Supplementary information of

A new microporous 12-ring zincosilicate THK-2 with many terminal silanols characterized by automated electron diffraction tomography

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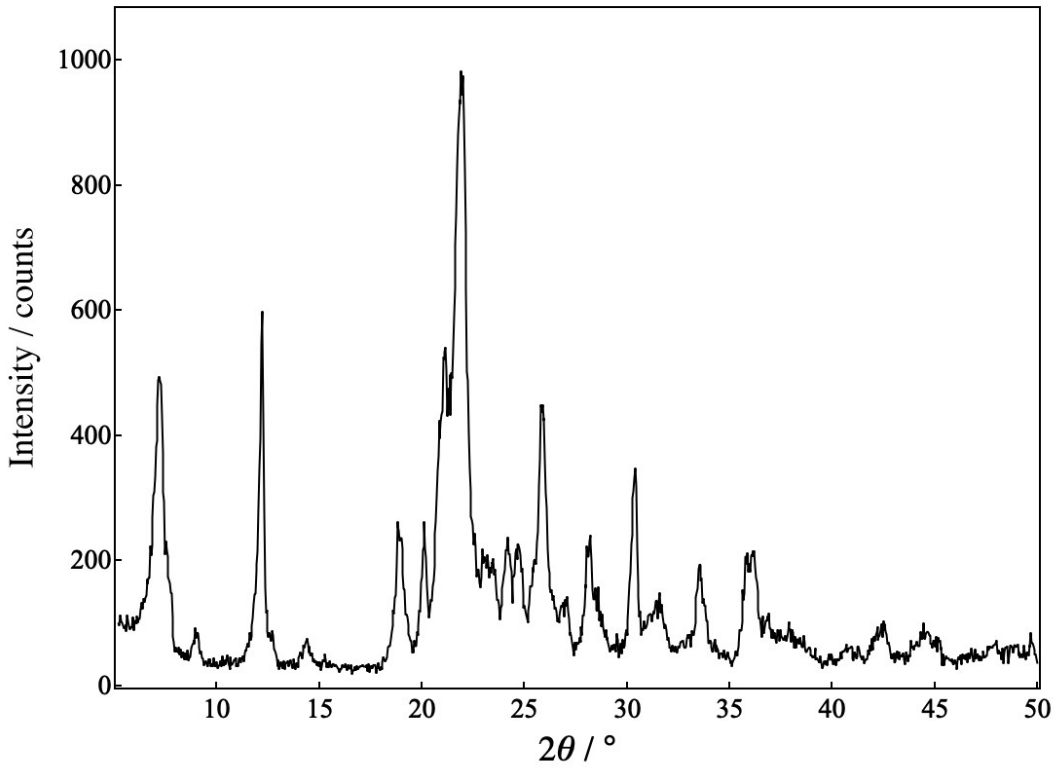

Figure S1. Powder XRD pattern of as-synthesized THK-2 without acetic acid washing.

