

Supplementary Information:

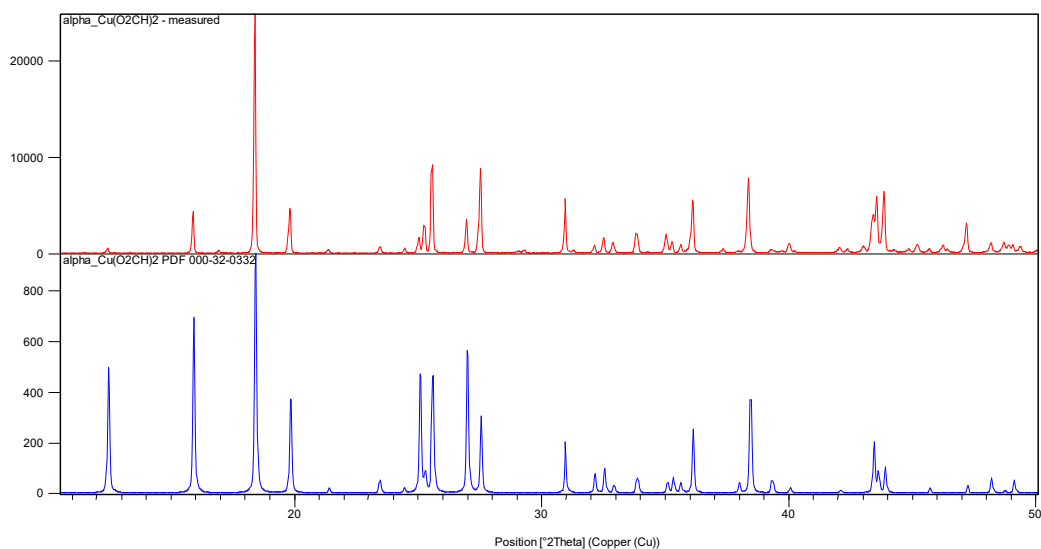


Fig. S1. X-Ray powder pattern of α - $\text{Cu}(\text{O}_2\text{CH})_2$ to confirm its purity (red curve = measured, blue curve = obtained from PDF-2 database, PDF no. 000-32-0332). Results of elemental analysis: Calc. for $\text{C}_2\text{H}_2\text{CuO}_4$: C, 15.6; H, 1.31. Found: C, 15.4; H, 1.03.

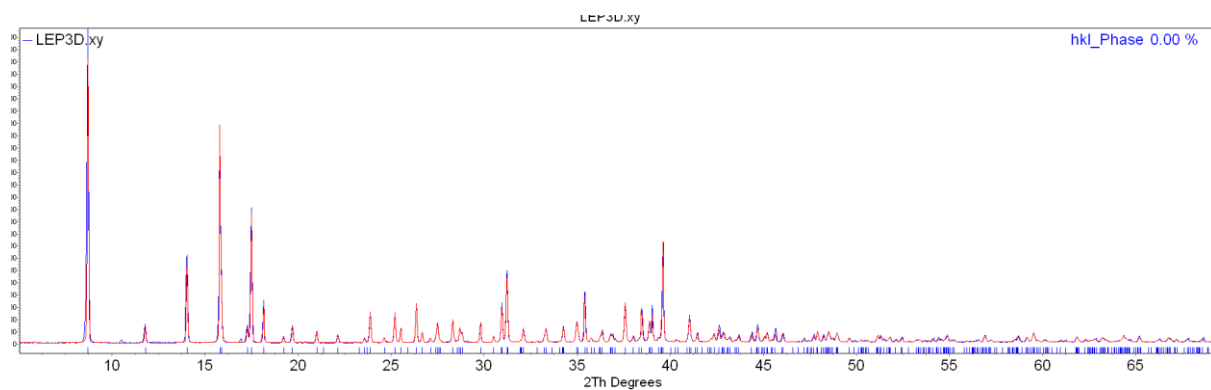


Fig. S2. LeBail fit of $[\text{Cu}_2(\text{O}_2\text{CH})_4(\text{Py})_2]$ to confirm its purity. The unit cell was obtained from CSD (refcode AFMPCB), unit cell parameters as follows: $a=10.190(1)$ Å, $b=11.134(1)$ Å, $c=7.675(1)$ Å $\beta=$, space group $P2_1/c$. For details on unit cell, please see Bernard *et al.*, Rev. Chim. Miner. 1979, 16, 124. Calc. for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{Cu}_2\text{O}_8$: C, 36.13; H, 3.03; N, 6.02. Found: C, 35.21; H, 2.91; N, 5.72.

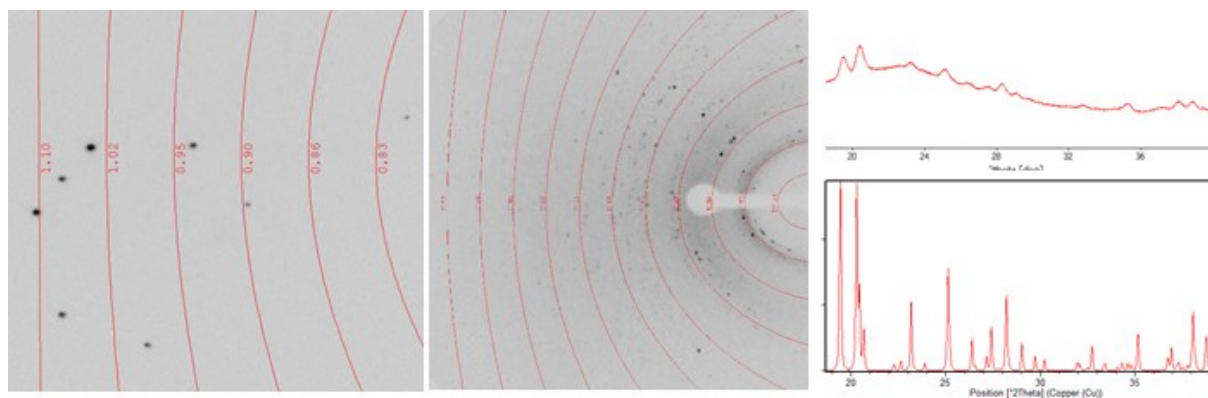


Fig. S3. Left: one frame of single crystal data collection of compound **1**. The single crystal was surrounded with vacuum grease and the experiment was run at 150 K in the stream of liquid nitrogen, yielding the unit cell with the following parameters: $a=12.242$ Å, $b=9.597$ Å, $c=15.563$ Å, $\beta=108.88^\circ$ characteristic for **1**.

Right: one frame of single crystal data collection of the same crystal as previously on left picture. The diffraction data were collected at ambient temperature after 24 h of exposure to air. The sample became polycrystalline and thus, only few frames were collected. From the single crystal experiment the powder pattern was extracted (upper powder pattern) and compared with the calculated one from structure **2** (lower powder pattern). The conversion of **1** to **2** can be confirmed only by matching the most intense diffraction peaks of **2**; all other diffraction peaks are hidden in the background, mainly due to small number of frames and extensive use of vacuum grease.