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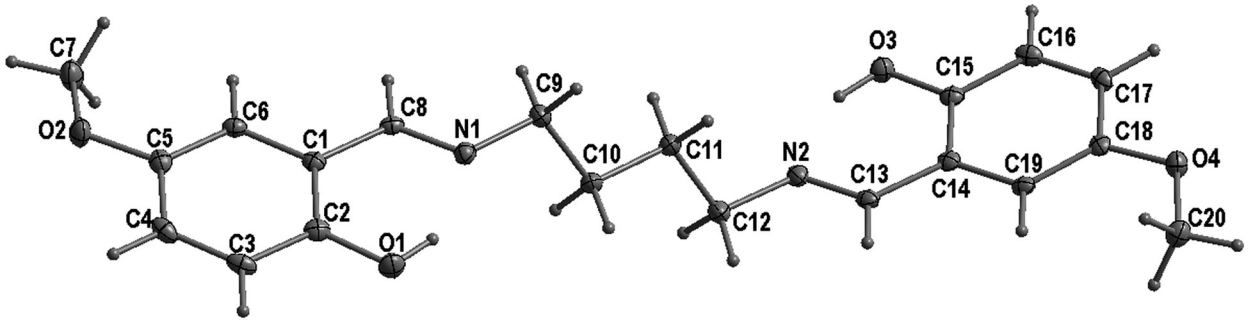
Crystal structure of 2,2′-(butane-1,4-diylbis(azanylylidene))bis(methanylylidene)) bis(4-methoxyphenol), $C_{20}H_{24}N_2O_4$



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Abstract

$C_{20}H_{24}N_2O_4$, monoclinic, $P2_1/n$ (no. 14), a = 11.0474(3) Å, b = 6.0853(2) Å, c = 13.9881(4) Å, β = 104.271(2)°, V = 911.36(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0290, $wR_{ref}(F^2)$ = 0.0728, T = 100(2) K.

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The molecular structure is shown in 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 parallelepiped
Size:	0.35 × 0.16 × 0.09 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.74 mm^{-1}
Diffractometer, scan mode:	Bruker SMART Apex-II, φ and ω
θ_{max}, completeness:	66.6°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9785, 2698, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2539
$N(param)_{refined}$:	241
Programs:	Bruker [1], SHELX [2], Diamond [3]

Source of material

The title compound was prepared from a 2:1 reaction ratio of substituted salicylaldehyde 2-hydroxy-5-methoxybenzaldehyde with 1,4-diaminobutane in dry ethanol under reflux for 1 h. The title compound was obtained as a yellow solid with yield = 96%. **Elemental analysis** for $C_{20}H_{24}N_2O_4$ (256.42 g/mol), Calculated: C, 49.77%; H, 5.64; N, 5.81; Found: C, 50.00%; H, 5.72; N, 5.64. Single crystals suitable for X-ray diffraction (XRD) studies were obtained after four weeks by slow diffusion and evaporation of hexane into a concentrated solution of the compound in DCM.

Experimental details

Crystal evaluation and data collection were done on a Bruker SMART 1000 CCD diffractometer with Mo Kα radiation (λ = 0.71073 Å) equipped with an Oxford Cryostream

