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Dehydration and rehydration process in boggsite: An in situ X-ray single-crystal study

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ABSTRACT

The dehydration-rehydration process in boggsite $|K_{0.06}Na_{0.36}Sr_{0.01}Ca_{7.00}Mg_{1.20}Fe_{0.05}(H_2O)_{70}|[Al_{17.52}Si_{78.62}O_{192}]$-**BOG**, a rare natural pentasil zeolite characterized by a 3D channel system of 10- and 12-rings, occurs in space group *Imma* with cell parameters $a = 20.291(1)$, $b = 23.840(1)$, $c = 12.807(1)$ Å, and $V = 6195.2$ Å^3 at 25 °C. Single-crystal X-ray data collections were carried out at room temperature, at 150, 350, and 500 °C in a hot nitrogen stream, and after the crystal was cooled down to 150 °C and then again at room temperature. During these processes, the variation in the unit-cell volume was always less than 1.4%. At room conditions boggsite is characterized by strong disorder in cation and H_2O molecule distribution, with many partially occupied sites weakly interacting with the framework. At 150 °C, where most of the water is lost, three extraframework cation sites were located. At 350 °C boggsite is fully dehydrated and five cation sites are present; three of these are fourfold coordinated and the others are sixfold coordinated. As a consequence of the migration of the cations, at 500 °C only four cation sites are present. The rehydration process causes distortion of the boggsite channels; the 10-ring reduces its free area by about 1 Å^2, whereas the area of the 12-ring channel grows from 40 to 42 Å^2. The dehydration process is rapid and completely reversible.