Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Chloro-*N*-*m*-tolylbenzamide

Aamer Saeed,[a]* Madiha Irfan[a] and Michael Bolte[b]

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and [b]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: aamersaeed@yahoo.com

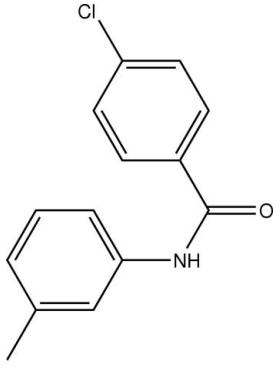
Received 7 May 2009; accepted 14 May 2009

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.066; wR factor = 0.178; data-to-parameter ratio = 13.7.

In the title compound, $C_{14}H_{12}ClNO$, the dihedral angle between the two aromatic rings is 11.29 (15)°. The crystal packing is stabilized by N—H···O hydrogen bonds linking the molecules into chains running along the *c* axis.

Related literature

For the biological activity of *N*-substituted benzamides and benzanilide derivatives, see Calderone *et al.* (2006); Beccalli *et al.* (2005); Yoo *et al.* (2005); Vega-Noverola *et al.* (1989); Olsson *et al.* (2002); Lindgren *et al.* (2001); Zhichkin *et al.* (2007). For related structures see: Saeed *et al.* (2008); Chopra & Guru Row (2008); Donnelly *et al.* (2008)



Experimental

Crystal data

$C_{14}H_{12}ClNO$
M_r = 245.70
Monoclinic, $P2_1/c$
a = 13.9721 (14) Å
b = 10.1922 (6) Å
c = 9.0154 (8) Å
β = 105.415 (7)°
V = 1237.67 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 173 K
0.26 × 0.24 × 0.23 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2009; Blessing, 1995)
T_{min} = 0.928, T_{max} = 0.936
9469 measured reflections
2193 independent reflections
1787 reflections with $I > 2\sigma(I)$
R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.178
S = 1.04
2193 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88 (3)	1.99 (3)	2.854 (3)	166 (3)

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2096).

References

Beccalli, E. M., Broggini, G., Paladinoa, G. & Zonia, C. (2005). *Tetrahedron*, **61**, 61–68.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Calderone, V., Fiamingo, F. L., Giorgi, I., Leonardi, M., Livi, O., Martelli, A. & Martinotti, E. (2006). *Eur. J. Med. Chem.* **41**, 761–767.
Chopra, D. & Guru Row, T. N. (2008). *CrystEngComm*, **10**, 54–67.
Donnelly, K., Gallagher, J. F. & Lough, A. J. (2008). *Acta Cryst.* C**64**, o335–o340.
Lindgren, H., Pero, R. W., Ivars, F. & Leanderson, T. (2001). *Mol. Immunol.* **38**, 267–277.
Olsson, A. R., Lindgren, H., Pero, R. W. & Leanderson, T. (2002). *Br. J. Cancer*, **86**, 971–978.
Saeed, A., Khera, R. A., Abbas, N., Simpson, J. & Stanley, R. G. (2008). *Acta Cryst.* E**64**, o2322–o2323.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Vega-Noverola, A. P., Soto, J. M., Noguera, F. P., Mauri, J. M. & Spickett, G. W. R. (1989). US Patent No. 4 877 780.
Yoo, C. L., Fettinger, J. C. & Kurth, M. J. (2005). *J. Org. Chem.* **70**, 6941–6943.
Zhichkin, P., Kesicki, E., Treiberg, J., Bourdon, L., Ronsheim, M., Ooi, H. C., White, S., Judkins, A. & Fairfax, D. (2007). *Org. Lett.* **9**, 1415–1418.

supporting information

Acta Cryst. (2009). E**65**, o1334 [doi:10.1107/S1600536809018236]

