^{4}He Outgassing Technical Report

CEES, TTU

Introduction:

The experiments for measuring helium outgassing from various materials used in the gas loading and electrochemical loading experiments in D2/Pd system is important in improving the overall quality of helium measurements. It is also important to know at what temperature the helium outgassing begins in these materials to make sure to avoid those temperatures in the experimental process. These experiments that are performed using helium leak detection technique provide a way to estimate the outgassing temperature and the amount of picomoles of helium outgassed from different materials.

Results and Analysis:

The ^{4}He leak rate was measured with respect to the time to find the temperature of helium outgassing and the amount of ^{4}He picomoles outgassed. The outgassing temperature was obtained by finding the exact time stamp, the "increase in ^{4}He outgassing" happened and correlating it with the temperature measurements with respect to time obtained from a LABVIEW program. The mount of picomoles outgassed are calculated using the ideal gas law.

Ideal gas law; $PV = nRT \Rightarrow n = \frac{PV}{RT}$

Integrating ^{4}He leak rate over time gives PV term in the ideal gas law. The temperature in this calculation is used as 298 K because the helium leak rate obtained from the leak detector corresponds to this temperature. Table 1.0 and Figure 1 summarizes the ^{4}He outgassing in picomoles, the outgassing temperature, and calculated values of outgassing per unit area and unit volume for tubing of different sizes.

Table 1.0: Total helium amount outgassed, release temperature of out-gassing and outgassing per unit area and unit volume for different size tubing, Swagelok, Wall thickness 0.035''.

Material	Vendor	Sample	4He Out Gassing (picomole)	4He outgassing/ cm^2	4He outgassing/ cm^3	Out Gassing Temperature (°C)
SS 316	Swagelok	12-11-17-3/8-SS	43	0.58	5.8	~400 °C from notebook
SS 316	Swagelok	02-12-18 -3/8-SS	30	0.40	4.1	
SS 316	Swagelok	01-26-18-1/2-SS	240	2.3	24	
SS 316	Swagelok	12-13-17-3/8-SS	70	0.94	9.5	
SS 316	Swagelok	03-23-18-1/4-SS	7.8	0.18	1.7	
SS 316	Swagelok	8-001, ½-SS316	64	0.61	6.4	400
SS 316	Swagelok	8-002, ½-SS316	92	0.88	9.2	---

SS 316	Swagelok	8-004, ½-SS316	58	0.55	5.8	386
SS 316	Swagelok	8-005, ½-SS316	36	0.34	3.6	386
SS 316	Swagelok	8-006, ½-SS316	15	0.14	1.5	427
SS 316	Swagelok	8-007, ½-SS316	92	0.88	9.2	427
SS 316	Swagelok	8-009, ½-SS316	50	0.49	5.1	401
SS 316	Swagelok	8-010, ½-SS316	35	0.34	3.6	410
SS 316	Swagelok	8-011, ½-SS316	22	0.21	2.2	442
SS 316	Swagelok	8-012, ½-SS316	42	0.41	4.3	401

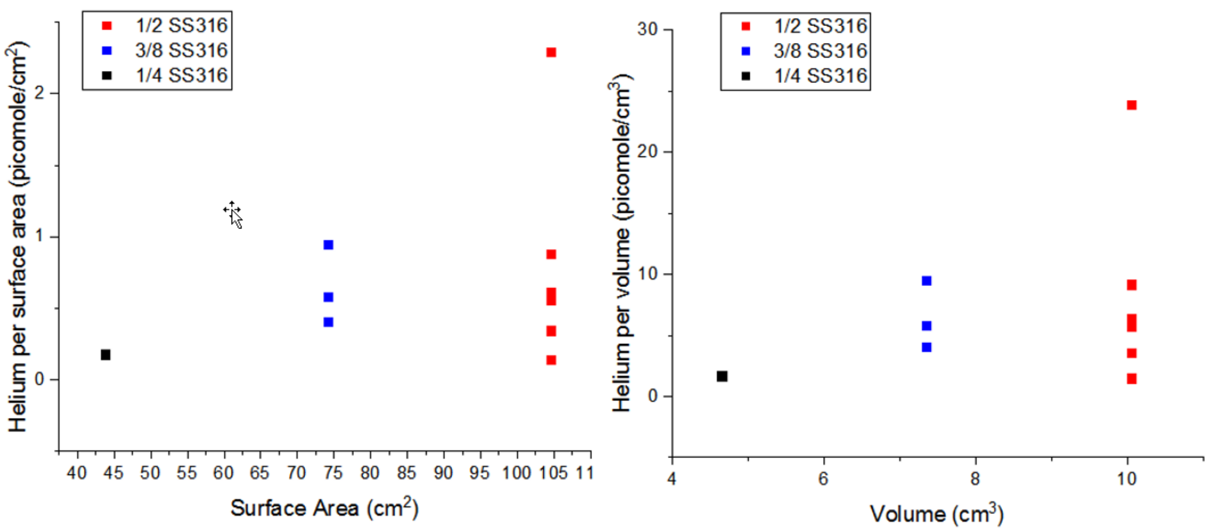


Figure 1 summarizes the ^{4}He outgassing in picomoles, the outgassing temperature, and calculated values of outgassing per unit area and unit volume for tubing of different sizes.

The stainless-steel tubes were pre-baked and kept exposed to atmosphere for 24 hours, 15 days and 27 days and used in the experiment to find the ^{4}He outgassing rates. Table 1.1 contains the resulted ^{4}He picomoles outgassed for the tubes under different conditions.

Table 1.1: Total helium amount outgassed and release temperature of out-gassing after being exposed to atmosphere for a certain time.

Material	**Size**	**Sample Status**	**He Out Gassing (picomoles)**	**4He outgassing picomoles / cm²**	**4He outgassing picomoles / cm³**	**Out Gassing Temperature (°C)**
SS 316	3/8 OD	Pre-baked and He soaked for 24 hours	16	2.2E-01	2.2	400
SS 316	3/8 OD	Pre-baked and kept in atmosphere for 24 hours	0.52	7.0E-03	7.0E-02	400

SS 316	3/8 OD	Pre-baked and kept in atmosphere for 15 days	18	2.4E-01	2.4	400
SS 316	3/8 OD	Pre-baked and kept in atmosphere for 27 days	0.88	1.2E-02	0.12	400

When performing ^{4}He outgassing experiment for various materials such as Teflon, MACOR and cathodes from CF cells, 3/8" or ½" stainless steel tube with a fused end, pre-baked at 600 ℃ was used as the sample holder. Table 1.2 summarizes the total number of picomoles of ^{4}He outgassed for different samples under different conditions. The experimental procedure followed in this experiment is described in detail in the appendix of the report. The relevant graphs of ^{4}He outgassing rates with respect to time for each experiment is also included in the appendix of the document.

Table 1.2: Total helium amount outgassed and release temperature of out-gassing from different materials.

Sample	Weight/ Size	Baking Temperature (°C)	^{4}He Out Gassing (picomoles)	^{4}He outgassing picomoles / gram	Out Gassing Temperature (°C)
Blank Palladium plate	---	600	2.7	---	---
Seashore PTFE	1.25 g	450	281	224.8	---
Large Seashore PTFE tube	1.009 g	600	50	49.55	above melting temperature of PTFE
Small Seashore PTFE tube	1006 g	500	87	0.086	
AWG PTFE tube	1.006g	500	48	47.71	above melting temperature of PTFE
MACOR as delivered	13 g	600	280	21.54	600
MACOR Prebaked, 2 days in atmosphere	13 g	600	110	8.46	---
MACOR Soaked in 4He 8 hours (25 **°C)**	13 g	RT	5400	415.38	RT
MACOR Soaked in 4He 8 hours (600 **°C)**	13 g	600	3800	292.31	---
CF Cell Diffusion Test	---	RT	500	---	RT
4He soaked CF Cell 2	---	RT	290000	---	RT
(4)3" Teflon Stripes, McMaster from our lab	7.061 g / 23.5 cm^2	600	7100	1005.5	~380
(4) Nylon Nuts	0.191 g	400	1.6	8.38	No sign of outgassing
Nylon Screws	0.184 g	400	3.6	19.57	No sign of outgassing
PTFE TWTT-22C	0.66 g	550	9.9	15	360
PTFE Plate washer	3.857 g	550	19	4.93	360
Nylon Nuts	0.807 g	400	0.8	0.99	No sign of sudden outgassing at high temperature
Nylon Screws	0.921 g	400	0.44	0.48	No sign of sudden outgassing at high temperature

- PTFE melting temperature: 327 ^{o}C
- Nylon melting temperature: 220 ^{o}C; decomposition temperature: 307 ^{o}C

APPENDIX:

Experimental Procedure:

The experiment for measuring amount of helium outgassed from various materials were performed using a tube furnace (LINDENBERG/BLUE) and a helium Leak Detector (Pfeiffer Vacuum ASM 340). The ASM 340 leak detector was calibrated to measure 4He leak rate. There are two possible test methods in ASM 340; Hard vacuum and Sniffing test. The hard-vacuum option was used in this experiment.