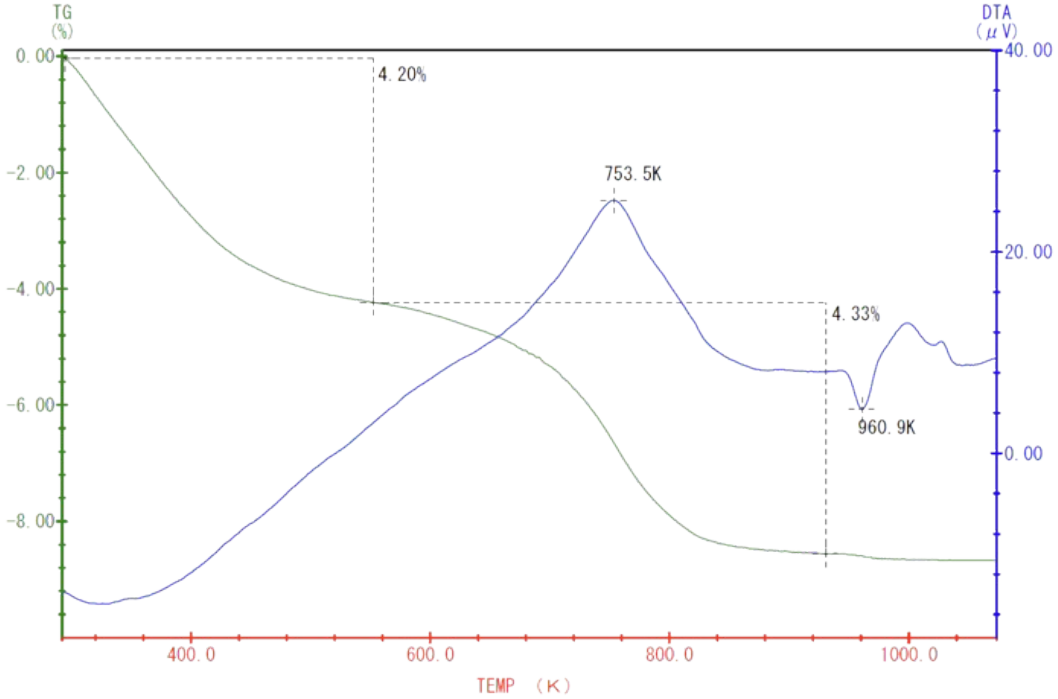

Figure S2. TG-DTA profiles of as-synthesized THK-2 in a temperature range of 292–1073 K.

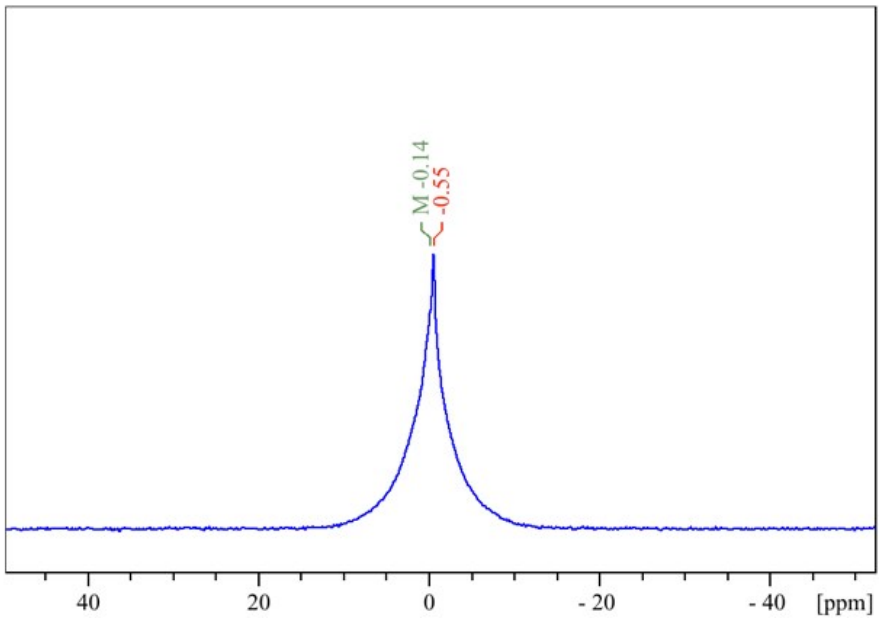

Figure S3. ^{7}Li MAS NMR spectra of as-synthesized THK-2 after HCl treatment (using 1 mM aqueous solution for 1 h at room temperature).

