Preliminary Experimental Volatiles Recovery from Carbonaceous Asteroid Simulants

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EGBOCHE UNOBE

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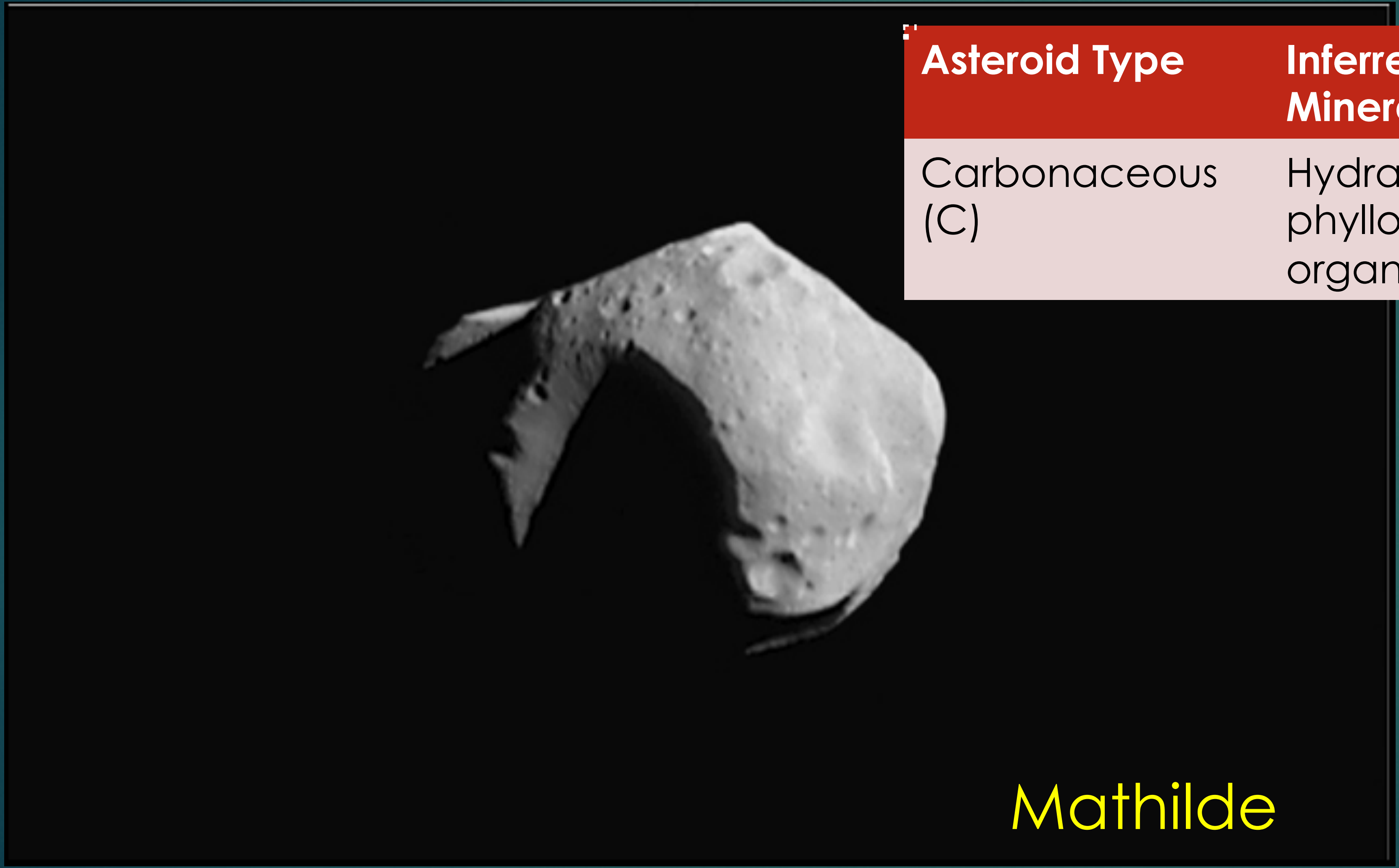
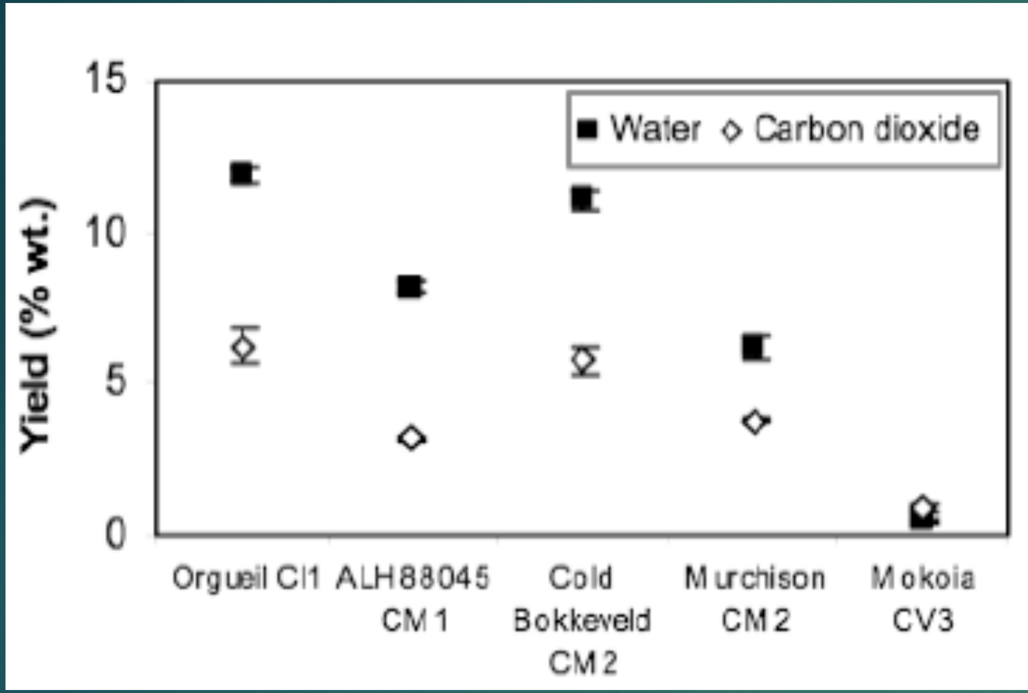
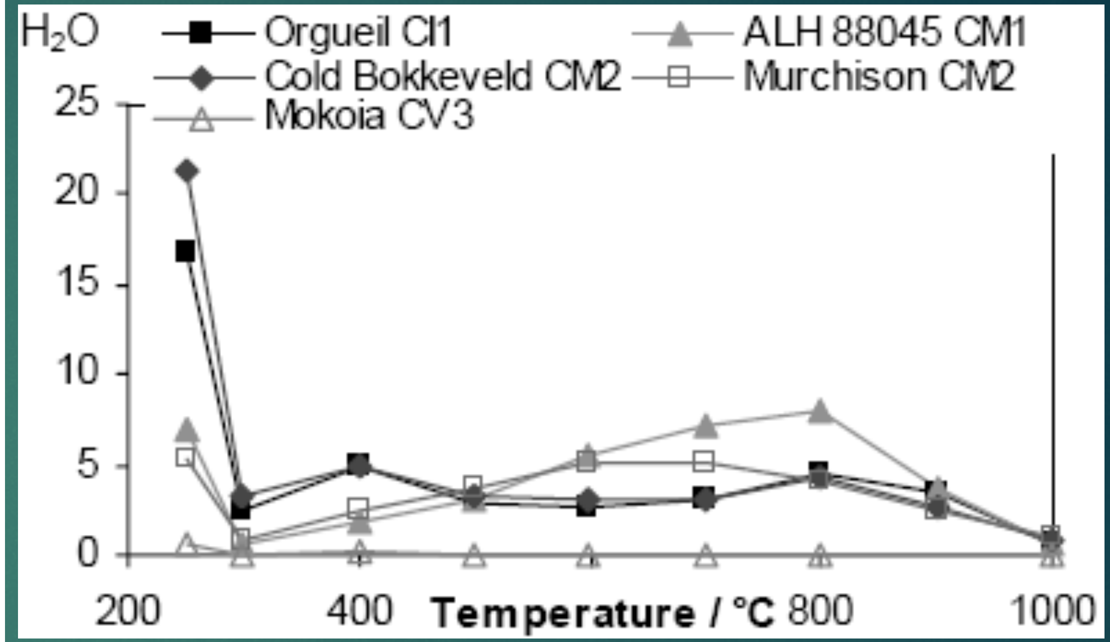
Background



Asteroid Type	Inferred Mineralogy	Expected Resources
Carbonaceous (C)	Hydrated phyllosilicates, organics	H_2O, CO_2, simple hydrocarbons

