



Horizon 2020
Programme

TRANSAT

Research and Innovation Action (RIA)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 754586.

Start date : 2017-09-01 Duration : 48 Months



Interim report concerning the state of the art. Completion of design of experimental study

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TRANSAT - Contract Number: 754586

Project officer: Project Officer: Angelgiorgio IORIZZO

Document title	Interim report concerning the state of the art. Completion of design of experimental study
Author(s)	Mr. Robert VALE, Mr. Tomas Rey, Mr. James Bromley
Number of pages	14
Document type	Deliverable
Work Package	WP04
Document number	D4.3
Issued by	UKAEA
Date of completion	2019-09-27 16:56:49
Dissemination level	Public

Summary

A description of the physical laws and processes connected with the permeation of hydrogen through steel together with the state of the art of current values for the permeability of tritium through disposal facility relevant materials is given. The design of an experiment to evaluate permeability/surface adsorption is proposed and developed. The experiment is proposed to investigate these materials at low temperatures of 50-200 oC for a gas consisting of tritiated water vapour.

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Abbreviations

CCFE	Culham Centre for Fusion Energy
UKAEA	United Kingdom Atomic Energy Authority
HLW	High Level Waste
ILW	Intermediate Level Waste
SF	Spent Fuel
EDX	Energy-Dispersive X-Ray Spectroscopy

Summary

A description of the physical laws and processes connected with the permeation of hydrogen through steel together with the state of the art of current values for the permeability of tritium through disposal facility relevant materials is given. The design of an experiment to evaluate permeability/surface adsorption is proposed and developed. The experiment is proposed to investigate these materials at low temperatures of 50-200 °C for a gas consisting of tritiated water vapour.



1 Introduction

While some options exist for the treatment of hard (including metallic) wastes to reduce the tritium inventory, options for recycling this material are limited and on an industrial scale are not expected to be available for many decades. Therefore, ultimately it is likely that such waste will require disposal in near surface or deep geological disposal repositories. Being a gas, the characteristic properties of tritium can make it a challenging isotope to manage in a repository setting ensuring that adequate barriers exist to prevent permeation which can sometimes reach a maximum after a number of decades.

Current European concepts for high-level waste and spent fuel disposal include containers made from: stainless steel, iron, copper, carbon steel and titanium (Bennett & Gens, 2008). Current major sources of tritiated solid waste include a large volume of graphite irradiated waste worldwide due to its wide use as a moderator and reflector for fission nuclear reactors. A likely option for final treatment of this waste is to dispose of it in a geological disposal facility (Girke, Steinmetz, Bosbach, Bushuev, & Zubarev, 2011). Experimental fusion power plants such as ITER are also predicted to produce metallic tritiated waste.

Most of the permeation experiments that have been conducted on metals have involved exposing a membrane to tritium gas. Little work has been done on the transport of tritium water vapour HTO through the materials being considered for HLW and spent fuel disposal. Experience from measuring drums of tritiated waste at UKAEA show that the gas usually contains a greater percentage of HTO than HT. This is corroborated by studies which show that for metal surfaces containing oxygen tritium transfers as HTO to the surrounding environment (Hayashi, Nakamura, & Isobe, 2012). Therefore, the basis of a decision regarding waste container material type solely on HT permeation rates could be inappropriate for metallic wastes with oxide layers.

2 Fick's Laws of Diffusion

Hydrogen transport through a material involves adsorption, dissociation, solution, diffusion, recombination and desorption. When the process reaches a steady state, it is limited by either surface adsorption or permeation through the bulk of the material. The rate of hydrogen permeation through steel is given by Fick's first law. In the case of the surface area of the hydrogen barrier being much greater than its thickness this can be expressed in one dimension. This dimension is perpendicular to the permeation surface.

$$J = -D \frac{dc}{dx} \quad (1)$$

Where D is the diffusion coefficient, dc/dx is the concentration gradient along the x axis (perpendicular to the permeation surface), and J is the diffusional flux. When steady state diffusion is achieved

$$J_{\infty} = -\frac{D(C_2 - C_1)}{l} \quad (2)$$

Where C_1 and C_2 are the hydrogen concentrations at the inlet and outlet surface respectively and l is the specimen thickness. Figure 1 shows the process that Fick's law describes.

