Supporting materials:

An Unprecedented 9-Fold [3×3] Interpenetrated Diamondoid Network Coordination Polymer Containing Cu(II)-based "Paddlewheels" as Connecting Node

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1. General Procedures

The IR spectrum was recorded as KBr pellets on Bomem MB102 FT-IR spectrometer. The elemental analysis was determined using a Vario EL III CHNOS elemental analyzer. The Diffuse-reflectance absorption spectrum was measured with a PerkinElmer Lambda900 UV/VIS/NIR Spectrometer. The spectrophotometer was calibrated against $BaSO_4$ surface for 100% reflectance over the wavelength range under consideration. Magnetic susceptibility data were measured using a Quantum Design MPMS-XL-5 SQUID Magnetometer, and were corrected for the sample holder and the diamagnetic contribution estimated using Pascal constants.

Single-crystal X-ray diffraction crystallographic data for **1** were collected on a Rigaku Saturn70 CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å) at room temperature. The CrystalClear program was used for the absorption correction. All structures were solved by direct methods and refined on F^2 by full-matrix least-squares methods using the SHELXL-97 program package. All non-hydrogen framework atoms were refined with anisotropic thermal parameters. The lattice water molecule, free chloride, hydroxide oxygen were refined with isotropic thermal parameters. One anion position was occupied by two kinds of atoms (Cl and O atom from hydroxide anion) based on the elemental analysis result, with the occupancy ratio of 1:1. The hydrogen atoms of lattice water molecule were not included in **1**.

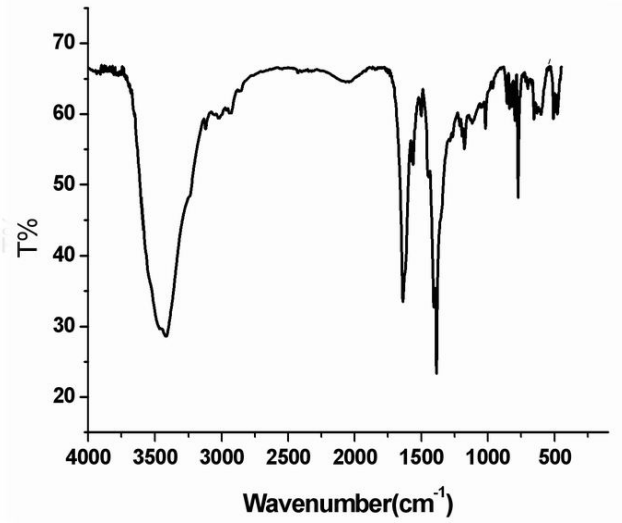

Fig. S1 IR spectrum of compound **1**.