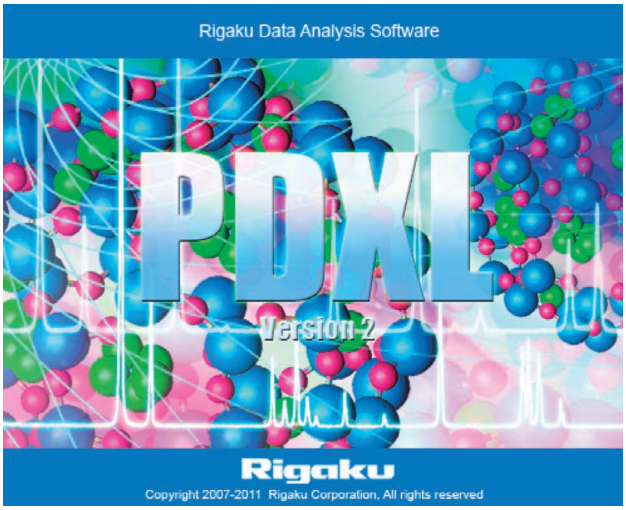
Advanced integrated X-ray powder diffraction suite

PDXL 2



1. Introduction

PDXL is a one-stop powder diffraction analysis software suite[1),2)]. The advanced engine and user-friendly GUI have been satisfying both experienced and novice users since PDXL was released in 2007.

PDXL provides various analysis tools such as automatic phase identification, quantitative analysis, crystallite-size analysis, lattice constants refinement, Rietveld analysis, *ab initio* structure determination, etc.

In May 2011, Rigaku released a new version "PDXL 2" with several excellent new features, which are introduced in the following sections.

2. New features

2.1. Fundamental parameter method

The peak shape in a powder diffraction pattern would appear to be a delta function if measured under ideal conditions. In reality, the peak shape changes depending on a number of measurement conditions: wavelength distribution of the source, optical systems, slit conditions, crystallite size and strain, and so on. The peak shapes obtained from measurements made under real-world conditions are described using an empirical function such as a split pseudo-Voigt function, or a split Pearson VII function which has a good agreement with the obtained peak shapes.

The fundamental parameter method (FP method) is a method to calculate peak shape by convolution of the shapes caused by all the instrumental and sample conditions as shown in Fig. 1.

The advantages of the FP method are as follow:

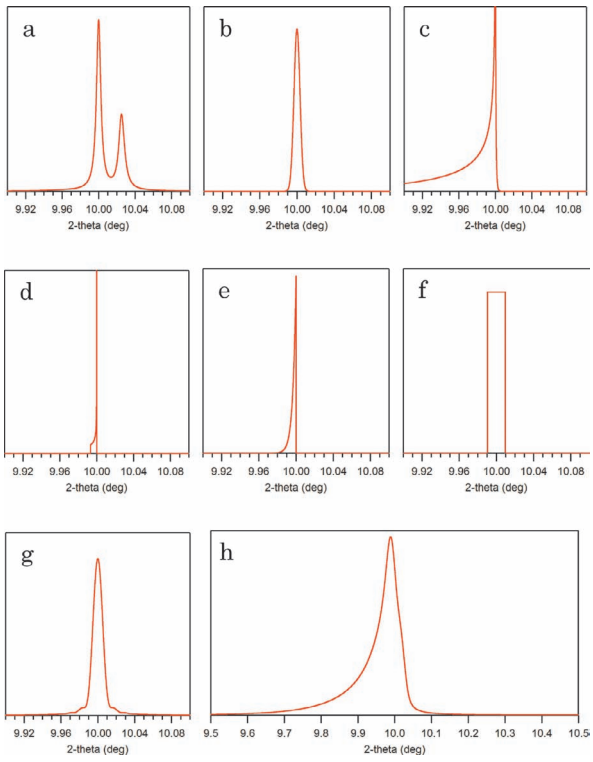


Fig. 1. Some examples of broadening caused by instrument and sample conditions. a: spectroscopic profile of X-ray radiation; b: effect of focus size of X-ray generator; c: axial divergence effect; d: flat-specimen effect; e: sample transparency effect; f: effect of receiving slit size; g: crystallite size and size distribution; h: a resultant profile convoluted the effects a–g.

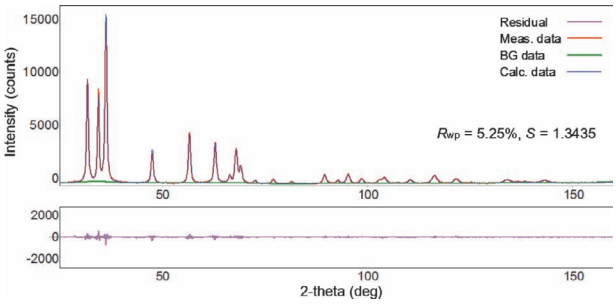


Fig. 2. An example of crystallite size, size distribution and strain analysis of ZnO nano-particles by the Rietveld method. Average crystallite diameters perpendicular and parallel to *c*-axis, normal size distribution and strain determined to be 19.09(13) nm, 22.9(2) nm, 0.589(9) and 0.200(4)%, respectively. Size distribution analyzed as a log-normal distribution function.

