Supporting Information for:

A Square Pyramidal-Triangular Framework Oxide: Synthesis and Structure of PKU-6

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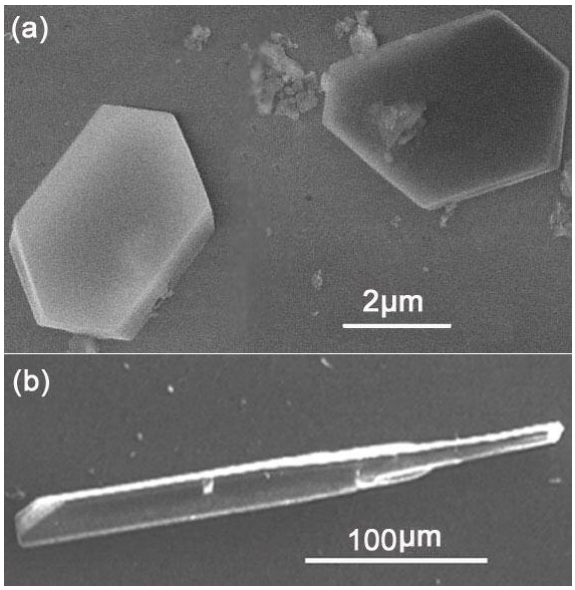

Figure S1. The morphologies of powder sample and single crystal of PKU-6.

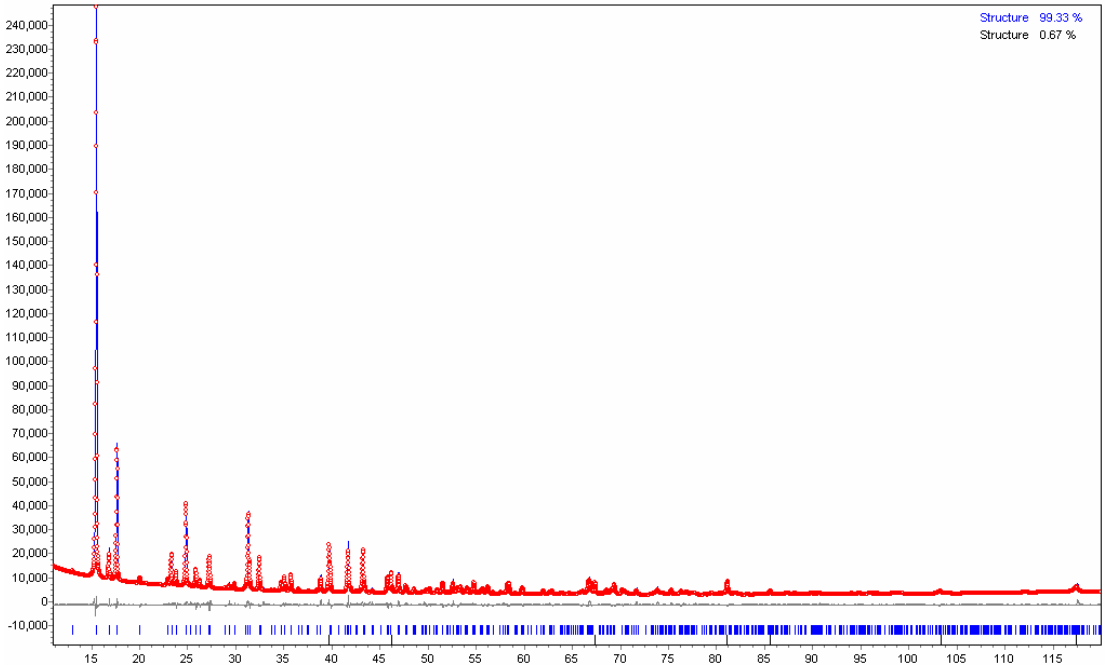

Figure S2. Rietveld plot of the powder X-ray diffraction pattern of PKU-6 ($Al_2(OH)B_3O_7$). The symbol ○ represents observed pattern and the solid line is the calculated pattern; the marks below the diffraction patterns are the reflection positions and, the difference curve is also shown below the diffraction curves. R_p = 0.033, R_{wp} = 0.048.

Table S1. Refined atomic coordinates of PKU-6* from the single crystal diffraction.

site	x	y	z
Al	0.05957(9)	0.38547(7)	0.06842(8)
O1	-0.0755(3)	1/4	0.1180(3)
O2	0.1790(3)	1/4	-0.0275(3)
O3	0.3212(2)	0.36117(17)	-0.2285(2)
O4	0.1197(2)	0.49305(16)	-0.08668(17)
O5	0.2022(2)	0.41853(18)	0.21930(19)
B1	0.2719(5)	1/4	-0.1593(4)
B2	0.2478(4)	0.4808(2)	-0.2012(3)
H1	-0.1707	1/4	0.1742

