PRECISION SUBSAMPLING SYSTEM FOR IN SITU ANALYSIS AT MARS (AND BEYOND).

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Introduction: The ability to analyze heterogeneous rock samples at fine spatial scales would represent a powerful addition to our planetary *in situ* analytical toolbox. This is particularly true for Mars, where the signatures of past environments and, potentially, habitability are preserved in chemical and morphological variations across sedimentary layers and among mineral phases in a given rock specimen. On Earth, microbial life often associates with surfaces at the interface of chemical nutrients, and ultimately retains sub-mm to mm-scale layer confinement in fossilization. On Mars, and possibly other bodies, trace chemical markers (elemental, organic/molecular, isotopic, chiral, etc.) and fine-scale morphological markers (e.g., microfossils) may be too subtle, degraded, or ambiguous to be detected, using miniaturized instrumentation, without some concentration or isolation. This is because (i) instrument *sensitivity* may not be high enough to detect trace markers in bulk averages; and (ii) instrument *selectivity* may not be sufficient to distinguish such markers from interfering/counteracting signals from the bulk. Moreover from a fundamental chemostratigraphic perspective there would be a great benefit to assessing specific chemical and stable isotopic gradients, over mm-to-cm scales and beyond, with higher precision than currently possible *in situ*.

We are developing a precision subsampling system (PSS) that addresses this need while remaining relatively flexible to a variety of instruments that may take advantage of the capability on future missions. While originally conceived primarily for the Astrobiology Field Lab mission [1], the PSS as further defined is highly relevant to a number of future lander/rover missions as well as Mars Sample Return. Our plan is to develop a specific PSS prototype and fully test it under Mars ambient conditions, on a variety of natural analog rocks and rock drill cores, using a set of complementary flight-compatible measurement techniques.

PSS Description and Characterization: We are taking a "secondary sampling" approach to the baseline design of the PSS. That is, it is assumed that a primary sample has been acquired by means of a coring drill, grasping tool, or scoop, that can be delivered to a holding station on a lander/rover, where precisely selected secondary samples (masses in the range of tens of μg to tens of mg) are obtained. This approach permits us to focus on subsystem development and testing with instruments, without the additional complexity of primary sampling (working directly with Mars surface) for which there are several existing technologies. For example, one PSS configuration assumes a drill core, such as produced by the Honeybee MiniCorer [2], positioned as shown in Fig. 1 enabling us to subsample any depth by lateral motion of a fine-scale drilling device. The PSS can also be used on hand-samples, with abraded or intact weathered surfaces. The subsample is passively collected on a tape-type collection and transport device (CTD) allowing it to be further analyzed under ambient and vacuum conditions, and optionally archived (with possible use for sample return).

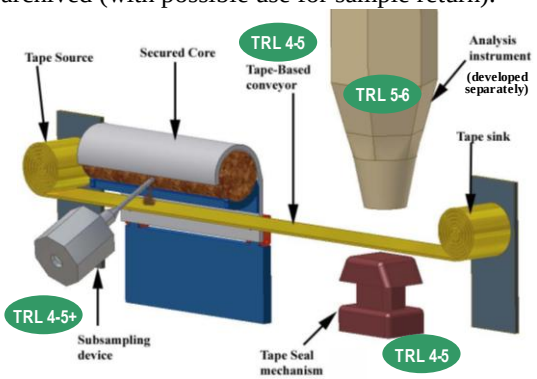

Fig. 1 Nominal spatial arrangement of the PSS applied to an 8 mm diameter core. Current technology readiness level (TRL) of key subsystems is indicated.

The core may be inspected with micro-imagers, spectrophotometers, laser excitation, etc. (not shown in Fig. 1), which can be used to select points for subsampling. Subsampling would be appropriate for measurements requiring separation of the selected material from the bulk, further chemical extraction, and/or analysis under vacuum conditions. Therefore three activities are involved in the PSS:

1. Precision subsampling. The most direct approach here uses a small rock milling device (dental-

type or other powdering drill tool) with a robust universal bur or set of exchangeable burs. Prior to finalizing the baseline drill for Mars, we are conducting preliminary tests of subsampling performance over a range of parameters, using a commercial dental hand tool (Brasseler/NSK Z-500). Example parameters include (1) rock characteristics such as hardness and composition, (2) drilling specifications (bur type, speed, contact force, angle of attack, etc.), and of course (3) the data obtained by measurements of subsamples, comparing point-by-point and fine-scale versus bulk composition.

