CHEMIN: A DEFINITIVE MINERALOGY INSTRUMENT ON THE MARS SCIENCE LABORATORY (MSL '09) ROVER.
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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 (XRD) instrument that has been chosen for the analytical laboratory of MSL [1]. CheMin uses a microfocus-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 energy-dispersive X-ray histograms from powdered samples. A diagram of the instrument geometry is shown in Figure 1.

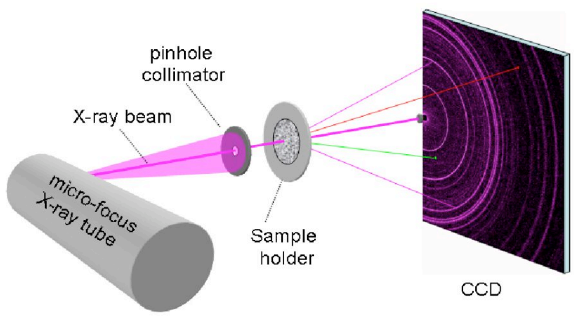


Fig. 1. Diagram of CheMin XRD geometry. Diffracted primary beam CoKα X-rays (magenta) are identified by their energy. A 2-D image of these constitutes the XRD pattern. All photons are summed into a histogram of photon energy vs. number of counts that provides a qualitative elemental composition of the sample.

X-ray Source and Detector: CheMin has a microfocus Co X-ray source optimized for 2θ resolution. The source, designed by Oxford X-ray Technology Group and Space Power Inc., consist of a ceramic high-vacuum X-ray tube integrated with a high-voltage power supply. The X-ray tube uses a conventional W cathode and a reflection-type Co anode. A focusing grid in the tube yields a 50 µm diameter photon source on the anode. The tube will be operated at 28 KeV accelerating voltage and 200 milliamps beam current. The tube and integrated power supply 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.*

Parameter	Value
2θ range	5-50° 2θ
2θ resolution	<0.35° 2θ
Total flux	5.6E5
Flux stability	<2% per hour
CCD detection	1-13.3 KeV
CCD resolution	<250 eV @CoKα KeV

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 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 4 sealed cells containing XRD reference standards. These standards can be analyzed at any time to evaluate the health of the instrument or to generate calibrated XRD reference patterns.

Data Collection Algorithms. Because the volume of raw data generated during a 10-hour analysis exceeds the down-link capacity of the spacecraft, some data processing must be performed on-board prior to its transmission to Earth. Several modes of data collection/processing are programmed into CheMin's Field Programmable Gate Array (FPGA) which are individually selectable during an experiment:

- *Raw frame mode:* A specified number of raw frames is collected and transmitted to Earth for diagnostic purposes.
- *Film mode:* Background-subtracted frames are summed into a single image, yielding a 2-D diffraction pattern.
- *Run-length encoded mode:* Background pixel values are set to zero and remaining pixel data are compressed by run-length encoding. The resulting data are reassembled into images after transmission to Earth.
- *Fully processed mode:* Energy-discrimination is used to create background-subtracted 2-D CoKα and CoKβ diffraction patterns. An X-ray energy histogram is constructed which provides the elemental composition of the sample.

Data Reduction: In its preferred mode of data processing, 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.

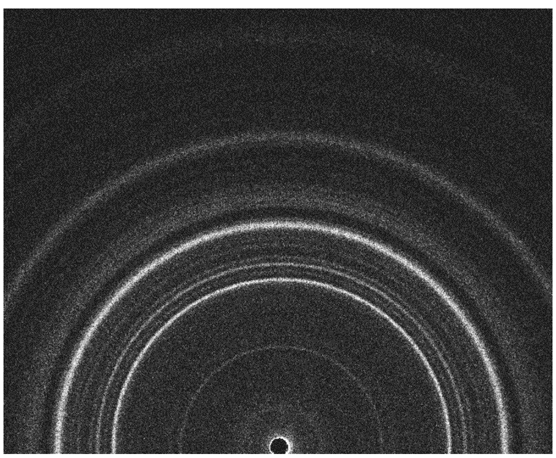
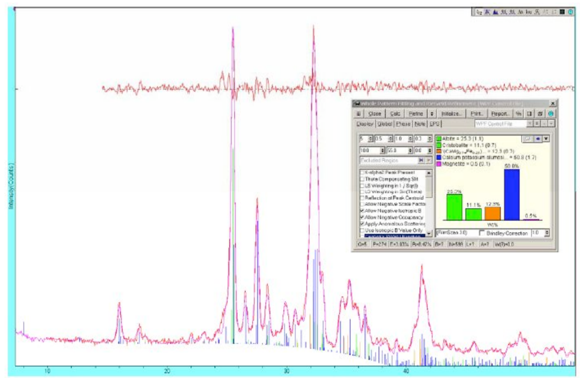


Fig. 2. *2-D CoKα diffraction pattern of P-52 Andesite standard using CheMin geometry and SA/SPaH-like sample preparation.*

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, several CheMin IV field instruments 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 the andesite pattern shown in Figure 2. 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.



