

2,4-Dichloro-*N*-*p*-tolylbenzamideAamer Saeed,^{a*} Rasheed Ahmad Khera,^a Hummera Rafique,^a Jim Simpson^b and Roderick G. Stanley^b^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and ^bDepartment of Chemistry, University of Otago, PO Box 56, Dunedin, New Zealand

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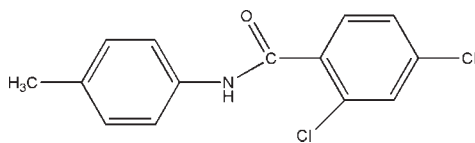
Received 2 July 2009; accepted 29 August 2009

Key indicators: single-crystal X-ray study; $T = 89$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.041; wR factor = 0.132; data-to-parameter ratio = 26.7.

In the title compound, $\text{C}_{14}\text{H}_{11}\text{Cl}_2\text{NO}$, the $\text{C}-\text{N}-\text{C}(=\text{O})-\text{C}$ amide unit is almost planar (r.m.s. deviation = 0.0317 Å) and subtends dihedral angles of 65.93 (6) and 29.45 (7)°, respectively, to the dichlorobenzene and tolyl rings. The two aromatic rings are inclined at 37.92 (6)° to one another. In the crystal structure, $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains along b . Additional weak $\text{C}-\text{H}\cdots\text{Cl}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds combine with $\text{C}-\text{H}\cdots\pi$ and very weak $\pi-\pi$ contacts [$\text{Cg}\cdots\text{Cg}$ distance = 4.0217 (12) Å] to stack the molecules down b .

Related literature

For background to our work on benzamide derivatives, see: Saeed *et al.* (2008). For related structures see: Zhou & Zheng (2007); Gowda *et al.* (2008*a,b,c*, 2009); Chopra & Guru Row (2005).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{Cl}_2\text{NO}$
 $M_r = 280.14$
 Monoclinic, $P2_1/c$
 $a = 9.0884$ (18) Å
 $b = 9.825$ (2) Å
 $c = 14.167$ (3) Å
 $\beta = 94.208$ (9)°

$V = 1261.6$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.50$ mm⁻¹
 $T = 89$ K
 $0.33 \times 0.26 \times 0.06$ mm

Data collection

Bruker APEXII CCD
 diffractometer

Absorption correction: multi-scan
 (SADABS; Bruker, 2006)
 $T_{\min} = 0.753$, $T_{\max} = 0.970$

20982 measured reflections
 4465 independent reflections

3463 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.132$
 $S = 1.15$
 4465 reflections
 167 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.57$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.86 (2)	2.14 (2)	2.9867 (17)	168 (2)
$\text{C12}-\text{H12}\cdots\text{Cl1}^{\text{ii}}$	0.95	2.91	3.7372 (17)	146
$\text{C6}-\text{H6}\cdots\text{O1}^{\text{iii}}$	0.95	2.67	3.619 (2)	175
$\text{C7}-\text{H7}\cdots\text{Cg2}^{\text{iv}}$	0.95	2.65	3.4865 (17)	147

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 1, -y + 1, -z + 2$; (iii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (iv) $x, -y + \frac{3}{2}, z + \frac{1}{2}$. Cg2 is the centroid of the C8–C13 ring.

Data collection: APEX2 (Bruker, 2006); cell refinement: APEX2 and SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and TITAN2000 (Hunter & Simpson, 1999); molecular graphics: SHELXTL (Sheldrick, 2008) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97, enCIFer (Allen *et al.*, 2004), PLATON (Spek, 2009) and publCIF (Westrip, 2009).

We thank the University of Otago for purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2252).