**Pnma* (No. 62), a = 7.4832(15), b = 10.528(2), c = 8.8525(18) Å, V = 697.4(2) Å^3

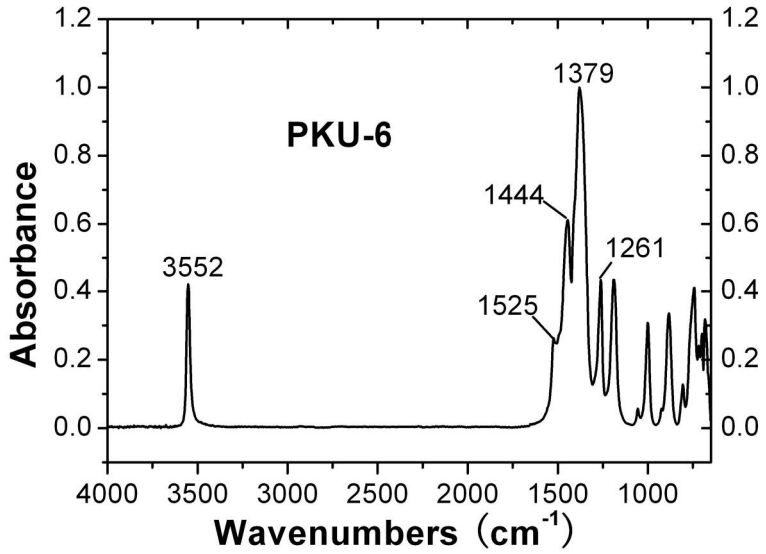

Figure S3. IR spectra of PKU-6 measured with a Nicolet Magna-IR-750 series II. The bands at 1261, 1379, 1444, and 1525 cm^{-1}belong to the B-O antisymmetric stretch in [BO_3]; the band at 3550 cm^{-1} belongs to the –OH bonded to [AlO_5].

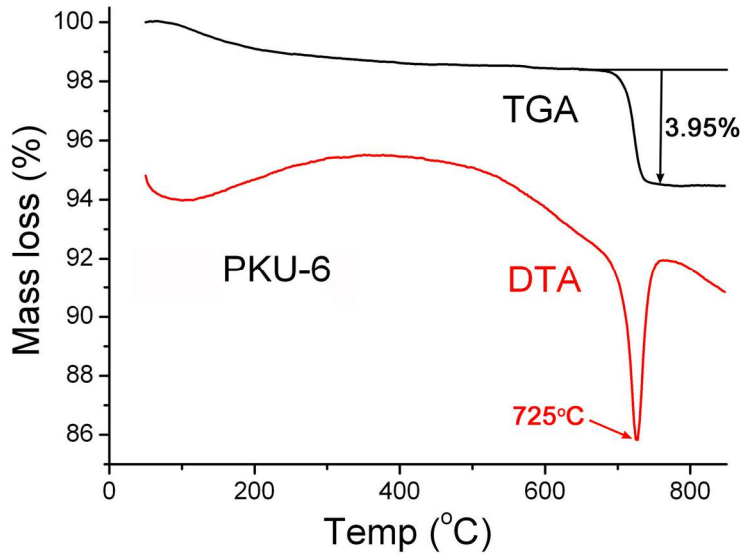

Figure S4. The TGA/DTA curve carried out on a NETZSCH STA449C instrument in a heating rate of 10 °C /min from 30 to 850°C.

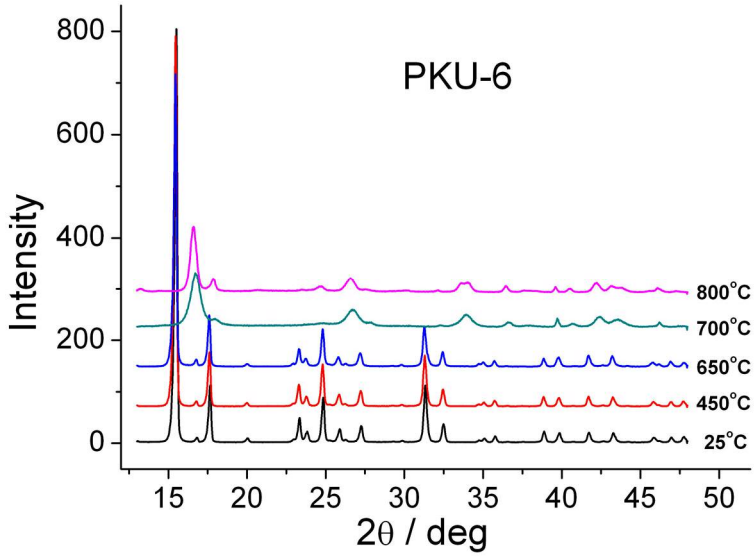

Figure S5. The as-synthesized PKU-6 powder was heated and cooled down to room temperature every step. The X-ray diffraction pattern shows that the open-framework collapses beyond 650 °C and then converts to a known phase $Al_4B_2O_9$, which is agree with the TGA/DTA curve.

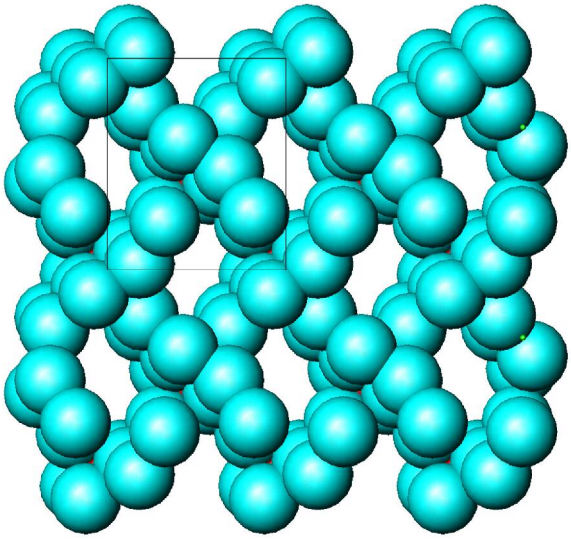

Figure S6. The channels in the structure of PKU-6, in which the oxygen atoms are expressed with Van de Waals radius (1.5 Å).