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## Structure Reports

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## 2,6-Dichloro-N-(4-methylphenyl)-benzamide

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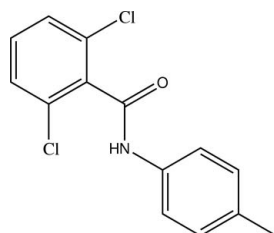
Received 25 March 2012; accepted 6 April 2012

 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.053;  $wR$  factor = 0.150; data-to-parameter ratio = 15.4.

In the title compound,  $\text{C}_{14}\text{H}_{11}\text{Cl}_2\text{NO}$ , the two benzene rings are non-coplanar [dihedral angle =  $60.9$  ( $3$ )°]. In the crystal, an amide  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond links the molecules into chains which extend along (001).

### Related literature

For the synthesis of the title compound, see: Houlihan *et al.* (1981). For bond-length data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

$\text{C}_{14}\text{H}_{11}\text{Cl}_2\text{NO}$   
 $M_r = 280.14$   
 Monoclinic,  $P2_1/c$   
 $a = 11.260$  (2) Å  
 $b = 12.786$  (3) Å

$c = 9.6700$  (19) Å  
 $\beta = 100.65$  (3)°  
 $V = 1368.2$  (5) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.46$  mm<sup>-1</sup>  
 $T = 293$  K

$0.30 \times 0.10 \times 0.10$  mm

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.874$ ,  $T_{\max} = 0.955$   
 2650 measured reflections

2518 independent reflections  
 1514 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$   
 3 standard reflections every 200 reflections  
 intensity decay: 1%

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$   
 $wR(F^2) = 0.150$   
 $S = 1.01$   
 2518 reflections

163 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.22$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.26$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}-\text{H}0\text{A}\cdots\text{O}^i$	0.86	1.98	2.839 (4)	173

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

The authors thank Liu Bo Nian from Nanjing University of Technology for useful discussions and the Center of Testing and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2197).

### References

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## supporting information

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## 2,6-Dichloro-*N*-(4-methylphenyl)benzamide

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### S1. Comment

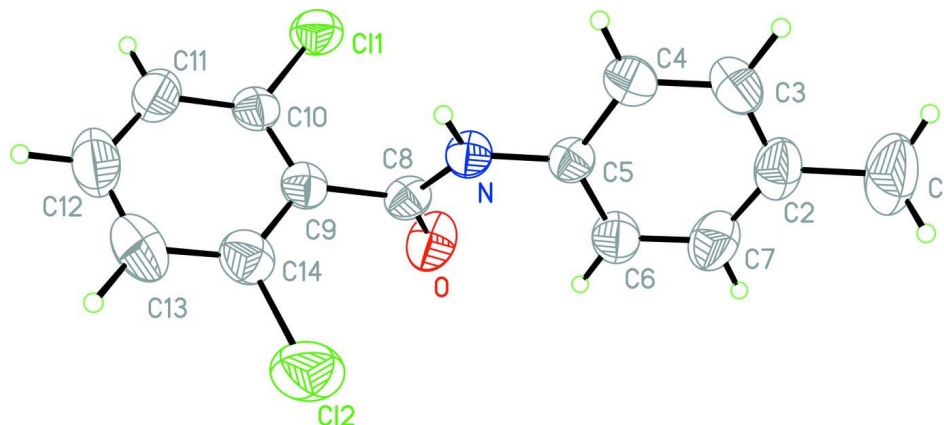
We report here the crystal structure of the title compound  $C_{14}H_{11}Cl_2NO$ . In this molecule (Fig. 1), the phenyl and dichlorophenyl rings are non-coplanar [dihedral angle  $60.9(3)^\circ$ ]. In the crystal structure an intermolecular amide  $N-H\cdots O$  hydrogen bond (Table 1) links the molecules, giving one-dimensional chains which extend along (001) (Fig. 2).

### S2. Experimental

A mixture of 4-methylbenzenamine (3.2 g, 0.03 mol), 2,6-dichlorobenzoyl chloride (6.3 g, 0.03 mol), and 6 ml of triethylamine in 50 ml of anhydrous tetrahydrofuran was refluxed with stirring for 8 h and then allowed to stand at room temperature. The resulting solids were filtered off and washed with water (2 x 30 mL) then dried, giving 7.2 g of product. Recrystallization from ethanol gave yellow crystals of the title compound. Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

