



# ETV VERIFICATION REPORT

Final

Technology HYDRO-1

Report N° 21DGMP13

Revision N° 00



ISP N° 069 E Membro degli Accordi di Mutuo Riconoscimento EA, IAF e ILAC Signatory of EA, IAF and ILAC Mutual Recognition Agreements

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## 1. INTRODUCTION

RINA SERVICES S.P.A. (RINA), commissioned by Iridra s.r.l., has verified the performance claim of the technology "HYDRO-1" according to the relevant procedures for EU ETV as for GVP Version 01 - July 7th, 2014 and the requirements set in the Specific Verification Protocol N° 21DGMP13, Revision N° 4.

#### 1.1. NAME OF TECHNOLOGY

HYDRO-1

#### 1.2. NAME AND CONTACT OF PROPOSER

Name: IRIDRA SRL Contact: Fabio MASI Address: Via Alfonso la Marmora 51. 50121, Florence, ITALY Telephone: +39 055470729 Email: masi@iridra.com

#### 1.3. NAME OF VERIFICATION BODY/VERIFICATION RESPONSIBLE

RINA SERVICES S.P.A, accredited EU ETV Verification Body, conform to the requirements of ISO/IEC 17020 for inspection bodies type A and of the GVP version 1.

Role	Last	First Name	Company		
	Name				
Scheme Manager and Technical Director	D'Angelo	Giovanni	RINA SERVICES S.p.A.		
Vice-Technical Director	Marti	Laura	RINA SERVICES S.p.A.		

#### Table 1: ETV Responsible of Verification

## 1.4. ORGANISATION OF VERIFICATION INCLUDING EXPERTS, AND VERIFICATION PROCESS

#### Table 2: Organization of Verification

Role/Qualification	Last Name	First Name
ETV Service Coordinator	D'Angelo	Giovanni
ETV Inspector	D'Angelo	Giovanni
ETV Technical Expert and Reviewer	Maffini	Andrea

The verification carried out by RINA Services included the following activities:

- Verification Proposal Assessment: The initial performance claim has been revised. RINA has provided a detailed cost estimate for the verification procedure. Based upon the cost estimate, the verification contract has been drafted and signed by IRIDRA.
- Eligibility Assessment: HYDRO-1 is a technology eligible for EU ETV. This technology falls within the scope of the EU ETV pilot programme and in the Technological Area 1 "Water Treatment and Monitoring" according to the GVP
- Specific Verification Protocol review and approve: Upon successful completion of the contact phase and proposal phase RINA developed the specific verification protocol following the provisions of the GVP. The drafted SVP was reviewed by an internal and by an external technical expert. The SVP includes: summary description of the technology, its intended application and associated environmental impacts; definition of verification parameters (revised performance claim); requirements on test design and data quality; requirements on test and measurement methods, definition of calculation methods for performance parameters; description of the way in which operational, environmental and additional parameters are to be dealt with in the verification process; and assessment of existing data and conclusions on the need or not for additional tests or measures.

#### **1.5. DEVIATIONS FROM THE VERIFICATION PROTOCOL**

Task	Date
Verification Proposal Assessment	January 2021
Eligibility Assessment	September 2021
Specific Verification Protocol	December 2022
Verification Reporting	February 2023

#### Table 3: ETV Time Schedule

## 2. DESCRIPTION OF THE TECHNOLOGY



Figure 1: Plan layout of the HYDRO-1 in Antissa, Lesbos island (Greece)



Figure 1. Anaerobic reactor (AR)



Figure 2. Saturated vertical flow constructed wetland (VF SAT)



Figure 3. Unsaturated vertical flow constructed wetland (VF UNSAT)

#### 2.1 SUMMARY DESCRIPTION OF THE TECHNOLOGY

HYDRO-1 technology is based on two processes: an anaerobic treatment followed by nature-based solutions (NBS) with constructed wetland (CW), which allows obtaining a treated effluent that is suitable to be reused for irrigation purposes.

The first anaerobic reactor (AR) is composed on two micro reactors with square-shaped body where anaerobic wastewater treatment takes place, i.e. biological wastewater treatment carried out without using

air or oxygen, leading to low amount of sludge produced, and offering the possibility to recover the biogas produced by the anaerobic metabolism. In this case two identical rectangular reactors have been installed (2.4 x 2.4 m) with a total height of 4 m. The total volume of the reactors (up to overflow) is 41 m<sup>3</sup>. Then, the AR effluent is directed to the CW stage, which consists in a hybrid combination of Vertical Subsurface Flow (VF) CWs. The CW is designed with two stages: 1<sup>st</sup> stage, saturated downflow (VF SAT – 250 m<sup>2</sup>); 2<sup>nd</sup> stage unsaturated intermittent load (VF UNSAT – 600 m<sup>2</sup>). VF SAT is filled with gravel, while VF UNSAT is filled with gravel and an intermediate sand layer.

