

Degradation of Tetraphenylboron at Hydrated Smectite Surfaces Studied by Time Resolved IR and X-ray Absorption Spectroscopies

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Abstract – The surface catalyzed redox reactivity of an organoboron probe molecule (tetraphenylboron or TPB) with fully hydrated clay mineral surfaces is described wherein one of both of two degradation pathways can occur. Attenuated total reflectance Fourier transform infrared (ATR-IR) spectroscopy can quantitatively measure, *in situ*, both acid hydrolytic and oxidative degradation of TPB as controlled by the exchange cation identity and the structural octahedral Fe content of the clay mineral. Oxidation of TPB at smectite surfaces is directly attributable to octahedral Fe(III) in the clay structure. The concomitant reduction of Fe(III) during TPB oxidation can be analytically measured by X-ray absorption near edge structure (XANES) spectroscopy. The resultant distortion of the clay mineral structure during the reduction of structural Fe could be probed directly by extended X-ray absorption fine structure (EXAFS) spectroscopy. The lack of evidence for clay mineral dissolution during reaction with TPB permits defining the clay as a catalyst. The combination of these three *in situ* spectroscopic techniques to measure the reaction in real time, provides powerful insight into the reactivity of clay mineral surfaces.