Background

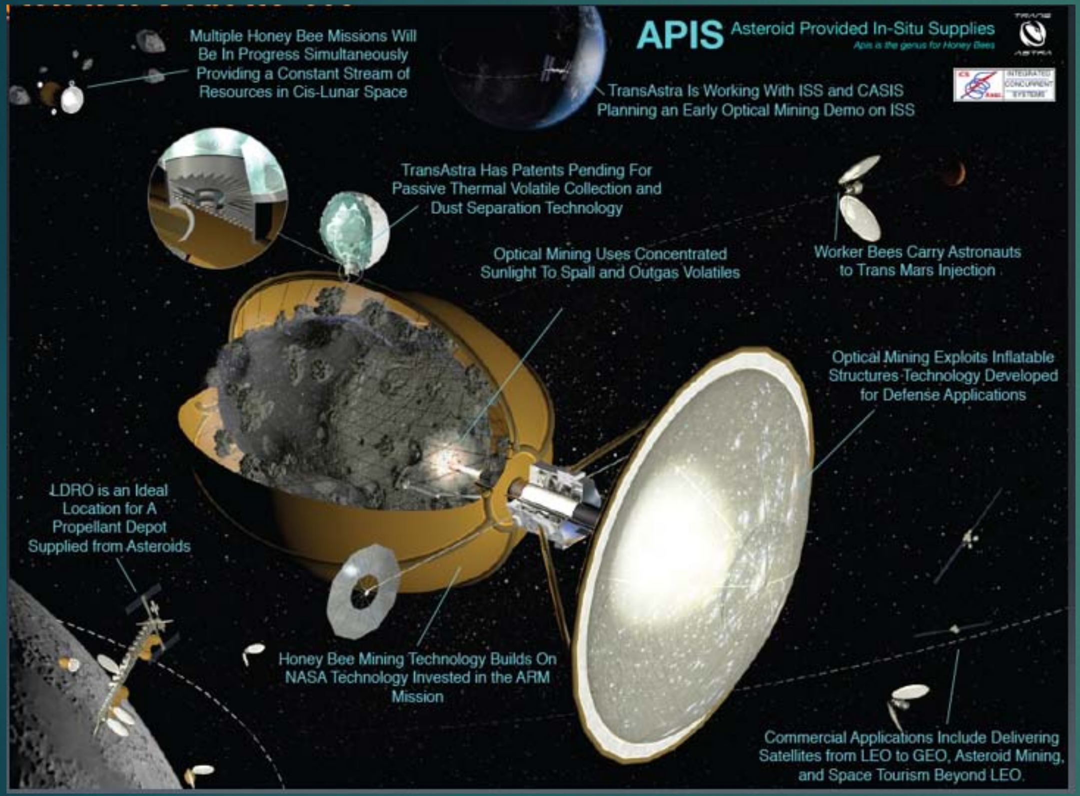




Court and Sephton (2009, 2011)

- Water and carbondioxide can be produced from hydrated mineral phases in Carbonaceous Meteorites by heating.
- The presence of such minerals on Asteroid bodies make them attractive as targets for volatile ISRU.

Background



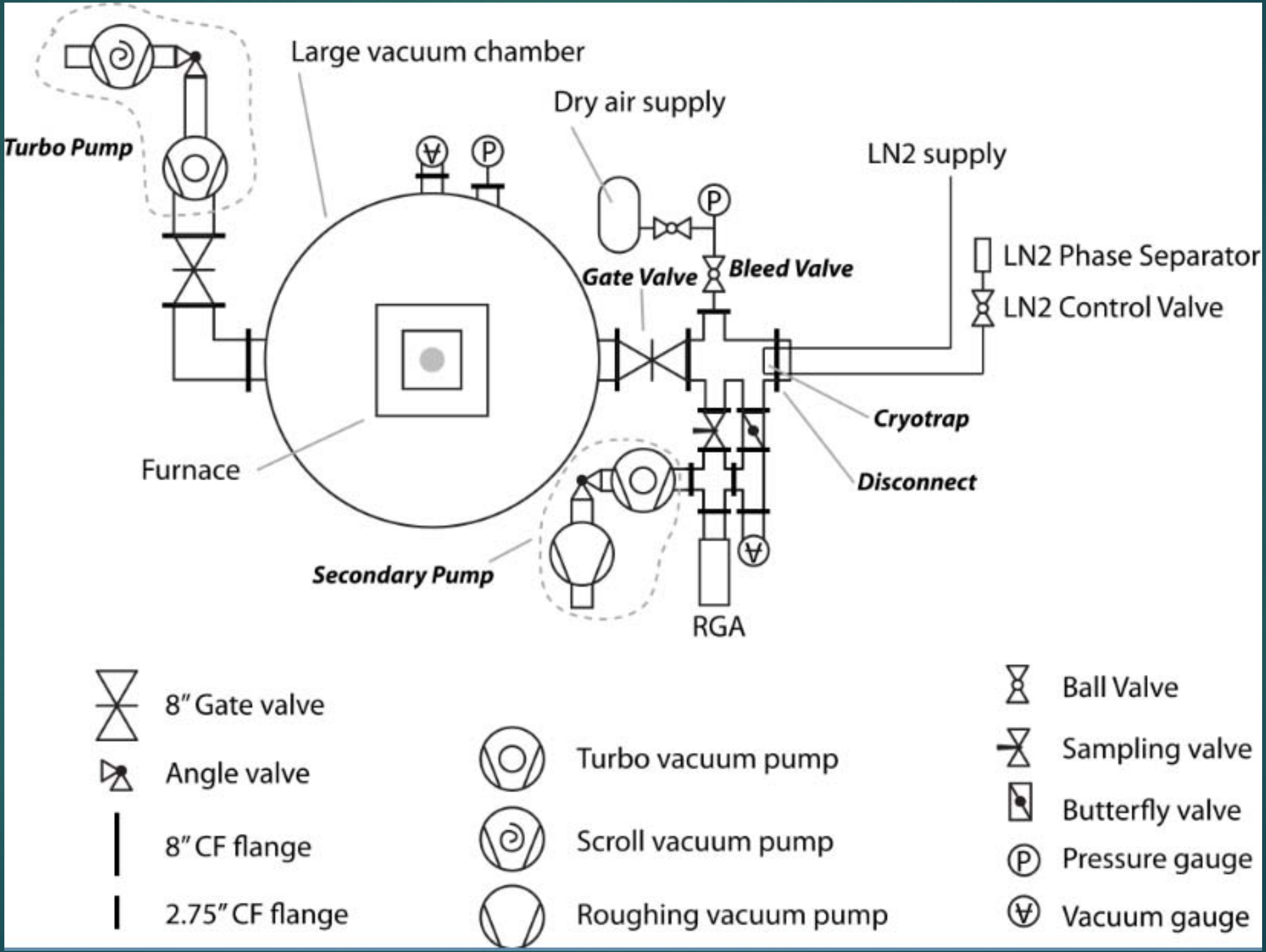
Asteroid Provided In-situ Supplies: ICS Associates

Planetary Resources Asteroid volatile mining concept.

Objective

- To develop greater understanding of the response of bulk volatile rich asteroidal minerals to radiative heating to enable effective asteroid ISRU as a means of supporting human activities in space.