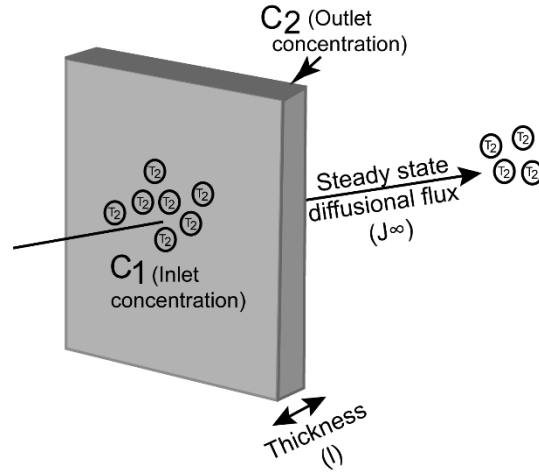


Figure 1: Steady state diffusional flux through a material.

Sievert's law gives the relationship between hydrogen concentration and pressure P

$$C = SP^{\frac{1}{2}} \quad (3)$$

Where S is the tritium solubility and C is the concentration. This establishes the important relation between solubility and pressure. Finally, the steady state equation can be re-written as

$$J_{\infty} = -\frac{DS\left(P_1^{\frac{1}{2}} - P_2^{\frac{1}{2}}\right)}{l} \quad (4)$$

Where P_1 , and P_2 are the equilibrium hydrogen pressure at inlet and outlet surfaces. Generally speaking P_2 is negligible.

$$P_1 \gg P_2$$

So DS can be the permeability

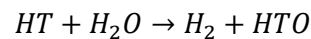
$$\Phi = DS \quad (5)$$

And finally

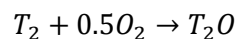
$$\Phi = \frac{J_{\infty} l}{P_1^{\frac{1}{2}}} \quad (6)$$

2.1 HT to HTO exchange reaction

Tritium gas can be transformed into tritiated water via the exchange reaction



The equilibrium constant of which is approximately 6.25 at 298K and 3.4 at 398K (Lasser, 1989). Or by oxidation



An example of an oxidation process is that caused by the oxide layer on stainless steel, this process has been recorded experimentally by (Longhurst, 2007) who derived the following formula for the conversion

$$\frac{C}{C_0} = \exp \left[-1.987S \exp \left(-\frac{62,840}{RT} \right) t \right]$$

Where C (m^{-3}) is the non-condensable or elemental tritium concentration, $C_0(m^{-3})$ is its initial value, S (m^{-1}) is the surface to volume ratio for the system, R is the universal gas constant (J/mole-K), T is



the temperature (K), and time is measured in minutes. However, the reverse is also true that HTO can react with a metal to form an oxide layer producing HT. A study by (Hayashi, Nakamura, & Isobe, 2012) showed that with pure D₂O the species that permeated through various metals was D₂ which was created during the formation of the corresponding oxide layer. This is supported by the results of (Yamasaki, et al., 2012) which demonstrated an increase in oxygen from at% 16.22 to at% 40.31 for EDX analysis of F82H martensitic steel exposed to 6480ppm water vapor at 700°C. In this case the permeation rates for hydrogen remained the same as that found by pure hydrogen studies which is supported by the increase in the oxide layer. If an inert barrier was applied to the metal such as Au the permeation dropped significantly as there was no route for D₂ production. (Hayashi, Nakamura, & Isobe, 2012) The same can therefore be assumed for tritium.

