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Tritium Extraction from Lithium–Lead Eutectic Alloy: Experimental Characterization of a Permeator against Vacuum Mock-Up at 450 °C

Francesca Papa ¹, Alessandro Venturini ^{2,*}, Daniele Martelli ² and Marco Utili ²

¹ Department of Astronautical, Electrical and Energy Engineering, Nuclear Section, Sapienza University of Rome, Corso Vittorio Emanuele II 244, 00186 Rome, Italy; f.papa@uniroma1.it

² ENEA Brasimone, Camugnano, 40032 Bologna, Italy; daniele.martelli@enea.it (D.M.); marco.utili@enea.it (M.U.)

* Correspondence: alessandro.venturini@enea.it

Abstract: Tritium extraction is one of the key open issues toward the development of the WCLL BB (Water-Cooled Lithium–Lead Breeding Blanket) of EU DEMO reactors, and different technologies have been proposed to address it. Among them, the Permeator Against Vacuum (PAV) has promising features, but it has never been tested in a relevant environment. This work presents the first experimental results ever obtained for a PAV mock-up. The experiments were carried out at ENEA Brasimone R.C. with the TRIEX-II facility on a mock-up characterized by a shell and tube configuration and using niobium as a membrane material. The experimental campaign was carried out with LiPb flowing at about 450 °C and 1.2 kg/s, while the hydrogen partial pressure was varied in the range 170–360 Pa. The characterization of the PAV performance was conducted by measuring the hydrogen partial pressure drop across the mock-up and the hydrogen permeated flux through a leak detector calibrated with an external hydrogen calibration cylinder. Moreover, the permeated flux was confirmed by a pressurization test performed measuring the pressure increase on the vacuum side of the PAV. The results constitute the first verification of the possibility to operate a PAV in flowing LiPb and to quantify its capabilities.

Keywords: tritium extraction; WCLL BB; Permeator Against Vacuum; lead-lithium eutectic



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1. Introduction

Among the tritium extraction technologies for the WCLL BB (Water-Cooled Lithium–Lead Breeding Blanket) [1], the Permeator Against Vacuum (PAV) has often been considered as the most promising one [2], but it has been analyzed mainly from theoretical and modeling points of view, while the experiments have been devoted to the measurements of fundamental quantities, such as the diffusivity of materials of interest (e.g., [3]) or the surface effects on permeation (e.g., [4]). Design activities of a PAV for DEMO have also been carried out, especially at CIEMAT and Politecnico di Torino [5].

Permeator Against Vacuums are based on the phenomenon of tritium permeation through a membrane that is, on one side, in contact with the LiPb, which carries tritium, while, on the other side, a vacuum is pumped. The vacuum is made to keep a tritium partial pressure gradient between the two sides of the membrane, so that permeation can take place. The membrane has to be a high tritium permeable material, such as α -iron [6], vanadium, or niobium.

Experimental activities devoted to the characterization of PAV technologies have started both at CIEMAT [7] (made of vanadium, planar configuration) and at ENEA Brasimone [8] (made of niobium, U-tubes configuration). Most recently, a new experimental facility, TEX, has been designed and built at Idaho National Laboratories to test a PAV

made of vanadium [9]. However, no results have been published before the experiments described in this paper.

This paper summarizes the results of the experiments performed in the TRIEX-II facility [10] with LiPb flowing at 1.2 kg/s and 450 °C and with the PAV mock-up described in [8]. Hydrogen in the partial pressure range between 170 and 370 Pa was used to simulate the behavior of tritium in these experiments. This partial pressure range was chosen as it covers the values that are currently considered the reference for design activities and modeling on the WCLL BB.

2. Materials and Methods

2.1. Facility and Test Section Description

TRIEX-II is a facility conceived to test different mock-ups of tritium extraction systems for the WCLL BB. As described in [10], TRIEX-II is a LiPb loop composed principally of a saturator and an extractor where the liquid metal flow is provided by a permanent magnet pump. A storage tank is used to store all LiPb during maintenance periods. The saturator works based on the gas–liquid contactor technology and consists of a column with two structured packings that allow increase of the gas–liquid interface for a better saturation of the hydrogen in the liquid metal. To work correctly, the saturator needs to have a gas dome in its top part, which is constantly kept in a slight overpressure with respect to the external environment by means of an automatic valve controlled by a relative pressure transducer (26.600 G by Barksdale, with a maximum reading of 10 barg and an accuracy of 0.5% of the full-scale output).

