Asian Journal of Chemistry; Vol. 25, No. 14 (2013), 8227-8228

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.14661

NOTE

Study on Novel Structure of Molybdenum Tungsten Imidazole Complex: $(H_{12}MoW_7O_{30})(C_3H_4N_2)_4(H_2O)_3$

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(*Received*: 9 October 2012; *Accepted*: 12 August 2013) AJC-13916

A novel molybdenum tungsten imidazole complex $(H_{12}MoW_7O_{30})(C_3H_4N_2)_4(H_2O)_3$ with the m.f. $C_{12}H_{34}MoN_8O_{33}W_7$ has been synthesized from a single solution reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. The Mo atom is coordinated by seven O atoms. There are two kinds coordinations mode for W atom. The crystal is stabilized by O-H···O and N-H···O hydrogen bonds interaction.

Key Words: Molybdenum tungsten complex, Imidazole, Structure analysis.

Supramolecular compounds based on polyoxometalates construct a small subset of the large family of polyoxometalates which have attracted considerable attention due to variety of their structures and potential application[1-3]. The self-assembly of supramolecular structure performs *via* ligand-metal coordination bonds, hydrogen bonds, electrostatic force and π-π stacking interaction[4]. Recently, the strategy of self-assembly through intermolecular weak interactions is a focus of the design of new polyoxometalates based hybrids[5], which have been intensively investigated in many important aspects such as catalysis, electrical conductivity, magnetic properties and biological chemistry[6]. In this paper, the novel molybdenum tungsten imidazole complex is reported.

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of $MnSO_4$ (0.01 mmol, 0.002 g), $(NH_4)_2WO_4$ (0.1 mmol, 0.03 g), $(NH_4)_2MoO_4$ (0.1 mmol, 0.02 g), imidazole (0.1 mmol, 0.007 g), citric acid (0.1 mmol, 0.02 g) and dilute HCl were added into 20 mL water with 20 % (v/v) ethanol and heated for 5 h at 343 K. The solution was obtained by filtration after cooling the reaction to room temperature. Colourless block single crystals suitable for X-ray measurements were obtained after a few weeks.

The title crystal structure (Fig. 1) is built up of molybdenum tungsten acid, imidazole and water molecular. The 1D chain structure extending along b axis is shown in Fig. 2. The View of the three-fold interpenetration network along the b axis is shown in Fig. 3. The crystal data and structure refinement is shown in Table-1. The Mo atom is coordinated by seven O atoms (one O1, two O1w, two O2w, two O3w). The W2 atom is coordinated by five O atoms and the others W atoms are coordinated by six O atoms. The μ2-O1 atom bridges Mo1 and W2. The μ3-O8 atom bridges W1, W3 and W4. The μ4-O7 atom bridges two W1, W2 and W4, while μ4-O13 atom bridges two W3, W4 and W5. The distances of d(Mo-O) and d(W-O) are in the range of 2.428-2.496 Å and 1.697-2.506 Å, respectively. The bond angles of Mo1-O1-W2 is 161.2°. Selected bond lengths and bond angles are shown in Table-2.

