American Mineralogist, Volume 84, pages 1433–1438, 1999

Solid-state ^{29}Si MAS NMR studies of illite and illite-smectite from shale

SØREN K. LAUSEN,[1,†] HOLGER LINDGREEN,[2] HANS J. JAKOBSEN,[1] AND NIELS C. NIELSEN[1,*,†]

[1]Instrument Centre for Solid-State NMR spectroscopy, Department of Chemistry, University of Aarhus, DK-8000 Aarhus C, Denmark
[2]Clay Mineralogical Laboratory, Geological Survey of Denmark and Greenland, DK-2400 Copenhagen NV, Denmark

ABSTRACT

A new method to extract quantitative information from poorly resolved ^{29}Si magic-angle spinning (MAS) nuclear magnetic resonance (NMR) spectra of natural mixed-layer illite-smectite (I-S) clays is presented. The Si-Al distribution in layered aluminosilicates are used to link the intensities of ^{29}Si resonances from all $Q^3(n\text{Al})$ sites (n = 0, 1, 2, 3) to the tetrahedral layer aluminum substitution by applying Loewenstein's aluminum avoidance principle (no Al-O-Al linkages) extended to ensure a homogeneous distribution of charge. In addition, correlations between ^{29}Si chemical shifts and the Al substitution are established for illite resonances by computer fitting of well-resolved phyllosilicate spectra. Combination of these two constraints led to a general procedure for iterative fitting of ^{29}Si MAS NMR spectra of clay minerals containing high-charge (illite-like) and low-charge (smectite-like) sites. The applicability of the new method is demonstrated for two I-S samples from Cambrian black shale in the Baltic area and two I-S samples from Upper Jurassic oil-source rock in the Central Trough of the North Sea. In combination with data from XRD and chemical analysis, the results from ^{29}Si MAS NMR enables determination of the composition for the entire I-S particles.