Characterization of Volatiles Loss from Soil Samples at Lunar Environments

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Mission

- Prospect for water at the lunar poles as a potential resource for In-Situ Resource Utilization (ISRU)
- Characterize the nature and distribution of water/volatiles in the lunar polar sub-surface materials

Mobility

Rover
- Mobility system
- Cameras
- Surface interaction

Sampling

Drill
- Subsurface sample acquisition
- Auger for fast subsurface assay
- Sample transfer for detailed subsurface assay

Processing & Analysis

Oxygen & Volatile Extraction Node (OVEN)
- Volatile Content/Oxygen Extraction by warming
- Total sample mass

Lunar Advanced Volatile Analysis (LAVA)
- Analytical volatile identification and quantification in delivered sample with GC/MS
- Measure water content of regolith at 0.5% (weight) or greater
- Characterize volatiles of interest below 70 AMU

Prospecting

Neutron Spectrometer System (NSS)
- Water-equivalent hydrogen > 0.5 wt% down to 1 meter depth

NIR Volatiles Spectrometer System (NIRVSS)
- Surface H2O/OH identification
- Near-subsurface sample characterization
- Drill site imaging
- Drill site temperatures
Resource Prospector (RP)  
Integrated Thermal Vacuum Test Program

- A series of ground based dirty thermal vacuum tests are being conducted to better understand the subsurface sampling operations for RP
  - Volatiles loss during sampling operations
  - Hardware performance
  - Concept of operations
- 5 test campaigns over 5 years have been conducted with RP hardware with advancing hardware designs and additional RP subsystems
  - Volatiles sampling 4 yrs
- Using flight-forward regolith sampling hardware, empirically determine volatile retention at lunar-relevant conditions
  - Use data to improve theoretical predictions
  - Determine driving variables for retention
  - Bound water loss potential to define measurement uncertainties
Overview

• The main goal of this talk is to introduce you to our approach to characterizing volatiles loss for RP.
  – Introduce the facility and its capabilities
  – Overview of the RP hardware used in integrated testing (most recent iteration)
  – Summarize the test variables used thus far
  – Review a sample of the results
VF13
Planetary Surface Simulation Facility

**Dedicated ‘dirty’ thermal vacuum chamber operated with up to 1-ton of lunar soil simulant**

**Dimensions**
- Maximum internal volume of 6.35 m³
- Internal dimensions: 3.6 m tall, 1.35 m diameter with cold wall, 1.5 m without cold wall
  - Fixed base 1.08 m deep + Removable cap 2.52 m tall

**Thermal capability**
- Removable cold wall in cap (top 2.5 m of chamber)
  - Temperature control from ambient to liquid nitrogen temperatures
  - 2 semi circular halves, independently controlled to achieve temperature gradients
  - Minimum temperature 80K (liquid nitrogen cooled)
- Fixed base has separate Liquid Nitrogen cooling, independent of cold wall
  - Supports cooling of soil bin (existing bin is 0.278 m diameter, 1.2 m tall)
- Liquid nitrogen is supplied from a 55,000 gallon dewar

**Vacuum capability**
- Achievable pressure on the order of 10⁻⁶ Torr, with soil
- Variety of customizable electrical and mechanical feed-throughs
- Four vacuum pumps to accommodate range of pressure regimes and pump rates
- (in process) Mars gas capability: Flow panel controlled with a Mass Spectrometer to maintain a Mars environmental conditions.

**Facility operation**
- PLC control allows for unattended operation for majority of pump down and cooling
- Customizable digital data acquisition system supporting over 80 channels
- Internal cameras for optical access
VF13 Research Hardware

Cylindrical Bin ("Drill Tube")
- 1.2m (48in) tall, 0.278m (11in) diameter
- Holds 100 kg of simulant
- Three side ports for soil embedded thermocouples (15, type T)
- Clamp on LN\textsubscript{2} Coolant system, soil temperature as low as -160° C

Square Bin
- 1 m x 1 m x 1 m
- Holds 800 kg of soil simulant

Robotic Translation Table (trolley)
- Enables lateral motion of research hardware to reach different locations on the soil bed surface
- Individual, Manual control of X and Y directions
- Position Encoders: ± 2 mm (approx.)
Soil Bin analysis methods

Soil Bin Preparation

- LHT-3M, ~100kg, doped with distilled water
  - Mixed in batches of 20-25kg (5 gal)
  - Samples taken from each batch to verify moisture
- Compacted into cylindrical bin
  - Vibratory compaction with 150lb surcharge weight
  - Compacted in layers, ~20 kg each

“Post Mortem”

- No in-situ moisture measurement during vacuum test
- Depth dependent moisture profile generated after test (thawed soil bin) using core sampling
  - Difference between thawed and frozen bin moisture profile only impacts the top ~10cm
  - Majority of desiccation occurs in top 30cm
RP Test hardware

