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Title: **THREE TRITIUM SYSTEMS TEST ASSEMBLY (TSTA) OFF-LOOP EXPERIMENTS**

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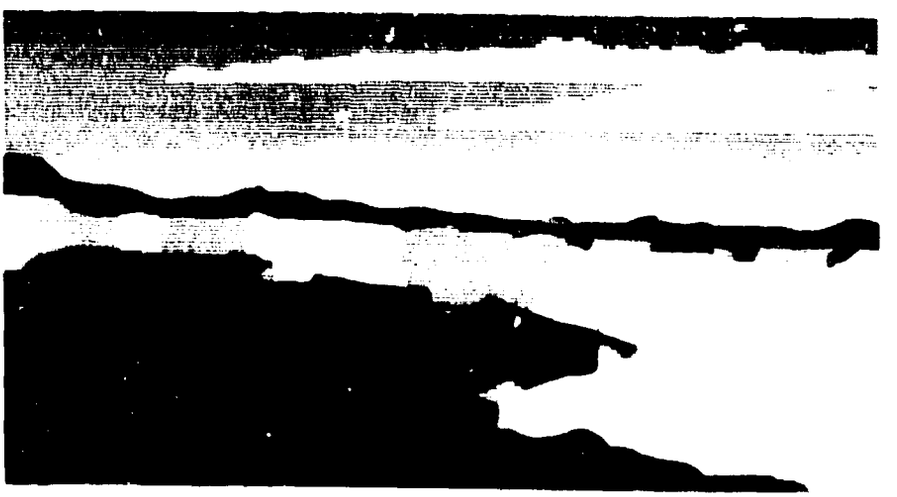

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Three Tritium Systems Test Assembly (TSTA) Off-Loop Experiments

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EXPERIMENT # 1
Effect of Tritium on Viton-A, Buna-n and EDPM Elastomers at 1, 40 and 400 Torr During Valve Cycling

ABSTRACT

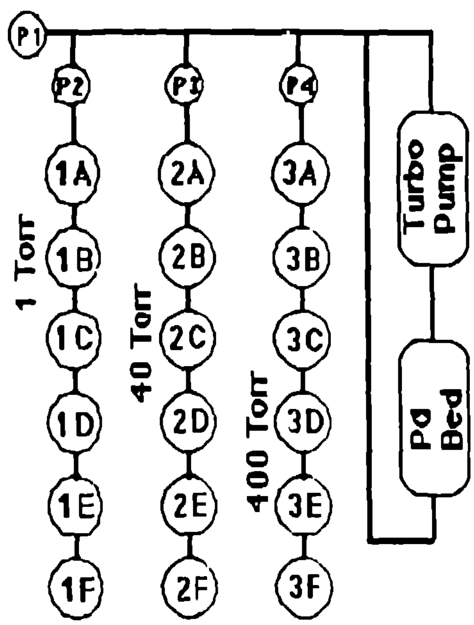
Two O-ring valve seals each of Viton-A, Buna-N, and EDPM were exposed to 1, 40, or 400 torr of tritium while being cycled open and closed approximately 11,500 times in 192 days. EDPM is the least susceptible to damage from the tritium. Both Buna-N and Viton-A showed deterioration following the first cycling at 400 torr .

INTRODUCTION

This work was initiated to establish the possibility of using a soft elastomer in ITER (International Thermonuclear Experimental Reactor) applications. Used in this application, the sealing material is anticipated to be in tritium at pressures in the range of $1x10^{-3}$ torr for many years. Accelerated tests over a range of higher pressures were used in this study in order to extrapolate the results to the low pressure range.

EXPERIMENTAL

Eighteen elastomer O-ring valve seals are studied to determine tritium compatibility under high use conditions for 192 days. Three sets of six test valves, labeled A-F in Fig. 1, are used. Each valve is opened and closed in tritium 1100 times during 11 days then tested for leakage across the valve seat. This procedure is repeated 11 times. During cycling, set 1 contained tritium at 1 torr while sets 2 and 3 contain tritium at 40 torr and 400 torr respectively. Valves not shown in the figure isolate the different valve sets during the cycling and leak test.