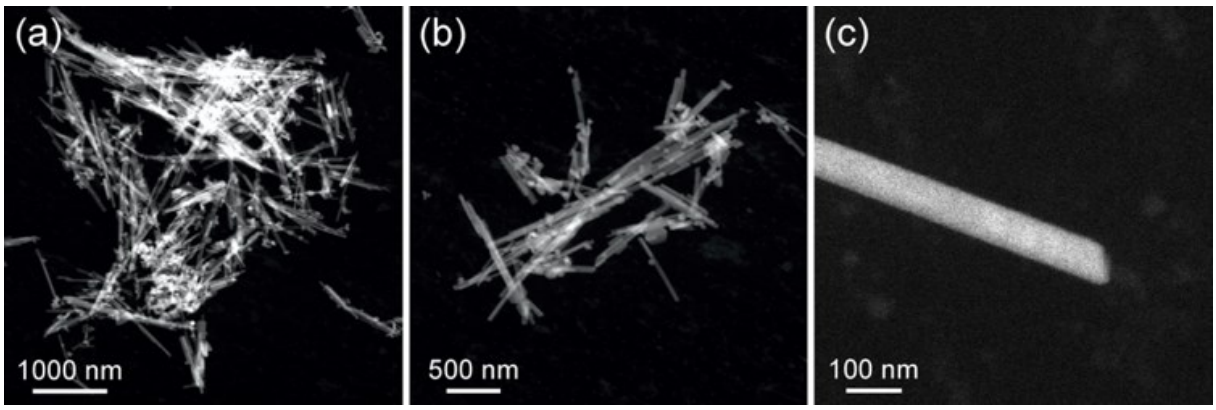

Figure S4. STEM images of cal-THK-2 (overview: a–b; c: single nano crystal).

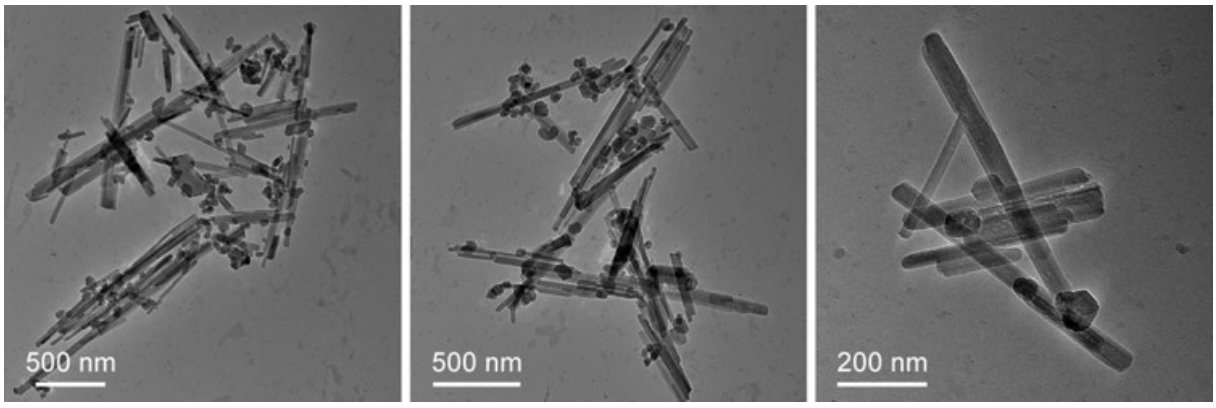

Figure S5. TEM images of cal-THK-2.

