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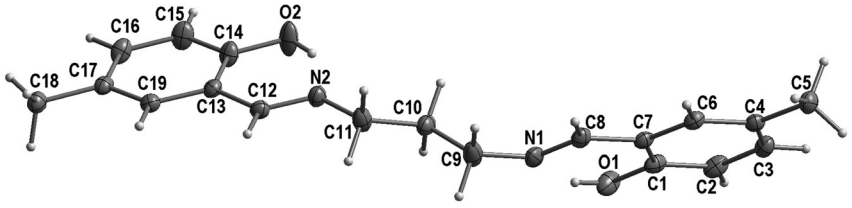
Crystal structure of 2,2′-(propane-1,3-dilylbis(azaneylylidene))bis(methanylylidene) bis(4-methylphenol), $C_{19}H_{22}N_2O_2$



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Abstract

$C_{19}H_{22}N_2O_2$, monoclinic, $P2_1/c$ (no. 14), a = 19.3063(4) Å, b = 5.83200(10) Å, c = 14.7996(3) Å, β = 92.715(1)°, V = 1664.48(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0423, $wR_{ref}(F^2)$ = 0.1102, T = 100(2) K.

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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.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.44 × 0.39 × 0.33 mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ:	0.64 mm^{-1}
Diffractometer, scan mode:	Bruker SMART Apex-II, φ and ω
θ_{max}, completeness:	66.8°, 96%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17,078, 2818, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2575
$N(param)_{refined}$:	213
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

The salicylaldimine compound was synthesized and obtained in good yields from a 2:1 reaction ratio of substituted salicylaldehyde 2-hydroxy-5-methylbenzaldehyde with 1,3-diaminopropane in dry methanol under reflux. The compound was obtained as a yellow solid with yields of 96%.

Experimental details

Single crystals suitable for X-ray diffraction (XRD) studies for the compound were obtained after four weeks by slow diffusion and evaporation of hexane into a concentrated solution of the compound in dichloromethane (DCM). Crystal evaluation and data collection were done on a Bruker Smart APEX2 diffractometer [1] Crystal evaluation and data collection were done on a Bruker SMART 1000