P1 0-2 Torr Pressure Transducer
P2 0-10 Torr Pressure Transducer
P3 0-100 Torr Pressure Transducer
P4 0-1000 Torr Pressure Transducer
1A-3F Test Valves
Pd Bed Palladium Bed used to store, supply and pump tritium.

Fig. 1 Simplified diagram of test valve arrangement for the elastomer test

All volumes associated with the test valves are calibrated so that pressure changes can be used to calculate leak rates. A palladium bed is used as the source of tritium during the process of loading tritium into the test valve lines by heating or cooling the bed as necessary to establish the desired tritium overpressure. Tritium transfer to and from the experiment is done with the Self Assaying [illegible] Uranium Bed (SAPUB)[1]

Initially, the [illegible] rates of the test valves are determined [illegible] valve [illegible] leak rate

1 Manuscript submitted Oct. 11, 1993

under $1x10^{-5}$ STP-cc/sec which is the specified limit. Tritium is added to the system in aliquots for a final leak check and to replace the hydrogen adsorbed on the system surfaces. After the valves are cycled for 11 days in the respective pressures, the tritium pressure is equalized in each valve set at approximately 140 torr. Leak rates are determined by closing all valves and evacuating the calibrated manifold beyond the valves in Fig. 1. Once the manifold is isolated from the vacuum, the pressure change in the manifold volume is used to calculate the leak rate of a test valve. Once this measurement is made, the test valve is opened and the pressure is noted to establish an accurate back-pressure behind the valve. The volume is again evacuated and the subsequent pressure rise is used to determine the leak rate through the next test valve. This procedure is used for the remaining valves. As a result, some of the valves at the end of the test cycle experience a pressure of 140 torr for up to 30 hours, although not during the cycling operations. Elastomer placement , shown in Table 1, was staggered in each line to avoid its position effecting the interpretation of the results

Table 1

Elastomer placement in each line

Pressure	Valve Positions					
Torr	"A"	"B"	"C"	"D"	"E"	"F"
1	V	B	E	V	B	E
40	B	E	V	B	E	V
400	E	V	B	E	V	B

V = Viton-A
B = Buna-n
E = EDPM

Fig. 2. Photograph of elastomer seal after tests

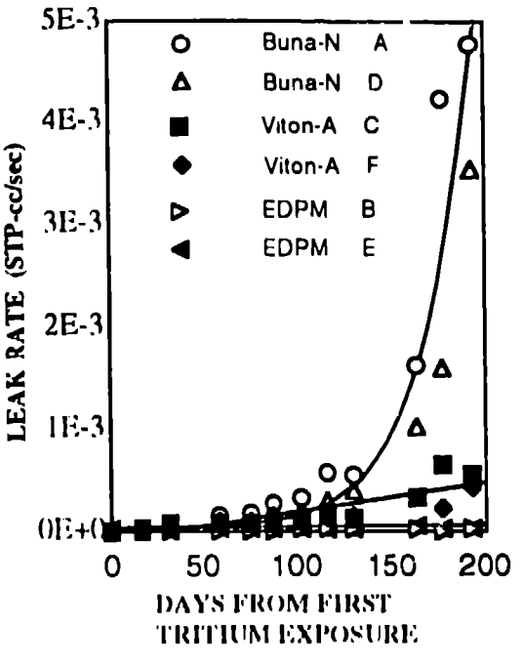


Fig. 3. Leak rate vs. days of tritium exposure during 40 Torr series.

RESULTS

Figure 2 shows a photograph of the three different materials after the test. Buna-n failed most dramatically. The elastomer is dull, hard, brittle and flattened. No large piece of the elastomer could be removed intact. Viton-A is intermediate in behavior. A few pieces could be extracted from the valve. The surface of this material is also flat and dull. EDPM shows the best compatibility with tritium. Although the surface of the elastomer shows dullness, it can be removed intact.