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supplementary materials

Acta Cryst. (2009). E65, o2527 [doi:10.1107/S1600536809034710]

2,4-Dichloro-*N-p*-tolylbenzamide

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Comment

The background to our work on benzamide derivatives has been described in a previous paper (Saeed *et al.*, 2008). In the title compound (I), Fig. 1, the C8–N1–C1(O1)–C2 amide unit is planar, r.m.s. deviation 0.0317 Å, and subtends dihedral angles of 65.93 (6)° and 29.45 (7)° respectively to the C2···C7 dichlorobenzene and C8···C13 tolyl rings. The two aromatic rings are inclined at 37.92 (6)° to one another. Bond distances within the molecule are normal and similar to those observed in comparable structures (Zhou & Zheng, 2007; Gowda *et al.* 2008a,b,c 2009; Chopra & Guru Row, 2005).

In the crystal structure N—H···O hydrogen bonds link molecules in a head to tail fashion into rows along *b*. C7—H7··· π and weak, inversion related π – π contacts involving adjacent dichlorobenzene rings [*Cg*···*Cg* distance 4.0217 (12), symmetry operation 1 – *x*, 1 – *y*, 1 – *z*] are also observed, Table 1, Fig 2. These together with additional C—H···Cl and C—H···O hydrogen bonds link the stacks of molecules alternately head to head and head to tail down the *b* axis, Fig. 3.

Experimental

2,4-Dichlorobenzoyl chloride (5.4 mmol) in CHCl₃ was treated with *p*-toluidine(21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with CHCl₃ and washed consecutively with aq 1 M HCl and saturated aq NaHCO₃. The organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Crystallization of the residue by evaporation from CHCl₃ afforded the title compound (84%) as colourless needles: Anal. calcd. for C₁₄H₁₁Cl₂NO: C, 60.02; H, 3.96; N, 5.00%; found: C, 60.06; H, 3.92; N, 5.10%

Refinement

The H atom bound to N1 was located in a difference Fourier map and its coordinates were refined with $U_{\text{iso}}=1.2U_{\text{eq}}$ (N). All other H-atoms were placed in calculated positions and refined using a riding model with $d(\text{C—H}) = 0.95$ Å, $U_{\text{iso}}=1.2U_{\text{eq}}$ (C) for aromatic and 0.98 Å, $U_{\text{iso}} = 1.5U_{\text{eq}}$ (C) for CH₃ H atoms.

Figures

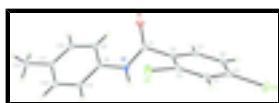


Fig. 1. The structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level.

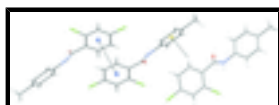


Fig. 2. π – π and C—H··· π interactions in (I). Contacts are shown as dotted lines, the coloured spheres represent the ring centroids.

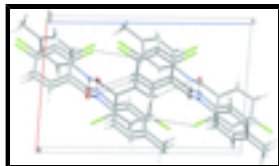


Fig. 3. Crystal packing of (I) viewed down the *b* axis, with hydrogen bonds drawn as dashed lines.

2,4-Dichloro-*N*-*p*-tolylbenzamide

Crystal data

$C_{14}H_{11}Cl_2NO$

$M_r = 280.14$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.0884$ (18) Å

$b = 9.825$ (2) Å

$c = 14.167$ (3) Å

$\beta = 94.208$ (9)°

$V = 1261.6$ (4) Å³

$Z = 4$

$F_{000} = 576$

$D_x = 1.475$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5859 reflections

$\theta = 2.5$ – 33.0 °

$\mu = 0.50$ mm⁻¹

$T = 89$ K

Irregular fragment, colourless

$0.33 \times 0.26 \times 0.06$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 89$ K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2006)

$T_{\min} = 0.753$, $T_{\max} = 0.970$

20982 measured reflections

4465 independent reflections

3463 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.044$

$\theta_{\text{max}} = 33.1$ °

$\theta_{\text{min}} = 2.9$ °

$h = -13 \rightarrow 13$

$k = -14 \rightarrow 13$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.132$

