Construction of a test facility and the test of an active tritium permeation barrier

Joshua KOHPEIß[1]*, Stefan WELTE[1], Ion CRISTESCU[1], Nancy TUCHSCHERER[1]

1- Karlsruhe Institute of Technology (KIT), Institute for Astroparticle Physics, Tritium Laboratory Karlsruhe, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

*Corresponding author: joshua.kohpeiss@kit.edu

Abstract - In future fusion or fission reactors, tritium permeation may present a serious challenge. In order to separate the water steam cycle from gas streams containing significant amounts of tritium, a permeation barrier is necessary. Tritium permeation into the environment through steam generators and heat exchangers can be a significant hazard regarding radiation and environmental safety.

In the scope of the project Transversal Actions for Tritium (TRANSAT) a facility has been set up to perform tests on various scaled and functioning permeation barrier mock-ups at the Tritium Laboratory Karlsruhe (TLK). The facility was built in a standard glove box unit in accordance to the technical terms and requirements of tritium handling at TLK. The behavior of an active permeation barrier was investigated.

Within the first series of TRANSAT experiments, four different mock-ups have been tested for tritium permeation. Migrated tritium is oxidized to tritiated water (HTO) using Carulite reactors and molecular sieves for HTO trapping. This paper will present the construction, set up and commissioning of the facility as well as the first series of TRANSAT experiments including their evaluation.

Keywords - Tritium permeation experiment, active permeation barrier, tritium test facility, Tritium Laboratory Karlsruhe

I. INTRODUCTION

Tritium permeation is a significant issue regarding radiation safety in large scale tritium facilities like fusion power plants. Because of high permeability at high temperatures, undesired tritium permeation may take place. Looking for example on the steam circuit for power generation of a future fusion power plant (as simplified in Figure 1), tritium permeation has to be avoided in order to mitigate the migration of tritium into the environment and to minimize the exposure risk for personnel.

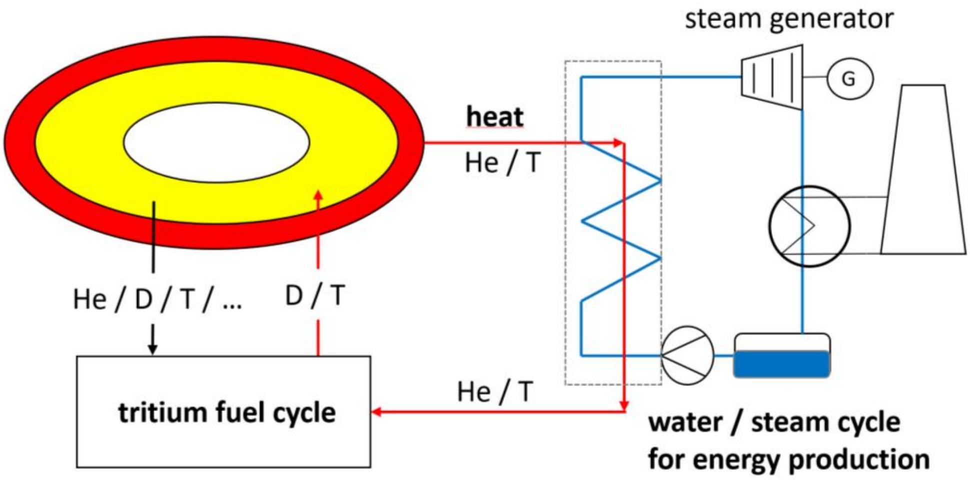


Figure 1 – Simplified schematic of the heat transfer from a fusion power plant into the steam circuit

Therefore, the interface for the heat transfer between the blanket and the water steam circuit for power generation, where tritiated gases are sent through heat exchangers, needs a reliable tritium permeation barrier.