- RP EDU Drill: *Honeybee Robotics*
- Near InfraRed Volatiles Spectrometer System (NIRVSS): NASA ARC
- Oxygen & Volatile Extraction Node (OVEN): NASA JSC
- Sample Capture Mechanisms
- Residual Gas Analyzer
Sample analysis methods

Test samples
- Sampling at 30 to 40cm depth
- Drilling in progressive 10cm bites
- The bottom 10 cm of auger captures sample on tapered auger flutes
- Sample dispensed into crucibles using a passive brush wheel and funnel on drill
- Solenoid actuated, spring driven seal mechanism with a knife edge-to-teflon seal, 100lbf clamp force

Sample analysis
- Moisture content of each sample is measured using ASTM standard
  - Bake at 110°C
  - Weight change
<table>
<thead>
<tr>
<th>Variables</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure</td>
<td>Low as possible 1e(^{-5}) Torr to 2e(^{-6}) Torr</td>
</tr>
<tr>
<td>Shroud Temperature</td>
<td>Controlled -50° C to -175° C</td>
</tr>
<tr>
<td>Soil Bin Temperature</td>
<td>Low as possible (Dependent on time, soil moisture) -80° C to -163° C</td>
</tr>
<tr>
<td>Soil Bin Moisture</td>
<td>Controlled ≤ 5wt% Stratification</td>
</tr>
<tr>
<td>Sample Crucible Temperature</td>
<td>Controlled 10° C Cold as possible -20° C to -70° C</td>
</tr>
<tr>
<td>Sample exposure time (in crucible)</td>
<td>Controlled 3min delay Fast as possible ~5min</td>
</tr>
<tr>
<td>Sample size</td>
<td>Target 15 g Average 12g (Range 4 g to 20 g)</td>
</tr>
</tbody>
</table>
Results from a drill sample bite:

Water release, according to RGA, show majority of release occurs when dispensing into the sample crucible.

Sample 2017_O3: 3.3g, 0.4wt%, 84% loss
Test Results, general observations

- Samples from a higher soil content retain higher percentage of water
- Data from low mass samples is less consistent (more scatter)
Test Results, general observations

• The rate of mass loss appears to be consistent for similar sample sizes. Samples with higher starting moisture content therefore lose less %.

• This mass loss could be correlated to sublimation rate.

• The sample is exposed to 4 temperatures: which is the driving temperature?
  – Soil bin
  – Cold wall
  – Drill bit
  – Sample Crucible
Test Results, general observations

The closest correlations are with:

**Bit temperature**
- But the lower temperature also have lower sample masses.

**Soil temperature:**
The 3 points at the lowest temperature are outliers to this trend. These are the 3 OVEN samples whose differences are:
- Sealed better than most of the SCM samples
- Warmer crucible temperature for OVEN crucibles (though on the bottom right graph this trends well with mass loss)
Summary

• To date we have conducted 4 test campaigns with volatiles sampling
  – 43 samples total
  – 4 soil moisture conditions with 26 samples from the same (~5wt%)
• Test performed with 3 RP subsystems: Drill, NIRVSS, OVEN
  – 3 tests were performed with the RP OVEN hardware, all the rest with the customized Sample Crucible Mechanisms (SCMs)
• Using flight-forward regolith sampling hardware, empirically determine volatile retention at lunar-relevant conditions
  – Use data to improve theoretical predictions
  – Determine driving variables for retention, adjust hardware and con-ops accordingly
  – Bound water loss potential to define measurement uncertainties

• Analysis of sample results in on-going, with a summary paper expected at the ASCE Earth and Space conference, April 2018
BACKUP
Test Results, example
Resource Prospector (RP) Overview

Mission:
• Characterize the nature and distribution of water/volatiles in lunar polar sub-surface materials
• Demonstrate ISRU processing of lunar regolith

Project Timeline:
✓ FY13: Pre-Phase A: MCR (Pre-Formulation)
✓ FY14: Phase A (Formulation)
✓ FY15: Phase A (Demonstration: RP15)
✓ FY16: Phase A (Risk Reduction)
✓ FY17: L2 Requirement Lockdown (July 11)
✓ FY18: MRD and PDR (Implementation)
✓ FY19: CDR (Critical design)
✓ FY20: I&T
✓ FY21: RP launch

RP Specs:
Mission Life: 6-14 earth days (extended missions being studied)
Rover + Payload Mass: 300 kg
Total system wet mass (on LV): 5000 kg
Rover Dimensions: 1.4m x 1.4m x 2m
Rover Power (nom): 300W
Customer: HEOMD/AES
Cost: ~$250M (excl LV)
Mission Class: D-Cat3
Launch Vehicle: EM-2 or ELV