

## **COLD AND CRYOGENIC CURATION OF LUNAR VOLATILE SAMPLES RETURNED TO EARTH.**

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**Introduction:** The study of volatile compounds and volatile elements, such as H, He, C, N, O, H<sub>2</sub>O, CH<sub>4</sub>, SO<sub>2</sub>, CO, CO<sub>2</sub>, NH<sub>3</sub>, HCN, etc., are commonly used for constraining evolutionary processes on planets, satellites, and asteroids, as well as formulating models of solar system formation. For Lunar science, the recent evidence of regolith and rocks containing small amounts of OH<sup>-</sup> and/or H<sub>2</sub>O has renewed scientific interest into the study of lunar volatiles [1, 2]. Future lunar sample return missions will include the study of volatiles as a high priority.

Comet particles from the Stardust mission, asteroid particles from Hayabusa, meteorites, and subsurface lunar samples all occupied subfreezing environments prior to collection. Valuable geochemical information on volatiles is often lost when these samples are allowed to reach ambient temperatures on Earth. The ability to store, document, subdivide, and transport extraterrestrial geologic samples while maintaining below freezing or cryogenic temperatures is required for the complete scientific study of such samples, as well as future samples from a wide range of solar system bodies.

**Lunar Temperatures:** Recent Lunar Reconnaissance Orbiter (LRO) Diviner radiometer observations have shown that cold traps in the polar regions of the moon have temperatures as low as 38 K [3]. Diviner data also estimates that the lunar surface (in the top 2 cm), daytime temperatures range from about 180 to 300 K and 60 to 120 K in semi-shaded areas [3]. Nighttime observations show an estimated temperature range from 38 to 90 K, where 38 K is in permanently shadowed craters [3]. Therefore, volatiles in regolith samples that would be collected and returned to Earth might need to be maintained at extremely low temperatures to fully preserve their scientific integrity.

**Curation at 250 K:** Subsurface lunar regolith samples collected and preserved at 250 K could contain ice and solar wind derived volatiles. Returning such samples from the lunar surface could require having the sample return capsule outfitted with several sample containers situated inside a freezer that would survive a reentry. Curation at 250 K on Earth could require that the samples be handled inside an insulated glovebox with an inert gas environment. An alternative option would

be a glovebox placed inside a walk-in freezer [4]. Storage of the samples would be in commercial freezers with redundant systems.

**Curation at 40 K:** Samples from extremely cold environments, including the lunar polar cold traps, could require curation at temperatures as low as 40 K. For cryogenic samples returning to Earth, a combination of passive cooling during cruise and active cooling during and after reentry might be required. Once on Earth, the sample return container could be placed into a helium shroud cryopump vacuum chamber. Cryocontainment would be maintained through delivery to a cryogenic chamber in the curation facility. This 40 K thermal vacuum chamber would include cameras and robotic manipulators for preliminary examination, subdivision of samples, and specialized sample allocation containers for shipment to laboratories. Cryogenic curation is feasible with current technologies developed for the superconductor industry. However, significant research and development costs would be required to tailor these technologies to the task of sample return and long term curation of lunar volatile samples at 40 K.

**Future Cold / Cryo Curation:** With over four decades of scientific investigation, the Apollo sample collection has given the science community the ability to study lunar materials with highly precise measurements made in multiple laboratories. High-resolution studies of lunar volatiles will require a sample return mission where cold or cryogenic curation preserves the scientific integrity of these fragile samples.

**References:** [1] Pieters, C.M. et al. (2009) *Science*, 326(5952): 568-572. [2] Anand, M. (2011) *Earth, Moon, and Planets*, 107(1): 65-73. [3] Paige, D.A. et al. (2010) *Science*, 330(6003): 479-482. [4] Herd, C.D.K. et al. (2011) *The Importance of Solar System Sample Return Missions to the Future of Planetary Science Workshop*, Abstract# 5029.

# Cold and Cryogenic Curation of Lunar Volatile Samples Returned to Earth

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(Background LRO Image: Terraced Craters in Aitken Crater)

# The Importance of Volatile Compounds and Elements

H, He, C, N, O, H<sub>2</sub>O, CH<sub>4</sub>, SO<sub>2</sub>, CO, CO<sub>2</sub>, NH<sub>3</sub>, HCN, etc., are commonly used for constraining evolutionary processes on planets, satellites, and asteroids, as well as formulating models of solar system formation.

Inherently, valuable geochemical information on volatiles are often lost when samples returned to Earth are allowed to reach ambient terrestrial temperatures and pressures.

