

⁴He Outgassing Technical Report

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

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

Integrating ⁴He 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 summarizes the ⁴He 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	⁴ He Out Gassing (picomole)	⁴ He outgassing / cm ²	⁴ He outgassing / cm ³	Out Gassing Temperature (°C)
SS 316	Swagelok	12-11-17-3/8-SS	43	0.29	0.76	~ 400 °C from notebook
SS 316	Swagelok	02-12-18 -3/8-SS	30	0.2	0.52	
SS 316	Swagelok	01-26-18-1/2-SS	240	1.2	2.2	
SS 316	Swagelok	12-11-17-3/8-SS	39	0.27	0.69	
SS 316	Swagelok	12-13-17-3/8-SS	64	0.44	1.1	
SS 316	Swagelok	03-23-18-1/4-SS	7.8	0.09	0.4	
SS 316	Swagelok	8-001, ½-SS316	64	0.31	0.57	400
SS 316	Swagelok	8-002, ½-SS316	92	0.45	0.82	---

SS 316	Swagelok	8-004, ½-SS316	58	0.28	0.51	386
SS 316	Swagelok	8-005, ½-SS316	36	0.18	0.32	386
SS 316	Swagelok	8-006, ½-SS316	15	0.07	0.13	427
SS 316	Swagelok	8-007, ½-SS316	92	0.44	0.81	427

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 ^4He outgassing rates. Table 1.1 contains the resulted ^4He 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	Vendor	Sample Status	He Out Gassing (picomoles)	^4He outgassing / cm ²	^4He outgassing / cm ³	Out Gassing Temperature (°C)
SS 316	Swagelok	Pre-baked and He soaked for 24 hours	16.12	0.1100	0.2850	~400
SS 316	Swagelok	Pre-baked and kept in atmosphere for 24 hours	0.52	0.0036	0.0092	
SS 316	Swagelok	Pre-baked and kept in atmosphere for 15 days	18.20	0.1250	0.3220	
SS 316	Swagelok	Pre-baked and kept in atmosphere for 27 days	0.88	0.0060	0.0160	

When performing ^4He 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 °C was used as the sample holder. Table 1.2 summarizes the total number of picomoles of ^4He 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 ^4He 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	1.006 g	500	87	86	
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
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- PTFE melting temperature: 327 °C
- Nylon melting temperature: 220 °C; decomposition temperature: 307 °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", 1/2") 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

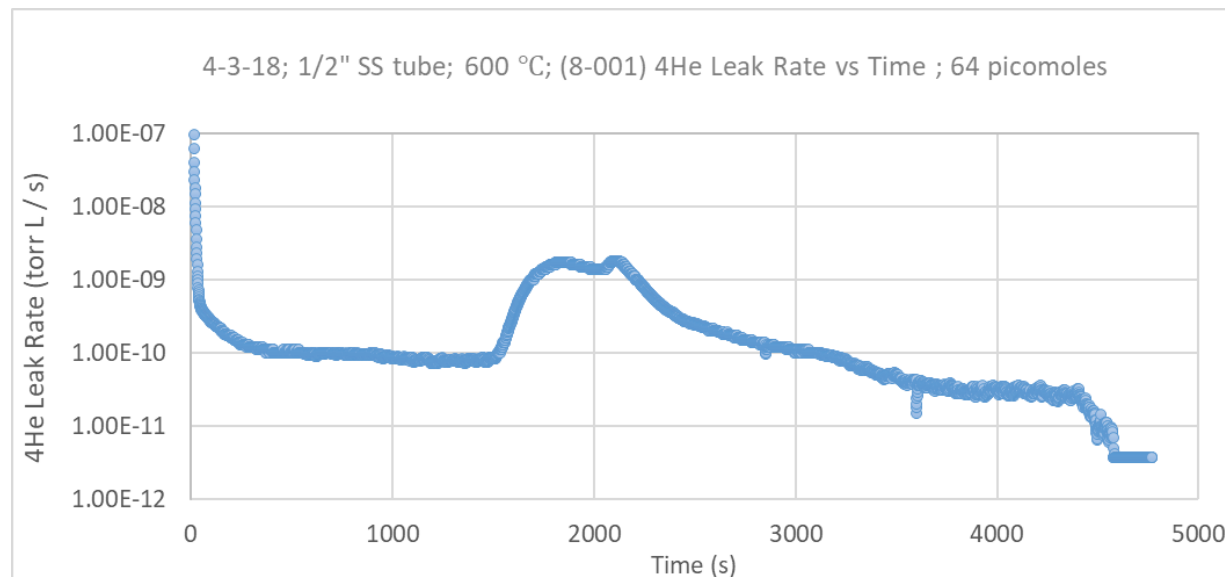
outgassed under increasing and decreasing temperature from these materials. A one end fused stainless steel tube is used to hold the sample inside the furnace. First the stainless-steel tube is placed inside the furnace and is connected to the ASM 340 leak detector. Then the stainless-steel tube is baked 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:

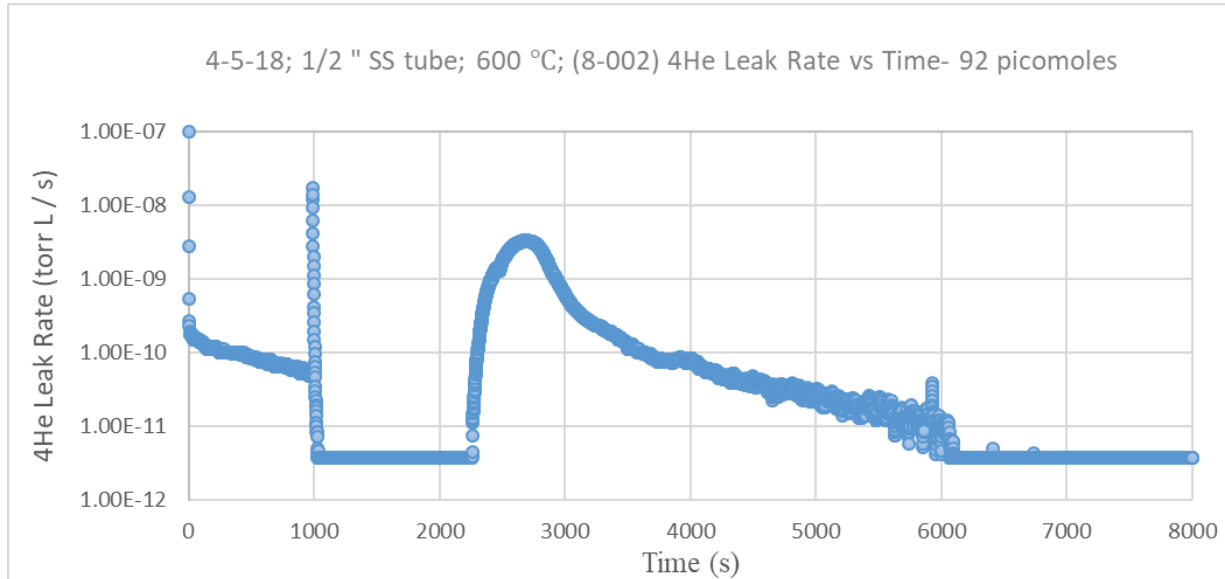
Some recent improvements to this experiment are connecting thermocouple to the tubing samples and using a LABVIEW program to record exact temperature at a given time during the experiment and modifying the furnace cap to hold up to three samples at the same time.

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.

