Charge Density: The Devil is in the Details

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Modern sources and detectors have refocused attention on data quality and the reasonable limits of instrumentation. Because area detectors, and especially detectors with large active area have dramatically reduced experiment time, it is possible to look to ways to extract more information from our samples. Many laboratories are beginning to investigate charge density. Charge density studies by X-ray diffraction require special care and attention to details. Charge density measurements demand the most robust instrumentation and most thoughtful experiments. Intensities must be measured with high accuracy and low uncertainty. Data collections must extend to very high resolution to ensure adequate data for all the additional parameters to be fit in the model. The Bruker D8 series of diffractometers are ideally suited to such studies. Able to reach beyond 150° in 2theta, reciprocal space is well-covered to 0.37 Å for molybdenum radiation. The PHOTON II and PHOTON III detectors exhibit high sensitivity, with single-photon counting for the PHOTON III, allowing for superior data at the extreme limit of the diffraction sphere. Other significant features of these detectors include low parallax effects and detection linearity, even at high count rates. In addition, the large active area of the detectors reduces total experiment time and facilitates the collection of extra redundant data.

To explore best practices for charge density studies, crystals of alanine and other small molecules were studied using a Bruker D8 QUEST diffractometer equipped with a PHOTON II detector and with a PHOTON III detector. These experiments required some time and consideration to acquire the highest quality data. Accurate, carefully measured data were paramount for success. Even absence of a single reflection strongly influenced the outcome. After learning the path to best data, the methodology and results will be presented so that users can be confident to pursue these experiments in their own labs.