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## SUMMARY

PREMISE is an internally-funded study by Southwest Research Institute (SwRI) to simulate the flux of gases and volatiles that would occur at the lunar surface due to a cometary impact or lunar outgassing event. A small chamber containing a sample cell was added to an existing vacuum system to permit the pump down and bakeout of JSC-1A Lunar Soil simulant. Once a good vacuum was achieved ( $1 \times 10^{-8}$  Torr), the sample was then exposed to various gases to observe their retention, if any, as a function of the exposure period and pressures used.

Gases included pure argon, a Mars mix and a mixture containing H<sub>2</sub>, He, Ne, N, and Ar. Preliminary results suggested that retention of gas was occurring at room temperature. This effect was investigated further and results are included below.

## BACKGROUND

Two issues motivated this effort. First, recent evidence of water trapped in the polar regions of both the Moon[1] and Mercury[2], have triggered a renaissance in the study of regolith environments. Laboratory simulation of these environments not only demands ultrahigh vacuum (UHV) conditions, but also the introduction of fine powders of either actual lunar regolith soil samples or regolith "simulants" into that same vacuum environment. Regolith materials are a rare laboratory commodity and in short supply. Regolith simulants are much more plentiful, but are fabricated from terrestrial basalts that place serious physical limits (particle size & distribution, morphology, composition, oxidation state) on their usefulness as analogues for regolith samples[3]. The second motivating factor was the past detection of Ar at the lunar surface by a mass spectrometer[4] deployed during the Apollo 17 mission (Dec '72) that has not yet been verified by remote sensing[5].

## TECHNICAL APPROACH

PREMISE consists of a stainless steel UHV system, a sample chamber and a gas inlet system. Both the vacuum system and gas inlet have separate turbomolecular pumping systems. In addition, the vacuum system also includes an ion pump for additional pumping once high vacuum conditions have been achieved by the turbopump.

The sample chamber consists of a Kimball Physics Magdeburg Hemisphere® (Fig. 1) miniature vacuum chamber having one 4.5" ConFlat® (CF)-style flange connection at the base, with two 2.75" CF flange connections and four 1.33" CF flange connections on the housing. Chamber connections lead to the vacuum system, gas inlet manifold, UV quartz window, thermocouple (TC) feedthrough, rotary feedthrough, residual gas analyzer (RGA) and sample cell.

The sample cell assembly consists of two copper gaskets (one solid, one standard) brazed together and gold plated (Fig. 2). This forms a flat cylindrical volume approximately 2 mm deep with a surface area of approximately 10 cm<sup>2</sup>.

The RGA is an Extorr XT300M quadrupole mass spectrometer (QMS) with mass scanning from 1 to 300 daltons. The full range of scanned masses is typically limited to 50 daltons to reduce data file size, as the heaviest mass used thus far has been that of CO<sub>2</sub> (44 daltons). However, bakeouts, laser ablation or disturbances to the regolith are covered by full-range scans (300 daltons) when necessary to capture any unanticipated features at higher molecular weights. A schematic of the complete system is shown in Figure 3.

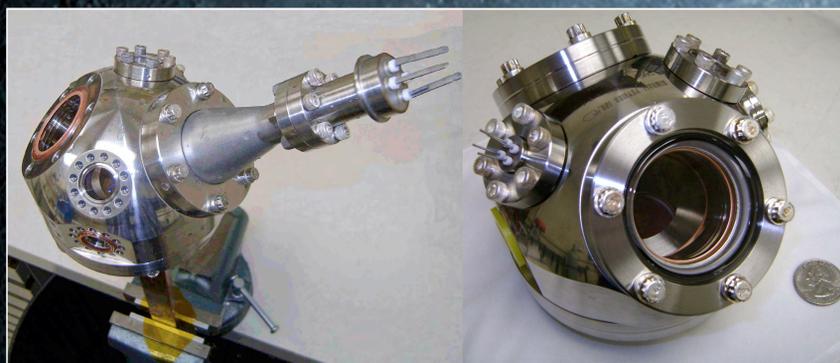


Figure 1 (above): The soil sample chamber uses seven ultrahigh vacuum (UHV) flange connections in three flange sizes (1.33", 2.75" & 4.5").

Figure 2 (at left): The soil sample cell is composed of two copper gaskets (one standard, one solid) brazed together and coated in gold. One thermocouple (TC) is attached and the other is suspended 1 mm above the bottom of the cell. The cell contains the sample and also acts as the vacuum sealing interface at the bottom of the chamber.

