KN-16 New methods for structural investigations of nanocrystalline and amorphous organic compounds

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Most methods for structure determination from powder diffraction data and Rietveld refinement only work, if the peaks of the simulated powder pattern overlap with those of the experimental diffractogram, i.e. if the lattice parameters of the structural model are very close to the correct ones. We developed a method for a FIt with DEviating Lattice parameters (FIDEL) [1]. The algorithm uses cross-correlation functions instead of a point-to-point comparison of the simulated and experimental diagrams. It can be used, e.g., as a pre-fit for a Rietveld refinement. The FIDEL method is also useful to fit, e.g., low-temperature crystal structures to a room-temperature powder diagram. Crystal structures can be solved from a non-indexed powder diagram by a crystal structure prediction with force fields, followed by FIDEL fit with subsequent automated Rietveld refinement. Structure determination from unindexed powder data is even possible directly with a FIDEL fit, starting from a large set of random crystal structures in various space groups with random values for lattice parameters, molecular position and orientation [1].

Local structures of crystalline, nanocrystalline and amorphous organic compounds can be investigated by analysis of the pair-distribution function (PDF) [2]. The pair-distribution function gives the probability of finding pairs of atoms separated by a distance r. Experimentally, the PDF is obtained by Fourier transformation of the intensities of a carefully measured powder diffractogram. Also electron powder diffraction data can be used [3,4]. The PDF is used to investigate local arrangements of neighbouring molecules, ordering lengths etc [5]. Examples will be shown. Recent software developments allow the fit of a crystal structural model to the PDF curve [6]. It is also possible to solve crystal structures from powder data by a PDF fit. The structure of allopurinol could even be successfully solved in P1 with four symmetrically independent molecules [6].

[1] S. Habermehl et al., Acta Cryst. B70 (2014), 347-359.

[2] D. Prill, P. Juhás, M.U. Schmidt, S.J.L. Billinge, J. Appl. Cryst. 48 (2015), 171-178.

[3] T.E. Gorelik, M.U. Schmidt, U. Kolb, S.J.L. Billinge, Microsc. Microanal. 21 (2015), 459-471.

[4] S. Billinge, C. Farrow, M. Kanatzidis, T.E. Gorelik, M.U. Schmidt, US-Patent 8921783 B2 (2014).

[5] M.U. Schmidt et al., Acta Cryst. B65 (2009), 189-199.

[6] D. Prill, P. Juhás, S.J.L. Billinge, M.U. Schmidt, Acta Cryst. A72 (2016), 62-72.

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MS1 SAXS in structural biology

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MS1-O1 Disentangling Structural Heterogeneity in Highly Disordered Biomolecular Complexes

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During the last decade multiple bioinformatics analysis have revealed that many key proteins involved in signalling and cell control contain large disordered regions and, in some cases, are fully unstructured. The existence of proteins that despite not having permanent structural elements are fully functional have required the reformulation of the traditional structure/function paradigm, one of the historical bases of biological sciences. The study of the relationship between the dynamic structure of Intrinsically Disordered Proteins (IDPs) and their function represents an enormous challenge due to their inherent conformational plasticity. IDPs do not crystallize, and solution methods provide ensemble averaged structural parameters that must be interpreted in terms of ensembles. These difficulties have been partially overcome by applying integrative structural biology approaches where multiple complementary structural data are integrated to derive ensemble models embedding the structure and dynamics of IDPs or their complexes with other biomolecules. In this presentation the study of disordered biomolecular complexes involved in regulation of gene transcription and DNA processivity will be presented. These studies integrate SAXS and NMR data with models built based on partial crystallographic structures. Developments aiming at disentangling conformational and species heterogeneity, which are very common in this family of biomolecular complexes, will be explained. Derived models provide the structural bases of their biological function.

Keywords: SAXS, NMR, biomolecular complexes, polydispersity