Crystal structure of bis{[4’-(4-tolyl)-2,2’:6’2’’-terpyridine-κ3N,N’,N’’]-[μ-5-hydroxybenzene-1,3-dicarboxylate-κ2O,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),

\[a = 13.0900(9) \text{ Å}, b = 14.8630(7) \text{ Å}, c = 14.061 \text{ Å},
β = 97.92°, V = 2709.6 \text{ Å}^3, Z = 4, \]

\[R_{gt}(F) = 0.067, \quad wR_{ref}(F^2) = 0.190, \quad T = 293 \text{ K}.\]

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 \times 0.20 \times 0.20 mm |
| Wavelength: Mo Kα radiation (0.71073 Å) |
| μ: 9.60 cm^{-1} |
| Diffractometer, scan mode: Oxford Diffraction Gemini R Ultra, ω |
| 2θ_{max}: 58.34° |
| N(hkl)_{measured}, N(hkl)_{unique}: 12617, 6241 |
| Criterion for I_{obs}, N(hkl)_{ref}: I_{obs} > 2 σ(I_{obs}), 3441 |
| N(\text{param})_{refined}: 370 |
| Programs: PLATON [2], SHELX-97, SHELXL-97 [4] |

* Correspondence author (e-mail: majf247nenu@yahoo.com.cn)
Table 2. Atomic coordinates and displacement parameters (in Å²).

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<th>Atom</th>
<th>Site</th>
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<th>y</th>
<th>z</th>
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Table 2. Atomic coordinates and displacement parameters (in Å²). Continued.

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Acknowledgments. We thank the Science Foundation for Young Teachers of Northeast Normal University (no. 20060304) and the Analysis and Testing Foundation of Northeast Normal University for support.

References