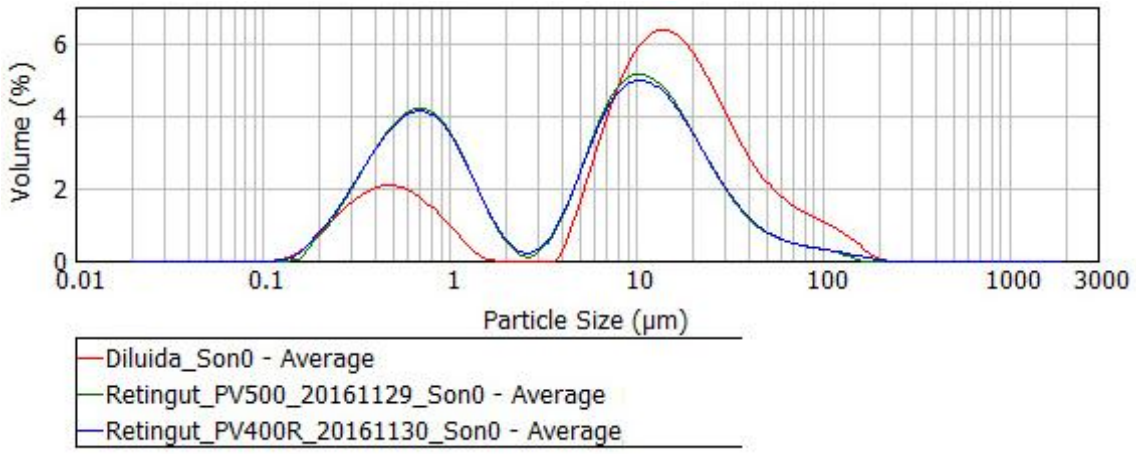



***Figure 3.70:*** *Retained particle size distribution for membrane 10 test*

### 3.3.3.11. Membrane 11: PES MF

Table 3.17 shows the results obtained in membrane 11, which was tested with the centrifuge liquid sample.

***Table 3.17:*** *Results obtained in membrane 11*

| parameter | value |
|---|---|
| **Pore size** | 0.200 µm |
| **Sample** | 4 - 20161107 (LC) |
| **Permeability with water in virgin membrane** | 163 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.23 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 1.77 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,800 mL |
| **Concentration factor** | 1.21 |
| **Initial turbidity** | 11.18 |
| **Detained turbidity** | 12.41 |
| **Permeate turbidity** | 0.02 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.75 |

Figure 3.71 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has peaks at 0.6 and 15 microns.

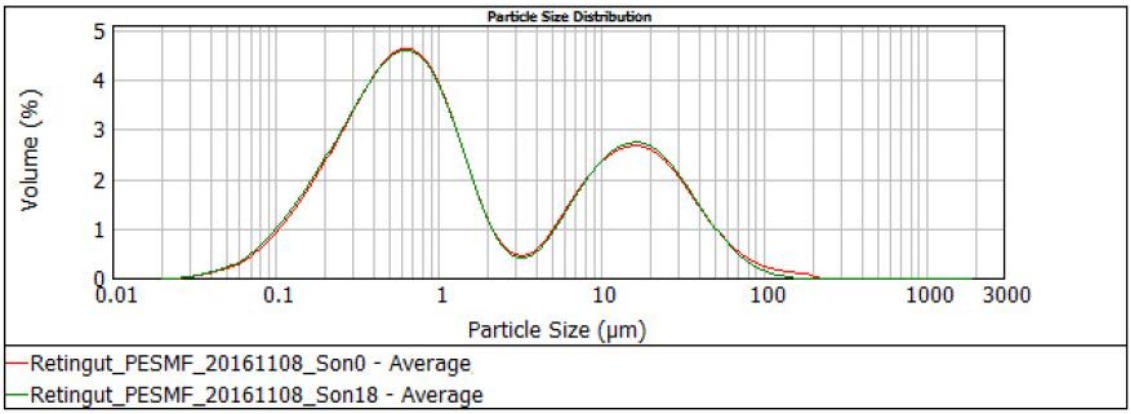



***Figure 3.71:*** *Particle size distribution with and without sonication for membrane 11 sample*

Figure 3.72 shows the decrease in permeability as a function of time which is 1.23 L/h/m$^2$/bar, after cleaning a permeability of 1.77 L/h/m$^2$/bar can be recovered with water.

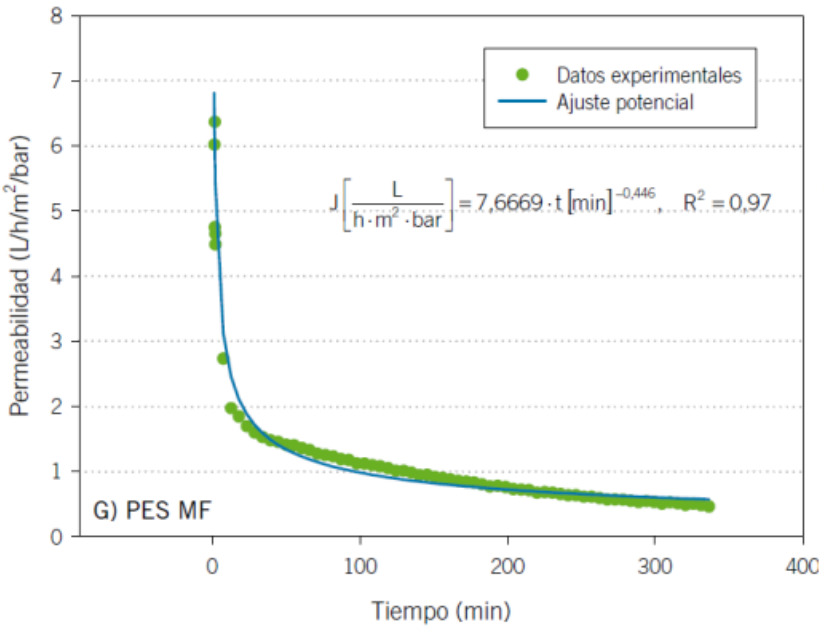



***Figure 3.72:*** *Permeability graph for membrane 11*

Figures 3.73 and 3.74 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.02.

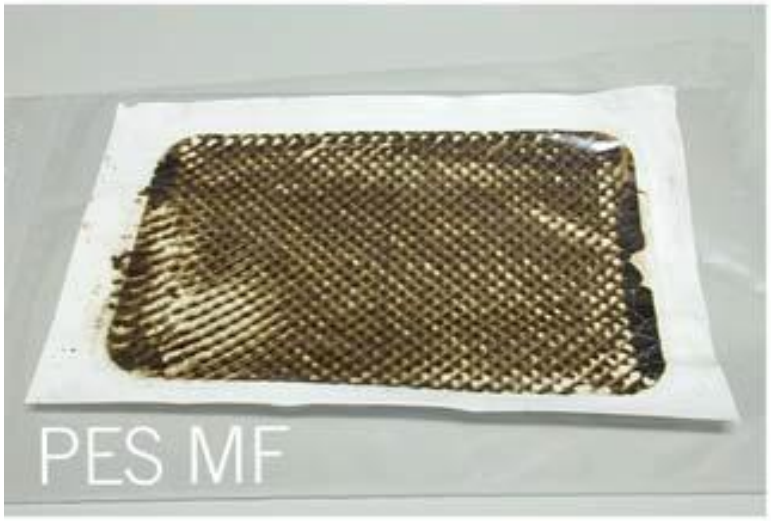



***Figure 3.73:*** *Photograph of membrane 11 after test*


***Figure 3.74:*** *Retained and permeate samples for membrane 11 test*

Figure 3.75 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

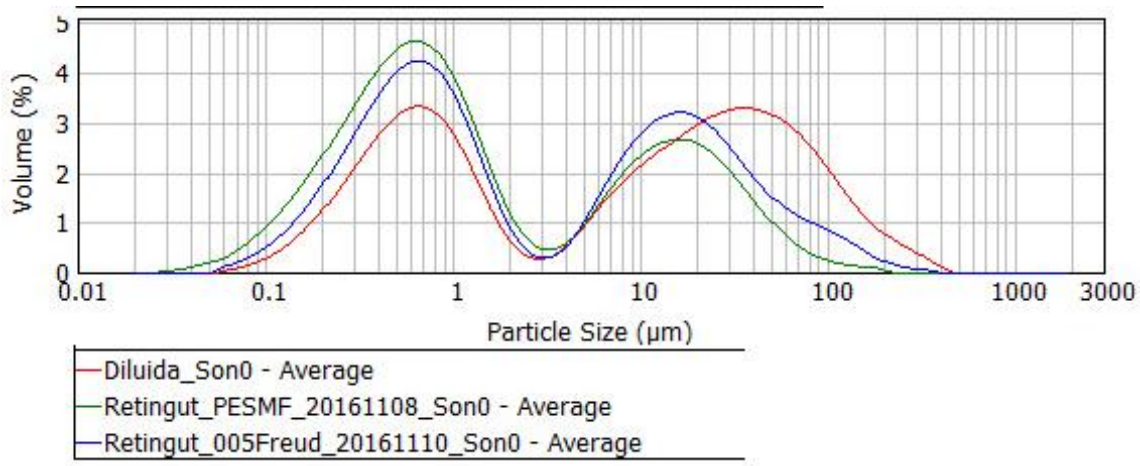



***Figure 3.75:*** *Retained particle size distribution for membrane 11 test*

### 3.3.3.12. Membrane 12: 1.0 on PE

Table 3.18 shows the results obtained for membrane 12, which was tested with the centrifuge liquid sample.

***Table 3.18:*** *Results obtained for membrane 12*

| parameter | value |
|---|---|
| **Pore size** | 1.000 µm |
| **Sample** | 2 - 20161017 (LC) |
| **Permeability with water in virgin membrane** | 2,824 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with sample** | 1.46 L/(h·m$^2$·bar) (after 60 minutes) |
| **Permeability with water in used membrane** | 69.99 L/(h·m$^2$·bar) (after 60 minutes) |
| **Sample volume** | 1,750 mL |
| **Concentration factor** | 1.35 |
| **Initial turbidity** | 11.34 |
| **Detained turbidity** | 14.06 |
| **Permeate turbidity** | 0.03 |
| **Permeate particle size distribution** | Without particles |
| **Detained particle size distribution** | Figure 3.80 |

Figure 3.76 shows the sieved and diluted sample particle size distribution with and without sonication. The sample has peaks at 0.6 and 15 microns.

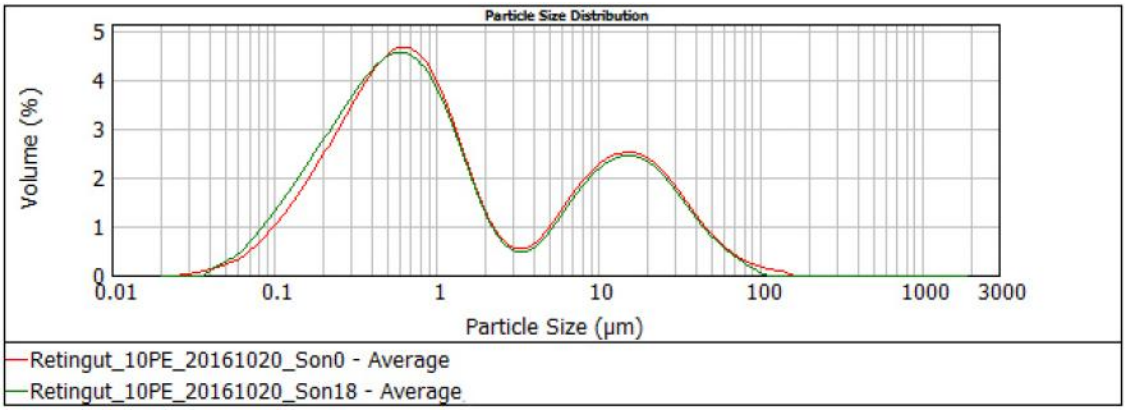



***Figure 3.76:*** *Particle size distribution with and without sonication for membrane 12 sample*

Figure 3.77 shows the decrease in permeability as a function of time which is 1.46 L/h/m$^2$/bar, after cleaning a permeability of 69.99 L/h/m$^2$/bar can be recovered with water.

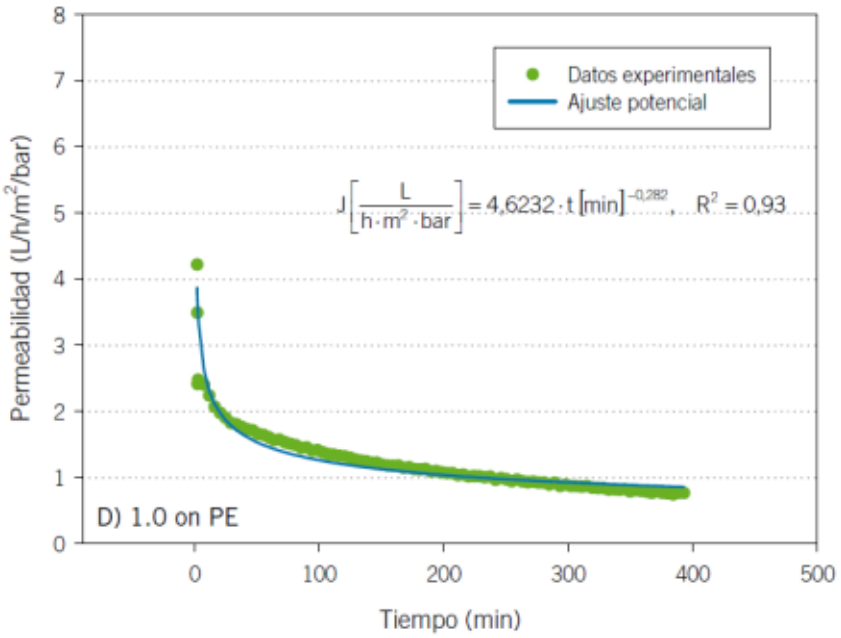



***Figure 3.77:*** *Permeability graph for membrane 12*

Figures 3.78 and 3.79 show the membrane after the test and the retained and permeate samples respectively. As shown in the figure, the permeate is free of suspended solids, with a turbidity of 0.03.


***Figure 3.78:*** *Photograph of membrane 12 after test*

***Figure 3.79:*** *Retained and permeate samples for membrane 12 test*

Figure 3.80 shows the retained particle size distribution. Compared with the initial sample, no significant differences were observed.

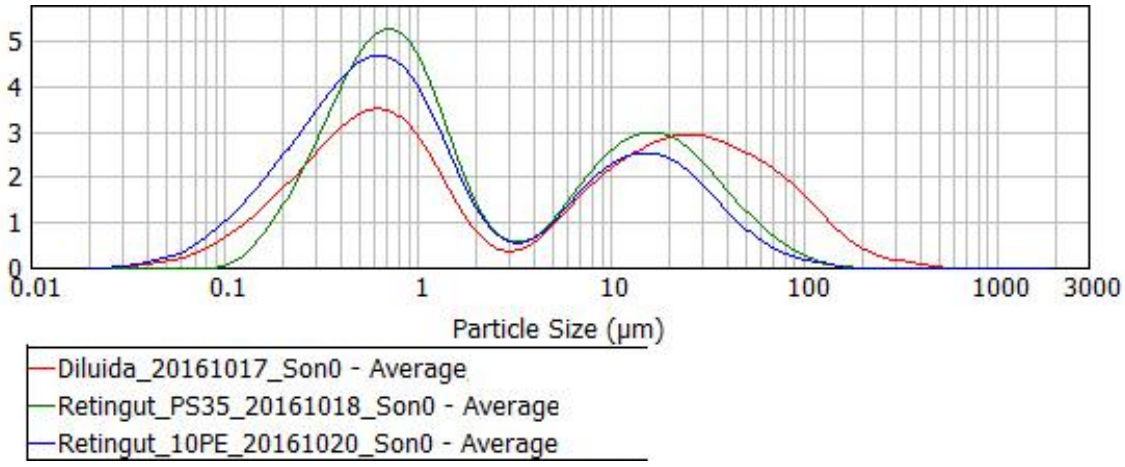



***Figure 3.80:*** *Retained particle size distribution for membrane 12 test*

### 3.3.3.13. Comparison between membranes

Figure 3.81 shows the particle size distribution of the six samples.

We can see four very similar distributions belong to the four centrifuge liquid samples. A distribution appears with two pronounced peaks at 0.15 and 0.7 microns and a smaller peak at 15 microns, which is the double stage centrifuge liquid sample. Finally,

there is a distribution with a pronounced peak at 15 microns and a smaller one at 0.6 microns that belongs to all the PW treated in the WWTP.

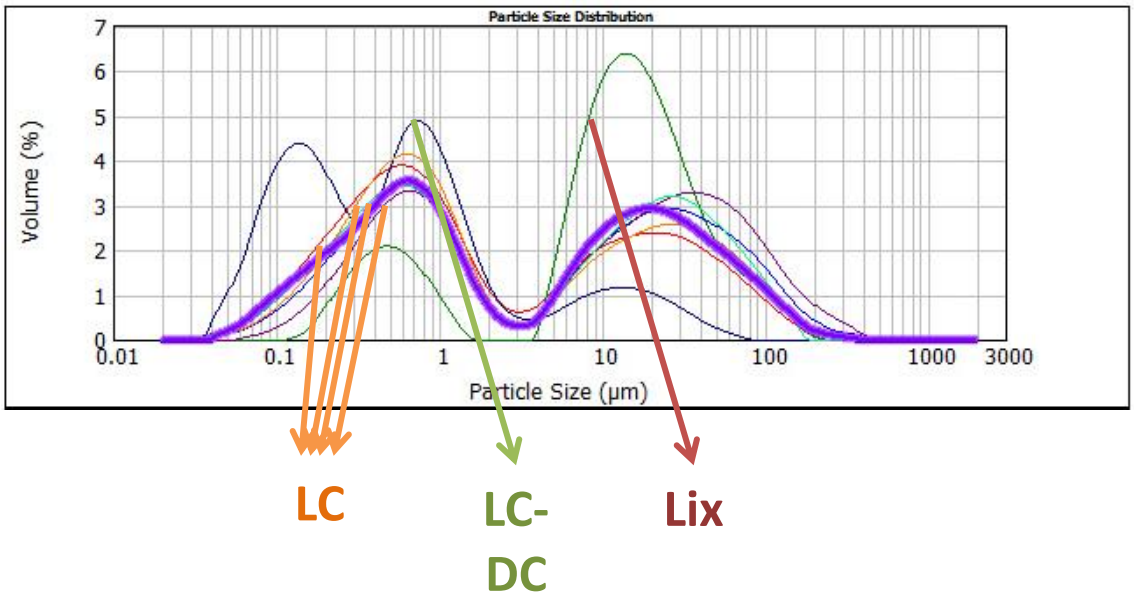



***Figure 3.81:*** *Particle size distribution of the six samples*

Figure 3.82 shows the permeability results obtained from higher to lower permeability.

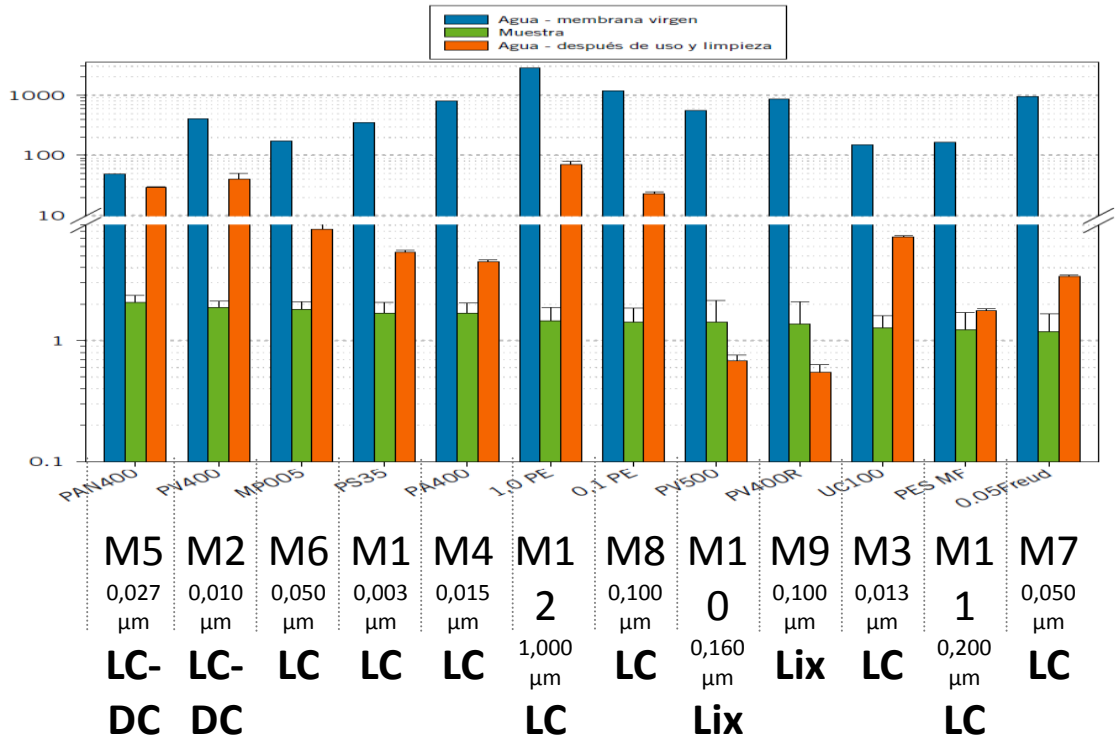



***Figure 3.82:*** *Permeability for all membranes*

We see that the two most favourable results belong to membranes 5 and 2 which were performed with the double stage centrifuge liquid sample. In the case of the membranes that were tested with the PW sample, permeability values after washing lower than the sample permeability were obtained.

Figure 3.83 shows the total and irreversible membrane soiling from minor to major fouling.

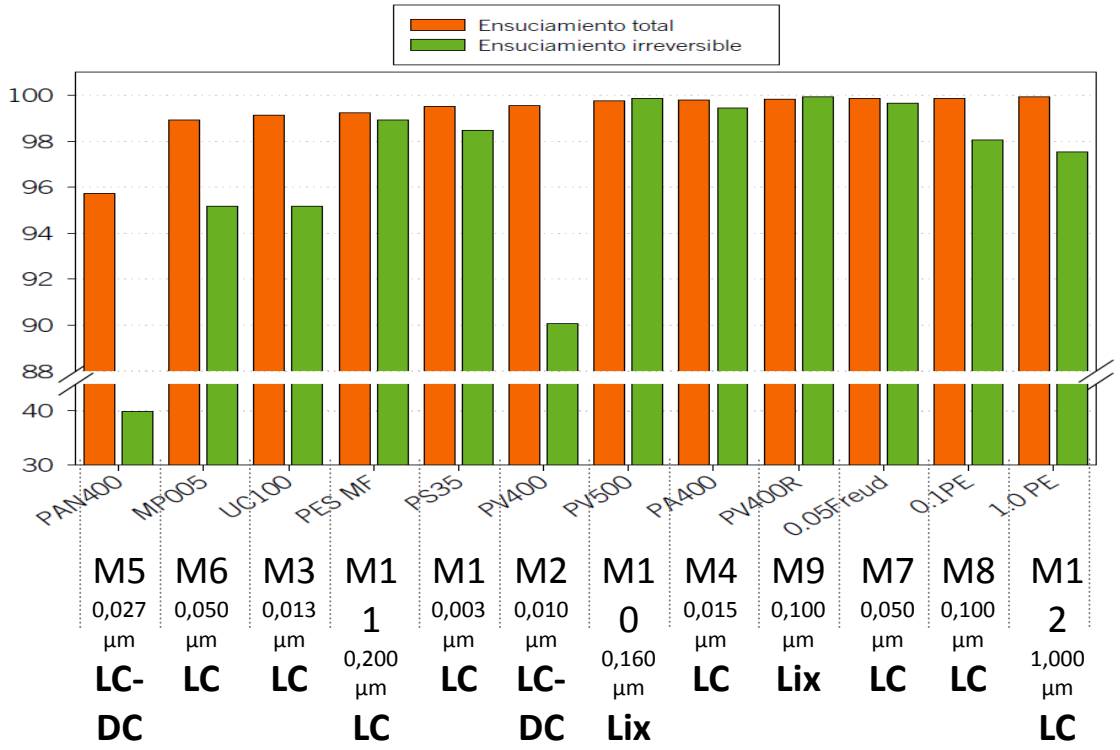



***Figure 3.83:*** *Total and irreversible soiling for all membranes*

It can be observed that the membranes that have been tested with the double stage sample obtain better irreversible soiling results than the other membranes. In the case of the membranes tested with the PW sample they produce an irreversible soiling superior to the total. The best results have been obtained with the membranes with smaller pore size, obtaining higher irreversible soiling results for membranes with a larger pore size.

Finally, we compared the samples, retained and permeate turbidity (figure 3.84).

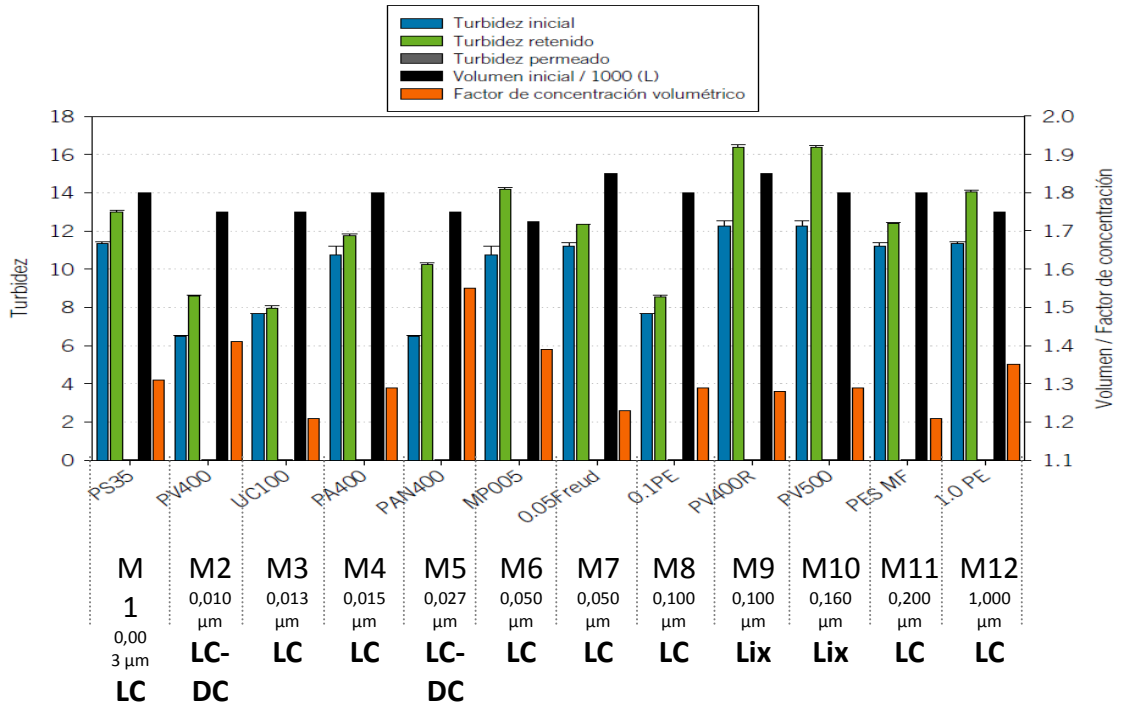



***Figure 3.84:*** *Sample, retained and permeate turbidity*

We observed that for the 12 membranes the permeate turbidity is practically non-existent. All membranes gave a good separation with the worst case giving a turbidity of 0.04.

Although anaerobic digestion PW accounts for between 75 and 97% of the PW to be treated, the other 3 – 25 % contain large amounts of solids which entail greater difficulties for membrane separation. Taking into account the modifications that are being carried out in the PW tanks in the WWTP, the addition of an already existing rotating sieve has been considered as a system for solids separation from the PW of other origins than the anaerobic digestion area (figure 3.85).

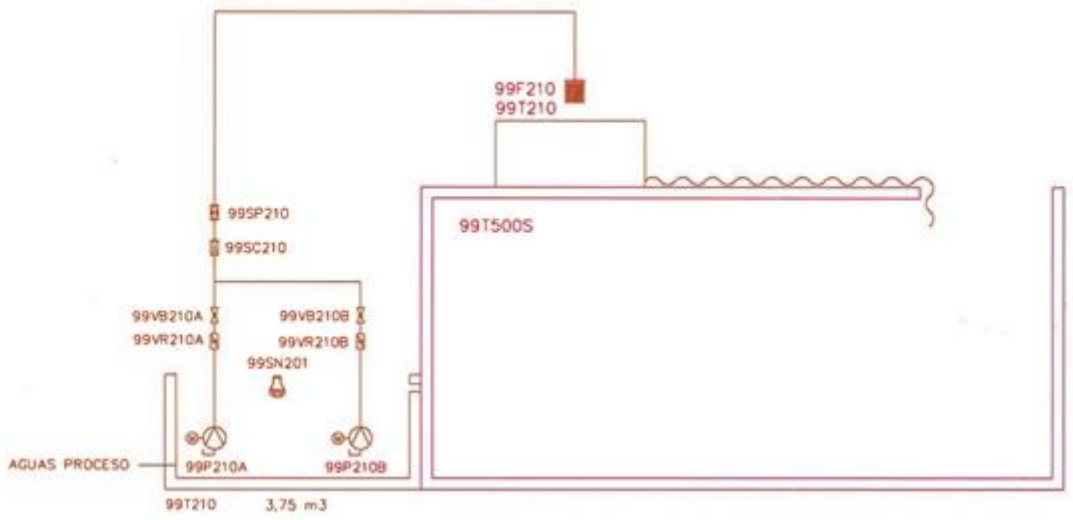



***Figure 3.85:*** *Solids separation from PW with origin other than the anaerobic digestion area*

## 3.4. Conclusions

Throughout this phase the following conclusions have been obtained:

- In the laboratory scale tests, we found that higher centrifuge speeds gave better results , so the speed was set to 9,000 rpm which is the maximum value of the Ecoparc 2 laboratory centrifuge. At this speed, with a duration of 20 centrifugation minutes, reductions of up to TS 26.3%, VS 43.4% and COD 37.5% were obtained. However, the ammonium was not affected by the solid-liquid phase separation by centrifugation. In the laboratory scale tests it was found that the longer the duration of the centrifuge the better were the results, although at longer durations this difference was less noticeable. As a result, it was not necessary to centrifuge for very long periods because the improvement was going to be negligible. In this test at 9,000 rpm, reductions of up to TS 34.2%, VS 56.4% and COD 49.2% were reached, again with no differences for ammonium. With these tests it was possible to determine that significant differences can be obtained in the COD and solids reduction by centrifugation.
  Industrial scale test were then carried out to determine the optimum parameters with the available centrifuges, which do not reach more than 3,000 rpm. Preliminary tests were performed without flocculant addiction, where good results were not observed. After consulting with an expert in industrial centrifuges, we were advised to divide the flocculant amount between the two centrifuges and, at the same time, to reduce the flocculant concentration from 5

g/L to 2.5 g/L as this would give better results. In a second attempt, 13 tests were performed with these conditions obtaining good separation results, of which 5 were also good with respect to the obtained solid and liquid phase appearance. When the working parameters were set for an industrial scale of 2,000 rpm in the second centrifuge with 5 relative speed, 7.5 $m^3$/h sludge flow versus 2,300 L/h of flocculant at 2.5 g/L concentration.

- Since March 24, 2017, the double centrifuge stage has been implemented as standard at Ecoparc 2. Since then real values have been tested, obtaining better results noting improvements in relation to the DM in the liquid phase of both centrifuges with values of up to 6.98% and 1.76% in phase 1 and 2 respectively. At this point, the parameters were 2.5 g/L concentration of Zetag 8185 flocculant, with 8.8 $m^3$/h sludge flow in the first stage with relative speed of 20, and 8.0 $m^3$/h sludge flow in the second stage with relative speed of 5.
  The double centrifugation process has proved to be very positive since the PW to be treated now contains lower COD and ammonium loads and lower TS contents of 25,000 mg $O_2$/L, 3,000 mg N/L and 2% TS compared to the historical values of 35,000 – 50,000 mg $O_2$/L, 4,500 – 6,000 mg N/L and 4% TS. This change implies an improvement in the process that allows increasing performance and efficiency in the WWTP, to decrease the viscosity and to improve the biological degradation by the nitrifying and denitrifying bacteria.

- In parallel, commercial membranes have been tested for solids separation in the PW as an alternative.
  Solids separation by membranes has proved to be a viable option for solids separation in the effluent from dehydration digest, being more effective in the double stage centrifugation processes. In the case of other areas PW, modifications are being made where a rotating sieve will be implemented to separate solids in this effluent.
  It has been found that it is necessary to perform a pretreatment consisting of sieving the sample to remove the larger particles (100 microns) and diluting it to reduce its viscosity. It is possible to dilute with permeate without need to use MW.
  The microfiltration range membranes are sufficient to retain all effluent solids, however, ultrafiltration membranes obtain better results due to less fouling.

# PHASE 4. NEW TECHNOLOGIES: HYDROLYSIS AND STRIPPING

## 4.1. Introduction

The process water between 75 – 97 % entering the WWTP comes from the digested products dehydration from the anaerobic digestion process (centrifuged liquid). This effluent contains an organic loading rate of COD 40,000 mg/L and an ammonium concentration of 5,000 mg/L.

In this phase it is intended to act on this effluent using two technologies for different purposes:

- **Thermal hydrolysis** (figure 4.1). By action of increased temperature the intention is to break the hydrocarbon chains to make the input matter more biodegradable and therefore obtain a carbon source that is more accessible to the wastewater treatment plant biological processes. The steam produced in the cogeneration process of the same facilities will be used as the heat source.

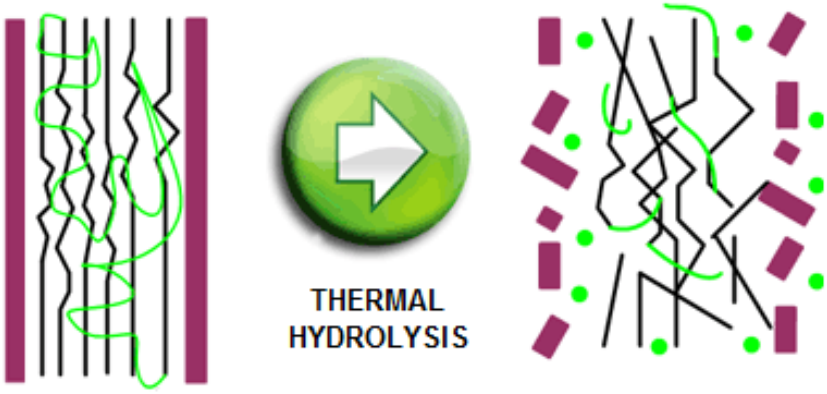



***Figure 4.1:*** *Thermal hydrolysis (source: http://www.hrs-heatexchangers.com)*

- **Stripping** (figure 4.2). By leveraging the hydrolysis temperature, shaking and airing the effluent, it is possible to strip ammonia from the liquid phase to the gas phase.

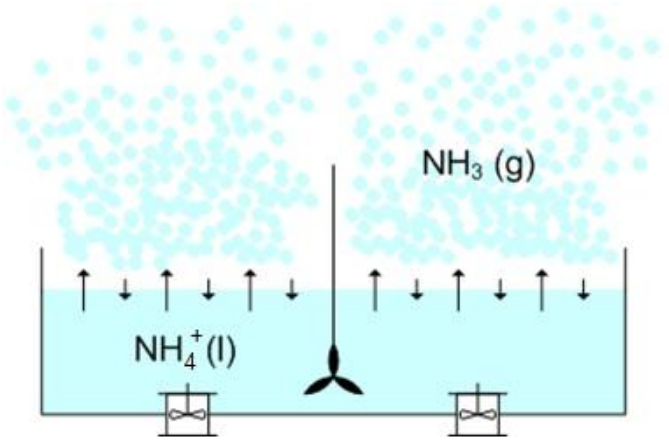



***Figure 4.2:*** *Stripping (image author: Eloísa Albacete)*

## 4.2. Thermal hydrolysis and biogas production

CambiTHP® is a patented thermal hydrolysis process since 1995. CambiTHP® has been carried out in more than 50 projects in different cities in Asia, Europe and America related to waste in order to reduce the amount of disposal, construction costs and operating digesters.

The process described by Cambi consists of the use of steam to increase the sludge temperature and pressure to dissolve the organic matter according to figure 4.3. This process takes place as a pretreatment prior to anaerobic digestion (figure 4.4) so as to increase biodegradability, biogas production and energy efficiency.

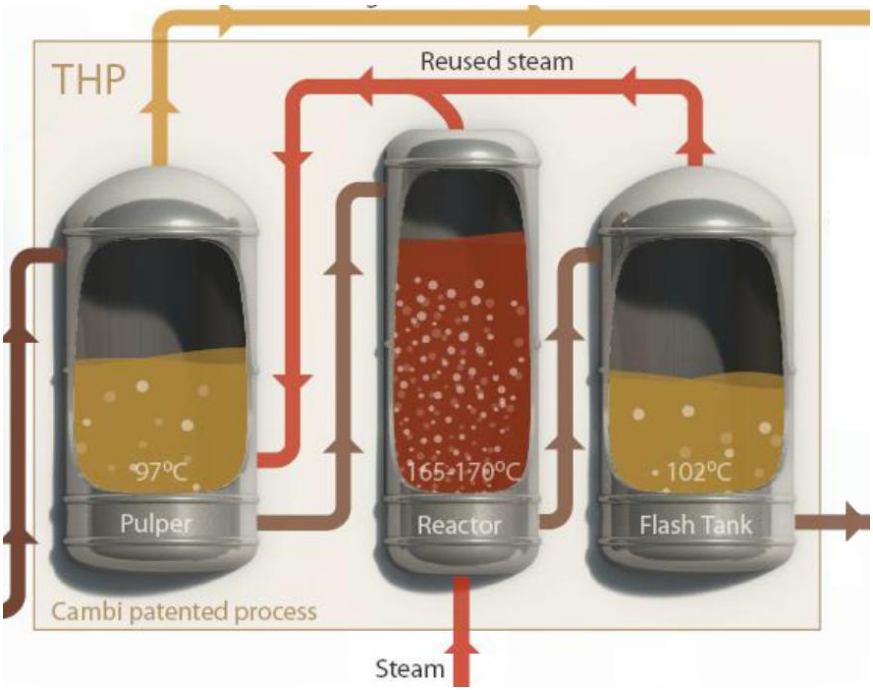



***Figure 4.3:*** *CambiTHP® process (source:)*

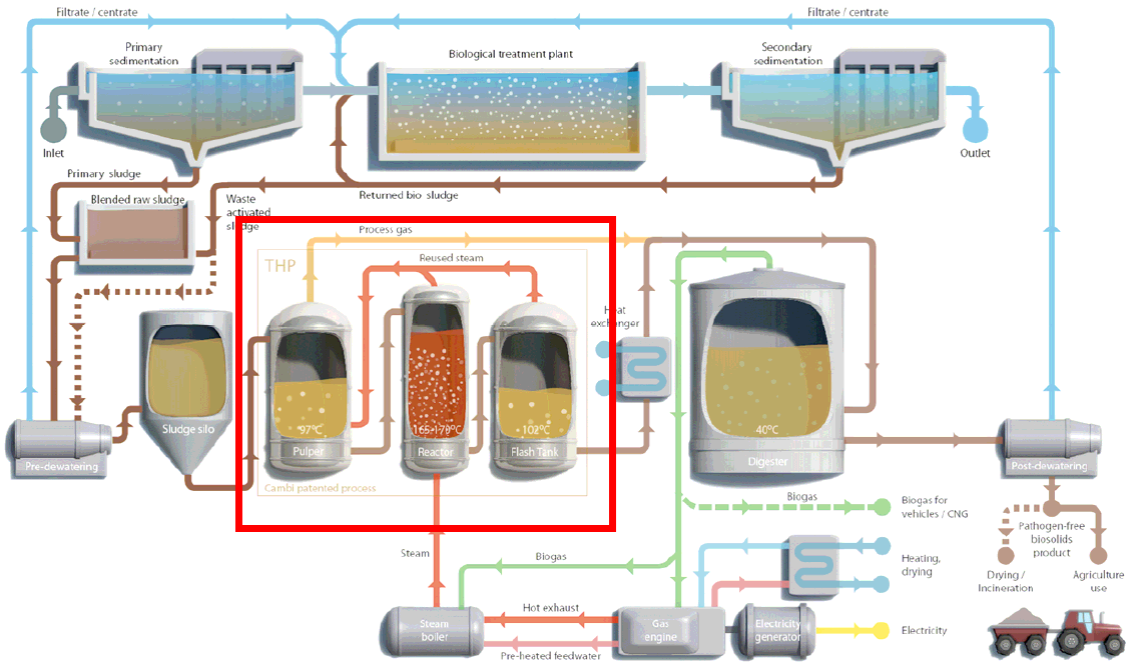



***Figure 4.4:*** *CambiTHP® integration in waste treatment (source: [www.cambi.com](http://www.cambi.com))*

*Sostaqua* is a CENIT project led by *Aguas de Barcelona* (Agbar) that studies the "Technological developments towards a self-sustaining urban water cycle" (*Desarrollos Tecnológicos hacia un ciclo urbano del agua autosostenible*). The study is carried out based on 4 vectors (water, waste, energy, health and environment) through 10 activities. One of these activities focuses on a systematic study of different sludge hydrolysis treatments:

- Enzymatic autohydrolysis (ENZIM-2)
- Thermal hydrolysis (HT-4)
- Thermochemical hydrolysis (HTQ-1)
- Mechanical hydrolysis (MOL-2)
- Ultrasonic treatment (US-H4)

One of the observed factors is the solubilisation, where the results obtained are given in figure 4.5. The conclusion is that the most optimal treatment process to increase the COD biodegradability is thermal hydrolysis.

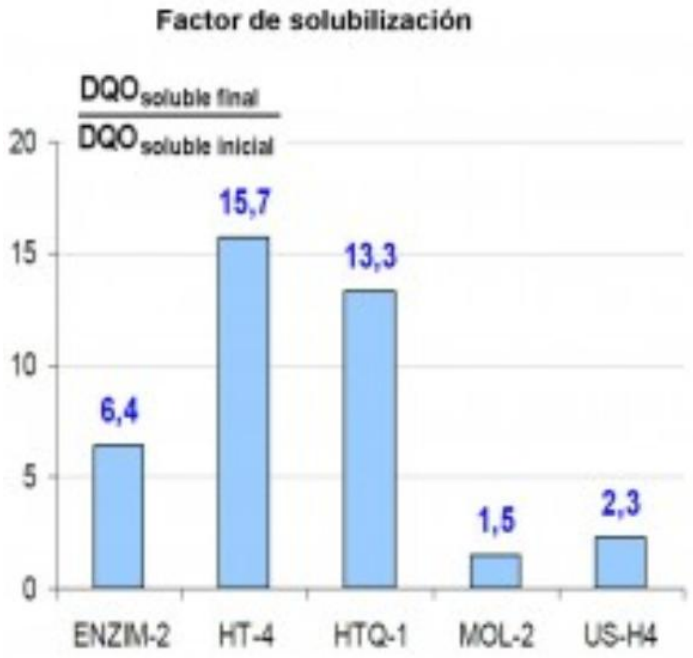



***Figure 4.5:*** *Solubilisation factor for different hydrolysis treatments (source: [www.sostaqua.com](http://www.sostaqua.com))*

*Enhanced excess sludge digestion using thermal and chemical pretreatments* is a study that aims to maximise the hydrolysis stage of anaerobic digestion to improve its process. For this reason, it evaluates the effect of thermal pretreatment on sludge as a function of temperature (30 – 200 ºC), hydrogen peroxide (0-1.0 g $H_2O_2$/g COD) and time.

The study concludes that there is an increase in COD biodegradability during the first treatment hour (THP), however after this first hour the change is not significant. COD solubility (biodegradability) increases with temperature. In the case of the solids, with a temperature of 80 ºC it was already possible to increase four times the TDS and VDS.

### *4.2.1. Laboratory scale tests*

We decided to perform hydrolysis tests to observe how the temperature affects the PW from anaerobic digestion regarding COD biodegradability (solubility). For this purpose, the analysis method given in point E of the section ANALYSIS AND METHODS of this project has been followed.

The effluent to be subjected to hydrolysis is the sludge that is introduced into the centrifuge. From this sludge two phases are obtained: a liquid phase of the anaerobic digestion PW destined to the WWTP, and a solid phase that is currently destined for agricultural application. In order to assign the solid phase to agricultural application, it is

necessary to subject the effluent to a sanitisation process, ensuring that the material has reached 70 ºC for a minimum period of 1 hour. Hydrolysis of the sludge entering the centrifuge would thus meet 2 objectives: to sanitise the solid phase, and increase the liquid phase biodegradability.

Given the results of the study *Enhanced excess sludge digestion using thermal and chemical pretreatments* (page 98 to 104, E.Torrens) and the appearance of a solid phase after subjecting the sludge to elevated temperatures, we decided to establish a maximum temperature of 75 ºC. In addition, we decided to perform 2 more tests at 55 and 65 ºC, and evaluate the evolution of COD, ammonia and solids at time periods of 30 minutes until 2 hours after the desired temperature had been reached.

#### 4.2.1.1. Thermal hydrolysis at 55 ºC

We analysed twelve samples, the soluble and total part of the initial sample before starting the test ($M_{55sol}$ and $M_{55tot}$) and after reaching a temperature of 55º C ($M_{55sol}$ and $M_{55tot}$) and after 30, 60, 90 and 120 minutes ($M_{55sol30}$, $M_{55tot30}$, $M_{55sol60}$, $M_{55tot60}$, $M_{55sol90}$, $M_{55tot90}$, $M_{55sol120}$ and $M_{55tot120}$ respectively).

Figures 4.6 – 4.8 show the values of TS and VS, total and soluble ammonia and COD over time by subjecting the sample to thermal hydrolysis at 55 ºC.

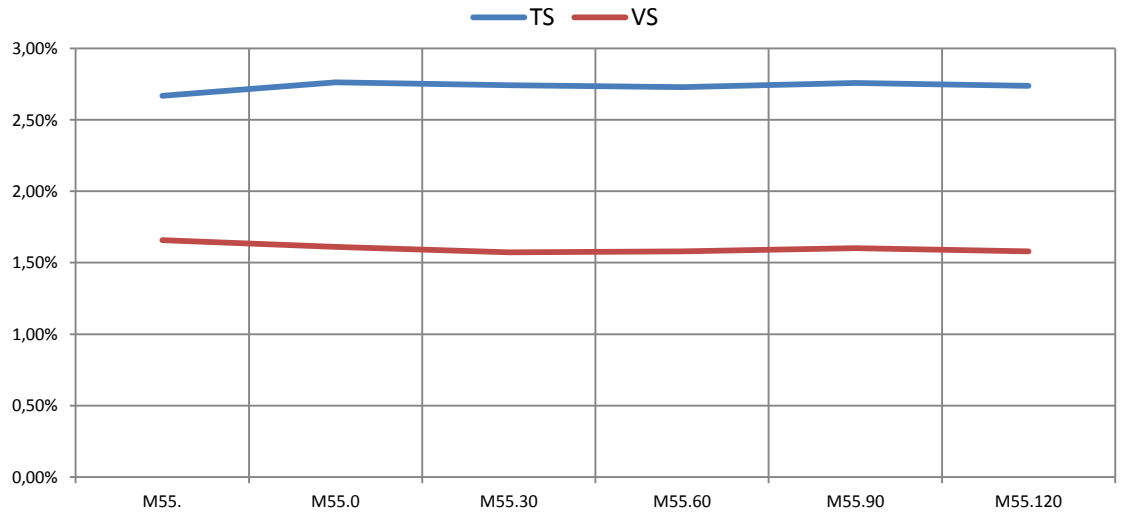



***Figure 4.6:*** *TS and VS values over time by thermal hydrolysis at 55 ºC*

The TS values vary between 2.67 and 2.76 % and in the VS case between 1.57 and 1.66 %. Both total and volatile solids show no significant variations over time after subjecting them to thermal hydrolysis at 55 ºC for 120 minutes.