In the experiment for finding helium outgassing from stainless steel tubing, the tubes are first fused from one end by welding. A known length of stainless steel tubing of different diameters (1/4", 3/8", ½") are used in this experiment. Then the tubes are marked with a sample Identification number using an engraving tool. The furnace cap was modified to support three tubing samples at the same time. The open end of the tube is connected to the ASM 340 leak detector with a valve in between to shut off when needed. J-type thermocouple is used to measure the temperature of the surface of the tube being analyzed. The temperature profile with time is recorded and plotted in real time using a LABVIEW program. The ASM 340 detector was set to give the rate of helium outgassing in torr liters per second every second. At the beginning of the experiment, the valve that is between the ASM 340 leak detector and the sample is closed, and the pumping is started. This will pump any moisture, dirt and unwanted gases out from the vacuum hose. The lab view program is also executed at the same time with the leak detector. After the base line of the pump is reached, the valve to the tube sample is slowly opened to evacuate the sample. After all the helium outgassing at room temperature is recorded, the temperature controller is turned on to heat the tube sample up to 600 °C within 20 minutes. The helium outgassing rate is continued to measure using the leak detector and the temperature profile is recorded by the LABVIEW program. Once the heating is completed, the temperature controller is turned off to cool down the sample back to room temperature. The two sets of data co-relate by the time of measurement in each set of data. After the valve is open the helium leak rate increases and then decreases back to the base line. The temperature at which this increase starts to happen is found by comparing the time stamp in the temperature measurement obtained from the LABVIEW program with the time stamp obtained from the helium leak detector. The amount of helium out gassed in picomoles are calculated by integrating the helium leak rate with time within the peak and using the ideal gas law.

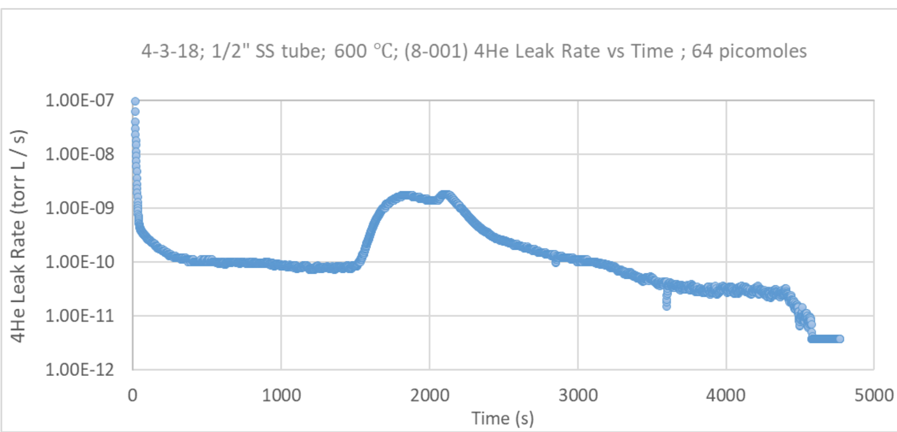
Materials such as Teflon, Nylon and MACOR were also used in a similar experiment in obtaining a rough estimate on the temperature that the helium outgassing starts, and the amount of helium outgassed under increasing and decreasing temperature from these materials. A one end fused stainless steel tube is used to hold the sample inside the furnace. First the stainless-steel tube is placed inside the furnace and is connected to the ASM 340 leak detector. Then the stainless-steel tube is baked at 600 °C for 1 hour while being connected to the helium leak detector to remove any helium previously absorbed by the tubing. Once the initial baking is completed, furnace is set to cool down before inserting the specific sample. Then, the sample is placed inside the stainless-steel tubing and the tubing is reconnected to the helium leak detector. The temperature controller in the furnace is set to reach 600 °C within 10 minutes. The leak detector and the temperature controller are switched on approximately at the same time. The temperature was assumed to be increased at a rate of 1°C per second during this preliminary experiment.

Improvements to the experiment:

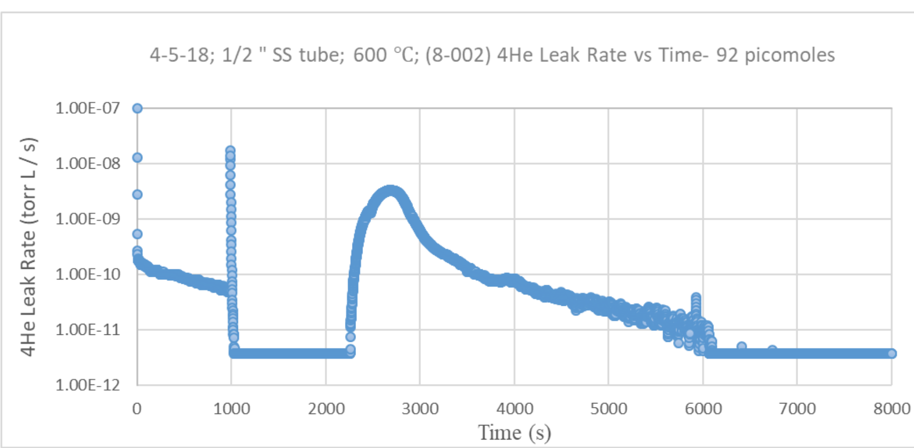
Some recent improvements to this experiment are connecting thermocouple to the tubing samples and using a LABVIEW program to record exact temperature at a given time during the experiment and modifying the furnace cap to hold up to three samples at the same time.

Some planned improvements in this experiment are extending the helium outgassing experiments for the tubing with different size, inner surface area and different manufactures. Performing helium outgassing experiments for capillary tubes, PTFE blocks and tubing and developing an automated data acquisition and analysis using LabVIEW.

