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The crystal structure of 2,2'-((1*E*,1'*E*)-(naphthalene-2,3 diylbis(azanylylidene))bis(methanylylidene))bis(4-methylphenol), $C_{26}H_{22}N_2O_2$

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Abstract

$C_{26}H_{22}N_2O_2$, monoclinic, $P2_1/n$ (No. 14), $a = 6.2362(2)$ Å, $b = 16.3148(6)$ Å, $c = 20.0521(7)$ Å, $\beta = 94.308(1)^\circ$, $V = 2034.38(12)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0439$, $wR_{ref}(F^2) = 0.1088$, $T = 173$ K.

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The asymmetric unit of 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.

Table 1: Data collection and handling.

Crystal:	Prism, red
Size:	0.43 × 0.23 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffraction, scan mode:	Bruker D8 Venture Photon, ω -scans
θ_{max} , completeness:	27°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	71,804, 4400, 0.026
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3997
$N(param)_{refined}$:	281
Programs:	Bruker programs [1], SHELX [2], Win GX [3], PLATON [4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.04502 (18)	0.56701 (7)	0.84538 (6)	0.0267 (2)
C2	−0.02471 (18)	0.61515 (7)	0.78952 (6)	0.0290 (2)
C3	−0.2084 (2)	0.66347 (8)	0.79207 (7)	0.0360 (3)
H3	−0.256288	0.696117	0.754641	0.043*
C4	−0.3210 (2)	0.66401 (8)	0.84886 (7)	0.0361 (3)
H4	−0.446258	0.697021	0.849589	0.043*
C5	−0.2559 (2)	0.61750 (8)	0.90511 (6)	0.0349 (3)
C6	−0.07337 (19)	0.56957 (8)	0.90212 (6)	0.0326 (3)
H6	−0.026736	0.537274	0.93988	0.039*
C7	0.23024 (18)	0.51351 (7)	0.84407 (6)	0.0279 (2)
H7	0.267685	0.479291	0.881473	0.033*
C8	0.52784 (17)	0.46098 (7)	0.79148 (5)	0.0252 (2)
C9	0.62047 (18)	0.41645 (7)	0.84422 (6)	0.0304 (2)
H9	0.555625	0.416804	0.885606	0.037*
C10	0.80979 (18)	0.37014 (7)	0.83849 (6)	0.0296 (2)
C11	0.9070 (2)	0.32391 (8)	0.89268 (7)	0.0394 (3)
H11	0.843501	0.323416	0.934281	0.047*
C12	1.0910 (2)	0.28010 (8)	0.88566 (8)	0.0412 (3)
H12	1.153802	0.24937	0.922342	0.049*
C13	1.1878 (2)	0.28022 (7)	0.82457 (7)	0.0388 (3)
H13	1.315396	0.249522	0.820161	0.047*
C14	1.09891 (19)	0.32438 (8)	0.77141 (7)	0.0347 (3)
H14	1.165854	0.324205	0.730389	0.042*
C15	0.90793 (17)	0.37043 (7)	0.77686 (6)	0.0277 (2)
C16	0.81133 (18)	0.41672 (7)	0.72296 (6)	0.0286 (2)
H16	0.8763	0.417534	0.681609	0.034*
C17	0.62594 (17)	0.46044 (7)	0.72913 (5)	0.0254 (2)
C18	0.62596 (17)	0.54356 (7)	0.63454 (5)	0.0268 (2)
H18	0.775122	0.552368	0.645323	0.032*
C19	0.52434 (17)	0.57827 (7)	0.57329 (5)	0.0252 (2)
C20	0.30617 (17)	0.56363 (7)	0.55348 (5)	0.0262 (2)
C21	0.21884 (19)	0.59586 (7)	0.49300 (6)	0.0308 (3)
H21	0.0726	0.585612	0.478716	0.037*
C22	0.3443 (2)	0.64256 (7)	0.45392 (6)	0.0319 (3)
H22	0.281255	0.664899	0.41331	0.038*
C23	0.5612 (2)	0.65815 (7)	0.47200 (6)	0.0315 (3)
C24	0.64721 (18)	0.62542 (7)	0.53185 (6)	0.0294 (2)
H24	0.794305	0.635234	0.545277	0.035*
C25	−0.3840 (2)	0.61715 (11)	0.96621 (7)	0.0518 (4)
H25A	−0.328547	0.574343	0.997168	0.078*
H25B	−0.535567	0.606249	0.952662	0.078*
H25C	−0.370861	0.670625	0.9884	0.078*
C26	0.6953 (2)	0.70756 (10)	0.42709 (7)	0.0471 (3)
H26A	0.839967	0.71502	0.448897	0.071*
H26B	0.628259	0.761238	0.418558	0.071*
H26C	0.704933	0.678529	0.384633	0.071*
N1	0.34438 (15)	0.51130 (6)	0.79374 (5)	0.0271 (2)
N2	0.51790 (15)	0.50122 (6)	0.67432 (5)	0.0272 (2)
O1	0.07909 (15)	0.61439 (6)	0.73288 (5)	0.0394 (2)
O2	0.17886 (13)	0.51870 (6)	0.59130 (4)	0.0337 (2)
H1	0.199 (3)	0.5790 (13)	0.7413 (10)	0.073 (6)*
H2	0.266 (3)	0.5026 (12)	0.6290 (10)	0.065 (5)*