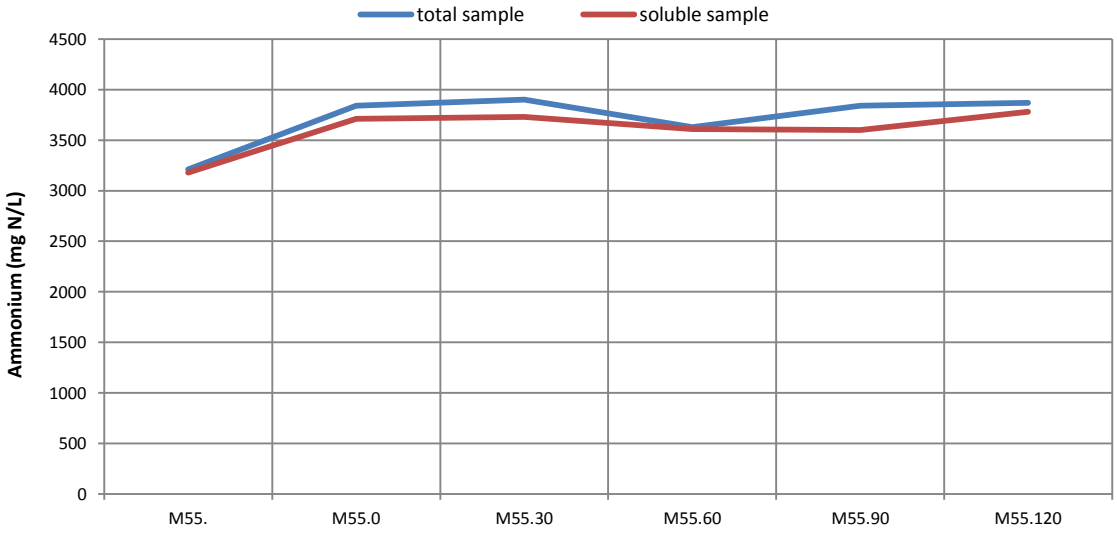



***Figure 4.7:*** *Ammonium values over time by thermal hydrolysis at 55 ºC*

In the case of ammonium, there is no relation between the concentration variations and time after subjecting the sample to 55 ºC during 120 minutes. The total sample values vary between 3,200 and 3,900 mg N/L and the soluble sample is between 93.8 and 99.4 % of the total. Therefore, the temperature also does not affect the ammonium solubility.

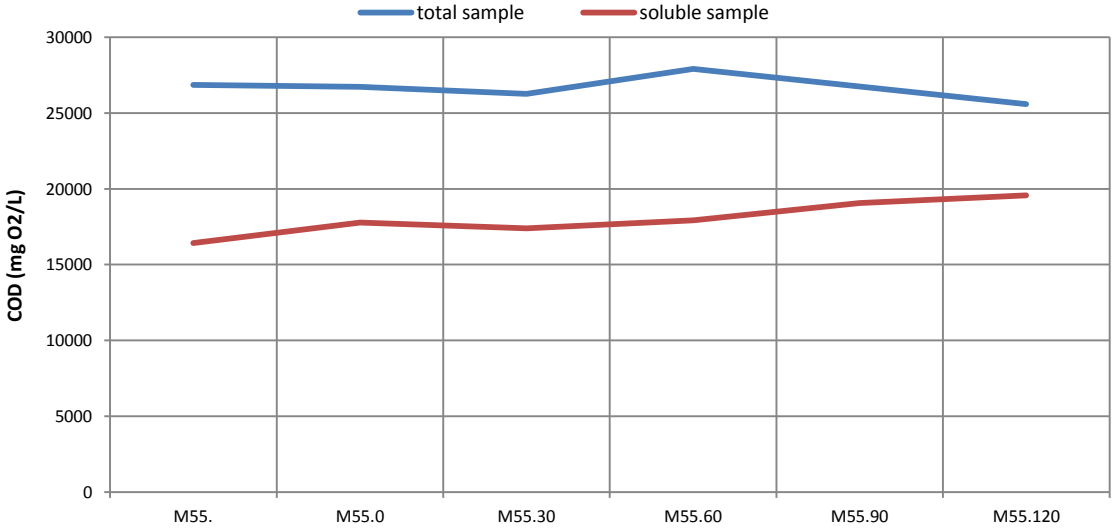



***Figure 4.8:*** *COD values over time by thermal hydrolysis at 55 ºC*

In the COD case, the total sample values remain stable between 25,600 and 28,000 mg $O_2$/L, however, it can be seen that the soluble sample increases its value as time passes, with soluble a COD 61.2 % in the initial sample until reaching 76.5 % after 120 minutes. Hence we observe that the temperature does affect the COD solubility. Figure 4.9 shows the solubilisation factor (soluble final COD/soluble initial COD) of five samples subjected to a temperature of 55 ºC at 0, 30, 60, 90 and 120 minutes respectively.

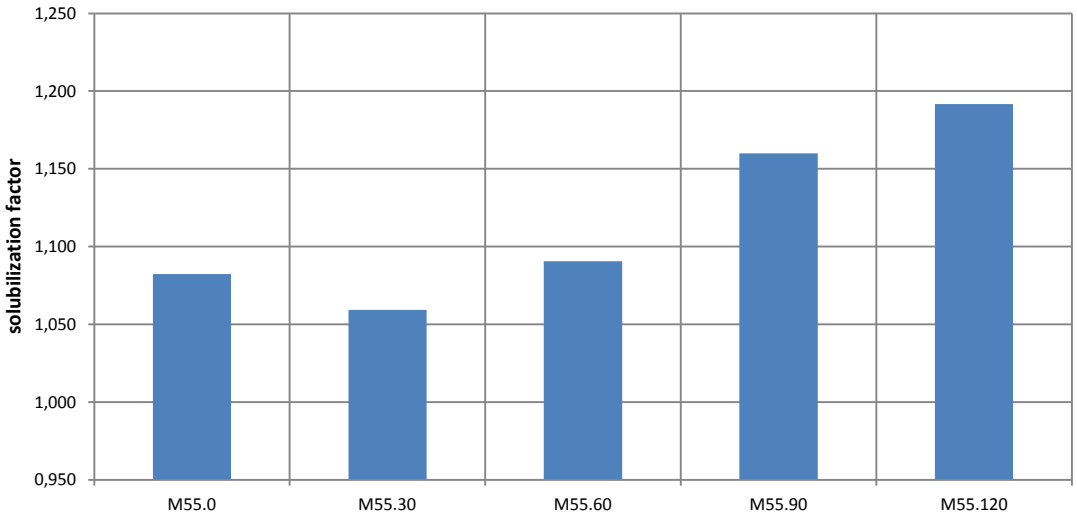



***Figure 4.9:*** *COD solubilisation factor over time by thermal hydrolysis at 55 ºC*

With the exception of the sample at 0 minutes, an increase in the COD solubilisation factor is observed as time passes, reaching a value of 1.19 after 120 minutes. This means that the effluent is 1.19 times more soluble after being subjected to a temperature of 55 ºC during 120 minutes.

#### 4.2.1.2. Thermal hydrolysis at 65 ºC

We analysed twelve samples, the soluble and total part of the initial sample before starting the test ($M_{65sol}$ and $M_{65tot}$) and after reaching a temperature of 65º C ($M_{65sol}$ and $M_{65tot}$) and after 30, 60, 90 and 120 minutes ($M_{65sol30}$, $M_{65tot30}$, $M_{65sol60}$, $M_{65tot60}$, $M_{65sol90}$, $M_{65tot90}$, $M_{65sol120}$ and $M_{65tot120}$ respectively).

Figures 4.10 – 4.12 show the values of TS and VS, total and soluble ammonia and COD over time by subjecting the sample to thermal hydrolysis at 65 ºC.

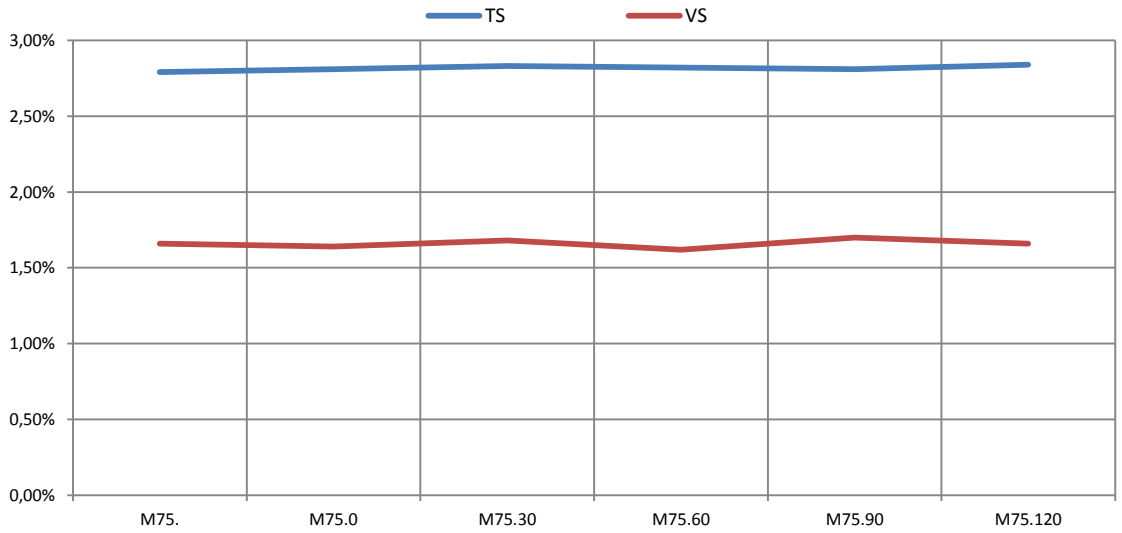



***Figure 4.10:*** *TS and VS values over time by thermal hydrolysis at 65 ºC*

The TS values vary between 2.79 and 2.84 % and in the VS case between 1.62 and 1.70 %. Both total and volatile solids do not show significant variations over time after subjecting the samples to thermal hydrolysis at 65 ºC for 120 minutes.

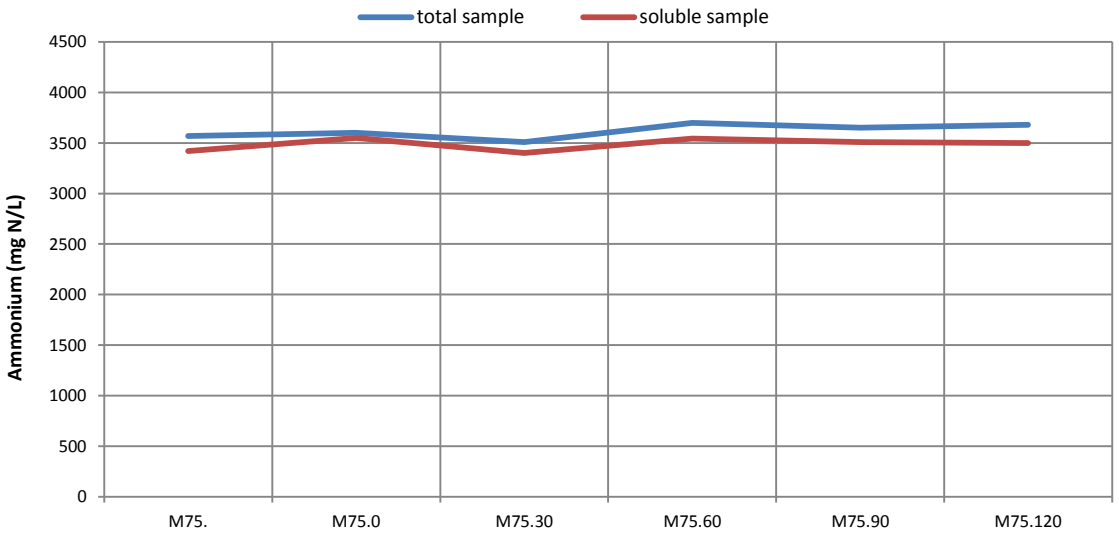



***Figure 4.11:*** *Ammonium values over time by thermal hydrolysis at 65 ºC*

In the case of ammonium, there is no relation between the concentration variations and time after subjecting the sample to 65 ºC during 120 minutes. The total sample values vary between 3,500 and 3,700 mg N/L and the soluble sample is between 95.1 and

98.6 % of the total. Therefore, the temperature also does not affect the ammonium solubility.

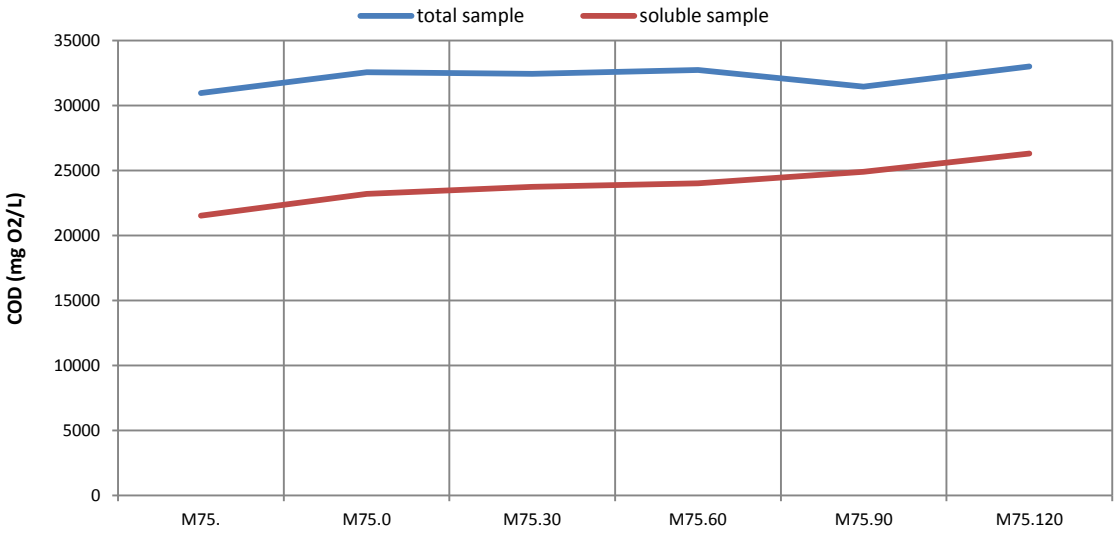



***Figure 4.12:*** *COD values over time by thermal hydrolysis at 65 ºC*

In the COD case, the total sample values remain stable between 30,900 and 33,000 mg $O_2$/L, however, it can be seen that the soluble sample increases its value as time passes, with soluble COD 69.5 % in the initial sample until reaching 79.7 % after 120 minutes. Hence we observe that the temperature does affect the COD solubility. Figure 4.13 shows the solubilisation factor (soluble final COD/soluble initial COD) of five samples subjected to a temperature of 65 ºC at 0, 30, 60, 90 and 120 minutes respectively.

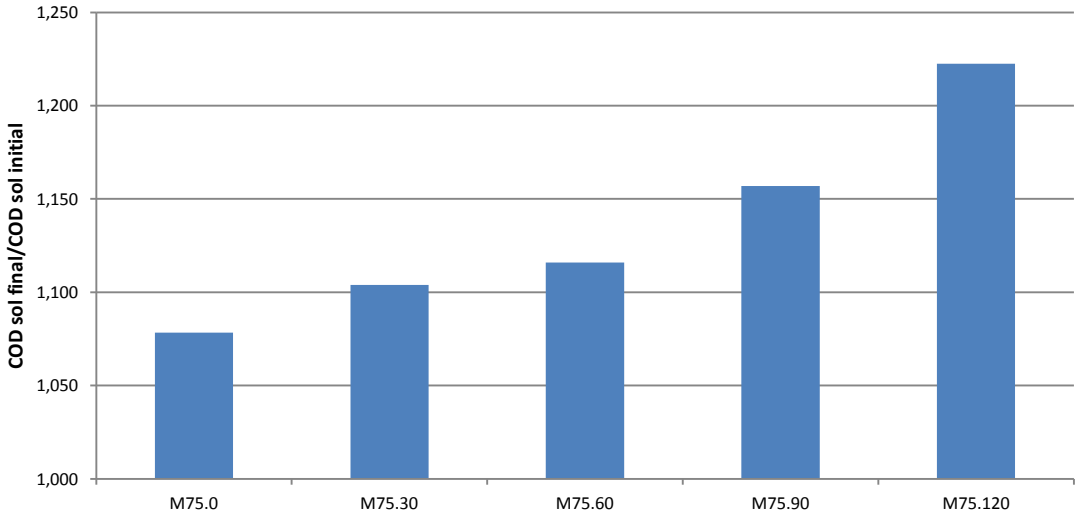



***Figure 4.13:*** *COD solubilisation factor over time by thermal hydrolysis at 65 ºC*

An increase in COD solubilisation factor is observed as time passes, reaching a value of 1.22 after 120 minutes. This means that the effluent is 1.22 times more soluble after being subjected to a temperature of 65 ºC during 120 minutes.

#### 4.2.1.3. Thermal hydrolysis at 75 ºC

We analysed twelve samples, the soluble and total part of the initial sample before starting the test ($M_{75sol}$ and $M_{75tot}$) and after reaching a temperature of 75º C ($M_{75sol}$ and $M_{75tot}$) and after 30, 60, 90 and 120 minutes ($M_{75sol30}$, $M_{75tot30}$, $M_{75sol60}$, $M_{75tot60}$, $M_{75sol90}$, $M_{75tot90}$, $M_{75sol120}$ and $M_{75tot120}$ respectively).

Figures 4.14 – 4.16 show the values of TS and VS, total and soluble ammonia and COD over time by subjecting the sample to thermal hydrolysis at 75 ºC.

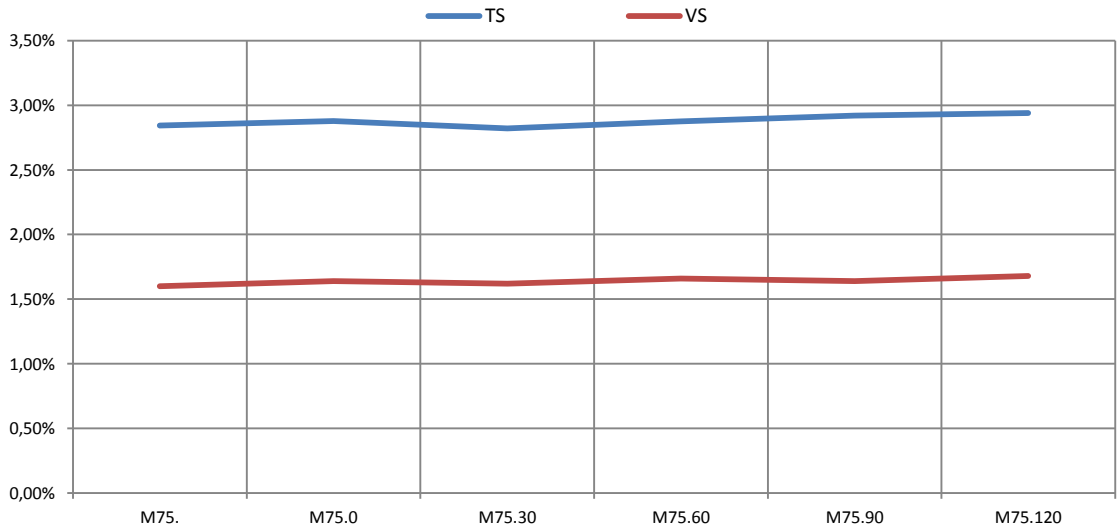



***Figure 4.14:*** *TS and VS values over time by thermal hydrolysis at 75 ºC*

The TS values vary between 2.82 and 2.94 % and in the VS case between 1.60 and 1.68 %. Both total and volatile solids do not show significant variations over time after subjecting the samples to thermal hydrolysis at 75 ºC for 120 minutes.

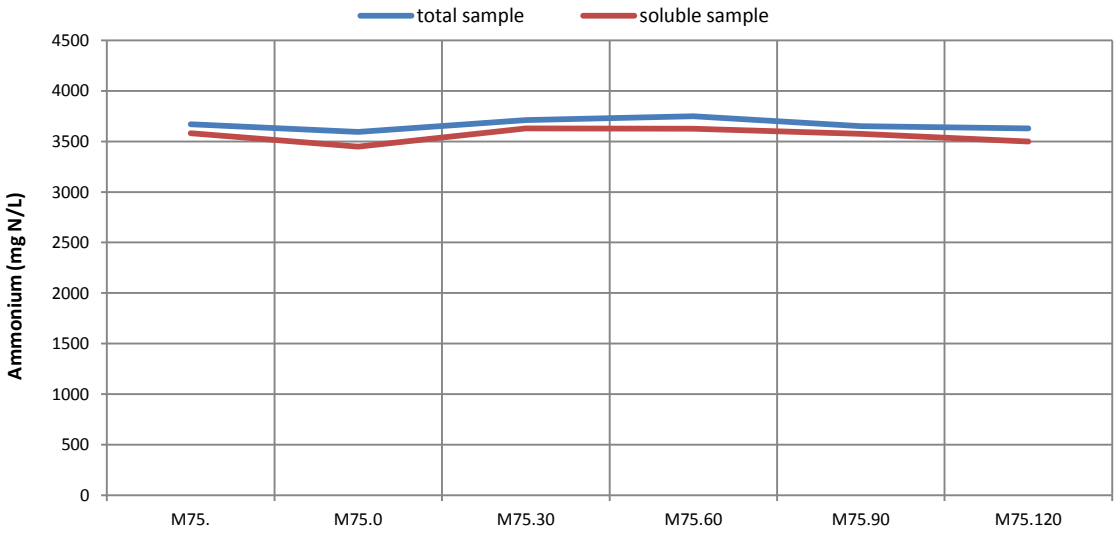



***Figure 4.15:*** *Ammonium values over time by thermal hydrolysis at 75 ºC*

In the case of ammonium, there is no relation between the concentration variations and time after subjecting the sample to 75 ºC during 120 minutes. The total sample values vary between 3,600 and 3,800 mg N/L and the soluble sample is between 96.0 and 97.9 % of total. Therefore, the temperature also does not affect the ammonium solubility.

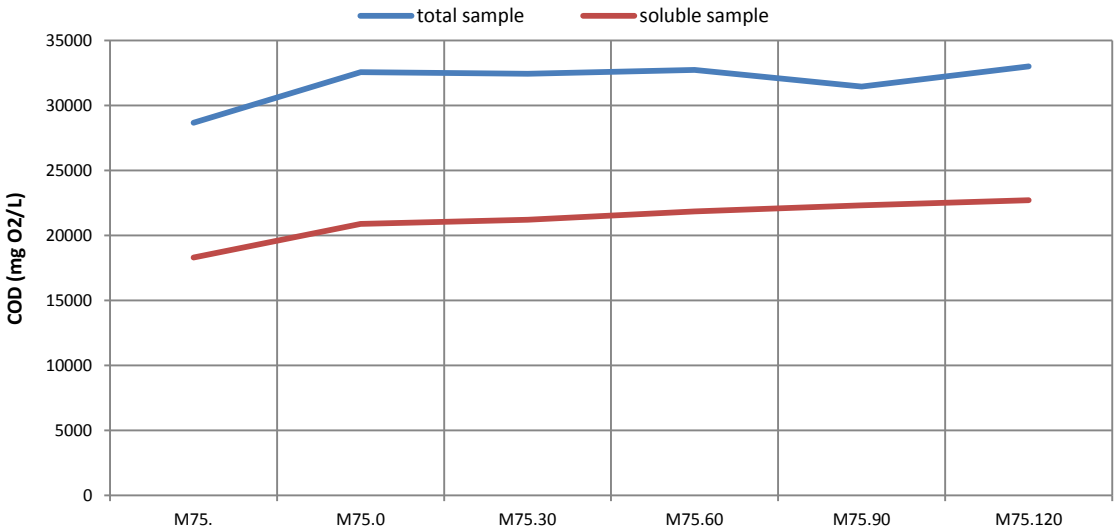



***Figure 4.16:*** *COD values over time by thermal hydrolysis at 75 ºC*

In the COD case, the total sample values remain stable between 28,600 and 33,000 mg $O_2$/L, however, it can be seen that the soluble sample increases its value as time passes, with soluble COD at 63.8 % in the initial sample until reaching 68.8 % after 120 minutes. Hence we observe that temperature does affect the COD solubility. Figure

4.17 shows the solubilisation factor (soluble final COD/soluble initial COD) of five samples subjected to a temperature of 75 ºC at 0, 30, 60, 90 and 120 minutes respectively.

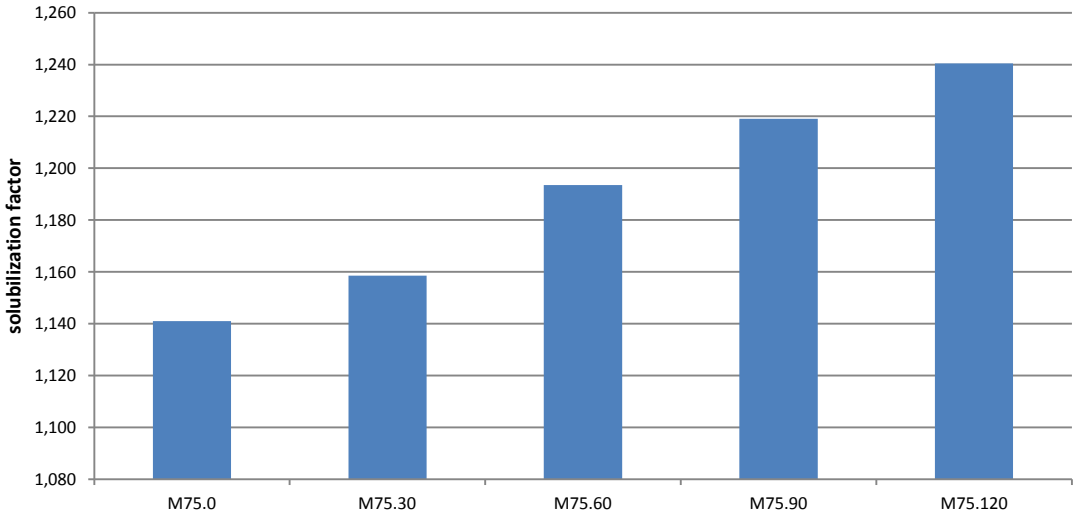



***Figure 4.17:*** *COD solubilisation factor over time by thermal hydrolysis at 75 ºC*

An increase in COD solubilisation factor is observed as time passes, reaching a value of 1.24 after 120 minutes. This means that the effluent is 1.24 times more soluble after being subjected to a temperature of 65 ºC during 120 minutes.

### 4.2.1.4. Thermal hydrolysis at 55, 65 and 75 ºC comparative

Table 4.1 shows the COD solubilization factor (SF) results at different temperatures in the same time period.

***Table 4.1:*** *Solubilisation factor at different temperatures and time periods*

| | Thermal Hydrolysis at 55 ºC | | Thermal Hydrolysis at 65 ºC | | Thermal Hydrolysis at 75 ºC | |
|---|---|---|---|---|---|---|
| Time (min) | COD (mg $O_2$/L) | SF | COD (mg $O_2$/L) | SF | COD (mg $O_2$/L) | SF |
| **Initial** | 16,428 | | 21,515 | | 18,300 | |
| **0** | 17,780 | 1.082 | 23,200 | 1.078 | 20,880 | 1.141 |
| **30** | 17,400 | 1.059 | 23,750 | 1.104 | 21,200 | 1.158 |
| **60** | 17,916 | 1.091 | 24,010 | 1.116 | 21,840 | 1.193 |
| **90** | 19,056 | 1.160 | 24,890 | 1.157 | 22,310 | 1.219 |
| **120** | 19,576 | 1.192 | 26,300 | 1.222 | 22,700 | 1.240 |

The difference in concentration between the different temperatures is due to the initial sample, since each of the tests were performed on different days, so despite picking the sample at the same point, their values differ from day to day. On the other hand, the SF should not be affected by this since it is calculated from the ratio between the value at a particular time with the initial one.

Regarding the SF obtained at the different temperatures, with the exception of the SF at 0 minutes at 65 ºC, we observe that the higher the temperature the higher the SF. Furthermore, the SF was found to increase over time.

Comparing the maximum SF of1.24 obtained from the sample at 75 ºC after 120 minutes with the values in figure 4.5 shows that it does not represent a very high value. However, in reality this values represents a 24 % increase in COD solubility which is avery significant improvement of the purification process. Moreover, since it is necessary to apply an increased temperature to the effluent for the sanitisation of solid phase, it does not entail an additional cost and facilitates the treatment in the WWTP by biological degradation.

## 4.3. Stripping

According to the study *Autotrophic nitrogen removal in granular sequencing batch reactors* (J.R.Vázquez) for concentrated wastewater with ammonium concentrations higher than 5,000 mg N/L physicochemical methods are technically and economically feasible. The main physicochemical processes applied for ammonium removal are air stripping, breakpoint chlorination and selective ion exchange. The last two options have the inconvenience of involving technologies with high operational costs and complex control. Contrarily, ammonia stripping is widely used because of its simple operation and high efficiency.

As shown in eq. 4.2, 2 moles of alkalinity are removed due to $CO_2$ stripping per mole of ammonium oxidised due to the release of protons during the ammonium oxidation (eq. 4.1). In the case when this amount of buffer is not available in the water, the pH of the medium drops and the ammonium oxidation rate decreases sharply.

$$NH_4^+ + 1.5\ O_2 + 2\ HCO_3^- \rightarrow NO_2^- + 3\ H_2O + 2\ CO_2 \qquad \text{(eq. 4.1)}$$

$$NH_4^+ + 1.382\ O_2 + 1.982\ HCO_3^- \rightarrow 0.018\ C_5H_7O_2N + 0.982\ NO_2^- + 2.927\ H_2O + 1.891\ CO_2 \qquad \text{(eq. 4.2)}$$

The oxidation of high ammonium concentrations causes a significant pH-decrease, which limits further ammonium conversion as already mentioned. Sludge reject water

typically contains equimolar amounts of bicarbonate and ammonium, so half of the produced protons are neutralised by CO2-stripping. As a result, for streams containing bicarbonate and ammonium in equimolar amounts and without additional pH control in the reactor, typically half of the ammonium is converted before a significant pH-decrease occurs, which prevents further ammonium oxidation

In the study *Coagulation-flocculation and air stripping as a pretreatment of young landfill PW* (T.Yilmaz) the optimal stripping flow and time for ammonium removal were determined: in ammonium removal with air stripping, the optimum flow rate was 1 L/min and the optimum aeration time was 8 hours.

In the study *Air stripping of ammonia from pig slurry: characterisation and feasibility as a pre- or post-treatment to mesophilic anaerobic digestion* (A.Bonmatí) the stripping effect on slurry at 80 ºC was checked.Here the ammonium stripping was performed fora desorption by entrainment with air with the ammonium being transferred from the liquid stream to the gas. It is combined with a second operation where the ammonia is absorbed from air to an aqueous acid medium. Both transfers depend on the relationship between air and liquid flow rates as well as the ammonium concentration. The equilibrium between ammonium and ammonia species is controlled by pH and temperature, obtaining recovery efficiencies higher than 90 % with a basic pH (8-10) and at temperatures above 50 ºC (figure 4.18).

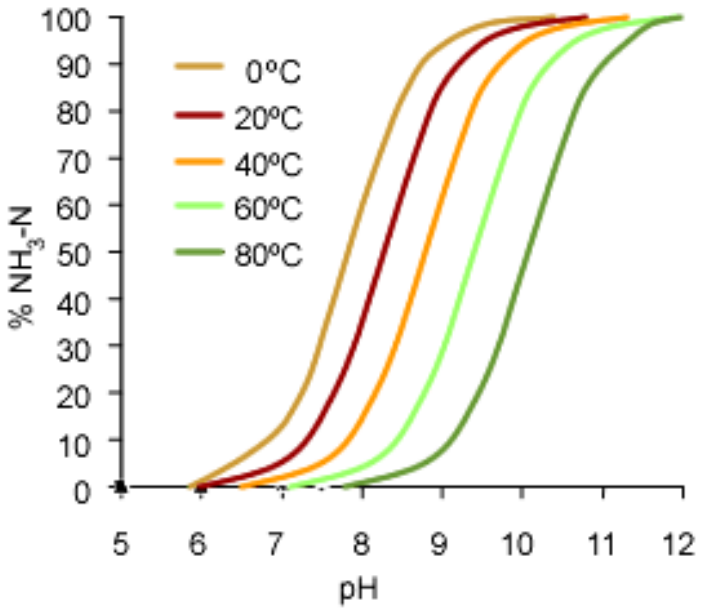



***Figure 4.18:*** *Ammonia (%) as a function of pH at different temperatures (source: A. Bonmatí)*

Since the effluent to be stripped comes from the thermal hydrolysis at a temperature of 75 ºC, the effluent to be treated will have a temperature of 70 – 75 ºC, so that, according to figure 4.18, without adjusting the pH, the ammonia-ammonium balance would be less than 5% ammonia (gas phase). Therefore, to obtain a good ammonia separation it is necessary to add a base which increases the pH. If a minimum 90 % ammonia/ammonium separation is required, it would be necessary to reach a pH > 10.5. For a minimum 50% ammonia/ammonium separation, a pH > 9.5 is necessary. In any case, the pH needs to be acted on to achieve a good performance.

### *4.3.1. Laboratory and pilot plant scale tests*

In May 2014 a poster of this project was presented with the main objectives and the phases in which the study had been divided (annex III). Initially we planned to divide the study into the following phases:

- Phase 1. Wastewater caracterisation
- Phase 2. Wastewater treatment plant optimisation
- Phase 3. New technologies: hydrolysis and stripping
- Phase 4. New biological processes

However, as the study progressed, the major problem caused by the solids contained in the PW was observed and this new phase was added.

FCC Aqualia has approved a LIFE project called METHAMORPHOSIS (annex IV). In November 2015, during a visit from Aqualia to Ecoparc 2, it was brought to the attention of this study that a great deal of information can be provided to the LIFE project, based on the poster published in 2014. Given that the LIFE project deals directly with the centrifuge liquid phase, special attention was paid to the application of new technologies (thermal hydrolysis and stripping) and a preliminary study was carried out in which they obtained positive results. As a result they added the incorporation of a stripping pilot plant to their project.

By this time, several companies specialised in stripping had already been contacted with respect to performing the tests at laboratory and pilot plant scales . However, since the LIFE project was programmed to be carried out at Ecoparc 2 with its own leach, in which a stripping pilot plant was to be incorporated, we decided to focus this study on the solids separation by centrifugation and membranes, leaving the stripping phase for the LIFE project.

**LIFE Project stripping test:**

In a previous study by Aqualia, a test was carried out at 65 ºC without adding any reagent to regulate the pH. Almost half of the ammonium had been reduced after 3 hours, from an initial 4,500 mg N/L to less than 2,500 mg N/L. In a second test also at 65 ºC temperature it was observed that by decreasing over time the alkalinity and increasing the pH to values of 9.5, as can be seen in figure 4.18, an ammonium reduction of 50 % was achieved.

## 4.4. Conclusions

Throughout this phase the following conclusions have been obtained:

- The anaerobic digestion PW accounts for between 75 and 97 % of the total PW to be treated in the WWTP. As a result the tests were carried out on the centrifuge liquid phase (anaerobic digestion PW) in order to increase the COD biodegradability, to facilitate the WWTP biological degradation process by thermal hydrolysis.

  The centrifuge solid phase is intended for agricultural application, which requires prior sanitisation at for 1 hour. In a previous test for this purpose, it had been observed that at temperatures higher than 70 ºC the solid phase appearance is more oily and makes agricultural application difficult, so that the maximum viable temperature is 75 ºC. For this reason, 3 thermal hydrolysis tests were carried out at 55, 65 and 75 ºC.

  In the results obtained no significant differences in ammonium concentration and solids contents were observed. However, a relationship with temperature and exposure time was observed for COD solubility.

- To calculate the COD biodegradability, the solubility factor (SF) corresponding to the ratio of soluble COD at a specific point to the initial soluble COD was used.

  The SF increases as the temperature and the exposure time increase. The maximum SF value that was obtained is 1.24 for a sample at 75 ºC during 120 minutes. This SF represents a 24 % increase in COD solubility. Since the centrifuge solid phase limits the temperature to 75 ºC, the optimum temperature for the thermal hydrolysis of effluent is 75 ºC. Moreover, it is necessary to apply

this heating for the solid phase sanitisation. If applied before centrifugation, the process is thus also valid for solid phase agricultural application and does not need an extra energy consumption for the thermal hydrolysis of the liquid phase.

- Stripping is a technology from which it is possible to reduce the ammonia concentration in the effluent by transferring ammonia to the gas phase from the aqueous phase. To obtain a good stripping performance, it is necessary to act on the temperature and pH, with the resulting economic cost.

  According to A.Bonmatí (figure 4.18) at a temperature of 75 ºC, a pH 9.5 is required for a 50 % ammonia reduction and a pH of 10.5 for a 90 % ammonia reduction.

  The LIFE METHAMORPHOSIS project being carried out at Ecoparc 2 has studied this technology in more depth and results of approximately 50 % ammonium reduction have been obtained after 3 hours without the need to add any reagents to regulate the pH. This is due to the fact that, as the aeration time increases, the total and partial alkalinity effluent decreases, thus increasing the pH. The higher the pH the greater the ammonium removal, reaching a pH of 9.5.

# GENERAL CONCLUSIONS

From the four phases of the thesis project carried out over the last 4 years we can conclude:

During the first phase, all the Ecoparc 2 wastewaters were determined, coding all the effluents, identifying all existing connection points, installing water meters and calculating the flow of effluents where possible. From all of this characterisation, it has been able to classify and distinguish the different wastewater types at Ecoparc 2.

The areas with the highest municipal water (MW) consumption are the WWTP, air treatment and anaerobic digestion.

The refrigeration towers and the evaporator vacuum pump in the WWTP area consume 35 % of the MW . The water that the refrigeration towers discharge contain very low COD and ammonium loads. For this reason, a deposit tank has been installed that captures the water discharged from these towers. The water is analysed to check if it is possible to reuse it as clean water for equipment cleaning and processing points and, if so, is sent to the rainwater tank for reuse.

The grey water (GW) is collected in a retention tank and analysed before it is discharged to determine if it needs to be treated. Approximately 100 $m^3$/day GW is produced, of which 26 % comes from the air treatment area. 26 % of the GW contains 55 % of the discharge of the total ammonia concentration. For this reason, we decided to use it for cleaning the centrifuges and for the preparation of the flocculant in the anaerobic digestion area. In this way, the GW with higher concentration pass to the process water (PW) ending their treatment by the WWTP, without being mixed with the rest of the GW. A pipeline is currently being installed that channels the GW from the air treatment and sends it to the anaerobic digestion, allowing this effluent to be reused.

Of the 26.4 $m^3$/day GW from air treatment, 30 % is related to the humidifiers and 16 % to the biofilters. To reduce these values, the humidifiers droplet separators have been modified thus minimising both the MW consumption and Grey Water. The biofilters irrigation needs to be regulated to maintain the required bed humidity without generating surplus water discharge. However, because of the current delicate state of the biofilters, it has not yet been possible to carry out this regulation.

The MW consumption at Ecoparc 2 has been significantly reduced, obtaining the best results from 2014 onwards. Of the 10 years registered (2007-2016), 2016 has the lowest consumption with 62,079 $m^3$ compared to 115,555 $m^3$ registered in 2009, which was the highest MW consumption at Ecoparc 2. This is a very substantial reduction of 46.3 % in relation to 2007. With respect to 2013, which was when this project began, the reduction of MW consumption is also a significant 33.8 %.

The changes made in the GW reuse from the air treatment area and WWTP serve several purposes: to reduce the MW consumption when being reused, to improve the discharge quality by not mixing the effluents with higher COD and ammonium concentration with the rest of the GW and to reduce the amount of discharge.

Being able to reuse part of the GW produces a reduction of the discharge of this water and keeps constant the amount to be treated in the WWTP. This is because they end their journey in the PW tank to be treated by the WWTP. As a result, the amount of permeate remains stable while GW discharge has been reduced by 41.2 %, which means a value of 40,681 $m^3$ less discharge in relation to the year 2014.

In addition, a tank has been added that collects the GW from the retention tank and the permeate in order to homogenise the discharge since its production is very variable in quantity, quality and frequency. In this way it is possible to avoid the effect of the high discharge produced during the peak hours compared to other hours in which there is practically none. The homogenisation tank increases the wastewater retention time and provides a smooth discharge in terms of quantity and helps avoid damaging the industrial collector pumps, it also stabilises the concentration with a more constant discharge.

The PW production is very variable with values between 75 and 122 $m^3$/day. Moreover, the variability of these PW is very high since between 75.4 and 96.6 % comes from the anaerobic digestion area, which differs greatly from the rest. A wide range in both production and origin caused this instability of the amount and PW properties.

The Ecoparc 2 WWTP is a biological process, as a consequence process variations in both PW and external factors (ambient temperature for example) create destabilisation and affect performance. For this reason, it is necessary to homogenise the PW in a way that smooths out the variations and favours stability in the system. Currently, modifications are being carried out in the feed tanks to the WWTP for this purpose.

On the other hand, the solids contained in the PW cause difficulties to the biological process since they increase the system viscosity, making solubility and diffusion of nutrients and dissolved oxygen more difficult. The end result is a poorer performance in the biological process. For this reason, it is necessary to act to minimise the solids introduced into the nitrification and denitrification tanks.

Different flocculants types have been tested in the anaerobic digestion centrifuge to retain the maximum amount of PW solids in this area, which accounts for 75.4 to 96.6 % of the total PW. It has been observed in the tests carried out with a double centrifuge stage that better results are obtained than in those of only one stage. The FLOPAM TH 4650 VHM flocculant gave the best results after subjecting it to the double centrifugal stage giving a liquid phase with a content between 1.43 and 2.28 % of DM, however, it requires a high flocculant consumption (14.86 kg/Tn DM). In contrast, the Zetag 8185 flocculant produces a liquid phase with values of 2.22% DM with 7.0 kg/Tn DM flocculant consumption. A good performance of HYFLOC XT-653 and FLOPAM TE 4650 SH flocculants has also been observed, which with a single centrifuge stage already obtain values of 2.06 % DM in the liquid phase with 7.4 kg/Tn DM flocculant consumption in both cases. The HYFLOC XT-653 and Zetag 8185 flocculants are currently being used with a double centrifuge stage, obtaining values of TS < 3% compared to those historically obtained of TS < 5 %. A rotary filter is also being incorporated to retain solids from the PW that do not come from the anaerobic digestion. At present the WWTP sludge contains 22 % DM compared to 35-40% obtained historically.

Control parameters and analytical tests have been incorporated to all the reactors to be able to determine the WWTP state, minimising the response times to process destabilisation. Equations have been established for the calculation of phosphoric acid, sodium hydroxide, methanol and purge to adjust them to the current needs of the process. In this way, it is possible to maintain optimal process conditions by avoiding the accumulation of solids and by obtaining a higher removal of forms of nitrogen (ammonium, nitrites and nitrates) and COD.

The methanol dosing, aeration and pause times of the combined reactors have been extended to favour the oxia/anoxia conditions thus improving the nitrification and denitrification processes.

At present, the PW to be treated contains 59.9 % less COD (22,000 mg $O_2$/L approximately) and 58.3 % less ammonium (approximately 3,000 mg N/L).

We are acting in a way to minimise the methanol consumption, thus reducing the economic process cost and optimising the COD content degradation.

At present, it is not necessary to resort to the evaporator as an auxiliary to the WWTP, since the biological WWTP is capable of treating all the PW. In this way it has also been possible to minimise the MW consumption of the evaporator refrigeration tower and vacuum pump. The COD and ammonium concentration in the permeate have also decreased, with values 39.3 % lower in COD and 62.2% lower in ammonium in relation to 2008 and 38.0 % less in COD and 10.9 % less in ammonium in relation to year 2013 when this project was started.

Furthermore, possible pretreatment processes have also been studied to improve the PW to be treated, facilitating the biological purification process.

In order to reduce the amount of solids from the anaerobic digestion area PW, the effect of centrifuging the effluent has been studied. For this, it must be subjected to a double centrifugation stage. In the tests carried out at the laboratory scale, it was observed that a higher centrifugation velocity gave a greater solids separation and COD reduction. In the industrial scale tests, the flocculant HYFLOC XT-653 was used with 2.5 g/L concentration. Five tests with favourable results were obtained in which the liquid and solid phases had a good aspect, determining as best parameters 2,000 rpm with 5 relative speed, 7.5 m3/h sludge flow and 2,300 L/h flocculant flow, in which 1.39 % DM concentration is obtained in the liquid phase with 8 kg/Tn DM flocculant consumption. Currently, the double centrifugation stage has been implemented as the standard procedure in the treatment of anaerobic digestion PW.

In parallel, the separation of the solids by commercial membranes has been studied. First, the particle size distribution of effluent was determined, obtaining two main distributions: one of microorganisms with 1 micron size and another of heterogeneous solids with measurements from 10 microns to the millimetric range. The effect of sonication on particle distribution was studied without obtaining significant differences, so it was concluded that the samples did not contain aggregates. The samples were given a pretreatment consisting of sieving the sample to 100 microns to remove the coarser solids and diluting the sample to decrease the high viscosity (from 16.92 cP to 3.83 cP), without obtaining significant differences in the particle size distribution. Twelve commercial membrane types with different pore size were tested for solids separation after the pretreatment of 100 micron screening and 50 % dilution. The tests were carried out with different samples: the centrifuge liquid phase, the centrifuge

double stage liquid phase, and the total PW before being introduced to the WWTP. In the PW case, it was observed that the solids contained different size distributions and caused major problems in which membrane fouling was high. However, in the case of the centrifuge liquid phase of both one and two stages, good results were obtained in which the permeate did not contain suspended solids and presented a low turbidity. The double stage sample gave the best results with respect to the fouling and recovery of membrane permeability. The membranes in the microfiltration range were sufficient for solids separation, but better results have been obtained in membranes of the ultrafiltration range.

