PROGRESS IN THE DEVELOPMENT OF CHEMIN: A DEFINITIVE MINERALOGY INSTRUMENT ON THE MARS SCIENCE LABORATORY (MSL '09) ROVER. D.F. Blake[1], P. Sarrazin[2], D. L. Bish[3], S. J. Chipera[4], D. T. Vaniman[4], S. Collins[5] , S.T. Elliott[5] and A. S. Yen[5]. [1]NASA ARC, MS 239-4, Moffett Field, CA 94035 (dblake@mail.arc.nasa.gov), [2] In-Xitu, Inc., 2551 Casey Ave. Ste A, Mountain View, CA 94042, [3]Dept. Geological Sciences, Indiana University, Bloomington, IN 47405, [4]Hydrology, Geochemistry, and Geology, Los Alamos National Laboratory, MS D469, Los Alamos, NM 87545, [5]Jet Propulsion Laboratory, California Institute of Technology Pasadena, CA 91109

Introduction: An important goal of the Mars Science Laboratory (MSL '09) mission is the determination of definitive mineralogy and chemical composition of Mars soil and rocks. CheMin is a miniature X-ray diffraction/X-ray fluorescence (XRD/XRF) instrument that has been chosen for the analytical laboratory of MSL [1] (see figure 1). CheMin uses a micro-focus-source Co X-ray tube, a transmission sample cell, and an energy-discriminating X-ray sensitive CCD to produce simultaneous 2-D XRD patterns and XRF spectra from powdered or crushed samples. A diagram of CheMin's geometry is shown in figure 2.

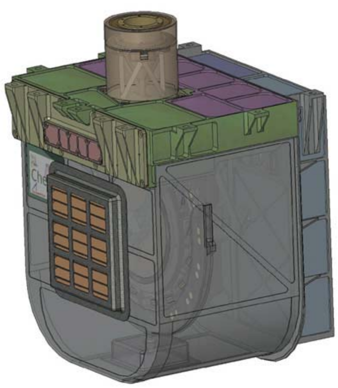


Fig. 1. *CAD model of CheMin MSL instrument. CheMin is ~ 30X30X30 cm, and has a mass of ~8 kg.*

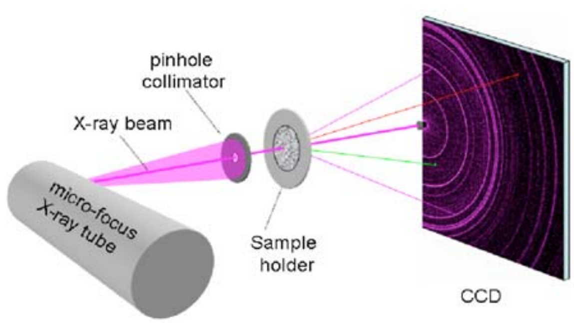


Fig. 2*. Diagram of CheMin XRD and XRF geometry. Diffracted primary beam CoKα X-rays (magenta) are identified by their energy. A 2-D image of these constitutes the XRD pattern. Fluorescence X-rays from the sample (multicolored) are summed into a histogram of photon energy vs. number of counts that constitutes the XRF spectrum.*

X-ray Source and Detector: CheMin has two X-ray sources: a high-resolution source optimized for 2θ resolution and a high-flux source optimized for flux. The sources, designed by Oxford X-ray Technology Group and Space Power Inc., consist of ceramic high-vacuum X-ray tubes integrated with a high-voltage power supply. The X-ray tubes use conventional W cathodes and reflection-type Co anodes. Focusing grids in the tubes yield a 50 µm diameter photon source on each anode. The tubes are nominally operated at 40 KeV accelerating voltage and 100 milliamps beam current. The tubes and integrated power supplies are contained in a sealed vessel of pressurized SF_6.

The CheMin CCD detector is a newly manufactured version of an E2V X-ray sensitive CCD-22 imager, called the CCD-224. The CCD-224 imager is a 600X600 front-illuminated frame transfer device having 40 µm square pixels, a deep depletion zone for high quantum efficiency of 7 KeV X-rays (CoKα), and a thin polygate structure for enhanced sensitivity to lower atomic number elements such as Mg.

Table 1: *Critical source and detector requirements for CheMin's high-resolution and high-flux geometries.*

Parameter	High-res. source	High-flux source
2θ range	5-50° 2θ	5-55° 2θ
2θ resolution	<0.35° 2θ	<0.5° 2θ
Total flux	5.6E5	2.8E6
Flux stability	<2% per hour	<2% per hour
CCD detection	1-13.3 KeV	1-13.3 KeV
CCD resolution		
@1.0 KeV	150 eV	150 eV
@5.9 KeV	200 eV	200 eV

Sample Acquisition and Delivery: During the one Mars year duration of MSL's mission, the Sample Acquision / Sample Preparation and Handling (SA/SPaH) system will deliver up to 74 samples of Mars soil and rock to the CheMin instrument. Samples are crushed and sieved to <150 µm particle size and delivered to one of 26 reusable transmission sample cells on the CheMin sample wheel. Samples are loaded at the top, analyzed, then discarded in a sump by rotating the sample cell to the bottom of the wheel.

