

A GAS EXTRACTION MANIFOLD FOR THE APOLLO 17 73001 CORE SAMPLE VACUUM CONTAINER. R. Parai¹, J. Rodriguez¹, A. Meshik¹, O. Pravdivtseva¹, P. Will¹, B. L. Jolliff¹, W. S. Cassata², Z. Sharp³, C. K. Shearer³, S. B. Simon³, and the ANGSA Science Team⁴. ¹Department of Earth & Planetary Sciences and Department of Physics, and the McDonnell Center for Space Sciences, Washington University in St. Louis, St. Louis, MO 63130; ²Nuclear and Chemical Sciences Division, Lawrence Livermore National Laboratory, Livermore, CA 94550; ³Institute of Meteoritics, University of New Mexico, Albuquerque, NM 87131; ⁴<https://www.lpi.usra.edu/ANGSA/teams/> (parai@wustl.edu).

Introduction: The Apollo 17 sample 73001 is the deep portion (> 22 cm depth) of a double drive tube core of the lunar regolith that was sealed in a core sample vacuum container (CSVC) at low temperature (potentially ~250 K [1]) on the lunar surface upon collection. The sample was returned to Earth under vacuum and sealed in a secondary container that was pumped to rough vacuum pressures [2]. The sealed core sample presents two important opportunities to study lunar volatiles. First, the regolith may have retained loosely-bound or cold-trapped volatiles that would have been lost from other Apollo samples upon return or during curation; these will form the head space gas in a sealed core at room temperature. Second, if the core has remained sealed, then the solid portion of 73001 was protected from contamination from spacecraft cabin gases and Earth's atmosphere, and represents the most pristine lunar samples for analysis of volatiles, including H, C, N and noble gases. Here we discuss the design and implementation plan for a gas extraction manifold for 73001.

Background: 73001 and 73002 are the lower and upper portion, respectively, of a double-drive tube core sample from Apollo 17 Station 3, sampling the light mantle avalanche deposit derived from the north face of the South Massif of Taurus Littrow Valley. 73001 was collected in an aluminum drive tube, secured with Teflon caps and sealed in a stainless steel CSVC by means of a stainless steel knife edge acting on an indium alloy sealing surface on the CSVC lid [3]. On visual inspection, the 73001 CSVC appeared to have been sealed properly, and it was sealed in a secondary container that was evacuated to $\sim 5 \times 10^{-2}$ torr [2].

The core sample comprises both a solid component (including regolith and clasts) and the head space gas. To properly handle and interpret results from both components, it is necessary first to determine whether the seals on both the primary and secondary vacuum containers have held, and then to extract, store and characterize the head space gas from the CSVC. To accomplish these goals, a piercing tool [4] will be coupled with the gas extraction manifold detailed here.

Determination of Secondary Container Pressure: It is important to characterize the pressure in the secondary container to determine the fidelity of the

secondary container seal to atmosphere and the indium alloy seal on the CSVC over nearly 50 years' time, and to thus form an expectation for the nature of the CVSC head space gas. There are three potential cases: (a) in the ideal case, the secondary container pressure is still near $\sim 5 \times 10^{-2}$ torr, and no leaks across either seal are suspected; (b) the pressure is greater than $\sim 5 \times 10^{-2}$ torr, in which case atmosphere has likely leaked into the secondary container and may also have leaked across the indium seal into the CSVC; and (c) the pressure is lower than $\sim 5 \times 10^{-2}$ torr, in which case the indium seal leaked and the secondary container gas may have leaked into or equilibrated with the CSVC head space gas over the decades; in this case, the secondary container seal to atmosphere likely held.

In cases (b,c), the solid component of 73001 will not have been fully protected from terrestrial atmospheric contamination. We can expect that the collected head space gas is contaminated, and it will be crucial to characterize the composition of the head space gas to understand the nature of the contaminant. In case (a), the head space gas may contain pristine lunar volatiles and the solid samples were likely well-protected.

Gas Extraction Manifold: Components of the gas extraction manifold to store, distribute and provide a preliminary characterization of 73001 gases are detailed below.

Sample tanks. Four stainless steel large volume cylinders are used as sample tanks (black cylinders in Fig. 1). Each is equipped with two all-metal ultra-high vacuum (UHV) angle valves that are bakeable to high temperatures ($\sim 200^\circ\text{C}$). The double valve forms a "pipette" for drawing aliquots from filled tanks for distribution to laboratories for analysis. Tanks and associated volumes will be baked and pumped to achieve extreme UHV pressures (10^{-10} torr) to ensure cleanliness prior to filling.

Vacuum lines. Stainless steel vacuum hardware with high conductance (all outer diameters greater than $\frac{3}{4}$ "") is used to ensure gases are not fractionated during extraction or distribution of the core head space gas. Vacuum lines will be baked to high temperatures along with the sample tanks.