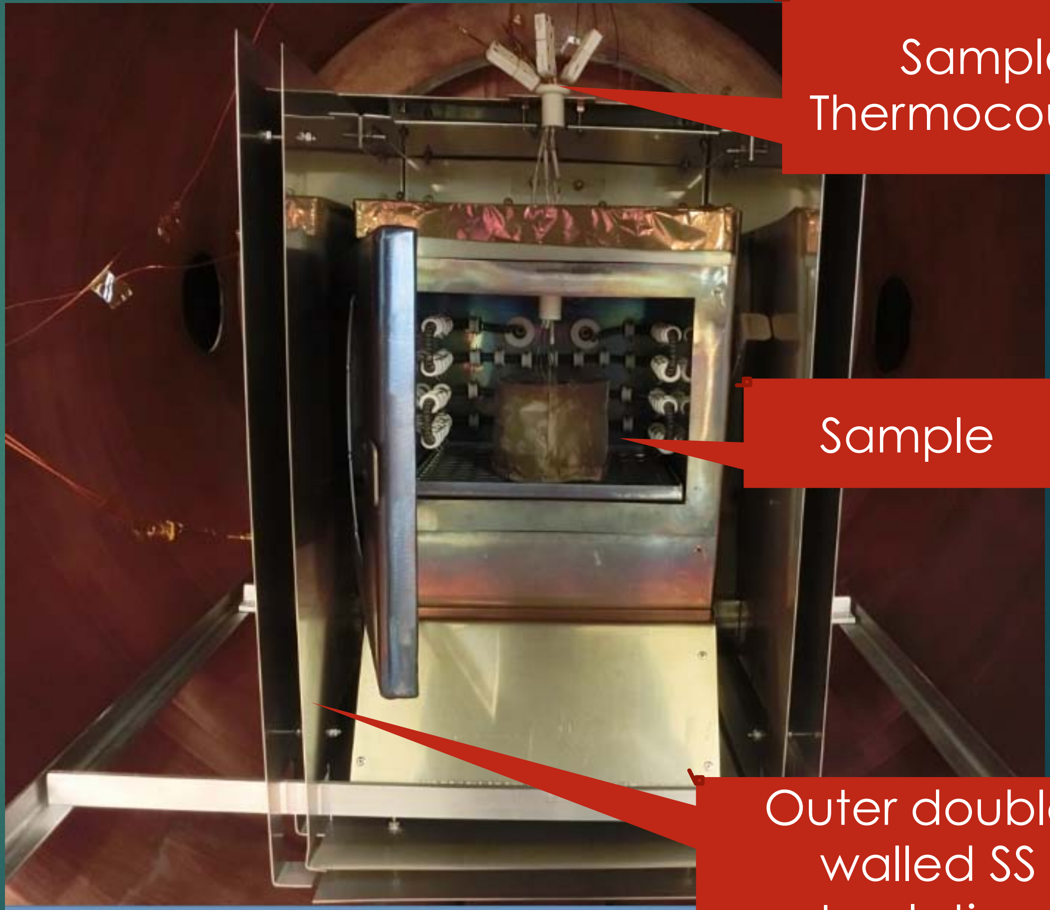
Laboratory System Schematic



Furnace Design

Original Furnace

Re-purposed Furnace

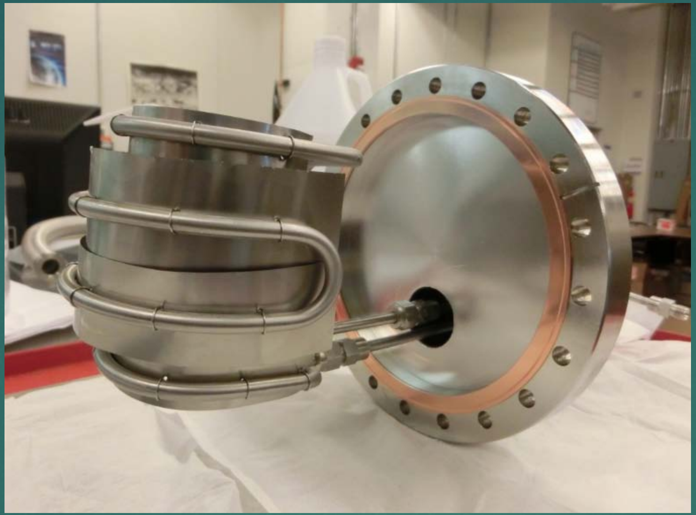


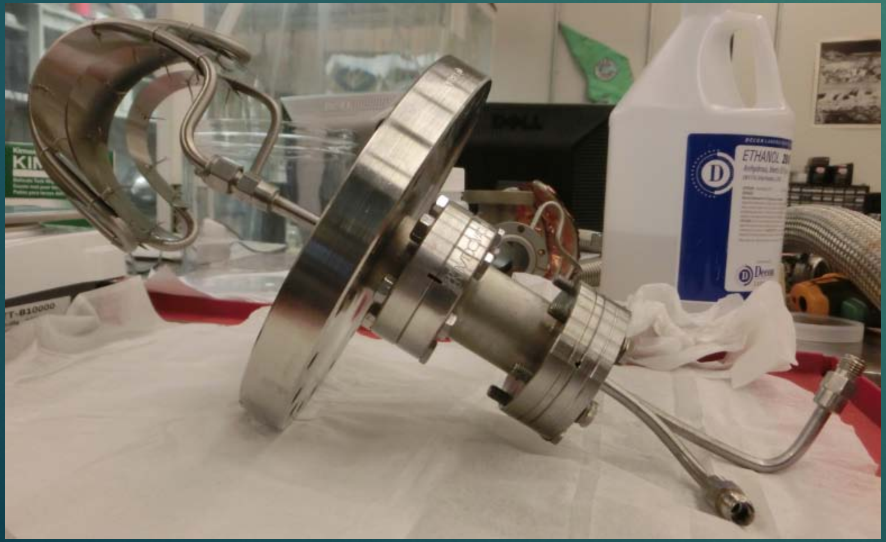
Sample Thermocouples

Sample

Outer double-walled SS Insulation

Cryotrap and Mass Spectrometer





LN$_2$ Inlet

LN$_2$ Outlet

MS Turbo Pump

MS Pressure gauge

Mass Spectrometer

Sample sets

Serpentine

- Major phase is Lizardite [$Mg_3Si_20_5(OH)_4$]
- A hydrated Magnesium phyllosilicate.
- Major hydrated phase on carbonaceous Asteroids.
- Detectable impurities includes carbonates.

Olivine

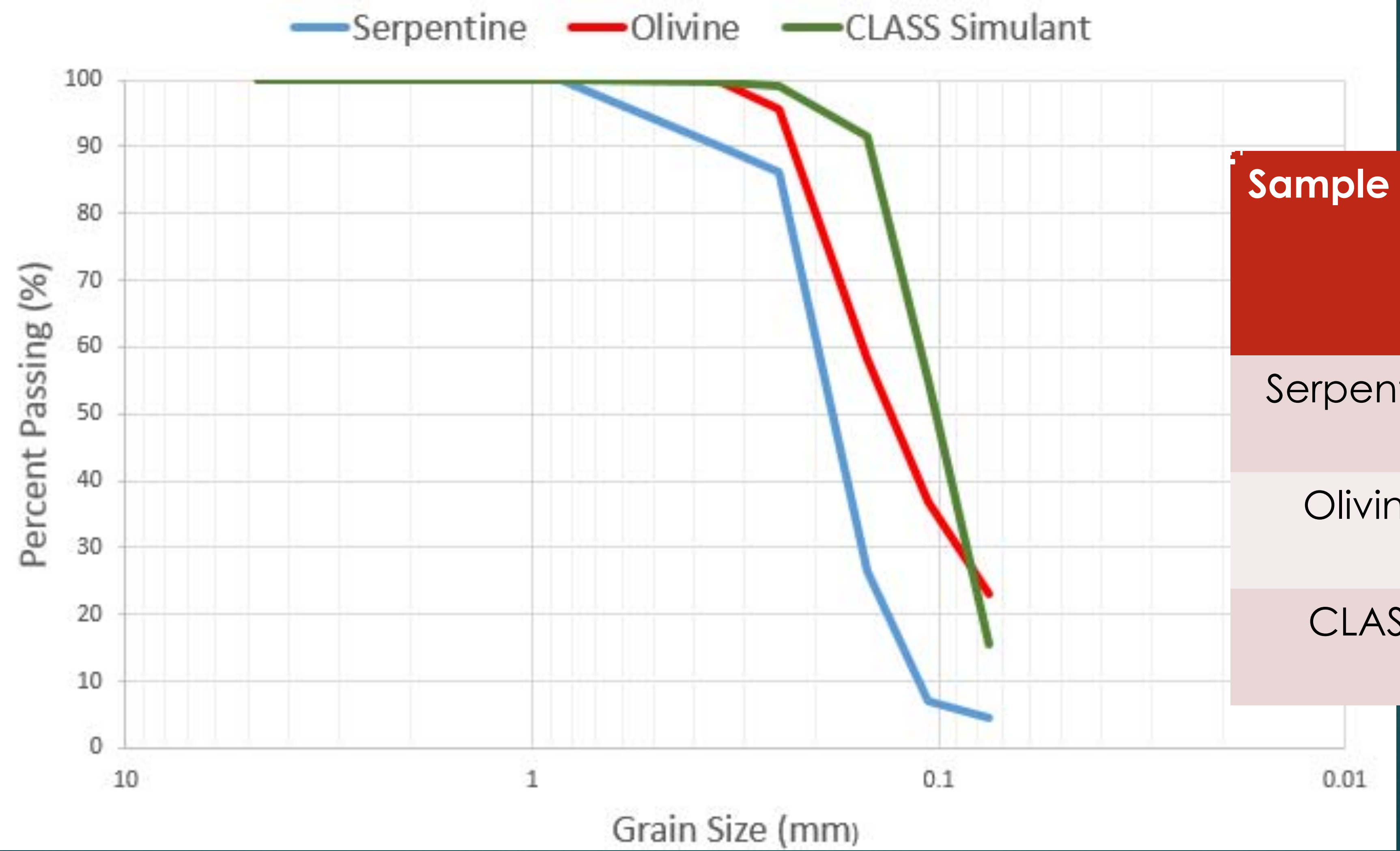
- Major phase is Forsterite [Mg_2SiO_4]
- Anhydrous Magnesium silicate
- Non-hydrated phase on carbonaceous Asteroids
- Detectable impurities include phyllosilicates

Center for Lunar and Atmospheric Surface Science (CLASS) Simulant

Mineral	Weight (%)
Serpentine	48
Olivine (Fo 90)	7.0
Magnetite	13.5
Vermiculite	9.0
Pyrite	6.5
Epsomite	6.0
Smectite	5.0
Sub-butiminous Coal	5.0

- "CLASS Simulant" nominal Composition (Britt, 2016; personal communication)
- Designed to simulate the Orgueil-type Carbonaceous Chondrite Meteorite

Grain Size Distribution



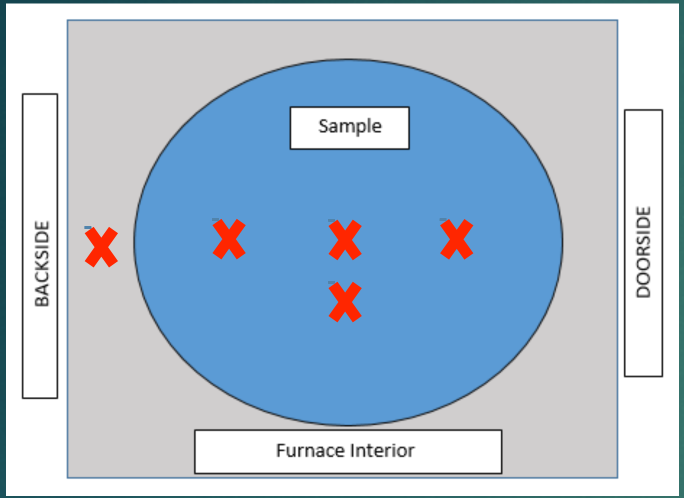
Sample type	Median grain diameter (D_{50}) (µm)
Serpentine	190
Olivine	140
CLASS	110

Sample Preparation

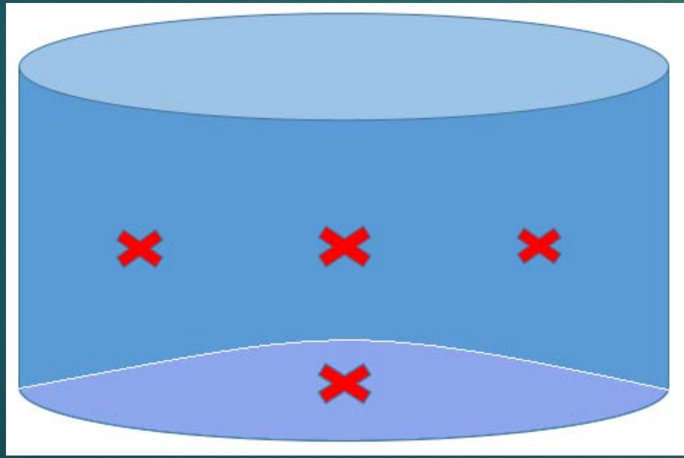
- Sample Size: 700 – 1000 grams
- Diameter: 3 - 4 Inch
- Height: 3 – 4 inch

✖ :Thermocouple location

A)



B)



Sample Mesh Container

A) Plan View B) Longitudinal View

Sample sitting in furnace with TCs going in through the top.

