

SOFTWARE NOTICE

Interactive software for calculating and displaying X-ray or neutron powder diffractometer patterns of crystalline materials

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ABSTRACT

Two computer programs, XPOW and XPOWPLOT, that generate and graph X-ray or neutron powder diffractometer patterns of crystalline materials are described. The input for XPOW requires only the radiation wavelength, cell dimensions, space group, and positional parameters for the atoms in the asymmetric unit. The output includes a listing of the d values, 2θ values, and the relative intensities for the nonequivalent Bragg reflections within a given 2θ interval. Using the XPOW output, the XPOWPLOT program creates menu-aided interactive color displays of up to five powder diffractometer patterns simultaneously on the PC monitor.

INTRODUCTION

The powder diffraction record is a set of data that can be used to identify and to characterize the structure of a crystalline material. Diffraction records for a large variety of such materials have been tabulated by the American Society of Testing Materials (ASTM). In addition, X-ray diffractometer patterns have been calculated and graphed for a variety of silicate minerals (Borg and Smith, 1969), and several programs have been coded that generate theoretical powder diffractometer patterns (Yvon et al., 1977; Smith et al., 1983; Bish and Post, 1989; and references therein; Smrčok and Weiss, 1993).

In this note, two programs written for the PC are described. The program XPOW calculates a theoretical X-ray or neutron powder diffraction record, and the program XPOWPLOT graphs it on the screen. The XPOW program requires a minimum amount of structural information, especially when compared with existing programs. The many options available within the XPOWPLOT program allow the user to make a careful study of the diffraction pattern through visual displays. The ability to input multiple sets of data into XPOWPLOT, for example, from an ASTM file, permits a comparison of patterns on the screen of the PC. The programs were written to obtain an unambiguous indexing of the observed diffraction pattern of a known material and as a pedagogical tool to help students understand and interpret powder

diffractometer patterns. The programs are user-friendly and will be especially useful for those who have a limited knowledge of space-group and structural theory.

PROGRAM XPOW

The execution of the XPOW program requires an ASCII input data file. This file may be created in free format using any standard DOS editor, or it may be one of several hundred data files provided with the software. Table 1 is an example of an input data file containing two data sets created for β eucryptite (Tscherry et al., 1972), which are separated by a blank line. The first data set uses X-ray radiation and the second data set uses neutron radiation. Each data set requires that the first line must contain a title, with a maximum length of 80 characters. The second must contain the radiation wavelength, λ , or the chemical symbol of the target X-ray source (Li-U) and the limits of a 2θ interval between which data will be calculated, $2\theta_{\text{left}}$ and $2\theta_{\text{right}}$. The chemical symbol of the target X-ray source represents $K\alpha$ radiation (e.g., Cu), and the chemical symbol followed by the number 1 (e.g., Cu1) or 2 (e.g., Cu2) represents $K\alpha_1$ or $K\alpha_2$ radiation, respectively. Furthermore, both $K\alpha_1$ and $K\alpha_2$ radiation may be entered simultaneously (e.g., Cu1 Cu2). If neutron radiation is used, then the radiation wavelength must be entered as a number and the limits of the 2θ interval must be followed by the letter "n."

The third line of a data set must contain the six unit-cell parameters ($a, b, c, \alpha, \beta, \gamma$) and a short space-group symbol or number as given in Table 6.2.1 of the *International Tables for X-ray Crystallography, Volume 1* (Henry and Lonsdale, 1965). In Table 1, the space group

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Table 1. An example of an XPOW data file.

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=====
X-ray beta eucryptite: Tscherry et al. (1972) Z Krist 135, 161-174
Cu 10 60
5.2409 5.2409 5.5875 90 90 120 P6_222
Si .5 0 0 .5 .41
Al .5 0 0 .5
Li 0 0 0 1 1.5
O .2017 .4034 .5

Neutron beta eucryptite: Tscherry et al. (1972) Z Krist 135, 161-174
1.541838 10 60 n
5.2409 5.2409 5.5875 90 90 120 P6_222
Si .5 0 0 .5 .41
Al .5 0 0 .5
Li 0 0 0 1 1.5
O .2017 .4034 .5
=====

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Note that each line is unformatted.

symbol is P6_222 and represents the space group $P6_222$. Any space group with a nonstandard setting is easily handled, the implementation being described fully in accompanying documentation. Each of the following lines must contain the chemical symbol of an atom and its three positional parameters, x , y , and z , in the asymmetric unit. Optional input includes the occupancy factor, O , and the isotropic temperature factor, B . For example, the data for the Si atom in Table 1 includes values of 0.5, 0, 0 for the positional parameters, 0.5 for the occupancy factor, and 0.41 \AA^2 for the temperature factor. The data for the Al atom illustrates mixed occupancy because it has the same positional parameters as the Si atom and 0.5 for its occupancy factor. If no values are entered for the occupancy or the temperature factors (Table 1, e.g., O), then the program assigns a default value for the temperature factor specific to the atom and an occupancy factor of 1.0. The occupancy factor may be entered without an isotropic temperature factor (Table 1, e.g., Al), whereas input of the temperature factor must be preceded by an occupancy factor (Table 1, e.g., Li).