The LiPb flow rate in the loop is measured by a thermal mass flow meter (TFM), designed by ENEA in collaboration with Thermocoax. This instrument is based on an enthalpy balance across a heating element: accurately measuring the temperature jump across the heater by resistance thermometers and the power given to the heater, it is possible to estimate the mass flow rate (given the LiPb-specific heat). Its measuring range is between 0.5 and 5 kg/s, and its accuracy is in the order of 0.1 kg/s. The gas system supplies helium, hydrogen, and deuterium flow rates by means of mass flow controllers (El Flow F-212AV by Bronkhorst, with a maximum flow of 250 nl/h and an accuracy of $\pm 0.5\%$ of the reading plus $\pm 0.1\%$ of the full-scale output), so that it is possible to inject a known concentration of Q₂. Moreover, the gas lines are also used to pump a low vacuum in the facility, at least twice, at 150 °C and before loading the alloy at the start of the operation in order to reduce as much as possible the contamination of LiPb by oxygen previously absorbed in the pipe walls.

With respect to the facility described in [10], TRIEX-II (Figure 1) underwent two major modifications before the start of this campaign. The first one is, of course, the installation of the PAV mock-up (called PAV-ONE, Figure 2), which replaces the gas–liquid contactor as the hydrogen extraction system in the facility. Described in detail in [8,11], PAV-ONE is composed of a collector, a vacuum chamber, and 16 U-pipes in niobium joined with a F22 plate. It is designed so that LiPb passes in the vacuum chamber of the mock-up twice before proceeding to flow toward the saturator. In the vacuum chamber, the test temperature of the LiPb is maintained by 4 infrared lamps and monitored by 45 thermocouples. A vacuum is pumped in the vacuum chamber by means of the vacuum station integrated in the leak detector that is described below. A fused silica porthole is installed on the top of the vacuum chamber to allow vision of the niobium tube bundle during operation of the facility. The idea behind this was to monitor the correct operation of the infrared lamps and to check that the thermocouples did not detach from the niobium tube walls.

The second modification was devoted to upgrading the hydrogen permeation sensors (HPS) [12], which are the instruments used to measure the hydrogen partial pressure inside the LiPb. These sensors are composed of a hollow helical-shaped tube made of α -iron connected to a vacuum capacitance gauge (CMR 372 by Pfeiffer Vacuum, with a full-scale output of 100 mbar and a precision of 0.15% of the measured value) and to a vacuum pumping station (HiCube 80 by Pfeiffer Vacuum). This vacuum station has a membrane

pump as the primary pump and a turbo molecular one for the higher vacuum levels. A valve can isolate the helix from the pumping station, allowing for the measurement of the pressure increase caused by the hydrogen permeation through the α -iron. The equilibrium pressure that is reached corresponds to the hydrogen partial pressure in the LiPb. These sensors were modified by minimizing as much as possible the internal volume of the piping that connects the helix with the vacuum gauge and the valve, thus improving the sensor response time, and by replacing most of the fittings with welded joints, thus improving the tightness and thus the accuracy of the sensor. Moreover, the α -iron helices were replaced with new ones.

