Dong-Dong Li, Hui Jiang, Bing-Xin Jia, Yuan Yuan, Ye-Shun Shi and Feng-Xia Ma*

Crystal structure of bis[diaqua(1,10-phenanthroline- κ^2N, N')-copper(II)] diphenylphosphopentamolybdate dihydrate, $C_{36}H_{38}Cu_2Mo_5N_4O_{27}P_2$

Table 1: Data collection and handling.

Crystal:	Blue bar-type
Size:	0.23 × 0.11 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.28 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Pro, ω
$\theta_{\max}$, completeness:	26.4°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	24274, 4925, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4449
$N(\text{param})_{\text{refined}}$:	349
Programs:	Olex2 [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.63257(2)	0.41491(2)	0.34264(2)	0.01789(6)
Mo2	0.60065(2)	0.65864(2)	0.30226(2)	0.01926(6)
Mo3	0.5000	0.27051(2)	0.2500	0.02044(7)
Cu1	0.46976(2)	0.83095(2)	0.37170(2)	0.02576(8)
P1	0.42135(4)	0.48870(5)	0.30294(2)	0.01573(12)
O1	0.37272(11)	0.58457(13)	0.28222(7)	0.0210(4)
O2	0.40688(11)	0.40478(13)	0.25774(7)	0.0189(3)
O3	0.51842(10)	0.51486(13)	0.31901(7)	0.0187(3)
O4	0.63088(13)	0.42165(16)	0.41386(8)	0.0304(4)
O5	0.72634(13)	0.35031(15)	0.33798(9)	0.0309(4)
O6	0.54722(12)	0.31127(13)	0.32586(7)	0.0208(4)
O7	0.42136(15)	0.19128(16)	0.26599(9)	0.0390(5)
O8	0.67551(11)	0.55065(13)	0.32967(7)	0.0210(4)
O9	0.67070(12)	0.74822(15)	0.28553(9)	0.0312(4)
O10	0.5000	0.69468(18)	0.2500	0.0227(5)
O11	0.56783(12)	0.70808(15)	0.36263(8)	0.0287(4)
O12	0.37107(13)	0.73368(15)	0.36318(8)	0.0304(4)
H12A	0.3753	0.6891	0.3362	0.046*
H12B	0.3219	0.7642	0.3505	0.046*
O13	0.42974(16)	0.88133(16)	0.29469(8)	0.0383(5)
H13A	0.3969	0.8470	0.2660	0.057*
H13B	0.3937	0.9303	0.2960	0.057*
N1	0.55633(15)	0.94344(18)	0.38536(10)	0.0277(5)
N2	0.49572(15)	0.81892(17)	0.45673(10)	0.0263(5)
C1	0.38319(16)	0.44382(19)	0.36589(10)	0.0202(5)
C2	0.31877(18)	0.3708(2)	0.36136(12)	0.0286(6)
H2	0.2969	0.3437	0.3258	0.034*
C3	0.2870(2)	0.3383(3)	0.40992(14)	0.0387(7)
H3	0.2438	0.2896	0.4070	0.046*
C4	0.3197(2)	0.3784(3)	0.46238(13)	0.0425(8)
H4	0.2989	0.3561	0.4950	0.051*
C5	0.3831(2)	0.4517(3)	0.46695(12)	0.0405(8)

<https://doi.org/10.1515/ncrs-2019-0032>

Received January 11, 2019; accepted March 11, 2019; available online June 18, 2019

Abstract

$C_{36}H_{38}Cu_2Mo_5N_4O_{27}P_2$, monoclinic, $C2/c$ (no. 15), $a = 15.6362(11)$ Å, $b = 13.2418(9)$ Å, $c = 23.6641(14)$ Å, $\beta = 98.858(6)^\circ$, $V = 4841.2(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0219$, $wR_{\text{ref}}(F^2) = 0.0522$, $T = 293(2)$ K.

CCDC no.: 1888567

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of $CuCl_2 \cdot 6H_2O$ (0.7 mmol, 0.1190 g), 1,10-phenanthroline (phen) (0.5 mmol, 0.0914 g), Na_2MoO_4 (0.6 mmol, 0.1500 g), phenylphosphonic acid (0.3 mmol,

*Corresponding author: Feng-Xia Ma, College of Biology and Pharmaceutical Engineering, Jilin Agricultural Science and Technology University, Jilin 132101, P.R. China, e-mail: fengxia_ma@126.com

Dong-Dong Li, Hui Jiang, Bing-Xin Jia, Yuan Yuan and Ye-Shun Shi: College of Biology and Pharmaceutical Engineering, Jilin Agricultural Science and Technology University, Jilin 132101, P.R. China

