

---

# Intercalation of Halloysite: A Raman Spectroscopic Study

Ray L. Frost<sup>1</sup> and Janos Kristof<sup>2</sup>

<sup>1</sup> Centre for Instrumental and Developmental Chemistry, Queensland University of Technology, 2 George Street, GPO Box 2434, Brisbane Queensland 4001, Australia

<sup>2</sup> Department of Analytical Chemistry, University of Veszprem, H8201 Veszprem, PO Box 158, Hungary

**Abstract:** Intercalates from an ordered halloysite with urea and potassium acetate were studied using Raman microscopy. The urea intercalate showed new Raman bands at 3387, 3410, 3497 and 3598  $\text{cm}^{-1}$  which were attributed to the formation of a urea- $\text{Si}_2\text{O}_5$  complex. New Raman bands were observed at 3585 and 3602  $\text{cm}^{-1}$  for the potassium acetate intercalate with concomitant loss of intensity of the bands at 3635, 3655, 3675 and 3696  $\text{cm}^{-1}$ . These new bands were attributed to the hydrogen bonds formed between the acetate and the inner surface hydroxyl groups. Remarkable changes in intensity in the lattice region of the halloysite were observed, the foremost being the reduction of the intensity of the bands at 243, 271 and 336  $\text{cm}^{-1}$ . Pronounced changes in the bands at 913 and 143  $\text{cm}^{-1}$  attributed to the Al-OH librations were also observed.

It is proposed that 2 distinct types of intercalation were present, as exemplified by: 1) urea intercalate, where the intercalating molecule hydrogen bonds to the Si-O of the halloysite layers and 2) potassium acetate intercalate, where the molecule is hydrogen-bonded to the inner surface hydroxyls of the halloysite layer and interacts with the tetrahedral sheet of the next adjacent halloysite layer. The Raman spectra of the intercalated halloysite strongly resembled that of an intercalated kaolinite.

**Key Words:** Halloysite • Intercalation • Kaolinite • Raman • Raman Microprobe • X-ray Diffraction

*Clays and Clay Minerals*; August 1997 v. 45; no. 4; p. 551-563; DOI: [10.1346/CCMN.1997.0450407](https://doi.org/10.1346/CCMN.1997.0450407)

© 1997, The Clay Minerals Society

Clay Minerals Society ([www.clays.org](http://www.clays.org))

---