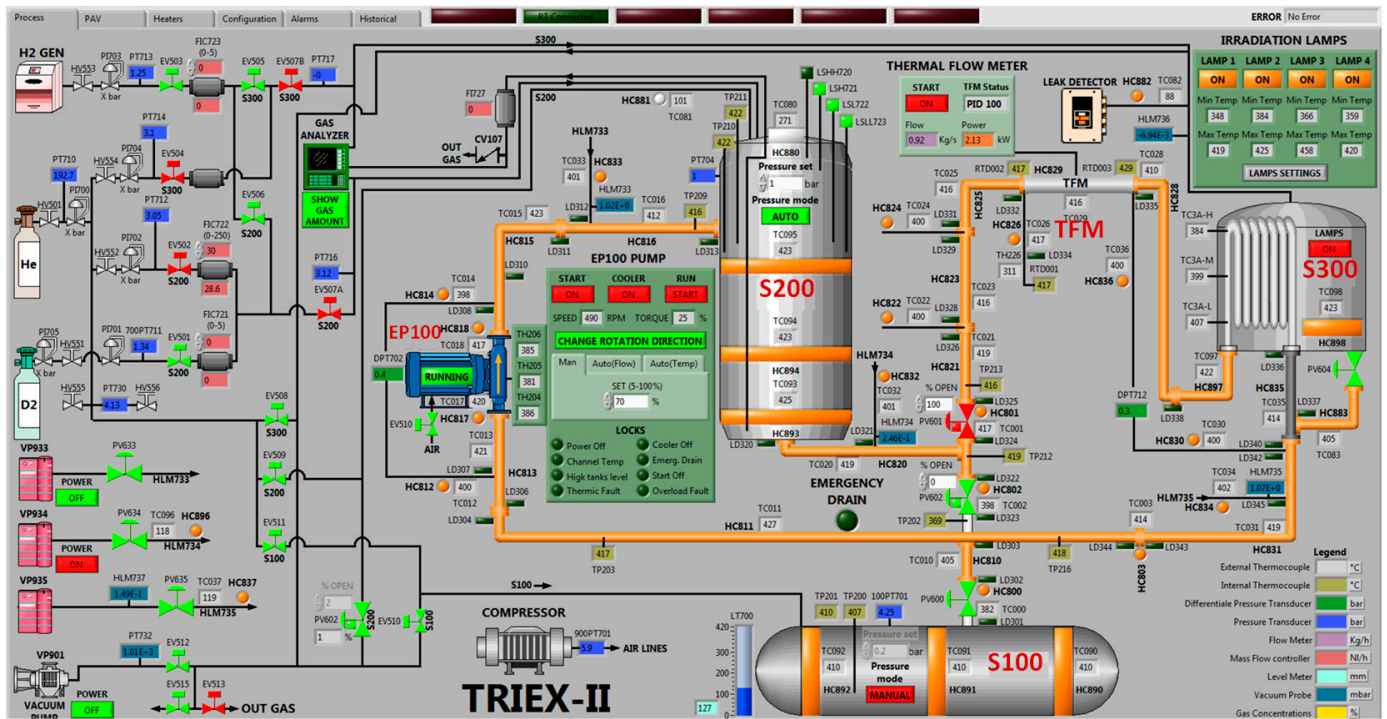


Figure 1. Main window of the Data Acquisition and Control System of the TRIEX-II facility during the heating up toward the test conditions. The main components are S100: storage tank; S200: hydrogen saturator; S300: PAV mock-up; EP100: permanent magnet pump; TFM: thermal flow meter.



Figure 2. Pictures of the PAV mock-up at the end of the manufacturing.

The three installed HPSs measure the hydrogen partial pressure in different locations of the facility: upstream of the saturator, between the saturator and the PAV mock-up, and downstream of the PAV mock-up. The sensors are labeled HLM 733, HLM 734, and HLM 735 and are indicated by Prussian blue boxes in Figure 1.

Besides the HPSs, the leak detector (LD) has been fundamental to characterizing the PAV mock-up by measuring the hydrogen permeated flux. The LD (ASM340 series by Pfeiffer Vacuum) is connected to the vacuum side of the PAV vessel through a vacuum-tight Swagelok line. The instrument is equipped with a vacuum station and a mass spectrometer that can recognize both hydrogen and helium and that performs an integral measurement of the permeated hydrogen in $\text{mbar}\cdot\text{L/s}$, from which it is possible to evaluate the average permeation flux in mol/s and the average flux per unit area.

A wide-range vacuum pressure sensor is installed on the vacuum chamber. This instrument is used to continuously monitor the vacuum level in the chamber and to perform an indirect confirmation of the leak detector measurement: indeed, by closing a gate valve installed on the line that connects the vacuum chamber to the leak detector, it is possible to measure the pressure increase in the vacuum chamber and to correlate it with a hydrogen flux permeating from the niobium tubes.