## PROCEDURE

**GAS DOSING:** After baking of the simulant to drive off atmospheric gases and volatiles, gas was introduced to the sample volume through a Varian sapphire leak valve at a selected pressure and for a selected period of time. At a base pressure of  $1 \times 10^{-8}$  Torr, the desiccated JSC-1A lunar simulant was exposed at room temperature to a simulated Mars atmospheric mixture. Gas was introduced until the background pressure reached approximately  $3 \times 10^{-8}$  Torr. After holding at a constant pressure for a total of 10 minutes, the leak valve was rapidly closed.

PREMISE was constructed for the purpose of monitoring gas-simulant interactions over a variety of temperatures and gas exposure pressures and times, but the persistence of certain peaks at room temperature precluded investigations of the sample under cryogenic conditions until this unanticipated peak persistence could be explained.

**PHOTO-DESORPTION:** Matrix-assisted laser desorption ionization mass spectrometry (MALDI-MS) and laser desorption resonance ionization mass spectrometry (LDRIMS) have become prominent analytical techniques employing lasers with possible applications for lunar and planetary research. Operation of a laser at the lunar surface could be part of a sample-handling protocol for analytical instruments that was unavailable during the Apollo era. Consequently, a laser was employed as a means to desorb volatiles from the JSC-1A lunar simulant.

**MECHANICAL PROCESSING:** Whether due to the disturbance of the upper layers of the lunar regolith by the impact of a landing spacecraft, or by the sample handling system of onboard analytical instrumentation, manipulation of lunar regolith soil by mechanical means is an absolute certainty for any landed probe. We used a vacuum feedthrough with a rotary shaft to manipulate a tiny stainless steel "flag" for disturbing the JSC-1A simulant in a manner analogous to similar mechanisms that would likely be used at the lunar surface.

## RESULTS

The desiccated JSC-1A regolith simulant was exposed at room temperature to carbon dioxide (CO<sub>2</sub>) in the simulated Mars atmosphere. Data reveal a persistence of the CO<sub>2</sub> molecule at 44 daltons (CO<sub>2</sub><sup>+</sup>) and for daughter ion peaks at 22 daltons (CO<sub>2</sub><sup>2+</sup>), 16 daltons (O<sup>+</sup>) and 12 daltons (C<sup>+</sup>). The data also show the time required for captured CO<sub>2</sub> molecules to reach equilibrium with evolving CO<sub>2</sub> molecules is inversely proportional to both temperature and pressure.

The argon mass peaks at 40 daltons (Ar<sup>+</sup>) and 20 daltons (Ar<sup>2+</sup>) were also monitored during exposure of the simulant to the Mars gas mix. No retention of Ar at the sample surface was observed. Figure 4 represents mass scans for the mass 40 channel (top) and mass 44 channel (bottom) representing Ar and CO<sub>2</sub>, respectively. Our tests suggest lunar regolith does not trap Ar at room temperature and these results are relevant to current interest in the sources and persistence of Ar detected by mass spectrometry [4] during the Apollo 17 mission. Though Ar has two doublets (92.0 & 93.2 nm and 104.8 & 106.7 nm) appearing in the UV portion of the spectrum, this gas has not been detected in the exosphere at the limb of the Moon by the Lyman-Alpha Mapping Project (LAMP) aboard the Lunar Reconnaissance Orbiter (LRO).

Use of a laser produced an evolved gas plume of sufficient magnitude to saturate the detector, so its use was abandoned. Subsequent inspection of the sample cell through the UV quartz window with a "white" LED flashlight resulted in an immediate and unexpected elevation in the chamber background pressure. It was assumed that this apparent photon desorption was due to the shorter wavelength "tail" in the blue segment of the composite "white" LED. This assumption was supported by subsequent tests using infrared, red, yellow, and green LEDs which resulted in no increase in the chamber base pressure. However, use of a UV LED flashlight produced even higher changes in base pressure.

At present, PREMISE does not permit bakeouts above the typical maximum surface temperature seen at the lunar equator (~120°C). However, after several bakeouts to this temperature and two months at UHV ( $1 \times 10^{-8}$  Torr), rotation of the metal flag through the simulant still resulted in significant gas evolution greater than that observed through photo-desorption. Figure 5 (right) shows an example of a mass spectrometer scan taken during efforts to disturb the simulant using both the UV LED flashlight and the rotary feedthrough.