Finally, the possibility of an increase in COD biodegradability contained in the PW has been determined by thermal hydrolysis. Taking into account that the solid phase must undergo a hygienisation process at 70 ºC and that at higher temperatures it presents an oily aspect not feasible for agricultural application of this material, the maximum temperature at which the effluent can be treated before the centrifugation is 75 ºC. Moreover, it does not constitute an increase in process cost, since it is necessary to apply this treatment for the solid phase hygiene. In the tests carried out, it was observed that better results are obtained at a higher temperature and a longer exposure time of effluent at this temperature. The best result being a 24 % increase in COD solubility after the effluent was subjected to a thermal hydrolysis at 75 ºC over a period of 120 minutes.

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# ANNEX

UNIVERSITAT ROVIRA I VIRGILI
OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2
Maria Eloisa Albacete Garcia

# ANNEX I. PHASE 5. NEW BIOLOGICAL PROCESSES

UNIVERSITAT ROVIRA I VIRGILI
OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2
Maria Eloisa Albacete Garcia

# PHASE 5. NEW BIOLOGICAL PROCESSES

## 5.1. Introduction

The possibility of testing a new biological process to eliminate COD and ammonia loading of Ecoparc 2 leachate was also considered as part of this project. Special attention was given to Anammox processes, which do not require the contribution of carbon sources (COD or methanol) and has lower oxygen requirements. Consequently an energy and economic saving is expected.

Figure 5.1 shows the most common form of nitrogen removal from wastewater via nitrification-denitrification.

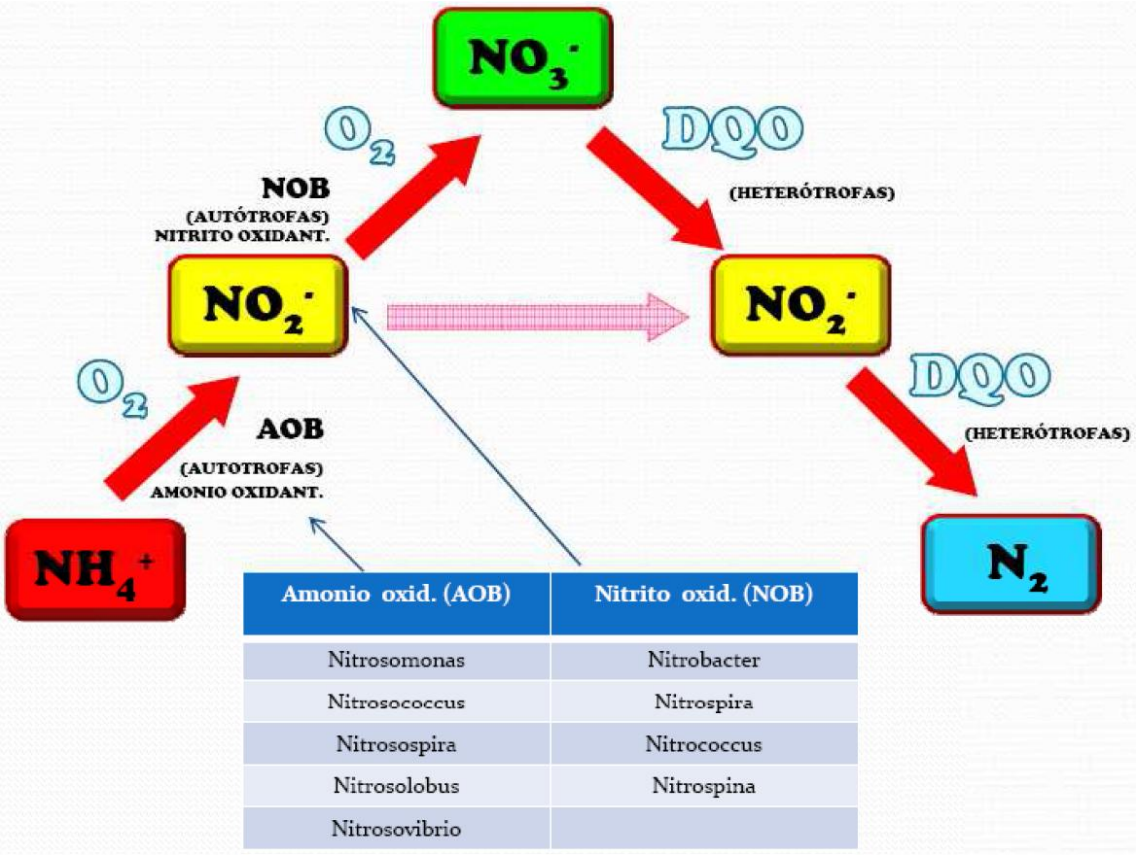


***Figure 5.1:*** *nitrogen removal via nitrification-denitrification*

Nitrification is a process that requires an oxygen supply to oxidise the ammonia to nitrites (eq. 5.1) and subsequently to nitrates (eq. 5.2). However, in the denitrification

process it is necessary to provide a carbon source to reduce nitrates to elemental nitrogen (eq. 5.3). The carbon source may come either from COD content or from an external input of methanol or acetic acid. Therefore, for nitrogen removal in wastewater via nitrification-denitrification it is necessary to provide more oxygen and a carbon source.

$NH_4^+ + 1{,}5\ O_2 \rightarrow NO_2^- + H_2O + 2\ H^+$ (eq. 5.1)

$NO_2^- + 0{,}5\ O_2 \rightarrow NO_3^-$ (eq. 5.2)

$4\ NO_3^- + 4\ H^+ + 5\ [C] \rightarrow 2\ N_2 + 5\ CO_2 + 2\ H_2O$ (eq. 5.3)

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$4\ NH_4^+ + 8\ O_2 + 5\ [C] \rightarrow 2\ N_2 + 4\ H^+ + 6\ H_2O + 5\ CO_2$ (eq. 5.4)

Figure 5.2 shows the most common form of nitrogen removal from wastewater via nitrites.

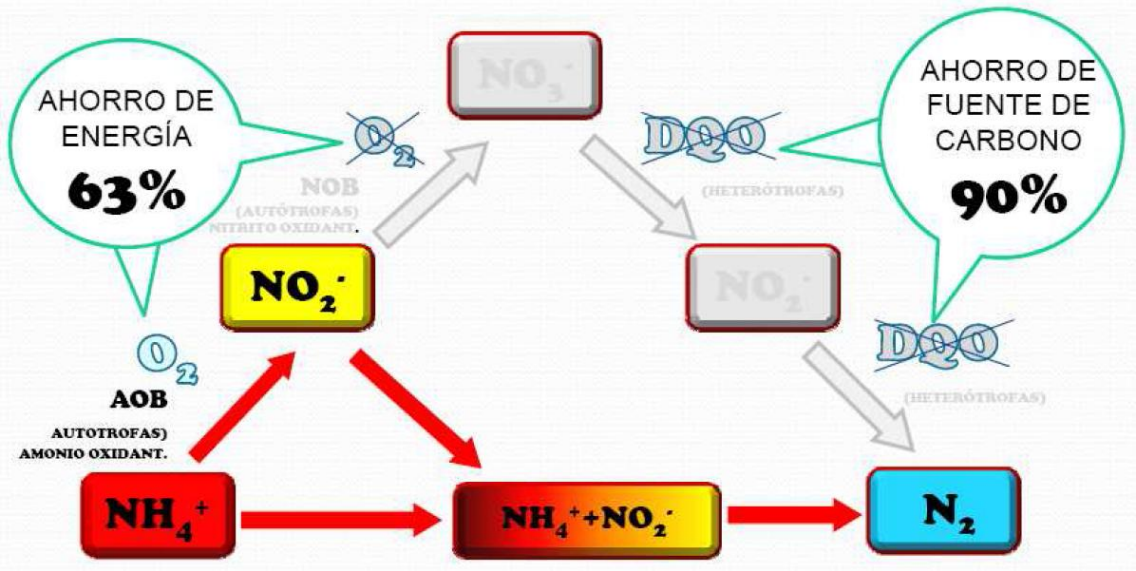


***Figure 5.2:*** *nitrogen removal via nitrites*

The nitrite form of removal is followed by the Anammox process (ANaerobic AMMonium OXidation). The ammonium part passes to nitrites in the same way as happens in the nitrification process, with an oxygen supply (eq. 5.1). However, less oxygen is needed as no oxygen supply is required to pass to the nitrates and gives an energy saving in the Anammox process. The other ammonium part reacts with the nitrites to form elemental nitrogen (eq. 5.5). Therefore, in the Anammox process, it is not necessary to contribute a carbon source unlike the denitrification process.

$NH_4^+ + 1{,}5\ O_2 \rightarrow NO_2^- + H_2O + 2\ H^+$ (eq. 5.1)

$NH_4^+ + NO_2^- \rightarrow N_2 + 2\ H_2O$ (eq. 5.5)

---

$2\ NH_4^+ + 1{,}5\ O_2 \rightarrow N_2 + 2\ H^+ + 3\ H_2O$ (eq. 5.6)

If we compare the two resulting equations in nitrification-denitrification (eq. 5.4) and in nitrite (eq. 5.6) we see that in each case four ammonia molecules are consumed, two elemental nitrogen, four protons, and six water molecules. However, it is also observed that in the nitrification-denitrification, eight oxygen molecules are required compared to three of Annamox, as well as an unnecessary carbon source in the Anammox process. In addition, the Anammox process does not produce $CO_2$ emissions while through the nitrification-denitrification process five $CO_2$ molecules are produced for every four ammonia molecules consumed.

$4\ NH_4^+ + 8\ O_2 + 5\ [C] \rightarrow 2\ N_2 + 4\ H^+ + 6\ H_2O + 5\ CO_2$ (vía nitrification-denitrification)

$4\ NH_4^+ + 3\ O_2 \rightarrow 2\ N_2 + 4\ H^+ + 6\ H_2O$ (vía Anammox)

According to the article *Proceso Anammox una aplicación en ingeniería. Revisión general de los aspectos microbianos* there exist several different Anammox process designs such as OLAND, CANON, SNAD, SHANON and other combined processes, of which the most studied and used on the industrial scale are:

- **CANON (Completely Autotrophic Nitrogen removal Over Nitrite)**

This process uses a single reactor in which the enriched microbial communities are autotrophs. It was conceived with the idea of treating 500 – 600 mg N/L ammonium concentrations. The reactions involved are as follows:

$NH_4^+ + 1.5\ O_2 \rightarrow NO_2^- + 2\ H^+ + H_2O$ (for nitrification, controlled until 1.32 $NO_2^-/NH_4^+$ ratio)

$NH_4^+ + 1.32\ NO_2^- \rightarrow 1.02\ N_2 + 0.26\ NO_3^- + 2.03\ H_2O$ (for Anammox reaction)

In this system, the aerobic nitrite oxidising bacteria present in the floccules experience a double limitation, they compete with Nitrosomas for oxygen and with Anammox for nitrite. In contrast, ammonium oxidisers have only one limitation: Nitrosomas for oxygen and Anammox for nitrite. The aeration control has a very important role in the

system for the nitrite oxidisers proliferation when there is a high concentration of dissolved oxygen and inhibition of aerobic ammonium oxidisers with low concentrations. A FISH biomass analysis of a CANON reactor showed that 40 % of the population corresponded to ammonium oxidising aerobic bacteria and Anammox cells constituted about another 40 %. Other advantages include the reduction of 20% in $CO_2$ emissions and low sludge production.

- **OLAND (Oxygen-Limited Autotrophic Nitrification Denitrification)**

This is a process in which ammonia is oxidised to molecular nitrogen under limited oxygen conditions, with which partial nitrification is achieved. In fact, it is based on the same CANON process principles already mentioned above, its process being similar but applied in different reactor conditions, specifically in a biofilter or adhered biomass reactor.

- **SHARON-Anammox**

This is a combined process of partial nitrification with Anammox in two separate reactors in series. It consists of an initial controlled aerobic ammonium transformation to nitrite to oxidise approximately 44 % of ammonium affluent in a SHARON type reactor . Afterwards, in a second Anammox reactor it is denitrificated to molecular nitrogen. This is a patented process, based on the partial nitrification by aerobic microorganisms and partial denitrification by facultative anaerobic heterotrophic microorganisms. Generally, the Anammox reactor is of a SBR (Sequential Batch Reactor) type. The process produces little sludge and only requires 40% of the aeration energy needed in a conventional nitrogen removal process. The modified SHARON reactor effluent contains a mixture of ammonium and nitrite, ideal for the Anammox process, in which these two compounds are converted to nitrogen gas. The treated wastewater must have ammonium concentrations higher than 500 mg N/L. It does not require biomass retention. The reaction in the SHARON reactor can be generalised as follows:

$$NH_4^+ + HCO_3 + 0.75\ O_2 \rightarrow 0.5\ NH_4^+ + 0.5\ NO_2^- + CO_2 + 1.5\ H_2O$$

There are several patents on Anammox processes that show that this biological process type was already studied in the 80's:

- *Anoxic ammonia oxidation*, publication number US5078884A dated January 7, 1992 and filing date February 2, 1989.

- *Method of treating ammonia-comprising waste water*, publication number US6383390B1 dated May 7, 2002 and filing date August 25, 1997.

Again, in this phase the LIFE METHAMORPHOSIS project will study this process for the anaerobic digestion leachate treatment at Ecoparc 2, and is the reason why in this project we decided to study the solids separation for a WWTP improvement at Ecoparc 2.

## 5.2. Conclusions

Throughout this phase the following conclusions have been obtained:

- In addition to the WWTP optimisation and study of other new technologies for the wastewater treatment, new wastewater treatment process options at Ecoparc 2 have been considered from other biological processes. For this purpose the Anammox process has been studied, which converts ammonia to elemental nitrogen via nitrites. This process has a lower energy consumption because less dissolved oxygen is needed, and a lower cost because an external carbon source contribution such as methanol or acetic acid is not required.
  On the other hand, it should be taken into account that in the Anammox process the leachate COD content is not consumed, so other ways of eliminating the organic load have to be sought.

- On comparing the nitrogen removal via the nitrification-denitrification process via nitrite, it has been observed that both processes produce two elemental nitrogen molecules for four ammonium molecules, obtaining in both processes two protons and six water molecules. However, via nitrification-denitrification, eight oxygen molecules are needed instead of three as in the Annamox process, which leads toa reduction of 62.5 % energy in the system. Moreover, the Anammox process does not produce $CO_2$ unlike the process via nitrification-

denitrification that emits five $CO_2$ molecules to the atmosphere4 for every four ammonia molecules.

- On the other hand, in the Anammox process, an external carbon source is unnecessary, which eliminates the methanol or acetic acid consumption.
  In CANON and OLAND processes, however, it has been observed that the Anammox reaction produces 0.26 nitrates molecules for each ammonia molecule consumed, so in this case a carbon contribution would be necessary to denitrify the nitrates to elemental nitrogen. In the case of Ecoparc 2 it would not be necessary due to the leachate COD content.

# ANNEX II. ANALYSES AND METHODS

UNIVERSITAT ROVIRA I VIRGILI
OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2
Maria Eloisa Albacete Garcia

REF 985 028

en

# Test 0-28 07.14
# *NANOCOLOR*® COD 15000
## Chemical Oxygen Demand

**Method:**
Photometric determination of chromium(III) concentration after oxidation with potassium dichromate / sulfuric acid / silver sulfate

| | |
|---|---|
| Range: | **1.0–15.0 g/L COD (1000–15000 mg/L COD)** |
| Factor: | **017.4** |
| Wavelength (HW = 5–12 nm): | **620 nm** |
| Reaction time: | **2 h** |
| Reaction temperature: | **148 °C** |
| Short time COD: | **30 min at 160 °C*** |

**Contents of reagent set:**
20 test tubes COD 15000
1 test tube with blank value "NULL"

**Hazard warning:**
Test tubes contain sulfuric acid 51–80 %, potassium dichromate 0.28–0.56 % and mercury(II) sulfate 0.37–0.74 %. Blank value "NULL" contains sulfuric acid 51–80 %.
H314, H340, H350, EUH203 Causes severe skin burns and eye damage. May cause genetic defects. May cause cancer. Contains chromium(VI). May produce an allergic reaction.
P201, P202, P260, P280, P301+330+331, P303+361+353, P304+340, P305+351+338, P308+313, P405, P501 Obtain special instructions before use. Do not handle until all safety precautions have been read and understood. Do not breathe vapors. Wear protective gloves / eye protection. IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water / shower. IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. IF exposed or concerned: Get medical advice / attention. Store locked up. Dispose of contents / container to regulated waste treatment. For further information ask for a safety data sheet. When shaking COD test tubes use safety bottle (REF 916 37).

**Interferences:**
For **chloride contents above 15000 mg/L** the test sample must be diluted. For determination of the concentration of chlorides we recommend a preliminary test with QUANTOFIX® Chloride (REF 913 21).
Turbidity in the COD test tube after reaction in the heating block will result in COD readings which are too high. Wait until turbidities caused by precipitation of mercury sulfate have deposited. When interpreting the results please remember the high dilution.

The method can not be applied for the analysis of sea water.

**Procedure:**
Requisite accessories: *NANOCOLOR*® heating block, piston pipette with tips

***Note:*** *For samples with high chloride concentrations it is important to shake the test tube* ***before*** *the water sample is added in order to suspend the deposit.*

**according to DIN ISO 15705 at 148 °C**

Open test tube, hold it **diagonally** and **slowly** add
**0.2 mL (= 200 µL)** test sample,
screw cap securely on to test tube, hold tube by the cap, place tube into the safety bottle and shake, then place tube into the heating block and start.
After 2 h remove test tube from heating block, after 10 min *(test tube is still warm)* shake once and allow to cool to room temperature.
Clean outside of the test tube and measure.

**Short time COD at 160 °C**

Open test tube, hold it **diagonally** and **slowly** add
**0.2 mL (= 200 µL)** test sample,
screw cap securely on to test tube, hold tube by the cap, place tube into the safety bottle and shake, then place tube into the heating block and start.
After 30 min remove test tube from heating block, after 10 min *(test tube is still warm)* shake once and allow to cool to room temperature.
Clean outside of the test tube and measure.

** In contrast to the conditions described in the ISO 15705, the short time COD is characterized by a higher digestion temperature and reduced reaction time. Therefore we recommend to compare the results of the short time COD from time to time with measurements made under the conditions of ISO 15705 (150 ± 5 °C / 2 h ± 10 min).*

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-28.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* COD 15000 (REF 925 28) or Multistandard Seepage water (REF 925 013)

**Storage:**
Store the test kit in a cool and dry place. Avoid exposing the test kit to sunlight.

**References:**
German standard methods for the examination of water, waste water and sludge (DIN 38 409 - H41-1)
British standard: Field and on-site test methods for the analysis of waters (BS 1427)

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Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***

PD 14122 / A011684 / 985 028 / 07412

REF 985 038

en

# Test 0-38 03.14
# *NANOCOLOR*® COD HR 1500
## Chemical Oxygen Demand

**Method:**
Photometric determination of chromium(III) concentration after oxidation with potassium dichromate / sulfuric acid / silver sulfate

| | |
|---|---|
| Range: | **20–1500 mg/L COD** |
| Wavelength (HW = 5–12 nm): | **620 mm** |
| Reaction time: | **2 h** |
| Reaction temperature: | **150 °C** |

**Contents of reagent set:**
20 test tubes COD HR 1500

**Hazard warning:**
Test tubes contain sulfuric acid 80–98 %, potassium dichromate 0.28–0.56 % and mercury(II) sulfate 0.74–1.50 %.
H314, H340, H350, EUH203 Causes severe skin burns and eye damage. May cause genetic defects. May cause cancer. Contains chromium(VI). May produce an allergic reaction.
P201, P202, P260, P280, P301+330+331, P303+361+353, P304+340, P305+351+338, P308+313, P405 Obtain special instructions before use. Do not handle until all safety precautions have been read and understood. Do not breathe vapors. Wear protective gloves / eye protection. IF SWALLOWED: rinse mouth. Do NOT induce vomiting. IF ON SKIN (or hair): Remove / Take off immediately all contaminated clothing. Rinse skin with water / shower. IF INHALED: Remove to fresh air and keep at rest in a position comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. IF exposed or concerned: Get medical advice / attention. Store locked up. For further information ask for a safety data sheet. When shaking COD test tubes use safety bottle (REF 916 37).

**Interferences:**
For **chloride contents above 2000 mg/L** the test sample must be diluted or use Chloride complexing agent (REF 918 911). For determination of the concentration of chlorides we recommend a preliminary test with QUANTOFIX® Chloride (REF 913 21).
Turbidity in the COD test tube after reaction in the heating block will result in COD readings which are too high. Wait until turbidities caused by precipitation of mercury sulfate have deposited.

The method cannot be applied for the analysis of sea water.

**Procedure:**
Requisite accessories: *NANOCOLOR*® heating block, piston pipette with tips

***Note:*** *For samples with high chloride concentrations it is important to shake the test tube* ***before*** *the water sample is added in order to suspend the deposit.*

1. Open test tube and **carefully** add **2.0 mL** sample *(Caution: Solution may heat up).*
2. Screw cap on the test tube, place tube into the safety bottle and shake.
3. Heat test tube for 2 h at 150 °C.
4. Sway test tube.
5. Allow test tube to cool to room temperature (20–25 °C).
6. Clean outside of test tube.
7. Insert the test tube in the photometer, measurement starts automatically.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-38.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* COD 1500 (REF 925 29) or Multistandard Sewage influx (REF 925 012)

**Storage:**
Store the test kit in a cool and dry place. Avoid exposing the test kit to sunlight.

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A045818 / 985 038 / 0440.5

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 014 1000 – 10000 mg/L

*Lichtgeschützt aufbewahren*
*Stocker à l'abri lumière*
*Conservare al riparo dalla luce*
*In het donker bewaren*
*Protect against light*

*Lagerhinweis*
*Stabilité*
*Conservazione*
*Houdbaarheid*
*Storage* +15°C ..... +25°C

## Datatabel / Data table

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***07/2000*** |
| ***LCK 014 *)*** • *F1 = 0 • F2 = 616.6 • K = -11.94* | |
| ***CADAS 30/30S/50/50S*** | ***09/2001*** |
| ***LCK 014 *)*** • *λ: 605 nm • Pro.: 1 • F1 = 0 • F2 = 6101 • K = -322.2* | |
| ***ISIS 6000/9000*** | ***09/2001*** |
| ***LCK 014 *)*** • *λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 6286 • K = -355.4* | |
| ***CADAS 100 / LPG 158*** | ***06/2000*** |
| ***LCK 014 *)*** • *λ: 605 nm • F1 = 6104 • F2 = -131.1* | |
| ***CADAS 100 / LPG 210*** | ***06/2000*** |
| ***LCK 014 *)*** • *λ: 605 nm • F1 = 6104 • F2 = -131.1* | |
| ***CADAS 200*** | ***09/2001*** |
| ***LCK 014 *)*** • *E1W1 • C1 = E1*F1-F2 • W1 = 605 nm • F1 = 6060 • F2 = 338.8* | |

****) CZV klassiek / HT***
***COD classic / HT***

NL

## LCK 014 CZV
## Chemisch zuurstof verbruik

***Let a.u.b. op de "Uitgave datum" (zie datatabel) en lees de "Opmerking". Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
Oxideerbare stoffen reageren met een zwavelzure kaliumdichromaatoplossing in aanwezigheid van zilversulfaat als katalysator. Chloride wordt met kwiksulfaat gemaskeerd. Gemeten wordt de groene kleur van het $Cr^{3+}$.

**Toepassingsgebied**
Afvalwater, procesanalyse

**Storingen**
De methode kan worden toegepast in water-monsters met een chloridegehalte van maximaal 5000 mg/L. Hogere chlorideconcentratie geven een te hoog resultaat.
De meetresultaten zijn via een plausibiliteitsonder-zoek te controleren (verdunning en/of standaard-additie).

**Opmerking!**
***In vergelijking met de klassieke CZV kuvettentest (CZV klassiek) is de hogere ontsluitingstemperatuur en korte ontsluitingstijd een belangrijk kenmerk van de HT-CZV.***
***In de praktijk wordt een vergelijking met de klassieke methode geadviseerd om er zeker van te zijn dat de HT-CZV voor de eigen monsters vergelijkbare resultaten oplevert.***

GB

## LCK 014 COD
## Chemical Oxygen Demand

***Please check the "Edition Date" (see data table) and read the "Note". Safety advice and expiry date on package.***

**Principle**
Oxidizable substances react with sulphuric acid – potassium dichromate solution in the presence of silver sulphate as a catalyst. Chloride is masked by mercury sulphate. The green coloration of $Cr^{3+}$ is evaluated.

**Range of Application**
Waste water, process analysis

**Interferences**
The method can be used for water samples with chloride concentrations of up to 5000 mg/L. Higher chloride concentrations cause high-bias results. The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**Note**
***In contrast to the classic COD Cuvette Test (COD classic) the HT-COD is characterised by a higher digestion temperature and shorter digestion time.***
***Users are advised to carry out a comparison with the COD classic, in order to be sure that the results obtained from their own samples when using the HT-COD are comparable to the standard.***

HACH LANGE
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# LCK 014 1000 – 10000 mg/L

| | |
|---|---|
| *Lichtgeschützt aufbewahren*<br>*Stocker à l'abri lumière*<br>*Conservare al riparo dalla luce*<br>*In het donker bewaren*<br>*Protect against light* | *Lagerhinweis*<br>*Stabilité*<br>*Conservazione*<br>*Houdbaarheid*<br>*Storage* +15°C ..... +25°C |

## *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***07/2000*** |
| ***LCK 014 **** • *F1 = 0 • F2 = 616.6 • K = -11.94* | |
| ***CADAS 30/30S/50/50S*** | ***09/2001*** |
| ***LCK 014 **** • *λ: 605 nm • Pro.: 1 • F1 = 0 • F2 = 6101 • K = -322.2* | |
| ***ISIS 6000/9000*** | ***09/2001*** |
| ***LCK 014 **** • *λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 6286 • K = -355.4* | |
| ***CADAS 100 / LPG 158*** | ***06/2000*** |
| ***LCK 014 **** • *λ: 605 nm • F1 = 6104 • F2 = -131.1* | |
| ***CADAS 100 / LPG 210*** | ***06/2000*** |
| ***LCK 014 **** • *λ: 605 nm • F1 = 6104 • F2 = -131.1* | |
| ***CADAS 200*** | ***09/2001*** |
| ***LCK 014 **** • *E1W1 • C1 = E1*F1-F2 • W1 = 605 nm • F1 = 6060 • F2 = 338.8* | |

****) CSB classic / HT***
***DCO classiques / HT***
***COD classica / HT***

D

## LCK 014 CSB
## Chemischer Sauerstoffbedarf

***Bitte "Ausgabedatum" (s. Datentabelle) und "Hinweis" beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Oxidierbare Stoffe reagieren mit schwefelsaurer Kaliumdichromatlösung in Gegenwart von Silbersulfat als Katalysator. Chlorid wird mit Quecksilbersulfat maskiert. Ausgewertet wird die Grünfärbung des $Cr^{3+}$.

**Anwendungsbereich**
Abwasser, Prozessanalytik

**Störungen**
Die Methode ist bis zu einem Chloridgehalt von 5000 mg/L in der Wasserprobe anwendbar. Höhere Chloridkonzentrationen führen zu Mehrbefunden. Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**Hinweis**
***Im Vergleich zum klassischen CSB Küvetten-Test (CSB classic) zeichnet sich der HT-CSB durch eine höhere Aufschlusstemperatur und kürzere Aufschlusszeit aus.***
***Für die Praxis wird der Vergleich mit dem CSB classic empfohlen, um sicherzustellen, dass der HT-CSB für die eigenen Proben vergleichbare Ergebnisse zur Norm liefert.***

F

## LCK 014 DCO
## Demande Chimique en Oxygène

***Vérifier la date d'édition (voir table des données) et lire la "Remarque". Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Les substances oxydables réagissent avec le bichromate de potassium sulfurique, en présence de sulfate d'argent. Le chlorure est masqué avec du sulfate de mercure. La coloration verte du $Cr^{3+}$ sera déterminée photométriquement.

**Domaine d'application**
Eaux de rejet, analyses en mode contenu

**Perturbations**
Cette méthode est applicable pour des échantillons d'eau ayant une teneur en chlorure de 5000 mg/L max. Les concentrations en chlorure plus élevées sont à l'origine des résultats trop élevés.
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**Remarque**
***En comparaison avec les Tests en Cuve DCO classiques (DCO classiques), le HT-DCO offre une température de désagrégation plus élevée, ainsi qu'un temps de désagrégation réduit.***
***Dans la pratique, la comparaison avec les DCO classiques est recommandée, afin de vous assurer que le HT-DCO fournit des résultats analogues dans les normes pour les différents échantillons.***

I

## LCK 014 COD
## Domanda Chimica di Ossigeno

***Si prega di verificare la "Data di Edizione" (vedi tabella dati) e di leggere le "Note". Avvertenze e data di scadenza sulla confezione.***

**Principio**
Reazione con soluzione di acido solforico e dicromato potassico più solfato di argento quale catalizzatore. I cloruri vengono mascherati col solfato di mercurio. La colorazione verde del $Cr^{3+}$ viene letta fotometricamente.

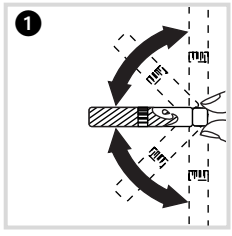
**Applicazione**
Acque di scarico, analisi di processo

**Interferenze**
Il metodo è valido per un contenuto di cloruri nel campione fino a 5000 mg/L. Più alte concentrazioni di cloruri danno risultati superiori.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**Note**
***In rapporto all'analisi classica del COD, con i test in cuvetta, l'HT-COD esegue l'ossidazione a una temperatura più alta e in tempi più rapidi.***
***E' consigliato ogni tanto eseguire dei COD secondo la metodologia classica (2h, 148°C) oltre che con l'HT-COD per accertarsi che i risultati siano confrontabili.***

AD 014 Q / Druckfarbe schwarz / 1

**LCK 014** *09/2001*



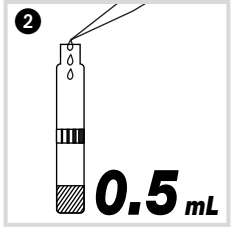
Bodensatz durch Schwenken in Schwebe bringen.

Mélanger le contenu pour avoir une solution homogène.

Agitare delicatamente per sospendere il fondo.

Bezinking door schudden in suspensie brengen.

Bring the sediment into suspension by inverting a few times.



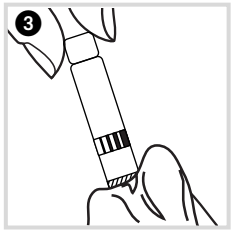

***0.5 mL*** Probe ***vorsichtig*** pipettieren.

Pipetter ***0.5 mL*** d'échantillon ***avec précaution***.

Pipettare ***attentamente 0.5 mL*** di campione.

***0.5 mL*** monster ***voorzichtig*** pipetteren.

***Carefully*** pipette ***0.5 mL*** sample.



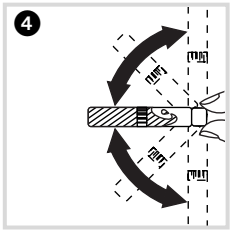
Küvette verschließen, von außen gut säubern.

Fermer la cuve et nettoyer l'extérieur de celle-ci.

Tappare la cuvetta, pulirla bene esternamente.

Kuvet sluiten, van buiten goed reinigen.

Close cuvette, thoroughly clean the outside.



Schwenken.

Mélanger.

Mescolare.

Zwenken.

Invert.

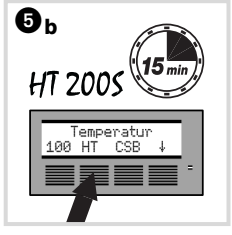

Im Thermostaten erhitzen.
***a) CSB classic:*** ***2 Std*** bei ***148°C***
***b) HT 200 S:*** ***15 min*** im Standardprogramm ***HT***

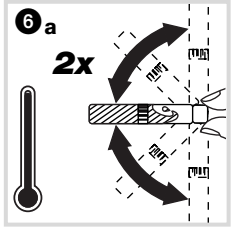
Chauffer dans le thermostat.
***a) DCO classique:*** ***2 h*** à ***148°C***
***b) HT 200 S:*** ***15 min*** avec le programme standard ***HT***

Riscaldare nel termostato.
***a) COD classica:*** ***2 h*** a ***148°C***
***b) HT 200 S:*** ***15 min*** nel programma standard ***HT***

In het thermostaat verhitten.
***a) CZV klassiek:*** ***2 h*** bij ***148°C***
***b) HT 200 S:*** ***15 min*** in standaard-programma ***HT***

Heat in the thermostat.
***a) COD classic:*** ***2 h*** at ***148°C***
***b) HT 200 S:*** in standard program ***HT*** for ***15 min***



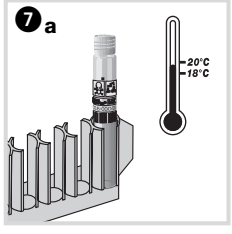

***Heiße*** Küvette entnehmen.
***a) CSB classic:*** ***2 x vorsichtig*** schwenken.
***b) HT 200 S:*** Nach Freigabe der Verriegelung ***2 x vorsichtig*** schwenken.

Sortir la cuve ***chaude***.
***a) DCO classique:*** Retourner ***2 x avec précaution***.
***b) HT 200 S:*** Après le déverrouillage, retourner ***2 x avec précaution***.

Estrarre la cuvetta ***calda***.
***a) COD classica:*** Agitare ***delicatamente 2 volte***.
***b) HT 200 S:*** Dopo il rilascio del dispositivo di bloccaggio, agitare ***delicatamente 2 volte***.

Het ***hete*** kuvet eruit nemen.
***a) CZV klassiek:*** ***2x voorzichtig*** zwenken.
***b) HT 200 S:*** Na de vrijgeving van de afsluitbeveiliging, ***2x voorzichtig*** zwenken.

Remove the ***hot*** cuvette.
***a) COD classic:*** ***Carefully*** invert ***twice***.
***b) HT 200 S:*** After the lock opens, ***carefully*** invert ***twice***.



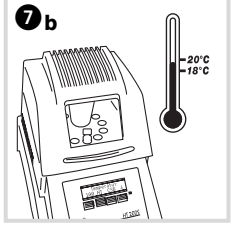

Auf Raumtemperatur abkühlen.
***a) CSB classic:*** im Küvettenständer
***b) HT 200 S:*** im Thermostaten

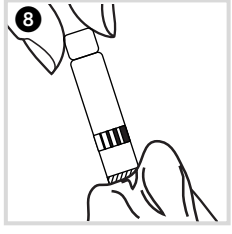
Laisser refroidir à température ambiante.
***a) DCO classique:*** dans le support de cuve
***b) HT 200 S:*** dans le thermostat

Lasciare raffreddare a temperatura ambiente.
***a) COD classica:*** in un portacuvetta
***b) HT 200 S:*** nel termostato

Laten afkoelen tot kamertemperatuur.
***a) CZV klassiek:*** in kuvettenstandaard
***b) HT 200 S:*** in thermostaat

Allow to cool to room temperature.
***a) COD classic:*** in a cooling rack
***b) HT 200 S:*** in the thermostat



| | |
|---|---|
| ***CSB classic:*** | Küvette außen gut säubern und auswerten. |
| ***HT 200 S:*** | Feststoffteilchen müssen vor der Auswertung vollständig abgesetzt sein! Küvette außen gut säubern und auswerten. |
| ***DCO classique:*** | Bien nettoyer l'extérieur de la cuve et mesurer. |
| ***HT 200 S:*** | Les résidus doivent être complètement éliminés avant l'évaluation. Bien nettoyer l'extérieur de la cuve et mesurer. |
| ***COD classica:*** | Pulire bene la cuvetta esternamente e leggere. |
| ***HT 200 S:*** | Prima dell'analisi il sedimento deve essersi completamente depositato. Pulire bene la cuvetta esternamente e leggere. |
| ***CZV klassiek:*** | Kuvet van buiten goed reinigen en meten. |
| ***HT 200 S:*** | De nog aanwezige vaste stof moet voor de meting volledig bezonken zijn. Kuvet van buiten goed reinigen en meten. |
| ***COD classic:*** | Clean the outside of the cuvette and evaluate. |
| ***HT 200 S:*** | Sediment must be completely settled before evaluation is carried out. Clean the outside of the cuvette and evaluate. |

## *Auswertung / Evaluation / Lettura / Meting*

| | Analysenküvette ❶<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|
| Barcode [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Test ❸<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Kontrollnr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|---|
| **LASA 1 / plus** | 590 nm | -- | CSB *) / HTCSB *) LCK 014 | 2 | ✔ |
| **LASA 10 / 20** | -- | _ : 46 | CSB *) / HTCSB *) LCK 014 | 2 | ✔ |

*) DCO / COD / CZV

**LP1W**

❼ (Ergebnis x 3.15) -119.4 = mg/L CSB
❼ (Résultat x 3.15) -119.4 = mg/L DCO
❼ (Risultato x 3.15) -119.4 = mg/L COD
❼ (Meetresultaat x 3.15) -119.4 = mg/L CZV
❼ (Result x 3.15) -119.4 = mg/L COD

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Test ❷<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Faktor ❸<br>Facteur<br>Fattore<br>Factor<br>Factor | Kontrollnr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❺<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) Null | Analysenküvette ❻<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette Ergebnis | ❼ |
|---|---|---|---|---|---|---|---|
| **LP1W** | 605 nm | -- | 1960 | -- | LCW 919 | ✔ | ✔ |
| **LP2W** | 605 nm / CSB 114 | CSB *) LCK 014 | -- | 7 | LCW 919 | ✔ | -- |

*) DCO / COD / CZV

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Mode ❸ | Test ❹<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Kontrollnr. ❺<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette, grüne Taste / Messen ❻<br>Cuve d'analyse, touche verte / Mesurer<br>Cuvetta d'analisi, tasto verde / Lettura<br>Analyse-kuvet, groene toets / Meten<br>Sample cuvette, green key / Read |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 46 | -- | 014 | 7 | ✔ |
| **ISIS 6000** | -- | _ : 46 | [2] | 014 | 7 | ✔ |
| **LASA 30** | 605 nm | -- | Dr. Lange | 014 | 7 | ✔ |
| **DR 1900** | -- | -- | [3] | 014 | 7 | ✔ |

[2] KÜVETTEN-TEST
[2] TEST EN CUVE
[2] CUVETTE-TEST
[2] KUVETTENTEST
[2] CUVETTE TEST

[3] BARCODE-PROGRAMME
[3] PROGR. CODE BARRE
[3] PROGRAMMI COD. A BARRE
[3] BARCODEPROGRAMMA'S
[3] BARCODE PROGRAMS

| | Mode ❶ | Symbol ❷<br>Symbole<br>Simbolo<br>Symbool<br>Symbol | Kontrollnr. ❸<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❹<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) NULL | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette MESS |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | $ 014 | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | 014 | 7 | LCW 919 | ✔ |

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 514

**100–2000 mg/L**

*In het donker bewaren*
*Protect against light*
*Işığa karşı koruma*

*Houdbaarheid*
*Storage*
*Saklama*
**+15 °C ..... +25 °C**

**T1**

## *Datatabel · Data table · Veri tablosu*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***06/1997*** |
| ***LCK 514 *)*** • *F1 = 0 • F2 = 2071 • F3 = -35.81* | |
| ***CADAS 30/30S/50/50S*** | ***07/2001*** |
| ***LCK 514 *)*** • *λ: 605 nm • Pro.: 1 • F1 = 0 • F2 = 2045 • K = -105.7* | |
| ***ISIS 6000/9000*** | ***07/2001*** |
| ***LCK 514 *)*** • *λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 2118 • K = -122.2* | |
| ***CADAS 100/LPG 158*** | ***06/1997*** |
| ***LCK 514 *)*** • *λ: 605 nm • F1 = 2046 • F2 = -37.39* | |
| ***CADAS 100/LPG 210*** | ***06/1997*** |
| ***LCK 514 *)*** • *λ: 605 nm • F1 = 2046 • F2 = -37.39* | |

**)* ***CZV klassiek/HT***
***COD classic/HT***
***COD klasik / HT***

**NL**

## LCK 514 CZV
## Chemisch zuurstof verbruik

***Let a.u.b. op de "Uitgave datum" (zie datatabel) en lees de "Opmerking". Veiligheidsadvies en houdbaarheidsdatum op de verpakking.***

**Principe**
Oxideerbare stoffen reageren met een zwavelzure kaliumdichromaatoplossing in aanwezigheid van zilversulfaat als katalysator. Chloride wordt met kwiksulfaat gemaskeerd. Gemeten wordt de groene kleur van het $Cr^{3+}$.

**Toepassingsgebied**
Afvalwater, procesanalyse

**Storingen**
De methode kan worden toegepast in monsters met een chloridegehalte van maximaal 1500 mg/L. De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaardadditie).

**Opmerking!**
***In vergelijking met de klassieke CZV kuvettentest (CZV klassiek) is de hogere ontsluitingstemperatuur en korte ontsluitingstijd een belangrijk kenmerk van de HT-CZV. In de praktijk wordt een vergelijking met de klassieke methode geadviseerd om er zeker van te zijn dat de HT-CZV voor de eigen monsters vergelijkbare resultaten oplevert.***

**Speciale aandachtspunten**
Voor een optimale stabiliteit tot de houdbaarheidsdatum, wordt opslag van de kuvettentest LCK 514 in een koelkast aanbevolen.

**EN**

## LCK 514 COD
## Chemical Oxygen Demand

***Please check the "Edition Date" (see data table) and read the "Note". Safety advice and expiry date on package.***

**Principle**
Oxidizable substances react with sulphuric acid – potassium dichromate solution in the presence of silver sulphate as a catalyst. Chloride is masked by mercury sulphate. The green coloration of $Cr^{3+}$ is evaluated.

**Range of Application**
Waste water, process analysis

**Interferences**
The method can be used for samples (or diluted samples) with chloride concentrations of up to 1500 mg/L.
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**Note**
***In contrast to the classic COD Cuvette Test (COD classic) the HT-COD is characterised by a higher digestion temperature and shorter digestion time. Users are advised to carry out a comparison with the COD classic, in order to be sure that the results obtained from their own samples when using the HT-COD are comparable to the standard.***

**Special note**
For optimal stability until it's expiry date, it is recommended the reagent LCK 514 is stored in a fridge.

**TR**

## LCK 514 COD
## Kimyasal Oksijen İsteği

***Lütfen "Baskı Tarihi"ni kontrol edin (bkz. veri tablosu) ve "Not"u okuyun. Güvenlik önerisi ve son kullanma tarihi ambalajın üzerindedir.***

**Prensip**
Okside edilebilir maddeler gümüş sülfatın katalizör olarak bulunduğu ortamda sülfürik asit – potasyum dikromat solüsyonuyla reaksiyona girer. Civa sülfatın bulunduğu ortamda klorür görünmez. $Cr^{3+}$ yeşil rengi aldığında değerlendirilir.

**Uygulama Alanları**
Atık su, proses analizi

**Girişim Yapan Maddeler**
Bu metot 1500 mg/L'ye kadar klorür konsantrasyonlu numunelerde (veya seyreltilmiş numunelerde) kullanılır.
Ölçüm sonuçlarında olasılık kontrolü yapılmalıdır (numuneyi seyreltin ve/veya katkılayın).

**Not**
***HT-COD testini klasik COD Küvet Testinden (COD klasik) ayıran özellikler daha yüksek sindirim sıcaklığı ve daha düşük sindirim süresidir. Kullanıcıların HT-COD kullanırken kendi numunelerinden aldıkları sonuçların standartlara uygun olduğundan emin olmaları için COD klasikle kıyaslamaları önerilir.***

**Özel not**
Son kullanma tarihine kadar stabilitesini koruması için, LCK 514'ün buzdolabında saklanması tavsiye edilmektedir.

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 514

**100–2000 mg/L**

Lichtgeschützt aufbewahren
Stocker à l'abri lumière
Conservare al riparo dalla luce

Lagerhinweis
Stabilité
Conservazione
+15 °C ..... +25 °C

T1

## Datentabelle · Table des données
## Tabella dati

| | |
|---|---|
| **DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000** | **06/2013** |
| **Software Download: www.hach-lange.com** | |
| **LP2W** | **06/1997** |
| **LCK 514 *)** • F1 = 0 • F2 = 2071 • F3 = -35.81 | |
| **CADAS 30/30S/50/50S** | **07/2001** |
| **LCK 514 *)** • λ: 605 nm • Pro.: 1 • F1 = 0 • F2 = 2045 • K = -105.7 | |
| **ISIS 6000/9000** | **07/2001** |
| **LCK 514 *)** • λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 2118 • K = -122.2 | |
| **CADAS 100/LPG 158** | **06/1997** |
| **LCK 514 *)** • λ: 605 nm • F1 = 2046 • F2 = -37.39 | |
| **CADAS 100/LPG 210** | **06/1997** |
| **LCK 514 *)** • λ: 605 nm • F1 = 2046 • F2 = -37.39 | |

***) CSB classic/HT**
**DCO classiques/HT**
**COD classica/HT**

DE

## LCK 514 CSB
## Chemischer Sauerstoffbedarf

***Bitte "Ausgabedatum" (s. Datentabelle) und "Hinweis" beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Oxidierbare Stoffe reagieren mit schwefelsaurer Kaliumdichromatlösung in Gegenwart von Silbersulfat als Katalysator. Chlorid wird mit Quecksilbersulfat maskiert. Ausgewertet wird die Grünfärbung des $Cr^{3+}$.

**Anwendungsbereich**
Abwasser, Prozessanalytik

**Störungen**
Die Methode ist bis zu einem Chloridgehalt von 1500 mg/L in der Probe (oder verdünnten Probe) anwendbar.
Messergebnisse sind durch eine Plausibilitäts-kontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**Hinweis**
***Im Vergleich zum klassischen CSB Küvetten-Test (CSB classic) zeichnet sich der HT-CSB durch eine höhere Aufschlusstemperatur und kürzere Aufschlusszeit aus.***
***Für die Praxis wird der Vergleich mit dem CSB classic empfohlen, um sicherzustellen, dass der HT-CSB für die eigenen Proben vergleichbare Ergebnisse zur Norm liefert.***

**Besonders beachten**
Für eine optimale Stabilität der Reagenzien bis zum Ablauf der Haltbarkeit empfehlen wir, den Test LCK 514 im Kühlschrank zu lagern.

FR

## LCK 514 DCO
## Demande Chimique en Oxygène

***Vérifier la date d'édition (voir table des données) et lire la "Remarque". Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Les substances oxydables réagissent avec le bichromate de potassium sulfurique, en présence de sulfate d'argent. Le chlorure est masqué avec du sulfate de mercure. La coloration verte du $Cr^{3+}$ sera déterminée photométriquement.

**Domaine d'application**
Eaux de rejet, analyses en mode contenu

**Perturbations**
Cette méthode est applicable pour des échantillons (ou échantillon dilué) ayant une teneur en chlorure de 1500 mg/L max.
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**Remarque**
***En comparaison avec les Tests en Cuve DCO classiques (DCO classiques), le HT-DCO offre une température de désagrégation plus élevée, ainsi qu'un temps de désagrégation réduit.***
***Dans la pratique, la comparaison avec les DCO classiques est recommandée, afin de vous assurer que le HT-DCO fournit des résultats analogues dans les normes pour les différents échantillons.***

**Remarque importante**
Afin d'optimiser sa stabilité jusqu'à la date d'expiration, il est recommandé de conserver le kit LCK 514 au réfrigérateur.

