

THE X-RAY FLUORESCENCE CAPABILITIES OF CHEMIN IV: DATA REDUCTION AND CALIBRATION. S. M. Chemtob¹, A. Yen², and D. F. Blake³. ¹Division of Geological and Planetary Sciences, Caltech, Pasadena, CA, 91125. (chemtob@gps.caltech.edu) ²Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA. ³NASA Ames Research Center, Moffett Field, CA.

Introduction: Quantitative mineralogical analysis is fundamental for characterizing and understanding the nature and history of geological unknowns. CheMin is the primary instrument on the Mars Science Laboratory (MSL) rover for determining mineralogy. CheMin is a miniaturized X-ray diffraction (XRD) instrument with transmission geometry that also features X-ray fluorescence (XRF) capabilities (see also [1]). It features a cobalt X-ray tube, sample wheel with reusable sample cells, and an energy-sensitive CCD detector. Sample cells are 8 mm in diameter and 175 μm thick, bounded on either side by Mylar or Kapton windows. During an analysis, collimated X-rays are transmitted through a sample, and then the CCD, positioned on the opposite side, detects diffracted and fluoresced photons. These detections are integrated over multiple exposures to produce both 2θ diffraction patterns and energy dispersive spectra (EDS).

Although CheMin is included in the MSL instrument suite primarily for its XRD, its XRF capabilities are potentially valuable for establishing additional context about unknowns on the martian surface. Chemical compositions derived from XRF spectra will be used to constrain the mineralogy derived from XRD patterns. In addition, CheMin XRF will be cross-calibrated with ChemCam and APXS, both instruments that provide information about chemical composition. XRF data will then provide confirmation that samples delivered to the MSL platform are the same materials as those measured by arm and mast instruments. Effective use of the CheMin XRF requires careful and thorough calibration. Here we present preliminary efforts to reduce CheMin EDS and to produce calibration curves for compositions of geological relevance. Because the developmental model (DM) is not yet available and the flight model (FM) is still in testing, all measurements were conducted on the CheMin IV, a laboratory testbed with similar geometry and capabilities.

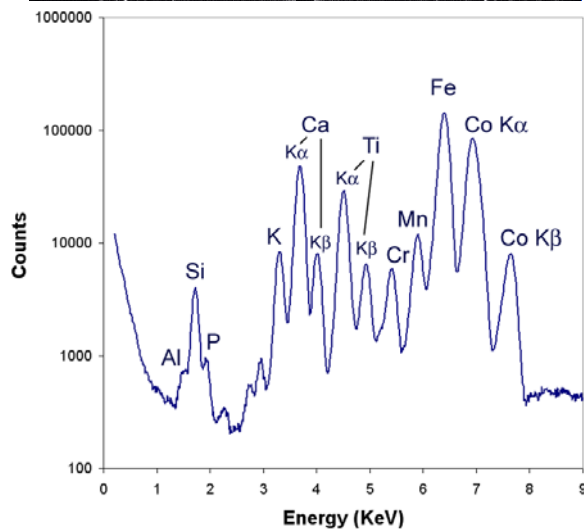
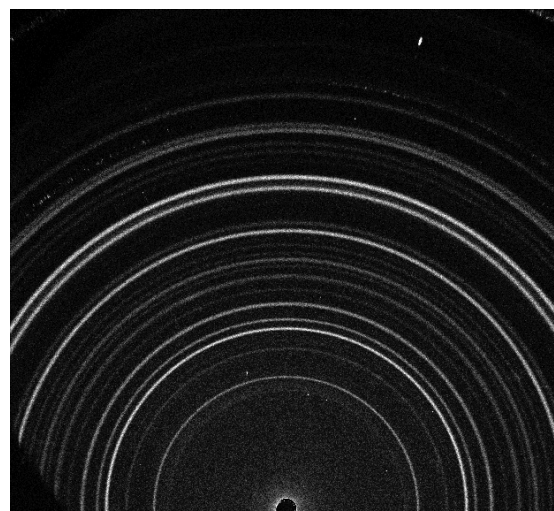


Figure 1. CheMin data products. a) An example of an integrated CCD display from the FM. Bright rings correspond to diffraction bands; fluoresced pixels have no spatial correlation. b) The EDS of BCR-2 basalt, collected on CheMin IV. Co peaks correspond to diffracted source radiation.

