FY24 Topic Areas Research and Technology Development (TRTD)

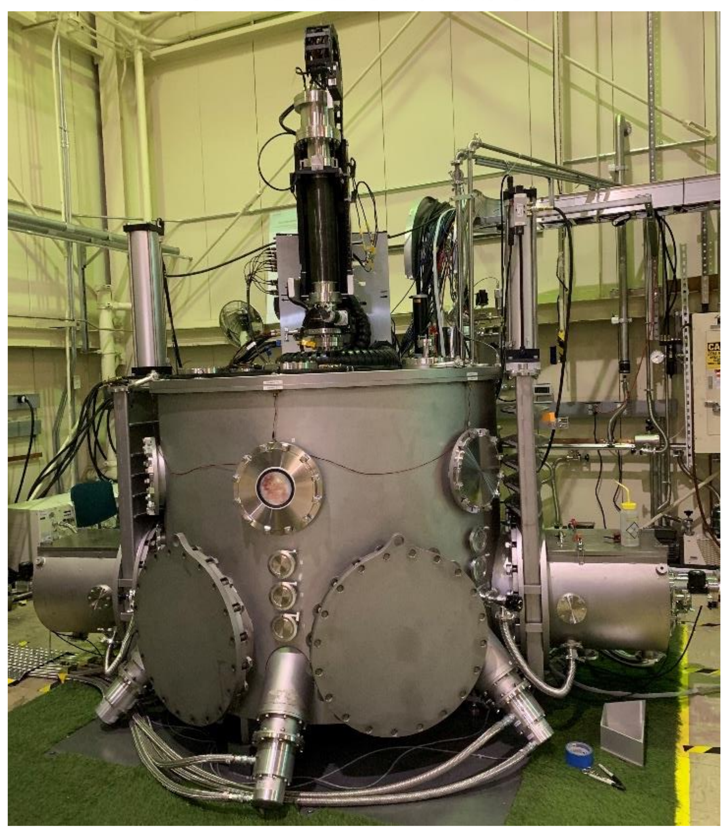
Sample Integrity Evaluation Methodology for Icy Worlds Applications

Principal Investigator: Anthony Wong (353); **Co-Investigators:** Daniel Green (352), Tatiana Jaimes (352), William Warner (353), Dallas Martinez (353), Lori Shiraishi (352), Kristopher Kriechbaum (352), Sun Yi (NC A&T), Paul Akangah (NC A&T)
Contributors: Maggie Potter (353), Nick Hatcher (353), Cambria Logan, Wade Smith, Sarah Yearicks

Objective

To develop a method of evaluating the scientific integrity of a collected water ice sample as compared to its parent material. Sample acquisitions leverage the development of the Ice Cutting and Encapsulation via Centrifugal Acceleration and Pneumatics (ICECAP) tool for use in the existing stat of the art Cryogenic Ice Transfer, Acquisition Development, and Excavation Laboratory (CITADEL) vacuum chamber.

