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Technical Note

Design, setup and routine operation of a water treatment system for the monitoring of low activities of tritium in water

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ABSTRACT

In the TRITIUM project, an on-site monitoring system is being developed to measure tritium (^{3}H) levels in water near nuclear power plants. The quite low-energy betas emitted by ^{3}H have a very short average path in water (5 μm as shown by simulations for 18 keV electrons). This path would be further reduced by impurities present in the water, resulting in a significant reduction of the detection efficiency. Therefore, one of the essential requirements of the project is the elimination of these impurities through a filtration process and the removal of salts in solution. This paper describes a water treatment system developed for the project that meets the following requirements: the water produced should be of near-pure water quality according to ISO 3696 grade 3 standard (conductivity < 10 μS/cm); the system should operate autonomously and be remotely monitored.

1. Introduction

Present detection systems based on liquid scintillation counting (LSC) allow measuring with high accuracy ^{3}H activities in the water of the order of 1 Bq/L. However, these measurements require a few days. This delay is not acceptable if anomalous activity concentrations of ^{3}H are released in the discharged waters of nuclear facilities, which may be used for public supply systems, because of the risk of underestimation of the dose to the public. However, there is no commercial solution for an in-situ real-time monitoring system for nuclear power plants.

The TRITIUM [1] project was funded by the European Union's INTERREG SUDOE programme and carried out by a consortium of 5 south-western European institutions: University of Aveiro (Portugal), CENBG Bordeaux (France), University of Extremadura (Spain), University of Valencia (Spain) and Junta de Extremadura (Spain). The main goal of the project is to develop a near real-time monitor to measure ^{3}H activity concentrations in continental and marine waters and to enable an in-situ surveillance of the water discharges in the vicinity of nuclear power plants. As an example, Fig. 1 shows the ^{3}H activity concentration measured in water samples taken monthly in the water discharge channel of the Almaraz Nuclear Power Plant (ANPP) and two other sampling points located in the Tagus River, upstream and downstream of the discharge channel. As can be seen, ^{3}H activity levels in the ANPP discharge channel are one or two orders of magnitude higher than those in the water samples taken at upstream and downstream sites. Furthermore, it is important to point out that the ^{3}H concentration in downstream water samples is lower than in upstream samples due to the contribution of the Tietar River (a Tagus River tributary). A detailed description of ^{3}H concentration patterns in the Tagus River can be found in ref. [2]. The TRITIUM project detector is described in ref. [1,3].

The energy of the beta emission of ^{3}H is very small (5.68 keV on average) which produces a very short (5 μm) mean free path of the beta particle in water. The presence of impurities in the water

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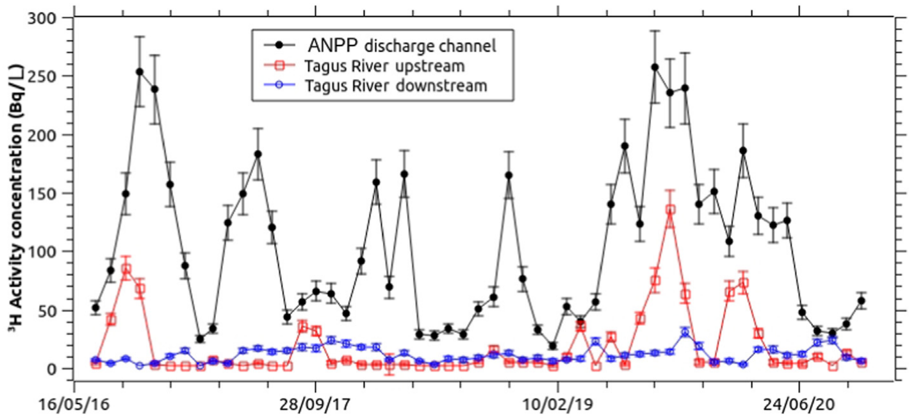

Fig. 1. ^{3}H activity levels measured in water samples taken in the Tagus River in different locations: ANPP discharge channel and upper and downstream of this location. The data, available in http://158.49.111.36/juntaex/vraex/map/., were extracted from the surveillance monitoring program around ANPP.

further reduces this mean free path, producing a further reduction of the detection efficiency. Additionally, continental and marine waters usually contain high concentrations of salts in solution, and particles forming colloids, as well as algae and other microorganisms. For ^{3}H measurement, a near-pure water quality is an essential requirement to avoid poor detection efficiency. This can be achieved by removing algae and sediments through a multi-stage filtration process and salt removal.