1. Correct peak positions can be obtained from a profile where many peaks overlap;
2. In quantitative analysis, more correct quantities can be obtained from a sample consisting of many phases;
3. Sample information, such as crystallite size, size distribution and strain, can be obtained precisely without using any standard reference materials in the measurement as shown in Fig. 2.

2.2. Phase identification using COD

The Crystallography Open Database (COD)[3] is a free, public-domain database of the crystal structures published in International Union of Crystallography, Mineralogical Society of America and so on.

Earlier versions of PDXL could only use paid databases such as ICDD's PDF-2 to perform automatic phase identification. PDXL 2 can incorporate both ICDD/PDF-2 and COD[4], adding the COD library of over 150,000 crystal structures to PDXL 2's already substantial capabilities.

2.3. Wizard for *ab initio* crystal structure analysis

Recently, there have been many published examples of *ab initio* crystal structure analysis performed on powder diffraction data. This development is attributed primarily to significant improvements in PC processing speed and in the efficiency of the algorithms used for structure determination.

PDXL has so far provided all of the functions required for *ab initio* crystal structure analysis, such as indexing (ITO, DICVOL and N-TREOR programs), structure determination (direct methods of EXPO2009, direct-space methods of EXPO2009 or PDXL, and charge flipping method) and structure refinement by the Rietveld method.

Now the "Structure Analysis Wizard" is available in PDXL 2 to provide support and guidance for users undertaking the complicated procedure of structure analysis, particularly of organic compounds. This wizard system will make it possible for even the beginner to achieve analytical success (Fig. 3).

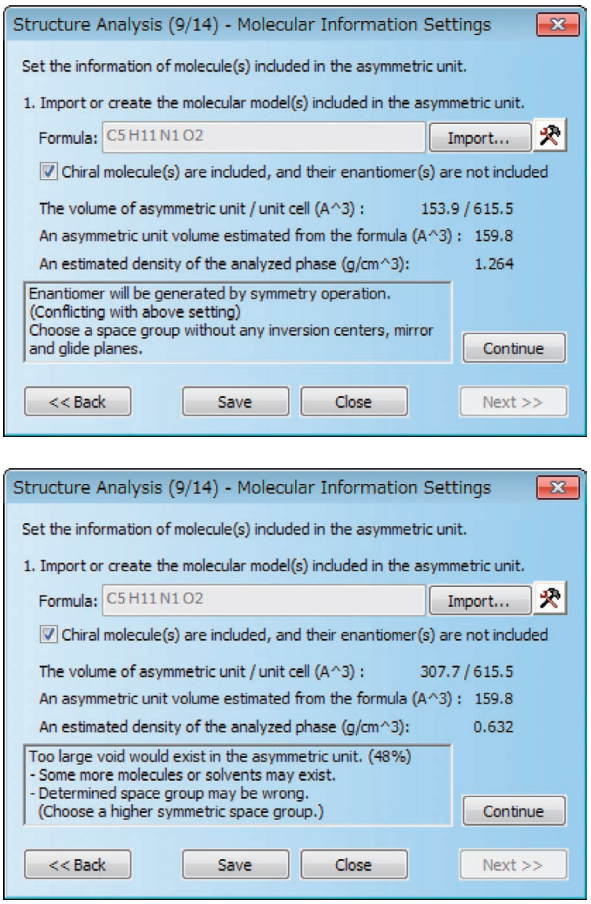


Fig. 3. Structure analysis wizard used to solve *L*-valine, which has space group $P2_1$ and with a Z', the number of molecules in the asymmetric unit, of 2. The top dialogue box indicates that the chosen space group of $P2_1/m$ is wrong. The bottom suggests that the volume of the set molecule (1 *L*-valine molecule) occupies only half the volume of the estimated asymmetric unit.

2.4. Clustering function

The PDXL clustering feature can group multiple scan data based on the similarity of powder diffraction patterns and peak positions, and displays the grouped data in an easy-to-read tree. This is particularly effective when it comes to classifying and screening the data from a large number of scans.

References

(1) *Rigaku Journal (Japanese version)*, **40** (2009), No.1, 36–40.
(2) *The Rigaku Journal (English version)*, **26** (2010), No. 1, 23–27.
(3) S. Grazulis, et al. *J. Appl. Cryst*, **42** (2009), 726–729.
(4) The database for PDXL can be downloaded from the COD homepage (http://www.crystallography.net/).