To run the program, type "xpow filename.pow filename.out," where filename.pow (File unit 4) is the name of the input data file and filename.out (File unit 7) is an arbitrary name given to the output file. After reading a data set, XPOW generates the positional parameters of all of the atoms within the unit cell and, for each atom, assigns neutral pseudoatom scattering factors for X-ray radiation (Lonsdale, 1985) or bound coherent scattering lengths for neutron radiation (Sears, 1992). Given the larger of the 2θ limits, $2\theta_{\max}$, the program determines the limits imposed on the indices of the Bragg reflections, (hkl) , according to

$$h_i = \frac{2 \sin(2\theta_{\max}/2)}{\lambda \sqrt{G'_{ii}}} \quad (1)$$

Table 2. An example of the file output by XPOW.

```

=====
X-ray beta eucryptite: Tscherry et al. (1972) Z Krist 135, 161-174
CELL PARAMETERS: 5.2409 5.2409 5.5875 90.000 90.000 120.000
SPACE GROUP: P6_222

ATOM      X      Y      Z      OCCUPANCY  ISO(B)
Si      .50000  .00000  .00000  .500      .410
Al      .50000  .00000  .00000  .500      .600
Li      .00000  .00000  .00000  1.000     1.500
O       .20170  .40340  .50000  1.000     1.000

X-RAY WAVELENGTH: 1.541838
BOUNDS ON TWO THETA: 10.0 60.0
LIMITS IMPOSED ON THE INDICES ARE: +/- 2 +/- 2 +/- 3
MAX. ABS. INTENSITY / VOLUME**2 : 33.27954719

2-THETA  INTENSITY  D-SPACING  H  K  L
19.56    4.55      4.5388    1  0  0
25.28   100.00    3.5229    1  0  1
34.22    2.44      2.6205    1  1  0
37.81    1.37      2.3792    1  0  2
37.92    .01       2.3725    1  1  1
39.72    5.02      2.2694    2  0  0
43.02    3.06      2.1026    2  0  1
47.58   20.34    1.9113    1  1  2
48.90    .04       1.8625    0  0  3
51.91    1.42      1.7615    2  0  2
53.16    .06       1.7231    1  0  3
53.41    .41       1.7155    2  1  0
=====

```

where $h = h_1$, $k = h_2$, $l = h_3$, and G'_{ii} represents the i th diagonal element of the reciprocal metrical matrix (Boisen and Gibbs, 1990). The program calculates $|F_{hkl}|^2 = A_{hkl}^2 + B_{hkl}^2$, where

$$A_{hkl} = \sum_{j=1}^n O_j f_j e^{(-B_j s^2)} \cos 2\pi(hx_j + ky_j + lz_j)$$

$$B_{hkl} = \sum_{j=1}^n O_j f_j e^{(-B_j s^2)} \sin 2\pi(hx_j + ky_j + lz_j)$$

n is the total number of atoms contained in the unit cell, O_j is the occupancy factor, f_j is the scattering factor or length, B_j is the isotropic temperature factor, x_j , y_j , and z_j are the positional parameters for the j th atom, and $s = \sin(\theta/\lambda)$ for the Bragg reflection (hkl) . The relative intensity, I_{hkl} , calculated for this reflection is given by $I_{hkl} = m I_o Lp |F_{hkl}|^2$, where the Lorentz factor, $Lp = (1 + \cos^2 2\theta) / (\sin^2 \theta \cos \theta)$ for X-ray radiation and $Lp = 1 / (\sin^2 \theta \cos \theta)$ for neutron radiation, m is the multiplicity or order of the orbit of (hkl) , and the factor $I_o = [I_o = 100 / \max(I_{hkl})]$ scales the highest relative intensity to 100.