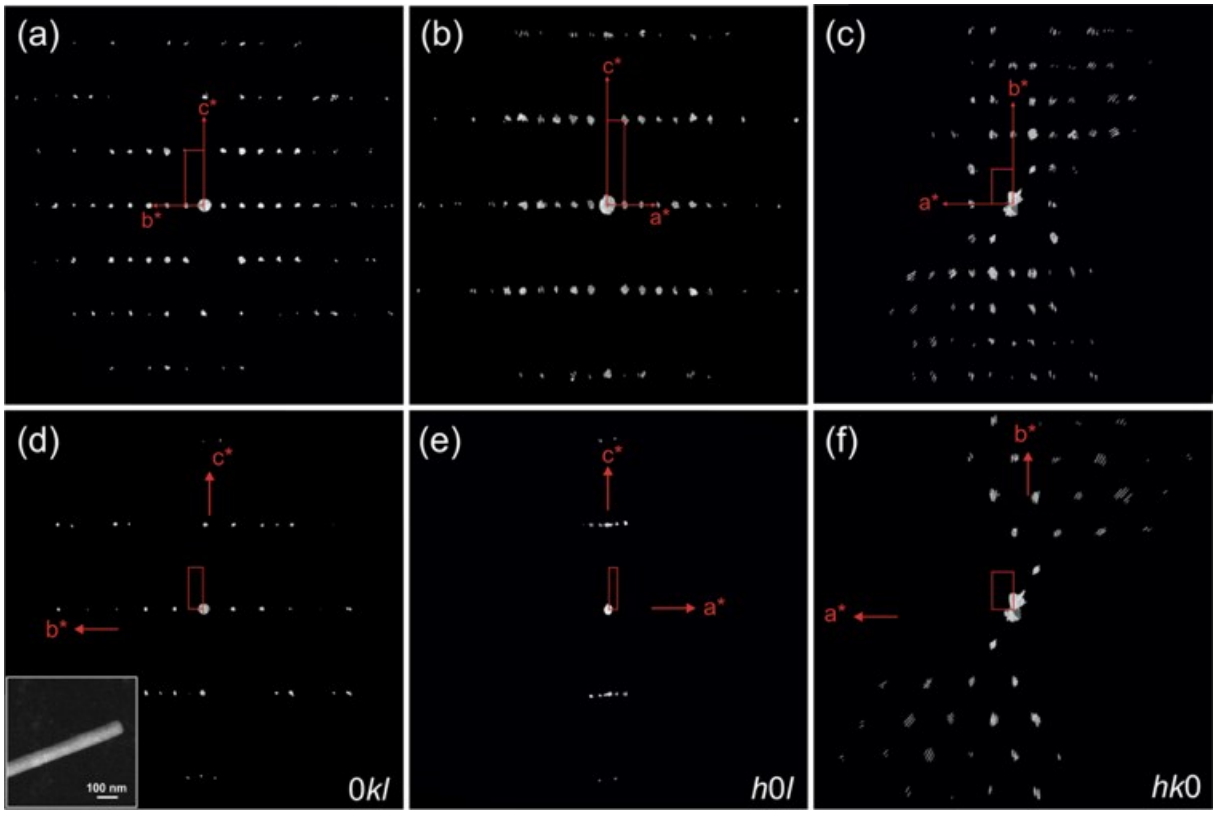

Figure S6. Reconstructed 3D diffraction volumes of cal-THK-2 (Cry2) obtained from ADT data viewed down the three main axis (a–c). 2D slices cut from reconstructed 3D reciprocal space corresponding to 0*kl* (d), *h*0*l* (e) and *hk*0 (f) planes. The crystal selected for the acquisition of ADT data is shown as an inset in (d).