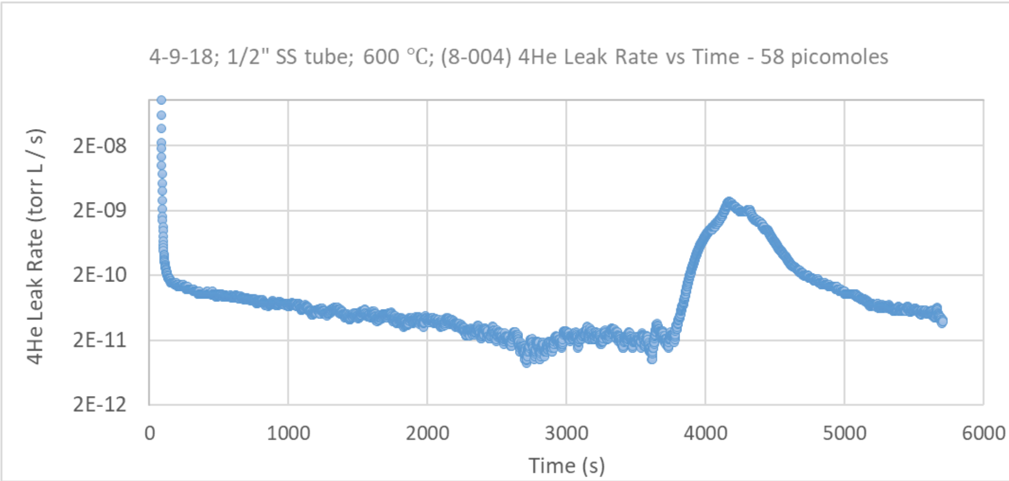
½'' SS316 tubing, 001, Swagelok



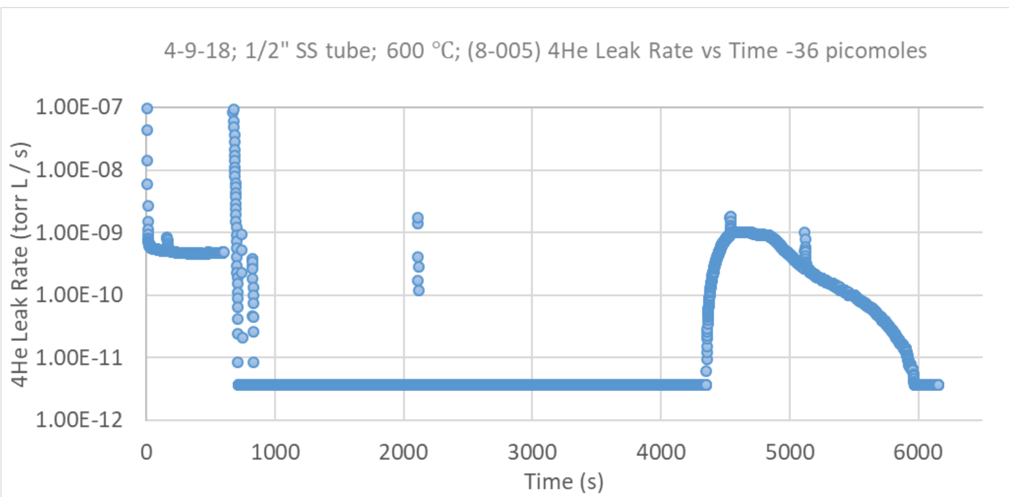
½'' SS316 tubing, 002, Swagelok



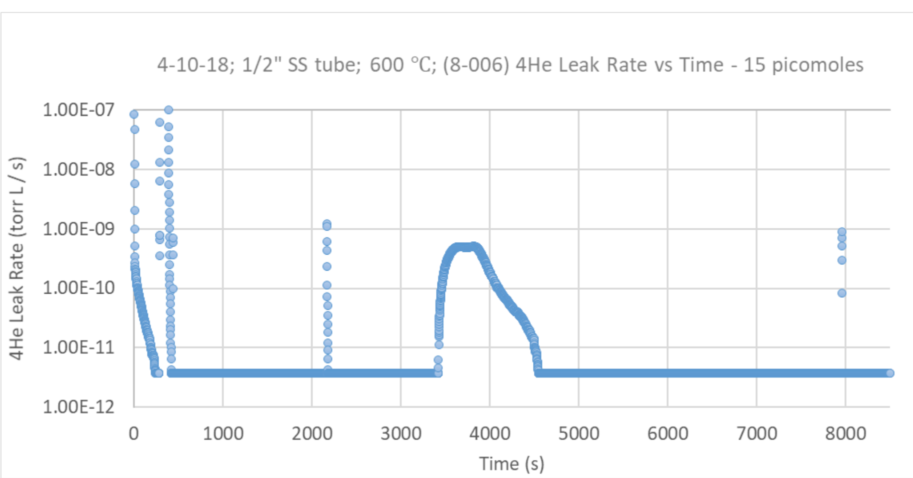
½'' SS316 tubing, 004, Swagelok



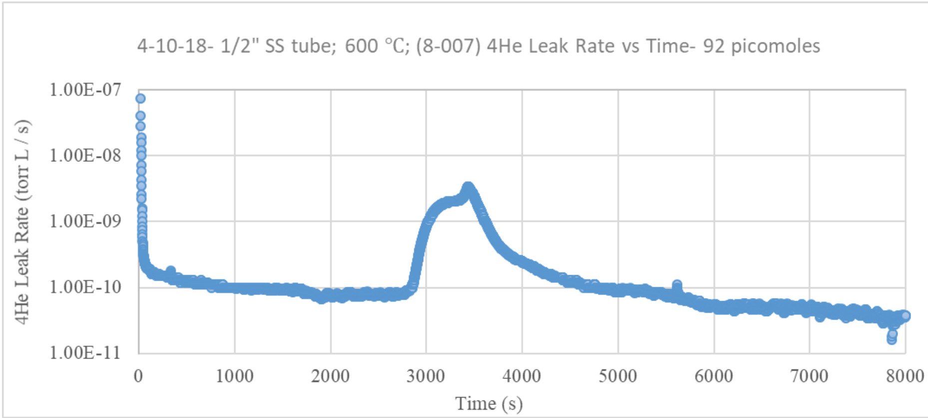
½'' SS316 tubing, 005, Swagelok

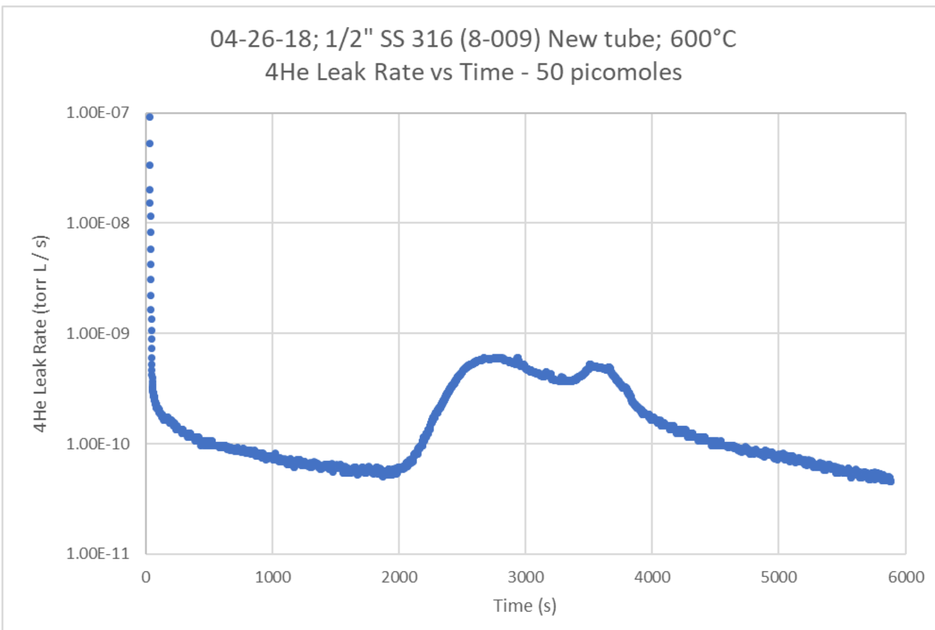


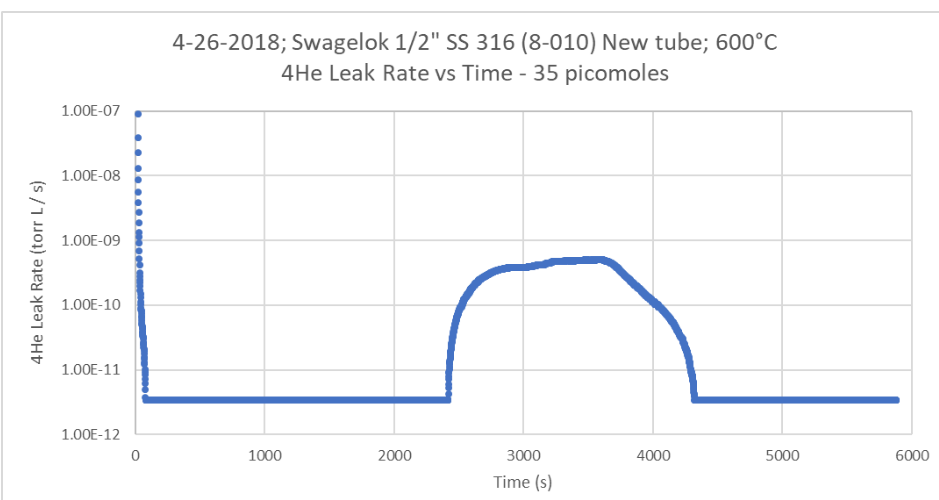
½'' SS316 tubing, 006, Swagelok

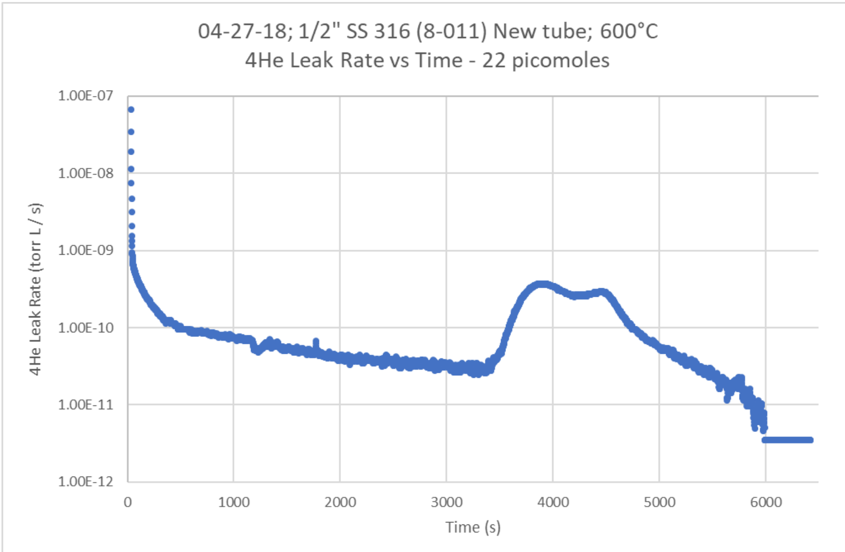


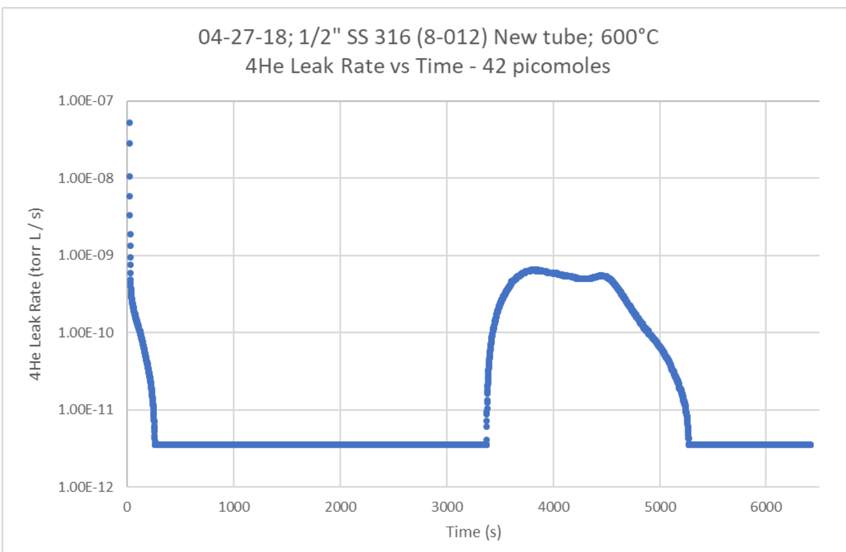
½’’ SS316 tubing, 007, Swagelok

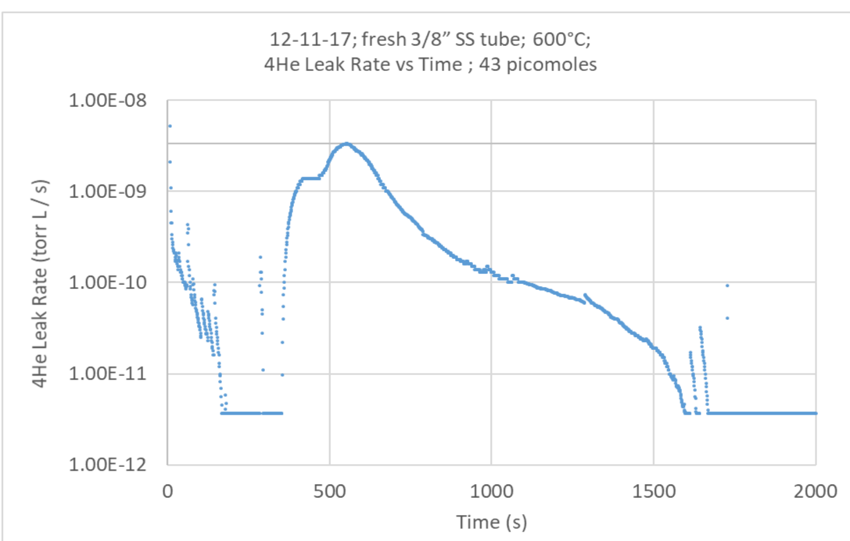


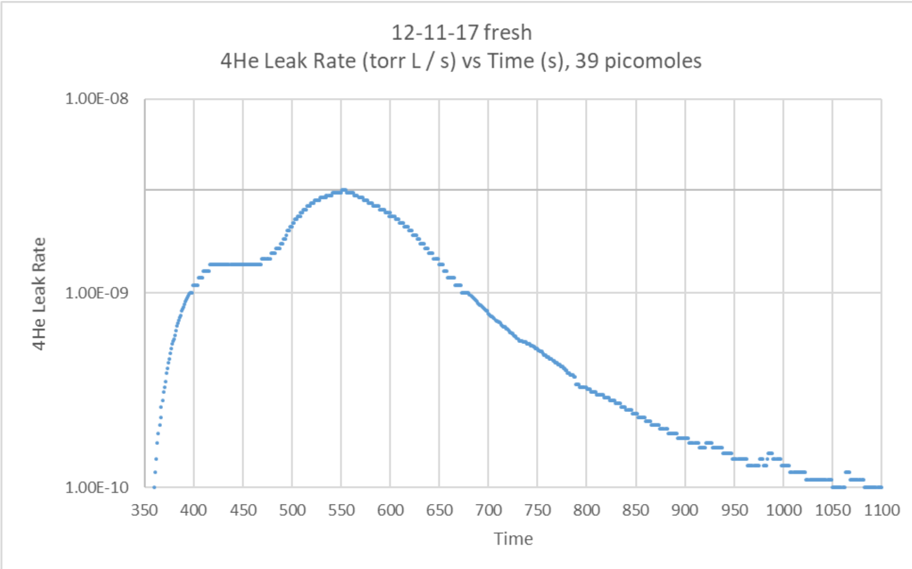


