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Review Article

Tritium Exposure and Decontamination: The TED Facility

Florian Priester, Alexander Marsteller, Michael Sturm, and Nancy Tuchscherer

Institute for Astroparticle Physics, Tritium Laboratory Karlsruhe, Karlsruhe Institute of Technology, Eggenstein-Leopoldshafen, Germany

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Abstract — *Ozone was used at the Tritium Laboratory Karlsruhe (TLK) for the first time for the decontamination of parts of the KATRIN experiment. The observed high decontamination efficiency led to the question of how to use ozone as an efficient cleaning tool for other facilities operated at TLK for years with tritium. To answer this question, we built the Tritium Exposure and Decontamination (TED) facility, where standard decontamination measures (purging, bake-out) can be quantitatively compared to the ozone method.*

To quantify the amount of ozone, and hence, the decontamination efficiency, we developed tritium-compatible in-line ozone sensors, which are presented alongside the TED facility and first results. A first series of tritium exposure at substantial tritium amounts (≈28 bar·h) and decontamination campaigns was performed using industrial-grade stainless steel, copper, and aluminum as sample materials, and applying ozone and conventional decontamination strategies.

The first results have indicated that ozone as a cleaning agent alone is not sufficient for tritium removal, and if possible, a bake-out is the most effective and least waste gas intense method. The ultimate goal of the TED facility is the development of a "plug-in" ozone generator suited for deployment in various facilities to achieve, e.g. the reduction of residual tritium in analytical systems or as a fast-acting cleaning tool prior to opening contaminated vacuum systems for modification and/or dismantling.

Keywords — *Ozone, tritium, decontamination, vacuum system, waste management.*

I. INTRODUCTION

Gaseous tritium is mainly handled and stored in vacuum systems made from stainless steel. Here, tritium has been shown to be incorporated in both the surface layer, and to a lesser degree, the bulk [1]. This contamination inside the vacuum system is usually of no concern, but can be challenging during maintenance or replacement when such systems need to be opened and residual tritium is released. Mostly, a second containment (glove box) keeps personnel safe when opening or dismantling a tritium plant, but sometimes work can only be facilitated by opening the second containment as well. An efficient decontamination prior to such work is mandatory.

Other than being a hazard to personnel, tritium can cause a background signal in various devices, such as ionization chambers or BIXS (beta-induced X-ray spectrometry) systems [2]. By reducing this so-called memory effect, regular decontamination can be beneficial for improving the limit of detection or measuring low activity concentrations after exposure to large amounts of tritium.

Another aspect to be considered is the handling of contaminated waste, such as a decommissioned vacuum system. Certain levels of activity per kilogram or cubic meter of noncombustible waste cannot be surpassed, requiring further treatment or long-term storage; hence reducing tritium contamination is necessary.

The standard decontamination strategy for most facilities at the Tritium Laboratory Karlsruhe (TLK), and probably others around the world, is purging with hydrogen (H_2) for

CONTACT Florian Priester florian.priester@kit.edu

isotopic exchange or (humid) air to enhance the release in the form of HTO. This procedure can be time consuming, while producing a large amount of slightly tritiated purge gas or water. If possible, a bake-out while continuously evacuating a system can help reduce surface contamination. However, baking, which is usually limited to a very few components due to the absence of heaters at all pipes, valves, etc., can cause tritium to migrate deeper into the bulk instead of being released.

At the KATRIN experiment [3], hosted at the TLK, we decontaminated a special surface (rear wall; termination of the magnetic flux tube) using ozone [4]. The rear wall is a gold-plated stainless steel surface that can be heated to approximately 160°C, and the surrounding vessel can be heated to 100°C. The combination of flushing, pumping, and heating alone did not make for a significant decrease in surface activity, but the use of ambient air in combination with a ultraviolet-C (UV-C) light source decreased the measured surface contamination by more than a factor of 1300 [4]. Other decontamination studies have been reported (e.g., Ref. [5]), but they used a combination of ultraviolet (UV)/ozone and did not mimic a real facility with arbitrary geometry.