2.2. Commissioning

Before starting the experimental campaign with PAV, commissioning tests were carried out. These tests are divided into two typologies: pressure and vacuum tests. All the tests were repeated twice, once close to ambient temperature and once at high temperature.

The first test was a pressure test of the LiPb loop at about $50\text{ }^{\circ}\text{C}$. The loop was filled with helium at 1 barg. After about 4 days, the pressure decreased about 0.3 bar ($0.12\text{ mbar}\cdot\text{L/s}$). Then, the temperature was raised to $350\text{ }^{\circ}\text{C}$ and the test was repeated, giving very similar results. After the two tests, the sealing of the loop was considered satisfactory for the needs of the experimental campaign.

The vacuum tightness of the PAV vessel was tested in cold conditions after the PAV installation in December 2021. The PAV vessel remained in vacuum conditions for more than one month. Later, in April 2022, the vacuum test was repeated with the LiPb loop at $450\text{ }^{\circ}\text{C}$ (the temperature on the external side of the vessel ranged from 80 to $150\text{ }^{\circ}\text{C}$, depending on the position).

A different test was devoted to check if the permeation sensors were able to reach a steady state or if possible leakages from the environment would cause the pressure to continue to increase. Figure 3 shows that there are not leakages and that the instruments are able to reach a constant value.

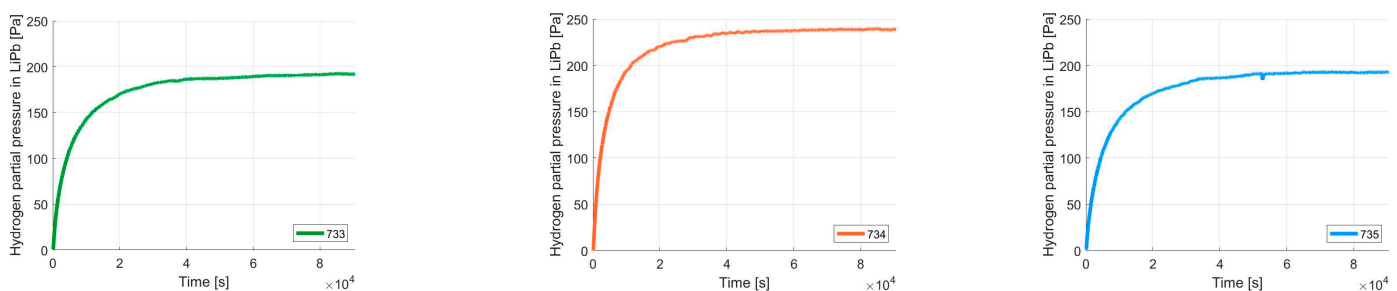


Figure 3. Example of commissioning test for sensors HLM733, HLM734, and HLM735.

At the end of the commissioning tests, calibration of the leak detector was performed with a calibrated leak of $1.23\cdot 10^{-4}\text{ mbar}\cdot\text{L/s}$ (with an accuracy of $\pm 5\%$ and temperature coefficient of 0.2% per $^{\circ}\text{C}$). The calibration was repeated every morning during the campaign to be sure to have a reliable measurement from the instrument. Moreover, the temperature close to the calibrated leak was measured during the calibration procedure in order to correctly take into account the error introduced by temperature changes.

Finally, LiPb was loaded into TRIEX-II facility. After the loading, a series of measurements with the HPSs was performed with the aim to check their reliability and repeatability. An example of two consecutive measurements is reported in Figure 4.

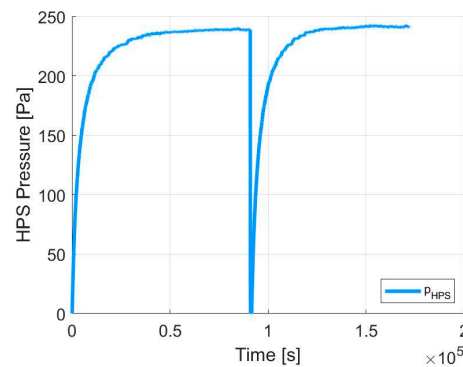


Figure 4. HPS pressure trend in two consecutive measurements.

