Crystal structure of bis{[4′-(4-tolyl)-2,2′:6′2″-terpyridine-κ^3N,N',N'']-[μ-5-hydroxybenzene-1,3-dicarboxylate-κ^2O,O']zinc(II)} tetrahydrate $[Zn(C_{22}H_{17}N_3)(C_8H_6O_5)]_2 \cdot 4H_2O$

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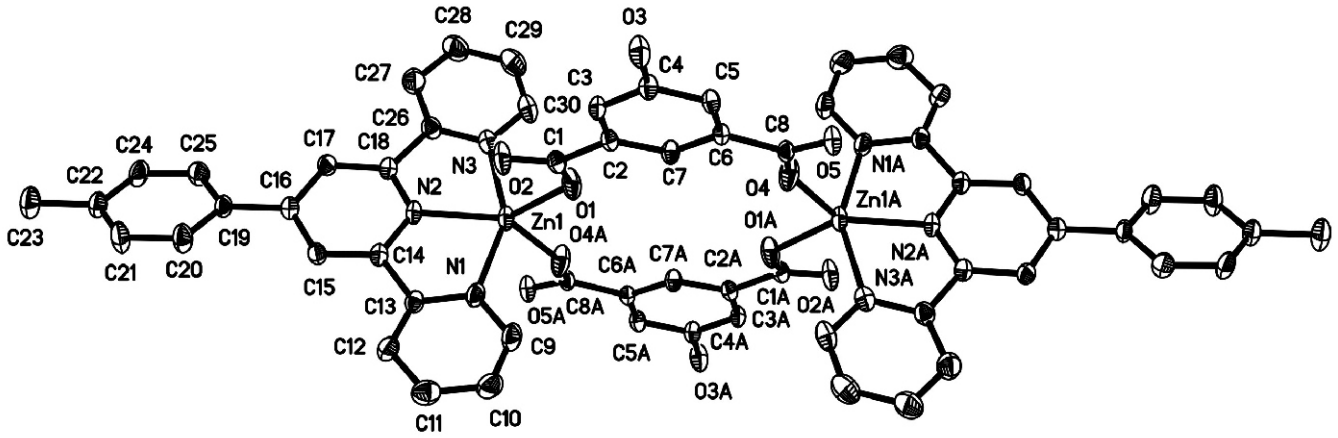

Abstract

$C_{60}H_{54}N_6O_{14}Zn_2$, monoclinic, $P12_1/c1$ (no. 14), a = 13.0900(9) Å, b = 14.8630(7) Å, c = 14.061 Å, β = 97.92°, V = 2709.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.067, $wR_{ref}(F^2)$ = 0.190, T = 293 K.

Source of material

All chemicals were purchased commercially and used without purification. 4′-(4-tolyl)-2,2′:6′2″-terpyridine was prepared according to [1]. The title compound was synthesized by the following procedures. A mixture of 4′-(4-tolyl)-2,2′:6′2″-terpyridine (0.0323 g, 0.1 mmol), 5-hydroxybenzene-1,3-dicarboxylic acid (0.0364 g, 0.2 mmol), and $Zn(CH_3COO)_2 \cdot 2H_2O$ (0.022 g, 0.01 mmol) in methanol/H_2O (1:1, v/v, 10 ml) was heated at 413 K in a Teflon-lined stainless steel autoclave for 3 days. The reaction system was then slowly cooled to room temperature. Colorless crystals suitable for X-ray diffraction analysis were collected by filtration (yield 25 %).

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.93 Å and U_{iso}(H) = 1.5 U_{eq}(C). The structure was checked with PLATON [2], which did not indicate problems. The hydrogen atoms bound to O1W and O2W atoms could not be located in reasonable positions.

Discussion

Recently, much attention in the field of metal-organic frameworks has been focused on the design and synthesis of metal-organic coordination networks. In this regard, the organic ligand plays a critical role in directing the final structures and topologies of the coordination compounds. The N-containing ligands are especially good candidates for the construction of coordination compounds [3].

