

NEW LEAK RATE MEASUREMENTS OF FLIGHT-LIKE MARS 2020 SAMPLE TUBES: INSIGHTS FOR POTENTIAL MARS SAMPLE RETURN. J. T. Osterhout¹, K. A. Farley^{1,2}, M. Wadhwa^{1,3}, J. Treffkorn², and E. Kulczycki¹. ¹Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA (jeffrey.t.osterhout@jpl.nasa.gov); ²California Institute of Technology, Pasadena, CA; ³Arizona State University, Tempe, AZ.

Introduction: NASA's Mars 2020 rover, *Perseverance*, is currently collecting samples on Mars in preparation for the planned Mars Sample Return campaign [1]. The sample tubes are designed to contain rocks, regolith, and atmospheric gases, and are hermetically sealed on the surface of Mars to minimize sample loss, alteration, and contamination [2]. Following a robust testing program, it was determined that the helium (He) leak rates of flight-like sample tubes sealed under a range of conditions were typically no greater than $\sim 10^{-10}$ standard cubic centimeters per second (scc/s) [3]. Leak rates below this value could not be measured since this is the detection limit of commercially available He leak detectors. This limit satisfies mission requirements, but concerns remain about potential sample loss, contamination, and chemical alteration (e.g., isotopic fractionation) for He leak rates at or below 10^{-10} scc/s, thus requiring a more sensitive technique for establishing leak rates.

Materials and Methods: This study investigated six flight-like sample tubes that varied in terms of their tube wall thickness, diametric tooth interference (i.e., tooth-wall contact), and sealing temperature. The He leak rate was determined for each sample tube using the experimental set up shown in Fig. 1 that resulted in lowering the detection limit by many orders of magnitude compared to commercial He leak detectors.

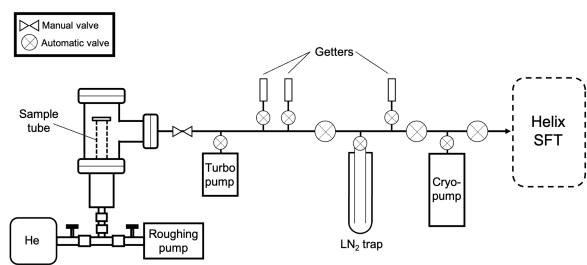


Figure 1. Schematic diagram of the experimental setup.

Each sample tube was filled with ~ 1 bar of He, and gas that leaked through the tube seal was accumulated in an evacuated chamber at room temperature for a minimum of ~ 12 h. Following gas purification within a vacuum line, ⁴He amounts were measured using a Thermo Helix SFT mass spectrometer. The He leak

rate for one sample tube (SN282) was analyzed at eight different temperatures (from -51 to $+42$ °C).

Results and Discussion: Helium leak rates of six sample tubes measured at room temperature range from $\sim 9 \times 10^{-17}$ to $\sim 5 \times 10^{-14}$ scc/s. Sample tube 282 was analyzed at multiple temperatures (Fig. 2) and yielded He leak rates correlated with temperature that varied from $\sim 2 \times 10^{-15}$ to $\sim 1 \times 10^{-13}$ scc/s, respectively.

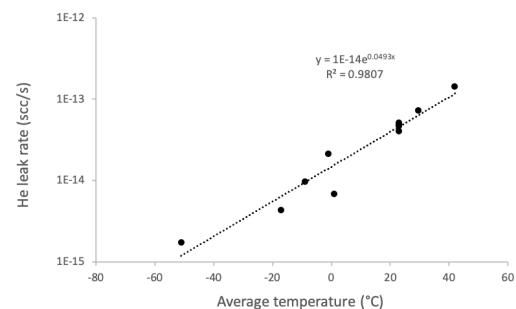


Figure 2. He leak rate data for SN282 plotted against the average temperature during static accumulation of He. Vertical axis is presented on a logarithmic scale.

The main periods of concern for sample loss, contamination, and chemical alteration are during the transit in space from Mars to Earth (when there will be vacuum outside the tubes) and following the return to Earth (when there will be ~ 1 bar of terrestrial atmospheric pressure outside the tubes).

For a He leak rate of 1×10^{-8} scc/s, leakage of He from the inside of the tube to ultra-high vacuum on the outside, over a two-year period, would result in $\sim 95\%$ of the original sample being retained in the sample tube. If the sample tube remains in space for 20 years (e.g., due to a delayed return of the Mars-orbiting sample container), then roughly 40% of the initial sample would be lost. If one assumes a He leak rate of 1×10^{-10} scc/s (the limit verified during sample tube seal development), the amount of sample lost under these conditions decreases significantly, and after 20 years in space $\sim 99.5\%$ of the original sample would still remain. In comparison, the highest He leak rate (i.e., worst-case) observed in this study ($\sim 1 \times 10^{-13}$ scc/s) is three orders of magnitude lower and indicates that $>99.99\%$ of the initial gas in a sample tube would be retained even after 50 years in space.

Once the sample tubes have been returned to Earth, the pressure difference between the inside of the sample tube and the ambient pressure of Earth's atmosphere (~ 1 bar) will generally lead to gas leakage from outside the sample tube to the inside, warranting concerns about potential terrestrial contamination. For an individual sample tube filled with Martian atmosphere, approximately 2.5×10^{-3} cc of N_2 will be contained within a sample tube with a volume of 12.5 cc (and $\delta^{15}N \approx 500\%$) [2,4]. Assuming the worst-case He leak rate measured here ($\sim 1 \times 10^{-13}$ scc/s), the amount of time needed to impart a 1‰ change to the $\delta^{15}N$ of Martian gas through a leak in the seal would be roughly two years.

In comparison to He, other atmospheric gases are expected to have lower leak rates. For example, the leak rate for water vapor (H_2O) is assumed to be lower by a factor of ~ 9 due to differences in the physical properties (e.g., molecular weights) of the two gases [2,3].

Conclusions: Our results suggest that the Mars 2020 sample tube seals are likely to be characterized by very low He leak rates, typically better than 10^{-13} scc/s (Fig. 3). Helium leak rates of the flight-like sample tubes studied here are roughly 3 to 6 orders of magnitude below the previous detection limit of 10^{-10} scc/s. As such, the leakage of gases into or out of the sealed sample tubes (during transit from Mars to Earth or after return to Earth) can be ignored for most practical purposes.

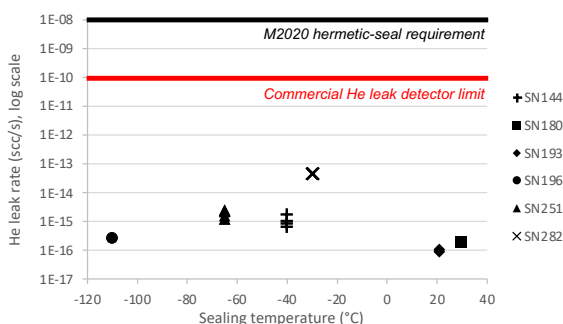


Figure 3. Results of He leak rate measurements for six flight-like sample tubes sealed at various temperatures. Vertical axis is plotted on a logarithmic scale; some data points overlap such that they are indistinguishable on this graph.

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References: [1] Farley K. A. et al. (2020) *Space Sci. Rev.*, 216, 1–41. [2] Swindle T. D. et al. (2022) *Astrobiology*, 22, S165–S175. [3] Redmond M. et al. (2015) *45th Intl. Conf. Env. Sys.*, p. 23. [4] Wong M. H. et al. (2013) *Geophys. Res. Lett.*, 40, 6033–6037.