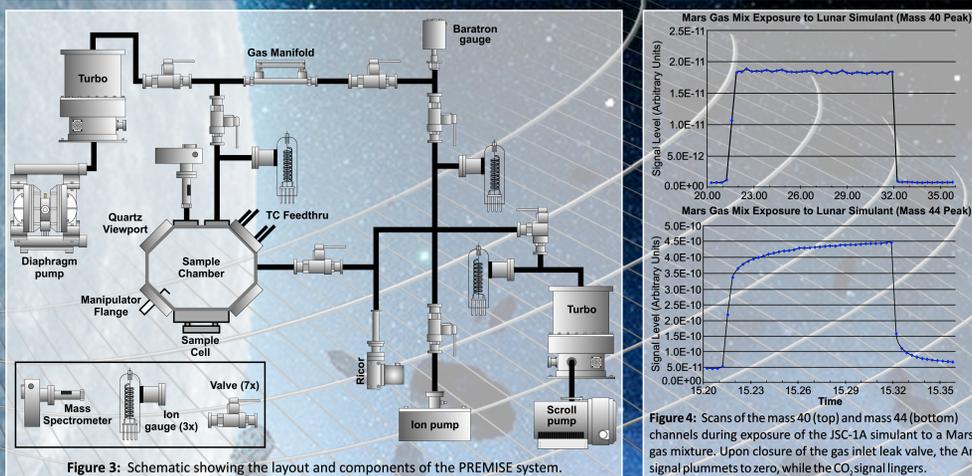


Figure 3: Schematic showing the layout and components of the PREMISE system.

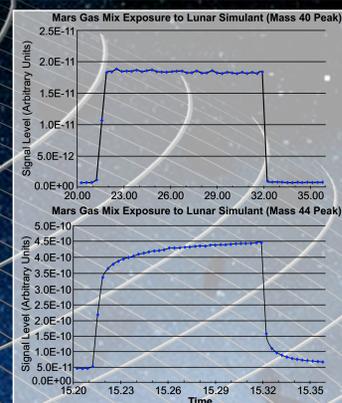


Figure 4: Scans of the mass 40 (top) and mass 44 (bottom) channels during exposure of the JSC-1A simulant to a Mars gas mixture. Upon closure of the gas inlet leak valve, the Ar signal plummets to zero, while the CO<sub>2</sub> signal lingers.

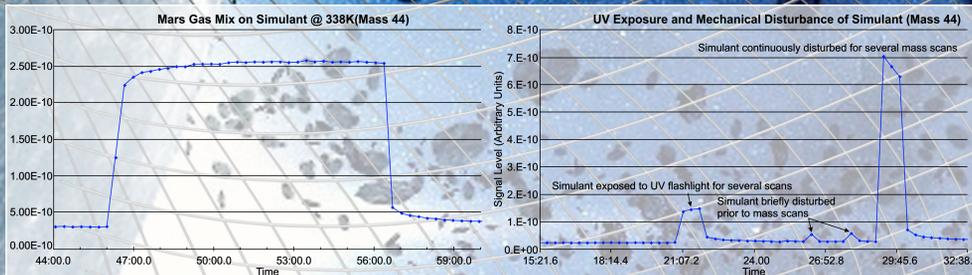


Figure 5: (Left) This scan of the mass 44 peak (CO<sub>2</sub>) was taken during exposure to the Mars gas mix at an elevated temperature (338K) and demonstrates that the gas reaches equilibrium with the simulant surface more rapidly. (Right) This scan of the mass 44 channel demonstrates the effect of a UV LED flashlight and a mechanical flag on the evolution of CO<sub>2</sub> from the desiccated JSC-1A simulant.

## CONCLUSIONS

1. The insertion and use of JSC-1A lunar soil simulant in a vacuum system is possible without incident provided the material is handled carefully, evacuation occurs slowly so as to avoid small particle migration, and the system is then slowly and carefully returned to atmospheric pressure during venting. Pressure "waves" simulating molecular impact from distant lunar outgassing events (cometary impacts or seismic events) can be conducted repeatedly without incident.
2. PREMISE data suggest that UV light emitting diodes, and even "white light" diodes are not only sufficient to evolve materials trapped within the lunar regolith, but can do so at much lower cost, mass and power than a typical laser system. Future investigations with diode lasers are warranted.
3. Argon is routinely sampled to determine ages of mid-ocean ridge basalts (MORBs) using the potassium-argon (K-Ar) geochron[6]. For this reason, Ar analysis at the lunar surface is relevant to the study of lunar basalts and to Earth-Moon comparative geology and planetary science.
4. This effort suggests further adsorbant/adsorbate studies in regolith materials is warranted due to current missions investigating the surface-bounded exospheres (SBEs) of the Moon (LRO, LADEE), Mercury (MESSENGER, BepiColombo) and asteroids (OSIRIS-REx).

## REFERENCES

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