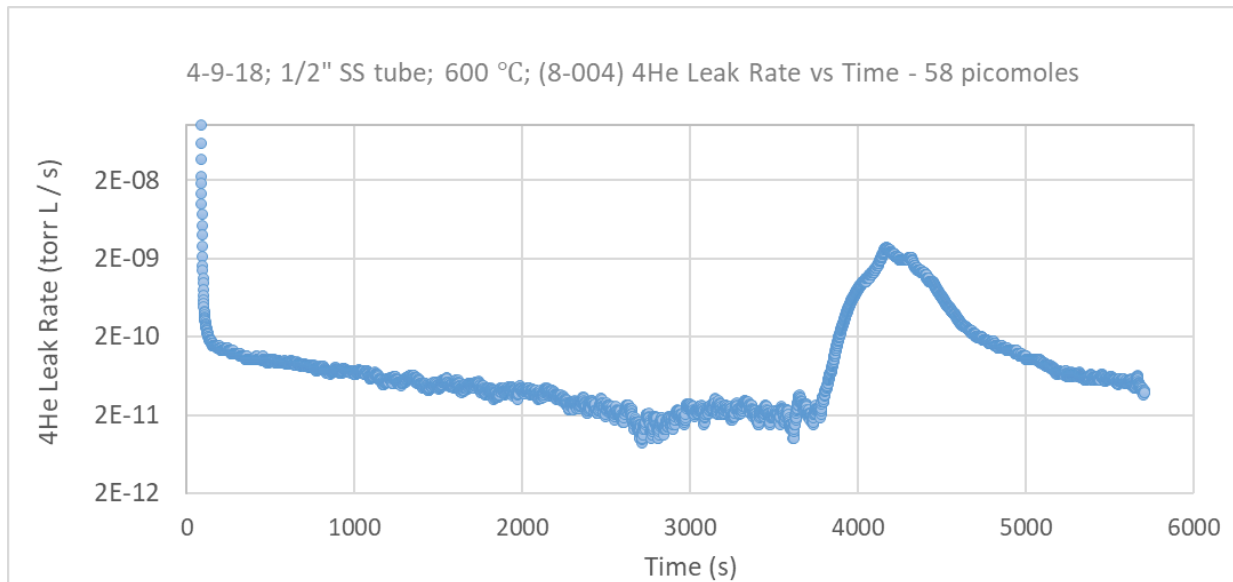
½” SS316 tubing, 001, Swagelok



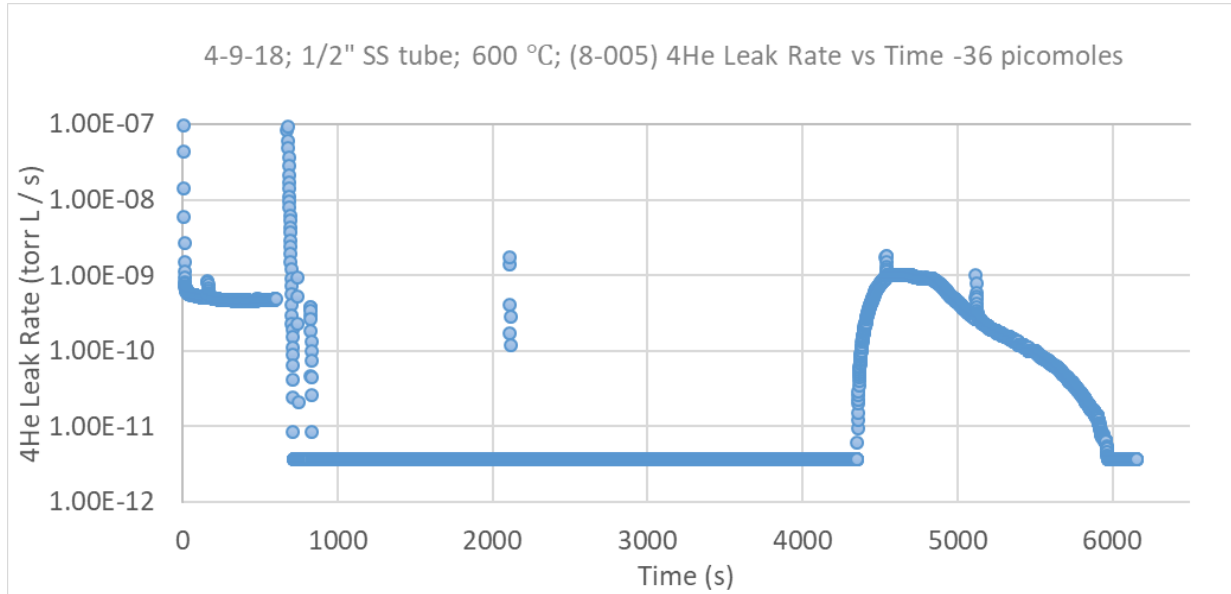
½" SS316 tubing, 002, Swagelok



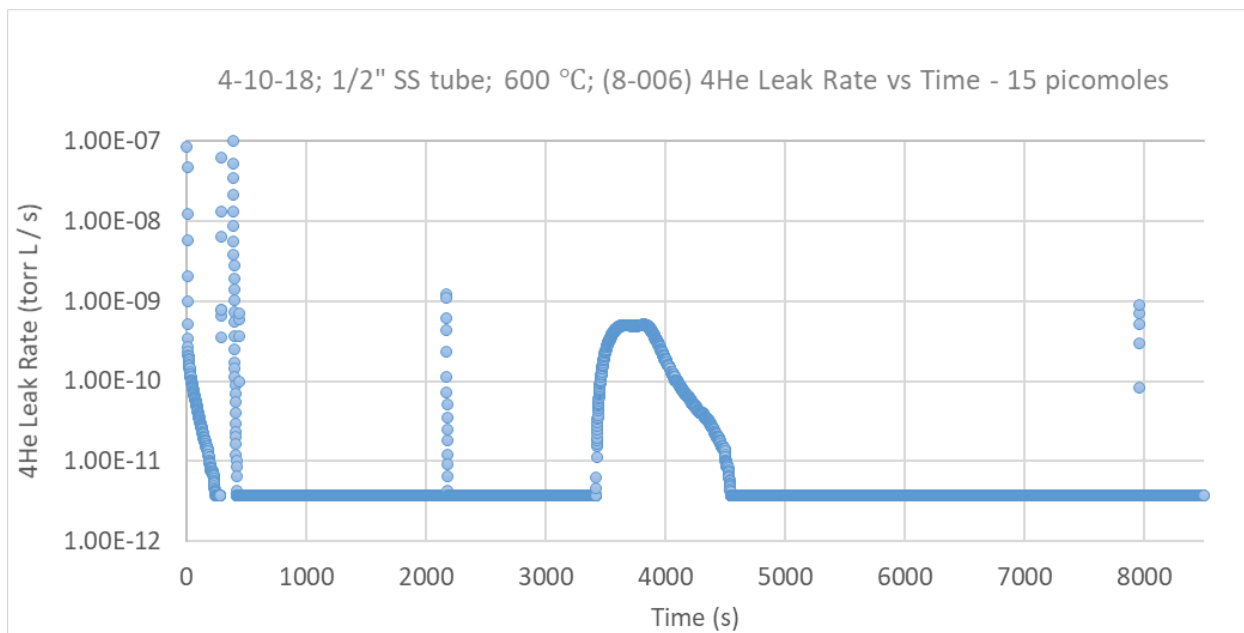
½" SS316 tubing, 004, Swagelok



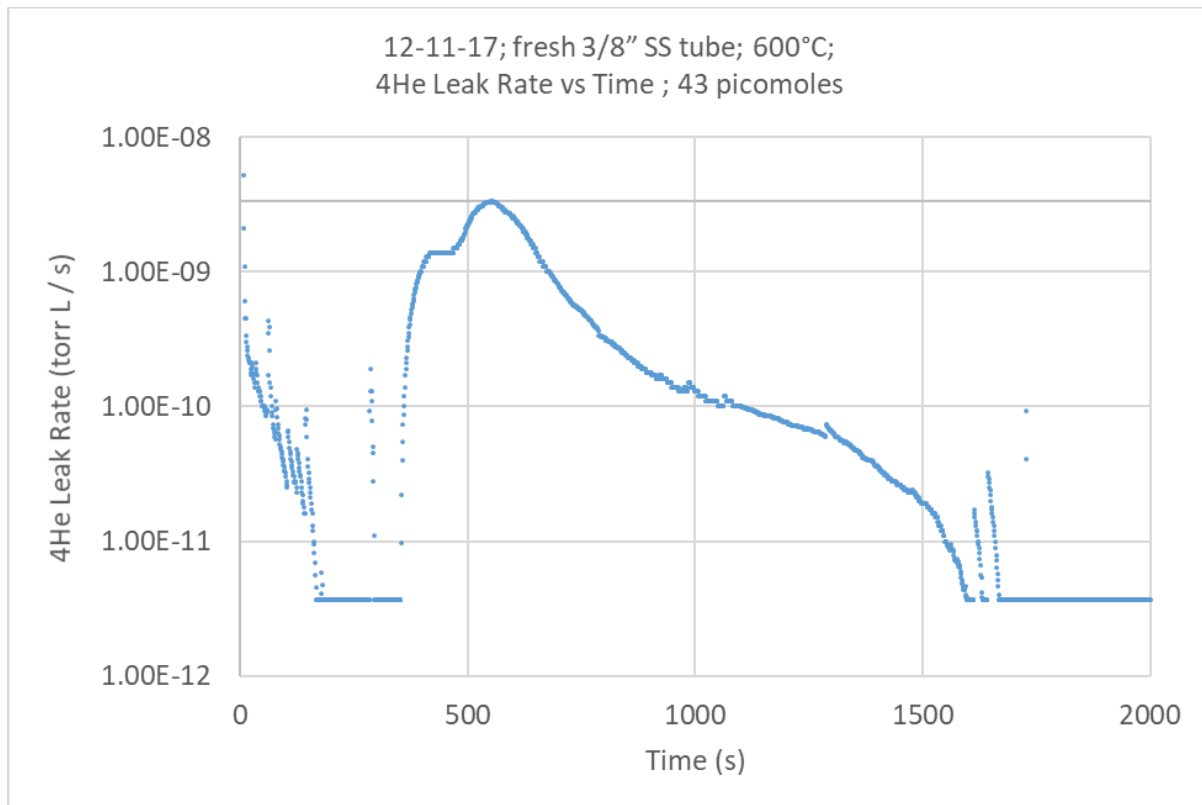
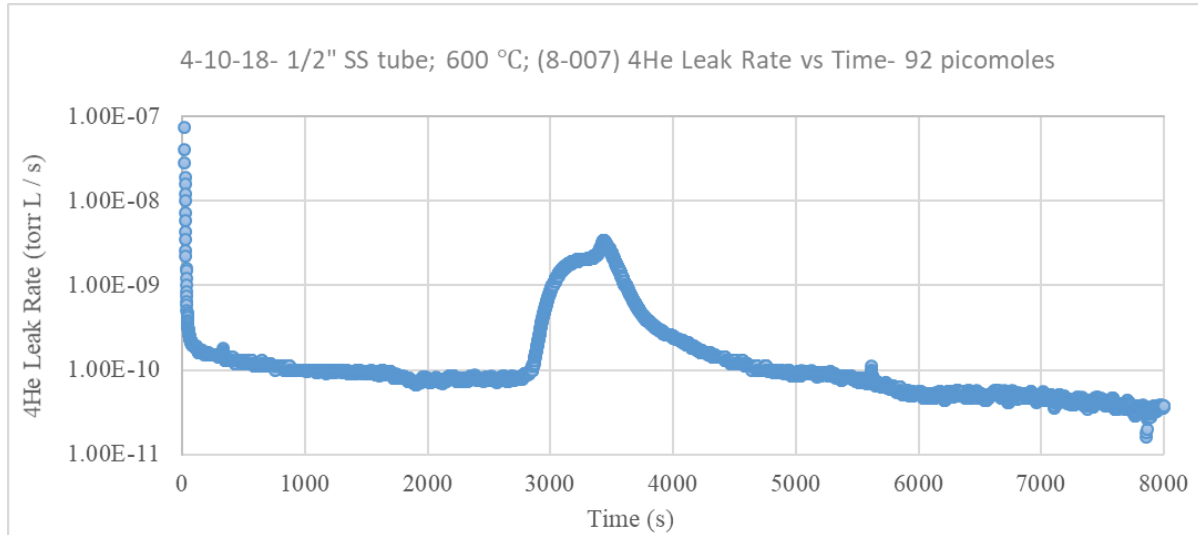
½" SS316 tubing, 005, Swagelok

