

New procedure to obtain Bragg-reflection intensities from FULLPROF suite for powder crystal-structure determination using GEST and PECKCRYST programs

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A useful procedure is described to rapidly obtain Bragg-reflection intensities from the FULLPROF suite, and the Bragg intensities can then be input into the GEST and the PECKCRYST programs for crystal-structure determination of small molecules. An example on using the new procedure for the structure determination from powder diffraction determination of hydrochlorothiazide ($C_7H_8ClN_3O_4S_2$) is presented, and the powder-structure results obtained by the PECKCRYST program are in good agreement with previously reported single-crystal results. © 2010 International Centre for Diffraction Data. [DOI: 10.1154/1.3501976]

Key words: GEST, PECKCRYST, crystal structure, SDPD, powder diffraction

I. INTRODUCTION

Over the past decade, the technology of structure determination from powder diffraction (SDPD) data has been greatly improved, although not completely routine (David and Shankland, 2008). The global optimization methods, i.e., the real-space methods, are widely and successfully used for molecular crystal-structure determination from powder diffraction data (David and Shankland, 2008; Harris *et al.*, 1994, 1998, 2004; Favre-Nicolin and Černý, 2002; Engel *et al.*, 1999; Brodski *et al.*, 2003; Cheung *et al.*, 2005; Hanson *et al.*, 2005). The main reason that these methods have been so successful in the procedure of SDPD is that the known molecular topology of the materials is used in the methods as far as applicable.

The GEST (Feng and Dong, 2007) and the PECKCRYST (Feng *et al.*, 2009) programs were developed for structure determination from powder diffraction data by us using genetic algorithm and particle swarm optimization algorithm, respectively. These two programs have been proved to be effective for molecular crystal-structure determination from powder diffraction data. However, we found that the preparation procedure of the input files for GEST or PECKCRYST is complicated that users may become discouraged and give up their efforts to obtain final solutions. If the input files for GEST or PECKCRYST are not prepared correctly, no solution will be obtained. For GEST or PECKCRYST, there are two main input files that need to be prepared. One is a “control” file containing crystal-structure information, known as molecular topology information and control parameters for algorithms. This file can be readily obtained by the recently developed GUI program for GEST and PECKCRYST. The second input file is the Bragg-reflection intensity file (i.e., *hkl* intensity data). This file can be obtained from the FULLPROF program (Rodriguez-Carvajal, 1990) using the Le Bail method (Le Bail *et al.*, 1988). However, the FULLPROF program is complex and may take time for new users to use it effectively.

In this paper, a new procedure for obtaining Bragg-

reflection intensities rapidly from the FULLPROF Suite is described. The Bragg intensities can then be used by the GEST or the PECKCRYST program for crystal-structure determination of small molecules.

II. METHOD FOR OBTAINING A BRAGG-REFLECTION INTENSITY FILE

Both the GEST and the PECKCRYST programs based on global optimization methods were developed in the past few years for molecular SDPD. The main difference between them is in the global optimization algorithms used by the programs: the genetic algorithm is used in the GEST program and the particle swarm optimization algorithm is used in the PECKCRYST program. Therefore, the main steps to use these two programs are similar. A friendly GUI program (as shown in Figure 1) was recently developed for GEST and PECKCRYST to help users to rapidly generate a control input file. Before using this GUI program to find the potential structure solution, the Bragg-reflection intensity data file should be obtained using the FULLPROF program. FULLPROF can be downloaded from <http://www.ill.eu/sites/fullprof/>. The steps for obtaining Bragg-reflection intensity file quickly from the FULLPROF suite are as follows:

1. Run the “Fullprof_Suite” program, then the “FullProf Suite ToolBar” will be opened.
2. Click on the “WinPlotr” program from the “FullProf Suite ToolBar.”
3. Click on “File->Open Pattern File” in the “WinPlotr” program, then, a “Format of data file” window will be opened. Then, select the data format to be used. It can be confusing for the user to determine which data format should be used. An easy way is to use POWDERX program (Dong, 1999) to change the data format into “GSAS” or “CPI” format which can be read into the WinPlotr program. The POWDERX program can read “*.xrd” format data which can be made manually. The format of .xrd file opened in notepad is shown below:

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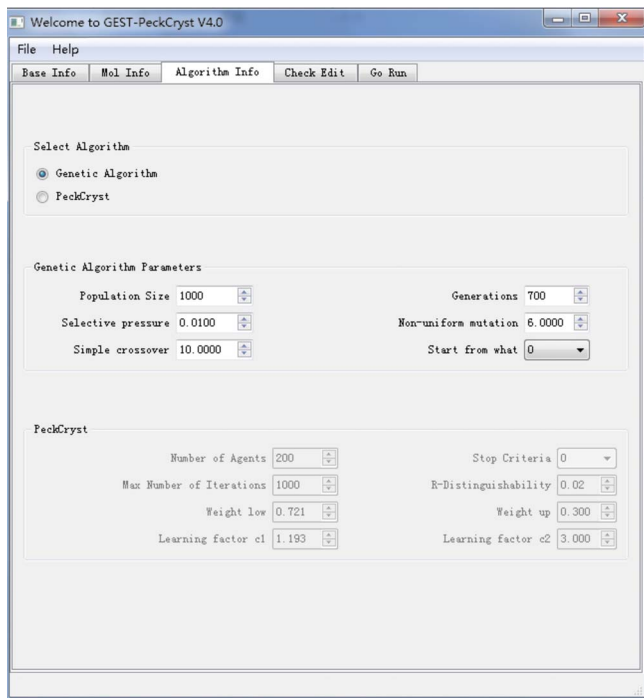


Figure 1. (Color online) The GUI program for GEST and PECKCRYST.

