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Abstract

Future fusion reactors must be able to breed the tritium they will consume. Several breeding and tritium extraction technologies are under investigation internationally. PbLi is of particular interest as a breeder material. A new forced convection PbLi loop, the Tritium Extraction eXperiment (TEX), is being designed and constructed to investigate tritium extraction from PbLi. Specifically, TEX will serve to investigate the extraction efficiency of various vacuum permeator membranes and configurations. The major components of the loop include a moving magnet pump, reverse permeator, furnace test section, analysis chamber, supply tank, and plenum. As significant hazards are present in such experiments, safety is an integral focus of the experimental design.

Keywords: PbLi, tritium, breeding blanket, permeation, membranes

1. Introduction

Tritium breeding is fundamentally required for a sustainable fusion fuel cycle, yet the technological readiness of blanket technology lags far behind other fusion systems [1]. Breeder concepts are divided into solid and liquid media. Solid breeders typically rely on a sweep gas, such as hydrogen doped helium, to carry away tritium from lithium containing ceramic materials. Liquid breeders produce tritium from lithium containing eutectics (e.g., PbLi) or molten salts (FLiBe). In each case, tritium must be harvested from the breeding medium. For liquid breeders, several methods for tritium extraction have been proposed [2]. One promising method for tritium extraction from PbLi is through a vacuum permeator [3], in which a concentration gradient from the tritium-containing PbLi promotes diffusion through a membrane with high hydrogen permeability to the vacuum.

Hydrogen permeation through materials has been studied for decades and is well characterized for many materials [4]. While permeable membranes are at the heart of tritium extraction, relatively little research has been conducted on using vacuum permeators to extract tritium from PbLi. The two facilities most relevant to the present research are the TRITEX and CLIPPER loops due to their methods for hydrogen injection and extraction. TRITEX was a forced convection loop constructed with the original intention to study tritium extraction from PbLi [5]. Permeators were used to introduce deuterium into the PbLi, and V getters were used for extraction. Over the course of its operation (late 1980's to 1996), the loop objectives shifted away from tritium extraction to material properties relating to corrosion and purification [6]. CLIPPER (Ciemat Lithium-Lead loop for Permeation exPERiments) is designed to test hydrogen and deuterium extraction under conditions applicable to the DEMO dual coolant lead lithium (DCLL) blanket concept [7-9]. PbLi has been studied in other facilities in order to investigate corrosion, magnetohydrodynamics, and other material properties (see Ref. [10] and references therein). Additional experimental facilities have investigated additional methods for tritium extraction, such as the packed columns, vacuum droplet towers, gas-liquid contactors, and getters [2].

As US, international, and commercial fusion efforts move forward, the need for significant advancements in tritium breeding and extraction are crucial. Herein, we present a design for a PbLi loop that will be used to investigate the tritium extraction efficiency of vacuum permeators. The primary goals for this new loop are to develop a versatile facility capable of testing multiple configurations and permeator materials under near-prototypic conditions. The design is presented in Section 2, followed by discussion on how the system will be commissioned and operated in Section 3, and some notes on safety in Section 4.

2. TEX loop design

A forced convection loop, the Tritium Extraction eXperiment, has been designed to test tritium extraction from PbLi. The loop is designed to be a versatile testbed to validate permeator modeling, test a wide range of permeator concepts, and examine the effects of permeator length, PbLi flow rate, temperature, tritium concentration, and transport properties on tritium permeation rates. The loop is designed to operate at isothermal temperatures up to 535°C, and an overview of basic operational parameters are summarized in Table 1. An overview schematic of the loop is shown in Figure 1. The major systems of the loop include the PbLi pump, a reverse permeator, the test section, the analysis chamber, a supply tank, and a plenum. Each of these systems will be described in detail in the following sections.

Table 1. Calculated operational parameters for TEX.