Data Reduction: The energy dispersive spectrum is derived from the CCD by recording the digital number (DN) associated with each pixel for each exposure, then plotting counts against DN (Fig. 1). DN can be converted to energy using the position of a recognizable peak, such as Co K α (6.93 KeV). An unprocessed EDS will have a high background, partially a result of continuum

bremsstrahlung radiation, but also due to photons split over two or more pixels. To reduce the background level, only single-pixel hits are counted. Post-processing of the raw CCD data is performed to maximize the signal-to-noise ratio.

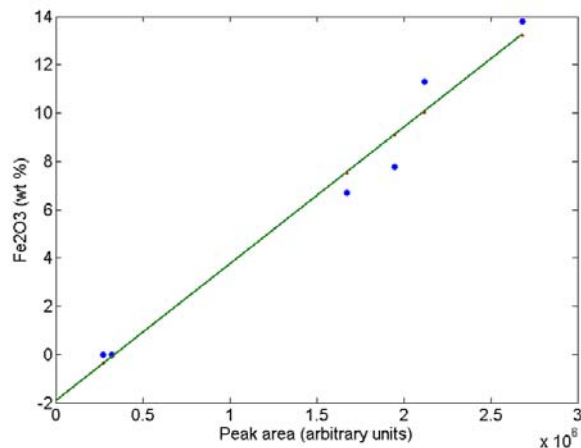


Figure 2. Calibration curve with geostandards for Fe. Predicted concentrations typically fell within 10% of certified levels.

Once the background level is optimized, a search algorithm is used to locate local maxima and minima; using these lists, the elements present are determined. Each elemental peak is fit to a theoretical shape, consisting of a Gaussian curve plus a low-energy tail [2]. Peaks that include significant overlap, such as Al and Si $K\alpha$, are fit simultaneously. If a $K\alpha$ peak of an element can be fit but the $K\beta$ peak interferes with another peak, the $K\beta$ peak is subtracted from the spectrum according to the theoretical $K\alpha/K\beta$ ratio. Peak areas are calculated for each element based on the functional form.

Calibration: We used certified geostandards and high-purity oxides for initial calibration on the CheMin IV. Although the mean grain size of these geostandards is typically well below 45-90 μm , the ideal size for CheMin, high-quality spectra were produced. Standards measured include BCR-2, BIR-1, AGV-2, DTS-2b, the MSL organic check material (OCM) consisting of amorphous silica, Mn_2O_3 , TiO_2 , and carbon. We first observed the relationship between concentration and peak area without considering matrix effects. To account for matrix effects, a basic Lucas-Tooth Pyne intensity correction model is employed [2]:

$$P_{nm}^{\text{x-ray}} = \alpha_n + P_{nm}^{\text{app}}(K_0 + \sum K_j P_{jm}^{\text{app}})$$

where P_{nm}^{app} is the apparent concentration of element n in sample m , K_0 and α_n are the slope and intercept terms and K_j are the interaction parameters for each element. Many analyses would be required to solve for all the interaction parameters uniquely, so two approaches can be taken: 1) solving for the most influential interaction parameters and leaving the rest equal to zero, and 2) using theoretically derived values for all parameters. Both approaches will be explored in future work.

Results: The optimum elemental range for analysis by CheMin XRF is $Z=14$ through 26. X-rays produced by elements lighter than Si are easily absorbed in transmission geometry, resulting in high detection limits for Na, Mg and Al. Only Co bremsstrahlung radiation is available to excite elements with $K\alpha$ energies above 6.93 KeV. Therefore, calibration efforts will focus on this optimum range.

Most elements perform very well under the linear model, even without matrix corrections (Fig. 2). The model generally fit each elemental concentration within 10-15% relative. One exception was Si, which suffered from strong matrix effects that will be addressed in the future. In addition, contamination in the CheMin IV led to nonzero intercepts for several elements, including Si, Ti and Fe. This issue should be mitigated or eliminated with the DM and FM.

These preliminary results suggest that CheMin XRF can provide accurate and precise chemical information for a range of geological compositions. Future work will include additional standard analyses, detection limit studies, and cross-calibration with the CheMin DM.

References: [1] Blake et al., this conference. [2] Jenkins et al. (1995) Quantitative X-ray spectrometry, 2nd ed., 484 p.