A water treatment plant was developed for the TRITIUM project and was installed to supply near-pure water grade to the TRITIUM detector prototype. There are many industrial solutions for medium and small size water filtration and deionization plants in remote locations [4–7]. These are usually designed for industrial facilities which sufficient space for on-site installation and maintenance operations. However, the requirements of the water purification plant proposed for the TRITIUM project are: 1) a small assembly space (less than 5 m^2); 2) most maintenance operations to be carried out remotely to reduce the cost of technical staff; 3) the water quality to be close to pure grade according to ISO 3696 grade 3 standard [8].

The installation of the water treatment plant and the prototype of TRITIUM detector was carried out in the spring of 2019 at the Arrocampo site, close to the water discharge channel of the ANPP into the Tagus River. High concentrations of ^{3}H could be detected at this site.

2. Materials and methods

2.1. Situation

The Arrocampo reservoir (39°49′00″N; 5°42′00″W) was built in 1976 on the Arrocampo stream and its landform to supply water to the secondary cooling of the ANPP. The water is taken from the Tagus River and describes a U-shaped circuit of 25 km which allows the cooling of the ANPP turbines. The water is discharged into the Tagus River when its temperature is close to the river temperature. The water treatment system was assembled in the water monitoring station of the Radiological Early Warning Network of Extremadura (RAREx) [9] (See Fig. 2) which is located in the Tagus River near the Arrocampo reservoir and the ANPP discharge channel.

The description and operation of the water monitoring station is described in ref. [10]. In the first stage, a pump continuously transfers water from the reservoir into a steel vessel shielded by a 10 cm thick lead layer. Gamma activity is measured by a 5 × 5 cm^2 NaI gamma scintillator detector. Water flow is continuously measured by an electronic flowmeter with a relative accuracy of 0.5%. Electromechanical flow distribution valves are installed and can be remotely or manually controlled. The average water flow rate is 25 L/min. After the gamma measurement, the water passes through the purification plant where it is treated before being measured by the TRITIUM detector. Finally, both rejected and pure water are mixed and discharged into the river. Fig. 2 shows a scheme of the RAREx monitoring station.

2.2. Measurement procedures and devices

The ^{3}H activity of the water from the ANPP discharge channel was measured by the LSC technique with the lowest minimum detectable activity. Water samples were collected in polyethylene bottles and filtered through 0.45 μm pore filters before being mixed with a liquid scintillation solution of Optiphase Hisafe 3 from LKB Wallac at a sample/scintillator ratio of 8/12 in 20 ml Teflon-coated polyethylene vials. ^{3}H activity was determined using a Quantulus-1220 liquid-scintillation alpha–beta spectrometer, equipped with a special background reduction system. The acquisition time was 500 min and the ^{3}H minimum detectable activity was 1.4 Bq/L. The spectrometer requires detection efficiency calibration as a function of the quenching. This calibration was performed with ^{3}H standards of known activity subjected to different degrees of quenching with CCl_4. The detection efficiency of the unquenched standards was 30%.

A γ-spectrometric analysis was carried out by an extended range HP(Ge) detector of 48% efficiency. The detector was shielded with a 15 cm thick old lead layer. A gamma cocktail with energies in the range of 46–2000 keV was used for efficiency calibration. Three spiked water samples were prepared in the same way as environmental samples to obtain the efficiency calibration curve. The gross relative uncertainty due to calibration, measurement and precision uncertainties was of the order of 10%.