Parameter	Value
Volumetric flow rate	0.5 L/s
Mass flow rate	4.7 kg/s
Permeator length	1-5 m
PbLi velocity in test section	≤ 4.2 m/s
PbLi velocity in tubing	≤ 1.3 m/s
Temperature	300-535°C
D concentration (mol/m ³)	$\leq 2 \times 10^{-2}$
Tritium capable	Yes

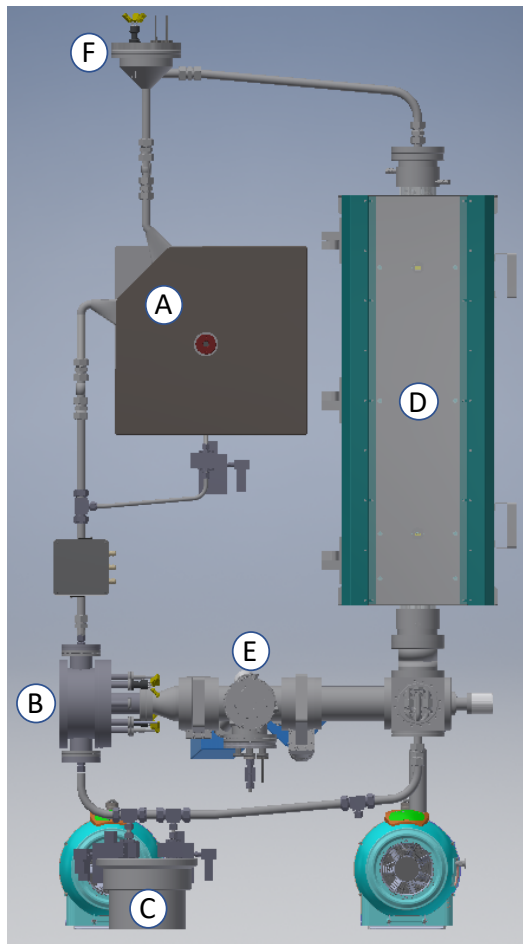


Figure 1. The Tritium Extraction experiment (TEX). As labelled in the figure: (A) EM pump, (B) reverse permeator, (C) supply tank, (D) furnace/test section, (E) analysis chamber, and (F) plenum.

2.1. Electromagnetic Pump

Molten metals can utilize a variety of pump types, and frequently electromagnetic (EM) pumps are preferred [11]. EM pumps can include annular linear induction pump (ALIP), direct current

induction pump, and moving magnet pump. The moving magnet pump type [12] has been chosen to be used in TEX (Figure 1A) as it meets the loop flow and temperature requirements, provides an economical choice, and the pumping channel can be replaced if necessary. The pump can be properly sized by considering the pressure drop through the loop components. One unique aspect of TEX is the modular and scalable nature of the test section, which, discussed later in Section 2.3, allows the tube length within the test section to range from 1-5 m in length within a reasonably compact configuration. Figure 2 shows the results of these head loss calculations for the loop with flowing PbLi with test sections in a serial configuration. Temperature-dependent density and dynamic viscosities are taken from the review article by Martelli, et al. [13]. To achieve flow rates of up to 0.5 L/s (~ 4.7 kg/s mass flow rate), using the tubing specified in Table 2 and flow loss factors from Ref [14], a pump that supplies 5 bar pressure is required.

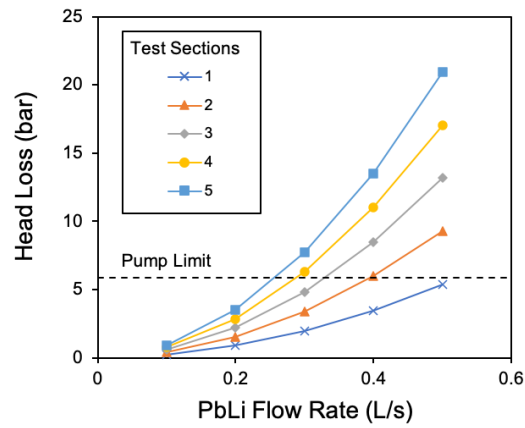


Figure 2. Head loss calculations are used to determine the pump pressure required to achieve desired flow rates. The effective loop length of TEX increases as the number of test sections increase in a serial configuration.

Table 2. Specifications and flow properties of the TEX loop and test section.

Tube parameter	Loop	Test section
Outer diameter (OD)	25.4 mm (1.0 in)	12.7 mm (0.5 in)
Inner diameter (ID)	22.1 mm (0.87 in)	12.3 mm (0.484 in)
Wall thickness	1.65 mm (0.065 in)	0.2 mm (0.0079 in)
Velocity (max.)	1.3 m/s	4.2 m/s
Tube roughness	1.5×10^{-5} m	1.5×10^{-5} m
Reynolds number	1.66×10^5	2.98×10^5
Friction factor	0.020	0.022

2.2. Reverse permeator

To test tritium extraction from PbLi without producing it through nuclear reactions requires artificially introducing tritium into the PbLi. Several potential methods for introducing tritium into PbLi are discussed. First, tritium gas could be bubbled into the molten metal. In practice, this is the most straight forward method, however, accurate measurement of the tritium partitioning between gaseous and liquid phase is challenging. The bubbling technique would most likely result in two-phase separation of the tritium from the PbLi, and the tritium would nucleate and

accumulate in the high points of the loop. Therefore, bubbling may not produce representative concentrations of volumetric tritium production in a breeding blanket.