For the investigation of such a tritium barrier and different permeation mock-ups in general, a facility was built at the Tritium Laboratory Karlsruhe (TLK), that enables tritium experiments under specific conditions, such as a wide range of temperatures, pressures and tritium concentration. The experiments shall enable the experimental validation of permeation mitigation concepts under relevant operation conditions.

Within the scope of Transversal Actions with Tritium (TRANSAT), four different heat exchanger mock-ups were tested with the goal to proof the principle of an active barrier concept. The experiments were performed within the safety framework of the Tritium Laboratory Karlsruhe[1].

II. EXPERIMENTAL SETUP

The experiment is confined in a standard glove box compartment at TLK. All TLK standards and rules in accordance with the TLK safety framework were applied[1]. Connections to tritium supply, tritium waste gas processing and auxiliary media supply are provided by the TLK infrastructure[2].

The general design considerations of the facility are:

- Tritium experiments of up to 10 TBq inventory at 70 bar and 450°C
- Technical rules for tritium handling and confinement at TLK[1]
- Pressure equipment directive (PED)
- AD 2000 directive

The experimental setup (see Figure 2) includes:

- Flexible barrier-mockup interface
- Tritium and media supply section
- Analytical section
- Exhaust interface

- Separate desorption setup for molecular sieves and catalysts

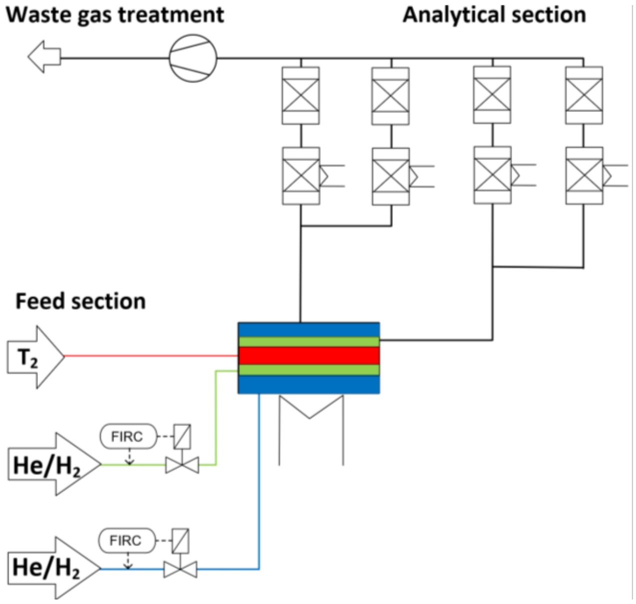


Figure 2 - Simplified flow chart

II.A. Barrier Mock-Up

The barrier mock-ups are functional and tritium compatible triple coaxial pipe in pipe assemblies. Nevertheless, they are not designed to function as heat exchangers. They have three chambers separated by thin walls, from which the intermediate chamber consists of small gaps that are purgeable, shown in Figure 3. The central chamber is usually loaded with a tritium sample. The barrier principle is to remove tritium that permeated from the central chamber into the purge gaps to decrease tritium permeation into the jacket (outer chamber).

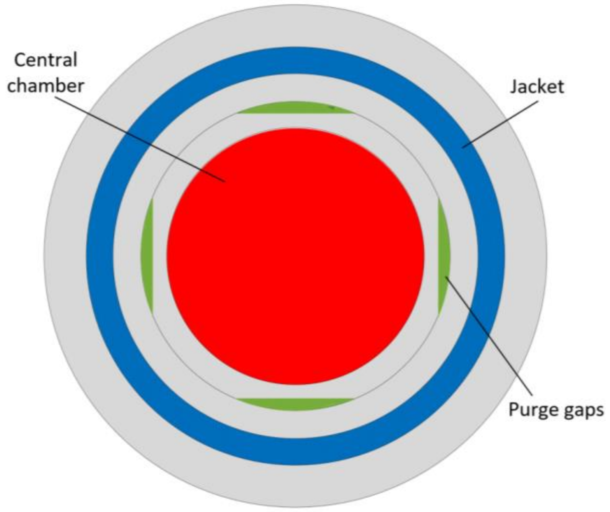


Figure 3 – Cross-section of the Barrier mock-up consisting of three volumes separated by walls (316L)