4-Chloro-*N*-*m*-tolylbenzamide

Aamer Saeed, Madiha Irfan and Michael Bolte

S1. Comment

The benzanilide core is present in compounds with such a wide range of biological activities that it has been called a privileged structure. N-Substituted benzamides are well known anticancer compounds and the mechanism of action for N-substituted benzamide-induced apoptosis has been studied, using declopramide as a lead compound (Olsson *et al.*, 2002). N-Substituted benzamides inhibit the activity of nuclear factor- B and nuclear factor of activated T cells activity while inducing activator protein 1 activity in T lymphocytes (Lindgren *et al.*, 2001). Various N-substituted benzamides exhibit potent antiemetic activity (Vega-Noverola *et al.*, 1989), while heterocyclic analogs of benzanilide derivatives are potassium channel activators (Calderone *et al.*, 2006). *o*-Aryloxylation of N-substituted benzamides induced by the copper(II)/trimethylamine N-oxide system has been studied. *N*-Alkylated 2-nitrobenzamides are intermediates in the synthesis of dibenzo[b,e][1,4]diazepines (Zhichkin *et al.*, 2007) and *N*-Acyl-2-nitrobenzamides are precursors of 2,3-disubstitued 3*H*-quinazoline-4-ones (Beccalli *et al.*, 2005). A one-pot conversion of 2-nitro-n-arylbenzamides to 2,3-dihydro-1*H*-quinazoline-4-ones has also been reported (Yoo *et al.*, 2005). As part of our work on the structure of benzanilides and related compounds, we report here the structure of the title 4-chlorobenzamide derivative, I, Fig 1.

The dihedral angle between the two aromatic rings is 11.29 (15) °. The crystal packing is stabilized by N—H···O hydrogen bonds linking the molecules to chains running along the *c* axis.

S2. Experimental

4-Chlorobenzoyl chloride (5.4 mmol) in $CHCl_3$ was treated with 3-methylaniline (21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with $CHCl_3$ and washed consecutively with aq 1 *M* HCl and saturated aq $NaHCO_3$. The organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Crystallization of the residue from $CHCl_3$ afforded the title compound (81%) as colourless blocks. Anal. calcd. for $C_{14}H_{12}Cl_{NO}1$: C 68.44, H 4.92, N 5.70%; found: C 68.39, H 4.90, N 5.67%

S3. Refinement

H atoms were located in a difference map but those bonded to C were geometrically positioned and refined using a riding model with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C) or U(H) = 1.5 U_{eq}(C_{methyl})] and C—H(aromatic) = 0.95Å or *C*—*H*(methyl) = 0.98 Å, respectively. The H atom bonded to N was refined isotropically, N-H 0.88 (3) Å.

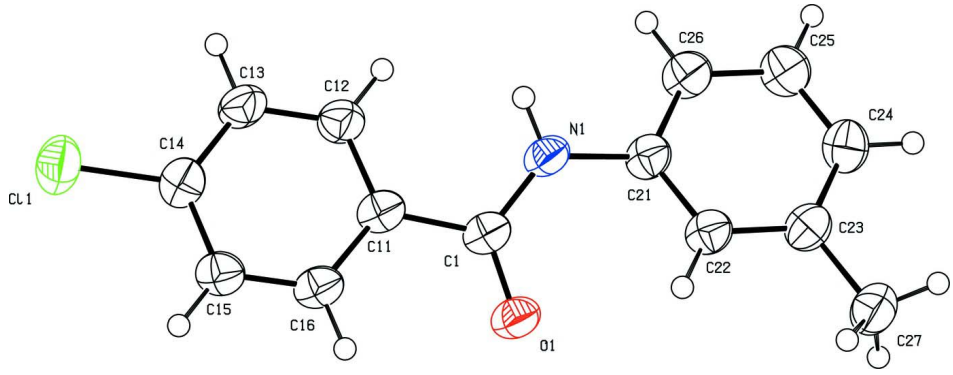


Figure 1

Perspective view of the title compound with the atom numbering scheme; displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

4-Chloro-*N*-*m*-tolylbenzamide

Crystal data

$C_{14}H_{12}ClNO$
$M_r = 245.70$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.9721\ (14)$ Å
$b = 10.1922\ (6)$ Å
$c = 9.0154\ (8)$ Å
$\beta = 105.415\ (7)°$
$V = 1237.67\ (18)$ Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.319$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10353 reflections
$\theta = 2.6–27.8°$
$\mu = 0.29$ mm^{-1}
$T = 173$ K
Block, colourless
0.26 × 0.24 × 0.23 mm

Data collection

Stoe IPDSII two-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2009; Blessing, 1995)
$T_{min} = 0.928$, $T_{max} = 0.936$
9469 measured reflections
2193 independent reflections
1787 reflections with $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
$h = -16\rightarrow16$
$k = -12\rightarrow12$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.178$
$S = 1.04$
2193 reflections
160 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.1231P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.018 (5)

