

## **Location and quantification of hydroxyl in wadsleyite: New insights**

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### **ABSTRACT**

Anhydrous and hydrous wadsleyite were synthesized at 13.3–13.5 GPa and 1150–1200 °C in a multianvil press and investigated by Fourier transform infrared (FTIR) spectroscopy, single-crystal X-ray refinement (SC-XRD), and electron microprobe analyses (EMPA). The FTIR spectra agree with previous data, i.e., the spectra are dominated by a broad band around 3380 cm<sup>-1</sup>, resolvable in three bands 3326 (ν<sub>2</sub>), 3382 (ν<sub>3</sub>), and 3546 (ν<sub>4</sub>) cm<sup>-1</sup> besides some weaker OH-bands around 3600 cm<sup>-1</sup>. We confirm that wadsleyite incorporates water in the wt% range and that the concentration strongly increases with decreasing temperature when using secondary ion mass spectrometry (SIMS) and Raman spectroscopy. The quantifications combined with FTIR spectra led us to develop the first IR calibration for water in wadsleyite, i.e., calculating an ε<sub>i,tot</sub> of 73 000 ± 7000 (L mol<sup>-1</sup><sub>H<sub>2</sub>O</sub> cm<sup>-2</sup>). A SC-XRD determination of hydrous wadsleyite FD0718, bearing 8000 ± 1000 wt ppm H<sub>2</sub>O, certifies the presence of Mg vacancies at the M3 sites as previously suggested. Furthermore, we found maxima in the electron density map close to the O atoms O1 and O3 of an M3 octahedron assuming the anhydrous structure. Based on our new data we suggest that the main protonation in wadsleyite occurs along the O1···O4 (3.1 Å) and O3···O4 (3.05 Å) edges of a vacant M3 octahedron. H-incorporation seems to be random leading to protonation of either two O1, two O3, or one O1 and one O3 of the vacant M3 octahedra. With this assignment, the observed ambient and high-pressure IR pattern can now be explained.

**Keywords:** Wadsleyite, FTIR spectroscopy, Raman spectroscopy, protonation, SIMS, absorption coefficient for water