In the title crystal structure, each Zn(II) cation is five-coordinated by three N atoms from 4′-(4-tolyl)-2,2′:6′2″-terpyridine and two O atoms from two 5-hydroxybenzene-1,3-dicarboxylic acid ligands forming a slightly distorted trigonal bipyramid. The distances d(Zn—N) are in the range of 2.056(4) - 2.181(4) Å, d(Zn—O) = 1.962(3) Å. The bond distances and angles are in normal ranges.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.15 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.60 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12617, 6241
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3441
$N(param)_{refined}$:	370
Programs:	PLATON [2], SHELXS-97, SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(5)	4*e*	0.8275	0.2602	0.0488	0.041
H(3)	4*e*	0.6683	0.2388	0.2723	0.039
H(7)	4*e*	0.5863	0.0927	0.0341	0.039
H(17)	4*e*	0.1267	0.1073	0.4373	0.044
H(15)	4*e*	0.3370	–0.0691	0.5589	0.043
H(25)	4*e*	0.0363	0.0744	0.5493	0.055
H(12)	4*e*	0.4810	–0.1429	0.5371	0.053
H(24)	4*e*	–0.0366	0.0750	0.6868	0.059
H(20)	4*e*	0.2938	–0.0279	0.6927	0.066
H(10)	4*e*	0.6595	–0.2204	0.3424	0.069
H(21)	4*e*	0.2197	–0.0250	0.8306	0.072
H(11)	4*e*	0.6190	–0.2268	0.4978	0.068
H(9)	4*e*	0.5599	–0.1353	0.2322	0.065
H(30)	4*e*	0.2380	0.1130	0.0478	0.064
H(23A)	4*e*	–0.0337	0.0529	0.8519	0.097
H(23B)	4*e*	0.0750	0.0679	0.9121	0.097
H(23C)	4*e*	0.0325	–0.0299	0.8924	0.097
H(28)	4*e*	0.0183	0.2483	0.1550	0.075
H(27)	4*e*	0.0721	0.1822	0.3035	0.065
H(29)	4*e*	0.1031	0.2107	0.0256	0.077