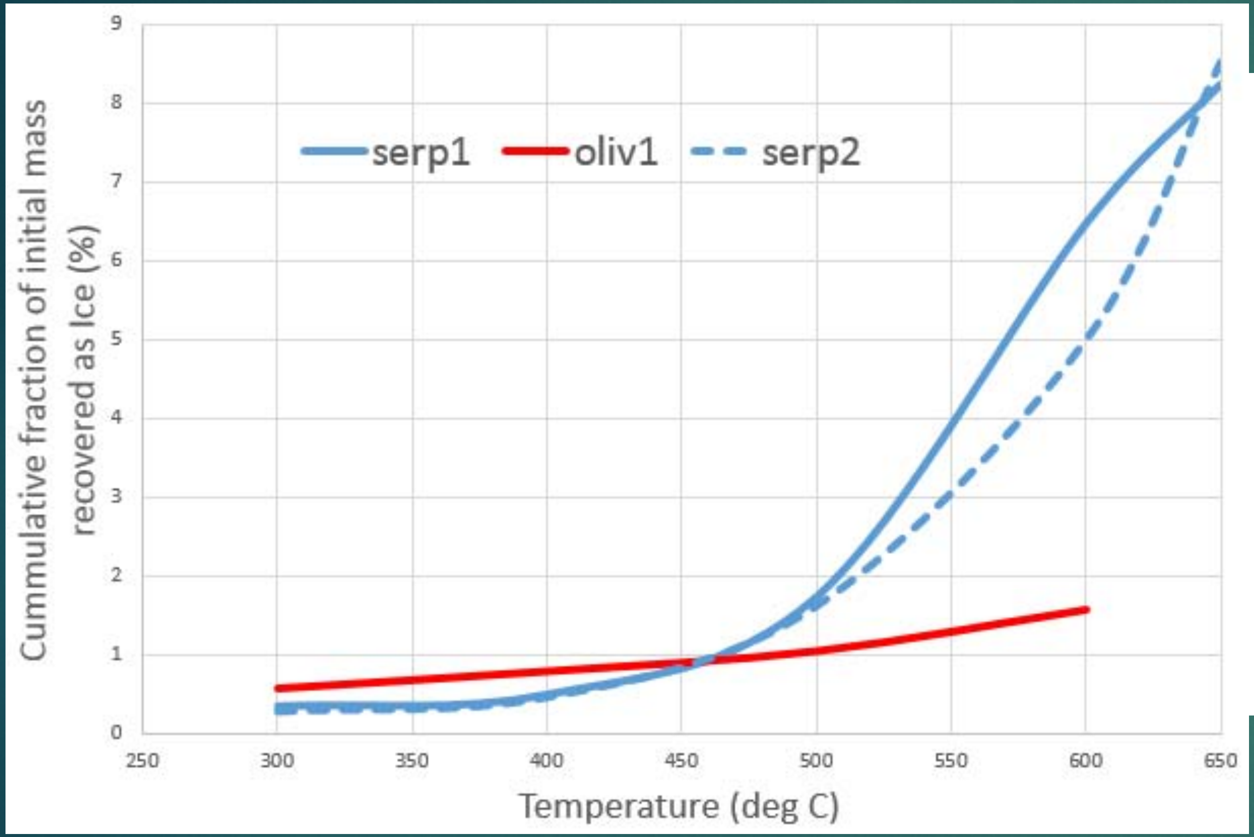
Experimental Procedure

- Sample Preparation (custom mesh, Handling, weighing)
- System Preparation and characterization (Pumpdown and bake-out, leak-up)
- Test run starts; sample is placed in chamber, system is pumped down to base pressure of ~5E-5 torr, 5 temperature plateaus to be held (300 ^{0}C, 400 ^{0}C, 500 , 600 ^{0}C and 650 ^{0}C), LN flowing through cryotrap, Mass spectrometer is sampling atmosphere, data is acquired on DAQ.
- Each plateau is held for at least 3 thermal time constants and until there is a significant drop in volatile production calculated from the mass spectra of the chamber atmosphere
- Trapped Ice is weighed and sampled at end of each temperature plateau.
- Post-test weight measured; value compared to pre-test weight and to weight of Ice collected.

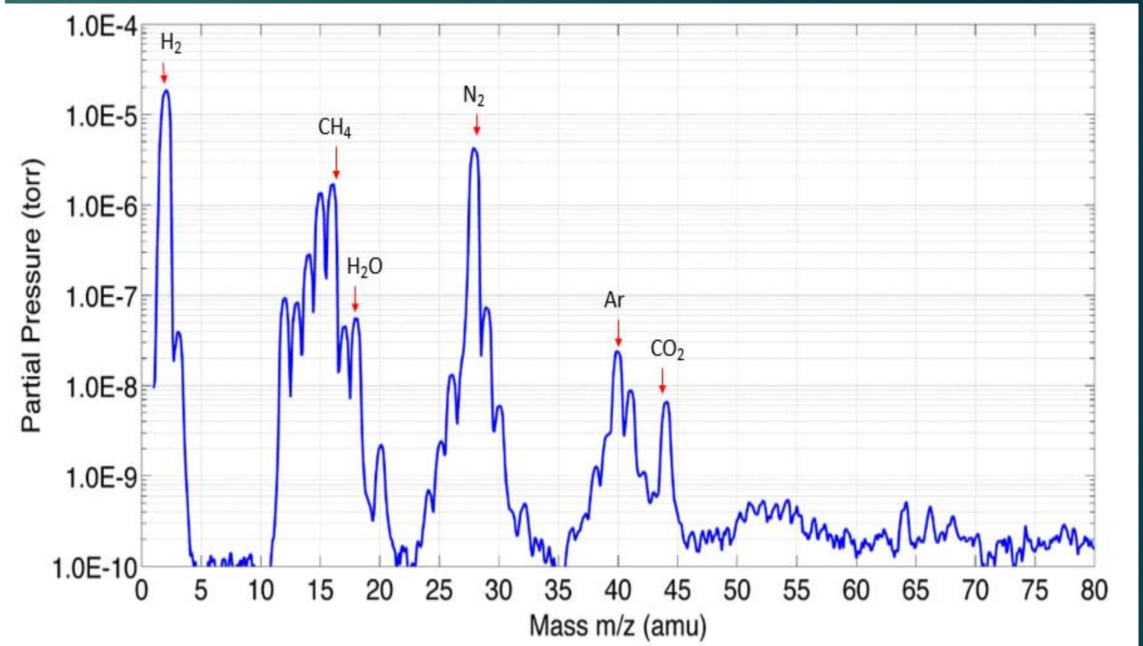
Preliminary Results

Samples Tested	Sample ID	Initial Sample Mass (grams)	Mass Loss after heating (grams)	Total Mass of Ice Trapped (grams)	Effective Recovery (%)	Major Volatile Species Produced
Serpentine	serp1	1019.8	80.6	78.0	96.7 %	H_2O
	serp2	717.8	64.5	61.2	94.8 %	H_2O
Olivine	oliv1	783.4	12.4	11.9	95.9 %	H_2O

Mass fraction of Ice Recovered, volatile species produced



Mass Fraction of Ice Vs Temperature

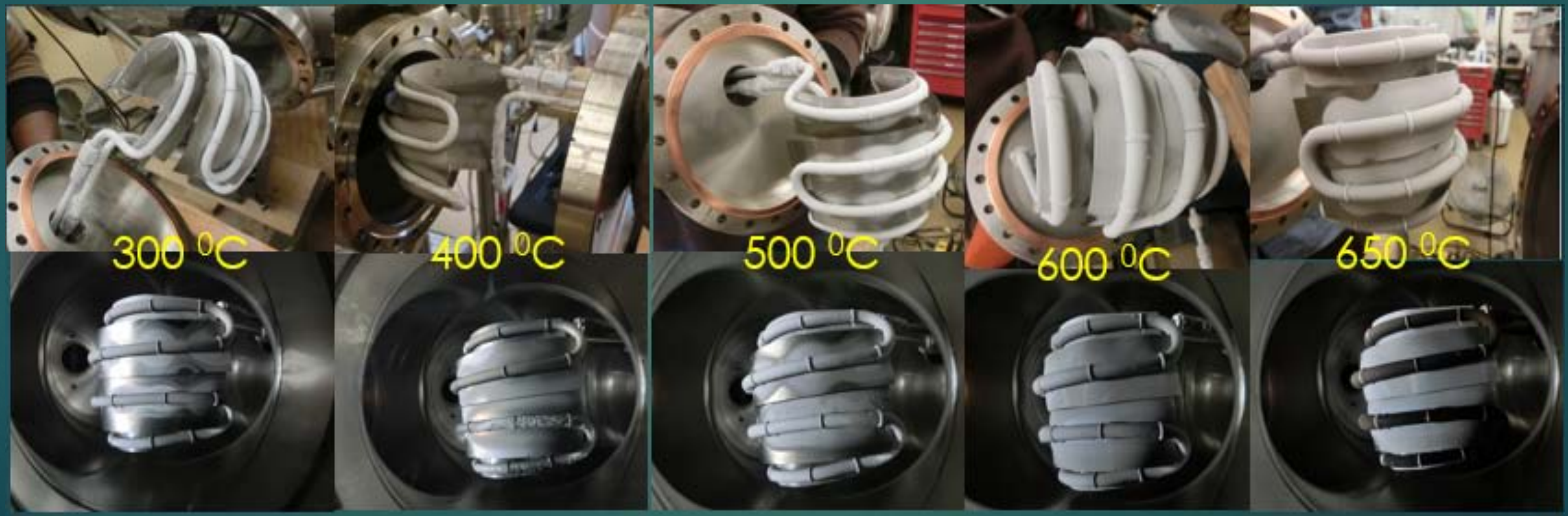


RGA Spectrum showing evolved gases within the vacuum system during tests.

