Volatile Loss from water bearing regolith simulant at Lunar Environments

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Lunar Polar Volatiles

- Permanently shadowed craters at the lunar poles contain water, ~5 wt% according to LCROSS
- Interest in water for ISRU applications
- Desire to ‘ground truth’ water using surface prospecting – e.g. Resource Prospector (RP) & RESOLVE
- How to access subsurface water resources and accurately measure quantity – Excavation operations and exposure to lunar environment may affect the results
Volatile capture tests

• A series of ground based dirty thermal vacuum tests are being conducted to better understand the subsurface sampling operations
  – Sample removal and transfer
  – Volatiles loss during sampling operations
  – Concept of operations
  – Instrumentation

• This presentation covers:
  – The capabilities of the VF-13 Thermal Vacuum Chamber (TVAC)
  – The Resource Prospector TVAC hardware
  – The summary and results of 5 years of RP volatiles tests
    • 43 viable samples
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
- Ports available for gas feed from portable bottles, to achieve customizable pressures and gas compositions (e.g., Mars environment)

**Facility operation**
- PLC control software 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

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Planetary Soil Simulants

- Available bin sizes
  - Square bin: 1 m³ holds 800 kg of soil simulant
  - Cylindrical Cooled bin: 0.278 m diameter, 1.2 m tall, holds 100 kg of simulant
    - Can be instrumented with 15 thermocouples embedded in the soil
- Two bins of each size so that one can be prepared while other is tested
- Variety of simulants currently at GRC: LHT3m, JSC1A, Chenobi, GRC3, GRC1
- Preparation in SLOPE lab to accommodate large quantities of soil
  - Preparation can include: compaction (vibrational), moisture control (drying/wetting)

Available Test-Support Hardware

- Robotic Translation Table to position hardware above soil bin
  - Enables lateral motion of research hardware to reach different locations on the soil bed surface
  - Separate control of X and Y directions, manual control
- Drill system
  - Simple 2 motor drive system can accommodate a 1 m tall drill tool
  - Mounted to translation table for multiple drill holes in the same bin
  - Encoders for feedback of rotation rate and drill depth
Resource Prospector: TVAC Hardware

**Drill:** Sample retrieval

**NIRVSS:** Soil surface assay

**OVEN or SCMs:** Sample containment for analysis
Lunar Prospector Drill

• Developed by Honeybee Robotics, and based on the Mars Icebreaker drill
• 100cm long, 2.5cm diameter auger
  – 10cm sample section has wider flutes at high pitch to capture granular material
  – Progressive "Bite sampling" approach to drilling
    • Retains depth stratigraphy of the holes
    • Less material conveyed to surface, less chance of stuck bit
• Sample delivery mechanism
  – Deployed to surface as stabilizing foot
  – Fully contains the 10cm sample when auger retracted
  – Passive brush that rotates as auger spins past. Material brushed off auger and through funnel for collection
• Actuators: Percussion, deployment Z-stage, Drill Z-stage, Auger rotation
Sample Capture Mechanisms (SCM)

- Capture soil from the auger and seal at vacuum conditions to retain volatiles
- Stepper motor actuated, spring driven mechanism with a knife edge-to-teflon seal, 100lbf clamp force
- Sealed 18ml crucibles easily removed for sample analysis
- 6 Sample Capture Mechanisms in each test for multiple samples.
Oxygen and Volatile Extraction Node (OVEN)

- Carousal system designed to reuse 12cc crucibles for sample capture and volatile evolution
  - Seal, Weigh, Heat, Dispose
  - Volatile analysis in downstream spectrometer, not available in TVAC

- TVAC alteration:
  - Sample capture
  - Seal
  - Store

- Sealed samples removed for analysis
  - Break vacuum to replace crucible
Near Infrared Volatiles Spectrometer Subsystem (NIRVSS)

- Views soil surface and drill cutting pile to evaluate surface water profile
- Components include:
  - Observation camera
  - Illumination sources (Lamp & LEDs)
  - Low wavelength Calibration Sensor
  - 2 Near Infrared Spectrometers (Fiber optic)
## Variables

<table>
<thead>
<tr>
<th>Variable</th>
<th>Target</th>
<th>Actual conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Pressure</strong></td>
<td>minimize</td>
<td>Average 4e⁻⁶ Torr Median 3e⁻⁶ Torr Range: 2e⁻⁵ Torr to 1.5e⁻⁶ Torr</td>
</tr>
<tr>
<td><strong>Cold Wall Temperature</strong></td>
<td>-50°C or -170°C (sunlight surface or cold as possible)</td>
<td>-50°C or -170°C</td>
</tr>
<tr>
<td><strong>Soil Temperature</strong></td>
<td>&lt;= -100°C</td>
<td>-80°C to -160°C (all but 3 under -100°C)</td>
</tr>
<tr>
<td><strong>Soil Moisture</strong></td>
<td>0.5 wt% to 5 wt%</td>
<td>4 test w/ 5 wt% 2 tests w/ 2.5 wt% 2 tests w/ 0.8 wt%. 2 tests w/ alternating layers of 5 wt% and 0.5 wt%</td>
</tr>
<tr>
<td><strong>Crucible temperature</strong></td>
<td>-20°C to +20°C</td>
<td>SCMS: -20°C or +10°C OVEN: ~+20°C</td>
</tr>
<tr>
<td><strong>Exposure Time</strong></td>
<td>minimize</td>
<td>~15 min to retrieve sample 5 min to fill crucible</td>
</tr>
<tr>
<td><strong>Sample size</strong></td>
<td>15 g</td>
<td>Average 12 g Median 14 g Range: 3.3 g to 20.7 g</td>
</tr>
</tbody>
</table>
Results

• The sampling process had a greater impact on drier soil beds:
  - Average loss for 5% bed: 30%
  - Average loss for 0.8% bed: 80%
• If the sublimation rate is consistent, then for same exposure time a sample with lower moisture will lose a greater percentage
Results: Mass loss

- Sample mass loss divided by exposure time (out of hole) to get mass loss rates
- Total sample mass (symbols) correlates strongly with loss rate
  - Smaller samples have slower loss rate
  - For samples of similar size, loss rates are similar, regardless of starting moisture content
Results: sublimation driver

• Assuming the loss rates correlate with sublimation rate, what is the driving factor?
• Pressure is similar for all tests, which leaves temperature
• Which temperature? Samples exposed to several temperatures before delivery.
Results: RGA, When is the water lost?
Conclusions

• VF-13 is a 6.35 m³ dirty thermal vacuum chamber available for testing with simulants at lunar or Martian conditions.

• Volatiles loss tests for Resource Prospector (RP) have been conducted in VF-13 for 5 years
  – Volatiles loss during drill sampling and transfer operations
  – 3 RP subsystems integrated in these tests
  – 43 viable simulant samples have been analyzed

• The complexity of the sampling process (number of variables, exposure variability, etc) have made definitive conclusion challenging
  – Mass loss rates have consistency for similar sample size, but scatter cannot be correlated to temperature conditions
  – Analytical correlations to sublimation rates at temperature and pressure are not sufficient for correlation.

• Mass spectrometer data shows majority of water loss occurs during ‘brushing’ operation, when the sample is agitated upon delivery to crucible