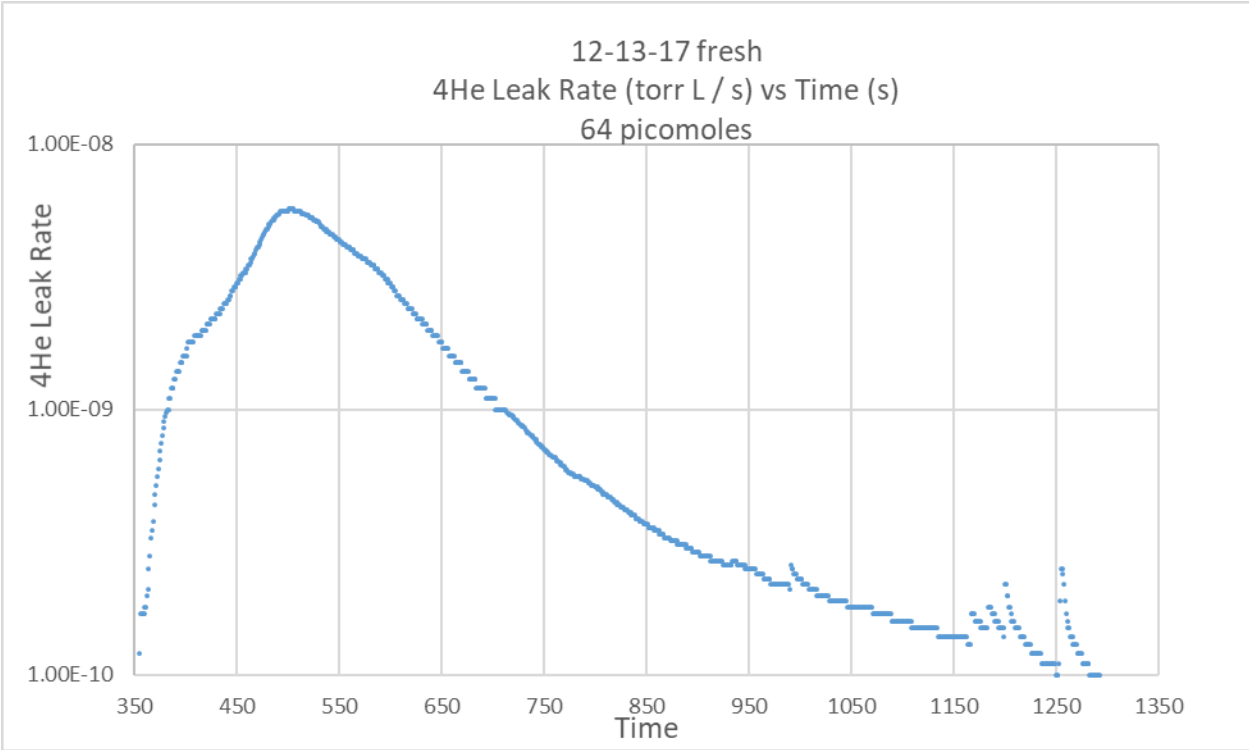
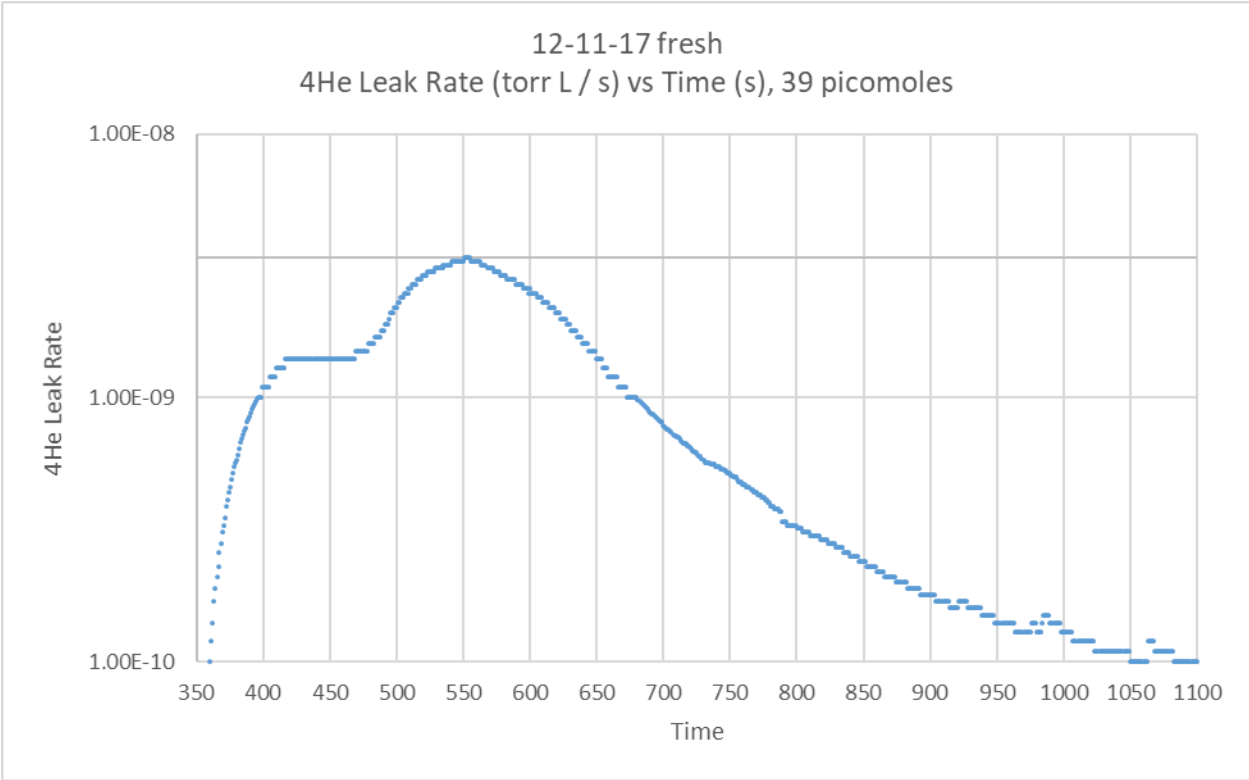


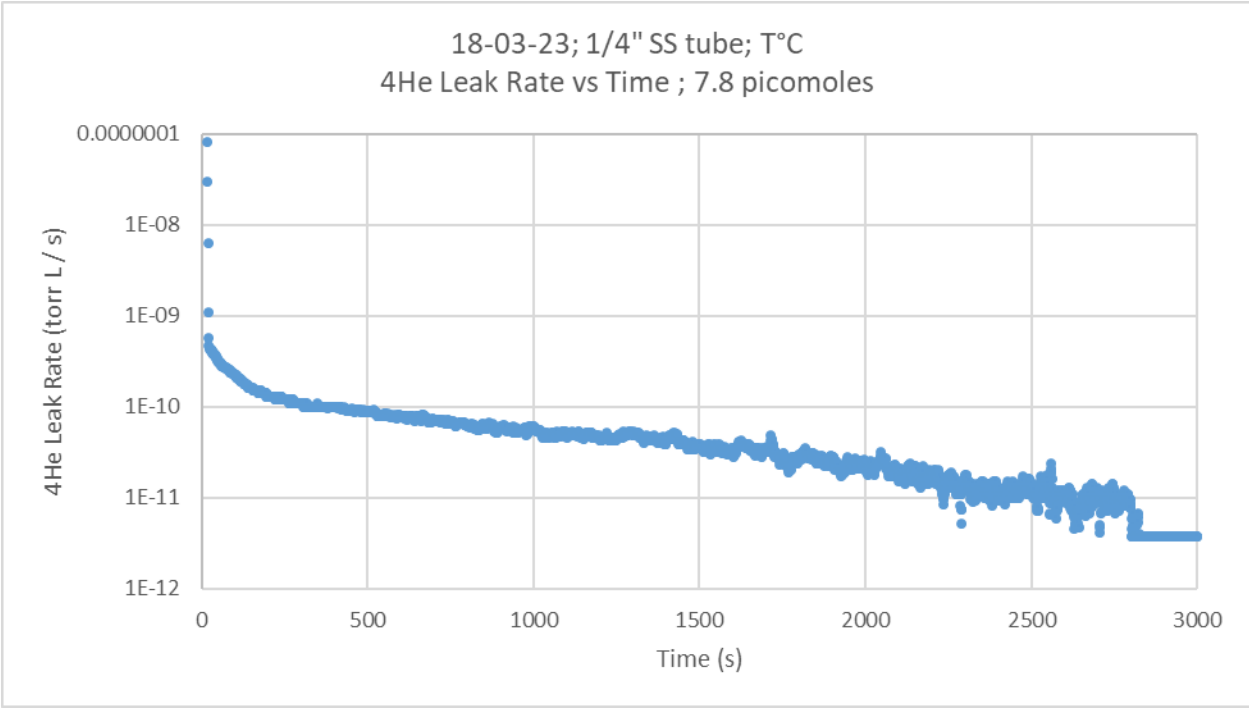
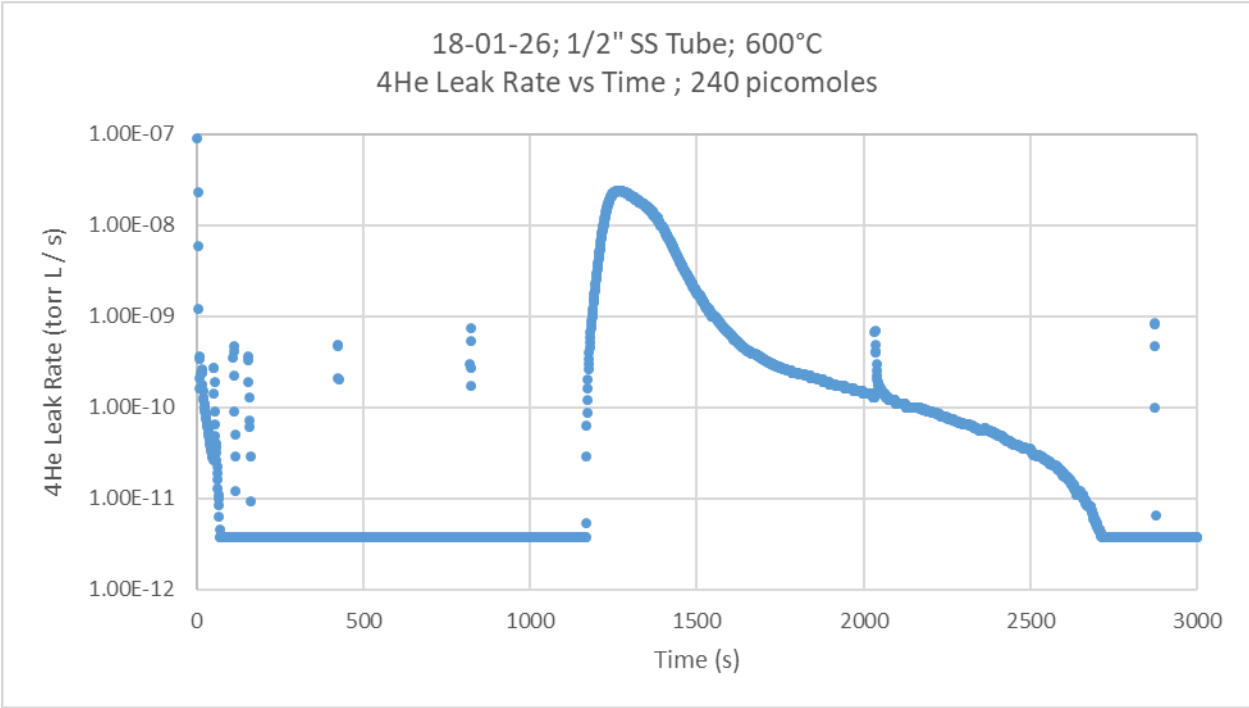
½" SS316 tubing, 006, Swagelok