The aim of the applied technology is to guarantee "class A" requirements for wastewater reuse in irrigation in terms of TSS, BOD<sub>5</sub>, and turbidity, according to the EU Regulation 2020/741 of the European Parliament and of the Council of 25 May 2020 on minimum requirements for water reuse in agriculture. Moreover, the integrated system also claims effective removal of COD and nitrification. The performance claims are intended to be robust against change of conditions that could be encountered in touristic areas of the Mediterranean region between winter (cold humid climate and lower number of residents) and summer (hot arid climate and higher number of residents, increased by the anticipated tourism). Finally, biogas from anaerobic process can be also collected and reused.

The process that takes place in the anaerobic reactor consists of the following phases. Wastewater flows upwards through a sludge bed composed by anaerobic biological sludge which occupies about half the volume of the reactor. There, the anaerobic microorganisms decompose the organic matter of sewage, generating biogas. The CW stage utilizes the complex physical – chemical – biological processes, dominant for the pollutants' removal. Saturated VF CW is continuously fed (with the effluent stream of the AR), over the top of the bed and for the whole surface, maintaining saturated conditions and developing anaerobic/anoxic conditions. Wastewater is intended to stay beneath the surface of the gravel bed and flow through the roots and rhizomes of the plants and the gravel pores. The inert material is maintained water saturated. This solution is suitable to remove organic and solid loads, as well as to provide partial denitrification, if nitrate nitrogen is available. In unsaturated vertical subsurface flow (VF) wetland, wastewater is intermittently pumped on the top of the beds and offers the possibility of an alternate feeding system, to enhance the prevalence of unsaturated conditions, which occur through the transfer of large quantities of oxygen inside the main bed filled with coarse sand. The high oxygen transfer is suitable to remove the organic matter and perform nitrification satisfactorily.

The system enables to reclaim a large amount of water and nutrients (TN and TP) that, if coupled with a disinfection unit (e.g. UV irradiation), can be reused in agriculture under class A reclaimed water quality of EU Regulation 2020/741, i.e. permitting a reuse and recover of water and nutrients with minimum operational and maintenance cost in comparison to conventional technologies (lower sludge production, and manpower) for the cultivation of all the crop categories defined by the European regulation, i.e. crops for food, feed, industrial, energy or seed production.

## 2.2. INTENDED APPLICATION (MATRIX, PURPOSE, TECHNOLOGIES, TECHNICAL CONDITIONS)

Technology area	Technology purpose	Technical conditions
Water Treatment and monitoring	The application of HYDRO-1	The performance of the technology will
	system enables the domestic	be inside the range shown in the next
	wastewater treatment at	table. The parameters will vary
	community level. The	depending on seasonal conditions. The
	HYDRO-1 will treat sewage	outlet minimal values of concentration

#### Table 4: intended application

with low carbon footprint	are the expected ones by design, but
requirement and at low cost,	could eventually be even lower under
since no oxygen supply is	favorable operative conditions. The inlet
needed for the	maximum values can also be related to
biodegradation and at the	90% of the samples, the system can
same time an	tolerate and buffer 10% higher values in
environmentally-friendly	the year.
energy source (biogas) is	
produced. Moreover, the	
technology contributes to the	
disinfection and removal of	
pollutants such as total	
suspended solids (TSS),	
organic matter (expressed as	
BOD5 and COD, biochemical	
and chemical oxygen	
demands) and total nitrogen	
(TN). The aim is to guarantee	
class A requirements for	
reclaimed water quality class	
in terms of TSS, BOD5, and	
turbidity, according to the EU	
Regulation 2020/741, also	
contributing to COD removal,	
nitrification and biogas	
production.	
To sum up, the proposed	
technology allows:	
No wastewater	
discharge in the sea	
during the dry weather	
Low-cost production of	
reclaimed Water	
<ul> <li>Increasing water supply</li> <li>Recycling putrients</li> </ul>	

