EVALUATION OF ROCK POWDERING METHODS TO OBTAIN FINE-GRAINED SAMPLES FOR CHEMIN, A COMBINED XRD/XRF INSTRUMENT. S. J. Chipera[1], D. T. Vaniman[1], D. L. Bish[2], P. Sarrazin[3], S. Feldman[4], D. F. Blake[4], G. Bearman[5], and Y. Bar-Cohen[6], [1]Los Alamos National Laboratory, MS D469, Los Alamos, NM 87545, [2]Indiana University, 1001 E 10th St., Bloomington, IN 47405, [3]Apparati Inc., 110 Pioneer Way, Suite I, Mountain View, CA 94041, [4]NASA Ames Research Center, MS 239-4, Moffett Field, CA 94035, [5]JPL, MS 306-336, Pasadena, CA 91109, [6]JPL, MS 82-105, Pasadena, CA 91109.

Introduction: A miniature XRD/XRF (X-ray diffraction / X-ray fluorescence) instrument, CHEMIN, is currently being developed for definitive mineralogic analysis of soils and rocks on Mars [1]. One of the technical issues that must be addressed to enable remote XRD analysis is how best to obtain a representative sample powder for analysis. For powder XRD analyses, it is beneficial to have a fine-grained sample to reduce preferred orientation effects and to provide a statistically significant number of crystallites to the X-ray beam [2]. Although a two-dimensional detector as used in the CHEMIN instrument will produce good results even with poorly prepared powder [3], the quality of the data will improve and the time required for data collection will be reduced if the sample is fine-grained and randomly oriented.

A variety of methods have been proposed for XRD sample preparation. Chipera et al. [4] presented grain size distributions and XRD results from powders generated with an Ultrasonic/Sonic Driller/Corer (USDC) currently being developed at JPL. The USDC was shown to be an effective instrument for sampling rock to produce powder suitable for XRD. In this paper, we compare powder prepared using the USDC with powder obtained with a miniaturized rock crusher developed at JPL and with powder obtained with a rotary tungsten carbide bit to powders obtained from a laboratory bench-scale Retsch mill (provides benchmark mineralogical data). These comparisons will allow assessment of the suitability of these methods for analysis by an XRD/XRF instrument such as CHEMIN.

Methods: Three samples representing potential target rocks for a Mars lander were prepared for this study. The samples included an igneous volcanic rock (andesite), a sandstone, and an evaporite (gypsum). To characterize the particle size distributions, each sample was wet-sieved through progressively finer mesh screens. XRD results for all samples were obtained by analyzing the <45μm size fraction on a commercial Siemens D500 diffractometer using CuKα radiation and fitted with incident- and diffracted-beam Soller slits and a Kevex solid-state PSi detector.

The USDC was driven with a 20 kHz AC voltage at resonance. In operation, the tip impacts a free-mass which in turn drives a drill bit in a hammering action. The free-mass rebounds to interact with the horn tip leading to a cyclic rebound at frequencies in the range of 60-1000 Hz [5].

The tungsten carbide rotary bit was rotated using a standard Dremel tool connected to a variac to control the rotational speed of the bit. Sufficient voltage was maintained to allow the bit to turn freely while under pressure. Slow speeds were maintained because excessive rotational speed resulted in poor sample recovery.

The crusher works by successive fracturing of a sample between two plates [6]. As fragments are created by fracturing and spallation, they fall through the exit throat at the bottom when their characteristic dimension is less than the width of the exit slit.

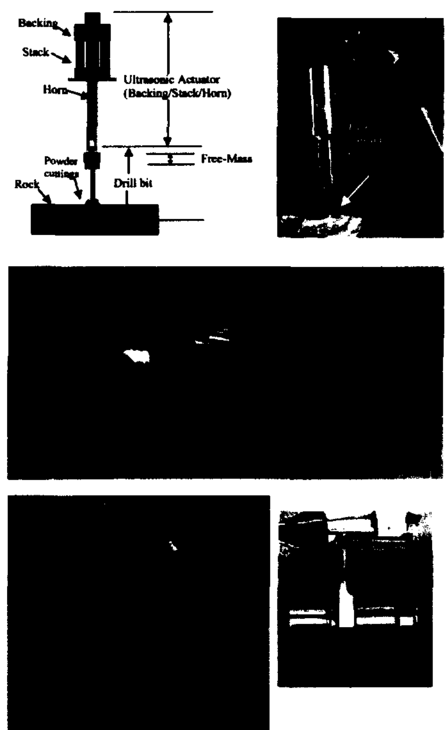

Figure 1: Powder producing methods used in this study. Top - Ultrasonic/Sonic Driller/Corer (USDC). Middle - Rotary tungsten carbide bit.