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}
Cl1	0.86033 (5)	0.92615 (6)	0.74007 (9)	0.0588 (3)
O1	0.41731 (13)	0.85570 (16)	0.23364 (18)	0.0439 (5)
N1	0.38823 (16)	0.72470 (18)	0.4215 (2)	0.0393 (5)
H1	0.408 (2)	0.702 (2)	0.519 (3)	0.041 (7)*
C1	0.44505 (17)	0.80750 (19)	0.3639 (3)	0.0366 (5)
C11	0.54665 (18)	0.83650 (19)	0.4646 (3)	0.0371 (6)
C12	0.59229 (19)	0.7638 (2)	0.5951 (3)	0.0435 (6)
H12	0.5574	0.6930	0.6253	0.052*
C13	0.68742 (19)	0.7929 (2)	0.6814 (3)	0.0456 (6)
H13	0.7172	0.7434	0.7711	0.055*
C14	0.73903 (18)	0.8948 (2)	0.6362 (3)	0.0422 (6)
C15	0.69544 (19)	0.9691 (2)	0.5060 (3)	0.0413 (6)
H15	0.7311	1.0387	0.4753	0.050*
C16	0.6000 (2)	0.94034 (19)	0.4222 (3)	0.0398 (6)
H16	0.5697	0.9917	0.3343	0.048*
C21	0.29378 (19)	0.6714 (2)	0.3444 (3)	0.0388 (6)
C22	0.22613 (19)	0.7341 (2)	0.2241 (3)	0.0404 (6)
H22	0.2426	0.8166	0.1884	0.049*
C23	0.13421 (19)	0.6770 (2)	0.1555 (3)	0.0429 (6)
C24	0.1115 (2)	0.5552 (2)	0.2080 (3)	0.0468 (6)
H24	0.0499	0.5142	0.1605	0.056*
C25	0.1784 (2)	0.4941 (2)	0.3291 (3)	0.0488 (6)
H25	0.1618	0.4118	0.3651	0.059*
C26	0.2693 (2)	0.5508 (2)	0.3988 (3)	0.0452 (6)
H26	0.3145	0.5084	0.4826	0.054*
C27	0.0604 (2)	0.7461 (3)	0.0252 (3)	0.0561 (7)
H27A	0.0720	0.7202	−0.0732	0.084*
H27B	0.0686	0.8413	0.0383	0.084*
H27C	−0.0073	0.7216	0.0264	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0465 (5)	0.0523 (5)	0.0671 (6)	−0.0050 (3)	−0.0031 (4)	−0.0004 (3)
O1	0.0528 (11)	0.0441 (9)	0.0317 (9)	−0.0007 (7)	0.0059 (8)	0.0023 (7)
N1	0.0477 (12)	0.0352 (9)	0.0296 (11)	−0.0020 (8)	0.0009 (9)	0.0012 (8)

C1	0.0479 (14)	0.0276 (10)	0.0321 (12)	0.0042 (9)	0.0067 (10)	−0.0024 (8)
C11	0.0481 (14)	0.0277 (10)	0.0340 (12)	0.0040 (9)	0.0081 (11)	−0.0020 (8)
C12	0.0481 (15)	0.0363 (11)	0.0433 (14)	−0.0019 (10)	0.0070 (12)	0.0059 (10)
C13	0.0485 (15)	0.0391 (12)	0.0435 (14)	0.0040 (11)	0.0021 (12)	0.0106 (10)
C14	0.0441 (14)	0.0334 (11)	0.0458 (14)	0.0025 (10)	0.0062 (12)	−0.0051 (10)
C15	0.0522 (14)	0.0306 (11)	0.0409 (13)	−0.0014 (10)	0.0119 (12)	−0.0010 (9)
C16	0.0539 (15)	0.0283 (10)	0.0345 (13)	0.0020 (9)	0.0070 (11)	−0.0005 (9)
C21	0.0468 (13)	0.0322 (11)	0.0343 (12)	−0.0028 (9)	0.0053 (11)	−0.0059 (9)
C22	0.0487 (14)	0.0343 (11)	0.0358 (13)	−0.0011 (9)	0.0069 (11)	−0.0032 (9)
C23	0.0471 (14)	0.0448 (13)	0.0350 (13)	−0.0002 (10)	0.0080 (11)	−0.0066 (10)
C24	0.0490 (15)	0.0410 (12)	0.0478 (15)	−0.0060 (10)	0.0081 (12)	−0.0116 (10)
C25	0.0566 (15)	0.0357 (12)	0.0523 (15)	−0.0047 (11)	0.0111 (13)	−0.0041 (10)
C26	0.0546 (16)	0.0336 (11)	0.0432 (14)	−0.0011 (10)	0.0058 (12)	−0.0013 (9)
C27	0.0510 (17)	0.0587 (15)	0.0500 (16)	−0.0056 (12)	−0.0017 (13)	0.0034 (12)