#### Table 5: winter condition

Operational	Temperature	Flow rate	Turbidity	COD	BOD <sub>5</sub>	TSS	N-NH <sub>4</sub>
Parameter	(°C)	inlet	inlet	inlet	inlet	inlet	inlet
		(m³/d)	(NTU)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Min	5	15	100	150	100	100	30
Max	20	30*	300	500	300	400	60
* The technology can be implemented by modules in order to fulfil different ranges of flow rate							
Performance	Turbidity	COL	)	BOD <sub>5</sub>	TSS		N-NH₄
Parameter	outlet	outle	et	outlet	outle	t	outlet
	(NTU)	(mg/l	L)	(mg/L)	(mg/L	.)	(mg/L)
Min	2	20		5	5		2
Max	5*	80		10*	10*		25
* Class A requirements for reclaimed water quality according to EU Regulation 2020/741							

Operational Parameter	Temperature (°C)	Flow rate inlet	Turbidity inlet	COD inlet	BOD₅ inlet	TSS inlet	N-NH₄ inlet
		(m²/a)	(NTU)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Min	20	50	100	400	200	200	30
Max	35	100*	300	1000	400	400	60
* The technology can be implemented by modules in order to fulfil different ranges of flow rate							
Performance	Turbidity	COL	)	BOD <sub>5</sub>	TSS		N-NH <sub>4</sub>
Parameter	outlet	outle	et	outlet	outle	t	outlet
	(NTU)	(mg/l	_)	(mg/L)	(mg/L	_)	(mg/L)
Min	2	20		5	5		2
Max	5*	80		10*	10*		25
* Class A requirements for reclaimed water quality according to EU Regulation 2020/741							

#### Table 6: summer condition

2.3 VERIFICATION PARAMETERS DEFINITION

Verification parameters, performance values and measurement methods are reported in the following table.

Parameter	Value	Existing legal Requirements	Test or measurement	Test /available data (+ performer			
		and/or BAT values	method(s)	of tests)			
	PERFORMANCE PARAMETERS						
COD effluent	< 80 mg/L		photometric				
TSS effluent	< 10 mg/L	< 10 mg/L*	Standard Methods				
			2540 D (APHA,				
			2005)				
BOD₅ effluent	< 10 mg/L	< 10 mg/L*	Standard Methods				
			5210 B				
			(APHA,2005)				
Turbidity	< 5 NTU	< 5 NTU*	Turbidity meter				
Sludge	<0.2		2540 B. Total Solids				
production	kgVS/kgCOD <sub>rem</sub>		Dried at 103-105 °C				
			method(APHA,				
			2005)				
Biogas	>0.3		flow meter				
production	Nm <sup>3</sup> /kgCOD <sub>removed</sub>						
COD removal	> 90% summer		photometric				
TSS removal	> 90% summer		Standard Methods				
			2540 D (APHA,				
			2005)				
N-NH <sub>4</sub> removal	> 60% summer		photometric				
Water recovered	> 10000 m <sup>3</sup> /y		flow meter				
	non-conventional						
	water						

 Table 7: parameters considered in the specific verification protocol

Resources	300 kN/y		flow meter for flow	
recovered	> 30 kP/y		rate	
			photometric for (TN	
			concentration	
			Standard Methods	
			4500-P E. Ascorbic	
			Acid Method	
			(APHA, 2005) for	
			TP concentration	
	0	PERATIONAL PARAM	ETERS:	<u> </u>
Flow	Summer: 100		flow meter	
	m <sup>3</sup> /d			
	AR: HLR			
	minimum 0.36			
	m3/m3/d			
	CW: HLR			
	minimum 0.018			
	m3/m2/d			
	Winter: 15 m <sup>3</sup> /d			
	AR: HLR			
	maximum 2.44			
	m3/m3/d			
	CW: HLR			
	maximum 0.118			
	m3/m2/d			
COD	150 – 1000 mg/L		photometric	
concentration			F	
inlet				
BOD <sub>5</sub>	100 – 400 mg/L		Standard Methods	
concentration			5210 B	
inlet			(APHA.2005)	
TSS	100 – 400 mg/l		Standard Methods	
concentration	100 100 mg/L		2540 D (APHA	
Concontration			2005)	
N-NH4	30 - 60  mg/l		photometric	
concentration	00 00 mg/L		photomotilo	
inlet				
Temperature	5 – 35 °C		sensor for T inside	
Pressure	positive		the reactors	
(hiogas)			meteorological	
(blogas)			station for air T	
Waste	<0.2		25/0 B Total Solida	
apparated	ka\/S/kaCOD		Dried at 102 105 °C	
yeneraleu				
(siuuge)			2003) TERS	
Man-powor				
needed				
	E0 dou/ucor		interview to staff	
operation and	50 day/year		Interview to staff	
maintenance				