Current sample return collections that occupied subfreezing environments prior to collection:

- ❖ Subsurface Apollo Lunar samples
- ❖ Comet particles from the Stardust mission
- ❖ Asteroid particles from Hayabusa
- ❖ Some meteorites (e.g. ANSMET, Tagish Lake, etc.)

## Lunar Daytime Temperatures:

180 to 300 K (about top 2 cm)

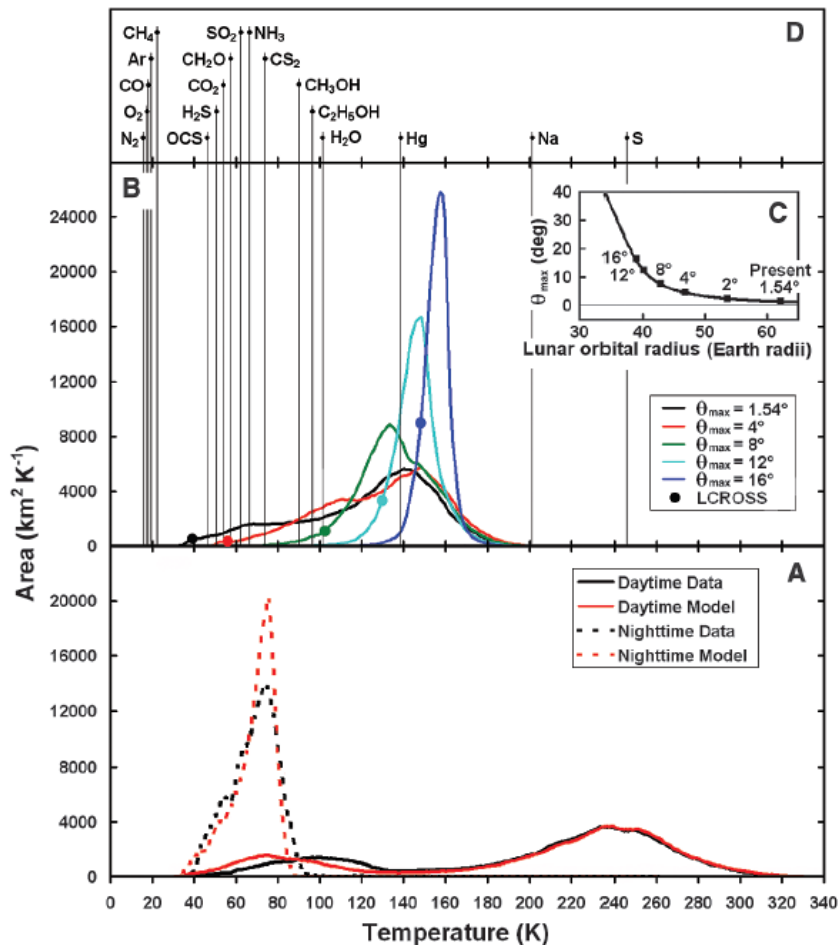
60 to 120 K (semi-shaded areas)

## Lunar Nighttime Temperatures:

38 to 90 K

(38 K permanently shadowed areas)