Ice Collected

serp1

serp2



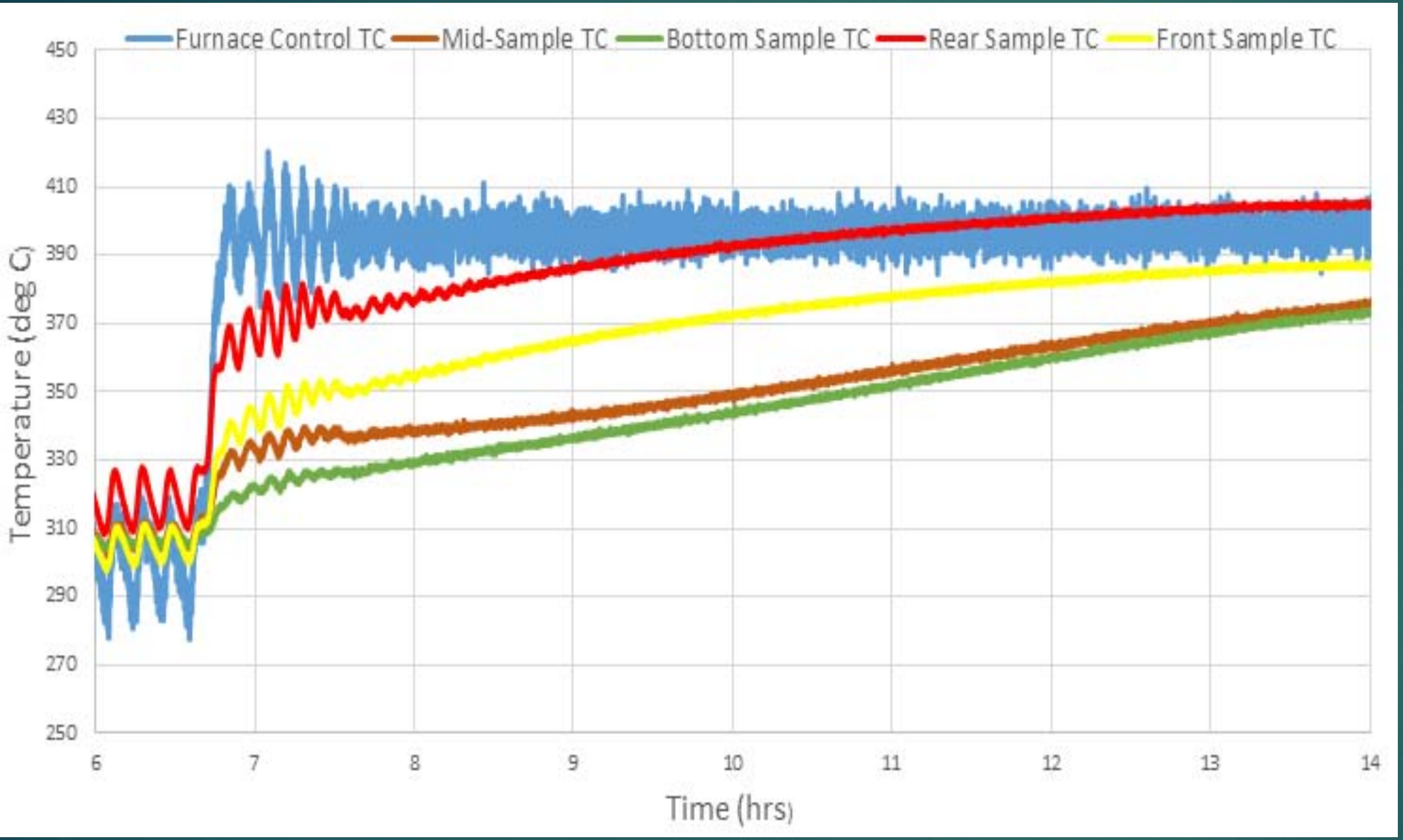
Operating in Vacuum

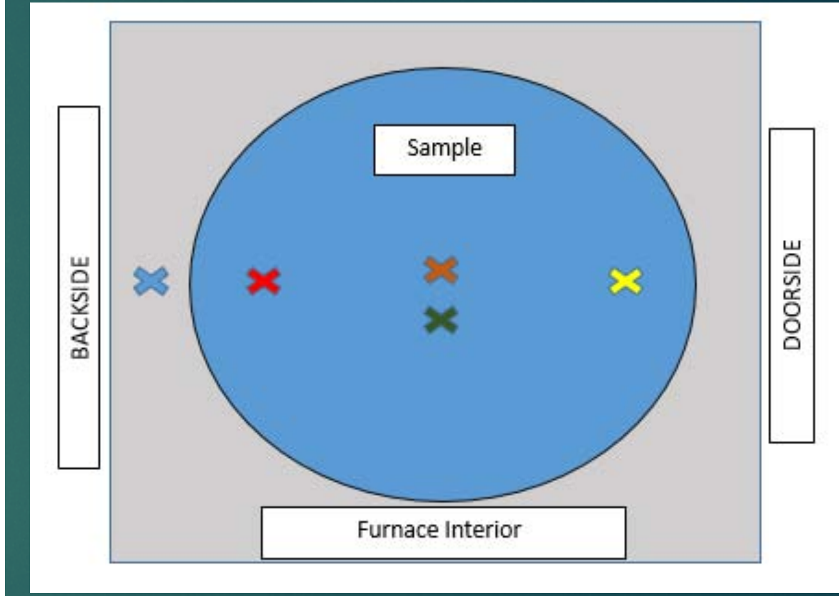
Removed, weighed & sampled

Removed, weighed & sampled

Operating in Vacuum

Characteristic heat profile in samples





serp1 at 400 ^{0}C

Preliminary Findings

- The cryotrap operates at Liquid nitrogen temperature and has proved capable of recovering H_2O, CO_2, and CH_4 at varying efficiencies.
- Overall heat input and heat transfer through the material would determine the pace to complete de-hydroxylation of bulk granular serpentine minerals
- Significant volatile production from serpentine minerals begins at the 500° C plateau and increases in rate at higher temperatures.
- The yield of desired products such as water may be affected by chemistry in the gas phase if total pressure in the system is allowed to build up from produced volatiles; a way to mitigate this is by continuously trapping.
- The likely chemical pathway to water formation from the hydroxyl group minerals under these conditions can be represented by the form;

$$H + OH \longrightarrow H_2O$$

$$H + H \longrightarrow H_2$$

Alternate pathway leading to the formation of Hydrogen gas.

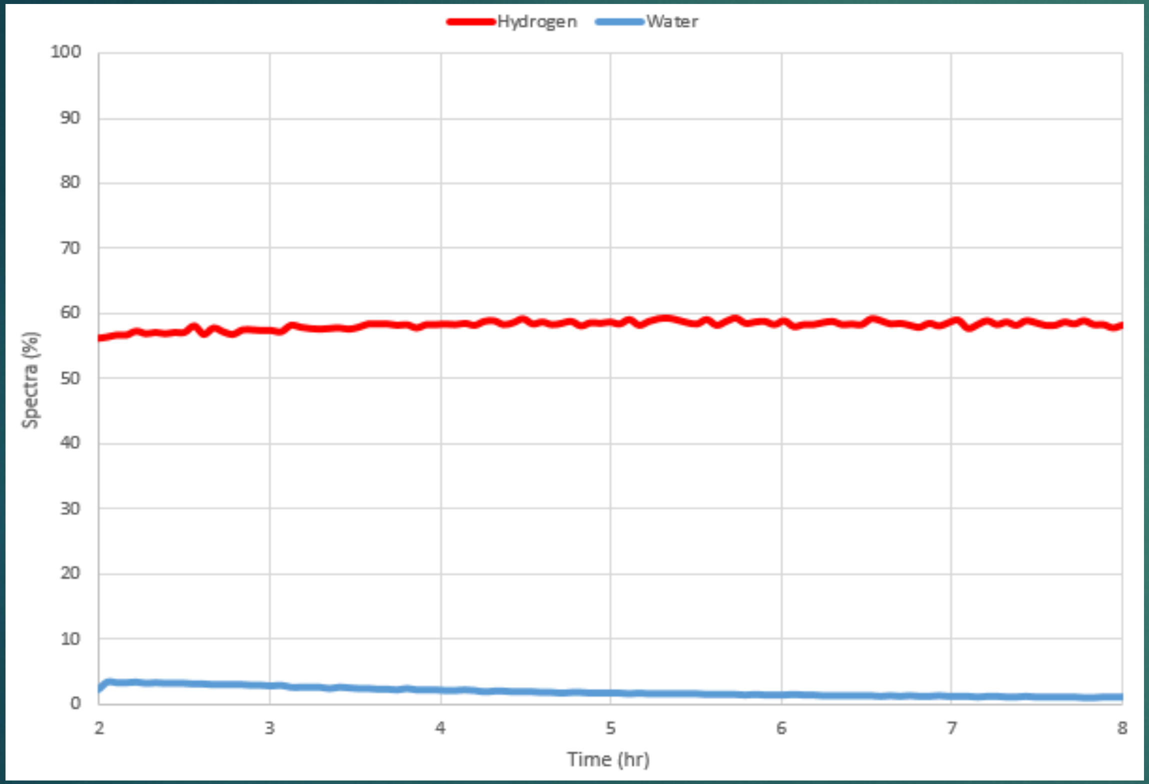
Moving Forward

- Complete experiments on Serpentine, Olivine and CLASS simulant.
- Complete before and after indentation tests on samples.
- Analyze sampled Ice
- Outline practical limitations to the extraction of volatiles from carbonaceous bodies.
- Complete and compare Equilibrium model of reaction process with actual results obtained.
- Apply validated model to simulate full scale application in space to predict yields and performance of various ISRU methods.