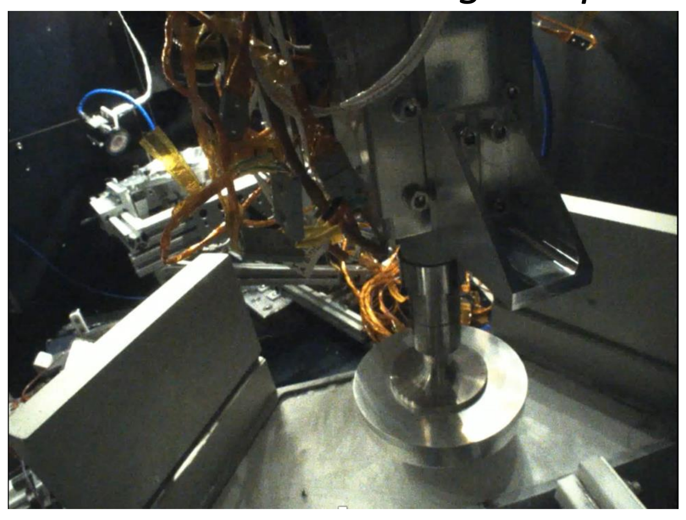
CITADEL Chamber



Background

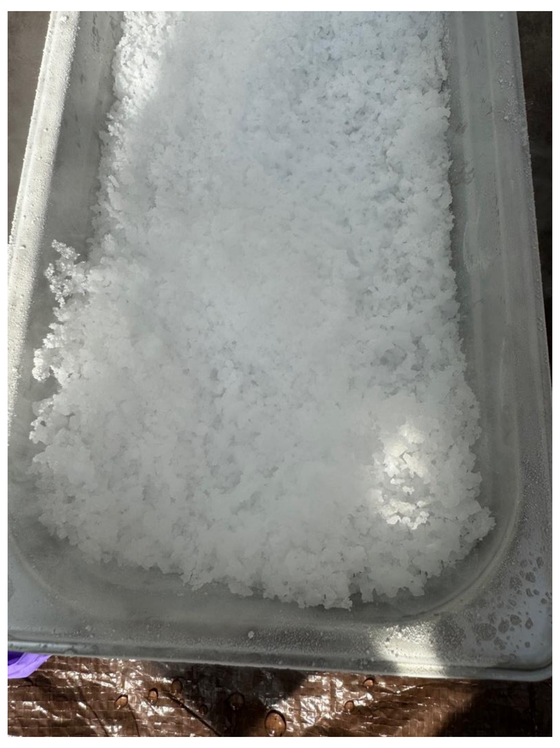
Icy Worlds are an ideal candidate to search for biologic indicators due to the existence of water. High sensitivity scientific instrument would be deployed to search for trace signs of past or present biology. To have confidence in these measurements, it is imperative for the science team to know that the collected samples have the same character and composition of the parent sample material, leading us to propose a sample integrity test campaign.

ICECAP tool collecting sample



Approach: Ice Sample Creation

Ice samples were precisely mixed with 2 salts in 1ppm concentrations. Water was then sprayed into a bath of liquid nitrogen to instantly freeze small ice particles to maintain chemical homogeneity. Granular ice was then loaded into trapezoidal CITADEL troughs and cold sintered in a -10C freezer before being stored in a -80C freezer.



Ice Sample Created by spraying into a bath of liquid nitrogen to create granular ice.

Approach: Sample Candidates

Due to the concern previously experienced over volatile samples, this test campaign selected 2 separate ionic salts at low concentrations to dope HPLC pure water for analysis. The two salts selected were $MgSO_4$, and KBr. The reasons for selecting 2 salts was to be able to exclude exogenous sources of the measured ions.

Approach: Sample Collection and Transfer

Samples were collected in the CITADEL chamber using the ICECAP rotary sampling tool. This tool then interfaced with the pneumatic transfer funnel and a "puff" of pre-chilled gas to transfer the collected ice into the glass vial. To ensure the successful transfer, the sample tube had to be directly plumbed into the pneumatic transfer line. Sample tubes were equipped with remotely operated valves to seal the tubes after the sample is transferred, to be removed from the chamber without contamination from re-pressurization, or from needing to warm the chamber.

Results

Collected ice samples extracted from the CITADEL chamber using the ICECAP tool were analyzed at the JPL Analytical Chemistry Lab using ion chromatography to measure the ionic species in the collected sample and was then compared to the pre-mixed water ice. 6 Ions were measured with 5 being detected in the collected ice, and 2 being detected in the doped ice. The Sulfate which was doped to a level of 1.7ppm, was initially measured at 30.4 ppm, decreasing with subsequent sampling to 4.1 ppm, and finally 3.9 ppm. The increase in the detected sulfate, and subsequent decrease in concentration is attributed to exogenous sulfate contamination in the sample transfer system.

