Fusion Science and Technology

ISSN: 1536-1055 (Print) 1943-7641 (Online) Journal homepage: www.tandfonline.com/journals/ufst20

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FUSION SCIENCE AND TECHNOLOGY

DOI: https://doi.org/10.1080/15361055.2026.2672904

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Received January 7, 2026
Accepted for Publication May 6, 2026

Abstract — *The KArlsruhe TRitium Neutrino (KATRIN) experiment aims to determine the effective mass of the electron antineutrino by investigating the tritium β-spectrum close to the kinematic end point. The Tritium Laboratory Karlsruhe hosts and operates the tritium parts of the experiment. A dedicated tritium loop system is tasked to provide the < 0.1% stabilized flow rate of tritium gas into the KATRIN source, maintaining a throughput of 40 gd^{-1} and a tritium purity >95%, while at the same time acting as the interface to the established tritium handling infrastructure of the laboratory, reliably working for more than 3 decades. Since KATRIN's start of tritium operation in May 2018, more than 1350 operation days of the tritium "loop" system combined with KATRIN's tritium source have been achieved. This paper summarizes the tritium operation experience gained with special emphasis on the permeator performance, a key component enabling direct internal recycling.*

Keywords — *Tritium, fuel cycle, permeator, direct internal recycling, KATRIN.*

I. INTRODUCTION

The aim of the KArlsruhe TRitium Neutrino (KATRIN) experiment is the determination of the effective mass of the electron antineutrino with a projected sensitivity of 0.2 eV/c^2 (90% confidence level) [1]. After first campaigns with deuterium and traces of tritium in 2018 [2,3], KATRIN started full tritium operation in March 2019. With data from the first five tritium measurement campaigns, a new upper limit of 0.45 eV/c^2 for the neutrino mass was set [4]. By the end of 2025 more than 20 tritium measurement campaigns had been conducted, culminating in more than 1350 days of high-purity tritium operation and an integral throughput over the KATRIN tritium source of more than 44 kg. In the present work, we give a brief description of the tritium handling (loop) and the operation with an emphasis on the permeator performance.

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II. THE KATRIN TRITIUM LOOP SYSTEM

The tritium loop system and its requirements are described in detail in Refs. [5] and [6], with a 24/7 core operational requirement at a throughput of up to 40 gd^{-1} and a tritium purity > 95% over the windowless gaseous tritium source (WGTS). A simplified flow diagram is shown in Fig. 1. Tritium is injected from buffer vessel (BV) 4 through a larger diameter transfer line and capillary into the injection chamber in the center of the WGTS. The capillary, injection chamber, and beam tube can be operated at ≈30 K, ≈80 K, or ≈100 K (stabilized to

