

## Unraveling unforeseen disorders in silicates with 3D electron diffraction

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Zeolites are often synthesized as small polycrystalline powders that make their structure determination by single crystal X-ray diffraction challenging. 3D electron diffraction (3D ED) methods, especially continuous rotation electron diffraction (cRED), overcome the size limitation and can reveal structures of sub-micrometer sized crystals with similar resolution as single crystal X-ray diffraction. In this work, we have utilized cRED to reveal unprecedented disordered chains that link together the *non* cages [4<sup>1</sup>5<sup>8</sup>] in nonasil (NON) to form its complete 3D framework. The refinement against the cRED data in the reported *Fmmm* space group revealed residual peaks in the electrostatic potential maps that clearly indicate two configurations of the zig-zag chains that link the *non* cages together. These atoms reside on a mirror plane perpendicular to the c-axis. Another mirror plane that is perpendicular to the b-axis prevents them from relaxing into either configuration, and replacing it with a two-fold rotation axis allows full relaxation. Hence, the structure is best described by superposition of two *Fm2m* models, or two 50% occupant chain configurations in *Fmmm*. Herein, with cRED and computational aid, we uncover the origin of the disorder and demonstrate that the same disorder is prevalent in all *non*-cage containing zeolite structures CIT-13 (\*CTH), ERS-18 (EEI), EMM-25, EU-1 (EUO), ITQ-27 (IWV), and NU-87 (NES), except for ITQ-32 (IHW) and IM-12 (UTL).

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