The use of ozone would be useful since gas can be fed to virtually any facility usually operated with tritium. Therefore, this is a promising approach, and the usage of ozone for cleaning purposes is known in other industries [6–8] where ozone cleaning apparatuses are commercially available.

For use with tritium contaminated systems, the ozone production and detection had to be redesigned in order to be compatible with the special demands, e.g., no polymers exposed to tritium. This paper describes the production and detection of ozone and introduces the Tritium Exposure and Decontamination (TED) facility that was used for the comparison of common decontamination techniques and ozone cleaning.

II. TED FACILITY

The TED facility follows the TLK guidelines for all tritium processing systems. Most notably, these are the use of stainless steel pipes, connections welded where possible, a single flange-connection leak rate of $<10^{-9}$ mbar l/s, and generally, only the exposure of metals, glass, or ceramics to the process gas. By complying with these regulations, compatibility with the highly reactive ozone (O_3) gas is ensured.

Process gas–wetted parts at TED are made entirely from stainless steel. The DN40CF standard is used for vessels, valves are 1/4-in. Swagelok VCR compatible, and pipes have a 4-mm inner diameter with 1/4-in. VCR connectors. The ozone sensors are based on a 1/2-in. Swagelok VCR. All seals are metallic. A circulation pump with an all-metal scroll unit (AirSquared) is used. The pressure sensors are gas independent, and the capacitive gauges were supplied by Wika WU20 or Leybold Ceravac. The UV light source (RBD Instruments mini-Z) is directly connected to the vacuum system with no viewport in between to maximize ozone production (see Sec. II.A for details on the production and cleaning mechanisms). Glass and ceramics, in addition to stainless steel, are exposed to the process gas.

Fig. 1 shows a simplified flow scheme of the TED facility displaying the most important parts for performing the intended experiments. The seven identical DN40CF vessels V_0 to V_6 contain the samples (small pieces of tube or sheet material with a known surface) that were exposed to tritium. Two vessels (V_3 and V_6) are equipped with electrical heaters. Four vessels (V_0 to V_3) are directly connected to the ozone source ("close" branch), while three additional vessels (V_4-, V_5, and V_6) are connected with a variable-length pipe ("far" branch).

The ozone sensors (see Sec. III) are placed close to each set of vessels. The O_3 is produced in a common volume (DN40CF cross piece) using dry air fed by a flow controller from the glove-box atmosphere and an UV-C light source. During standard operation, ozone will not be recirculated, but will be sent directly to the TLKs central tritium retention system (ZTS) after passing the sample

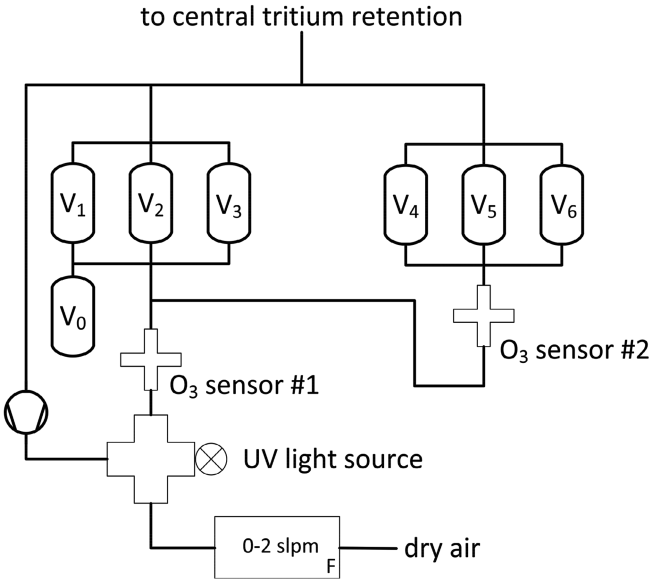

Fig. 1. Simplified TED flow scheme. For ozone sensor commissioning, the connection to the central tritium retention was replaced by a reference ozone meter.

volume. The flow rate is controlled by a flow controller from the Alicat Whisper series (0 to 2 slpm), which does not need to be tritium compatible.

