

Ming-Hu Wu,^{a*} Wen-Ju Liu,^a
Wei-Dong Zou^b and Hai-Yan
Wang^a^aDepartment of Chemistry and Life Science,
Xianning College, Xianning 437005, People's
Republic of China, and ^bDepartment of Physics,
Xianning College, Xianning 437005, People's
Republic of ChinaCorrespondence e-mail:
minghuwu@hotmail.com

Key indicators

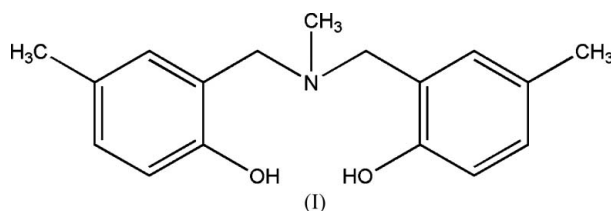
Single-crystal X-ray study
 $T = 292\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.055
 wR factor = 0.159
Data-to-parameter ratio = 18.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4,4'-Dimethyl-2,2'-(*N*-methyliminodimethylene)-
diphenol

In the title compound, $\text{C}_{17}\text{H}_{21}\text{NO}_2$, the dihedral angle between the benzene rings is $49.8(3)^\circ$. In the crystal structure, the molecules form layers parallel to the ac plane which are stacked along the b axis and which are stabilized by $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.

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Comment

The chemistry of Mannich bases has been the subject of investigations by an ever increasing number of researchers because of their wide applications (Arend *et al.*, 1998). However, the synthesis of an *N,N*-bis(5-substituent-2-hydroxybenzyl)amine from a *p*-substituted phenol by the Mannich reaction has not been reported. We have recently synthesized the title compound, (I), by reaction of 4-methylphenol, formaldehyde and methylamine, and its crystal structure is reported here.



In the molecule (Fig. 1), the two benzene rings are inclined with respect to each other with a dihedral angle of $49.8(3)^\circ$. The $\text{C}6-\text{C}1-\text{C}8-\text{N}1$ and $\text{N}1-\text{C}10-\text{C}11-\text{C}16$ torsion angles are $-45.55(19)$ and $66.4(18)^\circ$, respectively, showing that the aminomethyl group is not coplanar with either of the benzene rings.

The structure consists of layers of molecules, stacked along the b axis, stabilized by two intermolecular hydrogen-bonding interactions (Fig. 2 and Table 2).

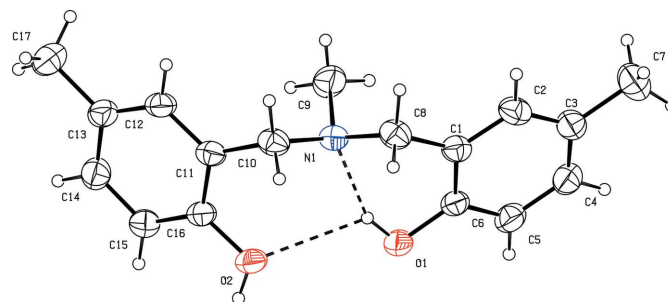


Figure 1

View of the molecular structure of (I), showing the atom-labeling scheme and displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

Experimental

Formaldehyde (8 ml, 40%, 0.1 mol) was added slowly with stirring to a mixture of methanol (35 ml), methylamine (6.5 ml, 25–30%, 0.05 mol) and 4-methylphenol (10.9 g, 0.1 mol) over a period of 3 h. The mixture was stirred for an additional 60 h at room temperature. The resulting bright-yellow solid was filtered off and washed with methanol. The solid residue was recrystallized twice from ethyl acetate–petroleum ether (1:5 v/v) to give colorless crystals of (I) in 63% yield (m.p. 325 K), which were suitable for X-ray analysis. MS (EI, 70 eV) *m/z*(%): 272 (100).