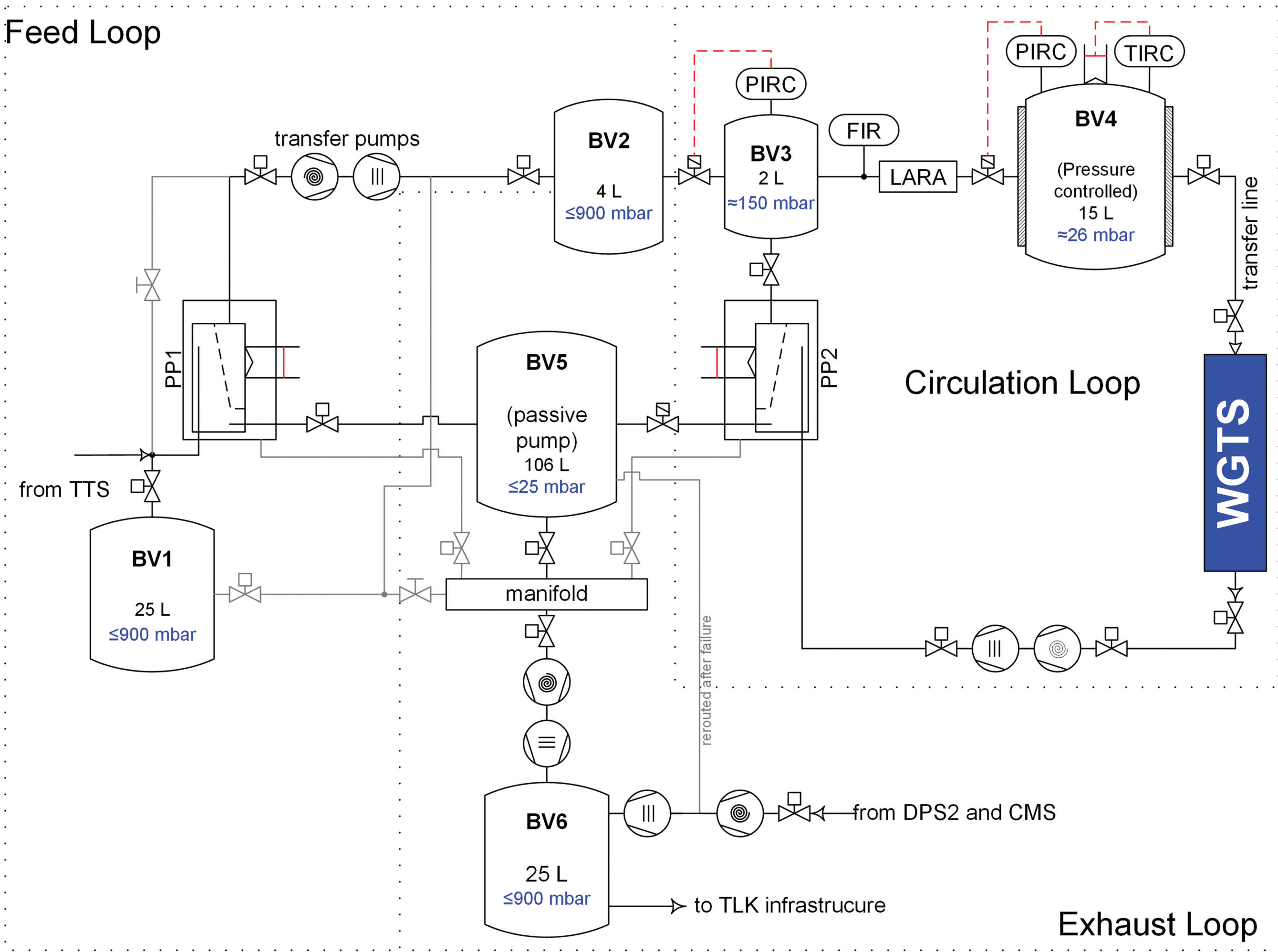

Fig. 1. Simplified flow scheme of the KATRIN tritium loop system. The pumping system at WGTS (not shown) consists of 18 TMPs. DPS2, CMS, and Cryogenic Pumping Section [7] are not shown. PIRC, TIRC, and FIR are the designators for pressure and temperature controllers and flow meters.

0.1%) [8]. The gas is pumped out by a cascaded pumping system consisting of 14 Leybold MAG W2800 turbomolecular pumps (TMPs) located at pump ports at both ends of the WGTS. Four groups of MAG W2800 are each pumped by a Pfeiffer HiPace300 pump. At the fore vacuum line a fraction of the gas can directly be reinjected via a second injection capillary [9]. The fore vacuum is provided by a combination of a Normetex and a metal bellows pump that pushes the gas through a palladium membrane filter ("permeator") [10] in the direction of BV3. From there the gas is fed through a laser Raman (LARA) sampling cell [11] and a regulation valve back into BV4. The permeator membrane can only be passed by hydrogen isotopes. All other species are removed from the circulating gas stream and eventually block it due to buildup at the feed side of the permeator. To prevent this blocking, a certain amount of gas ("bleed") is continuously extracted from the high-pressure side of the permeator and collected in BV5. A small amount of gas ($< 1.10^{-3}$ mbar Ls^{-1}) is streamed into the calibration and monitoring system (CMS) and differential pumping section (DPS) 2 [12] attached to rear and front side of the WGTS and is pumped via TMPs. Initially the exhaust gas was pumped via a Normetex and a metal bellows into BV6; after a pump failure the gas is pumped only via the Normetex in BV5. The amount of gas removed through CMS and DPS2 and as bleed at the permeator is continuously refilled from BV2. The gas stored in BV5 is transferred to BV6 each day to maintain the function of BV5 as a passive (fail-safe) pump. The accumulated gas in BV6 is transferred twice a week to the Tritium Laboratory Karlsruhe (TLK) infrastructure for purification. Processed tritium gas from TLK's tritium transfer system (TTS) is transferred weekly to BV1. Pure tritium is manually fed in batches from BV1 to BV2 [13,14]. The operation principle of this tritium loop is a demonstration of direct internal recycling [15].