IT

## LCK 514 COD
## Domanda Chimica di Ossigeno

***Si prega di verificare la "Data di Edizione" (vedi tabella dati) e di leggere le "Note". Avvertenze e data di scadenza sulla confezione.***

**Principio**
Reazione con soluzione di acido solforico e dicromato potassico più solfato di argento quale catalizzatore. I cloruri vengono mascherati col solfato di mercurio. La colorazione verde del $Cr^{3+}$ viene letta fotometricamente.

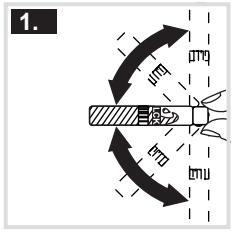
**Applicazione**
Acque di scarico, analisi di processo

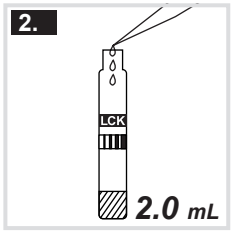
**Interferenze**
Il metodo è valido per un contenuto di cloruri nel campione (originale o diluito) fino a 1500 mg/L.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

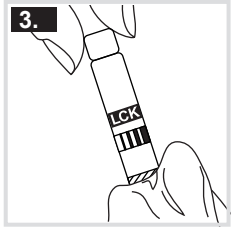
**Note**
***In rapporto all'analisi classica del COD, con i test in cuvetta, l'HT-COD esegue l'ossidazione a una temperatura più alta e in tempi più rapidi. E' consigliato ogni tanto eseguire dei COD secondo la metodologia classica (2h, 148 °C) oltre che con l'HT-COD per accertarsi che i risultati siano confrontabili.***

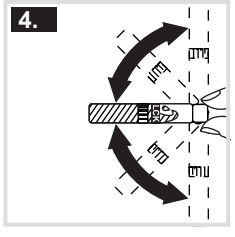
**Nota importante**
Per garantire una stabilità ottimale fino alla data di scadenza, è consigliabile conservare il reagente LCK 514 in frigo.

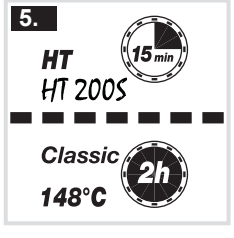
AD 514 P / Druckfarbe blau / 1

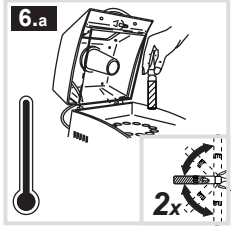


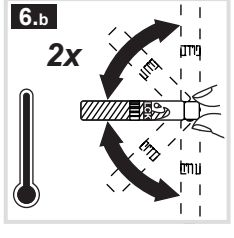



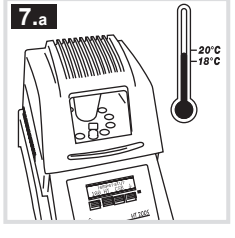



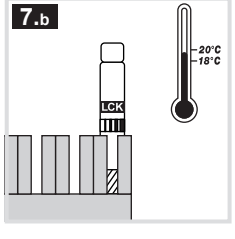



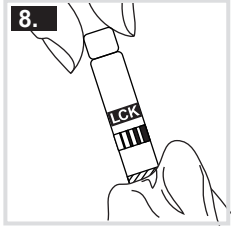




## DE

1. Bodensatz durch Schwenken in Schwebe bringen.
2. ***2.0 mL*** Probe ***vorsichtig*** pipettieren.
3. Küvette verschließen, von außen gut säubern.
4. Schwenken.
5. Im Thermostaten erhitzen.
   ***HT 200 S: 15 min*** im Standardprogramm ***HT***
   ***CSB classic: 2 Std*** bei ***148 °C***
6. ***Heiße*** Küvette entnehmen.
   a. ***HT 200 S:*** Nach Freigabe der Verriegelung ***2 x vorsichtig*** schwenken.
   b. ***CSB classic: 2 x vorsichtig*** schwenken.
7. Auf Raumtemperatur abkühlen.
   a. ***HT 200 S:*** im Thermostaten
   b. ***CSB classic:*** im Küvettenständer
8. ***HT 200 S:*** Feststoffteilchen müssen vor der Auswertung vollständig abgesetzt sein! Küvette außen gut säubern und auswerten.
   ***CSB classic:*** Küvette außen gut säubern und auswerten.

## FR

1. Mélanger le contenu pour avoir une solution homogène.
2. Pipetter ***2.0 mL*** d'échantillon ***avec précaution***.
3. Fermer la cuve et nettoyer l'extérieur de celle-ci.
4. Mélanger.
5. Chauffer dans le thermostat.
   ***HT 200 S: 15 min*** avec le programme standard ***HT***
   ***DCO classique: 2 h*** à ***148 °C***
6. Sortir la cuve ***chaude***.
   a. ***HT 200 S:*** Après le déverrouillage, retourner ***2 x avec précaution***.
   b. ***DCO classique:*** Retourner ***2 x avec précaution***.
7. Laisser refroidir à température ambiante.
   a. ***HT 200 S:*** dans le thermostat
   b. ***DCO classique:*** dans le support de cuve
8. ***HT 200 S:*** Les résidus doivent être complètement éliminés avant l'évaluation. Bien nettoyer l'extérieur de la cuve et mesurer.
   ***DCO classique:*** Bien nettoyer l'extérieur de la cuve et mesurer.

## IT

1. Agitare delicatamente per sospendere il fondo.
2. Pipettare ***attentamente 2.0 mL*** di campione.
3. Tappare la cuvetta, pulirla bene esternamente.
4. Mescolare.
5. Riscaldare nel termostato.
   ***HT 200 S: 15 min*** nel programma standard ***HT***
   ***COD classica: 2 h*** a ***148 °C***
6. Estrarre la cuvetta ***calda***.
   a. ***HT 200 S:*** Dopo il rilascio del dispositivo di bloccaggio, agitare ***delicatamente 2 volte***.
   b. ***COD classica:*** Agitare ***delicatamente 2 volte***.
7. Lasciare raffreddare a temperatura ambiente.
   a. ***HT 200 S:*** nel termostato
   b. ***COD classica:*** in un portacuvetta
8. ***HT 200 S:*** Prima dell'analisi il sedimento deve essersi completamente depositato. Pulire bene la cuvetta esternamente e leggere.
   ***COD classica:*** Pulire bene la cuvetta esternamente e leggere.

## NL

1. Bezinking door schudden in suspensie brengen.
2. ***2.0 mL*** monster ***voorzichtig*** pipetteren.
3. Kuvet sluiten, van buiten goed reinigen.
4. Zwenken.
5. In het thermostaat verhitten.
   ***HT 200 S: 15 min*** in standaard-programma ***HT***
   ***CZV klassiek: 2 h*** bij ***148 °C***
6. Het ***hete*** kuvet eruit nemen.
   a. ***HT 200 S:*** Na de vrijgeving van de afsluit-beveiliging, ***2 x voorzichtig*** zwenken.
   b. ***CZV klassiek: 2 x voorzichtig*** zwenken.
7. Laten afkoelen tot kamertemperatuur.
   a. ***HT 200 S:*** in thermostaat
   b. ***CZV klassiek:*** in kuvettenstandaard
8. ***HT 200 S:*** De nog aanwezige vaste stof moet voor de meting volledig bezonken zijn. Kuvet van buiten goed reinigen en meten.
   ***CZV klassiek:*** Kuvet van buiten goed reinigen en meten.

## EN

1. Bring the sediment into suspension by inverting a few times.
2. ***Carefully*** pipette ***2.0 mL*** sample.
3. Close cuvette, thoroughly clean the outside.
4. Invert.
5. Heat in the thermostat.
   ***HT 200 S:*** in standard program ***HT*** for ***15 min***
   ***COD classic: 2 h*** at ***148 °C***
6. Remove the ***hot*** cuvette.
   a. ***HT 200 S:*** After the lock opens, ***carefully*** invert ***twice***.
   b. ***COD classic: Carefully*** invert ***twice***.
7. Allow to cool to room temperature.
   a. ***HT 200 S:*** in the thermostat
   b. ***COD classic:*** in a cooling rack
8. ***HT 200 S:*** Sediment must be completely settled before evaluation is carried out. Clean the outside of the cuvette and evaluate.
   ***COD classic:*** Clean the outside of the cuvette and evaluate.

## TR

1. Çökeltiyi birkaç kez ters çevirerek karışmasını sağlayın.
2. ***Dikkatlice 2.0 mL*** numune ekleyin.
3. Küveti kapatın ve dışını iyice temizleyin.
4. Ters çevirin.
5. Termostatı ısıtın.
   ***HT 200 S:*** standart program ***HT'***de ***15 dk***.
   ***COD klasik: 148 °C'***de ***2 saat***.
6. Sıcak küveti ***çıkarın***.
   a. ***HT 200 S:*** Kilit açıldıktan sonra, ***dikkatlice iki kez ters*** çevirin.
   b. ***COD klasik: İki kez dikkatlice*** ters çevirin.
7. Oda sıcaklığına gelmesini bekleyin.
   a. ***HT 200 S:*** termostatta
   b. ***COD klasik:*** soğutma rafında
8. ***HT 200 S:*** Değerlendirme yapmadan önce çökelti tamamen çökmelidir. Küvetin dışını temizleyin ve değerlendirmeye alın.
   ***COD klasik:*** Küvetin dışını temizleyin ve değerlendirmeye alın.

| | DE: *Für folgende Barcode-Geräte erfolgt nach Einsetzen der Analysenküvette eine automatische Auswertung:* | FR: *Si vous utilisez un des instruments avec codes à barres suivants, une évaluation automatique est réalisée après l'insertion de la cuve d'analyse :* | IT: *Se si utilizza uno qualsiasi dei seguenti strumenti con codice a barre, dopo aver inserito la cuvetta d'analisi viene automaticamente visualizzato il risultato della misura:* | NL: *Wanneer een van de volgende barcode instrumenten worden gebruikt, wordt een automatische uitwaardering uitgevoerd zodra de analyse-kuvet geplaatst wordt:* | EN: *If any of the following barcode instruments is used, an automatic evaluation is carried out after the sample cuvette is inserted:* | TR: *Aşağıdaki barkod cihazlarından biri kullanılıyorsa, numune küveti takıldıktan sonra değerlendirme otomatik olarak yapılır:* |
|---|---|---|---|---|---|---|
| IBR IBR+ | **LASA 50 / 100, XION 500, CADAS 30 / 50 / 30S / 50S / 200 Barcode, ISIS 9000, DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000** | | | | | |

| DE | FR | IT | NL | EN | TR | ⬇ | LASA 1 / plus | LASA 10 / 20 | CADAS 200 Basis | ISIS 6000 | LASA 30 | DR 1900 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Filter | Filtre | Filtro | Filter | Filter | Filtre | 1 | 590 nm | – | – | – | 605 nm | – |
| Eprom | Eprom | Eprom | Eprom | Eprom | Eprom | 2 | _ : 46 | _ : 46 | _ : 46 | _ : 46 | – | – |
| Mode | Mode | Mode | Mode | Mode | Mod | 3 | – | – | – | KÜVETTEN-TEST[1] | Dr. Lange | BARCODE-PROGRAMME[3] |
| Test anwählen | Test choisir | Test selezionare | Test oproepen | Test select | Test seçme | 4 | CSB[2]/ HTCSB[2] LCK 514 | CSB[2]/ HTCSB[2] LCK 514 | 514 | 514 | 514 | 514 |
| Kontrollnr. | No. de contrôle | No. di controllo | Controlegetal | Control no. | Kontrol no. | 5 | – | 1 | 5 | 5 | 5 | 5 |
| Analysenküvette | Cuve d'analyse | Cuvetta d'analisi | Analyse-kuvet | Sample cuvette | Numune küveti | 6 | ✓ | ✓ | – | – | – | – |
| Analysenküvette, grüne Taste / Messen | Cuve d'analyse, touche verte / Mesurer | Cuvetta d'analisi, tasto verde / Lettura | Analyse-kuvet, groene toets / Meten | Sample cuvette, green key / Read | Numune küveti, yeşil düğme / Oku | 7 | – | – | ✓ | ✓ | ✓ | ✓ |

| DE | FR | IT | NL | EN | TR | ⬇ | LP1W | LP2W | CADAS 100 LPG158 | CADAS 100 LPG210 |
|---|---|---|---|---|---|---|---|---|---|---|
| Filter | Filtre | Filtro | Filter | Filter | Filtre | 1 | 605 nm (LZP 420) | 605 nm | – | – |
| Mode | Mode | Mode | Mode | Mode | Mod | 2 | – | – | TEST | TEST |
| Symbol | Symbole | Simbolo | Symbool | Symbol | Sembol | 3 | – | – | $ 514 | 514 |
| Test anwählen | Test choisir | Test selezionare | Test oproepen | Test select | Test seçme | 4 | – | CSB[2] LCK 514 | – | – |
| Faktor | Facteur | Fattore | Factor | Factor | Faktör | 5 | 2071 | – | – | – |
| Kontrollnr. | No. de contrôle | No. di controllo | Controlegetal | Control no. | Kontrol no. | 6 | – | 9 | – | 5 |
| Leerwert (dest. Wasser) | Valeur à blanc (l'eau dist.) | Bianco (acqua dist.) | Blanko (gedest. water) | Blank-value (dist. water) | Şahit-değer (saf su) | 7 | LCW 919 NULL | LCW 919 NULL | LCW 919 NULL | LCW 919 NULL |
| Analysenküvette | Cuve d'analyse | Cuvetta d'analisi | Analyse-kuvet | Sample cuvette | Numune küveti | 8 | ✓ ERGEBNIS | ✓ ERGEBNIS | ✓ MESS | ✓ MESS |
| Vom Ergebnis abziehen: | Soustraire au résultat: | Sottrarre dal risultato: | Van het resultaat aftrekken: | Substract from the result: | Sonuçtan çıkarın: | 9 | 35.81 mg/L | – | – | – |

**FR:**
[1] TEST EN CUVE
[2] DCO
[3] PROGR. CODE BARRE

**IT:**
[1] CUVETTE-TEST
[2] COD
[3] PROGRAMMI COD.A BARRE

**NL:**
[1] KUVETTENTEST
[2] CZV
[3] BARCODE-PROGRAMMA'S

**EN:**
[1] CUVETTE TEST
[2] COD
[3] BARCODE PROGRAMS

**TR:**
[1] KÜVET TESTİ
[2] COD
[3] BARKOT PROGRAMLARI

REF 985 099

en

# Test 0-99 06.14
# *NANOCOLOR*® TOC 600

## Total organic carbon

**Method:**
The determination of TOC is carried out in two steps:
1. Disposing of the inorganic carbon **(TIC)**
2. Decomposition of the organic carbon **(TOC)** and detection of the carbon dioxide formed by means of an indicator

| | |
|---|---|
| Range: | **40–600 mg/L C** |
| Factor: | **0410. (-)** |
| Wavelength (HW = 5–12 nm): | **585 nm** |
| Decomposition time: | **2 h** |
| Decomposition temperature: | **120 °C** |

**Contents of reagent set:**
10 dilution test tubes V
10 test tubes TOC 600
1 test tube with 6 mL TOC R0
1 brown glass bottle with 1 g TOC R2
1 measuring spoon 70 mm
1 test tube with blank value „NULL“
2 thermo caps
10 round stickers

**Hazard warning:**
Reagent R0 contains sodium hydrogen sulfate 10–25 %, reagent R2 contains sodium peroxodisulfate 20–100 %. H317, H318, H334 May cause an allergic skin reaction. Causes serious eye damage. May cause allergy or asthma symptoms or breathing difficulties if inhaled.
P261, P272, P280, P302+352, P304+340, P305+351+338, P333+313, P342+311, P363 Avoid breathing dust. Contaminated work clothing should not be allowed out of the workplace. Wear protective gloves / eye protection. IF ON SKIN: Wash with plenty of water / .... IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. If skin irritation or rash occurs: Get medical advice / attention. If experiencing respiratory symptoms: Call a POISON CENTER / doctor / .... Wash contaminated clothing before reuse. For further information ask for a safety data sheet.

**Interferences:**
The following quantities will not interfere: ≤ 10000 mg/L $Cl^-$; ≤ 5000 mg/L TIC.

This method can not be applied for the analysis of sea water.

**Procedure:**
Requisite accessories: piston pipette with tips, glass beaker 100 mL, magnetic stirring unit, mini-magnet, *NANOCOLOR*® heating block

Please use a magnetic stirrer which can run at least at 900 rpm. The stir bar's size should fit the dimension of the beaker. For example, in case of a 100 mL beaker with 4.5 cm diameter, we suggest a stir bar of 3 cm length and at least 0.5 cm width (please also see *NANOCOLOR*® accessory sets for TOC determinations).

Recommended accessories for disposing of TIC:
*NANOCOLOR*® accessory set for the determination of TOC (small), content:
1 magnetic stirrer (1 stirr position), 2 beakers 100 mL, 2 magnetic stirr bars 35 mm (REF 916 990)
*NANOCOLOR*® accessory set for the determination of TOC (big), content:
1 magnetic stirrer (15 stirr positions), 6 beakers 100 mL, 6 magnetic stirr bars 35 mm (REF 916 991)
*NANOCOLOR*® beaker 100 mL with magnetic stir bar 35 mm, pack of 2 (REF 916 992)

**1. Disposing of inorganic carbon (TIC)**
Open **dilution test tube V**, add
**1.0 mL** test sample *(the pH value of the sample must be between pH 1 and 12)*, close and mix.
Fill contents of the dilution test tube into a glass beaker 100 mL with a mini-magnet, add
**0.5 mL** R0 and stir for **10 min** at maximum speed.

If samples are high in carbonates (high TIC content), we recommend to increase the stirring time. Depending on the sample matrix, it is necessary to check and adjust stirring time individually. Recommendation: When running the test for the first time or if changes in the sample matrix occur, we suggest to test a series of samples with different stirring times (e.g. 10, 30, 60 min) until TOC contents remain constant.

**2. Decomposition** **2 h / 120 °C**
Open **TOC test tube**, add
**4.0 mL** of the solution from step 1 and
**1 measuring spoon** R2, close with **thermo cap** and mix.
Place test tube **standing on its head** *(thermo cap at the bottom)* into the heating block with the blue indicator solution on top.
Set heating block to 120 °C and 2 h and press start.
After 2 h remove test tube from the heating block and leave the tube **standing on its head to cool down for 60 min** *(do not cool with cold water!)*.
After 60 min turn test tube upside down, clean outside of tube and measure the colored solution in the photometer.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-99.

**Note:**
*NANOCOLOR*® thermo caps for TOC decomposition are reusable. After measurement replace the thermo cap by the black screw cap. Clean thermo cap with distilled water, dry and use for further determinations.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* COD 1500 (REF 925 29): 160 ± 20 mg/L C

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***

PD 14122 / A036708 / 985 099 / 0640.5

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 386 30 – 300 mg/L

*Verpakkingen rechtop bewaren!*
*Store package in an upright position!*

*Houdbaarheid*
*Storage*
+2°C ..... +8°C

## *ACHTUNG / ATTENTION / ATTENZIONE / LET OP / NB*

### *(D) Besonders beachten*

1. **Kontamination durch Raumluft:** Die blauen Indikatorküvetten ***niemals*** offen stehen lassen, da das $CO_2$ der Raumluft Mehrbefunde verursachen kann. Indikatorküvetten erst dann öffnen und mit dem Doppeldeckel verschließen, wenn die Probenvorbereitung der Aufschlussküvette beendet ist. Die Aufschlussküvette dann ***sofort*** mit der Indikatorküvette verschließen.
2. **TIC austreiben:** Es muss mit dem Rüttler **TOC-X5** gearbeitet werden.
3. **Thermostat (HT 200 S):** Aufschlussbedingungen (***95°C***, ***2 h***) am Gerät einstellen. Küvettenkombination einsetzen, Gerät starten.
   **Andere Lange-Trockenthermostate:** Auf ***100°C*** vorheizen, nach Erreichen der Solltemperatur Küvettenkombination einsetzen und Zeit (***2 h***) starten.
4. **Auskristallisierung:** Auskristallisierungen in der Aufschlussküvette ***beeinträchtigen nicht*** das Ergebnis.

### *(F) Remarque importante*

1. **Contamination par l'air ambiant :** ***Ne*** laissez jamais les cuves indicatrices bleues ouvertes, car le $CO_2$ dans l'air peut entraîner des résultats à forte erreur systématique. Attendez que la préparation de l'échantillon dans la cuve de digestion soit terminée, puis ouvrez la cuve indicatrice et vissez le double bouchon à membrane. Ensuite, fermez ***immédiatement*** la cuve de digestion avec la cuve indicatrice.
2. **Expulsion du CIT :** L'agitateur **TOC-X5** doit être utilisé.
3. **Thermostat (HT 200 S) :** Définissez les conditions de digestion (***95°C***, ***2 h***) sur l'instrument. Insérez la combinaison de cuves et démarrez l'instrument.
   **Autres thermostats secs Lange :** Préchauffez à ***100°C***. Une fois la température désirée atteinte, insérez la combinaison de cuves et lancez la minuterie (***2 h***).
4. **Formation de cristaux :** La formation de cristaux dans la cuve de digestion est ***sans effet*** sur le résultat.

### *(I) Pro memoria*

1. **Contaminazione attraverso l'aria circostante:** ***Non*** lasciare mai aperte le cuvette indicatrici blu, in quanto la $CO_2$ presente nell'aria può causare sovrastime dei risultati. Attendere che la preparazione del campione nella cuvetta di digestione sia completata, aprire la cuvetta indicatrice ed avvitarci sopra il doppio cap. Quindi chiudere ***immediatamente*** la cuvetta di digestione con quella indicatrice.
2. **Eliminazione del TIC:** E' necessario utilizzare l'agitatore **TOC-X5**.
3. **Termostato (HT 200 S):** Impostare le condizioni di digestione (***95°C***, ***2 h***) sullo strumento. Inserire le cuvette ed avviare lo strumento.
   **Altri termostati tradizionali Lange:** Preriscaldare a ***100°C***. Quando la temperatura desiderata è stata raggiunta, inserire le cuvette e far partire il timer (***2 h***).
4. **Formazione di cristalli:** La formazione di cristalli nella cuvetta di digestione ***non influenza*** i risultati ottenuti.

### *(NL) Speciale aandachtspunten*

1. **Contaminatie door buitenlucht:** Laat ***nooit*** de blauwe indicatorkuvet open, daar de $CO_2$ in de omgevingslucht grote afwijkingen op de resultaten kan veroorzaken. Wacht tot de monstervoorbereiding in het ontsluitingskuvet voltooid is alvorens de indicatorkuvet te openen, en de dubbele dop dicht te schroeven. Sluit dan ***onmiddellijk*** de ontsluitingskuvet af met de indicatorkuvet.
2. **Uitdrijving van TIC:** Men moet hiervoor de **TOC-X5** triller gebruiken.
3. **Thermostaat (HT 200 S):** Stel de ontsluitingsvoorwaarden (***95°C***, ***2 h***) in op het instrument. Plaats er de kuvettencombinaties in en start de thermostaat.
   **Andere Lange thermostaten:** Voorverwarmen tot ***100°C***. Wanneer de gewenste temperatuur bereikt is, de kuvettencombinaties er in plaatsen en start de timer (***2 h***).
4. **Ontstaan van kristallen:** Het ontstaan van kristallen in de ontsluitingskuvet heeft ***geen invloed*** op het resultaat.

### *(GB) Special note*

1. **Contamination by ambient air:** ***Never*** leave the blue indicator cuvettes open, as $CO_2$ in the air can cause high-bias results to be obtained. Wait until the sample preparation in the digestion cuvette is complete and then open the indicator cuvette and screw on the double cap. Then close the digestion cuvette ***immediately*** with the indicator cuvette.
2. **Expulsion of TIC:** The **TOC-X5** shaker must be used.
3. **Thermostat (HT 200 S):** Set the digestion conditions (***95°C***, ***2 h***) on the instrument. Insert the cuvette combination and start the instrument.
   **Other Lange dry thermostats:** Pre-heat to ***100°C***. When the desired temperature is reached, insert the cuvette combination and start the time (***2 h***).
4. **Formation of crystals:** The formation of crystals in the digestion cuvette ***does not affect*** the result.

---

NL

## LCK 386 TOC
## Totaal organische koolstof

***Let a.u.b. op de "Uitgave datum" (zie datatabel) en lees de "Opmerking". Veiligheidsadvies en houdbaarheidsdatum op de verpakking.***

**Principe**
In een proces in twee stappen, wordt eerst de totale anorganische koolstof (**TIC** - total inorganic carbon) uitgestoten met behulp van de **TOC-X5** triller, waarna de totale organische koolstof (**TOC** - total organic carbon) tot koolstofdioxide ($CO_2$) wordt geoxideerd.
De $CO_2$ gaat door een membraan en komt in de indicatorkuvet, waar het een kleurverandering teweeg brengt, die met een fotometer wordt uitgewaarded.

**Toepassingsgebied**
Afvalwater, oppervlaktewater, proceswater

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht. De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaardadditie). Voor het verdunnen van het monster mag alleen water gebruikt worden dat vrij van koolstof is.

***Als het monster vaste deeltjes bevat, raadt de fabrikant aan het monster te verdunnen voor analyse.***

**pH-waarde** monster.....3 – 10
**Temperaturen** monster/reagentia.....15 – 25°C

**Belangrijk!**
Van de vereiste temperatuur van ***100°C*** dient u absoluut niet af te wijken (bij ***148°C*** kan de kuvetten combinatie ***verbroken*** worden).
Let erop dat u ***voorzichtig*** omgaat met de ontsloten kuvettencombinatie, omdat het, bij de ontsluiting, gevormde zuurstof een overdruk in het kuvet te weeg brengt. Bij sterke mechanische druk, bijv. bij het stoten of laten vallen van de uitgereageerde kuvettencombinaties kunnen deze springen.

**Retourname en verwerking**
De kuvettencombinaties na de analyse ***niet*** uit elkaar draaien, maar de gehele combinatie in de blister terug plaatsen.

---

GB

## LCK 386 TOC
## Total organic carbon

***Please check the "Edition Date" (see data table) and read the "Note". Safety advice and expiry date on package.***

**Principle**
In a two-stage process, the total inorganic carbon (**TIC**) is first expelled with the help of the **TOC-X5** shaker, then the total organic carbon (**TOC**) is oxidized to carbon dioxide ($CO_2$).
The $CO_2$ passes through a membrane into the indicator cuvette, where it causes a colour change to occur, which is evaluated with a photometer.

**Range of Application**
Waste water, surface water, process water

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample). Use only carbon-free water to dilute the sample.

***If the sample contains particles the manufacturer recommends diluting the sample before analysis.***

**pH** sample .....3 – 10
**Temperature** sample/reagents .....15 – 25°C

**NB:**
Be sure to set the required temperature to ***100°C*** (at ***148°C*** the cuvette combinations may ***break*** apart).
Please note that cuvette combinations must be handled ***with care*** after the reaction is completed, because oxygen is formed under the digestion conditions and this results in a build up of pressure in the cuvette combination. If the cuvette combinations are subjected to strong mechanical stress after the digestion reaction, e.g. if they suffer a blow or a fall, they may shatter.

**Disposal**
Do ***not*** screw the cuvette combinations apart when the analysis has been completed, but press them back into the blister pack.

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 386 30 – 300 mg/L

*Packung aufrecht lagern!*
*Conserver la boîte de réactif l'ouverture vers le haut!*
*Tenere la confezione in posizione verticale!*

*Lagerhinweis*
*Stabilité*
*Conservazione*
+2°C ..... +8°C

**T1**

**2000 mg/L:** $Ca^{2+}$, $Mg^{2+}$, $NH_4$-N

**1400 mg/L:** $Cl^-$

**250 mg/L:** TIC

## *Datentabelle / Table des données / Tabella dati / Datatabel / Data table*

| | |
|---|---|
| ***CADAS 30/30S/50/50S*** | ***09/2007*** |
| ***TOC / COT*** • *λ: 435 nm • Pro.: 15 • F1 = 0 • F2 = -80.97 • F3 = 484.1 • F4 = 0 • K1 = -175.7 • K2 = 0* | |
| ***ISIS 6000/9000*** | ***09/2007*** |
| ***TOC / COT*** • *λ: 430 nm • Pro.: 15 • F1 = 0 • F2 = -64.48 • F3 = 453.3 • F4 = 0 • K1 = -168.3 • K2 = 0* | |
| ***CADAS 200*** | ***09/2007*** |
| ***TOC / COT*** • *E1^2*F1+E1*F2-F3 • W1 = 435 nm • F1 = -67.10 • F2 = 453.3 • F3 = 161.9* | |
| ***DR 2800/3800*** | ***09/2007*** |
| ***TOC / COT*** • *λ: 435 nm • F1 = -75.45 • F2 = 474.33 • F3 = 171.30 • U1 = 1.000* | |
| ***DR 5000*** | ***09/2007*** |
| ***TOC / COT*** • *λ: 435 nm • F1 = -80.90 • F2 = 480.29 • F3 = 167.90 • U1 = 1.000* | |

**D**

## LCK 386 TOC
## Gesamt organischer Kohlenstoff

***Bitte "Ausgabedatum" (s. Datentabelle) und "Hinweis" beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
In einem zweistufigen Verfahren wird zunächst der gesamte anorganische Kohlenstoff (**TIC**) mit Hilfe des Rüttlers **TOC-X5** ausgetrieben und anschließend der gesamte organische Kohlenstoff (**TOC**) zu Kohlendioxid ($CO_2$) oxidiert.
Das $CO_2$ gelangt durch eine Membran in die Indikatorküvette und verursacht dort einen Farbumschlag, der photometrisch ausgewertet wird.

**Anwendungsbereich**
Abwasser, Oberflächenwasser, Prozesswasser

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt. Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung). Zur Verdünnung der Probe darf nur Wasser, das frei von Kohlenstoff ist, verwendet werden.

***Der Hersteller empfiehlt, partikelhaltige Proben vor der Analyse zu verdünnen.***

**pH-Wert** Probe.....3 – 10
**Temperatur** Probe.....15 – 25°C

**Wichtig!**
Einstellen der Solltemperatur von ***100°C*** bitte unbedingt beachten (bei ***148°C*** können die Küvettenkombinationen ***auseinanderbrechen***).
Wir weisen darauf hin, dass der Umgang mit den ausreagierten Küvettenkombinationen ***vorsichtig*** erfolgen muss, da durch die Aufschlussbedingungen Sauerstoff gebildet wird. Dieser führt zu einem Überdruck in den Küvettenkombinationen. Bei starker mechanischer Beanspruchung, z. B. Stoß oder Fall, können die ausreagierten Küvettenkombinationen zerspringen.

**Entsorgung**
Die Küvettenkombinationen nach Beendigung der Analyse ***nicht*** auseinanderschrauben, sondern in den Blister zurückdrücken.

**F**

## LCK 386 COT
## Carbone Organique Total

***Vérifier la date d'édition (voir table des données) et lire la "Remarque". Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Au cours d'un processus en deux phases, le carbone inorganique total (**CIT**) est d'abord expulsé à l'aide de l'agitateur **TOC-X5**, puis le carbone organique total (**COT**) est oxydé en dioxyde de carbone ($CO_2$).
Le $CO_2$ passe à travers une membrane dans la cuve indicatrice, où il entraîne un changement de couleur, qui est évalué à l'aide d'un photomètre.

**Domaine d'application**
Eaux de rejet, eaux de surface, eaux de procédé

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.
Les résultats des mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).
Pour la dilution de l'échantillon, n'utilisez que de l'eau exempte de carbone.

***Si l'échantillon contient des particules en suspension, le fabricant recommande de le diluer avant analyse.***

**pH** échantillon .....3 – 10
**Température** échantillon/réactifs .....15 – 25°C

**Important:**
Veillez au réglage correct de la température à ***100°C***.
A ***148°C***, les cuves combinées ***peuvent se neutraliser***.
Il est particulièrement important que la manipulation des cuves combinées après réaction se fasse avec ***précaution***: les conditions d´oxydation produisent de l'oxygène, ce qui entraîne une surpression dans les cuves combinées. En cas de forte sollicitation mécanique, telle qu'un coup ou une chute, les cuves combinées qui ont déjà réagi peuvent éclater.

**Elimination**
A la fin de l'analyse, ***ne*** pas séparer les cuves combinées l'une de l'autre. Mettre la combinaison de cuves complète dans le blister.

**I**

## LCK 386 TOC
## Carbonio organico totale

***Si prega di verificare la "Data di Edizione" (vedi tabella dati) e di leggere le "Note". Avvertenze e data di scadenza sulla confezione.***

**Principio**
Il metodo si completa in due stadi: il carbonio inorganico totale (**TIC**) viene prima espulso con l'ausilio dell'agitatore **TOC-X5** e successivamente il carbonio organico totale (**TOC**) viene ossidato ad anidride carbonica ($CO_2$).
La $CO_2$ passa attraverso una membrana nella cuvetta indicatrice, dove provoca il cambio di colore dell'indicatore che viene valutato per via spettrofotometrica.

**Applicazione**
Acqua di scarico, acqua di superficie, acqua di processo

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).
Per la diluizione del campione si deve usare solamente acqua , che non contiene carbonio.

***Se il campione presenta del particolato, vi consigliamo di diluire il campione prima dell'analisi.***

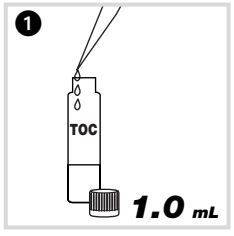
**pH** campione .....3 – 10
**Temperatura** campione/reagenti .....15 – 25°C

**Attenzione!**
Impostare la temperatura a ***100°C*** (a ***148°C*** le cuvette ***possono rompersi***).
Fare molta attenzione alla combinazione di cuvette: devono essere maneggiate con ***attenzione*** dopo che la reazione ha avuto luogo, poiché l'ossigeno formatosi durante la digestione determina un aumento notevole di pressione nella combinazione di cuvette. In seguito a colpi forti, p.es. la caduta delle cuvette, le cuvette possono rompersi.

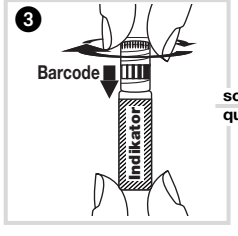
**Smaltimento**
Una volta terminata la reazione ***non*** svitare le cuvette, ma riporle come combinazione di cuvetta nei blister.

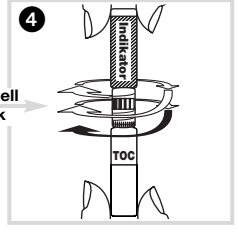
AD 386 G / Druckfarbe blau / 1

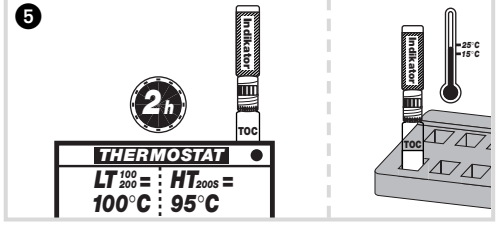
**LCK 386** 08/2005

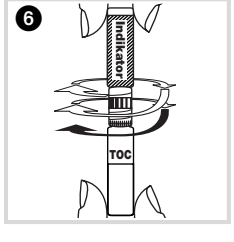


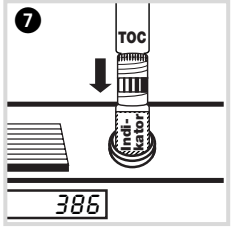





## D

### 1. Probenvorbereitung (TIC austreiben)

1. ***1.0 mL*** Probe in die Aufschlussküvette pipettieren.
2. Aufschlussküvette ***offen*** in den Rüttler **TOC-X5** einsetzen, bis zum Boden drücken, Ventilatordeckel über Küvette positionieren. Gerät einschalten. Nach ***5 min*** ertönt ein Signalton.

### 2. TOC-Bestimmung

3. Nach der Probenvorbereitung die blaue Indikatorküvette öffnen und ***sofort*** mit dem Membran-Doppeldeckel ***fest*** verschrauben. (**Achtung:** Barcode-Etikett muss zur Indikatorküvette zeigen).
4. ***Anschließend sofort*** die Aufschlussküvette mit der vorbereiteten Indikatorküvette ***fest*** verschließen. Küvettenkombination senkrecht halten und ***nicht*** schwenken.
5. Im Thermostat erhitzen (blaue Indikatorküvette oben).
   **a) HT 200 S:** ***2 h*** bei ***95°C***
   **b) $LT^{100}_{200}$:** Im vorgeheizten Thermostat ***(100°C) 2 h*** bei ***100°C***
   Anschließend auf Raumtemperatur abkühlen.
6. Küvettenkombination ***vor dem Umdrehen*** noch mal ***fest*** zudrehen.
7. Küvettenkombination umdrehen. Indikatorküvette außen gut säubern und auswerten.

## F

### 1. Préparation d'échantillon (expulsion du CIT)

1. Pipetter ***1.0 mL*** d'échantillon dans la cuve de digestion.
2. Insérez la cuve de digestion ***ouverte*** dans l'agitateur **TOC-X5**, en appuyant pour l'enfoncer le plus possible. Placez le couvercle du ventilateur sur la cuve. Mettez l'instrument sous tension. Après ***5 min***, un signal sonore est émis.

### 2. Détermination du COT

3. Lorsque la préparation d'échantillon est terminée, ouvrez la cuve indicatrice bleue et vissez ***immédiatement*** à fond le double bouchon à membrane. (**Attention :** l'étiquette du code à barres doit être dirigée vers la cuve indicatrice).
4. Fermez ***immédiatement*** à fond la cuve de digestion avec la cuve indicatrice préparée. Maintenir obligatoirement les cuves combinées à la verticale et ***ne pas*** les mélanger!
5. Chauffer dans le thermostat (la cuve indicatrice bleues toujours en haut).
   **a) HT 200 S:** ***2 h*** à ***95°C***
   **b) $LT^{100}_{200}$:** Dans un thermostat préchauffé ***(100°C) 2 h*** à ***100°C***
   Ensuite, laisser refroidir à température ambiante.
6. Serrez de nouveau la combinaison de cuves avant de la retourner.
7. Retourner la combinaison de cuves. Bien nettoyer l'extérieur de la cuve indicatrice et mesurer.

## I

### 1. Preparazione campioni (eliminazione del TIC)

1. Pipettare ***1.0 mL*** di campione in cuvetta di digestione.
2. Inserire la cuvetta di digestione ***aperta*** nell'agitatore **TOC-X5** spingendola più possibile verso il basso. Posizionare sopra la cuvetta il coperchio della ventola. Accendere lo strumento. Dopo ***5 min*** un segnale acustico segnalerà la fine della fase di agitazione.

### 2. Determinazione del TOC

3. Quando la preparazione del campione nell'agitatore è completata, aprire la cuvetta indicatrice blu ed avvitarci sopra ***immediatamente*** e saldamente il doppio cap a membrana. (**Attenzione:** l'etichetta del codice a barre deve essere rivolta verso la cuvetta indicatrice).
4. Chiudere ***immediatamente*** e saldamente la cuvetta di digestione con quella indicatrice. Mantenere le cuvetta doppia sempre in posizione verticale e ***NON*** agitare!
5. Riscaldare nel termostato (l'indicatore blu deve essere sempre rivolto verso l'alto).
   **a) HT 200 S:** ***2 h*** a ***95°C***
   **b) $LT^{100}_{200}$:** Nel termostato preriscaldato ***(100°C) 2 h*** a ***100°C***
   Successivamente lasciare raffreddare a temperatura ambiente.
6. Avvitare la combinazione di cuvette nuovamente prima di invertirla.
7. Capovolgere la combinazione di cuvette. Pulire bene la cuvetta con l'indicatore esternamente e leggere.

## NL

### 1. Monstervoorbereiding (uitdrijving van TIC)

1. ***1.0 mL*** monster in ontsluitingskuvet pipetteren.
2. Plaats het ***open*** ontsluitingskuvet in de **TOC-X5** triller, en duw de kuvet tot op de bodem. Plaats de ventilatorklep over de kuvetten. Zet de thermostaat aan. Na ***5 min*** weerklinkt een geluidssignaal.

### 2. TOC-bepaling

3. Wanneer de monstervoorbereiding voltooid is, op de blauwe indicatorkuvet ***onmiddellijk*** de dubbele membraandop stevig vast schroeven. (**Let op!:** Het etiket met de barcode moet naar de indicatorkuvet wijzen).
4. ***Sluit onmiddellijk*** de ontsluitingskuvet af met behulp van de voorbereide indicatorkuvet. Kuvettencombinatie beslist loodrecht houden en ***niet*** zwenken.
5. In het thermostaat verhitten (blauwe indicatorkuvet naar boven).
   **a) HT 200 S:** ***2 h*** bij ***95°C***
   **b) $LT^{100}_{200}$:** In de voorverwarmde thermostaat ***(100°C) 2 h*** bij ***100°C***
   Aansluitend laten afkoelen tot kamertemperatuur.
6. Zet de kuvettencombinatie weer ***vast*** alvorens ze om te draaien.
7. Draai de kuvettencombinatie om. Indicatorkuvet van buiten goed reinigen en meten.

## GB

### 1. Sample Preparation (expulsion of TIC)

1. Pipette ***1.0 mL*** sample into the digestion cuvette.
2. Insert the ***open*** digestion cuvette in the **TOC-X5** shaker, pushing it down as far as it will go. Position the fan cover over the cuvette. Switch on the instrument. After ***5 min*** an acoustic signal is emitted.

### 2. TOC determination

3. When the sample preparation is complete, open the blue indicator cuvette and ***immediately*** screw on the membrane double cap ***tightly***. (**NB:** The barcode label must point towards the indicator cuvette).
4. ***Immediately*** close the digestion cuvette ***tightly*** with the prepared indicator cuvette. Hold cuvette combination vertically. Do ***not*** invert.
5. Heat in the thermostat (blue indicator cuvette upwards).
   **a) HT 200 S:** ***2 h*** at ***95°C***
   **b) $LT^{100}_{200}$:** In the preheated thermostat ***(100°C) 2 h*** at ***100°C***
   Then allow to cool to room temperature.
6. ***Tighten*** the cuvette combination again ***before inverting*** it.
7. Invert cuvette combination, thoroughly clean the outside of the indicator cuvette and evaluate.

## *Auswertung / Evaluation / Lettura / Meting*

| | | | | | |
|---|---|---|---|---|---|
| IBR IBR+ | **Für folgende Barcode-Geräte erfolgt nach Einsetzen der Indikatorküvette eine automatische Auswertung:** | **Si vous utilisez un des instruments avec codes à barres suivants, une évaluation automatique est réalisée après l'insertion de la cuve indicatrice :** | **Se si utilizza uno qualsiasi dei seguenti strumenti con codice a barre, dopo aver inserito la cuvetta indicatrice viene automaticamente visualizzato il risultato della misura:** | **Wanneer een van de volgende barcode instrumenten worden gebruikt, wordt een automatische uitwaardering uitgevoerd zodra de indicatorkuvet geplaatst wordt:** | **If any of the following barcode instruments is used, an automatic evaluation is carried out after the indicator cuvette is inserted:** |

**LASA 50/100, XION 500, CADAS 30/50/30S/50S/200 Barcode, ISIS 9000, DR 2800/3800/3900/5000/6000**

| | D | F | I | NL | GB | | CADAS 200 Basis | ISIS 6000 | LASA 30 | DR 1900 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | **Filter** | **Filtre** | **Filtro** | **Filter** | **Filter** | 1 | -- | -- | 440 nm | -- |
| 2 | **Eprom** | **Eprom** | **Eprom** | **Eprom** | **Eprom** | 2 | _ : 50 | _ : 50 | -- | -- |
| 3 | **Mode** | **Mode** | **Mode** | **Mode** | **Mode** | 3 | 2) | 2) | Dr. Lange | 3) |
| 4 | **Test anwählen** | **Test choisir** | **Test selezionare** | **Test oproepen** | **Test select** | 4 | 386 | 386 | 386 | 386 |
| 5 | **Kontrollnr.** | **No. de contrôle** | **No. di controllo** | **Controlegetal** | **Control no.** | 5 | 3 | 3 | 3 | 3 |
| 6 | **Indikatorküvette der Küvetten-kombination einsetzen, grüne Taste / Messen** | **Introduire Cuve indicatrice des cuves combinées, touche verte / Mesurer** | **Usare cuvetta con l'indicatore (combinazione di cuvette), tasto verde / Lettura** | **Indicatorkuvet van de kuvetten-combinatie plaatsen, groene toets / Meten** | **Insert indicator cuvette of the cuvette combination, green key / Read** | 6 | ✔ | ✔ | ✔ | ✔ |

2) KÜVETTEN-TEST
2) TEST EN CUVE
2) CUVETTE-TEST
2) KUVETTENTEST
2) CUVETTE TEST

3) BARCODE-PROGRAMME
3) PROGR. CODE BARRE
3) PROGRAMMI COD. A BARRE
3) BARCODEPROGRAMMA'S
3) BARCODE PROGRAMS

REF 985 079

en

# Test 0-79 03.15
# NANOCOLOR® ortho- and total Phosphate 50

**Method:**
Photometric determination of the yellow phosphate-molybdate-vanadate complex after acidic hydrolysis and oxidation at 100–120 °C

| | | |
|---|---|---|
| Range: | **10.0–50.0 mg/L P ($PO_4$-P)** | **30–150 mg/L $PO_4^{3-}$** |
| Factor: | **027.5** | **0084.** |
| Wavelength (HW = 5–12 nm): | **436 nm** | |
| Decomposition: | **30 min** at 120 °C or **60 min** at 100 °C | |
| Reaction time: | **10 min (600 s)** at 20–25 °C | |

**Contents of reagent set:**
19 test tubes total Phosphate 50
1 tube *NANOFIX* total Phosphate 50 R2
2 test tubes with 11 mL total Phosphate 50 R3
1 test tube with blank value "NULL"

**Hazard warning:**
Test tubes contain sulfuric acid 5–15 %, reagent R2 contains sodium peroxodisulfate 20–100 %, reagent R3 contains sulfuric acid 15–30 %, blank value "NULL" contains sulfuric acid 5–15 %.
H314, H317, H334 Causes severe skin burns and eye damage. May cause an allergic skin reaction. May cause allergy or asthma symptoms or breathing difficulties if inhaled.
P260, P261, P272, P280, P301+330+331, P302+352, P303+361+353, P304+340, P305+351+338, P333+313, P342+311, P363, P501 Do not breathe vapors. Avoid breathing dust. Contaminated work clothing should not be allowed out of the workplace. Wear protective gloves / eye protection. IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN: Wash with plenty of water / … IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water / shower. IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. If skin irritation or rash occurs: Get medical advice / attention. If experiencing respiratory symptoms: Call a POISON CENTER / doctor / … Wash contaminated clothing before reuse. Dispose of contents / container to regulated waste treatment. For further information ask for a safety data sheet.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Phosphate (3–100 mg/L $PO_4^{3-}$, REF 913 20) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly.