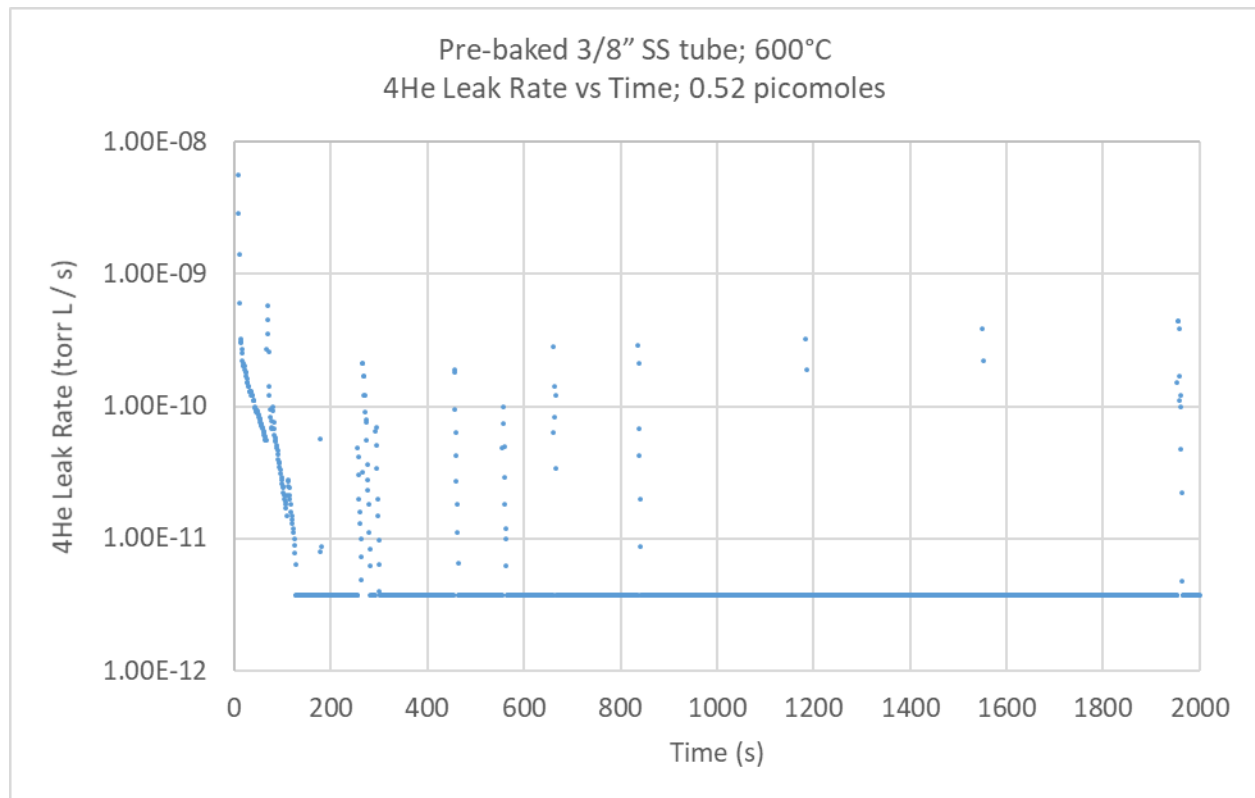
½" SS316 tubing, 007, Swagelok

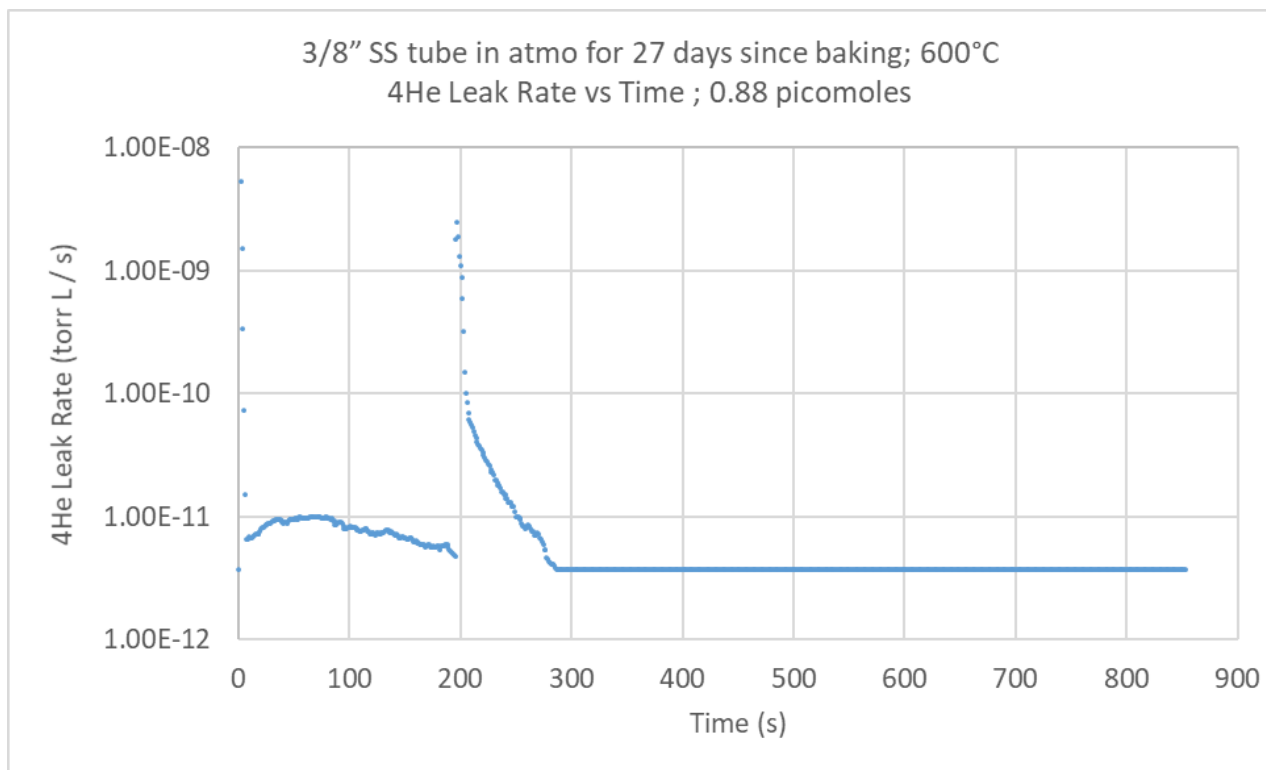
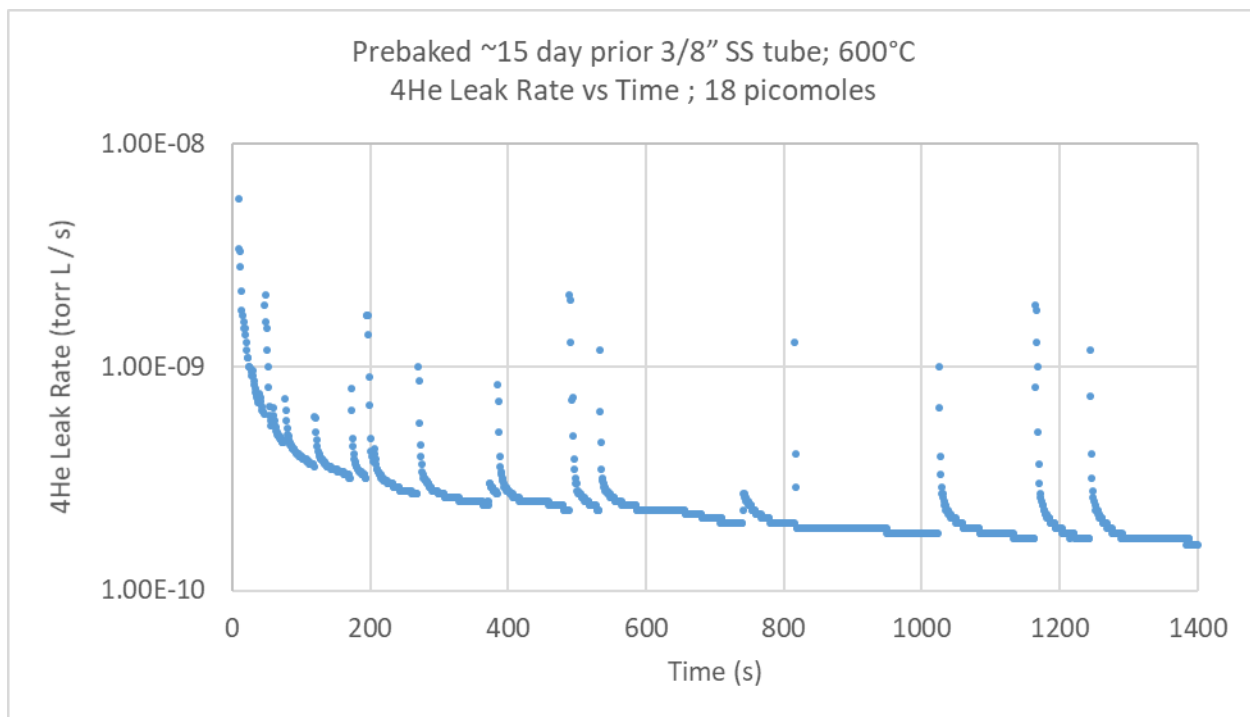


