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4-(*o*-Tolylamino)benzaldehyde

Li-Ying Wang,[a] Yong-Sheng Xie,[b] Ren-Min Wu[a] and Hua Zuo[a]*

[a]College of Pharmaceutical Sciences, Southwest University, Chongqing 400715, People's Republic of China, and [b]School of Chemical and Environmental Engineering, Chongqing Three Gorges University, Chongqing 404100, People's Republic of China
Correspondence e-mail: zuohua@swu.edu.cn

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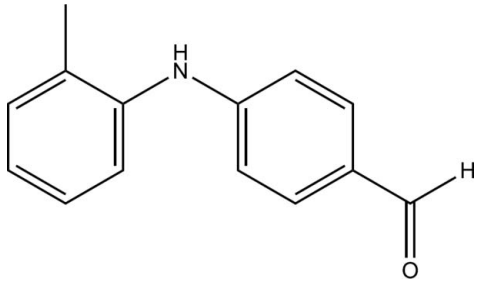
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.005 Å; R factor = 0.050; wR factor = 0.157; data-to-parameter ratio = 9.1.

In the title compound, $C_{14}H_{13}NO$, the dihedral angle between the aromatic rings is 49.64 (18)°. The crystal structure is stabilized by N—H···O, C—H···O and C—H···π hydrogen bonds.

Related literature

For applications and bioactivity of diarylamines, see: Ohta *et al.* (2008); Li *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{13}NO$
M_r = 211.25
Orthorhombic, $Pca2_1$
a = 14.193 (10) Å
b = 10.699 (10) Å
c = 7.677 (6) Å
V = 1165.9 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 273 K
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
6127 measured reflections
1397 independent reflections
1140 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.157
S = 1.06
1397 reflections
153 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C8–C13 tolyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.79 (4)	2.32 (4)	3.099 (5)	171 (3)
C14—H14A···O1^{i}	0.96	2.48	3.334 (6)	148
C9—H9···*Cg*1^{ii}	0.93	2.95	3.603 (5)	128

Symmetry codes: (i) $x-\frac{1}{2}, -y+1, z$; (ii) $-x+2, -y+2, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2297).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

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4-(*o*-Tolylamino)benzaldehyde

Li-Ying Wang, Yong-Sheng Xie, Ren-Min Wu and Hua Zuo

S1. Comment

Diarylamines represent an important class of compounds due to their wide applications and special pharmacological activities (Ohta *et al.* 2008; Li *et al.* (2008).). We report here the synthesis and the crystal structure of the title compound, $C_{14}H_{13}NO$, which consists of benzaldehyde and tolyl groups attached at the terminal nitrogen atoms (Fig. 1). The dihedral angle between the aromatic rings is 49.64 (18)°. The N1, C14 and H14A atoms are coplanar with the phenyl ring C8 to C13, with deviations of -0.053 (3) Å, -0.076 (4) Å, and -0.07 (1) Å from the ring plane, respectively. The non-planar conformation of the title molecule is not only due to the intramolecular C14-H14A···N1 hydrogen bond, but also owing to the repulsion of H14A and H1 together with packing effects and intermolecular interactions (Fig. 1 and Table 1).In the crystal, zigzag chains are formed along *a* through the intermolecular N—H···O and C—H···O hydrogen bonds (Fig. 2 and Table 1). The molecules are also stabilized by weak C—H···π interactions (Table 1: $Cg1^{ii}$ is the centroid of the tolyl ring C8 - C13).

S2. Experimental

To a magnetically stirred solution of *o*-toluidine (1.0 mmol) and Cs_2CO_3 (3.2 mmol) in dry DMF cooled by ice bath were added chloroacetyl chloride (1.2 mmol) and 4-hydroxybenzaldehyde (1.0 mmol). The reaction mixture was then stirred for 30 min at room temperature and placed into a microwave oven (600 W, 423K) and irradiated for 35 min. The solvent was removed under vacuum and water (20 ml) was added into the residue. The mixture was then extracted by ethyl acetate (4 *x* 30 ml). The combined organic layers were dried over anhydrous $MgSO_4$ and evaporated under vacuum to give the crude product, which was purified by column chromatography on silica gel using ethyl acetate/petroleum ether (yield 89%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid dissolved in ethyl acetate/petroleum ether at room temperature for 4 days.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aryl and 0.96 Å for methyl H atoms, and with U_{iso}(H) =1.2U_{eq}(C) for aryl H atoms, and 1.5U_{eq}(C) for methyl H atoms. The C1- and N1-bound H-atoms were located in a difference Fourier map, the U_{iso} values were freely refined. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