This oxide formation and the oxidation of HT should form an equilibrium at the metal if the oxide formed is stable. The formation of a stable oxide on the metal surface would reduce the permeation through the metal and as such the stability and permeation reduction effect of the metal oxide that would be produced is a key material property when considering container materials. The permeation reduction can also be achieved by adding a coating to the container material such as carbides oxides and nitrides (Nemanic, 2019).

Given these exchange reactions the starting T species can play an important role in container material choice. If HTO is the primary/exclusive T species present, then the key aim of the container material should be to prevent the formation of HT or T₂ which if successful would drastically limit permeation through the container material. If the primary species is HT/T₂ then the presence of a permeation barrier material is important to limit the permeation with the bulk materials capabilities to either form stable and effective barriers itself or its suitability for additional permeation barriers to be applied are key material properties.

2.2 Rate Limiting Factor

The steady state rate of the gas passing through the sample can be limited by either permeation through the material, or by surface effects such as adsorption and desorption. Adsorption occurs through a process of either physisorption or chemisorption. Surface layer processes contribute to an important distinction to be made regarding the transport of hydrogen (or water vapour) through materials. This concerns whether the rate limiting process is atomic hydrogen diffusing through the specimen (permeability), or whether it is surface process such as chemisorption, dissociation and recombination of hydrogen. Added to the surface processes are the HTO to HT exchange reactions described in the previous section. In particular, the definition of surface reactions can be broadened to include the reduction of HTO through the creation of an oxide layer and subsequent permeation of HT.

A typical way of evaluating what process is dominating the measured gas flux is whether the value measured is dependant on the square root of pressure or whether it follows a linear dependency (Forcey, Ross, Simpson, & Evans, 1988). An alternative to this is to vary the thickness of the membrane to see if it has an impact on measured gas flux.

For instance, for the case of (Yamasaki, et al., 2012) at temperatures of greater than 500 °C the permeability constant for F82H steel agrees well with that of (Serra, Perujo, & Benamati, 1997) which used 0.5-0.8 mm discs. This is even though water vapor is used in the first study and there is an increase in the oxide layer of the steel which shows it is having an effect. In this instance, the results can be interpreted to be indicating that oxidation of the HDO on the surface is happening at a quicker rate than permeation through the bulk. Therefore, it is the permeation rate that is recorded for the applied pressures and the disc thickness. In the case of (Hayashi, Nakamura, & Isobe, 2012) it was found that for heavy water applied to 1mm thick SS304 at 300°C there was a decrease in the expected deuterium permeation rate of approximately 1/100. So, it is likely that the surface oxidation rate is controlling the reaction under these conditions. At the temperatures expected of repository



conditions which can be well below 300°C this effect could also control flux for HTO in the materials suggested for containers.

3 Material Composition in Current Nuclear Waste Container Designs

With regards to the materials used for current nuclear waste containers, there are roughly 5 material groups currently used around the world (King, 2013):

1. Carbon steel, low-alloy steel, cast iron
2. Stainless steel
3. Copper, Cu-based alloys
4. Titanium, Ti-based alloys
5. Ni-based alloys

The cause of this wide variation of materials, including within the groups themselves, has to do with the advantages and disadvantages that each material type possesses. For example, while carbon steels allow for a cheaper and simpler container design, they suffer from higher corrosion rates. Conversely, while titanium has excellent corrosion resistance, it requires additional internal supports, in the form of steel or iron inserts, which greatly impacts the design.

This variation in materials choice is also present internationally, with each of the five group types being used as reference materials in at least one country, as seen in Table 1 below.