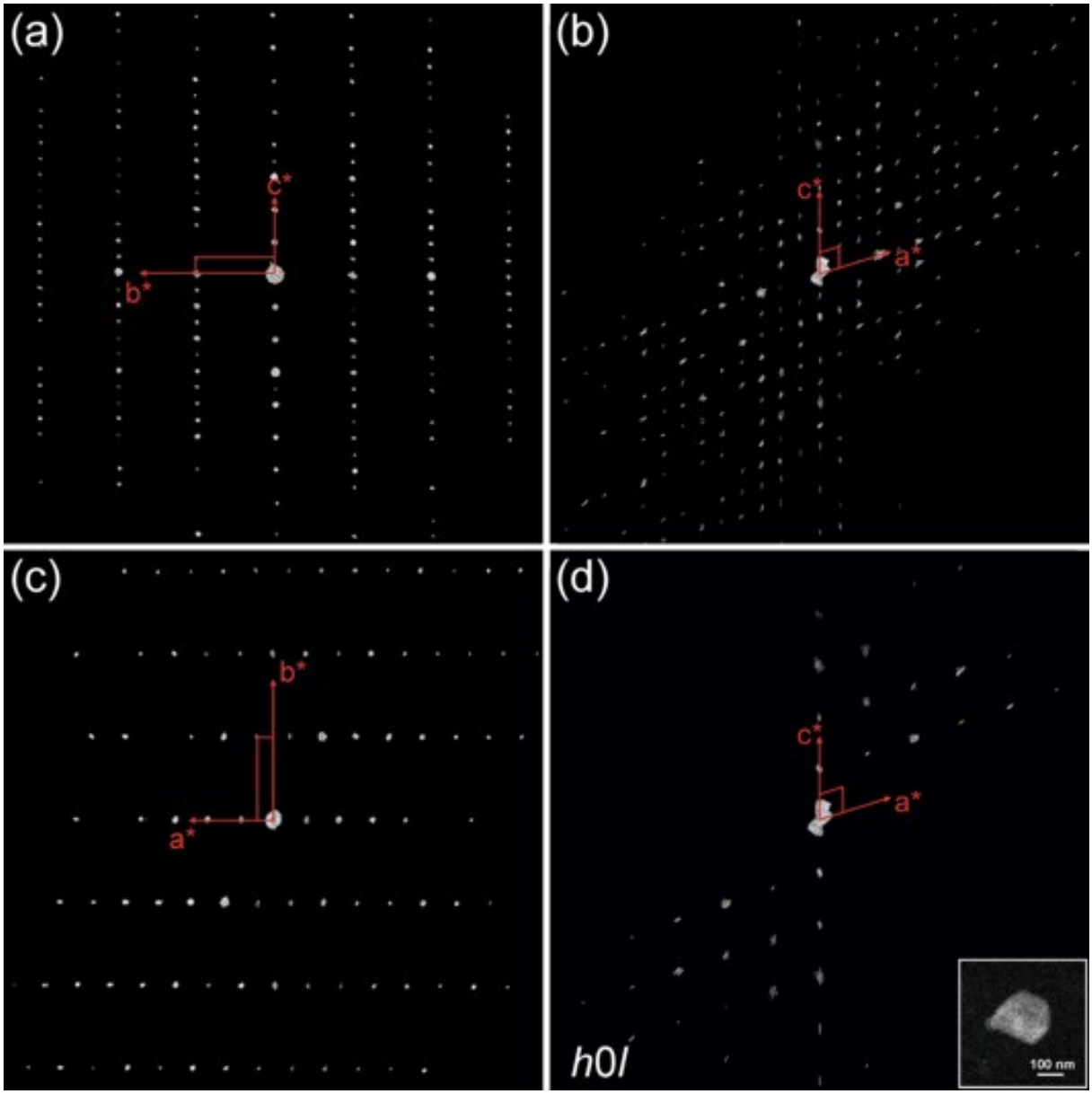

Figure S7 (a–c) Reconstructed 3D diffraction volumes of MTW obtained from ADT data viewed down the three main directions. A *C*-centred unit-cell ($h + l = 2n$ for hkl)) can be observed from the diffraction volume viewed down c^* direction in (c). (d) 2D slice of $h0l$ plane cut from reconstructed 3D reciprocal space. The reflection condition: $h = 2n$ and $l = 2n$ for $h0l$ plane. The selected crystal for the acquisition of ADT data is shown as an inset in (d).