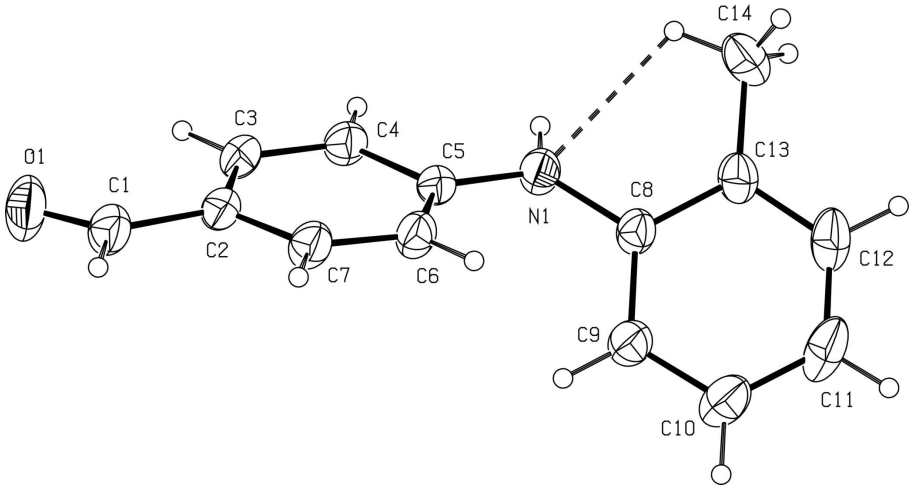

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

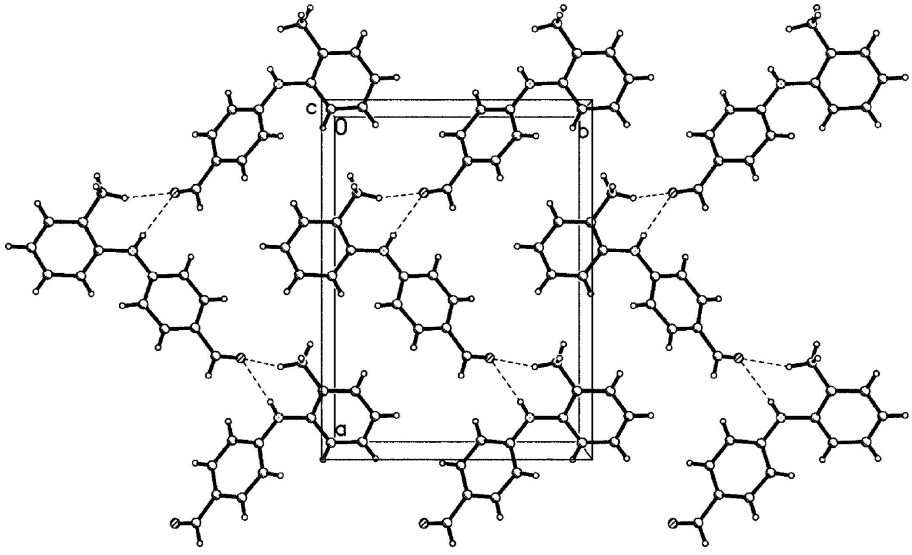

Figure 2

A section of the crystal packing of the title compound. Intermolecular hydrogen bonds are shown by dashed lines.