Tritium is fed to the TED facility via double-walled pipes from the TLK infrastructure (connection not shown), the scroll pump is able to compress tritium up to ≈200 mbar in the sample vessels (V_0 to V_6). All the vessels containing samples receive an equal amount of tritium. After a certain time of tritium exposure, gaseous tritium will be sent back to the TLK infrastructure. Using the scroll pump, the system can be evacuated down to an ultimate pressure of $\approx 10^{-2}$ mbar.

After exposure to tritium, the decontamination procedures start. These can include (1) further evacuation for a longer time, (2) flushing with a suitable gas like hydrogen, (3) bake-out up to 200°C, and (4) ozone. Flushing/evacuation and bake-out can be combined.

The O_3 sensors are attached to each set of vessels, and the amount of O_3 reaching the samples is monitored. This is of particular interest since the production of O_3 is, to some degree, proportional to the flow rate of the inlet gas.

The aim is to optimize the ozone concentration while minimizing the inlet flow rate to avoid a high gas load on the TLK infrastructure. Additionally, it can be ensured that different decontamination runs will be comparable by ensuring a similar level of ozone is present. Fig. 2 shows the final setup of TED in a glove box at the TLK. In the front, left, and right corner, the "close" and "far" O_3 detectors can be seen, while in the back, six DN40CF sample volumes (V_1 to V_6) are mounted side by side.

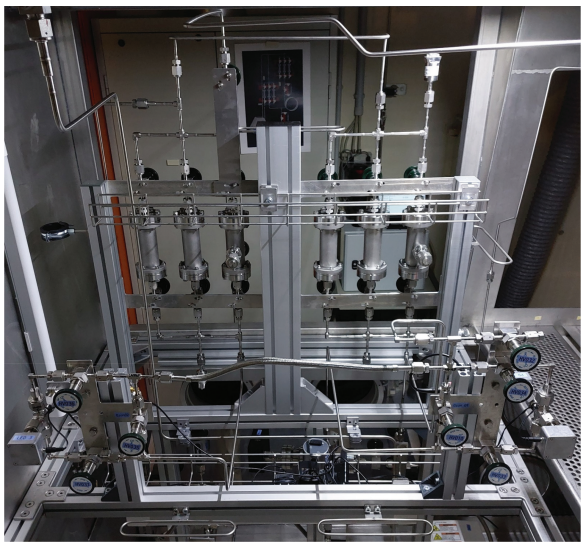

Fig. 2. TED vacuum system setup in glove box. Mixing vessels (six visible, backside) and ozone detectors (front, left, and right corner) can be identified.

For initial testing, a flexible metal hose was used as the connection between the close and far branches. This will be replaced by different lengths of pipe(s) in the future.

II.A. Ozone Cleaning Mechanism

Once ozone is produced by UV light from atmospheric oxygen, there are two vectors by which tritium can be removed from surfaces using ozone (this summary follows mainly that in Ref. [4]).

II.A.1. Desorption of Adsorbed Tritium

Tritium can adhere to metal surfaces via chemisorption (chemically bonded to the lattice) or it can be trapped in the oxide layer. The highly reactive atomic oxygen (atomic O due to ozone decomposing) and ozone (O_3) can attack these surface bonds. The oxidation reaction converts the surface-bound tritium into tritiated water vapor (HTO or less likely T_2O). The water can desorb from the surface and be pumped away.

II.A.2. Oxidation of Tritiated Hydrocarbons

Tritium can also bond to different molecules already sticking to the surface, like a thin layer of carbon (hydrocarbons/oils) that naturally forms on all technical surfaces during handling. The UV/ozone attacks the carbon backbone of these contaminants,

$$C_xH_yT_z + O_3(\text{or O}) \rightarrow CO_2 + H_2O + \text{HTO} \ .$$

This effectively removes the hydrocarbon layer, releasing tritium as tritiated and water vapor, as well as carbon dioxide, which can be pumped away.