III. START-UP AND OPERATION PROCEDURES OF THE LOOP SYSTEM

The overall loop system consists of 33 TMPs, four Normetex/metal bellows combinations, 223 sensors, and 231 valves. Tritium operation involves the whole TLK infrastructure: the interfaces to exhaust gas cleanup, isotope separation, tritium storage, and combined operation are described in Refs. [16,17]. The required main operation steps for starting tritium operation and maintaining it are briefly summarized in the following.

III.A. Gas Transfer from TLK Infrastructure and Feeding of Circulation Loop

Gas is transferred from TTS via the bypass line of permeator PP1 and the transfer pumps to BV1, followed by thoroughly evacuating the dead volumes of the transfer pumps afterward. BV2 is filled via PP1 with closed bleed. By this means, only purified gas is led into the circulation loop and nonhydrogen impurities are reduced to the lowest achievable level. The bleed is evacuated after the filling process has finished.

III.B. Start of Tritium Circulation

As a first step, the connection lines to the WGTS TMPs as well as the WGTS beam tube and pump ports are properly evacuated. Eighteen TMPs are installed at the WGTS, leading to a gas load of nonhydrogen species due to outgassing and permeation in the system of $\approx 2\ 10^{-5}$ mbar Ls^{-1}. Without proper evacuation, these impurities would be flushed to the permeator membrane and lead to poisoning of the membrane [10]. In a second step, BV3 and BV4 are filled to their required pressure levels by bypassing the flow controller between BV2 and BV3. The flow controller would restrict the flow from BV2 to BV3 to a maximum of 2 sccm. The required pressure set points are set in BV3 (usually 150 mbar) and BV4 (up to 30 mbar). They depend on the requirements on the throughput for an individual measurement). At permeator PP2 a fixed bleed rate of 1.4 sccm is chosen (independent on the circulation rate over the WGTS). As a final step, all required valves for the circulation are opened and the valves for evacuation of the system are closed.

III.C. Maintaining the Circulation

Once tritium circulation has started, five main operation steps (described in Sec. II) need to be performed routinely to maintain the circulation:

1. BV5 needs to be evacuated daily into BV6.

2. BV2 needs to be refilled from BV1 once a day.

3. A gas transfer from TTS to BV1 needs to be performed once a week.

4. BV6 is evacuated to the TLK infrastructure twice a week.

5. The vacuum jacket of permeator PP2 needs to be evacuated daily (see Sec. IV.C.3).

IV. OPERATION EXPERIENCE

IV.A. Overall Performance

KATRIN tritium operation started in May 2018 with traces of tritium [3], reaching full operation with tritium purities $>95\%$ in March 2019. During the first measurement phase with high tritium purity, radiochemical reactions of tritium with carbon deposits remaining from manufacturing and handling, even after proper cleaning and degreasing, caused a decrease in the system's overall performance. CO and tritiated methane were formed, mixed in the gas phase and frozen inside the 30 K cold part of the injection capillary (inner diameter 2.1 mm). This caused a severe reduction in conductance, and hence a reduced throughput. This blocking had to be removed by warming the capillary to 80 K prior to further operation of the loop system. After the first measurement phase, the generation of tritiated methane and CO was reduced to a negligible level that no longer influenced the performance of the system. The observed radiochemical reactions are discussed in detail in Ref. [13]. To date, a core team of six operators with on-call duty enabled more than 20 measurement campaigns, for more than 1350 days of tritium operation and an integral tritium throughput over the WGTS of more than 44 kg. The stabilization of pressure in the injection BV4 exceeds its requirement ($<0.1\%$) by more than an order of magnitude [18]. The tritium purity, monitored via in-line laser Raman spectroscopy [11], stayed above 97.5% the whole time, consistently surpassing the initial $>95\%$ requirement. Most of the time, purities $>98\%$ were achieved.