Pumping station. To achieve suitable vacuum, the assembly is pumped by means of a turbomolecular pump equipped with integrated backing pump, then with an ion pump (Fig. 2). An ion gauge will be used to monitor system pressure. All-metal UHV valves and stainless steel hardware are used to ensure the system can be baked to sufficient temperatures to achieve extreme UHV.

Quadrupole mass spectrometer. The manifold is equipped with a 200 amu quadrupole mass spectrometer. This mass spectrometer will be used to monitor system blanks, and to provide a preliminary characterization of the CSVC gas.

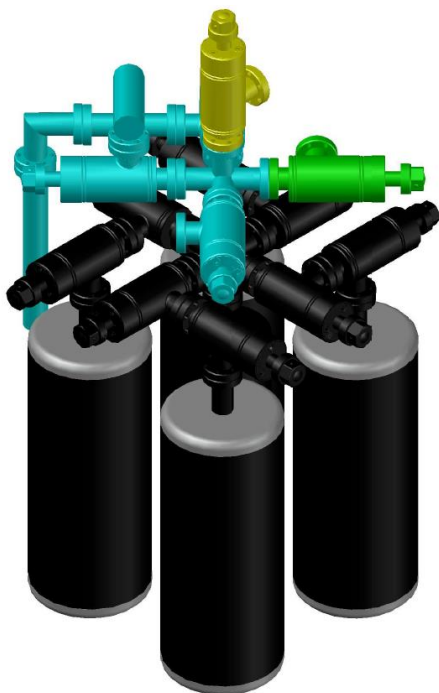


Figure 1. Tank assembly with valves to quadrupole volume (yellow), pumping station (green), and piercing tool (cyan valve at front).

Operational Plan: The manifold will be assembled, baked and tested at Washington University, then transferred to Johnson Space Center. There, it will be evacuated, baked and tested again in concert with the piercing tool. Tanks will be prepared for sampling through high-temperature baking and pumping, with blanks monitored using the quadrupole mass spectrometer.

Once the tanks are clean, they will be sealed off and the gas extraction manifold will be attached to a valve on the piercing tool assembly containing the CSVC. The gas extraction manifold will then be used to evacuate the piercing tool assembly, and all volumes

that saw the gas load from the piercing tool will be baked again to ensure very low ultimate pressures. Once suitable pressures are achieved and quadrupole scans are clean, valves to the pumping station will be closed off and valves to the sample tanks will be opened. The piercing tool will be used to pierce the end of the CSVC and head space gas will expand into the large-volume sample tanks. One tank will remain unfilled to serve as a process blank.

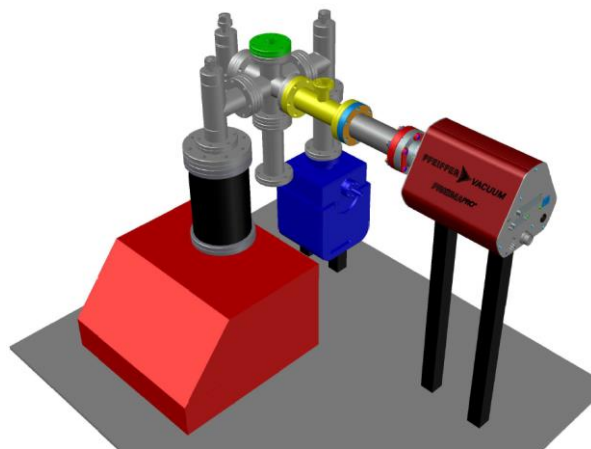


Figure 2. Pumping station and quadrupole mass spectrometer. The pumping station includes a turbo pump with integrated rough pump (red and black at left), ion pump (blue), and ion gauge. The quadrupole mass spectrometer (maroon at right) can be isolated from the pumping components. Bellows will attach the green and yellow flanges to their respective valves on the tank assembly.

Depending on the head gas pressure, preliminary head space gas characterizations may be done by drawing a shot from a sample tank using the dual-valve “pipette” and analyzing it on the quadrupole mass spectrometer. More detailed, high-precision gas analyses will be done on aliquots of the gas samples that can be drawn from the tanks and expanded into separate volumes for distribution to other laboratories.

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References: [1] Keihm S.J. and Langseth M.G. (1973) *Proc. 4th Lunar Sci. Conf.* 2503-2513. [2] Butler P. (1973) *Lunar Sample Information Catalog*, NASA Johnson Space Center. [3] Allton J. A. (1989) *JSC 23454*, NASA Johnson Space Center. [4] Schild, T. et al. (2021) *LPS LII*, this meeting.