Space needed	AR: 30 m <sup>2</sup>	as built drawings
	CW: 850 m <sup>2</sup>	
Service life	20 years	consumable of the
		material
Robustness/vuln	see claims of	
erability to	treatment	
changing	performance from	
conditions of	winter to summer	
use	conditions	

## **3. EXISTING DATA**

HYDRO-1 is a technology developed within the Horizon2020 EU funded project of **HYDROUSA** (Demonstration of Water Loops with innovative Regenerative Business models for Mediterranean Region, <u>www.hydrousa.org</u>), a project submitted under the call CIRC-02-2016-2017 Water in the context of the circular economy (Grant Agreement No. 776643).

HYDRO-1 is the main component of the HYDRO1 demo site of the project, realized in Antissa, in Lesbos island (Greece). HYDRO1 operation and monitoring are coordinated by the *Sanitary Engineering Laboratory of the National Technical University of Athens* (NTUA), which is also the coordinator of the project and by the *Water and Air Quality Laboratory of the University of Aegean* (UoA) (hereinafter "Aegean University Lab"), which is also a project partner.

The Sanitary Engineering Laboratory is included in the list of NTUA laboratories approved by NTUA Senate providing advisory and consultancy services. Furthermore, it is accredited by the Hellenic Accreditation System according to EAOT EN ISO/IEC 17025 (accreditation no 496) for chemical and microbiological analyses for water, wastewater and sludge in the field of water resources and wastewater management.

Moreover, the supervision to operational and monitoring of HYDRO1 is also provided by two other partners of the project, *AERIS* (Spain) for the anaerobic reactor and *IRIDRA* (Italy) for the part related to the constructed wetland stage.

Due to the lack of certified laboratories in Lesbos Island and the willingness to maximize the frequency of the sampling campaign, according to the available budget for monitoring activities, NTUA has decided to realize a control room with a laboratory of analysis in situ (hereinafter called "**in situ lab**") to monitor part of the pollutants of interest and to have daily presence on the HYDRO1 site to coordinate its operation and monitoring. The in situ lab is hosted in a portable container (ISOBOX – see Figure 4) and has an area dedicated to lab analysis (Figure 5) with the following equipment (Figure 5):

- Spectrometer WTW photoLab® 7600 UV-VIS
- Thermoreactor WTW CR 2200
- Magnetic Stirrer VELP Scientifica F203A0440
- Portable pH meter WTW MultiLine® Multi 3630 IDS with pH probe WTW IDS pH Electrode SenTix® 940
- Turbidity meter WTW Turb 430 IR
- Refrigerator

As described in the next sections and chapter, NTUA has provided skilled personnel to follow the activities of the in-situ lab. All personnel involved in the monitoring of the units are highly educated PhD candidates certified by the Sanitary Engineering Laboratory (SEL), that are well trained in all aspects of quality ETV\_07\_VReport\_RINA(12\_2020) page 10/22

assurance and quality control for performing monitoring of the treatment units. In addition, a clear organization that specifies the tasks for each member of the team have been developed to assure the quality of the monitoring and operating activities and to provide high quality results.





Figure 4. In situ control room

Figure 5. In situ lab within the control room

NTUA staff has access to the **Aegean University Lab** to do analyses on other pollutants of particular interest. In particular, the following instruments of the Aegean University Lab are used for the monitoring of HYDRO 1:

- Equipment for BOD measurement WTW Oxitop I set 6 (Appendix 2 Error! Reference source not found.)
- 105° oven (WTC BINDER) (Appendix 2 Error! Reference source not found.)
- KERN 770 analytical balance (Appendix 2 Error! Reference source not found.)
- Stirring heating plate SBS A-06 series H (Appendix 2 Error! Reference source not found.)

The Aegean University Lab is located at the Lesbos Island, in Mytilene, about one hour far by car from the HYDRO 1 site. The short distance between the Aegean University Lab and the HYDRO 1 site allows to perform lab analysis in the same day of the sampling activities, limiting any risk of deteriorating the quality of the collected samples, as described in detail in the dedicated quality assurance section.

The in-situ Lab and the Aegean University Lab permit to monitor all the parameters required to demonstrate the defined claims for the HYDRO-1.

### 3.1. ACCEPTED EXISTING DATA

The accepted existing data are following listed, describing the type of collected data.

