

X-ray Diffraction (XRD) has been used for over a century to describe the nature of crystal structures. In the early twentieth century upon the discovery of X-radiation by Röntgen it was shown by Laue that this radiation's atomic-scale wavelengths would cause it to diffract around the atoms and molecules in a crystal structure and through this process it is uniquely suited to measure the spacings and angles between these periodically organized molecules. From Laue's discovery to this day, when a new mineral is discovered, one of its researchers' goals is to use XRD to fully characterize the mineral by describing the internal arrangement of its atoms. Once a mineral structure is known, this method can be used to determine the effects of different changes to the crystal's structure such as those caused by different pressure and temperature regimes or subtle changes in its chemistry due to solid solution. To that effect, Smithsonite, ZnCO_3 , from Tsumeb mine, Tsumeb, Namibia shows varying levels of solid solution of Cu, Fe, Cd, Mn, and Co, for Zn and XRD was used to determine the effect of solid solution on the smithsonite crystal structure. In this experiment XRD was used to determine the effects of solid solution specifically between Zn and Cu^{2+} and Zn and Fe^{2+} in two Tsumeb smithsonite specimens, Cu-rich specimen, TS51 and Fe-rich specimen TS46. Preliminary results show that the unit cell dimensions for TS51 are $a = 4.665(3) \text{ \AA}$, $b = 4.665(3) \text{ \AA}$, and $c = 15.079(17) \text{ \AA}$, and the total cell volume is $284.2(5) \text{ \AA}^3$. TS46 is slightly smaller with unit cell dimensions of $a = 4.6532(4) \text{ \AA}$, $b = 4.6532(4) \text{ \AA}$, and $c = 15.047(4) \text{ \AA}$, and the total cell volume of $282.15(19) \text{ \AA}^3$. TS51 was problematic - many diffraction spots were indistinct. Natural mineral crystals are not typically anywhere close to perfect, and this causes challenges during structure solution. Minerals with many defects, twinning, and inclusions, for example, cause issues when solving a structure. This experiment will be used as an example to show the single crystal XRD process from start to finish and illustrate the capabilities of the method and the difficulties that can arise during this process.

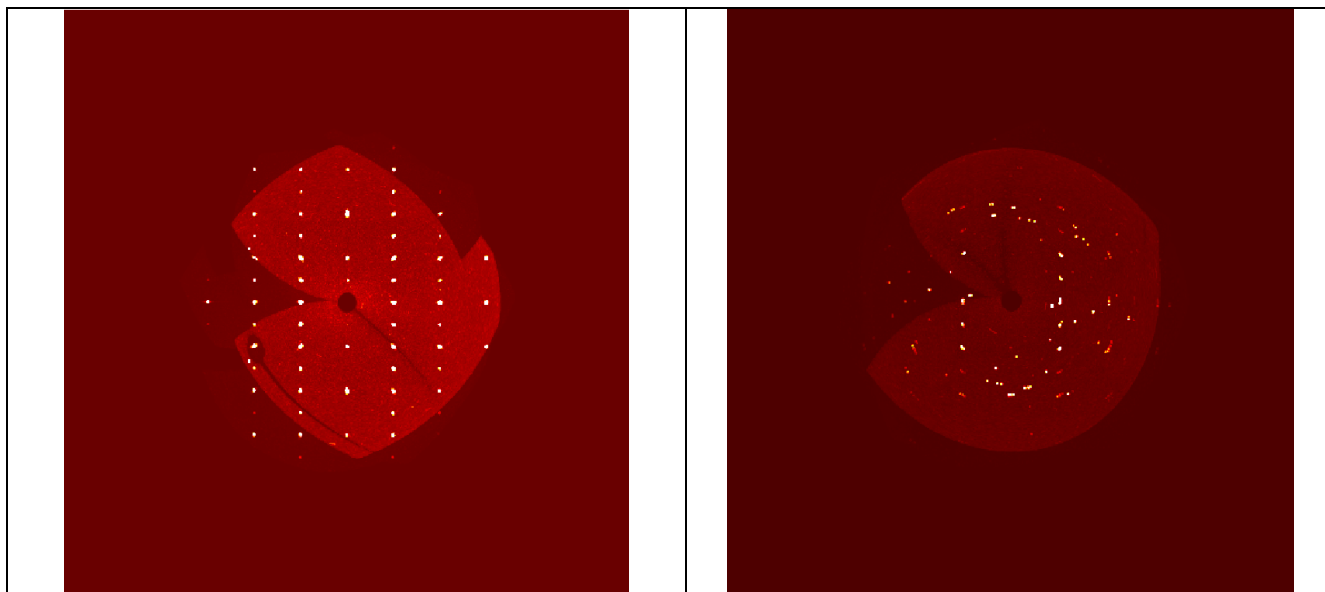


Figure 1: Precession image of specimen TS46 (left) compared to precession image of TS51 (right) showing the difference in quality between the two crystals. The diffraction spots for TS46 are clear individual spots while the diffraction spots for TS51 are smeared.



Figure 2: Specimen TS46, Fe-rich smithsonite courtesy of Malcolm Southwood.