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}
Zn(1)	4*e*	0.36823(4)	–0.00136(4)	0.21798(4)	0.0391(3)	0.0482(4)	0.0280(3)	–0.0117(3)	0.0150(2)	–0.0058(2)
O(5)	4*e*	0.8070(2)	0.1661(2)	–0.1038(2)	0.041(2)	0.062(2)	0.028(2)	–0.010(2)	0.014(1)	–0.002(2)
O(3)	4*e*	0.8201(3)	0.3264(2)	0.2129(2)	0.050(2)	0.063(2)	0.033(2)	–0.030(2)	0.013(2)	–0.012(2)
O(2)	4*e*	0.5236(3)	0.1295(3)	0.2920(2)	0.061(2)	0.077(3)	0.035(2)	–0.028(2)	0.025(2)	–0.007(2)
C(4)	4*e*	0.7572(3)	0.2621(3)	0.1680(3)	0.030(2)	0.045(3)	0.030(2)	–0.004(2)	0.007(2)	–0.003(2)
C(5)	4*e*	0.7736(3)	0.2351(3)	0.0766(3)	0.030(2)	0.044(3)	0.032(2)	–0.005(2)	0.012(2)	0.002(2)
C(3)	4*e*	0.6782(3)	0.2226(3)	0.2103(3)	0.036(3)	0.041(3)	0.024(2)	–0.004(2)	0.011(2)	–0.003(2)
C(19)	4*e*	0.1747(4)	0.0223(3)	0.6041(3)	0.035(3)	0.046(3)	0.030(2)	0.004(2)	0.013(2)	0.001(2)
C(2)	4*e*	0.6139(3)	0.1589(3)	0.1602(3)	0.030(2)	0.038(2)	0.029(2)	–0.003(2)	0.009(2)	0.004(2)
C(8)	4*e*	0.7298(3)	0.1395(3)	–0.0699(3)	0.034(3)	0.038(3)	0.021(2)	0.003(2)	0.006(2)	0.001(2)
C(7)	4*e*	0.6299(3)	0.1344(3)	0.0683(3)	0.030(2)	0.043(3)	0.025(2)	–0.006(2)	0.003(2)	–0.005(2)
N(2)	4*e*	0.3092(3)	0.0104(2)	0.3456(3)	0.031(2)	0.045(2)	0.025(2)	–0.006(2)	0.010(2)	–0.005(2)
C(17)	4*e*	0.1827(3)	0.0695(3)	0.4344(3)	0.032(3)	0.043(3)	0.036(3)	0.008(2)	0.011(2)	0.005(2)
C(1)	4*e*	0.5302(3)	0.1156(3)	0.2065(3)	0.030(2)	0.041(3)	0.034(2)	–0.004(2)	0.010(2)	0.004(2)
N(1)	4*e*	0.4639(3)	–0.0879(3)	0.3155(3)	0.043(2)	0.045(2)	0.046(2)	–0.002(2)	0.024(2)	–0.005(2)
C(18)	4*e*	0.2268(3)	0.0634(3)	0.3507(3)	0.033(3)	0.041(3)	0.028(2)	–0.002(2)	0.006(2)	–0.003(2)
C(16)	4*e*	0.2225(4)	0.0188(3)	0.5144(3)	0.036(3)	0.044(3)	0.030(2)	–0.001(2)	0.008(2)	–0.001(2)
C(6)	4*e*	0.7106(3)	0.1716(3)	0.0269(3)	0.027(2)	0.036(2)	0.022(2)	–0.000(2)	0.006(2)	0.000(2)
C(13)	4*e*	0.4413(3)	–0.0922(3)	0.4059(3)	0.033(2)	0.037(3)	0.035(2)	–0.006(2)	0.010(2)	–0.005(2)
N(3)	4*e*	0.2369(3)	0.0891(3)	0.1852(3)	0.043(2)	0.047(2)	0.032(2)	–0.009(2)	0.008(2)	0.000(2)
C(15)	4*e*	0.3082(3)	–0.0349(3)	0.5066(3)	0.036(3)	0.043(3)	0.030(2)	0.003(2)	0.008(2)	0.004(2)
O(4)	4*e*	0.6632(3)	0.0877(3)	–0.1124(2)	0.063(2)	0.075(3)	0.041(2)	–0.026(2)	0.022(2)	–0.028(2)
C(25)	4*e*	0.0743(4)	0.0537(4)	0.6059(3)	0.042(3)	0.060(3)	0.036(3)	0.013(3)	0.002(2)	0.006(2)
C(12)	4*e*	0.4976(4)	–0.1420(3)	0.4749(4)	0.043(3)	0.048(3)	0.043(3)	0.006(2)	0.013(2)	–0.006(2)
C(24)	4*e*	0.0308(4)	0.0546(4)	0.6884(4)	0.040(3)	0.066(4)	0.045(3)	0.011(3)	0.018(2)	–0.005(3)
C(14)	4*e*	0.3503(3)	–0.0379(3)	0.4228(3)	0.034(2)	0.039(2)	0.029(2)	–0.007(2)	0.008(2)	–0.005(2)
O(1)	4*e*	0.4693(3)	0.0645(2)	0.1538(2)	0.045(2)	0.071(2)	0.037(2)	–0.030(2)	0.016(2)	–0.007(2)
C(20)	4*e*	0.2267(4)	–0.0066(4)	0.6904(4)	0.043(3)	0.088(4)	0.036(3)	0.022(3)	0.013(2)	0.009(3)
C(26)	4*e*	0.1873(4)	0.1097(3)	0.2589(3)	0.035(3)	0.046(3)	0.038(3)	0.001(2)	0.008(2)	0.005(2)
C(10)	4*e*	0.6039(4)	–0.1882(4)	0.3596(4)	0.051(3)	0.054(3)	0.072(4)	0.011(3)	0.023(3)	0.000(3)
C(22)	4*e*	0.0846(4)	0.0259(4)	0.7748(3)	0.044(3)	0.059(3)	0.035(3)	0.004(3)	0.016(2)	–0.003(2)
C(21)	4*e*	0.1820(4)	–0.0047(4)	0.7737(4)	0.050(3)	0.101(5)	0.033(3)	0.022(3)	0.015(2)	0.015(3)
C(11)	4*e*	0.5801(4)	–0.1916(4)	0.4516(4)	0.045(3)	0.054(3)	0.069(4)	0.007(3)	0.007(3)	0.003(3)
C(9)	4*e*	0.5444(4)	–0.1368(4)	0.2948(4)	0.053(3)	0.062(3)	0.054(3)	0.000(3)	0.033(3)	–0.002(3)
C(30)	4*e*	0.2041(4)	0.1273(4)	0.0997(4)	0.060(4)	0.070(4)	0.030(3)	–0.009(3)	0.009(3)	0.006(3)
C(23)	4*e*	0.0350(5)	0.0295(5)	0.8662(4)	0.061(4)	0.093(5)	0.045(3)	0.006(3)	0.025(3)	–0.003(3)
C(28)	4*e*	0.0731(5)	0.2079(4)	0.1626(4)	0.068(4)	0.068(4)	0.050(3)	0.009(3)	–0.001(3)	0.015(3)
C(27)	4*e*	0.1051(4)	0.1689(4)	0.2507(4)	0.056(3)	0.062(4)	0.046(3)	0.007(3)	0.010(3)	0.002(3)
C(29)	4*e*	0.1238(5)	0.1858(4)	0.0859(4)	0.071(4)	0.074(4)	0.044(3)	–0.001(3)	–0.006(3)	0.016(3)
O(1W)	4*e*	0.9440(3)	0.3116(3)	–0.0848(3)	0.060(3)	0.082(3)	0.074(3)	–0.022(2)	0.019(2)	–0.001(2)
O(2W)	4*e*	0.3921(6)	0.0886(5)	–0.0524(5)	0.178(7)	0.154(6)	0.124(5)	–0.028(5)	–0.007(5)	0.013(5)

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