In both scenarios, there is the possibility of oxidizing the underlaying metallic surface. For the most commonly used structural material, stainless steel, this is not a disadvantage, but strengthens the already present passive layer protecting the bulk (Cr_2O_3). The same is true for aluminum, which naturally forms a Al_2O_3 protective layer. For copper, there is the possibility of discoloration when it comes in contact with ozone.

III. TRITIUM-COMPATIBLE IN-LINE OZONE DETECTORS

To assess the cleaning effect of ozone, it is necessary to know how much O_3 is reaching the sample chamber. For a comparison of different measurements, it is also crucial to know the O_3 concentration during each run.

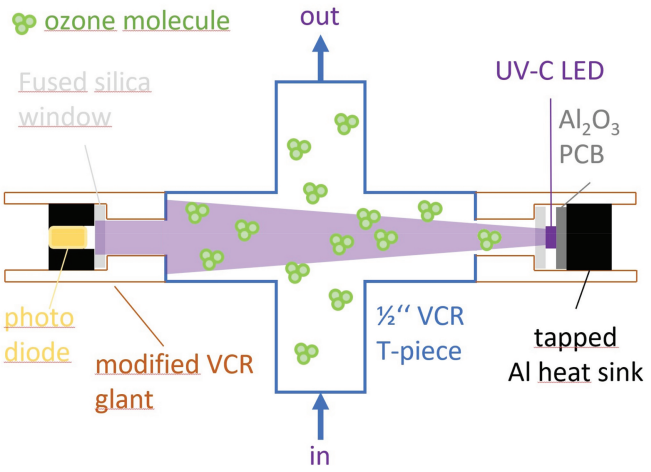

Fig. 3. In-line ozone monitor scheme.

With no commercial tritium and only a high vacuum-compatible solution readily available, we decided to develop our own O_3 detection unit, exposing only fused silica and metal to the process gas. The measurement (conceptual design shown in Fig. 3) of O_3 was based on the absorption of light around 254 nm by the ozone molecules. As a narrow-band light source, a UV-C LED (laser components PKB-35-F35) with a peak emission wave length of 255 nm was used. As a detector, we used a photodiode (laser components JAEA0,1S). The LED was surface mounted to an Al_2O_3 circuit board that was glued with thermal conductive epoxy resin to an aluminum heat sink. The photodiode was centered and glued to a second aluminum heat sink, both of which provide a Thorlabs SM005–compatible thread.

The voltage readout of the photodiode is facilitated with a transimpedance amplifier (Thorlabs AMP100) directly attached to a BNC connector (left side of Fig. 4), while the LED is powered by a 20-mA constant current source directly fitted in the housing (right side in Fig. 4). Digitalization was realized with 24-bit precision to be able to detect small changes in the output of the amplifier.

As viewports, we modified a 1/2-in. VCR gland so that it can accommodate a 1/2-in. fused silica window and provided a Thorlabs SM005–compatible thread for easy installation of the optical components. Both parts are sputter coated with a layered system that allows for soldering. A circle with a 7-mm diameter remains uncoated on the window for light transmission.

The window and gland are soldered together using soldering paste for reflow soldering at ≈275°C. Perfecting the procedure, we were able to consistently achieve He leak rates $<10^{10}$ mbar l/s. This design, which proved more useful and much easier to handle under glove-box conditions compared to traditional DN16CF-based windows, can be used for various other optical applications.

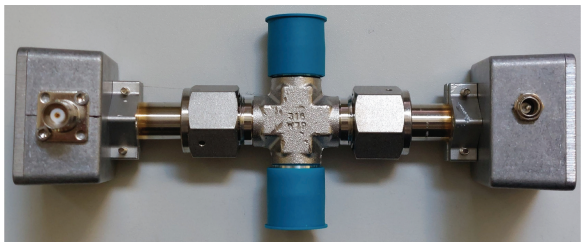

Fig. 4. Ozone in-line detector ready for use. On the left side is the photodiode with the BNC connector for read-out, and on the right side is the housing for the UV-C LED and the constant-current source.