The rock crusher is currently being developed at JPL and is intended as a facility rock preparation system for the Mars '09 MSL.

Results: XRD results are optimized with very fine particle sizes, and laboratory preparation usually strives for average particle sizes of 5 μm or less to reduce preferred orientation and other sample related effects [2]. Figure 2 compares the particle size distributions for the three methods on the sandstone sample. The bulk of the powders obtained using either the USDC or the rotary bit were in the finest (<45μm) fraction whereas the JPL crusher powders were coarser, on average. Recent advances in sample handling and sample manipulation methods that keep the grains in constant motion [7] greatly ease the grain size restriction. With particle movement, samples with average grain sizes of 150μm produce quality X-ray diffraction data. Accommodating larger particle sizes will greatly reduce the amount of sample preparation required to provide suitable powder to a CHEMIN instrument. With the USDC and rotary drills, much of the powder produced falls into the <150μm size range (20-95% depending on rock type). The JPL crusher will require more sample processing/sieving as only 5-20% of the powder falls into this range, but a suitable sample can be obtained from any of the three methods.

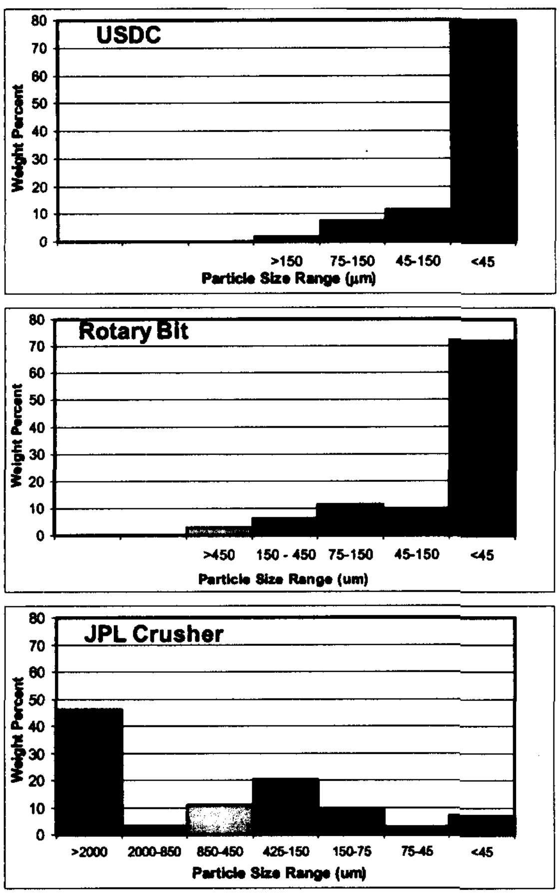

Figure 2: Particle size distributions obtained from the various sample-powdering methods for sandstone.

With regard to sample-to-sample contamination, both the JPL crusher and the rotary bit showed visible contamination between runs. However, much of the contamination issue can be addressed by simply conducting a dry run between samples. Sample accessibility is of concern because the USDC and rotary bits work at rock surface, requiring a sample-transfer capability. The crusher requires chunks of sample to be brought to it but powdered sample is readily obtained from the bottom exit port.

Lastly, mineral/phase segregation during the powdering process is of significant concern. It is important that the powder being delivered to the instrument be homogeneous and compositionally representative of the bulk rock from which it is derived. For all three rock types examined in this study, mineral compositions determined from USDC-prepared samples varied little from bulk-rock analyses. Powder prepared from the sandstone (comprised of hard quartz sand grains in a soft calcite and clay cement) using the rotary bit showed slight compositional deviation from the bulk-rock analysis. The JPL crusher did well with the andesite and the evaporite (gypsum) samples but showed considerable mineral segregation for the sandstone sample (Table 1).

Table 1: Quantitative XRD results for the various sample-powdering methods for the sandstone.

Mineral	Retsch	USDC	Rotary	Crusher
Quartz	73.6	73.1	68.0	39.8
Feldspar	0.1	0.2	1.3	2.4
Calcite	7.9	7.6	11.5	9.1
Mica	3.0	3.2	3.1	5.0
Kaolinite	12.1	11.3	11.8	34.3
Smectite	3.4	4.5	4.3	9.4

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