2.3. Experimental Procedure

After the loading, a mix of hydrogen and helium is injected into the saturator to reach and then maintain constant the hydrogen concentration during the test. The desired mix of hydrogen and helium is made by means of mass flow controllers, instruments that separately regulate the mass flow rates of each gas following the operator needs. The mass flow controllers, supplied by Bronkhorst, are composed of a thermal mass flow sensor, a precise control valve, and a controller that changes the position of the valve according to the input of the operator.

The concentration in LiPb reaches a value lower than the concentration in the gas, as the equilibrium value is a balance between the quantity of hydrogen solubilized in the saturator and that extracted by the PAV mock-up.

When the steady-state is reached, 3 kinds of measures are carried out:

- Partial pressure measurement with HPSs at the inlet and the outlet of PAV mock-up;
- Permeated flux with the LD;
- Pressure increase in the PAV mock-up vessel.

The HPSs positioned in LiPb loop at the inlet and at the outlet of the PAV mock-up measure different partial pressures, as shown in Figure 5a. This difference is due to the PAV mock-up extraction. The hydrogen partial pressure at the outlet of the PAV mock-up is actually measured twice: indeed, two HPSs are installed in two different positions (downstream of the PAV, HLM735, and upstream of the saturator, HLM733; as shown Figure 5b).

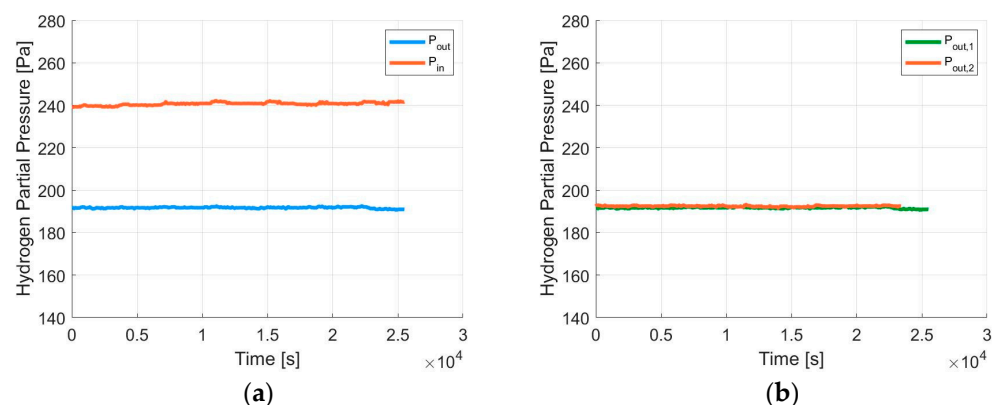


Figure 5. Examples of partial pressure measured by the HPSs at the inlet and at the outlet of the PAV mock-up (a) and of partial pressure measured by the HPSs installed at the outlet of the PAV mock-up by HLM733 (orange line) and HLM735 (green line) (b).

The LD is set to detect hydrogen, and it constantly measures the flux that permeates through the niobium tubes. Moreover, an additional indirect measurement of the flux is performed by isolating the vessel of the PAV mock-up by means of a specific gate valve

(pressurization test). The pressure inside the vessel increases, and, knowing the initial pressure, the final pressure, and the duration of the pressurization, it is possible to calculate the flux of hydrogen that permeates through the niobium tubes. This calculation has some uncertainties, but it is used to have a verification of the leak detector measurement. From the pressure increase, it is possible to calculate the permeation rate in mbar L/s:

$$\Phi[\text{mbar}\cdot\text{L/s}] = \frac{\Delta p}{\Delta t} \cdot V_{PAV} \quad (1)$$

where Δp is the pressure increase due to the permeation in the considered time Δt , and V_{PAV} is the volume of the PAV vessel. From the permeation rate, the permeated flux is derived by:

$$\Phi[\text{mol/s}] = \frac{\Phi[\text{mbar}\cdot\text{L/s}]}{10 \cdot R \cdot T_{av}} \quad (2)$$

where T_{av} is an average temperature in the PAV vessel. This temperature cannot be directly measured, and it is estimated by averaging the temperature of the PAV vessel wall and the temperature of the niobium tubes, but this process introduces an uncertainty into the calculation. For this reason, the pressurization test is conceived mainly as a way of confirming the order of magnitude of the flux measured by the leak detector.