The long absorption path of ≈75 mm results in a high sensitivity. Tests have shown that the detectors can effortlessly measure changes in the ozone concentration of 0.1 ppm.

IV. FIRST OZONE MEASUREMENTS

Prior to the operation with tritium, we needed to ensure that the performance of the in-line ozone detectors could match the demands of the TED facility; namely, a reliable operation over several hours and consistent readings in the final setup.

IV.A. Calibration Procedure

The output voltage of the photodiode is amplified and digitized as described in Sec. III. To convert that voltage reading to a useful ozone concentration, the detectors needed to be calibrated against another ozone meter with a known (better) precision, but that did not need to be tritium compatible.

For that, we used a desktop ozone detector (2B Technologies Model 106MH, 0 to 10 000 ppm and 0.01 ppm resolution). This reference system was attached at the position indicated in Fig. 1 with "to central tritium retention" prior to tritium operation. Attaching the two (self-built) in-line detectors in series to the reference ozone meter, we can get an ozone-concentration versus photodiode-amplifier output plot, directly allowing for fitting a calibration curve.

It was assumed that there was a neglectable amount of ozone vanishing due to wall interactions, which is reasonable considering the length (≈20 cm) and time of the gas between the inlet and outlet (<5 s). The fit to the data is individual for each in-line detector, accounting for a different light intensity of the LED, alignment, and detector (photodiode and amplifier characteristic) response.

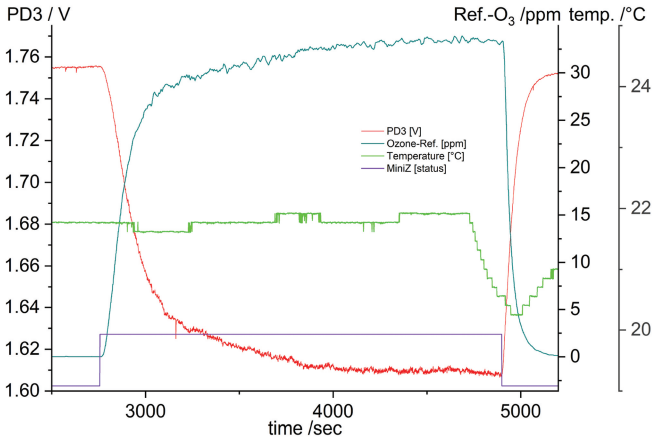

Fig. 5. Raw data for the calibration of one in-line ozone sensor (PD3) against the reference ozone detector (Ref.-O_3). The temperature was recorded to exclude any temperature-dependent effects.

Fig. 5 shows the data from a calibration for one in-line detector. Shortly (≈10 s) after switching on the UV light source, mini-Z (purple line), the signal of the reference ozone meter (ppm ozone) started to rise, while the signal of the photodiode was decreasing because the UV light intensity dropped due to absorption by O_3. The gas flow for this measurement was maintained by the internal pump of the reference ozone detector with a flow rate of ≈1 l/s. The relatively long time of ≈1500 s before a stable maximum ozone value was reached originated mainly from the warm-up time of the mini-Z UV light source.

By applying an exponential decay fit to the reading of the photo diode (PD3 in Fig. 5) against the measured ozone content in ppm, the conversion of the voltage readout to ppm ozone is facilitated. The derived, empirical fit function is

$$c_{O_3,\mathrm{ppm}} = ln\left(\frac{U-y}{A}\right)t + x + O\ .$$

The parameters for sensor 1 were as follows:

1. U = raw read voltage of the sensor/amplifier.
2. $y = 6.3514$.
3. $A = -1.37551$.
4. $t = 1072.5114$.
5. $x = -1293.4109$.
6. O = offset (to be determined in situ after the warm up of the sensor).

These were verified afterward with different gas flow rates, and hence, different ozone concentrations.

