Feng-Xia Ma*, Jin-Ke Jiang, Yuan Yuan and Dong-Dong Li

Crystal structure of tetrakis(1*H*-benzo[*d*]imidazol-3-ium) bis(μ_5 -phenylphosphonato)-pentakis(μ_2 -oxido)-decaoxo-penta-molybdenum dihydrate, $C_{40}H_{42}Mo_5N_8O_{23}P_2$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.14 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.32 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Pro, ω
$\theta_{\max}$, completeness:	26.4°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	47853, 10563, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 9260
$N(\text{param})_{\text{refined}}$:	716
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.39679(2)	0.07754(2)	0.26401(2)	0.02166(5)
Mo2	0.68880(2)	0.09653(2)	0.30293(2)	0.02173(5)
Mo3	0.75903(2)	0.30795(2)	0.25247(2)	0.02406(5)
Mo4	0.50035(2)	0.42947(2)	0.20406(2)	0.02417(5)
Mo5	0.25864(2)	0.30185(2)	0.23645(2)	0.02383(5)
P1	0.47018(5)	0.26055(4)	0.36868(3)	0.01958(11)
P2	0.55727(5)	0.22150(4)	0.13457(3)	0.01873(11)
O1	0.80069(16)	0.37732(12)	0.32148(12)	0.0359(4)
O2	0.89090(16)	0.28750(13)	0.18178(12)	0.0381(4)
O3	0.67170(14)	0.38395(10)	0.17596(10)	0.0264(3)
O4	0.51573(18)	0.51309(11)	0.26586(12)	0.0398(4)
O5	0.48999(16)	0.47792(12)	0.10303(11)	0.0354(4)
O6	0.32642(15)	0.41463(10)	0.24163(11)	0.0284(4)
O7	0.46742(13)	0.29602(10)	0.17229(10)	0.0227(3)
O8	0.48842(14)	0.13992(10)	0.13804(9)	0.0225(3)
O9	0.22771(16)	0.31522(12)	0.13192(11)	0.0354(4)
O10	0.11689(15)	0.32175(12)	0.29663(12)	0.0379(4)
O11	0.28352(14)	0.17760(10)	0.24896(10)	0.0248(3)
O12	0.33830(15)	0.04360(11)	0.36818(11)	0.0300(4)
O13	0.34987(16)	0.00435(11)	0.20184(11)	0.0328(4)
O14	0.50650(13)	0.17188(9)	0.32797(10)	0.0221(3)
O15	0.56922(14)	0.03172(10)	0.25976(10)	0.0232(3)
O16	0.69209(16)	0.04466(11)	0.40269(11)	0.0339(4)
O17	0.81769(15)	0.05271(12)	0.24159(11)	0.0339(4)
O18	0.75027(14)	0.20458(10)	0.32939(10)	0.0255(3)
O19	0.67094(13)	0.20448(10)	0.18098(10)	0.0228(3)
O20	0.55061(14)	0.33132(10)	0.32252(10)	0.0238(3)
O21	0.33355(14)	0.28558(10)	0.36557(10)	0.0253(3)
C29	0.5004(3)	0.2180(2)	0.65856(17)	0.0443(7)

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Abstract

$C_{40}H_{42}Mo_5N_8O_{23}P_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.9867(2)$ Å, $b = 15.4753(3)$ Å, $c = 15.5666(3)$ Å, $\alpha = 85.583(2)^\circ$, $\beta = 81.294(2)^\circ$, $\gamma = 84.844(2)^\circ$, $V = 2600.18(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0208$, $wR_{\text{ref}}(F^2) = 0.0485$, $T = 293(2)$ K.

