## Intercalation of Tetraalkylammonium Cations into Smectites and its Application to Internal Surface Area Measurements

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**Abstract:** Monolayer to bilayer (MTB) and bilayer to pseudotrimolecular (BTP) transitions were observed for smectites exchanged with symmetrical tetraalkylammonium cations of increasing sizes:  ${}^{+}NR_{4}$ , where  $R = (CH_{2})_{n}CH_{3}$ , with  $0 \le n \le 7$ . In the case of SWy-1, SHCa-1 and SWa-1, the variation in layer spacing observed from intercalation of tetramethylammonium up to tetraoctylammonium cations showed a behavior characteristic of smectites with homogeneously distributed interlayer cations. In the case of STx-1, the change in interlayer spacing with the increase of the alkyl chain length was characteristic of a very high charge heterogeneity. Higher charge smectites (SAz-1 and SCa-3, CEC > 1.20 mmol/g) were found to have decreasing cation exchange with increasing cation size, resulting in a "leveling off" of the interlayer spacing toward larger cations. The MTB and BTP transitions were used to determine the internal surface area of various smectites. The proposed method was found to be a quicker and simpler alternative to the polar liquid sorption method for this measurement, but was ineffective for high-charge smectites (CEC > 1.20 mmol/g).

**Key Words:** Hectorite • Intercalation • Internal surface area • Montmorillonite • Quaternary ammonium cations • Smectite • Tetraalkylammonium cations

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