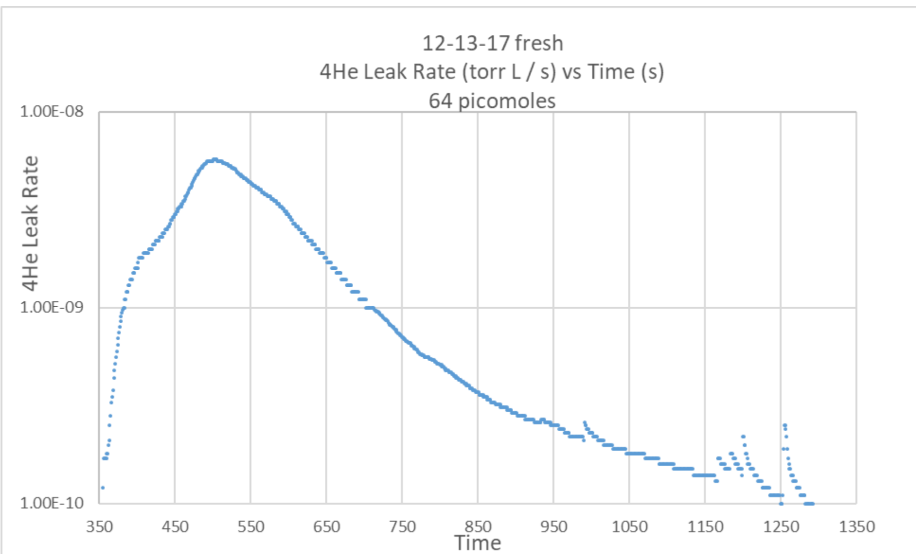


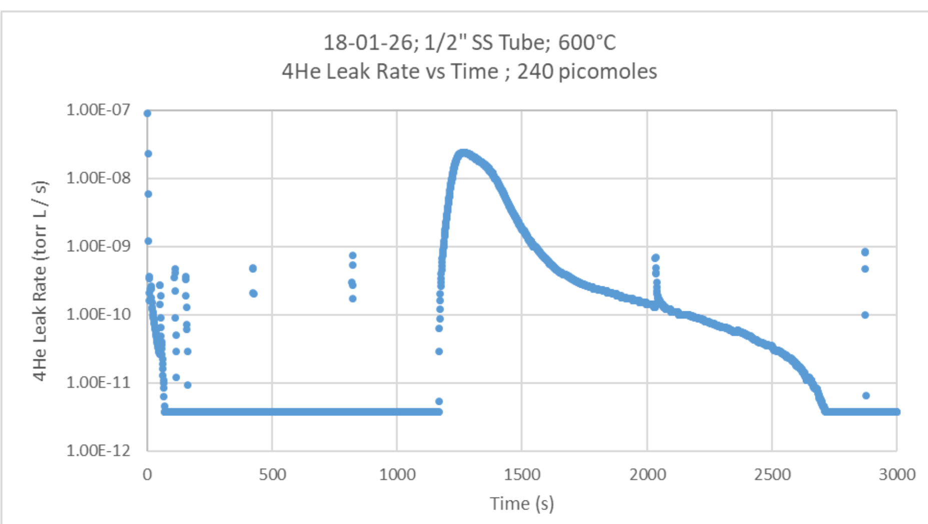

04-27-18; 1/2" SS 316 (8-011) New tube; 600°C
4He Leak Rate vs Time - 22 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
1000
2000
3000
4000
5000
6000
Time (s)

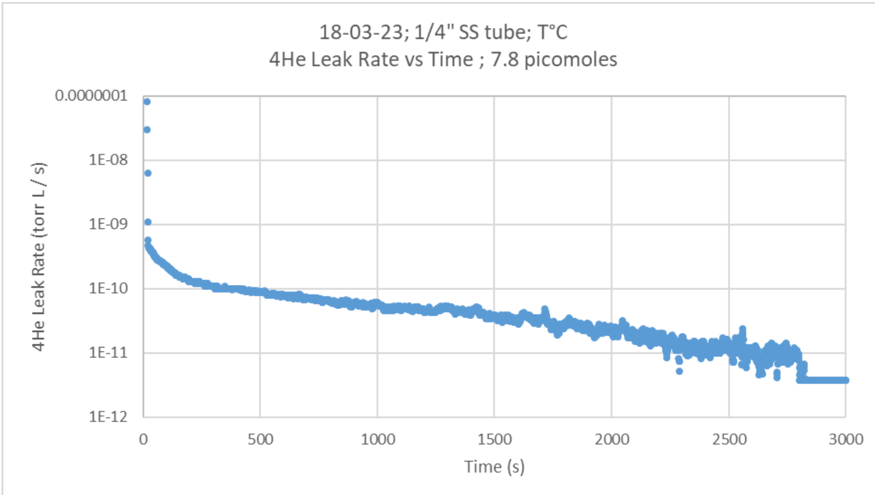

04-27-18; 1/2" SS 316 (8-012) New tube; 600°C
4He Leak Rate vs Time - 42 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
1000
2000
3000
4000
5000
6000
Time (s)


12-11-17; fresh 3/8" SS tube; 600°C;
4He Leak Rate vs Time ; 43 picomoles
4He Leak Rate (torr L / s)
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
500
1000
1500
2000
Time (s)