Four different permeation mock-ups with different purge gap geometries are tested: The purge gaps are milled on the outside of the central chamber shell with a width of 1.7 mm and a depth of 0.045 mm. For each mock-up respectively, there are 2, 4, 6 or 8 equally distributed gaps on the perimeter of the shell. For the central chamber, a ø16 x 1,5 mm tube was used. The purge gaps are surrounded by a ø20 x 2 mm tube. For the jacket, a ø33,7 x 3,5 mm tube was used. The tube material was SS316L. These mock-ups are connected to the experiment in a way, that they are easily exchangeable.

II.B. Tritium and Media Supply Section

To enable high pressure experiments, inactive media supply of up to 28 bar can be fed from a gas cabinet, which has an interface to the primary system in the glove box. Tritiated gases are fed at 0.95 bar using tritium transport cylinders at the tritium supply branch inside the glove box.

Figure 4 – The feed section comprises upstream pressure controllers, valves and mass flow controllers

As shown in Figure 4, four branches are fitted with mass flow controllers (max. 100 l h^{-1} stp) and an upstream pressure controller (max. 70 bar) for the regulation of the purge gas streams into the different chambers of the permeation mock-up. The temperature of the mock-up is heated by an electrical heater to 450°C.

II.C. Analytical Section

Tritiated gases from the central chamber, the purged gaps and the jacket are collected in analytical branches, oxidized to HTO, using Carulite reactors, and trapped on molecular sieves. Both the catalysts and the molecular sieves are designed exchangeable and can be transported through the air lock of the glove box. In order to determine the amount of tritium captured during an experiment, they have to fit either inside a calorimeter for the measurement of high tritium inventories[3] or into a desorption setup for lower tritium inventories. The catalysts are designed to have a sufficient water production potential, whereas the molecular sieves are designed to store the generated amount of water to enable a continuous conversion during the experiment.

II.D. Exhaust

Remaining gases are released into a vessel, compensating high pressures with a dedicated buffer volume. Depending on the composition, the gases are pumped into a specific area of the central tritium retention system for waste gas treatment[4].

II.E. Desorption Setup

For the experimental evaluation, a desorption setup was installed in a fume hood. Its main functions are:

- Recovering tritiated water from the molecular sieves and catalysts
- Regeneration of the molecular sieves
- Regeneration of the catalysts

Hence, the desorption setup is equipped with an air supply for oxidation, a nitrogen supply, a sheath heater and a cold trap.

In parallel with the manufacturing of the primary system and its implementation into the second containment, the technical documentation of the facility had to be prepared. The following documents are necessary:

- Safety description of the facility and the process summarizing the used mechanical and electrical components, the safety assessment, safety engineering and safety circuits.
- Flow diagram as an interface between design and process.
- Bill of implemented components for material traceability and replacement in case of maintenance
- Quality inspection documentation
- Operating manuals of components
- Operating instructions of the facility for reproducible experiments

III. FUNCTIONAL TESTING AND COMMISSIONING

For a safe and reliable experimental procedure, several tests are necessary during the facility commissioning:

- Leak rate proof ($<10^{-9}$ mbar l s^{-1} per connection) in vacuum and overpressure of the primary system
- Electrical safety check
- Loop check and test of safety circuit to proof the correct communication of the local control system
- Function of valves and plausibility of sensors
- Leak rate proof of the second containment (< 1 Vol.% h^{-1})
- Functionality of the below atmospheric pressure system and the tritium retention system

After functional testing, the commissioning is performed. The general functionality, accessibility and operability is tested applying usual operating parameters. Non-tritiated gases are used in order to prove the reliable operation of all parts of the experiment, with the following result:

The facility was operated successfully with a gas mixture to ensure the functionality of the experimental process, including flow through and pump down capabilities, pressure and flow controlling, H_2O generation and storage in the analytical section. The commissioning procedure also ensured the successful combined process of the different process controllers. In a test run, the desorption setup was able to recover up to 99% of the produced water that was stored on the molecular sieves.