IV.B. First Measurements in the Final Setup

The reference ozone meter was installed instead of the later-to-make connection to the central tritium retention system (Fig. 1), providing gas flow through the complete TED setup and a reference value for the ozone concentration. The valves were set in a fashion so that all the systems (Fig. 1: Ozone #1, Ozone #2, and reference ozone meter) were flushed. Only the valves to volumes V_3 and V_6 were opened, the flow controller was set to its maximum throughput (2 slpm), and the mini-Z UV light source was switched on. By pumping ambient air through the whole system, ozone reached all the detectors, and due to the flow rate of ≈1 l/min, a short connection between the close and the far branches and no samples were exposed in V_3 and V_6. Thus, a nearly identical ozone concentration was expected.

Fig. 6 displays the results of the very first ozone production and measurements in the TED facility. The same characteristic course of ozone increase can be seen here as with the earlier calibration measurements, but with a much more compact setup. In addition, Fig. 7 shows the time delay between switching on the mini-Z UV light source for O_3 production and the arrival of the O_3 at all of the three detectors. The farther away from the O_3 source the detector was located, the longer the it took the O_3 to reach it.

Since the ozone concentration in the TED setup was reaching similar values as with the more compact, initial setup for testing, the ozone lifetime was considered to be of no relevance for the experiment. Other ongoing investigations at TLK with ozone have pointed toward the same conclusion, that the lifetime of ozone at near-atmospheric pressures (order of 30 min) is several times larger than the time needed to pump the gas containing ozone from the point where it is produced to where the sampling volume of the ozone is applied to the samples.

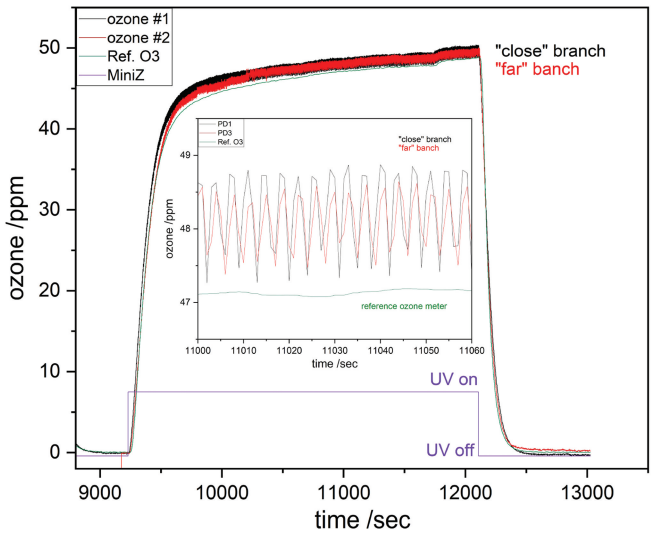

Fig. 6. First ozone measurement in the finalized TED setup. The pulsed pumping of the reference ozone meter (3 s pumping and 3 s measuring) is resolved in the data of ozone detectors 1 and 2 (inset).

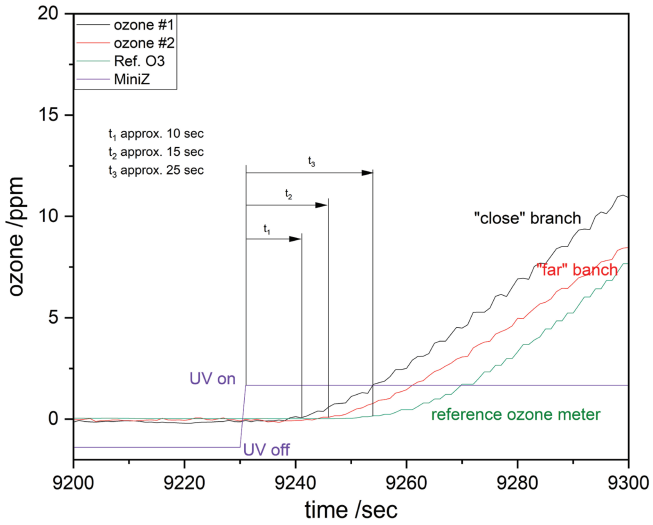

Fig. 7. Time delay between the start of ozone generation and the arrival at all the ozone detectors. The ripple, most prominent in the signal of sensor 1, originated from the pumping interval (3 s on and 3 s off) of the reference O_3 meter.