Source of materials

All reagents are commercially available and were used without further purification. The Schiff base compound was prepared by the condensation reaction of 2,3-diaminonaphthalene 0.356 g (0.22 mmol) and 2-hydroxy-5-methylbenzaldehyde 0.598 (0.44 mmol) in 10 ml dry methanol. The reaction mixture was heated under microwave irradiation (100 °C, 100 W) for 10 min. On cooling to room temperature an orange powder precipitated out of the reaction mixture. The solid obtained was filtered and washed with methanol to give the target product in 97% yield. Crystals of the title compound were obtained as deep orange needles by slow evaporation of an ethanol solution at room temperature over a period of five days.

Experimental details

Intensity data was determined on a Bruker Venture D8 Photon CMOS diffractometer with graphite-monochromated Mo K_{α1} (λ = 0.71073 Å) radiation at 173 K using an Oxford Cryostream 600 cooler. Data reduction was carried out using the program SAINT+, version 6.02 [1] and empirical absorption corrections were made using SADABS [1]. The structure was solved in the WinGX [2] Suite of programs, using intrinsic phasing through SHELXT [3] and refined using SHELXL-2017 [3]. All C bound hydrogen atoms were placed at idealized positions and refined as riding atoms with isotropic parameters 1.2 times or 1.5 times those of their parent atoms. O-bound hydrogen atoms were located in the difference Fourier map and their coordinates and isotropic displacement parameters refined freely. Diagrams and publication material were generated using ORTEP-3 [2], and PLATON [4].

Discussion

Salen-type ligands are a class of Schiff base compounds with nitrogen and oxygen donor atoms. These tetradentate ligands have received much attention due to their versatility and rich coordination chemistry [5–7]. The modularity of synthesis of these ligands allows for the selective modification of the steric and electronic properties by alteration of the diamine or salicylaldehyde moieties. The salen compounds have potential application in inorganic

biochemistry, catalysis, magnetism and medical imaging [8, 9]. When coordinated to transition metal ions; the complexes exhibit a broad range of biological activities including antifungal, antibacterial, antimalarial, antiproliferative and antiviral properties [5–8]. As a part of our current research focus on Schiff base compounds as ligands, we herein report the molecular structure of a new naphthalene functionalized salen-type ligand.

The title compound crystallizes in the monoclinic space group $P2_1/n$ (no. 14), with four molecules per unit cell. The bond lengths and angles are within the expected ranges and are comparable to related structures [10, 11]. As expected, selected carbon–carbon bond lengths within the aromatic naphthalene rings are shorter than typical aromatic double bonds, showing bond localization. The C8–C9, C16–C17, C11–C12 and C13–C14 bonds have values of 1.374, 1.372, 1.368 and 1.369 Å respectively which is ascribed to the Mills–Nixon effect which accounts for bond lengths changes during benzene ring annulation [12].

The molecule is non-planar, because there is a torsional twist observed about the naphthalene ring, with a (N1–C8–C17–N2) dihedral angle of -7.52° . While it is observed that the polyaromatic naphthalene ring is almost coplanar with one of the phenol rings of the molecule, the second phenol ring is rotated about the N2–C17 bond by *ca.* 27° . The conformation adopted may be due to steric and electronic effects experienced by the phenolic hydroxyl and imine groups of the molecule [13].

In the crystal, hydrogen bonds supported by the amine group can be observed. There are two strong intramolecular hydrogen bonds between the hydroxyl group and nitrogen atom of this compound, with O1–H1...N1 at 2.5984(13) Å and O2–H2...N2 at 2.6065(13) Å. In addition, π -stacking is a prominent feature in the crystal structure of the compound. There is intermolecular aromatic π – π interactions observed between the out-of-plane phenol rings.

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