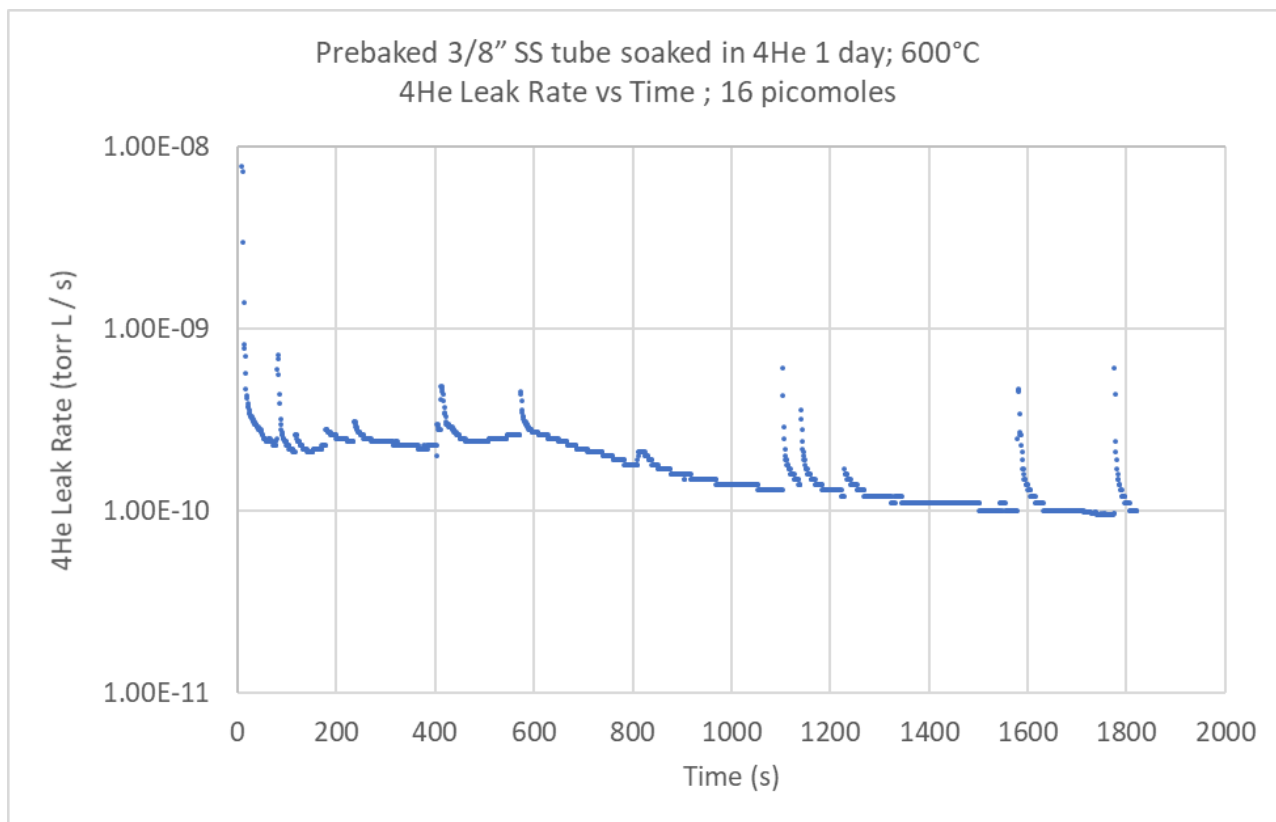




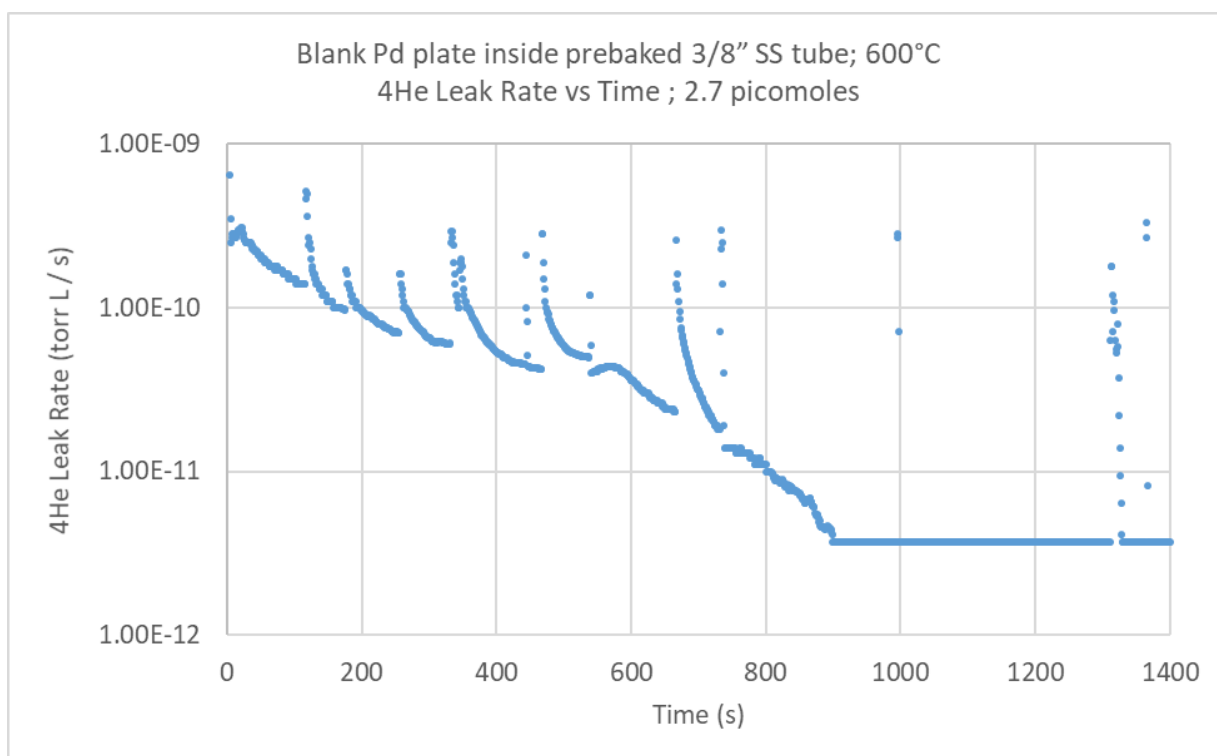
Pre-baked Stainless-Steel tubing

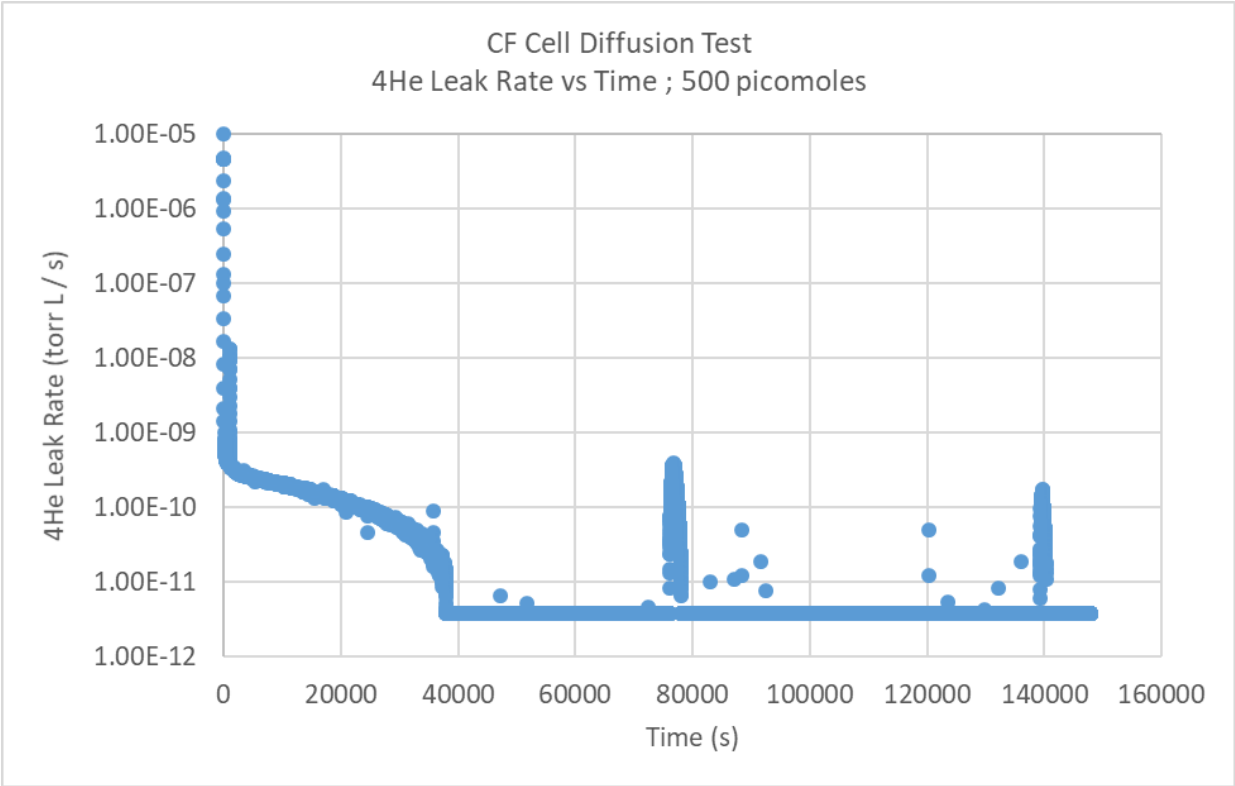




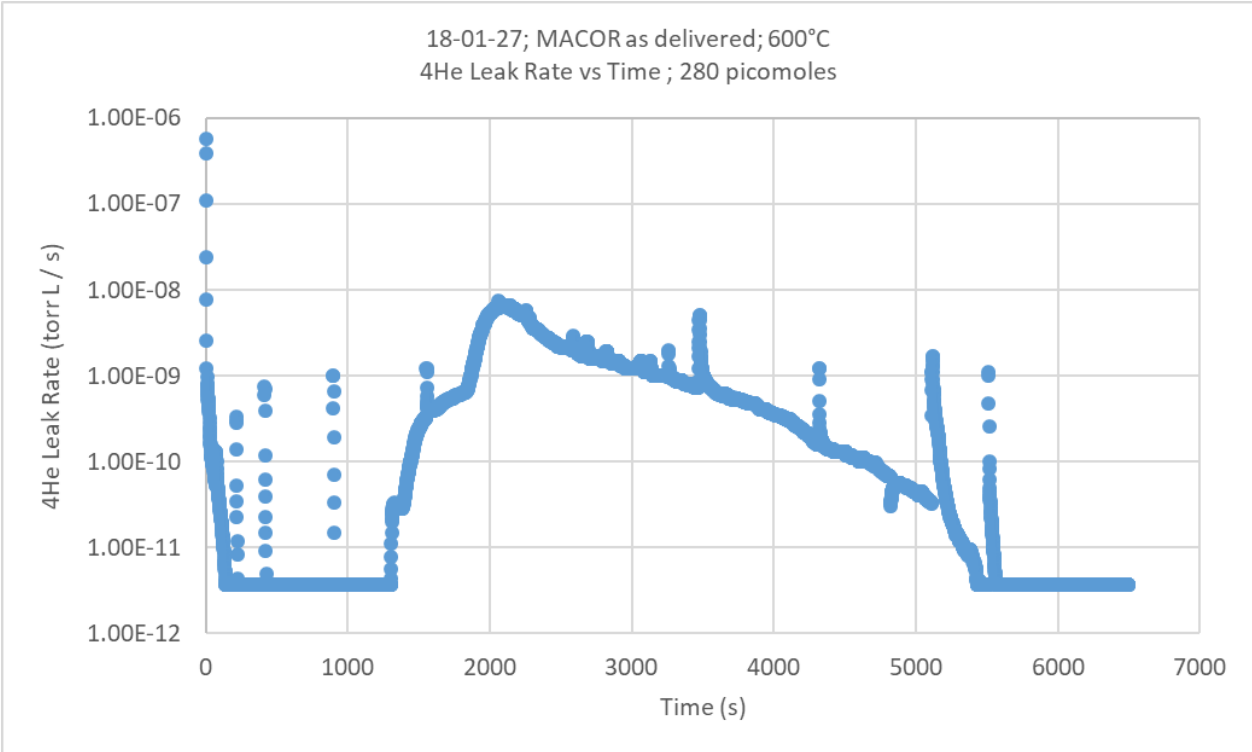


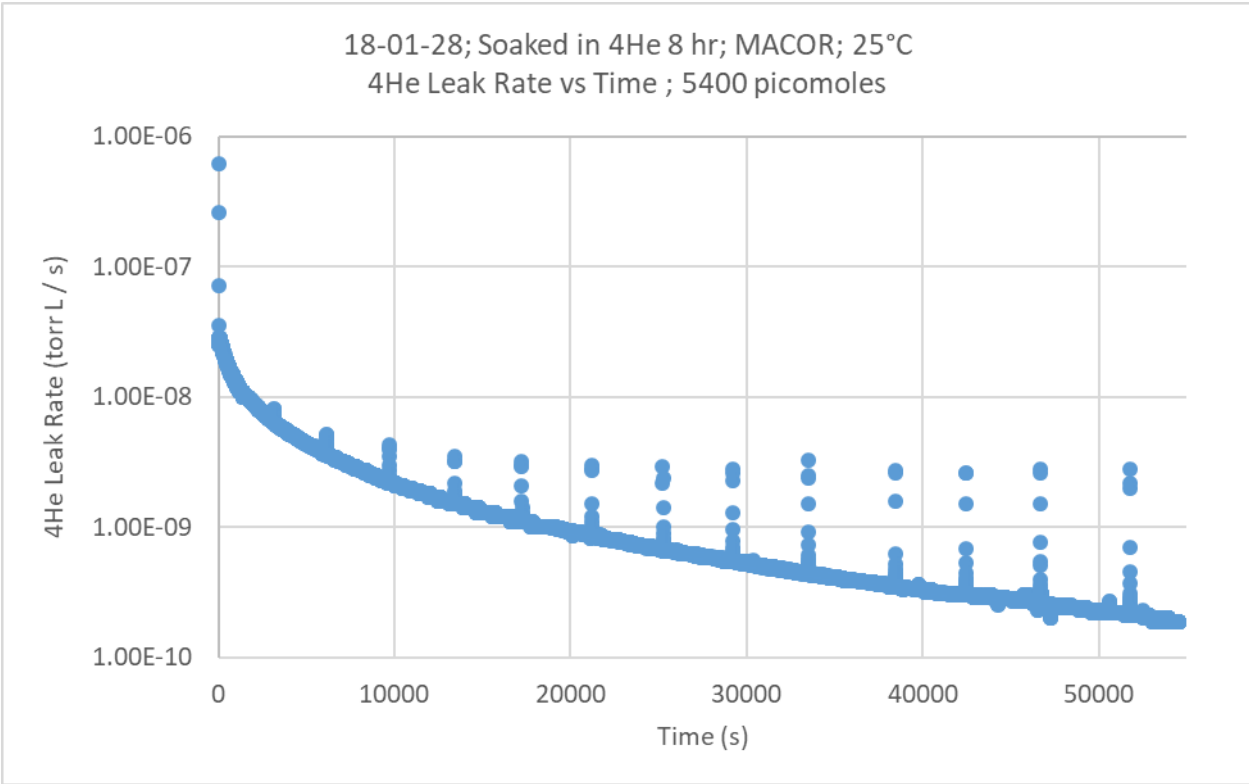
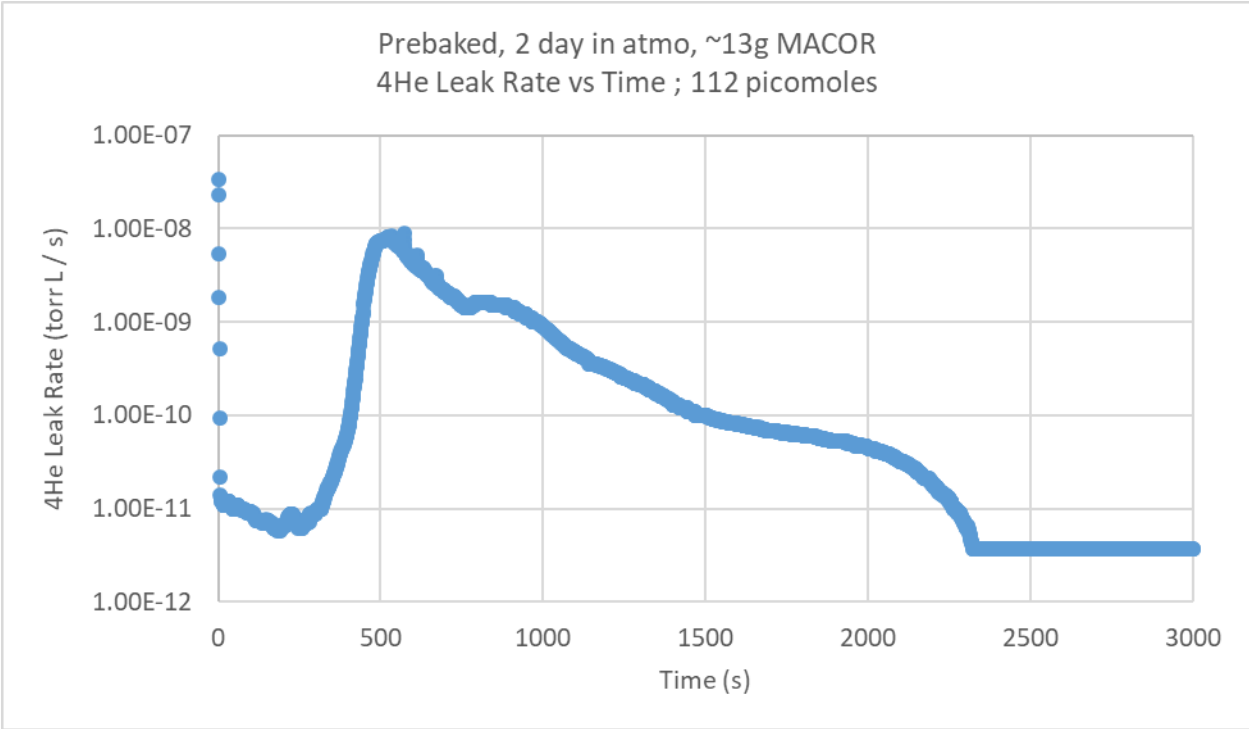
Other Materials:

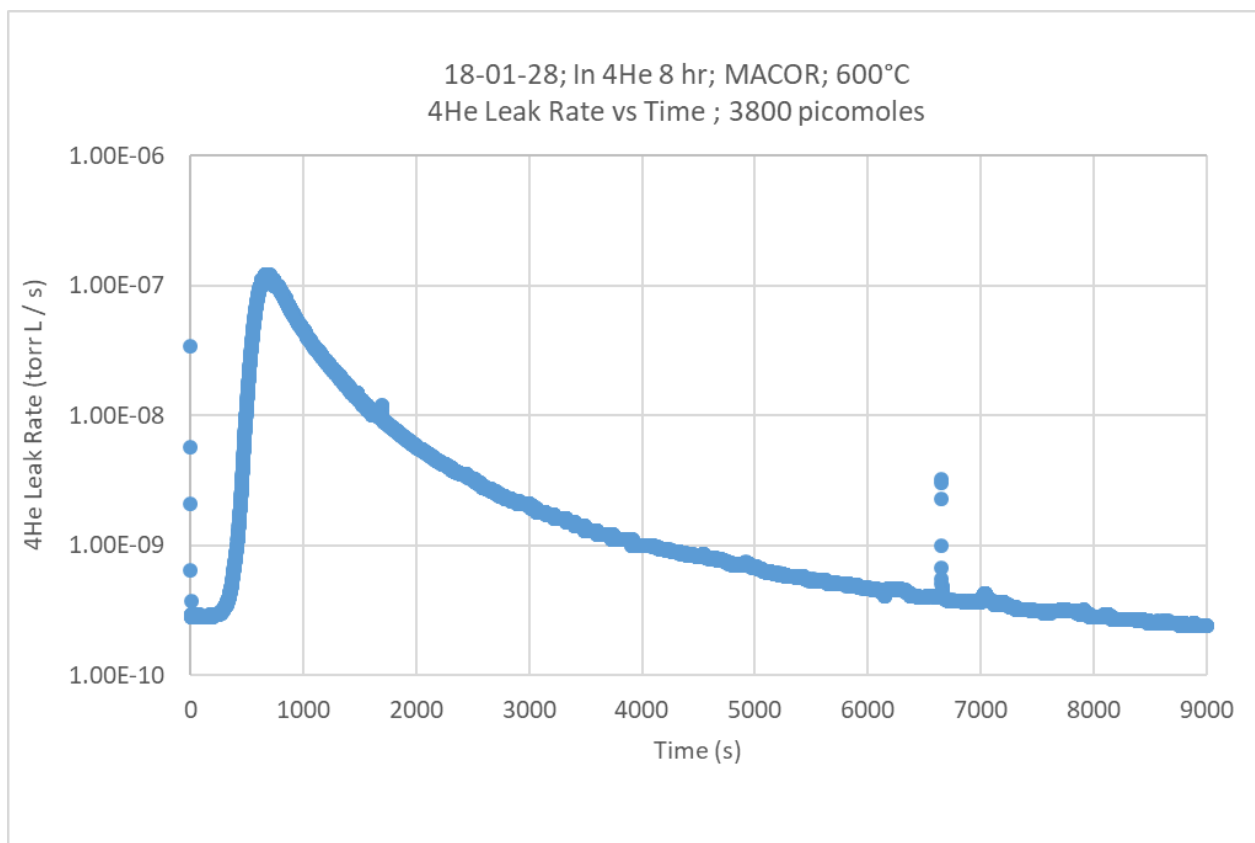




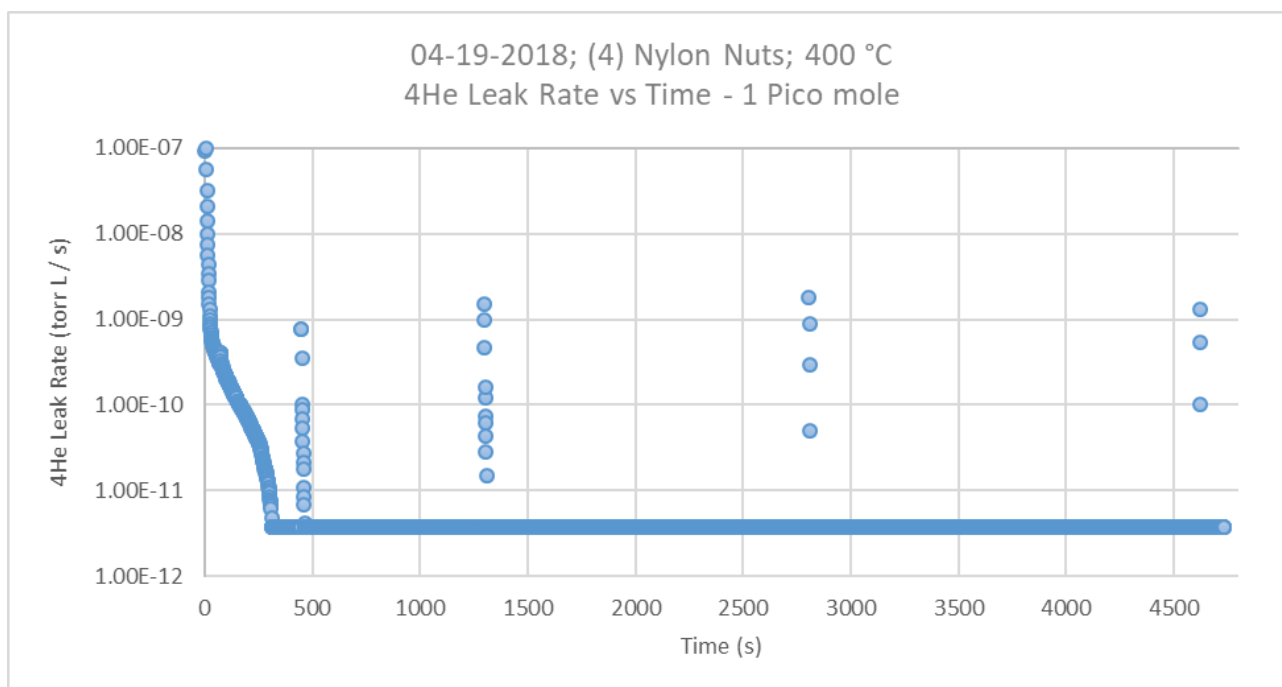
MACOR

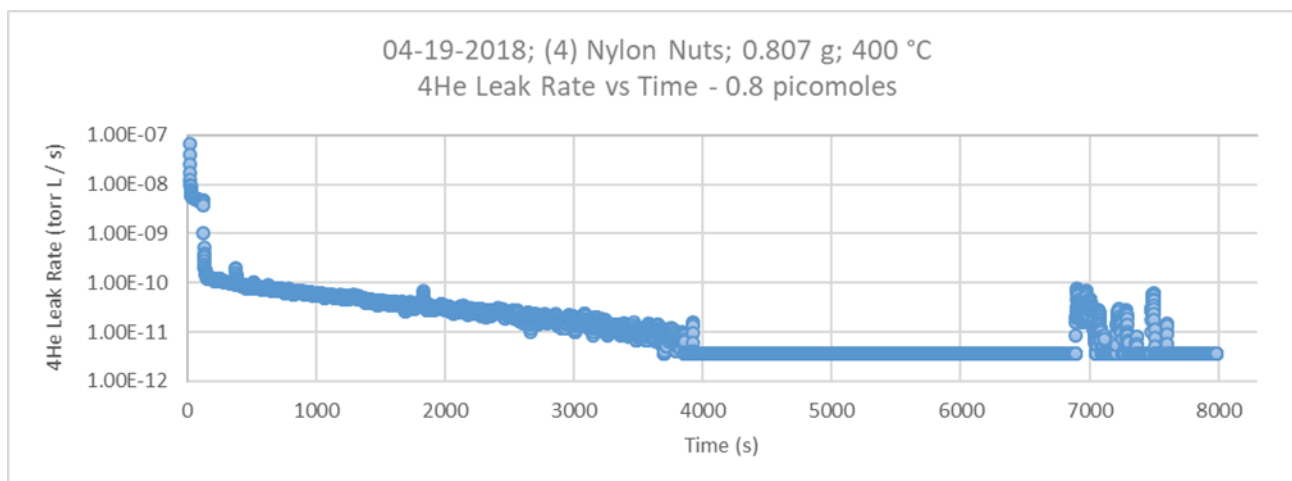
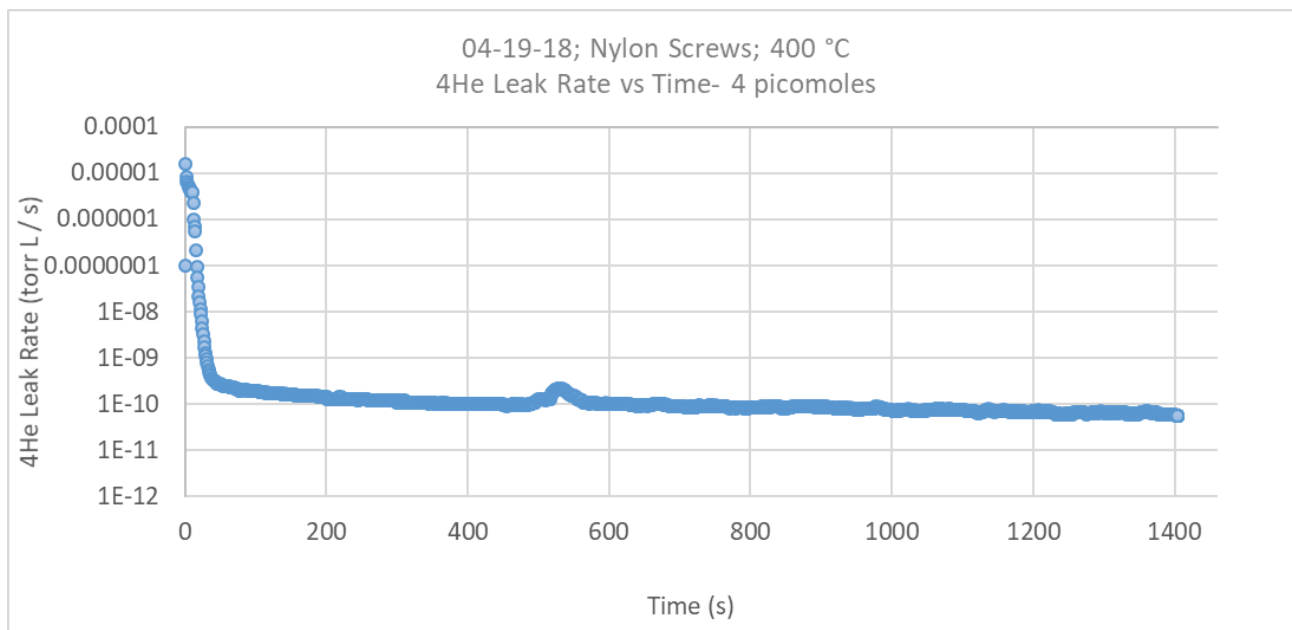