Reference samples provided by the IAEA were systematically used to confirm the precision and accuracy of the measuring devices. Moreover, the results provided by both devices were previously tested in several international comparison exercises with satisfactory results. The overall quality control of these analytical procedures is guaranteed by the accreditation of the laboratory to carry out radioactivity assays in environmental samples according to UNE-EN ISO/IEC 17025 [11]. Uncertainties were calculated according to ISO11929 [12].

For the measurement of the physicochemical parameters of the

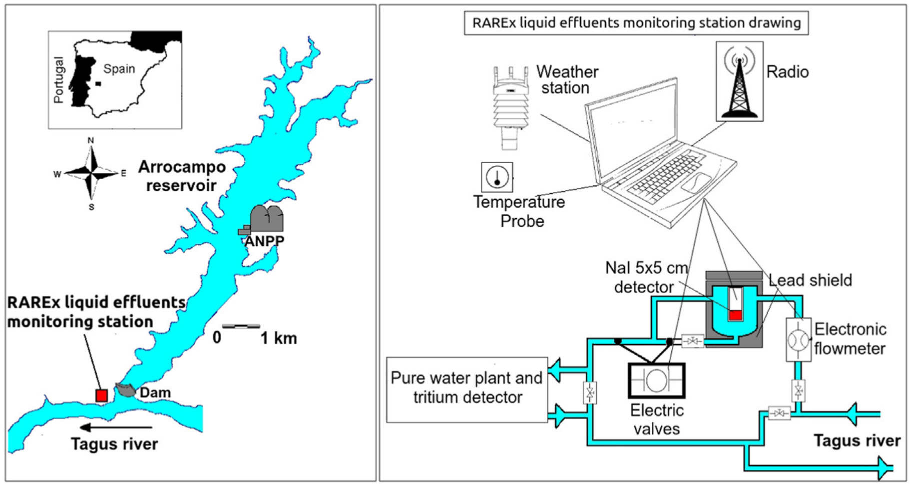

Fig. 2. (left) Location of RAREx water effluent monitoring station close to Arrocampo reservoir dam. (right) Scheme of the monitoring station.

water, a multiparameter measurement device, model Hanna HI9829, was employed. This device monitors up to 14 different water quality parameters. A microprocessor-based multi-sensor probe allows for the measurement of pH, redox potential ORP, conductivity, dissolved oxygen, turbidity, ammonium, chloride, nitrate, and temperature.

2.3. Water treatment plant design

For the design of the water treatment plant, it is very important to know the main physicochemical characteristics of the Tagus River water. Table 1 shows the chemical constituents of a Tagus River water sample taken in 2019 and measured in an external accredited analytical laboratory.

Two parameters directly affect the ^{3}H measurement: turbidity, which is the cloudiness of a fluid caused by a large amount of solid matter, and conductivity which is directly related to the total amount of dissolved solids. The water of the Tagus River can be very turbid due to the high concentration of algae caused by eutrophication, which is particularly noticeable during the summer months. In addition, the conductivity is of the order of 1100 μS/cm. Therefore, the water purification system designed for the TRITIUM project had to have the following characteristics: firstly, rough and filtration stages to remove all suspended solids, dissolved solids and even free cations and anions; secondly, taking into account the water flow conditions under which the RAREx water monitoring station operates, the plant should provide a flow rate of about 10–30 L/min of pure water; finally, all cleaning processes had to be automated. Based on these requirements, a water purification plant was designed and assembled at the Arrocampo monitoring station. Fig. 3 shows the schematics of the purification processes.