Reference and Other Candidate Container Materials in Various National Nuclear Waste Programs

	Waste Type	Reference Container Material(s)	Alternative Container Materials Considered	Ref.
U.K.	ILW	Type 304L/316L	BS4360 C-steel	16
U.K.	HLW (SF)	—	C-steel, copper, Ti alloys, Ni-Cr-Fe alloys	19
Germany	HLW/SF	TS1E 355 C-steel, Alloy C-4, Ti 99.8-Pd, pure copper	Alloy 625, Alloy 825, Cu10Ni, Cu30Ni, cast steel, cast iron	40
Spain	HLW/SF	TS1E 355 C steel	Type 316L, Alloy C-22, oxygen-free copper	40
Belgium	HLW (SF)	C-steel	Type 316L hMo, 309, 316Ti, 904L, 430; Alloy C-4, Alloy 625, Ti, Ti-0.2Pd	14,35,40
France	HLW/SF	C-steel	Undefined passive alloy	41
Switzerland	HLW/SF	C-steel	Copper	62
Sweden	SF	Copper, cast iron structural support	Ti alloy	43,54,56
Finland	SF	Copper, cast iron structural support	—	44
Japan	HLW	C-steel	Ti alloys, copper	45
Canada	SF	Copper, C-steel	Ti alloys	63-66
USA	SF/HLW	Alloy 22, Ti-7 drip shield	C-steel, various Ni-Cr-Mo alloys	20,23,24,61, 67-70

Table 1: Waste Container Materials used by each country (King, 2013)

And so, it is important to be able to sample representative materials from each group for tritium permeability, such as 304L steel, pure copper, low-carbon steel, and a Ni-Cr-Mo alloy.

The overall aim being to be able to put forth suggestions for a future novel waste container for tritiated waste. For example, once all material types are tested, it may be possible to draw conclusions on which materials are best suited as a tritium barrier. It may be the case that a solution would require two or more material layers to provide an adequate solution.

Looking at the Swedish waste containers for fission waste (figure 2), they make use of an outer copper canister and an internal cast-iron insert (SKB AB, 2010).



Figure 2: Copper and cast-iron waste container design used in Sweden (SKB AB, 2010)

The copper layer is meant to protect against corrosion, while the cast-iron insert is favoured due to its yield strength and fracture toughness. With this solution, the waste container is able to withstand high stresses and corrosive environments, all while providing effective shielding to prevent the release of radionuclides from spent fuel rods.

Tritiated waste poses its own issues, given the potential for significant tritium permeation, meaning that the popular solutions used for spent fuel rods may not be applicable to waste primarily contaminated with tritium. Because of this, studies considering these four existing material types specifically within the context of tritium (and its different forms) are necessary.

4 Temperature Conditions Expected in Repository Environments

The conditions a nuclear waste container will have to endure will have a large impact on the choice of material for the container. For example, if we consider a container filled with high-level waste (HLW), such as spent fuel rods, and the container is buried in a deep geological repository, it will be subject to two separate sources of heat. The first will come from the increased ambient temperature due to the depth of the repository. On average the temperature increases by 25-30 °C per km for dry rock formations (Fridleifsson, et al., 2008). For example, at depths of 4-5 km, associated with deep borehole disposal the ambient temperature alone can reach more than 100°C depending on the particular geothermal gradient (Gibb, Travis, McTaggart, & Burley, 2008). The 1km depths associated with most planned repositories can be expected to experience ambient conditions of 40 °C if the average surface temperature is 15 °C.

The second source comes from the heat generated from the nuclear waste itself. The temperature increase associated with this is highly variable, depending on factors including the intermediate storage time before final disposal, the amount of waste in the container and the distribution of containers per area. Because of this, estimating the temperature inside the waste container is very challenging (Mattsson, 1981). Nevertheless, there have been several studies conducted to model the expected temperature profile for different conditions. Figure 3 shows the thermal simulation of disposed strontium capsules over time (Payer, Finsterle, Apps, & Muller, 2019).