Figure 3 shows leak rates from the 40 torr set of valves as a function of time after initial exposure. This time is somewhat longer than the time during which the material was in actual contact with tritium. Buna-n clearly responds poorly to tritium, the Viton A slightly better, and the EDPM is best. As leak rates became large, valves at the end of the series lost significant back pressure. All back pressures were corrected to 140 torr to compensate for that pressure loss.

Figure 4 shows leak rate as a function of time after initial exposure for Buna-n at the three different pressures. Rapid failure occurs after a delay time that depends on the tritium pressure. This delay time is plotted in Figure 5 as a function of tritium pressure. Extrapolation of the figure 5 data to [illegible] torr suggests that Buna-n would last up to 1 year. The Viton A would clearly last longer and the EDPM would be best. Exact [illegible] could not be obtained from [illegible]

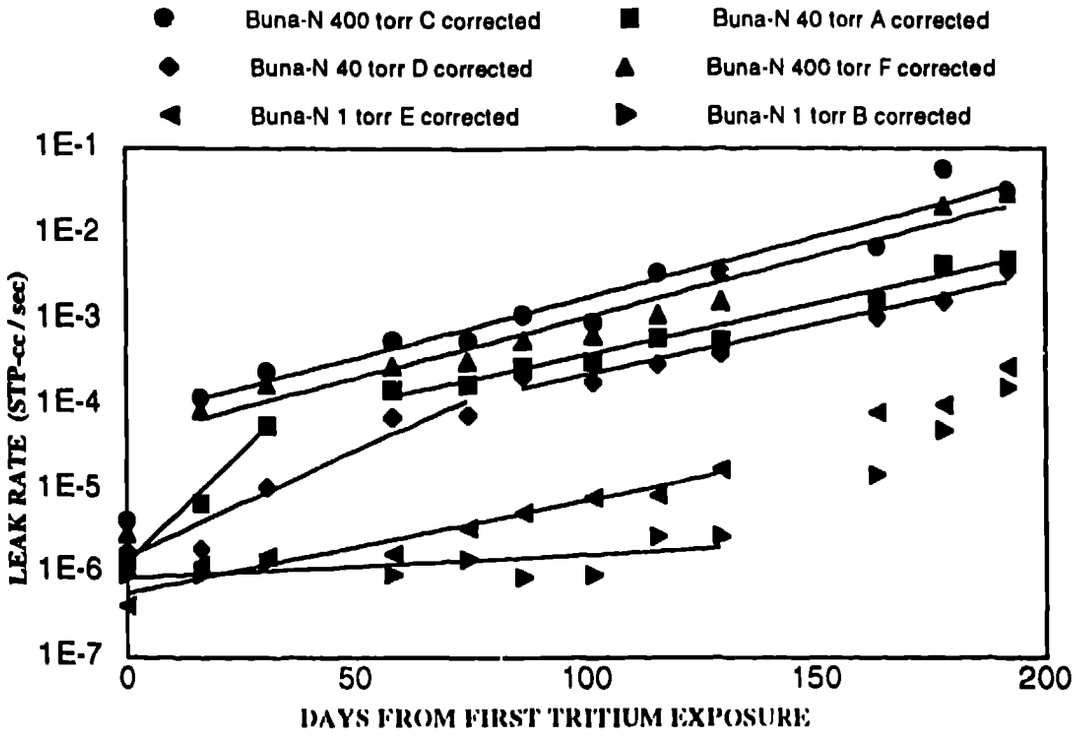


Fig. 4. Leak rate vs.days from first tritium exposure for Buna-n.

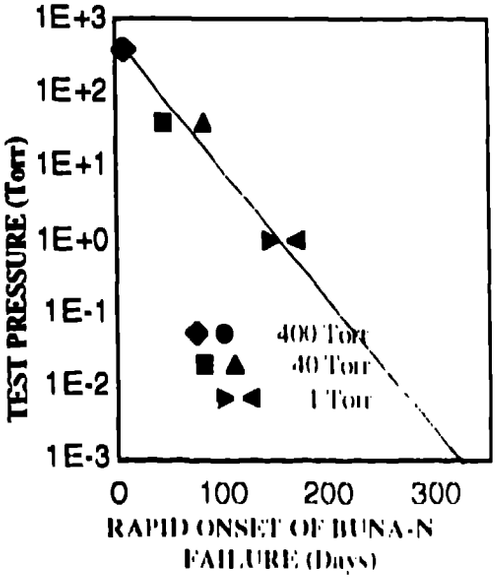


Fig. 5. Extrapolated service life at low pressures.