Hydrochlorothiazide

4000
 10.0 50.1
 10.02 52.3
 10.04 60.0

The first line is the name of the sample and the second line is the total number of the experimental points (Npoints), and it can be calculated according to the following formula:

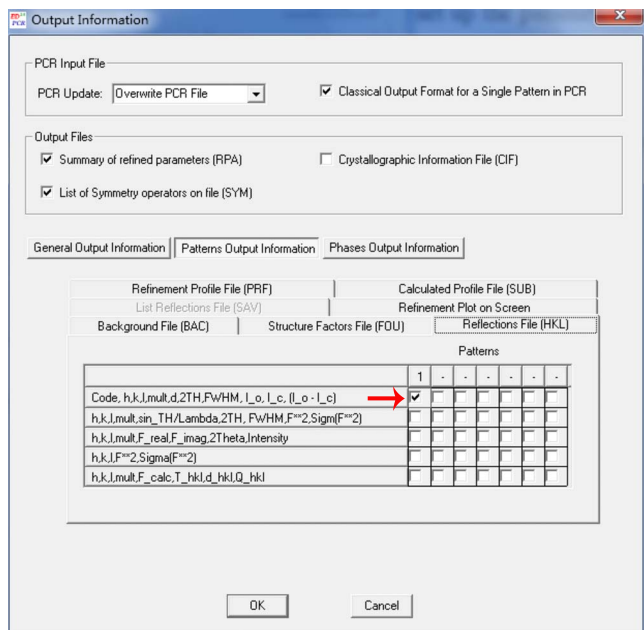


Figure 2. (Color online) The output setting, the check box pointed by an arrow must be checked.

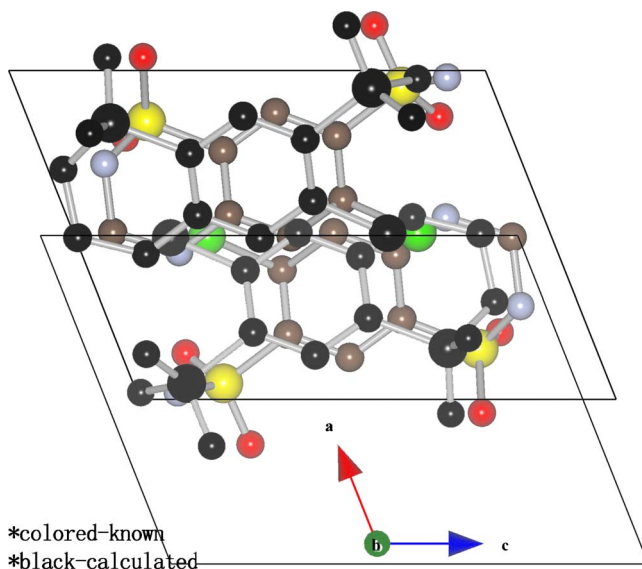


Figure 3. (Color online) Three-dimensional structures of hydrochlorothiazide, $C_7H_8ClN_3O_4S_2$: the calculated powder structure (black) and the single-crystal structure (color). The unit cell of the calculated powder structure is displaced slightly along both the a and the c axes.

$N_{points} = (StopAngle - StartAngle) / step$. Then, the following lines are the experimental points: the first column represents the 2θ and the second column represents the diffraction intensity.

- After the experimental data are read into WinPlotr correctly, click on the menu "Points Selection" -> "Automatic peak search." In this step, the Bragg-reflection peaks are determined for automatic indexing.
- Click on the menu "Points Selection" -> "Save as..." -> "Save as for DICVOL06." Then, a dialog window "Input parameters for DICVOL" is opened. Input the necessary parameters in this dialog and then click "OK." After the powder diffraction pattern is indexed, a file called "sample.pcr" is then generated.
- "* .pcr" file is the control file for the "Fullprof program." Click on the "Edpcr" program in the "Fullprof Suite Tool-Bar" or in the "WinPlotR" tool bar.
- In the "Edpcr" program, open the generated "sample.pcr" file. In EdPcr program, click the corresponding buttons to set up the parameter to do profile fitting using the Le Bail method. The important setting is in the "output" button as shown in Figure 2, and the check box pointed by an arrow should be checked. This setting will let the FULLPROF program generate the correct format Bragg-reflection intensity file for GEST or PECKCRYST.

III. EXAMPLE

The crystal structure of hydrochlorothiazide, $C_7H_8ClN_3O_4S_2$, was determined using the PECKCRYST program and the procedure described above. The powder diffraction data and data-collection parameters were downloaded from the website <http://www.powderdata.info/>. Bragg-reflection intensities were obtained using the FULLPROF program. The crystal-structure results obtained by PECKCRYST show that hydrochlorothiazide is monoclinic, with space group $P2_1$, unit-cell parameters $a=7.389 \text{ \AA}$, b

=8.494 Å, c =9.991 Å, and β =111.717°. The value of the modified Bragg R factor obtained by the PECKCRYST program is 0.1313 (Feng *et al.*, 2009). The VESTA software from Momma and Izumi (2008) was used to draw the three-dimensional crystal structure determined by SPDP and compared to the structure obtained from a single crystal (Dupont and Dideberg, 1972). As shown in Figure 3, the powder crystal structure does match the single-crystal structure.

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