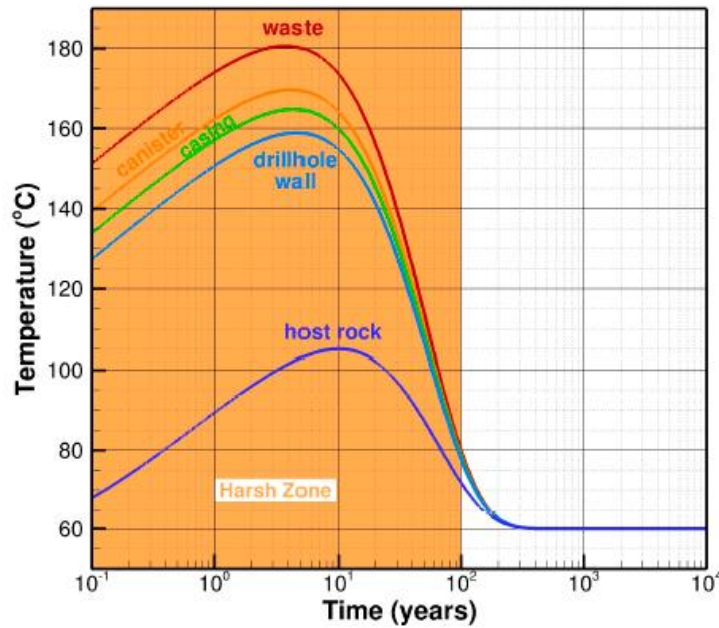


Figure 3: Temperature profile for HLW waste container over time

For this simulation, the initial heat output of the HLW was 164 W, the containers were spaced 0.6 m apart, and the ambient temperature of the container’s environment was set at 60°C. As the figure shows, the temperature due to the HLW steadily rises to a maximum of 180°C for the waste itself, and reaching roughly 170°C. For this material, the primary heating effects last around 100 years.

Given the possibility to condition HLW, by intermediate storage and other means, to reduce the heat output, the maximum temperature that a container is required to withstand can actually be set by governments. For example, Sweden specifies a maximum initial canister temperature of 70-80°C, the United Kingdom expects temperatures of up to 150°C, and for proposed salt dome repositories, testing is carried out at temperatures up to 300°C (Mattsson, 1981).

Indeed, more recent temperature simulations done in several countries give a similar spread of maximum temperatures found in the close vicinity of the HLW containers (Figure 4).