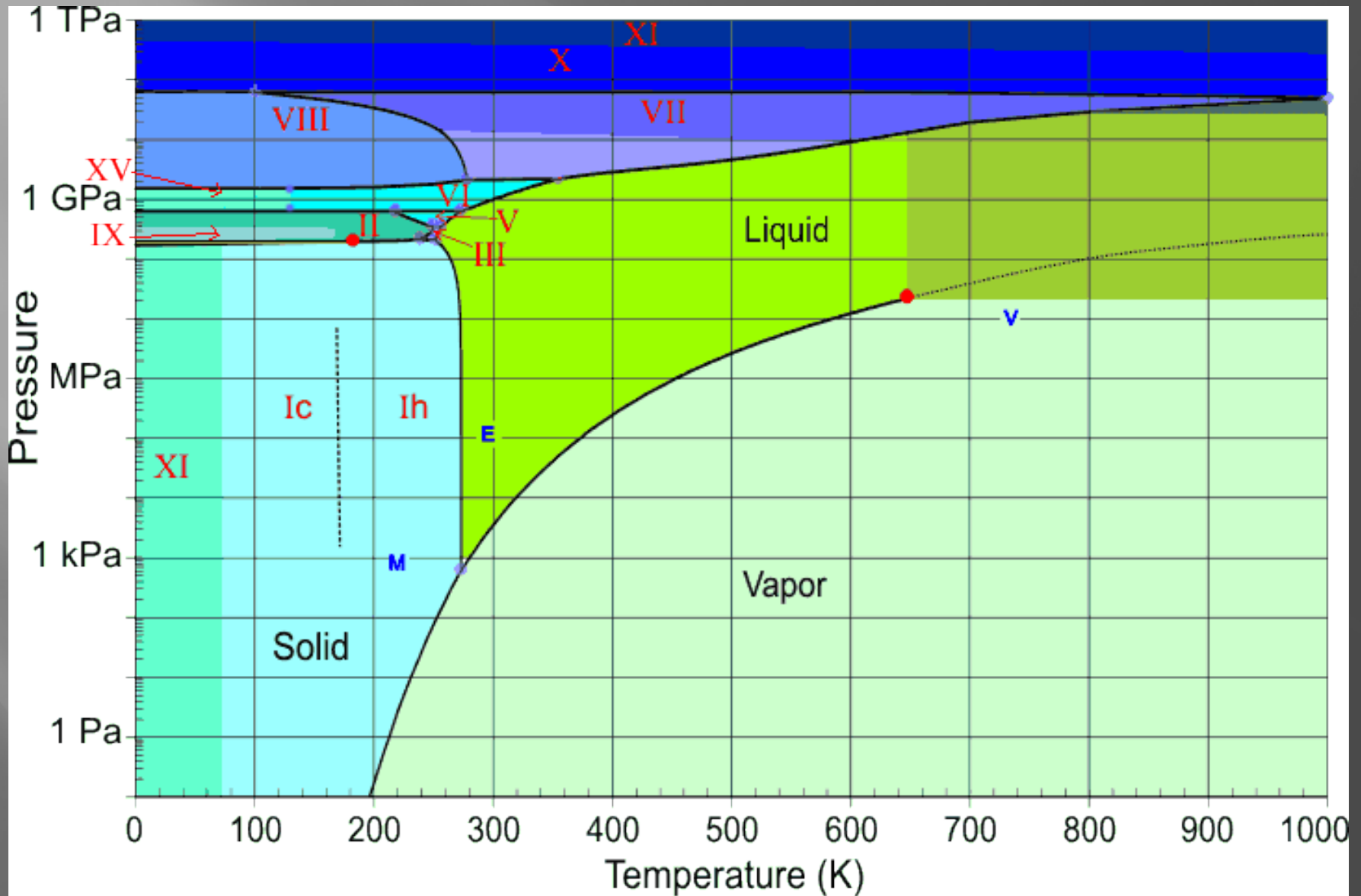
Therefore, volatiles in regolith and subsurface geological samples that would be collected and returned to Earth might need to be maintained at extremely low temperatures to fully preserve their scientific integrity.



**Fig. 2.** (A) Normalized histograms of measured daytime and nighttime bolometric brightness temperatures for the maps shown in Fig. 1, A and B, with comparisons to model-calculated surface temperatures at the same locations and times as those of the Diviner observations (fig. S3, A and B). (B) Histograms of model-calculated annual average temperatures at 2-cm depth for the maps shown in Fig. 1C and fig. S7, A to D, and at the LCROSS impact site for selected values of  $\theta_{\text{max}}$ , the mean maximum angle between the Moon's spin axis and the normal to the ecliptic plane.  $\theta_{\text{max}}=1.54^\circ$  for present-day conditions. (C) The recent evolution of  $\theta_{\text{max}}$  as a function of the Earth-Moon distance (29). (D) The volatility temperatures of a range of potential cold-trapped volatile compounds (21, 22).

Paige, D.A. et al. (2010) Diviner Lunar Radiometer Observations of Cold Traps in the Moon's South Polar Region, *Science* 330, p. 479 – 482.