IV. PROCEDURES

Several procedures are necessary in order to cope with all the experimental requirements (see Figure 5):

- Gas mixing
- Feeding
- Experiment
- Sampling
- Regeneration

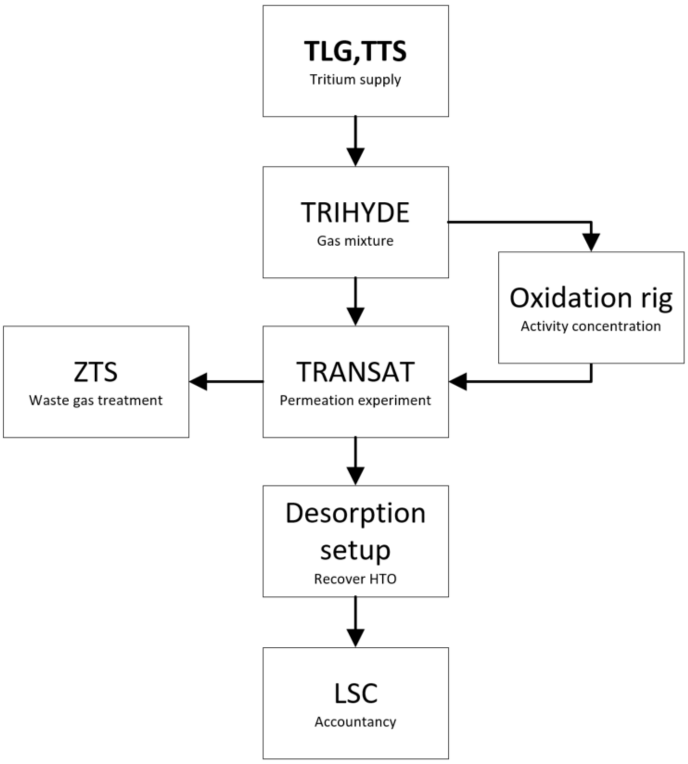


Figure 5 - TLK facilities used for production and analyzing sample gases and for treatment of waste gases (abbreviations in main text)

IV.A. Gas Mixture

In order to provide the experiment with the specified amount of tritium, several TLK facilities are necessary: Tritium needs to be extracted from the tritium storage beds on the Tritium storage system (TLG). The tritium is transferred via the Tritium Transfer System (TTS) into the Tritium Hydrogen Deuterium facility (TRIHYDE), where a specific mixture of helium and tritium with an accuracy of 1% is prepared[2, 5]. To analyze the gas mixture, samples of the mixture will be put into a separate rig, where the tritiated gas is oxidized, absorbed into water bubblers and measured by liquid scintillation counting (LSC).

IV.B. Feeding Procedure

The primary system will be evacuated completely, the gas mixture is then fed into the central chamber of the permeation mock-up. With the calculated and analyzed tritium activity concentration, the absolute amount of tritium injected into the experiment can be calculated via pVT (pressure, volume and temperature) calculation. The tubing of the primary system, except of the permeation mock-up, will be purged with helium and evacuated, to keep the activity background on a low level. For TRANSAT experiments, the central chamber was put to 28 bar at room temperature by adding helium with a tritium partial pressure of 0.7 bar. Before the experiment starts, the used tubing will be purged and evacuated with helium once more.

