Poster

Too small for XRD but perfectly sized for ED

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Whenever it is possible, for any small-molecule crystalline specimen scientist by default prefer to use the X-ray diffraction experiments for combined substance identification and structure analysis as it is a much cheaper, more convenient and much more established technique than any other diffraction method. However, some substances tend not to form crystals of sufficient size for X-ray diffraction and any attempts to grow larger samples result in polycrystalline clusters with quality not enough for obtaining a reliable model. In such cases electron diffraction might prove very useful. While usually the requirement of very limited crystal size range for electron diffraction technique is considered one of its highly restraining drawbacks, in some cases it is advantageous. Indeed, samples thicker than a few microns tend to absorb all of the primary electron beam and crystals thinner than a small fraction of a micron might not provide any useful diffraction signal at all, but with the perfectly fitting size the electron diffraction studies should provide information inaccessible for X-ray diffraction method.

At the main part, we would like to show a few cases where small-molecule compounds grew as tiny single-crystals with length of about a micron in each direction that were insufficient for X-ray diffraction studies but were perfectly sized for conducting electron diffraction experiments. In our approach, for each sample we collected multiple diffraction datasets that were then tested for possibility of merging to obtain better completeness. Then the independent atom model with kinematic approach was used with the dynamical effects accounted only by applying the extinction correction. While some of the refinement and data statistics, such as R_1, wR_2, R_{int} or mean I/σ might look much worse than in the typical X-ray diffraction study, the obtained models seem to be sensible. Even the typically occurring problem of similarly looking C-, N- and O-atoms is usually easily resolved with data collected up to a higher resolution.

To elaborate more on the topic, we would like to show some other structures obtained from electron diffraction data with independent atom model and compare them with X-ray diffraction results available in databases. One of the assets of the electron diffraction seems to be identification of hydrogen atoms presence. While typical X-ray data hardly ever identifies the electron density peaks that are sufficiently distinguishable from the background to allocate the hydrogen atoms, the routinely collected electron diffraction data seems to overcome this challenge much more often. What is more, electron diffraction provides quite reliable values of bond lengths between hydrogen and parent atoms as these are usually very consistent with neutron studies.

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