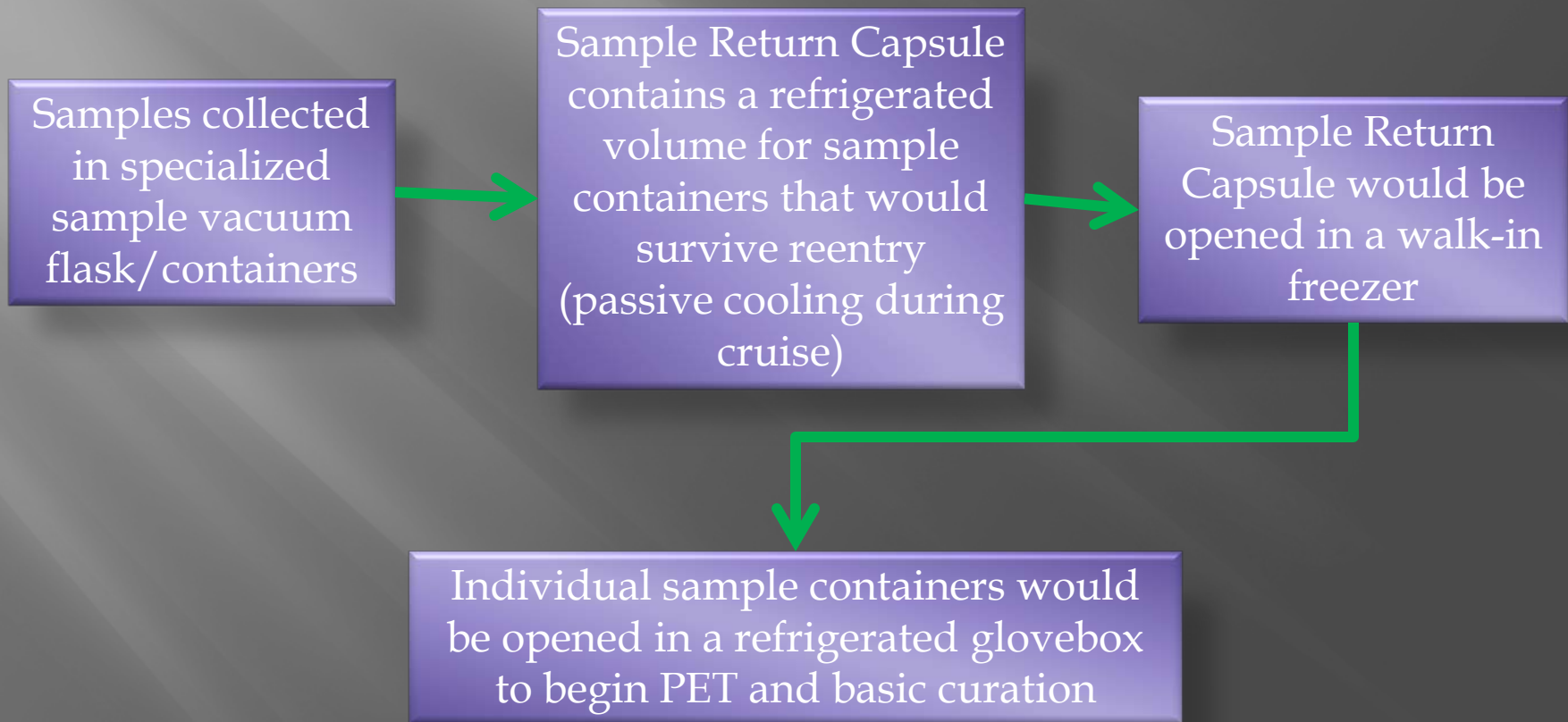
# H<sub>2</sub>O Phase Diagram



## Curation at 250 K (- 23.15 °C)

Preserved lunar regolith samples at 250 K, 1.0 atm could contain ice and solar wind derived volatiles.

Sample Return Steps:



## Commercial-Off-The-Shelf Cold Gloveboxes



Terra Universal  
Protein Analysis Glovebox  
- 20 °C, Class 100 environment



IP Systems  
Cold Testing Glovebox/Chamber  
- 40 °C, Class 100 environment

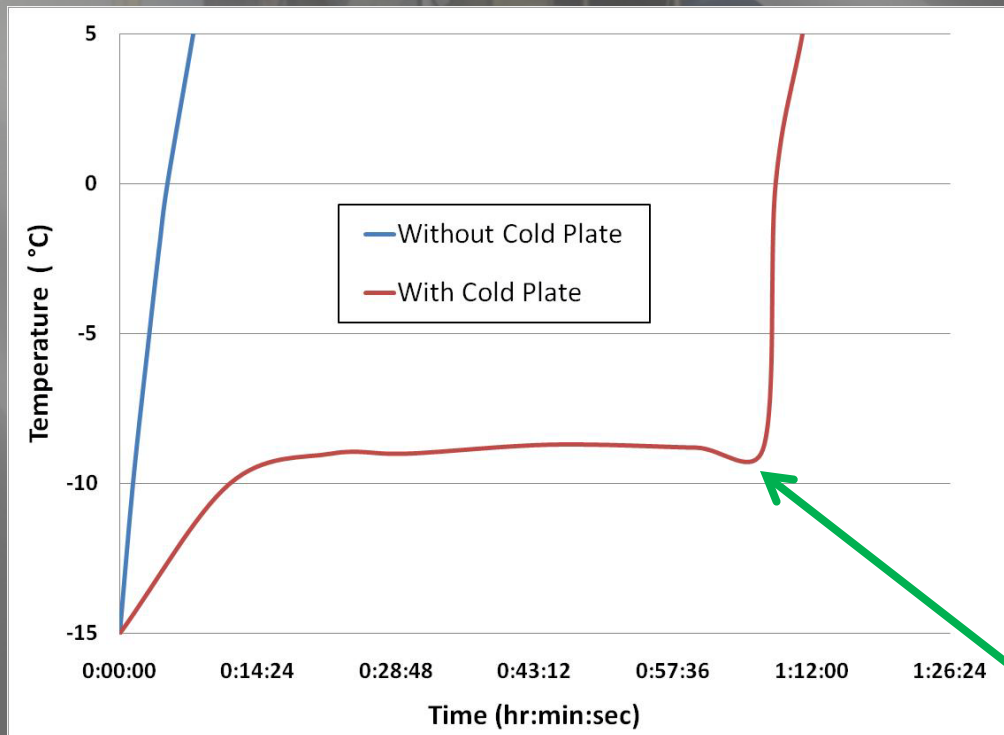


MBraun Glovebox at JSC



- 35 °C Sample Storage Freezer

- 35 °C Sample Cold Plate



Rock Warm-up Experiment  
Pampa J Meteorite  
19.631g ordinary chondrite

Average Time to Reach 0 °C  
in GN2 enriched environment  
from - 35 °C storage freezer

Without Cold Plate = 4 min. 59 sec.

With Cold Plate = Reached equilibrium at  
about 22 min. 2 sec. at - 9 °C  
when removed = 1 min. 16 sec. to 0 °C