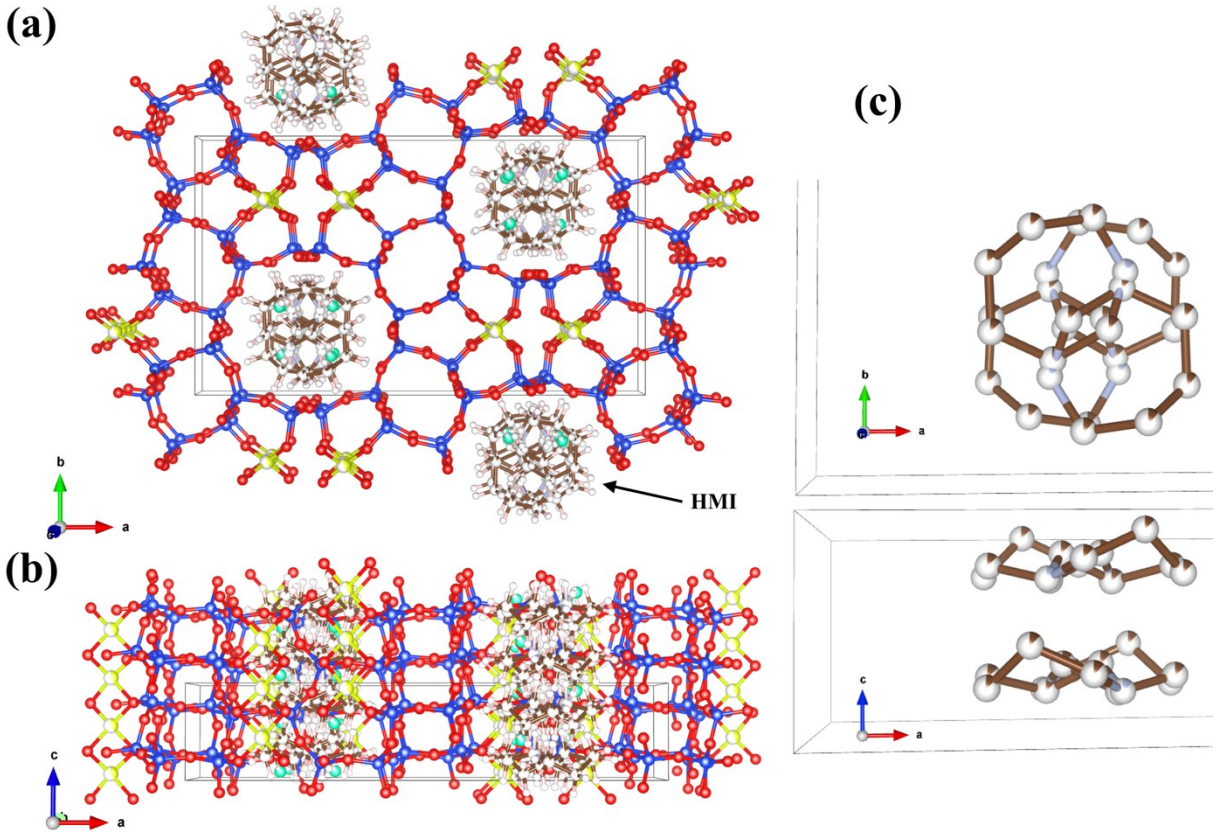

Figure S8. Refined crystal structure model of as-synthesized THK-2 viewed along (a) [001] and (b) [010] directions. Blue, red, yellow and green spheres indicate Si, O, Zn and H2O positions, respectively. (c) four equivalent HMI arrangement in a one 12-ring channel.

