CheMin: Software for X-ray Diffraction

1) Software for Conversion of 2D CCD images to 1D diffraction patterns

FILMSCAN. The 2-D patterns of CheMin CCD Debye rings are converted to 1-D intensity-2θ patterns using the program FILMSCAN. FILMSCAN is a commercially available program that can be purchased through MDI, Inc. (www.materialsdata.com/products.htm). FILMSCAN can read film raw data in 2-D and convert the data into a 1-D pattern for XRD mineral identification and quantification. Other software for performing this function is possible and may be developed as the mission progresses.

2) Software for analysis of CheMin instrumental parameters

LINEPROFILE. LINEPROFILE is a data reduction and calibration package being developed at Indiana University that will determine the instrumental broadening function from 1-D datasets. The program will describe the observed profiles with a combination of empirical profiles and back out the instrumental broadening function. LINEPROFILE will use datasets created by 2-D/1-D to test and validate the capability of the analysis to back out the instrument broadening function. Much of the analysis of line profiles will be done using the proprietary program TOPAS, which has very broad flexibility in terms of the number of analytical profile functions available and also includes the possibility of using a fundamental parameter description of the CheMin geometry.

3) Software for mineral identification and quantification

Several standard programs for handling CheMin 1D X-ray diffraction data are listed below. The examples provided are listed in an approximate sequence of increasing complexity and user knowledge, although ease of use is often subjective and depends on the user's background and training. Other programs for handling standard 1D X-ray diffraction data are available and use of such programs depends only on user preference.

XPOWDER. XPowder is a commercial program that supports phase identification using Powder Diffraction File (PDF) standard patterns (www.xpowder.com). XPowder can also be used with data in Crystallographic Information File (CIF) format available in the American Mineralogist Crystal Structure Database (rruff.geo.arizona.edu/AMS/amcsd.php). Quantification of multi-phase samples may be performed using least-squares pattern-fitting and normalized reference intensity ratio (RIR) methods.

JADE. Jade is a commercial program that supports the comprehensive analysis of X-ray diffraction patterns, including phase identification, peak profile fitting, indexing, unit cell refinement, Rietveld analysis, etc. JADE has been developed by Materials Data Incorporated (MDI) and requires a commercial license (www.materialsdata.com/products.htm).

TOPAS. TOPAS is a commercially available program that uses 1D XRD data to conduct qualitative and quantitative mineralogical analysis. TOPAS requires a commercial license

(www.bruker-axs.com/topas.html). TOPAS integrates many available profile fitting techniques as well as related applications such as single line fitting, whole powder pattern decomposition, ab-initio structure determination, and quantitative Rietveld analysis.

FULLPAT. FULLPAT is a quantitative X-ray diffraction methodology developed at Los Alamos National Laboratory (LANL) that merges the advantages of existing full-pattern fitting methods with the traditional reference intensity ratio (RIR) method. FULLPAT uses complete 1D diffraction patterns generated by CheMin, including the background, in conjunction with standard patterns measured on an equivalent instrument on Earth (e.g., the CheMin DM). FULLPAT uses as standard-reference patterns either instrument-specific measured patterns (preferred) or calculated patterns. FULLPAT has been coded into Microsoft EXCEL using standard spreadsheet functions. The program is available as freeware (see Appendix A) and has already been tested by the CheMin team. Effective use of FULLPAT with CheMin data will require measurement of a set of standard patterns in the laboratory on the CheMin DM. Calculated patterns can also be used if they include simulation of the CheMin profile shapes. Practical use of FULLPAT and free access to FULLPAT for CheMin data will be cited in publications by the CheMin team as DM results become available.

GSAS. GSAS (General Structure Analysis System) is a comprehensive package for the refinement of structural models using both X-ray and neutron diffraction data. The GSAS package can use 2D/1D powder diffraction data to perform Rietveld analysis, including analysis of peak profiles, refinement of unit-cell parameters, quantitative multicomponent analysis, and, in some cases, full crystal structure refinement. The package is in the public domain and requires no development (http://www.ncnr.nist.gov/xtal/software/downloads.html). However, use of GSAS requires considerable skill on the part of the operator and knowledge of crystallography. Similar analyses will be done with the program TOPAS (see above), which can include the ability to define fundamental instrument parameters that control measured profiles.

APPENDIX A: FULLPAT

FULLPAT: A FULL-PATTERN QUANTITATIVE ANALYSIS PROGRAM AND METHOD FOR X-RAY POWDER DIFFRACTION

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FULLPAT is a quantitative X-ray diffraction methodology that merges the advantages of existing full-pattern fitting methods with the traditional reference intensity ratio (RIR) method [1]. FULLPAT can explicitly analyze all phases in a sample including partially ordered or amorphous phases such as clay minerals, polymers, or glasses. Use of an internal standard allows unconstrained analyses to be conducted by direct fitting of library standard patterns to each phase in the sample. By fitting entire patterns, including background, all phases (including amorphous or disordered components) are explicitly included in a FULLPAT analysis. Amorphous content need not be determined as the difference from 100% (*i.e.*, %amorphous = 100% - Σ crystalline phases) but can be measured directly. If all individual library standards are normalized to be equal on the basis of the intensity of the corundum internal standard, an "external standard" or "adiabatic" method analysis can be conducted without addition of an internal standard to the unknown, but with the constraint that the abundances of all phases sum to 100%. The method is coded in Microsoft EXCEL using standard spreadsheet functions and uses least-squares minimization to optimize the fit between the sum of standard patterns and the observed pattern [2]. The method has been applied to many natural rock samples, including those containing disordered minerals such as clay minerals and amorphous components such as volcanic glass. Results for clay mineral-containing mixtures and for internal-standard analyses of amorphouscontaining mixtures were excellent, with values typically within several percent of the known amounts. Tests with numerous standard mixtures containing well-ordered, disordered, and amorphous components gave an overall uncertainty of 3.4 wt.% at the 95% confidence level. It is important to emphasize that these tests were done with known mixtures, using standards that were *different* from the components in the mixtures. The precision of analysis would be considerably improved for repetitive analyses using standards that were matched to the unknowns.

[1] Chipera, S.J., and Bish, D.L. (2002) FULLPAT: A full-pattern quantitative analysis program for X-ray powder diffraction using measured and calculated patterns. J. Applied Crystallography, 35, 744-749.
[2] The program can be downloaded from:
http://www.ccp14.ac.uk/ccp/web-mirrors/fullpat/