MS24-1-5 The influence of energy filtering on kinematical and dynamical structure refinement from 3D ED data #MS24-1-5

M. Cabaj [1], L. Palatinus [1], I. Andrusenko [2], M. Gemmi [2]
[1]Department of Structure Analysis, Institute of Physics, Czech Academy of Sciences, Prague, Czech Republic - Prague (Czech Republic), [2]Istituto Italiano di Tecnologia, Center for Materials Interfaces, Electron Crystallography, Pontedera, Italy - Pontedera (Italy)

Abstract
X-ray diffraction data is a well-established and reliable approach of solid-state structure solution and refinement. Compared to it, electron diffraction is still not as commonly used due to various reasons, one of them being higher figures of merit indicating poorer accuracy of structure refinement. There are many ways to address this problem, ranging from using more complex theoretical approach for structure refinement, to installing additional parts of equipment, which enable collection of higher quality data.
In this work we investigate the influence of energy-filtering on data refined with kinematical and dynamical approach.
Four sets of data – two filtered and two unfiltered – were collected from two crystals of metal-organic polymer containing Zn(II), water and 4,4'-biphenyldicarboxylic acid. For first crystal, the unfiltered data were collected first and the filtered – second. For the other crystal the order of data collection was reversed. This protocol ensures that the observed differences are not due to the radiation damage.
All datasets were processed with PETS2 [1], and then solved and refined with JANA2020 [2]. We processed all four data set in an identical manner to ensure a reliable and unbiased comparison.
In each case we observed some differences in R factors. For kinematical refinement the R factors were slightly lower for the unfiltered data (less than 1% of difference), with the except of R(all), which was higher for the filtered data. The situation was different for the dynamical approach – all R factors were higher for the unfiltered data, and again – the difference was usually less than 1%.
The obtained results confirm the preliminary hypothesis that the energy-filtering affects the kinematical and dynamical data refinement process in a different way. The change in all R factors is not considerable, but still measurable. Adding an energy-filter negatively affects kinematical refinement, but improves the dynamical one.

	Crystal 1			Crystal 2		
	Filter off	Filter on	Difference (filter off - filter on)	Filter off	Filter on	Difference (filter off - filter on)
Kinematical refinement						
R(obs)	24.94	25.13	-0.19	26.11	25.95	-0.16
wR(obs)	29.00	29.51	-0.51	30.02	28.91	-1.11
R(all)	32.47	30.78	1.69	29.35	31.46	2.11
wR(all)	29.91	30.35	-0.44	30.37	29.36	-1.01
Dynamical refinement						
R(obs)	10.27	9.78	0.49	12.46	12.94	0.48
wR(obs)	11.73	11.12	0.61	14.16	15.33	1.17
R(all)	16.71	16.24	0.47	15.35	16.26	0.91
wR(all)	13.34	12.78	0.56	14.82	15.94	1.12

This work was supported by the Czech Science Foundation, project number 21-05926X.

References
[1] L. Palatinus, P. Brázda, M. Jelínek, J. Hrdá, G. Steciuk and M. Klementová, "Specifics of the data processing of precession electron diffraction tomography data and their implementation in the program PETS2.0," Acta Cryst. B, vol. B75, pp. 512-522, 2019.
[2] V. Petricek, M. Dusek and L. Palatinus, "Crystallographic Computing System JANA2006: General features," Z. Kristallogr., vol. 229, no. 5, pp. 345-352, 2014.