CCDC no.: 2004515

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 $CoCl_2 \cdot 6 H_2O$ (0.5 mmol, 0.1190 g), benzimidazole (0.6 mmol, 0.0709 g), Na_2MoO_4 (0.6 mmol, 0.1500 g),

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H29	0.5010	0.2098	0.7183	0.053*
C30	0.5249(3)	0.2974(2)	0.61624(18)	0.0461(7)
H30	0.5441	0.3422	0.6472	0.055*
C31	0.5209(3)	0.31082(17)	0.52678(17)	0.0359(6)
H31	0.5374	0.3644	0.4982	0.043*
C32	0.4922(2)	0.24388(15)	0.48085(14)	0.0231(4)
C33	0.4709(2)	0.16290(16)	0.52389(16)	0.0318(5)
H33	0.4539	0.1174	0.4931	0.038*
C34	0.4750(3)	0.15039(19)	0.61274(18)	0.0406(6)
H34	0.4607	0.0965	0.6414	0.049*
C35	0.6012(2)	0.24905(15)	0.02064(14)	0.0247(5)
C36	0.7075(3)	0.2095(2)	−0.02331(18)	0.0523(8)
H36	0.7616	0.1744	0.0073	0.063*
C37	0.7339(3)	0.2222(3)	−0.1133(2)	0.0737(12)
H37	0.8055	0.1951	−0.1427	0.088*
C38	0.6546(3)	0.2744(2)	−0.15896(18)	0.0564(9)
H38	0.6725	0.2823	−0.2192	0.068*
C39	0.5501(3)	0.31477(19)	−0.11634(18)	0.0458(7)
H39	0.4974	0.3509	−0.1473	0.055*
C40	0.5222(3)	0.30186(17)	−0.02648(16)	0.0353(6)
H40	0.4501	0.3288	0.0024	0.042*
O1W	0.7235(4)	0.4936(3)	0.5835(3)	0.1100(14)
H1WA	0.661(4)	0.516(4)	0.557(3)	0.165*
H2WA	0.744(2)	0.512(5)	0.425(4)	0.165*
O2W	0.6641(5)	0.5118(3)	0.4173(3)	0.1456(19)
H2WB	0.672(6)	0.468(4)	0.382(4)	0.218*
N1	1.1052(3)	0.3308(2)	0.48403(19)	0.0634(8)
H1	1.1665	0.3175	0.4448	0.076*
N2	0.9265(3)	0.3848(2)	0.5438(2)	0.0734(9)
H2	0.8543	0.4121	0.5495	0.088*
C1	0.9495(4)	0.3345(3)	0.7002(3)	0.0704(11)
H1A	0.8726	0.3570	0.7262	0.084*
C2	1.0326(4)	0.2896(3)	0.7478(3)	0.0774(12)
H2A	1.0110	0.2808	0.8078	0.093*
C3	1.1479(4)	0.2569(3)	0.7093(3)	0.0786(12)
H3	1.2021	0.2273	0.7440	0.094*
C4	1.1837(3)	0.2673(3)	0.6206(3)	0.0689(10)
H4	1.2611	0.2457	0.5947	0.083*
C5	1.0999(3)	0.3112(2)	0.5721(2)	0.0531(8)
C6	0.9864(3)	0.3444(2)	0.6116(2)	0.0550(8)
C7	1.0003(4)	0.3734(3)	0.4702(3)	0.0688(10)
H7	0.9808	0.3930	0.4156	0.083*
N3	0.8219(2)	0.40483(16)	0.01056(15)	0.0427(6)
H3A	0.8046	0.3757	0.0596	0.051*
N4	0.7979(2)	0.48706(14)	−0.10405(15)	0.0376(5)
H4A	0.7626	0.5194	−0.1420	0.045*
C8	1.0545(3)	0.3737(2)	−0.0208(2)	0.0542(9)
H8	1.0650	0.3370	0.0281	0.065*
C9	1.1526(3)	0.3955(2)	−0.0824(3)	0.0656(11)
H9	1.2315	0.3720	−0.0751	0.079*
C10	1.1377(3)	0.4518(2)	−0.1553(3)	0.0579(9)
H10	1.2070	0.4651	−0.1947	0.069*
C11	1.0238(3)	0.4879(2)	−0.17045(19)	0.0443(7)
H11	1.0138	0.5260	−0.2185	0.053*
C12	0.9241(2)	0.46425(16)	−0.10978(16)	0.0303(5)
C13	0.9392(2)	0.40980(16)	−0.03627(17)	0.0343(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C14	0.7414(3)	0.45132(19)	−0.03139(19)	0.0425(6)
