

Structure Solution of Thermal Decomposition Compounds using Laboratory X-rays

Thierry Bataille, *Laboratoire de Chimie du Solide et Inorganique Moléculaire (UMR 6511 CNRS), Institut de Chimie, Université de Rennes 1, Rennes, France.* E-mail: thierry.bataille@univ-rennes1.fr

Powder X-ray diffraction at non ambient conditions is developing tremendously, thanks to the rapid progresses in diffraction techniques, methods and software. The present study focuses on the structure solution of inorganic powdered compounds resulting from thermal transformations, using the Bragg-Brentano optics with a conventional X-ray source. Some features related to *in situ* powder data collection are discussed. They include the sample surface displacement, which generates errors on peak positions for pattern indexing, the thermal stability of the products upon heating and the problem of line overlap. Indeed, the latter may arise from diffraction line broadening generated by the crystallite fragmentation during the thermal transformation. This is a major limiting factor for solving the crystal structure, since it strongly affects the structure solution with the direct methods and global optimisation approaches. The influence of the microstructure on the structure solution of the decomposition compound $\gamma\text{-Zn}_2\text{P}_2\text{O}_7$ is illustrated by a study from simulated patterns.

Representative examples of *ab initio* structure determination of thermal decomposition products will be described, such as those obtained by dehydration reactions of open-framework oxalate and phenylphosphonate materials, and by degradation of nitrate and squarate compounds.

Keywords: structure determination, thermal decomposition, powder diffraction