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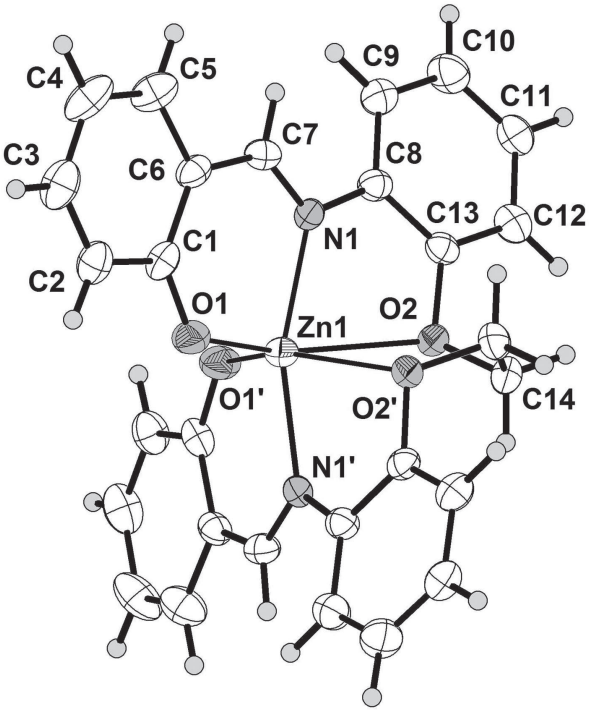
Crystal structure of [2,2′-((((ethane-1,2-diylbis-(oxy-κ^2*O*,*O*′))bis(2,1-phenylene))bis-(azanylylidene-κ^2*N*,*N*′))bis(methanylylidene))-diphenolato-κ^2*O*″,*O*‴]zinc(II), $C_{28}H_{22}N_2O_4Zn$



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Abstract

$C_{28}H_{22}N_2O_4Zn$, tetragonal, $P4_12_12$ (no. 92), $a = 10.986(5)$ Å, $b = 10.986(5)$ Å, $c = 18.187(8)$ Å, $V = 2195(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0440$, $wR_{ref}(F^2) = 0.0942$, $T = 293$ K.

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Table 1: Data collection and handling.

Crystal:	Red, block, size 0.18×0.21×0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.60 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
$2\theta_{max}$:	51.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11931, 2169
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1750
$N(param)_{refined}$:	160
Programs:	SHELX [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	8*b*	0.6547	0.5955	0.4207	0.057
H(3)	8*b*	0.7435	0.7798	0.4271	0.068
H(4)	8*b*	0.8748	0.8451	0.3364	0.080
H(5)	8*b*	0.9246	0.7184	0.2428	0.068
H(7)	8*b*	0.9109	0.5420	0.1779	0.045
H(9)	8*b*	0.9318	0.4850	0.0723	0.057
H(10)	8*b*	1.0112	0.3803	−0.0255	0.063
H(11)	8*b*	1.0103	0.1737	−0.0274	0.053
H(12)	8*b*	0.9150	0.0661	0.0649	0.050
H(14A)	8*b*	0.9216	0.0212	0.1902	0.053
H(14B)	8*b*	0.8094	0.0090	0.2433	0.053

The crystal structure is shown in the figure (′ = 1−*x*, 1−*y*, 0.5−*z*). Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

1,2-Bis(2-(2-oxidobenzylidene)aminophenoxy)ethane (H_2L) was synthesized following a reported synthetic protocol [1, 2], and the other reagents were obtained commercially and used directly without further purification. The title compound was prepared by a layering method. A buffer layer of methanol

