## In Situ Analytical Strategy For Mars Combining X-Ray And Optical Techniques

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Geological samples on earth rarely arrive “sight-unseen” in a laboratory before being analyzed by sophisticated instrumentation. Rather, the samples are carefully chosen by a geologist in the field using primarily optical methods. In the process of sample selection in the field, most samples are rejected in preference for a few which appear to warrant further scrutiny in the lab. But before their journey to the lab, the rocks have already been identified in some detail as to which basic geological category they belong to, and many mineral species within the rocks have been identified. Such information is gathered by the eyes and handlens of the geologist which establish geological context, structures, and sample color, texture, crystallinity, granularity, etc.

Despite the time-proven efficacy of this method of selecting and identifying rock samples for analysis, *in situ* missions to Mars have not placed heavy emphasis on instrumentation that can act as a surrogate field geologist. MER is an exceptional example in employing a microscopic imager as the eyes of the geologist. We propose to extend this approach with the MICA (Mineral Identification and Compositional Analyzer) instrument; a flight prototype is currently being developed through the NASA MIDP program. MICA would serve well on the MSL ‘09 mission, or on lunar or cometary venues. The instrument combines a microscopic imager (MI) with X-ray fluorescence spectroscopy (XRF) and X-ray diffractometry (XRD).

When the MICA instrument is placed in contact with a rock or soil sample, it bombards the surface with X-rays at a low glancing angle. The X-rays are generated by a miniature X-ray tube with a copper target and a nanotube array as the emission cathode. The glancing X-rays produce forward scattered diffraction cones from the sample surface that are intercepted by a CCD detector. Each cone is generated by constructive interference of the X-rays with the sample, and the full suite of cones provides a unique mineralogical signature (representing characteristic lattice spacing parameters). Diffraction X-rays are selected by a CCD pulse amplitude discriminator which selects a narrow energy band at the Cu target K-alpha energy of 8.05 keV.

Isotropically-scattered fluorescent X-rays from the sample surface are simultaneously generated by the X-ray beam, and these are also captured by the CCD. Each fluorescent X-ray photon has an energy corresponding to the atomic number of the element from which it was emitted. By binning these X-rays pulses from the CCD according to their energy (always selecting those with energy less than that of the diffraction photons), the instrument constructs a spectrum of energies that can be converted to a spectrum of elemental abundances. A single CCD detector serves for both XRD and XRF.

Situated between the X-ray tube and the CCD is a microscope consisting of a camera, lens barrel, and a special optical/electronic filter that enhances the depth of field above that of conventional microscopes (for comparable magnifications). This enables rough rock surfaces to be viewed while observing at a resolution of 4 microns. The microscope observes the same spot being bombarded by the X-rays and serves as a check on the beam position as well as being an analytical tool in its own right.

MICA can operate in two modes. In the “deployed” mode, the instrument is placed on the end of a robotic arm of a rover. This deployment is facilitated by the low mass and volume of the unit (approx. 1 kg, 1 liter). It is also facilitated by the fact that no sample acquisition or preparation is required: analysis is conducted by simply bringing the instrument into contact with a sample which can be either granular (powder or sand) or solid (rock flat or boulder). We are demonstrating the ability of this technique to provide quantitative XRF analysis (elemental abundances to within a few percent) and semi-quantitative XRD (positive mineral identification with first order evaluation of mineral abundance) on solid rocks. For powder surfaces such as the Martian dust, we expect to be able to conduct both quantitative XRF and XRD owing to the diffraction statistics associated with fine powders. In a “passive” operational mode, MICA can also be deployed on board a lander and be supplied by samples --for example, as planned by JPL for MSL. In this

case, quantitative XRD and XRF are theoretically achievable for all types of samples.

Our rationale for combining these analytical capabilities into one instrument derives from our belief that while it is critical to have unequivocal mineral identification, this alone cannot provide unequivocal rock identification. Mineralogy is not necessarily the key to lithology. Rather than seeing MICA as an XRD instrument with XRF and optical augmentation, we see it as a geologist with excellent optical skills and augmented lab capabilities of X-ray analysis.

We have conducted laboratory analyses of the basic rock types --sedimentary, igneous, and metamorphic. Their discrimination by instrument analysis derives primarily from optical inspection. A classic example is cited. A pink aeolian sandstone was analyzed. From our MI, it was immediately obvious that this sample was indeed a sandstone from the recognition of discrete sand grains composing the material. Furthermore, the individual grains were recognized as well-rounded, well sorted by size, having a brown ferruginous coating, and set within a white cementing matrix. From this simple but very revealing imagery, we were able to say that the sample was (a) of clastic sedimentary origin laid down in a relatively quiescent environment, (2) that the grains had undergone significant fluid (wind or water) transport, (3) that the grains had acquired a pre-diagenetic coating of ferruginous (and perhaps clay) material reminiscent of desert dune sands --indicating motional stasis in a dune system, and (4) diagenesis had occurred in a post-depositional environment to produce a pure siliceous or calcareous interstitial cement derived from the intergranular percolation of chemically-rich, supersaturated ground water. XRF confirmed the mineralogical implications of the optical analysis --revealing primarily silicon composing both grains and cement. XRD corroborated this by revealing a dominance of quartz mineralogy.

The understanding of the sample became quite comprehensive, but it is noted that the data flow sequence was from optical to chemical to mineralogical. If we had conducted only XRD, we would have learned that the material was dominantly quartz. XRF would have supported this. But the basic XRF fingerprint of the material was virtually indistinguishable from quartz-rich granite, quartzose gneiss, and a quartz-rich turbidite. There was absolutely nothing in the XRD or XRF data to tell us that the sample was granular (clastic), rather than polycrystalline, and that it was sedimentary, not igneous. In this example, our ability to recognize a sedimentary rock as distinct from an igneous one enabled the inference of fluid processes --a critical factor for Mars where the recognition of water-related processes is key to exobiology, geology, and paleoclimate. Even though clastic sedimentation of rounded grains can indicate either water or air transport, the grain coatings and interstitial cement are definite indicators of aqueous processes.

MICA’s non-destructive rock analysis also enables grain size distribution in the sample to be determined. Grain-size information (either clastic grain size or crystal size) can be important in understanding the origin of a sample. For sedimentary materials, grain size distribution defines the energy and stability of fluid transport (potentially enabling distinction between air, water, ice, colluvial, volcanic, and other transport mechanics), and for igneous/metamorphic materials, grain size defines cooling rates, thermal homogeneity of the system, flow conditions, and eruption-fragmentation processes. MICA determines grain size from both the MI and XRD. For the latter, software algorithms interpret the size of diffraction spots composing the diffraction arcs on the CCD detector.

Because MICA does not require sample acquisition or preparation, it is a non-invasive technique. Conventional XRD techniques require sample pulverizing and grain-size fractionation. If samples do not require acquisition, there are enormous savings of power, mechanical complexity, mass, and risk in the analysis of a sample. The acquisition, processing, and delivery of a sample is at least as complex, and certainly more risk than sample analysis itself. MICA circumvents this problem. Other benefits of proximity analysis include non-destructive interrogation of materials --perhaps of delicate mineralogical or biological growths on rock surfaces, and the ability to analyze thin surfaces such as weathering rinds or varnishes (indicators of weathering and climate). We have conducted analyses of desert varnish that would not be possible by boring or grinding the sample prior to analysis.