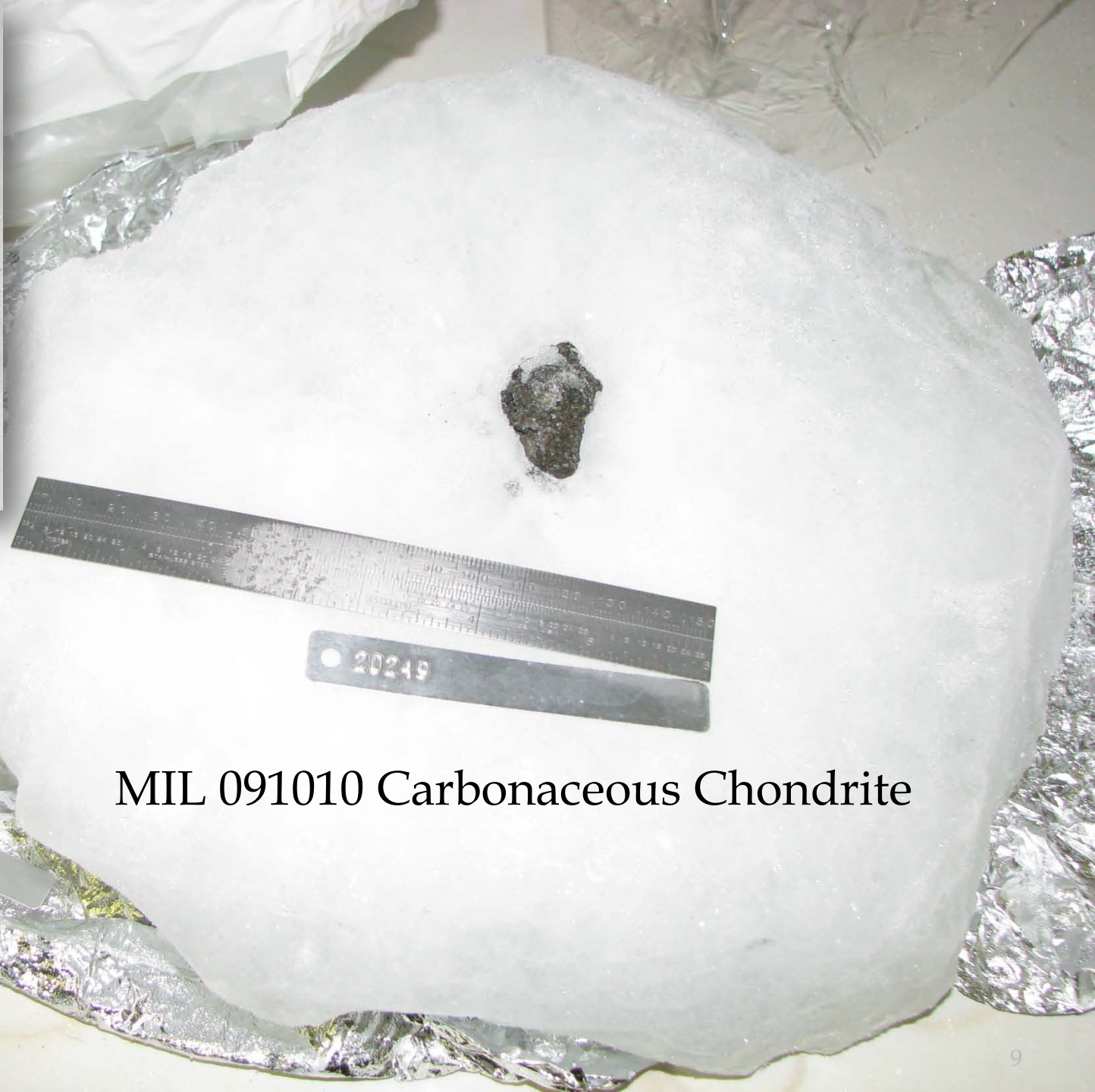


# U.S. Army Cold Regions Research and Engineering Laboratory (CRREL)

24 Cold Rooms Ranging from 24°F to - 50°F (- 4°C to - 45°C)



6' x 10' cold room, 10°F (-12°C) with laminar flow bench for 2010 Antarctic Meteorite Extraction from Ice



MIL 091010 Carbonaceous Chondrite



Ice was band sawed, then split with chisel



Sample Extracted with tweezers

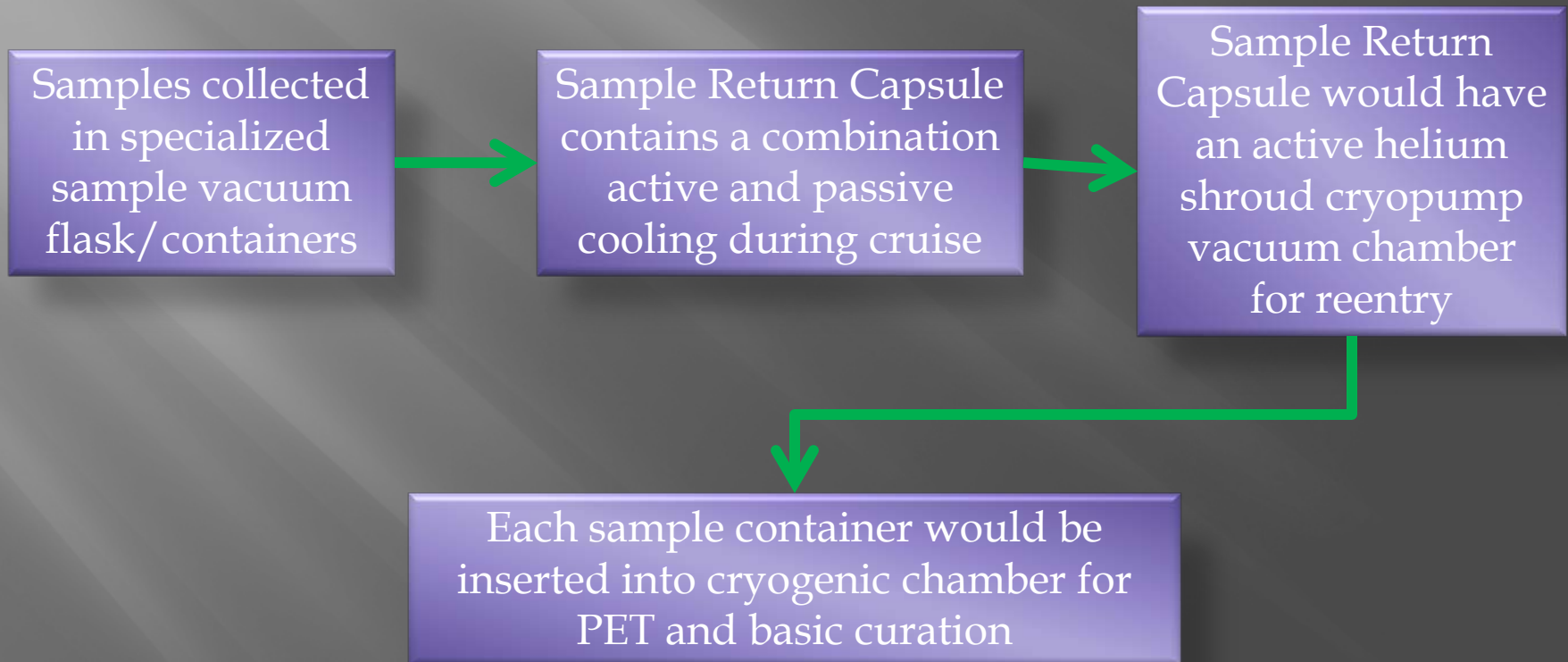


MIL 091010  
Carbonaceous Chondrite

## Curation at 40 K (- 233.15 °C)

Preservation of Lunar samples from extremely cold environments, including the lunar polar cold traps, could require curation at temperatures as low as 40 K and in vacuum

