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The Importance of Correlative Microscopy for Planetary Sample Return Missions

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This is an incredibly exciting time to be a planetary sample scientist. Fifty years ago, NASA's Apollo missions to the moon brought back approximately 382 kilograms of samples, including rocks, rock cores, pebbles, sand, and dust. Scientists have studied many of those samples over decades and have made some incredible discoveries. Recently new samples have been released as part of the Apollo Next Generation Sample Analysis (ANGSA) program [1]. There are a series of other sample return missions, too many to list, but some examples are the Japanese Space Agency (JAXA)-led Hayabusa mission returned a small amount of material from a carbonaceous asteroid Itokawa, June 2010 [2]; the JAXA-led Hayabusa2 returned carbonaceous material from Ryugu December 2020 [3] and a NASA-led mission OSIRIS-REx will be returning carbonaceous material from Bennu September 2023 [4].

One incredibly ambitious project that is ongoing is the Mars Sample Return Campaign, an international partnership that will result in delivery of the first martian samples to Earth (that are not serendipitously brought to Earth as meteorites). Currently as part of the Mars 2020 Mission, the Perseverance Rover is collecting and caching samples in and around Jezero Crater, an ancient river delta that a combination of mission team members and scientists around the world selected as a key landing site [5]. The Mars 2020 mission represents the first leg of a series of missions to return samples from the surface of Mars to Earth in the early to mid-2030s. Two Mars Sample Return (MSR) missions are planned to follow in Perseverance's footsteps; one to land near or in Jezero Crater, collect and then launch the sample cache off the surface of Mars. The second would capture the sample cache in Mars' orbit, and then bring the samples back to Earth.

While we do have martian meteorites for study currently, these samples have experienced incredible pressures and temperatures from the extraction of the surface of the planet, uncontrolled entry and exposure to terrestrial environments which could alter some of the primary signatures of the samples. With the ability to sample the Martian surface in a controlled way, collect the material and all the "field data" along with the sample, and return the sample under controlled conditions and with sufficient time, we can design a facility in advance of sample delivery to keep those samples in a pristine (i.e., as returned) state for an indefinite period. Also, samples returned from Mars will be "restricted" and need Biosafety Level 4 (BSL-4) containment until deemed safe for release. A Sample Receiving Facility (SRF) is where the Earth Entry System is opened, and the sample tubes are opened and processed after they land on Earth. Samples should be accessible for research in biocontainment for time-sensitive studies and eventually, when deemed safe for release after sterilization or biohazard assessment, should be transferred out of containment for allocation to scientific investigators in outside laboratories. There are two main mechanisms for allocation of samples outside the SRF: 1) Wait until the implementation of the Sample Safety Assessment Protocol (Planetary Protection) results conclude that the samples are non-hazardous, 2) Render splits of the samples non-hazardous by means of sterilization. To make these samples accessible, a series of observations and analytical measurements will need to be completed to produce a sample catalog for the scientific community.

Once these samples are accessible, objective-driven investigations are sample analyses and studies performed to address MSR Campaign L1 (Level 1) science objectives in support of campaign success criteria [7]. There will also be opportunity-driven investigations that are studies that are not explicitly conducted to address MSR Campaign L1 objectives and success criteria. Investigators will apply for sample access and–if deemed meritorious through a selection process–would be provided a sample allocation to conduct their research. These investigations will commence after samples have been returned to Earth and will continue indefinitely into the future. These selection review panels will likely take in consideration the amount of material that is required for the study, the impact of the study and how different types of measurements can be done on minimal sample.

Martian meteorites are the best material for us to be studying now, to prepare for these MSR samples**.** Over 100 Martian meteorites have been identified on Earth at the time of this report, ranging in age from 4,400 million years (Ma) to 165 Ma [8]. Analyzing these meteorites provides an opportunity to study the Martian atmosphere [9], Martian surface processes [10], and even Martian water that may have interacted with these rocks when they were once components of the planet's crust [11]. The extensive range in ages means that we can examine how volcanism, the Martian climate, and the availability of the ingredients of life on Mars have evolved over almost the entirety of Martian history. Shergottite meteorites are the most common type of Martian meteorite, the only samples we have from Mars, and have mafic and ultramafic compositions, like volcanic samples on Earth.

The analysis of Martian meteorites is complicated since all Martian meteorites are inherently shocked, undergoing intense deformation during ejection from the Martian surface before landing on Earth. These shock events can cause deformation, mineral transformations, and chemical reactions including the incorporation of Martian atmosphere into certain minerals and glasses through shock compression. The effects of shock metamorphism are so extensive that isotopic heterogeneities are often induced at the nanoscale, complicating efforts to accurately characterize Martian materials with conventional techniques. Using a variety of analytical techniques, such as Atom Probe Tomography (APT), Scanning Transmission Electron Microscopy (STEM), NanoSIMS, Scanning Electron Microscopy (SEM) Electron Backscatter Diffraction (EBSD) we are studying these affects and working out correlative methodology at the Pacific Northwest National Laboratory (PNNL) vial an Environmental Molecular Science Laboratory (EMSL) Large-Scale research grant.

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