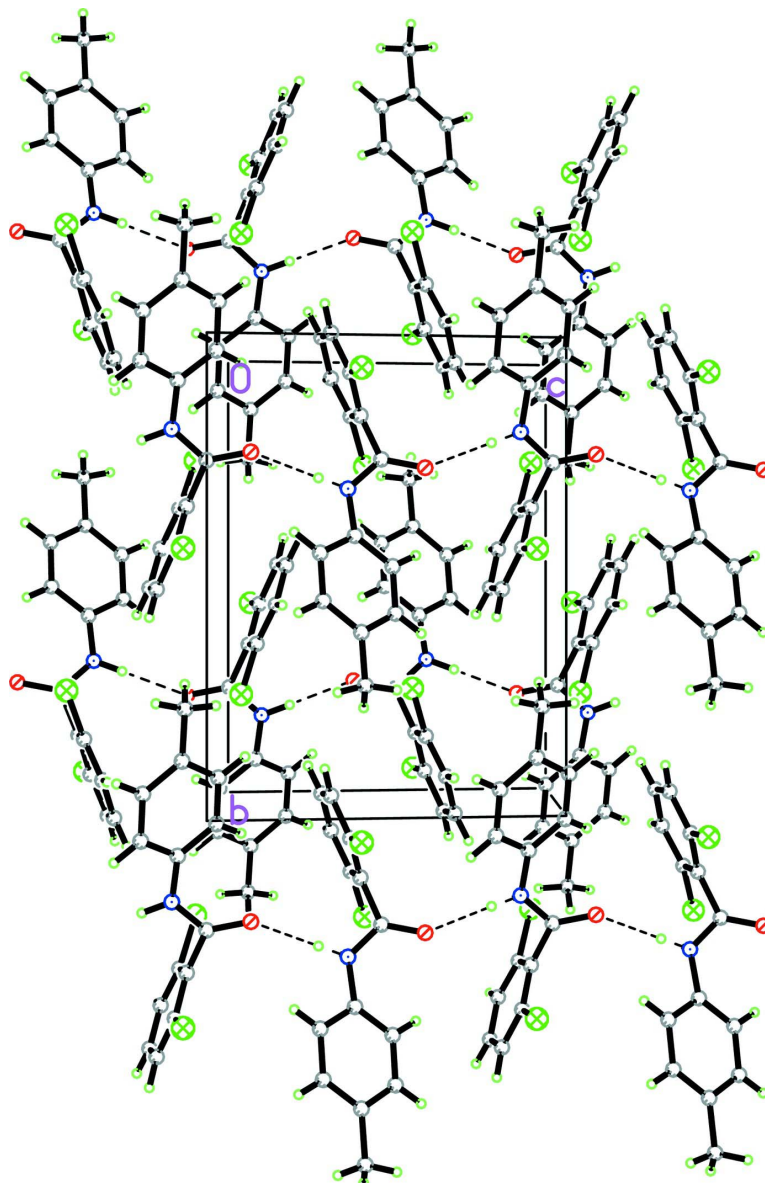
### S3. Refinement

Hydrogen atoms were positioned geometrically, with  $C-H = 0.93 \text{ \AA}$  (aromatic) or  $0.96 \text{ \AA}$  (methyl) and  $N-H = 0.86 \text{ \AA}$  and were allowed to ride on their parent atoms, with  $U_{iso}(H) = 1.2U_{eq}(\text{aromatic C, N})$  or  $1.5U_{eq}(\text{methyl C})$ .



**Figure 1**

The molecular structure of the title compound showing the atom-numbering scheme, with displacement ellipsoids drawn at the 30% probability level.



**Figure 2**

A packing diagram of the title compound viewed down  $a$ , with intermolecular hydrogen bonds shown as dashed lines.

### 2,6-Dichloro-*N*-(4-methylphenyl)benzamide

#### *Crystal data*

$C_{14}H_{11}Cl_2NO$

$M_r = 280.14$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 11.260\ (2)\ \text{\AA}$

$b = 12.786\ (3)\ \text{\AA}$

$c = 9.6700\ (19)\ \text{\AA}$

$\beta = 100.65\ (3)^\circ$

$V = 1368.2\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 576$

$D_x = 1.360\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 9\text{--}13^\circ$

$\mu = 0.46\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, yellow

$0.30 \times 0.10 \times 0.10\ \text{mm}$

*Data collection*

Enraf–Nonius CAD-4  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$  scans

Absorption correction:  $\psi$  scan  
(North *et al.*, 1968)

$T_{\min} = 0.874$ ,  $T_{\max} = 0.955$

2650 measured reflections

2518 independent reflections

1514 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 25.4^\circ$ ,  $\theta_{\min} = 1.8^\circ$

$h = -13 \rightarrow 0$

$k = 0 \rightarrow 15$

$l = -11 \rightarrow 11$

3 standard reflections every 200 reflections

intensity decay: 1%

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.150$

$S = 1.01$

2518 reflections

163 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-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.070P)^2]$