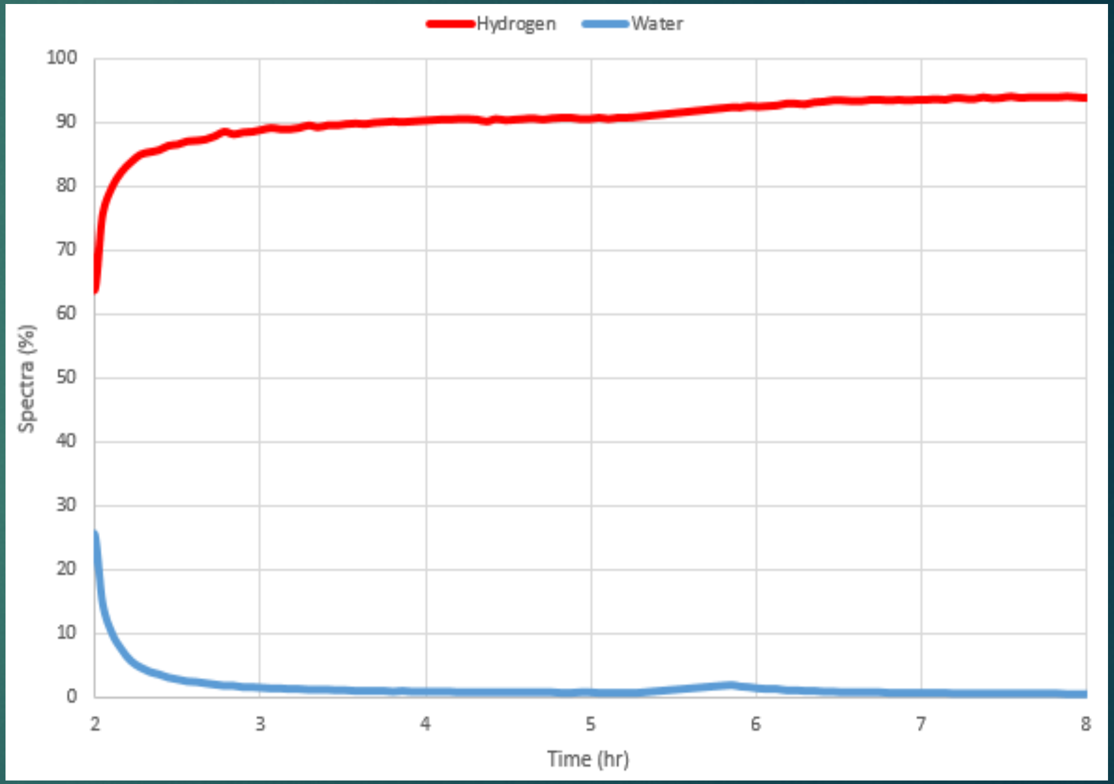
QUESTIONS?