With the central tritium retention system to be connected later, the gas flow through the TED facility will be adjusted with the installed flow controller and limited to <0.01 slpm (10 sccm) for regular operation. Instead of using dry air for ozone production, the flow controller can also be used for the regulated inlet of other gases (e.g., H_2, He, N_2) for flushing the samples.

V. TRITIUM EXPOSURE AND DECONTAMINATION

After verification of the proper functionality of the tritium-compatible O_3 sensors, a first set of samples was prepared, exposed to tritium, and analyzed for surface contamination. This section introduces the procedures and briefly summarizes the findings.

V.A. Experimental Procedures

To start with, we limited the setup to the close branch with four sampling cylinders (V_0 to V_3), identical tritium pressures of ≈168 mbar, an exposure time for all the samples of ≈1 week, and identical decontamination procedures with no variation in the temperature or gas species as follows:

1. 11-h evacuation at <0.01 mbar.

2. Flushing with ozone produced from dry box atmosphere for 8 h with a flow rate of 2 sccm.

3. Flushing with dry box atmosphere for 8 h with a flow rate of 2 sccm.

4. Bake-out for 8 h under vacuum (<0.01 mbar) at approximately 140°C (measured inside the sample volume).

The discharged gas was directly sent to the central ZTS with no further analysis. The ZTS provided a mild vacuum of ≈800 mbar, enough to maintain a constant flow through the setup. For the measurement of the surface activity of each sample, we relied on wipe testing (device Perkin Elmer TriCarb 4810TR with Insta-Gel Plus LSC cocktail). Since the wiped activity can be sensitive to the force applied during wiping, a special tool was designed to apply a constant pressure on each sample surface leading to a comparable wiping efficiency. This tool dictated the sample geometry to be a flat and thin (0.2 mm) sheet with a total surface per side of 10 cm^2.

Each sample, therefore, provided two wipe test results with three samples stored at the same time in one exposure vessel (V_0 to V_3) yielding a total of six measurements per decontamination method and material. All the samples were cleaned in an ultrasonic bath with water and wiped with acetone followed by ethanol prior to installation in the sample volume and subsequent tritium exposure. To securely separate the three samples in each volume from each other and ensure proper exposure of both sides to

Fig. 8. Samples in vessel with separation for exposure in place.

tritium gas (and O_3 later on), all the samples were separated by stainless steel wool (Fig. 8).

To provide representative results, the assessment of the surface contamination by the wipe test started immediately after stopping the decontamination process. This way, the loss of surface activity by outgassing was avoided to the extent possible.

V.B. Experimental Results and Discussion

A total of four campaigns were performed as the base for this paper. For each campaign, all sampling vessels were used and filled with the specified material. Only one cleaning method was applied to each sampling vessel. The following materials (see 6.1 for geometry) were exposed to pure tritium (>95%) as described in Section V.A.:

1. Campaigns 1 and 2 stainless steel (1.4307).

2. Campaigns 3 and 4 pure copper (CW004A) and aluminum (Al99.5).

Table 1 summarizes the results of the wipe tests, where six wipe test results per sample material were combined to an averaged value and the 1σ standard deviation is given (in italic). The data provided some useful insights:

1. Even if the number of samples is low, the uneven decontamination effect seems to be obvious due to the large standard deviation. This can be related to parameters like wipe test efficiency, despite the mentioned tool used, differences in surface cleanliness, reachability/mobility of the surface-bound tritium, or others.

2. Flushing with dry air seems to be the least effective method as it produces a contaminated waste gas stream that needs to be treated afterward; therefore, it is not recommended.

3. Heating the samples under vacuum yielded the best results, even if the temperature was rather low at 140°C. As a benefit, this method resulted in no additional waste gas that had to be treated.

