Crystal structure of *tetrakis*-(imidazol-1-ylmethyl)methane cobalt(II) tungstate-monohydrate, $[CoWO_4(C_{17}H_{20}N_8)](H_2O)$, $C_{17}H_{22}CoN_8O_5W$

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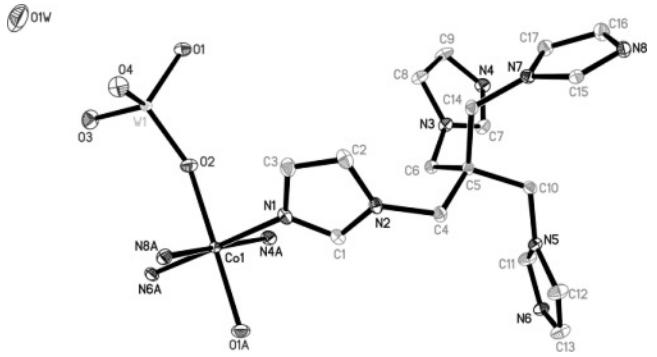

Abstract

$C_{17}H_{22}CoN_8O_5W$, monoclinic, $P2_1/n$ (no. 14), a = 9.4893(2) Å, b = 18.1400(4) Å, c = 12.2891(2) Å, β = 102.035(2)°, V = 2068.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.0252, $wR_{ref}(F^2)$ = 0.0716, T = 293 K.

Table 1. Data collection and handling.

Crystal:	purple blocks, size 0.16×0.18×0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	64.07 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9197, 4785
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4182
$N(param)_{refined}$:	295
Programs:	SHELX [3]

Source of material

$NaWO_4{\cdot}2H_2O$ (329.9 mg, 1mmol), $Co(OAc)_2{\cdot}2H_2O$ (50.0 mg, 0.2mmol) and *tetrakis*-(imidazol-1-ylmethyl)methane (L, 33.6 mg, 0.1mmol) were dissolved in 8 mL of distilled water with stirring at room temperature. The pH value of the mixture was adjusted to 6 with 1.0 mol·L^{-1} HCl, then the cloudy solution was put into a 18 mL Teflon-lined autoclave and heated to 170 °C for 72 h. After the solution was slowly cooled to room temperature at a rate of 10 °C·h^{-1}, purple block crystals were obtained in 51% yield.

Experimental details

All H atoms bound to carbon were refined using a riding model with d(C–H) = 0.93 Å, U_{iso} = 1.2 U_{eq}(C) for aromatic and 0.97 Å, U_{iso} = 1.5 U_{eq}(C) for CH_2 atoms. Water H atoms and hydroxyl H atoms were located in a difference Fourier map and refined as riding atoms with d(O–H) = 0.85-0.89 Å and U_{iso} = 1.5 U_{eq}(O). Because of series termination errors, there are some higher peaks after structure refinement.

Discussion

Among the *N*-donor bridging ligands, the *tetrakis*-(imidazol-1-ylmethyl)methane ligand (L) is expected to be a good candidate for construction of coordination polymers with diverse structures [1]. In the title compound, Co1 is in an octahedral coordination sphere, which is defined by two oxygen atoms from two different tungstate anions (Co1–O1A = 2.092 and Co1–O2 = 2.098 Å) and four nitrogen atoms from four different L ligands (Co1–N1 = 2.134, Co1–N4A = 2.220, Co1–N6A = 2.102, and Co1–N8A = 2.208(5) Å). All of the bond distances and bond angles are in normal ranges.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	1.1015	0.9085	0.3018	0.025
H(2)	4*e*	0.6854	0.8594	0.2427	0.031
H(3)	4*e*	0.8185	0.7582	0.1779	0.033
H(4A)	4*e*	0.9551	1.0052	0.3643	0.023
H(4B)	4*e*	0.8008	0.9832	0.3799	0.023
H(6A)	4*e*	0.9797	1.0435	0.1647	0.021
H(6B)	4*e*	0.8898	0.9730	0.1231	0.021
H(7)	4*e*	0.8925	1.1678	0.0490	0.026
H(8)	4*e*	0.7139	0.9790	–0.0669	0.029
H(10A)	4*e*	0.7655	1.1481	0.2116	0.021
H(10B)	4*e*	0.7387	1.1192	0.3254	0.021
H(11)	4*e*	1.0439	1.1714	0.2059	0.029
H(12)	4*e*	0.9395	1.1382	0.4960	0.035
H(13)	4*e*	1.1764	1.1966	0.5261	0.034
H(14A)	4*e*	0.6226	0.9869	0.1204	0.023
H(14B)	4*e*	0.6053	0.9832	0.2441	0.023
H(15)	4*e*	0.4641	1.0828	0.3141	0.022
H(16)	4*e*	0.3778	1.2159	0.0585	0.030
H(17)	4*e*	0.5355	1.1201	0.0122	0.027
H(9)	4*e*	0.6583	1.0732	–0.2125	0.032
H(1A)	4*e*	0.780(4)	0.510(2)	–0.063(3)	0.022
H(1B)	4*e*	0.768(5)	0.441(2)	–0.065(3)	0.022

