



Synchrotron powder X-ray diffraction study of the structure and dehydration behavior of palygorski te

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Rietveld refinements using synchrotron powder X-ray diffraction data were used to study the crystal structure and dehydration behavior of pure monoclinic palygorski te samples from Korea and Alaska. The 300 and 100 K palygorski te structures in air compare well with previous models but provide additional details about zeolitic H₂O sites and reveal that the Al atoms are ordered into the inner M2 octahedral sites and the Mg cations into the M3 sites at the edges of the tunnels. Real-time, temperature-resolved synchrotron powder X-ray diffraction data and Rietveld refinements were used to investigate the monoclinic palygorski te structure from 300 to 1400 K (in air). Rietveld refinements showed that most of the zeolitic H₂O is lost by ~425 K, accompanied by a decrease in the unit-cell volume of 1.3%, primarily owing to a decrease in the *a* unit-cell parameter and an increase in the β angle. The structurally bound H₂O is lost in two stages, at temperature intervals of 475–540 and 580–725 K. Above ~825 K in air a portion of the Korean sample transformed to a folded structure; the Alaskan sample folded at ~575 K under vacuum. A structure model was refined for the folded structure. At ~1015 K for the sample heated in air, β -quartz diffraction peaks appeared and increased in intensity as heating continued to the maximum temperature. Cristobalite formed above ~1050 K, along with a small amount of clinoenstatite, and both phases persisted to the maximum temperature studied.

Key Words: Palygorski te • Rietveld • synchrotron • X-ray diffraction • time-resolved

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