Three tests were performed at about 450 °C, and their conditions are shown in Table 1, which reports the average hydrogen partial pressure and LiPb temperature measured at the inlet of the PAV mock-up, together with the average LiPb flow rate measured by the thermal flow meter. Each test was repeated at least twice to check the repeatability of the results. Each experimental value is associated to a measuring uncertainty, evaluated by considering the instrument accuracy, the oscillation of the signal during the test, and the error introduced by the electrical noise in the signal chain. The same evaluation was performed for every acquired signal, but it was decided not to show an error bar in the plots to not jeopardize their readability.

Table 1. Test matrix for the PAV experimental campaign.

Test	Measurement	p_{in} [Pa]	T [°C]	LiPb $\dot{m}$ [kg/s]
1	1	170 ± 0.52	453 ± 2.56	1.19 ± 0.10
	2	170 ± 0.39	458 ± 2.24	1.19 ± 0.10
2	1	241 ± 0.83	458 ± 2.26	1.19 ± 0.10
	2	239 ± 0.60	443 ± 2.21	1.19 ± 0.10
	3	241 ± 0.82	444 ± 2.25	1.18 ± 0.10
	4	244 ± 0.53	446 ± 2.21	1.18 ± 0.10
3	1	359 ± 0.86	446 ± 2.22	1.20 ± 0.10
	2	366 ± 1.33	445 ± 2.22	1.20 ± 0.10
	3	366 ± 0.90	446 ± 2.21	1.20 ± 0.10

3. Results

The main results of the experimental campaign are summarized in Tables 2 and 3. Table 2 shows the hydrogen partial pressure at the inlet and at the outlet of the PAV mock-up. A partial pressure difference across the PAV mock-up exists in every measurement, demonstrating that the extraction is occurring and remains almost constant within each test, demonstrating the repeatability of the measurements. The duration of each measurement was at least 6 h, during which the signal remained practically constant.

Table 3 shows the permeated flux, which is the most relevant parameter for the design of the PAV system for the WCLL BB, as it can be used to estimate the size of a component able to extract the amount of tritium that has to be routed to the fuel cycle. The first column of data contains the measurements of the leak detector in mbar·L/s, which were then converted into mol/s by dividing the values by $10 \cdot R \cdot T_{LD}$ (the results of the conversion are shown in the second column of data), where R is the gas constant in J/(mol·K), 10 comes

from the conversions of mbar to Pa and liter to m^3 , and T_{LD} is the temperature in the chamber of the leak detector in K.

Table 2. Hydrogen partial pressure at the inlet and at the outlet of the PAV mock-up (with experimental uncertainty).

Test	Measure	P_{in} [Pa]	P_{out} [Pa]
1	1	170 ± 0.52	158 ± 0.58
	2	170 ± 0.39	159 ± 0.52
2	1	241 ± 0.83	192 ± 0.51
	2	239 ± 0.60	192 ± 0.46
	3	241 ± 0.82	194 ± 0.90
	4	244 ± 0.53	198 ± 0.48
3	1	359 ± 0.86	249 ± 0.79
	2	366 ± 1.33	256 ± 0.92
	3	366 ± 0.90	255 ± 0.74

Table 3. Measured permeation rate and hydrogen permeated flux calculated from the data of the leak detector and measured by pressurization test (with experimental uncertainty).