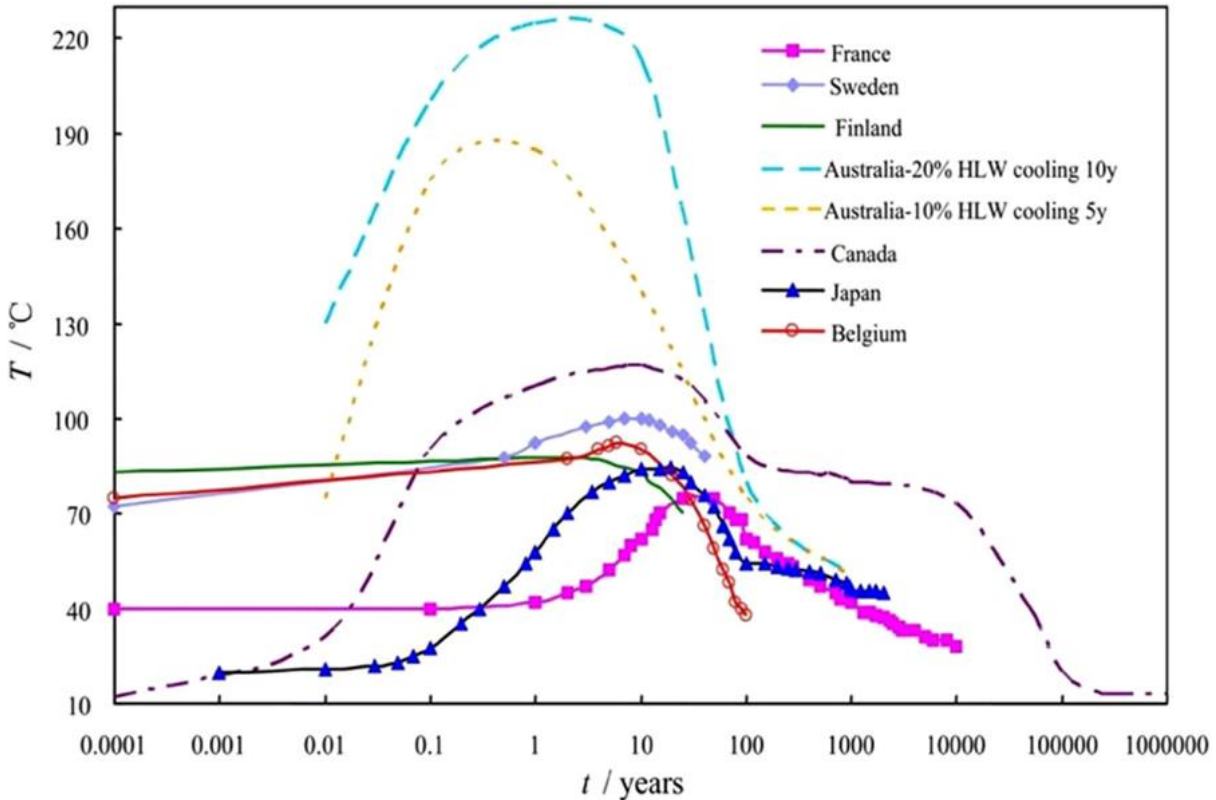


Figure 4. Temperature simulation near HLW containers for various countries (Zhang, et al., 2019)

Despite differing base values for ambient temperature and widely varying maximum temperatures, likely due to the heat output and amount of the simulated waste, the curves for each country show that any viable container material needs to be able to maintain its structural, anti-corrosive, and shielding properties at temperatures far above 100°C, possibly even at the upper limit of 300°C.

On the basis of this information it would be appropriate to test the container materials for tritium permeability at a temperature range of 50-200 °C.

5 Tritium Permeation Values for Different Metals

5.1 HT

As has already been stated the majority of permeation values in the literature have been derived for HT. The values for hydrogen permeability for a number of materials were compiled from the available literature by (San Marchi & Somerday, Technical Reference for Hydrogen Compatibility of Materials, 2012). Permeation figures from this report are given in table 2.

Material	H ₂ Permeability (293K) (mol H ² m ⁻¹ s ⁻¹ Mpa ^{-1/2})	Temperature Range of Derived Permeability K ⁻¹	Yield Strength approx. (MPa)	Cost (\$/ton)
Hastelloy-C22	7x10 ⁻¹⁵	434-1000	400	20,000
Austenitic Stainless Steel	2-3x10 ⁻¹⁵	423-700	650	2,000
Pure Cu-99.9% ¹	3x10 ⁻¹⁶	377-1250	70	7,000
Carbon Steel 1020	2x10 ⁻¹¹	500-900	350	600



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Table 2: Permeabilities of selected materials at 293K from (San Marchi & Somerday, Technical Reference for Hydrogen Compatibility of Materials, 2012). Permeability extrapolated for lower temperatures than experimental range.

6 Experimental Design

6.1 Container Material Selection

A review of the material types given in table 1 suggests that the following materials would make good candidates for an assessment of tritium permeability.

Material	National Programmes Suggested For Use
316L	UK (ILW), alternatives for Spain and Belgium.
Cu	Germany, Sweden, Finland, Canada, alternatives for UK (HLW), Switzerland, Japan.
C Steel (TStE 355 C-Steel)	Germany, Spain, Belgium, France, Switzerland, Japan, Canada, alternatives for UK (ILW and HLW), USA.
Hastelloy	Alternative for USA and Spain

Table 3: Suggested materials for experimental tests

The TStE 355 C-Steel is the steel selected for waste disposal in the German concept which conforms to the EU standard S355NL. Alternatives to this are the American ASTM A516 Grade 55 steel which has been suggested for the Yucca Mountain repository as an outer containment barrier (Larose & Rapp, 1997). ASTM A516 is primarily used for service in welded pressure vessels. Compositionally S355NL and ASTM A516 are very similar as table 4 shows. Therefore ASTM A516 should give useful information on both concepts.