**Interferences:**
If the ortho-phosphate content is higher than the total phosphorous content, destroyable colors (turbidities) falsely increase the ortho-phosphate reading.
Precipitations after hydrolysis can be removed by membrane filtration prior to the determination.
Silica < 1000 mg/L Si will not interfere.

The method ortho-P can be applied also for the analysis of sea water.

**Procedure:**
Requisite accessories: piston pipette with tips

**total Phosphorous**

Open test tube, add
**4.0 mL** test sample *(the pH value of the sample must be between pH 1 and 13)* and
**1** ***NANOFIX*** R2, screw cap back on to test tube, shake.
*(Close NANOFIX tube immediately after use.)*
Place tube in heating block and start heating block.
After 30 / 60 min remove test tube from heating block, shake once again and allow to cool down to room temperature.
Add
**1.0 mL** R3, mix.
Clean outside of test tube and measure after 10 min.

**ortho-Phosphate**

Filter sample solution.
Open test tube, add
**4.0 mL** test sample *(the pH value of the sample must be between pH 1 and 13)* and
**1.0 mL** R3, mix.
Clean outside of test tube and measure after 10 min.

***Note:***
*The concentration of condensed phosphates is the difference between total phosphorous* ***without*** *R2 and ortho-phosphate.*

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-79.

**Measurement when samples are colored or turbid:**
For all *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Multistandard Seepage water (REF 925 013)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
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PD 14122 / A011688 / 985 079 / 0352

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 049

## 5 – 90 mg/L $PO_4$ / 1.6 – 30.0 mg/L $PO_4$-P
## 3.7 – 70.0 mg/L $P_2O_5$

Lagerhinweis / Stabilité / Conservazione / Houdbaarheid / Storage: +15°C ..... +25°C

**T1**

**1000 mg/L:** $SO_4^{2-}$, $Cl^-$
**500 mg/L:** $K^+$, $Na^+$, $Ca^{2+}$
**50 mg/L:** $CO_3^{2-}$, $NO_3^-$, $Fe^{2+}$, $Fe^{3+}$, $Zn^{2+}$, $Cu^{2+}$, $Ni^{2+}$, $Cr^{3+}$
**5 mg/L:** $Pb^{2+}$

### *Datatabel / Data table*

| ***LP2W*** | ***06/1990*** |
|---|---|
| ***$PO_4$*** • *F1 = 0 • F2 = 92 • K = 0* | |
| ***$PO_4$-P*** • *F1 = 0 • F2 = 30 • K = 0* | |
| ***CADAS 30/30S/50/50S*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 435 nm • Pro.: 1 • F1 = 0 • F2 = 89 • K = -5.77* | |
| ***$PO_4$-P*** • *λ: 435 nm • Pro.: 1 • F1 = 0 • F2 = 29.2 • K = -1.94* | |
| ***$P_2O_5$*** • *λ: 435 nm • Pro.: 1 • F1 = 0 • F2 = 66.5 • K = -4.33* | |
| ***ISIS 6000/9000*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 405 nm • Pro.: 1 • F1 = 0 • F2 = 46.55 • K = -2.179* | |
| ***$PO_4$-P*** • *λ: 405 nm • Pro.: 1 • F1 = 0 • F2 = 15.18 • K = -0.709* | |
| ***$P_2O_5$*** • *λ: 405 nm • Pro.: 1 • F1 = 0 • F2 = 34.79 • K = -1.627* | |
| ***CADAS 100 / LPG 158*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 435 nm • F = 86.0* | |
| ***$PO_4$-P*** • *λ: 435 nm • F = 28.1* | |
| ***$P_2O_5$*** • *λ: 435 nm • F = 64.27* | |
| ***CADAS 100 / LPG 210*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 435 nm • F1 = 86.0* | |
| ***$PO_4$-P*** • *λ: 435 nm • F1 = 28.1* | |
| ***$P_2O_5$*** • *λ: 435 nm • F1 = 64.27* | |

**NL**

### LCK 049 Orthofosfaat

***Let a.u.b. op de "Uitgave datum" (zie datatabel). Veiligheidsadvies en houdbaarheidsdatum op de verpakking.***

**Principe**
Fosfaat-ionen geven met een vanadaat-molybdaatreagens een geel complex.

**Toepassingsgebied**
Water, bodenonderzoek, meststoffen, voeder en procesanalyse

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.

De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaard-additie).

**Speciale aandachtspunten**
Voor de bepaling van fosfaat totaal moet de kuvettentest LCK 348, LCK 349 of LCK 350 worden toegepast.

**pH-waarde** monster .....3 – 10
**Temperaturen** monster/reagentia.....15 – 25°C

**GB**

### LCK 049 Orthophosphate

***Please check the "Edition Date" (see data table). Safety advice and expiry date on package.***

**Principle**
Phosphate ions react with vanadate-molybdate reagent to form a yellow dye.

**Range of Application**
Water, soil analysis, fertilizers, animal feed, process analysis

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.

The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**For Special Attention**
Cuvette Tests LCK 348, LCK 349 or LCK 350 must be used for the determination of total phosphorus.

**pH** sample .....3 – 10
**Temperature** sample/reagents .....15 – 25°C

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 049

**5 – 90 mg/L $PO_4$ / 1.6 – 30.0 mg/L $PO_4$-P**
**3.7 – 70.0 mg/L $P_2O_5$**

*Lagerhinweis*
*Stabilité*
*Conservazione*
*Houdbaarheid*
*Storage*
+15°C ..... +25°C

**T1**

**1000 mg/L:** $SO_4^{2-}$, $Cl^-$

**500 mg/L:** $K^+$, $Na^+$, $Ca^{2+}$

**50 mg/L:** $CO_3^{2-}$, $NO_3^-$, $Fe^{2+}$, $Fe^{3+}$, $Zn^{2+}$, $Cu^{2+}$, $Ni^{2+}$, $Cr^{3+}$

**5 mg/L:** $Pb^{2+}$

## *Datentabelle / Table des données / Tabella dati*

| ***LP2W*** | ***06/1990*** |
|---|---|
| ***$PO_4$*** • *F1 = 0 • F2 = 92 • K = 0* | |
| ***$PO_4$-P*** • *F1 = 0 • F2 = 30 • K = 0* | |
| ***CADAS 30/30S/50/50S*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 435 nm • Pro.: 1 • F1 = 0 • F2 = 89 • K = -5.77* | |
| ***$PO_4$-P*** • *λ: 435 nm • Pro.: 1 • F1 = 0 • F2 = 29.2 • K = -1.94* | |
| ***$P_2O_5$*** • *λ: 435 nm • Pro.: 1 • F1 = 0 • F2 = 66.5 • K = -4.33* | |
| ***ISIS 6000/9000*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 405 nm • Pro.: 1 • F1 = 0 • F2 = 46.55 • K = -2.179* | |
| ***$PO_4$-P*** • *λ: 405 nm • Pro.: 1 • F1 = 0 • F2 = 15.18 • K = -0.709* | |
| ***$P_2O_5$*** • *λ: 405 nm • Pro.: 1 • F1 = 0 • F2 = 34.79 • K = -1.627* | |
| ***CADAS 100 / LPG 158*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 435 nm • F = 86.0* | |
| ***$PO_4$-P*** • *λ: 435 nm • F = 28.1* | |
| ***$P_2O_5$*** • *λ: 435 nm • F = 64.27* | |
| ***CADAS 100 / LPG 210*** | ***06/1990*** |
| ***$PO_4$*** • *λ: 435 nm • F1 = 86.0* | |
| ***$PO_4$-P*** • *λ: 435 nm • F1 = 28.1* | |
| ***$P_2O_5$*** • *λ: 435 nm • F1 = 64.27* | |

**D**

## LCK 049 Ortho-Phosphat

***Bitte "Ausgabedatum" (s. Datentabelle) beachten.***
***Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Phosphationen bilden mit Vanadat-Molybdat-Reagenz einen gelben Farbkomplex.

**Anwendungsbereich**
Wasser, Bodenuntersuchungen, Düngemittel, Futtermittel und Prozessanalytik

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.

Messergebnisse sind durch eine Plausibilitäts-kontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**Besonders beachten**
Zur Bestimmung von Gesamt-Phosphat muss mit dem Küvetten-Test® LCK 348, LCK 349 oder LCK 350 gearbeitet werden.

**pH-Wert** Probe .....3 – 10
**Temperatur** Probe/Reagenzien .....15 – 25°C

**F**

## LCK 049 Orthophosphate

***Vérifier la date d'edition (voir table des données).***
***Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Les ions phosphate réagissent avec le réactif vanadate-molybdate et donnent une coloration jaune.

**Domaine d'application**
Eaux, analyses des sols, engrais, fourrages et analyses en mode continu

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.

Les résultats de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**Remarque importante**
Pour la détermination du phosphate total, utilisez les Tests en Cuve LCK 348 ou LCK 349 ou LCK 350.

**pH** échantillon.....3 – 10
**Température** échantillon/réactifs .....15 – 25°C

**I**

## LCK 049 Orto-fosfati

***Si prega di verificare la "Data di Edizione" (vedi tabella dati).***
***Avvertenze e data di scadenza sulla confezione.***

**Principio**
Ioni fosfato formano con il reattivo di vanadato-molibdato una colorazione gialla.

**Applicazione**
Acqua e terreni, fertilizzanti, mangimi, analisi di processo

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.

I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**Pro memoria**
La determinazione dei fosfati totali si effettua con il cuvette-test LCK 348, LCK 349 opp. LCK 350.

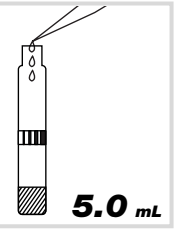
**pH** campione .....3 – 10
**Temperatura** campione/reagenti .....15 – 25°C

AD 049 J / Druckfarbe burgund / 1

**LCK 049** *06/1990*

**$PO_4$ / $PO_4$-P / $P_2O_5$**

❶




***5.0 mL*** Probe pipettieren.

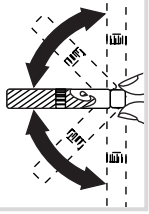
Pipetter ***5.0 mL*** d'échantillon.

Pipettare ***5.0 mL*** di campione.

***5.0 mL*** monster pipetteren.

Pipette ***5.0 mL*** sample.

❷



Küvette verschließen und schwenken.

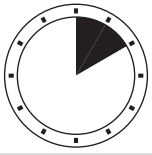
Fermer la cuve et mélanger le contenu en la retournant plusieurs fois de suite.

Tappare la cuvetta e mescolare.

Kuvet sluiten en zwenken.

Close cuvette and invert a few times.

❸ ***10 min***



Nach ***10 min*** Küvette noch einmal schwenken, außen gut säubern und auswerten.

Attendre ***10 min***, melanger de nouveau, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***10 min***, mescolare nuovamente, pulire bene la cuvetta esternamente e leggere.

Na ***10 min*** het kuvet opnieuw zwenken, van buiten goed reinigen en meten.

After ***10 min***, invert a few times more, thoroughly clean the outside of the cuvette and evaluate.

## *Auswertung / Evaluation / Lettura / Meting*

| | Analysenküvette ❶<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|
| IBR / IBR+ Barcode [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Test ❸<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Analysenküvette ❹<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|
| **LASA aqua** | △ 049 / ○ 049 P | _ : 12 | ***$PO_4$:*** △ 049 ***/ $PO_4$-P:*** ○ 049 P | ✔ |
| **LASA 1 / plus** | 440 nm | _ : 18 | PO4 / PO4-P / P2O5 LCK 049 | ✔ |
| **LASA 20** | -- | _ : 32 | PO4 / PO4-P LCK 049 | ✔ |

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Test ❷<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Faktor ❸<br>Facteur<br>Fattore<br>Factor<br>Factor | KontrollNr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (Probe) ❺<br>Valeur à blanc (échantillon)<br>Bianco (campione)<br>Blanko (monster)<br>Blank-value (sample) Null | Analysenküvette ❻<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette Ergebnis |
|---|---|---|---|---|---|---|
| **LP1W** | 435 nm | -- | ***$PO_4$:*** 92 ***/ $PO_4$-P:*** 30 ***/ $P_2O_5$:*** 68.75 | -- | LCW 919 | ✔ |
| **LP2W** | 435 nm | PO4 / PO4-P LCK 049 | -- | ***$PO_4$:*** 2 ***/ $PO_4$-P:*** 3 | LCW 919 | ✔ |

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Mode ❸ | Test ❹<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | KontrollNr. ❺<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette, grüne Taste / Messen ❻<br>Cuve d'analyse, touche verte / Mesurer<br>Cuvetta d'analisi, tasto verde / Lettura<br>Analyse-kuvet, groene toets / Meten<br>Sample cuvette, green key / Read |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 38 | -- | 049 | 5 | ✔ |
| **ISIS 6000** | -- | _ : 32 | [2] | 049 | 5 | ✔ |
| **LASA 30** | 440 nm | -- | Dr. Lange | 049 | 5 | ✔ |
| **DR 1900** | -- | -- | [3] | 049 | 5 | ✔ |

[2] KÜVETTEN-TEST
[2] TEST EN CUVE
[2] CUVETTE-TEST
[2] KUVETTENTEST
[2] CUVETTE TEST

[3] BARCODE-PROGRAMME
[3] PROGR. CODE BARRE
[3] PROGRAMMI COD. A BARRE
[3] BARCODEPROGRAMMA'S
[3] BARCODE PROGRAMS

| | Mode ❶ | Symbol ❷<br>Symbole<br>Simbolo<br>Symbool<br>Symbol | KontrollNr. ❸<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (Probe) ❹ NULL<br>Valeur à blanc (échantillon)<br>Bianco (campione)<br>Blanko (monster)<br>Blank-value (sample) | Analysenküvette ❺ MESS<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | ***$PO_4$:*** 049 ***/ $PO_4$-P:*** 049 E ***/ $P_2O_5$:*** 049 P | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | ***$PO_4$:*** 049 ***/ $PO_4$-P:*** 049 E ***/ $P_2O_5$:*** 049 P | ***$PO_4$:*** 9 ***/ $PO_4$-P:*** 6 ***/ $P_2O_5$:*** 5 | LCW 919 | ✔ |

**REF 985 088**

en

# Test 0-88 02.15

# *NANOCOLOR*® total Nitrogen $TN_b$ 220

**Method:**
Oxidative decomposition in the heating block with subsequent interference compensation and photometric determination with 2,6-dimethylphenol in sulfuric acid / phosphoric acid mixture

| | | |
|---|---|---|
| Range: | **5–220 mg/L N** | **5–220 mg/L N** |
| Factor: | **0319.–0400.** | **0190.** |
| Wavelength (HW = 5–17 nm): | **385 nm** | **365 nm** |
| Decomposition: | **30 min** at 120 °C / **60 min** at 100 °C | |
| Reaction time: | **10 min (600 s)** at 20–25 °C | |

**Content of reagent set:**

**Box A:** 20 decomposition tubes A
4 g decomposition reagent
1 tube *NANOFIX* compensation reagent
1 measuring spoon 85 mm orange

**Box B:** 20 test tubes total Nitrogen $TN_b$ 220
1 test tube with 11 mL $NO_3$ / N R2

**Hazard warning:**
The decomposition reagent contains potassium peroxodisulfate 20–100 % and sodium carbonate 20–100 %, the compensation reagent contains sodium sulfite 20–100 %, test tubes contain sulfuric acid 51–80 % and phosphoric acid 25–50 %, reagent R2 contains 2-propanol 20–50 %.
H314, H317, H334 Causes severe skin burns and eye damage. May cause an allergic skin reaction. May cause allergy or asthma symptoms or breathing difficulties if inhaled.
P260, P261, P272, P280, P301+330+331, P302+352, P303+361+353, P304+340, P305+351+338, P333+313, P342+311, P363, P501 Do not breathe vapors. Avoid breathing dust. Contaminated work clothing should not be allowed out of the workplace. Wear protective gloves / eye protection. IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN: Wash with plenty of water / … IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water / shower. IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. If skin irritation or rash occurs: Get medical advice / attention. If experiencing respiratory symptoms: Call a POISON CENTER / doctor / … Wash contaminated clothing before reuse. Dispose of contents / container to regulated waste treatment. For further information ask for a safety data sheet.

**Interferences:**
The following ions will not interfere: < 10000 mg/L $Cl^-$.

The method cannot be applied for the analysis of sea water.

***Note:***
*The pH value of the sample to be decomposed must be between pH 5 and 9, if necessary adjust with sodium hydroxide solution or sulfuric acid. Nitrogen concentrations above the double measuring range can simulate results within the measuring range and thus cause a wrong evaluation. Dilute the sample until the measured value is within the measuring range. For waters of unknown concentrations we recommend that you perform the test with very different dilutions (e.g. 1+9, 1+99) until the last dilution confirms the previous value. For samples which consume large amounts of oxidizing substances (e.g. for COD values above 10000 mg/L $O_2$), decomposition can be incomplete. In such cases repeat the decomposition with a diluted sample solution.*

**Procedure:**
Requisite accessories: *NANOCOLOR*® heating block, piston pipette with tips

**A) Decomposition (Box A)**

Open **decomposition tube A**, add
**500 µL** test sample *(the pH value of the sample must be between pH 5 and 9)* and
**1 level spoon decomposition reagent**, close and shake vigorously.
Place decomposition tube into the heating block and heat at 120 °C for 30 min or at 100 °C for 1 h.
Remove tube from heating block, shake gently and leave it to cool.
Open decomposition tube again, add
**1 *NANOFIX* compensation reagent**, close and shake vigorously.
→ decomposed solution

**B) Analysis (Box B)**

Open **test tube total Nitrogen $TN_b$ 220**, add
**500 µL** decomposed solution and
**500 µL** R2, close and mix by shaking gently.
Clean outside of test tube and measure after 10 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-88.
For exact measurements in the low range, the determination should be performed against a decomposed blank solution (use distilled water instead of the test sample).

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* multistandard Sewage influx (REF 925 012)

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PD 14122 / A030451 / 985 088 / 0257

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 338 20 – 100 mg/L LATON

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+15°C ..... +25°C

T1

**2500 mg/L:** CZV / COD

**5000 mg/L:** $Cl^-$

## *Datatabel / Data table*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***07/2004*** |
| ***LCK 338 **** • *F1 = 0 • F2 = 134.2 • K = -10.27* | |
| ***CADAS 30/30S/50/50S*** | ***07/2004*** |
| ***LCK 338 **** • *λ: 345 nm • Pro.: 1 • F1 = 0 • F2 = 120.3 • K = -15.73* | |
| ***ISIS 6000/9000*** | ***07/2004*** |
| ***LCK 338 **** • *λ: 360 nm • Pro.: 1 • F1 = 0 • F2 = 141.0 • K = -15.67* | |
| ***CADAS 100 / LPG 158*** | ***07/2004*** |
| ***LCK 338 **** • *λ: 370 nm • F1 = 180.4 • F2 = -8.22* | |
| ***CADAS 100 / LPG 210*** | ***07/2004*** |
| ***LCK 338 **** • *λ: 370 nm • F1 = 180.4 • K = -8.22* | |
| ***CADAS 200*** | ***07/2004*** |
| ***LCK 338 **** • *E1W1 = E1*F1-F2 • W1 = 345 nm • F1 = 119.7 • F2 = 16.14* | |

*) $TN_b$

NL

## LCK 338 Totaal-stikstof, $TN_b$

***Let a.u.b. op de "Uitgave datum" (zie datatabel).***
***Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
Anorganisch en organisch gebonden stikstof wordt door een ontsluiting met peroxodisulfaat tot nitraat geoxydeerd. Nitraat reageert in een zwavel- en fosforzure oplossing met 2.6-dimethylphenol tot een nitrophenol.

**Toepassingsgebied**
Water en afvalwater

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.

Aanwezigheid van reductiemiddelen kunnen leiden tot lagere meetresultaten.
De meetresultaten zijn via een plausibiliteitsonder-zoek te controleren (verdunning en/of standaard-additie).

**Speciale aandachtspunten**
1. Natronloog A / Oxidatiemiddel tablet B / MicroCap C
   Na toevoeging van de reagentia A, B en C moeten deze weer ***direkt*** gesloten worden.
2. Reactieglazen
   De reactieglazen dienen niet meer dan ***13 keer*** te worden gebruikt. Na ieder gebruik zijn ze met behulp van spoelborstels en leidingwater grondig te reinigen, en aansluitend met stikstofvrij gedestilleerd water na te spoelen en te drogen.
3. Troebeling
   Geringe troebelingen, storen niet. Sterke troebelingen, na toevoeging van het MicroCap C laten bezinken of met LCW 904 membraan-filtratie-set filtreren.

**pH-waarde** monster .....3 – 12
**Temperaturen** monster/reagentia.....15 – 25°C

GB

## LCK 338 Total Nitrogen, $TN_b$

***Please check the "Edition Date" (see data table).***
***Safety advice and expiry date on package.***

**Principle**
Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2.6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol.

**Range of Application**
Water, waste water

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.

Low-bias results are to be expected if the samples contain large amounts of reducing agents.
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**Special note**
1. Sodium hydroxide solution A / Oxidant tablet B / MicroCap C
   After addition of reagents A, B and C the bottles must be reclosed ***immediately***.
2. Reaction Tubes
   The reaction tubes should not be used more than ***13 times***. After use, clean thoroughly with a brush and water from the tap, then rinse well with nitrogen-free distilled water and dry.
3. Turbidity
   Slight turbidities present do not interfere; stronger turbidities after addition of the MicroCap C should be allowed to settle or filtered off using Membrane Filtration Set LCW 904.

**pH** sample .....3 – 12
**Temperature** sample/reagents .....15 – 25°C

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 338 20 – 100 mg/L LATONN

Lagerhinweis / Stabilité / Conservazione / Houdbaarheid / Storage: +15°C ..... +25°C

T1

**2500 mg/L:** CSB / DCO / COD

**5000 mg/L:** $Cl^-$

## *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***07/2004*** |
| ***LCK 338 *)*** • *F1 = 0 • F2 = 134.2 • K = -10.27* | |
| ***CADAS 30/30S/50/50S*** | ***07/2004*** |
| ***LCK 338 *)*** • *λ: 345 nm • Pro.: 1 • F1 = 0 • F2 = 120.3 • K = -15.73* | |
| ***ISIS 6000/9000*** | ***07/2004*** |
| ***LCK 338 *)*** • *λ: 360 nm • Pro.: 1 • F1 = 0 • F2 = 141.0 • K = -15.67* | |
| ***CADAS 100 / LPG 158*** | ***07/2004*** |
| ***LCK 338 *)*** • *λ: 370 nm • F1 = 180.4 • F2 = -8.22* | |
| ***CADAS 100 / LPG 210*** | ***07/2004*** |
| ***LCK 338 *)*** • *λ: 370 nm • F1 = 180.4 • K = -8.22* | |
| ***CADAS 200*** | ***07/2004*** |
| ***LCK 338 *)*** • *E1W1 = E1*F1-F2 • W1 = 345 nm • F1 = 119.7 • F2 = 16.14* | |

*) $TN_b$
NT

D

## LCK 338 Gesamt-Stickstoff, $TN_b$

***Bitte "Ausgabedatum" (s. Datentabelle) beachten.***
***Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Anorganisch und organisch gebundener Stickstoff wird durch einen Aufschluss mit Peroxodisulfat zu Nitrat oxidiert. Die Nitrationen reagieren in schwefel- und phosphorsaurer Lösung mit 2.6-Dimethylphenol zu einem Nitrophenol.

**Anwendungsbereich**
Wasser und Abwasser

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.

Minderbefunde sind zu erwarten, sofern die Proben große Mengen an Reduktionsmitteln enthalten. Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**Besonders beachten**
1. Natronlauge A / Oxidationsmittel-Tablette B / MicroCap® C
   Nach Zugabe der Reagenzien A, B und C müssen die Flaschen ***sofort*** wieder verschlossen werden.
2. Reaktionsgläser
   Die Reaktionsgläser sollten nicht mehr als ***13 mal*** benutzt werden. Nach jedem Gebrauch sind sie unter Einsatz von Spülbürste und Leitungswasser gründlich zu reinigen, und anschließend mit stickstofffreiem dest. Wasser gut nachzuspülen und zu trocknen.
3. Trübung
   Vorhandene geringe Trübungen stören nicht, starke Trübungen nach Zugabe des MicroCap® C absetzen lassen oder mit LCW 904 Membran-Filtrations-Set abfiltrieren.

**pH-Wert** Probe .....3 – 12
**Temperatur** Probe/Reagenzien .....15 – 25°C

F

## LCK 338 Azote Total, NT

***Vérifier la date d'édition (voir table des données).***
***Conseils de securité et date de péremption sur l'emballage.***

**Principe**
L'azote de composition organique et inorganique s'oxyde en présence de péroxydisulfate et se transforme donc en nitrate. Les ions nitrates réagissent dans une solution d'acides sulfurique et phosphorique avec du diméthylphénol-2.6 en formant du nitrophénol.

**Domaine d'application**
L'eau et eaux de rejet

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.

On peut s'attendre à des résultats par défaut si les échantillons contiennent des grandes quantités de réducteurs.
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**Remarque importante**
1. Solution d'hydroxyde de sodium A / Tablette d'oxydant B / MicroCap C
   Après l'ajout des réactifs A, B et C, refermer les flacons ***immédiatement***.
2. Eprouvettes de réaction
   Ne pas utiliser les éprouvettes plus de ***13 fois***. Avant chaque usage, elles doivent être nettoyées à la brosse de rinçage et à l'eau de distribution, puis rincées soigneusement à l'eau distillée non azotée et séchées.
3. Turbidité
   De légers troubles n'ont pas d'effet perturbateur, en cas de turbidité pronnoncée après l'ajout du MicroCap C, laisser décanter ou filtrer à l'aide du set de filtration à membrane LCW 904.

**pH** échantillon .....3 – 12
**Température** échantillon/réactifs .....15 – 25°C

I

## LCK 338 Azoto totale, $TN_b$

***Si prega di verificare la "Data di Edizione" (vedi tabella dati).***
***Avvertenze e data di scadenza sulla confezione.***

**Principio**
L'azoto in associazione organica ed inorganica viene ossidato in nitrato dissociandolo col perossidisolfato. Gli ioni nitrato reagiscono in soluzione solforica e fosforica col 2.6-dimetilfenolo dando il nitrofenolo.

**Applicazione**
Acqua e acque di scarico

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.

Se il campione contiene riducenti in concentrazioni elevate, il risultato sarà minore.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**Pro memoria**
1. Idrato di sodio A / Agente ossidante in pastiglia B / MicroCap C
   Richiudere i flaconi ***immediatamente*** dopo aver prelevato i reagenti A, B e C.
2. Provettoni
   Si sconsiglia di utilizzare i provettoni più di ***13 volte***. Dopo l'uso, pulire bene con una spazzola e acqua del rubinetto, poi risciacquare accuratamente con acqua distillata priva di azoto e lasciare asciugare.
3. Torbidità
   Debole torbidità non disturba. In caso di forte torbidità dopo l'aggiunta del MicroCap C, fare depositare o procedere alla filtrazione a membrana (LCW 904).

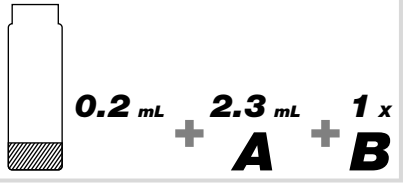
**pH** campione .....3 – 12
**Temperatura** campione/reagenti .....15 – 25°C

AD 338 P / Druckfarbe blau / 1

# LCK 338



❶




***Nacheinander zügig*** in ein trockenes Reaktionsglas dosieren:
***0.2 mL*** Probe, ***2.3 mL*** Lösung ***A*** (LCK 338 A), ***1 Tablette B*** (LCK138/238/338 B)
***Sofort*** verschließen. ***Nicht schwenken.***

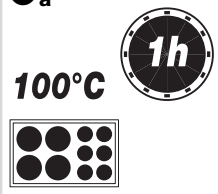
Doser ***à la suite***, ***consécutivement*** dans une éprouvette de réaction sèche:
***0.2 mL*** d'échantillon, ***2.3 mL*** de solution ***A*** (LCK 338 A), ***1 tablette B*** (LCK 138/238/338 B)
Fermer ***immédiatement***. ***Ne pas mélanger.***

Aggiungere in un provettone di reazione asciutto in ***rapida successione***:
***0.2 mL*** di campione, ***2.3 mL*** di soluzione ***A*** (LCK 338 A), ***1 pastiglia B*** (LCK 138/238/338 B)
Chiudere ***subito***. ***Non miscelare***.

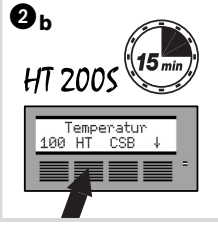
***Direct na elkaar*** in een droog reactieglas doseren:
***0.2 mL*** monster, ***2.3 mL*** oplossing ***A*** (LCK 338 A), ***1 tablet B*** (LCK 138/238/338 B)
***Onmiddellijk*** sluiten. ***Niet zwenken.***

Add in ***quick succession*** to a dry reaction tube:
***0.2 mL*** sample, ***2.3 mL*** solution ***A*** (LCK 338 A), ***1 tablet B*** (LCK 138/238/338 B)
Close ***immediately*** reaction tube. ***Do not invert.***

❷a




❷b




***Direkt*** erhitzen.
***a) Thermostat:*** ***60 min*** bei ***100°C***
***b) HT 200 S:*** ***15 min*** im Standardprogramm ***HT***

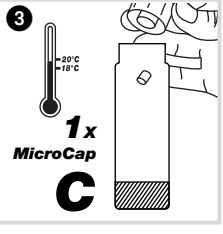
Chauffer ***directement***.
***a) Thermostat:*** ***60 min*** à ***100°C***
***b) HT 200 S:*** ***15 min*** avec le programme standard ***HT***

Riscaldare ***subito***.
***a) Termostato:*** ***60 min*** a ***100°C***
***b) HT 200 S:*** ***15 min*** nel programma standard ***HT***

***Direct*** verhitten.
***a) Thermostaat:*** ***60 min*** bij ***100°C***
***b) HT 200 S:*** ***15 min*** in standaard-programma ***HT***

Heat ***immediately***.
***a) Thermostat:*** ***60 min*** at ***100°C***
***b) HT 200 S:*** in standard program ***HT*** for ***15 min***

❸




Abkühlen und
***1 MicroCap® C***
(LCK138/238/338 C) zugeben.

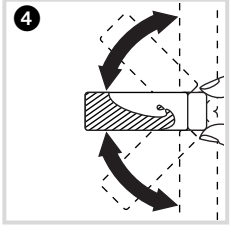
Refroidir et ajouter
***1 MicroCap C***
(LCK 138/238/338 C).

Raffreddare e aggiungere un
***1 MicroCap C***
(LCK 138/238/338 C).

Afkoelen en
***1 MicroCap C***
(LCK 138/238/338 C) toevoegen.

Cool down and add
***1 MicroCap C***
(LCK 138/238/338 C).

❹



Reaktionsglas verschließen und schwenken, bis das Lyophilisat ***vollständig*** und ***schlierenfrei*** aus dem MicroCap® C herausgelöst ist.

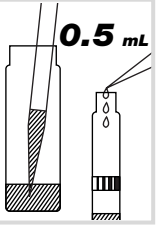
Fermer l'éprouvette de réaction et mélanger jusqu'à ce que le lyophilisat se soit ***complètement*** dissous du MicroCap C et qu'il ***n'y ait aucune particule restante***.

Chiudere il provettone e mescolare con cura finchè il liofilizzato contenuto nel MicroCap C si sia ***sciolto*** e miscelato ***perfettamente***, ***senza lasciare striature***.

Reactieglas sluiten en zwenken totdat het lyophilisaat ***volledig*** uit de MicroCap C opgelost is en ***homogeen*** verdeeld is.

Close reaction tube and invert a few times until the freezedried contents are ***fully removed*** from the MicroCap C and ***all streaks are vanished***.

❺




In Küvetten-Test ***langsam*** pipettieren:
***0.5 mL*** aufgeschlossene Probe.

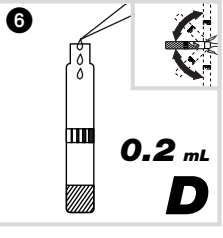
Pipetter ***lentement*** dans le Test en Cuve:
***0.5 mL*** d'échantillon désagrégé.

Pipettare ***con attenzione*** nella cuvetta-test:
***0.5 mL*** di campione preparato.

***Langzaam*** in kuvettentest pipetteren:
***0.5 mL*** ontsloten monster.

***Slowly*** pipette into the Cuvette Test:
***0.5 mL*** digested sample.

❻




***0.2 mL*** Lösung ***D*** (LCK 138/238/338 D) ***langsam*** pipettieren.
Küvette ***sofort*** verschließen und schwenken, bis ***keine*** Schlieren mehr zu beobachten sind.

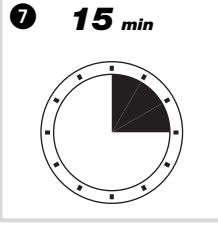
Pipetter ***lentement 0.2 mL*** de solution ***D*** (LCK 138/238/338 D). Fermer ***immédiatement*** la cuve et mélanger le contenu en la retournant plusieurs fois de suite jusqu'à qu'***aucun*** dépot ou agrégat ne soit observable.

Pipettare ***con attenzione 0.2 mL*** di soluzione ***D*** (LCK 138/238/338 D).
Tappare ***subito*** la cuvetta e mescolare fino a scioglimento completo ***(assenza di striature)***.

***Langzaam 0.2 mL*** oplossing ***D*** (LCK 138/238/338 D) pipetteren.
Kuvet ***onmiddellijk*** sluiten en zwenken totdat er ***geen*** slierten meer zichtbaar zijn.

***Slowly*** pipette ***0.2 mL*** solution ***D*** (LCK 138/238/338 D).
***Immediately*** close cuvette and invert a few times until ***no more*** streaks can be seen.

❼




Nach ***15 min*** Küvette außen gut säubern und auswerten.

Attendre ***15 min***, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***15 min*** pulire bene la cuvetta esternamente e leggere.

Na ***15 min*** het kuvet van buiten goed reinigen en meten.

After ***15 min*** thoroughly clean the outside of the cuvette and evaluate.

## *Auswertung / Evaluation / Lettura / Meting*

| | Analysenküvette ❶<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|
| IBR / IBR+ Barcode [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Test ❸<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Kontrollnr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|---|
| **LASA aqua** | ☐ 338 | _ : 50 | ☐ 338 | -- | ✔ |
| **LASA 1 / plus** | 330 nm | -- | TNb LCK 338 *) | 1 | ✔ |
| **LASA 10 / 20** | -- | _ : 50 | TNb LCK 338 *) | 1 | ✔ |

*) NT LCK 338

**LP1W**

❼ Vom Ergebnis abziehen: ***10.27 mg/L***
❼ Soustraire au résultat: ***10.27 mg/L***
❼ Sottrarre dal risultato: ***10.27 mg/L***
❼ Van het resultaat aftrekken: ***10.27 mg/L***
❼ Substract from the result: ***10.27 mg/L***

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Test ❷<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Faktor ❸<br>Facteur<br>Fattore<br>Factor<br>Factor | Kontrollnr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❺<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) Null | Analysenküvette ❻<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette Ergebnis | ❼ |
|---|---|---|---|---|---|---|---|
| **LP1W** | 340 nm / Nitrat 339 | -- | 134.2 | -- | LCW 919 | ✔ | ✔ |
| **LP2W** | 340 nm / Nitrat 339 | TNb LCK 338 *) | -- | 2 | LCW 919 | ✔ | -- |

*) NT LCK 338

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Mode ❸ | Test ❹<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Kontrollnr. ❺<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette, grüne Taste / Messen ❻<br>Cuve d'analyse, touche verte / Mesurer<br>Cuvetta d'analisi, tasto verde / Lettura<br>Analyse-kuvet, groene toets / Meten<br>Sample cuvette, green key / Read |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 50 | -- | 338 | 2 | ✔ |
| **ISIS 6000** | -- | _ : 50 | [2] | 338 | 2 | ✔ |
| **LASA 30** | 340 nm | -- | Dr. Lange | 338 | 2 | ✔ |
| **DR 1900** | -- | -- | [3] | 338 | 2 | ✔ |

[2] KÜVETTEN-TEST
[2] TEST EN CUVE
[2] CUVETTE-TEST
[2] KUVETTENTEST
[2] CUVETTE TEST

[3] BARCODE-PROGRAMME
[3] PROGR. CODE BARRE
[3] PROGRAMMI COD. A BARRE
[3] BARCODEPROGRAMMA'S
[3] BARCODE PROGRAMS

| | Mode ❶ | Symbol ❷<br>Symbole<br>Simbolo<br>Symbool<br>Symbol | Kontrollnr. ❸<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❹<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) NULL | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette MESS |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | $ 338 | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | 338 | 2 | LCW 919 | ✔ |

REF 985 006

en

# Test 0-06 05.14
# *NANOCOLOR*® Ammonium 200

**Method:**
Photometric determination as indophenol: At a pH value of about 12.6 ammonium reacts with hypochlorite and salicylate in the presence of sodium nitroprusside as catalyst to form a blue indophenol.

| | | |
|---|---|---|
| Range: | **30–160 mg/L $NH_4$-N** | **40–200 mg/L $NH_4^+$/$NH_3$** |
| Factor: | **0116.** | **0150./0142.** |
| Wavelength (HW = 5–12 nm): | **585 nm** | |
| Reaction time: | **15 min (900 s)** | |
| Reaction temperature: | **20–25 °C** | |

**Contents of reagent set:**
20 test tubes Ammonium 200
1 tube *NANOFIX* Ammonium 200 R2
1 test tube with blank value “NULL”

**Hazard warning:**
Reagent R2 contains sodium nitroprusside 5–33 % and dichloroisocyanuric acid sodium salt 10–20 %.
For further information ask for a safety data sheet.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Ammonium (10–400 mg/L $NH_4^+$, REF 913 15) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly.

**Interferences:**
The photometric analysis of water samples with own color or turbidity always requires determination of a correction value.

The method can be applied also for the analysis of sea water.

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**200 µL** (= 0.2 mL) test sample *(the pH value of the sample must be between pH 1 and 13)*
and
**1** ***NANOFIX*** R2, close and mix.
*(Close NANOFIX tube immediately after use.)*
Clean outside of test tube and measure after 15 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-06.

**Measurement when samples are colored or turbid:**
For all *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Multistandard Seepage water (REF 925 013)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***

PD 14122 / A011734 / 985 006 / 0544

REF 985 005

en

# Test 0-05 10.14
# *NANOCOLOR*® Ammonium 50

**Method:**
Photometric determination as indophenol: At a pH value of about 12.6 ammonium reacts with hypochlorite and salicylate in the presence of sodium nitroprusside as catalyst to form a blue indophenol.

| | | |
|---|---|---|
| Range: | **1.0–40.0 mg/L $NH_4$-N** | **1.0–50.0 mg/L $NH_4^+$/$NH_3$** |
| Factor: | **0026.** | **0033./0031.** |
| Wavelength (HW = 5–12 nm): | **690 nm** | |
| Reaction time: | **15 min (900 s)** | |
| Reaction temperature: | **20–25 °C** | |

**Contents of reagent set:**
20 test tubes Ammonium 50
1 tube *NANOFIX* Ammonium 50 R2
1 test tube with blank value “NULL”

**Hazard warning:**
Reagent R2 contains sodium nitroprusside 5–33 %.
For further information ask for a safety data sheet.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Ammonium (10–400 mg/L $NH_4^+$, REF 913 15) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly.

**Interferences:**
Good reproducibility is obtained in weakly polluted waters. High pollution causes errors and requires distillation prior to analysis.

The method can also be applied for the analysis of sea water.

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**0.2 mL** (= 200 µL) test sample *(the pH value of the sample must be between pH 1 and 10)* and
**1** ***NANOFIX*** R2, close and mix.
*(Close NANOFIX tube immediately after use.)*
Clean outside of test tube and measure after 15 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-05.

**Measurement when samples are colored or turbid:**
For all *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Multistandard Sewage influx (REF 925 012)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***
PD 14122 / A020703 / 985 005 / 10410

UNITED FOR WATER QUALITY

# LCK 302 47 – 130 mg/L $NH_4$-N / 60 – 167 mg/L $NH_4$

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+2°C ..... +8°C

**T1**

| | |
|---|---|
| **1000 mg/L:** | $Cl^-$, $SO_4^{2-}$ |
| **500 mg/L:** | $K^+$, $Na^+$, $Ca^{2+}$ |
| **50 mg/L:** | $CO_3^{2-}$, $NO_3^-$, $Fe^{3+}$, $Cr^{3+}$, $Cr^{6+}$, $Zn^{2+}$, $Cu^{2+}$, $Co^{2+}$, $Ni^{2+}$, $Hg^{2+}$ |
| **25 mg/L:** | $Fe^{2+}$ |
| **10 mg/L:** | $Sn^{2+}$ |
| **5 mg/L:** | $Pb^{2+}$ |
| **2 mg/L:** | $Ag^+$ |

## *Datatabel / Data table*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***08/2010*** |
| ***$NH_4$-N** • F1 = 0 • F2 = 133.5 • K = -17.30* | |
| ***$NH_4$** • F1 = 0 • F2 = 171.6 • K = -22.21* | |
| ***CADAS 30/30S/50/50S*** | ***08/2010*** |
| ***$NH_4$-N** • λ: 552 nm • Pro.: 1 • F1 = 0 • F2 = 152.7 • K = -21.06* | |
| ***$NH_4$** • λ: 552 nm • Pro.: 1 • F1 = 0 • F2 = 196.4 • K = -27.04* | |
| ***ISIS 6000/9000*** | ***08/2010*** |
| ***$NH_4$-N** • λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 49.82 • K = -7.694* | |
| ***$NH_4$** • λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 64.05 • K = -9.898* | |
| ***CADAS 100 / LPG 158*** | ***08/2010*** |
| ***$NH_4$-N** • λ: 550 nm • F1 = 161.8 • K = -17.30* | |
| ***$NH_4$** • λ: 550 nm • F1 = 208.0 • K = -22.22* | |
| ***CADAS 100 / LPG 210*** | ***08/2010*** |
| ***$NH_4$-N** • λ: 550 nm • F1 = 161.8 • K = -17.31* | |
| ***$NH_4$** • λ: 550 nm • F1 = 208.0 • K = -22.22* | |
| ***CADAS 200*** | ***08/2010*** |
| ***$NH_4$-N** • E1W1 • C1 = E1*F1-F2 •* | |
| *W1 = 550 nm • F1 = 162.7 • F2 = 22.68* | |
| ***$NH_4$** • E1W1 • C1 = E1*F1-F2 •* | |
| *W1 = 550 nm • F1 = 209.2 • F2 = 29.19* | |

**NL**

## LCK 302 Ammonium-Stikstof

***Let a.u.b. op de "Uitgave datum" (zie datatabel).***
***Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
Ammonium-ionen reageren bij een pH-waarde van 12.6 met hypochloriet-ionen en salicylaat-ionen in verbinding met natriumnitroprusside als katalysator en vormen zo de stof indofenol-blauw.

**Toepassingsgebied**
Influent, industrieel afvalwater

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.

Primaire aminen worden mee geregistreerd en geven een te hoog resultaat. Een hoeveelheid van 10000 maal de toegestane hoeveelheid ureum stoort niet. Alle reductiemiddelen storen en geven te lage resultaten.
***Een veel te grote hoeveelheid ammonium kan ertoe leiden dat een resultaat wordt aangegeven dat binnen het meetbereik ligt. Het verdient in dit geval aanbeveling, te verdunnen en een betrouwbaarheids-controle uit te voeren.***
De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaard-additie).

**pH-waarde** monster .....4 – 9
**Temperaturen** monster/analyse-kuvet .....20°C
***Afwijkende temperaturen beïnvloeden de nauwkeurigheid van het resultaat.***
Het monster dient zo snel mogelijk na de monstername te worden onderzocht.

**Afhankelijkheid van de tijd**
De eindextinctie is na een reactietijd van ***15 min*** gerealiseerd en blijft dan ***15 min lang constant***.

**Opmerking!**
***Verandering van de factoren in alle fotometers.***

**GB**

## LCK 302 Ammonium-Nitrogen

***Please check the "Edition Date" (see data table).***
***Safety advice and expiry date on package.***

**Principle**
Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitroprusside as a catalyst to form indophenol blue.

**Range of Application**
Waste water plant, industrial waste water

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.

Primary amines are also determined and cause high-bias results. A 10000-fold excess of urea does not interfere. All reducing agents interfere and cause low-bias results.
***A large excess of ammonium can cause result displays within the measuring range. It is advisable to carry out a plausibility check by making dilutions.***
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**pH** sample .....4 – 9
**Temperature** sample/sample cuvette.....20°C
***In case of not working at the right recommanded temperature an incorrect result may be obtained.***
The sample should be analysed as soon as possible after it has been taken.

**Time dependency**
The final absorbance is reached after a reaction time of ***15 min*** and then remains ***constant for a further 15 min***.

**Note**
***Change of factor for all types of photometers.***

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 302 47 – 130 mg/L $NH_4$-N / 60 – 167 mg/L $NH_4$

Lagerhinweis / Stabilité / Conservazione / Houdbaarheid / Storage: +2°C ..... +8°C

**T1**

| | |
|---|---|
| **1000 mg/L:** | $Cl^-$, $SO_4^{2-}$ |
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| **50 mg/L:** | $CO_3^{2-}$, $NO_3^-$, $Fe^{3+}$, $Cr^{3+}$, $Cr^{6+}$, $Zn^{2+}$, $Cu^{2+}$, $Co^{2+}$, $Ni^{2+}$, $Hg^{2+}$ |
| **25 mg/L:** | $Fe^{2+}$ |
| **10 mg/L:** | $Sn^{2+}$ |
| **5 mg/L:** | $Pb^{2+}$ |
| **2 mg/L:** | $Ag^+$ |

## *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
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| ***$NH_4$*** • *λ: 552 nm • Pro.: 1 • F1 = 0 • F2 = 196.4 • K = -27.04* | |
| ***ISIS 6000/9000*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 49.82 • K = -7.694* | |
| ***$NH_4$*** • *λ: 610 nm • Pro.: 1 • F1 = 0 • F2 = 64.05 • K = -9.898* | |
| ***CADAS 100 / LPG 158*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 550 nm • F1 = 161.8 • K = -17.30* | |
| ***$NH_4$*** • *λ: 550 nm • F1 = 208.0 • K = -22.22* | |
| ***CADAS 100 / LPG 210*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 550 nm • F1 = 161.8 • K = -17.31* | |
| ***$NH_4$*** • *λ: 550 nm • F1 = 208.0 • K = -22.22* | |
| ***CADAS 200*** | ***08/2010*** |
| ***$NH_4$-N*** • *E1W1 • C1 = E1*F1-F2 • W1 = 550 nm • F1 = 162.7 • F2 = 22.68* | |
| ***$NH_4$*** • *E1W1 • C1 = E1*F1-F2 • W1 = 550 nm • F1 = 209.2 • F2 = 29.19* | |

**D**

## LCK 302 Ammonium-Stickstoff

***Bitte "Ausgabedatum" (s. Datentabelle) beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Ammoniumionen reagieren bei pH 12.6 mit Hypochloritionen und Salicylationen in Gegenwart von Nitroprussid-Natrium als Katalysator zu Indophenolblau.