Another potential option for introducing tritium into the PbLi is to do so chemically. PbLi is produced by melting Pb with Li, and it may be possible to replace the Li with LiT. However, as these processes involve high temperatures, it is uncertain whether the initial tritium concentration would be maintained through the melting and dissolution process.

The third option that has been considered for introducing tritium into the loop is through a “reverse permeator.” This concept, which has been explored by others [6,9], involves the use of a high-tritium-concentration reservoir in contact with a permeable membrane to allow tritium to permeate into the loop. This method is viewed as being able to introduce tritium more uniformly into the PbLi as tritium is introduced in the atomic form eliminating the dissociation mechanistic step.

A schematic of the conceptual reverse permeator is shown in Figure 3 (see Figure 1B for context within TEX) where the permeator membrane tube is shown oriented vertically at the center of the chamber. The membrane in this case is approximately 152 mm in length and 10 mm outer diameter and could be a Group V metal or α -iron. The permeator tube passes through a tungsten filament, which will be used to heat the permeator. Because the entire chamber volume will be filled with tritium seeded gas, a water-cooled shroud will surround the filament. This is necessary in order to reduce radiative heating of the reverse permeator chamber and mitigate unwanted tritium permeation through the structural components. The pressure of the reverse permeator will be precisely monitored using baratron pressure gauges as well as an electronic controlled back pressure regulator. The baratron will measure the decrease in static pressure due to permeation into the PbLi. These sensors will be fundamental in determining how much tritium enters the PbLi. Figure 4 shows the temperature and pressure conditions of the reverse permeator necessary to achieve a desired deuterium concentration in the PbLi, using the analytical solutions from [15]. As shown, using α -iron in the reverse permeator will be able to provide deuterium concentrations relevant to helium cooled, water cooled, and dual cooled lead lithium (HCLL, WCLL, and DCLL) blanket concepts [9].

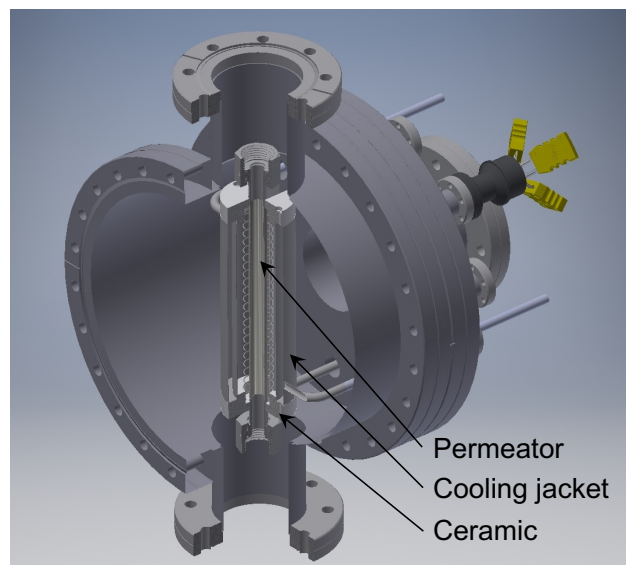


Figure 3. Conceptual design of the reverse permeator. As shown, PbLi will flow downward through the permeator.

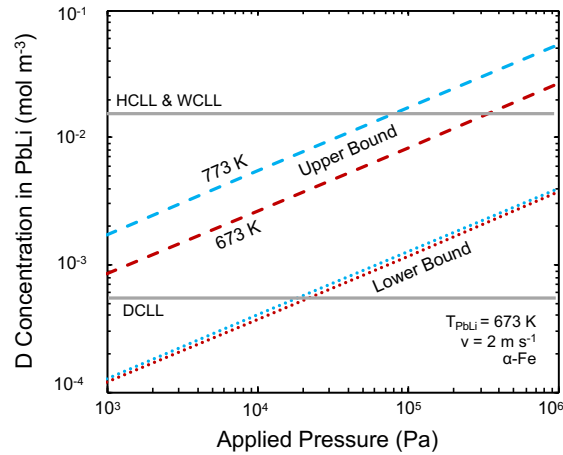


Figure 4. Temperature and hydrogen isotope pressure of the reverse permeator required to achieve a given concentration in the PbLi.