TABLE 1

Results of Wipe Tests*

Campaign Number	Material	Ozone	Heating	Evacuation	Dry Air
1	1.4307	553	319	402	645
	Standard deviation	*293*	*162*	*166*	*244*
2	1.4307	353	402	322	868
	Standard deviation	*125*	*197*	*229*	*125*
3	CW004A	574	309	761	1142
	Standard deviation	*357*	*128*	*230*	*128*
3	Al99.5	729	318	644	1109
	Standard deviation	*257*	*107*	*130*	*332*
4	CW004A	753	546	407	527
	Standard deviation	184	131	69	197
4	Al99.5	830	377	657	631
	Standard deviation	178	113	233	289

*"Ozone," "Heating," "Evacuation," and "Dry Air" refer to the decontamination method, with values given in kBq/cm^2. All the samples had a total area of 20 cm^2 and were exposed for the same time and under similar tritium pressure/purity conditions with an exposure equivalent of ≈28 bar·h.

4. Ozone showed virtually no added benefit, and the results were comparable to dry air alone.

5. The aluminum and copper samples showed a higher activity compared to stainless steel, but heating Al or Cu seemed to be able to release more surface-bound tritium than evacuation alone (see rows 3 and 4 in Table 1).

There were cases where the surface activity increased with the use of ozone (compared to dry air). This might be because of the mobilization of tritium from other parts of the vacuum system and deposition onto the sample, since this effect was only observed during the last campaign (see rows 5 and 6 in Table 1) when the facility had been in operation with tritium for several weeks. Prior to the additional measurements, this effect needs to be understood well and eliminated for any additional measurements.

VI. SUMMARY AND OUTLOOK

A new facility for the quantitative comparison of decontamination strategies was designed and set up at the TLK, based on the good experience of using ozone and UV light during the KATRIN experiment. The TED facility provides possibilities for exposing samples like metal sheets or short pieces of pipe with a well-known surface to a tritium atmosphere of up to ≈200 mbar. Utilizing decontamination strategies like evacuation, flushing with various gases, and bake-out up to 200°C, the TED facility offers in situ ozone treatment, in addition to the tritium process systems unique to the TLK.

To monitor ozone levels during the decontamination runs, a tritium-compatible monitor was developed and tested prior to tritium operation. This device, which was based on the absorption of 254 nm of UV light by ozone molecules, provides high sensitivity and good stability/repeatability.

A first series of exposures and decontamination campaigns was performed, exposing flat sheet material with a 0.2-mm thickness made from stainless steel, copper, and aluminum to a tritium atmosphere with an exposure equivalent to ≈28 bar·h. Different decontamination strategies, including the use of ozone, were applied.

It was apparent that the high decontamination factors observed during the KATRIN experiment could not be achieved. This might be linked to the absence of UV light in the TED facility in the sample chamber, which entertains the theory that only a combination of both can lead to significant decontamination achievements. Another possible thought is that the process only works for gold surfaces, as the coated and monitored contaminated surface in the KATRIN experiment provided only gold to tritium results. Further investigations are required for incorporating gold-coated samples and the addition of UV light to at least one sampling volume.

Summing up the results we have accumulated so far, a mild bake-out at ≈140°C yielded the best decontamination. This statement held true for all the tested materials.

After addressing all open questions arising from the first measurement campaigns, we plan to remeasure all previously tested materials and add tungsten to the ensemble of specimens because of its relevance as a fusion first-wall material.

The ultimate goal, after thorough testing and finding optimal parameters for ozone generation and duration of exposure, is a "plug-in" device for connection to other facilities for decontamination prior to opening tritium-contaminated vacuum systems for easier modification and/or dismantling.

Author Contributions

CRediT: **Florian Priester:** Conceptualization, Formal analysis, Investigation, Visualization, Writing – original draft; **Alexander Marsteller:** Investigation, Visualization, Writing – review & editing; **Michael Sturm:** Investigation, Visualization, Writing – review & editing; **Nancy Tuchscherer:** Conceptualization, Resources.

Disclosure Statement

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

ORCID

Florian Priester http://orcid.org/0000-0002-6840-9940

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