$S = 1.15$

4465 reflections

167 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 0.2064P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.57$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Primary atom site location: structure-invariant direct methods Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.52742 (16)	0.71474 (14)	0.71812 (10)	0.0111 (3)
C2	0.43510 (16)	0.67111 (13)	0.63071 (10)	0.0105 (3)
C3	0.29263 (16)	0.61878 (14)	0.63334 (10)	0.0121 (3)
C4	0.21096 (16)	0.57806 (14)	0.55106 (10)	0.0126 (3)
H4	0.1147	0.5414	0.5539	0.015*
C5	0.27317 (16)	0.59219 (14)	0.46479 (10)	0.0125 (3)
C6	0.41328 (16)	0.64737 (14)	0.45883 (10)	0.0130 (3)
H6	0.4536	0.6584	0.3993	0.016*
C7	0.49281 (16)	0.68595 (14)	0.54224 (11)	0.0125 (3)
H7	0.5887	0.7233	0.5391	0.015*
C8	0.66786 (16)	0.62192 (14)	0.86070 (10)	0.0109 (3)
C9	0.77909 (17)	0.71922 (15)	0.87165 (11)	0.0141 (3)
H9	0.7902	0.7855	0.8239	0.017*
C10	0.87396 (17)	0.71820 (15)	0.95361 (11)	0.0153 (3)
H10	0.9498	0.7847	0.9607	0.018*
C11	0.86130 (16)	0.62256 (14)	1.02572 (11)	0.0131 (3)
C12	0.74965 (17)	0.52520 (15)	1.01274 (11)	0.0142 (3)
H12	0.7388	0.4585	1.0603	0.017*
C13	0.65393 (16)	0.52412 (14)	0.93137 (10)	0.0123 (3)
H13	0.5789	0.4568	0.9238	0.015*
C14	0.96308 (18)	0.62590 (17)	1.11523 (11)	0.0184 (3)
H14A	0.9120	0.6683	1.1662	0.028*
H14B	1.0515	0.6788	1.1040	0.028*
H14C	0.9916	0.5328	1.1334	0.028*
N1	0.56897 (14)	0.61267 (12)	0.77821 (9)	0.0120 (2)
O1	0.56444 (13)	0.83472 (10)	0.72985 (8)	0.0168 (2)
Cl1	0.20933 (4)	0.60815 (4)	0.73967 (3)	0.01815 (11)
Cl2	0.17246 (4)	0.53821 (4)	0.36221 (3)	0.01910 (11)
H1N	0.530 (2)	0.534 (2)	0.7672 (16)	0.023*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0116 (6)	0.0104 (6)	0.0112 (6)	0.0005 (5)	0.0004 (5)	0.0007 (5)
C2	0.0122 (6)	0.0073 (6)	0.0117 (6)	0.0008 (4)	-0.0010 (5)	0.0009 (4)
C3	0.0135 (6)	0.0120 (6)	0.0107 (6)	0.0020 (5)	0.0011 (5)	0.0023 (5)
C4	0.0108 (6)	0.0127 (6)	0.0141 (7)	-0.0008 (5)	-0.0005 (5)	0.0009 (5)
C5	0.0145 (6)	0.0111 (6)	0.0115 (6)	0.0015 (5)	-0.0020 (5)	-0.0012 (5)
C6	0.0154 (6)	0.0114 (6)	0.0123 (6)	0.0003 (5)	0.0022 (5)	0.0005 (5)
C7	0.0127 (6)	0.0095 (6)	0.0153 (7)	-0.0011 (5)	0.0005 (5)	0.0005 (5)
C8	0.0121 (6)	0.0091 (6)	0.0112 (6)	0.0016 (4)	-0.0006 (5)	-0.0013 (4)
C9	0.0154 (6)	0.0127 (6)	0.0137 (6)	-0.0026 (5)	-0.0014 (5)	0.0035 (5)
C10	0.0141 (7)	0.0134 (6)	0.0179 (7)	-0.0031 (5)	-0.0015 (6)	0.0003 (5)
C11	0.0117 (6)	0.0141 (6)	0.0133 (6)	0.0011 (5)	-0.0008 (5)	-0.0002 (5)
C12	0.0166 (7)	0.0131 (6)	0.0125 (6)	-0.0014 (5)	-0.0008 (5)	0.0026 (5)
C13	0.0130 (6)	0.0103 (6)	0.0134 (6)	-0.0019 (5)	-0.0001 (5)	0.0005 (5)
C14	0.0182 (7)	0.0217 (7)	0.0145 (7)	-0.0031 (6)	-0.0037 (6)	0.0012 (6)
N1	0.0154 (6)	0.0084 (5)	0.0115 (6)	-0.0014 (4)	-0.0034 (5)	0.0006 (4)
O1	0.0210 (6)	0.0084 (5)	0.0199 (5)	-0.0009 (4)	-0.0056 (4)	0.0009 (4)
Cl1	0.01473 (18)	0.0284 (2)	0.01157 (18)	0.00040 (13)	0.00261 (13)	0.00371 (13)
Cl2	0.01772 (19)	0.0254 (2)	0.01362 (18)	-0.00149 (13)	-0.00263 (14)	-0.00494 (13)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.2338 (17)	C8—C13	1.400 (2)
C1—N1	1.3512 (18)	C8—N1	1.4240 (18)
C1—C2	1.5062 (19)	C9—C10	1.395 (2)
C2—C3	1.396 (2)	C9—H9	0.9500
C2—C7	1.401 (2)	C10—C11	1.399 (2)
C3—C4	1.394 (2)	C10—H10	0.9500
C3—C11	1.7379 (16)	C11—C12	1.397 (2)
C4—C5	1.391 (2)	C11—C14	1.514 (2)
C4—H4	0.9500	C12—C13	1.392 (2)
C5—C6	1.392 (2)	C12—H12	0.9500
C5—C12	1.7423 (15)	C13—H13	0.9500
C6—C7	1.392 (2)	C14—H14A	0.9800
C6—H6	0.9500	C14—H14B	0.9800
C7—H7	0.9500	C14—H14C	0.9800
C8—C9	1.392 (2)	N1—H1N	0.86 (2)
O1—C1—N1	124.29 (13)	C8—C9—C10	119.17 (13)
O1—C1—C2	120.84 (12)	C8—C9—H9	120.4
N1—C1—C2	114.82 (12)	C10—C9—H9	120.4
C3—C2—C7	118.10 (13)	C9—C10—C11	122.27 (14)
C3—C2—C1	122.99 (13)	C9—C10—H10	118.9
C7—C2—C1	118.90 (13)	C11—C10—H10	118.9
C4—C3—C2	121.42 (14)	C12—C11—C10	117.50 (13)
C4—C3—C11	117.94 (12)	C12—C11—C14	121.22 (14)