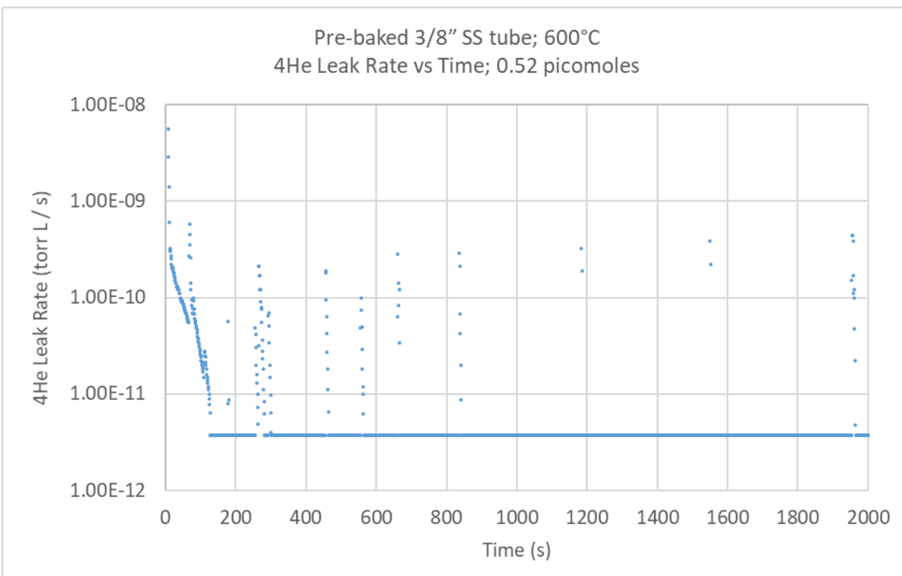

12-11-17 fresh
4He Leak Rate (torr L / s) vs Time (s), 39 picomoles
4He Leak Rate
1.00E-08
1.00E-09
1.00E-10
350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100
Time

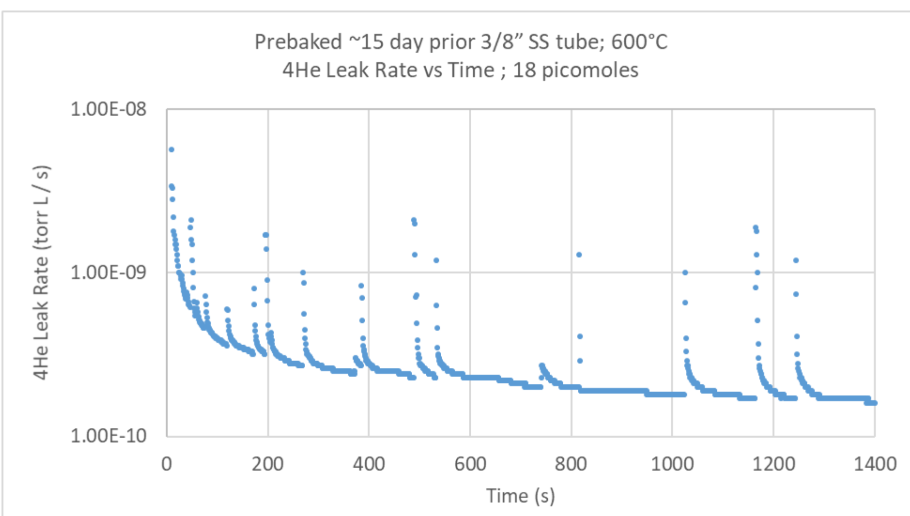

12-13-17 fresh
4He Leak Rate (torr L / s) vs Time (s)
64 picomoles
4He Leak Rate
1.00E-08
1.00E-09
1.00E-10
350 450 550 650 750 850 950 1050 1150 1250 1350
Time

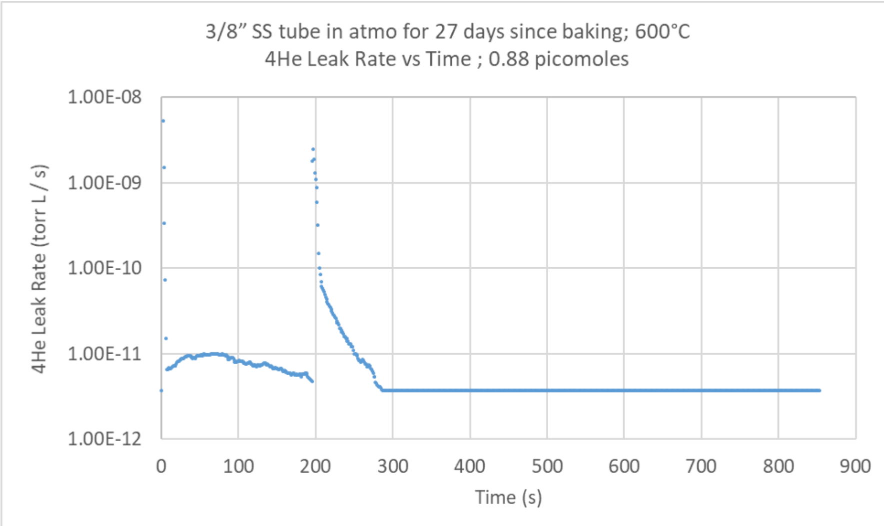

18-01-26; 1/2" SS Tube; 600°C
4He Leak Rate vs Time ; 240 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0 500 1000 1500 2000 2500 3000
Time (s)

