

LETTER

Structure determination of the 2.5 hydrate MgSO_4 phase by simulated annealing

HONGWEI MA,¹ DAVID L. BISH,^{1,*} HSIU-WEN WANG,¹ AND STEVE J. CHIPERA²

¹Department of Geological Sciences, Indiana University, 1001 East 10th Street, Bloomington, Indiana 47405, U.S.A.

²Chesapeake Energy Corporation, 6100 N. Western Avenue, Oklahoma City, Oklahoma 73118, U.S.A.

ABSTRACT

The crystal structure of the 2.5 hydrate MgSO_4 phase was determined by simulated annealing from laboratory X-ray powder diffraction data measured from 2–140 °2 θ using $\text{CuK}\alpha$ radiation. The 2.5 hydrate is monoclinic, space group $C2/c$, with unit-cell parameters $a = 18.8636(4)$ Å, $b = 12.3391(2)$ Å, $c = 8.9957(2)$ Å, $\beta = 94.568(2)^\circ$, $V = 2087.1(6)$ Å³, and $Z = 16$. The model was refined using fundamental-parameters Rietveld refinement, converging to $R_{\text{wp}} = 8.89\%$, $R_p = 6.61\%$, $R_{\text{exp}} = 3.33\%$, $R_{\text{Bragg}} = 3.95\%$, and $\chi^2 = 2.67$. The refined structure is consistent with a formula of 2.5 H_2O . Bond-valence calculations for the refined model show that the structure is chemically sensible. In the refined structure, $[\text{Mg}(\text{O},\text{H}_2\text{O})_6]$ octahedra and $[\text{SO}_4]$ tetrahedra build up 2-D double-sheet slabs by sharing vertex O atoms, which are held together by inter-slab H-bonds involving $(\text{SO}_4)^{2-}$ groups and H_2O molecules coordinated with Mg^{2+} cations to form the layer structure of the 2.5 hydrate phase.

Keywords: $\text{MgSO}_4 \cdot 2.5\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 2.4\text{H}_2\text{O}$, crystal structure, simulated annealing, structure determination, powder diffraction, Rietveld refinement