Supplementary Information

An Unprecedented Organic-Inorganic Hybrid Based on the first \( \{\text{Nb}_{10}\text{V}_{40}\text{O}_{40}(\text{OH})_{2}\}^{12-} \) Clusters and Copper Cations†‡

Peng Huang, Chao Qin, Xin-Long Wang*, Chun-Yi Sun, Guang-Sheng Yang, Kui-Zhan Shao, Yan-Qing Jiao, Kun Zhou and Zhong-Min Su*

Institute of Functional Material Chemistry, Key Lab of Polyoxometalate, Science of Ministry of Education, Faculty of Chemistry, Northeast Normal University, Changchun, 130024 Jilin, People’s Republic of China. Fax: +86-431-85684009; Tel: +86-431-85099108. E-mail: zmsu@nenu.edu.cn
1. Materials and Methods

All the chemicals were obtained from commercial sources, and were used without further purification. Elemental analyses (C, H and N) were measured on a Perkin-Elmer 2400 CHN elemental analyzer; Nb, V and Cu were determined with a Plasma-SPEC(I) ICP atomic emission spectrometer. IR spectrum was performed in the range 4000–400 cm⁻¹ using KBr pellets on an Alpha Centaurt FT/IR spectrophotometer. Powder X-ray diffraction measurement was recorded with radiation ranging from 5 to 50° at room temperature on a Siemens D5005 diffractometer with Cu–Kα (λ = 1.5418 Å). Thermogravimetric analysis (TGA) of the samples was performed using a Perkin-Elmer TG-7 analyzer heated from room temperature to 900 °C under nitrogen at the heating rate of 5 °C·min⁻¹.

2. Synthesis

Syntheses of 1: K₇H₂Nb₆O₁₉·13H₂O was prepared according to the literature method¹ and identified by IR spectra. 1,10-phenanthroline (0.099 g, 0.50 mmol) was added to the solution of Cu(Ac)₂·3H₂O (0.242 g, 1.00 mmol) in water (10 mL) under stirring. Then the resulting blue solution was added dropwise to the solution of K₇H₂Nb₆O₁₉·13H₂O (1.370 g, 1.00 mmol) and NaVO₃·2H₂O (0.3159 g, 2.00 mmol) in water (80 mL) under stirring. Subsequently, the mixture was adjusted to pH 11.2 using NaOH (1 molL⁻¹) solution, condensed to 50 mL at 58 °C for 8 h, filtered and then transferred to a straight glass tube. Over a period of 6 weeks, block-shaped Light-blue single crystals for X-ray crystallography were obtained, washed with distilled water and then air-dried to give 1 in 57% yield (based on K₇H₂Nb₆O₁₉·13H₂O). Elemental analysis: Anal. Calc.: H 2.01; Nb 27.00; V 5.92; Cu 11.08; C 25.11; N 4.88. Found: H 1.92; Nb 27.53; V 5.88; Cu 11.76; C 25.04; N 5.21%.
Fig. S1. Ball-and-stick representations of the I.

Fig. S2. The coordination geometric frameworks of the Cu$^{2+}$(1), Cu$^{2+}$(2), V$^{5+}$(1), Nb$^{5+}$(1) and Nb$^{5+}$(2).
Fig. S3. The IR spectrum of 1.

Fig. S4. The TGA curve of 1.
Fig. S5. The XRPD pattern (top) and simulated pattern (bottom) of 1.

References