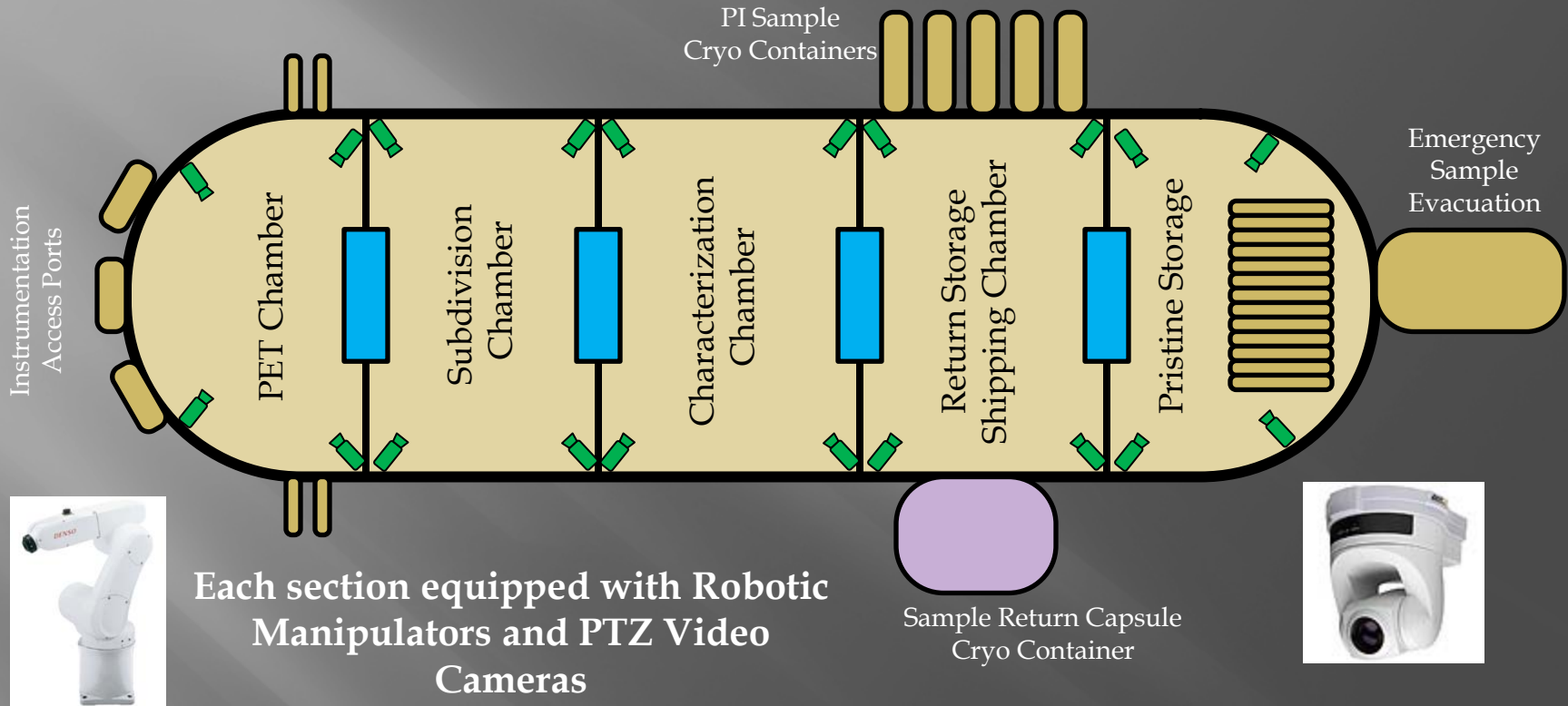
Sample Return Steps:



# 40 K Thermal Vacuum Curation Chamber

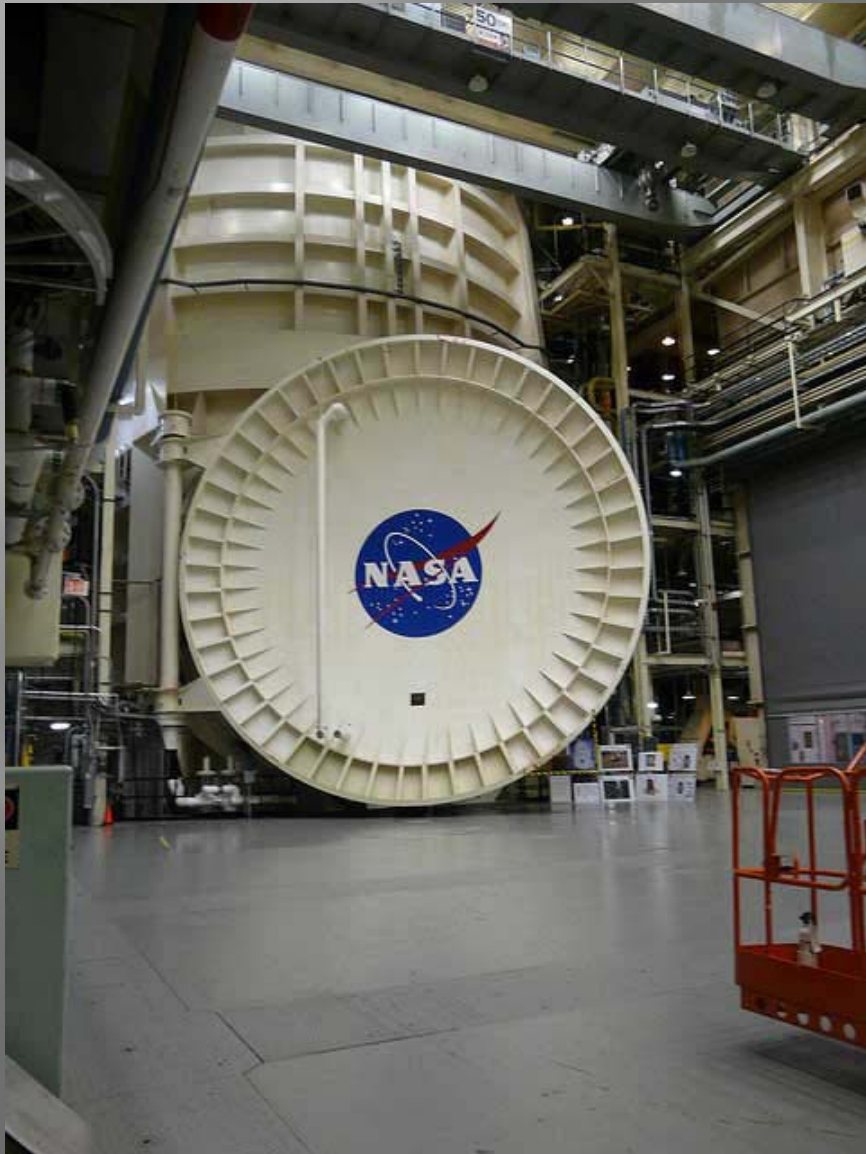
helium heat sink shroud at  $1 \times 10^{-6}$  Torr

12 m long x 3 m diameter



Cryogenic curation is feasible with current technologies developed for the superconductor industry. However, significant research and development costs would be required to tailor these technologies to the task of sample return and long term curation of lunar volatile samples at 40 K.

# Johnson Space Center Thermal Vacuum Chamber A Existing Technology and Expertise



# Johnson Space Center Thermal Vacuum Chamber A

## Current Configuration:

Working Dimensions: 55 feet (16.8 m) in diameter x 90 feet (27.4 m) high

Environment: 90 K liquid nitrogen heat sink shrouds at  $1 \times 10^{-6}$  Torr

## Post James Webb Space Telescope (JWST) Modifications:

Working Dimensions: 45 feet (13.7 m) in diameter x 80 feet (24.4 m) high

Environment: **20 K** helium heat sink shrouds at  $1 \times 10^{-6}$  Torr

Significant R&D investment for JWST is currently being developed with cold electronics, robotics manipulators, and testing chambers, all working at 20 K.



# The Importance of Sample Return and Long-term Curation

- ② High resolution and precision analytical results
- ② We have all the world's laboratories and instruments available for analysis
- ② Ability to use state-of-the-art instrumentation too big for remote sensing probes (Synchrotrons, SIMS, TEM, etc.)
- ② Ability to analyze samples at multiple laboratories with multiple methods for comparative results
- ② Provides ground-truth to remote sensing data
- ② Ability to analyze samples years/decades after samples have returned; to test new hypotheses and with new instrumentation as it is invented/developed to answer specific scientific questions



## Future Cold / Cryo Curation

With over four decades of scientific investigation, the Apollo sample collection has given the science community the ability to study lunar materials with highly precise measurements made in multiple laboratories.

High-resolution studies of lunar volatiles will require a sample return mission where cold or cryogenic curation preserves the scientific integrity of these fragile samples.

Cryogenic curation is feasible with current technologies developed for the superconductor industry

For JSC curation, cold curation at 250 K is the next step for developing advanced curation technologies for future missions with minimal investment, while monitoring the development of cryogenic technologies that could be used for future sample return missions that wish to preserve samples at very low temperatures