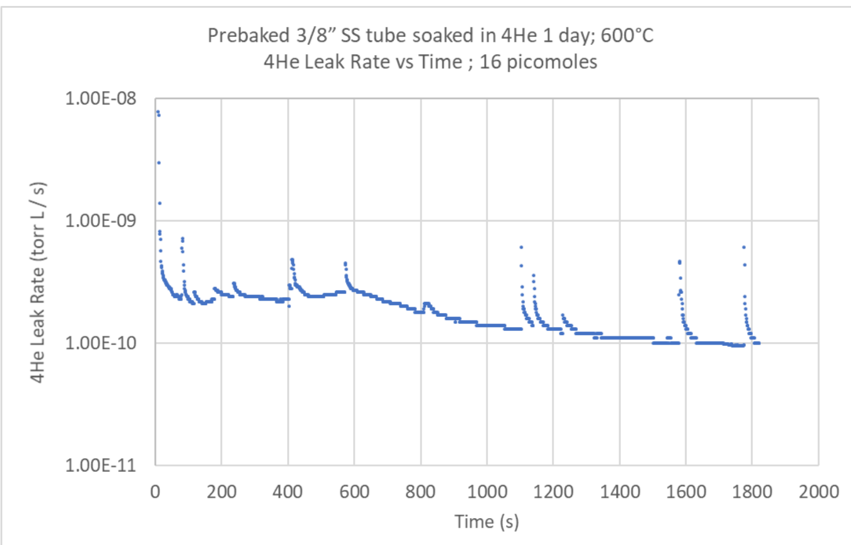


Pre-baked Stainless-Steel tubing

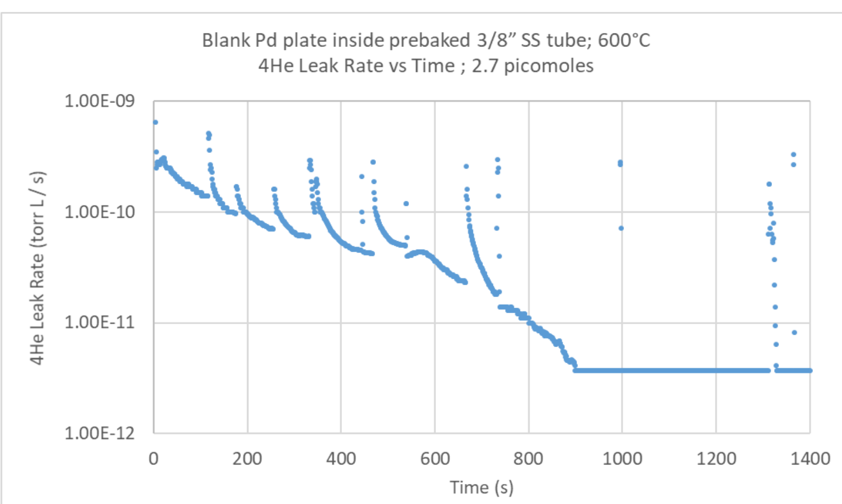


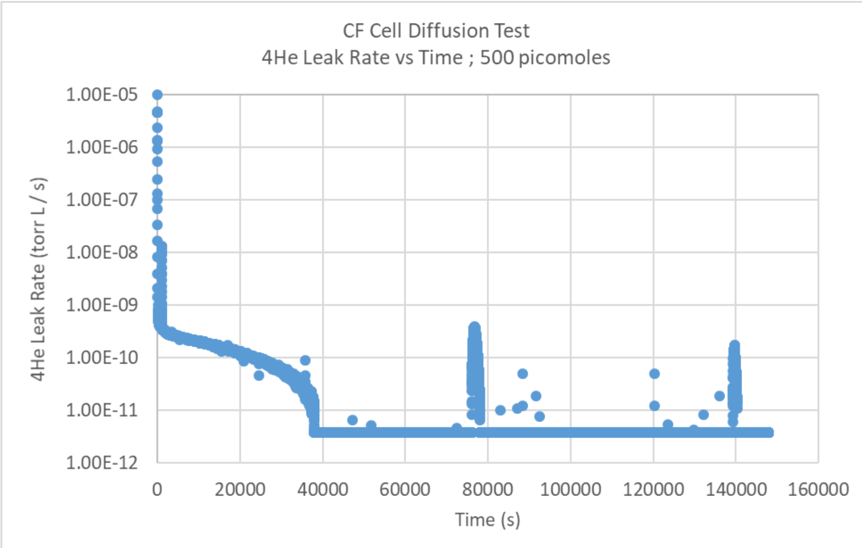




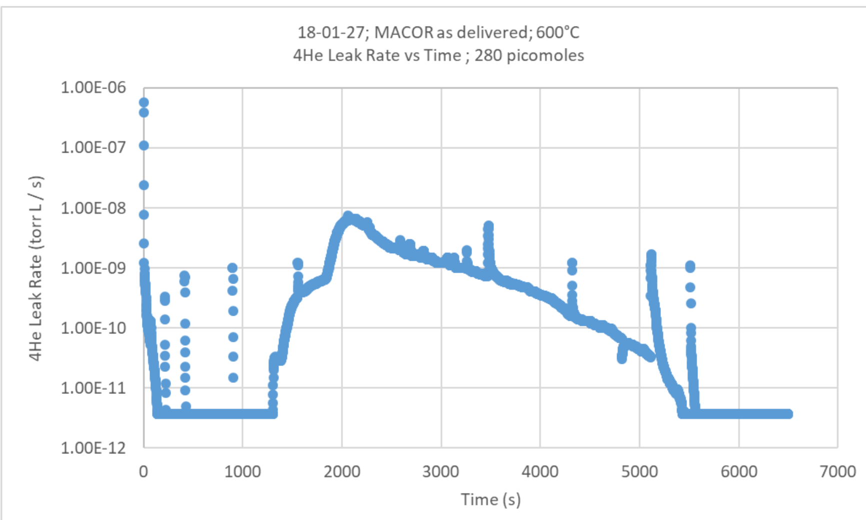


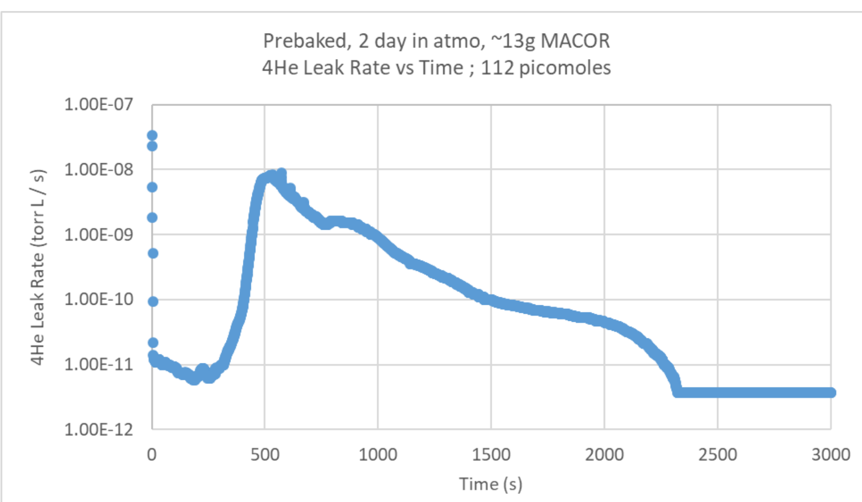
Other Materials:

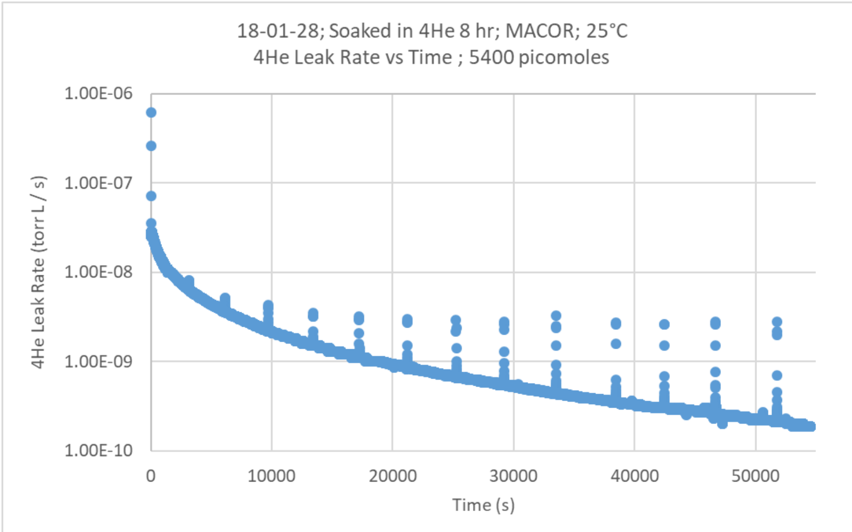


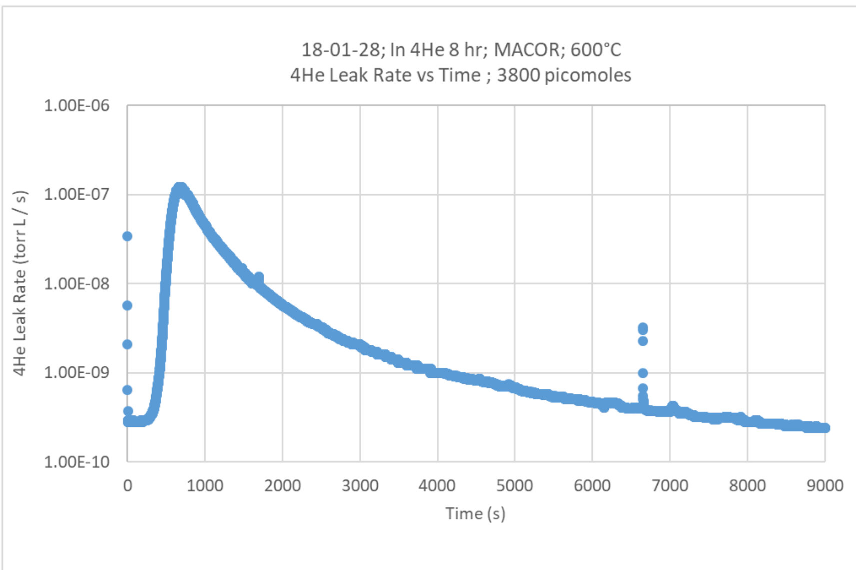

CF Cell Diffusion Test
4He Leak Rate vs Time ; 500 picomoles
4He Leak Rate (torr L / s)
1.00E-05
1.00E-06
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
20000
40000
60000
80000
100000
120000
140000
160000
Time (s)

MACOR

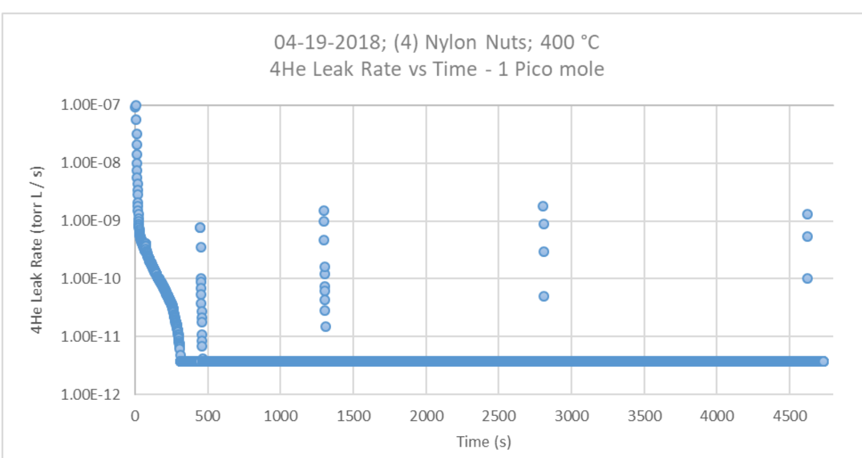

18-01-27; MACOR as delivered; 600°C
4He Leak Rate vs Time ; 280 picomoles
4He Leak Rate (torr L / s)
1.00E-06
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
1000
2000
3000
4000
5000
6000
7000
Time (s)