BACK-UP

High Hydrogen Atmosphere as a marker for Serpentine De-hydroxylation

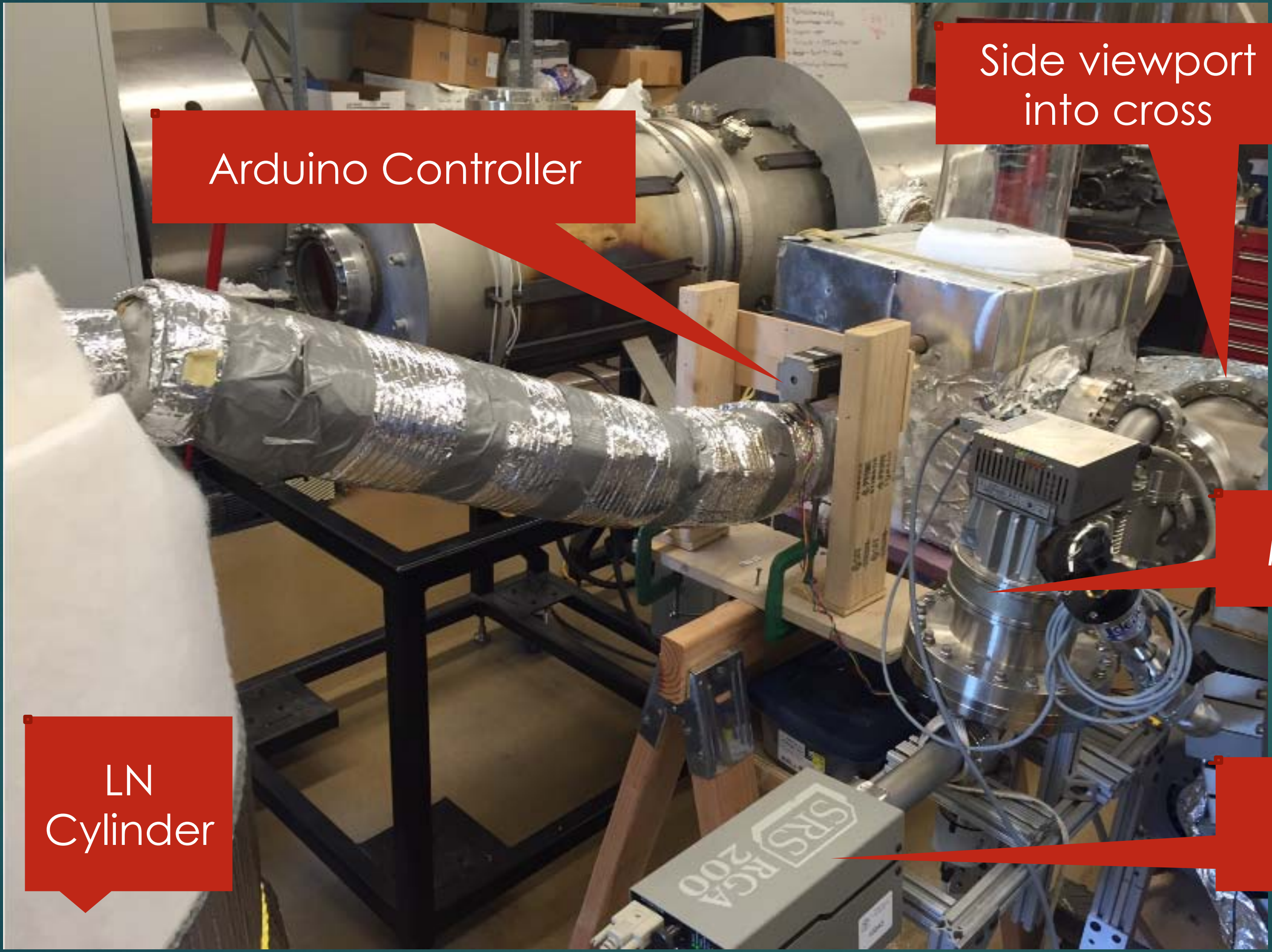


- Serpentine: 300 ^{0}C Plateau
- Spectra: ~ 60% Hydrogen

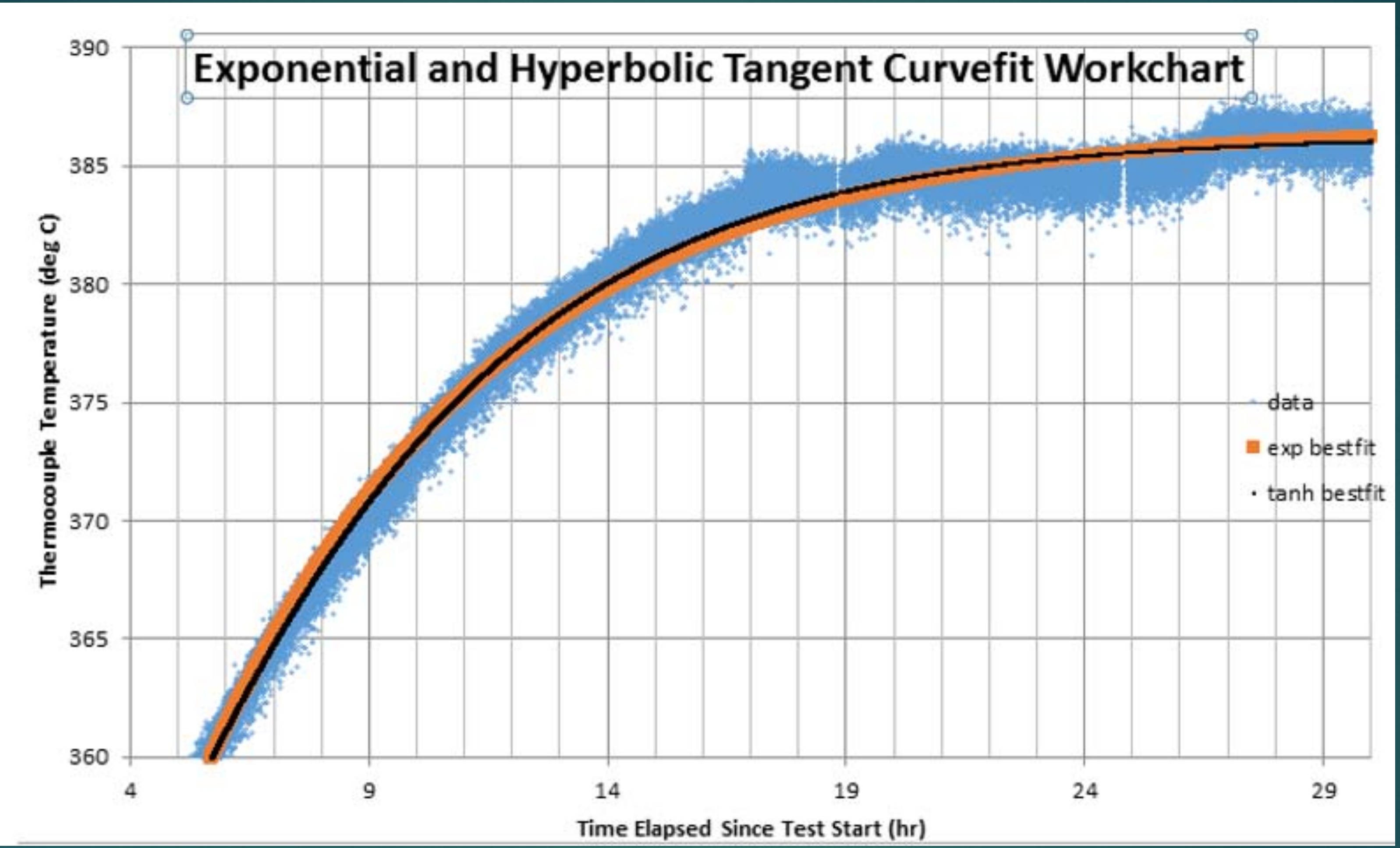


- Serpentine: 600 ^{0}C Plateau
- Spectra: ~ 90% Hydrogen

Cooling System & Mass Spectrometer



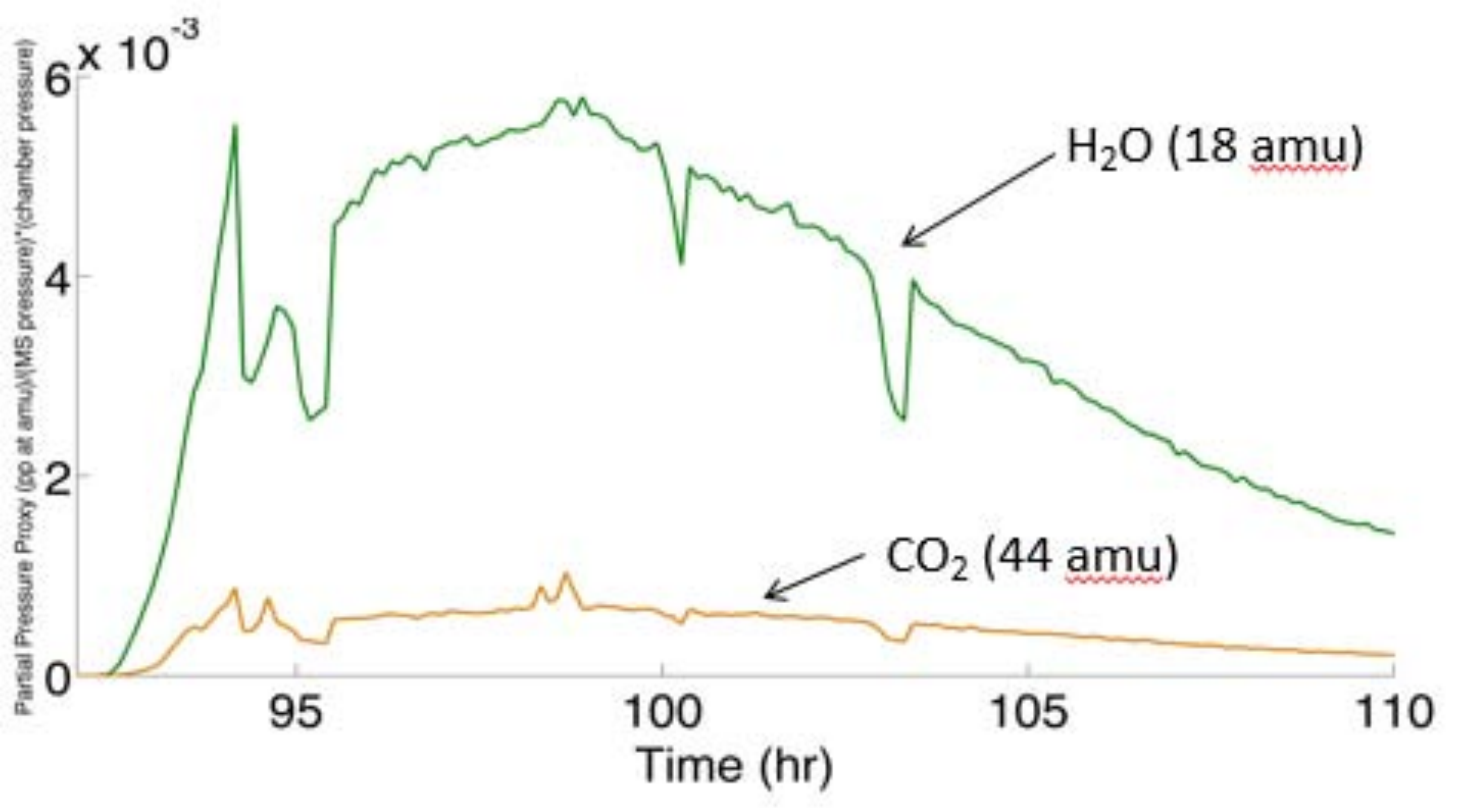
View from side port; cryotrap operating in vacuum (600 ⁰C)



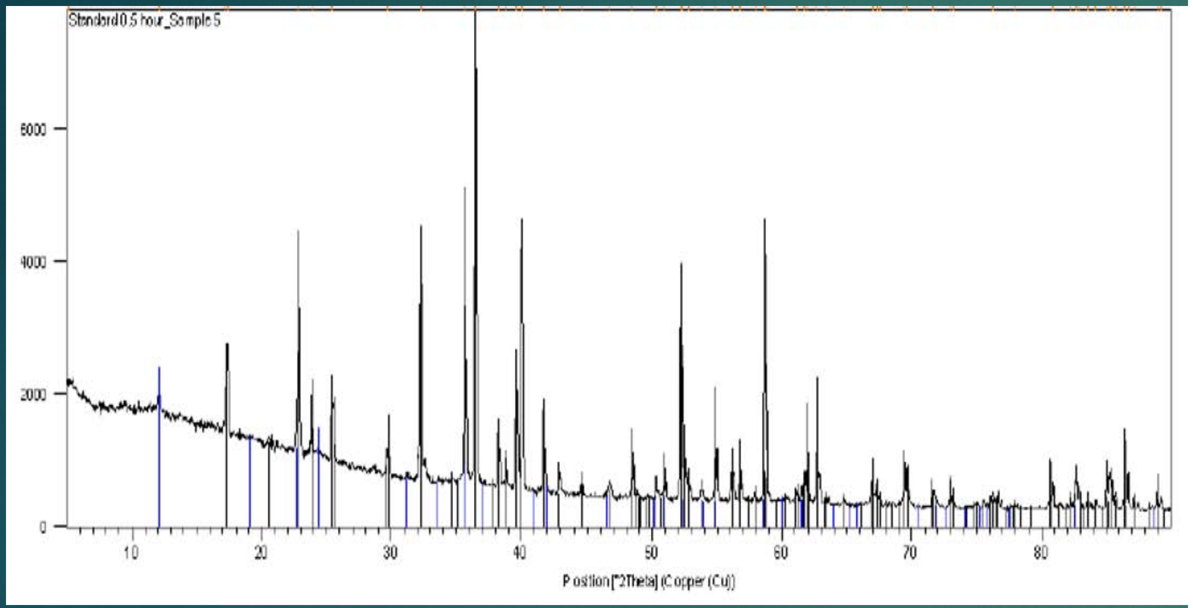
The thermocouple plateau duration is **greater than or equal to 3x the longest thermal time constant of the 4 sample thermocouples**, derived independently from all sample thermocouple readings (starting after initial transients have died out.

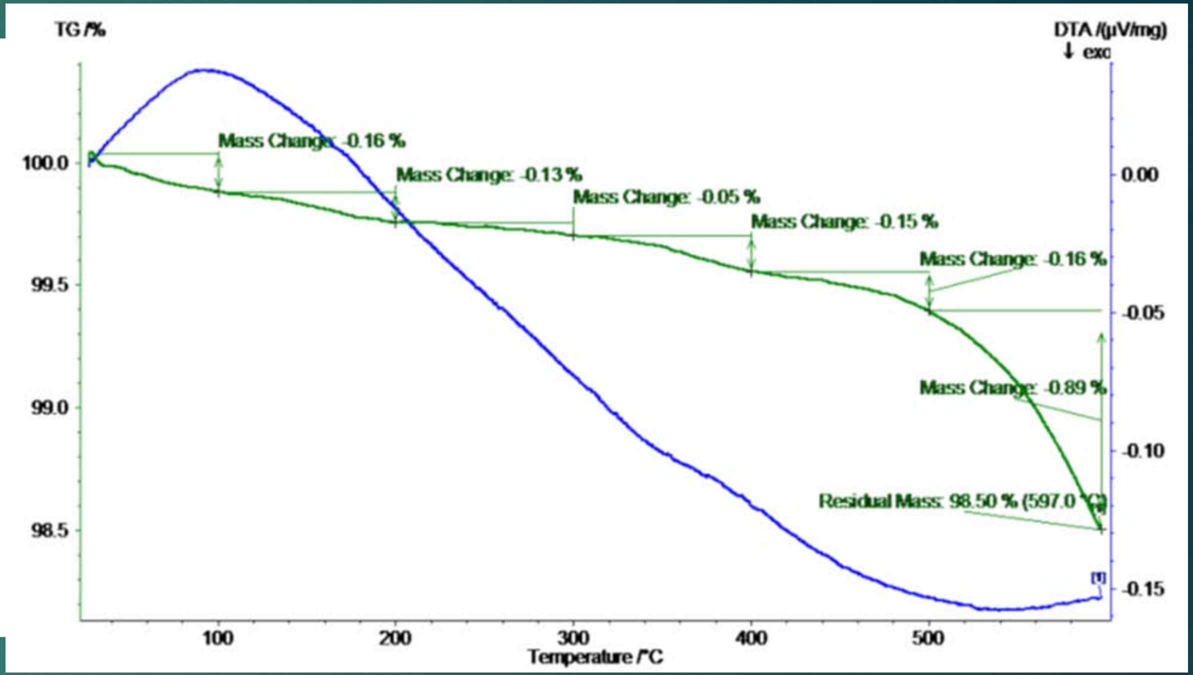
Ending a Plateau

- Gas production rate (proxied by the **water partial pressure** of the chamber atmosphere) is significantly lower than the peak (i.e., down 2 orders of magnitude from the peak).