The system consists of two roughing and filtration loops operating in parallel. The water is continuously pumped from the Tagus River by two 1500 W pumps. The operating pressure is between 4 – 6 bar. First, the water passes through the 25 L stainless-steel vessel to be measured by gamma spectrometry. Next, the water passes through the water treatment plant. In this first stage, the water flows at a constant pressure of 4 bar. Prior to filtration process, a dosing pump injects NaClO into the water to remove organic matter. The initial roughing is carried out in the first place by a silex-anthracite filter and then by a garnet filter. In both filters, the arrangement of the filtering material is from the larger to the smaller diameter in the water flow direction. Each filter loop has a self-cleaning 20 μm filter. Subsequently, water passes through an activated carbon filter to remove the chlorine anions. Finally, the filtration stage ends in a 1 μm polyethylene filter. Before the water enters the reverse osmosis system, sterilization is carried out by a 40 W ultraviolet lamp.

To remove all dissolved solids from water, the use of both ionic and anionic filtration resins was initially proposed. However, the disadvantage of this solution is that resins saturate after a short time and need to be replaced, which implies frequent and expensive maintenance. Therefore, it was decided to install a configurable double reverse osmosis system. The two osmosis branches can be operated either in series or in parallel. The appropriate selection of operation mode depends on the water purity grade required. Two reverse osmosis systems in series produce a pure water grade between 2 and 3 according to ISO 3696 [8], with a conductivity of less than 4 μS/cm. However, the power supply needed for series operation exceeds the uninterruptible power installed that is already significantly high (8 kW). A single reverse osmosis system produces water with a conductivity of the order of 5–20 μS/cm, which fulfils the requirement for the operation of the TRITIUM detector. To extend the lifespan of the osmosis membrane and reduce maintenance, the stages of the osmosis system work alternately every 96 h, with 1 min pauses between cycles.

Table 1
Physicochemical parameters measured in a Tagus River water sample taken in the year 2019.

Physicochemical parameters			
CO_3H^-	154 mg/L	**Mg^{2+}**	46 mg/L
Ca^{2+}	105 mg/L	**NO_3^-**	16 mg/L
Cl^-	196 mg/L	**NO_2^-**	0.03 mg/L
K^+	11 mg/L	**Na^+**	173 mg/L
Turbidity	29 FNU	**SO_4^{2-}**	217 mg/L
Dry residue	1029 mg/L	**Conductivity**	1550 μS/cm

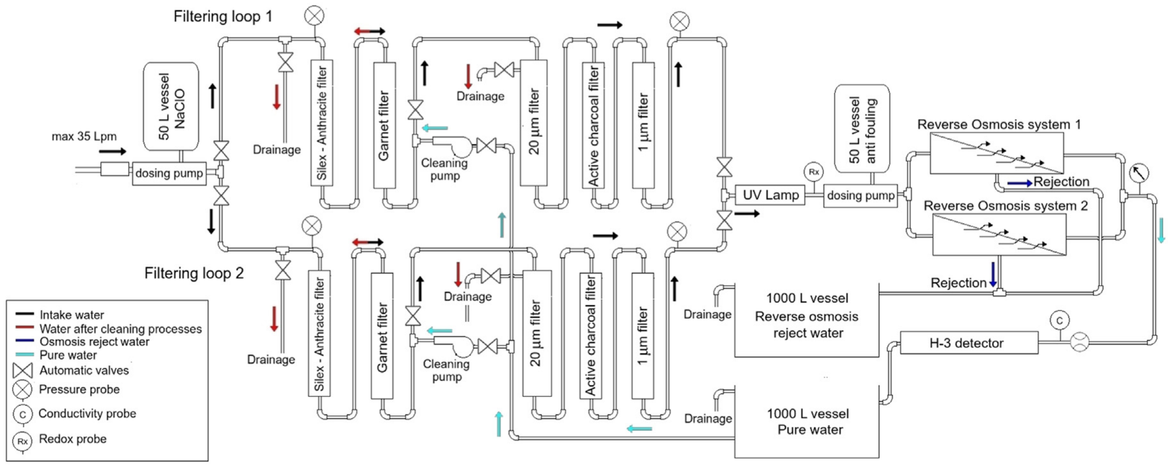

Fig. 3. Scheme of the water treatment plant installed in RAREx water monitoring station in the Tagus River.