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

Fig. 3. 1-D *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

XRF. Background and Gaussian peaks will be fitted to returned energy-dispersive X-ray spectra. Qualitative elemental compositions will be used to constrain quantitative mineralogic analyses. CheMin quantitative mineralogic analyses. CheMin will detect and quantify elements Mg-Fe in major, minor, and in some cases trace concentrations.

Level 2 Science Requirements for CheMin:

Detection limit – Ability to achieve a detection limit of at least 3 weight % for crystalline phases.

Quantitative XRD accuracy – Ability to determine abundance of crystalline mineral phases present at >4X the detection limit to an accuracy of 15% relative (1σ).

Quantitative XRD precision – Ability to achieve 1σ precision of better than 10% relative in abundance determination for crystalline phases present at >4X the detection limit.

Analysis time – Ability to meet detection limit, accuracy and precision specifications with an integration time of 10 hours on any given sol.

Cross-contamination limit – Ability to reuse sample chambers with <5% contamination from previous analyses.

Identification of minerals in mixtures – Ability to distinguish unique miner4als at abundances above detection limits in a rock matrix.

The CheMin IV field instrument: A field-portable CheMin instrument was built and tested in a variety of Mars analog environments including Death Valley, California (USA), Svalbard (Norway) and Rio Tinto (Spain). Raw data are collected by an imbedded computer and processed using the same algorithms to be implemented in the MSL CheMin instrument. Figure 4 shows the CheMin IV instrument during its deployment at Rio Tinto.

Fig. 4: *CheMin IV instrument at Rio Tinto with (optional) laptop computer for graphic user interface.*

Figure 5 shows an efflorescent salt that was hand collected and analyzed on-site by CheMin IV. Fig. 6 shows the resulting 2-D CoKα pattern that was collected after only 18 minutes of integration time (due to time constraints in the field, CheMin IV was operated at approximately 4X the flux of the MSL instrument).

Fig. 5. *Efflorescent salt collected in an evaporated pool of the river bed.*

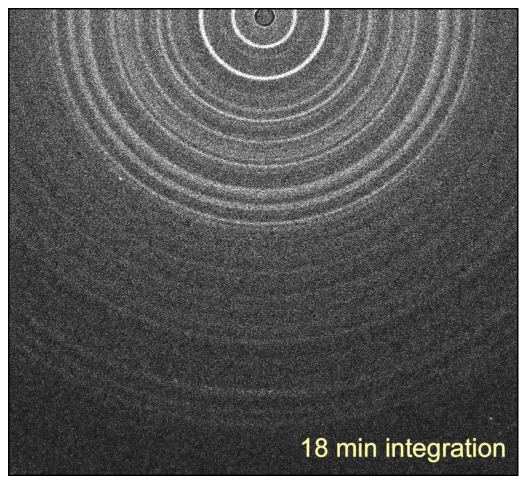


Fig. 6. *2-D CoKα pattern of efflorescent salt collected from Rio Tinto river bed.*

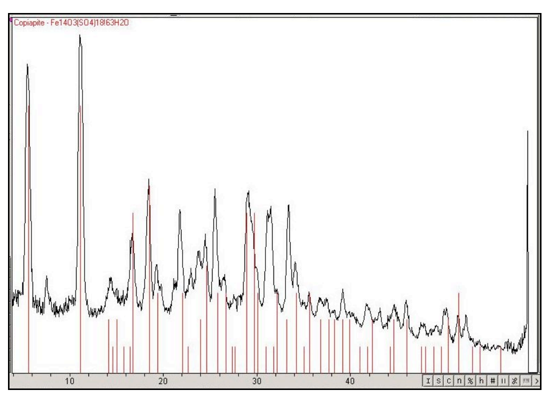


Fig. 7. *1-D diffractogram from Fig. 6. Phase identification (copiapite) was performed using the commercial search-match program JADE^{TM}.*

The CheMin MSL instrument: CheMin (Figure 8) will be a compact, lightweight autonomous instrument capable of collecting XRD data at resolutions acceptable for advanced data analyses such as Rietveld refinement. CheMin's unique sample vibration system allows it to accept powders from the SA/SPaH system and 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.

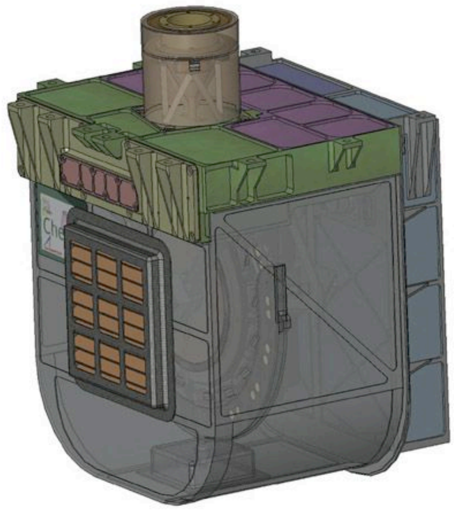


Fig. 8. *CAD model of CheMin MSL instrument. CheMin is ~ 30X30X30 cm, and has a mass of ~8 kg.*

References: [1] Sarazin 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.