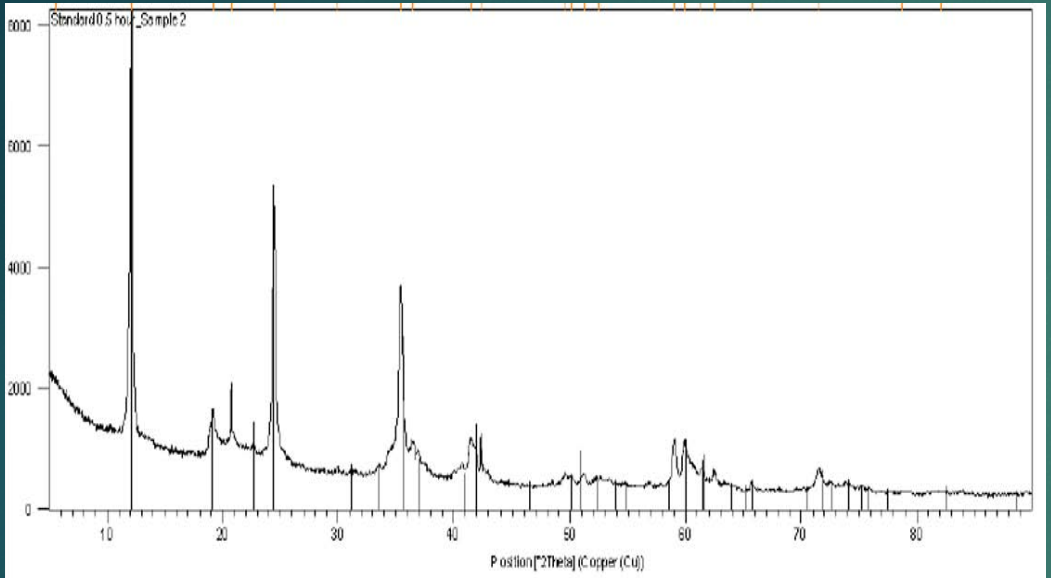
Forsterite XRD & TGA

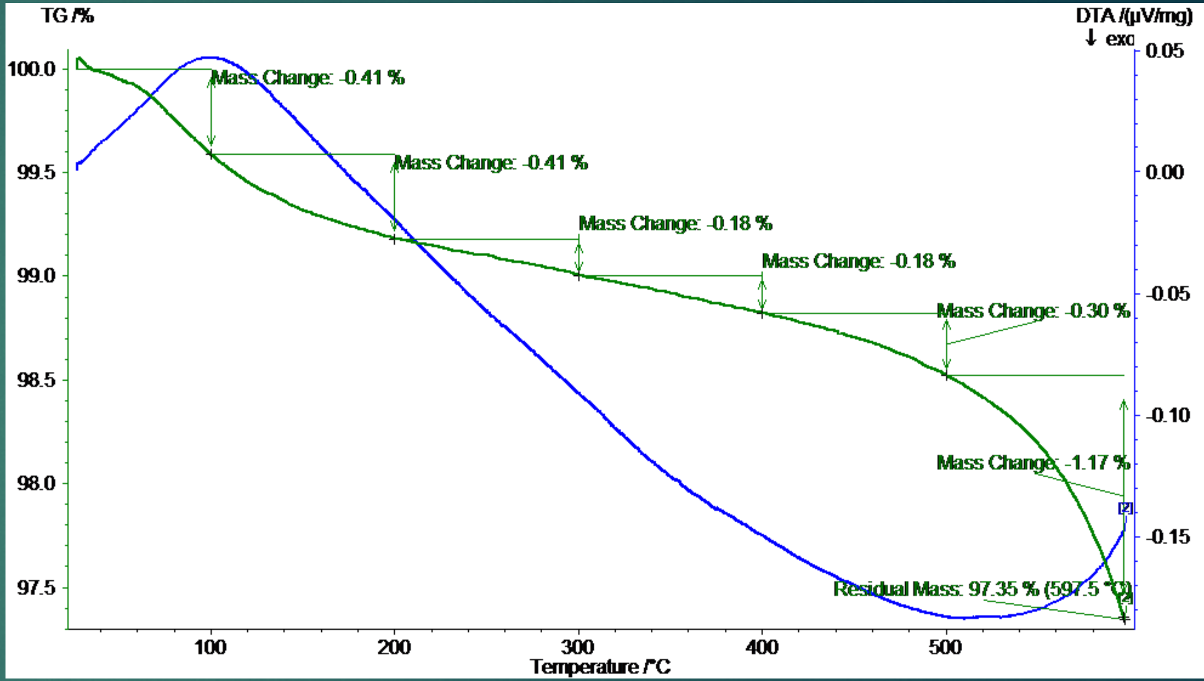




Olivine; major phase is Forsterite $Mg_2 SiO_4$

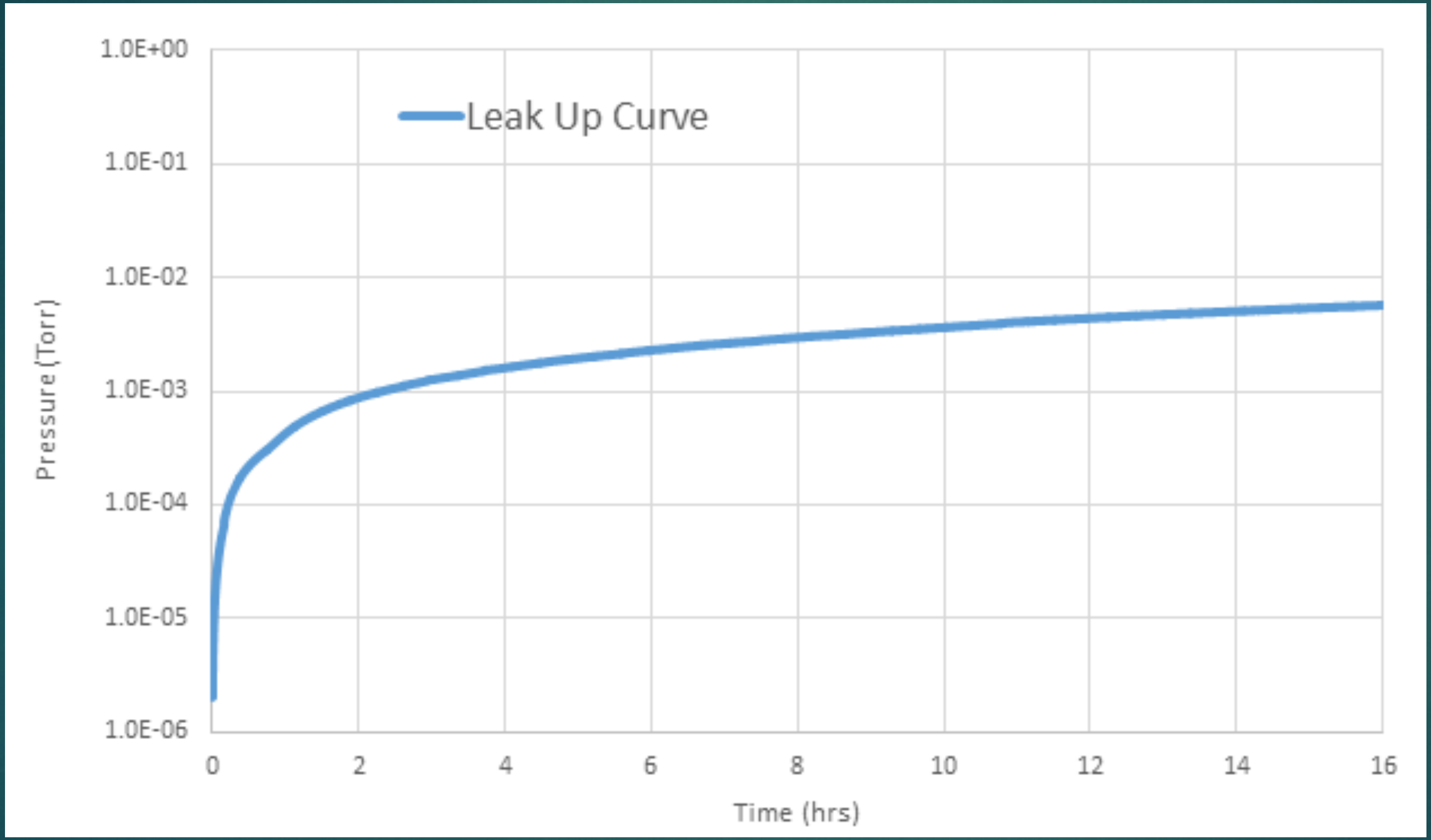
Lizardite XRD & TGA





- Serpentine; major phase is Lizardite [$Mg_3Si_20_5\ (OH)_4$]

Leak-up Test, Characteristic vacuum system background spectra



Gas Specie	Weight %?
Hydrogen	55.9
Nitrogen	15.5
Carbon dioxide	6.7
Water	8.1

Serp2 Ice