CONCLUSIONS

Buna-n is estimated to last up to 1 year at 10^{-3} torr before failing rapidly. Quantitative lifetimes have not been obtained for Viton A or EDPM. However, there is no question that of the three materials, EDPM sustains the least damage from tritium exposure. A comparison between this work and that of Wylie et. al.[2] shows that frequent cycling decreases the lifetime of an elastomer seal when in contact with tritium.

EXPERIMENT #2
Tests of a Portable Water Removal Unit Designed to Reduce Stack Emissions

ABSTRACT

Using commercially available materials, the Tritium Systems Test Assembly (TSTA) designed and built a Portable Water Removal (PWR) Unit to reduce tritium oxide emissions during glovebox breaches. The PWR removes >99% of all tritium and saves between 0.7 and 3.5 curies of tritium oxide from being stacked during each of the five tests.

INTRODUCTION

At TSTA it is often necessary to remove a glovebox window to perform maintenance. In order to keep atmospheric emissions of tritium oxide to a minimum, TSTA developed a device to reduce tritium emissions to the stack. The PWR is positioned between a glovebox

and the stack (see Fig. 6) during window removal operations. A more versatile and sophisticated system for tritium work is in use at Sandia Livermore[3]

EXPERIMENTAL

The system shown in Figure 6 is placed between a glovebox and the stack. The molecular sieve is contained between two barriers. The top barrier is a screen similar to that used on a screen door and keeps the sieve in the holder if the unit is tipped. The bottom frit is 5μm, 40μm or 100μm pore size fritted steel and keeps tritiated dust from entering the blower and ducts. Because of the large flow resistance, this frit is a major factor in restricting airflow. Any number of straight segments filled with sieve can be added. Temperature of the sieve is monitored as well as air flow. Gas is sampled for tritium concentration before and after the sieve. This gas is returned to the PWR.

Disposal of the sieve has not created a problem because the tritiated sieve is poured into solid-waste disposal barrels. These barrels contain tritiated plumbing and other large parts so that the sieve occupies the void spaces after the barrel is "filled" thus creating no additional waste barrels.

When a glovebox breach is planned, a moisture source is placed in the glovebox the night before the PWR is used. This exchanges HTO adsorbed on the glovebox walls with H_2O.

The PWR is connected to the glovebox through a glove port while a window or second glove-port opening supplies room air.

