

**QUANTIFICATION OF H<sub>2</sub>O AND CO<sub>2</sub> DOSING IN PREPARATION OF CRYOGENIC ICY REGOLITH SIMULANTS.** E. K. Lewis<sup>1</sup>, S. Ghosh<sup>2</sup>, Christopher L. Harris<sup>2</sup>, Cecilia L. Amick<sup>2</sup>, Crystal Mantilla<sup>2</sup>, Danielle Thompson<sup>2</sup>, Jeremy W. Boyce<sup>3</sup>, <sup>1</sup>NASA Texas State Texas-JETS Contract, JSC-ARES XI2 [ernest.k.lewis@nasa.gov](mailto:ernest.k.lewis@nasa.gov), <sup>2</sup>NASA Amentum Contract, JSC-ARES XI2, <sup>3</sup>NASA Civil Servant, Johnson Space Center, ARES XI2/XI3 Curation and Research

**Introduction:** The ability to simulate volatile-bearing regolith is critical for scientific and curatorial purposes. This work presents quantification of volatiles delivered in the generation of icy regolith simulant, over a range of volatile contents, for both H<sub>2</sub>O- and CO<sub>2</sub>-bearing regolith simulants. This is critical for both scientific and curatorial goals related to upcoming Artemis missions as well as other cryo-sample return missions. In this study, we use thermogravimetry to test our process on whether expected amounts of icy-volatile contents match delivered to icy regolith simulant.

**Methods:** Here we use two different methods that are applicable to separate scales of cryogenic icy regolith production. Sample vials and regolith were always pre-weighed, assigned ID numbers and pre-chilled to LN<sub>2</sub> temperatures prior to loading. For the first method—best suited for small batches up to 250-500g of total material—cryogenic ices are combined with pre-frozen regolith within a sample tube and simply shaken to achieve homogeneity. For the second method—best suited for 0.25-2 kg samples—we mix pre-frozen regolith and frozen volatiles in our low temperature bulk mixing system at cryogenic temperature. This bulk mixing currently takes place in a standard kitchen mixing bowl at cryotemperatures but could potentially be scaled up by orders of magnitude in the future using industrial mixers. In both cases, the samples were loaded under cryogenic clouds of either liquid nitrogen argon resulting in an atmosphere-free cloud for consistent sample preparation.

Two different icy volatiles were used for this work: H<sub>2</sub>O(s) and CO<sub>2</sub>(s). The H<sub>2</sub>O was formed by spraying water onto a cryogenic spray block and captured as low-density snow. The CO<sub>2</sub>(s) is from a liquid CO<sub>2</sub> tank, that has a CO<sub>2</sub>-Snow generator directly attached to the tank. The CO<sub>2</sub> snow is sprayed directly into the capture bag, and from that aliquots of CO<sub>2</sub> can be delivered to the regolith. The cryogenic spray block is held at LAr or LN<sub>2</sub> bath temperatures within a cryo-container. This allows for forming condensed volatiles at cryogenic temperatures.

**Thermogravimetry:** To validate our sample preparation methods, we have utilized both vacuum drying and freeze drying thermogravimetry. Vacuum drying involves placing all the samples with vented caps into a vacuum oven operating at 40°-50°C for 1-2 days. The freeze dryer operates at vacuum pressures with a condenser at -85°C resulting in the extraction of

volatiles well below the freezing point of species such as H<sub>2</sub>O. Because of size limitations in the freeze dryer, small batches of samples are dried as opposed to being able to dry many hundreds of grams worth of regolith in vials simultaneously as in the vacuum dryer. Also, we can place samples into a -20°C freezer which permits the sublimation of CO<sub>2</sub>(s) but the quantitative retention of H<sub>2</sub>O. This example demonstrates a stepwise separation of volatiles prior to complete drying to ascertain if any H<sub>2</sub>O accompanied the formation of CO<sub>2</sub> regolith, though it would also work for mixed H<sub>2</sub>O-CO<sub>2</sub> regolith simulants. Mass difference (preferably measured at the same temperature in both cases), yields the volatile content of the regolith. This can be compared with the expected mass to determine dosing efficiency.

