Structure determination from powder x-ray diffraction by direct method on $[Ni^{II}(H_2O)_2\ (pytz)_2]$

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Abstract

Geometric structure in atomic level is very important to understand the chemical and physical properties of a novel compound or phase transition in modern science. Traditionally, single crystal X-ray diffraction is the best way to obtain such three dimensional structure in atomic level. However, if no good quality of single crystal can be obtained, *ab initio* structure determination from powder X-ray diffraction (SDPD) is another way to obtain the crystal structure. In general, such SDPD can be performed in real space or reciprocal space. If the SDPD is carried out in reciprocal space, it needs high space and high angular resolution powder x-ray diffraction (PXRD) data. In this report, a molecular crystal structure $[Ni^{II}(H_2O)_2(pytz)_2]$ was determined in reciprocal space by direct method based on the high resolution data collected at TPS09A. In order to figure out the best data collection condition for direct method, both 12KeV (λ=0.82656Å) and 15KeV (λ=1. 03321Å) PXRD data were collected at room temperature (RT) and 100K. The results indicate that all data can be indexed to obtain the correct cell constants and space group by N-TREOR program in EXPO2014. However, the 15KeV data at RT cannot be solved by direct method. The best condition is 12KeV data collected at 100K. The obtained model was further refined by GSAS program. An interesting result is that using anisotropic displacement of thermal vibration model at Ni atom can significantly reduce the electron residual density from [-2.426, 1.056] (e/Å^3) to [-0.631, 0.553] (e/Å^3). In addition, the magnetic property and electronic structure of this compound will be also discussed in terms of Fe L_{II-III} –edge X-ray absorption spectroscopy (XAS) and density functional theory (DFT) calculation.

Keywords –XRD, SDPD, DFT