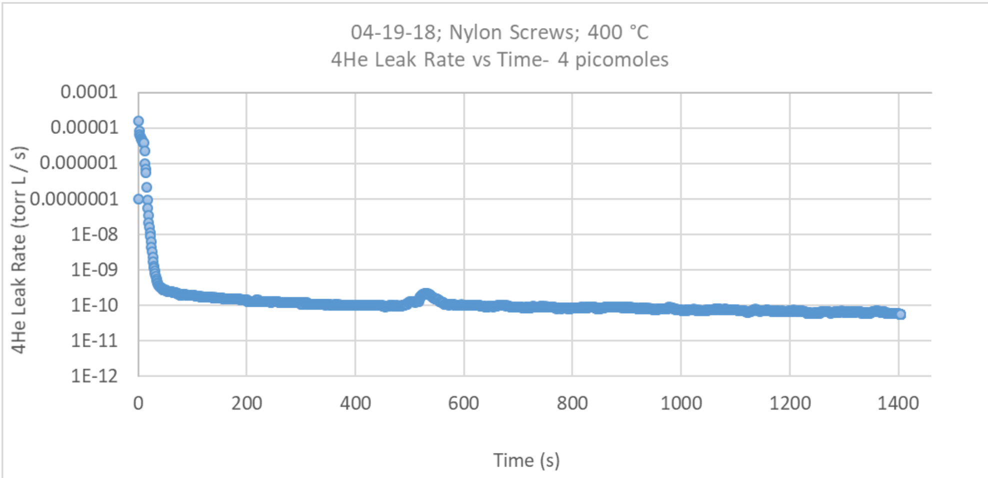

Prebaked, 2 day in atmo, ~13g MACOR
4He Leak Rate vs Time ; 112 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
500
1000
1500
2000
2500
3000
Time (s)


18-01-28; Soaked in 4He 8 hr; MACOR; 25°C
4He Leak Rate vs Time ; 5400 picomoles
1.00E-06
1.00E-07
1.00E-08
1.00E-09
1.00E-10
4He Leak Rate (torr L / s)
0
10000
20000
30000
40000
50000
Time (s)

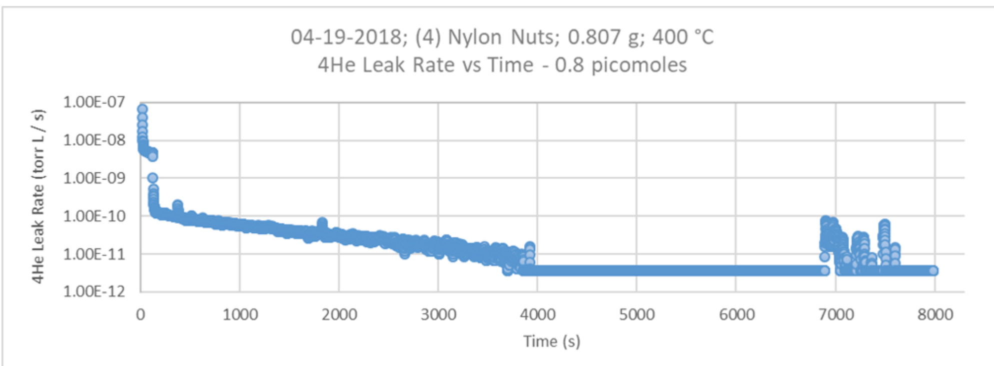

18-01-28; In 4He 8 hr; MACOR; 600°C
4He Leak Rate vs Time ; 3800 picomoles
1.00E-06
1.00E-07
1.00E-08
1.00E-09
1.00E-10
4He Leak Rate (torr L / s)
0
1000
2000
3000
4000
5000
6000
7000
8000
9000
Time (s)

Nylon

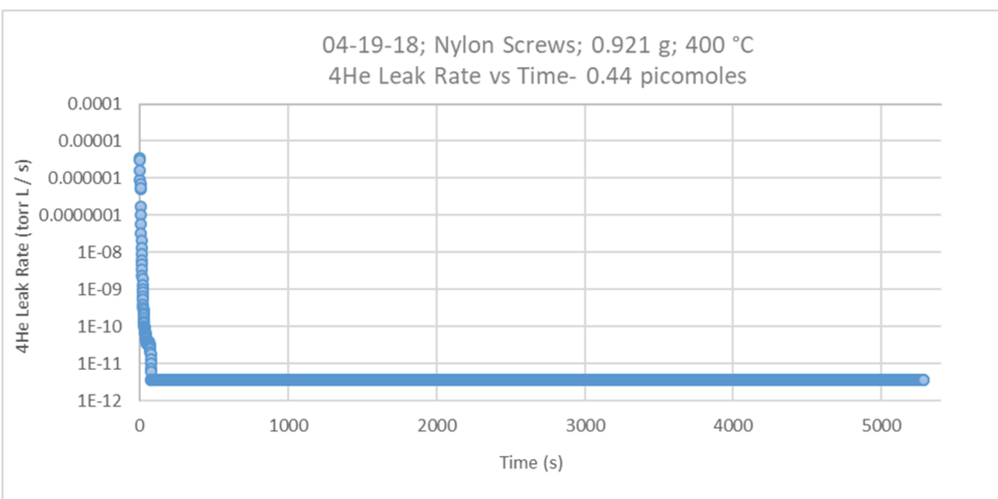

04-19-2018; (4) Nylon Nuts; 400 °C
4He Leak Rate vs Time - 1 Pico mole
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
4He Leak Rate (torr L / s)
0
500
1000
1500
2000
2500
3000
3500
4000
4500
Time (s)



04-19-18; Nylon Screws; 400 °C
4He Leak Rate vs Time- 4 picomoles
4He Leak Rate (torr L / s)
0.0001
0.00001
0.000001
0.0000001
1E-08
1E-09
1E-10
1E-11
1E-12
0
200
400
600
800
1000
1200
1400
Time (s)

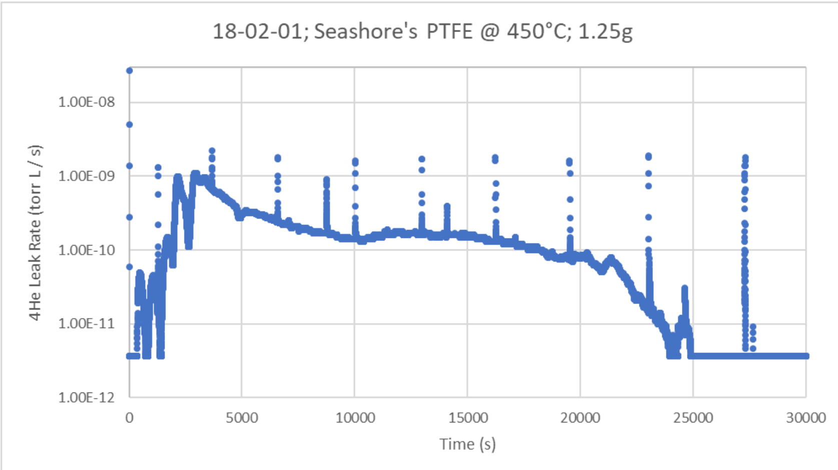


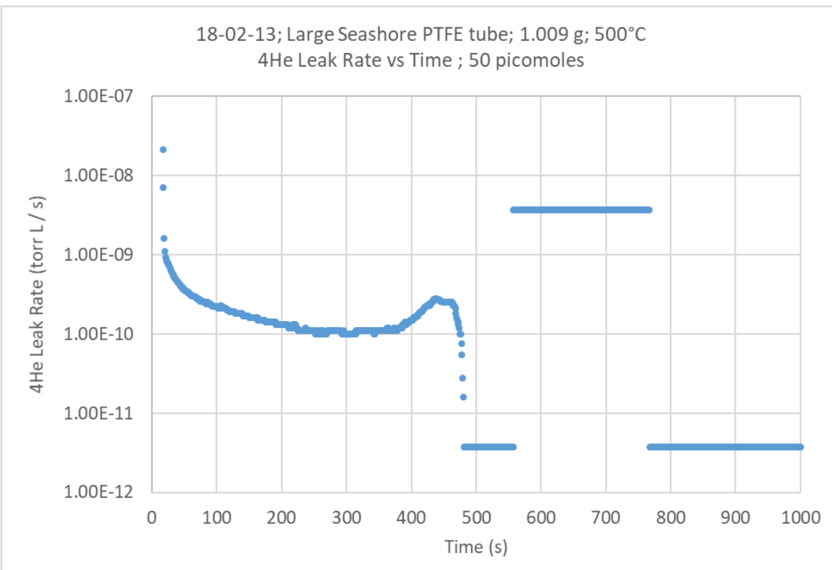
04-19-2018; (4) Nylon Nuts; 0.807 g; 400 °C
4He Leak Rate vs Time - 0.8 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
1000
2000
3000
4000
5000
6000
7000
8000
Time (s)