The osmosis system produces rejected water, which is approximately 50% of the input water. This rejected water is sent to a 1 m^3 vessel located in a steel structure built on the roof of the monitoring station (Fig. 4). The produced pure water passes through the TRITIUM detector and is sent to another 1 m^3 vessel also placed in the aforementioned structure. The overflow of both water tanks is mixed in a pipe located close to the station to dilute the rejected water before discharging it directly into the Tagus River.

The water treatment system is controlled by a programmable logic controller (PLC) which can be managed remotely. The PLC allows automatic running and cleaning operations of the water treatment plant. A safeguard mechanism avoids serious damage to the osmosis system in case of water pressure loss. Moreover, every 3 min, the PLC stores in a MySQL database the different monitoring parameters (conductivity, pressure, flow) which are sent to the Cáceres headquarters by the main computer of the RAREx water monitoring station.

Every 24 h, silex-anthracite and garnet filters are cleaned by countercurrent flow using the pure water stored in the 1 m^3 vessel. This operation is managed by the PLC which operates all the electronic valves. The cleaning operation lasts 10 min for each filter and consumes 150 L of pure water. The 20 and 1 μm pore size filters and active charcoal filters are replaced manually every two months.

There are four pressure probes located before and after the filtering loops. The pressure data are monitored by the PLC. If the pressure measured by a probe falls under an established level (1.0

Fig. 4. Above: Picture of the water treatment plant. Below left: 1 m^3 water storage tanks for rejected and pure water. Below right: Arrocampo water discharge monitoring station.

bar) for more than 30 s, the PLC stops the water treatment plant to protect the reverse osmosis system. In this situation, the water pumps stop, and water ceases to flow through the gamma monitoring system. The RAREx headquarter, which is situated in Cáceres city, alerts automatically the technical staff to re-establish the water flow through the stainless-steel vessel to continue the gamma monitoring system. For this purpose, an electronic valve situated before the water treatment plant, which can be operated remotely, opens to allow the water to flow through the gamma monitoring system.

3. Results and discussion

3.1. Preliminary test

In the commissioning of the treatment plant, the conductivity of raw and pure water samples was checked. In addition, ^{3}H concentration and gamma activity of these samples were measured. Table 2 shows conductivity and ^{3}H activity concentration values measured in the different tests performed during the commissioning. As is shown, the conductivity of the raw water is of the order of 1000 μS/cm. The conductivity measured in the produced pure water is less than 20 μS/cm, which meets the requirements of the TRITIUM detector. Also, as expected, the ^{3}H activity concentration in water was not modified by the water treatment processes.

Two water samples (raw and pure) were prepared to measure their gamma emitter radionuclide concentration in a low background laboratory. Both samples were desiccated and after that were placed in Petri dishes. They were measured over two days by an extended range HPGe detector shielded by a 15 cm thick lead layer. The radionuclides systematically analyzed were naturally occurring radionuclides (^{210}Pb, ^{226}Ra and their daughters (^{214}Pb and ^{214}Bi), ^{232}Th and its daughters (^{228}Ac, ^{208}Tl, ^{212}Pb, ^{212}Bi), ^{40}K and ^{7}Be) and long lived fission radionuclides ($^{141,144}Ce$, ^{51}Cr, $^{133,140}Ba$, $^{134,137}Cs$, ^{95}Zr, $^{58,60}Co$, ^{54}Mn, ^{59}Fe, ^{65}Zn, $^{124,125}Sb$ and ^{110m}Ag). ^{131}I was not measured because it is lost during the dessiccation process. For the precise determination of water activity, accredited procedures based on Standard 17025 [11], which is being based on Standard ISO 10703 [13], were used. Table 3 shows the activity levels measured both in raw and pure water samples.

