Supplementary material: Crystal-to-Crystal Transformation from $K_2[Co(C_2O_4)_2(H_2O)_2] \cdot 4H_2O$ to $K_2[Co(\mu\text{-}C_2O_4)(C_2O_4)]$

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Content

Sample Preparation

1: 300 mg $Co(NO_3)_2 \cdot 6H_2O$, 740 mg $K_2C_2O_4 \cdot H_2O$ were dissolved in 5 mL H_2O at 70°C. The small orange crystal was obtained on the bottom of baker after three days with yield of 60%. Elemental analysis calcd (%) for $C_4H_{12}O_{14}CoK_2$: C 11.40, H 2.87; found: C 11.17, H 2.85. IR (KBr, cm^{-1}): 3396(br,m), 1630(s), 1446(m), 1384(w), 1308(m), 804(m), 774(br,m), 510(m).

2: **1** was heated at 120ºC for three minutes, the colour of crystal change from orange to pink thoroughly. Elemental analysis calcd (%) for $C_4O_8CoK_2$: C 15.34; found: C 15.12. IR (KBr, cm^{-1}): 1659(s), 1624(s), 1427(m), 1384(w), 1338(m), 1307(w), 1284(m), 894(w), 803(m), 782(m), 520(m), 506(m).

Physical Characterization:

Element analysis of carbon and hydrogen were performed on an Elementar Vario EL analyzer. IR spectra were recorded on a Bio-rad FTS6000 spectrometer.

X-ray diffraction data was collected on Beijing Synchtron Radiation Facility with λ = 0.70 Å radiation (**1**) at 100K.[1] The structure were solved by direct method and refined by full-matrix least-square on F^2 using SHELX program, with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms of H_2O were located by different Fourier map and refined isotropically.[2] Crystallographic data of **1**: $C_4H_{12}O_{14}CoK_2$, M_r = 421.27, triclinic, space group $P\bar{1}$, a = 7.684(1) Å, b = 9.011(1) Å, c = 10.874(1) Å, α = 72.151(2)º, β = 70.278(2) º, γ = 80.430(2) º, V = 6701.0(1) Å^3, Z = 2, D_c = 2.085 g cm^{-3}, μ = 1.973 mm^{-1}, 5583 measured data, 3039 unique, R_{int} = 0.1139, R_1 = 0.0466 for 2281 observations of $I \geq 2\sigma(I_0)$, wR_2 = 0.1341 for all data, GOF = 1.004, CCDC842597. **2**: $C_4O_8CoK_2$, M_r = 313.17, monoclinic, space group $P2/c$, a = 8.460 Å b = 6.906 Å, c = 14.657 Å, α = 90 º, β = 93.11 º, γ = 90 º, V = 855.0 Å^3. It is the same as reported CCDC215207.

Thermogravimetric analysis was carried out on a Shimadzu DTG-60 analyzer at a 10°C/min heating rate from room-temperature to 550 °C with Al bag.

Powder X-ray diffraction pattern was obtained on a Rigaku RINT 2000 diffractometer at room temperature with Cu Ka radiation in a flat-plate geometry. The experiment on dehydrated sample was carried out as soon as possible in order to avoid decant in air while quality of X-ray became poor.

Magnetization measurements were performed against tightly packed polycrystalline sample in a capsule on a Quantum Design MPMS 7XL SQUID system under an applied field of 1000 Oe. Susceptibility data was corrected for diamagnetism of sample by Pascal constants (-169.8×10^{-6} cm^{3}mol^{-1} for **1**, -91.8×10^{-6} cm^{3}mol^{-1} for **2**) and background by experimental measurement on the sample holder.[3]

Reference:

1.   Z. Otwinowski and W. Minor, HKL2000, 1997.
2.   G. M. Sheldrick, SHELX-97, University of Gottingen, Göttingen, Germany, 1997.
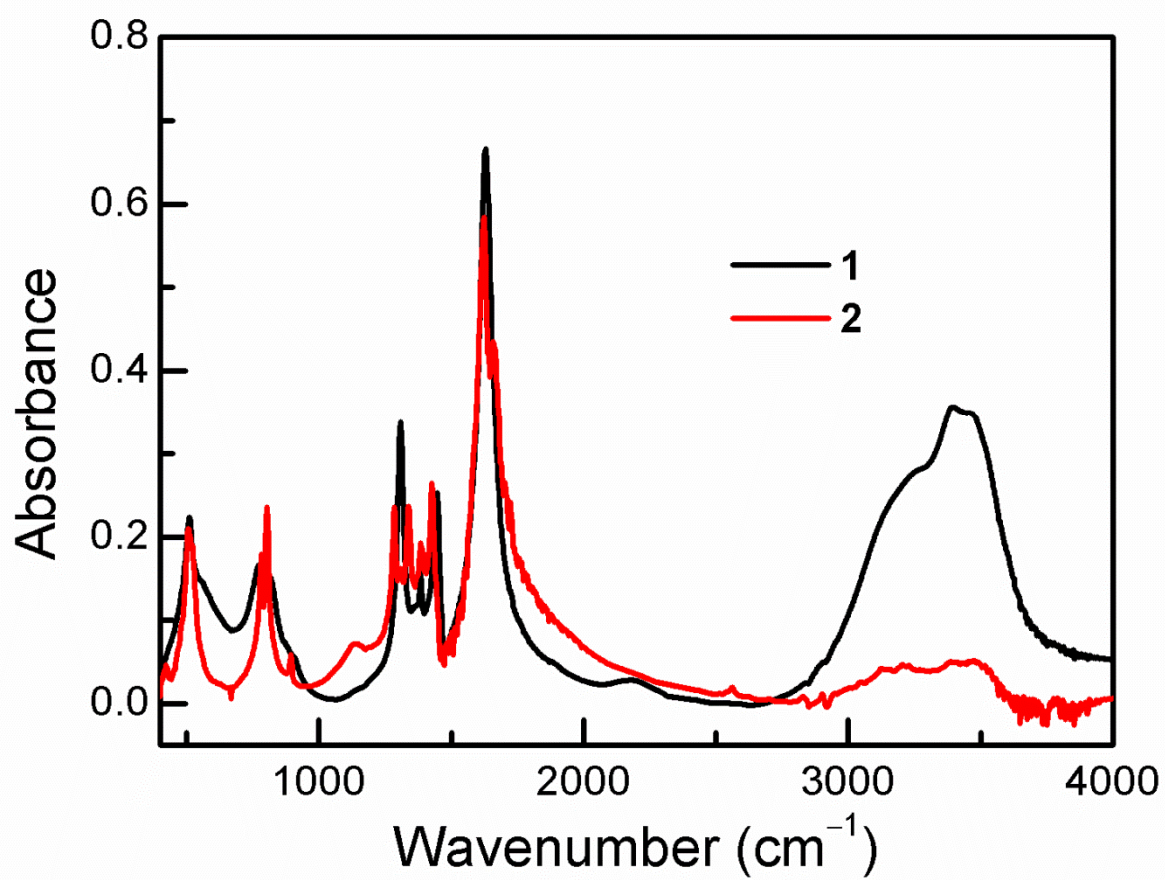3.   O. Kahn, Molecular Magnetism, Wiely, New York, 1993, pp. 10.
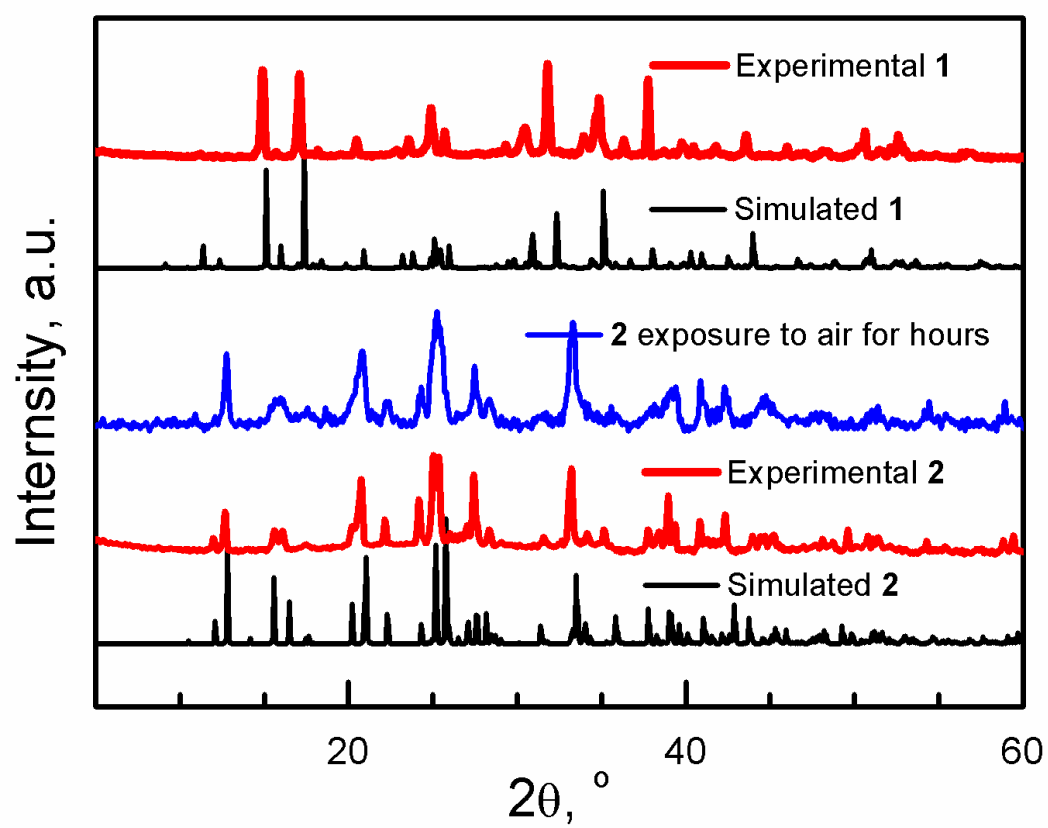
Figure S1. IR spectra of **1** and **2**.

Figure S2. Powder X-ray diffraction patterns of **1** and **2**. Blue one is dehydrated sample exposure to air for hours.