Elemental Composition (wt%)														
Steel Grade	C	Mn	Si	P	S	Fe	Cr	Mo	Ni	N	V	Cu	Al	Ti
S355NL	<0.18	0.90-1.65	<0.50	<0.025	<0.020	balance	<0.3	<0.1	<0.5	<0.015	<0.12	<0.55	<0.02	<0.05
ASTM A516 Grade 55	<0.26	0.55-1.30	0.13-0.45	<0.035	<0.035	balance								
SS316L	<0.03	<2	<0.75	<0.045	<0.03	balance	16-18	2-3	10-14	<0.10				
	C	Mn	Si	Cu	S	Fe	Cr	Mo	Co	W	V	Ni		
Hastelloy	<0.01	<0.5	<0.08	<0.5	<0.08	3	22	13	<2.5	3	<0.35	balance		

Table 4: Compositions of selected material types for permeation study

The thickness of the metallic membranes should be representative of the container materials in order to assess whether the surface rate or permeation is the rate determiner. For the purpose of the experiment membranes with a 1mm thickness will be tested at the higher end of the range (150-200°C). For the timescales available thinner membranes would have to be tested at the lower range of the temperature.

6.2 Tritium Permeation Apparatus

The design of the tritium permeation apparatus is shown in Fig.1. This apparatus is predominantly made from 1/4" stainless steel piping, including a laboratory grade oven capable of heating samples to 300 °C (LO), a tritium measurement furnace and bubblers containing a copper oxide catalyst (COF), a residual gas analyser comprising a mass spectrometer for low gas flows (RGA), 5 sample chambers (SC) each consisting of two 4cm wide mating CF 40 to KF 40 adaptors (with the samples held between the CF joints using a copper O-ring), a tritium delivery vessel and buffer for interim storage between experiments, roughing and turbo pumps for movement of the gas mixture, air and nitrogen lines and water and desiccant chambers for the introduction and regulation of moisture.