The activities of man-made radionuclides measured were below the detection limit in the raw water. The largest detection limit value measured was 0.039 Bq/L, corresponding to ^{140}Ba, due to its short half-life ($T_{1/2} = 12.75$ d). Concerning naturally occurring radionuclides, their activity levels were below the detection limit except for ^{40}K. This activity level corresponds to a K concentration of 11 mg/L, in agreement with the physicochemical analysis carried out in the year 2019 shown in Table 1. Regarding the pure water gamma analysis, the activities of all radionuclides both natural and man-made were below the detection limit (The ^{40}K activity detection limit was 0.077 Bq/L).

According to the commissioning results, the water treatment plant removes all dissolved salts before the water enters the TRITIUM detector and the beta contribution from beta emitters other than ^{3}H is negligible. In addition, the ^{3}H concentration in water is not modified by the water treatment plant.

Table 2
Conductivity and ^{3}H levels measured in a preliminary test. The measurement accuracy of the conductivity is 0.5%.

Test	Raw water	Rejected water	Pure water
	Average conductivity (μS/cm) ± SD (k = 1).		
1	960 ± 20	1630 ± 30	11.8 ± 0.2
2	970 ± 20	1730 ± 30	12.0 ± 0.2
3	980 ± 20	1700 ± 30	17.5 ± 0.4
4	990 ± 20	1690 ± 30	10.1 ± 0.2
	^{3}H activity concentration (Bq/L)		
1	24 ± 3	26 ± 4	26 ± 4

3.2. Automatic working and remote management

As described in section 2, the PLC allows the automatic operation and cleaning processes of the water treatment plant. The different monitoring parameters are sent to the Cáceres headquarters by the main computer of the RAREx water monitoring station. In this section, the parameters measured by the PLC in automatic mode from June 2019 to March 2020 are reported. All of them were received in the Cáceres headquarters in quasi-real time. Data taken during shutdowns due to issues not related to the water treatment plant, for example, water pressure losses caused by the power supply failures, are not considered.

In Fig. 5, the pressure values of the water inlet of the osmosis plant and the outlet pressure of the filtration system registered every 3 min are shown. As can be seen, the inlet water pressure provided by the pumps ranges from 4 to 6 bar. Sometimes, due to air present in the roughing system due to the cleaning operations, this pressure may fluctuate in a wider range. This is reflected in the water pressure in the outlet from the filtration system. In any case, this does not affect the production of pure water, although it causes the wear of the water supply systems due to frequent starts and stops when the limit pressure is reached.

Fig. 6 shows the pure water flow and conductivity measured every 3 min. It should be noted that a pattern is observed in the water flow production. Every 4 days, the flow reaches a maximum value after which it gradually decreases, due to the alternation of the osmosis systems programmed in the PLC. Besides, a decreasing trend is observed in the flow, which was reversed in October when the first maintenance and cleaning of the osmosis meshes were carried out. It is important to point out that water flow fluctuations do not affect the TRITIUM detector because it operates at a flow of less than 1 L/min. Fig. 6 also shows the conductivity measured in the produced pure water stream. The exceptionally high conductivities, well above the average value, are due to the osmosis stage change and cleaning operations, during which the conductivity increases by up to an order of magnitude. Discarding those high conductivity values, the mean value and its standard deviation is 15 ± 5 μS/cm, with a geometric mean of 14.2 μS/cm and a median of 15.4 μS/cm. Based on these results, the conductivity values of pure water produced, measured directly in the water treatment plant, do not reach grade 3 according to ISO 3696 [8]. However, these conductivity levels are satisfactory enough for the goal of the water treatment plant, which is to provide near-pure water quality to the TRITIUM detector, avoiding the presence of particles and dissolved solids that would significantly decrease the detection efficiency.

3.3. Verification test during routine operation

To check ^{3}H concentration and some physicochemical parameters both in raw and pure water during routine operations of the water treatment plant, water samples ($N = 19$) were periodically taken and analyzed. Table 4 shows the physicochemical parameters

Table 3
Natural and man-made radionuclide activity levels in pure and raw water samples measured by gamma spectrometry. The volume of the water samples measured were 21.5 L for raw water and 10.3 L for pure water.