C2—C3—C11	120.61 (11)	C10—C11—C14	121.27 (13)
C5—C4—C3	118.71 (14)	C13—C12—C11	121.19 (14)
C5—C4—H4	120.6	C13—C12—H12	119.4
C3—C4—H4	120.6	C11—C12—H12	119.4
C4—C5—C6	121.66 (13)	C12—C13—C8	120.20 (13)
C4—C5—C12	118.69 (11)	C12—C13—H13	119.9
C6—C5—C12	119.65 (12)	C8—C13—H13	119.9
C7—C6—C5	118.36 (14)	C11—C14—H14A	109.5
C7—C6—H6	120.8	C11—C14—H14B	109.5
C5—C6—H6	120.8	H14A—C14—H14B	109.5
C6—C7—C2	121.72 (13)	C11—C14—H14C	109.5
C6—C7—H7	119.1	H14A—C14—H14C	109.5
C2—C7—H7	119.1	H14B—C14—H14C	109.5
C9—C8—C13	119.66 (13)	C1—N1—C8	126.87 (12)
C9—C8—N1	123.01 (13)	C1—N1—H1N	117.5 (14)
C13—C8—N1	117.28 (12)	C8—N1—H1N	115.6 (14)
O1—C1—C2—C3	-115.79 (17)	C1—C2—C7—C6	179.87 (12)
N1—C1—C2—C3	66.64 (18)	C13—C8—C9—C10	-0.6 (2)
O1—C1—C2—C7	62.85 (19)	N1—C8—C9—C10	-178.01 (14)
N1—C1—C2—C7	-114.72 (15)	C8—C9—C10—C11	-0.1 (2)
C7—C2—C3—C4	2.0 (2)	C9—C10—C11—C12	0.6 (2)
C1—C2—C3—C4	-179.31 (13)	C9—C10—C11—C14	-178.33 (15)
C7—C2—C3—C11	-175.69 (10)	C10—C11—C12—C13	-0.5 (2)
C1—C2—C3—C11	2.96 (19)	C14—C11—C12—C13	178.50 (14)
C2—C3—C4—C5	-0.9 (2)	C11—C12—C13—C8	-0.2 (2)
C11—C3—C4—C5	176.86 (11)	C9—C8—C13—C12	0.8 (2)
C3—C4—C5—C6	-0.9 (2)	N1—C8—C13—C12	178.32 (13)
C3—C4—C5—C12	178.77 (11)	O1—C1—N1—C8	-4.3 (2)
C4—C5—C6—C7	1.5 (2)	C2—C1—N1—C8	173.21 (13)
C12—C5—C6—C7	-178.17 (11)	C9—C8—N1—C1	-26.7 (2)
C5—C6—C7—C2	-0.3 (2)	C13—C8—N1—C1	155.92 (15)
C3—C2—C7—C6	-1.4 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O1 ⁱ	0.86 (2)	2.14 (2)	2.9867 (17)	168 (2)
C12—H12...C11 ⁱⁱ	0.95	2.91	3.7372 (17)	146
C6—H6...O1 ⁱⁱⁱ	0.95	2.67	3.619 (2)	175
C7—H7...Cg2 ^{iv}	0.95	2.65	3.4865 (17)	147

