NOTE

Crystal Structure of Molybdate Pyridine Copper: $C_{10}H_{10}N_2O_{10}Cu_3Mo_2$

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A new multicore metal complex $C_{10}H_{10}N_2O_{10}Cu_3Mo_2$ has been synthesized from a hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. The compound consisted of three Cu atoms, two molybdate and two pyridine. The two-dimensional network crystal structure is formed through extensive O-H...O hydrogen bonding.

Key Words: Multicore complex, Hydrogen bond, Structure analysis.

The metal-organic framework can combine both inorganic and organic solids characteristics, which exhibits high porosity and a wide diversity of metallic centers and has broad prospect. The metal-organic framework matter have paid most attention due to their potential applications in diverse areas such as catalysis, optoelectronics, supramolecular storage of molecules, molecular magnetism, separation, nanoreactors, *etc*¹⁻⁵. The chemistry of polyoxometalates is a rapidly growing field because the nanomolecular entities exhibit a unique combination of tunable properties, including composition, size, shape, charge density, redox potentials and solubility⁶⁻⁸. One main feature of polyoxometalate is that they provide structurally well-characterized surfaces formed by approximately coplanar, closest-packed O atoms, forming a light, electricity and magnetism materials⁹⁻¹¹. In this paper, the novel molybdate pyridine multicore copper complex is reported.

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of $CuCl_2$ (0.1 mmol, 0.015 g), $(NH_4)_2MoO_4$ (0.1 mmol, 0.02 g), pyridine (5 mL) and benzene (1 mL) were added into 20 mL water with 50 % (v/v) ethanol and heated for 8 h at 413 K. The solution was obtained by filtration after cooling the reaction to room temperature. Green sheet single crystals suitable for X-ray measurements were obtained after a few days.

The crystal structure of molybdate pyridine multicore copper complex ($C_{10}H_{10}N_2O_{10}Cu_3Mo_2$) (Fig. 1) is build up of three Cu atom, two molybdate and two pyridine.

Fig. 1. Molecular structure of the title compound with atom-labelling scheme

The crystal data and structure refinement is shown in Table-1. The molecular contains multicore. The Cu1 atom is coordinated by one N atom and five O atoms (two O1, O2, O3, O5 atom) to form distorted octahedra. The Cu2 atom is coordinated by six O atoms (two O1, two O2, two O3). There are 3 μ_3 -O atoms, 1 μ_2 -O atom and one terminal O4 atom. The μ_2 -O5 bridges Cu1 and Mo atom. The μ_3 -O1 atom bridges Cu1, Cu1 and Cu2 atom, while μ_3 -O2 and μ_3 -O3 bridges Cu1, Cu2 and Mo atom. The distances Cu-O are in the range of 1.924-2.333 Å. The distance Cu-Cu is 3.046 Å. The angles of O1-Cu1-N, O2-Cu2-O1 are 99.9 and 86.3°. The flats of two pyridine ring are parallel. Selected bond lengths and bond angles are shown in Table-2.

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT
FOR THE TITLE COMPLEX

Empirical formula	C ₁₀ H ₁₀ Cu ₃ Mo ₂ N ₂ O ₁₀
Formula weight	700.70
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 5.5653(10) Å α = 93.9170(10)° b = 5.7312(11) Å β = 101.249(2)° c = 13.516(2) Å γ = 98.983(2)°
Volume	415.46(13) Å ³
Z, Calculated density	1, 2.801 Mg/m ³
Absorption coefficient	5.301 mm ⁻¹
F(000)	335
Crystal size	0.40 × 0.37 × 0.05 mm
Theta range for data collection	1.54 to 25.00°
Limiting indices	-6 ≤ h ≤ 6, -6 ≤ k ≤ 6, -16 ≤ l ≤ 14
Reflections collected / unique	2187/1440 [R _{int}] = 0.0586]
Completeness to theta = 25.00	98.6 %
Absorption correction	Semi-empirical from equivalents
Max and min transmission	0.7775 and 0.2256
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	1440/0/149
Goodness-of-fit on F ²	1.052
Final R indices [I > 2σ (I)]	R1 = 0.0686, wR2 = 0.1870
R indices (all data)	R1 = 0.0725, wR2 = 0.1936
Largest diff. peak and hole	2.468 and -2.234 e.Å ⁻³

TABLE-2
SELECT BOND LENGTHS [Å] AND
ANGLES [°] FOR THE TITLE COMPLEX

Mo(1)-O(4)	1.726(7)
Mo(1)-O(3)	1.751(7)
Mo(1)-O(5)	1.797(6)
Mo(1)-O(2)	1.805(6)
Cu(1)-O(1)	2.000(6)
Cu(1)-N(1)	2.016(6)
Cu(2)-O(2)	1.924(7)
Cu(2)-O(1)	2.035(6)
N(1)-C(1')	1.37(3)
N(1)-C(1)	1.3900
C(1)-C(2)	1.3900
C(2)-H(2)	0.9300
C(3)-C(2')	1.37(3)
O(4)-Mo(1)-O(3)	108.5(4)
O(3)-Mo(1)-O(5)	110.3(3)
O(4)-Mo(1)-O(2)	107.9(3)
O(3)-Mo(1)-O(2)	111.5(3)
O(5)-Mo(1)-O(2)	110.6(3)
O(1)-Cu(1)-N(1)	99.9(3)
O(1)#2-Cu(1)-N(1)	160.4(3)
O(2)-Cu(2)-O(1)	86.3(3)
Cu(1)-O(1)-Cu(2)	111.6(3)
Mo(1)-O(2)-Cu(2)	129.6(4)
C(1)-N(1)-Cu(1)	121.6(5)
C(5)-N(1)-Cu(1)	118.4(4)

Crystal is stabilized by lots of O-H...O hydrogen bonds interaction to form two-dimensional network structure

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