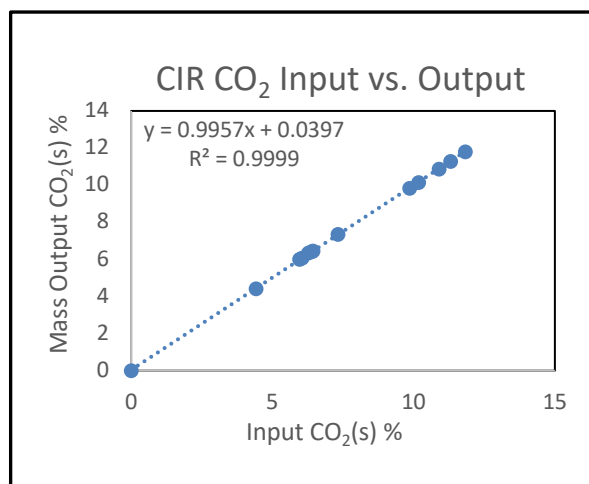


Figure 1 shows delivered CO<sub>2</sub> plotted as a function of input CO<sub>2</sub> for 10 different samples with different CO<sub>2</sub> abundances. A slope of nearly unity, intercept of zero, and strong correlation indicate that this technique makes accurate and precise icy regolith simulant.

**Results: Small volume synthesis.** We used the small-volume method to make 10 CO<sub>2</sub>-bearing regolith simulant samples (11 samples if you count the blank with zero added CO<sub>2</sub>; Fig. 1). We not only observe a linear relationship between expected and delivered volatiles, but the slope is very nearly exactly equal to unity and the intercept is very nearly equal to zero. This demonstrates that no significant losses of CO<sub>2</sub>(s) occur from handling, and that delivery is 100% efficient. Pre-freezing the regolith and keeping all materials at low temperatures minimizes sublimation. These results also

indicate that no significant gain of atmospheric H<sub>2</sub>O occurs even though we do not use a glove box, speaking to the efficacy of the “cryogen cloud” that reduces atmospheric contamination.

**Large volume synthesis.** In contrast to small volume synthesis of CO<sub>2</sub>-bearing regolith, delivery of H<sub>2</sub>O using the mixer is not 100% efficient, with a slope of only 90%. However, it is very precise, consistently delivering 90% ± 2% (2SD) that fraction. The source of the ice loss within the protocol is not known, but it likely occurs prior to mixing during the weighing stage before ice is added to the mixing bowl. Even though delivery is not 100% efficient, it is extremely reproducible, and this consistency suggests that once it is known, it would be possible to account for it by raising the input ice content.

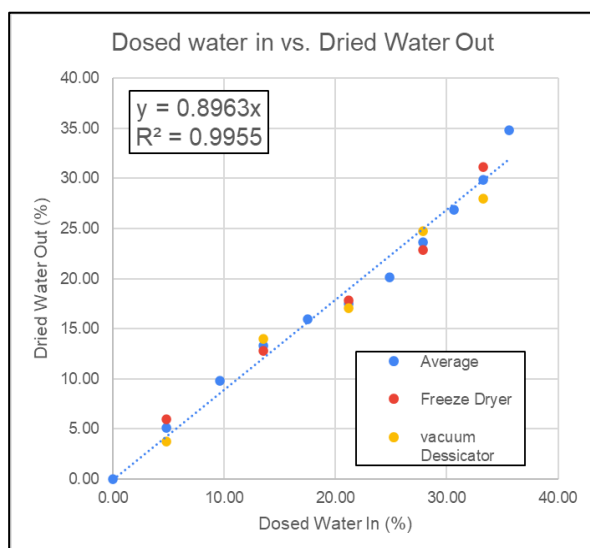


Figure 2 Sample aliquots of various percentages of cryogenic icy regolith loaded with H<sub>2</sub>O demonstrate a 90% delivery efficiency over a wide range of water contents, regardless of drying technique used (i.e., freeze drying vs. vacuum drying).

**Conclusions:** Thermogravimetric measurements of the products of two different methods of making volatile-dosed icy regolith simulant indicate that small batch, CO<sub>2</sub>-bearing regolith can be made with ~zero volatile loss. Larger batches require use of a mixing apparatus and in some part of the process there is a consistent loss of H<sub>2</sub>O-ice of ~10%. But because it is consistent it can be accounted for, and this method can still be used to make icy regolith simulants with precise and accurate volatile abundances at the ~kg scale.

In conclusion, these two methods of synthesizing quantified mixtures of cryogenic icy regolith (small batch, and large mixing bowl) provide high degrees of precision with measured standard deviations. Two

different sources of icy regolith were used: H<sub>2</sub>O and CO<sub>2</sub>(s) both of which were spray generated.

Furthermore, these methods can be readily applied by the cryogenic icy simulant community and lay the foundation for future comparisons with advanced experiments and melting of icy-regolith systems along with comparisons with computational models as they are developed.

Finally, it’s important to point out that these two methods compliment and support different applications. For larger bulk applications that may require small tool testing, or other conditions where the chemical precision isn’t required, the bulk mix method can be utilized. The bulk mixing method also works for experimenting with larger sample return vessel testing. For small volume applications such as working with rare materials and minerals in limited supply, such as with specific parent body compositions, the small batch method with higher precision is directly applicable and likely preferred.

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