The contents of the output file, filename.out, generated for the first data set in Table 1 is given in Table 2. It contains a listing of parameters used in the calculation, including default values assigned by the program and the limits imposed on (hkl) as computed by Equation 1. In addition, the maximum absolute intensity per unit-cell volume squared, $100 I_o / V^2$, is generated for use in multiple phase analyses. Finally, the calculated diffraction

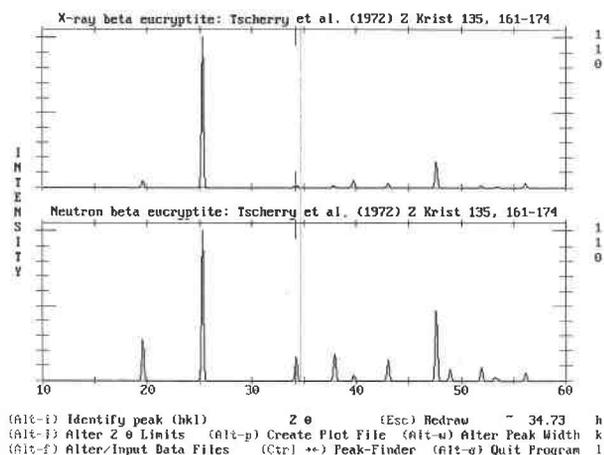


Fig. 1. An example of the image provided when viewing multiple data sets in XPOWLOT. The actual screen image is in color. The patterns for β eucryptite were generated from the XPOW output listed in Table 2.

record is tabulated in a manner similar to that used by the ASTM files.

PROGRAM XPOWLOT

To obtain an interactive, colored, graphical representation of the powder diffractometer patterns tabulated in filename.out, type "xpowplot." Appearing on the screen is the program title, a menu of various program options, and a prompt requesting the name of the file filename.out. At this point, the user may type the name of the file, filename.out, or execute various program options by striking the hot keys, which are listed in the menu. The user may view the list of the files in the current directory with the extensions pow or out by striking Alt P or Alt O, respectively. These options are useful when the extensions pow and out are adopted for naming XPOW input and output files. Alternatively, the program XPOW can be executed by striking Alt X. In addition, striking Alt D temporarily places the user in the DOS system. The user is then free to change directories, edit files, execute other programs, and so forth. Striking Esc terminates the program execution.

After the user has entered filename.out, the program reads the tabulated values of $2\theta_{hkl}$ and I_{hkl} and creates an xy plot of each diffraction pattern (Fig. 1). The intensity is calculated as the superposition of Gaussian functions centered at $2\theta_{hkl}$ and with half-widths, σ_{hkl} , according to

$$y = 0.0001 + \sum \frac{I_{hkl}}{\sigma_{hkl}} e^{-(x-2\theta_{hkl})^2/2\sigma_{hkl}^2}$$

The half-widths are calculated as linear functions of 2θ , adapted from Borg and Smith (1969): $\sigma_{hkl} = \sigma_0(1 + 2\theta_{hkl}/140)$, where σ_0 has a default value, $\sigma_0 = 1/80 + |2\theta_{left} - 2\theta_{right}|/800$. The extent and orientation of the plot is determined by the order in which the limits of the 2θ inter-

val are entered, with $2\theta_{left}$ on the left and $2\theta_{right}$ on the right.

A menu of various plot options can be displayed below the plot of striking the Enter key (Fig. 1). These options allow the user to analyze the diffraction pattern and to alter various settings of the plot. Different viewing limits may be entered by striking, Alt L. This feature can serve as a zoom-in function or to reverse the orientation of the plot. The user may enter a new half-width, σ_0 , by striking Alt W, overriding the default value. This option is used to manipulate the shape of the peaks. Using the arrow keys, a vertical line may be moved across the pattern with its 2θ position indicated below the lower right of the plot. By striking Alt I, the peak closest to the vertical line is identified with a tick mark and its indices (hkl) are displayed to the right of each plot (Fig. 1). The color of these indices is altered if the peak is composed of multiple reflections. Placing the vertical line over the tick mark can be used to determine the precise peak position. All of the above options may be interrupted by striking the Esc key. Striking Alt P produces an ASCII file containing the raw xy data that can be imported to a graphic package of the user's choice.

An option is also available for comparing multiple powder diffraction patterns contained in separate output files. By striking Alt F, a data file menu is displayed, wherein the user can enter or delete up to five XPOW output files. Program options invoked by striking Alt X, Alt O, Alt P, or Alt D are available within this menu. Upon exiting the menu, each of the patterns is separately drawn within a split-screen format that includes all of the display options described above (Fig. 1). Using this multiple pattern option, the user can easily compare diffraction patterns to study such things as polymorphism, solid solutions, the effects of chemical substitution, thermal vibrations, and change in radiation, or even to identify unknown patterns.

AVAILABILITY

The programs were written in Microsoft Fortran 5.1 and Microsoft QuickC 2.5 and occupy <200 K. XPOW should run on any PC, but XPOWLOT requires at least an EGA monitor with a graphics card. The program package (documentation, executable code, and an extensive set of about 300 data files) is available by sending a formatted diskette and a self-addressed stamped envelope or a check for US\$20.00 to any of the authors.

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