IV.C. Experiment

After feeding is completed, the temperature is increased to 450°C by the sheath heater, the pressure inside the chamber reaches 70 bar. Then, the purge gaps and the outer chamber are purged with a 5%-hydrogen-in-helium mixture. Mass flow controllers regulate the flow rate to the value defined by the experimenter whereas the pressure is regulated with the upstream pressure controller. Permeated tritium in the gaps and the outer chamber is sent to the analytical section. The experiment runs for typically seven days at constant temperature, pressure and gas flow.

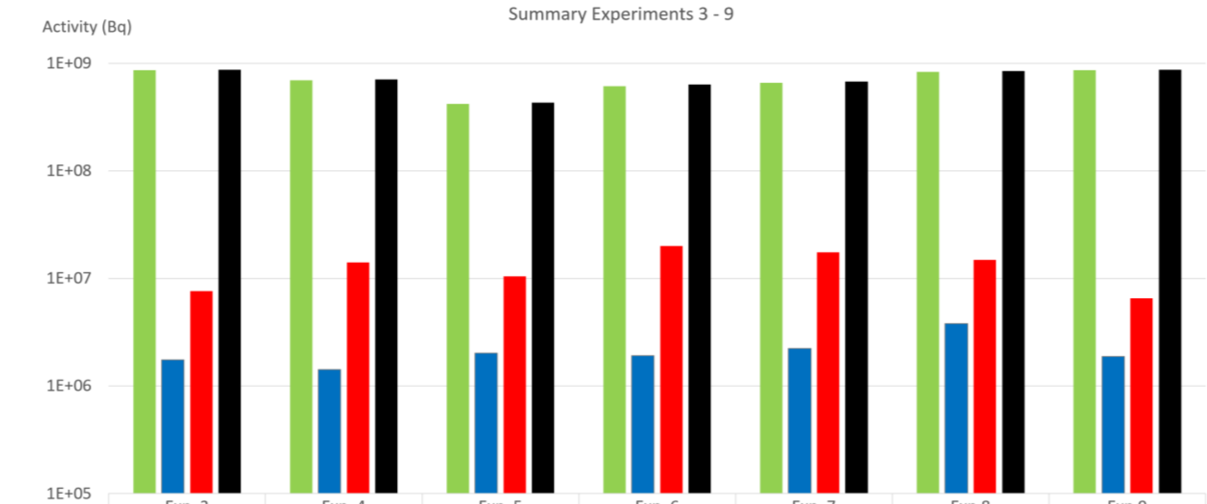
IV.D. Sampling

After purging the chambers, each gas stream is sent to a separate and redundant analytical branch respectively, which is a combination of a catalyst for tritium oxidation to HTO and a molecular sieve for HTO storage by adsorption. A hydrogen fraction of 5% in the helium carrier gas is used, so that the generated and stored amount of water is sufficient to withdraw a measurable amount for sampling. After the experiment, the remaining tritium gas mixture of the central chamber is released and stored inside a third analytical branch. The facility is put into a standby state. Catalysts and molecular sieves are disassembled, plugged and installed either in the desorption setup or in the calorimeter, depending on the activity level.

IV.E. Regeneration

In the desorption setup, the catalyst or molecular sieve is heated with a sheath heater to maximum 300°C. A molecular sieve is purged with nitrogen whereas a catalyst is purged with air. The HTO from the molecular sieve and the catalyst is trapped downstream in a LN2-cold trap. The captured HTO is determined by the weight difference of the cold trap. The purge gas allows the catalysts and molecular sieves to regenerate, so that they can be used in further experiments with their full oxidation potential for catalysts and full water storing capacity for molecular sieves. In the last step, the tritium activity of the trapped water is measured using LSC. As a result, the absolute activity of each cylinder used in the experiment is determined, enabling a complete tritium accountancy of the experiment.