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	4*a*	0.69425(5)	0.30575(5)	0.19246	0.0326(2)	0.0326(2)	0.0445(4)	−0.0046(4)	0.0021(2)	0.0021(2)
C(1)	8*b*	0.7344(4)	0.5356(4)	0.3271(2)	0.049(3)	0.031(3)	0.040(2)	0.005(2)	−0.008(2)	0.002(2)
C(2)	8*b*	0.7093(5)	0.6182(4)	0.3842(2)	0.060(3)	0.040(3)	0.044(3)	0.008(3)	−0.002(3)	−0.005(2)
C(3)	8*b*	0.7614(5)	0.7287(5)	0.3878(3)	0.074(4)	0.045(3)	0.050(3)	0.009(3)	−0.016(3)	−0.009(2)
C(4)	8*b*	0.8411(6)	0.7675(5)	0.3341(3)	0.102(5)	0.030(3)	0.068(3)	−0.009(3)	−0.016(3)	−0.003(3)
C(5)	8*b*	0.8696(5)	0.6922(5)	0.2783(3)	0.073(3)	0.041(3)	0.057(3)	−0.013(3)	−0.007(2)	0.001(3)
C(6)	8*b*	0.8186(4)	0.5748(4)	0.2723(2)	0.037(3)	0.029(2)	0.044(2)	−0.005(2)	−0.009(2)	0.003(2)
C(7)	8*b*	0.8567(4)	0.5041(4)	0.2098(2)	0.034(3)	0.038(3)	0.040(2)	−0.007(2)	0.001(2)	0.002(2)
C(8)	8*b*	0.8726(4)	0.3393(4)	0.1291(2)	0.034(2)	0.037(3)	0.035(2)	−0.002(2)	−0.001(2)	0.005(2)
C(9)	8*b*	0.9277(5)	0.4004(5)	0.0715(3)	0.051(3)	0.042(3)	0.050(3)	0.002(2)	0.008(2)	0.005(2)
C(10)	8*b*	0.9762(5)	0.3377(5)	0.0133(3)	0.056(3)	0.063(4)	0.039(3)	0.002(3)	0.008(2)	0.006(2)
C(11)	8*b*	0.9739(4)	0.2149(5)	0.0115(2)	0.037(2)	0.055(3)	0.040(3)	0.003(2)	−0.001(2)	−0.005(2)
C(12)	8*b*	0.9182(4)	0.1507(5)	0.0666(2)	0.035(3)	0.049(3)	0.040(3)	−0.003(2)	−0.005(2)	−0.009(2)
C(13)	8*b*	0.8669(4)	0.2133(4)	0.1246(2)	0.027(2)	0.040(3)	0.035(2)	−0.002(2)	−0.002(2)	0.001(2)
C(14)	8*b*	0.8651(5)	0.0664(4)	0.2207(3)	0.047(3)	0.034(3)	0.053(3)	0.009(2)	0.007(2)	0.003(2)
N(1)	8*b*	0.8252(3)	0.3952(3)	0.1925(2)	0.032(2)	0.027(2)	0.039(2)	0.001(2)	−0.000(2)	0.002(2)
O(1)	8*b*	0.6790(3)	0.4327(3)	0.3254(2)	0.052(2)	0.036(2)	0.057(2)	−0.006(2)	0.014(2)	−0.005(2)
O(2)	8*b*	0.7996(3)	0.1508(2)	0.1773(2)	0.034(2)	0.029(2)	0.047(2)	0.000(2)	0.001(2)	0.002(1)

(3 mL) was carefully layered over the CH_2Cl_2 solution (3 mL) of the protonated ligand (*H_2L*) (45.3 mg, 0.1 mmol). Then a solution of $Zn(CH_3COO)_2 \cdot 2H_2O$ (21.9 mg, 0.1 mmol) in 3 mL methanol was layered over the buffer layer. The resulting mixture was placed in a dark environment. Red block crystals were obtained after five days, in a yield of 58% (based on Zn).

Experimental details

The structure was solved by direct methods [3]. All hydrogen atoms were placed in the calculated positions.

Discussion

The condensation of an amine with an aldehyde or ketone, forming what is called a Schiff base, is one of the oldest reactions in chemistry [4]. Up to now, Schiff bases ligands have been attracted considerable attention of chemists due to the convenience in synthesis, favourable coordination ability and widely applications of its multifunctional complexes including biological [5], analytical [6] and industrial [7] processes. As part of our work, we report a Zn(II) complex of a Schiff base ligand based on N,O-bifunctional coordination group. The Zn(II) is six-coordinated with N_2O_4 binding set in a slightly distorted octahedral geometry. The axial positions are occupied by two N atoms with Zn—N bond lengths of 2.032(4) Å, and the N1—Zn1—N1′ bond angle is 161.72(19)°. The equatorial plane is defined by four oxygen atoms with Zn—O distance of 1.963(3) Å, 2.446(3) Å, and the bond angles around the Zn(II) atom are vary from 68.51(14)° to 155.83(13)°.

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