H14	0.6569	0.4580	−0.0125	0.051*
N5	0.3488(2)	0.06143(15)	−0.11989(14)	0.0393(5)
H5	0.3655	0.0423	−0.1711	0.047*
N6	0.3740(2)	0.10580(15)	0.00466(14)	0.0391(5)
H6	0.4100	0.1199	0.0463	0.047*
C15	0.1488(3)	0.1340(2)	0.0670(2)	0.0493(7)
H15	0.1603	0.1522	0.1205	0.059*
C16	0.0324(3)	0.1297(3)	0.0453(3)	0.0658(10)
H16	−0.0365	0.1457	0.0850	0.079*
C17	0.0161(3)	0.1018(3)	−0.0355(3)	0.0769(13)
H17	−0.0639	0.0994	−0.0476	0.092*
C18	0.1136(3)	0.0779(2)	−0.0975(2)	0.0599(10)
H18	0.1018	0.0605	−0.1513	0.072*
C19	0.2311(3)	0.08140(18)	−0.07532(18)	0.0383(6)
C20	0.2475(3)	0.10963(17)	0.00492(17)	0.0363(6)
C21	0.4302(3)	0.07691(18)	−0.06995(18)	0.0398(6)
H21	0.5154	0.0686	−0.0852	0.048*
N7	0.1139(2)	0.10011(16)	0.45920(17)	0.0425(6)
H7A	0.1904	0.0810	0.4557	0.051*
N8	−0.0582(2)	0.15757(15)	0.42120(14)	0.0366(5)
H8A	−0.1109	0.1815	0.3893	0.044*
C22	−0.0709(3)	0.0792(2)	0.67875(19)	0.0508(8)
H22	−0.0696	0.0601	0.7367	0.061*
C23	−0.1817(3)	0.1161(2)	0.6535(2)	0.0462(7)
H23	−0.2520	0.1208	0.6950	0.055*
C24	−0.1895(2)	0.14546(18)	0.56897(19)	0.0376(6)
H24	−0.2634	0.1700	0.5521	0.045*
C25	−0.0820(2)	0.13682(15)	0.51001(16)	0.0273(5)
C26	0.0286(2)	0.09963(16)	0.53476(17)	0.0315(5)
C27	0.0365(3)	0.07029(19)	0.6207(2)	0.0453(7)
H27	0.1102	0.0460	0.6379	0.054*
C28	0.0587(3)	0.13449(19)	0.3939(2)	0.0440(7)
H28	0.0969	0.1415	0.3366	0.053*
H1WB	0.711(7)	0.515(5)	0.626(4)	0.14(3)*

phenylphosphonic acid (0.3 mmol, 0.0550 g) were added to distilled water (15 mL). The pH of the mixture was adjusted to 2.0 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 160 °C for 5 days. Then the autoclave was cooled at 10 °C · h^{−1} to room temperature. Block colourless crystals of the unexpected cobalt free title compound 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

Metal-organic frameworks (MOFs) containing polyoxometalates have caught the increasing attention of researchers

due to their novel structural characteristics and applications in catalysis [5], magnetism [6], antibacterial drugs [7], and so on.

The title compound is synthesized under hydrothermal condition and its asymmetric unit contains a Strandberg-type anion, four protonated-benzimidazole counter cations and two water molecules (see the Figure). Its bond lengths and angles are in the expected ranges [8, 9]. Its structure is three-dimensional due to the existence of some weaker interaction such as $CH \cdots O$ and $NH \cdots O$ hydrogen bonds.

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