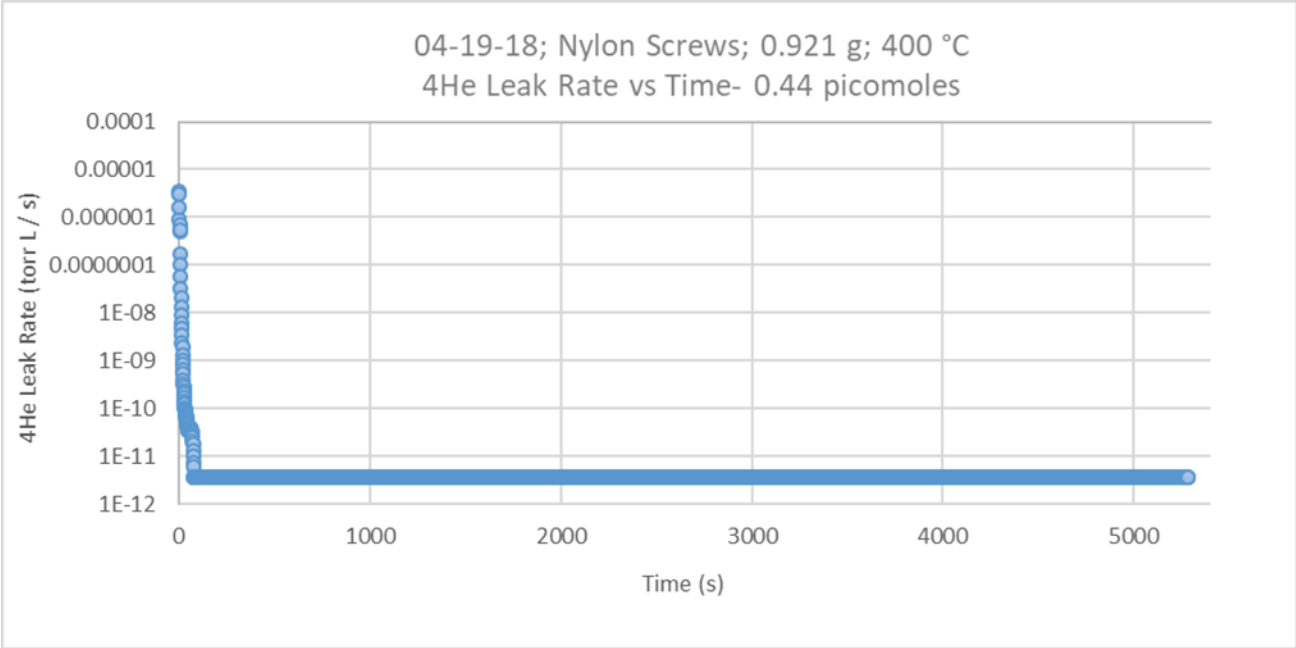




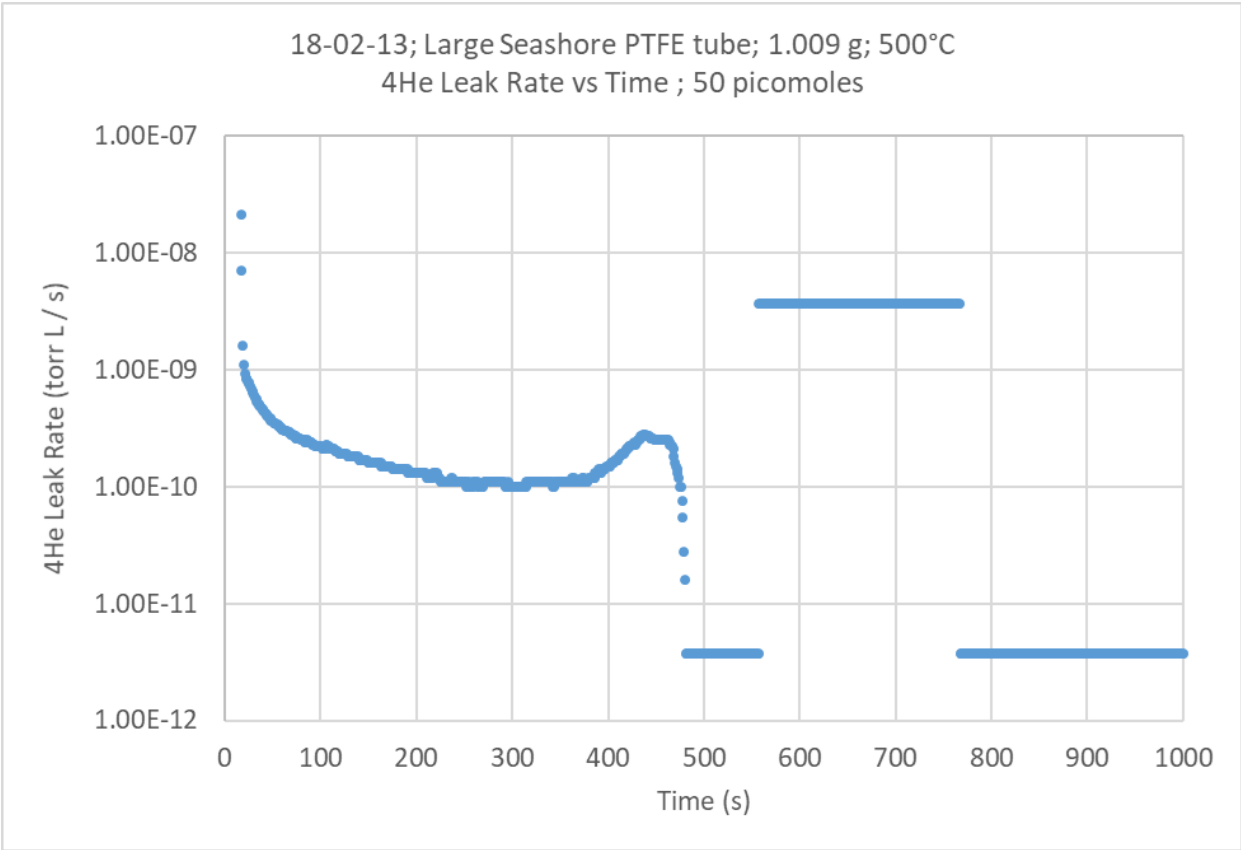
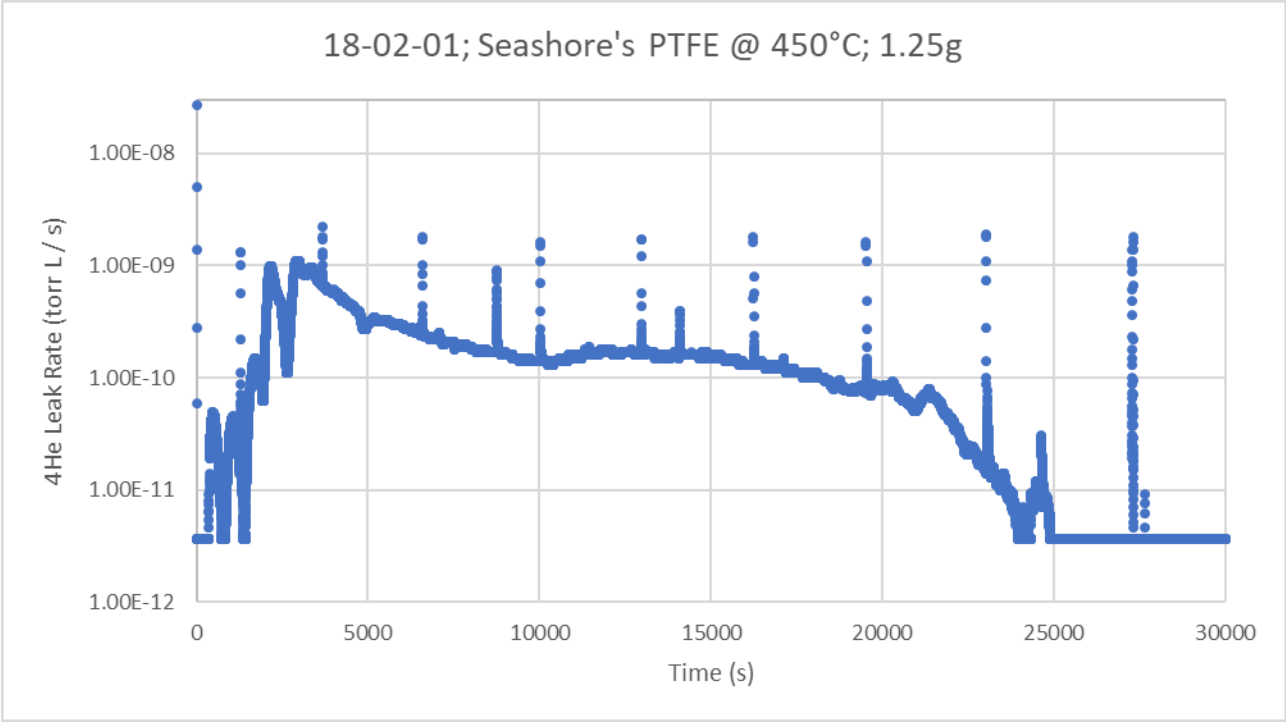
Nylon