#### PERFORMANCE PARAMETERS

- **COD outlet**: grab water quality sample, analysed with photometric method with WTW COD Cell Test kit C3/25 range: 10-150 mg/L, using the chromosulfuric acid oxidation/chromate determination method according to EN ISO 15705
- **TSS effluent:** grab water quality sample, analysed according to Standard Methods 2540 D (APHA, 2005). Total Suspended Solids Dried at 103-105 °C
- BOD<sub>5</sub> effluent: grab water quality sample, analysed according to Standard Methods 5210 B (APHA,2005) – 5 DAY BOD Test / WTW DIN 38409 H 52
- Turbidity effluent:

- o grab water quality sample, Turbidity meter, WTW Turb 430 IR
- online sensor, WTW Viso Turb® 700 IQ
- Sludge production:
  - <u>Height of sludge blanket</u> at different depths of the AR reactor (Appendix 2 **Error! Reference source not found.**), from bottom to the top
    - 0.4 m
    - 0.9 m
    - 1.4 m
    - 1.9 m
    - 2.4 m
    - 4.0 m (effluent)
  - <u>Total solid concentration of sludge blanket</u> grab water quality sample at each sampling point (each depth) of the AR reactor (from bottom to the top), analysed with 2540 B. Total Solids Dried at 103-105 °C method(APHA, 2005)
  - Sludge blanket threshold concentration: TS=10000 mg/l (S. K. Narnoli; Indu Mehrotra, 1997).
- **Biogas production:** flow meter COMBIMASS F03
- COD removal (COD concentration inlet):
  - grab water quality sample, analysed with photometric method with WTW COD Cell Test kit 14690 – range: 50-500 mg/L, using the chromosulfuric acid oxidation/chromate determination method according to EN ISO 15705
- TSS removal (TSS concentration inlet): grab water quality sample, analysed according to Standard Methods 2540 D (APHA, 2005). Total Suspended Solids Dried at 103-105 °C
- N-NH<sub>4</sub> removal:
  - <u>N-NH<sub>4</sub> concentration inlet</u>: grab water quality sample, analysed with photometric method with WTW NH<sub>4</sub> Cell Test kit A6/25– range: NH<sub>4</sub> 0.2-8.00 mg/L; N-NH<sub>4</sub> 0.26 10.30 mg/L, using the indophenol blue method according to ISO 7150-1
  - <u>N-NH<sub>4</sub> concentration outlet</u>: grab water quality sample, analysed with photometric method with WTW NH<sub>4</sub> Cell Test kit 1.14739– NH<sub>4</sub> 0.010-2.000 mg/L; N-NH<sub>4</sub> 0.01 2.58 mg/L, using the indophenol blue method according to ISO 7150-1
- Water recovered: electromagnetic flow meter SMC LFE
- Resources recovered:
  - <u>TN concentration outlet</u>: grab water quality sample, analysed with photometric method with WTWN Cell Test kit 1.14763– range: 10-150 mg/L, using the peroxodisulfate oxidation/2.6-dimethylphenol method according to EN ISO 11905-1
  - <u>TP concentration outlet</u>: grab water quality sample, analysed according to Standard Methods 4500-P E, Ascorbic Acid Method (APHA, 2005)

#### **OPERATIONAL PARAMETERS**

- Flow inlet: no. 2 flow meters iFm SM9000 (one for each reactor)
- **pH:** effluent AR and effluent CW, pH meter, WTW Multi 3630 IDS

- Temperature:
  - Heating tank (influent AR): temperature sensor iFM TD2541
  - Inside AR reactors: no. 2 temperature sensors PT100-V-PG (one inside each reactor)
  - o <u>Air Temperature</u>: meteorological station provided by the project partner AGENSO
- Precipitation: meteorological station provided by the project partner AGENSO
- Pressure (biogas collection): pressure sensor iFM PI008A

#### ENVIRONMENTAL PARAMETERS

• **Consumables:** water quality samples

#### ADDITIONAL PARAMETERS

- Man-power needed: based on interview to the NTUA staff
  - o Ordinary
    - AR:
- 1 visit per week (3 hour): Sample withdrawal, checking of the equipment, condensate emptying, delivery of samples in the laboratory.
- Daily remote control (1 hour/day) monitoring of the process and operational parameters.
- 1 per month (1 hour) cleaning of the feeding system.
- CW: 1 visit per month in winter (1 hour), 1 visit per week in summer (1 hour)

#### • Space needed:

- <u>AR</u>: as built drawings
- $\circ$  <u>CW</u>: as built drawings
- Service life: 20 years
- Robustness/vulnerability to changing conditions of use: removal efficiency claims (effluent COD, BOD<sub>5</sub>, TSS; COD, TSS, BOD<sub>5</sub>, N-NH<sub>4</sub> removal) confirmed under different operational conditions (winter no touristic; summer touristic)