Crystal data

$C_{17}H_{21}NO_2$ $V = 775.2 (2) \text{ \AA}^3$
 $M_r = 271.35$ $Z = 2$
 Triclinic, $P\bar{1}$ $D_x = 1.163 \text{ Mg m}^{-3}$
 $a = 5.8024 (9) \text{ \AA}$ Mo $K\alpha$ radiation $\mu = 0.08 \text{ mm}^{-1}$
 $b = 10.8867 (17) \text{ \AA}$ $T = 292 (2) \text{ K}$
 $c = 12.5346 (19) \text{ \AA}$ Block, colorless
 $\alpha = 93.980 (3)^\circ$ $0.30 \times 0.20 \times 0.20 \text{ mm}$
 $\beta = 90.847 (3)^\circ$
 $\gamma = 100.955 (3)^\circ$

Data collection

Bruker SMART CCD area-detector 7457 measured reflections
 diffractometer 3416 independent reflections
 φ and ω scans 2423 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.057$
 (SADABS; Sheldrick, 2001) $\theta_{max} = 27.3^\circ$
 $T_{min} = 0.978, T_{max} = 0.985$

Refinement

Refinement on F^2 H atoms treated by a mixture of
 $R[F^2 > 2\sigma(F^2)] = 0.055$ independent and constrained
 $wR(F^2) = 0.159$ refinement
 $S = 1.02$ $w = 1/[\sigma^2(F_o^2) + (0.0876P)^2]$
 3416 reflections where $P = (F_o^2 + 2F_c^2)/3$
 190 parameters $(\Delta/\sigma)_{max} = 0.002$
 $\Delta\rho_{max} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.22 \text{ e \AA}^{-3}$

Table 1

Selected torsion angles ($^\circ$).

C2–C1–C8–N1	135.25 (15)	N1–C10–C11–C12	–112.58 (15)
C6–C1–C8–N1	–45.55 (19)	N1–C10–C11–C16	66.44 (18)

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1–H1 \cdots N1	0.829 (9)	1.898 (13)	2.6594 (15)	152 (2)
O1–H1 \cdots O2	0.829 (9)	2.474 (19)	2.9910 (17)	121.4 (18)
C8–H8A \cdots O2 ⁱ	0.97	2.57	3.540 (2)	173
O2–H2A \cdots O1 ⁱⁱ	0.834 (9)	1.881 (10)	2.7065 (15)	170 (2)

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x, -y + 1, -z + 1$.

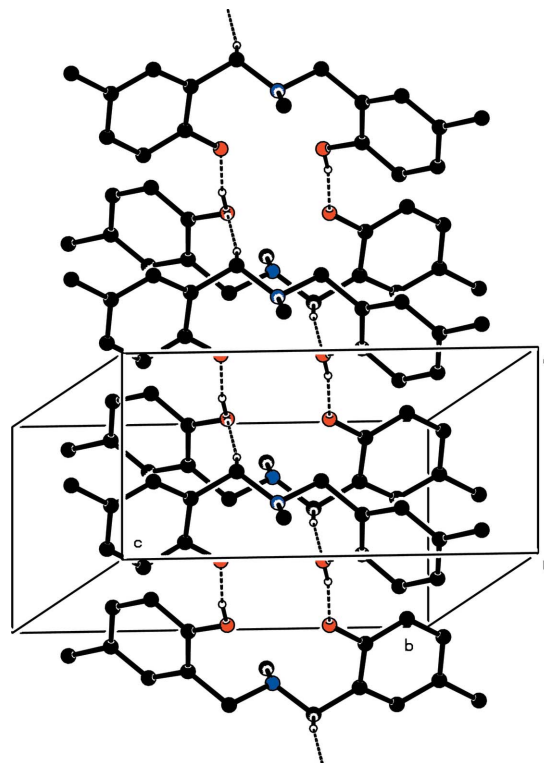


Figure 2

The crystal packing of (I), viewed down the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

All C-bound H atoms were placed in calculated positions and treated as riding atoms ($C-H = 0.93-0.97 \text{ \AA}$), with $U_{iso}(H)$ set equal to 1.5 (methyl H atoms) or 1.2 (other H) times $U_{eq}(C)$. The positions of O-bound H atoms were refined freely [$U_{iso}(H) = 1.5U_{eq}(O)$].

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

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