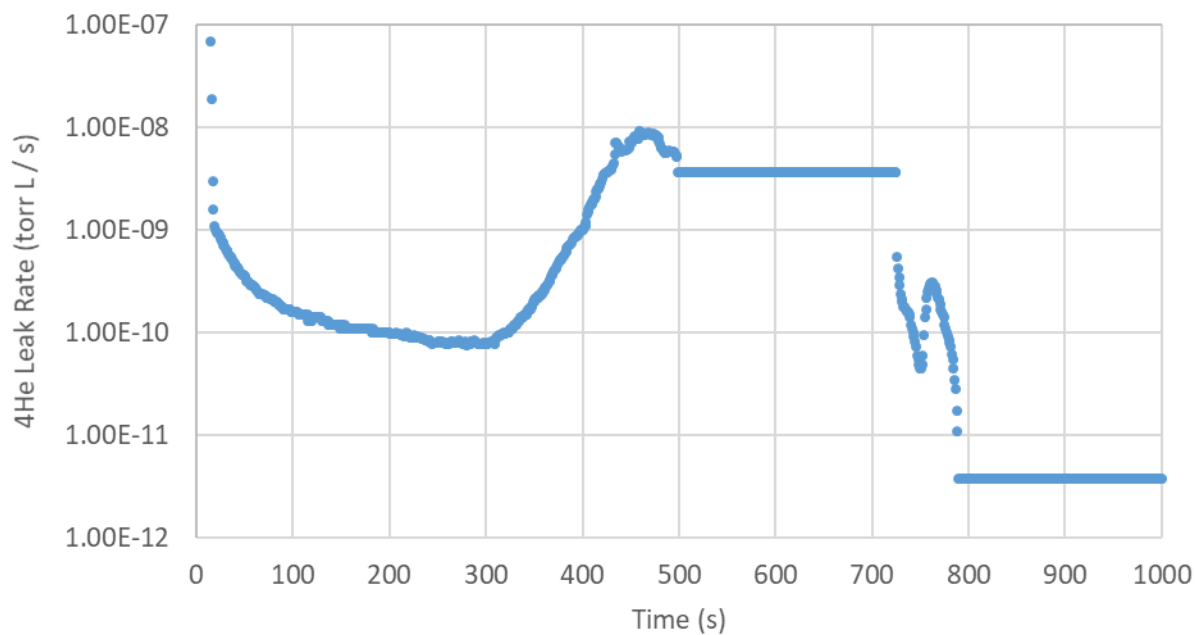




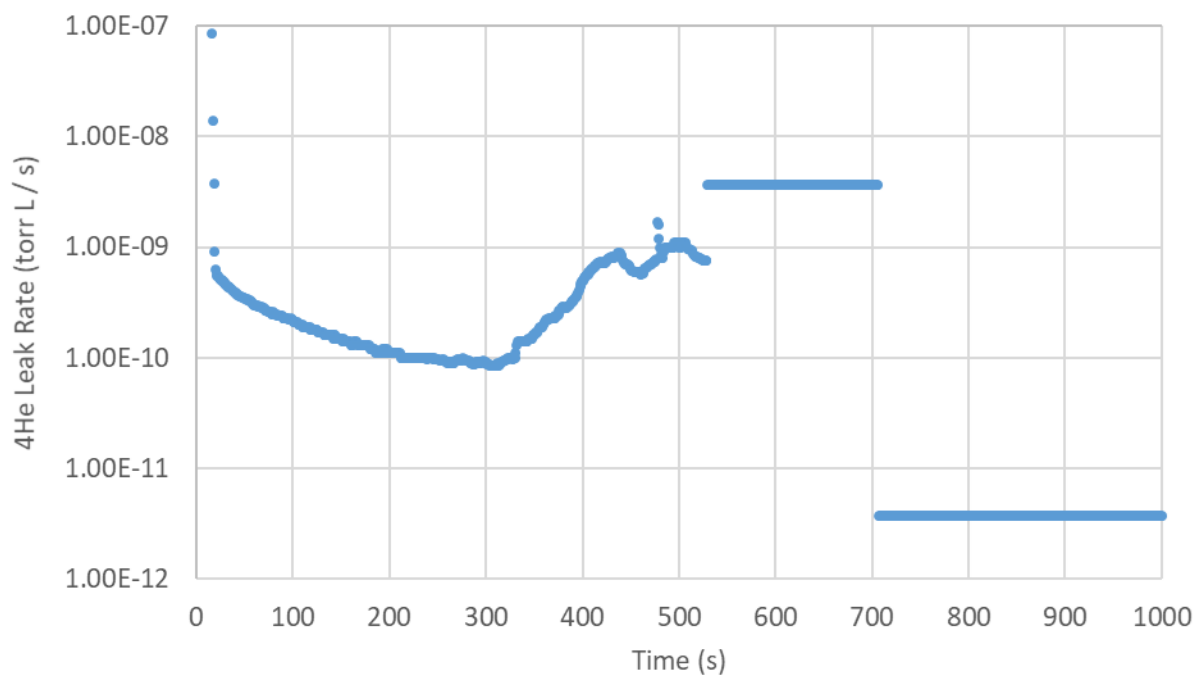
Teflon



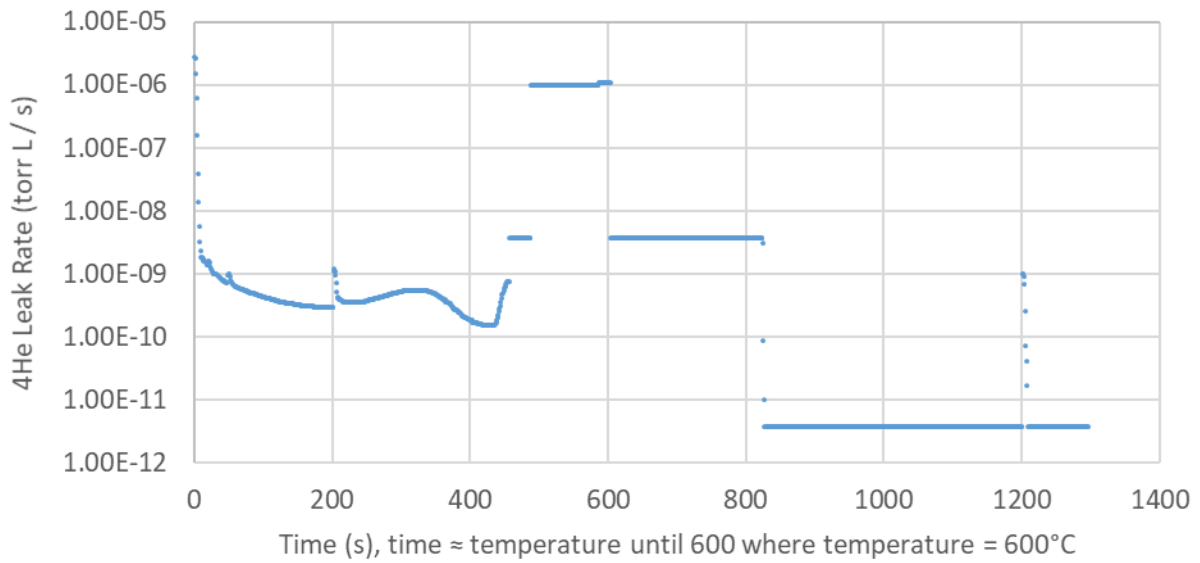
18-02-13; Small Seashore PTFE tube; 1.006 g; 500°C
4He Leak Rate vs Time; 87 picomoles



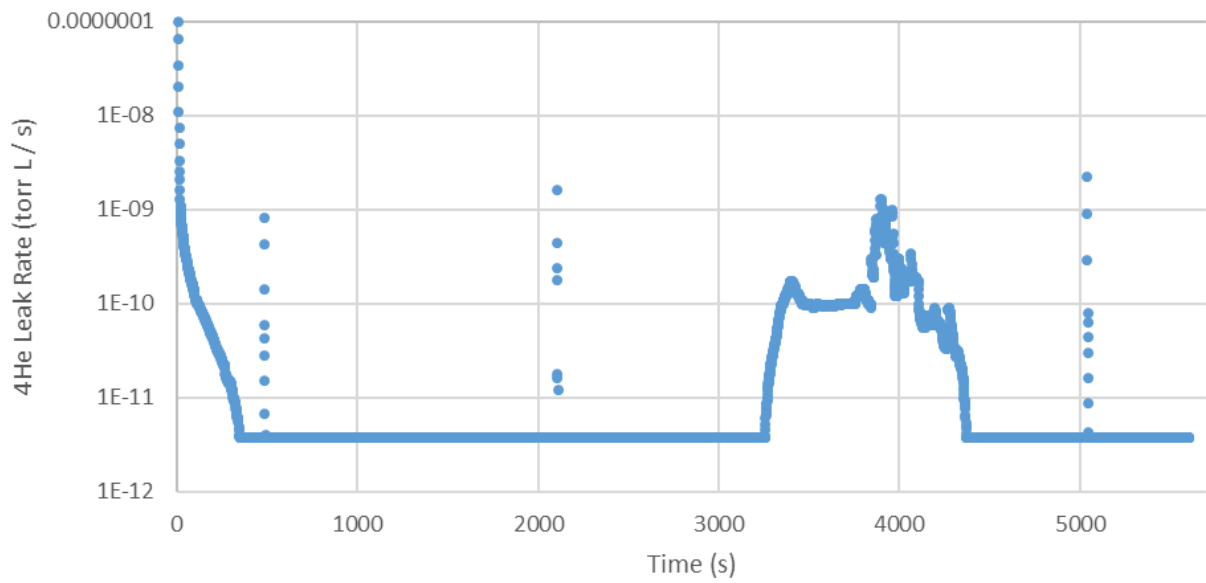
18-02-13; 24 AWG PTFE Tube; 1.006 g; 500°C
4He Leak Rate vs Time ; 48 picomoles



(4) 3" Teflon strips, m = 7.061 g, AS = 23.5 cm²; 7100 picomoles



4-20-18; Teflon-TWTT-22C; 550 °C
4He Leak Rate vs Time- 11 picomoles



4-20-18; PTFE plate washer; 550 °C

4He Leak Rate vs Time- 19 picomoles

