

LA-UR: 03-0329

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OBTAIN SAMPLE POWDER FOR CHEMIN, A COMBINED
XRD/XRF INSTRUMENT

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Submitted to: 34th Lunar and Planetary Science Conference, Houston TX,
March 17-21, 2003



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Form 836 (8/00)



USE OF AN ULTRASONIC/SONIC DRILLER/CORER TO OBTAIN SAMPLE POWDER FOR CHEMIN, A COMBINED XRD/XRF INSTRUMENT. S. J. Chipera¹, D. L. Bish¹, D. T. Vaniman¹, S. Sherrit², Y. Bar-Cohen², and D. F. Blake³, ¹Hydrology, Geochemistry, and Geology, Los Alamos National Laboratory, MS D469, Los Alamos, NM 87545, USA (chipera@lanl.gov), ²Science and Technology Development Section, Jet Propulsion Laboratory, MS 82-105, Pasadena, CA 91109 and ³NASA Ames Research Center, MS 239-4, Moffett Field, CA 94035.

Introduction: One of the technical issues that must be addressed before landing an XRD/XRF spectrometer on an extraterrestrial body is how best to obtain a representative sample powder for analysis. For XRD powder diffraction analyses, it is beneficial to have a powder that is extremely fine grained to reduce preferred orientation effects and to provide a statistically significant number of crystallites to the X-ray beam [1]. Although a 2 dimensional detector as used in the CHEMIN instrument will produce good results with poorly prepared powders [2], the quality of the data will improve with the quality of the sample powder.

An Ultrasonic/Sonic Driller/Corer (USDC) currently being developed at JPL (Figure 1) is an effective mechanism of sampling rock to produce cores and powdered cuttings. It requires low axial load (< 5N) and thus offers significant advantages for operation from lightweight platforms and in low gravity environments. The USDC is lightweight (<0.5kg), and can be driven at low power (<5W) using duty cycling. It consists of a piezoelectric stack, ultrasonic horn, free-mass, and drill bit and backing. The stack is driven with a 20 kHz AC voltage at resonance. The strain generated by the piezoelectric is amplified by the horn leading a factor of up to 10 times the displacement amplitude. The tip impacts the free-mass and drives it into the 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. It does not require lubricants, drilling fluid nor bit sharpening and it has the potential to operate at high and low temperatures using a suitable choice of piezoelectric material. To assess whether the powder from an ultrasonic drill would be adequate for analyses by an XRD/XRF spectrometer such as CHEMIN, we analyzed powders obtained from the JPL ultrasonic drill and compared the results to carefully prepared powders obtained using a laboratory bench scale Retsch mill.

Methods: Eight samples representing potential target rocks for a Mars lander were drilled for this study. They consisted of igneous volcanic rocks (basalt and andesite), sandstone, and evaporite/spring deposit rocks (limestone, calcite veins, and gypsum). To characterize the particle size distribution for sam-

ples obtained from the USDC, each sample was wet sieved through 100, 200, and 325 mesh sieves (150, 75, 45 μm respectively) and sample weights were recorded. Further analyses were conducted on the <325 mesh fraction using a Horiba CAPA-500 particle size distribution analyzer set up to bin from 0-50 μm using 5 μm bins.

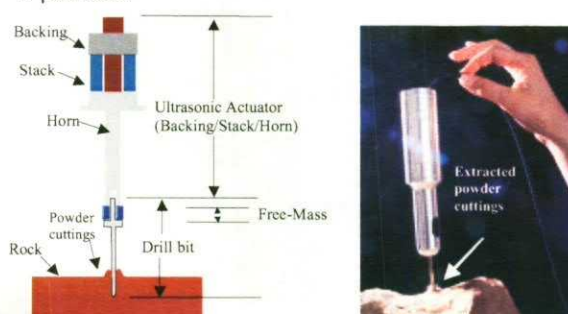


Figure 1: A schematic view of the USDC components. The USDC is shown to require relatively small preload to core a rock. The powder cuttings travel along the bit providing a removal mechanism for acquisition.

Results: Two types of rock powder were generated from the drill. Fine powder was generated from the cutting tip itself; the second product consisted of spallation detritus generated during the drilling operation. It was found that the softer materials tended to produce far more spallation detritus than the harder, more competent materials and that the orientation of the drill to the rock also affected spallation. Figure 2 shows results for a sample from the basal limestone of the Todilto Formation (Echo Amphitheater, New Mexico). This sample is composed mainly of calcite with minor quartz and gypsum. The top histogram shows that the bulk of the ultrasonic drill powder generated for this sample was composed of spallation detritus. However, the <325 mesh fraction (middle histogram), which is representative of the material generated at the cutting tip of the ultrasonic drill, shows that the drill does an excellent job of generating a fine powder for XRD analysis with much of the powder less than 10 μm in size. The bottom histogram shows the particle size distribution obtained on this sample from a laboratory Retsch mill for comparison.