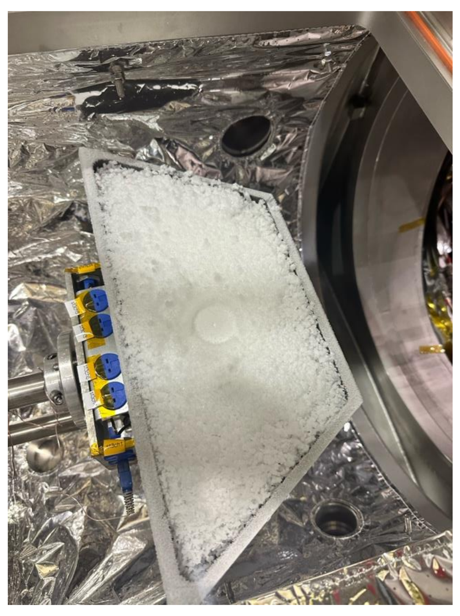
Conversely the bromide ions are measured at slightly elevated levels, 1.4 ppm at the first collected sample and maintaining concentrations (within experimental error) at 1.2 ppm, and 1.4 ppm through subsequent samples.

Other ions collected in the sample are also attributed to contamination sources in the sample transfer line and are common ions that are naturally found in aqueous systems.

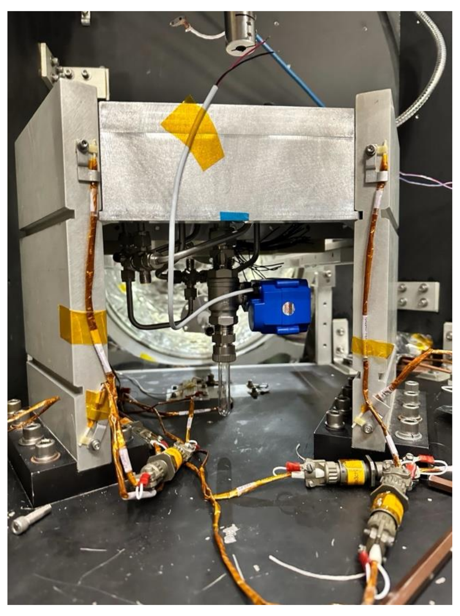
Sample (g)	"1.5"	1.5522	2.0211	1.2480
Anion (ppm)	Starting Water	Sample #1 (9/26/24)	Sample #2 (9/27/24)	Sample #3 (9/27/24)
Fluoride	-	0.15	0.14	0.15
Chloride	-	2.9	7.5	3.1
Sulfate	**1.7**	**30.4**	**4.1**	**3.9**
Bromide	**1.1**	**1.4**	**1.2**	**1.4**
Nitrate	-	1.2	0.64	0.34
Phosphate	-	-	-	-

Procedure

- Granular ice with chemical homogeneity was created doped with 1ppm of each Magnesium Sulfate, and Potassium Bromide.
- 3 Samples were collected using the ICECAP rotary sampling tool and transferred by pneumatic transfer system to glass sample vials in chamber.
- Sample vials were sealed by electrically activated ball valve and removed from the cryogenic chamber and disconnected from the pneumatic transfer line.
- Ice sample melts inside glass vial and is transferred to analytical chemistry lab for Ion Chromatography



Sample collected from granular ice



Sample transferred by pneumatics to vial

Vial with ice removed from cold chamber

Melted ice in vial measured by Ion Chromatography

Conclusions

Ice samples can be created, sampled and measured at JPL using state of the art facilities. This demonstration shows the ability to check the integrity of a collected sample for chemical changes as compared to the original material. This demonstration can be adapted to mission relevant sampling tools and scientifically relevant ice dopants.

Benefits to JPL and NASA

- Plans to use non-volatile organic molecules as ice dopant materials to prevent evaporation of collected samples.
- Modification to sample transfer and collection procedure to minimize the time the samples are exposed in air after being collected in-situ.
- Optimize sample tube design and in chamber monitoring for better visual confirmation of collected samples.

RPD-136 Clearance Number: CL#24-5689

PI/Task Mgr. Contact Information: Anthony Wong
818-354-9996, anthony.t.wong@jpl.nasa.gov