	Activity levels (Bq/L)							
	^{210}Pb	^{214}Pb	^{214}Bi	^{228}Ac	^{208}Tl	^{212}Pb	^{212}Bi	^{40}K
pure	<0.051	<0.009	<0.007	<0.013	<0.004	<0.006	<0.042	<0.077
raw	<0.167	<0.005	<0.006	<0.009	<0.002	<0.004	<0.026	0.31 ± 0.04
	^{141}Ce	^{144}Ce	^{51}Cr	^{133}Ba	^{140}Ba	^{134}Cs	^{137}Cs	^{95}Zr
pure	<0.004	<0.010	<0.042	<0.002	<0.047	<0.002	<0.003	<0.006
raw	<0.005	<0.014	<0.028	<0.003	<0.039	<0.001	<0.002	<0.003
	^{58}Co	^{60}Co	^{54}Mn	^{59}Fe	^{65}Zn	^{124}Sb	^{125}Sb	^{110m}Ag.
pure	<0.002	<0.003	<0.003	<0.007	<0.005	<0.005	<0.008	<0.004
raw	<0.002	<0.002	<0.002	<0.002	<0.004	<0.003	<0.004	<0.002

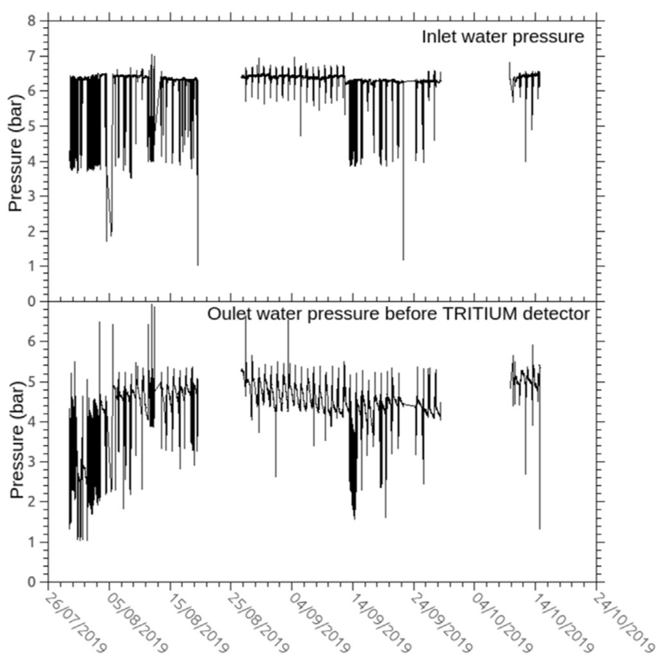

Fig. 5. Above) Inlet water pressure. Below) Outlet water pressure before entering the TRITIUM detector. To facilitate visualization only two months are shown.

measured in raw and pure water, and Fig. 7 shows the ^{3}H activity concentration in both water samples. The verification tests have shown that the water treatment plant has been operating automatically in accordance with the requirements of the TRITIUM detector.

4. Conclusions

This paper describes the design and commissioning of an automatic and remotely operated water treatment system installed at the RAREx water monitoring station, located in the Tagus River close to the ANPP water discharge channel. The objective of this system is to provide near-pure water for a real-time ^{3}H detection system. Initial tests have been carried out and show that the system provided water with a conductivity of less than 12 μS/cm.

The water treatment system was operated routinely for 6 months. During this time, several water quality parameters were measured. Although the average values of the water quality parameters measured in pure water are higher than those specified in the ISO 3696 standard for pure water quality grade 3, the parameters that could affect the ^{3}H detection efficiency, which are turbidity and total dissolved solids, have values that meet the requirements of the TRITIUM detector. It was also found that the water treatment system did not alter the ^{3}H concentration in the water as expected.