Geometric parameters (Å, º)

Cl1—C14	1.734 (3)	C16—H16	0.9500
O1—C1	1.236 (3)	C21—C22	1.390 (3)
N1—C1	1.353 (3)	C21—C26	1.398 (3)
N1—C21	1.426 (3)	C22—C23	1.396 (3)
N1—H1	0.88 (3)	C22—H22	0.9500
C1—C11	1.497 (3)	C23—C24	1.394 (3)
C11—C12	1.393 (3)	C23—C27	1.515 (4)
C11—C16	1.404 (3)	C24—C25	1.383 (4)
C12—C13	1.382 (4)	C24—H24	0.9500
C12—H12	0.9500	C25—C26	1.385 (4)
C13—C14	1.386 (4)	C25—H25	0.9500
C13—H13	0.9500	C26—H26	0.9500
C14—C15	1.393 (3)	C27—H27A	0.9800
C15—C16	1.378 (4)	C27—H27B	0.9800
C15—H15	0.9500	C27—H27C	0.9800
C1—N1—C21	127.7 (2)	C22—C21—C26	119.9 (2)
C1—N1—H1	119.2 (18)	C22—C21—N1	123.7 (2)
C21—N1—H1	112.9 (18)	C26—C21—N1	116.4 (2)
O1—C1—N1	123.0 (2)	C21—C22—C23	120.6 (2)
O1—C1—C11	120.3 (2)	C21—C22—H22	119.7
N1—C1—C11	116.7 (2)	C23—C22—H22	119.7
C12—C11—C16	118.3 (2)	C24—C23—C22	119.1 (2)
C12—C11—C1	123.7 (2)	C24—C23—C27	120.5 (2)
C16—C11—C1	118.0 (2)	C22—C23—C27	120.4 (2)
C13—C12—C11	121.1 (2)	C25—C24—C23	120.1 (2)
C13—C12—H12	119.4	C25—C24—H24	119.9
C11—C12—H12	119.4	C23—C24—H24	119.9
C12—C13—C14	119.5 (2)	C24—C25—C26	121.1 (2)
C12—C13—H13	120.2	C24—C25—H25	119.5
C14—C13—H13	120.2	C26—C25—H25	119.5

C13—C14—C15	120.6 (2)	C25—C26—C21	119.2 (2)
C13—C14—Cl1	119.28 (19)	C25—C26—H26	120.4
C15—C14—Cl1	120.04 (19)	C21—C26—H26	120.4
C16—C15—C14	119.3 (2)	C23—C27—H27A	109.5
C16—C15—H15	120.4	C23—C27—H27B	109.5
C14—C15—H15	120.4	H27A—C27—H27B	109.5
C15—C16—C11	121.2 (2)	C23—C27—H27C	109.5
C15—C16—H16	119.4	H27A—C27—H27C	109.5
C11—C16—H16	119.4	H27B—C27—H27C	109.5
C21—N1—C1—O1	5.0 (4)	C12—C11—C16—C15	0.8 (3)
C21—N1—C1—C11	−173.24 (19)	C1—C11—C16—C15	−177.3 (2)
O1—C1—C11—C12	−164.2 (2)	C1—N1—C21—C22	−28.5 (4)
N1—C1—C11—C12	14.2 (3)	C1—N1—C21—C26	153.4 (2)
O1—C1—C11—C16	13.8 (3)	C26—C21—C22—C23	−0.9 (4)
N1—C1—C11—C16	−167.84 (19)	N1—C21—C22—C23	−179.0 (2)
C16—C11—C12—C13	0.2 (4)	C21—C22—C23—C24	−0.7 (4)
C1—C11—C12—C13	178.2 (2)	C21—C22—C23—C27	179.5 (2)
C11—C12—C13—C14	−1.0 (4)	C22—C23—C24—C25	1.6 (4)
C12—C13—C14—C15	0.9 (4)	C27—C23—C24—C25	−178.6 (3)
C12—C13—C14—Cl1	−177.17 (19)	C23—C24—C25—C26	−1.0 (4)
C13—C14—C15—C16	0.1 (4)	C24—C25—C26—C21	−0.6 (4)
Cl1—C14—C15—C16	178.14 (18)	C22—C21—C26—C25	1.5 (4)
C14—C15—C16—C11	−1.0 (4)	N1—C21—C26—C25	179.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88 (3)	1.99 (3)	2.854 (3)	166 (3)

Symmetry code: (i) *x*, −*y*+3/2, *z*+1/2.