Test	Measurement	Permeation Rate [mbar·L/s]	Permeated Flux [mol/s]	Flux Press. [mol/s]
1	1	$1.10 \cdot 10^{-3} \pm 5.71 \cdot 10^{-5}$	$4.58 \cdot 10^{-8} \pm 2.75 \cdot 10^{-9}$	$4.42 \cdot 10^{-8} \pm 1.59 \cdot 10^{-8}$
	2	$1.20 \cdot 10^{-3} \pm 5.25 \cdot 10^{-5}$	$4.67 \cdot 10^{-8} \pm 2.58 \cdot 10^{-9}$	
2	1	$1.28 \cdot 10^{-3} \pm 6.30 \cdot 10^{-5}$	$3.00 \cdot 10^{-8} \pm 1.67 \cdot 10^{-9}$	$3.31 \cdot 10^{-8} \pm 1.18 \cdot 10^{-8}$
	2	$1.12 \cdot 10^{-3} \pm 4.18 \cdot 10^{-5}$	$2.56 \cdot 10^{-8} \pm 1.18 \cdot 10^{-9}$	
	3	$6.17 \cdot 10^{-4} \pm 8.79 \cdot 10^{-5}$	$1.73 \cdot 10^{-8} \pm 1.51 \cdot 10^{-9}$	
	4	$4.81 \cdot 10^{-4} \pm 2.19 \cdot 10^{-5}$	$1.15 \cdot 10^{-8} \pm 5.90 \cdot 10^{-9}$	
3	1	$1.41 \cdot 10^{-3} \pm 9.93 \cdot 10^{-6}$	$3.08 \cdot 10^{-8} \pm 6.84 \cdot 10^{-10}$	$6.93 \cdot 10^{-8} \pm 2.47 \cdot 10^{-8}$
	2	$1.71 \cdot 10^{-3} \pm 6.77 \cdot 10^{-5}$	$3.75 \cdot 10^{-8} \pm 1.88 \cdot 10^{-9}$	
	3	$1.80 \cdot 10^{-3} \pm 1.10 \cdot 10^{-4}$	$3.95 \cdot 10^{-8} \pm 2.80 \cdot 10^{-9}$	

Finally, the last column shows an estimation of the hydrogen permeated flux performed by closing the gate valve that connects the PAV chamber to the leak detector and measuring the pressure increase in the PAV chamber. The difficult estimation of the average temperature is reflected by the large uncertainties associated with these values. Nonetheless, the pressurization tests served as a qualitative confirmation that the used leak detector is reliable in this kind of experiment.

The permeated fluxes were also divided by the area of the niobium tubes to obtain an average flux per unit area, which is another useful input for designing the tritium extraction unit of the WCLL BB. The values are shown in Table 4.

Table 4. Hydrogen permeated flux per unit area (with experimental uncertainty).

Test #	Meas. Number	Flux per Unit Area [mol/(s·m ²)]
1	1	$1.08 \cdot 10^{-7} \pm 6.47 \cdot 10^{-9}$
	2	$1.10 \cdot 10^{-7} \pm 6.07 \cdot 10^{-9}$
2	1	$7.06 \cdot 10^{-8} \pm 3.93 \cdot 10^{-9}$
	2	$6.03 \cdot 10^{-8} \pm 2.77 \cdot 10^{-9}$
	3	$4.08 \cdot 10^{-8} \pm 3.55 \cdot 10^{-9}$
	4	$2.70 \cdot 10^{-8} \pm 1.39 \cdot 10^{-9}$
3	1	$7.26 \cdot 10^{-8} \pm 1.61 \cdot 10^{-9}$
	2	$8.82 \cdot 10^{-8} \pm 4.42 \cdot 10^{-9}$
	3	$9.30 \cdot 10^{-8} \pm 6.58 \cdot 10^{-9}$

The hydrogen fluxes in mol/s are reported in Figure 6 for the three tests. The order of magnitude of the fluxes was confirmed by performing the pressurization tests, whose results are also included in Figure 6. The pressurization tests were performed once per test.