IV.B. Hardware Defects

Since the start of tritium operation in 2018 the loop system suffered several hardware failures. Three metal bellows MB601DC pumps failed for different reasons [14]:

1. Failure of the inner bellows in May 2018 (< 3000 h of operation).

2. Failure of the outer bellows in spring 2021 (≈10 000 h of operation).

3. Blocking of the drive shaft or motor bearing in spring 2021 after ≈14 000 h of operation.

The pumps with bellows failure were replaced, the blocked metal bellows pump was left in place, and its function was taken over by rerouting the exhaust of the Normetex in BV5. Despite the failures, the double-contained design of the pumps prevented tritium release to the glove box, so the integrity of the primary system was preserved, and the currently installed pump in the circulation loop has accumulated more than 21 000 h of operation. The Normetex pumps are running without failures, two of them with more than 36 000 h of operation. Five automatic valves developed an internal leakage, and damage in the actuator occurred in two manual valves. Three pressure sensors ceased proper function, either due to a problem in the wiring or sensor error. Due to redundancy, these valves and sensors did not need replacement. Six TMP controllers failed with electrical or firmware errors but could always be replaced within one working day. One of the MAG W2800 at the WGTS began repeatedly shutting down in early 2025. We suspect a problem in the cabling or electronics at the pump. Due to redundancy, continued operation without this pump was possible. Two electrical failures in the control cabinets were solved in one working day. In summer 2025, the laser Raman cell in the circulation loops suffered a massive leak. The leak was detected by our pressure interlocks, causing the system to enter a safe state. The cell was replaced with a new unit, and operation resumed. Together, the incidents and their repairs resulted in less than 30 days downtime in more than 1350 days of tritium operation within the scheduled measurement phases.

IV.C. Permeator Performance

This section focuses on the performance of permeator PP2 of the circulation loop. The permeator is a finger-type permeator with 46 PdAg tubes with a geometric surface of ≈590 cm^2. The tubes are installed inside the permeation cell (volume ≈0.4 L, outer stainless steel surface ≈400 cm^2, wall thickness 6 mm). The permeation cell as well as the PdAg fingers are operated at 400°C and surrounded by a vacuum jacket ($V = 13.6$ L).

IV.C.1. Membrane Poisoning

As mentioned in Sec. III.B, the 18 TMPs at the WGTS, as well as the beam tube, have a combined outgassing and permeation rate of ≈2 × 10^{-5} mbar Ls^{-1} from glove box atmosphere as well as carbon-containing outgassing products. Special care must be taken when handling this gas. In the measurement breaks and maintenance phases, the Normetex and metal bellows pumps are routinely switched off, and the gas accumulates at the exhaust side of the TMPs in the volume of the connection lines. Evacuation is then only needed approximately every 4 weeks. If, during pump-down, the gas comes in contact with the permeator it can lead to a poisoning of the membrane. This in turn causes a high differential pressure across the membrane at the same throughput or a lowered throughput at the same differential pressure. This particular behavior was observed in 2018 in the commissioning phase (see Fig. 2). During pump-down, the permeator membrane came in contact with high concentrations of impurities. At the start of the circulation a high pressure drop over the membrane of $\Delta p \approx 320$ mbar at 28 sccm was observed. To revert this poisoning, the membrane was activated. The activation procedure consisted of seven ≈5-min exposures to 4% O_2 mixed with N_2 at a pressure of 350 to 400 mbar at the operating temperature (400°C). The gas mixture was chosen in order to stay below the minimum oxygen concentration in nitrogen for hydrogen. The pressure level was chosen to not exceed the pressure interlock levels in the