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H5	0.4044	0.4789	0.5026	0.049*
C6	0.41528(19)	0.4848(2)	0.41905(11)	0.0296(6)
H6	0.4581	0.5341	0.4223	0.036*
C7	0.4642(2)	0.7547(2)	0.49122(13)	0.0338(6)
H7	0.4239	0.7068	0.4756	0.041*
C8	0.4900(2)	0.7571(2)	0.55047(13)	0.0391(7)
H8	0.4667	0.7111	0.5736	0.047*
C9	0.5489(2)	0.8262(2)	0.57446(12)	0.0376(7)
H9	0.5657	0.8280	0.6139	0.045*
C10	0.58440(19)	0.8951(2)	0.53907(12)	0.0307(6)
C11	0.55495(17)	0.8878(2)	0.48025(11)	0.0251(5)
C12	0.58821(17)	0.9555(2)	0.44186(11)	0.0259(5)
C13	0.64948(18)	1.0285(2)	0.46210(13)	0.0328(6)
C14	0.6784(2)	1.0345(3)	0.52248(14)	0.0417(8)
H14	0.7192	1.0828	0.5367	0.050*
C15	0.6474(2)	0.9712(3)	0.55894(14)	0.0403(7)
H15	0.6672	0.9769	0.5979	0.048*
C16	0.6803(2)	1.0900(3)	0.42107(16)	0.0446(8)
H16	0.7220	1.1390	0.4325	0.054*
C17	0.6489(2)	1.0774(3)	0.36479(15)	0.0462(8)
H17	0.6688	1.1180	0.3375	0.055*
C18	0.5866(2)	1.0035(2)	0.34780(13)	0.0383(7)
H18	0.5656	0.9959	0.3091	0.046*
O14	0.32890(18)	1.0308(2)	0.28591(17)	0.0722(10)
H14A	0.2783	1.0358	0.2946	0.108*
H14B	0.3488	1.0896	0.2813	0.108*

0.0550 g) were added to distilled water (15 mL). The pH of the mixture was adjusted to 3.0 with 2 mol · L^{−1} HCl and then stirred for 30 min in air. The solution was then transferred into a 25 mL Teflon-lined autoclave and crystallized at 150 °C for 5 days. Then the autoclave was cooled at 10 °C · h^{−1} to room temperature. Blue bar-type crystals were separated and washed with distilled water.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Polyoxometalate multicomponent molecular species are a current field of research [4–8]. The title compound is depicted in the figure. The complex species shows a C₂ symmetry. Bond lengths and angles are in the expected ranges [9–11].

Acknowledgements: This work was supported by the Jilin Agricultural Science and Technology University Students Science and Technology Innovation and Entrepreneurship Training Program Project (No. 201811439007).

References

- Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
- Sheldrick, G. M.: SHELXT-integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
- Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
- Hill, C. L.: Introduction: polyoxometalates multicomponent molecular vehicles to probe fundamental issues and practical problems. *Chem. Rev.* **98** (1998) 1–2.
- Shi, S. Y.; Zou, Y. C.; Gui, X. B.; Xu, J. N.; Wang, Y.; Wang, G. W.; Yang, G. D.; Xu, J. Q.; Wang, T. G.; Gao, Z. M.: 0D and 1D dimensional structures based on the combination of polyoxometalates, transition metal coordination complexes and organic amines. *CrystEngComm* **12** (2010) 2122–2128.
- Qu, X.; Feng, H.; Ma, C.; Yang, Y.; Yu, X.: Synthesis, crystal structure and anti-tumor activity of a novel 3D supramolecular compound constructed from Strandberg-type polyoxometalate and benzimidazole. *Inorg. Chem. Commun.* **81** (2017) 22–26.
- Yuan, L.; Qin, C.; Wang, X. L.; Wang, E. B.; Chang, S.: Two extended organic-inorganic assemblies based on polyoxometalates and copper coordination polymers with mixed 4,4'-bipyridine and 2,2'-bipyridine ligands. *Eur. J. Inorg. Chem.* **31** (2008) 4936–4942.
- Kendell, S. M.; Nguyen, N. H.; Brown, T. C.: Electronic activity relationship for methacrolein formation over 4th period transition metal phosphomolybdates. *Catal. Lett.* **143** (2013) 61–70.
- Shi, S.; Chen, L.; Zhao, X.; Ren, B.; Cui, X.; Zhang, J.: Role of hydrogen-bonds in the crystal packing of three novel Strandberg-type polyoxoanion compounds. *Inorg. Chim. Acta* **482** (2018) 870–877.
- Ji, Y. M.; Fang, Y.; Han, P. P.; Li, M. X.; Chen, Q. Q.; Han, Q. X.: Copper(II) and cadmium(II) complexes derived from Strandberg-type polyoxometalate clusters: synthesis, crystal structures, spectroscopy and biological activities. *Inorg. Chem. Commun.* **86** (2017) 22–25.
- Feng, S. L.; Lu, Y.; Zhang, Y. X.; Su, F.; Sang, X. J.; Zhang, L. C.; Zhu, Z. M.: Three new Strandberg-type phenylphosphomolybdate supports for immobilizing horseradish peroxidase and their catalytic oxidation performances. *Dalton Trans.* **47** (2018) 14060–14069.