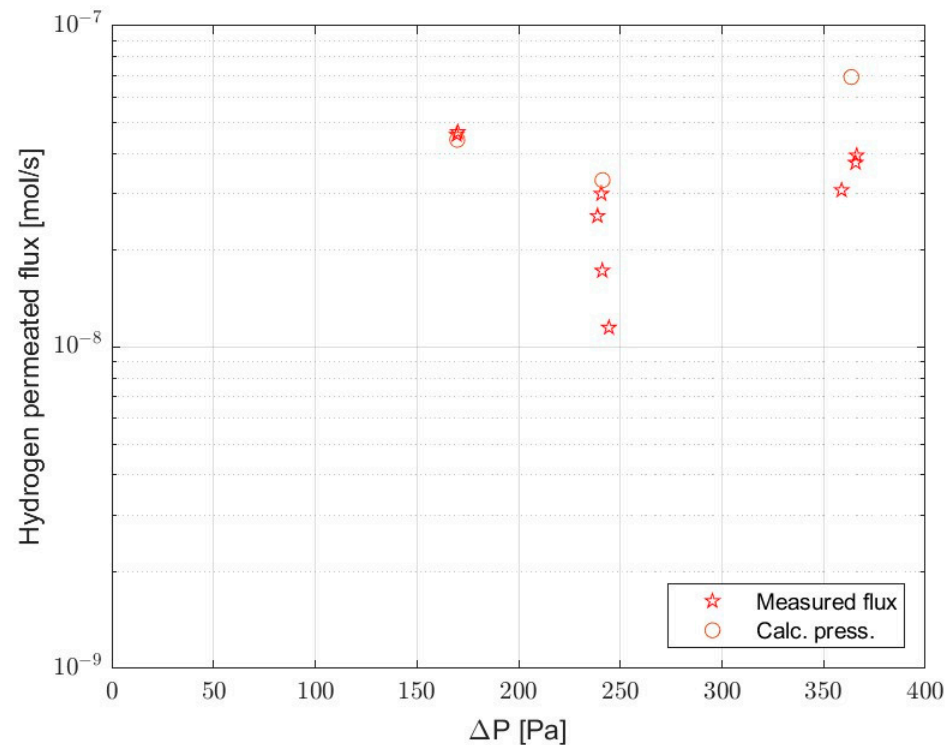


Figure 6. Hydrogen permeated fluxes for the 3 tests (stars) compared with the fluxes calculated from the data of the pressurization tests (circles). The X-axis refers to the pressure difference that drives the permeation (difference between the LiPb at the entrance of the niobium tubes and the pressure in the vacuum chamber).

4. Discussion and Conclusions

The activity to characterize the PAV technology started with a conceptual design of a PAV mock-up [5], which was engineered during the following years [8]. Afterwards, the techniques to manufacture the joint between the F22 plate and the Nb tubes of the mock-up were selected, and, in the meantime, a selection of the most suitable instruments and pieces of equipment was performed, in parallel with a refurbishment of the TRIEX-II facility to host the PAV mock-up [11]. The manufacturing of the first PAV mock-up for LiPb to ever be completed, and its successful commissioning, constitutes an important milestone toward the development of the WCLL TER (Tritium Extraction and Removal system).

After the installation of PAV-ONE in TRIEX-II, a first experimental campaign was carried out with the aim of characterizing the mock-up performances at different temperatures and hydrogen partial pressures. The experimental campaign demonstrated that the manufacturing techniques can withstand the harsh conditions of a LiPb flow, but also, more importantly, that the PAV technology can be used to extract hydrogen isotopes from a LiPb flow. This paper presented the results of the three tests carried out at about 450 °C, but four additional tests at 350 °C were performed, and their results are currently being analyzed.

The tests highlighted that the permeated flux is in the order of 10⁻⁸ mol/s for the PAV mock-up. These results are also confirmed by the pressurization tests, which indicate similar orders of magnitude for the permeated flux, and by the signals of the hydrogen permeation sensors, which show a significant drop in the hydrogen partial pressure across the mock-up that is repeatable within each test and that increases with the inlet partial pressure.

These results are the first experimental assessment of the performances of the PAV technology and mark a milestone for the development of this concept. The measured

permeated fluxes, and the associated drops in the hydrogen partial pressure in LiPb, are deemed to be encouraging, also considering that no treatments were performed on the niobium before its installation in TRIEX-II (e.g., no cleaning of the external surface to remove oxides). However, further activities will be needed to improve the knowledge of this technology and the assessment of its performances.

In the future, new tests with niobium tubes of different lengths and inner diameters would be interesting to better understand the impact on the extraction of the hydrogen transport in the liquid LiPb, leading to greater awareness of the scaling laws [13]. The scaling laws will be important to support a sound design of the PAV for the WCLL BB of EU DEMO.

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Conflicts of Interest: The authors declare no conflict of interest.

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