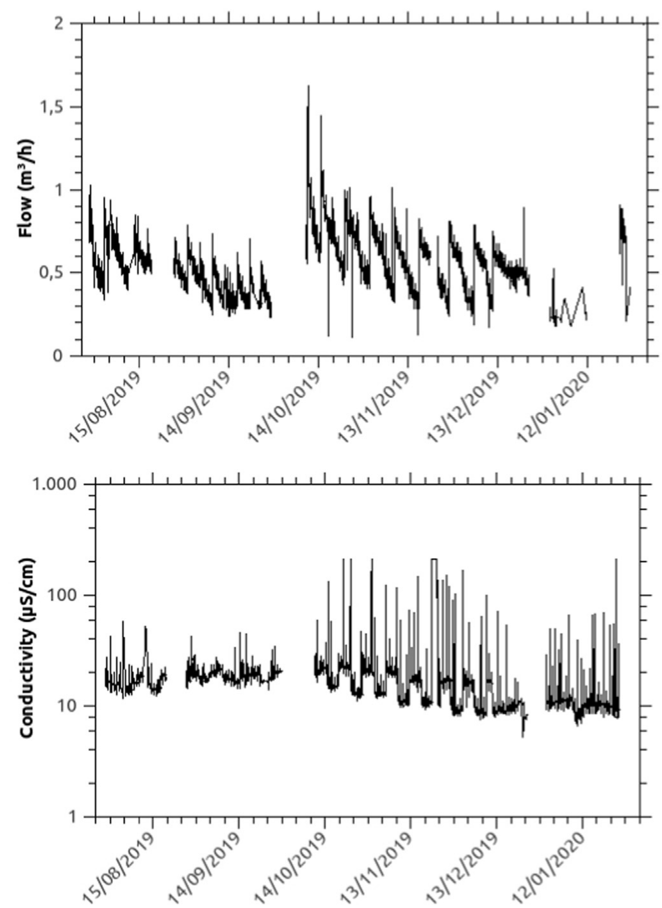

Fig. 6. Above) Pure water flow production over two months. Below) Pure water conductivity over the same period. The conductivity accuracy is 5%.

Table 4
Physicochemical parameters measured in 19 raw and pure water samples compared to ISO 3696 grade 3 reference values [8].

		pH	Oxygen (ppm)	Turbidity (FNU)	Resistivity (MΩ cm)	Conductivity (μS/cm)
Accuracy of measurement device		± 0.02	± 1.5%	± 2.0%	± 1.0%	± 1.0%
ISO 3696 grade 3 reference		5.0–7.5	0.4	Not specified	Not specified	5.0
Pure	**Av ± SD**	7.2 ± 0.7	5 ± 2	-	0.2 ± 0.1	11 ± 10
	Range	6.1–8.5	2 - 10	0 - 7	0.03–0.38	8 - 35
Raw	**Av ± SD**	7.4 ± 0.3	11 ± 4	-	$(9 \pm 2)\cdot 10^{-4}$	$(12 \pm 2)\cdot 10^{2}$
	Range	6.9–8.2	6 - 18	0.2 - 35	$(7 - 12)\cdot 10^{-4}$	800 - 1400

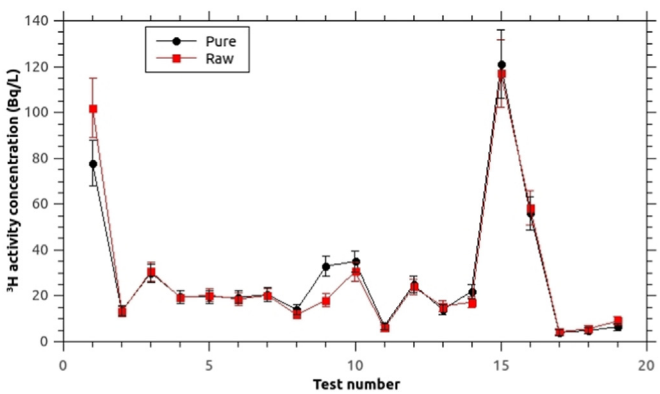

Fig. 7. ^{3}H activity levels measured in raw and pure water samples collected simultaneously during water treatment plant running in routine operations.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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