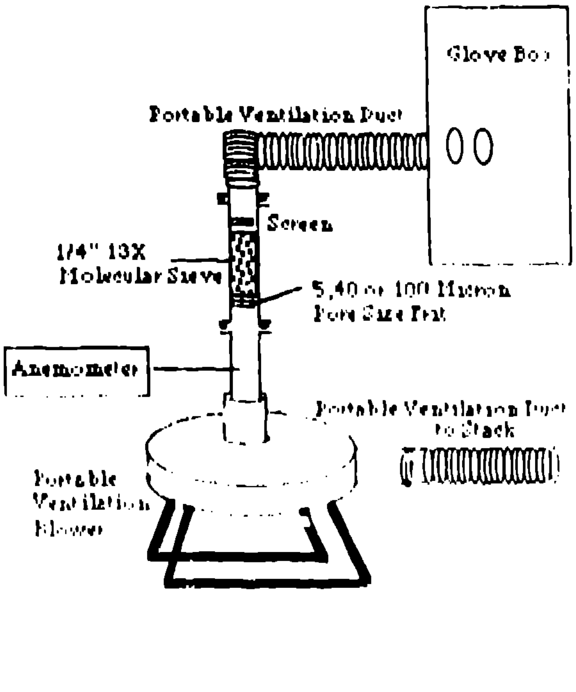


Fig. 6 A simplified Schematic of the PWR

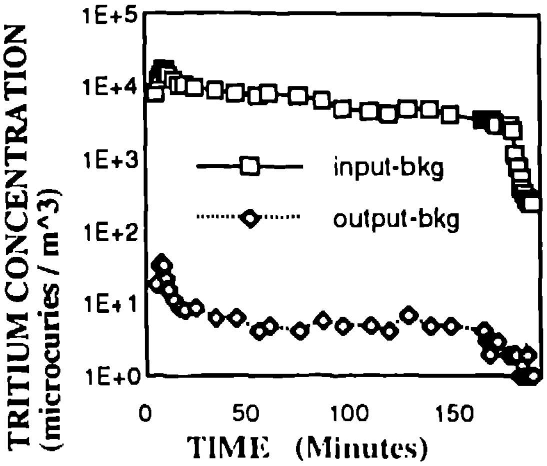


Fig. 7. Tritium input and output of the PWR. One Curie was collected in run 3.

ing supplies room air. Tritium concentration at the inlet and outlet of the PWR during a typical run is shown in Fig. 7. One curie of tritium was collected in the sieve during this run. * To be corrected

The following information is recorded: tritium concentration entering the PWR, tritium concentration leaving the PWR, flow rate of the gas stream in the PWR, total quantity of gas through the PWR, and time of the reading. Temperature of the sieve container is used as a guide to indicate when the sieve may be near saturation. Breakthrough was not observed during these tests. Table 2 shows that the PWR is very effective in reducing tritium emission. Additional studies are needed to determine the optimum gas flow rate and the effectiveness of catalytic-palladium coated sieve to convert T_2 to T_2O within the PWR[4]

Table 2
Results of PWR tests

Run	Pore Size micron	Efficiency[1] percent	Tritium Trapped curie
1	5	[illegible]	[illegible]
2	5	[illegible]	1.08
3	5	[illegible]	1.18
4	5	[illegible]	[illegible]
	100	[illegible]	[illegible]

[1] [illegible] PWR [illegible] tritium [illegible] 100

[2] Tritium [illegible] the run

ACKNOWLEDGEMENTS

Thanks are given to all TSTA personnel for their helpful suggestions and participation in PWR tests.

EXPERIMENT #3
Performance of Hydrogen /Oxygen Recombination Catalysts in the Presence of Tritium and SF_6

ABSTRACT

A series of tests are done to determine whether the presence of SF_6 changes the ability of palladium and platinum to catalyze the T_2-O_2 reaction to form T_2O. No deterioration of the catalytic activity is observed.

INTRODUCTION

Tokamak Fusion Test Reactor (TFTR) requires information about the effect of SF_6, an electrical insulator, on the catalytic behavior of Pt and Pd in a T_2 environment. This information is necessary for the accident analysis in the Safety Analysis Report for TFTR. This study is done using an apparatus supplied to TSTA by TFTR.

EXPERIMENTAL

Figure 8 shows a simplified schematic of the SF_6 system. The gases (SF_6, dry air and T_2) are mixed, preheated, and passed through the catalyst bed. Pd on alumina at 350°F (177°C) or 500°F (260°C), or Pt on alumina at 950°F (510°C) are used as catalysts. All T_2O resulting from the reaction is collected in a water bubbler. Unreacted T_2 passes through the bubbler and is converted to T_2O in an oxidized-copper furnace at 750°F (399°C). The T_2O formed in the Cu furnace is collected in a second bubbler. Remaining gas is returned to the mixing volume. An ion chamber with a small circulating pump is attached to the mixing volume and used to monitor tritium concentration in the gas. Gas circulation is continued for 30 minutes after the tritium level has dropped below 9% of the original value. Hydrogen is then flushed through the system to remove any adsorbed tritium. An additional step is required when the palladium catalyst is used. Because the alumina substrate tends to absorb water, the Pd catalyst is heated to 750°F. To drive any tritiated water into the bubbler.