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $-x+1, -y+1, -z+2$; (iii) $x, -y+3/2, z-1/2$; (iv) $x, -y+3/2, z+1/2$.

Fig. 1

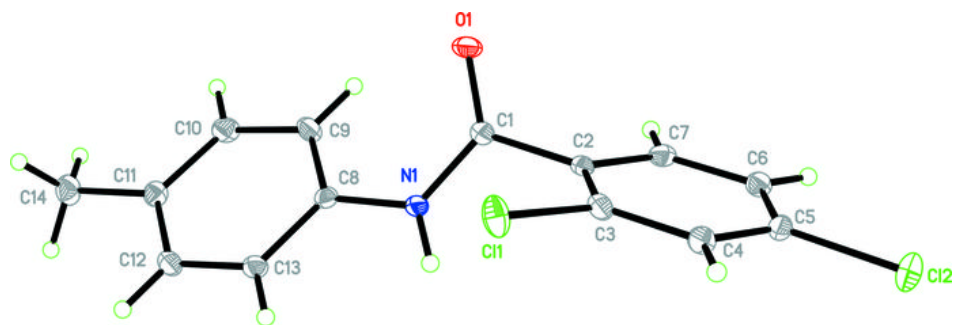


Fig. 2

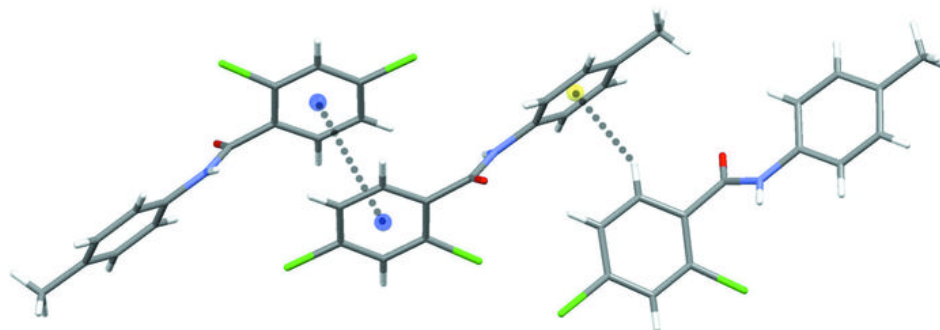


Fig. 3

