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X-ray Powder Diffraction Research

EXPO Software: Advances Tools for Structural Analysis of Crystalline Materials by X-ray Powder Diffraction

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Background and State of the Art

In recent years, the number of crystal structures solved from powder X-ray diffraction (PXRD) data has been increasing, as proved by the number of crystallographic information files (CIFs) deposited in the various crystal structure databases. Even though still not comparable to the single-crystal case, structure solution of microcrystalline powders has become widespread, especially in the case of small-size molecules. This success is well supported by the availability of software, among which we mention DASH,^[1] FOX,^[2] SUPERFLIP,^[3] and EXPO,^[4] the software developed by the Institute of Crystallography. This latter is able to solve crystal structures from powder diffraction data by using reciprocal- (Direct Methods) as well as real-space methods (Simulated Annealing). It is able to carry out the full pathway of the solution process: indexing, space group determination, estimation of the integrated intensities, structure solution and Rietveld refinement. Over the past few years, EXPO has been enriched with new powerful computing tools for enhancing the structure solution process and facing challenging cases.^[4] Our research activity is focused on developing advanced crystallographic methodologies that are implemented in EXPO for making the solution process successful and more easily accessible also to not experts in crystallography.

Results^[5,6]

2022 Additional Tools

> Crystal structure solution in case of a mixture of chemical phases





Rietveld Refinement

Capability to solve an UNKNOWN structure from a mixture composed of one UNKNOWN structure and one or more KNOWN structures

Bond Valence Sum (BVS) Tool





This tool provides details
on the oxidation states of
the metal ions in an

•	Label	Туре	x	У	z	Symm.Op.	Distance	Bond Valence
5	01	0	0.7449	-0.0131	0.5730	(x, y, z)	2.510	0.274
5	01	0	0.7551	0.4869	0.9270	(-x+1/2, y+1/2, -z+1/2)+(1,0,1)	2.282	0.508
6	02	0	0.7257	0.1770	1.2368	(x, y, z)+(0,0,1)	2.259	0.541
7	05	0	0.5711	0.1740	0.8702	(x+1/2, -y+1/2, z+1/2)	2.363	0.409
8	06	0	0.9406	0.2950	1.0802	(x, y, z)+(0,0,1)	2.532	0.259
9	03	0	0.6998	-0.1650	0.9334	(-x+1/2, y+1/2, -z+1/2)+(1,-1,1)	2.380	0.390
9	03	0	0.8002	0.3350	0.5666	(x, y, z)	2.585	0.224
10	07	0	0.9298	-0.0191	0.8490	(-x, -y, -z) + (2, -1, 1)	2.309	0.473

EISM Tool for searching in the COD database

This tool enables to import CIF files from the COD database^[7] by allowing the

structure



Crystal Structure Visualization

inorganic compound and serves as an additional support for the accuracy of the corresponding crystal structure.

Conclusions

Innovative computational and graphical tools have been implemented for advancing the crystal structure solution process from powder diffraction data using the EXPO software. These tools enabled to improve the capacity of the software to tackle challenging solution cases and make the structural analysis of microcrystalline materials more user friendly.

GII = 0.119317

Edit bond valence para

References

[1] W. I. F. David, K. Shankland, J. van de Streek, E. Pidcock, W. D. S. Motherwell, J. C. Cole, **2006**, J. Appl. Cryst. 39, 910 - 915. [2] V. Favre-Nicolin, R. Cerny, **2002**, J. Appl. Cryst. 35, 734-743. [3] L. Palatinus, G. Chapuis, **2007**, J. Appl. Cryst. 40, 786-790.) [4] A. Altomare, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, N. Corriero, A. Falcicchio, **2013**, J. Appl. Cryst., 46, 1231-1235. [5] C. Cuocci, N. Corriero, F. Baldassarre, M. Dell'Aera, A. Falcicchio, R. Rizzi, A. Altomare, software, J. Appl. Cryst. **2022**, 55, 411-419. [6] C. Cuocci, N. Corriero, M. Dell'Aera, A. Falcicchio, R. Rizzi, A. Altomare, Software, J. Appl. Cryst. **2022**, 55, 411-419. [6] C. Cuocci, N. Corriero, M. Dell'Aera, A. Falcicchio, R. Rizzi, A. Altomare, Comput. Mater. Sci. **2022**, 210, 111465. [7] S. Gražulis, D. Chateigner, R. T. Downs, A. F. T. Yokochi, M. Quirós, L. Lutterotti, E. Manakova, J. Butkus, P. Moeck, A. Le Bail, J. Appl. Cryst., **2009**, 42, 726-729.

user to provide the chemical formula or the chemical elements contained in the structure model *via* the graphical interface.

The EISM is also a handy tool for graphical visualization of COD entries.

🐞 Add fragme	ent from COD						
	_						
Tex	d:						
Form	iula: C	C8H9NO2					
Elem	ents:						
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COD Number	Spacegroup	Eormula (Cell parame	ters Cell vo	1.17		
1548348	Pcab		.232 11.76 17.16;90				
1552071	P 1 21/c 1	с8 н9 N 02 5	.6893 10.7024 12.40	02;90 95.454 751.61	999		
2006392	P 1 21/n 1	C8 H9 N 02 10	0.795 8.271 17.803;	90 92.957 90 1587.40	000		
2007205	P 1 21/n 1	C8 H9 N O2 7	.0939 9.2625 11.657	90 97.672 90 759.09	002		
2008620	P 1 21/c 1	C8 H9 N O2 0	5.7794 4.8525 9.877	1;90 97.952 9 749.010	001		
2009919	P 1 21/n 1	C8 H9 N O2 6	.664 16.83 7.153;90	107.898 90 763.40	002		
2013900	P 1 21/c 1	с8 н9 N 02 ⁸ 0	.5969 5.6053 15.539	7;90 96.172 9 744.48	999		
0014777	- 1		0505 1C 2220 0C 40	· · · · · · · · · · · · · · · · · ·	~~~		
Chemical name	es: Acetaminoph	en; 4-acetaminoph	enol				
Bibliography:		umov; Marina A. Va ic Form of Acetam	asilchenko; Judith A. K. Ho	ward			
bibliography.			, 1998, <mark>5</mark> 4, 653-655.				
	https://doi.or	g/10.1107/S010827	0197018386				
68 records foun	d.						
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