Prior to every run, dry air is passed through the system while only the Copper bed is heated. Once the Cu is fully oxidized, the catalyst is tested at each of the temperatures using a mixture of dry air and tritium. This test is followed by a similar study using a mixture of dry air, tritium and SF_6. Each gas sample contained approximately one curie of tritium, and about 35 Ci/m^3 in the total gas sample. All experiments are done using a single sample of each catalyst.

The bubblers are filled with a measured amount of ethylene glycol or water. After gas is circulated for 10-15 minutes, liquid samples are removed for scintillation analysis. Dilution is based on weight rather than volume measurement in order to reduce error.

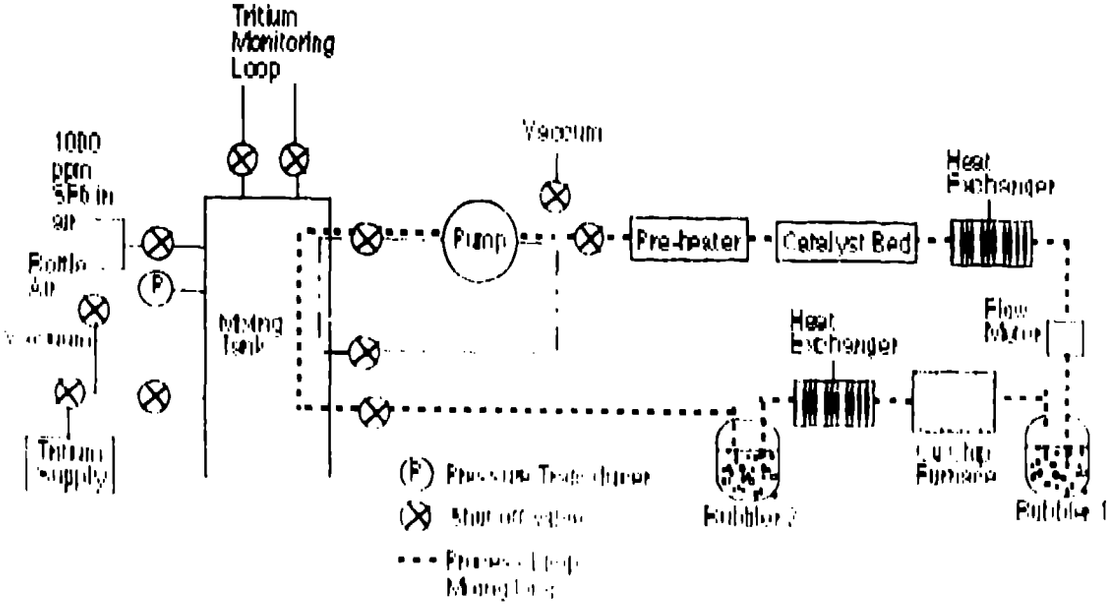


Fig. 8 Simplified [illegible]

RESULTS

Effectiveness of the catalyst is determined by dividing the tritium concentration in the first bubbler by the sum of the tritium concentrations in the first and second bubblers. The results are tabulated in Table 3. The two runs on Pd at 350°F show adequate repeatability for the supplied system. The presence of SF_6 appears to have no effect on the catalytic activity of these catalysts. TO BE CORRECTED

Table 3
Efficiency of T_2-O_2 reaction
with and without SF_6 present

Material	Temperature F	% Efficiency T_2	% Efficiency $T_2 + SF_6$ (ppm)
Palladium	350	97.8, 97.2	99.0 (35 ppm)
Palladium	500	96.9	96.5 (35 ppm)
Platinum	350	98.7, 98.5	*
Platinum	500	98.1	*
Platinum	950	98.5	98.2 (35 ppm)
			97.5 (100 ppm)

*Runs were determined to have less priority by TFTR and were preempted.

CONCLUSIONS

The presence of SF_6 appears to have no effect on the catalytic activity of these catalysts.

ACKNOWLEDGEMENTS

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The writer participated in all three of these experiments after the concepts and original construction were complete. I thank all of the people who made this possible. I also thank my husband, Edmund Storms, who patiently helped me edit this manuscript to produce a camera ready format

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