Figure 3 shows results for an andesite (Tschicoma

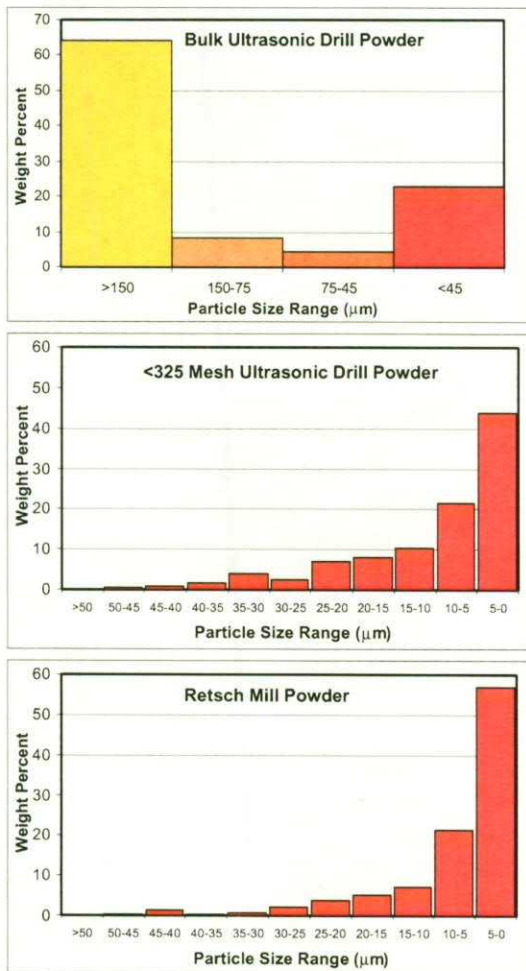


Figure 2: Basal Limestone, Todilto Formation

Peak, Jemez Mountains, New Mexico). The sample is a pyroxene-plagioclase porphyritic lava with fine-grained crystalite and a trachytic matrix. The top histogram plots the results of the size separations through the various sieves. However, compared to the Todilto basal limestone samples, this sample had very little spallation from ultrasonic drilling and most of the sample is in the fine fraction that passed through the 325 mesh sieve. The histogram of the <325 mesh fraction from ultrasonic drilling again shows that most of the sample is in the finest fractions, which is desirable for XRD analyses. The bottom figure compares XRD patterns obtained for the ultrasonic drill powder (blue) with the laboratory Retsch mill powder (red). Standard front packed mounts were utilized and the patterns compare extremely well, even though the andesite contains abundant feldspar and pyroxene that can show variable orientation effects.

The ultrasonic drill was found to do an outstanding job of generating quality XRD powders from all of the materials tested. XRD patterns obtained on

a laboratory Siemens D500 XRD unit for the mechanically screened ultrasonic drill powders (a simple process for excluding coarse chips) are essentially indistinguishable from powders obtained from a laboratory Retsch mill. The particle size distributions are also quite comparable between the two methods, demonstrating that the ultrasonic drill is more than adequate to generate powders for a landed XRD/XRF spectrometer. In practice, introduction of the powder into an XRD instrument may require passing the powder through a sieve to separate the drill bit powder from spallation detritus, but such sieving can be used to assist in the loading samples onto a specimen mount for analysis.

References: [1]Bish D. L. and Reynolds R. C. Jr. (1989) *Modern Powder Diffraction*, MSA Reviews in Mineralogy, **20**, 73-99. [2]Vaniman D. T. et al. (1998) *JGR*, **103**, 31477-31489.

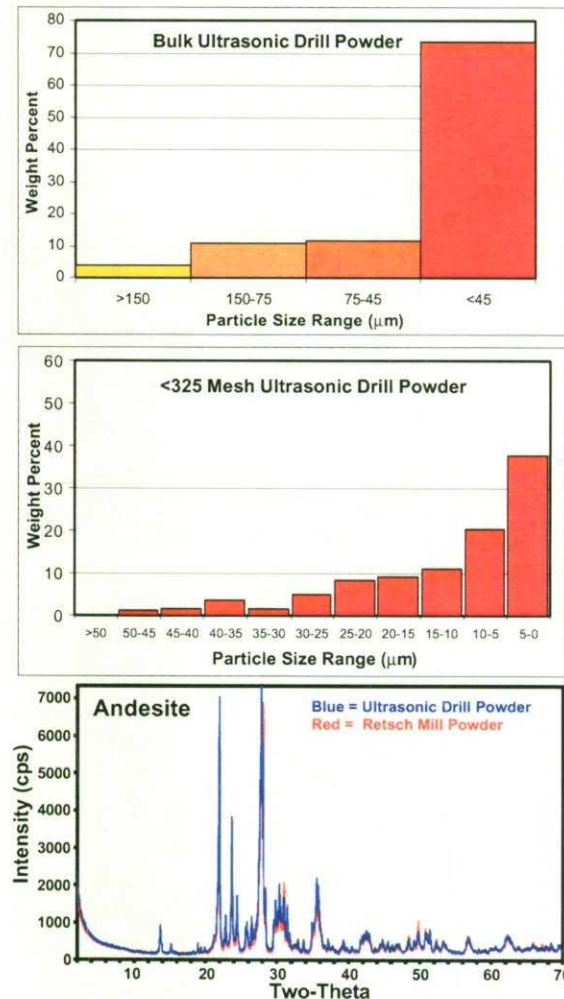


Figure 3: Andesite, Tschicoma Peak