Temperature dependent structure and dynamics in smectite interlayers: $^{23}$Na MAS NMR spectroscopy of Na- Hectorite

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Supporting Information

Table S1. Fitted peak maxima, full-widths at half-height (FWHH), and relative peak intensities of $^{23}$Na MAS NMR spectra of Na- Hectorite collected at the indicated temperatures and relative humidities (R.H.s).

| $T$ ($^\circ$C) | $\delta$ (ppm) | FWHH (ppm) | Relative Intensity/% |
|----------------|----------------|-------------|---------------------|
| 40             | -19.1          | 8.7         | 92.8                |
|                | -28.7          | 5.4         | 7.2                 |
| -60            | -19.4          | 7.9         | 86.2                |
|                | -29.0          | 6.6         | 13.8                |
| -80            | -19.5          | 7.8         | 87.7                |
|                | -29.2          | 6.7         | 12.3                |
| -100           | -19.4          | 7.8         | 86.4                |
|        | -29.3 | 6.9  | 13.6 |
|--------|-------|------|------|
| -120   | -19.3 | 8.2  | 85.8 |
|        | -29.3 | 6.5  | 14.2 |
| **29% R.H.** |      |      |      |
| 40     | -6.4  | 4.0  | 64.6 |
|        | -21.7 | 4.7  | 8.1  |
|        | -26.8 | 13.5 | 27.4 |
| 0      | -6.1  | 4.9  | 63.2 |
|        | -21.9 | 4.8  | 10.1 |
|        | -27.5 | 12.2 | 26.7 |
| -20    | -5.9  | 5.6  | 71.6 |
|        | -21.8 | 5.6  | 11.7 |
|        | -28.3 | 12.6 | 16.7 |
| -40    | -5.9  | 6.6  | 61.8 |
|        | -21.9 | 4.8  | 10.8 |
|        | -27.4 | 12.6 | 27.5 |
| -60    | -5.2  | 8.3  | 72.7 |
|        | -21.6 | 5.2  | 14.1 |
|        | -28.1 | 10.8 | 13.2 |
| -80    | -0.1  | 4.1  | 30.8 |
|        | -6.5  | 4.0  | 14.7 |
|        | -11.4 | 6.5  | 15.5 |
|        | -21.7 | 5.9  | 19.2 |
|        | -28.4 | 12.0 | 19.9 |
| -100   | 1.1   | 7.5  | 38.0 |
|        | -6.7  | 3.5  | 9.5  |
|        | -11.7 | 7.5  | 16.7 |
|        | -21.6 | 5.7  | 21.6 |
|        | -28.3 | 9.7  | 14.2 |
| -120   | 1.1   | 7.6  | 26.2 |
|        | -6.6  | 4.8  | 21.5 |
|        | -11.9 | 8.2  | 9.8  |
|        | -21.8 | 5.7  | 21.3 |
|        | -27.2 | 12.9 | 21.3 |
| **43% R.H.** |      |      |      |
| 40     | -5.3  | 3.4  | 73.0 |
|        | -19.4 | 6.7  | 27.4 |
| 20     | -5.5  | 4.0  | 65.8 |
|        | -19.6 | 6.7  | 34.2 |
| 0      | -4.4  | 2.8  | 26.7 |
|        | -6.5  | 3.7  | 45.3 |
|        | -19.7 | 6.5  | 28.0 |
| -20    | -4.1  | 3.2  | 27.8 |
|        | -6.7  | 4.2  | 44.0 |
|        | -19.8 | 6.4  | 28.2 |
| -40    | -4.0  | 4.0  | 31.8 |
|      | -7.0 | 5.5  | 40.2 |
|------|------|------|------|
| -19.8| 6.2  | 28.0 |
| -4.1 | 4.0  | 8.6  |
| -6.3 | 9.9  | 53.0 |
| -19.9| 6.9  | 38.4 |
|      |      |      |      |
| -80  | -0.1 | 4.1  | 22.3 |
| -5.8 | 5.2  | 22.2 |
| -11.1| 5.9  | 18.0 |
| -20.0| 7.2  | 37.1 |
|      |      |      |      |
| -100 | 0.6  | 5.8  | 25.5 |
| -6.3 | 4.1  | 15.0 |
| -11.6| 6.8  | 22.6 |
| -20.4| 6.7  | 37.0 |
|      |      |      |      |
| -120 | 1.0  | 7.0  | 26.3 |
| -6.3 | 5.5  | 17.3 |
| -12.0| 8.7  | 22.3 |
| -20.6| 6.1  | 33.2 |
| -27.4| 7.0  | 1.0  |

**70% R.H.**

|      |       |       |      |
|------|-------|-------|------|
| 40   | -2.7  | 1.9   | 76.2 |
|      | -21.1 | 6.2   | 23.8 |
| 20   | -2.2  | 2.0   | 83.9 |
|      | -19.8 | 6.5   | 16.1 |
| 0    | -2.2  | 2.4   | 79.8 |
|      | -19.4 | 5.5   | 20.2 |
| -20  | -2.0  | 2.9   | 74.2 |
|      | -19.7 | 6.0   | 25.8 |
| -40  | -1.7  | 3.9   | 74.4 |
|      | -21.1 | 6.2   | 25.6 |
| -60  | -1.2  | 3.8   | 36.2 |
|      | -3.9  | 9.6   | 35.0 |
|      | -20.0 | 6.0   | 28.9 |
| -80  | -0.2  | 2.7   | 42.5 |
|      | -5.5  | 11.4  | 26.1 |
|      | -20.0 | 6.4   | 31.4 |
| -100 | 0.4   | 5.0   | 45.2 |
|      | -6.0  | 6.0   | 32.0 |
|      | -20.1 | 5.2   | 22.8 |
| -120 | 1.1   | 6.6   | 50.8 |
|      | -6.2  | 7.5   | 33.9 |
|      | -20.0 | 5.3   | 15.3 |

**100% R.H.**

|      |       |       |      |
|------|-------|-------|------|
| 40   | -0.9  | 1.1   | 82.9 |
|      | -20.1 | 6.0   | 17.2 |
| 20   | -0.4  | 1.1   | 83.6 |
|      | -19.8 | 6.3   | 16.4 |
Figure S1: A representative example of the fitting of the $^{23}$Na MAS NMR spectra discussed in this paper using the DMfit. This spectrum is for Na- Hectorite exposed to 29% R.H. at -80°C and fit with five Lorentzian components. The blue and red lines are the experimental and fitted spectra, respectively.