2. Collection and transport. The CTD was inspired by the use of a similar approach in a terrestrial aerosol collection time-of-flight mass spectrometer (TOF-MS) [3]. The tape acts as an entrapment surface for fine particulates as well as a substrate for laser desorption, which can be done *in vacuo* with the tape serving as a demountable vacuum sealing gasket. With appropriate material selection, surface treatment, and electrostatic design, the tape-type system can efficiently and passively collect a thin, localized layer of subsample. In this approach each of potentially hundreds of subsamples occupies a pristine section of tape (a few mm in diameter). A separate CTD task is focusing on capture and analysis of subsampling-induced volatiles.

3. Ambient and lab analyses. Individual subsamples are analyzed under ambient or vacuum conditions as desired, nominally in a given sequence. As an example, a microscope combined with point spectrometers using ultraviolet fluorescence and infrared reflectance could be used to determine the presence of organic compounds which could then be thoroughly studied with various forms of mass spectrometry. In Fig. 1 the concept of a laser mass spectrometer is depicted as it only requires the thin layer of sample positioned on the tape surface. Subsamples of this scale would also be amenable to wet chemical extraction and analysis in microfluidic-based chemical laboratories.

Preliminary Tests: To refine PSS requirements we are examining a small set of initial analog samples with chemical heterogeneity consistent with our mm-class subsampling scale. Subsampling efficiency tests (extracted mass versus time, required load) over a drill speed range of 1000 to 10,000 rpm suggested that 5000 rpm is optimal for a variety of rock and mineral types. Selected subsamples obtained under reproducible conditions were further subject to evolved gas analysis to detect chemical differences. The Pilbara sample in Fig. 2 was cut from a dolomitic drill core from Hamersley Province in Western Australia. It displays finely laminated dolomite (lighter material) and kerogenous shale (darker material) with some mild faulting evident. It is known to contain extractable hydrocarbons including indigenous bacterial biomarkers [4]. We have extracted multiple in-layer subsamples from with a sub-mm radius diamond bur at 5000 rpm. As a baseline comparison, several-mg aliquots of light and dark layer subsamples were analyzed with a lab prototype of the pyrolysis-based quadrupole mass spectrometer system in the Sample Analysis at Mars (SAM) investigation on the Mars Science Laboratory. We are in the process of evaluating these results, but already it is apparent that there are characteristic evolved gas signatures associated with layer mineralogies and organics. In a separate test we subsampled few mm-scale masses of stilpnomelane in a quartz matrix. Trace volatile organic abundances in the dark mineral phase, as measured via aliphatic hydrocarbon fragments evolved over the range 300-500 C, could have been quite challenging to detect if diluted in the bulk quartz mineralogy.

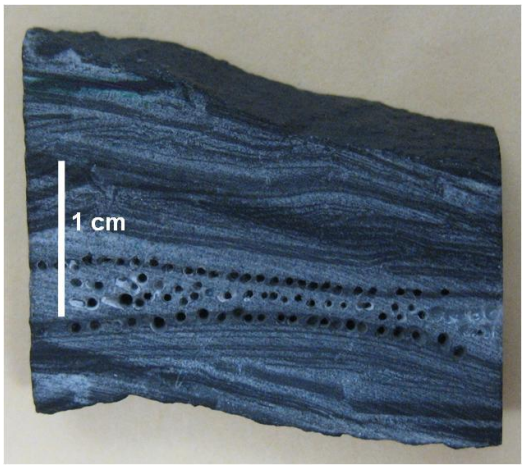

Fig. 2 Laminated Pilbara core sample with individual subsampling pits at scale and locating precision as expected for a Mars PSS.

As the complete PSS system is designed, we will be continuously analyzing subsamples using mass spectrometers and other instruments. Our long-term objectives include collaboration with interested parties, who may benefit from analyzing subsampled materials as part of their instrument development activities.

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Acknowledgments: This work is supported by NASA's Mars Instrument Development Project (MIDP).