### 4. EVALUATION

#### 4.1. CALCULATION OF PERFORMANCE PARAMETERS

HYDRO-1 candidates to ETV for the following performance parameters:

- Total suspended solids (TSS) concentration in the final effluent: < 10 mg/L in 90% of the samples, none of the values of the samples exceed the maximum deviation limit of 100% of the indicated value (Class A requirements for Reclaimed water quality according to EU Regulation 2020/741)
- BOD<sub>5</sub> concentration in the final effluent: < 10 mg/L in 90% of the samples, none of the values of the samples exceed the maximum deviation limit of 100% of the indicated value (Class A requirements for Reclaimed water quality according to EU Regulation 2020/741)

- Turbidity below < 5 NTU in 90% of the samples, none of the values of the samples exceed the maximum deviation limit of 100% of the indicated value (Class A requirements for Reclaimed water quality according to EU Regulation 2020/741)
- 4. COD concentration in the final effluent is always: < 80 mg/L
- 5. Sludge yield < 0.2 kg VS/ kg COD removed
- 6. Biogas production: > 0.3 m3 biogas/kg COD<sub>removed</sub>
- 7. Treatment load
  - a. winter period (tested flow rate minimum 15 m<sup>3</sup>/d)
    - i. AR: Hydraulic loading rate (HLR) minimum 0.36 m3/m3/d
    - ii. CW: HLR minimum 0.018 m3/m2/d
  - b. summer period (tested flow rate maximum 100 m<sup>3</sup>/d)
    - i. AR: HLR maximum 2.44 m3/m3/d
    - ii. CW: HLR maximum 0.118 m3/m2/d
- 8. > 60% N-NH<sub>4</sub> removal in summer period for 90% of the samples
- 9. > 90 % COD removal in summer period for 90% of the samples
- 10. > 90% TSS removal in summer for 90% of the samples
- 11. > 10000 m<sup>3</sup>/y of reclaimed water
- 12. > 300 kN/y and > 30 kP/y from reclaimed water for fertigation

#### 4.2. EVALUATION OF TEST QUALITY

NTUA has developed a detailed test design and quality procedure to assure the validity of the data collected, in order to successfully monitor the HYDRO 1 site in line with the quality required by both the H2020 project monitoring and ETV. The procedure for Test design, reference analysis and measurement, data management, quality assurance, and test report requirements are detailed in the SVP.

#### 4.2.1 CONTROL DATA

All the equipment employed for chemical analyses in the in situ lab and in the Aegean university lab <u>operate</u> <u>according to the manufacturer specifications</u>. Specifically, the following procedures are followed by the Analysts to calibrate the equipment:

- zero calibration of the spectrophotometer (COD, N-NH4+): According to specifications provided by the manufacturer zero calibration is performed prior to chemical analyses;
- Quality Control Samples (Standards): For all chemical analyses standards of known concentrations are employed, for method and equipment validation prior to sample analyses;
- Developing quality control charts: For all analytical methods quality control charts will be developed every two months using quality control samples of known concentrations
- Once a month inter calibration of all chemical analyses is performed by conducting analysis on a common sample both at the site and at SEL;
- turbidity meter calibration: as required by the meter, the calibration consists of using a standard turbidity solution to calibrate the turbidity measurement following the automated instruction of the meter;

• pH meter calibration: once per month, the calibration consists of using a standard pH solution to calibrate the pH measurement following the automated instruction.

For the **validation of each method** the following parameters where evaluated:

- Repeatability
- Reproducibility
- Combined standard Uncertainty
- Expanded uncertainty

In order to evaluate the uncertainty of each method of analysis multiple steps have been followed.

As a first step two kind of errors have been distinguished and was analyzed. That is Random errors and systematic errors.

The Random error is arising from unpredictable variations of influence quantities and these random effects give rise to variations in repeated observations of the measurand. In chemical analysis the repeatability result for each method is acceptable to be a good estimator for random error.

