Asian Journal of Chemistry; Vol. 25, No. 14 (2013), 8219-8220

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.14619

NOTE

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

HAI-XING LIU[1,*], LIN-TONG WANG[1], FANG-FANG JIAN[2], JING WANG[2], QING LIU[3], KAI-QI YE[3], GUANG ZENG[4] and XI-SHI TAI[1]

[1]Chemistry and Chemical and Environmental Engineering College, Weifang University, Weifang 261061, P.R. China
[2]New Materials and Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao 266042, P.R. China
[3]College of Chemistry, State Key Laboratory of Supramolecular Structure and Materials, Jilin University, Changchun 130012, P.R. China
[4]State Key Laboratory of Inorganic Synthesis and Preparative Chemistry, College of Chemistry, Jilin University, Changchun 130012, P.R. China

*Corresponding author: E-mail: haixingliu@tom.com

(*Received*: 25 September 2012; *Accepted*: 12 August 2013) AJC-13912

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 solubility[6-8]. 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 materials[9-11]. 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.

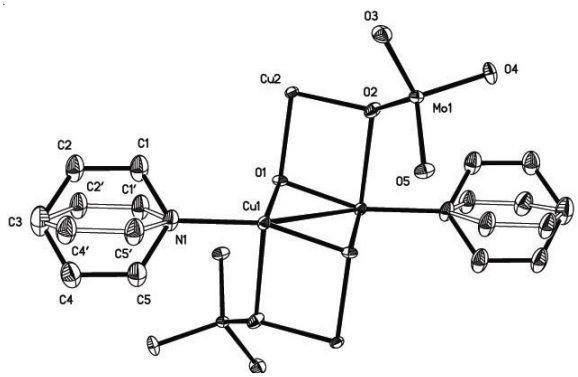

Fig. 1. Molecular structure of the title compound with atom-labling scheme

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 Cu_2 atom is coordinated by sixO 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, Cu_2 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 86.3º. 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	$C_{10}H_{10}Cu_3Mo_2N_2O_{10}$
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)º b = 5.7312(11) Å β = 101.249(2)º c = 13.516(2) Å γ = 98.983(2)º
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 25.00º
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

ACKNOWLEDGEMENTS

This study were supported by the Natural Science Foundation of Shandong Province (No. ZR2010BL025), State Key Laboratory of Inorganic Synthesis and Preparative Chemistry (Jilin University) (No. 2011-13), MOE Key Laboratory of Analytical Chemistry for Life Science (Nanjing University) (No. KLACLS1002) and the National Science Foundation of China (No. 21171132) and Open Project of State Key Laboratory of Supramolecular Structure and Materials, Jilin University (No. sklssm201323).

REFERENCES

1. W. Wang, L.-G. Qiu, G.-Q. Shao, Y. Wu and Y. Wang, *Asian J. Chem.*, **24**, 4041 (2012).
2. D.N. Lee, J.Y. Ryu, H. Kwak, Y.M. Lee, S.H. Lee, J.I. Poong, J. Lee, W. Shin, C. Kim, S.-J. Kim and Y. Kim, *J. Mol. Struct.*, **885**, 56 (2008).
3. U. Mueller, M. Schubert, F. Teich, H. Puetter, K. Schierle-Arndt and J. Pastre, *J. Mater. Chem.*, **16**, 626 (2006).
4. S. Konar, P.S. Mukherjee, E. Zangrando, F. Lloret and N.R. Chaudhuri, *Angew. Chem.*, **41**, 1561 (2002).
5. Y.K. Park, S.B. Choi, H. Kim, K. Kim, B.-H. Won, K. Choi, J.-S. Choi, W.-S. Ahn, N. Won, S. Kim, D.H. Jung, S.-H. Choi, G.-H. Kim, S.-S. Cha, Y.H. Jhon, J.K. Yang and J. Kim, *Angew. Chem.*, **119**, 8237 (2007).
6. N.H. Nsouli, B.S. Bassil, M.H. Dickman, U. Kortz, B. Keita and L. Nadjo, *Inorg. Chem.*, **45**, 3858 (2006).
7. M.T. Pope, *Comput. Coord. Chem. II*, **4**, 635 (2003).
8. J.J. Borras-Almenar, E. Coronado, A. Muller and M.T. Pope, Polyoxometalate Molecular Science, Springer (2003).
9. C.J. Gomez-Garcia, C. Gimenez-Saiz, S. Triki, E. Coronado, P. Le Magueres, L. Ouahab, L. Ducasse, C. Sourisseau and P. Delhaes, *Inorg. Chem.*, **34**, 4139 (1995).
10. E. Coronado, J.R. Galán-Mascarós, C. Giménez-Saiz, C.J. Gómez-García, C. Rovira, J. Tarrés, S. Triki and J. Veciana, *J. Mater. Chem.*, **8**, 313 (1998).
11. H.X. Liu, F.-F. Jian and J. Wang, *J. Chem. Crystallogr.*, **40**, 306 (2010).