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

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

$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N	0.2641 (2)	0.8121 (2)	0.1158 (3)	0.0404 (7)
H0A	0.2757	0.7889	0.2007	0.048*
O	0.2803 (3)	0.76570 (19)	-0.1064 (2)	0.0660 (8)
Cl1	0.14387 (9)	0.55959 (8)	0.07524 (10)	0.0635 (3)
C1	0.0707 (4)	1.2269 (3)	0.0646 (6)	0.0969 (17)
H1A	0.0283	1.2398	0.1404	0.145*
H1B	0.0162	1.2352	-0.0236	0.145*
H1C	0.1361	1.2758	0.0699	0.145*
Cl2	0.56778 (10)	0.75531 (9)	0.07081 (15)	0.0876 (4)
C2	0.1203 (3)	1.1167 (3)	0.0760 (5)	0.0601 (11)
C3	0.1026 (4)	1.0507 (3)	0.1828 (4)	0.0629 (11)
H3A	0.0596	1.0745	0.2500	0.076*
C4	0.1474 (3)	0.9499 (3)	0.1924 (4)	0.0533 (9)
H4A	0.1334	0.9065	0.2649	0.064*
C5	0.2128 (3)	0.9131 (2)	0.0952 (3)	0.0395 (8)

C6	0.2314 (3)	0.9779 (3)	-0.0124 (4)	0.0562 (10)
H6A	0.2750	0.9544	-0.0791	0.067*
C7	0.1848 (4)	1.0781 (3)	-0.0206 (4)	0.0661 (11)
H7A	0.1975	1.1210	-0.0941	0.079*
C8	0.2970 (3)	0.7476 (3)	0.0202 (3)	0.0423 (8)
C9	0.3616 (3)	0.6510 (3)	0.0813 (3)	0.0418 (8)
C10	0.3010 (3)	0.5611 (3)	0.1098 (3)	0.0445 (8)
C11	0.3614 (4)	0.4729 (3)	0.1667 (4)	0.0592 (10)
H11A	0.3192	0.4132	0.1837	0.071*
C12	0.4849 (4)	0.4751 (4)	0.1976 (5)	0.0730 (13)
H12A	0.5265	0.4165	0.2380	0.088*
C13	0.5485 (4)	0.5612 (4)	0.1707 (4)	0.0710 (12)
H13A	0.6324	0.5614	0.1921	0.085*
C14	0.4868 (3)	0.6472 (3)	0.1116 (4)	0.0550 (10)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N	0.0548 (17)	0.0427 (16)	0.0246 (13)	0.0042 (13)	0.0096 (12)	0.0009 (12)
O	0.112 (2)	0.0577 (16)	0.0291 (13)	0.0081 (15)	0.0157 (13)	-0.0012 (12)
C11	0.0531 (6)	0.0723 (7)	0.0669 (7)	-0.0039 (5)	0.0156 (5)	0.0100 (5)
C1	0.092 (4)	0.054 (3)	0.139 (5)	0.020 (3)	0.007 (3)	0.002 (3)
C12	0.0715 (8)	0.0718 (8)	0.1247 (11)	-0.0208 (6)	0.0314 (7)	-0.0195 (7)
C2	0.049 (2)	0.044 (2)	0.082 (3)	0.0040 (18)	0.000 (2)	-0.003 (2)
C3	0.062 (3)	0.060 (3)	0.072 (3)	0.008 (2)	0.027 (2)	-0.010 (2)
C4	0.060 (2)	0.054 (2)	0.050 (2)	0.0024 (19)	0.0204 (18)	-0.0009 (18)
C5	0.0430 (18)	0.0413 (19)	0.0337 (17)	-0.0001 (15)	0.0059 (14)	0.0015 (15)
C6	0.074 (3)	0.052 (2)	0.047 (2)	0.009 (2)	0.0222 (19)	0.0045 (18)
C7	0.083 (3)	0.051 (2)	0.063 (3)	0.003 (2)	0.010 (2)	0.013 (2)
C8	0.051 (2)	0.0449 (19)	0.0318 (18)	-0.0029 (16)	0.0087 (15)	0.0000 (16)
C9	0.050 (2)	0.045 (2)	0.0323 (17)	0.0039 (16)	0.0115 (15)	-0.0071 (15)
C10	0.050 (2)	0.051 (2)	0.0338 (17)	0.0035 (18)	0.0131 (15)	-0.0029 (17)
C11	0.072 (3)	0.051 (2)	0.057 (2)	0.008 (2)	0.018 (2)	0.0078 (18)
C12	0.073 (3)	0.067 (3)	0.077 (3)	0.027 (3)	0.011 (2)	0.009 (2)
C13	0.048 (2)	0.086 (3)	0.076 (3)	0.016 (2)	0.002 (2)	-0.004 (3)
C14	0.054 (2)	0.053 (2)	0.060 (2)	-0.0002 (19)	0.0143 (18)	-0.0102 (19)