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.81206 (8)	0.6886 (3)	0.30440 (10)	0.0287 (3)
C2	0.87955 (8)	0.7683 (2)	0.29892 (10)	0.0327 (4)
H2A	0.887618	0.914471	0.273133	0.039*
C3	0.93485 (8)	0.6371 (3)	0.33055 (10)	0.0296 (4)
H3	0.980550	0.694931	0.325968	0.036*
C4	0.92561 (7)	0.4208 (2)	0.36927 (9)	0.0256 (3)
C5	0.98661 (8)	0.2776 (3)	0.40219 (11)	0.0326 (4)
H5A	0.971548	0.118938	0.410774	0.049*
H5B	1.005590	0.339013	0.459826	0.049*
H5C	1.022393	0.281639	0.357412	0.049*
C6	0.85811 (7)	0.3428 (2)	0.37430 (9)	0.0248 (3)
H6	0.850499	0.196294	0.400062	0.030*
C7	0.80083 (7)	0.4712 (2)	0.34299 (9)	0.0243 (3)
C8	0.73110 (7)	0.3780 (3)	0.34833 (10)	0.0287 (3)
H8	0.725134	0.231154	0.374611	0.034*
C9	0.60952 (8)	0.3859 (3)	0.32523 (13)	0.0438 (5)
H9A	0.613306	0.249316	0.364905	0.053*
H9B	0.592026	0.334995	0.264545	0.053*
C10	0.55888 (8)	0.5540 (3)	0.36317 (11)	0.0322 (4)
H10A	0.554359	0.688536	0.322515	0.039*
H10B	0.577304	0.608376	0.422955	0.039*
C11	0.48792 (8)	0.4486 (3)	0.37346 (12)	0.0371 (4)
H11A	0.470656	0.384241	0.314726	0.044*
H11B	0.491539	0.321829	0.417903	0.044*
C12	0.37444 (7)	0.5928 (3)	0.38494 (10)	0.0267 (3)
H12	0.359468	0.460389	0.352108	0.032*
C13	0.32289 (7)	0.7585 (2)	0.41226 (9)	0.0242 (3)
C14	0.34274 (8)	0.9615 (3)	0.45794 (11)	0.0319 (4)
C15	0.29194 (9)	1.1161 (3)	0.48124 (13)	0.0381 (4)
H15	0.304830	1.253449	0.512165	0.046*
C16	0.22283 (8)	1.0720 (3)	0.45988 (11)	0.0324 (4)
H16	0.188915	1.180246	0.476460	0.039*
C17	0.20162 (7)	0.8730 (2)	0.41466 (10)	0.0256 (3)
C18	0.12648 (8)	0.8250 (3)	0.39069 (11)	0.0316 (4)
H18A	0.117236	0.849437	0.325719	0.047*
H18B	0.097430	0.928573	0.424766	0.047*
H18C	0.115783	0.665793	0.405955	0.047*
C19	0.25266 (7)	0.7184 (2)	0.39178 (9)	0.0248 (3)
H19	0.239309	0.581013	0.361196	0.030*
N1	0.67781 (7)	0.4894 (2)	0.31845 (9)	0.0340 (3)
N2	0.43897 (6)	0.6204 (2)	0.40381 (9)	0.0291 (3)
O1	0.75882 (6)	0.8209 (2)	0.27355 (8)	0.0400 (3)
H1	0.721521	0.747671	0.276516	0.060*
O2	0.41009 (6)	1.0089 (2)	0.47933 (10)	0.0473 (4)
H2	0.435110	0.903102	0.460415	0.071*

CCD diffractometer with Mo Kα radiation (λ = 0.71073 Å) equipped with an Oxford Cryostream low temperature apparatus operating at 100(1) K. The structure was solved by direct method using the SHELXS [2] program and refined with SHELXL [3].

All hydrogen atoms were placed in idealized positions and refined in riding models with U_{iso} assigned the values of 1.2 times or 1.5 times those of their parent atoms and the distances of C–H were constrained to 0.95 Å for all the aromatic H atoms, 0.98 Å for the CH_3 protons and 0.9 for CH_2 protons or 0.84 Å for the hydroxy group H atoms, respectively. The idealized tetrahedral OH refined as rotating group. The visual crystal structure information was performed using Diamond [4].

Comment

N,O-salicylaldimines are Schiff base ligands with the ability to coordinate transition metal ions *via* the hard nitrogen and oxygen donor atoms. This leads to a stabilization of the metal complexes against reduction and eventually give good thermal stability [5]. It is also easy to manipulate their steric and electronic properties. The early transition metal complexes have been shown to exhibit high oxophilic nature [6, 7]. Despite this shortcoming, a lot of research has been conducted on salicylaldimine complexes and their application as catalysts in polymerization reactions. For instance, group four metal-complexes of titanium and zirconium have been synthesized and tested, which showed significant (co) polymerization of ethylene [8–10]. Their salicylaldimine complexes have also been shown to exhibit stability, catalytic activity and greater tolerance for polar monomers in olefin polymerization [11].

The asymmetric unit of the title compound contains one salicylaldimine molecule. In the structure, the 2-(iminomethyl)-4-methylphenol moieties are on either sides of the propyl linker and enclose a dihedral angle of 52.40(3) comparable to that of in the structure of 4,4′-Dichloro-2,2′-[(1*E*,1′*E*)-propane-1,3-diylbis(nitrilomethylidyne)]diphenol [12].

In the crystal, this compound displays two intramolecular hydrogen bonds O–H···N with an N···O distance of 2.593(2) Å. The bond distances and angles are comparable to those of related compounds [13–16].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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