Supporting Information

On the Nature of Terminating Hydroxyl Groups and Intercalating Water in Ti$_3$C$_2$T$_x$ MXenes: A Study by $^1$H Solid-State NMR and DFT Calculations

Takeshi Kobayashi,$^a$ Yangyunli Sun,$^b$ Kaitlyn Prenger,$^c$ De-en Jiang,$^b$ Michael Naguib,$^c$ Marek Pruski$^{a,d,*}$

$^a$ Ames Laboratory, U.S. Department of Energy, Ames, IA 50011, United States

$^b$ Department of Chemistry, University of California, Riverside, CA 92521, United States

$^c$ Department of Physics and Engineering Physics, Tulane University, New Orleans, LA 70118, United States

$^d$ Department of Chemistry, Iowa State University, Ames, IA 50011, United States
**Synthesis of MAX phase Ti₃AlC₂**

MAX Ti₃AlC₂ was synthesized similarly to that reported by Huang and Mochalin.¹ Powders of titanium (Alfa Aesar, 99.5%, -325 mesh), aluminum (Alfa Aesar, 99.5%, -325 mesh), and graphite (Alfa Aesar, 99%, APS 7-11μm) were mixed in ratios of 3:1.2:1.88 in a Turbula T2F mixer for 3 h, then furnaced in a tube furnace under continuous flow of argon at 1600 °C for 2 h with a heating rate of 10°C/minute. After cooling, powders were ground to -325 mesh for use in etching.

¹H–¹H SQ/SQ correlation spectra of MXene annealed at 110 °C.

**Figure S1.** 2D ¹H–¹H SQ/SQ spectra of Ti₃C₂Tx MXene annealed at 110 °C. and 200 °C, taken with τ_{mix} = 50 ms, 100 ms, and 150 ms, 256 t₁ points, and Δt₁ = 55.6 μs.
DFT calculations

Figure S2. Computed $^1$H chemical shifts for Ti–OH terminations on AA-stacking MXene.

References