**Anwendungsbereich**
Kläranlagenzulauf, industrielles Abwasser

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.

Primäre Amine werden miterfasst und ergeben Mehrbefunde. Ein 10000facher Überschuss an Harnstoff stört nicht. Alle Reduktionsmittel stören und führen zu Minderbefunden.
***Ein hoher Überschuss an Ammonium kann zu Ergebnisanzeigen innerhalb des Messbereichs führen. Hier ist eine Plausibilitätskontrolle durch Verdünnen empfehlenswert.***
Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**pH-Wert** Probe .....4 – 9
**Temperatur** Probe/Analysenküvette .....20°C
***Abweichende Temperaturen beeinflussen die Ergebnisrichtigkeit.***
Die Probe sollte sobald wie möglich nach der Probenahme untersucht werden.

**Zeitabhängigkeit**
Die Endextinktion liegt nach einer Reaktionszeit von ***15 min*** vor und bleibt dann ***15 min konstant***.

**Hinweis**
***Faktoränderung bei allen Photometertypen.***

**F**

## LCK 302 Azote ammoniacal

***Vérifier la date d'edition (voir table des données). Conseils de securité et date de péremption sur l'emballage.***

**Principe**
En présence de sodium nitroprussique agissant comme catalyseur et à une valeur du pH d'environ 12.6, les ions ammonium réagissent avec les ions hypochloreux et salicyliques et donnent une coloration bleue indophénol.

**Domaine d'application**
Eaux de rejets industriels, entrée des stations d'épuration

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.

Les amines primaires sont aussi déterminées et sont donc à l'origine des résultats trop élevés. Un excédent 10000 fois plus élevé en urée ne gêne pas l'évaluation. Tous les réducteurs gênent et donnent des résultats trop faibles.
***Malgré un excédent important d'ammonium, l'appareil peut tout de même afficher un résultat d'analyse compris dans la gamme de mesure. Pour éliminer une telle erreur, il est recommandé ici de vérifier le résultat obtenu en effectuant une nouvelle analyse après avoir dilué l'échantillon (contrôle de plausibilité).***
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**pH** échantillon .....4 – 9
**Température** échantillon/cuve d'analyse .....20°C
***Des températures différentes influencent l'exactitude des résultats.***
L'analyse doit être réalisée immédiatement après la prise d'échantillon.

**Importance du temps**
L'extinction finale apparaît après un temps de réaction de ***15 min*** et reste ***constante*** pendant ***15 min***.

**Remarque**
***Modification de facteur pour tous les types de photomètres.***

**I**

## LCK 302 Ammonio/Azoto ammoniacale

***Si prega di verificare la "Data di Edizione" (vedi tabella dati). Avvertenze e data di scadenza sulla confezione.***

**Principio**
Ioni ammonio reagiscono a un pH 12.6 con ioni di ipoclorito e di salicilato, in presenza di nitroprussiato sodico quale catalizzatore, dando il blu indofenolo.

**Applicazione**
Ingresso depuratore, scarichi industriali

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.

Le ammine primarie possono reagire dando valori più elevati. Un contenuto di urea 10000 volte più elevato non interferisce. Tutte le sostanze riducenti interferiscono e danno valori minori.
***Concentrazioni molto elevate di ammonio rischiano di dare risultati che rientrano nel campo di misura. Verificare diluendo il campione.***
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**pH** campione .....4 – 9
**Temperatura** campione/cuvetta d'analisi .....20°C
***Variazioni della temperatura influenzano la correttezza del valore misurato.***
Fare l'analisi subito dopo aver prelevato in campione!

**Tempo**
Il valore definitivo dell'estinzione si ottiene dopo ***15 min*** di reazione; il valore rimane ***costante per 15 min***.

**Note**
***Variazione del fattore su tutti i fotometri.***

AD 302 N / Druckfarbe blau / 1

**LCK 302** 02/2000

**$NH_4$-N / $NH_4$**

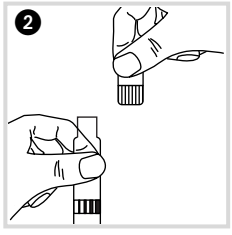

Siegelfolie von dem aufgeschraubten **DosiCap® Zip *vorsichtig*** abziehen.

Enlevez ***délicatement*** la feuille de protection du **DosiCap Zip** détachable.

Rimuovere ***con attenzione*** il foglio di alluminio.

Afdekfolie ***voorzichtig*** verwijderen.

***Carefully*** remove the foil from the screwed-on **DosiCap Zip**.



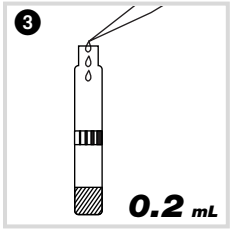

**DosiCap® Zip** abschrauben.

Dévissez le **DosiCap Zip**.

Svitare il **DosiCap Zip**.

**DosiCap Zip** afschroeven.

Unscrew the **DosiCap Zip**.



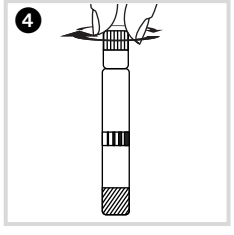

***0.2 mL*** Probe pipettieren.

Pipetter ***0.2 mL*** d'échantillon.

Pipettare ***0.2 mL*** di campione.

***0.2 mL*** monster pipetteren.

Pipette ***0.2 mL*** sample.



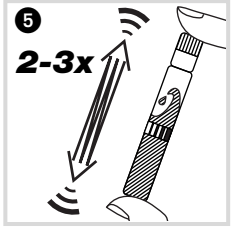

***Sofort*** **DosiCap® Zip** aufschrauben; Riffelung oben.

Vissez ***immédiatement*** le **DosiCap Zip**; dirigeant le cannelage vers le haut.

Avvitare ***subito*** il **DosiCap Zip**; scanalatura esterna verso l'alto.

***Onmiddellijk*** **DosiCap Zip** opschroeven; geribbelde zijde naar boven.

***Immediately*** screw the **DosiCap Zip** back; fluting at the top.



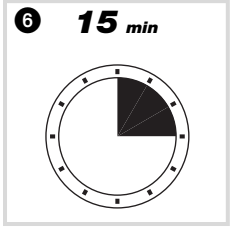

Kräftig schütteln.

Secouer énergiquement.

Agitare energicamente.

Krachtig schudden.

Shake firmly.




Nach ***15 min*** Küvette außen gut säubern und auswerten.

Attendre ***15 min***, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***15 min*** pulire bene la cuvetta esternamente e leggere.

Na ***15 min*** het kuvet van buiten goed reinigen en meten.

After ***15 min*** thoroughly clean the outside of the cuvette and evaluate.

## *Auswertung / Evaluation / Lettura / Meting*

| | Analysenküvette ❶<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|
| IBR / IBR+ Barcode [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

**LASA aqua / LASA 1 / plus**

❻ Zum Ergebnis addieren:
$NH_4$-N: ***2.17 mg/L*** /
$NH_4$: ***2.7 mg/L***

❻ Additionner au résultat:
$NH_4$-N: ***2.17 mg/L*** /
$NH_4$: ***2.7 mg/L***

❻ Addizionare al risultato:
$NH_4$-N: ***2.17 mg/L*** /
$NH_4$: ***2.7 mg/L***

❻ Bij het resultaat optellen:
$NH_4$-N: ***2.17 mg/L*** /
$NH_4$: ***2.7 mg/L***

❻ Add to the result:
$NH_4$-N: ***2.17 mg/L*** /
$NH_4$: ***2.7 mg/L***

**LASA 10 / 20**

❻ Zum Ergebnis addieren:
$NH_4$-N: ***4.88 mg/L*** /
$NH_4$: ***5.92 mg/L***

❻ Additionner au résultat:
$NH_4$-N: ***4.88 mg/L*** /
$NH_4$: ***5.92 mg/L***

❻ Addizionare al risultato:
$NH_4$-N: ***4.88 mg/L*** /
$NH_4$: ***5.92 mg/L***

❻ Bij het resultaat optellen:
$NH_4$-N: ***4.88 mg/L*** /
$NH_4$: ***5.92 mg/L***

❻ Add to the result:
$NH_4$-N: ***4.88 mg/L*** /
$NH_4$: ***5.92 mg/L***

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Test ❸<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Kontrollnr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|---|
| **LASA aqua** | ○ 302 N | _ : 28 | ***$NH_4$-N:*** ○ 302 N | -- | ✔ |
| **LASA 1 / plus** | 560 nm | _ : 28 | NH4-N / NH4 LCK 302 | 4 | ✔ |
| **LASA 10 / 20** | -- | _ : 30 **/** _ : 32 | NH4-N / NH4 LCK 302 | 4 | ✔ |

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Test ❷<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Faktor ❸<br>Facteur<br>Fattore<br>Factor<br>Factor | Kontrollnr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❺<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) | Analysenküvette ❻<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|---|---|
| **LP1W** | 550 nm | -- | ***$NH_4$-N:*** 133.4 ***/ $NH_4$:*** 171.6 | -- | LCW 919 | ✔ |
| **LP2W** | 550 nm | NH4-N / NH4 LCK 302 | -- | 4 | LCW 919 | ✔ |

**LP1W**

❼ Vom Ergebnis abziehen:
$NH_4$-N: ***22.21 mg/L*** /
$NH_4$: ***17.30 mg/L***

❼ Soustraire au résultat:
$NH_4$-N: ***22.21 mg/L*** /
$NH_4$: ***17.30 mg/L***

❼ Sottrarre dal risultato:
$NH_4$-N: ***22.21 mg/L*** /
$NH_4$: ***17.30 mg/Ll***

❼ Van het resultaat aftrekken:
$NH_4$-N: ***22.21 mg/L*** /
$NH_4$: ***17.30 mg/L***

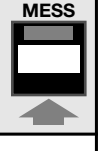
❼ Substract from the result
$NH_4$-N: ***22.21 mg/L*** /
$NH_4$: ***17.30 mg/L***

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Mode ❸ | Test ❹<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Kontrollnr. ❺<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette, grüne Taste / Messen ❻<br>Cuve d'analyse, touche verte / Mesurer<br>Cuvetta d'analisi, tasto verde / Lettura<br>Analyse-kuvet, groene toets / Meten<br>Sample cuvette, green key / Read |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 38 | -- | 302 | 4 | ✔ |
| **ISIS 6000** | -- | _ : 32 | [2] | 302 | 4 | ✔ |
| **LASA 30** | 535 nm | -- | Dr. Lange | 302 | 4 | ✔ |
| **DR 1900** | -- | -- | [3] | 302 | 4 | ✔ |

[2] KÜVETTEN-TEST
[2] TEST EN CUVE
[2] CUVETTE-TEST
[2] KUVETTENTEST
[2] CUVETTE TEST

[3] BARCODE-PROGRAMME
[3] PROGR. CODE BARRE
[3] PROGRAMMI COD. A BARRE
[3] BARCODEPROGRAMMA'S
[3] BARCODE PROGRAMS

| | Mode ❶ | Symbol ❷<br>Symbole<br>Simbolo<br>Symbool<br>Symbol | Kontrollnr. ❸<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❹<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | ***$NH_4$-N:*** $ 302 N ***/ $NH_4$:*** $ 302 | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | ***$NH_4$-N:*** 302 N ***/ $NH_4$:*** 302 | 4 | LCW 919 | ✔ |

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 303

## 2 – 47 mg/L $NH_4$-N / 2.5 – 60.0 mg/L $NH_4$

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+2°C ..... +8°C

**T1**

| | |
|---|---|
| **1000 mg/L:** | $Cl^-$, $SO_4^{2-}$ |
| **500 mg/L:** | $K^+$, $Na^+$, $Ca^{2+}$ |
| **50 mg/L:** | $CO_3^{2-}$, $NO_3^-$, $Fe^{3+}$, $Cr^{3+}$, $Cr^{6+}$, $Zn^{2+}$, $Cu^{2+}$, $Co^{2+}$, $Ni^{2+}$, $Hg^{2+}$ |
| **25 mg/L:** | $Fe^{2+}$ |
| **10 mg/L:** | $Sn^{2+}$ |
| **5 mg/L:** | $Pb^{2+}$ |
| **2 mg/L:** | $Ag^+$ |

### *Datatabel / Data table*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***08/2010*** |
| ***$NH_4$-N*** • *F1 = 0 • F2 = 23.01 • K = -0.84* | |
| ***$NH_4$*** • *F1 = 0 • F2 = 29.58 • K = -1.083* | |
| ***CADAS 30/30S/50/50S*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 690 nm • Pro.: 1 • F1 = 0 • F2 = 22.46 • K = -1.445* | |
| ***$NH_4$*** • *λ: 690 nm • Pro.: 1 • F1 = 0 • F2 = 28.88 • K = -1.856* | |
| ***ISIS 6000/9000*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 695 nm • Pro.: 1 • F1 = 0 • F2 = 22.98 • K = -1.865* | |
| ***$NH_4$*** • *λ: 695 nm • Pro.: 1 • F1 = 0 • F2 = 29.54 • K = -2.397* | |
| ***CADAS 100 / LPG 158*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 694 nm • F1 = 22.48 • F2 = -0.721* | |
| ***$NH_4$*** • *λ: 694 nm • F1 = 28.84 • F2 = -0.931* | |
| ***CADAS 100 / LPG 210*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 694 nm • F1 = 22.48 • F2 = -0.721* | |
| ***$NH_4$*** • *λ: 694 nm • F1 = 28.84 • F2 = -0.931* | |
| ***CADAS 200*** | ***08/2010*** |
| ***$NH_4$-N*** • *E1W1 • C1 = E1*F1-F2 •* *W1 = 694 nm • F1 = 22.48 • F2 = 1.465* | |
| ***$NH_4$*** • *E1W1 • C1 = E1*F1-F2 •* *W1 = 694 nm • F1 = 28.91 • F2 = 1.884* | |

**NL**

### LCK 303 Ammonium-Stikstof

***Let a.u.b. op de "Uitgave datum" (zie datatabel) en lees de "Opmerking". Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
Ammonium-ionen reageren bij een pH-waarde van 12.6 met hypo-chloriet-ionen en salicylaat-ionen in verbinding met natriumnitro-prusside als katalysator en vormen zo de stof indofenol-blauw.

**Toepassingsgebied**
Oppervlaktewateren, afvalwater, bodem, substraat

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.

Primaire aminen worden mee geregistreerd en geven een te hoog resultaat. Een hoeveelheid van 10000 maal de toegestane hoeveelheid ureum stoort niet. Alle reductiemiddelen storen en geven te lage resultaten.
***Een veel te grote hoeveelheid ammonium kan ertoe leiden dat een resultaat wordt aangegeven dat binnen het meetbereik ligt. Het verdient in dit geval aanbeveling, te verdunnen en een betrouwbaarheids-controle uit te voeren.***
De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaard-additie).

**pH-waarde** monster .....4 – 9
**Temperaturen** monster/analyse-kuvet .....20°C
***Afwijkende temperatuuren beïnvloeden de nauwkeurigheid van het resultaat.***
Het monster dient zo snel mogelijk na de monstername te worden onderzocht.

**Afhankelijkheid van de tijd**
De eindextinctie is na een reactietijd van ***15 min*** gerealiseerd en blijft dan ***15 min lang constant***.

**Opmerking!**
***Verandering van de factoren in alle fotometers.***

**GB**

### LCK 303 Ammonium-Nitrogen

***Please check the "Edition Date" (see data table) and read the "Note". Safety advice and expiry date on package.***

**Principle**
Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitroprusside as a catalyst to form indophenol blue.

**Range of Application**
Surface water, waste water, soils, substrates

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.

Primary amines are also determined and cause high-bias results. A 10000-fold excess of urea does not interfere. All reducing agents interfere and cause low-bias results.
***A large excess of ammonium can cause result displays within the measuring range. It is advisable to carry out a plausibility check by making dilutions.***
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**pH** sample .....4 – 9
**Temperature** sample/sample cuvette.....20°C
***In case of not working at the right recommanded temperature an incorrect result may be obtained.***
The sample should be analysed as soon as possible after it has been taken.

**Time dependency**
The final absorbance is reached after a reaction time of ***15 min*** and then remains ***constant for a further 15 min***.

**Note**
***Change of factor for all types of photometers.***

UNITED FOR WATER QUALITY

# LCK 303

## 2 – 47 mg/L $NH_4$-N / 2.5 – 60.0 mg/L $NH_4$

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+2°C ..... +8°C

**T1**

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| **25 mg/L:** | $Fe^{2+}$ |
| **10 mg/L:** | $Sn^{2+}$ |
| **5 mg/L:** | $Pb^{2+}$ |
| **2 mg/L:** | $Ag^+$ |

### *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***08/2010*** |
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| ***$NH_4$*** • *F1 = 0 • F2 = 29.58 • K = -1.083* | |
| ***CADAS 30/30S/50/50S*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 690 nm • Pro.: 1 • F1 = 0 • F2 = 22.46 • K = -1.445* | |
| ***$NH_4$*** • *λ: 690 nm • Pro.: 1 • F1 = 0 • F2 = 28.88 • K = -1.856* | |
| ***ISIS 6000/9000*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 695 nm • Pro.: 1 • F1 = 0 • F2 = 22.98 • K = -1.865* | |
| ***$NH_4$*** • *λ: 695 nm • Pro.: 1 • F1 = 0 • F2 = 29.54 • K = -2.397* | |
| ***CADAS 100 / LPG 158*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 694 nm • F1 = 22.48 • F2 = -0.721* | |
| ***$NH_4$*** • *λ: 694 nm • F1 = 28.84 • F2 = -0.931* | |
| ***CADAS 100 / LPG 210*** | ***08/2010*** |
| ***$NH_4$-N*** • *λ: 694 nm • F1 = 22.48 • F2 = -0.721* | |
| ***$NH_4$*** • *λ: 694 nm • F1 = 28.84 • F2 = -0.931* | |
| ***CADAS 200*** | ***08/2010*** |
| ***$NH_4$-N*** • *E1W1 • C1 = E1*F1-F2 •* | |
| *W1 = 694 nm • F1 = 22.48 • F2 = 1.465* | |
| ***$NH_4$*** • *E1W1 • C1 = E1*F1-F2 •* | |
| *W1 = 694 nm • F1 = 28.91 • F2 = 1.884* | |

**D**

## LCK 303 Ammonium-Stickstoff

***Bitte "Ausgabedatum" (s. Datentabelle) und "Hinweis" beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Ammoniumionen reagieren bei pH 12.6 mit Hypochloritionen und Salicylationen in Gegenwart von Nitroprussid-Natrium als Katalysator zu Indophenolblau.

**Anwendungsbereich**
Oberflächenwasser, Abwasser, Boden, Substrat

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.

Primäre Amine werden miterfasst und ergeben Mehrbefunde. Ein 10000facher Überschuss an Harnstoff stört nicht. Alle Reduktionsmittel stören und führen zu Minderbefunden.
***Ein hoher Überschuss an Ammonium kann zu Ergebnisanzeigen innerhalb des Messbereichs führen. Hier ist eine Plausibilitätskontrolle durch Verdünnen empfehlenswert.***
Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**pH-Wert** Probe .....4 – 9
**Temperatur** Probe/Analysenküvette .....20°C
***Abweichende Temperaturen beeinflussen die Ergebnisrichtigkeit.***
Die Wasserprobe sollte sobald wie möglich nach der Probenahme untersucht werden.

**Zeitabhängigkeit**
Die Endextinktion liegt nach einer Reaktionszeit von ***15 min*** vor und bleibt dann ***15 min konstant***.

**Hinweis**
***Faktoränderung bei allen Photometertypen.***

**F**

## LCK 303 Azote ammoniacal

***Vérifier la date d'édition (voir table des données) et lire la "Remarque". Conseils de securité et date de péremption sur l'emballage.***

**Principe**
En présence de sodium nitroprussique agissant comme catalyseur et à une valeur du pH d'environ 12.6, les ions ammonium réagissent avec les ions hypochloreux et salicyliques et donnent une coloration bleue indophénol.

**Domaine d'application**
Eaux de surface, eaux de rejet, sols, substrats

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.

Les amines primaires sont aussi déterminées et sont donc à l'origine des résultats trop élevés. Un excédent 10000 fois plus élevé en urée ne gêne pas l'évaluation. Tous les réducteurs gênent et donnent des résultats trop faibles.
***Malgré un excédent important d'ammonium, l'appareil peut tout de même afficher un résultat d'analyse compris dans la gamme de mesure. Pour éliminer une telle erreur, il est recommandé ici de vérifier le résultat obtenu en effectuant une nouvelle analyse après avoir dilué l'échantillon (contrôle de plausibilité).***
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**pH** échantillon .....4 – 9
**Température** échantillon/cuve d'analyse .....20°C
***Des températures différentes influencent l'exactitude des résultats.***
L'analyse doit être réalisée immédiatement après la prise d'échantillon.

**Importance du temps**
L'extinction finale apparaît après un temps de réaction de ***15 min*** et reste ***constante*** pendant ***15 min***.

**Remarque**
***Modification de facteur pour tous les types de photomètres.***

**I**

## LCK 303 Ammonio/Azoto ammoniacale

***Si prega di verificare la "Data di Edizione" (vedi tabella dati) e di leggere le "Note". Avvertenze e data di scadenza sulla confezione.***

**Principio**
Ioni ammonio reagiscono a un pH 12.6 con ioni di ipoclorito e di salicilato, in presenza di nitroprussiato sodico quale catalizzatore, dando il blu indofenolo.

**Applicazione**
Acque di superficie, acque di scarico, terreni, substrati

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.

Le ammine primarie possono reagire dando valori più elevati. Un contenuto di urea 10000 volte più elevato non interferisce. Tutte le sostanze riducenti interferiscono e danno valori minori.
***Concentrazioni molto elevate di ammonio rischiano di dare risultati che rientrano nel campo di misura. Verificare diluendo il campione.***
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**pH** campione .....4 – 9
**Temperatura** campione/cuvetta d'analisi .....20°C
***Variazioni della temperatura influenzano la correttezza del valore misurato.***
Fare l'analisi subito dopo aver prelevato in campione!

**Tempo**
Il valore definitivo dell'estinzione si ottiene dopo ***15 min*** di reazione; il valore rimane ***costante per 15 min***.

**Note**
***Variazione del fattore su tutti i fotometri.***

AD 303 P / Druckfarbe schwarz / 1

**LCK 303** *02/2000*

**$NH_4$-N / $NH_4$**

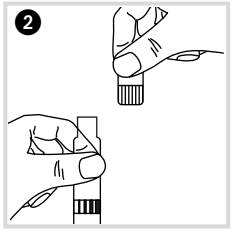

Siegelfolie von dem aufgeschraubten **DosiCap® Zip *vorsichtig*** abziehen.

Enlevez ***délicatement*** la feuille de protection du **DosiCap Zip** détachable.

Rimuovere ***con attenzione*** il foglio di alluminio.

Afdekfolie ***voorzichtig*** verwijderen.

***Carefully*** remove the foil from the screwed-on **DosiCap Zip**.



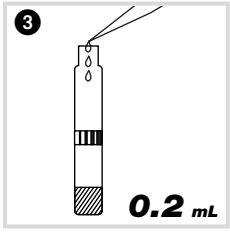

**DosiCap® Zip** abschrauben.

Dévissez le **DosiCap Zip**.

Svitare il **DosiCap Zip**.

**DosiCap Zip** afschroeven.

Unscrew the **DosiCap Zip**.



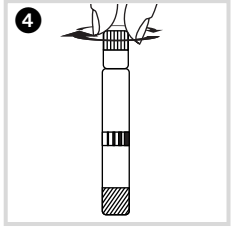

***0.2 mL*** Probe pipettieren.

Pipetter ***0.2 mL*** d'échantillon.

Pipettare ***0.2 mL*** di campione.

***0.2 mL*** monster pipetteren.

Pipette ***0.2 mL*** sample.



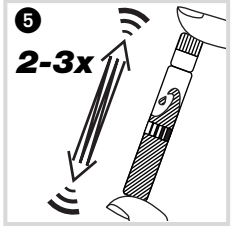

***Sofort*** **DosiCap® Zip** aufschrauben; Riffelung oben.

Vissez ***immédiatement*** le **DosiCap Zip**; dirigeant le cannelage vers le haut.

Avvitare ***subito*** il **DosiCap Zip**; scanalatura esterna verso l'alto.

***Onmiddellijk*** **DosiCap Zip** opschroeven; geribbelde zijde naar boven.

***Immediately*** screw the **DosiCap Zip** back; fluting at the top.



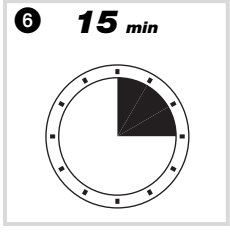

Kräftig schütteln.

Secouer énergiquement.

Agitare energicamente.

Krachtig schudden.

Shake firmly.




Nach ***15 min*** Küvette außen gut säubern und auswerten.

Attendre ***15 min***, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***15 min*** pulire bene la cuvetta esternamente e leggere.

Na ***15 min*** het kuvet van buiten goed reinigen en meten.

After ***15 min*** thoroughly clean the outside of the cuvette and evaluate.

## *Auswertung / Evaluation / Lettura / Meting*

| | **Analysenküvette** ❶<br>**Cuve d'analyse**<br>**Cuvetta d'analisi**<br>**Analyse-kuvet**<br>**Sample cuvette** |
|---|---|
| **Barcode** [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

**LASA aqua / LASA 1 / plus**

❻ Vom Ergebnis abziehen:
$NH_4$-N: ***0.252 mg/L*** /
$NH_4$: ***0.32 mg/L***

❻ Soustraire au résultat:
$NH_4$-N: ***0.252 mg/L*** /
$NH_4$: ***0.32 mg/L***

❻ Sottrarre dal risultato:
$NH_4$-N: ***0.252 mg/L*** /
$NH_4$: ***0.32 mg/L***

❻ Van het resultaat aftrekken:
$NH_4$-N: ***0.252 mg/L*** /
$NH_4$: ***0.32 mg/L***

❻ Substract from the result:
$NH_4$-N: ***0.252 mg/L*** /
$NH_4$: ***0.32 mg/L***

**LASA 10 / 20**

❻ $NH_4$-N: Ergebnis multiplizieren mit: ***1.12,***
vom Ergebnis abziehen: ***0.239 mg/L***
$NH_4$: Ergebnis multiplizieren mit: ***1.12***,
vom Ergebnis abziehen: ***0.311 mg/L***

❻ $NH_4$-N: Multiplier le résultat par: ***1.12***,
soustraire au résultat: ***0.239 mg/L***
$NH_4$: Multiplier le résultat par: ***1.12***,
soustraire au résultat: ***0.311 mg/L***

❻ $NH_4$-N: Moltiplicare il risultato per: ***1.12***,
sottrarre dal risultato: ***0.239 mg/L***
$NH_4$: Moltiplicare il risultato per: ***1.12***,
sottrarre dal risultato: ***0.311 mg/L***

❻ $NH_4$-N: Resultaat vermeningvuldigen met: ***1.12***,
van het resultaat aftrekken: ***0.239 mg/L***
$NH_4$: Resultaat vermeningvuldigen met: ***1.12***,
van het resultaat aftrekken: ***0.311 mg/L***

❻ $NH_4$-N: Result must be multiplied with: ***1.12***.
Substract from the result: ***0.239 mg/L***
$NH_4$: Result must be multiplied with: ***1.12***.
Substract from the result: ***0.311 mg/L***

| | **Filter** ❶<br>**Filtre**<br>**Filtro**<br>**Filter**<br>**Filter** | **Eprom** ❷ | **Test** ❸<br>**- anwählen**<br>**- choisir**<br>**- selezionare**<br>**- oproepen**<br>**- select** | **Kontrollnr.** ❹<br>**No. de contrôle**<br>**No. di controllo**<br>**Controlegetal**<br>**Control no.** | **Analysenküvette** ❺<br>**Cuve d'analyse**<br>**Cuvetta d'analisi**<br>**Analyse-kuvet**<br>**Sample cuvette** |
|---|---|---|---|---|---|
| **LASA aqua** | △ 303 N / △ 303 | _ : 46 | ***$NH_4$-N:*** △ 303 N ***/ $NH_4$:*** △ 303 | -- | ✔ |
| **LASA 1 / plus** | 690 nm | -- | NH4-N / NH4 LCK 303 | 9 | ✔ |
| **LASA 10 / 20** | -- | _ : 46 | NH4-N / NH4 LCK 303 | 9 | ✔ |

| | **Filter** ❶<br>**Filtre**<br>**Filtro**<br>**Filter**<br>**Filter** | **Test** ❷<br>**- anwählen**<br>**- choisir**<br>**- selezionare**<br>**- oproepen**<br>**- select** | **Faktor**<br>**Facteur**<br>**Fattore**<br>**Factor**<br>**Factor** | **Kontrollnr.** ❸<br>**No. de contrôle**<br>**No. di controllo**<br>**Controlegetal**<br>**Control no.** | **Leerwert (dest. Wasser)** ❹<br>**Valeur à blanc (l'eau dist.)**<br>**Bianco (acqua dist.)**<br>**Blanko (gedest. water)**<br>**Blank-value (dist. water)** | **Analysenküvette** ❺<br>**Cuve d'analyse**<br>**Cuvetta d'analisi**<br>**Analyse-kuvet**<br>**Sample cuvette** |
|---|---|---|---|---|---|---|
| **LP1W** | 695 nm | -- | ***$NH_4$-N:*** 23.01 ***/ $NH_4$:*** 29.58 | -- | LCW 919 | ✔ |
| **LP2W** | 695 nm | NH4-N / NH4 LCK 303 | -- | 9 | LCW 919 | ✔ |


**LP1W**

❼ Vom Ergebnis abziehen:
$NH_4$-N: ***0.84 mg/L*** /
$NH_4$: ***1.083 mg/L***

❼ Soustraire au résultat:
$NH_4$-N: ***0.84 mg/L*** /
$NH_4$: ***1.083 mg/L***

❼ Sottrarre dal risultato:
$NH_4$-N: ***0.84 mg/L*** /
$NH_4$: ***1.083 mg/L***

❼ Van het resultaat aftrekken:
$NH_4$-N: ***0.84 mg/L*** /
$NH_4$: ***1.083 mg/L***

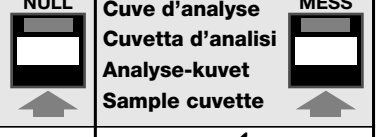
❼ Substract from the result
$NH_4$-N: ***0.84 mg/L*** /
$NH_4$: ***1.083 mg/L***

| | **Filter** ❶<br>**Filtre**<br>**Filtro**<br>**Filter**<br>**Filter** | **Eprom** ❷ | **Mode** ❸ | **Test** ❹<br>**- anwählen**<br>**- choisir**<br>**- selezionare**<br>**- oproepen**<br>**- select** | **Kontrollnr.** ❺<br>**No. de contrôle**<br>**No. di controllo**<br>**Controlegetal**<br>**Control no.** | **Analysenküvette, grüne Taste / Messen** ❻<br>**Cuve d'analyse, touche verte / Mesurer**<br>**Cuvetta d'analisi, tasto verde / Lettura**<br>**Analyse-kuvet, groene toets / Meten**<br>**Sample cuvette, green key / Read** |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 46 | -- | 303 | 9 | ✔ |
| **ISIS 6000** | -- | _ : 46 | [2] | 303 | 9 | ✔ |
| **LASA 30** | 695 nm | -- | Dr. Lange | 303 | 9 | ✔ |
| **DR 1900** | -- | -- | [3] | 303 | 9 | ✔ |

[2] KÜVETTEN-TEST
[2] TEST EN CUVE
[2] CUVETTE-TEST
[2] KUVETTENTEST
[2] CUVETTE TEST

[3] BARCODE-PROGRAMME
[3] PROGR. CODE BARRE
[3] PROGRAMMI COD. A BARRE
[3] BARCODEPROGRAMMA'S
[3] BARCODE PROGRAMS

| | **Mode** ❶ | **Symbol** ❷<br>**Symbole**<br>**Simbolo**<br>**Symbool**<br>**Symbol** | **Kontrollnr.** ❸<br>**No. de contrôle**<br>**No. di controllo**<br>**Controlegetal**<br>**Control no.** | **Leerwert (dest. Wasser)** ❹<br>**Valeur à blanc (l'eau dist.)**<br>**Bianco (acqua dist.)**<br>**Blanko (gedest. water)**<br>**Blank-value (dist. water)** | **Analysenküvette** ❺<br>**Cuve d'analyse**<br>**Cuvetta d'analisi**<br>**Analyse-kuvet**<br>**Sample cuvette** |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | ***$NH_4$-N:*** $ 303 N ***/ $NH_4$:*** $ 303 | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | ***$NH_4$-N:*** 303 N ***/ $NH_4$:*** 303 | 9 | LCW 919 | ✔ |



REF 985 066

en

# Test 0-66 09.14
# *NANOCOLOR*® Nitrate 250

**Method:**
Photometric determination with 2,6-dimethylphenol in sulfuric acid / phosphoric acid

| | | |
|---|---|---|
| Range: | **4–60 mg/L $NO_3$-N** | **20–250 mg/L $NO_3^-$** |
| Factor range (depends on wavelength): | **0068.–0081.** | **0303.–0355.** |
| Wavelength (HW = 5–17 nm): | **385 nm** | |
| Factor: | **0042.** | **0185.** |
| Wavelength (HW = 5–12 nm): | **365 nm** | |
| Reaction time: | **10 min (600 s)** | |
| Reaction temperature: | **20–25 °C** | |

**Contents of reagent set:**
20 test tubes Nitrate 250
1 test tube with 11 mL $NO_3$ / N R2

**Hazard warning:**
Test tubes contain sulfuric acid 51–80 % and phosphoric acid 25–50 %, reagent R2 contains 2-propanol 20–50 %.
H314 Causes severe skin burns and eye damage.
P260, P280, P301+330+331, P303+361+353, P304+340, P305+351+338, P501 Do not breathe vapours. Wear protective gloves / eye protection. IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water / shower. IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Dispose of contents / container to regulated waste treatment. For further information ask for a safety data sheet.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Nitrate / Nitrite (10–500 mg/L $NO_3^-$, REF 913 13) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly. In the same check it is possible to proof the interferences of nitrite.

**Interferences:**
Nitrite interferes > 3 mg/L (check with QUANTOFIX® Nitrite – REF 913 11). This can be circumvented by addition of 1 spoon of amidosulfonic acid (REF 918 973) to 10 mL test sample. Wait 10 min to determine nitrate.
The following ions will not interfere: < 2500 mg/L $Cl^-$, $CO_3^{2-}$; < 10 mg/L $Cl_2$.

The method can not be applied for the analysis of sea water.

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**0.2 mL** test sample *(the pH value of the sample must be between pH 1 and 13)* and
**0.5 mL** R2, mix by **shaking gently** *(Test tube becomes warm!)*.
Clean outside of test tube and measure after 10 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-66.

**Measurement when samples are colored or turbid:**
For all *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Multistandard Sewage influx (REF 925 012) or Multistandard Seepage water (REF 925 013)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***

PD 14122 / A020712 / 985 066 / 0943

UNITED FOR WATER QUALITY

# LCK 340

## 5 – 35 mg/L $NO_3$-N / 22 – 155 mg/L $NO_3$

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+15°C ..... +25°C

**T1**

| | |
|---|---|
| **2000 mg/L:** | $K^+$ |
| **1500 mg/L:** | $Na^+$ |
| **1000 mg/L:** | $Cl^-$ |
| **500 mg/L:** | CZV / COD *) |
| **250 mg/L:** | $Ca^{2+}$ |
| **100 mg/L:** | $Ag^+$ |
| **50 mg/L:** | $Pb^{2+}$, $Zn^{2+}$, $Ni^{2+}$, $Fe^{3+}$, $Cd^{2+}$, $Cu^{2+}$ |
| **20 mg/L:** | $Fe^{2+}$ |
| **10 mg/L:** | $Co^{2+}$ |
| **5 mg/L:** | $Cr^{6+}$ |
| **2 mg/L:** | $NO_2^-$ |

*) (Kaliumwaterstofftalaat)
(Potassium hydrogen phtalate)

### *Datatabel / Data table*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***06/2001*** |
| ***$NO_3$-N*** • *F1 = 0 • F2 = 45.59 • K = -0.405* | |
| ***$NO_3$*** • *F1 = 0 • F2 = 201.8 • K = -1.776* | |
| ***CADAS 30/30S/50/50S*** | ***06/2001*** |
| ***$NO_3$-N*** • *λ: 345 nm • Pro.: 1 • F1 = 0 • F2 = 42.2 • K = -3.007* | |
| ***$NO_3$*** • *λ: 345 nm • Pro.: 1 • F1 = 0 • F2 = 186.8 • K = -13.34* | |
| ***ISIS 6000/9000*** | ***06/2001*** |
| ***$NO_3$-N*** • *λ: 360 nm • Pro.: 1 • F1 = 0 • F2 = 47.33 • K = -3.001* | |
| ***$NO_3$*** • *λ: 360 nm • Pro.: 1 • F1 = 0 • F2 = 209.5 • K = -13.26* | |
| ***CADAS 100 / LPG 158*** | ***06/2001*** |
| ***$NO_3$-N*** • *λ: 370 nm • F1 = 60.65 • F2 = -0.607* | |
| ***$NO_3$*** • *λ: 370 nm • F1 = 268.6 • F2 = -2.679* | |
| ***CADAS 100 / LPG 210*** | ***06/2001*** |
| ***$NO_3$-N*** • *λ: 370 nm • F1 = 60.65 • K = -0.607* | |
| ***$NO_3$*** • *λ: 370 nm • F1 = 268.6 • K = -2.679* | |
| ***CADAS 200*** | ***06/2001*** |
| ***$NO_3$-N*** • *E1W1 • C1 = E1∗ F1-F2 • W1 = 370 nm • F1 = 59.46 • F2 = 3.217* | |
| ***$NO_3$*** • *E1W1 • C1 = E1∗ F1-F2 • W1 = 370 nm • F1 = 263.2 • F2 = 14.26* | |

**NL**

## LCK 340 Nitraat

***Let a.u.b. op de "Uitgave datum" (zie datatabel) en lees de "Opmerking!". Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
In zwavel- en fosforzuuroplossing reageren nitraat-ionen met 2.6-dimethylfenol tot 4-nitro-2.6-dimethyl-fenol.

**Toepassingsgebied**
Afvalwater (let op storingen!), drinkwater, ongezuiverd water, oppervlaktewateren, grond, substraat, voedingsstof

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aan-gegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.
Een hoge belasting van oxideerbare, organische substanties (CZV) leidt tot een verkleuring van de reagentia en daardoor ook tot een hoger resultaat. De test is daarom alleen bij onderzoek van afval-water te gebruiken, wanneer de CZV-waarde beneden de 500 mg/L ligt.
De meetresultaten zijn via een plausibiliteitsonder-zoek te controleren (verdunning en/of standaard-additie).

**Opheffen van storingen**
Nitriet-concentraties boven 2.0 mg/L storen (hogere resultaten!) en kunnen door toevoeging van een spatelpunt amidosulfonzuur worden geëlimi-neerd. De chloriden kunnen met zilversulfaat als zilverchloride worden neergeslagen.
Bij hogere calcium-concentraties ontstaat een troebeling, die de bepaling stoort. Door toevoeging van een spatelpunt EDTA aan het monster kan dit echter worden verhinderd.

**pH-waarde** monster .....3 – 10
**Temperaturen** monster/reagentia.....20 – 24°C
***Afwijkende temperaturen beïnvloeden de nauwkeurigheid van het resultaat.***
Het tijdstip waarop het monster wordt onderzocht, mag niet langer dan 3 uur na de monstername liggen. ***Koel bewaren!***

**Opmerking!**
***Verandering van de factoren in alle fotometers (met uitzondering van LASA aqua).***

**GB**

## LCK 340 Nitrate

***Please check the "Edition Date" (see data table) and read the "Note". Safety advice and expiry date on package.***

**Principle**
Nitrate ions in solutions containing sulphuric and phosphoric acids react with 2.6-dimethylphenol to form 4-nitro-2.6-dimethylphenol.

**Range of Application**
Waste water (beware of interferences!), drinking water, raw water, surface water, soils, substrates, nutrient solutions

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.
High loads of oxidizable organic substances (COD) cause the reagent to change colour and give high-bias results. The test can thus only be used for waste water analyses if the COD is less than 500 mg/L.
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**Removal of Interferences**
Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results) and can be removed by the addition of a spatula-tipfull of amidosulphonic acid. The chloride can be precipitated out as silver chloride by adding silver sulphate.
High calcium concentrations cause turbidity. This interferes with the determination but can be prevented by adding a spatula-tipfull of EDTA to the sample.

**pH** sample .....3 – 10
**Temperature** sample/reagents .....20 – 24°C
***In case of not working at the right recommanded temperature an incorrect result may be obtained.***
Not more than 3 hours should elapse between sampling and analysis. ***Store in a cool place!***

**Note**
***Change of factor for all types of photometers (except LASA aqua).***

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 340

## 5 – 35 mg/L $NO_3$-N / 22 – 155 mg/L $NO_3$

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+15°C ..... +25°C

**T1**

| | |
|---|---|
| **2000 mg/L:** | $K^+$ |
| **1500 mg/L:** | $Na^+$ |
| **1000 mg/L:** | $Cl^-$ |
| **500 mg/L:** | CSB / DCO / COD *) |
| **250 mg/L:** | $Ca^{2+}$ |
| **100 mg/L:** | $Ag^+$ |
| **50 mg/L:** | $Pb^{2+}$, $Zn^{2+}$, $Ni^{2+}$, $Fe^{3+}$, $Cd^{2+}$, $Cu^{2+}$ |
| **20 mg/L:** | $Fe^{2+}$ |
| **10 mg/L:** | $Co^{2+}$ |
| **5 mg/L:** | $Cr^{6+}$ |
| **2 mg/L:** | $NO_2^-$ |

*) (Kaliumhydrogenphtalat)
(Hydrogènphthalate de potassium)
(Potassio idrogenoftalato)

### *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000*** | ***06/2013*** |
| ***Software Download: www.hach-lange.com*** | |
| ***LP2W*** | ***06/2001*** |
| ***$NO_3$-N*** • *F1 = 0 • F2 = 45.59 • K = -0.405* | |
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| ***$NO_3$-N*** • *λ: 370 nm • F1 = 60.65 • F2 = -0.607* | |
| ***$NO_3$*** • *λ: 370 nm • F1 = 268.6 • F2 = -2.679* | |
| ***CADAS 100 / LPG 210*** | ***06/2001*** |
| ***$NO_3$-N*** • *λ: 370 nm • F1 = 60.65 • K = -0.607* | |
| ***$NO_3$*** • *λ: 370 nm • F1 = 268.6 • K = -2.679* | |
| ***CADAS 200*** | ***06/2001*** |
| ***$NO_3$-N*** • *E1W1 • C1 = E1∗ F1-F2 • W1 = 370 nm • F1 = 59.46 • F2 = 3.217* | |
| ***$NO_3$*** • *E1W1 • C1 = E1∗ F1-F2 • W1 = 370 nm • F1 = 263.2 • F2 = 14.26* | |

**D**

### LCK 340 Nitrat

***Bitte "Ausgabedatum" (s. Datentabelle) und "Hinweis" beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
In schwefel- und phosphorsaurer Lösung reagieren Nitrationen mit 2.6-Dimethylphenol zu 4-Nitro-2.6-dimethylphenol.

**Anwendungsbereich**
Abwasser (Störungen beachten!), Trinkwasser, Rohwasser, Oberflächenwasser, Boden, Substrat, Nährlösung

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.
Hohe Belastung von oxidierbaren, organischen Substanzen (CSB) führen zu einer Verfärbung des Reagenzes und damit zu Mehrbefunden. Der Test ist nur bei Abwasseruntersuchungen verwendbar, bei denen der CSB-Gehalt unter 500 mg/L liegt. Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**Beseitigung von Störungen**
Nitrit-Konzentrationen über 2.0 mg/L stören (Mehrbefunde) und können durch Zusatz von Amidosulfonsäure beseitigt werden.
Chloride können mit Silbersulfat als Silberchlorid gefällt werden.
Bei höheren Calcium-Konzentrationen tritt eine Trübung auf. Diese stört die Bestimmung, kann jedoch durch Zusatz von EDTA zur Probe verhindert werden.

**pH-Wert** Probe .....3 – 10
**Temperatur** Probe/Reagenzien .....20 – 24°C
***Abweichende Temperaturen beeinflussen die Ergebnisrichtigkeit.***
Zwischen Probenahme und Untersuchung der Probe sollten 3 Stunden nicht überschritten werden.
***Probe kühl lagern!***

**Hinweis**
***Faktoränderung bei allen Photometertypen (außer LASA aqua).***

**F**

### LCK 340 Nitrate

***Vérifier la date d'édition (voir table des données) et lire la "Remarque". Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Dans une solution d'acide sulfurique et phosphorique, les ions nitrate réagissent avec le 2.6-diméthylphénol pour donner du 4-nitro-2.6-diméthylphénol.

**Domaine d'application**
Eaux de rejet (voir perturbations!), eaux potables, eaux brutes, eaux de surface, sols, substrat, solutions nutritives

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.
Une présence importante de substances organiques oxydables (DCO) est à l'origine d'une coloration du réactif induisant des résultats trop élevés. Le test est donc applicable aux eaux de rejet, à condition que leur teneur en DCO soit en-dessous de 500 mg/L. Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**Solutions aux perturbations**
Les concentrations en nitrite au-dessus de 2.0 mg/L gênent l'évaluation (résultats trop élevés) et peuvent être éliminées en ajoutant un bout de spatule d'acide sulfamique.
Les chlorures peuvent être précipités par le sulfate d'argent sous forme de chlorure d'argent.
Les concentrations de calcium élevées génèrent une turbidité qui gêne la détermination, mais qui toutefois peut être évitée en ajoutant un bout de spatule d'EDTA à l'échantillon.

**pH** échantillon.....3 – 10
**Température** échantillon/réactifs .....20 – 24°C
***Des températures différentes influencent l'exactitude des résultats.***
Il ne doit pas s'écouler plus de 3 heures entre le prélèvement de l'échantillon et l'analyse.
***Conserver au frais!***

**Remarque**
***Modification de facteur pour tous les types de photomètres (à l'exception LASA aqua).***

**I**

### LCK 340 Nitrati

***Si prega di verificare la "Data di Edizione" (vedi tabella dati) e di leggere le "Note". Avvertenze e data di scadenza sulla confezione.***

**Principio**
Ioni nitrato reagiscono in soluzione di acido solforico-fosforico con 2.6-dimetilfenolo dando 4-nitro-2.6-dimetilfenolo.

**Applicazione**
Acque di scarico (v. "interferenze"), acqua potabile, acqua grezza, acque di superficie, terreni, substrati, soluzioni nutritive

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.
Con la presenza di sostanze organiche ossidabili (COD) in forti concentrazioni, il reattivo cambia colore e provoca risultati in eccesso. Per questo motivo, il test si può usare solamente per acque con concentrazioni COD inferiori a 500 mg/L.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**Eliminazione interferenze**
Concentrazioni di nitriti superiori a 2.0 mg/L disturbano (valori in eccesso) e possono essere eliminati con l'aggiunta di acido amidosulfonico.
I cloruri vanno precipitati sottoforma di cloruri d'argento con solfato d'argento.
In caso di forti concentrazioni di calcio la soluzione si presenta torbida. Può essere letta legando il calcio con EDTA.