*Geometric parameters (Å, °)*

N—C8	1.341 (4)	C4—H4A	0.9300
N—C5	1.414 (4)	C5—C6	1.376 (4)
N—H0A	0.8600	C6—C7	1.381 (5)
O—C8	1.225 (4)	C6—H6A	0.9300
C11—C10	1.739 (4)	C7—H7A	0.9300
C1—C2	1.513 (5)	C8—C9	1.498 (5)
C1—H1A	0.9600	C9—C14	1.387 (5)
C1—H1B	0.9600	C9—C10	1.390 (5)
C1—H1C	0.9600	C10—C11	1.379 (5)

C12—C14	1.740 (4)	C11—C12	1.367 (6)
C2—C7	1.376 (5)	C11—H11A	0.9300
C2—C3	1.377 (5)	C12—C13	1.365 (6)
C3—C4	1.381 (5)	C12—H12A	0.9300
C3—H3A	0.9300	C13—C14	1.368 (5)
C4—C5	1.379 (4)	C13—H13A	0.9300
C8—N—C5	128.5 (3)	C2—C7—C6	122.4 (4)
C8—N—H0A	115.8	C2—C7—H7A	118.8
C5—N—H0A	115.8	C6—C7—H7A	118.8
C2—C1—H1A	109.5	O—C8—N	124.2 (3)
C2—C1—H1B	109.5	O—C8—C9	121.6 (3)
H1A—C1—H1B	109.5	N—C8—C9	114.2 (3)
C2—C1—H1C	109.5	C14—C9—C10	116.5 (3)
H1A—C1—H1C	109.5	C14—C9—C8	120.8 (3)
H1B—C1—H1C	109.5	C10—C9—C8	122.7 (3)
C7—C2—C3	117.2 (4)	C11—C10—C9	122.1 (3)
C7—C2—C1	121.3 (4)	C11—C10—C11	118.5 (3)
C3—C2—C1	121.5 (4)	C9—C10—C11	119.4 (3)
C2—C3—C4	121.4 (4)	C12—C11—C10	118.5 (4)
C2—C3—H3A	119.3	C12—C11—H11A	120.7
C4—C3—H3A	119.3	C10—C11—H11A	120.7
C5—C4—C3	120.5 (3)	C13—C12—C11	121.5 (4)
C5—C4—H4A	119.8	C13—C12—H12A	119.2
C3—C4—H4A	119.8	C11—C12—H12A	119.2
C6—C5—C4	119.0 (3)	C12—C13—C14	119.0 (4)
C6—C5—N	122.8 (3)	C12—C13—H13A	120.5
C4—C5—N	118.1 (3)	C14—C13—H13A	120.5
C5—C6—C7	119.5 (3)	C13—C14—C9	122.3 (4)
C5—C6—H6A	120.2	C13—C14—C12	119.1 (3)
C7—C6—H6A	120.2	C9—C14—C12	118.6 (3)
C7—C2—C3—C4	-0.2 (6)	O—C8—C9—C10	-96.5 (4)
C1—C2—C3—C4	179.6 (4)	N—C8—C9—C10	85.6 (4)
C2—C3—C4—C5	0.8 (6)	C14—C9—C10—C11	0.7 (5)
C3—C4—C5—C6	-0.8 (5)	C8—C9—C10—C11	-179.7 (3)
C3—C4—C5—N	175.4 (3)	C14—C9—C10—C11	179.8 (2)
C8—N—C5—C6	-26.7 (5)	C8—C9—C10—C11	-0.5 (4)
C8—N—C5—C4	157.2 (3)	C9—C10—C11—C12	1.0 (5)
C4—C5—C6—C7	0.2 (5)	C11—C10—C11—C12	-178.2 (3)
N—C5—C6—C7	-175.8 (3)	C10—C11—C12—C13	-1.4 (6)
C3—C2—C7—C6	-0.4 (6)	C11—C12—C13—C14	0.2 (7)
C1—C2—C7—C6	179.8 (4)	C12—C13—C14—C9	1.6 (6)
C5—C6—C7—C2	0.4 (6)	C12—C13—C14—C12	-177.5 (3)
C5—N—C8—O	-4.5 (5)	C10—C9—C14—C13	-2.0 (5)
C5—N—C8—C9	173.4 (3)	C8—C9—C14—C13	178.4 (3)
O—C8—C9—C14	83.1 (4)	C10—C9—C14—C12	177.1 (2)
N—C8—C9—C14	-94.8 (4)	C8—C9—C14—C12	-2.5 (4)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N—H0 <i>A</i> $\cdots$ O <sup>i</sup>	0.86	1.98	2.839 (4)	173

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