2.3. Test section

The test section contains the primary vacuum permeator membranes. The test section is contained within a tube furnace (Figure 1D) that will have a 3-zone heated region sufficient to test 1m long test sections. The loop tubing will penetrate the hermetic test section at both the entrance (bottom) and exit (top) of the tube furnace. The test section tube furnace will be under ultra-high vacuum to ensure that the tritium concentration gradient will favor permeation from the PbLi. The bore size of the tube furnace will allow multiple configurations of permeator sections as illustrated in Figure 5. The effective length of the permeator membrane may be increased by adding serial passes through the test section. This design allows scaling a 1 cm diameter tubular membrane from a length of 1 m to a total length of 5 m. In addition, the test section can be configured to allow multiple parallel membrane legs. Additional configurations may be considered, which could include alternative membrane diameters, geometries, or other novel arrangements. This initial configuration envisions PbLi flow inside the tubes in order to investigate the coupled behavior of mass transport, potential surface effects, and permeation in the simplest configuration possible, which should also simply identification of the rate-limiting step in the resultant permeation.

Ideally, the vacuum tube will be constructed of quartz that transitions to metal conflat flanges on both ends in order to connect to the rest of the vacuum system. Quartz is the preferred material for the tube furnace as it is more transparent to the radiative heat, and more significantly, it is significantly less permeable to the tritium that will be released from the permeators. If a quartz vacuum boundary is not feasible, an oxidized thick-walled steel tube could be acceptable. As the entire steel tube would become a radiator, one challenge could be the copper gaskets that are used in connecting to the adjacent ultra-high vacuum system. In this case, the ends of the steel vacuum boundary would need to be water cooled to reduce the conductive heat transfer to avoid melting of the copper gaskets. This temperature gradient may possibly perturb the heat profile of the permeator membranes.

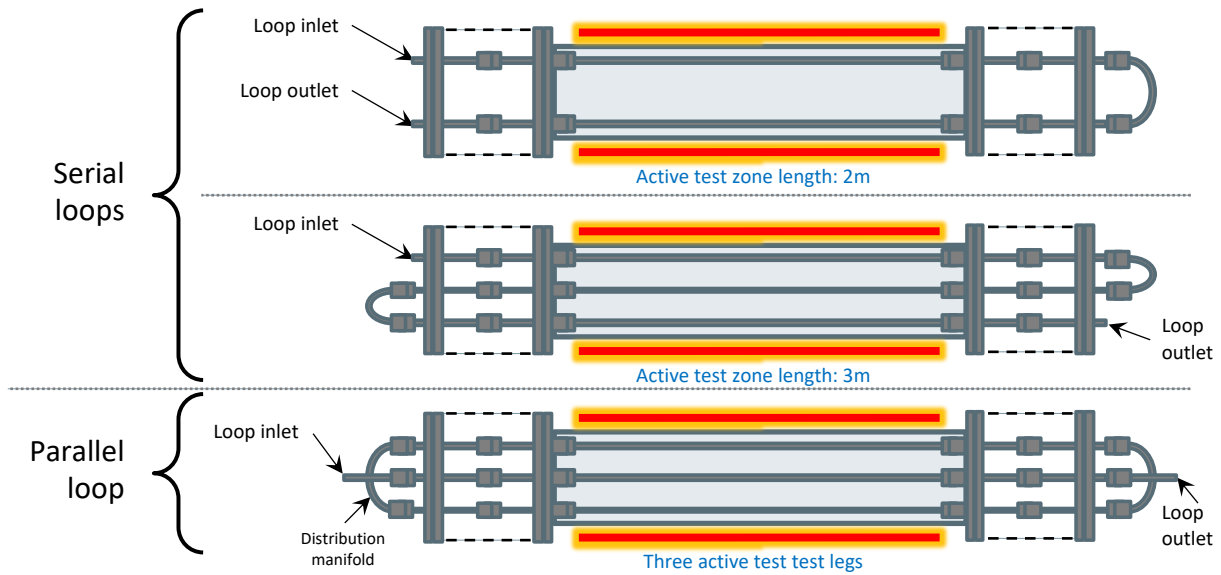


Figure 5. The versatile test section will allow serial and scaling of test section lengths. Expanding serial legs will increase the effective test section from 1 m up to 5 m in length. Increasing the number of parallel loop legs can offer reduced flow rates without altering the loop velocity.