4-(*o*-Tolylamino)benzaldehyde

Crystal data

$C_{14}H_{13}NO$
$M_r = 211.25$
Orthorhombic, $Pca2_1$
$a = 14.193\ (10)$ Å
$b = 10.699\ (10)$ Å
$c = 7.677\ (6)$ Å
$V = 1165.9\ (16)$ Å^3
$Z = 4$
$F(000) = 448$
$D_x = 1.204$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2572 reflections
$\theta = 2.4$–$26.6°$
$\mu = 0.08$ mm^{-1}
$T = 273$ K
Plate, brown
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
6127 measured reflections
1397 independent reflections
1140 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.4°$, $\theta_{min} = 1.9°$
$h = -18 \rightarrow 14$
$k = -13 \rightarrow 13$
$l = -6 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.157$
$S = 1.06$
1397 reflections
153 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1027P)^2 + 0.0736P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C6	1.07328 (19)	0.7362 (2)	0.3779 (5)	0.0546 (7)
H6	1.0775	0.8092	0.4432	0.066*
C5	0.99125 (16)	0.7114 (2)	0.2798 (4)	0.0476 (6)
N1	0.91447 (16)	0.7907 (2)	0.2760 (4)	0.0567 (7)
C8	0.91320 (18)	0.9212 (2)	0.3158 (4)	0.0496 (7)
C4	0.98692 (19)	0.5966 (2)	0.1871 (5)	0.0534 (7)
H4	0.9324	0.5758	0.1265	0.064*
C3	1.0628 (2)	0.5154 (2)	0.1858 (5)	0.0548 (7)
H3	1.0591	0.4418	0.1217	0.066*
C2	1.14506 (19)	0.5424 (2)	0.2797 (5)	0.0531 (7)
C13	0.83034 (19)	0.9730 (2)	0.3886 (5)	0.0557 (7)
O1	1.23816 (19)	0.36802 (18)	0.1876 (6)	0.0897 (10)
C1	1.2296 (2)	0.4612 (3)	0.2747 (6)	0.0660 (9)
C7	1.1475 (2)	0.6520 (2)	0.3770 (5)	0.0581 (8)
H7	1.2006	0.6694	0.4437	0.070*
C9	0.9897 (2)	0.9981 (3)	0.2718 (5)	0.0577 (7)

H9	1.0431	0.9638	0.2204	0.069*
C14	0.7450 (3)	0.8922 (3)	0.4262 (8)	0.0821 (11)
H14A	0.7589	0.8069	0.3968	0.123*
H14B	0.6926	0.9208	0.3580	0.123*
H14C	0.7295	0.8976	0.5477	0.123*
C12	0.8298 (3)	1.1013 (3)	0.4195 (6)	0.0742 (10)
H12	0.7765	1.1371	0.4693	0.089*
C10	0.9854 (2)	1.1256 (3)	0.3053 (7)	0.0728 (11)
H10	1.0365	1.1764	0.2781	0.087*
C11	0.9052 (3)	1.1773 (3)	0.3790 (7)	0.0814 (12)
H11	0.9023	1.2627	0.4011	0.098*
H1A	1.286 (2)	0.483 (3)	0.339 (5)	0.063 (9)*
H1	0.869 (3)	0.755 (3)	0.244 (5)	0.070 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0502 (14)	0.0407 (13)	0.0730 (19)	0.0008 (10)	−0.0053 (14)	−0.0097 (14)
C5	0.0395 (11)	0.0388 (12)	0.0645 (17)	−0.0005 (9)	0.0042 (12)	−0.0001 (14)
N1	0.0375 (10)	0.0445 (11)	0.0881 (19)	−0.0002 (9)	−0.0052 (12)	−0.0093 (14)
C8	0.0417 (12)	0.0427 (13)	0.0645 (18)	0.0050 (10)	−0.0062 (12)	−0.0031 (13)
C4	0.0441 (12)	0.0446 (12)	0.0716 (19)	−0.0062 (10)	−0.0046 (14)	−0.0073 (15)
C3	0.0586 (15)	0.0355 (11)	0.0704 (18)	−0.0036 (11)	0.0043 (15)	−0.0069 (15)
C2	0.0487 (13)	0.0375 (11)	0.0730 (19)	0.0034 (10)	0.0040 (14)	0.0039 (14)
C13	0.0469 (14)	0.0554 (15)	0.0646 (18)	0.0144 (11)	−0.0045 (14)	0.0001 (15)
O1	0.0794 (16)	0.0617 (12)	0.128 (3)	0.0276 (12)	−0.0038 (18)	−0.0164 (18)
C1	0.0559 (17)	0.0496 (15)	0.093 (3)	0.0101 (13)	−0.0029 (18)	0.0025 (19)
C7	0.0478 (14)	0.0446 (13)	0.082 (2)	0.0010 (11)	−0.0105 (15)	−0.0024 (15)
C9	0.0470 (13)	0.0486 (13)	0.077 (2)	0.0005 (10)	−0.0054 (15)	0.0012 (16)
C14	0.0541 (16)	0.090 (2)	0.102 (3)	0.0158 (18)	0.0239 (18)	0.008 (2)
C12	0.0644 (19)	0.0643 (18)	0.094 (3)	0.0275 (15)	−0.0133 (19)	−0.015 (2)
C10	0.0631 (17)	0.0484 (15)	0.107 (3)	−0.0040 (13)	−0.022 (2)	0.006 (2)
C11	0.083 (2)	0.0442 (15)	0.117 (3)	0.0170 (17)	−0.035 (2)	−0.010 (2)