On the other hand, the systematic errors are defined as a component of error which, in the course of a number of analyses of the same measurand, remains constant or varies in a predictable way. <u>The most</u> <u>important sources of errors that have been chosen for the selected methods are</u>:

- Absorbance of measurement error
- Dilution error
- Pipette error
- Calibration curve error
- observed measurement error (C Cm)
- Standard solution preparation error
- lincubator and drying oven temperature error
- Balance error

The above list then was quantified by calculating the standard uncertainty and the relative standard uncertainty for each parameter. The following table explains the calculation procedure

Uncertainty contributor	Calculation procedure and assumtions
Absorbance of measurement	Instrument error (AU) that is given by the manufacturer usually or can be retrieved after verification of system
Volumetric flask error	Is calculated by assuming a rectangular distribution, with a standard deviation of a/ $\!\sqrt{3}$
Pipette error	Instrument error (in $\mu$ L or mI) that is given by the manufacturer usually or can be retrieved after verification of system
Dilution error	Is calculated taking account the volumetric flasks and pipette errors and using the propagation of uncertainty formula
Calibration curve error	A first-order least-squares fit of the data is computed as well as the standard error of the fit curve
observed measurement error (C - Cm)	Is the difference between the observed and the known value of certified material

#### Table 8: calculation procedure

Incubator and drying oven temperature	Obtained from manufacturer or verification but can be neglected as can be assumed to be involve also in reproducibility result
Balance error	Instrument error (in mg) that is given by the manufacturer usually or can be retrieved after verification of system
Standard solution preparation	Is calculated by combining the balance and volumetric flask error

All the above errors (systematic and random) are being expressed as relative standard uncertainties, that is, as relative standard deviations and then the <u>combined standard uncertainty</u> was calculated. Finally, by applying the appropriate coverage factor (k=2 for 95% confidence interval) the <u>expanded uncertainty</u> was calculated.

The goals of **quality assessment** were to determine when an analysis has reached a state of statistical control, to detect when an analysis falls out of statistical control, and to suggest possible reasons for this loss of statistical control. The methods that have been adopted from our lab for the evaluation of quality test is

- DUPLICATE SAMPLES
- ANALYSIS OF BLANKS
- STANDARD SAMPLES

which provide immediate data about the control of analysis.

#### 4.2.2 AUDITS

The existing data provided by IRIDRA has been fully accepted by RINA.

#### 4.2.3 DEVIATION

No deviations were identified by RINA during the SVP phase.

#### 4.3. VERIFICATION RESULTS (VERIFIED PERFORMANCE CLAIM)

#### 4.3.1. DESCRIPTION OF STATISTICAL METHODS USED

Data were collected for two years (2021-2022) and were analyzed with standard statistical analysis, calculating:

- mean
- standard deviation
- minimum
- 1<sup>st</sup> quartile (25° percentile)
- 2<sup>nd</sup> quartile (50° percentile median)
- 3<sup>rd</sup> quartile (75° percentile)
- maximum

The statistical analysis is graphically represented with box-whisker plots, which displays the five-number summary of a set of data. The five-number summary is the minimum, first quartile, median, third quartile, and maximum.

#### 4.3.2 VERIFICATION PARAMETERS

In the table below, is presented a sample of analysis results in the summer and winter period from the real system. The results consider the overall performance of the HYDRO-1 Demo Scale.

Parameter	IN (mg/L)	St.dev.	OUT (mg/L)	St.dev	% rem
COD	555	±254	31	±13	94
BOD5	256	±105	6	±3	98
TSS	260	±131	4	±3	99
N-NH4	53.5	±18.3	4.2	±3.5	92
Turbidity (NTU)	218	±89	3	±1	-
COD	555	±254	31	±13	94
BOD5	256	±105	6	±3	98
TSS	260	±131	4	±3	99
N-NH4	53.5	±18.3	4.2	±3.5	92

Table 9: Quality of the inlet and outlet wastewater observed in the monitoring period 2021-2022

Table 10: Recovered bioproducts observed in the monitoring period 2021-2022

Sludge production (t/y)	Biogas production (m3/y)	Water recovered (m3/y)	Resources recovered tN/y; tP/y
1.05	2500	20000	1.1 tN/y 0.15 tP/y

In the following Figures there are reported the concentration trends vs. time. as also the measured daily flow (red line), for the main observed chemical parameters (BOD<sub>5</sub>, COD, N-NH4<sup>+</sup>, Turbidity, TSS).

About the organic matter (BOD<sub>5</sub>), it can be observed that the final outlet concentrations have been always under the limits for Class A of the Reuse Directive, until the flow has been lower than 50 m<sup>3</sup>/d. Above this threshold, a simple operational variation was required to bring again BOD<sub>5</sub> concentrations constantly below the desired 10 mg/L, i.e. maintaining saturated the 30 cm of the bottom part of the VF UNSAT (just regulating the existing outlet pipe in the appropriate manhole). All the other parameters are instead under the norm limits in all the cases.