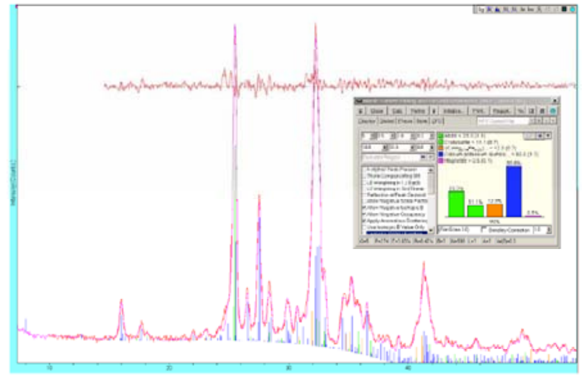
Sample Analysis: Samples are analyzed for up to 10 hours during multiple sols. During analysis, sample cells are shaken at sonic frequencies by a piezoelectric actuator that causes convection and granular flow of the powdered material. The result is that all components of the sample are passed through CheMin's ~50µm diameter beam in random orientations during the course of an analysis. In addition to the 26 reuseable sample cells, CheMin has 6 sealed cells containing XRF and XRD reference standards. These standards can be analyzed at any time to evaluate the health of the instrument or to generate XRF elemental working curves or calibrated XRD patterns.

Data Reduction: For each analysis, CheMin will produce energy-selected 2-D CoKα and CoKβ patterns (similar to that shown in Figure 2), as well as a histogram of X-ray energy vs. number of photons.

Downlinked data from Mars will be validated in three ways: First, on-board standards will be used to calibrate unknowns and to control for non-optimal analysis conditions during the lifetime of the mission. Second, laboratory-based CheMin instruments will be used to analyze Mars analog materials. These instruments include the CheMin Engineering Model, the CheMin IV field instrument, and a laboratory INEL diffractometer equipped with a Mars atmosphere chamber and CheMin-like sample cell. Third, a ray-tracing model has been developed to simulate the CheMin geometry and corroborate diffraction results from specific sample mineralogies.

XRD. 2-D patterns are converted into conventional 1-D 2θ diffractograms by summing the patterns circumferentially about the central beam. The diffractograms are analyzed using Fullpat [2] and Rietveld refinement [3] methods. Figure 3 shows a 1-D XRD pattern and Rietveld refinement of an andesite standard (P-52) from Los Alamos National Laboratory. This pattern was obtained using CheMin MSL geometry and X-ray fluxes and acquisition times typical of those planned for the Mars instrument. Table 2 shows quantitative results for this analysis vs. a full laboratory analysis performed using a Siemens D500 instrument.

XRF. Background and Gaussian peaks will be fit to returned XRF spectra. Working curves will be used to produce quantitative elemental abundances with calculated uncertainties [4]. CheMin will detect and quantify elements Mg-Fe, and qualitatively determine the presence of other elements in major, minor, or in some cases trace concentrations.



Refinement above used albite, bytownite, augite, cristobalite, and magnetite

Fig. 3. *XRD pattern and Rietveld refinement of P-52 Andesite standard using CheMin geometry and SA/SPaH-like sample preparation.*

Table 2: *Quantitative XRD analysis of P-52 (wt. %) using a laboratory Siemens D500 instrument vs. a CheMin prototype. "Normalized data" are observed minerals from CheMin, normalized to 100% of amount observed.*

Mineral	Siemens D500	Siemens D500 (Normalized)	CheMin
Plagioclase	71	79	76
Augite	4	10	12
Enstatite	5		nd*
Cristobalite	8	9	11
Magnetite	3	3	0.5
Smectite	5		nd
Carbonate	3		nd
Apatite	1		nd
Olivine	1		nd

* not detected

Summary: CheMin's unique sample vibration system allows it to accept powders from the SA/SPaH system and to obtain representative intensity data little affected by particle statistics or preferred orientation. Diffraction data will be analyzed by methods that use the entire diffraction profiles, and quantitative mineralogic results will be obtained through a combination of diffraction and chemical data. CheMin will for the first time, provide definitive mineralogic information on remote samples on the martian surface.

References: [1] Sarrazin et al. (2005) Powder Diff. **20**(2),128-134. [2] Chipera and Bish (2002) J. Appl. Cryst., **35**,744-749. [3] Bish and Post (1993) Amer. Min. **78**,932-942. [4] Sherman (1955) Spectrochim. Acta. **7**,283-306.