[MS17-05] Using FOCUS and Superflip to solve structures from 3D electron and powder diffraction data.

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The program FOCUS was originally developed to solve zeolite structures from X-ray powder diffraction (XPD) data [1]. It uses zeolite-specific chemical information (3-dimensional 4-connected framework structure with known bond distances and angles) to supplement the diffraction data. In this way, it is possible to compensate, at least in part, for the ambiguity of the reflection intensities resulting from reflection overlap, and the program has proven to be quite successful. Recently, advances in electron microscopy have led to the development of automated diffraction tomography (ADT)[2] and rotation electron diffraction (RED)[3] techniques for collecting 3-dimensional electron diffraction (ED) data on very small crystallites. Reasoning that such data are also less than ideal (dynamical scattering, low completeness, beam damage) and that this can lead to failure of structure solution by conventional direct methods for very complex zeolite frameworks, FOCUS was modified to accommodate ED data. The modified program was applied successfully to five different data sets (4 ADT and 1 RED) collected on zeolites of different complexities. One of these, IM-5, could not be solved completely by direct methods, but emerged easily in the FOCUS trials [4]. For two of the samples, IM-5 and SSZ-45, synchrotron XPD data were also available. It was thought that by combining the ED and XPD data sets, the deficiencies of each could be compensated by the other, to generate a data set more correct than either one alone. A pragmatic approach was taken by using the ratios of the structure factor amplitudes obtained from the ED data to repartition reflections that overlap in the XPD data. The reflections in the XPD pattern were assigned to overlap groups based on their *d*-spacings and half-widths, and then each overlap group was categorized, based on the number of XPD and ED reflections present in the group. The repartitioning procedure takes into account that ED reflections may be absent. The combined data set was then treated as single-crystal X-ray diffraction data and tried with FOCUS and the more generally applicable charge flipping routine in the program Superflip [5]. With both programs, the combined data set was found to improve the results significantly. With FOCUS, using the same input file as for the tests with ED data alone, the selectivity and number of solutions generated by the algorithm could be increased in both cases. In other words, structure solution using the combined data was substantially better than using XPD or ED data individually. The structures of both zeolites could also be recovered using charge flipping. For SSZ-45, up to 50% of the electron density maps generated by Superflip were fully interpretable and nearly all framework atoms could be identified, whereas structure solution from ED data alone yielded only partial solutions. In the case of IM-5, up to 80% of the framework atoms can be identified in the better density maps. When several maps are merged, the complete framework structure can be recovered. A reliable procedure for determining a common origin for the individual maps in order to combine them effectively is currently under investigation.

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