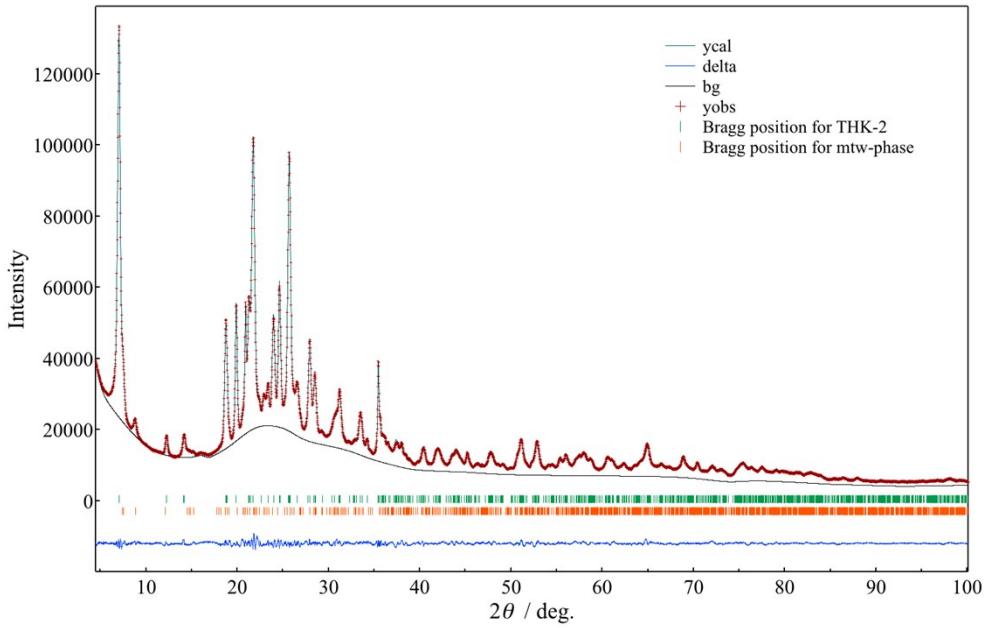

Figure S9. Observed, calculated and difference patterns, and background obtained by the Rietveld refinement for as-synthesized THK-2.

The structural information of cal-THK-2 in CIF format.

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# CRYSTAL DATA
#----------------------------------------------------------------------
data_VESTA_phase_1

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_cell_length_c                      5.0368(1)
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_cell_angle_gamma                   90
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_space_group_IT_number              56

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   'x+1/2, y+1/2, -z'
   '-x, y+1/2, -z+1/2'
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loop_
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   _atom_site_fract_y
   _atom_site_fract_z
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Si2    0.0708(3)   0.6007(9)   0.712(3)   1          8 e  Uiso   0.0262(6)    Si
Si3    0.8968(4)   0.5078(7)   0.788(3)   1          8 e  Uiso   0.0262(6)    Si
Si4    0.7821(4)   0.4179(6)   0.821(3)   1          8 e  Uiso   0.0262(6)    Si
O1     0.9067(7)   0.615(1)    0.727(5)   1          8 e  Uiso   0.035(1)     O
O2     0.7959(6)   0.310(1)    0.797(5)   1          8 e  Uiso   0.035(1)     O
O3     0.8326(8)   0.487(1)    0.772(5)   1          8 e  Uiso   0.035(1)     O
O4     0.0115(9)   0.641(1)    0.737(6)   1          8 e  Uiso   0.035(1)     O
Zn1    0.8366(8)   0.241(2)    0.538(6)   0.2605     8 e  Uiso   0.053(7)     Zn
O5     0.1116(8)   0.686(1)    0.705(5)   1          8 e  Uiso   0.035(1)     O
O6     0.767(1)    0.433(1)    0.124(6)   1          8 e  Uiso   0.035(1)     O
O7     0.915(1)    0.478(2)    0.078(5)   1          8 e  Uiso   0.035(1)     O
O8     0.928(1)    0.451(2)    0.566(5)   1          8 e  Uiso   0.035(1)     O
O9     0.9424(8)   0.764(2)    0.537(8)   1          8 e  Uiso   0.035(1)     O
WO1    0.235(3)    0.197(5)    0.28(1)    0.32(3)    8 e  Uiso   0.03(1)      O
WO2    0.266(5)    0.142(9)    0.28(2)    0.17(3)    8 e  Uiso   0.03(1)      O
WO3    0.445(2)    0.727(3)    0.462(9)   0.44(2)    8 e  Uiso   0.03(1)      O
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*Note: The WO site represents the center of gravity of the adsorbed water. A virtual atom consisting of one O atom and two H atoms was applied for all WO sites.

The structural information of as-synthesized THK-2 in CIF format.