V. RESULTS



	Exp. 3	Exp. 4	Exp. 5	Exp. 6	Exp. 7	Exp.8	Exp.9
Gap LSC	8.6E+08	6.9E+08	4.2E+08	6.1E+08	6.6E+08	8.3E+08	8.6E+08
Jacket LSC	1.8E+06	1.4E+06	2.0E+06	1.9E+06	2.2E+06	3.8E+06	1.9E+06
Central LSC (End)	7.6E+06	1.4E+07	1.0E+07	2.0E+07	1.8E+07	1.5E+07	6.5E+06
Sum End	8.7E+08	7.1E+08	4.3E+08	6.4E+08	6.8E+08	8.5E+08	8.7E+08

Figure 6 - Experimental results: Six reproducible experiments were performed including all four heat exchangers. The figure contains the accounted activities for the purge gap, the jacket, the remaining gases from the central chamber and their sum for each experiment respectively.

The results of the experiments with the four permeation mock-ups are shown in Figure 6. The measured tritium activity in total, of the central chamber, the purged gaps and the jacket are visualized:

Experiments 1 and 2 are commissioning experiments necessary to prove the tritium compatibility of the plant and to find the optimal processing parameters. They were not done with a reproducible procedure, hence they are not representative. Since experiment no. 3 all experiments were performed with the same parameters (Chapter IV) and consistent results: The central chamber was fed with a helium-tritium mixture of 1 GBq tritium activity. Between 430 and 870 MBq were accounted with the measurement procedures. The largest relative fraction with at least 97% from the tritium activity was measured in the purge gaps. Less than 1% was found in the jacket. The remaining 1-3% were measured as the remaining gaseous activity of the central chamber. The influence of the various amounts of purge gaps of the different heat exchangers is not significant, because the measured activities did not exceed the calculated precision.

In the performed experiments, almost the complete amount of tritium permeated through the wall, where it was removed in the purge gaps, so that only a remarkable small amount could migrate into the jacket. The activity measured in the jacket was at least two orders of magnitude lower than the activity measured in the purge gaps.

VI. CONCLUSIONS

Tritium permeation is a significant challenge to radiation safety of tritium plants. A tritium permeation barrier will be necessary in all sections of a plant, where hot tritiated gas streams are contacting non tritiated systems (e.g. heat exchangers). At TLK, a facility has been set up that enables tests of permeation mock-ups and devices at high temperatures and pressures in a wide range of tritium activity with high reproducibility.

The tested active tritium permeation barrier successfully decreases the permeating amount of tritium into the jacket by two orders of magnitude. Within the experiments, the barrier principle is proven to be successful. The examined active barrier concept is a suitable option for tritium permeation mitigation.

This work was done in the frame of the TRANSAT project. The views and opinions expressed herein do not necessarily reflect those of the European Commission. The authors would thank the staff from TLK for the fruitful collaboration during the set-up of the facility.

VII. REFERENCES

1. S. WELTE, U. BESSERER, D. OSENBERG & J. WENDEL (2015) Tritium Laboratory Karlsruhe: Administrative and Technical Framework for Isotope Laboratory Operation, Fusion Science and Technology, **67**, *3*, 635-638, https://doi.org/10.13182/FST14-T98
2. L. DÖRR et al. (2005) The Closed Tritium Cycle of the Tritium Laboratory Karlsruhe, Fusion Science and Technology, **48**, *1*, 262-26, https://doi.org/10.13182/FST05-A924
3. D.DEMANGE et al. (2012) Tritium Laboratory Karlsruhe: Overview of R&D at TLK for Process and Analytical Issues on Tritium Management in Breeder Blankets of ITER and DEMO, Fusion Engineering and Design, **87**, 1206-1213
4. U. BESSERER, L. DÖRR & M. GLUGLA (2008) Tritium Confinement, Retention, and Releases at the Tritium Laboratory Karlsruhe, Fusion Science and Technology, **54**, *1*, 160-163, https://doi.org/10.13182/FST08-A1786
5. S. NIEMES et al. Accurate Reference Gas Mixtures Containing Tritiated Molecules: Their Production and Raman-Based Analysis. Sensors 2021, **21**, 6170, https://doi.org/10.3390/s21186170