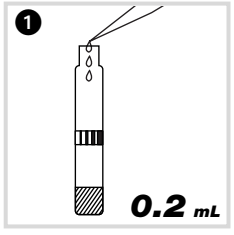
**pH** campione.....3 – 10
**Temperatura** campione/reagenti .....20 – 24°C
***Variazioni della temperatura influenzano la correttezza del valore misurato.***
Fra il prelievo del campione e l'analisi non devono passare più di 3 ore. ***Mettere in fresco!***

**Note**
***Variazione del fattore su tutti i fotometri (eccetto LASA aqua).***

AD 340 N / Druckfarbe burgund / 1

**LCK 340** *11/2005*

**$NO_3$-N / $NO_3$**



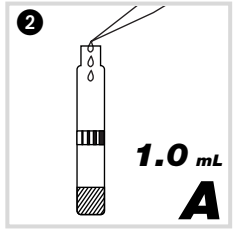

***0.2 mL*** Probe ***langsam*** pipettieren.

Pipetter ***lentement 0.2 mL*** d'échantillon.

Pipettare ***con attenzione 0.2 mL*** di campione.

***Langzaam 0.2 mL*** monster pipetteren.

***Slowly*** pipette ***0.2 mL*** sample.



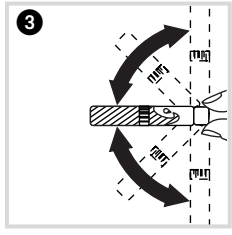

***1.0 mL*** Lösung ***A*** (LCK 340 A) ***langsam*** pipettieren.

Pipetter ***lentement 1.0 mL*** de la solution ***A*** (LCK 340 A).

Pipettare ***con attenzione 1.0 mL*** di soluzione ***A*** (LCK 340 A).

***Langzaam 1.0 mL*** oplossing ***A*** (LCK 340 A) pipetteren.

***Slowly*** pipette ***1.0 mL*** solution ***A*** (LCK 340 A).



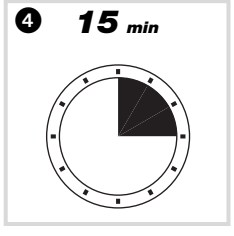

Küvette verschließen und schwenken, bis keine Schlieren mehr zu beobachten sind.

Fermer la cuve et mélanger le contenu en la retournant plusieurs fois de suite jusqu'à ce que le mélange soit complet.

Tappare la cuvetta e mescolare accuratamente fino a miscelazione completa (assenza di striature).

Kuvet sluiten en zwenken tot er geen stroopdraden meer aanwezig zijn.

Close cuvette and invert a few times until no more streaks can be seen.




Nach ***15 min*** Küvette außen gut säubern und auswerten.

Attendre ***15 min***, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***15 min*** pulire bene la cuvetta esternamente e leggere.

Na ***15 min*** het kuvet van buiten goed reinigen en meten.

After ***15 min*** thoroughly clean the outside of the cuvette and evaluate.

## *Auswertung / Evaluation / Lettura / Meting*

| | Analysenküvette ❶<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|
| IBR / IBR+ Barcode [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

**LASA aqua**

❻ $NO_3$-N: ***(display x 4.82) - 0.88***
$NO_3$: ***(display x 4.82) - 3.9***

❻ $NO_3$-N: ***(affichage x 4.82) - 0.88***
$NO_3$: ***(affichage x 4.82) - 3.9***

Das errechnete Ergebnis muss innerhalb des Messbereichs liegen.

Le résultat calculé doit être dans la gamme de mesure.

Il risultato cosi calcolato deve rientrare nel campo di misura.

Let op de grenzen van het meetbereik.

Please observe the measuring range limits.

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Test ❸<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | KontrolInr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette | ❻ |
|---|---|---|---|---|---|---|
| **LASA aqua** | ☐ 339 N / △ 339 | _ : 30 | ***$NO_3$-N:*** ☐ 339 N ***/ $NO_3$ :*** △ 339 | -- | ✔ | ✔ |
| **LASA 1 / plus** | 330 nm | -- | NO3-N / NO3 LCK 340 | 2 | ✔ | -- |
| **LASA 10 / 20** | -- | _ : 46 | NO3-N / NO3 LCK 340 | 2 | ✔ | -- |

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Test ❷<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Faktor ❸<br>Facteur<br>Fattore<br>Factor<br>Factor | KontrolInr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Nulllösung ❺<br>Solution zéro<br>Bianco<br>Nulkuvet<br>Zero-solution<br>Null | Leerwert (dest. Wasser) ❻<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water)<br>Null | Analysenküvette ❼<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette<br>Ergebnis |
|---|---|---|---|---|---|---|---|
| **LP1W** | 340 nm / Nitrat 339 | -- | ***$NO_3$-N:*** 45.59 ***/ $NO_3$ :*** 201.8 | -- | LCW 918 | -- | ✔ |
| **LP2W** | 340 nm / Nitrat 339 | NO3-N / NO3 LCK 340 | -- | 5 | -- | LCW 919 | ✔ |

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Mode ❸ | Test ❹<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | KontrolInr. ❺<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette, grüne Taste / Messen ❻<br>Cuve d'analyse, touche verte / Mesurer<br>Cuvetta d'analisi, tasto verde / Lettura<br>Analyse-kuvet, groene toets / Meten<br>Sample cuvette, green key / Read |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 46 | -- | 340 | 5 | ✔ |
| **ISIS 6000** | -- | _ : 46 | [2] | 340 | 5 | ✔ |
| **LASA 30** | 340 nm | -- | Dr. Lange | 340 | 5 | ✔ |
| **DR 1900** | -- | -- | [3] | 340 | 5 | ✔ |

[2] KÜVETTEN-TEST [3] BARCODE-PROGRAMME
[2] TEST EN CUVE [3] PROGR. CODE BARRE
[2] CUVETTE-TEST [3] PROGRAMMI COD. A BARRE
[2] KUVETTENTEST [3] BARCODEPROGRAMMA'S
[2] CUVETTE TEST [3] BARCODE PROGRAMS

| | Mode ❶ | Symbol ❷<br>Symbole<br>Simbolo<br>Symbool<br>Symbol | KontrolInr. ❸<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❹<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water)<br>NULL | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette<br>MESS |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | ***$NO_3$-N:*** $ 340 N ***/ $NO_3$ :*** $ 340 | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | ***$NO_3$-N:*** 340 N ***/ $NO_3$ :*** 340 | 5 | LCW 919 | ✔ |

**LP1W**

❽ Vom Ergebnis abziehen:
$NO_3$-N: ***0.405 mg/L***
$NO_3$: ***1.776 mg/L***

❽ Soustraire au résultat:
$NO_3$-N: ***0.405 mg/L***
$NO_3$: ***1.776 mg/L***

❽ Sottrarre dal risultato:
$NO_3$-N: ***0.405 mg/L***
$NO_3$: ***1.776 mg/L***

❽ Van het resultaat aftrekken:
$NO_3$-N: ***0.405 mg/L***
$NO_3$: ***1.776 mg/L***

❽ Substract from the result:
$NO_3$-N: ***0.405 mg/L***
$NO_3$: ***1.776 mg/L***

REF 985 069

en

# Test 0-69 10.13
# NANOCOLOR® Nitrite 4

**Method:**
Photometric determination with sulfanilamide and *N*-(1-naphthyl)ethylenediamine

| | | |
|---|---|---|
| Range: | **0.1–4.0 mg/L $NO_2$-N** | **0.3–13.0 mg/L $NO_2^-$** |
| Factor: | **004.9** | **016.0** |
| Wavelength (HW = 5–12 nm): | **540 nm** | |
| Reaction time: | **15 min (900 s)** | |
| Reaction temperature: | **20–25 °C** | |

**Contents of reagent set:**
20 test tubes Nitrite 4
1 tube *NANOFIX* Nitrite 4 R2

**Hazard warning:**
This tube test does not contain any harmful substances which must be specially labelled as hazardous.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Nitrite (1–80 mg/L $NO_2^-$, REF 913 11) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly.

**Interferences:**
Free chlorine, organic colloids and humic acids can cause interferences.
The following ions will not interfere:
< 1000 mg/L $Ca^{2+}$, Cr(III), $Cu^{2+}$, $Fe^{3+}$, $Mg^{2+}$, $Mn^{2+}$, $Ni^{2+}$, $Zn^{2+}$, $Cl^-$, $NO_3^-$, $PO_4^{3-}$, $SO_4^{2-}$
< 10 mg/L Cr(VI)

This method can be applied also for the analysis of sea water.

***Note:***
*For removal of emulsions, turbidities and colour prior to the test, e.g. for nitrite in cooling lubricants, seepage water from waste deposits etc., use Reagents for sample preparation by clarification precipitation (REF 918 937).*

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**200 µL** (= 0.2 mL) test sample *(the pH value of the sample must be between pH 4 and 13)* and
**1** ***NANOFIX*** R2, close and mix.
*(Close NANOFIX tube immediately after use.)*
Clean outside of the test tube and measure after 15 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-69.

**Measurement when samples are colored or turbid:**
For all *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Nitrite (REF 925 68)
Dilute 100+ addition solution with distilled water (1+1): 2.1 mg/L $NO_2$-N
Confidence interval: 1.9–2.3 mg/L $NO_2$-N

**Reference:**
German standard methods for the examination of water, waste water and sludge (DIN EN 26 777-D10)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***
PD 14122 / A031581 / 985 069 / 1033

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# LCK 341

## 0.015 – 0.6 mg/L $NO_2$-N / 0.05 – 2.0 mg/L $NO_2$

Lagerhinweis / Stabilité / Conservazione / Houdbaarheid / Storage: +15°C ..... +25°C

**T1**

| | |
|---|---|
| **2000 mg/L:** | $Cl^-$, $SO_4^{2-}$ |
| **1000 mg/L:** | $K^+$, $NO_3^-$ |
| **500 mg/L:** | $NH_4^+$, $PO_4^{3-}$, $Ca^{2+}$ |
| **100 mg/L:** | $Mg^{2+}$ |
| **50 mg/L:** | $Cr^{3+}$ |
| **25 mg/L:** | $Co^{2+}$, $Zn^{2+}$, $Cd^{2+}$, $Mn^{2+}$, $Hg^{2+}$ |
| **12 mg/L:** | $Ni^{2+}$ |
| **10 mg/L:** | $Ag^+$, $Fe^{2+}$ |
| **5 mg/L:** | $Sn^{4+}$, $Fe^{3+}$ |

### *Datatabel / Data table*

| ***LP2W*** | ***04/2005*** |
|---|---|
| ***$NO_2$-N*** • *F1 = 0 • F2 = 0.539 • K = -0.024* | |
| ***$NO_2$*** • *F1 = 0 • F2 = 1.763 • K = -0.078* | |
| ***CADAS 30/30S/50/50S*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 515 nm • Pro.: 1 • F1 = 0 • F2 = 0.483 • K = -0.035* | |
| ***$NO_2$*** • *λ: 515 nm • Pro.: 1 • F1 = 0 • F2 = 1.585 • K = -0.11* | |
| ***ISIS 6000/9000*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 500 nm • Pro.: 1 • F1 = 0 • F2 = 0.524 • K = -0.036* | |
| ***$NO_2$*** • *λ: 500 nm • Pro.: 1 • F1 = 0 • F2 = 1.726 • K = -0.119* | |
| ***CADAS 100 / LPG 158*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 515 nm • F = 0.481 • F2 = -0.019* | |
| ***$NO_2$*** • *λ: 515 nm • F = 1.586 • F2 = -0.065* | |
| ***CADAS 100 / LPG 210*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 515 nm • F1 = 0.481 • K = -0.019* | |
| ***$NO_2$*** • *λ: 515 nm • F1 = 1.586 • K = -0.065* | |
| ***CADAS 200*** | ***04/2005*** |
| ***$NO_2$-N*** • *E1W1 • C1 = E1∗F1-F2 •* *W1 = 515 nm • F1 = 0.481 • F2 = 0.036* | |
| ***$NO_2$*** • *E1W1 • C1 = E1∗F1-F2 •* *W1 = 515 nm • F1 = 1.576 • F2 = 0.118* | |

**NL**

### LCK 341 Nitriet

***Let a.u.b. op de "Uitgave datum" (zie datatabel) en lees de "Opmerking". Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
In zure oplossing reageert nitriet met primaire, aromatische aminen en vormen daarbij diazoniumzouten. Deze geven met aromatische verbindingen die een amino- of hydroxylgroep bevatten, een intensief gekleurde azo-kleurstof.

**Toepassingsgebied**
Afvalwater, drinkwater, mineraalwater, oppervlaktewateren

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.

Chroom(VI)-ionen storen de bepaling.
Koper(II)-ionen storen de bepaling al bij een concentratie van minder dan 1 mg/L.
De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaardadditie).

**pH-waarde** monster .....3 – 10
**Temperaturen** monster/reagentia.....15 – 25°C
Het tijdstip waarop het monster wordt onderzocht, mag niet langer dan ***3 uur*** na de monstername liggen.

**Opmerking!**
***Verandering van de factoren in alle fotometers.***

**GB**

### LCK 341 Nitrite

***Please check the "Edition Date" (see data table) and read the "Note". Safety advice and expiry date on package.***

**Principle**
Nitrites react with primary aromatic amines in acidic solution to form diazonium salts. These combine with aromatic compounds that contain an amino group or a hydroxyl group to form intensively coloured azo dyes.

**Range of Application**
Waste water, drinking water, table water, surface water, mineral water

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.

Chromium(VI) ions interfere with the determination.
Copper(II) ions interfere with the determination even at concentrations below 1 mg/L.
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**pH** sample .....3 – 10
**Temperature** sample/reagents .....15 – 25°C
Not more than ***3 hours*** should elapse between sampling and analysing the sample.

**Note**
***Change of factor for all types of photometers.***

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UNITED FOR WATER QUALITY

# LCK 341

**0.015 – 0.6 mg/L $NO_2$-N / 0.05 – 2.0 mg/L $NO_2$**

*Lagerhinweis*
*Stabilité*
*Conservazione*
*Houdbaarheid*
*Storage*
+15°C ..... +25°C

**T1**

| | |
|---|---|
| **2000 mg/L:** | $Cl^-$, $SO_4^{2-}$ |
| **1000 mg/L:** | $K^+$, $NO_3^-$ |
| **500 mg/L:** | $NH_4^+$, $PO_4^{3-}$, $Ca^{2+}$ |
| **100 mg/L:** | $Mg^{2+}$ |
| **50 mg/L:** | $Cr^{3+}$ |
| **25 mg/L:** | $Co^{2+}$, $Zn^{2+}$, $Cd^{2+}$, $Mn^{2+}$, $Hg^{2+}$ |
| **12 mg/L:** | $Ni^{2+}$ |
| **10 mg/L:** | $Ag^+$, $Fe^{2+}$ |
| **5 mg/L:** | $Sn^{4+}$, $Fe^{3+}$ |

## *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***LP2W*** | ***04/2005*** |
| ***$NO_2$-N*** • *F1 = 0 • F2 = 0.539 • K = -0.024* | |
| ***$NO_2$*** • *F1 = 0 • F2 = 1.763 • K = -0.078* | |
| ***CADAS 30/30S/50/50S*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 515 nm • Pro.: 1 • F1 = 0 • F2 = 0.483 • K = -0.035* | |
| ***$NO_2$*** • *λ: 515 nm • Pro.: 1 • F1 = 0 • F2 = 1.585 • K = -0.11* | |
| ***ISIS 6000/9000*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 500 nm • Pro.: 1 • F1 = 0 • F2 = 0.524 • K = -0.036* | |
| ***$NO_2$*** • *λ: 500 nm • Pro.: 1 • F1 = 0 • F2 = 1.726 • K = -0.119* | |
| ***CADAS 100 / LPG 158*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 515 nm • F = 0.481 • F2 = -0.019* | |
| ***$NO_2$*** • *λ: 515 nm • F = 1.586 • F2 = -0.065* | |
| ***CADAS 100 / LPG 210*** | ***04/2005*** |
| ***$NO_2$-N*** • *λ: 515 nm • F1 = 0.481 • K = -0.019* | |
| ***$NO_2$*** • *λ: 515 nm • F1 = 1.586 • K = -0.065* | |
| ***CADAS 200*** | ***04/2005*** |
| ***$NO_2$-N*** • *E1W1 • C1 = E1∗F1-F2 •* | |
| *W1 = 515 nm • F1 = 0.481 • F2 = 0.036* | |
| ***$NO_2$*** • *E1W1 • C1 = E1∗F1-F2 •* | |
| *W1 = 515 nm • F1 = 1.576 • F2 = 0.118* | |

**D**

## LCK 341 Nitrit

***Bitte "Ausgabedatum" (s. Datentabelle) und "Hinweis" beachten. Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
In saurer Lösung reagieren Nitrite mit primären, aromatischen Aminen unter Bildung von Diazoniumsalzen. Diese bilden mit aromatischen Verbindungen, die eine Amino- oder Hydroxylgruppe enthalten, intensiv gefärbte Azofarbstoffe.

**Anwendungsbereich**
Abwasser, Trinkwasser, Tafelwasser, Oberflächenwasser, Mineralwasser

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.

Chrom(VI)-Ionen stören die Bestimmung.
Kupfer(II)-Ionen stören die Bestimmung schon bei einer Konzentration unter 1 mg/L.
Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**pH-Wert** Probe .....3 – 10
**Temperatur** Probe/Reagenzien .....15 – 25°C
Zwischen Probenahme und Untersuchung der Probe sollten ***3 Stunden*** nicht überschritten werden.

**Hinweis**
***Faktoränderung bei allen Photometertypen.***

**F**

## LCK 341 Nitrite

***Vérifier la date d'édition (voir table des données) et lire la "Remarque". Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Les nitrites réagissent en solution acide avec les amines primaires et aromatiques pour donner des sels diazonium. Ceux-ci forment avec des composés aromatiques, contenant un amino-groupe ou un hydroxyle, un colorant azoique de couleur intense.

**Domaine d'application**
Eaux de rejet, eaux potables, eaux de table, eaux de surface, eaux minérales

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.

Les ions chrome(VI) gênent la détermination.
Les ions cuivre(II) gênent la détermination à partir d'une concentration de: 1.0 mg/L.
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**pH** échantillon.....3 – 10
**Température** échantillon/réactifs .....15 – 25°C
Il ne doit pas s'écouler plus de ***3 heures*** entre le prélèvement de l'échantillon et l'analyse.

**Remarque**
***Modification de facteur pour tous les types de photomètres.***

**I**

## LCK 341 Nitriti

***Si prega di verificare la "Data di Edizione" (vedi tabella dati) e di leggere le "Note". Avvertenze e data di scadenza sulla confezione.***

**Principio**
I nitriti reagiscono in soluzione acida con ammine aromatiche primarie formando sali di diazonio. Questi formano con complessi aromatici, contenenti un gruppo ammino o idrossilico, coloranti azoici intensamente colorati.

**Applicazione**
Acqua potabile, acqua da tavola, acqua minerale, acque di superficie, acque di scarico

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.

Ioni cromo(VI) disturbano.
Ioni rame(II) disturbano l'analisi anche in concentrazioni inferiori a 1 mg/L.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**pH** campione .....3 – 10
**Temperatura** campione/reagenti .....15 – 25°C
Fra il prelievo del campione e l'analisi non devono passare più di ***3 ore***.

**Note**
***Variazione del fattore su tutti i fotometri.***

AD 341 Q / Druckfarbe blau / 1

**LCK 341** *06/2001*

**$NO_2$-N / $NO_2$**

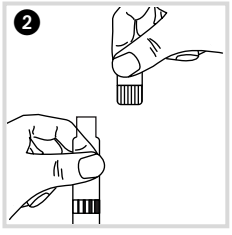

Siegelfolie von dem aufgeschraubten **DosiCap® Zip *vorsichtig*** abziehen.

Enlevez ***délicatement*** la feuille de protection du **DosiCap Zip** détachable.

Rimuovere ***con attenzione*** il foglio di alluminio.

Afdekfolie ***voorzichtig*** verwijderen.

***Carefully*** remove the foil from the screwed-on **DosiCap Zip**.



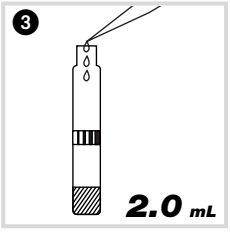

**DosiCap® Zip** abschrauben.

Dévissez le **DosiCap Zip**.

Svitare il **DosiCap Zip**.

**DosiCap Zip** afschroeven.

Unscrew the **DosiCap Zip**.



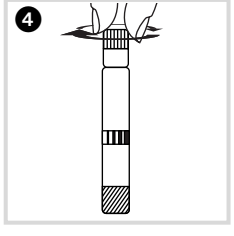

***2.0 mL*** Probe pipettieren.

Pipetter ***2.0 mL*** d'échantillon.

Pipettare ***2.0 mL*** di campione.

***2.0 mL*** monster pipetteren.

Pipette ***2.0 mL*** sample.




Sofort **DosiCap® Zip** aufschrauben; Riffelung oben.

Vissez immédiatement le **DosiCap Zip**; dirigeant le cannelage vers le haut.

Avvitare subito il **DosiCap Zip**; scanalatura esterna verso l'alto.

Onmiddellijk **DosiCap Zip** opschroeven; geribbelde zijde naar boven.

Immediately screw the **DosiCap Zip** back; fluting at the top.

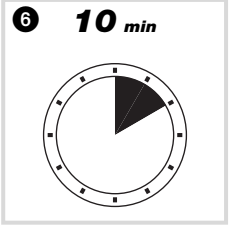

Kräftig schütteln, bis Lyophilisat gelöst ist.

Secouer énergiquement jusqu'à dissolution du lyophilisat.

Agitare energicamente fino a scioglimento completo del liofilizzato.

Krachtig schudden tot het lyofilisaat is opgelost.

Shake firmly until the freeze-dried contents are completely dissolved.




Nach ***10 min*** Küvette noch einmal schwenken, außen gut säubern und auswerten.

Attendre ***10 min***, melanger de nouveau, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***10 min***, mescolare nuovamente, pulire bene la cuvetta esternamente e leggere.

Na ***10 min*** het kuvet opnieuw zwenken, van buiten goed reinigen en meten.

After ***10 min***, invert a few times more, thoroughly clean the outside of the cuvette and evaluate.

## Auswertung / Evaluation / Lettura / Meting

| | Analysenküvette ❶<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette |
|---|---|
| IBR / IBR+ Barcode [1] | ✔ |

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Test ❷<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | Faktor ❸<br>Facteur<br>Fattore<br>Factor<br>Factor | KontrollNr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❺<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water) Null | Analysenküvette ❻<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette Ergebnis | Vom Ergebnis abziehen: ❼<br>Soustraire au résultat:<br>Sottrarre dal risultato:<br>Van het resultaat aftrekken:<br>Substract from the result: |
|---|---|---|---|---|---|---|---|
| **LP1W** | 535 nm | -- | ***$NO_2$-N:*** 0.539 ***/ $NO_2$:*** 1.763 | -- | LCW 919 | ✔ | ***$NO_2$-N:*** 0.024 ***/ $NO_2$:*** 0.078 |
| **LP2W** | 535 nm | NO2-N / NO2 LCK 341 | -- | 5 | LCW 919 | ✔ | -- |

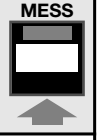
| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Mode ❸ | Test ❹<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | KontrollNr. ❺<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette, grüne Taste / Messen ❻<br>Cuve d'analyse, touche verte / Mesurer<br>Cuvetta d'analisi, tasto verde / Lettura<br>Analyse-kuvet, groene toets / Meten<br>Sample cuvette, green key / Read |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 48 | -- | 341 | 5 | ✔ |
| **ISIS 6000** | -- | _ : 48 | [2] | 341 | 5 | ✔ |
| **LASA 30** | 535 nm | -- | Dr. Lange | 341 | 5 | ✔ |
| **DR 1900** | -- | -- | [3] | 341 | 5 | ✔ |

[2] KÜVETTEN-TEST
[2] TEST EN CUVE
[2] CUVETTE-TEST
[2] KUVETTENTEST
[2] CUVETTE TEST

[3] BARCODE-PROGRAMME
[3] PROGR. CODE BARRE
[3] PROGRAMMI COD. A BARRE
[3] BARCODEPROGRAMMA'S
[3] BARCODE PROGRAMS

## Auswertung / Evaluation / Lettura / Meting

| | Filter ❶<br>Filtre<br>Filtro<br>Filter<br>Filter | Eprom ❷ | Test ❸<br>- anwählen<br>- choisir<br>- selezionare<br>- oproepen<br>- select | KontrollNr. ❹<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette | Zum Ergebnis addieren: ❻<br>Additionner au résultat:<br>Addizionare al risultato:<br>Bij het resultaat optellen:<br>Add to the result: |
|---|---|---|---|---|---|---|
| **LASA aqua** | ***$NO_2$-N:*** ○ 341 N ***/ $NO_2$:*** □ 341 | _ : 48 | ***$NO_2$-N:*** ○ 341 N ***/ $NO_2$:*** □ 341 | -- | ✔ | ***$NO_2$-N:*** 0.027 ***/ $NO_2$:*** 0.089 |
| **LASA 1 / plus** | 540 nm | -- | NO2-N / NO2 LCK 341 | 3 | ✔ | ***$NO_2$-N:*** 0.027 ***/ $NO_2$:*** 0.089 |
| **LASA 10** | -- | 11 : 48 / 99 : 48 | NO2-N / NO2 LCK 341 | 3 | ✔ | ***$NO_2$-N:*** 0.092 ***/ $NO_2$:*** 0.304 |
| **LASA 10** | -- | 98 : 48 | NO2-N / NO2 LCK 341 | 3 | ✔ | ***$NO_2$-N:*** 0.033 ***/ $NO_2$:*** 0.109 |
| **LASA 20** | -- | _ : 48 | NO2-N / NO2 LCK 341 | 3 | ✔ | ***$NO_2$-N:*** 0.033 ***/ $NO_2$:*** 0.109 |

| | Mode ❶ | Symbol ❷<br>Symbole<br>Simbolo<br>Symbool<br>Symbol | KontrollNr. ❸<br>No. de contrôle<br>No. di controllo<br>Controlegetal<br>Control no. | Leerwert (dest. Wasser) ❹<br>Valeur à blanc (l'eau dist.)<br>Bianco (acqua dist.)<br>Blanko (gedest. water)<br>Blank-value (dist. water)   | Analysenküvette ❺<br>Cuve d'analyse<br>Cuvetta d'analisi<br>Analyse-kuvet<br>Sample cuvette   |
|---|---|---|---|---|---|
| **CADAS 100 LPG158** | TEST | ***$NO_2$-N:*** $ 341 N ***/ $NO_2$:*** $ 341 | -- | LCW 919 | ✔ |
| **CADAS 100 LPG210** | TEST | ***$NO_2$-N:*** 341 N ***/ $NO_2$:*** 341 | 5 | LCW 919 | ✔ |

REF 985 019

en

# Test 0-19 12.14
# *NANOCOLOR*® Chloride 200

**Method:**
Photometric determination with mercury(II) thiocyanate and iron(III) nitrate

| | |
|---|---|
| Range: | **5–200 mg/L $Cl^-$** |
| Factor: | **not linear** |
| Wavelength (HW = 5–12 nm): | **470 nm** |
| Reaction time: | **3 min (180 s)** |
| Reaction temperature: | **20–25 °C** |

**Contents of reagent set:**
20 test tubes Chloride 200
2 test tubes with 11 mL Chloride 200 R2
1 test tube with blank value “NULL”

**Hazard warning:**
Test tubes contain nitric acid 5–20 %, reagent R2 contains mercury(II) thiocyanate 0.32–0.64 % in methanol 50–100 %.
H301, H311, H314, H331, H370 Toxic if swallowed. Toxic in contact with skin. Causes severe skin burns and eye damage. Toxic if inhaled. Causes damage to organs.
P260, P264, P270, P280, P301+310, P301+330+331, P302+352, P303+361+353, P304+340, P305+351+338, P308+311, P361+364, P405, P501 Do not breathe vapors. Wash with water thoroughly after handling. Do not eat, drink or smoke when using this product. Wear protective gloves / eye protection. IF SWALLOWED: Immediately call a POISON CENTER / doctor / … IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN: Wash with plenty of water / … IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water / shower. IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. IF exposed or concerned: Call a POISON CENTER / doctor / … Take off immediately all contaminated clothing and wash it before reuse. Store locked up. Dispose of contents / container to regulated waste treatment. For further information ask for a safety data sheet.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Chloride (500–3000 mg/L $Cl^-$, REF 913 21) or with *VISOCOLOR*® *HE* Chloride CL 500 (REF 915 004) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly.

**Interferences:**
Thiocyanate, sulfide, thiosulfate, bromide and iodide all interfere, since they react in the same way as chloride. A fluoride concentration in excess of 20 mg/L interferes with the chloride determination, and the concentrations read off are lower than those actually present in the test sample.

The method can also be applied for the analysis of sea water after dilution (1:200).

**Note:**
For the determination of chloride up to 1.00 g/L $Cl^-$ please contact MACHEREY-NAGEL for special working instructions.

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**1.0 mL** sample solution *(the pH value of the sample must be between pH 1 and 13)* and
**1.0 mL** R2, close and mix.
Clean outside of test tube and measure after 3 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-19.

**Measurement when samples are colored or turbid:**
For *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify calibration curve for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Multistandard Metals 1 (REF 925 015)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***

PD 14122 / A011691 / 985 019 / 1244

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 311

**1–70 mg/L Cl I**
**70–1000 mg/L Cl II**

Houdbaarheid
Storage
+2 °C ..... +8 °C

**T1**

| | |
|---|---|
| **1000 mg/L:** | $SO_4^{2-}$, $NO_3^-$ |
| **50 mg/L:** | $Pb^{2+}$, $Zn^{2+}$, $Ni^{2+}$, $Cu^{2+}$, $Cr^{3+}$, $Cr^{6+}$ |
| **10 mg/L:** | $Cd^{2+}$ |
| **0.4 mg/L:** | $CN^-$, $S^{2-}$ |

(I): ***Cl I – Chloride Meetbereik I***
***Cl I – Chloride Measuring range I***

(II): ***Cl II – Chloride Meetbereik II***
***Cl II – Chloride Measuring range II***

## *Datatabel · Data table*

***LP2W*** ***10/2011***

***Cl I*** • *λ: 470 nm • F1 = 0 • F2 = 110.3 • K = 1.82*
***Cl II*** • *λ: 470 nm • F1 = 0 • F2 = 880.4 • K = -9.05*

***CADAS 30/30S/50/50S*** ***10/2011***

***Cl I*** • *λ: 468 nm • Pro : 8 • F1 = -93.26 • F2 = 108.4 • K = -0.375*
***Cl II*** • *λ: 468 nm • Pro : 8 • F1 = -876.0 • F2 = 876.0 • K = -15.33*

***ISIS 6000/9000*** ***10/2011***

***Cl I*** • *λ: 455 nm • Pro: 8 • F1 = -95.46 • F2 = 111.0 • K = -0.25*
***Cl II*** • *λ: 455 nm • Pro: 8 • F1 = -907.6 • F2 = 907.6 • K = -16.7*

***CADAS 100/LPG 240*** ***10/2011***

***Cl I*** • *λ: 468 nm • Pro : 1 • F = 108.9 • K = 1.58*
***Cl II*** • *λ: 468 nm • Pro : 1 • F = 865.7 • K = -18.94*

***CADAS 200*** ***10/2011***

***Cl I*** • *L1W1.(M.E1W1) • C1= (E1-(0.86*L1))*F1-F2 •*
*W1=468 nm • F1 = 108.8 • F2 = 0.096*
***Cl II*** • *L1W1.(M.E1W1) • C1= (E1-L1)*F1-F2 •*
*W1=468 nm • F1 = 871.8 • F2 = 10.4*

***DR2800/3800/3900/5000/6000:*** ***03/2012***

***NL/EN: www.hach-lange.com ➔ LCK311 ➔ Download ➔ Software***

**NL** **LCK 311 Chloride**

***Let a.u.b. op de "Uitgave datum" (zie datatabel). Veiligheidsadvies en houdbaarheidsdatum op de verpakking.***

**Principe**
Bij het omzetten van chloride-ionen met kwikthiocyanaat ontstaat het nauwelijks gedissocieerde kwik(II)-chloride. Tegelijkertijd komt er een equivalente hoeveelheid thiocyanaat-ionen vrij, die met ijzer(III)-zouten tot ijzer(III)-thiocyanaat reageren.

**Toepassingsgebied**
Afvalwater, drinkwater, oppervlaktewateren, toevoerketelwater, procesanalyse, constructiebeton

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.

Zilver stoort doordat zilverchloride neerslaat (resultaat te laag). Kwik verhindert de reactie (resultaat te laag). Bromiden en jodiden, die met name in talrijke soorten mineraalwater voorkomen, leiden tot dezelfde reactie (resultaat te hoog). Stoffen die met ijzer(III)-zouten gekleurde produkten vormen, zorgen eveneens voor een storing in de gemeten waarden.
De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaardadditie).

**pH-waarde** monster ..... 3–10
**Temperaturen** monster/reagentia ..... 15–25 °C

**EN** **LCK 311 Chloride**

***Please check the "Edition Date" (see data table). Safety advice and expiry date on package.***

**Principle**
During the reaction of chloride ions with mercury thiocyanate the slightly dissociated mercury(II) chloride is formed. Simultaneously an equivalent amount of thiocyanate ions are set free, which react with iron(III) salts to form iron(III) thiocyanate.

**Range of Application**
Waste water, drinking water, surface water, boiler feed water, process analysis, structural concrete

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions.

Silver interferes due to the precipitation of silver chloride (low-bias results). Mercury hinders the reaction (low-bias results). Bromides and iodides, which are found in particular in many mineral waters, undergo the same reaction (high-bias results). Substances which form coloured complexes with iron(III) salts interfere with the determination.
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**pH** sample ..... 3–10
**Temperature** sample/reagents ..... 15–25 °C

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 311

**1–70 mg/L Cl I**
**70–1000 mg/L Cl II**

Lagerhinweis
Stabilité
Conservazione
+2 °C ..... +8 °C

**T1**

| | |
|---|---|
| **1000 mg/L:** | $SO_4^{2-}$, $NO_3^-$ |
| **50 mg/L:** | $Pb^{2+}$, $Zn^{2+}$, $Ni^{2+}$, $Cu^{2+}$, $Cr^{3+}$, $Cr^{6+}$ |
| **10 mg/L:** | $Cd^{2+}$ |
| **0.4 mg/L:** | $CN^-$, $S^{2-}$ |

(I): ***Cl I – Chlorid Messbereich I***
***Cl I – Chlorure Gamme de mesure I***
***Cl I – Cloruri Campo di misura I***

(II): ***Cl II – Chlorid Messbereich II***
***Cl II – Chlorure Gamme de mesure II***
***Cl II – Cloruri Campo di misura II***

## *Datentabelle · Table des données · Tabella dati*

***LP2W*** ***10/2011***

***Cl I*** • *λ: 470 nm • F1 = 0 • F2 = 110.3 • K = 1.82*
***Cl II*** • *λ: 470 nm • F1 = 0 • F2 = 880.4 • K = -9.05*

***CADAS 30/30S/50/50S*** ***10/2011***

***Cl I*** • *λ: 468 nm • Pro : 8 • F1 = -93.26 • F2 = 108.4 • K = -0.375*
***Cl II*** • *λ: 468 nm • Pro : 8 • F1 = -876.0 • F2 = 876.0 • K = -15.33*

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***Cl I*** • *λ: 455 nm • Pro: 8 • F1 = -95.46 • F2 = 111.0 • K = -0.25*
***Cl II*** • *λ: 455 nm • Pro: 8 • F1 = -907.6 • F2 = 907.6 • K = -16.7*

***CADAS 100/LPG 240*** ***10/2011***

***Cl I*** • *λ: 468 nm • Pro : 1 • F = 108.9 • K = 1.58*
***Cl II*** • *λ: 468 nm • Pro : 1 • F = 865.7 • K = -18.94*

***CADAS 200*** ***10/2011***

***Cl I*** • *L1W1.(M.E1W1) • C1= (E1-(0.86*L1))*F1-F2 •*
*W1=468 nm • F1 = 108.8 • F2 = 0.096*
***Cl II*** • *L1W1.(M.E1W1) • C1= (E1-L1)*F1-F2 •*
*W1=468 nm • F1 = 871.8 • F2 = 10.4*

***DR2800/3800/3900/5000/6000:*** ***03/2012***

***DE/IT: www.hach-lange.com ➔ LCK311 ➔ Download ➔ Software***
***FR: www.hach-lange.com ➔ LCK311 ➔ Télécharger ➔ Logiciel***

**DE** **LCK 311 Chlorid**

***Bitte "Ausgabedatum" (s. Datentabelle) beachten.***
***Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Bei Umsetzung von Chloridionen mit Quecksilberthiocyanat entsteht das wenig dissoziierte Quecksilber(II)-chlorid. Gleichzeitig wird eine äquivalente Menge Thiocyanationen freigesetzt, die mit Eisen(III)- Salzen zu Eisen(III)-thiocyanat reagieren.

**Anwendungsbereich**
Abwasser, Trinkwasser, Oberflächenwasser, Kesselspeisewasser, Prozessanalytik, Bauwerksbeton

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.

Silber stört durch Ausfällung von Silberchlorid (Minderbefund). Quecksilber verhindert die Reaktion (Minderbefund). Bromide und Jodide, wie sie besonders in vielen Mineralwässern vorkommen, gehen die gleiche Reaktion ein (Mehrbefund). Stoffe, die mit Eisen(III)-Salzen farbige Komplexe bilden, stören die Bestimmung. Messergebnisse sind durch eine Plausibilitätskontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**pH-Wert** Probe ..... 3–10
**Temperatur** Probe/Reagenzien ..... 15–25 °C

**FR** **LCK 311 Chlorure**

***Vérifier la date d'édition (voir table des données).***
***Conseils de securité et date de péremption sur l'emballage.***

**Principe**
La réaction d'ions chlorure avec du thiocyanate de mercure donne du chlorure mercurique(II) peu dissocié. Il y a simultanément libération d'une quantité équivalente d'ions thiocyanate qui forment avec des sels ferriques(III) du thiocyanate ferrique(III).

**Domaine d'application**
Eaux de rejet, eaux potables, eaux de surface, eaux d'alimentation de chaudières, analyses en mode continu, béton de construction

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.

L'argent gêne la détermination par précipitation de chlorure d'argent (résultat trop faible). Le mercure perturbe la réaction (résultat trop faible). Les bromures et les iodures, contenus en particulier dans de nombreuses eaux minérales, réagissent de la même façon (résultat trop élevé). Les substances formant des complexes colorés en présence de sels ferriques(III) gênent la détermination.
Les résultats des mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**pH** échantillon ..... 3–10
**Température** échantillon/réactifs ..... 15–25 °C

**IT** **LCK 311 Cloruri**

***Si prega di verificare la "Data di Edizione" (vedi tabella dati).***
***Avvertenze e data di scadenza sulla confezione.***

**Principio**
Trattando soluzioni contenenti cloruri con tiocianato di mercurio si forma il poco dissociato cloruro di mercurio(II). Allo stesso momento si libera una quantità equivalente di ioni di tiocianato che formano in presenza di sali ferrici(III) il tiocianato ferrico(III).

**Applicazione**
Acqua potabile, acque di superficie, acque di scarico, acque di caldaia, analisi di processo, cemento armato

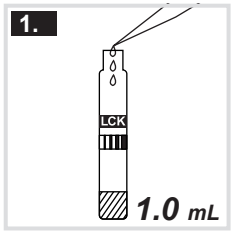
**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.

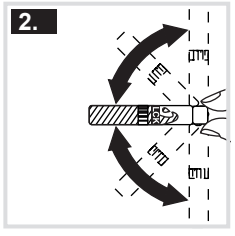
L'argento disturba la determinazione con la precipitazione di cloruri d'argento (valori ridotti) e la presenza di mercurio impedisce la reazione (valori ridotti). Bromuri e ioduri, presenti in molte acque minerali, danno reazioni analoghe e portano a valori in eccesso. Disturbano le sostanze che legati a sali ferrici(III) danno dei composti colorati.
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

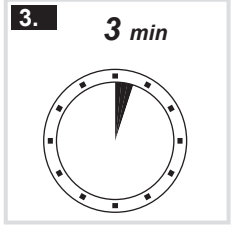
**pH** campione ..... 3–10
**Temperatura** campione/reagenti ..... 15–25 °C

AD 311 M / Druckfarbe schwarz / 1

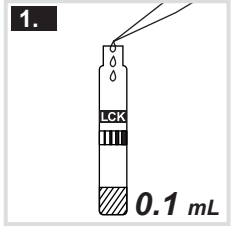
### 1–70 mg/L – (I)

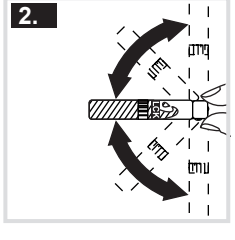


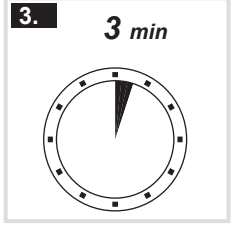




### 70–1000 mg/L – (II)




**DE**

**1–70 mg/L – Messbereich (I)**

1. ***1.0 mL*** Probe pipettieren.
2. Küvette verschließen und schwenken.
3. Nach ***3 min*** Küvette außen gut säubern und auswerten.

**70–1000 mg/L – Messbereich (II)**

1. ***0.1 mL*** Probe pipettieren.
2. Küvette verschließen und schwenken.
3. Nach ***3 min*** Küvette außen gut säubern und auswerten.

**FR**

**1–70 mg/L – Gamme de mesure (I)**

1. Pipetter ***1.0 mL*** d'échantillon.
2. Fermer la cuve et mélanger le contenu en la retournant plusieurs fois de suite.
3. Attendre ***3 min***, bien nettoyer l'extérieur de la cuve et mesurer.

**70–1000 mg/L – Gamme de mesure (II)**

1. Pipetter ***0.1 mL*** d'échantillon.
2. Fermer la cuve et mélanger le contenu en la retournant plusieurs fois de suite.
3. Attendre ***3 min***, bien nettoyer l'extérieur de la cuve et mesurer.

**IT**

**1–70 mg/L – Campo di misura (I)**

1. Pipettare ***1.0 mL*** di campione.
2. Tappare la cuvetta e mescolare.
3. Dopo ***3 min*** pulire bene la cuvetta esternamente e leggere.

**70–1000 mg/L – Campo di misura (II)**

1. Pipettare ***0.1 mL*** di campione.
2. Tappare la cuvetta e mescolare.
3. Dopo ***3 min*** pulire bene la cuvetta esternamente e leggere.

**NL**

**1–70 mg/L – Meetbereik (I)**

1. ***1.0 mL*** monster pipetteren.
2. Kuvet sluiten en zwenken.
3. Na ***3 min*** het kuvet van buiten goed reinigen en meten.

**70–1000 mg/L – Meetbereik (II)**

1. ***0.1 mL*** monster pipetteren.
2. Kuvet sluiten en zwenken.
3. Na ***3 min*** het kuvet van buiten goed reinigen en meten.

**EN**

**1–70 mg/L – Measuring range (I)**

1. Pipette ***1.0 mL*** sample.
2. Close cuvette and invert a few times.
3. After ***3 min*** thoroughly clean the outside of the cuvette and evaluate.

**70–1000 mg/L – Measuring range (II)**

1. Pipette ***0.1 mL*** sample.
2. Close cuvette and invert a few times.
3. After ***3 min*** thoroughly clean the outside of the cuvette and evaluate.

***Auswertung · Evaluation · Lettura · Meting***

**DE:** *Für folgende Barcode-Geräte erfolgt nach Einsetzen der Nulllösung eine automatische Auswertung:*

**FR:** *Si vous utilisez un des instruments avec codes à barres suivants, une évaluation automatique est réalisée après l'insertion de la Solution zéro :*

**IT:** *Se si utilizza uno qualsiasi dei seguenti strumenti con codice a barre, dopo aver inserito la bianco viene automaticamente visualizzato il risultato della misura:*

**NL:** *Wanneer een van de volgende barcode instrumenten worden gebruikt, wordt een automatische uitwaardering uitgevoerd zodra de nulkuvet geplaatst wordt:*

**EN:** *If any of the following barcode instruments is used, an automatic evaluation is carried out after the zero solution is inserted:*

## LASA 50 / 100, XION 500, CADAS 30 / 50 / 30S / 50S / 200 Barcode, ISIS 9000, DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

| DE | FR | IT | NL | EN | ⬇ | CADAS 200 Basis | ISIS 6000 | DR 1900 | LP2W | CADAS 100 LPG158 | CADAS 100 LPG210 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Filter | Filtre | Filtro | Filter | Filter | 1 | – | – | – | 470 nm | – | – |
| Eprom | Eprom | Eprom | Eprom | Eprom | 2 | _ : 50 | _ : 50 | – | – | – | – |
| Mode | Mode | Mode | Mode | Mode | 3 | – | [1] | [2] | – | TEST | TEST |
| Symbol | Symbole | Simbolo | Symbool | Symbol | 4 | – | – | – | – | [4] | [4] |
| Test anwählen | Test choisir | Test selezionare | Test oproepen | Test select | 5 | 311 | 311 | 311 | [3] | – | – |
| Faktor | Facteur | Fattore | Factor | Factor | 6 | – | – | – | – | – | – |
| Kontrollnr. | No. de contrôle | No. di controllo | Controlegetal | Control no. | 7 | 7 | 7 | 6 | 5 | – | 7 |
| Nulllösung | Solution zéro | Bianco | Nulkuvet | Zero-solution | 8 | – | – | – | ✓ NULL | ✓ NULL | ✓ NULL |
| Analysenküvette | Cuve d'analyse | Cuvetta d'analisi | Analyse-kuvet | Sample cuvette | 9 | – | – | – | ✓ ERGEBNIS | ✓ MESS | ✓ MESS |
| Nulllösung, blaue Taste / Null ▲ | Solution zéro, touche bleue / Zéro ▲ | Bianco, tasto blu / Zero ▲ | Nulkuvet, blauwe toets / Nulstellen ▲ | Zero-solution, blue key / Zero ▲ | 10 | ✓ | ✓ | ✓ | – | – | – |
| Analysenküvette, grüne Taste / Messen | Cuve d'analyse, touche verte / Mesurer | Cuvetta d'analisi, tasto verde / Lettura | Analyse-kuvet, groene toets / Meten | Sample cuvette, green key / Read | 11 | ✓ | ✓ | ✓ | – | – | – |

**DE:** [1] KÜVETTEN-TEST
**FR:** [1] TEST EN CUVE
**IT:** [1] CUVETTE-TEST
**NL:** [1] KUVETTENTEST
**EN:** [1] CUVETTE TEST

**DE:** [2] BARCODE-PROGRAMME
**FR:** [2] PROGR. CODE BARR
**IT:** [2] PROGRAMMI COD.A BARRE
**NL:** [2] BARCODE-PROGRAMMA'S
**EN:** [2] BARCODE PROGRAMS

**DE:** [3] Chlorid LCK 311 / **Cl II:** TEST
**FR:** [3] Chlorure LCK 311 / **Cl II:** TEST
**IT:** [3] Cloruri LCK 311 / **Cl II:** TEST
**NL:** [3] Chloride LCK 311 / **Cl II:** TEST
**EN:** [3] Chloride LCK 311 / **Cl II:** TEST

**DE:** [4] **Chlorid**: 311 / **Chlorid II**: 311 B
**FR:** [4] **Chlorure**: 311 / **Chlorure II**: 311 B
**IT:** [4] **Cloruri**: 311 / **Cloruri II**: 311 B
**NL:** [4] **Chloride**: 311 / **Chloride II**: 311 B
**EN:** [4] **Chloride**: 311 / **Chloride II**: 311 B

AD 311 M / Druckfarbe schwarz / 2

REF 985 086

en

# Test 0-86 07.14
# *NANOCOLOR*® Sulfate 200

**Method:**
Photometric determination as barium sulfate

| | |
|---|---|
| Range: | **10–200 mg/L $SO_4^{2-}$** |
| Accuracy: | **±10 %** at 100 mg/L |
| Factor: | **non linear** |
| Wavelength (HW = 5–12 nm): | **436 nm** |
| Reaction time: | **2 min (120 s)** |
| Reaction temperature: | **20–25 °C** |

**Contents of reagent set:**
20 test tubes Sulfate 200
1 bottle with 5 g Sulfate 200 R2
1 measuring spoon 85 mm

**Hazard warning:**
Reagent R2 contains barium chloride 25–83 %.
H301 Toxic if swallowed.
P301+310, P330, P405, P501 IF SWALLOWED: Immediately call a POISON CENTER / doctor / … Rinse mouth. Store locked up. Dispose of contents / container to regulated waste treatment. For further information please ask for a safety data sheet.