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	1.0151(4)	0.8833(2)	0.2793(3)	0.019(2)	0.021(2)	0.022(2)	0.003(1)	0.005(1)	0.003(1)
C(2)	4*e*	0.7843(5)	0.8570(2)	0.2470(3)	0.021(2)	0.022(2)	0.039(2)	0.002(2)	0.013(2)	0.006(2)
C(3)	4*e*	0.8592(4)	0.8014(2)	0.2114(3)	0.024(2)	0.019(2)	0.041(2)	–0.001(2)	0.010(2)	–0.001(2)
C(4)	4*e*	0.8634(4)	0.9847(2)	0.3269(3)	0.025(2)	0.020(2)	0.015(2)	0.005(2)	0.006(1)	–0.000(1)
C(5)	4*e*	0.7956(4)	1.0357(2)	0.2288(3)	0.016(2)	0.014(2)	0.015(1)	0.000(1)	0.004(1)	0.001(1)
C(6)	4*e*	0.8827(4)	1.0254(2)	0.1369(2)	0.021(2)	0.019(2)	0.011(1)	0.001(1)	0.005(1)	0.001(1)
C(7)	4*e*	0.8410(4)	1.1327(2)	0.0015(3)	0.031(2)	0.018(2)	0.017(2)	–0.004(2)	0.005(2)	0.001(1)
C(8)	4*e*	0.7429(4)	1.0281(2)	–0.0610(3)	0.029(2)	0.022(2)	0.018(2)	–0.009(2)	0.000(2)	0.000(1)
N(4)	4*e*	0.7765(4)	1.1460(2)	–0.1010(3)	0.027(2)	0.023(1)	0.017(1)	0.000(1)	0.003(1)	0.005(1)
C(10)	4*e*	0.8025(4)	1.1155(2)	0.2734(3)	0.015(2)	0.019(2)	0.019(2)	0.002(1)	0.003(1)	0.000(1)
C(11)	4*e*	1.0482(4)	1.1702(2)	0.2822(3)	0.023(2)	0.035(2)	0.016(2)	–0.009(2)	0.005(2)	–0.000(1)
C(12)	4*e*	0.9893(5)	1.1515(2)	0.4413(3)	0.028(2)	0.044(2)	0.016(2)	–0.008(2)	0.007(2)	–0.002(2)
C(13)	4*e*	1.1198(4)	1.1841(2)	0.4573(3)	0.027(2)	0.038(2)	0.018(2)	–0.009(2)	0.003(2)	–0.004(2)
C(14)	4*e*	0.6331(4)	1.0150(2)	0.1887(3)	0.020(2)	0.013(2)	0.024(2)	–0.001(1)	0.005(1)	–0.001(1)
C(15)	4*e*	0.4620(4)	1.1044(2)	0.2452(3)	0.019(2)	0.020(2)	0.016(2)	–0.002(1)	0.002(1)	–0.000(1)
C(16)	4*e*	0.4154(5)	1.1769(2)	0.1046(3)	0.031(2)	0.024(2)	0.021(2)	0.005(2)	0.005(2)	0.005(1)
C(17)	4*e*	0.5025(4)	1.1241(2)	0.0781(3)	0.024(2)	0.025(2)	0.020(2)	–0.000(2)	0.007(2)	–0.001(1)
N(1)	4*e*	1.0037(3)	0.8179(2)	0.2321(3)	0.020(2)	0.020(1)	0.026(2)	0.004(1)	0.006(1)	0.004(1)
N(2)	4*e*	0.8863(3)	0.9091(2)	0.2908(2)	0.022(2)	0.016(1)	0.019(1)	0.003(1)	0.007(1)	0.004(1)
N(3)	4*e*	0.8243(3)	1.0619(2)	0.0309(2)	0.024(2)	0.018(1)	0.016(1)	–0.003(1)	0.005(1)	0.001(1)
C(9)	4*e*	0.7131(4)	1.0804(2)	–0.1413(3)	0.030(2)	0.032(2)	0.017(2)	–0.014(2)	0.000(2)	–0.002(1)
N(5)	4*e*	0.9444(3)	1.1419(2)	0.3287(2)	0.018(2)	0.019(1)	0.017(1)	–0.002(1)	0.003(1)	–0.002(1)
N(6)	4*e*	1.1564(3)	1.1961(2)	0.3565(2)	0.019(2)	0.022(2)	0.019(1)	–0.006(1)	0.002(1)	–0.001(1)
N(7)	4*e*	0.5329(3)	1.0774(2)	0.1688(2)	0.015(1)	0.020(1)	0.017(1)	0.000(1)	0.004(1)	0.001(1)
N(8)	4*e*	0.3897(4)	1.1648(1)	0.2103(3)	0.022(2)	0.020(2)	0.020(2)	0.003(1)	0.005(1)	0.000(1)
O(1)	4*e*	0.8182(3)	0.7107(2)	–0.1652(2)	0.028(2)	0.032(1)	0.020(1)	0.002(1)	–0.004(1)	–0.002(1)
O(2)	4*e*	1.0375(3)	0.7210(2)	0.0424(2)	0.028(2)	0.029(2)	0.022(1)	–0.001(1)	–0.005(1)	–0.001(1)
O(1W)	4*e*	0.7363(5)	0.4770(2)	–0.1059(4)	0.068(3)	0.034(2)	0.084(3)	–0.015(2)	0.014(2)	–0.012(2)
O(3)	4*e*	1.0484(3)	0.6037(2)	–0.1153(2)	0.033(2)	0.035(2)	0.037(2)	0.003(1)	0.015(1)	–0.008(1)
O(4)	4*e*	0.8258(3)	0.6038(2)	0.0089(2)	0.035(2)	0.035(2)	0.028(1)	–0.004(1)	0.012(1)	0.006(1)
Co(1)	4*e*	1.17519(5)	0.75517(2)	0.19024(3)	0.0130(2)	0.0148(2)	0.0140(2)	0.0004(2)	0.0009(2)	–0.0003(2)
W(1)	4*e*	0.93340(1)	0.660450(7)	–0.05756(1)	0.01319(9)	0.01761(9)	0.01344(8)	0.00001(5)	0.00138(6)	–0.00037(4)

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