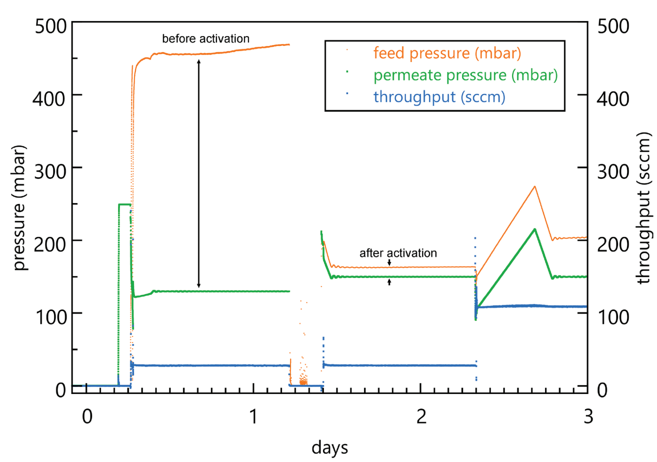

Fig. 2. Activation of the permeator membrane: the PdAg membrane was poisoned by impurities and needed an activation. Shown are the permeator throughput as well as the pressures on the feed and permeate side. Before activation the pressure drop over the membrane was $\Delta p \approx 320$ mbar at 28 sccm. After activation we achieved $\Delta p \approx 12$ mbar at 28 sccm. Afterwards the throughput was raised to 108 sccm, resulting in $\Delta p \approx 54$ mbar.

system. After activation, we achieved $\Delta p \approx 12$ mbar at 28 sccm, an improvement by more than a factor of 20.

IV.C.2. Long-Term Behavior

Fig. 3 shows the evolution of the differential pressure and corresponding flow over a period of 28 months. The pressure on the permeate side was kept constant at approximately 150 mbar. The continuous impurity gas load from WGTS and the TMPs caused a small progressively upward trend in the range of 1 to 2 mbar month^{-1} in feed pressure (maximum allowed 650 mbar, restricted by inventory and safety considerations) due to slow membrane poisoning. The pressure difference started out at ≈38 mbar at a flow rate of 71 sccm and increased to ≈67 mbar over time. In order to "reset" the membrane, an activation would be needed. From a practical perspective, the slight increase did not require activation, as the flow rate could still be maintained at reasonable operating pressures. Except for electrical safety and interlock tests, the permeator has been kept at the operating temperature since 2018 to avoid unnecessary thermal cycling.

IV.C.3. Behavior of the Vacuum Jacket

The permeator operation at 400°C caused a significant permeation of tritium from the permeation cell to the vacuum jacket. Fig. 4 shows the behavior during normal operation (short rise time, near linear) and standby (longer, exponential rise time) with no gas circulating. Once tritium operation has stopped, the pressure rise slows down significantly while depleting all hydrogen isotopes stored in the inner stainless steel wall. To keep the heat transfer to the outer wall low, the insulation vacuum is renewed approximately every 24 h during normal operation. Taking the technical data of the permeator and the recorded pressure rise of up to 0.4 mbar/day into account, a total gas transfer by permeation of $\approx 1.1 \times 10^{-7}$ mbar Ls^{-1} cm^{-2} was found.