**Preliminary tests:**
If the order of magnitude of the concentration in a sample is not known, a preliminary test with QUANTOFIX® Sulfate (REF 913 29) or with *VISOCOLOR® ECO* Sulfate (REF 931 092) rapidly gives this information. From the order of magnitude the required dilution can be calculated and prepared directly.

**Interferences:**
Turbidities of sample interfere and test sample must first be filtered before the determination. In drinking, surface and ground water the test results are accurate. In polluted waste water the test result can be smaller than the real concentration.

The method can not be applied for the analysis of sea water.

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**4.0 mL** test sample *(the pH value of the sample must be between pH 1 and 13)*, close and mix.
Place test tube in photometer as blank value, adjust to zero.
Open test tube again, add
**1 level spoon** R2, close and **immediately** after addition shake vigorously for **10 s**.
Clean outside of test tube and measure after 2 min.

**Measurement:**
For *NANOCOLOR*® photometers and PF-12 see manual, test 0-86.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify calibration for each type of instrument by measuring standard solutions.

**Analytical quality control:**
*NANOCONTROL* Multistandard Metals 1 (REF 925 015) or Multistandard Drinking water (REF 925 018)

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***

PD 14122 / A011705 / 985 086 / 0744

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 153

## 40–150 mg/L $SO_4$

Houdbaarheid
Storage
+15 °C ..... +25 °C

### Datatabel · Data table

| | |
|---|---|
| ***LP2W*** | ***06/1990*** |
| ***$SO_4$ • F1 = 0 • F2 = 165 • K = 0*** | |
| ***CADAS 30/30S/50/50S*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • Pro.: 1 • F1 = 0 • F2 = 255.6 • K = -24.8*** | |
| ***ISIS 6000/9000*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • Pro.: 1 • F1 = 0 • F2 = 121.8 • K = -0.652*** | |
| ***CADAS 100/LPG 158*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • F = 194*** | |
| ***CADAS 100/LPG 210*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • F1 = 194*** | |

NL

### LCK 153 Sulfaat

***Let a.u.b. op de "Uitgave datum" (zie datatabel). Veiligheidsadvies en houdbaarheidsdatum op de verpakking.***

**Principe**
Sulfaat-ionen reageren in waterige oplossing met bariumchloride, waarbij het moeilijk oplosbare bariumsulfaat ontstaat. De troebeling die daarbij ontstaat, wordt met de fotometer geanalyseerd.

**Toepassingsgebied**
Afvalwater, grond, ongezuiverd water, drinkwater, constructiebeton, procesanalyse

**Storingen**
De meetresultaten zijn via een plausibiliteitsonderzoek te controleren (verdunning en/of standaardadditie).

**pH-waarde** monster ..... 3–10
**Temperaturen** monster/reagentia ..... 15–25 °C

EN

### LCK 153 Sulphate

***Please check the "Edition Date" (see data table). Safety advice and expiry date on package.***

**Principle**
Sulphate ions react with barium chloride in aqueous solution to form barium sulphate, which is only sparingly soluble. The resulting turbidity is measured photometrically.

**Range of Application**
Waste water, soils, raw water, drinking water, structural concrete, process analysis

**Interferences**
The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**pH** sample ..... 3–10
**Temperature** sample/reagents ..... 15–25 °C

HACH LANGE
UNITED FOR WATER QUALITY

# LCK 153

**40–150 mg/L $SO_4$**

Lagerhinweis
Stabilité
Conservazione
+15 °C ..... +25 °C

## Datentabelle · Table des données · Tabella dati

| | |
|---|---|
| ***LP2W*** | ***06/1990*** |
| ***$SO_4$ • F1 = 0 • F2 = 165 • K = 0*** | |
| ***CADAS 30/30S/50/50S*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • Pro.: 1 • F1 = 0 • F2 = 255.6 • K = -24.8*** | |
| ***ISIS 6000/9000*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • Pro.: 1 • F1 = 0 • F2 = 121.8 • K = -0.652*** | |
| ***CADAS 100/LPG 158*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • F = 194*** | |
| ***CADAS 100/LPG 210*** | ***06/1990*** |
| ***$SO_4$ • λ: 430 nm • F1 = 194*** | |

## DE LCK 153 Sulfat

***Bitte "Ausgabedatum" (s. Datentabelle) beachten.***
***Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Sulfationen reagieren mit Bariumchlorid in wässrigen Lösungen zum schwerlöslichen Bariumsulfat. Die dadurch hervorgerufene Trübung wird photometriert.

**Anwendungsbereich**
Abwasser, Boden, Rohwasser, Trinkwasser, Bauwerkbeton, Prozessanalytik

**Sulfat in Zement**
Für diese spezielle Auswerteform hat HACH LANGE eine Applikation ausgearbeitet, die Sie kostenlos bei HACH LANGE in Düsseldorf anfordern können.

**Störungen**
Messergebnisse sind durch eine Plausibilitäts-kontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**pH-Wert** Probe ..... 3–10
**Temperatur** Probe/Reagenzien ..... 15–25 °C

## FR LCK 153 Sulfate

***Vérifier la date d'édition (voir table des données).***
***Conseils de securité et date de péremption sur l'emballage.***

**Principe**
Les ions sulfate réagissent en solution aqueuse avec le chlorure de baryum pour donner du sulfate de baryum difficilement soluble. La turbidité en résultant est mesurée par photométrie.

**Domaine d'application**
Eaux de rejet, sols, eaux brutes, eaux potables, constructions en béton, analyses en mode continu

**Perturbations**
Les résultat de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**pH** échantillon..... 3–10
**Température** échantillon/réactifs ..... 15–25 °C

## IT LCK 153 Solfati

***Si prega di verificare la "Data di Edizione" (vedi tabella dati).***
***Avvertenze e data di scadenza sulla confezione.***

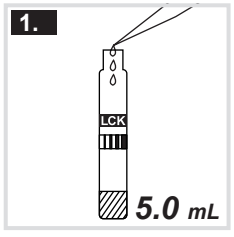
**Principio**
Ioni solfato formano con cloruro di bario in acqua un solfato di bario difficilmente solubile, la cui torbidità viene letta per via fotometrica.

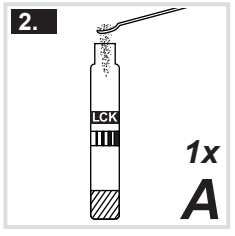
**Applicazione**
Acqua potabile, acqua grezza, acque di scarico, terreni, cemento armato, analisi di processo

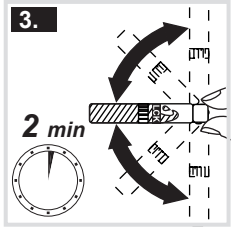
**Interferenze**
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

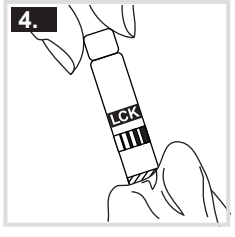
**pH** campione ..... 3–10
**Temperatura** campione/reagenti ..... 15–25 °C

AD 153 J / Druckfarbe burgund / 1




**DE**

1. ***5.0 mL*** Probe pipettieren.
2. ***1 Löffel*** Reagenz ***A*** (LCK 153 A) dosieren.
3. Küvette verschließen und ***sofort 2 min schwenken***.
4. Küvette außen gut säubern und auswerten.

**FR**

1. Pipetter ***5.0 mL*** d'échantillon.
2. Doser ***1 cuillère*** du réactif ***A*** (LCK 153 A).
3. Fermer la cuve et mélanger ***immediatement le contenu en la retournant plusieurs fois de suite pendant 2 min***.
4. Bien nettoyer l'extérieur de la cuve et mesurer.

**IT**

1. Pipettare ***5.0 mL*** di campione.
2. Aggiungere ***1 cucchiaino*** di reattivo ***A*** (LCK 153 A).
3. Tappare la cuvetta e ***subito mescolare per 2 min***.
4. Pulire bene la cuvetta esternamente e leggere.

**NL**

1. ***5.0 mL*** monster pipetteren.
2. ***1 doseerlepel*** reagens ***A*** (LCK 153 A) doseren.
3. Kuvet sluiten en ***onmiddellijk 2 min zwenken***.
4. Kuvet van buiten goed reinigen en meten.

**EN**

1. Pipette ***5.0 mL*** of sample.
2. Add ***1 dosing spoon*** reagent ***A*** (LCK 153 A).
3. Close cuvette and ***invert repeatedly for 2 min immediately***.
4. Thoroughly clean the outside of the cuvette and evaluate.

| | | | | |
|---|---|---|---|---|
| **DE:** *Für folgende Barcode-Geräte erfolgt nach Einsetzen der Analysenküvette eine automatische Auswertung:* | **FR:** *Si vous utilisez un des instruments avec codes à barres suivants, une évaluation automatique est réalisée après l'insertion de la cuve d'analyse :* | **IT:** *Se si utilizza uno qualsiasi dei seguenti strumenti con codice a barre, dopo aver inserito la cuvetta d'analisi viene automaticamente visualizzato il risultato della misura:* | **NL:** *Wanneer een van de volgende barcode instrumenten worden gebruikt, wordt een automatische uitwaardering uitgevoerd zodra de analyse-kuvet geplaatst wordt:* | **EN:** *If any of the following barcode instruments is used, an automatic evaluation is carried out after the sample cuvette is inserted:* |

**LASA 50 / 100, XION 500, CADAS 30 / 50 / 30S / 50S / 200 Barcode, ISIS 9000, DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000**

| DE | FR | IT | NL | EN | ⬇ | LASA aqua | LASA 1 / plus | LASA 20 | CADAS 200 Basis | ISIS 6000 | LASA 30 | DR 1900 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Filter | Filtre | Filtro | Filter | Filter | 1 | ☐ 153 | 440 nm | – | – | – | 440 nm | – |
| Eprom | Eprom | Eprom | Eprom | Eprom | 2 | _ : 12 | _ : 18 | _ : 32 | _ : 38 | _ : 32 | – | – |
| Mode | Mode | Mode | Mode | Mode | 3 | – | – | – | – | KÜVETTEN-TEST[1] | Dr. Lange | BARCODE-PROGRAMME[3] |
| Test anwählen | Test choisir | Test selezionare | Test oproepen | Test select | 4 | ☐ 153 | SO4 LCK 153 | SO4 LCK 153 | 153 | 153 | 153 | 153 |
| Kontrollnr. | No. de contrôle | No. di controllo | Controlegetal | Control no. | 5 | – | – | – | 7 | 7 | 7 | 7 |
| Analysenküvette | Cuve d'analyse | Cuvetta d'analisi | Analyse-kuvet | Sample cuvette | 6 | ✓ | ✓ | ✓ | – | – | – | – |
| Analysenküvette, grüne Taste / Messen | Cuve d'analyse, touche verte / Mesurer | Cuvetta d'analisi, tasto verde / Lettura | Analyse-kuvet, groene toets / Meten | Sample cuvette, green key / Read | 7 | – | – | – | ✓ | ✓ | ✓ | ✓ |

| DE | FR | IT | NL | EN | ⬇ | LP1W | LP2W | CADAS 100 LPG158 | CADAS 100 LPG210 |
|---|---|---|---|---|---|---|---|---|---|
| Filter | Filtre | Filtro | Filter | Filter | 1 | 435 nm | 435 nm | – | – |
| Mode | Mode | Mode | Mode | Mode | 2 | – | – | TEST | TEST |
| Symbol | Symbole | Simbolo | Symbool | Symbol | 3 | – | – | 153 | 153 |
| Test anwählen | Test choisir | Test selezionare | Test oproepen | Test select | 4 | – | Sulfat[2] LCK 153 | – | – |
| Faktor | Facteur | Fattore | Factor | Factor | 5 | 165 | – | – | – |
| Kontrollnr. | No. de contrôle | No. di controllo | Controlegetal | Control no. | 6 | – | 3 | – | 5 |
| Leerwert (Probe) | Valeur à blanc (échantillon) | Bianco (campione.) | Blanko (monster) | Blank-value (sample) | 7 | LCW 919 NULL | LCW 919 NULL | LCW 919 NULL | LCW 919 NULL |
| Analysenküvette | Cuve d'analyse | Cuvetta d'analisi | Analyse-kuvet | Sample cuvette | 8 | ✓ ERGEBNIS | ✓ ERGEBNIS | ✓ MESS | ✓ MESS |

**FR:**
[1] TEST EN CUVE
[2] SULFATE
[3] PROGR. CODE BARRE

**IT:**
[1] CUVETTE-TEST
[2] SOLFATI
[3] PROGRAMMI COD.A BARRE

**NL:**
[1] KUVETTENTEST
[2] SULFAAT
[3] BARCODE-PROGRAMMA'S

**EN:**
[1] CUVETTE TEST
[2] SULFATE
[3] BARCODE PROGRAMS

AD 153 J / Druckfarbe burgund / 2

REF 985073

en

# Test 0-73 06.17
# *NANOCOLOR*® Sulfide 3

**Method:**
Photometric determination as methylene blue

| | |
|---|---|
| Range: | **0.05–3.00 mg/L $S^{2-}$** |
| Wavelength (HW = 5–12 nm): | **620 nm** |
| Reaction time: | **10 min (600 s)** |
| Reaction temperature: | **20–25 °C** |

**Contents of reagent set:**
20 test tubes Sulfide 3
1 bottle with 1.5 g Sulfide 3 R2
1 test tube with 5 mL Sulfide 3 R3
1 measuring spoon 70 mm

**Hazard warning:**
Test tubes contain sulfuric acid 51–65 %, reagent R2 contains sulfamic acid 90–100 %.
H314 Causes severe skin burns and eye damage.
P260sh, P280sh, P303+361+353, P305+351+338, P310 Do not breathe dust / vapors. Wear protective gloves / eye protection. IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water [or shower]. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. For further information ask for a safety data sheet.

**Interferences:**
Sulfide concentration is tested in an acidic medium and, therefore, if the reagents are not mixed gently, some sulfide may escape as hydrogen sulfide, leading to lower test results.
The following ions will not interfere: < 100 mg/L $NO_3^-$, $NO_2^-$; < 20 mg/L $SCN^-$; < 10 mg/L $SO_3^{2-}$.

The method can be applied also for the analysis of sea water after dilution (1+3).

**Procedure:**
Requisite accessories: piston pipette with tips

Open test tube, add
**1 level measuring spoonful** of R2 and
**4.0 mL** test sample *(the pH value of the sample must be between pH 7 and 10)*, close and shake gently. Wait **1 min**.
Add
**200 µL** (= 0.2 mL) R3, close and mix.
Clean outside of test tube and measure after 10 min.

**Measurement:**
For MACHEREY-NAGEL photometers see manual, test 0-73.

**Measurement when samples are colored or turbid:**
For all *NANOCOLOR*® photometers see manual, use key for correction value.

**Photometers of other manufacturers:**
For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

**MACHEREY-NAGEL GmbH & Co. KG** · Neumann-Neander-Str. 6–8 · 52355 Düren · Germany
Tel.: +49 24 21 969-0 · Fax: +49 24 21 969-199 · info@mn-net.com · ***www.mn-net.com***
PD 14122 / A035100 / 985073 / 0670.6

HACH LANGE
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# LCK 653 0.1 – 2.0 mg/L

Lagerhinweis
Stabilité
Conservazione
Houdbaarheid
Storage
+15°C ..... +25°C

**T1**

**900 mg/L:** $S_2O_3^{2-}$, $SCN^-$
**700 mg/L:** $SO_3^{2-}$
**20 mg/L:** $I^-$
**2 mg/L:** $CN^-$

## *Datatabel / Data table*

| | |
|---|---|
| ***CADAS 30/30S/50/50S*** | ***07/2004*** |
| ***LCK 653*)*** • *λ: 666 nm • Pro.: 1 • F1 = 0 • F2 = 1.30 • K = -0.132* | |
| ***ISIS 6000/9000*** | ***07/2004*** |
| ***LCK 653*)*** • *λ: 695 nm • Pro.: 1 • F1 = 0 • F2 = 2.60 • K = -0.243* | |
| ***CADAS 200*** | ***07/2004*** |
| ***LCK 653*)*** • *E1W1 • C1 = E1∗F1-F2 • W1 = 665 nm • F1 = 1.29 • F2 = 0.133* | |

****) Sulfide***
***Sulphide***

**NL**

## **LCK 653 Sulfide** opgelost

***Let a.u.b. op de "Uitgave datum" (zie datatabel).***
***Veiligheidsadvies en houdbaarheids-datum op de verpakking.***

**Principe**
Dimethyl-p-fenylendiamine reageert met waterstofdisulfide tot een intermediaire verbinding die overgaat in leucomethyleenblauw.
Dit leucomethyleenblauw wordt door ijzer(III)-ionen geoxideerd tot methyleenblauw.

**Toepassingsgebied**
Afvalwater, ongezuiverd water

**Storingen**
De, in ***T1*** genoemde ionen, zijn tot aan de aangegeven concentratie afzonderlijk onderzocht en storen niet. De invloed van het cummulatief effect en invloed van andere ionen is niet door ons onderzocht.
De storingen zijn overgenomen uit DIN 38 405 D 26, pag. 1.

Indien geen vergelijkbaarheid met de referentie-methode verkregen wordt, dan raden wij u aan om een monstervoorbereiding analoog met de referentiemethode uit te voeren (bijvoorbeeld DIN 38405 D26 of D27).

De meetresultaten zijn via een plausibiliteitsonder-zoek te controleren (verdunning en/of standaard-additie).

**pH-waarde** monster.....3 – 10
**Temperatuur** monster/reagentia.....15 – 25°C

***De analyse moet onmiddellijk na de monstername worden uitgevoerd.***

**GB**

## **LCK 653 Sulphide** dissolved

***Please check the "Edition Date" (see data table).***
***Safety advice and expiry date on package.***

**Principle**
Dimethyl-p-phenylenediamine reacts with hydrogen sulphide to form an intermediate compound which turns into leucomethylene blue. The leucomethylene blue is oxidized to methylene blue by iron(III) ions.

**Range of Application**
Waste water, raw water

**Interferences**
The ions listed in ***T1*** have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions. This list of interferences has been taken from DIN 38 405 D 26 p. 1.

If there is no comparability to the reference method, we recommend the use of a sample preparation similar to that of the reference method (e. g. 38405 D26 or D27).

The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

**pH** sample.....3 – 10
**Temperature** sample/reagents .....15 – 25°C

***The analysis must be carried out immediately after the sample has been taken.***

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# LCK 653 0.1 – 2.0 mg/L

Lagerhinweis / Stabilité / Conservazione / Houdbaarheid / Storage: +15°C ..... +25°C

**T1**

**900 mg/L:** $S_2O_3^{2-}$, $SCN^-$
**700 mg/L:** $SO_3^{2-}$
**20 mg/L:** $I^-$
**2 mg/L:** $CN^-$

## *Datentabelle / Table des données / Tabella dati*

| | |
|---|---|
| ***CADAS 30/30S/50/50S*** | ***07/2004*** |
| ***LCK 653*)*** • *λ: 666 nm • Pro.: 1 • F1 = 0 • F2 = 1.30 • K = -0.132* | |
| ***ISIS 6000/9000*** | ***07/2004*** |
| ***LCK 653*)*** • *λ: 695 nm • Pro.: 1 • F1 = 0 • F2 = 2.60 • K = -0.243* | |
| ***CADAS 200*** | ***07/2004*** |
| ***LCK 653*)*** • *E1W1 • C1 = E1∗F1-F2 • W1 = 665 nm • F1 = 1.29 • F2 = 0.133* | |

****) Sulfid***
***Sulfure***
***Solfuri***

**D**

## LCK 653 Sulfid gelöst

***Bitte "Ausgabedatum" (s. Datentabelle) beachten.***
***Sicherheitshinweise und Verfallsdatum auf der Packung.***

**Prinzip**
Dimethyl-p-phenylendiamin reagiert mit Schwefelwasserstoff zu einer Zwischenverbindung, die in Leucomethylenblau übergeht. Das Leucomethylenblau wird durch Eisen(III)-Ionen zu Methylenblau oxidiert.

**Anwendungsbereich**
Abwasser, Rohwasser

**Störungen**
Die in ***T1*** aufgeführten Ionen wurden bis zu den angegebenen Konzentrationen einzeln überprüft und stören nicht. Die summarische Wirkung sowie der Einfluss weiterer Ionen wurden von uns nicht ermittelt.
Angabe der Störungen entnommen DIN 38 405 D 26 S. 1.

Wenn keine Vergleichbarkeit zum Referenzverfahren erreicht wird, empfehlen wir den Einsatz einer Probenvorbereitung analog zum Referenzverfahren (z. B. DIN 38405 D26 oder D27).

Messergebnisse sind durch eine Plausibilitäts-kontrolle zu überprüfen (Verdünnung und/oder Aufstockung).

**pH-Wert** Probe .....3 – 10
**Temperatur** Probe/Reagenzien .....15 – 25°C

***Die Analyse muss unmittelbar nach Probe-nahme erfolgen.***

**F**

## LCK 653 Sulfure dissous

***Vérifier la date d'edition (voir table des données).***
***Conseils de securité et date de péremption sur l'emballage.***

**Principe**
La diméthyl-p-phénylènediamine réagit avec l'hydrogène sulfuré et donne une substance intermédiaire qui se transforme en bleu de leucométhylène. Le bleu de leucométhylène est oxydé par les ions fer(III) en bleu de méthylène.

**Domaine d'application**
Eaux de rejet, eaux brutes

**Perturbations**
Les ions mentionnés dans ***T1*** ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires.
Mention des perturbations selon DIN 38 405 D 26 p. 1.

Si aucune comparaison avec la méthode de référence n'est obtenue, nous recommandons l'utilisation d'une préparation d'échantillon analogue à celle utilisée par la méthode de référence (par ex. DIN 38405 D26 ou D27).

Les résultats des mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).

**pH** échantillon .....3 – 10
**Température** échantillon/réactifs .....15 – 25°C

***L'analyse doit être réalisée immédiatement après la prise d'échantillon.***

**I**

## LCK 653 Solfuri disciolto

***Si prega di verificare la "Data di Edizione" (vedi tabella dati).***
***Avvertenze e data di scadenza sulla confezione.***

**Principio**
Dimetil-p-fenilendiammina forma con idrogeno solforato un componente intermedio che si trasforma in blu di "leuco". Questo viene poi ossidato con ioni ferrici(III) in blu di metilene.

**Applicazione**
Acque di scarico, acqua grezza

**Interferenze**
Gli ioni elencati in ***T1*** sono stati verificati singolarmente fino alle concentrazioni specificate e non causano interferenze. Non sono stati verificati eventuali effetti cumulativi e l'influenza di altri ioni.
Vedere DIN 38 405 D 26 pag. 1.

Se i risultati non sono comparabili con i risultati della metodica di riferimento, raccomandiamo l'utilizzo di un sistema di preparazione simile a quelli indicati nelle procedure di riferimento (DIN 38405 D26 o D27).

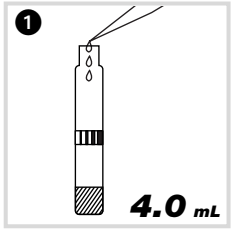
I risultati sono da verificare con un controllo (diluizione e/o soluzione additiva).

**pH** campione .....3 – 10
**Temperatura** campione/reagenti .....15 – 25°C

***Fare l'analisi subito dopo aver prelevato il campione.***

AD 653 E / Druckfarbe burgund / 1

**LCK 653** *07/2004*



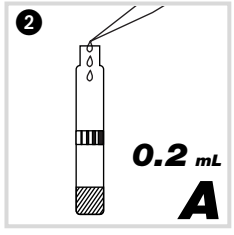

***4.0 mL*** Probe pipettieren.

Pipetter ***4.0 mL*** d'échantillon.

Pipettare ***4.0 mL*** di campione.

***4.0 mL*** monster pipetteren.

Pipette ***4.0 mL*** sample.



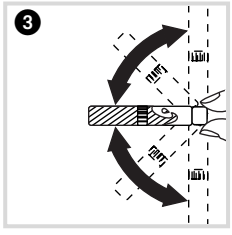

***0.2 mL*** Lösung LCK 653 ***A*** pipettieren.

Pipetter ***0.2 mL*** de la solution LCK 653 ***A***.

Pipettare ***0.2 mL*** di soluzione LCK 653 ***A***.

***0.2 mL*** oplossing LCK 653 ***A*** pipetteren.

Pipette ***0.2 mL*** solution LCK 653 ***A***.



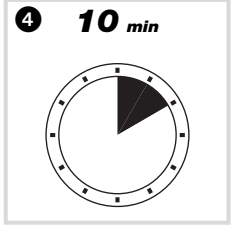

Küvette verschließen und schwenken.

Fermer la cuve et mélanger le contenu en la retournant plusieurs fois de suite.

Tappare la cuvetta e mescolare.

Kuvet sluiten en zwenken.

Close cuvette and invert a few times.




Nach ***10 min*** Küvette außen gut säubern und auswerten.

Attendre ***10 min***, bien nettoyer l'extérieur de la cuve et mesurer.

Dopo ***10 min*** pulire bene la cuvetta esternamente e leggere.

Na ***10 min*** het kuvet van buiten goed reinigen en meten.

After ***10 min*** thoroughly clean the outside of the cuvette and evaluate.

| | **Analysenküvette** ❶<br>**Cuve d'analyse**<br>**Cuvetta d'analisi**<br>**Analyse-kuvet**<br>**Sample cuvette** |
|---|---|
| IBR / IBR+ **Barcode** [1] | ✔ |

## *Auswertung / Evaluation / Lettura / Meting*

[1] LASA 50 / 100
XION 500
CADAS 30 / 50 / 30S / 50S / 200 Barcode
ISIS 9000
DR 2800 / DR 3800 / DR 3900 / DR 5000 / DR 6000

| | **Filter** ❶<br>**Filtre**<br>**Filtro**<br>**Filter**<br>**Filter** | **Eprom** ❷ | **Mode** ❸ | **Test** ❹<br>**- anwählen**<br>**- choisir**<br>**- selezionare**<br>**- oproepen**<br>**- select** | **KontrollNr.** ❺<br>**No. de contrôle**<br>**No. di controllo**<br>**Controlegetal**<br>**Control no.** | **Analysenküvette, grüne Taste / Messen** ❻<br>**Cuve d'analyse, touche verte / Mesurer**<br>**Cuvetta d'analisi, tasto verde / Lettura**<br>**Analyse-kuvet, groene toets / Meten**<br>**Sample cuvette, green key / Read** |
|---|---|---|---|---|---|---|
| **CADAS 200 Basis** | -- | _ : 50 | -- | 653 | 1 | ✔ |
| **ISIS 6000** | -- | _ : 50 | [2] | 653 | 1 | ✔ |
| **LASA 30** | 695 nm | _ : 50 | Dr. Lange | 653 | 1 | ✔ |
| **DR 1900** | -- | -- | [3] | 653 | 1 | ✔ |

| [2] KÜVETTEN-TEST | [3] BARCODE-PROGRAMME |
|---|---|
| [2] TEST EN CUVE | [3] PROGR. CODE BARRE |
| [2] CUVETTE-TEST | [3] PROGRAMMI COD. A BARRE |
| [2] KUVETTENTEST | [3] BARCODEPROGRAMMA'S |
| [2] CUVETTE TEST | [3] BARCODE PROGRAMS |

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OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2
Maria Eloisa Albacete Garcia

# ANNEX III. POSTER PUBLICATION

UNIVERSITAT ROVIRA I VIRGILI
OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2
Maria Eloisa Albacete Garcia

# Optimisation of wastewater treatment at Ecoparc 2

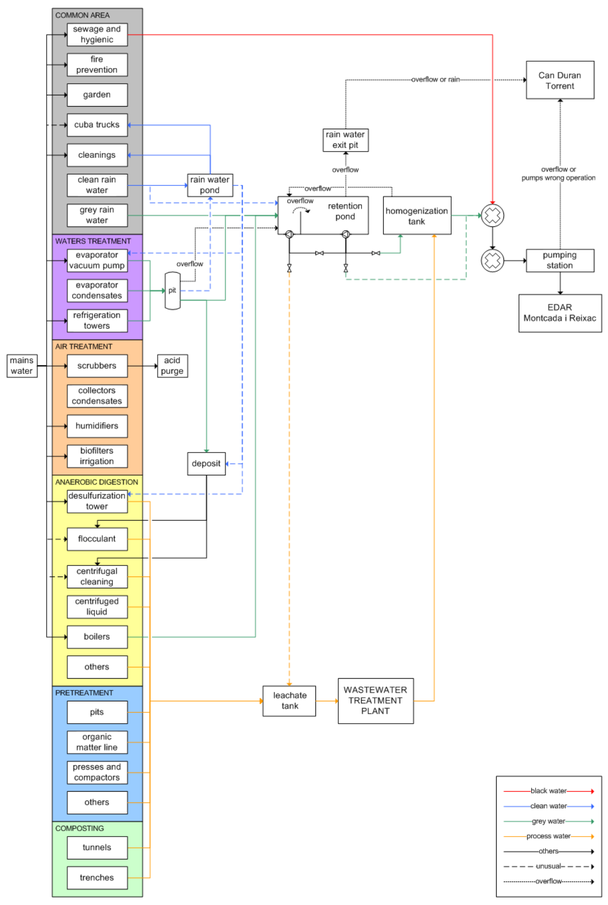
Eloísa Albacete[a,b], Josep Tàrraga[b], Inmaculada Sanz[a], and Allan D. Mackie[a]

[a]Departament d'Enginyeria Química, ETSEQ, Universitat Rovira i Virgili, avinguda Països Catalans, 26 , 43007 Tarragona, Spain
[b]Ecoparc del Besòs, avinguda Torre Mateu, s/n, 08110 Montcada i Reixac, Spain

## INTRODUCTION

Ecoparc 2 is a Urban Solid Waste (RSU) treatment plant managed by UTE Ebesa which belongs to the Barcelona Metropolitan Area (AMB) and is located in Montcada i Reixac.
In its facilities are treated the RSU fractions of packaging (yellow container), kitchen waste (brown container) and non recyclable mixed waste (grey container), with the objective of transforming waste into compost, biogas and recovering materials that have the potential to be recycled, in order to receive a previous treatment to their final destination.
It occupies an area of 11.7 ha and has a nominal capacity of 27,500 tons/year of packaging, 120,000 tons/year of kitchen waster and 160,000 tons/year of non recyclable mixed waste.
As a result of waste treatment, a large amount of leached liquids is obtained which also requires treatment before being discharged.

## PHASE 1. WASTEWATER CHARACTERISATION




Annually, Ecoparc 2 consumes about 70,000 $m^3$ of municipal water network and discharges approximately 50,000 $m^3$ to the collector from the municipal wastewater treatment plant in Montcada i Reixac.

In Ecoparc 2, four wastewater networks are distinguished with different origins and destinations.

- **BLACK WATER**
**From**: changing rooms, toilets, and sewage in general.
**To**: collector from the municipal wastewater treatment plant.

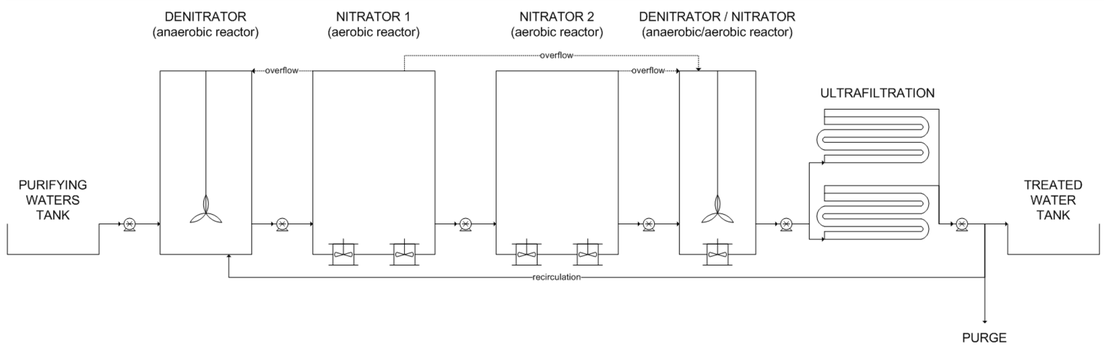
- **CLEAN WATER**
**From**: rain water collected from the installation covers which have not had contact with waste.
**To**: rain water pond for later use in the process, reducing the consumption of the municipal water network.

- **GREY WATER**
**From**: rain water collected from the ground and that may have been in contact with waste, and water collected from air treatment (condensates, humidifiers, and biofilters).
**To**: retention pond, if within legal limits discharges to collector, otherwise it is treated in the wastewater treatment plant.

- **PROCESS WATER**
**From**: water collected from waste treatment processes, which contains a high organic content and a high ammonium concentration.
**To**: wastewater treatment plant to later be discharged into the collector.

**OBJECTIVES**:
- To characterise all wastewater from Ecoparc 2 in order to be able to have a solid basis for the project, and to act on the most problematic areas (in terms of both concentration and quantity), for which the flow, ammonium concentration, COD, total solids and volatile solids of the maximum possible effluents are determined .
- To minimise the consumption of municipal water.
- To reuse any grey water exceeding legal limits, so the process water can pass and finish its journey in the wastewater treatment plant, and minimise the consumption of municipal water.

## PHASE 2. WASTEWATER TREATMENT PLANT OPTIMISATION




Ecoparc 2 has a wastewater treatment plant with MBR system (Membrane Bio Reactor) to treat basically the COD and the nitrogen such as ammonia, nitrites and nitrates.

Its initial design consists of a nominal capacity of 142 $m^3$/day with a COD of 10,000 mg/L, which consisted of three reactors:

- **DENITRATOR.** Reduction of nitrate to nitrogen gas consuming carbon compounds (COD) and $H^+$ ions, therefore making the medium more basic. Reactions:

$2\ NO_3^- + 2\ H^+ + 10\ [H] \rightarrow N_2 + 6\ H_2O$
$4\ NO_3^- + 4\ H^+ + 5\ [C] \rightarrow N_2 + 5\ CO_2 + 2\ H_2O$

- **NITRATOR**. Oxidation of ammonium to nitrite and nitrite to nitrate, releasing $H^+$ ions and thereby acidifying the medium. Reactions:

Nitritation: $2\ NH_4^+ + 3\ O_2 \rightarrow 2\ NO_2^- + 2\ H_2O + 4\ H^+$
Nitration: $2\ NO_2^- + O_2 \rightarrow 2\ NO_3^-$

- **COMBINED (DENITRATOR/NITRATOR).** It is possible to operate either as denitrator, using methanol (carbon source) and stopping the aeration (anoxic conditions), or as nitrator airing (oxic conditions). A supply of methanol as a carbon source is necessary for the denitrator reactions.

4 years later, it had to be expanded by installing a second nitrator reactor, so that it could handle 40,000 mg/L COD from the input.

Nowadays it is difficult to treat all process water discharged into the collector, without exceeding the legal allowable limits in COD (< 1,500 mg/L) and ammonium (< 60 mg/L). Also, only the inlet and outlet of the wastewater treatment plant is being analysed, regardless of what happens in the intermediate reactors, as if it were a black box, and it's necessary to review the analytical methods used.

**OBJECTIVES**:
- To review the analytical procedures and replace them with standardised methods consistent with the analysed waste.
- To change to track the wastewater treatment plant sampling point, analytical and its periodicity.
- To check the well operation of the wastewater treatment plant and optimise its performance.

## PHASE 3. NEW TECHNOLOGIES: HYDROLYSIS AND STRIPPING

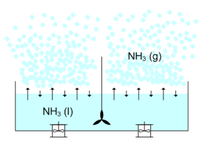
Approximately 75% of the process water entering the wastewater treatment plant comes from the dehydration of digested products from the anaerobic digestion process (centrifuged liquid). This effluent contains an organic loading rate of 40,000 mg/L COD and ammonium concentration of 5,000 mg/L.

This work intends to act on this effluent using two technologies for different purposes:

- **THERMAL HYDOLYSIS**. By the action of temperature the intention is to break the hydrocarbon chains to make the input matter more biodegradable and therefore obtain a carbon source more accessible to the biological processes of the wastewater treatment plant. The steam produced in the cogeneration process of the same facilities will be used as a heat source.

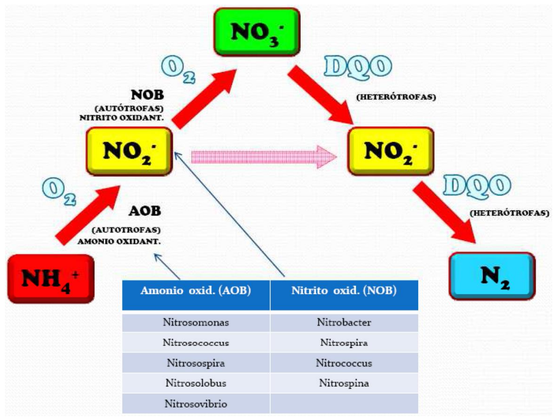

- **STRIPPING**. Leveraging the hydrolysis temperature, shaking and airing the effluent, to divert the ammonia equilibrium in the liquid phase to the gas phase.

$NH_3\ (l) \rightleftharpoons NH_3\ (g)$




**OBJECTIVES**:
- To increase the biodegradability of the organic matter contained in the effluent with more volumetric weight by thermal hydrolysis.
- To find the optimal temperature for thermal hydrolysis.
- To reduce the ammonia concentration in the liquid effluent that comes from anaerobic digestion by aeration and agitation (stripping).

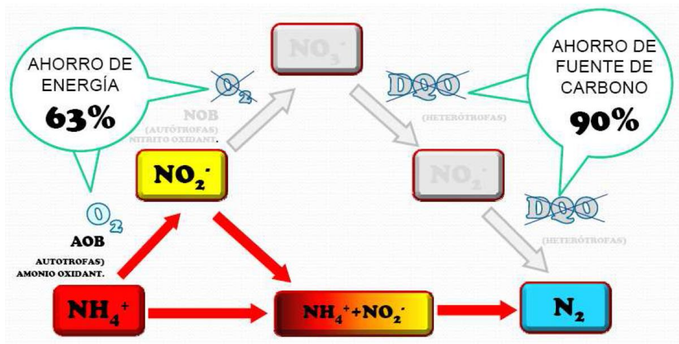
## PHASE 4. NEW BIOLOGICAL PROCESSES




Another existing alternative is a change in the biological process.

Up to now, the biology used in the wastewater treatment plant transforms ammonia nitrogen contained in nitrogen gas, firstly oxidising to nitrite, these to nitrate and, finally, reducing these to nitrogen gas.

It is planned to make a change in biology, so that nitrites are reduced to nitrogen gas without going through nitrates. In this way it is possible to achieve energy savings and a decrease in the needs of the carbon source, which result in a reduction of methanol consumption.




**OBJECTIVES**:
- To consider other alternatives for the process water treatment from Ecoparc 2.
- To achieve energy savings in water purifying.
- To reduce methanol consumption, previously required for water purifying in the current wastewater treatment plant.

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# ANNEX IV. METHAMORPHOSIS LIFE PROJECT

UNIVERSITAT ROVIRA I VIRGILI
OPTIMISATION OF WASTEWATER TREATMENT AT ECOPARC 2
Maria Eloisa Albacete Garcia


# PROYECTO LIFE METHAMORPHOSIS

Con la contribución del instrumento financiero LIFE de la Unión Europea

metha MORPHOSIS

## Tratamiento de aguas residuales para obtención de agua de reúso y biometano para el sector transporte mitigando las emisiones del gas invernadero

### Descripción del Proyecto

El proyecto LIFE+ Methamorphosis tiene como objetivo la valorización energética de residuos sólidos orgánicos de origen tanto municipal como agroganadero y la obtención de combustibles alternativos y sostenibles.

El primer objetivo consiste en la optimización energética de la depuración de las aguas procedentes del tratamiento de la fracción orgánica de los residuos sólidos urbanos mediante la implantación de innovadores procesos anaerobios y autótrofos aplicados en serie:

1) el sistema AnMBR (reactor anaerobio de membranas) y
2) el sistema ELAN® de eliminación autótrofa de nitrógeno.

La aplicación de este innovador tren de tratamiento en el ECOPARC2 de Barcelona permitirá maximizar la recuperación energética de los residuos urbanos y supondrá importantes mejoras medioambientales en el tratamiento de dichos residuos. Se espera una reducción del 70% en la demanda energética para el proceso de tratamiento y una reducción del 80% en las emisiones de $CO_2$ asociadas al mismo en comparación con la tecnología aerobia de membranas instalada actualmente.

En paralelo se implantarán sistemas novedosos basados en la absorción en agua (sistema ABAD en el ECOPARC2) y la utilización de membranas (sistema METHAGRO en una planta agroganadera) para la producción de biometano a partir de biogás y su utilización directa en el sector del

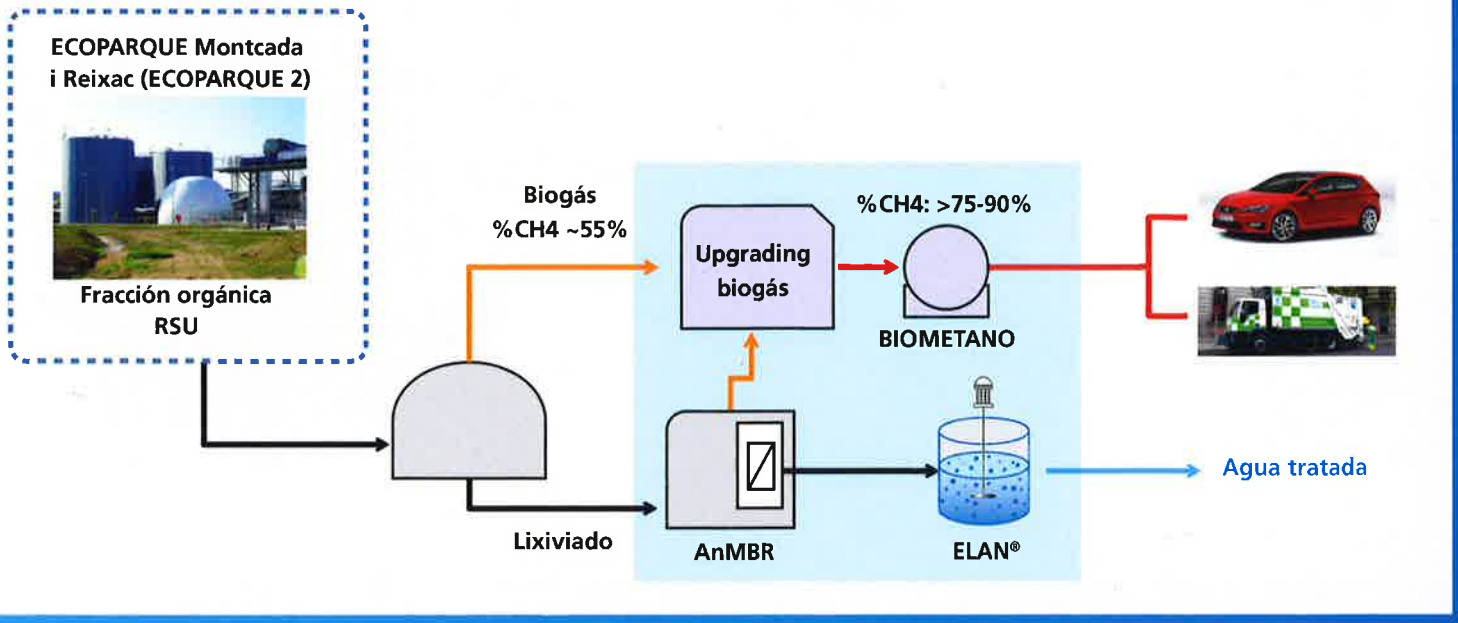
aqualia

**Ubicación:** Ecoparc2 de Barcelona

**Duración:** Del 16 de julio de 2015 al 30 de junio de 2019

**Presupuesto Total:** 3.642.167 € **Aqualia:** 837.049 €

## DIAGRAMA DE FLUJO DEL PROYECTO




transporte o bien su inyección en la red de distribución de gas natural. Las tecnologías que se demostrarán en instalaciones reales durante el proyecto contribuirán a la mejora de la calidad del aire, especialmente en zonas densamente pobladas, ya que las emisiones de un vehículo alimentado con biometano son inferiores a las emisiones de vehículos alimentados por gasolina (-25%) a nivel de $CO_2$ y una reducción muy considerable en emisiones contaminantes locales como óxidos de nitrógeno comparado con los motores diésel (-85%). El proyecto contribuirá al objetivo general sobre la transición hacia una economía eficiente en el uso de recursos y a la protección y mejora de la calidad del medioambiente. Tiene como objetivo específico contribuir al desarrollo y demostración de tecnologías, métodos e instrumentos de mitigación del cambio climático, y su escalado, transferencia o incorporación en otros sectores.

## ORGANIZACIONES PARTICIPANTES

- FCC Aqualia, S.A. (líder)
- FCC S.A.
- AMB
- Gas Natural
- ICAEN
- SEAT

## DETALLES DE LA AYUDA

**Ayuda:** Programa de Medioambiente y Acción por el Clima (LIFE): Subprograma Acción Climática (CCM).

**Organismo:** Comisión Europea (CE).

**Número expediente:** LIFE14/CCM/ES/000865

**Modalidad de la ayuda:** Subvención del 60% del presupuesto.

**Financiación recibida**

**Total:** 2.089.200 €

**Aqualia:** 837.049 €

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