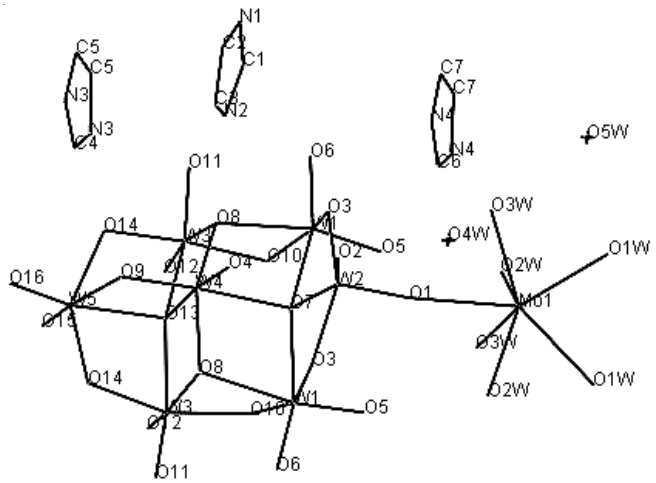

Fig. 1. Molecular structure of the title complex

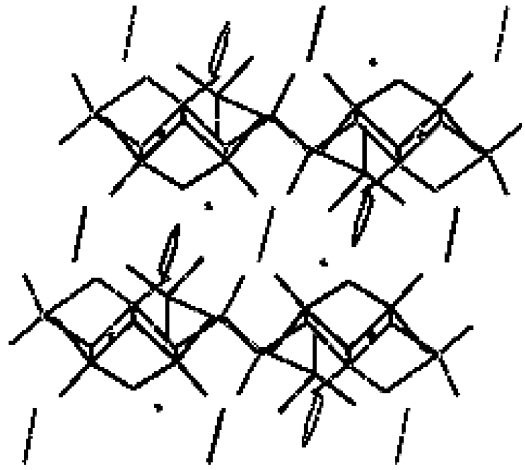

Fig. 2. 1D chain structure extending along b axis

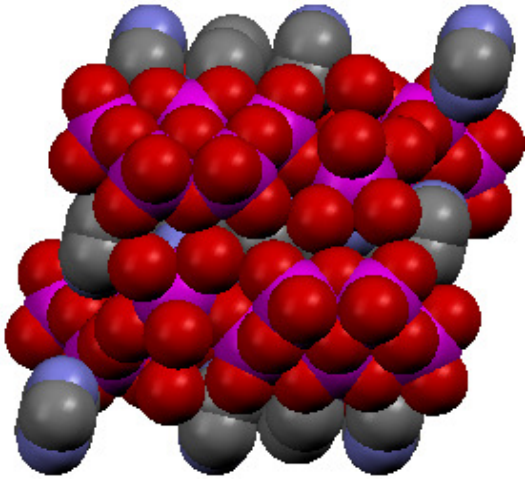

Fig. 3. Perspective view of the 3D packing diagram along the b axis

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

Empirical formula	$C_{17}H_{34}MoN_8O_{33}W_7$
Formula weight	2201.36
Temperature	291(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C^2/m
Unit cell dimensions	a = 15.6529(8) Å α = 90° b = 19.3356(8) Å β = 103.975(4)° c = 14.1045(5) Å γ = 90°
Volume	4142.5(3) $Å^3$
Z, Calculated density	4, 3.530 Mg/m^3
Absorption coefficient	19.755 mm^{-1}
F(000)	3944
Crystal size	0.28 × 0.24 × 0.22 mm
Theta range for data collection	1.49 to 26.00°
Limiting indices	-8 <= h <= 19, -23 <= k <= 22, -17 <= l <= 17
Reflections collected/unique	12725/4205 [R(int) = 0.0141]
Completeness to θ = 26	99.8 %
Absorption correction	Semi-empirical from equivalents
Max and min transmission	0.02 and 0.01
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4205/0/296
Goodness-of-fit on F^2	1.092
Final R indices [I > 2σ (I)]	R1 = 0.0210, wR2 = 0.0653
R indices (all data)	R1 = 0.0237, wR2 = 0.0676
Largest diff. peak and hole	0.618 and -1.868 $e.Å^{-3}$

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

C(1)-N(1)	1.310(8)	O(4W)-H(4X)	0.8500
C(1)-N(2)	1.366(7)	W(1)-W(2)	3.1993(3)
C(1)-H(1)	0.9300	N(1)-C(1)-N(2)	108.0(5)
C(2)-C(3)	1.307(9)	C(3)-C(2)-N(1)	107.8(5)
C(2)-N(1)	1.377(9)	C(2)-C(3)-N(2)	109.0(6)
C(2)-H(2)	0.9300	N(2)-C(3)-H(3)	125.5
C(3)-N(2)	1.343(8)	N(4)-C(6)-H(6)	123.9
C(4)-N(3)	1.290(7)	C(1)-N(1)-C(2)	107.8(5)
C(5)-N(3)	1.372(7)	C(4)-N(3)-C(5)	108.5(5)
C(6)-N(4)	1.286(8)	C(7)-N(4)-H(4A)	127.3
C(7)-N(4)	1.375(9)	W(2)-O(1)-Mo(1)	161.2(3)
Mo(1)-O(3W)	2.428(4)	W(4)-O(4)-W(2)	105.1(2)
Mo(1)-O(1)	2.470(5)	W(4)-O(9)-W(5)	108.9(2)
Mo(1)-O(1W)	2.470(4)	O(8)-W(1)-W(2)	87.34(8)
Mo(1)-O(2W)	2.496(4)	O(1)-W(2)-O(2)	105.5(2)
N(2)-H(2A)	0.8600	O(2)-W(2)-O(7)	150.6(2)
O(1)-W(2)	1.704(5)	O(2)-W(2)-W(1)	135.66(5)
O(2)-W(2)	1.756(5)	O(7)-W(2)-W(1)	42.25(3)
O(3)-W(1)	1.941(4)	O(4)-W(2)-W(1)	83.32(8)
O(4)-W(4)	1.732(5)	O(9)-W(4)-O(4)	105.9(2)
O(5)-W(1)	1.707(4)	O(4)-W(4)-O(13)	173.0(2)
O(6)-W(1)	1.697(4)	O(4)-W(4)-O(7)	83.6(2)
O(7)-W(2)	2.141(5)	O(15)-W(5)-O(16)	105.0(2)
O(8)-W(4)	1.894(3)	O(15)-W(5)-O(13)	106.1(2)
O(9)-W(4)	1.723(5)	O(15)-W(5)-O(9)	174.7(2)
O(1W)-H(1X)	0.9600	O(16)-W(5)-O(9)	80.3(2)

Crystal is stabilized by O-H···O and N-H···O hydrogen bonds interaction

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).

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