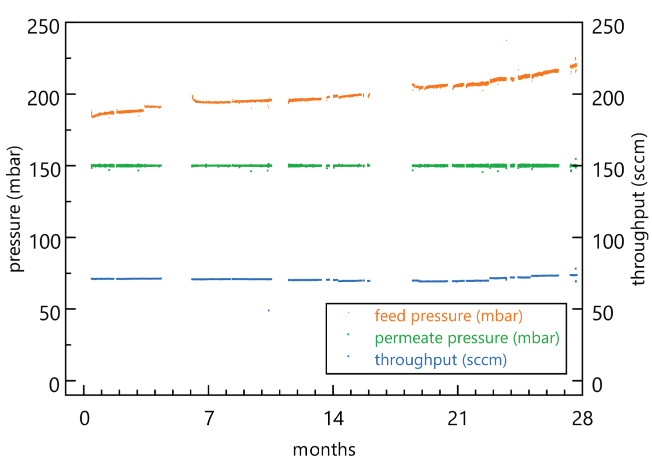

Fig. 3. Long-term evolution of the pressure drop over the PdAg membrane over a period of 28 months. No in-between activation needed to be performed. The slow increase in the pressure drop due to poisoning of the membrane is known, e.g., from Ref. [10] and cannot be avoided.

V. CONCLUSION

The KATRIN tritium loop system has now (December 2025) operated for more than 1350 days with a tritium throughput over the WGTS of more than 44 kg. Despite several hardware failures (metal bellows pumps, Raman cell), its overall duty cycle remained high, thanks to the redundant design, experienced operators, and thorough preparation allowing for rapid repair measures. Less than 30 days of downtime due to failures and repair in the loop system during the measurement phases enabled a high duty cycle (>98%) and efficient data taking for KATRIN. The initial requirements for injection pressure stability (<0.1%) and tritium purity (>95%) were surpassed, and a stability <0.01% and purity >98% were reached. The experience gathered over more than 7 years of operation provides a valuable basis for further optimization and future experiments that also need to handle large amounts of tritium safely. The loop system will be used in the coming years for KATRIN beamline operation for systematic measurements for neutrino mass analysis and then for the search for sterile neutrinos [19]. For this search, the injection

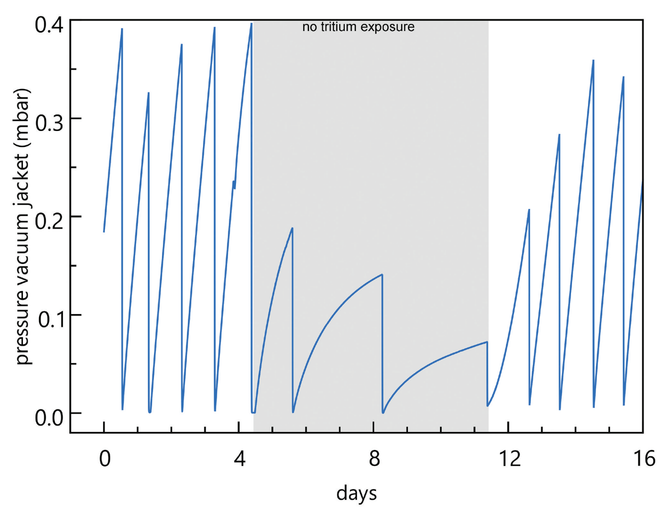

Fig. 4. Tritium permeation in the permeator vacuum jacket: during tritium operation of the permeator, tritium permeates from the permeation cell through its stainless steel cell (operating temperature 400°C) in the insulation vacuum.

rate needs to be lowered by a factor of 50 to 100. The capability of our regulation system to handle the injection rates below 0.1 sccm required to achieve this was already successfully demonstrated.

Author Contributions

CRediT: **Michael Sturm:** Data curation, Formal analysis, Investigation, Methodology, Visualization, Writing – original draft; **Florian Priester:** Data curation, Formal analysis, Investigation, Validation, Writing – review & editing; **Marco Röllig:** Investigation, Writing – review & editing; **Alexer Marsteller:** Investigation, Writing – review & editing; **Simon Niemes:** Investigation, Writing – review & editing; **Johanna Wydra:** Investigation, Writing – review & editing; **David Hillesheimer:** Investigation, Writing – review & editing; **Stefan Welte:** Resources, Validation, Writing – review & editing; **Lutz Bornschein:** Investigation, Writing – review & editing; **Beate Bornschein:** Conceptualization, Project administration, Resources, Supervision, Writing – review & editing.

Disclosure Statement

No potential conflict of interest was reported by the author(s).

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