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.64008 (17)	0.9164 (4)	0.13709 (13)	0.0162 (4)
C2	0.57652 (17)	1.1180 (4)	0.12601 (13)	0.0184 (5)
C3	0.59650 (18)	1.2685 (4)	0.05643 (14)	0.0207 (4)
H3A	0.553564	1.405082	0.048317	0.025*
C4	0.67834 (18)	1.2199 (4)	−0.00069 (14)	0.0221 (5)
H4	0.690968	1.323514	−0.048090	0.027*
C5	0.74297 (18)	1.0207 (4)	0.01003 (14)	0.0199 (5)
C6	0.72360 (17)	0.8693 (4)	0.07859 (14)	0.0181 (5)
H6	0.766908	0.733040	0.086129	0.022*
C7	0.8705 (2)	0.7766 (4)	−0.05657 (15)	0.0302 (5)
H7A	0.920348	0.730498	0.008381	0.045*
H7B	0.923010	0.776059	−0.103758	0.045*
H7C	0.800605	0.674659	−0.078953	0.045*
C8	0.61923 (16)	0.7529 (4)	0.20764 (13)	0.0160 (4)
H8	0.660896	0.615342	0.212042	0.019*
C9	0.52678 (18)	0.6169 (4)	0.33066 (14)	0.0201 (5)
H9A	0.555757	0.668093	0.399719	0.024*
H9B	0.577009	0.486794	0.322409	0.024*
C10	0.39022 (17)	0.5529 (4)	0.31019 (14)	0.0194 (5)
H10A	0.361685	0.500408	0.241300	0.023*
H10B	0.340043	0.683757	0.317511	0.023*
C11	0.36864 (17)	0.3739 (4)	0.37980 (14)	0.0199 (5)
H11A	0.393288	0.428978	0.448467	0.024*
H11B	0.421949	0.245726	0.374869	0.024*
C12	0.23267 (17)	0.3020 (4)	0.35600 (14)	0.0230 (5)
H12A	0.178671	0.431631	0.356440	0.028*
H12B	0.209525	0.236698	0.289134	0.028*
C13	0.15127 (16)	0.2041 (4)	0.49011 (14)	0.0182 (5)
H13	0.115172	0.346668	0.483727	0.022*
C14	0.13610 (16)	0.0626 (4)	0.57031 (13)	0.0166 (4)
C15	0.18869 (17)	−0.1481 (4)	0.58234 (14)	0.0179 (5)
C16	0.18253 (17)	−0.2721 (4)	0.66495 (13)	0.0192 (5)
H16	0.218728	−0.414604	0.673975	0.023*
C17	0.12403 (17)	−0.1884 (4)	0.73355 (14)	0.0192 (5)
H17	0.121833	−0.272869	0.790210	0.023*
C18	0.06823 (17)	0.0180 (4)	0.72087 (14)	0.0179 (4)
C19	0.07448 (16)	0.1445 (4)	0.63968 (13)	0.0171 (4)
H19	0.037210	0.286097	0.630955	0.021*
C20	−0.05252 (18)	0.2871 (4)	0.78128 (15)	0.0239 (5)
H20A	0.009102	0.405347	0.787026	0.036*
H20B	−0.098280	0.304664	0.832568	0.036*
H20C	−0.111223	0.293737	0.716170	0.036*
N1	0.54579 (14)	0.7912 (3)	0.26385 (11)	0.0188 (4)
N2	0.21211 (14)	0.1411 (3)	0.42787 (11)	0.0201 (4)
O1	0.49423 (13)	1.1699 (3)	0.18013 (10)	0.0244 (4)
H1	0.490835	1.066750	0.219273	0.037*
O2	0.82349 (13)	0.9927 (3)	−0.04984 (10)	0.0266 (4)
O3	0.24919 (12)	−0.2351 (3)	0.51775 (9)	0.0221 (3)
H3	0.250825	−0.142609	0.473486	0.033*
O4	0.00990 (12)	0.0797 (3)	0.79304 (9)	0.0215 (4)

low temperature apparatus operating at 100(1) K. The structure was solved by direct method using the SHELXS [2] program and refined with SHELXL [2]. 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 and 0.99 for CH_2 protons or 0.84 Å for the hydroxy group H atoms, respectively. The idealised tetrahedral OH refined as rotating group. The visual crystal structure information was performed using Diamond [3].

Comment

The salicylaldimine ligands have drawn much interest from many researchers because they can easily bind to many transition metals like nickel, palladium, copper, manganese, iron, zinc and ruthenium [4–9]. These complexes have found a wide range of applications in catalysis like olefin polymerization and (co)polymerization of substituted norbornene, ethylene polymerization and (co) polymerization [10–13], ring-opening and ring-closing metathesis polymerization [9, 14–16].

The asymmetric unit of the title compound contains one salicylaldimine molecule. In the structure, the aminomethyl methoxyphenol moieties are on either sides of the butyl linker and are almost co-planar with a dihedral angle of 12.78(3)°.

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