Geometric parameters (Å, º)

C6—C7	1.386 (4)	C13—C12	1.393 (4)
C6—C5	1.412 (4)	C13—C14	1.516 (5)
C6—H6	0.9300	O1—C1	1.207 (5)
C5—N1	1.381 (3)	C1—H1A	0.97 (4)
C5—C4	1.421 (4)	C7—H7	0.9300
N1—C8	1.429 (4)	C9—C10	1.390 (5)
N1—H1	0.80 (4)	C9—H9	0.9300
C8—C9	1.403 (4)	C14—H14A	0.9600
C8—C13	1.415 (4)	C14—H14B	0.9600
C4—C3	1.383 (4)	C14—H14C	0.9600
C4—H4	0.9300	C12—C11	1.381 (6)
C3—C2	1.403 (4)	C12—H12	0.9300

C3—H3	0.9300	C10—C11	1.386 (6)
C2—C7	1.391 (4)	C10—H10	0.9300
C2—C1	1.481 (4)	C11—H11	0.9300
C7—C6—C5	120.1 (2)	O1—C1—C2	125.5 (4)
C7—C6—H6	119.9	O1—C1—H1A	113.4 (19)
C5—C6—H6	119.9	C2—C1—H1A	121.0 (19)
N1—C5—C6	123.1 (2)	C6—C7—C2	122.2 (3)
N1—C5—C4	119.1 (2)	C6—C7—H7	118.9
C6—C5—C4	117.7 (2)	C2—C7—H7	118.9
C5—N1—C8	127.2 (2)	C10—C9—C8	119.8 (3)
C5—N1—H1	111 (2)	C10—C9—H9	120.1
C8—N1—H1	122 (2)	C8—C9—H9	120.1
C9—C8—C13	120.6 (2)	C13—C14—H14A	109.5
C9—C8—N1	120.7 (2)	C13—C14—H14B	109.5
C13—C8—N1	118.5 (2)	H14A—C14—H14B	109.5
C3—C4—C5	120.8 (3)	C13—C14—H14C	109.5
C3—C4—H4	119.6	H14A—C14—H14C	109.5
C5—C4—H4	119.6	H14B—C14—H14C	109.5
C4—C3—C2	121.0 (3)	C11—C12—C13	122.5 (3)
C4—C3—H3	119.5	C11—C12—H12	118.7
C2—C3—H3	119.5	C13—C12—H12	118.7
C7—C2—C3	118.0 (2)	C11—C10—C9	120.2 (3)
C7—C2—C1	119.3 (3)	C11—C10—H10	119.9
C3—C2—C1	122.7 (3)	C9—C10—H10	119.9
C12—C13—C8	117.3 (3)	C12—C11—C10	119.6 (3)
C12—C13—C14	121.7 (3)	C12—C11—H11	120.2
C8—C13—C14	121.1 (2)	C10—C11—H11	120.2

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C8–C13 tolyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···N1	0.96	2.40	2.880 (6)	110
N1—H1···O1^{i}	0.79 (4)	2.32 (4)	3.099 (5)	171 (3)
C14—H14*A*···O1^{i}	0.96	2.48	3.334 (6)	148
C9—H9···*Cg*1^{ii}	0.93	2.95	3.603 (5)	128

Symmetry codes: (i) *x*−1/2, −*y*+1, *z*; (ii) −*x*+2, −*y*+2, *z*−1/2.