Figure 7. Time series during the monitoring period

As explained above, the red line shows the increasing treated flow along the two observed summer and winter periods 2021 and 2022. It clearly appears that the outflow BOD<sub>5</sub> concentrations are constantly below the 10 mgO2/L limit with the exception of a few samples in summer 2022. when operating the 2nd stage VF UNSAT beds in the completely drained configuration; once we have noticed the increase of the outlet concentrations, we started operating those beds keeping the 30 cm at the bottom of each of them watersaturated (just closing a tap in the outlet well). The creation of an anoxic environment at the bottom of the beds is improving the denitrification process, which is consuming the faster degradable Carbon and therefore immediately decreasing the BOD<sub>5</sub> outlet concentrations. The conclusion from these observations is, therefore, that when the treatment plant is operated in summer at the highest flow, about 100 m<sup>3</sup>/day, for maintaining all the time the outlet characteristics in full respect of the Class A limits for the wastewater reuse, the 2<sup>nd</sup> stage VF UNSAT beds have to be operated with the + 30 saturated bottom configuration.

The TSS outlet concentrations are all time respecting the required quality for the further usage by fertigation. Indeed, the filtration process which occurring inside the sand-based 2<sup>nd</sup> stage VF UNSAT beds is sufficient, and there is no need of any other filtration step before the reuse practice.

The quality of the reported dataset can be observed by the following whisker plots graphics.





Figure 8. Graphical representation of statistical analysis of monitored parameters with box-whisker plots

#### **4.3.3 ADDITIONAL PARAMETERS, WITH COMMENT OR CAVEATS WHERE APPROPRIATE** None.

#### 4.4. RECOMMENDATIONS FOR STATEMENT OF VERIFICATION

Only the verification protocol and verification report require external review according to EU ETV pilot programme GVP. The review was performed by Andrea MAFFINI. RINA Services reviewed and approve the test plan and review the test report.

## **5. QUALITY ASSURANCE**

The personnel and experts responsible for quality assurance as well as the different quality assurance activities are described in the next table.

#### Table 11: Personnel and experts responsible for quality assurance

Role	Inspector	TE	ITR	Proposer
Responsible	Giovanni	Giovanni	Andrea	Fabio

	D'ANGELO	D'ANGELO	MAFFINI	MASI
Specific Verification Protocol	Draft	Draft	Review	Review and
				approval
Test Plan	Approval	Review	N.R.	N.R.
Test System at test site	Audit	Audit	N.R.	N.R.
Test Performance	Audit	Audit	N.R.	N.R.
Test Report	Approval	Review	N.R.	N.R.
Verification Report	Draft	Draft	Review	Acceptance
Statement of Verification	Draft	Draft	Review	Acceptance

## 6. REFERENCES

EU Environmental Technology Verification Pilot Programme. General Verification Protocol. version 1.3 of 01 April 2018

## **APPENDIX 1 TERMS AND DEFINITIONS**

"EU ETV – European Environmental Technology Verification" is the EU programme providing for thirdparty verification, on a voluntary basis, of the performance claims made by technology manufacturers in business-to-business relations.

"GVP – General verification protocol" means the description of the principles and general procedure to be followed by the ETV pilot programme when verifying an environmental technology.

"SVP – Specific verification protocol" means the protocol describing the specific verification of a technology and applying the principles and procedures of the General verification protocol.

"**Performance claim**" means a set of quantified and measurable technical specifications representative of the technical performance and environmental added value of a technology in a specified application and under specified conditions of testing or use.

**"Verification"** means the provision of objective evidence that the technical design of a given environmental technology ensures the fulfilment of a given performance claim in a specified application, taking any measurement uncertainty and relevant assumptions into consideration.

"Deviation" is a change to a specific verification protocol or a test plan done during the verification or test step performance.

**'Test system assessment'** means determining whether the test system and quality management system applied by a test body to generate data for verification purposes comply with the requirements of the General Verification Protocol and of the specific verification protocol. It includes the review of the relevant accreditations, and may include a test system audit.

**'Test system audit'** means the examination of a test system and of a quality management system. It is achieved through the review of relevant procedures, observation of actual practices and evaluation of test performance. Where applicable, it includes the examination of control data for relevant period, participation in proficiency testing and/or control of calibration of measurement devices. It is aimed to provide the necessary evidence for the test system assessment.

"Accreditation" has the meaning assigned to it by Regulation (EC) No 765/2008.