2.4. Analysis chamber

The analysis chamber (Figure 1E) is an ultra-high vacuum chamber that is connected to the test section. Hydrogen isotopes in the PbLi will permeate through the membrane into the vacuum chamber. The analysis chamber will be pumped by a 250 L/s turbo molecular pump and backed by an oil free scroll pump. Two quadrupole mass spectrometers (QMS) will detect the amount of tritium that has permeated into the vacuum system. Hydrogen isotopes diffuse through metals in atomic form and recombine into molecular form at the surface. Tritium may then recombine as T_2 or HT molecules. Initial commissioning and benchmarking phases of the loop will investigate deuterium extraction from helium. Since helium and molecular deuterium have the same atomic mass number, it is prudent to use two separate QMS detectors: one in a standard 1-100 amu range for detecting typical vacuum impurities (water, oxygen, nitrogen, etc.) as well as a high resolution QMS sensitive enough to resolve D_2 and He.

The analysis chamber will also be connected to the primary loop tubing. This is necessary during the initial fill phase of the loop. The loop will be evacuated for several hours or days to remove impurities prior to the initial fill with PbLi. Heating the loop during this pump-out phase will help remove adsorbed impurities. Experimentally measured hydrogen solubility constants for PbLi span several orders of magnitude [16]. Removing as many impurities as possible from the loop prior to introducing the PbLi will help mitigate the potential influence of unknown impurities on the behavior of tritium in the system.

Because the vacuum system will have a direct connected to loop tubing, it will be necessary to include an inline freeze filter. This filter will have a very large cold surface area that will freeze any PbLi that may inadvertently pass from the loop into the vacuum system.

2.5. Supply tank

PbLi will be loaded into a supply tank located at the bottom of the loop, as shown in Figure 1C. After melting, the loop will be loaded into the pre-heated loop by opening the primary valve

and then pressurizing the supply tank with inert gas. The pressurized gas will push the molten PbLi into the loop. This process will be facilitated by evacuating the loop prior to filling. PbLi will be elevated from the supply tank approximately 3 m to the top of the loop, which corresponds to approximately 3 bar inert gas pressure to fill the loop. The size of the supply tank is dictated by the volume required to melt ~3.5 L of fresh PbLi ingots. Such volumes, gas pressures, and local regulator codes require the supply tank to certify as a pressure vessel.

The supply tank will also serve as the dump tank when it is necessary to drain the loop. PbLi can be highly corrosive, and the high temperature ball valves may become unreliable once wetted with PbLi. Therefore, a secondary valve will be reserved exclusively as an emergency drain valve.

2.6. Plenum

A free surface will be located at the highest point of the loop in the plenum, as shown in Figure 1F. Gases that precipitate from the PbLi, or hydrogen isotopes in excess of the solubility limit will naturally accumulate in the plenum. Plenum gases can be exhausted by a pressure relief valve or periodical pumping. In addition, the composition of the gases can be monitored by gas chromatography. Additional diagnostics could be housed in the plenum. These may include electrochemical sensors, oxygen or hydrogen sensors, and pressure gauges.

The plenum additionally serves as a buffer volume for thermal expansion of the PbLi, which has a volumetric thermal expansion coefficient between $1.2\text{-}1.3 \times 10^{-4} \text{ 1/K}$ between the range of $235\text{-}650^\circ\text{C}$ [17]. Depending on the diameter of the plenum, this expansion will be observed and monitored by using a series of sheathed thermocouples positioned at different heights. Thermocouples in direct contact with the PbLi will read a higher temperature than thermocouples in contact with gas only.

3. System operation

Systematic and phased campaigns will ensure a successful operational and research mission for TEX. The first phase will be a deuterium-helium once-through campaign to commission TEX systems and components. Phase two will utilize PbLi and deuterium, and phase three will introduce tritium.