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#======================================================================
# CRYSTAL DATA
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_cell_length_b                      14.3869(4)
_cell_length_c                      5.05369(8)
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   _atom_site_type_symbol
Si1    0.9536(2)   0.6855(3)   0.718(1)   1         8 e  Uiso   0.0175(3)    Si
Si2    0.0723(2)   0.6121(3)   0.692(1)   1         8 e  Uiso   0.0175(3)    Si
Si3    0.8989(1)   0.4974(3)   0.828(1)   1         8 e  Uiso   0.0175(3)    Si
Si4    0.7816(1)   0.4320(3)   0.816(1)   1         8 e  Uiso   0.0175(3)    Si
O1     0.9133(3)   0.6025(6)   0.773(2)   1         8 e  Uiso   0.0204(6)    O
O2     0.7894(3)   0.3170(5)   0.796(2)   1         8 e  Uiso   0.0204(6)    O
O3     0.8368(3)   0.4863(6)   0.786(2)   1         8 e  Uiso   0.0204(6)    O
O4     0.0156(3)   0.6618(5)   0.742(2)   1         8 e  Uiso   0.0204(6)    O
Zn1    0.8354(2)   0.2481(6)   0.561(1)   0.3894    8 e  Uiso   0.030(1)     Zn
O5     0.1150(3)   0.6917(5)   0.699(3)   1         8 e  Uiso   0.0204(6)    O
O6     0.7622(4)   0.4661(5)   0.102(2)   1         8 e  Uiso   0.0204(6)    O
O7     0.9149(3)   0.4737(6)   0.125(2)   1         8 e  Uiso   0.0204(6)    O
O8     0.9270(3)   0.4219(5)   0.621(2)   1         8 e  Uiso   0.0204(6)    O
O9     0.9405(3)   0.7393(9)   0.436(2)   1         8 e  Uiso   0.0204(6)    O
C1     0.8353      0.73903     0.71444    0.108     8 e  Uiso   0.02(1)      C
C2     0.8381      0.84444     0.75685    0.108     8 e  Uiso   0.02(1)      C
C3     0.78085     0.69461     0.78292    0.108     8 e  Uiso   0.02(1)      C
C4     0.80084     0.89757     0.57137    0.108     8 e  Uiso   0.02(1)      C
C5     0.73232     0.74267     0.6583     0.108     8 e  Uiso   0.02(1)      C
C6     0.74591     0.91315     0.69597    0.108     8 e  Uiso   0.02(1)      C
N7     0.71643     0.83071     0.77793    0.108     8 e  Uiso   0.02(1)      N
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H8	0.86852	0.7029	0.81431	0.108	8 e	Uiso	0.02(1)	H
H9	0.84473	0.72534	0.50636	0.108	8 e	Uiso	0.02(1)	H
H10	0.87907	0.86765	0.72058	0.108	8 e	Uiso	0.02(1)	H
H11	0.82517	0.85864	0.95928	0.108	8 e	Uiso	0.02(1)	H
H12	0.77575	0.68686	0.99591	0.108	8 e	Uiso	0.02(1)	H
H13	0.78193	0.62154	0.72515	0.108	8 e	Uiso	0.02(1)	H
H14	0.79582	0.86027	0.38094	0.108	8 e	Uiso	0.02(1)	H
H15	0.81859	0.96623	0.53677	0.108	8 e	Uiso	0.02(1)	H
H16	0.69729	0.69747	0.65965	0.108	8 e	Uiso	0.02(1)	H
H17	0.73946	0.74982	0.44312	0.108	8 e	Uiso	0.02(1)	H
H18	0.72113	0.94913	0.5494	0.108	8 e	Uiso	0.02(1)	H
H19	0.75281	0.95275	0.87623	0.108	8 e	Uiso	0.02(1)	H
H20	0.71912	0.8261	0.9804	0.108	8 e	Uiso	0.02(1)	H
WO	0.3062(3)	0.1570(6)	0.586(1)	0.832(5)	8 e	Uiso	0.06333	O

*Note: The WO site represents the center of gravity of the adsorbed water. A virtual atom consisting of one O atom and two H atoms was applied for all WO sites. Atomic coordinates for all C, H, N atoms were fixed at those obtained by the direct space method analysis. Occupancies for C, H, N atoms were fixed at 0.108 based on the result of CHN analysis.