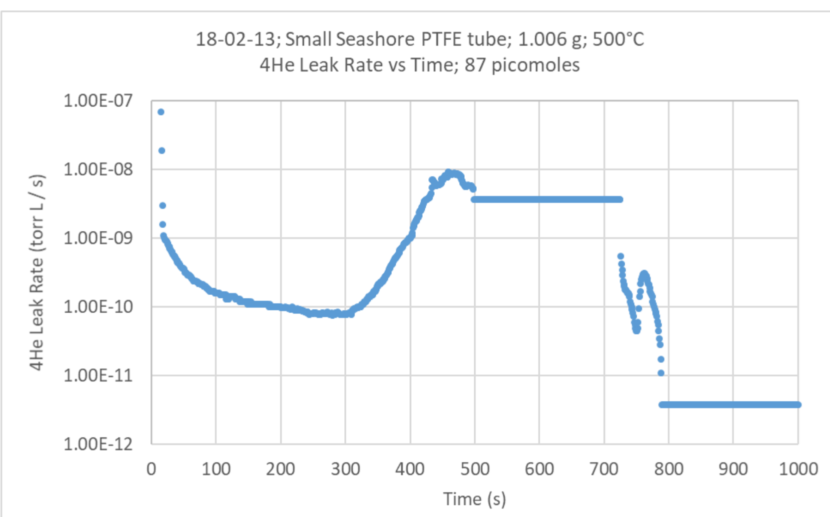


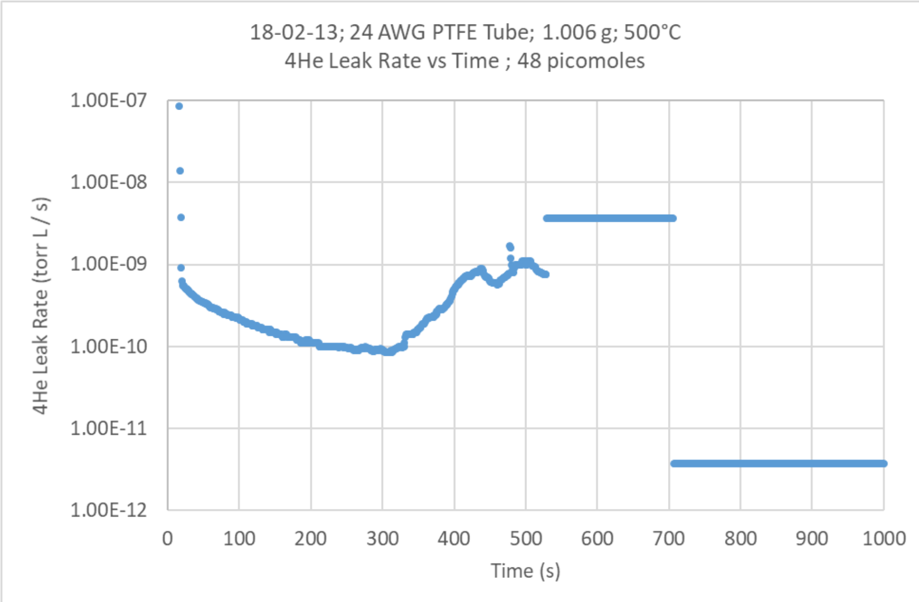
04-19-18; Nylon Screws; 0.921 g; 400 °C
4He Leak Rate vs Time- 0.44 picomoles
4He Leak Rate (torr L / s)
0.0001
0.00001
0.000001
0.0000001
1E-08
1E-09
1E-10
1E-11
1E-12
0
1000
2000
3000
4000
5000
Time (s)

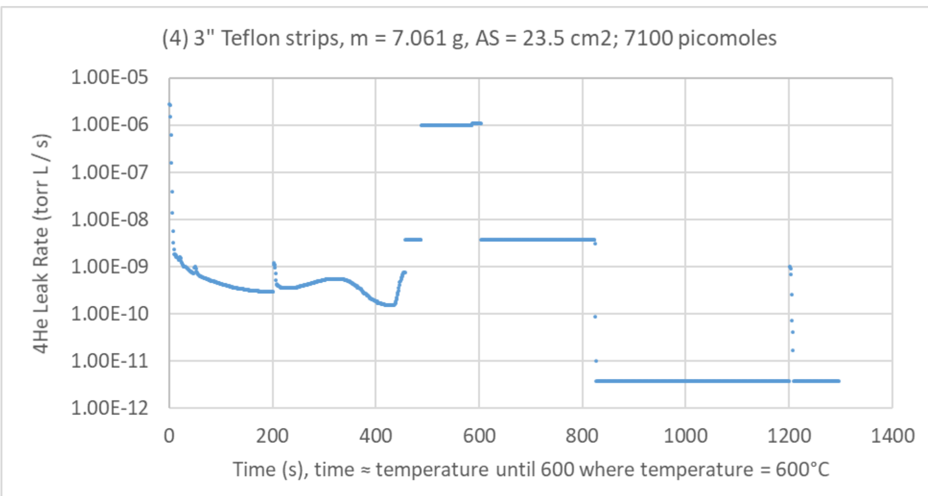
Teflon

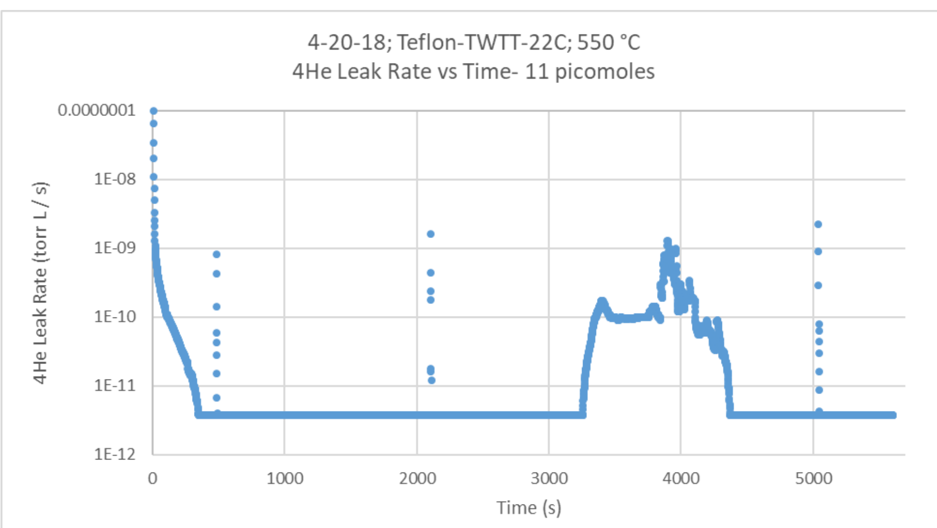

18-02-01; Seashore's PTFE @ 450°C; 1.25g
4He Leak Rate (torr L / s)
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
5000
10000
15000
20000
25000
30000
Time (s)

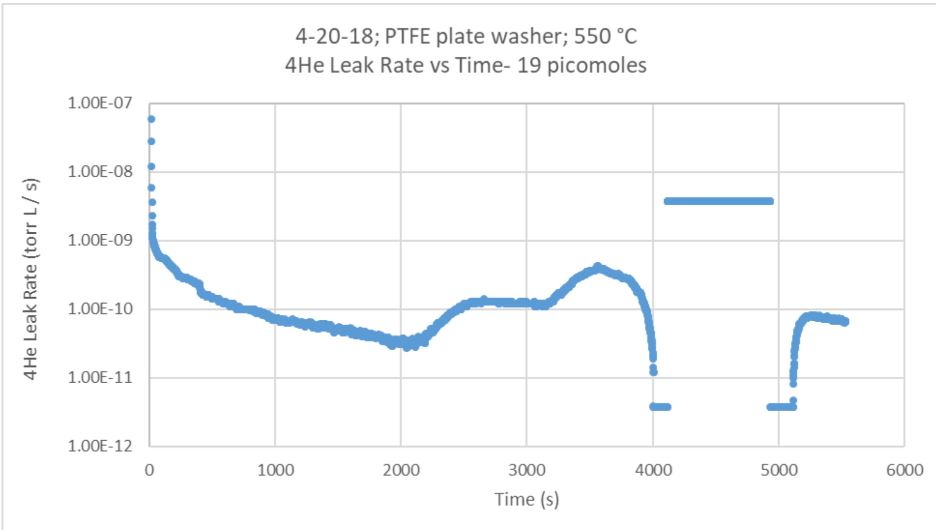

18-02-13; Large Seashore PTFE tube; 1.009 g; 500°C
4He Leak Rate vs Time ; 50 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
100
200
300
400
500
600
700
800
900
1000
Time (s)


18-02-13; Small Seashore PTFE tube; 1.006 g; 500°C
4He Leak Rate vs Time; 87 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
100
200
300
400
500
600
700
800
900
1000
Time (s)


18-02-13; 24 AWG PTFE Tube; 1.006 g; 500°C
4He Leak Rate vs Time ; 48 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
100
200
300
400
500
600
700
800
900
1000
Time (s)


(4) 3" Teflon strips, m = 7.061 g, AS = 23.5 cm2; 7100 picomoles
4He Leak Rate (torr L / s)
1.00E-05
1.00E-06
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
200
400
600
800
1000
1200
1400
Time (s), time ≈ temperature until 600 where temperature = 600°C


4-20-18; Teflon-TWTT-22C; 550 °C
4He Leak Rate vs Time- 11 picomoles
4He Leak Rate (torr L / s)
0.0000001
1E-08
1E-09
1E-10
1E-11
1E-12
0
1000
2000
3000
4000
5000
Time (s)


4-20-18; PTFE plate washer; 550 °C
4He Leak Rate vs Time- 19 picomoles
4He Leak Rate (torr L / s)
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
0
1000
2000
3000
4000
5000
6000
Time (s)