Currently, no reliable diagnostics are available for measuring hydrogen isotopes in PbLi. Commissioning the equipment for deuterium extraction from helium is integral in establishing the measurement techniques and analytical confidence required for operating with PbLi. Measuring the extraction efficiency of the vacuum permeator requires precise knowledge of the hydrogen (generic for all hydrogen isotopes) concentration in the loop as well as the hydrogen that permeated through the test section membrane. The reverse permeator will utilize a control volume and precision pressure gauges in order to quantify the amount of hydrogen (C_0) that permeated from the reverse permeator into the helium loop. C_0 will be verified in the helium gas via gas chromatography. The hydrogen concentration downstream the test section (C_1) will also be sampled, revealing the quantity of hydrogen that permeated through the membrane into the vacuum, D_{extract} . Calibrated quadrupole mass spectrometers (QMS) will verify the value of D_{extract} . Efficiency of the extraction experiment is determined as

$$\eta = \frac{C_0 - C_1}{C_0} = \frac{D_{\text{extract}}}{C_0}.$$

Verification of C_0 and C_I through gas chromatography will not be possible for the PbLi loop. Therefore, determining the extraction efficiency from PbLi will rely entirely on the reverse permeator conditions and the QMS measurements.

PbLi will re-circulate within the loop and deuterium extraction from the vacuum permeator will be $< 100\%$. Therefore, PbLi at the inlet of the reverse permeator may contain a non-trivial hydrogen concentration. One advantage of the reverse permeator over alternative hydrogen injection methods is that permeation is driven by a concentration gradient. As a result, if the PbLi is already saturated with hydrogen, hydrogen will cease to be injected at the given reverse permeator temperature and pressure.

Operating with tritium in the final phase will introduce challenges, namely contamination and exhaust from the vacuum pumps will need to be captured on getter beds. However, enhanced opportunities become available. Tritium diagnostics enable exceptional sensitivity, which will result in improved quantification of the tritium extraction efficiency.

4. Safety

The TEX loop exhibits a number of hazards that must be mitigated and protected against. First, the loop will be actively heated to temperatures as high as 535°C . PbLi brings significant chemical and toxicity concerns. The introduction of tritium requires special measures to avoid radiological contamination. Accordingly, TEX will be mounted inside of a safety enclosure. The safety enclosure will be an engineered structure capable of bearing the weight loads of the furnace, pump, and all other required equipment.

An example of such a ventilated enclosure is shown in Figure 6. The enclosure protects personnel from inadvertent contact with hot surfaces by providing physical isolation. The enclosure will be continuously exhausted to provide a negative pressure for drawing room air through the enclosure. In the event of a leak or spill, this ensures that potentially volatile hazardous materials do not escape into the room air. Tritium containment strategies also frequently rely on continuous ventilation. The negative enclosure pressure will help ensure that the tritium that permeates into the enclosure will be swept through the exhaust. Getter beds and HEPA filters will be used in the exhaust stream to prevent the release of tritium and hazardous particulates, respectively.

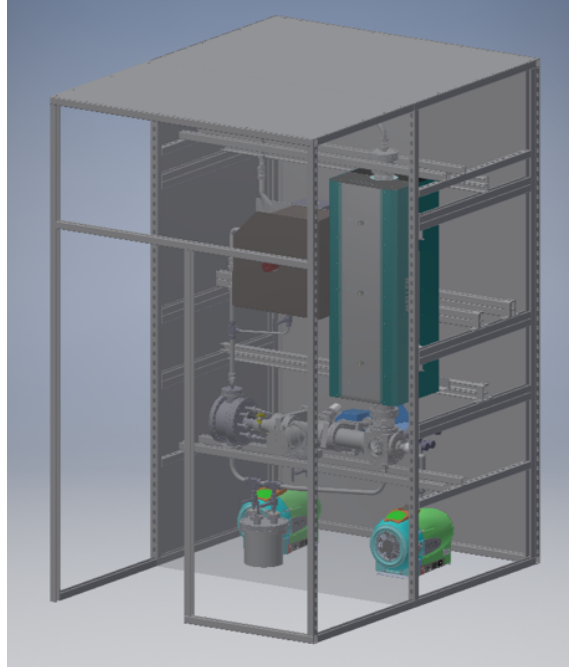


Figure 6. TEX safety enclosure.

5. Summary

A new facility, the Tritium Extraction eXperiment (TEX) is being designed and constructed to investigate the extraction efficiency of vacuum permeators. The unique features of TEX include the ability to investigate multiple test sections that can expand from 1-5 m in length. This versatility will aid in validating permeation models [15]. The loop is designed to operate under isothermal conditions from 300-535°C. The major components include a moving magnet pump, reverse permeator, vacuum furnace test section, analysis section, supply tank, and a plenum. Commissioning of the system will be performed by testing deuterium extraction from helium. This methodology will provide the confidence necessary for testing tritium extraction from PbLi.

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