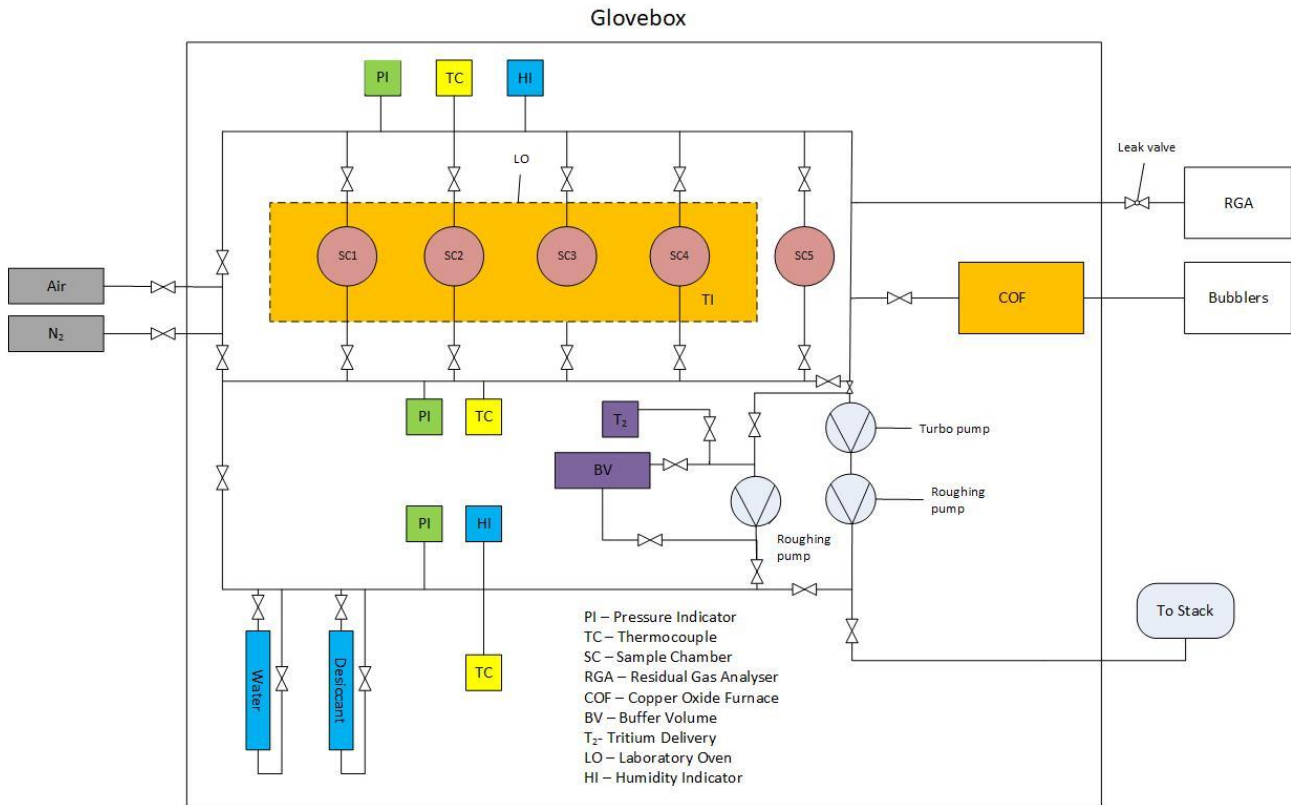


Figure 4: Design of the tritium permeation apparatus

The procedure for the tritium measurements will be to clean the pipework of the apparatus from any residual tritium using a combination of heater tape and a nitrogen purge. Once this has been completed tritium will be introduced into the system from the buffer volume and the required level of humidity will be reached through passing the gas through the water chamber. After this the gas will be introduced into the sample chamber, heated to the set temperature by the oven. Periodic measurements of the gas that has permeated through the membranes will be made by opening the valves downstream of the sample holders. A nitrogen purge through the downstream side of the apparatus will act to flush the tritium through the oxidising furnace and bubbler system. Alternatively, a small pump after the bubblers can be used to draw the tritium through the furnace system at an appropriate rate. The permeation experiments will be made for set temperatures ranging from 50-200 degrees representative of expected disposal facility conditions. The humidity of the gas mixture will be varied and the effect of this upon the recorded permeation/transport rate will be evaluated.

7 Conclusions

The conditions that will be found in near surface repositories and future planned deep geological repositories have potential implications on tritium transport processes through container materials. The majority of experiments regarding permeation of tritium through metals have been conducted at



temperatures higher than 300°C and with HT mixed with an inert gas. Temperature simulations of repository conditions taking into account the ambient temperatures and decay heat of the waste suggest an upper limit of 220°C. Experience of dealing with tritiated metallic waste suggests that the majority of off-gassing will be in the tritiated water form rather than HT. The transport of tritium through a metal membrane can be controlled by surface processes or permeation through the bulk of the material. Research conducted by (Hayashi, Nakamura, & Isobe, 2012) and (Yamasaki, et al., 2012) suggest that for 1mm thick SS304 the process is limited by surface reactions. There is a scarcity of available information regarding the different surface rate reactions of HTO with proposed container materials. This is potentially an important factor in the determination of the tritium performance of different container materials. A selection of different materials has been made based on existing concepts for nuclear waste containers. An experimental apparatus will test these materials to determine whether at the lower temperature range the process is controlled by permeation or by surface reaction. If the process is indeed controlled by surface reactions the different container materials will be evaluated on this basis.



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